UNIVERSITEIT GENT

Faculty of Arts and Philosophy
January 2008

ARCHAEOMETALLURGICAL ANALYSES OF PRE-ISLAMIC ARTEFACTS FROM ED-DUR

(Emirate of Umm al-Qaiwain, U.A.E.)

Parsival DELRUE

Promotor: Prof. dr. E. Haerinck
Department of Languages and Cultures of the Near East and North Africa – Research Unit Archaeology

Co-promotor: Prof. dr. ir. Y. Houbaert
Department of Materials Science and Metallurgy

Dean: Prof. dr. F. Mortier

Vice-chancellor: Prof. dr. P. Van Cauwenberge

PhD dissertation submitted to obtain the degree of Doctor in the Archaeology – 2007-2008

UNIVERSITEIT GENT

Faculty of Arts and Philosophy – January 2008

ARCHAEOMETALLURGICAL ANALYSES OF PRE-ISLAMIC ARTEFACTS FROM ED-DUR (Emirate of Umm al-Qaiwain, U.A.E.)

Parsival DELRUE

Promotor : Prof. dr. E. Haerinck
Department of Languages and Cultures of the Near East and North Africa – Research Unit Archaeology

Co-Promotor : Prof. dr. ir. Y. Houbaert
Department of Metallurgy and Materials Science

Dean : Prof. dr. F. Mortier

Rector : Prof. dr. P. Van Cauwenberge

PhD dissertation submitted to obtain the degree of Doctor in the Archaeology – 2007-2008

UNIVERSITEIT GENT

Faculty of Arts and Philosophy – January 2008

ARCHAEOMETALLURGICAL ANALYSES OF PRE-ISLAMIC ARTEFACTS FROM ED-DUR (Emirate of Umm al-Qaiwain, U.A.E.)

Parsival DELRUE

Dedicated to the ones I lost during this research

"*Still, it is an error to argue in front of your data, you find yourself impossibly twisting them around to fit your theories.*"

Sherlock Holmes

TABLE OF CONTENTS

PART II – The unknown

PART III – The new

PART IV – The tail

ABSTRACT & KEYWORDS – SAMENVATTING & SLEUTELWOORDEN

• **Abstract**

The aim of this PhD thesis is the archaeometallurgical analysis of the metal samples collected during excavations by Ghent University at ed-Dur (Umm al-Qaiwain, United Arab Emirates). The site of ed-Dur is situated on the west coast of the Oman Peninsula. It is the only large coastal site identified so far between Qatar and the straits of Hormuz with a main occupation from the 1st c BC until the 1st half of the 2nd c AD. The excavations at the site revealed the remains of at least one small fort and more importantly a square temple with altars dedicated to the Semitic sun god Shams/Shamash. A number of beach-rock buildings and plastered floors were found, but most living structures were probably made of perishable materials. The vast majority of the architectural remains belong to stone-built tombs of various types.

The metallurgical samples include copper and copper-base alloys, lead, silver, litharge, a collection of local SE-Arabian coinage, iron and metal slag. This wide array of materials is studied for its microstructure by optical metallography, for its chemical bulk composition by SEM-EDX and for the lead isotopic ratio and trace elements by ICP-MS. All the obtained information is used to characterise the metal assemblage from ed-Dur and to place this information in a broader frame. The ultimate goal is to better understand the function and reason for the relatively short-lived heydays of ed-Dur. The many foreign objects excavated at ed-Dur evidence that the site was in one way or another involved in the international trading networks operating during the 1st c BC until the 1st half of the 2nd c AD.

The lead isotope analyses showed that the lead was of European origin, most probably from Spain or Sardinia. This conclusion in backed by the textual evidence of the *Periplus* that states that the Roman Empire was exporting lead (to India) via the Red Sea route. The lead might however have arrived at ed-Dur from the second major trading artery, the *Characenean corridor* or by a re-exportation from India Subcontinent towards the Gulf. Two fragments from ed-Dur stand apart and are very likely from Indian origin. If this is true, then the *Periplus* is contradicted in that it states that India was not producing any lead at the period under consideration.

The lead isotopic fingerprint of the silver shows significant overlap with the ores from Great Britain. This again underlines the extensive trade network operating during the occupation of ed-Dur.

A very unexpected determination were the three pieces of litharge. The litharge fragments from ed-Dur are related with the extraction of silver from a silver-copper alloy. This technology of cupellation was previously unattested in the Gulf region during the period under consideration.

Next to the expected copper and bronze object, two other categories were attested, i.e. brass and gunmetal. A significant part of the analysed samples and objects were made of brass, a rather unexpected result. There are very strong indications that the brass is from Roman origin, although it was sometimes used for object that certainly were not Roman in origin. Some interesting conclusions on the circulation of this metal can be drawn from that.

For the first time a relative large collection of local SE-Arabian coins was systematically analysed (*ca*. 8% of all published specimens). The analyses showed that there is a broad relation between the typology and the chemical composition of the types, pointing towards intentional use of specific alloys. Additionally evidence was found for a process of silver surface enrichment or *pickling* of some of the coins. A limited experimental part was undertaken to evaluated the confines of this technique.

The iron objects were to severely corroded to extract any useful information. The slags found on the site prove however that iron smithing activities took place at ed-Dur and that the slag is not related to any other metallurgical process. The chemical composition of the slags is in accordance with the limited published evidence of the Gulf region.

Keywords:
Archaeometallurgy, SE-Arabia, United Arab Emirates, Umm al-Qaiwain, ed-Dur, optical metallography, SEM-EDX, chemical composition, numismatics, local coins, lead, litharge, cupellation, silver, copper, copper-base alloys, bronze, brass, gunmetal, iron, metal slags, ICP-MS, lead isotope analyses, trace elements, trade.

• **Samenvatting**

Het doel van deze doctoraatsscriptie is de archeometallurgische analyse van de metaalmonsters verzameld tijdens de Belgische opgravingen van de Universiteit Gent te ed-Dur (Umm al-Qaiwain, Verenigde Arabische Emiraten). De site van ed-Dur is gelegen op de westkust van de Peninsula van Oman. Het is de enige grote kustsite die tot nu toe is geattesteerd tussen Katar en de Straat van Hormuz. De belangrijkste bewoningsfase van ed-Dur is tussen de 1ste eeuw v. Ch. en de eerste helft van de 2de eeuw n. Ch. te dateren. De opgraving bracht de resten van minstens één fort en een tempel aan het licht. De tempel was gewijd aan de Semitische zonnegod Shams/Shamash en rond het gebouw werden verschillend altaren aangetroffen. Verder zijn er nog een aantal gebouwen in *beach-rock* en gepleisterde vloeren opgegraven, maar het merendeel van de bewoningstructuren was waarschijnlijk uit vergankelijke materialen vervaardigd. De overgrote meerderheid van de architecturale resten zijn afkomstig van verschillende types van graven.

De metallurgische monsters omvatten koper en koperlegeringen, lood, zilver, loodglit, een collectie locale Zuidoost Arabische munten, ijzer en metaalslakken. De microstructuur van deze ruime waaier aan materialen werd eerst via optische metallografie onderzocht. De globale chemische samenstelling werd bepaald via SEM-EDX. De loodisotopen radio's en de sporenelementen concentraties werden onderzocht met ICP-MS. Al deze informatie werd aangewend om de metallurgische collectie te karakteriseren en de verkregen informatie in een ruimer kader te plaatsen. Het ultieme doel is om gegevens aan te brengen die kunnen dienen om de functie van het relatie kort bewoonde ed-Dur te verduidelijken. De vele buitenlandse objecten die opgegraven werden tonen aan dat ed-Dur op één of andere manier was geïntegreerd in de grote handelsnetwerken van toen.

De loodisotopen van de loden objecten tonen aan dat het metaal van Europese oorsprong is, hoogstwaarschijnlijk van Spanje of Sardinië. Deze conclusie wordt ondersteund door de tekstuele informatie van de *Periplus*. Deze tekst zegt dat het Romeinse Rijk lood exporteerde (naar India) via de Rode Zee. Het lood van ed-Dur zou echter ook via een andere weg aangevoerd kunnen zijn, namelijk lang de tweede grote handelsader de *Characenean corridor*. Een derde optie is dat lood geherexporteerd werd vanuit het Indisch Subcontinent. Twee geanalyseerde fragmenten hebben echter een volledige andere isotopische signatuur en zijn erg waarschijnlijk van Indische herkomst. Als dit waar is, dan wordt de stelling in de *Periplus* dat India op dat moment geen lood produceerde weerlegd.

De loodisotopische signatuur van het zilver toont een duidelijke overlapping met ertsen afkomstig uit Groot-Brittannië. Dit onderlijnt nog eens het uitgebreide handelsnetwerk dat bestond in de periode dat ed-Dur werd bewoond.

Een erg onverwachte vaststelling was de ontdekking van drie stukjes loodglit. Deze fragmenten zijn het afvalproduct van een proces om zilver te ontrekken aan een zilver-koperlegering. De technologie van cupellatie was voorheen nog niet geattesteerd in de Golf regio tijdens 1ste eeuw v. Ch. en de eerste helft van de 2de eeuw na Ch. te dateren.

.

Naast de verwachte koperen en bronzen objecten, werden er ook twee andere legeringen vastgesteld: messing en *gunmetal*. Het feit dat een belangrijk deel van de geanalyseerde objecten van messing was gemaakt, is een eerder verrassend resultaat. Er zijn sterke aanwijzingen dat de messing van Romeinse oorsprong is, hoewel het soms is gebruikt om objecten te vervaardigen die zeker niet Romeins zijn. Hieruit kunnen een aantal interessante conclusies in verband met de circulatie van messing worden gedistilleerd.

Voor het eerst werd een relatief grote collectie localel Zuidoost Arabische munten systematisch geanalyseerd (*ca*. 8% van alle gepubliceerde exemplaren). De analyses tonen aan dat er een brede relatie is tussen de typologie en de chemische samenstelling van de verschillende types. Dit wijst op een bewuste keuze van bepaalde legeringen. Daarnaast werden er aanwijzingen gevonden die impliceren dat het zilveren oppervlak van sommige munten kunstmatig werd verrijkt. Een beperkt experimenteel luik werd opgezet om de beperkingen van een dergelijk proces te onderzoeken.

De ijzeren objecten waren te gecorrodeerd om bruikbare informatie op te leveren. De metaalslakken die werden gevonden tonen echter aan dat smeedactiviteiten hebben plaatsgevonden op ed-Dur en dat de slakken niet gerelateerd zijn aan een ander metallurgisch proces. De chemische samenstelling

van de slakken is vergelijkbaar met de beperkte gepubliceerde gegevens voor andere slakken in de Golf regio.

Sleutelwoorden:
Archeometallurgie Zuidoost Arabië, Verenigde Arabische Emiraten, Umm al-Qaiwain, ed-Dur, metallografie, SEM-EDX, chemische samenstelling, numismatiek, lokale munten, lood, loodglit, cupullering, zilver, koper, koperlegeringen, brons, messing, *gunmetal*, ijzer, metaal slakken, ICP-MS, lood istotopen analyses, sporenelementen, handel.

ACKNOWLEDGMENTS

"Illusion is the first of all pleasures."

O. Wilde

To start with a cliché: *this work would not have been possible without the help, advice and support of many others and my gratitude goes out to all of them*. Although this covers the lot, I may have to get a bit more personal for this.

Primary thanks are due to Prof. dr. E. Haerinck for giving me the opportunity to do this research and being supportive for every additional angle I wanted to add to this study. Second in line for thanks would be Prof. dr. ir. Y. Houbaert for letting me use the facilities of the *Department of Materials Science and Metallurgy* to perform the analytical part of this PhD.

At the *Department of Materials Science and Metallurgy* several people were crucial for a wide range of reasons. Dr. T. Ros Yanez and dr. A. Carmen Da Costa Reis introduced me in the mysterious world of the SEM-EDX analyses. Later on P. Mast took their place as the central point were all microscope problems could be reported. Next to this he also was an interested college. 'Master of polish' E. De Temmerman initiated me in that other mysterious world of sample preparation and more importantly giving these samples a scratch-free surface. I must admit that the disciple never surpassed the master in this case and his retirement is visible in the quality of the metallographic pictures. The Spanish crew from the Department was the best coffee partner ever, a special mentioning of Pablo, Lucia and Patricia is needed, next to Ludovic from the French connection.

My gratitude also goes to D. Tokar, whom I never met in person, for casting the experimental coins used in this PhD.

I would also like to thank drs A. De Waele (soon to be dr.) for being an excellent college and providing me with essential details on the copper-base alloys from her PhD in progress. Dr. A. Daems and T. De Schacht and E. Smekens were part in creating a nice atmosphere at the *Department of Languages and Cultures of the Near East and N-Africa.* I'm also grateful to H. De Paepe for taking care of the unavoidable administrative work that comes with working at a large institution such as Ghent University.

Some of the analyses included in this PhD were performed at different departments. I would like to thank Prof. dr. L. Moens and especially Prof. dr. F. Vanhaecke and drs D. De Muynck for the lead isotope analyses performed at the *Department of Analytical Chemistry – UGent*. Some slags were analysed by powder-XRD at the *Labratory of Soil Sciences – UGent*, headed by Prof. dr. E. Van Ranst. The analyses were performed by N. Vindevogel and for the interpretation dr. F. Mees spent several hours behind the computer, trying to figure out the meaning of the spectra. Prof. dr. M.A Elburg, *Department of Geology and Soil Sciences*, looked at pertrographical composition of two slices of slags.

Extra archaeological information was made available by the directors of the other teams that worked at ed-Dur, e.g. D.T. Potts, O. Lecomte and C.S. Phillips. The former director of the *Department of Museums and Heritages, Umm al-Qaiwain*, dr. I. Kadam Sarhan Al-Naqeeb, provided accommodation and made it possible to study the artefacts from ed-Dur in real time. Additionally it was allowed to export a considerable

amount of additional samples (e.g. the coins). Prof. dr. A. Avanzini and A. Paven provided some additional samples from the site of Khor Rori (Oman).

V. Serneels, M. van Nie, A.M. Feuerbach, C. Vlachou, L. Weeks and J. Bayley provided fruitful information by the digital medium of email. Prof. dr. Th. Rehren was very helpful upon a visit to the Archaeological Institute – University College London, as were Prof. dr. V. Piggot and X. Veldhuijzen. L. Weeks also gave me the basic lead isotope database used in this PhD and was ever willing to help.

Although I never intended to bother family, friends and acquaintance with the burdens that come with writing a PhD, I undoubtfully did. Their willing ear and enthusiasm was much appreciated.

A very special mentioning is for J. Vanmeirhaeghe who pointed out to me that the different chapters could be fused with PDF creator, omitting the step of first getting everything in one word document. This saved me blood, sweat (and who knows) tears at the very last minute.

Last but certainly not least I would like to thank my Persian princess Baharak for all the help in completing this project and her everlasting critical opinion on everything I wrote. Her support in good and bad moments was essential to finish this assignment and range from doing the dishes, till tainting the final draft of this PhD red with the correction pen.

Thanks to all, also those who are not mentioned by name.

PART I

The known

Chapter 1. GENERAL INTRODUCTION, STATUS QUAESTIONIS, TERMINOLOGY & STRUCTURE

"You may admire a girl's curves on the first introduction,
but the second meeting shows up new angles."

Mae West

• **General introduction**

The genesis of this PhD topic is found in the idea to study the metal samples collected by Ghent University during the eight successive excavations campaigns at the site of ed-Dur (Emirate of Umm al-Qaiwain, United Arab Emirates). The sample collection was very diverse and came to me in a box full of plastic bags. The assemblage consisted out of lead, silver, copper-base alloys, slag and mainly iron fragments. The research project itself is a joined project of the *Department of Languages and Cultures of the Near East and N-Africa – Research Unit Near Eastern Archaeology* (Promotor Prof. dr. E. Haerinck) and the *Department of Materials Science and Metallurgy* (Co-promotor Prof. dr. ir. Y. Houbaert). In the run up to the submission of this research project some preliminary analyses were preformed and the results looked promising enough to justify a full-time PhD research.

The results of this PhD are part of a larger program of PhD's in order to fully study the excavated material from ed-Dur and are to be published in a series of volumes that make up the final excavation report of the work done at ed-Dur by the Belgian team. Till now a volume on the glass[1] found at ed-Dur and on the tombs and their contents[2] has appeared. Recently the research on the ceramic assemblage[3] was finished and the study of the small finds[4] from ed-Dur is on its way.

The original goals for this PhD as defined in the BOF-project were:

- The determination of the alloys used and the possible identification of new ones.
- The analyses of the slag will contribute to a better understanding of the metallurgical activities/knowledge of the region and period under consideration. Possibly the slag will provide an answer on the origin of the ores used.
- Next to the archaeometallurgical study of the samples an archaeological and/or anthropological angle of view is necessary, to see the local evolution of the metal technology and to establish which object could be imported and which are of local origin.
- All this information is important to clarify the regional and interregional cultural processes at the period under consideration.

The *Department of Materials Science and Metallurgy* made the infrastructure for the research available and the basic toolset was to exist out of:

- *Optical metallography* for the study of the microstructures of the samples and slags.
- *Scanning electron microscopy* (SEM) combined with chemical analyses by *electron dispersive X-ray spectrometry* (EDX) for bulk and local chemical analyses
- *Atomic absorption spectrometry* (AAS), *spark emission spectrometry* and *wet chemistry* may be used as additional tools.

[1] Whitehouse, 1998.
[2] Haerinck, 2001.
[3] Rutten, 2006.
[4] In preparation by A. De Waele.

It soon became clear that the result generated from the available samples would not suffice to write a PhD. As mentioned above the largest part of the sample collection was made up of iron fragments. Logically this metal was going to be the main focus of the research, moreover no research has been done on iron objects from SE-Arabia. After considerable time being spent on the iron, it however also became clear that the metal was too corroded to be useful from an archaeometallurgical point and a change of course imposed itself.

The remaining sample set of a few copper-base alloy samples, lead fragments and the slags would not suffice to close the gap. Luckily the set of sample could be expanded during a visit to the Archaeological Museum of Umm al-Qaiwain, kindly made possible by the former director of the *Department of Museums and Heritages, Umm al-Qaiwain*, dr. I. Kadam Sarhan Al-Naqeeb. Additional samples were taken from objects and it was also made possible to temporally export the collection of the local SE-Arabian coins excavated by the Belgian team at ed-Dur.

New research questions were formulated and new goals were set. The 'research tools' were supplemented by *inductively coupled plasma mass spectrometry* (ICP-MS) and *powder X-ray diffraction* (XRD).

1. The main body of the research would stay as formulated in the original project, e.g. the determination of the alloys used and the identification of possible new ones. More concretely this involved the full characterisation of the expanded dataset of the copper-base alloy, lead and silver (new) samples and the slags by optical metallography and SEM-EDX. New was however that many samples now came from identifiable objects and this added the possibility to link the chemical analyses to the object class. This also made it possible to truly combine the metallurgical data with the archaeological perspective, i.e. true archaeometallurgical results could be obtained. Six additional samples were also purchased from the more or less contemporaneous site of Khor Rori (Oman) for comparative reasons. Seen in retrospect it would have been more logic that this dissertation would also have treated the typo-chronology of the copper-base alloy artefacts. This coincides much better with the analytical data presented. But the initial frame of this project was laid out in a different grid. For this reason I will use some results of drs A. De Waele on the copper-base alloy artefacts in advance of her own PhD on the small finds of ed-Dur.

2. The coins are treated somewhat as a separate group. They were also analysed by SEM-EDX for their chemical composition and this information was projected on the typology, to see if there is any link between the two. Additional question were found in the determination of the production technique of the coins. This is the first extensive collection (104 specimens) of SE-Arabian coins to be analysed in a systematic way.

3. The basic question towards the slag was to determine to which metallurgical process they belonged. The slags were characterized as mentioned above, but it was thought useful to gather information on their mineralogical composition. For that reason a selection of slags were submitted for powder-XRD analyses to determine their mineralogical composition. Linking the slag to possible ore sources proves impossible and this objective was abandoned.

4. To properly address the possible provenance of the metal artefacts an additional research track was opened. A selection of samples was submitted for lead isotope analyses (by ICP-MS) and some were also analysed for their trace elements in the frame of an undergraduate thesis.

5. The ultimate goal was and is to link all this new data and add information to a better understanding of the site of ed-Dur.

The diversity of the material made it necessary to conduct the research on such a broad. This may however mean that depth is lost at certain moments, since each of the metals is actually a separate field of speciality. I hope however that the shortcomings are minimal.

Research is often directed by the available means, being *expertise*, *analytical techniques and equipment*, *libraries* and *historical and archaeological sources*.

The archaeometallurgical expertise was not present within Ghent University and archaeometallurgy was a completely new terrain. All what is written here is the result of self-teaching/study, sometimes trial-and-error experiments and some very useful (short) discussions with specialists abroad. The lack of specialist guidance was a serious shortcoming at the onset, but was/is hopefully largely bridged during this PhD research. It is hoped that the results and analytical methods are sound and can measure up to the international standards of archaeometallurgical studies.

The analytical techniques were summed up in the original project proposition and mainly consisted out of SEM-EDX and optical metallography. It was hoped that EDX-analyses would provide information on a whole array of elements, but the semi-quantitative nature of this technique is not to be underestimated. This became particularly clear when comparative data on the same samples became available with data obtained by ICP-MS data and some test-analyses on another SEM (XL Phillips). This more recent SEM is equipped with a different software program. Although the analyses are in the same direction, the Phillips-SEM gave slightly better results. All analyses presented here were done on a Zeiss DSM 962 SEM however. This was out of practical consideration (i.e. analysis time availability for the large amount of measurements needed). I am confident that the broad alloy groups used for this PhD are reliable enough for the angle of interpretation taken. When other elements than the main alloy elements are discussed more care has to be taken and the results should not be 'over-interpreted'.

One major mistake that was made from analytical perspective is the limited amount of measurements on certified standards. I would like to add that this is due to ignorance at the onset of this study. At the very end of the PhD some additional AAS measurements were made to crosscheck the EDX results. The measurements were in acceptable accordance to the EDX data so these can be considered reliable.

As mentioned above the array of analytical techniques used, was expanded with XRD and ICP-MS analyses, both asking a special approach for archaeological material. In case of the powder-XRD, the interpretation of the spectra proved to be less straightforward than thought. The expertise in XRD analyses at the *Laboratory of Soil Science, Ghent University* (Prof. dr. E. Van Ranst) is in the field of the study of clays. The available software was not much of a help for understanding the complex silica and oxide phases present in slag.

To properly address the question of provenancing the artefacts and metals the possibility to perform lead isotope and trace elemental analyses on some of the samples was explored. In first instance renowned and specialized European Institutes were contacted, but it became immediately clear that the available budget would not allow contracting out these analyses. In a second attempt the *Laboratory of Analytical Chemistry Institute for Nuclear Sciences* was contacted (first via Prof. dr. L. Moens, who referred me to Prof. dr. F. Vanhaecke). There, well hidden from the archaeologist, Drs D. De Muynck is working on a PhD in order to refine the use of ICP-MS and by coincidence he was working on archaeological material. More specifically on the relation of lead isotopes present in the soil and in skeletal remains buried in it. The lack of knowledge on lead isotope analyses of metals was more then compensated by the enthusiasm and persistence of D. De Muynck.

For the study of the Gulf region the library of the *Department of Languages and Cultures of the Near East and N-Africa* is well equipped. The archaeometallurgical front on the other hand was poor, but due to several visits to the excellent libraries of the *Université Libre de Bruxelles* and especially the one of the *Archaeological Institute - University College London* most of this lacuna was filled. Where necessary the interlibrary book service bridged most shortcomings.

The available historical and archaeological sources of the region and period under consideration do have an impact on the information presented here. While going through this dissertation the reader will get many references to the Roman world (for technology, trade, etc.). This should not be misinterpret in the sense that ed-Dur is influenced by the Romans, even not in the light of the evident link based on the archaeological material (e.g. the glass). This is merely the result of the much larger body of archaeometallurgical studies and information on trade, available from the Roman world. To this we have to add the antique texts (especially the *Periplus Maris Erythraei*) that survived the ravage of time. This 'wealth' of information is in sharp contrast to the rather limited work on the archaeometallurgy of the Parthian Empire or at least the availability of the results. On the Indian Subcontinent archaeometallurgical work is undertaken, but is hard to track down and most of the references quoted are actually studies done by European institutes. Also the recent archaeological material from Iran and India regions is not easily accessible. Here it was tried to include as much information as possible and put forward an as complete picture as possible.

Repetition is bound to happen since I tried to let every chapter stand on its own and make it possible to read them separately. Where necessary chapters are cross-referenced.

- **Status quaestiones of archaeometallurgical research in the Gulf region**[5]

One particular publication that was published in the coarse of this PhD research was extremely informative. The book *Early Metallurgy of the Persian Gulf* (2004) by L.R Weeks, is the most recent and up to date information source of the archaeometallurgical research in the Gulf region. Although it treats the earlier periods of the Gulf (basically till the end of the Wadi Suq period, *ca*. 1700 BC), the general introduction on the geology of the region, the lead isotope research, etc. are very complete and helpful. All these items are treated in a thorough way and the list of references is extensive. This book will be referred to on many occasions in the text, and in a certain way it is used as a primary source. It would have been a possibility to recheck all the references and thicken my bibliography considerably, but I chose not to do so. This is not out of laziness but because it seems to me needless to redo the work of others, if of course this is evaluated and used in a critical way.

The earliest scientific research on copper production in the Gulf region took place already in the 1920s as a component of research into the sources of copper used by the Sumerians[6]. The research was based on the attestation of high percentages of nickel characterizing both the ores from Oman and the early copper objects from Mesopotamia. Based on that H. Peake concluded that at least part of the copper used by the Sumerians had to originate from Oman. Modern geological and archaeometallurgical research indicates that nickel occurs in many copper deposits of Western Asia, and therefore high nickel levels cannot be used to prove a definite link between early Mesopotamian copper objects and Oman ore deposits. Moreover, nickel does not occur characteristic of all the copper deposits of the Oman Peninsula, meaning that copper produced in SE-Arabia could have very low levels of nickel as well.

[5] Summery based on Weeks, 2004b: 18-33. For an extensive bibliography I would like to refer to this publication.
[6] Peake, 1928.

The first significant archaeological and scientific study of the material remains of ancient smelting operations within Oman started in the 1970s. This was triggered by the discovery of evidence for copper production from the 3rd millennium BC onwards. This had come to light by research programs conducted by the geological survey company *Prospections Limited Oman*[7], *Harvard University*[8] and the *Institute for Human Palaeontology* in Rome[9]. An additional trigger was the book by G. Bibby, *Looking for Dilmun*[10]. The archaeological and geological work carried out in Oman between 1973 and 1975 was able to demonstrate that significant copper production took place in the region in early times. Theories were proposed regarding the technologies and processes of copper smelting at various periods in the region's past, although archaeometallurgical and related analyses were extremely limited. Estimation of the periods of copper production also proved problematic, while calculations of the volume of copper production in the various periods of extraction were not possible due to chronological uncertainty. Additionally the surveys were still incomplete and lacked the detailed archaeometallurgical analyses.

In the late 1970s and early 1980s a number of archaeometallurgical studies were published by scholars from the *Centre National de la Recherche Scientifique (CNRS)*, the *Commissariat à l'Energie Atomique* and the *Laboratoire de Recherche des Musée de France* that included analyses of material from SE-Arabia. The articles represented an effort to characterize the evolution of alloying techniques in early W-Asia. They also wanted to scientifically determine the provenance of the copper used in various regions bordering the Gulf in the 4th and 3rd millennia BC. The provenance program was based on two series of compositional data: one on copper ores from various ancient mining regions in western and central Asia, the second on copper objects from Iran, Mesopotamia and SE-Arabia. They concluded amongst other things, that S-Mesopotamia and Khuzistan obtained their copper from SE-Arabian sources at least by the Early Dynastic III period and perhaps as early as the ED II period. The analytical approach of T. Berthoud[11] has been questioned on the grounds that the analyses were of accuracy and precision insufficient to allow the stated conclusions of the work. He also only used at a limited number of analyses to characterize copper produced in different areas.

The work in Oman of the *German Mining Museum* began in 1977[12]. German research in the Sultanate of Oman with a strong, though far from exclusive, emphasis upon copper production continues to this day[13]. The early survey work was particularly focused upon the importance of Oman and SE-Arabia in general as a potential location of ancient *Magan*. Over a course of several field seasons in the Sultanate of Oman, the German Mining Museum expedition was able to provide an outline of the periods of copper production, characterize the development of copper mining and extraction technology, estimate the volume of copper produced in some historic and prehistoric periods and begin to address the social and economic implications of this industry for SE-Arabia. Additionally, as the archaeology of SE-Arabia was so poorly known in the 1970s, the fieldwork of the German mission was crucial in the development of a basic chronological framework for discussion of the archaeology of the region.

The work of L. Weeks is the most recent contributions to archaeometallurgical work in the Gulf region, in addition to some recent articles.[14] His main focus is on chronological

[7] Goettler, Firth & Huston, 1976.
[8] Hastings, Humphries & Meadow, 1975.
[9] Tosi, 1975.
[10] Bibby, 1972.
[11] Berthoud, 1979.
[12] Weisgerber 1978a, 1978b, 1980a, 1980b, 1980c, 1981, 1983, 1984, 1987, 1988, 1991; Hauptmann & Weisgerber, 1980; Hauptmann, 1985, 1987; Hauptmann, Weisgerber & Bachmann, 1988.
[13] Yule 1996; Yule & Weigerber, 1996; Prange, Götze, Hauptmann & Weisgerber, 1999.
[14] Weeks, 1996; Weeks 1997; Weeks, 1999; Weeks, 2000a; Weeks, 2000b; Weeks 2003; Weeks, 2004b; Weeks, 2005; Weeks & Collerson, 2005.

evolutions of alloys, inter-site alloy differences based on lead isotope analyses and trace elements and the notorious problem of the origin of tin during the Middle Eastern Bronze Age. All this information is used to reconstruct the level of technology and trading networkes operating during the Bronze Age.

All these studies are however focused on the earlier chronological periods from the U.A.E. For the late pre-Islamic period under consideration in this PhD, only limited work has been done. L. Weeks[15] analysed 33 samples from ed-Dur and some of the metallurgical remains from the contemporaneous inland site of Mleiha were analysed[16]. In the light of this overview it is obvious that a thorough study of an extensive dataset from the late pre-Islamic period would be a welcome addition to the existing research.

- **Terminology**

Most of the used terminology is explained in the appropriate parts in the text, but some terms should already be defined at the beginning to avoid confusion.

The period that is considered here has received a diversity of names in literature, some of which are rather confusing. By many scholars the term *Hellenistic* has been used because of the presents of artefacts evidently originating from regions of Hellenistic culture or states ruled by Hellenistic monarchs. In this sense I would like to quote R. Boucharlat and M. Mouton[17]:

> *"It is significant that the most outstanding "hellenistic feature" assimilated by East-Arabian communities during the Seleucid period is the Alexander's coinage, a means of commercial exchange. These exchanges have confused the archaeological perception of the Arabian cultures; … In fact, it was just goods that circulated: the way of life, the language, the writing, the religious beliefs and rituals did not integrate any Hellenistic features. Only after they had evolved themselves and after long exchanges did the Arabian communities adopt some Hellenistic elements, in a superficial way, at the beginning of the 1st c AD."*

The more specific term *Seleuco-Parthian* has been applied again because of the presents of material clearly comparable (or even imported) to that from Mesopotamia and Iran during the reign of the Seleucid and the Parthian Empire. But by now we know that SE-Arabia was not directly ruled by any of these powers, so the region may have been influenced but had a clear own political and cultural identity. M. Mouton proposed a culturally neutral term for this period: the *Pré-Islamique Récent*, abbreviated as PIR A more elaborate description of this term will follow later, but I will use this term throughout this dissertation, because of the neutral status.

Chronological Phases SE-Arabia	Cultural periods & dating in the U.A.E.	
Bronze Age	**Hafit Period**	3100-2700 BC
	Umm al-Nar Period	2700-2000 BC
	Wadi Suq Period	2000-1700 BC
	Late Bronze Age	1700-1300 BC
Iron Age	**Iron I**	1300-1100 BC
	Iron II	1100-900 BC
	Iron III	900-300 BC
Late pre-Islamic period	**PIR A**	3rd – first half 2nd c BC
	PIR B	second half of 2nd – 1st c BC
	PIR C	1st c BC – 2nd c AD
Sasanian period	**PIR D**	3rd – (4th) c AD
	Sasanian dominance	4th – 7th c AD

Table 1: Archaeological chronology of the U.A.E.

[15] Weeks, 2004a.
[16] Final report: Ploquin, Orzechowski, & Briand, 1999.
[17] Mouton, 1999b: 14, footnote 8.

Throughout this dissertation many references to geographic regions are inclused. For the sake of clarity I will define them at the beginning:

- The Arabian Peninsula: the region bordered in the west by the Red Sea and the Gulf of Aqaba on the W-side; the Arabian Sea on the NE-side; the Gulf of Oman, Straits of Hormuz and the Gulf on the SE-side; to the north the Zagros mountains are the limit and it merges with the Syrian Desert with no clear demarcation line. Nowadays the Arabian Peninsula comprises the countries of Bahrain, Kuwait, Oman, Qatar, Saudi Arabia, the United Arab Emirates and Yemen.
- SE-Arabia: the southeastern corner of the Arabian Peninsula (U.A.E. and eastern Oman), this term will be used interchangeable with the *Oman Peninsula*
- S-Mesopotamia: Southern part of Iraq, Kuwait and the Susiana plain in southwest Iran.
- NE-Arabia: northern side of Saudi Arabia, Qatar and Bahrain.
- Indian Subcontinent: southeast Pakistan, east and southeast India and Sri Lanka
- Mediterranean: Especially the eastern part of the Mediterranean
- SE-Iran: Kerman and western Baluchistan
- S-Arabia: the south and southwestern part of the Arabian Peninsula and the largest part of Yemen.

The discussion on the terminology of the Gulf-region (Arabian *versus* Persian Gulf) is a long-standing one and both terms cause irritation. To avoid any politization of the subject, I choose to use the neutral term *Gulf-region* or the *Gulf*, and speak of the Arabian side or the Persian side when necessary.

• Structure of the PhD

This work is as much a personal quest in the, for me, new world of archaeometallurgy, and lead isotope research for that matter, as that it is the writing down of what could be learned from the metal *archaeologica* and metallurgical remains of ed-Dur. For that reason some introductions might be too exhaustive and/or detailed to the readers, maybe even a bit 'besides the question'. In my opinion a thesis is not only the presentation of results but also that of the way they were obtained.

All the chapters are built up in the same way. In the first part a general introduction on the subject is given, followed by its production techniques and a short overview of the geological setting of the region. The geological information is very basic and not meant to give an exhaustive overview. This is primarily because no indication of extractive metallurgy were found at ed-Dur. This provides the *tools* for the interpretation. A second part presents the data obtained by the analytical methods applied. Every chapter ends with a preliminary conclusion, where the tools are used to interpret the presented data.

A last point to take into account is the fact that I am writing for a very heterogeneous audience, with on the one end of the spectrum archaeologists and on the other end metallurgical engineers. Both have their own back-ground that probably has little overlap concerning this study. I thought it better to take the chance of 'over-informing' then to risk leaving the readers in the dark.

The metals and their alloys determine the general structure of this thesis. Every group is treated separately and linked to the artefact where possible. There is only one exception to this and this is the collection of coins. I thought it was better to treat this collection on its own then to split it up and treat the alloys in their appropriate chapters.

At the same time I find it important to incorporate the data obtained by archaeometallurgical analyses in a larger historical, geographical, archaeological and political frame. This is a step often omitted in this kind of research and this leads to an incomplete interpretation of the data, hence the many appendices on archaeometallurgical data. As if it is crucial to have the

research done for the sake of completeness and to show that 'also' this angle was explored. After all I am more of an archaeologist than a metallurgist and instead of seeing this as a drawback I used it in my advantage in my attempt to bridge these entirely different research fields, indeed even research mentalities.

More concrete the PhD is split up in three major parts:
Part I serves as an introduction. It gives the archaeological, historical and geographical context (*Chapter 2*) and a more detailed description of the trade during the period under consideration (*Chapter 3*). A focus is laid on the trade in metals. This can easily be omitted by the readers who are familiar with the region and the period under consideration. But it seems to me crucial to fully comprehend the position of ed-Dur and anchor any further analysis. *Chapter 4* gives an overview of the analytical methods used in this dissertation.

Part II (*Chapters 5* till *9*) presents the results of the archaeometallurgical research on the different metals conducted within the frame of this PhD research, i.e. the copper and copper-base alloys; the lead, silver, their alloys and litharge; the coins; the iron and the slag. Originally a additional chapter was to be dedicated to the typo-chronological study of the iron artefacts found at ed-Dur. At the last moment this chapter was omitted, since the study was not completed before the deadline of this dissertation. Instead it was replaced by a case study of one artefact group (the *ring-pommel daggers*) in included in *Chapter 10* to show the potential of a combined archaeological, iconographic, historical and metallurgical study. To bring all this together has to be the objective of an archaeometallurgical study. Of course this is not always possible, but it has to be the target. For this reason it is to me also justified to exclude the typo-chronological study. No metallurgical information could be added since the microstructure of the iron was completely eradicated by corrosion.

Part II can be seen as the *dataset* and includes the 'original' research done in this study. Parts I and II are brought together in Part III.

Part III is the synthesis (*Chapter 10*) of the information previously given, supplemented by the case study on the ring-pommel daggers. This chapter in combination with all the interim conclusions given in-between sum up all the conclusions of this PHD. The general conclusion in Chapter 11 serves as a more to be seen as a evaluation of the research itself. This is followed by the obligated Dutch summery of the results and, last but not least, the reference list of the works cited and (ab)used.

Part IV includes all *appendices*.

Chapter 2. HISTORICAL, GEOGRAPHICAL & ARCHAEOLOGICAL CONTEXT

"For me CONTEXT is the key - from that comes the understanding of everything."

K. Noland

2.1. Introduction

This chapter is serves as the general political and historical frame for the period of occupation at ed-Dur. In this way the context can be easily referred to in the appropriate chapters and does not need to be repeated at every step. For obvious reasons Mleiha and ed-Dur are treated in greater detail. Mleiha is discussed first, since that site provided the basic chronology for SE-Arabia.

A single site as ed-Dur, or even a small political entity with ed-Dur and Mleiha at their core, in SE-Arabia cannot be placed correctly without the necessary context. This context has to include the major political, economical and/or military powers at play at that time. Politics, economics and military actions were (and still are) always closely interwoven to each other. Maybe the disappearance of ed-Dur as an economical entity (whatever its function) is related to regional or interregional political change, which made its existence obsolete, unnecessary or simply impossible.

The political situation between 3rd c BC till 4th c AD in the surrounding empires and territories must have had its impact on the situation in SE-Arabia. The major players at work at that period were:

- The Seleucids, successors to Alexander the Great
- The city of Gerrha
- The Parthian Empire
- The kingdom of Mesene – Charax Spasinou
- The Sasanian Empire
- The Roman Empire & their policy towards the East
- The Nabataean kingdom
- The city of Palmyra
- The South Arabian kingdoms
- The kingdoms in the Indian Subcontinent

2.1.1. The Seleucids, successors to Alexander the Great

The first contact between Arabia and the Greek world was in 323 BC, the final year of the reign of Alexander the Great. Babylon was the new capital of the eastern part of his kingdom. One of his commanders, Nearchus, sailed from the river Indus to the Arabian Sea, the gulf of Oman, and then along the Persian coast of the Gulf till he reached Susa. The mission was to scout the coast, the inhabitants, its anchorages, water supplies, etc. in the region, prior to an invasion of the unknown lands of the Arabian Peninsula. To insure a better communication between Babylon and the sea, a city (Alexandria) was founded at the head of the Gulf. This city or new ones in its immediate surrounding was refounded several times. Alexander planned to conquer Arabia with a double aim:

- On the one hand Alexander wanted to complete the sea-route by exploring the section between Babylon and Egypt and so completing the sea-route.
- On the other hand it was much safer to attack Arabia from the sea then on land because of the very inhospitable environment.

Unfortunately Alexander died before any of his plans could be executed.[1] The Indo-Greek and Graeco-Bactrian culture on sites such as *Taxila (Sirkap)*, *Aï Khanoumon* and *Bégram* are the remains of the campaigns of Alexander in that region. After the dead of Alexander his empire was divided amongst his generals and by far the larges territory came under the rule of Seleucos. Originally the Seleucid Empire comprised C-Anatolia, the Levant, Mesopotamia, Persia, Turkmenistan, Pamir and the Indus Valley, but even under the first ruler the territory

[1] al-Saud, 1997: 43.

started crumbling because of internal and external stress. A major part of the lands was lost when the Parthians seized control over the former Persian realm.

With the conquering of his world empire, Alexander the Great brought the Gulf back into the picture as a main trading corridor between East and West. From the end of the 4th c BC onwards, the Seleucid kings showed much interest in the Gulf. Alexander's foundation of Alexandria at the Shatt el-Arab and its renewal by Antiochus IV probably reflected the Greek desire to establish a new maritime centre at the head of the Gulf. This settlement would function both as the capital of the Empire and as port off-call for long-distance trading vessels and the navy. They tried to control this waterway as well as possible and to protect the trade activities and therefore a few fortresses were built at crucial points in the Gulf (e.g. Failaka). The Seleucids did however never dominate the Gulf politically and the trade was mainly under control of Arab middlemen (e.g. Gerrha), who took the goods by caravan across the desert of the Arabian Peninsula to the Levantine coast or via the Euphrates and the northern trans-Syrian land route.[2]

The island of *Failaka* at the head of the Gulf was known as *Ikaros* to the Greeks. The island was strategically situated for seafaring activity in the region and ceramics indicate connections with Babylon, Bahrain, E-Arabia and the S-Gulf coast. Archaeological excavations unearthed a fortified enclosure containing two temples and a number of rubble houses, a "terracotta workshop" and a small shrine dedicated to Artemis. The site was founded by the first Seleucid king, Seleucos I (312-281 BC), or one of his successors with the eye on controlling the trade traffic in the Gulf. Its function would have been military (as well as religious) although a lack of weapons could point towards a development into a civil settlement. Around the middle of the 3rd c BC, the local power would have become stronger at the cost of the Greek influence. Antiochus III seems to have reinstalled Seleucid domination on Failaka. The fortress was rebuilt and strengthened and more Seleucid coins came in circulation. The main occupation is to be situated between the beginning of the 3rd c BC till the middle of the 2nd c BC. With the end of the Seleucid Empire, Failaka was largely abandoned. A reduced occupation during the Parthian period is attested however.[3]

The decline and abandonment of the Seleucid settlement on Failaka around the middle of the 2nd c BC appears as a noticeable time-mark. At that time the political situation dramatically changed in the Middle East. The Seleucid authority over Babylonia and the Gulf was challenged and then destroyed by the Parthians who progressively took over the area. More precisely the client kingdom of Characene situated in the same region as the original city founded by Alexander the Great became to dominate the Gulf maritime trade. They would keep their influence on the "Characenean Corridor" at least up to the end of the 2nd c AD.[4]

2.1.2. The city of Gerrha

The east of the Arabian Peninsula witnessed a great increase in settlement during the Seleucid period. From the end of the 4th c BC onwards the trade relations along the coast of the E-Arabian Peninsula developed and without question the single most important trading entity in the Gulf region was the wealth ancient city of Gerrha. It is mentioned by classical writers such as Polybius, Strabo, Pliny and it appeared on Ptolomy's map of Arabia, still the origins of the metropolis and its location have been topics of debate for many years. Its merchants, dealers in frankincense and myrrh among other luxuries, served as middlemen in the network between the S-Arabians, Indians, Nabataeans, Seleucids and Ptolemies.[5] It was

[2] Salles, 1987: 75; Potts, 1990b: 15; Salles, 1993: 493-494; Salles, 1996a: 260-262.
[3] Ray, 2003: 174; Callot, 1990: 230; Potts, 1990b: 178 & 195-196.
[4] Salles, 1993: 494.
[5] Groom, 1982: 97; Potts, 1984: 105; Potts, 1990b: 85-87; Ray, 2003: 176.

only by paying a huge tribute of silver, frankincense and myrrh to the Seleucid king Antiochus III in 205 BC that they were able to maintain their independence. His objective was probably to extract tribute from the fabulously wealthy city. But perhaps also to gain more control over the Indian and Arabian trade and to divert it from its hereditary enemy, Egypt, which had profited greatly by contact with Gerrha through her Nabataean vassals.[6]

R.G. Hoyland notices that at some point there was a shift in the trajectory of Gerrha's foreign trade, before Gerrha shipped merchandise to Babylonia by sea, but in the coarse of the 3rd c BC Gerrha began to export its goods by land. This probably meant towards Egypt and Syria, and certainly we hear of Gerrhaean incense in the Mediterranean world at that time. In the 2nd c BC they seem to have begun trading with the Nabataeans, presumably they were exporting Indian goods to Petra, since S-Arabian goods would have been passed on along the W-Arabian route or via the Red Sea. Later on in the 1st c BC and 1st c AD, the Gerrhaeans supplied the Parthian Empire.[7]

Anyhow, from the 1st c BC and 1st c AD onwards, the importance of Gerrha diminished for several reasons. With the rise of the Parthian Empire, the commercial policy concerning the Gulf changed and the land route towards the East developed. Because of a better knowledge of the monsoon winds the ships could have sailed straight to the top of the Gulf where Spasinou Charax was situated, omitting a stop at Gerrha. Moreover, the Romans started trading 'directly' with the Indians via the Red Sea. Consequently, the city of Gerrha lost its important role and disappeared from the international scene by the 2nd c AD.[8]

All in all it is not inconceivable that Thaj, although it is situated inland, is to be identified with Gerrha. From the accounts of Strabo it would seem that there was both a port and an inland town of Gerrha. A bi-polar organisation with an entity in the inland and a more distant harbour at the sea is also known from other examples in history.[9]

2.1.3. The Parthian Empire[10]

The history of the Parthian Empire is somewhat of a problem, since it is primary based on sources from outside the Parthian world (e.g. Roman). The Parthians established an autonomous kingdom somewhere around 238 BC, headed by the first Parthian ruler Arsaces, hence the Parthians are also called the *Arsacids*. They became independent under the rule of the Seleucid ling Seleucos II, who was unable to repress this revolt in the east of his empire. Shortly before also the satrap of Bactria had pronounced its independence. The struggle for independence and for the control of power over the satrapy of Parthia might actually have started earlier, in 247 BC when the governor Andragoras achieved a large amount of autonomy in his province. Arsaces and the Parni might have subsequently overthrown him. The Seleucid kings did not stand down as easily and several wars were fought and it was only under Artebanus I peace was made with the Seleucid king Antiochus III. The Parthians formally accepted a feudal status but the rapid withdrawal of the Seleucid forces in 189 BC, left the new client kingdom in a semi-independent state.

The real expansion of the Pathians began under Mithradates I about 171 BC, and he planted the seeds of the world-empire Parthia was to become. The power of Mithradates I could grow because of the weak control that was exerted by the Seleucid kings at that time, who were caught-up in internal struggles for power. In the final months of Mithradates' reign (138-137 BC) his empire reached its largest expanse, but a source of trouble arose at the eastern

[6] Nodelman, 1960: 83-87.
[7] Hoyland, 2001: 24-26.
[8] Hoyland, 2001: 26.
[9] Hoyland, 2001: 24.
[10] The summery of the Parthian history is largely taken from Bivar, 1983: 21-99, unless stated otherwise.

frontier in the form of the tribal confederation of the *Yüeh-chih* (the later *Kushans*). The central Asian nomads moved westwards as part a larger westward migration gulf. The successor of Mithradates I, Phraates II (138-128 BC), was also faced with a renewed attempt of the Seleucid king Antiochus VII to regain control over the eastern part of the former Seleucid kingdom. In a first phase the Seleucid military campaigns were successful. In 129 BC the luck turned and the main Seleucid army was defeated and Antiochus IV was killed. The celebration of this victory was quickly disturbed by trouble with the *Sakas* (pushed forward by the advancing Yüeh-chih) at the eastern frontiers and Phraates II was killed in an attempt to repress the Sakas. It was only under Mithradates II (124-87 BC) that the eastern frontier was secured again. He also stabilised the Parthian administration and made Babylonia (with the cities of Seleucia and Cteisphon) the heart of the kingdom.

During the reign of Phraates III (*ca.* 70-57 BC) the Parthians are for the first time confronted with the expansion military power of the Roman. As a consequence Parthia appears for the first time in Roman historical treatises. At first the Parthians maintained their neutrality but a military clash was inevitable. This soon followed with the destruction of the Roman army under Crasses at Carrhae in 53 BC and the seizure of the Roman legionary standards. The boarder between the two empires was fixed at the Euphrates and several Parthian intrusions in the Roman East followed. In order to retrieve the standards a large, but unsuccessful, military campaign was set up, headed by Marcus Antonius and also Armenia was lost to the Parthians. The ruling Parthian king, Phraates IV, was victorious but faced internal political problems and was exiled for a period of time. After negotiations with Octavianus (the later Augustus) the Roman standards and prisoners of war from the battle at Carrhae were returned to Rome in 20 BC. This was the beginning of the *Pax Romana*. After the dead of Phraates IV a long period of dynastic conflict in the Parhian Empire followed.

The next conflict between Parthia and Rome was occurred between Vologeses I and Nero, again the bone of contention was the control over Armenia. A stalemate was eventually achieved with an agreement that both empires would have to give their consent on the ruler of Armenia and peace was re-established in 64 AD. But as during the previous period of peace, internal political struggles weakened the Parthian rule. The rise to power of Osroes I and his unilateral appointment of a new king in Armenia provoked the anger of the Romans. The Roman Emperor Trajan set out to punish the Pathians and headed a military campaign first to Armenia (114 AD) and then into the Parthian hearth land. This latter campaign ended with the sacking of the Parthian capital of Cteisphon (116 AD). Dura Europos became under Roman control for the first time. During that time two Parthian rulers were struggling for power: Osroes I, controlling Babylonia, and Vologeses II residing in Iran. Such a division in the control over the Parthian territories was becoming increasingly usual during the 2nd c AD and contributed to the weakening of Parthian defence against external invaders. The territories won by Trajan were however returned to the Parthians by his successor Hadrian.

Only with the accession of Marcus Aurelius in 161 AD the threat of hostilities returned. The new king Vologeses IV launched an offensive and poured across the Euphrates into Syria, after retaking Edessa. A counterattack by the Romans in 165 AD under Lucius Verus stabilised Armenia, retook Dura Europos and Cteisphon was burned. The oasis kingdom of Palmyra, a Roman colony since the time of Hadrian, contributed to the stability on the southern flanks by acting as policeman of the Syrian Desert. In 191 AD Vologeses IV was replaced by a rival, Vologeses V and wars were fought with the Roman troops of Septimus Severus. In 197 AD Cteisphon was again captured by the Romans and Septimus Severus created the new province of Osrhoene. The new network of roads allowed e.g. Nisibis to be more firmly integrated into Rome's eastern defences, giving her valuable access to the Tigris and a choice of invasion routes.[11]

[11] Dodgeon & Lieu, 1991: 1.

A final act of aggression towards the Parthian was by Emperor Caracalla towards Artabanus V. Caracalla is said to have attacked the unsuspecting Parthians during a state visit. The Roman army ravaged a large part of Media. After the dead of Caracalla the Parthians invaded Mesopotamia and defeated the Romans near Nisibis. In the end the Parthian dynasty was not ended by the external struggles with the Romans but by the new Iranian dynasty of the Sasanians. The Sasanian Ardashir I defeated and killed the last Parthian king Artabanus V in 224 AD.

On the position of the Parthians in (international) trade the sources are even scantier. Although there is no record of Parthian attacks on Arabia or direct political or commercial influence, the Parthian sphere of influence clearly included both Gulf coasts. By 100 AD the scene had altered considerably from the days when Gerrha was the region's principal middleman. The 'Parthian' merchants, sailing out of Charax, learned to navigate the monsoon winds, making coastal port-hopping unnecessary. Trade now proceeded directly down and up the Gulf to points in S-Arabia, India and Sri Lanka. The Parthians controlled the Tigris-Gulf water connection and the overland routes through N-Iran. Roman traders were forced to use the route via the Red Sea. This transfer to a northern or western axis of trade left Nabataean Petra deserted, while Palmyra became the major distribution point by the 2nd c AD.[12]

The pivot of the peculiar alliance between self-governing cities and the central Parthian authority may however well have been the international trade networks. Trade brought fantastically large revenues both to the cities and to the Parthian ruling house. The fate of the caravans that set out from the harbours of the Gulf for Syria, or which proceeded through Ecbatana to Palmyra, depended largely on the central government, which insured their safety. Besides this the central government dictated the routes to be taken, fixed uniform tariffs, etc. Of course this 'alliance' depended entirely on the strength of the Parthian state and the effectiveness of its influence on the trade routes. When any crisis arose in Parthian Iran, or its rulers lost their footing in the Mesopotamian area, the trading cities sharply changed their orientation.[13]

The principal commodities for Parthian Iran were the Chinese raw silk trade from the 2nd c BC onward and also Indian goods, such as precious stones, perfumes, opium, eunuch slaves and spices. Caravans from Mesopotamia delivered Syrian glass, silk fabrics, woollen fabrics dyed in purple, metal articles, wine and butter to the eastern regions of the Parthian realm, from where they were carried of by local merchants to China and India.[14]

2.1.4. The kingdom of Mesene – Charax Spasinou

A city was founded at the confluence of the Tigris and the Euphrates (the Shatt el-Arab) by Alexander the Great known as *Alexandria-on-the-Tigris*. After damage by floods Antiochus IV restored the city and renamed it as *Antioch*. Later the city became the capital of the "Satrapy of the Erythraean Sea", probably created by Seleucos II or III. The local governor Hyspaosines, who gave it the name of *Spasinou Charax*, refounded it a second time in 127 BC. With turning success the city/kingdom retained a large amount of independence till the 3rd c AD. There were three other Characenean cities of importance in the Mesene territory, which is a strictly geographical term, being: Forat, Apologos and Apamea.[15] Spasinou Charax was connected by road with the 'silk-routes' at Seleucia, by river and road with Palmyra and Syria and by desert-route across N-Arabia with Petra.[16] There were also sea

[12] Potts, Mughannum, Frye, & Sanders, 1978: 10-12; Potts, 1990b: 228.
[13] Lukonin, 1983: 722.
[14] Lukonin, 1983: 739.
[15] Potts, 1988: 140; Bin Seray, 1996: 16.
[16] Warmington, 1974: 30; Potts, 1988: 138.

routes linking the port of Apologos with Qana' in S-Arabia and with the NW-Indian port of Barbarikon.[17]

Little is known of the fate of Alexandria-on-the-Tigris under the early Seleucids. Different views have been expressed on the amount of attention that they gave the Gulf region in the interest of commercial exploitation. During the internal twists in the Seleucid Empire, the local governor Hyspaosines found himself in the position of ruling a virtual independence city-state, Spasinou Charax ("the fort of Hyspaosines", from now on referred to as Charax). He was in the position to profit immensely from the trade with the East during the second quarter of the 2nd c BC. The prosperity of Seleucia also indicates that there was no significant interruption of traffic between the city and the Gulf. This may infer that Hyspaosines made no open rebellion against whatever Seleucid controlled. In 141 BC the Parthian king Mithradates I moved on Mesopotamia and defeated the Seleucid king Demetrius II. In 121/120 BC the whole of Mesene was overrun by the Parthians and even Charax must have fallen. Despite the absoluteness of his defeat, Hyspaosines was permitted to retain the little kingdom of Mesene as a vassal of Parthia. During the anarchy in Syria in the early 1st c BC and the constant upheaval in Parthia, the Tigris/Euphrates-route was unsafe and alternatives emerged. The first was across the desert to Petra, a route organised and dominated by the Nabataeans. The second was the sea route around the Arabian Peninsula through the Red Sea. More trouble came with the continuous wars between Parthia and Rome from 54 to 33 BC, where the Characenean policy showed a more pro-Roman attitude. By the early 2nd c AD, following Trajan's retreat from Babylonia in 116 AD, the kingdom of Characene, which had joined the side of the Roman emperor, lost its autonomy and was taken over by the Parthians. Sometime prior to 131 AD a Parthian prince, Meredates, was put on the throne of Characene.[18]

Next to the economical influence Characene had on the Golf, it is clear that they also had political influence in the N-Gulf and especially on Bahrain. King Meredates appointed a governor of Palmyrene origin, Yarhai, in 131 AD on the island of Bahrain. Meredates also minted coins in 142 AD with the title "King of the Omani" and depicts himself with the double crown, a symbol normally reserved only for the Parthian King. D.T. Potts suggests that Characenean authority was not only practised on Bahrain but also by the annexation of the *emporium* Omana and/or even part of SE-Arabia. Meredates was defeated and banned from Characene by Volgases III in 150/151 AD.[19] It has been suggested that this is actually the consequence of Characene being a Rome client state for some time during the 2nd c AD. This would also explain the influence of the Palmyrenes, who were also a Roman client state. The involvement of the Palmyrenes in the trade organised from Charax is important to stress. Vologeses' attack can thus be seen the removal of a source of Roman influence within his own realm. The most likely time for the beginning of the Roman influence would be during Trajan's Parthian campaign (114-117 AD), since Trajan was received hospitably at Charax. Nevertheless the caravan trade recommenced (156 AD) after the Parthians took control, but it is striking that whenever the war broke out between the 2 powers, there are no indications of caravans coming and going.[20] The eventual collapse of the Kingdom of Charcene occurred soon after the Sasanians king Ardashir I rose to power in the early 3rd c AD.[21]

The location of Charax was ideal to take over the trade once controlled by the city of Gerrha. It is thought that Gerrha supplied the caravan routes to the Mediterranean during the 1st c BC and possibly also still in the 1st c AD. The shift in these activities to other centres is linked to the expansion of the sea trade and the growth of Palmyra at the end of the 1st c BC.

[17] Nodelman, 1960: 102; Bin Seray, 1996: 18-19
[18] Nodelman, 1960: 87-97; Bivar, 1983: 40; Potts, 1996: 279.
[19] Potts, 1990b: 145-146; Rutten, 2006: 402-403.
[20] Young, 2001: 146-147.
[21] Potts, 1997c: 103.

Characenean ceramics are found in considerable amounts on Failaka, Bahrain and ed-Dur. Bahrain played an important role as a place to bunker water and food, and as a trading market in the Characenean commercial network. The large amounts of Characenean pottery on ed-Dur may point to a similar function.[22]

2.1.5. The Sasanian Empire[23]

Around 205 AD the local king Papak revolted and probably united much of the province of Persis (Fars). During this period his Parthian overlord Vologeses IV was occupied with the Romans under Septimus Severus invading Mesopotamia. The true rise of the Sasanians came under Ardashir. He became king probably about 216 AD and began to expand his realm into Kirman in the east and Elymais to the west. The overthrow of the Parthians seems to have been the result of a coalition headed by Ardashir. In 224 AD Ardashir had overthrown the last Parhian king Artabanus. The Sasanian dynasty introduced a new phase of conflict between Rome and the Persian Empire. Ardashir found that his relations with Rome were dictated, in the first instance, by the need to secure the safety of his regime. In the later 220s pressure on the Roman frontier became acute. The first Persian attack appears to have come in 230, and the Roman legions in N-Mesopotamia seem to have been incapable of handling the crisis.[24] Ardashir besieged Nisibis in 230 AD and his forces raided Syria and elsewhere in the Roman east. Septimus Severus marched against Ardashir in 232 AD and claimed victory. But Septimus Severus' murder in 235 AD and the subsequent troubles in the Roman Empire may have induced Ardashir to attack again. The dates are uncertain, but towards the end of his reign, probably in 238 AD, he took Carrhae and Nisibis. Also Hatra was taken and this might have provoked a Roman counterattack.

Because of internal trouble and the rapid change of Roman emperors, it was not until 243 AD that the Roman emperor Gordian III advanced against Shapur I. At first the Romans were victorious, but in a second battle, in 244 AD, the Romans were defeated and Gordian III died and Philippus Arabs was appointed the new emperor. Philippus bought peace with the Sasanians. In the 250s AD new trouble arose after a possible Roman interference in Armenia. Again the Sasanians were victorious, among others an army of 60.000 legionaries was destroyed at Barbalissus, Syria was overrun, Antioch sacked and Dura Europos retaken. As a final triumph at the end of the 250s the Roman Emperor Valerius was captured in person, an event commemorated in the rock-carvings of Naqsh-I Rustam and Bishapur. Shapur's forces again ravaged Syria and also invaded Cappadocia. Eventually they were made to retreat to their homeland because of the attacks of Odenathus, ruler of Palmyra, in 262 AD. Shapur I saw himself as the heir to Cyrus the Great: he re-established much of the ancient borders of the Persian Empire, reformed the administration, encouraged religious tolerance, but at the same time reaffirmed Zoroastriasm as the state religion.

At the onset of his reign Bahram II faced a Roman invasion under emperor Carus in 283 AD. The Romans captured Ctesiphon and would have extended their conquest if the emperor had not died the same year. Peace was made, and this permitted the Romans to regain the province of Mesopotamia. The reason for the acceptance by Bahram II of such enormous terms was possibly a revolt in the eastern provinces, which he was able to put down. The strong ruler was Narseh. He set out to regain the Sasanian territories lost by Bahram II. He defeated the Roman army under co-emperor Galerius in 297 AD and Mesopotamia was recovered. However Narseh was taken by surprise the following year and the Romans scored a major victory. They forced Narseh to make major territorial concessions. Diocletian made peace at the request of Narseh whereby the Romans not only regained suzerainty

[22] Rutten, 2006: 401-402.
[23] The summery of the Sasanian history is largely taken from Frye, 1983: 116-177, Dodgeon & Lieu, 1991, Pollard, 2000 and Ball, 2000, unless stated otherwise.
[24] Potter, 2004: 165-166.

over N-Mesopotamia and Armenia but obtained additional land. Furthermore, trade between the two empires was to be channelled through Nisibis as the sole place of exchange, at the request of the Romans. After this defeat the Persians and the Romans remained at peace for forty years.

The Roman Emperor Constantius II waged a war against Shapur II but neither side came out victorious and by 350 Constantius returned to the west. Shapur II resumed hostilities once more in 359. In 361 the Roman Emperor Julian assembled a huge army and proceeded down the Euphrates. But they encountered a flooded land to slow them down and once on the other side of the river the Sasanians used the tactics of the scorched earth to starve the Roman army, all the time attacking them in guerrilla style. In one of these encounters Julian died and the army was forced to retreat. The new emperor was forced to sign a thirty-year peace treaty and give up a lot of the Roman Eastern possessions.

This is where the historical overview ends, since by the end of the 4th c AD ed-Dur seems to have been completely deserted. The information on the Arabian side of the Gulf is much more extensive for the Sasanian period till the rising of Islam.

The Sasanians wanted military and political control of the Gulf, what also meant that domination of the commercial sea-routes. There is reason to believe that the Sasanians wanted to regain the mercantile supremacy in the region and beyond at the expense of the Romans. The first attempt was already made by Ardashir I (224-241 AD). After the conquest of the central districts of Persis he would have crossed the Gulf from Iran in 240 AD and attacked E-Arabia and killed the local king of Bahrain (Sanatruq). The fact that this was a Parthian name (often used in Parthian royal families) could point to the presence of a Parthian ruler in Arabia and could explain why Ardashir I invaded the east of the Peninsula, which formed no threat to the Sasanian empire.[25] Early in his career Ardashir I also seems to have fought with the "natives of Mazun", the name given to Maka (Oman) in Middle Iranian, Syriac, Armenian and Arabic sources. [26] Sasanian outpost such as Sohar, the fort at Siraf and Jazirat al-Ghanam, were installed in the after match of Ardashir's campaign in Arabia. The pearling industry flourished and the Gulf trade was stimulated by the Sasanians[27]. The dearth of Sasanian archaeological material in the area between modern Qatar and Kuwait can be explained by the fact that the Sasanians mainly exerted their dominance through local rulers and clients.[28]

The great Ka'aba of Zoroaster inscription at Naqsh-I Rustam near Persepolis lists Mazun as the 27th land in the empire of Shapur I (241-272). Archaeologically however there is little solid evidence of Sasanian presence in the form of Sasanian pottery and coins in the U.A.E., and by the end of the 3rd c AD the control over the province may already have been lost.[29]

Early in the 4th c AD Shapur II (309-379 AD) invaded Arabia in retaliation for Arab raids on the Persian coast. He landed somewhere in the Eastern Province around 325 AD before sweeping across the whole peninsula. How devastating the disruption was, is unclear. But several towns apparently continued to serve as minor ports and security outposts for the Sasanians to retain control over the coast. From all Sasanian coins found in E-Arabia the largest number is attributed to Shapur II. This can point to the fact that after the campaign the Sasanian more actively dominated the region. It is only in the 6th c AD that a more direct control from the centre of the Sasanian Empire, through the appointment of governors, can

[25] Potts, Mughannum, Frye, & Sanders, 1978: 10; Piacentini, 1985: 60-61; Potts, 1996: 281; al-Saud, 1997: 54; Hoyland, 2001: 28.
[26] Kennet, 2005: 109.
[27] Hellyer, 1998: 114; Ray, 2003: 200.
[28] Kennet, 2005: 108.
[29] Hellyer, 1998: 114; Kennet, 2005: 109.

be noticed.[30] In the SE of the peninsula, most of the interior mountain areas and the E-Coast overlooking the Gulf of Oman were governed by the Al Julanda dynasty of Oman. There was however a Sasanian governor at Oman's inland city of Rostaq, while the less hospitable desert areas are likely to have been effectively autonomous under their own tribal leaders.[31]

By the end of the 6th c AD the Sasanians had manoeuvred themselves into a position which enabled them to dominate maritime trade in the Gulf and the Arabian Sea. The region of Oman had considerable strategic and commercial importance to the Sasanians. Its location at the entrance of the Gulf made it an important centre from which Persian activities could be directed throughout the Indian Ocean and S-Arabia. Therefore they controlled the major centres of maritime trade in Oman, the most important of them being Sohar and Dibba. These two cities also acted as the main military and administrative bases for Sasanian operations in and around Oman. The inhabitants of the coastal areas of Oman engaged in wide-scale trade and fishing activities, as well as serving as sailors in the Sasanian navy.[32]

The Sasanians did not only try to control the sea-routes, but also the land-routes (i.e. the 'Silk Road' from China to the West) that past over their vast territory. Caravans were directed to official crossing-points and the duty collected on goods in transit contributed to the wealth of Sasanian rulers. In the first phase the control over the sea-trade in the Gulf was sufficient but quickly the commercial interests expanded to India, Sri Lanka and perhaps even Malaysia.[33] By the 5th and 6th c AD Iran probably was the major maritime power in the western Indian Ocean.[34]

2.1.6. The Roman Empire & their policy towards the East

This part on the Roman Empire will only include the policy towards the East and the importance of Roman Egypt towards the Roman-Indian trade. The Romans, as always, took a gradual path in annexing territories in the East. Pompey added Syria to the Empire in 64 BC. The map draw of the region in this way was a patchwork of large cities with their hinterland, client kingdoms, etc. Some of these entities preserved their autonomy till the end of the 1st c AD.[35]

During the civil war at the end of the Triumviral period major kings such as in *Commagene*, Judea (where Herod was formally recognised as king in 40 BC) and in *Nabataea*, gained considerable power. The battle of Carrhae in 54 BC with the Parthians crushed the Roman forces in the East. It was only in 40 BC that Octavian, the later Augustus, finally got the lost standard back and the surviving prisoners were returned[36]. Marcus Antonius engaged in battle, but had to withdraw in 39 BC and control was only restored in 37 BC. This changed after the victory at Actium (31 BC) by Octavian and the Roman civil war came to an end. One of the most crucial steps in the strategic shaping of the Empire in the Near East seems almost certainly to have been taken as an immediate consequence of the sea battle of Actium. Seleucia on the Euphrates (later Zeugma – "the Bridge", since 64 BC part of the kingdom of Commagene) appears to have been placed under direct Roman control.[37] The Romans finally took complete control of the Eastern Mediterranean region after 31 BC with the surrender of Ptolemaic Egypt. At that time they acquired territories in which the long-distance trade via the Red Sea with the East was already well established.[38]

[30] Potts, Mughannum, Frye, & Sanders, 1978: 12; al-Saud, 1997: 54.
[31] Hellyer, 1998: 114.
[32] al-Naboodah, 1992:81-82.
[33] Whitehouse, 1996: 339.
[34] Warmington, 1974: 137; Ball, 2000: 133.
[35] Gawlikowski, 1997: 40.
[36] Bivar, 1983: 66-67.
[37] Millar, 1993: 27-29.
[38] Avanzini, 2002: 19.

Augustus undertook a full-scale invasion of Arabia in 26 or 25 BC and either then or latter the port of Eudaimon Arabia was successfully attacked. The Roman Empire was most probably not directly involved in the trade with the East but profited in an indirect way by asking custom duties for the imported goods. As a consequence it was in their interest that the routes stayed as safe as possible.[39]

Peace was established at a great ceremony on an island in the Euphrates in 20 BC, between Augustus and the Parthian ruler Phraates IV. Over half a century of war was officially ended and the *Pax Romana* took shape. After 54 years and tens of thousands of lives after the battle of Carrhae, the frontier between the Roman and Parthian empires reverted back to where it had been in the first place: the Euphrates. After the Augustan peace, Roman borders in the Near East remained more or less static for over a century as Rome and Persia enjoyed a period of stable, even friendly, relations.[40] During the peaceful and secure rule of Augustus the demand for 'exotic' products boomed and the Red Sea became the royal route, omitting the Gulf route. But how tempting this might seem this is an oversimplification and a complete shift of the trade patterns from the Gulf to the Red Sea cannot be seriously argued.[41]

After this period there was a shift from the mixture of client kingdoms and organized provinces, which characterized the republican and Augustan east, to the Flavian situation. The new policy annexed most of the frontier areas to produce a zone of directly controlled provinces. There was renewal of the conflict between Rome and the Pathians over the alignment of Armenia, including a threat to Syria and the deployment of Roman forces east of the Euphrates in 64 AD. This war was resolved in Rome's favour in 66 AD. Most changes came in the Flavian period. Commagene was permanently annexed in 72 AD. Client kingdoms in S-Syria gradually came under direct control, and Palmyra became part of the Roman province by this time, if not before. By the time of the appointment of Trajan as emperor in 96 AD, the eastern frontier consisted of a string of directly controlled provinces (Cappadocia, Syria, Judaea and Egypt) bordered by the Armenian kingdom, Parthia and the Nabataean kingdom.[42]

The military operation undertaken by Trajan in the East must have had a severe impact on the region. The annexation of the Nabataean kingdom in 106 AD was perhaps made inevitable after the annexation of Palestine. The secret of Palestine's security lay not in controlling the coasts, Rome's initial reason for entering Palestine, but in the control of its hinterland beyond the Jordan. Control of the lands beyond the Jordan also had a powerful economic as well as strategic attraction. Beyond laid the immensely wealthy Nabataean state with all the trans-Arabian trade routes.[43] The annexation of Nabataea therefore has to be seen in the light of the creation of a secure boarder combined with commercial motives[44].

The main reason for Trajan to cross the Euphrates was the need to preserve a Roman friendly client kingdom in Armenia. The replacement by the Parthians of the Armenian king for one that was more sympathetic to brought Trajan eastwards in 114-115 AD, resulting in the annexation of Armenia, Mesopotamia and Assyria. He marched down the Euphrates and captured Ctesiphon the Parthian capital in 115-116 AD, profiting from the weak Parthian rule at the time. He placed a puppet Parthian king on the throne and continued down the Euphrates to the shores of the Gulf to Mesene. Although Trajan's advance into Iranian territory was one of the most successful campaigns by an European in the East since Alexander, it achieved little in the short term. His eastern conquests, apart from Arabia, were

[39] Casson, 1989: 37-38.
[40] Ball, 2000: 15.
[41] Salles, 1993: 495-496.
[42] Pollard, 2000: 16-17.
[43] Pollard, 2000: 17; Ball, 2000: 15.
[44] Treister, 1990: 34.

given back by his successor Hadrian in the face of instability elsewhere. On the long term a major shift in ideas came. After Trajan Rome looked increasingly to the East as the only place where real expansion still lay and there were more frequent incursions across the Euphrates, and even the establishment of short lived Roman provinces there.[45]

There was another war with the Parthian in 161-165 AD after the appointment of Marcus Aurelius, again caused by the Roman refusing to accept a pro-Iranian king on the Armenian throne. Seleucia on the Tigris and Ctesiphon were destroyed again. This time Marcus Aurelius' co-emperor, Lucius Verus, took charge of the East, basing himself at Antioch for four years. For the first time Rome had an emperor for the East, ruling from an eastern capital. The Roman victories against Iran must be questioned somewhat as exaggerated. After all they appear to have had very little effect on the real Iranian power and they resulted in few permanent gains for the Romans.[46]

Septimus Severus (193 – 211 AD) spent the first 5 years of his reign in civil war and the consolidation of power. The rest was spent in aggressive expansion, apparently in an attempt to improve the Empire's strategic position on several major frontiers.[47] Severus made his way to the East and attacking the fortress city of Nisibis by the Euphrates. The excuse being that the Parthians had taken advantage of the distraction in the West to attack the city of Nisibis. The Parthian king had probably saw the increased Roman presence at Nisibis as an violation upon his traditional territory. Severus, on the other hand, may have seen the conflict as an opportunity to win easy glory by defeating a ruler weakened by internal political struggle. In 197 Severus launched his invasion, he failed to capture Hatra, but did capture and plunder Ctesiphon. The Romans took the Parthian king completely by surprise, implying perhaps that there was a truce at the moment. The military operations were completed by 199 AD and Rome had arrived on the banks of the Tigris to stay. Septimus Severus established his new conquests as the Roman province of Mesopotamia with the capital of Nisibis and he divided the old large province of Syria into two.[48]

In 216 AD Caracalla advanced across Mesopotamia and into the Parthian heartland. In 217 he reached the city of Edessa at the head of a large army. Before he could however move into battle, Caracalla was murdered in 217. When the Parthian king Artabanus learned of Caracalla's death, he gathered his army for an invasion of Roman Mesopotamia. The armies met outside of Nisibis in the autumn of 217, and the Romans were defeated. The new emperor Macrinus bought peace for an enormous amount of money.[49]

The reign of Marcus Aurelius marks the turning point in the general well being of the Roman world, and from this time we see the decline and the breaking up of the Western Empire, often described as the "crisis of the third century". From the 2nd half of the 2nd c AD onwards there is a decline in the trade via the sea route and a general economic decline in the Roman Empire in the 3rd c AD due to political and military problems. The Roman emperors were many and most of them died a violent death. At the same time and for the same reasons Palmyra reached an extraordinary degree of prosperity. The absence of an effective imperial force in the East since 260 AD may have contributed to the feeling at Palmyra that they had the leadership of the East. Just as other parts of the Empire such as Britain and Gaul were tearing off at this time due to perceived failure of the central administration to protect them.[50]

A sharply contrasted picture emerges after the mid-3rd c AD. The threats in the East increased with the Sasanian overtake of Parthian rule. The Sasanians with their more

[45] Parker, 1986: 123; Pollard, 2000: 17; Ball, 2000: 17.
[46] Warmington, 1974: 104; Pollard, 2000: 17-18; Ball, 2000: 17.
[47] Parker, 1986: 130-131.
[48] Young, 2001: 173; Potter, 2004: 110-115.
[49] Potter, 2004: 144.
[50] Warmington, 1974: Young, 2001: 181; 136; Rutten, 2006: 405.

centralized government and professional army with sophisticated siege capacity, were a more serious danger to the eastern frontiers than the Parthians had ever been and this remained so for a long time. Major Persian invasions of Syria and eastern Anatolia occurred in the 250s AD. Antioch was sacked twice and the fortress of Dura Europos destroyed. The emperor Valerian himself was captured in 259 AD outside Edessa. The eastern frontier was in a disaster by 260, with Roman Mesopotamia, Syria and Cappadocia in Persian hands, the Roman eastern armies defeated and the Roman emperor a Persian captive. The Persian forces were only expelled from the eastern provinces by an unexpected force, the client state of Palmyra. The Palmyrene usurpation of the power in the East was however not taken well by the Romans. In 273 AD he Palmyrene forces were defeated and the city destroyed by emperor Aurelian.[51]

Before the arrival of the Romans, the Ptolemies had already begun to exploit the trade with both India and Arabia by constructing ports along their Red Sea coast. The sea trade was well established by the end of the Hellenistic period. With the absorption of the Ptolemy kingdom in the Roman Empire, these trade routes came in Roman hands. The amount of cargo passing through grew exponentially however after the Romans entered the game.[52] The zenith of the Roman trade through Egypt was from the reign of Augustus and lasted until the mid- to late 2nd c AD. Thereafter the trade seems to have declined dramatically. As seen above the 3rd c AD was a period of political instability, military turmoil, and economic chaos in many areas of the Roman world. This atmosphere was not very encouraging for long-distance maritime or overland caravan trade. There seems however to have been a revival of this commerce in the 4th – 7th c AD.[53]

2.1.7. The Nabataean kingdom

The Nabataeans are known from Hebrew and Assyrian sources from as early as the 7th c BC as a nomadic tribe inhabiting the desert regions of NW-Arabia. The Nabataeans may originally have come from S-Arabia. There is no sudden invasion of S-Jordan, but a long process of usually peaceful infiltration and gradual displacement of original inhabitants between the 6th and the 4th c BC. They assimilated many cultural ideas in the process and absorbed much of the population. By the end of the 4th c BC they had established a base at Petra, which gradually grew in wealth from its privileged location alongside several important trade routes. By the end of the 2nd c BC, if not earlier, the Nabataeans were ruled by kings from their capital at Petra. At the peak of the Nabataean power their territory spread from the south of Israel and Syria, Jordan and the Sinai to Hegra/Egra (modern Meda'in Saleh) and Dumat al-Jandal (modern al-Jawf) in NW-Saudi Arabia.[54]

The trade routes the Nabataeans controlled were an essential link between the Roman Empire and S-Arabia, India and E-Africa. Some goods arrived by ship at Leuke Kome on the Arabian shore of the Red Sea. From Leuke Kome the goods went by caravan through several stopovers to finally arrive at Petra. Other routes radiated from the Red Sea port, including a western road across the Sinai to Egypt. From the Nabataean capital a route extended west to finally reach the Mediterranean at Gaza. A second major road led north from Petra, over Bosra (ancient Bostra) and then to Damascus. From Damascus several routes branched of to the Mediterranean ports. Great land routes also ran from Petra to the Gulf and to S-Arabia. All these routes carried S-Arabian (frankincense, myrrh, incense and perfumes) and possibly Indian (spices) goods destined for Syria and the rest of the

[51] Parker, 1986: 132.
[52] Young, 2001: 19.
[53] Sidebotham, 1991: 33.
[54] Ball, 2000: 60-61.

Mediterranean[55]. In addition they also seem to have been engaged in marketing asphalt from the Dead Sea. But the Nabataean economy also involved pastoral herding and brigandage.[56]

The first not very successful Roman intrusion into Nabataea was in 63 BC following the incorporation of Judaea. This was followed up by another inconclusive campaign in 55 BC. From this time onwards Nabataea seems to have become a client kingdom of Rome, but the sources are unclear. It is certainly true that Nabataean kings from then onwards appear to be more answerable to Rome.[57] Nabataea was finally annexed by the Romans in 106 AD and transformed in the Roman *Provincia Arabia*. This has to be viewed as one component of Trajan's overall policy of expansion and Rome inherited a vast desert frontier, or rather the control of a vast desert area where no fixed line would be conceivable[58].

The Nabataean international traffic reached its peak during the reign of Aretas IV (9 BC-40 AD)[59], but the Nabataean control of the trade from S-Arabia started to decline in the 1st c AD. Advance in maritime technology made the sea routes more feasible than the land routes traditionally controlled by Petra. Roman Egypt opened the new port of Myos Hormus on the Egyptian side of the Red Sea and absorbed much of the trade passing through. This is reflected in S-Arabia, where a major shift in the pattern occurred from the old trading kingdoms of Ma'an, Saba and Qataban on the inland desert fringes, to the new kingdoms of Himyar on the highlands. On the other hand the emergence of Palmyra from the 2nd c AD onwards along alternative land routes from the east through the northern Syrian Desert, absorbed another part of the trade. The latter should be regarded with some care since it is not necessarily true that the trade that went through Palmyra had formerly passing through Petra. Moreover G.K. Young states that the commodities traded through Palmyra were entirely different from those of the Nabataeans[60]. However, despite the decline in long-distance trade, the substantial developments that the Nabataeans had made in hydrology, water management and agriculture ensured that Petra continued to flourish and even expand.[61] Petra was replaced by a new capital, Bosra[62].

2.1.8. The city of Palmyra

The great rise of Palmyra is mainly situated between 130 and 273 AD. During the 2nd c AD it was the chief centre in Mesopotamia for trade with the East. Its main advantage was the fact that Palmyra lay 'upon' the shortest route between Rome and India, and was placed between the Parthian/Sasanian and Roman Empires, maintaining good relations with both as middleman and buffer state. After the disappearance or weakening of large trading centres such as Petra and Gerrha in the 1st c AD, Palmyra found itself in the perfect niche to take control and exploit this advantage.[63] Although the Palmyrenes did not extending their territories much outside the Syrian Desert, they organized a trade network spanning much of W-Asia and the Mediterranean that channelled merchandise through Palmyra. Cities like Seleucia-Ctesiphon, Babylon, Vologeses, and Spasiou Charax played an important role in that network. Oddly enough Palmyra was not so much part of a trans-Asian caravan network as suggested by its location, but formed virtually as much a part of the sea trade network as any seaport did. Palmyrene merchants travelled directly across the desert to the Euphrates, from where they would sail down to the Gulf to Charax. From there, the trade became part of the sea routes to India and the Far East, with Palmyrene agents established in ports of call

[55] G.K. Young however mentions that the Nabataeans were solely involved in the aromatic trade from S-Arabia (Young, 2001: 19).
[56] Warmington, 1974: 12; Parker, 1986: 115-116; Ray, 2003: 175.
[57] Ball, 2000: 63.
[58] Parker, 1986: 118, 123; Gawlikowski, 1997: 42
[59] Potts, 1991: 142.
[60] Young, 2001: 137-138.
[61] Goodman, 1997: 259; Hoyland, 2001: 72-73.
[62] Ball, 2000: 63.
[63] Warmington, 1974: 100-101.

on the way. In this way Palmyrenes established a commercial empire that reached throughout the known world. Although Palmyra's merchants were directly involved in the shipping business and importation of goods, the majority of the Palmyrene business was arranged around the transport of products from the Euphrates' western shore to Palmyra and further on to the Mediterranean. Today, evidence for the presence of Palmyrene merchant has been found as far apart as Charax, Bahrain, the Indus Delta, Merv, Rome and Newcastle-upon-Tyre in England.[64]

The opinions on when Palmyra was incorporated in Roman Empire vary. It presumably must have been by 75 AD or earlier. It was certainly no later than 114 AD, perhaps as part of Trajan's annexation of Nabataea. But it was not a Roman province in the same way that the rest of Syria was. The Romans instead were satisfied with merely installing a garrison there to counter Parthian influence.[65] By the second half of the 1st c AD it had the standard regime of a provincial city. In spite of its cultural particularity Palmyra remained under the firm Roman control with no hint of independence until the 3rd c AD. The territory of Palmyra formed a buffer zone, while the city itself remained a part of the province. The regular caravan traffic to the Euphrates and down to the Gulf presupposed conditions of security that only a complex web of relations with the nomads could have guaranteed. Moreover the Palmyrenes had to diverged a trade route that passed through their city, since the city is not located on a 'natural' caravan route. The natural trade route goes up the Euphrates to just east of Aleppo, before branching off directly to Antioch, which was after all the main market and enterpôt. Diverting from the Euphrates to cross the desert to Palmyra and thence to Emesa or Damascus, from where the nearest ports were relatively minor ones, was certainly neither natural nor logical. Palmyra is the only example in history when this route was the main one. To maintain this itinerary Palmyra had to offer protection from brigand tribes and provide the caravans with water and shelter. No one but the nomad tribesmen could have provided the necessary growth potential for Palmyra. The same basic nomadic knowledge was needed for the creation of the desert ranches in the north-western hills, essential for the mass breeding of pack animals for the Palmyrene caravans. Palmyra appears to be a successful settlement of nomads within the pre-existent frame of sedentary life. The success is undoubtfully triggered by the opportunities for trade that opened with the Roman peace, an activity always pursued by Arabic nomads.[66]

From 250-260 AD onwards Syria experienced a slowing of economic activities which is generally considered as a part of the "crisis of the 3rd c AD". It should be noted that the evidence of inscriptions indicates that the pause of activity was brief, lasting no longer than 20 years, and that and that after the defeat of the Romans by the Sasanians in 259 AD the power of Palmyra increased again.[67]

A rather important political event in the history of Palmyra was the military power accumulated by its leader Odenathus, who led the Palmyrene army to victory against the Sasanians. His accumulation of power is related with the weakness of Rome, following the death of Emperor Severus Alexander in 235 AD. Odenathus was assassinated in 267 AD and as a result his wife Zenobia came to power as a regent. She became increasingly independent of Roman control and after defeating a Roman army Zenobia conquered all of Syria, Cappadocia, Palestine and Egypt and at least N-Arabia by 269. She established a short-lived Palmyrene Empire but was however defeated by the Roman emperor Aurelian in 273 AD and Palmyra was destroyed.[68] The destruction of Palmyra also meant the end of its importance in the trading network and there are no more inscriptions after 272 AD. There is however evidence for continued trade elsewhere in Roman Syria and Mesopotamia, and

[64] Mattingly, 1995: 227; Ball, 2000: 76; Young, 2001: 80.
[65] Ball, 2000: 74.
[66] Gawlikowski, 1997: 44; Ball, 2000: 74; Hoyland, 2001: 76; Young, 2001: 137-138.
[67] Tate, 1997: 58-59; Ray, 2003: 176 (footnote 6).
[68] Parker, 1986: 132.

there is no reason to believe that this trade only appeared after the fall of Palmyra. It is far more likely that it had coexisted all throughout the period. It would seem that mainly the Palmyrenes themselves used the route through Palmyra. The Greek, Jewish and Syrian merchants active in Mesopotamia had probably continued to use the Euphrates route all along, and no doubt continued to do so after the fall of Palmyra.[69]

2.1.9. The South Arabian kingdoms[70]

One should not forget that strong kingdoms emerged in S-Arabia in the late first half of the 1st millennium BC, which established early contacts with the E-Mediterranean.[71] The history of S-Arabia is easier to reconstruct than that of E-Arabia in that some ten thousand inscriptions are known from the region. The downside is that the majority cannot be exactly dated. The very mountainous terrain prohibited the formation of a single regime, and political power was in general fragmented among the various peoples of S-Arabia.

From the end of the 2nd – beginning of the 1st millennium BC till the beginning of Islam, several strong, centralized and powerful states emerged in S-Arabia, being the kingdoms of *Saba'*, *Qataban*, *Ma'in*, *Hadramawt* and *Himyar*. Because of their well-developed agricultural system, but mainly due to the presence and export of frankincense and myrrh, and the strategic position between Africa and Asia, these states were very prosperous.[72] Aromatics were extremely favoured in the Roman Empire and elsewhere, and S-Arabia and E-Africa are the only places where the vegetation grows from which these products are made.[73] Till the 2nd c BC, these aromatics were marketed by means of the Arabian overland trade routes: on the one hand from Najran to Yathrib (modern Medina), Dedan, Hegra, Petra and Gaza; on the other hand from Najran to Qaryat al-Fau, Aflaj and Gerrha.[74] All these cities were on the fringes of the desert, because it was via the desert that aromatics passed on their long journey to the Mediterranean and Mesopotamia. Whoever wished to participate in this trade was therefore obliged to take up a position near to this route. The S-Arabians even expanded their power to East Africa where colonists founded the kingdom of Aksum, in present-day Ethiopia.

Saba' (8th c BC – 275 AD) with the capital at Ma'rib, was situated at the junction of the trade routes connecting the frankincense lands with the Mediterranean ports, and NE-Arabia. The economy was based on agriculture, the producing of frankincense and myrrh, gold, honey and wax. However the Sabaeans owed their enormous wealth to trade with distant nations, including the transit of all goods shipped westwards from India. The trade passed through the S-Arabian ports of Qana' and Aden, from where it went by caravans along the frankincense routes to the Mediterranean. The great dam of Ma'rib is an indication of the strength and power of the Sabaean kingdom.[75] The Sabaeans' territory was probably limited initially to Mar'ib and its environs, but the military expansion of various leaders added new lands. It is quite possible that the Roman attack against Ma'rib in 25 BC greatly weakened this kingdom. The Sabaeans had been weakened so severely that they were forced to look for a coalition with *Himyar* or *dhu-Raydan* and formed the united monarchy of *Saba and dhu Raydan*. In the 2nd c AD the Sabaean people however campaigned against their coalition partner, initiating a Sabaean renaissance for a century and a half, but at the end of the 3rd c AD this dynasty seems to simply die out.

[69] Mare, 1995: 203; Young, 2001: 183-184.
[70] This summery is mainly based on Sedov, 1996: 11-35; Hoyland, 2001: 36-47 and Avanzini, 2002: 18-21, unless stated otherwise.
[71] Salles, 1996b: 295.
[72] Doe, 1983: 4.
[73] Doe, 1983: 252.
[74] Deblauwe, 1991: 143-146.
[75] al-Saud, 1997: 36-37.

The kingdom of *Qataban* (4th c BC – 200 AD) was the southwestern neighbour of Saba'. Their capital was Timna' (Hajar Qulan) in wadi Baihan. They were able to become a separate state from Sana' around 400 BC and reached their zenith of power in the 3rd and 2nd c BC. The Qatabanite territory went as far as the Indian Ocean, what gave them the change to control the coastal trade[76]. Around the 2nd c BC the balance of power broke down. Arab nomads, who were no doubt already present in all the foothills bordering the desert, seized control of Jawf, one of the principal valleys. The Qatabanite territories in the highlands broke down and part was annexed by the coalition of Sana and Himyar.

The state of *Ma'in* (8th c BC – 100 AD) had its capital at Karna (Qarnawa), and the territory was located in the large river oasis al-Jauf. The kingdom was situated on the Red Sea side of the Arabian Peninsula. They also became a separate state from Sana' around 400 BC. They controlled most of the long distance trade route where the caravans took on their way to the Mediterranean. To protect this route the Minaeans established a colony far out in the NW-Arabia, in an area called al-'Una in the oasis of Dedan.[77] Their power peaked in the first halve of the 3rd c BC. The Minaeans had already lost their influence in the course of the 1st c BC.

The kingdom of *Hadramawt* (8th c BC – 300 AD) became independent of the Sabaean Empire in the 4th c BC. They seem to have controlled the frankincense-growing region (Dhufar) and became the main supplier. Their capital was Shabwa (Sabata). The state probably reached its climax of wealth in the 1st c AD, when Indian and Roman ships landed at Qana' bringing goods to Shabwa[78]. When the kingdom of Qataban broke down, a part of it was absorbed into Hadramawt. The troops of Hadramawt led successful wars against the neighbouring kingdoms and occupied their territories. The settlement of *Moscha Limen* or *Sumhuram* (Khor Rori) was founded in the early 1st c AD. It was a storehouse for incense, the departure point for short sea transport to Qana' and a stopping point for some Indian mariners who had arrived too late to profit from the monsoon and were forced to spend the winter. Hadramawt became the largest and probably one of the most powerful kingdoms among the S-Arabian states.

The capital of the kingdom of *Himyar* or *dhu-Raydan* (2nd c BC – 520 AD) was situated at Zafar in the fertile southern highlands. From Zafar a road led to the port of *Muza* (modern Mocha) at the northern end of the straits of Bab al-Mandab, where Arabia almost touches Africa. Already in the mid-1st c AD they achieved a prominent position in the region. Himyar was to face serious menace when at the very beginning of the 3rd c AD the Abyssinians attacked Arabia. Allied initially with Saba, their troops posted themselves in the whole of W-Yemen. An internal struggle between the allies broke out however and in the late 3rd c AD the king of Himyar conquered Hadramawt and what remained of Saba, and S-Arabia became a unified state for the first time.[79]

The passage from land to sea trade can help throw light in general on the relationship between S-Arabian power and commerce. A sudden change of the trade route from desert to the sea, motivated by the Roman arrival on the Red Sea in the 1st c BC and better knowledge of the monsoon winds, is too simplistic however. An additional factor that has to be considered is that in the period of the 1st and 2nd c AD a number of nomadic Arab groups in the S-Arabia began to migrate from their homeland. Literary and epigraphic evidence indicates that they went to the north (Syrian desert), to the east (Bahrain/Oman) and the south (Yemen). What triggered this migration is unclear, but is probably connected with upheavals in the kingdoms of Yemen. The old S-Arabian kingdoms, located around the

[76] al-Saud, 1997: 38-39.
[77] al-Saud, 1997: 38.
[78] al-Saud, 1997: 39-40.
[79] Robin, 1997: 52-53.

desert, which had dominated the region's affairs up until this point, were gradually overtaken by the tribes of the highlands.

Ma'in, the caravan kingdom *par excellence*, dies about the year zero, and *Qataban's* collapse a couple of centuries later is also linked to its lack of access to the sea after the wars with the Himyar kingdom. This illustrates the strong connection between commerce and power in S-Arabia. The entire history depends on the ability to manage trade over long distances, whether by sea or by land. Trade was important but it should not be forgotten that irrigated agriculture was as important to the survival of these kingdoms. The foundation and further development of the port of Qana' resulted from the involvement of the kingdom of Hadramawt in the international sea-trade between Roman Egypt, Arabia and India. Frankincense and aloe, the main local products traditionally exported from Hadramawt via caravan routes, started to be sent now in large quantities by sea through this port of trade as well. On the other hand the direct sailing between Egypt and Indian subcontinent which became regular and very intensive from the 1st c AD onwards, probably necessitated the foundation of a transit point on the southern coast of the Arabian Peninsula supplying water and food products. Furthermore it is clear that not all trade was in luxury items such as perfumes, spices or pearls. There were also (completely different) routes for essential goods such as foodstuffs and other items. These routes ran along the coasts of the Arabian Peninsula and India without necessarily taking the open sea.

During the mid 2nd – 4th c AD Qana' probably had its heyday. The trade connections with the Mediterranean region which was reduced somewhere in the second half of the 2nd c AD were with no doubt reactivated in the late 2nd – early 3rd c AD, although not in the same quantities as before. In the late 2nd – early 3rd c AD Qana' was transformed from a small coastal settlement used mostly as a transit point for vessels on their run from Egypt to India into the extensive port-city of the Hadramawt and later Himyarite kingdoms. The increased material evidence of the Gulf and Indian imports as well as the reduction of Mediterranean items show, that in contrast with the previous period there was a change in the character of the sea-trade. The commerce was now concentrated in the hands of Hadramawt middlemen and/or foreign merchants who had their temporary residence in Hadramawt. As early as the 4th c AD the incense markets began to collapse. There are three reasons that could be the cause of this collapse. First of all the spread of Christianity in the Roman Empire had an impact on the use of incense, since no incense was used at that time in the church ceremonies. A second reason could be that due to the weakening of the Roman power, the insecurity along the incense routes increased. A last reason are the many wars that took place between the S-Arabian States, especially between the Late Himyarite kingdom and the Aksumite kingdom. Amphorae material from the 5th c – early 7th c AD shows very strong trade links between late Qana' and the regions of S-Palestine or SW-Jordan[80]. The connections with the Indian subcontinent were reduced almost completely.

2.1.10. The kingdoms in the Indian Subcontinent

Indian contact with the Gulf region is a long-standing one and goes back to the 3rd millennium BC. The Harappans had extensive trade-links within and outside India with amongst other goods the export of lapis lazuli.

During the period under consideration the India subcontinent was far from anything that resembled a national unity. While the *Magadha* kings ruled over a part of NE-India, the *Andhra Satavahanas* ruled C-India from coast to coast. The NW-India was the battleground of Scythians/Sakas and the Parthians, and eventually was conquered by the *Kushans* (1st c AD), stretching into C-Asia. In the south, there were three kingdoms, the *Chera* on the SW-

[80] al-Saud, 1997: 50.

coast, the *Pandya* in the southernmost part and the most powerful and richest *Chola* kingdom in the SE-part (including the port of Arkamendu).[81]

The *Kushan kingdom* stretched over a region that nowadays consists of parts of Tajikistan, Afghanistan, Pakistan, down in the Ganges valley of Northern India. They are also the rulers of *Gandhara* (which is situated above Bactria) during their heydays. They ruled from the 1st c BC till the 3rd c AD and are of C-Asia origin, the same or related to the Indo-European *Yüeh-chih.* The kingdom had extended friendly and unfriendly contacts with Rome, Persia and China and formed a centre of contact between East and West. The kingdom lay at the junction of three culture spheres: the Indian subcontinent, Iran and the Hellenised Orient, and the steppes of Central Asia. The power of the Kushans was only overthrown in 460 AD with the invasion of the Hephthalites.[82]

The two most important ports in India for the Roman trade were *Barygaza* and *Muziris*. Up until the 2nd c AD, the trade was mainly with the Buddhist Tamil kingdoms in S-India, i.e. the Cheras, the Cholas and the Pandyas. A secondary sea route reached the Indo-Scythian and later Kushan kingdoms at the mouth of the Indus at *Barbarikon* in NW-India.[83]

The Roman trade with India started around 25 BC and continued for more than three centuries. There are written accounts of the Greek origin residing around Egyptian Alexandria and serving under the Roman Empire. Following the annexation of Egypt by Augustus in 31 BC, Rome opened its direct trade route with India, and within a few years (in 22 BC) a Pandyan king sent an ambassador to Augustus. During the reign of Augustus the monsoon wind, crucial for direct shipping to India without following the Arabian and Persian coast, was 'discovered' for the first time by a Graeco-Roman sailor, Hippalos. Before the commencement of the direct trade, the Romans used to obtain Indian goods through intermediaries such as the Syrians, Jews, Parthians, Axumites, etc.[84] *Denarii* and *aurei* minted by Augustus and the 1st c AD Roman emperors (till Nero) are found in thousands in the Indian soil. Tiberius pursued a careful and successful financial policy, and expressed his anxiety at the great increase of oriental trade. He censured the wearing of silk. The fact that at the time of Tiberius' reign a lot of money flowed to India is confirmed by the numerous coins of his reign found in India.[85]

The trade seems to have gone through a decline after that first period of booming. Ethiopians and Arabians most likely became the middlemen again rather than the Egyptian Greeks from Alexandria. Barter trade replaced gold, hence showing up less in the archaeological record. The trade again picked up its momentum until Marcus Aurelius (AD 161 – 180) caused a second break in trade on account of reversed trade balance, plague, war expenses, etc. This break lasted till the reign of Commodus (177 – 192 AD) after which the last phase of the Indo-Roman trade started, which was eventually terminated by the Arabs and Persians.

With the revival of the trade after the 4th c AD, Sri Lanka was the main trade market. After the 5th c AD the Nestorian Church became prominent in the trade. Being mainly Syrian in origin, the Nestorians were simply inheriting a tradition that begun by the Palmyrenes. By the 6th c AD Iran ruled the waves from naval bases throughout the Gulf as well as in S-Arabia, the mouth of the Indus and possibly Sri Lanka, with mercantile colonies stretching from the E-African coast to the South Chinese Sea.[86]

[81] Biswas, 1996: 289; Ball, 2000: 129-133.
[82] Rosenfield, 1967: 1; Masia, 2000: 216.
[83] Warmington, 1974: 291; Ball, 2000: 129-133.
[84] Biswas, 1996: 289-291.
[85] Warmington, 1974: 41.
[86] Warmington, 1974: 52-54; Biswas, 1996: 291; Ball, 2000: 129-133.

There is no doubt that there were extensive trade connections between Rome and the Indian Subcontinent. In the Roman times there were three possible routes between the Roman Empire and India: the sea-route via the Red Sea, the route via the Gulf and the overland routes through Mesopotamia and Persia.[87] Of the few bits of both archaeological and literary evidence that we have by far the bulk supports a mainly sea route from the Gulf and the Red Sea to NW-India, through the Kushan Empire. Other overland routes were almost negligible in terms of Roman trade links with C-Asia.[88]

[87] During Caspers, 1988: 25.
[88] Ball, 2000: 138.

2.2. SE-Arabia – United Arab Emirates

2.2.1. Mleiha (Sharjah, U.A.E.)

2.2.1.1. Geographical context

Mleiha is situated in the Emirate of Sharjah (U.A.E.), in the central part of the Oman Peninsula at the western piedmont of the Oman Mountain chain, in between the plains of Dhaid of al-Madam[89]. Dune sands locally cover this basin of fluviatile deposits of Pleistocene and Holocene date. The site itself lies upon the floodplain of *wadis*. The plain is covered with fine material, gabbros, basalts, ophiolithes from the mountains and sand, all of them covering a marl layer at 0,5 to 1 m beneath the present surface.[90] The location is well protected from the sands blowing from the west by an anticlinal Miocene limestone outcrops (Jebal Faiyah). The Mleiha area has benefited from favourable environmental conditions for agricultural activities in the past, as is still the case today. The mountains regularly provide alluvial deposits and water is retained in the *jebals* and was accessible by wells. The site extends over more than 3 km².[91]

2.2.1.2. History of research

The site of Mleiha was first noticed in the 1960s as the result of the finding of an inscription in S-Arabian script. The first archaeological attention given to the site was by the Danish archaeologist K. Frifelt in 1968, who was one of the pioneers in this part of the Gulf-region. B. de Cardi visited the site that same year. In 1973 an Iraqi archaeological mission carried out a survey of the U.A.E, which had just become independent. Besides survey work and some soundings, limited excavations were undertaken at the site of Mleiha. In the tomb they opened, a Mediterranean *amphora* fragment from Rhodos was found that allowed a dating to the end of the 3rd c BC – beginning 2nd c BC. The *French Archaeological Mission* first started working on the site in 1986, in the frame of a broader study on the ancient environment and archaeology of the U.A.E. Since Mleiha was (and still is) the only site with a continuous stratigraphy from the end of the Iron Age till the 1st c AD, it offered great opportunities to gain new insights in the badly known history of the region. Eight campaigns (directed by R. Boucharlat and M. Mouton) took place between 1986 and 1995, and a methodical survey of the site was undertaken combined with thorough excavation where needed.[92] Since 1999 the work has continued under the direction of A. Benoist, who finished the excavation of the fort and is focussing mainly on the processing of the archaeological data[93]. In 1993 and 1994 additional excavations were undertaken by the *Department of Antiquities of Sharjah*, headed by S.A. Jasim[94].

2.2.1.3. Archaeological chronology & context of Mleiha in another nutshell

- **Chronology**

In his PhD-dissertation M. Mouton devised the main chronological sequence of Mleiha. He proposed a chronology of four4 phases called the *Pré-Islamique Récent*, e.g. PIR A, PIR B, PIR C and PIR D. This division is primarily based on material from Mleiha and ed-Dur, but material from some additional sites is also included.[95] This matter will be treated more extensively because of the obvious importance to the archaeological chronology of ed-Dur. Mleiha was occupied from the 3rd c BC till the 4th c AD and is the only major inland settlement in the Oman Peninsula for that period. By the time ed-Dur emerged on the shore of the Gulf, Mleiha had already been occupied for 3 centuries.[96]

[89] Mouton, 1992: 20; Boucharlat & Mouton, 1994: 13.
[90] Boucharlat & Mouton, 1991: 23.
[91] Boucharlat, 1991: 291.
[92] Mouton, 1999b: 9-11. For an extensive bibliography on Mleiha see Mouton, 1999c.
[93] Benoist, Mouton & Schiettecatte, 2003.
[94] Jasim, 1999; Jasim, 2001.
[95] Mouton, 1992; Mouton, 1999b: 13-19.
[96] Boucharlat & Mouton, 1991: 23.

The fact that a clear chronology could be developed for Mleiha is caused by the presence of well-stratified profiles in certain areas of the site. These are the result of a continuous occupation over a long period of time in the same area. This is in sharp contrast to the situation at ed-Dur that basically is a one-layered site that was occupied for a 'short' window in time. Based on a cross-trench in the 6 m deep archaeological deposits of area L, several occupation phases could be identified. The deepest occupational level lies directly on the alternation of the silt sediment and is only attested by a few potsherds. This layer was covered by a heavy sandy horizon (*ca*. 1 m thick) with a few potsherds and a hearth structure. Next were a number of successive sand deposits, darker in colour and mixed with ashes (total thickness of 0,8 - 1 m). These layers yielded an abundance of ceramic material and the remains of a badly preserved mudbrick structure. This layer is covered by thin layers of courser grains resulting from the disintegration of mudbrick walls. The levels with granules alternated with sandy deposits (not thicker than 20-30 cm), in some places intermingling and yielding relatively less pottery. A continuous sand layer (20 to 30 cm deep) closed of these alternating levels. On top of this sandy stratum, two successive occupation levels were connected with two waste deposits containing abundant material.

The 'absolute' dates are based on the presence of imported pottery and other artefacts that have well dated reference material elsewhere, present in this sequence. By making associations between the local and the imported material, it was also possible to date layers without foreign objects.[97] The chronology of the site is summarised in Table 1.

Level	Deposits	'Absolute' dates	PIR	Extra
Level I	Sand deposit on alternation silt sediment	Virgin soil	**-**	
Level II	Horizontal sand deposits mixed with ash	3rd – first half 2nd c BC	**PIR A**	Iron objects first occur
Level IIIA	Succession of building levels	Second half of 2nd – 1st c BC	**PIR B**	
Level IIIB	Two successive occupation levels	1st c BC – 2nd c AD	**PIR C**	Occupation of Ed-Dur
Level IV	Occurs only on limited part of the site, Areas DA & CW	3rd – (4th) c AD	**PIR D**	

Table 1: Chronology of Mleiha.

The material culture of the PIR-period is differs considerably from previous Iron Age material. The fabric of the ceramics still has some parallels with that of the Iron Age, but on the typological field the pottery is more closely related to that found with the C- and E-Arabian communities. The typology of the stone chlorite vessels is also radically different from that of Iron Age material. The turning wheel is introduced for their production. Iron metallurgy occurs for the fist time at a large scale in the area, although copper-base alloys stay in use mainly for vessels production and decorative elements. Also glass vessels are attested for the first time in the region, together with other imported products (ceramics from Mesopotamia, Greece, etc.).[98]

There is not only a break in the material culture, but also in the cultural behaviour. The habitation is completely different. There is no continuity in the construction techniques, settlement organization and domestic organization. The first settlers of Mleiha built structures made of light materials (*barasti*-type structures), the remains of their dwellings being post-holes, stone lines and occupation floors. The traditional Iron Age architecture, stone or mudbrick houses, seems to be forgotten or voluntarily ignored. It is only in the PIR B period mudbrick work reappears and slowly evolves into more elaborate structures. A clear sign of sedentarisation of an originally nomadic people. There is also a compete break in the burial

[97] Mouton, 1999b: 14-16.
[98] Mouton, 1992: 274-275 & 280.

practises. A somewhat clearer hierarchical organisation can be observed, absent in the Iron Age. The tombs at Mleiha are placed in organised cemeteries, more simple tombs are clustered around monumental tombs of prestigious people (ancestor or communal chief). This points towards strong family or clan-like ties. This change is more indicative for a cultural shift than the new site organisation, since it is linked to the religious believes and social structure of the society. The use of writing, S-Arabian script, is observed for the first time.[99]

The imported materials point to the importance of exchange. The origin of the PIR culture at Mleiha could be found in a group of new migrants. The most favourable region of origin would be S-Arabia since the iconographic representations on the copper-base alloy vessels have the closest parallels with S-Arabia. The funeral architecture has some similarities with S-Arabia, but also with the Nabataeans and to a lesser extent Syria. To this we can add the use of the S-Arabian script and the worship of the sun-god Shams/Shamash. The construction of monumental tombs in materials chose to survive time, correspond well with the psychology of the nomad. After a long life of moving a permanent structure is chose to impress later groups that pass by. The iron weapons correspond with a warring, moving society that meets other groups like hem, but also with the need to defend newly acquired home territory.[100]

• **PIR A context** (3rd – first half 2nd c BC)
During PIR A the site of Mleiha extends to its maximum surface: it looks like an immense camp that stretches over an area of 1 km (N-S) by 1,5 km (E-W). The closeness of the post-holes on the occupation levels indicates a rapid succession of dwellings, which might indicate that the population still is mobile, maybe following a seasonal rhythm. The continuity and stability of the settlement at Mleiha, as a community is proven by the installation of dozens of small cemeteries that border the site to the E and the S. In these cemeteries solid square mudbrick monuments were built, a sort of massive towers, decorated with crenellated stone ornaments. They cover a rectangular underground individual cist burial chamber richly furnished with funerary objects (imported pottery and glass, weapons, ornaments, etc.). Around these monumental tombs, built in the middle of each cemetery, there are plainer tombs, simple grave pits with mudbrick facing, sometimes associated with a smaller monument. The architectural features of these monumental tombs, as well as their decoration have no antecedents in the region. They do recall the funerary traditions of other communities that settled on the borders of the desert of Arabia at about the same time however, namely at Petra (Jordan) and Qaryat al-Fau (SW-Saudi Arabia). This similarity in a domain that has deep connection with religious beliefs and practices bears witness of some cultural relationship, which could come from a period prior to their respective sedentarisation.[101]

The archaeological material preserved in the funerary deposits show that the community of Mleiha was well integrated into the trans-Arabian trade. As much as the preceding Iron Age communities seem to have been rather isolated, so the population of Mleiha, even since the ancient period, seems to actively participate in the trans-Arabian commercial. The trade connected the Seleucid Empire with S- and E-Arabia that constituted the wealth of the caravan-towns in Arabia during the last centuries BC. In that period there existed as yet no harbour on the long desert coast between Qatar and the Straits of Hormuz. Caravan trade would have been the only way goods from Western production centres could have reach SE-Arabia mainly through N- and NE-Arabia. The trade, the contacts with the communities living in E-Arabia and the needs of the local economy have naturally led to the use of 'money'.[102] The coins are only small units, probably brought there by caravan merchants, but they are

[99] Mouton, 1992: 275-276 ; Mouton, 1999b: 20-21.
[100] Mouton, 1992: 279.
[101] Boucharlat & Mouton, 1998: 28-29; Mouton, 1999b: 21; Andersen, 2005: 291.
[102] Mouton, 1999b: 22.

not in themselves a proof of commercial exchange. These early coins could well have been a prestige coinage, minted by a ruler who wanted to assert his power and independence. Before it had any commercial use, this currency was mainly a political tool, and possibly a military tool used, for instance, to pay mercenaries, as often happened in the Hellenistic world.[103]

The few potsherd found in Level I (the deepest level) of area L were of common and coarse wares that might date to the Iron Age or later. No definitive date could be proposed however, moreover this level was nowhere else encountered at Mleiha.

The distinctive element in dating Level II was the present of sherds of Greek *amphorae*, absent in later levels. Although not numerous, their presents in some of the tombs made the link to other objects possible. The *amphorae* originating from Rhodos can be precisely dated to the second half of the 3rd c BC until the first quarter of the 2nd c BC. Additionally glazed pottery and moulded glass, typical materials from Mesopotamia, Iran and the Eastern Mediterranean are also usually dated to the 3rd and 2nd c BC.[104]

The position of Mleiha in this early period was most likely that of the seat of a small local kingdom, rather than a trading *emporium* as it was not located directly on one of the important international trade routes. Due to its location in a well-watered and fertile plain, agriculture was probably the main activity around the site. It could also have served as a local market, distributing objects further afield towards the Gulf of Oman.[105]

• **PIR B context** (second half of 2nd – 1st c BC)
At PIR-B, the inhabitants of Mleiha started to build their houses in mudbrick, a material only used until then for the tombs. But until the end of the 1st c BC the houses remain sets of units, most often separated. The settlement seems to be less extensive at this period. The organisation and position of the cemeteries are the same as those of the previous period. The monumental tombs, however, are larger, with real substructures consisting of one or two chambers, stairs and an access passage. They are associated with massive monuments made of mudbrick, but their elevation was not sufficiently preserved to know if they were just platforms or towers. The excavations have brought to light several workshops, where copper, iron, soft stone and bone were worked. These industries relied mostly on raw materials from the Oman Mountains.[106]

Level IIIA actually did not contain any elements that allowed dating. The overall assemblage of the ceramics is quite distinct as a whole from the previous level. No Greek *amphorae* are present anymore, as are some types of common wares characteristic for the previous phase. New ceramic types appear on the other hand. The separation is based on these differences and the beginning of this period is estimated somewhere after the last date given by the *amphorae*, towards the middle of the 2nd c BC. The absence of blown glass (which appears in the Middle East in the 1st c AD) is used as a *terminus anti quem* as well as the absence of some forms of glazed wares, widely spread in the 1st and 2nd c AD (which are present at ed-Dur). All this lead M. Mouton to put the end date of this phase towards the end of the 1st c BC.[107]

This period corresponds to the time that the Seleucid power ceased to exist in the Gulf, after the death of Antiochus IV in 164 BC. It was replaced in the north by the small Characenean kingdom, which soon became an essential link in the trade between the Mediterranean countries and Persia, and S-Arabia and India. During this transition the major commercial

[103] Callot, 2004: 141.
[104] Mouton, 1999b: 16-17.
[105] Haerinck 1999: 127.
[106] Mouton, 1999b: 23; Callot, 2004: 141.
[107] Mouton, 1999b: 17.

routes did not disappear, but they seem to have given way to a different exchange pattern. Some communities of E-Arabia participated more actively and minted their own currency. The fact that the majority of the coins found in NE- and SE-Arabia are identical in both regions, leads to the conclusion that they used the same currency for commercial transactions mainly from the 3rd c to maybe the 1st c BC. In a certain way it was a common market, with NE-Arabia as the main provider of foreign goods. Although NE-Arabia probably acted as middleman, we should not forget that the flourishing kingdoms in S-Arabia could equally be considered as distribution centres. The main trade particularly for Western goods was done with caravans, coming in from NE-Arabia.[108] A closer contact between both coast sides of the Gulf is seen by the presents of a parfum bottle of *Londo Ceramic* from Baluchistan (Iran) and the parallels between the Mleiha painted ceramic and those from Tepe Yahya.[109]

• **PIR C context** (1st c BC – 2nd c AD)
At the beginning of PIR C the occupied area of Mleiha appears reduced in size. Some, but not many, large-sized buildings are spread on a wide space, alternating with poorer dwellings still formed by separate units. The cemeteries are still situated at the borders of the settlement, but only one tomb was excavated dating to PIR C. This is a stone monumental tomb, with a subterranean rectangular burial chamber and an open mausoleum, oriented more or less oriented N-S. The trade network seemed to remain as before, indicated by the Mesopotamian and Iranian pottery, glassware from Syria and Palestine and a few rare objects from S-Arabia. The most remarkable fact of this period however is the development of the site of ed-Dur on the coast. Both sites belong to the same culture and most probably to the same political entity. The coins attributed to the Oman Peninsula are exactly the same on both sites. Like Mleiha at its beginning, ed-Dur gives the impression of a large meeting place, undoubtedly connected with the sanctuary and its cult, which is in accordance with the number of cemeteries. [110]

At ed-Dur hundreds of coins have been collected over time, local issues as well as coins from NE-Arabia and foreign coins, e.g. Parthian, Characenean, Roman, S-Arabian and Indian, unknown in the previous periods. These coins were not then intended for trade, but they do indicate that international exchange took place. Although this trade did exist before, it seems to take another dimension in the PIR C period. It would seem that sea trade supplemented the major caravan routes of the previous phases and that a place such as ed-Dur became a favourable stopover. It is possible that the seat of power moved to ed-Dur at the times of meetings, but Mleiha was the capital of this region and most probably the seat of government.[111]

The fact that only the material of one tomb is known limits the information from Mleiha on this phase. M. Mouton mainly uses finds from the tombs at ed-Dur to fill this hiatus. Level IIIB brings a more distinct pottery assemblage. Some handles belonging to cups of Roman *skyphos* were collected and can be compared to examples from Eastern sites dating to the 1st c AD. In general the pottery assemblage can be compared with that from ed-Dur. The numerous fragments of blown glass (i.e. *pillar moulded* glass vessels, also encountered at ed-Dur) found, confirm a date after the beginning of the 1st c AD.[112] Imported material is much more abundant at Mleiha and ed-Dur now. To name but a few items (see also *Chapter 3*: xx-xx): Mesopotamian or SW-Iranian glazed ceramics, some E-Mediterranean *terra sigilata* fragments, glass from Mesopotamia or the E-Mediterranean, ceramics of possible Indo-Pakistani origin and some possible S-Arabian sherds).[113]

[108] Callot, 2004: 141.
[109] Mouton,1992: 255-256.
[110] Mouton, 1999b: 24; Callot, 2004: 143.
[111] Callot, 2004: 143-144.
[112] Mouton, 1999b: 18.
[113] Mouton ,1992: 256-259.

• **End PIR C – beginning PIR D context** (3rd – (4th) c AD)
At the end of PIR C and the beginning of PIR D (end 2nd c AD – beginning of the 3rd c AD), Mleiha had become a large village, with dwelling clustered around a large fortified residence that was built around that time. The fort in Area CW is a large, roughly square mudbrick building about 60 x 65 m. It is surrounded by a thick fortification wall, with eight towers, one placed at each corner and one in the centre of each side. Rooms were built on the interior against the wall and around a large central courtyard. The entrance was on the eastern side. The fort was built in three main phases and most of the material can be dated to the late PIR D phase. The oldest occupational layer is above a layer of broken mud-bricks laid by the builders to consolidate the soil of the slope. The second occupational layer is a simple reconstruction of the floors in some places, separated from the previous occupation by a thin layer of construction debris. This phase has yielded a huge quantity of material, which provides evidence of the rapid abandonment of the fort. The third occupation phase overlies the remains of the collapsed walls. During PIR D the settlement was limited to the central part of the site, concentrated around the fort and two or three additional large buildings.[114] The fort seems to have accumulated a wide range of activities: storage areas, dwelling areas, metallurgical activities, rotary stone grinders may indicate the processing of cereals, a defensive function, place of refuge in time of need, … The possible presence of a minting place shows an additional link to a politico-economical power.[115]

On the Gulf coast, ed-Dur was almost completely abandoned and only Area F yielded any remains dated to PIR D. However Mleiha still imported pottery, glassware and various objects from the north, from Mesopotamia and from the Mediterranean, as well as from the Persian coast and India, the African coast and even from Egypt. All these archaeological finds indicate that Mleiha had access to the sea trade routes of the Indian Ocean, which connected Egypt with India via the Red Sea. This opening to sea trade coincides with the foundation of the seaport of Sohar on the eastern coast of Oman, probably under the stimulating influence of the Sasanians, who sought to control the navigation routes from land. The foundation of Sohar was followed shortly after by that of Kush, a seaport on the Gulf, in the north of the Peninsula. These two ports were to remain the main ports throughout the Middle Ages and into modern times. It should be added that the Characenean kingdom lost its importance by the middle of the 2nd c AD and became totally controlled by the Parthian kingdom and later by the Sasanians.[116]

The date when Mleiha was abandoned, as well as the reasons that led to it, are still to be determined. The upper level of the fort has kept numerous broken transport and storage jars on the floors. In the neighboring houses, the floors were covered with luxury pottery, common vessels, storage containers, tools and countless objects. At the northern end the fortified residence shows evidence of fierce fire. All this seems to indicate that the site was apparently abandoned suddenly, perhaps as a result of some dramatic event. No trace of pre-Islamic occupation after the end of Mleiha has been found along the western foothills of the Oman Mountains. On the other hand, an increase in the density of the population of the coastal regions is evident. Along the coast of the Persian Gulf, traces of a later pre-Islamic occupation have been found on the islands off the coast of the Emirate Abu Dhabi and to the north, near Dubai at Jumeirah. There is also an occupation at Kush near the city of Ras al-Khaimah, as well as at Jazirat al-Ghanam, an island of the Musandam. But the major site for the last centuries before the Islam is without any doubt Sohar on the Oman Sea.[117]

The ceramic assemblage of Level IV is quite similar to the previous level, although some new distinctive types appear (e.g. *Fine Orange Painted Ware* imported from S-Iran). Additional clues for dating are an Egyptian jar and several lamps that seem to be of African origin. The

[114] Benoist, Mouton & Schiettecatte, 2003: 60-64.
[115] Benoist, Mokaddem & Mouton, 1994: 14.
[116] Mouton, 1999b: 26-27; Benoist, Mouton & Schiettecatte, 2003: 72; Callot, 2004: 145-146.
[117] Mouton, 1999b: 28-29.

time of abandonment of Mleiha still remains to be established, but considering the total absence of any Sasanian material dating to the 5th and 6th c AD, a date during the 3rd or 4th c AD is possible.[118]

The local SE-Arabian coinage disappears in this period, and only some very debased examples are known. Sasanian coins are found for the first time, however still in limited amounts. Mleiha lost its position as a center of regional exchanges with the interior of Arabia to the new sea-ports under Sasanian influence. It is the disappearance of the coinage that could clarify the reason for its appearance in the first place. The small state of Mleiha was for more than four centuries one of the links in the caravan network of pre-Islamic Arabia. The coins that were minted are not found outside the Oman Peninsula. This is important, because it is a clear sign that these issues were not intended for international trade, but rather for local or regional commerce. This regional character partly explains why the coins were minted only in metals of low value since this currency was intended to circulate only within a limited area.[119]

[118] Mouton, 1999b: 19.
[119] Callot, 2004: 147-148.

2.2.2. Ed-Dur (Umm al-Qaiwain, U.A.E.)

2.2.2.1. Geographical context

The site of ed-Dur (which means "the houses" in Arabic) is situated on the west coast of the Oman Peninsula, United Arab Emirates (Emirate of Umm al-Qaiwain; 25°33' N – 55°35' E) near the sheltered lagoon of Khor al Beidah. This is the only large coastal site identified so far with a main occupation from the 1st c BC until the 1st half of the 2nd c AD between Qatar and the straits of Hormuz. The shallow lagoon of Khor al-Beidah contained several islands, some with mangrove, and is protected by the narrow peninsula (where the modern town of Umm al-Qaiwain is situated) and the island of As Siniyyah. The lagoon may have been dry during the 1st c AD[120]. The site is positioned behind a high dune, several meters high (*ca*. 14 to 18 m above sea level) by which it was hidden from foreigners, The dune also protected ed-Dur from the NW-winds coming from the sea and responsible for the parallel running NW-SE sand dunes which cover this part of SE-Arabia. Ed-Dur lies in a flatter area (mainly between 7 to 9 m above sea level) and extends to about 1 km inland. During its heyday the site witnessed an occupation spread over some 2 to 3 square km, if not more. However the real extend of the site is difficult to judge as sherds were found over a vast area.[121]

2.2.2.2. History of research

An Iraqi team discovered the site of ed-Dur in 1973 during an archaeological survey of the United Arab Emirates and conducted a sounding. Preliminary reports were published on the limited excavation of a small square fort with round corner towers built of locally available beach rock (*farush*, a conglomerate of shells and sediment found in the lagoon).[122] The surface collections of ceramics and coins were the subject of two studies by J.-F. Salles[123]. These preliminary investigations highlighted the presence of 1st c – 2nd c AD Characene coins, glazed Parthian pottery and Roman *millefiori* glass, rare sherds of *Eastern Sigillatta C* and local coin issues representing barbarised Alexander imitations bearing the name of a ruler, Abi'el in Aramaic at the site[124].

In 1986 the site was threatened by local development and the construction of a new airport. After a two-week reconnaissance in late October – begin November 1986, a European consortium of four countries (Belgium, Denmark, France and Great Britain) was created to conduct full-scale excavations. These teams were respectively headed by E. Haerinck (Ghent University), D.T. Potts (University of Copenhagen), R. Boucharlat and O. Lecompte (Lyon/CNRS), and C. Phillips (University of Edinburgh). Based on a rotating system the first team set out in 1987. Over time different sites caught the attention of the other teams and eventually only the Belgian group remained. They conducted nine continuous seasons at ed-Dur between 1987 and 1995, with eight excavation campaigns and a final study season.[125]

In the frame of the final excavation reports already two volumes were published. The first volume by D. Whitehouse[126] discusses the glass vessels found by the Belgian team. The second volume, by E. Haerinck[127], deals with the tombs and their content. Still to come are a volume on the pottery by K. Rutten of which the study was recently finalised in a PhD-dissertation[128], a volume on the religious and secular architecture (temple, altars, wells, houses, etc.) and a volume on the small finds in general[129].

[120] Potts, 1990b: 274.
[121] Haerinck, 2001: 3-5.
[122] Salman, 1972; al-Qaisy, 1975.
[123] Salles, 1980b; Salles, 1984.
[124] Boucharlat, Haerinck, Phillips & Potts, 1988: 1; Haerinck, Phillips, Potts & Stevens, 1993: 183.
[125] For full referencing of preliminary excavations reports see Haerinck, 2001.
[126] Whitehouse, 1998.
[127] Haerinck, 2001.
[128] Rutten, 2006.
[129] The small finds are currently under study by A. De Waele.

Because of the fact that different teams were working on the same site, a (more or less) uniform registration system was implemented, this was to simplify the exchange and comparison of data. For the sake of clarity I will briefly describe the registration system used during the excavation. An *Area* refers to the excavations carried out in a particular location and is referred to by one or two letter(s). Architectural features within each area are allocated a *Locus* number. Religious and secular architecture are given a *L*. locus, standing for location, while a grave is a *T.* or *G* locus (respectively *Tombe or Grave*) and a *F. locus* (*Fosse*) for a pit. If a location is more complex (e.g. a number of rooms), then each separate entity (e.g. every room) is given another L. locus. In some cases individual walls are given an *M.* locus (*Mur*). Each layer or context is given a *UF number*. Small finds are registered by a number preceded by the area code. The same system is used for the samples, but there an additional *s* is added in front of the area code.

2.2.2.3. Archaeological context of ed-Dur in another nutshell

The main occupation of ed-Dur, and the phase under consideration here, dates to the late 1st c BC till the 1st half of the 2nd c AD, often referred to as the *'Hellenistic period'* or the *'ed-Dur period'*, there are however traces of earlier occupation.

• **Earliest periods** (5th millennium BC – 1300 BC)
The presence of Ubaid material (5th – 4th millennium BC) points to the occupation of the dune belt during this period and is materialized by some silex arrowheads. In the southern part of the site Umm an-Nar-type pottery and stone vessel fragments have been located, dating to 2700 – 2000 BC. Scattered finds of chipped stone, chlorite or steatite vessel fragments (dated from the Wadi Suq period to the Iron Age, 2000 – 1300 BC), and pottery are also attested in the central area of the site, amongst the later occupation. These finds point to the continuous but probably scattered occupation of ed-Dur since pre-historic times.[130] Along the crest of the lagoon itself there are shell-mounds of variable ages.

• **Iron Age** (1300 – 300 BC)
In 1982 an area of Iron Age occupation of uncertain size was located in the northern part of the site. At this time, two characteristic types of Iron Age pottery were recovered. Most of the material collected in 1986 came from this area, estimated to cover more than one hectare. This area was later excavated by the British team but still awaits full publication. Scattered Iron Age sherds were also picked up around the perimeter and the central area of the site. These sherds can be dated between 1300 and 400/300 BC.[131] Although there is evidence of earlier activity, the site of ed-Dur was deserted afterwards, probably for some centuries.

• **Main occupation: PIR C and D** (late 1st c BC – 1st half 2nd c AD/3rd c AD)
The site was reoccupied during the last decades of the 1st century BC and there is no evidence of PIR A and B occupation. The settlement must have profited from its position, particularly as the coast itself between Umm al-Qaiwain and Dubai is renowned for its dangerous shoals and reefs. The proximity of the lagoon provided possibilities for fishing and collecting shellfish, while the sea allowed fishing in open sea and international commercial traffic. On the other hand ed-Dur would have enjoyed communication with the interior via traditional routes leading to the fertile inland plain of al-Madam. There the other important pre-Islamic site of Mleiha is located, where several routes from the south converge[132]. Camels could cover the distance of approximately 70 km between these two sites, in one or one and a half days travel. The possible function and position of ed-Dur will be discussed later. Ed-Dur, but also Mleiha, probably ceased to be of any importance by the middle of the 2nd c AD, if not earlier. The later occupation (PIR D) at both sites is probably only of

[130] Boucharlat, Haerinck, Phillips & Potts, 1988: 2-3.
[131] Boucharlat, Haerinck, Phillips & Potts, 1988: 3.
[132] Boucharlat, Haerinck, Phillips & Potts, 1988: 2.

squatters, who mainly made use of the ruins of the building in Area F to bury their dead and to shelter.[133]

Ed-Dur does not exhibit a concentration of building remains within a fairly restricted area, nor does it have any blocks of houses separated by streets, alleys or open areas. Rather it has the character of an extensive, sandy area of isolated building-ruins set amidst a more or less continuous surface scatter of shell, sherds and stone rubble. Houses vary in size from small one- or two-room dwellings (e.g. Areas B, O, P) to substantial buildings of four rooms or more (Areas E, F, Z).[134] Around the fort is a grouping of buildings, but the excavations conducted there by C. Phillips still await full publication.

I will not give a complete overview of all architectural remains found at ed-Dur, only mention the more important ones. When necessary the context will be sketched further on.

Secular architecture
A small roughly square fort or fortified house (22 x 25 m) with round corner towers dominates the site. It was built of dressed beach rock or *farush*. The round towers point to a Parthian date at the earliest, since square towers seem to have been the rule before. It was first sounded by the Iraqi team in 1973, but was reinvestigated by the British team, bringing to light the remains of an earlier building underneath. The walls survive to a height of approximately 1 m, and it is likely that the superstructure was constructed of mudbrick. Postholes indicate the presence of substantial structures built of organic material. Evidence of ovens and burnt animal bone deposits indicates that domestic activities were undertaken in the courtyard of the fort.[135] On the south side of the fort a large contemporaneous building was excavated. To the east of this building a large room was found filled entirely with ash, some oven-like structures and a lot of bone material. On the east side a previously investigated building by the Iraqi team was re-excavated and turned out to be a tomb with a *dromos*-type entrance (Area K).[136]

Areas of domestic housing have been recovered at ed-Dur. Excavations indicate that the majority of houses were widely separated from each other, although a denser cluster was noted in the vicinity of the fort[137]. The majority of the living quarters probably must have consisted of *barasti* (palm branches) dwellings, leaving little material traces apart from numerous fire-places, animal and fish bones, some plaster floors, etc. Nevertheless some simple and more elaborate houses made of beach rock and more rarely of mudbrick, were encountered. In Area C (excavated by the Danish) a complex building (30 x 15 m) with four rooms was unearthed of which the walls might have been completely plastered (remains on the base). The most unusual aspects are the exterior, semi-circular buttresses on the eastern, western and southern wall. This building is more architecturally decorated than the other buildings and might have been the residence of an elite family.[138]

In Area F a stone building was investigated by the French team, situated on the highest point of the large dune that separates ed-Dur from the lagoon. This square building has sides of about 25 m, with round towers on three corners and is organised around a central courtyard with a monumental entrance to the east. Although the building that it dominates the surrounding has a defensive appearance, the walls are not very thick if compared to the fort. Worth mentioning are the two stone eagles statues found at this entrance, paralleling examples from Hatra. In this courtyard a burial was excavated with next to it a sacrificed dromedary. In front of the entrance a second camel burial is probably also related to this

[133] Haerinck, 1998c: 24.
[134] Potts, 1990b: 274-277.
[135] Mouton, 1992: 90; Weeks, 2004a: 240.
[136] Unpublished information.
[137] Weeks, 2004a: 240.
[138] Boucharlat, Haerinck, Lecomte, Potts, & Stevens, 1989: 15-20; Potts, 1990b: 277.

tomb. This could give the status of a mausoleum to the structure. In front of the building some hearths were encountered, some containing the remains of a meal (luxury ceramics, glass, knife, bone, etc.). After this initial occupation the building seems to have been left and covered by a layer of sand. In a next phase simple intrusive burial pits were dug in and around the building (58 were excavated) and the burials date to the late PIR D/Sasanian period. It is not clear whether the small hearths mentioned are related to these interments or date to the previous occupation.[139]

Based on the architectural style and the material found O. Lecomte initially dated the complex to between 225 AD and the end of the 3rd c AD[140]. The ceramics however are only indirectly linked to the building, since they were found on the walking surface in front of the building (the so called *zone de rejet*) and in several pits with the remains of burned funeral meals. According to K. Rutten[141], who reanalyses the ceramic remains, a clear division should however be made between the *in situ* ceramics coming from the funerary pits and the sherds from the floors within and especially in front of the building. The pit material can be dated to the 3rd c AD, based on parallels on other sites. The floor-material has parallels within Parthian and Sasanian assemblages, but the most typical late 2nd c AD ceramic types are absent. Most have clear *comparanda* within the ed-Dur assemblage and are without doubt contemporaneous. This material is only partly dating to the 3rd c AD, but has a fair amount of residual 1st/2nd c AD sherds.

Two hypothetic functions can be proposed for this building. It could have had a military function, protecting the south side of the site and offering a lookout over the ships that possibly harboured in the lagoon. But this is however partly contradicted by the relatively thin walls. A second possibility is an economic function as a storehouse. Whatever the original purpose, the structure was reused for the central burial and again deserted. In a last phase, after the building was largely destroyed it was used as a cemetery and some tombs were cut through the walls.

Important to notice are also several deep, circular or square stone-built wells that provided the water for the inhabitants of ed-Dur.

Religious architecture

The excavations at ed-Dur revealed the presence of a square temple (Area M), most probably dedicated to the Semitic sun-god Shams/Shamash. The temple at ed-Dur is unique in the fact that it is the only sanctuary known from this period in SE-Arabia and it differs considerably from sanctuaries in S-Arabia. It was build with care, is oriented E-W and measures 8 x 8,3 m. It was well preserved due to the encasement by a dune. There are two entrances, the main one on the E-side, and a secondary one in the opposing wall. The walls were build in beach rock, but afterwards they were covered with a layer of gypsum and lines were drawn on the exterior to give the impression of well cut ashlars. Around the doors and on other places decorative elements were added and on some places traces of ochre colour is preserved. The whole was built on a protruding plinth.

Outside in front of the main entrance, a plastered low podium has a square stone with a slight depression adjoined to the eastern edge. This stone shows signs of intense burning. A small canal runs under the plinth of the building. Important finds include a Roman bronze oil-lamp, a bronze pedestal with a male bust in relief, a short Aramaic inscription and a limestone sculpture of a bird. Also outside the temple four structures interpreted as alters have been excavated. One of these altars was topped by a stone basin that had an Aramaic inscription of seven lines on the front and three lines on the side. The exact content is not yet known, but the sun god Shamash is mentioned in the longest inscription. Therefore it is very

[139] Boucharlat, Haerinck, Lecomte, Potts, & Stevens, 1989: 29-50; Mouton, 1992: 126.
[140] Boucharlat, Haerinck, Lecomte, Potts, & Stevens, 1989: 29-56; Lecomte, 1993: 195-203.
[141] Rutten, 2006: 141-142.

possible that this sanctuary was dedicated to this god, an idea further supported by the E-W orientation of the entrances. To the NW-corner of the temple a round stone lined well was situated. Several traces of large fires were observed around the temple.[142]

Funeral architecture
Tombs are by fare the most numerous architectural remains. At ed-Dur the graves occur all over the site, which indicates that the living camped amongst the buried. This is in contrast to Mleiha were the tombs are concentrated in certain areas of the site. A lot of effort was invested to built the tombs in stone. Several types can be distinguished going from the individual, rectangular cists to large semi-subterranean multiple interment tombs covered by a barrel vault and accessible via an entrance, occasionally with stairs. This energy input is in sharp contrast to the remains of domestic structures. The tomb chambers were built with smooth wall faces on the interior. In general the more elaborate tombs have some resemblance to the Parthian tombs as found at Assur (N-Mesopotamia).[143]

Burials belong to all age groups, with both female and male interments, without differentiation and without specific rules related to gender and age. Probably most tombs were visible on the surface but there were no surviving markers. From the 121 tombs excavated only 14 were not plundered (Table 2).

Type	***Description***	***#***
Type A	Simple pit burial, the place was indicated above the body of a child by 2 standing stones.	1
Type B	Simple burials (children) covered with pottery fragments.	2
Type C	Tombs delineated by some stones or by one wall only.	2
Type D	Rectangular cist graves with stones set up on their short side and covered by capstones, for newborn or very young children.	9
Type E	Rectangular cist graves constructed of horizontal layers of stone, covered by large capstones. There is quite a lot of variation in size. Most are single inhumations.	99
Type F	Rectangular cist graves made of horizontal layers of stone with a door and covered capstones. Similar in structure to type E, but they had a door.	2
Type G	Rectangular cist graves made of horizontal layers of stone, with door and entrance passage and covered by capstones. Similar to type F, but with additional entrance passage. The French and British team also excavated this type of tomb.	2
Type H	Rectangular cist grave made of horizontal layers of stone, with door and entrance passage and covered by capstones. Related to Type F and G, but had a stepped entrance.	1
Type I	Large tombs with stepped entrance and regular vaulted chamber. The Danish and British team also excavated similar tombs.	2
Type J	Large rectangular above-ground tower-like burial chamber with door (Area N). This was a freestanding monument.	1

Table 2: Overview of the types of tombs attested.

Two tombs stand out from the rest, i.e. G 5156 in area AV and G 3831 in area N. Area AV appeared as a very low mound with some stones on the surface. The main structure discovered turned out to be a large, semi-subterranean, vaulted tomb (G 5156). The tomb must have escaped the attention of the looters, since it was found still largely intact with only some stones of the vault missing. The entire grave included an outer enclosure, had a length of 9,5 m and was oriented N-S. It had no stairway leading to the chamber however. The skeletal remains were found all over the floor of the tomb and belonged to a minimum of 27 individuals. Many objects were encountered throughout the grave, the majority being at the

[142] Haerinck, 1990: 15; Haerinck, Metdepenninghen & Stevens, 1991: 33-34; Haerinck, Metdepenninghen & Stevens, 1992: 45.
[143] Haerinck, 2001: 7-15.

back of the tomb, where they were found undisturbed and complete. The frequent discolorations in the sand point to the fact that many perishable materials and foods were joining the dead. On the outside, next to the entrance a more or less square annex construction was excavated, that maybe can be seen as an altar attached to the tomb. The only evidence for PIR D occupation encountered by the Belgian team was an intrusive female burial (UF 4272) in the large tomb. Further a small plundered grave and two sacrificed camelids (UF 5527) were excavated in the vicinity.[144]

Area N was chosen because it showed a small circular mound. Four small plundered tombs were excavated in 1987 and in 1988 an additional 13 tombs were opened. In the middle of this area stood a large rectangular tomb (G.3831, 6 x 5,4 m) with a paved flour, built on a plinth. This tomb has thick walls and was a freestanding monument, with an entrance to the south, facing the temple. The large tomb contained at least two individuals (one man and one woman) and although it was plundered a fair amount of objects was still retrieved. Among them some 20 decorated bone/ivory plaques (with incised humans, lions and circle-dot decoration), bone reinforcement lathes of a bow, spindle whorls, a silver obol, an oval shaped agate intaglio, beads, some large iron nails and some fragments of glass.[145] The large tomb differs of all the others at ed-Dur since it was not a subterranean or semi-subterranean grave. In this respect it is related to the earlier monumental tombs at the inland site of Mleiha, which showed a solid tower-like upper structure of mudbrick, but with a subterranean burial chamber. The presence of crenellations (although not proven to be related to G.3831) further strengthens the correlation with Mleiha, where they were used as decoration on the tombs[146]. In 1989 two additional tombs were localized, bringing the total on 19 for this area. Area N is the only area where we can speak of a possible concentration of tombs around a monumental aboveground burial.

On the Island of Ghallah (SW of the lagoon in front of ed-Dur) 26 tombs were discovered showing that the occupation traces are not only restricted to the site of ed-Dur[147].

2.2.2.4. Function & thoughts on the of the site

- **Bedouin nature**

To fully understand the nature of 'habitation sites' in SE-Arabia, we must be aware of the influence of the Bedouin nature of its occupants. This will not be elaborated here since it is to far from the topic at hand. Some basic thought can be put forward however based on ethnographic research in Ja'alan, an area of the SE-coast of Oman.[148]

Local inhabitants of this coast say that not one area or resource is able to sustain a livelihood over the year. People therefore have to depend on a multitude of resources and their mobility. Fishing and herding gives a continue source of food and provides surpluses which are processed for storage or can be traded. The boats and animals also enable them to provide transport services and to trade surpluses. This system to survive in a harsh environment is done without a central government, but is embedded in social infrastructures based on a shared moral background. This system can be found over the wider Arabian Peninsula and over a long span of time. Bedouin tribal identity works through descent in the male line. Their social processes are based on the grounds of juridical equality before god and individual autonomy. This premise implies that access to resources, products from them and distribution of these are available to all participants. Relationships arising from these activities are essentially symmetrical and cannot give rise to patron-client relationships nor to stratification. Families of herders do move to the coast and spend a winter fishing with their

[144] Haerinck, Metdepenninghen & Stevens, 1991: 53-58; Haerinck, 1992: 191-201; Haerinck, 2001: 41.
[145] Haerinck, Metdepenninghen & Stevens, 1991: 40-45; Haerinck, Metdepenninghen & Stevens, 1992: 49-50
[146] Haerinck, 2001: 29.
[147] Boucharlat, Haerinck, Lecomte, Potts, & Stevens, 1989: 8; Lecomte, 1990: 14.
[148] Lancaster & Lancaster, 1996: 141-145.

own boat. Formerly the fish were dried or salted and carried to the interior or traded to India. Coastal Bedouin were and are active in both local and inter-regional markets and trading.[149]

- **Religious**

An interesting side remark towards the function of ed-Dur can be made concerning the presence of a 'temple' at the site. During a lecture K. Damgaard[150] mentioned the importance of the Ka'ba to Mecca. The city became the first of all Islamic holly places due to the presence of this 'temple'-like building. Mecca was not a large trading place or an important political centre as such in the early Islamic period, but derived its importance from this shrine. Maybe a similar interpretation (not of the same magnitude of coarse) can be proposed for the temple of ed-Dur. After all this is the only structure in SE-Arabia clearly identified as a religious building. At the larger and more complex site of Mleiha no temple structure was identified so far, making the ed-Dur structure even more significant. The care taken to build and decorate the temple shows the importance that was attributed to. Importance to the local community in the first place but it is not inconceivable that it stretched over a wider area. The presence of a religious building could have attracted pilgrims or be an attracting pole for annual fairs in the vicinity. It could also explain the (till now) almost complete absence of tombs from PIR C at Mleiha, maybe people were buried more closely to their holly place at that time. This will not be explored any further here since it is not part of this PhD and suitable sources to do so do not directly spring to mind. Still it is an interesting experiment of thought.

- **Pearling**

The growing body of evidence suggests a sea-oriented society before Islam in SE-Arabia and thereafter, and this raises the question of the role of the islands of the Gulf, including the large number off the Abu Dhabi Coast[151]. The pearl-banks of Ceylon and the Gulf were for long the main sources of natural pearls for the old world and SE-Arabia was involved in diving and traded pearls as mentioned in the *Periplus*. Further evidence can be found in almost all musea on the local history of the U.A.E. that incorporate an exhibit on pearl diving in the recent past[152]. Pliny also informs us that the best pearls came from the Arabian side of the Gulf.[153] Pearls and shells of large oysters were found at ed-Dur, as well as a bell-shaped lead weight identical to the stone weights used by recent pearl divers. The recovery of large pearl-oyster shells in and in the vicinity of the tombs at ed-Dur also suggests that the pearl was a valued commodity and stressing the importance of these sea products for the community.[154]

Generally speaking fishing is confined inshore and is a seasonal occupation of the winter months. Pearling on the other hand, is a summer occupation. Yet the two do not fully complement each other, partly because the pearl-banks are situated in different areas from the richest fishing grounds, and partly because pearling is organized on commercial lines, whereas fishing tends to form part of the subsistence economy.[155] The main concentration of the pearl-banks in the Gulf are in the shallow waters on the Arabian coast. Here the principal pearling-banks are located on either side of the Qatar Peninsula; those to the north-west include the famous Bahrain fisheries, while those of the Lower Gulf are enclosed by a line drawn from the tip of the Peninsula to a point just west of Dubai (so well to the SW of ed-Dur).[156]

[149] Lancaster & Lancaster, 1996: 141-145.
[150] K. Damgaard was the speaker on the archaeological research afternoon of 13-03-2007 and presented a lecture on 'Early Islamic Settlements in Jordan'.
[151] King & Tonghini, 1998: 119.
[152] Wilkinson, 1977: 20.
[153] Haerinck, 1998a: 274.
[154] Potts, 1997c: 92-93.
[155] Wilkinson, 1977: 19.
[156] Wilkinson, 1977: 20.

• **Gathering place**

There is ethnographic data from Islamic sources that there were co-operative markets that took place under the protection of certain clans, on religious places or during festivities or religious events. There is a real possibility that this kind of gatherings also took place at ed-Dur. The absence of a large living and agricultural areas makes ed-Dur ideally suited to receive a large group of people at once. A religious connotation can be found in the presence of the temple. The coins can be seen as the expression of a political identity and power within the region, providing safety for the market. Studies on the traditional trade in Oman show that two different economic systems were operating. The trade of mainly agricultural goods between villages in the Hajjar Mountains exists next to an interregional trade that specializes on local goods that were suited for export (pearls, dried fish and fruits, dromedaries, etc.) that was situated on the coastal cities. This situation is largely mirrored by the situation at ed-Dur where we see import of local goods and of foreign products, a local exchange and redistribution of both, and the presence of local and foreign coins.[157]

• **Port/marketplace of trade & identifying *Omana***

The Gulf has always been a major route by which (distant) goods from different origins travel. Commercial activities in this sea contributed without doubt to international contacts and exchange of commodities between different populations and nations. Since ed-Dur is located at the seaside it was possibly involved in this international trade network. It remains a mystery if the site is to be interpreted as a marketplace or has to be seen as a port of transhipment. The site could also be a terminus, where no real trade took place but from where goods were further distributed in the hinterland, within a local commercial system. In any case the vast amount of foreign objects found on the site shows that the local population was rather wealthy and could acquire foreign goods from different sources and regions.[158]

M. Mouton states that care should be taken in asserting 'wealth' to the inhabitants of ed-Dur. He says that the wealth of the objects recovered at ed-Dur is mainly the result of the intensive archaeological work, concentrated on the excavation of tombs, where luxury objects were preserved. A much smaller number of tombs were excavated in Mleiha, hence the difference in 'rich finds'. The large number of imported objects found at ed-Dur is therefore no proof that the site actively participated in any sea trade, but rather stresses the continuity of the long distance trade.[159] This comment of M. Mouton is not entirely correct, in that most tombs of ed-Dur were plundered and that most objects come from the few tombs that were more or less intact. So the large amount of funeral deposits is not due to the number of tombs excavated, but due to the presence of some rich tombs. That this is not conclusive proof of active participation in the trade can be correct on the other hand, but the large amounts of S-Mesopotamian transport ceramics are hard to explain if they did not arrive at ed-Dur by boat. The shapes of these ceramic transport *amphorae* (pointed base) are more typical for shipping than land trade (also see *Chapter 3*: xx).[160]

Certain signs also point to a close relation with the desert and caravan trade, e.g. the custom of sacrificing and burying camelids when certain (important?) people died. Whatever the nature of the trade going on, ed-Dur and Mleiha were closely linked, both culturally and economically, and they were in all probability politically connected too, considering the homogeneity of the coinage series found in both sites.[161] The fact that ed-Dur was reoccupied after centuries, was probably organised from Mleiha. Trade on a local level in that ed-Dur provided sea products and mangrove wood to Mleiha and that Mleiha supplied ed-Dur with cereals, dates, palm wood, raw products from the inland, etc. is undisputed. Both sites were interdependent and the archaeological remains of local commerce are

[157] Rutten, 2006: 416-418, referring to Potts, 1990b: 339-340 and Hoyland, 2001: 109-110.
[158] Haerinck, 1998a: 274.
[159] Mouton, 1999b: 25-26.
[160] Rutten, 2006: 420 & 428-429.
[161] Mouton, 1999b: 25-26.

materialised by minting of local coinage. Regular contact was not a real problem since a camel can cover the distance of some 70 km between both sites in one long day or more likely in one and a half days of travel.[162]

The presence of potable water at the site, attested by the presence of some stone build wells, should not be neglected in this discussion. The bunkering of drinking water was very important from sailing merchants, especially those passing along arid coasts such as those of the Arabian side of the Gulf. The site was probably a (major) stopping place between Characene and NW-India for taking on fresh water and other supplies. Another supply point is most likely to be found on Bahrain.[163] Based on the presence of large quantities of S-Mesopotamian pottery it is also possible that Characene played a role in the reoccupation of ed-Dur. Contacts between Mleiha and NE-Arabia are already attested in the 3rd c BC and it is very possible that the Characenean traders in the 1st c BC extended these. Maybe Characene wanted to bypass NE-Arabia and at the same time saw the opportunity to extend its influence and trade further to the East, even maybe beyond the Straits of Hormuz. This can be seen in the light of and as the result of the increase in traffic in the Gulf and the Indian Ocean, linked to a higher demand from the West to obtain more goods from the East. For this purpose, and to achieve a direct link, the coastal site of ed-Dur could have been reoccupied. The Gulf must have been the scene of intensified maritime trade. Not only Western merchants sailed in the Indian Ocean, but also Arabs, Persians and Indians probably all participating in this lucrative business. In any case the position of ed-Dur at the mouth of the Gulf provided a stopping place for Characenean ships and a market for their products (local exchange and redistribution to S-Arabia and the Indian Subcontinent).[164]

The discussion to identify ed-Dur as Oman(n)a is a longstanding one and it will not be repeated here. A short overview will suffice[165]. Moreover new conclusive evidence to advocate for or against a positive assertion is not found within the frame of this research.

> "*After sailing by the mouth of the Gulf, six runs further on you come to another port of trade of Persis called Omana. Customarily the merchants of Barygaza deal with it, sending out big vessels to both of Persis's ports of trade* [Apologos and Omana] *with supplies of copper, teakwood, and beams, saplings, and logs of sissoo and ebony; Omana also takes frankincense from Kanê and sends out to Arabia its local sewn boats … Both ports of trade export to Barygaza and Arabian pearls in quantity but inferior to the Indian; purple cloth; native clothing; wine; dates in quantity; gold; slaves.*"[166]

The first literary documents talking about and mentioning people and places in SE-Arabia date to the 1st c AD, e.g. Pliny's *Natural History*, the anonymous *Periplus of the Erythraean Sea* and the 2nd c AD map of Arabia by Ptolemy. The area of the U.A.E. seemed to have been full of settlements, tribes, and physical features, the names of which Ptolemy recorded. Connecting these literary place names to actual sites has proven difficult however. Ed-Dur is one of the several places proposed as the location of ancient *Omana*. Certainly the archaeological remains of ed-Dur leave no doubt that the site was the most important coastal settlement in the lower Gulf during the 1st c AD, but to link both remains speculative.[167] L. Casson is of the opinion that Omana is not situated in the Gulf but on the southern coast of Iran, since the Periplus states says "sailing by the mouth of the Gulf"[168].

[162] Haerinck, 1998c: 27.
[163] Haerinck, 1998a: 274; Haerinck, 2002: 42.
[164] Haerinck, 1998c: 25-26; Haerinck, 2003: 200; Rutten, 2006: 429.
[165] A detailed discussion of the pro's and contra's is given by D.T. Potts (1990b: 305-310). He plies for a positive correlation.
[166] Casson, 1989: 73, chapter 36 in the translation of the *Periplus*.
[167] Potts, 1997: 61-62.
[168] Casson, 1989: 19 & 180-181.

M. Mouton sums up several arguments that advocate against an identification of ed-Dur as Omana[169]:

- There is no indication of a harbour function or even a marine oriented organisation at ed-Dur.
- The most 'powerful' people of ed-Dur were buried with their camel, which indicates a clear link to the desert.
- The material remains do not show a link with commerce when compared to the assemblage of a true commercial centre such as Sohar.
- Charancene money show contact with Charax, who are not very maritime, and the NE-Arabian coins also point more towards caravan trade and contacts with the Arabian neighbours. Most of the coins are from copper-base alloys, so not very useful for trade where silver coinage was used. These foreign coins attested can be the result of 'travelling' (~ caravan) rather than 'commerce'.

Although I do not whish to advocate in favour of an identification of ed-Dur as Omana, some counter arguments can be brought up:

- Harbour installations do not need to be complex (see *Chapter 3*). The absence of any permanent docking facilities is certainly not a premise for an important role as a port.
- That there was still a link to the possible desert origin of the people living at ed-Dur is not surprising. If goods arrived at ed-Dur they had to be dispersed and the only way to do that is by camel in this arid environment. The political and cultural link to Mleiha, that is certainly desert oriented, can be used to underpin that. It is not a 'because … then' equation. It is not *because* camel remains are present that the community was desert oriented.
- The comparison to Sohar, a true port, is not completely justified since many different levels and types of harbour and ports existed.
- The local SE-Arabian coinage was probably used for inter- and intra site commerce and the fact that all coins contain at least some silver shows the metal had an intrinsic value. The local coins are not attested outside the SE-Arabia and should indeed be placed within the frame of local trade. The foreign coins are indeed most probably present as objects without a real monetary value (all in all few coins from one area occur and they all are of small nomination) and the result of contact. The trade that went on with territories further away most probably was more like barter trade than a full monetary trade.

All we can say is that the inhabitants of ed-Dur had access (directly or indirectly) to a large variety of luxury foreign good from different places and that the involvement of the site in this trade network should at least be considered.

- **Human bones**

P. Stone[170] studied a selection of human bones found at ed-Dur, those from tomb G 5156 (Area AV), and J. Littleton[171] studied another part of the collection. The conclusions of P. Stone can be summarised as follows. The bones from did not show any sign of dietary stress, but the pathologies do suggest that people were working long hours at labour intensive activities from childhood. Pathologies indicate trauma by prolonged habitual kneeling on the toes. This suggests constant kneeling practices, which could be the result of work related to activities associated with grinding grains, or could be due to squatting for other tasks, and even socializing. Other stress symptoms (even at young age) are the result of different activities that utilize the head and neck, by carrying heavy loads on their head supported. Other show symptoms related to occupational stressors, which involve habitual activities that utilize the upper body, such as carrying, or pulling, of heavy loads on or by the

[169] Mouton, 1992: 257-258.
[170] Stone, 2001.
[171] Littleton, 2005.

upper body/shoulder area from early childhood into adulthood. A last pathology that has remained somewhat of an enigma is a specific deformation on the feet of some of the skeletons. Similar traumas can however be attested on many soldiers and some athletes. When stress is placed on the feet this can result in fractures of various bones of the foot. These stress fractures are referred to as 'marchers fractures'.[172]

The findings of J. Littleton indicate that the bone inventory contains the remains of all ages and both sexes. This argues against the idea that ed-Dur was a settlement with a high proportion of immigrant males. Not everyone had lost teeth before they died. This is in striking contrast to remains of the same date from Bahrain where nearly every adult had lost teeth before they died. Two possible explanations are offered. The first is that the two, i.e. ed-Dur *versus* Bahraini, populations differed in the amount of reliance upon carbohydrates. The second being that the rate of dental wear at ed-Dur is higher so that even when dental decay developed it was removed by the grit in the died. A further factor that needs to be considered is the extent of seasonal variation in diet that may contribute to both caries and more dental wear. J. Littleton did however attest at least two individuals that had experienced multiple episodes of growth arrest during their lifetime. Nevertheless the overall population clearly had access to abundant food resources.[173]

What can be deduced from these findings? It is unfortunately that no association was made between the pathologies and the sex of the individuals of the analysed bones. We don't know if some are more often present with male or female individuals. This is due to the small amount of bones analysed and the fragmented state of preservation and storage of the bones.

There were no indications towards injuries obtained through fighting, although the tomb had a lot of weapons included.

The pathologies related to carrying can be related to the carrying of products from boots mooring in the bay to site or the transport of rather heavy loads within the site. Pathologies indicative of pulling can maybe be situated within the same context: pulling boots out of the water, rowing, pulling in fishnets (or maybe even from shooting a bow?). That the community was involved in fishing activities is certain, exemplified by the presence of many fish remains, 'netsinkers' or 'line weights' (both ceramic and from lead), some anchors and some iron fishing hooks. Pathologies symptomatic for prolonged habitual kneeling show that food was probably processed on-site. Unprocessed cereals might have been imported on the site or dried fish might have been processed to fish flour for internal use or export, hence the presence of grindstones at ed-Dur. The 'marchers feet' syndrome can be related to walking long distances in the desert (e.g. ed-Dur to Mleiha and back). Camelids were known and used at that time, but rather then a riding animal the 'ships of the desert' was an animal of burden, used to transport heavy load over long distances in a dry climate. The absence of consistent growth stress symptoms and the good state of the dental remains show that shortage of food and/or a monotonous diet was not an issue.

The age and sex profile of the diseased is normal, in that the amount of male, female and child remains are in balance. This means that ed-Dur was a 'normal' settlement where a whole community was living and not a specialised site that was involved in specific activities that only requested part of the population. Examples of the latter could be seasonal fishing or temporal trading activities, which would most probably result in a higher fraction of the male population present. The presence of many burials and some permanent stone buildings, including a fort and a temple further reinforces the idea of a permanently inhabited core settlement, possible expanded with visitor at certain points of the years (fairs, etc.). The

[172] Stone, 2001: 57-61.
[173] Littleton, 2005: 5-7.

absence of any structure, e.g. streets, squares, etc., is not in contradiction with that because the much later residential places of the Emirs also did not have them. The absence of a structuralized town is a characteristic of Bedouin nature of its inhabitants. The 19th c AD photographs from the Emirate 'capitals' also only show a fort where the leader resided, some permanent stone building for the socially more important people, a mosque, surrounded by impermanent living place in perishable materials. A striking similarity with the remains found at ed-Dur, a central fort, some stone building, a temple, surrounded with the remains of impermanent *barasti*. The major difference is that in the Islamic period the graveyard was situated in a well defined area, whereas at ed-Dur the burials are located among the living area. At Mleiha the cemetery is more concentrated however.

Al this sketches a picture of a permanently inhabited settlement that was involved in a variety of activities (some probably seasonal) and had no shortage of food.

- **Piracy**

A last, rather romantic, hypothesis proposed by E. Haerinck is that of piracy. Although the open sea could be reached quite easily, the site was so well protected by the natural topography that it was impossible to see it from the sea and any trade or visitor would have to know the exact location. Could this very well chosen location indicate that the locals and foreign visitors had to protect themselves against enemies and bandits? Or were the inhabitants of ed-Dur themselves involved in piracy? Piracy existed in the Gulf and is in fact mentioned in several ancient texts. It justified the intervention of a Sasanian king in the Arabian coastal lands, but this happened 200 years after the first settlement at ed-Dur[174]. The coasts of SE-Arabia stayed the home base of pirates. A malice only rooted out in the 19th c AD when the English stabilised the region.

- **New people moving into the region**

According to the Arab chroniclers an important migration of the *Azd* tribe from S-Arabia took place at the beginning of our era. Their movement triggered a chain of movements across the Arabian Peninsula and also the northward displacement of some of the older, sedentary population of the region who previously inhabited the new territory of the Azd.[175] Based on the toponimic name change by the classical authors (Pliny *versus* Ptolemy) in NE-Arabia, D.T. Potts suggested that by the 1st c AD a new population had entered Arabia[176]. Although he does not treat the Oman Peninsula, the coincidence of the occupation of the site of ed-Dur at this period has to be mentioned. The penetration of a second group of Arabs in the Peninsula in the 1st c AD can be the origin of the settlement of ed-Dur, maybe a tribe pushed out by the Azd. The archaeology does not contradict this: the importance of the camel points to a desert oriented community connected with exchange. Moreover the settlement of ed-Dur recalls the first occupation of Mleiha: light constructions, few (mudbrick) stone buildings and well-constructed tombs. This similarity can of course also be used the other way around and makes it difficult to see it as the movement of a different group of people. At this time Mleiha seems to have been less densely populated, with some large dispersed buildings that were grouped around the fort. Ed-Dur however does not have the urban structures to justify a movement of the population from Mleiha to ed-Dur.[177]

- **Environmental causes of desertion**

The site of ed-Dur as a whole was largely abandoned by the end of the 1st c – beginning of the 2nd c AD, and completely deserted by the end of 3rd c AD. A possible lowering of the water table may have made the site less attractive or uninhabitable. Alternatively the small

[174] Mouton, 1999b: 25-26.
[175] Potts, 1990b: 197.
[176] Potts, 1990b: 226 & 325.
[177] Mouton, 1992: 287-289; Hoyland, 2001: 231.

inlet of the Khor al-Beida over which the site looks had perhaps become silted so that it was no longer suitable for commercial shipping, if it had ever been used as a port.[178]

• **Politic-economical causes of desertion of the site**

When the ceramic assemblage of ed-Dur is evaluated, a gradual but steady decline of the imported pottery can be observed from the late 1st c AD onwards. At the same time some of the houses excavated at Mleiha show traces of burning and M. Mouton links this to a possible entering of new tribes in this area. At ed-Dur the central fort is build with a concentration of stone building in the vicinity. This is in contrast with the more dispersed nature of stone buildings before. Based on the monetary devaluation of the Parthian coins at the end of the 1st c AD we can see an economic decline of the super power. A process accelerated by the Roman-Parthian wars of Trajan (between 114-116 AD). The destruction of many Parthian cities (Seleucia, Ctesiphon, Niniveh, etc.) and the many assaults of both camps weakened Parthian government. The situation of Characene is less clear. Apparently the king at the time (Attambelos) surrendered to Trajan in order to save his capital. But as soon as Trajan died the Characene king was replaced by the Parthians. After a period of recuperation the trade was reinstalled, but by now the Palmyrene traders take a dominant position, peaking in the second half of the 2nd c AD.[179]

Originally it was thought that also the site of Mleiha was abandoned in the 2nd c AD, but more recently the fort present there was dated to this period. At that time ed-Dur was already completely abandoned. Together with the revival of Mleiha there is a closer link to the archaeological material from the site of Sohar (Oman). The foundation of Sohar (in the late 2nd c AD) is connected with the rising of the Sasanian domination after they had overthrown the Parthians. The Sasanians had a much more centralised government and sought to actively control the Indian Ocean trade. The site of Sohar functioned as a new centre of trade to them.[180]

2.2.3. Summary

There is no doubt that ed-Dur and Mleiha were the main towns in the S-Gulf during the last decades of the 1st c BC and the 1st c AD. They were in close contact with each other and interdependent. Ed-Dur probably acted as a harbour and provider of food from the sea, while Mleiha was able to deliver agricultural products and other commodities, which were lacking at ed-Dur on the coast. It is quite remarkable that from the 3rd to the middle of the 1st c BC there was no harbour site along the whole coast between Qatar and the Straits of Hormuz even though, at Mleiha, foreign objects such as Greek black-glazed sherds, fragments and stamped handles of Rhodian *amphorae*, glazed pottery, and moulded glass were imported. These goods could only have reached the site by means of camel caravan, coming from NE-Arabia or less likely S-Arabia. The locally minted coins are indicative for the fact that SE-Arabia wanted to express itself as an independent political and economical entity. In the second half of the 1st c BC the coastal site of ed-Dur was re-occupied. It is clear that SE-Arabia replaced the NE-Arabian middlemen, who had been providing foreign commodities from the 3rd to the middle of the 1st c BC. SE-Arabia now more actively participated in the Gulf trade, what does not mean that the importance of camel trade should be neglected. A re-occupation of ed-Dur can also be seen against the background the growing power of the Kingdom of Characene. The Characenean merchants possibly wanted to by-pass NE-Arabia and at the same time extend their influence and trade further to the east. This can be seen in the light of an increased traffic in the Gulf and Indian Ocean, linked in turn to a higher demand in the West for goods from the East.[181]

[178] Hellyer, 1998: 112.
[179] Rutten, 2006: 434-435.
[180] Rutten, 2006: 434-435.
[181] Haerinck, 2003: 199-200.

2.3. SE-Arabia – Sultanate of Oman[182]

• **The inland Samad culture**

Omani history really begins with an account of how the first Arab tribes came to the area. It tells of the way clans of the *Malik b. Fahm Azd*, accompanied by some so-called Quda'a groups, left Sarat and the Tihama (in SW-Arabia) and migrated along the fringes of S-Arabia to arrive in Oman. This story forms a part of the legend of the Azd diasporas which began when the Sayl al-Aram, the flood which reputedly burst the Marib dam, caused the Azd to move away by major genealogical groupings from their homeland. They first moved into W-Arabia and then, in the course of time, further afield into the Arabian Peninsula until they reached the fringes of the Fertile Crescent. Now the Marib part of the story can be proven wrong. There does however appear to have been a period of active, though periodic, migration of Arab tribes away from SW-Arabia which started roughly at the time when the old civilizations associated with the Marib dam were collapsing, and it lasted for several centuries. These tribes followed three main routes in their dispersal:

- Northwards towards Syria and Iraq
- Eastwards through C-Arabia
- South-eastwards along the settled fringe of S-Arabia to Oman

The first identifiable 'Arab' migrants to Oman established themselves in the western desert borderlands in pre-Sasanian times (possibly the 1st or 2nd c AD). Their final success in taking control of all of Oman did not occur until the middle of the 7th c AD, while Azd migration into the region continued at least until well into the 8th c AD.[183]

Oman/Mazun was a possession of the Achaemenids and later, according to the *Periplus*, of the Parthians. A decentralized Parthian power was succeeded by a more centralized Sasanian one, but few details are known about the earliest foreign occupation. The Persians taxed merchandize arriving here from further east and south before being transited to the north and north-west. Mazun must have served as a market for Persian raw materials and manufactured goods. In return it offered foodstuffs and copper. In all likelihood the Sasanians ruled at certain times by means of vassals. For the Sasanians, Oman's strategic role was probably more important than its commercial one. But this tells us little about Central Oman's population, which is known through excavations.

Around 1200 BC there seems to be the arrival of a new people. The new settlers are taken to be the population of the Samad culture because their material remains represent a break with those of the Early Iron Age Lizq/Rumaylah culture (1200 – post 300 BC). Written sources complement archaeological information and provide an indication that the arrival of the early tribes coincides with the rise of the Samad culture. No other large group is known at this time, which could be responsible for changes in pottery, settlement patterns and burial customs.

Initially, without stratigraphy and with few radiocarbon determinations, the chronology of pre-Islamic cultures was largely theoretical, based on groupings of typologically associated artefacts loosely dated by outside comparisons. In 1979 the *German Archaeological Mission* began fieldwork in the interior, at al Maysar. The mission set out to trace the history of copper-producion in Magan/Makkan, known from cuneiform text of the late 3rd and early 2nd millennia BC. It was only in 1980 that excavations at Maysar-9 revealed an unknown hand-made pottery together with iron arrowheads and daggers in tombs. After some more excavations of these tombs, Samad-culture was chosen as a name because of its chronological neutrality. Previously, prehistoric pottery not precisely attributable to the 3rd and 2nd millennium BC, by comparison with material from SW-Iran, was catalogued as Iron Age.

[182] This summery is largely based on Yule, 1999b: 121-146, unless stated otherwise.
[183] Wilkinson, 1977: 126-128.

Excavations at Lizq, Rumaylah and elsewhere reveal an Early Iron Age culture largely contemporary with that of the iron-using neighbours in Iran and Iraq. The final date of the Early Iron Age is not yet very clear and the beginning date of the Samad period is even less clear. Recently progress has been made in firming up the definition of the end of the Early Iron Age on north-west Oman at around 300 BC, largely on the basis of Iranian parallels.

The Samad culture is the main archaeological manifestation at the turn of the 1st millennium BC/AD in what is the present-day Sultanate of Oman. It is represented at over 20 different sites. Except that it is still usually hand-made, Samad-period pottery differs entirely from that of the preceding period. Nonetheless, a few vessels from the Samad-period graves seem transitional in their shape and/or fabric from those known from the preceding Lizq/Rumaylah period.

In the 1980s the Samad period/culture was dated to the so-called Hellenistic/Parthian age, or alternatively to the last 3rd or 4th c BC. The dating rested on the appearance of iron arrowheads and other implements, a bronze bowl with constricted rim and an iron sword. The Samad culture was considered to succeed the Lizq/Rumaylah culture in a linear fashion and to ended with the Islamization of the country in the early 7th c AD.

The first ^{14}C-dates pointed to a dating for the Samad period overlapping with or immediately succeeding the Lizq/Rumaylah period. Later on and with more dating there seemed to be a clustering from 250 to 1050 AD. Based on artefact parallels one could take the beginning of the Samad period at the end of the Lizq period, but then a hiatus of 500 years exists to the first firm dates. The gap in the ^{14}C chronology is a result of archaeological sampling and not due to the measurement of the decay of ^{14}C. There is no clear indication that the Samad population declined or was assimilated immediately following the onset of Islam. One reason is that early Islamic graves which might illuminate this matter have not been excavated. Nor does it seem likely that the battle of Samad al Shan in 893 AD directly resulted in the demise of the non-Muslim Samad people. The last cluster of dates is at around 900 AD, conventionally the end of the Samad-period[184].

The Samad cemetery occupies a gravel terrace which is bordered by the Wadi Samad in the west, by some ridges and hillocks and a tributary of the main wadi in the north and a mountain range in the east. It was discovered in 1976 by the expedition of the German Mining Museum. A total number of 2000 to 3000 or even 4000 graves is not unlikely. Three major groups of grave types are distinguished[185]:

- Aboveground graves: nearly circular in plan, the diameter varied between 2,5 - 4,5 m. No entrance to the circular or oval burial chamber could be located due to the limited height to which these structures were preserved (max. 0,5 m). The ceiling was probably rather flat. The interior dimensions of the burial chamber measured between1,2 - 1,5 m, and a one-person interment in a flexed position is suggested. The state of preservation was bad and based on some pottery a date to the second quarter of the middle of the 1st millennium BC is proposed.
- Aboveground-subterranean burial structures: the above ground part is badly preserved, only one layer of stones remains from an oval ring wall. The burial cist was underground, dug in the gravel terrace and sometimes lined with upright standing boulders. The cist walls tapered towards the bottom which is 0,5 - 0,6 m below the surface. Skeletal material is almost never preserved, but grave goods were fairly abundant (personal ornaments, iron arrowheads, iron swords and daggers, etc.).
- Subterranean graves: These were the most common grave type. Some are marked by an aboveground stone circle or a flat mound of gravel, but these are missing in most cases. These were single burials. The ceiling was about 0,25 - 0,70 m below the surface. Their

[184] Yule, 1999b: 138-140.
[185] Vogt, 1984: 271 & 272-277.

orientation varies from E-W to SE-NW. Three types are distinguished: infant graves, small and large shaft graves. The small shaft graves had a rectangular ground plan. The larger tombs are constructed in the same way as the smaller ones, their size being the only difference.

The large diversity of Iron Age grave types all over the Oman Peninsula suggests different burial customs as a result of local cultural processes. During the earlier phases of the 1st mill. BC a more or less uniform cultural assemblage is spread all over the Oman. The appearance of iron weapons in a large quantity in Oman is of strong chronological relevance. At midst 1st mill. BC sites weapons are exclusively made of bronze. At Samad however apart from a few mountings all metal objects were made of iron.[186]

- **The coastal settlement of Khor Rori/*Sumhuram***

Khor Rori/*Sumhuram*, also know as ancient *Moscha Limen* of the *Periplus*, was founded on the Dhofar coast (Oman) in the early 1st c AD[187]. Khor Rori was a colony of the Hadramawt kingdom founded on the coast far from the capital and from the state's territory. It had nothing to do with the previously discussed Samad culture. The political relations between the two are still a matter of debate. It was a port for the most part used for the incense trade. Its foundation is connected to the increase in maritime trade between Rome and India at the beginning of the era. Khor Rori was a storehouse for incense, the departure point for short sea transport to Qana' and a stopping point for some Indian seamen who had arrived too late to profit from the monsoon and were forced to spend the winter. Possibly Khor Rori was built on top of an earlier settlement. The earlier settlement may not have specialized in the frankincense trade, but might instead have been a stopping place on the ancient sea routes to India or N-Oman for trade in everyday goods or metals. A. Avanzini states there is evidence of important iron and bronze production throughout the history of Khor Rori and probably a commercial link for metals existed with Hadramawt. Furthermore she mentions a hypothesis on a northern route that joined Khor Rori to inland Oman, tied to the copper trade.[188] In the literature gone through for this study no further mentioning of this route or the importance of the site in connection to metal production and/or trade was found.

Khor Rori is mentioned here because some samples for analyses were obtained from that site[189]. They include six copper-base alloy fragments and six pieces of slag. They are indicated further on by the prefix KR. The archaeological remains on the site point to a possible smithy and also small crucibles for copper(-base alloy) melting were encountered. The fact that the site is mentioned by A. Avanzini in connection with the commerce of metals, made it an interesting site for comparison to the material with that found at ed-Dur and to see if they share a similar origin or composition.

[186] Vogt, 1984: 276.
[187] Sedov, 1996, p. 23-24.
[188] Avanzini, 2002: 14-15 & 20.
[189] My gratitude goes to Prof. A. Avanzini and PhD student A. Pavan.

2.4. NE-Arabia – Bahrain

The history of the island of Bahrain (ancient *Tylos*) during the Tylos Period is still rather badly understood. It covers a long span of time between the end of the 4th c BC and the rise of Islam in the 7th c AD.[190] The remains of the Early and Late Tylos period (*ca*. 325 BC to 250 AD) prove close contacts to the Hellenistic world. It is unclear which role Tylos played at that time, how strongly it was affected by Seleucid and Parthian control and to what extent local customs changed due to western influences.[191] Contrary to NE-Arabia (Gerrha) and Failaka, the exact role of Bahrain during the period of the Seleucid Empire is unknown. Its location and natural environment suggest its importance as a stopping place for bunkering fresh water and food on the trade route towards the East. Moreover, the Bahrain pearls were products much sought after by the Romans.[192] During the Parthian period, the role of Bahrain as a halting place for the Persian Gulf merchants would not have changed much.[193] Generally, it would have been an autonomous region, sometimes under influence of the dynasties that controlled the Gulf trade. The only evidence of a foreign rule is an inscription of Palmyra from 131 AD where it is made clear that the satrap of Thiloua/Tylos was under the reign of the Characenean governor Meredat.[194] On Bahrain, just a few objects dating to the Sasanian period were excavated.[195]

Only very few settlement remains have been discovered. *Ra's al-Qal'at* is the Hellenistic settlement on Bahrain with to the north a fort build from the pre-Islamic period, *Qal'at al-Bahrain*. The fort also has a second phase of use dating to the Islamic period. The exterior fortification walls were 2,4 m wide and a good defensive position was assured by positioning the entrance to the least vulnerable side and providing the towers with arrowslots. The fact that round towers are used in the fortress suggests a date after the Seleucid period, when square or rectangular towers were universally used (see the fortresses at Thaj and Failaka). The fortress should be dated around the 2nd or 3rd c AD. The causes for the dearth in settlement remains are uncertain. Maybe the occupational areas are still covered by the present-day habitation or they might have been destroyed by cultivation activities. It is also possible that they were not situated in the vicinity of the cemeteries.[196]

Whatever the reason the consequence is that we must rely on the study of burials to learn more about this still rather unknown chapter of Bahrain's history. While settlement remains are scanty during the Tylos period cemeteries are scattered all over the fertile region of Bahrain.[197] Despite the excavation of a rather large amount of burials however, the shortage of detailed publications and lack of settlement sites obscure our knowledge of these centuries. The most common burial practice in Tylos was individual burial. Cemeteries were placed away from the settlements and were above ground. Often, at the heart of the necropolis, there was one elaborate grave covered by an impressive burial mound. The dead were joined with a whole series of burial goods, but it must be said that the presence of iron weaponry was rather exceptional.[198] There is an evolution from single burials with a mound, to more single burials in one mound towards multiple burials in one tomb[199].

[190] Salles, 2000: 133-134.
[191] Herling, 1994: 225-226.
[192] Boucharlat & Salles, 1989: 84; Herling, 2003: 25.
[193] Boucharlat & Salles, 1989: 84.
[194] Potts, 1990b: 145.
[195] Boucharlat & Salles, 1989: 85; Højlund & Andersen, 1997: 13.
[196] Potts, 1990b: 111-115.
[197] Herling, 1994: 225-226.
[198] Herling, 2000: 136-140.
[199] Andersen, 2005: 276.

The period between *ca*. 300 BC (the Seleucid time) and 600 AD (the Islamic time) is called the Tylos period. The chronology of the period can be summarized as follows[200]:

- The Early Tylos period or 'Failaka horizon': *ca*. 300-100 BC
- The Middle Tylos-1 period or 'Bahrain horizon': 1st c BC - early 1st c AD
- The Middle Tylos-2 period or 'ed-Dur horizon' 1st - 2nd c AD
- The Late Tylos period: *ca*. 250-600 AD.

This subdivision is somewhat refined by the recent study of S.F. Andersen who attested four phases based on the ceramic sequence. He furthermore states that the earliest Tylos period should not be dated earlier than the late 3rd/early 2nd c BC. His statement is based on the comparing a large selection of pottery from the tombs excavated by the Bahraini authorities with the pottery sequence from Qala'at al-Bahrain. An overall lack of burials from early Tylos period, which is also evident in the Achaemenid Period indicates that a burial were performed without much material splendour.[201]

The ceramic assemblage of *Phase I* (*ca*. 200 - 50 BC) gives the overall impression of a mixed collection of vessels representing a number of traditions, some of which can be assigned to specific areas, whereas others lack comparable materials. A significant proportion of the vessels is influences by Greek pottery, but also by vessels types in Near Eastern and Arabian traditions.[202]

The ceramic types from *Phase II* (*ca*. 50 BC - 50 AD) do not find exact parallels outside the Gulf-region. The assemblage gives a very coherent impression. Most of the types show strongly influence from Roman or Eastern Mediterranean pottery.[203]

Phase III (*ca*. 50 - 150 AD) has a lot of good parallels at ed-Dur. The high number of vessels (both imported glass and local Gulf pottery) indicates more burials than in the other phases, either due to a larger population or caused by the fact that a larger part of the population adopting the beliefs and traditions manifested in the tomb burials. A generally higher wealth among the population enabling more resources to be spend in grave construction and more grave goods to be placed in the tombs. The material gives the impression of a time of prosperity.[204]

The Iranian influence that can be noticed in the assemblage of *Phase IV* (*ca*. 150 - 450 AD) did not necessary start in the middle of the 2nd c AD. This assemblage can be associated with the rise of the Sasanians in the beginning of the 3rd c AD and their successful expansion over the next decades[205]

The beginning of Phase II corresponds well with the eastern expansion of the Roman Empire, and the increased import of East Mediterranean glassware. T•he adoption of Roman types of pottery by local potters could well be the result of increased contacts between NE-Arabia and the Roman Empire. Since we have no evidence for an active Roman policy in E-Arabia, the driving force of the contacts is likely to have been commercial. There is written evidence for trade between E-Arabia and the Mediterranean via Nabataeans or via the Characenean kingdom and Palmyra. In this time the NE-Arabian entity, which played a crucial role in the introduction of the new burial tradition on Bahrain in the previous period may have lost some importance.[206]

[200] Herling & Salles, 1993: 161 & 177.
[201] Andersen, 2005: 8.
[202] Andersen, 2005: 228.
[203] Andersen, 2005: 229.
[204] Andersen, 2005: 231.
[205] Andersen, 2005: 232.
[206] Andersen, 2005: 294.

Towards the end of the 1st c AD or the first half of the 2nd c AD it seems as if the close contact to the Mediterranean came to a sudden halt. Thereafter we no longer find the abundance of Roman wares or their influence on the style of the pottery. Not only did the style and origin of the grave goods change, the amounts of grave goods also fell significantly. It seems that the whole pattern of trade and distribution changed, and the inhabitants on Bahrain no longer got the same share of the profit as in the previous periods. Multiple burials and the reuse of tombs became very common, perhaps indicating that the resources available for building new tombs were limited. The cemeteries and choice of categories of grave goods remained, however, the same as before and a radical change in belief and thus burial custom seem therefore not to have taken place.[207]

The economic crisis is difficult to link to the rise of the Sasanian power, since that only started later. A decline in the demand of eastern products by Roman consumers would be an obvious explanation. It is likely that the Romans in beginning of the 2nd c AD were accustomed to eastern products and that they no longer possessed the same aura of luxury.[208]

[207] Andersen, 2005: 296.
[208] Andersen, 2005: 303.

Chapter 3. TRADE

"Don't waste time learning the tricks of the trade. Instead, learn the trade."

Anonymous

3.1. Introduction: definitions & sources

• **Definitions**

If we discuss *trade* some things have to be made clear. In this context trade is more than the *buying and selling*, since this implies a monetary society. Although local and foreign coins were found at ed-Dur it is far from clear that these are enough proof for a full-blown monetary economy. The, all by all, few foreign coins are all of small denomination and are in the pocket money category. The local coins on the other hand were only found in Mleiha and ed-Dur and do point towards (very local) use as a means of payment, most probably existing next to older exchange networks. Even a fully monetarily society as the Romans Empire still used barter trade in many of the ports mentioned in the *Periplus*. Putting these coins in the right perspective is an important factor to understand the true function of ed-Dur, since they tell a story of a rather exceptional short-lived event (the first local coinage) but with rather limited impact on the larger scale. Trade probably triggered this event.

A second factor to bear in mind is that the Gulf has always been an axis of trade from its earliest days till now. The most obvious means of transportation would be by ship, but it should not be forgotten that there also were land-routes for transportation by caravan. Although often complementary, from time to time one route had the upper hand without completely eliminating the other however.

G. Plisson states that the study of the exchange systems in antiquity involves *three aspects*[1]:

- Identifying the origins of the materials and techniques that are identified as imports.
- Determining the chronological framework in which exchange took place, along with any changes that might have occurred.
- Reviewing the historical implications of the evidence and comparing this with the various theoretical models proposed for the explanation of political and economic development in antiquity.

The *first aspect* is traditionally covered by the typological study of the *archaeologica* and comparing that with reference material from the places of origin. For ed-Dur this has been done for the ceramics and is under construction for the other small finds. In this dissertation the iron objects are treated in this way. Additional information based on the chemical composition and the lead isotope signature of the metals is presented. This is supplemented with a focus on the historical data on the trade in metals during the period under consideration. Finally the available information derived from the ceramics and the other small finds is summarized to complete the picture.

The *second* and *third aspects* are treated as one since politics and economics (e.g. trade in this case) are closely interwoven. For this the economic and political situation of the wider region was sketched in *Chapter 2*. This is necessary since the historical lifetime of ed-Dur was limited and no real internal site chronology is found so any external factor is a welcome element to help explain the existence of ed-Dur.

The word *harbour* or port should also be put in its right context. It has to be noted that sea-trade does not always require 'western'-style big harbours, with permanent structures. Even for the Roman period no ports with harbour installations and structures similar to those in the Mediterranean are known along the Red Sea coast. This in spite of archaeological surveys and exploration work in the vicinity of ports of major importance, e.g. Berenike. Trading activity in the region, as mentioned in the *Periplus*, was marked by heterogeneity. Muza on the S-Arabian shores at the mouth of the Red Sea, for example, is described as a port that "though without a harbour, offers a good road-stead for mooring"[2].

[1] Plisson, 2005: 67.
[2] Ray, 2003: 189.

A great deal of Roman trade was coastal trade carried out by small ships, which loaded and unloaded at ports with minimal facilities or no facilities at all. There is no reason to believe that this was any different in the Gulf region. The *beaching* (stranding) of small vessels or *mooring* just off an unadjusted open beach must have played an important role throughout antiquity. In tidal areas beaching remained a widespread practice well into the 19th c AD and in non-tidal areas ships can either be winched up onto the shore or moored to posts in shallow water. In all these cases, ships can be readily loaded and unloaded either by men wading through the shallow water or directly into small vessel or rafts brought alongside the ship. At Dubai on the Gulf, modern *dhows* anchor, load and unload in shallow water along the banks of Dubai Creek where there are no quays, all unloading apparently being done by men wading through the water. Where beaching is not practised the simplest and cheapest solution would be to anchor offshore and unload into lighter/smaller vessels. With these economically cheaper alternatives ships of all sizes can be handled and the commercial activity of the port may be considerable even in the virtually total absence of quays or piers.[3]

When considering the sea and land trade routes it is very important to keep in mind that at now point in time only one or the other was in use, but rather that one or the other was more important. Geographically it is impossible to reach all areas by sea. Certain areas can however be crossed by land or by sea, e.g. the head of the Gulf can be reached over land as well as by sea from S-Arabia. The two networks are complementary and goods shipped by sea had to go back at land at some point or another for further transported over land, i.e. by the land routes.

- **Textual sources**

The first group of sources are the Roman classical texts written at the time or preserved (and often distorted) in later copies. Next to that there are clues found in old text from India, China, the Talmud, etc. All these were used to the level that they were accessible (e.g. often indirectly) since this is not a PhD in history, with the exception of the anonymous *Periplus Maris Erythraei* (referred to as the *Periplus* in this study) and to a lesser extent Pliny's *Naturalis Historia.* These last two texts were written at the time ed-Dur was inhabited and they provide direct information concerning the Roman-India trade, the goods shipped, the itineraries followed and the harbouring places called in. Next to this textual information there is also a growing body of archaeological evidence to support (or reject) them.[4]

The *Periplus Maris Erythraei* (= The Periplus of the Erythraean Sea)[5] is a Greek commercial trade handbook generally accepted to date from the 1st c AD or shortly after. Literally the Erythraean Sea means "red sea", but it does not refer to the Red Sea (called Arabian Gulf by the ancients) we know. It reference to a much larger body of water including the Red Sea, Gulf of Aden and the western Indian Ocean. According to L. Casson the Gulf is not included, since it is mentioned separately as the *Persian Gulf*.[6] The anonymous author (most probably an Egyptian Greek) wrote it as a technical guide for merchants (and sailors) who set sail from the ports of Roman Egypt at the Red Sea to the coast of Africa, the western and southern coasts of Arabia and the western coast of India. It gives the itineraries, harbours, navigation and especially cargoes handled in the trade network. The detailed information on the traded goods is rather atypical for a *periplus*, which is foremost a guide for seamen whereas this work is a guide for merchants in the first place. The fact that it was never intended as a work of literature, but as a practical manual, we may reasonably expect the provided accounts to

[3] Houston, 1988: 560-562.
[4] R. Tomber (2004: 351) mentions the Roman finds from Timna, Qani, Shabwa, Khor Rori, Mleiha and ed-Dur and the current excavations at Red Sea sites and in Jordan.
[5] For this study the translation and comments of L. Casson (1989b) are used and in the text this work is referred to as the *Periplus*. The older translation by W.H. Schoff (1912, second print of 1974 used here) was checked but no additional information was provided.
[6] Casson, 1989b: 94.

be reliable and accurate. This is what makes the *Periplus* such an exceptional document.[7] For an extensive list of the traded goods mentioned in the Periplus see *Appendix 1*.

The *Naturalis Historia* (= Natural History) can be seen as an encyclopaedia of the knowledge of the 1st c AD and was composed by Pliny the Elder (23-79 AD). Especially book VI is important here. When Pliny is mentioned in the text, this is the work that is referred to.

The oldest first hand information on parts of the Indian Ocean and the Gulf was collected by the captains (Nearchus, Androsthenes, Onesicritus, etc.) of Alexander the Great. They were sent out on expeditions in the region to scout these coasts. Although the information collected under Alexander was sound and trustworthy, it suffered from centuries of copying and results in a patchwork of information.[8]

Other more sporadic and indirect references to the subject are provided by the geographer Strabo (63 BC-24 AD). Claudius Ptolemy's *Geography* was written in the 2nd c AD but has possible later additions. It offers similar information then the *Periplus* on the routes and the locations of ports. It is however less informative and in some cases confuses the earlier picture drawn in the *Periplus*.[9] *Papyri* and *ostraca* have also provided important information on the eastern commerce and the bulk of the evidence is from the Red Sea ports in Egypt. These refer to Egyptians, Greeks, Arabs and Indians residents in these ports who controlled the Indian trade.[10] Inscriptions in relation to the trade are another source of textual evidence and are well attested in Palmyra. Many of them refer directly to the caravan trade of Palmyra and usually commemorate the successful return of trading expeditions to the Gulf.[11]

- **Archaeological sources**

A completely different kind of source is the material remains left behind. The first obvious group would be object that can be clearly defined as imports, so foreign objects found on site. Ed-Dur has a large variety of *archaeologica* that originated elsewhere. A short overview of these objects will be given at the end of this chapter to sketch an as wide a picture as possible.

A second group of remains would be shipwrecks, in the case of this topic those that carried metals as (part of) their cargo. They can tell an enormous deal on trade routes, commodities, etc. Unfortunately no shipwrecks are known from the Gulf region dating to the period under consideration[12]. The Mediterranean has a much richer catalogue and considering the importance of the Roman-India trade during this period, they might also provide some information. These ships were of course not (directly) involved in the Roman-Indian trade, but they are an indictor of what the Romans were transporting in the Mediterranean at that time and can at least give some clues on what was possibly shipped to other places.

[7] Casson, 1989a: 187; Casson, 1989b: 8; Thapar, 1997: 14-15; Young, 2001: 5-6.
[8] Salles, 1996b: 295; Ray, 2003: 168.
[9] Chami, 1994: 25; Thapar, 1997: 14-15.
[10] Ball, 2000: 123.
[11] Young, 2001: 8-9.
[12] A shipwreck roughly dated to the Parthian or Sasanian era, based on some amphora sherds, was recently discovered at adepth of 70 m in the Gulf. No further details are provided however (11-11-2006). http://www.tehrantimes.com/Description.asp?Da=11/11/2006&Cat=10&Num=2

3.2. Caravan trade

The term of caravan trade within this context needs some additional refining. The transport of goods from the sea to the inland and *vice versa* has always been by means of caravan in Arabia. Here only the caravan trade between Arabian Peninsula and the Mediterranean is considered, a network that could partly be replaced by sea routes.

There were rarely ever 'roads' or even trade mechanisms in the organised sense, with all the characteristics that define roads and trade routes in more recent history. Ancient routes were nothing more than amorphous networks, with many minor branches each with their own variants. They even varied from journey to journey as well as from period to period, depending upon numerous factors such as supplies, water, trading opportunities, political circumstances and security. Any ancient *route* therefore was at best simply a broad channel of communications across a region for the movement of people, goods and ideas. It rarely followed any fixed pathway.[13]

The initial networks of routes lay between India and S-Arabia on the one side and the Eastern Mediterranean and Egypt on the other side. The land fraction of these routes passed through Iran and the Middle East. The network combined transportation by river on the Nile, sea transportation in the Mediterranean Sea, coastal traffic along the Egyptian and Levantine coasts and transportation by land and river in the territories separating the Levant from Iran and India.[14] The second main possibility, i.e. by sea, is discussed below.

On the western side of this network, the Red Sea had in early times the effect of isolating rather than uniting Egypt with SW-Arabia. Therefore the Arabs developed camel routes along the whole western side of their peninsula. Additionally piracy was (and is) wide spread in these waters. Conditions in the Gulf were better, but there too there was a lack of fresh water on both sides, and the number of islands encouraged piracy.[15]

The spiders in the middle of this network connecting S-Arabian, India and the Mediterranean were the people of Gerrha and the Nabataeans as seen in the previous chapter. The basic core of their business was providing high value 'exotic' commodities from S-Arabia, India and the Far East to the Mediterranean area, e.g. frankincense, myrrh, pearls, etc. A lot more ordinary goods such as agricultural produce, salt, honey, etc. probably accompanied these luxury products[16]. Generally less valuables goods made the backwards trip. This trans-Arabian caravans trade is persistently referred to in the Bible, the Mesopotamian sources and the Classical and Hellenistic texts[17].

A prerequisite for this trade overland was the camel. There is no reason to assume the camel caravans really existed before the 1st millennium BC, which seems to be the earliest period of overland S-Arabian trade.[18] The overland incense trade route northwards from Saba' bifurcated at Najran. The eastern arm continued north via Qaryat al-Fau, and then in an arc across al-Kharj to the al-Hasa oasis and its Gulf port at Ugair. The al-Hasa oasis plus its port at Ugair comprised the Gulf *emporium* known as Gerrha to classical writers.[19]

The route that linked the SE-Arabia to the region of Hofuf, and with Jabal Kenzan (Saudi Arabia) was still used in the 18th c AD. Although the sea-route was used more often, two

[13] Ball, 2000: 138.
[14] Plisson, 2005: 67.
[15] Hourani, 1963: 5.
[16] Deblauwe, 1991: 133.
[17] Salles, 1996b: 301-302.
[18] Deblauwe, 1991: 134.
[19] Mitchiner, 2004: 477.

routes were known to cover the 700 km from the al-Ain Oasis to Hofuf, in the time span of 30 days.

> *"There is no track that can be followed, as the sand is blown about by the wind, but there appear to be two general routes, one of which is used more in winter, the other in summer, the first is straighter and shorter, the other passes near the sea, is more winding, and after leaving the* sabkheh *turns north for three days. The journey is not considered dangerous or difficult, as water is found in great many places, though usually very brackish, and they seldom have to carry a supply for more than two days"*[20].

Once the goods had reached NE-Arabia they could be taken straight through the desert, along routes used by the Nabataeans or they could be moved further northwards to the Kingdom of Characene. From Characene the commodities were shipped up the rivers of Tigris and especially the Euphrates. From the frontier fort/caravan town of Dura Europos there they were taken through the desert to Palmyra and further on to Damascus, Philadelphia, Antioch, etc. or more to the south to Petra.[21] The use of this 'Euphrates routes' has to be seen as an essential part and extension of the trade dominated by the Palmyrenes. The use of this route was of course inseparably linked to the safety that could be provided along it by the Parthian Empire. There is evidence that the use of the shift towards the sea-route along the Red Sea may be induced due to upheaval in the Parthian realm.[22]

[20] Mouton, 1992: 261.
[21] Mare, 1995: 189; Young, 2001: 18.
[22] Warmington, 1974: 14; Retsö, 2006: 330.

3.3. Sea trade

Three main sea routes can be distinguished. The first went down the Red Sea, along the S-Arabian coast, across the mouth of the Gulf, following the coast till it reached NE-India. The second route also went down the Red Sea, but followed the coast further on till Cape Guardafui, from where ships took off to cross the open sea till they reached India. The third route, starting in India, runs up the Gulf till the Kingdom of Characene, hence sometimes named the 'Characenean corridor', from where the goods were further shipped up the Tigris and Euphrates Rivers. The merchandise was than taken to the E-Mediterranean coast by caravan via Dura Europos and Palmyra. All these routes obviously worked in both ways. On the first two routes 'Roman' traders were active, but these seem to have been completely absent on and/or ignorant of the third route. If ed-Dur played any part in the international trade of its time, it was connected in the network in the Gulf.[23]

- **The Roman Red Sea & 'far side' ports**

As mentioned above the Red Sea had in early times the effect of isolating rather than uniting Egypt with SW-Arabia. The northern half of the sea in particular presents some obstacles. It is flanked on both sides by hundreds of kilometres of waterless desert and good harbour places are scars. Immense coral reefs lay along both coasts and skill was required to avoid being wrecked on them. Sailing up the Red Sea was hard to early seafarers, because northerly winds blow down this part of the sea all year long.[24] It was only after the Red Sea became an outlet from the Roman Empire after the annexation by Augustus of Egypt that this situation changed. Sea trade was already going on to a limited extend during the preceding Ptolemaic period, but during the Egyptian occupation this trade expanded exponentially. Alexandria was the major port of Egypt and exported Egyptian goods (dates, grain, papyrus, etc.) next to other products from the Empire to the East. It also re-exported, as the end point of a long journey, most of the goods that came in from the East to other places in the Mediterranean.[25]

The *Periplus* tells us about the routes taken by ships from Myos Hormos (modern Abu Sha'ar' or Quseir al-Qadim[26]) and Berenike (modern Berinike or Bernis/Medinet al-Haras[27]) on the Egyptian side of the Red Sea. From the *Periplus* it is clear that the Romans did in an initial phase not really leave the Red Sea or at least did not sail past S-Arabia. They also did not appear to have entered the Gulf and apparently the Indian and Arab merchants filled this niche. The Indians playing an intermediary role between the Indian subcontinent and the Arabian communities. In the beginning of our era the Romans did not know the origin of a certain number of spices and aromatics and associated them with the Arabian Peninsula, where in reality they came from further away. This could indicate that Indian merchants at their turn were not active in the Red Sea at that time.[28]

The goods to be exported out of Egypt and the Mediterranean were shipped up the Nile to Koptos and then brought by camel or donkey across the desert to Myos Hormus or Berenike. Myos Hormus offered a shorter desert route but Berenike, further down the Red Sea coast, had the advantage that it was the first port to be encountered when returning to the Red Sea. Ships did not have to sail further up the Red Sea with it sometimes unfavourable winds and could be unloaded immediately.[29] Goods brought back from overseas did the reverse journey. From either port ships sailed straight down the Red Sea to the Arabian port of Muza, then they followed the coast along the S-Arabian shores as far as Qana'. From there one

[23] Mare, 1995: 189.
[24] Hourani, 1963: 5.
[25] de Vallerin, 1978: 102.
[26] Tomber, 2004: 351.
[27] Tomber, 2004: 351.
[28] Ball, 2000: 131; Plisson, 2005: 68.
[29] Casson, 1989b: 13.

branch took off for the ports of NW-India, Barygaza and Barbarikon, and another for those of SW-India. An alternative route to SW-India, probably used by sailors who worked the Ethiopian coast (Adulis, modern Zula) and the 'far-side' ports on N-Somalia, took off from Cape Guardafui, to the ports of Muziris and Nelkynda on the Malabar Coast. The African trade network basically yielded ivory, tortoise shell, frankincense, myrrh and various grades of cassia (actually of SE-Asian origin, but the sale took place at Africa's 'far-side' ports). What made these routes possible was the very special nature of the prevailing winds, the so-called *monsoon* winds.[30]

The seasonal monsoon winds were very determining for the East – West trade. These blow steadily from the northeast in the winter but during the summer conveniently switch to just the opposite direction, the southwest. Vessels were able to sail from Arabia to Africa in the winter and return in the summer or sail to India in the summer and return in the winter.[31] The importance of the *emporium* of Eudaimon Arabia in the first phase of this trade can be attributed to the lack of knowledge of Western sailors of the monsoon-winds so they could not sail any further than the Gulf of Aden. At a certain moment the Greeks did not stop anymore at Eudaimon Arabia and aware of the monsoons continued all the way to India. Traditionally this discovery is attributed to Hippalos and might already have taken place in 116 BC. But even then the number of Greek ships making the complete voyage was limited.[32]

- **The Arabian Red Sea & S-Arabian ports**

As mentioned above the discovery of the use of the monsoon winds greatly facilitated the travelling of ships towards and from India. After the discovery of the monsoon winds the role of S-Arabian was largely reduced to that of a watering point, between the Red Sea and India. This however did not affect the trade in aromatics, the main sources of income of the S-Arabian states.[33] Arabia basically yielded frankincense, myrrh and aloe.[34]

The *Periplus* lists three ports at the Arabian side of the Red Sea coast[35]:

1. Leuke Kome (modern Al Wadjh or Khuraybah) was a minor port located far up the coast just below the entrance to the Gulf of Aqaba. It was in Nabataean territory, and the chief reason for its existence was that from it a route went inland to the capital at Petra, although it also served for the handling of cargoes brought by small coastal vessels from Arab regions to the south.
2. Muza (modern Mocha) was the main port of the Kingdoms of the *Sabaeans* and the later *Himyarites* on the Red Sea coast and was situated far down the coast more or less where Mocha is today. This port provided a market for various articles of cloth and metalwork, as well as wine, grain and money. In return traders picked up myrrh, as well as some marble and some goods brought to Muza from Somalia in Arab boats.
3. Qana' (modern Bir 'Ali) was the prime ocean port of the "frankincense-bearing land" of the Hadramawt, on the site of present-day Husn al Ghurab. Next to Qana', the port Moscha Limen/Sumhuram (modern Khor Rori) was an important outlet for frankincense. The major trade item was of course frankincense, both local and imported from Somalia. Mocha Limen apparently only received boats sent there from Qana' or boats coming back from India and that stayed there during the winter.

Today the chief port of the southern coast is Aden, thanks to its good natural harbour. It had been a centre for international trade before the *Periplus* was written. Ships from Egypt went only as far as Aden, not daring to venture into the open waters further on. At Aden they were

[30] Casson, 1989a: 188-189 ; Casson, 1991: 8.
[31] Casson, 1989a: 188-189 ; Casson, 1991: 8.
[32] Casson, 1989b: 12; Retsö, 2006: 330
[33] Young, 2001: 19.
[34] Casson, 1989b: 17-18.
[35] Casson, 1989a: 188; Young, 2001: 35.

met by vessels from India and cargoes were exchanged, so this harbour functioned as a transfer market. Aden has been an *emporium* (ancient Eudaimon Arabia), importing and exporting, serving the masters of the interior, rather than becoming a power in its own right. But by the time of the *Periplus* the Roman Egyptian sailors had learned about the monsoon-winds and were sailing to India on their own, in this way eliminating the function of a transfer harbour of Ocelis and Eudaimon Arabia that seem to have been reduced to watering stations. The port of Ocelis was reserved for vessels arriving from India, which the *Periplus* tells us was "not a market town, but the first landing for those sailing into the Gulf"[36]. By the 1st c AD the ports of Muza (Himyar*) and* Qana' (Hadramawt) had taken the position of most important harbours at the expense of the port of Aden that was reduced to a harbour of refuge and watering station. Because of its good location it did not take long for Aden to regain its importance and by the 2nd c AD the city had gained a renewed importance.[37]

- **The Indian connection**

For the Romans the Indian west coast was the most important and the east coast played a distinct second role. The Malabar coast trade can be further divided in two spheres: the ports of Barbarikon (modern Karachi) and Barygaza (modern Broach) in the NW and Muziris (possibly modern Cranganore/Kodungallur) and Nelkynda (modern Niranam) in the SW. Barbarikon was merely a port where all goods were forwarded to the Royal capital of Minnagar. Based on the raw products imported and the bulk exported Barygaza must have had an industrial area. The same was true for Muziris and Nelkynda, which are treated as one in the *Periplus*. There is good reason to believe that the 'Roman' ships did not go any further then the waters between India and Ceylon. Still there is archaeological evidence that a colony of Westerners was present at Arikamendu on the SE-coast. Also Muziris seems to have had a Western group of people. Of course land or/and sea routes interconnected all these Indian centres.[38] The ports in the NE of India were reached by sealing along the coast, whereas a more direct route across the Indian Ocean reached the SW ports.[39]

Between India and the Mediterranean world a lot of exotic luxury goods were traded, e.g. spices, pearls, semi-precious stones and silk from China. It should however not be forgotten that everyday raw materials to supply workshop centres, foods, timber, metals, manufactured products such as pottery, textiles, glassware, etc. were also part of the transported goods. The volume of these was probably not great however, since ships were small and it is almost certainly true that the demand for low-bulk, high-value luxuries drove the trade. Common commodities exported by the West were wine, grain, olive oil, glass, metals, coral, textiles, as well as Roman money and frankincense (picked up in Arabia during journey to India).[40]

In early Imperial Roman times, judging from the archaeological remains found in India and the statements in various Greek and Latin texts, there appears to have been a growth in the trade between the Red Sea and India concentrating on W- and S-India from the late 1st c BC to the mid 1st c AD. A later phase dates to the 2nd c AD when the E-coast of India becomes more prominent. The question as why the Roman input seems to decline after the 2nd c AD and early 3rd c AD still remains largely unanswered. Much of the answer would lie in the historical changes that took place in those regions of India most closely connected with the trade, W-Asia and the eastern Mediterranean.[41] The serious monetary inflation in the Roman Empire during the 3rd c AD is one factor that would have greatly reduced the ability of citizens to purchase luxury products. A second reason could be a saturation of the Western markets and a loss of interest in the by then known 'exotic' products. Based on the number of

[36] Schoff, 1915: 230.
[37] Casson, 1989a: 190.
[38] Casson, 1989b : 23-25.
[39] Young, 2001: 29-30.
[40] Schwarzer & Deal, 1986: 57; Glover, 1996b: 369; Thapar, 1997: 14. See *Appendix 1* for an extended list of goods as mentioned in the *Periplus*.
[41] Thapar, 1997: 13 & 39.

shipwrecks found in the Mediterranean Sea from the late Hellenistic and Roman periods, it can be concluded that the greatest activity took place from *ca*. 200 BC to 200 AD, with a significant dip in the 3rd c AD.[42]

- **The 'Characenean corridor'**

A last major line of commerce is scarcely mentioned in the *Periplus* and started at the head of the Gulf in the Kingdom of Characene and went down the Gulf to S-Arabia and possible beyond to Indian harbours and *vice versa*. It may be safely concluded that 'Roman' traders were not active in the Gulf. This segment of the international trade network was dominated by Palmyrenes, Arabs and Iranians and was pre-eminent in the India trade long before the Roman Period.[43] Goods desired from India and further on, ended up in the West by sea-borne trade up the Gulf. From Characene the commodities travelled up the Euphrates and the Tigris to be taken by caravans to the E-Mediterranean. The *Periplus* does however mention Apologos (near modern Basra), Spasinou Charax, both at the head of the Gulf, and Omana. There is a long-standing discussion on the actual location of the *emporium* of Omana, amongst the suggestions is the site of ed-Dur.[44] The 'Euphrates routes' mentioned above is the extension of the trip up the Gulf by ship, controlled by Characene and Palmyra.

[42] Young, 2001: 85.
[43] Ball, 2000: 131.
[44] See Potts, 1990b: 305-310 and pp. 63-65.

3.4. The position of SE-Arabia

A last word should be said on the position of SE-Arabia in this entire network. The western goods that were found in the archaeological sites of the Gulf could have ended up there along several different routes. Moreover it should be kept in mind that goods do not always need to travel along the shortest routes.

The first route would be along the non-Roman segment Apologos-Barygaza of the Indian Ocean trade. The goods had first reached the north Indian harbours on Roman ships, from Alexandria through the Red Sea and the Indian Ocean navigation. They were then re-exported from Barygaza and Barbarikon to the Gulf by Arabo-Persian merchants and sailors.[45] This does also not need to be directly to ed-Dur, since regular contact with the other side of the Gulf by means of small boats is very possible. The presence of SE-Iranian wares in SE-Arabia can be explained in this way.[46]

The second possibility was that goods went on land in S-Arabia and were further distributed to SE-Arabia by means of caravan or by costal navigation. There was a busy contact between local traders from Qana', Barygaza and Apologos-Omana in the Gulf. The *amphorae* on ed-Dur could be shipped from the S-Arabian and Oman-coast from Qana' and/or Sumhurum.[47]

The third option is that the commodities were brought in via Characene, after they had travelled through Palmyra, down the Euphrates and then down the Gulf. The part down the Gulf could have been covered by land or by sea. The large amounts of imported ceramics from S-Mesopotamia are evidence of regular contact. The shear quantity of these ceramics in the overall assemblage at ed-Dur is in contrast with the smaller amount found at Mleiha. This large amount of vessels might imply an arrival by larger ships.[48]

[45] Salles, 1993: 516-517; Haerinck, 1998c: 25-26; Haerinck, 2003: 200.
[46] Rutten, 2006: 413-415.
[47] Rutten, 2006: 408.
[48] Rutten, 2006: 413-415.

3.5. Trade in metals

3.5.1. Introduction

There is a dearth of archaeological data in the Gulf for the Hellenistic period. Deducing from the *Peripus* this limited knowledge can be attributed to the nature of the exchange products, which in most cases would have hardly left any archaeological remains. These commodities being: foods and drinks, textiles and clothing, household items and tools, raw materials, costly materials, spices and aromatics, drugs and dyes, and slaves. The merchandise that travelled from the Eastern Mediterranean and/or Arabia to India that could have left material remains are: recipients containing oil and wine, glassware, drinking vessels (copper, etc.), tools and precious stones. Material evidence of products going in the reverse direction, from India to Arabia and/or the Eastern Mediterranean, could be found in the presence of: iron and steel, tortoise shell, ivory, marine shells, pearls and precious stones. It is easy to realize how little would have been preserved of the goods that were imported into the West such as dates, frankincense, textiles and silk, aromatics, spices, woods, slaves, etc. and they will remain permanently absent from the archaeological levels of any harbour-site. Because of the nature of the cargo many more traces are preserved of western exports to the East than Indian or Arabian imports in the Eastern Mediterranean.[49]

The main source on trade in metals between East and West during the 1st c AD is again the information given by the *Periplus* and a thorough discussion is presented by L. Casson[50] on that matter. The information in the *Periplus* should of course be treated with the necessary criticism. L. Casson for example finds difficulties in the references of the *Periplus* on the Indian import of certain metals, such as copper, tin and lead[51]. They were imported, although at some ports these metals must have been available from indigenous Indian sources that were located in the not to distant vicinity. Several reasons for this phenomenon can be offered, such as the fact that traders invariably buy commodities from the cheapest available source, in this case the Roman Empire. In antiquity the transport of heavier items by sea was also substantially cheaper than by land. Ships engaged in the Indian trade were large enough to safely cope with the monsoon, and some heavy commodity cargoes would have provided welcome ballast.[52] The import of an excess to re-export it to other areas (e.g. the Gulf) should also be considered[53].

The *Periplus* reveals several lines of trade next to the well-known movement of Eastern luxuries to the ports of Egypt. There was also a trade in commodities from India to the coasts of Persia, Arabia, and Africa that had nothing to do with the West. Arabs and Indians handled the bulk of the goods in this network. Inscriptions reveal a line of trade that ran from the head of the Gulf to the mouth of the Indus and back carried on by merchants and shippers of Characene and other cities in the area. We can even distinguish certain local forms of trade, so local that the means of transport were small crafts and rafts. From the other side Roman Egypt sent out to Africa, Arabia and India a mix that ranged from everyday tools and cheap clothing to the costliest of luxuries for the courts of regional rulers.[54]

[49] Salles, 1996b: 296.
[50] Casson, 1989b.
[51] Casson, 1989b: 27-29.
[52] Mac Dowall, 1996: 94.
[53] Biswas, 1996: 309.
[54] Casson, 1989b: 21.

Concerning the trade of metals or metal wares the information contained in the *Periplus* can be summarized as follows:

- Export from Roman Egypt to overseas ports: [basic products] copperware, copper honey pans[55] (?), copper drinking vessels, ironware and iron tools (axes, adzes & knives) – [expensive products] bronze and brass vessels, silverware, goldware, statuary (might be from metal) – [Raw materials] iron, tin, lead and copper.
- Export from India to ports other than those in Roman Egypt: iron, steel and copper.

From the Mediterranean shipwrecks known from the 22nd to 12th c BC it is clear that metal represented the most important and dominant cargo. This seems to have changed by the classical period (between 400 BC – 400 AD) where metal and ore cargoes are significantly less numerous (only *ca*. 10%) than the most common cargoes, *amphorae* (*ca*. 54%). Metal is most often found in the form of ingots.[56] A.J. Parker lists the following cargoes found on ancient shipwrecks in the Mediterranean: lead ore (2 cargoes), lead ingots (46), lead (7), iron ore (1), iron bars (16), copper ingots (27), tin ingots (16), other ingots (8), ore (5), copper-base metal goods (19) and not copper-base metal goods (18)[57]. Most of the known shipwrecks that yielded metal ingots are dated to the Augustan period. The rarity of 3rd c AD wrecks with metal ingots and objects is surprising and it seems that small mines in the Mediterranean world were satisfying the local needs, while no mines played a role of international importance at that time. This evidence finds confirmation in archaeological and literary sources on mining in the Late Roman Empire. Most of the wrecks dated to the 4th – 6th c AD consisted of cargoes of scrap metal.[58]

Below only the information on the trade in metals is presented in greater detail and supplemented with any other historical or archaeological relevant data.

3.5.2. Copper-base alloys

According to the *Periplus* brass and bronze vessels were exported from Roman Egypt via the African route to the first major stop at Adulis (Abyssinian kingdom of Axum, Ethiopia). This port did not only imported goods for the local need, but also for the court that was situated more land inwards. The imported brass was used for making personal adornments and coins, and the bronze vessels were used as vessel or were cut up and made into ornaments. A second line of export, direction Indian subcontinent, stopped at the S-Arabian port of Qana' were amongst other products bronzeware was delivered for the local courts.[59]

A. Avanzini mentions that there is evidence that Khor Rori (Sumhuram or Moscha Limen) was important for the production of iron and bronze and that probably a commercial link for metals existed with Hadramawt. Furthermore she mentions a hypothesis on a northern route that joined Khor Rori to inland Oman, tied to the copper trade.[60] In the literature gone trough for this study no further mentioning of this route or the importance of the site in connection to metal production and/or trade was found.

One of the more puzzling statements in the *Periplus* is the fact that copper was exported to the Indian subcontinent. More specifically Western copper (together with tin and lead) via Roman Egypt was shipped to Barygaza and Muziris/Nelkynda[61]. Pliny states that India

[55] A.K. Biswas (1996: 307) says: "Meliephtha Khalka, *literally copper cooked in honey, possibly means sheets of ductile or soft copper … This ductile copper in thin sheets was called '*honey copper' *because the sheets were shaped like honey-cakes. Ductile copper in Roman times generally meant an alloy with 5 to 10% lead*".
[56] Treister, 1995: 20; Jurisic, 2000: 41.
[57] Parker, 1992: 18-19.
[58] Treister, 1995: 19 & 25.
[59] Casson, 1989b: 18-21 & 53; Habashi, 1994: 61.
[60] Avanzini, 2002: 14-15 & 20.
[61] Casson, 1989b: 22.

lacked copper (as well as lead) and exchanged precious stones and pearls for those metals. But the Indian subcontinent is not void of copper sources and Ptolemy speaks of numerous copper mines in further India[62]. There are deposits near Ajmer in Rajasthan that, if worked in ancient days, would have been convenient for transport to Barygaza and deposits near Madras that would have been convenient for Muziris/Nelkynda, to mention only the closest sources. According to H. Chakraborti there is clear evidence that either these or other deposits were worked[63]. Moreover the *Periplus* itself reports that Barygaza who took-in copper, did also sent back out shipments of copper to Apologos at the head of the Gulf and to Omana, a port passed on route there[64]. The (much) later *Geniza documents*[65] also refer to trade in metals from India and regular export of brass and bronze vessels.

So the information on the origin of the copper used in India is contradictory. A possible explanation could be that during the 1st AD (when the *Periplus* was written) India was not, or to a limited extent, producing copper and depended on the West for the supply[66]. This could be due to hostilities in NW-India connected with the settling of the *Kushans*. A situation that might have improved by the time of Ptolemy, and by the time of the Geniza documents export of copper (products) was normal[67]. A second possibility is that India imported a surplus of copper, only to re-export it to the ports of the Gulf. Due to the fact that war broke out between the Roman and Parthian Empire it is possible that some more direct trading routes were suspended and this indirect trade was established[68].

Only one wreck in the Mediterranean (Les Magnans B - France) carried small yellow ingots (20-50 cm long) of plano-convex form. Analyses showed that these contained 79% of copper and 21% of zinc, making it brass. There is no good dating evidence for the wreck but a date of at the earliest in the second half of the 1st c BC is suggested. That date would correspond to the general chronology of the early use of brass. An alloy with such high zinc content was typical of the Augustan period, when an alloy like this was used for coining *sestertii* and *dupondii* at two Imperial mints (Rome and *Lugdunum*)[69]. This shows, based on the evidence available, that brass was not a very common trade item. Copper ingots and copperware on the other hand are the second most (after lead ingots) encountered cargoes on wrecks dating from the 2nd c BC to the 2nd c AD, with the greatest frequency in the 1st c AD[70].

3.5.3. Lead

The same lack of clarity on the copper trade to India is seen for the trade in lead. Pliny states that India has no lead but exchanges precious goods for it. India required supplies of base metals chiefly for the native currency, which was mostly of lead alloyed with small amounts of copper and tin. Lead was also alloyed with a little tin and made in thin foils for the manufactory of mirrors. Lead was produced in quantities in the Roman Empire as a by-product of the process of extracting silver from argentiferous lead ores and it was extensively used for drainpipes, aqueducts, etc. The sole market for it outside the Roman Empire seems to have been India and the *Periplus* lists lead among the imports arriving to Barygaza on the NW-coast and to Muziris/Nelkynda on the SW-coast of India. This imported metal might have been supplement with indigenous supplies from Rajasthan.[71] It is also possible that lead ores

[62] Warmington, 1974: 267-268.
[63] Chakrabarti, 1966: 253.
[64] Casson, 1989b: 18 & 21.
[65] The Ginezah is a kind of storeroom in a synagogue where also documents of secular nature were deposited (e.g. on the Indian trade). These *Geniza* documents span a period from as early as the 9th c till the 19th c AD.
[66] Warmington, 1974: 267-268.
[67] Biswas, 1996: 309.
[68] Chakrabarti, 1966: 253 ; Warmington, 1974: 268.
[69] Treister, 1995: 23; Treister, 1996: 350-352.
[70] Jurisic, 2000: 43.
[71] Chakrabarti, 1966: 274; Warmington, 1974: 267-268.

were worked in India but solely for the extraction of the silver it contained, regardless of the waste product[72].

Lead was a relatively uncommon metal in ancient India, until the exploitation of the large sources at Zawar in Rajasthan, and its use in coinage suggest its *pseudo-precious metal* status. N.J. Seeley and P.J. Turner examined Indian lead coins and found two distinct isotopic groups. The LIA of the first group match those of the Sardinian and Spanish lead sources exploited by the Romans. This substantiates the statements of Pliny and in the *Periplus* that lead was import into India during the 1st c AD. The importance of the Spanish and Sardinian mines staidly declined from 50 AD onwards because of the opening of the British lead mines. These were more economical to work and dominated the markets by about 70 AD. The LIA signature of the second group (dated slightly later) falls close to those obtained for ores from Zawar, implying the use of indigenous metal.[73]

The amount of lead found at ed-Dur is rather unusual, since it seems to be rare on other sites. Here I would just like to mention the small lead *bulla* excavated at ed-Dur imitating a Roman coin of Augustus or Tiberius that was most probably produced in India.[74]

The trade of lead ingots in the Mediterranean has been recorded already from Hellenistic period (4th – 1st c BC). Several shipwrecks yielded lead ingots mainly of Greek origin cast in oval moulds resembling *Pinnas nobilis* shells. They could weight up to 40 kg. These ingots reflect the trade in lead between the Iberian mines and Italy via Gaul. During the 1st c BC and 1st c AD lead from Hespania dominated the Mediterranean, followed by that of Britain. Three quarters of the shipwrecks with cargoes of lead are dated to the 1st c BC and 1st c AD. A smaller number of lead ingots are found at Roman shipwrecks as part of the ships equipment, since lead was often used for minor repairs during a voyage.[75]

The Arabian geographer al-Muqaddasi writing in the 10th c AD mentions the export of lead from Yemen to Oman[76].

3.5.4. Silver

The *Periplus* states that on the African coast and the 'far-side' ports the import of staples products was predominant but some imports were more costly, among them silverware. Also Arabia took in staples but the royal courts and the courts of local governors also desired a whole range of high-priced items among them again silverware.[77]

Although there are remains of silver extraction in antiquity, the import of silver was important in the history of India. Pliny referred to India as a country where silver was obtained through trade with the Romans. The *Periplus* records that silver (and gold) plate was imported at Barbaricum, and that silver (and gold) coinage was imported at Barygaza. Silver entered the ports by the sea-route from the west what not necessary means that the silver was from Mediterranean origin. From the coast the silver could be moved further up the Indus to Taxila (the Parthian city of Sirkap). The Parthians however controlled large part of Afghanistan where argentiferous lead ores are abundant. There can then also be little doubt that they obtained their main supply of silver from that part of the world.[78] In the later Sasanian period silver was mined within their territories and principally used throughout the empire for the

[72] Casson, 1989b: 28.
[73] Seeley & Turner, 1984: 331.
[74] Haerinck, 2003: 205.
[75] Treister, 1995: 19; Treister, 1996: 347-349 & 390; Jurisic, 2000: 42.
[76] Weisgerber, 1980b: 119.
[77] Casson, 1989b: 20-21.
[78] Chakrabarti, 1966: 278-279; Casson, 1989b: 21.

purpose of minting coins. Both Bahrain and Oman appear to have imported silver at that time.[79]

A different way of passing silver (and gold) from the West to India was under the form of coinage. Apparently relatively large amounts of coinage went to Muziris/Nelkynda. In India this phenomenon is archaeologically attested by numerous coin hoards, especially in S-India. The north is much poorer in Roman coins. One explanation could be that in the north the coinage was re-melted to produce local coins and that in the south actually Roman coins were circulating. An alternative explanation is that the north was more involved in barter trade whereas the south wanted cash payments.[80] Being was it is this is a secondary stream of metal that went from the West to the East that was important enough to be a concern to the Roman Emperor Tiberius.

3.5.5. Iron

The *Periplus* mentions that Adulis, Malao and Avalites on the E-Africa coast as importers of Roman wrought iron, next to finished ironware and tools such as axes, adzes and knives. Adulis used this iron to manufacture points for hunting and war spears and, in addition, imported Indian iron and steel[81].

Pliny's *Natural History* mentions the import of iron and steel from Seres and that *Seric* iron (*Sericum ferrum*) is considered to be the best followed by Parthian iron. In this case Seric does not refer to China but to an intermediate source for which the Ferghana Valley, NE-Iran and the ancient southern Indian Kingdom of Tamil Chera have been suggested.[82] From the possible regions mentioned Tamil Chera can be ruled out. The people from this kingdom appear in the *Periplus* as the Cerobotha. Their chief port, Muziris, was an active centre of shipping towards Arabia and Roman Egypt. Iron is however not mentioned among the product exported from there. While some of the Indian iron might have been shipped through the Cheraean ports, it is probably true that most of it went through the port of Barygaza. Much of the Roman knowledge on India came apparently from Sri Lanka and the origin of Indian iron attributed to the Chera Tamils, might have been the result of a confusion of the name of *Chera* and that of the *Seres* of Turkmenistan.[83] Additional argument to also exclude China from the list can be found in the Chinese sources themselves, who refer to steel as coming from India and Persia[84]. This leaves us with the large region of NE-Iran and Turkmenistan as the possible provider of good quality iron (see *Chapter 8* for a more detailed account related to crucible steel), a role they fulfilled later on in history. The fact that Parthian iron is mentioned on the second place implies that Iran was at least involved in the trade, if not the actual manufacture, of good-quality iron and/or steel[85].

In the Roman world iron was probably a relatively low-value commodity and it is likely that most was moved by water, travelling by river to transhipment points for short land transport or longer sea transport. Evidence for regular transport of iron by sea has been provided by a number of Roman wrecks off the coasts of Sicily, S-France and NE-Spain. These 15 wrecks date from between the 2nd c BC to the 5th c AD. Iron was mostly transported as bars. The nature and patterns of trade between the Empire and the 'barbarian world' is much less understood. But we do know that during the 2nd half of the 2nd c AD the Roman Emperors

[79] al-Naboodah, 1992: 86.
[80] Casson, 1989b: 30-31.
[81] Casson, 1989b: 20 & 28.
[82] Schoff, 1915: 236; Srinivasan, 1997: 111.
[83] Schoff, 1915: 236-237.
[84] Casson, 1989b: 114.
[85] Gilmour, 2000b: 46.

Marcus Aurelius and Commodus subjected the import of Indian iron and steel to customs duty.[86]

The references to iron and steel in the *Periplus* and by Pliny have often been interpreted as being to a special kind of iron, i.e. good quality steel or even crucible steel. This is based on the idea that if an otherwise cheap bulk material is transported over a long distance it had to be something special, since the Roman importers were quite capable to produce ordinary iron themselves. No detailed descriptions of the antique cargoes survived, but later (Late Medieval and early Post Medieval periods) documents indicate ordinary iron was shipped in considerable quantity and at low unit cost especially to the Middle Eastern markets. It should not be forgotten that the bloomery process requires large quantities of charcoal and that timber was a rather scarce and by consequence rather precious resource in the Middle East. S-India on the contrary was very heavily wooded so iron could be produced much more cheaply over there.[87] Whatever the quality of the metal, the *Periplus* shows that the export from the Indian subcontinent started as early as the 1st c AD and the importance of India would only grow over time.

Written sources indicate that the Yemen had become an important region for sword making by the late Himyarite period (6th c AD), and they hint towards the idea that the basic iron used was crucible steel, partly made locally and partly imported from the Indian region[88]. Al-Kindi (9th c AD) described the variant sources where iron for swords was production and where finished blades were made. Places as far apart as Yemen, Sri Lanka, Ceylon, N-India, the Ferghana Valley, Khurasan, Fars, Egypt, Damascus, S-Iraq and the region around Baghdad were all involved in a big trading network, exchanging raw materials and finished products[89]. Marco Polo in 1298 mentioned Kerman in Persia as making all the equipment for a mounted warrior. Till the 19th c AD a considerable manufacture and export of arms from Persia, in particular from the Eastern province of Khurasan is well documented. This changed over the 19th c AD as European manufactured goods replaced Persian products and Persia became an exporter of raw materials instead.[90]

Such goods almost inevitably passed through the two main funnels of Muslim Indian Ocean trade, the Bab el-Mandeb and the Straits of Hormuz (respectively the entrances to the Red Sea and the Gulf), on their way to destinations further west and north. During the 10th c AD Oman was described as "the *emporium* of the world", of which it was said that "all commodities of the east, west, south and north are brought to this town and from there carried to different places".[91] An account of commerce in the Gulf written in 1790 lists iron as one of the many categories of imports at Muscat from the Indian subcontinent. In 1835/6 iron reached the ports of the lower Gulf (Ras al-Khaimah, Umm al-Qaiwain, Dubai and Abu Dhabi) from Bombay and the Makran coast, and by the early 20th c AD iron ore and tools were being imported in sizeable amounts by the Gulf countries from various European producers.[92]

3.5.6. Tin & pewter

The questions on the transport and origin of the tin used for the bronzes in early times is a notorious one, but not so much an issue in the 1st c AD. Tin was exported by the Romans to Avalites, the "far-side" ports, Qana', Barygaza and Muzis/Nelkynda. There are however rich deposits of tin in Burma, Thailand and Malay some of which were exploited in ancient times,

[86] Cleere, 1995: 214-215. For examples on shipwrecks see Treister, 1995: 27-34; Treister, 1996: 353; Jurisic, 2000: 43.
[87] Craddock, 2003: 243.
[88] Hoyland & Gilmour, 2006: 54 & 59.
[89] Hoyland & Gilmour, 2006: 54-56.
[90] Schoff, 1915: 233; Elgood, 1994: 2.
[91] Nicolle, 1983: 234 (quoting Hudud al'alam, 148).
[92] Potts, 1990b: 273; Mouton, 1992: 204.

but India does not seem to have had contact with these places at the time of the *Periplus*.[93] In the Mediterranean tin ingots are only found in a limited amount of Roman shipwrecks[94].

Pewter (lead-tin alloy) ingots within the Roman Empire are only reported from the River Thames. There six Roman ingots were found at Battersea, dated to the 1st c BC – 1st c AD. One ingot contained 95% of tin, three around 67% and two with about 52% of tin. It is suggested that these compositions were not randomly chosen.[95]

[93] Casson, 1989b: 28.
[94] Treister, 1995: 23-24.
[95] Treister, 1995: 30.

3.6. Overview imported goods at ed-Dur

• **Introduction**
It is not my intention to give an exhaustive overview of all foreign objects found at ed-Dur since this would overlap with the topic of A. De Waele[96]. This short summery is only based on the already published data and serves to give an impression of the trading network existing at that time. We should however keep in mind that trade contacts need not be direct and objects could have made a long *down-the-line* trip before ending up at ed-Dur.

• **Glass**[97]
D. Whitehouse studied the glass vessels and fragments from ed-Dur, which make up about 8% of the total amount of registered objects (pottery not included) and about 10% of all registered samples. Technologically two types of vessels were distinguished, *cast* and *blown*. All fragments formed by casting have abundant parallels in the Roman Empire and there are no indications that examples found outside the frontiers of the Empire were made near their find-place. Similarly, among the blown objects many have Roman parallels and it seems likely that they too were imported from the Mediterranean. Some objects (8 in total) however have a likeness to Roman objects but are equally close or closer to parallels from Mesopotamia, and they may well be Parthian or Sasanian. However since much more is known about the glass produced in the Roman Empire around the 1st c AD than about the contemporaneous glass production by Rome's eastern neighbours, care has to be taken in assigning all Roman looking products to the Roman Empire.

The dating of these vessels depended entirely on dated parallels from the Roman Empire. In the case of the cast vessels the first specimen could have arrived at ed-Dur as early as 25 BC or as late as 25-50 AD. The last cast vessels to have reached the site can be placed as early as *ca*. 25 AD or as late as the early 2nd c AD. For the blown vessels many were or could have been made in the 1st c AD but a few may be significantly later (2nd c AD).

The presence of considerable amounts of glass from the Mediterranean region at ed-Dur, but also at Mleiha, shows that it was a frequently 'imported' commodity. D. Whitehouse suggests three possible ways of contact. The first option is the arrival by sea from Egypt, the second option is that the glass was exported from Egypt to India and then re-exported to the Gulf and the last possibility is the overland route to the head of the Gulf (Characene) and subsequent shipment to the Arabian Peninsula, or of course any combination of routes.

Two types of evidence (literary and archaeological) show that the Roman glass could have arrived at ed-Dur from Egypt. The literary evidence indicates that Egyptian ports in the Red Sea were already engaged in long-distance trade at the period. Pliny, Strabo and the *Periplus* write about the trade between Egypt and India. Moreover the *Periplus* lists glass objects among the goods carried by Roman merchants. Archaeologically there is some evidence at Leukos Limen, a Roman harbour in the Red Sea, where glass fragments were found with parallels at ed-Dur. A powerful objection to the idea of the Red Sea route however is that the *Periplus* does not seem to mention the Persian Gulf as such, what led to the idea that Roman merchants were not active in the Gulf. The re-exportation from India on the other hand is possible since the Gulf was frequented by Indians.

• **Ceramics**
As on many archaeological sites ceramics were the largest group of artefacts encountered at ed-Dur. Pottery can be a useful indicator for contacts, since next to it's possible intrinsic value it is often traded indirectly for the goods that it contained. The study of the ceramic

[96] A. De Waele is currently preparing her PhD-dissertation on all the small finds (except the iron objects) from ed-Dur.
[97] Whitehouse, 1998: 59-67.

from ed-Dur was recently finished by K. Rutten and presented as a PhD[98]. Here only a short summary of the conclusions and the possible places of origin is given.

The first large group of ceramics present are *locally produced ceramics*. Some caution is warranted however since no actual production centres are known. The ceramic assemblage at ed-Dur is totally different from that of the earlier Iron Age, but this can be explained by the three centuries of occupational hiatus between the Iron Age and the re-occupation of ed-Dur. Mleiha had a more continuous habitation and there it can be seen that the assemblage went through an evolution away from the Iron Age material. The raw material used however is similar to that of the Iron Age since there are only a limited number of places were clay can be found in the region. K. Rutten proposed three different production regions who got their clay from wadi's and sedimentary fans on both sides of the Hajjar Mountains. There is a clear difference between the assemblage of ed-Dur and Mleiha. This could be due to the different sample size studied (the exact amount studied from Mleiha is unclear) and the slightly different period that the sites peaked, but it is probably not caused by the different geographical location or the access to different production centres. There is a limited but clear link to the Samad culture ceramics leading to the hypothesis that there was only sporadic contact.[99]

The majority of the studied ceramics were imported however (72% of the total), mainly from S-Mesopotamia (42,4%) and NE-Arabia (23,2%). The Indian subcontinent represents 3,6%, SE-Iran 2% and the Mediterranean 0,3%[100].

- Large numbers of Parthian glazed pottery (40%) were present. The main producer of this type of ceramics was Characene. The multi-functional and high quality table ware was a wanted product, the transport vessels (for date-wine, bier, oil, fat, honey, dried fruit cereals, …) on the other hand point to other needs and forms of contact.[101]
- A particular group in the ed-Dur assemblage is what is labelled *Thaj ware*, a type of ceramic well known throughout NE-Arabia[102]. Strange enough NE-Arabian ceramics are not present at Mleiha[103].
- Little bowls in a yellow ceramic have exact parallels at the site of Janussan North and South, Bahrain. Other examples may be found at Qala'at al-Bahrain, Thaj and on Failaka[104].
- Indian pottery is present under the form of *Red Polished Ware.* The exact date of this kind of ceramics is not clear and can vary between the 1st c BC to the 5th- 6th c AD, nor is their exact place of origin known[105]. It is strange that Indian ceramics (mostly cooking ware) are not present at Mleiha. K. Rutten speculates that these wares were left behind by Indian sailors as personal items after a period of staying[106].
- Eastern connections are shown by the common occurrence of *Namord ware* from SE-Iran or Baluchistan[107].
- A buff-coloured sherd of the body of an amphora was discovered in Area Z, dated to the 1st c AD with a (part) of a Roman inscription. Stamped inscriptions of this form, on whatever part of an amphora, are common on a whole series of Mediterranean *amphorae*

[98] Rutten, 2006.
[99] Rutten, 2006: 138-140.
[100] Rutten, 2006: 151.
[101] Haerinck, Phillips, Potts & Stevens, 1993:187; Whitehouse, 1998: 67.
[102] Haerinck, Phillips, Potts & Stevens, 1993: 187.
[103] Rutten, 2006: 421.
[104] Salles, 1984: 243-244.
[105] Kervran, 1996: 38-39; Whitehouse, 1998: X (Preface by Haerinck).
[106] Rutten, 2006: 421.
[107] Haerinck, Phillips, Potts & Stevens, 1993: 187.

produced between the late 2nd c BC and 2nd c AD or later, particularly in North Africa, Spain and Italy.[108]

Sigillata ware is present in the form of *Eastern Sigillata A (ESA)*, *Eastern Sigilatta B1 (ESB1), Eastern Sigilatta C (ESC)* and *Italian Sigilatta*. The exact provenance of *ESA* remains unknown but recent research suggests that it was probably produced at several centres on the Syrian-Lebanese coast and in the Syrian interior. This type of ceramics appears on the oriental Hellenistic market around the middle of the 2nd c BC and quickly dominated the production of fine, red-slipped wares in the Levant until the end of its manufacture around the middle of the 2nd c AD. Archaeological confirmation is still lacking for the precise identification of the origin of *ESB1*, current data points to the region of Tralles/Caesarea in western Asia Minor. The distribution is largely restricted to the Aegean region and Egypt. Based on *comparanda* a date between 25 BC and 75 AD is suggested. *ESC* originated from the region of Pergamon in Asia Minor. The typical composition and colour of the fabric and slip dates mainly from the last third of the 1st c BC, and is characteristic of the Augustan series, produced up to the 2nd c AD. The geographic distribution of *ESC* is restricted to the Aegean region and Asia Minor, with occasional finds in Italy, Cyprus and S-Russia. *Italian Sigilatta* is very common throughout the whole of the Mediterranean region, the type of the late 1st c BC and 1st c AD is completely absent at sites in Mesopotamia and the Gulf with the exception of ed-Dur. Sigilatta from various regions in Italy are, however, present in small quantities in S-Arabia and on the E-African coast and at Arikamedu. It is very common at Myos Hormos and Berenike on the Red Sea Coast. They are dated within the range of the end of the 1st c BC till 70 AD.

Of the six fragments of moulded lamps four originated from the Levant and the other two could as well be from the Levant as from Egypt. They date to the 1st half of the 1st c AD.

A last type of sherds encountered are examples of the so-called *Roman green lead-glazed ware*, which was produced in small quantities from the late 1st c BC onwards at different centres in Asia Minor the examples of ed-Dur most probably originate from SE-Asia Minor). The date from the last quarter of the 1st c BC to the middle of the 1st c AD, with diminishing production continuing into the 2nd half of the 1st c AD. [109]

- Only three fragments originated from S-Arabia, showing the limited contacted with that region.
- Letters were attested on some vessel fragments. They belong to two alphabets, old south Arabian and Aramaic. The use of both alphabets for inscriptions is not unusual for this period and is connected to the use of the Hasaitic dialect in N-Arabia and the Arabian side of the Gulf, and the spread of the Aramaeans from Mesopotamia from the 1st c AD onwards.[110]

General conclusions based on the study of the ceramics are[111]:

- The ceramics point to an intensive exchange and transport of local goods between the different regions of SE-Arabia. Agricultural products are brought from the northern and central zone to ed-Dur. This is mainly done over land routes. These local products were used to feed the population of ed-Dur and were probably exchanged for sea-products and to a lesser extent imported products.
- The import of large quantities of ceramics and the flowering of the sea-trade has had a profound impact on the local ceramic production. This is seen in the flexibility of the potters by change in the pottery shapes and the production of larger quantities of transportable vessels.

[108] Papadopoulos, 1994: 276-278.
[109] Rutten, 2006: 9, 14-17 & 151.
[110] Rutten, 2006: 60.
[111] Rutten, 2006: 427-428.

- The exchange on a local level encloses a small quantity of imported ceramics that are spread or exclusively via ed-Dur or by other local networks. Mleiha is the main consumer of these imports and possibly functions as middlemen for local and import products.
- The ceramic collection shows a clear involvement in an intensive interregional trade, in which ed-Dur focuses on the exchange by sea-trade between Characene and the Indian Subcontinent. This includes a direct supply of large quantities of Mesopotamian, Bahraini and NE-Arabian ceramics and an indirect supply of Mediterranean pottery. Via the coastal S-Arabian route ed-Dur gets a direct supply of S-Arabian pottery and an indirect one of S-Indian and Mediterranean material. The sea-route provides a direct supply from NW-Indian and an indirect one of Mediterranean material. There is also a direct interaction with the coasts of SE-Iran. The ceramic assemblage is by consequence very complex, dynamic and interwoven and the result of contacts mainly made by the two main sea-routes, but also along smaller regional routes. The international trade is not only oriented in supplying local use and distribution to the inland, but it is highly likely that some of the products are also redistributed back in the same network.
- The fact that large amounts of ceramics from the N-Gulf are present shows a clear link to that region. A regular transport by ship is the only good explanation for their numerous presences. The large number of Bahraini material shows the importance of this island in this wider network of Gulf-trade[112].
- Two main groups of Roman fine wares can be distinguished. The first comprises the *ESA* vessels from Syria and Palestine. A movement of this pottery from its region of production along routes through Zeugma or Palmyra in the direction of S-Mesopotamia is suggested by K. Rutten. The distribution within the Gulf is unclear and this is partly due to the lack of evidence from Characene. This type of ceramics is however absent in the Gulf with the exception of Mleiha and ed-Dur. The type was however also widely distributed in the E-Mediterranean, so a trajectory via the Red Sea to the S-Arabian ports and from there to ed-Dur cannot be excluded.

  The second group comprises *ESB1*, *ESC*, *Italian Sigillata* and *lead-glazed ware* display a different and more specific pattern of circulation, beginning in the reign of Augustus and reaching its greatest extent during the 1st c AD. Almost none of these fine wares have been found in Parthian Mesopotamia or the Gulf. They are also only present in small amounts in Syria and the Levant. A direct shipment from Alexandria is most likely.[113]

Based on the ceramic collection an occupational date for ed-Dur from 25 BC till 125 AD is proposed. Common ware from Mediterranean origin disappear slightly earlier, between 75 AD and the end of the 1st c AD. *Amphorae* follow at the end of the 1st c - beginning 2nd c AD. This earlier end date corresponds largely with the dating of the glass, of which the bulk dates from between 25 BC and 75 AD.[114]

• Stone vessels[115]

Ch. Zutterman studied the softstone vessels of ed-Dur and, next to one 3rd millennium vessel fragment and 11 Iron Age fragments, 49 artefacts are dated to 1st c BC – early 2nd c AD (PIR-C) and have *comparanda* from all over the Arabian Peninsula. Among the sites mentioned with similar material are: Site 208-95 (Eastern Province survey, Saudi Arabia), Thaj, Tarut, Jazirat al Ghanam, Asimah, Sharm, Mleiha, Samad, Maysar, Al Bustan, Rawdah/Muqatta, Sama'il for the smooth surface dark grey vessels and Site 6 (Central Province survey, Saudi Arabia), Timna and Hajar Bin Humeid for the rough surface dark grey vessels.
Due to the fact that comparable softstone vessels have been found at other sites it is clear that these items were traded and retained a certain value. It is possible to distinguish two

[112] Rutten, 2006: 420.
[113] Rutten, 2007: 17-18.
[114] Rutten, 2006: 395.
[115] Zutterman, 2003: 84-86.

import routes. The first and most important originated in present-day Oman and led to ed-Dur, most probably via Mleiha. The second, which brought rough, undecorated softstone vessels with chisel marks, originated in S-Arabia. Although restricted in numbers, the ed-Dur assemblage is part of the grey softstone collection which was traded during the last 3rd c BC and the first 3rd c AD. It is possible that the popularity of other materials like glass contributed to a decline in the production of softstone vessels in the late pre-Islamic era.

The last stone vessel to be mentioned is a bell-shaped calcite vessel of S-Arabian origin[116].

• **Figurines**[117]

Eighteen human and animal figurines were found during the work of the Belgian team. Most of them are surface finds without a clear context and those found in the excavated trenches had no special context. Here we will only mention the figurines that could be linked to reference material found on other sites.

- BR 077 is a hand-made bird figurine with parallels in the Seleucid contexts of and at Babylon and Seleucia-on-the-Tigris and Thaj.
- A hand-made neck fragment of a camel figurine was found (BR 040). This figuring most closely resembles a figurine from Uruk of Iron Age (early 1st millennium BC) date, where several camel figurine fragments were found in and around houses. Because of their large numbers they have been interpreted as toys. At Susa a somewhat similar figurine identified as a horse was found, dating to the Sasanian period. Closer to ed-Dur several rather similar fragments have been found during excavations and surveys in various parts of Arabia, but little information is available as to date the archaeological context. Comparable camel figurine fragments material were found at Dumat Al-Jandal, at Rumeilah (near Al-Ain), on the al-Madam plain (interior Sharjah, during survey), Tell Khazneh (Failaka Island), Muweilah (Iron Age II), Thaj and on the surface of Ain Jawan (west of Tarut Island).
- Figurine M 035 (a human head) has some clear parallels with other Arabian and Mesopotamian finds dated to the first half of the 1st millennium BC. Mention should be made of over 20 Late Dilmun human figure fragments found by the Danish expedition and the more than 100 fragments found by the French expedition at Qal'at al-Bahrain. The French objects were found in a trace layer that has been associated with a cultic space of the 10th - 7th c BC. They were labelled votive because of their high number and the presents of an incense burner-like ceramic support. Significantly, in the vicinity of one of the ed-Dur altars, three similar objects were found, that were interpreted as incense burners. This figure also shows close parallels with human figurines found at Tell Khazneh on Failaka (dated to post-Isin II/Pre-Hellenistic). Other figurine fragments with similar facial features are known from Seleuco-Parthian layers of Uruk and Susa.
- Figurine AV 163 (stylised human head) has similarity with Thaj-material (date between 3rd c BC and 0). Outside Thaj figurines from this type are said to come from Wadi al-Jawf at Yemen and an example somewhat resembling from Mleiha.
- BR 078 (torso of a human figurine) is of similar type to AV 163 and has parallels at Thaj, Qal'at al-Bahrain and with survey material of al-Ukhdud (Najran).
- BR 076 (mould-made torso) is without doubt an import with Hellenistic parallels found at Failaka.

The human figurines could not have been made in a local workshop and their close parallels to other figurines present at other sites on a much more significant scale, suggests that the ed-Dur figurines were either imports or "souvenirs" brought or received elsewhere.

[116] Haerinck, 2003: 205.
[117] Daems, 2004a: 2-4; Daems, 2004b: 94-96.

• **Coins**
The information connected to the coins, more specifically on the foreign coins will be presented in the appropriate *Chapter 7*. Here it will suffice to sum up the number of foreign coins with their place of origin that are known from ed-Dur: Charance (11 coins), S-Arabia (2), Persis/Parthian Empire (4), Nabataean/E-Mediterranean (6), Roman (4), Indian (5) and from Ghallah Island 2 Sasanian coins.

• **Beads**
The beads excavated by the Belgian team were analysed by K. De Corte in the frame of an undergraduate thesis at the *Department of Geology and Soil Science – Ghent University.*[118] Next to the scientific analyses, one chapter was spent on pinpointing possible sources where the raw material or the finished beads originated. Of these samples only the mineral garnet could be allocated to a well-defined source area. Based on the colour and the spatial spread of inclusions one beads could be linked to Sri Lanka, five others to Tanzania[119]. In the *Periplus* Tanzania is mentioned as *Anzania*. A small elephant bead/pendant in a private collection could be originating from the same region, although India/Sri Lanka might be a more likely place of origin[120].

A second very distinct group of beads are the etched carnelian beads. Banded agate and carnelian does appear in SE-Arabia (i.e. the Emirate of Ras al-Khaimah) and there is some evidence of open-cast mining. However there is no evidence of a major stone bead production in the area and certainly not of etched beads. The etched carnelian beads therefore can be seen as an import product from workshops on the Indian Subcontinent, whether through direct or indirect contacts[121].

Numerous other beads (glazed frit in different shapes e.g. altars, fists, grapes, melon-shaped, etc.) can be dated to the Parthian period and produced somewhere in the Parthian territory[122].

• **Other hints of international contacts**
Two Roman/E-Mediterranean agate intaglios were excavated. One shows the goddess Victory with a shield and lance, while the other shows a standing figure in a long, classical dress[123]. The recent excavation of a tomb at Dibba Al Hisn also yielded three similar insets[124].

The two stone sculptures of eagles found in the building of area F seem to be inspired on examples known from Hatra dating to the 2nd and 3rd c AD, where they are generally associated with the solar god Shamash.[125]

In the vicinity of one of the altars next to the temple an incense burners was excavated that still containing the remains of incense in the bowl and in the large tomb of Area AV some 20 grams of myrrh and/or frankincense was encountered, products originating from S-Arabia.[126]

Among the archaeozoological remains, two pieces are considered as imports: an antler fragment of *Dama mesopotamica* and a pharyngeal bone of a large freshwater fish of the genus *Barbus*. This genus of fish does not occur in Arabian but it was impossible to establish whether it originated from the Shatt-al-Arab or from one of the coastal rivers of present-day

[118] De Corte, 1994.
[119] De Corte, 1994: 144-147.
[120] Haerinck, 2003: 92.
[121] De Waele & Haerinck, 2006: 38.
[122] Haerinck, 2003: 204.
[123] Whitehouse, 1998: X (Preface by Haerinck); Haerinck, 2003: 203.
[124] Jasim, 2006: 227.
[125] Lecomte, 1996: 196.
[126] Haerinck, 2003: 203; Daems, 2004b: 94.

Iran. The antler fragment of the Mesopotamian fallow deer is also considered as an import. This species occurred until recently in W-Iran, S-Turkey and N-Arabia and this also seems to have been the case in prehistoric times.[127] The large *Bursa bardeyi* shells frequently encountered at ed-Dur originate from the Omani coast[128].

[127] Van Neer & Gautier, 1993: 110-113.
[128] Haerinck, Metdepenninghen & Stevens, 1992: 50.

Chapter 4. ANALYTIC METHODS

"Science is spectral analysis. Art is light synthesis."

K. Kraus

4.1. Introduction

"Choice of technique is often based upon access to instruments and cost rather than the suitability of the resulting data to solve the formulated question.[1]"

The working-space of archaeometallurgy can be defined as the study of metalworking structures, tools, waste products and finished metal artefacts. This includes the identification and interpretation of *in situ* structures, as well as the post-excavation analyses of any metal work, waste products, crucibles and moulds, … or anything that is related to the production of processing of metals. The ultimate goal is to understand and reconstruct the technologies used in the past. Archaeometallurgical investigations can provide evidence for both the nature and scale of mining, smelting, refining and metalworking trades, and aid understanding of other structural and artefactual evidence. They can be crucial in understanding the economy of a site, the nature of the occupation, the technological capabilities of its occupants and their cultural affinities. For this a wide array of analytical techniques are available and used in the different studies.

This dissertation (unfortunately) is only concerned with the post-excavation phase of the different metal samples exported to Belgium. The main questions to be answered are:

1. Which alloys were used?
2. Which processes did the copper and copper-base alloys go through?

As I was a novice in this research field the analytical techniques used in this study were not chosen as such, but rather offered. The main instrument used in this study is a *scanning electron microscope* (SEM) equipped with an *energy dispersive X-ray spectrometer* (EDX) for chemical analyses. This devise is user-friendly, relatively low in cost and combines two kinds of analyses, i.e. visual examination at high magnification and chemical compositional analyses. Additionally the *Department of Metallurgy and Materials Science – UGent* has extensive experience with this analytical technique. The major advantage for myself was that I could operate this microscope alone and was not dependent of anyone to do the analyses.

The SEM-EDX results are supplemented with the study of the microstructure of the samples by *optical metallographic microscope*. These two techniques form the basic core of the research. Metallography is a low-cost analytical technique that gives a good impression of the different working phases the metal object went through. This method is quite straightforward and could also be performed without any help.

During this study a second line of research was opened, focussing on the possible origin of the metal used to manufacture the objects. For this, contact was made with the *Laboratory of Analytical Chemistry, Institute for Nuclear Sciences – UGent*. Provenance studies of metals are based on the measurement of the trace elements and the lead isotope ratios present in the material. These results can then be compared to the published data of different ore fields or other object. They can also be used to compare the analysed samples amongst each other. To collect that data *inductively coupled plasma mass spectrometry* (ICP-MS) was used. Originally it was thought to use the updated version of this technique, *multi collector ICP-MS* (MC-ICP-MS), which gives results with a higher precision and accuracy. Due to unforeseen circumstances however, i.e. the delay in the installation of this new equipment, the traditional form had to be used instead. The drawback of this technique is that it is rather expensive[2] and a specialised activity, not to be preformed by a layman. Luckily D. De Muynck was very helpful on this part, investing a lot of time and accuracy in the samples provided to him. He is currently preparing a PhD on the optimisation of ICP-MS for *lead isotope analyses* (LIA). If coincidence does exist here is a nice example, the material he uses

[1] Young & Pollard, 2000: 22.
[2] Prof. dr. L. Moens and Prof. dr. F. Vanhaecke were however so kind to perform these analyses for a 'give-away' price.

for the optimisation up till then were archaeological samples. The coincidence did however not stretch as fare that they were also metallic samples, but soil samples instead. Nevertheless he is able to incorporate the findings on the ed-Dur material in his own research, creating a win-win situation for both of us.

One additional technique was used for the study of the slags found at ed-Dur, being *X-ray diffraction* (XRD). This allows determining the mineral phases present in the slag, rather than the separate chemical elements. This was performed at the *Laboratory of Soil Science – UGent*, on powdered samples. The interpretation of these results was however not as straight forward as hoped for. The lab had no experience with this kind of material since they normally analyse clays. Also the software that should 'simplify' the determination was of little help, rather the opposite actually. These rather expensive analyses were on the verge to be a measure for nothing. Luckily the human brain of F. Mees was available to make something of the data.

Other analytical techniques are mentioned throughout this PhD, but they will be briefly explained when they are brought up. This chapter is not meant to be an exhaustive in-depth technical description of the analytical techniques used. After all it was not the objective to evaluate their usefulness, since they are not new and innovative methods but established tools for archaeometallurgical analyses. The chapter serves as an introduction for the reader who is not familiar with them and to summarize the sample preparation, the analytical parameters and the problems encountered during this specific study.

Concerning the quote above the *instrumentarium* used was of course determined by their accessibility at Ghent University, and primarily by their ease in use and my ability to use them. The cost was, as always, an issue but not a major one and certainly not to that degree that it prevented the use of the desired method. Relating to the answering of 'formulated questions' I think the technique were appropriate. Not that all questions were answered, but they were at least considered to the best of my abilities. Moreover not all 'questions' were formulated at the onset of this study and many came up, as usual, along the way. The interpretation was another pair of sleeves and as a rookie in the archaeometallurgical research the insights were accumulated throughout this study. Things were learned as questions came up and many questions did come by, considering the great variety in materials that needed study. I hope the overall results, conclusions, applied strategies and techniques are also satisfying to the reader.

4.2. SEM-EDX – microstructure & chemical elemental composition

4.2.1. Introduction[3]

SEM is the abbreviation of *scanning electron microscope*. A complete explanation of the functioning of a SEM and the EDX is not presented here. Enough good handbooks and descriptions are available on the market[4]. This basic principle of this type of microscope is relatively simple, a very narrow beam of electrons is generated by heating a wire filament (as in a light bulb) to a high temperature under a strong electrical potential. This beam passes down a cylindrical *column*, around which are a series of electromagnetic *lenses*, which allow the beam to be focused and moved. At the base of the column, the electrons hit the sample and generate a number of different types of signal. These signals from the sample can be converted electronically to provide an image of the sample. The big difference with an optical microscope is that the magnification that can be reached is much larger (x1500 *versus* theoretically unlimited) and that, within a reasonable margin, the topography of the sample surface can be resolved, meaning that at the same time the *hills* and the *valleys* of the surface can be in focus.

A SEM can generate two types of images: Secondary electron (SE) images and back-scatter electron images (BSE). For this study the second type was used (see Fig. 1-1).

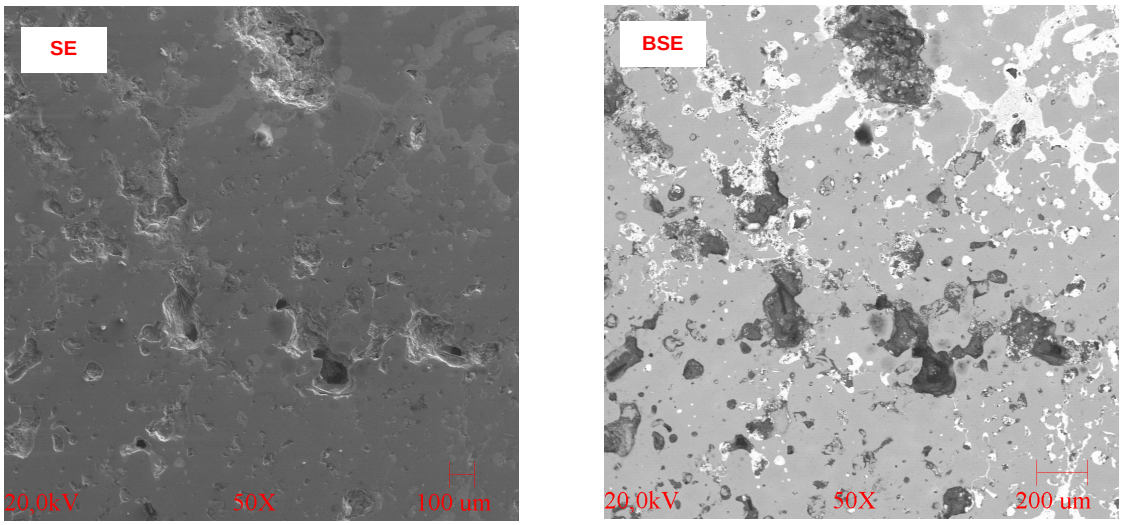


Fig. 1: SE-picture and BSE-picture of same area and magnification of sample KR 009.

• **Secondary electron images (SE)**
These are based on the signal of the electrons knocked out of the surface of the sample by the colliding electrons from the beam. These secondary electrons have low energies, and can easily be drawn into a collector from all over the surface of the sample. Stronger signals received from the *hills* than the *valleys* result in a SEM-image which shows the topography or morphology of the sample in shades of grey, grading in to one another without sharp boundaries.

• **Back-scattered electron images (BSE)**
BSE-electrons are the primary electrons of the beam bounced back from the sample. Heavier elements are more efficient electron reflectors than light elements, thus areas rich in a heavy element such as iron or lead appear brighter than those rich in a light element, e.g.

[3] This introduction is largely based on Freestone, 1985: 67-68 and Northover, 1998a: 94-113.
[4] To give only two (consulted here): Friel, 1995 and Gabriel, 1985.

carbon, silicon or oxygen. BSE-images show the make-up of an object in terms of the different phases or chemical compounds of which it is composed. These are usually separated by sharp boundaries, which show up in microphotographs as sharp changes in the grey values.

- **EDX or EDS**

EDX or EDS stands for *electron dispersive X-ray spectrometry* or *electron dispersive spectrometry* and is the technique used for chemical analyses (after this abbreviated by EDX). This is based on a third kind of signal that is generated by the electron bombardment of the sample surface, the emission of X-rays. These X-ray spectra are analysed based on the energies of the X-ray peaks. Elements from boron (Z = 5) upwards (but carbon and oxygen do not give very reliable results) can be detected with the exception of the strongly radioactive elements. Every element has its own spectrum that can be detected down to about 0,1 weight percentage (wt%), but accuracy and precision are poor for concentrations below 0,5 wt% (Table 3).

Results in wt%	Description	Absolute error in %
100 - 5	Major elements	2 to 4%
5 - 1	Minor elements	10 to 20%
1 - 0,2	Trace elements	50% (up to 100%)

Table 3: Errors on the values obtained by EDX.

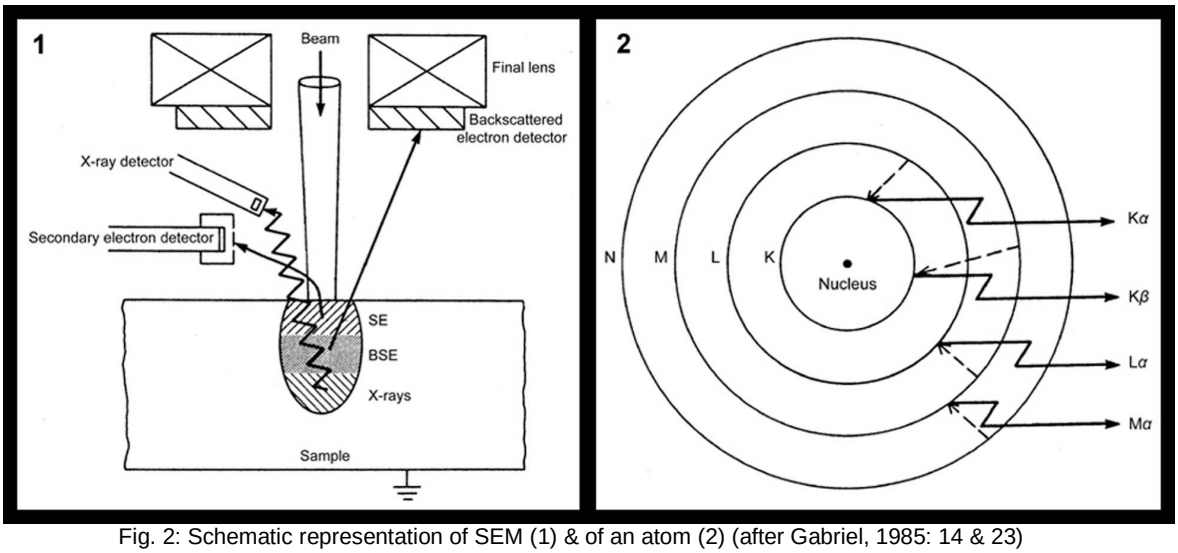


Fig. 2: Schematic representation of SEM (1) & of an atom (2) (after Gabriel, 1985: 14 & 23)

An atom is built up from a nucleus and one or more 'shells' around it in which the electrons turn. The first shell is called the K-shell, the next the L-shell, etc. When an electron of sufficient energy strikes an atom, an inner shell electron may be ejected. To return to its lowest energy state, an outer shell electron (of greater energy) fills the vacancy in the lower energy shell. In doing so energy equal to the transitions that occurred within the atom is released as an X-ray. There are three families of X-ray lines in the spectrum that are relevant to EDX: the K, L and M lines. K lines are generated from the inner electron shells of the atoms and require the highest energies to remove an electron from them (see Fig. 2-2). They also emit the most energetic X-rays. The spectrum is different for every chemical element and in this way different elements can be detected by looking at the peaks in the spectrum. The intensity of the line (peak height in the spectrum) gives the abundance of the element. Different atoms present in the sample affect the emitted X-ray signal, known as the *matrix effect*. As atomic number increases so does electron back scattering and the yield of X-rays

decreases; furthermore one atom may absorb the X-rays produced by another, or actually be excited to emit X-rays by an X-ray generated in another atom. Statistical methods have been developed to correct this. There is however no single perfect solution and, since different systems use different physical assumptions, there can be small variations in the results. The correction standard provided by the analytical software used here was the *ZAF-correction*[5].

Because raw data are impossible to compare, these are submitted to a modification process. This *normalisation-process* manipulates the elemental data for each individual to sum to a uniform value of 100%.

4.2.2. Microscope & analytical parameters

- **Microscope & sample preparation**

The microscope used for the analyses within this PhD was a *Zeiss DSM 962 Electron Scanning Microscope*. The software to process the chemical analyses was *EDXi 2000 analytical software package*. SEM-EDX is a semi-quantitative technique, which means that the results are not the exact composition of the sample. This is due to the fact that it is only possible to determine the chemical composition of a relatively small surface (max. *ca.* 1 x 1 mm) with a limited penetration depth of max. 1 µm. Basically this is a near surface analyses of the sample and this can differ from the internal composition and thus only a small volume of the specimen is analysed. To partly overcome this problem, or at least level the compositional differences out, it was decided to make measurements on an as large as possible area of the samples. The areas were chosen in such a way that the least corroded surfaces were analysed and at least three areas per sample were analysed in this way. The mean was calculated and was accepted as the composition of the alloy. The size of the analysed area was adapted to the size of the sample and/or analysable surface. All measurements were done at a 50 to 200x enlargement (again depending on the sample), with a 20kV current, a live time of 120 and a count rate of about 1500. When necessary specific phases or inclusions were analysed separately.

Much better results are obtained if the sample to be analysed is flat and polished, although untreated specimens can be analysed as well. The electrons and the X-rays reflected from a flat surface are far less dispersed over different directions and a higher quantity can be sensed by the detector. The samples for this study were imbedded in bakelite and submitted to metallographic polishing with increasing fineness, ending with a 1 micron diamond paste to insure a good positioning in respect to the electron beam. The only exceptions are the analyses done on the drillings from some objects. These were fixed on a small holder with a sticky carbon layer on top. These samples were physically pressed as flat as possible. The analyses were found acceptable since the count rate was well within the desired percentage.

The slag fragments asked for an additional treatment since they are not or poorly conductive. These samples were coated with a thin layer of gold before SEM-EDX analyses were undertaken in order to improve their conductivity and prevent the sample from charging and giving erroneous results.

- **Elements analysed**

In an initial run the elements analysed for were selected on the basis of their applicability to this study and the percentile composition was calculated taking only the following elements in account: sulphur (S), iron (Fe), nickel (Ni), copper (Cu), zinc (Zn), silver (Ag), tin (Sn) and lead (Pb). Other metallic elements were looked for but not present or present below the

[5] The factors taken into account are: the *atomic number effect* (Z), which describes the depth of electron penetration and the fraction of electrons that backscatter from the sample; the *absorption correction* (A), which describe the absorption of X-rays in the matrix and the *fluorescence correction* (F) which describe the secondary fluorescence of one element by the others present.

detection limit of EDX. If they were encountered they are mentioned in the text. Eventually only Cu, Zn, Sn, Ag and Pb were evaluated. This means that the other elements were excluded from the quantification and the normalisation to 100%. The reasoning behind this is that the metal producers in antiquity did not intentionally alloy their metals with elements such as S or Fe, they just happened top be present in the alloying metals. By excluding these elements the resulting ratios are probably closer to the intended composition. Moreover the effect of excluding these elements does not fundamentally change the results, and mostly affects the amount of copper present. Copper is however not evaluated as such, since it is the alloying element that defines the type of alloy. For zinc and tin the effects are minimal and none of the samples changed alloy group if one or the other dataset was evaluated.

For the slag a different approach was needed, since they a completely different group of material and all elements present were evaluated and normalised.

• Sulphur & lead

There is the possibility that sulphur could be present in the samples as a result of corrosion, in the form of sulphate minerals such as *brochantite* ($Cu_4SO_4(OH)_6$)[6]. Sulphur, like chlorine, could also potentially have been derived from the ground water[7].

Metallographic analyses carried out on copper-base objects from the Gulf-region have shown that numerous copper-sulphide inclusions are present in objects dating to the Bronze and Iron Age. These could indicate the use of copper sulphide ores in the production of the artefacts[8]. The presence of iron and sulphur in finished objects is related to the occurrence of these elements in the raw copper used for their production[9]. Sulphur can occur in relative great quantities under the form of *matte* (Cu_2S) in the raw copper. This is caused by a limited efficiency of secondary refinement[10].

A particular problem related to SEM-EDX is the peak overlap of the Lα Pb- and Kα S-peak. This is not a problem if only sulphur is present, but when both occur. Moreover the software package used gives very low sulphur-levels when lead is attested. It should also be kept in mined that inaccurately high lead concentrations for some samples measured by EDX, were already reported by L. Weeks[11].

Peak overlap also occurs between lead and arsenic. Arsenic was however excluded from the quantification based on three arguments. First of all copper-arsenic alloys are mainly used in the earlier periods of the Bronze Age after which they are replaced by copper-tin alloys and do not seem to make a re-entry in metallurgical history ever after. Secondly the study of L. Weeks of some of the ed-Dur material did not show arsenic present in higher values than 0,5%[12]. The percentages obtained when arsenic was quantified were all above 5%. A last argument is that these high values arsenic were exclusively associated with lead inclusions, whereas if they would have been an alloying element they should also show up in the copper matrix.

• Iron

Of all the minor and trace elements regularly found in early copper, iron is the most dependent on the smelting process and provides a good indication of the smelting process used. In primitive processes where high-grade ores were smelted at low temperatures and

[6] Hauptmann, Weisgerber & Bachmann, 1988: 36.
[7] Weeks, 2004b: 76.
[8] Weeks, 2004b: 76
[9] Weeks, 1997: 61.
[10] Weeks, 1997: 61-62.
[11] Weeks, 1997: Appendix A; Weeks, 2004b: 138.
[12] Weeks, 2004a: 234.

rather poor reducing conditions the iron minerals that may have been present were not sufficiently reduced to become incorporated in the copper. Average concentrations of around 0,05% are characteristic of such copper.[13]

In the later slagging process the situation was very different. In this process the temperatures were higher and the conditions generally more reducing, and the gangue materials were removed by the formation of a liquid slag. The principal gangue materials were oxides of iron and silica, and if quantities of both were present under suitable conditions they could react to form a liquid iron silicates slag. The gangue would very rarely be of the correct composition and a flux had to be added. If the ore was silica-rich then a flux of iron oxides could be added, if the ore was iron-rich then quartz could be added. The important point is that whatever ore was smelted a good deal of iron was present in the system. In highly reducing conditions some of the iron minerals could react with the copper going through the slag to form copper-iron alloys. Iron is soluble in molten copper, especially in the presence of even small amounts of sulphides. The majority of iron can be removed from the raw copper by simple remelting in an open crucible. The iron-rich phase will floats to the surface where the iron preferentially oxidised after which it can be skimmed off or allowed to form a crucible slag. Experiments[14] have also demonstrated the strong uptake of iron in smelting operations with mixed oxide, carbonate and sulphide ores, with concentrations of up to 8%.[15] This last bit of iron does influence the properties of the copper very little, so the smelters would not have bothered to remove it[16].

The iron present may also have been introduced as contaminate by soil and rock particles incorporated during corrosion[17].

• Nickel

The high Ni-content, which is stressed as typical for the Oman Peninsula deposits in the previous archaeometallurgical literature, is not typical for all deposits. Nickel is low in the massive sulphide deposits but is clearly concentrated with cobalt and arsenic in veins situated in peridotitic rocks (Ni up to 0,6%, Co up to 0,12% and As up to 0,2%). It is probable that the nickel levels recorded for the prehistoric bronzes from SE-Arabia are the un-intentional result of the use of such ores. The changes in the mechanical and physical properties of copper, which might have been caused by the presence of nickel, are unclear other than the fact that it may have led to a silvering in the appearance of the material.[18]

• Zinc

Measuring low zinc levels in a high copper matrix by means of EDX proved not to be as straightforward as thought. This problem was also noted by L. Weeks, who says that accurate readings are only likely with zinc levels of greater than 1%[19]. In addition to that the software used to quantify the spectra sometimes had problems in distinguishing the Kα Zn-peak. It is thought that this is due to the fact that the Kα Zn-peak is close to the Kα Cu-peak, and high copper values tend to elevate the zinc levels. This was overcome by visually evaluating the spectra. Where inaccurate high zinc levels were obtained, zinc was manually removed before normalising the composition. The samples where the zinc levels were found inaccurate and were manually excluded are indicated with a grey square table cell.

[13] Craddock, 1995: 137-138; Weeks, 2004b: 106-107.
[14] Lechtman & Klein: 1999: 515-516
[15] Craddock, 1995: 137-138; Weeks, 2004b: 106-107.
[16] Craddock & Gale, 1988: 179.
[17] Weeks, 2004: 78.
[18] Hauptmann, Weisgerber & Bachmann, 1988: 35; Weeks, 1997: 63.
[19] Weeks, 2000a: 89.

Reg. nr.	SEM-EDX Zn wt%		ICP-MS Zn wt%	
AV 083	19,33	± 0,11	17,30	± 0,9
AW 063-4	19,12	± 0,47	18,00	± 0,2
BL 014	18,92	± 0,60	19,10	± 0,6
C 079	1,08	± 0,10	0,558	± 0,006

Table 4: EDX Zn-values *versus* ICP-MS Zn-values

This problem did also cast doubt on the other zinc percentages measured, but some parallel analyses by ICP-MS and on certified samples showed relatively good correspondence for the higher zinc values (Table 4). Moreover some samples where the zinc content was manually removed, the element was only present as a trace element in the ICP-MS results.

• Corrosion products

Corrosion can introduce some elements, which would not originally have been incorporated into the metal object at its time of production and use. The process of corrosion can not only change the concentrations of elements relative to those seen in the original, but can introduce wholly foreign material such as surrounding soil and mineral particles and corrosive salts. In such case the data for these elements haven been removed prior to normalisation, and normalised compositional values are thus presented only for those elements, which would have comprised the metal object in its original form. Silicon (Si), chlorine (Cl), calcium (Ca) and potassium (K) are the major indicators of the intrusion of soil and mineral particles into the matrix, next to oxygen (O), which points to oxidation. Chlorine was sometimes present in large amounts as a result of the formation of copper hydroxy-chlorides, which are likely to form in a saline environment[20]. These elements were left out before the normalisation, but were used as parameters to see how *pure* the analysed surfaces were and if they could be used for further calculations or not. Heavily corroded pieces were also examined, but a reliable alloy composition is impossible to achieve. These were excluded for compositional data, but where possible the broad alloy group was determined, e.g. unalloyed copper, brass or bronze.

The corrosion can be a real problem when uncleaned surfaces of ancient metal objects are analysed. In ancient copper-base alloys corrosion products have usually replaced some or all of the micro-constituents. At an early stage of corrosion in some soils the tin-rich phase in bronze can been converted by local electrolytic action into spongy copper filled with tin oxide. Later on the copper turns to *cuprite* (Cu_2O) and eventually to green *malachite* [$Cu_2CO_3(OH)_2$].[21] Brass can suffer from a phenomenon known as *dezincification*, whereby the zinc may be totally lost from the surface of a brass.[22] The analyses presented here were all done on a fresh cut section of the samples, which reduces the effect of corrosion. Moreover the safety guard by evaluating the elements connected with corrosion, as described above, excluded any objects that were too severely corroded.

[20] Weeks, 1997: 75.
[21] Smith, 1981d: 85-86.
[22] Craddock, 1978: 4.

4.3. Optical microscopy – metallography of the microstructures

4.3.1. Introduction

Metallography is the scientific of the *internal structure* or *microstructure* of metals and is mainly carried out by optical reflecting light microscopy. Since this is such a common instrument an extensive description seems irrelevant. The formation of different microstructures in alloys of two metals or one metal and one non-metallic constituent (e.g. carbon in iron) can be interpreted and explained by phase diagrams (Appendix XX). These summarize the behaviour of the components in function of their concentration and temperature. More complex ternary phase diagrams can also be used.

Pure metals consist of a cell-like arrangement of polyhedral grains or crystals. This is because the atoms that make up a metal will normally arrange themselves in regular geometric form, of which the cube and hexagon are the most common. When a metal melts, however, this orderly lattice breaks down, and the arrangement of the atoms becomes random. When a pure molten metal cools slowly, small crystals begin to form from nuclei (small impurities still present) more or less evenly distributed throughout the liquid. As cooling continues more and more atoms attach themselves to these nuclei in the arrangement of the *crystal habit* typical for the metal. On yet further cooling these 'tree-like' *dendrites* will grow until they come into contact with each other, preventing any further outward growth. At this point the remaining liquid metal between the branches of the dendrites will solidify to fill the spaces. Ultimately each dendrite will become a roughly *equi-axial crystal* with its surfaces in contact with those of its neighbours. These crystals, which are irregular in boundary, are usually referred to as *grains.* If a pure metal is very slowly cooled than very small no dendritic structure will appear. If, however, cooling is rapid or uneven, as for example when molten metal is poured into a cold mould, nuclei will appear first near the mould face and the grains will grow rapidly inwards, until they come in contact with one another. The faster the cooling the smaller the dendrites formed. In a *chill cast* metal the grains near the surface are not equi-axial but elongated, their long axes being at perpendicular angles to the mould face.[23]

Porosities can be present in the metal and are the result of dissolved gases in the melt or the interdendritic holes and channels that were not completely filled. Gases can exsolve upon cooling and react with the metal to form oxides (e.g. Cu_2O).[24]

Alloys consisting of mixtures of two or more metals usually have more complex structures than pure metals. This is largely depending upon the size of the several varieties of atoms and the crystal habits of the metals. The atom size and the crystal habit of two metals may be sufficiently similar to permit the atoms of one metal to fit in the space lattice of the other. A structure of this kind may be referred to as a *solid solution* of one metal in another (e.g. gold and silver). However, although the atoms of one metal may fit into the space lattice of another in any proportions, the lattice of the solvent metal is bound to be somewhat distorted by the presence of the solute atoms, since the atoms of no two metals are identical in size. As a result, the slip planes running through the lattice become have an effect on the strength of the alloy. The strength of the solvent metal is usually increased. It is for this reason that alloys are frequently stronger than either pure constituent metal.[25]

Normally the alloyed metals have different melting points and when the molten alloy starts to cool, the component with the highest melting point begins to solidify first, resulting in a segregation of the metals. As most metals are not completely soluble in each other in the solid state, the alloy composition will rarely be consistent throughout the metal. The nuclei

[23] Hodges, 1968: 210; Scott, 1991: 5; La Niece, 1998: 114-115.
[24] Scott, 1991: 6.
[25] Hodges, 1968: 212.

and the first branches of the dendrites contain higher proportions of the higher melting metal than the in present in the liquid. As the dendrites grow, the proportion of lower melting metal in the remaining molten material increases, so that the final infilling around the dendrites is proportionally far richer in the lower melting metal than are the dendrites themselves. A dendrite can contain more of a certain metal in the centre than at the surface a phenomenon referred to as *coring*.[26]

In many cases, instead of two metals being mutually soluble in one another, they will only dissolve in one another to a limited degree. As a result two or more types of grain may be formed in an alloy, each being a solution of one metal in the other. These different solid solutions are referred to as *phases*, and usually occur when metals of rather different atom size are alloyed. Bronzes and some brasses are typical of this kind of alloy. In some instances the metals are completely insoluble in each other and are completely separated in the microstructure (e.g. lead and copper).[27]

Many alloys undergo structural changes on cooling after solidification. Some alloys will form one phase on solidification, which when further cooled will break down into other phases. Usually some of the solute metal is ejected from the space lattice of the solvent metal (they may form new solutions) and a *eutectoid* is formed. This eutectoid is often composed of alternating thin layers of the expulsed metal and the solid solution. An example of this is pearlite in steel or the alpha-delta eutectoid in bronze.[28]

If a cast metal is reheated for a certain amount of time the dendritic structure disappears, differences of composition are largely removed by diffusion, and rather regular grains are formed. This heating stage is known as *annealing*, and it causes the structure of metal to recrystallise, restoring its original ductility and malleability so that working can continue. The persistence of a cored structure with a fine-grained structure on top of the coring indicates that the *annealing* temperatures were low and the annealing times were short. This occurs when the metal was heated at temperatures below 800°C. [29]

Cold-working is the process of shaping solid metal without heating it, for example by hammering or cutting. Copper-base alloys can be easily worked at room temperature. The properties of the metal however are affected when it is cold-worked. As the alloy is hammered, bent or twisted into shape, it becomes work-hardened. This increased hardness is often desirable but it also leads to increased brittleness. The strength of a metal depends to a very large degree on the fact that the adjacent grain surfaces are cohesive and these are normally less liable to fracture than the grains themselves. Because atoms of a metal are arranged in a regular lattice, there are also planes of weakness running through the lattice. When a metal is hammered lightly whole volumes of lattice may become momentarily distorted, to return to their original position after each blow, but under heavier hammering these volumes of lattice may become permanently deformed, with the result that the crystal grain is given a new shape. This kind of distortion, known as s*train* or *slip lines*, will appear under the microscope on the etched metal in fine lines parallel in each grain and strain lines change direction from grain to grain. As a result of slipping under severe hammering the grains themselves will become flattened.[30]

If a large amount of working is required to produce a particular object, the metal must be heated between successive cycles of working otherwise it will eventually break. The new grains formed after annealing will show typical lines crossing them, called *annealing twins*. Annealing twins are mirror images with respect to lattice plane orientation, appearing in

[26] Hodges, 1968: 211-213; Scott, 1991: 5; La Niece, 1998: 114-115.
[27] Hodges, 1968: 213.
[28] Hodges, 1968: 213.
[29] Unglik, 1991: 102; De Ryck, Adriaens & Adams, 2003: 586.
[30] Unglik, 1991: 102; Hodges, 1968: 211.

structures as wide straight-sided bands either dark or light in colour depending on how the incident light is reflected. The annealing twins indicate that these objects were fabricated either by cold working followed by annealing or hot working alone.

Metal worked at high temperature has an undistorted twinned structure identical to that resulting from cold working followed by annealing. When an annealed metal is again cold-worked the annealing twins may be distorted and strain lines can occur again. Repeated cycles of cold-working and annealing will normally produce progressively smaller grains.[31]

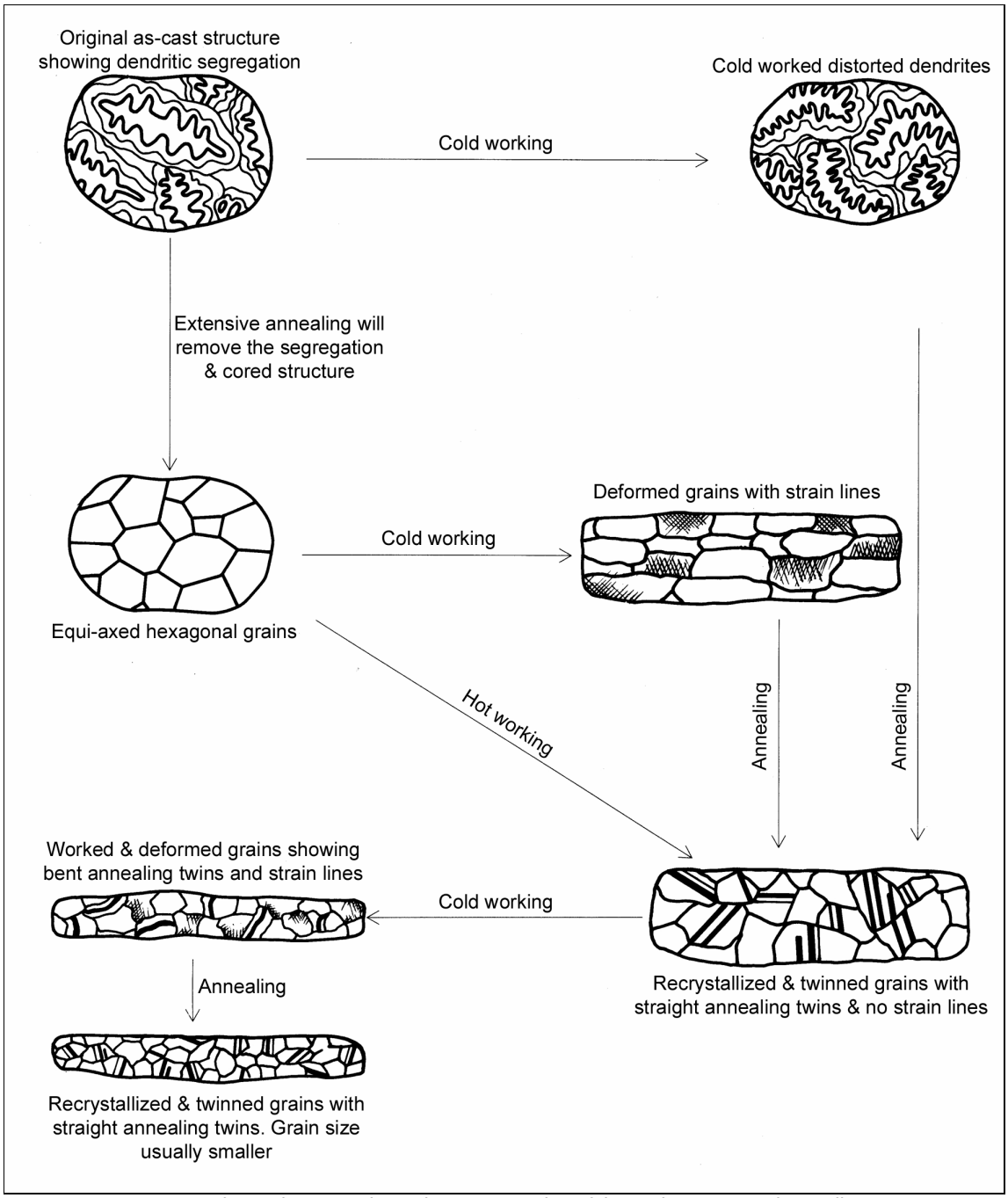


Fig. 3: shows a schematic representation of the cycles a copper-base alloy goes through when worked (after Scott, 1991: 8).

[31] Hodges, 1968: 211; Scott, 1991: 8; Unglik, 1991: 103; An., 2001: 17.

This is only a very basic overview of metallographic terms and processes that can be studied (Fig. 3). When necessary more profound information will be provided.

4.3.2. Sample preparation

The principle of the optical reflecting light metallographic microscope will not be explained here because I presume the reader is familiar with this instrument.

All samples in the original dataset could be approached in a fully destructive way, so there was little constrained in how and where samples were taken. The additional samples brought back from the Museum in March 2005 were taken with more care, since they were from preserved objects. Small fragments were detached by a small wire-cutter or saw. Additional some complete objects were brought back for sectioning at the lab and some large objects were sampled by drilling.

At the workshop samples were cut with a high speed, water-cooled cut-off wheel. The samples where then mounted in bakelite and grinded down using four abrasive papers of increasing finer grain size over which a thin sheet of water washes. The samples were then polished further on three successive motorized turning wheels to which diamond paste was added till 1 µm. Kerosene was used as a lubricant. Finally the samples were polishing with an acidic alumina suspension (Struers, OP-A suspension). This greatly increased the quality of the polished surface[32]. The objective of this polishing is the preparation of a polished surface, which on the one hand will readily reflect light under the optical microscope and on the other hand will enhance the imaging and accuracy of the SEM-EDX (as mentioned above).

After the study of the polished surfaces, the samples were etched with a solution of aqueous ferric chloride ($FeCl_3$) to enhance the microstructural structure. Etching brings out the microstructure much clearer and defined the grain boundaries, annealing twins, strain or slip lines, etc. The etching only took 3 to 5 seconds after which the surface was cleaned with methanol and dried in a hot air flow to prevent staining by the etchant and the methanol. The few iron samples that were selected for microstructural analyses were etched by different products (nital 2%, nital 4%, picral saturated and *stead reagens*) and for different time spans. None of them produced a satisfactory microstructure since the matrix was entirely made up by iron oxides that 'absorbed' the etchant and left the small metallic inclusions unaffected (see *Chapter 8* for more details).

[32] I would like to apologise to the reader for the sometimes mediocre quality of the polished surfaces illustrated in the text. After the retirement of the technician E. - Master of polishing - De Temmerman, I had to perform this secure operation myself, something that was harder than thought and the result was not as wanted. Extra time spend at the 'turning tables' did not result in much better results.

4.4. ICP-MS – lead isotopic analyses (LIA) & trace elements

4.4.1. Introduction & sample preparation

Inductively coupled plasma - mass spectrometry (ICP-MS) is generally acknowledged as one of the most powerful techniques for the quantitative determination of ultra trace elements. ICP-MS combines the ease of sample introduction and the use of an ICP as an ion source operated under atmospheric pressure with the sensitivity and selectivity of mass spectrometry.[33] One of the attractive features of ICP-MS is its capability of rapid quantitative detection of elements present in a sample.

In its standard configuration, ICP-MS needs a digested sample in order to perform the analyses. The liquid sample is vaporized into a fine aerosol by use of a nebuliser and a spray chamber. The inert carrier gas argon leads this aerosol into the inductively coupled plasma, formed at the plasma torch. The extreme heat (6000K) of the ICP breaks down the introduced molecules to their atomic constituents, and these atoms are converted into singly charged positive ions. The ions are then extracted by the interface into the mass spectrometer, where they are separated according to mass-to-charge ratio. Finally, separated ions are sent to the detector and counted. The resulting signal on a mass-to-charge ratio is proportional to the amount of ions present with that mass-to-charge ratio. ICP-MS has a linear dynamic range of 9 orders of magnitude, but in order to determine the concentration of major elements, the sample has to be diluted to prevent saturation of the detector and hamper accurate determination of trace elements.[34]

Laser ablation allows the analysis of solid samples. The impact of the laser beam vaporizes a part of the surface. A laser beam can carry sufficient energy to remove a tiny sample from a solid specimen and also vaporize, atomise, excite and ionise this sample. Laser ablation devices can vaporize a hole of a few micrometers in diameter from a solid sample and send it directly into the ICP-MS, eliminating the need for a sample solution. This saves considerable preparation time dissolving and diluting samples and is extremely attractive to archaeologists because it is considerable less destructive.[35] This technique was only used on one coin, since it is semi-quantitative due to the small area blasted. Moreover the sample chamber for this procedure is small and could not hold the samples presented for this research.

With the introduction of ICP-MS in the early 1980s there was great hope that this would be an interference free technique, but this is not the case however. The interferences, which occur in ICP-MS, fall broadly into two groups, *spectral interferences* and *non-spectral interferences* or *matrix effects*.

The spectral interferences can be subdivided into four areas: those due to *isobaric overlap*, *polyatomic* or *adduct ions*, *refractory oxide ions* and *doubly charged ions*. An isobaric overlap occurs when two elements have an isotope on the same nominal mass (*e.g.*, ^{87}Sr and ^{87}Rb). Polyatomic ions are thought to result mainly from ion molecule reactions during the expansion process (*e.g.*, $^{56}Fe^{+}$ and $^{40}Ar^{16}O^{+}$). Refractory oxide species, as their name suggests, may in fact be present in the plasma as the strong oxide bonds are not always broken down. Elements with low second-ionisation energies will be present partly as doubly charged species (*e.g.*, $^{138}Ba^{2+}$ and $^{69}Ga^{+}$).

The non-spectral interferences are more complex, and less well understood, but may be broadly divided into *suppression and enhancement effects* and *physical effects* caused by high total dissolved solids. The extent of the interference problems in most cases is related to

[33] Williams, 1998: 253-254.
[34] Renfrew & Bahn, 1997: 344; Williams, 1998: 253-254; Young & Pollard, 2000: 41; D. De Muynck pers. comm.
[35] Young & Pollard, 2000: 37.

the nature of the sample matrix and much can be done to minimize or even eliminate potential problems by careful sample preparation.[36]

Before samples were taken, a small area of the artefact surface was cleaned to expose unaltered material free of corrosion products that may introduce elements foreign to the original metal. A small quantity of this unaltered material was then removed using a small hardened tungsten carbide drill. Approximately 50 mg of the sample powder was weighed, and dissolved in a mixture of concentrated subboiled HNO_3 and H_2SO_4 in a beaker on a hotplate. The Pb recovery for the sample digestion was shown to be quantitative. After digestion, the pure Pb fraction was isolated from the sample. For this purpose, an extraction chromatographic separation, based on a Pb-selective crown ether, was used. The isolation process gave rise to a quantitative Pb recovery. The Pb isotope ratio measurements were performed on a PerkinElmer SCIEX Elan DRC*plus* ICP-MS instrument. The external precision obtained on the Pb isotope ratios was below 0,17% RSD for ratios with ^{204}Pb, and below 0,09% RSD for the other ratios. More information on the Pb isolation methodology and the measurement protocol is given in D. De Muynck, Ch. Cloquet and F. Vanhaecke.[37]

4.4.2. Lead isotopic analyses

> "*Often archaeologists are blinded by the exactness of the analytical results themselves that seems to imply equal exactness in the conclusions derived therefrom*[38]*"*

4.4.2.1. Introduction & geochemistry of lead isotopes analysis (LIA)

- **Geological application**

Lead is an omnipresent element in ore deposits, rocks and minerals. It occurs in major amounts in lead-zinc and lead-silver ore deposits and to a lesser extent in copper and iron ore deposits. It even occurs in at least ppm-levels in all rocks and non-metallic minerals.[39] This first observation is one of the cornerstones of lead isotope studies. Next to this lead has an interesting isotopic feature.

Isotopes are varieties of a same chemical element whose atoms have a common number of protons and electrons (i.e. their atomic number Z is the same) but they vary in the number of neutrons in their nucleus (i.e. their atomic weight A differs)[40]. Isotopes of the same element display the same chemical properties, but their physical properties related to mass are different. A distinction has to be made between stable and non-stable (radioactive) isotopes. Non-stable isotopes will evolve into stable isotopes via radioactive decay. In the case of Pb isotopic analysis, the focus is on the stable Pb isotopes. Lead has four such stable isotopes, namely ^{204}Pb, ^{206}Pb, ^{207}Pb and ^{208}Pb, with a respective atomic mass of 204, 206, 207 and 208. Lead present on/in the earth today is a mixture of lead, which was present when the earth was formed (*primordial lead* - ^{204}Pb) and lead subsequently derived from radioactive decay of uranium and thorium (*radiogenic lead* - ^{206}Pb, ^{207}Pb & ^{208}Pb). For the great majority of chemical elements the relative proportions of their isotopes are the same regardless of where the elements are found in the earth's crust. Lead, however, is unusual in a way that the relative proportions of its isotopes vary somewhat among ores occurring in different geographical areas. These variations reflect differences in the geological ages of the ore deposits and occur from the fact that ^{206}Pb, ^{207}Pb and ^{208}Pb are formed as an end product of the radioactive decay of uranium and thorium[41] ($T_{1/2}$ is the half-life):

[36] Williams, 1998: 254-255.
[37] De Muynck, Cloquet & Vanhaecke, 2007; D. De Muynck pers. comm.
[38] Pernicka, 1995: 63.
[39] Gale & Stos-Gale, 2000 : 503.
[40] Allaby & Allaby, 1999: 293.
[41] Healy, 1978: 219-220; Barnes, Shields & Murphy, 1974: 2; Rohl & Needham, 1998:1-3; Weeks, 2004b: 130.

$^{238}U \rightarrow {}^{206}Pb$ $T_{1/2} = 4{,}4683 \times 10^9$ years
$^{235}U \rightarrow {}^{207}Pb$ $T_{1/2} = 0{,}70381 \times 10^9$ years
$^{232}U \rightarrow {}^{208}Pb$ $T_{1/2} = 14{,}01 \times 10^9$ years[42]

It is normally assumed that in any small part of the Earth's crust and mantle that contained primordial lead, uranium and thorium at the formation, did not contain any radiogenic lead. With the passing of time atoms of radiogenic ^{206}Pb, ^{207}Pb and ^{208}Pb gradually replace uranium and thorium atoms at a known speed. If at a certain point in time all the lead in a deposit is removed (and separated from the uranium and thorium source) and it deposited again as an ore then the *signature* of this moment is preserved and the decay is stopped. The fourth isotope (^{204}Pb) is of non-radiogenic origin and the amount of this isotope has not changed since the earth was formed some 4,5 to 4,6 billion years ago, so it can be used as a standard to compare the other isotopes against.[43]

Objects with low $^{208}Pb/^{206}Pb$ (1,750-1,850) and very high $^{206}Pb/^{204}Pb$ (24,00-26,00) ratios, are generally described as *radiogenic*, referring to the fact that their lead isotopic compositions have been affected by high levels of uranium and/or thorium in the ores from which they were smelted[44].

Different factors are at play. The original U-Th-Pb ratios present in the system during its formation (cooling and segregation into core, mantle and crustal components) differ from region to region. This led to differing ratios of U/Pb and Th/Pb in different parts of the world and is the origin of the different ratios of the radiogenic lead isotope values across the planet. The mixing of lead from different sources should however not be overlooked. So even though ore deposits may be very similar in type, host rock and age, they can still have very different isotopic compositions depending on the source material.[45]

From a geological perspective, the lead isotope compositions of ore deposits are primarily determined by the age of the deposit and the sources of the lead that they incorporate. Dating these deposits is the main question.

- **Archaeological application**

Based on the cardinal rules and parameters set out by the geological research, an interesting archaeological side effect was born. Here, the age of the deposits is not of direct importance, but the different *fingerprints* of different geographical/geological deposits have the potential of provenancing lead containing artefacts made from them. This is because the finished objects should have the same isotopic *fingerprint* as the parent ore. Isotopic compositions remain constant independent of any physical-chemical changes, natural or artificial, such as smelting, refining, casting, oxidation, corrosion, etc., i.e. there is no *fractionation*[46]. Hence the signature of the object can potentially be linked to that of the ore. Comparisons can also be made between the isotopic characteristics of finished objects from different sites and areas. It is these ore-to-artefact and/or artefact-to-artefact links that are the prime interest of archaeological lead isotope analysis (LIA).[47]

The fact that there is no fractionation is the major advantage of this technique over an ore-artefact comparison based on trace elements. This has two important consequences. First of all neither the exact pathway from ore-to-artefact, nor the metallurgical processes used, needs to be known in order to predict the trace element(s) behaviour. Secondly, the isotopic

[42] Barnes, Shields & Murphy, 1974: 2.
[43] Allaby & Allaby, 1999: 313.
[44] Weeks, 2004b: 146.
[45] Healy, 1978: 219-220; Barnes, Shields & Murphy, 1974: 2; Rohl & Needham, 1998:1-3; Weeks, 2004b: 130; Weeks & Collerson, 2005: 318.
[46] Rohl & Needham, 1998: 4; Ortiz, 2003: 21.
[47] Begemann, Schmitt-Strecker, & Pernicka, 1989: 269; Rohl & Needham, 1998:1; Weeks, 2004b: 129.

composition is not dependent from how the lead is distributed between the different phases that might be present in the metal. Different segregation phases in artefacts may have major differences in their lead contents but the lead will have the same isotopic composition. The heterogeneity of the sample, which is important when applying elemental analysis, is of no relevance to LIA. For this reason, just a single analysis of a metal object will be representative for the isotopic composition of the entire object.[48] This is not the case if an inhomogeneous substance is analysed, such as soil, bone, etc.

In first instance LIA was used on lead or objects including a large amount of lead (such as lead glazes, pigments, glass, etc.) but it was quickly expanded to silver artefacts. The link between the metal used and the ores are evident, since lead logically originates from lead ores and the majority of the silver in antiquity came from argentiferous galena deposits.[49] In a last step the potential of the technique was also seen for provenance studies of copper and copper-base alloy artefacts. This development is based on the fact that small amounts of lead are commonly present in copper and copper-base alloys, not due to intentional addition of lead, but as a result of their initial presence in lead bearing copper ores. With the present methods the minimum lead levels needed for detection and analysis are so small that samples with only 0,5 µg of lead per gram of material can be analysed[50]. Analysis of the lead isotope characteristics of this lead offers the potential to determine the geological source of the copper used in these artefacts. Linking these two datasets holds an important prerequisite however in that a representative database with lead isotopes analyses of potential ore bodies has to be available.

The earliest LIA performed on archaeological material already dates back to the 1970s. The initial enthusiasm for this technique was great since some open archaeological questions could be tackled. But as with every new analytical method also lead isotope analysis had to go through a period of debate, problems and criticism. This lasted through most of the 1980s and 1990s. The major scholarly debates can be separated in two waves. The first wave discussed issues as mixing and remelting of copper supplies, the production of intentionally leaded bronzes, the possible contribution of lead to a bronze by the addition of tin or cassiterite, and the possible role of polymetallic ore bodies as suppliers of different types of metal. The second major debate covered the nature of samples used to define ore fields, the statistical treatment of lead isotopic data in the removal of outliers and the delineation of ore fields, and the reliability of laboratory measurements of lead isotope ratios.[51]

Especially the growing body of available lead isotope data tempered the initial optimism. The growing dataset showed the limits of the technique as it became clear that not all ore deposits had the unique isotopic signature as originally thought and considerable amount of overlap occurred. The potential of the method to delineate *exclusively* the sources of the copper used in archaeological object was undermined.[52] Although this realisation shacked the fundaments of the lead isotope provenance studies, it did not bring the technique down but rather brought it back to its true usefulness. In the next part the 'problems' that may arise with LIA are explained.

LIA can also serve to compare objects from a same context amongst each other or with those of other locations. This side step from the strict provenance question can also provide interesting information on (changing) trade and exchange patterns.[53]

[48] Brill & Wampler, 1967: 72; Healy, 1978: 221; Begemann, Schmitt-Strecker, & Pernicka, 1989: 269; Ortiz, 2003: 23.
[49] Weeks, 2004b: 132.
[50] Ortiz, 2003: 23.
[51] Weeks, 2004b: 133.
[52] Weeks, 2004b: 133.
[53] Weeks, 2004b: 133.

4.4.2.2. Considerations, criticism and limits

Several general areas of concern can be expressed when discussing LIA[54]:

1. Earlier lead isotope measurements are of insufficient high precision and these data can be erroneous and difficult to use together with newly acquired data. This concern can even be valid for recent analyses and comparing data from different laboratories may proof difficult since each uses their own procedures, parameters, etc. A wide variety of reference standards are however used to minimize these problems. The database used here does not consider this and all relevant LIA are included.
2. It has been suggested that fractionation of the isotopes can occur during smelting with each isotope undergoing processes at a slightly different rate, making it impossible to link the ore with the end product[55]. Although fractionation is theoretically possible, experimental work indicates that in practical situations fractionation does not introduce errors of a greater magnitude than the analytical precision commonly obtained in most laboratories[56].
3. The isotopic signatures of different ore sources are insufficiently unique for each ore deposit to separate all of the fields. This problem cannot be overcome with statistical treatment of the data.
4. A major setback is the effect of possible mixing and recycling of metal from more than one ore source.

Points 3 and 4 will be treated is some more detail beneath.

- **Defining ore fields**

The exact definition of an isotopic source field and its extension must be based on geographical, geochemical, mineralogical and statistical data. Grouping geographically and geologically related deposits, with the aim of obtaining homogeneous isotopic fields of meaningful size is important to keep the data interpretable. The limit of increasing the size of source fields on a geographical basis is difficult and must rely upon criteria of statistical homogeneity. Two things should be taken into consideration in defining ore fields. The first is that individual ore deposits represent the smallest possible grouping of data points and can never be split up among different fields. The second is that geographically, or geologically, unrelated deposits can never be merged in a single field, even if they show extensive isotopic superposition. D. Attanasio, G. Bultrini and G.M Ingo define a *source field* as "*the largest grouping of statistically homogenous, geographically related ore deposits*".[57]

So the first premises to compare the isotopic signature of an artefact to that of an ore field, is the clear delimitation of the fields isotopic ratios. While it would be very convenient if each deposit or mine had its own unique isotopic signature, this is far from true in reality[58]. Two entirely different deposits can have the same signature due to geological similarity, making them indistinguishable[59]. In such a case the combined study of trace elements and isotopic ratios can possibly provide better separation. This was not done here since not all studies combine these two tracks and within the time and money frame of the LIA this was impossible. Some ore bodies on the other hand may be heterogeneous and have a lead isotopic signature with a very broad range (*anomalous* ore bodies) due to incomplete mixing of the ores.[60]

The analyses of sufficient samples from the mineral deposits are crucial to grasp the true isotopic extent of the deposits or mining region. The quantity of samples needed, will vary

[54] Tite, 1996: 960.
[55] Budd, Pollard, Scaife & Thomas, 1995c.
[56] Rohl & Needham, 1998: 5; Weeks, 2004b: 131.
[57] Attanasio, Bultrini & Ingo, 2001: 535.
[58] Ortiz, 2003: 20.
[59] Healy, 1978: 220; Ortiz, 2003: 20.
[60] Rohl & Needham, 1998: 3-4; Ortiz, 2003: 20.

according to the characteristics of the ore field, but to obtain a reliable isotopic definition of a mineral deposit, a number of samples between 12 and 20 has been suggested[61].

One cannot hope to use isotopic evidence alone to prove conclusively that the lead from some given object was taken from some given area. Only measuring the isotopic composition of random ore bodies is useless without considering their archaeological relevance, *e.g.* data of ores that could not be worked at a certain period cannot be used for a study of that particular period. Also the geological context cannot be overlooked, *e.g.* when studying copper artefacts it is useless to include lead ores in the reference database. To keep the field surveyable different parameters have to be considered, rather than 'blind' comparison of isotopic ratios.[62]

• **The effect of mixing of ores or metals from different origins**
One of the primary assumptions of LIA in archaeology is that ores and metals from different sources have not been mixed together to produce an object. Alloying or mixing can however emerge in different ways and may involve mixing different ores together, adding crushed ore to the base metal during the melting, or melting different metals together. The lead isotope composition of metal originating from a mixture of metals of different origin will fall within the region defined by drawing straight lines between the isotopic ratios of the original ore deposits (*e.g.* if 3 origins are involved this will form a triangle). Its exact position will depend very strongly on the amount of lead present in each fraction added to the alloy.

• *Lead and silver objects*
The main common lead mineral is galena (PbS). Secondary lead minerals such as cerussite ($PbCO_3$) or anglesite ($PbSO_4$) have the same isotopic composition as that of the galena from which they were formed. Many factors can however intervene, induced by the geological evolution of the ores. The lead present in the rocks and in the mineral deposits shows a complex range of variations that reflects their particular geological history.[63]

In antiquity almost all silver was extracted from argentiferous galena, and always retained some of the lead from the parent material. This small amount of lead would have the same isotopic characteristics as the galena.[64] Silver metal artefacts should however be treated with the necessary precautions. Silver has always been a precious metal so recycling and remelting the metal must have been common practise. If silver of the same origin is remelted together this does not pose a problem, it is only when silver of different origins is mixed that the signature is blurred. A second problem might be the fact that silver is often to a certain extent debased with copper that brings its own fingerprint[65]. A last point to consider is the presence of some litharge fragments at ed-Dur. These are most probably the residues of silver extraction from a copper-silver alloy in a process that involves adding lead to the system (for further details see *Chapter 6*). The addition of lead would seriously distort the original signature of the resulting purified silver. M.H. Ortiz states that in such a case the metal should be considered as an alloy as far as its isotopic composition is concerned[66]. We do not know to what extent this process was used, but it shows that the knowledge was at least present.

• *Copper-base alloys (bronze, leaded bronze & brass)*
Tin
The major alloying element of copper to produce bronze is tin, so if tin-bronzes are analysed for their lead isotopic fingerprint the addition of lead via the tin metal or ore should be

[61] Ortiz, 2003: 23.
[62] Gale & Stos-Gale, 2000: 539-540.
[63] Ortiz, 2003: 19.
[64] Weeks, 2004b: 132.
[65] Gale & Stos-Gale, 2000: 541.
[66] Ortiz, 2003: 25.

considered. The potential contribution of lead from tin or cassiterite (SnO_2), which is found relatively pure in nature, is seen as minimal since tin deposits rarely contain any lead. Even if lead would be present it is in such small amounts that the addition of 10% of tin would not significantly alter the copper isotopic signature. For this reason the addition of tin is not considered a factor of contamination.[67] This is not completely true, since some tin deposits do contain amounts of lead. The limited amount of lead isotope analysis on tin ingots does show however that only very small amounts of lead are present, but a contribution cannot be excluded.[68] A second parameter to keep in mind is the fact that copper can also be very poor in lead and then even the small amount of lead introduced by the tin can have an impact[69].

Lead

The limits to define 'leaded' alloys differ from author to author. The lead isotope analyses of a heavily leaded bronze containing 8 to 15% of lead, will basically determine the provenance of the added lead and not that of the lead present in the copper ore. Lead and copper can however also come from the same deposit and in this case the LIA are representative. Smelting experiments and analyses of batches of raw copper have shown that certain copper ores can generate smelted copper with up to 4 to 5% of lead, already present in the original ore. According to N.H. Gale and Z.A. Stos-Gale it seems safe to say that copper alloy artefacts containing lead in the range from 50 ppm up to 4% are suitable for the provenancing of their copper by LIA.[70] D.R. Hook and P.T. Craddock are more pessimistic however and say that lead isotopes of leaded bronzes are unlikely to produce meaningful data, given the large number of potential lead sources and the prevalence of mixing and re-use of metal[71]. So if the lead levels are 'low', they are unlikely to represent intentional alloying and the lead is likely derived from the parent ores[72].

It is generally accepted that the use of leaded bronze in the Near East and E-Mediterranean was not important until the first millennium BC. Analyses of prehistoric copper-base objects from Oman and the U.A.E. indicate only a handful of objects with more than 1% of lead before the end of the Iron Age. All the rest of the objects had very low lead values.[73] The lead levels in the ed-Dur artefacts analysed by L. Weeks are high in comparison with the older SE-Arabian material, but are still low when compared to contemporaneous Roman material[74]. The lead values are probably due to the use of a different copper ore source, rather than the intentional adding of lead to the alloy. Many samples do however contain lead in amounts that are unquestionably deliberately added.

Zinc

The lead isotopic analysis of brasses is difficult. Lead is always associated with zinc ores, often in substantial proportions and it is fairly volatile. In the sublimation process (see *Chapter 5*) some lead oxide would inevitably be carried over with the zinc oxide.[75] As such this would not pose a problem since the resulting zinc oxide would have the same signature as the lead from the mixed ore body (i.e. containing zinc and lead). The problem is however that this zinc oxide was added to copper during the brass making process, mixing the two different isotopic ratios.

In the Mediterranean the production of brass is likely to have been a rather specialised activity concentrated in some major centres. At these centres, metal from a variety of

[67] Rohl & Needham, 1998: 5; Gale & Stos-Gale, 2000: 539; Ortiz, 2003: 26.
[68] Weeks, 2004b: 139.
[69] Weeks & Collerson, 2005: 319. Umm an Nar copper-base objects have low levels of lead and are therefore highly susceptible to contamination by lead of different isotopic composition introduced by the tin (Weeks, 2000a: 196).
[70] Gale & Stos-Gale, 2000: 538; Ortiz, 2003: 26; Weeks, 2004b: 137.
[71] Hook & Craddock, 1996: 152.
[72] Weeks, 2004b: 151.
[73] Weeks, 2004b: 138.
[74] Weeks, 2004a: 248.
[75] Craddock, La Niece & Hook, 1998: 74.

sources may have been available, including recycled scrap, and thus there is little potential for provenancing the copper by composition.[76] In the frame of the ed-Dur brasses it has to be noticed that many of them are rather pure (i.e. no tin and lead present) and have a rather high zinc level (see *Chapter 5* for details). Both assessments are indicative of limited recycling. On the one hand the original brass was not alloyed with scrap bronze otherwise a certain amount of tin (and if from a leaded bronze, lead) would have been introduced. The high zinc value on the other hand point to a limited amount of remelting, since every time brass is remelted a certain amount of zinc is lost as vapour. Whatever the value of the obtained lead isotope ratios of the analysed brasses in order to pinpoint a certain place of origin, they are valuable for intra-site comparison of the brass artefacts.

- **The effect of metal recycling on LIA**

The recycling of scrap metal or objects from different origins will lead to a mixing of their specific lead isotopic signatures and will make it very difficult or even impossible to extract any useful information.[77] Recycling is more likely to happen on sites that are distant from primary smelting centres. Metal was expensive, largely because complicated technology was required to extract it from its ores and because it was commonly traded over long distance.[78]

Specifically for the Oman Peninsula it is important to notice that widespread deposition of certain amounts of metalwork is attested in Bronze Age, Iron Age and late Pre-Islamic tombs. The removal from circulation of sometimes large quantities of metal in the form of burial goods can be seen as an argument for limited recycling and the necessity to acquire 'new' metal. However plundering just those rich tombs from previous periods may have relieved the need for new metal. The archaeological record shows that this was common practise in the region. Also the reuse of older tombs (especially attested in the Iron Age or more recent) may have brought people in contact with previously buried goods.[79] Recycling however only poses a problem if metal (objects) of different origins are melted together. If recycling however occurred within the same 'metal batch' the isotopic signature will be unaltered. Hypothetically speaking (!), if Umm an-Nar objects produced of local ores are recycled in later periods and the resulting objects are analysed they will still point towards an SE-Arabian origin. The difference would be that the metal was not freshly produced at that time, so the logical assumption of metal extraction at these later periods would be invalid.

- **Presentation of results**

The large majority of published archaeological LIA are presented in the ratios: $^{204}Pb/^{206}Pb$ or $^{206}Pb/^{204}Pb$, $^{207}Pb/^{206}Pb$ and $^{208}Pb/^{206}Pb$. Other ratios are sometimes used but much less frequently. In any case it is possible to calculate any ratio if the necessary lead isotope(s) is available (from $^{204}Pb/^{206}Pb$ the values of $^{206}Pb/^{204}Pb$ can be calculated or $^{208}Pb/^{207}Pb$ from $^{207}Pb/^{206}Pb$ and $^{208}Pb/^{206}Pb$). These three ratios are presented in a pair of bi-variant graphs. Two such plots are needed to show the full isotopic variation of the objects, since each lead isotope measurement gives the parameters of a point in three-dimensional space. If an object falls within the boundaries of an ore field on both plots this shows that both may be related. Differences on one such plot is however sufficient to show that there is an inconsistency. If different clusters are formed on these plots they are indicative of different ore sources.[80]

It seems clear that a basic approach in which artefacts can visually be assigned to ore fields using *nearest neighbour* procedures, is the most appropriate for archaeological LIA. Such a minimal approach, whereby enveloping lines around the outer error bars of the data

[76] Hook & Craddock, 1996: 152.
[77] Gale & Stos-Gale, 2000: 531.
[78] Weeks, 2004b: 140-142.
[79] Weeks, 2004b: 140-142.
[80] Stos-Gale, 1998: 351; Gale & Stos-Gale, 2000: 522; Weeks & Collerson, 2005: 318.

delineates ore field boundaries, is used for simplicity and in order to avoid the over-interpretation predicted by some researchers.[81]

Although the above shows that a strict positive relation between the ore and the object might be difficult, or even impossible, to prove a negative relation can also be useful. Negative conclusions regarding provenance can be strong whereas positive assignations are by their nature weaker and dependent upon the extent of isotopic sampling of potential ore sources.[82]

- **The lead isotope database**

An important shortcoming was the lack of a reference database to compare the new results to. Several thousand accurately determined data points are available in the literature, and on the bases of these measurements a database was composed[83].

We decided to include as much data as possible, since it was not clear where the material might have originated. The copper-base alloys (raw or finished product) could be locally produced, but in the same time import from the Mediterranean Roman World, the Parthian Empire or even India are very likely. Four datasets were composed. The first consists of all possibly relevant copper ore deposits (including ingots and slag), the second of copper-base alloy finished objects, the third of lead/silver ores, and the fourth of lead and silver finished artefacts. We are aware of the fact that the dataset is not homogenous, some measurements are old, precisions are different, analyses techniques differ, etc. But on the other hand what is the value of all these published data if they cannot be used.

Roman world material

Probably the best researched part of the world for the archaeological application of lead isotopes is the Mediterranean. Literary thousands of analyses were preformed on all kinds of materials (ores, slag, ingots, objects, etc.) [84]. To bring some order in this massive amount of data, all analysed artefacts dated earlier than the 2nd c BC were excluded. Some later Islamic artefacts were included however based on the idea that the metal used then originated from within the Islamic world that included the Gulf. The relevant mining regions were selected based on the thought that the metal circulating in the Roman Empire originated from within the territories[85]. During the lifetime of ed-Dur the Roman Empire reached its largest expansion and many mining districts were exploited. The LIA on material from Pompeii can serve as an example for the complexness of the supply areas. The results indicate that 40% of the material has links to Sevilla and Hueva in S-Spain, 10% could be correlated to Sardinia, 12% originated from deposits in the ophiolotic Troodos complex of Cyprus and for 16% no match was found however[86].

Parthian world material

The wealth of information at hand on the Mediterranean is not mirrored in the available analysis from the Parthian territories. For this reason all available data on ores was included.

Some data are available for copper ores and prehistoric slags from the Iranian Plateau, based on recent archaeometallurgical research by a combined Iranian-German team, and geological research at Iran's largest copper mine at Sar Cheshmeh. The analysed copper ores and slags from the Kashan region have $^{207}Pb/^{206}Pb$ ratios of less than 0,843, and show a similar range to the analysed sulphide concentrates from rocks hosting the Sar Cheshmeh deposit. There are no exact isotopic matches between the Iranian and SE-Arabian groups.

[81] Weeks, 2004b: 143.
[82] Ortiz, 2003: 21; Weeks & Collerson, 2005: 321.
[83] I would like to thank L. Weeks for providing the basic reference LIA-database. This list was however largely expanded with, *e.g.*, more recently published data. A full list of the references used is given in Appendix 2.
[84] Rohl & Needham, 1998:1.
[85] Shepherd, 1993: 151-152. R. Shepherd publishes a list of the date when new regions were added to the Roman Empire and their mineral wealth became accessible.
[86] Wagner, 2000: 615.

The slag analyses do indicate however that more LIA on Iranian copper ores and slags may make the isotopic distinction between metal from Oman and Iran problematic since their geological genesis is similar. Moreover ore bodies with a wide variety of isotopic characteristics can be expected in Iran, many of which may match ores from SE-Arabia.[87]

India
The same dearth of information on Parthian material is also found for material originating from the Indian Sub-continent. Also here as much data as possible was included sometimes neglecting the dating of the analysed objects.

Lead was a relatively uncommon metal in ancient India, until the exploitation of the large sources at Zawar in Rajasthan. The use of lead as a coining material suggest its 'pseudo-precious metal' status. N.J. Seeley and P.J. Turner examined Indian lead coins and found two distinct isotopic groups. The LIA of the first group match those of the Sardinian and Spanish lead sources exploited by the Romans, confirming the statement in the 1st c AD *Periplus* that lead was imported into India. These mines became relatively unimportant after the opening of the British lead mines, which were more economical to work. The British mines came into use not much later than 50 AD. The LIA signature of the second group (dated slightly later) falls close to this obtained for ores from Zawar, implying the use of indigenous metal.[88] Some copper ores have also been analysed isotopically. These deposits are hosted in geologically old rocks of Precambrian age and their $^{207}Pb/^{206}Pb$ ratios are commonly greater than 0,900.[89]

SE-Arabia
There are numerous copper deposits in the Arabian shield and SE-Arabia had prominent periods of copper extraction. Still the number of isotopically analysed copper ore samples is rather limited and future analyses may broaden the potential isotope range of copper in Oman[90]. There is a significant overlap in the isotopic data for SE-Arabian copper ore deposits and those of Cyprus and the Taurus Mountains. This reflects the similar age and geological context of the copper ores from those regions.[91]

The accumulated body of geological lead isotope data from SE-Arabia indicates a relatively broad range of lead isotope data from SE-Arabia. This broad range is also to be expected in the copper-bearing massive sulphide deposits of the Semail Ophiolite. An examination of the isotopic variability of individual ore deposits from Oman indicates that: ores from the *'Arja mine'* show a very limited isotopic variation; the ores from *Raki mine* have a slightly larger variation, which might indicate *anomalous* lead; and the *Al Ajal deposit* has a clearly *anomalous* linear isotopic signature. Small deposits from mantle and lower crustal formations of the Semail Ophiolite are poorly characterised. There are also no lead isotope analyses of copper ores from Masirah Island, off the SE-coast of Oman. These deposits are known to have been exploited from at least the early second millennium BC.[92] A non-Omani source for some objects with the highly *radiogenic* fingerprint seems very likely[93].

The importance of a lead isotope analysis program on the ed-Dur material was already expressed by L. Weeks: "*The investigation of the sources of lead used for lead objects and leaded copper alloys … would be a worthwhile research programme, particularly with regard to determining the local production of copper alloys …* ".[94] But he also stressed that the scientific determination of provenance for *unleaded bronze* and *brass* objects would be more

[87] Weeks, 2004b: 159-160.
[88] Seeley & Turner, 1984: 331.
[89] Weeks, 2004b: 159.
[90] Weeks, 2004b: 156.
[91] Prange, Götze, Hauptmann & Weisgerber, 1999: 191; Weeks, 2004b: 135 & 160.
[92] Weeks, 2004b: 136.
[93] Weeks, 2004b: 158.
[94] Weeks, 2004a: 248.

difficult, given the overwhelming evidence for recycling and mixing of fresh and scrap metals by Roman times.

The analyses performed within this study are the first on 1st c BC – 2nd c AD material from the region, so all results will be an (valuable) addition to the existing body of analyses. The analysed samples do not come from a site that has evidence of copper/lead production or working (difficult to attest in the case of lead), so trade or import must explain their presents. Their origin could be far from the Gulf region considering the archaeological context of ed-Dur. So the LIA can potentially provide information on the wider trading networks present within or connecting to the Gulf.

The lab that performed the LIA had obtained experience concerning lead isotopic analysis for archaeological purposes. One project focused on obtaining insight into possible lead poisoning during the Roman Period by investigating infant human bone tissue, surrounding soil and other possible 'contributors' endmembers, such as *amphorae* and lead objects. An analytical methodology, comprising sample digestion with quantitative Pb-recovery, quantitative and pure Pb-isolation from its matrix and isotope ratio measurements under fully optimised conditions using ICP – dynamic reaction cell – MS was developed. For the lead isotopic analysis of the objects originating from ed-Dur, new digestion procedures leading to quantitative Pb-recovery were developed. The previously developed isolation procedures were shown to be useful for metal objects as well, and the measurements were carried out using the previously developed instrumental protocol. This complete analytical strategy was successfully applied to the metal objects found at ed-Dur.[95]

In a first step the lead isotope ratios of the analyses objects will be compared amongst each other. In a next step they will also be compared to other relevant published data of ores, slag and other objects.

- **Some thoughts on the ed-Dur material**

Recycling in general

In the case of ed-Dur there was no re-use of elder tombs on the site, a phenomenon also not seen on the site of Mleiha, since no older tombs are present. Other contemporaneous sites in the region show isolated interments of individuals in older collective graves. However these are often close to the surface and it does not seem that the older burial gifts were removed. An important difference between the late Pre-Islamic Period and the previous periods is the introduction of iron, so the demand for copper-base metal was much lower. Although copper-base alloy vessels are still used, the presence of large quantities of glass vessels also reduced the need for copper-base recipients. The absence of traces of copper working on ed-Dur does not support the local recycling of metal. The limited evidence from Mleiha does not really change this picture, since these remains are linked to a limited primitive primary extraction technique. All these remarks are of course of no consequence for imported metal and the practises in the exporting countries should be know.

Lead objects

In comparison to other site in SE-Arabia lead 'objects' are plentiful. The absence of lead on other sites is indicative for the import of this metal at ed-Dur from elsewhere, otherwise it would be more commonly used on other sites. The lead 'ingot' (if indeed it is an ingot) found at ed-Dur supports this assumption. The production of lead was in antiquity most often not the purpose in its own but the result of the by-product of silver extraction from argentiferous lead ores. There is little reason to believe that the metal for lead object would have originated from different ore deposits. Moreover the ease of lead production and the large gain from the

[95] All analyses were performed by D. De Muynck, to whom I am very grateful, at the *Laboratory of Analytical Chemistry - Institute for Nuclear Sciences – UGent* (headed by Prof. Dr. F. Vanhaecke) by ICP-MS. A more detailed description of the procedure can be found in De Muynck, Cloquet, & Vanhaecke, 2007.

reduction of its ores does also not indicate multiple ore sources. For this study we will use the working hypothesis that the lead is not mixed and has the same fingerprint as its mother ore.

Silver objects
In discussing the silver metal artefacts caution is needed since the recycling and remelting of this precious metal is known to have been common practise in antiquity. If silver of the same origin is remelted together this does not pose a problem, it is only when silver of different origins is mixed that the signature is blurred. A second problem might be the fact that silver is often to a certain extent debased with copper that brings its own fingerprint and causes additional distortion of the signature. As mentioned above a last point to consider is the presence of some litharge fragments at ed-Dur. These most probably are the residues of silver extraction from a copper-silver alloy in a process that involves adding lead to the system. This lead would also leave its own signature. We do not know to what extent this process was used, but it shows that the knowledge was at least present.

Copper objects
Corrosion does not alter lead isotope compositions by fractionation. The burial environment could however introduce lead, i.e. present in the soil[96]. The lead levels in the ed-Dur artefacts analysed by L. Weeks are high in comparison with the older SE-Arabian material, but are still low when compared to contemporaneous Roman material, so many objects were not intentionally leaded (a common practise in the Roman world)[97]. The fact that the lead levels are not super low makes an influence of the environment improbable. Moreover all analyses were done on corrosion free samples.

Bronze objects
Tin can hypothetically add some lead to the copper, a problem if the copper metal itself is very poor in lead. As stated above this does not seem to be the case for the ed-Dur objects and tin as a contributor can be safely excluded.

Brass
As stated above brass is a difficult (maybe unusable) alloy to submit to LIA. Still it was decided to include the brass objects. Most of the brass is rather pure and has a high zinc content, indicating that only limited recycling occurred. Whatever the value of the obtained lead isotope ratios of the analysed brasses are in order to pinpoint a certain place of origin, they are valuable for intra-site comparison of the brass artefacts.

Leaded objects
In analysing the leaded object it is important to know that the majority of the isotopic signature is formed by the lead and not by the copper.

- ***Erratum***

Initially it was also thought to compare the ed-Dur information with objects, but this proved rather un-useful. For many objects the origin is also not known, moreover there is a heavy emphasis on European and Mediterranean artefacts from the Bronze Age in the literature. It does not seem relevant to me to use these. Of course many Roman coins were analysed, but using these only is an intermediate step to get to the ore sources, and every step brings its own errors. It was decided to omit the comparison with artefacts in this study, but a future more detailed comparison with a selective dataset might still be useful and add additional information. It must also be admitted that the interpretation of lead isotope data is not as straightforward as originally thought. The rather simple presentation of the data in two bi-

[96] Gale & Stos-Gale, 2000: 527.
[97] Weeks, 2004a: 248.

plots can give the falls impression that the pinpointing of sources is no more than looking for ore data that plots together with the LIA of the artefacts. This is of course the basis, but next to that a thorough knowledge of the geological context, and the historical and archaeological information is needed to correctly interpret the data.

LIA is not the work of a layman or novice, but needs some more expertise and the obtained data was not explored to its full potential. They are kept for future research and publication.

4.4.3. Trace elements

All metal ores contain small amounts of other metallic elements, which are geochemically related to the main metal present. Some of these will be transmitted to the finished metal artefact and thus allow certain characteristics of the parent ore to be found in the smelted and refined metal. Furthermore, there will also be small amounts of similar metals associated with the flux added to assist smelting and, of course, with the alloying metals themselves. During the processes of smelting, refining and alloying, further changes will take place with the amounts of these contaminants, which will depend on the technology of these processes and the chemical properties of the individual elements themselves. By understanding these processes and their chemistry, it is possible to gain an insight into the ore types used, the technology employed in their processing and therefore to characterize the artefacts on a basis largely independent of intentional human intervention.[98]

Analyses of minor and trace elements present in copper-base artefacts have frequently been used as the basis for discussions of provenance and reconstructions of trade patterns. The feasibility of using diagnostic trace elements concentrations alone to outline ore sources has been seriously questioned over the last twenty years and more. When metallurgical industries had developed to the point where a variety of ores were being used under different conditions, with or without fluxes, alloys were commonplace. Recycling of copper work was also a normal practice. Therefore provenience studies depending on elemental analysis are as likely to confuse, as they are to clarify the relationships of source areas and distant production centra. However, compositional studies can play an important role in discussions of trade, if interpreted within a framework that allows for the complexity involved in metal extraction and object fabrication and the compositional heterogeneity of many ore sources.[99]

I am fully aware of the potential of the combined study of the lead isotope ratios and the trace elements present in the samples, but due to budgetary reasons and time restrained this was not undertaken to a large extent. Trace and minor elements were however determined for some samples and where possible and necessary these are included in this study

Trace and minor elements were also determined for all the coins analysed by ICP-MS[100]. These are not evaluated in this PhD, but may be used in a later publication. The main reason for omitting this data is the fact that their analysis did not yield a coherent picture and no pattern was found that added new insight in the typological grouping of the coins.

[98] Ponting, 1999: 1317.
[99] Weeks, 2004b: 105.
[100] The samples analysed were: AT 013, AV 083, AV 005, AV 104, AW 063-3, BL 014, C 079, K 203, M 007 and S 0020, next to all the coins analysed for their lead isotope ratios. The elements analysed for were Cr, Fe, Mn, Ni, Co, As, Se, Ag, Sb, Te, Hg and Bi.

4.5. Powder-XRD – mineralogical composition

4.5.1. Introduction[101]

Powder X-ray diffraction (XRD) is a technique of particular value in determining the crystalline lattice structures present in a sample. Solid matter can be described as *amorphous* or *crystalline*. In amorphous matter the atoms are arranged in a random way similar to the disorder we find in a liquid (e.g. glass). In crystalline matter the atoms are arranged in a regular pattern. It is the smallest volume element that by repetition in the three dimensions describes the crystal. This smallest volume element is called a *unit cell*. The dimension of the unit cell is described by three axes (a, b and c) and the angles between them (alpha, beta and gamma). About 95% of all solid materials can be described as crystalline.

An electron in an alternating electromagnetic field will oscillate with the same frequency as the field. When an X-ray beam hits an atom, the electrons around the atom start to oscillate with the same frequency as the incoming beam. In almost all directions there will be destructive interference, that is the combining waves are out of phase and there is no resultant energy leaving the solid sample. However the atoms in a crystal are arranged in a regular pattern, and in a very few directions we will have constructive interference. The waves will be in phase and there will be well-defined X-ray beams leaving the sample at various directions. A diffracted beam may thus be described as a beam composed of a large number of scattered rays mutually reinforcing one another.

In XRD two main applications can be distinguish: *single crystal* and *polycrystalline* or *powder* applications. The single crystal sample is a perfect crystal and all unit cells are aligned in a perfect extended pattern. The single crystal diffractometer is used mainly to clarify the molecular structure of new compounds, either natural products or man made molecules. Powder-XRD is mainly used for 'finger print identification' of various solid materials. The ideal sample is homogeneous and the crystallites are oriented randomly.

When X-rays interact with a crystalline substance (phase), a diffraction pattern is obtained. Every crystalline substance gives a unique pattern and the same substance always gives the same pattern. As a consequence, in components of a mixture of phases they will each produce their own pattern independently of the others.

The powder-XRD method is ideally suited for characterisation and identification of polycrystalline phases. Today about 50.000 inorganic and 25.000 organic single diffraction patterns have been collected and stored in a database as standards. The main use of powder-XRD is to identify the different components present in a sample by a search/match procedure. Furthermore, the areas under the peaks are related to the amount of each phase present in the sample and this can subsequently be quantified. The particular crystallographic structure can provide information on the temperatures at which the crystal was formed.

4.5.2. Sample preparation & analytical parameters

Powder-XRD is to provide additional information on the mineral phases present within the slag. This data will supplement the EDX-analyses that only provide the semi-quantitative amount of the individual chemical elements present, but no information on the minerals they make up.

[101] http://epswww.unm.edu/xrd/xrdbasics.pdf

The samples were broken up manually and then made into a fine powder in an agate ball mill. The powder-XRD analyses were performed by the *Laboratory of Soil Science – UGent*, headed by Prof. Dr. E. Van Ranst. The XRD-patterns were collected on a Philips X'PERT SYSTEM with a PW 3710 based diffractometer, equipped with a Cu tube anode, a secondary graphite beam monochromotor, a proportional xenon filled detector and a 35 position multiple sample changer. The incident beam was automatically collimated. The irradiated length was 12 mm. The secondary beam side comprised a 0,1 mm receiving slit, a soller slit, and an anti-scatter slit. The tube was operated at 40 kV and 30mA, and the XRD data collected in a theta, 2-theta geometry 3,00' onwards, at a step of 0,020° 2-Theta, and a count time of 1 sec.[102]

[102] Description provided by Prof. E. Van Ranst and is only included for the sake of completeness.

PART II

The unknown

Chapter 5. COPPER & COPPER-BASE ALLOYS

"Some minds improve by travel, others, rather, resemble copper wire,
or brass, which get the narrower by going farther."

Th. Hood

5.1. Introduction

5.1.1. General overview of copper production

• Ores

Although no archaeological traces were found that could point to the fact that copper (from ore to raw copper) was actually produced at ed-Dur, we will briefly summarize the production processes. This is only to put the copper and copper-base alloys in a broader frame.

Copper (Cu) occurs naturally in small quantities in various parts of the world. The existence of *native copper* is well known and compared to the other metals treated here it is relatively 'abundant'. All large and small copper ore deposits seem to hold some native copper. This was most probably the source for the very first copper objects.

There are many ores that contain copper, but they can be split up in two large groups. The first group contains the oxides, carbonates and silicates and the best know minerals are *cuprite* (red oxide of copper that is widely distributed), *melaconite* (CuO, black oxide of copper), *malachite* (green carbonate found in many regions), *azurite* (blue carbonate of rather limited distribution) and *chrysocolla*[1]. These ores can in principle be refined by heating and reduction with charcoal or wood, combined with a blast of air and a suitable furnace.[2]

The second group are the sulphide ores, mainly *chalcopyrite* of *copper pyrites*, which is de most plentiful and widely distributed of the sulphide ores, *chalcocite* (copper glance) and *bornite* (peacock ore). These ores are more difficult to reduce because they contain many impurities (mainly sulphur and to a lesser extent arsenic and antimony) that can have a negative effect on the quality of the copper produced. The harmful constituents had to be removed prior to smelting.[3] This was done by *roasting*, a process of prolonged heating at a temperature not exceeding 800°C to convert both the iron present in the ore and the copper sulphides to their oxide form[4].

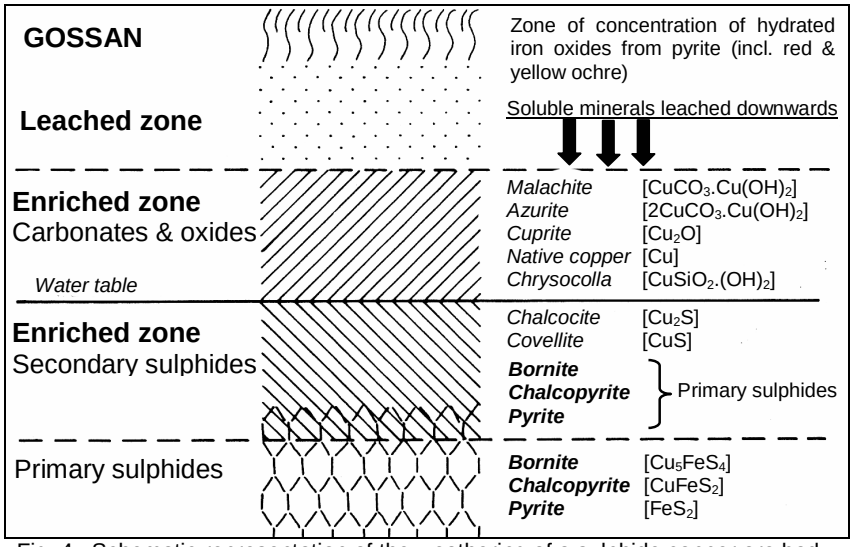


Fig. 4: Schematic representation of the weathering of a sulphide copper ore body (after Pigott, 2004: 31).

[1] This is a mineral with a variable composition but essentially a hydrated silicate with as a general formula of (Cu, Al)$_2$H$_2$Si$_2$O$_5$(OH)$_4$.nH$_2$O. The variant with predominantly copper in the formula is called *dioptase* (CuSiO$_2$.(OH)$_2$).
[2] Coghlan, 1975: 20; Moorey, 1994: 242.
[3] Coghlan, 1975: 20; Moorey, 1994: 242.
[4] Tylecote, 1976: 8.

The main primary copper ore-bodies have weathered into a succession of layers (Fig. 4). The upper zone can be very variable in thickness, from a few meters to several hundred. It is in this zone that the native copper, the oxides and the carbonates appear, resulting from the decomposition of sulphide ores underneath. This zone is usually associated with iron oxide on the weathered surface (*gossan* or *iron hat*). In the cementation zone below it, at the subterranean water level, a region of secondary enrichment forms by the precipitation of minerals which are more concentrated in copper. The are sometimes described by the general term of *fahl ores*, after the German term *Fahlerze* ('faded or pale discoloured ores')[5]. The thickness and depth of this layer of secondary sulphides (chalcocite and covellite) varies with the local conditions of deposition. Underneath this zone the parent ore of unaltered primary sulphides (pyrite, chalcopyrite and bornite) lays.[6]

When studying ore deposits used in the past it is important to keep in mind that the deposits available and used then were often not those that are used or appear today. Obviously some of the ancient beds have been worked out and completely exhausted. The oxide ores would in general have been available by simple collection or opencast working. The often bright colours of these ores may have made these deposits easy to find. Before deep mining the deeper horizons could only have appeared at the surface or in outcrops as a result of deep erosion. Additionally these sulphide ores are technically more difficulties to reduce.[7]

The history of the development of copper ore smelting is much more complex that the short summery below may suggest and the earliest copper production phases are not yet completely understood. A general division can be made, although this is not rigid at all, in that in the earliest phases especially the copper oxide and carbonate ores were used and later on the sulphide

- '**Simple' smelting of copper oxides, carbonates & sulphides**[8]

The temperature of a large, hot, wood fire will attain around 700°C. The oxide and carbonate ores of copper require temperatures in the range of 700-800°C for their reduction. In normal smelting practice when copper and slag are liquefied, a higher furnace temperature is required, 1100°C as a minimum. If the campfire is forced by a strong wind, there should be just enough temperature available.[9]

Basically the earlier processes (4th millennium BC) from the Middle East were neither sufficiently hot nor reducing to produce a real free-flowing slag. The actual installations must have been varied, but where refractories do survive there seems to be evidence of large rough open vessels or crucibles that appear to have been heated only from above. They probably formed the base or hearths of primitive furnaces, although little evidence has been found for any clay superstructure. Tuyeres are generally absent which may well suggest that bellows were not used. The use of blowpipes may be an alternative. The use of bellows during the smelting process may have been the crucial advance in energy production that enabled a true slag-forming process to develop as it did in the Middle East in the 3rd millennium BC.

When enough air is blown in the furnace the charcoal will burn to carbon monoxide (CO) and the oxide or carbonate ore will be reduced. Expressed in a simplified ore formula this gives:

Copper oxide: $Cu_2O + CO \rightarrow CO_2 + 2\ Cu$
Copper carbonate: $CuCO_3 + CO \rightarrow 2\ CO_2 + Cu$

[5] Craddock, 1995: 28.
[6] Tylecote, 1976: ix; Moorey, 1994: 242; Pigott, 2004: 31.
[7] Coghlan, 1975: 20-21.
[8] Mainly taken from Craddock, 1995: 127 & 135-139
[9] Coghlan, 1975: 28.

If too much air is present much of the carbon monoxide will be burnt to carbon dioxide ($2\ CO + O_2 \rightarrow 2\ CO_2$), leaving insufficient CO to reduce the ores.

Experimental smelts have shown that there is no problem in smelting copper from high-grade carbonate ores, producing little or no slag in an installation that would itself leave no enduring evidence. The archaeological evidence from the mines suggests that a wide range of minerals were encountered in the partially oxidised near-surface zones, including primary sulphides.

The general conclusion based on excavations of mines and smelting places is that whatever copper available mineral encounter was smelted. The initial thought that primary sulphide ores could not be reduced in these primitive installations has been contradicted by experimental work. Moreover chalcopyrite, when pre-treated, correctly can quite easily be reduced directly from their ores to metallic copper. When chalcopyrite is roasted the forming copper oxide reacts with the unchanged iron sulphides to give copper and iron oxides with the expulsion of sulphur dioxide. At temperatures of about 1230°C the following reactions take place:

$3\ Cu_2O + FeS \rightarrow FeO + SO_2 + 6\ Cu$
$5\ CuO + CuFeS_2 \rightarrow FeO + 2\ SO_2 + 6\ Cu$
$3\ CuO + FeS \rightarrow FeO + SO_2 + 3\ Cu$
$2\ CuO + S \rightarrow SO_2 + 2\ Cu$ (the S originates from the decomposition of the pyrite)

By roasting an ore (in the experimental case containing 71% of chalocpyrite, 17% of pyrite and 10% of silica gangue) at 700°C the chalcopyrite was converted first to copper sulphate and then to copper oxide, and the pyrite to pyrrhotite (FeS). The ore was covered with charcoal and heated to *ca.* 1250°C in crucibles. In all experiments metallic copper was recovered. If too little copper oxide was present, then the ores tend to form *matte* of copper sulphide (see below). Without the formation of slag the process still worked but was much slower and less efficient.

The process can be executed without the formation of a liquid *fayalite* slag, but specific care has to be taken in the pre-treatment of the ores. The small pieces of chalocpyrite had to be *dead roasted* (i.e. all sulphur was driven off from the surface and the core was left intact). These nodules were then crushed and smelted in a closed crucible. If there is more mineral than gangue, the copper will form metallic prills. Alternatively a portion of dead roasted ore could be mixed with untreated ore. This was than smelted in a crucible or a simple hearth covered with charcoal. Chalcopyrite melts already at 880°C. This molten mass would take the function of liquid slag to protect the forming copper droplets from reoxidation and the pool of molten copper beneath it. The molten metal however is expected to be high in sulphides (which is the case in much of the Bronze Age British metal).

The iron content can give an indication of the smelting process practised. In the simple process described above the iron minerals were not sufficiently reduced to be incorporated in the copper (i.e. around 0,03%), a situation different from the more complex slagging technique.

- **More complex smelting of copper sulphides or the *matte* process**[10]

Due to higher demand for copper the simpler process could not be used anymore. First of all the easily treated ores were not very abundant and the yield of a single smelt was low (a few hundred grams). The major advantages of the new slagging technique (already in use in the 3rd millennium BC in the Middle East) were the more efficient heat energy use and the higher yield of metal from lower grade ores. Additionally by tapping the slag, which otherwise

[10] Mainly taken from Forbes, 1964b: 18-19; Habashi, 1994: 53-54; Craddock, 1995: 146-153.

choked up the furnace, the process could be maintained for longer periods of time, allowing larger amounts of metal to be produced in a single smelt.

The process described so far basically consists of three steps: roasting, smelting and purification. Little is know however on the origins of the more complex treatment of sulphides by the *matte* smelting process. This is a process for treating the low-grade primary sulphide ore (e.g. chalcopyrite, which always occurs with other minerals such as pyrite) by successive transformation and concentration operations, finishing with a rather impure metal that required sophisticated refining.

In the first stage the beneficiated ore was partially roasted (roasting stage 1) to remove the sulphur as sulphur dioxide and chemically converts sulphides, chlorides and carbonates to oxides, which could then be removed by slagging. Additionally roasting renders the ore more friable, largely removes the arsenic, antimony and bismuth, as volatile oxides, and it drives off water present in the crystallisation. This process requires little fuel as it develops sufficient heat to maintain the roasting until the end.

Pyrite: $4\ FeS_2 + 11\ O_2 \rightarrow 2\ Fe_2O_3 + 8\ SO_2$ (slagging to remove iron oxides)
Chalcopyrite: $4\ CuFeS_2 + 7\ O_2 \rightarrow 2\ Fe_2O_3 + CuS + 4\ SO_2$ (slagging to remove iron oxides)

Most ores contain a good deal of gangue, so even after elaborate dressing the ore added to the furnace would typically only contain 20 to 40% of copper, the rest being silica in one form of another or iron oxides. This gangue had to be removed, and in more advanced furnaces this was done by slagging. A suitable flux was added and this combined with the gangue to form a molten slag that, because of a lower density, accumulated on top of forming copper and matte. This was the matting stage, and converted the remaining chalcopyrite ore to a mixture consisting predominantly of iron oxides and molten copper sulphides with some iron sulphides. Most copper production slags are iron silicates (*fayalite*, Fe_2SiO_4) and thus if the gangue was silica-rich iron oxides had to be added as flux, if the gangue was iron-rich, silica in the form of sand or crushed quartz had to be added tile the balance was correct. This fluid slag could be tapped periodically, while the matte accumulated at the bottom of the furnace. Additionally the liquid slag prevented the oxidation of already forming copper. Inadequate control at this stage leads to loss of copper in the slag and a quantity of impurities in the matte.[11]

$CuFeS_2 + 5\ O_2 + 2\ FeS_2 \rightarrow CuS + FeS + 2\ FeO + 4\ SO_2$ (slagging to remove iron oxides)
chalcopyrite *pyrite* *matte*

The *matte* was cooled, broken up and again roasted (roasting stage 2) to convert the sulphides to oxides, which could then be smelted in the usual manner by adding the necessary fluxes and an air blast. Now the remaining iron sulphide oxidizes and the iron oxide formed combines with the flux to form slag. The blast air further oxidizes the sulphides present and the sulphur escapes in the form of sulphurous gases, the charcoal present reduces the copper oxide forming copper. The second roasting stage and the smelting can also be performed in one step.

Roasting stage 2: $CuS + FeS + 3\ O_2 \rightarrow CuO + FeO + 2\ SO_2$
Smelting stage: $CuO + CO \rightarrow Cu + CO_2$

This raw so-called *blister* or *black copper* would then be refined. It typically contained only about 95-97% of copper, the rest being residual iron and sulphur from the ore. The last stage is the refining of the black copper and requires considerable skill. This is done by a combination of oxidation, slagging and volatilisation. To purify the black copper it was molten in an open vessel so the remaining impurities would oxidised. Care had to be taken not too much are was blown through the molten metal since the copper itself will then reoxidize.

[11] Craddock & Gale, 1988: 178-179; Charles, 1996: 22.

Plunging green twigs into the melt, a process known as poling, reduced the copper oxide inclusions. The great heat created clouds of steam and hydrogen and carbon-rich gases, which rapidly reduced the oxides back to metal. In the Roman period the technique of poling might already have been used, but the first textual evidence is only found in a text by Theophilus (*ca*. 950 AD). The refined copper has a copper percentage of 98 to over 99,5%.[12]

Complex though this appears, it is only a simplified outline of the reality as practised in post medieval operations throughout Europe, where a dozen or more operations of alternate roasting and smelting were performed.

• **Presence of iron, sulphur and/or matte**
In the Middle Eastern process of melting copper ore, some of the iron present in the ore or the flux was itself reduced to metal, and this metal appears in the slag in the form of finely dispersed particles. Iron is soluble in copper and did dissolve readily in the steady stream of copper droplets draining trough the iron-rich slag. Raw copper from Near Eastern sites regularly contains from 1-10% of iron. The necessary refining could be accomplished quite easily by melting the copper in an open crucible, the light iron-rich phase would rise to the surface and could be skimmed off, and as the iron oxidised it would react readily with clay of the crucible. Alternatively more silica in the form of sand or crushed quartz could be sprinkled on the surface. In this way it is possible to bring the iron content down to 0,5-0,3% in a few minutes. Iron could however be almost completely removed by yet another step of refining by blowing air over the surface of the molten metal, speeding-up the oxidation of the iron. However this would not have made a fundamental difference to the properties of the copper and this last step was probably not carried out.[13]

In contrast the early European bronzes contain almost no iron and the same goes for the early Near eastern cultures (Predynastic Egypt, Cycladic period in the Aegean, etc.), pointing to another technology in melting copper ore. It seems that the molten copper was not brought in contact with metallic iron, either because the technique was non-slagging or that the conditions in the furnace were only sufficient to reduce the copper oxides, but not the iron oxides[14].

Slags from prehistoric sites in Oman have been interpreted as the remains of matte smelting operation. The presence of copper sulphide droplets in the slag does however not automatically prove matte smelting. Any remaining copper sulphides either from an incomplete roasted of the sulphide primary ore, or a secondary ore where the presence of sulphides was not expected would have melted to form matte on smelting under reducing conditions. These finds could also represent practical lack of skill rather than technical sophistication.[15]

5.1.2. Historical frame of SE-Arabia

Because of the important role the Oman Peninsula played in its early days as copper provider for the Near East, this introduction will be somewhat more extensive.

One of the main interests of archaeological, archaeometallurgical and Assyriological research during the 20th c AD in the Gulf region was to locate the lands of *Dilmun*, *Magan* and *Meluhha.* These regions are already mentioned in the Bronze Age Sumerian, Akkadian and Babylonian cuneiform texts in connection to the supply of copper to Mesopotamia (notoriously poor in ores) in the 3rd and 2nd millennium BC. It has become progressively clear

[12] Craddock, 1995: 167.
[13] Craddock & Gale, 1988: 179.
[14] Craddock & Gale, 1988: 179.
[15] Craddock, 1995: 153.

that two of these entities are to be placed within the Gulf region. *Dilmun* was situated in the central Gulf, incorporating E-Saudi Arabia and particularly Bahrain and Failaka from the later 3rd millennium BC onwards. *Magan* was located at the southern end of the Gulf, incorporating SE-Arabia and, perhaps, some areas of SE-Iran. *Meluhha* probably refers to the Indus region (Harappan culture).[16]

From the textual evidence it can be deduced that in the 3rd millennium BC the land of Magan was the main provider of copper to the Mesopotamian heartland. Around 2000 BC Magan and Meluhha disappear from the economic texts and they seem to have been replaced by the land of Dilmun. Dilmun came to monopolise the trading network as the middleman between Mesopotamia, the Gulf-region and the maritime trade of the Indian Ocean. It is important to remind that in this period maritime trade flourished, but this does not mean that the traditional land routes were abandoned, but rather that they were supplemented by sea traffic. After *ca*. 1800 BC, Mesopotamia turned to alternative copper sources in Anatolia and Cyprus for its supply.[17]

To link the Mesopotamian textual information to the archaeological data from SE-Arabia a short overview of the region is given, starting in the Hafit Period dating to 3100-2700 BC. Till now there is little indication for local copper production during this period, although copper was already circulating as evidenced by the presence of copper objects in tombs and in the upper levels of some shell-middens dating to the 5th – 3rd millennium BC. In all periods of copper production in SE-Arabia, ore crushing and concentration was carried out using hand held hammerstones and large stone anvils. The earliest evidence of copper production in SE-Arabia occurs at Maysar 1 and is thought to be prior to the late 3rd millennium BC. A second smelting site at al-Batin was dated to 2500 BC by thermoluminescence .[18]

Small smelting furnaces or crucibles must have been used and oxides, carbonates and secondary sulphur-bearing copper minerals were processed. The reduction of the copper and the formation of liquid slags were reached under difficulties. The separation of the copper from the slag was far from complete, caused by the not fully liquid state of the slags during the smelting process under relatively low temperatures. The copper content of these slags, sometimes reached up to 30% and the presence of inclusions of metallic copper, cuprite and Cu-Fe-oxides point to a very inefficient copper production with a low technical level. The nature of these remains strongly suggests that these operations represent a phase of 'trail and error'.[19]

The first extensive copper production seems to have started during the Umm an-Nar Period (2700-2000 BC), as shown by the numerous slag heaps all over the Oman Mountains. A. Hauptmann mentions at least 20 sites with evidence of copper smelting. Un-doubtfully the evidence for many other sites was completely covered or destroyed by later mining activities, particularly during the early Islamic Period.[20] It is in this period that SE-Arabia is brought into connection with the land of Magan. Copper was probably exported and the island of Umm an-Nar might have acted as SE-Arabia's copper outlet. There is however evidence for extensive copper working in other regions too. South central Iran was also an important copper producer at this time with numerous mines in Kerman. Copper sources are also found in Central Iran and further to the East. The Iranian borderlands and SE-Arabia had close cultural and possibly economic relations during the 3rd millennium BC, which would fit the idea that Magan referred to regions on both sides of the Gulf.[21]

[16] Hauptmann, Weisgerber & Bachmann, 1988: 34; Carter, 2003: 31; Weeks, 2004b: 1 & 14-17.
[17] Carter, 2003: 31.
[18] Hauptmann, Weisgerber & Bachmann, 1988: 35; Weeks, 2004b: 24 & 29.
[19] Hauptmann, 1985: 113; Weeks, 2004b: 29-30.
[20] Hauptmann, 1985: 116-117.
[21] Carter, 2003: 37-38.

Calculations based on the amount of slag remaining by A. Hauptmann suggest a theoretical production output of 2.000 – 4.000 tonnes of copper in Oman during the Umm an-Nar and the following Wadi Suq period. This level of production is unknown from any other area of the Middle East at this time. The evidence for such large-scale production of copper is a strong indication to identify the modern Oman Peninsula as ancient Magan.[22]

The linking of the geographic region of SE-Arabia and the historical land of Magan based on the analyses of trace elements in copper-base objects is not as straight forward as originally thought. Early result seemed promising since various objects from the N-Gulf contained elevated levels of nickel, an element present in ores from the mountains of Oman. This in combination with the absence of nickel in ores from known mines in Cyprus, Anatolia, Iran and the Sinai, seemed conclusive evidence. Considerable doubt has now been cast on these results since high nickel content is also found in certain ore sources in India, also a possible provider of early copper. More recent and detailed analyses have also shown that the trade in metals, specifically copper, bronze and tin, is far more complex than previously thought.[23]

A limited amount of fully quantitative and semi-quantitative compositional analysis of SE-Arabian metalwork from this period has been undertaken. The great majority of the analyses showed that tin-bronze was very rarely used. Objects were predominantly of unalloyed copper, with the elements arsenic and nickel occurring in quantities of up to 4% or higher. This pattern is however strongly contradicted by the analyses of material from Tell Abraq where tin-bronze was used for more than half of the 31 objects analysed [24]. The importance of this should not be underestimated, since the Arabian Peninsula does not have tin sources. This suggests that an important external source of pre-alloyed bronze or unalloyed tin was supplying Tell Abraq during the 3rd and early 2nd millennia. This fact is not mirrored on other sites in SE-Arabia, where tin-bronze stays rather rare until the Iron Age. The tin-bronze metal at Tell Abraq could have originated from Afghanistan or Central-Asia and this single site. Even the unalloyed copper of Tell Abraq could not be linked to SE-Arabian ores, though this is not conclusive proof that no SE-Arabian copper was reaching the site.[25]

During this period mostly secondary copper minerals were smelted under low reducing conditions. Less frequently also sulphide ores were worked. Following the shape of the furnace fragments from Maysar 1, smelting furnaces with a height of 40 cm, a diameter of 40-50 cm and a volume of 10-15 litres were used. They were constructed of leaned clay. Probably ores with a copper content of up to 30% after dressing were available. Iron oxides and hydroxides were used as a flux. These ores are intensively intergrown with the siliceous country rocks. The sulphur-containing ores were not roasted. They were reduced in a one-step smelting process to metallic copper and concentrated matte, which separated easily from each other in the liquid state due to the coexistence of two liquid phases in the system Cu-Cu_2S. The formation of liquid slags was not always complete and the working temperatures in the furnace were determined at 1150-1190°C. Sometimes tapping has been carried out with only partially liquid slags. The raw copper produced together with the matte was separated from the slag by crashing. The sometimes high levels of sulphur and iron in analysed samples from the region indicate that significant amounts of matte can remain in this copper. In a next step the prills and lumps of copper were remelted to bigger pieces and finally cast into plano-convex ingots. There is no evidence of further pyrotechnological refining also not of the raw copper or the ingots. The further treatment of the matte is not clear. In the Umm-an-Nar period copper was produced in Maysar 1 only in a small workshop and probably for domestic use. At numerous other smelting sites from this time, however,

[22] Hauptmann, 1985: 108 & 115; Weeks, 1997: 16; Weeks, 2004b: 24.
[23] Carter, 2003: 39.
[24] Pedersen & Buchwald 1991; Weeks 1997.
[25] Carter, 2003: 39-40.

slag heaps of up to 4.000 tons are found (e.g. Wadi Salh, Tawi Ubaylah). They clearly demonstrate a copper production on an industrial scale in Oman during this period.[26]

The situation in the early 2nd millennium BC suggests isolation and lack of integration of SE-Arabia into the trading network. The material culture of SE-Arabia became very local in character and the cultural affinities with the Indo-Iranian borderlands are terminated. The new power that controlled the networks was Dilmun and it is likely that Dilmun gathered it's copper from a variety of sources and not only, if even, from SE-Arabia.[27] After the extensive evidence of production during the Umm an-Nar Period the situation during the Wadi Suq and Late Bronze Age period (1900-1300 BC) is less clear[28]. Numerous copper-base objects are known from burial contexts, but if primary copper production continued remains hypothetical at this moment. The only clear evidence for contemporaneous primary production are the copper mines on Misirah Island, which have been radiocarbon dated to *ca*. 1800 BC[29]. The very few Wadi Suq settlements that are known show no evidence of primary smelting. Moreover the presence of copper-base objects cannot be directly linked to contemporary production, since robbing of earlier tombs, recycling and trade were commonplace at that time.[30]

An increase in copper production has been suggested for the Iron Age[31] (1300-600 BC) linked to the first exploitation of the massive sulphide deposits in SE-Arabia. At least 20 archaeological sites related to copper extraction are recorded including large-scale mines, slag fields, and settlements for which copper processing was an important economic activity.[32] But also here the original number of site may have been much higher since the early Islamic activities were concentrating on the same ore bodies. Based on A. Hauptmann's calculations a minimum production of 7.000 to 10.000 tons of raw copper is suggested.[33]

A significant gap seems to exist in the evidence for copper production in SE-Arabia between the mid 1st millennium BC and the mid 1st millennium AD. The occupational phase of ed-Dur is situated in this period. Pottery from the Samad Period (4th-3rd c BC – 900 AD) has not been reported from any smelting site in Oman. Small-scale copper smelting in crucibles within settlements cannot be excluded though as is suggested by excavated material from Mleiha[34]. Ed-Dur did not yield any indication for this kind of on-site activity. The next evidence for production is provided by some slag samples from 'Arja (radiocarbon dated between the 5th – 7th c AD) and fall within the period when the Sasanians took control over the region.[35] At the pre-Islamic site of Jabal Kenzal (Saudi Arabia, North-East Province) NE of Hofuf, eight copper ingots (98-99% Cu) were reported. They weigh 4,5 kg each and are slightly pyramidal in shape. It is possible that this copper is from Omani origin, and was brought there to be used as minting material.[36]

The remains found at Mleiha need special mentioning since copper slag was found in several excavation areas (AH, BE, BV, CI, CZ/Area 7, E and L) and 'copper working remains' were attested in the fort (CW, room P715 and P740). The most informative areas are summed up beneath.

[26] Hauptmann, 1985: 113-114; Weeks, 2004b: 31 & 42.
[27] Carter, 2003: 40-42.
[28] Hauptmann, Weisgerber & Bachmann, 1988: 35.
[29] Weisgerber, 1988: note 6.
[30] Carter, 2003: 39; Weeks, 2004b: 24-25.
[31] Lizq Period in present-day Oman and Iron Age I & II in present-day U.A.E.
[32] Hauptmann, 1985: 116-117; Hauptmann, Weisgerber & Bachmann, 1988: 35.
[33] Hauptmann, 1985: 108-109; Weeks, 2004b: 26.
[34] Ploquin & Orzechowski, 1994: 30-32; Weeks, 2004b: 31.
[35] Weeks, 2004b: 26.
[36] Mouton, 1992: 200.

Area CW – the fort (see p. 403 for an plan).
In the fort there are two rooms that had traces indicating 'copper working'. At location P740 immediately under the first walking surface (with iron working traces) part of another living surface was encountered that yielded 'bronze' fragments and the remains of a furnace. P715 at the W-side of the internal courtyard also had some drops of a copper-base alloy and two furnaces, but no indication of metallurgical activities inside them.[37] The excavators connect these remains to a minting activity because three fragments of coin moulds were also found in the fort. It appears that metalworking was usually carried out in the storage rooms opening onto the courtyard, and that 'bronze working' and iron working occurred in the same areas, which could indicate that it was done by the same craftsmen.[38]

Area 7 (previously area CZ)
More recent excavations in 1993-1994, by the French archaeologists in cooperation with S.A. Jasim of the Sharjah Archaeological Museum, revealed a workshop for manufacturing 'copper or bronze' objects in area CZ. Within a wide trench measuring 20 x 20 m large quantities of copper slag and some iron slag were found. In the northern part of the trench there is a rectangular structure (4 x 3 m) containing two hearths near its southern wall. The hearth contained copper slag along with thick, heavy burnt pieces of pottery, possibly from a crucible that must have been about 20 cm in height. The floor of the room yielded a large quantity of copper slag. Outside the room three fire pits were attested associated with parts of another crucible. Nearby were several pits containing copper slag, one of which contained no less than 8 kg, small formless slags and a few small fragments of ceramic furnace/hearth lining. These pits may have served as waste pits and all date to PIR A. An 'ingot' (12 x 1,5 cm) of leaded tin-bronze pierced by an iron rivet was recovered from this sector, as well as several fragments of spouts. Whether this ingot was locally made or from imported ores/metal, was not investigated.[39]

Area E
Area E is presently situated on a farm but was originally an area at the edge of the eastern necropolis. This area with a level dating to PIR A yielded a large amount of copper slag, easily spotted at the surface due to regularly worked soil. The ancient walking level (opened up for 37 m²) is marked by a group of holes, similar to postholes, 20-30 cm in diameter and a depth of 15-35 cm. Their relation to metalworking could be considered. To the north, a ditch of 2 m wide and 0,85 m deep contained a fill of sandy earth and the traces of hearths. On the edge of a pit, 0,70 m in diameter, a ridge of reddened earth was registered. No other structures were found. Spread over the whole excavated area some 12 kg of slags was recovered. Some slags showed a regular, slightly convex side partly covered with a film of burnt earth. It is however not very likely that the ore was transported to the site because of the large volume of ore needed to produce copper. No moulds or tuyeres were found, but the slag indicates that some phase of a metallurgical process was going on.[40]

General
The copper slags from Mleiha are all fragmentary (2 to 3 cm), some are more angular than others. They are rich in metallic copper and often show a thin layer of lining attached to their outer side. No charcoal is visible in these slags. Their morphology shows great similarity with slags from the Iron Age recorded from the al-Madam plain. The initial thought of the researches was to interpret the slag as (fragmentary) 'furnace bottoms' (of smelting), since the production of bronze from its metallic components in principle does not produce slags. However certain morphologies were not very coherent with this model. This in

[37] Ploquin, Orzechowski & Briand, 1999: 175.
[38] Benoist, Mokaddem & Mouton, 1994: 13 ; Ploquin, Orzechowski & Briand, 1999: 175; Benoist, Mouton & Schiettecatte, 2003: 64.
[39] Ploquin & Orzechowski, 1994: 35 ; Ploquin, Orzechowski & Briand, 1999: 180; Jasim, 2001: 128.
[40] Mouton, 1992: 200-201; Mouton, Mokaddem & Garczynski, 1997: 26; Ploquin, Orzechowski & Briand, 1999: 173.

combination with the presence of crucibles fragments that show slaggy cuprous products on the concave face, points to a crucible process.[41]

It remains however un-clear if the slag is the result of small-scale on-site copper working, or of limited local copper extraction, or simply of the working of semi-finished imported metal. If they are the remains of small-scale production of copper in crucibles, the technique used was archaic. Iron ore in the form of the local pisolith deposits was added as a flux but still a considerable amount of copper was lost to the slag. The rather inefficient way of production (if it was production!) puts the slag much closer to similar but not identical Bronze Age slag found on the Peninsula than to the Early Islamic slag from Oman.[42]

No Late pre-Islamic copper extraction sites are known, but according to M. Mouton it is very possible that the copper that was worked at Mleiha, came from the Oman Mountains. To the north of Sohar and to the east of the Al-Aïn oasis a dozen extraction places were registered, more specifically in the region of Lasail and in Wadi Jizi. Most were exploited in the 3rd – 2nd millennium and during the Islamic period. Other Islamic extraction places were attested in Wadi Safarfir and Wadi Helu. All these places are situated close enough to the site of Mleiha to be potential extraction points.[43] Some late Sasanian period exploitation is also hypothesised on the deposits of 'Arja, Bayda and Raki[44].

Copper production in SE-Arabia reached its greatest extent in the Early Islamic Period, peaking during the 9th and 10th c AD. Working the massive sulphide deposits in the Oman Mountains generated enormous quantities of waste material (e.g. up to 100.000 tons of slag reported from Lasail) and destroyed most traces of earlier activity. A complicated and relatively advanced extraction technology was developed to deal with the primary, unweathered ores of pyrite, chalcopyrite and bornite. The process is best seen as a process of repeated stages of roasting and smelting. Each smelting operation produced 20-50 kg of tapslag and 5-6 kg of copper matte containing 50-60% Cu. Following this initial smelt, the refined copper matte was mechanically separated from the iron-rich slag, was roasted and re-smelted a number of times before finally being reduced to metallic copper. Most of the important sites in this period were located in the hinterland of Sohar, which provided the trading outlet for the enormous surplus of production. The output might have been between 48.000 and 60.000 ton of copper.[45]

From the middle Islamic to modern times (12th – 19th c AD) copper extraction is represented by much less archaeological evidence, reflecting drastically reduced levels of production and lower levels of technological understanding in comparison to earlier smelting operations.[46] Smelting was done in a one-step-process using very simply constructed bowl hearths, which were dug in the ground. At that time copper was produced from recycling the ancient slags that had sufficiently high copper content.[47] This shift is also found in textual evidence. The Early Islamic reports of the Arab historian and geographer Abul Hasan Ali Al-Mas'udi, dating to the late 9th – early 10th c AD, are amongst the earliest sources that state that copper was produced in the region of Sohar (see above). A later Persian manuscript from the 14th c AD by Al-Mustaufi mentions the production of gold, silver and iron in Arabia, but does not mention copper anymore.[48]

[41] Ploquin & Orzechowski, 1994: 31; Ploquin, Orzechowski & Briand, 1999: 179.
[42] Ploquin, Orzechowski & Briand, 1999: 180.
[43] Mouton, 1992: 199.
[44] Weeks, 2004a: 247.
[45] Hauptmann, 1985: 108-109; Hauptmann, Weisgerber & Bachmann, 1988: 35; Weeks, 2004b: 26.
[46] Weeks, 2004b: 26.
[47] Hauptmann, 1985: 114.
[48] Weisgerber, 1980b: 119; Weeks, 2004b: 17.

As can be seen above the copper production in SE-Arabia seems to show a kind of periodicity. Periods of intense production are alternate with period of no or very meagre evidence of production. Several reasons can be taken into account to explain this. First of all the enormous fuel requirements for big scale copper exploitation may have exhausted the region at times, limiting the output. In second factor was the demand. As today the growth of a region, depending on one major export product stands or falls with the demand. Social, political or economical instability on the markets can seriously disturb such a system. A third factor is the reuse of copper through grave robbing, since this provided an easy access to considerable amounts of metal previously taken out of circulation and reducing the need to produce new (much more labour intensive) copper.[49]

5.1.3. Geological context of Gulf region

The copper ore riches from the Oman Peninsula are known since long and I will limited myself here to a general overview of the occurrences and ores present. The geological context of the region has recently been thoroughly reviewed and discussed by L. Weeks and there is no reason to repeat this here.[50]

All of the copper deposits of SE-Arabia, with the exception of those on Masirah Island, are to be found within various geological units of the northern Oman or Al Hajjar Mountains. The Oman Mountain range consists mostly of basic and ultrabasic rocks. They build up the so-called *Samail Ophiolite* or *Samail Nappe*, which is part of an ancient oceanic crust. These copper, iron and zinc-rich ores are exhalative sedimentary deposits and were formed either in sea-floor depressions near oceanic ridges or as a result of sea-mount volcanism. The ore metals themselves originated in the volcanic rocks, from which they were dissolved by hydrothermal seawater activity. The copper deposits occur in nearly all lithological units of the ophiolite. All these deposits are believed to be of similar origin and are comparable in form and type with the famous copper deposits in Cyprus. It is estimated that there are approximately 50 major copper deposits and more than 100 minor deposits in the mountains of N-Oman. These deposits can be divided in two groups: the *massive sulphide deposits* and the *veins*. [51]

The massive sulphide deposits are generally composed predominantly of pyrite (FeS_2) with a copper content of 2-2,5%, chalcopyrite and sphalerite (ZnS). There are well-developed *gossans* of brightly coloured iron oxides, hydroxides (the *iron hat*) and sulphates in addition to secondary copper minerals such as malachite, azurite and rare native copper (see Fig. XX). The copper and sometimes many other metals, have been removed by downward percolating water. So, normally, in a deeper level around the water table a zone of mineral enrichment (cementation zone) occurs. In this zone mining was usually most intensive.

In Oman the situation was to some extent different, since there remained a lot of copper in the upper parts of the gossan. Rich ores are rare in these deposits, and zones of secondary enrichment are not known. The three largest sulphide deposits occur in the hinterland of Sohar (sites of Bayda, Lasail and ‘Arja), next to locations at Zuha, Raki, Hayl as-Safil and Daris. They were mostly exploited and worked partly by the ancient miners. These deposits of which only a few occur in the mountain range, are economically important today (for example at Lasail, ‘Arja and Bayda).[52]

An overwhelming part of the mineralizations and ore deposits in Oman form veins in gabbros and peridotitic rocks. The main copper mineral under the surface is chalcopyrite which is

[49] Weeks, 2004b: 35-36.
[50] Weeks, 2004b.
[51] Weeks, 2004b: 12.
[52] Weisgerber, 1980b: 115; Hauptmann, Weisgerber & Bachmann, 1988: 35; Weeks, 2004b: 7-13.

intergrown with small amounts of Fe-Co-Ni-As containing minerals. The outcrops are characterized by a distinctive and rich secondary copper mineralization with malachite, brochantite ($Cu_4SO_4(OH)_6$) and chrysocolla. These ores, which are of no economic value today, easily reach copper content of up to 30% or more after hand picking. Most probably these ores were the decisive impulse for the earliest copper production in Oman.[53]

In addition to copper deposits within rock units of the Semail Ophiolite, small copper mineralization can be found within the stratigraphic units that underlie the Semail Nappe. Both massive sulphide and vein-type mineralization is found within Hawasina rocks in the Sultanate of Oman (e.g. site of Al Ajal) and the U.A.E., but at the present there is no archaeological evidence for exploitation of these deposits.[54]

Although there is significant mineralogical variations between each of the major massive sulphide copper deposits in SE-Arabia, the levels of As, Sb and Ni in these deposits are generally very low. In contrast, the oxidized copper deposits of the lower crustal and mantle sequence of the Semail ophiolite, which are exceptionally abundant in comparison to other ophiolites, show much higher concentrations of Ni, As and Co (Ni up to 0,6%, Co up to 0,12% and As up to 0,2%). The most conspicuous feature of Bronze Age and Iron Age copper objects from Oman and Bahrain is their high levels of As and Ni (each up to 4%). These result from the smelting of arsenic- and nickel-rich ores embedded in ophiolithic rocks of Oman[55]. Copper ores with similar concentrations can however also be found outside SE-Arabia, most significantly in some of the copper deposits of the Iranian Plateau.[56]

Masirah Island (64 km long, up to 14 km wide) is located 24 km off the SE-coast of Oman. Geologically it contains a group of rocks that are very similar, but not identical, to the Semail Nappe. They are now believed to have been obducted later than the Oman Mountains.[57] The common feature of ophiolites is the occurrence of massive sulphide deposits. There are thus strong *a priori* reasons to suspect the presence of copper deposits on the Island. Copper mineralizations was reported already in the 1840's. However little archaeological work has been done on Masirah Island, but ancient slagheaps were reported and some radiocarbon dates indicate that copper mining was taking place at least as early as the 18th c BC.[58]

Fragments of cupels dating to the Abbasid period were found at sites situated to the NE of Medina (Saudi-Arabia). The mineralization of the Al-Naqrah South consists of polymetallic sulphides. According to preliminary research, copper was the main metal extracted, but gold and silver are present in economically profitable amounts and may also have been produced as secondary metals. This is one of the largest ancient copper mines on the Arabian Shield and was, therefore, a key industry in Abbasid Arabia.[59]

Important copper deposits are recorded in central and SE-Iran. The largest copper deposit in Iran is the Sar-Cheshmeh deposit. A number of copper deposits of Iran occur in geologically similar contexts to those of Oman (e.g. ophiolite-hosted massive sulphide deposits). Differentiating the metal produced from these Iranian and Omani deposits may prove difficult, as more samples become available, since they will increase the compositional overlap.[60]

[53] Weisgerber, 1983: 274; Hauptmann, Weisgerber & Bachmann, 1988: 35; Moorey, 1994: 243; Weeks, 2004b: 12.
[54] Weeks, 2004b: 14.
[55] Prange, Götze, Hauptmann & Weisgerber, 1999: 187.
[56] Weeks, 2004b: 110-111.
[57] Weeks, 2004b: 10.
[58] Weeks, 2004b: 13-14.
[59] de Jesus, Al-Surgiran, Rihani, Kesnawi, Toplyn & Incagnoli, 1982: 63-77.
[60] Hauptmann, Weisgerber & Bachmann, 1988: 42; Weeks & Collerson, 2005: 322.

5.1.4. Terminology

• **Definition of alloys**

This chapter treats the copper-base alloys. An alloy is the result of the intentionally or accidentally combination of two or more chemical elements of which at least one is an elemental metal. In this study only metallic alloys are considered of which the major element is copper. Alloying happens in the presence of heat and can be achieved in different ways: by smelting a poly-metallic ore, by co-smelting different ores, by co-smelting an ore and a metal or by combining previously prepared metals.

Alloy terminology	Composition
Unalloyed copper	Less than 2% of either tin or zinc and less than 4% of lead. In some cases more than 3% of impurities can be present.
Low or natural tin-bronze	Copper with between 2% and 5% of tin
Medium tin-bronze	Copper with between 5% and *ca*. 15% of tin
High tin-bronze	Copper with more than *ca*. 15% of tin
Brass	Copper with more than 5%[61] of zinc and less than 5% of tin
Gunmetal	Copper with more than 5% of zinc and more than 5% of tin
Leaded …	Alloy with more than 4% of lead

Table 5: Composition of copper-base alloys as used in this study.

In antiquity copper was basically alloyed on purpose with one or more of the following elements: tin, zinc and lead. Respectively creating *tin-bronze*, *brass*, *gunmetal* (when both tin and zinc are present) and the *leaded* variants if lead is present in a certain amount. To this we can add *unalloyed copper* and *billon* (if alloyed with silver, see *Chapter 6*). Table 5 gives the compositional definitions that are used for this study. In the literature there are by no means standard definitions of these alloys, and the terminology used in archaeological articles again differs from that used for industrially produced alloys nowadays. Modern metallurgists attach very rigid criteria to these definitions and would even suggest that some of the alloys described below, as e.g. brass is not a true brass at all. The ancient metalworkers used different criteria to name and use a certain alloy and the archaeometallurgical definitions try to incorporate (as far as possible) these consideration. The ancient approach was based on clearly noticeable physical changes, such as hardness, brittleness, colour, castability, etc.[62] One clear example, although not related to copper, is the use of the word *steel* (see also *Chapter 8*). In modern metallurgy this is iron to which one or more elements are added, no matter how small the amount. In the case of carbon (the most common of ancient steels), the archaeometallurgist will only speak of steel when 0,3% or more carbon is present. This is the minimum to make steel quenchable and significantly harder.

Table 5 is the result of the combined definitions given in several archaeometallurgical articles[63]. A more detailed discussion of every alloy will be given at the beginning of the appropriate chapters. The broad definition of the different alloy groups is also the result of the semi-quantitative character of the EDX-analyses that does not allow a very detailed subdivision.

A few more words should be said on the presence of lead. The border to determine whether an alloy is leaded or not, is not straightforward, also here different authors use different limits

[61] M. Ponting (2002: 559) uses 10% Zn as the minimum level. I did not do so, but even if this is applied only one sample defined as a brass falls outside this category.
[62] Ponting, 2002: 558-559.
[63] Dungworth, 1997b: 906; Ponting & Segal, 1998: 112; Ponting, 1999: 1312; Ponting, 2002: 559; Ponting, 2003: 87; Weeks, 2004b: 5.

varying from 1 to 5%. In fact all analyses should be considered and than it should be seen if there is a sharp break in the lead values.

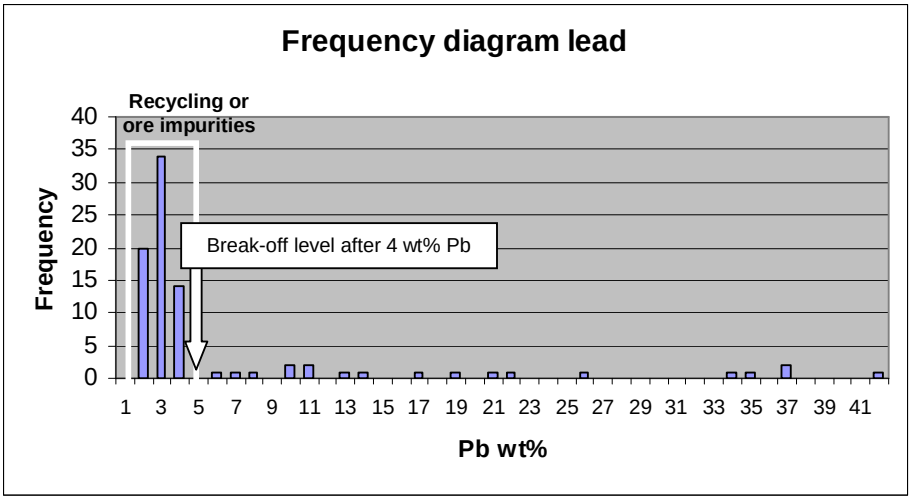


Fig. 5: Frequency diagram of lead wt% in all copper-base alloys.

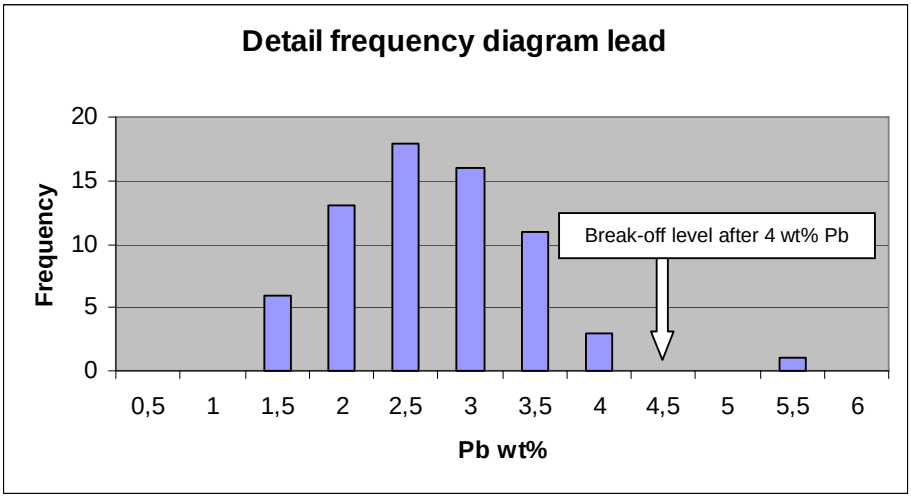


Fig. 6: Detail of brake-off level in frequency diagram of lead wt%.

Fig. 5 shows all the lead values of all copper-base alloys and the frequency that every value occurs. A clear break can be seen after 4 wt%. A detail of the diagram with a smaller interval can be seen on Fig. 6. For that reason 4 wt% of lead is used as the break-off level. Alloys with higher amounts of lead present are considered to be intentionally leaded. It should however be emphasised that the analyses presented here are semi-quantitative and that inaccurately high lead concentrations for some samples measured by EDX, were already reported by L. Weeks[64]. This was confirmed by some parallel ICP-MS at certain samples made by D. De Muynck. So care should be taken in considering the lead values. When 4% of lead is measured by EDX, this means that lead is present, but the amount is not reliable and certainly (much) less than 4%. No means to correct this error was found, but again since only broad groups are defined I think this will not bias the results and conclusions and in any case heavily leaded alloys can be sorted out.

[64] Weeks, 1997: Appendix A; Weeks, 2004b: 138.

Some additional information has to be given on the reason why some alloys were intentionally ***leaded***. Lead is always present in raw copper, as minor or trace element due to incomplete ore separation. There is however a big difference in the effect of these small amount (negligible) and the intentional addition of larger amounts of lead. The addition of lead can has several positive effects on an alloy. Lead lowers the temperature at which the alloy solidifies. It also improves the fluidity of the alloy (noticeable till 2% of lead[65]) for casting purposes, especially where the alloy was to be used in the casting of large and complex objects such as statues[66].

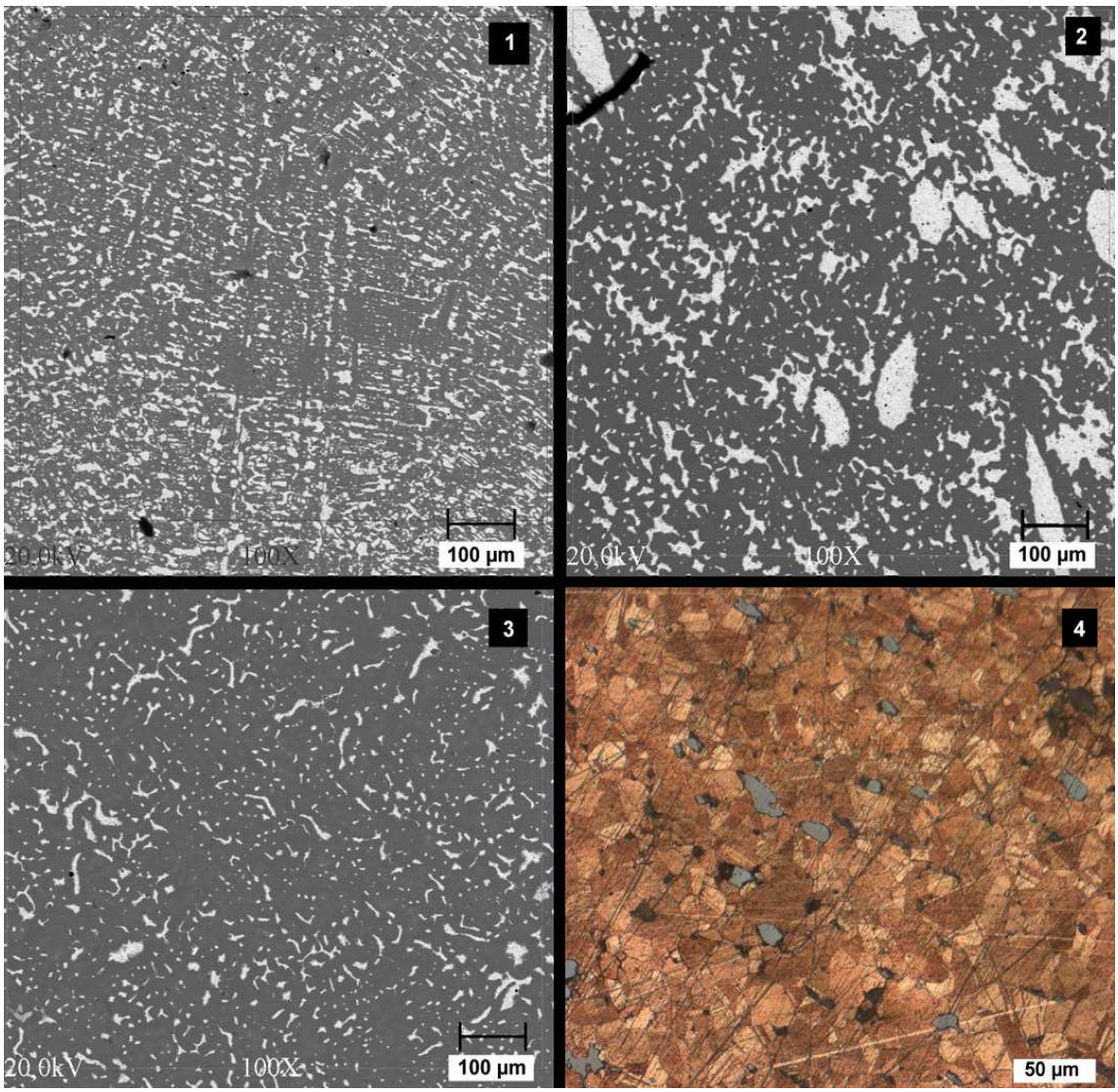


Fig. 7: Distribution of lead. (1: KR 008 – *ca*. 16 wt% Pb; 2: BS 154 – *ca*. 33 wt% Pb;
3: Z 146 – *ca*. 10 wt% Pb & 4: BQ 070 – *ca*. 3 wt% Pb).

The addition of lead also has the disadvantage in that lead does not dissolve into copper and forms minute globules throughout the metal.[67] Fig. 7 show examples of the segregation of lead. The microstructure consists of two distinct phases: lead and copper (or a copper-base alloy). Lead is seen as the white phase in 7-1 to 7-3 (SEM-BSE images) and grey in 7-4 (optical microscope). Practically all the copper will solidify before the lead-copper eutectic

[65] Staniaszek & Northover, 1983: 265; Treister, 1996: 341-342; Ponting & Segal, 1998: 115.
[66] Rosenfeld, Ilani & Dvorachek, 1997: 863; Weeks, 2004a: 248.
[67] Hodges, 1968: 216; Thornton & Ehlers, 2003: 6.

forms. This lead-copper eutectic is almost pure lead, as it consists of 99,9% of Pb and 0,1% of Cu. Ordinarily the separation of lead globules would be expected to result in massive segregation (still see the uneven distribution on Fig. 7-2) producing an unusable material. In practice the gross segregation is limited by the formation of a dendritic microstructure in copper-rich alloy castings (especially clear on Fig. 7-1). With high cooling rates, the lead is finely dispersed among the dendrites.[68] Still values of lead in analysed samples should be viewed with caution because lead can segregate in the liquid state (i.e. due to the impact of gravity when casting a large object). This can result in large differences in composition in different parts of the casting. Especially the analysis of a small sample without microscopic examination (e.g. drillings from large objects) may not be representative to determine the average composition[69].

Levels of above 10% of lead weaken the metal and it is more likely to crack when hammered and forged. Copper-base alloy sheets and wires almost never have more than 1% of lead[70]. Despite of this, alloys with high levels of lead were produced. This could be for the effect on the colour of the alloy[71], to add 'weight' to the alloy or to use a relatively 'inexpensive' metal in an alloy instead of the more 'expensive' copper and tin[72]. Pliny wrote in the 1st c AD that tin costing 11 times more than lead. This difference in value is largely due to the fact that lead was a waste by-product of silver extraction[73] or because it was a metal that could be produced at a low cost (i.e. easily reducible at low temperatures). This could explain the chronological changes in the alloy types used in the Roman world. There was an increased use of leaded bronze and leaded gunmetal, from 27% of the artefacts in the 1st c AD to 64% during the economical crisis in the 4th c AD[74].

In the *Pappae Clavicula*, a technical treatise probably written in the 8th c AD, an alloy is mentioned which contains one part of lead and four parts of copper or bronze, i.e. a 20% leaded alloy. This alloy is called *caldarium* (= 'copper for kettles') and is described as being very white in colour and useful for castings. The colour of the alloy is an interesting feature, and suggests a primary aesthetic consideration in the choice of alloy. The colour would have been a surface effect caused by *inverse segregation* of the lead or lead/tin-rich phase where the low melting point phase is squeezed to the surface of the object during solidification as a "lead sweat". In the Islamic period a similar alloy was use but instead of copper or bronze, brass was added as the copper-base, i.e. a leaded brass. This creates an interesting decorative possibility. If the white silvery-grey surface was pierced it would expose the golden brassy metal beneath, in this way giving the opportunity to have the two colours contrast without going through the trouble of making real inlay.[75]

- **Copper & copper-base alloy working**[76]

The basic principle of copper and copper-base alloy working, and the related terminology was already explained in *Chapter 4* (pp. 110-113). The most simple and earliest form of working copper was by cold hammering of a piece of native copper. To extend the process and reduce brittleness native copper can be annealed. The working of native copper is predominantly associated with the early metallurgy and is of no importance here. Very early the step was made to produce metallic copper from its ores. The raw copper was cast in ingots to facilitate transport and handling. This was done by letting the copper solidify at the bottom of the furnace, by poring it in simple open moulds or more complex two-piece moulds.

[68] Scott, 1991: 23.
[69] Unglik, 1991: 96.
[70] Staniaszek & Northover, 1983: 265; Dungworth, 1997b: 902; Northover, 1997: 328.
[71] Unglik, 1991: 99.
[72] Weeks, 2004a: 248.
[73] Treister, 1996: 341-342; Ponting & Segal, 1998: 115.
[74] Dungworth, 1997b: 907.
[75] Craddock, 1979: 75; Ponting, 1999: 1317.
[76] Coghlan, 1975; Renfrew & Bahn, 1997: 323-329.

The alloying of copper can be done at different stages of the process and these will be discussed in more detail in the parts on the respective alloys.

Once a block of raw copper or one of its alloys is produced this can be cold worked, possibly in combination with (repeated cycles of) annealing. It can also be hot worked, which makes annealing unnecessary. This technique is only suited for simple shapes, e.g. the production of simple blades or sheets. Of course these object can also be cast into shape. When more complex forms were desired the casting of the metal (pure or alloyed) was essential. Sometimes different part were cast separately and later joined together, e.g. by solder, or by the casting-on process. Two basic end results can be distinguished: the production of a solid object or of a hollow object.

The most widespread casting technique for more elaborate shapes is the *lost-wax* (*cire perdue*) process. Basically the desired object is first made from wax and then carefully encased with fine clay to make a mould around the object. The mould is then backed and the wax can be poured out. The clay mould is left over and took the negative impressions of the details of the original wax object. This mould can then be back-filled with molten metal. For larger object the wax was moulded around a clay core that was held in place and connected with the outer mould by pins. The lost wax technique allows producing objects with a lot of detail, but has the disadvantage that only one cast can be made. Removing the clay core after the metal has solidified makes a hollow cast. Casting seams, imperfections, etc. were removed afterwards and the object could be polished to take a smooth surface. To produce a series of identical object a more permanent mould had to be made that could be opened without having to be broken first.

After casting object could still be subjected to additional cold and/or hot working. They could also be decorated by incising, punching, coating with more valuable metals etc. This very basic overview will suffice as an introduction.

The microstructures included in this chapter are not always as 'good' as they should be, this is partly because of the 'skill' (and patience) needed to make good polished surfaces and partly due to the printing quality. Better resolution pictures can be found in the digital version of the dissertation included.

5.1.5. General conclusions on ed-Dur material analysed by L. Weeks[77]

L. Weeks analysed 33 copper-base alloy samples from ed-Dur by PIXE (proton induced x-ray emission) for the following elements: sulphur (S), titanium (Ti), vanadium (V), manganese (Mn), iron (Fe), cobalt (Co), nickel (Ni), copper (Cu), zinc (Zn), arsenic (As), selenium (Se), bromine (Br), silver (Ag), tin (Sn) and lead (Pb). Antimony (Sb), gold (Au) and bismuth (Bi) were also sought for but were below the detection limits. In a recent article on the assemblage of ed-Dur, only S, Fe, Co, Ni, Cu, Zn, As, (Se), Ag, Sn, and Pb are included[78]. PIXE could not be used in this research because it is not available at Ghent University. PIXE is a quantitative method and has a much lower detection than EDX and by consequence many of the elements included in his study could not be evaluated here. To present an as complete picture as possible the results of L. Weeks will be summarized here.

- Copper, tin, zinc and in some cases lead are the major and intended alloy elements. A number of alloys not revealed in earlier periods in SE-Arabia were detected at ed-Dur, being Copper-base alloys incorporating significant amounts of zinc and lead (i.e. *leaded tin-bronze*, *brass* and *gunmetal*).

[77] Weeks, 2000a; Weeks, 2004a: 242-248. The full analytical results are reproduced in *Appendix 3*.
[78] Weeks, 2004a.

- The concentrations of sulphur, iron and cobalt are similar to levels recorded in copper-base objects from contemporary and earlier archaeological contexts in SE-Arabia. In general these elements (Fe, S & Co) are contaminants coming from the used ores and going through the whole smelting process to partly end up in the finished products. Iron and sulphur can be addressed together because they frequently appear combined in non-metallic inclusions in copper objects as result of the exploitation of Cu-Fe sulphides and are both impurities that are generally removed during the refining process to improve the quality of the finished object.
- Arsenic and nickel are generally present below 1%. Two objects (sBS 1181 & sN 255) have a Ni-content over 1%, but this can still be explained by nickel-baring copper ores and is no indication of nickel alloying. Objects with significant Ni- and As-concentrations are typical for Bronze Age SE-Arabia, and are thought to represent the smelting of locally available As-Ni-rich ores. There are however also local As-Ni-poor deposits, so the low values of ed-Dur are not necessarily indicative of import. There is a difference between Ni-level linked to the alloy type: finished objects with more than 1% of nickel are from copper, and not tin-bronze.
- According to L. Weeks the lead-levels in the objects analysed are very high in comparison to the levels in samples from previous periods in SE-Arabia. The levels remain low however in the majority of the tin-bronzes (mostly < 5%) and very low in the copper samples (generally < 2500 ppm) when compared to much of the metalwork of from the Roman world (often more than 15 wt% of Pb).
- High sulphur concentrations are seen in the 4 objects (sBR 1176, sBS 1151, sBS 1326 & sBS 1487) with the highest lead concentrations. The median level of sulphur in the ed-Dur assemblage is much higher than in the previous periods. It is possible that this correlation reflects mineralogical/technological factors as lead is generally extracted from its primary sulphide ore (galena, PbS) and is thus likely to contain some sulphur. Additionally silver and lead values are commonly much higher than in the rest of SE-Arabia. This becomes clear if the material is compared to the Ag and Pb levels present in objects from the Iron Age. Whilst the Ag-concentrations are in all cases less than 1% and indicative of contamination from smelting, refining or alloying. It should also be kept in mind that silver was often produced from argentiferous galena.
- A positive statistical correlation between tin and lead might indicate that some lead was introduced into the copper along with the tin. This correlation is only clear for objects that are not intentionally leaded (less than *ca.* 2% Pb). It can be seen that objects with more than 2% tin almost always have higher lead (and silver) values.
- A correlation between silver and tin is also seen, and it seems that the alloying practices used to create the ed-Dur tin-bronzes may have introduced not only lead, but also small amounts of silver. Silver tends to be more correlated with tin-bronzes than with the unalloyed copper objects. It is possible that silver (and perhaps lead) were introduced with the tin during the process of alloying. Cassiterite (SnO_2) deposits are sometimes associated with silver mineralization. The ed-Dur samples are characterised by much higher lead and silver levels than found in objects from earlier periods in SE-Arabia.

The findings on other chemical elements were not very informative to use, since they could not be measured by EDX.

5.2. Unalloyed & leaded copper

5.2.1. Introduction

Pure copper has a melting point of 1084°C, a temper ature relatively easy to reach. The metal can be cast or can be wrought into shape. Copper is very ductile and soft, and can be drawn in long wires or hammered into thin sheets. Copper, as-cast, is soft and unless hardened is not suitable for the cutting edges of tools and weapons, but for the production of vessels from sheet metal a soft condition may be crucial. Pure copper is hardened either by the introduction of impurities (alloying, see below), which alter its physical characteristics. A second possibility is that the metal is work-hardened through the action of cold hammering (forging). This changes the elastic properties of the metal so the harder it becomes the less malleable it is, until it will eventually crack. Annealing may reverse this and returns the metal to a soft and workable state.[79]

'Pure copper' was available to the Romans but was rarely used to manufacture objects[80]. This is for examples completely different for Indian material, where copper had a more symbolic function and was used intentionally for certain object classes[81].

5.2.2. Sample description

- **This study**

Of the 97 samples analysed here 18 are of unalloyed copper and one is heavily leaded. The leaded specimen is actually an alloy and thus does not fall under the group of *unalloyed copper*. This is however the only example of this composition in the collection and is described here. If the samples from Khor Rori are not considered then 17 are of unalloyed copper and one is heavily leaded. Samples described as 'fragments' are pieces that could not be designated to a certain object. They are however not the remains of a 'production process' of any kind, but small parts of objects, except for sBO 1275. This fragment is a 'shapeless blob' that could be the remains of a spilling during casting (Table 6).

Reg. nr.	Area	UF	Sq	Loc	Team	Description
AW 063-1	AW	4541	XVI 5-6	G 5437	British	Ring-pommel dagger, scabbard
BK 005	BK	-	-	-	Belgian	Lion bead
BQ 153	BQ	-	-	-	Belgian	Lock plate
BQ 154	BQ	-	-	-	Belgian	Nail
BS 302	BS	6762	X 4	-	Belgian	Altar bead (gold plated)
ED 009	-	-	-	-	-	'Hook'
KR 012	-	-	-	-	-	Fragment from *Khor Rori*
M 084	M	-	-	-	Belgian	Nail
N 138	N	2420	IV 6	G 3840	Belgian	Altar bead
S 0012	-	-	-	-	Belgian	Shapeless fragment
sBJ 1237 A (2)	BJ	-	-	-	Belgian	Flat fragment
sBO 1275 B	BO	-	-	-	Belgian	Shapeless fragment (solidified 'blob'?)
sBR 1157 C	BR	-	-	-	Belgian	Rivet or end pin/needle
sBS 1129 A	BS	6609	VI 4	-	Belgian	Shapeless fragment
sBS 1429	BS	6751	VII 4		Belgian	Thick fragment
sM 1250 C	M	-	-	-	Belgian	Rivet
sN 1251 A	N	-	-	-	Belgian	Shaped fragment, square section
SX 001	C	01	C I	409	Danish	Fragment flat pin
Z 092	Z	-	-	-	Danish	Point pin/nail

Table 6: Co-ordinates of copper objects and samples.

[79] Hodges, 1968: 64; Coghlan, 1975: 76; Moorey, 1994: 249.
[80] Dungworth, 1997b: 904.
[81] See for examples Lahiri, 1995.

Table 6 gives the excavation co-ordinates, the excavation team and the description of each sample.

• **Previous analyses by L. Weeks**[82]

Of the 33 samples analysed by L. Weeks nine are of unalloyed copper. Six are described as flat fragments, one as a pin/awl and two as a possible chisel/nail. No specific details are given on the copper samples, but some things can be remarked based on the published results. Two samples (BS 1147 & BS 1181) have elevated levels of cobalt and arsenic and one of these samples (BS 1181) has the highest level of nickel attested in the analyses (1,27%). The presence of these elements is related to the use of ores containing these impurities and could be indicative of local ore use. The silver and lead levels are generally low, the only exception being BQ 1005 which has 950 ppm of silver associated with 0,2% of lead. Tin occurs in one sample (BQ 992) above 1% and four other samples have levels between 0,2 and 0,4%. The element selenium was only detected in six samples, five of which are unalloyed copper.

5.2.3. Microstructural observations – optical microscope

Sixteen samples were looked at under the optical metallographic microscope. First in the polished state, after which they were etched wit $FeCl_3$ to reveal the microstructure in more detail. The results of the observations are summarized in table 7.

Reg. nr.	As-cast	Annealed	Twin-lines	Strain lines	Extra
BQ 153		x	x	x	Annealing twins deformed near surface. Mechanical deformation twins (?). Strain lines. Large variety in grain size.
BQ 154		x	x	x	Heavily deformed grains and annealing twins in the head of the nail, indicating the final shape was given by cold working. A section through the shaft has a much less deformed microstructure. Grains between 50-100 µm.
BS 302	x				Remains of coring, but no dendrites, possibly moderately annealed after casting.
ED 009		x	x		Heavily cold worked near surface with grains deformed parallel to the surface. Deformed annealing twins more to the centre. Elongated inclusions in line of working. Grains *ca.* 50 µm or smaller (some larger ones too).
KR 012		x	x		Straight annealing twins. Grains not very well visible (*ca.* 50 µm) with remnant coring. Inclusions more or less banded.
M 084		x	x		Straight annealing twins, some possibly slightly deformed. Grains in the middle much larger (*ca.* 50 µm) than at surface (*ca.* 20 µm). Elongated inclusions.
S 0012	x	?			Large grains with remains of coring. Many pores and areas with 'eutectoid/eutectic' structure.
sBJ 1237 A (2)		x	x	x	Slightly deformed annealing twins in large grains (> *ca*. 100 µm). Strain or slip lines in certain areas.
sBO 1275 B	x				Many pores and large inclusions. As-cast structure with prominent coring.
sBR 1157 C		x	x	?	Heavily deformed grains and annealing twins. Grains small (< *ca*. 30 µm in length)
sBS 1129 A	x	x		x	Completely annealed to remove as-cast structure, but remnant coring. Deformed structure but no final annealing. No clear grains. Many small inclusions and cracks.
sBS 1429		x		x	Equi-axed large grains (200 µm or more), some deformed. Areas with 'eutectoid/eutectic' structure.
sM 1250 C		x	x		Straight annealing twins, grains *ca.* 50 µm or smaller.
sN 1251 A		x	x	x	Deformed annealing twins, some strain lines. Large variation in grain size from smaller than 50 µm to larger than 100 µm.
SX 001		x	x	x	Deformed annealing twins and strain lines. Grain size in centre larger (50 µm or larger) than near surface (10-30 µm)
Z 092		x	x	?	Heavily deformed grains and annealing twins near surface, not in centre. Elongated inclusions in line of working. Grains between 50-100 µm.

Table 7: Microstructural observations of copper objects and samples.

The copper samples can be divided in two large groups. The first group has an as-cast structure with coring. Equi-axed grains can sometimes be observed, but no annealing twins, indicating that the metal was not annealed after working. Dendrites are not observed, since the copper is relatively pure and no visually separate phases are formed upon cooling.

[82] Weeks, 2004a.

sBO 1275 B (Fig. 8-1) has very prominent coring. The sample was slightly over-etched, which explains the dark areas. Many cracks and pores are seen. The large light grey inclusions do not seem to be affected by the etchant (white arrow Fig. 8-1). S 0012 (Fig. 8-2) shows a similar microstructure with pores, remnant coring and large grains. The inclusions seem to be of a different nature then in sBO 1275 B colouring black and much more finely dispersed in the grains.

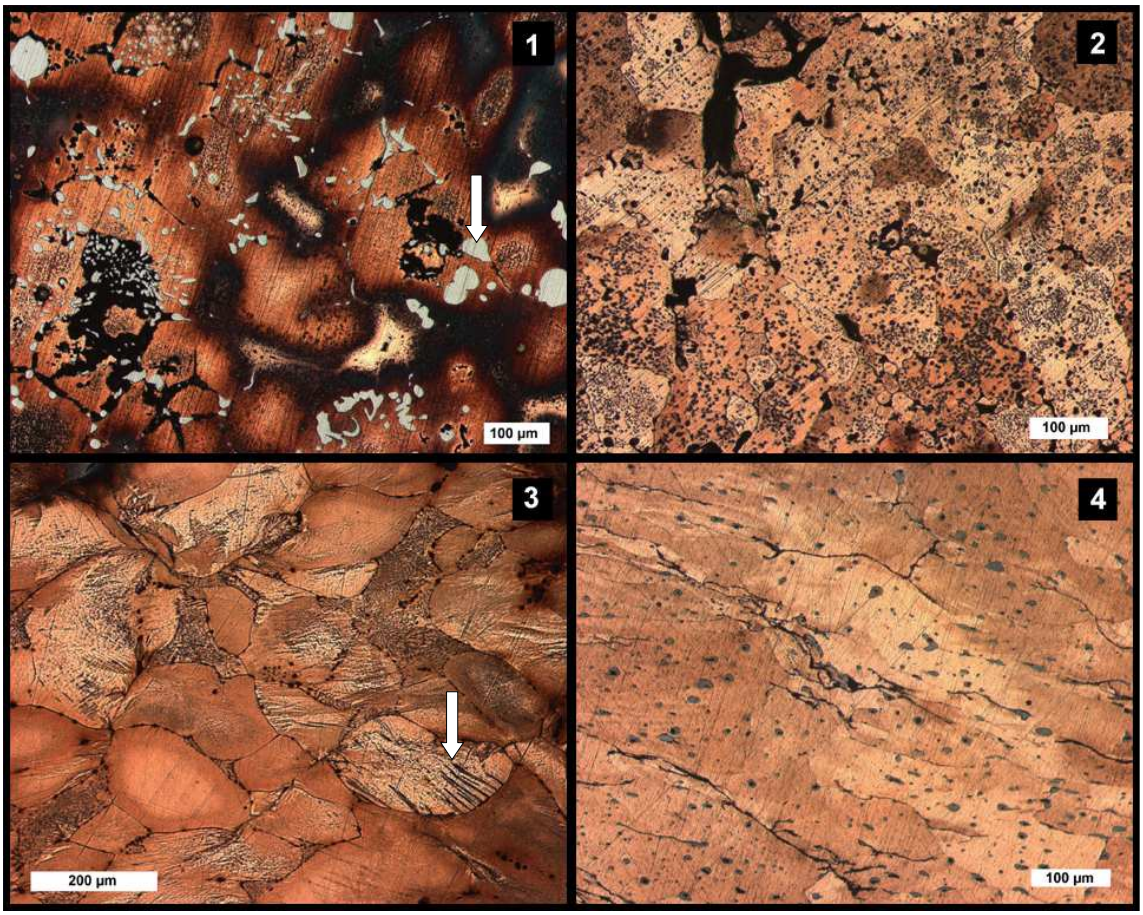


Fig. 8: Microstructures of sBO 1275 B (1), S 0012 (2), sBS 1429 (3) & sBS 1129 A (4).

sBS 1429 (Fig. 8-3) exhibits nicely formed rounded more or less equi-axed grains. No annealing twins were seen on the sample however, and the metal was annealed before working and left in this state. Some slip lines are visible (white arrow Fig. 8-3) and some of the grains were clearly deformed in a longitudinal direction. Not all grains have the small rounded black inclusions. sBS 1129 A (Fig. 8-4) has many (inter-granular?) cracks all in the same direction. These pores are most probably caused by the shrinkage of the cast metal when it was cooled. The etching did not reveal any particular microstructure. Remnants of the coring structure can be observed.

These four samples cannot be attributed to a certain object. They are rather amorphous and some (sBO 1275 B & S 0012) seem to be solidified spillage. These might be the sole shreds of evidence that metal might have been molten on the site. It must be stressed however that there is no other evidence to support the casting or production of copper or copper-base alloy objects from ed-Dur. Alternatively the fragments might have ended up as a fragment that accompanied imported objects or even originate from Mleiha, were some evidence of 'copper' melting was found.

BS 302 (Fig. 9-1) is the polished base of an 'altar bead'. No clear microstructure was found after etching, but coring and the remains of the dendritic as-cast structure can be resolved. The black lines that run more or less along the grain boundaries are not porosities of cracks, but inclusions. This small bead was cast to its definitive shape and did not receive any additional working, at least not in the base.

The inclusions seen in these first five samples are further discussed in the part with the SEM-EDX analyses below.

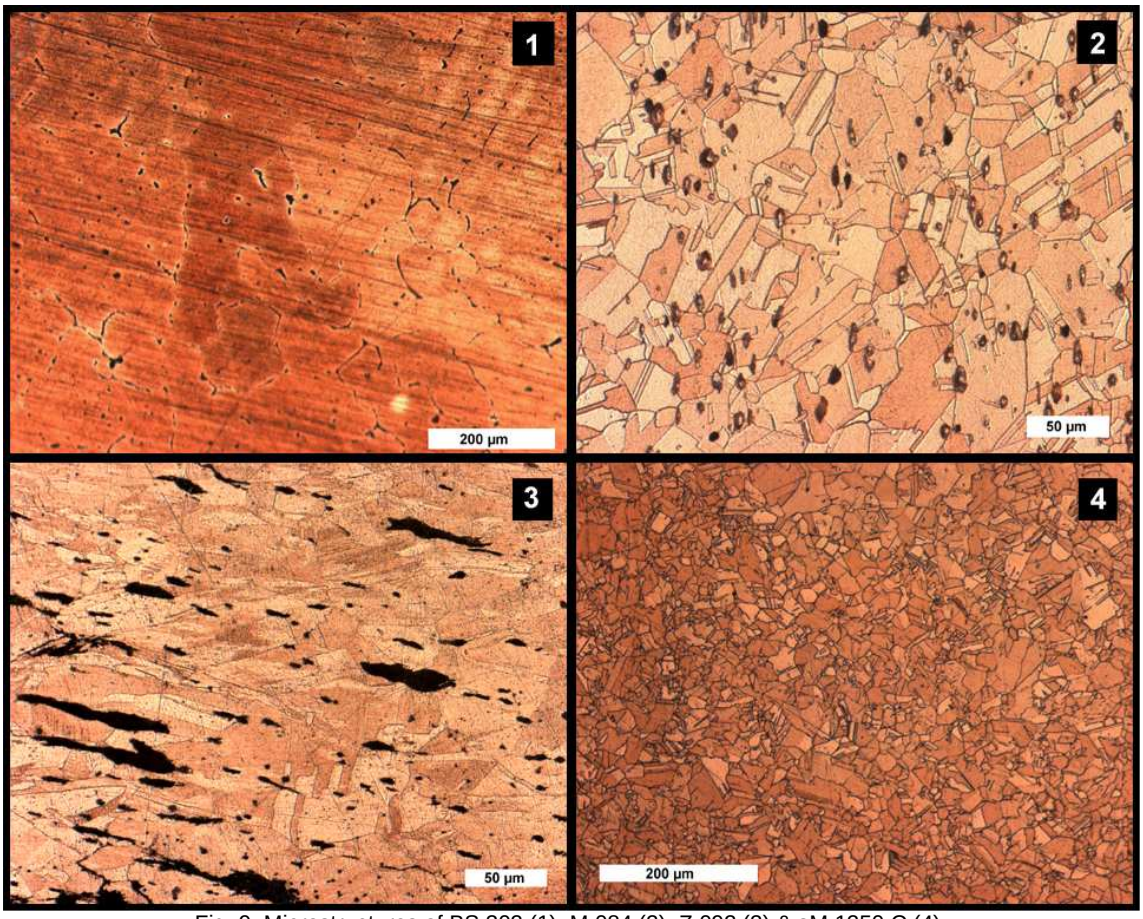


Fig. 9: Microstructures of BS 302 (1), M 084 (2), Z 092 (3) & sM 1250 C (4).

All other samples have annealing twins and show a more complex way of working. All were at least ones annealed after they had been worked. Many samples also show additional cold working to their final shape.

M 084 (Fig. 9-2) is a section through the shaft of a nail. No deformed annealing twins are seen and the variation in the grain size is rather large. The grains in the centre are much larger than at the surface. Many black inclusions are seen that are slightly elongated in the direction of working. The structure and grain variation is very similar to the one observed in sM 1250 C (Fig. 9-4), which is a small rivet. Also KR 012 can be added, although the grain structure is less well developed or at least less well brought out by etching.

Z 092 is the point of a pin or nail. A large crack in the middle of the metal can be visually seen in a longitudinal section, other elongated pores are also seen in the metal (Fig. 9-3). Some of the black dots are inclusions and some are pores that were originally filled-up. The centre of the pin exhibits nicely formed grains with annealing twins that are not deformed. At

the surface however the grains and annealing twins are deformed, indicating that the final shape was given to the tip after an annealing phase. The absence of any strain lines might indicate that this final phase was by hot working and not cold deformation.

ED 009 is a hook-shaped object and could possible be a handle of a vessel. The area near the surface shows severely deformed grain and proof that the object was cold worked to its final shape (Fig. 10-1). The inclusions are elongated along the way of working and the core material is much less deformed (Fig. 10-2).

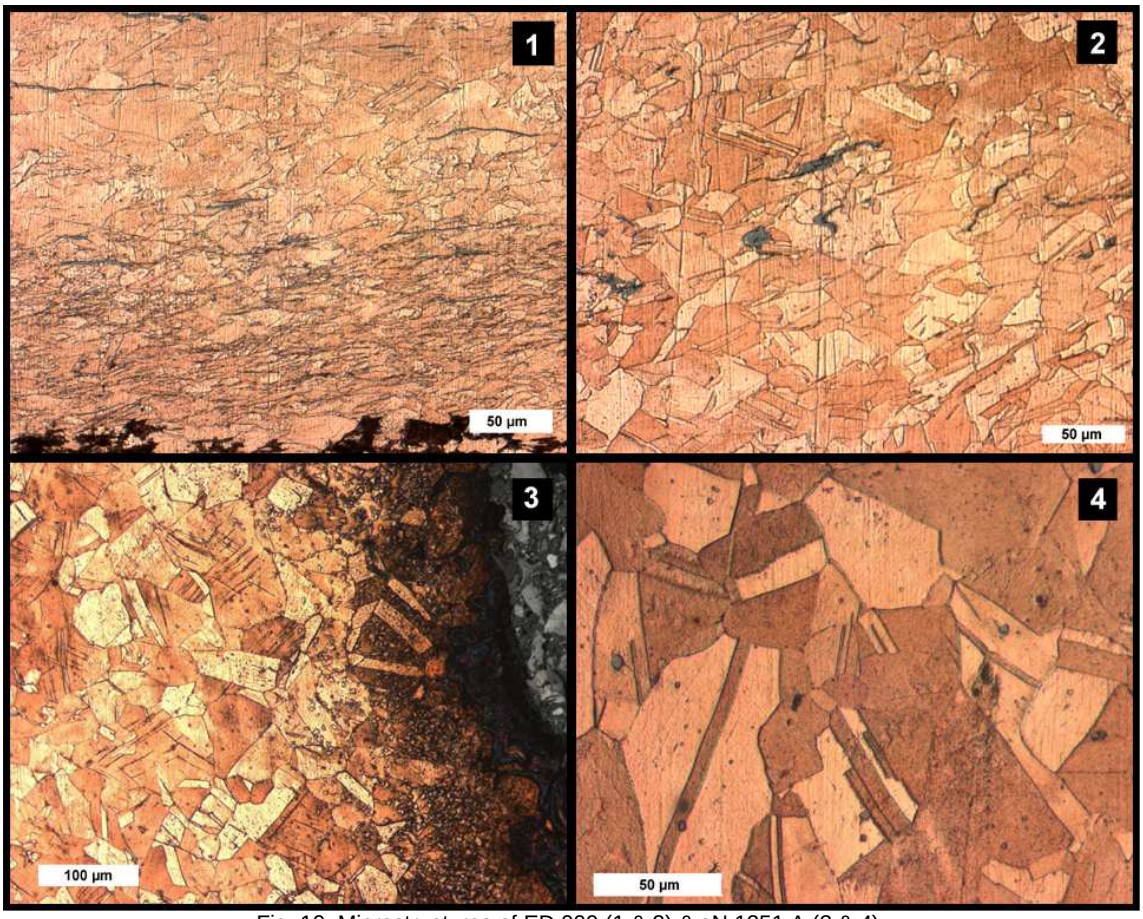


Fig. 10: Microstructures of ED 009 (1 & 2) & sN 1251 A (3 & 4).

Fragment N 1251 A (Fig. 10-3 & 4) has deformed annealing twins and strain lines in the grains near the surface. This piece might have been finished by some cold working, or by hot working.

BQ 154 is a nail and Fig. 11-1 and 2 show the microstructure along a longitudinal section. Fig. 11-2 shows where the head is bent over, so the transition between shaft and the head. The grains are severely deformed, as are the annealing twins. Fig. 11-1 is taken from the centre of the head and also exhibits a very distorted microstructure. The head was obviously hammered in shape after casting. The grain structure of the shaft (not illustrated) is less deformed.

The small rivet BR 1157 C (Fig. 11-3) has small very heavily deformed grains. Elongated inclusions, deformed annealing twins and cracks are all indicative for the fact that this object was heavily cold worked to its final shape.

The flat fragment BJ 1237 A (Fig. 11-4) possibly is a part of a vessel. It shows large grains with slightly deformed annealing twin. The metal for this plate was probably worked to shape and annealed, followed by some additional working.

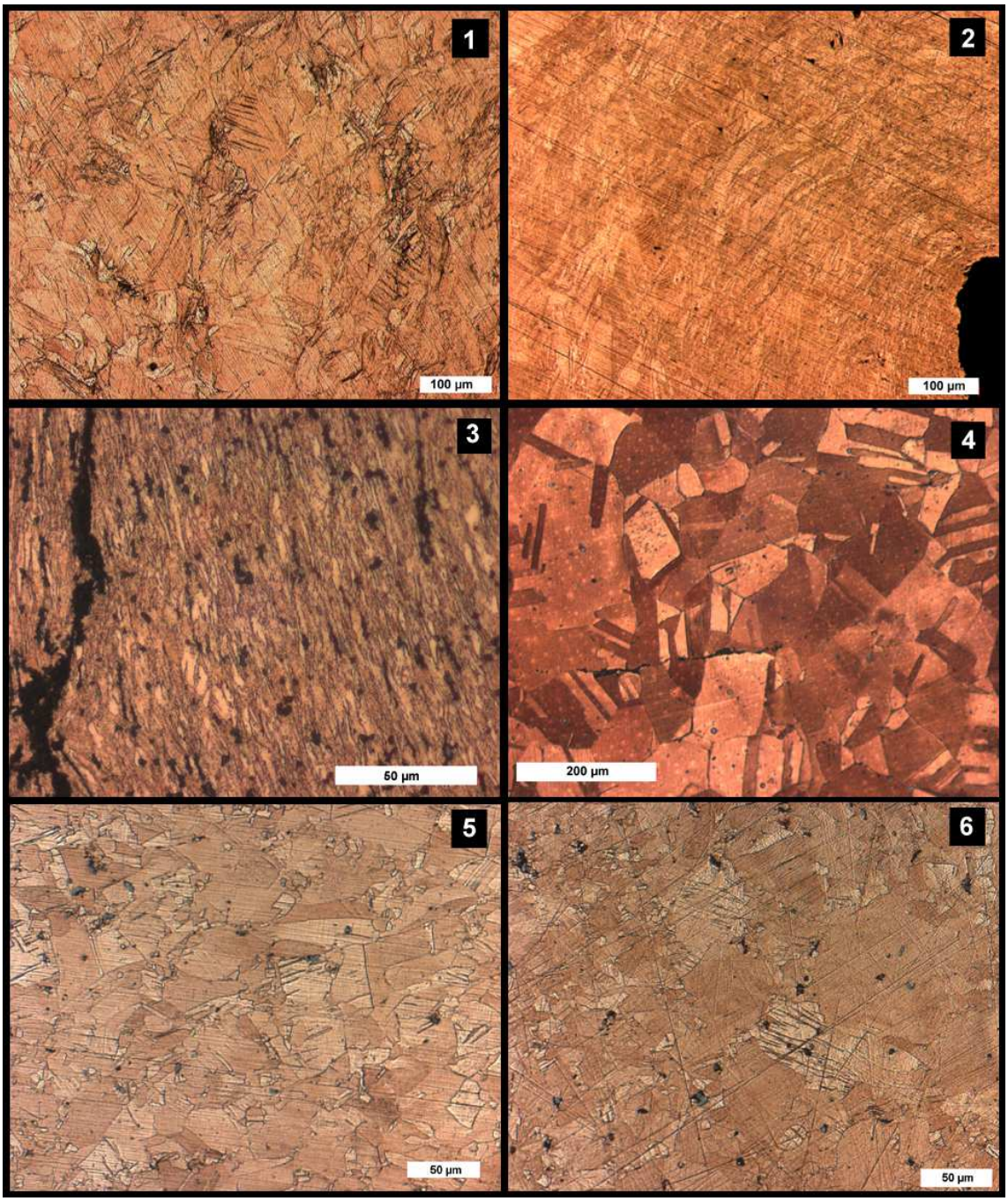


Fig. 11: Microstructures of BQ 154 (1 & 2), sBR 1157 C (3), sBJ 1237 A2 (4), SX 001 (5) & BQ 153 (6).

Deformed grains and annealing twins and strain lines are observed in the pin SX 001 (Fig. 11-5). The grains are larger in the interior than near the surface.

BQ 153 (Fig. 11-6) is a fragment of a lock plate. Deformed annealing twins and strain lines are seen in a greater amount near the surface. Mechanical deformation twins are possibly

also attested. The final handling of this lock plate was cold working to flatten it and hammer in the decoration, or to fit the plate on the wooden (?) substrate.

5.2.4. Microstructure & chemical composition – SEM-EDX results

Reg. nr.	Cu	Zn	Ag	Sn	Pb	Extra
AW 063-1	98,15	-	-	0,44	1,42	Considerable amounts Cl & O
BK 005	56,35	-	-	0,87	42,78	1,62 wt% Fe - 0,59 wt% Ni – 0,50 wt% Ag
BQ 153	96,14	-	-	0,89	2,97	
BQ 154	96,25	-	-	0,57	3,18	
BS 302	98,39	-	-	-	1,61	
ED 009	97,73	-	-	-	2,27	
KR 012	98,33	-	-	-	1,67	
M 084	96,17	-	-	0,56	3,27	
N 138	96,58	-	-	1,10	2,32	
S 0012	97,12	-	-	0,56	2,33	
sBJ 1237 A (2)	97,51	-	1,38	-	1,11	
sBO 1275 B	95,93	-	-	-	3,64	0,73 wt% S - 2,43 wt% Fe
sBR 1157 C	97,80	-	-	0,61	1,59	
sBS 1129 A	98,43	-	-	-	1,57	
sBS 1429	96,93	0,83	-	-	2,24	
sM 1250 C	95,46	-	-	1,01	3,53	0,67 wt% Fe
sN 1251 A	96,66	-	-	1,05	2,29	
SX 001	96,35	-	-	0,97	2,68	
Z 092	95,41	-	0,94	0,45	3,20	

Table 8: Chemical compositional data of copper samples and objects in wt%. Grey squares in the Zn-column indicate that the zinc values were removed before quantification.

The only leaded copper object, BK 005, is a small lion bead. The high amount of lead present (Table 8) in this alloy probably served to increases the liquidity of molten copper. To preserve the detail in such small cast objects a very liquid melt is needed. The fast cooling and the small volume of the object may have resulted in an unequal distribution of the lead in the metal. Possibly the lead level measured here is too high because a lead rich zone was analysed.

In AW 063-1, a piece of metal fitted on the scabbard of a ring-pommel dagger, chlorine and oxygen were present in relative large amounts. This sample does provide some information though, since not even traces of zinc or tin could be detected, it is possible to conclude that a rather pure copper was used for this part of the scabbard sheeting.

In more than half of the analyses small amounts of tin were attested. This can be the result of the ore used, but also due to recycling and the inclusion of small fragments of bronze during remelting. This latter explanation can also explain the silver and zinc found in a few samples.

Four types of inclusions were attested in the unalloyed copper samples. The inclusions were already pointed out in the part on the microstructure. The inclusions of the 'eutectic' structure seen in BS 1249 A under the optical microscope (Fig. 12-1) turned out to be Cu-O particles. They appear in an elongated and a more rounded form (Fig. 12-2, ***1*** & ***2***). The white dots (***3***) are lead globules. The rounded inclusions in S 0012 (Fig. 12-4, ***6***) are also Cu-O enclosures. Similar black Cu-O inclusions are seen in many of the unalloyed copper samples. These are attacked more aggressively by the etchant ($FeCl_3$) and turn black under the optical microscope. They are often associated or 'imbedded' in a lead inclusion. D.A. Scott states

that unalloyed copper is difficult to cast without the formation of copper oxide[83]. This is confirmed by their presence in many of the samples looked at here and Cu-O inclusions appear more frequently in the unalloyed copper than in the other copper-base alloys.

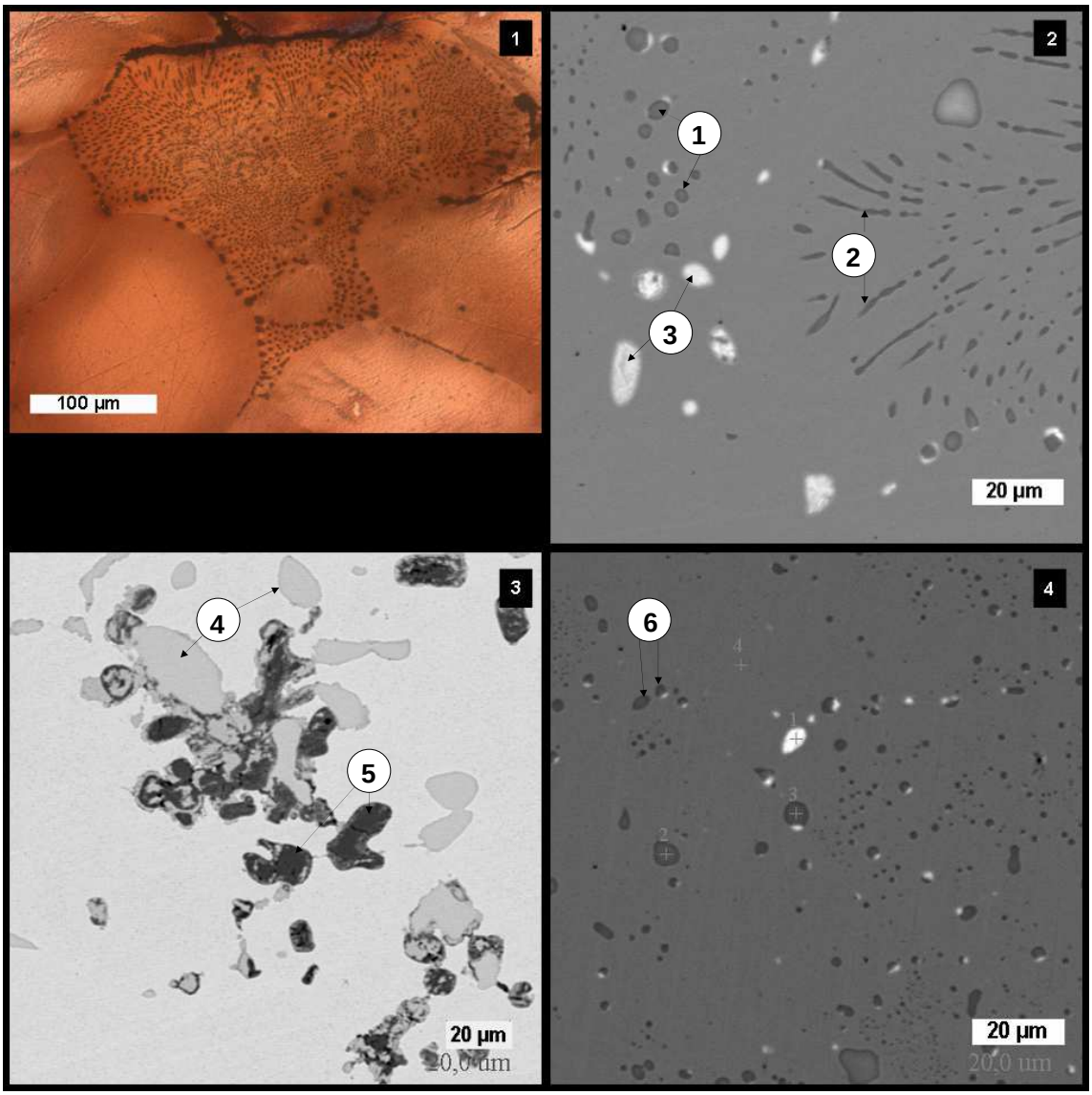


Fig. 12: Inclusions in samples sBS 1249 A (1: OM; 2: BSE-image), sBO 1275 (3) & S 0012 (4).

The inclusions in sBO 1275 (Fig. 12-3) are of a different composition. The large light grey particles (***4***) contain Cu-S. These were not discoloured by the etchant and this is conform to the statement that Cu-S inclusions are not attacked by the $FeCl_3$[84]. The dark grey particles are (Cu)-Fe-O enclosures (***5***). The presence of these impurities may be in accordance with the remains of *matte*. As seen above (pp. 135-137) this matte can originate during the smelting of a sulphide ore by a simple process or by using the more complex *matte process*. The iron rich inclusion can be the remnants of the addition of an iron oxide flux to facilitate the formation of a liquid slag. The inclusions explain the relative high levels of sulphur and iron in this sample.

[83] Scott, 1991: 92.
[84] Scott, 1991: 92.

5.3. Tin-bronzes

5.3.1. Introduction

After the discovery of copper smelting the invention of alloying copper with other elements must have been one of the most important achievements of the early metallurgists. *Bronze* is probably the best-known and most commonly used copper-alloy from antiquity and it remained popular long after. Many artefacts with a green corrosion layer are automatically classified as bronzes, although this is by no means certain. Within this dissertation the SE-Arabian coins are an example of that, many coins were described as being of bronze, whereas in this collection no real bronze coins are present. A second example is the *brass* attested within this study. These objects would have been classified as bronzes without compositional analysis. It should be made clear that when referring to bronzes, these are tin-bronze (since other 'bronze' alloy types do exist e.g. arsenic-bronzes, etc.).

The alloy element for a tin-bronze obviously is *tin*. Tin is a soft, white metal with a low melting point of 232°C. The main minerals of tin are *cassiterite* (tin oxide, SnO_2) and to a much lesser extent *stannite* (a sulphide, Cu_2FeSnS_4). Tin does not occur in nature in its metallic form. Tin deposits are somewhat of a rarity and they are associated with certain types of granite and occur in only a few well-known localities such as Malaya, China, Bolivia, Cornwall, Saxony-Bohemia and Nigeria[85]. An important secondary source next to the ore veins was *stream tin*, alluvial deposits as a result of erosion of cassiterite deposits. The tin in these deposits is very resistant to chemical and physical attack, and it was easy to exploit.[86] The origin of the tin used in early tin-bronzes is a notorious problem in the archaeology of the early Bronze Age, especially in the Middle Eastern region. Although much progress has been made in recent years, some of the enigma still remains. L. Weeks exhaustively discussed this subject[87]. This is however not of any consequence for the period treated here, where trading networks were well established and tin could have been imported from 'anywhere'.

What is important to stress is that all tin present in objects from SE-Arabia was *certainly* imported. Tin deposits are not known and are unlikely to occur in the basic and ultrabasic rocks, which comprise the majority on the Oman Mountains. Geological studies report tin concentrations in local rocks in the order of approximately 10 ppm or less. L. Weeks states that if more than 0,5% of tin is found in a SE-Arabian object it certainly includes foreign material.[88] The question remains if this tin was imported as ingots and alloyed with local or also imported copper, or if it was brought in the form of pre-alloyed tin-bronze ingots or finished objects. A last sources may have been the recycling of older tin-bronze object.

In the case of ed-Dur it is very likely that finished objects reached the site since there is no indication that copper-base alloys were produced or processed at the site (e.g. no crucibles, moulds, production waste, etc.). Activities that do not leave any material evidence such as the repairing of objects or manufacturing of new objects by cold or even hot-working scrap bronze can of course not be excluded and are even very likely to have occurred. It might be interesting to mention that a small bent fragment (K 066) was made of unalloyed tin. Admittedly the sample was completely corroded, but some sound metal islands were preserved and moreover the corrosion products also did not hold any copper-oxides. It is safe to conclude that this piece was unalloyed tin.

Several methods do exist to produce a tin-bronze. A first and obvious one would be the addition of tin to molten copper. It is however more probable that tin (if available as metal) was put in the crucible with pieces of copper under a charcoal cover and both were heated together. The tin will melt at 232°C and some of th is will diffused into the copper and lower

[85] Tylecote, 1976: 14.
[86] Forbes, 1964b: 126-127; Moorey, 1994: 297.
[87] Weeks, 1999; Weeks, 2004b: 165-195, also see these publications for additional references.
[88] Weeks, 2004b: 121.

the melting point of the latter. This was an interesting side effect that eased the problem of melting. A second method was to add tin as an oxide under the form of crushed cassiterite. When this is done under reducing conditions the oxide is reduced to metal. This is an easier process than reducing the cassiterite separately to tin first. A last possibility would be the use of naturally occurring copper-tin ores or the smelting of a copper-ore together with a tin-ore (i.e. cassiterite).[89]

The addition of tin to copper hardens the metal as long as it is not excessive, since this makes the alloy brittle. It also lowers the melting point of the copper-tin melt from 1083°C to 1050°C if 5% tin is present, to 1005 °C with 10% of tin and even to 960 °C with 15% of tin. A last advantage is that tin increases the liquidity of the metal and creates a considerable change in colour.[90] For many purposes tin-bronze is much more useful than unalloyed copper.

Theoretically a subdivision can be made between *low tin-bronze*, *medium tin-bronze* and *high tin-bronze*. A phase diagram of the behaviour of the copper-tin system at different temperatures and concentrations can be found in *Appendix 6*.

- **Low or natural tin-bronze**

Lower percentages of tin are problematic, since they involve making the distinction between a natural and an artificial alloy. This limit has been much debated and is, anyway, difficult to define. There is a tendency to argue that 5% is a good division since the ancient metalworkers are unlikely to have appreciated the effect of tin in lower concentrations[91]. Bronze with less than 5% of tin does not differ much in colour and mechanical properties from pure copper. It is only when more than 5% of tin is present that copper becomes noticeably harder.[92] With low amounts of tin 2-5% it is possible that all tin is absorbed in the dendritic growth, depending on the cooling rate resulting in single alpha phase (α-phase) alloy.[93]

These bronzes can be the result of the smelting of a tin-containing copper ore and are as such not intended to be an alloy. Low amounts of tin can also be the result of recycling scrap bronze together with copper. Care should however be taken in using this 5% boarder for SE-Arabian material. Natural alloys from local ores cannot contain more than 0,5% of tin since the amount of tin present is always low (see above).

- **Medium tin-bronze**

When copper is alloyed with a small proportion of tin, a solid solution of tin in copper, the α-phase, is formed on solidification. Initially at a certain temperature the dendrites will contain only a small proportion of tin, but as the dendrites grow the proportion of tin and copper in the remaining liquid phase will increase. The further outgrowth of the dendrites will contain increasingly larger proportions of tin, while the final infilling will contain an even higher proportion of tin. As cooling in antiquity was fairly rapid, as-cast medium tin-bronzes often appear in cross-section as cored structures of copper-rich dendrites with an infilling of tin-rich material, alpha-delta intermetallic *eutectoid* (α+δ eutectoid, 27% Sn). A *eutectoid* structure is formed by the decomposition from a solid phase (*ca*. at 520°C) into two finely dispersed solid phases[94]. In this case the α-phase is copper-rich and the δ-phase ($Cu_{31}Sn_8$, 32,6% Sn) tin-rich. The α+δ eutectoid is hard, brittle and 'blue' in colour.[95] This medium tin-bronze region has a wide *solidus/liquidus* separation, i.e. wide freezing range. This produces the coring

[89] Coghlan, 1975: 35; Tylecote, 1976: 15-16.
[90] Moorey, 1994: 252.
[91] Moorey, 1994: 252.
[92] Dungworth, 1997b: 905.
[93] Scott, 1991: 25.
[94] Scott, 1991: 140.
[95] Scott, 1991: 15.

within dendrites and can readily lead to *"tin sweat"* on the surface of cast bronze. Tin sweat actually involves the expulsion of a tin-enriched alloy and the formation of the α+δ eutectoid microstructure on the surface, rather than tin itself.[96]

A bronze with 10% of tin seems to have been the optimum composition in antiquity. The maximum theoretical limit of the solubility of tin in the copper-rich solution is 17%, but in practice this is 15% and it is rare to find bronze with this tin content in a single phase[97]. Till 10% of tin bronze can still be easily cold worked, once above this amount bronzes cannot be satisfactory cold deformed without some danger of breaking. This is due to a hard and brittle α+δ eutectoid formed in the metal. Therefore most ancient alloys have less than 15% tin present and can be cold worked if periodically annealed. Medium tin-bronze consists mainly of the α-solid solution of tin in copper and the α+δ eutectoid normally starts to appear in limited amounts in the structure of cast bronze containing more than 8% tin. However proper annealing of bronze with up to 15% of tin can result in a solid solution of only the α-phase.[98]

The addition of tin to copper also has an effect on the colour of the alloy. It gets a golden appearance up to about 15% after which the alloy becomes increasingly silvery in colour[99]. With the introduction of brass in the 1st c BC – 1st c AD the use of bronze declines, a trend that continued during the 1st millennium AD in Europe and the Middle East. In this context it is noticeable that major statuary continued to be of bronze right into the Byzantine period[100], pointing to a sort of 'prestige' still held by bronze (e.g. a bronze medal and not a brass one).

- **High tin-bronze**

Within an archaeometallurgical context *high-tin bronze* is the term generally applied to *archaeologica* made from bronze having between 15% and 30% of tin present, often with about 5% lead added. Once above 10% of tin the physical properties change quickly with increasing tin content. The colour changes less rapidly and does not become particularly silver coloured until well over 20%. The shift in colour towards silvery is due to the increasing presence of the hard, brittle and silver-white coloured δ-phase. When polished the metal gets a bright reflecting surface, which is normally corrosion resistant but benefits from occasional repolishing. The colour is only truly silver-white when the intermetallic compound is present without any α-phase, which adds a shade of copper colour to the alloy. If a little lead is added to a high tin-bronze the metal is very suitable for casting items that need a durable, silver-like polished finish. The best-known artefact classes for which high-tin bronzes (20-30% Sn) are used are jewellery, bells and mirrors. A typical Roman mirror composition varies between 18 and 30% of tin and 5 to 10% of lead[101]. The term *speculum metal* is often used to describe this material. More recently this term is specifically used for hyper-eutectoid alloys with *ca*. 30-40% tin, that have next to the δ-phase also a ε-intermetallic compound present. As a side remark it should be mentioned that the Romans also used low-tin bronze in the manufacture of mirrors. The low-tin bronzes were tinned to produce the colour of the expensive silver metal mirrors.[102]

Roman high tin-bronzes that were left in the as-cast state are almost always *leaded* up to 20% for thin-walled bodies. Sometimes less lead was added for thicker or less complex sections. Any part designed to be cold worked will be lead-free or have a low lead content since bronze with more than a few percent of lead is difficult to work after casting.[103]

[96] Hodges, 1968: 213; Meeks, 1993b: 252-253.
[97] Scott, 1991: 25.
[98] Tylecote, 1976: 166; Craddock, 1977: 102; Healy, 1978: 228; Scott, 1991: 26. Meeks, 1993b: 252-253.
[99] Pigott, 1989b: 457; Ponting & Segal, 1998: 113.
[100] Hook & Craddock, 1996: 151-152.
[101] Srinivasan & Glover, 1997: 82; Angelini, de Caro, Grassini, Ingo & Rosalbino, 2003: 545.
[102] Scott, 1991: 26; Meeks, 1993a: 64-65; Meeks, 1993b: 253; Northover, 1998b: 115.
[103] Craddock, 1977: 105; Northover, Brooks, Lister & Lloyd-Morgan, 1991: 722.

If the tin content is between 17-19% it has been found that the alloy is unworkable (cold or hot) since a film of delta forms a brittle phase that coats the grain boundaries with the result that the alloy breaks upon working. However above 19% of tin the bronze can be hot-worked. In order to shape these bronzes, containing 22-24% of tin, they must be forged at a temperature above 586℃ (more like 650-750℃), foll owed by quenching to prevent decomposition. At that temperature the bronze takes a single beta-phase (β-phase) and becomes soft and malleable. The result of the quenching is the presence of martensitic needles. This bronze is still hard but rather tough than brittle in comparison to a slowly cooled bronze of the same composition. The principal reason for the use of this alloy was its colour that resembled gold and because of their resonant tone, hence they are especially utilized for bowls and gongs. Beta-bronzes were first made in SE-Asia (e.g. S-India, Thailand, Korea, China, the Philippines, etc.) from the end of the 2nd c BC onwards and they slowly spread to the Near East. When brass became more widely known, the use of high-tin bronze diminished (pointing towards the importance of the golden colour). Forged high tin-bronzes have not been encountered in the Roman world. The later Islamic alloy 'white bronze' or *safidruy* is an example of a high-tin bronze.[104]

5.3.2. Sample description

• **This study**

Of the 97 samples analysed here 48 are of tin-bronze, some of which are leaded. If only the samples of ed-Dur are considered (minus the ones originating from Khor Rori) then 43 fall within the tin-bronze category. Samples described as 'fragment' are pieces that could not be designated to a certain object.

Table 9 gives the excavation co-ordinates, the excavation team and the description of each sample. The ten registration numbers indicated by a grey square are samples that were too severely corroded to produce reliable data, i.e. the tin levels are too high due to selective corrosion of the copper. However the fact that tin was detected proves that the metal originally used, was a tin-bronze. These samples cannot be evaluated in more detail.

Reg. nr.	Area	UF	Sq	Loc	Team	Description
AD 031	AD	2348	-	-	British	Bracelet
AV 005	AV	4269	I 1-2	G 5156	Belgian	Ram's head *patera*
AV 007	AV	4269	I 1-2	E 5157	Belgian	Fragment sieve
AV 016	AV	4271	I 1-2	G 5158	Belgian	Bracelet/anklet
AV 055	AV	5502	II 1-2	G 5156	Belgian	Fragment vessel
AV 056	AV	5502	II 1-2	G 5156	Belgian	Fragment vessel
AV 104	AV	5503	II 1-2	G 5156	Belgian	Horse spout
AV 115	AV	5503	II 1-2	G 5156	Belgian	Fragment ladle
AW 021-1	AW	-	-	-	British	Decorative element with snake head
AW 038	AW	4541	XVI 5-6	G 5437	British	Disc, possibly mirror
BQ 007	BQ	5961	I 1	G 6266	Belgian	Fragment vessel
BQ 016	BQ	5921	I 3	-	Belgian	'Mirror'
BQ 070	BQ	5970	A 3	-	Belgian	Bracelet
BR 026	BR	6026	III 2	-	Belgian	Bracelet
BS 064	BS	-	V 3	-	Belgian	Horse bit?
BS 092	BS	6563	IV 4	-	Belgian	Fragment handle vessel?
BS 154	BS	-	-	-	Belgian	Shaped fragment
C 079	C	103	C V	418	Danish	Fragment handle?
K 005	K	382	VI 8	G 922	British	Horse head at the of a ladle

[104] Pigott, 1989b: 457; Scott, 1991: 26; Meeks, 1993a: 65; Meeks, 1993b: 253; Craddock, La Niece & Hook, 1998: 77.

Reg. nr.	Area	UF	Sq	Loc	Team	Description
K 070	K	2187	-	G 3669	British	Fragment vessel
K 149	K	2182	-	G 3666	British	Fragment vessel
K 153	K	2164	X-XI 35-36	-	British	Fragment vessel
K 203	K	2153	-	G 3651	British	Handle vessel
KR 007	-	-	-	-	-	Fragment from *Khor Rori*
KR 008	-	-	-	-	-	Fragment from *Khor Rori*
KR 009	-	-	-	-	-	Fragment from *Khor Rori*
KR 010	-	-	-	-	-	Fragment from *Khor Rori*
KR 011	-	-	-	-	-	Fragment from *Khor Rori*
M 007	M	632	IV 4	R 1114	Belgian	Pedestal statuette
M 038	M	2352	V 3	3812	Belgian	Mirror?
N 118	N	2420	IV 6	G 3840	Belgian	Bead (found together with 'torque')
N 121	N	2420	IV 6	G 3840	Belgian	Anklet (ring)
N 122	N				Belgian	Anklet (ring)
S 0003	-	-	-	-	Belgian	Flat fragment
S 0020	-	-	-	-	-	Female head attachment
S 0021	-	-	-	-	-	Fragment ladle
S 0023	-	-	-	-	-	Mirror
sAV 412 A	AV	4273	II 1	G 5156	Belgian	Iron with tin-bronze sheeting
sBK 1238 A	BK	-	-	-	Belgian	Flat fragment
sBQ 1058 A	BQ	-	-	-	Belgian	Flat fragment
sBQ 1058 B	BQ	-	-	-	Belgian	Shaped fragment, square section
sBQ 1173 A	BQ	-	-	-	Belgian	Shaped fragment, square section
sBR 1041 C	BR	6065	I 3	-	Belgian	Flat fragment
sBR 1157 A	BR	-	-	-	Belgian	Shapeless fragment (solidified 'blob'?)
sBS 1276	BS	6575	IV 2	-	Belgian	Flat fragment
sFO 1308 A	FO	Fort	-	-	Belgian	Shapeless fragment (solidified 'blob'?)
Z 012	Z	1727	Z V	3305	Danish	Large needle
Z 146	Z	-	-	-	Danish	Bent fragment

Table 9: Co-ordinates of bronze objects and samples. The samples in a grey square were corroded.

• **Previous analyses Mleiha**

Three objects from Mleiha were analysed by M. Drieux and C. Degrigny: two anthropomorphic figurines and a 'bar' of undefined function. The first figurine came from a funerary deposit probably dating to PIR B and the second was a surface find of two broken pieces. The 'bar' came from a PIR C context. The objects were of a copper-tin-lead ternary alloy, with the 'bar' object having more lead than tin present. The analytical results of the two fragments of the second figurine turned out to be of different composition: the upper part contained more iron and less lead than the lower part. Different explanations were offered for this phenomenon:

- The two parts are not from the same object.
- Upon cleaning the surface, part of the preserved lead was removed.
- Because of the solidification process it is possible that the amount of lead is higher in the lower part of the casting due to the effect of gravity.

No exact analytical data were published, nor the general ratios of the copper, tin and lead present.[105] The results are to be summarized as tin-bronzes with a 'high' lead content. It is not clear if the 'ingot' described in A. Ploquin, S. Orzechowski and B. Briand[106] is the same object as the 'bar' mentioned above, since the context dates differ (i.e. PIR C for the 'bar' and PIR A for the 'ingot').

[105] Mouton, 1992: 201-202.
[106] Ploquin, Orzechowski & Briand, 1999: 176.

• **Previous analyses by L. Weeks** [107]

Of the 33 samples analysed by L. Weeks *ca*. 60% (~ 20 samples) are tin-bronzes. Of this *ca*. 30% (~ 9 samples) are labelled as *leaded*. The Iron Age witnesses the first widespread use of tin-bronzes in SE-Arabia, although they were in use much earlier (e.g. Tell Abraq)[108]. Very high lead levels of between 12 and 21% are found in four objects: two flat fragments, a chisel/nail and a fragment tentatively designated as a horse bit. The lead levels in the ed-Dur materials are very high if compared to material from the previous periods in SE-Arabia. Although small numbers of leaded objects are also known from Iron Age context.

L. Weeks states that there is a positive correlation between the tin and lead concentrations, which might indicate that some lead was introduced into the copper along with the tin. This correlation is only clear for objects that are not intentionally leaded, i.e. those with less than *ca*. 2% of lead. A correlation between tin and silver is also seen, and it seems that the alloying practices used to create the ed-Dur tin-bronzes may have introduced not only lead but also small amounts of silver. A possible explanation for that is that the lead was produced from argentiferous galena ores and small amounts of silver remained in the lead.

5.3.3. Microstructural observations – optical microscope

Thirty-three samples were looked at under the optical metallographic microscope. First in the polished state, after which they were etched wit $FeCl_3$ to reveal the microstructure in more detail. The results of the observations are summarized in table 10.

Reg. nr.	As-cast	Annealed	Twin-lines	Strain lines	Extra
AD 031		x	x	x	Annealing twins (some slightly deformed). Strain lines. Grains *ca.* 40 µm or smaller. Inclusions elongated in line of working. Some small α+δ-eutectoid islands.
AV 007		x	x	?	Straight annealing twins (possibly some strain lines). Grains < 50 µm.
AV 115		x	x	?	Few annealing twins and strain lines. Small grains (< 50 µm). Inter- and intra-granular corrosion. Some α+δ-eutectoid islands.
AW 021-1	x	x	x		Dendritic structure and coring. Intermetallic phase, α+δ-eutectoid around the copper-rich grains. Many large lead inclusions.
BQ 016		x	x	x	Deformed annealing twins and strain lines. Grains *ca.* 50 µm or smaller. Some small α+δ- eutectoid islands.
BQ 070		x	x		Straight annealing twins. Large slightly elongated lead inclusions. Grain size: < 40 µm.
BR 026		x	x	x	Deformed annealing twins and grains with strain lines near surface. Grain size < 50 µm. Some small α+δ- eutectoid islands (?).
BR 104	x	x			Equi-axed grains (< 50 µm) with remains of coring. Some α+δ-eutectoid islands. Areas with large lead inclusions, concentrated near outer surface. Inter-granular corrosion.
BS 064	x	?			As-cast with coring. Possible annealing phase. Small α+δ-eutectoid islands, lead inclusions and shrinkage pores.
BS 092		x	x		*Corroded. Remains of straight annealing twins in corroded grains.*
BS 154	x	?			As-cast with coring and possibly an annealing phase. Large lead inclusions.
K 005		x	x	x	Straight (?) annealing twins and strain lines. Grain size 80 µm or larger.
K 149		x	x		Straight annealing twins. Large grains (> 50 µm) and inter-granular corrosion
K 153		x	x		Straight annealing twins and strain lines. Grain size 50 µm or larger.
KR 007	x	x	x	x	Remains of coring. α+δ-eutectoid and lead inclusions. Small grains (< 30 µm) with annealing twins and strain lines.
KR 008	x				Fine dendrites, as-cast with coring. Many small lead inclusions and shrinkage pores between copper-rich dendrites.
KR 009	x	?			As-cast with coring and possibly an annealing phase. Lead inclusions.
KR 011	x				Remains of dendritic structure and coring. Intermetallic phase, α+δ-eutectoid around the copper-rich grains. Many lead inclusions.

[107] Weeks, 2004a: 248.
[108] Weeks, 1997: 25; Weeks, 2004b: 3.

Reg. nr.	As-cast	Annealed	Twin-lines	Strain lines	Extra
M 038		x	x	?	*Corroded. Remains of larger grains with straight annealing twins, next to much smaller and distorted grains*
N 121		x	x	x	Straight annealing twins and strain lines. Grains between 30 & 50 µml. Possibly some α+δ-eutectoid islands. Elongated lead inclusions. Inter-granular corrosion.
N 122	x	x	x	?	Remains of coring. Some small lead inclusions. Small grains (< 30 µm) with annealing twins and some with strain lines.
S 0023	x				Intermetallic phase, α+δ-eutectoid throughout the section.
sBK 1238 A		x	x		Straight annealing twins of *ca.* 50 µm. Inter-granular corrosion.
sBQ 1058 A		x	x	x	Straight annealing twins. Strain lines. Large variety in grain size.
sBQ 1058 B		x	x	x	Annealing twins (some seem slightly deformed). Strain lines. α+δ-eutectoid islands. Large lead inclusions.
sBQ 1173 A		x	x	?	Straight annealing twins and possibly strain lines(?). Grain size *ca.* 50 µm
sBR 1041 C	x				Intermetallic phase, α+δ-eutectoid throughout the section.
sBR 1157 A	x	?			As-cast with coring and possibly an annealing phase. Some strain lines?? Small α+δ- eutectoid islands. Large lead inclusions.
sBS 1276	x	?			As-cast with coring and possibly an annealing phase. Some small α+δ-eutectoid islands
sFO 1308 A	x				Fine dendrites, as-cast with coring. Small α+δ- eutectoid islands, small lead inclusions and shrinkage pores between copper-rich dendrites.
sM 1250 B		x	x	x	Remains of coring. Equi-axed grains (between 50 & 100 µm large) with strain lines. Few annealing twins. Some α+δ-eutectoid islands.
Z 012		x	x	?	Few annealing twins in small grains (< 30 µm). Inter-granular corrosion.
Z 146	x	?			As-cast with coring and possibly an annealing phase. Small α+δ-eutectoid islands. Large lead inclusions.

Table 10: Microstructural observations of bronze objects and samples.

S 0023 and sBR 1041 C (Fig. 13-4) have an identical microstructure, completely existing out of the intermetallic α+δ-eutectoid. The grey matrix is the δ-phase, whereas the brownish islands are the α-phase. The larger dark rounded spots are lead inclusions. S 0023 is a sample from an object identified as a mirror. The microstructure seen here is in complete accordance with those seen in Roman or Chinese high tin-bronze mirrors. The flat fragment sBR 1041 C must originate from a similar object, i.e. a mirror.[109] The tin content of this alloy approaches the eutectoid composition and the microstructure took on a lath-like appearance. This probably is due to chill casting conditions.[110] A mirror with this microstructure was directly cast to its shape since this metal is brittle and cannot be worked to shape so it can be labelled as-cast. High tin-bronze mirrors (20-25% Sn and 5% Pb) were first produced in China before 200 BC but the alloy rapidly became established over the Old World and was commonly used by the Romans throughout the Imperial period[111].

[109] Meeks, 1993a: 96 & Scott, 1990: 28.
[110] Meeks, 1993b: 264.
[111] Craddock, 1979: 76.

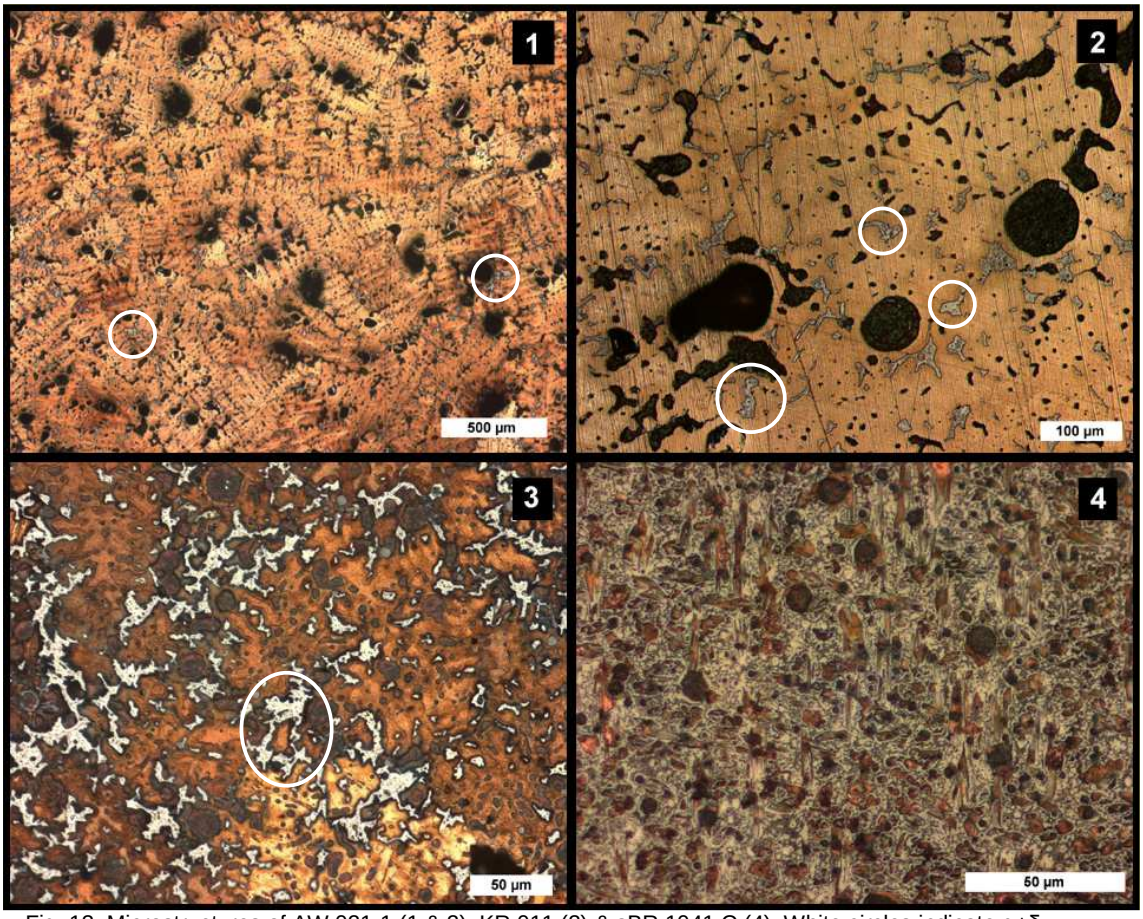


Fig. 13: Microstructures of AW 021-1 (1 & 2), KR 011 (3) & sBR 1041 C (4). White circles indicate α+δ-eutectoid islands.

When a cored bronze is annealed the dendritic structure gradually disappears as the copper-rich dendrites absorb tin from the infilling and a completely homogeneous structure of polyhedral grains is eventually formed. At an intermediary point, however, in which this change has only been partly completed, one may see shadows of the original dendrites. The new grain structure is superimposed upon this. If, on the other hand a cored bronze is cold hammered or drawn and left in this state, the dendrites will be seen as distorted. This is not attested in any of the samples looked at here. Simple annealing of such a bronze will result in the production of a fresh grain structure, finer than that resulting simply from the annealing of a cast and un-worked bronze. It is generally true that the more a bronze has been cold worked and annealed, the finer will be the grain structure. Additionally, twinned grains will appear. These are pairs of identical grains whose lattice structures are mirror images of each other formed each side of a common plane.[112]

[112] Hodges, 1968: 214; Scott, 1991: 25.

Both samples AW 021-1 (decorative element) and KR 011 have an as-cast structure (Fig. 13-1 to 3) and show coring. The α+δ-eutectoid phase (bright grey) is concentrated around the more copper-rich grains. Many large lead inclusions are observed together with shrinkage pores. AW 021-1 was probably cooled slower or went through a short annealing phase after casting, so the α+δ-eutectoid is more finely dispersed and the copper-rich matrix shows larger grain size.

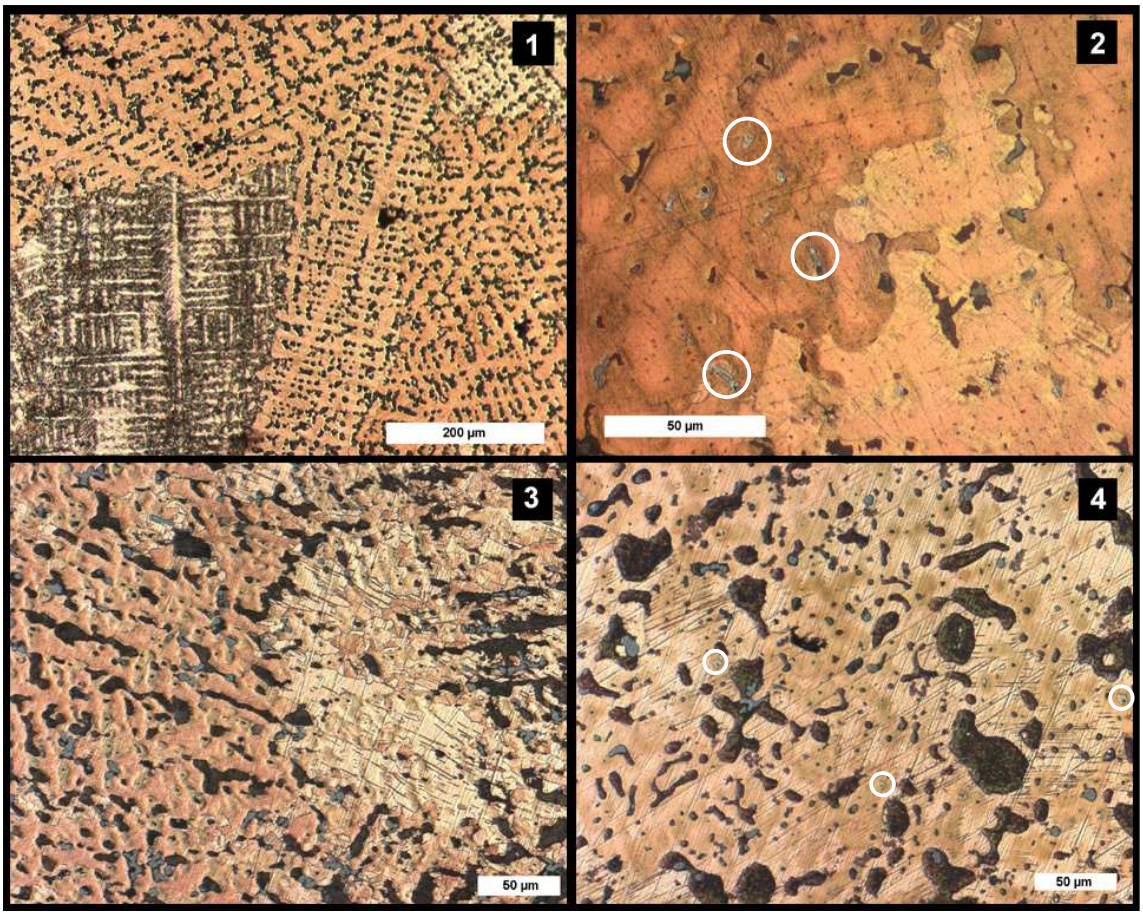


Fig. 14: Microstructures of sFO 1308 A (1 & 2), KR 008 (3) & sBR 1157 A (4). White circles indicate α+δ-eutectoid islands.

Both sFO 1308 A and KR 008 (Fig. 14-1 & 2, and 3) have an as-cast structure with coring. The dendrites in sFO 1308 A are nicely developed (long and fine) within the large grains. The black infill between the dendritic arms consists out of some lead, shrinkage pores, gas vacuoles (?) and α+δ-eutectoid. These fine dendrites are indicative for a fast cooling. The structure of KR 008 is comparable but without the α+δ-eutectoid and much more lead inclusions. The dendrites are also less nicely formed. BS 064 has the same microstructure.

The structure of sBR 1157 A (Fig. 14-4) is very similar to those of BS 154 and Z 146. The large black inclusions are lead globules. They are coloured black (normally grey) because the polishing did not produce a flat surface due to the softness of the lead. The absence of a flat surface prevents the reflection of the incoming light, creating black areas. The remains of coring can be observed although true dendrites are absent, suggesting an annealing phase after the casting. Sample BS 154 does not have any α+δ-eutectoid islands however. Fig. 14-4 might exhibit some strain lines in some of the grains, indicating some cold working. Experimental work by B.E.P. Staniaszek and J.P. Northover report that the use of a clay mould for casting leaded bronze can result large pools of lead. This is because of the fact

that some time (7 minutes, which is a slow cooling) passed before the freezing point of the lead was reached. This gave time for the rejection of lead from the bronze matrix and its accumulation in large pools.[113]

BS 1276 (Fig. 15-1) has a cored as-cast structure, comparative with KR 009. KR 009 has much better defined lead inclusions and pores in its structure.

KR 007 (Fig. 15-2) has a remnant coring structure, but well developed small grains can be observed indicating an annealing phase. The grains have annealing twins (some of which seem deformed) and strain lines. A limited amount of α+δ-eutectoid islands are seen in the section. The lead inclusions are somewhat elongated. N 122 (Fig. 15-3) is very similar with the difference that no α+δ-eutectoid islands were noticed.

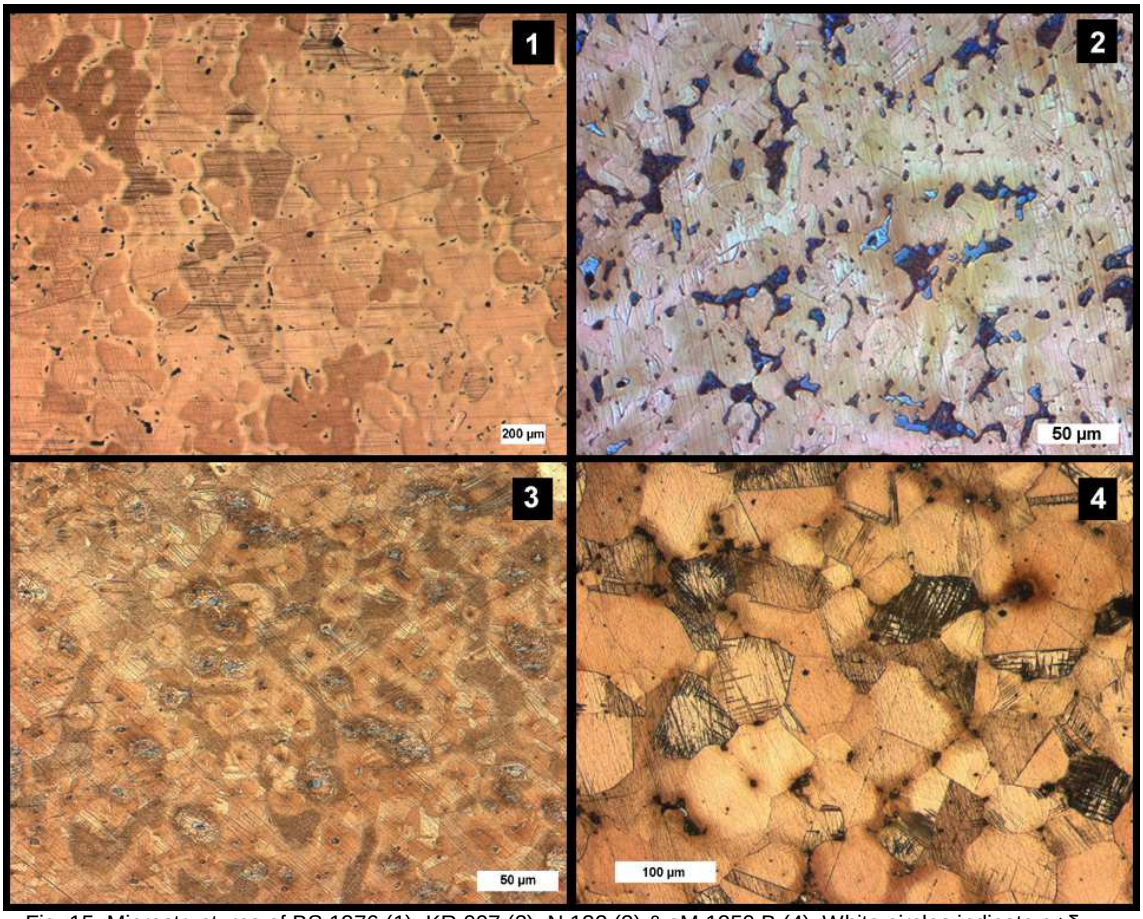


Fig. 15: Microstructures of BS 1276 (1), KR 007 (2), N 122 (3) & sM 1250 B (4). White circles indicate α+δ-eutectoid islands.

The vessel fragment K 153 (Fig. 16-1) and sieve fragment AV 007 have completely recrystallized angular grains with annealing twins. The grains seen in AV 007 are somewhat smaller though. The objects were finished with a final annealing phase and all stress was relieved from the metal. K 005 (Fig. 16-2) is a section through a decorative horse head element, probably the ending of a ladle. Annealing twins are less numerous and not deformed, and strain lines are still visible as the result of an incomplete annealing phase.

[113] Staniaszek & Northover, 1983: 263-264.

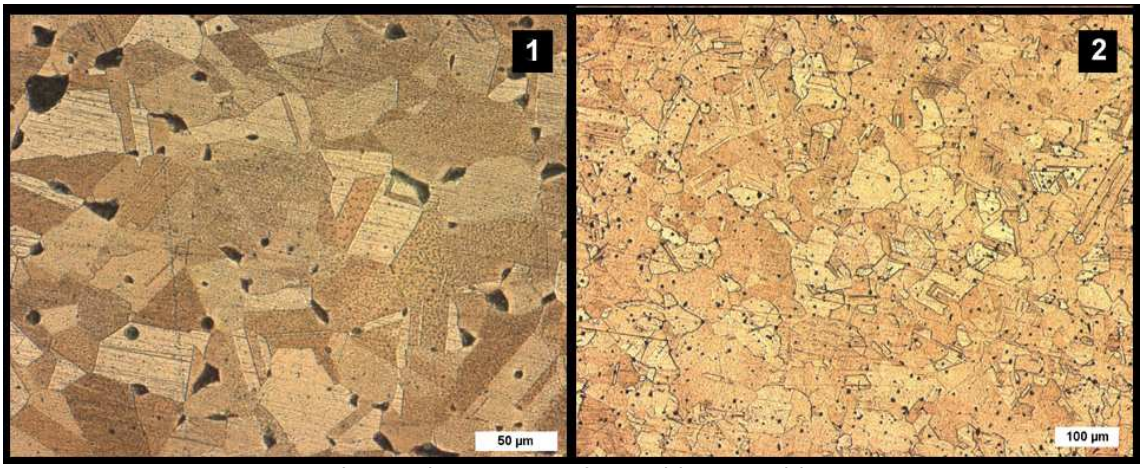


Fig. 16: Microstructures of K 153 (1) & K 005 (2).

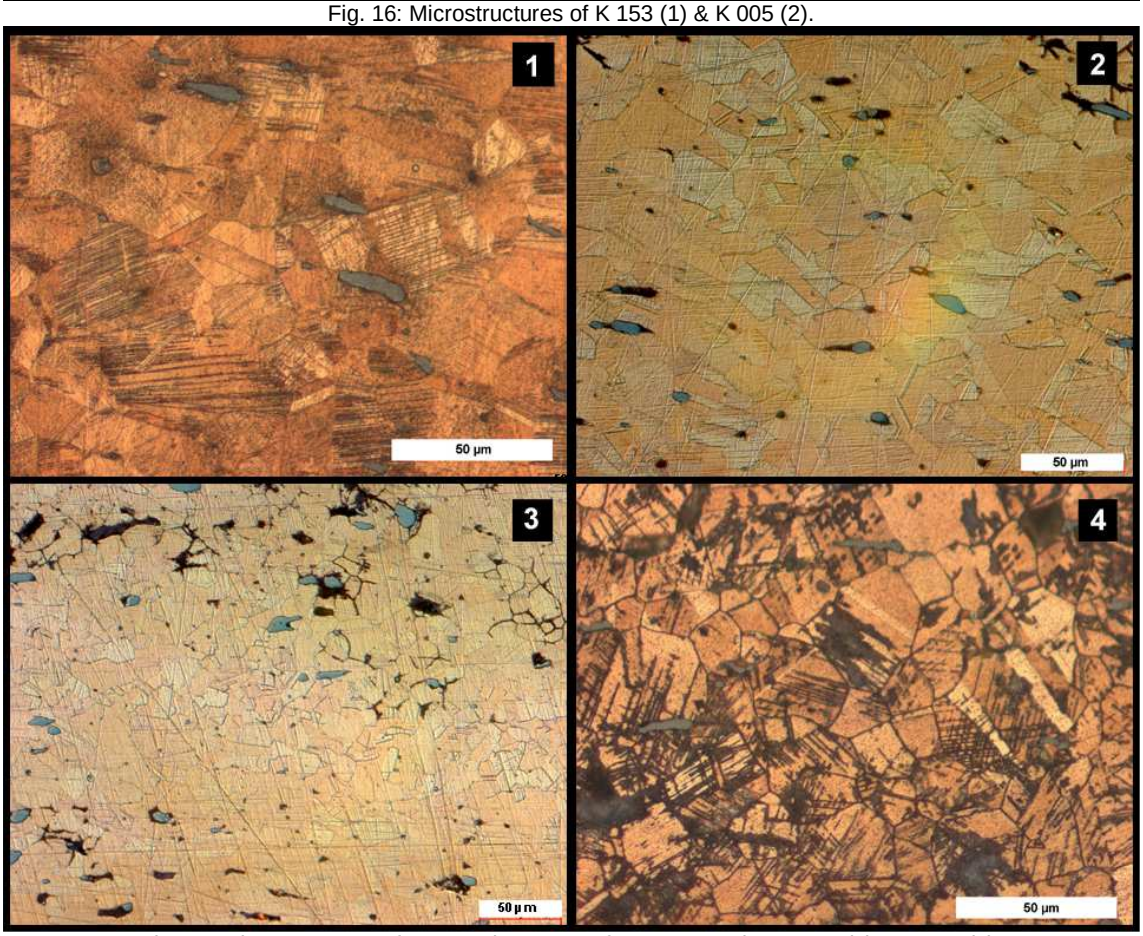


Fig. 17: Microstructures of AD 031 (1: near surface & 2: core), BQ 070 (3) & N 121 (4).

AD 031 is a section through a bracelet (Fig. 17-1 & 2). Annealing twins are present and some are slightly deformed. Near the surface strain lines are seen, something not attested in the centre of the sample. The lead inclusions are elongated. BQ 070 (Fig. 17-3) also is a section through a bracelet and has a similar microstructure, without the strain lines that is. This bracelet seems to have been annealed after it had been given its final shape.

Several samples has a structure similar to the anklet N 121 (Fig. 17-4). They include AV 115 (the handle of a ladle), sBK 1238 A (flat fragment) and Z 012 (a needle). The grain size varies but they all have a limited amount of annealing twins. Inter-granular corrosion is seen

together with corrosion along the strain lines. Some also exhibit small α+δ-eutectoid islands. The vessel fragment K 149 is comparable, but the grains are larger and strain lines are absent. All these objects were finished by annealing and do not seem to have received any additional working afterwards.

BQ 016 (Fig. 18-1) was tentatively described as a mirror, but the microstructure is completely different from that seen in the two samples of mirrors presented above (S 0023 & sBR 1041 C). This sample has many strain lines and deformed annealing twins. The final shaping happened after the final annealing phase. The different microstructure does not automatically imply that this object was not a mirror, since the cheaper version of the Roman mirrors had a tinned surface and the core was of an alloy poorer in tin. But in the light of the presence of high tin-bronze mirrors at ed-Dur this might be unlikely. In any case no evidence of a tinned surface was not found, but it has to be admitted that these tinned surface rarely survive the effect of corrosion. The large flat fragment BQ 016 can equally likely be a piece of a vessel or of some sort of plating. The shaped fragment sBQ 1058 B (not depicted) has a very similar microstructure.

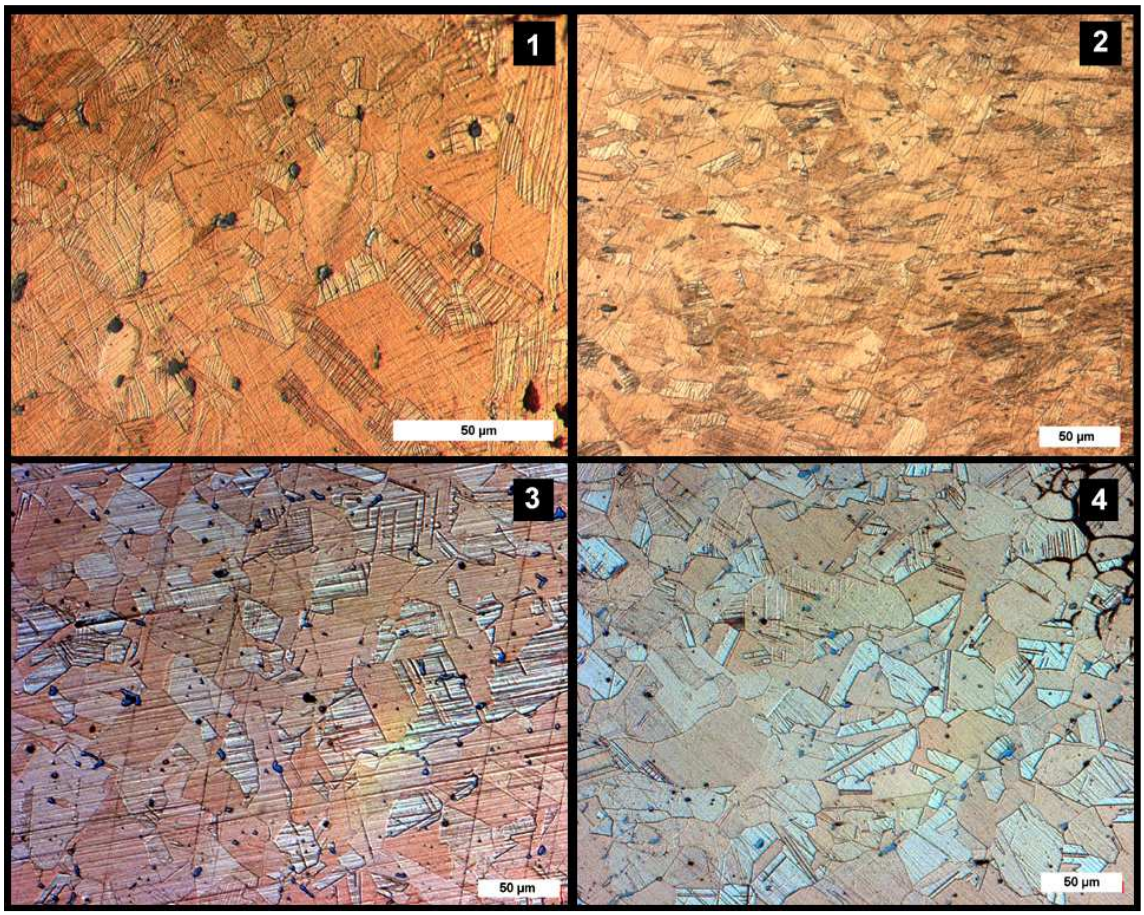


Fig. 18: Microstructures of BQ 016 (1), BR 026 (2), sBQ 1173 A (3) & sBQ 1058 A (4).

The microstructure of BR 026 (Fig. 18-2, near surface), a bracelet, shows heavily deformed grains and annealing twins (near the surface), strain lines and elongated inclusions are all indictors of a reduction in thickness and working without a final annealing phase.

BQ 1173 A (Fig. 18-3) and sBQ 1058 A (Fig. 18-4) both have annealing twins and angular grains of similar size. The lines in Fig. 18-3 are no part of the microstructure but due to bad polishing. sBQ 1058 A exhibits some strain lines however.

5.3.4. Microstructure & chemical composition – SEM-EDX results

• **Tin**

In the introduction of the part on the tin-bronzes three large groups were defined, being: *low* or *natural* tin-bronze (till 5 wt% of Sn), Medium tin-bronze (less than *ca.* 15 wt% of Sn) and high tin-bronze (more than 15 wt% of Sn). Is this division sustained by the EDX-results obtained here?

Fig. 19 gives a frequency diagram of all the copper-base alloys that contain tin. Two clear breaks can be seen. The first is between 4 and 6 wt% of tin and that group falls nicely within the definition of a low tin-bronze. These are bronzes that originated from recycling or where the tin entered the alloy via the ore source. The second break is after 18 wt%. There is no clear distinction between what was defined as medium and high tin-bronzes. Within the medium tin-bronzes there are two small peaks, one at 8 and another at 12 wt%. In broad lines the division suggested in the introduction can thus be found in the samples from ed-Dur.

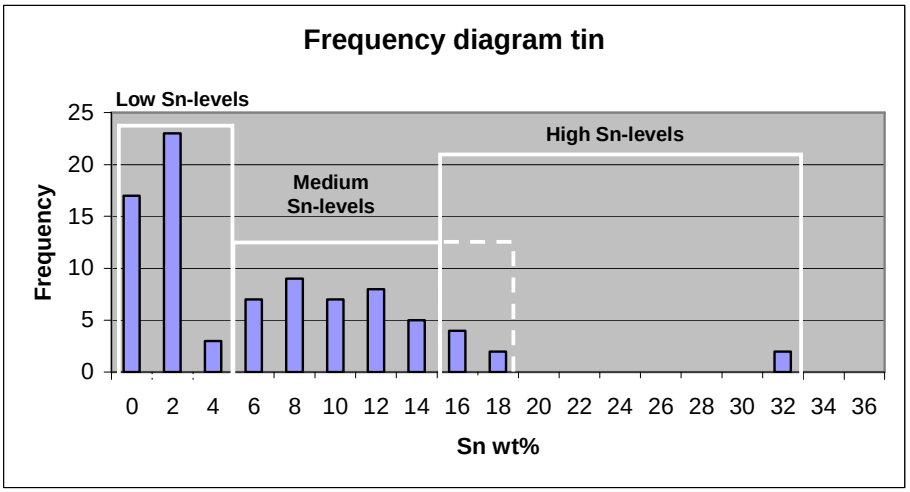


Fig. 19: Frequency diagram of tin wt% in all copper-base alloys.

The mean, average, minimum and maximum tin levels for the three different bronze classes are summarized in Table 11.

Alloy	Mean Sn wt%	Average Sn wt%	Min. Sn wt%	Max. Sn wt%	n
Low tin-bronze	4,6	4,4	3,6	4,9	4
Medium tin-bronzes	8,8	9,3	5,3	14,1	27
High tin-bronzes	16,6	20,3	15,1	31,9	7

Table 11: Mean, average, minimum & maximum Sn levels for different bronze classes.

Actually only two true high tin-bronze samples were encountered (sBR 1041 C & S 0023). The other five samples in this class might as well be medium tin-bronze if the semi-quantitative results of EDX are considered. None of these actually contains more than 17 wt% of tin. All are thus still workable and not too the brittle.

A major peak in the Roman bronze artefact of N-Britain is seen around 9% of Sn and a minor peak in low tin-bronzes around 2%. This type of bronze (till 5% of Sn) would have a similar colour and mechanical properties to pure copper (i.e. pinkish colour and malleable). It was widely used for the production of sheet and wire objects in N-Britain.[114]

[114] Dungworth, 1997b: 904-905.

Reg. nr.	Cu	Zn	Ag	Sn	Pb	Extra
High tin-bronze & leaded high tin-bronze						
sBR 1041 C	61,17	0,49	-	31,89	6,45	
S 0023	65,99	-	-	30,55	3,46	Sn level by AAS: 23,1 wt%
KR 011	69,91	-	-	16,76	13,33	
sBK 1238 A	81,31	-	-	16,63	2,06	
Z 012	80,86	-	-	15,73	3,42	
S 0020	59,58	-	-	15,28	25,15	Drilling – Pb level by ICP-MS: 22,4 wt%
AW 021-1	78,30	-	-	15,12	6,58	Sn level by AAS: 13 wt%
Medium tin-bronze & leaded medium tin-bronze						
AD 031	85,05	-	-	12,58	2,36	
AV 005	56,83	0,77	-	7,61	34,79	Drilling – Pb according to ICP-MS: 17,3 wt%
AV 007	84,56	-	-	12,53	2,91	
AV 016	86,43	-	-	10,55	3,02	
AV 115	91,08	-	-	6,16	2,76	
BQ 016	91,58	-	-	6,19	2,23	Cu-S-Fe-inclusions from slag? Sn level by AAS: 5,56 wt%
BQ 070	89,65	-	-	7,12	3,23	
BR 026	85,27	-	-	11,94	2,79	
BS 064	83,86	-	-	10,39	5,75	
C 079	80,66	1,08	-	7,42	10,84	Drilling – Pb according to ICP-MS: 12,2 wt%
K 005	83,42	-	-	13,89	2,69	
K 149	83,48	-	-	14,11	2,41	
K 153	83,51	-	-	13,17	3,32	Sn level by AAS: 12,5 wt%
K 203	69,97	-	-	10,70	19,33	Drilling; 0,67 wt% Ni – Pb according to ICP-MS: 12,2 wt%
KR 007	77,19	-	-	11,02	11,79	
KR 009	88,61	-	-	8,56	2,83	0,79 wt% Fe; 0,79 wt% Ni
M 007	55,93	0,72	-	6,16	37,18	Drilling – Pb according to ICP-MS: 21,7 wt%
N 118	79,87	-	-	6,82	13,31	
N 121	85,57	-	-	11,63	2,81	
N 122	85,42	-	-	11,61	2,97	
S 0021	90,60	-	-	6,77	2,64	
sBQ 1058 A	91,73	-	-	5,82	2,45	
sBQ 1058 B	88,47	-	-	10,00	1,53	
sBQ 1173 A	89,11	-	-	8,26	2,63	
sBS 1276	92,12	-	-	5,25	2,63	
sFO 1308 A	89,61	-	-	8,84	1,55	
Z 146	78,37	-	-	5,54	16,09	Sn level by AAS: 5,85 wt%
Leaded low tin-bronze						
AV 104	58,47	-	-	4,77	36,76	Drilling – Pb according to ICP-MS: 22,2 wt%
BS 154	76,67	-	-	3,58	19,74	
KR 008	79,34	-	-	4,45	16,21	
sBR 1157 A	80,11	-	-	4,87	15,02	

Table 12: Chemical compositional data of bronze objects and samples in wt%. Grey squares in the Zn-column indicate that the zinc values were removed before quantification. Parallel analyses by ICP-MS and AAS are indicated in the column 'Extra'.

All low tin-bronzes have a similar amount of tin present and are heavily leaded, including the sample from Khor Rori. This high lead level would make these alloys impossible to work without breaking. This statement is sustained by the microstructures of these samples, since they all exhibit an as-cast structure.

The leaded medium and high tin-bronzes show the same feature and have an as-cast structure. Only one exception is to be noted (KR 007) where a limited amount of annealing

twins are seen. The grains of this sample are small and could indicate repeated annealing phases.

Four more medium tin-bronzes have an as-cast structure. These are sFO 1308 (a shapeless blob) and KR 008 with an tin contents of *ca.* 9 wt% and two flat fragments sBQ 1058 A and sBS 1276 (with 5-6 wt% of Sn). The latter two would have been suited for working since they were not leaded and could be parts of vessels.

All other bronzes have annealing twins and this shows that they went through at least one annealing phase after they were worked. This in turn shows that the metalworkers were well aware of the brittleness that is introduced by working bronze. Moreover α+δ-eutectoid islands are not seen in samples where they could be expected. The removal of the eutectoid is only possible when the alloys are carefully annealed.

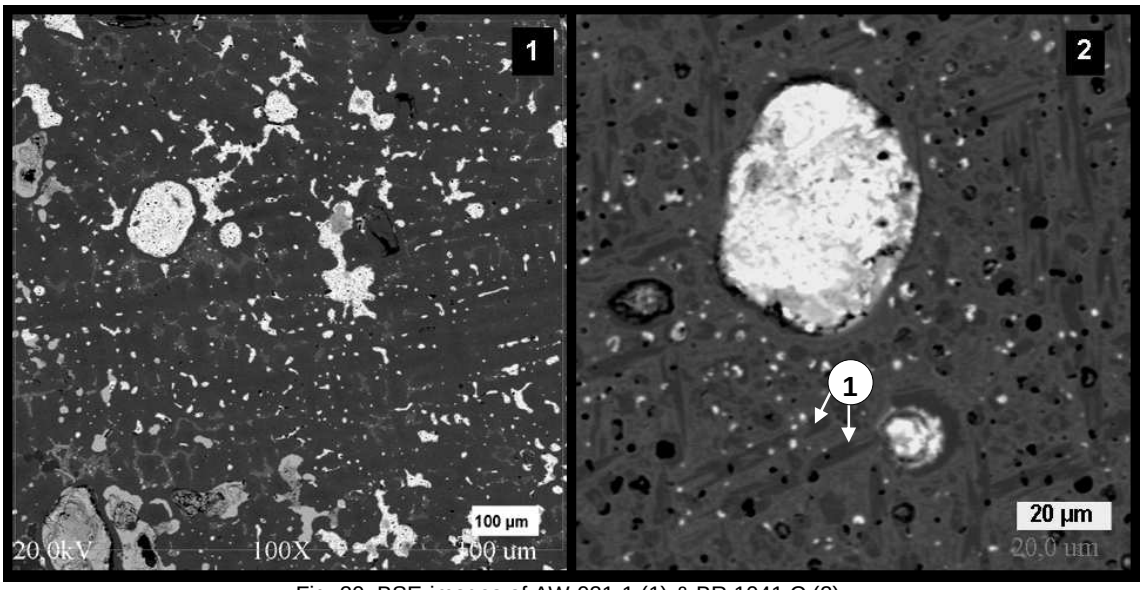


Fig. 20: BSE-images of AW 021-1 (1) & BR 1041 C (2).

On Fig. 20-1 the dark grey matrix is copper-rich, the light grey network in-between is the α+δ-eutectoid and the large white inclusions are lead globules. The eutectoid is found at the boundaries of dendrite-like structures.

Fig. 20-2 is a close-up of the mirror sample BR 1041 C that has a complete α+δ-eutectoid microstructure with large and small lead inclusions. The darker laths (1) are surrounded by a lighter grey matrix. The tin content of the α-laths varies between 18 and 20 wt% and in the light grey δ-matrix between 32 and 36 wt%. The fixed composition of the δ-compound, $Cu_{31}Sn_8$, is 32,6% of tin[115]. Considering the semi-quantitative nature of the EDX results this is in relative good correspondence. Moreover the average composition as published for a α+δ-eutectoid phase is around 27% wt%. The average for the two mirror samples, S 0023 and BR 1041 C, respectively are 30,5 and 31,9 wt% of tin. It can be suggested that this is somewhat at the high side and that this is inherent to the EDX. These high values can also be due to the overwhelming presence of the δ-phase that contributes much of the tin detected by EDX. Recent mirrors made in the Kerale region (S-India) come very close to these samples and are actually defined as δ-bronzes.[116] The presence of lead is to be explained by the fact that it improves casting conditions since it reduces the melting point and increases fluidity during casting[117].

[115] Meeks, 1993a: 65.
[116] Srinivasan & Glover, 1997: 81 & 83.
[117] Meeks, 1993b: 264.

Many Roman and Chinese mirrors have a black surface. There has been a lot of debate on the origin of this layer, i.e. was it applied intentionally or is it the result of post-burial corrosion processes. N. Meeks has convincingly argued that the black layer is the result of corrosion processes[118]. A small fragment of S 0023 was cleaned in lemon juice to remove some of the corrosion products present at the surface. Underneath a black layer was found. EDX analyses showed that this was mainly a tin oxide layer (some copper and lead present). Fig. 21-2 shows a BSE-image of the mineralised surface. It is crazed and still shows the *ghost structure* of the eutectoid microstructure. The black spots include leached lead globule porosity, mineralised lead globules, residual copper oxides, etc. The *pseudomorphic ghost structures* at the surface proves that no significant layers were applied to the original bronze surface. The tin oxides present arose solely from the natural corrosion of the δ-intermetallic compound of the surface material. The black surfaces found on high tin-bronze mirrors can be accounted for by natural patination mechanisms[119].

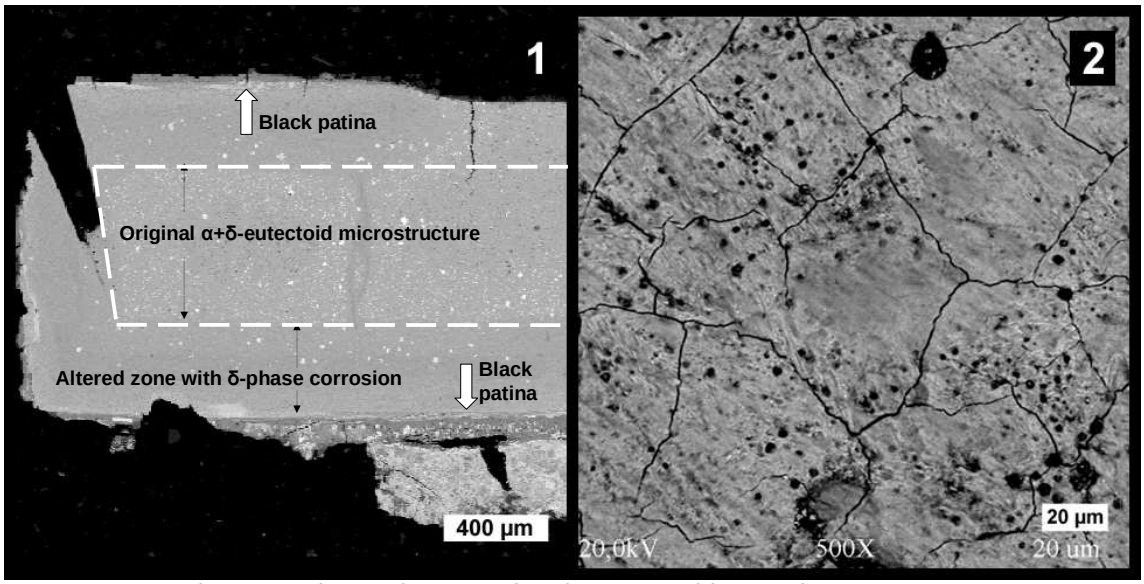


Fig. 21: BSE-image of cross-section of BR 1041 C (1) & BSE-image of the black patina on the surface of S 0023 (2).

When the corrosion of bronze leads to the formation of tin oxide there is a tendency towards *tin enrichment*, since tin oxide is very insoluble and stable and remains at the surface of the object. The copper on the other hand is often leached away. This enrichment is particularly noticeable on high tin-bronzes as the α-phase (copper-rich) of the eutectoid is always corroded. The δ-phase is mineralised and this leads to severe loss of copper and relative tin concentration of about 60-70%.[120] In our analyses the EDX registered *ca.* 75 wt% of tin (additional copper might have been removed by the lemon juice). Underneath there is a zone which is selectively corroded and in the core unaltered metal is seen. The presence of the stable tin oxide results in a protection of the core metal in the centre, leavening it in an uncorroded state.

The upper side of the Fig. 21-1 was probably the polished side of the mirror. Research on Roman and Chinese polished black patinated mirror has shown that this layer tends to be thinner on the polished side of the mirror[121].

[118] Meeks, 1993a & b.
[119] Meeks, 1993a: 82.
[120] Meeks, 1993b: 265.
[121] Meeks, 1993a: 75.

• **Zinc**
Only a few samples had a detectable zinc level. This must surely be the result of recycling of small amount of brass with bronze. It is interesting to notice that the two objects (AV 005, *patera* & M 007, pedestal) both have a similar amount of zinc present. Both objects are very likely of Roman/Mediterranean origin. The only other sample that contained some zinc is C 079.

• **Lead**
The high lead levels in the measurements made on drilling are higher than in reality, since the lead seems to be 'smeared out' due to the drilling. Lead probably acted as a kind of *lubricant* for the drill and the EDX values should be reduced with a certain factor. Still they are heavily leaded objects as shown by some parallel ICP-MS analysis where they range between 12 and 22 wt% (Table 12, column Extra).

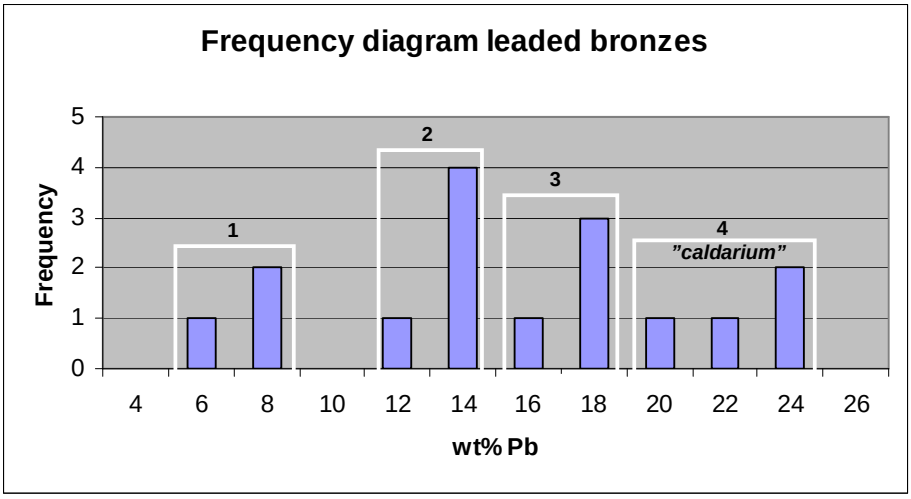


Fig. 22: Frequency diagram of leaded bronzes (including ICP-MS results where possible).

The amount of lead measured ranges from 5,75 wt% and 22,4 wt% (by ICP-MS). Four groups can tentatively be defined. The first group has an average tin level of 6,3 wt%, the second group of 12,6 wt%, the third group of 16,2 wt% and the fourth of 21,5 wt%.

Only the last group can be related to textual evidence, where an alloy is mentioned which contains one part of lead and four parts of copper or bronze, i.e. a 20% leaded alloy. This alloy is called *caldarium* and is described as being very white in colour and useful for castings. The colour of the alloy is an interesting feature, and suggests a primary aesthetic consideration in the choice of alloy.[122] The objects AV 005 (a *patera*), AV 104 (a horse spout), M 007 (a pedestal) and S0020 (a female head appliqué) were made of such an alloy.

[122] Craddock, 1979: 75; Ponting, 1999: 1317.

5.4. Brass

5.4.1. Introduction

Brass is a copper-zinc alloy and although the alloying element zinc (Zn) is widespread in the earth's crust it is very reactive and by consequence never occurs as a native metal. Its most dominant mineral occurrences are as the carbonate *calamine*[123] or *smithsonite* ($ZnCO_3$, zinc spar), the sulphide *sphalerite* (ZnS, blende) and *heminorphite* ($Zn_4[Si_2O_7(OH)_2].H_2O$) once called calamine as well. Zinc ores are common and easily recognised and they are regularly found in association with copper and lead ores, in fact sulphide copper ore almost always contains some zinc. It is however rare for prehistoric artefacts to contain more than traces of zinc, since the zinc is lost due to evaporation during the smelting of the copper rather than being absorbed by it.[124]

Zinc was the last of the common metals to be smelted and its late appearance in history in its metallic form is linked to the extreme volatile nature of the metal. The metal zinc melts at 419°C but boils at 907°C, this is at a much lower t emperature than any other common metal (except for tin and lead). In order to reduce zinc from its ores, it needs to be heated in contact with charcoal at *ca*. 1000°C. Unfortunately this is above the boiling p oint, thus instead of a molten metal descending to form an ingot at the base of the furnace, the zinc vapour would rise and re-oxidise in the upper part of the furnace or be lost in the fumes.[125]

The fusion of zinc with copper produces an alloy, *brass*, with increased strength, hardness and toughness when compared to unalloyed copper. In concentrations of greater than 5% it improves the castability of the metal to an even greater extent than tin[126]. The tensile strength is comparable to that of tin-bronze[127]. Brass with less than 15% of zinc is extremely ductile and malleable at room temperatures. They can be cold worked by many processes and this increases tensile and yield strength, and also hardness. After cold working these brasses can be annealed at temperatures ranging from 370 till 760°C to reduce hardness and render them more malleable and ductile again.[128] When the alloy is composed of 10 to 20% of zinc it has a golden yellow colour, and when polished it can shine like gold[129]. This made it attractive for decorative metalwork such as *fibulae*, rings, trappings, etc. A particular advantage of brass is that *niello* (a mixture of copper and silver sulphides) adheres well to it, in contrast to a substrate of bronze. During the 1st c AD large numbers of trappings were made of brass covered with silver foil and with niello inlaid throughout the silver into the brass beneath.[130] When more than 20% zinc is present the colour becomes more green-yellow[131]. The addition zinc also increases corrosion resistance (especially for sea water), but when the zinc content exceeds about 15 to 20%, copper may corrode by dezincification. Small amounts of tin greatly improve the strength and resistance of brasses to dezincification.[132] For modern industrial purposes brass contains about 40% of zinc when used for casting and 10 to 30% when it has to undergo processes involving working[133].

In antiquity the maximum percentage of zinc that could be alloyed with copper was about 28% (see below), so these *high-zinc brasses* in antiquity actually correspond with a *low-zinc brass* of nowadays. This is an important contradiction in terms that has to be kept in mind when going through archaeometallurgical literature. Most brasses from antiquity would

[123] This old term covered the present minerals heminorphite and smithsonite.
[124] Moorey, 1994: 254; Craddock, 1998b: 1.
[125] Craddock, 1978: 2; Craddock, 1998b: 1; Tylecote, 1962: 53.
[126] Craddock, 1978: 11.
[127] Habashi, 1994: 59.
[128] An., 1972: 7.
[129] Habashi, 1994: 59; Biswas, 1996: 351; Biswas, 2001: 141.
[130] Craddock, 1978: 11.
[131] An., 1972: 7.
[132] An., 1979: 41.
[133] Coghlan, 1975: 36.

correspond to modern alloys termed *red brass*, *gilding metal* and *commercial bronze*[134]. All the brasses discussed here show a single phase at room temperature and are alpha-brasses (α-brass)[135] and contain less than 30% of Zn. The earlier brasses with high zinc content are almost free of tin and the small amounts of lead present (1 or 2%) is very likely to come from the zinc ore used in the process[136]. The presence of about 2% of lead will however slightly lower the alloy's melting point and improve the fluidity, thus making a small amount of lead desirable in alloys used for casting[137].

The term *primary brass* is used when a freshly melted brass is meant that maximally went through one cycle of remelting, e.g. from the ingot to the object. These have a high zinc value, but the actual amount can vary according to the technical skills of the producers. In the Roman period primary brasses would have a zinc level of between 20 up to the maximum amount of 28%. *Secondary brasses* are brasses that went through repeated cycles of remelting and recycling. This progressively drives of the zinc at every melt and averages values of between 10 and 15% can be expected. The spreading of the use of brass is closely connected with the emergence of *gunmetal* (see below) and although this alloy is treated separately some information is inevitably included in this part.

5.4.2. Production techniques

Zinc ores are often found in association with copper and lead ores and the zinc could easily have found its way into the smelt by accident. In reality however the majority of the zinc present in the ores would vaporize, leaving only a trace (up to 1 wt%) in the resulting copper metal. The vaporization can only be prevented if deliberate measures were taken to avoid the loss of zinc by reintroducing the vapour back into the smelt.

The early history of zinc and brass is not very clear. This is caused in part by misleading comments by some ancient authors, some questionable analyses, poor excavation reports, and numerous misconceptions.[138] There are three main processes to produce brass, *co-smelting of ores*, the *cementation process* and *co-smelting of metals*.

- **Co-smelting of ores**

Co-smelting of ores is the simplest and probably often unintentionally used method to create an alloy of volatile and non-volatile metals. In this process a mixed ore was smelted to produce a *natural alloy*, i.e. *natural brass*. Arsenical copper can also be a result of such an unconscious process. In rare instances, such as under extremely reducing conditions, the non-volatile component would be reduced to form the molten metal, while the mineral of the volatile metal would be reduced to a vapour. This vapour would in part rise with the waste gases to re-oxidise and be deposited on the furnace walls and in part leave the furnace. Some of the zinc vapour however would be absorbed by the droplets of the non-volatile metal, in this way directly creating a natural alloy.[139] Natural brass with up to 7 or 8 wt% of zinc could be produced by mixing copper and zinc ores in a furnace[140]. This process can be taken as the unintended production of an alloy, since it is very hard to control. This does not mean that ancient smelters could not have deliberately used zinc-rich ores to produce 'an' alloy with certain properties, but only that they had little or no control on the outcome. This process is of no concern to the brasses examined here.

[134] Ponting, 2002: 557-558.
[135] An., 1972: 8.
[136] Craddock & Eckstein, 2003: 223.
[137] Ponting, 1999: 1316.
[138] Craddock, 1995: 292-293; Thornton & Ehlers, 2003: 3.
[139] Craddock, 1995: 284-285.
[140] Craddock, 1995: 292-293; Thornton & Ehlers, 2003: 3.

• **Cementation**

The second technique, and probably the earliest intentional method of actually producing brass, was *cementation* (also known as the *calamine process*[141]). This is the term used for the process where one metal is heated in the solid state to absorb another element, forming an alloy. In this process copper in the form of small fragments, thin sheets or foil and ideally with as less as possible impurities, was mixed and packed in a sealed crucible with charcoal and zinc oxide. The zinc could be added as finely crushed smithsonite ore or as an oxide powder (*philosopher's wool*). This powder was the re-oxidised zinc vapour that sometimes settled on the cooler parts of the furnace walls and could be collected. The closed crucible, to keep the highly reactive and elusive zinc vapour in close contact with the copper metal, was then heated to a temperature between 900 and 1000°C . This temperature is hot enough for the zinc to vaporise but not so hot that the copper would melt and run to the bottom of the vessel. The zinc vapour in the sealed vessel readily diffuses in the widely dispersed copper and was absorbed into the solid copper to form brass. The temperature had to be carefully regulated as zinc does vaporise below 907°C and pur e copper melts at 1083°C. [142]

As zinc diffuses into copper the melting point of the new alloy falls below 1000°C by the time it contains about 30% zinc. Values of up to 33,3% of zinc are theoretically possible but usually the maximum would have been between 20 and 28%. These ancient high-zinc brasses normally contain a maximum of a few per cent of additions such as tin or lead. The presence of either tin or lead will reduce the uptake of zinc in the alpha phase of copper, by the amount known as the *zinc equivalent*. For lead this is one, for tin two, i.e. copper with 2% of tin will absorb 4% less zinc than it would otherwise have done. Additionally these metals reduce the melting point of the copper. Once the copper is molten the specific surface available for the diffusion of the zinc vapour is much reduced and the process will result in a brass with a lower amount of zinc.[143]

It is clear that whole range of factors including temperature, pressure, atmosphere composition and other alloying elements present influences the exact limit of zinc uptake. The process was often quite efficient but cementation allowed little control over the composition, and it was difficult to stop other undesirable elements from joining the alloy. Brass cementation generally requires closed crucibles. At the end of the process the temperature was raised and the then molten brass was stirred to form a uniform alloy.[144] Th. Rehren however points to the absence of metal prills or slag remains within recovered Roman brass production vessels, strongly indicating that cementation was done as a solid-vapour reaction, with no liquid metal/alloy or slag formation[145]. P.T. Craddock and K. Eckstein contradict this by saying that in antiquity zinc oxide reacted with molten copper to produce brass and that only in Renaissance Europe 'true' cementation (the solid state process) was employed. This 'true' process needed longer operating-times but at temperatures about 100°C lower than previously, the reby raising the potential maximum zinc content from 28% to 33%.[146]

When zinc oxide or carbonate ores (calamine/smithsonite), found as a natural ore, are used the cementation process can be achieved without any extra ore preparation. If zinc sulphide (sphalerite) was used on the other hand an additional refining step was essential. During this step the sphalerite ore was submitted to roasting and sublimation in a special furnace to collect the zinc vapour in the form of zinc oxide. This had the added advantage of separating

[141] Thornton & Ehlers, 2003: 3.
[142] Bayley, 1998: 9; Craddock, 1978: 9; Craddock, 1995: 285; Rehren, 1999a: 253; Craddock & Eckstein, 2003: 223.
[143] Craddock, Burnett & Preston, 1980: 60.
[144] Bayley, 1998: 9; Craddock, 1978: 9; Craddock, 1995: 285; Rehren, 1999a: 253; Rehren, 1999b: 1085; Craddock & Eckstein, 2003: 223.
[145] Rehren, 1999a: 254.
[146] Craddock & Eckstein, 2003: 216.

the zinc from any contaminants such as iron and lead minerals (although some of the lead was inevitably also sublimated), which are often found together with those of zinc.[147]

• **Co-smelting of metals**

The third process is to mix metallic copper with metallic zinc or *co-smelting* both metals. To do this, metallic zinc has to be produced by a process of *distillation* and this clearly separates this technique from the others, where no actual metallic zinc was used. The element of distillation allowed separating the volatile metal as a vapour and condensing it away from contaminants or the danger of oxidisation. Distillation gave a purer product, but the processes were more expensive and needed more sophisticated equipment.[148] Since the cementation process can only produce brass with a zinc level up to 28-30%, artefacts containing higher levels of zinc must have been prepared by mixing the two metals together. The co-smelting of these metals is a process known as *speltering*.[149]

The manufacture of zinc metal by reduction and distillation was a well-guarded Indian secret and put to large commercial practice in the 12th – 13th c AD[150]. At that period the European countries knew about the cementation route for making brass, but the art of making metallic zinc and high-zinc brasses with more than 30% of zinc, was unknown in the West[151]. Even after the discovery of the distillation process to obtain metallic zinc the tradition of cementation persisted[152]. From the 16th c AD onwards, metallic zinc was introduced from the East as an import product[153], but it was only in the early part of the 18th c AD that the knowledge travelled from India, and William Champion of Bristol (England) utilised it to manufacture zinc metal[154].

• **Remelting brass**

A *fresh brass* made by the cementation process could contain between 20 and 30% of zinc, with a conventional maximum around 28%. If cementation brasses were repeatedly recycled then the volatile zinc would be progressively driven off producing low zinc values[155]. Every time a brass is remelted some 10% of the zinc is lost[156], so theoretically remelting a 28% zinc alloy will result in a 25 % alloy. Recycling could also result in new alloys. If a brass was remelted together with scrap tin-bronze, not only the zinc content would be reduced by evaporation, but the alloy would also be diluted by the introduction of other metals. This results in an ill-defined ternary alloy of copper, tin and zinc or quaternary alloy with additional lead, respectively *gunmetal* and *leaded gunmetal* (see below) used for many everyday implements and castings.[157] Probably brass was often at least once remelted since the fresh brass was made into an ingot and to produce a cast object it has to be liquefied again. Here I would like to point the fact that the maximum of zinc present in many Roman objects is around 28% and that many of these objects are cast. The question is if these artefacts were immediately produced (so the prepared alloy went straight from the crucible into the mould), if not the original alloy composition must have been around 31% of zinc.

[147] Bayley, 1998: 10; Thornton & Ehlers, 2003: 3.
[148] Craddock, 1995: 284; Freestone, Craddock, Hegde, Hughes & Paliwal, 1996: 229.
[149] Craddock, 1978: 9.
[150] Freestone, Middleton, Craddock, Gujar & Hook, 1991: 617; Biswas, 1996: 354.
[151] Biswas, 1996: 354.
[152] Biswas, 2001: 148.
[153] Craddock, Freestone, Gurjar, Middleton & Willies, 1989: 61; Weeks, 2004a: 245.
[154] Biswas, 1996: 354.
[155] Dungworth, 1997b: 905-906.
[156] Craddock, 1978: 13.
[157] Rehren, 1999a: 252.

5.4.3. Historical frame

• **West (Europe & the Mediterranean)**
Among the earliest brass artefacts known in the *West* are the fibulae excavations at the Gordion Tomb in Phrygia, dating to the 8th – 7th c BC. From the 7th c BC, the Greeks also seem to start using the word *oreichalkos*, literary "mountain copper" which later on became the standard word for brass.[158] It was used to describe an expensive, exotic metal not produced in Greece. No zinc was detected in analyses of early Greek bronzes[159]. In Marian and Hittite words with similar meaning are used to describe a metal as "copper of the mountain"[160].

From the 5th c BC, more literary references to *oreichalkos* (= *Aurichalcum* in Latin), would suggest that brass became more common. Unfortunately, direct evidence is not yet available, on the one hand this is due to the lack of analysis of items of everyday metalwork from well-dated Hellenistic origin. On the other hand this is caused by the predominate presence of more prestigious art metalwork in the collections of Western museums and art galleries. However, the frequency of brass items in groups of analysed metalwork from the periphery of the Hellenistic world does suggest that brass was becoming more popular.[161] Some Etruscan 'bronzes' of the 5th c BC contained up to 11% of zinc with less than 3% of tin[162]. By the late 2nd c BC there is evidence of more intense brass usage at both ends of the Anatolian-Persian land mass. From the beginning of the 1st c BC, the occurrence of brass rose and its spread through Eurasia was swift. The expansion in use, which appears uniform in a large-scale overview, however was often uneven and brass was quite selectively used.[163]

Next to brass items one example of an almost pure zinc plate is known from the Agora in Athens, dating to the 4th c BC. There has been a long-standing debate if this object is genuine or not. Other early zinc objects from Europe have largely been proven to be later in date or counterfeits. P.T. Craddock evaluated all the textual and archaeological evidence in Europe and came to the conclusion, that zinc was surely not intentionally made but could have occasionally be produced by accident as a by-product of silver refining (hence the name *mocksilver* sometimes used). Zinc oxide was recovered from furnace walls were part of it sublimated instead of disappearing in the furnace fumes. This oxide was used for medical purposes early on and not for the production of brass. If the conditions were right however, small droplets of metallic zinc can also form on the cooler parts of the furnace wall. This was the only source of metallic zinc and thus for brass prior to the introduction of the cementation process. The small amounts and rarity of the formation of zinc metal can also explain the great value given in early texts to the brass produced from it.[164]

The Roman period is marked by the widespread introduction of brass. The technique was well under control by then and the result of a long period of experimentation. The date when and the place where the cementation production process started is not known, but part of the evidence points to N-Anatolia (Phrygia and Bithynia). There the process might have evolved from at least the 4th c BC onwards.[165] By the 1st c BC the Romans were using the cementation process to produce brass. In the Augustan period, brass with high zinc content was used for coining *sestertii* and *dupondii* at two Imperial mints (Rome and *Lugdunum*). Based on these coins the onset of large-scale production can be reasonably well dated.[166]

[158] Craddock & Eckstein, 2003: 217.
[159] Biswas, 1996: 352; Biswas, 2001: 144.
[160] Craddock & Eckstein, 2003: 217.
[161] Craddock & Eckstein, 2003: 217.
[162] Craddock, 1978: 1.
[163] Craddock & Eckstein, 2003: 217.
[164] Craddock, 1998b: 2-5.
[165] Craddock, 1995: 297.
[166] Treister, 1996: 352.

Next to coinage, brass was widely used within the Roman army[167]. Despite this seemingly 'state controlled' use, it rapidly became popular in other fields, especially for decorative metalwork where it partly replaced bronze[168]. Zinc ores are much more abundant than tin ores, so for large-scale metal production such as coinage, brass had the obvious advantage of cheapness once practical ways of manufacture had been developed[169]. The loss of the tin-producing regions of the western Roman Empire of *Hispania* and *Britannia* together with the loss of easy access to the Bohemian tin fields with the loss of *Pannonia* in the late 4th and 5th c AD, is also seen as a major reason for brass replacing tin-bronze[170]. During the 2nd and 3rd c AD however the zinc content of the coinage fell and brasses with high zinc content ceased to be used. Nevertheless brass continued to be as popular as ever accounting for about 30% of Roman copper-base alloys. It appears that the production of fresh brass largely ceased even before the collapse of the Roman Empire[171].

The use of bronze declines after the introduction of brass in the 1st c AD, a trend that continued during the 1st millennium AD in Europe and the Middle East. The analyses of small Roman copper-base alloy artefacts from Gaul show that the predominant metal used was brass, except for pieces that are believed to be imports. In the NW of the Roman Empire brass seemed to have been the main copper-base alloy for everyday small-scale metalwork already in the Imperial period. There are two ways to interpret this pattern. On the one hand it could be that small everyday items tended to be brass throughout the Empire, whereas prestige and art metalwork continued to be of bronze. In this context it is noticeable that major statuary continued to be of bronze right through into the Byzantine period. On the other hand it may prove that there really was a preference towards brass in the NW-Empire based on the zinc ore deposits at Solberg near Aachen (Germany) when compared to Italy and the Mediterranean centres of the Empire, e.g. local small statuettes are from brass, whereas the Rome counterparts are in bronze.[172]

In his study of Roman alloys P.T. Craddock published a histogram where the tin percentage is plotted against the zinc percentage. He distinguishes three groups. The first group contains less than 4% of zinc, and the presence of zinc here almost certainly arose from the use of scrap brass in the alloy. The second group contains between 4 and 20% of zinc with an essentially normal distribution around 13%. It would seem that these alloys were made by mixing freshly made brass with scrap and that an alloy containing about 13% of zinc was preferred. Alternatively this can be explained due to repeated remelting of the brass. This is the approximate zinc content used now, and in Roman times, for decorative metal. The third group contains between 20 and 28% zinc. These are clearly fresh brasses straight from the smelter, without the addition of any copper or scrap bronze to reduce the overall zinc content. The overall picture that emerges is that of the brass smelters producing a metal containing between 20 and 28% zinc. The Imperial coiners and legionary smiths mixed this with pure copper, whereas the small commercial producers seemed to have used brass mixed with scrap tin-bronzes for reasons of utility and economy.[173]

The conclusions of P.T. Craddock were largely confirmed by a study of D.B. Dungworth on copper-base alloy artefacts from N-Britain[174]. He analysed 1163 artefacts from N-Britain and took particular care in selecting them from different contexts, i.e. *vici*, towns, smaller roadside settlements, villas, hillforts, farmsteads, caves, ritual hoards and a temple, in order to get a more global view of the distribution of certain alloy groups. Also the sample strategy of the artefacts was designed to address artefact types that are normally ignored (e.g. sheet, wire

[167] Craddock, 1978: 1; Rehren, 1999a: 252.
[168] Craddock, 1978: 1.
[169] Craddock, 1978: 11.
[170] Craddock, La Niece & Hook, 1998: 73.
[171] Craddock, 1978: 1; Rehren, 1999a: 252.
[172] Hook & Craddock, 1996: 151-152.
[173] Craddock, 1978: 12-13.
[174] Dungworth, 1997b.

and droplets *versus* the normally more artistic or elite artefacts). A major peak around brass artefacts containing 18% of zinc is reported, although fresh Roman brass should have a somewhat higher zinc ratio. The reduction in the zinc content of many Roman brass artefacts may have arisen from the loss of volatile zinc during the melting of fresh cementation brass prior to casting. Very few brass Roman objects have high levels of lead and if they do these are usually military and indicate the fairly restricted use of this particular alloying technique.[175] The limited amount of low-zinc brasses (i.e. alloys with 2-10% Zn) within the Roman material shows that brass was not recycled on its own. Repeated recycling would progressively have driven off the zinc and produced such low-zinc brasses, but this is not the case. The absence of these alloys shows that if brass was recycled then it was mixed with scrap bronze or pure copper.[176]

The use of brass for coins and military equipment has suggested to some that brass was produced and supplied by a *state monopoly.* If this was true, the highest amount of brass would be expected on highly Romanised settlements such as towns and forts in N-Britain. These settlements show an average amount of 20% of the objects being from brass, interesting however the small rural sites display a much higher occurrence of brass. This high portion of brass on small rural sites seen in N-Britain is similar to that seen in late Iron Age and 'Celtic' metalwork. The widespread use of brass in the early Empire suggests that there was not an official monopoly on the production. Brass was at least in N-Britain widely available.[177]

A study by M. Ponting and I. Segal on the comparison of Eastern Mediterranean Roman military brasses from Masada (Palestine, 1st c AD) with other published analyses of Western material shows strong similarity to the findings of the rest of the Roman Empire. The average zinc content was about 19-20%. Brass with higher amounts of zinc is rather rare, indicating that brass was not used in its as-cast state and most often went through at least one phase of remelting and when diluted this was done with relatively pure copper[178]. In both European and Masada examples, the cast brass pieces contain small but significant amounts of lead, presumably to assist in casting. The wrought pieces, on the other hand, which make up the majority of the Masada material, contain little or no added lead, the small amount of lead detected by the analyses being impurities from the copper ores or from the ore used as the source of the zinc.[179]

The only known shipwreck from the Mediterranean (*ca.* second half of the 1st c BC, Les Magnans B - France) carried small yellow ingots (20-50 cm long) of plano-convex form. They contained 21% of zinc. These ingots most probably are made from *primary brass*, still the zinc value is 'only' 21%. This may suggest that the zinc contents of fresh brass might often have been closed to this value than to the theoretical maximum amount of 28%.

It has been suggested on the basis of coin analyses alone that the knowledge of brass smelting was lost after the 1st c AD and that remelting scrap brass provided the only source of metal for the later coins. However when the growing amount of analyses on objects other than coins are considered this is not correct. The fall of the zinc content for other metalwork is much less pronounced and on the contrary the use of brass increases. It is true that the average zinc content fell slightly and the use of high zinc brasses ended. But this hiatus was filled with a new alloy, i.e. *gunmetal*. The emergence of gunmetal is not to be seen in terms of debasement, but rather that a more suitable alloy for general purposes was being developed (see below for more detail).[180]

[175] Dungworth, 1997b: 903-905.
[176] Tylecote, 1962: 53; Craddock, 1978: 1; Dungworth, 1997b: 904-906; Ponting, 2002: 559.
[177] Dungworth, 1997b: 908.
[178] Ponting, 2002: 559.
[179] Ponting & Segal, 1998: 116-117.
[180] Craddock, 1978: 14; Dungworth, 1997b: 903.

Some information on Byzantine brasses is included in at the next point, since they are compared to Early Islamic copper-base alloys.

- **Middle East & Islamic period**

In a recent study C.P. Thornton and C.B. Ehlers give a list of sites where the oldest zinc containing artefacts from the Middle East were reported: Nuzi (N-Iraq, *ca*. 1400 BC), Ugarit (Syria, *ca*. 1400 BC), Tepe Yahya (S-Iran, *ca*. 15th c BC[181]), Altyn Depe (Turkmenistan, mid 3rd millennium BC) and Umm an-Nar (U.A.E., late 3rd millennium BC)[182], but only few are from controlled excavations. L. Weeks adds to this list some shaft-hole axes from the Royal Cemetery at Ur (S-Iraq, mid 3rd millennium BC) and the site of Ikiztepe (Black Sea coast of Turkey, 3rd millennium BC)[183]. C.P. Thornton and C.B. Ehlers focus on the 2nd millennium BC contexts at the sites of Tepe Yahya and Nuzi[184]. The results from their study show that copper-zinc alloys existed almost 2000 years before the date generally accepted, suggesting that the history of brass is longer and more complex than generally believed.[185] Although generally attributed to the 1st millennium BC, the invention of the cementation process is probably related to the invention of the co-smelting and other ore smelting techniques in the Chalcolithicum of the Near East, documented as early as the 4th millennium BC. These experiments are likely responsible for the explosion in use of copper-base alloys in the 3rd millennium BC with elements such as arsenic, lead, nickel, antimony and tin. Despite the wide spread presence of zinc ores that naturally occur in association with copper ore, there is still little evidence of widespread copper-zinc alloying before the 1st millennium BC[186]. The limited amount of objects C.P. Thornton and C.B. Ehlers discussed do not really change that view, it is however a sign that straight lines are difficult to draw and that also in the Middle East early brasses were part of the array of alloys produced, intentionally or not.

In the frame of this PhD-dissertation we should point out the unusual composition of a small number of Bronze Age objects from Umm an-Nar Island that suggests the possibility of the unintentional production of low-zinc brasses in the Gulf-region. The objects (which contain between 2 and 10% zinc) are highly unusual in the early metallurgy of SE-Arabia, and separated from the ed-Dur material by more than two millennia. Quantitative analysis of a ring from Shimal Site 5 demonstrated the presence of substantial amounts of tin, lead and zinc (i.e. the object was actually a leaded gunmetal, but no exact amounts given). The composition of the ring was in fact used to date the burial in which it was found, after the 1st c BC. A ring from the Iron Age tomb at Bithnah contains 13,1% of zinc and 5,6% of lead (in addition to 2,2% nickel), and most probably belongs to the Late Pre-Islamic reuse of the structure in the 1st c BC – 1st c AD. Although these Umm an-Nar objects are compositional rarities in 3rd millennium SE-Arabia they are paralleled in compositional terms by a number of contemporary finds as mentioned above.[187] Among the finds in the tumuli of Dhahran (SE-Saudi Arabia) bracelets made of an alloy of copper, zinc and lead are mentioned in two instances (Artefact 20/A/7 and Collection 27). No further detail is given on how the excavator came to the conclusion that zinc was present in these alloys, so any further evaluation of these finds is impossible. They are only mentioned in the light of completeness. These artefacts are dated to the late pre-Islamic period and not older than the 1st millennium BC.[188]

One of the earliest literary references to brass is possibly the 8th c BC inscription in the palace of the Assyrian King Sargon II (722-705 BC) at Khorsabad. It mentions the covering

[181] Thornton, Lamberg-Karlovsky, Liezers & Young, 2002: 1457.
[182] Thornton & Ehlers, 2003: 4.
[183] Weeks, 2004b: 57.
[184] Two rings were found of which one has a composition of 14,4% Zn, 0,4% Sn and 3,35% Pb. The second ring actually was made from gunmetal (see below).
[185] Thornton & Ehlers, 2003: 7.
[186] Thornton & Ehlers, 2003: 3.
[187] Weeks, 2004b: 57 (After Corboud, Castella, Hapka & im Obersteg, 1996: Fig. 59)
[188] al-Mughannam & Warwick, 1986: 27: 11-22.

of wooden doors with a sheet of 'white bronze' from Musasir, the mountain region west of the Tigris. Some Assyrian 'bronze' bowls were found to contain about 6% of zinc although still with the usual amount of tin, actually making them *gunmetal*. This suggests that the smith was unaware of the zinc, yet the metal would have been appreciable more golden in colour[189].

The Persians might have used brass from the 5th c BC onwards, and Darius (521-486 BC) had a '*cup which looked like gold but had a disagreeable smell'*, a description that would fit a brass cup.[190] M.Y. Treister points to the possibility that metallic zinc was produced by the Parthians. This is based on the analyses of a letter that Pliny the Younger wrote to Emperor Trajanus about a certain Callidromus. The letter mentions a piece of metal brought from the Parthian mines that, according to M.Y. Treister, could have been a piece of metallic zinc. If this really true is difficult to evaluate since little work has been done on the metallurgy of the Persians, or their successors the Parthians and the Sasanians. The zinc mines in the territory of Iran are however well known. The remains of ancient exploitation were found in the north, in the region of Teheran and Deh-Kvaleh, as well in the centre of the country in the vicinities of Isfahan, Yezd, Kush, Kerman, Saavand, Shiraz, etc. On these sites fragments of clay bars and cones were found. These were used as sublimation surfaces in the furnaces for the zinc vapour. Regrettably the production remains cannot (or could not) be dated[191] and large-scale production of brass does not seem to begin before the 6th c AD in Persia[192].

An interesting study was done by M. Ponting on the copper-base alloy artefacts found at Bet She'an (Palestine)[193]. This is manly due to the fact that objects from different periods were found at the same site, from Byzantine, Umayyad till the Mamluk period. It is interesting that the Umayyad domestic metalwork at Bet She'an is characterized by leaded bronzes and the total absence of brass. This is a marked change from the preceding late Byzantine period, where secondary brasses were the norm for all classes of objects. We have to be aware of the lack of comparative analyses however. The observation of the use of leaded low tin-bronzes is somewhat in contradiction with the analyses of early Arab vessels and 'art' pieces, where brass is predominant from the 9th c AD onwards. Moreover tin was almost certainly not available from local Near Eastern sources and had to be brought in from British Cornwall or SE-Asia (Persia, Afghanistan and Turkmenistan). Tin was therefore likely to have been generally expensive and its supply not always assured. Bet She'an may have been different in this way since it was important and prosperous at this period and was also on the 'silk-route' connecting the Mediterranean with the Far East. The low tin-bronzes can also be the result of recycling Roman bronzes during the Umayyad times. The absence of access to zinc ores may also have induced the recycling of older brass. The zinc sources in Anatolian would still have been in Byzantine hands, but zinc oxide (*tutiya*) could have been imported from Persia and Afghanistan. When Anatolia was annexed in the 9th c AD (the Abbasid period) brass seems to have retaken it's predominate role. Cultural impact and deliberate choice of certain alloys for certain objects should however never be overlooked.[194]

As said above the early Islamic Umayyad (661 – 750 AD) objects are predominantly of low-tin bronze whereas contemporaneous Byzantine copper-base alloy artefacts are predominantly of brass. Gunmetal appears in both cultural spheres however. It has been suggested[195] that brass largely replaced bronze in the Late Antique world and that subsequently brass remained the usual Islamic copper-base alloy up until modern times. This conclusion is primarily based on the analyses of the fine quality metalwork present in

[189] Craddock, 1995: 293.
[190] Forbes, 1964a: 268 & 273; Habashi, 1994: 61.
[191] Treister, 1990: 37-38.
[192] Forbes, 1964a: 268 & 273; Habashi, 1994: 61.
[193] Ponting, 1999.
[194] Ponting, 1999: 1319-1320.
[195] Craddock, 1979; Craddock, La Niece & Hook, 1998.

many museum collections, but another view emerges if domestic objects are brought into the equation. The more common objects seem to be of low tin-bronze (often leaded).[196] The Byzantine brass material shows an increase of the amount of tin present over time. The overall picture of the period between the 5th and 8th c AD shows that the zinc levels of the brasses were relatively low when compared to the previous Roman material and the mean amount is *ca*. 9% (*versus ca.* 18-20% in the Roman period).[197] P.T Craddock describes that the majority of the alloys fall into two basic groups however. The *first* group is relatively pure brass with an average zinc content of about 17%, with little or no tin and/or lead. This alloy was used for sheet or raised metalwork and some castings. The *second* group has a much greater variety in composition, with an average of 12% of zinc, up to 25% of lead and a few percents of tin. This alloy was used exclusively for casting.[198] The first group of brasses would seem to be the fresh product from the cementation. The lower zinc levels might be attributed to a less careful control over the process or to a technological difference and that scrap bronze was used in the cementation instead of 'pure' copper, preventing the uptake of zinc. The second group is rather intriguing because of the often high lead fraction. The persistent occurrence of small amounts of tin is significant. The use of scrap bronze might account for some of the tin but it is also possible that a tin-lead alloy (i.e. *soft solder* or *pewter*, see *Chapter 6*) was used to dilute the fresh brass. [199]

- **East (Afghanistan & India)**

The earliest evidence for brass and zinc in the East, particularly from the Indian subcontinent, is almost all textual. Little relevant archaeological material evidence has yet been uncovered or scientifically examined. Many texts dealing with zinc are known from India, but there is a problem in dating them. Most are copies of ancient texts, themselves only a few centuries old. Despite this drawback they still provide a wealth of documentary evidence. Many texts list zinc as a metal quite separate from tin or lead, some specifying it as a metal made by distillation, and others giving quite detailed descriptions of how this distillation had to be carried out. From the texts it seems certain that brass was in use in the later part of the 1st millennium BC, and that zinc was known by 500 BC. During the 1st millennium AD reference is made in the literature to the production of brass by the cementation process and also of distillation to produce metallic zinc.[200]

A.K. Biswas states that the earliest artefacts containing an appreciable amount of zinc anywhere in the world are from the Indian sites of Lothal (2200 – 1500 BC) and Atranjikhera (ca. 1200-600 BC)[201]. In the light of the information presented above this is not completely correct, but there is a growing body of evidence that in the later part of the 1st millennium BC brass usage may have become more frequent. This is exemplified by the vessels of leaded brass excavated at Begram in the Kabul Valley in Afghanistan (2nd c BC), which join that already published from Taxila (Bhir mound, 4th - 2nd c BC), now N-Pakistan. The Taxila vessel contains 34,34% of zinc, 4,25% of tin and 3% of lead. This high zinc percentage, next to a considerable amount of tin and lead, suggests that metallic zinc was available from at least the 2nd c BC in this region. It may have been introduced by the Hellenistic invaders from the West since the word *arakuta* appears in the book of *Arthasastra*[202], where it is regarded as a recent loan word derived from the Greek *oreichalkos.*[203] On the other hand it is also possible that the Greeks had carried the material or the technology that existed in Taxila

[196] Ponting, 1999: 1313.
[197] Ponting, 1999: 1315-1316.
[198] Craddock, 1979: 73; Craddock, La Niece & Hook, 1998: 77.
[199] Craddock, 1979: 73; Craddock, La Niece & Hook, 1998: 77; Ponting, 1999: 1315-1316.
[200] Craddock, Freestone, Gurjar, Middleton & Willies, 1998: 27.
[201] Biswas, 1996: 352; Biswas, 2001: 142. The object from Lothal contains *ca*. 6% Zn and the two objects from Atranjikhera respectively contain 11,68% Sn and 6,28% Zn, and 20,72% Sn and 16,2% Zn. These last two examples are to be classified as gunmetal.
[202] *Arthashastra* (also spelt *Arthasastra*) is an ancient Indian treatise on economics and politics written sometime between the 4th c BC and 150 AD during the early years of the Mauryan Empire.
[203] Biswas, 1996: 352-353; Craddock, Freestone, Gurjar, Middleton & Willies, 1998: 27.

back to their homeland. This could explain the 4th c BC roll of sheet zinc excavated at the Agora in Athens[204]. So it is not yet clear whether there was a parallel evolution towards the production of brass in the East and the West, or if the technology was transmitted from one region to the other[205], but on the basis of archaeological and numismatic evidence it is clear that brass was already in common use in ancient India during the 1st c BC[206]. A situation more or less mirrored in the West.

The first large-scale distillation of zinc ore to metallic zinc seems to have started in India less than a thousand years ago, around the 12th c AD. Indirect and circumstantial evidences suggest however that the distillation method was already used to a limited extent probably from the later 1st millennium BC onwards[207]. At the site of Zawar (Province Rajasthan) in India evidence was found of the extraction of lead, silver and mainly zinc. The ore at Zawar is principally sphalerite with some marmatite, pyrite and galena, all sulphides. Before any reduction could take place it was essential to remove both the lead and sulphur. The roasting must been done very carefully, because even a few percent of residual sulphides would severely inhibit the reduction process. The ore was then packed in an open-ended clay vessel on which a funnel-shaped clay condenser was fitted. The content of these vessels was a mix of the powdered roasted ore, organic materials, dolomite (as an inert carrier) and other fluxes (e.g. salt), which were shaped in balls. The balls were placed in the open vessel (the *retort*), a stick inserted, and the condenser sealed in place. The stick prevented the charge from falling out when the retort was inverted in the furnace. The reaction is strongly endothermic, requiring a large heat input from outside and was largely governed by the easy movement of gases. This free movement allowed the carbon monoxide to bring about the reduction, and the zinc vapour to escape. On heating the organics would burn, but the fluxes (e.g. salt) would help the sintering of the dolomite preserving the open structure. The carbon monoxide generated from the organic material and the charcoal would reduce the zinc oxide to zinc vapour, which could escape to the condenser down the central passage left by the now burnt stick. It was essential to maintain very reducing conditions, as the reaction is easily reversible. The forming zinc vapour condensed and dripped in its liquid metallic form out of the condenser into the cool chamber below, where it solidified.[208]

- **Remark and conclusion**

Some general remarks can be made on the early brass artefacts discussed above. Many have a zinc content ranging between 5 to 15% (excluding some exceptions). These pieces often contain as much tin as ordinary tin-bronzes. Moreover associated similar objects are made of tin-bronze. This rather suggests that the presence of zinc was not always deliberate and that the smith might not always have been aware of its presence. Although it should be noted that from 12% of zinc onwards there is an observably change in appearance and mechanical properties of the metal. Zinc might also have been present in the copper ore and, under special circumstances during the smelting process (highly reducing, but rather low temperature), had become dissolved in the copper, rather than evaporated and lost to the furnace fumes. When noticed these special furnace conditions could have been carefully replicated to make a zinc-alloy at order.[209] Certain ores might have been preferentially used and/or combined with others, being the start of the co-smelting process. This in time may have led to the addition of pulverised zinc-rich ores to metallic copper and the cementation process.

[204] Biswas, 2001: 144-145.
[205] Craddock & Weirong, 2003: 285.
[206] Biswas, 1996: 360.
[207] Freestone, Middleton, Craddock, Gurjar & Hook, 1991: 617; Biswas, 1996: 358.
[208] This process is described in several articles: Craddock & Gurjar, 1991; Craddock, Freestone, Gurjar, Middleton & Willies, 1989; Craddock, Freestone, Gurjar, Middleton & Willies, 1998.
[209] Craddock & Eckstein, 2003: 217.

The production of brass almost certainly developed during the 1st millennium BC in the Middle East – North India-region. Where the initial spark took place is not clear, nor if the technology evolved independently in different regions or was transmitted from one region to another. The interest in an alternative to tin-bronze was probably due to the general absence of suitable tin deposits in the region. Europe was better served, but even so already during the Roman period, but especially after, brass became dominant.[210]

The geological context for zinc ores will not be discusses since there is no indication that zinc was ever extracted or brass produced in SE-Arabia during the period under consideration. Moreover the production of brass (and/or zinc) is not so much an issue of the presence of the right ores, but of the right technology and knowledge. Suffice it to say that zinc is widespread and present in many complex ores and is often associated with lead, silver, copper, antimony and arsenic[211]. Sphalerite does appear in the certain regions of Saudi Arabia and in the Oman Mountain range[212], but as will be seen below this ore is very unlikely to have provided the zinc that was used to produce the brass in the artefacts found at ed-Dur.

5.4.4. Sample description

- **This study**

Of the 97 samples analysed here 23 are from brass, of which one is leaded. No brass sample was seen amongst the material from Khor Rori. Samples described as 'fragment are pieces that could not be designated to a certain object. They are however not the remains of a 'production process' of any kind, but small parts of objects. Table 13 gives the excavation co-ordinates, the excavation team and the description of each sample.

Reg. nr.	Area	UF	Sq	Loc	Team	Description
AT 013	AT	4264	I – II	G 5155	Belgian	Ring-pommel dagger (drilling)
AV 083	AV	5504	II 1-2	G 5156	Belgian	Large rivet, with traces of iron blade? (drilling)
AV 160	AV	5502	II 1-2	5502	Belgian	Nail from lock plate
AW 021-2	AW	-	-	-	British	Piece of guard (~ AW 063)
AW 062	AW	4541	XVI 5-6	G 5437	British	Sheeting/fitting around iron core
AW 063-2	AW	4541	XVI 5-6	G 5437	British	Ring-pommel dagger, guard attached to handle
AW 063-3	AW	4541	XVI 5-6	G 5437	British	Ring-pommel dagger, nail-shaped fragment guard
AW 063-4	AW	4541	XVI 5-6	G 5437	British	Ring-pommel dagger (drilling)
BL 014	BL	5753	I 2	G 6150	Belgian	Ring-pommel dagger (drilling)
BO 029	BO	5907	I 3	-	Belgian	Small bell pendant
BO 047	BO	-	-	-	Belgian	Ring (finger)
BO 049	BO	-	-	-	Belgian	Ring (finger)
BR 096	BR	6069	I 3	G 6319	Belgian	Decorative element?
BR 103	BR	-	-	-	Belgian	Ring (finger)
BS 066	BS	6557	IV 3	-	Belgian	Stick or small chisel
F 326	F	4423	-	-	French	Ring (finger)
F 330	F	4436	-	-	French	Needle
N 051	N	2404	IV 5	-	Belgian	Vessel fragment
N 251	N	2432	V 6	G 3847	Belgian	Ring (finger)
S 0027	-	-	-	-	Belgian	Small nail
Sample 223	F	1835	-	S 3551	British	Thick fragment
sAV 412 E1	AV	4273	II 1	G 5156	Belgian	Iron fragment with sheeting
sBS 1288 A	BS	Subsurf.	II /III/IV-5	-	Belgian	Thick shaped fragment

Table 13: Co-ordinates of brass objects and samples.

[210] Craddock & Weirong, 2003: 285.
[211] Forbes, 1964a: 261.
[212] Cottrell, 1980 : 570-572; Weeks, 2004b: 12.

• **Previous analyses by L. Weeks** [213]

Among the 33 copper-base alloy objects from ed-Dur analysed by L. Weeks, two brass fragments were recognised. The flat fragments (sBC 593, sN 255) both came from a funeral context (resp. G 6802 and G 3831) and had a zinc content of respectively 10 and 13,7%. Two other pieces also had an elevated zinc content, but also tin was present, placing them in the broad alloy group of *gunmetal* (see *5.5*.). sBC 593 contained 0,4 % of iron, but was free of any other additional elements. sN 255 on the other hand contained 1,1% of tin, 1,1% of iron and 1,1% of nickel.[214] This means that on this small sample of analysis already four (~ 12%) had a considerable amount of zinc incorporated. There is a strong negative correlation between lead and zinc concentrations, i.e. lead is only used in low-zinc objects.[215]

5.4.5. Microstructural observations – optical microscope

Nineteen samples were looked at under the optical metallographic microscope. First in the polished state, after which they were etched wit $FeCl_3$ to reveal the microstructure in more detail. The results of the observations are summarized in table 14.

Reg. nr.	As-cast	Annealed	Twin-lines	Strain lines	Extra
AV 160		x	x		Limited amount of annealing twins (deformed?). Grains > 50 µm.
AW 021-2		x	x	x	Deformed annealing twins. Grains *ca.* 50-100 µm (some 200 µm).
AW 062		x	x		Straight annealing twins
AW 063-2		x	x	x	Deformed annealing twins. Cold working is limited the surface and the thinnest part of the object. Grains *ca.* 50-100 µm.
AW 063-3		x	x	x	Straight annealing twins (some deformed?). Cold working is limited the surface. Grains *ca.* 100 µm (some 200 µm).
BO 029		x	x	x	Straight annealing twins. Grains *ca.* 30 µm
BO 047		x	x		Large variation in grain size across the sample, *ca.* 50 µm but larger and smaller grains appear.
BO 049		x	x	x	Maybe some slight deformation annealing twins in certain areas. Large grains 100-200 µm.
BR 096		x	x	x	Straight annealing twins. Grains *ca.* 50 µm or larger.
BR 103	x			?	Many large lead inclusions
BS 066		x	x	x	Lead inclusions spread along the working direction. Deformed annealing twins, heavily worked at surface. Grains lager than 50 µm (big variety).
F 326		x	x		Straight annealing twins. Large grains, *ca.* 200 µm.
F 330		x	x	x	Heavily worked, deformed annealing twins. Seems to be three-layered, less cold working on middle layer (largest grains). Grains < 20µm near surface.
N 051		x	x	x	Straight annealing twins. Grains 30-40 µm and smaller.
N 251		x	x	x	Straight annealing twins (some deformed?). Cold working is limited the surface. Grains *ca.* 100 µm (some 200 µm).
S 0027		x	x	x	Deformed annealing twins. Small grains *ca.* 20 µm.
Sample 223		x	x	x	Unclear microstructure. Grains weakly developed and areas without grains 30-50 µm others with annealing twins.
sAV 412 E1		x	?	x	Deformed annealing twins. Small grains < 20 µm.
sBS 1288 A	x				Beginning formation of large grains

Table 14: Microstructural observations of brass objects and samples.

All brasses examined here contain less than 36 wt% zinc, actually less than 28 wt%. They are all single phased and a solid solution, the α-phase, where the zinc atoms entered the space lattice of the copper. On cooling from the melt coring takes place so the final appearance is one of copper-rich dendrites in a zinc-rich infilling.[216] These α-brasses are

[213] Weeks, 2004a.
[214] Weeks, 2004a: 245-247.
[215] Weeks, 2004a: 244.
[216] Hodges, 1968: 215.

much more suitable to be cold worked and annealed than hot worked because impurities tend to segregate at the grain boundaries and make the brass weak[217].

Two samples (BR 103 & sBS 1288 A) stand out of the assemblage of microstructure in that they have an as-cast structure (Fig. 22). Part of sample sBS 1288 A was affected by corrosion, but a large part was un-corroded. This area showed the as-cast structure seen on Fig. 22-1. Coring and a dendritic structure can be observed in large grains. A part of the sample had a microstructure not encountered in any other sample (Fig. 22-2). Small rounded dark grey inclusion where imbedded in a matrix that had a distinct different colour (more 'red'). This structure will be further discussed in the SEM-EDX part below. The small grey inclusions are lead (see white circles on Fig. 22-1).

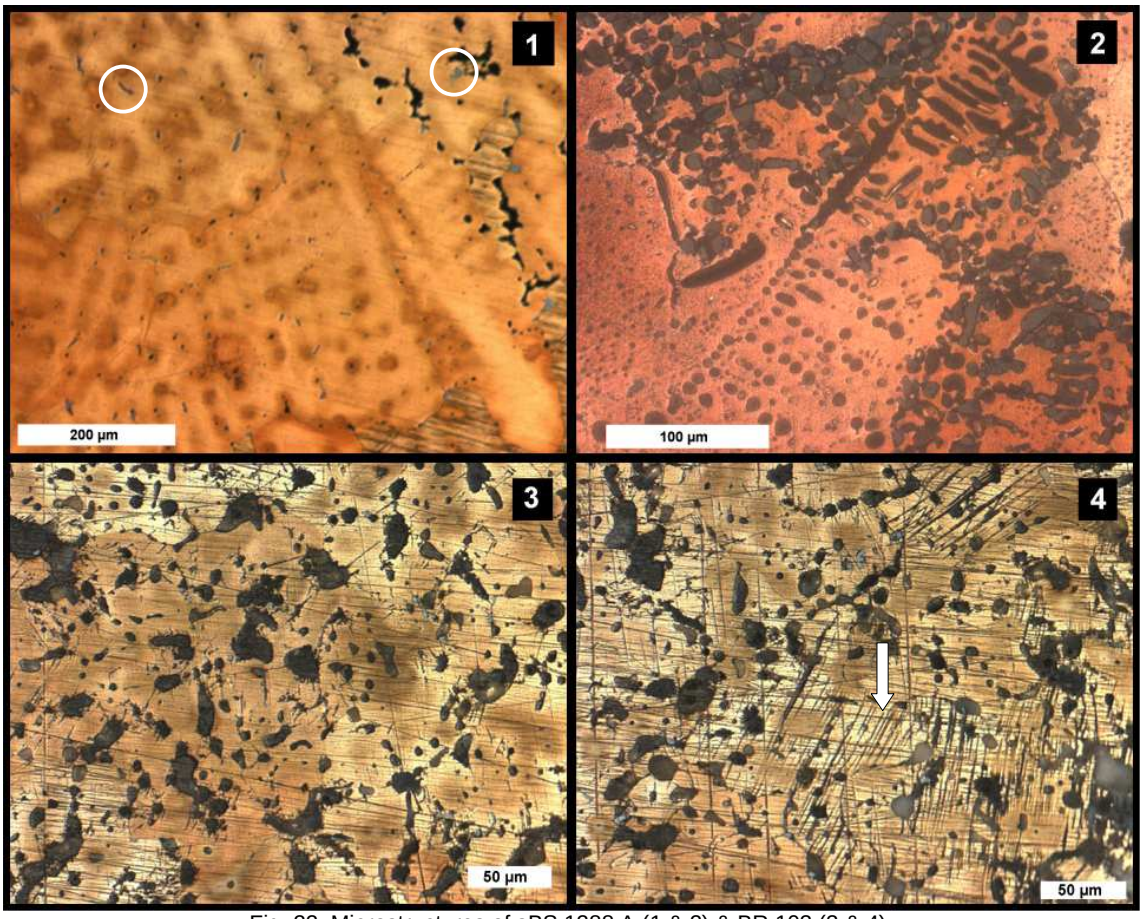


Fig. 22: Microstructures of sBS 1288 A (1 & 2) & BR 103 (3 & 4).

BR 103 is a finger-ring that also has an as-cast structure with clear coring. The black areas seen in Fig. 22-3 and 4 are large inclusions of lead (as verified by EDX). An alloy with such large amount of lead is not suited for cold working, however at least some of the lines seen on Fig. 22-4 seem to be strain lines caused by cold working (white arrow in Fig. 22-4). To produce this ring brass was alloyed with lead and cast to shape. Some additional cold working took place to give the object it final form.

All other brass samples show annealing twins and went through at least one phase of annealing. This annealing was not always sufficient to remove the strain lines completely. Some show evidence of additional working after the annealing phase in the form of deformed annealing twins or the presence of strain lines at the border of the grains. The microstructure

[217] Scott, 1991: 19-20.

depicted below are a representative selection of structures that appear amongst the samples.

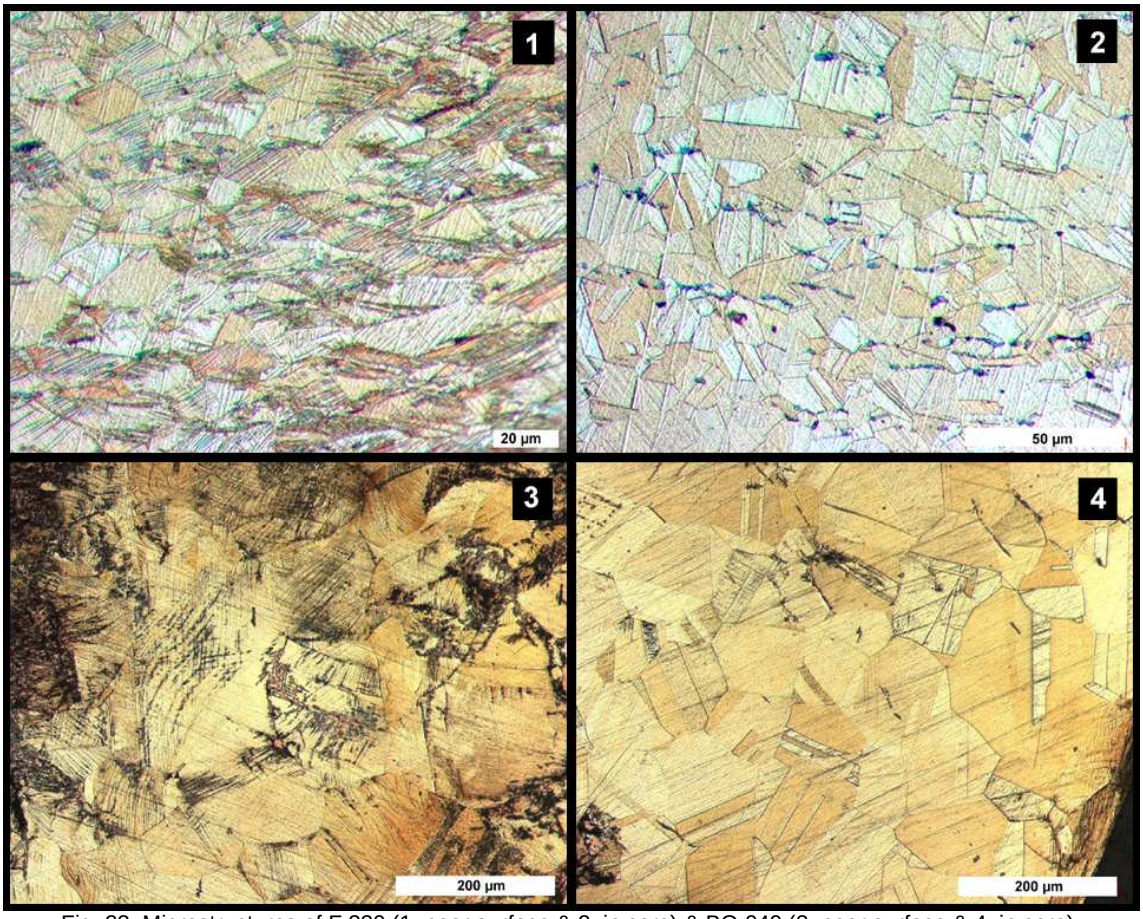


Fig. 23: Microstructures of F 330 (1: near surface & 2: in core) & BO 049 (3: near surface & 4: in core).

F 330 is a small needle (diameter *ca.* 1 mm) and shows that brass was not only used for decorative elements but also for more common daily objects. It could also be a pin (e.g. to hold cloths together) and in this case it would have served a more decorative function. The cast needle was first annealed and all as-cast and coring structures were removed. The section of the objects exhibits three distinct layers of which the middle shows no strain lines or deformed grains (Fig. 23-2). The annealing twins are straight and do show the area was worked but then fully annealed. The lead inclusions are elongated along the line of working. The two outer layer show evidence of extensive cold working in a final stage in the form of deformed annealing twin, strain lines and elongated grains (Fig. 23-1). S 0027 shows severely deformed annealing twins due to the deformation of the small grain received during the hammering. Overall the structure is comparable with F 330.

The finger-ring BO 049 exhibits large grains with annealing twins (Fig. 23-4) that are not deformed. Certain parts however show corrosion along the strain lines and possibly some slightly deformed annealing twins. The cast object was worked and thoroughly annealed (large grains), but not to the extent that all strain lines were removed. In any case the ring was cast, worked to shape and annealed. The microstructure of the other rings BO 047, F 326 and N 251 are similar, although the grains of F 326 are much larger. BO 029 (small bell) and BR 096 (decorative element) also have similar microstructures. They both have straight annealing twins, but strain lines are still visible. Inter-granular corrosion is observed in both samples.

BS 066 is tentatively described as a 'stick or small chisel, but this is only a suggestion. The surface (Fig. 24-1) demonstrates heavily deformed grains and elongated lead inclusions. This is the result of heavy cold working in the final stage of manufacturing. More to the centre (Fig. 24-2) larger grains are seen, but also these are deformed and strain lines and deformed annealing twins are present. The brass was annealed before it was give it final shape.

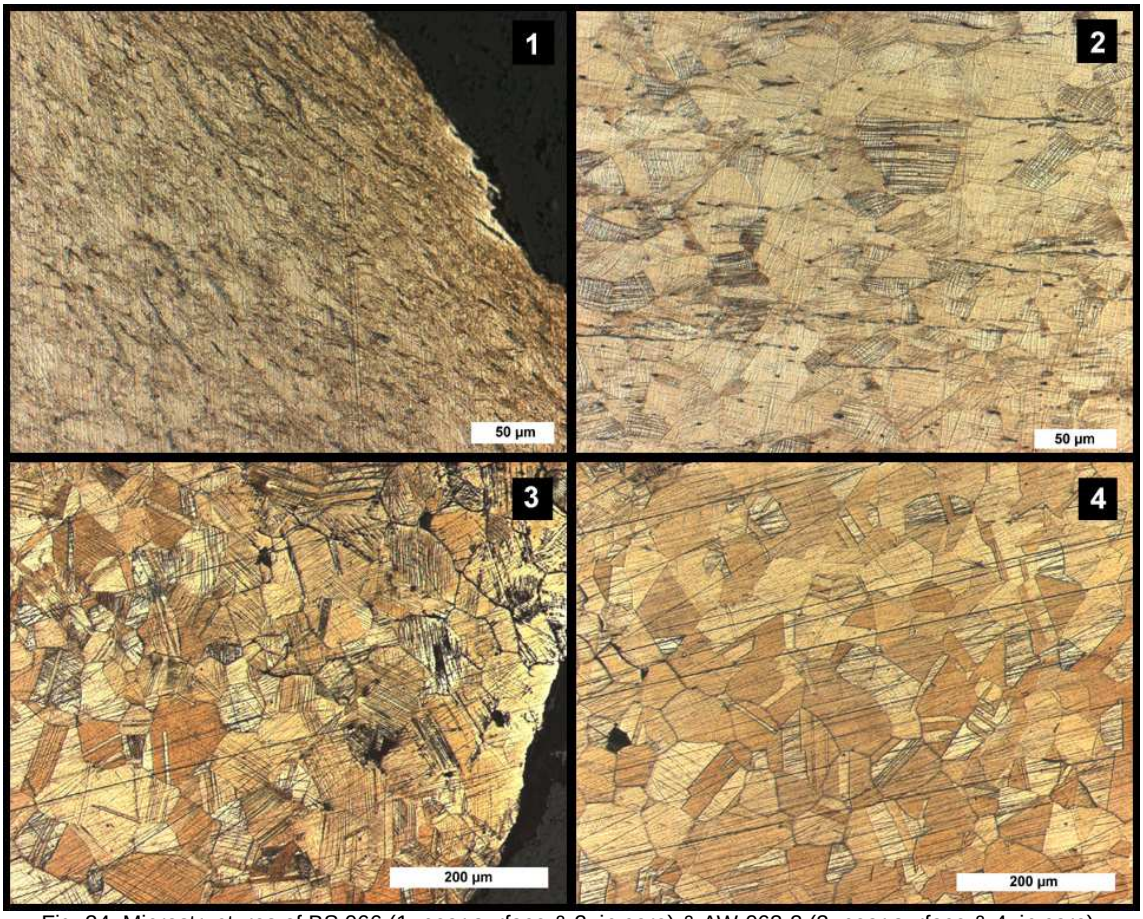


Fig. 24: Microstructures of BS 066 (1: near surface & 2: in core) & AW 063-2 (3: near surface & 4: in core).

AW 063-2 is a small part of the guard of a ring-pommel dagger (see Fig. 47 for a drawing of the object) that was still attached at the base of the handle. AW 063-3 (Fig. 47-1) is from a section through the nail head-shaped extremity of the same guard. Their very similar chemical composition (see EDX part below) serves to underpin the fact that the two samples were originally part of the same guard. The guard was cast in one piece with an opening in the middle. An almost identical piece can be seen on Fig. 25 (AW 021-2). The black arrow indicates the opening the opening where the tang of the dagger past through till above the part where the blade started. The other side of the guard is a mirror of the picture seen. The microstructure of this sample is also similar to AW 063-2 & 3.

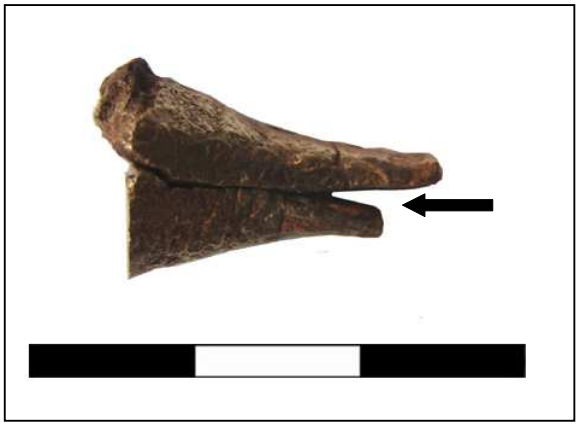


Fig. 25: Fragment of guard AW 021-2 is almost identical to AW 063-3.

The grains show annealing twins and are the result of an annealing phase after the guard was worked to shape (Fig. 24-4). Near the surface corrosion made the strain lines visible, but the annealing twins are not deformed in this part. AW 063-3 (Fig. 26-1) has a similar microstructure, but some slightly deformed annealing twins were noticed, indicating some working after the annealing phase.

N 051 is a large flat fragment probably from a vessel. Fig. 26-2 shows the uneched polished surface and the corrosion made the numerous strain lines visible. After etching only a limited number of annealing twins were seen, suggesting that a plate was cast, annealed, worked and finished by only a limited time of annealing at a low temperature.

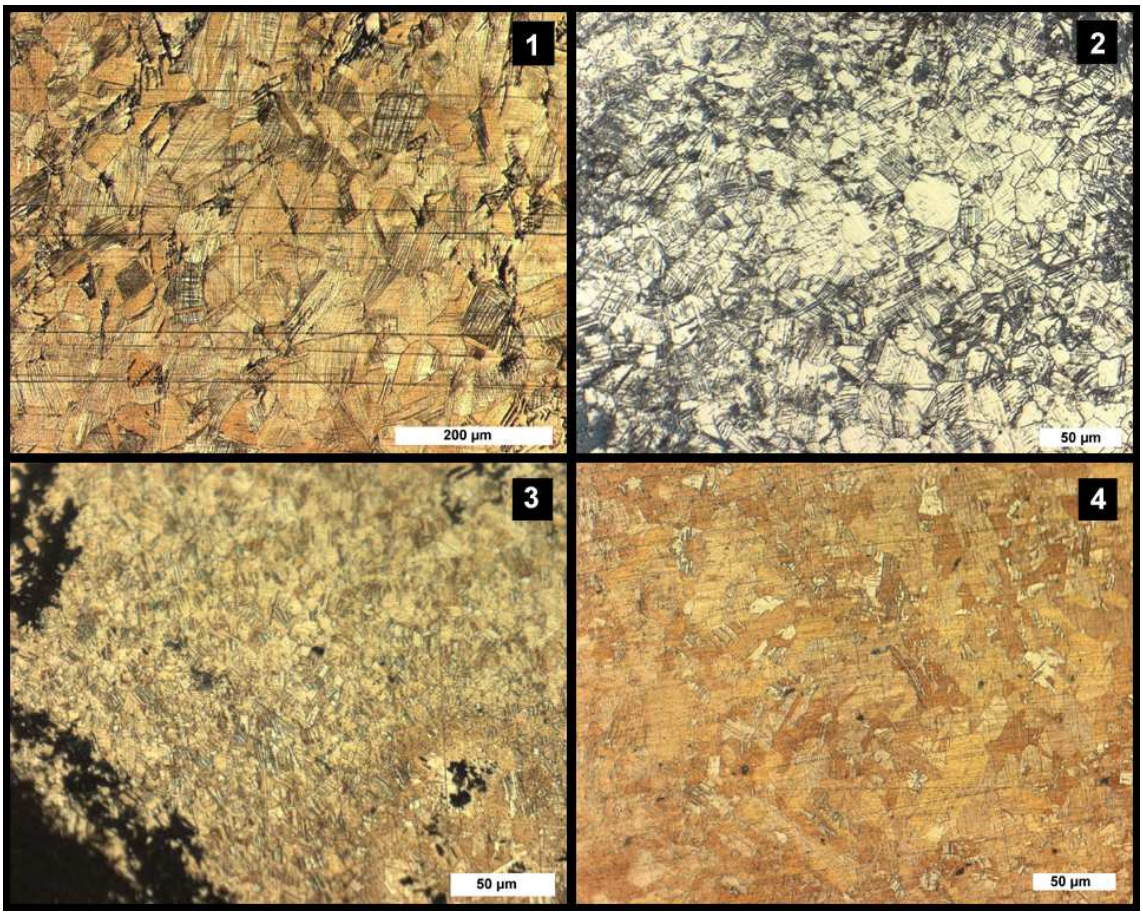


Fig. 26: Microstructures of AW 063-3 (1), N 051 (2, unetched), sAV 412 E1 (3) & Sample 223 (4).

AV 412 E1 is a piece of brass sheet attached to iron, and can be seen as part of the sheeting of a scabbard. The very small grains show deformed annealing twins and strain lines, evidencing cold working after annealing. The small grains suggest that several annealing cycles went on.

Sample 223 is a thick flat piece and might be part of a thick walled vessel, although this is only a suggestion. The microstructure is different from the others in that not the whole surface has similar grains. This may be due to the remains of the coring structure, annealing twins can be observed however.

AW 062 (part of a sheeting around an iron core, scabbard?) shows nicely formed grains with annealing twins and no evidence of any working after the final annealing phase.

AV 160 is a small nail that was used to attach a lock plate. Some of the annealing twins are deformed. This possibly occurred when the nail was hammered in.

A last remark to be made is that inter-granular corrosion, sometimes intra-granular, often appears and that pores are also frequently seen in the samples.

5.4.6. Microstructure & chemical composition – SEM-EDX results

Upon interpreting the analytical results it is important to keep the effect of corrosion processes in mind, e.g. *dezincification*. This is selective corrosion that can severely change the surface composition of an object. Brasses with over 15% of zinc are susceptible to this form of corrosion and the tendency for it to occur generally increases with the zinc content. This can be the result of the re-deposition of copper as a porous outer layer. A second mechanism can be the selective removal of zinc, resulting in a copper-rich residue alloy[218]. To avoid this bias as much as possible, only 'clean' un-corroded surfaces in the centre of the sample were analysed. In the case of the analyses of drillings, the corroded top layer was first removed from the object, before the actual sample was taken, this to avoid as much surface contamination as possible.

Reg. nr.	Cu	Zn	Sn	Pb	Extra
AT 013	76,08	18,86	2,73	2,33	Measurement on drilling
AV 083	77,57	19,33	1,03	2,07	Measurement on drilling
AV 160	78,55	17,63	1,31	2,52	1,40 wt% Fe
AW 021-2	81,04	15,70	0,75	3,26	
AW 062	76,65	20,80	-	2,55	
AW 063-2	78,28	20,24	-	1,48	
AW 063-3	78,34	20,13	-	1,53	
AW 063-4	79,36	19,12	-	1,52	Measurement on drilling
BL 014	79,52	18,92	-	1,56	Measurement on drilling
BO 029	78,25	19,09	-	2,65	
BO 047	79,02	18,87	-	2,11	
BO 049	75,89	18,88	0,62	3,48	0,66 wt% Ag
BR 096	82,91	13,94	0,69	2,46	0,51 wt% Ag
BR 103	79,72	12,22	0,92	7,14	
BS 066	81,60	14,50	1,48	2,41	1,77 wt% Ni
F 326	81,13	16,59	-	2,29	
F 330	82,39	14,05	0,79	2,86	
N 051	80,30	16,76	0,64	2,29	
N 251	77,09	19,72	-	3,19	
S 0027	77,13	19,98	0,56	2,89	
Sample 223	74,55	23,12	-	2,34	
sAV 412 E1	77,13	19,98	0,70	2,89	Some Ag inclusions (few)
sBS 1288 A	87,05	9,03	2,17	1,76	0,56 wt% Ni

Table 15: Chemical compositional data of brass objects and samples in wt%.

The minimum level of zinc present that needs to be present to define an alloy as a brass in this study is 5 wt%. As can be seen in table 15 all (but one, sBS 1288 A) samples are well above this minimum. The 10% cut-off line could have be used as well. One sample can be seen as an 'outlier' (sBS 1288 A with only 9 wt%). For the remaining samples the average zinc content is 17,7 wt% and the mean is 18,9 wt%, with a range from 12,3 to 23,1 wt% (n = 22). Many samples contain some tin, but only in small amounts (average: 0,7 wt%, mean: 0,6

[218] Weisser, 1975: 207.

wt% & range: 0 – 3,7 wt%). A phenomenon noticed on the measurements on standard is that the tin levels detected by EDX tend to be higher than in reality when zinc is also present in the matrix (*Appendix 4*). The figures found here are very likely to be too high, so it can be concluded that in general the tin levels are low and are probably the result of tin present in the copper used or by using small amounts of bronze to dilute the brass.

Only one of the samples is leaded (BR 103) and all the rest is below the limit set to consider them as intentionally leaded. The absence of abnormal lead concentrations shows that when diluting the brass, care was taken not to use any leaded copper (or bronze).

Of all analysed copper-base alloy samples only five contained more than 0,5% of nickel, two of which are brasses and amongst them the highest nickel value found in any of the samples (BS 066: 1,8 wt%).

Silver was found in five samples in amounts equal or above 0,5 wt%, two of which are brasses.

The diagram in Fig. 27 shows the frequency of the objects with a certain amount of zinc is found. The brasses have a considerable range, but a concentration can be seen between 18-22 wt%. This corresponds to mildly diluted brass or the limited remelting of fresh cementation brass. As stated above the mean zinc level is 18,9 wt% and this corresponds well with the data from the Roman World.

It is interesting that some objects have a zinc content in the region of a fresh brass, since this means they were directly cast or produced by limited remelting a preformed ingot. The fact that a 'peak' exists around *ca.* 20 wt% zinc can point to a preferred use of an alloy with such composition. Nowadays brasses of 15 to 20% zinc are often used for low-priced jewellery and in foil-form as a cheap substitute for gold leaf, since this composition has a golden yellow colour. A multitude of terms is used to refer to these modern brasses. The overall terms for the 15 to 20% spectrum alloys are *gilding metal* or *Dutch metal*. Although they are used as cheap substitutes for gold, they are not as noble as gold and are much more prone to discolouration and tarnishing in polluted environments, but they are very corrosion resistant. This kind of alloy must have given the impression that they were of gold, especially when they were polished. This golden colour is very clear on a fresh cut corrosion free section.

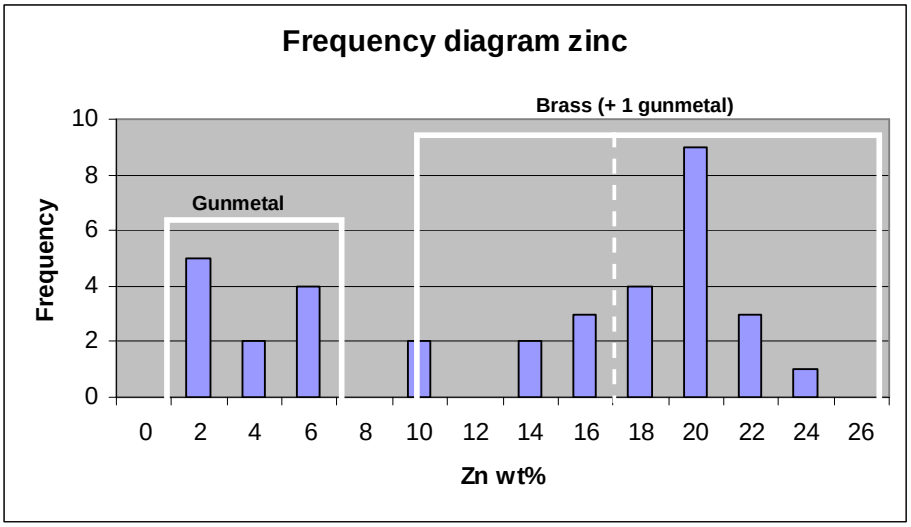


Fig. 27: Chemical compositional data of all zinc containing brass objects and samples.

Three sample stand out at a fist inspection of the results. sBS 1288 A has a low zinc value and is abnormal within this assemblage. The tin value is among the higher ones attested. In first instance sBS 1288 A can be interpreted as the result of repeated remelting and/or the diluting with a low tin-bronze.

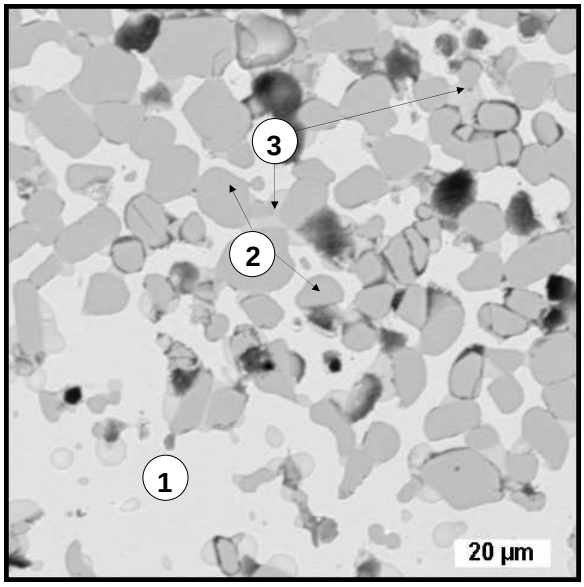


Fig. 28: BSE-image of sBS 1288 A.

The intriguing structure seen on Fig. 28-2 was further examined by SEM-EDX. The light grey matrix is basically copper (***1***), the dark grey rounded inclusions (***2***) contain zinc and oxygen (Zn-O inclusions) and the medium grey areas (***3***) in-between the inclusions are copper containing small amount (2-4 wt%) of zinc (Fig. 28). An alternative explanation for this structure might thus be that this part of the sample is the result of the interruption of the cementation process somewhere along the way. The majority of the sample however has an as-cast structure and shows brass metal without any of these Zn-O inclusions. Moreover this zone is situated along one of the sides of the sample. Possibly something might also have gone wrong during the cementation and part of the crucible charge did not react as planned. If this is the case then the zinc containing areas (***3***) around the Zn-O inclusions show the first stage of zinc being absorbed in the copper. The presence of fairly pure copper explains the 'red' colour seen under the optical microscope (Fig. 22-1 & 2).

The second sample to be mentioned is Sample 223. It has the highest zinc content of all and is close to the maximum uptake of zinc.

The third sample is BR 103 since it is the only leaded brass within the analysed samples. The lead could account for the missing zinc, since they add up to about 20 wt%. It is however more likely that the lead was added to an unleaded brass and was not part of the crucible charge during cementation.

The fragment of the dagger guard (AW 063-2) that was still attached to the handle of the ring-pommel dagger (AW 063) and the nail-shaped fragment that was found next to the dagger (AW 063-3) have almost identical composition, so we can safely conclude that they belong to the same guard.

The brass used for the objects found at ed-Dur was certainly not locally produced. They are almost definitely from Roman origin as will be seen in the use of smithsonite and not sphalerite (see trace elements ICP-MS below). This is supported by the mean zinc contents of the objects, and the historical and archaeological context (see above). Sample 223 is the only fragment that could be been from a fresh brass. It cannot be attributed to a certain object and nothing can be said about this sample, it might however be a piece of a rather thick plate (*ca*. 5 mm thick).

The brass can thus be divided in two compositional groups: the first group are brasses with a high zinc contents and the second group contains brasses that were diluted and/or went through more than one cycle of remelting (Table 16). It should be kept in mind however that a range of factors including temperature, pressure, atmosphere composition and other

alloying elements present, control the exact limit of zinc uptake. The cementation process was often quite efficient, avoiding the production of the volatile metal as a separate stage. But cementation allowed little control over the composition[219] and it is far from clear if the maximum uptake of zinc was always achieved.

Limited remelted or slightly diluted brass (more than 18 wt% Zn)		**Remelted or diluted brasses (less than 18 wt%)**	
Sample 223	Thick fragment	**AV 160**	Nail from lock plate
AW 062	Sheeting/fitting around iron core	**N 051**	Vessel fragment
AW 063-2	Ring-pommel dagger, guard attached to handle	**F 326**	Ring (finger)
AW 063-3	Ring-pommel dagger, nail-shaped fragment guard	**AW 021-2**	Piece of guard (~ AW 063)
S 0027	Small nail	**BS 066**	Stick or small chisel
sAV 412 E1	Sheeting on iron fragment	**F 330**	Needle
N 251	Ring (finger)	**BR 096**	Decorative element?
AV 083	Large rivet, with traces of iron blade?	**BR 103**	Ring (finger)
AW 063-4	Ring-pommel dagger	**sBS 1288 A**	Thick shaped fragment
BO 029	Small bell pendant		
BL 014	Ring-pommel dagger		
BO 049	Ring (finger)		
BO 047	Ring (finger)		
AT 013	Ring-pommel dagger		

Table 16: Two large groups of brasses (arranged according to decreasing Zn content).

The largest amount of objects only went through a limited amount of remelting cycles or was only slightly diluted. The relation between the alloys and the objects will be further discussed below (see *5.6.3.*).

The two flat brass fragments that were attested amongst the samples analysed by L. Weeks had a relatively low zinc level, i.e. sBC 593 contained 10 wt% and sN 255, 13,7 wt%. Only six of the samples analysed here fall within this range. sBC 593 and sN 255 are the result of multiple remelting or diluting of a fresh brass with unalloyed copper.

[219] Bayley, 1998: 9; Craddock,1978: 9; Craddock, 1995: 285; Rehren, 1999a: 253.

5.5. 'Gunmetal'

5.5.1. Introduction, production technique and historical frame

Copper-base alloys of three metallic components, i.e. copper, zinc and tin, are generally grouped under the term *gunmetal*. Many types of gunmetal also contain a few percents of lead. Nowadays they are used for various everyday implements and castings. Mechanically the tin helps the strength of the alloy, the zinc acts as deoxidant, and the lead improves the casting properties and machinability of the gunmetal alloy.[220]

The history of gunmetal is not very clear but is closely linked to the introduction of brass. The oldest gunmetal objects in the Mediterranean are three fibulae from Gordion in Phrygia and date to the 1st millennium BC. They appear next to brass specimens and roughly have a composition of 15 % of tin and above 10 % in zinc.[221] One older gunmetal ring is from a 15th c BC context at Nuzi (12,2% Zn, 6,3% Sn and 4,73% Pb). It is interesting to notice however that other contemporary pieces from Nuzi were of tin-bronze.[222]

Brass and bronze were the most prominent among the Roman copper-base alloys. Gunmetal can be seen as an intermediate alloy in-between these two main groups. There does seem to be a significant chronological change in the alloy types used however. The average zinc content in brasses steadily declined between the 1st and 4th c AD in the Roman world and high-zinc brasses seem to disappear, after which the (leaded-) gunmetal rapidly established itself.[223] In the 1st c AD brass made up 37% of the metal used for Roman objects, but in the 4th c AD it is only used in 4% of the cases. In the same time there is a significant increasing in the use of leaded bronze and leaded gunmetal, from 27% in the 1st c AD to 64% in the 4th c AD.[224]

Roman gunmetal has a fairly wide range of compositions (between 2-8% of Sn and between 3-15% of Zn) and do not show any clustering around a certain composition. The lack of such a peak implies that there was not one preferred composition in the same way as there was for bronze and brass. From this it might be thought that gunmetal was formed by using whatever scrap metal available to hand and so had a random composition[225]. Generally however the fraction of zinc is larger than the fraction of tin. The lack of gunmetal with high levels of zinc and tin on the one hand indicates that brass was not mixed directly with tin but with bronze. If pure tin would have been added, gunmetal with high levels of tin and zinc would be the result (i.e. *ca*. 20% of Zn and *ca*. 10% Sn), but analysed Roman artefacts do not have such compositions. Most of the lead present in gunmetal was probably introduced by the use of leaded bronze rather than the addition of lead to brass.[226] An alternative way in which gunmetal could have come into existence is by using scrap bronze in the cementation process to produce brass[227].

The production of gunmetal was not difficult and was most probably just the result of remelting scrap bronze and brass, since there is no indication that gunmetal was deliberately produced.

[220] Craddock, 1978: 14; Dungworth, 1997b: 901.
[221] Craddock, 1978: 14.
[222] Thornton, Lamberg-Karlovsky, Liezers & Young, 2002: 1457; Thornton & Ehlers, 2003: 7.
[223] Craddock, 1978: 14.
[224] Dungworth, 1997b: 907.
[225] The analyses on material from Masada (1st c AD) showed that the gunmetal objects form a small but distinct group clustering around 14% of zinc and 4% of tin (Ponting & Segal, 1998: 117).
[226] Tylecote, 1962: 53; Craddock, 1978: 1; Dungworth, 1997b: 904-905.
[227] Ponting & Segal, 1998: 117.

5.5.2. Sample description

• **This study**
Seven of the 97 analysed objects fall within the gunmetal group and none were found amongst the Khor Rori material. Table 17 gives the excavation co-ordinates, the excavation team and the description of each sample.

Reg. nr.	Area	UF	Sq	Loc	Team	Description
BR 104	BR	-	-	-	Belgian	Small bell-shaped bead
BS 054	BS	6525	V 3	G 7001	Belgian	Vessel fragment
BS 088	BS	6611	VI-3	-	Belgian	'Spatula', square section
F 128	F	1892	-	T 3521	French	Earring?
S 0022	-	-	-	-	-	Bracelet
sBS 1493	BS	6765	X 4	-	Belgian	Vessel fragment
sM 1250 B	M	-	-	-	Belgian	Shapeless fragment (solidified 'blob'?)

Table 17: Co-ordinates of gunmetal objects and samples.

• **Previous analyses by L. Weeks** [228]
The analyses by PIXE of 33 copper-base alloys samples from ed-Dur showed that two fragments of gunmetal were present. sAT 365 had a composition of 7,7% zinc, 19% of tin and 1,3% of lead, whereas sAT 374 had 5,2% of zinc, 12,9% of tin and 0,5% of lead present. L. Weeks states that the presence of brass and gunmetal at ed-Dur is almost certainly linked to the significant contacts that existed between the site and the Roman world, although the ultimate point of manufacture of these alloys is uncertain[229].

According to L. Weeks the absence of leaded gunmetal at ed-Dur is interesting, as these alloys are known from other metal assemblages in Western Asia and the Mediterranean, and from Late Pre-Islamic sites in the U.A.E.[230]

5.5.3. Microstructural observations – optical microscope

All five samples were looked at under the optical metallographic microscope. First in the polished state, after which they were etched wit $FeCl_3$ to reveal the microstructure in more detail. The results of the observations are summarized in table 18.

Reg. nr.	As-cast	Annealed	Twin-lines	Strain lines	Extra
BR 104	x	x			Equi-axed grains (< 50 µm) with remains of coring. Some α+δ-eutectoid islands. Areas with large lead inclusions, concentrated near outer surface. Inter-granular corrosion.
BS 054		x	x	x	Cold worked and not completely annealed, surviving strain lines. Grains (*ca.* 30-40 µm.
BS 088	x			x	Mildly cold worked with remains of the as-cast structure. Sliver inclusions.
F 128		x	x		Worked and annealed. Large lead inclusions oriented in the line of working. Grains *ca.* 25-40 µm.
S 0022		x	x		Cold worked and afterwards annealed. Large lead inclusions oriented in the line of working. Grains *ca.* 10-30 µm.
sBS 1493		x	?	x	Annealed after casting (no as-cast structure) remains of working in the form of corrosion along strain lines. Grains *ca.* 50-60 µm.
sM 1250 B		x	x	x	Remains of coring. Equi-axed grains (between 50 & 100 µm large) with strain lines. Few annealing twins. Some α+δ-eutectoid islands.

Table 18: Microstructural observations of gunmetal objects and samples.

[228] Weeks, 2004a.
[229] Weeks, 2004a: 247.
[230] Weeks, 2004a: 244. For Shimal only an alloy containing Cu, Zn and Sn is mentioned, but no analytical results were published (Craddock, 1985: 98 & Tab. 10).

BS 054 (Fig. 29-1) is a vessel fragment and shows inter-granular corrosion and pores in the matrix. The lead inclusions are elongated in the line of working at the grain boundaries, this means that they are parallel to the surface and follow the direction that the metal spread when worked. The same goes for the pores. The annealing twins in the grains are straight, but not all strain lines were removed by the final annealing phase. The small size of the grains, the limited amount of annealing twins and the remnants of the strain lines suggest that the final annealing was at a moderate temperature and for a relatively short period of time. This microstructure is in accordance with the driving-out of a cast sheet with a short final annealing period.

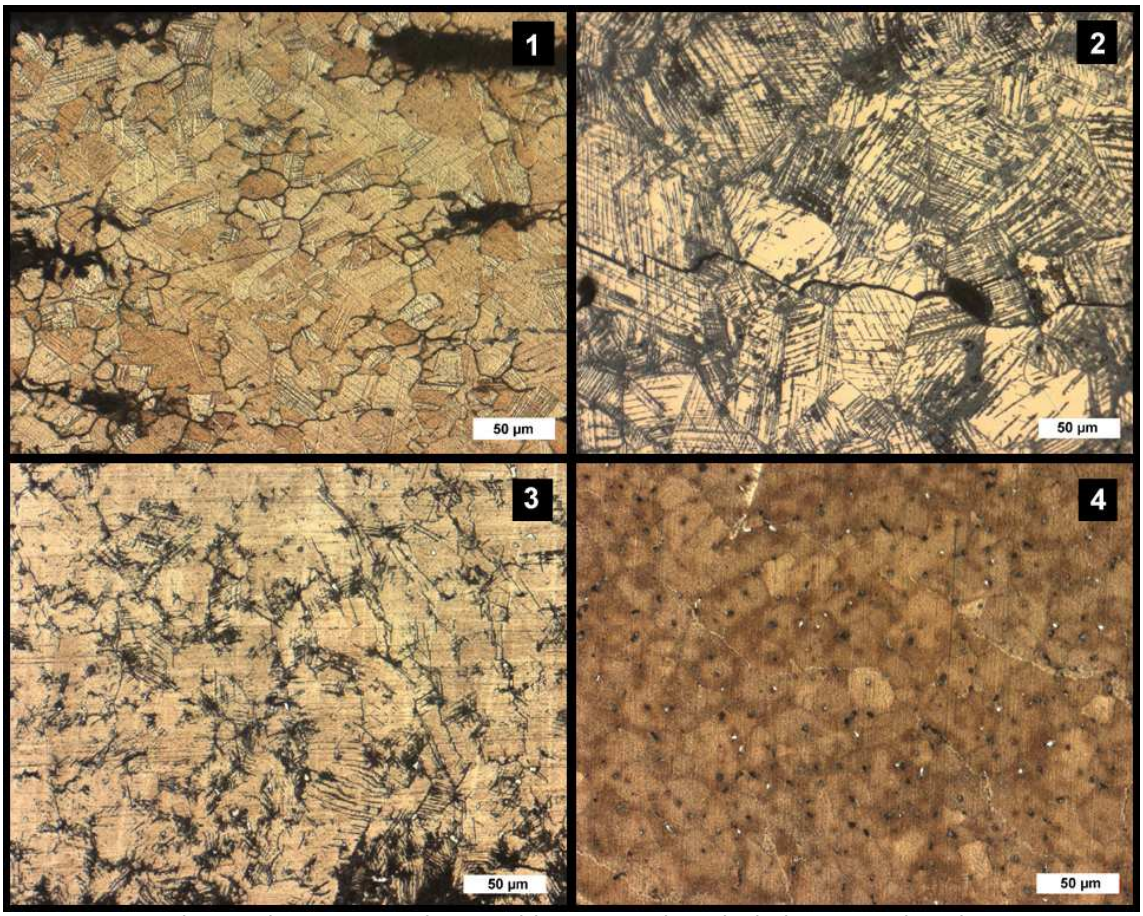


Fig. 29: Microstructures of BS 054 (1), sBS 1493 (unetched, 2) & BS 088 (3 & 4).

sBS 1493 (Fig 29-2, vessel fragment) shows extensive corrosion along the strain lines of the grains and some pores. Some (deformed?) annealing twin shows that the cast was annealed after a phase of working. Additional (cold) worked generated many strain lines and some deformed annealing twins. The corrosion made it impossible to obtain a clear microstructure after etching.

BS 088 is a 'spatula' (?). Fig. 29-3 shows the microstructure near the surface, whereas Fig. XX-4 is a picture of the core of the sample. The white dots are silver inclusions and the grey ones lead globules. They are not deformed showing that only limited cold working was done after casting this object. Fig. 29-4 has a cored structure, remnant of the as-cast structure. Grains can however be already observed, but no annealing twins. The cast was thus annealed for a short period of time at/or moderate temperatures. The surface shows corrosion along the grain boundaries and possibly the lines seen in Fig. 29-3 are strain lines implying some working to give the object it definitive shape.

The 'earring', F 128 (Fig. 30-1 & 2), has lead inclusions that are elongated in the line of working. Inter-granular corrosion can be observed. As a whole the annealing twins are limited, but since they are straight the object was finished with a final annealing stage.

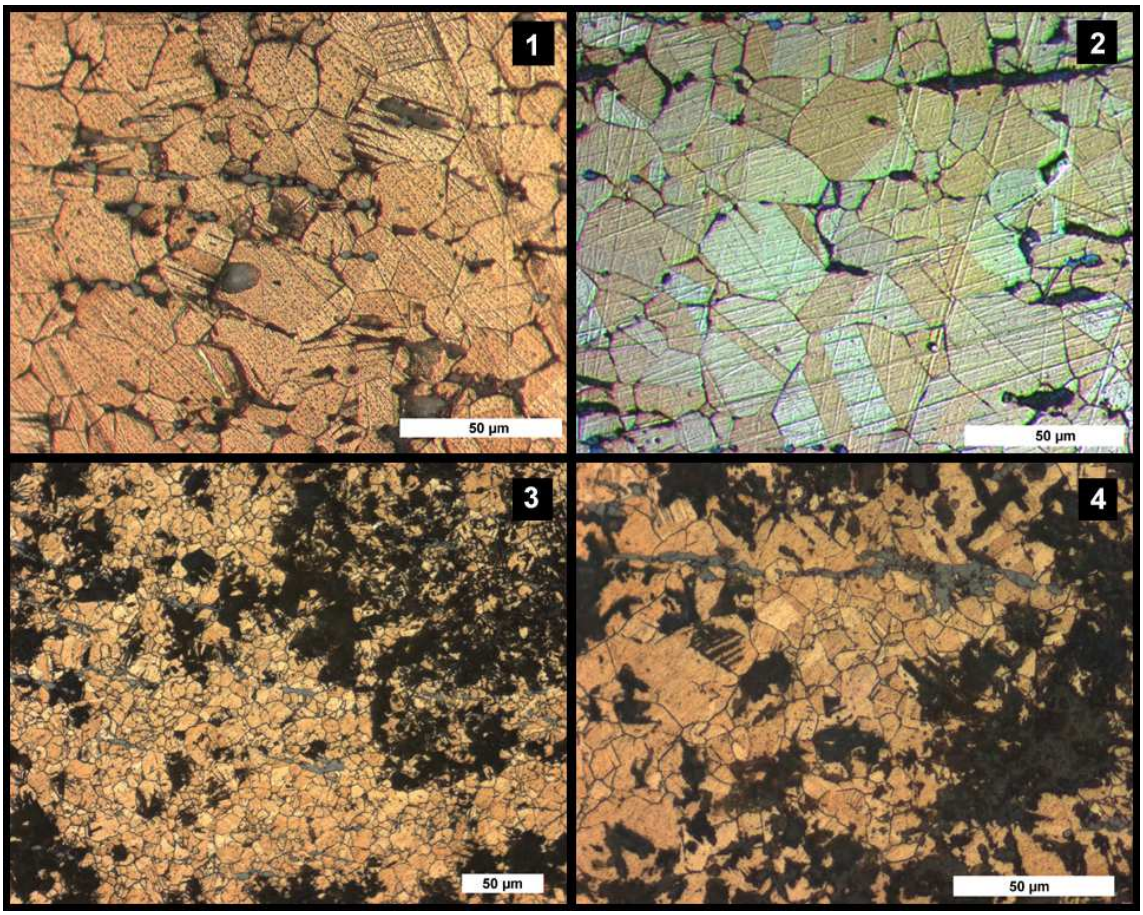


Fig. 30: Microstructures of F 128 (1 & 2) & S 0022 (3 & 4).

S 0022 (a bracelet) is severely corroded along the and inside the grains. The small grains suggest repeated cycles of working alternated with annealing, finished with a final annealing phase (straight annealing twins). The lead inclusions are elongated along the line of working.

Sample sM 1250 B (Fig. 15-4, due to a mistake this sample was included in a different plate) has nicely formed equi-axed grains with relatively few annealing twins. Strain lines are however seen in many grains and some α+δ-eutectoid islands were also present. All this may indicate that this fragment was worked before annealing and that the annealing did not remove all stress. Alternatively the fragment may have been annealed, worked and annealed again to a limited extent. A similar microstructure but with smaller equi-axed grains is attested in BR 104 (a small bell). No strain lines and annealing twins were seen, indicating this object was cast to its shape and fully annealed, although some remnants of the coring might be present. There is extensive inter-granular corrosion.

Except for BS 088 all samples were annealed at least once and the as-cast structure and coring was removed.

5.5.4. Chemical composition – SEM-EDX results

The original limit set to define an alloy as *gunmetal* was copper with more than 5 wt% of zinc and more than 5 wt% of tin. Among the samples listed in table 19 actually only one (BS 054) is a true gunmetal. The other samples listed in table 19 however clearly stand out of the analysed assemblage and it was felt better to treat them separately. Moreover it can be argued what the minimum limit of zinc (and tin for that matter) should be. Gunmetals are the result of recycling bronze and brass together, and the fractions introduced in the melt determine the fractions present in the eventual alloy. Maybe the less rigid definition like the one D. Dungworth used for Roman material from N-Britain has to be adopted, i.e. a range for tin between 2-8% and 3-15% of zinc[231]. This however does not enclose all alloys seen in table xx, since the range of tin values at ed-Dur is larger. For the purpose of this study I will use a bit of 'wet finger work'[232] and define gunmetal with both too much tin and zinc to be classified as a brass or a bronze.

Reg. nr.	Cu	Zn	Ag	Sn	Pb	Extra
BR 104	81,68	4,25	-	8,89	5,18	1,37 wt% Fe
BS 054	82,45	9,59	-	5,34	2,62	-
BS 088	79,33	3,46	5,76	9,13	2,32	Silver present (AAS: 7,55 wt% Sn)
F 128	85,90	4,08	-	7,33	2,70	-
S 0022	79,74	4,93	-	11,78	3,55	0,54 wt% Fe
sBS 1493	78,65	4,78	-	13,72	2,85	
sM 1250 B	85,54	3,41	-	8,10	2,95	

Table 19: Chemical compositional data of gunmetal objects and samples in wt%.

The tin levels detected are probably somewhat on the high side, since it was noticed that the tin values tend to be elevated if zinc is also present in the matrix. This was realized at a too advanced state of this PhD to effectively adjust the data and no means were found to correct this error. This point needs attention if any further research is to be performed. This flaw does however not affect the global trend and conclusions drawn.

Two groups of gunmetal can be distinguished. The *first group* contains BR 104, F 128, S 0022, sBS 1493 and sM 1250 B. Their tin level is high than their zinc level. To this group also BS 088 can be added although this samples has a considerable amount of silver included. If the silver is taken out of the analyses and normalized to sum up to 100% the composition would be: 84,2 wt% Cu, 6,1 wt% Zn, 9,7 wt% Sn and 2,4 wt% Pb. This fits the profile of the first group defined. The two samples analysed by L. Weeks, sAT 365 and sAT 374[233], also have a larger tin fraction present than zinc. S 0022 and sBS 1493 have a similar composition then sAT 374. sAT 365 has an exceptional composition, since it contains 19 wt% of tin and 7,7 wt% of zinc. This type of alloy was not encountered among the samples analysed here and can be considered as an abnormality. As mentioned above this *first group* are not 'real' gunmetals according to the definition.

The *second 'group'* is only represented by one sample (BS 054) and has a higher zinc than tin level. This sample is thus to be considered as an exception and is a 'true' gunmetal.

The compositional results obtained for the *first group* here do not fit the conclusions of analysed Roman material where the tin level is generally lower than the zinc fraction. The *second 'group'* on the other hand is in better accordance with that.

How did the gunmetal of the *first group* come into existence then? A first hypothesis would be that pure tin was mixed with brass. This however implies that the alloying was intentional,

[231] Dungworth, 1997: 904.
[232] Flemish saying that means 'to do something off the top of one's head'.
[233] sAT 365: 7,7 wt% of Zn & 19,0 wt% of Sn – sAT 374: 5,2 wt% of Zn & 12,3 wt% of Sn.

since tin was an 'expensive' metal. The fact that brass just became popular because it was a cheaper alternative for bronze is not to be forgotten. The argument made here is that it would be illogic to add the metal that you are substituting. A second argument to reject this hypothesis is that the resulting alloy from such an action would contain both high levels of zinc and tin (i.e. *ca*. 20% of Zn & *ca*. 10% Sn). This is not the case for the ed-Dur gunmetal, nor is it for the Roman material.

A second hypothesis is that the gunmetal was the result of the use of scrap bronze instead of copper in the brass cementation process. The presence of tin (and lead) in this process would reduce the amount of zinc that could be taken up in the resulting alloy. The tin values in the gunmetal imply that in this case a medium tin-bronze was used as scrap bronze. The presence of a given percentage of lead will reduce zinc absorption by the same amount, whereas tin will reduce the zinc absorption by twice its own concentration[234]. Theoretically most of the samples in table 19 could be made in this way, i.e. the levels of tin and lead can account for the 'missing' zinc. But brass production in the 1st c AD seems to have been a rather specialised activity, restricted to a certain amount of production centres. It seems unlikely to me that they would use the more valued tin-bronze scrap to produce brass. Bronze could be recycled on its own and reused, without wasting it to an alloy such as gunmetal. Moreover if scrap bronze was used for cementation on a regular basis, more gunmetal of the composition attested at ed-Dur should turn up among the ones distributed in the Roman Empire. This is not the case if the small amount of published data on gunmetals is considered as representative of the reality.

This leaves the remaining third hypothesis of recycling. The compositional results suggest that in any case a medium tin-bronze was recycled with a small amount of brass. This would yield relatively high tin and low zinc values. Alternatively brass that had been recycled on its own for several times, every time reducing the amount of zinc present, could have been added to a medium tin-bronze. This would give a similar result.

The broad difference in composition of *group 1* with Roman material suggests that the gunmetal found at ed-Dur was probably not made in the Roman World. This does not mean that the metals used for the alloying could not have been 'Roman', but only that the process of alloying might have taken place outside the Roman sphere.

The lead levels measured in the gunmetal are not different to these of unleaded bronze or brass, and in any case no lead was intentionally added. The small bell BR 104 is the only leaded gunmetal, but for the rest care was taken not to include lead in the alloy. The tin values must originate from the use of medium tin-bronze (as defined in this study) in the recycling process and possibly a small amount of brass (unconsciously?) was mixed in the melt.

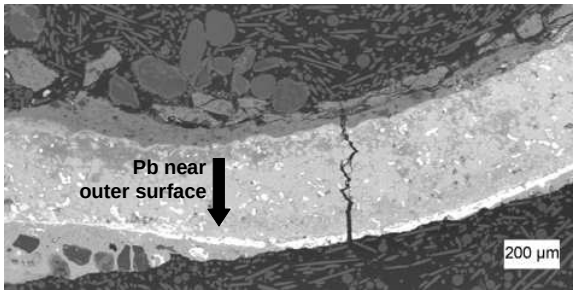


Fig. 31: Cross-section BR 104 (BSE-image) showing the lead enrichment at the outer surface.

The presence of lead in the small bell BR 104 is probably to ease the casting of this small object. No indication of any working after casting is seen (no annealing twins), but equi-axed grains are present and the small bell was annealed after casting. The grains are rather small (< 50 µm) and the remains of coring are still visible, indicating a short annealing phase at low temperatures. It is interesting to notice that the lead is very unevenly distributed and a large concentration is seen near the outer

[234] Craddock, 1978: 12; Ponting, 2002: 560.

surface of the bell (Fig. 31). Upon cooling after the cast the lead was expulsed from the metal towards the fastest cooling outer surface. The presence of lead on the surface would have given this object a grey appearance instead of the more golden colour of the bronze.

The silver present in the 'spatula' BS 088 is intriguing. What would be the benefit of a Zn-Ag-Sn-Cu alloy? The silver might have been introduced by recycling a silver containing copper-base alloy (e.g. hard solder) with brass. Or in the light of the pickling process (see *Chapter 7*, pp. xx-xx) suggested for the coins, the silver might have been added to get a desired surface effect, e.g. a silvery gloss.

5.6. Alloys & objects

5.6.1. Alloy groups

In this point the previous presented information will be further evaluated. The defined alloys will be compared amongst each other and to the artefact they belong to. The contextual information on the artefacts was kindly provided by A. De Waele and is only added for the completeness. I would like to refer to her coming PhD for more details and references.

During the Belgian excavations 156 objects and 90 samples were registered in the initial database as being made from copper-base-alloys. The total amount of samples and fragments of objects from ed-Dur analysed here is 91, of which 41 came from objects and 22 from samples collected during the Belgian excavations, the remaining samples (28) were found by the other teams that worked on ed-Dur. To this we have to add the 33 samples analysed by L. Weeks, bringing the total amount of copper-base alloy fragments from ed-Dur examined for their chemical composition at 124. Six samples were also obtained from the site of Khor Rori. Table 20 summarizes the composition of the assemblage.

Alloy	# objects & samples		# per team	Total #
Copper	*Objects*	*11*	6 Belgian 1 British 2 Danish 1 unknown *1 Khor Rori - Italian*	**19**
	Samples	*8*	8 Belgian	
Bronze	*Objects*	*38*	19 Belgian 8 British 3 Danish 3 unknown *5 Khor Rori - Italian*	**48**
	Samples	*10*	10 Belgian	
Brass	*Objects*	*20*	13 Belgian 5 British 3 French	**23**
	Samples	*3*	2 Belgian 1 British	
Gunmetal	*Objects*	*5*	3 Belgian 1 French 1 unknown	**7**
	Sample	*2*	2 Belgian	

Table 20: Composition of the assemblage analysed in this study.

Ten of the samples were to severely corroded to be included in the more detailed grouping of the alloys. They however all contained large amounts of tin, making them the corroded remains of bronzes. To present an as broad as possible picture of the alloys used at ed-Dur the analyses done by L. Weeks will also be include wherever possible (Table 21).

	All analysed samples (incl. Khor Rori)	All analysed samples (excl. Khor Rori)	All samples from ed-Dur (incl. Weeks, 2004a)
General alloy groups			
Copper	19	18	27
Tin-bronze	48	43	63
Brass	23	23	25
Gunmetal	7	7	9
Total	*97*	*91*	*124*

	All analysed samples (incl. Khor Rori)	All analysed samples (excl. Khor Rori)	All samples from ed-Dur (incl. Weeks, 2004a)
	Detail alloy groups		
Copper (unalloyed)	18	17	26
Copper (leaded)	1	1	1
Tin-bronze (corroded)	10	9	9
Low tin-bronze (leaded)	4	3	3
Medium tin-bronze	19	18	22
Medium tin-bronze (leaded)	8	7	14
High tin-bronze	3	3	10
High tin-bronze (leaded)	4	3	5
Brass	22	22	24
Brass (leaded)	1	1	1
Gunmetal	6	6	8
Gunmetal (leaded)	1	1	1
Total	*97*	*91*	*124*

Table 21: Composition of the assemblage analysed in this study.

Fig. 32 gives a graphic representation of the different main alloy groups attested at ed-Dur in this study and in the publication of L. Weeks. The third (yellow) bar gives the combined result of both studies and the most complete picture of the alloys attested at ed-Dur. The absolute amounts of samples are difficult to compare because of the difference in the number of samples analysed in both studies. To overcome this the results of both studies are converted to their percentile fraction.

Except for the gunmetal the results tend to differ considerably. The amount of brasses found in this study is much higher. Whereas they form a minority in L. Weeks study, they are the second most dominant alloy attested in this study. For both bronze and unalloyed copper the percentile fraction in this study is lower. The combined data are in broad lines similar to the results obtained here.

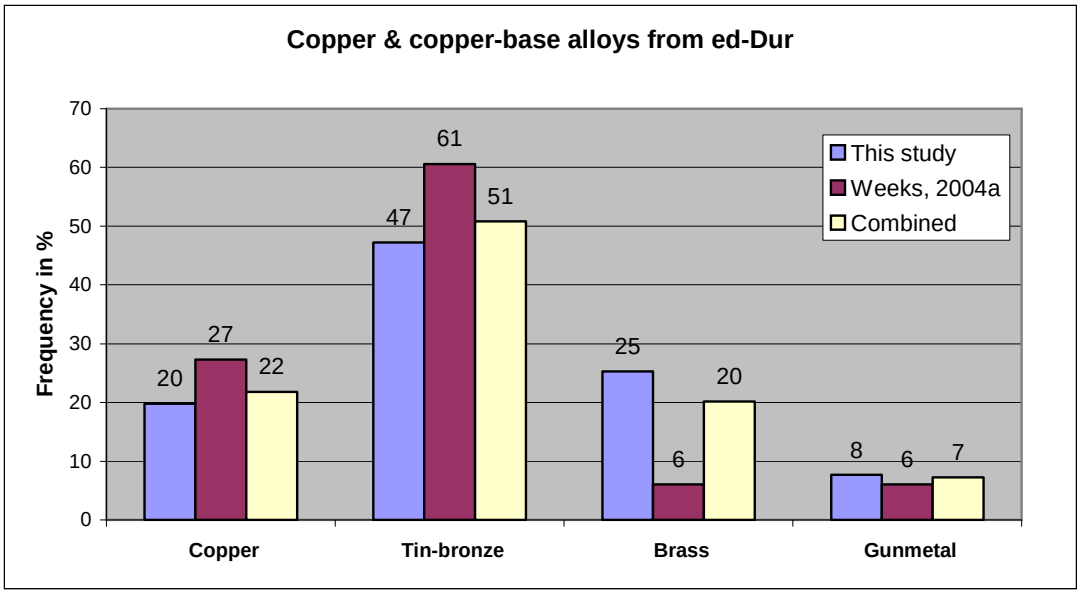
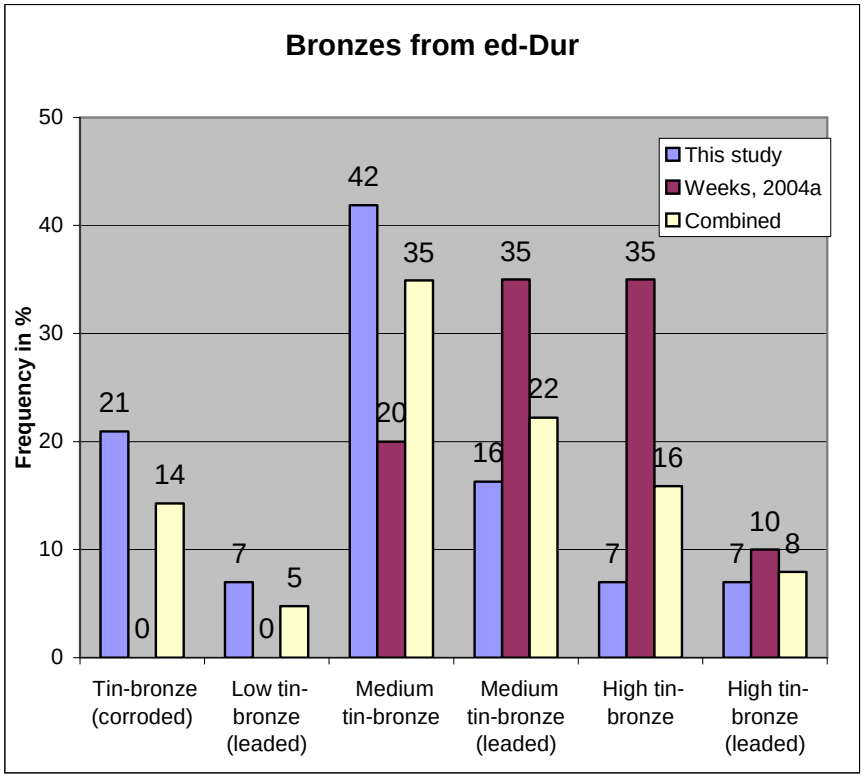


Fig. 32: Frequency (in %) diagrams of the distribution of the main alloy groups from ed-Dur.

To have a more detailed look at the results on the bronzes in L. Weeks' analyses and the results obtained here, the data is plotted on two frequency diagrams (Fig. 33) in the same

way as described above. The first diagram includes all analyses on samples and objects from ed-Dur. In the second plot the nine fragments that were too corroded to give reliable results were removed.



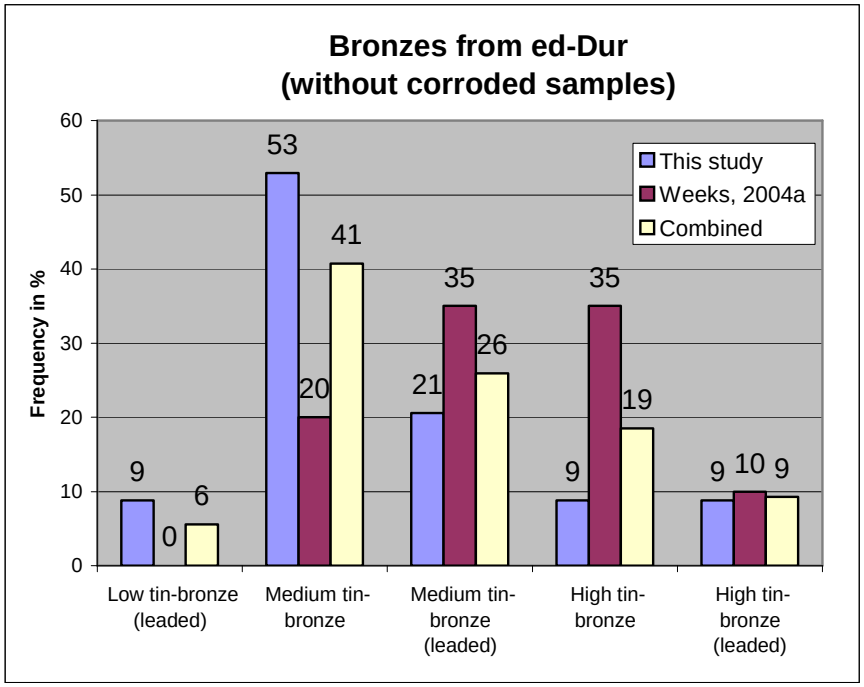


Fig. 33: Frequency diagrams giving the percentile fraction of every alloy type.

The first difference to be noted between the two datasets is the absence of low tin-bronze in amongst L. Weeks' samples. The amount of medium tin-bronzes is overwhelmingly higher in our analyses, although more leaded medium tin-bronzes are seen in L. Weeks' dataset.

The amount of high tin-bronzes is much larger in L. Weeks' data. For the leaded high tin-bronzes the amount is similar. It can be concluded that the analyses performed here are an essential contribution to get an idea of the alloys used and attested at ed-Dur.

It is felt that some of the data published by L. Weeks might be biased by corrosion effect, since bronzes are seen with more than 33,8 wt% of tin. It should be noted that the PIXE analyses used in his study does not have the same semi-quantitative character as EDX and produces more accurate measurements. Such high tin levels are strange and no information on such alloys was found in literature.

The overall picture shows that medium tin-bronzes and leaded medium tin-bronzes were the most important bronze alloy used. Followed by high tin-bronzes and their leaded variant. Leaded low tin-bronzes are the least numerous. Considering all alloys, bronze is the most important alloy, followed by unalloyed copper and brass. Gunmetal is the least well represented alloy.

5.6.2. Alloy metals

Three alloy metals can be considered, i.e. tin, zinc and copper. These are plotted in function of each other in three scatter plots (Fig. 34, 35 & 36).

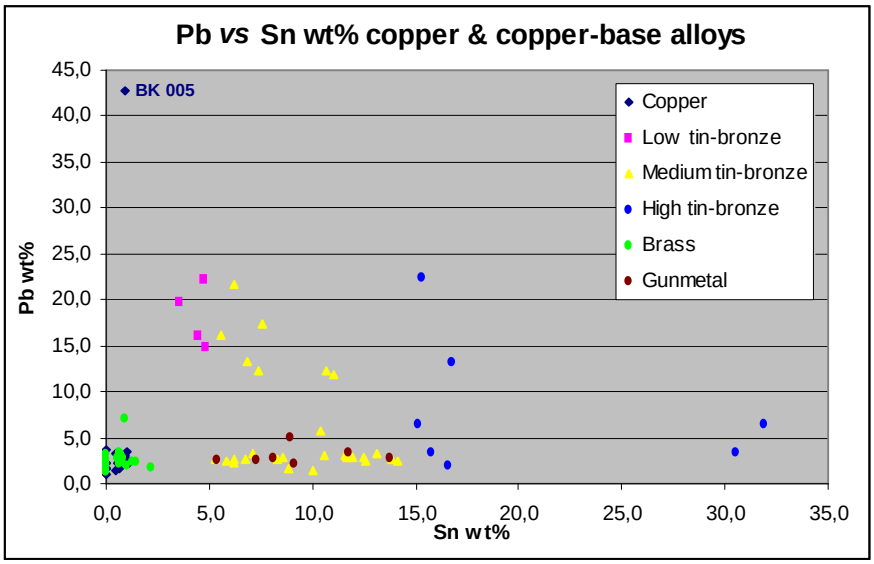


Fig. 34: Scatter plot of the lead wt% *versus* tin wt% of all copper & copper-base alloys.

Fig. 34 gives the plot of the lead values in relation to the tin values. The different alloy groups are clearly separated, except for the unleaded medium-bronzes and the gunmetal. This shows that the gunmetal was made by mixing medium tin-bronze and brass.

A tentative division can be made for the leaded artefacts. The mildly leaded examples with less than 10 wt% of lead and the heavy with more than 10 wt% of lead. The outlier BK 005 is likely to have a too high lead value. This is the lion bead that was analysed on a small polished section of the object itself. Due to segregation when cast the lead was concentrated at the surface. Probably not enough of the surface was removed of this already small object before analyses.

Fig. 35 gives the plot of the lead values in relation to the zinc values. The different alloy groups are less clearly separated, except for the brass and the gunmetal. It (obviously) shows that except for these two alloy groups, the zinc levels are low for the other alloys.

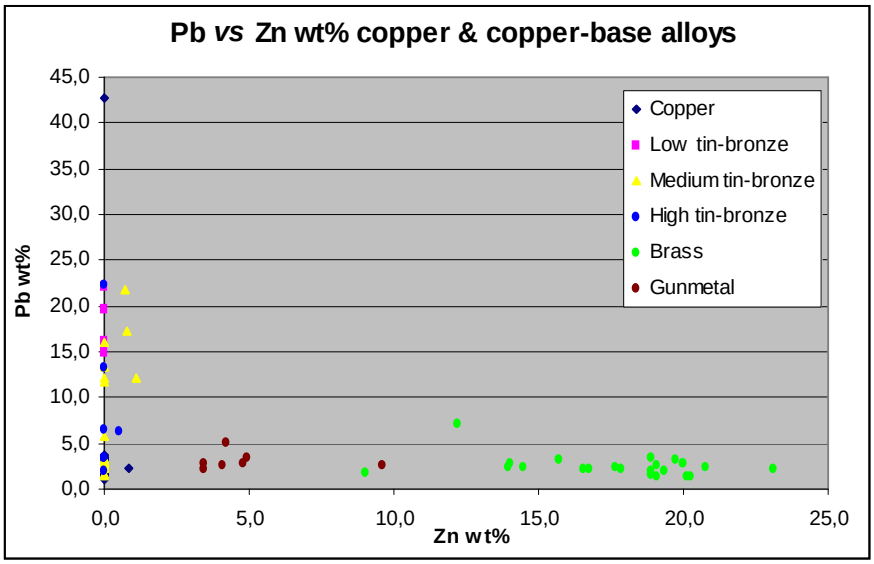


Fig. 35: Scatter plot of the lead wt% *versus* zinc wt% of all copper & copper-base alloys.

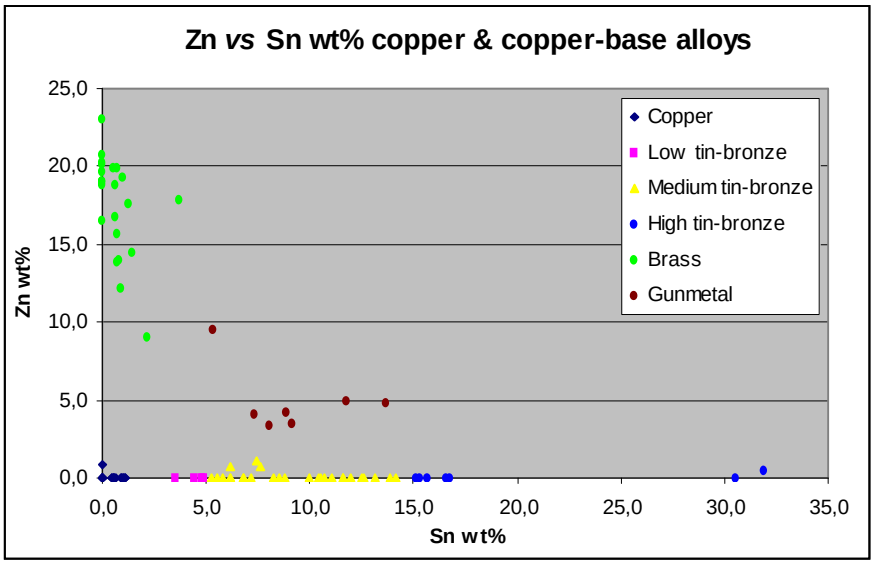


Fig. 36: Scatter plot of the zinc wt% *versus* tin wt% of all copper & copper-base alloys.

Fig. 36 gives the plot of the zinc values in relation to the tin values. Of course the different alloy groups are clearly separated. A clear cluster appears in the brasses around the 20 wt% of zinc. The medium tin-bronzes tend to be split up in two groups with more and less than 10 wt% of tin. The same can be said about the high tin-bronzes. Two samples fall well out of the range of the other high tin-bronzes and are to be considered 'true' high tin-bronzes.

The complete EDX dataset of all analyses of the copper and copper-base alloys is to be found in *Appendix 7.*

5.6.3. Objects & alloys

• Introduction

At ed-Dur, 187 copper-base-alloy artefacts have been registered by the Belgian team. A total of 94 of the samples analysed are copper-base alloys, but not all originated from the Belgian excavations. Six more samples from Khor Rori were also looked at.

This section has to be seen as the culmination of the information obtained by optical microscopy and the SEM-EDX results, presented above. This is brought into connection with the archaeological information on the artefacts kindly provided by A. De Waele. I would like to refer to her coming PhD for more details and references and stress that the contextual information is not the fruit of my labour. Here only the samples of identifiable objects are treated and where possible a drawing of the object is given. Where possible some of the samples are also mentioned in connection to one of the artefact groups. A line indicated by the letter 's' indicates the analysed surfaces.

• Mirrors

Four fragments were tentatively described as 'mirrors': AW 083, BQ 016, M 038 and S 0023 (Fig. 37). S 0023 was as surface find from the first survey at ed-Dur in 1986. AW 083 came from a tomb excavated by the British team that worked on the site. BQ 016 and M 038 came from the occupation level below the surface.

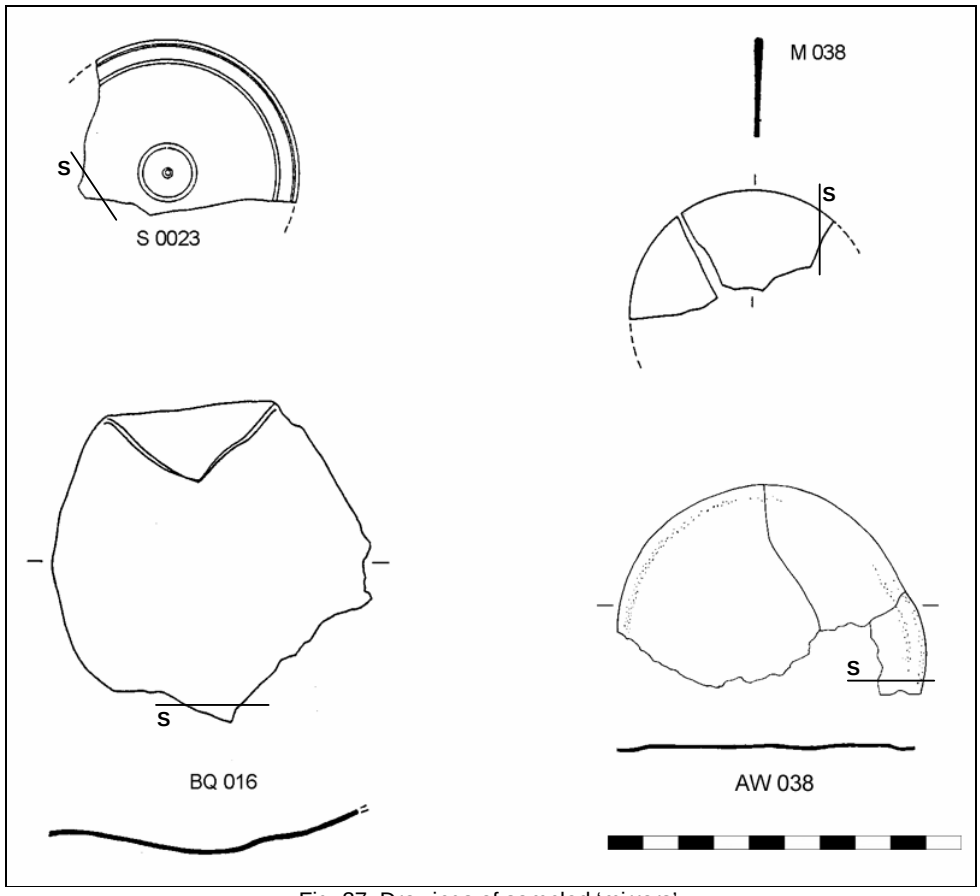


Fig. 37: Drawings of sampled 'mirrors'.

Two of the objects were unfortunately too severely corroded to give any reliable result about their composition (AW 038 & M 038). All what can be said is that these two objects certainly

are made of tin-bronze. Their shape and size does however still suggest that they can be the remains of mirrors.

The microstructure and to a certain extent the composition of S 0023 is very suggestive towards the interpretation of a mirror. Also the shape and design can underpin that. The microstructure is made up of a α+δ-eutectoid copper-tin alloy, a feature seen in Roman and Chinese (Han Dynasty) mirrors from approximately the same date. Roman mirror are however often leaded (5-10%) to improve the casting conditions since it reduces the melting point and increases fluidity during casting[235]. S 0023 on the contrary is not leaded although lead globules are seen in the microstructure.

Fig. 38: Roman mirror (1st – 3rd c AD).

Fig. 39: Chinese 1st c AD mirror (from Castello, 2005: Fig. 2).

Further research towards the typology of undecorated Roman and Chinese might give more clues towards the origin of this mirror, but this is not part of this PhD. Lead isotope analyses for the mirrors may also resolve the problem. Unfortunately no sample of the mirrors was included in the dataset for the LIA[236]. A quick search on the internet (Fig. 38 & Fig. 39) did result in a comparable example from Roman origin[237] and a one from Chinese origin[238]. A more accurate analysis of the composition may also give a clue since Chinese mirror on average contain somewhat higher amount of tin (between 18-23% *vs* 22-26%[239]). An additional AAS analyses only gave a tin value of 23,1 wt% of tin. This value unfortunately neatly falls in the overlap region of the tin amount of Roman and Chinese mirrors. By consequence it does not provide any argument for or against one or the other origins. Roman mirrors are also much thinner than the Chinese and often have drilled or turned decorative patterns on them. The, sometimes massive, Chinese mirrors on the other hand often have fine cast-in decorations. Fig. 39 shows however that this is not always the case and that more simple decorated Chinese mirrors are also known. S 0023 is a thin fragment (as is BR 1041 C) with circle pattern. It would be tempting to allocate this mirror to a Roman origin in the light of the many Roman objects found at ed-Dur. Caution has to be taken however, since a Chinese origin cannot be excluded, considering the global trading network and the many contacts between the West and the East that existed during the 1st c AD.

[235] Meeks, 1993b: 264.
[236] A useful article in that respect would be Mabuchi, Hirao & Nishida, 1985.
[237] www.dcancientart.com/prodimages/low/RB703.gif, *ca*. 1st – 3rd c AD. This only serves as an illustration and is not to be considered of any scientific value.
[238] Costello, 2005.
[239] Meeks, 1993b: 253.

The flat fragment BR 1041 C has an identical microstructure as S 0023 and is almost certainly also a fragment of a mirror. The lead contents is somewhat higher and that fits the information on Roman and Chinese mirrors better. A sample analysed by L. Weeks and described as a flat fragment (sN 152) has a composition of 25,5 wt% tin and 4,3 wt% of lead and could tentatively be classified as a mirror fragment.

Many Roman and Chinese mirrors have a black surface. This was not originally intended but is the result of post-burial corrosion processes. A small fragment of S 0023 was cleaned in lemon juice to remove some of the corrosion products present at the surface. Underneath a black tin oxide layer was found (Fig. 21, p. 174).

BQ 016 has a very different in composition since it only contains a low amount of tin (*ca*. 6 wt%). If a mirror (and this is a big 'if') than it is from a total different type than S 0023. This is possible of course, since next to the high tin-bronze mirrors a 'cheaper' variant is known from the Roman world, i.e. a low or medium tin-bronze mirror that was finished with a tin layer at the surface. This layer however only rarely survives the effect of corrosion and also here no tin-enriched layer was observed. The alternative would be that this rather large fragment is part of a vessel or some kind of plating. The vessel-possibility may be rather unlikely since the vessel fragments analyses in this study have a higher tin content and the overall shape of BQ 016 does also not suggest that it is the remain of a vessel. In any case this fragment was annealed and afterwards heavily worked as evidenced by the numerous strain lines and deformed annealing twins.

- ***Patera* (ram's head, AV 005)**

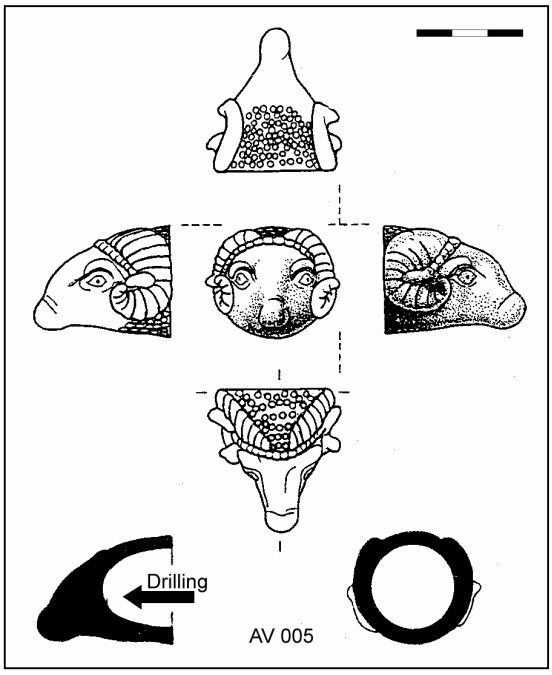


Fig. 40: Drawing of *patera* AV 005.

AV 005 was unearthed in the large tomb with rich grave goods G 5156 (area AV). It is a *patera*, which is a bowl or pan with a long, round, frequently ribbed handle and often with a central navel or *omphalos*. Many times the handle is decorated with a ram's head. These vessels are known from the Hellenistic world, but they became widespread in the Roman Empire during the 1st c AD. *Paterae* were used for cooking, serving food or as tableware, but they would also have been used for religious activities.[240] It is unquestionable that this object is of Roman/Mediterranean origin. The head seems to have broken-off at the onset of the handle.

The analysis of this object was done on drillings obtained from the inside of the head as indicated on the drawing (Fig. 40), so no microstructural information could be obtained. The ram's head is made from a medium tin-bronze (7,6 wt% Sn) and is heavily leaded (17,3 wt% by ICP-MS). A small amount of zinc was also detected. This is interesting to notice in connection to the pedestal of a statuette (see below, M 007), which also contains some zinc and is most probably also Roman/Mediterranean. The tin level falls nicely within the expected values of Roman bronzes, which seemed to have preferred a tin level of around 10% or a little less, which is of course a rough rule of thumb.

[240] Kozloff, 1981: 183-184 (provided by A. De Waele).

Fig. 41: Example of a 1st c AD Roman *patera* handle from Exeter (U.K.)[241].

The amount of lead would have made this object almost impossible to work and obviously it was cast directly to its final shape. The amount of lead present might fit the description of the alloy known as *caldarium* that contained one part of lead and four parts of copper or bronze, i.e. a 20% leaded alloy. It is described as being very white in colour and useful for castings. The colour of the alloy is an interesting feature, and suggests a primary aesthetic consideration in the choice of alloy. The colour would have been a surface effect caused by *inverse segregation* of the lead or lead/tin-rich phase where the low melting point phase is squeezed to the surface of the object during solidification as a "lead sweat".[242]

- **Pedestal statuette (M 007) & female appliqué (S 0020)**

The pedestal for a statuette (M 007) was found in the 'temple' of ed-Dur. On this supposed 'Roman' pedestal very little information is available. No typology exists on this type of objects and only two similar pedestals are reported by A. De Waele, one from Syria and one from Austria both from Roman contexts. This does not contradict the supposed Roman origin of this object. It is interesting to notice that M 007 was found inside the temple, together with two more 'Roman' objects, i.e. another pedestal (M 015) and an oil lamp (M 014).[243]

The female head appliqué (S 0020) was a surface find done during the first survey of ed-Dur in 1986. The attachment in the form of a female head (S 0020) is Hellenistic in style, but the workmanship is crude. It is unknown to which object this appliqué was attached.[244] A similar head was found at Mleiha and dated to the PIR B. This object was analysed at the time and was reported as being a leaded tin-bronze of which the fraction of lead was greater than that of tin[245]. This is not contradicted by the analyses done here, where S 0020 *ca*. 15 wt% of tin and 22,4 wt% of lead (by ICP-MS).

Both objects were sampled by drilling and the EDX analyses were done on those drillings. The compositional results for M 007 are close to that of the *patena* discussed above, with *ca.* 6,2 wt% of tin and 21,7 wt% (by ICP-MS) of lead. The composition seems to underpin the possible Roman origin of this object. The high lead content would have given these objects a white/grey colour.

Two more fragments that could not be related to a certain object can be mentioned in this context. sBR 1157 A (4,9 wt% Sn & *ca*. 15 wt% Pb) and BS 154 (3,6 wt% Sn & *ca*. 19,8 wt% Pb) are both heavily leaded and have a low tin value. Both fragments were in an as-cast state, which fits the 'impossibility' to work such a highly leaded alloy.

[241] http://www.exeter.gov.uk/timetrail/02_romanfortress/object_detail.asp?photoref=2_44
[242] Craddock, 1979: 75; Ponting, 1999: 1317.
[243] A. De Waele pers. comm.
[244] A. De Waele pers. comm.
[245] Mouton 1992: 76, footnote 214.

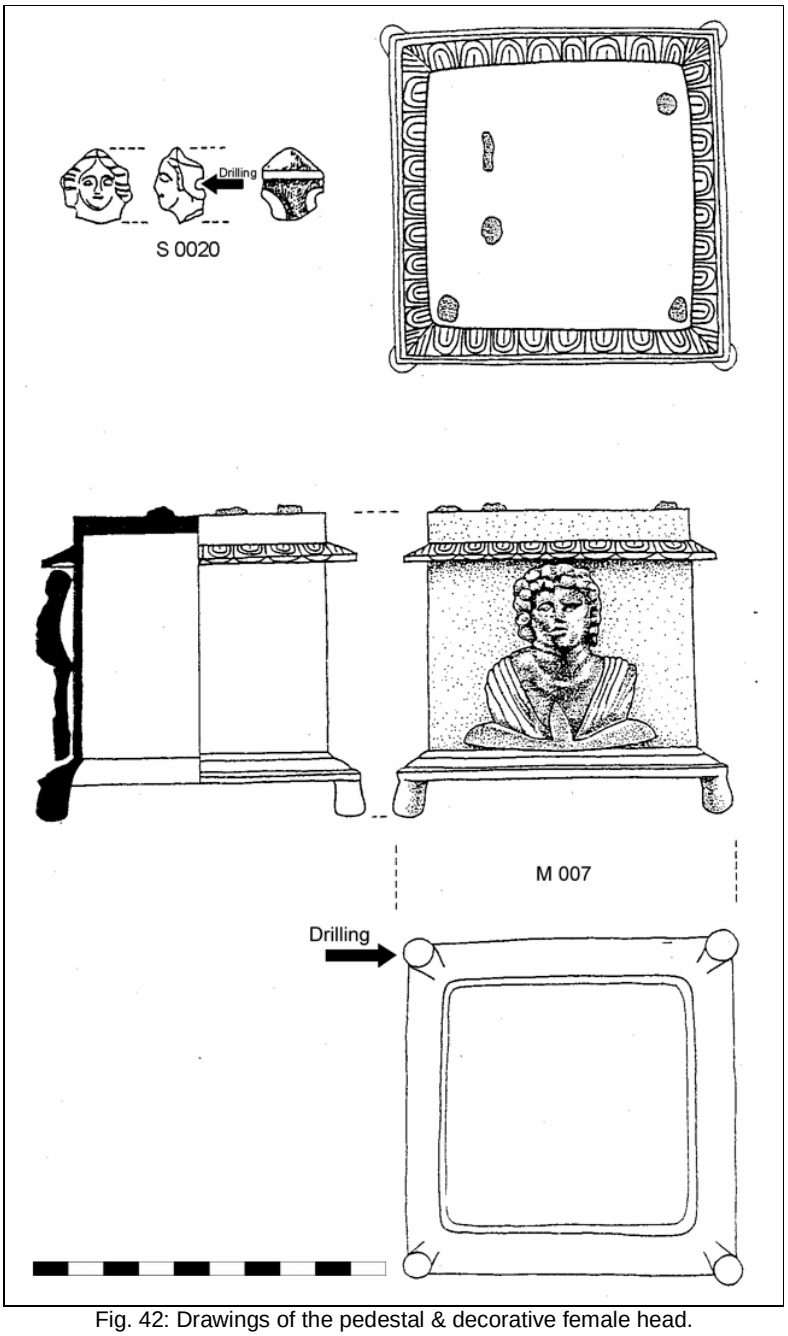


Fig. 42: Drawings of the pedestal & decorative female head.

- **The 'wine set'**

The samples from objects analysed related to the 'wine sets' are: AV 007 (fragment sieve), AV 104 (horse spout), AV 115 (part of the ladle), K 005 (horse head-ending of ladle) and S 0021 (possible part of a handle of a ladle)

S 0021 had no coordinates and K 005 was excavated by the British team in Tomb 922 in area K (no further details available). AV 104 and AV 115 were found inside Tomb 5156 in area AV whereas AV 007 was unearthed at the entrance of the same tomb. Next to the Belgian excavators the Danish also found a similar wine set.

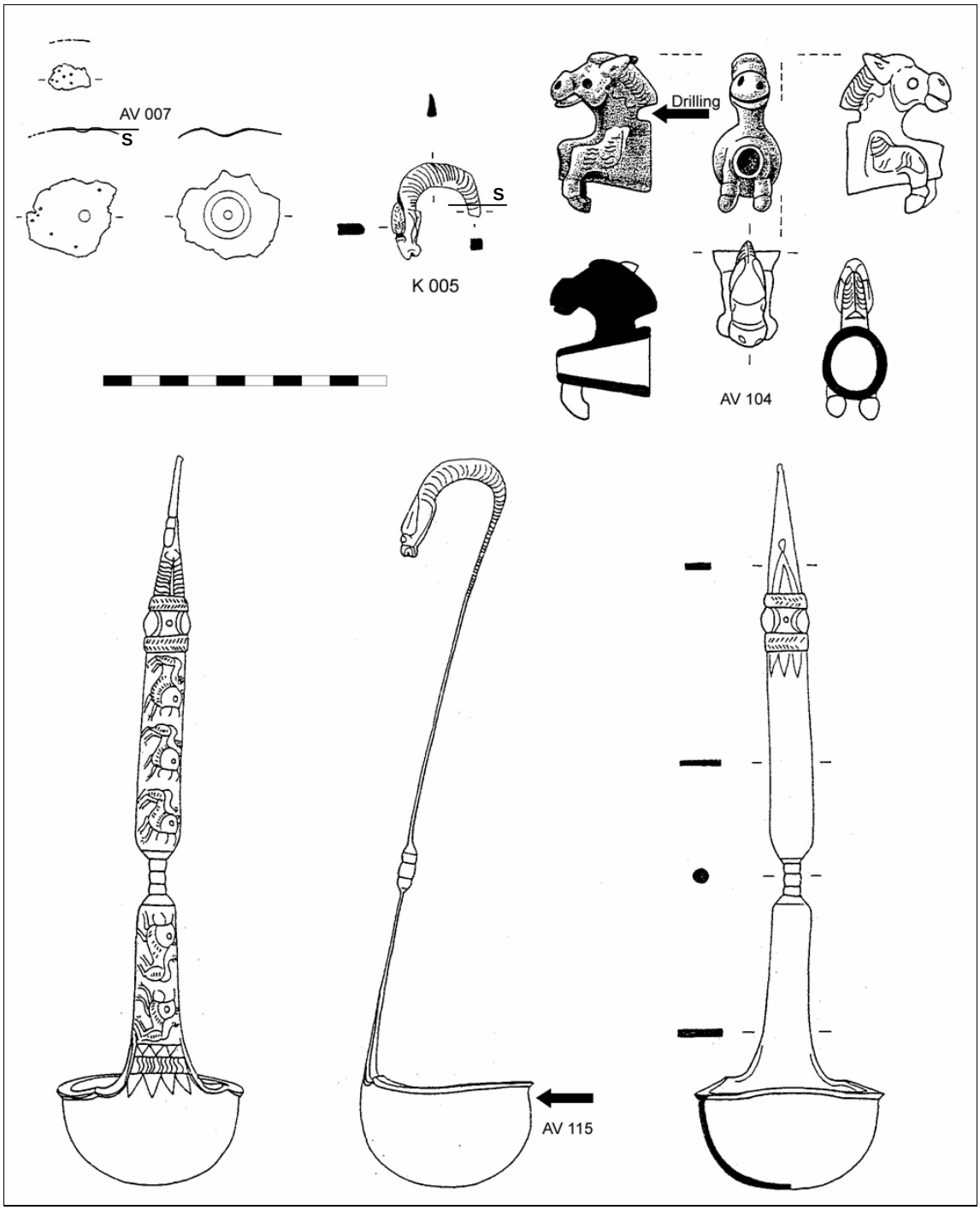


Fig. 43: Drawings of sampled parts of the 'wine set'.

In the ancient Near East, drinking and banquet scenes are already known from the prehistoric period. During the Assyrian and Achaemenid period, drinking sets became popular and they were used during banquets and other activities. They also had a great success both during the Graeco-Roman and Seleucid-Parthian periods, with changes in the individual objects as well as in the composition of the sets. Parts of drinking sets have been discovered in the Arabian Peninsula (particularly in the Oman Peninsula), but apart from these objects not much is known about their function and use during this late pre-Islamic period. Were they used in daily life or was their use rather religious (e.g. banquet in honour of the gods, libations, etc.) or funerary (e.g. funerary meal, for the afterlife, etc.)?

Regarding the animals portrayed on the wine sets, the horse appears to be most prominently present. In antiquity, animals were often used to represent or symbolize deities. In SE-Arabia, the very important sun god Shamash would have been connected with the horse. The fact that artefacts which look very much like the ed-Dur wine set elements (especially the spouts) are almost exclusively found in the Oman Peninsula, could point to local/regional manufacture.[246]

AV 007, AV 115, K 005 and S 0021 are all unleaded medium tin-bronzes. The two ladle fragments (AV 115 & S 0021) have a similar tin content, respectively 6,2 and 6,8 wt%. The horse head-shaped element K 005, has a higher tin level (13,9 wt%), which might suggest it was not part of a ladle. The sieve fragment AV 007 has a tin level of 12,5 wt%. This is in the same region as two other vessels analysed. All have straight annealing twins (microstructure of S 0021 was not evaluated) and this indicated that the objects were finished by an annealing phase after being worked.

AV 104 is the only object that stands out if the composition is considered. It is a leaded (22,2 wt% by ICP-MS) low tin-bronze (4,7 wt%). It was definitely cast to shape and not further worked. The high lead content is similar to the 'Roman' alloys mentioned above, but the tin value is slightly lower. The tin present would not have altered the colour of the metal and must be seen as residual from recycling or present to lower the melting point of the alloy. The lead however would have given the spout a white/grey appearance. This is interesting since a similar spout from SE-Arabia was reported of being from silver.

- **Vessels**

At ed-Dur, six copper-base alloy vessels were excavated by the Belgian team. They are all bowls with a diameter larger than the height and quite simple forms. AV 055 and AV 056 come from the large grave with rich burials gifts G 5156 (area AV) in which also the wine set elements were found. BS 054 and K 149 were also found in tomb contexts. The function of these specific bowls remains unknown, but copper-base alloy vessels appear to be far less common than ceramic vessels and with the eye on their presence in the rather rich graves G 5156 and G 3840 they were possibly only affordable by the more wealthy inhabitants. However, we have to keep in mind the severe plundering of the ed-Dur tombs by which a lot of metal vessels probably disappeared. Some vessel fragments were also listed. Few similar copper-alloy bowls have been found in the Arabian Peninsula.

BS 054 is a vessel made of gunmetal (*ca*. 9,6 wt% Zn *&* *ca*. 5,4 wt% Sn). The composition of this gunmetal is atypical when compared to the other gunmetal attested, in that the fraction of zinc is larger than that of tin. For the other gunmetals at ed-Dur this is just the other way around. The composition fits the data for gunmetal published from Roman contexts. The vessel was most probably produced by hammering a cast sheet into the shape of the vessel. The deformation might have been quite severe and pores in many strain lines are still visible in the microstructure. The lead inclusions are spread out in the line of working. A final annealing phase finished the production process and the annealing twins are straight. A cramp found together with BS 054 indicates that this metal vessel was repaired and thus must have had a certain intrinsic value.

The flat fragment sBS 1493 most probably is also a fragment of a vessel. The composition is however different from that of BS 054 and the alloy contains *ca*. 13,7 wt% of tin and only *ca*. 5 wt% of zinc. The microstructure as a whole is however similar to that of BS 054 and the production procedure must have been similar as well, i.e. the casting of a sheet that was hammered into shape (in or over a mould) in several steps with annealing in-between. The tin value of sBS 1493 is similar to that of the vessels K 149 and K 153. This may suggest that the zinc was unintentionally added to the alloy.

[246] A. De Waele pers. comm.

The two fragments that were taken from vessels K 149 and K 153, both were made of medium tin-bronze (respectively *ca*. 14 & 13 wt% Sn). Lead was not attested in significant amounts, what is to be expected for a metal that has to be worked into shape, i.e. a vessel. The microstructure revealed rather large grains and straight annealing twins. This must mean that the vessels were properly annealed at the end of the working to remove the stress in the metal.

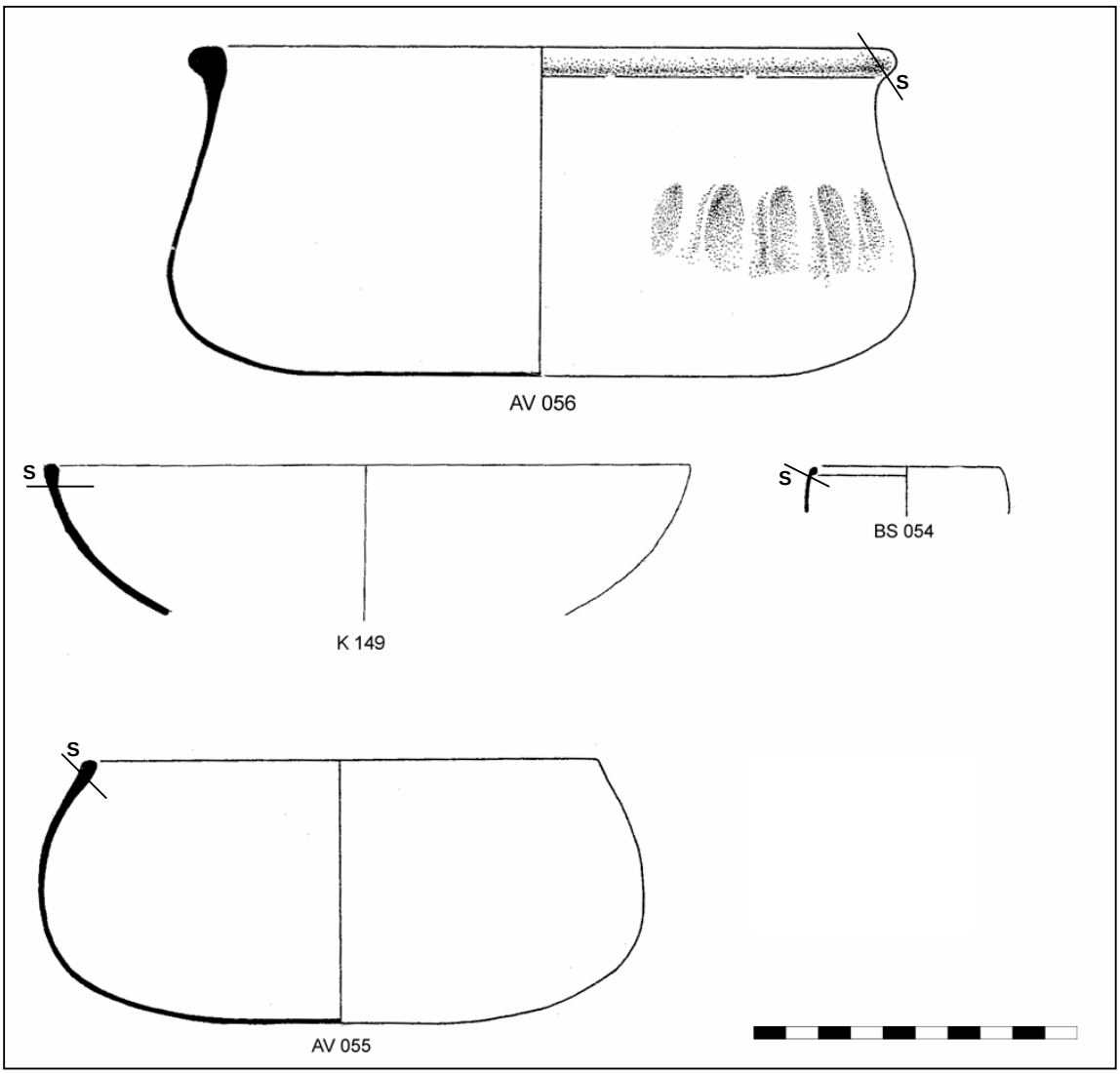


Fig. 44: Drawings of some sampled vessels.

A fragment of the bowls AV 055 and AV 056 was analysed, but the metal was too corroded to give any reliable results. They were made of a tin-bronze however, since large amounts of tin were detected. The detected fraction is biased however by the corrosion.

Several flat fragments (sBK 1238 A, sBQ 1058 A & sBS 1276) were attested amongst the samples and they can be tentatively linked to vessels, although this is with the necessary precaution. sBK 1238 A was severely corroded, although islands of uncorroded metal survived. These contained around 16,6 wt% of tin. Although this fragment is already in the high tin-bronze category it has straight annealing twins, indicative of working and final annealing. The annealing twins are however more numerous and may whiteness several annealing phases. In broad lines it fits the observations and composition of the vessels K 149

and K 153. Both other fragments (sBQ 1058 A & sBS 1276) had a similar amount of tin between 5 and 6 wt% of tin, i.e. a medium bronze. They have an as-cast structure and were possibly annealed after casting. If these two fragments are parts of vessels, then two different alloy types are attested. The fragments that definitely came from vessel have a rather high tin value (between 13-16 wt% Sn), a feature probably related to the colour this type of alloy has. Two other samples fall in the lower tin region of a medium tin-bronze and were in the as-cast state, indicating that sheets were cast.

- **Ornaments**

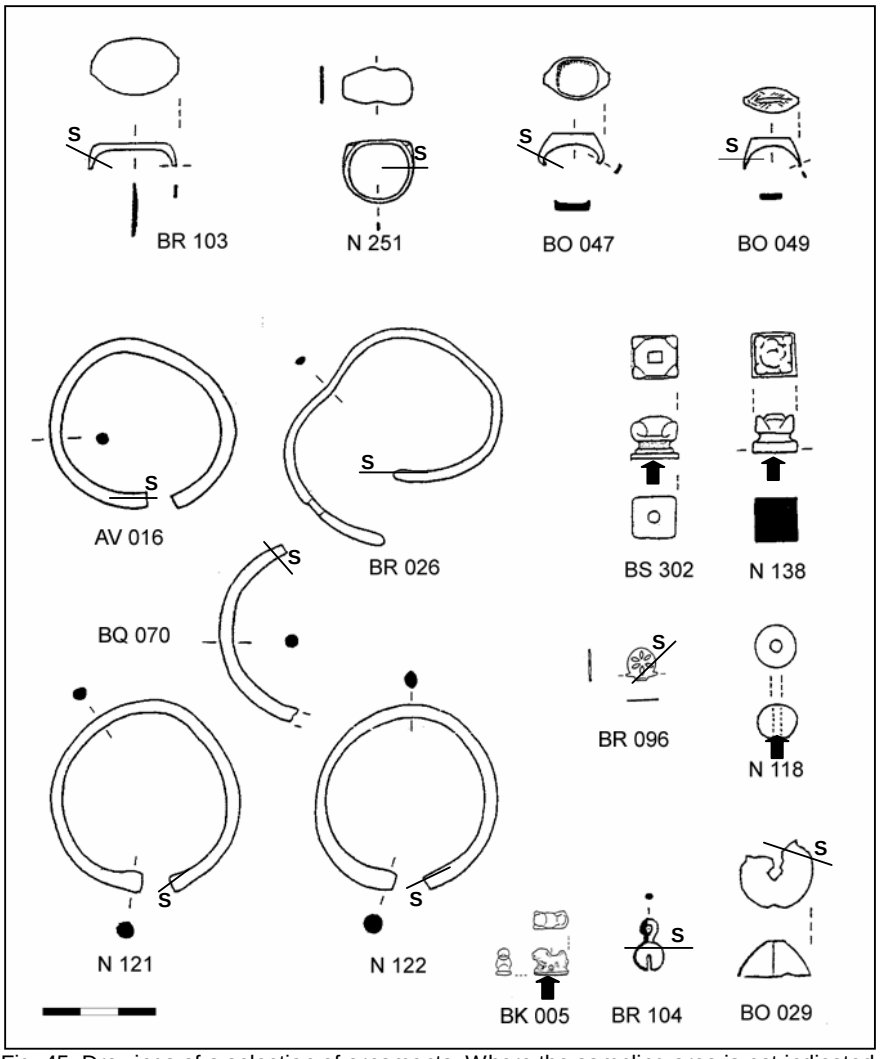


Fig. 45: Drawings of a selection of ornaments. Where the sampling area is not indicated part of the object was polished and analysed (indicated by black arrows).

Rings

At ed-Dur 26 finger-/toe-/earrings were unearthed[247]. Thirteen of these are simple objects with a round cross-section and open ends. Eleven are *bezel-rings* (flat, engraved, or with inset). Such finger-/toe-/earrings were quite widespread and examples are known from the U.A.E., Bahrain, Oman, Failaka, the Parthian and Sasanian Empire and the Roman world. Bezel-rings had a long history in the ancient Near East. Because of that these rings are not

[247] For the Belgian team: 12 from graves, 3 from the walking area and 7 were surface finds.

very diagnostic and difficult to compare typologically, since they are fairly simple objects and widespread.

Five rings were subjected to chemical analyses: BO 047, BO 049, BR 103, F 326, and N 251. BO 047, BO 049 and BR 103 were found on an excavation dump and have no archaeological context. F 326 cam from an occupational layer outside the large building in area F excavated by the French. N 251 came from a burial (G 3847). All these rings were made of brass. Three of the rings (BO 047, BO 049 & N 251) are very similar in composition, with a high zinc content (between ca. *19* wt%). This is rather indicative of primary 'fresh' brass was somewhat diluted or that went through a limited number of remelting cycles. The composition might suggest that these rings were produced and exported as a finished object and that they might be Roman/Mediterranean in origin. D. Dungworth mentions Roman finger-rings from N-Britain that were made of brass[248]. F 326 contains a somewhat lower amount of zinc (16,7 wt%). All these rings have gone to a phase of working and subsequent annealing, with (some) additional cold working as the final step.

The last ring (BR 103) is completely different in composition. This object is made of leaded brass (12 wt% of Zn, 1% wt of Sn & 7 wt% of Pb). It is the only leaded brass that was attested. The microstructure is also very different from the other rings since it is in the as-cast condition with coring. The high lead level would have eased the casting, but make the final object difficult to work. Some strain lines might be observed (although I am not sure) and could be related to the deformation to give the the ring its final shape. As a whole the alloy and microstructure fit the casting of a ring with possibly a small amount of cold deformation.

The French excavators tentatively described F 128 as an earring. This object was made of gunmetal (*ca*. 7 wt% Sn, *ca*. 4 wt% Zn and some lead). The straight annealing twins point to proper annealing as the final phase of working. The lead inclusions are elongated in the line of working.

Bracelets & anklets

Sixteen bracelets/anklets were registered. Fourteen of them came from graves (nine from children's graves). Eight of these are simple objects with a round cross-section and open ends. N 121 and N 122 have thickened ends. Bracelets/anklets were quite widespread and they were found at Failaka, Bahrain, the Parthian and Sasanian Empire, the Graeco-Roman world and India.[249] In this study five bracelets (AD 031, AV 016, BQ 070, BR 026 & S 0022) and two anklets (N 121 & N 122) were analysed.

AD 031 apparently came from an occupational layer (no further detail available). AV 016 was unearthed in a tomb (G 5158), BQ 070 was found at the occupational layer beneath the surface and BR 026 was registered near an altar with oyster shells. S 0022 is an orphan without any context. N 121 and N 122 both came from a tomb (G 3840).

Four bracelets are made of medium tin-bronze (AD 031, AV 016, BQ 070 & BR 026) with respective tin levels of: 12,6 wt%, 10,6 wt%, 7,2 wt% and 11,9 wt%. Except for BQ 070 maybe, the composition is very consistent. All were worked and afterwards annealed. This was however not the final step and additional cold working to some extent was done after the annealing.

The bracelet S 0022 is made of gunmetal (*ca*. 12 wt% Sn & *ca*. 4 wt% of Zn). This object was not cast to its final shape and received (repeated?) cycles of working (creating small grains). The final stage was one of annealing since the annealing twins are not deformed. It should be mentioned that although S 0022 is from gunmetal, the tin level is consistent with the other

[248] Dungworth, 1997b: 909.
[249] A. De Waele pers. comm.

bracelets analysed and may indicate that the zinc entered the alloy unintentionally in the form of a small amount of scrap brass.

Both anklets (N 121 & N 122) are from a medium bronze with an identical tin level (i.e. 11,6 wt%). This is in good accordance with the alloy used for the bracelets. The microstructure however shows a difference in treatment for both objects. N 122 shows evidence of mild working after casting and is partly annealed (annealing twins appear, but also the remains of the cored structure). The anklet N 122 on the other hand shows evidence of working (strain lines and elongated lead inclusions) and final annealing (twins). No coring is present.

Round bead

One quite simple necklace of the 'torque' type with a round cross-section and open twisted ends was found in a child tomb (G 3840). The round bead N 118 came from the same context. This bead is heavily leaded (*ca*. 21 wt% by EDX) medium tin-bronze (*ca*. 7 wt% of Sn). A lead enriched layer can be noticed at the rim of the polished section. The high fraction of lead detected is thus due to segregation upon cooling, a process known as *lead sweat*. This would have given the bead a grey appearance.

Bells

Nine small bells were found at ed-Dur. Six are very alike and spherical in shape (e.g. BR 104, from an excavation dump), while the other three bells are rather conical (e.g. BO 029, from an occupational layer beneath the surface). In the ancient Near East both men and animals often wore metal bells as protective amulets against evil. They were part of jewellery or were sewn on garments. During the 1st millennium AD, bells were popular as grave gifts everywhere in the Near and Middle East and are found in Nabataean contexts, the Parthian Empire and Roman Syria. They are also attested in Christian and Jewish burials.[250] This strong link to the Near and Middle East can be indicative for a local production, for local needs.

BR 104 is the only leaded (*ca.* 5 wt% of Pb*)* gunmetal (*ca*. 9 wt% of Sn & *ca*. 4 wt% of Zn) object. Except for the lead level it fits the composition of the earring (F 128) very well. The presence of a larger amount of lead is probably to ease the casting of this small object. No indication of any working after casting is seen (no annealing twins), but equi-axed grains are present so the small bell was annealed after casting. The grains are rather small (< 50 µm) and the remains of coring are still visible, indicating a short annealing phase at low temperatures. It is interesting to notice that the lead is very unevenly distributed and a large concentration is seen near the outer surface of the bell. Upon cooling after the cast the lead was expulsed from the metal ('lead sweat'). The presence of lead on the surface would have given this object a grey appearance. The fact that the composition of the gunmetal does not really correspond to Roman gunmetal might indicate that the alloy was made elsewhere. Something that would fit the more Near and Middle Eastern character of this bell pendant.

BO 029 is an example of the more conical shaped bells. It was most probably provided with a (iron) clapper, as seen on other examples of this type of bell. It was made of a 19 wt% zinc brass. The metal was at least ones annealed, but not sufficiently to remove the strain lines.

Lion beads

Three (BQ 156, BS 141 and N 311) additional examples of small lion bead BK 005 (no context) were found at ed-Dur. Similar beads are known from India where the powerful lion was one of the most favourite animals for the manufacturing of amulet. The lion appears to have been popular between the 1st c BC and 1st c AD and these small amulets might originate from the Indian subcontinent.[251] BK 005 is the only object made of heavily leaded

[250] A. De Waele pers. comm.
[251] Information kindly provided by A. De Waele

copper. The high amount of lead present in this alloy probably served to increases the liquidity of molten copper. To preserve the detail in such small cast objects a very liquid melt is needed. The fast cooling and the small volume of the object may have resulted in an unequal distribution of the lead in the metal. Possibly the lead level measured here is too high (*ca*. 42 wt%) because a lead rich zone was analysed. Small amounts of silver, tin, iron and nickel were seen in the spectrum of this object.

Altar beads

Two altar-shaped beads (BS 302 and N 138) were analysed. BS 302 was found on an excavation dump and N 138 came from tomb G 3840. They slightly resemble the faience altar-shaped beads (e.g. found at Dura Europos), and according to E. Haerinck these artefacts resemble Hellenistic altars.[252] No exact parallels are known however for these metal examples.

The altar bead BS 302 was cast of unalloyed copper (some silver present) and was apparently gilded afterwards. Four basic techniques can be used to gild a metal artefact[253]:

- Covering the object with gold foil and attaching it *mechanically* by rivets, etc.
- Gold foil can be hammered out to a leaf, this thin leaf can then be attached by an *adhesive* (e.g. egg-white).
- Silver artefacts were often gilded by *diffusion bonding*, whereby gold foil was burnished onto a hot surface. The inter-diffusion of gold and silver results in a strong metallurgical bond. This technique is not well suited for a copper(-base alloy) substrate, since surface oxidation caused by the heating, prevents an efficient bonding.
- *Fire*, *amalgam* or *mercury gilding* are different terms for the same technique. It was developed during the 3rd c BC in China and the 1st c BC in Europe as an alternative methode that was also applicable to copper and copper-base alloy objects. By the 2nd – 3rd c AD it was the predominant technique for gilding metals in Europe and the Middle East. The basic concept of this method is to grind-up gold leaf in mercury, creating a paste of gold amalgam. This paste is then applied to the metal substrate and heated to 250-300°C. A large part of the mercury evaporate an d the gold is left on the surface. The object was then burnished to compress the porous structure and create a smooth and brilliant surface. An alternative method is to first apply the mercury and then cover it with gold leaf. When this is heated an *in situ* amalgam is formed.

The way of gilding was not researched to the fullest, but amalgam gilding would be a good candidate. The EDX-spectrum of mercury and gold shows severe overlap and it seems impossible to distinguish between them if both are present. The EDX software did not automatically detect mercury, but when manually added the software calculated a fraction of 6,6 wt% of mercury. A residual mercury value of between 8-25 % can be expected after *fire gilding*[254], although much lower values were reported for the *mercury silvered* 3rd c AD Roman coins analysed by Valouch *et. al.*[255]

A clear as-cast structure is absence. This is not abnormal since an as-cast structure can be difficult to resolve in a pure metal. Still the remains of coring can be observed. Moreover if BS 302 was fire gilded, then the metal went through a short heating phase (*ca*. 15 min at about 250°C), which would have removed part of the as-cast structure. Some lead and Cu-O inclusions are found at the grain boundaries. Copper oxides are easily formed during the casting or heating of copper and thus to be expected.

A similar altar bead, N 138, had no evidence of gilding, but was also made of unalloyed copper (*ca*. 1 wt% of Sn present). It is to be remarked that both beads are *not leaded*,

[252] Personal communication with E. Haerinck & A. De Waele.
[253] Anheuser, 1997a: 58-59.
[254] Anheuser, 1997a: 58.
[255] Vlachou, McDonnell & Janaway, 2002: II9.2.3 - http://www.mrs.org/publications/epubs/proceedings/fall2001/ii/.

something that might be expected in the light of the other objects. On the other hand it has to be noted that the gilding of leaded alloys poses severe problems[256]. So for BS 302 the absence of lead can to be explained in this way.

Decorative element

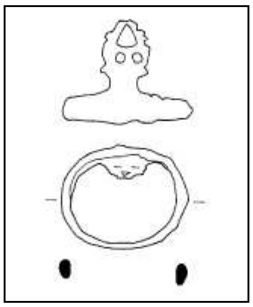


Fig. 46: Silver ring (∅: 1,8 cm).

BR 096 is a small floral decorative element and came from tomb G 6319. It is broken of on one side and thus was attached to something else. A tentative interpretation would be that it was part of a ring. A silver ring with decorative elements was found in a Tylos period grave at Bahrain[257] (Fig. 46). Fragment BR 096 might have been attached to a ring in a similar way.

The fragment is made of brass (*ca*. 20 wt% Zn and some Sn). After working this object was annealed, but strain lines are still visible, indicating that the working might have been quite severe.

• **Dagger elements**

Table 22 lists the fragments that are (or very likely are) parts of daggers. Three are the pommels of what is called a *ring-pommel dagger* (AT 013, AW 063 & BL 014). The chronological, cultural and typological aspect is treated in *Chapter 10*. AW 063 came from the British excavations at ed-Dur and was kindly made available for analysed by C.S. Phillips.

Reg. nr.	Description	Zn wt%	Sn wt%	Extra
AT 013	Ring-pommel of dagger	18,86	2,73	-
AV 079	*Ring-pommel of dagger*	-	-	*Not sampled*
AV 083	Large rivet, with traces of iron blade?	19,33	1,03	-
AW 021-2	Nail shaped fragment guard (~ AW 063)	15,70	0,75	No drawing, see Fig. 25
AW 062	Sheeting/fitting around iron core	20,80	-	-
AW 063-1	Ring-pommel dagger, scabbard sheeting	Copper		Fig. 47-1
AW 063-2	Ring-pommel dagger, guard attached to handle	20,24	-	Fig. 47-2
AW 063-3	Ring-pommel dagger, nail shaped fragment guard	20,13	-	Fig. 47-3
AW 063-4	Ring-pommel of dagger	19,12	-	Fig. 47-4
BL 014	Ring-pommel of dagger	18,92	-	-
sAV 412 E1	Iron fragment with sheeting	19,98	0,70	-

Table 22: Dagger fragments.

To this we can add AW 021-2 (see p. 191 for a picture), a nail-shaped fragment very similar to AW 063-3. The overall shape of AV 083 is also similar, but if this is the guard of a dagger, then the production technique was different. In the first cases the guard was slid over the tang or grip of the iron dagger, whereas in the latter the iron would have been bent around the guard.

[256] Anheuser, 1997a: 61.
[257] Jensen, 2003: 142, Fig. 9.9.

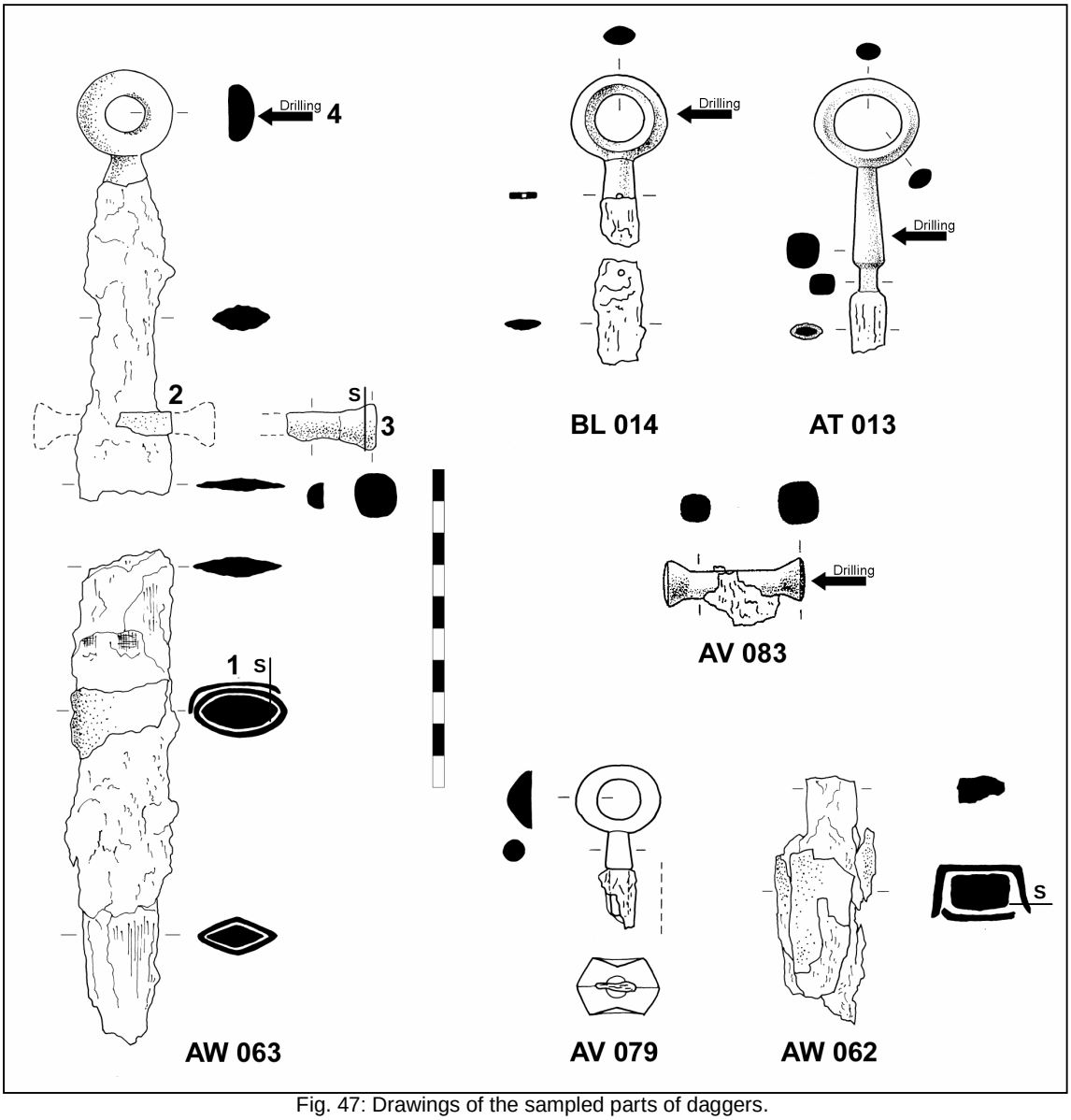


Fig. 47: Drawings of the sampled parts of daggers.

All analyses of the pommels were done on drillings (indicated on Fig. 47), since these objects could not be sampled in any other way. AT 013, AW 063-4 and BL 014 all contain similar amounts of zinc, between 18 and 20 wt%. This is not as much as a 'fresh' Roman cementation brass, which would normally contain 22 to 28% of zinc. The low amounts of tin detected in some of the samples indicates that the brass was at least ones remelted and probably diluted with metal that contained a small amount of tin. The fact that the fraction of zinc is quite uniform in all analysed fragments, can point to an intentional choice for an alloy of about 20% zinc. Nowadays brasses of 15 to 20% zinc are often used for low-priced jewellery and in foil-form as a cheap substitute for gold leaf, since this composition has a golden yellow colour. A multitude of terms is used to refer to these modern brasses. Overall terms for the 15 to 20% spectrum are such as gilding metal or Dutch metal. When a brass is composed of 10 to 20 % of zinc it has a golden yellow colour and after polishing it shines like gold[258]. To this we can add the 'large rivet' AV 083.

[258] Biswas, 1996: 351; Biswas, 2001: 141.

Additionally, the fragment of the guard (AW 063-2) that was still attached and the 'nail-shaped' fragment that was found together with the dagger (AW 063-3), have almost identical composition, so we can safely conclude that they belong to the same guard.

Where the microstructure was observed the metal was always subjected to working and annealing, with sometime a final cold working phase. Single phased (α-)brasses are well suited for cold working.

It is intriguing that almost all brass fragments associated with these daggers have a zinc content that is indicative of at least one remelting phase. The brass used for the objects found at ed-Dur is very likely from a European or Mediterranean origin, but it used for object that clearly originated in the Parthian cultural sphere (seen *Chapter 10*). The composition of this brass would nicely fit the hypothesis that the Parthian World was importing (or receiving in one way or another) fresh brass ingot. Upon remelting the metal part of the zinc was lost and/or the brass was diluted with copper or scrap bronze, producing an alloy with less zinc.

One exception is found in the list. AW 021-2 is a nail-shaped object very similar to the extremities of the guard of dagger AW 063. The zinc content is somewhat lower than in the other samples (15,7 wt%) and is indicative for diluting or remelting.

Two more samples are to be considered here: AW 062 (from the same tomb as AW 063), a kind of sheeting around an iron core and sAV 412 E1 also a piece of sheeting (?) attached to an iron fragment. Both have a zinc level around 20 wt%.

- **Pins, spatulas & needles**

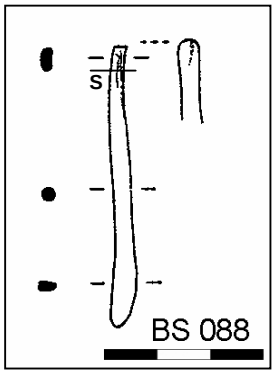


Fig. 48: Drawing of a sampled 'spatula'.

Thirty-seven artefacts can be grouped as 'pins', spatulas and needles. The 'pins' (23) are sticks with a round cross-section, most are more or less pointed and they can be decorated with mouldings. Eleven pins have a hook at one end. The needles (7) are pins with an eye, and the spatulas (4) have a broader, flat tip. The pins and needles could have been used on many different ways, for example as spindles, cloth and hairpins or sewing needles. The spatulas could have been used for stirring (e.g. cosmetics, aromatic substances, medical liquids, etc.).

BS 088 (found on an excavation dump) might have functioned as an applicator for cosmetics (*spatula* or *kohlstick*). It is made from gunmetal (*ca.* 3,5 wt% Zn & *ca*. 7,3 wt% Sn), but has a fairly large fraction of silver present (*ca.* 5,8 wt%). BS 088 was cast to shape and the beginning formation of equi-axed hexagonal grains with coring remnants show that it was not worked (no annealing twins) and annealed for a short period of time at/or moderate temperatures after casting. In the light of the pickling process described in *Chapter 7* this may be related to a process of silver surface enrichment.

It may be worth mentioning object M 019 (see *Chapter 6*, p. 270) in this context. This artefact is described a possibly part of a *kohlstick*, an object used to apply cosmetics. M 019 is basically made of unalloyed silver and bears the remains of a gilded surface. The use of silver and gold point towards the exclusivity of this object. The rather high silver contents in BS 088 can maybe also be explained in this way. The object came from the floor level of the temple.

SX 001 is a flat pin (rectangular section) and came from the house that was excavated in Area C by the Danish team. It is made of a copper (*ca*. 1 wt% of Sn). The microstructure

shows deformed annealing twins and strain lines, so the metal was deformed in the final phase of working and not annealed afterwards.

F 330 (from the occupation level in Area F) is a small needle (diameter *ca.* 1 mm) from brass (*ca*. 14 wt% Zn & 0,8 wt% of Sn). It could also be a pin (e.g. to hold cloths together) and in this case it would have served a more decorative function. The section of the objects exhibits three distinct layers of which the middle shows no signs strain lines or deformed grains. The annealing twins are straight and do show the area was worked but then fully annealed. The lead inclusions are elongated along the line of working. The two outer layer show evidence of extensive cold working in a final stage in the form of deformed annealing twin, strain lines and elongated grains.

Z 012 is a 'high' tin-bronze (*ca*. 15,7 wt% of Sn) needle with an eye. The needle was preserved up to a length of *ca*. 11 cm, but the point was missing. The diameter of the corroded needle was *ca*. 4 mm. The annealing twins show that this object was at least ones annealed after working. A very tentative hypothesis is that this needle was used to repair fishing nets, since it is rather large for other purposes of sewing.

Z 092 is the point of either a nail or a pin/needle (no drawing), but since the section is round and not square and rather pointed an assignment as pin/needle is more likely. The metal it was produced from is unalloyed copper (*ca.* 1 wt% of Ag and some Sn) and the microstructure indicates that the point was made by heavy cold working a rod. The deformation of the inclusions, grains and annealing twins is only seen at near the surface and not in the centre of the section. At least one annealing phase went on prior to the final shaping of the object. Cracks and elongated porosities in the line of working, near the surface, may be the result of 'over working' the metal.

One pin analysed by L. Weeks was also made from unalloyed copper.

- **Nails & rivets**

At ed-Dur, 13 copper-base alloy nails were excavated. They have different forms ranging from small nails with circular cross sections to large nails with square profiles. Three nails have large disc-shaped heads with a 'decoration'. The presence of these objects is quite interesting since they probably are the witnesses of furniture made in perishable materials. They were found in graves, on the walking area, on the (sub)surface and on dumps.

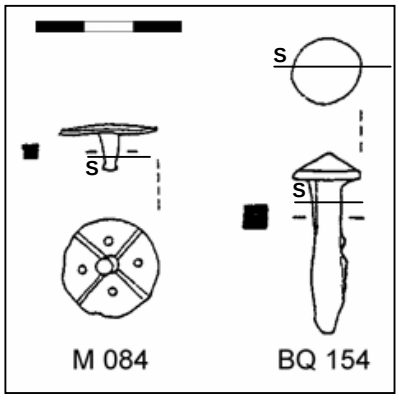


Fig. 49: Drawings of some sampled nails.

The 'decoration' mentioned in connection with the unalloyed copper nail (*ca*. 0,6 wt% of Sn & some lead) M 084 needs some additional discussion. The lines and dots on M 084 are actually not 'decorations', since they are at the bottom side of the nail head and would be invisible when the nail is hammered into something. The lines and dots are also in relief and not punched. The head was obviously flattened when this nail was hammered in.

A first possibility would be that the 'decorations' were already present from the cast, meaning that it was integrated in the casting mould. The 'decoration' would serve as extra 'point of attachment', but the relief is rather shallow for that. Probably the 'decorations' would have been more deformed in that case and would not run to de edge of the nail head. They do however not seem to be deformed and do run till the edge.

A second possibility is that they are the remains of the use of a mould in four parts, with the ridges being molten metal that ran in-between the four parts. The shape of these ridges is however to regular and the dots are not explained in this way.

A last explanation would be that the underside of the nail head took the shape of a decoration already present in the substrate it was hammered into. In this case the original decoration was incised and the copper took the positive mirror of this. The microstructure of the square shaft of this nail indicates that the metal was worked after casting (annealing twins present and large grains). There was no deformation of the annealing twins in the shaft of the nail so the substrate this nail was hammered into, was probably quite soft (e.g. wood?). This seems to me the best explanation.

BQ 154 (surface find) is also of unalloyed copper with a small amount of tin and lead present (*ca*. 0,6 wt% of Sn). The shaft of the nail was cast to shape, worked and annealed. Additional deformation in the annealing twin can be noticed, possible relate to deformation when the nail was hammered-in. The head of the nail was however shaped after the casting as evidenced by the heavily distorted microstructure, especially at the transition of the shaft and the nail head.

The use of the 'softest' metal to produce nails might at first sight seem illogic, since it could be hypnotized that a harder alloy would be more useful. This however might be a misconception since for example during the American Colonial period iron nails were deliberately not made of steel but wrought iron. The reason is that a softer metal is easily deformed and in this way is better anchored in the substrate.

sM 1250 C seems to be a small unalloyed copper rivet. The microstructure of this samples is however not deformed, something expected for a rivet that was used to connect things. Maybe this object was never used. The large grains and straight annealing twins point towards a thorough annealing phase.

sBR 1157 C probably is also part of an unalloyed copper rivet (some tin present). The heavily deformed microstructure would be in accordance with the compression of a rivet in order to connect different elements.

S 0027 is the point of a small square sectioned nail (2 x 2 mm) of brass with high zinc content (*ca*. 20 wt% & some tin). The microstructure shows severely deformed annealing twins indicating cold working after annealing, possibly during hammering-in the nail.

The small nail AV 160 (see Fig. XX) with round head and square section (rather similar to S 0027 in dimensions) is from brass (*ca.* 18 wt% Zn & 1,3 wt% of Sn). Some of the annealing twins seen in the microstructure are deformed, possible related to the hammering-in of the nail. The choice of brass in this case is probably related to the decorative aspect of brass.

Two nails analysed by L. Weeks turned out to from unalloyed copper.

- **Lock plate**

BQ 153 is a lock plate with an opening to insert a key. It most likely was part of the mountings of a wooden box, decayed over time. BQ 153 was a surface find and has a punched decoration. One more example is known from ed-Dur (AV 160) and was found in the rich tomb G 5156 (area AV).

BQ 153 is made of a copper (*ca.* 1 wt% of Sn and some lead). The grain size in the microstructure shows a large variety, with large grains appearing next to small ones. The

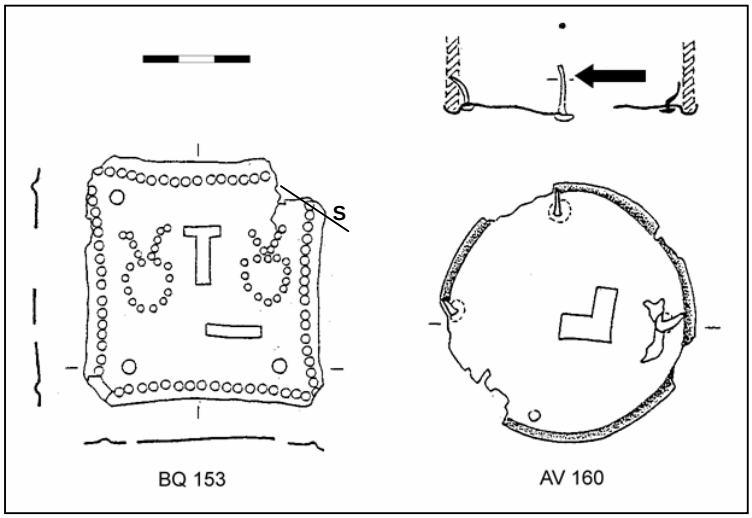


Fig. 50: Drawings of the lock plates.

annealing twins are deformed. Strain lines and mechanical deformation twins appear near the surface, indicative of heavy working after the annealing phase. The plate must have been cast, worked and annealed. When attached to the substrate (a wooden box?) the plate was hammered into place, generating the 'symptoms' of working described above.

Unfortunately no sample was taken from the lock plate AV 160, only the small nail was sampled (see above, *Nails & rivets*). The reconstruction shown on Fig. 50 of the substrate to which AV 160 was attached seem incorrect to me. It is more probable that the wooden board is located *parallel* to the plate with an opening underneath the L-shaped cut-away. In this opening the locking mechanism must have been placed. So the nails were not hammered into a wooden board perpendicular to the plate but parallel to it.

• Miscellaneous & undefined fragments

Fragment BS 066 (from the occupation level) was tentatively described as a 'stick or small chisel' by the excavators. The object is made of brass (14,5 wt% of Zn) with some tin present (1,48 wt%) and some lead. The metal was heavily worked to shape, exhibiting deformed annealing twins and grains, and strain lines. The reduction in thickness was considerable, judging from the elongation of the lead inclusions.

The flat fragment N 051 (from occupation level) is a brass with *ca.* 16,8 wt% of zinc and some tin (0,6 wt%). Although annealed at least ones the metal was extensively cold worked to its final shape and not annealed after that. It is tempting to seen this flat fragment as part of a vessel. This would fit the information of the *Periplus* where it is stated that the Romans exported brass vessels to Adulis (in present-day Ethiopia)[259]. If brass vessels were exported in this direction, they may as well have travelled via different trading channels to the Gulf-region.

ED 009 (surface find) is an unidentified unalloyed copper object and tentatively described as a 'hook'. At the broadest end the remains of a perforation are seen, and this may indicate that this piece was originally attached to something by means of a rivet or a nail. The microstructure of this sample evidences heavy cold working (elongated inclusions, deformed grains and annealing twins) after at least one annealing phase to give it the final shape.

K 203 and C 079 are identical objects, as is AV 150 (not analysed). It is not clear what these objects were used for, but it can be suggested that they were handle attached to a vessel. Both analyses were done on drilling, so no microstructural information is at hand. The alloy used for these object was leaded bronze (in both cases 12,2% of Pb by ICP-MS). The tin levels respectively were *ca*. 11 and *ca*. 7 wt%. Most probably these objects were cast to their final shape, with no or little additional working. The high lead content would make the working of such an alloy difficult anyway.

[259] Casson, 1989b: 18-21 & 53.

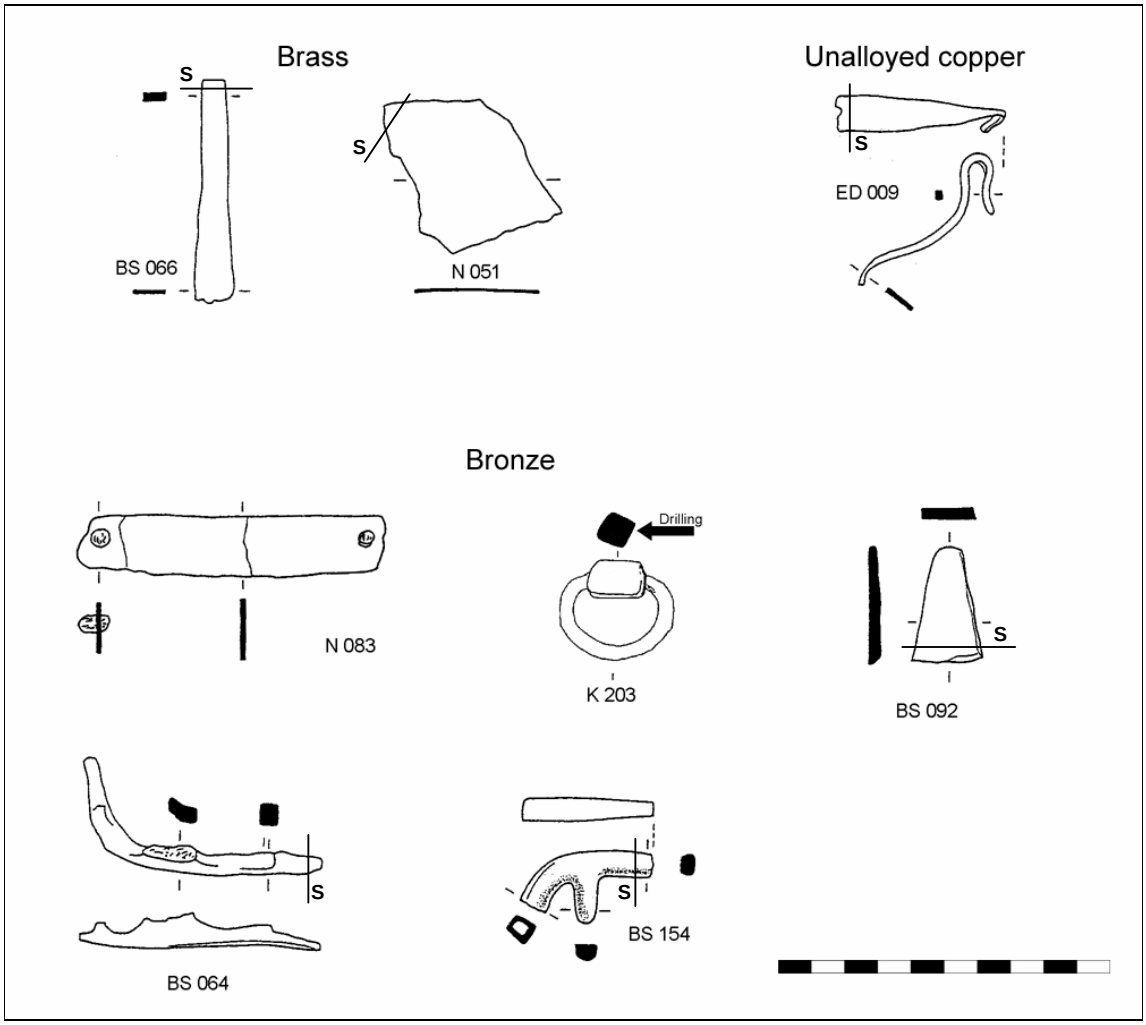


Fig. 51: Drawings of some unidentified objects.

BS 092 (occupation level) was made of bronze, but the metal was too corroded to abstract any further information. Small islands of uncorroded metal exhibited straight annealing twins however.

BS 064 (from an excavation dump) is tentatively described by the excavators as a 'horse bit'. It was made of a medium tin-bronze (10,4 wt% of Sn) that was lightly leaded (5,8 wt% of Pb by EDX). Coring is still seen in the microstructure and no grains were formed. The cast was not optimal since many (shrinkage?) pores are present.

A sample analysed by L. Weeks and described as 'horse bit?' (sBR 1176) had a similar tin contents, but was heavily leaded.

BS 154 (surface find) was cast from a heavily leaded (*ca*. 26 wt% of Pb) low tin-bronze (*ca.* 3,6 wt% of Sn). The cast might have occurred in a clay mould (see p. 167) and no further treatment of the metal is evidenced.

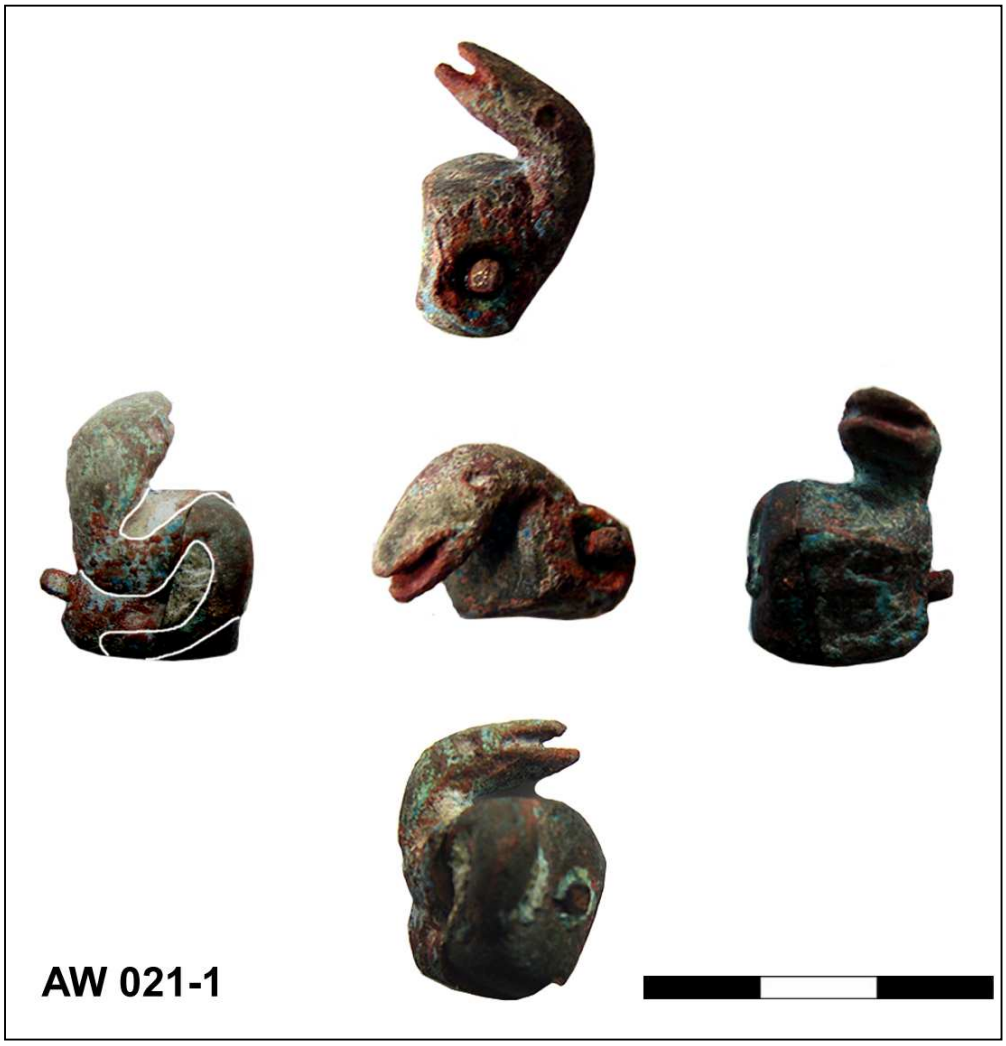


Fig. 52: Pictures of decorative element AW 021-1 (white line added to bring out the snakes body).

AW 021-1 was a completely corroded object, but after the oxidation layer was removed it turned out to be a decorative element. A snake is seen crawling around a more or less cubic element. The white line on Fig. 52 is added to bring out the body of the snake better. This also shows that this part of the element was meant to be seen. A metal wire runs through the entire element. The wire was undoubtfully used to attach the element to something (whether it be a vessel or something else), so the snake was depicted as crawling up to something, just looking over the rim. No parallel pieces are known to me and since the object was found by the British team working on ed-Dur no contextual information is available, but it likely was found in a tomb (G 5437). Depictions of snakes are known from the period preceding that of ed-Dur, i.e. the Iron Age, where they for example appear on ceramic vessels.

AW 021-1 was cast to its final shape from a medium tin-bronze (*ca*. 15 wt% - 13 wt% by AAS) with many α+δ-eutectoid islands present in the microstructure. The object is leaded (*ca*. 10 wt% by EDX) and large lead globules are present throughout the examined section. The large amount of lead is certainly present to ease the casting. There does not seem to have been an annealing phase, since the dendritic structure and coring is present.

- **Artefacts and cultural tradition**

One point that is often overlooked in archaeology and more specific archaeometallurgy is the fact that the use of a certain alloy can also be linked to a cultural tradition. The *Western* view often only considered the metallurgical advance from a technological point of view. But this is not always sufficient to grasp the complete picture and other cultural motivations can influence the chose of alloy. This however is a difficult issue to evaluate to the fullest, especially if textual evidence is absent. The persistence in use of copper-base alloys in SE-Arabia, although iron was already known and present in the same area might be an example of this.

N. Lahiri summarizes this hiatus in relation to Indian copper working as follows[260]:

> "(1) The persistent and numerically dominant tradition of working in copper of high purity That one observes in the early Indian archaeological record does not have any technological implication and, on the contrary, fits in with what we know about the ritual importance of pure copper in ancient Indian texts. The continuity of this tradition and the position of superiority of craftspersons in pure copper to those dealing with various alloys in the caste hierarchy is underlined in the more recent ethnographic background of metal-related craft traditions. (2) The factor of variation in the elemental compositions of the Indian metal artefacts also does not have any technological dimension. This must be understood in relation to a very dominant and ethnographically well-documented tradition of recycling objects and scraps of old metal. As some textual and archaeological sources indicate, this tradition goes back to the ancient period as well. (3) In some cases, metal or metal-related objects are focused around specific historical events and folk beliefs; the stories/myths and artefacts are linked to each other in ways which suggest that in such contexts the latter can only be understood in a symbolic sense, as signifiers of social and cultural beliefs."

D. Dungworth also mentions an alloy choice for spiral rings from N-Britain. They are divided in two groups, the larger type was worn as a finger-ring and are composed of brass, while the smaller type used as earrings are made of bronze. This may also indicate a social and symbolic meaning to the wearer.[261] A tentative comparison to the ed-Dur material can be made, where the finger-rings are made of brass, whereas anklets and bracelets are all made from a rather uniform medium tin-bronze.

This facet, how interesting it might be, will not be further developed in this study, since this is mainly a *technological* study and the crucial key of textual evidence is not available from the region and period under evaluation. It is mentioned however and has to be kept in mind as a possible influence when looking at ancient material.

[260] Lahiri, 1995: 117.
[261] Dungworth, 1997b: 909.

5.7. Minor & trace elements – SEM-EDX & ICP-MS

5.7.1. Minor & 'trace' elements – SEM-EDX

The minor elements that are detected by EDX were sulphur, iron and nickel. Relatively little can be done with those results, since they are all very low and not very reliable. Keeping this in mind some things can be said however on the iron and sulphur levels. These are addressed together because they frequently occur combined in non-metallic inclusions in copper objects as a result of the exploitation of Cu-Fe sulphides, and are both impurities that are generally removed during the refining process to improve the quality of the finished object.[262] Average iron concentrations of around 0,05% are characteristic of copper produced during using a non-slagging extraction process. Whereas a slagging process commonly produced iron concentrations an order of magnitude higher.[263]

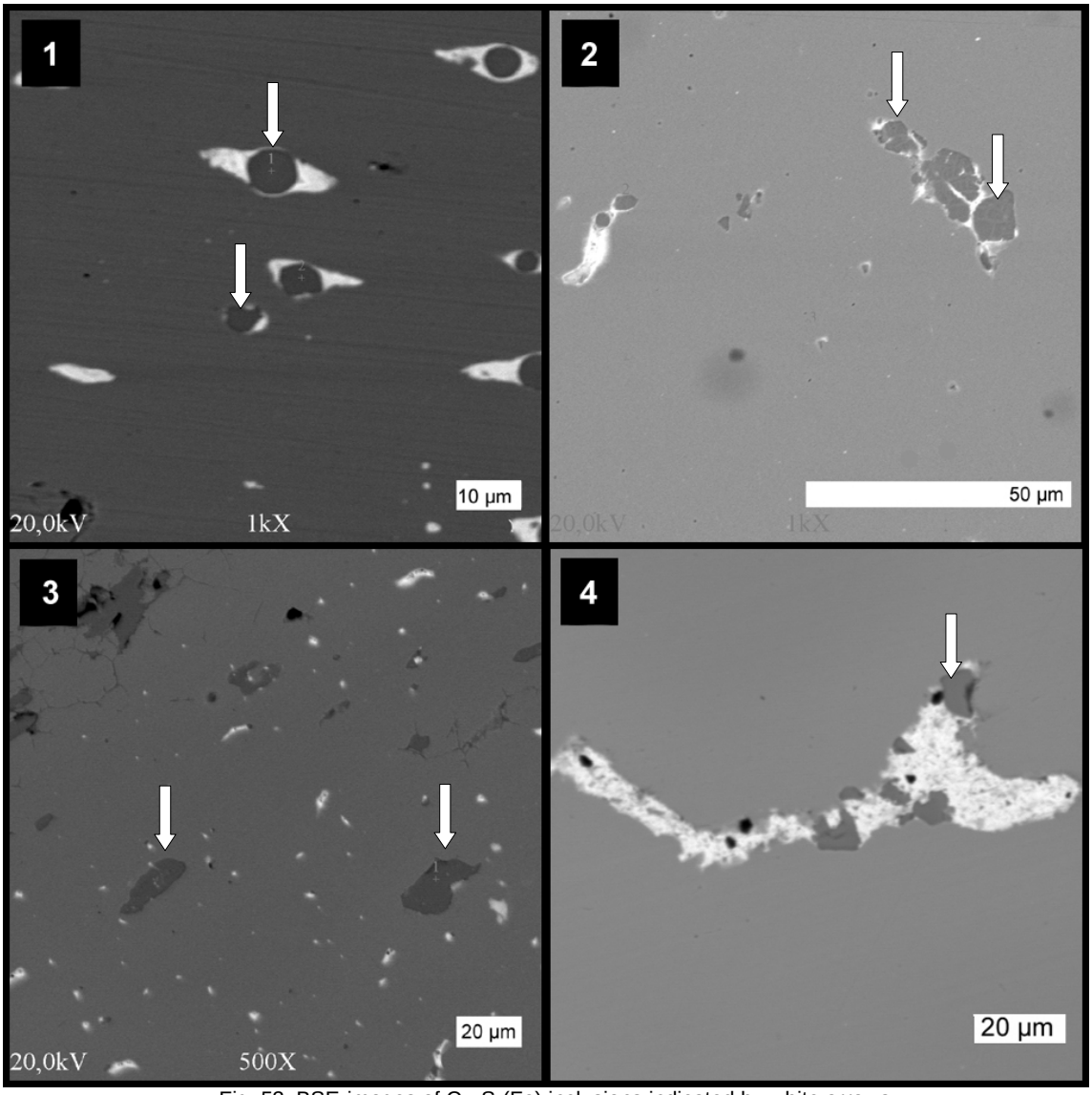


Fig. 53: BSE-images of Cu-S-(Fe) inclusions indicated by white arrows
(1: M 084; 2: BQ 016; 3: BQ 070 & 4: BS 1276).

[262] Weeks, 2004b: 106.
[263] Craddock, 1995: 139.

According to L. Weeks the median sulphur level in the tin-bronzes of ed-Dur is 'higher' than in previous periods in SE-Arabia. The same is not seen in the copper samples.[264] Cu-S-(Fe) inclusions are frequently present in the samples analysed (at least 52, see Fig. 12 BIJ COPPER & Fig. 53 for examples) and it has to be admitted that they were not systematically looked for at the beginning of this research. No relation was found between the alloy and the presence of these inclusions. The inclusions could be indicative of a matte smelting process or at least of the treatment of copper sulphide ore to produce copper.

Further relations between the silver and lead, lead and sulphur and nickel and iron were sought for, but no meaningful pattern was seen. Moreover it has to be concluded that the semi-quantitative technique used here is of no use to evaluate low concentration of elements detected. The question whether part of the sulphur and the silver were introduced by the lead has to remain open.

5.7.2. Minor & trace elements – ICP-MS

5.7.2.1. Introduction

The evaluation of the trace elements is only a preliminary attempt to use the limited analyses available. Due to time and money constraints only ten samples were submitted to this kind of research and this dataset is far too small to draw any definitive conclusions. On the other hand it would be a pity not to include them in the overall picture, since some interesting results did emerge. The manganese and iron levels are evaluated in relation to brass production and are compared with the bronzes analysed. The arsenic, cobalt, nickel and antimony levels are considered in relation to the ores used to produce copper. But again, especially on the ore sources, no conclusive statements can be made only a few interesting remarks that might be useful for further research in this complex field.

	Brass				***Bronze***					
					Low tin	**Medium tin**				**'High' tin**
	AT 013	**AV 083**	**AW 063-4**	**BL 014**	**AV 104** 4,8 wt% Sn	**M 007** 6,2 wt% Sn	**C 079** 7,4 wt% Sn	**AV 005** 7,6 wt% Sn	**K 203** 10,7 wt% Sn	**S 0020** 15,3 wt% Sn
Fe	0,10000	0,10300	0,38000	0,26000	0,00260	0,04000	0,08000	0,05400	0,02900	0,00229
Mn	0,00470	0,00210	0,01090	0,01600	0,00004	0,00024	0,00038	0,00024	0,00024	0,00005
Ni	0,01710	0,01610	0,01260	0,00680	0,02600	0,03630	0,06900	0,07400	1,22000	0,10100
Co	0,00130	0,00062	0,00022	0,00023	0,00231	0,00580	0,00480	0,02550	0,09340	0,03290
As	0,00750	0,02600	0,03930	0,02770	0,03530	0,02800	0,00150	0,17000	0,00230	0,00310
Se	0,00100	0,00410	0,00230	0,00048	0,00120	0,00100	0,00016	0,00090	0,00033	0,00011
Ag	0,02310	0,03940	0,02600	0,05900	0,04700	0,09100	0,12900	0,06400	0,06500	0,04700
Sb	0,02500	0,03600	0,05700	0,04930	0,19000	0,01600	0,00970	0,07200	0,00040	0,00250

Table 23: Trace elemental ICP-MS analyses in wt%.

Ten samples were subjected to ICP-MS trace elemental analyses by P. Rogiers in the frame of an undergraduate thesis at the *Department of Analytical Chemistry – Ghent University*.[265] The results for the different trace elements are presented in Table 23. The major metallic alloying elements Cu, Sn, Zn and Pb are not included in the table. All bronzes are actually leaded and it needs mentioning that the lead levels in the brasses were low (AW 063-4: 0,097%, AT 013: 0,330%, AV 083: 0,140% & BL 014: 0,027%) and none were intentionally leaded. This is in accordance with the EDX results. An additional word on the iron levels needs to be said. Iron is one of the notoriously difficult elements to measure by ICP-MS. This is because the atomic mass of the main iron isotope $^{56}Fe^{+}$ is the same as the combined masses of argon (^{40}Ar) and oxygen (^{16}O), which makes it indistinguishable for the detectors.

[264] Weeks, 2000a: 78.
[265] Rogiers, 2006.

Argon is present as the carrier gas used in the ICP-MS machine and oxygen is always present to a limited extent[266].

5.7.2.2. Manganese & iron

- **General**

As mentioned in the part on the brass above *smithsonite* or *calamine* ($ZnCO_3$, carbonate ore) can be easily reduced to its oxide ready to be used in the cementation process. Sphalerite (ZnS, sulphide ore) on the other hand had to be first roasted to driven off the sulphur. This also caused the zinc to vaporise, but unlike the sulphur that was mainly lost in the fumes, the zinc vapour sublimated in the cooler parts of the roasting furnace. An additional advantage of the roasting was that it also purified the zinc oxide to a certain extent in that only the volatile elements would sublimate (i.e. zinc and some lead if present in the ore). Other non-volatile impurities of the ore (e.g. iron, manganese, etc.) remain behind. The *calamine process* to the contrary uses the crushed ore with all its impurities. Consequently some of the iron, lead and manganese present in the calamite/smithsonite ore will also be transferred to the brass. Based on the presence of especially iron and manganese it should be possible to make a distinction between brass made from calamine/smithsonite on the one hand and sphalerite on the other hand.[267]

From an archaeological point of view this has an interesting consequence. In Europe and the Mediterranean the common zinc mineral utilized in antiquity was smithsonite, whereas in the Near and Middle East sphalerite was used from an early date. The reason is that few viable smithsonite deposits are to be found in the Near and Middle East.[268] Sphalerite on the contrary is found in deposits in E-Turkey, to the east of the Black sea, N-Saudi Arabia and E-Azerbaijan. For the sake of completeness it needs to be mentioned that many sphalerite ore body are also known within Europe, but they seem not to have been used. This is probably due to the more extensive preparation needed to obtain usable zinc oxide, something not needed for the calamine/smithsonite at hand. Significant levels of iron and manganese in finished alloys are thus indicative for the utilization of natural smithsonite with the absorption of these elements into the smelt. By consequence Near and Middle Eastern brass is likely to have lower levels of impurities such as iron and manganese[269]. Does this hypotheses stand up to the analytical results obtained?

In the three Middle Eastern early brass fragments from Tepe Yahya manganese was not a significant trace element[270]. This is consistent with the view of the use of sphalerite in that region, since no smithsonite is present. The low iron content of the 1st c BC Hellenistic brass coins (average Fe content 0,22% and no Mn) is indicative of the use of treated sphalerite as well, rather than the untreated smithsonite which was apparently used in Roman issues (average Fe content 0,47% and regularly Mn between 0,001-0,045%).[271] A comparison of the iron content of the Roman provincial coins dating up to the 2nd c AD coins with near contemporary Roman issues shows that for in the provincial coins the range of the iron content remains very similar. The alternating high and low iron concentrations (under 0,25%) during the 1st c BC to 2nd c AD suggest that both sphalerite and smithsonite were used at that time. For Rome however the iron content is high in the 1st c BC, but this is much lower from the 1st c AD onwards (between 0,1 and 0,5%). In the 1st c AD they do not reach as low as the provincial mint issues but in the 2nd c AD there are a number of brass coins with very low iron content. This may be due to the increasing use of sphalerite but a more likely explanation is the probable recycling of brass for Rome issues, thereby reducing both the iron and zinc

[266] Also the other isotopes of iron cause problems (D. De Muynck pers. comm.).
[267] Bayley, 1998: 10; Ponting, 1999: 1317; Weeks, 2004a: 246.
[268] Bayley, 1998: 10; Ponting & Segal, 1998: 118; Ponting, 1999: 1317; Cowell, Craddock, Pike & Burnett, 2000: 677; Ponting, 2002: 562.
[269] Bayley, 1998: 10; Ponting, 1999: 1317; Cowell, Craddock, Pike & Burnett, 2000: 677; Thornton & Ehlers, 2003: 3.
[270] Thornton, Lamberg-Karlovsky, Liezers & Young, 2002: 1459.
[271] Craddock, Burnett & Preston, 1980: Tables on 56 & 58.

content. The difference between the provincial and the Rome issue coinage, along with the apparent regional difference in the use of alloys, suggests a high degree of autonomy of metal acquirement and coin production in the province of Asia. This is in contrast to the situation for brass supplies to the Roman army in Palestine (see below, Masada and Gamla) where it has been suggested that smithsonite brass may have been imported from the western frontiers of the Empire.

It can even be argued that it was the local availability of smithsonite on the Rhine frontier which lead to the wholesale adoption of brass by the Roman military in the 1st c AD rather than any metallurgical advantage which brass had over bronze[272]. The coin production however seems to have followed local tradition.[273]

Significant traces of both manganese and iron were detected in Roman military brasses from Masada and Gamla (Palestine), Roman coinage and artefacts from the Roman site of Camerton (Great-Britain). Especially when the levels are compared to the levels of these elements in contemporaneous bronze and copper artefacts (see Table 24). The concentrations of manganese and iron in the military brasses from Masada and Gamla are similar to those found in European brasses and could suggest a common (European) origin for these alloys.[274]

	Brass		**Bronze/copper**	
	Mn	**Fe**	**Mn**	**Fe**
Masada & Gamla (Palestine)	0,0051	0,23	< 0,0005	0,19
Camerton (Great Britain)	0,0011	0,32	0,0005	0,27
Roman coinage	0,0035	0,28	0,0005	0,20
Bet She'an (*Scythopolis*, Palestine)	0,0007	0,20	0,0006	0,21
Islamic	< detection limit	0,20	No information	

Table 24: Avearge manganese & iron levels (wt%) in brass &bronze/copper from different periods (after Ponting, 1999: 1318).

The manganese and iron levels in the Bet She'an (Palestine) brasses, which has a sequence of objects from the Roman, early and late Byzantine, over the Umayyad Abbasid to Mamluk period, are low and more consistent with the amounts found in copper and bronzes. When compared to the European brasses it can be suggested that the technology is different and better fits the Near eastern sphalerite-brass tradition. Unfortunately no comparative data exists for the Late Antiquity/Early Mediaeval brass from the Levant and no direct comparison are yet possible. Moreover iron and manganese are not always looked for or the techniques that are used are not sensitive enough to detect them. If individual pieces are looked at there are some pieces with high iron and/or manganese levels. The single Mamluk piece is a leaded low tin-bronze with exceptionally high manganese values (0,0042%) and this is probably related to a very different copper or flux source. The remaining pieces with elevated iron and manganese levels are predominantly Late Byzantine and Abbasid in date. This may reflect either the reuse of earlier Roman brass imported from Europe or the occasional use of (Anatolian?) smithsonite as a zinc source. Given the low overall zinc level, it is likely we are dealing with recycled brass however.[275]

The only comprehensive analytical survey of Islamic copper-base alloy metalwork so far published suggests on the basis of the iron content that the 9th c AD brass produced in the Near East was made by using the sublimated zinc oxide from sphalerite ores. The European brass is generally higher in iron and some brasses have several percent of the metal. The use of sphalerite could explain why the majority of the Islamic brass does not contain more

[272] Ponting & Segal, 1998: 117-119.
[273] Cowell, Craddock, Pike & Burnett, 2000: 676-677.
[274] Ponting & Segal, 1998: 117-118.
[275] Ponting, 1999: 1318.

iron than would be expected to come from the copper alone, typically around *ca.* 0,2%.[276] Contemporaneous mediaeval European brasses can contain up to a several percents of iron[277]. The limited study by Z. Al-Saa'd on three high zinc brass and two leaded medium zinc level brass objects from the Islamic period (dating between 13th to 18th c AD), from Jordan does however report high iron content (0,33 %, Mn was not looked for)[278]. Which again would imply the use of smithsonite.

The summary above shows that manganese and iron can be used as a guide to identify brasses made from the two different ores, i.e. smithsonite and sphalerite. For the completeness it should be mentioned that manganese could also enter the brass in other ways. It is possible for manganese to be introduced into copper during smelting with an iron oxide flux or in mixed copper-manganese ores, but it will generally pass through the smelt into the slag rather than into the copper.[279] It might be argued that manganese is actually a better determinant for the separation of both techniques, since the difference in iron levels iron levels is less well defined. Using manganese as a marker also solves the problem of the problem of the difficulty to detect the exact amount iron with ICP-MS.

What can this conclusion tell us about the brass from ed-Dur.

- **Ed-Dur**

In Fig. 54 the manganese level is plotted against the iron level, to evaluated the two diagnostic elements for brass made from calamine/smithsonite. The four brasses are clearly separated from the bronzes, suggesting that these two elements were introduced by the zinc. The variation of manganese in the four brasses from ed-Dur is greater than that seen for the Roman material. Especially the upper limit is higher for the ed-Dur material. The manganese levels of the bronzes correspond well with those given for Roman bronze. The iron values are lower than these given for the Roman material, but still well above these of the bronzes. Moreover the overall iron content of the ed-Dur bronze is also lower than that given for the Roman material.

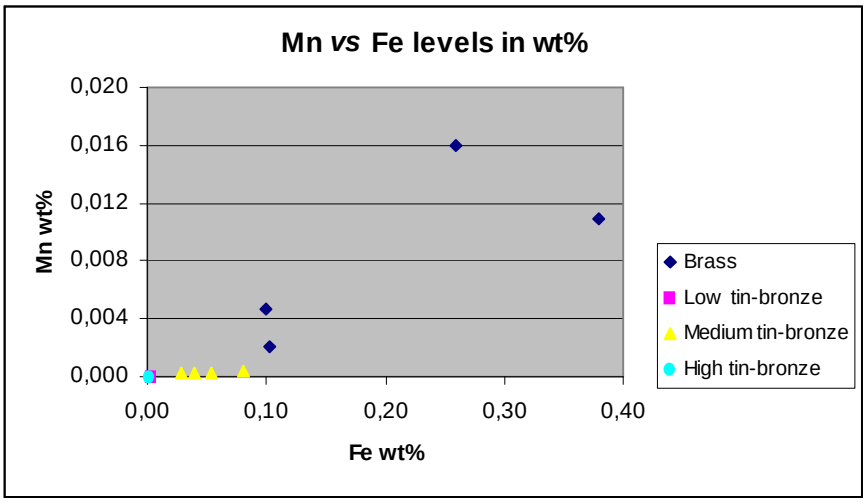


Fig. 54: Manganese *versus* iron levels in wt%.

[276] Craddock, La Niece & Hook, 1998: 78.
[277] Craddock, La Niece & Hook, 1998: 78.
[278] Al-Saa'd, 2000.
[279] Ponting, 2002: 563-564.

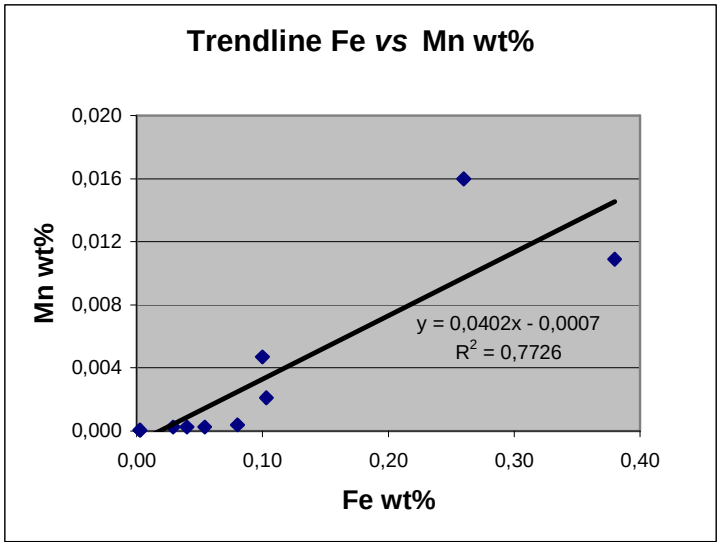


Fig. 55: Trendline manganese *versus* iron levels in wt%.

The elevated levels of manganese and iron levels in the brass in comparison to those of the bronze are a clear indication that they are *smithsonite brasses*. This in turn suggests a European or Mediterranean and not a Near Eastern origin. Moreover there is a clear positive correlation between manganese and iron (see Fig. 55).

5.7.2.3. Arsenic, cobalt & nickel

• **General**

Only a limited amount of studies were used to evaluate the analytical results for arsenic, cobalt, nickel and antimony obtained in this PhD. These publications are all mainly concerned about the early copper production and copper and copper-base objects in SE-Arabia. This is mainly due to the late decision to incorporate this chapter[280]. These four elements are related to the copper ores used to produce the copper.

At a smelting temperature of about 1200°C in the fu rnace the following elements would follow the copper instead of being absorbed in the slag: Co, Sb, Ni, As Pb, Bi, Se, Te and Ag. The average nickel and arsenic concentrations of Oman and Bahrain copper are both about 0,5%, ranging from 0,01 to 10%. Copper with such levels of nickel and arsenic was used from the Early Bronze Age to the Iron Age. The average nickel and arsenic concentrations in the ores are in the same range as the metal, but show larger variations. This similarity in element concentration indicates that the As-Ni copper metal used is a *natural alloy*. In addition some copper metal inclusions in smelting slags also show the same trend of elevated nickel and arsenic levels. This all indicates that the metal used for the objects must have been produced from local ores. The average As-level from locally available ores appears to be somewhat lower than in the objects. This is a well-known, but poorly understood phenomenon in archaeometallurgy. A possible but unverified explanation could be that the copper which separates from the slags is enriched in arsenic, in contrast to metal inclusions remaining in the silicate slag, since any concentrations of trace and minor elements will lower the melting point of copper and facilitate this separation.[281]

There is a strong positive correlation between arsenic, nickel and cobalt (and perhaps antimony) for copper material from the Umm al-Nar period. This relation is less clear for the

[280] Being: Hauptmann, Weisgerber & Bachmann, 1988; Prange, Götze, Hauptmann & Weisgerber, 1999; Weeks, 2000a & Weeks 2004b.
[281] Hauptmann, Weisgerber & Bachmann, 1988: 41; Prange, Götze, Hauptmann & Weisgerber, 1999: 188-190.

tin-bronzes. They probably reflect the mineralogy of the copper deposits that were the source. Enormous numbers of analyses of copper-base (alloy) objects have clearly shown that in many areas of Europe, Asia and the Americas, copper objects with significant levels of arsenic and other elements (e.g. antimony and nickel) were a feature of early metallurgy. Ore containing arsenic, antimony, nickel and cobalt can be found associated with copper in the oxidized, enriched and primary ore zone of many weathered ore deposits. Arsenic and antimony are often concentrated in the secondary enriched zone of copper deposits as copper sulpharsides, where they form the mineralogical series of tennantite ($Cu_{12}As_4S_{13}$)-tetrahedrite ($Cu_{12}Sb_4S_{13}$) as found in a number of copper mines in E-Turkey and Iran.[282]

High nickel and arsenic (and cobalt[283]) content has subsequently often been considered as characteristic for copper originating from ores found in SE-Arabia. Caution should however be taken, in that also deposits outside this region can contain similar amounts of nickel (i.e. Iranian ores). If the copper is smelted in a simple way (e.g. without the formation of matte) from a pure copper ore the recovery of nickel in the metal is high, up to 80% of the amount originally present in the ore. When copper sulphides are smelted however the nickel is always concentrated in the iron sulphide phase in the matte. This phase is removed by slagging and the resulting metal will contain much less nickel than the ore or slag.[284] There are no indications that copper was being produced in SE-Arabia during the 1st c BC – 2nd AD, and if the limited evidence of Mleiha is considered the process was very primitive and certainly not related to the matte smelting.

- **Ed-Dur**

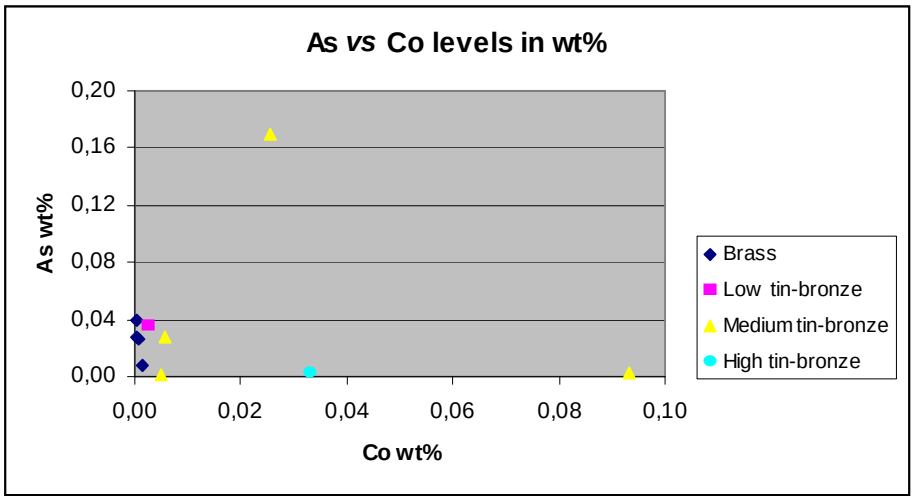


Fig. 56: Arsenic *versus* cobalt levels in wt%.

In comparison to copper and bronze samples analysed from the older contexts from SE-Arabia L. Weeks reports that the ed-Dur samples[285]:

- Arsenic levels are lowest of all periods in SE-Arabia.
- Cobalt levels are the lowest of all periods in SE-Arabia.
- Nickel levels are the lowest of all periods in SE-Arabia, but much higher in bronzes and brasses than in copper.

[282] Weeks, 2004b: 97-98 & 108-110.
[283] Ponting, 1999: 1318.
[284] Hauptmann, Weisgerber & Bachmann, 1988: 41.
[285] Weeks, 2000a: 84-97.

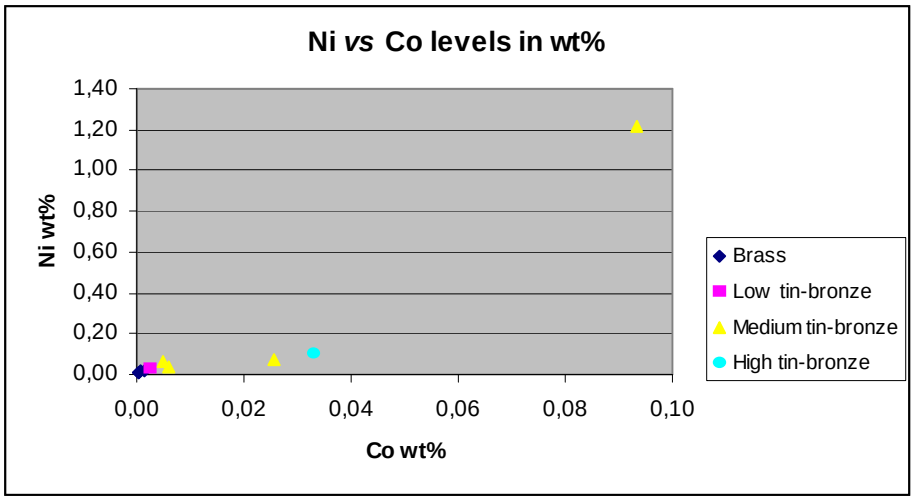


Fig. 57: Nickel *versus* cobalt levels in wt%.

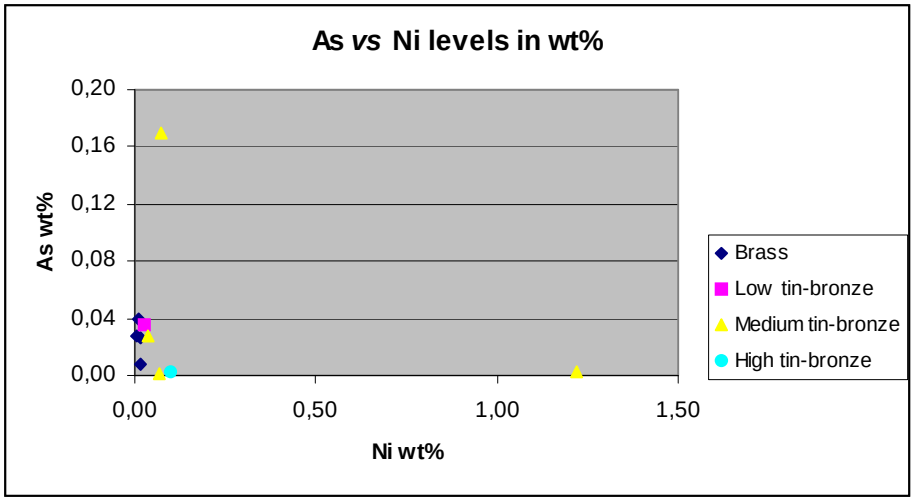


Fig. 58: Arsenic *versus* nickel levels in wt%.

Three samples show a different signature (AV 203, K 203 & S 0020). The rest groups in the low arsenic, nickel and cobalt region relatively close to each other.

Arsenic, cobalt and nickel are elements often associated with the copper ores used. I will not go as far as to try to link these the results to possible ore bodies, but only to compare the samples amongst each other. The nickel and arsenic levels are in accordance to L. Weeks' results. The cobalt levels in the analyses presented above are generally lower than those published by L. Weeks. The three samples with the highest cobalt levels also have the highest nickel levels (AV 005, K 203 & S 0020) and all three are bronzes. Cobalt and nickel seem to be correlated to each other (r^2 = 0,89), but the number of samples included is too small to draw definite conclusions. This is no significant correlation between arsenic and nickel (r^2 = 0,04), and arsenic to cobalt (r^2 = 0,03). The nickel and cobalt levels are very low for the brasses, setting them apart from the bronze (except for the low tin-bronze AV 104). This might suggest that a different copper source was used for the brass then for (some of) the medium and high tin-bronzes. If we accept that the copper contributes the nickel, arsenic and cobalt then it can be suggested that more than one copper source was used.

The difference in the nickel and arsenic levels in the ed-Dur material with those of Bronze and Iron Age Oman object, ingots and ores show that they were not made from the same ore

deposits and most probably not from ore from SE-Arabia. Additionally the copper-base objects from ed-Dur are also not the result of recycling older copper-base objects made from local ores, since then the nickel and arsenic levels should have been higher. On exception might be K 203. This object (a handle) has an exceptional high nickel level. Within the small dataset the cobalt level is also relatively high, but still low when compared to older material from SE-Arabia. It could be hypothesised that this metal is the result of recycling of older metal and/or of locally produced metal, but all-in-all it does not really fit the overall composition of metal from SE-Arabian origin. If the origin for the metal at ed-Dur from SE-Arabia can be excluded, also an origin from ores of S-Iran can be excluded on the basis that these deposits are very similar to and maybe even indistinguishable from those of SE-Arabia.

5.7.2.4. Silver, selenium & antimony

The antimony can also be introduced as an impurity by the lead added to the alloy, next to a contribution of the copper ore. The presence of antimony suggests that the lead was smelted at a high temperature which preserves the impurities in the ore and therefore probably the by-product of silver extraction. Simple lead extraction for the lead metal itself is a low temperature process. Pliny mentions two types of lead: *plumbum argentarum* and *plumbum nigrum*. Recent research has shown that the former was used to indicate lead that was smelted primarily for its silver content, whereas the latter was smelted at low temperatures, for the lead itself.[286]

Antimony levels were below the detection limit of PIXE to be evaluated in the work of L. Weeks. Additionally selenium levels are the lowest of all periods in SE-Arabia. Lead and silver levels at ed-Dur are much higher however than in any previous periods in SE-Arabia (especially in the bronzes) [287]. Selenium was only found in six samples by L. Weeks, five of which were of unalloyed copper.[288] This could not be verified in the small dataset here, since no copper samples were present among the samples for the trace elemental analyses.

The silver levels seen here are in accordance with those published by L. Weeks. The amount of silver is generally higher in the heavily leaded bronzes, indicating that some of the silver may be introduced by the lead. This is however not statistically supported (r^2 = 0,1656), so the lead cannot have been the only contributor as for example the 'lead-free' brass BL 014 contains more silver than the leaded bronze S 0020. The only common major element in the alloys that might be suggested as a contributor is the copper.

The selenium and antimony levels in the high tin-bronzes are lower than in the other alloys, but no further conclusions can be drawn from this.

[286] Ponting, 1999: 1318-1319.
[287] Weeks, 2000a: 84-97.
[288] Weeks, 2004a: 243.

5.8. Lead isotope analyses – ICP-MS

Fig. 59 gives the scatter plot of the lead isotope ratios of all the analysed copper and copper-base alloys in this study. Error bars indicate the error on the measurements. Six samples of Khor Rori are also included. The leaded samples have to be treated with the necessary cautions, since it is not clear which part of the lead isotope ratio is contributed by the copper and which part by the lead. All these are 'heavily' leaded and logically the input of the lead will be dominant in the isotopic make-up. The complete dataset can be found in *Appendix 5*.

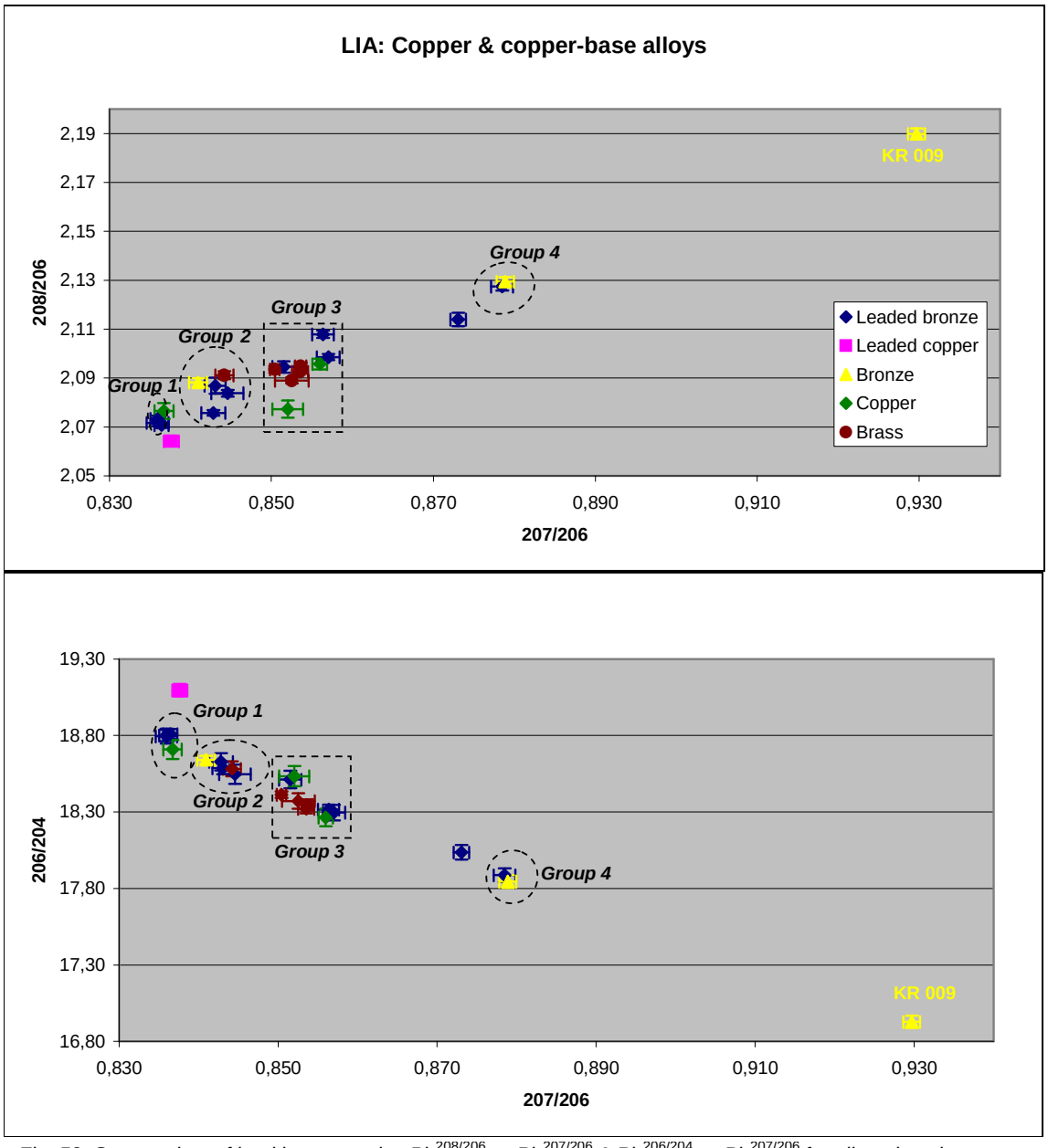


Fig. 59: Scatter plots of lead isotope ratios $Pb^{208/206}$ *vs* $Pb^{207/206}$ & $Pb^{206/204}$ *vs* $Pb^{207/206}$ for all analysed copper & copper-base alloys (the error is indicated by error bars).

For the ease of comparison the data is split-up in four 'groups' and the outlier KR 009. I will first discuss the outlying lead isotope ratios of KR 009 from Khor Rori, so it can be removed from the next plots and the groupings are made better visible.

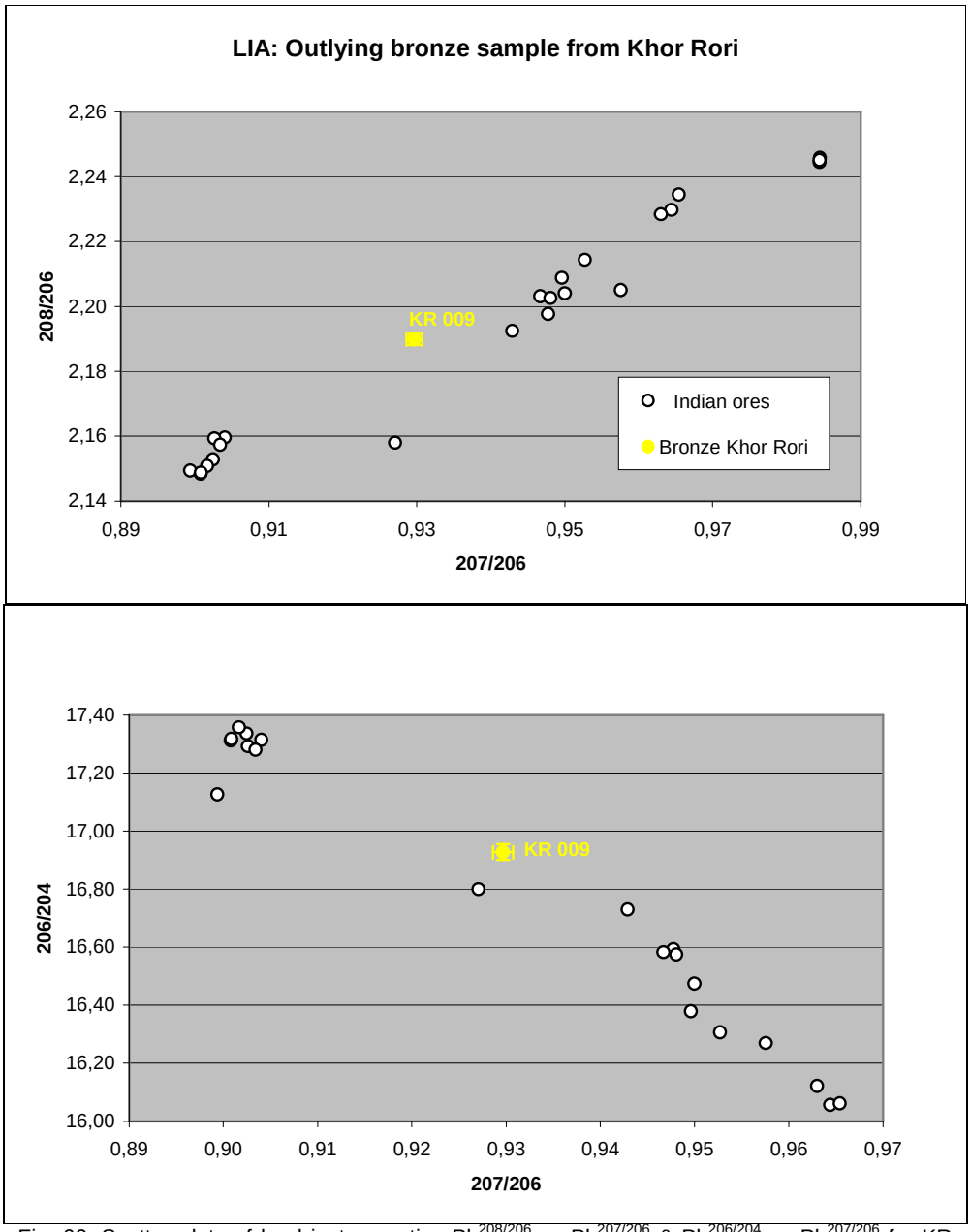


Fig. 60: Scatter plots of lead isotope ratios $Pb^{208/206}$ *vs* $Pb^{207/206}$ & $Pb^{206/204}$ *vs* $Pb^{207/206}$ for KR 009 and Indian ore sources (the error is indicated by error bars).

The only ore sources that plot on in neighbourhood of KR 009 are ores from the Indian Subcontinent (Fig. 60). The ore samples were taken from sources in the Rajasthan region. The ores are polymetallic and contain copper, zinc and lead.[289] The lead isotope ratio of the ores do not fall completely together with the sample from Khor Rori, so the copper does not come from one particular sampled ore region. The copper metal can however be a mix of metal originating from several regions in India, which would give an intermediate isotopic signature. Alternatively and equally possible, the bulk of the isotopic signature of the Khor Rori sample may be made up from a contribution of the Indian copper. Copper from different origin may contribute the rest. A third possibility would be an as yet unidentified copper ore sources in India, possibly located in the Rajasthan region.

[289] Ericson & Shirahata, 1985: 207-209.

The implication of an attribution of the sample from Khor Rori to an Indian origin is intriguing. The *Periplus* and Pliny both state that copper was imported into India from the Roman World and suggest that no indigenous copper was produced. The findings here would contradict that and suggest that at least to a limited extent copper was produced in India, and apparently exported to Oman. H. Chakraborti states that there is clear evidence that sources in Rajasthan were worked at 'early times'[290]. This region would have had an outlet via the harbour of Barygaza. Moreover Barygaza is mentioned in the *Periplus* as sending out shipments of copper to the Gulf. The signature of KR 009 would fit the idea that India was producing and even exporting some indigenous copper as well. The dependence of India on imported Roman copper might have been less significant than suggested in the *Periplus* and by Pliny and rather meant to create a surplus than to fill a shortage.

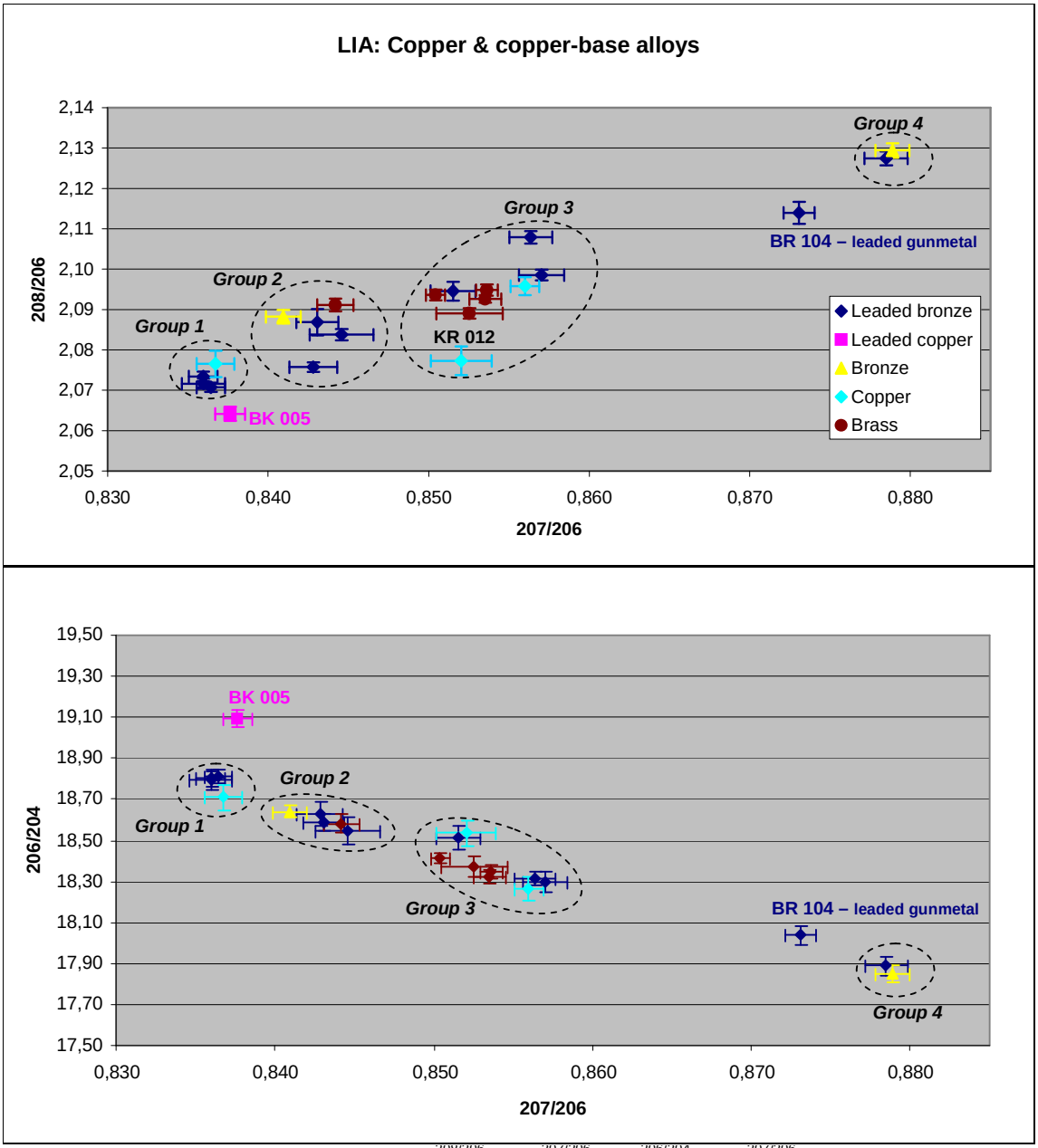


Fig. 61: Scatter plots of lead isotope ratios $Pb^{208/206}$ *vs* $Pb^{207/206}$ & $Pb^{206/204}$ *vs* $Pb^{207/206}$ for all analysed copper & copper-base alloys (the error is indicated by error bars). The outlier KR 009 is removed.

[290] Chakraborti, 1966: 253.

Fig. 61 shows the same plots then Fig. 59, but with the outlier K 009 removed. This is to make the groups more visible. It has to be stressed that these groups have no other meaning then to make it data better comparable.

- ***Group 1*** contains one copper (N 138, altar bead) and three leaded bronze (AV 005, *patera* - AV 104, horse appliqué - S 0020, female head appliqué) objects.
- ***Group 2*** contains one brass (BO 029, small bell bead), one bronze (AV 115, fragment ladle) and three leaded bronze (C 079, handle vessel - M 007, pedestal statuette - N 118, bead) objects.
- ***Group 3*** contains two copper (BS 169, tetradrachm & fragment KR 012), three leaded tin-bronze (K 203, handle vessel - KR 007 & KR 008 - 2 fragments from Khor Rori) and four brass (AT 013, AW 063-4 & BL 014, ring-pommels) objects. KR 012 only is closely associated with the other samples on one of the two plots however.
- ***Group 4*** contains one tin-bronze (KR 010, fragment from Khor Rori) and one leaded tin bronze (KR 011, fragment from Khor Rori) object.
- The leaded gunmetal BR 104 (bell) and the leaded copper BK 005 (lion bead) are not really part of any of the groups.

The three leaded bronzes in *Group 1* are isotopically almost identical and this must point to a common place of origin. All three are heavily leaded (more than 17 wt% of lead) and their signature is thus more indicative of the lead used in the objects than the copper. It is interesting that amongst these three artefacts the rams head *patera* is seen (AV 005). This object is almost without any doubt from Roman origin and thus made from metals extracted in the Roman Empire. What is more intriguing however is the fact that the horse-shaped spout (AV 104) is also part of this cluster. This object seems to be typical for the SE-Arabian Peninsula with several comparable pieces in the region. Intuitively it would be labelled as a local SE-Arabian product. Several explanations can be suggested for this.

- The first is that unalloyed 'Roman' metals came to SE-Arabia from the same origin as the metals used for the *patera* and that these metals were locally alloyed to produce the spout.
- The second possibility is that pre-alloyed metal came to SE-Arabia from the same origin as the *patera* and was locally processed.
- The third possibility is that imported 'Roman' objects were recycled and recast in new objects.
- A forth suggestion could be that these horse spouts are not local at all, but imported as finished objects.
- A last possibility could be that only the lead used for the spout was the same as that used for the *patera* and that is completely masks the isotopic signature of the copper metal

Not enough evidence is at hand to solve this question, but maybe the last two suggestions are most acceptable. The third piece in this cluster is the female head appliqué (S 0020), but this object is not allocated to a certain point of origin.

Group 2 includes the pedestal (M 007), which almost without any doubt is Roman, it does however not seem to have anything in common with the *patera.* The ladle fragments AV 115 is thought to be a local product. The small brass bell has a possible link to the Middle East. The other objects in this group are not very diagnostic.

The most interesting thing of brasses *Group 3* is that it contains most of the brasses (ring-pommels and large rivet) group in, except for BO 029 (a small bell). That these brasses have a similar signature point towards the fact that they were made up from the same 'ingredients', regardless of their individual origin. As mentioned in *Chapter 4* brass is however difficult to provenance based on its isotopic composition. This is because zinc ores often appear together with lead ores and that part of that lead contributes to the isotopic signature, next to the copper.

The two samples in *Group 4* are plotted close together, it should however be noted that one leaded and the other one not. It is thus not clear if this closeness has anything to do with a similar origin or is a coincidence.

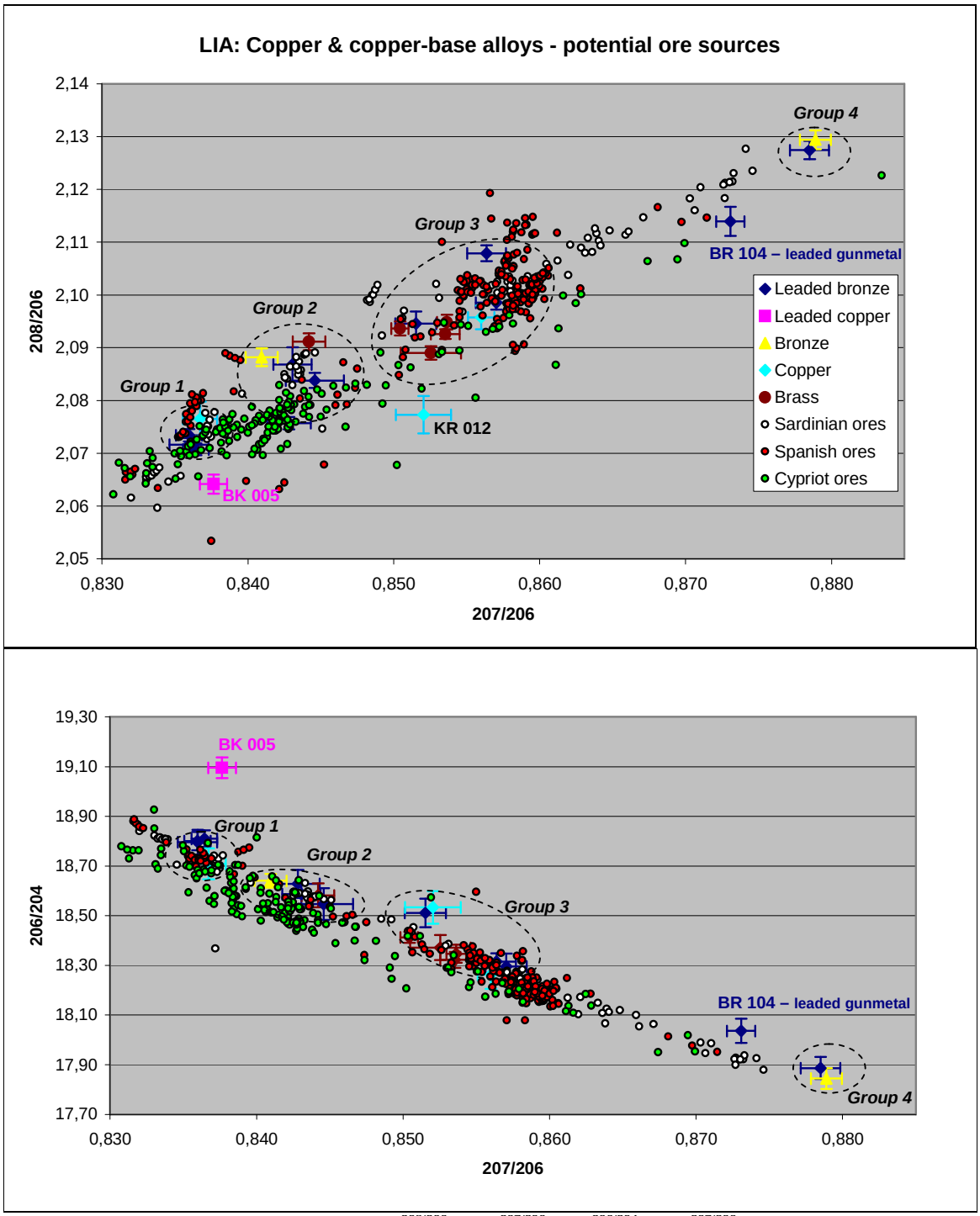


Fig. 62: Scatter plots of lead isotope ratios $Pb^{208/206}$ *vs* $Pb^{207/206}$ & $Pb^{206/204}$ *vs* $Pb^{207/206}$ for all analysed copper & copper-base alloys, with potential ore sources from Sardinia, Spain & Cyprus (the error is indicated by error bars).

The comparison of the obtained lead isotope ratios with the collected data in the database, proved difficult. Many ore field have significant overlap and are indistinguishable from each

other. The plot in Fig. 62 show the three potential sources (Sardinia, Spain and Cyprus) that gave the best match. It has to be stressed however that this is a very basic comparison and that several other ore sources show overlap to a lesser extent. The most prominent amongst these are Turkey and Bulgaria, but only for the samples that plot below the 0,850 $Pb^{207/206}$ value. More research is needed to tackle this problem. Moreover the analyses of the leaded copper-base alloys pose the additional problem that the input of the lead to the make-up of the signature is unknown. The fact that these samples were analysed is because they were submitted for the LIA before the actual composition was determined. Additionally they came from the more 'diagnostic' objects and it was hoped to pinpoint their origin. Of the three source regions potted Fig. 62 the Spanish and Sardinian ores seem to me to provide the best fit.

Several samples show no identification with any of the data in the database. BK 005 does not fit any of the data from the database, not for the lead and not for the copper. Even when a mixed isotopic signatures is considered, i.e. when a contribution of both metals is considered.

The leaded gunmetal bell BR 104 and the two samples in *Group 4* also do not show a good similarity with any of the ore sources. It might argued that the samples from *Group 4*, both from Khor Rori, contain some copper from the Indian Subcontinent making their signature shift towards the Indian ore sources. In the light that KR 009 is probably Indian of origin and the evidence that copper or copper-base alloy were melting at Khor Rori, it is entirely possible that copper from both locations (Roman World and India) were mixed.

As a whole the lead isotope signature from the Khor Rori samples is different of that of ed-Dur. Of the five samples analysed from Khor Rori, four are seen as atypical. KR 009 plots in the Indian source region, the two samples in *Group 4* do not really show a clear overlap with any of the source data and KR 012 is also not really a part of *Group 3*. It is tempting to conclude that the metal supply to Khor Rori is different to that of ed-Dur.

5.9. Interim conclusions copper & copper-base alloys

• **Unalloyed copper**
Unalloyed copper makes up for 22% of the analysed samples. One small lion-shaped bead is made from leaded copper. The nails analysed were made of unalloyed copper. One copper 'altar' bead shows the remains of gilding. This was most probably done by a technique known as *amalgam* or *fire gilding.*

• **Tin-bronze**
Tin-bronze, as might be expected, is the most abundant alloy accounting for 51% of the analysed samples. The bronzes can be further divided based on the tin level present, i.e. low (less than 5 wt% of tin), medium (between 5 & 15 wt%) and high (more than 15 wt%).

The low tin-bronzes are characterized by the fact that they are all heavily leaded. The alloy is left in the as-cast state, since working an alloy with such high lead levels is rather impossible.

Most popular are the medium tin-bronzes. This needs not be a surprise since this alloy suited for many purposed. Within this group functional objects such as vessels are included, next to ornamental objects such are anklets and bracelets.

Only two 'real' high level tin bronzes are attested and these are fragments of a typical kind of mirrors. The high tin levels produce an alloy with a grey/silvery colour, that is hard and brittle, but can be highly polished. The typical black patina described in the literature was also observed. These high tin-bronze mirrors appear in the Roman Empire, but are also known from China. On analytical grounds not one of the regions can be excluded, but considering the whole context at ed-Dur, a Roman origin is more likely.

• **Brass**
The rather large amount of brasses attested in this study (20% of the samples) is surprising. Brass is to be considered a typical Roman alloy that appeared around the 1st c BC and quickly became popular. The average zinc level is rather high and indicative of brass that was remelted only a limited number of times or slightly diluted with copper or bronze. The latter option can explain the small amounts of tin sometimes detected in the alloy. Care was taken not to included any leaded, since only one of the samples is leaded. This is important because the presence of lead hinders the uptake of zinc In general the zinc levels in de ed-Dur brasses are in good accordance with the values published for Roman brasses.

The trace elements (i.e. manganese & iron) strongly suggest that the zinc used in the brass came from smithsonite. Smithsonite was the ore used to produce Roman brass by cementation, whereas the Eastern tradition was centred on sphalerite ores as source of zinc. This makes it highly likely that the brass found at ed-Dur is from Roman origin. Moreover textual evidence from the *Periplus* states that the Romans exported brass vessels to Africa. If brass was exported to Africa, it may also have been exported to other regions.

The metallography shows that almost all brass objects were worked (cold or hot?), a procedure that α-brass is ideally suited for.

Two categories of objects are exclusively made of brass: i.e. the copper-base alloy finger-rings and the ring-pommels. A likely explanation for the choice of brass is the golden colour of the alloy if it contains *ca.* 15-20%. The overall picture is that brass was rather used for decorative purposes.

• **Gunmetal**
Together with brass this is a rather unexpected alloy to turn up in the assemblage studied, still they make up 7% of the copper-base alloys. This alloy does not seem to have been used for a certain class of objects and is most likely not an 'intentional' alloy but the result of recycling. More over the composition of the gunmetal at ed-Dur tends to differ from that of Roman gunmetal. The zinc levels are lower than the tin levels (except for one sample BS 054), this is the opposite of the average Roman gunmetal. Only BS 054 can be considered as a 'true' Roman style gunmetal.

The most likely explanation of for the existence of the gunmetal at ed-Dur would be the recycling of medium tin-bronzes with a small amount of brass. Moreover the bracelet S 0022 has a tin level that is similar to that in the tin-bronze bracelets analysed and this may show that the zinc got mixed in by accident.

• **Lead isotope analyses**
I am fully aware of the fact that the potential of the lead isotope analyses was far from explored to its fullest extend. A more thorough evaluation of these data is first in line for future research and I am confident that more information can be extracted from them. For a moment it was thought to omit this part of the research from this PhD because it got stuck on a fairly basic level. Some interesting result did appear however and this made it necessary to include this first sketch of the results.

The most interesting piece within the dataset did actually not come from ed-Dur but Khor Rori. Sample KR 009 almost certainly originated from ores from the Indian Subcontinent, most probably from the region of Rajasthan. The implication of an attribution of the sample from Khor Rori to an Indian origin is considerable. The *Periplus* and Pliny both state that copper was imported into India from the Roman World and suggest that no indigenous copper was produced. The findings here would contradict that and suggest that at least to a limited extent copper was produced in India, and apparently exported to Oman. This region would have had an outlet via the harbour of Barygaza. Moreover Barygaza is mentioned in the *Periplus* as sending out shipments of copper to the Gulf. The signature of KR 009 fits the idea that India was also producing copper (and its alloys) and even exporting some indigenous copper as well. The dependence of India on imported Roman copper might have been less significant than suggested in the *Periplus* and by Pliny and rather meant to create a surplus than to fill a shortage.

The two other samples that (*Group 4* as defined above) also come from Khor Rori have an isotopic signature that can be explained by mixing Indian and 'Western' copper or in one case maybe lead. The attestation of same crucibles used for copper or copper-base alloy melting at Khor Rori indicates that this is a viable hypothesis.

The attestation of Roman artefacts, e.g. the *patera* and the pedestal, next to objects that are though to be local of design, e.g. the horse spout and the wine set ladle, is interesting. Although the objects themselves point to a different origin, the lead isotope signature shows they may be made from the same base metals. These metals are most likely from ore sources within the Roman Empire.

Towards the interpretation of the ore sources used extracted to extract the metal from, the study is not completed. It can only be mentioned that the ed-Dur dataset shows significant visual overlap with ores from Spain, Sardinia and Cyprus. But several other regions also have ores that provide similar signatures. A more detailed study of the history of all these mining regions during the Roman period is needed to truly tackle this problem.

Most of the brasses plot close together and suggests they were made from similar alloying metals. Designating the brasses to a certain place of origin may prove impossible however.

As a whole the lead isotope signature from the Khor Rori samples is different of that of ed-Dur. Of the five samples analysed from Khor Rori, four are seen as atypical within the studied assemblage. It is tempting to conclude that the metal supply to Khor Rori was different from that of ed-Dur.

Chapter 6. LEAD, SILVER, THEIR ALLOYS & LITHARGE

"One's real life is often the life that one does not lead."

O. Wilde

6.1. Introduction

Lead and silver are treated together here because their history and extractive metallurgy are closely linked. Nowadays about half of the silver produced comes from lead ores but in antiquity this proportion must have been even higher[1]. The number of silver and lead objects or fragments analysed is limited and for that reason the results are presented in the same chapter.

This chapter will give an introduction to the basic production technique of lead from ore, not so much for the lead itself but as a basis to understand the extraction of silver from lead. The lead artefacts and samples are discussed first. Secondly the silver and silver alloys objects and samples are treated and a final part discusses the three *litharge* fragments encountered at ed-Dur. Litharge consists mainly out of lead oxides and is the typical waste product of a silver extraction process. These are quite unique and the first to be identified in the region dating to the period under consideration.

The majority of the coins found at ed-Dur are made from silver or an alloy of silver and copper, sometimes with silver and other times with copper as the main fraction. To be in line with the general structure of this dissertation the coins should be presented in the chapters on their respective alloys. This was not done since to my opinion it seemed more useful to present this artefact group as one unit in a separate chapter (*Chapter 7*) and not to disperse the data of this coherent collection over several chapters. Many simple angles of approach would be severely hampered and to much cross-referencing would be needed. This would not facilitate the study and presentation of the results. The lead isotope data obtained from the few silver coins that could be sampled are however including in the presentation of the lead isotope analyses of silver below. The object as such is of secondary importance when looking for the origin of the silver and determining if more than one ore sources was used. It is only after this has been established that the results can be projected onto the object itself. A second reason is the limited amount of silver objects available for LIA. I hope this explanation justifies and clarifies the decision made here.

[1] Tylecote, 1962: 73.

6.2. Production techniques lead & silver

6.2.1. Introduction

Lead (Pb) is the most abundant of the heavy non-ferrous metals but *native lead* is very rare in nature. The principal lead ore source exploited by man has probably always been the mineral *galena* (lead sulphide, PbS), which is of very common occurrence and is frequently associated with silver ore minerals. Next to galena, two other lead ores can be mentioned: *cerussite* (lead carbonate, $PbCO_3$), a widely distributed secondary ore mineral of lead formed by the action of carbonated waters on galena, and of lesser importance *anglesite* ($PbSO_4$), that normally occurs in the oxidized zone of lead deposits and often surrounds a core of galena.[2]

Silver (Ag) is fairly widely dispersed in minerals in levels less than 0,5% and it is almost invariably found in sulphides. *Native silver* is rare however and is usually found in quantities not worth melting to make larger workable lumps. It generally appears deep underground and if on the surface, it is likely to have been converted into a chloride by traces of chloride present in rainwater. Native silver as such was probably never used and if so it must have been exhausted by historical times because of the limited amounts available. The principal ores of silver are the so-called *dry ores* such as silver sulphide (*argentite* or silver glance, Ag_2S), silver chlorides (*cerargyrite* or horn silver, AgCl) and mixed ores[3] and argentiferous *fahl ores*[4]. These dry ores will yield up their metal by simple direct smelting, without the necessity of a *cupellation process* (see below). Next to this, silver can be found in the natural gold-silver alloy, *electrum*. Pyrite ores can also contain silver, as can *jarosite*[5] ores.[6]

The principle sources of silver in antiquity however were *argentiferous lead ores*, notably *galena, cerussite* and *anglesite*. It is however possible that the dry ores were used more often than presently assumed.[7]

6.2.2. General overview of lead production

Lead ores are wide spread, easy to treat to almost pure metal and its oxide can be reduced at below 800°C in a domestic fire burning charcoal or dry wood[8]. Because of the ease of this process it is unlikely to leave much permanent evidence. As a consequence there is little direct knowledge of the early history of lead, but it seems likely that lead may have been among the first metal to be smelted.[9]

Galena (PbS) is the most common lead ore and undoubtedly it was the main mineral used in antiquity. Generally it contains small quantities of silver (*argentiferous* galena)[10]. Galena is a lead sulphate and should first be reduced to its oxide form by roasting. This is done by gently heated the ore in open trenches with a blast of air. The sulphur-lead compound is decomposed and most of the sulphur escapes as sulphur dioxide gas (SO_2) although some remains as lead sulphate. Because galena however often contains about 80% of lead and is easily decomposed, the roasting step can be eliminated and the ore can be directly smelted under a moderately oxidising atmosphere. The simplified reaction that take place:

[2] Forbes, 1964a: 204; Moorey, 1994: 292; Craddock, 1995: 205; Allaby & Allaby, 1999: 24.
[3] Examples of mixed ores are *pyragyrite* (Ag_3SbS_3), *proustite* (Ag_3AsS_3) and *stephanite* (Ag_5SbS_4) (Craddock, 1995: 211).
[4] *Fahl ores* are to be found in the enrichment zone of copper ore bodies and are in this way similar to the *jarosite* ores.
[5] *Jarosite* is a complex partially oxidised sulphide ore, $KFe_3(SO4)_2.(OH)_6$, that is associated with the enrichment zone of an ore body, between the gossans and the primary deposits. They may have been a significant contributory source of silver in antiquity (Craddock, 1995: 212).
[6] Tylecote, 1962: 73; Forbes, 1964a: 202-203; Moorey, 1994: 232; Craddock, 1995: 211-212.
[7] Craddock, 1995: 211
[8] Tylecote, 1962: 75; Craddock, 1995: 206.
[9] Craddock, 1995: 205.
[10] Habashi, 1994: 46-47.

$$2\ PbS + 3O_2 \rightarrow 2\ PbO + 2\ SO_2$$
$$2\ PbO + PbS \rightarrow 3\ Pb + SO_2$$

and

$$2\ PbS + 4O_2 \rightarrow 2\ PbSO_4$$
$$PbS + PbSO_4 \rightarrow 2\ Pb + 2\ SO_2$$

The lead that is produced falls to the bottom of the fire. This is known as the double decomposition reaction, since both components (the sulphide and the oxide) are decomposed to give lead. The disadvantage of his process is that more lead is lost to the slag.[11]

Cerussite ($PbCO_3$) was heated with charcoal to remove the oxygen so that it passes off as gas[12]:

$$PbCO_3 \rightarrow PbO + CO_2$$
$$PbO + CO \rightarrow Pb + CO_2$$

Apart from the extraction of the silver (and gold if present) primary produced lead was probably never refined. The simple nature of the smelting operation had the advantage of providing a relatively pure metal, as far as base metals were concerned.[13] The metal obtained in this way is sometimes called *crude lead* (other terms are *work-lead*, *base-bullion*) and can contain substantial amounts of silver next to a number of impurities (among them antimony, arsenic, copper, tin, etc.). To ameliorate the de-silvering process a second phase of purification can be introduced. An additional advantage of removing these impurities is that the lead become more malleable, hence the name *soft lead*[14].

This step in the process is called *liquation* and is based on the fact that lead has a lower melting point (327°C) if compared to most of the co ntaminants. By slowly melting the crude lead at a low temperature the lead is separated and flows away, leaving behind the *dross*, a mixture of copper, lead, antimony and arsenic. The silver however remains in the lead. In an additional oxidation phase the lead was melted and exposed to a current of air. The impurities left over by the liquation oxidized first and the dross formed was skimmed off from time to time.[15] This process is sometimes termed *scorification*[16].

The process does seem to have been very different depending whether ore was smelted primarily for the lead or for the silver it contained. In the hearths where lead was the principal product, the reducing conditions were poor and the temperatures low resulting in a poor yield. The lead produced however was of high quality because the harmful impurities were not reduced to metal. They went into the slag instead (i.e. iron or copper minerals) and other impurities (e.g. arsenic) were oxidised and went out with the fumes. If the objective was to produce silver then the lead ores were smelted under much more rigorously controlled conditions to ensure that all the silver minerals were reduced and absorbed by the lead.[17]

[11] Tylecote, 1962: 75; Forbes, 1964a: 227-228; Healy, 1978: 179; Craddock, 1995: 206.
[12] Moorey, 1994: 292.
[13] Tylecote, 1962: 79.
[14] Forbes, 1964a: 229-230; Healy, 1978: 180-181.
[15] Forbes, 1964a: 229-230; Healy, 1978: 180-181. This process was already used in Roman Period for the extraction of silver from copper ore.
[16] Bayley & Eckstein, 1997: 109.
[17] Craddock, 1995: 211.

6.2.3. General overview of silver production

• **Cupellation of lead to obtain silver**

Silver can be extracted from argentiferous lead ores in a two-stage process. The most common lead ores containing reasonable quantities of silver are, as mentioned above, galena and cerussite. These ores have to be smelted under reducing conditions to produce argentiferous lead. Lead has the property to absorb silver but it is not mixable with it. In the second step the silver that was absorbed had to be separated by a process called *cupellation*, i.e. the removal of the lead by selective oxidation under the form of *litharge* or lead oxide. Silver stays almost entirely unaffected by this oxidation process.[18]

This process can be preformed in an open clay vessel or a small hearth with a low wall, screen or hood of clay. Small-scale cupellation could be carried out on a small shallow dish, or disc, known as a *cupel* or *test*. The crucible was filled with the molten silver-enriched lead that had to be treated and under an oxidizing air blast the lead was converted to an oxide. It took with it any other base metals (e.g. copper, antimony, arsenic, tin, iron and zinc) or other impurities present. In this way it removed them from the silver that remains un-oxidized. The hearth or vessel had to be shallow so that the maximum amount of molten lead was exposed to the air provided by bellows and charcoal or wood must be used to maintain the temperature at about 1000-1100°C. Part of the base metals are consumed, or *drossed*, and together with the lead oxides it is necessary to remove them as very little oxygen can pass through this layer once the surface of the metal is completely covered. This results in a very slow oxidation of the remaining metallic lead. Nowadays this happens by careful skimming off or tapping of the lead oxide but in antiquity a different technology was used. The cupellation hearth was lined with porous material so the litharge was absorbed rather than skimmed off the surface and the silver was retrieved as a small button of metal. The litharge cakes often take the shape of these cupels or hearths and show a depression in the top where the molten silver lay. This process could be repeated several times to purify the silver and is very efficient in separating the silver from the impurities.[19]

The most fundamental characteristic of the lining was that it needed to be porous to allow the absorption of the liquid litharge by capillarity. The lining of the cupellation hearth or vessel played a crucial role in the process and materials were selected for their inert behaviour against the liquid litharge. The lining has two major functions. Firstly, as mentioned, its porous nature soaks up the liquid litharge in order to separate it from the molten lead so that the latter is always exposed to air. Secondly it prevents chemical attack of the molten litharge on the hearth itself and causing disintegration.[20]

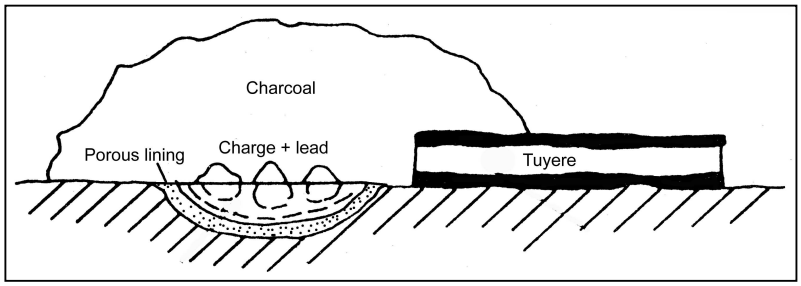


Fig. 63: Cupellation hearth (after Tylecote, 1981a: 111, Fig. 7).

Two large groups of materials used for the porous lining can be distinguished. On the one hand there are linings from carbonate-rich material, like lime, crushed shell or highly calcareous clay (marl). Pliny mentions a special clay, *tasconium*, that was used for ancient cupels. On the other hand phosphate-rich materials could be used like bone

[18] Tylecote, 1962: 79-80; Tylecote, 1976: 166; Bayley & Eckstein, 1997: 108; Pernicka, Rehren & Schmitt-Strecker, 1998: 123.
[19] Tylecote, 1962: 79-81; Tylecote, 1976: 61-62; Hodges, 1968: 92-93; Moorey, 1994: 218 & 233; Bayley & Eckstein, 1997: 108; Ingo, Agus, Ruggeri, Amore Bonapasta, Bultrini & Chiozzini, 1997: 413; An., 2001: 19; Kassianidou, 2003: 205; Bayley & Eckstein, 2006: 146.
[20] Pernicka, Rehren & Schmitt-Strecker, 1998: 125, 131.

ash. This is certainly attested from the 1st millennium AD onwards and the Romans seem to have used this material from the 1st c AD in certain occasions.[21] The first group generates litharge cakes with high calcium and silica values, the second group has high phosphorous levels[22].

The reason that calcium-rich material was preferred for lining is that lead oxide behaves very aggressively towards silica, easily forming a lead silicate glass. This reaction may cause the breakdown of a silicate-rich hearth linings or vessels if too much lead oxide is present. The silica content of the lower parts of the litharge cakes may however be as high as 30 wt% and sometimes a thin layer of silicate material is present at their base. This points to a silica-rich base supporting the calcium-rich lining.[23] This problem was later solved by using bone ash since this did not react with the litharge but only absorbed it, resulting in better separation.[24]

Cupellation was not always a crucible process. Simple hearths were in use and may have been a small hole in the ground. The Romans used bowl furnaces, excavated on the spot with air blown over the surface through tuyeres (Fig. 63). Small-scale cupellation was carried out on small shallow dishes and even potsherds could be used. These ceramic cupels have highly coloured lead-rich vitrified surfaces.[25]

Although it is sometimes said that the silver was *floating* on top of the liquid lead oxide this is actually impossible because silver is heavier than litharge (10,5 g/cm³ *versus ca*. 9,5 g/cm³ [26]). The explanation is that the liquid litharge was absorbed by capillary action into the porous lining. In the same time it attacked the clay component of the recipient to form lead silica. In reality most of the cakes are really the lining impregnated and reacted with the litharge, with the relict structure of the linings preserved in the cakes. It was on this that the silver sat as a coherent pool.[27] A second factor is that silver metal has a much higher surface tension than the lead oxides, making it easier to stay on top of the hearth. Only when the lining contains relatively large cracks the molten lead metal could penetrate the hearth material, resulting in the loss of noble metals dissolved in the lead. To prevent this, the hearth lining was prepared and applied with the necessary care.[28]

Possibly the silver enriched lead was first subjected to a process to further concentrate the silver content. One passage of Pliny has been used to suggest that the Romans had invented the *Pattinson process* for de-silvering lead by which precious metals are concentrated nowadays. With this technique, crystals of pure lead crystallising from the molten charge are removed, leaving a liquid that becomes more enriched in silver as more lead crystals are skimmed of. This process will go on until the remaining lead contains about 2,4% of silver. Then the remaining molten metal will set all at once. By pouring off the molten metal before this happens the silver is concentrated as far as possible and the cupellation process can then be used to de-silver the enriched lead.[29]

Over time the level to which argentiferous lead could be refined changed. During the Aegean Bronze Age the lowest limit considered economically interesting seem to have been about 0,04 – 0,07% of silver[30]. Pure lead remains rare until Roman times[31], but the silver levels generally dropped to about 0,01% in the Roman Empire but they show great variation (from

[21] Forbes, 1964a: 173; Craddock, 1995: 229; Rehren & Klappauf, 1995: 19; Pernicka, Rehren & Schmitt-Strecker, 1998: 125, 131.
[22] Craddock, 1995: 229; Rehren & Kraus, 1999: 263.
[23] Bayley & Eckstein, 1997: 108 & 111; Bayley & Eckstein, 2006: 146.
[24] Bayley, 1992: 6.
[25] Bayley, 1992: 6; Habashi, 1994: 49.
[26] Hess, Hauptmann, Wright & Whallon, 1998: 60.
[27] Craddock, 1995: 223.
[28] Rehren & Kraus, 1999: 263; Pernicka, Rehren & Schmitt-Strecker, 1998: 125.
[29] Forbes, 1964a: 230-231; Lang & Hughes, 1984: 81.
[30] Pernicka & Wagner, 1982: 424; Pernicka, Rehren & Schmitt-Strecker, 1998: 129; Rehren & Prange, 1998: 189.
[31] Forbes, 1964a: 208 & 225.

0,002 till 0,026%) depending to some degree on the skill of the workmen[32]. According to R.F. Tylecote however the limit of economic de-silvering was only about 0,06% for the Roman period[33]. The same phenomenon of variation is also seen during the European Middle Ages (from 0,0004 to 0,03 % of Ag)[34].

The identification of de-silvered lead is however not always as straightforward as thought. Lead with low silver content can also be the result of smelting a silver-poor primary ore. A second possibility is the recovery of the lead used during the cupellation process by re-melting the litharge. The resulting lead would contain significant quantities of metals such as arsenic, bismuth, antimony and copper (which render it much harder) but low levels of silver[35].

K. Butcher and M. Ponting mention that evidence of traditional silver production tells us that silver directly from the cupellation hearth was, on average, only 94% pure, the remainder being mostly lead. An additional refining in a smaller furnace or test was needed. The second refining process required considerable skill to produce silver that was about 98% pure.[36] Th. Rehren and K. Kraus state however that it is generally assumed that when the refining of debased silver is done carefully the silver will contain as little as 1% of copper and lead[37].

- **Cupellation of debased alloys to retrieve silver**

The mechanism to extract silver from a debased alloy works just in the same way as the cupellation process described above and involves the melting of the impure silver with lead metal. The melt was oxidised by blowing air across it, forming litharge which then oxidised any base metals that were present and dissolved them, before being absorbed by the hearth lining. Silver dissolves readily in lead metal, but is insoluble in litharge. The pure silver was thus left behind on the surface of the hearth at the end of the process.[38]

The metal most often alloyed with silver in antiquity was copper and the obvious reason to remove the silver was the metals' value. This technique was often used to recover the silver from debased coinage. Instead from starting with argentiferous lead ore the debased alloy is molten together with three to four times its weight of lead and stirred so the lead absorbed the silver from the copper. If necessary lead had to be added at frequent intervals to assure that all the silver was taken from the copper. Copper and lead are virtually insoluble and when stirring stopped the two molten metals would separate, the silver-enriched lead settled underneath the de-silvered copper. These cakes were heated on a hearth at a temperature which allowed the lead (containing the silver) to melt and drain off, leaving the copper behind. This is called *liquation*.[39] The step of liquation is only necessary when the alloy to be treated was severely debased, especially when tin and copper (e.g. tin-bronze) are present in too high concentrations. In this case the success of the cupellation depended on the preparation of the right bullion.[40]

The temperature required depends on the degree of debasement of the silver. Oxidation produced litharge, which reacts with the copper, giving a melt with a eutectic of 680°C at just under 20 wt% of CuO_2. Relatively low temperatures produce complete oxidation of impurities and kept the losses of the volatile lead to a minimum. As lead and copper are oxidised, the proportion of silver in the melt increased so the process temperature also had to increase

[32] Friend & Thorneycroft, 1929: 115-116; Healy, 1978: 180; Habashi, 1994: 48; Pernicka, Rehren & Schmitt-Strecker, 1998: 129.
[33] Tylecote, 1976: 140.
[34] Friend & Thorneycroft, 1929: 115-116; Pernicka, Rehren & Schmitt-Strecker, 1998: 129.
[35] Tylecote, 1962: 82; Craddock, 1995: 211; Rehren & Prange, 1998: 188.
[36] Butcher & Ponting, 2005: 187.
[37] Rehren & Kraus, 1999: 263.
[38] Bayley & Eckstein, 2006: 145.
[39] Tylecote, 1962: 80; Craddock, 1989: 208-209; Bayley & Eckstein, 1997: 108.
[40] Craddock, 1995: 229; Rehren & Kraus, 1999: 263.

towards 1000°C. Temperatures this high were only ne eded towards the end of the process.[41] The silver-enriched lead was than cupelled in the same way as described above.

The practise of cupellation goes back to as early as the Early Bronze Age and it is still a commonly used method to assay noble metal, mainly for low concentrations. Though the process is chemically quite simple, the success depends on the careful workmanship. The control of temperature is important. The operation was controlled by the observations of colours and the formation of the yellow litharge crystals on the cold parts of the crucible.[42] In a first phase cupellation was used to produce silver from argentiferous lead ores, but later cupellation also became a refining process, notably during the Roman period[43].

In the *Ain-I-Akbari* an example of how debased silver can be treated is preserved and goes as follows:

> "*They dig a hole, and having sprinkled into it a small quantity of wild cow dung, they fill it with the ashes of* mughita *(a kind of acacia) wood, then they moisten it and work it up into the shape of a dish; into this they put the adulterated silver, together with a proportionate quantity of lead. First they put a fourth part of the lead on top of the silver and having surrounded the whole with coals, blow the fire with a pair of bellows, till the metals are melted, which operation is generally repeated 4 times. The proofs of the metal being pure are a lightning-like brightness and its beginning to harden at the sides. As soon as it is hardened in the middle they sprinkle it with water when flames resembling in shape the horns of wild goats, issue from it. It then forms itself into a disc and is perfectly refined.*"[44]

6.2.3. Geological context Gulf region

Argentiferous galena sources in the mountains of Oman Peninsula have long been known. D.T. Potts suggests that these sources may have been among those that produced the lead found at ed-Dur[45]. Lead and zinc deposits are situated in Wadi Hawasinah (between Suhar and Muscat) and at Saih Hatat (south of Muscat)[46]. The rest of the Arabian Peninsula is notoriously poor in silver and lead ores. Exceptions are the silver-rich galena deposits in the Midian mountains to the east of the Gulf of Aqaba. The Nabataeans exploited these sources and the silver that they produced was exported.[47] Fragments of cupels dating to the Abbasid period were found at An-Nuqrah South, Ash Shumta I, Al Koom al-Gharbi, Al Koom al-Sharki and Mawan, all situated to the NE of Medina (Saudi-Arabia). The mineralization of the Al-Naqrah South consists of polymetallic sulphides (Cu, Pb, Zn, Ag). According to preliminary research, copper was the main metal extracted, but gold and silver are present in economically profitable amounts and were worked as secondary metals. It should be noted that at the present the silver content of the ores is too low to be worked, but the cupels encountered show that in the past, by now exhausted, ores might have been worked.[48] Adding weight to this hypothesis are the manuscripts of Arabian geographer Muqaddasi (10th c AD) mentioning the export of lead from Yemen to Oman.[49] This statement is repeated in a later Persian manuscript from the 14th c AD by al-Mustaufi who notes the production of gold and silver in S-Arabia[50].

[41] Bayley & Eckstein, 2006: 146.
[42] Kuppuram, 1989a: 181.
[43] Rehren & Kraus, 1999: 265.
[44] Kuppuram, 1989: 183. The *Ain-I-Akbari* is a 16th c AD Persian text written for the Indian ruler Akbari.
[45] Potts, 1990b: 287.
[46] Tosi, 1975: 202.
[47] Forbes, 1964a: 210; Moorey, 1994: 234-235.
[48] de Jesus, Al-Surgiran, Rihani, Kesnawi, Toplyn & Incagnoli, 1982: 63-77.
[49] Weisgerber, 1980b: 119.
[50] Weeks, 2004b: 17.

In Persia many important galena deposits are found. Ancient mines are known in Khurasan, Kerman, Fars, in the Central Mountains, etc.[51]. Important deposits are also found in Armenia and Kurdistan[52]. Other significant sources for silver and lead are to be found to the north of India in Bactria and Transoxania which have been worked from the Persian period onwards and possibly already earlier but the evidence for that is slim. Afghanistan and the regions to the north of the Oxus contain many deposits of lead ores, which like those in Iran are always argentiferous.[53]

There are many argentiferous galena deposits on the Indian subcontinent, some of which have been worked for a very long time[54]. Remains of cupellation were found in the silver mine of Argucha and Dariba in the Province of Rajasthan dating to the 3rd c BC. At the sites of Agucha evidence was found of the extraction of lead, silver and zinc. The mine is however dated to the 3rd century BC based on only 2 radiocarbon dates. The dates of the mine of Dariba go back to the 2nd millennium BC, but the period of most intensive work is after 500 BC.[55]

Anatolia has the greatest quantity of geologically identified silver-bearing ores of any of Mesopotamia's neighbours and probably was the major source of the lead used in Mesopotamia[56].

During the Roman Period the *silver* deposits in Spain were extensively exploited (jarosite ores), notably at the Rio Tinto mine. Oxidized ores were more widely distributed and the best-known area of exploitation in antiquity probably is Laurion in Greece. Other areas with argentiferous lead sources are quite widespread and mines known to have been active in antiquity are recorded for Tuscany, Sardinia, central France, southern Pyrenees, central and northern Serbia and Macedonia. Germany also has a number of sources (e.g. Thuringerwald and the Erzgebirge), but these were outside the Roman imperial frontiers and so presumably did not supply the Roman Empire[57].

Also for the extraction of *lead* Spain was well known in the Roman period. The central source was Carthage Nova, and other supplies were obtained from the Ebro Valley, Baetica, Gaul and other regions, including Capraria of the Balearic Islands and especially from Britain[58].

[51] Forbes, 1964a: 212-213.
[52] Momenzadeh, 2004: 16.
[53] Forbes, 1964a: 212-213; Habashi, 1994: 23; Moorey, 1994: 234-235.
[54] Forbes, 1964a: 211; Habashi, 1994: 23.
[55] Craddock, 1995: 223; Craddock, Freestone, Gurjar, Middleton & Willies, 1989: 58-59.
[56] Moorey, 1994: 234-235 & 293.
[57] Butcher & Ponting, 2005: 189.
[58] Warmington, 1974: 267-268.

6.3. Lead & alloys

6.3.1. Lead

Lead metal has a low melting point of 327°C and lead ores (oxides) can be reduced to lead metal below 800°C. Lead has a high specific gravity, is very soft and easily formed into sheets.[59] As mentioned before the most important function of lead was probably as a source of silver and its use in purifying noble metals. It was only from the Romans onwards that lead took a more prominent role in civil use, e.g. water pipes, etc. A second important function was as an alloying element to copper (or copper-base alloys) to improve the casting properties and when alloyed with tin it functioned as a soft solder. Lead was also used from early times in cosmetics and in pigments and later on in the production of glass and glazes.[60]

Artefacts made of lead are among the oldest metal objects known, and lead was probably the first smelted metal that was not already known in its native state. Compared to the other early metals in antiquity, i.e. gold, silver and copper, lead does not have an aesthetic appeal or superior working properties. It can however be easily melted, cast and shaped into form. Hence, the very early uses of lead was mainly for small implements and figurines, fittings for mending broken pots, etc., while later the softness and high specific gravity of the relatively cheap metal led to its use also as weights, net sinkers, bullets, etc.[61]

The first lead object reported from the Oman Peninsula comes from Mleiha and dates to the PIR-B period. It is described as a flat base of a vessel or goblet[62]. At ed-Dur however lead appears in some quantity for the first time in the history of the region. The initial database composed during the excavations contained 34 lead objects and 75 more were entered as samples. Bringing the total amount of registered fragments at 109. The total number of individual fragment is still higher since many of the sample bags contain more than one fragment.

Most of the objects are not very spectacular and are just flat strips of lead (see Fig. 64 for some examples). One group of 29 folded pieces was described as 'net sinkers' and to this 18 additional pieces can be added that were recognised among the samples as net sinkers. The identification as net or line sinkers is based on the resemblance that they have to the modern weights used by fishermen. A recent publication on the tumuli of mounds near pylon T158 show two similar folded strips of lead[63]. Other lead objects were three spindle whorls, a dromedary pendant, two shell-shaped pieces and a bell-shaped artefact with iron ring attachment resembling a pearl divers weight. During a visit to the Museum of Umm al-Qaiwain one extra sample was taken from Z 019 (from the Danish mission) because it was a rather large piece of lead. It had an oval, bowl-shape (*ca*. 12 x 7 x 3 cm). The bottom side was rather rough as if the molten lead was poured in a roughly hewn stone hollow (in *farush*?). Two exceptional objects need special mentioning however, a *bulla* (BS 269, Fig. 64) and an 'ingot'-like object (S 0024, Fig. 64), since they are part of the artefacts that were analysed in this study.

A *bulla* is a coin or an Indian imitation of a Roman coin manufactured in clay, lead, gold, silver or faience. These coins were often *denarii* and *aurei* of the Roman emperors Augustus (31 BC – 14 AD) and Tiberius (14 – 37 AD), although all sorts of human, animal, floral and geometric motifs were pictured. The example of ed-Dur depicts a human face, something that appears to have been typical of the first centuries AD. The *bullae* had two loops at the back so they could be attached to something, a feature also seen on BS 269. They were

[59] An., 2001: 18.
[60] Moorey, 1994: 293.
[61] Rehren & Prange, 1998: 183.
[62] Mouton, 1992: 76.
[63] Jensen, 2003: 144-145.

primarily used as decorative pendants but might have had other functions as well. A small part (one of the attachment loops) of this object was sampled. The fact that this item surely was made in India makes it very valuable as a 'standard' to compare the other lead fragments of ed-Dur too.

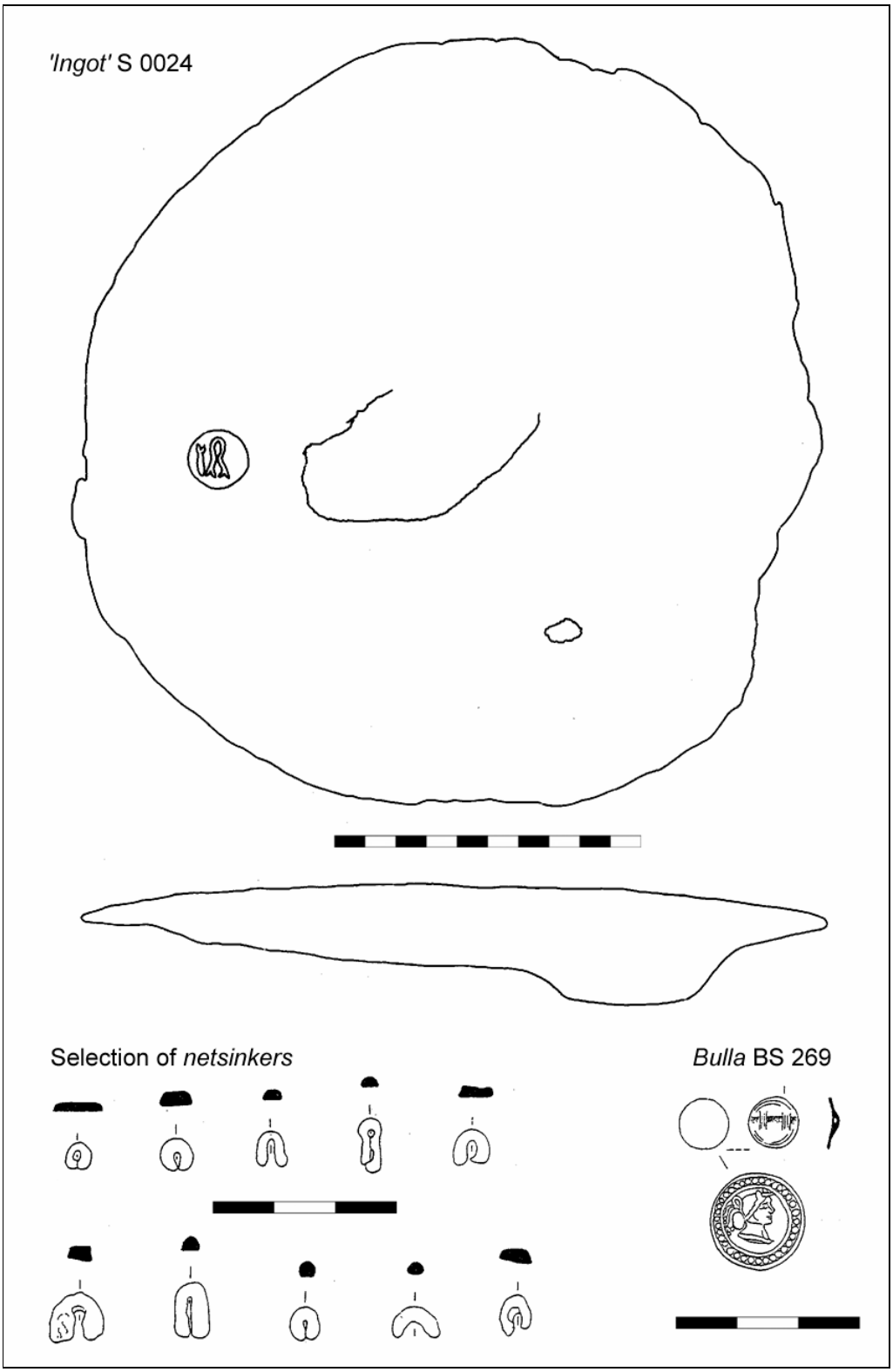


Fig. 64: Drawings of some sampled lead objects.

Before the official excavations started at ed-Dur, an amateur archaeologist picked up a large disc-shaped lead object. Luckily it was possible to obtain a drilling from this object (new registration number S 0024), in order to analyse the lead isotopic composition[64]. The object is possible an ingot (a tradable form and amount of lead) with a diameter of *ca*. 24 cm and is provided with an imprinted monogram. The same/similar monograms were also noticed on some of the SE-Arabian coins and on some pottery sherds. It resembles monograms on coins from Characene and Seleucia, and on an *intaglio* of a Characenean finger-ring found at Kharg. The meaning of this monogram is unfortunately unknown but it could be the abbreviation of Attambelos, a Characenean king. The monogram is an important additional clue in the provenancing of the object and the lead used on the site of ed-Dur. If this lead disc indeed is an ingot, it is direct evidence that lead was imported in 'bulk' and used to manufacture the desired objects on site. An activity that is very easy due to the low melting point of lead. This object can also serve as a 'standard' on which the other lead fragments can be checked.[65]

From all the lead samples brought back to Belgium, 12 were selected for SEM-EDX analyses. The selection was based on a visual examination. Three categories could be distinguished:

- Pieces that looked as if they had been molten and showed a flowing structure. These probably represent leftovers after casting an object, etc. It is interesting to mention two lead objects in the shape of a shell (BS 140 & M 085) that might be the result of poring some excess lead in a 'recipient', in this case a shell.
- Although lead is not a particularly noble metal, it is relatively stable under atmospheric conditions. It is converted immediately with a protective coherent corrosion layer of basic lead carbonates, reducing further corrosion[66]. Two colours of oxides can be observed on the lead pieces. The majority has a white patina, but some fragments had a brownish oxide skin. Based on the idea that this might be the result of a different base metal, examples from both types were selected for further analyses.
- The last group are actual objects or processed fragments, e.g. net sinkers, folded strips of lead, etc.

In a first trail the pieces were cut with an abrasive water-cooled wheel, but this was not an appropriate technique since too much lead was lost and/or got stuck on the wheel. The sampling method was altered and pieces were cut of with a small iron hacksaw or a snap-off blade knife instead. The samples were taken carefully so no corrosion products were present. This corrosion skin might contain elements that were attracted from the surrounding soil and give results that are unrepresentative for the original metal.

The samples retrieved were then mounted in bakelite and polished. The polishing caused some problems since the grains from the grinding paper and the diamond paste got stuck in the soft lead matrix. A second method was tried and the lead was treated in the same way as magnesium. The results were better but they still were not satisfactory, still too much polishing paste got stuck on the surface. To resolve this problem contact was made with Prof. M. Verhaege of the non-ferrous lab and he proposed electrolytic polishing and etching of the samples. This had the desirable effect and a flat 'clean' surface was prepared for SEM-EDX analyses.

[64] This disc is part of a private collection, the owner was however so kind to provide a drilling (registered as S 0024) for which I would like to express my sincere gratitude.
[65] The information of the three last paragraphs are part of the PhD of A. De Waele on the small finds by, for further details I would like to refer to her work.
[66] Rehren & Prange, 1998: 183.

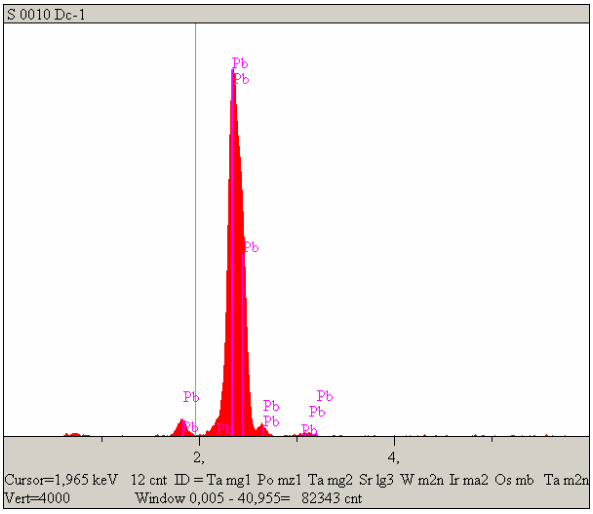


Fig. 65: EDX-spectrum of analysis on S 0010 D.

The EDX results were all similar and only gave lead peaks in the spectra (e.g. Fig. 65). The automatic assessing via the software sometimes found other elements, but they were all in very low wt% (less than 0,2) and not considered any further. The only elements that systematically turned up as a major element was technetium (Tc). This was a very improbable determination since it is an artificially produced element. This was due to peak overlap and the software seemed incapable of separating the two elements. As a double check some of the samples were also reanalyse on a Phillips ESEM. The EDAX software program was able to distinguish both elements and the Tc was not quantified. In the next analyses the element Tc was removed manually. The 12 analysed samples all gave the same picture, neither contained other elements then lead (not even at minor elemental level). Based on these analytical results it was concluded that they were 'pure' lead. If any information on the trace elements is to be gathered, SEM-EDX is of no use and ICP-MS should have been used.

In conclusion we can say that the EDX-analyses did not provide any other information then that the samples all were unalloyed lead. Any other elements present were under the reliable detection level of the technique and therefore omitted from quantification.

6.3.2. Lead as a major element in alloys

A second set of samples has lead as a major alloying element. In this part we will not discuss the *leaded* copper-base alloys since these were already discussed in *Chapter 5*. The samples presented here are actually tin-lead alloys since the amount of tin is higher than the fraction of lead. Because the number of samples is so limited and lead is still a major alloying element, they are included in this chapter.

Two broad groups can be distinguished among the ancient alloys that contain lead and tin, being *pewter* and *soft solders*. There is no clear distinction between the two but the term pewter is used for cast objects.

• **Pewter**

In ancient contexts the term pewter is used for an alloy that has as the basic component tin and lead as the second major element. Sometimes 1 to 5% of copper is also added to increase the hardness of this alloy, e.g. for Roman spoons[67]. The relative high lead percentages in ancient pewter were rather dangerous since it can cause lead poisoning and therefore lead was replaced by antimony from the 19th c AD onwards[68]. A standard modern pewter composition may contain 15-30% of copper and 5-10% of antimony, with the remaining percentage being tin[69].

There are several motives why an alloy such as pewter was produced, next to the accidental contamination of tin with other metals during recycling[70]. The original reason to add lead may

[67] Brownsword & Pitt, 1983: 119; Beagrie, 1989: 171 & 169.
[68] Smythe, 1937: 263; Tylecote, 1962: 67-68.
[69] Scott, 1991: 142.
[70] Beagrie, 1989: 172.

have been to make the tin more corrosion resistant and to prevent a process known as *tin-disease*. It is a well-established fact that the number of tin objects encountered during excavations is very limited, due to the poor corrosion resistance of pure tin. The result of corrosion is a powder of tin-oxide, easily overlooked. There is evidence that the addition of at least 5 to 10% of lead to tin slows this process down, resulting in the fact that pewter is more often encountered on archaeological sites.[71] The lead slows down the corrosion process but does not stop it and also pewter disintegrates over time, especially in dry conditions.[72] A *second* important factor was of a more technical nature. Adding lead improves the hardness and the casting properties of the alloy. A *third reason*, from an economic point of view, is the fact that lead has always been cheaper than tin, so adding lead would reduce the total cost of the base materials[73].

Although there might have been standard ratios of pewter composition in the past, the tin contents in for example Roman vessels ranges between 95% and 40%[74]. Next to the accidental contamination mentioned above, conscious recycling of pewters would result in ratios different from the original. There would have been few ways of assessing the tin content of scrap pewter. Once a significant portion of scrap was introduced into the smelt, its composition would become largely unknown.[75] Some broad lines can be seen in different alloys analysed:

- Alloys with 5 to 10% of lead (see above) were probably used to partly prevent tin-disease[76]. High-tin pewter was also used to make objects similar to contemporary silver objects, because of the similar colour and lower cost[77].
- Alloys with 10 to 20% of lead were used for wares such as pots or flagons[78].
- Alloys with about 20% of lead (4:1 ratio) were used for large bowls and all plates[79].
- A ratio of about 3:1 (25% of lead) was often used to cast larger vessels. These objects needed a shorter solidification time because of their large surface area. This ratio approaches the eutectic point of the tin-lead system (61,9% of tin - 38,1% of lead), being the point where an alloy has its lowest melting temperature (183°C) and fastest transition from liquid to solid. Using this eutectic point or just above it, gives the best casting.[80]
- A 1:1 ratio was mostly used for small vessels (dimensions less than 20 cm)[81].
- Late medieval period ware could contain much higher levels of lead, in some cases as much as 75%[82].

Pewter dating to the Roman period is mainly found in Britain and some examples across Belgium, the Netherlands and France. The rest of the Empire is almost completely pewter-free. They mainly originate from Late Roman contexts of between the 3rd – 4th c AD and dates of 42 – 49 AD are exceptionally early.[83] From the medieval period onwards the alloy was much more widely used for the manufacture of domestic metal ware[84]. That all literature mentioned here is from Europe and the Roman period is due to the fact that there are no analytical data available (yet?) from Near Eastern contexts[85]. One piece of 'proto-pewter'

[71] Smythe, 1937: 255.
[72] Smith, 2003: 7.
[73] Tylecote, 1962: 68-69.
[74] Smythe, 1937: 255.
[75] Pollard, 1983: 86-88; Beagrie, 1989: 172.
[76] Smythe, 1937: 263.
[77] Beagrie, 1989: 173; An., 2001: 21.
[78] Beagrie, 1989: 169.
[79] Pollard, 1983: 88; Smith, 2003: 4.
[80] Hughes, 1980: 42; Pollard, 1983: 86; Smith, 2003: 5.
[81] Pollard, 1983: 88; Smith, 2003: 4.
[82] Beagrie, 1989: 169.
[83] Beagrie, 1989: 175; Smith, 2003: 2 & 7.
[84] Brownsword & Pitt, 1983: 119.
[85] Moorey, 1994: 293.

was however recently reported from Tepe Yahya (75% Pb & 25% Sn) dating to 1800-1400 BC.[86]

• **Soft solder**

The difference between pewter and a soft solder is not well defined if only the alloy composition is taken into consideration, but there is a clear difference in use. Solders are used to attaching different metallic parts to each other or to repair objects. A solder can be defined as a metal, or more usually an alloy, with a lower melting point than the metal(s) that it has to join together[87]. Two large groups of solders can be distinguished *soft* and *hard solders*. Both have the same function but are different in alloy composition and working temperature. Here only the soft solder is considered, since this is an alloy of tin and/or lead.

Soft solders are composed mainly of tin and/or lead. The union of the different elements is done without fusion between the parent metal and the applied metal. The solder actually is nothing more than *metallic glue*. This is a low temperature process, below 430°C (e ven as low as 183°C in some cases). [88]

In the Roman period, soft solders were used to attach *silver foil* to small decorative copper-base alloy items with rather complex shapes. This gave the impression that they were of solid silver. The silver foil was bonded by a continuous layer of solder, not just patches at key points. This layer was obtained by coating the object with molten solder that was afterwards allowed to cool. Silver foil was worked to fit the object closely. The object was then heated gently until the solder flowed. To plate all surfaces, more than one piece of silver foil was needed. This method produces a good silver imitation, but is not strong enough to withstand working after the foil is applied. This technique later became known as *close-plating.*[89]

• **SEM-EDX results**

All fragments listed in Table 25 were too severely corroded (high O and Cl levels) to consider the SEM-EDX data as representative of the original composition. All contained lead and tin as their major components however, making them either pewter or soft solder. These alloys are not very corrosion resistant, hence the poor results.

Reg. nr.	Area	UF	Sq	Loc	Team	Description
AF 137	AF	2502	II 1	-	British	Large shapeless fragment
AO 002	AO	4212	-	T 5121	British	Underneath 'button'
AP 009	AP	4163	-	T 5102	British	Flat fragment, rim vessel?
AW 037	AW	4541	XVI 5-6	T 5437	Belgian	Copper-base alloy appliqué, inside filling
K 066	K	358	VIII 7-8	T 904	British	Flat bent over fragment, rim vessel?
M 072	M	645	IV 4	R 1114	Belgian	Shapeless fragment
M 075-2	M	-	-	-	Belgian	Fragment from underneath nail head/button

Table 25: Coordinates of lead-tin objects and samples.

In three cases we can certainly speak of a solder (AO 002, AW 037 & M 075-2). These analysed fragments came from the inside of a 'button', the inside of an 'appliqué' and from underneath a nail head. The large shapeless fragment AF 137 is also likely to be a piece of unused solder. One fragment only contained tin (K 066), which is exceptional. The fragment could not be attributed to an object, but looks like the bent over rim of a small vessel, a purpose for which tin is not very suitable. The only point we can make here is that tin-lead alloys were present at the site and that soft solders were certainly used. The question about the tin-lead ratios has to remain unanswered. One flat small fragment (AP 009) might be part of a vessel, although this is far from clear, and could therefore be made of pewter. Taking the

[86] Thornton, Lamberg-Karlovsky, Liezers & Young, 2002: 1457.
[87] La Niece, 1993a: 202.
[88] Lang & Hughes, 1984: 84.
[89] La Niece, 1993a: 202 & 204.

late dates of the majority of Roman pewter into account and the absence of any pewter reported from the Near and Middle East, makes an determination as pewter very unlikely.

Reg. nr.	Alloy	Description
AF 137	Sn/Pb	Fractions *ca.* equal
AO 002	Sn/Pb	Fractions *ca.* equal → probably solder
AP 009	Sn/Pb	Fraction Sn larger than lead
AW 037	Sn/Pb	Fractions *ca.* equal → probably solder
K 066	Sn	No Pb detected
M 072	Sn/Pb	Fraction Sn larger than lead
M 075-2	Sn/Pb	Too corroded → probably solder

Table 26: Compositional data of lead-tin objects and samples.

• **Trace elements by ICP-MS**

L. Weeks suggested that the high sulphur concentrations in four samples that also had a high lead content could be correlated and that further analyses of the lead objects could clarify the relation between these elements[90]. The initial thought was to evaluate this in the trace elemental research by ICP-MS. Sulphur however is a difficult element to measure by ICP-MS. Time constraints and the fact that a completely new procedure had to be developed for a relatively small gain in information, led to the decision to set this part of the research aside.

In order to address the relation between lead and silver, seven lead objects and samples were analysed by ICP-MS for their trace elemental levels of silver. The results are presented in Table 27.

Reg. nr.	µg/g Ag	wt% Ag	Description
BS 269	72,73	0,007273	Indian *bulla*
S 0024	42,91	0,004291	'Ingot'
sBS 1360	5,04	0,000504	Netsinker
sBS 1441 A	11,75	0,001175	Long folded strip
sBS 1466	20,31	0,002031	Flat fragment
Z 019	118,11	0,011811	Large piece of lead

Table 27: Silver levels attested in some objects and samples.

Five samples (BS 269, S 0024, sBS 1360, sBS 1441 A & sBS 1466) have very low silver levels. This would be in accordance with lead that was de-silvered or of course lead originating from silver-poor primary ore. Additionally it could be caused by the lead production from ores at low temperatures. The result being that only the lead is reduced and melted leaving behind all other elements that have a higher melting point. Moreover the idea that the Romans de-silvered lead in consistent way is far from proven. That it was done at certain places is correct, but to generalise this conclusion more research has to been done[91]. Z 019 has a more elevated silver level (0,0118%) but can still be the residue of a de-silvered lead ore. De-silvered lead from the Roman world shows great variation (from 0,002 till 0,026%) but an average of 0,01 to 0,06% is put forward[92].

[90] Weeks, 2004a: 242.
[91] Pers. comm. M. van Nie.
[92] Friend & Thorneycroft, 1929: 115-116; Healy, 1978: 180; Habashi, 1994: 48; Pernicka, Rehren & Schmitt-Strecker, 1998: 129.

L. Weeks suggested that the elevated levels of silver in many of the copper-alloy samples (if compared with older SE-Arabian material) might have been introduced by the lead. The average silver content found in the copper-base objects analysed by him is 0,06% with a minimal detection limit of 0,022%.[93] The hypothesis of silver entering the alloys *via* the lead is not negated by our results. The ICP-MS analyses to show however that the lead found on the site must have a different origin than the lead present in the copper-base alloy samples, otherwise they would much lower levels of silver.

[93] Weeks, 2004a: 243 & 245.

6.4. Silver & alloys

6.4.1. Silver

Silver has a melting point of 961,8°C (which is quite close to that of copper) and is relatively soft. For that reason silver was often alloyed with some percents of copper, which produced a harder alloy.[94]

Finds of pure silver at Ur, Troy and other places, point to the fact that the cupellation process was probably invented in NE-Asia Minor in the 1st half of the 3rd millennium BC. It is significant that silver and lead first appear on excavations at the same time and by 600 BC the cupellation process was well known in many places.[95] The early history of silver as recorded in the archaeological remains might be biased because of the fact that silver is easily converted to its chloride (AgCl). The chlorine is present in chloride-containing surface waters, in rain or salt sprays from the sea, and the result is a whitish-grey mass easily overlooked by excavators.[96]

As mentioned above Arabia is notably poor in workable galena (PbS) deposits. The first silver objects in SE-Arabia date to the later Umm al-Nar Period. They appear as numerous silver beads of various shapes from the sites of Tell Abraq, Hili North Tomb A and Moweihat.[97] The number of silver objects retrieved during the excavations at ed-Dur is limited, but this does not mean that the metal was not well represented. The fact that most tombs were severely plundered gives a distorted picture of the reality. It can be imagined that the precious metals present in the graves were probably among the prime objectives of the robbers. One silver bracelet was found at Asimah 24[98] and recently three silver finger-rings were excavated in Dibba Al Hisn (two of which had a carnelian intaglio inlay) together with some silver beads[99]. All coins studied here (see *Chapter 7*) contain silver and point towards the fact that silver had an intrinsic value. Among the analyses done by L. Weeks, one flat fragment was of silver (AV 415, G 5156). Other chemical elements present were: 2,63% of bromine, 1,65% of gold, 0,18% of bismuth, 0,16% of copper and 0,3% of lead[100].

6.4.2. Silver as an alloying element

Several alloys of silver are known, but for this study only *hard solder* and *billon* are of interest.

- **Hard solder**

As opposed to the previous soft solder which is a tin-lead alloy, hard solder comprise a whole series of silver-copper alloys, hence sometimes the name *silver solder*. This technique of using hard solder is often referred to as *brazing* or *silver soldering*. These alloys are intended as filler metals used to bridge a very narrow space between closely adjacent surfaces of the metal parts that have to be joined.[101]

As opposed to the previous soft solder which is a tin-lead alloy, hard solder comprise a whole series of copper alloys, amongst them silver-copper alloys or *silver solder*. The technique of using hard solder is often referred to as *brazing* although this refers to the use of a copper-zinc alloy as solder. For the silver-copper alloys it is better to use the term *silver soldering* or also *silver brazing*.

[94] An., 2001: 19.
[95] Habashi, 1994: 22-25.
[96] Tylecote, 1962: 73-74; Forbes, 1964a: 203.
[97] Weeks, 2004b: 57.
[98] Mouton, 1992: 172.
[99] Jasim, 2006: 227-228.
[100] Weeks, 2000a: 313.
[101] Coghlan, 1975: 118.

Silver soldering is a joining process where the solder is heated to it smelting point. The composition of a typical hard silver-copper solder alloy is close to the eutectic composition (72% of Ag and 28% of Cu) of the alloy. This is the composition of the alloy with the lowest melting point (*ca*. 780°C). These alloys are intended as filler metals used to bridge a very narrow space between closely adjacent surfaces. They are distributed between two or more close-fitting parts by capillary action. The melting temperatures of the solder range from 450 up to 830°C depending on the exact composition of the solder, and are considerable higher those of soft solders. It was often used to attach a silver foil to a copper object. The working temperature is well below that of the silver foil (about 950°C) or the for example copper-base core (around 1083°C). This kind of solder was used at its liquid temperature, the molten filler metal and the flux, to keep the joining surface clean, interacts with a thin layer of the base metal, cooling to form an exceptionally strong bond. One of the main functions of hard solders in antiquity was to connect several metal pieces to a more complex shape and the joint is much stronger than that obtained by soft-solders. The main difference with a soft solder is that a hard solder does not act as a *metallic glue*, but forms a true metallurgical bond due to defussion.[102]

- **Billon**

Billon is an ill-defined term used for any copper-silver alloy, especially in numismatic contexts where it is used as the base metal and not as a layer to connect two metal parts. Billon is not to be mistaken with *bullion*, a term used to define unprocessed silver or gold. The overwhelming majority of the SE-Arabian coins analysed in *Chapter 7* are made of some sort of billon.

- **SEM-EDX results & optical microscope observations**

Nine of the samples analysed by EDX turned out to be from silver or a silver alloy. They are listed in Table 28. The compositional results are given in Table 29 and drawings of some of the analysed artefact are shown in Fig. 66.

Reg. Nr.	Area	UF	Sq	Loc	Team	Description
AV 058	AV	5503	II 1 – 2	G 5156	Belgian	Copper-base alloy vessel with silver bottom
BJ 010	BJ	-	-	-	Belgian	Bar-shaped piece with rounded corners
ED 016	Surface	-	-	-	Belgian	Small ring
M 019	M	647	III-IV/ 3-4	F 1122	Belgian	*Kohlstick*?, traces of gold plating present
M 075-1	M	Dump	-	-	Belgian	Nail head/button
S 0025	-	-	-	-	-	Nail head, silver plated on copper- base alloy
sAV 505-13	AV	5503	II 1	G 5156	Belgian	Flat fragment, vessel?
sBJ 1237A (1)	BJ	Dump	-	-	Belgian	Fragment of hard solder?
sBS 1469	BS	6761	X 4	-	Belgian	Flat fragment, vessel?

Table 28: Co-ordinates of silver and silver alloy objects and samples.

Five silver objects were only analysed by ICP-MS for their lead isotopic ratios. The objects are: F 107, F 108, F 113, K 205 and N 301 (see Fig. 66 for drawings). They respectively are samples from two bracelets, two finger rings and a small twisted wire/pin.

[102] Campbell, 1933: 1; Coghlan, 1975: 118; Lang & Hughes, 1984: 85-86; La Niece, 1993a: 202.

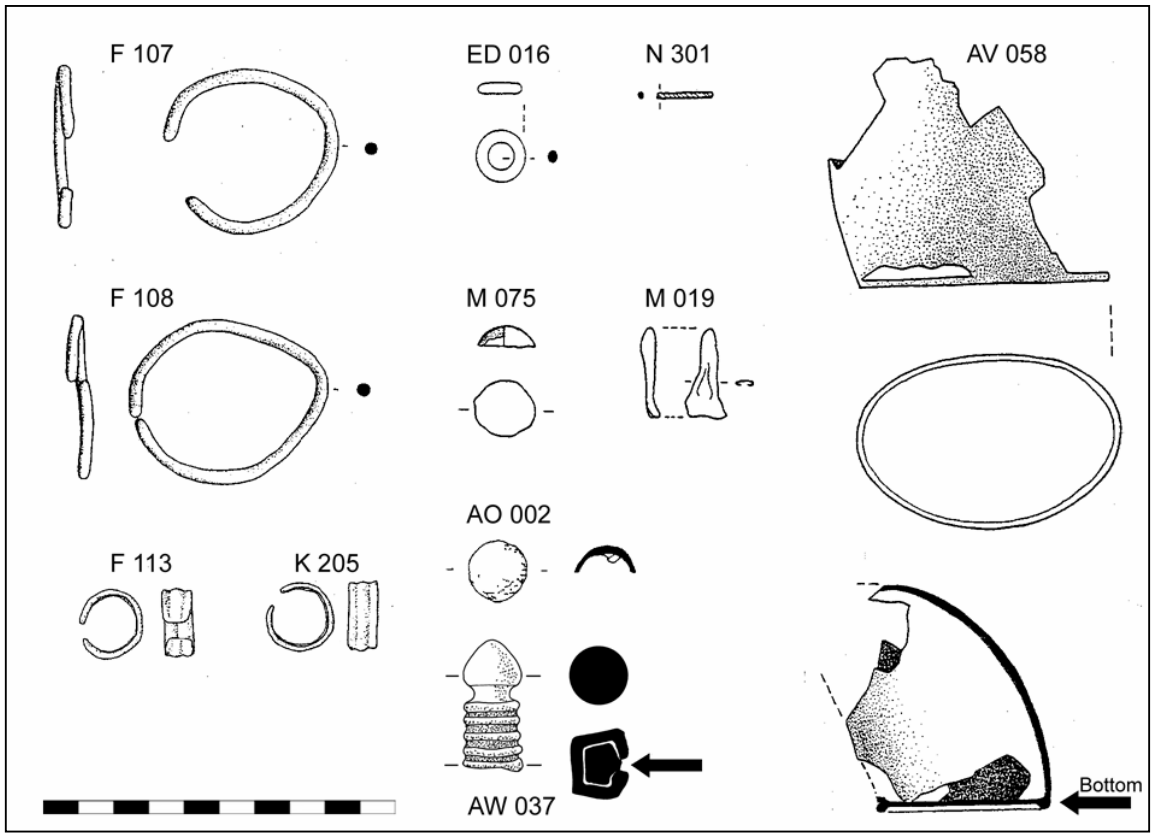


Fig. 66: Drawings of most sampled silver & solder fragments.

Reg. nr.	Cu	Ag	Au	Pb	*2-sig Cu*	*2-sig Ag*	*2-sig Au*	*2-sig Pb*	Alloy
AV 058	2,23	94,55	1,57	1,65	*0,15*	*0,98*	*0,33*	*0,40*	Silver
BJ 010	64,86	32,25	1,51	1,39	*0,97*	*0,69*	*0,41*	*0,46*	Billon
ED 016	2,07	95,40	1,14	1,40	*0,12*	*0,82*	*0,25*	*0,32*	Silver
M 019	1,60	95,10	1,63	1,67	*0,13*	*0,98*	*0,34*	*0,40*	Silver
M 075-1	69,38	27,54	0,65	2,42	*0,67*	*0,42*		*0,38*	Billon
S 0025	1,61	95,88	1,17	1,34	*0,12*	*0,91*	*0,27*	*0,34*	Silver
sAV 505-13	-	-	-	-	-	-	-	-	High Cl content, corroded silver
sBJ 1237A (1)	22,39	74,88	1,26	1,47	*0,38*	*0,67*	*0,24*	*0,31*	Hard solder?
sBS 1469	2,21	94,62	1,56	1,62	*0,17*	*1,12*	*0,38*	*0,45*	Silver

Table 29: Compositional silver and silver alloy objects and samples.

All silver samples contain some copper and some gold. The gold is probably the result of an incomplete purification. The copper could also be related to incomplete separation, but can also been deliberately added to harden the alloy.

BJ 010 (Fig. 67-1 & 2) could be a piece of hard solder, but the microstructure does only show patches of eutectic composition and corresponds more to the structures observed on the coins. There is a clear silver enrichment at the surface. The presence of a eutectic corresponds to the experiments preformed by L. Beck *et. al* where inverse segregation of copper-silver alloys with silver levels between 72% and 15% (i.e. an eutectic at the surface

and copper dendrites in the interior) was observed after casting[103]. It is possible that this fragment is related to the coining alloys used (see below).

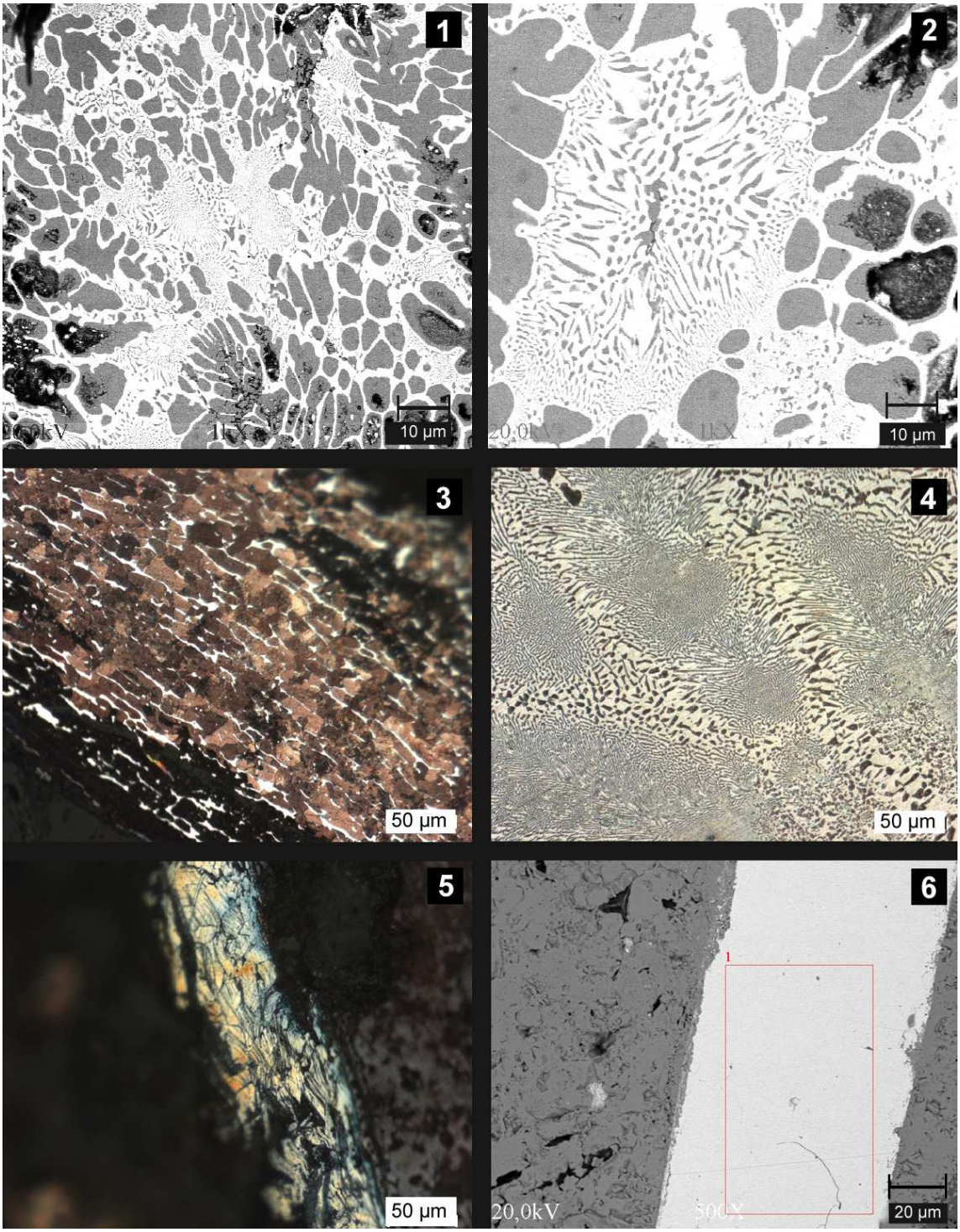


Fig. 67 : Silver and silver alloys (1 & 2: BJ 010; 3: M 075-1; 4: sBJ 1237A (1); 5 & 6: S 0025).

M 075-1 (Fig.67-3) is the cross-section of a nail head or a button. The silver grains are elongated in the direction of working, the copper grains seem to be less deformed.

[103] Beck, Bosonnet, Reveillon, Eliot & Pilon, 2004: 156. But see *Chapter 7* on the coins for a more detailed discussion.

The small fragment sBJ 1327A (1) could very well be a piece of hard solder, the microstructure has a eutectic composition as can be seen on Fig. 67-4. The light colored phase is silver-rich and the dark phase is copper-rich. The eutectic microstructure is confirmed by the EDX analyses and the silver-copper ratio (74,9 wt% – 22,4 wt%) is close to the eutectic composition (71,9 wt% – 28,1 wt%). The chemical composition of sBJ 1327A (1) gives the alloy a melting point around 800°C, and would make it suitable for soldering.

S 0025 is a decorative nail with a large rounded head and had no coordinates. The nail was complete covered with green (copper) oxides, but when sectioned a bright layer turned up underneath the corrosion skin (Fig.67-5 & 6). Apparently this was a copper-base alloy nail (completely corroded, so no composition could be determined) covered with a thin sheet of silver. This sheet was cold-worked as can be seen at the strain lines in the elongated silver grains.

AV 058 most probably is the silver bottom of a copper-base alloy vessel. Apparently the vessel was fixed on a silver base. This is a bit odd since the silver would be invisible. Maybe this object had an entire different function.

ED 016 is a small ring made of silver. It probably had some kind of decorative function (e.g. sewn on clothing, etc.)

M 019 was tentatively described as a *kohlstick*, which is an implement used to apply cosmetics. The most interesting bit of this object is that it showed the remains of gilding at the surface. This in combination with a fair proportion of silver in the core material indicates that this must have been a luxury object, a conclusion that is in line with cosmetic implements. The gilding technique was not further investigated here but some general information can be found above (*Chapter 5*, p. 223). The presence of silver might be evidence of *diffusion bonding*, instead of *fire gilding* as suggested for the copper altar bead BS 302.

Both sAV 505-13 and sBS 1469 are small flat fragments of silver and it is tempting to link them to vessel fragments.

6.5. Litharge

6.5.1. General introduction

The cupellation process usually gives two different kinds of waste material, being solid litharge and litharge-impregnated hearth lining. Additional archaeological indications can be crucible fragments, tuyere fragments, etc.[104] Although these remains are often not very plentiful, their presence on a site is a strong indication for silver production, silver recycling or silver assaying, since they do not occur naturally and are always man-made[105].

As described above the litharge soaked into the lining of the hearth or the cupel, shaping the litharge cakes. As soon as the litharge solidifies, the penetration in the lining stops. Usually litharge cakes are bowl-shaped almost like a ceramic vessel, plano-convex or concave-convex circular lumps, typically 8-15 cm in diameter. They have a high specific gravity and are usually grey in colour, but reddish and green colouration can also appear. The cakes are rarely found complete, the fractures showing a variety of textures ranging from massive to powdery or granular. Since the dry hearth lining is mostly loose and porous, the solidified litharge mixed with the lining can easily be separated from the hearth after cooling.[106]

One way to identify litharge is the rather typical shape. A second characteristic is of course their chemical and mineralogical compositions. Cupellation hearth material typically contain about 60 to 80 wt% of lead oxides (mostly weathered to lead (hydro-)carbonates), in the ranging between 5 and 10 wt% of silica and/or lime and minor amounts of magnesium, aluminium and iron oxide[107]. The phase composition within the litharge cakes is also variable and it seems to be related to the efficiency of the cupellation process, which can be evaluated on the basis of the amount of silver still present[108].

The major difference between the litharge cakes originating from extracting silver from lead and those resulting from de-silvering a debased alloy is the amount of copper (oxides) or even tin present. These metals are also oxidised during the cupellation together with the lead and make up part of the cakes. A characteristic of litharge cakes from production sites is their low content of *related oxides* (even when polymetallic ores were being exploited). In contrast, litharge cakes from recycling contain far higher levels of *related oxides* (CuO, cuprite - Cu_2O - or calcium-tin-oxide) as lead was added to copper-silver alloys to separate the silver. Their total CuO content is at least 7 wt% and in some cases considerably higher. Also the sizes of the litharge cakes may differ since the de-silvering of lead was done on a larger scale, the cakes tend to be bigger.[109]

6.5.2. Sample description & macroscopic examination

A considerable amount of metal samples was brought back to Belgium for further analyses, including some 3 kg of metal slags (see *Chapter 9*). Among these slags, two fragments stood out because of a different morphology, colour and specific gravity. They did not have the lobbed, rusty appearance and there was no magnetic reaction either.

Both fragments were recovered in the same area, Area BO. In this Area nine tombs were excavated and some platform remains linked to domestic structures. Sample BO 722 was found in the surface layer above one of the tombs. Sample BO 724 A came from the surface layer above one of the “rooms”. No further links can be made between the find context,

[104] Pernicka, Rehren & Schmitt-Strecker, 1998: 124; Kassianidou, 2003: 205.
[105] Tylecote, 1976: 76; Craddock, 1989: 208; Bayley & Eckstein, 1997: 107.
[106] Bayley, 1995; Pernicka, Rehren & Schmitt-Strecker, 1998: 125-126; Kassianidou, 2003: 205.
[107] Pernicka, Rehren & Schmitt-Strecker, 1998: 125; Rehren, 2003: 186.
[108] Bayley & Eckstein, 2006: 152.
[109] Tylecote, 1962: 81; Bayley & Eckstein, 1997: 108; Pernicka, Rehren & Schmitt-Strecker, 1998: 128.

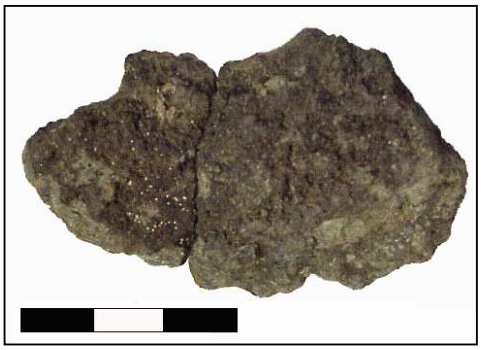
associated structures and the pieces of litharge. Upon a recent visit to the archaeological museum of Umm al-Qaiwain two additional fragments collected by the British team (AW 008 and AW 013) were "excavated" in the depot. Although the two samples had different coordinates (see Table 30), they belonged to the same fragment (see picture AW 008 & AW 013). They will be treated as such and referred to by AW 013 (the largest fragment was used for analyses). A possible fourth fragment (S 0006, Area G, UF 252) was registered by the French team as *lead slag*[110], an obvious mistake to make considering the physical appearance of the litharge. That combined with the fact that there is no indication that any lead production ever took place at the site, makes it safe to determine this sample as a piece of litharge. Unfortunately this fragment could not be localised during our visit to the museum depot and it will not be considered any further.

Reg. nr.	Area	UF n°	Sq n°	State	Thickness in mm	Width in mm	Length in mm	Weight in g	Volume in cm^3	Specific gravity in g/cm^3
BO 722	BO	5756	II 3	Frag.	14	42	55	69,3	11,5	6,03
BO 724 A	BO	5901	II 2	Frag.	20	58	51	116,2	22	5,28
AW 008 & AW 013	AW	4536 4539	XV-XVI 5,6 XIV 7	Frag. Frag.	20	42	65	-	-	-

Table 30: Physical description litharge.

BO 722

BO 724 A



AW 008 & AW 013

The weight, size, coordinates (Table 30) and a photo of the each fragment can be seen above. They have a grey to dark grey colour, with a greenish tint at some places (picture BO 724 A turned out more yellow than in reality).

For BO 722 and BO 724 A a rough calculation of the specific gravity was made. This was obtained by dividing the weight by the approximate volume (see Table 30). The average specific gravity is 5,7 g/cm^3, which is still far below the specific gravity of pure lead oxide (9,53 g/cm^3). This is explained by the fact that a litharge cake consists out of the mixture of lead oxide with the remains of the hearth material (which is lighter). In comparison with the average specific gravity calculated for the metal slags, being only *ca*. 3 g/cm^3 (see *Appendix 8*) the difference is striking however.

Deciding which way is the bottom side of the pieces of litharge is rather difficult, primarily because rather small fragments survived. It must be admitted that the suggestion done towards the bottom side here can be just the other way around[111].

[110] Notes provided by O. Lecomte.

[111] Advice was asked via e-mail to J. Bayley, who was kind enough to reply and give some suggestions. The problem of course was that 2-D pictures are difficult to judge a 3D object. M. Van Nie, who saw the fragments gave some suggestions. It is a combination of their suggestions that will be followed here. But again this is a tentative positioning.

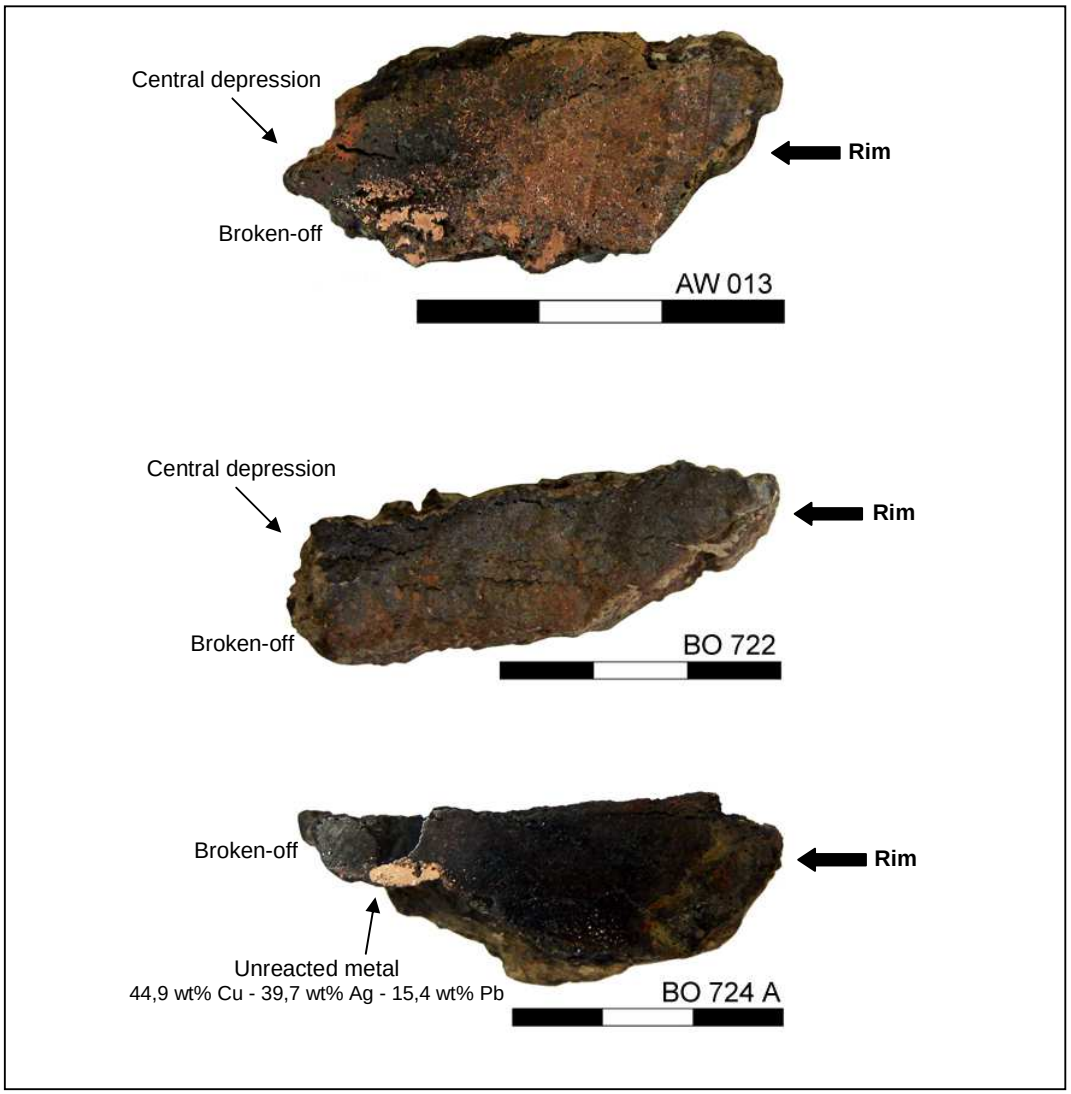


Fig. 68: Sections of the litharge fragments (AW 013 is a fresh cut surface, BO 722 is a natural brake & BO 724 A is a polished section).

The fragments have a rather flat bottom, but two show the remains of a small depression at the top-side were the silver-rich button must have collected. All have part of the rim preserved and indicate that the original shape must have been more or less circular. The diameter is difficult to reconstruct, since the rim sections are too small, but can be estimated between 10 and 15 cm, which would be in the line of other published data[112]. Samples BO 722 and BO 724 A have what seems a 'two-stepped rim'. A possibly explanation could be that this is the first litharge that solidified and combined with the lining.

The topside is grey, rather flat and rough, whereas the bottom side has more brownish and greenish shades and is rather smooth. A fresh brake has a dark grey metallic colour, with a reddish spotted band near the lower surface. When polished the copper-rich inclusions can be clearly distinguished. The size and amount of these inclusions is much higher at the bottom side and decreases towards the top. AW 013 has much more copper-rich particles included and is red copper coloured. BO 724A has a large piece of un-reacted or partially reacted base metal preserved (due to an incomplete process?). Many small cavities and

[112] An., 2001: 20: 6-60 cm; Kassianidou, 2003: 203: 12 cm; Bayley, 1992: 6: 3-15

cracks are present throughout the samples, but they do not make them crumbly. As a whole the samples can be described as massive.

All three fragments were cut in three parts in such a way that a representative cross-section from top to bottom was exposed. One part was polished and studied by optical microscope and SEM-EDX, a second part was crushed to fine powder and submitted for powder-XRD analyses and LIA by ICP-MS, and the third piece was kept for the reference collection and possible future research.

6.5.3. Microstructure & chemical composition – SEM-EDX results

The following elements were analysed for by EDX: O, Mg, Al, Si, S, K, Ca, Fe, Cu, Ag and Pb. The problem of the overlapping Kα-peak of sulphur with the Lα-peak of lead was already mentioned, nevertheless sulphur was included here. This is because an alternative analysis by means of XRF at the *University College of London* confirmed the presence of sulphur[113]. Other metallic elements (Au, Sn and Zn) and especially phosphorous were looked for but were not found. This shows on the one hand that only the binary alloy of copper and silver was treated and on the other hand that no phosphorous was present in the hearth lining.

	Average wt%				Average calculated oxide wt%		
	AW 013	**BO 722**	**BO 724 A**		**AW 013**	**BO 722**	**BO 724 A**
O	7,7	9,0	5,5				
Mg	0,4	0,2	0,4	**MgO**	1,6	0,8	1,6
Al	0,4	1,0	0,4	**Al_2O_3**	1,0	2,5	1,0
Si	4,8	5,4	3,3	**SiO_2**	17,0	19,5	11,3
S	0,6	0,9	1,5	**SO_3**	1,9	2,8	4,5
K	0,1	0,3	0,1	**K_2O**	0,1	0,4	0,1
Ca	7,2	6,0	7,3	**CaO**	17,9	15,2	17,5
Fe	1,5	3,3	0,4	**FeO**	1,8	4,0	0,5
Cu	19,8	16,9	24,6	**CuO**	31,0	26,9	37,3
Ag	0,9	0,4	1,0	**Ag_2O**	0,4	0,2	0,4
Pb	56,6	56,6	55,5	**PbO**	27,2	27,7	25,8

Table 31: Average chemical composition of litharge samples in wt% & calculated oxide composition.

The BSE-images of the litharge show a wide range of different textures and considerable variations in bulk composition. The values given in Table 31 are the average from 15 independent measurements throughout the section of the litharge samples. Areas with obvious large metallic inclusions were avoided. It is not clear if the reported data is reliable, since it differs considerably from the XRF data in *Appendix 9* and the calculated PbO fraction is far below the amount of lead oxide expected. A possible explanation would be that a large fraction of the litharge is represents the remains of the lining. A second factor is that not all these elements are of course present in they oxide form, for example metallic copper does occur too.

The three litharge fragments show in broad lines a similar composition. The major elements are lead, copper, calcium and silicon. Lead and copper are both also present in their metallic form and not as their oxides. The copper level is rather higher and could indicate that an alloy with a high copper content was treated. The silver level is low however, showing that the process must have been rather efficient. Important to notice is the fact that no

[113] With thanks to X. Veldhuijzen for performing the analyses (see *Appendix 9* for the unused results).

phosphorous was detected, so the cupellation hearth was certainly not made from or including bone ash.

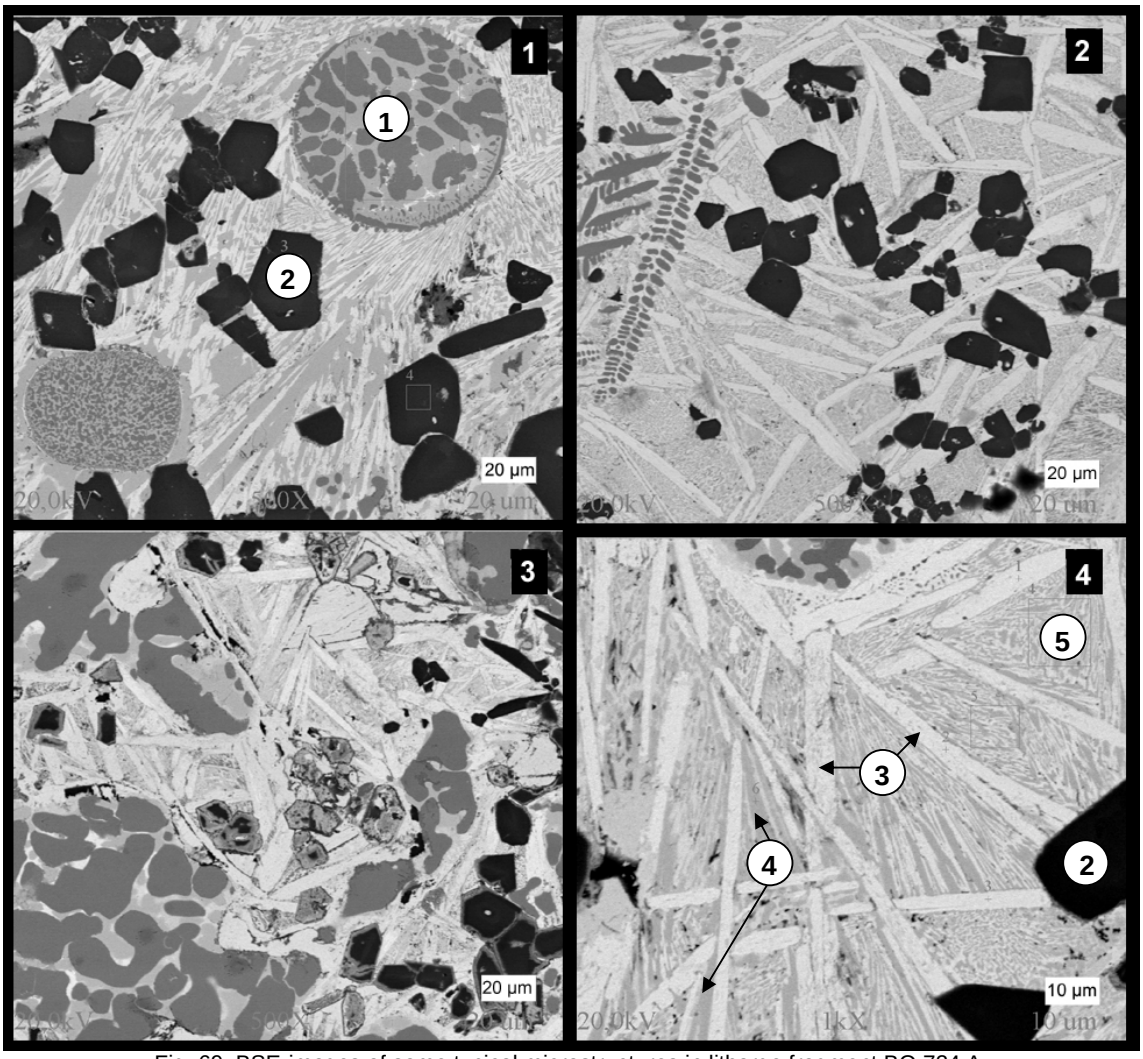


Fig. 69: BSE-images of some typical microstructures in litharge fragment BO 724 A.

Fig. 69 and 70 give an impression of the microstructure of the litharge. The large, platy white needle crystals (*3*) are PbO (massicote and/or litharge) and the darker grey ones (*4*) contain more copper (probably $PbO.CuO_2$). The space between these large needles is filled-in with a eutectic mixture of both crystals (*5*). The black inclusions (*2*) were part of the original lining. The main chemical elements detected in these inclusions are: silicon, calcium, magnesium, potassium and aluminium. Not all these elements are present in every particle and the ratios differ from one grain to another. In literature these inclusions are reported as e.g. calcium magnesium silicates and magnesium alumino-silicates[114]. The lighter rims that can be seen around some of the black inclusions are due to the reaction between the particles and the liquid lead oxide. The globule indicated with (*1*) is a copper-silver alloy. These are small quantities of the alloy that had to be treated that were lost to the litharge.

[114] Pernicka, Rehren & Schmitt-Strecker, 1998: 126; Hess, Hauptmann, Wright & Whallon, 1998: 60-64.

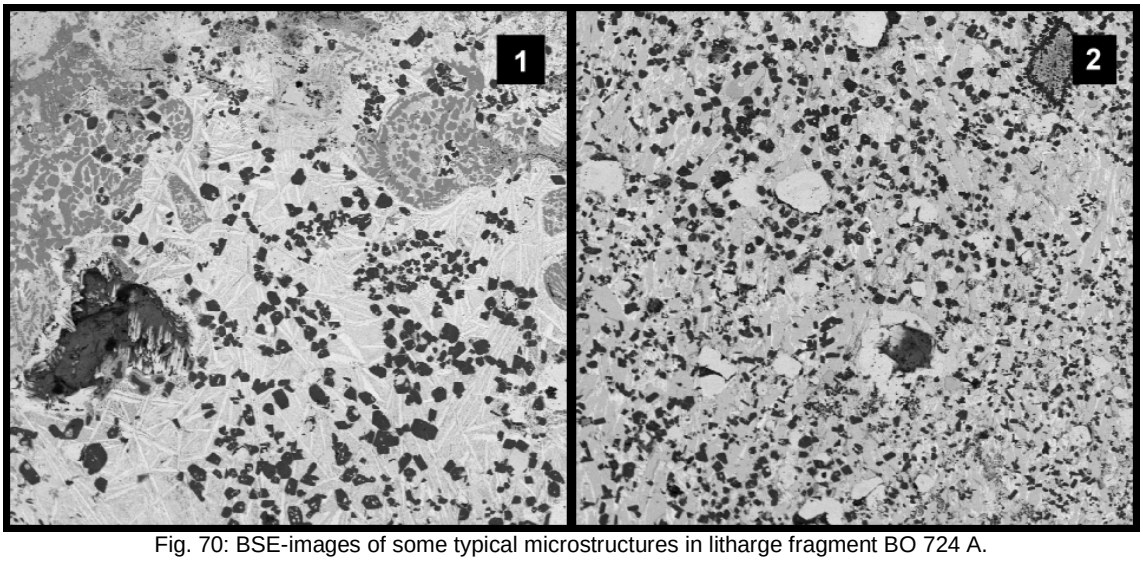


Fig. 70: BSE-images of some typical microstructures in litharge fragment BO 724 A.

Th. Rehren and L. Klappauf suggested that if the lead (actually PbO) is removed from the bulk analyses and the new results are normalized to 100%, an idea can be formed of the original hearth material[115]. Here we also have to eliminate the copper and silver, since they are the remains of the treated metal and no part of the original hearth. Sulphur was also removed, since the software identified the removed lead Lα-peak as the Kα-peak of sulphur, resulting in unrealistic high sulphur content. The figures given are the average of more than 9 measurements per sample.

In wt%	AW 013 hearth	BO 722 hearth	BO 724 A hearth
MgO	3,4	2,1	4,8
Al_2O_3	3,0	4,8	3,5
SiO_2	43,2	50,6	37,5
K_2O	0,5	1,0	0,7
CaO	43,7	29,4	51,4
Fe_2O_3	6,3	12,0	2,0

Table 32: Tentative calculation of the hearth composition.

These results can be compared to the chemical analyses of local and non-local ceramics from ed-Dur analysed for their composition[116]. The SiO_2 and MgO levels in the hearth fall more or less in the range of those of the ceramics, although the silicon level is at the low side. Only the Fe_2O_3 value of AW 013 falls within the range seen in the ceramics. The K_2O and Al_2O_3 values are much lower than in any of the ceramics and the CaO levels are higher. In general there is no similarity to be found in the chemical composition of the clays used for the ceramics and the (hypothetical) composition of the hearth.

The results for the litharge hearth composition calculated for Fatmali-Kalecik (E-Anatolia, 4th millennium BC) in the same way, show some similarity, but additional elements were quantified. This makes a direct comparison difficult. The general conclusion in the study on the Fatmali-Kalecik litharge was that the CaO values came from a carbonate mineral and that the silica came from the presence of quartz. Alumina, potassium and magnesium were

[115] Rehren & Klappauf, 1995: 23.
[116] De Paepe, Rutten, Vrydaghs & Haerinck, 2003: 217 & 219.

attributed as components of clay minerals. The researcher concluded that the porous lining of the hearth was made from a mixture of quartz and marl.[117]

- **Powder-XRD**

An attempt was made to characterise the mineral phases present in the litharge fragments. As with the slag (*Chapter 9*) this was not very successful and it was impossible to identify all peaks. This made the quantification of the spectra difficult. The spectra are included in *Appendix 10.*

As expected[118] several lead containing mineral were attested such as *massicote* (PbO), *litharge* (PbO) and *cerussite* ($PbCO_3$). Cerussite appears when lead oxide is weathered. Other minerals attested were mainly calcite (CaO) and quartz (SiO_2). No further time was spend on the deciphering of the spectra.

[117] Hess, Hauptmann, Wright & Whallon, 1998: 63.
[118] Pernicka, Rehren & Schmitt-Strecker, 1998: 126; Hess, Hauptmann, Wright & Whallon, 1998: 60-64.

6.6. Lead isotope analyses – ICP-MS

- **Lead, solder & litharge**

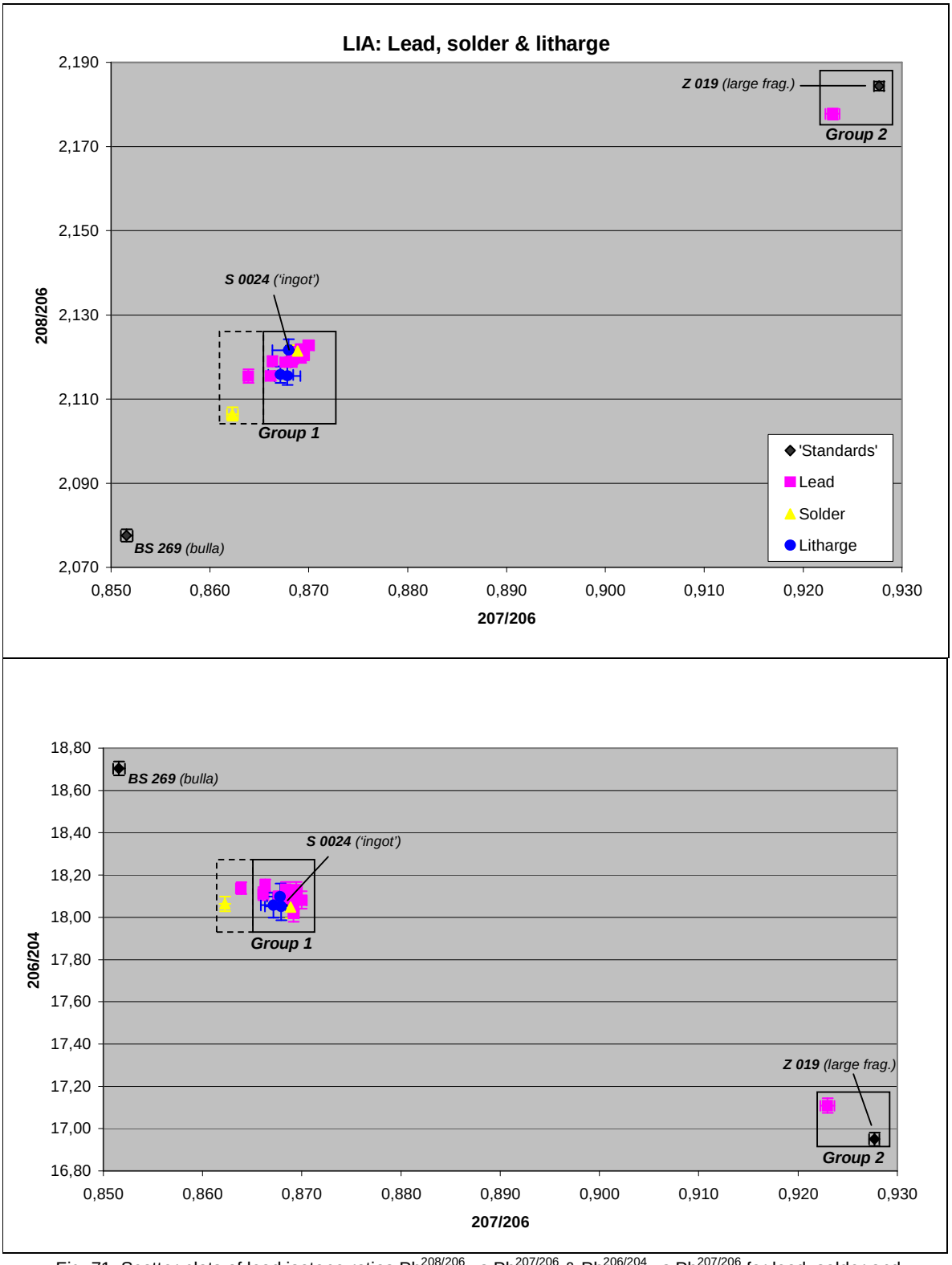


Fig. 71: Scatter plots of lead isotope ratios $Pb^{208/206}$ *vs* $Pb^{207/206}$ & $Pb^{206/204}$ *vs* $Pb^{207/206}$ for lead, solder and litharge (the error is indicated by error bars).

Fig. 71 shows the scatter plots for the isotope ratios of the lead (15), solder (2) and litharge (3) fragments analysed. Error bars indicate the error on the measurements. The complete dataset can be found in *Appendix 5*. Three samples were tentatively used as 'standards'. The first is the 'ingot' (S 0024) and as can be seen the large majority of the samples group around it (*Group 1*). This is a very interesting feature since it is a strong indication that S 0024 actually *is* an ingot. It also shows that the majority of the small fragments are made of lead from the same origin and that this lead was used for the cupellation process. It can be safely concluded that all this lead originated from the same source. The second 'standard' is the large lead fragment Z 019. It is situated at the extreme end of the plot, together with one other lead fragment. Both samples are not close enough together to definitely link them to a single source, but they definitely originate from a source different then *Group 1*. The third 'standard' is the Indian *bulla* (BS 269) that also falls to far outside of *Group 1* to be from a similar source.

For the moment I will leave aside all possibilities of mixing and recycling. From this first visual examination of the plots it can then be suggested that the majority of the lead found at ed-Dur came from a single source. This was the lead the inhabitants used for their daily activities.

At least one different source of lead is attested. The sample came from a rather large fragment of lead and might as well be some kind of 'ingot' (Z 019). One small fragment of lead groups in the region of Z 019 but the isotope ratios do not overlap. The *bulla* falls outside this, but this needs not to be a surprise, since this object was certainly not produced at ed-Dur. The source of the lead of the bulla will not tell more about the metal used at ed-Dur, only on the metal used to make this object (most probably in India).

I will first discuss the two outlying samples of *Group 2*. The only ore sources that plot in this region of the plot are the ones from the Indian Subcontinent (Fig. 72). The ore samples were taken from ore sources in the Rajasthan region, i.e. Zawar, Rajpura Dariba and Khankaria. The ores are polymetallic and contain zinc and lead. Silver and copper deposits are also present.[119] The lead isotope ratio of the ores do not fall completely together with the two objects from ed-Dur, so the lead does not come from one particular sampled ore region. The lead metal can however be a mix of metal originating from several regions, which would give an intermediate isotopic signature.

Alternatively and equally possible, the bulk of the isotopic signature of the two ed-Dur samples may be made up from a contribution of the Indian lead metal. The rest may be contributed by lead from a different origin. Mixing Indian lead with a small portion of the lead samples composing *Group 1*, would result in such a signature. The ratios would shift along an imaginary line connecting the two original ore sources.

A third possibility would be an as yet unidentified ore sources in India, possibly located in the Rajasthan region. This last possibility is to me the most likely.

[119] Ericson & Shirahata, 1985: 207-209.

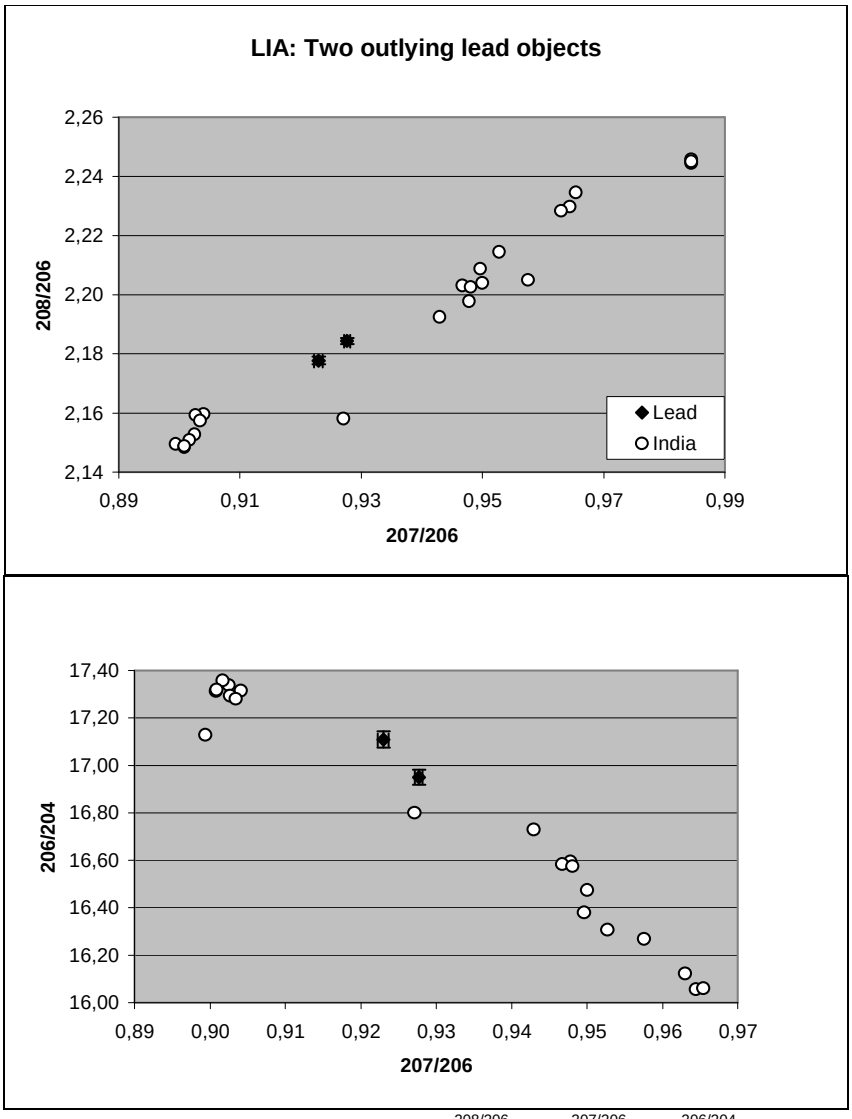


Fig. 72: Scatter plots of lead isotope ratios $Pb^{208/206}$ *vs* $Pb^{207/206}$ & $Pb^{206/204}$ *vs* $Pb^{207/206}$ for *Group 2* and Indian ore sources(the error is indicated by error bars).

Unfortunately no ores sources or objects plotted close by the Indian *bulla* (BS 269). This is a pity since it might have provided interesting information. Some points from Great Britain come close on the $Pb^{208/206}$ *vs* $Pb^{207/206}$ plot, but not on the $Pb^{206/204}$ *vs* $Pb^{207/206}$.

Next the lead objects from *Group 1* will be discussed, together with the silver object and silver coins analysed (Fig. 73). To ease the comparison two additional groups are defined (*Group 3* & *4*). The spread within group 1 may suggest that this cluster can be split in two, i.e. *Group 1* and *Group 1-bis*. Thus three different regions of origin for the silver can tentatively be suggested. Silver objects appear in *Group 1-bis* (possibly related to the cluster of lead), *Group 2* and *Group 3*. The four objects in *Group 3* are closely related isotopically and are most likely from the same ore source. There seems to be a division between the coining silver used for the obol and tetradrachms, with and overlap in *Group 3*.

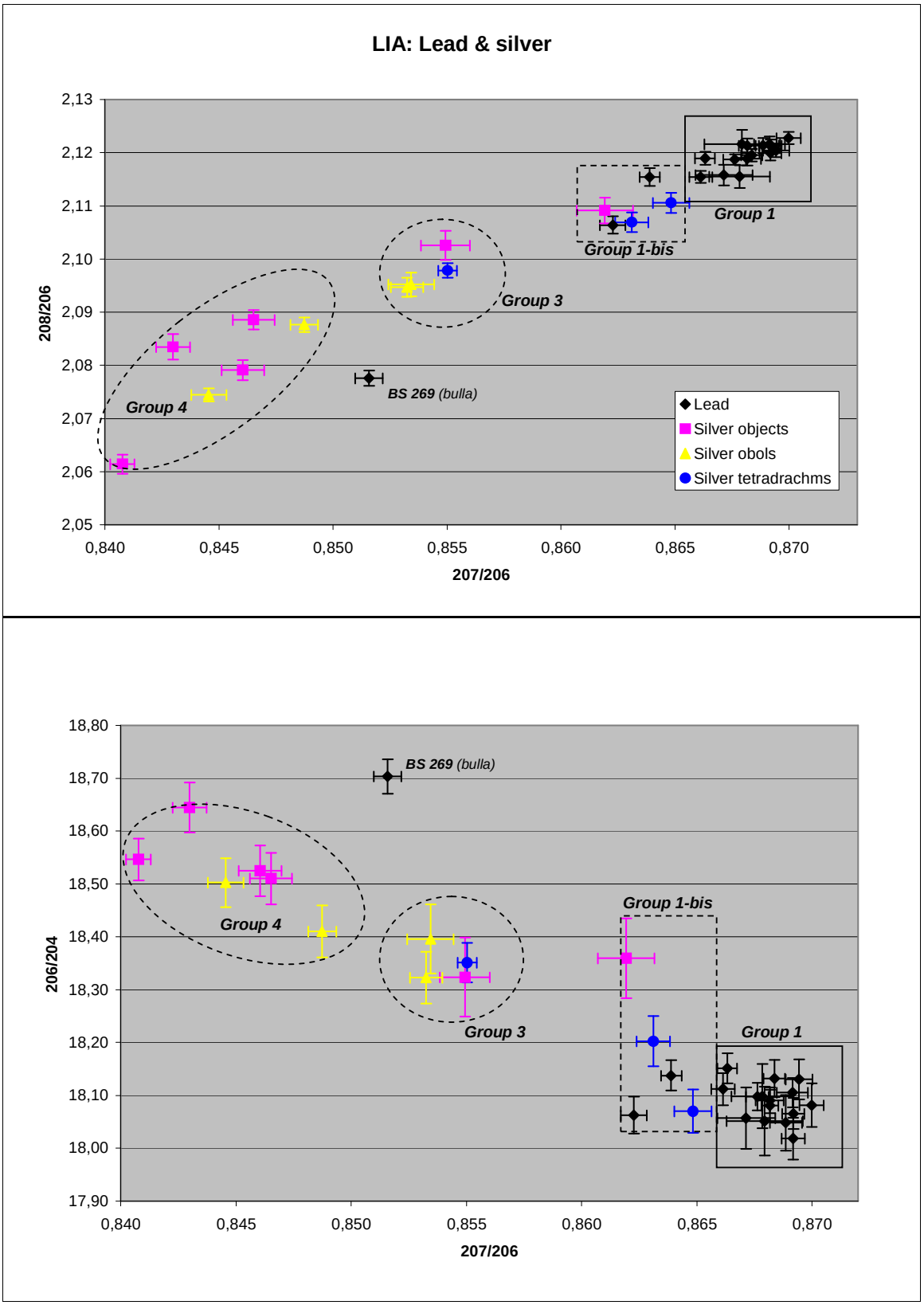


Fig. 73: Scatter plots of lead isotope ratios $Pb^{208/206}$ *vs* $Pb^{207/206}$ & $Pb^{206/204}$ *vs* $Pb^{207/206}$ for silver and lead (the error is indicated by error bars).

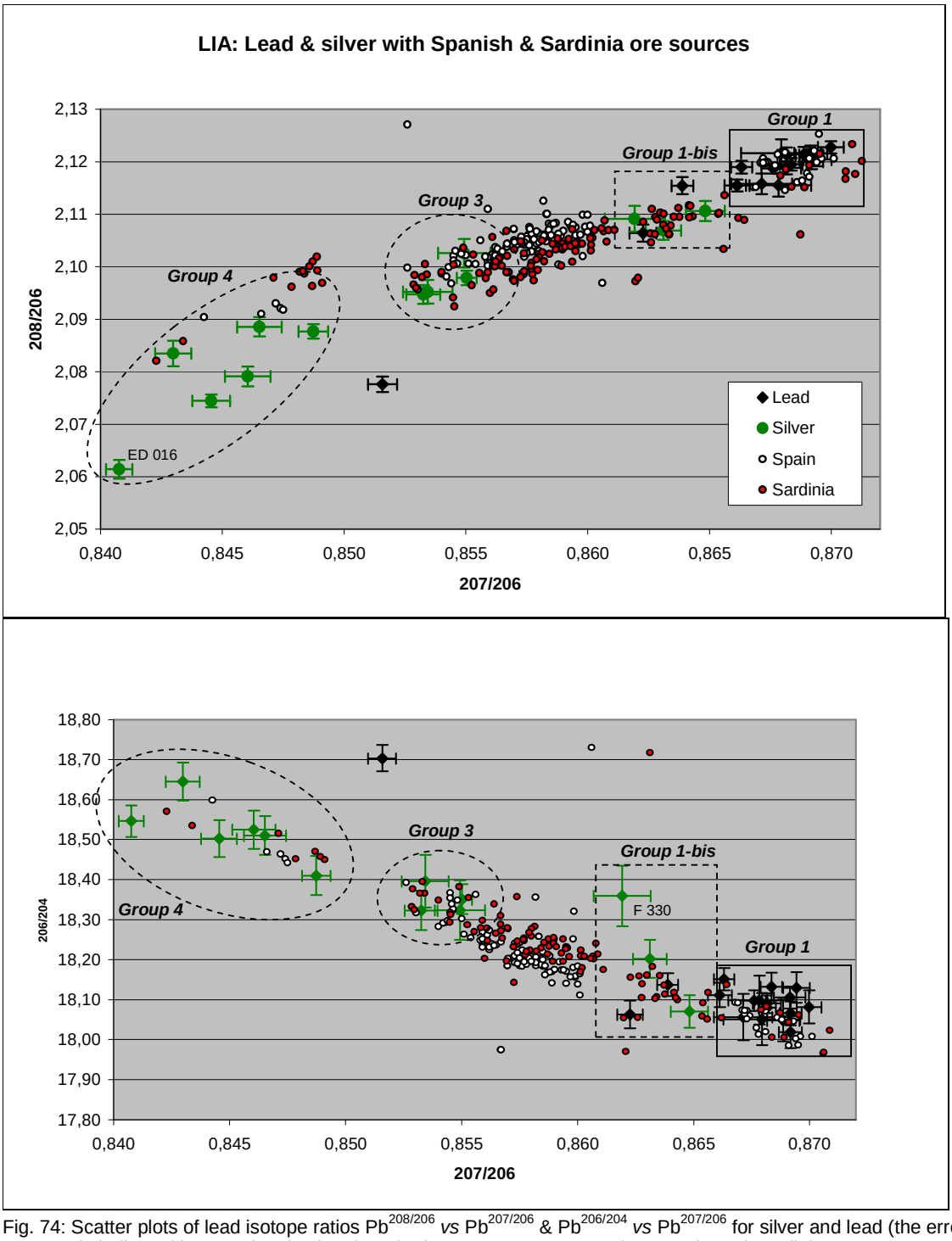


Fig. 74: Scatter plots of lead isotope ratios $Pb^{208/206}$ *vs* $Pb^{207/206}$ & $Pb^{206/204}$ *vs* $Pb^{207/206}$ for silver and lead (the error is indicated by error bars). The plotted relevant ore sources are from Spain and Sardinia.

The ore field of Spain and Sardinia nicely fall in the *Group 1* lead cluster. These are the only sources that plot inside this region and it can be safely concluded that this lead comes from one or both of these regions. In favour of Sardinia is that also the samples within *Group 1-bis* fall together with those of Sardinia, except for one silver sample (F 330) that falls outside. Both regions were extensively mined by the Romans till up to around 70 AD, after which the British ore field became the main providers of lead and silver. *Group 3* shows the same good accordance, but *Group 4* is rather poorly defined by the Spanish/Sardinian ores (especially

for the $Pb^{208/206}$ *vs* $Pb^{207/206}$ ratios). A different origin all together may be likely for these silver samples.

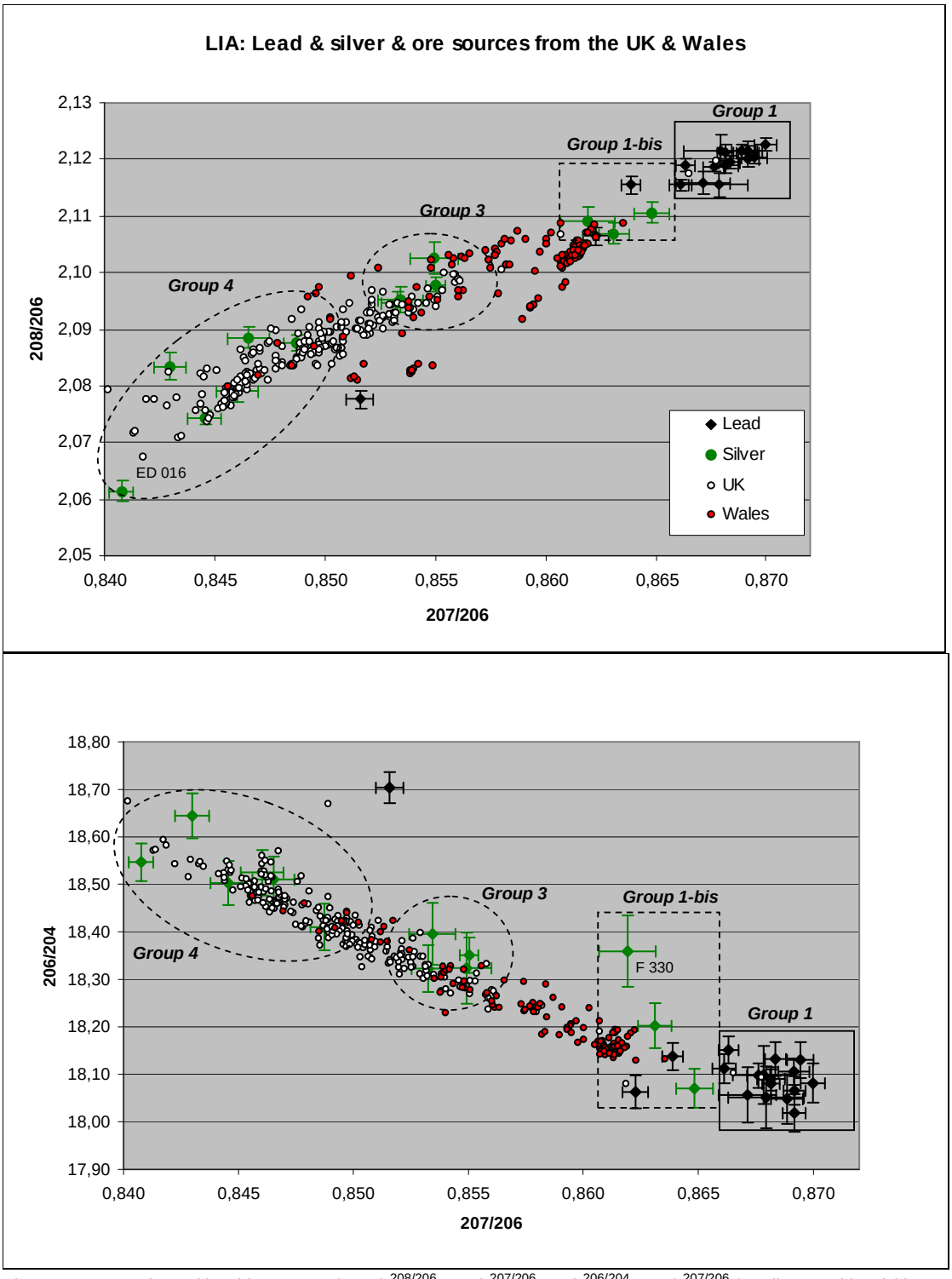


Fig. 75: Scatter plots of lead isotope ratios $Pb^{208/206}$ *vs* $Pb^{207/206}$ & $Pb^{206/204}$ *vs* $Pb^{207/206}$ for silver and lead (the error is indicated by error bars). The plotted relevant ore sources are from the UK and Wales.

The ores from the UK and Wales (Fig. XX) show significant overlap with the silver objects in *Group 3* and *4* and would complete the source attribution for almost all samples analysed from ed-Dur.

Of course the solution is probably not that straight forward an certain other ore sources also showed overlap with the data from ed-Dur. For the sake of correctness Fig. 76 shows the plotted ore fields from Germany, France, Iran and Oman. Other region present in the lead isotope database did not cluster or only to a very limited extent around the data from ed-Dur so they are not further discussed.

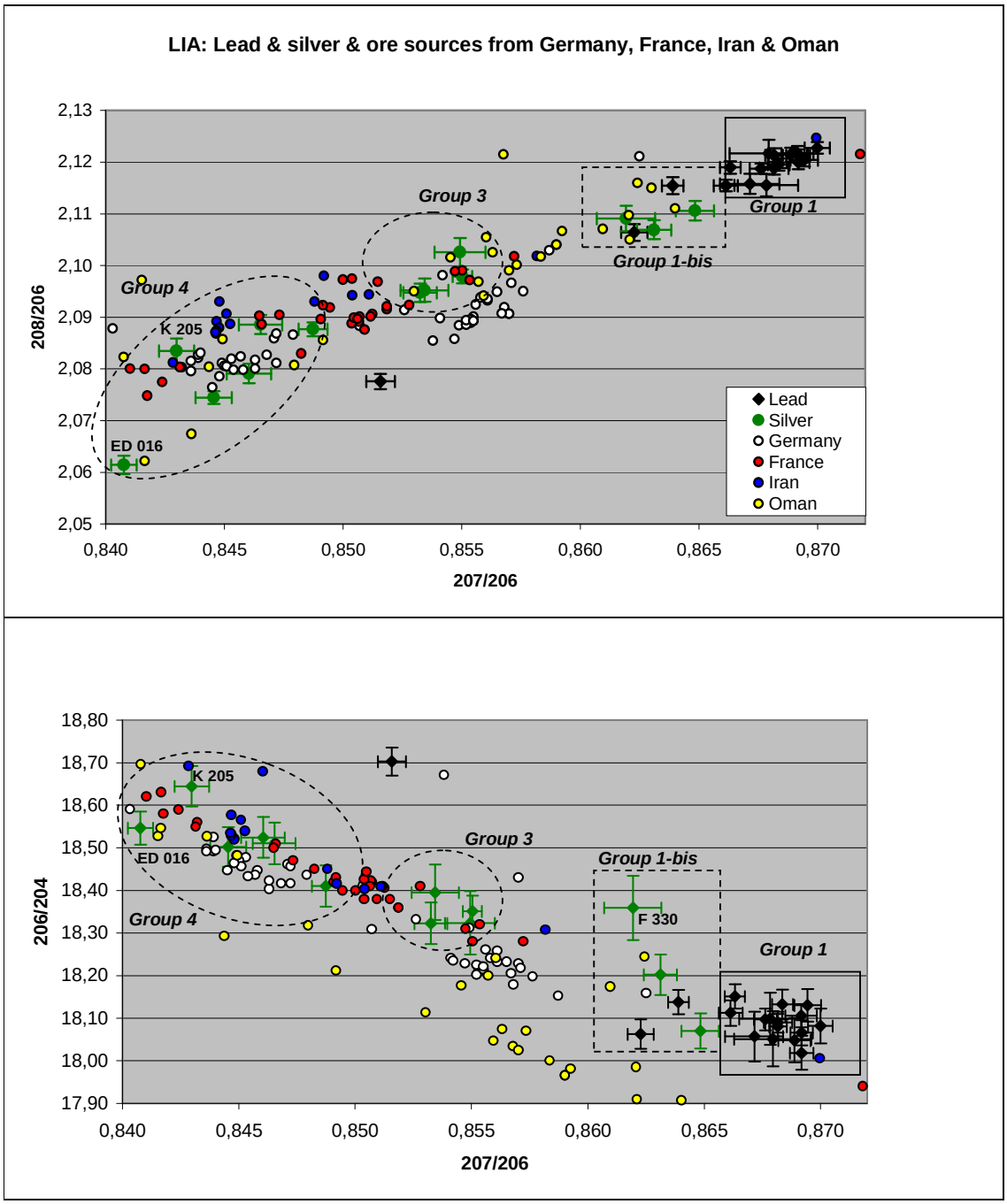


Fig. 76: Scatter plots of lead isotope ratios $Pb^{208/206}$ *vs* $Pb^{207/206}$ & $Pb^{206/204}$ *vs* $Pb^{207/206}$ for silver and lead (the error is indicated by error bars). The plotted relevant ore sources are from the Germany, France, Iran & Oman.

The German ore fields were potentially accessible to the Romans although I did not find any information that they were exploited at that time. It is however known that the zinc ores in Germany were extensively used, as evidenced by the archaeometallurgical remains of brass production found at *Xanten*[120]. There is some overlap with *Group 4* and one point plots within *Group 3*. As a whole however the overlap is rather limited and probably ores from Germany might be excluded as a potential provider.

A better fit is seen for the ore fields from France, especially for some samples in *Group 3* and *Group 4*. France can thus not be excluded as a potential provider.

The limited evidence from Iran is plotted since there is a clear link between the Parthian or at least Characenean territory and ed-Dur. One point plots at the limit of the lead in *Group 1* but has probably nothing to do with this group. The silver ring K 205 that is poorly covered by the previously suggested ore sources is covered in both plots by one of the Iranian ores. For the rest of the points no good overlap is seen.

The ores from Oman are included for the obvious reason that ed-Dur is close by. It should be stressed however that there is as yet no evidence that during the period under consideration any mining or extractive metallurgical activities were taking place. As a whole there is little overlap. Two points plot within *Group 1-bis* however and one point plots close to ED 016 (a small silver ring). This is the only point that can be connected to ED 016. This is not evidence of course that the metal originated from Oman, but an interesting occurrence.

Some samples such as F 330 and the *bulla* BS 269 do not really have any relation with any of the lead isotope ratios in the database.

[120] Rehren, 1999b.

6.7. Interim conclusions lead, silver, their alloys & litharge

• **Lead & alloys**
The lead found at ed-Dur turned out to be, 'how surprising', lead. The EDX analysis is not sensitive enough to address the occurrence of trace elements present and the only conclusion is that the lead is unalloyed.

The suggestion by L. Weeks that the elevated levels of silver he found in many of the copper-alloy samples from ed-Dur were introduced by the lead can only be partly evaluated. What can be said is that the five lead samples analysed by ICP-MS had very low levels of silver present. This only shows that if the lead indeed introduced a small amount of silver to the copper-base alloys, this lead was from a different source then that of the lead objects. Which is of course entirely possible. In the literature low levels of silver in lead are often related to a de-silvering process of the lead. Other possibilities are however the production of lead from silver-poor primary lead ore or the production of lead at low temperatures, i.e. the silver present is not extracted. Z 019 is stands out of the samples analysed for their silver content, in that it has a more elevated level.

L. Weeks' second suggestion, that the high sulphur concentrations in some leaded copper-base alloy samples analysed by him, were introduced by the lead could not be evaluated. This due to the fact that sulphur is rather difficult to detected with ICP-MS and needed a special sample preparation. Time constraints and the fact that a completely new procedure had to be developed for a relatively small gain in information, led to the decision to set this part of the research aside.

Some 'new' alloys were found amongst the samples first thought to be lead. Seven samples turned out to be from a tin-lead alloy. The samples were however too severely corroded to give any reliable compositional results. Tin-lead alloy can be defined as either *soft solder* or *pewter*. There is no clear distinction between the two but rather a functional one. Solder is as the term implies used to solder two parts of an object together. The term pewter is used for objects made of a tin-lead alloy. Some of the samples certainly are solder, i.e. they were taken from underneath the head of a button. One flat fragment however could be part of vessels and would be pewter then. If this is the case, this is the first reported pewter from the region. Also one fragment of unalloyed tin was found.

• **Silver & alloys**
Six silver fragments were analysed by EDX. All silver samples contain some copper, some gold and some lead. The gold and lead is probably the result of an incomplete purification. The copper could also be related to incomplete separation of the ore, but is more likely to have been added deliberately to harden the alloy.

M 019 needed mentioning since remains of gilding are seen at the surface. The gilding technique was not further investigated, but it might have been obtained by *diffusion bonding*, instead of *fire gilding* as suggested for the copper altar bead BS 302 in the previous chapter.

The remaining silver objects were only analysed for their lead isotope ratios.

• **Litharge cakes**
The three litharge fragments discovered at ed-Dur are the remains of a previously unattested technological process at the site and in the wider region at the period under consideration. They are also the sole witnesses of a cupellation process, since no cupellation hearths, crucibles or crucible fragments, or completely/partly preserved tuyeres were encountered. No phosphorous was detected by the SEM-EDX, so the cupellation was not done on bone ash cupels. This in combination with the absence of any crucible fragments can lead to the conclusion that the process was done in small hearths dug in the sand and lined with a

calcium-rich matter (hence the high Ca levels). Clay or marl are not available in the near surrounding of ed-Dur, but a possible nearby source of calcium can be found in grinded up shells or *farush* (the beach-rock that is used as building material and is made up by a concretion of shell). The remains of 'plastering' was found on different occasions, e.g. the walls of the temple in Area M and the domestic building in Area C, the remains of floor levels, on walls of tombs, etc. Although the plaster was not analysed we can assume that this substance is and porous. This shows that the inhabitants of ed-Dur had access to calcium-rich matter that could be used to line a cupellation hearth. Moreover marl or calcareous clay has also been reported from the surroundings of Mleiha[121].

The high copper levels in the litharge cakes indicate that the process was one of extracting silver from copper-silver alloys. This process seems to have been well mastered by the inhabitants of ed-Dur since the residual silver levels are low. It is tempting to link these residues to the recycling of coining metal, since this is the only group of artefacts that is made of copper-silver alloys. This is however only a suggestion, since there is no circumstantial evidence to back this up. Moreover the fact that one decorative nail head or button (M 075-1) was made from a copper-silver alloy shows that the alloy was also used for other purposes.

To my knowledge there are not other litharge fragments reported from SE-Arabian sites, but this could be due to the fact that these remains were not recognised as such. Alternatively the absence of large quantities of litharge can be explained by the fact that litharge cakes can be re-smelted to recover the lead[122]. If recycling took place at ed-Dur is not clear, but the relatively abundance of metallic lead at the site (when compared to the absence on other sites) would plea for a non-recycling model. The nearest litharge remains come from Saudi Arabia, but they date to the much later Abbasid period (8^{th} – 13^{th} c AD) where they seem to have been the remains of the extraction of especially gold and/or silver from complex polymetallic sulphides. They were found on the sites of An-Nuqrah South, Ash Shumta I, Al Koom al-Gharbi, Al Koom al-Sharki and Mawan, all situated to the NE of Medina[123]. Unfortunately no details, pictures or chemical analyses of this material were published.

- **Lead isotope analyses**

The lead isotope analyses of lead and silver were much more willing for interpretation than those of the copper-base alloys in the previous chapter. This allowed a more in-depth approach, unfortunately not (yet) achieved for the copper-base alloys.

Lead, solder & litharge

The large majority of the lead samples analysed plotted closely together indicating that they originated from a single source. The object tentatively identified as an 'ingot' (S 0024) falls nicely in the middle of the lead cluster and this indeed makes it very likely that this object actually also *is* an ingot. Also the litharge fragments plotted in this cluster and show that the same lead was used in the cupellation process.

Their isotopic signature is in very good accordance for lead originating from ore field in Spain and Sardinia. Moreover these are the only sources that plot inside this region of the lead isotopic plots and it can be safely concluded that this lead comes from one or from both of these regions. In favour of Sardinia is that also the samples that fall somewhat outside the tight lead group are also covered by the Sardinian ores. Both regions were extensively mined by the Romans till up to around 50 AD, but by 70 AD the British ore field became the main providers of lead (and silver). These date falls well within the occupation phase of ed-Dur. The determination of a Sardinian and Spanish origin of the lead is in accordance with the

[121] Briand, Dalongeville & Ploquin, 1997: 49.
[122] Tylecote, 1962: 82.
[123] de Jesus, Al-Surgiran, Rihani, Kesnawi, Toplyn & Incagnoli, 1982: 63-77; Kisnawi, de Jesus & Rihani, 1983: 78.

results published on 1st c AD Indian lead coinage by N.J. Seeley and P.J. Turner. They also found that the lead isotopic signature fitted that of the Sardinian and Spanish ores.[124]

Two lead samples, amongst them the large fragment Z 019, plot well away from the main group discussed above. This shows that at least one additional source of lead is attested. It might be suggested that Z 019 actually also is an ingot. The only ore sources that plot around these two samples are the ores from the Indian Subcontinent, i.e. the Rajasthan region. The lead isotope ratios of the ores do however not fall completely together with the samples objects from ed-Dur. The main reason for that is that the number of LIA available from India is rather limited and not enough to clearly define the ore fields. Based on the lack of any other alternative I would like to suggest that the lead is from Indian origin, but possibly from an as yet unidentified ore sources in India. Z 019 also contains more silver as a trace element than the other samples analysed, adding to the atypical character of this sample.

The interesting stamped monogram on the ingot and the possibility that Indian lead is attested at ed-Dur will be further discussed in *Chapter 10*, in relation to the possible routes the lead metal could have travelled to get to ed-Dur.

Silver

The lead isotopic fingerprints of the Sardinian and Spanish ores only partly cover that of the silver objects from ed-Dur. The ores from the United Kingdom and Wales show significant overlap with the other silver objects. These four regions cover the majority of the silver samples from ed-Dur.

Other ore sources however also gave a certain amount of overlap, and the actual origin question for the silver objects may be more complex than sketched above. Metal from ore sources in France has to be considered. The limited evidence from Iran available only shows a significant overlap with one silver sample (a finger-ring, K 205). The ores from Oman as a whole show little overlap, however the small silver ring ED 016 only covered by a data point from Oman origin.

Some samples such the *bulla* BS 269 showed no association with any of the lead isotope ratios in the database.

The research done here is the first step, but further refinement is needed. A more detailed study of the results may narrow the possible mines and ore sources down and give a more exact origin of the region the metal actually originated from.

[124] Seeley & Turner, 1984: 331.

Chapter 7. THE COINS

"Before you can hit the jackpot, you have to put a coin in the machine."

F. Wilson

7.1. Introduction

Until a couple of decades ago local pre-Islamic coins from SE-Arabia were totally unknown. This picture changed when new coin types started to appear on the antiquities market in the 1980s, particular in the wake of the First Gulf War. By 1998 a minimum of 750-800 coins were known from SE-Arabia, of which an estimated amount of 290-300 came from the site of Mleiha and 470-500 from the site of ed-Dur (of which 20% out of official excavations).[1] Most of these coins were picked up by private collectors before the start of official excavations at ed-Dur (and Mleiha), and dispersed in different collections worldwide. A substantial number of the coins were published in an attempt to document as much information as possible before these coins disappeared into private hands.[2]

The 112 coins presented here are the result of official excavations, of which the team of Ghent University collected 109. Seven more obols were found, but these are on display in the Archaeological Museum of Umm al-Qaiwain and could not be included in this analytical study. They are however included in the typological part. Despite the fact that all of these coins originated from official excavations, little contextual information is known. Ed-Dur is essentially an un-stratified site and most coins were just surface finds. C.S. Phillips, the former director of the British team, made three additional coins available. Next to the 104 Arabian issues, 8 foreign coins found at ed-Dur were also analysed and the results will be presented. They will however not be discussed any further. The reasons are multiple: some are difficult to assign to a place of origin or there is limited reference material available. The main reason however is the fact that their metallurgical analysis does not contribute to a better understanding of SE-Arabia coinage, which is the main goal of this study. They do however supply considerable information on the trade contacts and networks existing at that time, but this will touched upon in *Chapter 3*.

After a general introduction on the coins of the Arabian Peninsula and the typology used for this study, the analytical results obtained by SEM-EDX will be presented. The compositional results and visual information retrieved are combined with the typology, weight and possible production methods of the coins. The compositional and microstructural observations raised certain questions however. To tackle these questions it was decided to add a small experimental section. Several coins were cast and treated in the same way the ancient coins are supposed to have been produced. The second last point gives the data on trace elements and lead isotopes collected by ICP-MS analyses. In the final part of this chapter all the presented information is brought together in a preliminary conclusion. A condensed form of this chapter, without the ICP-MS data was presented at the *Seminar for Arabian Studies 2006* and is published in the proceedings[3].

[1] Haerinck, 1998b: 279.
[2] Salles, 1980a; Boucharlat & Drieux, 1991; Potts, 1991; Callot, 1994a; Potts, 1994b; Senior, 1994; Grave, Bird & Potts, 1996; Haerinck, 1998a, 1998b, 1999 & 2002d; Callot, 2004.
[3] Delrue, 2007.

7.2. Historical frame of coinage on the Arabian side of the Gulf

• **Historical frame**[4]

It was only after 1974, when C. Robin[5] published the first general assessment of the indigenous coinage of E-Arabia that the numismatic attention was pulled to this region. Before that date the only important minting centres known in the Arabian Peninsula were the S-Arabian kingdoms of Saba, Ma'in, Qataban and Himyar, and the NE-Arabian Nabataean kingdom. Even the early Islamic writers were unaware of any indigenous coining outside the Himyaritic kingdom. The discovery of several 3rd c BC hoards of silver tetradrachms on Failaka and Bahrain by Danish archaeologists in the 1960s changed this picture. They revealed the presence of issues that could not be assigned to any of the known categories of Seleucid, S- and NE-Arabian or Ptolomaic coinage.[6]

More discoveries and further research showed that from the 3rd c BC onwards coins were minted at different centres in Arabia, copying the coins introduced by Alexander the Great. On the one hand the adoption of the Hellenistic design can be explained by the fact that the Alexandrian coins were highly valued because of their good quality and high silver/gold content. This resulted in their use as a favoured coinage for trade even some hundred years after their original minting. Although Arabia was not a part of the Hellenistic world there were contacts and obviously also influence. On the other hand no indigenous coins were made before the Hellenistic period, and the obvious source of inspiration would have been the widely spread Alexandrian coins.

The appearance of the first indigenous coins in NE-Arabia (during the second half of the 3rd c BC) can be linked to a period of political weakness in the Seleucid power that dominated the area. Under the Seleucid king Seleucos II (246-226) there was a weakening of the influence on in the eastern part of the Empire that amongst others resulted in the independence of Bactrian satrap and the birth of the Parthian Empire. It is probably during this feeble period that the local Arabian rulers and people profited from the benefits of the trade routes that were not longer controlled by the Seleucids. Issuing their own coinage helped to affirm their independence and to underline that they formed a federation of states grouped around the common cult of the Semitic sun god Shams/Shamash. They did so however without completely abandoning the monetary types in use by the Seleucids, since these must have been the reference value for them and they did want to maintain privileged relations with the Seleucids. Besides a limited use for commercial transactions it has been suggested that these Arabian issues were also used to pay soldiers or mercenaries for protection.

The earliest coinages essentially consists of tetradrachms, drachms and obols, in good quality silver, all more or less skilful imitations of coins of Alexander the Great. Some of these coins have legends in S-Arabic writing, showing the name Shams, others present the names of a local ruler, either in S-Arabian or Aramaic. The exact origin of these different issues is not know, but there is reasons to believe that mints were situated on Ikaros (Failaka), Tylos (Bahrain) and on the east coast of Saudi Arabia where the famous trading city of Gerrha (Thaj?) was situated. It is also probable that certain regions in the Oman Peninsula had their own mint. The entities that first produced these coins must have been powerful enough to compete with the Seleucid dominance, but must also have been close enough to adapt the technology from the Seleucid Empire considering the good quality of these early issues.

The production of these first silver coins seems to have stopped rather suddenly at the end of the 3rd c BC or the beginning of the 2nd c BC. This can be connected to the presence of Antiochos III in the Gulf after he returned from his *Anabasis* in 205 BC and restored order in

[4] Unless stated otherwise all information in this chapter is based on Callot, 1994b: 255-256; Callot, 2004: 20, 122, 128 & 140-145; Haerinck, 1998c: 25-28 & Haerinck, 1999: 126-127.
[5] Robin, 1974.
[6] Grave, Bird & Potts, 1996: 76.

the region. He travelled through NE-Arabia and Gerrha was forced to pay tribute, Tylos probably underwent the same measure and on Ikaros, which was taken back by force, a Hellenistic colony was installed. Antiochos III did put an end to a number of local coinages, which competed with his own currency. During this period (PIR A, 3rd - ½ 2nd c BC) Mleiha was a large site occupied by dwellings made of light materials with a rapidly sedentarising nomadic population. The position of Mleiha in this early period was most likely that of the seat of a small local 'kingdom'. The area may have been so remote from the main Seleucid marketplaces in SW-Iran, Mesopotamia and the West, that direct trade with the Seleucid Empire probably never took place.

However the Seleucid domination on the Arabian coast of the Gulf was short-lived and they abandoned the region not long after the death of Antiochus IV (around 160-150 BC). The vacuum left by the disappearance of the Seleucid Empire was slowly replaced in the north by the small kingdom of Characene. The material remains excavated at Mleiha dating to PIR B (middle of the 2nd c BC - 1st c BC) show the growing importance of the site as a trading centre in the Oman Peninsula. The coins, only known from Mleiha, of good quality and style are still limited however and they might still correspond to a prestige coinage minted for political and possibly military purposes.

At the beginning of PIR C (1st c BC - 2nd c AD), the occupied area of Mleiha appears to reduce in size. Many cultural characteristics change, without any major break with the previous periods. In this phase the site of ed-Dur developed, forming a cultural and political entity with Mleiha. During the 1st c AD the maritime trade between the West and the Indian subcontinent boomed. One of the most speaking pieces of evidence is the *Periplus Maris Erythraei.* Many actors participated in this trade network (Romans, Greeks, Persians, Indians, etc.), among them also Arabians. Although SE-Arabia is not directly situated on this major trade artery, it is on one of the contributories through the Gulf from and towards Characene. This is the context when the bulk of the SE-Arabian coins appeared. Within this frame foreign coins show up for the first time in the Oman Peninsula. The SE-Arabian coins dating to PIR C are marked by the appearance of a new feature on all coins, the trident monogram symbol. The sudden emergence of this symbol and the minting of bigger amount of coins may be a sign of political and economical change in the Oman Peninsula induced by commercial activities. It is in this period that SE-Arabia omitted the NE-Arabian middlemen and either organised its own trade or sea-borne trade came to there. The role of Characene is probably considerable in organizing this new system and maybe it was the latter that wanted to bypass NE-Arabia and extend its influence and trade network. This does not mean however that the traditional trans-Arabian caravan routes disappeared, but rather that they were supplemented by sea trade contacts.

Between the end of PIR C and the beginning of PIR D (end 2nd c AD – beginning of 3rd c AD), if not earlier, ed-Dur was abandoned and Mleiha shrunk to the size of a large village, with dwellings clustered around a large fortified residence. The *archaeologica* found at Mleiha from that period however still included imported objects from Mesopotamia and the Mediterranean, as well as from the Persian coast and India, the African coast of the Red Sea and even from Egypt. This points to the fact that there was access to the sea trade routes of the Indian Ocean, which connected Egypt with India via the Red Sea. This opening to sea trade coincides with the foundation of the seaports of Sohar and, shortly after, Kush. The major changes in the Gulf region were the disappearance of the semi-independent kingdom of Characene and the emergence of a new power, the Sasanians. The organisation of the Sasanian economy was much more centralised than that of the Parthians and they sought full control over the Gulf traffic. Sasanian coins started circulating from the 3rd c AD. None were encountered at ed-Dur, but in SE-Arabia two Sasanian coins are known from the Island of Ghallah (situated in the bay in front of ed-Dur) and one from Tell Abraq[7]. The importance

[7] Potts & Cribb, 1995: 126-130.

of the coins found at Ghallah lies in the fact that they prove that at least some activity was still going on during the 3rd c AD in the region. Probably the very stylised Arabian coins were the last to be minted in the Peninsula and they must date from after the heydays (PIR C) of ed-Dur because none were found during the Belgian excavations. The French team on the contrary found one such coin in area F (dated to the 3rd/4th c AD). The fact that these iconographically very deteriorated coins occur all over E-Arabia indicates that the camel caravan traffic between NE- and SE-Arabia, that had never completely disappeared, was restored and/or that a limited sea-borne trade between both regions was going on.

- **Abi'el**

A last word should be dedicated to the name Abi'el. This must be the name of an important person during the 3rd or early 2nd c BC. Linking the name to an actual historic figure has proven difficult and different hypothesis have been proposed. The name Abi'el is present as an Aramaic inscription behind the seated figure on the reverse side of many of the Arabian coins. A second group of coins has a more extended inscription, "*Abi'el, son/daughter of*", although most of the time it is only partly legible.[8]

The first suggestion was that Abi'el was a NE-Arabia king who ruled relatively close to Gerrha and Bahrain. The appearance of his name on the SE-Arabian coins was just the result of copying earlier NE-Arabian issues.[9]

D.T. Potts, followed by E. Haerinck, suggested however that his realm has to be located in SE-Arabia and that the coins date back to the early 2nd c BC[10]. Some of the early Abi'el coins were found on Bahrain, Thaj and Jebel Kenzan, which suggest that this was also an accepted currency in NE-Arabia. Moreover some were found at Susa where they most likely ended up as a result of commercial contacts between Arabia and SW-Iran. If this suggestion were accepted it would put SE-Arabia during this early period in a completely different daylight, which also has implication for the political, economic and historical situation in NE-Arabia[11]. The second and largest group of these Abi'el coins date to the 1st c BC - 1st c AD and were only found and most probably also only used in SE-Arabia. These latter issues may have repeated the name of an important political figure in the region long after that individual died, just as many Asiatic issues that were based on those of Alexander repeated the name of the Macedonian king over and over again, even centuries after his lifetime.[12]

A third suggestion came from O. Callot in that there might have been a second ruler with the name of Abi'el[13].

[8] Maraqten, 1996: 304.
[9] Callot, 1994b: 356.
[10] Potts, 1991: 109; Potts, 1994b: 82; Haerinck, 1999: 126. E. Haerinck sees no reason to push this date further back to the 3rd c BC.
[11] Haerinck, 1999: 126-127.
[12] Potts, 1997a: 65.
[13] Callot, 2004: 126-127.

7.3. Typological groups

7.3.1. Introduction

In the frame of this study the typology devised by D.T. Potts was used to classify the coins[14]. Typologically he distinguished roughly sixty different sub-categories that could be broadly assigned to two different traditions: one large group of issues with considerable stylistic homogeneity that originates from half a dozen sites in NE-Saudi Arabia (e.g. Thaj, Hofuf, Jabel Kenzan, etc.), and a second group is primarily known from the sites of ed-Dur and Mleiha (U.A.E.). The earliest issues probably date to the late 3rd c BC, while the latest were minted at least as late as the 1st c AD, but they must have stayed in circulation into the 3rd or early 4th c AD considering the coins found by the French team in Area F at ed-Dur.[15]

A more recent study by O. Callot[16] on the coin collection of the Sharjah Archaeological Museum proposes a different, but in broad strokes similar, typological grouping. This study was evaluated but not used as a typological frame since some types of coins presented here do not occur among the Sharjah collection and it was thought better not to mix the two systems. The classes defined by D.T. Potts that are relevant for the studied coin assemblage will be briefly presented. The registration numbers of the coins belonging to these classes are listed according to their denomination (tetradrachm, drachm or obol). Most of the coins were already classified and discussed by E. Haerinck[17], his designations is largely followed, except for some instances where the coin in question was left un-classified because of bad preservation. Then the ed-Dur assemblage is placed in a wider frame, and here we will mention the broad typological evolutions as described by D.T. Potts and O. Callot and the subject of struck *versus* cast coins is touched upon.

7.3.2. Typological groups present in the ed-Dur assemblage

As mentioned before the vast majority of the coins were modelled on the coinage of Alexander the Great. They show the iconographically deteriorated head of Heracles wearing the pelt of the Nemean lion on the obverse or, less likely, the head of Alexander with the Amon horn. On the reverse a seated figure on a throne holding a staff is depicted, based on that of Zeus Aetophorus. In SE-Arabia however the person is to be interpreted as the representation of Shams/Shamash, the solar deity worshipped by the inhabitants of this region. On coins from NE-Arabia the name of the Semitic sun god Shamash is sometimes written in full in S-Arabian characters, but most of the time only the letter *shin* is represented (abbreviated as s^2). One of the peculiarities of SE-Arabian coinage is that no Greek inscriptions appear on their issues, but an angular lapidary-style Aramaic inscription mentioning the name Abi'el. The seated figure is usually shown supporting an eagle, a horse protome/rython or a full horse on his outstretched right arm and various monograms occur around him.[18]

Although the iconography and typology are not dealt with in detail in this study I will just briefly mention some of the monograms typical for the SE-Arabian coins:

- The palm tree is always present on the Abi'el issues with horse protome or full horse, but not on the eagle issues, except maybe on some obols. This could be the monogram of Mleiha, which was largely a provider of agricultural products.[19]
- The trident/anchor monogram must have had a special meaning. It is suggested to be the symbol of the coastal site of ed-Dur, since it is the most common monogram found at the

[14] Potts, 1991; Potts, 1994b.
[15] Grave, Bird & Potts, 1996: 77.
[16] Callot, 2004.
[17] Haerinck, 1998b.
[18] Grave, Bird & Potts, 1996: 77; Potts, 1994b: 43 & 1997a: 65; Haerinck, 1999: 127.
[19] Haerinck, unpublished text.

'late' Abi'el issues. It is always present, appearing together with the full horse on the hand of the seated person and the palm tree. This trident/anchor could point to the maritime activities at ed-Dur.[20] According to D.T. Potts this monogram probably derived from the three-pronged anchor, one of the royal symbols of the Seleucids and an image frequently found on their coinage and on the coins of Elymais[21]. For the moment this derivation seems more satisfactory than an attempt to link it to the fork-shaped *Blitzbündel*-symbol widely attested in S-Arabia[22]. O. Callot has doubt towards this interpretation because the Seleucid anchors all have an additional 'anchor-stick', not presented on the SE-Arabian symbol. The comparison with the Elymaic coins is more acceptable. But maybe this monogram, HE, imitates a Greek prototype that through copying and re-use by non-Greeks became a simple symbol. The only thing that can be said is that the use of this monogram is definitely linked to the Oman Peninsula.[23] One coin is known where the monogram was added after it was struck on the obverse instead of on the reverse side[24].

- A particular group of coins show the calliper-like monogram. The meaning of this symbol is unknown, but it also appears on a pottery sherd from ed-Dur, on a large disc-shaped lead 'ingot' (see *Chapter 6*), on a stone seal and on a finger ring. Identical or closely related monograms are also to be seen on coins from Characene and some issues from Seleucia. It also appears on a Characenean seal found at Kharg Island. It is suggested that there was probably a link between the signs on all of these objects and the coins from Mesopotamia and SE-Arabia. Commercial and possibly political ties with Characene may be relevant in this regard.[25]

- Iconographically deteriorated Abi'el issues where the seated person on the reverse has a full horse depicted on his outstretched arm are very common at ed-Dur. With the other hand he holds a staff. Behind the staff is a cursive or lapidary Aramaic inscription of 3 or 4 letters with the name Abi'el (though sometimes the inscription is defective or illegible). Coins of this full horse type have never been found outside Mleiha or ed-Dur in controlled excavations (except 1 coin recently found at the site of Khor Rori). At ed-Dur they represent slightly more than 70% of the excavated coins and we may conclude that this type was in local use only. One of the particularities of this group is the fact that quite often a dot, most likely a wart, is indicated on the cheek of the head on the obverse side of the coin. A palm tree and a trident monogram are always present.[26]

• **Class II**

There is very probably a chronological evolution to be observed within the group, there are clear stylistically differences. On the reverse a seated person is shown with his right arm raised, supporting a horse or a horse protome and the left arm curled around the staff. A date palm is depicted in front of the knees. An Aramaic legend is present but is largely illegible.[27] BO 044, BS 107, BS 068 & BK 004) are 4 obols that are stylistically very similar, especially the first 3. The staff of the seated person on the reverse is represented by a line of dots.

Obols: BK 004, BO 044, BQ 041, BS 039, BS 068, BS 082, BS 107, BS 294 & N 036 - see Fig. 77.

[20] Haerinck, unpublished text; Haerinck, 1999: 127.
[21] This was a semi-independent kingdom at the foothills of the Zagros (SW-Iran), with as capital Susa. Around 100 AD a Parthian line of rulers replaced the local dynasty.
[22] Potts, 1991: 80.
[23] Callot, 2004: 30.
[24] Haerinck 1999: 127.
[25] Haerinck, 1998b: 295-296; Haerinck, unpublished text.
[26] Haerinck, 1998b: 287-288.
[27] Potts, 1991: 20; Potts, 1994b: 53.

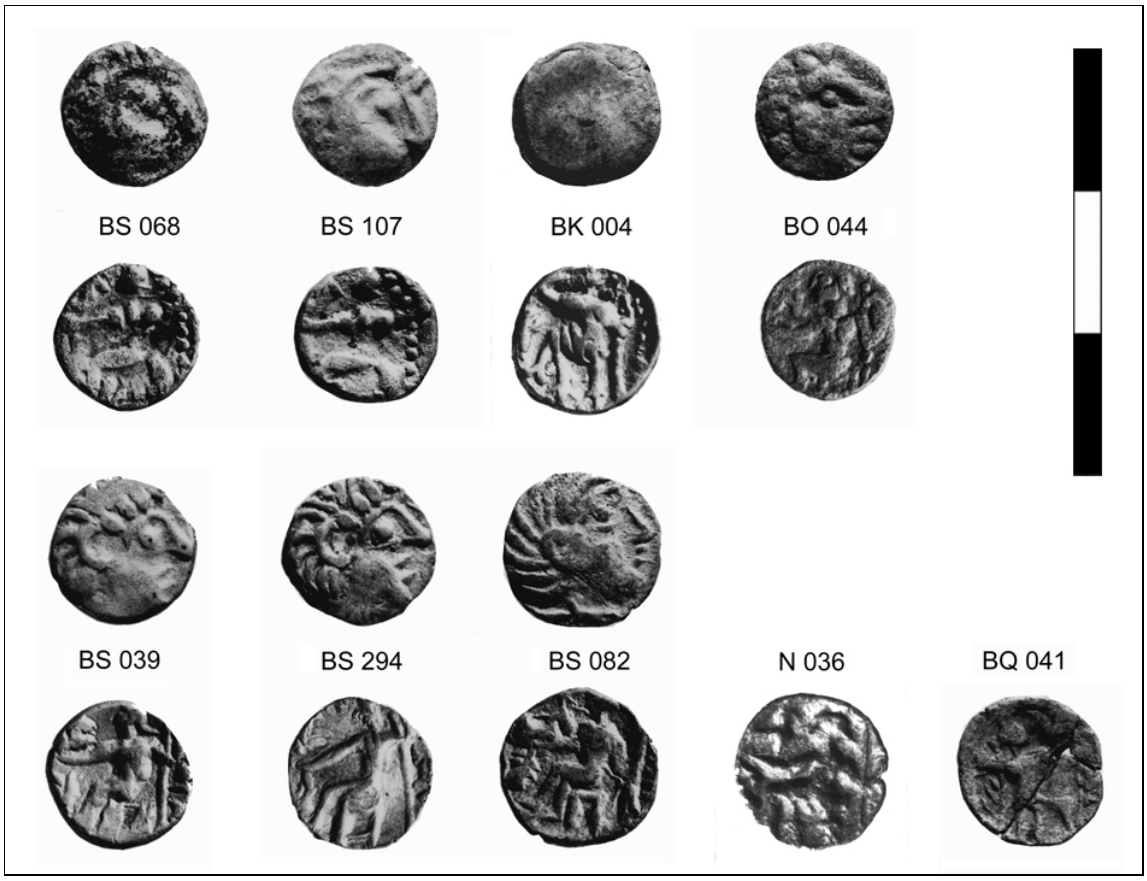


Fig. 77: coins belonging to class II.

• **Class V**

This class has in most cases a blank obverse and a seated figure with outstretched arm supporting an eagle is found on the reverse. The left arm is curled around the staff with a stylised date palm in front of the knees. Normally no legend is shown.[28]

Obol: N 114 - see Fig. 79.

• **Class XIVc**

All of these coins have a black obverse. On the reverse a seated figure with outstretched right arm supports an eagle (which is not always as clear). The left arm is curled around a staff and a vertical *shin* is shown in front of the knees. As to their chronology D.T. Potts states that they probably were already circulating in the 3rd c BC. At any rate they were still in circulation in the 1st c AD during the heyday of ed-Dur, as shown by the excavations.[29]

Obols: AH 058, AV 014, BM 027, BO 055, BQ 157, BS 027, BS 098, BS 157 & BS 160 - see Fig. 78.

[28] Potts, 1991: 23; Potts, 1994b: 48.
[29] Potts, 1994b: 18; Haerinck, 1998b: 280.

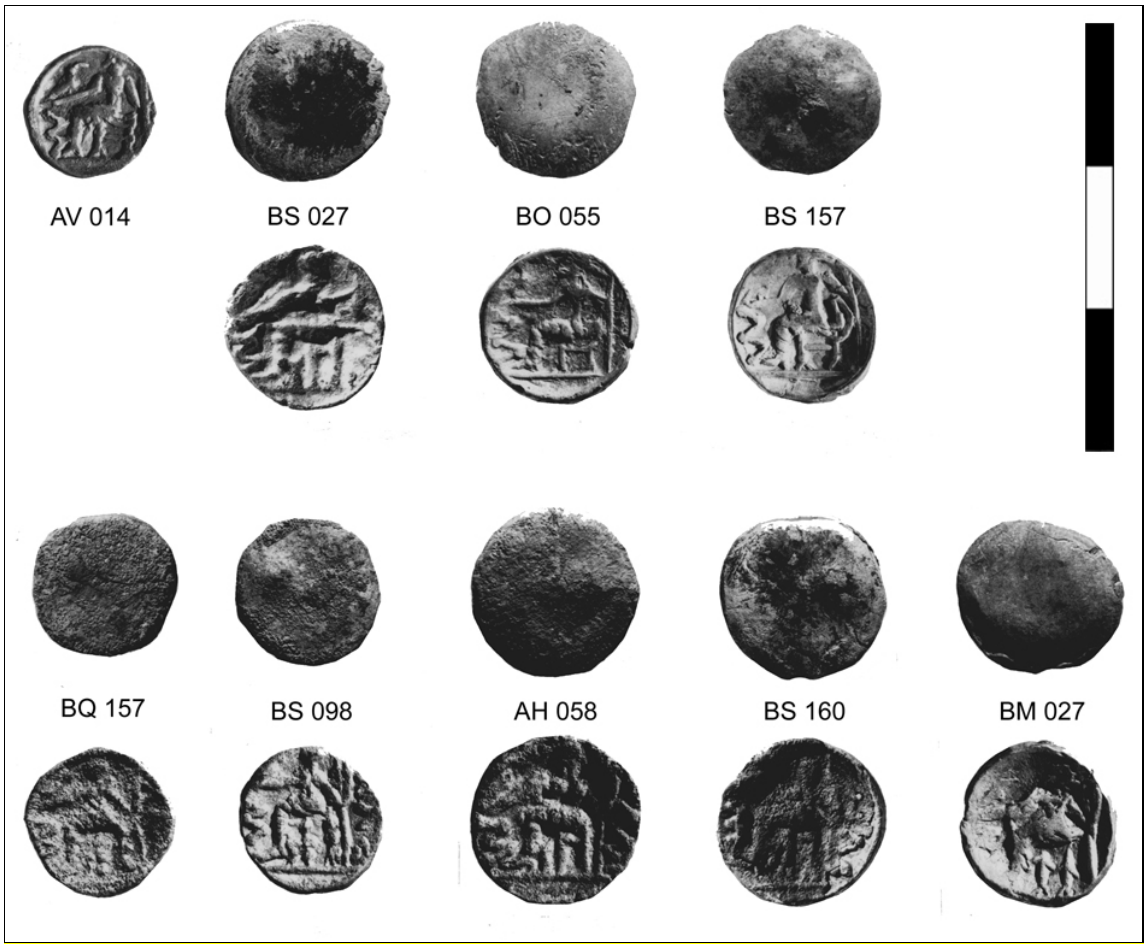


Fig. 78: Coins belonging to class XIVc.

• **Class XXII-XXIII**

These iconographically deteriorated coins have a blank obverse and a very stylised figure on the reverse with on the outstretched right arm a horse (XXII) or eagle (XXIII). In front of the knee of the seated person a vertical *shin* is depicted. As to chronology, these most likely represent the final stage of the local coin issues in E-Arabia. It is important that not a single coin of this type was found during the Belgian excavations. A coin of this type was however found during the French excavations at area F. The latest occupation of this area dates to the 3rd/4th c AD. This points to a late use, and possibly date, for these iconographically deteriorated issues.[30]

• **Class XLI**

This class groups all abstract coin that do not really fit the other types described by D.T. Potts. Each coin has a blank obverse[31]. The main reason that we place 3 coins in this group is the absence of clear dots and circles (typical for the other abstract groups).

Obols: AV 161, BS 109 & ED 010 - see Fig. 79.

[30] Potts, 1991: 47-48; Potts, 1994b: 25-26; Haerinck, 1998b: 281.
[31] Potts, 1991: 76; Potts, 1994b: 37.

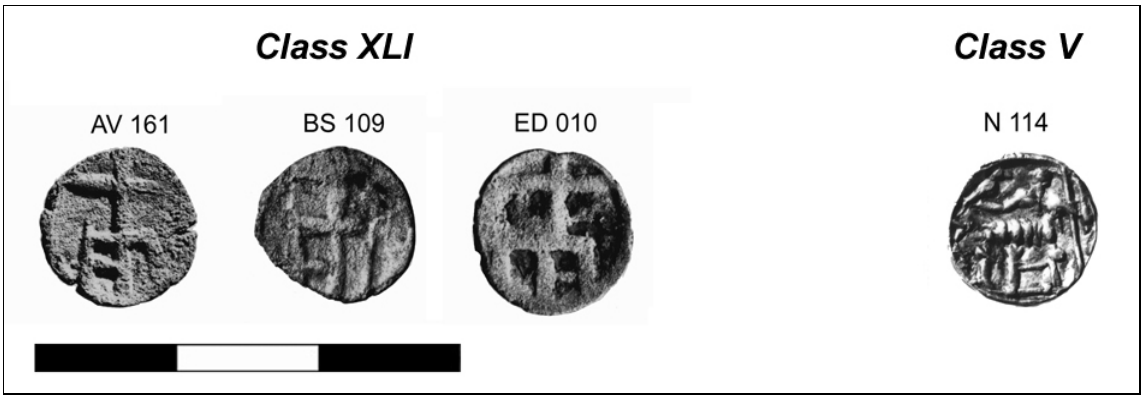


Fig. 79: coins belonging to class XLV & class V.

• **Class XLIV**

This class has an iconographically deteriorated head of Heracles on the obverse and the facial features are more exaggerated than is normally the case in NE-Arabia. The nose, eye and ear are often large. The pelt of the Nemean lion is rendered variously, but in this class it has the appearance of a series of chevrons in a herringbone pattern that envelop the entire face, covering the neck as well. The open jaw of the lion has been transformed into an unattached crescent, resembling a curved horn. The reverse shows an enthroned male, nude from the waist up, and draped around the legs. The rather realistic body is broad shouldered, has a narrow waist, and is slightly modelled. Three parallel lines sometimes indicate the stomach muscles. The figure wears a brimmed hat not unlike a cavalry helmet. The enthroned figure supports a realistic-looking horse, on his outstretched right arm. The left arm is bent at the elbow and curved around the staff. Sometimes nothing is to be seen above the throne, but two or more parallel, horizontal bars or a vertical dotted line are occasionally observed behind the back of the figure. Pointing in, towards the chest of the seated figure, is the trident monogram. Further to the left we find a stylised palm tree. The text is written in lapidary, i.e. rectilinear style.[32]

Obols: BS 050 & BS 275 (possibly same die, but not completely same die orientation) - see Fig. 80.

Drachms: BQ 054, BR 105, BS 091, BS 101 & M 073 (BS 091 & BS 101 possibly same die) - see Fig. 80.

[32] Potts, 1991: 79-80; Potts, 1994b: 62-63; Haerinck, 1998b: 288-289.

Fig. 80: Coins belonging to class XLIV.

- **Class XLV**

This class of coins is generally similar to the previous with however some important differences. The obverse portraits are generally comparable though slightly more barbarised, with the eye, nose, lips, and chin all somewhat exaggerated. The torso, arms, and legs of the reverse figure tend to be very flat and featureless, and the person always faces right. Normally no drapery is shown, but exceptions exist. The palm tree on these coins is usually linear, the base of which is bulbous. Often though not always, a short bar runs parallel to the base of the trident monogram. The legend is problematic and is similar to that of XLIV. This group only exists in the tetradrachm denomination.[33]

Tetradrachms: BM 026, BQ 137, BS 254, BS 284, & BS 285 - see Fig. 81.

[33] Potts, 1991: 81; Potts, 1994b: 65; Haerinck, 1998b: 289-290.

Fig. 81: Coins belonging to class XLV.

- **Class XLVI**

The iconography of this class is identical to that of the foregoing two, but there are clear stylistic differences. There is considerable variety in the representation of the obverse portrait, ranging from the relatively delicate features to more iconographically deteriorated. The reverse figure faces left and is distinguished stylistically by his generally triangular torso with narrow waist and broad shoulders, which is often accentuated by dots indicating the nipples and the navel. The right arm, which may be either bent or straight, supports a horse. In general the trident monogram is shown under the arm of the seated figure, pointing in towards the chest, although on several examples it is either pointing away or missing altogether. On the tetradrachms the palm in front of the figure's knee is rendered more realistically than in classes XLIV and XLV. There is a certain amount of diversity in the rendering of the reverse legend.[34]

Obols: BQ 144, BQ 152, BS 102, BS 114, BS 158, BS 259, ED 001 & ED 007 (very similar are BS 102 & ED 001 and BQ 144 & BQ 152) - see Fig. 82.
Drachms: BO 040, BQ 143, BS 033 (trident pointing outwards), BS 183 & M 078 (all very similar) - see Fig. 82.
Tetradrachm: BS 096 (has a very realistic obverse head) - see Fig. 82.

[34] Potts, 1991: 84; Potts, 1994b: 66.

Fig. 82: Coins belonging to class XLVI.

- **Class XLVII**

This is by far the most common group of coins in SE-Arabia. This class shares the iconography of classes XLIV-XLVI, but the style and execution are very different. The obverse portrait is normally very iconographically deteriorated, showing a large nose, thick lips, almond-shaped eye with a marked dot in the centre, and large 'horn' in stead of the jaw of the Nemean lion. The lesion or wart is commonly depicted. The arms of the enthroned figure on the revere are now shown as a horizontal line running across the coin face. The torso is rendered as 2 parallel, vertical lines with an open space between them. Occasionally a dot may represent the navel. The head tends to appear as a sunburst with spikes of hair emanating from a round, featureless skull. The drapery on the thighs resembles a spiky fringe. The horse supported by the outstretched right arm is normally very stylised. The palm in front of the figure's knees tends to be larger and fuller, with more individual palm fronds. The trident monogram is usually horizontal, running directly parallel to the right arm and pointing in towards the stomach. A vertical row of dots representing the back of the throne is sometimes seen behind the figure's back, but more commonly the back and the back legs of the throne are shown as a single vertical line which also represents the staff. The legend is often very deteriorated, but in some cases appears to be the same as that on the coins of class XLIV. In other cases, it may represent a different legend.[35]

Here two additional subgroups were made within the obols. *Obols-1* are more 'realistic' and the depictions are more detailed, e.g. the trident monogram is clearly rendered. *Obols-2* are much more iconographically deteriorated and this is especially clear with the trident monogram, it is depicted rather by 3 lines than by the entire monogram.

Obols-1:	BK 003, BQ 145, BQ 147, BS 156, BS 159, BS 162, BS 170, M 077 & N 002 (BK 003 & BS 159 possibly same die) - see Fig. 83.
Obols-2:	BS 262, BQ 138, BQ 139, BQ 148, BQ 149, BQ 150, BS 279, FO 001, M 068 & M 074 (BQ 150, BS 262, FO 001 & M 068 very similar) - see Fig. 83.
Drachms:	BO 062, BQ 104, BR 100, BS 106, BS 127 & M 079 - see Fig. 84.
Tetradrachms:	BJ 008 (calliper monogram), BO 043, BQ 005, BQ 142, BS 169 & N 310 - see Fig. 85.

[35] Potts, 1991: 86; Potts, 1994b: 68; Haerinck, 1998b: 292-293.

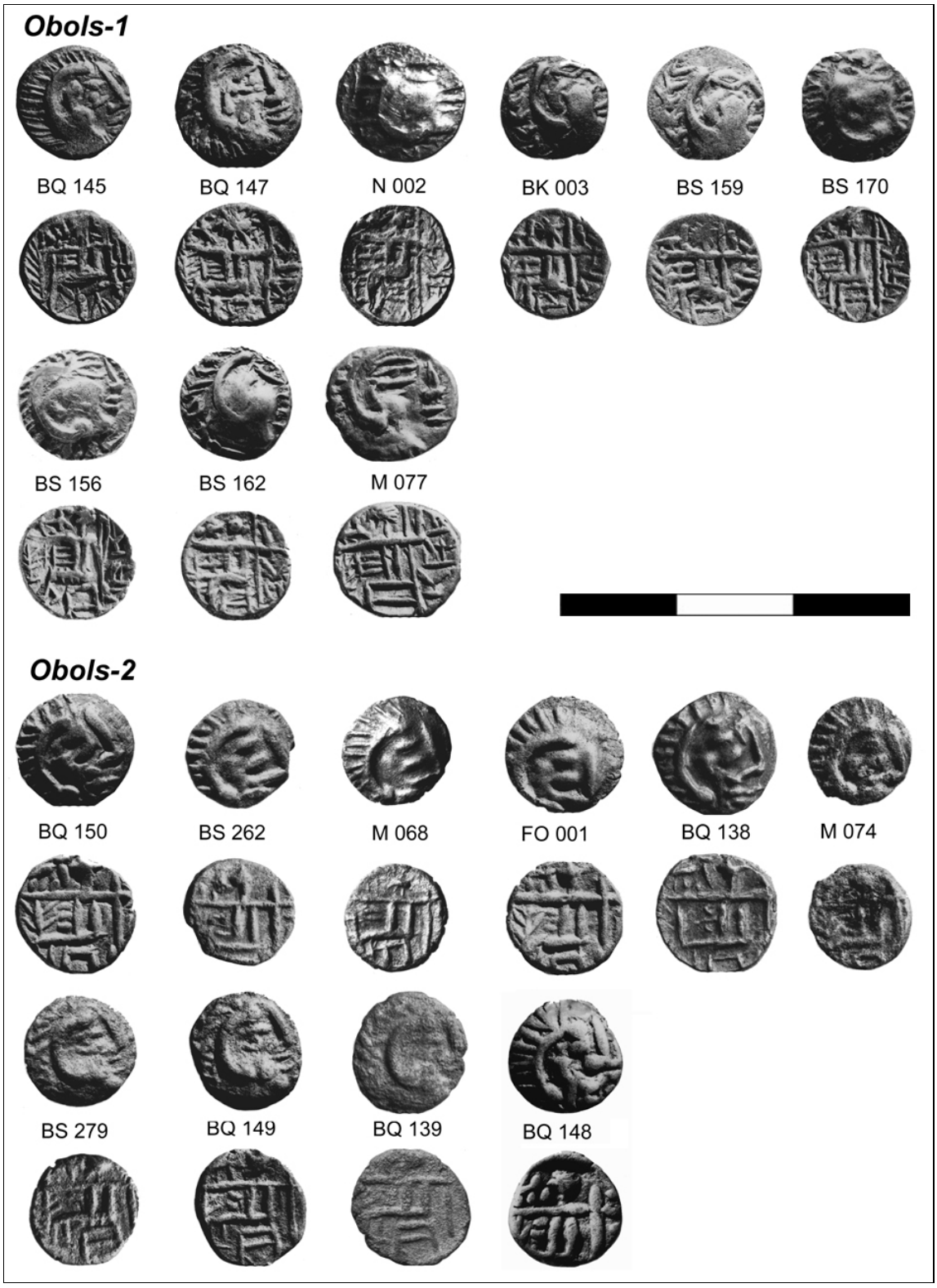


Fig. 83: Coins belonging to class XLVII - obols.

Fig. 84: Coins belonging to class XLVII – drachms.

Fig. 85: Coins belonging to class XLVII - tetradrachms.

• **Class XLVIIIa & b**

This class is stylistically speaking closely related to class XLIV. This applies both to the obverse head of Heracles, where the lesion or wart sometimes appears, and the enthroned person on the reverse. The latter is usually shown with some degree of naturalistic modelling. This may be extremely life-like where the pectorals and the stomach muscles are clearly defined. In other cases, the figure is flat. The reverse figure is usually shown wearing a hat, the brim of which sometimes resembles the cavalry helmet of class XLIV, and is sometimes less broad, more like a woollen cap. The horse supported by the outstretched right arm of the enthroned figure is often very naturalistic. The legend most closely resembles that of class XLIV. The trident monogram is now depicted behind the back of the figure in an upward position. The space in front of the figure has been filled by an *'l* (Λ-monogram). The palm tree normally has a broad trunk and may be an attempt to depict a young palm tree in a realistic way. The division between *type a* and *type b* is based on the presence (*a*) or absence (*b*) of drapery on the legs. This class only appears in the tetradrachm denomination.[36]

Among the photographs published by D.T. Potts several coins may have originated from the same die. The depictions are also neatly imprinted on the coins, this could point to the fact that better quality dies were used (harder) or to the fact that the alloy used was better suited for striking.

Tetradrachms *type a*: AC 012, AO 018, AV 023, BS 043, BS 071, BS 148, & BS 172 - see Fig. 86.
type b: BS 235, BS 236, BS 237, BR 106, ED 005, M 063 & M 064 - see Fig. 86.
unclear: AD 025[37] - see Fig. 86.

• **Class XLIX**

In the treatment of the pelt of the Nemean lion the obverse head on the tetradrachms is unlike that of any other series. Here the fur resembles tightly braised rows of hair. The lesion or wart is found just below the right eye. Stylistically the reverse figure most closely resembles that of class XLV with its flat, featureless torso and thin arms, supporting a horse. There is an obvious link with class XLVIII in that the trident monogram occurs in the same place in both classes. The place of the *'l* (Λ-monogram) is now taken by a large cone-shaped object (a club as one of the symbols of Heracles?).[38]

Tetradrachms: BS 080 & BS 097 - see Fig. 87.

• **Class S5**

On the reverse the seated figure is depicts with outstretched arm supporting a horse protome and the left arm curled around a staff. There is a date palm in front of the knees and behind the figures staff an upward pointing trident monogram can be seen.[39]

Drachms: BS 069 & BS 171 - see Fig. 87.

[36] Potts, 1991: 90; Potts, 1994b: 74-75; Haerinck, 1998b: 297-298.
[37] The 3 underlined coins are from the British excavations at ed-Dur.
[38] Potts, 1991: 94; Haerinck, 1994b: 10-11; Potts, 1994b: 76. Concerning this 'club' G. De Wilde (1994: 249) makes an interesting comparison to a *lingam* (an Indian fertility symbol of Shiva).
[39] Potts, 1994b: 60.

Fig. 86: Coins belonging to class XLVIIIa & b.

Fig. 87: Coins belonging to class XLIX & class S5.

• **Undetermined**

Almost all coins from ed-Dur could be attributed to one of the classes in the typology of D.T. Potts. What follows are the coins that could not be classified (see Fig. 88):

- ED 002 could not be allocated to one specific group but the obverse head is realistically rendered, possibly related to the Seleucid-inspired obols in class XII as described by D.T. Potts[40].
- Two silver tetradrachms (AG 003 & BQ 136) could not be determined. AG 003 has some resemblance to Potts' class LI and BQ 136 to class II[41]. Both have high silver contents (respectively 94 and 96 wt%, see below), which makes them exceptional among the tetradrachm assemblage.
- Some coins were so badly preserved that none of the iconography could be seen or too much detail was lost, to classify them (Obols: BM 028, BQ 125, BR 099, BR 101, BS 040, BS 108, BS 234 & ED 012; drachm: BS 286).

[40] Potts, 1991: 32.
[41] Respectively Potts, 1991: 96; Potts, 1994b: 41 & Potts, 1991: 20.

Fig. 88: Undetermined coins.

7.3.3. The ed-Dur assemblage in a broader context

• **Arabian coins**

Table 33 combines the coins published by D.T. Potts, E. Haerinck and O. Callot in order to give a general impression of the number of coins and their place of origin. Only the types that are attested (whether by excavation or from private collection) at Mleiha or ed-Dur are included, so the types that only occur in NE-Arabia are left out. It should however be kept in mind that the majority of these coins are from private collections and that the place of origin mentioned, has to be treated with caution. The excavated coins from ed-Dur are entered in a separate column. Coin types that are thought to be of SE-Arabian origin are grouped in the lower part of the table.

To the coins definitely originating from the SE-Arabia (so far only attested at ed-Dur or Mleiha) only a few can be added from other regions:

- A coin belonging to class XLVIIIb was recently excavated at the site of Khor Rori (Oman).
- A small collection of coins picked up near Hofuf (Saudi Arabia) is said to have contained a tetradrachm assignable to one of the classes found at ed-Dur (British Museum note made available to D.T. Potts by the late N.M. Lowick)[42].
- A coin of class XLVII first reported to have been found at Shabwa in S-Arabia (Yemen) and present in the National Museum catalogue of Yemen[43] later appeared to be unprovenaced[44].
- One (type XLVII) tetradrachm was found at Sialkot (Afghanistan)[45].
- Recently some 12 coins (?) were found in the Emirate of Fujairah, but as yet no further details are available[46].

The coins of Fujairah should however be seen in a different light. Recently a collective tomb at Dibba Al Hasn was excavated by the Sharjah Archaeological Museum, which yielded material identical or at least very similar to that of ed-Dur[47]. This lends support to the obvious, but till now materially unproven, hypothesis that next to the big sites of ed-Dur and Mleiha smaller sites existed. The location of Dibba is very interesting, since it is located at the coast of the Gulf of Oman, the other side of the U.A.E., and actually situated much closer to the know sea-trading networks from the 1st c BC – 1st AD. This would place Dibba within the *inter*-site trade because of the identical or at least very similar cultural artefacts.

Within the ed-Dur coin assemblage excavated by the Belgian team the obols (61) are best represented, than the tetradrachms (28) and as last the drachms (19). In the total SE-Arabian numismatic collection however the obols are the least represented (see Table 33 above) followed by the drachms and with the de tetradrachms making up 61% of all coins. Within the coins that are supposed to originate from NE-Arabia (the first part of Table 33) the obols have the overwhelming majority, followed by the drachms. What does this tell us? First of all tetradrachms must have been a denomination used on a local SE-Arabian scale, only used as currency between the sites in the region or within the settlements. Why the coin assemblage studied here does not confirm this, is not clear. Secondly the obols probably were the main form of payment in the rest of E-Arabia, where tetradrachms are few.

[42] Potts, 1991: 107.
[43] Potts, 1990b: 290; 1991: 107.
[44] Potts, 1994b: 68.
[45] De Wilde, 1994: 249 (note 1). This is the only reference to this coin and the statement of G. De Wilde is based on a personal comment by D.T. Potts.
[46] Haerinck pers. comm.
[47] Jasim, 2006.

Typology Potts	Denomination	**Ed-Dur**	**Ed-Dur (exc.)**	**Ghallah island**	**Mleiha**	**Ash-Sha'ba**	**Ayn Jawan**	**Salt Mine Site**	**Jabal Kenzan**	**Hofuf**	**Thaj**	**Failaka**	**Yemen**	**Khor Rori**	**Bahrain**	**? / Other sites**	*Total # per type & denomination*
I	T										1						1
	D								2								2
	O				1				3	1							5
	?											10				6	16
XVII	O				1	2			8	4	13						28
XI	O				2				6		1						9
XXI – XXIII	-	5			3				9		7					2	26
XXV	-		2*		1				5		2						10
XXVI	-				1	2			15		8					1	27
XXVII	D	6				1			10	1	3						21
XXX	-	2							5		3					1	11
XXXI	D	4				2	1		11	1	6						25
XXXII	-	14				2			32		19						67
XXXIII	-	7							6								13
XXXVIII	-	2							9		1						12
XXXIX	-	1				2			3		4						10
XLI	-		3			2			7	1							13
XLII	O				3				6	1	1	1					12
II	T				1											1	2
	D				2												2
	O	8	5**		57				8	3	1					6	88
	?														77		77
V	O	15	1		1	2	2		4	2	3						30
VIII	D	1			3	1											5
XIVa	O	1			4						1						6
XIVb/c	O	27	9		14	2	2	3	15	9	1						82
		SE-Arabian issues															
XLIV	T	3			1											1	5
	D	11	5		19											7	42
	O		2		4												6
XLV	T	76	5	3	22									1			107
XLVI	T	19	1		2												22
	D	3	5		1												9
	O	8	8		12												28
XLVII	T	55	6		38								1			8	108
	D	17	6		30												53
	O	13	19		9											2	43
XLVIIIa	T	26	8		7												41
XLVIIIb	T	18	7		7									1			33
XLIX	T	11	2		1												14
	D	1			1												2
L	T	31			7											1	39
	O	3			1												4
LI	T	1	1?		1												3
S1	O	1			1					1							3
S2	T				15												15
	D	1			7***											1	9
	O				3												3
S3	-				2				1								3
S4	T				3												3
	D	1			5												6
	O	2			8												10
S5	T	1			4											1	6
	D	2	2		3												7
	O	1			8				1								10
Undetermined	-		17		1												18
Total # per site		398	114	3	317	18	5	3	166	24	75	11	1	2	77	38	*1252*

Table 33[48]: Published numismatic data according to the classes defined by D.T. Potts[49].

[48] ***** Found in area F by the French team, 1 drachm and 1 obol; ****** Potts proposes a late 2nd c BC date for class II (Potts, 1994b: 82), which does not correspond well with the main occupation phase of ed-Dur; ******* Found by the French team at Mleiha (Haerinck, 1999: 124).

[49] Based on the typology defined by Potts, 1991 and expanded in Potts 1994b. Additional coins published by Boucharlat & Drieux, 1991; Haerinck, 1998b & 1999 and Callot, 2004 are included.

• **Indication of internal chronology**

Most coins collected at ed-Dur were just surface finds and have no meaningful stratigraphy attached to them; moreover the site itself shows no or very little stratigraphic sequencing. This makes it difficult to put the different classes in a chronological sequence. The only real hint from ed-Dur is a coin of Class XXII-XXV found by the French team in area F, generally dated to PIR D, suggesting that iconographically more stylised coins are younger than realistic ones[50]. One of the coins presented here from the British team was found at the lower foundation level of the small fort of ed-Dur[51].

The site of Mleiha is stratified and could be more helpful on this topic, but also there most coins are surface finds. Some exceptions are known however. R. Boucharlat and M. Drieux state that one is generally tended to attribute earlier dates to the more realistic coins and a later to the more stylised, iconographically deteriorated varieties. A realistic coin (tetradrachm of class L) found in a deep level dating to the late 3rd – mid 2nd c BC, seems to confirm this view. However another realistic coin originated from a 1st c AD context[52]. So there seems to be an inconsistency with the statement that younger coins are more stylised. They do however tend to forget that some coins stayed in circulation for a considerable length of time, and that it is by no means sure that a coin found in a 1st c AD context, was also struck during that period.

D.T. Potts also says that a different state of debasement does not necessarily imply a chronological evolution. Copies of widely differing quality can be made at the same time in different areas, depending on factors such as the skills of the craftsmen, the availability of good examples to copy, etc. What the coins excavated at ed-Dur do show is that the chronological range of some of the types is between the late 1st c BC - 1st c AD and that some NE-Arabian coins struck earlier were still in circulation at that time[53].

Despite the absence of a clear chronological frame, it is still possible to point out some general lines of iconographic evolution. The earliest coins present at Mleiha are some silver Athenian owl imitations but none were reported from ed-Dur. These are inspired on coins from S-Arabia (Hadramaut) with whom commercial contact certainly existed from an early date[54]. O. Callot dates these coins slightly later, to the transitional period between PIR A and PIR B[55].

More important for the coins presented here are the imitation Alexander and Seleucid inspired Arabian issues. Almost all types of these coins occur in NE-Arabia and only a few of these coins have been reported from ed-Dur. A rather limited number of issues are known and they were probably minted for local exchange only. Some have a vertical *shin* on the reverse and are generally attributed to NE-Arabia.[56]

The next group of coins, although still inspired by the Alexander types, are rather freely copied imitations. On the obverse the open jaw of the lion pelt worn by Heracles gradually becomes a sort of large horn. On the reverse, the eagle held by Shamash is replaced by a rython in the shape of a horse protome. Other symbols are added such as a small stylised palm tree (symbol of Mleiha?) typical for the SE-Arabian issues. These coins are only known from the site of Mleiha and were minted in small quantities for local use. The name of Abi'el

[50] Haerinck, 1998b: 281.
[51] Pers. comm. C.S. Phillips, I do not know which one from the 3 coins is from this context, but this is relatively unimportant since they all belong to the same class, i.e. XLVIII.
[52] Boucharlat & Drieux, 1991: 114, 116 & Fig. 6. The latter coin is compared to classes X-XIII, but I do not see why. If part of one of these classes, then class XIII, but nor the eagle, nor the horizontal s², nor the name of Alexander (all typical for this type) are to be distinguished.
[53] Potts, 1990b: 64-65; Potts, 1991: 108.
[54] Haerinck, 1998c: 24.
[55] Callot, 2004: 141.
[56] Haerinck 1999: 126-127; Callot, 2004: 20, 122 & 140-141.

is still present, although it is not clear if this is a second ruler with the same name or a copying of the name of a by then legendary historic figure of Arabia. The fact that the name is often incomplete or spelled incorrect, might be an indication of the latter. Some of these coins are clearly of poorer quality.[57]

At ed-Dur some 16% of the coins found are silver obols of the vertical *shin* and eagle on the outstretched hand type (Class XIVc & S2) and coins of the protome type (S4, S5, Class II & V). Class XIVc must have been already circulating in the 3rd c BC (if the dating of the Failaka coins is correct) and they apparently stayed in use at least till the late 1st c BC, as evidenced by the coins found at ed-Dur. Potts' Class S2 is only known from Mleiha (and 1 from ed-Dur from uncertain context[58]) so they were only used or accepted in SE-Arabia. The other groups of Abi'el coins, especially class II, with horse protome could represent the next stage since they are also recorded on sites in NE-Arabia (on Bahrain, Thaj, Jebel Kenzan, etc., two were even found at Susa). It is certainly not excluded, however that they are contemporary or very close in date to Class S2. Class S4 and S5 circulated mainly in SE-Arabia while Class II (group 1) many are reported from Mleiha, but some were found at Jabal Kenzan, Hofuf and Thaj. These were likely the coins used in inter-regional Arab relations within the Gulf.[59]

The iconographically deteriorated local SE-Arabian Abi'el coins date to PIR C and are marked by the appearance of the trident monogram and the full horse on the outstretched hand of the seated person. The majority of the coins from ed-Dur belong to these types. None of these coins have been reported outside the SE-Arabia (except for the one registered example recently found at Khor Rori), but they make up about 70% of all excavated coins at ed-Dur making it likely that they were minted locally.[60]

This typical SE-Arabian iconography seems however to have been preceded by a group of transitional coins. These coins already have the trident monogram, but the sitting figure still holds a rython as a horse protome. This must be the oldest category, which O. Callot dates to the end of 1st c AD.[61] This class is not attested at ed-Dur.

Three obols of very linear style are classified under XLI and can be regarded as late issues. The very stylised classes XXII-XXV are probably the last coins struck in E-Arabia, and as mentioned before only one was found at ed-Dur by the French team in area F (dated to the 3rd – early 4th c AD)[62].

- **Foreign coins**

A brief excurse is made here to the foreign coins found at ed-Dur and Mleiha, this is only to complete the information on the coins and to point to their importance within the politico-economical reconstruction of the region. The coins will not be discussed in detail, only summed up, so when necessary they can be referred to (Table 34). The analytical results of the 8 issues excavated by the Belgian team will be presented later on.

About 7% of the coins excavated at ed-Dur are foreign, and this figure stays more or less the same if we include the coins from private collections (8 of the 116 excavated coins; 32 in total). The 32 known foreign coins include[63]:

[57] Callot, 2004: 142-143.
[58] Haerinck, 1999: 124.
[59] Haerinck, 1998b: 280; Haerinck, 1998c: 25, 27; Haerinck, 1999: 127; Haerinck, 2003: 202.
[60] Callot, 2004: 128.
[61] Callot, 2004: 129.
[62] Haerinck, 1998b: 281.
[63] Haerinck, 1998c: 30; Haerinck, 2003: 202; Whitehouse, 1998: X (Preface by Haerinck) & 67.

Place of origin	# Ed-Dur	# Mleiha	Extra
Seleucia	-	1	Dating to *ca.* 42-41 BC.
Characene	11	4	Dating to the 1st c AD. Reigns of Attambelos II (44/45 AD), Attambelos IV (58/58 AD) and Attambelos VI (104/105 AD).
S-Arabia	2	7	Hadrami coins.
Persis/Parthian	4	1	Coins of a virtually independent kingdom in Fars.
Nabataean/Palestine/ Other Mediterranean	6	1	Three coins of Aretas IV (9-40 AD), 1 from Gaza and 2 more that are difficult to attribute.
Roman	4	1	One silver from Augustus (2 BC – 11 AD), 1 copper of Tiberius (minted under Augustus, 10-11 AD) and 2 golden coins of Tiberius (14-37 AD).
Indian	5	2	Dating to *ca.* 150-75 BC.
	32	***17***	

Table 34: Foreign coins at ed-Dur and Mleiha[64].

As can be seen most of these coins date to the 1st c AD, the heydays of ed-Dur. Whether the foreign coins are the result of monetarised trade or rather reached the site as a curiosum is difficult to assess. In general the number of foreign issues of Hellenistic date registered in Arabia is small[65]. Also Roman coins are rare but there is a 17th c AD report that mentions that numerous coins of Tiberius were discovered at the coastal site of Sohar. Interesting enough coins minted under Augustus and Tiberius occur in considerable number in Indian coin hoards[66].

The number of Sasanian coins dating to 3rd and 4th c AD is rather limited in the SE- and NE-Arabia, but their presence shows that they replaced the local issues after the Sasanians came to power and started controlling the Gulf-trade. In SE-Arabia 2 Sasanian coins are know from the Island of Ghallah (a tetradrachm Ardashir I and an obol Shapur II) and 1 from Tell Abraq (drachm of Shapur II). The number of coins is higher in NE-Arabia with 2 coins from Thaj (Ardashir I), 1 from Jabal Kerzan (Shapur I), 9 from Jabal Berri (Shapur II), 2 from Al Khobar (Shapur II), 7 from Jabal Kerzan (Shapur II) and 1 from Hofuf (Shapur II). It should be mentioned that the majority of these coins were surface finds and picked up by private collectors[67]

[64] According to Haerinck, 1998a: 297.
[65] Potts, 1990: 58.
[66] Haerinck, 1998c: 30.
[67] Potts & Cribb, 1995: 126-130.

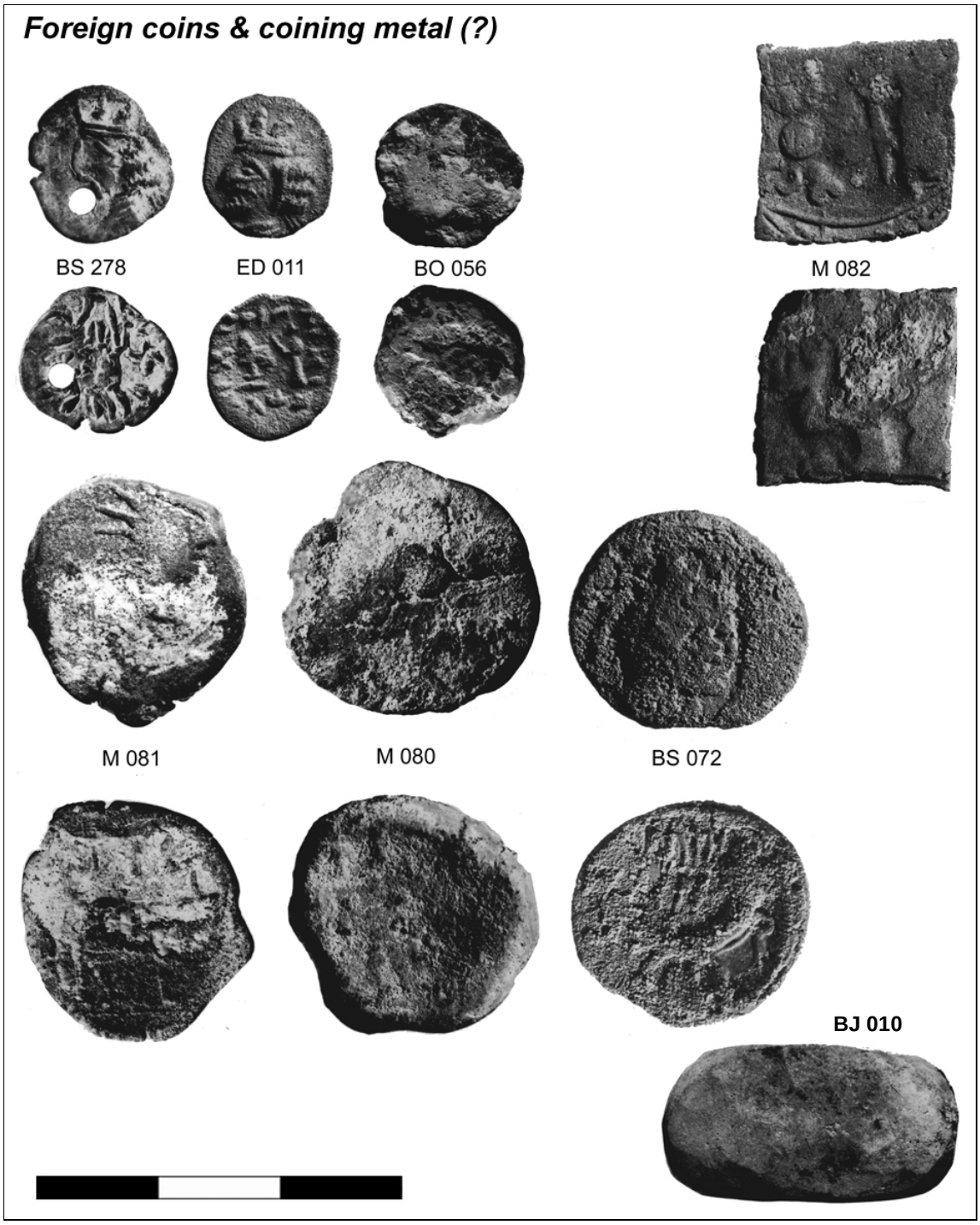


Fig. 89: Foreign coins & a possible piece of coining material (BJ 010).

7.3.4. Weight

As mentioned before there are three denominations within the SE-Arabian coins, normally described as obols, drachms and tetradrachms since they more or less follow the old Attic standard, although there is considerable variation. This standard dates back to the time of Alexander the Great (4th c BC) and remained popular long after and over a vast area. Generally we can say that the tetradrachms and the drachms are lighter than the original standard and the obols are slightly heavier (Table 35). The 'Parthian Attic standard' is also given in Table 35, and although for the obols the difference is even bigger, for the tetradrachms and the drachms the values tend to fit better. This should come as no surprise, considering the (indirect) impact that the Parthian Empire had on the Gulf. An important factor that should be taken into account is the effect of corrosion and the weight loss that results when this layer is removed. All coins were weighted after they were cleaned but some were also weighted before. The loss of weight is particularly obvious among the coins with a high copper content and much less with those that have a high silver content (more corrosion resistant).

Denomination	**Original Attic standard**	**Parthian[68] 'Attic standard'**	**ed-Dur**			
			Average	Mean	Maximum	Minimum
Tetradrachm	17,2 g	*ca.* 15,5 g	14,8 g	15,0 g	17,3 g	13,2 g
Drachm	4,25 g	4,25 - 3,75 g	3,6 g	3,7 g	4,0 g	2,9 g
Obol	0,7 g	*ca.* 0,55 g	0,8 g	0,9 g	0,4 g	1,1 g

Table 35: Average and median values for the weight of the different denominations.

The link with the original Attic standard is possible, but impossible to evaluate completely due to the effect of corrosion and debasement over time.

[68] De Wilde, 1994: 234.

7.4. Microstructural & chemical composition – SEM-EDX results

7.4.1. Introduction

In order to have a representative surface the coins were put in a holder and a small surface of the rim was polished till clean, corrosion-free metal was seen.[69] This surface was accepted as representative for the originally metal used to mint the coins. The fact that low values of chlorine, oxygen and other corrosion elements were measured, is used as circumstantial evidence to support this. The elements retained for the analyses of the coins are silver, copper, tin and lead. Others elements were looked for but not detected (gold, zinc, etc.). Gold was detected in too low levels to be correctly interpreted, but the ICP-MS analyses showed that it was present in trace elemental levels. Tin was only present in a limited number of coins and in fairly low weight percentages. The lead values were all quite uniform with an average of 1,6 wt% (minimum of 0,75 and a maximum of 2,66 wt%). These values are too low to speak of intentional alloying and the presence of lead can originate from the use of scrape metal or be introduced as impurities present in the copper or silver used[70]. For the bulk analyses essentially only the silver and copper were evaluated, with the exception of one typological group that seems to be characterized by the presence of some tin.

Only two previous studies have reported on the chemical composition of SE-Arabian coins. The first study was by R. Boucharlat and M. Drieux and seven coins from Mleiha were analysed by means of XRF (x-ray fluorescence spectrometry)[71]. XRF is a technique rather analogue to EDX since it is also based on the detection of the X-rays emitted by the different chemical elements present. It is also a non-destructive method and under vacuum conditions all elements with atomic number above 12 can be analysed. The minimum detection limit of the individual chemical elements however is much lower than with EDX, so also elements present at trace levels can be measured. The biggest drawback is, like EDX, the low penetrability of the analysis so only the surface is analysed[72]. The analyses on the Mleiha coins were done at the *Laboratoire de Electricité de France* – St-Denis after the corrosion products had been removed. The conclusions were rather general and no actual data were published. They attested three different alloys: one obol consisted of silver, with lead as impurity and a small amount of copper (alloying element or also impurity), two more coins were made of tin-bronze, and the remaining four coins were of a similar silver-copper alloy. The spectra obtained however did not permit a clear determination of which metal was dominant, the peaks of each being similar in size.

The second contribution was by P. Grave, R. Bird and D.T. Potts and analysed eight coins[73]. These analyses were done by PIXE/PIGME (proton induced x-ray/gamma-ray emission). With PIXE no vacuum is needed and the samples do not need to be conductive. This technique has much lower detection limits than EDX. They distinguished three compositional types of silver coinage in their dataset: a relatively silver-rich group, silver coins debased with lead and silver coins showing elevated levels of iron and nickel. Although the dataset of eight coins was too small to make definitive conclusions, statistical clustering (based on PCA) did appear and the potential to distinguish between groups was acknowledged.[74]

As far as I know these were the only coins from the SE-Arabian region ever subjected to compositional analyses, therefore I think that the dataset of 112 coins presented here is a valuable addition on the numismatic field of SE-Arabia. To sum up: the collection analysed here are all the coins excavated by the Belgian team, minus seven obols on display in the museum, supplemented with three coins found by the British team. The compositional data

[69] A similar approach was used by King & Northover, 1993, to study a large collection of 3rd c AD Roman coins.
[70] Deraisme, Beck, Pilon & Barrandon, 2006: 471.
[71] Boucharlat & Drieux, 1991: 114-115.
[72] Ortiz, 2003: 14.
[73] Grave, Bird & Potts, 1996.
[74] Grave, Bird & Potts, 1996: 75.

of the coins will be presented in relation to their weight and their typology. The arguments of internal chronology combined with the iconographic decline is used here as a hypothetical chronological frame.

7.4.2. Compositional data versus *typology and weight*

To give an impression of the complete assemblage analysed here an overview of the silver content plotted against the copper value is given in Fig. 90. Copper and silver are the main components in all coins. In this plot the lead and tin are not taken into account, but the dots that fall somewhat out of the linear projection have tin and/or lead present in the alloy. Two things can already be noted. Firstly, most obols are situated in the higher silver concentration region (blue square), and secondly most tetradrachms group in the lower silver concentration region (yellow square), but there is a number of tetradrachms on the other end of the scale as well.

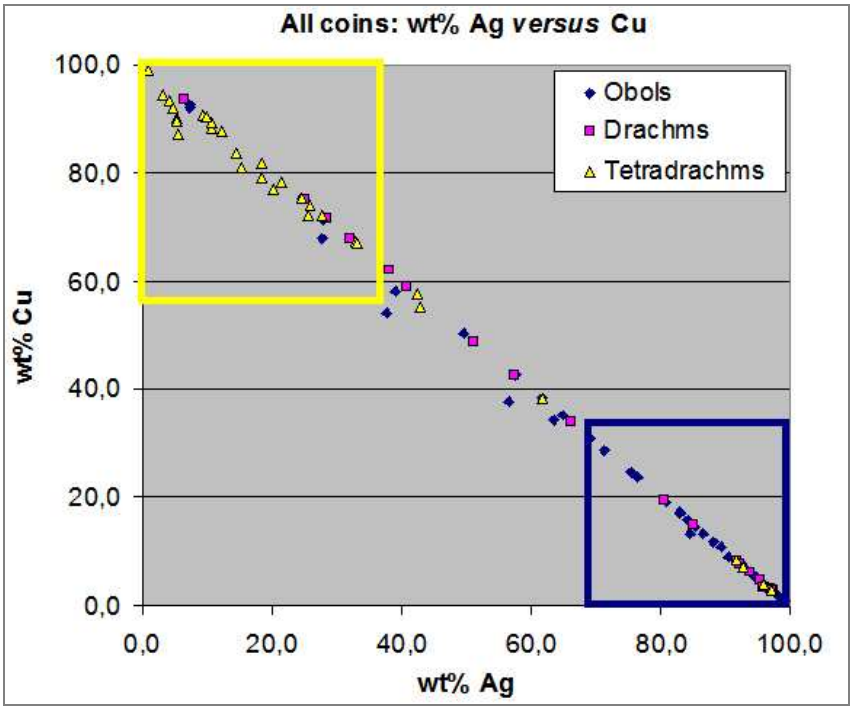


Fig. 90: Wt% copper *versus* silver of all the analysed SE-Arabian coins.

The different denomination will be look at separately and the silver/copper ratio will be evaluated for the different classes. The silver levels are used because silver was the most precious metal present in the coins and for that reason might say something on the intrinsic value of the coins. The box-plots on Fig. 92, 94 and 96 show the range in silver content (in wt%) per typological class and combine the range of the weight (in grams) with the different classes. The black line in the middle of the box is the *mean* value. The box shows 25% of the data above and below the mean and represent 50% of all data. The T-shaped lines above and below the box are the remaining 50% of the data, bordered by the minimum and maximum silver level. Dots outside the plot are statistic outliers as defined by statistical program (SPSS 11.5). The numbers on the plot indicate the number of coins included in the analyses. A picture of a representative coin of each class is included to add the iconographic dimension. The full dataset is given in *Appendix 11.*

• **Obols**

The smallest denomination is the obol. Numerically they are the group that is best represented at ed-Dur (with 54 coins, but 10 could not be classified) and they could be allocated to six different classes. Within class XLVII two extra subtypes were distinguished (XLVII-1 & XLVII-2) based on the 'cleanness' of the depiction (see above).

Classes	Average weight	Median weight
XIVc	0,89 g	0,90 g
II	0,73 g	0,90 g
XLIV	0,95 g	0,95 g
XLVI	0,92 g	0,90 g
XLVII-1	0,81 g	0,80 g
XLVII-2	0,79 g	0,80 g
XLI	0,67 g	0,60 g

Table 36: Average and median weight per obol class.

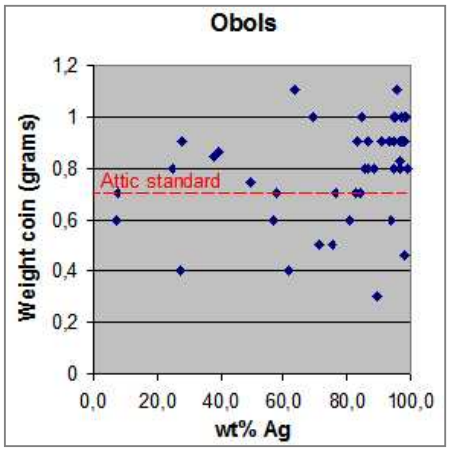


Fig 91: Weight *versus* silver content (incl. Attic standard).

In Table 36 the classes are placed in the hypothetic sequence based on the decline of the iconography. The general trend is that the median weight tends to drop and a break is seen after class XLVI. As mentioned before most obols group in the higher silver region and their weight is well above the original Attic standard (Fig. 91). Although there is no real correlation, silver-poorer coins tend to be lighter.

All obols contain at least some copper even those with very high silver content. Three reasons can be suggested for this[75]:

- The addition of some copper makes a harder and more wear resistant alloy.
- It also lowers the melting point of the silver and makes it easier to produce cast blanks.
- The presence of small amounts of copper can be due to the effects of metal recycling and/or incomplete metal separation during smelting.

Next to the main elements some coins have tin and/or elevated lead levels (Table 37). All obols that have tin added have relatively low silver content and higher lead values. Most of these belong to class XLVII-2, a class with deteriorated iconography. BS 262, M 068 and M 074 are three very similar coins and all three have a lead value higher than the average present in all obols.

Reg. Nr.	Type	Cu	Ag	Sn	Pb
BS 275	XLIV	13,26	84,56	-	*2,18*
BS 262	XLVII-2	34,17	63,66	-	*2,17*
M 068	XLVII-2	53,97	37,74	*6,22*	*2,08*
BS 260	?	68,05	27,74	*2,14*	*2,07*
M 074	XLVII-2	37,57	56,69	*3,70*	*2,04*
BQ 139	XLVII-2	58,02	39,09	*2,89*	1,77
BS 234	?	92,04	7,31	*0,64*	1,91

Table 37: Obols with tin and/or relatively high lead levels.

[75] Scott, 1991: 21; Zwicker, Oddy & La Niece, 1993: 224.

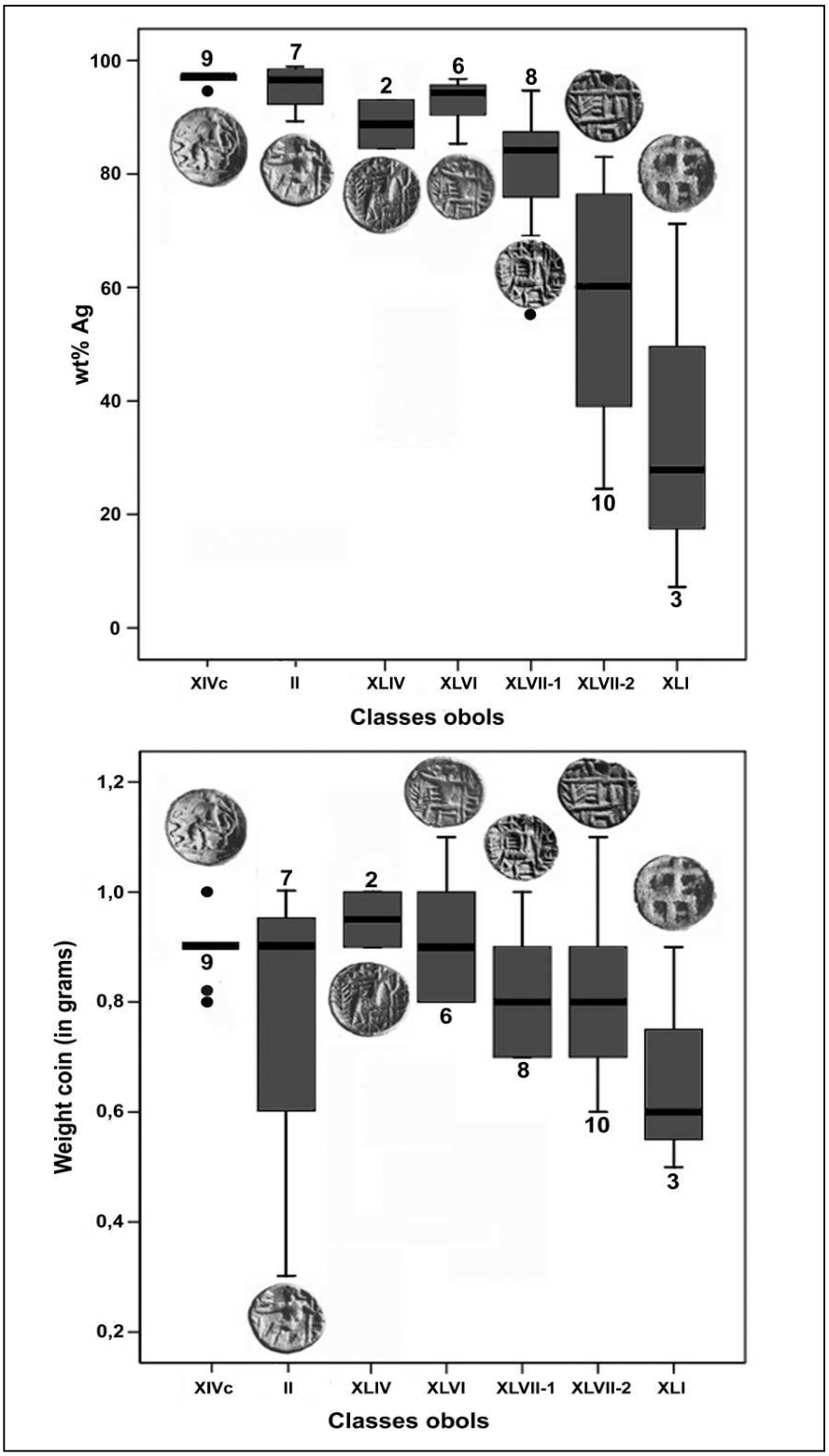


Fig. 92. Obols. Box plot showing the range in silver content (in wt%) per typological class.
Obols. Box plot showing the range in weight (in grams) per typological class.

Other observations to be noted in the dataset are that the iconographically similar coins BS 107, BS 068 and BO 044, from class II, have comparable silver content (*ca*. 95 wt%). The same goes for two coins from class XLVI (ED 001 & BS 102). Some very similar coins, maybe even struck with the same die, also have very parallel compositions. All this points to intentional use of specific alloys for specific groups, this is not an exclusive relation, but the trend is obvious.

If we accept the tentative chronology in Fig. 92 there is a trend among the obols that with dropping silver content, the range of the silver content increases and the iconography appears more stylised. So the less carefully made coins generally contain less silver and show a greater variation in their silver content. To a certain extent this is also the case for the weight. More debased coins tend to be lighter. Class XIVc is a coherent group both in composition and weight. Class II has little variation in the silver content, but has a wide range in weight. These two classes do not have the trident monogram and can be found in SE-Arabia as well as in NE-Arabia, their rather similar high silver values are typical. The first two types that show the trident monogram (XLIV & XLVI) still have a high silver content and a rather realistic depiction. After class XLVI the median silver level starts to drop. The division made here between XLVII-1 and XLVII-2 is supported by the differences in silver content. It is less clear for the weight, but XLVII-2 has a larger range and is thus less standardised. The trend is continued in the iconographically deteriorated class XLI, where the median silver level and weight are lower than in the other classes and the range is large. It should be noted, however that this class is only represented by three coins.

- **Drachms**

The second denomination is the drachm and only 19 were present in the assemblage, of which one could not be classified. In the overall Arabian numismatic collection (as summarised in Table 38 above) the drachms are also the smallest group. They could be classified in four different classes.

Classes	Average weight	Median weight
XLIV	3,41 g	3,42 g
S5	3,70 g	3,70 g
XLVI	3,80 g	3,93 g
XLVII	3,54 g	3,61 g

Table 38: Average and median weight per drachm classes.

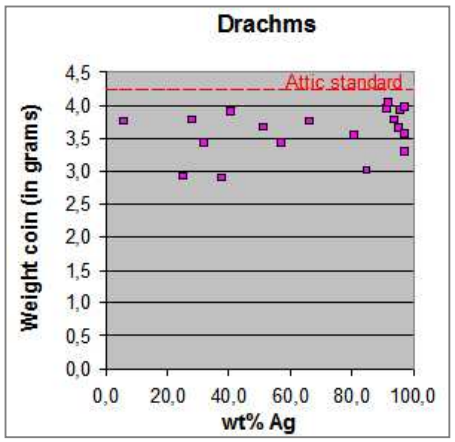


Fig 93: Weight *versus* silver content (incl. Attic standard).

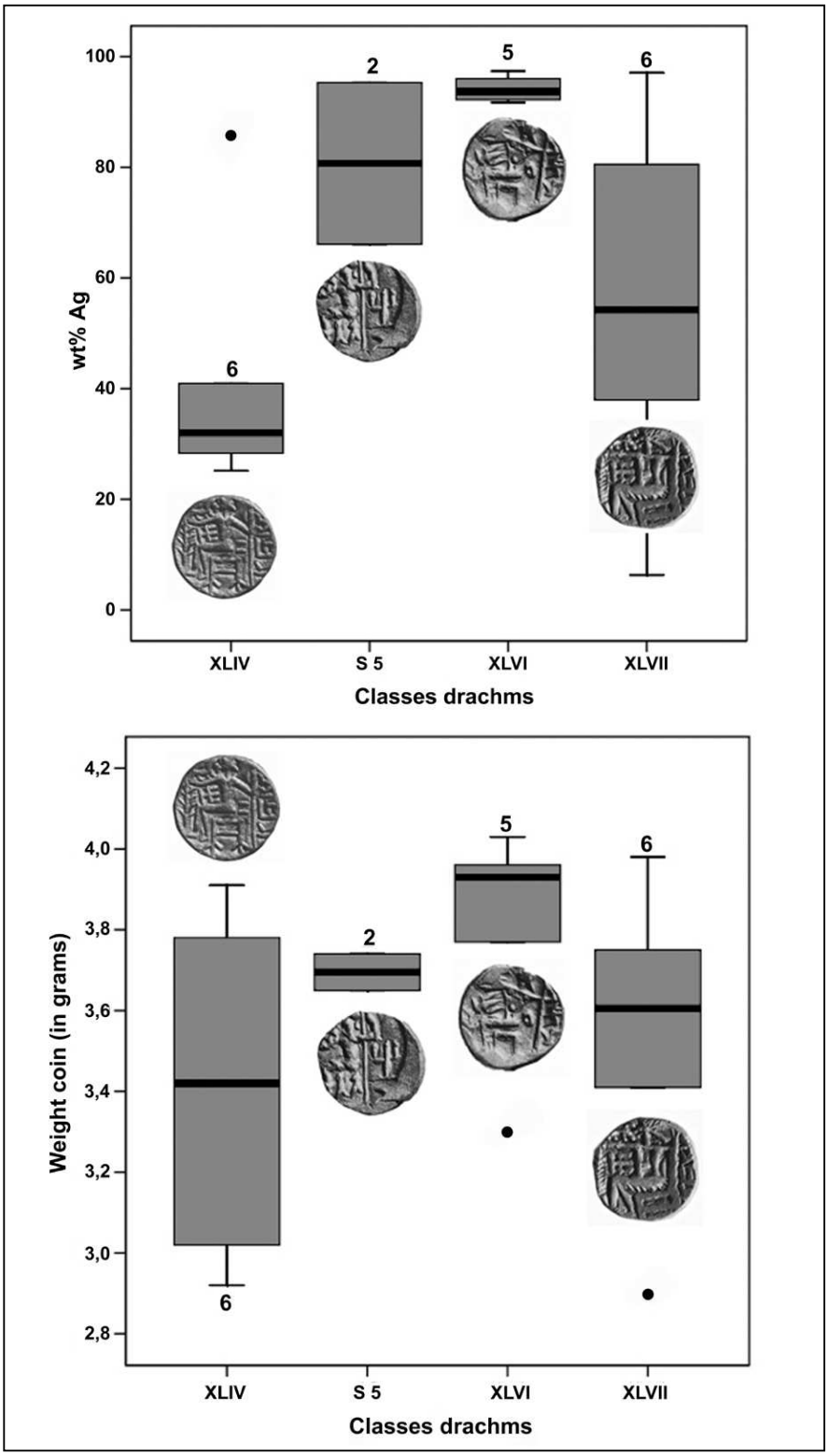


Fig. 94. Drachms. Box plot showing the range in silver content (in wt%) per typological class.
Drachms. Box plot showing the range in weight (in grams) per typological class.

In table 39 the classes are again placed in the hypothetic order based on the deterioration of the iconography. A trend as seen with the obols (dropping median weight) is not found with the drachms. Rather the opposite actually, where the iconographical less deteriorated coins are the lighter ones. The weight of all coins is under the original Attic standard. There is a large variation in the amount of silver in the alloys used to coin the drachms, but still there is a defined group in the high silver region. The iconographic decline does not seem to be linked to the silver content.

Reg. Nr.	Type	Cu	Ag	Pb
BS 286	?	2,90	94,71	2,12
BR 100	XLVII	41,54	56,37	1,90
BO 062	XLVII	60,51	37,53	1,89
M 078	XLVI	7,54	90,48	1,85

Table 39: Drachms with relatively high lead levels.

None of the drachms have any tin present and only four have significant lead values. In the case of BS 286 and M 078 the elevated lead levels might be related to the high silver fraction.

The general trend in the box plot (Fig. 94) of the silver content is mimicked in that of the weight (Fig. 94), so there might be a relation between both parameters. The drachms with higher silver values are also the heavier coins. But the iconographic debasement does not seem to be linked to the silver content. It is surprising to see that class XLIV, with realistic iconography, turned out to have the lowest silver values and the largest weight range. Based on the visual examination of the coins oxidation cannot solely account for this. In class XLVI three almost identical coins (BO 040, BQ 143 & M 078), most probably struck with the same die, also have a comparable high silver fraction. The only tetradrachm of this class (BS 096) also has a very high silver content. The variation of silver within class XLVII is very high, what does fit the stylised iconography.

- **Tetradrachms**

The largest denomination are the tetradrachms and 31 are present in this collection of which one, although very well preserved and with high silver content, could not be classified. Within the overall SE-Arabian numismatic assemblage, the tetradrachm is the most numerous denomination. Seven classes could be distinguished. The one coin placed in class LI, is an uncertain attribution and will not be evaluated any further. Coin BS 096 is the only one that belongs to class XLVI and for that reason not taken up in the box plots below, so five classes are represented on Fig. 96.

Classes	Average weight	Median weight
LI?	16,07 g	16,07 g
XLVIIIa	15,28 g	15,17 g
XLVIIIb	14,48 g	14,60 g
XLV	13,21 g	13,15 g
XLVI	15,78 g	15,78 g
XLIX	15,05 g	15,05 g
XLVII	15,24 g	15,20 g

Table 40: Average and median weight per tetradrachm classes.

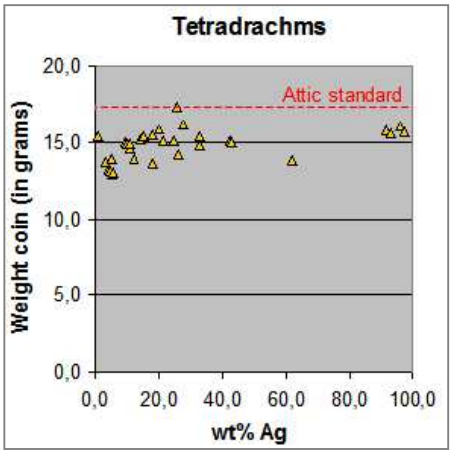


Fig 95: Weight *versus* silver content (incl. Attic standard).

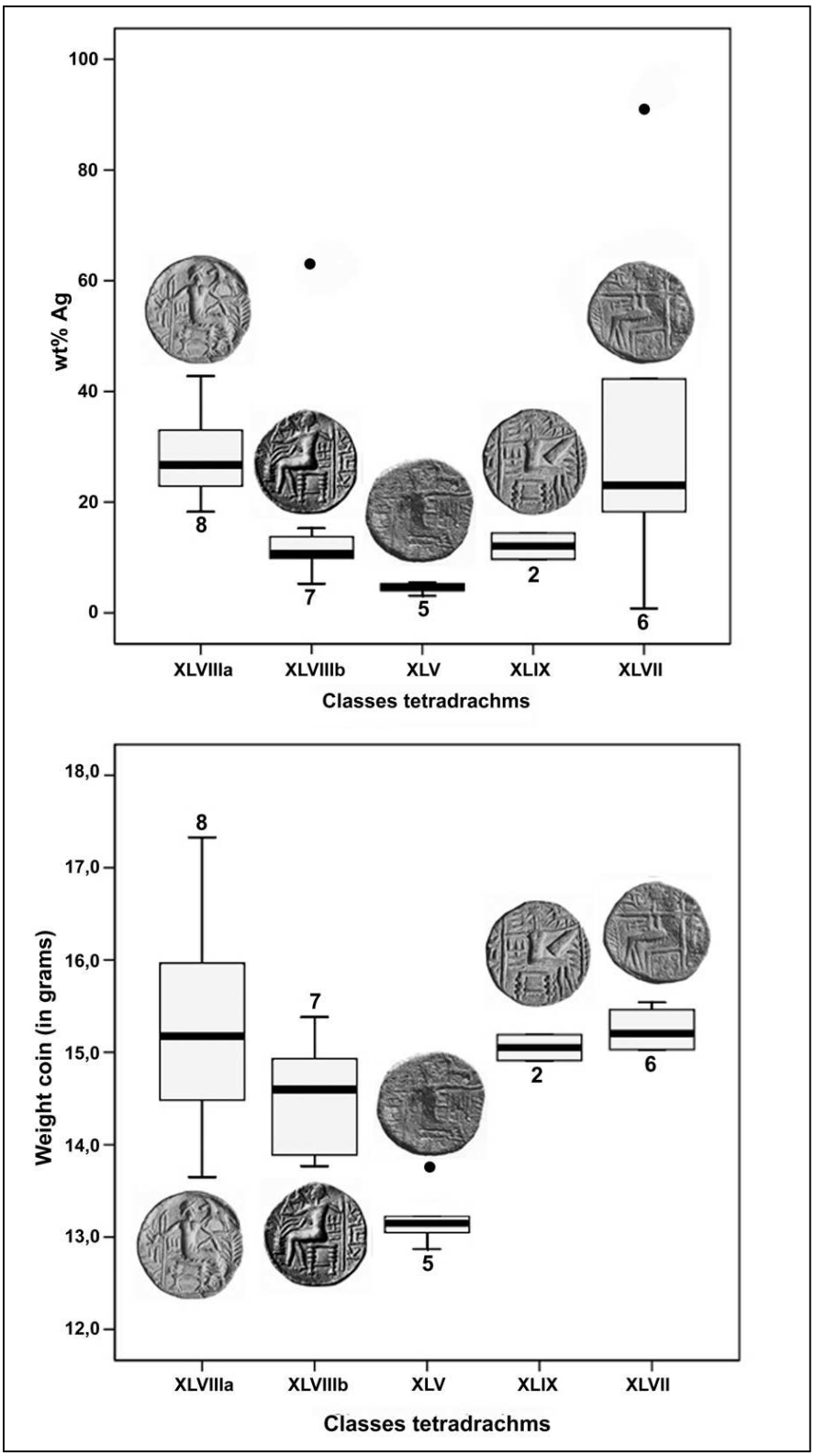


Fig. 96. Tetradrachms. Box plot showing the range in silver content (in wt%) per typological class.
Tetradrachms. Box plot showing the range in weight (in grams) per typological class.

In table 41 the classes are ordered in the hypothetic sequence based on the deterioration of the iconography. A clear trend as seen with the obols (dropping median weight) is not found. There is a break after type XLV, after which the average and median weight rise again. Strange enough this is also the break between the realistic and stylised coins (see below). There is a large variation in the amount of silver in the alloys used to mint the tetradrachms, but a small group can be distinguished with a high silver content and these coins are also among the heavier ones. This group is however not related to a specific class but surpasses the typology and show great iconographic variety. Except for one example, all coins are lighter than the original Attic standard.

Reg. Nr.	Type	Cu	Ag	Sn	Pb
BQ 137	XLV	89,61	5,21	5,18	1,59
BM 026	XLV	86,90	5,47	4,95	***2,65***
BS 285	XLV	91,98	4,61	3,41	1,62
BS 254	XLV	93,21	4,06	2,48	1,88
BS 284	XLV	94,52	3,12	2,36	1,82
BS 237	XLVIIIb	90,07	5,26	2,52	***2,14***
BS 235	XLVIIIb	80,78	15,28	3,94	1,65
BR 106	XLVIIIb	88,35	10,55	1,10	0,75
AD 025	XLVIII	76,76	20,17	3,07	1,63
AV 023	XLVIIIa	79,05	18,24	0,49	***2,22***
BS 172	XLVIIIa	72,12	25,65	-	***2,23***
BS 071	XLVIIIa	55,13	42,77	-	***2,10***
BS 080	XLIX	83,58	14,40	-	***2,03***
BS 097	XLIX	84,94	12,97		1,91

Table 41: Tetradrachms with tin and/or relatively high lead levels.

The number of coins containing tin is higher among the tetradrachms than among the other denominations and is associated with the coins that have lower silver levels. Class XLV stands out because all coins contain a certain amount of tin (3-5 wt%) and the silver levels (4-5 wt%) are the lowest of all tetradrachms. The relation between this Cu-Ag-Sn-alloy and this class is exclusive and two tetradrachms of type XLV analysed and published by P. Grave, R. Bird and D.T. Potts[76], also had significantly higher tin levels. It is interesting to notice that the addition of tin appears to be fairly effective in increasing the whiteness of a copper-silver alloy. This method was for example used during the reign of the Roman emperor Gallienus (253-268 AD) to give the debased coins a more silvery look. There are narrow practical limits to the amount of tin which can be added to silver-copper alloy without causing serious embrittlement[77]. It is thus striking that among the analysed coins only few have detectable levels of tin, and all these coins have relatively low levels of silver. This could be linked to the whitening effect of tin as mentioned above. The effect of adding tin was evaluated by casting some test coins (see below). Alloys with above 5% of tin are hard enough to shorten the working life of coining dies. A small amount of tin also lowers the casting temperature considerably, making the casting of blanks much easier[78].

Other facts to be noted are:

- The two coins in class XLIX (BS 080 & BS 097) have a very similar iconography and also the alloy composition is very parallel.
- Within class XLVII, BJ 008 can be seen as somewhat of an outlier (contains more silver), the only thing that is really different from the others coins within this class is the presence of the *calliper* monogram.

[76] Grave, Bird & Potts, 1996: 75.
[77] Cope, 1972a: 271; but now actual limit is mentioned.
[78] D. Tokar, pers. comm.

- Within class XLVIIIb, M 064 contains considerable more silver than the other coins, it also has the best depiction (clear, detailed…) and is very close in style to AO 018 and AC 012 (compositions do not correspond however).

Class XLVIIIa and XLVIIIb are iconographically closely related, but in composition there is a clear separation. Three coins within class XLVIIIb (BR 106, BS 235 & BS 237) also have some tin present. They differ from the other coins in that class in the fact that the seated figure on the reverse side is facing to the right, something not seen among the other issues of this class. The depiction is not well preserved, a feature also seen among the coins of class XLV. Class XLV is a very well defined group, compositionally (see above), but also on the level of their weight and iconography. Among these three classes the iconographic debasement is linked to a dropping silver content and to a certain extent weight, although class XLVIIIa has a large range. The two coins in class XLIX are very similar as mentioned before and although their iconography is already more stylised, they were made with great care and well-struck. These characteristics can also be seen on coins from this class that were published from other sites. The last and iconographical most deteriorated group, class XLVII, has a relatively high silver content if compared to the other classes, but the range is large. The weight on the other hand shows little variation.

7.4.3. Foreign coins

As mentioned before, these coins will not be discussed any further, but since they were analysed it was thought useful to present the data anyway. The results are summarised in the table below, also the origin and if possible date are mentioned (see Fig. 42 for illustrations).

	Origin	Cu	Ag	Sn	Pb	Extra
BO 056	Mediterranean	78,50	-	6,66	14,84	Ascalon?, 1st – 2nd c AD
BS 070	Mediterranean	83,62	-	10,47	5,91	Letter M, typical for Gaza
M 080	Greek	63,76	-	4,50	31,74	Imperial Greek?
BS 072	Nabataean	94,96	-	2,42	2,62	Aretas IV (*ca*. 8 BC-ca 40 AD)
BS 278	Persis (Fars)	3,25	96,75	-	-	2nd half 1st c BC
ED 011	Persis (Fars)	2,35	97,65	-	-	2nd half 1st c BC
M 081	South Arabia (Hadramawt)	72,69	-	7,01	20,30	Late 2nd – late 4th c AD?
M 082	Indian (Ujjan)	97,81	(0,24)	(0,24)	(1,71)	*ca*. 150-75 BC, local issue from West Malawa in Madya Pradesh

Table 42: Foreign coins analysed[79].

Based on table 42 some things can be pointed out:

- Both coins, BO 056 and BS 070, are true leaded tin-bronzes. Also M 080 is a leaded tin-bronze, although the lead level is much higher. In a certain sense it can be compared to the S-Arabian coin (M 081).
- The Nabataean coin (BS 072) is from leaded copper. The tin content is low and could easily be the result of unintentional addition through recycling of metals.
- The coins from Persis (BS 278 & ED 011) are both of a similar good silver quality, indicative of a strong, centralized coining policy. The copper in the coins of Persis may

[79] Determination by Haerinck, 1998a.

be intentional to harden the alloy, but may as well be the result of remelting or residual copper left after the incomplete refining of the silver.
- The Indian coin is from an unalloyed copper, but the fact that very low levels of silver and tin were detected might point to the fact that this metal is likely to be the result of recycling. The levels are too low however to be truly evaluated and are not really reliable.
- It might be interesting to mentioning that eleven coins were found in area M, where the temple is situated, three of them are foreign (M 080, M 081 & M082).

The foreign coins analysed here differ in several aspects from the SE-Arabian once analysed. The lead values are considerable in some of the tin-bronze coins, a phenomenon not seen in any of the SE-Arabian coins. None of the coins are of an intentionally debased copper-silver alloy.

7.4.4. Surface silver enrichment

As outlined above all coins contain silver and copper as their main compositional elements. Surface enrichment is the term used to describe the higher concentrations of silver with respect to copper at the surface as compared to the interior[80]. Some of the published coins are described as being "coated with a silver layer"[81]. Several methods were known in antiquity to give coins a more silvery appearance without actually using pure silver as the core material. During the 3rd and 4th c AD the Romans used such techniques to make coins that appeared to be from silver, but actually had a much baser core. In this way less of the more expensive silver was needed.

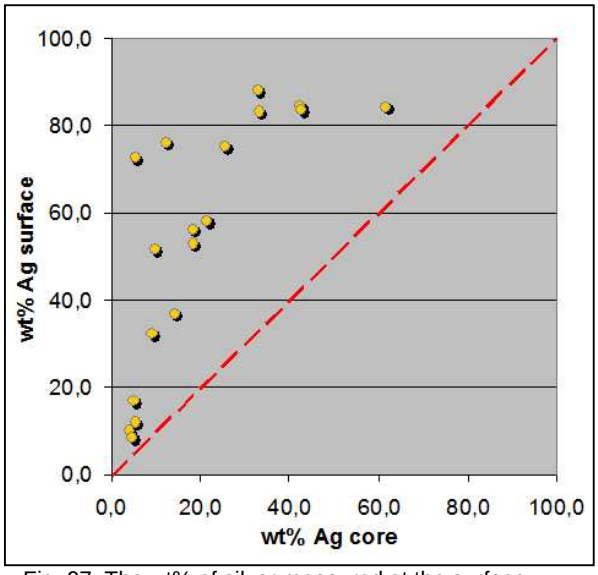


Fig. 97. The wt% of silver measured at the surface *versus* that measured on a polished surface.

Fig. 97 shows the weight percentage of silver measured at the surface before polishing[82] *versus* the values measured on a polished section that is supposed to be representative of the original coining alloy used. Not all are included, because not all were analysed in this way. For the sake of clarity also the 'silver' issues are omitted since the effect of surface enrichment is negligible for these coins. The graph clearly shows that the amount of silver at

[80] Tite, 1972: 338.
[81] E.g. Haerinck, 1998c: 28 and Callot, 2004: 145.
[82] The surface was previously cleaned in lemon juice so the analyses were not done on the oxide layer originally present on the coins.

the surface is sometimes considerably higher than in the core. If the values had been similar then the dots would have plotted on or around the dashed line. The results point to a mechanism that generated a silver enriched layer at the surface. To evaluate if this was an intentional process or the result of post-processional changes, an overview will be given of the possible silvering techniques used in antiquity. A second conclusion from this graph is that one should be careful in using un-cleaned/polished surface to do compositional analyses. In some cases the difference between the values can be considerable.

Several methods were known in antiquity to give an object a silver appearance without actually using pure silver as the base material. In the case of coins it was often thought in the past that this deliberately silvering was the work of counterfeiters, but now it has become clear that for example the official mint in the Roman Empire sometimes used these techniques in periods of crisis. So it is often a problem to evaluate if such coins are to be regarded as genuine official issue, or a false one. This problem is most probably of no relevance to the coins found at ed-Dur and Mleiha. I think that their relative limited number and restricted area of use do not easily allow fake coins to be brought in circulation. Fakes would probably have been spotted immediately. Remains the question of course whether the present observed surface and enrichment was originally intended or if it is the accidental result of selective chemical corrosion and surface-enrichment processes after the coins became buried, or even from the cleaning process used after excavation.

The techniques presented are silver coating or enrichment by: mechanical attachment; soft-, hard- and self-soldering; amalgam or mercury silvering; depletion silvering or pickling; hot-dipping and electrochemical replacement. A last possibility is a natural post-burial process that took place over time.

- **Mechanical attachment**

This very first method was just to attach a thin silver sheet by simple mechanical methods (nails, hammering into keying grooves, etc.) or with non-metallic adhesives (organic glues). This was primarily done on organic base materials and is attested from at least as early as the 3rd millennium BC[83]. This technique however is of no use in coining and will not be discussed any further.

- **Soldering**

The second method is soldering. The two large groups of solders that can be distinguished, soft and hard solders, were discussed above and only the details relevant to coining will be mentioned here.

Soft-soldering

In the Roman period, soft solders were used to attach silver foil, but this technique can be excluded from the possibilities here since it is not (well) suited for coins that have to be struck. Moreover if it was used detectable raised levels of tin and/or lead should have shown up in the EDX-spectra and any layer of solder would also have been seen on the BSE-images of the SEM. Neither was the case, so this possibility can be safely excluded.

Hard-soldering

Plated Roman coins have revealed that for the most part these specimens consist of a copper core, to which a silver sheet has been fixed by a silver-copper solder. The edges are carefully doubled over to prevent exposure of the core and the application of a flux contributed to the efficiency of the soldering action. The thickness of the silver plate was usually quite sufficient to make sure that the core was not seen at the surface due to the deformation when struck. It is important to notice that the silver foil was attached to a blank before it was struck, so the details of the die were not lost. It is not clear whether such

[83] La Niece, 1993a: 201-202.

plated coins are wholly the work of counterfeiters or if the official mints sometimes used the same technique[84]. The use of silver-copper solders for silvering goes back to at least the 5th c BC where it can be seen on early coin forgeries imitating Greek types. The method stayed in use for small, flat items, to improve the permanence of foil silvering and in the Roman Empire the use of silver foil at least continued until the 1st c AD[85].

Although silver eutectic spots were seen on the BSE-images, they were not continuous, and to few to be the remains of a hard solder.

Self-soldering or Sheffield plating

Self-soldering is known from the 18th c AD as Sheffield plating, but the technique could have occurred centuries earlier, conclusive evidence is lacking however[86]. This process is based on the fact that silver can be bonded to copper by simply heating the two metals in close contact with each other with no intermediate joining material or solder, as see above. The limited diffusion between the metals will form a low melting-point alloy at the interface of the two pure metals. The resulting bi-metal can be worked and shaped as if it was solid silver[87].

The problem is that the end product cannot be distinguished from a joining created by a silver-copper solder. So the use of Sheffield-plating cannot be proven in antiquity, but it neither can be ruled out[88]. Analysis showed that the surface layer of experimental coins (copper core covered with silver foil and heated at 800℃ for 15 to 20 min.) contains about 40 % of Cu, rather more than the eutectic composition (*ca*. 28%). The best results were achieved at a temperature 800℃, or even a few degr ees less, and only heating the coin for very short times (3 to 10 min)[89]. A. Deraisme *et al.*[90] conducted a similar experiment, wrapping a copper blank in silver foil, at temperatures of at least 950℃ for a time period of more than 4 minutes. The result was a layer that contained 30% of silver due to self-soldering.

Instead of using foil it is also possible to sprinkle the copper core with powdered silver or silver-copper alloy and then heat them strongly, till the powder melted and runs over the surface. In this way a liquid hard-solder covered the surface[91].

A last general remark on all technique used to add a silver layer to a base metal core is the difficulty to successful cover the rim of the coin[92].

- **Amalgam or mercury silvering**

Amalgam silvering is done in the same way as the historically better attested technique of mercury gilding. This gilding technique was used extensively in the Roman period and there are many references to gilding with amalgam in early texts whereas the use of amalgam silvering is never cited and even in much later texts it is rarely mentioned[93].

The technique is based on the fact that silver and mercury readily react to form an amalgam that can be used for silver-plating of copper and copper-base alloys. Silver leaf was ground in a mortar with an excess of mercury. This paste of silver-mercury amalgam is applied to the clean surface of a copper(-base alloy) object and is heated to 250-350℃ for a few minutes till a diffusion bond is formed between the substrate and the silver. A thin layer of silver is left

[84] Sellwood, 1976: 68.
[85] La Niece, 1993a: 201-202; Zwicker, Oddy & La Niece, 1993: 242.
[86] Campbell, 1933: 144; La Niece, 1993a: 201.
[87] La Niece, 1993a: 204.
[88] La Niece, 1993a: 205.
[89] Zwicker, Oddy & La Niece, 1993: 229.
[90] Deraisme, Beck, Pilon & Barrandon, 2006: 479.
[91] Campbell, 1933: 144.
[92] Zwicker, Oddy & La Niece, 1993: 228.
[93] Vlachou, McDonnell & Janaway, 2002: II9.2.3 - http://www.mrs.org/publications/epubs/proceedings/fall2001/ii/.

after the excess mercury is evaporated. Alternatively the object could be first coated with mercury and when the silver leaf was applied a silver amalgam was formed *in situ*. Afterwards the surface is polished, till it takes a silvery colour. The role of the mercury in this process is mainly to maintain close contact between the silver and the core metal. The discovery that mercury could be used to plate a thin layer of silver onto copper or a copper-base alloy was a major advance in plating technology. The method could be used to plate complex shapes without the joins that go with all the methods of plating with foil. In Europe it is only from the 13th c AD onwards that the technique became widely available in spite of the widespread use of mercury for gilding. In China on the other hand it was already used at least as early as the Han-dynasty (1st c BC – 1st c AD), but examples are rare however.[94]

Recent analyses however by C. Vlachou in the frame of a PhD thesis to evaluate the changes in the Roman numismatic metallurgical technology in the 3rd - 4th c AD, have reported mercury concentrations present in association with the silver layer on the coins. For this research the combined application of LA-ICP-MS and EDX analyses were used to shown for the first time, that mercury was correlated with the silver in the plating area. Much higher concentrations of mercury occurred on the surface than in the silver rich pools in the coins core. This evidence strongly implies that the possible method for the production of the plating was amalgam silvering and that it was already know in Europe in the 3rd – 4th c AD[95]. Apparently C. Vlachou concluded that a few percent of tin and silver in a copper-base alloy are required to make effective mercury silvering[96].

In the EDX-spectra no indication of the presence of mercury could be detected. Because the detection limit of SEM-EDX is rather high and the expected values of mercury very low, one coin was therefore analysed by LA-ICP-MS. This coin (AV 023) was selected because part of the surface had what appeared to be a piece of silver 'foil' preserved (see Fig.113). The bulk of this coin was previously also analysed by ICP-MS and showed that no mercury was present in the core material. This then could be used as an internal standard to compare the results of the surface analyse. The data generated by LA-ICP-MS did confirm that no significant mercury levels were present. Although only one coin was analysed in this way it is a usable observation. It should not be forgotten that mercury silvering is not expected. The technique was only attested on 3rd – 4th c AD Roman coins, which is considerable later then the ed-Dur coins. Still it was thought useful to exclude this by an analytical spot check[97]. What the origin of this 'piece of silver foil' might be is not completely clear, especially why it is only preserved on part of the coin (corrosion effect combined with the initial cleaning?). The 'foil' has a Cu/Ag-ratio of *ca*. 20/80, whereas the core is *ca*. 80/20. According to Beck *et al*. a cast blank of this composition should generate an eutectic layer (28/72) at the surface[98]. The somewhat higher amount of silver measured would be in accordance to such a blank additionally treated by a pickling process.

- **Depletion silvering or pickling**

A completely different technique is depletion silvering or pickling. This is not strictly a plating method, although the end result is also a silver-rich surface on a baser copper-base alloy core. To make this technique work the base copper alloy must contain a certain percentage of silver from the start. Basically the method involves the removal of copper from the surface. This is done by chemically removing copper(-oxides) so the silver-rich components of the alloy will be left at the surface. After polishing it can obtain a bright silvery colour. This technique could be exploited for larger scale production than would be possible with many of the other silvering techniques. The clearest example of the technique outside the New World

[94] La Niece, 1993a: 201 & 207; Anheuser, 1997b: 127-128.
[95] Vlachou, McDonnell & Janaway, 2002: II9.2.1 - http://www.mrs.org/publications/epubs/proceedings/fall2001/ii/; Vlachou, C. from abstract posted on the Arch-Metals website (29/09/2005).
[96] M.J. Ponting, pers. comm.
[97] In retrospect this was not the best coin to test this on, but unfortunately due to time restrain it was impossible to repeat the test on one of the coins with low silver content and some tin added.
[98] Beck, Bosonnet, Reveillon, Eliot & Pilon, 2004: 156.

is in the Roman coinage dating to between 63 AD till *ca*. 260 AD. Blanks containing as little as 12% of silver in the alloy can be successfully treated to produce a bright silvery surface, as long as the microstructural continuity of the silver-phase does not break down.[99]

One simple way to remove the copper-oxides chemically is by boiling or quenching the hot surface-oxidized blank in a warm citrus fruit acids or vinegar. This was done before the coin was struck, with the result that the silver-rich surface was consolidated during the striking and formed an almost continuous skin of silver[100]. We should however be aware of the fact that natural corrosion processes that take place during burial and/or post excavation cleaning can also result in a silver-rich surface. Analytically this is indistinguishable from deliberate surface enrichment during minting, but a metallographic section should reveal whether the oxidation and leaching of the copper-rich phases occurred before or after striking. If before, the silver-rich lenses will be compacted together to form a denser concentration at the surface, but if the leaching occurred after minting, the silver-rich lenses will be evenly distributed in a more porous matrix of corrosion products.[101]

An alternative method where a pickling bath is also used was suggested by L.H. Cope[102], and has more recently been evaluated by K. Anheuser and P. France[103]. L.H. Cope suggested that the thin coatings of the later Roman coinage could be achieved by the segregation of lead from leaded copper-silver alloys. The technique is related to the cupellation process for the purification of silver. He assumed that by cold hammering and annealing of a leaded argentiferous copper blank, molten lead together with the silver would have migrated to the surface where the lead was oxidized and removed in a pickling bath. If this were true the coins should contain enough silver and lead and the metallographic examination should show lead enrichment near the surface.[104] This is not the case with the coins looked at here.

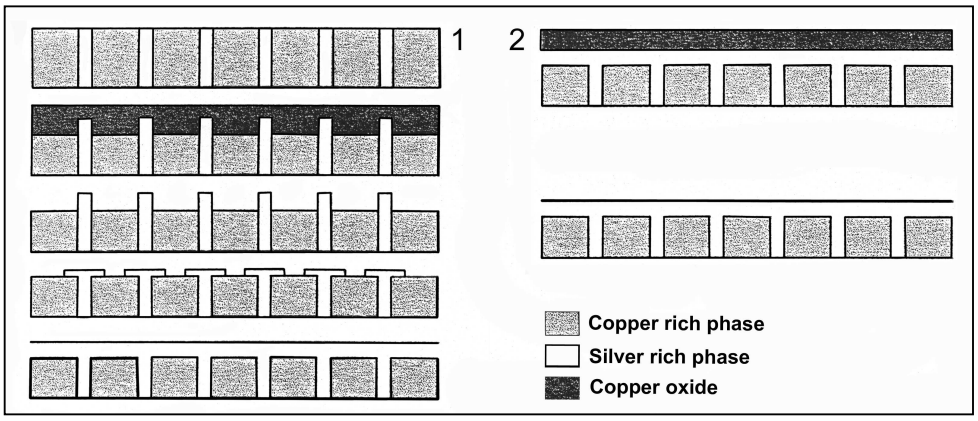


Fig. 98: Schematic representation of pickling process (after Beck *et al.*, 2003: 565).

The silver-copper system is an eutectic system with low solid solubility of silver in copper and *vice versa* and upon cooling the silver-copper alloys undergo phase separation. At the surface the arrangement of these phases follows different behaviours according to the initial alloy composition. Recent experimental work by L. Beck, S. Reveillon, S. Bosonnet, D. Eliot and F. Pilon with silver-copper alloy blanks has clarified these processes[105]. Basically three alloy groups can be distinguished.

[99] Cope, 1972a: 267-269; Tylecote, 1987: 287; La Niece, 1993b: 227; Zwicker, Oddy & La Niece, 1993: 240.
[100] Cope, 1972a: 267-269; La Niece, 1993a: 206.
[101] La Niece, 1998: 120.
[102] Cope, 1972a: 275.
[103] Anheuser & France, 2002.
[104] Anheuser & France, 2002: 19-20.
[105] Beck, Reveillon, Bosonnet, Eliot & Pilon, 2003; the same information was published in Beck, Bosonnet, Reveillon, Eliot & Pilon, 2004.

- The first group are silver-rich hypereutectic alloys with more than 72% of silver and the presence of the silver-rich phase at the surface of the sample is due to the normal segregation of the components. The higher melting point metal (i.e. silver *versus* the eutectic silver-copper alloy infill) is concentrated at the outside of the button during solidification. The connection between the silver-rich grains leads to the formation of a quasi-continuous layer containing about 92% of silver. This layer can be 20 to 80 µm thick. This phenomenon already occurs when the blanks are cast.[106]

- The second group are hypoeutectic silver-poor alloys with a silver content between 72 and 15%. It was thought that the natural process of segregation could not give an explanation to the surface enrichment. Upon casting an inverse segregation was observed by L. Beck *et al.* for these alloys, with the formation of a eutectic layer at the surface[107]. The blank will have a black copper-oxide layer at its surface. This layer will also extend beneath the surface, the depth being linked to the length of time the blank is kept at red heat[108]. L.H. Cope already mentioned a process of surface silvering caused by oxidation phenomena. He suggested that the oxidation occurred during fabrication of coins and particularly during the preparation of the blanks or flans. The sequence he described was that a copper-silver alloy blank was cast and then heated in the air to form a copper-oxide layer. Acid-pickling was then used to remove these oxides and to reveal the silver-rich phase beneath. By striking the coin this silver-phase was spread sideways and consolidated (Fig. 98-1 shows the successive steps). The recent experiments by L. Beck *et al.* have shown however that the silver enrichment can already be achieved at the first step (during the casting) when an oxide layer is already formed and no additional oxidation process has to be performed. Only pickling is needed to remove the copper-oxides (schematic representation Fig. 98-2). Furthermore, this process directly induces the formation of a continuous eutectic layer (~ 72% of silver) as a continuous and regular layer of 10 to 40 µm thick, although the atmosphere is important in regulating this process. Multiple hot-working operations can however still enhance this phenomenon.[109]

- The third group are silver-poor alloys with less than 15% of silver present. The silver proportion seems to be too low for forming a spontaneous eutectic layer. In this case the model proposed by L.H. Cope, i.e. an additional oxidation phase before pickling, is suitable. R.F. Tylecote suggests the lower limit of this system might be somewhere around 12% of silver[110]. Essentially a blank can be treated until the interdendritic network of the copper-silver eutectic breaks down and is not interconnected anymore[111].

• Dipping and electrochemical replacement

In the past it has been suggested that coins were plated by dipping a copper core into a bath of *liquid eutectic silver-copper* alloy or rather one richer in silver[112]. Several practical objections can be used to reject this hypothesis:

- The difficulty of keeping the temperature of the bath high enough to keep the silver molten when the item is dipped, but low enough to prevent the copper core from melting.
- There is also little economic sense in having a bath of silver that will become progressively more contaminated by the copper dissolving from items dipped into it[113]. This will eventually result in a bath where the copper content is to high to be used to coat, but where the silver content is still considerable. Although it is possible to extract the silver from such an alloy, it is time and energy consuming.

[106] Beck, Reveillon, Bosonnet, Eliot & Pilon, 2003: 562-563.
[107] Beck, Bosonnet, Reveillon, Eliot & Pilon, 2004: 156.
[108] Butcher & Ponting, 2005: 173.
[109] Cope, 1972a: 265-267; Beck, Reveillon, Bosonnet, Eliot & Pilon, 2003: 563-564.
[110] Tylecote, 1987: 287.
[111] P. Northover, Pers. comm.
[112] Campbell, 1933: 144; Tylecote, 1987: 287.
[113] Tylecote, 1987: 287; La Niece, 1993a: 205.

- Also the thickness of the silver layer and the weight of the coin are difficult to control[114].
- This is in addition to the practical difficulties associated with hot-dipping in mass production[115].

L.H. Cope[116] has suggested that copper blanks could be dipped into a bath of *molten silver chloride* (melting point 455°C and a by-product of the cupel lation of gold[117]) instead of silver or a silver-copper alloy. Silver chloride was available in antiquity as hornsilver (keragyrite), but there is no reference to this method in ancient literature[118]. This should have led to the deposition of a thin silver layer on the copper by electrochemical replacement. Experimental work by K. Anheuser and P. France has shown that if a copper sheet is dipped into molten silver chloride, silver immediately precipitates as a thick, dark and porous deposit, not as a thin sheet. Alternatively *heating* pieces of copper together with *solid silver chloride* in a crucible to a temperature above the melting point of the silver chloride followed by removing the copper from the liquid salt produced equally poor results, with thick and porous silver deposits. Such a process would also have been inefficient in its use of silver[119].

K. Anheuser and P. France suggested a fourth possibility in which the silvering is done by bringing clean copper blanks into contact with silver salts or their aqueous solution, *without heating* them. Although it is usually assumed that silver salts only became available with the post-medieval introduction of nitric acid, experiments have demonstrated that a silvering paste could equally well be obtained from silver and commonly available corrosive salts (sodium chloride, potassium nitrate and alum). According to K. Anheuser and P. France a silver chloride past would be the most likely agent for Roman electrochemical replacement plating. The metallographic investigation of the thin silver-plating on mass-produced Roman 3rd c AD coins showed that the silvering was carried out cold with a paste or liquid which penetrated into the cracks and holes. Their replication experiments showed that macro- and microscopically identical plating could be produced by electrochemical replacement silvering with a silver chloride paste. This type of operation would have takes place after striking and was used in Andean cultures.[120]

There are no positive elements to reject this technique, only a circumstantial one. Why would one use a copper-silver alloy blank, if the same result can be obtained by using a copper core instead? Where is the gain in saving silver? Admitting this is a modern point of view that may not have played in the past, I still think it is valid.

• **Natural processes**

The question remains however to what extent the results of analysis today are representative of the original composition of the alloy used. Several natural processes and/or post-excavation cleaning can also result in a silver-rich surface. Because copper is a less noble element than silver, it is the component that is most subjected to corrosion in a copper-silver alloy. Because of this there could be a variable loss of the copper due to oxidation, thus leaving an increased average percentage of silver at the surface[121]. This process however has a negligible effect on the average composition of the blank and if core material is analysed good results are still produced. In the case of the coins analysed here, the surface corrosion was removed, but care had to be taken not to damage the coins too much. There is a slight possibility that the analysed surfaces still had part of the enriched/depleted layer present and the results are influenced in favour of the silver fraction.

[114] Anheuser & Northover, 1995: 28.
[115] Anheuser & France, 2002: 20.
[116] Cope, 1972a: 275.
[117] Sellwood, 1976: 67.
[118] Vlachou, McDonnell & Janaway, 2002: II9.2.3 - http://www.mrs.org/publications/epubs/proceedings/fall2001/ii/.
[119] Anheuser & France, 2002: 20-21.
[120] Anheuser & France, 2002: 19-22; Vlachou, McDonnell & Janaway, 2002: II9.2.3 - http://www.mrs.org/publications/epubs/proceedings/fall2001/ii/.
[121] Condamin & Picon, 1964: 98.

The fact that the coins were soaked in lemon juice after excavation to partly remove the corrosion layer is important to notice, especially in the frame of a pickling process. This could result in a similar appearance as when performed prior to striking. If the coins were not treated at all when they were made and a corrosion layer was formed after they became buried, this would give a copper-oxide layer at the surface, leaving a silver-enriched layer beneath. If then after excavation this copper-oxide were removed by lemon juice, this would give a similar effect as when the coins were pickled before burial. Compositionally this is indistinguishable from deliberate surface enrichment. Microscopically there should however be a difference in the consolidation of a silver enriched surface generated by pickling and striking afterwards, and that resulting from post-burial process. The silver-rich lenses generated by natural processes should be evenly dispersed in a more 'porous' matrix of corrosion produces. Whereas when the lenses underwent the action of striking they would be compacted together to form a denser concentration at the surface. In practice this has however proven not to be as clear as thought. This is mainly due to the fact that both processes generate a relative thin layer of silver and that pickled surfaces are not immune for additional post-burial transformations.

- **Observations on the studied coins**

Of the possibly techniques used to create a more silver-rich layer most (soft- and hard soldering, amalgam silvering, dipping and electrochemical replacement) can be ruled out based on the analytical results obtained and circumstantial evidence. One technique is left standing: pickling or depletion silvering.

To evaluate all coins they are divided in 3 categories according to L. Beck, *et al.*[122], being coins with more than 72% of silver present in their alloy, coins with between 72 and 15% of silver and coins with less than 15% of silver. To the first group belong 4 (of the 31) tetradrachms, 10 (of the 19) drachms and 39 (of the 54) obols. These alloys generate a silver-rich phase at the surface due to the normal segregation of the components, since the higher melting point metal (i.e. silver) is concentrated at the outside of the button and the inside exists out of silver dendrites filled in with the silver-copper eutectic. These will not be considered any further since they automatically have a silver-rich surface, without extra interventions. To the second group (72 to 15 wt% Ag) belong 15 tetradrachms, 8 drachms and 13 obols. These coins qualify for a pickling process and under the right circumstances the extra oxidation phase can be omitted. To the last group (less than 15 wt% Ag) belong 12 tetradrachms, 1 drachm and 2 obols. Here the silver proportion seems to be too low and an additional oxidation phase before pickling is needed to obtain an enriched surface. Within the coins of this group no visual indications was found that more silver was present at the surface. There are 2 obols in this group and one belongs to class (XLI), the most iconographically deteriorated, and on the other one the depiction is not preserved, the drachm is also of a very deteriorated type. The tetradrachms of this group consist out of: 4 coins of class XLVIIIb (2 additional ones are at the margin, containing respectively 16 and 15 wt% Ag), the 2 coins of class XLIX, 1 of class XLVII and all coins of class XLV.

I will only focus on the second group and all BSE-images shown in Fig. 99 and 100 are from coins within this group. Since SEM also provides optical information, it was hoped that a distinct silver layer would be seen on the small rim-section. This was unfortunately not the case on all coins, although it was clear on some. It was difficult to visualise the silver layer in the small polished rim-section even though it was readily recognizable on the surface of some of the coins. The dashed lines on Fig. 99 and 100give the somewhat arbitrary boundary between the core material and the silver enriched surface. Remains the question whether the present observed surface enrichment was originally intended or if it is the accidental result of selective chemical corrosive and surface-enrichment processes after the coins became buried.

[122] Beck, Reveillon, Bosonnet, Eliot & Pilon, 2003; Beck, Bosonnet, Reveillon, Eliot & Pilon, 2004.

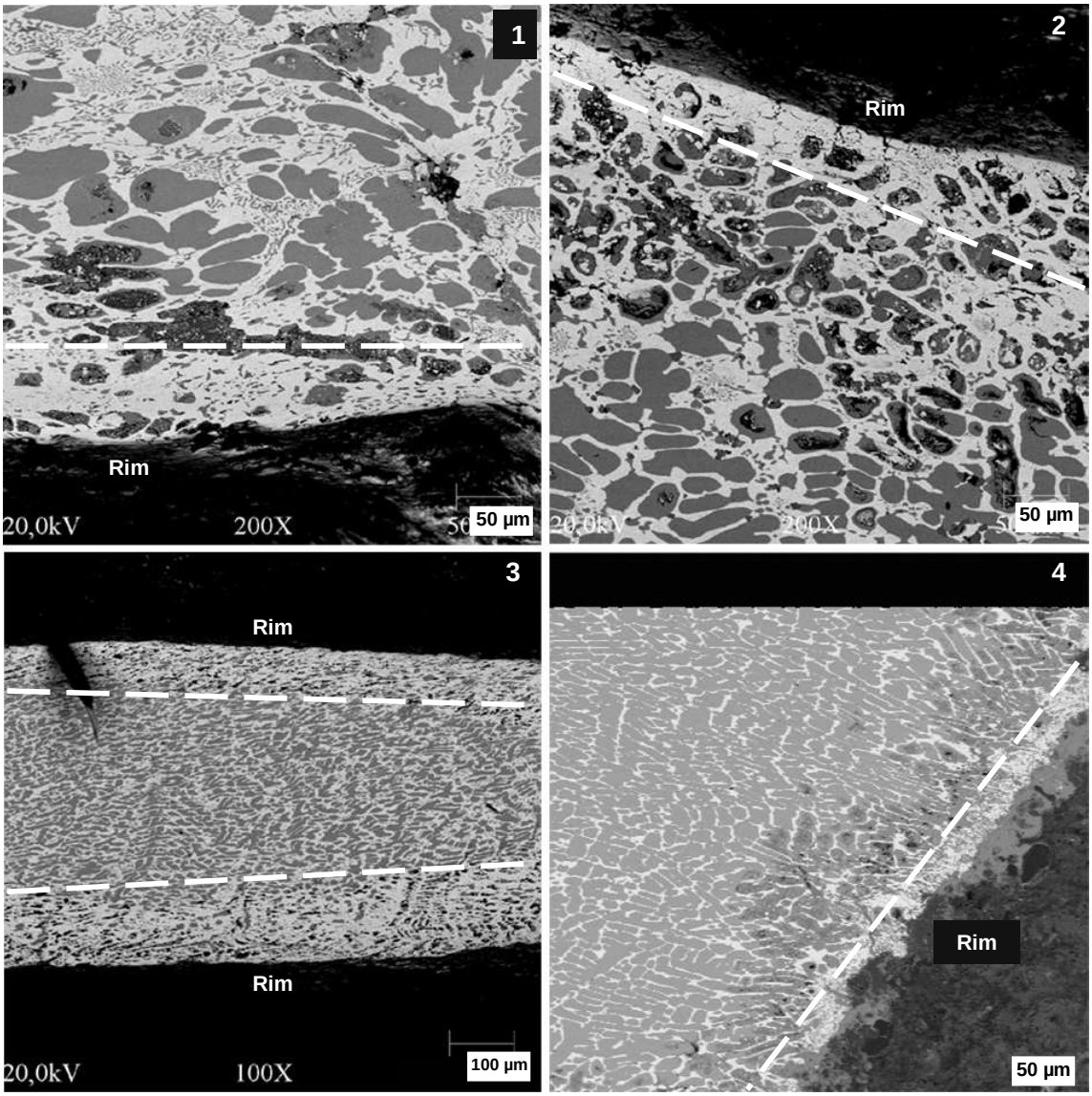


Fig. 99: BSE-pictures of silver enriched layers (1: BK 003, 2: BQ 138, 3: BM 028 & 4: AC 012).

All the pictures show a layer where the silver fraction is more abundant than it the core material. This layer is between 50 µm and 150 µm, what is more than in the experimental work described above (10 – 40 µm). Two reasons can be offered for that. The first is a practical one. Since the coins were not cut in two, mounted in bakelite and polished to a perfectly flat section, the natural rounding of the coins plays a role. The sections seen are rather rounded and part of the silver layer is actually already part of the curve going towards the coined surface. The second reason could be that the coins went through a prolonged heating phase that generated a more thorough oxide layer and by consequence a thicker silver layer. The only example that might be due to post-burial changes is Fig. XX-4 where the silver layer is more porous and corrosion can also be seen underneath within the copper matrix.

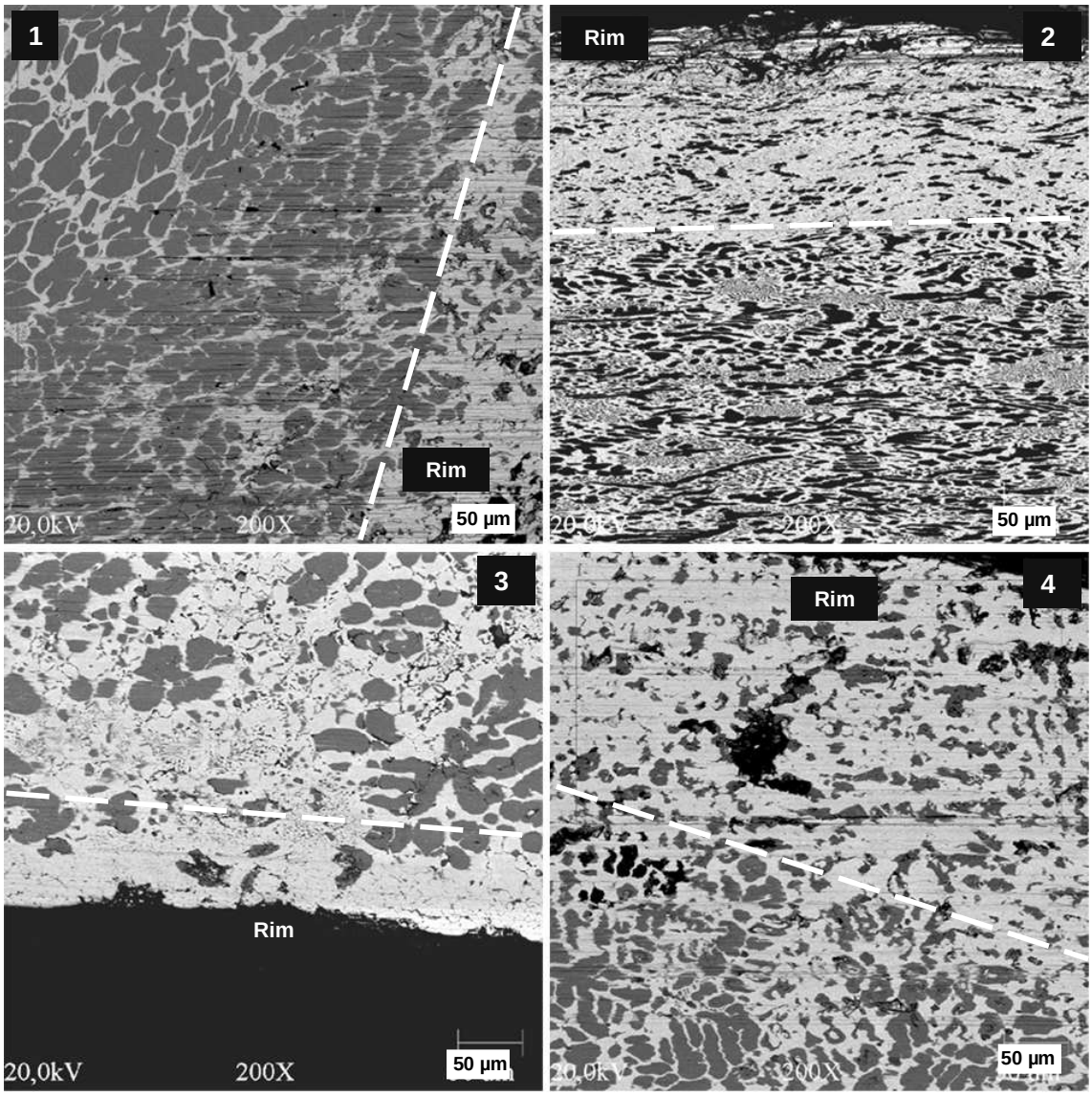


Fig. 100: BSE-pictures of silver enriched layers (1: BS 091; 2: BS 171; 3: BR 100 & 4: BQ 104).

It is impossible to exclude a natural enrichment process but some circumstantial evidence should be taken into account. Firstly, the fact that complete silver coins do exist shows that silver coins were circulating. This does not mean that there could not be a second type of coin that was made of another alloy, but the silver issues probably set the standard. Producing them with a lower silver content but similar appearance might have been an objective.

Secondly, the fact that the base metal always contained a least some silver is puzzling, because if the coins were silvered out of economic considerations the core should be of the cheapest base metal possible, e.g. copper. This is attested in Roman coinage that suffered from severe debasement during the 3rd c AD. By 250 AD, the production of complex copper alloy (Cu-Sn-Pb-Ag) coins with a silvered surface became common practice. This layer was only a few microns thick and in the bulk composition only a small quantity of silver inclusions was present[123]. One particular group of tetradrachms (class XLV) of silver-poor alloy has a small amount of tin present (below 5 wt%). From a metallurgical point of view it was

[123] Vlachou, McDonnell & Janaway, 2002: II9.2.2 - http://www.mrs.org/publications/epubs/proceedings/fall2001/ii/

suggested that this could generate a whitening effect of the silver. So with less silver a more silvery appearance is created. Since now actually figures on this technique were found, it was decided to test the effect of 5% of tin in a Cu-Ag-Pb-alloy (see experimental coins below).

Finally, SEM also provided optical (BSE) images and on some polished rim-sections the silver enrichment can be clearly distinguished (Fig. 99-3 & 100-2). This implies that they underwent consolidation after the silver-rich layer was formed and can thus not be the result of a post-burial process. Taking all this into account, it is probable that a process of pickling was used on at least some of the coins in order to give them a silver-enriched surface before striking them.

7.4.5. Mould casting versus *die striking*

Why talk about cast coins? The first obvious reason is that even if coins are struck the blanks had to be cast (the dendritic microstructure seen on Fig. 99 are remnant to that). The second more enigmatic reason is the find of three fragments of stone coin moulds in the fort of Mleiha[124]. The three mould fragments were made to produce a finished product, in this case a type of tetradrachm close to D.T. Potts class XLIV[125]. In the best-preserved fragment five identical heads were carved (Fig. 101). The mould is to be positioned up straight and the arrow in Fig. 101 shows the funnel where the molten metal could be poured in. The four holes were most probably to fix the other side of the mould. The fact that stone is used as mould material is rather exceptional since most known moulds (e.g. Roman) were of clay. Moreover they were only used to cast the blanks of which the coins were struck[126]. The most complete mould fragment was found in an uncertain context, collected by workers digging a trench across the fort at Mleiha in 1990. The two others fragments come from an eroded area in the courtyard and from the floor of the SW-tower of the fort. The excavators point to the fact that their presence in the fort together with the traces of 'bronze' casting could be evidence of minting. But on the other hand they are also aware of the problem that there is a total absence of any registered cast coins of this type in the numismatic collection from SE-Arabia[127]. A point also made clear by R.C. Senior[128].

Fig. 101: The best-preserved coin mould found at Mleiha.

It is sometimes difficult to differentiate between both techniques, but two basic criteria can be used. Generally, struck coins have much more detail and clearer rendering of the depiction.

[124] In one publication the mould is said to be made of a fine-grained, hard white limestone (Boucharlat & Drieux, 1991: 112), but on another occasion it is described as being made of grey sandstone (Benoist, Mouton & Schiettecatte, 2003: 66).
[125] Callot, 1994a: 22.
[126] Boucharlat & Drieux, 1991: 111-113.
[127] Benoist, Mouton & Schiettecatte, 2003: 66.
[128] Senior, 1994: 15.

A second criterion is the shape of the coin itself, die-struck coins are often convex-concave and have a peripheral flange. The surface of cast specimen on the other hand is most often flat, pitted and detail is lost. They also often exhibit the remains of the casting (casting seams, etc.). The use of the mould-technique for coin manufacture was much less widespread and less well esteemed than the die-technique, which generally produced a coin of superior quality and was by consequence less subject to imitation and forgery. But given the mediocre quality of the average coin from SE-Arabia, the moulds from Mleiha could have produced cast coins of similar quality.[129]

Dies are rarely found on excavations and ed-Dur is no exception to that. The scarcity of dies is to be explained by the fact that they were destroyed when not needed anymore, in order to prevent the dies being used by counterfeiters[130]. The known Roman die examples were made of a high tin-bronze alloy (more than 15 wt% Sn), although some iron examples are also known[131]. The usual way of minting was to lay a blank on a fixed lower die, hold the upper die above it and then impress both designs by hitting the upper die with a hammer[132]. The lifetime of these dies is partly depended on whether the die was an anvil die or a pile die and the alloy composition of the blanks. The pile die has much less external support and is more prone to damage. This can result in finding coins with identical depictions on one side but different ones on the other (not at ed-Dur)[133]. Originally this was thought to be an explanation for the appearance of some obols that only have depictions on one side. The other die may have been worn out, so one-sided coins were struck instead. But curiously it is the side that lay at the anvil side that lacks the depiction. This can be seen in the fact that this side of the coin is convex at this side, indicating that is was placed in a hollow anvil die. The alternative would suggest that the anvil die was rounded, a very unpractical fact. An interesting detail is that the obverse of the coins, e.g. 'the Hercules head' side, is always on the convex side. This is a feature seen on most ancient coins, i.e. that the head of a divinity, a hero or a ruler was engraved in the anvil die[134]. A tentative practical explanation for this might be that the 'simpler' obverse depiction was easier to carve out in the slightly hollowed-out anvil, than the more 'complex' reverse depiction on the pile die.

It is impossible to know how long a die was used and some coins found at ed-Dur were clearly struck by the same die. Experimental work and numismatic studies have shown that a good die can be used to strike more than 200 coins. The hardness of the die-alloy and the coining metal, next to the cold or hot striking of the blanks are of course crucial factors in this equation.[135] The large variation within the SE-Arabian coins could point to a relative short period of use. But without an idea of the total output of coins at the period under consideration, it is impossible to fully evaluate this conclusion.

None of the coins examined for this study gave any clear indication that they were cast. This can be observed in the slightly convex-concave shape of most coins. In some cases the die was bigger or smaller than the blank, resulting in incomplete depictions or an empty rim. In other cases the die was not nicely placed in the middle of the blank and part of the coin face is lost. Additional arguments can be found in the differences in the orientation of the obverse towards the reverse side of coins struck by the same dies, and in the deformed microstructure of the coins (Fig. 102).

[129] Sellwood, 1976: 69; Boucharlat & Drieux, 1991: 111-113.
[130] Zograph, 1977: 37.
[131] Zograph, 1977: 45.
[132] Sellwood, 1976: 63.
[133] Hill, 1922: 23.
[134] Zograph, 1977: 44.
[135] Zograph, 1977: 45; Hu, Nash & Fleming, 2007; http://www.archaeologystudent.com/coinarch/. D. Sellwood even states that 10.000 coins could be struck with a single pare of dies (1976: 72).

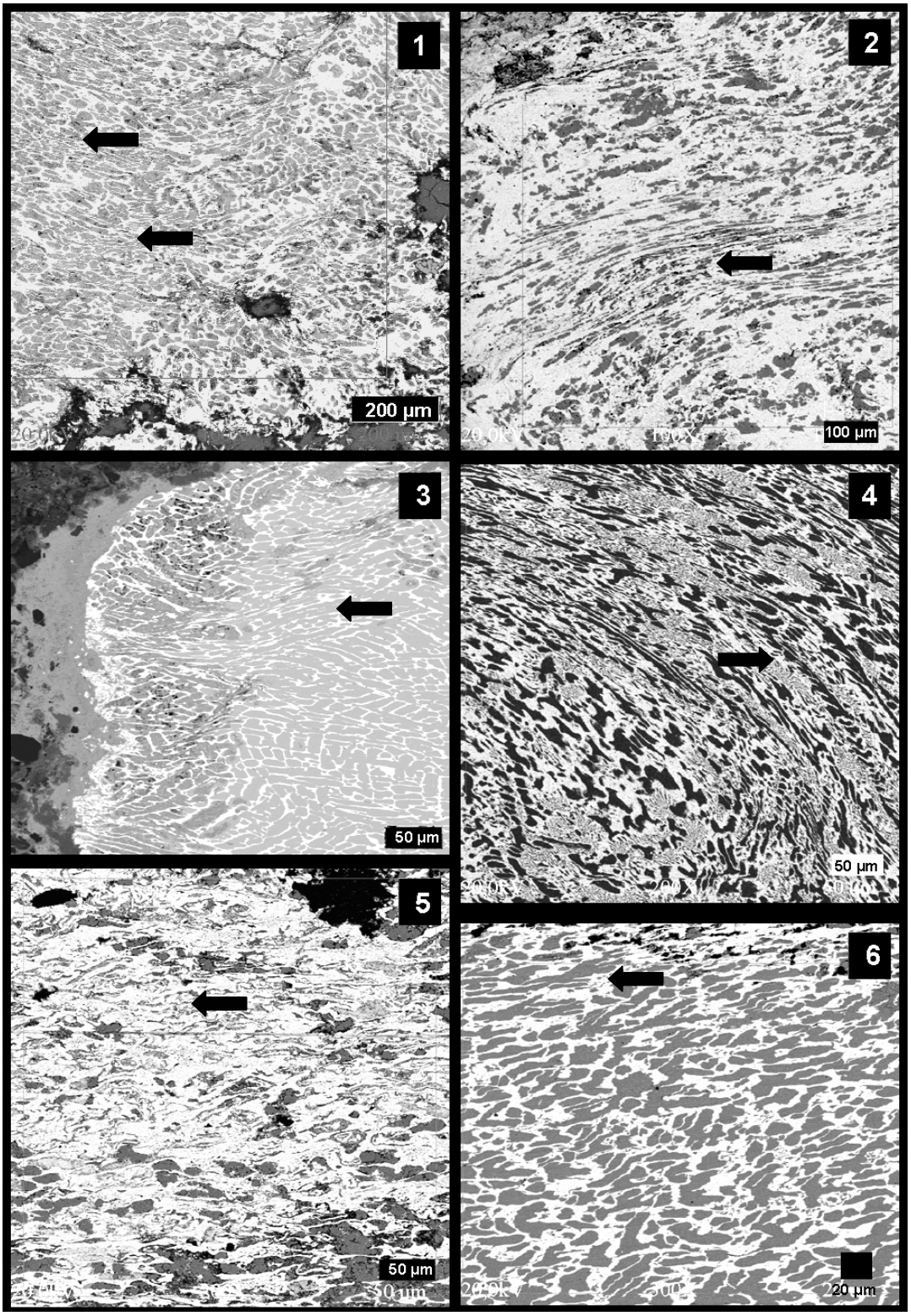


Fig. 102: Some BSE-pictures of microstructural deformation (1: M 064, 2: BS 106, 3:AC 012, 4: BS 171, 5: BQ 041 & 6: BM 028).

The BSE-SEM images of many coins show deformed grains close to the surface, especially at the rims, something that would not occur if the coins were cast. This microstructural observation eliminated some of the coins that 'casted' doubt. The deformation was

unfortunately only evident in coins with a high enough silver content. The two phases, silver-(white) and copper-rich (grey) present shows the deformation very well (see Fig. 102 for some examples indicated by the black arrows). In the coins with low silver content, the network of the silver-rich phase was not present and only 'lines' of silver-rich grains were to be observed. If these were elongated than they were however also accepted to have undergone deformation.

Still some coins remained difficult to assign. There the circumstantial evidence of the depictions on the coin was taken into account. If the obverse depiction in the coin-moulds were made with such care as the fragments found at Mleiha, it would be obvious that also the reverse was made with the same care and the complete face of the depiction would be present on the coin. This is however not always the case, leading to the conclusion that the coins were struck. For the completeness we should mention that two coins (BJ 008 & BS 285) have what looks like a 'scare' of something that was broken off from the rim, possibly the remains of the casting channel that connected several coins (see mould Mleiha). Such a scare can on the other hand also occur when blanks are cast in a mould with a *feeder* (see the experimental coins for examples). Based on the microstructure BJ 008 can however be characterized as struck. Two more coins are difficult to classify: BQ 137 (but here the obverse depiction is not nicely centred, indicative of a misplaced die) and BS 254.

According to O. Callot some specimens within a certain series of tetradrachms could be moulded (his type C). He also admits however that no traces of any casting seams were found. But absence of evidence is no evidence of absence and he does not exclude the possibility of the use of moulds, or at least an attempt to do so. He suggests that the method of manufacture could be related to the metal used: silver being die-cast, and 'bronze' occasionally moulded. O. Callot does publish two 'coins' that were cast, since they have the casting seams still attached. It is not clear however if these are coins as such.[136]

All these arguments lead to the conclusion that the coins studied here were die-struck. Beyond reasonable doubt all obols and drachms were struck. On the part of the tetradrachms it cannot be completely ruled out that some exceptions are the result of a casting process. I think however this is not the case within the assemblage studied here. An additional question that arises is if were the coins were *cold* or *hot struck*. This will be further discussed in the next point (see below) on the microstructure of the coin BR 106.

It would be interesting to analyse some of the so-called 'bronze' prills found at Mleiha in the same sector as the coin mould fragments. If they were in fact associated with a coin production, they should contain at least some silver, since all coins analysed here do. Minting was generally a royal privilege in the ancient world and the association of the Mleiha mould fragments and the fort leads D.T. Potts to suggest that the fort represents the central seat of power of the site and the region[137].

One additional object should be mentioned in this context. BJ 010 (see Fig. XX) is a small bar-shaped piece of metal with rounded corners, weighing 12,1 g. The piece is from a copper-silver alloy with about 33 wt% of silver. Although the weight does not seem to be related to one of the coins denominations, the composition of the alloy suites well within the alloys use for some of the coins. No archaeological context is recorded for this object.

[136] Callot, 1994a: 22; Callot, 2004: 67, 135 & 161: fig. 93 & 94.
[137] Potts, 1997a: 64-65.

7.4.6. Iron

In some coins considerable amounts of iron (above 0,5 wt%, but up to 8,7 wt% and 15,6 wt% in two cases) were detected (see *Appendix 11*), but there was no consistency in this. The only type for which most of the coins have elevated levels of iron are the obols of type XLVII-2. Two sources for the presence of iron can be suggested. The first possibility is that it was present in the copper that was used to compose the alloy. This can be an explanation in most of the cases, but two silver coins (BO 024 & BQ 125) also have more than 1 wt% of iron.

A second explanation would be that iron present in the soil was included in the corrosion products. If the surface was not completely corrosion free, e.g. corrosion products could be present in small cracks in the surface, this will affect the EDX results.

No further conclusions could be drawn based on the presence iron accept for maybe that not all copper used for the coinage was refined to the same extent.

7.5. Microstructure of BR 106 & experimental coins

7.5.1. Microstructure BR 106

> *"Although archaeologists are generally willing to contemplate the complete destruction of unique sites, by excavation, they often show an inconsistent reluctance to destroy even fairly common artefacts for the scientific information which they can provide."* [138]

Only one coin (tetradrachm BR 106) could be sacrificed, cut in two, mounted in bakelite and polished, to study the microstructure of the cross-section by optical microscope. Two questions are to be addresses:

1. Can additional information be found on the presence of a silver enriched layer, and if so how was it applied or generated?
2. Was the coin cold or hot struck?

The polishing of the sample did not pose any problem, but the etching procedure did. The technician tried different methods, but the results stayed poor and no clear microstructure could be resolved. The reason for this is uncertain and no solution was found.

Fig. 103 gives an overview of the different microstructures observed. BR 106 is from a copper-silver alloys (respectively 88-10 wt%) with a little tin and lead present. Copper-silver alloys differ from bronzes and brasses in that no intermediate phases are formed when the melt cools down. The white phase in the pictures is silver-rich, the orange/pink background is the copper-rich matrix, the dark grey inclusions are lead pockets and the black spots are vacuoles.

Fig. 103-1 to 3 shows the rim of the coin and the arrows point to the presence of thin silver-lenses at the surface. This is of course not conclusive evidence for silver enrichment at the surface, but it is reported in the literature that this silver skin can be very thin and difficult to detect. Moreover the silver skin is not continuous, but the effect of corrosion can play an important role in the destruction of such a thin layer. The silver inclusions underneath the rim are also often deformed and elongated in a direction parallel to the surface, indicating the effect of compression during the striking of the coin. The reduction might have been considerable in order to produce such elongated silver structures. Silver is softer than copper and will more easily be deform upon impact than the copper matrix. In the middle part this effect is not observed throughout the coin and it is rather concentrated near the surface (Fig. XX-3). At the rim the silver inclusions are more spherical, possibly because the material could deform without any constrained at the side (Fig. XX-6).

The mildly etched microstructure on Fig. XX-4 shows coring from the material (often around the silver inclusions) and the lighter coloured areas are somewhat richer in silver than the darker matrix that is more copper-rich. Fig. XX-5 to 7 exhibits weakly formed small angular grains. This in not seen everywhere in the section and it proofed impossible to make the microstructure clearer. The persistence of a cored structure with a fine-grained structure on top of the coring is indicative of a short annealing time at low temperatures[139]. The grains are better developed in the centre of the coin than on the sides. They exhibit darker lines inside the grains and these could be annealing twins or mechanical deformation twins. In the first case the coin was annealed after the striking, in the second case they can be the result of the striking after an annealing phase. To me however some of the twins are definitely annealing twins, as can be seen on Fig. 104-2. The small lines indicated by the white arrows in Fig. 103-6 and 7 might be strain lines resulting from working after the grains were formed.

[138] Cope, 1973: 221.
[139] De Ryck, Adriaens & Adams, 2003: 586.

The poor quality of the microstructure made it however impossible to make this assertion with any degree of certainty.

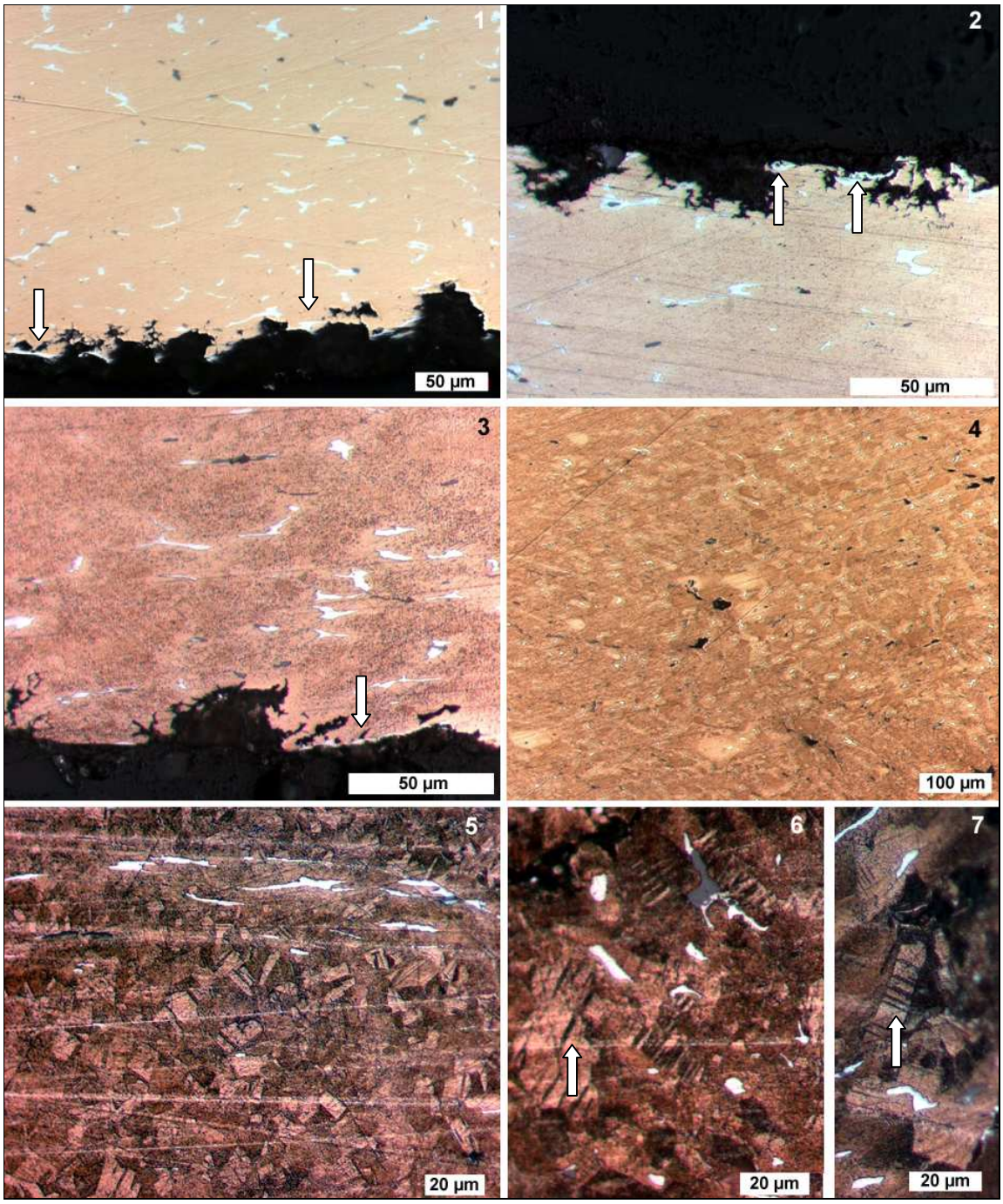


Fig. 103: OM-pictures of section coin BR 106 (88 wt% Cu – 10 wt% Ag).

This microstructure will be further discussed in the conclusions (*7.5.3.)* of this sub-chapter. First the results on the test coins will be presented, since they may contribute to the understanding of the microstructure of coin BR 106.

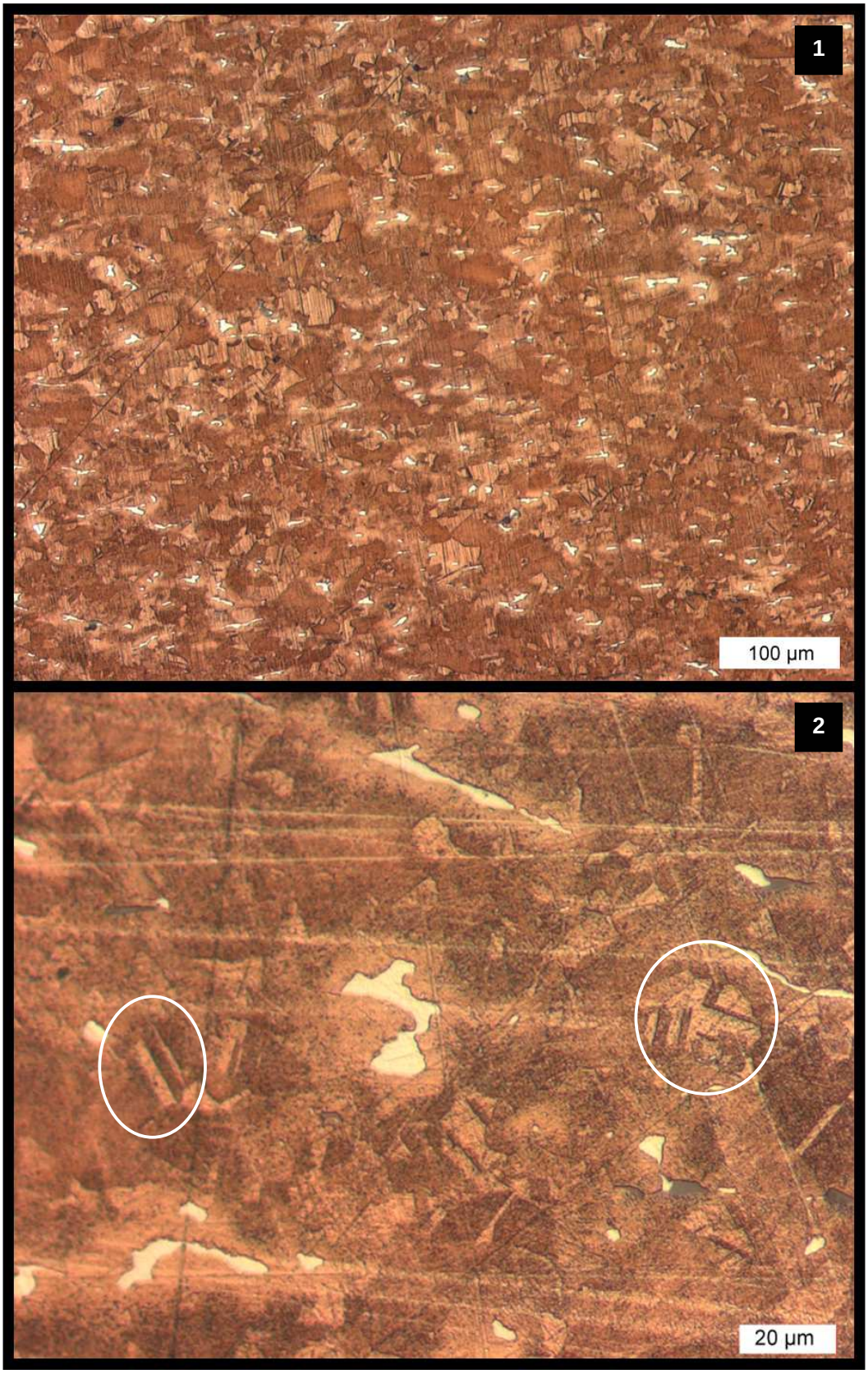


Fig. 104: OM-pictures of section coin BR 106 (88 wt% Cu – 10 wt% Ag). White circles indicating annealing twins.

7.5.2. Experimental coins

7.5.2.1. Experimental conditions

In order to resolve some of the questions around the low silver level coins, it was decided to cast some experimental coins of about the same size as the tetradrachms[140]. Three different alloys were chosen (see Table 43). Beck *et al*. have shown that coins with up to 15% of silver can be successfully treated with a pickling process and a silvery surface can be obtained[141]. In order to see if this was also possible with lower values of silver two different alloys were tested, one with 10% and one with 5% of silver. Additionally an alloy with 5% of silver and 5 % of tin was made, to see what the effect was of the addition of tin. It was also hoped that the microstructure would clarify the structure observed on coin BR 106.

Sample nr.	Alloying elements (the rest is copper)	Treatment	Observation
1A	5% Ag – 5% Sn – 2% Pb	Struck as-cast	No silver layer
1B	5% Ag – 5% Sn – 2% Pb	Low temp. roasted (*ca*. 700°C)	Oxide layer
1C	5% Ag – 5% Sn – 2% Pb	Low temp. roasted + vinegar (5%) → struck	Weak silver layer, copper visible ~ 1D
1D	5% Ag – 5% Sn – 2% Pb	High temp. roasted + vinegar (5%) → struck	Weak silver layer, copper visible ~ 1C
2A	10% Ag – 1% Pb	Struck as-cast	No silver layer
2B	10% Ag – 1% Pb	Low temp. roasted (*ca*. 700°C)	Dark oxide layer
2C	10% Ag – 1% Pb	Low temp. roasted + vinegar (5%) → struck	Weak silver layer, copper visible
2D	10% Ag – 1% Pb	High temp. roasted + vinegar (5%) → struck	Good silver layer, smooth
6	10% Ag – 1% Pb	High temp. roasted + vinegar (5%) → struck	Good silver layer, rough
3A	5% Ag – 2% Pb	Struck as-cast	No silver layer
3C	5% Ag – 2% Pb	Low temp. roasted + vinegar (5%) → struck	Weak silver layer, copper visible ~ 3D
3D	5% Ag – 2% Pb	High temp. roasted + vinegar (5%) → struck	Weak silver layer, copper visible ~ 3C
5	5% Ag – 2% Pb	Silvered, melted Ag/Sn on one side	Relative good silver layer

Table 43: Summery of alloy types, treatments and visual observations.

The alloying elements for the blanks were weighted with a 0,1 g precision scale and melted in a graphite crucible in an open forge fire to imitate the ancient conditions. The blanks were cast in a steel mould, coated with a carbon layer. After removal the casting sprues were sheared off with a chisel. One blank of every alloy was struck in the as-cast state and no further preparation was done (1A, 2A & 3A). A second blank of every alloy was oxidized or roasted in a forge fire for 15 min at a low red heat (1B, 2B, alloy 3 was not among the received samples). A third blank was treated in the same way but was afterwards put in vinegar with an acidity of 5% for 48 hours at room temperature (1C, 2C & 3C). Then it was brushed with a nylon brush, cleaned with a weak ammonia[142] solution and struck. A fourth blank (1D, 2D & 3D) was raised to a higher temperature in the forge, up to a point where it looked 'shiny' as silver alloy sweated to the surface. This took about two minutes. Afterwards it was treated in the same way in vinegar, cleaned and struck. Sample 5 was treated in a different way and a silver-tin alloy was melted on the surface. Sample 6 was treated the same as 2D, but heated for a longer time (the amount was not mentioned).

[140] I am very grateful to D. Tokar who made and treated the blanks.
[141] Beck, Reveillon, Bosonnet, Eliot & Pilon, 2003: 563-564.
[142] Ammonia (NH_3) is already mentioned by Pliny as *hammoniacus sol*. During the European Middle Ages ammonia was used by dyers in the form of fermented urine to alter the colour of vegetable dyes (www.wikipedia.org). This note only serves to show that the use of ammonia, as in this experiment, was at least possible.

7.5.2.2. Visual examination

All three alloys have a red copper colour in the as-cast state, but 2A and 3A have some more 'golden' coloured spots on their rim. Oxides appear as darker spot on the surface. No real difference can be noted among these blanks. Only two blanks were available that were submitted to a low-temperature (*ca*. 700°C) roasting phase (1B and 2B) and left untreated. 1B has a spotted oxide skin coloured brown to black, whereas 2B has a uniform dark grey/black oxide layer covering the surface.

Fig. 105: Visual results (obverse and reverse) of blanks of alloy 1 (5 wt% Ag – 5 wt% Sn – 2 wt% Pb) after different treatments.

Fig. 106: Visual results (obverse and reverse) of blanks of alloy 2 (10 wt% Ag – 1 wt% Pb) after different treatments.

Of the blanks roasted at a low temperature and treated in vinegar all show a more silvery surface. This is not always nicely shiny continuous layer, but it is very different to the un-

treated blanks, especially for alloy 2 and 3. The best results were apparently obtained with alloy 1C, but the difference in appearance is small with 2C and 3C.

For the blanks subjected to a higher temperature roasting phase 1D and 3D gave similar results. The surface layer is less smooth though when compared to the low-temperature roasting and is very thin and flaky in the case of 3D (can be scratched of). The most exceptional result is seen on blank 2D, which has a smooth silvery layer covering the complete coin blank. Sample 6 had the same composition as 3D but was heated for a longer time. The surface is rough (orange peal-like) and has a bright silvery colour.

Fig. 107: Sample 6

As mentioned above the surface of sample 5 was obtained by melting a silver-tin alloy on it. The result was a nice uniform layer, but less smooth than the layer obtained by pickling.

Fig. 108: Visual results (obverse and reverse) of blanks of alloy 3 (5 wt% Ag – 2 wt% Pb) after different treatments.

7.5.2.3. Metallographic examination

One thing that has to be pointed out here to begin with is the 'danger' of idea fixes. When the idea arose of making some experimental coins I was convinced that the ed-Dur coins were cold struck and only this path was explored here. A shortcoming that was only realised when the insight came that coin BR 106 to the contrary was most probably hot struck. A true comparison between the microstructures seen in BR 106 and the test coins is thus impossible. Still it was though relevant to present the results of this small experimental part.

All three alloys have similar as-cast structures with copper-rich dendrites and silver-rich islands in-between the dendrite arms (Fig. 109 1A & 2A and Fig. 110-6). The dendrites are fine in structure and are arranged in the same direction within on large grain. These polyhedral crystals are filled with dendrites in an orderly way according to the crystal habit of the metal. After annealing equi-axed grains are seen in some parts of the sample with the silver concentrated at the grain boundaries. Alloy 3 is the only alloy that has more massive and short dendrites.

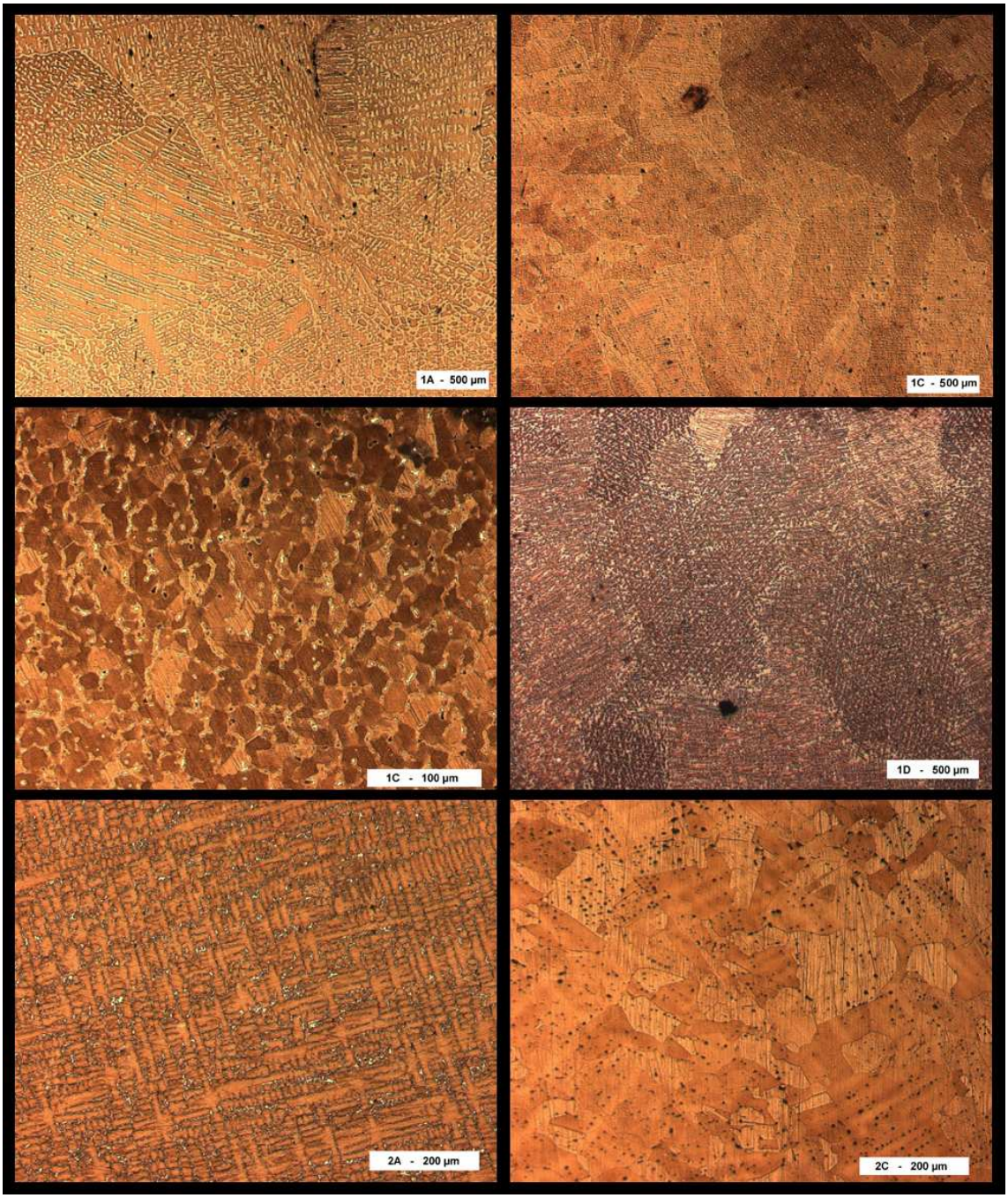


Fig. 109: Metallographic images alloy type 1 (5 wt% Ag – 5 wt% Sn – 2 wt% Pb: 1A: struck as-cast; 1C: low temperature roasting, pickled & struck; 1D: high temperature roasting, pickled & struck) & alloy type 2 (10 wt% Ag – 1 wt% Pb; 2A: struck as-cast; 2C: low temperature roasting, pickled & struck) at different stages of the process.

As a whole most of these structures are not seen on the sectioned coin BR 106 and nowhere the small grains appear in the test coins. These microstructures as seen in Fig. 109-1C and 2C do have some similarity however. It can be concluded that the structures appearing in the experimental coins do not really elucidate those seen in BR 106 and at no point the elongated silver phase is found.

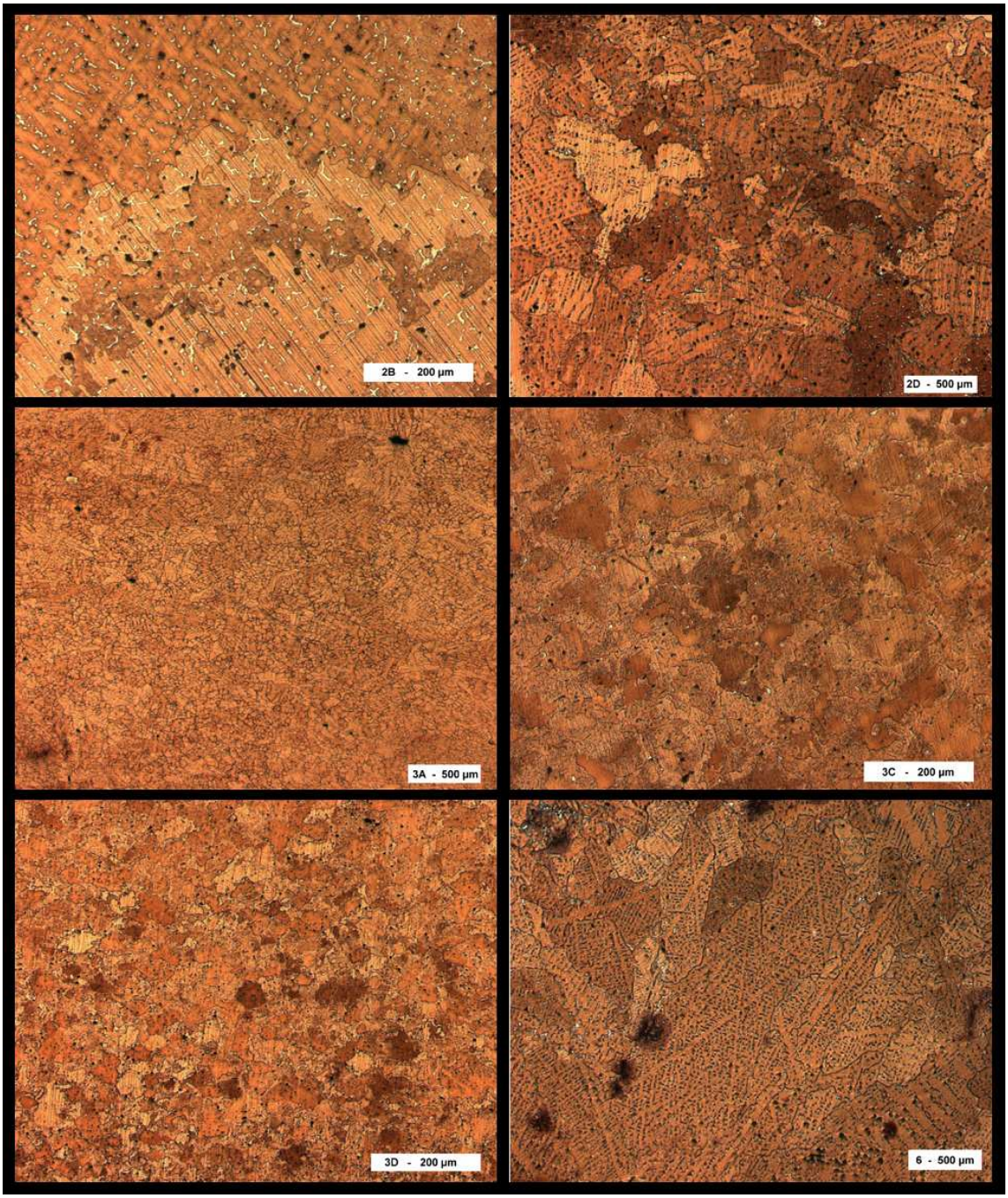


Fig. XX: Fig. 110: Metallographic images alloy type 2 (10 wt% Ag – 1 wt% Pb; 2B: low temperature roasting; 2D: high temperature roasting, pickled & struck; 6: high temperature roasting, pickled & struck) & alloy type 3 (5 wt% Ag – 2 wt% Pb; 3A: struck as-cast; 3C: low temperature roasting, pickled & struck; 3D: high temperature roasting, pickled & struck) at different stages of the process.

7.5.2.4. SEM-EDX analyses

All the experimental coins were analysed by SEM-EDX in order to see what the effect of the different treatments were on the different alloys. In order to evaluate the surface effect a second set of measurements was done on the surfaces. The results are summarized in Table 44. An interesting side effect was that the results of the compositional bulk analyses could be compared with the initial composition of the alloys used. The results show that there is a fairly good accordance with the obtained results and the expected values, considering that EDX is only a semi-quantitative method. The amount of silver seems to 1 to 2 % above or below the initial amount present in the alloy. Tin always gave results of between 1 and 1,5 % below the original amount present. Lead was found in higher values than the amount added to the alloy (between 1 and 3% higher), a problem already mentioned before. It is interesting to notice that the error is the largest when lead is present in small amounts. Overall it is safe to conclude that the EDX-results are reliable enough to use them and draw broad conclusions from the compositional analyses obtained for the coins,

	Core								**Surface**									
	Cu	**Ag**	**Sn**	**Pb**	*2 sig Cu*	*2 sig Ag*	*2 sig Sn*	*2 sig Pb*	**O**	**Cu**	**Ag**	**Sn**	**Pb**	*2 sig O*	*2 sig Cu*	*2 sig Ag*	*2 sig Sn*	*2 sig Pb*
1A	89,6	5,3	3,4	1,8	*1,6*	*0,4*	*0,2*	*0,9*	-	83,7	4,1	9,1	3,1		*1,2*	*0,3*	*0,4*	*0,8*
1B	87,6	5,4	4,3	2,8	*1,0*	*0,3*	*0,2*	*0,5*	-	88,1	2,3	2,3	7,3		*1,6*	*0,2*	*0,2*	*1,4*
1B	-				-				3,1	85,4	2,2	2,3	7,0	*0,3*	*1,3*	*0,3*	*0,3*	*1,1*
1C	87,6	6,0	3,6	2,8	*1,3*	*0,4*	*0,2*	*0,8*	-	83,0	10,9	3,4	2,6		*1,2*	*0,4*	*0,2*	*0,7*
1D	88,6	5,4	3,9	2,1	*1,1*	*0,3*	*0,2*	*0,6*	-	48,9	47,6	1,0	2,5		*0,8*	*0,8*	*0,1*	*0,6*
2A	84,9	12,7	-	2,3	*1,0*	*0,5*	-	*0,6*	-	87,4	10,2	-	2,4		*1,1*	*0,4*	-	*0,6*
2B	86,3	11,7	-	2,0	*1,0*	*0,5*	-	*0,5*	-	90,5	0,8	-	8,7		*1,2*	*0,1*	-	*1,2*
2B	-				-				6,9	84,3	0,7	-	8,1	*0,5*	*1,1*	*0,1*	-	*1,1*
2C	88,6	8,8	-	2,6	*1,3*	*0,4*	-	*0,7*	-	60,0	37,8	-	2,2		*0,9*	*0,7*	-	*0,6*
2D	85,9	11,8	-	2,3	*1,0*	*0,5*	-	*0,6*	-	17,7	80,4	-	1,9		*0,5*	*1,0*	-	*0,5*
6	85,9	11,9	-	2,2	*1,0*	*0,4*	-	*0,5*	-	7,4	90,6	-	1,9		*0,3*	*1,0*	-	*0,5*
3A	91,6	5,7	-	2,7	*1,7*	*0,5*	-	*1,0*	-	91,4	6,5	-	2,1		*1,1*	*0,3*	-	*0,6*
3C	92,0	5,9	-	2,1	*1,1*	*0,3*	-	*0,6*	-	41,1	57,3	-	1,7		*0,7*	*0,9*	-	*0,5*
3D	92,4	5,2	-	2,4	*1,1*	*0,3*	-	*0,6*	-	37,3	60,8	-	1,9		*0,7*	*0,9*	-	*0,5*
5	91,5	5,8	-	2,7	*1,6*	*0,5*	-	*0,9*	-	9,2	88,9	-	1,8		*0,3*	*1,0*	-	*0,4*

Table 44: Summarized EDX-results from measurements on the core and the surface.

In the bulk analyses no real differences can be spotted concerning the composition. One exception is 2C where the silver amount is considerably different than the other measurements on the blanks of the same alloy. The error however is similar, but on the negative side of the balance. No explanation can be given for this anomaly. The treatments do not effect the global composition of the alloys.

At the surface the evolution towards an enriched layer can be clearly seen. Alloy 1 has higher amounts of tin and lead present at the surface in the as-cast condition (1A). This is the result of a process of inverse segregation, where the low melting point metals solidify first in the coldest area of the mould, i.e. the contact zone. The phenomenon that causes a higher concentration of tin at the surface is sometimes described as *tin sweat* where tin is expulsed from the alloy and concentrated at the surface. This can also explain the lower values obtained in the bulk analyses, part of the tin was concentrated at the surface and subsequently lost by further treatments. The measurement (1B) on the roasted surface detected oxygen as part of the oxides formed. These must be mainly copper oxides since the values of the tin and silver are lower than on the as-cast surface. The behaviour of lead is different in that it is present in much higher amount than in the as-cast state, upon roasting lead also 'sweated' to the surface. When the blank is subjected to a low-temperature roasting process and cleaned afterwards (1C), the result of the silver enrichment can already be seen

and the amount of silver is about the double if compared to the bulk. It is only with the high-temperature roasting (1D) that the effect of surface silver enrichment becomes really evident where about 9 times the amount of silver is found at the surface if compared to the bulk. The tin value is very low, showing that even more tin is lost during this high temperature process. The differences in the level of silver present at the surface can also be visually noticed (see Fig. 105).

The amount of silver found at the surface of alloy 2 in the as-cast state (2A) is somewhat lower than in the bulk, showing the inverse segregation mentioned above. After a roasting the percentage of copper in the form of oxides is considerable higher and the silver level is very low (2B). This is also visually observed in the massive black oxide layer seen on Fig. 106. For the lead a similar phenomenon is attested as with 1B, i.e. a much higher concentration at the surface then in the bulk. The effect of a low-temperature roasting followed by cleaning (2C) already generates an enrichment of silver at the surface, but becomes really clear after a high-temperature roasting phase, where up to 80% of silver is found at the surface (2D). The blank 2D produced the best results in this experiment and shows that with only 10% of silver included in the original alloy, a very nice silvered layer can be obtained. The differences in the level of silver present at the surface can also be visually noticed (see Fig. 106). In sample 6 the silver levels on the surface were even higher, but the visual result was entirely different. The surface was rough and did not exhibit the whished for. This shows that the process needs to be well controlled to obtain the desired effect.

Alloy 3 was used to evaluate the effect of the 5% of tin added in alloy 1. In contrary to the other two alloys the silver content at the surface in the as-cast state is somewhat higher than in the bulk (3A). No roasted blank was provided for this alloy. The difference between a low- and high temperature roasting process is minimal (3C *versus* 3D). Visually the result of 3C is even better than for 3D. The layer is however not very consistent, nor complete and tends to flake-off (Fig. 108). This experiment shows that the effect of tin does not create a better or brighter silver layer in an alloy with 5% of silver. The whitening effect of tin on silver as described above is not obtained by this experiment and within this study the presence of 3 to 4% of tin in some of the low silver content coins cannot be explained as a catalyst to produce a silver enriched surface. This is not sustained by the analytical or by the visual effects on the experimental coins (Fig. 108).

Sample 5 was also made from alloy 3 and the backside shows a similar surface as 3C. The obverse side was treated with a different technique, a silver-tin alloy was molten on the surface[143]. This resulted in a nice silvered surface, with a high silver content. No tin was detected on the surface by EDX, indicating that all tin must have evaporated upon heating. This method will not be evaluated any further however.

To complete the picture of the microstructure given by the optical microscopy some representative SEM-BSE images are given (Fig. 111). Alloy type 2 is the only alloy that shows a somewhat continuous network of the silver phase (2B). The other alloys have a discontinuous silver phase, especially clear in sample 5. Alloy 1 (1A, 1B & 1D) has tin included. A lighter grey phase surrounds the white inclusions (especially clear on 1D), both imbedded in the darker grey matrix. The matrix is copper-rich (only 0,5% Sn), the light grey phase has about 5% of tin and the white inclusions are silver with a small amount of copper, but this could originate from the matrix around the inclusions. So there is a coring of the tin around the silver. The small black elongated inclusions are lead and they are also most often found within or along the silver inclusions.

[143] D. Thokar did this on his own initiative.

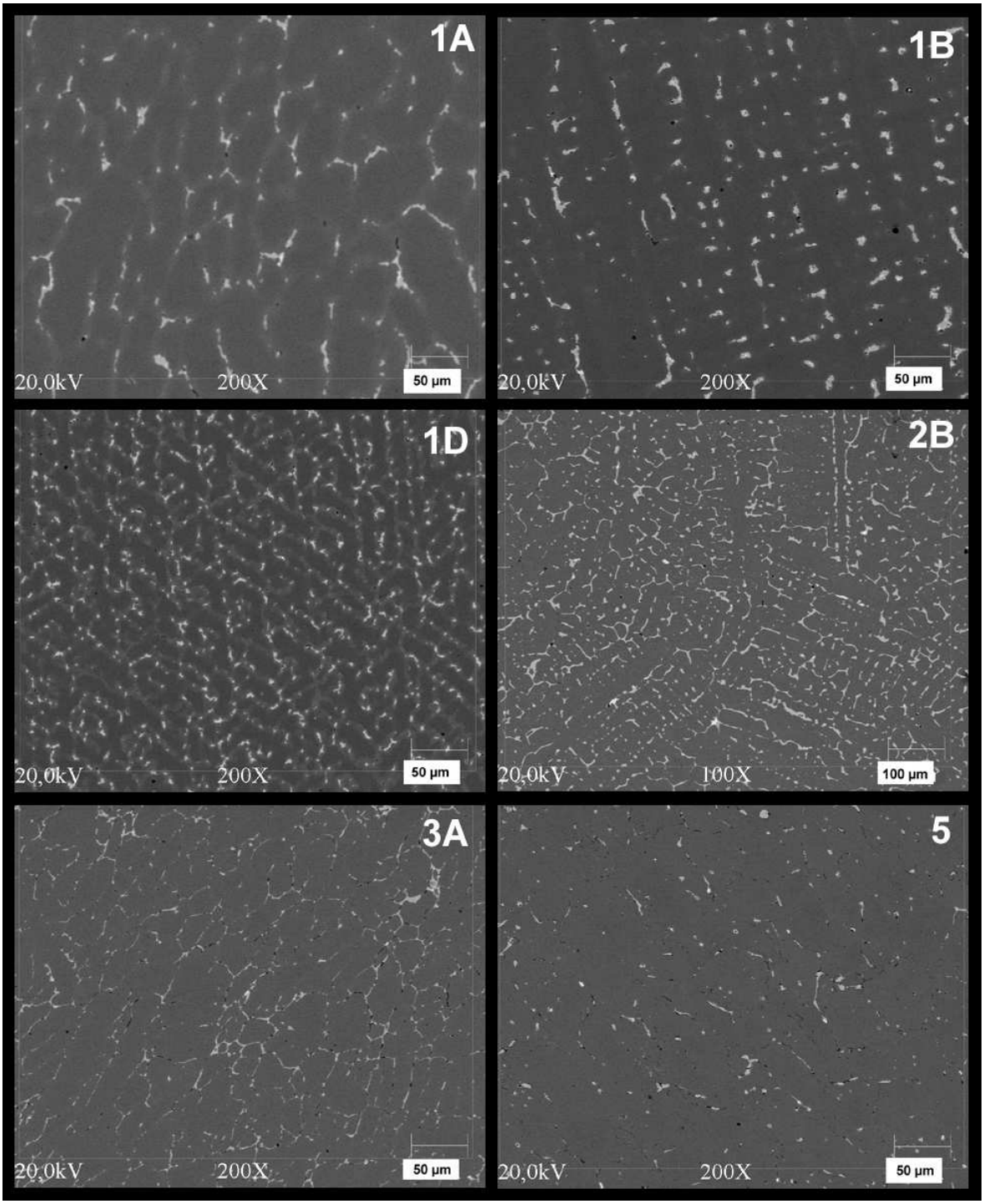


Fig. 111: SEM-BSE images experimental coins.

7.5.2.5. Surface silver enrichment layer

A last element to be discussed is the silver enriched layer itself. Only four blanks produced a layer that was sufficiently continuous and thick to be studied by optical microscope and SEM-BSE. These are the blanks 2D, 3D, 5 and 6.

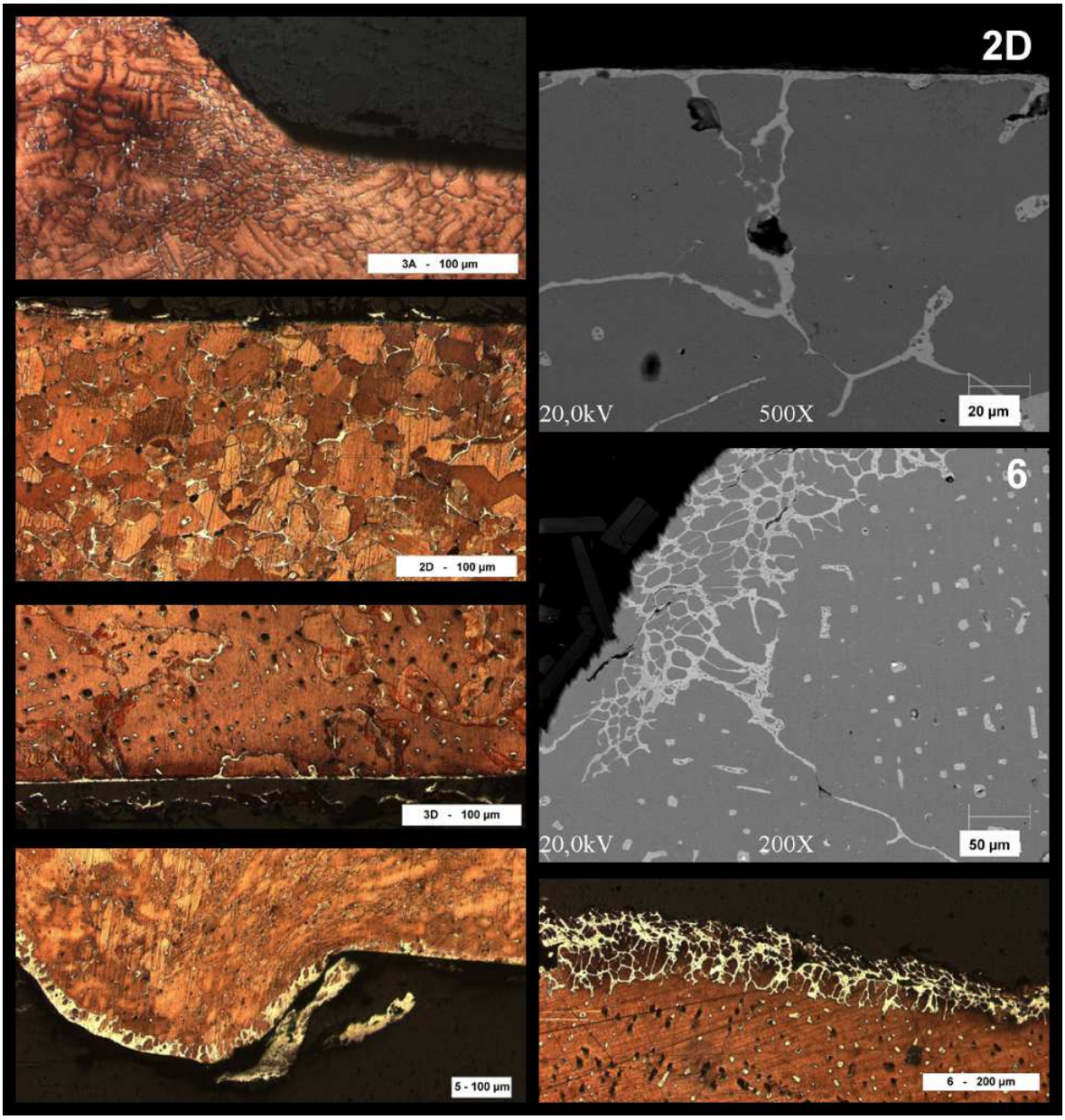


Fig. 112: Optical microscope & SEM-BSE images of the silver enriched surface layers.

Fig. XX-3A and 6 show a section through the impressed depiction. The deformed grains microstructure can be clearly observed. The blanks 2D and 3D had a nice silver surface, but it can be seen that resolving this layer in section is not clear since it is so thin that it is easily damaged by during sectioning. Coin 6 is only included to show that the technique of adhering silver by means of a tin-silver alloy created a much thicker silver-rich layer. This technique is however of no interest for this study since there is no indication that it was used. Blank 6, apart from the 'orange-peel'-like surface, also has a well-developed silver enriched surface. The silver network extends some distance into the surface. This microstructure is seen on Roman coins with a silver enriched surface. If this blank were to be used for coining some phase of polishing and smoothening the surface had to be conducted prior to striking.

7.5.3. Conclusions

- **Experimental coins**

The first problem that is addressed by the experimental coins is the amount of silver needed to still be able to produce a silvery surface by a pickling process. Of the three alloys tested here the best result was obtained with the alloy that contained 10% of silver, roasted at a high temperature and treated in vinegar. The coin was not boiled however, but just left to soak in the solution for 48 hours. Boiling might have speed up the process or enhanced the eventual result. 2D has a nice silvery look although the core material only contained 10% of silver. Coin AV 023 is the only coin that clearly has an area of silver-skin preserved and has a silver level of about 20%. This experiment shows that the lower limit of silver that needs to be present to have a successful pickling can be as low as 10%, but higher than 5%. When 5% of silver is present it is still possible to have a nice visual result, but the silver layer is not wear-resistant and the skin flakes of very easily. These coins also have a 'coppery' shine to them, revealing the copper-rich matrix underneath. Previously the lower limit was estimated to be around 12% of silver and the interconnection of the silver network was seen as crucial. This limited experimental project shows that this is not entirely true, since a 10% silver blank can still be treated successfully. The silver network is not completely interconnected in this blank as seen in the microstructures presented above.

Fig. 113: Experimental coin 2D & reproduction of the preserved silver-skin on AV 023.

The presence of 5% of tin did not improve the quality of the silver skin, to the contrary even the skin tended to flake-off due to the addition of tin. The whitening effect of tin on silver as described above is not obtained by this experiment and within this study the presence of 3 to 4% of tin in some of the low silver content ed-Dur coins cannot be explained as a catalyst to produce a silver enriched surface. The presence of tin has to be explained in another way. Two suggestions can be made. The first is that the addition of a small amount of tin lowers the melting temperature of the coining alloy and thus eases the casting of the blanks. The second possibility is that when the coining alloy was composed not enough 'pure' copper was available and scrap bronze was used instead or at least added to the alloy. I would tend to go with the first explanation, since the tin content is fairly uniform and low tin-bronzes are rare among the copper-base alloys from ed-Dur analysed in this study. So the chance they were used as scrap metal is small.

- **Coins BR 106**

The last objective for making the experimental coins was to clarify the microstructure of coin BR 106. Coin BR 106 does not have a continuous silver layer preserved at its surface, but elongated silver grains do appear at the surface and underneath. Possibly the silver-skin was more continuous but effected by corrosion processes. The experimental coins show that the silver skin generated by pickling can be very thin and even when very clear before sectioning the coin, it was difficult to resolve it under the microscope. The mechanical effect of sectioning is important to understand this and the abrasive cut-off wheel destroyed the thin skin on many places. The elongated silver grains (and some lead inclusions) parallel to the

surface however show clearly that they were deformed and thus the coin was definitely struck.

The appearance of the small grains inside coin BR 106 with some obvious annealing twins ,and possibly also the present of mechanical twins (?) and strain lines are more difficult to clarify. Still a plausible explanation can be given when the whole *châine d'opération* of the coin is considered.

What does the microstructure of coin BR 106 tell us then? The first step would have been to create a cast blank. Cutting or punching blanks is in theory possible but there is no real evidence that this was done in antiquity. Four basically different ways of producing blanks are known:

1. Blanks can be cast in a closed two-part mould, creating similar blanks of the desired thickness and size[144]. This was mostly done in a mould that produced more than one blank at the time and the depressions in the mould were interconnected with a casting channel and additional channels to let the air out. Such a mould is to be positioned upright in order to allow the metal to run through it and functioned as the coin mould found at Mleiha. The result is that the blanks have 'scares' remaining from the casting and air channels that had to be removed. The mould probably had to be coated with a fine layer (e.g. carbon dust) to facilitate the removal of the blank. There is no real evidence of this on the SE-Arabian coins (except possibly on two of the coins in the ed-Dur collection as mentioned above). Also the weight difference as noticed between the coins does not plea in the advantage of this explanation.
2. Coin blanks can be cast in a mould with a depression for every blank, without any interconnection. Such a mould is essentially flat mould and is placed horizontally. Whit the necessary practise blanks of similar weight can be produced, but still a fair amount of variation might occur.[145] Since these mould are most often open moulds the surface of the metal exposed to the air is prone to oxidation and the upper surface is not completely flat due to the effect of surface tension and the result of the pouring of the metal. This convex upper surface can be flattened out by hammering the blank into shape.
3. The third possibility is that the molten metal is just poured on a smooth and flat surface and due to the physical effect of surface tension contracts to form a round disc (like a drop of water that falls on the table). As with the previous method these pellets have to be flattened by hammering.[146]
4. A forth possibility is that the blanks were actually made by reusing older coins. The previous depictions were then removed by hammering out the coin[147]. This is most probably not the case in SE-Arabia, since there is some relation between the composition and the typology of the coin.

In general option 2 and 3 can be considered within the context of the SE-Arabian coins (although option 1 cannot be completely ruled-out). Alternatively pre-alloyed blanks could have been imported into SE-Arabia and only the striking of the coins was done locally.

The fact that a clear as-cast structure is absent (as opposed to the structures seen in the experimental coins) shows that the blank used for coin BR 106 was thoroughly annealed at some point. Self-annealing can occur when a cast is slowly cooled, but in antiquity this cannot be as thorough to completely remove the as-cast structure. The remains of coring are however still observed and this means the annealing was not as complete to also remove that. Following the hypothesis of the pickling the coin blank was heat-treated before striking in order to generate an oxide layer, annealing would simultaneously occur. Such an oxidation

[144] Zograph, 1977: 36.
[145] Zograph, 1977: 36.
[146] Sellwood, 1976: 66.
[147] Zograph, 1977: 37.

phase would have been crucial for coin BR 106 since it only contains 10 wt% of silver and the automatic generation of a silver enriched layer would not occur.

The microstructures of the experimental coins (see above) exhibit larger more or less hexagonal equi-axed grains with coring and as-cast remnants at the end of the treatment (i.e. cast, oxidized, pickled and *cold struck*). This is the result of the oxidation phase these blanks went through. Cold striking generated deformation but this is only evident at near the surface. This structure is fundamentally different from that seen in coin BR 106, where small angular grains and *annealing twins* are seen. These twins can only be the result of an annealing phase after deformation. If the blanks for the SE-Arabian coins were produced by option 2 and/or 3 as described above, the deformation would have occurred after casting and before the oxidizing heat treatment. This would result in grains with annealing twins. But since the heat that had to be applied to the blank would have had to be considerable to get the necessary oxidation layer and subsequent silver enrichment below, large grains with annealing twins might be expected.

The next phase of striking introduced a next phase of deformation. The annealing twins are however also not deformed (white circle on Fig. XX-2) and the grains are small, (possibly) indicating an additional annealing phase after deformation. But again the microstructure was rather difficult to resolve and near to the surface no clear grains were seen. It is at this point that the idea that the BR 106 was actually hot struck sprung to mind. The initial idea of cold striking was prompted by the cracks that appeared in some of the coins, but this can equally be the result of hot striking if the force of impact is large enough. Moreover the small cracks appear more regularly on the small silver obols than on the large tetradrachms.

Coin blanks can be struck either cold or hot and the metallurgical evidence for this is not always easy to interpret. Heating above a certain temperature causes recrystallization of the metal. Cold striking causes distortion of the microstructure near the surface, which will not occur if the coin is struck when hotter than the recrystallization temperature. But if the struck impression is shallow, little deformation may be caused however[148]. L.H. Cope stated that it was normal Roman practice to hot strike coins from a temperature sufficiently high to provide good plasticity. The blanks were heated to about dull red-heat before final single-blow striking between much cooler dies. This is according to him sustained by the microstructures observed and the residual hardness of the coins.[149]

By combining the microstructure published by L.H. Cope[150] and D.A. Scott[151] an idea can be formed of the microstructure that can be expected in coins that most likely were hot struck. The basic characteristics are:

- An elongated (so deformed) silver-rich phase or network parallel to the working surface within a copper-rich matrix.
- Small angular grains with sometimes (straight) annealing twins.
- The remnants of the as-cast structure or the presence of coring, indicating partial recrystallization.
- Strain lines can be present due to the superficial chilling effect of the dies in the final split-second of striking.

Recent experimental work by D. Hu to evaluate the practise of cold and hot striking on silver and debased silver coinage (60% of silver) produced some interesting results.[152] The experimental blanks were cast in an open mould and hammered to shape. The silver blanks were easy to hammer-out because of their softness but they were more susceptible to stress

[148] La Niece, 1998: 119.
[149] Cope, 1972a: 265.
[150] Cope, 1973: 228-229 'especially Plate 3 & 4).
[151] Scott, 1991: 86, 101 & 117.
[152] Hu, Nash & Fleming, 2007; http://www.archaeologystudent.com/coinarch/.

fractures in comparison to the debased alloy. The cold or hot working of the pellets did not change this and such stress fractures are seen on many Roman coins. In a second step the prepared blanks were die struck. The major difference between cold and hot striking was the 'bouncing effect' of the dies, i.e. the shifting of the dies due to the hammer impact. By striking pre-heated blanks this effect was much reduced for both the silver and the debased coins. Heat makes the metal more malleable so most of the force is directly transmitted to the blank and not back to the upper die. Cold striking the debased pellets led to severe bouncing and poor quality. Several blows were necessary to transmit a good image. This however progressively hardened the blank, making it almost impossible to ameliorate the image after more than four blows. The hot striking of the debased blanks improved the result a lot. The debased blanks were heated to around 600°C and they obtained a grey oxide layer. This oxide layer however flaked-off when struck, leaving a silvery surface. It should be remarked here that these experiments were done with an alloy containing 60% of silver. This is much higher than most of the tetradrachms from ed-Dur and indeed blanks with such high silver levels do not need an additional oxidation phase. The silver surface described by D. Hu is in accordance with the experimental work done by L. Beck *et al.* (see above)[153].

D. Hu concluded that the hardness of the coining metal turned out to be a crucial factor in the minting process. Although cold striking and hot striking debased coins took roughly the same time, hot striking created much higher quality coins. For silver coins, the Romans probably had the option of either hot or cold striking efficiently. For debased silver coins however hot striking was clearly advisable.

Putting all this evidence together it can be suggested that the tetradrachm was hot struck. The blank was cast, probably in an open mould, and may have received a first phase of (cold) working to flatten the blank out. Simultaneously with the oxidation phase, the metal was also annealed. If the blank was previously cold worked, grains with annealing twins would evolve. The oxide skin formed during this heat treatment most probably was removed before striking to improve the quality of the coin. When the coin was heated prior to the hot striking two things happened the metal deformed by the impact of the die and recrystallized creating smaller grains than before with straight annealing twins. This is most clear in the centre of the coin that held a higher temperature for a somewhat longer period than the surface. The small lines as observed on Fig. XX-6 and 7 can indeed be strain lines due to the superficial chilling effect of the dies in the final split-second of striking.

[153] Beck, Reveillon, Bosonnet, Eliot & Pilon, 2003; Beck, Bosonnet, Reveillon, Eliot & Pilon, 2004.

7.6. Lead isotope analyses – ICP-MS

7.6.1. Introduction

In order to address the question of possible origin of the silver and/or copper used as coining alloy, a series of ICP-MS analyses were performed. Only a small amount of coins could be used for this study due to the sampling technique. Only 50 µg of un-corroded core material was needed to do have reliable analytical results. To obtain this material a small hole was drilled into the side of the coin. Obviously only the coins with that are sufficiently thick (e.g. the tetradrachms) could be sampled. In theory the sampling could be omitted by using the laser ablation technique, which vaporises part of the surface by using a focused laser beam. This gas is then inserted into the mass spectrometer and analysed. Two practical factor however made this solution unusable. First of all the limited penetration depth of the laser beam might not allow to reach "corrosion-free" core material because. Directing the laser on the polished surface of the coin can solve this, but the sample chamber of the machine used here is to small to put the coin in the up-right position.

The second criteria that was used to select candidate coins, was their bulk-composition measured by EDX. When the amount of silver and copper are to similar, than it is useless to measure the trace elements and lead isotopes, since they are not representative for one metal or the other. The values obtained are rather an average and not usable.

Eventually only 18 of the 32 available tetradrachms were selected and the results are presented below.

7.6.2. Trace elements & lead isotopes analyses

The 18 coins were analysed for the following trace elements: Cr, Fe, Mn, Ni, Co, Zn, As, Se, Sb, Te, Au, (Sn), Hg, Pb and Bi, and their lead isotopic ratios. The results are however not evaluated in this PhD due to time restrain and the rather complex nature of the results, i.e. no clear pattern arose that linked the typology, alloy and trace elements. This data will be used for a future publication on the coins. This further study will also include the LIA.

The only evident conclusions were that the three coins of type XLV (BM 026, BS 254 & BS 284) had a very similar trace elemental composition, something also true to a lesser extent for the two coins of type XLIX (BS 080 & BS 097). The major elemental composition of the coins within both types is also very similar. This points towards the use of alloying metals of the same or similar origins. Something that can be interpreted in two very different ways: on the one hand this can be the result of using a (pre-alloyed?) metal for coining that was available at the moment and that all coins were made at the same time.

For the other types a larger variety appeared within one type. This might be indicative of the intentional alloying to the desired alloy for a certain coin type, regardless of the origin of these metals.

D.T. Potts speculated on the origin for the metal used for the SE-Arabian coinage and he suggested that the region probably provided the ore from the minting metal.[154] This interesting remark deserved more attention. A basic dataset to tackle this suggestion was collected and as said above this will be used for a later publication.

[154] Potts, 1997c: 93.

7.7. Interim conclusions coins

This chapter presents the first systematic analyses of a rather large collection of local SE-Arabian coinage (e.g. 104 specimens), which account for about 8% of all registered NE- and SE-Arabian coins. The coins are analysed for their composition and these results are projected on the typology of the coinage.

A first question to be addressed is the reason why these coins appeared in SE-Arabia. There are, at least, two possible answers or major points of view on that. The first is that the coins were minted in connection with political power, the urge of the local people and their rulers to define their identity and coins seem to be one of the means to do that. The second possibility is the more obvious function of money, as a currency for trade. Coins are issued as one of the primary tools for trade and commerce. A great increase in trade will lead to a growth in the amount of coinage struck to facilitate exchange[155].

If these coins were struck for a political agenda, we must say that their function as a *political messenger* was not very successful, since hardly any coins were found outside the ed-Dur/Mleiha territories, indicative for the fact that they were only used as a political symbol on a very local scale. I would like to add however that even if the coins were only used locally, the issuing of (to a certain extent) standardised coins over a longer period of time requires considerable organisation and centralisation. In this way they do suggest political power, but on a regional scale. This can be used as circumstantial evidence for the fact that these sites were more than temporally gathering points of otherwise nomadic people.

To evaluate the coinage on the site of ed-Dur in the frame of a trade currency, it is first of all important to define ed-Dur within a trade network. This is not an easy task, since the site is not very helpful on its behalf. On the one hand there are a lot of foreign products present (Roman glass, etched carnelian Indian beads, Parthian glazed ware, etc.) and for that matter the 32 foreign coins (of which 8 out of official excavations), on the other hand there is no clue on how they got there.

As a trade currency the importance of the SE-Arabian coins has to be brought back to its true proportions. The SE-Arabian coins listed in table 33 are undoubtfully only a part of all coins found in the region, but even taking that into consideration their overall amount is limited and again restricted to SE-Arabia. To me the local SE-Arabian coins found at ed-Dur and Mleiha were used for the *inter*- and *intra*-site trade, and must therefore have been produced locally. The generally low silver content of the tetradrachms seems to indicate that the value of the coins was no longer related to the metal they contained, but rather retained only a face value[156]. If ed-Dur was involved in bigger trade networks, than some other means of payment were used otherwise SE-Arabian coins, even to a limited extent, are bound to turn up at other sites. This has hardly been the case. If not a monetary based trade, than one based on exchange of goods has to be presumed. I will not go into this issue, but options could be the supply of food and water for the passing ships, pearling, retailing some of the luxury goods as found in the tombs, …

What do the foreign coins at ed-Dur tell us? Are they the result of actual commercial transactions or merely rarities that reached the site, but had lost their monetary function? I would be inclined to believe the second option, especially in the case of the Indian and Mediterranean coins. Moreover one foreign coin from Persis (BS 278) was perforated, which can indicate that it was wore as ornament and not used as currency.
The SEM-EDX analyses brought some interesting new results although the findings are not always as straightforward as I would like them to be. Several broad trends emerge.

[155] Senior, 1994: 1.
[156] Callot, 1994: 22.

Different metallic chemical elements were looked for. The two major components of all coins were copper and silver, so basically ***billon*** (an ill-defined copper-silver alloy) was used. However, there is a great variety in the silver/copper ratios, but only one unalloyed copper coin was encountered and all 'silver' coins contained at least a fraction of copper, probably to produce a more wear-resistant alloy or to lower the melting point of the alloy[157]. Generally the obols contain much more silver and the tetradrachms more copper. This could fit the idea of the more widespread use of obols (so their intrinsic value is determined by their silver content), whereas the tetradrachms were used locally and had a more 'token' value. The drachms are somewhere in the middle, but have a distinct group in the higher silver region.

Two other metallic elements were detected, i.e. tin and lead. Lead was present in all coins, but in small amounts (an average of *ca*. 1,6 wt%) and never exceeded the 4 wt% limit used to define leaded alloys. It can be safely conclude that none of the coins was intentionally leaded, something that does appear among some of the foreign coins analysed. Moreover the lead values are generally lower than the values seen in the copper-base alloys samples. Lead trends to embrittle copper-silver alloys[158], a problem when cast blanks are to be struck. The fact that the lead values are low in the coins shows that lead was probably intentionally excluded and care was taken in producing the coining metal.

Not one tin-bronze coin was encountered, shedding doubt on the many coins that are published as being made of "bronze"[159]. Tin was moreover only detected in a limited amount of coins, but only in low concentrations. These coins all had a relatively low silver level. The tetradrachms of class XLV are characterised by the consistent presence of some tin (less than 5 wt%). L.H. Cope[160] stated that from a metallurgical point of view a small amount of tin has a whitening effect on the silver, accentuating the silvery colour. To evaluated this statement some test coins were made with 5% of tin in the copper-silver alloy. The effect of 5% of tin in the test blanks had however no visual effect on the appearance of the alloy. The presence of the tin does thus not seem to be liked to a whitening effect and other reasons for the presence of tin have to be considered. A first explanation could be the use of recycled tin-bronze, instead of copper as alloying metal. A second possibility is that tin was added to lower the melting point of the alloy and make the casting of the blanks easier. It has to be remembered that the coins containing tin are low in silver and that the melting point of such an alloy would be closer to that of copper (1084°C) . The addition of silver lowers the melting point as well. If the amount of silver is reduced the melting point of the alloy steadily rises again. Adding tin would have the effect that the melting point is lowered again.

Most of the different classes defined by D.T. Potts are mirrored to a certain extend in the compositional data of this study. This is seen in the grouping of coins of the same class, meaning that they have a similar composition. This in turn can suggest that certain alloy compositions were intentionally used for certain classes. Coins that are probably struck with the same dies often have a very similar chemical composition. The word *intentional* of is coarse to be used with care, since the association between iconography and the alloy can also be the result of the available metal when a certain class was struck. The consistent presence of silver does however suggest that not any alloy was used.

Debasement of the coins can be seen in three ways, sometimes combined, sometimes separate. Lower silver levels are often associated with the more stylistic iconography (especially clear among the obols); if this is not the case greater variety of the silver content within the class is seen, so less care was taken to produce coins of similar alloy. A last way of debasement only present among the obols is a lower median weight of the more stylistic depictions. The lack of a true chronological sequence hinders the real evaluation of these

[157] Sellwood, 1976: 63-64; Scott, 1991: 21; Zwicker, Oddy & La Niece, 1993: 224.
[158] Cope, 1973: 224.
[159] E.g. Callot, 2004: 145.
[160] Cope, 1972a: 265-267.

parameters (weight, silver content and iconography) and this study could not change that. It is clear that the most realistic issues are the earliest coins and the most stylised are the last, but if the evolution between these two extremes was linear remains unclear.

Evidence was brought forward to suggest that an artificial process of pickling or depletion silvering was used to obtain an artificial silver enriched surface on some of the low silver coins. Other processes to apply an artificial silver layer could be ruled out on the bases of that analytical results or circumstantial evidence, although post-burial processes cannot be completely excluded. To evaluate this issue further some test coins were made and treated by pickling. The best result was obtained with the alloy that contained 10% of silver, roasted at a high temperature and treated in vinegar. The coin was not boiled however, but just left to soak in the solution for 48 hours. Boiling might have speed up the process or enhanced the eventual result. This limited experiment shows that a blank with 10% of silver, when treated correctly, can obtain a silvery surface.

As things stand at present, it is impossible to evaluate the true function of the moulds from Mleiha. No conclusive evidence was found that any of the coins were cast, so all are probably struck. It is beyond reasonable doubt that all obols and drachms were struck. In the case of the tetradrachms it cannot be completely ruled out that some were cast, but none were encountered in this collection. The great variation in depictions points to the limited lifetime of the dies used and provides a possible explanation for the one-sided obols, i.e. the wearing out of one of the dies.

There is still question concerning the possibility of hot or cold striking. Only one coin (BR 106) could be sectioned to tackle this problem. Originally it was thought that all coins were cold struck and the experimental coins were treated in that way as *comparanda*, i.e. they were cast and cold struck to observe the microstructure. Further research however cast doubt on this original view and putting all the new evidence together it can be suggested that the tetradrachm BR 106 was hot struck. Most probably the blank was cast, probably in an open mould, and may have received a first phase of (cold) working to flatten the blank out. Simultaneously with the oxidation phase to obtain a silver enriched surface, the metal was also annealed. This would explain the grains with annealing twins observed. The oxide skin formed during this heat treatment most probably was removed before striking to improve the quality of the coin. When the coin was heated prior to the hot striking two things happened the metal deformed by the impact of the die and recrystallizes again. This creating smaller grains than before with straight annealing twins. This is most clear in the centre of the coin that held a higher temperature for a somewhat longer period than the surface. Moreover hot striking coin blanks is the most wide spread technique of creating struck coins. If the technique of striking were adopted from abroad (lets say taken from the Seleucid Empire early on) then it would be logic that the coins were hot instead of cold struck.

.

Chapter 8. IRON

"Do not wait to strike till the iron is hot; but make it hot by striking."

W.B. Yeats

8.1. Introduction

Of all metals encountered and excavated at ed-Dur, iron is by far the largest group and the majority of the samples brought back to Belgium were iron fragments. It seemed obvious at the beginning of this project that iron was going to receive the major attention. A short preliminary investigation by Dr. ir. T. Ros Yanez appeared to confirm the possibilities of retrieving some interesting data from the iron. This in combination with the fact that not a lot of work has been done on iron in SE-Arabia was thought to be the right angle of research and a world of exploring and refinement of study-techniques was in front of it. An additional advantage was that the retrieved scientific information could be projected directly on the archaeological material, since that is the second pillar of this PhD, i.e. the typological (if possible chronological) study of the iron artefacts of ed-Dur.

This chapter will start with some general information on iron and a short overview of the production process of iron and its path towards a finished object. Not a lot of attention will be spent on the actual production of iron, since there is no evidence that reduction of iron ore was undertaken at the site or in the vicinity. The shaping, e.g. smithing, will be touched upon here but the residues of this process will be discussed in the chapter on the slag remains found at ed-Dur. The geological conditions and possible iron ore sources in the region will be summed up briefly, but again since there is no indication of primary iron production it was thought to be useless to give an exhaustive survey of the region.

A second part will define and motivate the preset questions that were to be answered by the experimental part. This was intended as the bases for the experimental part, which unfortunately has not achieved the preset goals, but I thought it useful to include this anyway since time and effort was spend on this.

The chapter is concluded with a short overview on the iron artefacts found in the SE-Arabian Peninsula, to illustrate the importance of the massive occurrence of iron artefacts in the PIR periods. Originally it was intended as an introduction to the typo-chronology of the iron objects from ed-Dur, but since this chapter was omitted from this PhD this short overview is included here.

8.2. General overview of iron production

Iron (Fe) is one of the most important metallic elements in the earths crust (ca. 7%) and is present in all kinds of rocks. Strictly speaking iron is the pure metal (the chemical element Fe) and as such was never produced in antiquity. The manufactured iron was instead an alloy of iron and other impurities. In many areas terrestrial ores are far more readily available than copper ores. Many lie so close to the surface that they are easily recoverable and they can be broadly grouped into oxides, carbonates and sulphides. Red oxide (haematite, Fe_2O_3) and the hydrated form (limonite, $2Fe_2O_3.3H_2O$; goethite α-FeO(OH)) both contain a high proportion of iron and are relatively free from sulphur and phosphorus. The magnetic oxide (magnetite, $FeO.Fe_2O_3$) and the carbonate (siderite, $FeCO_3$) can also easily be worked. The sulphide ores (pyrites, marcasite, FeS_2, and pyrrhotite, FeS) are more problematic, since any sulphur left in the metal affects its working properties and use. Iron ores usually contain 30-40% of unwanted materials.[1] In the past only relative iron-rich ores could be used since significant amounts of iron were lost to the slag.

Two additional sources of early iron should be mentioned, being *native iron* and *meteoric iron*. Native iron is rare and found only in some natural occurrences (none of which are known from the Near East and Middle East) since it is only formed under very specific conditions. Not all meteorites on the other hand contain iron in readily available form and the once held view on the meteoric origin of the first used iron, now is not that solid anymore. It is true that in ancient Egyptian and Hittite iron is sometimes referred to as “iron from heaven” and “iron from the sky”, but it is very unlikely meteoric iron was the only source of iron known.[2]

It is not known which kinds of ores were preferred in the Near East. Ethnographical parallels suggest the possibility of significant variations in the ores and the types of furnaces used[3]. For several different reason it may be difficult to identify the original sources when looking through the glasses of present-day geology, where economic principles are used to pinpoint ore sources. Many other parameters may have influenced the choice of ore in the past, such as availability and ore composition. It should also be taken into account that the traces of ancient mining are often completely destroyed by modern activities at the same places, or the ore sources might have been completely exhausted by the ancient miners.

As it is possible under certain conditions to produce iron when smelting copper, it is probable that a significant proportion of the iron used in the Near East, at least before the Late Bronze Age, was produced as a by-product. If iron oxide was accidentally or deliberately added to the furnace charge as a fluxing agent the iron would combine with the silica in the ore to form slag that melted and eventually run off. In circumstances of high temperatures and an extremely reducing atmosphere, small bits of relatively pure iron would have formed.[4]

- **Ore preparation & smelting**

Most of the time the processing of the ore happened close to the extraction site, to avoid unnecessary transportation. A fist step was to wash the ores in order to enrich and/or clean them. Often the ores themselves contained a certain amount of water, so in a second step this excess of water had to be removed by a process called *roasting*. This operation is preformed at temperatures between 400 and 500°C. This is easily achieved in an open charcoal fire or by burning green wood with the aid of bellows. The roasting process also expels a large part of the sulphur present in certain ores. Although sulphur does not influence the process, concentrations exceeding 0,035% cause the final metal to be brittle and to crumble when forged. Another advantage of this action was that the water caught in the ore

[1] Coghlan, 1977: 8-11; Moorey, 1994: 280.
[2] Coghlan, 1977: 1-6; Moorey, 1994: 279; Serneels, 2004: 25.
[3] Moorey, 1994: 280.
[4] Moorey, 1994: 279.

was turned into steam. This internal pressure made the ore shatter and produced smaller and better manageable pieces. Roasted ore was also more porous and easy to break down further. The size of the ore pieces charged to the furnace is important, too large chunks of ore will not be thoroughly reduced and too finely divided iron ore will restrict the air flow and thus prevent the slag from becoming free-flowing (see below and Chapter 9). Smaller pieces provide a larger surface area per unit volume of material in order to improve the chemical reduction reaction. An important additional function of roasting in the case of carbonate is to drive off the CO_2 and to produce iron oxide ready for smelting:

$$FeCO_3 \rightarrow FeO + CO_2$$
$$4\ FeO + O_2 \rightarrow 2\ Fe_2O_3$$

If these reactions are not preformed before the ore is reduced, they will occur during the smelting operation, robbing time, heat, and efficiency from the actual production of iron. The furnace is charged with alternating layers of iron ore and charcoal and sometimes recharged during the process when the charge moves down the furnace-shaft.[5]

The modern system of producing iron is an *indirect method*. Iron ore is put into the blast furnace together with calcium (limestone) and coke and heated until they melt into a liquid. High temperatures are needed for this and these generally were beyond the possibilities of the small furnaces used in antiquity. The added calcium replaces iron ions and frees more iron otherwise caught in the slag. The slag is removed and liquid iron is left that can be processed further into steel and cast iron. However, until the 15th c AD, iron was extracted from its ore by what is known as the *direct method* or the *bloomery process*, so the ore was directly reduced to iron.[6]

The smelting and processing of iron is considerably more complicated than that of the other metals used in antiquity. Iron is much more difficult to reduce and requires much stronger reducing conditions. The “late” introduction of iron in history and the elaborate technology associated with its manufacture arose from the fact the iron was the only metal of antiquity to be produced below its melting point. Iron oxide will reduce to iron at a temperature of 800°C, but in practice the operating temperature of a bloomery furnace is in the region of 1200°C, much lower than the melting point of iron (1540°C). In very simple terms the direct method of iron production involves heating the ore in a reducing atmosphere (low in oxygen) of carbon monoxide (CO), which is provided by burning charcoal. The CO is formed by partial combustion of charcoal stimulated by the air blown in the furnace through the air inlets or tuyere(s).[7] The carbon has a strong affinity for oxygen allowing it to form carbon dioxide (CO_2) where possible. In this way the oxygen is removed from the ore and iron is left. The CO_2-gas escapes through the top of the furnace.

$$Fe_2O_3 + 3\ CO \rightarrow 2\ Fe + 3\ CO_2$$

In addition to the chemical process, the iron has to be physically separate from the other mineral impurities such as silica (SiO_2) and alumina (Al_2O_3), which are the basic compounds of sands and clays. A second crucial parameter is the formation of a liquid slag from the waste products of the ore. As the temperature in the furnace raises these components in the ore melt. At about 1150°C *wüstite* (iron monoxide, FeO) reacts with silica to form a glassy wüstite *fayalite* slag, which seals the iron particles from further contact with the furnace gases and permits them to pass into the high-temperature oxidizing zone above the tuyeres.

$$2\ FeO + SiO_2 \rightarrow Fe_2SiO_4\ (\textit{fayalite})$$

[5] Sim, 1998:3; Sim & Ridge, 2002: 44-45; Ehrenreich, 1985: 20-21.
[6] Ehrenreich, 1985: 22; Sim & Ridge, 2002: 43.
[7] Tylecote, 1962: 188.

With high-grade ores some silica must be added to form the protective slag. The intentional addition of fluxes is not attested with certitude until the middle Ages (e.g. limestone, etc.) and a portion of the iron had to be scarified in order to make a slag compound.[8]

Below the tuyere the atmosphere is again reducing. The most fluid slag (80% FeO, 20% SiO_2) drains down into the bowl or is tapped by periodically opening a channel from the furnace hearth. The pasty iron particles and some fluid slag are filtered on the solid bed of charcoal and stay above the bowl and sinter together in a pasty mass called a *bloom*. This process continues till the bloom is too large and the air cannot circulate anymore and the process stops or till when the slag blocks the air-inlet.[9]

Although no fluxes were added the furnace lining was significantly consumed in the direct iron-making process, especially when rich ores were used. The analysis of non-ferruginous matter in slags shows that it derived from the furnace lining. If clay had not been available from the furnace walls, it would have been necessary to add it.[10]

• **Smithing & forging**

The result of the direct reduction process is a spongy mass of iron with slag impurities and is known as the *bloom*. In general the carbon content was low, in the range of about 0,02 to 0,15%[11]. It should however be kept in mind that the bloom could have parts that had a much higher carbon content, even bits that were of cast iron. The bloom as such is not usable and during the *primary smithing* or *bloomsmithing* phase the bloom is consolidated and reduced to a workable piece of metal, a *billet*. A billet is a piece of iron (often bar-shaped, so sometimes the term *barsmithing* is used) that was much more manageable to produce a end product. Although blooms were traded as such the majority of the iron was traded in the form of billets.

Bloomsmithing is carried out in a hearth at a temperature of approximately 1100°C, because the slag still trapped inside the bloom needs to be sufficiently fluid to be expelled, but not liquefied so it would flows out of its own. If this happens the resulting voids considerably weaken the bloom, which would fall apart as it was handled.[12] The main waste product is the *primary smithing slags* that are still quite close to the chemical composition of the production slags. The resulting iron is called *wrought iron*, which is an iron alloy, in which often many other elements are still present (carbon, sulphur, silicon, phosphorous, manganese, nickel, etc.).

Not all slag can be expelled from the wrought iron and these slag inclusions become elongated in the direction of working (*stringers*), which has an important effect on the properties of the metal (strongest along the direction of working). The malleability of wrought iron is considerably increased when the temperature is raised to above 700°C. Its toughness makes it ideal for weapons or tools that are going to be subjected to heavy shock forces such as might be encountered when using a hammer or a sword.

In the whole *châine d'opérations*, *secondary smithing* or *forging* is the last step the iron has to undergo. This step can be combined in one process with the primary smithing. Forging is the actual production of an object, by repeatedly heating, hammering, cutting, welding, etc. the iron is shaped to the desired form. Before the coming of iron metalworking was dominated by casting and additional cold working. To process iron however a complete new set of tools had to be developed that allowed extensive forging on heated iron.[13] The new

[8] Sim & Ridge, 2002: 43.
[9] Tylecote, 1962: 183; Avery, van der Merwe & Saitowitz, 1988: 264-264; Sim & Ridge, 2002: 44 & 51.
[10] Sim & Ridge, 2002: 51, but he quotes Fulford & Allen, 1992: 197.
[11] Sim, 1998: 2.
[12] Sim & Ridge, 2002: 44 & 65.
[13] Ehrenreich, 1985: 25.

tools include: hammers, fullers (to elongate iron), chisels, wedges, tongs, files and rasps, anvil, etc. Of course stone tools could also still be used. Next to a new toolset the blacksmith also had to adopt new techniques such as welding, carburising, quenching, etc. The secondary smithing phase also comprises repair and recycling of objects. The waste products of this last phase are secondary smithing slags, hammer scale, and small droplets of slag or pieces of iron that were pressed out during hammering. We will come back on the waste products in the chapter on the slags.

The nature of the smithing activity itself may leave very little evidence, in terms of features and/or structures in the archaeological record. The smithing hearth can be very simple and a small pit in the ground with one tuyere attached to a bellow can suffice. In the pit opposite the tuyere charcoal is heated with a forced draft. The hearth can be lining with a clay fabric and normally the remains of the lining are vitrified and have suffered from slag attack. The vitrification normally occurs at the place of the air inlet. Little work has been undertaken on the smithing evidence. There are numerous reasons for this but, in particular, the smithing residues are mid-way along the cycle ore-to-artefact. Studies of smelting residues provide evidence for smelting technology and potentially an approach to sourcing of ores. Similarly, the study of the artefacts provides a great deal of information on smithing technology and also, potentially (trace elements) another approach to the sourcing of the iron. The smithing residues, at present, are not regarded as sources of such information. The evidence for smithing is divided into 2 broad groups, firstly, the structural features, and secondly the residues.[14] At ed-Dur only residues were collected, no indications of a smithing hearth were recognized as such, although numerous small hearths were excavated.

The main difference between iron and copper-base alloys is that it is possible to join two pieces of iron, without the use of a solder, a process described as *fire* or *forge welding*. In this process the 2 pieces to be joined must be heated to a high temperature (yellow heat, *ca.* 1100°C). It is important to have the temperature ju st right, as at this temperature the metal is very soft but not molten, and is in its most plastic state. The 2 pieces are then placed on top of each other and hammered together. The hammering forces out any slag and then fuses or welds them together. This technique is and always was a highly skilled operation. If the temperature is too low, the parts will not weld; if the temperature is too high, the iron will burn and the resulting weld will be brittle. This technique is considerably complicated when two pieces of iron with different carbon content have to be welded together, since they both have a different optimal welding temperature. The reason why forgers are often located inside in a dark environment, is because the colour of the glowing iron is the major parameter a blacksmith can use to see if the iron is ready to be welded or not.

When fire welding it is important that the material is clean and as free as possible from surface contamination such as iron oxide. In modern operations a flux is often applied to the surface (e.g. borax), which dissolves the oxide film and prevents the formation of new ones. In ancient times products as sand, clay, salt, a mix of hammer scale and sand, etc. were used as de-oxidants, they produced a thin layer of fluid slag on the surface and upon forging this layer is squeezed out. Even when fluxes are used, each time a fire weld is made large quantities of iron may be lost if the smith is not careful.[15] Around the anvil these small fragments of oxide can often be observed and they are termed *hammer scale*. They are thin scales or small droplet-shaped fragments, very magnetic and often completely oxidized.

[14] McDonnell, 1984: 47.
[15] Sim & Ridge, 2002: 61.

8.3. Geological context Gulf-region

Iron is an element that can be present in a whole range of forms, and is common on many places on the earth. We will briefly present some of the more important iron deposits in the region.

A mineral survey in the Emirate of Abu Dhabi revealed the presence of iron oxide, like limonite and haematite, in a number of locations in the Emirates, including on some the off-shore islands (e.g. Sir Abu Nuair Island, Sir Bani Yas). Haematite was discovered in some layers of igneous rocks but its quantity and quality are such that its extraction would be economically uninteresting to modern processing[16]. Iron ores have also been located in Fujairah[17] and iron oxide was recorded on Dalma Island (haematite)[18]. The *Foreign Office Handbook* on Arabia reports that iron was mined commercially in the Emirates of Sharjah and Ras al-Khaimah in 1916-17[19].

The ophiolitic formations[20], which are the skeletal structure of the Oman Mountains, are known for their richness in copper but the iron ore appears more elusive. Gossans associated with copper deposits are certainly predictable and have been sited. A few veins of oligist quartz have been mapped in Oman, near the frontier. However, in the external range of Jebal Faiyah (close to Mleiha), Jebal Emalah, Jebal Rumaylah and Jebal Buhays, ferrous continental alterations have been discovered: ferruginous laterite (i.e. weathered product of the rock-base) on ophiolites, pisolith[21] zones on the surface of drainage debris on Maestrichian limestone, or at their feet, as well as some blocks of dismantled cuirasse, some ferric incrustations which carpet the limestone in certain fissured and slightly karstic zones. The latter, though the most visible in the landscape, offers only very slight amounts of iron oxides; the pisoliths appear to provide the most easily accessible iron ore (at a few kilometres from Mleiha). On the western edge of the Oman Mountains this continental alteration does not exist and birberites occupy the upper surface of the ophiolites[22]. The copper mining areas are concentrated in the Sumail Nappe, the ophiolitic belt running round both sides of the limestone platform of Jebal Akhdar. The mineralizations present, both oxides and sulphates, are all extensively associated with iron ore. Iron ore is present in all stages of degradation down to haematites and limonites[23]. Ores were attested on Masirah and at Marbat and specular (i.e. having a bright shiny surface) iron ore was discovered on the Tumb Islands. The presence of iron in the mountains of inner Oman was noted in the 19th c AD. Iron in the mountains around Muscat was noted. Iron oxide deposits on Abu Musa were worked briefly in 1906-7 by the Wönckhaus firm[24]. Possibly exploitable amounts of iron ore have also been located between Buraimi and Suhar, in Oman[25].

Iron minerals are present in the Oman Mountains, sometimes associated with copper deposits. But these minerals present in the iron hat are mainly hydroxides (limonite essentially) more often exploited as ochre pigment or as flux in the copper smelting. Other minerals as chalcopyrite need additional treatment (roasting) because of the sulphur content. So it is generally accepted that the iron deposits in the Peninsula were not used or only to a very limited extend.[26]

[16] An., 1984-1985: 149-151.
[17] Cottrell, 1980: 572.
[18] Mouton ,1992: 204 ; Haerinck pers. comm.
[19] Potts, 1990b: 273; Mouton ,1992: 204.
[20] Ophiolites are a sequence of rock types consisting of deep-sea sediments (after Allaby & Allaby, 1999: 382).
[21] A pisolith is a spherical to subspherical, inorganic carbonate particle and are characterized by a internal concentric lamination (after Allaby & Allaby, 1999: 414).
[22] Ploquin & Orzechowski, 1994: 25-26; Potts, 1997a: 60.
[23] Tosi, 1975: 196.
[24] Potts, 1990b: 273.
[25] Cottrell, 1980: 572.
[26] Mouton ,1992: 204.

In Saudi Arabia iron deposits are found along the Red Sea at places such as Wadi Sawawin, Wadi Fatima and Jizan[27].

Further away, but in the light of the close contact the principal iron deposits in Iran lie at Shamsabad, Khumain near Isfahan, Simnan, Qum, Mahallat, Kirman and Arak. Large deposits are found at Ghoghart near Bafq and Pul-I Gawhar. Islands such as Qishm and Hurmuz have iron oxide[28].

Throughout history much of the iron used in the Gulf region has been imported. The Arabian geographer Muqaddasi (10th c AD) mentions the iron from Yemen to Oman[29]. An account written in 1790 of commerce in the Gulf lists iron as one of the many categories of imports at Muscat from the Indian subcontinent. In 1835/6 iron reached the ports of the lower Gulf (Ras al-Khaimah, Umm al-Qaiwain, Dubai and Abu Dhabi) from Bombay and the Makran coast, and by the early 20th c iron ore and tools were being imported in sizeable amounts by the Gulf countries from various European producers.[30]

There is no question about the presents of usable iron ore deposits in the region, the question is if they were actually exploited and if local iron production took place, and if so to what extend. For the moment there is no evidence known to me that during the period of occupation of ed-Dur any iron production activity took place. It might be interesting to mention that at ed-Dur some fragments of haematite[31] were collected but these probably reached the site as “pretty” or exceptional stones.

[27] Cottrell, 1980: 572.
[28] Cottrell, 1980: 570-571.
[29] Weisgerber, 1980b: 119.
[30] Potts, 1990b: 273.
[31] Determination by B. Bashar.

8.4. Preset questions & production techniques

Two important questions are to be addressed. The first is to see if there is any trace of the use of steel or steeled objects. If yes, is this material widespread or rather rare (maybe related to imported objects)? The second question is to see if there is any indication of the use of a particular kind of steel, i.e. *crucible steel*. At this point I would like to motivate why these two questions were posed. I will start with a short overview on why steel was important and how it could be produced. A second point will focus on the different production processes for crucible steels.

8.4.1. Steel

8.4.1.1. Introduction

If we speak of steel it is necessary to first define this term: steel is an alloy of iron (ferrite) and a relatively small amount of one or more other elements. In antiquity the main and most important alloy element was carbon, and the carbon-steels are the only type treated here. It is important to note that seen from a modern metallurgical point of view all alloys of iron with one or more other elements are called steel, even when only a very small amount of an additional element is present. In archaeometallurgical terms the definition of what and when iron is to be called steel is not always clear, different authors tend to use different definitions, but in practise a carbon contents between 0,3 and 2% is used. The 0,3% value is used because from that amount onwards it becomes possible to quench the steel and a noticeably improvement of the mechanical properties is achieved. Iron with a lower amount of carbon is called *wrought iron* (sometimes also called *soft* or *mild steel*) and with a value higher than 2% (till 5%) it is termed *cast iron* (which is hard and brittle).[32] Iron with a carbon value between 0,3 and 2% is *hard steel*. Inevitably cast iron would have been made by accident in smelting hearths that were producing wrought iron, but is was useless within an iron technology that was based on forging.[33]

When carbon steel is produced, the carbon atoms find spaces between the layers of atoms in the iron crystals so they move through the hot metal even when it is solid, far below the melting point. The effect is that when the metal cools it has increased in hardness and strength in proportion to the amount of carbon dissolved. The importance of steel lays in the fact that unless wrought iron is “steeled” in one way or another, hardened by quenching, and heat-treated by tempering to reduce brittleness and induce strength, it is not generally superior to work-hardened bronze.[34]

The origin of making steel is not very clear. The way in which iron was made by the bloomery process meant that it was exposed to a readily available source of carbon, so it is hardly surprising that a carbon-iron alloy occurred in parts of the bloom. From evidence of the Luristan iron swords we may conclude that the knowledge of iron technology, including at least an awareness of the existence and potential of steel-making, was well advanced in western Iran by ca 1000 BC, but its development during the 1st millennium BC is uncertain[35]. Metallography indicated that the iron in analysed objects was very heterogeneously carburised wrought iron (e.g. from Hasanlu, 9th c BC[36]). The carbon present was, most probably a result of the smelting process and this carbon percentage was further diminished during forging. Carburisation depth would depend on the temperature and the amount of time spent in oxidizing circumstances.[37]

[32] Sim & Ridge, 2002: 117.
[33] Moorey, 1994: 278.
[34] Moorey, 1994: 278.
[35] Gilmour, 2000b: 43.
[36] Pigott, 1989c.
[37] Pigott, 1999d: 6.

Steel was not always desirable, for example in wire drawing and sheet metal production, the more ductile wrought iron was preferred[38]. Even in much later times, during the 19th c AD steel tools were sometimes deliberately avoided in the more remote areas of the United States for the simple reason that softer tools could more easily be sharpened or repaired if necessary than sharper but also more brittle ones[39]. Wrought iron also has a better resistance to corrosion than steel. This is due to the inclusions of slag, which chemically is rather like glass. Hammering elongates the slag and the grains of iron. Corrosion attack starts from the surface and penetrates inwards until it reaches a slag inclusion where it ceases to go inwards, and travels along the boundary of the inclusion and the grain. When the corrosive media reaches the end of the inclusion, the process can continue inwards and be repeated[40].

8.4.1.2. Steel from the bloom

As mentioned above the way in which iron was made by a direct reduction process meant that the carbon source (i.e. charcoal) was readily available, so it is hardly surprising that with the right conditions a carbon-iron alloy occurred. The production of a steel bloom is part of the normal functioning of the bloomery process in a tall shaft furnace, if stoked in a certain way. By carefully controlling the ratio of the charcoal to ore some of the particles of iron formed will absorb carbon. This in turn lowers their melting point and they become a liquid, dripping down in the furnace to collect in a steely bloom (or at least partly). In the end the bloom is broken up in smaller parts and the harder fractions are separated from the softer ones.[41] However this view is not sheared by all, especially on the point that part of the iron is liquefied opinions seem to differ. Apart from this it is clear that some sections of the bloom had higher carbon rates and could be selected out during the further processing of the bloom. This is confirmed by ethnographical information. Some bloomery processes, notably in Africa produced iron which often contained small amounts of carbon, but this steel is not produced an a regular basis, and the carbon content was not consistent throughout the bloom.[42] A typical type of furnace, the Zelechovice underground furnace, was specially designed to produce steel blooms (8th – 9th c AD, Moravia)[43].

The separated steely parts of the bloom could than be selectively and consciously used when necessary and welded on an iron core. As mentioned above this ability to weld 2 pieces of iron together was the biggest advantage of the metal. Hardened carbon steels are brittle in their full-hardened state. To get a long lasting implement that did not break when flexed, it was necessary to weld an edge of hardened steel to an unhardened iron or soft steel back that was ductile and had a degree of elasticity.[44] Of course steel obtained by other processes (see below) can also be used in forge-welding. Many different combinations and methods were developed to combine steel and a softer core but I will not go in to this.

8.4.1.3. Cementation

This technique is related to the *in situ carburisation* in the crucible process (see below), but is conducted at lower temperatures, 800-950/1100°C. It is a more superficial method of carburisation and is sometimes called *case-hardening*. For this technique wrought iron was put in a closed recipient together with a source of carbon (charcoal powder, plant material, etc.) or just immersed in fine charcoal a slight distance away from the tuyere to avoid rapid oxidation of the charcoal, but with enough consumption of the charcoal in front of the tuyere to maintain a high enough temperature. At these temperatures the carbon diffuses into the

[38] Pleiner, 2000: 137.
[39] Ehrenreich, 1985: 79.
[40] Tylecote, 1962: 248.
[41] Pleiner, 2000:137; Sim & Ridge, 2002: 121.
[42] Craddock, 1998a: 43.
[43] Pleiner, 2000: 190-193.
[44] Tylecote & Gilmour, 1986: 2.

surface of the iron, so forming steel, is very slow and depending on the temperature.[45] At a temperature of 920°C a case-hardened coat with 0,02 % of carbon, 1 mm thick is formed in about one hour[46]. To get a 'usable' carbon content of 0,8% a time span of at least 10 hours is needed at a temperature of 950°C [47]. The fundamental difference with the crucible steels is the thoroughness of carburisation. With this technique only a thin layer of steel was formed on the surface but sometimes thin objects were completely treated in this way.

This technique requires a very skilled smith and, while completed objects could still be treated, it is best done before an object is made. Because the diffusion of carbon into steel is so slow it was necessary to beat the iron thin and then, when the carbon had been absorbed, to fold it over and weld it into a piece thick enough to make the artefact required. Subsequent heating can even out the carbon variations, and uncareful smithing would undo the carburisation.[48]

8.4.1.4. Decarburisation of cast iron

Instead of adding carbon to the iron carbon is removed in this process. The earliest evidence of cast iron production is to be found in China and dates back to the 5th c BC at the latest (but possible even from the 9th c BC) and by the 1st c BC they developed the process of reducing the carbon content by working the molten iron in an oxidising atmosphere.[49] In this way steel could be made by carefully controlled partial decarburisation of cast iron. Despite the very advanced state of their ferrous metallurgy generally the Chinese never seem to have produced liquid steel (see below), but obtained supplies of crucible steel apparently from India or Central Asia[50]. The knowledge of producing cast iron seemed to have stayed within the China till much after the period considered here. Consequently this technique is of no importance to other regions.

It should be mentioned that pieces of cast iron are sometimes encountered on excavations, they are the result of uncareful handling of the temperature in the furnace so locally the temperature could go up to 1600°C. Most of the time they can be considered as waste products because in a technology that is based on forging wrought iron, cast iron is an unwelcome result, since it is too hard and brittle to be forged. In the frame of the production of crucible steel by the co-smelting method however these fragments of cast iron may have played a crucial role (see below).

8.4.2. Crucible steel

8.4.2.1. Introduction

Crucible steel can be defined as steel with a moderate to high carbon content, usually in the range of 0,3% right up to 2%, which has been homogenised and purified of slag inclusions and other impurities by melting[51]. Processing wrought iron in the solid state as explained above meant that impurities from the smelting process were trapped in the metal as slag stringers, resulting in weaknesses in the metal and inferior strength properties. Liquefying the wrought iron, however, would make the slag float to the surface allowing it to be skimmed off or solidify, so improved properties would be achieved. Also more carbon would readily dissolve in the molten iron and be more evenly distributed than when introduced at the surface in solid state.[52]

[45] Sim & Ridge, 2002: 111 & 120.
[46] Mangin, 2004: 217.
[47] Godfrey, 2006.
[48] Tylecote & Gilmour, 1985: 15.
[49] Craddock, 1998a: 43.
[50] Craddock & Lang, 2004: 36.
[51] Craddock, 1998a: 41.
[52] Craddock & Lang, 2004: 35.

The crucible steel of India has attracted widespread interest for centuries in Europe as the steel from which the famous Damascus blades were fashioned. Damascus blades are often associated with the term *wootz*. *Wootz* has been used to refer to any crucible steel produced in antiquity or by traditional methods. The term however seems to be a European corruption of an Indian word that only appeared in the 18th c AD.[53] New research[54] has shown that the *wootz* steel from S-India and Sri Lanka generally was not suited to produce the Damascus 'watery' pattern, but that crucible steel from N-India and C-Asia was used instead, i.e. not the crucible steel referred to as *wootz* but as *pulad*. The crucible steel labelled *pulad* has a long history and its derivates can be found in many C-Asian and European languages.[55] It is becoming clear that production of crucible steel took place over large areas of C-Asia as well as in the C-India, S- India and Sri Lanka (the region production has traditionally been confined to).

The reason that crucible steel is touched upon here is twofold. First of all at Mleiha similar slag to the one of ed-Dur was excavated and for Mleiha the archaeometallurgical study suggested that these slags could be related to some form of crucible process[56]. The evidence is however circumstantial. On the one hand the slag recovered at Mleiha cannot be linked to an iron reduction process (i.e. smelting slag). On the other hand their chemical composition differs considerably from that known from European smithing slags so they cannot be classify as such. This led the archaeometallurgist to suggest that the slags were the result of a crucible process. If this were true, this would be the first evidence of a crucible process in this part of the Middle East, with an early date on top of that. If crucible steel was used it should be looked for in the samples and objects.

The second reason is that the *Periplus*[57] states that iron was exported from India to the Roman Empire. India has always taken an important leading position in iron and steel production, and it is possible that the iron mentioned here as an Indian export was in fact a (crucible) steel. In order to address this problem properly an overview of these crucible processes will be presented to better understand the context.

8.4.2.2. Production processes

There are two main processes (with many variants) to produce crucible steel that have a long history in the Indian subcontinent, Sri Lanka and C-Asia[58]:

1. *In situ carburisation*: Is the carburisation or cementation of wrought iron by diffusion of carbon into the wrought iron in a crucible.[59]

   For this process of producing steel in a crucible small pieces of wrought iron were placed in a crucible with carbon-rich material (small dried pieces of wood, green leaves, fruit skin, etc.), together with some slag. Although charcoal was rarely used it would have worked just as well, even the charred plant material mixed with the clay of the crucible walls might have provided enough carbon.[60] The crucibles were sealed and heated till *ca*. 1450℃ for between 1 and 25 hours, after which it was cooled and the desired microstructure structure was formed. The high amount of carbon present lowered the melting point of the iron and allowed the steel to become liquid. Sometimes the complete liquid state was not reached. The result was a very 'clean' steel, free from

[53] Bronson, 1986: 15.
[54] See references under Verhoeven (*et al.*) in reference list.
[55] Craddock, 1998a: 45; Craddock, 2003: 231. Feuerbach, 2002: 253-255; On the origin of the use of *Damascus steel* see Feuerbach, 2002: 182-185.
[56] Ploquin & Orzechowski, 1994: 29-30; Ploquin, Orzechowski & Briand, 1999: 180.
[57] Casson, 1989: 28.
[58] Feuerbach, Merkel & Griffiths, 1997: 106; Biswas, 2001: 122; Feuerbach, Griffiths & Merkel, 2003: 258; Craddock, 2003: 242; Craddock & Lang, 2004: 36.
[59] Bronson, 1986: 15.
[60] Bronson, 1986: 40.

slag inclusions and high in carbon[61]. Much of the literature on crucible steel fails to stress or even consider that although the ingot was produced in a crucible it may or may not necessary have the inherent metallographic pattern that can produce a Damascus pattern (i.e. the watery pattern on the finished product). The result may also have been a good quality homogenous steel product[62]. The *in situ* carburisation is the process already described in the 3rd c AD by Zosimos (see below).

2. The *co-fusion* method involves the use of two different kinds of iron as raw materials, low-carbon iron (wrought iron) and high-carbon iron (steel or cast iron) are placed together in a crucible. The crucible is heated to a high enough temperature (estimated over 1300°C) to liquefy both or at least one (the cast iron) of the components for a number of hours and than cooled slowly. The mixture of the high and low carbon iron will form a more or less homogeneous alloy with intermediate carbon content. The co-fusion method is fundamentally different from the *in situ* carburisation in that the carbon is not derived from the addition of organic matter, but is already present in part of the iron.[63]

   Variations of the co-fusion process have been found primarily in Persia and C-Asia, but also in Hyderabad (India), and might be referred to as the "Persian method"[64]. This process was certainly carried out into India and in C-Asia in the recent past. Some of the early Islamic descriptions of crucible steel making also seem to refer to co-fusion of wrought and cast iron. The process only began to be adopted in Europe in the mid 19th c AD, after details of the Indian process had been published. It is clear that the production of crucible steel by co-fusion of wrought and cast iron was well established in the early Islamic world.[65]

   The production, modification and use of cast iron are at present poorly documented throughout this vast region, yet there is both written evidence of the blast-furnace process and surviving artefacts of cast iron[66].

Other methods of making steel in a crucible are known, i.e. the direct reduction of high quality iron ore, melting and casting steel in a crucible, decarburisation of cast iron by oxidizing liquid cast iron and decarburising of cast iron with iron oxide.[67] These will not be developed any further since there is no evidence that they were used.

A side note to be made is that not only skilled control of the technique of melting iron and producing steel had to be mastered to make these processes a success, but also highly refractory crucibles had to be produced. In producing molten steel the early craftsmen had to achieve temperatures far in excess of anything previously attained (between 1150 and 1450°C), even in metallurgical processes, and crucibles used up till then could never withstand such temperatures without themselves melting, leave alone that the process took many hours. Very different solutions to the problem of refractories seem to have been developed in different parts of the world.[68]

8.4.2.3. Textual evidence

There are many references in the ancient Classical literature to a variety of special irons originating in India. Many of these have been interpreted as evidence for crucible steel. B.

[61] Sim & Ridge, 2002: 122-123.
[62] Craddock, 1998: 44-45; Feuerbach, Merkel & Griffiths, 1998: 38-39.
[63] Bronson, 1986: 43; Feuerbach, Merkel & Griffiths, 1998: 38-39.
[64] Feuerbach, Merkel & Griffiths, 1998: 38-39.
[65] Craddock, 1998a: 44.
[66] Craddock, 2003: 239-240.
[67] Fuerebach, 2002: 112.
[68] Craddock, 1998a: 44; Craddock & Lang, 2004: 35.

Bronson discussed (and dismissed) these references in full[69]. Here we will list the most important references to Indian iron and steel, because of the prominent role India played in the production and export of iron and steel and the role that the Mediterranean-Indian trade route in general played during the ed-Dur period.

- Herodotus wrote that the Indians in the Xerxes' army at the battle of Thermopylae in 480 BC were equipped with cane arrows tipped with iron[70]. Some excavated examples from contemporary sites show surface carburisation according to A.K. Biswas[71].

- The Greek physician Ctesias (late 5th c BC) referred to 'two magical swords of Indian iron' presented to him by the king of Persia and his mother made from Indian steel (*ferrum Indicum*). The text does however not make clear whether the swords actually originated from India[72].

- The Roman historian Quintus Curtius Rufus (1st c AD) reported that Alexander the Great received 100 talents (ca. 3 tons) of *ferrum candidum* ('white iron') or bright iron from the defeated Indian Malloi and Oxydrakai (326 BC)[73].

- Some words in ancient Indian texts seem to point to 'steel', e.g. in the *Arthasastra*, *vrtta* refers to steel[74].

- Pliny's (1st c AD) *Natural History* mentions the import of iron and steel from Seres and that *Seric* iron (*Sericum ferrum*) is considered to be the best followed by Parthian iron. In this case Seric is often thought to refer not to China but to an intermediate source for which Ferghana, NE-Iran and the ancient southern Indian Kingdom of Tamil Cheres have been suggested[75]. Additional argument can be found in that Chinese sources themselves refer to steel as coming from India and Persia[76]. The fact that Parthian iron is mentioned on the second place implies that Iran was at least involved in the trade, if not the actual manufacture, of good-quality iron and/or steel[77].

- In the 1st c AD the *Periplus* mentions the Red Sea trade in iron and steel at a time when Roman trade with India was at its peak. The metal mentioned here (and by Pliny) has often been interpreted as being crucible steel. The reason for that is that it would be uneconomical to transport a relatively abundant and cheap bulk material such as iron across thousands of kilometres of ocean, unless it was something special. However no detailed descriptions of the antique cargoes survive, the ones from the Late Medieval and early Post Medieval periods do refer just to iron, in considerable quantity and at low unit cost. It was on in the markets of the Middle East. The bloomery process of making iron requires very large quantities of charcoal, and timber was a precious resource in the Middle East. The south of India was very heavily wooded and thus iron could be produced much more cheaply over there.[78]

- The first certain reference to crucible steel is the detailed description by the Alexandrian alchemist Zosimos, in the 3rd c AD. Among his work there is a chapter entitled 'The tempering of Indian steel' and it describes the production of crucible steel by the *in situ* carburisation process[79]. Zosimos is quite specific in saying that this technique came to the

[69] Bronson, 1986; Craddock, 2003: 243.
[70] Gilmour, 2000b: 44.
[71] Biswas, 1996: 386.
[72] Bronson, 1986: 18; Biswas, 1996: 386.
[73] Bronson, 1986: 18; Biswas, 1996: 386; Gilmour, 2000b: 44; Biswas, 2001: 121.
[74] Biswas, 1996: 386; Biswas, 2001: 121. The *Arthasastra* was compiled by a chief minister in the Mauryean Empire in the 4th c BC.
[75] Srinivasan, 1997: 111.
[76] Casson, 1989: 114.
[77] Gilmour, 2000b: 46.
[78] Craddock, 2003: 243.
[79] Biswas, 1996: 386; Craddock, 2003: 243.

West from Iran, and it is likely to have been in use in Iran at this time even if it was invented in India. In this crucible steel beautiful swords were made by the Indians, but traded by the Persians[80].

- There is a tradition that says that Diocletian's (4th c AD) armoury at Damascus was supplied with Indian iron or steel[81].

- Another reference to the international trade in Indian iron is to be found in the Babylonian Talmud, compiled between the 3rd and the 5th c AD, when there was a sizeable Jewish community in the Sasanian Empire. The sale by Jewish merchants of iron to other nations was forbidden because of the likelihood of this material being used to make weapons and harm the Jewish people. The export of Indian iron to the Iranians on the other hand was allowed since they were protectors of the Jews. The restriction on trade is mirrored by 4th and 6th c AD classical authors, who refer to a corresponding Roman ban on the export of high-grade iron ore to Persia which was said to have lacked its own ore resources[82]. These Talmudic references suggest that the trade of Indian iron between India and Persia was controlled by Jewish merchants between the time of Zosimos and the Islamic writers. The Indian iron is described as 'iron bars made as ingots' and suggests that this refers to crucible steel. This is reinforced by the use of the word *hinduan*, which is related to the word *hinduwani*, later used by the early Islamic writers for Indian crucible steel. From Zosimos it would seem however that the Mediterranean world also got its crucible steel indirectly from the Persians in Late Antiquity, at the same time that the Chinese report (3rd – 6th c AD) that they received special steel with a pattern of "wavy lines" from the Sasanians.[83]

- Late Classical authors, such as Libanius and Procopius, refer to a ban on the export of high-grade iron ore from the Roman Empire to Persia, which was said to have lacked good resources of its own[84].

- Middle Persian (Pahlavi) contains many words for iron and steel, including 'Indian iron' suggesting different varieties were used. Two pre-Islamic Iranian references to what may have been watered-steel blades reinforces the idea that crucible-steel making had become well established in Iran by the 6th c AD. As yet we have no evidence of when the crucible-steel industry was established in NW-Iran. It would seem likely that the arrival of cast-iron technology from China and the establishment of the trade links via the Silk Route between Iran and China would have had some influence on the later development of the industry in Iran.[85]

- The early Islamic technical authors such as al-Kindi, Jabir al-Hayyan, al-Biruni and al-Tarsusi, writing between the 8th and the 13th c AD describe the production of steel for swords in Iran and Central Asia in some detail, and recent archaeological fieldwork has identified several early Islamic production sites in Central Asia, notably at Merv[86] and at Akhsiket (see below)[87]. At both sites production terminated in the 1220s due to the Mongol invasions, and the evidence for the continuity of this production in Central Asia is not very clear.

  It is important to know that by the 9th c AD al-Kindi clearly mentions the existence of two types of iron, *natural* or *mined* and *not-natural* or *unmined*. Mined iron refers to the production of iron (soft or hard) as a one-step process direct from the ores (i.e. the

[80] Gilmour, 2000b: 46; Simpson, 2001: 15.
[81] Biswas, 1996: 386.
[82] Lang, Craddock & Simpson, 1998: 11.
[83] Craddock, 1998a: 47-49; Gilmour, 2000b: 47; Craddock, 2003: 244.
[84] Craddock, 2003: 244.
[85] Gilmour, 2000b: 47-48; Craddock, 2003: 244.
[86] Feuerbach, Griffiths & Merkel, 2003.
[87] Rehren & Papakhristu, 2000.

bloomery process). By contrast, unmined iron must refer to another (secondary) process in which wrought iron is converted into a specific form of steel. The steel produced by a crucible process that liquefied the iron was regarded as a purified form of iron. Al-Kindi described the process as mined iron placed in a crucible so it melted and became steel. This describes what happens when a mixture of low carbon bloomery steel is molten together with (white) cast iron. Later Arabic authors (11th – 12th c AD) clearly describe the use of a cast iron and bloomery iron mixture, with the addition of manganese dioxide or magnesia. This would have combined with any sulphur present in the cast iron, rendering it harmless as small particles of manganese sulphide dispersed through the resultant cast steel.[88] Written sources indicate that the Yemen had become an important region for sword making by the late Himyarite period (6th c AD), and they hint that the basis was crucible steel, partly made locally and partly imported from the Indian region.[89]

That Indian iron and/or steel existed and was traded internationally from at least the 1st c AD onwards seems indisputable, but the evidence does not permit more fare reaching conclusions. There is no evidence that Indian steel was made in crucibles during the Roman and ed-Dur period, or that it was outstanding in quality. It is only from the 3rd c AD onwards that crucible steel was produced in C-Asia, as evidenced from the account of Zosimus. In the middle of the 1st millennium AD the situation seemed to have changed and Indian iron rather suddenly appeared as the standard component for sword-production and the Islamic writers make clear that South Indian steel was used in many production centres.[90] The Delhi wrought-iron pillar (7 m high, weighing 7 tons and dating to the 5th c AD), illustrates the advanced iron technology of India at that time[91].

8.4.2.4. Different production centres, different variants on the processes

Many sites were documented in the Indian subcontinent during the 19th c AD. Two distinct technological complexes can be defined, more or less grouped geographically. A third group can be found in Central Asia and is the result of recent excavations.

- **South India and Sri Lanka**

The South Indian process practised at Mysore and its surroundings, Tamil Nadu and Sri Lanka, was based on the *in situ* carburisation of wrought iron in crucibles. A hermetically sealed crucible was charged with vegetal matter, although one sources indicates that the lid had small holes pierced into it so gas could escape.[92]

On the process used in the Periyar District of Tamil Nadu (megalithic site of Kodumanal) there is only limited evidence available that originates from some excavated crucibles, dating to between 3rd c BC and 3rd c AD[93]. Next to the main slag heaps a large oval furnace was uncovered, surrounded by 12 smaller ones. Inside one of them there was a small heavily vitrified crucible and many more fragments in the vicinity. If their identification as crucibles used to make steel is correct, these are by far the oldest remains.[94]

The general uniformity of the microstructure of the iron prills stuck on crucible fragments suggests that the ingot had a uniform pearlitic structure of high carbon hypereutectoid steel, while presence of rusty splashes and globules on the crucible walls and lids, and the curled glassy fins inside the crucibles all suggest the metal was molten. Other sites in the region as Mel-Siruvalur in Tamil Nadu, and Tintini and Machnur in Karnataka, yielded surface samples

[88] Feuerbach, 2002: 156-157; Hoyland & Gilmour, 2006: 51-52.
[89] Hoyland & Gilmour, 2006: 54 & 59.
[90] Bronson, 1986: 18-19.
[91] Gilmour, 2000b: 44.
[92] Bronson, 1986: 39.
[93] Biswas, 2001: 121; Craddock, 2003: 245.
[94] Craddock, 1998a: 49; Feuerbach, 2002: 166.

of crucible all similar enough to suggest they are also from crucible steel manufacturing.[95] In the same region during the 19th c AD a process of partly decarburising small lumps of cast iron produced during the bloomery process was practised. This was however never an important activity[96].

Sri Lanka shared with South India a reputation as a producer of high quality steel (exported to Yemen, Khorasan and Fars in Iran, and Mansura in Pakistan where swords were made of it). Excavations at a Sri Lankan site lying east of the Knuckles Mountain range, northeast of the Central Highlands, dated between 530 - 1160 AD, yielded crucibles (very similar to the much later site of Mawalgaha, in the Balangoda region of the Central Highlands).[97] Sri Lanka also took a special place by the fact that at least from the 7th c AD, till the 11th c AD, high carbon steel was produced, not in crucibles but by wind powered furnaces. In the 12th c AD crucible steel production seems to have taken over, although it probably started some time earlier.[98]

At Mysore relatively small crucibles were used. They were heated over a short time and there was a tendency to ignore the supposed benefits of very slow cooling[99]. The production in this region dates from in the 19th c AD[100] and the fundamental understanding of the technique and the advantages of producing crucible steel seem to have been partly lost.

The crucible steel produced in S-Indian/Sri Lanka is generally referred to as *wootz*. The crucibles are tempered with rice husks (organic *versus* inorganic in C-Asia) and their shape is more conical, with a rounded or pointed base. The crucible charge used was one type of iron and either wood and/or leaves. The crucibles were stacked together in the furnace, often in a cone shape. At most sites the steel solidified relatively quickly, since the crucibles were usually removed from the furnace when still hot.[101]

- **Central India**

The remains found in central India, once the state of Hyderabad, are from the co-fusion process and the steel produced in Hyderabad was better suited for the making of Damascus blades and dates to post-medieval times and the earliest record of Hyderabad as a steel producer dates to the 17th c AD[102].

The crucibles in this process are composed of charred rice husks and clay and are uniformly fine-textured, porous and black. The colour suggests that firing was performed under heavy reducing conditions[103]. Sources show that the Deccan exported iron and steel to the Middle East in the 11th and 12th c AD[104].

The difference with the S-Indian process is the type of crucible charge used. Here two types of iron (i.e. cast iron and wrought iron) were combined in a process of co-smelting. It is also possible that instead of wrought iron, iron objects were recycled. If only the material remains are considered, the C-Indian material has more in common with the C-Asian material than with the S-Indian remains. The crucible shape of the Hyderabad is similar to those of C-Asia, both are cylindrical in shape and have a flat base.[105]

[95] Srinivasan, 1997: 111, 122; Feuerbach, 2002: 167; Craddock, 2003: 245.
[96] Craddock, 1998a: 43, 52-53.
[97] Rehren & Papakhristu, 2000: 55; Craddock, 2003: 245.
[98] Wayman & Juleff, 1999: 26; Gilmour, 2000b: 50.
[99] Bronson, 1986: 41.
[100] Craddock, 1998a: 52-53.
[101] Feuerbach, 2002: 172 & 252.
[102] Bronson, 1986: 15; Craddock, 1998a: 52-53.
[103] Feuerbach, Griffiths & Merkel, 2003: 265.
[104] Biswas, 2001: 102.
[105] Feuerbach, 2002: 172-173 & 253.

• **Central Asia**

Crucible steel was certainly in production and quite widely known during the early centuries of our era. Many of the early sources mention Persia. Recently crucible-steel production sites dated to the end of the 1st millennium AD (early Islamic period) have been discovered in Central Asia at Merv (Turkmenistan) and Akhsiket (Eski Achsy, Uzbekistan)[106]. These production centres, contemporary with the early crusades, predate the Indian evidence by several centuries except for that from Sri Lanka[107].

At the successive sites of the Merv oasis remains of crucible fragments, green glassy slag and 4 furnaces dating to the early Islamic period (9th - early 10th c AD) were excavated. These crucibles were used in the production of crucible steel. Previously these remains were thought to come from a co-fusion process because prills of cast iron were found attached to the crucible walls. The recent PhD dissertation of A.M. Feuerbach did however suggested a different origin for the prills (see *Chapter 9*), the main argument being that cast iron was not known at that time in C-Asia. The possibility that cast iron was imported from China, were it was produced, should not be totally neglected however. The new hypothesis is that slag rich bloomery iron together with plant matter was packed in the crucible. It is interesting to notice that the slag rich bloomery iron would have been a low quality product and that the plant matter used could also have come from ordinary domestic waste. The result would be that from low quality products a high quality end product was made.[108]

The region of Khurasan, the area to which the city of Merv, Heart and Balkh belong, is mentioned specifically as a steel-manufacturing centre by the Islamic scholar al-Kindi (9th c AD), where swords were manufacturing made of local iron and iron from Sarandib, modern Sri Lanka[109]. The discovery of a crucible steel industry at Merv confirms contemporary Early Islamic written sources. This suggests that Khurasan was the second most important Iranian province for the manufacturing of iron and steel. The towns of Heart, Tus, Nishapur and Ghur being specially mentioned as manufacturing centres for iron arms, armour, knives and needles whereas a locksmithing industry is attested in Merv. This pattern of production was developed during the Sasanian period, if not earlier.[110]

At the sites of Akhsiket, Pap and Kuva, all situated in the Ferghana Valley (E-Uzbekistan) good archaeological evidence for large-scale production of crucible steel within the Islamic world, during the 9th to 12th c AD was found[111]. The substantial amounts of slag cakes preserved are unique among the known finds of steel making crucibles and the production of crucible steel seems to have been undertaken at a more industrial scale. This initially let Th. Rehren and O. Papakhristu[112] to suggest that the crucible charge consisted of a significant proportion, if not entirely, of ore which had been smelted to metal within the crucible. This would be in contrast to all the other known crucible processes (i.e. *in situ* carburisation or co-fusion). The idea of ore smelting was further reinforced by the nature of the slag, being similar in composition and colour to early blast furnace slags[113]. This hypothesis was rejected however by the same authors and replaced by a proposed process of *in situ* carburisation of slag rich blooms. The ceramics appear to be somewhat larger than in Merv and they have domed lids (flat at Merv). The crucibles at these sites had a flat base. The compositions of the ceramics are fairly similar, having a highly refractory, light grey firing and alumina-rich body. The calcium content in ceramics from Merv is much higher however than at Akhsiket.

[106] Lang, Craddock & Simpson, 1998: 7.
[107] Rehren & Papakhristu, 2000: 55.
[108] Griffiths, Feuerbach & Merkel, 1997: 116; Feuerbach, 2002: 31-32; 124-125 & 253; Feuerbach, Griffiths & Merkel, 2003: 259
[109] Feuerbach, Griffiths & Merkel, 2003: 258.
[110] Simpson, 2001: 15.
[111] Rehren & Papakhristu, 2000: 55; Feuerbach, 2002: 129-144.
[112] Rehren & Papakhristu, 2000.
[113] Rehren & Papakhristu, 2000: 58.

The slag present at the inside of the Uzbek crucibles is much thicker than the one found on the Merv crucibles.[114]

The remains of the crucible steel production from Merv and Uzbekistan share many common characteristics. They do however not result from an entirely similar process. The crucibles used in C-Asia were made of refractory clay with quartz, so inorganic, temper. The crucible sat on the furnace floor and gravel like material was placed in-between the crucibles. The nature of the furnace charge is still uncertain but the best hypothesis is bloomery iron and plant matter. The crucibles were fired until the steel was liquid and then left to slowly cool inside the furnace. The result was a high carbon steel ingot virtually free from slag and non-metallic impurities.[115]

8.4.2.5. Early crucible steel artefacts

Next to the literary references mentioned very few artefacts could be recognized as being of crucible steel dating to the 1st millennium AD. Possibly the earliest crucible blade comes from Luristan (Iran). The problem is that this object did not come from a controlled excavation and that the usual date for these objects (1st millennium BC) and provenance cannot be confirmed. [116]

The earliest evidence of the use of crucible steel was found amongst the ironwork excavated at the ancient city of Taxila (N-Pakistan) and consist out of a sword from a 1st c AD Parthian context, and a second sword and adze fragment probably dating to the 5th c AD.[117]

The only other early example to be recognised so far is a double-edge, Sasanian sword with a silver scabbard, dated to the 6th – 9th c AD, from the Dailaman region NW-Iran. The metallographic conclusion is that the purity of the metal and the small quantity of slag inclusions indicate an efficient steel making process. The dendritic macrostructure is typical of a metal that has solidified from the molten state and can be seen in ingots of crucible steel.[118]

8.4.2.6. Conclusion

It is very clear that the routes taken to achieve usable crucible steel in the Far East, Central Asia and the Indian subcontinent, and later in Europe (not discussed here because of the very late introduction, 19th c AD), were very different. The extent of interchange of technology, if any, between the three major zones is still unclear.[119]

Within the two basic methods of making crucible steel – *in situ* carburisation and co-fusion – there is a very wide range of reported procedures. Although crucible steel has always been seen as a typical product of southern India, in fact production was widely spread throughout the Middle East and Central Asia as well as in India.[120] There is however a fundamental difference in the materials and techniques used to produce crucible steel in C-Asia with those used in South India/Sri Lanka (e.g. the shapes and materials of the crucibles and the cooling rate of the ingots). The crucible steel in C-Asia was clearly not exclusively an Indian import. This does however not mean that mutual trade links did not exist.[121]

[114] Rehren & Papakhristu, 2000: 65; Feuerbach, 2002: 148.
[115] Feuerbach, 2002: 154.
[116] Feuerbach, 2002: 229.
[117] Feuerbach, 2002: 229-230.
[118] Lang, Craddock & Simpson, 1998: 10; Anantharamu, Craddock, Nagesh Rao, Murthy & Wayman, 1999: 13, this artefact was obtained through the art-market and the exact archaeological context is not known.
[119] Craddock, 2003: 231.
[120] Lang, Craddock & Simpson, 1998: 7.
[121] Feuerbach, 2002 : 252.

There is no doubt that India was exporting good quality iron or even steel from an early period onwards, as evidenced by the *Periplus*. But the question remains of this iron was a crucible steel and if the ingots much later known under the general term *wootz*-steel, were already produced in this early period. As far as we can see, there is no indication that crucible steel was exported on a considerable scale during the 1st and 2nd c AD from India.

The initial origin of the crucible steel technique probably lies decades earlier than the first few centuries AD, since it would have taken considerable time to improve and refine a technique as complex as crucible steel production.[122]

This discussion is not relevant for the iron objects found at ed-Dur, since none of them has its microstructure preserved. This overview is important to fully understand the implication of the suggestion by A. Ploquin, S. Orzechowski and B. Briand[123] that the slag found at Mleiha and ed-Dur could be related to a crucible process. If this were so, then this would be one of the earliest crucible steel processes, in a region that previously had no or very little experience with the production of iron. This will be further expanded in the chapter on the slag.

[122] Feuerbach, 2002: 259.
[123] Ploquin & Orzechowski, 1994: 29-30; Ploquin, Orzechowski & Briand, 1999: 180.

8.5. Sample description, analytical results & conclusions

"*Iron artefacts that are found in conditions of damp with a good supply of oxygen are very often only iron oxide in the shape of the original artefact, with at times a small core of iron left at the centre of the object. Others may be completely oxidized and all that is available for inspection is the shape of the artefact, and other than being a ferrous metal, little can be determined concerning its original composition*[124]."

To start the analytical work a visual selection was made of all the samples brought back from ed-Dur to Belgium for further research. An emphasis was put on the recognisably of the fragment, since any information retrieved that cannot be linked to an artefact might be interesting but brings no deeper understanding. The second selection criterion was the solidity of the fragments, although all were corroded some seemed to be in better shape than other. The poor oxidation resistance of iron and steel is a serious drawback in studying iron artefacts. The corrosion of iron is severely accelerated by moist conditions, especially in the presence of salt[125], the perfect description of the subsoil of ed-Dur. Nevertheless it was thought that with the more solid samples the chance was bigger that some un-corroded metal was left in the core.

The selected samples were cut with a water-cooled abrasive cut-off wheel with a thickness of 0,8 mm. Afterwards they were mounted in bakelite and polished according normal metallography standards (see *Chapter 4*).

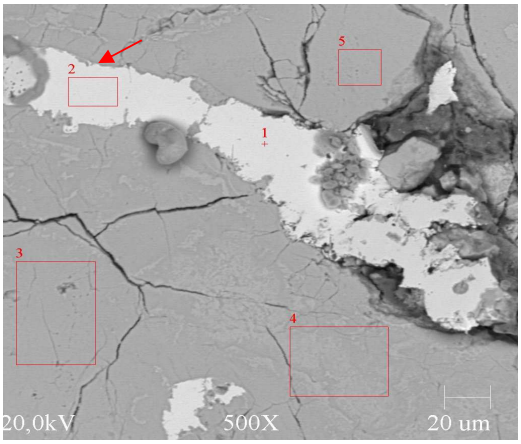


Fig. 114: SEM-picture of iron inclusion.

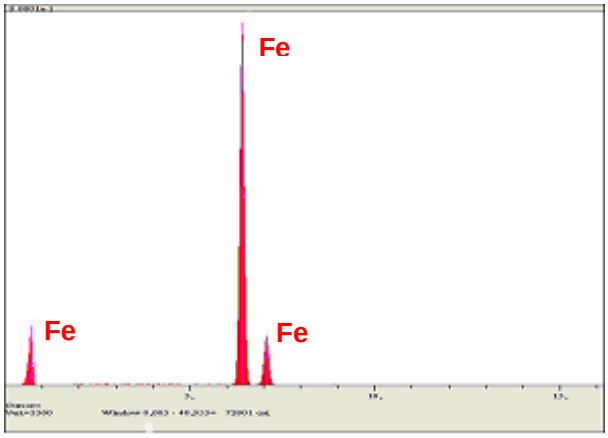


Fig. 115: EDX-spectrum of Point 2.

Under the optical microscope and SEM the majority of the polished samples showed some small white inclusions (the largest some 100 µm long and some 20 µm wide, see for example Fig. 114). SEM-EDX analyses confirmed the suspicion that they were ferritic grains that had survived the corrosion process. On the EDX-spectrum (FIG. 115) no oxygen peak appears, proving them to be un-oxidized. The next step was to determine if these islands were only ferritic grains or if there was any indication of a more complex microstructure, e.g. pearlite. The presence of carbon, which would be materialized in the form of cementite, would point to steel. R. Knox analysed an iron dagger from Hasanlu (Iran, dating to ca. 800 BC) and was able to show that the original object was steel because of the presence of nodular carbides in both the metal particles and the oxide, and of pearlite in the metal particles[126].

[124] Sim, 1998:3.
[125] Unglik, 1991: 106.
[126] Knox, 1963: 45.

To reveal this microstructure the samples needed to be etched. Etching is a process where the surface is attacked (chemically, mechanical, electrolytic, etc.) to create a relief at the surface that shows the microstructure of the metal. The basic principle is that harder parts are attacked less than the softer parts. Under the metallographic optical microscope this relief is reflected and can be interpreted. Following the analyses of R. Knox mentioned above the samples were etched with a 2% Nital solution for different time periods, starting with 10, than 20 and eventually 30 seconds. No reaction whatsoever was seen. In a second attempt a 4% Nital solution was tried for 10 and 20 seconds. But also here no reaction could be observed. A third etchant, *Stead* reagens[127] had a very different effect, the islands could not be resolved anymore under the optical microscope. This could mean 2 things, or they were so aggressively attacked by the etchant that they were dissolved or fell out of the oxidised matrix; or that the etchant changed the colour of the grains so much they could not be detected against the oxide background. The technician tried some more etchants, but none had any effect (see table 45).

Etchant	Time	Result
Nital 2%	10, 20, 30 sec.	No result
Nital 4%	10, 20 sec.	No result
Picral saturated	+ 1 min.	No result
Stead reagens	30 sec.	White inclusions gone

Table 45: Etchants used on the iron inclusions.

Despite the use of a wide array of etchants in an attempt to resolve some microstructure in these grains of iron, no result was obtained. This could mean that these inclusions were the remains of a single grain, so no boundaries can be resolved or that the effect of the etchant was neutralised by the oxide matrix.

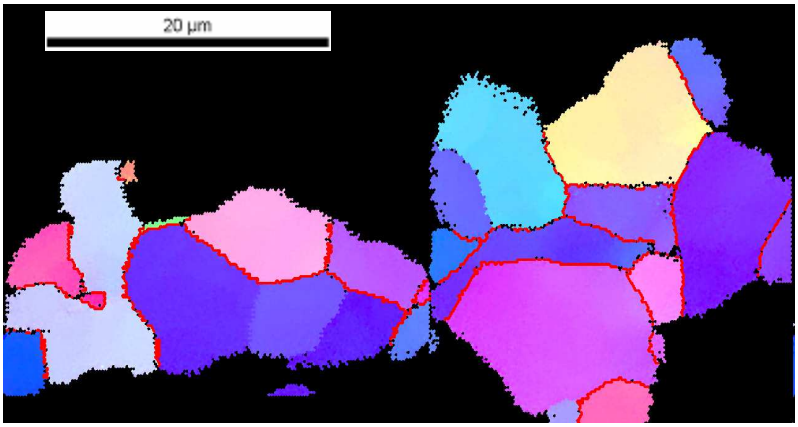


Fig. 116: OIM-scan of an iron inclusion.

To test this an OIM-scan was made of one of these inclusions[128]. This technique is of no further importance to this study, so only a brief description of it will be given. *Orientation Imaging Microscopy* (OIM) is the most complete solution available for analysing orientation and related aspects of crystalline microstructures and is based on the detection of diffraction of the electron beam on the crystallographic planes of the specimen. These diffractions produces a pattern composed of intersecting bands, termed electron backscatter patterns and can be imaged by placing a suitable film or phosphor screen in close proximity to the sample. The result is an image with different coloured section, where the same or adjacent colours indicate the same or similar oriented planes. In the case of Fig. 116 the result is 26

[127] "Iets dat altijd goed is tegen een hoestje" quote E. De Temmerman.
[128] With thanks to dr. ir. P. Rodriguez Calvillo, for suggesting this test and performing the analysis.

grains of α-iron[129] with an average confidence index of 0,70. This analysis shows that these inclusions are multi-grained, but the boundaries cannot be made visible by etching. The overwhelming oxide-matrix 'absorbed' the etchant, leaving the island untouched. In the case of the Stead reagens the etchant was to strong, and the iron inclusions fell out of their oxide matrix. α-Iron points to the fact that the lattice of the iron at least offers the possible for carbon atoms to be present in the iron matrix. SEM-EDX analyses did not show a carbon peak however, but this is also not surprising due to the corroded nature of the iron.

A second method to collect information was not the study of the un-corroded iron inclusions, but that of the corrosion products themselves. The line of reasoning is that the corrosion products might preserve relics of the original structure. These *ghost-structures* have been studied by several scientists, and have produced some interesting results[130]. Considerable time was spent on looking for these remnant structures under the optical microscope, but either due to inexperience or absence, no relevant structures were found.

To conclude we can say that despite the time and effort spent on the iron samples, no usable information could be deducted from them. The preserved iron grains on the one hand were too small and to few to be etched and therefore it was impossible to observe their microstructure under the optical microscope. The corrosion products on the other hand had not preserved the remnants of the original microstructure as was hoped. After a talk with and on advise of Th. Rehren, Professor for *Archaeological Materials and Technologies* at the *Archaeological Institute – University College London*, it was decided to leave the metallurgical analyses of the iron for what it was and focus on the other materials covered in this PhD.

[129] This is iron in its BCC (body-centred cubic) state. The atoms are closely packed together and the internal spaces in-between these atoms is too small to allow the entry of any carbon atoms (Congdon, 1971: 18).
[130] Some examples can be found in Agrawal, Narain & Prakash, 1991; Knox, 1963 and Notis, 2002.

8.6. Iron artefacts prior to the late pre-Islamic period

The question of the introduction of iron in SE-Arabia is an interesting one. It is noteworthy that although the period spanning 1300-300 BC is called the 'Iron Age', this metal was very little used and spread. This terminology was taken from the chronology of the Iranian Plateau, and introduced in the U.A.E. after the Iranian revolution when archaeological missions within Iran became impossible. The archaeologists previously working in Iran, spread out, some of them starting excavations on the Arabian side of the Gulf, taking with them their chronologies and the for them much better known Iranian background of the material culture.[131] Iron artefacts appeared on a more widespread base in the Oman Peninsula only at the end of the 3rd c BC as shown by excavations on the site of Mleiha. This is centuries after its use had become common in neighbouring regions[132].

This does however not mean that iron was entirely unknown, but that it was an exotic product that occurred only in exceptional circumstances. The list of iron object dating to the Iron Age is short and can be summarized as followed[133]:

- A ring fragment was found in the more recent levels of Rumeilah (Emirate of Al Ain), dating to the Iron Age III.
- An iron point fitted in a bone handle (an awl) is known from the graves of al-Qusais (Emirate of Dubai), but this stays largely unpublished.
- Two fragmented iron arrowheads and hook was excavated by C.Phillips at Wa'ab 4 (situated in Wadi al-Qawr, Emirate of Ras al-Khaimah)[134]. The exact stratigraphic position is not published but the occurrence of glazed pottery could point to a later date. Apparently during a later campaign at Fashgah (located in Wadi al-Qawr) more iron objects were uncovered in a tomb[135].
- Two fragments were encountered at Bithnah (Emirate of Fujairah), an arrowhead or a burin point[136]. These are possibly intrusive fragments, seen the fact that a "Hellenistic" interment was present.
- At Muweilah (Emirate of Sharjah) further evidence for the presence of iron in the Iron Age II period is provided by the find of 19 iron artefacts, of which one dagger is published[137]. These represent the only iron found in the SE-Arabian that incontestably date to the Iron Age II period. P. Magee states that iron assumes an important role as a symbol of status and power when it first appears elsewhere in the Near East. Its rarity in SE- Arabia suggests a similar role in that region. The lack of any evidence for local production suggests that it was probably imported from elsewhere. W-Iran appears as a likely source and some of the artefacts have parallels in this region. The iron objects at Muweilah were found in Building II. This columned building is associated with the local power.[138]
- Shimal Site 2 (Emirate of Ras al-Khaimah) yielded four corroded fragment of a knife blade in a collective tomb dating to the first quarter of the 1st millennium BC. The blade possibly was one-sided, but if there are traces of a mid-rib then two-sided.[139]
- At al-Thuqaibah (Emirate of Sahrjah) an iron slag and one shapeless fragment of iron is reported, dated to the 8th – 4th c BC.[140]

The exact reason for the late introduction of iron in this region must have different reasons since iron containing deposits do occur in the Hajjar mountains of Oman. These deposits are often associated with those of copper, as an *iron hat* above the copper-rich deposits. It needs

[131] Ploquin & Orzechowski, 1994: 25; Magee, 1998: 112.
[132] Lombard, 1989: 26 ; Ploquin & Orzechowski, 1994: 25.
[133] Lombard, 1984: 255; Magee, 1998: 112.
[134] Phillips, 1987: 15 & Fig. 38.
[135] Lombard, 1989: 32, footnote 16.
[136] Corboud, Castella, Hapka & im-Obersteg, 1996: 77.
[137] Magee, 1998: 113; since then some more iron daggers were excavated (Magee, pers.comm.).
[138] Magee, 1998: 113; Magee, 2002: 166.
[139] Donaldson, 1984: 257-258.
[140] Millet, 1997: 37.

to be mentioned that these iron deposits are basically hydrated oxides (limonite), which is not the best ore to be used for direct reduction. These ores were more often used as a colorant (ochre) or as a flux in the copper smelting process. Iron ores present in the form of chalcopyrite, need a good pre-smelting treatment (roasting) to remove the sulphides and are only exploited much later. The copper smelters of the 1st millennium must have known iron, since it is often used as a flux.[141] This is reinforcing by analyses on copper-base alloy artefacts from Tell Abraq by L. Weeks, where iron occurs in quantities between 0,5 and 1%. This can also be linked to the incomplete purification of iron containing copper ores.[142]

The wide spread use of bronze from a very early period on in SE-Arabia and the abundant copper sources close by could have slowed the introduction of a new metal, i.e. iron. Bronze objects did their job and no other material was needed. Competition to acquire a good that is used by a neighbouring group of people is often a powerful motor to spread new technology. But if no one used iron this effect is lost.[143] It should also not be forget that iron originally was not such an attractive metal to use. It oxidises easily, the production is much more labour/energy intensive, every object had to be made individually, technically it is more difficult to process, in the beginning it was not harder than the bronzes, etc. Without dramatic stress (e.g. shortage of tin, copper, etc.) the step towards the use of iron might be taken.[144]

Why some centuries later the step was taken is not clear, since there are no sites that have a stratigraphy spanning this transitional period. The youngest Iron Age site (Rumeilah, *ca.* 450 BC) where copper-base alloys are exclusively used is still separated by a gap of two centuries from the earliest widespread use of iron at Mleiha.[145] P. Lombard suggests several factors that may have contributed to the transition: a shortage in the supply of tin, shortage of wood to produce copper and the arrival of the Seleucid power in the Gulf-region[146].

It is interesting to notice that the massive use of iron occurs roughly at the same time on the both sides of the Oman Mountains, e.g. Mleiha and the Samad culture. Both are located in very different areas: Mleiha not far from the shores of the Gulf and Samad in a rather remote country, hardly in contact with each other. The abundant and sudden use of iron and the large amounts deposited in the tombs could indicate a local production but no convincing evidence of iron smelting in the region was found so far.[147]

The iron objects from their part are not very helpful in solving the problem. The badly preserved state of most of the iron recovered at Mleiha and ed-Dur does not often permit stylistic comparisons with finds from other areas. As a result, it is difficult if not impossible to say in most cases, whether a particular object was imported or locally produced.[148]

[141] Lombard, 1989: 32-33.
[142] Weeks, 1997: 61-62.
[143] Lombard, 1984: 233.
[144] Lombard, 1989: 34-35.
[145] Lombard, 1989: 36; Boucharlat, 1991: 289.
[146] Lombard, 1989: 36.
[147] Boucharlat, 1991: 297.
[148] Potts, 1990b: 274-275.

Chapter 9. IRON SLAG

"Making me(n)tal connections is our most crucial learning tool, the essence of human intelligence; to forge links; to go beyond the given; to see patterns, relationships, context."

M. Ferguson (brackets added)

9.1. Introduction

Slag is a term used to indicate the silicate complexes formed by combination of the gangue from the ore (mainly silicate complexes), with some of the iron oxide in the charge acting as a flux. The majority of slags produced in ancient times are therefore iron silicates. High temperature processes determine the structure of slag and their chemical composition makes them very resistant against environmental weathering factors.[1] Despite their good preservation over time, it is often not easy to pinpoint at which point of the production process the slag originated, especially if no additional archaeological context is known.

Many pyro-technologies form slag as a waste product, but here we will only consider the by-product of iron working. Slag is produced at different stages during the *châine d'opération* of iron (see *Chapter 8*). They can be divided in three large groups: *smelting slag* (i.e. bloomery slag), *primary smithing slag* or *refining slag* formed by purifying the bloom to wrought iron and *secondary smithing slag* formed during the forging or repairing of an object. A fourth kind of slag important in this discussion is *crucible slag*, generated during a crucible processes as described in *Chapter 8*. Other waste products (e.g. furnaces, hearths, ore, etc.) can also be indicative for pyro-metallurgical processes, but are more easily overlooked than slag during fieldwork. The main waste products from the smelting phase (of no importance here) are: *tap* slag, excess slag that is tapped from the furnace, *furnace bottoms* and *slag pits*.[2]

The slag types of the successive phases can tell us different things, smelting slag being the most informative. Chemical analyses can give an idea of the bulk composition of the slags, e.g. is the acidic slag (e.g. the presence of SiO_2, Al_2O_3, TiO_2, etc.) or basic (e.g. the presence FeO, MnO, CaO, MgO, etc). It can also give an indication towards the efficiency of the process and give an idea how much metal was lost. Determination of the crystalline phases formed says something on the solidification temperatures of the originally liquid bulk of slag and provides information on the reducing conditions. The microstructure and the non-metallic phases and metallic inclusions give additional information on the structure of the slag (segregation, which impurities are present, etc).[3]

In an ideal situation with enough circumstantial evidence one would think that it is easy to distinguish between all these types of slag, but the reality is often different. Certain slags tend to be rather un-characteristic in appearance and composition. So the use of slag morphology to identify the metal-working processes involved can be extremely difficult. A bulk chemical composition of the slags is also often off little help in that smelting, welding and smithing can all produce slags with very similar composition. Copper and iron-working slags can be very difficult to distinguish, although chemical analysis should be able to separate them because of enhanced copper concentrations in the former. The study of slag therefore is a complicated and specialized issue. For that reason they will be treated in a rather general way. A wide array of techniques is needed, from visual characterization over optical, petrographical and electron microscopy till EDX, XRD, etc. to study slag. But even with all this morphological, chemical, mineralogical and physical data it is not always possible to distinguish between the different types of slag, if no additional evidence is present. Moreover the fact that the slags dealt with here most likely are iron smithing slags, the gain in information will be limited since they stand at the end of the production chain. Information potentially held in smelting (or even primary smithing slag) on the relation to the ore source and the efficiency of the reduction process are completely absent or obscured in secondary smithing slag.

[1] Tylecote, 1962: 183; Morton & Wingrove, 1969a: 1156; Ortiz, 2003: 24.
[2] Schrüfer-Kolb, 2004: 9-10.
[3] Tholander, 1989: 36; Pelet, 1993: 91.

This chapter starts with an introduction on the study of slags and the presentation of the reference material found in the region, after which the results of the samples and their macroscopic observations are given. A selection of slags is made and looked at in more detail under a normal metallographic optical microscope. The microstructures seen are further analysed on their chemical elemental composition by SEM-EDX. Prof. dr. M. Elburg studied two samples on their petrographical features. Finally an attempt is made to determine the crystalline minerals present by powder-XRD. An excurse is made to some specific ceramic remains that might be related to metalworking and the chapter is rounded-off by a general conclusion of the obtained results.

9.1.1. Smelting slag

I will only briefly touch upon the characteristics of smelting slag (see also *Chapter 8*) since to my opinion none were found at ed-Dur, but a basic introduction will help to discuss the difference with smithing slags.

Two large groups can be distinguished, *furnace bottoms* and *tap slag*. In non-slag-tapping furnaces the slag forms as a dense lump at the bottom of the furnace. If the bottom is slightly concave to collect the slag, as in the case of the bowl or advanced bowl furnace, the slag has a characteristic plano-convex shape and is very dense. This is called a *furnace bottom*. They can also have a plate form, if the bottom of the furnace is level. There are also very dense slags that display fuel imprints, charcoal or ash remains, which indicate the slag has cooled inside the furnace. These can be labelled under the general term of *furnace slag*. A more advanced form of a non-tapping furnace is the slag pit furnace. In this case the slag is collected in a pit dug below the furnace. Depending on the shape of the pit different shapes and sizes of *slag blocks* develop.[4]

The second group are slags that were tapped from the furnace while smelting. The reason was to prevent that the accumulating slag would block the tuyere, stopping air supply and eventually the reduction process. Tap slag has a smooth, sometimes with a ropy surface that shows the flow of the fluid slag, and a rather irregular bottom with impressions of the soil. In section it is very dense. Sometimes the upper surface shows reddish coating, which is the result of re-oxidation processes during cooling. Frequently large vacuoles are present because of the gases trapped in the slag.[5]

To free the iron from its ore it is necessary to physically separate it from the gangue. The gangue is made up by components of oxygen, silica, aluminium and calcium in particular, as well as small amounts of other elements (Mg, Mn, P, etc.). The individual minerals, such as quartz and alumina, have very high melting points (over 1700°C), but the presence of iron oxide acts as a flux, lowers the melting point. At about 1150°C *wüstite* (iron monoxide, FeO) reacts with silica and forms *fayalite* (Fe_2SiO_4), one of the dominant phases in solidified slag. Next to this, other complex glassy silicate minerals can be present, as well as iron oxides or even droplets of metallic iron. During the smelting process the slag also reacted with the ash from the charcoal introducing Na and K that again reduce the melting point. The reaction of the clay lining of the furnace contributing silicates and CaO to the slag. The final bulk composition of a bloomery smelting slag reflects not only that of the original ore, but also that of the other contributors.[6]

Slags produced in the early solid state smelting processes typically contained 50% or more iron. This means that iron could only be produced from high-grade ores, since part of the iron was (unintentionally) sacrificed to generate the low melting point free-flowing fayalite. The most fluid slag has a composition of 80% of FeO and 20% of SiO_2. Since these high-grade

[4] Salter, 1987: 201; Schrüfer-Kolb, 2004: 10.
[5] Schrüfer-Kolb, 2004: 10.
[6] Bachmann, 1982: 10.

ores have too little silica present to make the slag liquid, it had to be added. The intentional use of fluxes (e.g. sand, limestone, etc.) is not attested with certitude until the European Middle Ages, but the digestion of the minerals present in the furnace wall solved that shortage.[7]

Iron production on any scale would produce large amounts of slag present in noticeable concentrations or slag heaps[8].

9.1.2. Primary smithing slag (bloomsmithing)

The primary product of the smelting process, the raw bloom has to be reheated to welding heat (around 1200°C), and hammered to fuse the iron particles together and to squeeze out excess slag. This stage of smithing is very slow and requires great patience. The entrapped slag makes it difficult to heat the bloom completely and only a few hammer blows can be given at each re-heat, otherwise the bloom would fall apart. Initially the slag liquates from the bloom leaving small voids in the iron, which are closed by the hammering. If the bloom is left too long in the hearth at this stage, the slag can liquate out and the unconsolidated iron can literally fall apart and be lost to the hearth.[9] During this process of refining, part of the slag ended up at the base of the smithing hearth used for reheating and part was scattered around the anvil/workspace as small particles. A small portion stayed in the wrought iron as *slag stringer*. The slag cake collecting at the base of the hearth was for the majority made up of the remains of smelting slag that was still present in the bloom and is therefore difficult to distinguish on mineralogical and/or chemical bases, especially if any archaeological context is missing. The appearance of smelting type and smithing type slags in the same context can be indicative of primary smithing, as bloom refining is a mixture of both processes. The smithing of the bloom also squeezed out small particles of iron, encapsulated in slag.[10]

M. Leroy suggests a general chemical composition of primary smithing slag. The CaO levels can be 7 to 25%, less Al_2O_3 (< 5%) is present than in smelting slag, the silica component is more or less the same as other slag (in-between 15 and 30%) and the fraction of iron oxides is higher than in smelting slags (up to 75% or even more). This is only a general indication and exceptions do occur regularly.[11] V. Serneels adds that elements such as Mn, V, Cr etc. can be present in elevated levels in bloomsmithing slag and also chemical elements related to the ashes of the charcoal (Ca and K) contributes to the general composition of this slag[12].

9.1.3. Secondary smithing slags & other waste materials (forging)

Secondary smithing slag is produced during the final phase of iron working, being the production or reparation of an object. During the smithing process different kinds of waste are generated. In the hearth, *smithing slag* is formed by the melting of various materials accumulated just under the tuyere or at the base of the hearth. A different kind of slag comes into existence due to the reaction of the charcoal with the hearth lining and forms *fuel ash slags*. On the surface of the worked object itself an oxidised iron skin is formed. This *hammer scale* is scattered around the anvil and can fall into the smithing hearth. A last group of debris are *metallic fragments* that are lost or cut off by the blacksmith during the forging of the artefact.[13]

In general already refined iron was traded to the blacksmith and the making of artefacts was carried out at a place other then the primary production site. This could be anything from a specialised workshop to a small temporary forge. The amount of slag produced is much less

[7] Tylecote, 1962: 188; Rostoker, Bronson & Dvorak, 1989: 14; Scott, 1990: 155; Sim & Ridge, 2002: 43.
[8] Greenough, 1987: 27.
[9] Crew & Salter, 1991:18-19.
[10] Bachmann, 1982: 31; Scott, 1990: 156-157.
[11] Leroy 1997: 185.
[12] Serneels, 1993: 49 & 239.
[13] Scott, 1990: 156-157; Serneels & Perret, 2003: 469.

then during the previous stages. Any site with only a small quantity of slag, if the sample is representative of course, and with no real slag concentration is very likely to be a secondary smithing site.[14]

• **Hammer scale**
Hammer scales are the remains of the oxide skin that is formed during forging. When the iron is shaped in an object it has to go through repeated cycles of heating and hammering. To forge iron together a temperature of 1250°C is need ed. Every time the iron is heated an oxide layer forms on the surface. This layer breaks or flacks off when the iron is hammered or because of 'thermal expansion'. Part of it falls in the forge and is one of the components that contribute to the formation of smithing slag and part accumulates around the anvil. It is fairly pure oxidised iron and a characteristic feature of scale is that it is strongly magnetic. The hammer scale comes of in thin flakes and is very susceptible to corrosion, resulting in a poor preservation. Sometimes only a fine rusty reddish-brown powder remains and it is easily overlooked during excavations, if not specifically looked for (e.g. by dragging a magnet over the ground). Next to the scale form, small spherical droplets of liquid slag can be expelled from the metal when hammered, forming a second residual product typical of smithing activities. The droplets can also be of the same composition of the scales and highly magnetic. They both are a strong indication of the former presence of a forge.[15]

To prevent or limit the loss of iron to hammer scale a flux is applied to the surface. Additionally the elimination of any oxide layer on the surface of iron that has to be welded is crucial if the weld is to be successful. Sand (or clay) was added to the surface forming a liquid slag film, with a relative low melting point, protecting the surface from oxidation. This liquid *fayalitic* film also creates a metastable layer that helps the fusion of the parts and acts as a kind of 'glue'[16]. Upon the impact of the hammer this thin layer of slag is expelled. When the blows are either too hard or to soft the slag is trapped within the weld, creating a weak or incomplete fusion. Exerting the right pressure is the part of the crucial skills of the blacksmith, next to the recognition of the right temperatures based on the colour of the heated metal. In the finally phase, after the object was completed, sand was used again to minimize the oxidation of the iron if additional heat treatments were to be preformed (e.g. *tempering*). When steel had to be worked a different reason to protect the surface played, i.e. to minimize the decarburisation of the steel.[17]

• **Fuel ash slag and cinders**
Fuel ash slag is a highly siliceous, low-density fused ash. It results from the reaction of the inorganic fractions in the fuel and its ashes[18], and other siliceous or calcareous material from the surrounding hearth lining or soil, under oxidising conditions and at high temperature[19]. This type of slag can form during any high temperature process and their chemical composition suggests that they would only become molten at the sort of temperatures that would be found near the tuyere (1400-1500°C) [20]. A second intermediary waste product is called a *cinder*. They are a conglomerate of hearth material that is not completely fused, containing mainly fuel ash, hearth lining and/or perhaps welding sand. These are easily recognised by their light, crumbly nature and brownish colour. Cinder can also form in a smelting furnace in connection with unreduced ore and fuel, called *bear*.[21]

[14] Bachmann, 1982: 5; Crew, 1996.
[15] Bachmann, 1982: 31; McDonnell, 1984: 48; McDonnell, 1988: 286; Crew & Salter, 1991:18-19; Schrüfer-Kolb, 2004: 11.
[16] Pers. comm. Van Nie.
[17] Fluzin, Ploquin & Dabosi, 2004: 169.
[18] According to V. Serneels and S. Perret (2003: 472) charcoal can contain a mineral fraction between 0,5 and 5%, mainly lime and potash (respectively CaO and K_2O).
[19] McDonnell, 1984: 48.
[20] Salter, 1987: 197.
[21] Bachmann, 1982: 30; Schrüfer-Kolb, 2004: 11.

• **Smithing slag**

Smithing slag is believed to have formed under oxidising conditions. Quite often the iron oxides, generally identified as wüstite, in the slag are re-oxidised to a higher form of iron oxide called magnetite (Fe_3O_4). This may be due to post depositional oxidation[22]. Many different components contribute to the formation of smithing slag: hammer scale, small pieces of metallic iron, inclusions of silica sand used as flux, hearth lining that mainly contained Si and minor amounts of Al, K and Ca, charcoal (ashes - Ca, Na and K) and some smelting slag still present in the wrought iron. The amount depends on the thoroughness of refining. All of this fell into the base of the hearth and formed either randomly shaped pieces of slag termed *smithing slag lumps* (SSL) or the more diagnostic/characteristic *smithing hearth bottom* (SHB). The latter is also sometimes called a *plano-convex bottoms* (PCB) because of the plano-convex or concave-convex shape. It is assumed that the convex surface was the bottom side of the PCB and the flat surface formed the upper surface, often with a central depression due to the air blast. The slag does not actually fall to the bottom of the hearth, but solidifies in the cooler zone under the tuyere. Depending on the depth of the hearth they can touch the bottom or not. Smithing slag lumps can be considered as irregularly shaped, partly formed hearth bottoms that have not been incorporated into the slag cake. They can also result from the hearth being cleaned when hot, so that the pasty slag shape became distorted. When solidified in the hearth, imprints or pieces of the charcoal or hearth material can regularly be observed. Smithing slag can vary considerably in size from about 5 cm in diameter, and a few cm in depth, up to *ca*. 20 cm in diameter and a maximum of 10 cm in depth. Their weight can also fluctuate considerably, from 100 g till 2 kg, but the majority seem to between 200-500 g.[23]

Three main types of smithing slags (*calottes* or *culots* in French) can be distinguish, based on their general composition. The distinction is not always clear and some slags show characteristics of more than one of these types.[24]

1) SAS (*scorie argilo-sableuse*). Slag principally made up from clay and/or sand that is completely or partially fused, resulting in a matrix rich in silica and other minerals commonly found in granitic rocks. Minerals of the same chemical composition also make up sandstone. They often show relictic grains of quartz-veldspate or fragments of baked clay integrated. In general the amount of sodium is reduced in relation with the potassium and calcium content. Magnesium amounts are very low, as is the iron content. Sometimes dendrites of magnetite can be observed though. These originated from the hammer scale that went through a liquid phase. The shape can be very irregular, with a size of a few mm till 10 cm and some slags have the typical PCB-shape. The colour range is large going from black, brown, beige over areas with even bright colours like blue and green. They have a vitrified aspect, are porous and have a low specific gravity. Because of the very heterogeneous appearance they are easily mistaken for fragments of the hearth lining. SAS are formed when the smith adds a large amount of flux to the surface of the metal for welding or steel working, and it is sometimes present as a separate phase on top surface of a slag.

2) SGD (*scorie grise dense*). Grey dense slags are the result from the cooling of liquid silica, rich in iron oxide, making up an assembly of fayalite with a variable amount of iron oxide (mainly plated magnetite or/and wüstite) and a small amount of interstitial glass. Sometimes it is possible to identify rounded or elongated ghost structures formed by iron oxide minerals that probably are the not completely dissolved fragments of hammer scale crust. Iron is also frequently present in metallic form, but the distribution is irregular. The slags are generally fragmented, but some have a

[22] McDonnell, 1984: 48.
[23] Greenough, 1987: 27; McDonnell, 1988: 286; McDonnell, 1991: 23 -24; Salter, 1994: 383; Crew, 1996; Mihok & Pribulova, 2003: 163; Serneels & Perret, 2003: 471-472; Schrüfer-Kolb, 2004: 10.
[24] Dunikowski, Leroy, Merluzzo & Ploquin, 1998: 147-149; Serneels & Perret, 2003: 475-476; Serneels, Merluzzo & Leroy, 2004: 101-102.

clear 'bowl-shape'. Their size can differ from 5 to 30 cm, although the majority is between 1 to 10 cm and their weight can vary from 100 g to as much as 2 kg (or even 5 kg). They are often slightly elliptic of shape. The lower surface can be convex and can have impressions of the material it was formed on (e.g. charcoal, small stones, hearth base or baked earth). The upper surface is flat, convex or concave. The internal structure is variable and often includes particles of sand, clay or incompletely fused pieces of rock. The slag can be divided in zones from porous to dense or have a more homogeneous composition. The fracture is metallic-grey. The upper surface will be smooth and dark-grey, eventually sometimes even wine-coloured. The bottom will be more porous with a grey-rusty colour. This type of slag is very similar to the slag produced by smelting or primary refining operations, but is mainly formed by hot oxidation of the metal with a small input of silica from various sources. They are mainly generated during the heating of iron for hot forging.

3) SFR (*scorie ferreuse rouillée*) are slags richer in iron oxides or even metallic iron. They often have earthy or rusty colours and are magnetic. Most have no specific form, but some are kidney or bowl-shape. A fresh brake normally has a dull grey colour and frequently includes fragments of charcoal. The matrix is fayalitic and rich in wüstite. The porosity is very variable, irregular and spread, but normally a slag rich in SFR is dense. The metallic particles mainly originate from the loss of metallic particles during the work of a poorly compacted metal. It can also occur when the smith is working at temperature close to the melting point of the metal, for example welding operations.

The smithing slags stand on the end of the process and have no or very little chemical traces left that can link them to the minerals originally used, except possibly some *siderophile elements*. The chemical composition of the charcoal might actually even dominate the composition.[25] *Siderophile elements* are chemical elements that are close to iron in the periodic table (i.e. transition metals) that exhibit metallic bonding. They do not easily combine with oxygen and sulphur i.e. P, Co, Mo, Ni, Ru, Rh, Pd, Re, As, Ir, Pt and Au. They prefer to go into the iron and not into the slag. *Lithophile elements* on the contrary combine easily with oxygen and are present in many silicates. These elements preferentially go into the slag, e.g. Mg, Al, Si, K, Ca, Cu, Ti, V, Cr, Mn, Y, Zr, etc.[26]

- **Smithing hearth**

The smithing hearth can take many forms, from a simple pit in the ground with a diameter from 25 to 100 cm and a depth of 10 to 30 cm would suffice. Only a small wall to protect the bellows might be needed[27]. More permanent forges were raised to working height. The simple hearth is difficult to identify archaeologically, since they do not differ from other domestic fireplaces. This is the reason why smithing hearths do not show up as widely in the archaeological record as might be expected, and even when identified they appeared to have been multifunctional[28]. Blacksmiths in small communities did not limit their work to only iron often they also repaired all kinds of metal object. This sometimes results in e.g. small fragments of copper-base alloys intermixed in the hearth and being trapped in a typical smithing slag. These inclusions are strong circumstantial evidence that smithing activities were undertaken.[29]

The lining of the hearth is mostly made of a clay fabric. The lining that remains is normally heavily burnt and vitrified and may have suffered from the slag attack. Because of the

[25] Dunikowski, Leroy, Merluzzo & Ploquin, 1998: 145.
[26] Serneels, 1993: 49.
[27] Serneels, 1998: 29.
[28] Schrüfer-Kolb, 2004: 33.
[29] Schrüfer-Kolb, 2004: 31.

reaction between the slag and the lining elements such as silicon, calcium, aluminia, etc. can enter into the smithing slag.[30]

If a blacksmith was making artefacts it was in his best interest to work in a clean hearth. In this way the process could be controlled in the best way (e.g. temperature estimation based on the colour of the metal, preventing pollution, etc.). To keep the hearth clean, regular cleaning out of the slag is important. A normal PCB is formed between the lighting and the extinguishing of the fire. PCB's are produced during one unit of work. Although it is not possible to demonstrate the length of such a session (anthropological and modern observations would suggest a maximum of three to four hours), it would be logically that it was a one-day operation at the most.[31]

An important side remark can be made considering the amount of smithing slag produced. If the temperature of the forge is good there is little oxidation of the iron. Using charcoal of hardwood at high temperatures also reduces the amount of slags formed. Working a classical forge to shape iron produced little magnetic oxides and did not need the addition of deoxidising products. The presence of slag could thus be linked to the skills of the blacksmith and to specific activity, such as the working of bad quality iron, joining fragments, working steel, recycling activities, etc.[32]

- **Conclusion**

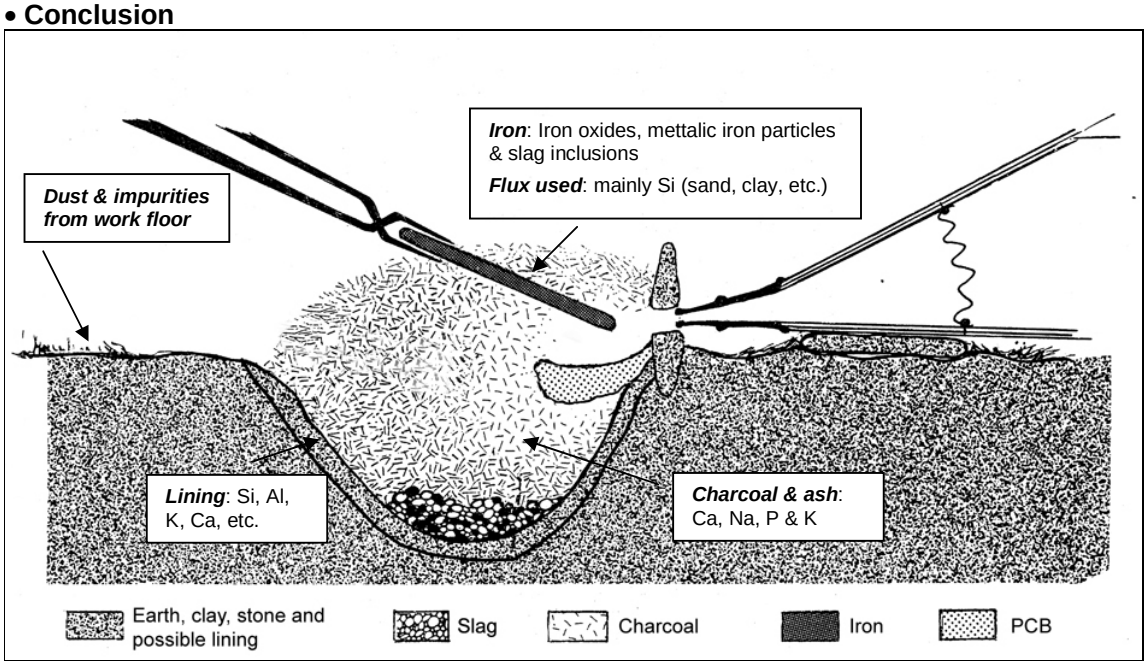


Fig. 117: Schematic representation of smithing hearth and the elements that can contribute to the formation of smithing slags (after Scott, 1990: 168. The hearth is rather deep, but this is a typical Irish feature).

Relatively little can be learned from smithing slag since they stand at the end of the *châine d'opération*. At first sight, it might seem that smithing slags should show a significantly higher degree of iron oxides than smelting slags. But this can only be taken as a broad generalisation, since we may expect a proportion of these slags to have ended up under reducing conditions at the base of the hearth. Another trademark often present is the appearance of magnetite coupled with high silica levels (Gallo-Roman slags have an average silica content of 23,5 till 38% SiO_2[33]). The rather fast cooling of the smithing slag

[30] McDonnell, 1984: 47-48.
[31] Serneels & Perret, 2003: 472; Fluzin, Ploquin & Dabosi, 2004: 167-168.
[32] Dunikowski, Leroy, Merluzzo & Ploquin, 1998: 150.
[33] Maréchal, 1982: 306-307.

creates mineralogical heterogeneity.[34] The chemical fingerprint is not likely too be linked to the original minerals used since the vast majority of the bulk material does not come from the gangue of the ores. Fig. 117 shows the possible contributors to the formation of a smithing slag. On this drawing the hearth is rather deep and the PCB is formed in the charcoal. If the hearth were shallower, what is perfectly possible, the PCB would form in contact with the base of the hearth. The contact region with the lining would be much larger, allowing the inclusion/reaction of more of the lining with the slag.

9.1.4. Crucible slags

The main problem with crucible slags is that few analyses have been published. The reason to include this type of slag is due to the fact that A. Ploquin, S. Orzechowski and B. Briand suggested that the slag found at Mleiha, in all aspects very similar or identical to the slag from ed-Dur, could be the remains of a crucible process[35].

Crucible slags are formed inside the crucibles used to produce crucible steel (see *Chapter 8*). They are made up by the impurities present in the (bloomery) iron in combination with the digested crucible wall, elements of the vegetal matter included as a carbon source and possible sand and/or crushed slag added to facilitate the formation of a liquid slag. Their exact formation process is still poorly understood, as is the whole crucible steel process for that matter. If this type of slag is separated from its original context it is often impossible to like them to their true origin, since similar slags can also form during other high temperature processes (e.g. glass making, etc.). An important characteristic of a crucible slag however that sets it apart from any other type of slag mentioned above is that it is formed on top of the (liquid) iron contained by the crucible and not beneath it. The fact that some of the Mleiha/ed-Dur slag has a typical PCB-shape (taking the shape of the 'bottom' of something) is difficult to explain within this. Crucible slags floated on top of the charge or are stuck to the crucible side at the height of the load.

Table 46 gives an overview of the few published compositional analyses encountered in the literature. GH-1 and GH-2 are from the 19th c AD site of Ghattihosahalli (S-India)[36]. The material of Merv (Turkmenistan)[37] dates to the 9th-10th c AD and the Ferghana[38] slag (Uzbeksistan) to the 9th-12th c AD.

Oxide wt%	**GH-1**	**GH-2**	**Merv**	**Ferghana** ICP-OAS	**Ferghana** SEM-EDX
SiO_2	58,2	58,8	45 - 53	-	60
Al_2O_3	14,2	14,6	13 – 18	3 – 15	20
Fe_2O_3	6,9	7,2	0,2 – 2,4	3 – 15	2
MgO	2,4	2,6	1,4 – 4,2	-	-
CaO	11,5	11,6	9 – 21	5 & 15	3
Na_2O	1,1	0,9	0,2	-	-
K_2O	4,5	4,7	ca. 2 – 6,4	-	1
TiO_2	0,8	0,7	0,5	-	-
MnO	0,2	0,2	0,1 – 4 & 9 – 17	15 – 20	15

Table 46: Comparative compositional data published on crucible slag.

[34] Scott, 1990: 157.
[35] Ploquin, Orzechowski & Briand, 1999: 179.
[36] Anantharamu, Craddock, Nagesh Rao, Murthy & Wayman, 1999: 18.
[37] Feuerbach, Merkel & Griffiths, 1997: 105-107; Feuerbach, Griffiths & Merkel, 2003: 261.
[38] Rehren & Papakhristu, 2000: 57-58.

The slag from Ghattihosahalli shows a close morphological resemblance to the much earlier crucible slag from Sri Lanka, east of the Knuckles Mountains dating to the middle of the 1st millennium AD. They all have a glassy appearance, with colours ranging from green to blue. Their iron content is lower than usually encountered in smelting or smithing slag.[39]

At Merv similar loose fragments of glassy shiny green to a darker, dull, slightly more bluish green slag was excavated mixed with crucible fragments in the refuse pits or attached to the remains of the interior crucible walls. No notable crystalline inclusions are seen. Some of the fragments appear to be of a homogeneous colour whereas others are not and display a wavy pattern. The slag mainly contains silica and considerable amounts of iron oxide (although less than in standard bloomery iron-smelting slag), alumina, lime, magnesia and potash. Na_2O, TiO_2 and MnO are present in smaller quantities, and there are traces of Cu, Zn, Ni, Co, Zr, Sb, As, Cr and V. Two groups of slag can be distinguished based on their Mn content, a high manganese group (9 – 17 wt%) and a low manganese group (0,1 – 4 wt%). In certain cases droplets of cast iron and steel were trapped in the slag and on the crucible walls, probably a result of small droplets being splashed upwards due to a 'carbon boil'. Alternatively they could have originated by the *in situ* carburisation of some residual bloomery iron[40]. These iron inclusions can appear as rusty spots on the surface. The bulk slag composition varies among samples and too few samples have yet been analysed to present a detailed discussion of the composition. The overall composition given by the authors is shown in Table 46.[41]

The Ferghana process generated more massive slag cakes adhering to the inside of the crucibles. They are typically 2 to 8 cm thick and situated 15 to 20 cm above the base of the vessel. Although the slag cakes appear to be solid, they are highly vesicular with on average half their volume being made of bubbles. They range in diameter from a few mm up to 2 cm, indicating the relatively high viscosity of the liquid slag throughout the process. The colour of the slag ranges from opaque grey and turquoise to bright blue and eventually translucent dark green. The mean bulk composition covers a rather wide range of oxide levels. The ICP-OES analyses show a CaO content that clusters around two groups, respectively *ca*. 5 wt% and 15 wt%. According to Th. Rehren and P. Papakhristu the analyses are hampered by the varying amounts of unreacted lime-rich stone fragments present and the frequent occurrence of ferrous metal prills trapped in the slag. The SEM-EDX analyses where therefore performed on inclusion-free areas of slag, resulting in a tighter compositional field.[42]

In general we can say that crucible slag are glassy in appearance, rather small and have a wide range of (bright) colours, going till greenish and bluish. They do contain iron, but generally far less than smelting or smithing slag. The iron is mainly present in its metallic form and has a high carbon content with cast iron prills regularly appearing. Purely based on the morphological appearance of the Mleiha/ed-Dur slag it is difficult to identify them as crucible slag, keeping in mind all difficulties to link morphology to origin. Neither the colours nor the shape seem to sustain such a classification. Also the amount of iron present in the material from Mleiha is much higher than would be expected for a crucible slag.

[39] Anantharamu, Craddock, Nagesh Rao, Murthy & Wayman, 1999: 18; Wayman & Juleff, 1999: 29.
[40] A carbon boil occurs during the formation of CO from the cast iron when reacting with oxides (Feuerbach, 2002: 125).
[41] Feuerbach, Merkel & Griffiths, 1997: 105-107; Anantharamu, Craddock, Nagesh Rao, Murthy & Wayman, 1999: 18; Feuerbach, 2002: 75-77; Feuerbach, Griffiths & Merkel, 2003: 261.
[42] Rehren & Papakhristu, 2000: 56-58.

9.2. Reference material in the Gulf-region

Not much iron slag was reported from the neighbourhood of ed-Dur. At Mleiha similar or identical slag was discovered and studied in detail. The results will be summarised below. Next to Mleiha some slag was encountered at Thaj and a few fragments at Qal'at al-Bahrain.

9.2.1. Thaj, Qal'at al-Bahrain & some surface finds

On a low dark mound (the "Iron Mound" or Tell al-Hadid) just south of the Thaj city wall the remains of substantial walling were noticed. A number of pieces of iron slag and a large amount of pottery was dispersed on the surface. Because of this it was hypothesised that the tell might have been the location of a metallurgical workshop. Excavations revealed a building oriented NNW, which measured *ca*. 7,5 x 13,5 m, with an extension measuring *ca*. 8 x 2,5 m in the middle of the eastern wall. The walls varied between 1,10-1,45 m, and consisted of an inner and outer skin of well-cut ashlars, with a rubble core. Several more fragments of iron slag were recovered both inside and outside the building but the initial thought that it might be a workshop area was not confirmed.[43] No details on the slag were published.

C. Salter published the slag fragments from Qal'at al-Bahrain (in Trench C level 5, a period IVd context: 400-300 BC) in greater detail[44]. They are described as having a microstructure typical of an iron smithing slag, with an inhomogeneous distribution of primary iron oxide in a matrix of olivine laths and alkali components in-between. The iron oxide occurred in dendritic form in those regions with moderate iron contents and in a semi-dendritic globular form in those regions with a high iron content (up to 50% of the cross-sectional area). The morphology suggested that the iron oxide was predominantly wüstite. The slag does show some unusual features however. Many of the olivine crystals were zoned, this was due to the presence of relatively high calcium (0,8%) and magnesium (0,4-0,5%) concentrations. In these regions with little or no free iron oxides the material between the olivine crystals has separated out into two distinct components, one definitely crystalline and the other probably glassy. These features together with the relatively large size of the olivine laths (over 100 µm wide) indicate that the slag was cooled from the liquid relatively slowly. Such features are unusual in smithing slags, which normally cool fairly rapidly. An additional unusual feature was the presence of small spherical iron sulphide particles that often contained small amounts of copper. Although this could indicate that the slag was the result of some form of small-scale copper refining process, no sign of such an activity was found. C. Salter concludes that the slag was most likely generated during a smithing process. The presence of calcium and magnesium could be from the clay used for the lining and gypsum (present in the soil of Bahrain) could be the origin of the sulphur. The copper could be an indication that copper was being worked in the same hearth.

One photograph of an "iron ingot" was published from the Islamic settlement on Bahrain[45]. Considering the picture, size and the extreme rarity of iron ingots to be found, this most probably is also a piece of smithing slag.

One piece of iron slag was reported without any further description from al-Thuqaibah (Emirate of Sharjah) dated to the fine stage of the Iron Age.[46] In Saudi-Arabia (Northern Hijaz) surface finds made during a survey included an iron slag from site 200-41 dated to the late 2nd to mid-1st millennium BC[47] and another piece at Al-Hawra (seaport) dating to the

[43] Gazdar, Potts & Livingstone, 1984: 68-69; Potts, 1993b: 94-95.
[44] Salter, 1994: 382-385.
[45] Frifelt, 2001: 142 (Fig. 279), no further details mentioned.
[46] Millet, 1997: 37.
[47] Ingraham, Johnson, Rihani & Shatla, 1981: 74.

Islamic period.[48] Further details on all these finds are lacking however and it is impossible to evaluate these finds any further.

9.2.2. Mleiha

The interdisciplinary team that worked at Mleiha took a more wide-angled approach in studying the slag. The preset questions were: to find out if the iron objects were local fabrication or if imported iron was worked, and to see where in the process the slag originated. They tried to link the slag found at Mleiha to the geological iron deposits in the neighbourhood. Some 20 samples were collected and subjected to different analyses: macro- and microscopic examination, XRD- analysis and chemical analysis.

• **Geology**

The short-term geological survey in the calcareous mountain chains of Jabel Faiyah and Jabel Buhays turned out three types of iron minerals potentially suited for iron production. In the immediate surrounding of Mleiha several sources were sampled and the analytical results can be summarized as followed[49]:

- The laterite at Jabel Buhays has an iron content of *ca*. 45% (Fe_2O_3) and the associated elements are not harmful for a reduction process. The chemical elements that could serve as a 'fingerprint' to establish a connection between the ore and the slag are: elevated levels of SiO_2, MgO, Na_2O and the elements of Ni, Cr, and reduced levels of Be, Mo, Pb, U, W, etc. This composition is related to the basal mother-rocks, e.g. an ophiolithic substrate altered by a continental milieu. Some samples from Jabel Buhays are *chromite* ($FeCr_2O_4$) and have a very high chromium and magnesium (about 40% MgO) content and little iron. This chromite (actually magnesiochromite, $Mg(Fe)Cr_2O_4$) could not have provided the ore necessary for iron production.
- The second group of samples are nodules found in the deep fractures of Jabel Faiyah. Pisoliths with a diameter from some millimetres to some centimetres appear in the sandy filling of cavities in the Maestrichian limestone, but these occurrences are limited. In these areas some blocs of pisoliths fused together with ferruginous cement were found, they were the remains of a laterite different from the one mentioned above. The iron content was 70% (Fe_2O_3) and the associated elements are not harmful for reduction. There is a certain affinity between Al-Ca and silica, together with elevated levels of Be, Cr, Ni, V, W, Zn, etc. This chemical composition reflects a more complex continental origin. Upon smelting the majority of the elements present in the pisoliths will go into the slag.

• **Slag**

In Mleiha slag is well represented, but they are very dispersed and never concentrated in one spot. An average of 0,5 to 2 slags per square metre is given, with a maximum of 3 fragments. Morphologically the majority of the slag is of a type described as *skullcaps* (*coupelle*), comparable to the PCB and the SHB as described above. They are not directly comparable to similar material known from Europe. The diameter varies between 5 and 8 cm and a depth of 1 to 3 cm. The concave side often has backed clay attached sometimes with a 0,5 cm thickness. The interior side is often irregular with bulbs but some are rather flat (~ smithing hearth lumps as mentioned above). The bulbs often have spots that are red oxidised. In section the grain is fine, grey or black and has a low porosity. The slags are magnetic, with a high specific gravity and little visual internal heterogeneity. Little globular drops with a 1 to 3 cm length and 0,3 to 1 cm thickness are also attested. Their interior surface is also grey to black but is sometimes more porous (termed *poupée* by the analysts). They have been found in association with the skullcaps but also with hammer scale. The hammer scale is well

[48] Ingraham, Johnson, Rihani & Shatla, 1981: 78.
[49] Ploquin & Orzechowski, 1994: 25-26; Briand, Dalongeville & Ploquin, 1997: 51; Ploquin, Orzechowski & Briand, 1999: 172.

preserved and rather large if compared to the European material, with sizes of 1 cm up to 2 cm and a thickness less than 1mm. The scale seems to be never associated with the skullcap-shaped slags.[50]

Microscopical the skullcaps show iron oxides that are present in a smithing slag, but the structure is different. The olivine phases are much more trapped, and not present as skeletal laths or dendrites. Magnetite is very abundant in the glass phase, sometimes with massive granular texture, paved with globular inter-granular relics of wüstite. Within this mass of magnetite small island or waves with fine dendrites of wüstite are seen, with or without olivine. On some places banded structures are visible that remind the hammer scale in smithing slags. If compared to European material these slags fundamentally differ from the reduction slags of low-furnaces and smithing slags. The hammer scale on the other hand has the classical mineralogy, crest of massive magnetite and zones of molten wüstite and olivine in various proportions. XRD-analyses confirmed the omni-presence of wüstite, magnetite, olivine and sometimes hematite.[51]

The slag of Mleiha was analysed for their chemical composition by ICP-MS. The amount of SiO_2, Al_2O_2, Fe_2O_3, MnO, MgO, CaO, Na_2O, K_2O, TiO_2 and P_2O_5 were determined in wt% and the elements Sc, As, Ba, Be, Co, Cr, Cu, Mo, Ni, Pb, Rb, Sb, Sn, Sr, Th, U, W, Y, Zn and Zr in ppm. All slag had high Mg, Cr, Sr, Zr and to a lesser degree Ni-content. Some slags contained copper. This could indicate that iron and copper were worked in the same room. Or alternatively that iron containing ore from the gossan above the copper ore body was used.

According to the report the chemical analyses on the skullcap slags would tend to eliminate here the smithing slag interpretation and their interpretation therefore would be in the direction of a *smelting furnace bottom* or *crucible smelting slag*. The following conclusions are these drawn by A. Ploquin, S. Orzechowski and B. Briand, who illuminated the smithing slag hypotheses. The local ores would have a strong affinity to their ophiolithic origin and would be rich in chromium and nickel. Upon smelting the chromium will concentrate into the skullcap slag and the nickel will more easily concentrate into the iron produced. The examination of a furnace wall demonstrates that the operating temperature was around 1300°C and that the iron produced would have been characterized by high nickel content and still an appreciable chromium content, despite the fact that most would go into the slag. As a result the waste products at the forge (hammer scale, slags other than skullcaps, etc.) are also expected to contain elevated levels of nickel and some chromium. This hypothesis is partly sustained by the analytical data on the chromium levels in the slag and in some of the ore nodules. But other factors oppose this idea. Sr, Ca and K are present in the slag in too high concentrations when compared to the available ores. It should however be kept in mind that the two latter elements can be introduced by the charcoal (ash) and hearth lining. Also the Titanium levels are much to high to link them to the sampled ores. Other elements (e.g. Al, Mn, Cr, etc.) are on the contrary underrepresented. All this opposes a link between the slag and the ores available in the neighbourhood. Finally one piece of corroded iron was also analysed. It had elevated levels of As and Cu and low levels of Co, Cr, Ni and V and does also not fit in with local ore use. Only one piece was analysed however, so any conclusions are extremely preliminary. One argument that could hamper the ore-slag link is the fact that locally produced iron was mixed with imported iron obscuring the possible chemical relation between the ore and the iron.[52]

[50] Ploquin & Orzechowski, 1994: 26; Ploquin, Orzechowski & Briand, 1999: 173 & 176-177.
[51] Ploquin, Orzechowski & Briand, 1999: 117-178.
[52] Ploquin & Orzechowski, 1994: 27; Briand, Dalongeville & Ploquin, 1997: 51; Ploquin, Orzechowski & Briand, 1999: 178.

The chemical signature of the pisoliths (up to 75% of Fe) does not collide with that of the slag[53]. The widely spread pisolith deposits do show signs of exploitation but they cannot be dated. Although less clear, also the connection with the laterite (45% of Fe) cannot be shown. An ancient deep (man-made?) trench in one of the deposits cannot be dated and could also be related to copper production.[54]

- **Archaeological contexts**

During a survey of the site carried out in 1989, 4 of the 44 tested areas had iron slags present (areas BE, BS, BT and CI[55]). On two of these areas, BE and BT, the slag was rare, whereas on the two others, BS and CI, there was a high concentration within a radius of about 20 m.[56] Indications of metalworking were also found inside the fort (area CW) and in area CY. More recently area 7 also yielded some iron slag[57].

Area BS

In Area BS a first trench of 12 m² uncovered occupation levels of PIR-B. A total of 4,7 kg of iron slag was gathered from the trench. A partly preserved floor of burned soil and ashy pockets was found. The layer is pierced with shallow postholes. A light construction was associated with this surface, but no traces of ovens or of heating places were exposed. The excavators suggest that this is because of the fact that only the surroundings and not the actual metallurgical working places were excavated. The concentration of slag on the site demonstrates that at least one stage of iron metallurgy was preformed there in PIR-B.[58]

Two extra trenches were opened in the next excavation season. The NE-trench produced a cylindrical structure suggesting a small furnace of low shaft type in mud brick, it contained no slag in place (but they do appear in the filling) and no trace of interior coating or of firing was observed. However skullcaps slags, as well as iron fragments and pieces of lining were recovered in the associated layer around the structure and from a pit. The pit also contained flakes of hammer scale. At the same level several heaps of some broken bricks could be evidence of similar dismantled structures. The context would tend to an interpretation of this cylindrical structure as a metallurgical furnace, but no additional arguments can be given.[59]

The SW-trench yielded a line of pits dug in the sand containing plenty of hammer scale as well as a few little rounded slags and very corroded iron fragments. Two hearths contained a few small slags and terracotta fragments. No skullcap slags were encountered, though a fragment of copper slag was found.[60]

Area CI

Area CI is situated on a slight rise and a concentration of iron slag was seen at the surface. The excavation of a surface of 31 m² did however not reveal any clue to iron metallurgy and the iron slags were limited to the loose surface layer. The excavators concluded that the workshop probably belonged to PIR-C and had been totally destroyed by bulldozer during preparation of cultivated land. Under the surface, copper slags without significant structure were found on the hardened silty sediment upon which rests the occupation of PIR-A.[61]

[53] Additionally it needs to be said that pisoliths are notoriously difficult to reduce to produce metallic iron (pers. comm. M. van Nie).
[54] Ploquin & Orzechowski, 1994: 27 & 29; Briand, Dalongeville & Ploquin, 1997: 51; Mouton, 1999a: 274; Ploquin, Orzechowski & Briand, 1999: 178.
[55] Some iron slag appear also to have been found in areas AI, BE, BF, BO, BV and E, but nothing is mentioned on the contexts.
[56] Mouton, 1992: 207.
[57] Jasim, 2001: 128.
[58] Mouton, 1992: 207-208; Mouton, Mokaddem & Garczynski, 1997a: 33; Ploquin, Orzechowski & Briand, 1999: 175.
[59] Ploquin & Orzechowski, 1994: 27-28.
[60] Ploquin & Orzechowski, 1994: 28.
[61] Ploquin & Orzechowski, 1994: 27.

Area CW - the fort

Previously only two locations were mentioned in the fort to have yielded metallurgical residues related to iron working, room P740 and P715[62]. A more recent article by A. Benoist, M. Mouton and J. Schiettecatte[63] included a map (reproduced in Fig. 118) with the spread of the metallurgical remains within the fort. Various crafts were practised in the fort of which the working of iron, and 'copper and bronze' are the most common. Concentrations of iron slag together with hammer scale, hearths, charcoal, filings, grinding stones and anvils were recovered together in three places. Individual remains are registered in several other spots. This gives a more diversified picture.

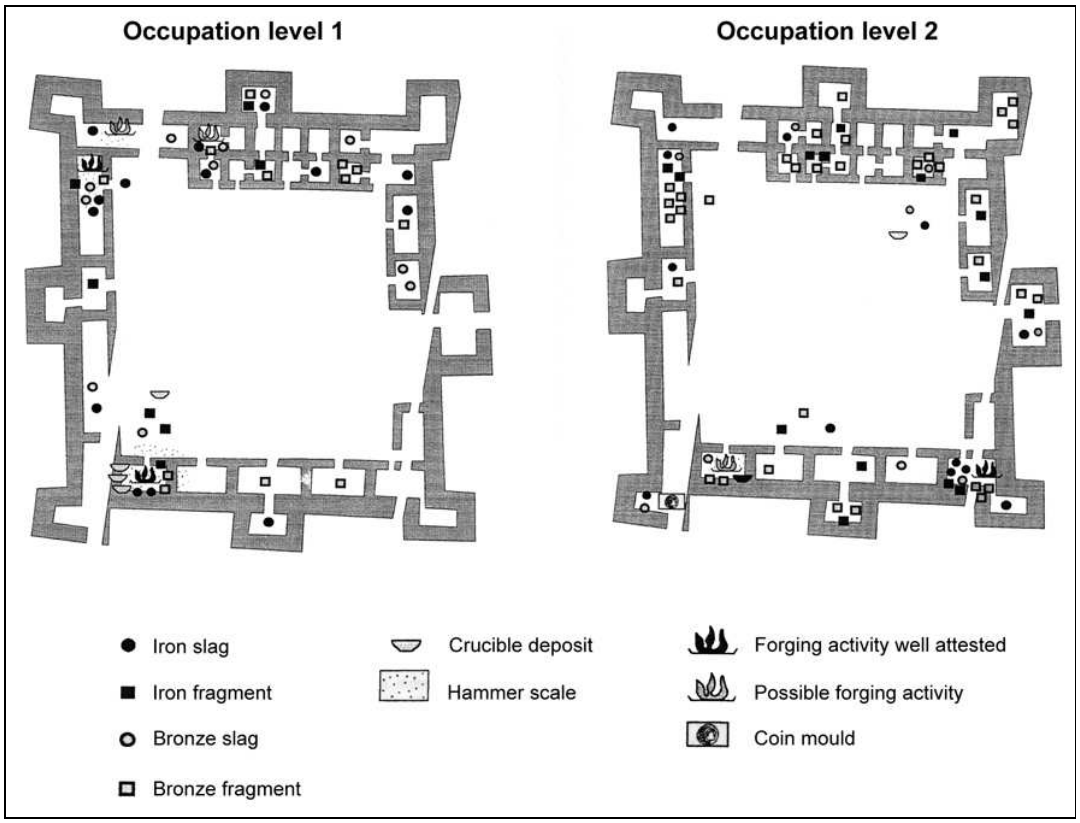


Fig. 118: Plan of the fort at Mleiha showing the distribution of the metallurgical remains (after Benoist, Mouton & Schiettecatte, 2003: Figure 4 & 5).

The presence of iron hammer scales helped to identify these places as iron workshops for the fabrication and repair of tools and weapons. In the fort[64] several locations with metallurgical residues were excavated, found in two successive occupation levels. During the oldest occupational phase iron was worked in two distinct locations. Room P715 is situated along the S-side of the fort and contained the remains of a smithing hearth of *ca*. 1 m² with a black, charcoal surface. Other residues found were some hammer scale, some small slags and fragments of corroded iron. The second room is placed on W-side of the fort and there 'bronze' was also worked. During the earlier occupational phase the iron metalworking appears to move from P740 to room P858 in the SE-part of the building. Room P715 has a continued activity of iron working associated with 'bronze' working indicated by the 'bronze' slag found. Room P740 does show some 'bronze' working remains though. A third metal workshop has been found in room P739. It appears that metalworking was usually carried out in the storage rooms opening onto the courtyard, and that bronze working and iron

[62] Ploquin, Orzechowski & Briand, 1999.
[63] Benoist, Mouton & Schiettecatte, 2003.
[64] The fort was build in PIR D, but had preceding occupation layer dating to PIR B and C.

working occurred in the same areas. This could indicate that it was performed by the same craftsmen.[65]

The fact that no skull-cap slag where found in the immediate vicinity of smithing hearth does not have to mean that they were not related to these structures. When the smith cleaned out the hearth it is very likely he deposited the waste outside the smithy. Their presence in a habitation structure with possible storage functions, does also not plead in favour of a relation to smelting. Smelting is a high-temperature process with all risks that brings. Normally it is conducted close to the ore source and, of importance here, not in living areas. Blacksmithing on the contrary is attested in many residential contexts.

Area CY
In area CY a pit next to a small building containing some slag and the remains of a specific ceramic vessel (see *pot-bellows* below)[66].

Area 7 (previously area CZ)
More recently during the excavations conducted by S.A. Jasim in 1993-1994 a workshop for manufacturing 'copper or bronze objects' was excavated. Within a wide trench measuring 20 x 20 m large quantities of copper slag and also some iron slag were found.[67]

- **Conclusion**

In the 3rd c BC iron metallurgy appeared for the first time in SE-Arabia at the site of Mleiha. Two blacksmithing workshops were found. Area BS yielded the remains of iron working and according to the archaeologists also iron production. The second group of workshops were excavated inside the fort, which dates to the 1st - 2nd c AD. Evidence of both iron and 'bronze and copper' working was found in the same place. The excavators suggested that the 'bronze and copper' working is probably related to minting activities undertaken in the fort. Another area, Cl, had many iron slags on the surface but was bulldozed to create cultivation space. This destroyed all evidence of the original function of this area. However on a deeper level that was perturbed by modern activities, evidence for 'copper working' was found.

This small overview shows that the metallurgical remains at Mleiha are present at different parts of the site, but nowhere in huge quantities. In spite of the special attention paid to the archaeometallurgy, no real conclusions could be drawn. Smithing was certainly going on, but the evidence for iron production is very slim. Numerous iron objects were found at Mleiha, however some of them seemed rather large in comparison to the size of the slag found in the forges. No relation based on the chemical composition could be made between the iron slag and the sampled iron deposits in the region. Of the different types of waste material reported from Mleiha (e.g. slag cakes, small spherical slag and hammer scale) only slag was recorded at ed-Dur.

Here I would like to add some additional conclusions drawn from the Mleiha analytical dataset. When the chemical analysis of the only analysed iron fragment is compared to those of the slag, the object is in general richer in the siderophile elements As, Co, Mo and Ni and poorer in the lithophile elements (except for Sb and Cu). Although this is an expected pattern it was not mentioned in the report and could actually be used to underpin an interpretation as smithing slags. The two fragments of hammer scale analysed are for the majority made up from magnetite, but siderophile elements are present in lower quantities that in the iron object, indicating that the scale is probably not linked to the iron used for the object.

[65] Benoist, Mokaddem & Mouton, 1994: 13; Ploquin, Orzechowski & Briand, 1999: 175; Benoist, Mouton & Schiettecatte, 2003: 64.
[66] Ploquin, Orzechowski & Briand, 1999: 172 & 175.
[67] Jasim, 2001: 128.

9.2.3. Khor Rori

At the site of Khor Rori room A27a was interpreted as a possible blacksmith workshop. Against the NW-wall a semi circular fireplace (0,3 x 0,5 m) was constructed from medium-sier un-worked stones. The filling consisted of a dark brown loam mixed with ashes and charcoal. Part of another construction made from five elongated roughly dressed stone blocks was discovered in the SE-corner of the room. Four hammerstones were found *in situ* lying above the two central stones of the construction, and two bigger stones, round and rectangular in shape were probably used as anvils. They were found on the floor of the room. A number of iron tools were also attested and hey consist of a nail, a fragment of a pointed tool with a copper-base alloy handle, two fragments of another pointed tool, two iron axes (exact location not clear) and a piece of iron slag. From another location, room A 32, four more iron slags were recovered.[68]

The initials “KR” precede the slags from Khor Rori investigated.

9.2.4. Ed-Dur

C.S. Phillips submitted four samples (AW 001, AW 008[69], AW 020 and AX 008) for EDX analyses already in 1993[70]. The results were not normalized to 100% and are difficult to compare to our findings. Most probably the major element that was left out is oxygen, if this is the case the results are in accordance with the trends seen in the slag analysed here.

The study by A. Ploquin, S. Orzechowski and B. Briand included two samples from ed-Dur of the type they described as “coupelle”.[71] The results will be evaluated further on.

[68] A. Pavan pers. comm.
[69] AW 008 is also the registration number of a piece of litharge (see *Chapter 6*).
[70] C.S. Phillips, pers. comm. and unpublished information.
[71] Ploquin, Orzechowski & Briand, 1999: 184.

9.3. Sample description & macroscopic examination

At ed-Dur slags were already picked-up by J.-F. Salles during his visit in 1979[72]. During the Belgian excavations *ca*. 3 kg of slag fragments en some compete specimens were collected (the other teams also registered an unknown amount). However no concentrations were registered. During a recent visit to the site, several additional pieces were found. The metric and contextual data on these slags originally collected by the Belgian team as well as a tentative typology is presented in *Appendix 8*[73]. No real correlation was found between these 'types' and the other metrical or analytical data, so their shape is related to the formation circumstances and not to different technical processes. The *ca*. 3 kg of slag from the Belgian excavations is thus not to be seen as a definitive amount, it only shows that slag is widely dispersed over the site and found in a low area concentration. Only one slag was found in a possible relevant context, BR 982 (context UF 6055). This is a burned spot, which had some baked clay fragments in its infill. The spot had a surface of 80 x 110 cm and a depth of 8 cm. No further details are available, but this could be a remains of a temporal smithing hearth.

C. Salter states that the specific gravity of slag can vary from relatively inhomogeneous low (2-3 g/cm³) through to higher values (approaching 4 g/cm³)[74]. The average specific gravity of the ed-Dur slag is *ca.* 3 g/cm³ (this is also the mean value). The highest specific gravity calculated for a single piece is *ca*. 5,6 g/cm³ when fragments are considered and 3,3 g/cm³ if only complete specimens are taken into account. The lowest values are respectively *ca*. 1,9 g/cm³ and 2,1 g/cm³ (See *Appendix 8* for details). Still these values fall nicely within the limits put forward by C. Salter.

The diameter of complete slags varies between 3 and 9 cm and the thickness between of 1 to 4 cm, with an average of 2 cm, none seem to have been bigger than *ca*. 10 cm in diameter. A yellowish-white crust often appears over the whole surface of the slag. This must have originated after burial and is probably due to a post-processional phenomenon where calcareous rich particles are deposited on the slag.

Several slags have what seems to be backed clay attached to their concave (bottom-) side. This can be 1,5 cm thick and is grey in appearance, pointing towards reducing condition when backed. This crust must be the remains of the hearth lining (see Fig. 122 - BO 1349-2, 3 & 4). The metal slag itself only forms a thin layer on top of this lining. On a transversal section made by a water-cooled diamond cut-off wheel the transition between the slag and the lining is not visually detectable. Although the bulk of the slag is dense and fine grained, large and small cavities are always visible. The colour is metallic dark grey to black and relatively homogeneous. Brownish spots (due to weathering?) do however sometimes appear in the section. The real specific gravity of the slag (without the air filled gaps) must therefore be higher than calculated, but still in correspondence with C. Salter. Many slags have a bulbous surface on the topside, but some also exhibit this on the bottom side. The extremities often have a reddish-brown iron oxide colour.

The ceramic component is lighter grey in colour but has a dark grey to black appearance on a fresh cut (Fig. 122 - BS 1349-2, 3 & 4). Sometimes glassy vitrified bulbs appear. Cavities are also visible, but these are smaller than in the metal slag layer. The cavities are typically coated on the inside with a white (calcareous?) layer. The last *ca*. 2 mm of the bottom side has more finely dispersed hollows. These ceramic fragments resemble a coarse ceramic type found at ed-Dur, the so-called 'black ware'. Their outer side is unfinished, the thickness varies considerably within one piece and the tempering is much finer than the thickness would make believe. These features would be consistent with an interpretation as lining

[72] Salles, 1980: 98.
[73] Some additional pieces brought back from the others teams are not included.
[74] Salter, 1987: 200-201.

smeared out in a hollow shape. The clay was also heavily vitrified, making it harder than backed pottery examples. The clay probably came from the north of the U.A.E., very likely from the wadi's in the neighbourhood of Ras al-Khaimah[75]. The fact that such lining is preserved shows that the hearths were more elaborate than only a pit in the ground. Since they were at least partly lined they might have had a somewhat more permanent structure (Fig. 121, BS 1131). Possibly the hearths were therefore also used for other activities. The lining is often as magnetic as the slag and must be the result of a reaction between the slag and the original ceramic component. No such heaths were found *in situ* however. In one case (sample 228 – French team) two slags were formed on top of each other, something that can occur when the smithing hearth is used twice without cleaning in-between. The separation is indicated by a dashed line (Fig. 122-5).

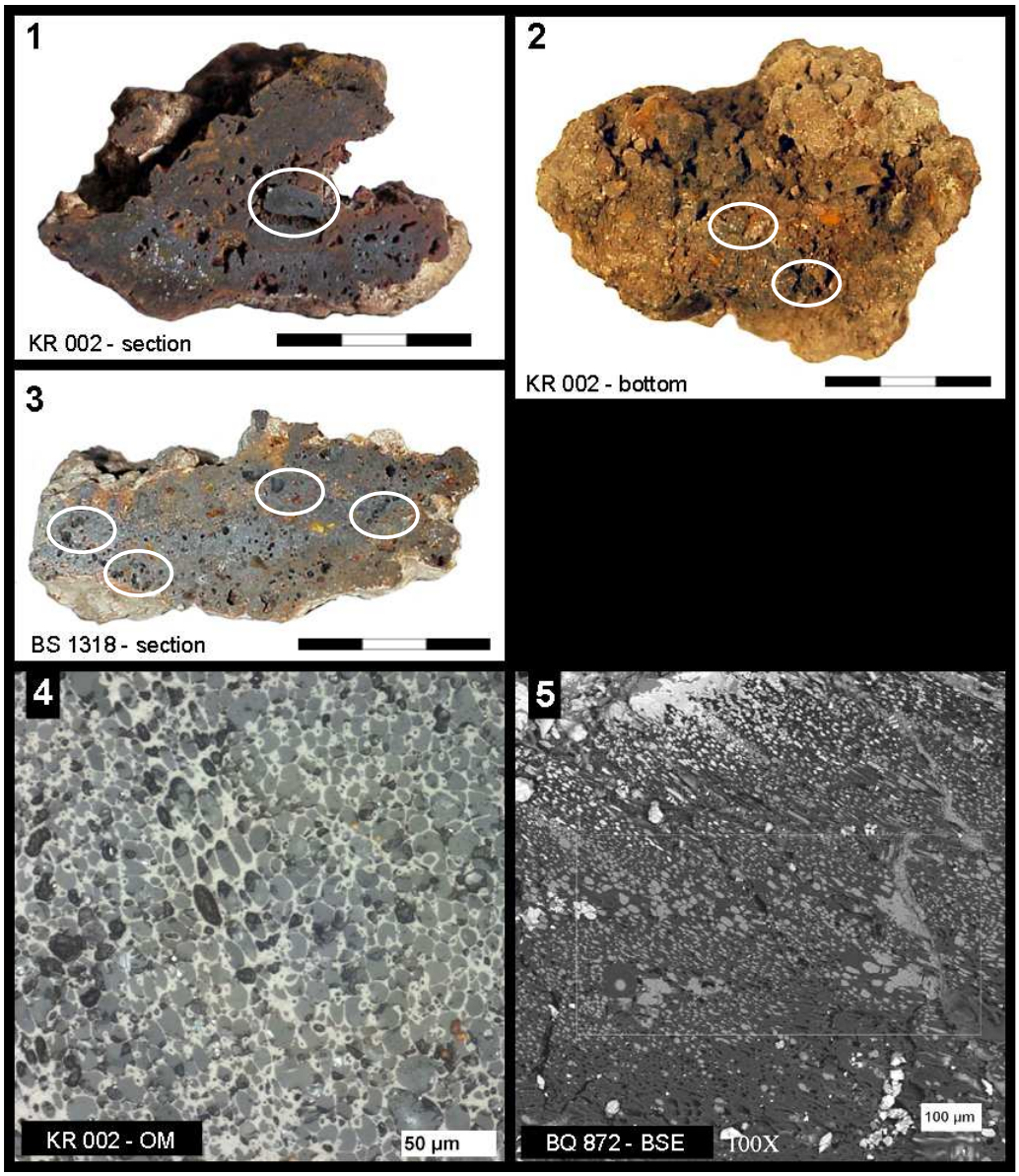


Fig. 119: Charcoal inclusions indicated by white circles.

[75] K. Rutten, pers. comm. The fact that the clay is supposed to originate in Ras al-Khaimah does create an interesting problem, since it implies that wet clay was transported. Something that is feasible but not attested for other purposes. On the other hand it might be possible that the 'lining' was transported as finished bowl-shaped objects, something that seems a little far stretched to me. Although interesting, this subject will not be further developed.

In three of the sample from ed-Dur pieces of charcoal are preserved. BQ 1008B has a fragment stuck in a cavity between the lining and the slag. The section of BS 1318 has several small particles of charcoal dispersed over the transversal section (Fig. 119-3). BQ 872 had a fragment of mineralised charcoal included in the bottom of the slag (an SEM-BSE picture is shown on Fig. 119-5). Much better examples are seen on the slag from Khor Rori, where charcoal particles are well represented on the 'bottom side' of the slags (Fig. 119-2). In one case a completely mineralised charcoal piece was also found inside the slag (see Fig. 119-1 and a picture by optical microscope on Fig.119-4).

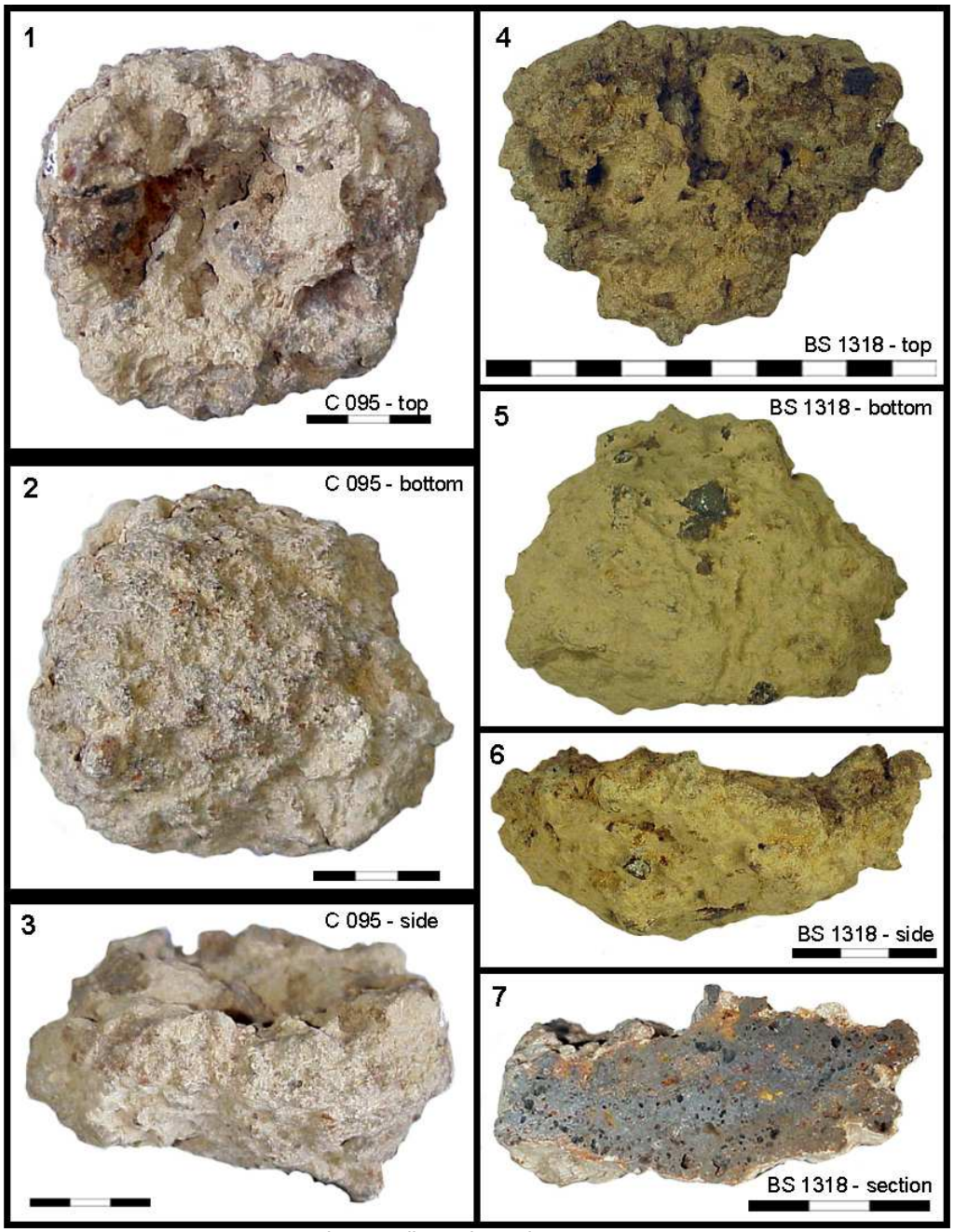


Fig. 120: Illustrations of two PCB's.

Fig. 120 shows two very nice examples of a PCB's. These bowl-shaped slags have a ventral depression due to the air blast. The bottom-side is rather smooth, while the top-side has a more bulbous appearance. One slag is particularly instructive (Fig. 121: BS 1131) because it

is preserved in two parts. The lower part is cup-shaped and consists mainly of lining. On top of that a slag was found that perfectly fitted on the lining but for one reason or another was not fussed to it. This slag has a rather smooth bottom and a bulbous topside. Interesting to notice is also the fact that the surface of the lining that was not covered with slag has a very porous layer on top of the typical grey lining. This must be due to the reaction of the lining under high temperature.

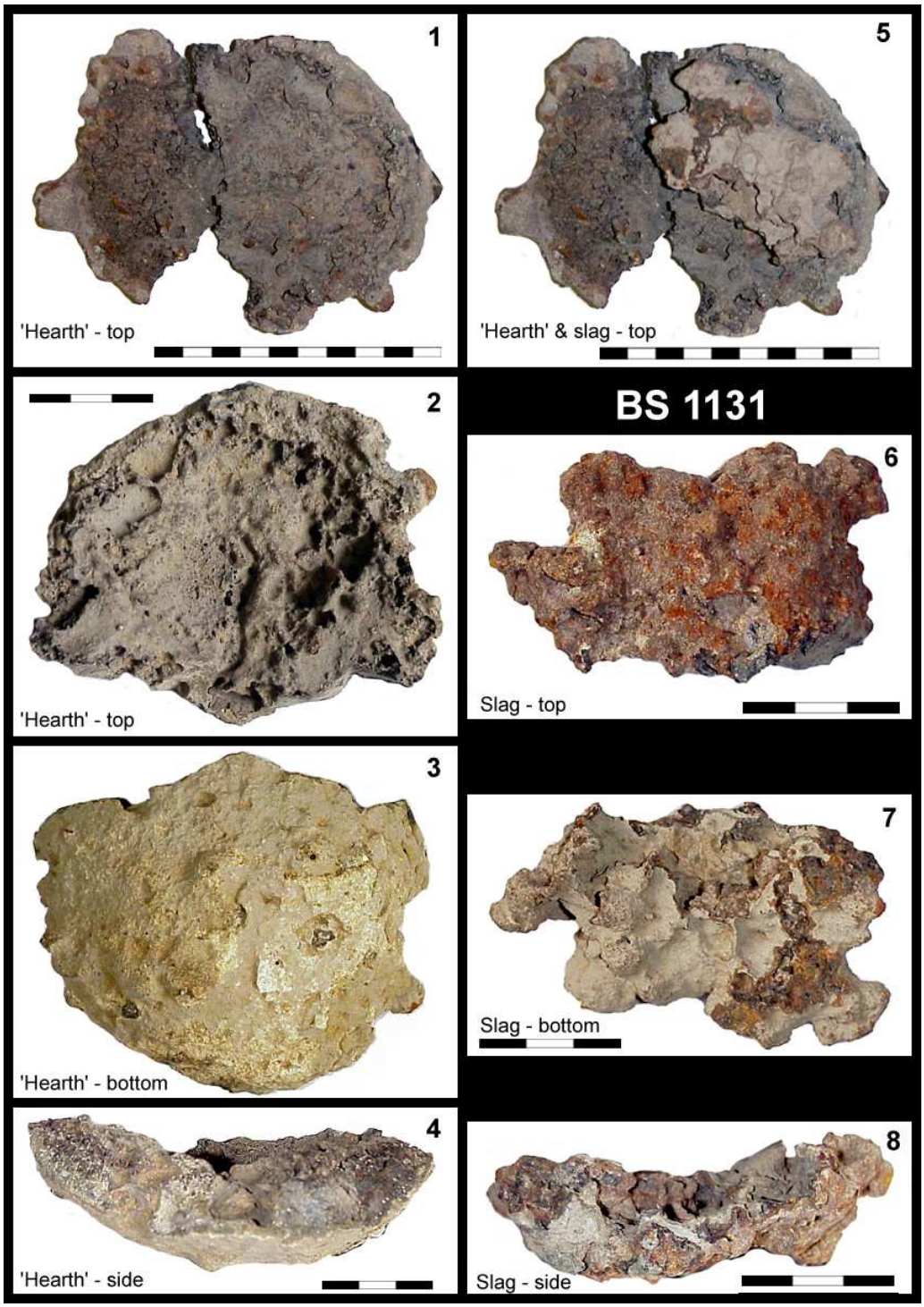


Fig. 121: Illustrations of 'hearth' remains and slag BS 1131.

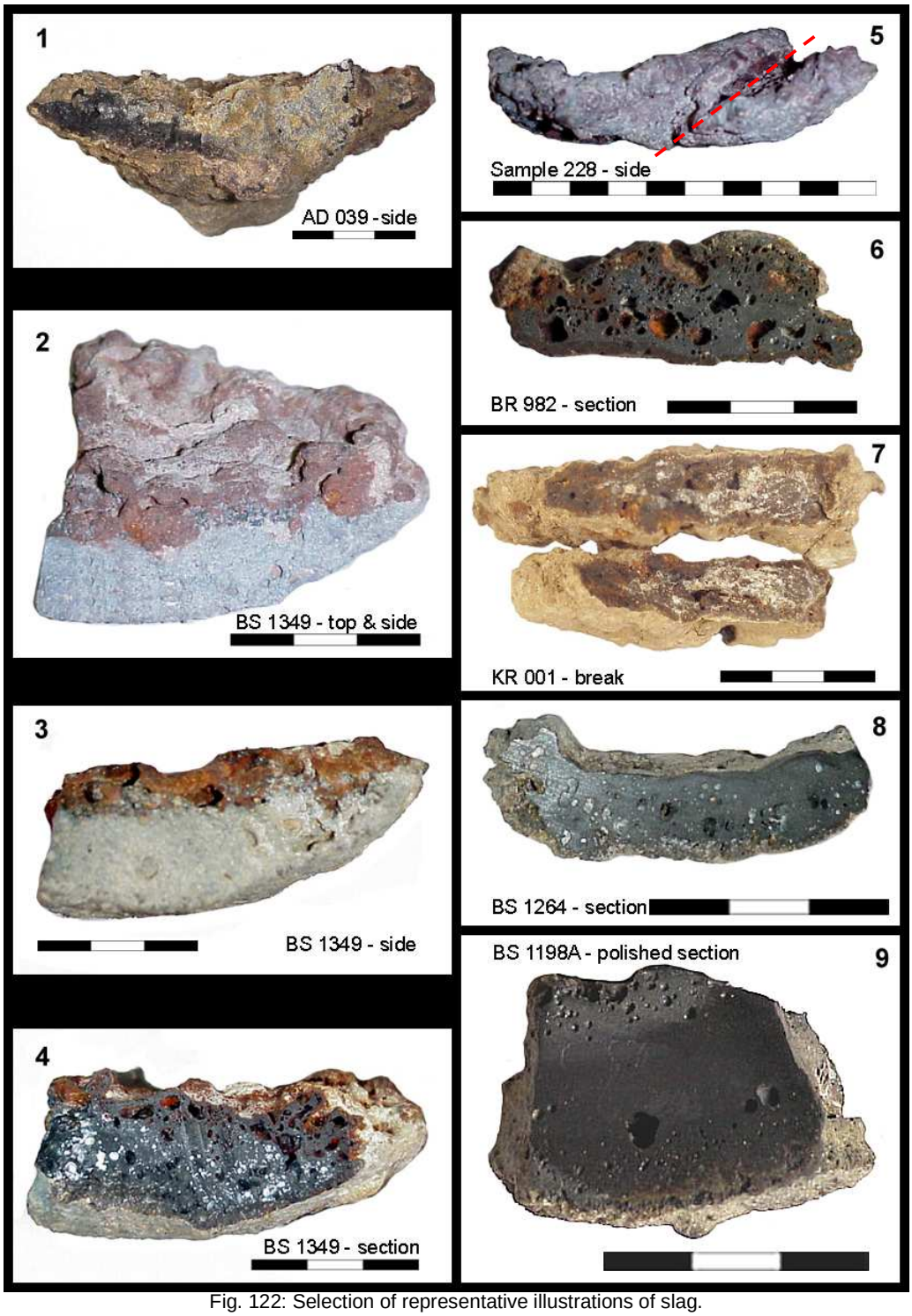


Fig. 122: Selection of representative illustrations of slag.

Fig. 122-6 shows an example of the many vacuoles that can be present, this is however not always the case as can be seen on Fig. 122-9. Most often the slag has the typical reddish-brown colour of oxidised iron (Fig. 122-2), in section however the slag most often appears dark grey to black (Fig. 122-8 & 9). Fig. 122-7 is one of the many examples that the slag is not always cup-shaped and can be rather flat (the pictures shows both sides of the break). The white coating in some of the vacuoles can be seen on Fig. 122-4, in this case in the

lining, but also Fig. 122--8 exhibits this feature. Smithing slags can have a zoned build up. The top layer contains the last material that joined the hearth. In the middle the fully developed texture can be observed with underneath the evidence of the reaction of the first slag, the ash and the hearth bottom. An additional layer can be present at the base that is the result of the reaction of the slag and the hearth lining. The lower region is often more vesicular and shows the 'normal' appearance of this slag. The central region is often denser since this region was exposed to higher temperatures so the structure became denser and the air holes were compressed. The lower temperatures at the base of the hearth make it possible to preserve the more vesicular structure. This can be observed on several examples shown above, where the vacuoles are concentrated at the bottom-side of the slag (e.g. Fig. 122-6, 8 & 9).

Table 47 lists the selection of slag made for further research. The selection was based on: the different external appearance and size of the slags, and on a basic scan by optical microscope. Care was taken to include as many of the different microstructures attested as possible. The selection made here can be seen as a representative sample of the collection. An additional factor at play was the porosity of the slag, although this was not always clear from a visual examination. This hindered the establishment of sufficient vacuum conditions in the sample room of the SEM and prevented any analyses. As solution for this would have been the preparation of petrographical slices of the slags. Because these are thin, the total amount of vacuoles is much less important than when a large fragment of slag is introduced in the vacuum room of the SEM. This solution was thought of when writing up this PhD and can only offer an alternative for future studies.

Sample nr.	OM	SEM-EDX	XRD	Extra
AH 233	**x**			
BO 716		**x**		Small slag
BO 845A	**x**	**x**	**x**	
BO 1349			**x**	Too fragmented for SEM
BQ 819	**x**	**x**	**x**	
BQ 872	**x**	**x**	**x**	
BQ 875	**x**	**x**	**x**	
BQ 1008B		**x**		
BS 1068		**x**		
BS 1110	**x**	**x**		
BS 1131			**x**	Completely pulverized
BS 1185A		**x**		
BS 1198A	**x**	**x**	**x**	
BS 1215		**x**		
BS 1264	**x**	**x**	**x**	
BS 1318	**x**			
BS 1417			**x**	
KR 002	**x**	**x**	**x**	
KR 003	**x**		**x**	Impossible to make vacuum in SEM

Table 47: Sample numbers and methods of the analysed slags.

9.4. Microstructural observations – optical microscope

9.4.1. Optical microscopy

A large number of slags were cut in three by a water-cooled diamond incrusted cut-off wheel. These were polished according to normal metallographic standards till 1 µm. This gave good microstructural results under an optical reflective metallographic microscope. Although not exactly the same, the microstructure of the many slag was similar enough to take some representative examples to present here (eight from ed-Dur and one from Khor Rori). From several slags a complete profile was composed to show the microstructural evolution throughout the section. Close-ups of the different microstructures are includes.

- **AH 233**

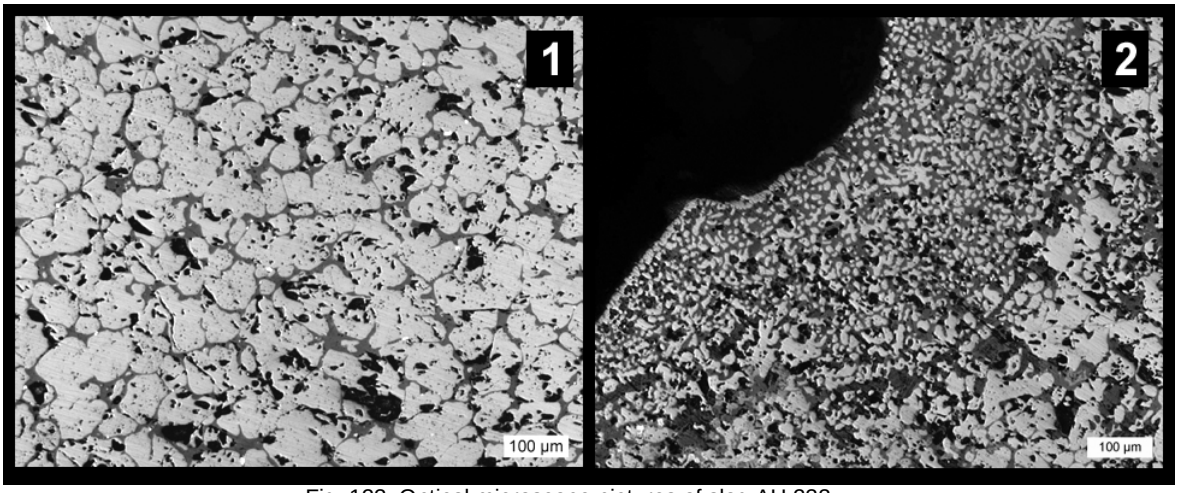


Fig. 123: Optical microscope pictures of slag AH 233.

AH 233 is one of the larger slags (*ca*. 7 x 6,5 cm, specific gravity: *ca.* 3,8 g/cm³). The slag has a smooth bottom and a bulbous upper surface. Fig. XX-1 shows the most common microstructure seen in the slags looked at, rounded 'tiles' of iron oxide (wüstite or magnetite) with a network of a second phase in-between. The white dots are metallic iron particles and the black areas are porosities. Fig. XX-2 shows the same phases but in a different ratio. The size of the iron oxide globules when they solidified can vary considerably, even within one sample. They can go from closely connected tiles, over more dispersed globules to dendritic form. This amount of iron oxides would be typical for a smithing slag, since if this amount of iron was lost to a smelting slag the operation should at least be called unsuccessful.

- **BO 845A**

BO 845A is small fragment of slag (*ca.* 4 x 4,5 cm, specific gravity: *ca*. 3,8 g/cm³) with a glassy surface. Vacuoles are more numerous at the bottom of the slag. On Fig. 124-1 and 2 the bright white spots are inclusions of metallic iron. Fig. 124-1 shows two phases a light grey one (1) and darker grey laths (2), next to dark grey rounded inclusions (3). The light grey phase in Fig. 124-2 is the same as in Fig. XX-1, but it is much less abundant. The matrix that appears in medium grey (4) is not seen on the first picture. The dark laths are probably the same as on Fig. 124-1. These phases are analysed by EDX (see below). The black areas are vacuoles.

In general the slag has a zoned build-up. The brightest phase is most abundant at the top and disappears towards the bottom. (Fig. 124-1 is from the top and Fig. 124-2 from near the bottom). Metallic iron particles are dispersed in small amount throughout the slag. In a restricted area a dendritic form of iron oxide is also appeared (not depicted).

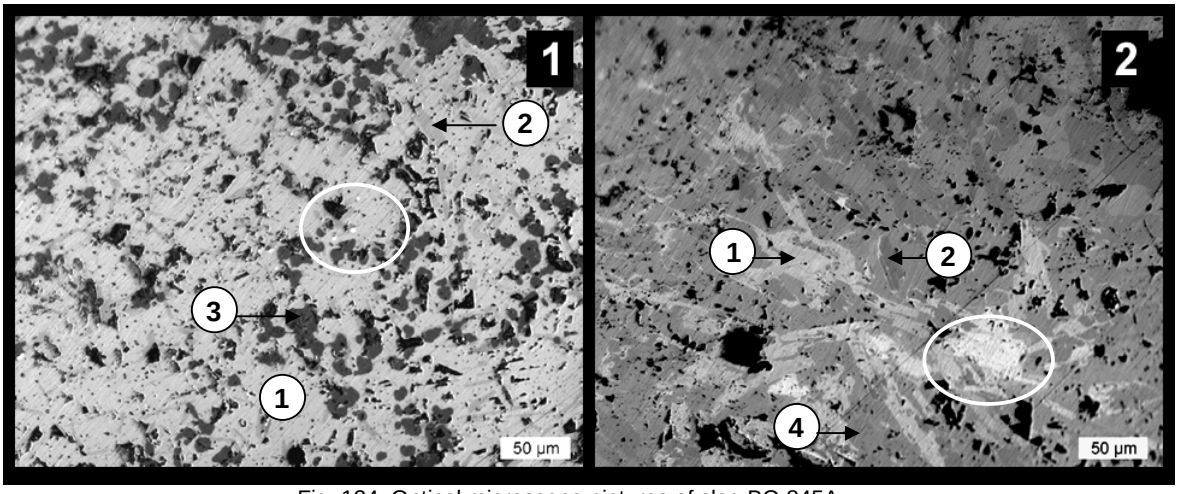


Fig. 124: Optical microscope pictures of slag BO 845A.

- **BQ 819**

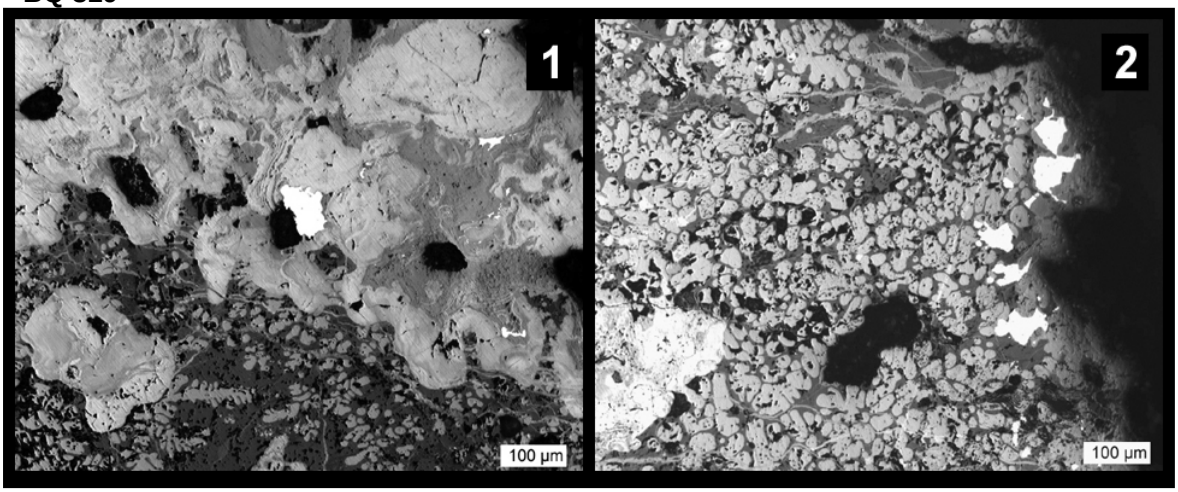


Fig. 125: Optical microscope pictures of slag BQ 819.

BQ 819 is a small fragment of slag (*ca*. 3 x 4 cm, specific gravity: *ca*. 2,9 g/cm³) The grey mass at the top of Fig. 125-1 is a hydroxide form of iron and is the result of postburial hydration of the slag. Beneath is a zone of a dark grey matrix with wüstite dendrites grown into it. The bright white areas are metallic iron inclusions. Fig. 125-2 shows an area with larger and more closely packed dendrites in a similar dark grey matrix. This slag was analysed by SEM-EDX.

- **BQ 872**

BQ 872 is also a small fragment of slag (*ca.* 4 x 4 cm, specific gravity: *ca*. 3,4 g/cm³). Fig. 126 shows a complete section of a slag. This is mainly to illustrate the inhomogeneous build-up of slags, a feature that does not simplify their characterisation and analyses. The same goes for the other sections presented below.

This slag actually has no nice microstructural features and shows that the microstructure can differ not only within one slag, but also between similar specimens from one location. At the top sand grains can be seen fused to the slag. These are probably to be seen as the result of a post-depositional process and are stuck in the secondary iron oxides formed over time. The large wavy area is a hydroxide of iron. Metallic iron particles are dispersed throughout the section. The light grey phase is an iron oxide and the structure gets finer towards the bottom. Two more phases appear next to the iron oxide.

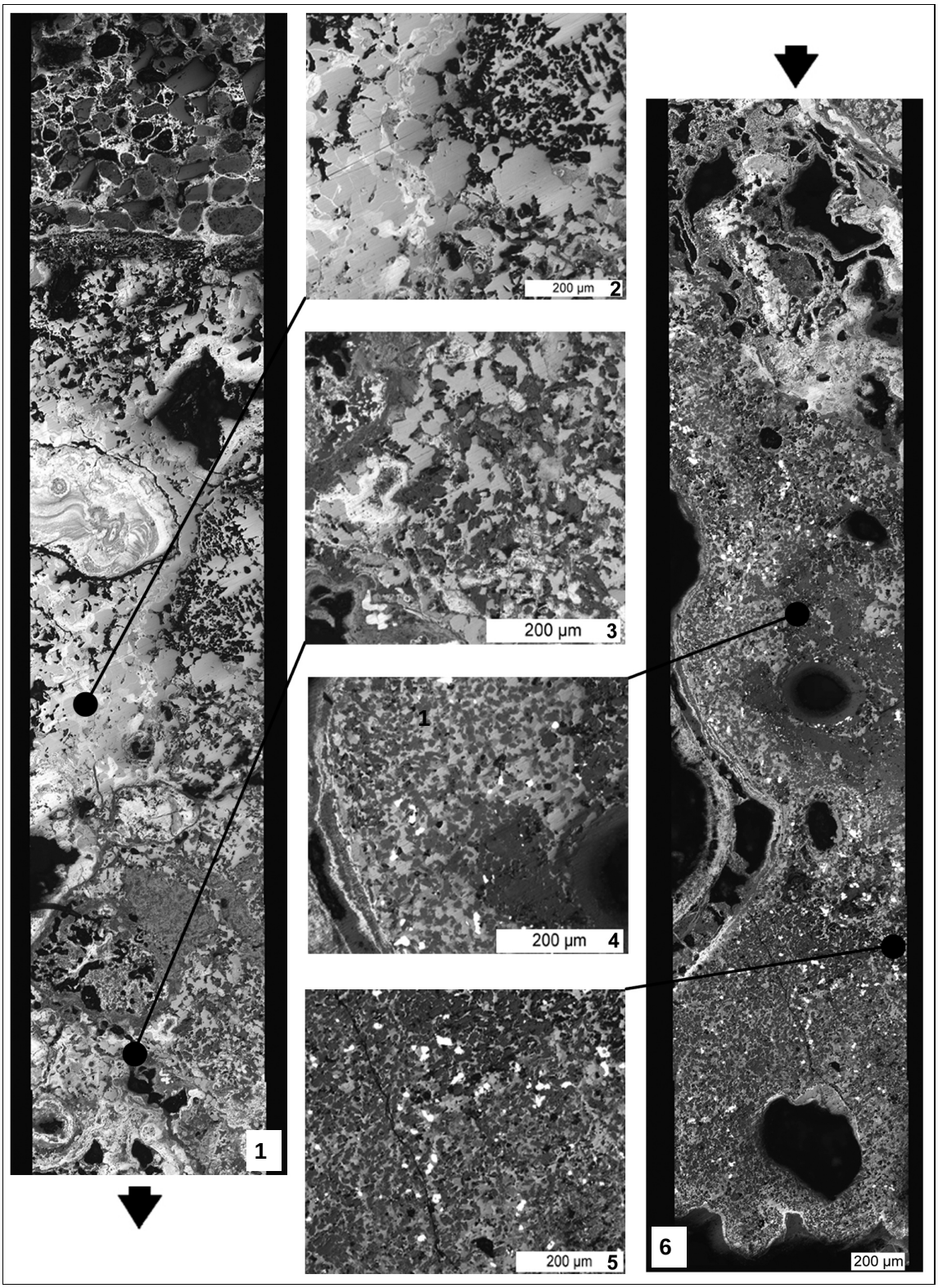


Fig. 126: Section of BQ 872, microstructure by optical microscope.

As a whole this slag is little informative concerning its microstructure, except for the fact that it certainly is not the product of a smelting operation and by consequence must be related to a smithing process. Smelting slags tend to be more homogeneous and always exhibit iron oxide dendrites.

- **BS 1198A**

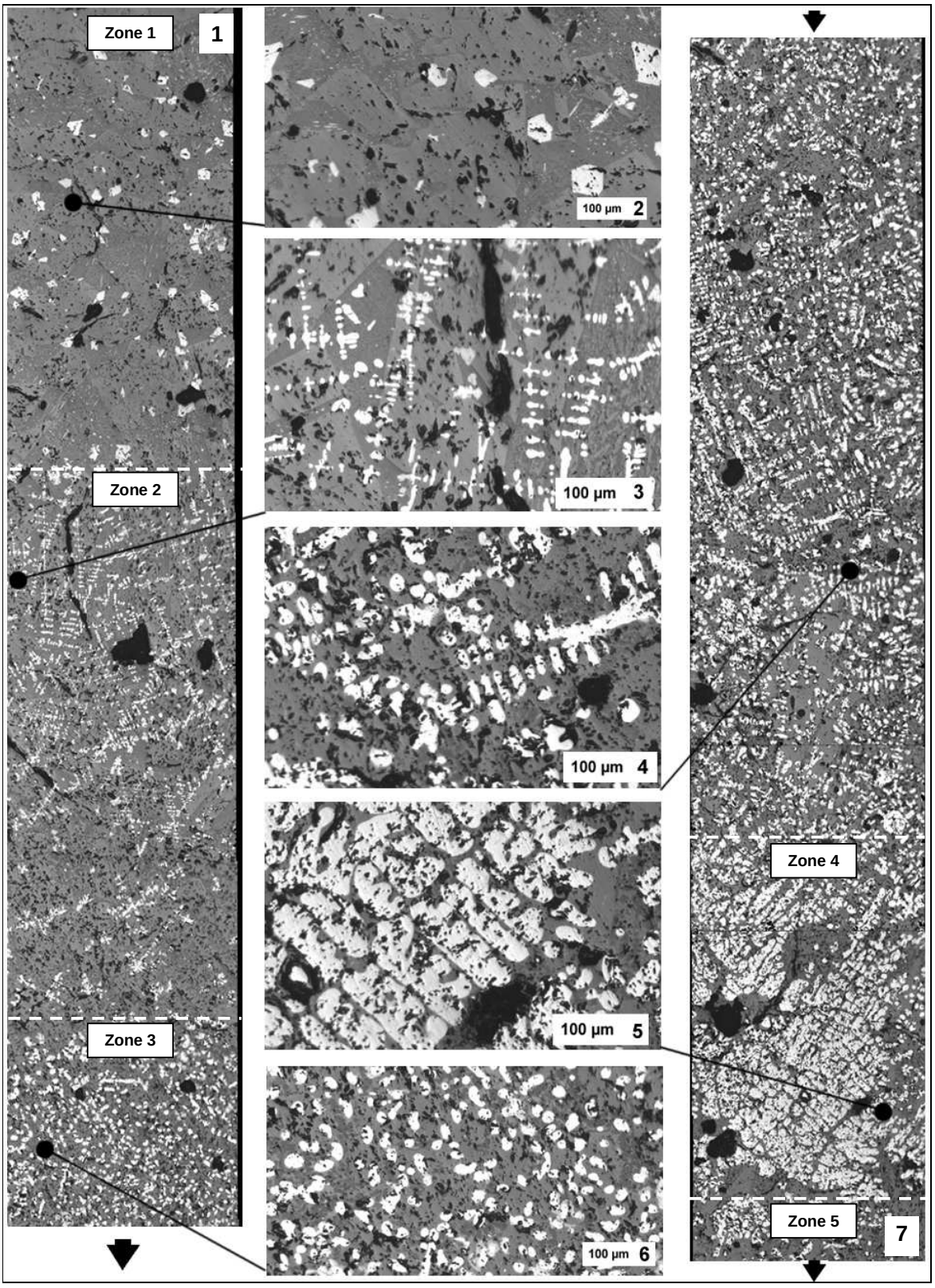


Fig. 127a: Section of BS 1198A, microstructure by optical microscope.

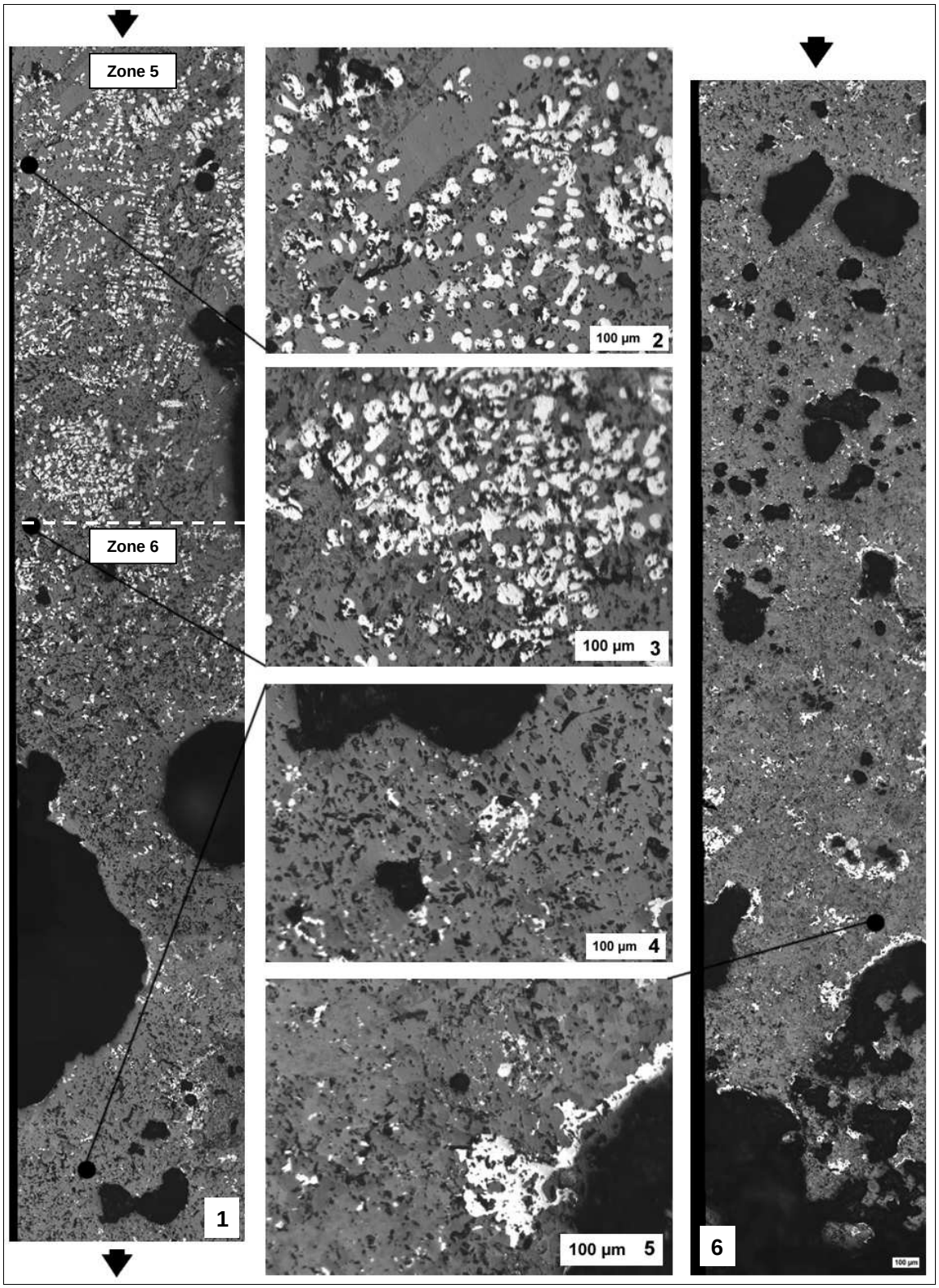


Fig. 127b: Section of BS 1198A, microstructure by optical microscope.

BS 1189A is a fragment of a rather thick slag (*ca*. 8 x 6 cm, specific gravity: *ca*. 3,2 g/cm³). The microstructure is completely different from the previous slag and if only a small part of the section would have been studied it might have been classified as a slag resulting from a smelting operation. Especially the images shown on Fig. XXa-2 and 3 and Fig. XXb-2 remind of the microstructure generated in a typical smelting slag. The overall build-up of the slag is however much too heterogeneous for such an identification and the presence of dendrites here is indicative for a long forging or welding operation. Large smithing slags can contain dendrites in certain zones or in small areas that reached the melting temperature in the hottest zone of the forge. A picture of the polished section can be seen at Fig. XX-9.

The section shows six different zones and they are separated on Fig. 127a and b by a dashed line. The black spots and areas are vacuoles and they get noticeably larger and more numerous towards the bottom of the slag. *Zone 1* shows nicely formed angular iron oxide crystals (wüstite or magnetite) with light grey laths. There is an interstitial phase that shows a mixture of this light grey phase and a darker grey one, intergrown with small secondary iron oxide dendrites (Fig. 128-1). The angular crystals are not seen anymore in *zone 2* but instead the iron oxides appear as larger nicely formed dendrites. Both grey phases are still present, as is the mixed infill. The small secondary dendrites are absent however. The larger dendrites are less common towards the base of this zone and are replaced by semi-dendritic globules in *zone 3*. The light grey laths get smaller as do the areas with infill. The two phases are still present but in larger crystals, and the darker grey phase is dominant. Overall the light grey phase dominates the matrix of *zone 3*. Towards the base of this zone larger dendrites appear again and they are even bigger than in *zone 2*. *Zone 4* is dominated by large tile-shaped iron oxides packed together. These could be the remains of a oxidised and partly dissolved fragment of metallic iron that fell in the hearth and was absorbed by the slag. The two grey phases are still present. *Zone 5* is similar to *zone 3* but the light grey laths are better developed. The infill existed out of a mixture of larger crystals of the light and dark grey phase and no secondary small dendrites are present. *Zone 6* makes up 1/3 of the slag section. Iron oxides appear dispersed as single particles or larger areas, but no dendrites are to be found. The vacuoles are large and numerous. The light grey phase is omnipresent and the dark grey phases appears in smaller areas till the bottom of the slag.

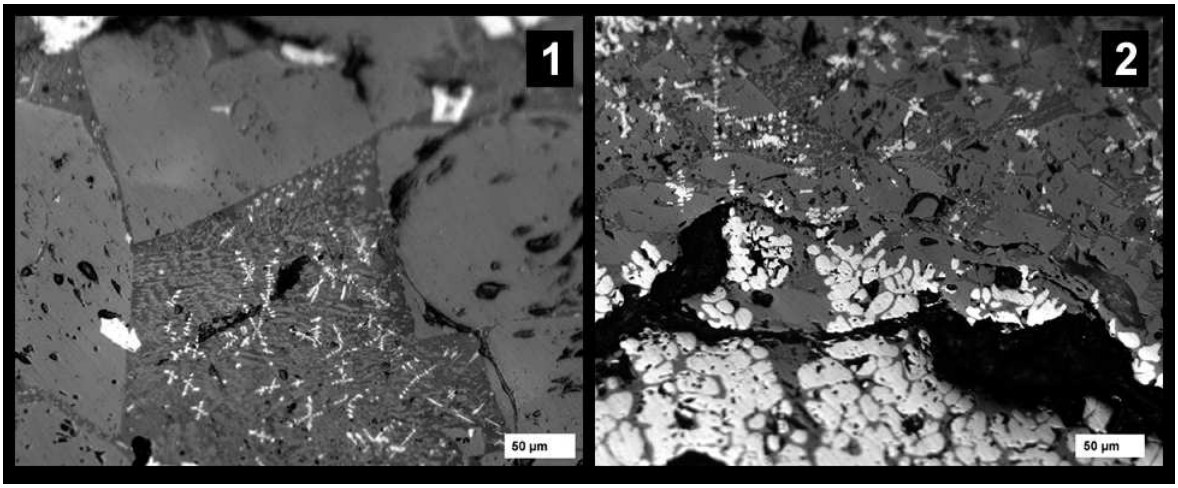


Fig. 128: Optical microscope pictures of slag BS 1198A.

Fig. 128-2 (not from section) shows that the transition between an area poor in iron oxide and one rich in iron oxides can be very abrupt.

• **BS 1110**

BS 1110 is a fragment of slag that has a glassy bulb at the surface and has a high amount of ceramic components, originating from the 'hearth lining' (specific gravity: *ca*. 2,1 g/cm³). BS 1110 is a slag very poor in iron oxides and they appear nowhere more abundant than in Fig. 129-1. Next to the white iron oxides two grey phases are present. Fig. 129-2 is almost void of iron oxides, but three grey phases appear, indicated by the numbers 1, 2 and 3. This slag was analysed by SEM-EDX.

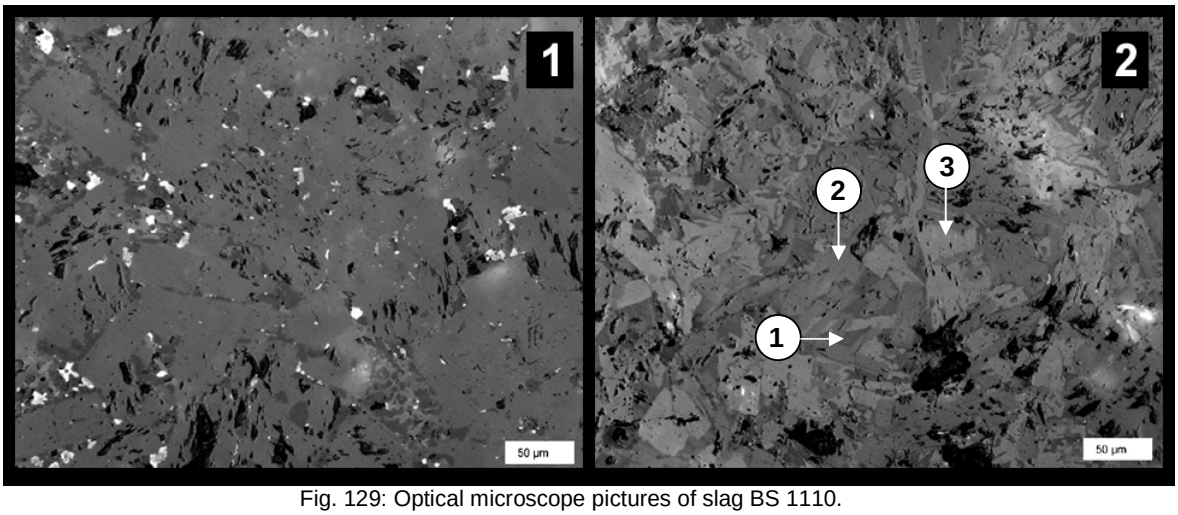


Fig. 129: Optical microscope pictures of slag BS 1110.

• **BS 1318**

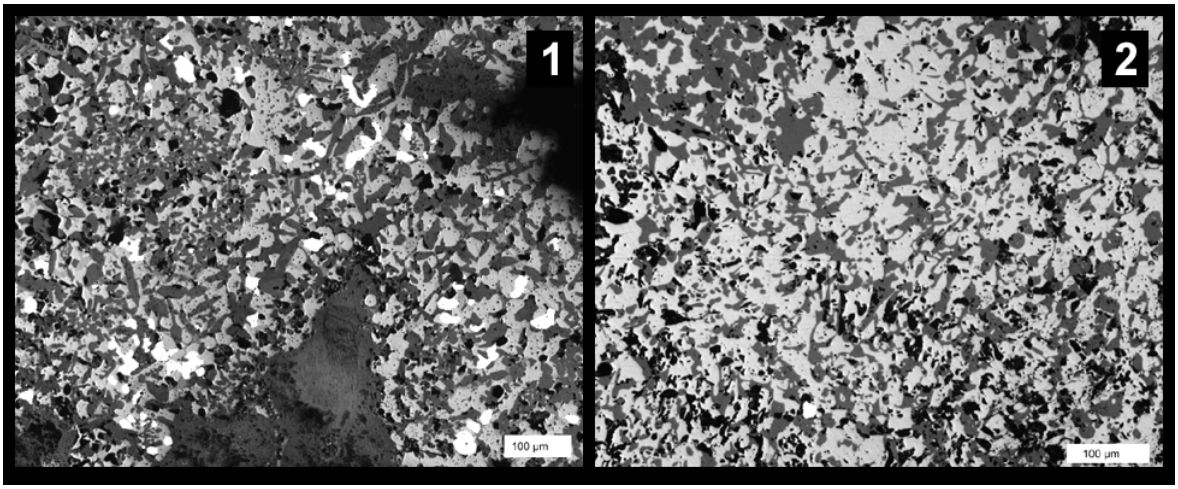


Fig. 130: Optical microscope pictures of slag BS 1318 (1) & KR 003 (2).

BS 1318 shows a area of the microstructure of one of the large bowl-shaped slags (*ca*. 9 x 7 cm, specific gravity: *ca*. 3 g/cm³, see Fig. 121-4 to 8 for pictures). Metallic iron particles appear throughout the section, next to a light grey iron oxide phase and a darker grey phase.

• **KR 003**

KR 003 comes from Khor Rori and essentially has the same phases present as BS 1318, being a light grey phase and a dark grey one. A small metallic iron particle is to be seen at the base of the picture. The slag has the same build-up throughout although the ratios of the light and dark grey phase tend to differ in different regions, and basically more of the dark phase is seen at the bottom part (Fig. 130-2 comes from the top part).

.• **BS 1264**

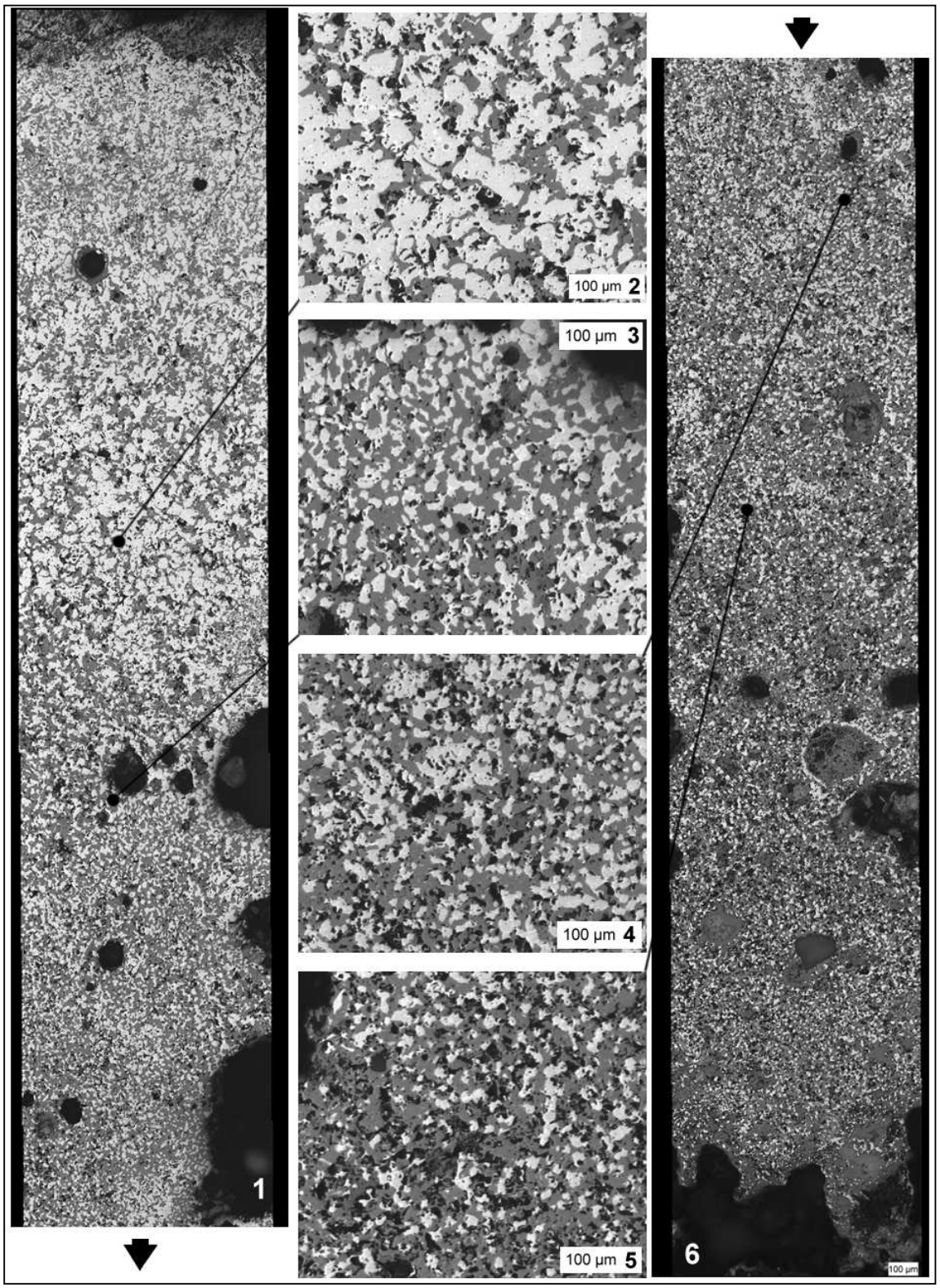


Fig. 131: Section of BS 1264, microstructure by optical microscope.

BS 1264 is a fragment of a slag of which the section has a dark grey colour (*ca*. 6 x 4,5 cm, specific gravity: *ca*. 3,3 g/cm³, see Fig. 131-8). Only two phases can be seen and the light

grey phase is concentrated to the top of the slag where they form angular areas with little infill. More to the bottom the dark grey phase predominates and the light grey phase appears globular (see Fig. 131). Small metallic iron particles are seen throughout the section

In restricted areas the two phases appear as if they have been molten with small more dendritic shaped iron oxides grown in the dark grey matrix. Very small inclusions of metallic iron are observed throughout the slag, associated with this light grey phase. The number of large vacuoles is concentrated in the lower part, together with many small vacuoles, seen as the black dots on the metallographic pictures. This slag was analysed by SEM-EDX.

• **Discussion & conclusions**

All, but one (BS 1110), slags show the presence of a form of iron oxide. The oxides can be present in different forms: massive tiles (probably magnetite or wüstite), rhomboid crystals sometimes partly decomposed, nicely formed dendrites (wüstite), small secondary dendrites or as a 'groundmass' without rounded form. The hydroxide is also sometimes present, but this is due to post-depositional processes and the weathering of the slag. Small particles of metallic iron do appear on regular bases, throughout the sections.

Next to the phases classified as iron oxides, two other phases appear in darker grey shades. In the slags AH 233, BQ 819, BS 1264, BS 1318 and KR 003 only one phase is present. From the iron oxide-rich slags only BO 845A, BQ 872 and BS 1198 have both phases. The first phase can be seen as the basic matrix or infill between the iron oxides. If the second phase appears it is found in the form of laths or well-defined crystals.

Complete or partly decomposed sand grains did not show up in any of the samples looked at. This casts doubt on the use of sand as a flux to prevent the oxidation of the surface when forging. This does however explain the absence of typical 'fayalite' laths as seen in many European smithing slags. Sand grains are found on the outer surface, but this is probably due to the fact that iron corrosion products incorporated them. Their presence is thus the result of as secondary process.

Two slags are exceptional, in the sense that they are different from the others. The first is BS 1198A. The microstructure has a resemblance to a smelting slag if only part of it is considered. The grey phase is present in nicely developed crystals, with in-between a finely dispersed infill of the same phase together with a second darker grey phase. Secondary small iron oxide dendrites are also seen in this infill. These structures are however not uncommon in smithing slags that were exposed at high temperatures. The top part of this slag must have formed close to the hottest zone of the hearth, what allowed the material to crystallize in better-developed phases. Moreover the bottom that must have solidified in the cooler region of the hearth does not exhibit these structures.

The second exceptional slag is BS 1110. Almost no iron oxides are present in the slag and two phases clearly appear on the top-side of the slag. An additional phase is found in near the bottom-side. This slag is not to be seen as a metal slag as such, but as part of the lining from the hearth that reacted with the slag. The fact that some iron oxide particle are seen, do show however that it is related to metal working and not just the lining of any hearth that reacted to high temperatures. It is interesting to notice that this is one of the only slags that is not magnetic and the specific gravity is also among the lowest attested.

It can be concluded that none of the slags have a microstructure that allows to identify them as the product of a smelting process. Their inhomogeneous microstructure and often zoned build-up can on the contrary be seen as indications towards an identification as smithing slag. This next to their morphological appearance that already pointed in that direction.

9.4.2. Petrographical analyses

Two fragments, BS 1110 and S 0007, were also submitted at the *Department of Geology and Soil Sciences – Ghent University* and were looked at by Prof. dr. M.A. Elburg. A slice was made of these two fragments in order to examine them under a transmission petrographical microscope, with polarized light. The following paragraph is what she reported, but no pictures were provided to illustrate the findings.

> *"The same minerals largely make up both slags. A first group consists of opaque minerals, which do not provide any information with transmitting light. Silicate minerals make up the second group. A first type of these minerals is a kind of olivine, probably* kirschsteinite *a calcium-iron-olivine ($CaFeSiO_4$). A second type of silicate (of which BS 1110 contains more than S 0007) is probably* melilite*, a transitional series between $Ca_2Al(Al,Si)_2O_7$ and $Ca_2MgSi_2O_7$. Another possibility would be* vesuvianite *that has similar optical properties but is formed at lower temperatures. Both slags have a surface layer of finely dispersed carbonate, but this is a secondary phenomenon. S 0007 has more opaque minerals (iron oxides?) than BS 1110 and primarily has olivine as silicate mineral.* Melilite *is concentrated more at the bottom side. Both slags have a zoned build-up of which the top layer has cooled faster so glass was formed. BS 1110 has some quartz grains included in its top layer."*[76]

The presence of a carbonate film on the surface of the slag was also noted at Mleiha. In order to see if the calcium levels that were measured were biased by that, some slags were cleaned before analyses. The results showed that the calcium levels remained the same and by consequence the calcium was present in the matrix of the slag itself[77], i.e. in the kirschsteinite and melilite minerals.

[76] Pers. comm. Prof. dr. M.A. Elburg.
[77] Ploquin, Orzechowski & Briand, 1999: 176.

9.5. Microstructure & chemical composition – SEM-EDX results

The slag was looked at with an optical microscope to study the homogeneity of the samples. Part of the slag was then imbedded, polished and their mineralogy and petrography were examined under the SEM-EDX. The bulk composition of the slag was calculated by taking the average of three or more 'large surface' analyses by EDX. This in turn is supplemented by the analyses of specific phases and/or inclusions. As mentioned above the slag samples were rather difficult to analyse in the SEM-EDX. The porosity of the slag made it difficult to make a vacuum in the sample chamber of the SEM and it often took up to two hour to reach the minimal vacuum circumstances. The fact that slags are poorly conductive made it necessary to coat them with a thin layer of gold in order to prevent the sample from charging and giving biased measurements.

The EDX software gives the results of the measurements in concentration weight percentages. The results of the elements analysed for are, including oxygen, normalized to 100%. In many publications the composition of slag is however given by the amount of the oxides of the detected elements. Therefore a basic conversion to the amount of oxides that could be present is given. This is however analytically not completely precise, since it is impossible to know which oxides or oxide forms are present. Iron can be present in its metallic form or in any or a combination of their oxidised states, i.e. as wüstite (FeO), as hematite (Fe_2O_3), as magnetite (Fe_3O_4) or a hydroxide form. It should be mentioned that wüstite is actually a non-stoichiometric compound and the ratio Fe to O cannot be well-defined in natural numbers. The actual stoichiometry is closer to $Fe_{0,95}O$ and for each missing Fe^{2+}-ion, the crystal contains two Fe^{3+}-ions to balance the change and a more correct way of writing the chemical formula is $Fe_{1-x}O$.

Oxide form	Chemical formula	wt% Fe	wt% O	Extra
Metallic iron	Fe	100,00	-	
Wüstite	FeO	77,62	22,38	
Hematite	Fe_2O_3	69,81	30,19	
Magnetite	Fe_3O_4	72,23	27,77	
Hydroxide form	FeO(OH)	63,43	36,57	H is not detected by EDX and not evaluated

Table 48: Ideal composition of the different oxides that should be detected by EDX in wt%.

In smithing slag the iron oxide is generally identified as wüstite although higher oxides do occur, but this may be due to post depositional oxidation[78]. In many publications FeO is therefore used as the basic iron oxide present, a suggestion followed here since the microstructure of the oxide is consistent with published microstructures of wüstite. The more massive form of the iron oxides present may however be magnetite.

The calculation is as follows: the concentration weight percentage was converted to the number of moles of each element present. These were combined with the necessary moles of oxygen to produce the expected oxide (i.e. FeO is a combination of 1 mol of Fe with 1 mol of O). These results were then again normalized to 100%. An important drawback is that oxygen is a light element and the results obtained by EDX are not always reliable. Still an satisfactory stoichiometric equation was often found, the amounts of oxygen not always in perfect balance with the other elements but acceptable[79]. This conversion was needed in any case to make the data more accessible for comparison to other published data. It is not always clear how the amount of oxides is calculated in other studies and the long-term corrosion of slags can also lead to the partial oxidation of some of the minerals[80].

[78] McDonnell, 1984: 48.
[79] My sincere gratitude goes out to B. Bashar for showing me how to tackle this problem, P. Mast to somewhat confusing this solution and P. Gobernado Hernandez for providing the necessary third opinion, coinciding with the first.
[80] Craddock, 1995: 17.

The analyses will be treated in greater detail and per slag underneath and average compositions of the total slag and the different phases present will be given. It is important however to keep in mind that slags are very heterogeneous in their composition and that compositions can differ greatly from one area to another. The only way to get the real global composition of a slag would be to completely dissolve the specimen and measure the elements present. This was not done in this study. Moreover the visual information obtained by SEM largely compensates the semi-quantitative character of the analyses, since different phases can be analysed individually. Whatever the two digits behind the comma might suggest, the results are only an indication of the composition and are not quantitative.

• **BO 716**

BO 716 is a small piece of slag (*ca*. 2,5 X 2 cm, specific gravity: *ca*. 2,5 g/cm³). That difference from many of the other slags in that it has a low iron oxide content. Fig. 132-1 shows these are present in two distinct forms: finely dispersed wüstite dendrites (1) and a larger more angular form that might be magnetite (2). Two different large crystal forms are also attested and iron oxides are often concentrated around the grain boundaries. One type of crystals appears as a darker grey phase (3), whereas the other is lighter grey (4) (Fig. 132-2). Their main compositional difference is that the lighter phase has a much higher amount of iron oxide and lower amounts SiO_2 and CaO present. The light grey phase is grown around the dark grey one and a certain amount of coring appears in the light grey phase, due to an inhomogeneous iron distribution. The brighter areas contain more iron. A similar amount of MgO is found in both phases. In-between these large crystals a mixture of both is finely dispersed within a black glassy interstitial phase (5). Metallic iron particle do not seem to appear in this slag.

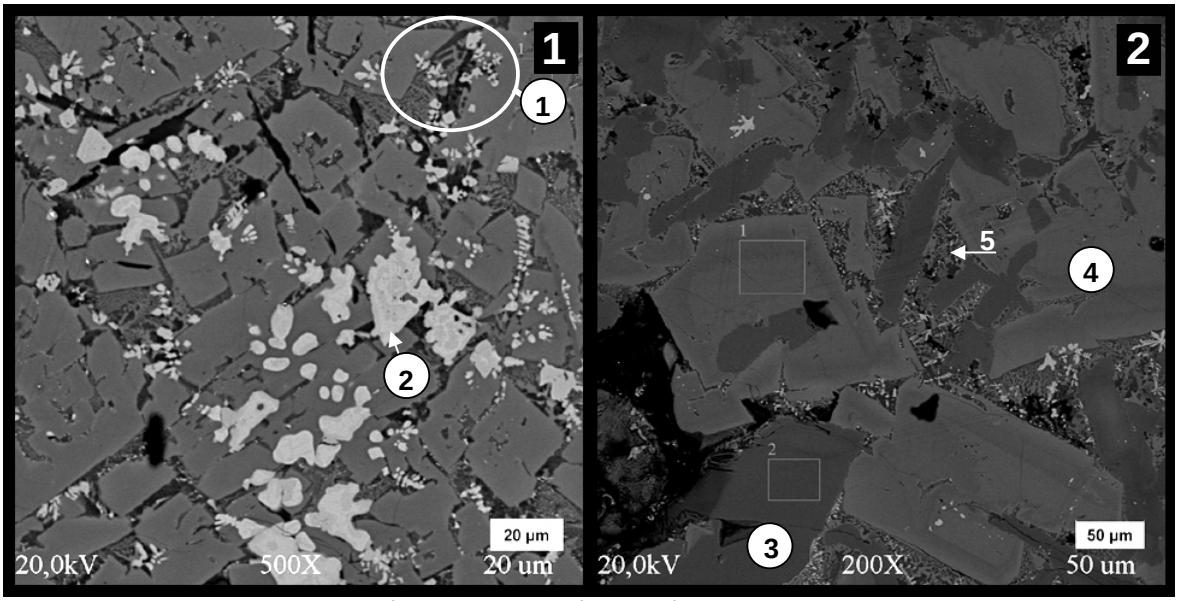


Fig. 132: SEM-BSE images of BO 716.

Phases	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	CrO_x	FeO
Dark grey phase (3)	-	7,02	-	44,13	-	-	-	45,05	-	-	3,80
Light grey phase (4)	-	6,29	-	37,89	-	-	-	40,59	-	-	15,24
Average grey phase	-	6,65	-	41,01	-	-	-	42,82	-	-	9,52
Iron oxide (1 & 2)	-	0,54	0,48	2,39	-	-	-	2,38	-	-	94,21
Average composition	-	*3,16*	*2,16*	*42,51*	-	-	*1,08*	*37,74*	-	-	*13,31*

Table 49: Compositional data of different phases and global composition in oxide wt% of BO 716 (**-** : not present).

As a whole this slag shows many similarities with certain zones of BS 1198A and BS 1110 as seen in the part on the optical microscopy (see above), and BS 1215 (below). They are probably the result of a reaction between the hearth lining and the forming slag, this explains the presence of relatively high amounts of MgO, Al_2O_3 and K_2O in the average composition.

• **BO 845A**

This slag was already discussed in the section on the optical microscopy, where three different grey phases and dark grey inclusions were observed. BO 845A has an high overall iron oxide content and the small white inclusions seen on Fig. 133-1 are metallic iron particles. The iron oxide grains are large and only a fine network of another phase is seen between them. This is an image from the top side of the slag. Fig. 1333-2 shows the three phases together with rounded dark grey inclusions (3) that contain SiO_2 and CaO. The light grey matrix is iron oxide phase (1) with only a small amount of CaO. In that matrix two phases can be distinguished, becoming darker (2 & 4) when the iron content is lower and the CaO content is higher. The three phases correspond with the three phases seen under the optical microscope (see above, they are designated the same numbers on the illustrations), but their ratios differ in the pictures.

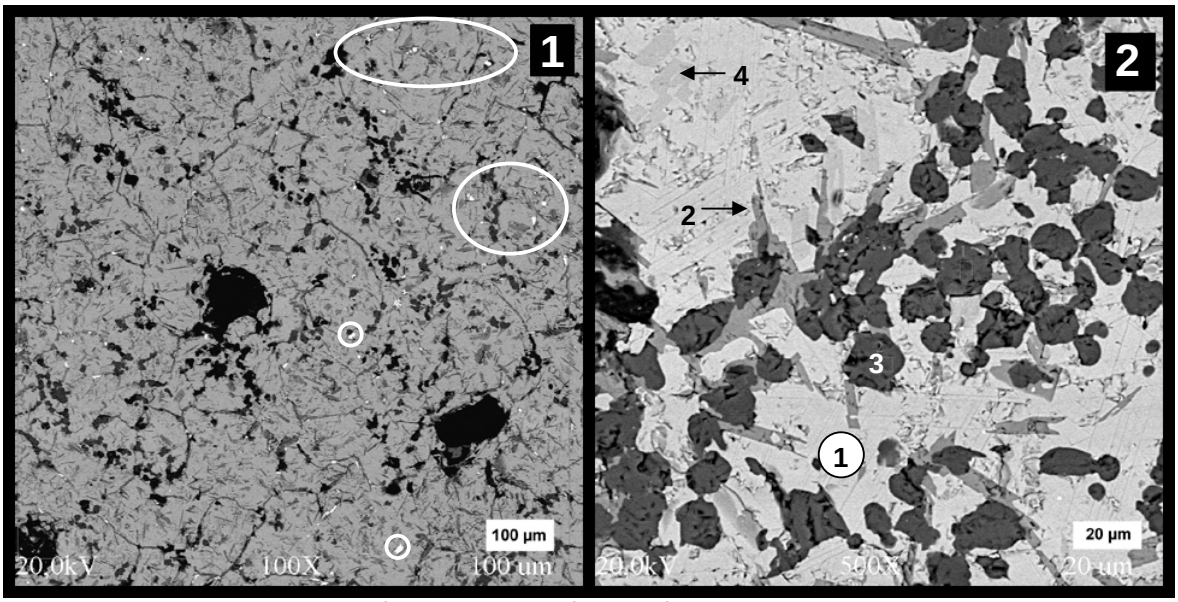


Fig. 133: SEM-BSE images of BO 845A.

Phases	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	CrO_x	FeO
Dark grey phase (2)	-	-	-	-	-	-	-	57,57	-	-	42,43
Iron oxide (Ca-rich) (4)	-	-	-	-	-	-	-	19,10	-	-	80,90
Iron oxide (1)	-	-	-	-	-	-	-	1,92	-	-	98,08
Inclusions (3)	-	-	-	37,00	-	-	-	62,29	-	-	0,71
Average composition	-	-	-	*0,85*	-	-	-	*13,28*	-	-	*85,87*

Table 50: Compositional data of different phases and global composition in oxide wt% of BO 845A (**-** : not present).

• **BQ 819**

This slag was already discussed in the section on the optical microscopy. Fig. 134-1 shows the inhomogeneous of this slag. The round inclusions (1) at the top are 'sand' grains incorporated in an iron oxide matrix. Large laths of grey crystals appear (2) next to large globular iron oxides (3) and finer dendrites (4). Fig. 134-2 is an enlargement of a zone in-between the grey laths (2) and shows an infill of the same phase and a black glassy phase (5). Some of the iron oxides include some SiO_2 and CaO, whereas in others only oxygen and

iron was detected. The glass infill has Na_2O, Al_2O_3, SO_3, K_2O not attested in the other phases.

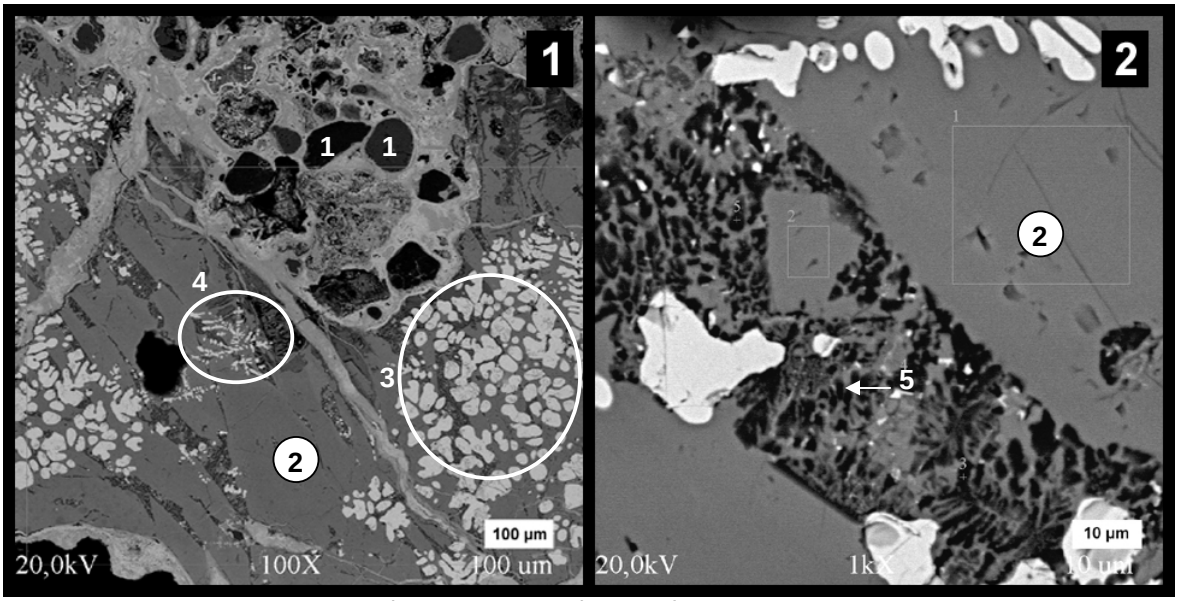


Fig. 134: SEM-BSE images of BQ 819.

Phases	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	CrO_x	FeO
Black inclusions in glass infill (5)	4,92	-	12,71	56,31	-	3,43	7,01	9,46	-	-	6,17
Grey lath phase (2)	-	-	-	38,12	-	-	-	37,51	-	-	24,37
Iron oxide (+Si)	-	-	-	3,85	-	-	-	0,73	-	-	95,42
Iron oxide	-	-	-	-	-	-	-	-	-	-	100,00
Average composition	-	-	-	*23,67*	-	-	-	*14,93*	-	-	*61,40*

Table 51: Compositional data of different phases and global composition in oxide wt% of BQ 819 (**-** : not present).

- **BQ 872**

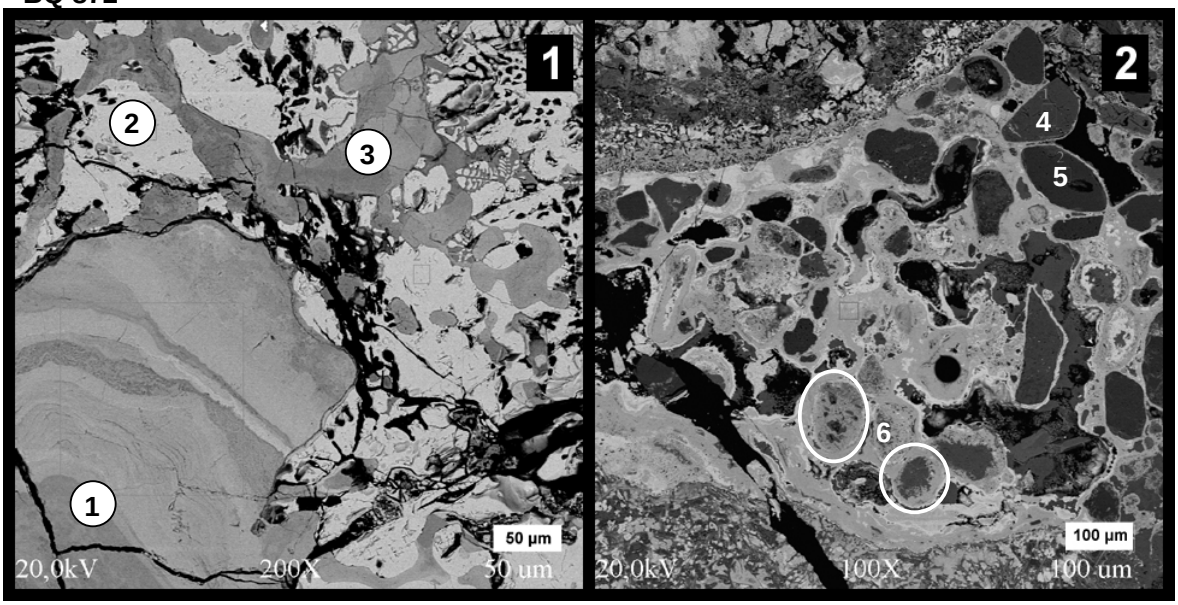


Fig. 135: SEM-BSE images of BQ 872.

This slag was already discussed in the section on the optical microscopy. It is slag rich in iron oxide. Fig. 135-1 only shows iron oxides in different states of oxidation. The large zoned area is probably a partly decomposed form and probably an iron hydroxide[81] (1). The other grey areas are iron oxides that contain a various amount of SiO_2 and CaO (2 & 3). The inclusions in Fig. 135-2 are 'sand' in an iron oxide matrix and the white rim around them shows only limited decomposition by the attack of the slag (4 & 5). Other grains might be the remnants of the original grain almost completely absorbed by the slag (6), if so this is the only instance encountered where this is seen.

Phases	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	CrO_x	FeO
Iron oxide (2)	-	-	-	0,45	-	-	-	4,99	-	-	94,57
Iron oxide (+Si) (3)	-	-	-	14,07	-	-	-	3,18	-	-	82,75
'Sand' (4)	-	-	22,88	66,07	-	-	-	10,05	-	-	1,00
'Sand' (5)	-	-	-	100,00	-	-	-	-	-	-	-
Average composition	-	-	-	*7,42*	-	-	-	*6,64*	-	-	*85,94*

Table 52: Compositional data of different phases and global composition in oxide wt% of BQ 872 (**-** : not present).

- **BQ 875**

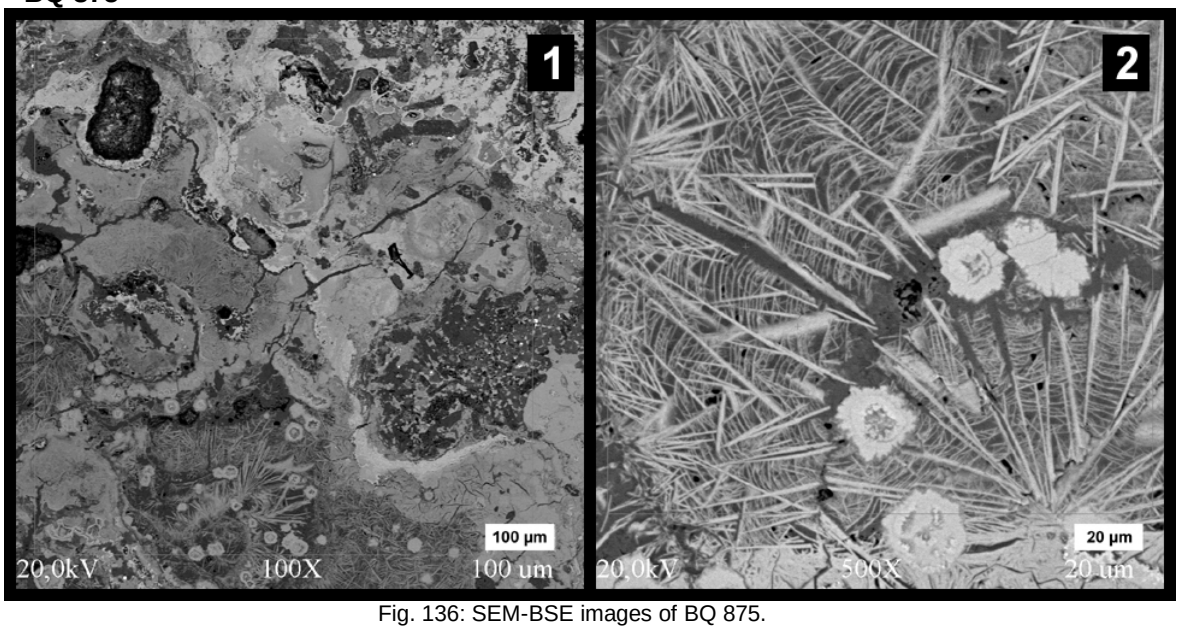


Fig. 136: SEM-BSE images of BQ 875.

Phases	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	CrO	FeO
White phase 'feathered' structure	-	-	-	10,20	-	-	-	19,90	-	-	69,90
Grey phase 'feathered' structure	-	-	-	-	-	-	-	96,01	-	-	3,99
Average 'feathered' structure	-	0,51	-	2,79	-	-	-	57,75	-	-	38,95
Average composition	-	-	-	*1,92*	-	-	-	*31,72*	-	-	*66,37*

Table 53: Compositional data of different phases and global composition in oxide wt% of BQ 875 (**-** : not present).

BQ 875 is a complete slag (*ca*. 6 x 4,5 cm, specific gravity: *ca*. 2,8 g/cm³) and has a very 'messy' microstructure (Fig. 136-1) with a rather high overall iron oxide content. A particular microstructure was seen in, and only in, BQ 875, described as 'feathered' (Fig. 136-2). It shows white iron rich needless in a grey CaO matrix. No comparable structures were found in the literature and no explanation for the origin of this microstructure can be offered.

[81] Bachmann, 1982: 33.

• **BQ 1008B**

BQ 1008B (*ca*. 6 x 6 cm, specific gravity: *ca*. 3,6 g/cm³) is a rather complex slag and shows great variation in the composition of different phases.

Fig. 137-1 shows an overview of an iron oxide-rich part of the top of the slag. On the other pictures (Fig. 137-2, Fig. 137-1 & 2) the numbers 1, 4 and 7 indicate iron oxides. They do however differ considerably in the other elements detected. The presence of Cr and Ti are rather exceptional and point towards the use of an original iron ore that contained high values of both elements. The sources of the Ti could be the Ti-rich inclusions (5 on Fig. 137-1). The MgO levels are also amongst the highest found in any of the slags iron oxides (BS 1264 also has 5,4 % of MgO). As a whole the high values of other elements in the oxides is atypical for the slag looked at here. Also the grey phases have a specific feature in that they contain phosphorous, up to 10%.

Phases	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	CrO_x	FeO
Grey phase (P-poor) (8)	0,22	1,81	0,32	36,87	1,00	-	1,06	51,65	0,20	-	6,87
Grey phase (P-medium) (2)	-	1,39	-	32,41	5,91	-	3,68	53,81	-	-	2,80
Grey phase (P-rich) (3)	-	1,68	-	35,94	10,63	1,58	-	46,27	-	-	3,90
Grey inclusions (Ti-rich) (5)	-	-	-	22,24	0,70	-	0,70	50,47	18,42	-	7,48
Dark grey phase (6)	-	0,34	1,66	45,51	5,55	3,49	1,51	34,12	0,68	-	7,15
Iron oxide (Mg-rich) (1)	-	27,61	-	-	-	-	-	0,86	0,76	-	70,77
Iron oxide (Si & Ca-rich) (7)	-	5,78	1,01	7,97	-	-	0,66	6,73	1,45	0,69	75,71
Iron oxide (Mg-poor) (4)	-	6,35	-	-	-	-	-	0,70	1,13	0,58	91,25
Average oxide	-	*11,76*	*0,40*	*3,38*	-	-	*0,26*	*3,02*	*0,92*	*0,34*	*79,92*
Average composition	-	*0,74*	-	*16,21*	-	-	-	*15,24*	-	-	*67,81*

Table 54: Compositional data of different phases and global composition in oxide wt% of BQ 1008B (**-** : not present).

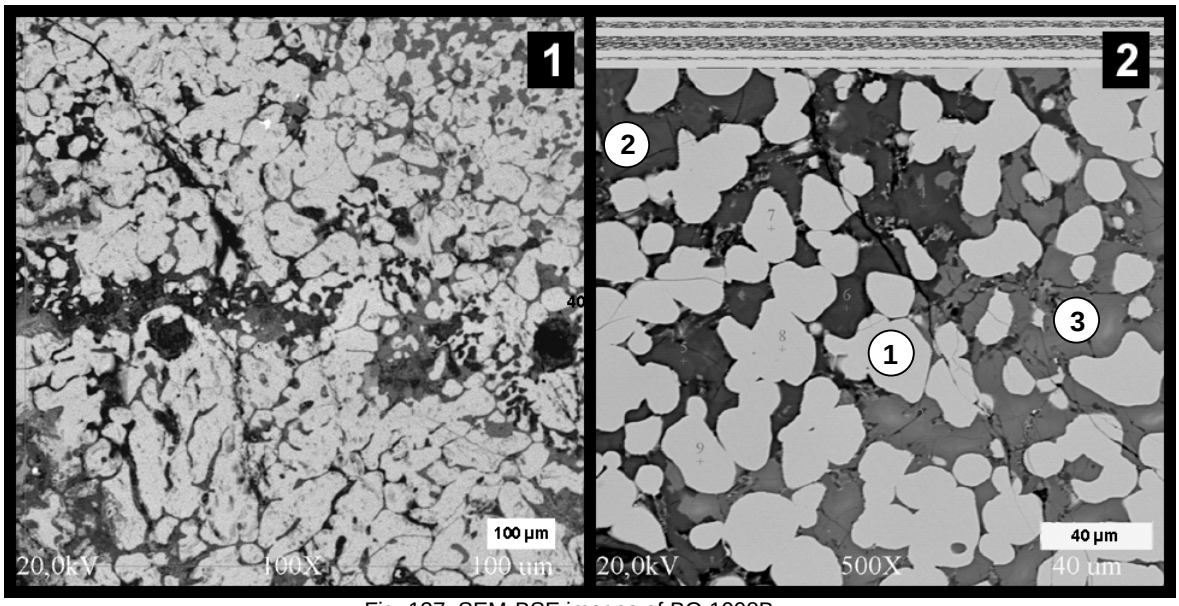


Fig. 137: SEM-BSE images of BQ 1008B.

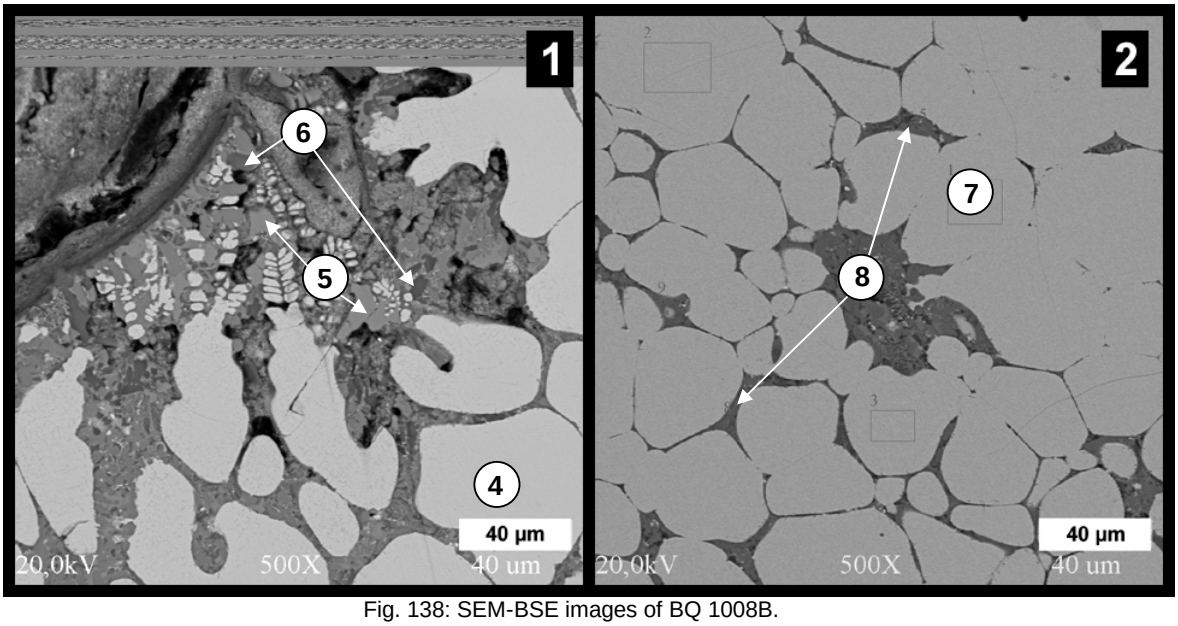


Fig. 138: SEM-BSE images of BQ 1008B.

• **BS 1068**

BS 1068 (*ca*. 6 x 2 cm, specific gravity: *ca*. 2,5 g/cm³) has large iron oxides present in the form of large tiles (Fig. 139-1) at the top and in a more semi-dendritic form towards the bottom of the slag (Fig. 139-2). The ground matrix exists out of laths and a mixed infill. The iron oxides have only a small amount of other elements present. The laths are rich in CaO and SiO_2.

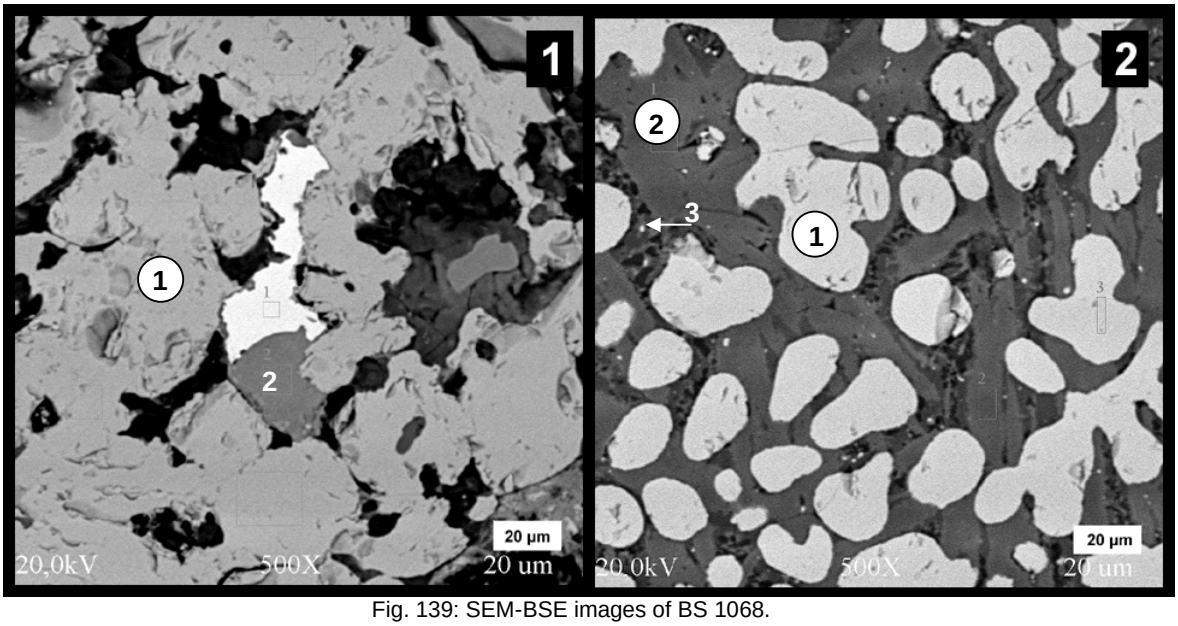


Fig. 139: SEM-BSE images of BS 1068.

Phases	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	CrO_x	FeO
Dark grey phase (2)	0,33	0,84	-	28,61	1,20	-	0,68	57,30	-	-	11,03
Iron oxide (1)	-	-	-	0,69	-	-	-	1,92	-	-	97,40

Table 55: Compositional data of different phases in oxide wt% of BS 1068 (**-** : not present).

- **BS 1110**

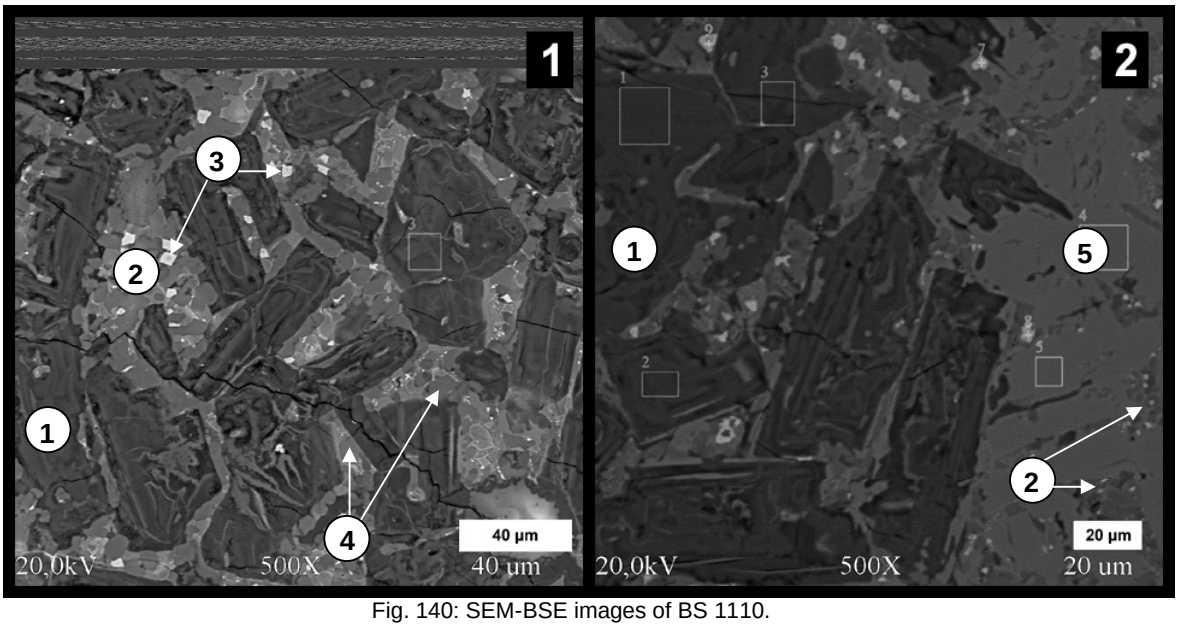


Fig. 140: SEM-BSE images of BS 1110.

This slag was already discussed in the part on the optical microscopy. BS 1110 is a fragment of slag that has a glassy bulb at the surface and has a high amount of ceramic components, originating from the 'hearth lining', this actually is a piece of the hearth lining and as such very different from the other slags discussed. Two very different crystals appear, one is dark and the other light (Fig. 140-1 & 2, resp.1 & 5). Their main difference is the SiO_2/CaO ratio, where the dark phase much more SiO_2 included, next to a higher MgO level. Also here Ti-rich inclusions are found, but they are associated with high iron content, whereas in slag BQ 1008B the Ti-rich inclusions have lower Fe-levels. The small grey inclusions contain Na_2O, MgO, Al_2O_3, SiO_2, K_2O and some CaO, what would be consistent with some form of clay. The infill indicated by (2) can be seen as a mixture of the small grey inclusions, and the dark and light grey crystals, reflected in an 'average' composition.

Phases	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	CrO_x	FeO
Small grey crystals (4)	2,99	3,89	27,02	57,43	-	-	6,61	1,39	-	-	0,67
Grey phase (5)	-	11,04	1,58	47,23	-	-	0,33	38,20	-	-	1,54
Dark crystals (1)	-	4,55	5,85	81,55	-	-	0,77	5,07	0,03	-	2,18
Infill (2)	1,03	4,48	13,26	55,64	-	-	4,57	15,14	0,34	-	5,54
Fe-Ti-rich inclusions (3)	-	11,73	10,18	5,06	-	-	0,49	1,32	17,20	3,39	50,62
Average composition	*0,25*	*7,60*	*1,78*	*50,85*	-	-	*0,59*	*34,67*	-	-	*4,26*

Table 56: Compositional data of different phases and global composition in oxide wt% of BS 1110 (**-** : not present).

To evaluate the concentration of the different elements detected visually, a mapping was done of one area (Fig. 141). This shows that the grey laths (5 on Fig. 140-2) are mainly composed of Ca, Si and some Mg. The infill between (2 on Fig. 140-2) contains also contain Si, Mg and to a lesser extent Ca, but the major difference is the exclusive occurrence of K and Al. Fe is only present to a very limited extent and many concentrated in some iron inclusions in-between. The mapping is in accordance with the different composition presented in Table 56 for the light grey phase and the infill.

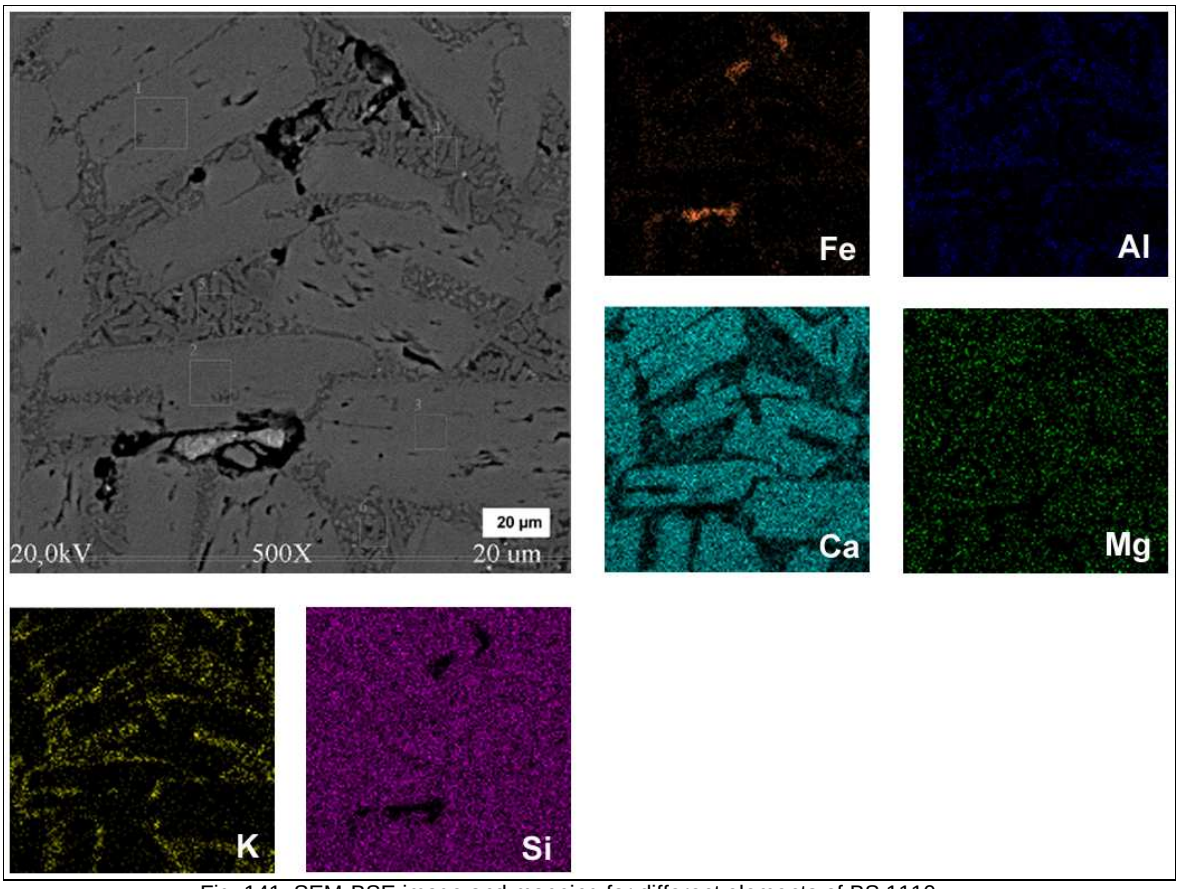


Fig. 141: SEM-BSE image and mapping for different elements of BS 1110.

- **BS 1185A**

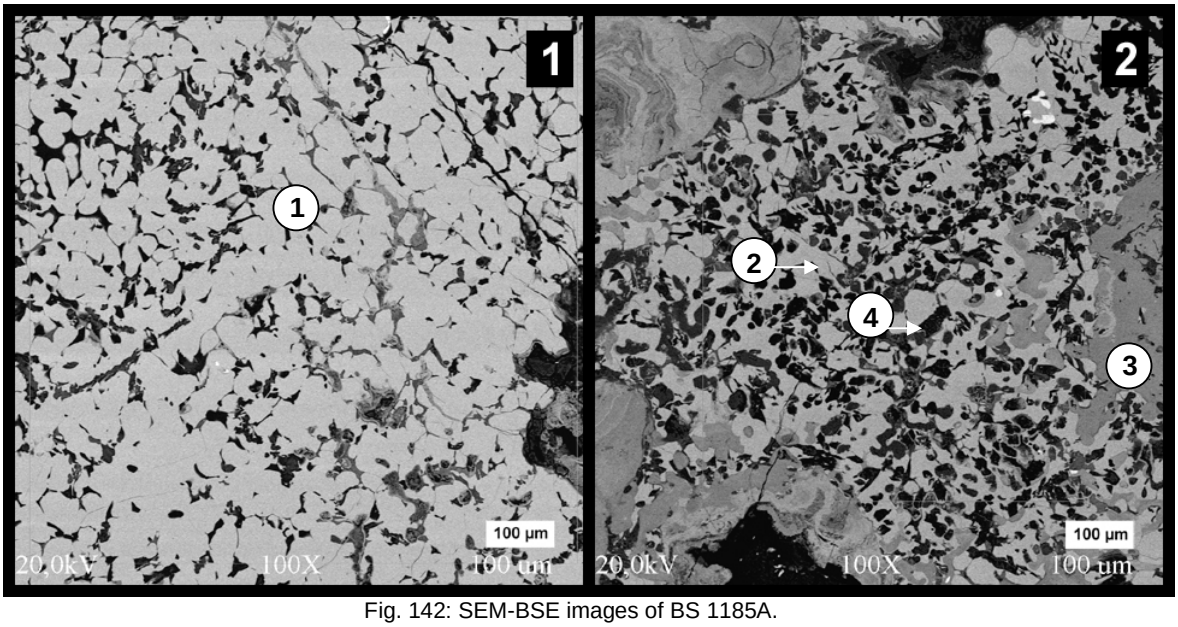


Fig. 142: SEM-BSE images of BS 1185A.

.

BS 1185A (*ca*. 4 x 2 cm, specific gravity: *ca*. 3,2 g/cm³) is an iron oxide-rich slag with a clear zoning. Fig. 142-1 is situated at the top of the slag and Fig. 142-2 near the bottom. The main difference is the presence of the black inclusions. Their occurrence cause higher levels of MgO, SiO_2 and CaO in the lower region of the slag.

Phases	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	CrO_x	FeO
Iron oxide (1 & 2)	-	-	-	-	-	-	-	7,83	-	-	92,17
Iron oxide (+Si) (3)	-	-	-	10,61	-	-	-	2,79	-	-	86,60
Black inclusions (4)	-	16,07	-	59,45	-	-	1,47	9,71	-	-	13,30
Average zone without black inclusions ~ Fig. XX-1	-	*4,04*	-	*8,24*	-	-	-	*8,89*	-	-	*78,84*
Average zone with black inclusions ~ Fig. XX-2	-	*5,13*	-	*15,79*	-	-	-	*11,29*	-	-	*67,78*
Average composition	-	*4,58*	-	*12,02*	-	-	-	*10,09*	-	-	*73,31*

Table 57: Compositional data of different phases and global composition in oxide wt% of BS 1185A (**-** : not present).

- **BS 1198A**

This slag was extensively discussed in the section on the optical microscopy. Due to the inattention of the researcher (*mea culpa*) this slag was not studied with the same thoroughness by SEM-EDX. Only one phase of the two phases seen under the optical microscope is attested. The black inclusions are rich in Na_2O, Al_2O_3, SiO_2 and K_2O, whereas the light grey phase has a high level of MgO and CaO.

Phases	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	CrO_x	FeO
Black inclusions (1)	3,96	-	27,50	50,50	-	-	12,26	3,85	-	-	1,94
Light grey phase (2)	-	10,47	0,41	38,50	-	-	-	34,98	-	-	15,64
Iron oxide (3)	-	-	-	-	-	-	-	1,28	-	-	98,72

Table 58: Compositional data of different phases in oxide wt% of BS 1198A (**-** : not present).

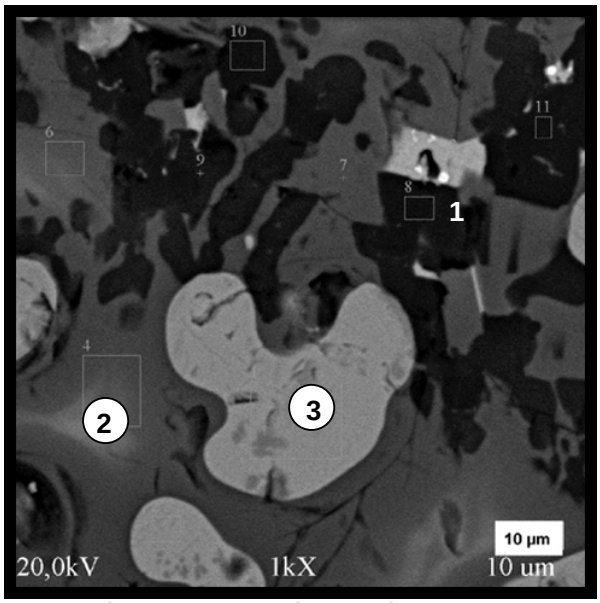


Fig. 143: SEM-BSE images of BS 1198A.

- **BS 1215**

BS 1215 (*ca*. 6 x 4,5 cm, specific gravity: *ca*. 2,4 g/cm³) exhibits a similar microstructure as BO 716 (Fig. 132-1) and the description given there can be copied for this specimen. The slag has part of the lining still attached hence the low specific gravity of the slag and shows a structure similar to smelting slag. The results for a similar grey phase (Fig. 144, ***1*** & ***4***) show a variation in the amount of MgO and FeO present, but their main characteristic seem to be the presence of MgO, not seen in the dark grey phase.

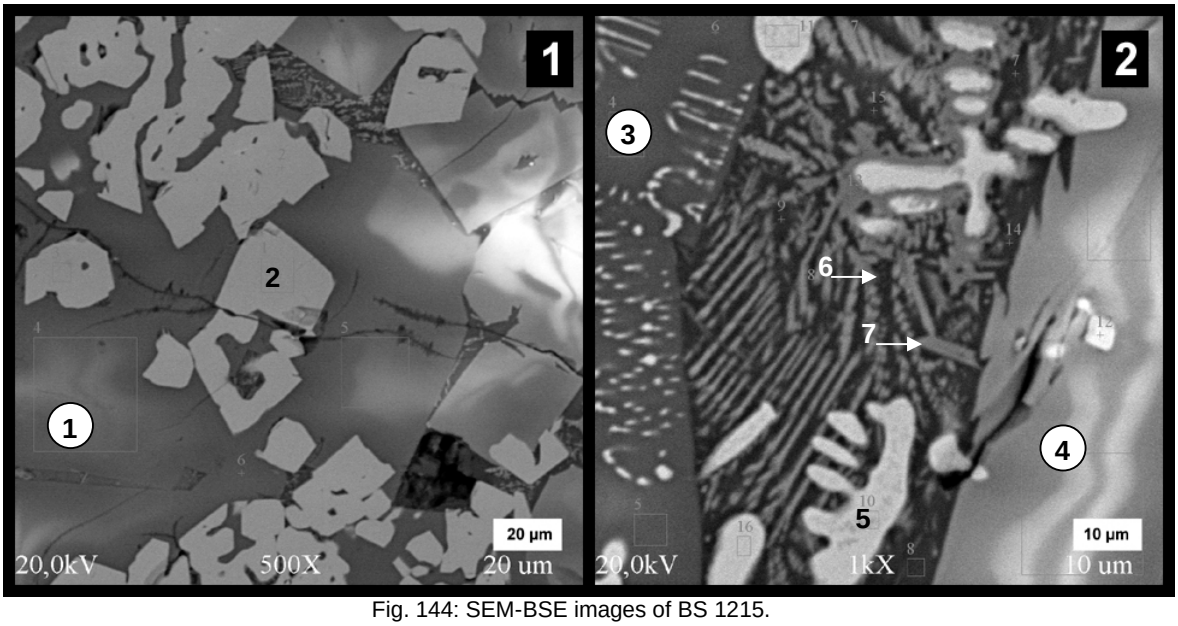


Fig. 144: SEM-BSE images of BS 1215.

Phases	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	CrO_x	FeO
Light grey phase (1)	-	11,37	-	45,08	-	-	-	42,28	-	-	1,27
Dark grey phase (3)	-	-	-	49,74	-	-	-	41,11	-	-	9,14
Light grey phase (4)	-	5,04	-	47,70	-	-	-	39,54	-	-	7,71
Light grey phase infill (7)	0,25	3,00	2,82	45,08	-	-	0,48	28,78	-	-	19,59
Glass (6)	2,32	-	17,79	56,85	-	-	4,57	10,29	-	-	6,96
Iron oxide (2 & 5)	-	-	3,10	3,23	-	-	-	2,63	0,67	0,11	90,26

Table 59: Compositional data of different phases in oxide wt% of BS 1215 (**-** : not present).

- **BS 1264**

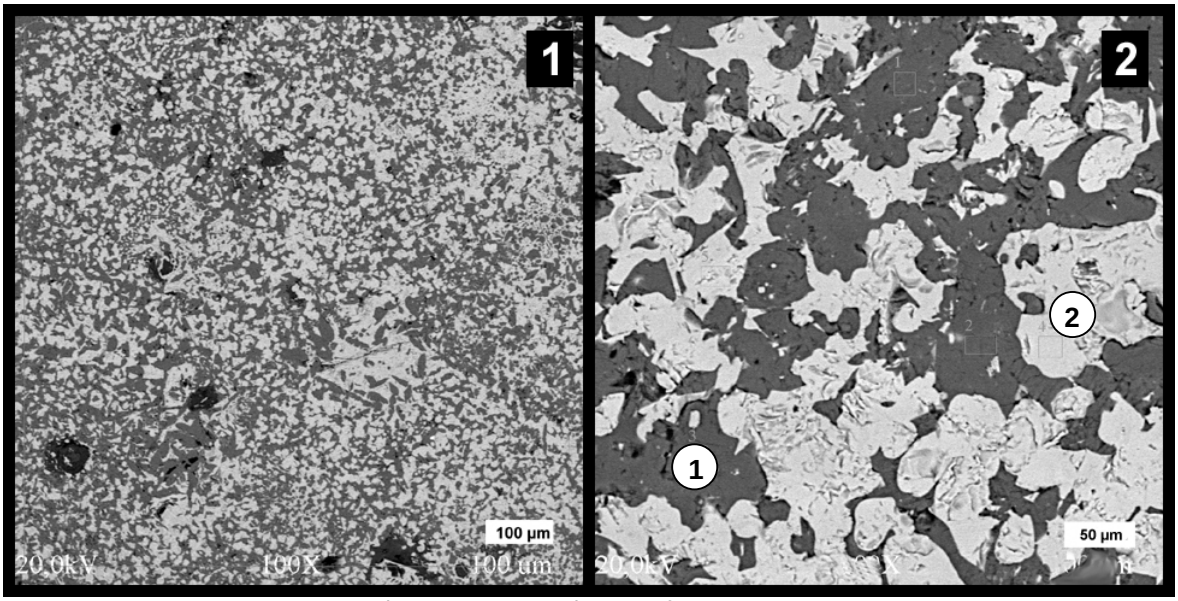


Fig. 145: SEM-BSE images of BS 1264.

BS 1264 is a fragment of a slag of which the section has a dark grey colour and was already discussed in the section on the optical microscopy. The upper part contains more iron oxides than the lower part, which has more vacuoles. Fig. 145-1 belongs to the middle part of the

slag. BS 1264 only exhibits two different phases. The light grey phase are iron oxides that occur in certain areas as the typical globules, but in others are much more irregular in shape. The overall iron content is low and the dark grey infill in rich in Ca and Si.

Phases	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	CrO_x	FeO
Dark grey phase (1)	-	-	-	35,04	-	-	-	63,61	-	-	1,35
Iron oxide (2)	-	5,45	-	-	-	-	-	3,10	-	-	91,45
Average composition	-	-	-	*24,07*	-	-	-	*41,97*	-	-	*33,97*

Table 60: Compositional data of different phases and global composition in oxide wt% of BS 1264 (**-** : not present).

- **KR 002**

Slag KR 002 comes from Khor Rori and was found together with some others, in a architectural structure that was defined by the excavators as a smithing workshop. A hearth structure in the corner of the building is seen as a smithing hearth (Fig.146). The fill was ashy grey and yielded a small fragment of slag and a small piece of iron. The absence of slag inside the hearth does not be a surprise in that a more permanent hearth would have been cleaned out on regular bases. The hole on the side might be the inlet for the air blast provided from a type of bellows (see inset in Fig. 146).

Fig. 146: 'Hearth structure' excavated at Khor Rori.

The global analysis of the slag shows that a high amount of iron oxide is present, in the form of wüstite and/or magnetite. The grains are big and arranged closely together with only a little glassy infill in-between, resulting in low Si and Ca content. The slag shows a clear zoning where the top is much richer in iron oxides and the bottom-side shows much more black inclusions (respectively Fig. 147-1 and 2). The higher amounts of Mg, Al, Si and Ca in the lower part of the slag are due to the reaction with the forming slag and the ashes and/or lining of the hearth. The presence of S seem to be related with the black inclusions.

Unique for this slag is that the black inclusions contain Cu and S. C.J. Salter reported a similar phenomenon in the slag from Qal'at al-Bahrain[82]. The occurrence of such inclusions can be the result of a small scale crucible copper smelting or refining process. The low amount of copper in these inclusions and the absence of any metallic copper particles does

[82] Salter, 1994: 383-384.

however exclude this option and it is very unlikely that this slag is in any way related to a copper working process. It may however point to the fact that copper was worked in the same workshop as iron, and that small bits may have ended up in the hearth reacting with the rest of the slag. A hypothesis underpinned by the finding at Khor Rori of at least two small crucibles used to melt copper or one of its alloys[83].

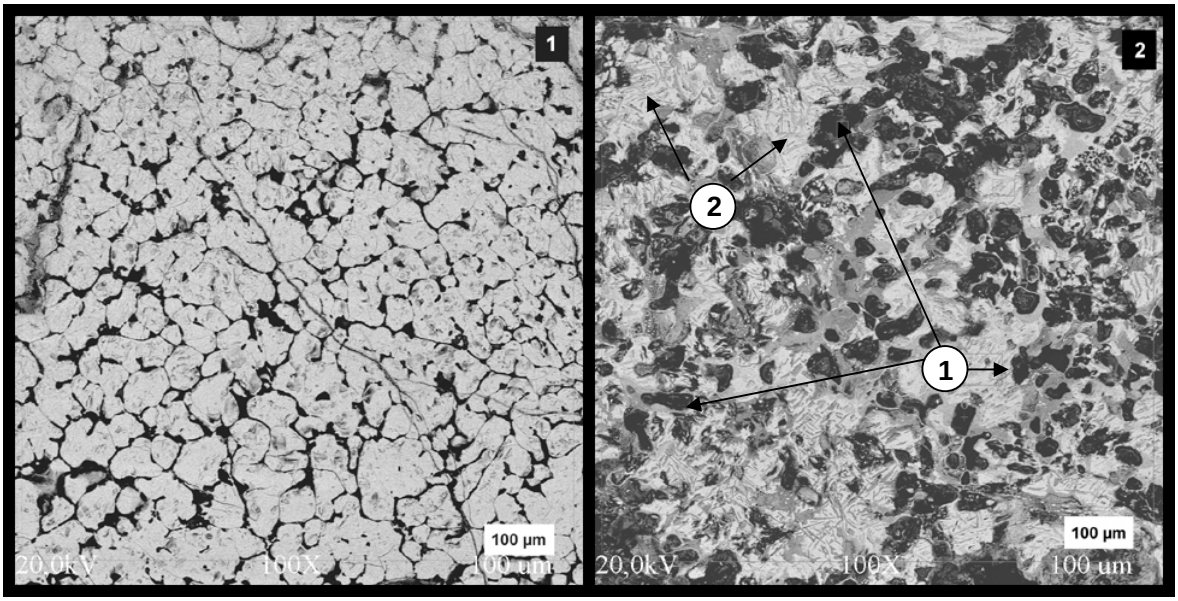


Fig. 147: SEM-BSE images of KR 002.

Phases	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	CrO_x	FeO	CuO
Dark inclusions (1)	-	-	4,96	7,76	-	1,47	-	6,71	-	-	78,24	0,87
Iron oxide (2)	-	2,76	-	1,54	-	-	-	1,80	-	-	93,90	-
Average zone top ~ Fig. XX-1	-	*0,64*	-	*4,38*	-	-	-	*3,46*	-	-	*91,52*	-
Average zone bottom ~ Fig. XX-2	-	*3,35*	*0,67*	*9,24*	-	*2,52*	*0,17*	*10,37*	-	-	*73,67*	-
Average composition	-	*1,65*	-	*6,73*	-	*0,84*	-	*6,08*	-	-	*84,41*	-

Table 61: Compositional data of different phase and global composition in oxide wt% of KR 002 (**-** : not present).

[83] A. Pavan pers. comm.

• **Discussion & conclusions**

An overall view of the results is shown in Table 62. It lists the oxides attested, the number of occurrences throughout all phases, the number of slags where a certain oxide was detected, the minimum and maximum amount of this oxide and the registration numbers of the slags under analysed. The different oxides will be discussed individually and in relation to each other underneath.

Oxide	# times	# slags	Min. amount in wt%	Max. amount in wt%	Slag nr.'s
Na_2O	8	6	0,2	4,9	BQ 819 – BQ 1008B – BS 1068 – BS 1110 – BS 1198A – BS 1215
MgO	26	10	0,3	27,6	BO 716 – BQ 875 – BQ 1008B – BS 1068 – BS 1110 – BS 1185A – BS 1198A – BS 1215 – BS 1264 – KR 002
Al_2O_3	17	8	0,3	27,5	BO 716 – BQ 819 – BQ 872 – BQ 1008B – BS 1110 – BS 1198A – BS 1215 – KR 002
SiO_2	40	All	0,5	81,5	All
P_2O_5	6	2	0,7	10,6	BQ 1008B – BS 1068
SO_3	4	3	1,5	3,5	BQ 819 – BQ 1008B – KR 002
K_2O	16	7	0,3	12,2	BQ 819 – BQ 1008B – BS 1068 – BS 1110 – BS 1185A – BS 1198A – BS 1215
CaO	49	All	0,2	96,0	All
TiO_2	9	3	0,2	18,4	BQ 1008B – BS 1110 – BS 1215
CrO_x	2	2	0,7	3,4	BQ 1008B – BS 1110
FeO	50	All	0,7	100,0	All

Table 62: Summery of the oxides attested.

• Na_2O

Sodium was only detected a few times. It always appears in the infill between large crystals or in small inclusions and could be related to the glassy phase. It is associated with elevated levels of K_2O and Al_2O_3. Na_2O was also detected in the interstitial phase in the slag from Qal'at al-Bahrain, likewise connected with elevated K_2O and Al_2O_3 levels[84]. The sodium could originate from the ash of the charcoal or from natural salt crystals present in the soil, after-all ed-Dur is situated next to the sea. The association with potassium and aluminium could also suggest that a clay containing lining introduced it.

• MgO

The occurrence of MgO can be split up in three different cases:

- In the first case it is attested in the iron oxides, with values around 5%, although one exception is to be noticed (BQ 1008B), where a very high value of 27% was measured.

- The second phase often associated with MgO are the grey laths with amounts between 5 and 12%. The same crystals occur in BO 716, BS 1198A and BS 1215. BS 1068 also has similar laths, but the MgO levels are rather low. The three slags first mentioned have in common that they show a microstructure resembling smelting slags. This indicates that the slag, or part of it, was formed at a higher temperature and that the mineralogical phase containing MgO can only appear when high enough temperature are reached. BS 1110 is non-magnetic and is part of the original 'hearth lining', but the morphology of the MgO containing phase is similar to that seen in the slag, i.e. grey lath or angular tile-shaped.

[84] Salter, 1994: 383.

- In two cases elevated MgO levels were seen associated with inclusions. For BS 1110 this is with the Fe-Ti-rich inclusions and for BS 1185A with black inclusions incorporated in the bottom of the slag. A similar phenomenon is seen in the KR 002. The MgO amount is higher in the bottom part of the slag. Also there inclusions are seen, but in the ones analysed no MgO was detected.

Possible contributors of MgO can be the charcoal ash that can contain between 2 and 11% of MgO[85] or the hearth lining used.

• Al_2O_3
If Al_2O_3 is found in iron oxides the values are always low, with an exception in slag BS 1215 where up to 3% is present. The Fe-Ti-rich inclusions in BS 1110 also have a rather high quantity of Al_2O_3 (5%).

Most often the Al_2O_3 is however seen in the glassy infill between the larger crystals (BS 1215 & BQ 819), inclusions (BS 1198A, BQ 1008B & KR 002) of the slag or in one type of crystal (BS 1110). The association with the interstitial phase is also seen in the analyses of the slag from Qal'at al-Bahrain.

The hearth lining most probably introduces aluminium, as can be seen in BS 1110 that has high levels of Al_2O_3. The occasionally associated with elevated levels of MgO, suggesting that both could be associated with a contribution to the slag by the hearth lining.

• SiO_2
Fragments of free silica do occur in the form of sand grains, but this is due to post-depositional processes, where sand got stuck in the oxides forming at the surface due to weathering. Only in one instance sand grains might be included inside the slag (BQ 872), most show little evidence of chemically attack by the surrounding slag material before solidification had occurred.

The iron oxides can contain up to 15% of SiO_2 till as little as 0,5%. The other phases have a varying level between 20 and 60% of SiO_2. This is in accordance with the analyses on the Qal'at al-Bahrain slag.

The main contributor of SiO_2 in smithing slag normally is the sand used as a flux, what leads to the formation of fayalite. The absence of fayalite in the slag from ed-Dur could on the one hand indicate that sand was not used. On the other hand it is possible that the working temperature in the hearth were higher and other higher melting point minerals will form. This could explain the absence of fayalite and the presence of Ca-Si-rich minerals. Silicon could also originate from sand included in the lining of the hearth.

• P_2O_5
High phosphorous amounts make the iron *cold short*, which means that it is hard and brittle and cracks when cold worked. In the past the use of phosphorous rich ores had the just the interesting side effect that the iron produced was somewhat harder than normal wrought iron.[86]

P_2O_5 was only found in two slags. In BS 1008B phosphorous is attested in the grey infill between the iron oxides, amounts vary between as little as 1% till 10%. In BS 1068 P_2O_5 is associated with the grey laths or in inclusions in small amounts.

[85] Feuerbach, 2002: 124.
[86] Wertime, 1962: 17; Avner, 1974: 248; Ehrenreich, 1985: 67, 76 & 95.

An origin of P_2O_5 from the use of phosphorous containing iron ores is unlikely, since the P is not associated with the iron oxides found, but rather with inclusions and other mineral phases in the slag. Ash from the charcoal can be a possible contributor.

• SO_3

Sulphur is not a wanted element in iron production or iron working. Iron with too high sulphur content is harmful, since causes *hot shortness*, meaning that the metal is too brittle to be forged and fractures. When sulphur is combined with iron it forms FeS (iron sulphide) a low melting point eutectic alloy. This tends to concentrate at the grain boundaries. Due to the melting of the iron sulphide eutectic the cohesion between the grains is destroys and the metal cracks.[87]

Three slags contained sulphur. In KR 003 the SO_3 is associated with the black Fe-rich inclusions, what could mean they are actually FeS. This can enter the slag if iron sulphide ore is used to produce iron that was not sufficiently roasted to drive of the sulphur. It should however be noticed that these inclusions also contained a small amount of copper and the sulphur can alternatively be associated with the copper. In the light of possible copper working at Khor Rori in the same location as iron working, small bits might have ended up in the smithing hearth. Maybe the hearths were multifunctional.

In BQ 1008B SO_3 is found in one of grey phases that is seen as small inclusions and also P-rich phase. In slag BQ 819 the SO_3 is found in the black glassy phase part of the mixed infill between the larger grey laths.

A last source of sulphur could be the use of coal a fuel, but this cannot be the case in these examples.

• K_2O

K_2O is only found in the glassy infill and in some inclusions. Besides carbon and hydrogen, plant matter contains some inorganic compounds that remain in the ash such as K_2O.[88] The lining used for the hearth is the second source of potassium.

• CaO

CaO was detected in all slags and in the many different phases. Iron oxide phases can contain up to 25% of other elements. The highest levels of CaO are around 7%, with one exception that contains 17%. The rest varies from about 4% to as little as 0,3%.

In all other instances CaO is found in other phases then the iron oxides often associated with high SiO_2 levels. The glassy infill has some 10% of CaO included and the other phases have a variable amount of CaO, ranging from *ca.* 30 to *ca*. 65%. It cannot be stressed enough that this is a typical feature for the slag from the region, maybe even the larger region since a smithing hearth bottom analysed from Merv also contained up to 35% of CaO[89].

The first explanation often given for this is the deposition of calcium out of the environment in the slag due to the effect of ground water and rain, but this can hardily be the case at ed-Dur. This can lead to the formation of $CaCO_3$ or $CaSO_4$. The site is situated in the desert and the environment consists out of sand. Moreover the CaO is held within the minerals attested in the slag and not as a separate element deposited at a later stage.

A second source would be that the calcium present in the ash reacted with the slag and formed Ca-rich minerals, but this would not account for more than 7-10% of the Ca. It should

[87] Wertime, 1962: 17; Avner, 1974: 248; Ehrenreich, 1985: 71.
[88] Feuerbach, 2002: 124.
[89] Feuerbach, 2002: 92.

be noted that the CaO content of European smithing slags is often (much) higher than the amount found in slags originating from the direct reduction process, so a contribution during the smithing process is evident[90]. Still the 10% border is rarely crossed although higher amounts are not unknown[91].

A third sources could be the use of iron produced from Ca-rich ores. Maybe the iron worked on site was not of very high quality and still had some Ca-rich slag included in the wrought iron. This would have to be purified first in a primary smithing process and the slag included in the wrought iron would have found its way into the smithing slag forming in the hearth.

A fourth source of CaO might be the use of a Ca-rich lining for the hearth. Maybe it was made from a mixtures of pulverised *farush* and other matter. The calcium would have reacted with the slag forming and took up part of it.

A last possibility is the use of a Ca-rich flux when forging, instead of the more commonly used sand. The latter might be far fetched because of the omnipresence of sand on the site.

• TiO_2 & CrO_x
Titanium is only attested in three slags (BQ 1008B, BS 1110 & BS 1215) and in two of these also chromium is found (BQ 1008B & BS 1110). These two elements are lithophile what means that they preferentially will go into the slag. The chromium seems to be related to the Ti- and Fe-rich phases.

BQ 1008B is the only slag that contains TiO_2 in all its iron oxide phases. It does not appear in the global grey phase, but individual grey inclusions have the highest Ti-levels found anywhere. The presence of titanium and chromium is probably due to their occurrence in the original iron ores used to produce the iron.

• FeO
As can be expected iron oxides are omnipresent in slags related to iron working, but also in almost all other phases attested at least some iron oxide (or iron) was found.

The different morphological forms in which the iron oxides appear were already mentioned above and will not be repeated here. It suffices to add that large wüstite crystals are typical for smithing slag. Wüstite dendrites in a fayalitic bulk (not present here) are typical for a fast cooling from liquid state. The amount of wüstite is much more variable within smithing slags, whereas in smelting slags it more uniformly divided.[92] On the other hand the presence of dendrites in smithing slags can also be related to growth in a plastic state, rather than rapid cooling from the liquid state. They represent low temperature growth rather than high temperature growth[93].

• Comparison to the Mleiha and Qal'at al-Bahrain material
Because of the fact that the publication on the Qal'at al-Bahrain slag published analytical results of the different phases, these could be included in the discussion above. In general the results are in good accordance. In the study of the Mleiha slags only global analyses are published and no separate phases were analysed. One of the ed-Dur slags analysed together with the Mleiha material had a very high iron oxide content (95,55%) and this type was not encountered among the slags analysed here. The composition of the other slag is in accordance with the findings here, especially with BO 845, BQ 872 and KR 002.

[90] Eschenlohr & Serneels, 1991: 115.
[91] Seen e.g. Serneels, 1995b: 127 (8^{th} c – 12^{th} c AD, Liestal-Röserntal – Switserland, due to the use of Ca-rich ores) and Photos, Filippakis & Salter, 1996: 191 (Hellenistic, Knossos – Crete, no explanation).
[92] Dunikowski, Leroy, Merluzzo & Ploquin, 1998: 147-148.
[93] McDonnell, 1991: 26.

All slags from Mleiha contained MgO. This is not the case for all the slag from ed-Dur slag, but if MgO was present it was in similar amounts. The amount of CaO was also high, but values above 30% do not appear in Mleiha. It should however be kept in mind that a different analytical technique was used, ICP-MS with a complete solution of the samples. This provides full quantitative information on the elements present. The EDX results are only semi-quantitative.

• Overall composition & interpretation

Table 63 gives the overall oxide composition of ten of the thirteen slags selected for analyses.

Sample nr.	Na_2O	MgO	Al_2O_3	SiO_2	SO_3	K_2O	CaO	FeO
BS 1110	0,25	7,60	1,78	50,85	-	0,59	34,67	4,26
BO 716	-	3,16	2,16	42,51	-	1,08	37,74	13,36
BS 1264	-	-	-	24,07	-	-	41,97	33,97
BQ 819	-	-	-	23,67	-	-	14,93	61,40
BQ 875	-	-	-	1,92	-	-	31,72	66,37
BQ 1008B	-	0,74	-	16,21	-	-	15,24	67,81
BS 1185A	-	4,58	-	12,02	-	-	10,09	73,31
KR 002	-	1,65	-	6,73	0,84	-	6,08	84,70
BO 845A	-	-	-	0,85	-	-	13,28	85,87
BQ 872	-	-	-	7,42	-	-	6,64	85,94

Table 63: Average oxide wt% composition of slags.

High values of above 60 wt% of FeO can be seen in seven of the samples and are expected in slag related to iron smithing. In smithing slag the iron oxide is generally identified as wüstite (FeO), although higher oxides do occur, but this may be due to post-depositional oxidation.[94] The low value in BS 1110 is due to the fact that the fragment analysed was largely a piece of the glassy ceramic 'hearth lining' that was attached to the slag, also seen in the presence of Na_2O, MgO, Al_2O_3, K_2O and a high amount of SiO_2, all pointing towards ceramic components. This kind of lining was observed on many slags and shows that the smithing hearth was more elaborate than just a pit dug in the sand.

Although the FeO content is higher in BO 716, the overall picture is the same. This small slag is the result of the reaction of the slag with some sort of hearth lining. This slag, together with BS 1215 and areas of BQ 819, BQ 1008B, BS 1068 and BS 1198A, have microstructures that resemble those of smelting slags exhibiting dendritic iron oxide and a (lath-shaped) matrix. This is explained by the fact that these slags formed in the hottest zones of the smithing hearth, going through a phase of actual melting or at least reaching a temperature close by. BS 1264 had a glassy black appearance indicative for high temperatures and the presence of ceramic components. The iron oxide content is relatively low, but the microstructure fits with the next group of slags. BO 716 and BS 1215 might fit the type of slag defined as SAS (*scorie argilo-sableuse*, see p. 394). They are formed when the smith adds a large amount of flux to the surface of the metal for welding or steel working, and it is sometimes present as a separate phase on top surface of a slag.

A second group of slag has iron oxide amounts higher than 60% often present in a massive form and in a layered structure. The amount of iron oxide decreases towards the bottom of the slag whereas the amount of CaO and SiO_2 increases, showing the reaction of the forming slags with the lining.

[94] McDonnell, 1984: 48.

KR 002 and BQ 872 are very similar in their global composition, although they originate from a different site, the first came from Khor Rori, the latter from ed-Dur. This shows that these were most probably formed under the same conditions and in a similar process. These slags fall within the definition of SGD (*scorie grise dense*) and SFR (*scorie ferreuse rouillée*) as discribed above. The separation between the two is not very clear, but the main difference is that the latter group contains more particles of metallic iron. These do occur in the slag analysed but are rather small and never very abundant.

9.6. Mineralogical phases – powder-XRD

• **Introduction**
The basic scientific ground on which XRD is based was already explained in *Chapter 4* and will not be repeated here.

The apparently featureless slags are in fact made up of complex mixtures of crystalline and amorphous minerals. An attempt was made to examine the crystalline mineralogical phase composition of 11 slags using X-ray powder diffraction spectrometer[95] (XRD) at the *Laboratory of Soil Science - Ghent University* headed by Prof. dr. E. Van Ranst. N. Vindevogel preformed the analyses. For an introduction to the interpretation of the obtained spectra the help of dr. F. Mees was indispensable and very appreciated. XRD does however not detect any amorphous (glassy) phases and also minor crystalline phases are difficult to distinguish, since they get lost in the background noise. Additionally the count rate was rather low, making the effect of the background even more profound. The reasons for the low count rate are introduced by two main factors: the effect of the matrix (a large portion of amorphous material) and the individual character of the minerals present. The geometry of some crystals causes little reflection and therefore low peak intensity. The whole dataset is included in *Appendix 10*.

The mineralogical composition of slags dependents on the temperature in the hearth and the cooling rate of the slag. The chemical and mineral texture of smelting and smithing slags can be indistinguishable. The heterogeneity of bulk analyses, both within a sample and between different types of slags, particularly of smithing slags, makes their use as comparative data of restricted use.[96]

• **Discussion & conclusions**
'Normal' smithing slags are made up by three phases, *silicates* (normally fayalite, often impure with small amounts of Mg, Mn & Ca present), *free iron oxides* and a *glassy matrix*. In some cases the glass phase can be absent however. These main phases can be associated with oxide minerals (hercynite (Al_2FeO_4), ferro-hyrcynite ($AlFe_2O_4$), hematite (Fe_2O_3), maghemite, wüstite and iron oxy-hydroxides) and silicates (quartz, pyroxenes & melilite). Particles of iron are frequently present. Also the presence of unfused pieces of rock or ceramic are often observed. The chemical composition is generally dominated by iron and silicon oxides, ranging from material poor (5%) to materials rich in iron oxides.[97]

Fayalite does not seem to appear in the slag from ed-Dur and the ones from Khor Rori. Fayalite (Fe_2SiO_4) is part of the olivine group of silicates and has a melting point of 1205℃ on the other side of the olivine series is fosterite ($MgSiO_4$) with a high melting point of 1890℃. In-between there is a whole series of miner als that include calcium or magnesium such as kirschsteinite ($CaFeSiO_4$) or monticellite ($CaMgSiO_4$) with a melting point around 1340℃. Their melting point is more towards that of fayalite if they contain more iron. Another possibility would be pyroxene minerals (e.g. diopsite, $CaMgSi_2O_6$, or hedenbergite, $CaFeSi_2O_6$), but these have higher melting points and are rarely found in ancient slags.[98] Locally temperature of up to *ca.* 1400-1500℃ could be reached in a smithing hearth, since this is the temperature needed to fuse two pieces of iron.

[95] In a first attempt XRD was tried on solid samples at the *Department of Materials Science and Metallurgy – Ghent University*, but due to the porosity of the material the results were too poor to be considered.
[96] McDonnell, 1984: 52.
[97] McDonnell, 1991: 24; Serneels & Perret, 2003: 473.
[98] Serneels, 1993: 24-27.

In spite of considerable time spend on deciphering the EDX-spectra, these *sudoki* were not solved satisfactory. The interpretation does not exceed the level of confirming the presence of some mineral phases. Not all spectral peaks could however be assigned and several intense peaks remained undetermined. The software used did not prove to be of great help, since a hole list of exotic minerals were suggested that could impossibly be present. The results presented have to stay at a very general level. Also the quantification of these phases was impossible, since not all peaks were identified.

Some things can be concluded however[99]:

- No fayalite (Fe_2SiO_4) was found. This is rather unexpected, since in the majority of the European smithing slags this mineral is prominently present.
- Wüstite (FeO) and to a lesser extent magnetite (Fe_3O_4) was attested in all slags, confirming the visual identification of these phases. This confirms the conclusions drawn from the visual and BSE-images of the slags.
- Some have a hematite (Fe_2O_3) may be present as segregations in magnetites if the furnace conditions had been highly oxidizing.
- Quartz (SiO_2) and calcite (CaO) are the important constituents of all slags. Especially the presence of calcium seems to be a phenomenon typical for the Gulf-region.
- Two slags had a very different EDX spectrum from the others, being BS 1131 and BS 1198. Two mineral phase were attested not seen in the other spectra (maybe as a minor constituent), åkermanite ($Ca_2MgSi_2O_7$) and kirschsteinite ($CaFeSiO_4$). Åkermanite is part of the melilite silicates group. The mineral is often present in blast furnace slag and other smelting slags of non-ferrous metals, but also in *slagged furnace lining*. When high amounts of calcium are present kirschsteinite can form and has a relatively low melting point compatible with the range of temperatures at which iron working processes took place[100]. The presence of kirschsteinite and melilite (i.e. a more general term for åkermanite) was also reported by Prof. dr. M.A. Elburg in the pertrographical slices of and to a lesser extent in S 0007. Based on this information the square/rectangular minerals seen in the optical microscopical and BSE-images, can be identified as åkermanite and kirschsteinite minerals. Additionally they can also be seen in BO 716 and BS 1215[101]. The presence of these minerals is certain related to the slagging of the smithing heart lining.
- Other Ca-Si compounds may also be present, such as wollastinite ($CaSiO_3$), and such as hedenbergite ($CaFe(Si_2O_6)$).
- BO 1349 is definitely a part of hearth lining. The main constituents were calcite and quartz, with a little wüstite. This can be indicative that the lining was made of calcite and quartz material, and may provide a source for the high calcium levels in the slag. The wüstite probably was trapped in the slagging lining when iron oxides fell into the smithing hearth.

It has to be admitted that the analyses of complex materials such as slag is not a trade that can be picked up in a hurry. Without the necessary in-depth knowledge and reliable *a priori* assumptions and eliminations, the data is a forest or rather a mountain range of peaks. The third panel of the triptych, i.e. the determination of the mineral phases present, after the optical microscopic information and the chemical compositional data, is only a sketch. More research and time is needed to use the XRD-data to its fullest extend. Both were not at hand so only very general conclusions can be drawn.[102]

[99] General information on the minerals taken from Bachmann, 1982: 14-16.
[100] Photos, Filippakis & Salter, 1996: 192.
[101] In Hauptmann, Weisgerber & Bachmann, 1988: 40, a micrograph with a similarly shaped mineral was published, which was also identified as kirschsteinite. Seen, Fig.128 for BS 1198, Fig. 129 & 140 for BS 1110, Fig. 132 for BO 716 and Fig. 144 for BS 1215.
[102] M. Van Nie informed me that XRD to investigate slag is actually rather *passé*, because of the problems in determining the iron phases (few distinctive reflection peaks) especially when the lab performing the analyses have no experience with iron oxide rich material. I was only made aware of this *after* the analyses had been done.

9.7. Portable forges *versus* pot-bellows

In area CY at Mleiha a pit next to a small building contained some slag and the remains of a specific ceramic vessel[103]. This vessel is basin-shaped, has an opening with a spout at the base and has a flat bottom (see Fig.148). Traces caused by fire can be seen till the middle of the vessel. Fragments of similar spouts were also reported from Area 7/Area CZ[104]. One of the Pakistani labourers at Mleiha pointed out that a similar vessel was used as a 'forge' in Pakistan, but with a lid and with the aide of bellows (making the spout a tuyere), called a *doukan*. According to the A. Ploquin, S. Orzechowski and B. Briand it is also possible that crucibles (i.e. to produce crucible steel) were placed inside this kind of furnace.[105]

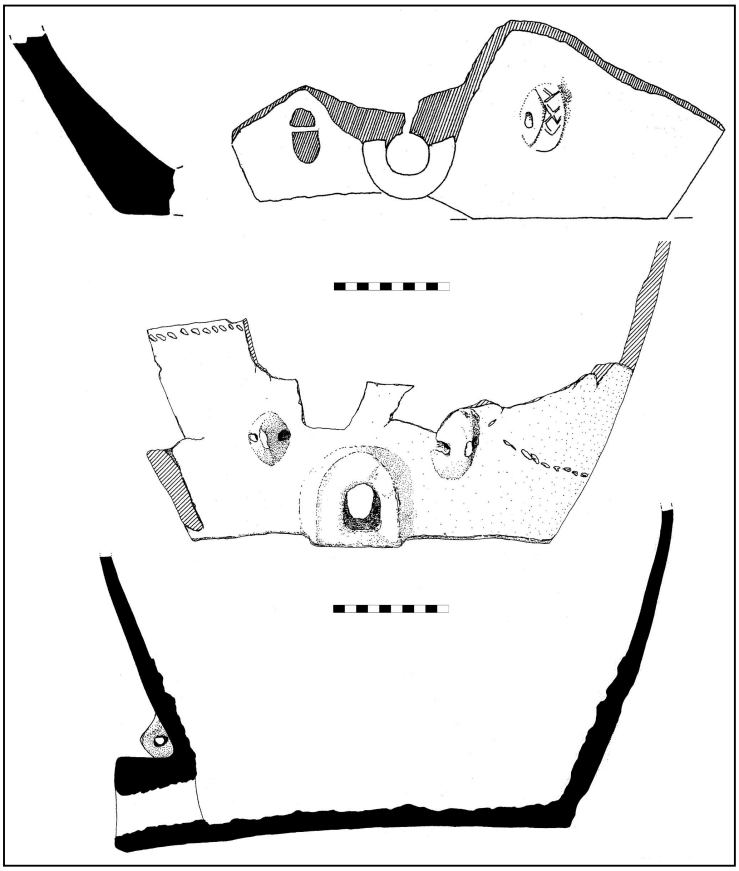


Fig. 148: Ceramic vessels from Mleiha (after Ploquin, Orzechowski & Briand, 1999: 199).

Here I would like to propose a different function for the basin-shaped vessels, being a *pot-bellow*. To make many metallurgical process work in an efficient way a forced draft is necessary to achieve the high temperatures. There are several techniques for that. A natural forced draft by making an opening in the furnace in the direction of the wind is the easiest, but rather unpredictable. The use of bellow is probably the best know technique to create a artificial air blast. Therefore the bellow is also one of the attributes often associated with a blacksmith, next to the forge, hammer and anvil. Often they do not leave any archaeological evidence behind since bellows were often made of perishable materials (wood, animal skin, leather etc.). One kind of bellow, the *pot-* or *bowl-bellow*, is different in that a ceramic or stone vessel, covered with a leather skin, formed the air reservoir. This kind of bellow is know to have been used in many parts of Africa an Asia for smelting and forging activities in the past and is still in use. The oldest representations are found in a pair of foot-operated bellows portrayed in the wall painting of the Egyptian tomb of Rekhmire (dating to *ca*. 1450 BC). The earliest archaeological remains are found on 2nd millennium BC sites in C-Anatolia and Babylonia. By the mid 2nd millennium BC they are in use in N-Syria and S-Palestine and found their way into Egypt and Cyprus (see Fig. 149 for examples). Later examples were excavated in Sudan on Roman period sites.[106] In S-Indian and Sri Lanka pot-bellows were used for smelting iron till the early years of the 20th c AD, and they remain the common bellows type for African smiths. Although rubber from the inner

[103] Ploquin, Orzechowski & Briand, 1999: 172 & 175.
[104] Ploquin & Orzechowski, 1994: 35.
[105] Ploquin, Orzechowski & Briand, 1999: 179.
[106] Davey, 1979: 101 & 109-110.

tubes of old truck tyres has largely replaced the leather covering[107]. Recent examples for smelting and forging operations are reported and documented from Ethiopia[108]. These vessels were dug in the ground and an air outlet was situated in the middle of the vessel side (similar to the Qal'at al-Bahrain bellows, see below and Fig. 151-2).

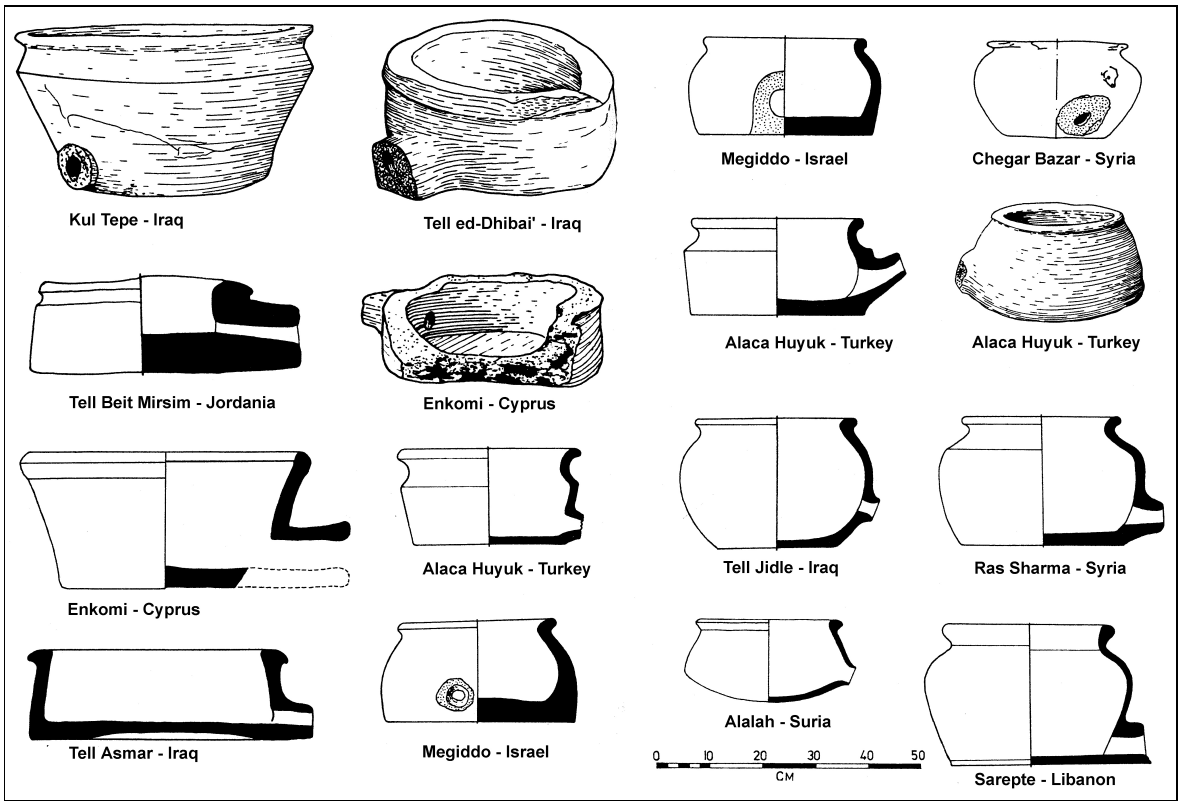


Fig. 149: Examples of pot-bellows (after Davey, 1979: 104-105).

There are two basic systems to operate pot-bellows (Fig. 150). The first is to have a diaphragm in the skin covering which will open as the skin is raised, allowing air to enter the bellows. The diaphragm is closed when the skin is depressed and the air is forced directly into the furnace or hearth. This system requires a very well designed diaphragm if the bellows are to function efficiently. The second technique is easier to use, in this case the air enters the bellows through a gap between the tuyere and the bellows pipe as the skin covering is raised. When the skin is depressed air is forced in. To prevent hot air being drawn back from the furnace or hearth, a system of using two alternating bellows blowing in one tuyere was developed. When the air from the first bellow is blown in the single tuyere the second bellow is filled again with air. Then the second bellow is emptied, while the first is filled again, in this way alternating each other. While a certain quantity of hot gas and ash was drawn into the bellows by this system, the continued induced draught effect into the furnace or hearth through the single tuyere minimised it so the bellow did not overheat. This technique is still used by some African smiths. The bellows themselves can be manipulated by hand or operated by foot.[109]

[107] Craddock, 1995: 180-181.
[108] Todd & Charles, 1977; Todd & Charles, 1978.
[109] Davey, 1979: 101; Craddock, 1995: 180-181.

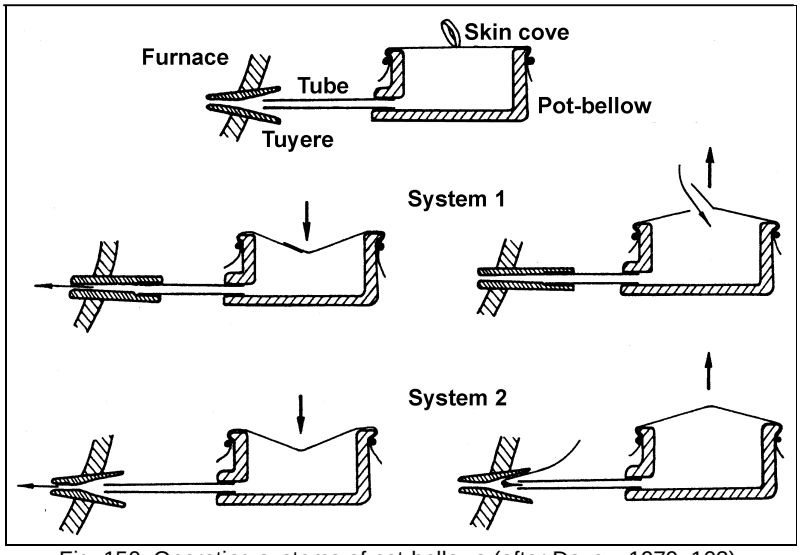


Fig. 150: Operating systems of pot-bellows (after Davey, 1979: 103).

The shape of the vessel used as bellow is quite distinctive when found in a metallurgical context. The opening at the top must be wide so that the hide covering it can draw in a maximum amount of air. The covering is attached to the bowl with a cord, which is fastened under an out-turned rim or in a groove made in the side of the pot, just bellow the rim. At the base or in the side of the vessel a hole of a diameter varying between 1 to 4 cm is located. To prevent the direct contact of the bellows with the intense heat, a separate tuyere was inserted in the wall or side of the furnace or hearth. The air that was pushed from the bellow towards the actual tuyere was transferred by intermediate inserted tube (e.g. a reed). A tuyere has a short life since it is exposed to intense heat and has to be replaced quite often. By not attaching the pot-bellow directly to the furnace or hearth it could be used many times and transported if necessary. Pot-bellows are roughly made with a coarse tempering, typical for appliances. The size varies from 30 to 60 cm in diameter and between 13 and 25 cm in height.[110]

Why are the vessel sherds from Mleiha likely to be the remains of pot-bellows?
The first possible explanation offered by the archaeometallurgists that studied the Mleiha-material was that these ceramic vessels were actually portable forges. The spout at the base acted as the tuyere to force air through the charcoal. The fact that the vessels are corroded by fire on the inside is explained as the result of the reaction to the intense heat of the charcoal. To this explanation several objections can be made. First of all the use of a tuyere at the base of a forge is not attested in antiquity, and is to be considered a recent phenomenon[111]. In the past air was always blown from above. In this way part of the charcoal could be brought to the desired temperature by focussing the blast and limiting the charcoal used. If the charcoal was to be heated from below more would be consumed. The air was blow onto the charcoal and not through it. A more practical reason to place the tuyere above the bottom is the formation of the smithing slag. They drip down till they reach the cooler zone under the tuyere, where they solidify (see above). If the air inlet already were at the base, the forming slag would easily block it. This problem is avoided nowadays by including a device to crush slag when formed and a vault underneath to remove the fragments. The walls of the vessel are also thin (0,8 cm at the top and 1,6 cm at the base), what makes them venerable when iron pieces are handled, especially if the ceramic is hot. They do not fit into the picture of the 'rough' nature of the blacksmiths work.

[110] Davey, 1979: 101-102; Craddock, 1995: 180-181.
[111] Serneels, 1998: 29.

The second possible use put forward by A. Ploquin, S. Orzechowski and B. Briand, is that of portable furnaces to heat crucibles in order to produce crucible steel. Some problems or lack of evidence have to be pointed out there. The slag brought into connection with the crucible process does not look at all like other published crucible slags. The first unarguable historical and archaeological data evidences that crucible steels were being produced in considerable amounts only dates from the middle half of the 1st millennium AD onwards (see above). On top of that it happened in regions with a rich metallurgical past. So the use of this process in SE-Arabia would not only have a very early date (as old as the S-Indian production), but also unique in the fact that iron was produced locally. Archaeologically the statement is also not sustained since no crucible fragments were found, something likely to happen since the rather widespread presence of slag and the retrieval of 'furnace fragments'. The burn marks on the inside of the vessels also tell a different story. If crucibles were heated inside the pot, they would have been completely covered with charcoal in order to stimulate a uniform heating of the crucible and its charge. By consequence burn marks should be present along the whole inside surface, but these traces are restricted to the lower part only.

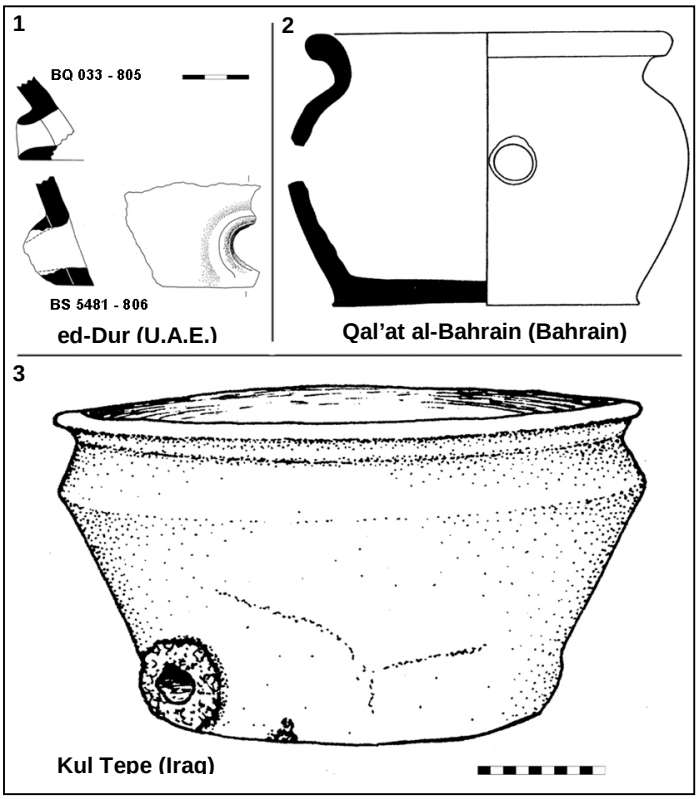


Fig. 151: Pot-bellows & possible fragments from ed-Dur
(2: Højlund & Andersen, 1997: 167; 3: Davey, 1979: 104).

I would like to propose that these vessels are the remains of pot-bellows. To show the morphological resemblance of the Mleiha material the pot-bellow from Kul Tepe example is reproduced again, this time at the same scale (Fig. 149 *versus* Fig. 151-3). The dimensions of the best preserved Mleiha fragment has a minimal height of 27 cm, a maximum diameter of 48 cm and the two spouts are between 3 and 4 cm. These measurements are in accordance with the general dimensions published on other pot-bellows. On the lower profile of Fig. 149 the onset of a declining vessel side can be seen. The small ears above the spout could serve different functions. They could be anchor point to secure the bellow to the ground/floor or they could serve as attachments for the tube to the tuyere or the tuyere itself. A last possibility is that they served to bind to or pas through the rope that fixed the bellow skin. The lower part that seems to have undergone corrosion by heat would not be exceptional. It is normal to find the internal surfaces of bellows blackened with ash and on occasions even fragments of slag are found inside. Severe burning is not common however, and is probably due to incorrect use or an accident that occurred[112]. Maybe the reason whey the pots at Mleiha were discarded. At ed-Dur two fragments were found that resemble the spout of an Mleiha-like vessel (805 – 806; BQ 033, BS 5481 – Fig. 151-1). The ceramics belong to Group 10 as defined by K. Rutten. They are orange to brown

[112] Davey, 1979 : 102.

and have a coarse vegetal tempering. The ceramics within this group are local SE-Arabian in production[113].

That the pot-bellow technology was known in the wider region can be seen in two installations with pot-bellows excavated at Qal'at al-Bahrain[114]. Each installation covered an older, similar installation. The type of the pot-bellow is different from the vessels from Mleiha in that they were integrated in some sort of a bench and that the tuyere exit is situated more in the middle of the vessel side (see Fig.151-2). The tuyere hole was *ca*. 3 cm wide. The dating of these installations is not without problems, since no diagnostic artefacts were found in association with them. The excavators however give a tentative dating to period IVc/d of Qal'at al-Bahrain, which falls in the middle Tylos period (100 BC – 250 AD). The installation was associated with fragments of crucibles, used for copper working in this case, and copper working slag.

All this leads me to conclude that the sherds found at Mleiha are more likely to be the remains of pot-bellows than those of portable forges or furnaces to hold crucibles.

- ***Erratum***

Fig. 152: Vessel with spout from Mleiha on exhibition in Sharjah Museum (U.A.E.)

At the very last stage of writing out this PhD dissertation I was made aware of this vessel from Mleiha on exposition in the Archaeological Museum of Sharjah (U.A.E.)[115].

It has to be admitted that this casts doubt on the interpretation presented above. This clearly is not a pot-bellow, since it is much too high to be efficiently worked. This vessel seems to be a recipient for liquid, equipped with a spot at the bottom to tap. It is very possible that the bottom fragments show above are from similar vessels and are not related to any metallurgical activity at all. The traces caused by fire as reported by the excavators from Mleiha, are then likely to be secondary and not related to the original use of the vessel.

Still, in the light of this more or less complete vessel, it is incomprehensible that similar bottom fragments were linked by the archaeometallurgist working on the Mleiha material to a furnace or a smithing hearth.

[113] Rutten, pers. comm.
[114] Højlund & Andersen, 1997: 165-171.
[115] Provided by Prof. dr. E. Haerinck & dr. A. Daems (October 2007).

9.8. Interim conclusions iron slag

The initial gut feeling that the slags found at ed-Dur were smithing slags is confirmed by the analyses, although not all slag show the typical microstructure of this type of residue[116]. The slag from ed-Dur is definitely not related to copper production or working, since not even traces of copper were found. Slag from Mleiha that were connected to small-scale copper production all contained copper in various amounts between 3 and 4 wt%. The circumstantial evidence (e.g. the morphology, the amount and the history of iron working in the region, etc.) however makes this attribution highly likely. Also the slag looked at from Khor Rori can be put in this class. The optical microscope, SEM-EDX and limited petrographical and XRD results are in broad accordance with each other.

It is clear that the bulk composition of the smithing slags from ed-Dur is completely different from those analysed in Europe. Fayalite the main silica constituent of European smithing slags does not appear and the calcium levels are much more elevated. This is however not a characteristic of the slag from ed-Dur alone. It appears in the slag from Mleiha, Khor Rori and the single published slag from Qal'at al-Bahrain and should thus be considered a feature typical for slags originating from the Gulf region.

The origin of these high calcium levels is however not clear. A tentative explanation would be that it is due to post-depositional processes. The analyses done here show however that the calcium is present *in* the minerals making up the slag (e.g. kirschsteinite) and is part of the slag itself. The most probably source would be the lining of the smithing hearths and through high temperature processes the calcium became mixed with the forming smithing slag. Another possibility is that a calcium-rich flux was used. Normally sand is used as flux during smithing to keep the surface oxide free. No partly dissolved and particles are seen in the slags from ed-Dur however. It has to be admitted that this suggestion is rather far stretched, considering the omni-presence of sand in the dessert. Still it remains an option.

The absence of partly fussed sand particles can also be explained by the fact that no flux was used, indicative of a primitive smithing technology or the working of wrought iron. When 'soft' iron is worked, little magnetite and oxides are formed at the surface, so the smith does not has to add a lot of sand. When PCB's do contain a lot of silica this can be indicative for special processes. This can result from working bad quality iron, welding operations, steel working and recycling[117]. This would fit the small dimensions of the smithing slags, i.e. small pieces of iron were 'worked' or shaped, without the use of more complex smithing techniques, such as welding. I would like to suggest that for example the more simple forms of arrowheads and nails could have been made on site from imported iron, but that the large objects such as the swords were certainly not local. The preliminary results (not included) on the typological study of the swords tend to support that. The long swords attested in the grave of ed-Dur are atypical for the region and are comparable to contemporaneous swords from the Parthian and later Sasanian Empire.

One of the items to evaluate at the onset of this study was the possible use of crucible steel in SE-Arabia. As mentioned and shown above the corroded nature of the iron objects did not allow this kind of evaluation. So we can only turn to circumstantial evidence to prove or counter that statement. As it stands now, no evidence (under the form of slag, crucibles, furnaces, objects microstructure and historical sources) can be brought forward to found such an assertion. On the contrary the closing piece of the puzzle, the Mleiha *portable furnace*, is very likely to have had a very different function e.g. a *pot-bellow* or as seen in the *erratum* above, just a spouted vessel. The slag is on the one hand not similar to the typical smithing slag found in Europe, but on the other hand they are also very different to typical

[116] An opinion shared by V. Serneels and M. van Nie.
[117] Dunikowski, Leroy, Merluzzo & Ploquin, 1998: 150.

crucible slag. The archaeological and historical information does not bring additional arguments to the hypothesis of local iron production. Iron always, even into recent times, seems to have been imported to SE-Arabian.

There are no positive analytical arguments within the slags found at Mleiha and ed-Dur to classify them as the remains of a crucible process. Moreover according to A.M. Feuerbach crucibles fragments are the most abundant material remains from the crucible process[118]. The process can generate a limited amount of slag, but cannot be performed without a crucible. These crucible remains are also quite distinctive since they undergo a high temperature process that generates a typical glaze. If crucible fragments were present they should have turned up in the ceramic collection, if not at Mleiha, certainly at ed-Dur, where a large sample was studied. This is not the case. The crucible technique is also a rather specialised procedure that needs specialised skills. For this reason the evidence known from India and C-Asia is concentrated in specialised workshops. Also for this, there is no evidence at Mleiha or ed-Dur. So based on only the circumstantial evidence a crucible process can already be safely excluded.

A small side remark that is not further explored here is the position of the blacksmith in the late Pre-Islamic SE-Arabian community. In general the slag are spread all over the site of ed-Dur (Mleiha provides a slightly different picture). The smithing technology might have been widespread among the people and used when needed. Nothing at ed-Dur points to the existence of a 'village smith' with his own workshop, since that would have left more concentrated refuse areas. This implies that the methods used must have been 'basic' and not a specialised activity performed by certain individuals, hence the technical level must have been rather low. Moreover the formation of one PCB is seen as the result of one smithing session (e.g. one days work, for about three to four hours)[119]. An interesting link to make would be between the size of the slag generated and the skill of the blacksmith. The less experienced the blacksmith the more iron (oxide) that is lost during the forging, i.e. more cycles of reheating would be needed to get to the same end result. The rather low number of slags retrieved from ed-Dur, e.g. *ca*. 70 fragments, is indicative of only two months of work over an occupational phase of over one and a half century. Even if the total amount of slag remains were considerable higher, this would still not be indicative of a regular activity.

Remains of forges were present in the fort of Mleiha. This building is clearly associated with the elite of the community. That it had a residential function next to its clear defensive/military role is shown by the presence of a living area in the northern part of the fort. Around the residence storage rooms are located, probably in connection with the function of the owner. They could of course also have served as a depository for reserve stocked by the authorities and/or inhabitants of the site for times of danger. Workshops were also set up in these storage spaces, particularly for metalwork. An interesting detail is however that they do not seem to have a permanent character.[120] Iron working seems to have been an *ad hoc* activity, preformed when and where it was needed.

[118] Feuerbach, 2002: 176.
[119] V. Serneels and M. van Nie pers. comm.
[120] Benoist, Mouton & Schiettecatte, 2003: 71.

PART III

The new

Chapter 10. SYNTHESIS

"Get the habit of analysis - analysis will in time enables synthesis to become your habit of mind."

F.L. Wright

10.1. General synthesis

• Introduction

Much of the information deducted from the archaeometallurgical analyses was already summarized in the interim conclusions throughout this PhD. These are an integrated part of the synthesis and final conclusions. There is no point in repeating this here and I would like to refer the reader to the appropriate chapters. To this section *5.6.* has to be added, since it combines the metallographical, SEM-EDX and archaeological information on the recognisable copper-base alloy artefacts, and this will also not be repeated. Here only the most important findings are combined to draw some conclusions and do some suggestions towards the 'trade question'. This chapter is rounded off with a case study that exploits all the data gathered on one particular artefact class (the *ring-pommel daggers*), and shows the full potential of an archaeometallurgical study.

• Copper & copper-base alloys

One thing that has to be made clear here is that much of the information deduced from the metal samples and artefacts, does actually tell little about the technological level of the inhabitants of ed-Dur and SE-Arabia. This is particularly true for the copper and copper-base alloys and probably also for the silver objects. This is mainly because there is no substantial evidence that copper and copper-base alloys were worked at ed-Dur. The evidence of from Mleiha is also minimal and related to a relative primitive process of possible copper extraction. The small amount of 'bronze spillage' attested in the fort does however show that copper or/and copper-base alloys were melted and cast. What was made remains unknown, although the excavators link the fragment to the coin production due to the discovery of three fragmented moulds.

The expected metals of copper, tin-bronze and their leaded variants were attested. Bronze was the most common alloy used for object manufacturing. This is no surprise since bronze has been the most prominent alloy since early times. The metalworkers were fully aware of the limits and possibilities of different composition of alloy, e.g. leaded alloys are generally still in the as-cast condition and show no sign of working. This would have been difficult to cold works such alloys since large amounts of lead weaken the metal considerably. Hot working would have been entirely impossible. An interesting observation is that some leaded alloys approach or are similar to the composition of an ancient alloy described as *caldarium*. This is an alloy made up from of one part of lead and four parts of copper or bronze, i.e. a 20% leaded alloy. Due to inverse segregation it creates a white alloy.

Two fragments are from a high tin-bronze (in excess of 17 wt% of tin) and have a α+δ-eutectoid microstructure. Such an alloy is very brittle and impossible to work after casting. It is silvery in colour however end ideally suited to produce mirrors. The two fragments are then also the remains of such mirrors.

The rest of the bronzes are low- and mainly medium tin-bronzes. The average tin contents of the medium bronzes is around 10% and is in good accordance with the average tin contents of Roman bronzes. This an all duty alloy, although there seem to have been preferences towards certain compositions for certain objects.

The low tin-bronzes (less than 5 wt% of tin) are all heavily leaded and the tin attested is most likely present to reduce the melting point of such alloys. Moreover they are all heavily leaded, to improve the casting properties or to obtain a desired aesthetic effect (white surface).

An interesting feature is that the lead isotopic signature of objects that are 'surely' Roman, e.g. the *patera* and the pedestal, shows a good match with the signature of objects that are

though to be SE-Arabian, e.g. the wine set and the horse spout. This illustrates that they were made of a similar metal batch. From this it might be deduced that SE-Arabia was not providing in its own needs of metal at the period under consideration and was obtaining these basic metals from elsewhere.

Two 'new' alloy groups were attested: brass (copper-zinc alloy) and gunmetal (copper-zinc-tin alloy), both typical Roman. Brass even made up a considerable part of the studied assemblage and can be labelled as a common metal at the time. The introduction of brass appeared around the 1st c BC in the Roman Empire and the fact that brass is found in considerable amounts among the copper-base alloy samples from ed-Dur, shows that the site was not isolated from the rest of the world. The history of gunmetal is closely linked to that of brass, since it is the result of recycling brass together with bronze. The gunmetal from ed-Dur is rather different from that found in the Roman Empire, since the fraction of zinc is smaller than that of tin, the mirror of the Roman situation. One fragment has a 'true' Roman style gunmetal composition.

- **Slag, litharge & coins projected on the local technological level**

The slag, the litharge fragments and the collection of local SE-Arabian coinage does however provide information on the question of local technology.

The slag is *smithing slag* and is therefore proof that smithing activities took place on the site. The slag is certainly not related to a crucible process of any kind. The absence of any evidence of iron production in SE-Arabia in the period under consideration shows that most probably imported iron was worked. The evidence related to iron production at Mleiha is vague and in any case no production slag was found. Moreover the history of SE-Arabia strongly suggests that iron production was never important. This can be seen in the late introduction of iron in the material culture and in the importation of iron into the historic periods.

The absence of partly fussed sand particles can also be explained by the fact that no flux was used, indicative of a primitive smithing technology or the working of wrought iron. When 'soft' iron is worked, little magnetite and oxides are formed at the surface, so the smith does not has to add a lot of sand. A primitive smithing technology fits the small dimensions of the slags, i.e. small pieces of iron were 'worked' or shaped, without the use of more complex smithing techniques, such as welding. I would like to suggest that the more simple forms of arrowheads and nails could have been made on site from imported iron, but that the large objects such as the swords were certainly not local. The preliminary results (not included) on the typological study of the swords tend to support that. The long swords and trilobate arrowheads attested in the grave of ed-Dur are atypical for the region and are comparable to contemporaneous examples from the Parthian cultural sphere and later Sasanian Empire.

The chemical analyses of the slags from ed-Dur show that their composition clearly stands apart from analysed smithing slags from Europe, in that fayalite does not appear. The ed-Dur slag is also characterized by high levels of calcium. The analyses do however fit in with the very limited published data from other slags from the Gulf region.

The litharge fragments are on the other hand proof of an unattested technical process. The lead used is from the same origin than the lead objects, and excludes the possibility that the litharge is of later date. The litharge remains are related to a silver extraction process from copper-silver alloys (possibly the coins). This has to be kept in mind when the silver objects are to be examined in detail. This is not a 'simple' technical procedure and shows that the knowledge was available and used rather efficiently, since the silver levels in the litharge are low and not much silver was lost to the process. The cupellation was done in hearths from

calcium-rich material and not the historically better-known phosphor-rich substrate, i.e. bone ash.

The coins almost certainly are a local product, which does not mean that the alloying metals (i.e. silver and copper) have to be local. This study presented the first systematic analyses of a large collection of local SE-Arabian coinage (e.g. 104 specimens), which account for about 8% of all registered NE- and SE-Arabian coins. There is a broad correspondence between the types defined and the alloys used to produce the coins. This shows that certain compositions were strived for and 'intentionally' chosen. The debasement of the coins can be seen in three ways, sometimes combined, sometimes separate from each other. Lower silver levels are often associated with the more stylised iconography (especially clear among the obols). If this is not the case greater variety of the silver content within the classes is seen, so less care was taken to produce coins of similar alloy. A last way of debasement only present among the obols is a lower median weight of the more stylistic depictions. The lack of a true chronological sequence however hinders the real evaluation of these parameters (weight, silver content and iconography).

Evidence was brought forward to suggest that an artificial process of *pickling* or *depletion silvering* was used to obtain an artificial silver enriched surface on some of the low silver coins. This is a technological process that was not attested yet in the region, but it is know from large monitarized economies such as the Roman. To evaluate this issue further some experimental coins were made and treated by pickling. This limited experiment showed that a blank with 10% of silver, when treated correctly, could obtain a silvery surface.

No coins were encountered in the studied collection that were cast. The question whether the blanks were cold or hot struck could not be addressed completely since only one coin could be sectioned. The microstructure of BR 106 did not fully answer the question at hand, but the metallographic evidence tends to tip the balance to hot striking.

All this, the 'standardised' alloys, the hot striking and the process of pickling, suggests that the minting of the inhabitants of SE-Arabia were well aware of the technical procedures of minting. The coins never reached the technical level of coins minted by large Empires, but at least an attempt was made.

It can be suggested that the larger issues (tetradrachms) were used on a local level, on the site of ed-Dur and for the trade between the sites of Mleiha and ed-Dur. This is sustained by the almost complete absence of these coins in any other contemporaneous site. The smallest denomination (obols) has on average a much larger fraction of silver in their alloy, if not completely made of silver. This can be evidence that obols had a more intrinsic value and could be used in a wider region. Indeed the small silver obols turn up in a larger area. This points to partly monetary system that existed next to the undoubtfully still practiced barter trade. The number of coins seems to me too small to sustain a completely monitarized system.

Next to a currency the SE-Arabian coins most probably also had a political function. They materialized the urge of the local people and their rulers to define their identity. The foreign coins found on the site are also most likely not connected to trade, although they can serve to show certain regions were (in)directly connected.

10.2. Metals, isotopes & trade

• Introduction

The lead isotope data was not fully evaluated in this PhD, but certain interesting result already came forward and can be integrated in a more general approach towards the trade routes operating in the 1st c BC – beginning of the 2nd c AD.

• Lead

The large majority of the lead samples analysed plotted closely together indicating that they originated from a single source. With a high degree of certainty they can be linked to ores sources from Spain and Sardinia, the main providers of lead (and silver) in the Roman Empire during this period.

It is known from the *Periplus* that during the 1st c AD the Romans were exporting lead towards the Indian Subcontinent and apparently part of that also ended up at ed-Dur. The question remains via which channel provided ed-Dur. Three possible routes are known from textual and archaeological research.

1. The main trade route between the Roman Empire and the Indian Subcontinent was via the Red Sea along the S-Arabian coast, past the mouth of the Gulf towards India. Later on a direct route across open sea was also opened. The lead arrived to the Indian Subcontinent via the harbours of Barygaza on the NW-coast and Muziris/Nelkynda on the SW-coast of India.

   The metal that arrived in India could then be re-exported towards the Gulf. Again the *Periplus* illustrates this, but in this case for copper metal. The harbour of Barygaza that took in copper metal also shipped it back out to Apologos, at the head of the Gulf, and to Omana, a port on the route there[1]. Lead is however nowhere listed in the *Periplus* as travelling this way. But absence of evidence is not evidence of absence and theoretically it is entirely possible that lead was brought to the Gulf region by this trade network.

2. A second possibility is that Roman lead was already disembarked in one of the S-Arabian ports and then taken by local trade routes up the Gulf by ship or across land by caravan.

3. The third option is that the lead came from an entirely different direction, via the Palmyrene-Characenean network. This means that the metal travelled from the Eastern Mediterranean, across Syria to Palmyra. From where it was taken to the Euphrates and travelled downstream to the city of Characene, boarded on ship (or even loaded on caravans) and continued its rout down the Gulf to eventually arrive at ed-Dur. This trade route can be labelled as the 'Characene corridor'.

I will leave the second hypothesis aside, since there is only scanty evidence of contact between S-Arabian and ed-Dur and is thus the least probable. Still it remains an option.

A clue to solve the puzzle might be found on the object tentatively identified as an 'ingot' (S 0024). The isotopic signature of this object falls nicely in the middle of the lead cluster and this indeed makes it very likely that this object actually also *is* an ingot. Also the litharge fragments plotted in this cluster and show that the same lead was used in the cupellation process. Moreover I cannot think of another function for this large disc-shaped artefact.

[1] Casson, 1989b: 18 & 21.

An interesting feature on the ingot is that is has a stamped monogram. The same or similar monograms were also noticed on some of the SE-Arabian coins and on some pottery sherds. It resembles monograms on coins from Characene and Seleucia, and on an intaglio of a Characenean finger-ring found at Kharg. The meaning of this monogram is unfortunately unknown but it could be the abbreviation of Attambelos, a Characenean king. If we accept the hypothesis that the monogram is 'Characenean' it indicates that at some point the lead metal passed through the city of Characene.

We know that the links between ed-Dur and Characene were tight. This is evidenced by the mass of S-Mesopotamian ceramics found at ed-Dur and the fact that the largest amount of foreign coins (11 in total) is from Characene. Moreover a more general link to the Parthian cultural sphere is also found in the appearance of ring-pommel daggers (see below) and the similarity in some of the iron objects, e.g. the long swords, the trilobate arrowheads, the spear-/lance heads and the appearance shears in the tombs. The assemblage of these iron objects is somewhat mirrored in Parthian tombs excavated in e.g. Hassani Mahale (Iran), Ghalekuti (Iran)[2], Noruzmahale/Khoramrud (Iran)[3] and Tall Sheikh Hamad/Magdala (Syria)[4]. And in any case does not appear to be indigenous to SE-Arabia.

If we add together:

- The apparently strong link between ed-Dur and Characene.
- The appearance of a monogram on the ingot that may be Characenean in origin.
- And the absence of lead listed as an export from the Indian Subcontinent in the otherwise detailed accounts of the *Periplus*

An itinerary of the lead ingot via the 'Characenean corridor' becomes very plausible.

But as always it is the mavericks that are the most interesting. Two analysed samples from ed-Dur have to originate from a different ore source. One sample came from a rather large fragment of lead and might as well be some kind of 'ingot' (Z 019). One fragment of lead shows no overlap with Z 019, but plots in the close neighbourhood.

The only ore sources that plot in this region of the isotope plots, are the ones from the Indian Subcontinent. The published ore samples came from ore sources in the Rajasthan region, i.e. Zawar, Rajpura Dariba and Khankaria. The ores are polymetallic and contain zinc and lead ore, but silver and copper deposits are also present.[5]

The implications of an attribution of the two ed-Dur samples to an Indian origin are considerable. The *Periplus* and Pliny both state that lead was imported into India and suggested that no indigenous lead was produced. The findings here would contradict that and suggest that to a certain extent lead was produced in India, and apparently even exported to the Gulf region[6]. Two ports would be likely candidates for the export, Barygaza and Barbarikum.

It is possible that a chronological factor is at play here. The Romans extensively mined the Sardinian and Spanish ores during the 1st c AD. But after the annexation of Great Britain to the Roman Empire, more easily accessible lead sources became available and by *ca.*70 AD the British ore fields had become the main providers of lead. Moreover N.J. Seeley and P.J. Turner report a similar disappearance of the use of Sardinian and Spanish lead during the 1st c AD. They analysed Indian lead coinage and found that in the second half of the 1st c AD

[2] Sono & Fukai, 1968.
[3] Egami, Fukai & Masuda, 1966.
[4] Novak, Oettel & Witzel, 2000.
[5] Ericson & Shirahata, 1985: 207-209.
[6] This was already suggested by E.H. Warmington (1974: 268) and H. Chakraborti (1966: 253), but without any proof.

the lead isotopic signature drastically changed. The coins from the second half of the 1st c AD had a signature closely related to ores from ores from Zawar (Rajasthan).[7]

The data from the two ed-Dur samples fit the idea that India was producing (and even exporting) at least some lead, and that this metal reached the Gulf and ed-Dur, probably in the later phase of its occupation.

- **Silver**

The lead isotopic signature from the silver objects from ed-Dur is only partly overlapped by that of the Spanish and Sardinian ores. The most likely candidates to fill this gap are the ores from the Great Britain (i.e. the UK and Wales). A similar chronological effect as described for the lead may be at play here, but more research is needed to clarify this.

Other ore sources can however not be excluded for the silver. Isotope ratios from ores from Germany and France also show overlap and can be seen as potential providers. One silver finger-ring (K 205) that is poorly covered by any other ore data has a good overlap with some data points from Iran.

- **Bronze sample KR 009 from Khor Rori**

A last 'odd man out' point is a sample that came from Khor Rori. Sample KR 009 almost certainly originated from ores from the Indian Subcontinent, most probably from the same region were the lead originated discussed above, e.g. the region of Rajasthan. The lead isotopic signature of the copper bronze samples is very similar to that of the ingot. Moreover the ores in Rajasthan are polymetallic and some contain lead as well as copper.

The implication of an attribution of the sample from Khor Rori to an Indian origin is considerable. The *Periplus* and Pliny both state that copper was imported into India from the Roman World and suggest that no indigenous copper was produced. The findings here would contradict that and suggest that at least to a limited extent indigenous copper was produced in India, and apparently exported to Oman. This region would have had an outlet via the harbour of Barygaza. Moreover Barygaza is mentioned in the *Periplus* as sending out shipments of copper to the Gulf.

The signature of KR 009 fits the idea that India was also producing and even exporting (some) indigenous copper as well. The dependence of India on imported Roman copper might have been less significant than suggested in the *Periplus* and by Pliny. The importation of copper may have been rather meant to create a surplus than to fill a shortage.

[7] Seeley & Turner, 1984: 331.

10.3. Case study: *Ring-pommel daggers*

"It's the stories we're after. We love the stories and the artefacts can tell the stories."

D. Elliott

10.3.1. Object description & archaeological context

This chapter is meant to show the added value that metallurgical analyses can bring to the usual typo-chronological study of artefacts. The artefact group are the *ring-pommel daggers*, since this is the only iron object class that also has a copper-base alloy element included that could be analysed.[8] This chapter contains some repetition of things mentioned in the preceding chapters, but it serves to combine all info and place this against the archaeological back-ground.

Many descriptions can be found in the literature to classify cutting or thrusting weapons, such as daggers, dirks, short swords, long swords, rapiers, etc. but no clear definitions seem to exist for all these terms and the classification of a weapon in to one or the other group is often based on personal opinion.[9] Moreover the definitions can change over the course of time (e.g. a Bronze Age "sword" can be of the same length as a Medieval "dagger", so a metric division also creates problems) and the functional use of the object, although often unknown, should also be taken into account. For these reasons I will use the classification *dagger* for the objects presented here. I define a dagger as a blade with a double cutting edge, tapering to the tip, so it is to be used mainly as a thrusting weapon. Although the term "loop-handle" is used in literature, I prefer to name it a ring-pommel because the ring is most probably not used as a handle but as a decorative pommel.

The Belgian team excavated three ring-pommels (AV 079, BL 014 & AT 013). To this at least two better preserved specimens have to be added unearthed by C.S. Phillips during the British campaign at ed-Dur in 1990[10]. These more complete specimens confirmed the suspicion that these ring were in fact part of a type of dagger. One of the British examples (AW 063) was also analysed in the frame of this PhD and is included. The other one was conserved at the Archaeological Institute of London (UCL) and was the subject of a report[11]. So a total amount of five ring-pommels is known from ed-Dur.

AV 079 was not analysed. This pommel came from a large, unplundered, semi-subterranean, vaulted tomb G 5156 in Area AV. The shape of the pommel closely resembles that of AW 063. The entire grave including an outer enclosure, had a length of 9,50 m and was oriented north-south. At least 27 individuals were interred in this tomb. Since the tomb was undisturbed a whole array of burial gifts was found, amongst them iron weaponry and the ring-pommel. It is likely that the pommel AV 079 is associated with a male interment.

Pommel AT 013 was excavated in Area AT in one of the three plundered graves found in this area. Next to the ring-pommel, some bone, some small 'bronze' fragments, a fragment of an iron finger-ring (?), a bent iron fragment with wood traces on the inside (possibly from a scabbard), two arrowhead fragments and one tang of an arrowhead were retrieved. The arrowhead fragments suggest this was a male burial. The remains of the iron handle seem to envelope the central core of the pommel and it was certainly not cast-on the iron tang. It is possible that further down this shaft a rivet was used to keep the two elements together.

BL 014 originated from area BL, where a badly preserved square structure was excavated, possibly the remains of an altar. Some plundered tombs were also encountered. Next to the

[8] Published as Delrue, 2006.
[9] Potts, 1998: 191.
[10] Pers. comm. C.S. Phillips.
[11] This report was unavailable.

ring-pommel, only shells, animal and human bone were found in G 6150. There is no additional indication of the sex of the buried person. A small rivet was used to attach the pommel to the handle.

Pommel AW 063 was excavated in 1990 by the British team in a tomb (G 5437) in area AW. It is the most complete specimen presented here. Since G 5437 is an unpublished tomb, nothing can be said about the excavation context. A lot of iron weaponry was recovered from this tomb however, including arrowheads, swords, spear-/lance-heads, and knife fragments in addition to the presented ring-pommel dagger. Also several 'bronze' objects were attested. We can safely say that this was a tomb with multiple interments, were at least some were male judging from the weaponry.

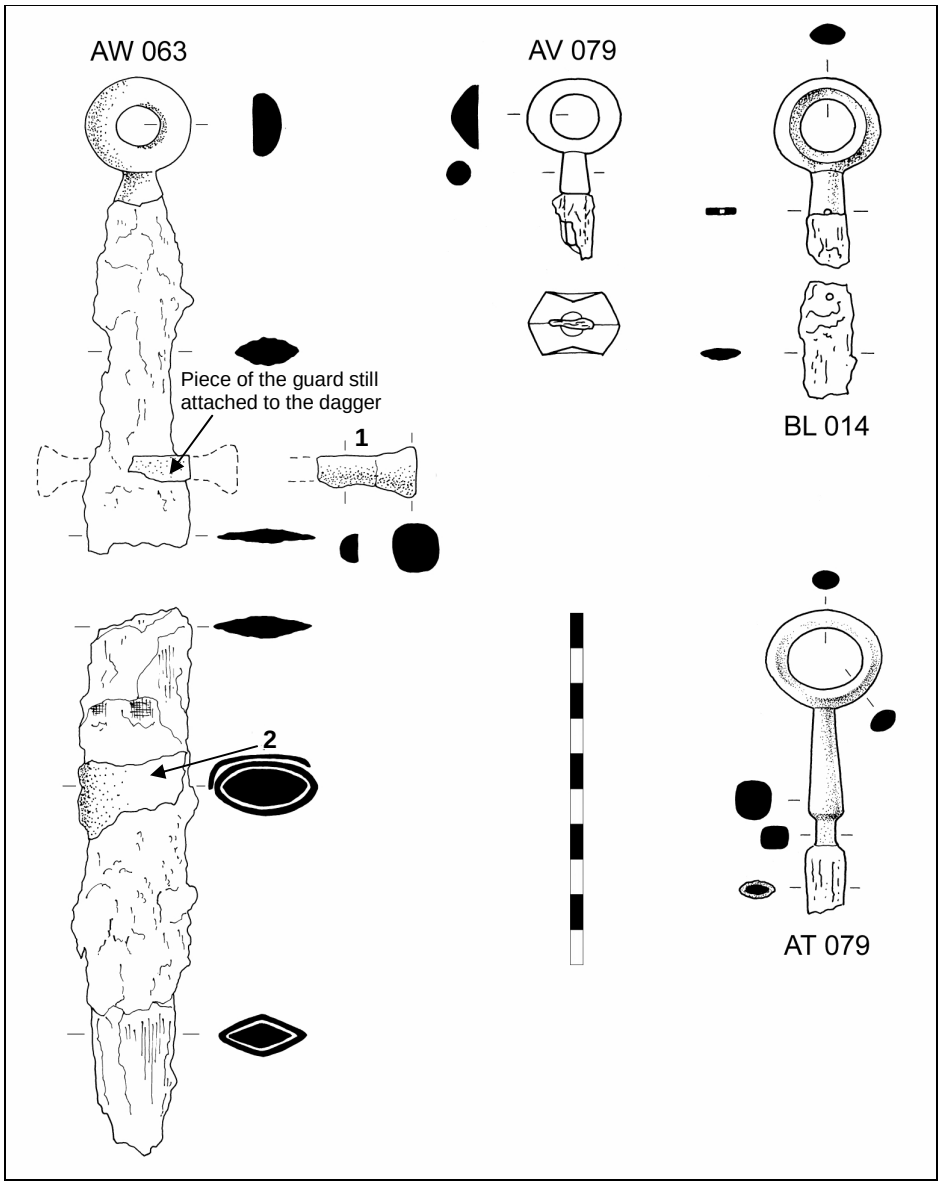


Fig. 153: Drawings of ring-pommel fragments found at ed-Dur.

The attachment system of the pommel of AW 063 on the handle cannot be seen, because of the corrosion on the handle, but the most likely system is with small rivets. The shape of the pommel closely resembles that of AV 079. A small fragment of the copper-base alloy guard is still attached to the handle and a nail-shaped guard fragment was found next to the

handle. The very similar composition of this fragment and the piece still attached to the dagger suggests they were part of the same guard. The preserved blade fragment is oval in section, which implies a double-edged cutting-blade, measuring 28 x 7 mm and no pronounced mid-rib or fuller can be observed. Some information on the scabbard can be deduced from the mineralised remains. Two wooden plates were used as the base for the scabbard. These wooden plates seem to have been enveloped by a thin iron sheet. As mentioned above the British team excavated one more ring-pommel dagger[12].

One fragment of a copper-base alloy fitting can be seen (AW 063-1). This fitting seems to have been a decorative element, since there is no sign of a complete sheeting of the scabbard. It is equally possible that it had something to do with the suspension mechanism. The construction of the scabbard can be compared with that observed on some of the sword fragments at ed-Dur. Since the blade fragment does not actually fit the handle the exact length of the dagger cannot be given, but a minimal overall length of 30 cm with a minimal blade length of 20 cm should be considered. This dagger was previously unpublished.

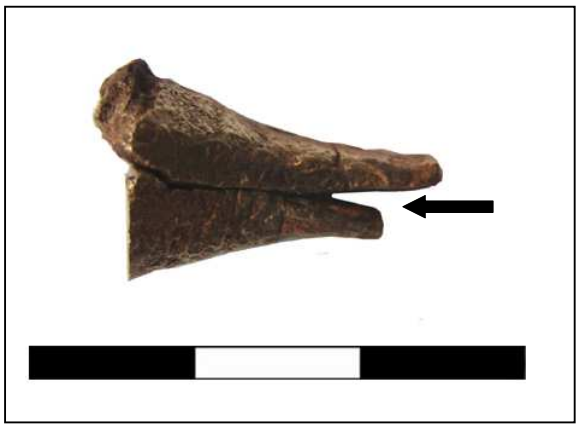


Fig. 154: Fragment of guard AW 021-2 is almost identical to AW 063-3

The nail-shaped fragment AW 021-2 came from the same grave (G 5437) and might thus be part of the same dagger.

These dagger guard was cast in one piece with an opening in the middle. The black arrow indicates the opening the opening where the tang of the dagger past through till above the part where the blade started.

In one case (BL 014) the pommel was attached by small rivets onto the rest of the iron handle. The same technique seems to have been used for AV 079 and AW 063. The iron handle was undoubtfully finished-off with organic elements on the grip, but these were not preserved. In the fourth case (AT 013) the attachment technique was not very clear. AT 013 is also a bit different from the other examples, in that the ring is bigger and has a long round to square stem, maybe with a more functional use. Whereas the other daggers only had a copper-base alloy pommel with a small stem, this one most probably also had part of a copper-base alloy handle.

10.3.2. Microstructural & chemical analyses

10.3.2.1. Metallography

Most samples taken from the daggers were by drilling a small hole and obviously for these samples no microstructural information is available. Only part of the guard (Fig. 153, *1*) of AW 063 and AW 021-2 could be examined under the optical microscope.

The alloy is single phased. The microstructure shows that the metal was worked to shape to a certain extent and properly annealed afterwards to relieve the stress in the metal. The fact that the annealing was done at sufficient high temperature and/or length of time resulted in rather large grains. In the nail-shaped part the annealing twins are straight, indicating that no addition al cold working took place after this annealing phase. For the small part of the

[12] C.S. Phillips pers. comm., who showed me a drawing of the almost completely preserved dagger. No details on the context of this dagger were available however.

dagger still attached to the dagger this is different and slightly deformed annealing twins can be seen near the surface. This part was probably hammered to place after it was slid over the tang of the dagger, to make sure that the guard was fixed.

10.3.2.2. Bulk analyses – SEM-EDX

All analysed ring-pommel drillings (AT 013, AW 063 & BL 014) contain similar amounts of zinc, between 18 and 20 wt%. This makes them brasses with a relatively high zinc level and not the initially thought bronze. The zinc level in the brass is however not as much as a 'fresh' Roman cementation brass, which would normally contain 22 to 28% of zinc. The low amounts of tin detected in some of the samples indicates that the brass was at least ones remelted and probably diluted with metal that contained a small amount of tin. The fact that the fraction of zinc is quite uniform in all analysed fragments, can point to an intentional choice for an alloy of about 20% zinc. Nowadays brasses of 15 to 20% zinc are often used for low-priced jewellery and in foil-form as a cheap substitute for gold leaf, since brass has a golden yellow colour when 10 to 20 % of zinc is present. Polishing improves this effect even more.

The detached nail-shaped fragment of the guard of dagger AW 063 had an almost identical composition to the piece still attached to the dagger. This is a strong indication that they were originally part of the same piece. The nail-shaped fragment AW 021-2 came from the same grave (G 5437) as dagger AW 063. The chemical composition is different however to the piece of guard of AW 063. It is also made of brass but the zinc levels are considerably lower than in the nail-shaped fragment from AW 063 (i.e. *ca*. 15,7 wt% *versus ca*. 20 wt% of zinc). This suggests that one more dagger might have been present in this tomb and excludes the possibility that this fragment was the other side of the same guard.

The fragment of a copper-base alloy fitting on dagger AW 063 turned out to be too corroded to give any accurate compositional information, but no other metallic elements then copper were detected however. A small amount of tin was present (*ca*. 0,4 wt%), but this is no indication for a tin-bronze since the tin should be present in elevated levels due to selective corrosion of the copper. The most obvious explanation for this small amount of tin would be that it entered the copper from the ore or is the result of recycling. It shows however that the copper-base alloy components of the dagger were chosen and different metals were used for different decorative components.

10.3.2.3. Trace elements – ICP-MS

Two main ores can be used to produce brass: *smithsonite* or *calamine* ($ZnCO_3$, carbonate ore) and Sphalerite (ZnS, sulphide ore). The first can be easily reduced to its oxide to be readily used in the cementation process, the latter however has to be roasted first in order to driven off the sulphur. This also caused the zinc to vaporise, but unlike the sulphur that was mainly lost in the fumes, the zinc vapour sublimated in the cooler parts of the roasting furnace. An additional advantage of the roasting was that it purifying the zinc oxide to a certain extent in that only the volatile elements would sublimate (i.e. zinc and some lead if present in the ore). The *calamine process* to the contrary uses the crushed ore with all its impurities. Consequently some of the iron, lead and manganese present in the calamite/smithsonite ore will also be transferred to the brass. Based on the presence of especially iron and manganese it is thus possible to make a distinction between brass made from calamine/smithsonite on the one hand and sphalerite on the other hand.[13]

From an archaeological point of view this has the interesting consequence. In Europe and the Mediterranean the common zinc mineral utilized in antiquity was smithsonite, whereas in the Near and Middle East sphalerite was used from an early date. The reason is that few

[13] Bayley, 1998: 10; Ponting, 1999: 1317; Weeks, 2004a: 246.

viable smithsonite deposits are to be found in the Near and Middle East, but sphalerite on the other hand is available.[14] Significant levels of iron and manganese in finished brass are thus not only indicative for the ore used, but can also give a first clue to the broad origin of the brass. Research on brass has shown that this division largely stands up to the test.

The four brasses from ed-Dur all have elevated levels of manganese and iron. Especially when the brasses are compared to the bronzes. Moreover there is a strong correlation between manganese and iron. This is a strong indication that the brass found at ed-Dur was made from *smithsonite* and this in turn suggests a European or Mediterranean and not a Near Eastern origin.

10.3.2.4. Lead isotope analyses – ICP-MS

Lead isotope ratios can not only be used to look for possible ore sources of the metal used, but it can also be used to compare the artefacts amongst each other. The lead isotope ratios of the brass elements of the ring-pommel daggers group nicely together on the lead isotope ratio plots. This point towards the fact that the same 'ingredients' were used and since brass production seem to have been a specialised activity, and also towards an origin from the same place.

Brass is not the ideal alloy to be analysed for its isotopes. Zinc containing ores almost always also contain have some lead. This lead contributes to the isotopic signature and can blur the origin of the copper used. So the signature is a mixture of both and is makes it hard to determine either of the ore sources. Well aware of this problem three copper ore sources plotted together with the brasses: Spain, Yugoslavia and Cyprus. Spain gave the best overlap, provided that the zinc did not significantly alter the ratios, a fact that cannot be evaluated.

[14] Bayley, 1998: 10; Ponting & Segal, 1998: 118; Ponting, 1999: 1317; Cowell, Craddock, Pike & Burnett, 2000: 677; Ponting, 2002: 562.

10.3.3. Archaeological & iconographic comparanda

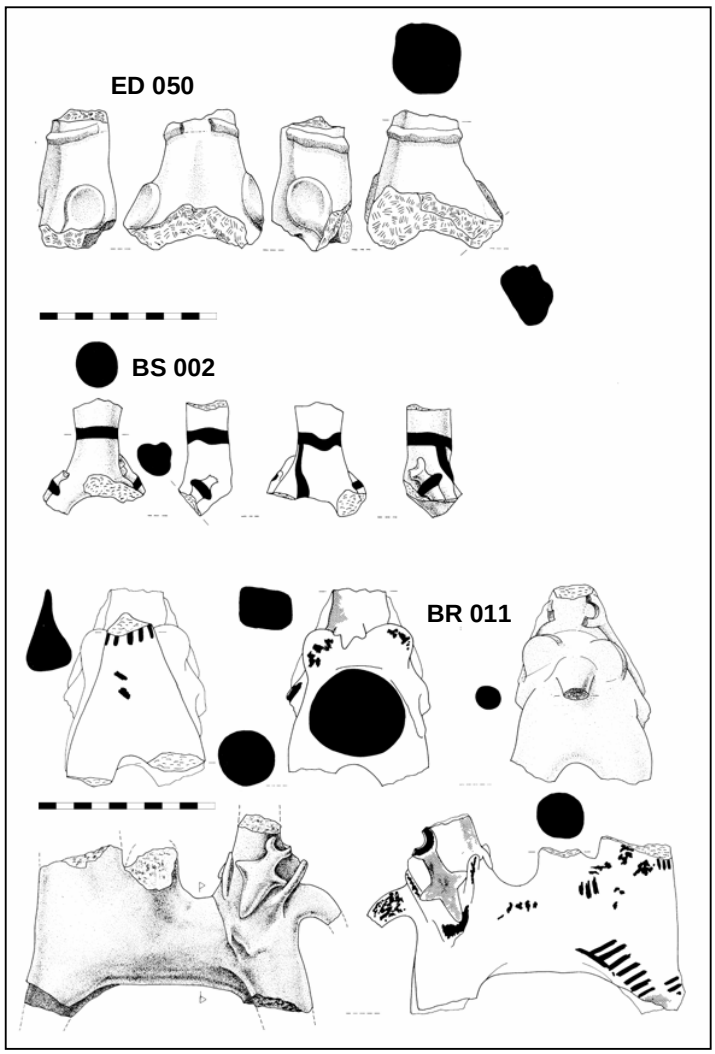


Fig. 155: Three rider-on-animal figurines found at ed-Dur with daggers on both hips.

Next to the ring-pommels three rider-on-animal figurines were found at ed-Dur with the representation of daggers attached to their left and right upper legs or hips (Fig. 155). In one case (ED 050) the pommels are clearly ring-shaped, in the two other examples (BS 002 and BR 011) the pommels are damaged and could also have a crescent-shape. One of the figurines clearly represents a male. Due to the fragmentary preservation no indication of the sex of the person can be seen on the other two fragments. A. Daems already gave some references to similar daggers in other areas in her article on the human figurines from ed-Dur[15].

The wearing of a dagger as a standard piece of equipment has a long history. If we leave aside the ring-pommel for a moment, a kind of lobed dagger with four protrusions seems to originate somewhere in the Altai regions in the 5th c BC[16]. The origin of the ring-shaped pommel as a decorative (or maybe functional) element can be traced back to the Scythians[17], Huns or even the Chinese Han Dynasty from where it spread among the Central Asian Steppe people[18].

Next to the long swords, also short swords (50 to 60 cm) and daggers (30-35 cm), all generally having a right-angled guard and a ringed pommel (Fig. 156-4) are part of the *Middle Sarmatian period* (1st c BC – 1st c AD) weaponry. The characteristic thigh scabbards for short ring-pommel swords and daggers appear from the 2nd c BC and are found in burial-mounds all over the Sarmatian territory[19].

[15] Daems, 2004b: 97-99.
[16] Tanabe, 1985: 111-112.
[17] See Ginters, 1928: Tafel 7c, for an example of an *Aikinakes* weapon with a ring-pommel excavated at Jelisavetovskaja and dated to the Scythian period.
[18] Seyrig, 1937: 30.
[19] Ginters,1928: 56; Trousdale, 1975: 104; Moshkova, 1995: 139-140; Brzezinski & Mielczarek, 2005: 33.

Fig. 156: Iconographic reference material and the most complete dagger from ed-Dur (6).[20]

Such short swords or daggers, probably evolved out of the *akinakes*-sword (45-60 cm), were worn on the right thigh with a leather-strap rope from the scabbard passed around the leg to secure it. The use of the *akinakes* was wide spread among the Indo-Iranian people.

[20] Fig. 1.1 Drawing after Rostovtzeff, 1922: Plate XXX-2; Fig. 1.2 Drawing after Curtis, 2001: 325 (Pl. XIIa); Fig. 1.3 Drawing after Seyrig, 1937: Plate 1; Fig. 1.4 Moshkova, 1995: 140; Fig. 1.5 Drawing from Lombard & Kervran, 1989: 124; Fig. 1.7 Drawing after Boehmer & von Gall, 1973: Tafel 31; Fig. 1.8 Drawing after al-Salihi 1989: 175; Fig. 1.9 Drawing after Harper, 1981: 225.

Sometimes the top pair of straps, or an additional pair of straps, led to a belt hidden under the skirts of the jacket, allowing the height of the scabbard on the thigh to be adjusted.

Several 1st c AD grave steles from the peninsula of Kerch in the Bosporus (Fig. 156-1) show riders in Sarmatian-like dress with a ring-pommel dagger on the right upper leg[21]. This weapon apparently remains popular until the 3rd century AD in this area[22]. From Hungary examples are known dating to the beginning of the 4th century AD[23]. These rings were of iron and as with the swords probably evolved by the closing of the circle on the arc-shaped and antennae-pommel of the previous swords[24].

The *Parthians* were in origin a Central Asian power[25] and it was a Parthian custom to wear one or sometimes two daggers[26]. This can be seen on several steles and rock-reliefs of the Parthian period[27] or on the bronze statue of Shami (wears two daggers, but without pommel)[28]. The preservation of these reliefs is most of the time poor so aside from a rather crude outline of the dagger no details can be observed and no ring-pommels can be attested. If one dagger is worn it is always on the right upper leg. One of the horse riding figurines from Susa also has a crude representation of a dagger on the right upper leg[29]. As pointed out by A. Daems, two belt buckles from the British Museum show a horse-riding figure both with a ring-pommel dagger attached to their upper leg[30]. One buckle shows the dagger on the right upper leg, while the other shows the person with a dagger on the left upper leg (Fig. 156-2). These objects were purchased on the antique market, so they have no real archaeological value[31]. They do however point out that in the Parthian period, Parthians wore daggers with a ring-pommel and a lobed attachment-system. To my knowledge no such daggers have been excavated in the Parthian territories.

The cultural dominance of the Parthians on their client and buffer states seems to have been strong enough to make them imitate the Parthian way of dressing. Parthian costume, as seen after ca. 100 AD, was rarely complete without a lobed dagger attached to the lower right side of the tunic[32]. In *Palmyra* a dagger was worn on the right upper leg, sometimes even on both legs. Some of these daggers ended on a large ring (Fig. 156-3, funerary statue from Kasr el-Abiad, but many others are known)[33]. Daggers with lobed sheaths were used not only by the Palmyrene military or the caravan guards on the desert highways, also civilians and even priests display it as part of their formal Iranian dress. This is a sure indication that the dagger had been transformed from weapon to decorative accessory[34]. Some of the statues at the necropolis of Palmyra were dressed with a ring-pommel dagger on the right upper leg. The *Hatraeans* (Fig. 156-8, this statue actually has a dagger on the right and the left hip) often wear a similar dagger on the right hip and also at *Commagene* (Fig. 156-7, Antiochos I, Nimrud-Dagh) parallels can be found of ring-pommels[35]. The depictions of a ring-pommel on the reliefs of Antiochus are probably the earliest examples that can be closely date, e.g. 69-34 AD[36].

[21] Rostovtzeff, 1922: 169, Plate XXX-2; Ginters,1928: Tafel 30.
[22] Trousdale, 1975: 104.
[23] Ginters,1928: 56.
[24] Brzezinski & Mielczarek, 2005: 33.
[25] Ball, 2000: 12.
[26] Seyrig, 1937: 30; Khorasani, 2006: 81-82.
[27] For examples see Mathiesen, 1992: 150, 155, 156, 155, 168 & 175.
[28] Curtis, 2000: 26, daggers on both legs.
[29] Martinez-Sève, 2002: 495.
[30] Curtis, 1989: 61, Fig. 73; Curtis, 2001: 306, Pl. XIIa, b.
[31] Daems, 2004b: 99; Curtis, 2001: 325.
[32] Colledge, 1976: 153.
[33] Amy & Seyrig, 1936: 239; Seyrig, 1937: 29; Homès-Fredericq, 1963: 35.
[34] Goldman, 1993: 212-213.
[35] Seyrig, 1937: 30.
[36] Winkelmann, 2003: 55.

From the Bahrain tumuli at least one ring-pommel dagger is known from excavations (Fig. 156-5). The dagger is published in the catalogue of the Bahrain Museum and was found in tumulus 36/2 at Jidd Hafs on Bahrain and cannot be dated more closely than to the Early to Middle Tylos period (*ca*. 300 BC - 250 AD) since no information is available on the associated ceramics or material[37]. On the basis of ceramics of adjacent tombs this period may be narrowed down to the "Parthian period" or the Middle Tylos period (100 BC – 250 AD), a date that would fit nicely with the evidence presented here[38]. This Jidd Hafs iron dagger had an iron scabbard (as can also be seen on AW 063), which it is corroded together so the blade is no longer visible, but it clearly tapers to the point and has a two-sided cutting edge. The pommel is of a copper-base alloy as is the guard most probably. The overall length of the dagger is 36,5 cm, the scabbard length is 26,6 cm and is 2,9 cm wide.

According to tradition the basic weapons of a *Sasanian* prince or king were the long sword, a dagger or short sword and a knife. Where we have seen that the wearing of a dagger was a cultural tradition among the Iranian and Semitic peoples such as the Parthians, Commagenians, Hatraeans and Palmyrenes, it seems to have been exclusively worn by the King of Kings in the Sasanian period[39]. Two rock-reliefs that clearly depict daggers date to the period of Shapur I. At Naqsh-i Rajab and Darabgird the king has a dagger attached to his right thigh, although these do not seem to have a ring but a disc as pommel[40]. Discs (or possibly ring-pommels) attached to some daggers, short swords or swords are represented on some of the famous silver bowls of Sasanian origin (Fig. 156-9, silver vessel with Shapur III killing a leopard)[41]. So continuing a Parthian custom, the Sasanian kings often wore a lobed dagger on the right thigh, sometimes partly hidden by the legging. Based on the study of iconographic representation it is not easy to retrieve detailed information on the shape of the daggers. All we can see is that the scabbard tapers to the tip, which ends on a round knob, a detail that also can be seen on the dagger from Bahrain[42]. Often four, or sometimes two, round or U-shaped elements protrude from the scabbard side. The upper two often give the impression that they represent the guard of the dagger, but their link to an attachment system is more plausible. This weapon seems to be attached to the upper right leg and not directly to the arms belt as for example the sword. Based on the size of the representation and the height of the person a rough estimate on the length of the dagger can be made. It should be around 35-45 cm long.

Ring-pommels are one amongst a number of pommel types depicted on Sogdian wall paintings. All of these date to the 7th - 8th c AD and this may be a reflection of a Sasanian influence on the culture of Sogdiana[43]. The custom to sometimes wear two daggers might be echoed by the Tocharian horsemen of the 7th c AD[44].

A last striking appearance of the ring-pommel can also be found in Europe. A type of *spatha* with a ring-shaped pommel (*Ringknaufschwerter*) is known from Germany and surrounding regions[45]. This type can be traced back to the middle of the 2nd c AD, when the use is spread in Roman *Germania* and *Germania Libra*. The spread of this sword (with a possible origin

[37] Boucharlat & Salles, 1989: 123-124; also mentioned by Potts, 1990b: 116, but there it's place of origin seems to be the al-Maqsha cemetery nearby. Based on the reference to Bibby (1954) given by both authors the same dagger is meant. Pers. comm. by Haerinck says that there is probably a second dagger of this type in the reserves of the Bahrain Museum, although this is not confirmed by Andersen (Pers. comm. and Andersen, 2005).
[38] Potts, 1990b: 116.
[39] Tanabe, 1985: 111-112.
[40] For the rock reliefs of Darabgird see Trümpelmann, 1975: Tafel 1 and for Naqsh-i Radjab Vanden Berghe, 1983: 188. When a mounted Sasanian king is represented the dagger is not hanging vertically, but follows the bent of the leg.
[41] For some examples seen on Sasanian silver vessels see Harper, 1981: 217, 225, 229, 230.
[42] Although only small fragments it might be interesting to note that a similar knob was excavated at ed-Dur (by the British team, pers. observation), one at Mleiha (depicted in Mouton, 1992: Fig. 40 (PIR.B) and one at Bithnah (Corboud, Castella, Hapka & im Obersteg, 1996: 157, Pl 25).
[43] Masia, 2000: 214-215.
[44] Seyrig, 1937: 30.
[45] Feugère, 2002: 154.

among the Sarmatians) can be extended to the whole of non-Mediterranean Europe, from Normandy to the Carpathians[46]. A shorter type of this weapon is also known and is generally dated to the 3rd c AD. These have convergent sides or parallel sides and are between 40 and 46 cm long[47].

It is important to notice that two different points are to be made. A first one is that the use of lobed daggers attached to the right upper leg was a tradition that was widely spread in Central Asia and the Middle East. The oldest examples can be found among the Central Asian steppe people. The Parthians, who had their roots in those same steppes, might have brought the tradition to Iran and influenced their neighbours (Palmyra, Commagene, Hatra, Tylos), although direct influence by the Sarmatians can also not be excluded. They also handed down the torch to their successors, the Sasanians, who in their turn seem to have influenced the Sogdians. It is interesting to notice that on the Kushana reliefs no daggers appear[48]. Although the dagger fragments from ed-Dur did not leave any clues on how these daggers were worn, this hiatus is partially filled in by the three rider-figurines, where a two-lobed suspension is shown on the right and left upper leg (although these two lobes could also be the guard). The second point is that the ring-pommel was equally widespread in Central Asia and the Middle East, this of coarse does not mean that all daggers had this kind of pommel.

10.3.4. Conclusions

Both the compositional analyses and the historical setting of the ring-pommel daggers strengthen the picture that emerged from the artefact assemblage of the site, being that ed-Dur was not a site cut of from the world, but was in one way or another connected with the overall trends that appeared.

The uniformity of the alloy, the relatively high zinc percentage and the fact that other metallic elements are present only in small quantities, suggests that the brass was not extensively recycled or diluted. Moreover the average zinc content of about 19,5 wt% is in good accordance with Roman (military) objects with high zinc content and the use of *primary brass*. The uniformity in the zinc percentage points to a deliberate choice of alloy, most probably because of the colour resembling gold. AW 021-1 is somewhat different in composition and has a zinc content that point towards dilution of the alloy or recycling. The colour of the alloy would have been similar however.

Brass appeared as a common alloy during the 1st c BC in the Roman Empire. The trace elemental composition of the brasses found at ed-Dur are strongly indicative for a Roman/European origin and for smithsonite as the source of zinc. The similarity of the isotopic fingerprint is indicative that the 'ingredients' used to produce the brass (i.e. the copper and zinc ores) are very similar, if not the same. On the provenancing question the lead isotope signature is much less useful, since brass contains lead attributed by the copper and the zinc component of the alloy. For what it is worth a Spanish, Yugoslavian or Cypriot origin for the copper can be suggested. The main point is that the overall lead isotopic fingerprint matches best with a Western origin and certainly not an Indian origin.

Although theoretically brass could come from the India Subcontinent, there is a preference for a Roman origin. This is underpinned by circumstantial evidence from the lead isotopes and trace elements detected. Archaeologically the early brass production in India is not completely clear and based on the finds at ed-Dur the contacts with the Roman world (direct or indirect) were much more intense than with India. The *Periplus* states that the merchants

[46] Feugère, 2002: 157.
[47] Feugère, 2002: 159-160.
[48] Winkelmann, 2003: 67.

from Roman Egypt exported among other goods, brass to Adulis on the African shore, where it was used for making personal adornments[49]. Nowhere is it mentioned that India exported brass to the West. If Roman brass was exported to Adulis, it is very possible that it was also available on other markets.

There is no material proof that copper-base alloys were worked at ed-Dur and certainly not that brass was produced. At the inland site of Mleiha traces of "bronze"-working were found, but there no ring-pommel daggers were attested so far. As described above there is a clear link between the Parthian Empire and their client states, and the use of ring-pommel daggers. The suggestion that these daggers were imports is almost certain. Unfortunately, except for the dagger from Bahrain, no direct archaeological *comparanda* are known. The Sarmatian examples seem to have iron pommels instead of the copper-base alloy ones attested at Bahrain and ed-Dur. For the time being this detail can be seen as a typical NE- and SE-Arabian characteristic.

The use of 'Roman' brass for 'Parthian' objects is an interesting suggestion, since it is indicative of trade between the two empires. In this frame the rather friendly relation between the two sworn enemies during the first part of the 1st c AD can be important. The high zinc level would be in accordance with exported brass ingots that were remelted ones to produce an object, in this case the dagger elements. Moreover the only known brass ingots found in a shipwreck in the Mediterranean Sea contained around 21% of zinc. Brass could have reached the Parthian Empire in different ways. The first would be via the Red Sea to S-Arabia, from where it went up the Gulf by a secondary trade network. The *Periplus* makes clear that brass objects travelled in that direction to the African ports. The second way would be that the brass was exported to the Indian Subcontinent and from there re-exported to the Gulf. This route is also mentioned in the *Periplus*, but brass is not amongst the exported goods listed. The third option is that the brass went overland via Palmyra towards Characene, or to any other place in the Parthian Empire.

To me the last hypothesis seems the most likely, since the others are rather time-consuming. It has to be admitted however that this is not a very strong argument. Still I would like to suggest that 'fresh' brass ingots or high zinc brass objects reached the Parthian territories. There the brass was remelted (maybe slightly diluted) and used for the production of local objects, e.g. the elements for ring-pommel daggers. The finished daggers were then exported to ed-Dur via the 'Characenean corridor' down the Gulf. That the link between ed-Dur and Characene was strong is, as mentioned above, also evidenced by some coins and more importantly the mass of S-Mesopotamian ceramics found at the site. The ring-pommel dagger from Bahrain fits very well in this puzzle, since the island was most probably part of the Characenean network.

The daggers are likely to have been part of the standard male weaponry at ed-Dur. In origin these ring-pommel daggers seem to have been associated with riders and the attachment system on the upper leg was undoubtedly designed not to hinder the horsemen in their movement. Later on the attachment system becomes less clear. The dagger is worn on the upper leg, but seems to be attached directly on the trousers or the tunic, since no straps can be seen running around the leg. If this is a realistic depiction or rather an artistic formality is not clear, but if these daggers were directly attached to the clothing they would have lost their practical advantage. Such an evolution would fit the interpretation that these daggers were more a decorative/prestige element than a functional one. This is illustrated by for example the banquet scenes in the Palmyrene tombs and the standing statues from Hatra.

The cultural tradition of wearing a (ring-pommel) dagger was carried by a wide variety of people and over a vast territory and time span. These daggers have long been attested on

[49] Casson, 1989b: 20.

the sculptures in the Parthian, Palmyrene, Hatraean and Commagenean territories, now we can also add NE- and SE-Arabia to this cultural tradition. SE-Arabia was not just a territory at the fringes of the large Parthian realm, but was an actual part of its cultural sphere.

Chapter 11. GENERAL CONCLUSIONS

"A conclusion is simply the place where someone got tired of thinking."

Anonymous

• Introduction

Ed-Dur is not a metallurgical important site in the sense that it is not a site that was involved in metal production or the large scale working of metal. Still the archaeometallurgical study of its remains provides an interesting additional insight in the larger picture. This is born out of the broad spectrum of metals and metallurgical remains looked at here. The remains include an array of metals (iron, copper and its alloys, silver, billon, and lead and its alloys) and some working remains (iron smithing slag and litharge). Additionally a large coin collection was analysed. Next to the pure metallurgical study the results were as much as possible fed back to the archaeological context to draw conclusions that add to the understanding of the site. The fact that all these metals were researched in a single study brings a wide range of information, although some depth may be lost.

A broad range of materials needs an equally wide range in analytical techniques to characterize these materials. The basic analytical tools used were optical metallography and SEM-EDX (scanning electron microscope with electron dispersive x-ray spectrometry). In the second line ICP-MS (inductively coupled plasma mass spectrometry) and XRD (x-ray diffraction) were deployed. To tie up some loose ends a limited number of AAS (atomic absorption spectrometry*)* and pertrographical analyses were preformed. The main analytical objective was to study the microstructures and determine the bulk chemical composition, supplemented with local analyses of inclusions. To this first objective the study of trace elements, lead isotopes and mineral composition was added.

• Balance between set & achieved goals

The analytical results were presented in the interim conclusions and *Chapter 10.* In this final chapter I would like to evaluate the balance between the set and the achieved goals, a moment of introspection. The five goals summed-up here were defined in *Chapter 1*, the sixth point gives the last general remarks.

1. The determination of the alloys used and the identification of possible new ones.

This was done for all available samples and generated a good insight in the copper-base alloys 'used' at ed-Dur. The word 'used' is however misleading since it implies that metal was worked and alloyed at the site to produce objects. This is however not the case and the alloy assemblage only tells something about the alloys that reached the site. The participation in the choice of alloy might have been minimal or even non-existing for the inhabitants of ed-Dur. No remains were found at ed-Dur that could be related to copper and/or copper-base alloy working. For the moment it is impossible to completely evaluate the extent of the metalworking that went on at Mleiha. In Mleiha remains related to copper and/or copper-base alloy working were found but the archaeometallurgical study on them is not clear on the amount, the type, etc. As it stands now this cannot be evaluated.

Copper and bronze are of course the most expected candidates to turn up in the assemblage. The metalworkers seem to have been well aware of the bronze variants they used and certain compositions seem to have been preferred for certain object classes. But this needs not to be a surprise in the period under consideration.

The brass and gunmetal can be considered 'new' or rather unexpected alloys. Brass makes up a fair potion of the analysed material and seems to have been mainly used for decorative functions. The compositional results show that most of the brass was only remelted a limited number of times and based on the trace elemental levels of manganese and iron, a 'Western' origin is suggested.

The identification of litharge fragments was a complete surprise. The samples were originally labelled as slag. This is the first evidence of a cupellation technology in SE-Arabia and the litharge from ed-Dur is the oldest evidence of this technique in the whole of Arabia.

The lead and silver were less informative since they were rather pure. Some soft and hard solder and possible a pewter fragment turned up amongst the samples however. The low trace elemental levels of silver in the lead suggest that the lead was possibly the waste product of silver extraction. One sample had more silver included, and as it turned out this sample is also from a different origin.

SEM-EDX is a good tool to do a fast bulk analyses, but care has to be taken, since it is a semi-quantitative technique. More time should have been spent on the analysis of certified standards, and to 'calibrate' certain results when necessary. In a few cases the EDX data was crosschecked by quantitative techniques (i.e. ICP-MS & AAS) and the results were in most cases in rather good accordance. I do not think the analytical results were 'over interpreted'. The broad alloy groups determined here would survive a more accurate analysis.

2. The analyses of the collection of SE-Arabian coins.

The coins are treated as a separate group. They were analysed by SEM-EDX for their chemical composition. The majority of the coins were made from silver or a copper-silver alloy. One copper coin was attested and one specific group contained small amounts of tin. True bronze coins were not attested, and it can be questioned if bronze coins were ever produced. It also shows that the intuitive determination of alloys is often wrong. The same 'mistake' was made with the brass artefacts and as seen in this study the correct interpretation of the material can lead to new and additional insights.

The compositional data of the coins was projected on the typology of the coins and there is a certain relation between the alloys used and the type of coins. Evidence was brought forwards for a pickling process used to enrich the coin surface with silver in an artificial way. It is also suggested that the coin blanks may have been hot struck.

The hypothesis that the SE-Arabian coins were made locally also implies that the coining metal may be alloyed locally. This can be true, since the alloys used in the (limited amount of) foreign coins analysed in this study, are completely different. If the production hypothesis is accepted then an additional circumstantial clue is found to address the local technological level. As stated several metallurgical technique are attested amongst the coins and although the depiction level never reached that of large monitory systems, a process of pickling does suggest a certain technological level. In any case the analysis of the coin collection is a valuable addition to the numismatic study of the coins of the region.

The trace elemental and lead isotopic analyses performed on 18 coins were not evaluated in this study and are kept for a future publication. The data is however not very willing to release its secrets and can be seriously biased by recycling of the coin metal. Therefore the analytical results have to be approached with the necessary caution.

3. Determining to which metallurgical process the slag belongs.

The analyses of the slags made it clear that they are related to smithing. No iron was produced at ed-Dur, nor was a previously suggested crucible process applied to obtain steel. The smithing activities seem to have been on a rather basic level. The absence of fayalite shows that sand was not added as a flux to keep the working surface free of the iron oxide. This is essential when more complex smithing activities are undertaken such as welding or steel working. The absence of the use of a flux can also be related with working wrought iron, since little oxides are generated. All this fits the small size of the slags and the shaping of small objects, i.e. small scale working of wrought iron.

The time spent on the slag analyses and the page space given to the slag may seem too much for the rather simple conclusion: the slags are the remains of smithing activities. Slag is however not a simple material to study and much time was needed to get an understanding of the basic slag analyses. The distinguishing between several types of slags is even for a trained archaeometallurgist not straightforward. The many microstructural and SEM-images included are meant to fully characterise the analysed slags and ease any comparison in the future. The high calcium levels in the slag, which seems to be a characteristic for the Gulf iron smithing slags, could not be explained. The only suggestion that can be made is that it originates from the lining used in the smithing hearths.

4. Addressing the provenance of the metals.

To address the provenance problem the analytical techniques suggested in the original project were not sufficient. For this reason it was decided to start an additional research track on the lead isotope composition of a selection of samples, by far the most common method within archaeology to tackle the question of metal origin. Unfortunately it is also a technique that has some drawbacks and needed the necessary caution in interpreting the data.

The lead isotope research is not completely finished and some data remains unevaluated, i.e. that of the coins and part of the copper-base alloy samples. Still the results were illuminating. The metal that arrived at ed-Dur as objects or raw material seems to primarily originate from the 'West'. Especially for the lead this is very clear and without any doubt Spain and Sardinia were the providers of the lead. Two fragments are related to an Indian origin however. This is completely new information and seems to contradict the *Periplus*. It could however be that a chronological argument is at play here and that the two fragment of Indian origin date from the second half of the 1st c AD. This is the period that the Indian mines of Zawar (Rajasthan) opened-up, as evidenced by the study of Indian lead coinage. In any case the two ed-Dur samples are the first to give an analytical backing to the fact that India was exporting (and thus certainly producing) metallic lead before the 2nd c AD. Also one of the Khor Rori bronzes seems to originate from the same Indian region.

The silver originated from several sources and probably the ore fields from Great Britain were the main providers, next to Sardinia and Spain. Other regions could however not be excluded. The copper-base alloys are the least clear to interpret, Spanish and Sardinian ores together with ore from Cypriot cover a large portion of the samples, but other ore sources also show overlap with the copper-base alloys samples. Moreover some of the samples are not covered by any of the data from the composed database, showing a shortcoming in the database. It is also possible that these samples from ed-Dur originated from an as yet unanalysed ore sources. Further research is needed for the copper and copper-base alloy samples.

The lead isotope research would have benefited from a more conscious selection of the copper-base alloy samples. A large portion of the selected copper-base alloy samples was leaded and this makes it difficult to interpret their lead isotope signature, since it is a mix of

the isotopes contributed by the copper and lead. It should however be said that these samples were submitted for LIA, before their composition was determined. These samples originated from the most interesting artefacts analysed The selection of the samples to be analysed was thus not based on their alloy, but on their archaeological relevance.

It was also experienced that the interpretation of LIA is not as straightforward as thought and to get a thorough interpretation, some more expertise is needed. The results presented in this PhD have to be seen as a sketch of the situation and a steppingstone to further research. This research will undoubtfully add new information to the already presented results.

5. Link the new data and add new information to a better understanding of the site of ed-Dur.

Up until now especially the analyses of the brasses and the lead added an interesting new angle of information. Based on the analytical results, a hypothesis on the way these metals and/or objects reached ed-Dur was proposed. It seems likely that both the lead 'ingot' and the brass used in the ring-pommel daggers passed through the Parthian and Characenean territories. This makes it very likely that they reached ed-Dur via the '*Characenean corridor'*. This hypothesis reinforces the already proposed strong link that ed-Dur had with (the commercial activities of) Characene.

The appearance of two lead fragments at ed-Dur and the one bronze sample from Khor Rori from Indian origin however shows that the network providing metals to the Gulf region was complex.

6. Some general remarks

As a whole the original project would have benefited from a better defined frame and phrasing of the questions that had to be addressed, at the very beginning. This would have made it possible to include the necessary analytical tools from the onset, i.e. lead isotope analyses combined with trace elemental analyses. Considerable time was lost in setting up this additional hatch and the definitive results came rather late. The lead isotopes and the limited amount of trace elemental data however proved their value and probably make up the most innovating part of the PhD.

The XRD results did not provide the additional information hoped for, i.e. the complete mineral characterization of the slags. Looking in retrospect this analytical technique could have been left out. On the other hand nothing ventured, nothing gained.

The exclusion of the chapter on the typo-chronology of the iron artefacts may be considered a pity in the overall study of the *archaeologica* from ed-Dur, but on the other hand it would have added nothing to the archaeometallurgical study presented here. Again looking in retrospect this study would have benefited more from the inclusion of the typo-chronological study of the copper, copper-base alloy and silver objects, since this adds information to the metallurgical study. Luckily A. De Waele was willing to share some of her work on the small finds and let me include some of her information, in advance of her own PhD.

Looking back, *Chapter 2* is to elaborate for this PhD, since it serves no other purpose then to situate ed-Dur in the overall historical/political/economical frame of the 2nd c BC – 5th c AD. How interesting this may be, it does only add little to the archaeometallurgical work presented here. *Mea culpa,* but cutting it out of the final draft was a bit too painful.

- **Future research**

As a whole I think the metallic assemblage of ed-Dur is sufficiently characterized by this study, and the remaining unanalysed portion of the collection, would probably not yield much additional information. A more elaborate trace elemental analysis program by ICP-MS to supplement the lead isotopic data would have added an additional dimension. Especially when such a research project was done over a larger amount of contemporaneous sites in the wider region. Especially the inclusion of some material from Mleiha would have been interesting for the study of the complete chronology of the PIR period, and not only one facet of it, i.e. PIR C. Also the metallurgical remains from Mleiha related to copper and/or copper-base alloy working deserve further study in relation to the data presented here and the understanding of the origin of the metals used.

A more profound examination and interpretation of the lead isotopic data would definitely yield more insight, keeping the problem of recycling in mind of course. A broader research program that includes artefact (*e.g*. Parthian, Bahraini, S- and SE-Arabian, … material and ore samples) from the larger Gulf area would be useful. Despite a lot of research on the Oman copper ores their lead isotopic fingerprinting is far from complete. The (published) dataset remains rather limited for crucial regions such as Oman, Iran and the Indian Subcontinent.

In the margin of this research an interesting research project concerning the analyses of metal slags can be suggested. Slag is a difficult material to examine and different research groups use different methods. The research of slags might benefit from a systematic study of a selection of slags by different analytical techniques, and especially the way the data generated are related to each other. In this way analytical parameter for the different techniques could be synchronised and more easily compared. Within this analytical package the following techniques should be included: registration strategies and simple metrical recording, visual examination, sample strategies and preparation, optical microscopy (reflective, but also by polarized light), pertrographical microscopy (transmission), SEM-EDX, XRD, XRF, ICP-MS/OES and wet chemistry. The result could be combined in some kind of 'manual' that can be consulted to place the results of the analytical data obtained by one of the techniques in a broader frame. This might trigger more systematic analyses of slag and their inclusion in reports on archaeometallurgical remains.

The original plan was to also include the typological study of the iron artefacts found at ed-Dur. This study is in a very advanced state, but not far enough to include it in the final version of this PhD. In the end the chapter was omitted, "close but no cigar", due to time restrain (yes even after five years). The iron objects were well enough preserved to study their basic typology, but the metal was too severely corroded to yield any metallurgical information. Hence it can be argued that their study is not part of an archaeometallurgical based PhD. Moreover except for the archaeological and cultural information they can yield, the iron objects do not add any significant information to the archaeometallurgical knowledge of SE-Arabia. The microstructures were much to corroded to yield any metallurgical information. One exception within this study are the ring-pommel daggers. These were include to show that the combination of metallurgical and archaeological/historical information can lead to new insights. This is what *archaeometallurgy* has to stand for.

"There is nothing in a caterpillar that tells you it's going to be a butterfly."
B. Fuller

I do certainly not intend to say that this PhD is a butterfly, but the carton box in which all the plastic sample bags came to me, did not even hint towards the fact that they would yield any useful information. At least for me they did.

DUTCH SUMMARY – '*NEDERLANDSCHE*' SAMENVATTING

"Hebban olla uogala nestas bigunnan hinase hi(c) (e)nda thu uuat unbidan uue nu"
"Hebben alle vogels nesten begonnen, behalve ik en jij. Waarop wachten we nu?"

1. Introductie[1]

Het doel van deze doctoraatsverhandeling is de archeometallurgische analyse van de metaalmonsters verzameld tijdens de Belgische opgravingen van de Universiteit Gent te ed-Dur (Umm al-Qaiwain, Verenigde Arabische Emiraten). De site van ed-Dur is gelegen op de westkust van de Peninsula van Oman. Het is de enige grote kustsite die tot nu toe is geattesteerd tussen Katar en de Straat van Hormuz. De belangrijkste bewoningsfase van ed-Dur is gedateerd tussen de 1ste eeuw v. Ch. en de eerste helft van de 2de eeuw n. Ch. De opgraving bracht de resten van minstens één fort en een tempel aan het licht. De tempel was gewijd aan de Semitische zonnegod Shams/Shamash en rond het gebouw werden verschillend altaren aangetroffen. Verder zijn er nog een aantal gebouwen in *beach-rock* en gepleisterde vloeren opgegraven, maar het merendeel van de bewoningstructuren was waarschijnlijk uit vergankelijke materialen opgetrokken. De overgrote meerderheid van de architecturale resten zijn afkomstig van verschillende types van graven.

2. Analytische kader

De metallurgische monsters omvatten koper en koperlegeringen, lood, zilver, loodglit, een collectie locale Zuidoost Arabische munten, ijzer en metaalslakken. De microstructuur van deze ruime waaier aan materialen werd via optische metallografie onderzocht. De globale chemische samenstelling werd bepaald via SEM-EDX. De analyse van de loodisotopen en de sporenelementen concentraties werd uitgevoerd via ICP-MS op een aantal monsters. Een beperkt aantal slakken werd onderzocht op hun mineralogische samenstelling door middel van XRD.

Al deze informatie werd aangewend om de metallurgische collectie te karakteriseren en de verkregen informatie in een ruimer kader te plaatsen. Het ultieme doel is om gegevens aan te brengen die kunnen dienen om de functie van het relatief kort bewoonde ed-Dur te verduidelijken. De vele buitenlandse objecten die opgegraven werden tonen aan dat ed-Dur op één of andere manier was geïntegreerd in de grote handelsnetwerken van toen.

3. Koper en koperlegeringen

Naast de verwachte koperen en bronzen objecten, werden er ook twee andere legeringen vastgesteld: messing (een koper-zink legering) en *gunmetal* (een koper-zink-tin legering).

De koperen monsters vertegenwoordigen 22% van de totale collectie. Het meeste uitzonderlijk object is een 'altaar' kraal die verguld was. De techniek die waarschijnlijk gebruikt werd om het goud aan te brengen is amalgaam vergulding.

Brons is de meest omvangrijke groep en vertegenwoordigd 51% van de geanalyseerde stalen. De bronzen monsters kunnen in drie sub-groepen worden opgedeeld, brons met lage (minder dan 5%), middelmatige (5 tot 15%) en hoge tin (meer dan 15%) fractie.

[1] In deze samenvatting komen geen referenties voor. Deze samenvatting is enkel bedoeld om als introductie en vermeldt enkel de belangrijkste resultaten. De uitgebreide historische inleiding en het hoofdstuk over de handel is dan ook volledig weggelaten. Voor de volledig gerefereerde en gedetailleerde versie moet ik de lezer jammergenoeg doorverwijzen naar de Engelse hoofdtekst.

De bronzes samples met lage concentratie tin bevatten grote hoeveelheden lood en het tin is waarschijnlijk aanwezig om het smeltpunt te verlagen. De bronzen voorwerpen met een medium concentratie aan tin zijn de grootste groep. Dit hoeft geen verrassing te zijn gezien dit een legering is die voor vele toepassingen kan dienen. Sommige artefact groepen bestaan uit een redelijke uniforme samenstelling, wat wijst op bewuste legereingskeuze.

Twee monsters hebben een uitzonderlijk hoge tin concentratie. Deze legering heeft een zilver grijze kleur en is erg bros, en is dus maar voor weinig toepassing geschikt. De twee fragmenten zijn afkomstig van spiegels. Deze hoge tin legering is uitermate geschikt om spiegels te vervaardigen, gezien ze gepolijst kan worden om een spiegelend effect te verkrijgen. Dergelijke spiegels zijn gekend uit het Romeinse en het Chinese Rijk. Op analytische basis kan geen van deze twee gebieden worden uitgesloten, maar in de algemene archeologische context van ed-Dur is een Romeinse oorsprong realistischer.

Het feit dat een belangrijk deel van de geanalyseerde objecten (20%) van messing was gemaakt, is een verrassend resultaat. Er zijn sterke aanwijzingen dat de messing van Romeinse oorsprong is, hoewel het soms is gebruikt om objecten te vervaardigen die zeker niet Romeins zijn. De zinkwaarden zijn relatief hoog (gemiddeld 18 wt% Zn) en dit wijst op een beperkt aantal hersmeltingen van het oorspronkelijk 'primaire' messing. Een andere optie is dat een beperkte hoeveelheid koper of misschien zelfs brons werd toegevoegd om het primaire messing te verdunnen. In grote lijnen komen de zinkwaarden in het messing gevonden te ed-Dur goed overeen met de gemiddelde waarden gepubliceerd voor het Romeinse Rijk. De voorkeur voor een bepaald percentage zink in de legering is hoogstwaarschijnlijk gelinkt aan de gouden kleur die het metaal heeft.

De sporenelementen (mangaan en ijzer) geattesteerd in de messingmonsters zijn een sterke aanduiding voor het gebruik van *smithsoniet* als zinkerts. Dit is op zich weer een aanwijzing voor een Europese of Mediterrane oorsprong, gezien in het Nabije en Midden Oosten een ander erst, *sfaleriet*, werd gebruikt als bron van zink. De export van messingen vaatwerk vanuit het Romeinse Rijk naar de oostkust van Afrika, wordt in de tekst van de *Periplus* (dateert uit de 1ste eeuw n. Chr.) vermeld. Voor één bepaalde artefact groep, *ring-pommel daggers*, zijn de degenknopen uit messing vervaardigd. Deze objecten zijn cultureel echter in het Parthische invloedsfeer te plaatsen. We zitten dus met een typisch 'Parthisch' object dat metaal bevat dat uit het 'Romeinse Rijk' komen.

Dit vertelt iets over de weg die het metaal kan hebben afgelegd. Er bestaan tekstuele bewijzen bestaan dat messing werd uitgevoerd tijdens de 1ste eeuw n. Chr. vanuit het Romeinse Rijk naar Oost-Afrika. Als dit mogeleijk was dan kan dit ook in een andere richting zijn verlopen, nl. richting het Parthische Rijk. Tijdens de 1ste eeuw n. Chr. waren de relaties tussen deze anders gezworen aartsvijanden eerder ontspannen en handel zal dus zeker mogelijk zijn geweest. Het messing werd dan verwerkt in locale produkten die dan op hun beurt werden geëxporteerd naar ed-Dur.

Naast messing is ook *gunmetal* (kanonmetaal) een eerder onverwachte legering (7% van de totale assamblage). De geschiedenis van *gunmetal* is echter dicht verweven met deze van messing. G*unmetal* is het resultaat van het samen recycleren van brons en messing, wat een drievoudige legering oplevert, nl. met koper, zink en tin. *Gunmetal* mag op dit moment van de geschiedenis hoogstwaarschijnlijk niet als een bewust gemaakte legering worden gezien. De samenstelling van het *gunmetal* gevonden te ed-Dur is echter anders dan dat van Romeinse oorsprong. Bij de Romeinse variant is het gehalte aan zink normaal groter dan het gehalte aan tin. Bij de meeste monsters van ed-Dur is dat juist het omgekeerde, behalve voor één object. Het *gunmetal* op ed-Dur is hoogstwaarschijnlijk het resultaat van de recyclage van een brons met middelmatige tin niveau en messing. Dat dit hoogwaarschijnlijk onbewust gebeurde kan afgeleid worden uit de samenstelling van een armband. Deze *gunmetal* armband (S 0022) heeft een tin fractie die overeenkomt met de concentratie van tin

in bronzen armbanden. S 0022 heeft daarnaast echter een klein percentage zink in de legering. De kans dat het zink per toeval in de legering terecht kwam is dus reëel.

De loodisotopen analyses van een selectie koper en koperlegerings monsters is nog niet volledig afgerond. Ze vormen echter een eerste schets van het potentieel.

Het meest interessante monster komt echter niet van ed-Dur maar van een gelijktijdig bewoond site in Oman (Khor Rori). De isotopische analyse toont aan dat dit fragment hoogstwaarschijnlijk uit de Indische regio van Rajasthan komt. De *Periplus* en Plinius vertellen beiden echter dat India in de 1ste eeuw n. Chr. koper invoerde van het Romeinse Rijk, wat dan weer impliceert dat er in India geen locale productie was. De attributie van het fragment van Khor Rori aan een Indische oorsprong impliceert echter dat er wel een locale productie was en zelfs een export. India lijkt dus een surplus aan koper te hebben ingevoerd om het dan terug uit te voeren (staat in de *Periplus*) aangevuld met locaal metaal.

Het feit dat bepaalde objecten die hoogstwaarschijnlijk Romeins zijn, vb. een *patera* en een sokkel voor een figurine, een isotopische signatuur hebben die zeer dicht licht bij die van objecten die van locale ZO-Arabische makelij lijken, vb. een giettuit in de vorm van een paard en de wijn diggustatie set, is onverwacht. Dit toont aan dat de metalen die gebruikt zijn van een gelijkaardige oorspong zijn en dat in dit geval Romeins metaal ingevoerd moet zijn in in ZO-Arabië.

Het exact bepalen van de erts gebieden waar het metaal van afkomstig is, is voorlopig nog niet afgewerkt. Een eerst selectie van erstvelden lijken een oorsprong voor het koper in Spanje, Sardinië en/of Cyprus te suggereren. Andere ertsgebieden tonen echter ook overlappingen en op dit moment kan nog niet uitgemaakt worden welke regio's de juiste zijn.

4. Lood, silver, hun legeringen en loodglit

De SEM-EDX analyses hebben weinig informatie opgeleverd in verband met het lood, gezien het lood relatief zuiver is. Wel zijn er enkele fragmenten zacht soldeersel (tin-lood legering), eventueel een fragment *pewter* (tin-lood legering) en een tin fragment geattesteerd.

Het zilver was relatief zuiver, maar bevatte een kleine hoeveelheid koper. Dit kan het gevolg zijn van een onvolledige purificatie, maar kan ook intensioneel toegevoegd zijn om het zilver harder te maken. Er is ook een fragmentje hard soldeersel of *billon* (zilver-koper legering) aangetroffen.

De loodisotopen van de loden objecten tonen aan dat het metaal van Europese oorsprong is, hoogstwaarschijnlijk van ertsen in Spanje of Sardinië. Deze conclusie wordt ondersteund door de tekstuele informatie van de Periplus. Deze tekst zegt dat het Romeinse Rijk lood exporteerde via de Rode Zee naar India. Een tweede optie is dat lood opnieuw werd geëxporteerd vanuit het Indisch Subcontinent. Het lood van ed-Dur zou echter ook via een andere weg aangevoerd kunnen zijn, namelijk lang de tweede grote handelsader de Characenean corridor. Een object met een stempel die gelijkenissen vertoont met voorbeelden uit Characene, zou er echter op kunnen wijzen dat het lood via de Characenean corridor werd aangevoerd. Twee geanalyseerde fragmenten hebben een volledige andere isotopische signatuur dan de andere lood samples en zijn erg waarschijnlijk van Indische herkomst. Als dit waar is, dan wordt de stelling in de Periplus dat India op dat moment geen lood produceerde weerlegd.

De loodisotopische signatuur van het zilver toont een duidelijke overlapping met ertsen afkomstig uit Groot-Brittannië. Dit onderlijnt nog eens het uitgebreide handelsnetwerk dat bestond in de periode dat ed-Dur werd bewoond.

Een erg onverwachte vaststelling was de ontdekking van drie stukjes loodglit. Deze fragmenten zijn het afvalproduct van een proces om het meer kostbare zilver te ontrekken aan een zilver-koperlegering. De technologie van cupellatie was voorheen nog niet geattesteerd in de Golf regio tijdens de 1ste eeuw v. Ch. - de eerste helft van de 2de eeuw na Ch.

5. Locale ZO-Arabische munten

Voor het eerst werd een relatief grote collectie locale ZO-Arabische munten systematisch geanalyseerd (*ca*. 8% van alle gepubliceerde exemplaren). De analyses tonen aan dat er een brede relatie is tussen de typologie en de chemische samenstelling van de verschillende types. Dit wijst op een bewuste keuze van bepaalde legeringen. Daarnaast werden er aanwijzingen gevonden die impliceren dat het zilveren oppervlak van sommige munten kunstmatig werd verrijkt met zilver. Een beperkt experimenteel luik werd opgezet om de beperkingen van een dergelijk proces te onderzoeken en bevestigde dat een legering met maar 10% zilver via de juiste behandeling een mooi zilveren oppervlak kan krijgen.

Maar één munt kon in twee gezaagd worden om de microstructuur te bestuderen. De sectie was minder informatie en duidelijk als gehoopt, maar als alle observaties naast elkaar worden gelegd kan er gesuggereerd worden dan deze munt warm geslagen is.

6. IJzer

De ijzeren objecten waren te gecorrodeerd om bruikbare informatie op te leveren en het onderzoek kon dus jammergenoeg niet uitgebreid worden voor dit metaal.

7. Metaal slakken

De metaalslakken die werden gevonden tonen aan dat er smeedactiviteiten hebben plaatsgevonden op ed-Dur en dat de slakken niet gerelateerd zijn aan een ander metallurgisch proces. De chemische samenstelling van de slakken is vergelijkbaar met de beperkte gepubliceerde gegevens voor andere slakken in de Golf regio, maar verschilt van deze van Europees materiaal.

Het verschil zit hem in het hoge calcium gehaltes en de afwezigheid van fayaliet (Fe2SiO4, een ijzer-silicaat verbindinging) in het ed-Dur materiaal. De afwezigheid van fayaliet wijst op het feit dat er geen zand als flux is gebruikt om het smeedoppervlak oxidevrij te houden. Dit is cruciaal als men verschillende delen wil samenwelden. Anderzijds kan het ook wijzen op het gebruik van ijzer met een zeer laag koolstofgehalte, dan is er geen flux nodig. De kleine afmetingen van de slakken wijzen dan weer op het vervaardigen van kleine objecten.

Het kan dus gesuggereerd worden dat het technisch niveau van de ijzerbewerking te ed-Dur relatief laag was en dat er enkel klein objecten werden vervaardigd (vb. nagels of simpele pijlpunten). Meer complexe smeedactiviteiten (vb. produktie van de zwaarden) zoals het welden van ijzer en het bewerken van staal hebben dus niet plaatsgevonden.

8. Conclusies

Het ruime spectrum van geanalyseerde materialen heeft een aantal technologische processen aan het licht gebracht die nieuw zijn voor ZO-Arabië. De projectie van de lood isotopen data lijken aanvullende bewijzen aan te brengen voor een dichte betrokkenheid van ed-Dur en de commerciële activiteiten van Characene.

REFERENCES

"County library? Reference desk, please. Hello? Yes, I need a word definition.
Well, that's the problem. I don't know how to spell it and I'm not allowed to say it.
Could you just rattle off all the swear words you know and I'll stop you when ... Hello?"

Calvin & Hobbes

AGRAWAL, O.P., NARAIN, H. & PRAKASH, J. (**1991**)
Metallographic Studies of Iron Artifacts of Domestic Use, Tools and Weapons from Khairadih (100-400 AD), Uttar Pradesh. IN: *Role of Chemistry in Archaeology*, (Ganorkar, M.C. & Rama Rao, N., eds): 103-111. Hyderabad: Birla Institute of Sientific Research.

ALLABY, A. & ALLABY, M. (ED.) (**1999**)
A Dictionary of Earth Sciences. Oxford - New York: Oxford University Press.

AL-MUGHANNUM, A.S. & WARWICK, J. (**1986**)
Excavations of the Dhahran Burial Mounds Third Season, 1405/1985-86. *Atlal. The Journal of Saudi Arabian Archaeology* **10**: 9-27.

AL-NABOODAH, H.M. (**1992**)
The Commercial Activity of Bahrain and Oman in the Early Middle Age. *Proceedings of the Seminar for Arabian Studies* **22**: 81-96.

AL-QAISY, R. (**1975**)
Archaeological Investigations and Excavations at the State of the United Arab Emirates - Arabian Gulf. *Sumer* **31**: 75-157.

AL-SAA'D, Z. (**2000**)
Technology and provenance of a collection of Islamic copper-based objects as found by chemical and lead isotope analysis. *Archaeometry* **42** (2): 385-398.

AL-SALIHI, W. (**1989**)
Stele of Brymryn from Hatra. *Mesopotamia* **24**: 176-180.

AL-SAUD, A.S. (**1997**)
Central Arabia during the early Hellenistic period. Riyadh: King Fahd National Library.

AMY, R. & SEYRIG, H. (**1936**)
Recherches dans la nécropole de Palmyre. *Syria* **17**: 230-245.

AN. (**1972**)
Copper and copper alloys. Welding - soldering - brazing - surfacing. American Welding Society.

AN. (**1979**)
Source Book on Copper and Copper Alloys. Ohio: American Society for Metals.

AN. (**1984-1985**)
A mineral survey of the United Arab Emirates. IN: *Petroleum Industry in the UAE - 1984-1985 Yearboor*: 147-155. Abu Dhabi: A.R.I.P.

AN. (**2001**)
Archaeometallurgy. Centre for Archaeological Guidelines **1**. English Heritage.
http://www.english-heritage.org.uk/Filestore/archaeology/pdf/cfa_archaeometallurgy.pdf.

ANANTHARAMU, T.R., CRADDOCK, P.T., NAGESH RAO, K, MURTHY, S.R.N. & WAYMAN M.L. (**1999**)
Crucible steel of Ghattihossahilli, Chitradurga District, Karnataka, southern India. *Historical Metallurgy* **33** (1): 13-25.

ANDERSEN, S.F. (**2005**)
The Tylos period burials on Bahrain. Grave goods and chronology. Aarhus: Unpublished Ph.D. Thesis - Afteling for Klassisk Arkaeologi ved Institute for Antropologi, Arkaeologi og Lingvistik, Aarhus Universitet.

ANGELINI, E., DE CARO, T., GRASSINI, S., INGO, G.M. & ROSALBINO, F. (**2003**)
Microstructural evolution of Cu-based alloys in Roman mirrors. IN: *Archaeometallurgy in Europe. 24-25-26 September 2003 - Milan, Italy. Proceedings Vol. 2.*: 545-552. Milan: Associazione Italiana di Metallurgia.

ANHEUSER, K. (**1997**a)
The Practice and Characterization of Historic Fire Gilding Techniques. *Journal of Metals* **49** (11): 58-62.

ANHEUSER, K. (**1997**b)
Where is all the amalgam silvering? IN: *Materials Issues in Art and Archaeology V*, (Vandiver, P.B., Druzik, J.R., Merkel, J.F. & Stewart, J.): 127-134. Materials Research Society Symposium Proceedings **462**. Pittsburgh - Pennsylvania: Materials Research Society.

ANHEUSER, K. & FRANCE, P. (**2002**)
Silver plating technology of the late 3rd century Roman coinage. *Historical Metallurgy* **36** (1): 17-23.

ANHEUSER, K. & NORTHOVER, J.P. (**1995**)
Silver plating on Roman and Celtic coins from Britain - a technical study. *The British Numismatic Journal* **64**: 22-32.

ATTANASIO, D., BULTRINI, G. & INGO, G.M. (**2001**)
The Possibility of Provenancing a series of Bronze Punic Coins at Tharros (Western Sardinia) using the Literature Lead Isotope Database. *Archaeometry* **43** (4): 529-547.

AVANZINI, A. (ED.) (**2002**)
Khor Rori Report 1. Arabia Antica **1**. Pisa: Universita di Pisa.

AVERY, D.H., VAN DER MERWE, N.J. & SAITOWITZ, S. (**1988**)
The Metallurgy of the Iron Bloomery in Africa. IN: *The Beginning of the Use of Metals and Alloys*, (Maddin, R., ed.): 261-282. Cambridge - Massachusetts - London: M.I.T. Press.

AVNER, S.H. (**1974**)
Introduction to Physical Metallurgy. Tokyo: McGraw-Hill Kogakusha, Ltd.

BACHMANN, H-.G. (**1982**)
The Identification of Slags from Archaeological Sites. Institute of Archaeology. Occasional Publication **6**. London: Institute of Archaeology.

BALL, W. (**2000**)
Rome in the East: The Transformation of an Empire. London - New York: Routledge.

BARNES, I.L., SHIELDS, W.R. & MURPHY, T.J. (**1974**)
Isotopic Analysis of Laurion Lead Ores. IN: *Archaeological Chemistry*, (Beck, C.W., ed.): 1-10. Advances in Chemistry Series **138**. Washington D.C.: American Chemical Society.

BAYLEY, J. (**1992**)
Metalworking ceramics. *Medieval Ceramics* **16**: 3-10.

BAYLEY, J. (**1998**)
The production of brass in antiquity with particular reference to Roman Britain. IN: *2000 Years of Zinc and Brass*, (Craddock, P.T., ed.): 7-26. British Museum Occasional Paper **50**. London: British Museum Press.

BAYLEY, J. & ECKSTEIN, K. (**1997**)
Silver Refining - Production, Recycling and Assaying. IN: *Archaeological Sciences 1995. Proceedings of a conference on the application of scientific techniques to the study of archaeology*, (Sinclair, A., Salter, E. & Gowlett, J., eds): 107-111. Oxbow Monographs **64**. Oxford: Oxbow books.

BAYLEY, J. & ECKSTEIN, K. (**2006**)
Roman and Medieval Litharge Cakes: Structure and Composition. IN: *34th International Symposium on Archaeometry, 3 - 7 May 2004, Zaragoza, Spain*: 145-153. Zaragoza: Sociedad Cooperative, Librer.

BEAGRIE, N. (**1989**)
The Romano-British Pewter Industry. *Britannia* **20**: 169-191.

BECK, L., BOSONNET, S., REVEILLON, S., ELIOT, D. & PILON, F. (**2004**)
Silver surface enrichment of silver-copper alloys: a limitation for the analysis of ancient silver coins by surface techniques. *Nuclear Instruments and Methods in Physics Research B* **226**: 153-162 .

BECK, L., REVEILLON, S., BOSONNET, S., ELIOT, D. & PILON, F. (**2003**)
Experimental evidence of direct silver surface enrichment on silver-copper alloys. Application to the analysis of ancient silver coins. IN: *Archaeometallurgy in Europe. 24-25-26 September 2003 - Milan, Italy. Proceedings Vol. 2*: 561-568. Milan: Associazione Italiana di Metallurgia.

BEGEMANN, F., SCHMITT-STRECKER, S. & PERNICKA, E. (**1989**)
Isotopic Composition of Lead in Early Metal Artefacts. IN: *Archäometallurgie der Alten Welt / Old World Archaeometallurgy. Proceedings Symposium Heidelberg 1987*, (Hauptmann, A., Pernicka, E. & Wagner, G.A., eds): 269-278. Der Anschnitt, Beiheft **7**. Bochum: Deutsches Bergbau-Museum.

BENOIST, A., MOKADDEM, K. & MOUTON, M. (**1994**)
Excavations at Mleiha site, the 1993 and 1994 seasons. IN: *Archaeological Surveys and Excavations in the Sharjah Emirate, 1993 and 1994. A Seventh Interim Report*, (Mouton, M., ed.): 11-19. Lyon - Sharjah: CRNS - Directore of Antiquities and Heritage, Department of Culture.

BENOIST, A., MOUTON, M. & SCHIETTECATTE, J. (**2003**)
The artefacts from the fort at Mleiha: distribution, origins, trade and dating. *Proceedings of the Seminar for Arabian Studies* **33**: 59-76.

BERTHOUD, T. (**1979**)
Etude par l'analyse de traces et la modélisation de la filiation entre minerai de cuivre et objets archéologiques du Moyen-Orient (IVe et IIIe millénaires avant notre ère). Paris: Unpublished Ph.D. Thesis - University of Paris VI.

BIBBY, G. (**1954**)
Five among Bahrain's Hundered Thousand Grave-mounds. *KUML*: 132-141.

BIBBY, G. (**1972**)
Looking for Dilmun. Harmondsworth: Penguin Books Ltd.

BIN SERAY, H.M. (**1996**)
Spasinou Charax and its commercial relations with the east through the Arabian Gulf. *Aram* **8** (1 & 2): 15-23.

BISWAS, A.K. (**1996**)
Minerals and Metals in Ancient India. Vol.1 Archaeological Evidence. New Delhi: D.K. Printworld (P) Ldt.

BISWAS, A.K. (**2001**)
Minerals and Metals in Pre-Modern India. New Delhi: D.K. Printworld (P) Ltd.

BIVAR, A.D.H. (**1983**)
The Political History of Iran under the Arsacids. IN: *The Cambridge History of Iran, Volume 3 (1). The Seleucid, Parthian and Sasanian Periods*, (Yarshater, E., ed.): 21-99. Cambridge: Cambridge University Press.

BOEHMER, R.M. & VON GALL, H. (**1973**)
Das Felsrelief bei Batas-Herir. *Baghdader Mitteilungen* **6**: 65-77.

BOUCHARLAT, R. (**1984**)
Les périodes pré-islamiques récentes aux Émirates Arabes Unis. IN: *Arabie Orientale, Mésopotamie et Iran Méridional de l'âge du fer au début de la période Islamique*, (Boucharlat, R. & Salles, J.-F.,eds): 189-199. Mémoire **37**. Paris: Editions de recherche sur les civilisations.

BOUCHARLAT, R. (**1989**)
Documents Arabes Provenant des Sites "Hellenistiques" de la Péninsula d'Oman. IN: *L'Arabie préislamique et son environnement historique et culturel. Actes du Colloque de Strasbourg 24-27 juin 1987*, (Fahd, T., ed.): 109-126. Strasbourg: Publications de la Faculté des Lettres, Univ. De Strasbourg.

BOUCHARLAT, R. (**1991**)
From the Iron Age to the "Hellenistic" Period: Some Evidence from Mleiha (United Arab Emirates). IN: *Golf-Archaologie: Mesopotamien, Iran, Kuwait, Bahrain, Vereinigte Arabische Emirate und Oman*, (Schippmann, K., Herling, A. & Salles J.-F., eds): 289-300. Internationale Archaologie **6**. Rahden: Verlag Marie Leidorf GmbH.

BOUCHARLAT, R. (**1997**)
Second Archaeological Survey in the Sharjah Emirate. A preliminary Report - II.5 Observations. IN: *Archaeological Surveys in Sharjah Emirate (U.A.E.), Second Report (1985)*, (Boucharlat, R., ed.): 26-39. Sharjah Archaeology **2**. Sharjah: Directorate of Archaeology Department of Culture & Information.

BOUCHARLAT, R. & DRIEUX, M. (**1991**)
Appendix 1. A Note on Coins and the Coin Mold from Mleiha, Emirate of Sharjah, U.A.E. IN: *The Pre-Islamic Coinage of Eastern Arabia*, (Potts, D.T.): 110-117. Carsten Niebuhr Institute Publications **14**. Copenhagen: Museum Tusculanum Press.

BOUCHARLAT, R. & GARCYZYNSKI, P. (**1997**)
The Mleiha Area - An Archaeological Map. IN: *Archaeological Surveys in Sharjah Emirate (U.A.E.), Fourth Report (1988)*, (Boucharlat, R., ed.): 47-72. Sharjah Archaeology **4**. Sharjah: Directorate of Archaeology Department of Culture & Information.

BOUCHARLAT, R. & MOUTON, M. (**1991**)
Cultural change in the Oman Peninsula during the late 1st millennium BC as seen from Mleiha, Sharjah Emirate (U.A.E.). *Proceedings of the Seminar for Arabian Studies* **21**: 23-33.

BOUCHARLAT, R. & MOUTON, M. (**1993**a)
Importations occidentales et influence de l'hellénisme dans la péninsule d'Oman. IN: *Arabia Antiqua: Hellenistic Centres around Arabia*, (Invernizzi, A. & Salles, J.-F., eds). Serie Orientale Roma **70** (2). Rome.

BOUCHARLAT, R. & MOUTON, M. (**1993**b)
Mleiha (3e s. avant J.-C.-1er/3e s. après J.-C.). IN: *Materialien zur Archäolgie der Seleukiden- und Partherzeit im südlichen Babylonien und im Golfgebiet*, (Finkbeiner, U., ed.): 219-250. Tübingen: Deutsches Archäologisches Institut, Abteilung Baghdad.

BOUCHARLAT, R. & MOUTON, M. (**1994**)
Mleiha (Emirate of Sharjah, UAE) at the beginning of the Christian era. *Proceedings of the Seminar for Arabian Studies* **24**: 13-26.

BOUCHARLAT, R. & MOUTON, M. (**1997**)
Excavations at Mleiha Site: A preliminary report. IN: *Archaeological Surveys in Sharjah Emirate (U.A.E.), Third Report (1986)*, (Boucharlat, R., ed.): 35-46. Sharjah Archaeology **3**. Sharjah: Directorate of Archaeology Department of Culture & Information.

BOUCHARLAT, R. & MOUTON, M. (**1998**)
Les pratiques funéraires dans la péninsule de Mleiha (E.A.U.). IN: *Arabia and its Neighbours. Essays on prehistorical and historical developments presented in honour of Beatrice de Cardi*, (Phillips, C.S., Potts, D.T. & Searight, S., eds): 15-32. Abiel, New Research on the Arabian Peninsula **2**. Brussels: Brepols.

BOUCHARLAT, R. & SALLES, J.-F. (**1981**)
The history and archaeology of the Gulf from the fifth century BC to the seventh century A.D.: A review of the evidence. *Proceedings of the Seminar for Arabian Studies* **11**: 67-94.

BOUCHARLAT, R. & SALLES, J.-F. (**1984**)
Céramiques de surface à ed-Dour, Emirats Arabes Unis. IN: *Arabie Orientale, Mésopotamie et Iran Méridional de l'âge du fer au début de la période Islamique*, (Boucharlat, R. & Salles, J.-F., eds): 241-270. Mémoire **37**. Paris: Editions de recherche sur les civilisations.

BOUCHARLAT, R. & SALLES, J.-F. (**1989**)
The Tylos Period (300 BC - 600 AD). IN: *Bahrain National Museum Archaeological Collections Volume I. A Selection of Pre-Islamic Antiquities from Excavations 1959-1975*, (Lombard, P. & Kervran, M., eds): 83-134. State of Bahrain: Museum and Heritage, Ministry of Information.

BOUCHARLAT, R., HAERINCK, E., LECOMTE, O., POTTS, D.T. & STEVENS, K.G. (**1989**)
The European Archaeological Expedition to ed-Dur, Umm Al-Qaiwayn (U.A.E.). An Interim Report on the 1987 and 1988 Seasons. *Mesopotamia* **24**: 5-72.

BOUCHARLAT, R., HAERINCK, E., PHILLIPS, C.S. & POTTS, D.T. (**1988**)
Archaeological Reconnaissance at ed-Dur, Umm al-Qaiwain U.A.E. *Akkadica* **58** (2): 1-26.

BRIAND, B., DALONGEVILLE, R. & PLOQUIN, A. (**1997**)
The Industries of Mleiha Mineralogical and Archaeological Date Recovered from the site and its Environment in 1990. IN: *Archaeological Surveys in Sharjah Emirate (U.A.E.), Sixth Report (1990-92)*, (Boucharlat, R., ed.): 49-52. Sharjah Archaeology **6**. Sharjah: Directorate of Archaeology Department of Culture & Information.

BRILL, R.H. & WAMPLER, J.M. (**1967**)
Isotope Studies on Ancient Lead. *American Journal of Archaeology* **71** (1): 63-77.

BRONSON, B. (**1986**)
The Making and Selling of Wootz, a Crucible Steel of India. *Archeomaterials* **1** (1): 13-51.

BROWNSWORD, R. & PITT, E.E.H. (**1983**)
A note on some medieval pewter spoon alloys. *Historical Metallurgy* **17** (2): 119.

BRZEZINSKI, R. & MIELCZAREK, M. (**2005**)
The Sarmatians, 600 BC - AD 450. Men-at-Arms **373.**

BUDD, P., POLLARD, A.M., SCAIFE, B. & THOMAS, R.G. (**1995**)
The possible fractionation of lead isotopes in ancient metallurgical processes. *Archaeometry* **37** (1): 143-150.

BUTCHER, K. & PONTING, M. (**1995**)
Rome and the East. Production of Roman provincial silver coinage for Caesarea in Cappadocia under Vespasian, AD 69-79. *Oxford Journal of Archaeology* **14** (1): 63-77.

BUTCHER, K. & PONTING, M. (**2005**)
The Roman denarius under the Julio-Claudian Emperors: mints, metallurgy and technology. *Oxford Journal of Archaeology* **24** (2): 163-197.

CALLOT, O. (**1990**)
Les monnaies dites 'arabes' dans le Nord du Golfe arabo-persique à la fin du IIIe siècle avant notre ère. IN: *Failaka: Fouilles Françaises 1986-1988*, (Calvet, Y. & Gachet, J., eds): 221-240. TMO (Travaux de la Maison de l'Orient) **18**. Lyon: Maison de l'Orient Méditerrenéen.

CALLOT, O. (**1994**a)
Mleiha: Local issues of the Arabian pre-islamic coinage. IN: *Archaeological Surveys and Excavations in the Sharjah Emirate, 1993 and 1994. A Seventh Interim Report*, (Mouton, M., ed.): 21-22. Lyon - Sharjah: CRNS - Directore of Antiquities and Heritage, Department of Culture.

CALLOT, O. (**1994**b)
Un trésor de monnaies d'argent et monnaies diverses. IN: *Qala'at al-Bahrain, volume 1. The Northern City Wall and the Islamic fortress*, (Højlund, F. & Andersen, H.H., eds): 351-358. Jutland Archaeological Society Publications **30**. Moesgård: Aarhus.

CALLOT, O. (**2004**)
Catalogue des Monnaies du Musée de Sharjah (Emirates Arabes Unis). Essai sur les monnayages arabes préislamiques de la péninsule d'Oman. Collection de la Maison de l'Orient et de la Méditerranée **30** [Série Archéologique 15]. Lyon: Maison de l'Orient et de la Méditerranée.

CAMPBELL, W. (**1933**)
Greek and Roman plated coins. Numismatic notes and monographs **57**. New York: American Numismatic Society.

CARRADICE, I. & COWELL, M.R. (**1987**)
The minting of Roman Imperial bronze coins for circulation in the East: Vespasian to Trajan. *Numismatic Chronicle* **147**: 26-50.

CARTER, R. (**2003**)
Restructuring Bronze Age Trade: Bahrain, southeast Arabia and the Copper Question. IN: *The Archaeology of Bahrain. The British contribution*, (Crawford, H., ed.): 31-42. BAR International Series **1189**. Oxford: Archaeopress.

CASSON, L. (**1989**a)
South Arabia's maritime trade in the first century A.D. IN: *L'Arabie préislamique et son environnement historique et culturel. Actes du Colloque de Strasbourg 24-27 juin 1987*, (Fahd, T., ed.): 187-194. Strasbourg: Publications de la Faculté des Lettres, Univ. De Strasbourg.

CASSON, L. (**1989**b)
The Periplus Maris Erythraei. Princeton: Princeton University Press.

CASSON, L. (**1991**)
Ancient Naval Technology and the Route to India. IN: *Rome and India. The Ancient Sea Trade*, (Begley, V. & De Puma, R.D., eds): 8-11. Madison: The University of Wisconsin Press.

CHAKRABARTI, D.K. (**1976**)
The Beginning of Iron in India. *Antiquity* **50** (198): 114-124.

CHAKRABARTI, D.K. (**1992**)
The Early Use of Iron in India. Delhi: Oxford University Press.

CHAKRABORTI, H. (**1966**)
Trade and Commerce of Ancient India (c. 200 B.C. - c. A.D. 650). Calcutta: Academic Publishers.

CHAMI, F. (**1994**)
The Tanzanian Coast in the First Millennium AD. An archaeology of the iron-working, farming communities. Studies in African Archaeology **7**. Uppsala: Societas Archaeologica Upsaliensis.

CHARLES, J.A. (**1996**)
Determinative mineralogy and the origins of metallurgy. IN: *Furnaces and Smelting Technology in Antiquity*, (Craddock, P.T. & Hughes, M.J., eds): 21-28. British Museum Occasional Paper **48**. London: British Museum Press.

CLEERE, H. (**1995**)
Reflections on the iron trade in the Roman world. IN: *Paléométallurgie du fer et Cultures. Actes du Symposium international du Comité pour la sidérurgie ancienne de l'Union internationale des sciences préhistoriques et protohistoriques, Belfort - Sévenans 1-2-3 novembre 1990*, (Benoit, P. & Fluzin, P., eds): 213-216. Belfort: Institut Polytechnique de Sévenans.

COGHLAN, H.H. (**1975**)
Notes on the Prehistoric Metallurgy of Copper and Bronze in the Old World, 2nd edition. Occasional Paper on Technology **4**. Oxford: Oxford University Press.

COGHLAN, H.H. (**1977**)
Notes on Prehistoric and Early Iron in the Old World, 2nd edition. Occasional Paper on Technology **8**. Oxford: Pitt Rivers Museum.

COLLEDGE, M.A.R. (**1976**)
The Art of Palmyra. London: Thames and Hudson.

CONDAMIN, J. & PICON, M. (**1964**)
The Influence of Corrosion and Diffusion on the Percentage of Silver in Roman denarii. *Archaeometry* **7**: 98-105.

CONGDON, L.O.K. (**1971**)
Steel in Antiquity: a Problem of Terminology. IN: *Studies Presented to George M.A. Hanfmann*, (Mitten, D.G., Pedley, J.G. & Scott, J.A.., eds): 7-27. Fogg Art Museum - Harvard University Monographs in Art and Aarchaeology **2**. Mainz: Verlag Philipp von Zabern.

COPE, L.H. (**1972**a)
Surface-Silvered Ancient Coins. IN: *Methods of Chemical and Metallurgical Investigation of Ancient Coinage*, (Hall, E.T. & Metcalf, D.M., eds): 261-278. Royal Numismatic Society, Special Publication **8**. London: Royal Numismatic Society.

COPE, L.H. (**1973**)
The Metallurgical Examination of a Debased Silver Coin of Maximinus Daza Issued by Constantine I. *Archaeometry* **15** (2): 221-228.

CORBOUD, P., CASTELLA, A.-C., HAPKA, R. & IM OBERSTEG, P. (**1996**)
Les Tombes Protohistoriques de Bithnah, Fujairah, Emirats Arabes Unis. Terra Archaeologica **1**. Mainz: Phillip von Zabern.

COSTA, P.M., GRAZIOSI COSTA, G., YULE, P., KUNTER, M., PHILLIPS, C., & 'ALI B. A.B.B. AL SHANFARI (**1999**)
Archaeological Research in the Area of Muscat. IN: *Studies in the Archaeology of the Sultanate of Oman*, (Yule, P., ed.): 1-90. Orient-Archäologie **2**. Rahden: Verlag Marie Leidorf.

COSTELLO, S.D. (**2005**)
An Investigation of Early Chinese Bronze Mirrors at the Harvard University Art Museums. *ANAGPIC 2005 Student Papers*: 1-15
www.buffalostate.edu/depts/artconservation/Revised%20Website%202005/ANAGPIC%20Page/Costello%20ANAGPIC.pdf.

COTTRELL, A.J. (ED.) (**1980**)
The Persian Gulf States. A general Survey. Baltimore - London: The Johns Hopkins University Press.

COWELL, M.R., CRADDOCK, P.T., PIKE, A.W.G. & BURNETT, A.M. (**2000**)
An analytical survey of Roman provincial copper-alloy coins and the continuity of brass manufacture in Asia Minor. IN: *XII. Internationaler Numismatischer Kongress Berlin 1997. Akten - Proceedings - Actes I*, (Kluge, B. & Weisser, B., eds): 670-677.

CRADDOCK, P.T. (**1977**)
The Composition of the Copper Alloys used by the Greek, Etruscan and Roman Civilizations. 2. The Archaic, Classical and Hellenistic Greeks. *Journal of Archaeological Science* **4**: 103-123.

CRADDOCK, P.T. (**1978**)
The Composition of the Copper Alloys used by the Greek, Etruscan and Roman Civilizations. 3. The Origins and Early Use of Brass. *Journal of Archaeological Science* **5**: 1-16.

CRADDOCK, P.T. (**1979**)
The Copper Alloys of the Medieval Islamic World. *World Archaeology* **11** (1): 68-79.

CRADDOCK, P.T. (**1985**)
Technical appendix 1. The composition of the metal artefacts. IN: *Prehistoric Tombs of Ras al-Khaimah*, (Donaldson, P., ed.). Oriens Antiquus **24**.

CRADDOCK, P.T. (**1989**)
The scientific investigation of early mining and smelting. IN: *Scientific Analysis in Archaeology and its Interpretation*, (Henderson, J., ed.): 178-212. Oxford University Committee for Archaeology Monograph **19**. Oxford: Oxford University Committee for Archaeology.

CRADDOCK, P.T. (**1995**)
Early Metal Mining and Production. Edinburgh: Edinburgh University Press.

CRADDOCK, P.T. (**1998**a)
New Light on the Production of Crucible Steel in Asia. *Bulletin of the Metals Museum* **29**: 41-66.

CRADDOCK, P.T. (**1998**b)
Zinc in Classical Antiquity. IN: *2000 Years of Zinc and Brass*, (Craddock, P.T., ed.): 1-6. British Museum Occasional Paper **50**. London: British Museum Press.

CRADDOCK, P.T. (**2003**)
Cast Iron, Fined Iron, Crucible Steel: Liquid Iron in the Ancient World. IN: *Mining and Metal Production through the Ages*, (Craddock, P.T. & Lang, J., eds): 231-257. London: British Museum Press.

CRADDOCK, P.T. & ECKSTEIN, K. (**2003**)
Production of Brass in Antiquity by Direct Reduction. IN: *Mining and Metal Production through the Ages*, (Craddock, P.T. & Lang, J., eds): 216-230. London: British Museum Press.

CRADDOCK, P.T. & GALE, D. (**1988**)
Evidence for early mining and extractive metallurgy in the British Isles: problems and potentials. IN: *Science and Archaeology, Glasgow, 1987. Proceedings of a conference on the application of Scientific techniques to archaeology, Glasgow, September 1987*, (Slater, E.A. & Tate, J.O., eds): 167-191. BAR British Series **196** (i). Oxford: Archaeopress.

CRADDOCK, P.T. & GURJAR, L.K. (**1991**)
Traditional Zinc Distillation in India and China. IN: *Role of Chemistry in Archaeology*, (Ganorkar, M.C. & Rama Rao, N., eds): 112-120. Hyderabad: Birla Institute of Sientific Research.

CRADDOCK, P.T. & LANG, J. (**2004**)
Crucible Steel - Bright Steel. *Historical Metallurgy* **38** (1): 35-46.

CRADDOCK, P.T. & WEIRONG, Z. (**2003**)
Traditional Zinc Production in Modern China: Survival and Evolution. IN: *Mining and Metal Production through the Ages*, (Craddock, P.T. & Lang, J., eds): 266-292. London: British Museum Press.

CRADDOCK, P.T., BURNETT, A.M. & PRESTON, K. (**1980**)
Hellenistic copper-base coinage and the origins of brass. IN: *Scientific Studies in Numismatics*, (Oddy, W.A., ed.): 53-64. British Museum Occasional Paper **18**. London: British Museum Press.

CRADDOCK, P.T., FREESTONE, I.C., GURJAR, L.K., MIDDLETON, A. & WILLIES, L. (**1989**)
The production of lead, silver and zinc in ancient India. IN: *Archäometallurgie der Alten Welt / Old World Archaeometallurgy. Proceedings Symposium Heidelberg 1987*, (Hauptmann, A., Pernicka, E. & Wagner, G.A., eds): 51-70. Der Anschnitt, Beiheft **7**. Bochum: Deutsches Bergbau-Museum.

CRADDOCK, P.T., FREESTONE, I.C., GURJAR, L.K., MIDDLETON, A. & WILLIES, L. (**1998**)
Zinc in India. IN: *2000 Years of Zinc and Brass*, (Craddock, P.T., ed.): 27-72. British Museum Occasional Paper **50**. London: British Museum Press.

CRADDOCK, P.T., LA NIECE, S.C. & HOOK, D.R. (**1998**)
Brass in the Medieval Islamic World. IN: *2000 Years of Zinc and Brass*, (Craddock, P.T., ed.): 73-114. British Museum Occasional Paper **50**. London: British Museum Press.

CREW, P. (**1996**)
Archaeology Datasheet No. 6: Bloom refining and smithing slags and other risidues.
http://ourworld.compuserve.com/homepages/PeteHutch/start.htm.

CREW, P. & SALTER, C. (**1991**)
Comparative Data from Iron Smelting and Smithing Experiments. IN: *From Bloom to Knife. International Symposium of the Comité pour la sidérurgie ancienne de l'UISPP, Kielce - Ameliowka 18-22 September 1989*: 15-22. Materialy Archeologiczne **26**. Krakow: Muzeum Archeologiczne w Krakowie.

CURTIS, J. (**1989**)
Ancient Persia. London: British Museum.

CURTIS, V.S. (**2000**)
Parthian Culture and Costume. IN: *Mesopotamia and Iran in the Parthian and Sasanian Periods: Rejection and Revival c. 238 BC - AD 642*, (Curtis, J.E., ed.): 23-34. London: British Museum Press.

CURTIS, V.S. (**2001**)
Parthian Belts and Belt Plaques. *Iranica Antiqua* **36**: 299-327.

DAEMS, A. (**2004**a)
The terracotta figurines from ed-Dur (Umm al-Qaiwain, U.A.E.): The animal representations. *Arabian Archaeology and Epigraphy* **15**: 1-11.

DAEMS, A. (**2004**b)
The terracotta figurines from ed-Dur (Umm al-Qaiwain, U.A.E.): The human representations. *Arabian Archaeology and Epigraphy* **15**: 92-104 .

DAEMS, A. & HAERINCK, E. (**2001**)
Excacations at Shakhoura (Bahrain). *Arabian Archaeology and Epigraphy* **12**: 90-95 .

DAEMS, A., HAERINCK, E. & RUTTEN, K. (**2001**)
A burial mound at Shakhoura (Bahrain). *Arabian Archaeology and Epigraphy* **12**: 173-182 .

DAVEY, C.J. (**1979**)
Some Ancient Near Eastern Pot Bellows. *Levant* **11**: 101-111 .

DE CORTE, K. (**1994**)
Niet-destructief fysisch onderzoek van kralen uit de archeologische site ed-Dur (Umm al-Qaiwain, Verenigde Arabische Emiraten). Ghent: Unpublished licentiaatsthesis - Vakgroep Geologie en Bodemkunde, Universiteit Gent.

DE JESUS, S., AL-SURGIRAN, S., RIHANI, B., KESNAWI, A., TOPLYN, M. & INCAGNOLI, J. (**1982**)
Preliminary Report of the Ancient Mining Survey 1981 (1401). *Atlal. The Journal of Saudi Arabian Archaeology* **6**: 63-79 .

DE MUYNCK, D., CLOQUET, CH. & VANHAECKE, F. (**2007**)
Development of a new method for Pb isotopic analysis of archaeological artefacts using single-collector ICP-dynamic reaction cell-MS. *Journal of Analytical Atomic Spectrometry*: In press. .

DE PAEPE, P., RUTTEN, K., VRYDAGHS, L. & HAERINCK E. (**2002**)
A Petrographic, Chemical and Phytolith Analysis of Late Pre-Islamic Ceramics from ed-Dur (Umm al-Qaiwain, U.A.E.). IN: *Archaeology of the United Arab Emirates. Proceedings of the First International Conference on the Archaeology of the U.A.E.*, (Potts, D., Al Naboodah, H. & Hellyer, P., eds): 208-228. Galway: Trident Press.

DE RYCK, I., ADRIAENS, A. & ADAMS, F. (**2003**)
Microanalytical metal technology study of ancient near eastern bronzes from Tell Beydar. *Archaeometry* **45** (4): 579-590.

DE VALLERIN, G.G. (**1978**)
Le Periple de la Mer Erythree. *Dossiers de l'archéologie* **29**: 102-105.

DE WAELE, A. (**2005**)
Composite bows at ed-Dur (Umm al-Qaiwain, U.A.E.). *Arabian Archaeology and Epigraphy* **16** (2): 154-160.

DE WAELE, A. & HAERINCK, E. (**2006**)
Etched (carnelian) beads from northeast and southeast Arabia. *Arabian Archaeology and Epigraphy* **17**: 31-40.

DE WILDE, G. (**1994**)
Zuid-Oost arabische numismatiek. Van ca. De 3de eeuw v.Chr. tot ca. de 3de eeuw na Chr. Een typologische, iconografische en chronologische studie. Gent: Unpublished MA Thesis - Archeologie van het Oude Nabije Oosten, Universiteit Gent.

DEBLAUWE, F. (**1991**)
Old South Arabian Trade Routes. *Orientalia Lovaniensia Periodica* **22**: 133-158 .

DELRUE, P. (**2006**)
Ring-pommel daggers from ed-Dur (Umm al-Qaiwain, U.A.E.), an archaeometallurgical and typological analysis. *Arabian Archaeology and Epigraphy* **17**: 201-213 .

DELRUE, P. (**2007**a)
Flip the coin. Preliminary results of compositional EDX analyses on SE-Arabian coins from ed-Dur (Umm al-Qaiwain, U.A.E.). *Proceedings of the Seminar for Arabian Studies* **37**: 79-92.

DELRUE, P. (**2007**b)
Trilobate arrowheads at ed-Dur (U.A.E, Emirate of Umm al-Qaiwain). *Arabian Archaeology and Epigraphy* **18**: 239-250.

DERAISME, A., BECK, L., PILON, F. & BARRANDON, J.-N. (**2006**)
A study of the silvering process of the Gallo-Roman coins forged during the third century AD. *Archaeometry* **48** (3): 469-480.

DODGEON, M.H. & LIEU, S.N. (**1991**)
The Roman Eastern Frontier and the Persian Wars (AD 226-363). A Documentary History. London - New York: Routledge.

DOE, D.B. (**1983**)
Monuments of South Arabia. Cambridge: Falcon - Orleander Press.

DONALDSON, P. (**1984**)
Prehistoric Tombs of Ras al-Khaimah. *Oriens Antiquus* **23** (3-4): 191-312.

DONALDSON, P. (**1985**)
Prehistoric Tombs of Ras al-Khaimah. *Oriens Antiquus* **24** (1-2): 85-142.

DUNGWORTH, D.B. (**1997**a)
Caley's 'zinc decline' reconsidered. *Numismatic Chronicle* **156**: 228-234.

DUNGWORTH, D.B. (**1997**b)
Roman Copper Alloys: Analysis of Artifacts from Northern Britain. *Journal of Archaeological Science* **24**: 901-910 .

DUNIKOWSKI, C., LEROY, M., MERLUZZO, P. & PLOQUIN, A. (**1998**)
Des déchets paléométallurgiques: quels indices pour une forge? IN: *Les métaux antiques : travail et restauration. Actes du colloque Les de Poitiers, 28-30 septembre 1995*, (Nicolini, G. & Dieudonné-Glad, N., eds): 145-152. Monographies instrumentum **6**. Montagnac: Mergoil.

DURING CASPERS, E.C.L. (**1988**)
Contacts between India and the West during early historical times. *Indian Museum Bulletin* **21**: 22-33.

EGAMI, N., FUKAI, S. & MASUDA, S. (**1966**)
Dailaman II. The Excavations at Noruzmahali and Khoramrud 1960. The Tokyo University Iraq-Iran Archaeological Mission Report **7**. Tokyo: The Yamakawa Publishing Co., Ltd.

EHRENREICH, R.M. (**1985**)
Trade, Technology, and the Ironworking Community in the Iron Age of Southern Britain. BAR British Series **144**. Oxford: Archaeopress.

ELGOOD, R. (**1994**)
The Arms and Armour of Arabia in the 18th-19th and 20th Centuries. Hants: Scolar Press.

ERICSON, J.E. & SHIRAHATA, H. (**1985**)
Lead Isotope Analysis of Ancient Copper and Base Metal Ore Deposits in Western India. IN: *Application of Science in Examination of Works of Art. Proceedings of the Seminar: September 7-9, 1983*, (England, P.A. & van Zelst, L., eds): 207-212. Boston: Museum of Fine Arts.

ESCHENLOHR, L. & SERNEELS, V. (**1991**)
Les bas fourneaux mérovingiens de Boécourt, Les Boulies (JU/Suisse). Cahiers d'archéologie jurassienne **3**. Porrentruy: Office de patrimoine historique société jurassienne d'émulation.

FEUERBACH, A., MERKEL, J.F. & GRIFFITHS, D.R. (**1997**)
Production of Crucible Steel by Co-Fusion: Archaeometallurgical Evidence From the Ninth-Early Tenth Century at the Site of Merv, Turkmenistan. IN: *Materials Issues in Art and Archaeology V*, (Vandiver, P.B., Druzik, J.R., Merkel, J.F. & Stewart, J.): 105-110. Materials Research Society Symposium Proceedings **462**. Pittsburgh - Pennsylvania: Materials Research Society.

FEUERBACH, A.M., MERKEL, J.F. & GRIFFITHS, D.R. (**1998**)
An examination of crucible steel in the manufacture of Damascus steel, including evidence from Merv, Turkmenistan. IN: *Metallurgica antiqua: in honour of Hans-Gert Bachmann and Robert Maddin*, (Rehren, Th., Hauptmann, A. & Muhly, J., eds): 37-44. Der Anschnitt, Beiheft **8**. Bochum: Deutsches Bergbau-Museum.

FEUERBACH, A.M. (**2002**)
Crucible Steel in Central Asia: Production, Use, and Origins. London: Unpublished Ph.D. Thesis - Institute of Archaeology, University College London.

FEUERBACH, A.M., GRIFFITHS, D.R. & MERKEL, J.F. (**2003**)
Early Islamic Crucible Steel Production at Merv, Turkmenistan. IN: *Mining and Metal Production through the Ages*, (Craddock, P.T. & Lang, J., eds): 258-266. London: British Museum Press.

FEUGÈRE, M. (**2002**)
Les armes des Romains de la République à lAntiquité tardive. Paris: editions errance.

FLUZIN, PH., PLOQUIN, A. & DABOSI, F. (**2004**)
Chapitre 4: Approches métallurgiques et archéométriques. IN: *Le Fer*, (Mangin, M., ed.): 113-173. Paris: éditions Errance.

FORBES, R.J. (**1964**a)
Studies in Ancient Technology - Volume VIII. Leiden: E.J. Brill.

FORBES, R.J. (**1964**b)
Studies in Ancient Technology - Volume IX. Leiden: E.J. Brill.

FREESTONE, I.C. (**1985**)
Scanning electron microscopy and X-ray microanalysis. IN: *The Archaeologist and the Laboratory*, (Phillips, P., ed.): 67-68. CBA Research Report **58**. Oxford: Council for British Archaeology London.

FREESTONE, I.C., CRADDOCK, P.T., HEGDE, K.T.M., HUGHES, M.J. & PALIWAL, H.V. (**1996**)
Zinc production at Zawar, Rajasthan. IN: *Furnaces and Smelting Technology in Antiquity*, (Craddock, P.T. & Hughes, M.J., eds): 229-224. British Museum Occasional Paper **48**. London: British Museum Press.

FREESTONE, I.C., MIDDLETON, A.P., CRADDOCK, P.T., GURJAR, L.K. & HOOK, D.R. (**1991**)
Role of materials analysis in the reconstruction of early metal extraction technology: zinc and silver-lead smelting at Zawar, Rajasthan IN: *Materials Issues in Art and Archaeology II*, (Vandiver, P.B., Druzik, J. & Wheeler, G.S., eds): 617-626. Materials Research Society Symposium Proceedings **185**. Pittsburgh - Pennsylvania: Materials Research Society.

FRIEL, J.J. (**1995**)
X-ray and Image Analysis in Electron Microscopy. Princeton: Princeton Gamma-Tech, Inc.

FRIEND, J.N.A. & THORNEYCROFT, W. (**1929**)
The Silver Content of Ancient and Medieval Lead. *Journal of the Institute of Metals* **41**: 105-116.

FRIFELT, K. (**1975**)
On Prehistoric Settlements and Chronology of the Oman Peninsula. *East and West* **25**: 329-423.

FRIFELT, K. (**1990**)
A third millennium kiln from the Oman Peninsula. *Arabian Archaeology and Epigraphy* **1**: 4-15.

FRIFELT, K. (**2001**)
Islamic Remains in Bahrain. Jutland Archaeological Society Publications **37**. Moesgaard: Jutland Archaeological Society.

FRYE, R.N. (**1983**)
The Political History of Iran under the Sasanians. IN: *The Cambridge History of Iran, Volume 3 (1). The Seleucid, Parthian and Sasanian Periods*, (Yarshater, E., ed.): 116-180. Cambridge: Cambridge University Press.

FULFORD, M.G. & ALLEN, J.R.L. (**1992**)
Iron-making at the Chesters villa, Woolaston, Gloucestershire: Survey and Excavation 1987-91. *Britannia* **23**: 159-215.

GABRIEL, B.L. (**1985**)
SEM: A User's Manual for Materials Science. Metal Park, Ohio: American Society for Metals.

GALE, N.H & STOS-GALE, Z.A. (**2000**)
Lead isotope analyses applied to provenance studies. IN: *Modern Analytical Methods in Art and Archaeology*, (Ciliberto, E. & Spoto, G., eds): 503-584. Chemical Analyses Series **155**. New York: Wiley-Interscience.

GAWLIKOWSKI, M. (**1997**)
The Syrian Desert under the Romans. IN: *The Early Roman Empire in the East*: 37-54. Oxbow Monograph **95**. Oxbow: Oxbow Books.

GAZDAR, M.S., POTTS, D.T. & LIVINGSTONE, A. (**1984**)
Excavations at Thaj. *Atlal. The Journal of Saudi Arabian Archaeology* **8**: 55-85.

GILMOUR, B. (**2000**b)
The development of iron and steel technology in southern Asia. IN: *Persian Steel: The Tanavoli Collection*, (Allan, J. & Gilmour B.J.): 41-79. Oxford Studies in Islamic Art **15**. Oxford: University of Oxford Press.

GINTERS, W. (**1928**)
Das Schwert der Skythen und Sarmaten in Südrussland. Vorgeschichtliche Forschungen **2** (1). Berlin: Verlag von Walter de Gruyter & Co.

GLOVER, I.C. (**1996**)
The Archaeological Evidence for Early Trade between India and Southeast Asia. IN: *The Indian Ocean in Antiquity*, (Reade, J., ed.): 365-400. London - New York: Kegan Paul International.

GODFREY, E.G. (**2006**)
The Technology of Ancient and Medieval Directly Reduced Phosphoric Iron. Bradford: Unpublished Ph.D. Thesis - Department of Archaeological Sciences, University of Bradford.

GOETTLER, G.W., FIRTH, N. & HUSTON, C.C. (**1976**)
A Preliminary Discussion of Ancient Mining in the Sultanate of Oman. *Journal of Oman Studies* **2**: 43-51.

GOLDMAN, B. (**1993**)
The later pre-Islamic riding costume. *Iranica Antiqua* **28**: 201-239.

GOODMAN, M. (**1997**)
The Roman World, 44 BC-AD 180. London - New York: Routledge.

GRAVE, P., BIRD, R. & POTTS, D.T. (**1996**)
A Trail PIXE/PIGME analysis of Pre-Islamic Arabian Coinage. *Arabian Archaeology and Epigraphy* **7**: 75-81.

GREENOUGH, A.P. (**1987**)
An examination of Roman iron working residues from Loughor, West Glamorgan, Wales. *Historical Metallurgy* **21** (1): 25-27.

GRIFFITHS, D.R., FEUERBACH, A.M. & MERKEL, J.F. (**1997**)
Islamic Crucible Steel Manufacture in Merv. IN: *Archaeological Sciences 1995. Proceedings of a conference on the application of scientific techniques to the study of archaeology*, (Sinclair, A., Salter, E. & Gowlett, J., eds): 116-118. Oxbow Monographs **64**. Oxford: Oxbow books.

GROOM, N. (**1982**)
Gerrha - A 'Lost' Arabian City. *Atlal. The Journal of Saudi Arabian Archaeology* **6**: 97-108.

HABASHI, F. (ED.) (**1994**)
A history of metallurgy. Québec: Métallurgie Extractive Québec, Enr.

HAERINCK, E. (**1989-1990**)
Ed Dur (Umm al Qiwain, United Arab Emirates) (2nd season by the Belgian Team). *The Arabian Gulf Gazetteer* **1** (1): 15-16.

HAERINCK, E. (**1992**)
Excavations at ed-Dur (Umm al-Qaiwain, U.A.E.) - Preliminary report on the fourth Belgian season (1990). *Arabian Archaeology and Epigraphy* **3**: 190-208.

HAERINCK, E. (**1993**)
Excavations at ed-Dur (Umm al-Qaiwain, U.A.E.) - Preliminary report on the fifth Belgian Season (1991). *Arabian Archaeology and Epigraphy* **4**: 210-225.

HAERINCK, E. (**1994**a)
Excavations at ed-Dur (Umm al-Qaiwain, U.A.E.) - Preliminary report on the sixth Belgian Season (1992). *Arabian Archaeology and Epigraphy* **5**: 184-197.

HAERINCK, E. (**1994**b)
Héraclès dans l'iconographie des monnaies pré-islamiques d'Arabie du Sud-Est? *Akkadica* **89-90**: 9-13.

HAERINCK, E. (**1996**)
The seventh and eight Belgian archaeological expeditions to ed-Dur (Umm al-Qaiwain, U.A.E.). *Arabian Archaeology and Epigraphy* **7**: 69-74.

HAERINCK, E. (**1998**a)
International Contacts in the Southern Persian Gulf in the late 1st Century B.C./1ste Century A.D.: numismatic evidence from ed-Dur (Emirate of Umm al-Qaiwain, U.A.E.). *Iranica Antiqua* **33**: 273-302.

HAERINCK, E. (**1998**b)
More pre-Islamic coins from Southeastern Arabia. *Arabian Archaeology and Epigraphy* **9**: 278-301.

HAERINCK, E. (**1998**c)
The shifting pattern of overland and seaborne trade in SE-Arabia: foreign pre-islamic coins from Mleiha (Emirate of Sharjah, U.A.E.). *Akkadica* **106** (jan.-feb.): 22-40.

HAERINCK, E. (**1999**)
Abi'el, the ruler of Southeastern Arabia. *Arabian Archaeology and Epigraphy* **10**: 124-128.

HAERINCK, E. (**2001**)
Excavations at ed-Dur (Umm al-Qaiwain, United Arab Emirates), Vol. II: The Tombs. The University of Ghent South-East Arabian Archaeological Project. Leuven: Peeters.

HAERINCK, E. (**2002**a)
Boom and Burst at ed-Dur. *Minerva* **13** (4): 41-44.

HAERINCK, E. (**2002**b)
Internationalisation and Business in Southeast Arabia during the Late 1st century B.C./1st Century A.D.: Archaeological Evidence from ed-Dur (Umm al Qaiwain, U.A.E.). IN: *Archaeology of the United Arab Emirates. Proceedings of the First International Conference on the Archaeology of the U.A.E*, (Potts, D., Al Naboodah, H. & Hellyer, P., eds): 196-206. Galway: Trident Press.

HAERINCK, E. (**2002**c)
Textile remains from Eastern Arabia and new finds from Shakhoura (Bahrain) and ed-Dur (Umm al-Qaixain, U.A.E.). *Arabian Archaeology and Epigraphy* **13**: 246-254.

HAERINCK, E. (**2002**d)
The Indigenous Coinage of Pre-Islamitic South-East Arabia. *Minerva* **13** (2): 54-56.

HAERINCK, E. (**2003**)
Metal animal figurines from southeastern Arabia. *Arabian Archaeology and Epigraphy* **14**: 88-94.

HAERINCK, E. (**2004**)
Coins for Identity and Coins for Trade. Remarks on Late Pre-Islamic Numismatics in SE-Arabia (3rd. C. BCE - 3rd. C. AD). Unpublished text.

HAERINCK, E., METDEPENNINGHEN, C. & STEVENS, K.C. (**1991**)
Excavations at ed-Dur (Umm al-Qaiwain, U.A.E.) - Preliminary report on the second Belgian season (1988). *Arabian Archaeology and Epigraphy* **2**: 31-60.

HAERINCK, E., METDEPENNINGHEN, C. & STEVENS, K.C. (**1992**)
Excavations at ed-Dur (Umm al-Qaiwain, U.A.E.) - Preliminary report on the third Belgian season (1989). *Arabian Archaeology and Epigraphy* **3**: 44-60.

HAERINCK, E., PHILLIPS, C., POTTS, P.T. & STEVENS, K.G. (**1993**)
Ed-Dur, Umm al-Qaiwain (U.A.E.). IN: *Materialien zur Archäolgie der Seleukiden- und Partherzeit im südlichen Babylonien und im Golfgebiet*, (Finkbeiner, U., ed.): 183-193. Tübingen: Deutsches Archäologisches Institut, Abteilung Baghdad.

HAERINCK, E., VRYDAGHS, L. & DOUTRELEPONT, H. (**1998**)
Des feux sacrificiels pour la divinité solaire à ed-Dur. *Arabian Archaeology and Epigraphy* **9**: 125-130.

HARPER, P.O. (**1981**)
Silver Vessels of the Sasanian Period I. Royal Imagery. New York.

HASTINGS, A., HUMPHRIES, J.H. & MEADOW, R.H. (**1975**)
Oman in the third millennium BCE. *Journal of Oman Studies* **1**: 9-56.

HAUPTMANN, A. (**1985**)
5000 Jahre Kupfer in Oman. Band 1: Die Entwicklung der Kupfermetallurgie vom 3. Jahrtausend bis zur Neuzeit. Der Anschnitt, Beiheft **4**. Bochum: Deutsches Bergbau-Museum.

HAUPTMANN, A. (**1987**)
Kupfer und Bronzen der südostarabischen Halbinsel. *Der Anschnitt* **39**: 209-218.

HAUPTMANN, A. & WEISGERBER, G. (**1980**)
Third millenium BC copper production in Oman. *Revue D'Archeometrie* **3**: 131-138.

HAUPTMANN, A., WEISGERBER, G. & BACHMANN, H.-G. (**1988**)
Early Copper Metallurgy in Oman. IN: *The Beginning of the Use of Metals and Alloys*, (Maddin, R., ed.): 34-51. Cambridge - Massachusetts - London: M.I.T. Press.

HEALY, J.F. (**1978**)
Mining and Metallurgy in the Greek and Roman World. London: Thames and Hudson.

HELLYER, P. (**1998**)
Hidden Riches. An Archaeological Introduction to the United Arab Emirates. United Arab Emirates: Union National Bank United Arab Emirates.

HERLING, A. (**1994**)
Excavations at Karranah Mound I, Bahrain. A preliminary report. *Iranica Antiqua* **29**: 225-239.

HERLING, A. (**2000**)
Necropolis and Burial Customs in the Tylos Era. IN: *Traces of Paradise: The Archaeology of Bahrain 2500BC-300AD*, (Rice, M. & Crawford, H., ed.): 136-141. Bahrain: BahrainNational Museum.

HERLING, A. (**2003**)
Tyloszeitliche Bestattungspraktiken auf der Insel Bahrain. Präsentation und Interpretation der befunde der deutsche-bahrainischen Ausgrabungen bei Saar in Vergleich mit der Ergebnissen anderer Untersuchungen zum Thema. (Göttinger philosophische Dissertation) Göttinger: Cuvillier Verlag.

HESS, K., HAUPTMANN, A., WRIGHT, H. & WHALLON, R. (**1998**)
Evidence of fourth millennium BC silver production at Fatmali-Kalecik, East Anatolia. IN: *Metallurgica antiqua: in honour of Hans-Gert Bachmann and Robert Maddin*, (Rehren, Th., Hauptmann, A. & Muhly, J., eds): 57-67. Der Anschnitt, Beiheft **8**. Bochum: Deutsches Bergbau-Museum.

HILL, G.F. (**1922**)
Ancient Methods of Coining. *Numismatic Chronicle* **5** (2): 1-42.

HODGES, H. (**1968**)
Artifacts. An introduction to early materials and technology. London: John Baker.

HØJLUND, F. & ANDERSEN, H.H. (EDS) (**1994**a)
Qala'at al-Bahrain, volume 1. The Northern City Wall and the Islamic fortress. Jutland Archaeological Society Publications **30**. Moesgård: Aarhus.

HØJLUND, F. & ANDERSEN, H.H. (EDS) (**1997**)
Qala'at al-Bahrain, volume 2. The central Monumental Buildings. Jutland Archaeological Society Publications **30** (2). Moesgård: Aarhus.

HOMÈS-FREDERICQ, D. (**1963**)
Hatra et ses sculptures parthes étude stylistique et iconographique. Uitgaven van het Nederlands Historisch-Archeologisch Instituut te Istanbul **15** .

HOOK, D.R. & CRADDOCK, P.T. (**1996**)
Appendix: The scientific analysis of the copper alloy lamps: Aspects of classical alloying practices. IN: *A Catalogue of the Lamps in the British Museum IV: Lamps of Metal and Stone, and Lampstands*, (Bailey, D.M., ed.): 144-163. London: British Museum Press.

HOURANI, G.F. (**1963**)
Arab Seafaring in the Indian Ocean in ancient and early medieval times. Beirut: Khayats.

HOUSTON, G.W. (**1988**)
Ports in Perspective: Some Comparative Materials on Roman Merchant Ships and Ports. *American Journal of Archaeology* **92**: 553-564.

HOYLAND, R.G. (**2001**)
Arabia and the Arabs. From the Bronze Age to the coming of Islam. London - New York: Routledge.

HOYLAND, R.G. & GILMOUR, B. (**2006**)
Medieval Islamic Swords and Swordmaking. Kindi's treatise "On swords and their kinds". Oxford: Oxbow Books.

HUGHES, M.J. (**1980**)
The analysis of Roman tin and pewter ingots. IN: *Aspects of Early Metallurgy*, (Oddy, W.A., ed): 41-50. British Museum Occasional Paper **17**. London: British Museum Press.

INGO, G.M., AGUS, T., RUGGERI, AMORE BONAPASTA, A., R., BULTRINI, G. & CHIOZZINI, G. (**1997**)
Lead and silver production in the Montevecchio basin (western Sardinia). IN: *Materials Issues in Art and Archaeology V*, (Vandiver, P.B., Druzik, J.R., Merkel, J.F. & Stewart, J.): 411-416. Materials Research Society Symposium Proceedings **462**. Pittsburgh - Pennsylvania: Materials Research Society.

INGRAHAM, M.L., JOHNSON, T.D., RIHANI, B. & SHATLA, I. (**1981**)
Saudi Arabian Comprehensive Survey Program: Preliminary Report on a Reconnaissance Survey of the Northwestern Province. *Atlal. The Journal of Saudi Arabian Archaeology* **5**: 59-107.

JASIM, S.A. (**1999**)
The excavation of a camel cemetery at Mleiha, Sharjah, U.A.E. *Arabian Archaeology and Epigraphy* **10**: 69-101.

JASIM, S.A. (**2001**)
Excavations at Mleiha 1993-94. *Arabian Archaeology and Epigraphy* **12**: 103-135.

JASIM, S.A. (**2006**)
Trade centres and commercial routes in the Arabian Gulf: Post-Hellenistic discoveries at Dibba, Sharjah, United Arab Emirates. *Arabian Archaeology and Epigraphy* **17**: 214-237.

JENSEN, S.T. (**2003**)
Tylos burials from three different sites on Bahrain. *Arabian Archaeology and Epigraphy* **14**: 127-163.

JURISIC, M. (**2000**)
Ancient Shipwrecks of the Adriatic. Maritime transport during the first and second centeries AD. BAR International Series **828**. Oxford: Archaeopress.

KASSIANIDOU, V. (**2003**)
Early Extraction of Silver from Complex Polymetallic Ores. IN: *Mining and Metal Production through the Ages*, (Craddock, P.T. & Lang, J., eds): 198-206. London: British Museum Press.

KENNET, D. (**2005**)
On the eve of Islam: archaeological evidence from Eastern Arabia. *Antiquity* **79**: 107-118.

KERVRAN, M. (**1996**)
Indian Ceramics in Southern Iran and Eastern Arabia: Repertory, Classification and Chronology. IN: *Tradition and Archaeology, Early Maritime Contacts in the Indian Ocean. Proceedings of the International Seminar "Techno-Archaeological Perspectives of Seafaring in the Indian Ocean 4th cent. B.C. - 15 th cent. A.D." (New Dheli, Febr. 28 - March 4, 1994)*, (Ray, H.P. & Salles, J.-F., eds): 37-58. New Delhi: Manohar.

KHORASANI, M.M. (**2006**)
Arms and Armor from Iran The Bronze Age to the End of the Qajar Period. Tübingen: Legat-Verlag GmbH & Co.

KING, C.E. & NORTHOVER, J.P. (**1992**)
The Analyses. IN: *Der Münzhort aus dem Gutshof in Neftenbach. Antoniniane und Denare von Septimius Severus bis Postumus*, (von Kaenel, H.-M., ed.): 101-117. Zürcher Denkmalpflege Archäologische Monographien **16**. Zürich: Velag Zürich und Egg.

KING, G. & TONGHINI, C. (**1998**)
The Study of Islamic sites on the Abu Dhabi Islands. IN: *Arabia and its Neighbours. Essays on prehistorical and historical developments presented in honour of Beatrice de Cardi*, (Phillips, C.S., Potts, D.T. & Searight, S., eds). Abiel, New Research on the Arabian Peninsula **2**. Brussels: Brepols.

KISNAWI, A., DE JESUS, P.S. & RIHANI, B. (**1983**)
Preliminary report on the mining survey, Northwest Hijaz, 1982. *Atlal. The Journal of Saudi Arabian Archaeology* **7**: 76-83.

KNOX, R. (**1963**)
Detection of Iron Carbide Structure in the Oxide Remains of Ancient Steel. *Archaeometry* **6**: 43-45.

KOZLOFF, A.P. (ED.) (**1981**)
Animals in Ancient Art. From the Leo Mildenberg Collection. Cleveland: Cleveland Museum of Art.

KUPPURAM, G. (**1989**a)
Ancient Indian Mining, Metallurgy and Metal Industries, Volume I. Delhi: Sundeep Prakashan.

LA NIECE, S. (**1993**a)
Silvering. IN: *Metal Plating and Patination. Cultural, Technical and Historical Developments*, (La Niece, S. & Craddock, P.T., eds): 201-210. Oxford: Butterworth-Heinemann Ltd.

LA NIECE, S. (**1993**b)
Technology of Silver-Plated Coin Forgeries. IN: *Metallurgy in Numismatics, Volume 3*, (Archibald, M.M. & Cowell, M.R., eds): 227-239. Royal Numismatic Society, Special Publication **24**. London: Royal Numismatic Society.

LA NIECE, S. (**1998**)
Metallography in Numismatics. IN: *Metallurgy in Numismatics, Volume 4*, (Oddy, W.A. & Cowell, M., eds): 114-133. Royal Numismatic Society, Special Publication **30**. London: Royal Numismatic Society.

LAHIRI, N.D. (**1995**)
Indian metal and metal-related artefacts as cultural signifiers: an ethnographic perspective. *World Archaeology* **27**: 116-132.

LANCASTER, W. & LANCASTER, F. (**1996**)
Reflections on the Social Organization of the Arabian Bedu in Coastal Oman. IN: *The Prehistory of Asia and Oceania. Colloquium XXXII, Trade as a Subsistence Strategy. Post Pleistocene Adaptions in Arabia and Early Maritime Trade in the Indian Ocean*, (Afanas'ev, G., Cleuziou, S., Lukacs, J.R. & Tosi, M., eds): 141-154. A.B.A.C.O. Edizioni.

LANG, J. & HUGHES, M.J. (**1984**)
Soldering Roman Silver Pate. *Oxford Journal of Archaeology* **3** (3): 77-97.

LANG, J., CRADDOCK, P.T. & SIMPSON, ST. J. (**1998**)
New evidence for early crucible steel. *Historical Metallurgy* **32** (1): 7-14.

LECHTMAN, H. & KLEIN, S. (**1999**)
The Production of Copper Arsenic Alloys (Arsenic Bronze) by Cosmelting: Modern Experiment, Ancient Practice. *Journal of Archaeological Science* **26**: 497-526.

LECOMTE, O. (**1993**)
Ed-Dur, les occupations des 3e et 4e s. ap. J.-C.: contexte des trouvailles et matériel diagnostique. IN: *Materialien zur Archäolgie der Seleukiden- und Partherzeit im südlichen Babylonien und im Golfgebiet*, (Finkbeiner, U., ed.): 195-218. Tübingen: Deutsches Archäologisches Institut, Abteilung Baghdad.

LEROY, M. (**1997**)
La Sidérurgie en Lorraine avant le haut fourneau. L'utilisation du minerai de fer oolithique en réduction directe. Monographie du CRA **18**. Paris: Editions CNRS.

LITTLETON, J. (**2005**)
The Human Remains from the Belgian, French and Danish Excavations - ed-Dur, Umm al-Qawain. *Unpublished Preliminary Report for the Umm al-Qawain National Museum*: 1-12.

LOMBARD, P. (**1984**)
Quelques Eléments sur la Métallurgie de l'Age du Fer aux Emirats Arabes Unis. IN: *Arabie Orientale, Mésopotamie et Iran Méridional de l'âge du fer au début de la période Islamique*, (Boucharlat, R. & Salles, J.-F., eds): 225-236. Mémoire **37**. Paris: Editions de recherche sur les civilisations.

LOMBARD, P. (**1989**)
Age du Fer sans fer: Le cas de la Péninsule d'Oman au 1er millénaire avant. J.-C. IN: *L'Arabie préislamique et son environnement historique et culturel. Actes du Colloque de Strasbourg 24-27 juin 1987*, (Fahd, T., ed.): 25-37. Strasbourg: Publications de la Faculté des Lettres, Univ. De Strasbourg.

LOMBARD, P. & KERVRAN, M. (EDS) (**1989**)
Bahrain National Museum Archaeological Collections Volume I. A Selection of Pre-Islamic Antiquities from Excavations 1959-1975. State of Bahrain: Museum and Heritage, Ministry of Information.

LUKONIN, V.G. (**1983**)
Political, Social and Administrative Institutions: Taxes and Trade. IN: *The Cambridge History of Iran, Volume 3 (2). The Seleucid, Parthian and Sasanian Periods*, (Yarshater, E., ed.): 681-746. Cambridge: Cambridge University Press.

MABUCHI, H., HIRAO, Y. & NISHIDA, M. (**1985**)
Lead isotope approach to the understanding of early Japanese bronze culture. *Archaeometry* **27** (2): 131-159.

MAC DOWALL, D.W. (**1996**)
The Evidence of the Gazetteer of the Roman Artefacts in India. IN: *Tradition and Archaeology, Early Maritime Contacts in the Indian Ocean. Proceedings of the International Seminar "Techno-Archaeological Perspectives of Seafaring in the Indian Ocean 4th cent. B.C. - 15 th cent. A.D." (New Dheli, Febr. 28 - March 4, 1994)*, (Ray, H.P. & Salles, J.-F., eds): 79-95. New Delhi: Manohar.

MAGEE, P. (**1998**)
New Evidence of the Initial Appearance of Iron in Southeastern Arabia. *Arabian Archaeology and Epigraphy* **9**: 112-117.

MAGEE, P. (**2002**)
The Indigenous Context of Foreign Exchange between South-eastern Arabia and Iran in the Iron Age. *Journal of Oman Studies* **12**: 161-168.

MANGIN, M. (ED.) (**2004**)
Le Fer. Paris: éditions Errance.

MARAQTEN, M. (**1996**)
Notes on the Aramaic script of some coins from East Arabia. *Arabian Archaeology and Epigraphy* **7**: 304-315.

MARE, W.H. (**1995**)
Abila and Palmyra: Ancient Trade and Trade Routes from Southern Syria into Mesopotamia. *Aram* **7**: 189-215.

MARÉCHAL, J.-R. (**1982**)
Thermodynamique des scories gallo-romaines. IN: *Mines et Fonderies Antiques de la Gaule. Table ronde du CRNS, Université de Toulouse-Le Mirail, 21-22 novembre 1980*, (Domergue, C., ed.): 305-318. Paris: Editions CNRS.

MARTINEZ-SÈVE, L. (**2002**)
Les figurines de Susa. De l'époque néo-élamite à l'époque sassanide. Tome 2. Paris: Musée du Louvre Département des Antiquités orientales.

MASIA, K. (**2000**)
The Evolution of Swords and Daggers in the Sasanian Empire. *Iranica Antiqua* **35**: 185-244.

MATHIESEN, H.E. (**1992**)
Sculpture in the Parthian Empire. A Study in Chronology. Aarhus: Aarhus University Press.

MATTINGLY, G.L. (**1995**)
The Palmyrene Luxury Trade and Revelation 18: 12-13: A Neglected Analogue. *Aram* **7**: 217-231.

MCDONNELL, J.G. (**1984**)
The Study of Early Iron Smithing Residues. IN: *The Crafts of the Blacksmith. Essays presented to R.F. Tylecote at the 1984 Symposium of UISPP Comité pour la Sidérurgie Ancienne held in Belfast, N. Ireland 16th-2&st September 1984*, (Scott, B.G. & Cleere, H., eds): 47-59. Belfast: Ulster Museum/UISPP Comité pour la Sidérurgie Ancienne.

MCDONNELL, J.G. (**1988**)
Ore to artefact - a study of early ironworking technology. IN: *Science and Archaeology, Glasgow, 1987. Proceedings of a conference on the application of Scientific techniques to archaeology, Glasgow, September 1987*, (Slater, E.A. & Tate, J.O., eds): 283-293. BAR British Series **196** (i). Oxford: Archaeopress.

MCDONNELL, J.G. (**1991**)
A Model for the Formation of Smithing slags. IN: *From Bloom to Knife. International Symposium of the Comité pour la sidérurgie ancienne de l'UISPP, Kielce - Ameliowka 18-22 September 1989*: 23-26. Materialy Archeologiczne **26**. Krakow: Muzeum Archeologiczne w Krakowie.

MEEKS, N. (**1993**a)
Patination phenomena on Roman and Chinese high-tin bronze mirrors and other artefacts. IN: *Metal Plating and Patination. Cultural, Technical and Historical Developments*, (La Niece, S. & Craddock, P.T., eds): 63-84. Oxford: Butterworth-Heinemann Ltd.

MEEKS, N. (**1993**b)
Surface characterization of tinned bronze, high-tin bronze, tinned iron and arsenical bronze. IN: *Metal Plating and Patination. Cultural, Technical and Historical Developments*, (La Niece, S. & Craddock, P.T., eds): 247-275. Oxford: Butterworth-Heinemann Ltd.

MIHOK, L. & PRIBULOVA (**2003**)
Metallurgy of Iron at the Beginning of the 1st Millenium AD in Slovakia. IN: *Prehistoric and Medieval Direct Iron Smelting in Scandinavia and Europe. Aspects of Technology and Society. Proceedings of the Sandbjerg Confrence 16th to 20th September 1999*, (Norbach, L.C., ed.): 163-170. Acta Jutlandica [Humanities Series 75] **76** (2). Aarhus: Aarhus University Press.

MILLAR, F. (**1993**)
The Roman Near East 31 BC - AD 337. Cambridge - Massachusetts: Harvard University Press.

MILLET, M. (**1997**)
The Lithic Industry, 1988 report.IN: *Archaeological Surveys in Sharjah Emirate (U.A.E.), Fourth Report (1988)*, (Boucharlat, R., ed.): 33-43. Sharjah Archaeology **4**. Sharjah: Directorate of Archaeology Department of Culture & Information.

MITCHINER, M. (**2004**)
Ancient Trade and Early Coinage, Volume I.

MOMENZADEH, M. (**2004**)
Metallische Bodenschätze in Iran in antiker Zeit. Ein kurzer Überblick. IN: *Persiens Antike Pracht. Bergbau - Handwerk - Archäologie. Katalog der Ausstellung der Deutschen Bergbau-Museums Bochum von 28. November 2004 bis 29. Mai 2005. Band 2*, (Stöllner, T., Slotta, R. & Vatandoust, A., eds): 8-21. Bochum: Deutschen Bergbau-Museums.

MOOREY, P.R.S. (**1985**)
Materials and Manufacture in Ancient Mesopotamia: The evidence of Archaeology and Art. Metals and metalwork, glazed materials and glass. BAR International Series **237**. Oxford: Archaeopress.

MOOREY, P.R.S. (**1994**)
Ancient Mesopotamian Materials and Industries. The Archaeological Evidence. Oxford: Clarendon Press.

MORTON, G.R. & WINGROVE, J. (**1969**a)
Constitution of Bloomery Slags: Part I: Roman. *Journal of the Iron and Steel Institute* **207**: 1556-1564.

MOSHKOVA, M.G. (**1995**)
Middle Sarmatian Culture. IN: *Nomads of the Eurasian Steppes in the Early Iron Age*, (Davis-Kimball, J., Bashilov, V.A. & Yablonsky, L.T., eds): 137-147. Berkeley, CA: Zinat Press.

MOUTON, M. (**1990**)
Les pointes de flèches en fer des sites préislamiques de Mleiha et ed-Dur, E.A.U. *Arabian Archaeology and Epigraphy* **1**: 88-103.

MOUTON, M. (**1992**)
La peninsule d'Oman de la fin de l'age du fer au début de la periode sassanide (250 av. - 350 ap. JC). Volume I: Texte, Volume II: Planches. Paris: Unpublished Ph.D. Thesis - Near Eastern Archaeology, Université de Paris I.

MOUTON, M. (**1999**a)
Mleiha: Environment, survival strategies and crafts. IN: *Mleiha I. Environnement, stratégies de subsistance et artisanats*, (Mouton, M., ed.): 265-281. TMO (Travaux de la Maison de l'Orient) **29**. Lyon: Maison de l'Orient Méditerrenéen.

MOUTON, M. (**1999**b)
Mleiha: présentation du site et périodisation; Mleiha: Discription and Dating of the Site. IN: *Mleiha I. Environnement, stratégies de subsistance et artisanats*, (Mouton, M., ed.): 9-32. TMO (Travaux de la Maison de l'Orient) **29**. Lyon: Maison de l'Orient Méditerrenéen.

MOUTON, M. (ED.) (**1999**c)
Mleiha I. Environnement, stratégies de subsistance et artisanats, (Mouton, M., ed.) TMO (Travaux de la Maison de l'Orient) **29**. Lyon: Maison de l'Orient Méditerrenéen.

MOUTON, M., MOKADDEM, K. & GARCZYNSKI, P. (**1997**)
Excavations at Mleiha, 1990 and 1992 campaigns. IN: *Archaeological Surveys in Sharjah Emirate (U.A.E.), Sixth Report (1990-92)*, (Boucharlat, R., ed.): 24-48. Sharjah Archaeology **6**. Sharjah: Directorate of Archaeology Department of Culture & Information.

NICOLLE, D. (**1983**)
Arms Manufacture and the Arms Trade in South-Eastern Arabia in the Early Muslim Period. *Journal of Oman Studies* **6** (2): 231-238.

NODELMAN, S.A. (**1960**)
A Preliminary History of Characene. *Berytus* **13** (2): 83-121.

NORTHOVER, J.P. (**1997**)
Appendix: The Analysis of Early Copper and Copper Alloys. IN: *Art of Ancient Iron, Copper & Bronze*, (Mahboubian, H., ed.): 325-341. London: Philip Wilson Publishers Limited.

NORTHOVER, J.P. (**1998**a)
Analysis in the Electron Microprobe and Scanning Electron Microscope. IN: *Metallurgy in Numismatics, Volume 4*, (Oddy, W.A. & Cowell, M. eds): 94-113. Royal Numismatic Society, Special Publication **30**. London: Royal Numismatic Society.

NORTHOVER, J.P. (**1998**b)
Exotic alloys in antiquity. IN: *Metallurgica antiqua: in honour of Hans-Gert Bachmann and Robert Maddin*, (Rehren, Th., Hauptmann, A. & Muhly, J., eds): 113-121. Der Anschnitt, Beiheft **8**. Bochum: Deutsches Bergbau-Museum.

NOTHOVER, P., BROOKS, H., LISTER, J. & LLOYD-MORGAN, G. (**1991**)
Fine metalwork from the Roman cemtery at Stansted Airport, Essex tinning and high tin alloys. IN: *Materials Issues in Art and Archaeology II*, (Vandiver, P.B., Druzik, J. & Wheeler, G.S., eds): 719-724. Materials Research Society Symposium Proceedings **185**. Pittsburgh - Pennsylvania: Materials Research Society.

NOTIS, M.R. (**2002**)
A Ghost Story: Remnant Structures in Corroded Ancient Iron Objects. IN: *Materials Issues in Art and Archaeology VI*, (Vandiver, P.B., Goodway, M. & Mass, J.L., eds). Materials Research Society Symposium Proceedings **712**. *http://www.lehigh.edu/~inarcmet/papers/notis%20mrs%20fall%202001.pdf*.

NOVAK, M., OETTEL, A. & WITZEL, C. (**2000**)
Der Parthisch-Römische Friedhof von Tall Seh Hamad/Magdala. Teil I. Berichte der Ausgrabung Tall Seh Hamad/Dur-Katlimmu (Batsh) Band **5**. Belin: Dietrich Reimer Verlag.

ORTIZ, M.A.H. (**2003**)
Prehistoric Mining and Metallurgy in South West Iberian Peninsula. BAR International Series **1188**. Oxford: Archaeopress.

PAPADOPOULOS, J.K. (**1994**)
A Western Mediterranean amphora fragment from ed-Dur. *Arabian Archaeology and Epigraphy* **5**: 276-279.

PARKER, A.J. (**1992**)
Ancient Shipwrecks of the Mediterranean and the Roman Provinces. BAR International Series **580**. Oxford.

PARKER, S.T. (**1986**)
Romans and Saracens: A History of the Arabian Frontier. American Schools of Oriental Research Dissertation Series **6**. Winona Lake: American School of Oriental Research.

PEAKE, H. (**1928**)
The Copper Mountain of Maggan. *Antiquity* **2**: 452-457.

PEDERSEN, C.H. & BUCHWALD, V.F. (**1991**)
An examination of metal objects from Tell Abraq, U.A.E. *Arabian Archaeology and Epigraphy* **2**: 1-9.

PELET, P.-L. (**1993**)
Une industrie reconnue: Fer, Charbon, Acier dans le Pays de Vaud. Cahiers d'Archéologie romande **60**. Lausanna: Cahiers d'Archéologie romande.

PERNICKA, E. (**1995**)
Crisis or Catharsis in Lead Isotope Analysis? *Journal of Mediterranean Archaeology* **8** (1): 59-64.

PERNICKA, E. & WAGNER, G.A. (**1982**)
Lead, Silver and Gold in Ancient Greece. IN: *Nordic conference on the application of scientific methods in archaeology, 2nd, Helsinger (Elsinore), Denmark, 17-19 August 1981*, (Mejdahl, V., ed.): 419-423. PACT: Journal of the European Study Group on Physical, Chemical and Mathematical Techniques applied to Archaeology **7** (2).

PERNICKA, E., REHREN, TH. & SCHMITT-STRECKER, S. (**1998**)
Late Uruk silver production by cupellation at Habuba Kabira, Syria. IN: *Metallurgica antiqua: in honour of Hans-Gert Bachmann and Robert Maddin*, (Rehren, Th., Hauptmann, A. & Muhly, J., eds): 123-134. Der Anschnitt, Beiheft **8**. Bochum: Deutsches Bergbau-Museum.

PHILLIPS, C.S. (**1987**a)
Ed-Dur, 1986/7. Progress Report. Unpublished.

PHILLIPS, C.S. (**1987**b)
Wadi al-Qawr, Fashgha 1. The excavations of a prehistoric burial structure in Ras al-Khaimah, U.A.E., 1986. Project Paper **7**. Edinburgh: Department of Archaeology, University of Edenburgh.

PHOTOS, E., FILIPPAKIS, S.J. & SALTER, C.J. (**1996**)
Preliminary investigations of some metallurgical remains at Knossos, Hellenistic to 3rd c AD. IN: Furnaces and Smelting Technology in Antiquity, (Craddock, P.T. & Hughes, M.J., eds): 189-197. British Museum Occasional Paper **48**. London: British Museum.

PIACENINI, V.F. (**1985**)
Ardashir I Papakan and the wars against the Arabs: working hypoyhesis on the Sasanian hold of the Gulf. *Proceedings of the Seminar for Arabian Studies* **15**: 57-77.

PIGOTT, V.C. (**1989**b)
Bronze in pre-Islamic Iran. IN: *Encyclopaedia Iranica* **4** (5), (Yarshater, E., ed.): 457-471. New York:Routledge & Kegan Paul.

PIGOTT, V.C. (**1989**c)
The Emergence of Iron Use at Hasanlu. *Expedition* **31** (2-3): 67-79.

PIGOTT, V.C. (**1999**d)
The Archaeometallurgy of the Asian Old World. Introductory Comments. IN: *The Archaeometallurgy of the Asian Old World*, (Pigott, V.C., ed.): 1-13. MASCA Reasearch Papers in Science and Archaeology [Univesity Museum Monograph 89; University Museum Symposium Series VII] **16**. Pennsylvania: Science Press.

PIGOTT, V.C. (**2004**)
Zur Bedeutung Irans für die Erforschung prähistorischer Kupfermetallurgie. IN: *Persiens Antike Pracht. Bergbau - Handwerk - Archäologie. Katalog der Ausstellung der Deutschen Bergbau-Museums Bochum von 28. November 2004 bis 29. Mai 2005. Band 1*, (Stöllner, T., Slotta, R. & Vatandoust, A., eds): 28-43. Bochum: Deutschen Bergbau-Museums.

PLEINER, R. (**2000**)
Iron In Archaeology. The European Bloomery Smelters. Prague: Archeologicky Ustav Avcr.

PLISSON, G. (**2005**)
Some Thoughts on Exchange Systems in the Red Sea Region and Indian Ocean. IN: *People of the Red Sea. Proceedings of Red Sea Project II. Held in the British Museum October 2004*, (Starkey, J.C.M., ed.): 67-73. BAR International Series **1395**. Oxford: Archaeopress.

PLOQUIN, A. & ORZECHOWSKI, S. (**1994**)
Palaeo-metallurgy at Mleiha. Preliminary notes. IN: *Archaeological Surveys and Excavations in the Sharjah Emirate, 1993 and 1994. A Seventh Interim Report*, (Mouton, M., ed.): 25-32. Lyon - Sharjah: CRNS - Directore of Antiquities and Heritage, Department of Culture.

PLOQUIN, A., ORZECHOWSKI, S. & BRIAND, B. (**1999**)
Paléométallurgie à Mleiha: Une première approche. IN: *Mleiha I. Environnement, stratégies de subsistance et artisanats*, (Mouton, M., ed.): 171-190. TMO (Travaux de la Maison de l'Orient) **29**. Lyon: Maison de l'Orient Méditerrenéen.

POLLARD, A.M. (**1983**)
X-ray fluorescence analysis of the Appleford hoard of Romano-British pewter. *Historical Metallurgy* **17** (2): 83-90.

POLLARD, N. (**2000**)
Soldiers, Cities & Civilians in Roman Syria. Michigan: The University of Michigan Press.

PONTING, M. & SEGAL, I. (**1998**)
Inductively Coupled Plasma-Atomic Emission Spectroscopy analyses of Roman military copper-alloy artefacts from the excavations at Masada, Israel. *Archaeometry* **40** (1): 109-122.

PONTING, M.J. (**1999**)
East meets West in Post-Classical Bet She'an: The Archaeometallurgy of Culture Change. *Journal of Archaeological Science* **26**: 1311-1321.

PONTING, M.J. (**2002**)
Roman military copper-alloy artefacts from Israel: Questions of organization and ethnicity. *Archaeometry* **44** (4): 555-571.

PONTING, M.J. (**2003**)
From Damascus to Denia: scientific analysis of three groups of Fatimid period metalwork. *Historical Metallury* **37** (2): 85-105.

POTTER, D.S. (**2004**)
The Roman Empire at Bay, AD 180-395. London - New York: Routledge.

POTTS, D., MUGHANNUM, A.S., FRYE, J. & SANDERS, D. (**1978**)
Preliminary Report on the Second Phase of the Eastern Province Survey 1397/1977. *Atlal. The Journal of Saudi Arabian Archaeology* **2**: 7-28.

POTTS, D.T. (**1984**)
Northeastern Arabia in the Later Pre-Islamic Era. IN: *Arabie Orientale, Mésopotamie et Iran Méridional de l'âge du fer au début de la période Islamique.*, (Boucharlat, R. & Salles, J.-F., eds): 85-144. Mémoire **37** . Paris: Editions de recherche sur les civilisations.

POTTS, D.T. (**1988**)
Arabia and the Kingdom of Characene IN: *Araby the Blest. Studies in Arabian Archaeology*, (Potts, D.T., ed.): 137-168. Carsten Niebuhr Institute Publications **7**. Copenhagen: Museum Tusculanum Press.

POTTS, D.T. (**1990**a)
The Arabian Gulf in Antiquity, Vol. I: From Prehistory to the Fall of the Achaemenid Empire. Oxford: Clarendon Press.

POTTS, D.T. (**1990**b)
The Arabian Gulf in Antiquity, Vol. II: Alexander the Great to the Coming of Islam. Oxford: Clarendon Press.

POTTS, D.T. (**1991**)
The Pre-Islamic Coinage of Eastern Arabia. Carsten Niebuhr Institute Publications **14**. Copenhagen: Museum Tusculanum Press.

POTTS, D.T. (**1994**)
Supplement to the Pre-Islamic Coinage of Eastern Arabia. Carsten Niebuhr Institute Publications **16**. Copenhagen: Museum Tusculanum Press.

POTTS, D.T. (**1996**)
The Parthian Presence in the Arabian Gulf. IN: *The Indian Ocean in Antiquity*, (Reade, J., ed.): 269-285. London - New York: Kegan Paul International.

POTTS, D.T. (**1997**a)
Before the Emirates: An Archaeological and Historical Account of Developments in the Region ca. 5000 BC to 676 AD. IN: *Perspectives on the United Arab Emirates*, (Ghareeb, E. & Abed, I.A., eds): 36-73. London: Trident Press.

POTTS, D.T. (**1997**b)
The Roman Relationship with the "Persicus sinus" from the Rise of Spasunou Charax (127 BC) to the Reign of Shapur II (AD 309-379). IN: *The Early Roman Empire in the East*, (Alcock, S.E., ed.): 89-107. Oxbow Monograph **95**. Oxford: Oxbow Books.

POTTS, D.T. & CRIBB, J. (**1995**)
Sasanian and Arab-Sasanian coins from Eastern Arabia. *Iranica Antiqua* **30**: 123-139.

PRANGE, M.K., GÖTZE, H.-J., HAUPTMANN, A. & WEISGERBER, G. (**1999**)
Is Oman the Ancient Magan? Analytical Studies of Copper from Oman. IN: *Metals in Antiquity*, (Young, S.M.M, Pollard, A.M., Budd, P. & Ixer, R.A., eds): 187-192. BAR International Series **792**. Oxford: Archaeopress.

RAY, H.P. (**2003**)
The Archaeology of Seafaring in Ancient South Asia. Cambridge: Cambridge University Press.

REHREN, TH. (**1999**a)
"The same … but different": A juxtaposition of Roman and Medieval brass making in Central Europe. IN: *Metals in Antiquity*, (Young, S.M.M, Pollard, A.M., Budd, P. & Ixer, R.A., eds): 252-257. BAR International Series **792**. Oxford: Archaeopress.

REHREN, TH. (**1999**b)
Small size, large scale: Roman brass production in Germania Inferior. *Journal of Archaeological Science* **26**: 1083-1087.

REHREN, TH. & KLAPPAUF, L. (**1995**)
... ut oleum aquis - Vom Schwimmen des Silbers auf Bleiglätte. *Metalla* **2**: 19-28.

REHREN, TH. & KRAUS, K. (**1999**)
Cupel and crucible: the refining of debased silver in the Colonia Ulpia Traiana, Xanten. *Journal of Roman Archaeology* **12**: 263-272.

REHREN, TH. & PAPAKHRISTU, O. (**2000**)
Cutting Edge Technology - The Ferghana Process of medieval crucible steel smelting. *Metalla* **7** (2): 55-69.

REHREN, TH. & PRANGE, M. (**1998**)
Lead metal and patina: a comparison. IN: *Metallurgica antiqua: in honour of Hans-Gert Bachmann and Robert Maddin*, (Rehren, Th., Hauptmann, A. & Muhly, J., eds): 183-195. Der Anschnitt, Beiheft **8**. Bochum: Deutsches Bergbau-Museum.

RENFREW, C. & BAHN, P. (**1997**)
Archaeology. Theories, Methods and Practise. Second Edition. London: Thames and Hudson Ltd.

RETSÖ, J. (**2003**)
The Arabs in Antiquity. Their history from the Assyrians to the Umayyads. London & New York: RoutledgeCurzon.

ROBIN, C. (**1974**)
Monnaies provenant de l'Arabie du nord-est. *Semitica* **24**: 83-125.

ROBIN, C. (**1997**)
The Date of the Periplus of the Erythraean Sea in the Light of South Arabian Evidence. IN: *Crossings. Early Mediterranean Contacts with India*, (De Romanis, F. & Tchernia, A., eds): 41-65. New Delhi: Manohar.

ROGIERS, P. (**2006**)
Spoorelementanalyse van metallische objecten van de archeologische site Ed-Dur (VAE, Umm Al-Qaiwain) middels ICP-massaspectrometrie. Ghent: Unpublished licentiaatsthesis - Vakgroep Analytische Chemie, Universiteit Gent.

ROHL, B. & NEEDHAM, S.P. (**1998**)
The Circulation of Metals in the British Bronze Age / The Application of Lead Isotope Analysis. British Museum Occasional Paper **102**. London: British Museum Press.

ROSENFELD, A., ILANI, S. & DVORACHEK, M. (**1997**)
Bronze Alloys from Canaan During the Middle Bronze Age. *Journal of Archaeological Science* **24**: 857-864.

ROSENFIELD, J.M. (**1967**)
The Dynastic Arts of the Kushans. Berkeley and Los Angeles: University of California Press.

ROSTOKER, W., BRONSON, B. & DVORAK, J.R. (**1989**)
Smelting to Steel by the Japanese Tatara Process. *Archeomaterials* **3** (1): 11-25.

ROSTOVTZEFF, M. (**1922**)
Iranians and Greeks in South Russia. Oxford: Clarendon Press.

RUTTEN, K. (**2006**)
Het aardewerk van ed-Dur (Umm al-Qaiwain, V.A.E.) uit de late 1ste eeuw v. tot de vroege 2de eeuw n. Chr. Technologische, typologische en vergelijkende studie met een analyse van de ruiltelijke verspreiding en handel in en voorbij de Perzische Golf. Ghent: Unpublished Ph.D. Thesis - Vakgroep Talen en Culturen van het Nabije Oosten en Noord-Afrika, Universiteit Gent.

RUTTEN, K. (**2007**)
The Roman fine wares of ed-Dur (Umm al-Qaiwain, U.A.E.) and their distribution in the Persian Gulf and the Indian Ocean. *Arabian Archaeology and Epigraphy* **18**: 8-24.

SALLES, J.-F. (**1980**a)
Monnaies d'Arabie Orientale: éléments pour l'histoire des Emirats Arabes Unis à l'époque historique. *Proceedings of the Seminar for Arabian Studies* **10**: 97-109.

SALLES, J.-F. (**1980**b)
Notes on the archaeology of the Hellenistic and Roman periods in the United Arab Emirates. *Archaeology in the United Arab Emirates* **2-3**: 79-91.

SALLES, J.-F. (**1984**a)
Bahrain "Hellénistique": Données et Problèmes. IN: *Arabie Orientale, Mésopotamie et Iran Méridional de l'âge du fer au début de la période Islamique*, (Boucharlat, R. & Salles, J.-F., eds): 151-163. Mémoire **37**. Paris: Editions de recherche sur les civilisations.

SALLES, J.-F. (**1984**b)
Introduction. IN: *Failaka: Fouilles Françaises 1983*, (Salles, J.-F., ed.): 9-19. TMO (Travaux de la Maison de l'Orient) **9**. Lyon: Maison de l'Orient Méditerrenéen.

SALLES, J.-F. (**1987**)
The Arab-Persian Gulf under the Seleucids. IN: *Hellenism in the East. The interaction of Greek and non-Greek civilizations from Syria to Central Asia after Alexander*, (Kuhrt, A. & Sherwin-White, S., eds): 75-119. Berkeley - Los Angeles: University of California Press.

SALLES, J.-F. (**1993**)
The Periplus of the Erythraean Sea and the Arab-Persian Gulf. *Topoi* **3** (2): 493-523.

SALLES, J.-F. (**1996**a)
Achaemenid and Hellenistic Trade in the Indian Ocean. IN: *The Indian Ocean in Antiquity*, (Reade, J., ed.): 251-267. London - New York: Kegan Paul International.

SALLES, J.-F. (**1996**b)
Hellenistic Seafaring in the Indian Ocean a Perspective from Arabia. IN: *Tradition and Archaeology, Early Maritime Contacts in the Indian Ocean. Proceedings of the International Seminar "Techno-Archaeological Perspectives of Seafaring in the Indian Ocean 4th cent. B.C. - 15 th cent. A.D." (New Dheli, Febr. 28 - March 4, 1994)*, (Ray, H.P. & Salles, J.-F., eds): 293-309. New Delhi: Manohar.

SALLES, J.-F. (**2000**)
Bahrain, from Alexander the Great to the Sasanians. IN: *Traces of Paradise: The Archaeology of Bahrain 2500BC-300AD*, (Rice, M. & Crawford, H., ed.): 132-135. Bahrain: Bahrain National Museum.

SALMAN, I. (**1972**)
Iraqi Archaeological Expedition to Arab Princedom States. *Sumer* **28**: 7-8.

SALTER, C. (**1987**)
Ferrous metallurgy and other slags. IN: *Hengistbury Head, Dorset, Vol.1: The prehistoric and roman settlement, 3500 BC - AD 500*, (Cunliffe, B., ed.): 197-205. Oxford University Committee for Archaeology Monograph **13**.

SALTER, C. (**1994**)
Analysis of iron smithing slag. IN: *Qala'at al-Bahrain, volume 1. The Northern City Wall and the Islamic fortress*, (Højlund, F. & Andersen, H.H., eds): 374-385. Jutland Archaeological Society Publications **30**. Moesgård: Aarhus.

SCHOFF, W.H. (**1915**)
The eastern iron trade of the Roman empire. *Journal of American Oriental Society* **35**: 224-239.

SCHOFF, W.H. (**1974**)
The Periplus of the Erythraean sea : travel and trade in the Indian ocean by a merchant of the first century / Transl. from the Greek and annotated by Wilfred H. Schoff. New Delhi: Oriental Books Reprint Corporation.

SCHRÜFER-KOLB, I. (**2004**)
Roman Iron Production in Britain. Technological and socio-economic landscape development along the Jurassic Ridge. BAR British Series **380**. Oxford: Archaeopress.

SCHWARZE, J. & DEAL, E.C. (**1986**)
A Sword Hilt from the Serçe Liman Shipwreck. *MASCA Journal* **4** (2): 50-59.

SCOTT, B.G. (**1990**)
Early Irish Ironworking. Belfast: Ulster Museum.

SCOTT, D.A. (**1991**)
Metallography and Microstructure of Ancient and Historic Metals. Los Angeles: Getty Conservation Institute.

SEDOV, A.V. (**1996**)
Qana' (Yemen) and the Indian Ocean: the archaeological evidence. IN: *Tradition and Archaeology, Early Maritime Contacts in the Indian Ocean. Proceedings of the International Seminar "Techno-Archaeological Perspectives of Seafaring in the Indian Ocean 4th cent. B.C. - 15 th cent. A.D." (New Dheli, Febr. 28 - March 4, 1994)*, (Ray, H.P. & Salles, J.-F., eds): 11-35. New Delhi: Manohar.

SEELEY, N.J. & TURNER, P.J. (**1984**)
Metallurgical investigation of three early Indian coinages: implications for metal trading and dynastic chronology. IN: *South Asian archaeology 1981: proceedings of the sixth International Conference of the Association of South Asian Archaeologists in Western Europe*, (Allchin, B., ed.): 331-333. University of Cambridge oriental publications **34**. Cambridge: Cambridge University Press.

SELLWOOD, D. (**1976**)
Minting. IN: *Roman Crafts*, (Strong, D. & Brown, D., eds): 62-73. London: Gerald Duckworth & Co. Ltd.

SENIOR, R.C. (**1994**)
Trade and coinage in Eastern Arabia c. 100 BC - 100 AD. London.

SERNEELS, V. (**1993**)
Archéométrie des scories de fer. Recherches sur la sidérurgie ancienne en Suisse occidentale. Cahiers d'Archéologie romande **61**. Lausanne: Cahiers d'Archéologie romande.

SERNEELS, V. (**1995**b)
Du minérai à l'objet: un village de sidérurgistes du VIIIe siècle à Liestal-Röserntal (BL/Switzerland). IN: *The Importance of Ironmaking, Technical Innovation and Social Change, Volume I. Papers presented at the Norberg Conference on May 8-13, 1995*, (Magnusson, G., ed.): 124-131. Jernkontorets Berghistoriska Utskott **58** . Stockholm.

SERNEELS, V. (**1998**)
La chaîne opératoire de la sidérurgie ancienne. IN: *Recherches sur l'économie du fer en Méditerranée nord-occidentale*, (Feugère, M. & Serneels, V., eds): 7-44. Monographies instrumentum **4**. Montagnac: Mergoil.

SERNEELS, V. (**2004**)
Chapitre 1: Le fer et ses minerais. IN: *Le Fer*, (Mangin, M., ed.): 25-48. Paris: éditions Errance.

SERNEELS, V. & PERRET, S. (**2003**)
Quantification of smithing activities based on the investigation of slag and other material remains. IN: *Archaeometallurgy in Europe. 24-25-26 September 2003 - Milan, Italy. Proceedings Vol. 1.*: 469-486. Milan: Associazione Italiana di Metallurgia.

SERNEELS, V., MERLUZZO, P. & LEROY, M. (**2004**)
Chapitre 3: Les activités de forge: Le travail du métal. IN: *Le Fer*, (Mangin, M., ed.): 81-112. Paris: éditions Errance.

SEYRIG, M.H. (**1937**)
Armes et costumes iraniens de Palmyra. *Syria* **18**: 4-53.

SHEPHERD, R. (**1993**)
Ancient Mining. London - New York: Elsevier Applied Science.

SIDEBOTHAM, S.E. (**1991**)
Ports of the Red Sea and the Arabia-India Trade. IN: *Rome and India. The Ancient Sea Trade*, (Begley, V. & De Puma, R.D., eds): 12-38. Madison: The University of Wisconsin Press.

SIM, D. (**1998**)
Beyond the Bloom: Bloom refining and iron artifacts production in the Roman world. BAR International Series **725**. Oxford: Archaeopress.

SIM, D. & RIDGE, I. (**2002**)
Iron for the Eagles: the Iron Industry of Roman Britain. Stroud: Tempus Publishing Ltd.

SIMPSON, ST. J. (**2001**)
The Early Islamic crucible steel industry at Merv. *IAMS Journal* **21**: 14-15.

SMITH, CH. (**2003**)
A Gift to the Gods or a Cache in Crisis - Pewter in Roman Britain. *Journal of the Pewter Society* **Autumn**: 2-9.

SMYTHE, J.A. (**1937**)
Notes on Ancient and Roman Tin and its Alloys with Lead. *Transactions of the Newcomen Society* **18**: 255-265.

SONO, T. & FUKAI, S. (**1968**)
Dailaman III. The Excavations at Hassani Mahali and Ghalekuti 1964. The Tokyo University Iraq-Iran Archaeological Mission Report **8**. Tokyo: The Yamakawa Publishing Co., Ltd.

SRINIVASAN, S. & GLOVER, I. (**1997**)
The archaeometallurgical implications of new findings of traditional crafts of making high tin "delta" bronze mirrors and "beta" bronze vessels in Kerala State of South India. IN: *Materials Issues in Art and Archaeology V.*, (Vandiver, P.B., Druzik, J.R., Merkel, J.F. & Stewart, J.): 81-103. Materials Research Society Symposium Proceedings **462**. Pittsburgh - Pennsylvania: Materials Research Society.

STANIASZEK, B.E.P. & NORTHOVER, J.P. (**1983**)
The properties of leaded bronze alloys. IN: *The Proceedings of the 22nd Symposium on Archaeometry, University of Bradford, Bradford, U.K. March 30th - April 3rd 1982*, (Aspinall, A. & S.E. Warren, S.E., eds): 262-272. Braford: University of Bradford.

STONE, P. (**2001**)
Human remains from Area AV G 5156. IN: *Excavations at ed-Dur (Umm al-Qaiwain, United Arab Emirates), Vol. II: The Tombs. The University of Ghent South-East Arabian Archaeological Project*, (Haerinck, E.): 57-64. Leuven: Peeters.

STOS-GALE, Z.A. (**1998**)
Lead isotope analysis of coins - a review. IN: *Metallurgy in Numismatics, Volume 4*, (Oddy, W.A. & Cowell, M., eds): 348-366. Royal Numismatic Society, Special Publication **30**. London: Royal Numismatic Society.

TANABE, K. (**1985**)
Date and Significance of the So-Called Investiture of Ardashir II and the Images of Shapur II and III at Taq-i Bustan. *Orient* **21**: 111-112.

TATE, G. (**1997**)
The Syrian Countryside during the Roman Era. IN: *The Early Roman Empire in the East*, (Alcock, S.E., ed.): 55-71. Oxbow Monograph **95**. Oxbow: Oxbow Books.

THAPAR, R. (**1997**)
Early Mediterranean Contacts with India: An Overview. IN: *Crossings. Early Mediterranean Contacts with India*, (De Romanis, F. & Tchernia, A., eds): 11-40. New Delhi: Manohar.

THOLANDER, E. (**1989**)
Microstructure examination of slags as an instrument for identification of ancient iron-making processes. IN: *Archaeometallurgy of Iron 1967-1987. International Symposium of the Comité pour la sidérurgie ancienne, Liblice 5 - 9 October 1987*, (Pleiner, R., ed.): 35-42. Prague: Geofyzika n.p. Brno.

THORNTON, C.P. & EHLERS, C.B. (**2003**)
Early brass in the ancient Near East. *IAMS Journal* **23**: 3-8.

THORNTON, C.P., LAMBERG-KARLOVSKY, C. C, LIEZERS, M. & YOUNG, S.M.M. (**2002**)
On Pins and Needles: Tracing the Evolution of Copper-base Alloying at Tepe Yahya, Iran, via ICP-MS Analysis of Common-place Items. *Journal of Archaeological Science* **29**: 1451-1460.

TITE, M.S. (**1972**)
Methodes of Physical Examination in Archaeology. London - New York: Seminar Press.

TITE, M.S. (**1996**)
In defence of lead isotope analysis. *Antiquity* **70** (270): 959-962.

TODD, J.A. & CHARLES, J.A. (**1977**)
The Analysis of non-metallic inclusions in Ancient Iron. IN: *L' analyse par microfluorescence X appliquée à l'archéologie / X-ray Microfluorescence Analysis Applied to Archaeology. Proceedings of the Symposium Organized by Mmn Madeleine Hours at the Laboratoire des Musées de France, Paris, in June 1977*, (Hours, M., ed.): 204-220. PACT: Journal of the European Study Group on Physical, Chemical and Mathematical Techniques applied to Archaeology **1**. Strasbourg: Council of Europe.

TODD, J.A. & CHARLES, J.A. (**1978**)
Ethiopian Bloomery Iron and the significance of inclusion analysis in iron studies. *Historical Metallurgy* **12** (2): 63-87.

TOMBER, R. (**2004**)
Rome and South Arabia: new artefactual evidence from the Red Sea. *Proceedings of the Seminar for Arabian Studies* **34**: 351-360.

TOSI, M. (**1975**)
Notes on the Distribution and Exploitation of Natural Recources in Ancient Oman. *Journal of Oman Studies* **1**: 187-206.

TREISTER, M.Y. (**1990**)
About the Early Production of Metallic Zinc in Parthia. *Bulletin of the Metals Museum* **15**: 33-40.

TREISTER, M.Y. (**1995**)
Once More about the Trade in Metals in the Greek and Roman World. *Bulletin of the Metals Museum* **23**: 19-35.

TREISTER, M.Y. (**1996**)
The Role of Metals in Ancient Greek History. Mnemosyne: bibliotheca classica Batava. Supplementum **156**. Leiden - New York - Köln: E.J. Brill.

TROUSDALE, W. (**1975**)
The Long Sword and Scabbard Slide in Asia. Smithsonian Contributions to Anthropology **17**. Washington D.C.: Smithsonian institution Press.

TRÜMPELMANN, L. (**1975**)
Iranische Felsreliefs B. Das Sasanidische Felsrelief van Darab. Iranische Denkmäler, Lieferung 6 enthaltend Reihe II. Berlin: Dietrich Reimer Verlag.

TYLECOTE, R.F. (**1962**)
Metallurgy in Archaeology: A Prehistory of Metallurgy in the British Isles. London: Edward Arnold Publishers Ltd.

TYLECOTE, R.F. (**1976**)
A History of Metallurgy. London: The Metals Society.

TYLECOTE, R.F. (**1987**)
The early history of metallurgy in Europe. London - New York: Longman.

TYLECOTE, R.F. & GILMOUR, B.J.J. (**1986**)
The Metallography of Early Ferrous Edge Tools and Edged Weapons. BAR British Series **155**. Oxford: Archaeopress.

UNGLIK, H. (**1991**)
Structure, Composition, and Technology of Late Roman Copper Alloy Artifacts from Canadian Excavations at Carthage. *Archeomaterials* **5** (1): 91-110.

VAN NEER, W. & GAUTIER, A. (**1993**)
Preliminary report on the faunal remains from the coastal site ed-Dur, 1st-4th century A.D. Umm Al-Quwain, United Arab Emirates. IN: *Archaeozoology of the Near East. Proceedings of the first international symposium on the archaeozoology of southwestern Asia and adjacent areas*, (Buitenhuis, H. & Clason, A.T., eds): 110-118. Leiden: Universal Book Services/Dr. W.Backhuys.

VANDEN BERGHE, L. (**1983**)
Oud-Iraanse rotsreliëfs. Brussels: Koninklijke Musea voor Kunst en Geschiedenis.

VERHOEVEN, J. (**2001**)
The Mystery of Damascus Blades. *Scientific American* **284**: 62-67.

VERHOEVEN, J.D. (**1987**)
Damascus Steel Part I: Indian Wootz Steel. *Metallography* **20**: 145-151.

VERHOEVEN, J.D. & JONES, L.L. (**1987**)
Damascus Steel, Part II: Origine of the Damask Pattern. *Metallography* **20**: 153-180.

VERHOEVEN, J.D. & PENDRAY, A.H. (**1992**a)
Experiments to reproduce the pattern of Damascus steel blades. *Materials Characterization* **29**: 195-212.

VERHOEVEN, J.D. & PENDRAY, A.H. (**1992**b)
A reconstructed method for making Damascus steel blades. *Metals Materials Proceeding* **4**: 93-106.

VERHOEVEN, J.D. & PENDRAY, A.H. (**1993**a)
Studies of Damascus steel blades, I: Experiments on reconstructed blades. *Materials Characterization* **30**: 175-186.

VERHOEVEN, J.D. & PENDRAY, A.H. (**1993**b)
Studies of Damascus steel blades, II: Destruction and reformation of the pattern. *Materials Characterization* **30**: 187-200.

VERHOEVEN, J.D. & PETERSON, D.T. (**1993**c)
What is a Damascus Steel? *Materials Characterization* **29**: 335-341.

VERHOEVEN, J.D., PENDRAY, A.H. & DAUKSCH, W.E. (**1998**)
The Key Role of Impurities in Ancient Damascus Steel Blades. *Journal of Metals* **50** (9): 58-64.

VERHOEVEN, J.D., PENDRAY, A.H. & GIBSON, E.D. (**1996**)
Wootz Damascus Steel Blades. *Materials Characterization* **37**: 9-22.

VLACHOU, C., MCDONNELL, J.G. & JANAWAY, R.C. (**2002**)
Experimental Investigation of Silvering in Late Roman Coinage. IN: *Materials Issues in Art and Archaeology VI*, (Vandiver, P.B., Goodway, M. & Mass, J.L., eds). Materials Research Society Symposium Proceedings **712**. *http://www.mrs.org/publications/epubs/proceedings/fall2001/ii/*.

VOGT, B. (**1984**)
1st Mill. B.C. Graves and Burial Customs in the Samad Area (Oman). IN: *Arabie Orientale, Mésopotamie et Iran Méridional de l'âge du fer au début de la période Islamique*, (Boucharlat, R. & Salles, J.-F., eds): 271-284. Mémoire **37**. Paris: Editions de recherche sur les civilisations.

WAGNER, R. (**2000**)
Searching ancient copper sources - a lead isotope study of Roman bronze vessels from Pompeii. *Kölner Jahrbuch* **33**: 615-616 .

WARMINGTON, E.H. (**1974**)
The Commerce between the Roman Empire and India. London & Dublin: Curzon Press Ltd.

WAYMAN, M.L. & JULEFF, G. (**1999**)
Crucible steelmaking in Sri Lanka. *Historical Metallurgy* **33** (1): 26-42.

WEEKS, L. (**1996**)
Area B. IN: *Excavations at Al Sufouh: A third Millenium Site in the Emirate of Dubai*, (Benton, J.N., ed.): 21-31. Abiel, New Research on the Arabian Peninsula **1**. Brussels: Brepols.

WEEKS, L. (**1997**)
Prehistoric Metallurgy at Tell Abraq, United Arab Emirates. *Arabian Archaeology and Epigraphy* **8**: 11-85.

WEEKS, L. (**1999**)
Lead Isotope Analyses from Tell Abraq, United Arab Emirates: New Data regarding the "Tin Problem in Western Asia". *Antiquity* **73** (279): 49-64.

WEEKS, L.R. (**2000**a)
Pre-Islamic Metallurgy of the Gulf. Sydney: Unpublished Ph.D. Thesis - Near Eastern Archaeology, University of Sydney.

WEEKS, L. (**2000**b)
Metal artefacts from Sharm tomb. *Arabian Archaeology and Epigraphy* **11**: 180-198.

WEEKS, L. (**2002**)
Prehistoric Metallurgy in the U.A.E.: Bronze Age-Iron Age Transition. IN: *Archaeology of the United Arab Emirates. Proceedings of the First International Conference on the Archaeology of the U.A.E.*, (Potts, D., Al Naboodah, H. & Hellyer, P., eds): 115-121. Galway: Trident Press.

WEEKS, L. & COLLERSON, K. (**2005**)
Chapter 9: Archaeometallurgical studies. IN: *The Early Dilmun Settlement at Saar*, (Killick, R. & Moon, J., eds): 309-324. London-Bahrain Archaeological Expedition Saar Excavation Report **3**. Ludlow: Archaeology International Ltd.

WEEKS, L.R. (**2004**a)
An Analysis of Late Pre-Islamic Copper-Base Artefacts from Ed Dur, U.A.E. *Arabian Archaeology and Epigraphy* **15**: 240-252.

WEEKS, L.R. (**2004**b)
Early Metallurgy of the Persian Gulf. Technology, Trade, and the Bronze Age World. American School of Prehistoric Research Monograph Series. Boston - Leiden: Brill Academic Publishers, Inc.

WEISGERBER, G. (**1978**a)
Evidenceof ancient mining sites in Oman: a preliminary report. *Journal of Oman Studies* **4**: 15-28.

WEISGERBER, G. (**1980**a)
Archäologische und Archäometallurgische Untersuchungen in Oman. *Allgemeine und Vergleichende Archäologie - Beiträge* **2**: 67-90.

WEISGERBER, G. (**1980**b)
Patterns of Early Islamic Metallurgy in Oman. *Proceedings of the Seminar for Arabian Studies* **10**: 115-126.

WEISGERBER, G. (**1980**c)
" ... und Kupfer in Oman". Das Oman-Projekt des Deutschen Bergbau-Museums. *Der Anschnitt* **32** (2-3): 62-110.

WEISGERBER, G. (**1981**)
Mehr als Kupfer in Oman - Ergebnisse der Expedition 1981. *Der Anschnitt* **33** (5-6): 174-263.

WEISGERBER, G. (**1983**)
Copper Production during the Third Millenium B.C., in Oman and the question of Makkan. *Journal of Oman Studies* **6**: 269-276.

WEISGERBER, G. (**1984**)
Makkan and Meluhha - Third Millenium BC Copper Production in Oman and the Evidence of Contact With the Indus Vally. IN: *South Asian archaeology 1981: proceedings of the sixth International Conference of the Association of South Asian Archaeologists in Western Europe*, (Allchin, B., ed.): 196-201. University of Cambridge oriental publications **34**. Cambridge: Cambridge University Press.

WEISGERBER, G. (**1987**)
Archaeological evidence of copper exploitation at 'Arja. IN: *The Hinterland of Sohar. Archaeological Surveys and Excavations within the Region of an Omani Seafaring City*, (Costa, P.M. & Wilkinson, T.J.): 145-172. Journal of Oman Studies **9**.

WEISGERBER, G. (**1988**)
Oman: a bronze-producing centre during the 1st Half of the 1st millennium BC. IN: *Bronzeworking Centres of Western Asia c. 1000-539 B.C.*, (Curtus, J., ed.): 285-295. London: Keegan Paul International.

WEISGERBER, G. (**1991**)
Die Suche nach dem altsumerischen Kupferland Makan. *Das Altertum* **37** (2): 76-90.

WEISSER, T. (**1975**)
The de-alloying of copper alloys. IN: *Conservation in Archaeology and the Applied Arts, IIC Congress, Stockholm*: 207-214. Stockholm.

WERTIME, T.A. (**1962**)
The Coming of the Age of Steel. Chicago: University of Chicago Press.

WHITEHOUSE, D. (**1996**)
Sasanian Maritime Activity. IN: *The Indian Ocean in Antiquity*, (Reade, J., ed.): 339-349. London - New York: Kegan Paul International.

WHITEHOUSE, D. (**1998**)
Excavations at ed-Dur (Umm al-Qaiwain, United Arab Emirates), vol. I: The Glass Vessels. The University of Ghent South-East Arabian Archaeological Project, (Haerinck, E., ed.). Leuven: Peeters.

WILKINSON, J.C. (**1977**)
Water and Tribal Settlement in South-East Arabia. A Study of the Aflaj of Oman. Oxford: Clarendon Press.

WILLIAMS, J.G. (**1998**)
Inductively Coupled Plasma Mass Spectrometry: a New Scientific Method for Investigating Coins and Coinage. IN: *Metallurgy in Numismatics, Volume 4*, (Oddy, W.A. & Cowell, M., eds): 253-261. Royal Numismatic Society, Special Publication **30**. London: Royal Numismatic Society.

WINKELMANN, S. (**2003**)
Eurasisches in Hatra? Ergebnisse und Probleme bei der Analyse partherzeitlicher Bildquellen. IN: *Mitteilungen des SFB. "Differenz und Integration": Nomaden und Sesshaft - Fragen, Methoden, Ergebniss*, (Holzwarth, W., ed.): 21-140. Halle - Saale: Orientwissenschaftliches Zentrum der Martin-Luther-Universität Halle-Wittenberg.

YOUNG, G.K. (**2001**)
Rome's Eastern Trade. International commerce and imperial policy 31 BC-AD 305. London - New York: Routledge.

YOUNG, M.M., & POLLARD, M.A. (**2000**)
Atomic spectroscopy and spectrometry. IN: *Modern Analytical Methods in Art and Archaeology*, (Ciliberto, E. & Spoto, G., eds): 21-54. Chemical Analyses Series **155**. New York: Wiley-Interscience.

YULE, P. (**1996**)
The 1995 German archaeological mission to the Sultanate of Oman. *Proceedings of the Seminar for Arabian Studies* **26**: 175-176.

YULE, P. (**1999**a)
Studies in the Archaeology of the Sultanate of Oman. Orient-Archäologie **2**. Rahden: Verlag Marie Leidorf GmbH.

YULE, P. (**1999**b)
The Samad Period in the Sultanate of Oman. *Iraq* **61**: 121-146.

YULE, P. & WEISGERBER, G. (**1996**)
Die Deutsche Archäologische Oman-Expedition. *Mitteilungen der Deutschen Orient-Gesellschaft* **128**: 135-155.

ZOGRAPH, A.N. (**1977**)
Ancient Coinage. Part i. BAR Supplementary Series **33** (1). Oxford.

ZUTTERMAN, CH. (**2003**)
The softstone vessels from ed-Dur, Umm al-Qaiwain, United Arab Emirates. *Arabian Archaeology and Epigraphy* **14**: 81-87 .

ZWICKER, U., ODDY, A. & LA NIECE, S. (**1993**)
Roman techniques of manufacturing silver-plated coins. IN: *Metal Plating and Patination. Cultural, Technical and Historical Developments*, (La Niece, S. & Craddock, P.T., eds): 223-246. Oxford: Butterworth-Heinemann Ltd.

PART IV

The tail

Appendix 1: **Traded goods & ports as mentioned in the *Periplus***[1]

From	***To***	***Goods***
India	Apologos, Omana	**copper**, wood
	Socotra	grain, rice, cotton cloth, female slaves
	Moscha Limen	grain, sesame oil, cotton cloth
	N-Somalia: "far-side'" ports	grain, rice, sesame oil, ghee, cane sugar, cotton cloth, girdles
	Adulis	**iron, steel**, lac, cotton cloth, girdles, cloaks, fine cotton garments
Ptolemais Theron & Adulis	Roman Red Sea ports	ivory, tortoise shell, rhinoceros horn
Avalites		ivory, tortoise shell, aromatics, little myrrh
Malao, Mundu, Masyllon, Spice Port & Opone		myrrh, frankincense cassia, aromatics, drugs, slaves ivory, tortoise shell
Rhapta		ivory, tortoise shell, rhinoceros horn, nautilus shell
Muza		myrrh, white marble
Qana'		aloe, frankincense
Moscha Limen		frankincense (exceptional surcomstances)
Barabarikon & Barygaza		costus, bdellium, *lykion*, nard, Indian myrrh (?), indigo, turquoise, lapis lazuli, onyx, agate, ivory, fine cotton, silk, yarn, Chinese pelts pepper
Muziris/Nelkynda & Argaru		nard, malabathron, pepper, pearls, ivory, tortoise shell, transparent gems, diamonds, sapphires, silk, fine cotton
Desarene, Ganges		nard, malabathron, pearls, ivory, fine cotton
Adulis	Omana	(cheap) cotton cloth
	N-somalia	(cheap) cotton cloth
	Socotra	(cheap) cotton cloth
Muza	African ports & Barygaza	whatever they imported from Adulis
Qana'	African ports, Omana & S-Iran	whatever they imported
Roman Red Sea ports	Adulis	**tools (axes, adzes, knives)**, **iron**, cheap clothing, **brass** and **bronze vessels**, glass, olive oil, wine, **silverware**, **goldware**, cloaks
	Avalitis + "far-side" ports	cheap clothing, grain, wine, **iron**, **ironware**, **tin**, **silverware**, glassware, **drinking vessels**
	Arabia	clothing, textiles, grain, oil, wine, **copper**, **tin**, horses, pack mules, **silverware**, **goldware**, **bronzeware**, deluxe clothing, statuary
	India	**lead**, **tin**, **copper**, drugs, cosmetics, silverware, glassware, coral, multicolored textiles, wine, deluxe clothing, slaves.
	Barabarikon	glassware
	Barygaza	raw glass, **copper**, **tin**, **lead** + more luxury products, wine The Arabian wine destined for Barygaza was probably picked up at Muza
	Qana'	**copper**
	Muziris/Nelkynda	raw glass, **copper**, **tin**, **lead**

[1] After Casson, 1989.

Appendix 2: References used to compose the lead isotope database

AL-SAA'D, Z. (**2000**)
Technology and provenance of a collection of Islamic copper-based objects as found by chemical and lead isotope analysis. *Archaeometry* **42**(2): 385-398.

ATTANASIO, D., BULTRINI, G. & INGO, G.M. (**2001**)
The Possibility of Provenancing a series of Bronze Punic Coins at Tharros (Western Sardinia) using the Literature Lead Isotope Database. *Archaeometry* **43**(4): 529-547.

BARNES, I.L., SHIELDS, W.R. & MURPHY, T.J. (**1974**)
Isotopic Analysis of Laurion Lead Ores. IN: *Archaeological Chemistry.*, (Beck, C.W., ed.): 1-10. Advances in Chemistry Series **138**. Washington D.C.: American Chemical Society.

BEGEMANN, F. & SCHMITT-STRECKER, S. (**1994**)
Das Blei von Schiff und Ladung: Seine Isotopie und mögliche Herkunft. IN: *Das Wrack. Der antike Schiffsfund von Mahdia. Band 2.*, (Hellenkemper Salies, G., von Prittwitzund Gaffron, H.-H. & Bauchhenss, G., eds): 1073-1076. Köln: Rheinland Verlag.

BEGEMANN, F., SCHMITT-STRECKER, S., PERNICKA, E. & LO SCHIAVO, F. (**2001**)
Chemical composition and lead isotopy of copper and bronze from Nuragic Sardinia. *European Journal of Archaeology* **4**(1): 43-85.

BONI, M., DI MAIO, G., FREI, R. & VILLA, I.M. (**2000**)
Lead isotope evidence for a mixed provenance for Roman water pipes from Pompeii. *Archaeometry* **42**(1): 201-208.

BRILL, R.H. & SHIELDS, W.R. (**1972**)
Lead Isotopes in Ancient Coins. IN: *Methods of Chemical and Metallurgical Investigation of Ancient Coinage.*, (Hall, E.T. & Metcalf, D.M., eds): 279-303. Royal Numismatic Society, Special Publication **8**. London: Royal Numismatic Society.

BRILL, R.H. & WAMPLER, J.M. (**1985**)
Isotope Ratios in Archaeological Objects of Lead. IN: *Application of Science in Examination of Works of Art. Proceedings of the Seminar: September 7-9, 1983.*, (England, P.A. & van Zelst, L., eds): 155-171. Boston: Museum of Fine Arts.

BRIQUEU, L., MEVEL, C. & BOEDIER, F. (**1991**)
Sr, Nd and Pb isotopic constraints in the genesis of a calc-alkine plutonic suite in the Oman ophiolite related to obduction processes. IN: *Ophiolite genesis and evolution of the oceanic lithosphere: proceedings of the Ophiolite Conference, held in Muscat, Oman, 7-18 January 1990.*, (Peters, Tj., Nicolas, A. & Coleman, R.G., eds): 517-542. Petrology and structural geology **5**. Sultanate of Oman: Ministry of Petroleum and Minerals.

BUTCHER, K. & PONTING, M. (**2005**)
The Roman denarius under the Julio-Claudian Emperors: mints, metallurgy and technology. *Oxford Journal of Archaeology* **24**(2): 163-197.

CALVEZ, J.Y. & LESCUYER, J.L. (**1991**)
Lead isotope geochemistry of various sulphide deposits from the Oman mountains. IN: *Ophiolite Genesis and the Evolution of the Oceanic Lithosphere.*, (Peters, Tj., Nicolas, A. & Coleman, R.G., eds): 385-397. Sultanate of Oman: Ministry of Petroleum and Minerals.

CHALKIAS, S., VAVALIDIS, M., SCHMITT-STRECKER, S. & BEGEMANN, F. (**1988**)
Geologische Interpretation der Blei-isotopen-verhältnisse van Erzen der Insel Thasos, Der Ägäis und Nordgriechenlands. IN: *Antike Edel- und Buntmetallgewinnung auf Thasos.*, (Wagner, G.A. & Weisgerber, G., eds): 59-74. Der Anschnitt, Beiheft **6**. Bochum: Deutschen Bergbau-Museums.

CHEGINI, N.N., MOMENZADEH, M., PARZINGER, H., PERNICKA, E., STÖLLNER, T., VATANDOUST, R. & WEISGERBER, G. (**2000**)
Preliminary report on archaeometallurgical investigations around the prehistoric site of Arisman near Kashan, western Central Iran. *Archäologische Mitteilungen aus Iran und Turan* **32**: 281-318.

CHEN, J.H. & PALLISTER, J.S. (**1981**)
Lead isotopic studies of the Samail Ophiolite, Oman. *Journal of Geophysical Research* **86**(B4): 2699-2708.

CRADDOCK, P.T., FREESTONE, I.C., GALE, N.H., MEEKS, N.D., ROTHENBERG, B. & TITE, M.S. (**1996**)
The investigation of a small slag heap of silver smelting debris from Rio Tinto, Huelva, Spain. IN: *Furnaces and Smelting Technology in Antiquity.*, (Craddock, P.T. & Hughes, M.J., eds): 199-219. British Museum Occasional Paper **48**. London: British Museum Press.

DOE, B.R. (**1982**)
The lead and strontium isotope geochemistry of metalliferous sediments associated with Upper Cretaceous ophiolitic rocks in Cyprus, Syria, and the Sultanate of Oman: Discussion. *Canadian Journal of Earth Sciences* **19**: 1720-1724.

EISEMAN, C.J. (**1978**)
The Porticello shipwreck: lead isotope data. *MASCA Journal* **1**: 18.

ERICSON, J.E. & SHIRAHATA, H. (**1985**)
Lead Isotope Analysis of Ancient Copper and Base Metal Ore Deposits in Western India. IN: *Application of Science in Examination of Works of Art. Proceedings of the Seminar: September 7-9, 1983.*, (England, P.A. & van Zelst, L., eds): 207-212. Boston: Museum of Fine Arts.

GALE, N.H. (**1978**)
Lead isotopes and Aegean metallurgy. IN: *Thera and the Aegean World Vol. I.*, (Doumas, C., ed.): 529-545. London.

GALE, N.H. (**1979**)
Some Aspects of Lead and Silver Mining in the Aegean. *Miscellanea Graeca* **2**: 8-60.

GALE, N.H. (**1989**)
Archaeometallurgical Studies of Late Bronze Age Ox-hide Copper Ingots from the Mediterranean Region. IN: *Archäometallurgie der Alten Welt / Old World Archaeometallurgy. Proceedings Symposium Heidelberg 1987.*, (Hauptmann, A., Pernicka, E. & Wagner, G.A., eds): 247-268. Der Anschnitt, Beiheft **7**. Bochum: Deutsches Bergbau-Museum.

GALE, N.H. & SPOONER, E.T.C. (**1982**)
The lead and strontium isotope geochemistry of metalliferous sediments associated with Upper Cretaceous ophiolitic rocks in Cyprus, Syria, and the Sultanate of Oman: Reply. *Canadian Journal of Earth Sciences* **19**: 1724-1726.

GALE, N.H. & SPOONER, E.T.C. & POTTS, P.J. (**1981**)
The lead and strontium isotope geochemistry of metalliferous sediments associated with Upper Cretaceous ophiolitic rocks in Cyprus, Syria, and the Sultanate of Oman. *Canadian Journal of Earth Sciences* **18**: 1290-1302.

GALE, N.H. & STOS-GALE, Z.A. (**1981**)
Cycladic Lead and Silver Metallurgy. *Annual of the British School at Athens* **76**: 169-224.

GALE, N.H. & STOS-GALE, Z.A. (**1987**)
Oxhide ingots from Sardinia, Crete and Cyprus and the Bronze Age copper trade: new scientific evidence. IN: *Studies in Sardinian Archaeology III. Nuragic Sardinia and the Mycenaean World.*, (Balmuth, M.S., ed.): 159-198. BAR International Series **387**. Oxford: Archaeopress.

GALE, N.H., GENTNER, W. & WAGNER, G.A. (**1980**)
Mineralogical and Geological Silver Sources of Archaic Greek Coinage. IN: *Metallurgy in Numismatics, Volume 1.*, (Metcalf, D.M. & Oddy, W.A., eds): 3-49. Royal Numismatic Society, Special Publication **13**. London: Royal Numismatic Society.

GALE, N.H., PICARD, O. & BARRANDON, J.N. (**1988**)
The archaic Thasian silver coinage. IN: *Antike Edel- und Buntmetallgewinnung auf Thasos.*, (Wagner, G.A. & Weisgerber, G., eds): 212-222. Der Anschnitt, Beiheft **6**. Bochum: Deutschen Bergbau-Museums.

GALE, N.H., STOS-GALE, Z., RADUNCHEVA, A., PANAYOTOV, I., IVANOV, I., LILOV, P. & TODOROV, T. (**2003**)
Early Metallurgy in Bulgaria IN: *Mining and Metal Production through the Ages.*, (Craddock, P.T. & Lang, J., eds): 122-173. London: British Museum Press.

GALE, N.H., STOS-GALE, Z.A. & DAVIS, J.L. (**1984**)
The Provenance of Lead Used at Ayia Irini, Keos. *Hesperia* **53**(4): 389-406.

GALE, N.H., STOS-GALE, Z.A., MALIOTIS, G. & ANNETTS, N. (**1997**)
Lead isotope data from the Isotrace Laboratory, Oxford: Archaeometry data base 4, ores from Cyprus. *Archaeometry* **39**(1): 237-246.

GRAESER, S. & FRIEDRICH, G. (**1970**)
Zur Frage der Altersstellung und Genese der Blei-Zink-Vorkommen der Sierra de Cartegena in Spanien. *Mineralium Deposita* **5**: 365-374.

GRÖGLER, N., GEISS, J., GRÜNENFELDER, M. & HOUTERMANS, F.G. (**1966**)
Isotopenuntersuchungen zur Bestimmung der Herkunft römischer Bleirohre und Bleibarren. *Zietschrift für Naturforschung* **21a**(7): 1167-1172.

HAMELIN, B., DUPRÉ, B. & ALLÈGRE, C.J. (**1984**)
The lead isotope systematics of ophiolite complexes. *Earth and Planetary Science Letters* **67**: 351-366.

HAMELIN, B., DUPRÉ, B., BRÉVART, O. & ALLÈGRE, C.J. (**1988**)
Metallogenesis at Paleo-spreading centers: lead isotopes in sulfides, rocks and sediments from the Troodos ophiolite (Cyprus). *Chemical Geology* **68**: 229-238.

HAUPTMANN, A., BEGEMANN, F., HEITKEMPER, E., PERNICKA, E. & SCHMITT-STRECHER, S. (**1992**)
Early Copper Produced at Feinan, Wadi Araba, Jordan: The Composition of Ores and Copper. *Archeomaterials* **6**(1): 1-33.

HAUPTMANN, A., SCHMITT-STRECKER, S., BEGEMANN, F. & PALMIERI, A. (**2002**)
Chemical Composition and Lead Isotopy of Metal Objects from the "Royal" Tomb and Other Related Finds at Arslantepe, Eastern Anatolia. *Paléorient* **28**(2): 43-70.

HEGDE, K.T.M. & ERICSON, J.E. (**1996**)
Ancient Indian copper smelting furnaces. IN: *Furnaces and Smelting Technology in Antiquity.*, (Craddock, P.T. & Hughes, M.J., eds): 59-69. British Museum Occasional Paper **48**. London: British Museum Press.

INGO, G.M., AGUS, T., RUGGERI, AMORE BONAPASTA, A., R., BULTRINI, G. & CHIOZZINI, G. (**1997**)
Lead and silver production in the Montevecchio basin (western Sardinia). IN: *Materials Issues in Art and Archaeology V.*, (Vandiver, P.B., Druzik, J.R., Merkel, J.F. & Stewart, J.): 411-416. Materials Research Society Symposium Proceedings **462**. Pittsburgh - Pennsylvania: Materials Research Society.

JOEL, E.C., TAYLOR, J.J., IXER, R.A. & GOODWAY, M. (**1997**)
Lead Isotope Analysis and the Great Orme Mine. IN: *Archaeological Sciences 1995. Proceedings of a conference on the application of scientific techniques to the study of archaeology.*, (Sinclair, A., Salter, E. & Gowlett, J., eds): 123-131. Oxbow Monographs **64**. Oxford: Oxbow books.

KLEIN, S., LAHAYE, Y., BREY, G.P. & VON KAENEL, H.-M. (**2004**)
The early Roman imperial aes coinage II: tracing the copper sources by analysis of lead and copper isotopes - copper coins of Augustus and Tiberius. *Archaeometry* **46**(3): 469-480.

MABUCHI, H., HIRAO, Y. & NISHIDA, M. (**1985**)
Lead isotope approach to the understanding of early Japanese bronze culture. *Archaeometry* **27**(2): 131-159.

MCGEEHAN-LIRITZIS, V. & GALE, N.H. (**1999**)
Chemical and lead isotope analyses of Greek Late Neolithic and Early Bronze Age metals. *Archaeometry* **30**(2): 199-225.

ORTIZ, M.A.H. (**2003**)

Prehistoric Mining and Metallurgy in South West Iberian Peninsula. BAR International Series **1188**. Oxford: Archaeopress.

PERNICKA, E., BEGEMANN, F., SCHMITT-STRECKER, S. & GRIMANIS, A.P. (**1990**)
On the composition and provenance of metal artefacts from Poliochni on Lemnos. *Oxford Journal of Archaeology* **9**(3): 263-298.

PERNICKA, E., REHREN, TH. & SCHMITT-STRECKER, S. (**1998**)
Late Uruk silver production by cupellation at Habuba Kabira, Syria. IN: *Metallurgica antiqua: in honour of Hans-Gert Bachmann and Robert Maddin.*, (Rehren, Th., Hauptmann, A. & Muhly, J., eds): 123-134. Der Anschnitt, Beiheft **8**. Bochum: Deutsches Bergbau-Museum.

PERNICKA, E., SEELIGER, T.C., WAGNER, G.A., BEGEMANN, F., SCHMITT-STRECKER, S., EIBNER, C., ÖZTUNALI, Ö. & BARANYI, I. (**1984**)
Archäometallurgische Untersuchungen in Nordwestanatolien. *Jahrbuch des Römisch-Germanisches Zentralmuseums Mainz* **31**: 533-599.

PONTING, M.J. (**2003**)
From Damascus to Denia: scientific analysis of three groups of Fatimid period metalwork. *Historical Metallury* **37**(2): 85-105.

PONTING, M., EVANS, J.A. & PASHLEY, V. (**2003**)
Fingerprinting of Roman mints using laser ablation MC-ICP-MS lead isotope analysis. *Archaeometry* **45**(4): 591-597.

PRANGE, M.K., GÖTZE, H.-J., HAUPTMANN, A. & WEISGERBER, G. (**1999**)
Is Oman the Ancient Magan? Analytical Studies of Copper from Oman. IN: *Metals in Antiquity.*, (Young, S.M.M, Pollard, A.M., Budd, P. & Ixer, R.A., eds): 187-192. BAR International Series **792**. Oxford: Archaeopress.

ROHL, B.M. (**1996**)
Lead isotope data from Isotrace Laboratory, Oxford: Archaeometry data base 2, galena from Britain and Ireland. Archaeometry 38(1): 165-180.

ROHL, B. & NEEDHAM, S.P. (**1998**)
The Circulation of Metals in the British Bronze Age / The Application of Lead Isotope Analysis. British Museum Occasional Paper **102**. London: British Museum Press.

SANTOS ZALDUEGUI, J.F., GARCIA DE MADINABEITIA, S., GIL IBARGUCHI, J.I. & PALERO, F. (**2004**)
A lead isotope database: the Los Pedroches - Alcudia Area (Spain); implications for archaeometallurgical connections across southwestern and southeastern Iberia. *Archaeometry* **46**(4): 628-630.

SAYRE, E.V., JOEL, E.C., BLACKMAN, M.J., YENER, K.A. & ÖZBAL, H. (**2001**)
Stable lead isotope studies of Black Sea anatolian ore sources and related Bronze Age and Phrygian artefacts from nearby archaeological sites. *Archaeometry* **43**(1): 77-115.

SAYRE, E.V., YENER, K.A., JOEL, E.C. & BARNES, I.L. (**1992**)
Statistical Evaluation of the Presently Accumulated Lead Isotope Data from Anatolia and Surrounding Regions. *Archaeometry* **34**(1): 73-105.

SEELIGER, T.C., PERNICKA, E., WAGNER, G.A., BEGEMANN, F., SCHMITT-STECKER, S., EIBNER, C. ,OZTUNALI, O. & BARANYI, I. (**1985**)
Archäometallurgische Untersuchungen in Nord- und Ostanatolien. *Jahrbuch des Romisch-Germanisches Zentralmuseums Mainz* **32**: 597-659.

SEGAL, I., HALICZ, L. & KAMENSKI, A. (**2002**)
The Metallurgical Remains from Ashqelon-Afridar, Israel, and their source. IN: *Archaeometry '98: Proceedings of the 31st Symposium, Budapest, April 26 - May 3 1998. Volume II.*, (Jerem, E. & Biró, K.T., eds): 461-473. BAR International Series [Archaeolingua Central European Series 1] **1043**(II). Oxford: Archaeopress.

SHAHABPOUR, J. & KRAMERS, J.D. (**1987**)
Lead isotope data from the Sar-Cheshmeh porphyry copper deposit, Iran. *Mineralium Deposita* **22**(4): 278-281.
SPOONER, E.T.C. & GALE, N.H. (**1982**)

The lead and strontium isotope geochemistry of metalliferous sediments associated with Upper Cretaceous ophiolitic rocks in Cyprus, Syria, and the Sultanate of Oman: Discussion. *Canadian Journal of Earth Sciences* **19**: 1715-1719.

SRINIVASAN, S. (**1999**)
Lead isotope and trace element analysis in the study of over one hundred South Indian metal icons. *Archaeometry* **41**(1): 91-116.

STACEY, J.S., DOE, B.R., ROBERTS, R.J., DELEVAUX, M.H. & GRAMLICH, J.W. (**1980**)
A Lead Isotope Study of Mineralization in the Saudi Arabian Shield. *Contributions to Mineralogy and Petrology* **74**: 175-188.

STOS-GALE, Z.A. (**1989**)
Cycladic Copper Metallurgy. IN: *Archäometallurgie der Alten Welt / Old World Archaeometallurgy. Proceedings Symposium Heidelberg 1987.*, (Hauptmann, A., Pernicka, E. & Wagner, G.A., eds): 279-291. Der Anschnitt, Beiheft **7**. Bochum: Deutsches Bergbau-Museum.

STOS-GALE, Z.A. (**2001**)
The Impact of the Natural Sciences o,n Studies of Hacksilver and Early Silver Coinage. IN: *Hacksilber to coinage: New Insights into the Monetary History of the Near East and Greece. A Collection of Eight Papers Presented at the 99th Annual Meeting of the Archaeological Institute of America.*, (Balmuth, M.S., ed.): 53-76. Numismatic Studies **24**. New York: The American Numismatic Society.

STOS-GALE, Z.A. & GALE, N.H. (**1982**)
The sources of Mycenaean silver and lead. *Journal of Field Archaeology* **9**: 467-485.

STOS-GALE, Z.A., GALE, N.H. & ANNETTS, N. (**1996**)
Lead isotope data from the Isotrace Laboratory, Oxford: Archaeometry data base 3, ores from the Aegean, part 1. *Archaeometry* **38**(2): 381-390.

STOS-GALE, Z.A., GALE, N.H. & ZWICKER, U. (**1986**)
The copper trade in the South-East Mediterranean region. Preliminary scientific evidence. *Report of the Department of Antiquities, Cyprus, 1986*: 122-144. Nicosia: The Department of Antiquities, Cyprus.

STOS-GALE, Z.A., GALE, N.H., ANNETTES, N., TODOROV, T., LILOV, P., RADUNCHEVA, A. & PANAYOTOV, I. (**1998**)
Lead isotope data from the Isotrace Laboratory, Oxford: Archaeometry data base 5, ores from Bulgaria. *Archaeometry* **40**(1): 217-226.

STOS-GALE, Z.A., GALE, N.H., HOUGHTON, J. & SPEAKMAN, R. (**1995**)
Lead isotope data from the Isotrace Laboratory, Oxford: Archaeometry data base 1, ores from the Western Mediterranean. *Archaeometry* **37**(2): 407-415.

STOS-GALE, Z.A., MALIOTIS, G., GALE, N.H. & ANNETTS, N. (**1997**)
Lead isotope characteristics of the Cyprus copper ore deposits applied to provenance studies of copper oxhide ingots. *Archaeometry* **39**(1): 83-123.

THORPE, R.I. (**1982**)
The lead and strontium isotope geochemistry of metalliferous sediments associated with Upper Cretaceous ophiolitic rocks in Cyprus, Syria, and the Sultanate of Oman: Discussion. *Canadian Journal of Earth Sciences* **19**: 1710-1715.

TRINCHERINI, P.R., BARBERO, P., QUARATI, P., DOMERGUE, C. & LONG, L. (**2001**)
Where do the lead ingots of Saintes-Maries-De-La-Mer wreck come from? Archaeology compared with physics. *Archaeometry* **43**(3): 393-406.

VAVELIDIS, M., BASSIAKOS, I., BEGEMANN, F., PATRIARCHEAS, K., PERNICKA, E., SCHMITT-STRECKER, S. & WAGNER, G.A. (**1985**)
Geologie und Erzvorkommen. IN: *Silber, Blei und Gold auf Sifnos. Prähistorische und antike Metallproduktion.*, (Wagner, G.A. & Weisgerber, G., eds): 59-80. Der Anschnitt, Beiheft **3**. Bochum: Deutsches Bergbau-Museum.

WAGNER, G.A., GENTNER, N., GROPENGIESSER, H. & GALE, N.H. (**1980**)

Early Bronze Age lead-silver mining and metallurgy in the Aegean: The ancient workings on Siphnos. IN: *Scientific Studies in Early Mining and Extractive Metallurgy.*, (Craddock, P.T., ed.): 63-80. British Museum Occasional Paper **20**. London: British Museum.

WAGNER, G.A., PERNICKA, E., SEELIGER, T.C., LORENZ, I.B., BEGEMANN, F., SCHMITT-STRECKER, S., EIBNER, C. & OZTUNALI, O. (**1986**)
Geochemische und isotopisch Charakteristika früher Rohstoffquellen für Kupfer, Blei, Silber und Gold in der Türkei. *Jahrbuch des Römisch-Germanisches Zentralmuseums Mainz* **33**(2): 723-752.

WEEKS, L. (**1999**)
Lead Isotope Analyses from Tell Abraq, United Arab Emirates: New Data regarding the "Tin Problem in Western Asia". *Antiquity* **73**(279): 49-64.

WEEKS, L.R. (**2000**)
Pre-Islamic Metallurgy of the Gulf. Sydney: Unpublished Ph.D. Thesis - Near Eastern Archaeology, University of Sydney.

WEEKS, L.R. (**2004**)
Early Metallurgy of the Persian Gulf. Technology, Trade, and the Bronze Age World. American School of Prehistoric Research Monograph Series. Boston - Leiden: Brill Academic Publishers, Inc.

WOLF, S., STOS, S., MASON, R. & TITE, M.S. (**2003**)
Lead Isotope Analyses of Islamic Pottery Glazes from Fustat, Egypte. *Archaeometry* **45**(3): 405-420.

YENER, K.A., SAYRE, E.V., JOEL, E.C., ÖZBAL, H., BARNES, I.L. & BRILL, R.H. (**1991**)
Stable Lead Isotope Studies of Central Taurus Ore Sources and Related Artifacts from Eastern Mediterranean Chalcolithic and Bronze Age Sites. *Journal of Archaeological Science* **18**: 541-577.

ZWICKER, U. (**1989**)
Untersuchungen zur Herstellung von Kupfer und Kupferlegierungen im Bereich des östlichen Mittelmeeres (3500-1000 v.Chr.). IN: *Archäometallurgie der Alten Welt / Old World Archaeometallurgy. Proceedings Symposium Heidelberg 1987.*, (Hauptmann, A., Pernicka, E. & Wagner, G.A., eds): 191-201. Der Anschnitt, Beiheft **7**. Bochum: Deutsches Bergbau-Museum.

Appendix 3: PIXE analytical data on ed-Dur material published by L. Weeks[1]

- **Sample description & contextual data**

Reg. Nr.	Area	UF	Sq	Loc.	Object
sAT 365	AT	4261	I	G 5153	Flat fragment
sAT 374	AT	4263	I	G 5154	Flat fragment
sAV 410	AV	4270	II 1 - 2	G 5156	Flat fragment
sAV 626	AV	-	-	G 5156	Flat fragment
sBC 593	BC	5606	-	G 6082	Flat fragment
sBI 624	BI	5723	-	G 6103	Flat fragment
sBM 680	BM	5775	III 2	G 6130	Flat fragment
sBO 705	BO	5900	II 3	G 6154	Flat fragment
sBO 1127	BO	Dump	-	-	Curved fragment
sBQ 1005	BQ	5982	A 4	G 6276	Pin/awl
sBQ 856	BQ	5969	I 2	G 6270	Flat fragment
sBQ 865	BQ	5968	I 2	G 6272	Ring
sBQ 899	BQ	5971	A 3	G 6272	Rough lump
sBQ 992	BQ	5981	A 4	G 6275	Flat fragment
sBR 1046	BR	6065	I 3	-	Folded sheet
sBR 1176	BR	Dump	-	-	Horse bit (?)
sBR 776	BR	5825	II 1	G 6219	Flat fragment
sBR 836	BR	6019	II 4	G 6307	Flat fragment
sBR 970	BR	6041	IV 3	G 6311	Curved flat fragment
sBS 1147	BS	Dump	-	-	Chisel/nail (?)
sBS 1151	BS	6558	IV 3	G 7041	Flat fragment
sBS 1181	BS	6533	V 4	-	Chisel/nail (?)
sBS 1244	BS	6620	VI 4	-	Flat fragment
sBS 1326	BS	6641	VII 3	-	Flat fragment
sBS 1487	BS	6727	X 5	-	Chisel/nail (?)
sBS 1488	BS	6723 (dump)	X 5	-	Folded sheet
sN 152	N	2406	V 6	G 3831	Flat fragment
sN 253	N	2419	IV 6	G 3836	Flat fragment
sN 255	N	2405	IV - V 6	G 3831	Flat fragment
sN 290	N	2427	IV 5 - 6	G 3839	Flat fragment
sN 316	N	2407	V 6	G 3832	Flat fragment
sN 317	N	2405	V 6	G 3831	Flat fragment
sN 344	N	4255	IV 7	G 3849	Flat fragment

Co-ordinates of the samples analysed by L. Weeks.

[1] Weeks, 2004a.

• Analytical data samples

Reg. Nr.	S	Fe	Co	Ni	Cu	Zn	As	Se	Ag	Sn	Pb
sAT 365	0,25	1,06	0,04	0,66	69,7	7,70	0,10	-	1200	19,0	1,34
sAT 374	-	0,88	0,05	0,40	80,4	5,24	0,09	-	1250	12,3	0,51
sAV 410	0,17	0,24	0,12	0,21	63,8	0,18	0,40	-	450	33,8	0,94
sAV 626	0,28	0,19	0,11	0,08	78,0	0,17	0,28	-	750	20,1	0,67
sBC 593	-	0,36	0,02	-	89,3	10,0	0,05	50	350	0,05	0,09
sBI 624	-	0,17	0,03	-	74,9	0,11	-	-	500	24,4	0,27
sBM 680	0,15	0,40	0,02	-	98,8	0,15	-	-	-	0,33	0,11
sBO 705	0,38	0,87	0,05	0,09	81,1	0,69	0,08	-	1450	15,4	1,20
sBQ 1005	0,29	0,93	0,03	-	98,2	0,09	0,05	80	950	-	0,21
sBO 1127	1,06	1,77	0,07	0,02	78,7	0,13	0,15	-	450	13,1	4,94
sBQ 856	0,64	0,97	0,08	0,02	89,8	0,09	0,14	-	850	6,43	1,76
sBQ 865	0,83	0,40	0,07	0,06	86,6	0,09	0,06	-	900	7,74	4,03
sBQ 899	0,80	1,08	0,08	-	60,7	0,16	0,28	-	1100	35,9	0,88
sBQ 992	0,49	0,42	0,04	-	97,8	0,11	-	50	300	1,09	0,02
sBR 1046	0,54	0,19	0,05	0,05	89,1	0,12	0,04	-	650	5,49	4,34
sBR 1176	2,95	0,15	0,02	0,01	66,5	0,19		-	700	9,52	20,6
sBR 776	1,40	0,26	0,03	0,01	88,0	0,11	0,07	-	650	9,73	0,37
sBR 836	-	0,15	0,03	-	99,6	0,09	-	-	-	0,05	0,11
sBR 970	0,51	0,23	0,04	-	91,5	0,29	0,02	-	-	7,36	0,08
sBS 1147	-	0,29	0,16	-	98,9	0,09	0,40	60	250	0,04	0,06
sBS 1151	2,67	0,07	0,01	-	73,0	0,12	-	-	600	11,4	12,6
sBS 1181	0,14	0,04	0,12	1,27	97,6	0,08	0,33	160	100	0,31	0,04
sBS 1244	-	0,32	0,03	-	99,1	0,09	0,04	80	300	0,21	0,12
sBS 1326	4,23	0,04	0,03	0,05	71,3	0,13	0,04	-	1100	7,82	16,3
sBS 1487	2,22	0,05	0,01	-	69,8	0,14	-	-	600	6,62	21,1
sBS 1488	-	0,98	0,03	0,17	91,2	0,94	0,03	-	450	5,89	0,70
sN 152	0,60	0,99	0,10	0,18	67,5	0,16	0,52	-	1800	25,5	4,26
sN 253	0,38	0,59	0,04	0,16	78,2	0,36	0,08	-	300	18,1	1,97
sN 255	-	1,14	0,09	1,11	82,8	13,7	0,04	-	-	1,08	0,13
sN 290	0,35	0,64	0,04	-	98,5	0,13	-	-	-	0,04	0,32
sN 316	-	0,01	0,03	-	99,3	0,10	0,02	-	-	0,41	0,14
sN 317	0,59	0,34	0,07	0,19	75,8	0,13	0,28	-	650	18,8	3,66
sN 344	0,28	0,43	0,09	0,37	49,5	0,09	0,51	-	1450	47,1	1,54

PIXE compositional data as published by L. Weeks (2004a: 242). The amounts are expressed in *weight %*, except for Se and Ag which are given in *ppm.*

Appendix 4: EDX measurements on standards

- **Standards**

Standard values		Measurement at x100 magnification	Measurement at x200 magnification	Measurement at x200 magnification	Measurement at x500 magnification	Average	Standard value *minus* average	Standard deviation 2 sigma
Cu	58,5	54,8	55,4	55,6	55,7	55,4 ± *0,7*	3,1	0,8
Zn	39,8	42,0	42,0	41,6	41,7	41,8 ± *0,7*	-2,0	0,4
Pb	1,13	2,5	2,2	2,4	2,1	2,3 ± *0,5*	-1,2	0,4
Fe	0,32	-	-	-	-	-	0,3	0,0
Cu	89,93	89,5	89,3	89,1	89,5	89,4 ± *1,0*	0,6	0,4
Zn	9,94	10,5	10,7	10,9	10,5	10,7 ± *0,4*	-0,7	0,4
Cu	98,41	96,7	97,5	96,8	96,9	97,0 ± *1,0*	-1,4	0,7
Sn	0,43	0,3	0,3	0,3	0,4	0,3 ± *0,1*	0,1	0,1
Pb	0,73	3,0	2,2	2,8	2,7	2,7 ± *0,5*	-1,9	0,7
Cu	87,44	84,1	84,6	84,5	-	84,4 ± *1,1*	3,0	0,5
Zn	4,23	4,7	4,3	4,8	-	4,6 ± *0,3*	-0,4	0,5
Sn	7,46	9,2	9,7	9,2	-	9,4 ± *0,3*	-1,9	0,6
Pb	0,26	1,8	1,3	1,5	-	1,5 ± *0,5*	-1,3	0,5
Fe	0,48	0,1	0,1	0,1	-	0,1 ± *0,1*	0,4	0,0
Cu	93,8	93,1	92,8	92,2	92,6	92,7 ± *1,0*	1,1	0,8
Sn	5,99	5,7	5,8	6,0	5,9	5,9 ± *0,2*	0,1	0,3
Pb	0,4	1,2	1,4	1,7	1,5	1,5 ± *0,4*	-1,1	0,4
							0,0	
Cu	60,44	60,7	60,7	59,3	60,6	60,3 ± *0,8*	0,1	1,4
Zn	38,84	36,4	37,1	38,0	36,9	37,1 ± *0,7*	1,7	1,3
Pb	0,58	2,9	2,3	2,7	2,5	2,6 ± *0,6*	-2,0	0,5
Cu	68,09	66,8	67,2	66,6	66,1	66,7 ± *0,8*	1,4	0,9
Zn	30,76	31,6	30,6	31,4	31,3	31,2 ± *0,7*	-0,4	0,8
Pb	0,48	1,6	2,1	2,0	2,6	2,1 ± *0,5*	-1,6	0,8
Fe	0,5	-	-	-	-	-	0,5	-
Zn	98,95	98,5	98,0	98,0	98,6	98,3 ± *1,2*	0,7	0,6
Pb	1,03	1,5	2,0	2,0	1,4	1,7 ± *0,5*	-0,7	0,6
Cu	88,13	85,7	85,8	84,4	84,2	85,0 ± *0,9*	3,1	1,7
Zn	4,36	4,4	4,5	4,0	5,1	4,5 ± *0,2*	-0,1	0,9
Sn	6,65	7,9	8,0	9,3	9,2	8,6 ± *0,3*	-1,9	1,5
Pb	0,3	1,8	1,6	2,2	1,4	1,7 ± *0,4*	-1,4	0,7
Fe	0,42	0,2	0,2	0,2	0,2	0,2 ± *0,1*	-0,2	0,1

The values given are in wt%.

• Experimental coins

'Standard' values		Measurement at x100 magnification	Measurement at x200 magnification	Measurement at x200 magnification	Measurement at x500 magnification	Average	Standard value *minus* average	Standard deviation 2 sigma
Alloy 1								
Cu	88,0	89,6	87,6	87,6	88,6	88,4 ± *1,3*	-0,4	1,9
Ag	5,0	5,3	5,4	6,0	5,4	5,5 ± *0,4*	-0,5	0,6
Sn	5,0	3,4	4,3	3,6	3,9	3,8 ± *0,2*	1,2	0,8
Pb	2,0	1,8	2,8	2,8	2,1	2,4 ± *0,7*	-0,4	1,0
Alloy 2								
Cu	89,0	84,9	86,3	88,6	85,9	86,4 ± *1,06*	2,6	3,1
Ag	10,0	12,7	11,7	8,8	11,8	11,3 ± *0,46*	-1,3	3,4
Pb	1,0	2,3	2,0	2,6	2,3	2,3 ± *0,58*	-1,3	0,5
Alloy 3								
Cu	93,0	91,6	92,0	92,4	91,5	91,9 ± *1,4*	1,1	0,8
Sn	5,0	5,7	5,9	5,2	5,8	5,6 ± *0,4*	-0,6	0,6
Pb	2,0	2,7	2,1	2,4	2,7	2,5 ± *0,8*	-0,5	0,6

• Other standards and parallel analyse on samples

The lab of non-ferrous metallurgy provided a piece of bronze that was dissolved in *aqua regia* and subjected to full quantitative analyses by *atomic absorption spectrometry* (AAS).

'Standard' values		Measurement at x100 magnification	Measurement at x200 magnification	Measurement at x500 magnification	Average
Sn	9,20	8,87	8,84	8,69	8,80 ± *0,8*

Reg. nr.	Sn wt% by EDX	Sn wt% by AAS
AW 021-1	15,12	13,0
BQ 016	6,19	5,65
BS 088	9,13	7,55
K 153	13,17	12,5
S 0023	30,55	23,1
Z 146	5,54	5,85

The EDX analyses are in fairly goof accordance, although some samples show significant error.

Appendix 5: Lead isotope ratios of analysed objects

- **Lead isotope ratios of the copper & copper-base alloys**

Copper & copper-base alloys		$^{206}Pb/^{204}Pb$	$^{207}Pb/^{206}Pb$	$^{208}Pb/^{206}Pb$	Alloy
Ring-pommel dagger	**AT 013**	18,412812	0,850424	2,093647	Brass
Large rivet, traces iron blade?	**AV 083**	18,346616	0,853628	2,094850	Brass
Ring-pommel dagger	**AW 063-4**	18,321515	0,853529	2,092615	Brass
Ring-pommel dagger	**BL 014**	18,370930	0,852543	2,089033	Brass
Small bell	**BO 029**	18,581834	0,844188	2,091152	Brass
Altar	**N 138**	18,708693	0,836722	2,076531	Copper
Fragment	**KR 012**	18,533269	0,852034	2,077325	Copper
Coin: tetradrachm XLVII	**BS 169**	18,263049	0,855995	2,095764	Copper
Lion bead	**BK 005**	19,095608	0,837641	2,064173	Copper (leaded)
Bell	**BR 104**	18,036386	0,873071	2,113945	Gunmetal (leaded)
Fragment ladle	**AV 115**	18,640359	0,840946	2,088205	Tin-bronze
Fragment	**KR 010**	17,844875	0,878891	2,129387	Tin-bronze
Fragment	**KR 009**	16,928109	0,929685	2,189859	Tin-bronze
Handle vessel	**C 079**	18,546931	0,844581	2,083798	Tin-bronze (leaded)
Handle vessel	**K 203**	18,295583	0,857038	2,098556	Tin-bronze (leaded)
Female head appliqué	**S 0020**	18,796038	0,835969	2,071641	Tin-bronze (leaded)
Horse appliqué	**AV 104**	18,808976	0,836434	2,070765	Tin-bronze (leaded)
Patera	**AV 005**	18,800202	0,835960	2,073415	Tin-bronze (leaded)
Pedestal statuette	**M 007**	18,628154	0,842813	2,075720	Tin-bronze (leaded)
Bead (with torque)	**N 118**	18,586346	0,843063	2,086849	Tin-bronze (leaded)
Fragment	**KR 011**	17,886408	0,878493	2,127436	Tin-bronze (leaded)
Fragment	**KR 008**	18,314950	0,856363	2,107899	Tin-bronze (leaded)
Fragment	**KR 007**	18,511064	0,851513	2,094554	Tin-bronze (leaded)

- **Lead isotope ratios of the lead objects & litharge**

Lead objects & litharge		$^{206}Pb/^{204}Pb$	$^{207}Pb/^{206}Pb$	$^{208}Pb/^{206}Pb$
Solder	**AF 137**	18,062765	0,862266	2,106415
Lead fragment	**sBK 1238 B**	18,090607	0,868162	2,118841
Lead fragment	**sBM 1225 B**	18,132331	0,868357	2,119526
Lead fragment	**sBM 1225 C**	18,137508	0,863886	2,115443
Lead fragment	**sBM 1225 D**	18,018630	0,869176	2,121278
Lead fragment	**sBS 1139 A**	18,151250	0,866317	2,118963
Lead fragment	**sBS 1360**	18,065807	0,869182	2,119977
Lead fragment	**sBS 1441 A**	18,130176	0,869437	2,120401
Lead fragment	**sBS 1453 A**	18,105590	0,869152	2,121642
Lead fragment	**sBS 1453 B**	18,097401	0,867620	2,118709
Lead fragment	**sBS 1466**	17,108771	0,922963	2,177735
Bullae	**BS 269**	18,703234	0,851576	2,077588
Lead fragment	**ED 1309**	18,081454	0,869982	2,122741
Solder	**M 072**	18,048369	0,868863	2,121385
Lead fragment	**S 0010 C**	18,111857	0,866131	2,115452
'Ingot'	**S 0024**	18,080672	0,868163	2,121320
Large fragment	**Z 019**	16,949497	0,927678	2,184403
Litharge	**AW 013**	18,051580	0,867944	2,121648
Litharge	**sBO 722**	18,098759	0,867834	2,115534
Litharge	**sBO 724 A**	18,056873	0,867141	2,115801

- **Lead isotope ratios of the silver objects (incl. silver coins)**

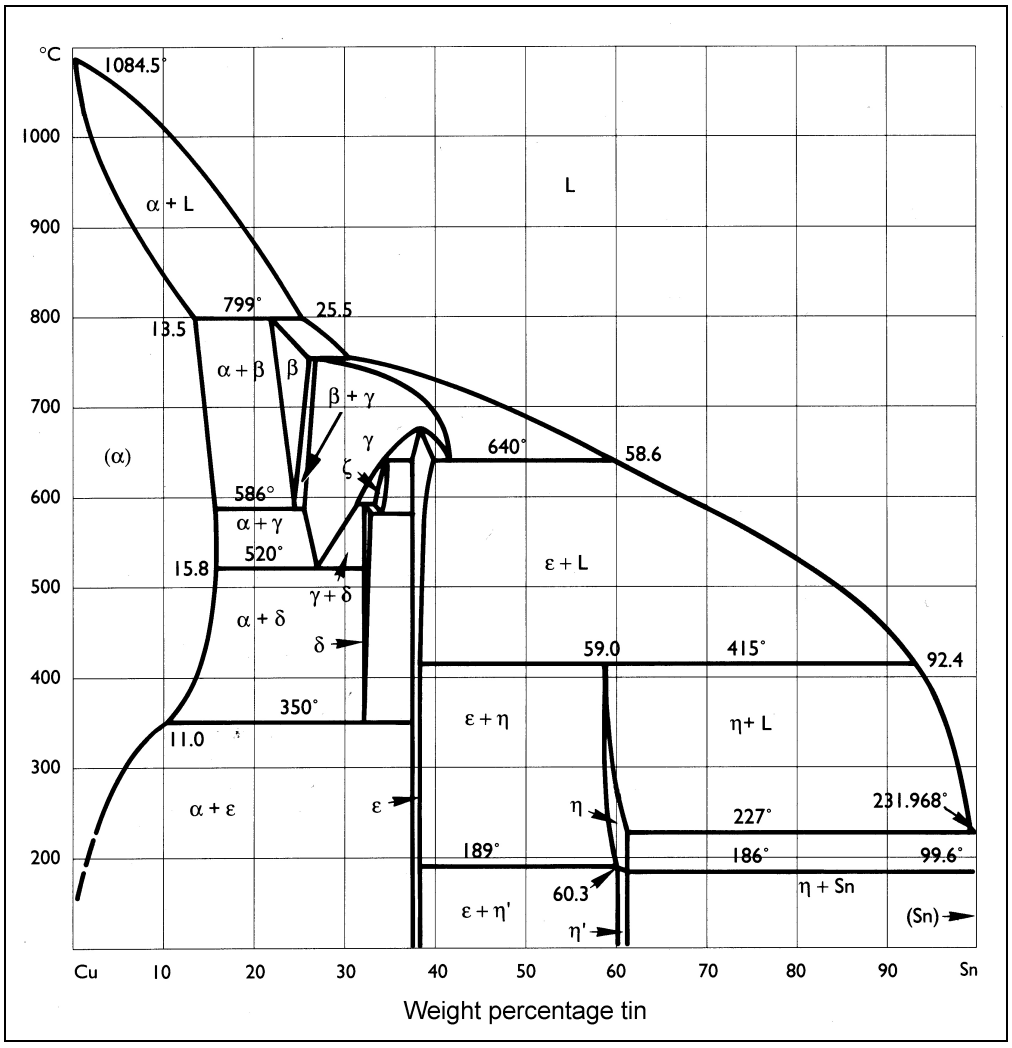
Silver		$^{206}Pb/^{204}Pb$	$^{207}Pb/^{206}Pb$	$^{208}Pb/^{206}Pb$
Coin: obol XLI	**AV 161**	18,410152	0,848739	2,087663
Coin: obol II	**BQ 041**	18,395930	0,853436	2,095222
Coin: obol ?	**BQ 125**	18,322945	0,853250	2,094714
Coin: obol II	**N 036**	18,502624	0,844548	2,074446
Coin: tetradrachm LI?	**AG 003**	18,070333	0,864830	2,110593
Coin: tetradrachm ?	**BQ 136**	18,202409	0,863109	2,106904
Coin: tetradrachm XLVII	**BQ 142**	18,351039	0,855031	2,097871
Small ring	**ED 016**	18,546343	0,840766	2,061424
Bracelet	**F 107**	18,510093	0,846524	2,088565
Bracelet	**F 108**	18,524785	0,846039	2,079095
Ring, finger	**F 113**	18,359412	0,861924	2,109115
Ring, finger	**K 205**	18,644569	0,842985	2,083479
Small twisted wire	**N 301**	18,323755	0,854930	2,102579

- **Lead isotope ratios of the copper-silver alloy coins (not used in this PhD)**

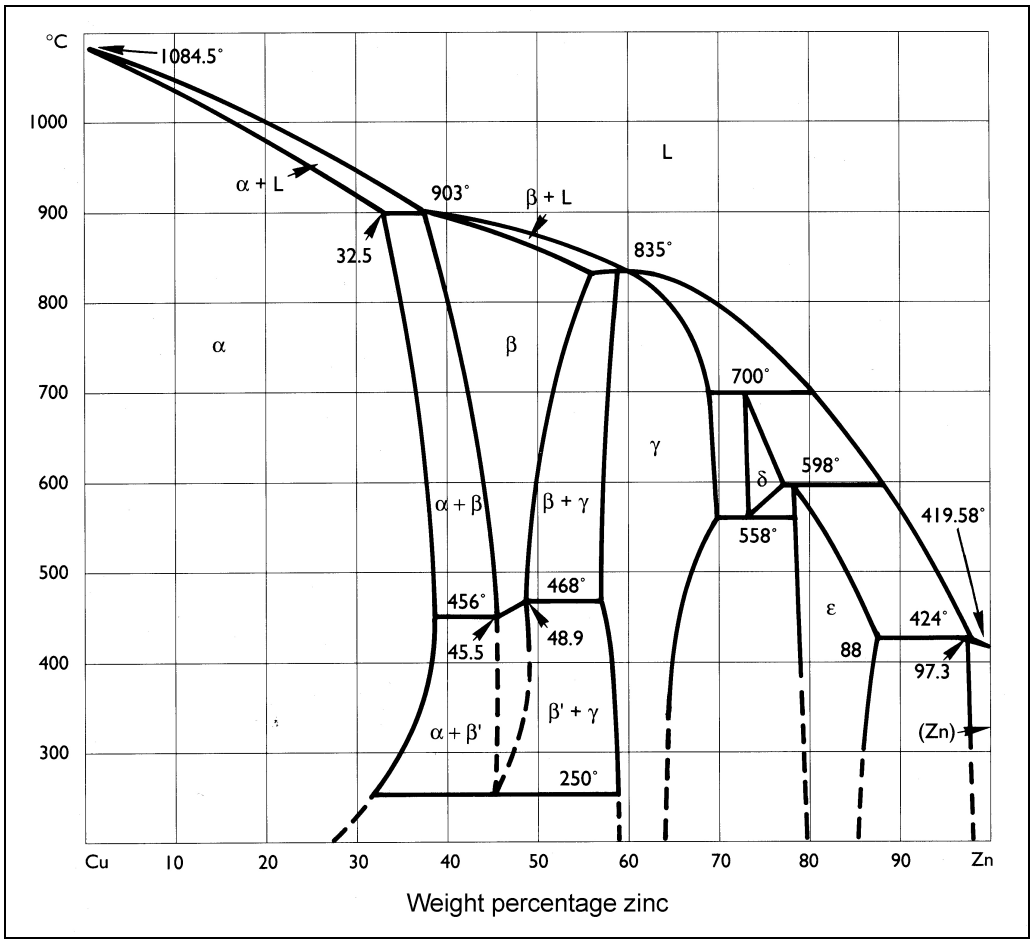
Coins		$^{206}Pb/^{204}Pb$	$^{207}Pb/^{206}Pb$	$^{208}Pb/^{206}Pb$
Coin: tetradrachm XLVIII	**AD 025**	18,339287	0,855396	2,100780
Coin: tetradrachm XLVIIIa	**AV 023**	18,485593	0,847639	2,085823
Coin: tetradrachm XLV	**BM 026**	18,470417	0,846289	2,088567
Coin: tetradrachm XLVII	**BO 043**	18,545768	0,848639	2,088618
Coin: tetradrachm XLVII	**BQ 005**	18,448232	0,849602	2,088239
Coin: tetradrachm XLVIIIb	**BR 106**	18,424409	0,852193	2,095394
Coin: tetradrachm XLVIIIa	**BS 043**	18,311710	0,854514	2,095248
Coin: tetradrachm XLIX	**BS 080**	18,095503	0,863096	2,108113
Coin: tetradrachm XLIX	**BS 097**	18,223490	0,857288	2,092670
Coin: tetradrachm XLVIIIa	**BS 148**	18,321167	0,852073	2,093315
Coin: tetradrachm XLVII	**BS 169**	18,263049	0,855995	2,095764
Coin: tetradrachm XLVIIIa	**BS 172**	18,203396	0,856394	2,097548
Coin: tetradrachm XLVIIIb	**BS 235**	18,409573	0,848101	2,077138
Coin: tetradrachm XLVIIIb	**BS 236**	18,362644	0,857512	2,102088
Coin: tetradrachm XLVIIIb	**BS 237**	18,319134	0,855062	2,094325
Coin: tetradrachm XLV	**BS 254**	18,357028	0,852949	2,087734
Coin: tetradrachm XLV	**BS 284**	18,545616	0,846241	2,082925
Coin: tetradrachm XLVIIIb	**ED 005**	18,310426	0,855333	2,101336
Coin: tetradrachm XLVII	**N 310**	18,446924	0,848621	2,096447

Appendix 6: Phase diagrams

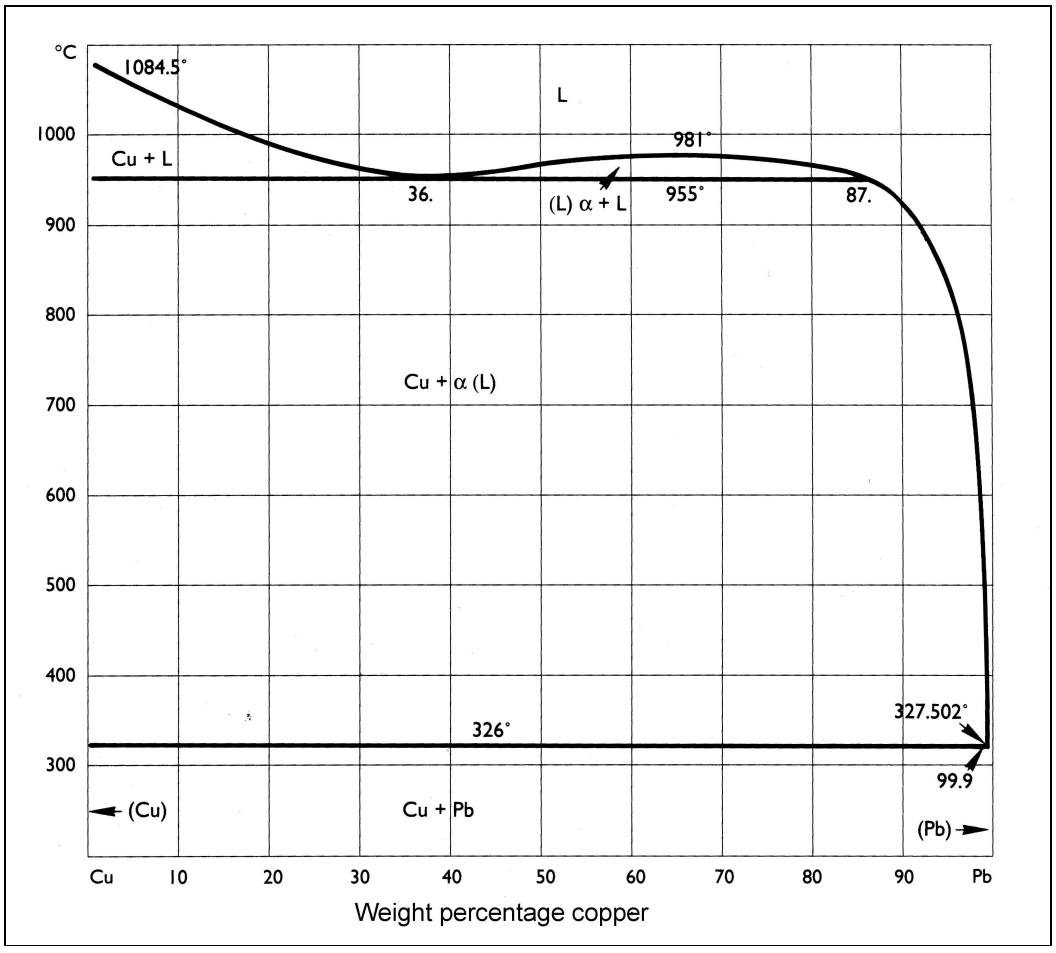
- **Phase diagram bronze (Cu-Sn)**



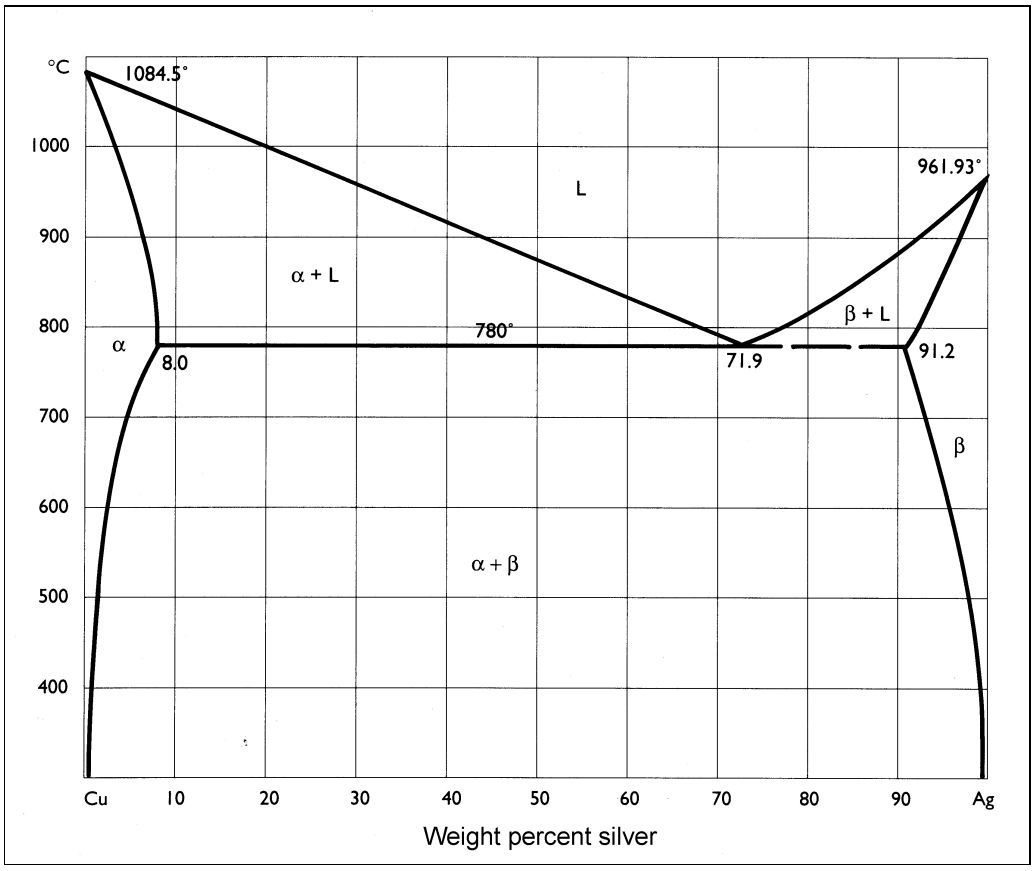
• Phase diagram brass (Cu-Zn)



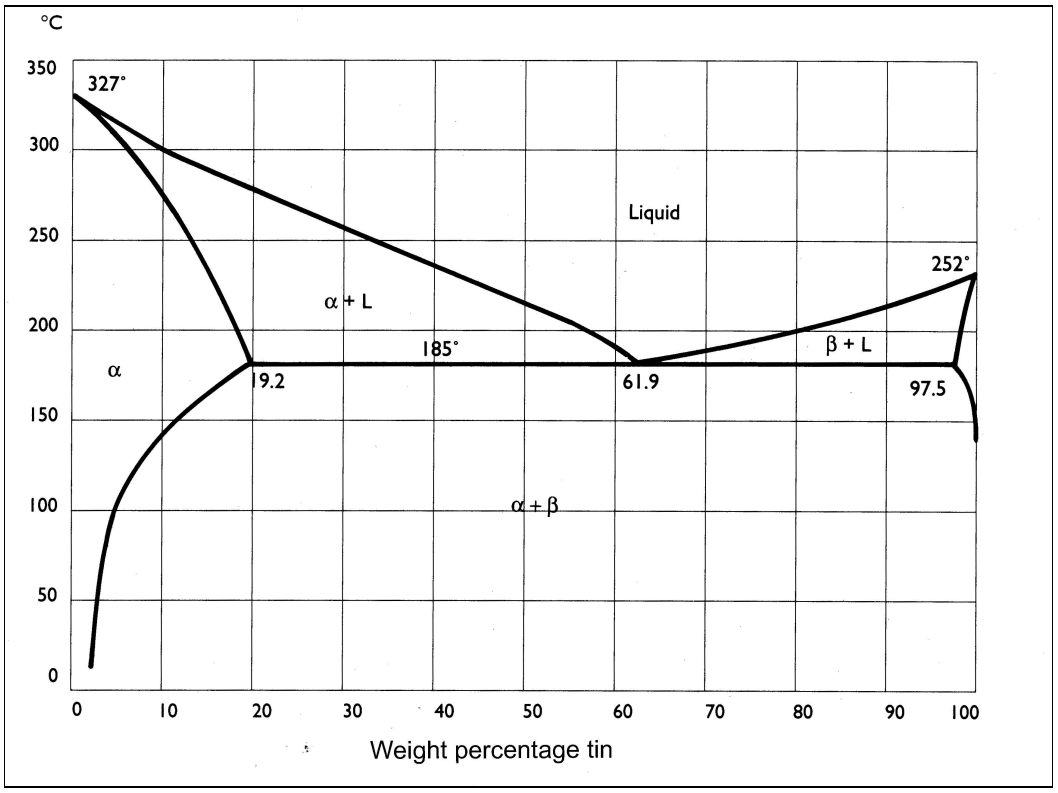
- **Phase diagram leaded copper (Cu-Pb)**



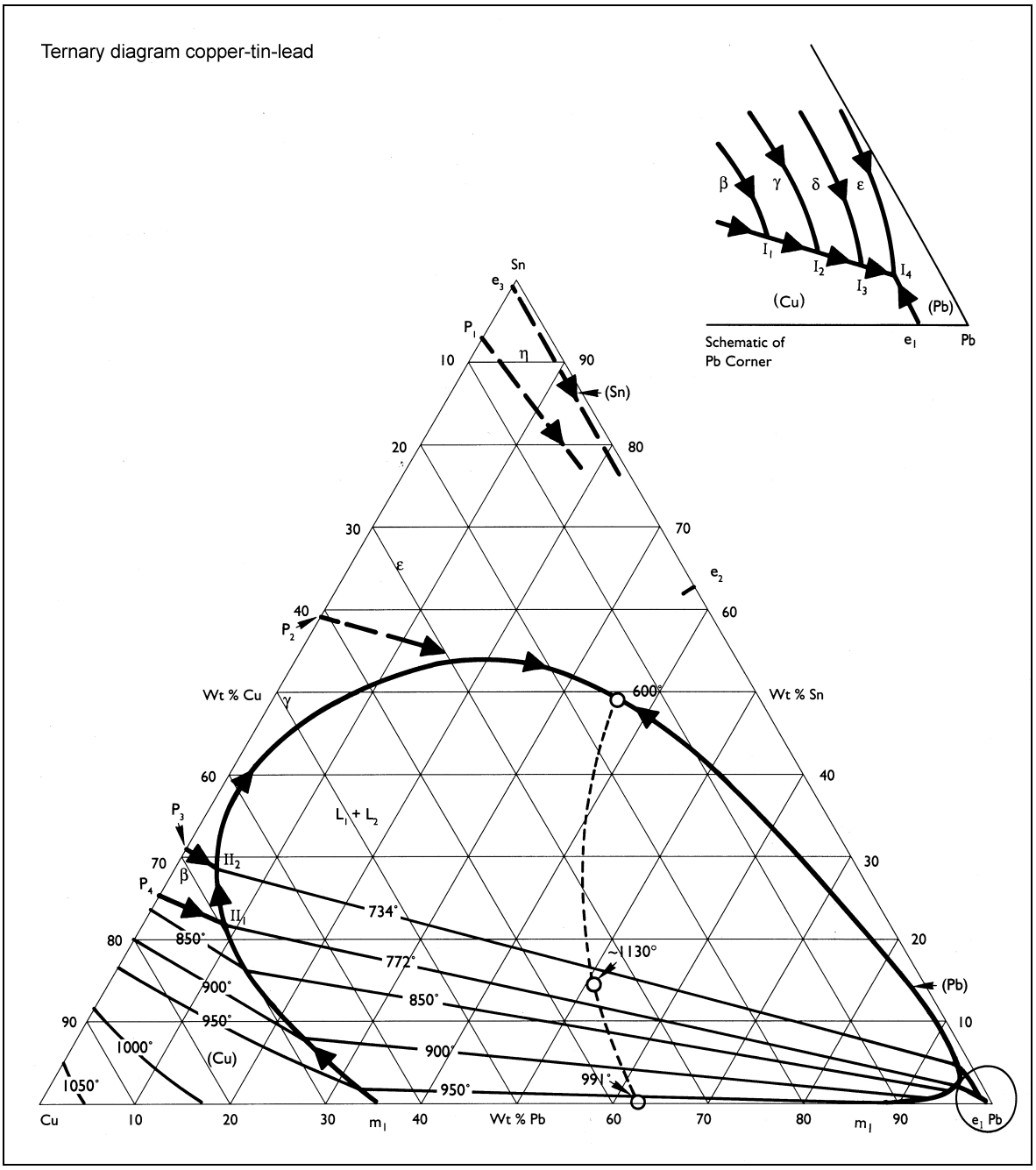
- **Phase diagram brass (Cu-Zn)**



- **Phase diagram pewter (Pb-Sn)**



• Ternary phase diagram leaded bronze (Cu-Sn-Pb)



Appendix 7: Full & restricted EDX dataset copper & copper-base alloys

1. Full EDX-dataset

• Unalloyed copper

Reg. nr.	S	Fe	Ni	Cu	Zn	Ag	Sn	Pb	2-sig S	2-sig Fe	2-sig Ni	2-sig Cu	2-sig Zn	2-sig Ag	2-sig Sn	2-sig Pb
AW 063-1	0,03	0,27	0,07	97,41	0,12	0,27	0,43	1,41	*0,01*	*0,03*	*0,02*	*1,16*	*0,04*	*0,06*	*0,07*	*0,46*
BK 005	-	1,62	0,59	55,00	-	0,50	0,82	41,49	-	*0,14*	*0,10*	*1,22*	-	*0,12*	*0,17*	*3,43*
BQ 153	0,07	0,27	0,12	95,40	-	0,29	0,89	2,95	*0,03*	*0,05*	*0,02*	*0,93*	-	*0,08*	*0,20*	*0,63*
BQ 154	0,17	0,19	-	95,51	-	0,43	0,55	3,13	*0,01*	*0,02*	-	*0,92*	-	*0,06*	*0,07*	*0,51*
BS 302	-	0,36	0,19	96,93	0,05	0,49	0,40	1,59	-	*0,05*	*0,04*	*1,53*	*0,02*	*0,11*	*0,09*	*0,64*
ED 009	0,21	0,17	0,41	96,09	-	0,35	0,40	2,37	*0,01*	*0,02*	*0,02*	*0,64*	-	*0,04*	*0,04*	*0,30*
KR 012	-	0,07	0,14	97,31	0,20	0,43	0,19	1,65	-	*0,02*	*0,03*	*1,20*	*0,06*	*0,08*	*0,05*	*0,52*
M 084	0,07	0,10	0,05	95,54	-	0,47	0,54	3,22	*0,01*	*0,02*	*0,01*	*0,85*	-	*0,05*	*0,05*	*0,51*
N 138	-	0,25	0,21	95,64	0,07	0,48	1,05	2,27	-	*0,04*	*0,04*	*1,28*	*0,03*	*0,09*	*0,14*	*0,82*
S 0012	0,06	0,11	0,02	96,99	-	0,34	0,22	2,26	*0,01*	*0,01*	*0,01*	*0,60*	-	*0,04*	*0,03*	*0,29*
sBJ 1237 A (2)	0,02	0,13	0,06	97,12	-	1,38	0,20	1,09	*0,01*	*0,02*	*0,02*	*1,17*	-	*0,13*	*0,05*	*0,41*
sBO 1275 B	0,73	2,43	0,14	93,54	-	0,26	-	2,90	*0,04*	*0,06*	*0,02*	*0,63*	-	*0,03*	*0,02*	*0,39*
sBR 1157 C	-	0,10	-	97,37	0,31	0,24	0,38	1,59	-	*0,01*	-	*0,68*	*0,04*	*0,03*	*0,04*	*0,44*
sBS 1129 A	0,23	0,11	0,06	97,73	0,11	0,22	0,19	1,35	*0,02*	*0,01*	*0,01*	*0,68*	*0,02*	*0,03*	*0,03*	*0,40*
sBS 1429	0,02	0,13	0,02	96,17	0,81	0,32	0,31	2,22	*0,01*	*0,02*	*0,01*	*0,96*	*0,10*	*0,05*	*0,05*	*0,64*
sM 1250 C	0,14	0,67	-	94,79	-	0,43	0,58	3,38	*0,01*	*0,04*	-	*0,91*	-	*0,06*	*0,07*	*0,57*
sN 1251 A	-	0,23	0,03	96,40	-	0,29	0,75	2,28	-	*0,02*	*0,01*	*0,81*	-	*0,04*	*0,07*	*0,43*
SX 001	0,09	0,13	0,13	95,71	-	0,32	0,96	2,66	*0,01*	*0,03*	*0,01*	*0,83*	-	*0,06*	*0,16*	*0,54*
Z 092	0,31	0,18	-	95,01	-	0,94	0,43	3,13	*0,02*	*0,02*	-	*0,69*	-	*0,06*	*0,04*	*0,38*

• Bronze

Reg. nr.	S	Fe	Ni	Cu	Zn	Ag	Sn	Pb	2-sig S	2-sig Fe	2-sig Ni	2-sig Cu	2-sig Zn	2-sig Ag	2-sig Sn	2-sig Pb
High tin-bronze & leaded high tin-bronze																
AW 021-1	0,13	0,17	0,31	77,69	-	0,20	15,01	6,52	*0,06*	*0,03*	*0,02*	*0,67*	-	*0,08*	*0,52*	*0,91*
KR 011	-	0,27	0,27	69,37	0,13	0,10	16,63	13,23	-	*0,03*	*0,03*	*0,57*	*0,03*	*0,02*	*0,30*	*0,80*
S 0020	0,03	0,12	0,10	59,32	-	0,21	15,19	25,03	*0,01*	*0,02*	*0,02*	*0,50*	-	*0,03*	*0,28*	*1,12*
S 0023	-	0,14	0,20	65,63	0,20	-	30,39	3,44	-	*0,02*	*0,03*	*0,71*	*0,04*	-	*0,52*	*0,71*
sBK 1238 A	-	0,33	0,26	80,79	-	-	16,53	2,05	-	*0,04*	*0,02*	*0,55*	-	-	*0,41*	*0,37*
sBR 1041 C	-	0,24	0,05	60,89	0,48	-	31,89	6,45	-	*0,03*	*0,02*	*0,73*	*0,07*	-	*0,56*	*0,89*
Z 012	-	0,19	0,38	80,36	-	-	15,64	3,39	-	*0,03*	*0,01*	*0,90*	-	-	*0,46*	*0,50*
Medium tin-bronze & leaded medium tin-bronze																
AD 031	0,11	0,22	0,22	84,45	-	0,21	12,50	2,35	*0,02*	*0,03*	*0,01*	*0,69*	-	*0,05*	*0,40*	*0,42*
AV 005	0,04	0,22	0,04	56,74	0,76	0,46	7,43	34,31	*0,01*	*0,04*	*0,01*	*0,77*	*0,10*	*0,08*	*0,37*	*1,97*
AV 007	0,15	0,19	0,26	83,96	-	0,15	12,45	2,89	*0,02*	*0,04*	-	*0,75*	-	*0,06*	*0,49*	*0,47*
AV 016	0,06	0,21	0,22	84,90	-	0,49	10,35	2,96	0,00	*0,07*	*0,01*	*1,11*	-	*0,07*	*0,63*	*0,97*
AV 115	-	0,20	0,20	90,44	-	0,32	6,13	2,74	-	*0,06*	*0,02*	*0,67*	-	*0,05*	*0,38*	*0,40*
BQ 016	0,04	0,18	0,11	91,12	0,12	0,10	6,03	2,30	*0,01*	*0,03*	*0,03*	*1,21*	*0,05*	*0,04*	*0,31*	*0,63*
BQ 070	0,12	0,24	0,22	88,87	-	0,33	7,08	3,20	*0,04*	*0,03*	*0,01*	*0,74*	-	*0,05*	*0,33*	*0,44*
BR 026	-	0,26	0,15	85,37	-	0,12	11,75	2,38	-	*0,03*	*0,01*	*0,70*	-	*0,05*	*0,37*	*0,43*
BS 064	-	0,32	0,23	83,35	-	0,15	10,34	5,71	-	*0,07*	*0,01*	*1,11*	-	*0,07*	*0,63*	*0,97*
C 079	0,02	0,13	0,01	78,58	1,08	0,16	7,21	10,81	*0,00*	*0,02*	*0,01*	*0,66*	*0,09*	*0,03*	*0,31*	*0,82*
K 005	0,13	0,17	0,24	83,05	-	0,31	13,50	2,63	*0,01*	*0,02*	*0,02*	*0,89*	-	*0,08*	*0,47*	*0,46*
K 149	-	0,16	0,22	83,01	-	0,25	14,04	2,40	-	*0,03*	*0,01*	*0,64*	-	*0,04*	*0,45*	*0,37*
K 153	-	0,24	0,21	82,76	-	0,22	13,34	3,26	-	*0,05*	*0,01*	*0,84*	-	*0,06*	*0,51*	*0,54*

Reg. nr.	S	Fe	Ni	Cu	Zn	Ag	Sn	Pb	2-sig S	2-sig Fe	2-sig Ni	2-sig Cu	2-sig Zn	2-sig Ag	2-sig Sn	2-sig Pb
K 203	0,37	0,15	0,67	69,27	-	0,02	10,55	18,98	*0,03*	*0,03*	*0,06*	*0,76*	-	*0,01*	*0,45*	*1,38*
KR 007	-	0,16	0,28	76,62	0,14	0,11	10,68	12,00	-	*0,04*	*0,05*	*1,12*	*0,06*	*0,02*	*0,44*	*1,40*
KR 009	0,04	0,79	0,79	86,98	0,05	0,12	8,45	2,77	*0,18*	*0,07*	*0,07*	*1,08*	*0,02*	*0,04*	*0,35*	*0,63*
M 007	-	0,17	0,07	55,91	0,71	0,35	6,03	36,75	-	*0,03*	*0,02*	*0,80*	*0,10*	*0,07*	*0,35*	*2,12*
N 118	-	0,17	0,18	79,47	-	0,15	6,79	13,24	-	*0,09*	*0,05*	*0,82*	-	*0,00*	*0,38*	*2,26*
N 121	-	0,18	0,30	84,94	-	0,21	11,55	2,79	-	*0,06*	*0,03*	*1,04*	-	*0,11*	*0,74*	*0,82*
N 122	0,1	0,16	0,22	84,77	-	0,36	11,53	2,95	*0,03*	*0,03*	*0,02*	*0,80*	-	*0,05*	*0,36*	*0,49*
S 0021	-	0,24	0,19	89,91	-	0,34	6,73	2,62	-	*0,09*	*0,06*	*1,04*	-	*0,02*	*0,49*	*0,74*
sBQ 1058 A	0,12	0,19	0,25	90,96	-	0,32	5,79	2,43	*0,01*	*0,02*	*0,02*	*0,62*	*0,05*	*0,02*	*0,17*	*0,42*
sBQ 1058 B	0,20	0,22	0,05	87,93	0,28	0,07	9,93	1,34	*0,02*	*0,02*	*0,01*	*0,63*	*0,04*	*0,01*	*0,21*	*0,35*
sBQ 1173 A	-	0,22	0,17	88,54	-	0,27	8,22	2,61	-	*0,03*	*0,01*	*0,74*	-	*0,06*	*0,35*	*0,39*
sBS 1276	0,12	0,19	0,22	91,23	-	0,42	5,03	2,85	*0,01*	*0,03*	*0,02*	*0,92*	*0,16*	*0,04*	*0,25*	*0,60*
sFO 1308 A	0,10	0,17	0,18	89,12	0,14	0,05	8,79	1,45	*0,01*	*0,02*	*0,02*	*0,61*	*0,02*	*0,01*	*0,19*	*0,34*
Z 146	0,32	0,23	0,41	77,41	-	0,32	5,49	15,88	*0,07*	*0,03*	*0,03*	*0,79*	-	*0,08*	*0,37*	*0,91*
Leaded low tin-bronze																
AV 104	-	0,16	0,06	58,21	-	0,23	4,73	36,60	-	*0,02*	*0,01*	*0,57*	-	*0,04*	*0,18*	*1,42*
BS 154	0,31	0,20	0,24	75,95	-	0,23	3,56	19,56	*0,08*	*0,03*	*0,01*	*0,57*	-	*0,10*	*0,24*	*1,23*
KR 008	-	0,12	0,27	78,99	-	0,07	4,44	16,12	-	*0,03*	*0,04*	*1,08*	-	*0,03*	*0,27*	*1,58*
sBR 1157 A	-	0,22	0,20	79,45	-	0,43	4,82	14,89	-	*0,03*	*0,02*	*0,68*	-	*0,09*	*0,28*	*1,29*

- **Brass**

Reg. nr.	S	Fe	Ni	Cu	Zn	Ag	Sn	Pb	2-sig S	2-sig Fe	2-sig Ni	2-sig Cu	2-sig Zn	2-sig Ag	2-sig Sn	2-sig Pb
AT 013	0,07	0,33	0,05	75,49	18,72	0,33	2,70	2,31	*0,01*	*0,04*	*0,01*	*0,90*	*0,49*	*0,06*	*0,21*	*0,53*
AV 083	0,07	0,18	0,01	77,13	19,21	0,33	1,01	2,06	*0,01*	*0,03*	*0,00*	*0,97*	*0,54*	*0,06*	*0,11*	*0,53*
AV 160	0,19	1,40	0,04	79,34	15,15	0,29	1,31	2,29	*0,01*	*0,05*	*0,01*	*0,56*	*0,29*	*0,04*	*0,08*	*0,29*
AW 021-2	0,09	0,22	0,13	80,17	15,30	0,32	0,75	3,02	*0,01*	*0,04*	*0,01*	*1,03*	*0,71*	*0,09*	*0,09*	*0,64*
AW 062	0,15	0,19	0,11	75,80	20,55	0,37	0,39	2,52	*0,01*	*0,06*	*0,01*	*1,08*	*0,58*	*0,07*	*0,15*	*0,57*
AW 063-2	0,03	0,19	0,14	77,43	20,01	0,41	0,33	1,46	*0,01*	*0,03*	*0,03*	*1,18*	*0,67*	*0,09*	*0,08*	*0,55*
AW 063-3	0,02	0,15	0,09	77,62	19,93	0,37	0,30	1,51	*0,01*	*0,03*	*0,02*	*0,98*	*0,56*	*0,07*	*0,06*	*0,46*
AW 063-4	-	0,18	0,10	78,63	18,94	0,29	0,36	1,50	-	*0,02*	*0,02*	*0,85*	*0,47*	*0,05*	*0,06*	*0,40*
BL 014	0,06	0,16	0,10	78,77	18,73	0,31	0,31	1,55	*0,02*	*0,03*	*0,02*	*1,08*	*0,59*	*0,07*	*0,07*	*0,51*
BO 029	-	0,19	0,10	77,18	19,04	0,39	0,47	2,61	-	*0,05*	*0,05*	*1,32*	*0,70*	*0,11*	*0,13*	*0,86*
BO 047	-	0,18	0,11	78,18	18,66	0,38	0,41	2,08	-	*0,03*	*0,01*	*0,70*	*0,47*	*0,06*	*0,06*	*0,71*
BO 049	-	0,22	0,10	76,40	18,55	0,66	0,62	3,42	-	*0,05*	*0,02*	*1,14*	*0,85*	*0,11*	*0,12*	*0,84*
BR 096	0,19	0,15	0,25	82,12	13,68	0,51	0,68	2,41	*0,02*	*0,04*	*0,04*	*0,69*	*0,40*	*0,05*	*0,14*	*0,62*
BR 103	-	0,32	0,16	79,17	12,14	0,19	0,92	7,08	-	*0,07*	*0,03*	*1,17*	*0,70*	*0,09*	*0,22*	*1,51*
BS 066	-	0,33	1,77	79,58	14,17	0,30	1,49	2,35	-	*0,08*	*0,17*	*1,11*	*0,70*	*0,10*	*0,27*	*0,81*
F 326	-	0,18	0,12	80,09	16,36	0,49	0,49	2,25	-	*0,04*	*0,00*	*1,09*	*0,75*	*0,10*	*0,11*	*0,78*
F 330	0,10	0,19	0,25	81,77	13,82	0,28	0,79	2,81	*0,01*	*0,03*	*0,04*	*0,72*	*0,43*	*0,05*	*0,13*	*0,66*
N 051	0,06	0,19	0,30	79,75	16,51	0,30	0,64	2,25	*0,04*	*0,05*	*0,09*	*1,21*	*0,79*	*0,10*	*0,21*	*0,81*
N 251	-	0,19	0,09	76,18	19,50	0,42	0,47	2,50	-	*0,03*	*0,01*	*0,66*	*0,48*	*0,05*	*0,05*	*0,65*
S 0027	0,09	0,17	0,11	76,16	19,71	0,35	0,56	2,84	*0,01*	*0,03*	*0,00*	*0,79*	*0,53*	*0,07*	*0,11*	*0,86*
Sample 223	-	0,16	0,13	73,67	22,82	0,46	0,41	2,31	-	*0,02*	*0,01*	*0,70*	*0,54*	*0,06*	*0,09*	*0,75*
sAV 412 E1	-	0,19	0,22	75,98	19,66	0,41	0,70	2,84	-	*0,02*	*0,00*	*0,81*	*0,61*	*0,07*	*0,06*	*0,67*
sBS 1288 A	0,10	0,13	0,56	86,36	9,03	0,15	2,02	1,66	*0,01*	*0,02*	*0,03*	*0,61*	*0,23*	*0,02*	*0,10*	*0,39*

- **Gunmetal**

Reg. nr.	S	Fe	Ni	Cu	Zn	Ag	Sn	Pb	*2-sig S*	*2-sig Fe*	*2-sig Ni*	*2-sig Cu*	*2-sig Zn*	*2-sig Ag*	*2-sig Sn*	*2-sig Pb*
BR 104	-	1,37	0,22	80,32	4,18	0,11	8,75	5,09	-	*0,03*	*0,02*	*0,58*	*0,30*	*0,03*	*0,33*	*0,45*
BS 054	0,05	0,16	0,14	81,83	9,52	0,40	5,30	2,60	*0,00*	*0,03*	*0,02*	*0,74*	*0,36*	*0,06*	*0,21*	*0,66*
BS 088	-	0,24	0,25	78,90	3,44	5,73	9,08	2,31	-	*0,03*	*0,03*	*0,66*	*0,24*	*0,22*	*0,31*	*0,59*
F 128	-	0,12	0,18	85,42	4,06	0,21	7,30	2,68	-	*0,05*	*0,03*	*1,09*	*0,41*	*0,08*	*0,51*	*0,79*
S 0022	-	0,54	0,24	79,01	4,89	0,12	11,68	3,51	-	*0,08*	*0,03*	*0,72*	*0,27*	*0,04*	*0,42*	*0,75*
sBS 1493	-	0,28	0,41	77,62	3,79	0,15	15,47	2,29	-	*0,04*	*0,02*	*0,59*	*0,18*	*0,00*	*0,29*	*0,44*
sM 1250 B	-	0,24	0,32	84,86	3,39	0,21	8,05	2,92	-	*0,02*	*0,02*	*0,74*	*0,14*	*0,03*	*0,18*	*0,48*

2. Restricted EDX-dataset

- **Unalloyed copper**

Reg. nr.	Cu	Zn	Ag	Sn	Pb	*2-sig Cu*	*2-sig Zn*	*2-sig Ag*	*2-sig Sn*	*2-sig Pb*
AW 063-1	98,15	-	-	0,44	1,42	*1,17*	-	-	*0,07*	*0,47*
BK 005	56,35	-	-	0,87	42,78	*1,25*	-	-	*0,17*	*3,53*
BQ 153	96,14	-	-	0,89	2,97	*1,37*	-	-	*0,29*	*0,92*
BQ 154	96,25	-	-	0,57	3,18	*1,34*	-	-	*0,10*	*0,81*
BS 302	98,39	-	-	-	1,61	*1,55*	-	-	-	*0,65*
ED 009	97,73	-	-	-	2,27	*1,23*	-	-	-	*0,39*
KR 012	98,33	-	-	-	1,67	*1,21*	-	-	-	*0,53*
M 084	96,17	-	-	0,56	3,27	*1,25*	-	-	*0,09*	*0,77*
N 138	96,58	-	-	1,10	2,32	*1,62*	-	-	*0,16*	*0,83*
S 0012	97,12	-	-	0,56	2,33	*1,22*	-	-	*0,04*	*0,62*
sBJ 1237 A (2)	97,51	-	1,38	-	1,11	*1,22*	-	*0,13*	-	*0,46*
sBO 1275 B	95,93	-	-	-	3,64	*1,03*	-	-	-	*0,60*
sBR 1157 C	97,80	-	-	0,61	1,59	*1,12*	-	-	*0,05*	*0,48*
sBS 1129 A	98,43	-	-	-	1,57	*1,03*	-	-	-	*0,40*
sBS 1429	96,93	0,83	-	-	2,24	*1,28*	*0,10*	-	-	*0,65*
sM 1250 C	95,46	-	-	1,01	3,53	*1,35*	-	-	*0,08*	*0,85*
sN 1251 A	96,66	-	-	1,05	2,29	*1,20*	-	-	*0,10*	*0,45*
SX 001	96,35	-	-	0,97	2,68	*1,26*	-	-	*0,23*	*0,81*
Z 092	95,41	-	0,94	0,45	3,20	*1,39*	-	*0,06*	*0,10*	*0,55*

- **Bronze**

Reg. nr.	Cu	Zn	Ag	Sn	Pb	*2-sig Cu*	*2-sig Zn*	*2-sig Ag*	*2-sig Sn*	*2-sig Pb*
High tin-bronze & leaded high tin-bronze										
AW 021-1	78,30	-	-	15,12	6,58	*1,00*	-	-	*0,78*	*1,35*
KR 011	69,91	-	-	16,76	13,33	*0,58*	-	-	*0,30*	*0,81*
S 0020	59,58	-	-	15,28	25,15	*0,78*	-	-	*0,44*	*1,63*
S 0023	65,99	-	-	30,55	3,46	*0,98*	-	-	*0,72*	*0,72*
sBK 1238 A	81,31	-	-	16,63	2,06	*0,75*	-	-	*0,55*	*0,38*
sBR 1041 C	61,17	0,49	-	31,89	6,45	*0,84*	*0,07*	-	*0,64*	*0,89*
Z 012	80,86	-	-	15,73	3,42	*0,90*	-	-	*0,46*	*0,50*
Medium tin-bronze & leaded medium tin-bronze										
AD 031	85,05	-	-	12,58	2,36	*1,05*	-	-	*0,61*	*0,65*
AV 005	56,83	0,77	-	7,61	34,79	*0,78*	*0,10*	-	*0,37*	*1,99*
AV 007	84,56	-	-	12,53	2,91	*1,14*	-	-	*0,74*	*0,72*
AV 016	86,43	-	-	10,55	3,02	*1,10*	-	-	*0,35*	*0,64*
AV 115	91,08	-	-	6,16	2,76	*1,00*	-	-	*0,56*	*0,40*
BQ 016	91,58	-	-	6,19	2,23	*1,28*	-	-	*0,34*	*0,66*

Reg. nr.	Cu	Zn	Ag	Sn	Pb	*2-sig Cu*	*2-sig Zn*	*2-sig Ag*	*2-sig Sn*	*2-sig Pb*
BQ 070	89,65	-	-	7,12	3,23	*1,14*	-	-	*0,50*	*0,69*
BR 026	85,27	-	-	11,94	2,79	*1,03*	-	-	*0,54*	*0,43*
BS 064	83,86	-	-	10,39	5,75	*1,12*	-	-	*0,63*	*0,98*
C 079	80,66	1,08	-	7,42	10,84	*0,66*	*0,09*	-	*0,31*	*0,83*
K 005	83,42	-	-	13,89	2,69	*0,90*	-	-	*0,48*	*0,46*
K 149	83,48	-	-	14,11	2,41	*0,97*	-	-	*0,65*	*0,57*
K 153	83,51	-	-	13,17	3,32	*1,08*	-	-	*0,66*	*0,68*
K 203	69,97	-	-	10,70	19,33	*0,77*	-	-	*0,46*	*1,39*
KR 007	77,19	-	-	11,02	11,79	*1,10*	-	-	*0,44*	*1,41*
KR 009	88,61	-	-	8,56	2,83	*1,10*	-	-	*0,35*	*0,64*
M 007	55,93	0,72	-	6,16	37,18	*0,80*	*0,10*	-	*0,36*	*2,13*
N 118	79,87	-	-	6,82	13,31	*1,26*	-	-	*0,59*	*2,31*
N 121	85,57	-	-	11,63	2,81	*1,02*	-	-	*0,72*	*0,53*
N 122	85,42	-	-	11,61	2,97	*1,04*	-	-	*0,47*	*0,46*
S 0021	90,60	-	-	6,77	2,64	*1,28*	-	-	*0,58*	*0,65*
sBQ 1058 A	91,73	-	-	5,82	2,45	*0,93*	-	-	*0,26*	*0,28*
sBQ 1058 B	88,47	-	-	10,00	1,53	*0,86*	-	-	*0,28*	*0,35*
sBQ 1173 A	89,11	-	-	8,26	2,63	*1,10*	-	-	*0,52*	*0,57*
sBS 1276	92,12	-	-	5,25	2,63	*1,18*	-	-	*0,34*	*0,46*
sFO 1308 A	89,61	-	-	8,84	1,55	*0,81*	-	-	*0,25*	*0,35*
Z 146	78,37	-	-	5,54	16,09	*1,18*	-	-	*0,54*	*0,91*
Leaded low tin-bronze										
AV 104	58,47	-	-	4,77	36,76	*0,82*	-	-	*0,27*	*2,12*
BS 154	76,67	-	-	3,58	19,74	*1,11*	-	-	*0,49*	*2,70*
KR 008	79,34	-	-	4,45	16,21	*1,08*	-	-	*0,27*	*1,59*
sBR 1157 A	80,11	-	-	4,87	15,02	*0,94*	-	-	*0,37*	*1,31*

- **Brass**

Reg. nr.	Cu	Zn	Sn	Pb	*2-sig Cu*	*2-sig Zn*	*2-sig Sn*	*2-sig Pb*
AT 013	76,08	18,86	2,73	2,33	*0,91*	*0,49*	*0,21*	*0,53*
AV 083	77,57	19,33	1,03	2,07	*0,97*	*0,54*	*0,11*	*0,53*
AV 160	78,55	17,63	1,31	2,52	*1,13*	*0,62*	*0,15*	*0,44*
AW 021-2	81,04	15,70	0,75	3,26	*1,04*	*0,72*	*0,09*	*0,65*
AW 062	76,65	20,80	-	2,55	*1,10*	*0,59*	-	*0,58*
AW 063-2	78,28	20,24	-	1,48	*1,19*	*0,68*	-	*0,55*
AW 063-3	78,34	20,13	-	1,53	*0,99*	*0,56*	-	*0,39*
AW 063-4	79,36	19,12	-	1,52	*0,86*	*0,47*	-	*0,25*
BL 014	79,52	18,92	-	1,56	*1,09*	*0,60*	-	*0,35*
BO 029	78,25	19,09	-	2,65	*1,33*	*0,71*	-	*0,87*
BO 047	79,02	18,87	-	2,11	*1,07*	*0,72*	-	*0,49*
BO 049	75,89	18,88	0,62	3,48	*1,16*	*0,86*	*0,12*	*0,85*
BR 096	82,91	13,94	0,69	2,46	*1,06*	*0,61*	-	*0,40*
BR 103	79,72	12,22	0,92	7,14	*1,19*	*0,71*	*0,23*	*1,55*
BS 066	81,60	14,50	1,48	2,41	*1,16*	*0,73*	*0,28*	*0,85*
F 326	81,13	16,59	-	2,29	*1,10*	*0,76*	-	*0,79*
F 330	82,39	14,05	0,79	2,86	*1,08*	*0,65*	*0,20*	*0,42*
N 051	80,30	16,76	0,64	2,29	*1,09*	*0,71*	*0,19*	*0,51*
N 251	77,09	19,72	-	3,19	*1,01*	*0,74*	-	*0,66*
S 0027	77,13	19,98	0,56	2,89	*1,17*	*0,83*	*0,11*	*0,56*
Sample 223	74,55	23,12	-	2,34	*1,06*	*0,82*	-	*0,49*
sAV 412 E1	77,13	19,98	0,70	2,89	*1,00*	*0,75*	*0,08*	*0,54*
sBS 1288 A	87,05	9,03	2,17	1,76	*0,84*	*0,30*	*0,12*	*0,31*

- **Gunmetal**

Reg. nr.	Cu	Zn	Ag	Sn	Pb	*2-sig Cu*	*2-sig Zn*	*2-sig Ag*	*2-sig Sn*	*2-sig Pb*
BR 104	81,68	4,25	-	8,89	5,18	*1,22*	*0,32*	-	*0,63*	*1,34*
BS 054	82,45	9,59	-	5,34	2,62	*1,11*	*0,54*	-	*0,34*	*0,48*
BS 088	79,33	3,46	5,76	9,13	2,32	*1,00*	*0,35*	*0,22*	*0,31*	*0,42*
F 128	85,90	4,08	-	7,33	2,70	*1,10*	*0,42*	-	*0,51*	*0,80*
S 0022	79,74	4,93	-	11,78	3,55	*1,05*	*0,42*	-	*0,64*	*0,51*
sBS 1493	78,65	4,78	-	13,72	2,85	*0,86*	*0,26*	-	*0,42*	*0,30*
sM 1250 B	85,54	3,41	-	8,10	2,95	*0,96*	*0,18*	-	*0,26*	*0,38*

Appendix 8: Metrical, typological and contextual data on the slag

• Metrical data

The specific gravity is only an approximate calculation. The volume was obtained by submerging the slag in a recipient filled with water and measuring the water displacement in millilitres. Dividing the weight by the volume cubic centimetres (1 ml = 1 cm^3) gives the specific gravity. Considering the rather large errors on the volume estimate, the results should be seen only as an indication of the true specific gravity.

Original Reg. nr	New sample nr.	Type	State	Height ca. mm	Width ca. mm	Depth ca. mm	Weight in grams	Volume in cm^3	Specific gravity in g/cm^3	Magnetic
S 0004	S 0004	?	Frag.	15	19	20	5,8	3	1,93	++
S 0007	S 0007	T1	Frag.	20	47	41	37,0	13,5	2,74	-
sAH 233	AH 233	T1	Frag.	20	72	65	113,4	30	3,78	++
sBM 679	BM 679A	?	Frag.	12	23	17	6,0	3	2,00	+
sBN 689	BN 689	T2	Frag.	17	20	29	10,0	4	2,50	+
sBO 1136	BO 1136	?	Frag.	20	50	31	32,5	12	2,71	++
sBO 716	BO 716	?	Frag.	10	23	22	3,8	1,5	2,53	++
sBO 724	BO 724B	T1	Frag.	23	58	41	56,9	17	3,35	++
sBO 740	-	-	-	-	-	-	-	-	-	?
sBO 769	-	-	-	-	-	-	-	-	-	?
sBO 845	BO 845A	T3?	Frag.	2	44	40	64,9	17	3,82	+
sBQ 1006	BQ 1006B	T1	Compl.	18	32	25	16,7	5	3,34	++
sBQ 1008*	BQ 1008A	T2	Frag.	13	30	?	17,5	6	2,92	++
sBQ 1008*	BQ 1008B	T3	Frag.	23	57	64	123,6	35	3,53	+
sBQ 1171	-	-	-	-	-	-	-	-	-	?
sBQ 777	BQ 777B	T2	Frag.	19	32	24	12,0	5	2,40	+
sBQ 777	BQ 777A	T2	Frag.	26	50	40	51,7	17	3,04	++
sBQ 788	BQ 788	T2?	Frag.	14	18	33	10,8	4	2,70	++
sBQ 819	BQ 819	T2	Frag.	28	37	32	35,2	12	2,93	++
sBQ 872	BQ 872	T2	Frag.	18	37	35	24,0	7	3,43	++
sBQ 875	BQ 875	T1	Compl.	17	59	45	46,6	16,5	2,82	++
sBQ 893	BQ 893A	?	Frag.	26	45	30	59,4	20	2,97	-
sBQ 893	BQ 893B	T2	Frag.	17	39	35	21,0	7	3,00	+
sBQ 893	BQ 893D	T3?	Frag.	27	36	27	55,7	13,5	4,13	+
sBQ 940	BQ 940E	?	Frag.	-	-	-	8,4	2,5	2,84	++
sBQ 940	BQ 940A	T2	Compl.	37	47	46	56,0	17	3,29	++
sBQ 940	BQ 940D	T2	Compl.	15	47	32	22,7	8	2,83	++
sBQ 940	BQ 940C	T3	Frag.	21	50	42	61,0	21,5	2,83	++
sBQ 940	BQ 940B	T3?	Frag.	24	39	30	37,8	10	3,78	++
sBQ 955	BQ 955	T2?	Frag.	14	46	23	20,9	6,5	3,22	++
sBQ 967	-	-	-	-	-	-	-	-	-	?
sBQ 976	BQ 976A	?	Frag.	12	43	26	14,7	6	2,45	+
sBQ 976	BQ 976C	?	Frag.	17	27	21	10,5	4	2,63	++
sBQ 976	BQ 976B	?	Frag.	26	42	32	42,6	15	2,84	++
sBQ 976	BQ 976E	T2	Frag.	19	23	35	14,0	6	2,33	++
sBQ 976	BQ 976D	T2	Frag.	17	57	48	48,9	17	2,88	++
sBQ 989	BQ 989	?	Frag.	11	18	17	3,2	1,5	2,13	+
sBR 1021	-	-	-	-	-	-	-	-	-	?
sBR 1041	BR 1041B	?	Frag.	9	22	15	3,3	1	3,90	+
sBR 1041	BR 1041A	T2	Frag.	14	22	16	3,9	2	1,65	+
sBR 938	-	-	-	-	-	-	-	-	-	?

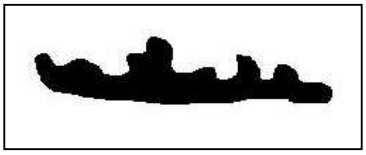
Original Reg. nr	**New sample nr.**	**Type**	**State**	**Height** ca. mm	**Width** ca. mm	**Depth** ca. mm	**Weight** in grams	**Volume** in cm^3	**Specific gravity** in g/cm^3	**Magnetic**
sBR 982	BR 982	T4	Compl.	20	77	70	109,1	36	3,03	++
sBR 987	BR 987C	?	Frag.	12	60	20	2,7	1	2,70	++
sBS 1068*	BS 1068	T3	Compl.	12	60	20	100,0	40	2,50	+
sBS 1110*	BS 1110	T1?	Compl.	30	40	?	37,2	18	2,07	-
sBS 1116	BS 1116	T1	Frag.	14	53	52	54,5	16	3,41	++
sBS 1138	-	-	-	-	-	-	-	-	-	?
sBS 1152	BS 1152	?	Frag.	19	37	36	26,9	8,5	3,16	+
sBS 1185	BS 1185A	T2	Compl.	27	38	10 à 20	17,6	5,5	3,20	++
sBS 1185	BS 1185B	T2	Compl.	28	43	15	16,4	5	3,28	++
sBS 1198	BS 1198B	T3	Frag.	29	55	48	101,7	34	2,99	+
sBS 1198	BS 1198A	T3	Frag.	33	76	60	178,1	56	3,18	+
sBS 1214	BS 1214	T1	Frag.	14	35	25	10,3	3	3,43	++
sBS 1215	BS 1215	T2	Frag.	31	57	45	65,8	27	2,44	++
sBS 1264	BS 1264	T4	Frag.	12	50	45	40,1	12	3,34	++
sBS 1318	BS 1318	T3	Frag.	20-30	87	67	219,5	73	3,01	++
sBS 1361	BS 1361	T4	Frag.	12	51	43	35,8	12	2,98	++
sBS 1400	BS 1400	T1	Frag.	21	48	46	51,6	17	3,04	++
sBS 1402	BS 1402A	T1	Compl.	16	65	42	47,8	16,5	2,90	++
sBS 1402	BS 1402B	T2	Compl.	23	40	35	23,5	7,5	3,13	++
sBS 1407	BS 1407	T2	Frag.	17	40	30	33,7	10	3,37	++
sBS 1414	BS 1414B	T1	Compl.	17	60	41	59,6	21	2,84	+
sBS 1417	BS 1417	T1	Frag. ?	18	60	54	71,2	20,5	3,47	++
sBS 1425	BS 1425	T1	Frag.	18	35	35	35,6	6,5	5,48	++
sBS 1431	BS 1431	T4	Frag.	15	60	39	49,8	15	3,32	++
sBS 1458	BS 1458A	?	Frag.	12	39	21	8,3	3,5	2,37	++
sBS 1458	BS 1458B	?	Frag.	25	52	28	61,6	16	3,85	++

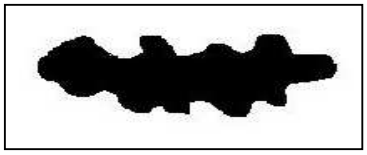
Metrical data of the slag. Registration numbers in **bold cursive** are slags given to A. Ploquin for analyses before this study. No report or results were available but two analyses seem to be included in Ploquin, Orzechowski & Briand, 1999: 184. An **asterisk** (*) indicates that these slags were sampled before they were measured and weighed, so the figures are from after the sampling. **Compl**. indicates that the piece of slag was complete, whereas **Frag**. is used when the slag was in a fragmented state. The magnetic properties as determined with a handheld magnetic are labelled strongly magnetic (**++**)**,** little magnetic (**+**) or not magnetic (**-**). In the first case the magnet stayed in place even when held upside down, in the second case attraction was clearly felt but was not strong enough to keep the magnet in place, and in the last case no magnetism was attested.

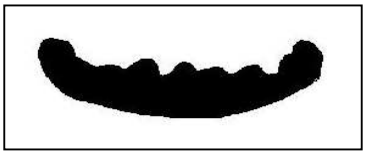
• Typological data

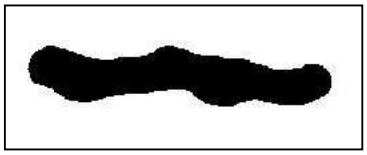
Four morphological types were distinguished, but no further conclusions could be linked to that.

- *Type 1* (T1): Spots of reddish iron oxides, flat on the bottom and nodules on the topside.
- *Type 2* (T2): Spots of reddish iron oxides, nodular both on and topside.
- *Type 3* (T3): Spots of reddish iron oxides, "bowl-shaped" bottom and nodular topside.
- *Type 4* (T4): Spots of reddish iron oxides, flat on the bottom and topside.


Type 1


Type 2


Type 3


Type 4

Type of slag	**Total weight** per type in grams	**Average weight** individual slag in grams	**Total volume** per type in cm^3	**Average volume** individual slag in cm^3	**Average specific gravity** per type in g/cm^3
T1	601,2	50,1	182,5	15,2	3,3
T1 + T1?	638,4	49,1	200,5	15,4	3,2
T2	473,9	27,9	163,0	9,6	2,9
T2 + T2?	505,6	26,6	173,5	9,1	2,9
T3	783,9	130,7	259,5	43,3	3,0
T3 + T3?	942,3	104,7	300,0	33,3	3,1
T4	234,8	58,7	75,0	18,8	3,1

Weight, volume and specific gravity information per type.

• Contextual data

Reg. nr	Area	UF n°	Sq n°	Loc n°	Context
S 0004	-	-	-	-	No associated structures
S 0007	-	-	-	-	No associated structures
sAH 233	AH	2491	III 2	-	Subsurface
sBM 679	BM	5775	III 2	G 6130	In tomb G 6130, found when sieving content but not recognized as slag
sBN 689	BN	5754	I 1		Found in filling of a wall
sBO 716	BO	5900	II 3	G 6154	In tomb G 6154
sBO 740	BO	5907	I 3	-	In surface layer above tombs
sBO 769	BO	5911	I 2	-	In surface layer (nothing beneath)
sBQ 777	BQ	5914	-		In surface layer
sBQ 788	BQ	5961	I 1	-	Found in concentration of stone, ceramic, shells, ... to the east of G 6266
sBQ 819	BQ	5965	I 2	-	In surface layer (neighborhood of tombs)
sBO 845	BO	Surface	-	-	In surface layer (nothing beneath)
sBQ 872	BQ	5935	C 1	-	In surface layer
sBQ 875	BQ	5970	A 3	-	In surface layer (above G 6273)
sBQ 893	BQ	5938	C 1	G 6234	Found in G 6234 (very disturbed)
sBR 938	BR	6040	IV 2	-	Above G 6312
sBQ 940	BQ	5940	C 3	-	Subsurface
sBQ 955	BQ	5944	B 4	G 6235	Found in G 6235
sBQ 967	BQ	5978	A 4	-	Surface layer (above G 6275 and G 6276)
sBQ 976	BQ	5978	A 4	-	Surface layer (above G 6275 and G 6276)
sBR 982	BR	6055	V 3	-	In a burned place
sBR 987	BR	6056	V 4	G 6314	In tomb G 6314
sBQ 989	BQ	5943	B 4	-	Subsurface
sBQ 1006	BQ	5951	B 5a	-	In surface layer
sBR 1021	BR	6058	VI 3		Surface layer
sBR 1041	BR	6065	I 3	-	Subsurface
sBS 1068	BS	6504	III 4	-	Surface layer
sBS 1110	BS	6554	IV 3		Subsurface
sBS 1116	BS	6555	IV 3		Under subsurface
sBO 1136	BO	Dump	-	-	Found on dump
sBS 1138	BS	6611	VI 3	-	Surface layer
sBS 1152	BS	6557	IV 3	-	In neighborhood G 7041
sBQ 1171	BQ	Dump East	-	-	Dump
sBS 1185	BS	6557	IV 3	-	In neighborhood G 7041
sBS 1198	BS	6563	IV 4	-	Found in between concentration of shell and bone
sBS 1214	BS	6538	V 4	-	At 40 cm depth, no related structures
sBS 1215	BS	6540	V 2	-	Surface find
sBS 1264	BS	6625	VI 2	-	Surface layer
sBS 1318	BS	6639	VII 3	-	Subsurface (gravel, plaster structure)
sBS 1361	BS	6705	VIII 5	-	Surface layer
sBS 1400	BS	6712	VII 5	-	Layer with mud floor
sBS 1402	BS	6757	VII 4	-	Surface layer
sBS 1407	BS	6658	VI 1	7102	To the side of a small clay wall
sBS 1414	BS	6712	VII 5	-	Layer with mud floor
sBS 1417	BS	6760	I X 4	-	No associated structures
sBS 1425	BS	6719	IX 5	-	Subsurface
sBS 1431	BS	6751	VII 4	-	Subsurface
sBS 1458	BS	6760	IX 4	-	No associated structures

Contextual data on the slag. The surface layer is the present-day walking level and the subsurface is the layer just beneath it, together they make up between 10 and 35 cm.

Appendix 9: XRF-analyses performed on two litharge samples

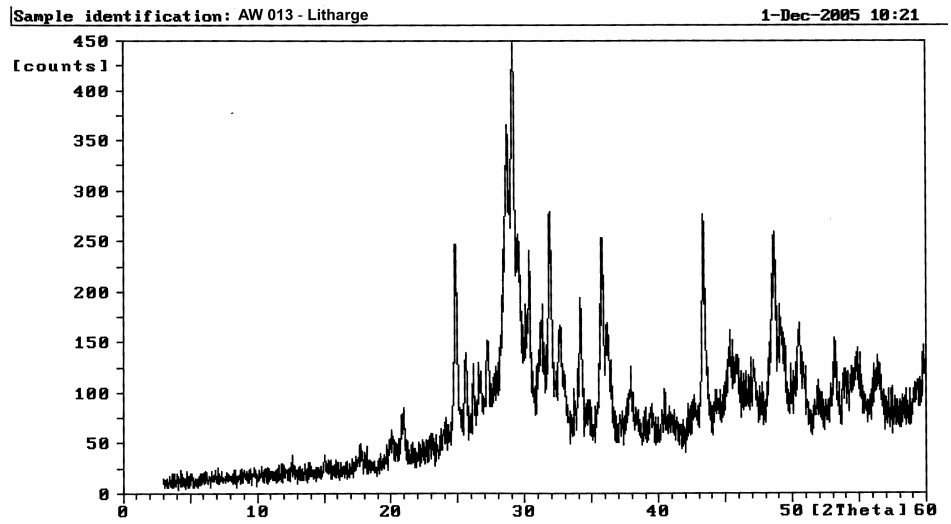
		BO 724A	BO 722
Na	%	-	-
Mg	%	0,613	0,604
Al	%	1,099	1,330
Si	%	5,676	5,797
P	%	< 0,0027	< 0,0027
S	%	> 2,2280	> 1,9160
Cl	%	-	-
K	%	-	-
Ca	%	-	-
Ti	%	0,0539	0,1089
V	%	< 0,0025	< 0,0026
Cr	%	< 0,0081	0,0165
Mn	%	< 0,0060	0,0191
Fe	%	0,645	1,278
Co	%	< 0,0042	0,0203
Ni	%	0,0271	0,0398
Cu	%	19,81	15,84
Zn	%	0,0450	0,0612
As	%	< 0,0001	0,0230
Zr	%	< 0,0049	< 0,0043
Nb	%	< 0,0038	< 0,0052
Mo	%	< 0,0016	< 0,0025
Rh	%	< 0,0008	< 0,0011
Pd	%	< 0,0008	< 00010
Ag	%	> 0,2415	0,1699
Cd	%	0,0012	< 0,0007
Sn	%	< 0,0001	< 0,0001
Sb	%	0,02701	0,0092
Te	%	< 0,0005	< 0,0007
Ba	%	< 0,0017	0,0080
Au	%	-	0,0159
Pb	%	69,53	72,74
Total		100	110,02

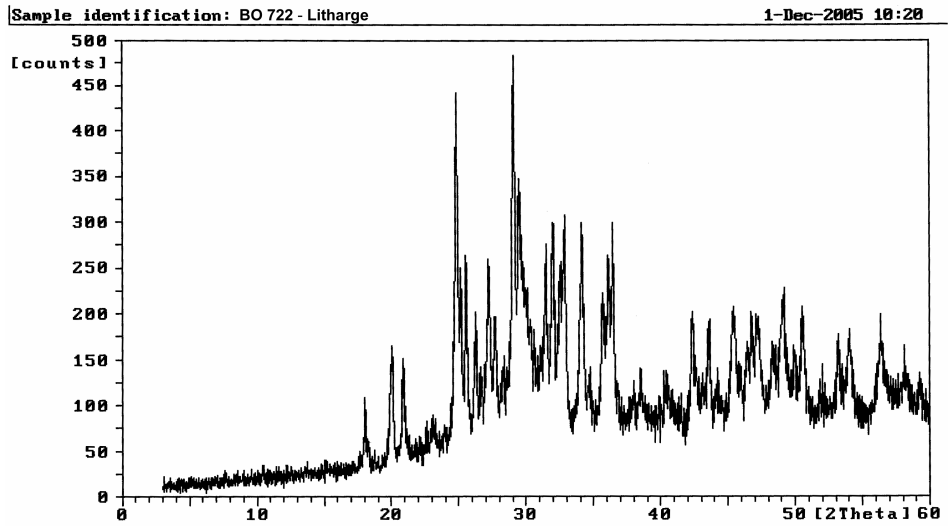
		BO 724A	BO 722
Na_2O	%	2,480	3,260
MgO	%	2,17	1,278
Al_2O_3	%	3,056	2,204
SiO_2	%	17,70	10,850
P_2O_5	%	< 0,0140	< 0,0076
SO_3	%	3,412	1,455
Cl	%	< 0,0063	< 0,0031
K_2O	%	0,320	0,2330
CaO	%	5,519	2,966
TiO_2	%	0,0582	0,0649
V_2O_5	%	< 0,0047	< 0,0025
Cr_2O_3	%	< 0,0120	< 0,0052
MnO	%	< 0,0068	0,0099
Fe_2O_3	%	0,4986	0,569
CoO	%	< 0,0034	0,0034
NiO	%	0,0197	0,0147
CuO	%	13,440	6,0690
ZnO	%	0,0064	0,0051
As_2O_3	%	< 0,0013	0,0187
ZrO_2	%	0,0032	< 0,0016
Nb_2O_5	%	< 0,0013	< 0,0010
Mo	%	< 0,0008	< 0,0007
Rh	%	-	-
Pd	%	-	-
Ag	%	0,2475	> 0,0113
Cd	%	0,0024	< 0,0002
SnO_2	%	0,0200	0,0629
Sb	%	0,0075	0,0017
Te	%	< 0,0003	< 0,0002
Ba	%	0,0018	16
Au	%	-	-
PbO	%	28,71	18,44
Total		77,72	63,54

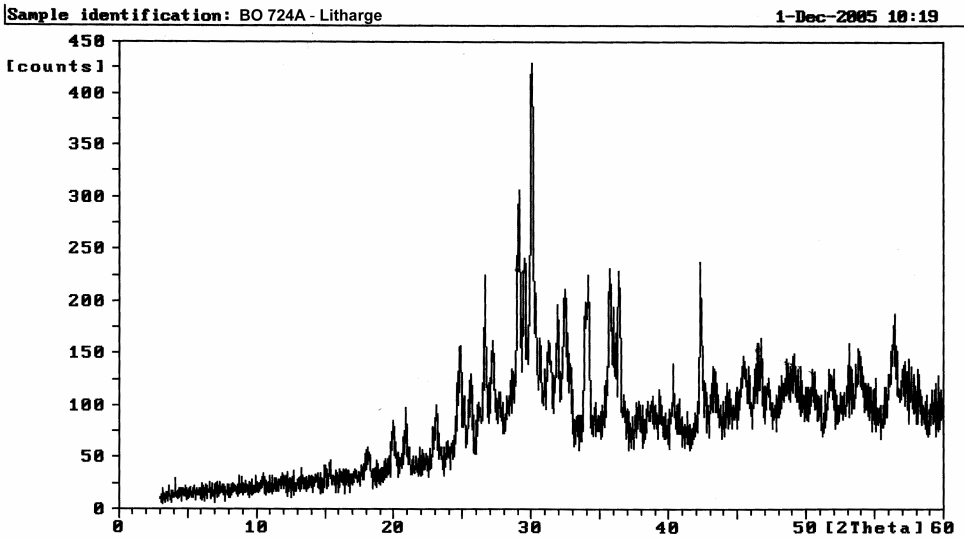
X. Veldhuijzen preformed these analyses when I visited the *Institute of Archaeology – UCL*. Eventually these analyses were not used since I am not familiar with the technique. Still I found it useful to present the obtained data in appendix, rather than throwing it away. The two samples were each measured twice and the data was processed by different software packages, the first only taking in account the element, the other calculating the oxides of every element. The results tend to differ considerably. The reason that the totals do not add up to 100% is due to the fact that the data was not normalised and that certain elements not included in the analysis were present.

Appendix 10: Raw XRD-spectra and analytical data

- **Litharge**







Sample identification: **AW 013**
Data measured at: 9-Aug-2005 22:56:00

Diffractometer type: PW3710 BASED
Tube anode: Cu
Generator tension [kV]: 40
Generator current [mA]: 30
Wavelength Alpha1 [Å]: 1.54060
Wavelength Alpha2 [Å]: 1.54439
Intensity ratio (alpha2/alpha1): 0.500
Divergence slit: AUTOMATIC
Irradiated length [mm]: 12
Receiving slit: 0.1
Monochromator used: YES

Start angle [°2θ]: 3.010
End angle [°2θ]: 59.990
Step size [°2θ]: 0.020
Maximum intensity: 353.4400
Time per step [s]: 1.000
Type of scan: CONTINUOUS
Intensities converted to: FIXED

Minimum peak tip width: 0.00
Maximum peak tip width: 1.00
Peak base width: 2.00
Minimum significance: 0.75
Number of peaks: 34

Angle [°2θ]	d-value α1 [Å]	d-value α2 [Å]	Peak width [°2θ]	Peak int [counts]	Back. int [counts]	Rel. int [%]	Signif.
15.050	5.8820	5.8965	0.120	38	20	10.9	1.11
17.645	5.0224	5.0347	0.640	21	23	6.0	1.40
20.035	4.4283	4.4392	0.240	28	30	7.9	1.45
20.965	4.2339	4.2443	0.280	62	34	17.7	2.05
24.890	3.5744	3.5832	0.060	219	45	62.0	0.78
25.600	3.4769	3.4854	0.200	98	45	27.7	3.00
26.160	3.4037	3.4121	0.100	61	46	17.2	0.79
26.630	3.3447	3.3529	0.160	69	46	19.5	1.76
27.190	3.2771	3.2851	0.160	102	46	28.9	1.16
28.675	3.1106	3.1183	0.240	310	48	87.6	2.59
29.190	3.0569	3.0644	0.140	353	49	100.0	3.11
30.350	2.9427	2.9499	0.080	182	49	51.6	0.93
31.290	2.8564	2.8634	0.160	110	50	31.2	1.02
31.870	2.8057	2.8126	0.080	202	50	57.1	1.12
32.700	2.7364	2.7431	0.240	94	52	26.6	2.25
34.200	2.6197	2.6262	0.200	110	53	31.2	3.15
35.755	2.5093	2.5154	0.080	166	55	47.1	1.06
36.255	2.4758	2.4819	0.200	74	55	20.9	0.91
37.915	2.3711	2.3770	0.200	41	56	11.6	1.03
39.580	2.2751	2.2807	0.240	12	58	3.5	0.81
40.375	2.2321	2.2376	0.240	14	58	3.9	1.75
42.625	2.1194	2.1246	0.240	18	61	5.0	0.89
43.360	2.0851	2.0903	0.080	144	61	40.7	0.79
45.285	2.0009	2.0058	0.240	44	64	12.3	1.16
45.870	1.9767	1.9816	0.240	42	66	12.0	1.04
47.130	1.9268	1.9315	0.120	38	67	10.9	1.14
48.655	1.8699	1.8745	0.280	106	69	30.0	5.15
49.195	1.8506	1.8552	0.400	56	69	15.9	1.84
50.500	1.8058	1.8102	0.160	52	71	14.7	0.86
51.945	1.7589	1.7632	0.400	14	74	3.9	1.41
53.155	1.7217	1.7259	0.160	38	76	10.9	0.99
53.915	1.6992	1.7034	0.240	18	76	5.2	1.38
54.845	1.6726	1.6767	0.400	25	77	7.1	1.20
56.570	1.6256	1.6296	0.280	18	79	5.0	1.17

Sample identification:	BO 722
Data measured at:	9-Aug-2005 22:08:00
Diffractometer type:	PW3710 BASED
Tube anode:	Cu
Generator tension [kV]:	40
Generator current [mA]:	30
Wavelength Alpha1 [Å]:	1.54060
Wavelength Alpha2 [Å]:	1.54439
Intensity ratio (alpha2/alpha1):	0.500
Divergence slit:	AUTOMATIC
Irradiated length [mm]:	12
Receiving slit:	0.1
Monochromator used:	YES
Start angle [°2θ]:	3.010
End angle [°2θ]:	59.990
Step size [°2θ]:	0.020
Maximum intensity:	436.8100
Time per step [s]:	1.000
Type of scan:	CONTINUOUS
Intensities converted to:	FIXED
Minimum peak tip width:	0.00
Maximum peak tip width:	1.00
Peak base width:	2.00
Minimum significance:	0.75
Number of peaks:	39

Angle [°2θ]	d-value α1 [Å]	d-value α2 [Å]	Peak width [°2θ]	Peak int [counts]	Back. int [counts]	Rel. int [%]	Signif.
18.030	4.9160	4.9281	0.100	128	28	29.2	1.37
20.110	4.4120	4.4228	0.140	161	28	36.9	2.12
20.910	4.2449	4.2554	0.080	125	28	28.7	0.92
23.105	3.8464	3.8559	0.240	55	28	12.5	0.96
24.885	3.5751	3.5839	0.120	428	28	98.1	3.66
25.180	3.5339	3.5426	0.120	246	28	56.4	0.94
25.550	3.4836	3.4922	0.100	266	28	60.8	2.10
26.290	3.3872	3.3955	0.060	193	28	44.2	1.45
26.695	3.3367	3.3449	0.160	102	28	23.4	0.95
27.250	3.2700	3.2780	0.120	231	28	52.9	1.63
27.695	3.2185	3.2264	0.200	161	28	36.9	3.05
28.305	3.1505	3.1582	0.160	106	28	24.3	0.88
29.125	3.0636	3.0711	0.160	437	28	100.0	6.07
29.460	3.0295	3.0370	0.240	276	28	63.1	2.97
31.525	2.8356	2.8426	0.120	231	28	52.9	1.77
32.030	2.7921	2.7989	0.200	237	28	54.3	3.98
32.550	2.7486	2.7554	0.120	182	28	41.7	0.78
32.895	2.7206	2.7273	0.100	246	28	56.4	1.44
34.210	2.6190	2.6254	0.160	210	28	48.1	3.31
34.755	2.5791	2.5855	0.200	79	28	18.1	0.79
35.700	2.5130	2.5192	0.160	144	28	33.0	1.40
36.125	2.4844	2.4905	0.060	185	28	42.3	0.80
36.480	2.4610	2.4671	0.160	188	28	43.0	2.26
38.535	2.3344	2.3401	0.160	81	28	18.5	1.15
40.455	2.2279	2.2334	0.240	79	28	18.1	1.44
42.335	2.1332	2.1385	0.120	117	28	26.7	1.31
43.595	2.0745	2.0796	0.200	94	28	21.5	2.19
44.170	2.0488	2.0538	0.400	56	28	12.9	1.32
45.490	1.9923	1.9972	0.440	104	28	23.8	8.64
46.780	1.9404	1.9451	0.120	100	28	22.9	1.26
47.255	1.9220	1.9267	0.240	96	28	22.0	0.89
48.330	1.8817	1.8863	0.120	79	28	18.1	0.79
49.195	1.8506	1.8552	0.280	110	28	25.2	3.15
50.580	1.8031	1.8076	0.280	94	28	21.5	3.90
53.185	1.7208	1.7250	0.200	76	28	17.3	1.72
54.035	1.6957	1.6999	0.400	74	28	16.9	2.94
56.365	1.6310	1.6350	0.120	69	28	15.8	0.83
58.190	1.5841	1.5880	0.240	56	28	12.9	1.14
59.255	1.5582	1.5620	0.240	46	28	10.6	1.12

Sample identification: **BO 724A**
Data measured at: 9-Aug-2005 21:20:00

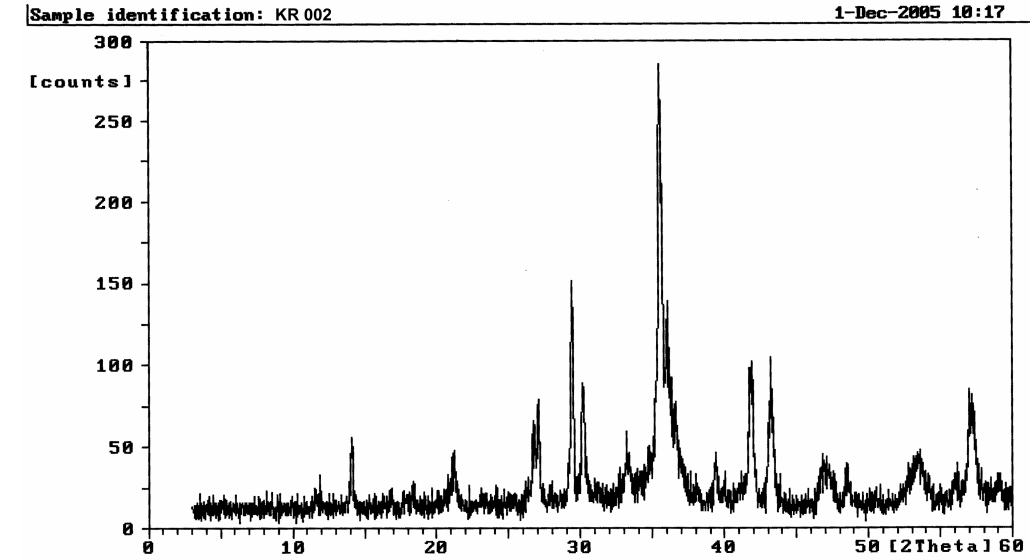
Diffractometer type: PW3710 BASED
Tube anode: Cu
Generator tension [kV]: 40
Generator current [mA]: 30
Wavelength Alpha1 [Å]: 1.54060
Wavelength Alpha2 [Å]: 1.54439
Intensity ratio (alpha2/alpha1): 0.500
Divergence slit: AUTOMATIC
Irradiated length [mm]: 12
Receiving slit: 0.1
Monochromator used: YES

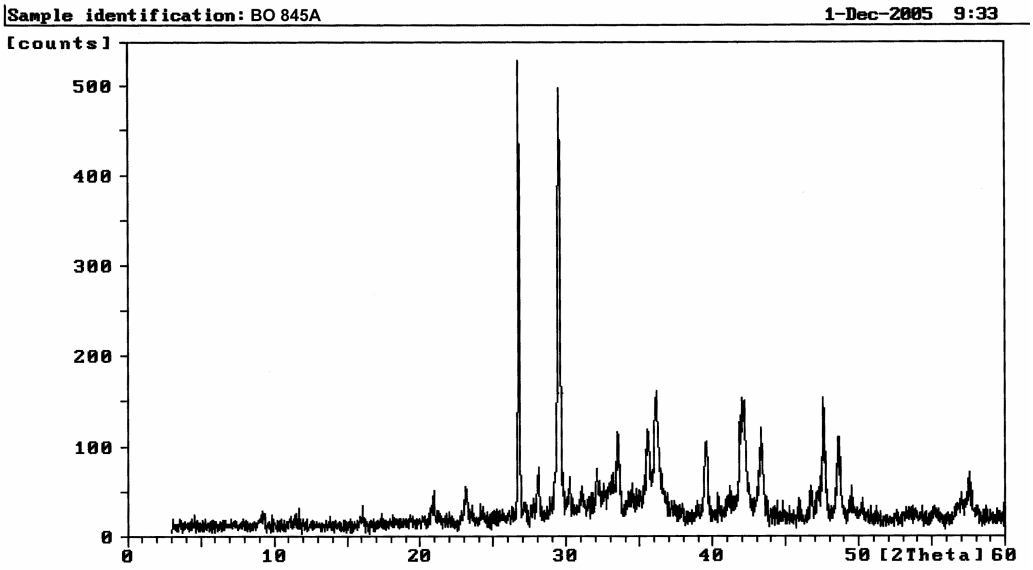
Start angle [°2θ]: 3.010
End angle [°2θ]: 59.990
Step size [°2θ]: 0.020
Maximum intensity: 361.0000
Time per step [s]: 1.000
Type of scan: CONTINUOUS
Intensities converted to: FIXED

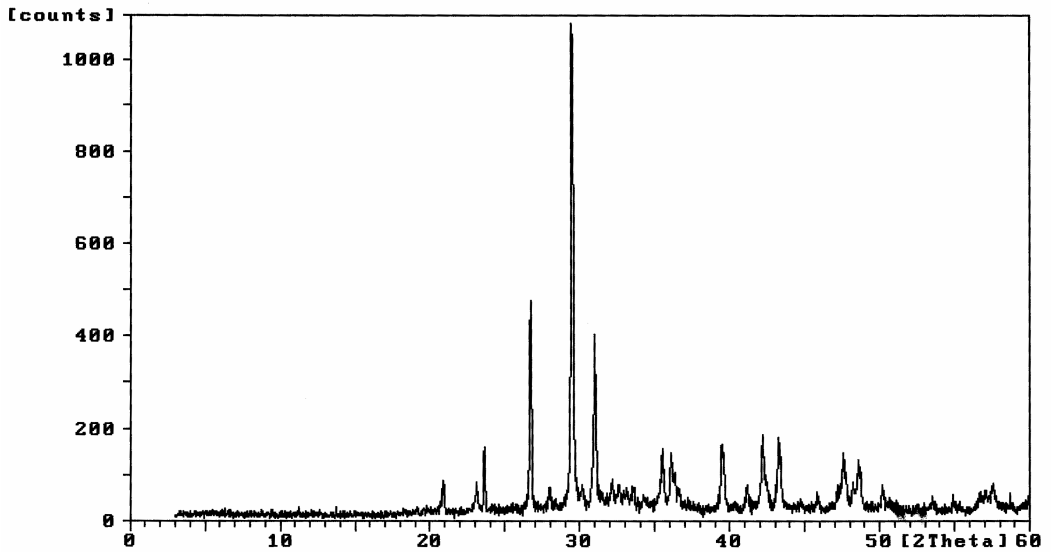
Minimum peak tip width: 0.00
Maximum peak tip width: 1.00
Peak base width: 2.00
Minimum significance: 0.75
Number of peaks: 35

Angle [°2θ]	d-value α1 [Å]	d-value α2 [Å]	Peak width [°2θ]	Peak int [counts]	Back. int [counts]	Rel. int [%]	Signif.
18.120	4.8918	4.9038	0.320	31	31	8.7	1.28
19.945	4.4481	4.4590	0.200	61	34	16.9	1.29
20.885	4.2500	4.2604	0.200	61	36	16.9	1.17
23.110	3.8456	3.8550	0.160	71	38	19.5	1.09
24.850	3.5801	3.5889	0.240	121	41	33.5	3.00
25.610	3.4756	3.4841	0.280	81	42	22.4	3.72
26.625	3.3453	3.3535	0.080	199	44	55.1	0.95
27.210	3.2747	3.2828	0.160	117	45	32.3	0.90
29.090	3.0672	3.0748	0.100	253	48	70.0	1.09
29.535	3.0220	3.0294	0.080	193	49	53.5	0.78
30.015	2.9748	2.9821	0.080	361	49	100.0	1.08
30.665	2.9132	2.9203	0.120	100	50	27.7	1.62
31.315	2.8542	2.8612	0.240	94	52	26.1	0.86
31.905	2.8027	2.8096	0.160	104	52	28.8	1.16
32.455	2.7565	2.7632	0.240	132	53	36.6	3.00
32.920	2.7186	2.7253	0.160	59	53	16.4	1.03
33.875	2.6441	2.6506	0.060	117	55	32.3	0.88
34.135	2.6245	2.6310	0.100	139	56	38.6	0.89
35.680	2.5144	2.5205	0.100	139	58	38.6	1.40
36.360	2.4689	2.4750	0.120	137	59	37.9	1.93
37.770	2.3799	2.3857	0.320	22	61	6.1	0.98
38.710	2.3242	2.3300	0.320	25	62	6.9	0.90
40.350	2.2335	2.2390	0.120	38	66	10.6	0.96
42.285	2.1356	2.1409	0.060	119	69	32.9	0.93
43.350	2.0856	2.0907	0.320	37	71	10.3	1.34
44.285	2.0437	2.0487	0.320	21	71	5.9	0.85
45.415	1.9955	2.0004	0.200	41	72	11.3	0.80
46.540	1.9498	1.9546	0.320	36	74	10.0	3.92
47.260	1.9218	1.9265	0.240	28	76	7.8	1.19
48.845	1.8630	1.8676	0.960	30	77	8.4	1.76
50.480	1.8065	1.8109	0.240	25	81	6.9	0.98
51.695	1.7668	1.7712	0.320	26	83	7.2	0.95
53.115	1.7229	1.7271	0.240	26	85	7.2	1.93
53.910	1.6994	1.7035	0.400	32	86	9.0	1.85
56.395	1.6302	1.6342	0.240	38	94	10.6	1.66

- **Slag**


Sample identification: KR 002
1-Dec-2005 10:17
[counts]
300
250
200
150
100
50
0
0
10
20
30
40
50 [2Theta] 60


Sample identification: BO 845A
1-Dec-2005 9:33
[counts]
500
400
300
200
100
0
0
10
20
30
40
50 [2Theta] 60


Sample identification: BS 1264
1-Dec-2005 9:43
[counts]
1000
800
600
400
200
0
0
10
20
30
40
50 [2Theta] 60

Sample identification:	KR 002
Data measured at:	9-Aug-2005 19:44:00
Diffractometer type:	PW3710 BASED
Tube anode:	Cu
Generator tension [kV]:	40
Generator current [mA]:	30
Wavelength Alpha1 [Å]:	1.54060
Wavelength Alpha2 [Å]:	1.54439
Intensity ratio (alpha2/alpha1):	0.500
Divergence slit:	AUTOMATIC
Irradiated length [mm]:	12
Receiving slit:	0.1
Monochromator used:	YES
Start angle [°2θ]:	3.010
End angle [°2θ]:	59.990
Step size [°2θ]:	0.020
Maximum intensity:	222.0100
Time per step [s]:	1.000
Type of scan:	CONTINUOUS
Intensities converted to:	FIXED
Minimum peak tip width:	0.00
Maximum peak tip width:	1.00
Peak base width:	2.00
Minimum significance:	0.75
Number of peaks:	22

Angle [°2θ]	d-value α1 [Å]	d-value α2 [Å]	Peak width [°2θ]	Peak int [counts]	Back. int [counts]	Rel. int [%]	Signif.
11.710	7.5511	7.5697	0.480	20	11	9.1	0.79
14.135	6.2606	6.2760	0.120	98	11	44.1	0.98
16.820	5.2668	5.2798	0.240	18	12	8.3	0.98
18.325	4.8375	4.8494	0.240	22	12	10.0	1.30
21.250	4.1778	4.1881	0.160	48	13	21.4	0.96
26.685	3.3379	3.3461	0.160	50	15	22.7	1.04
27.050	3.2937	3.3018	0.060	83	16	37.3	0.81
29.440	3.0315	3.0390	0.100	164	18	73.8	1.81
30.115	2.9651	2.9724	0.100	76	18	34.1	1.16
33.270	2.6908	2.6974	0.320	26	19	11.7	1.36
35.470	2.5288	2.5350	0.060	222	21	100.0	0.94
36.030	2.4907	2.4969	0.120	96	22	43.3	1.15
39.390	2.2857	2.2913	0.200	23	15	10.4	1.45
41.820	2.1583	2.1636	0.280	69	14	31.0	4.97
43.225	2.0913	2.0965	0.280	61	14	27.4	3.83
46.825	1.9386	1.9434	0.560	16	13	7.2	1.84
47.255	1.9220	1.9267	0.480	15	13	6.9	0.95
48.515	1.8749	1.8796	0.320	13	14	5.8	1.47
53.390	1.7147	1.7189	0.400	18	16	7.9	0.93
56.075	1.6388	1.6428	0.320	7	16	3.3	0.90
57.105	1.6116	1.6156	0.560	32	16	14.6	7.02
59.020	1.5638	1.5677	0.400	8	16	3.5	1.27

Sample identification:	**BO 845A**
Data measured at:	9-Aug-2005 12:42:00
Diffractometer type:	PW3710 BASED
Tube anode:	Cu
Generator tension [kV]:	40
Generator current [mA]:	30
Wavelength Alpha1 [Å]:	1.54060
Wavelength Alpha2 [Å]:	1.54439
Intensity ratio (alpha2/alpha1):	0.500
Divergence slit:	AUTOMATIC
Irradiated length [mm]:	12
Receiving slit:	0.1
Monochromator used:	YES
Start angle [°2θ]:	3.010
End angle [°2θ]:	59.990
Step size [°2θ]:	0.020
Maximum intensity:	428.4900
Time per step [s]:	1.000
Type of scan:	CONTINUOUS
Intensities converted to:	FIXED
Minimum peak tip width:	0.00
Maximum peak tip width:	1.00
Peak base width:	2.00
Minimum significance:	0.75
Number of peaks:	28

Angle [°2θ]	d-value α1 [Å]	d-value α2 [Å]	Peak width [°2θ]	Peak int [counts]	Back. int [counts]	Rel. int [%]	Signif.
9.300	9.5018	9.5252	0.240	32	12	7.6	1.32
11.370	7.7762	7.7953	0.640	10	12	2.4	0.85
15.995	5.5366	5.5502	0.240	13	12	3.0	1.23
20.930	4.2409	4.2514	0.120	35	14	8.1	1.06
23.145	3.8398	3.8493	0.120	49	14	11.4	1.04
24.225	3.6710	3.6801	0.240	14	15	3.4	1.37
26.740	3.3312	3.3394	0.100	428	17	100.0	5.99
28.085	3.1746	3.1825	0.100	48	18	11.1	0.77
29.510	3.0245	3.0319	0.140	376	19	87.8	5.64
30.215	2.9555	2.9628	0.120	28	20	6.6	0.97
31.075	2.8757	2.8827	0.240	23	21	5.4	2.28
32.085	2.7874	2.7943	0.100	37	22	8.7	1.07
33.500	2.6728	2.6794	0.200	58	23	13.5	2.20
34.390	2.6057	2.6121	0.400	13	24	3.0	0.76
35.585	2.5209	2.5271	0.200	69	25	16.1	2.28
36.055	2.4891	2.4952	0.100	90	25	21.1	0.99
39.610	2.2735	2.2791	0.180	49	21	11.4	3.77
41.085	2.1952	2.2006	0.400	12	21	2.9	0.91
41.880	2.1553	2.1607	0.160	62	21	14.6	0.85
42.200	2.1397	2.1450	0.240	74	21	17.3	2.45
43.330	2.0865	2.0917	0.200	53	21	12.4	2.60
46.740	1.9419	1.9467	0.200	14	20	3.4	1.71
47.610	1.9085	1.9131	0.080	74	20	17.3	1.20
48.615	1.8713	1.8759	0.240	46	20	10.8	4.36
49.465	1.8411	1.8457	0.320	10	20	2.4	1.45
53.620	1.7079	1.7121	0.960	3	18	0.7	1.34
55.270	1.6607	1.6648	0.480	4	18	0.9	1.56
57.500	1.6015	1.6054	0.280	18	18	4.1	2.23

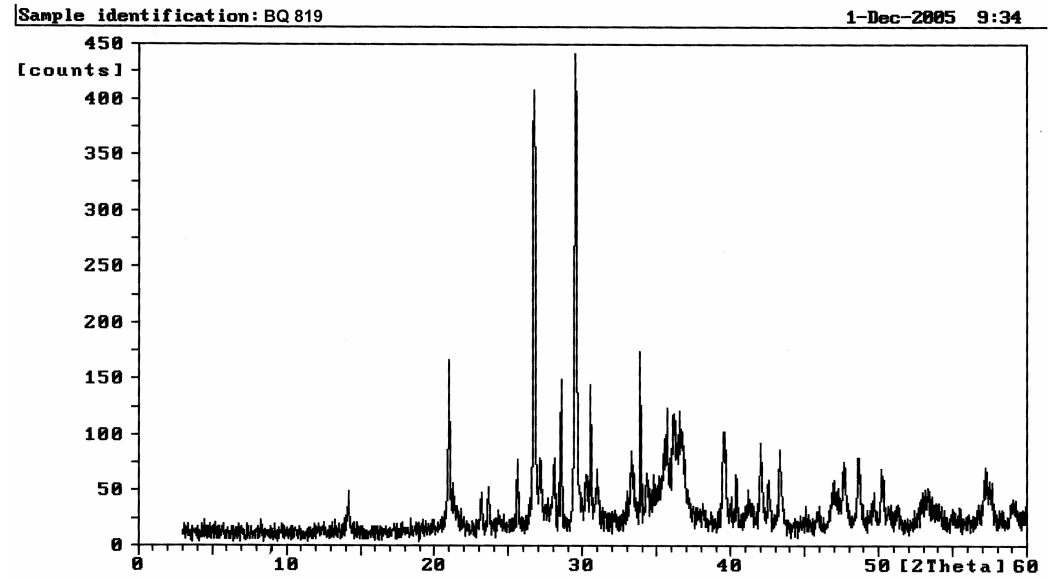
Sample identification: **BS 1264**
Data measured at: 9-Aug-2005 18:08:00

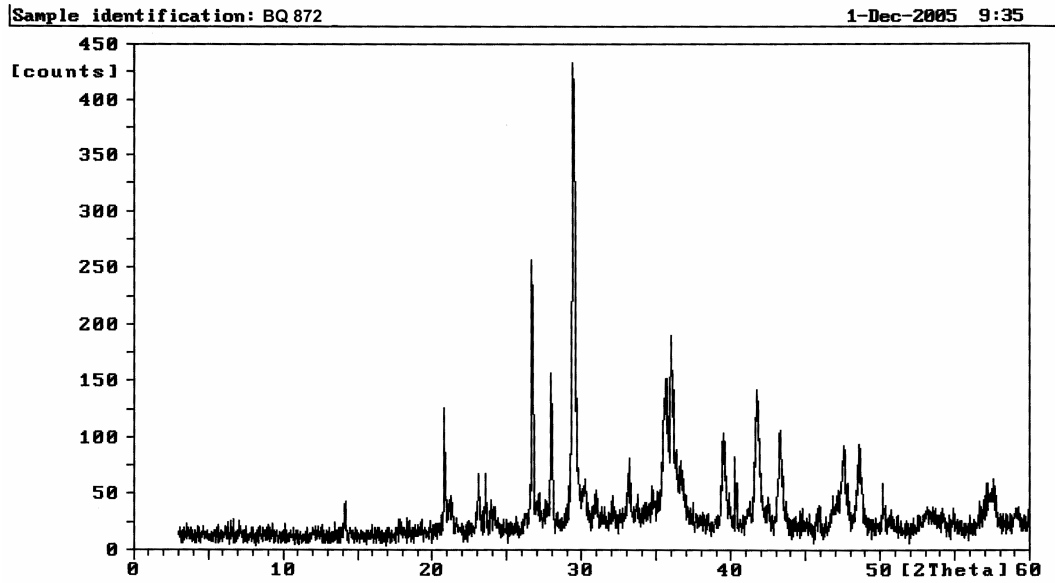
Diffractometer type: PW3710 BASED
Tube anode: Cu
Generator tension [kV]: 40
Generator current [mA]: 30
Wavelength Alpha1 [Å]: 1.54060
Wavelength Alpha2 [Å]: 1.54439
Intensity ratio (alpha2/alpha1): 0.500
Divergence slit: AUTOMATIC
Irradiated length [mm]: 12
Receiving slit: 0.1
Monochromator used: YES

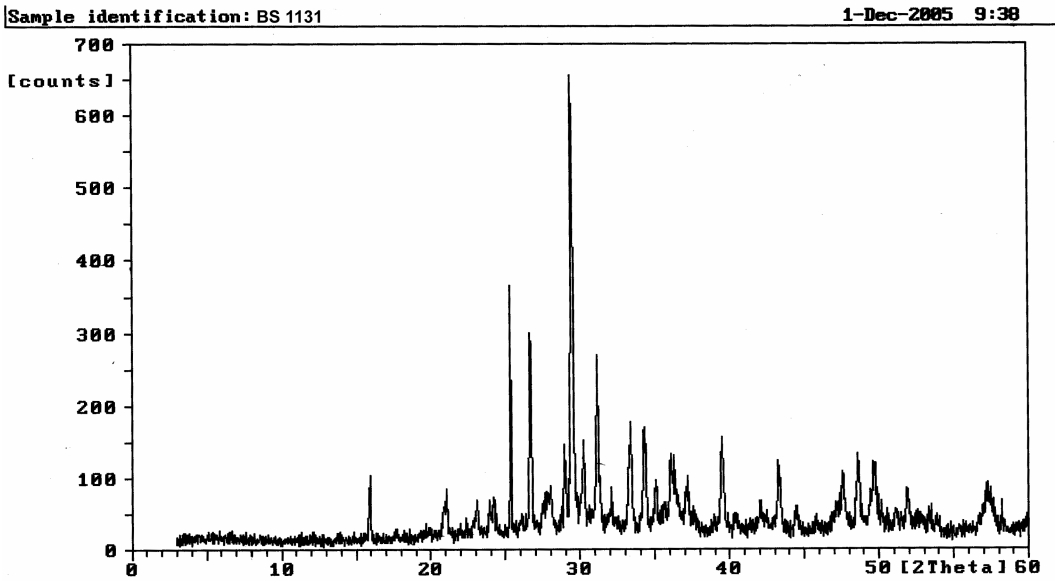
Start angle [°2θ]: 3.010
End angle [°2θ]: 59.990
Step size [°2θ]: 0.020
Maximum intensity: 1043.290
Time per step [s]: 1.000
Type of scan: CONTINUOUS
Intensities converted to: FIXED

Minimum peak tip width: 0.00
Maximum peak tip width: 1.00
Peak base width: 2.00
Minimum significance: 0.75
Number of peaks: 30

Angle [°2θ]	d-value α1 [Å]	d-value α2 [Å]	Peak width [°2θ]	Peak int [counts]	Back. int [counts]	Rel. int [%]	Signif.
13.745	6.4374	6.4532	0.060	40	12	3.8	1.71
20.910	4.2449	4.2554	0.080	96	19	9.2	1.05
23.125	3.8431	3.8526	0.080	79	21	7.6	0.89
23.615	3.7645	3.7737	0.100	159	22	15.2	3.70
26.690	3.3373	3.3455	0.120	471	24	45.1	8.08
27.950	3.1897	3.1975	0.160	48	24	4.6	1.30
29.445	3.0310	3.0385	0.160	1043	24	100.0	16.46
30.170	2.9598	2.9671	0.160	49	24	4.7	1.09
30.995	2.8829	2.8900	0.100	369	24	35.3	4.49
32.155	2.7815	2.7883	0.100	58	24	5.5	0.81
32.615	2.7433	2.7501	0.160	49	24	4.7	1.21
33.595	2.6655	2.6720	0.160	37	24	3.6	1.11
34.295	2.6127	2.6191	0.240	21	24	2.0	1.53
35.505	2.5264	2.5326	0.200	100	24	9.6	3.97
36.055	2.4891	2.4952	0.120	81	24	7.8	1.01
36.605	2.4529	2.4590	0.120	37	24	3.6	0.83
39.485	2.2804	2.2860	0.100	102	24	9.8	1.35
41.165	2.1911	2.1965	0.240	34	25	3.2	2.71
42.180	2.1407	2.1460	0.080	94	25	9.0	1.18
43.245	2.0904	2.0956	0.120	110	25	10.6	1.89
45.860	1.9771	1.9820	0.240	14	25	1.3	2.06
47.585	1.9094	1.9141	0.200	71	25	6.8	2.18
48.215	1.8859	1.8905	0.120	34	25	3.2	1.25
48.590	1.8722	1.8768	0.240	59	25	5.7	3.55
50.155	1.8174	1.8219	0.080	26	25	2.5	0.92
53.495	1.7116	1.7158	0.060	18	23	1.7	0.75
54.950	1.6696	1.6737	0.160	9	23	0.9	1.79
56.665	1.6231	1.6271	0.240	16	23	1.5	0.81
57.555	1.6001	1.6040	0.200	27	24	2.6	1.47
58.720	1.5711	1.5750	0.240	8	25	0.8	1.23


Sample identification: BQ 819
1-Dec-2005 9:34
[counts]
450
400
350
300
250
200
150
100
50
0
0
10
20
30
40
50 [2Theta] 60


Sample identification: BQ 872
1-Dec-2005 9:35
[counts]
450
400
350
300
250
200
150
100
50
0
0
10
20
30
40
50 [2Theta] 60


Sample identification: BS 1131
1-Dec-2005 9:38
[counts]
700
600
500
400
300
200
100
0
0
10
20
30
40
50 [2Theta] 60

Sample identification: BQ 819
Data measured at: 9-Aug-2005 13:30:00

Diffractometer type: PW3710 BASED
Tube anode: Cu
Generator tension [kV]: 40
Generator current [mA]: 30
Wavelength Alpha1 [Å]: 1.54060
Wavelength Alpha2 [Å]: 1.54439
Intensity ratio (alpha2/alpha1): 0.500
Divergence slit: AUTOMATIC
Irradiated length [mm]: 12
Receiving slit: 0.1
Monochromator used: YES

Start angle [°2θ]: 3.010
End angle [°2θ]: 59.990
Step size [°2θ]: 0.020
Maximum intensity: 376.3600
Time per step [s]: 1.000
Type of scan: CONTINUOUS
Intensities converted to: FIXED

Minimum peak tip width: 0.00
Maximum peak tip width: 1.00
Peak base width: 2.00
Minimum significance: 0.75
Number of peaks: 35

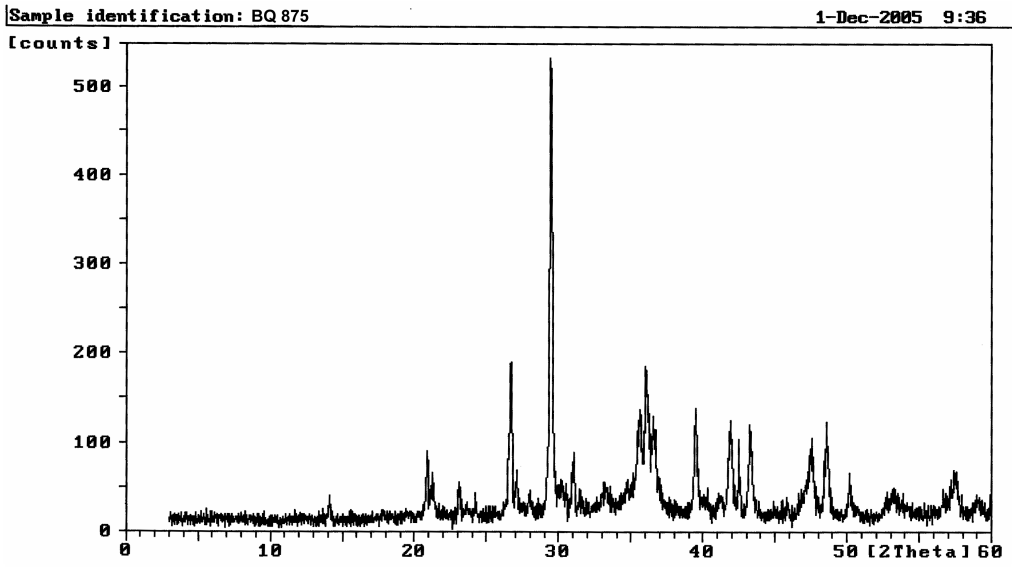
Angle [°2θ]	d-value α1 [Å]	d-value α2 [Å]	Peak width [°2θ]	Peak int [counts]	Back. int [counts]	Rel. int [%]	Signif.
14.185	6.2387	6.2540	0.160	46	11	12.3	1.31
20.970	4.2329	4.2433	0.100	190	14	50.6	3.21
23.170	3.8357	3.8452	0.080	37	14	9.9	0.84
23.645	3.7598	3.7690	0.040	44	14	11.6	1.05
25.570	3.4809	3.4895	0.100	64	15	17.0	1.78
26.680	3.3385	3.3468	0.140	376	15	100.0	9.25
27.145	3.2824	3.2905	0.120	59	15	15.8	2.75
28.075	3.1757	3.1836	0.160	50	15	13.4	1.56
28.530	3.1261	3.1338	0.060	125	15	33.3	3.80
29.510	3.0245	3.0319	0.140	320	15	85.1	9.38
30.260	2.9512	2.9585	0.160	35	15	9.2	1.83
30.520	2.9267	2.9339	0.060	117	15	31.0	1.20
31.040	2.8788	2.8859	0.240	29	15	7.7	2.42
33.290	2.6892	2.6958	0.200	45	16	11.9	1.63
33.880	2.6437	2.6502	0.060	90	16	24.0	1.21
34.340	2.6093	2.6158	0.160	35	16	9.2	1.05
35.615	2.5188	2.5250	0.240	56	16	14.9	0.92
36.170	2.4814	2.4875	0.200	74	16	19.7	2.02
36.740	2.4442	2.4502	0.200	58	16	15.3	1.09
39.535	2.2776	2.2832	0.240	55	17	14.5	5.22
40.405	2.2306	2.2360	0.120	18	17	4.9	2.23
41.215	2.1886	2.1940	0.320	14	17	3.6	0.84
42.010	2.1490	2.1543	0.060	49	17	13.0	1.35
42.525	2.1241	2.1294	0.100	27	17	7.2	0.97
43.300	2.0879	2.0930	0.100	37	17	9.9	1.28
45.890	1.9759	1.9808	0.240	6	18	1.5	0.94
46.970	1.9330	1.9377	0.240	15	18	4.0	0.99
47.700	1.9051	1.9097	0.240	24	18	6.4	2.51
48.625	1.8710	1.8756	0.100	34	18	8.9	1.27
49.705	1.8328	1.8373	0.240	9	18	2.4	1.38
50.205	1.8157	1.8202	0.100	28	18	7.5	1.06
51.270	1.7805	1.7849	0.240	7	18	1.9	1.35
53.135	1.7223	1.7265	0.640	12	19	3.1	1.83
57.190	1.6094	1.6134	0.240	16	20	4.3	0.76
59.055	1.5630	1.5668	0.320	6	20	1.7	1.02

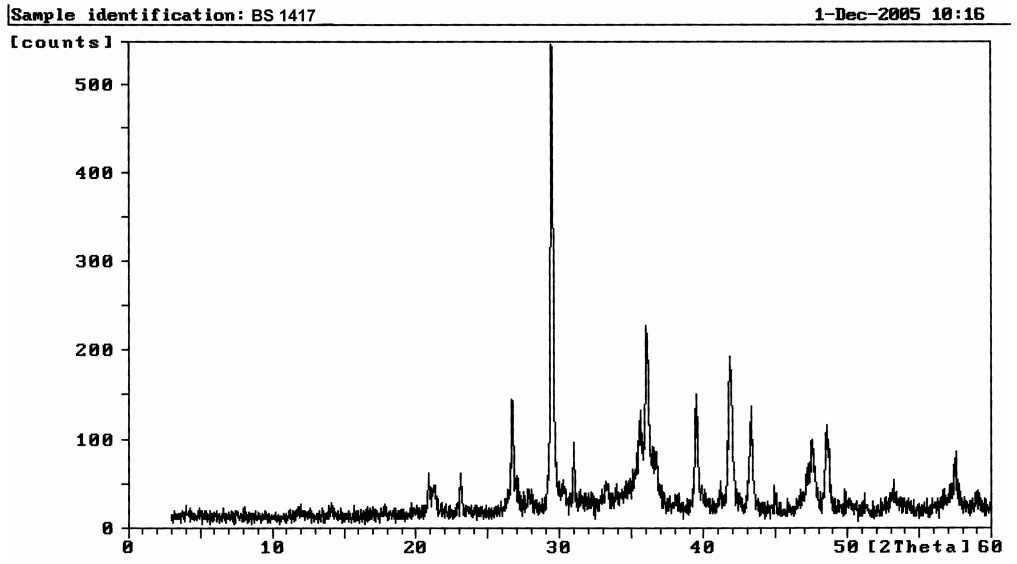
Sample identification:	BQ 872
Data measured at:	9-Aug-2005 14:18:00
Diffractometer type:	PW3710 BASED
Tube anode:	Cu
Generator tension [kV]:	40
Generator current [mA]:	30
Wavelength Alpha1 [Å]:	1.54060
Wavelength Alpha2 [Å]:	1.54439
Intensity ratio (alpha2/alpha1):	0.500
Divergence slit:	AUTOMATIC
Irradiated length [mm]:	12
Receiving slit:	0.1
Monochromator used:	YES
Start angle [°2θ]:	3.010
End angle [°2θ]:	59.990
Step size [°2θ]:	0.020
Maximum intensity:	424.3600
Time per step [s]:	1.000
Type of scan:	CONTINUOUS
Intensities converted to:	FIXED
Minimum peak tip width:	0.00
Maximum peak tip width:	1.00
Peak base width:	2.00
Minimum significance:	0.75
Number of peaks:	33

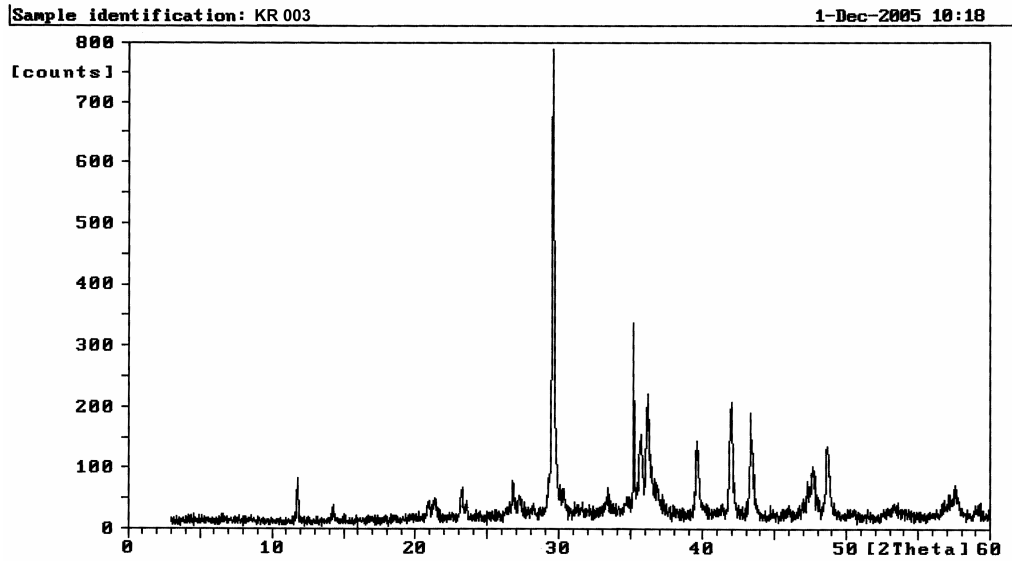
Angle [°2θ]	d-value α1 [Å]	d-value α2 [Å]	Peak width [°2θ]	Peak int [counts]	Back. int [counts]	Rel. int [%]	Signif.
6.740	13.1040	13.1362	0.960	14	12	3.2	1.02
9.190	9.6153	9.6390	0.800	9	12	2.1	0.76
12.265	7.2107	7.2284	0.640	6	11	1.5	0.76
14.170	6.2453	6.2606	0.060	67	12	15.8	0.84
17.820	4.9734	4.9857	0.480	10	12	2.4	0.79
18.495	4.7934	4.8052	0.240	9	12	2.1	0.83
20.825	4.2621	4.2726	0.060	102	12	24.0	3.83
21.265	4.1749	4.1851	0.160	35	12	8.2	0.87
23.105	3.8464	3.8559	0.160	55	13	12.9	1.36
23.550	3.7747	3.7840	0.080	66	13	15.5	0.86
24.050	3.6973	3.7064	0.240	18	13	4.4	1.05
26.655	3.3416	3.3498	0.080	276	13	64.9	2.31
27.945	3.1902	3.1981	0.080	149	14	35.1	1.19
29.440	3.0315	3.0390	0.100	424	14	100.0	2.80
29.530	3.0225	3.0299	0.060	324	14	76.4	0.88
30.250	2.9522	2.9594	0.200	41	14	9.7	0.87
31.020	2.8806	2.8877	0.120	35	14	8.2	0.97
32.090	2.7870	2.7938	0.240	18	14	4.4	1.00
33.115	2.7030	2.7097	0.160	42	14	10.0	1.02
35.540	2.5239	2.5302	0.320	96	14	22.6	5.38
35.985	2.4937	2.4999	0.160	132	14	31.2	1.82
36.705	2.4465	2.4525	0.160	37	15	8.8	0.87
39.490	2.2801	2.2857	0.200	56	15	13.3	2.86
40.270	2.2377	2.2432	0.040	49	15	11.5	3.88
41.730	2.1627	2.1681	0.280	81	16	19.1	5.77
43.260	2.0897	2.0949	0.280	52	16	12.2	5.20
45.825	1.9786	1.9834	0.240	9	16	2.1	0.90
47.595	1.9090	1.9137	0.120	45	17	10.6	2.39
48.600	1.8719	1.8765	0.160	40	17	9.4	1.13
50.150	1.8176	1.8221	0.160	17	17	4.0	0.97
53.260	1.7185	1.7228	0.800	7	19	1.6	1.92
57.600	1.5989	1.6029	0.320	15	19	3.6	0.97
59.120	1.5614	1.5653	0.320	7	19	1.7	0.96

Sample identification:	**BS 1131**
Data measured at:	9-Aug-2005 16:32:00
Diffractometer type:	PW3710 BASED
Tube anode:	Cu
Generator tension [kV]:	40
Generator current [mA]:	30
Wavelength Alpha1 [Å]:	1.54060
Wavelength Alpha2 [Å]:	1.54439
Intensity ratio (alpha2/alpha1):	0.500
Divergence slit:	AUTOMATIC
Irradiated length [mm]:	12
Receiving slit:	0.1
Monochromator used:	YES
Start angle [°2θ]:	3.010
End angle [°2θ]:	59.990
Step size [°2θ]:	0.020
Maximum intensity:	635.0400
Time per step [s]:	1.000
Type of scan:	CONTINUOUS
Intensities converted to:	FIXED
Minimum peak tip width:	0.00
Maximum peak tip width:	1.00
Peak base width:	2.00
Minimum significance:	0.75
Number of peaks:	38

Angle [°2θ]	d-value α1 [Å]	d-value α2 [Å]	Peak width [°2θ]	Peak int [counts]	Back. int [counts]	Rel. int [%]	Signif.
13.875	6.3774	6.3931	0.320	14	12	2.3	0.92
15.950	5.5521	5.5657	0.060	161	12	25.4	3.72
17.700	5.0069	5.0192	0.480	15	12	2.4	0.96
19.965	4.4437	4.4546	0.200	20	13	3.2	0.90
21.065	4.2141	4.2244	0.120	71	14	11.1	1.98
22.395	3.9667	3.9765	0.120	38	14	6.1	1.24
23.105	3.8464	3.8559	0.100	69	14	10.8	0.85
23.945	3.7133	3.7225	0.120	55	14	8.6	1.31
24.235	3.6695	3.6786	0.120	66	14	10.3	0.77
25.315	3.5154	3.5240	0.100	396	14	62.4	5.81
26.670	3.3398	3.3480	0.140	313	15	49.3	7.33
28.035	3.1802	3.1880	0.120	64	15	10.1	1.01
28.975	3.0791	3.0867	0.100	130	15	20.5	1.79
29.445	3.0310	3.0385	0.120	635	16	100.0	6.10
30.260	2.9512	2.9585	0.060	130	16	20.5	1.35
31.155	2.8685	2.8755	0.140	190	16	30.0	4.10
32.125	2.7840	2.7909	0.120	52	16	8.2	3.21
33.400	2.6806	2.6872	0.080	139	17	21.9	1.21
34.265	2.6149	2.6213	0.080	123	17	19.4	1.04
35.070	2.5567	2.5630	0.160	61	17	9.6	1.86
36.065	2.4884	2.4945	0.080	88	18	13.9	0.84
36.295	2.4732	2.4792	0.060	94	18	14.8	1.00
37.190	2.4157	2.4216	0.160	61	18	9.6	1.24
39.510	2.2790	2.2846	0.160	96	18	15.1	4.56
40.400	2.2308	2.2363	0.240	18	18	2.9	1.86
42.045	2.1473	2.1526	0.160	27	19	4.3	0.92
43.235	2.0909	2.0960	0.060	69	19	10.8	1.16
44.430	2.0374	2.0424	0.240	21	19	3.3	2.34
45.800	1.9796	1.9844	0.320	11	20	1.7	0.85
47.615	1.9083	1.9130	0.200	50	20	7.9	2.23
48.635	1.8706	1.8752	0.240	56	21	8.9	4.75
49.690	1.8333	1.8378	0.200	56	21	8.9	1.76
51.065	1.7871	1.7915	0.120	18	21	2.9	0.78
51.875	1.7611	1.7655	0.200	32	21	5.1	1.79
53.390	1.7147	1.7189	0.160	12	22	1.9	0.84
53.890	1.6999	1.7041	0.160	9	22	1.4	1.32
57.295	1.6067	1.6107	0.400	27	26	4.3	1.97
58.240	1.5829	1.5868	0.060	16	26	2.5	1.35


Sample identification: BQ 875
1-Dec-2005 9:36
[counts]
500
400
300
200
100
0
0
10
20
30
40
50 [2Theta] 60


Sample identification: BS 1417
1-Dec-2005 10:16
[counts]
500
400
300
200
100
0
0
10
20
30
40
50 [2Theta] 60


Sample identification: KR 003
1-Dec-2005 10:18
800
[counts]
700
600
500
400
300
200
100
0
0
10
20
30
40
50 [2Theta] 60

Sample identification:	**BQ 875**
Data measured at:	9-Aug-2005 15:44:00
Diffractometer type:	PW3710 BASED
Tube anode:	Cu
Generator tension [kV]:	40
Generator current [mA]:	30
Wavelength Alpha1 [Å]:	1.54060
Wavelength Alpha2 [Å]:	1.54439
Intensity ratio (alpha2/alpha1):	0.500
Divergence slit:	AUTOMATIC
Irradiated length [mm]:	12
Receiving slit:	0.1
Monochromator used:	YES
Start angle [°2θ]:	3.010
End angle [°2θ]:	59.990
Step size [°2θ]:	0.020
Maximum intensity:	475.2400
Time per step [s]:	1.000
Type of scan:	CONTINUOUS
Intensities converted to:	FIXED
Minimum peak tip width:	0.00
Maximum peak tip width:	1.00
Peak base width:	2.00
Minimum significance:	0.75
Number of peaks:	27

Angle [°2θ]	d-value α1 [Å]	d-value α2 [Å]	Peak width [°2θ]	Peak int [counts]	Back. int [counts]	Rel. int [%]	Signif.
14.140	6.2584	6.2738	0.160	44	12	9.2	1.10
20.890	4.2490	4.2594	0.100	104	16	21.9	1.88
21.285	4.1710	4.1813	0.160	55	16	11.5	0.86
23.110	3.8456	3.8550	0.160	42	16	8.9	1.50
24.255	3.6666	3.6756	0.120	21	16	4.5	1.09
26.680	3.3385	3.3468	0.080	193	16	40.7	1.43
27.110	3.2866	3.2946	0.200	45	16	9.4	1.37
28.010	3.1830	3.1908	0.240	20	16	4.3	1.08
29.480	3.0275	3.0350	0.200	475	16	100.0	15.79
31.055	2.8775	2.8845	0.200	56	16	11.8	2.81
33.175	2.6983	2.7049	0.480	25	16	5.3	2.03
35.635	2.5174	2.5236	0.120	86	16	18.2	0.89
36.045	2.4897	2.4959	0.200	128	16	26.9	2.63
36.535	2.4575	2.4635	0.160	85	16	17.8	1.16
39.525	2.2782	2.2838	0.140	77	16	16.3	2.56
41.215	2.1886	2.1940	0.240	15	16	3.2	1.55
41.895	2.1546	2.1599	0.280	67	16	14.1	5.63
42.460	2.1272	2.1325	0.060	64	16	13.5	0.76
43.210	2.0920	2.0972	0.080	74	16	15.6	0.77
45.835	1.9781	1.9830	0.200	8	16	1.8	1.17
47.625	1.9079	1.9126	0.280	45	16	9.4	3.38
48.605	1.8717	1.8763	0.080	67	16	14.1	0.97
50.170	1.8169	1.8214	0.080	28	16	5.9	1.13
53.165	1.7214	1.7256	0.320	14	16	3.0	1.11
56.030	1.6400	1.6440	0.120	5	16	1.1	0.82
57.555	1.6001	1.6040	0.560	22	16	4.6	3.54
59.080	1.5624	1.5662	0.240	10	16	2.2	0.87

Sample identification: **BS 1417**
Data measured at: 9-Aug-2005 18:56:00

Diffractometer type: PW3710 BASED
Tube anode: Cu
Generator tension [kV]: 40
Generator current [mA]: 30
Wavelength Alpha1 [Å]: 1.54060
Wavelength Alpha2 [Å]: 1.54439
Intensity ratio (alpha2/alpha1): 0.500
Divergence slit: AUTOMATIC
Irradiated length [mm]: 12
Receiving slit: 0.1
Monochromator used: YES

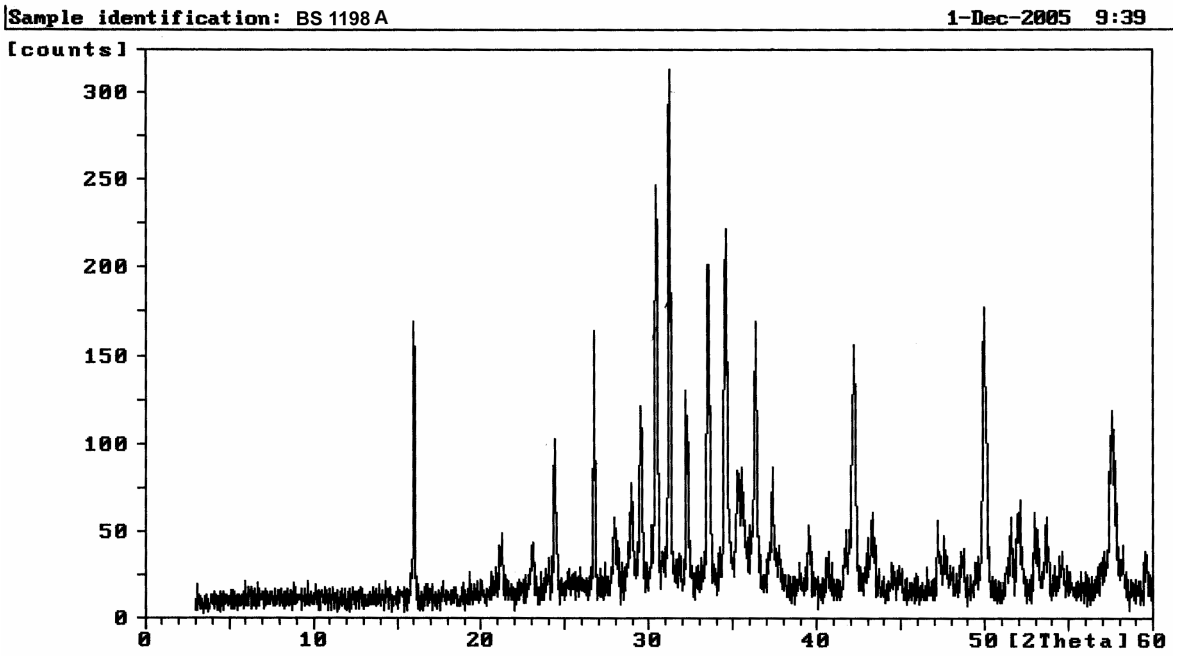
Start angle [°2θ]: 3.010
End angle [°2θ]: 59.990
Step size [°2θ]: 0.020
Maximum intensity: 533.6100
Time per step [s]: 1.000
Type of scan: CONTINUOUS
Intensities converted to: FIXED

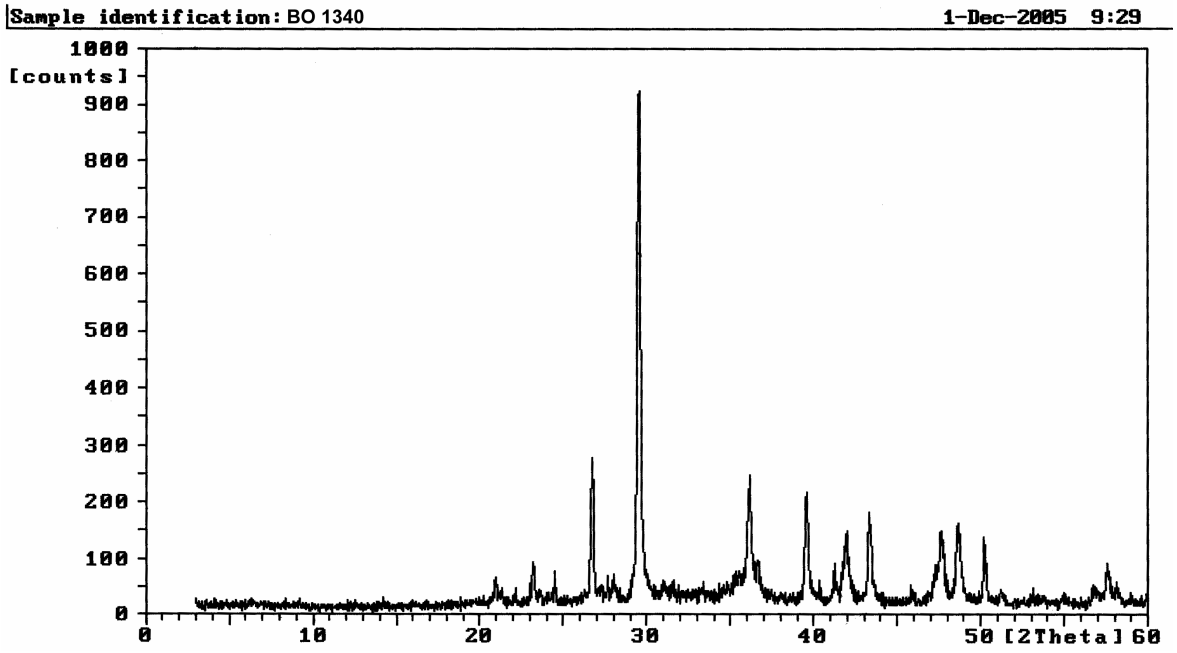
Minimum peak tip width: 0.00
Maximum peak tip width: 1.00
Peak base width: 2.00
Minimum significance: 0.75
Number of peaks: 27

Angle [°2θ]	d-value α1 [Å]	d-value α2 [Å]	Peak width [°2θ]	Peak int [counts]	Back. int [counts]	Rel. int [%]	Signif.
11.885	7.4403	7.4586	0.640	18	12	3.3	0.89
14.150	6.2540	6.2694	0.320	22	12	4.1	0.92
17.780	4.9845	4.9968	0.240	11	13	2.0	0.76
20.870	4.2530	4.2634	0.060	46	16	8.7	0.80
21.295	4.1691	4.1793	0.200	32	16	6.1	0.82
22.090	4.0208	4.0307	0.120	14	16	2.7	0.91
23.115	3.8447	3.8542	0.120	42	17	7.9	3.32
26.670	3.3398	3.3480	0.120	142	17	26.5	2.31
27.890	3.1964	3.2043	0.160	19	17	3.6	1.01
29.455	3.0300	3.0375	0.220	534	18	100.0	17.56
30.985	2.8838	2.8909	0.140	64	18	12.0	2.78
33.290	2.6892	2.6958	0.240	27	18	5.1	0.82
35.585	2.5209	2.5271	0.200	74	18	13.9	1.17
36.045	2.4897	2.4959	0.200	164	18	30.7	3.92
36.750	2.4436	2.4496	0.120	50	18	9.4	0.88
39.470	2.2812	2.2868	0.080	85	18	15.9	1.29
39.590	2.2746	2.2802	0.040	77	18	14.5	0.75
41.175	2.1906	2.1960	0.160	18	18	3.3	0.89
41.820	2.1583	2.1636	0.240	117	18	21.9	6.07
43.255	2.0900	2.0951	0.200	71	18	13.2	3.58
44.945	2.0152	2.0202	0.120	11	18	2.0	1.28
47.550	1.9107	1.9154	0.240	48	18	8.9	2.65
48.545	1.8739	1.8785	0.200	52	18	9.7	2.68
50.075	1.8201	1.8246	0.480	5	18	0.9	1.15
53.215	1.7199	1.7241	0.320	12	19	2.3	0.99
57.505	1.6014	1.6053	0.160	24	23	4.5	1.31
59.010	1.5641	1.5679	0.400	6	22	1.1	1.42

Sample identification:	**KR 003**
Data measured at:	9-Aug-2005 20:32:00
Diffractometer type:	PW3710 BASED
Tube anode:	Cu
Generator tension [kV]:	40
Generator current [mA]:	30
Wavelength Alpha1 [Å]:	1.54060
Wavelength Alpha2 [Å]:	1.54439
Intensity ratio (alpha2/alpha1):	0.500
Divergence slit:	AUTOMATIC
Irradiated length [mm]:	12
Receiving slit:	0.1
Monochromator used:	YES
Start angle [°2θ]:	3.010
End angle [°2θ]:	59.990
Step size [°2θ]:	0.020
Maximum intensity:	702.2500
Time per step [s]:	1.000
Type of scan:	CONTINUOUS
Intensities converted to:	FIXED
Minimum peak tip width:	0.00
Maximum peak tip width:	1.00
Peak base width:	2.00
Minimum significance:	0.75
Number of peaks:	25

Angle [°2θ]	d-value α1 [Å]	d-value α2 [Å]	Peak width [°2θ]	Peak int [counts]	Back. int [counts]	Rel. int [%]	Signif.
11.785	7.5032	7.5217	0.080	174	11	24.8	1.46
14.225	6.2212	6.2365	0.160	30	11	4.3	0.76
20.950	4.2369	4.2473	0.160	35	18	5.0	0.99
21.380	4.1527	4.1629	0.200	29	18	4.2	0.84
23.205	3.8300	3.8395	0.140	53	18	7.6	1.93
23.560	3.7731	3.7824	0.120	28	18	4.0	0.94
26.770	3.3275	3.3357	0.060	52	22	7.4	0.97
27.205	3.2753	3.2834	0.200	34	23	4.8	1.41
28.165	3.1658	3.1736	0.240	8	24	1.2	0.76
29.595	3.0160	3.0234	0.140	702	27	100.0	10.32
33.405	2.6802	2.6868	0.320	18	26	2.6	1.22
35.155	2.5507	2.5570	0.060	246	28	35.1	2.05
35.660	2.5157	2.5219	0.200	98	29	14.0	2.94
36.130	2.4841	2.4902	0.120	149	29	21.2	1.92
39.595	2.2743	2.2799	0.140	81	23	11.5	2.38
41.970	2.1509	2.1562	0.100	132	22	18.8	1.58
43.310	2.0874	2.0926	0.060	104	21	14.8	2.91
45.545	1.9901	1.9950	0.120	5	20	0.7	0.77
46.000	1.9714	1.9763	0.240	8	19	1.2	0.78
47.710	1.9047	1.9094	0.200	48	19	6.8	1.98
48.645	1.8702	1.8748	0.120	71	19	10.0	2.22
50.395	1.8093	1.8138	0.480	4	18	0.6	1.09
53.400	1.7144	1.7186	0.480	8	19	1.2	1.24
57.635	1.5981	1.6020	0.320	18	22	2.5	1.43
59.040	1.5633	1.5672	0.400	4	22	0.6	0.95


Sample identification: BS 1198 A
1-Dec-2005 9:39
[counts]
300
250
200
150
100
50
0
0
10
20
30
40
50 [2Theta] 60


Sample identification: BO 1340
1-Dec-2005 9:29
1000
[counts]
900
800
700
600
500
400
300
200
100
0
0
10
20
30
40
50 [2Theta] 60

Sample identification:	BS 1198 A
Data measured at:	9-Aug-2005 17:20:00
Diffractometer type:	PW3710 BASED
Tube anode:	Cu
Generator tension [kV]:	40
Generator current [mA]:	30
Wavelength Alpha1 [Å]:	1.54060
Wavelength Alpha2 [Å]:	1.54439
Intensity ratio (alpha2/alpha1):	0.500
Divergence slit:	AUTOMATIC
Irradiated length [mm]:	12
Receiving slit:	0.1
Monochromator used:	YES
Start angle [°2θ]:	3.010
End angle [°2θ]:	59.990
Step size [°2θ]:	0.020
Maximum intensity:	278.8900
Time per step [s]:	1.000
Type of scan:	CONTINUOUS
Intensities converted to:	FIXED
Minimum peak tip width:	0.00
Maximum peak tip width:	1.00
Peak base width:	2.00
Minimum significance:	0.75
Number of peaks:	39

Angle [°2θ]	d-value α1 [Å]	d-value α2 [Å]	Peak width [°2θ]	Peak int [counts]	Back. int [counts]	Rel. int [%]	Signif.
3.175	27.8051	27.8735	0.240	18	8	6.3	0.76
16.010	5.5314	5.5450	0.080	279	11	100.0	3.39
18.475	4.7986	4.8104	0.240	7	11	2.4	0.86
21.245	4.1788	4.1890	0.240	40	11	14.2	2.33
23.075	3.8513	3.8608	0.160	38	11	13.8	1.34
24.370	3.6495	3.6585	0.060	106	11	38.0	2.21
25.190	3.5325	3.5412	0.480	13	11	4.6	0.91
26.705	3.3355	3.3437	0.100	156	11	56.0	3.34
27.940	3.1908	3.1986	0.120	44	11	15.6	1.67
28.960	3.0807	3.0883	0.160	61	11	21.8	1.38
29.510	3.0245	3.0319	0.120	88	11	31.7	2.90
30.380	2.9398	2.9471	0.080	159	11	56.9	1.09
30.470	2.9314	2.9386	0.040	219	11	78.5	3.49
30.845	2.8966	2.9037	0.640	25	11	9.0	2.29
31.215	2.8631	2.8701	0.100	253	11	90.6	3.44
32.225	2.7756	2.7824	0.060	96	11	34.4	2.24
33.560	2.6682	2.6748	0.080	146	11	52.5	1.38
34.580	2.5918	2.5982	0.080	166	11	59.7	1.34
35.290	2.5413	2.5475	0.120	53	11	19.1	1.31
36.355	2.4692	2.4753	0.120	121	11	43.4	2.33
37.355	2.4054	2.4113	0.080	44	11	15.6	0.83
39.510	2.2790	2.2846	0.160	27	11	9.7	1.39
40.640	2.2182	2.2237	0.200	14	11	4.9	1.45
41.705	2.1640	2.1693	0.200	18	11	6.3	0.84
42.215	2.1390	2.1443	0.080	100	11	35.9	1.88
43.250	2.0902	2.0953	0.160	27	11	9.7	1.18
44.685	2.0263	2.0313	0.480	7	11	2.6	0.93
47.190	1.9245	1.9292	0.160	20	11	7.3	1.13
48.580	1.8726	1.8772	0.200	14	11	4.9	1.25
49.965	1.8239	1.8284	0.060	96	11	34.4	2.66
50.105	1.8191	1.8236	0.080	71	11	25.3	0.83
51.520	1.7724	1.7768	0.160	22	11	7.9	1.14
51.950	1.7588	1.7631	0.240	24	11	8.6	2.12
52.935	1.7283	1.7326	0.200	18	11	6.3	1.55
53.595	1.7086	1.7128	0.160	17	11	6.0	0.76
54.645	1.6782	1.6823	0.280	10	11	3.7	1.47
57.595	1.5991	1.6030	0.280	50	11	18.1	4.58
58.240	1.5829	1.5868	0.240	11	11	3.9	0.82
59.570	1.5507	1.5545	0.120	10	11	3.7	1.47

Sample identification:	**BO 1349**
Data measured at:	9-Aug-2005 11:54:00
Diffractometer type:	PW3710 BASED
Tube anode:	Cu
Generator tension [kV]:	40
Generator current [mA]:	30
Wavelength Alpha1 [Å]:	1.54060
Wavelength Alpha2 [Å]:	1.54439
Intensity ratio (alpha2/alpha1):	0.500
Divergence slit:	AUTOMATIC
Irradiated length [mm]:	12
Receiving slit:	0.1
Monochromator used:	YES
Start angle [°2θ]:	3.010
End angle [°2θ]:	59.990
Step size [°2θ]:	0.020
Maximum intensity:	852.6400
Time per step [s]:	1.000
Type of scan:	CONTINUOUS
Intensities converted to:	FIXED
Minimum peak tip width:	0.00
Maximum peak tip width:	1.00
Peak base width:	2.00
Minimum significance:	0.75
Number of peaks:	29

Angle [°2θ]	d-value α1 [Å]	d-value α2 [Å]	Peak width [°2θ]	Peak int [counts]	Back. int [counts]	Rel. int [%]	Signif.
6.375	13.8534	13.8875	0.480	31	14	3.7	0.81
9.195	9.6101	9.6337	0.480	18	12	2.2	0.90
12.250	7.2194	7.2372	0.080	26	12	3.1	0.82
14.135	6.2606	6.2760	0.480	12	12	1.4	1.07
20.960	4.2349	4.2453	0.120	56	20	6.6	2.97
23.225	3.8268	3.8362	0.080	92	21	10.8	0.96
24.475	3.6341	3.6430	0.060	52	22	6.1	1.47
26.750	3.3300	3.3382	0.180	256	25	30.0	8.75
27.655	3.2230	3.2309	0.100	42	27	5.0	1.20
27.995	3.1846	3.1925	0.160	34	27	3.9	0.95
29.560	3.0195	3.0269	0.180	853	34	100.0	16.45
33.275	2.6904	2.6970	0.240	8	32	1.0	0.85
35.410	2.5329	2.5391	0.400	28	30	3.3	1.24
36.140	2.4834	2.4895	0.120	151	29	17.7	1.42
36.675	2.4484	2.4544	0.160	46	28	5.4	2.31
39.575	2.2754	2.2810	0.160	142	26	16.6	4.31
40.370	2.2324	2.2379	0.120	18	25	2.2	0.89
41.215	2.1886	2.1940	0.080	36	25	4.2	0.90
41.965	2.1512	2.1565	0.240	74	24	8.7	3.64
43.300	2.0879	2.0930	0.240	100	23	11.7	6.91
45.910	1.9751	1.9799	0.240	11	21	1.3	0.80
47.630	1.9077	1.9124	0.120	77	20	9.1	1.20
48.600	1.8719	1.8765	0.280	76	20	8.9	6.81
50.175	1.8167	1.8212	0.100	66	19	7.7	2.33
51.200	1.7827	1.7871	0.160	14	18	1.7	0.91
54.900	1.6710	1.6751	0.200	6	19	0.7	0.78
56.790	1.6198	1.6238	0.320	12	18	1.4	1.29
57.540	1.6005	1.6044	0.120	30	18	3.5	0.98
58.190	1.5841	1.5880	0.240	12	19	1.4	1.51

Appendix 11: Full & restricted EDX dataset coins

- **Obols**

Complete dataset												Restricted dataset			
Reg. nr.	**Class**	**Fe**	**Cu**	**Ag**	**Sn**	**Pb**	***2-sig Fe***	***2-sig Cu***	***2-sig Ag***	***2-sig Sn***	***2-sig Pb***	**Cu**	**Ag**	**Sn**	**Pb**
BM 028	?	0,24	37,35	60,71	-	1,70	*0,04*	*0,64*	*0,82*	-	*0,44*	38,25	61,75	-	-
BQ 125	?	1,18	1,68	95,62	-	1,53	*0,07*	*0,12*	*0,92*	-	*0,36*	1,73	98,27	-	-
BR 099	?	0,13	1,34	97,25	-	1,28	*0,03*	*0,10*	*0,87*	-	*0,31*	1,37	98,63	-	-
BR 101	?	0,43	6,09	91,88	-	1,60	*0,05*	*0,21*	*0,84*	-	*0,34*	6,25	93,75	-	-
BS 040	?	0,61	23,95	74,01	-	1,43	*0,06*	*0,49*	*0,87*	-	*0,38*	24,55	75,45	-	-
BS 108	?	0,16	2,98	95,57	-	1,28	*0,03*	*0,15*	*0,86*	-	*0,31*	3,04	96,96	-	-
BS 234	?	0,22	90,01	7,22	0,64	1,91	*0,03*	*1,01*	*0,28*	*0,09*	*0,49*	92,04	7,31	0,64	-
BS 260	?	0,43	67,78	27,60	2,13	2,06	*0,05*	*0,87*	*0,56*	*0,17*	*0,49*	68,05	27,74	2,14	2,07
ED 002	?	0,13	3,29	95,22	-	1,36	*0,02*	*0,16*	*0,86*	-	*0,32*	3,36	96,64	-	-
ED 012	?	0,51	13,09	85,01	-	1,39	*0,05*	*0,31*	*0,79*	-	*0,32*	13,40	86,60	-	-
BO 044	II	1,11	0,75	96,82	-	1,32	*0,07*	*0,08*	*0,88*	-	*0,32*	0,77	99,23	-	-
BQ 041	II	0,67	10,39	87,19	-	1,75	*0,06*	*0,31*	*0,91*	-	*0,40*	10,70	89,30	-	-
BS 039	II	0,15	5,53	92,95	-	1,37	*0,02*	*0,19*	*0,86*	-	*0,35*	4,69	95,31	-	-
BS 068	II	0,12	1,76	96,86	-	1,27	*0,02*	*0,12*	*0,87*	-	*0,31*	1,79	98,21	-	-
BS 082	II	0,13	2,12	96,41	-	1,34	*0,03*	*0,13*	*0,88*	-	*0,32*	2,17	97,83	-	-
BS 107	II	0,16	1,84	96,81	-	1,20	*0,02*	*0,08*	*0,59*	-	*0,20*	1,87	98,13	-	-
N 036	II	0,37	1,01	96,94	-	1,69	*0,05*	*0,09*	*0,94*	-	*0,39*	1,04	98,96	-	-
AH 058	XIVc	0,20	2,43	95,83	-	1,55	*0,03*	*0,14*	*0,90*	-	*0,36*	2,45	97,55	-	-
AV 014	XIVc	0,20	3,15	95,16	-	1,50	*0,03*	*0,17*	*0,95*	-	*0,37*	3,22	96,78	-	-
BM 027	XIVc	0,16	5,24	93,47	-	1,13	*0,03*	*0,20*	*0,83*	-	*0,28*	5,34	94,66	-	-
BO 055	XIVc	0,15	2,51	95,95	-	1,39	*0,03*	*0,14*	*0,87*	-	*0,33*	2,57	97,43	-	-
BQ 157	XIVc	0,17	3,33	94,73	-	1,77	*0,03*	*0,17*	*0,90*	-	*0,38*	3,42	96,58	-	-
BS 027	XIVc	0,17	2,73	95,54	-	1,57	*0,03*	*0,15*	*0,92*	-	*0,37*	2,79	97,21	-	-
BS 098	XIVc	0,16	2,45	95,69	-	1,70	*0,03*	*0,15*	*0,93*	-	*0,39*	2,51	97,49	-	-
BS 157	XIVc	0,14	4,88	93,41	-	1,57	*0,03*	*0,19*	*0,84*	-	*0,34*	5,00	95,00	-	-
BS 160	XIVc	0,14	2,58	95,65	-	1,64	*0,03*	*0,15*	*0,91*	-	*0,37*	2,65	97,35	-	-
AV 161	XLI	8,71	26,24	63,54	-	1,51	*0,23*	*0,52*	*0,81*	-	*0,40*	28,78	71,22	-	-
BS 109	XLI	0,16	90,85	7,14	0,24	1,62	*0,03*	*0,96*	*0,27*	*0,05*	*0,42*	92,54	7,22	0,24	-
ED 010	XLI	0,97	70,19	27,43	-	1,41	*0,07*	*0,78*	*0,49*	-	*0,36*	71,40	27,86	-	-
BS 050	XLIV	0,12	6,86	91,69	-	1,32	*0,02*	*0,23*	*0,86*	-	*0,32*	7,00	93,00	-	-
BS 275	XLIV	0,46	13,21	84,16	-	2,17	*0,05*	*0,35*	*0,89*	-	*0,45*	13,26	84,56	-	2,18
BQ 152	XLVI	0,51	14,25	83,64	-	1,60	*0,05*	*0,37*	*0,91*	-	*0,39*	14,64	85,36	-	-
BS 102	XLVI	0,24	6,19	92,16	-	1,41	*0,04*	*0,22*	*0,87*	-	*0,34*	6,32	93,68	-	-
BS 114	XLVI	0,23	8,43	89,96	-	1,38	*0,04*	*0,27*	*0,90*	-	*0,35*	8,99	90,46	-	-
BS 158	XLVI	0,13	3,17	95,24	-	1,45	*0,03*	*0,15*	*0,84*	-	*0,32*	3,25	96,75	-	-
ED 001	XLVI	0,12	4,31	94,28	-	1,30	*0,02*	*0,19*	*0,89*	-	*0,33*	4,39	95,61	-	-
BS 259	XLVI?	0,18	4,93	93,55		1,35	*0,03*	*0,19*	*0,85*		*0,32*	5,03	94,97		-
BK 003	XLVII-1	0,25	41,60	56,72	-	1,42	*0,03*	*0,60*	*0,71*	-	*0,36*	42,45	57,55	-	-
BQ 145	XLVII-1	0,23	15,44	82,68	-	1,65	*0,03*	*0,35*	*0,83*	-	*0,37*	15,82	84,18	-	-
BQ 147	XLVII-1	0,29	11,52	86,55	-	1,64	*0,04*	*0,30*	*0,83*	-	*0,36*	11,82	88,18	-	-
BS 156	XLVII-1	0,12	5,17	93,48	-	1,23	*0,02*	*0,19*	*0,83*	-	*0,30*	5,27	94,73	-	-
BS 159	XLVII-1	2,75	16,34	79,06	-	1,85	*0,12*	*0,38*	*0,85*	-	*0,41*	17,18	82,82	-	-
BS 170	XLVII-1	0,15	13,12	85,47	-	1,26	*0,03*	*0,34*	*0,89*	-	*0,34*	13,37	86,63	-	-

Complete dataset												Restricted dataset			
Reg. nr.	**Class**	**Fe**	**Cu**	**Ag**	**Sn**	**Pb**	***2-sig Fe***	***2-sig Cu***	***2-sig Ag***	***2-sig Sn***	***2-sig Pb***	**Cu**	**Ag**	**Sn**	**Pb**
M 077	XLVII-1	0,95	29,98	67,34	-	1,74	*0,07*	*0,54*	*0,82*	-	*0,42*	30,92	69,08	-	-
BQ 138	XLVII-2	0,21	49,43	48,88	-	1,49	*0,03*	*0,65*	*0,65*	-	*0,36*	50,43	49,57	-	-
BQ 139	XLVII-2	0,58	56,51	38,31	2,83	1,77	*0,05*	*0,73*	*0,60*	*0,19*	*0,42*	58,02	39,09	2,89	-
BQ 148	XLVII-2	0,86	18,62	78,78	-	1,74	*0,07*	*0,43*	*0,90*	-	*0,42*	19,20	80,80	-	-
BQ 149	XLVII-2	1,50	22,79	73,99	-	1,72	*0,09*	*0,46*	*0,83*	-	*0,40*	23,62	76,38	-	-
BQ 150	XLVII-2	0,33	16,66	81,65	-	1,35	*0,04*	*0,38*	*0,86*	-	*0,35*	17,03	82,97	-	-
BS 262	XLVII-2	0,70	33,99	63,16	-	2,16	*0,07*	*0,64*	*0,88*	-	*0,51*	34,17	63,66	-	2,17
BS 279	XLVII-2	0,40	73,72	24,15	-	1,74	*0,04*	*0,86*	*0,50*	-	*0,43*	75,45	24,55	-	-
FO 001	XLVII-2	15,59	29,80	52,92	-	1,69	*0,30*	*0,54*	*0,71*	-	*0,41*	35,19	64,81	-	-
M 068	XLVII-2	0,77	53,62	37,40	6,15	2,07	*0,06*	*0,71*	*0,60*	*0,28*	*0,45*	53,97	37,74	6,22	2,08
M 074	XLVII-2	4,45	36,21	53,88	3,48	1,97	*0,15*	*0,57*	*0,70*	*0,21*	*0,42*	37,57	56,69	3,70	2,04

- **Drachms**

Complete dataset												Restricted dataset			
Reg. nr.	**Class**	**Fe**	**Cu**	**Ag**	**Sn**	**Pb**	***2-sig Fe***	***2-sig Cu***	***2-sig Ag***	***2-sig Sn***	***2-sig Pb***	**Cu**	**Ag**	**Sn**	**Pb**
BS 286	?	0,27	2,90	94,71	-	2,12	*0,04*	*0,16*	*0,92*	*0,43*	*0,04*	2,88	95,00	-	2,12
BS 069	S 5	0,12	4,60	93,93	-	1,35	*0,02*	*0,18*	*0,83*	*0,31*	*0,02*	4,70	95,30	-	-
BS 171	S 5	0,22	33,06	65,01	-	1,71	*0,03*	*0,54*	*0,77*	*0,39*	*0,03*	33,87	66,13	-	-
BQ 054	XLIV	3,74	14,15	80,72	-	1,39	*0,13*	*0,33*	*0,79*	*0,33*	*0,13*	14,87	85,13	-	-
BR 105	XLIV	0,24	66,57	31,56	-	1,64	*0,03*	*0,72*	*0,50*	*0,37*	*0,03*	67,99	32,01	-	-
BS 091	XLIV	0,17	58,01	40,42	-	1,40	*0,03*	*0,69*	*0,58*	*0,35*	*0,03*	59,08	40,92	-	-
BS 101	XLIV	0,29	70,37	28,01	-	1,32	*0,04*	*0,76*	*0,49*	*0,34*	*0,04*	71,63	28,37	-	-
M 073	XLIV	0,62	73,26	24,74	-	1,37	*0,05*	*0,76*	*0,45*	*0,34*	*0,05*	74,85	25,15	-	-
BO 040	XLVI	0,12	2,58	95,79	-	1,51	*0,03*	*0,16*	*0,95*	*0,37*	*0,03*	2,64	97,36	-	-
BQ 143	XLVI	0,12	8,05	90,23	-	1,61	*0,03*	*0,28*	*0,94*	*0,39*	*0,03*	8,24	91,76	-	-
BS 033	XLVI	0,19	6,16	92,04	-	1,60	*0,03*	*0,24*	*0,95*	*0,39*	*0,03*	6,32	93,68	-	-
BS 183	XLVI	0,18	3,49	94,86	-	1,46	*0,03*	*0,17*	*0,90*	*0,35*	*0,03*	3,55	95,99	-	-
M 078	XLVI	0,13	7,54	90,48	-	1,85	*0,03*	*0,26*	*0,91*	*0,40*	*0,03*	7,75	92,25	-	-
BO 062	XLVII	0,08	60,51	37,53	-	1,89	*0,02*	*0,73*	*0,58*	*0,42*	*0,02*	62,01	37,99	-	-
BQ 104	XLVII	0,17	47,80	50,36	-	1,68	*0,03*	*0,63*	*0,66*	*0,38*	*0,03*	48,87	51,13	-	-
BR 100	XLVII	0,19	41,54	56,37	-	1,90	*0,03*	*0,67*	*0,79*	*0,46*	*0,03*	42,62	57,38	-	-
BS 106	XLVII	0,22	19,01	79,13	-	1,65	*0,03*	*0,38*	*0,79*	*0,36*	*0,03*	19,48	80,52	-	-
BS 127	XLVII	0,17	2,86	95,42	-	1,56	*0,04*	*0,19*	*1,13*	*0,45*	*0,04*	2,93	97,07	-	-
M 079	XLVII	0,13	92,32	6,28	-	1,27	*0,02*	*0,86*	*0,22*	*0,33*	*0,02*	93,67	6,33	-	-

• Tetradrachms

		Complete dataset										Restricted dataset			
Reg. nr.	**Class**	**Fe**	**Cu**	**Ag**	**Sn**	**Pb**	***2-sig Fe***	***2-sig Cu***	***2-sig Ag***	***2-sig Sn***	***2-sig Pb***	**Cu**	**Ag**	**Sn**	**Pb**
BQ 136	?	0,22	2,06	96,00	-	1,72	*0,04*	*0,16*	*1,11*	-	*0,46*	2,93	97,07	-	-
AG 003	LI?	0,12	3,95	94,04	-	1,89	*0,03*	*0,24*	*1,17*	-	*0,51*	4,06	95,94	-	-
BS 080	XLIX	0,14	83,47	14,37	-	2,02	*0,04*	*1,32*	*0,55*	-	*0,68*	83,58	14,40	-	2,03
BS 097	XLIX	0,18	84,94	12,97	-	1,91	*0,04*	*1,31*	*0,51*	-	*0,65*	90,31	9,69	-	-
BM 026	XLV	1,19	85,87	5,40	4,89	2,65	*0,12*	*1,49*	*0,37*	*0,37*	*0,86*	86,90	5,47	4,95	2,68
BQ 137	XLV	0,22	87,90	5,14	5,15	1,59	*0,04*	*1,06*	*0,26*	*0,27*	*0,47*	89,61	5,21	5,18	-
BS 254	XLV	0,71	88,48	5,50	3,44	1,88	*0,06*	*1,06*	*0,26*	*0,22*	*0,51*	93,21	4,06	2,48	-
BS 284	XLV	0,19	90,44	4,26	3,30	1,82	*0,04*	*1,37*	*0,30*	*0,27*	*0,64*	94,52	3,12	2,36	-
BS 285	XLV	0,14	90,29	4,56	3,39	1,62	*0,03*	*1,10*	*0,25*	*0,22*	*0,49*	91,98	4,61	3,41	-
BS 096	XLVI	0,15	5,96	92,27	-	1,62	*0,04*	*0,28*	*1,12*	-	*0,46*	8,33	91,67	-	-
BJ 008	XLVII	0,23	56,67	41,68	-	1,43	*0,04*	*0,80*	*0,87*	-	*0,51*	57,74	42,26	-	-
BO 043	XLVII	0,48	76,65	21,19	-	1,68	*0,06*	*1,03*	*0,55*	-	*0,50*	78,45	21,55	-	-
BQ 005	XLVII	0,38	73,95	24,27	-	1,41	*0,03*	*0,95*	*0,55*	-	*0,43*	75,40	24,61	-	-
BQ 142	XLVII	0,10	5,09	93,13	-	1,68	*0,03*	*0,27*	*1,17*	-	*0,49*	7,14	92,86		-
BS 169	XLVII	0,16	97,39	0,80	0,26	1,39	*0,02*	*1,20*	*0,11*	*0,06*	*0,48*	98,94	0,81	0,26	-
N 310	XLVII	0,53	78,42	19,43	-	1,63	*0,06*	*1,04*	*0,52*	-	*0,49*	81,71	18,29	-	-
AD 025	XLVIII?	1,93	73,98	19,49	2,97	1,63	*0,09*	*0,81*	*0,42*	*0,18*	*0,39*	76,76	20,17	3,07	-
AC 012	XLVIIIa	0,23	66,00	32,01	-	1,77	*0,04*	*0,84*	*0,79*	-	*0,47*	67,20	32,81	-	-
AO 018	XLVIIIa	0,33	70,82	27,24	-	1,61	*0,04*	*0,77*	*0,48*	-	*0,38*	72,35	27,65	-	-
AV 023	XLVIIIa	0,17	78,92	18,21	0,49	2,22	*0,04*	*1,27*	*0,61*	*0,10*	*0,69*	79,05	18,24	0,49	2,22
BS 043	XLVIIIa	0,75	65,28	32,48	-	1,48	*0,07*	*0,95*	*0,67*	-	*0,46*	66,86	33,14	-	-
BS 071	XLVIIIa	1,60	54,38	41,95	-	2,07	*0,11*	*0,91*	*0,81*	-	*0,57*	55,13	42,77	-	2,10
BS 148	XLVIIIa	0,20	72,99	25,63	-	1,19	*0,04*	*1,02*	*0,59*	-	*0,34*	74,10	25,90	-	-
BS 172	XLVIIIa	0,21	71,98	25,59	-	2,22	*0,05*	*1,20*	*0,73*	-	*0,69*	72,12	25,65	-	2,23
BR 106	XLVIIIb	0,14	90,52	7,83	0,77	0,75	*0,02*	*0,86*	*0,30*	*0,10*	*0,36*	88,35	10,55	1,10	-
BS 235	XLVIIIb	0,18	79,19	15,08	3,90	1,65	*0,04*	*1,11*	*0,49*	*0,27*	*0,53*	80,78	15,28	3,94	-
BS 236	XLVIIIb	0,37	84,02	14,23	-	1,39	*0,06*	*1,43*	*0,59*	-	*0,60*	89,34	10,66	-	-
BS 237	XLVIIIb	0,45	89,66	5,24	2,51	2,14	*0,07*	*1,43*	*0,34*	*0,24*	*0,73*	90,07	5,26	2,52	2,15
ED 005	XLVIIIb	0,33	85,70	12,40	-	1,58	*0,06*	*1,49*	*0,57*	-	*0,66*	90,76	9,24	-	-
M 063	XLVIIIb	0,15	81,85	16,14	-	1,86	*0,04*	*1,33*	*0,59*	-	*0,66*	87,81	12,19	-	-
M 064	XLVIIIb	0,29	29,99	68,26	-	1,46	*0,05*	*0,63*	*0,96*	-	*0,44*	38,16	61,84	-	-

• Foreign coins

		Complete dataset										Restricted dataset			
Reg. nr.	**Class**	**Fe**	**Cu**	**Ag**	**Sn**	**Pb**	***2-sig Fe***	***2-sig Cu***	***2-sig Ag***	***2-sig Sn***	***2-sig Pb***	**Cu**	**Ag**	**Sn**	**Pb**
BO 056	Mediterranean	1,20	77,53	0,05	6,59	14,63	*0,08*	*0,93*	*0,02*	*0,29*	*1,32*	78,50	-	6,66	14,84
BS 070	Mediterranean	0,41	83,28	-	10,43	5,88	*0,04*	*0,85*	-	*0,31*	*0,74*	83,62	-	10,47	5,91
BS 072	Nabataean	0,56	94,41	-	2,43	2,61	*0,04*	*0,91*	-	*0,14*	*0,50*	94,96	-	2,42	2,62
BS 278	Persis	-	3,19	95,52	-	1,30	-	*0,15*	*0,83*	-	*0,30*	3,25	96,75	-	-
ED 011	Persis	0,25	2,30	96,07	-	1,37	*0,04*	*0,13*	*0,87*	-	*0,33*	2,35	97,65	-	-
M 080	Greek?	0,80	63,26	-	4,47	31,47	*0,06*	*0,79*	-	*0,23*	*1,80*	63,76	-	4,50	31,74
M 081	S-Arabian	0,64	72,08	0,22	6,94	20,12	*0,05*	*0,83*	*0,05*	*0,28*	*1,38*	72,69	-	7,01	20,30
M 082	Indian	1,00	96,85	0,26	0,27	1,63	*0,05*	*0,95*	*0,05*	*0,05*	*0,41*	100,0	-	-	-

Appendix 12: Glossary & other general information[1]

• Glossary

Alloy: the result of combining intentionally or inadvertently 2 or more metals in the presence of heat. Made through co-smelting various ores; combination of ores and metals; combining previously prepared metals. A substance having metallic properties and being composed of 2 or more chemical elements of which at least one is an elemental metal.

Annealing: the process of heating steel to red heat, holding it at this temperature for a time, followed by slow cooling. This remedies brittleness caused by hammering, rendering the steel in the softest possible condition and relieving stress.
Heating to and holding at a suitable temperature and then cooling at a suitable rate, for such purposes as reducing hardness, improving machinability, facilitating cold working, producing a desired microstructure, or obtaining desired mechanical, physical or other properties. When used to ferrous alloys, the term annealing implies full annealing (heating the steel to the proper temperature and then cooling slowly through the transformation range). When applied to non-ferrous alloys the term annealing implies a heat treatment designed to soften a cold worked structure by recrystallization or subsequent grain growth.
The process of softening a metal hardened by cold working (e.g. hammering). The lowest temperature at which a metal will soften varies with the degree of cold working, great amounts of working tending to reduce it.

Austinite: a solid solution of carbon (or other elements) in face centred γ-iron. In carbon steels, austenite appears only above the critical temperature (about 720°C in metal with about 0.02% C, 906°C in that with about 0.7% C, up to about 18% at temperatures above 1150°C). Cooling of austenitic structures leads to crystallization of ferrite and pearlite, while rapid cooling (quenching) develops martensite.

Billet: in recent archaeometallurgical literature a well-forged iron bloom, suitable for making a bar. Only a small part of the slag is still present.

Bloom: a mass of unrefined wrought iron with large quantities of entrapped slag and voids in the structure. The product of a bloomery process.

Bloomery iron: in antiquity the first product of smelting iron ore in charcoal. It is a relatively pure iron with small amounts of slag. Product of *direct iron smelting* process. Iron that is produced in a solid condition directly as a result of reduction of iron ore. The carbon content is variable, but usually low. High carbon bloomery irons have properties similar to modern carbon steel.

Brass: an alloy of copper and zinc.

[1] Tylecote, 1962: 312-316; Avrin, 1974: 667-688; Tylecote, 1976: 166-168; Knox, Maddin, Muhly & Stech, 1983: 98; Moorey, 1985: XXV-XXVI; Tylecote, 1989: XVIII-XXV; Pleiner, 2000: 287-292; Sim, 2002: 143-148; Tylecote, 1989: XVIII-XXV.

Brazing (Hard-Soldering) & Soft-Soldering: are joining techniques involving an agent (filler metal). Conventionally *soldering* refers to processes below 450°C; *brazing* to processes above 450°C.

Carbon in iron: as an alloying component, carbon increases its hardness (carbon steels) and simultaneously brittleness. Carbon is absorbed by iron in its austenitic state during the reduction of ore in the smelting furnace or during secondary carburisation.

Carburisation or Case hardening: introduction of carbon (process of diffusion) into iron in the austenitic state, mostly at temperatures above 780-906°C in contact with carbon-containing substances, essentially charcoal, by carbon monoxide or by carbon. The process takes place, under favourable conditions, in the furnace in the advanced phase of the smelting process, in spite of the fact, that in the bloomery at the tuyere mouth much of the metal would be secondarily oxidized (decarbonised). Prolonged heating (over several hours) of ready-made cutting edges of tools and weapons in contact with carbonaceous material was used to carbonise items of steel. Nitrogenous substances accelerate the carburisation; phosphorus blocks it to a certain extent.

- The process of heating bloomery iron in direct contact with charcoal. Carbon is absorbed into the iron converting the areas in which they combine into steel. So the surface of the metal is hardened while the interior is still soft.

Casting-on: a method employed not only in repairing broken or damaged objects, but also in manufacture, to form joins between elements in copper/copper alloys, or between elements in copper/copper alloys and iron.

Cast iron: (see also pig iron), the product of a *'Flossofen'* or blast furnace, an iron-carbon alloy of which the carbon content exceeds the solubility in austenite at the eutectic temperature (above 1148°C from 2% C onwards). This impure iron also contains other impurities besides carbon, and these are silicon and phosphorus. This renders the metal very brittle and not malleable either hot or cold. **Grey cast iron** contains pearlite, as well as segregated flakes of graphite, which cause a grey-coloured fracture. **Malleable** or **ductile cast iron** has the graphite granulated; in the **white cast iron** the excess carbon appears as cementite or ledeburite and is very hard and brittle and makes the metal unmalleable. In modern metallurgy, cast iron is the product of special cupola furnaces and is destined for foundry purposes.

Cementation (brass production): involves heating finely-divided copper metal together with zinc oxide or carbonate (calamine) and charcoal in closed crucibles. The zinc ore is reduced to metallic zinc vapour, which defuses in the copper, forming brass. At the end the crucible content is melted to homogenise the alloy. This process was developed in Asia Minor in the 1st century BC[2].

Cementation (steeling): a process used for carburising soft iron bars to make steel. The bars are heated for several days in contact with carbon.

[2] Bayley, 1988: 196.

Cementite: Fe_3C, hard and brittle iron carbide which occurs in steel and cast iron either as an intercrystalline phase in very low carbon steel ('tertiary cementite') or as a component of pearlite or as white cells (appearing as a network in section) surrounding the pearlite grains in hypereutectic steels. It also appears in the grain boundaries of wrought iron containing about 0,02% carbon, and in irons containing more than 0,89% carbon. In the latter case it may produce a Widmanstätten structure. It appears in white cast iron in the pearlite and as a separate constituent. The carbon in cementite is normally referred to as 'combined carbon' to distinguish it from the form of carbon known as graphite.

Cinder: are partially smelted ores, which have been in such a position in the furnace that reduction has been unable to proceed to completion. Cinders are very porous and the shape of individual ore lumps can still be distinguished. Although some low-melting point constituent has bonded the whole together, the remains of pieces of charcoal can be seen.

Cold working: metals, when hammered at low temperatures, become hardened and stronger. If the temperature of working is increased, a point is reached at which hardening no longer occurs, i.e. the hot working temperature is reached.

Cupellation: process that is used separate silver from argentiferous lead by melting the unrefined metal in an open hearth. This oxidises the lead to litharge that can be skimmed off, eventually leaving behind a small button of pure silver. The same can be done in a shallow open crucible, the cupel[3].

Decarburisation: the process for depleting surface layers of carbon. Carbonised iron is heated in an oxidizing atmosphere so that the carbon in the surface layers combines with oxygen and is given of as gas.

Dendrite: a crystal that has a tree-like branching pattern, being most evident in cast metals slowly cooled through the solidification range. This structure formed by a solid metal or constituent growing from the liquid. Many pure metals and alloys solidify in this way, as do some constituents of slags, such as wüstite and magnetite in fayalite.

Equiaxial: Term applied to crystals which are roughly as broad as they are long.

Eutectic: 1. an isothermal reversible reaction in which a liquid solution is converted into two or more intimately mixed solids on cooling, the number of solids formed being the same as the number of components in the system.
2. an alloy having the composition indicated by the eutectic point on an equilibrium diagram.
3. an alloy structure of intermixed solid constituents formed by a eutectic reaction.

[3] Bayley, 1988: 196.

Eutectoid: 1. an isothermal reversible reaction in which a solid phase (usually a solid solution) is converted into two or more intimately mixed solids on cooling, the number of solids formed being the same as the number of components in the system.
2. an alloy having the composition indicated by the eutectoid point on an equilibrium.
3. an alloy structure of intermixed solid constituents formed by a eutectoid reaction.

Fayalite: iron orthosilicate Fe_2SiO_4 from the family of olivines, melting at 1175-7780℃, is a constituent of iron and most of copper slags or occurring in volcanic rocks. It appears as dark grey laths or fields on polished sample blocks, studied by microscope. Combined with other elements (Ca, Mg, Mn) it forms different mineralogical phases.

Ferrite: ductile crystals of almost pure body-cantered α-iron, containing no carbon or a minimal amount (0.02-0.03%) which does not appear as pearlite. This is unsteeled iron. During the very first phase of reduction ferrite creates a thin skin around the ore fractions or charcoal particles. Ferrite iron is easily malleable and relatively soft. In the optical field of the microscope, ferrite appears as light structures with a network of grain boundaries. The crystalline form of iron that is stable below 910℃. This form of iron is magnetic.

Flux: a substance lowering the melting point of metals and minerals, promoting the liquefaction of slag. In metallurgy, siliceous or calciferous components; according to the ore composition, may be added to the charge. Lime fluxes were exceptional in bloomery technology; silica could be added in case of high-grade iron oxides. Old bloomery slag with its high FeO content was used as flux as well. Sand flux (fine quartz) was used to remove hammer-scale during forging by melting to fayalitic slag.

Fire welding (forge welding): the process in which 2 pieces of iron are heated to white heat and hammered together, thus causing them to fuse.

Forging: the process by witch a piece of metal is shaped through hammering either at its normal temperature (cold working) or when heated.

Gangue: non-metallic constituents of any ore. Unwanted mineral.

Gossan: The part of a metalliferous deposit from which the wanted metal has been leached and which is rich in iron.

Hypereutectoid: steels containing more than 0,8% carbon. In these steels, austenite also transforms in a range of temperatures during slow cooling, producing increasing amounts of 'proeutectoid cementite' as it cools. The cementite precipitates in the austenite grain boundaries and some extent along certain crystallographic planes of the austenite. When the carbon content of the austenite has decreased to the eutectoid amount (0,8%) at 727℃, pearlite forms. The microstructure of such a steel consists of a carbide network in a pearlite network with occasional needles of cementite in the pearlite.

Hypoeutectoid: steels containing less than 0,8% carbon. In these steels, austenite transforms in a range of temperatures during slow cooling, forming increasing amounts of 'proeutectoid ferrite' as it cools. Carbon increases in the remaining austenite as its quantity diminishes in the cementite component of the cooled steel. When the eutectoid composition and temperature are reached, the remaining austenite transforms to pearlite. The microstructure of such a steel consists of a mixture of ferrite and pearlite.

Liquation: the separation of metals by graded fusion of the metals themselves or their eutectic mixtures.

Litharge: the lead oxide α-PbO, formed as a result of cupellation.

Melting/smelting: *melting* is changing a metal from the solid to the liquid state, whereas *smelting* is the process by which a metallic ore is converted to metal through the agency of heat and chemical energy.

Matte: a liquid or solid mixture of sulphides, usually FeS and Cu_2S, but other sulphides may dissolve in the mixture. Usually present at some stage in the production of copper from sulphide minerals.
A compound of metals and sulphur, often produced in the first state of smelting copper in which case it is a mixture of iron and copper sulphides.

Pearlite: a lamellar conglomerate of ferrite and cementite plates found in carbon steel. Its pearly lustre is due to the fine and regular alternation of the two constituents. Cementite being more resistant than ferrite throws, after etching of polished samples with acids, minute shadows. Thus pearlite appears darker, when observed under the microscope. When annealed, the lamellae of cementite coagulate into globular (globular cementite). Pearlite is the basic crystalline microstructure of carbon steel.
- the product arises when austenite is cooled more slowly. The name derives from the decomposed appearance of the austenite: alternate lamellae of **ferrite** (pure iron) and **cementite** (a compound of iron and carbon) with a "pearly" appearance under the optical microscope.

Pig iron: in modern metallurgy, crude impure iron produced in blast furnaces, containing 2 – 4.4% C, and destined for foundries. Melts below 1200°C. According to the ore used or smelting practices applied, pig iron contains Mn, which facilitates the absorption of carbon; Si hinders it causing the formation of graphite flakes. P penetrates into the pig iron completely. S represents an unwanted impurity. It is difficult to distinguish between pig- and cast irons produced in the early phases of the indirect process.

Phase: anything which is homogeneous and physically distinct, this is a visual distinction under the microscope (so not on the atomic level).

Quenching: a kind of heat treatment applied to steel, comprising a rapid cooling usually from temperatures above 900°C in water or m ore drastic acidic liquids etc. The result is the coarse or fine acicular martensite structure. Quench hardened steels are stronger but also more brittle, the brittleness increasing with higher carbon content and speed of cooling.
- plunging a hot metal into water or some other cooling medium; most commonly used for hot steel (austenite).

Roasting: the ore is dried to prepare it for smelting; in the case of iron carbonate roasting reduces it to iron oxide.

Sintering: in modern metallurgy the fritting together of powder particles of metal with different melting points. In the bloomery process the austenitic iron particles coagulate in the liquid or viscous slag to larger complexes (spongy iron), without being artificially welded.

Solid solution: a single solid homogeneous crystalline phase containing two or more chemical specimens.

Spheroidite: when martensite is reheated to a temperature in the vicinity of 500-600°C, it decomposes, precipitating iron carbide with agglomerates in the form of spheroids rendering a less hard but tougher steel.

Steel:
- an alloy of pure iron and carbon, often forming a solid solution. Steel containing more than 0,3% carbon can be flame hardened. The carbon content does not exceed 1,8%. Steel is extremely hard, tough and strong; it is fairly subject to rusting (oxidation). *Carbon steel* is the earliest and most common type.
- some modern steels do not contain carbon in more then trace amounts, but are alloys of iron and some other element(s); other contain iron, carbon and other elements.

Tempering: the reheating of quench hardened steel below the critical range (usually up to 500-600°C, according to purposes required), in order to decrease the brittleness and, to certain extent, hardness. Martensite structures decay to pearlite-based fine structures (formerly known as **sorbite** and **troostite**).
- the process for relieving the brittleness of martensite, accomplished by heating to a temperature in excess of 150°C and than cooling at the desired rate..

Twins: faults in crystals which show that the structure has at one time been strained mostly by hammering or bending.

Welding: heating 2 pieces of metal to be joined, almost to melting-point, and then hammering them together. In antiquity it could not have been used to join copper or copper alloys, only iron.

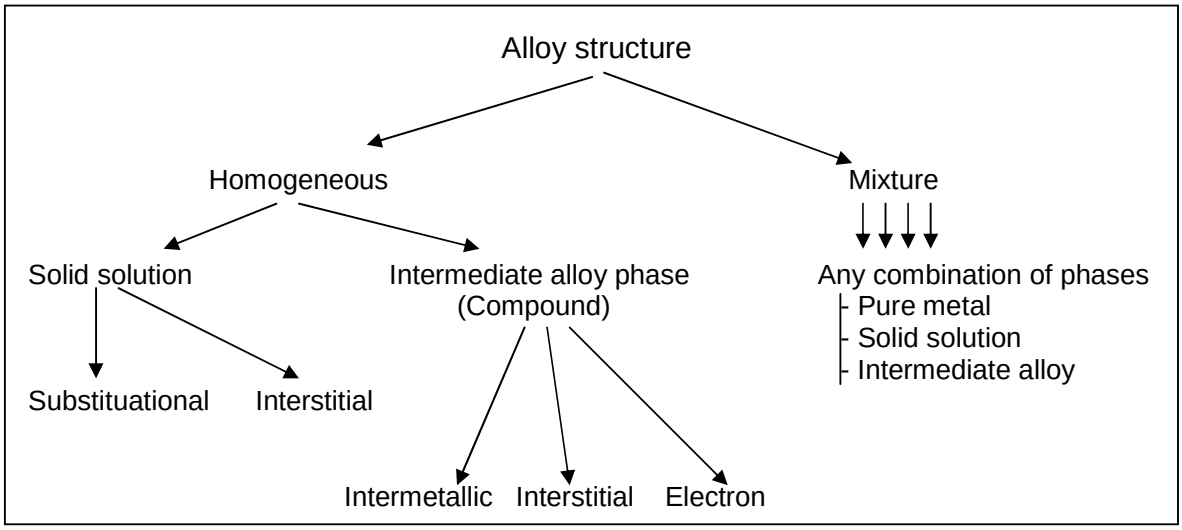
Widmanstätten structure: a microstructure characterized by a tendency of ferrite to form plates, during fairly rapidly cooling from above 710°C. Ejection of ferrite or cementite takes place along certain crystallographic planes of the parent austenite. A structure characterized by a geometrical pattern resulting from the formation of a new phase along certain crystallographic planes of the parent solid solution. The orientation of the lattice in the new phase is related crystallographically to the orientation of the parent phase.
- The Widmanstätten structure results from the precipitation of a new solid phase within an existing solid phase. It is the result of one solid phase at a high temperature decomposing into two solid phases at a lower temperature. This precipitation usually occurs at the grain boundaries of the initial crystals and as plates or needles within the grains themselves.

Wrought iron: a malleable, tough and relatively soft iron, low in carbon (at most a few 10^{th} percent), containing much entrapped slag. Bloomery iron belongs to this category. Melting point ca. 1540°C. Iron made either by the direct process or resulting from a convertion process such as puddling or fining. Is normally low in Mn, Cu, Ni but may contain appreciable amounts of P.

Wüstite: iron monoxide FeO can contain some MnO or NiO, a phase in the reduction of iron ore, forming subsequently, combined with SiO_2, the fayalite. Excess wüstite appears in bloomery slags and slag inclusions, in carbon-poor parts of iron/steel as light crystals (also arranged to dendrites).

• General information

Alloy structure[4]:



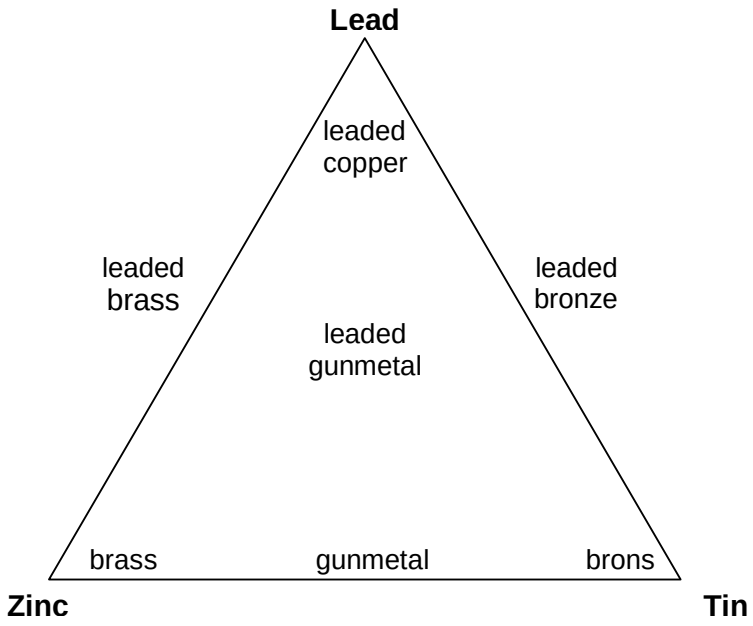
An alloy may be homogeneous (uniform, single phase) or mixed (several phases). A phase is anything that is homogeneous and physically distinct, this is a visual distinction under the microscope (so not on the atomic level). When a metal undergoes a change in the crystal structure, it undergoes a phase change, since each type of crystal structure is physically distinct. In the solid state there are 3 possible phases:

- ***Pure metal***
- ***Intermediate alloy phase or compound***: each element in the compound has its own chemical properties, when they are combined they get new ones (ex. NaCl: Cl is toxic and Na has to be kept in kerosene, but when combined you can eat it and expose it to the air; H_2O: H and O are both gases at room temperature, when combined they are liquid).
- ***Solid solution***: is composed of 2 parts: a *solute* (minor part, the material that is dissolved) and a *solvent* (major part of the solution), ex. sugar + water. Is simply a solution in the solid state and consists of 2 kinds of atoms combined in one type of space lattice.

Fe and C are in ***interstitial solid solution***. The atoms of C are small enough to fit in between the Fe-lattice. This increases the strength because they interfere with the movement of dislocations on slip planes and will therefore strengthen the alloy.

[4] Avner, 1974: 154.

***Alloys terminology of copper with lead, tin and zinc*[5]:**



Data on metallic elements discussed:

Metal	Symbol	Melting point	Specific gravity g/cm³
Tin	Sn	232℃	7,29
Lead	Pb	327℃	11,34
Zinc	Zn	419℃	7,16
Silver	Ag	960℃	10,53
Gold	Au	1063℃	19,30
Copper	Cu	1083℃	8,95
Iron	Fe	1535℃	7,88

[5] After Bayley, 1988: 204; Bayley, 1998: 8.

"*You admire a girl's curves on the first introduction, but the second meeting shows up new angles*."

Mae West

UNIVERSITEIT GENT