

## Appendix A: extended methods and procedures

### A.1 Apatite fission track (AFT) method and procedure

Apatite grains were handpicked using a Leica MZ 16 FA stereo zoom microscope with both dia- and episcopic illumination, with preference for large, transparent, inclusion-free and complete grains. The grains were embedded in transparent epoxy (Struers CaldoFix-2) and cured for 12h at 40°C. After grinding and polishing to reveal internal grain surfaces, the embedded apatite was etched for 20s in a 5.5 mol/l nitric acid solution, at 21°C (Donelick, 1993). The mounts were then covered with a 0.025 mm thick mica sheet (Goodfellow Clear Ruby muscovite), which is kept in place by Scotch tape, and stacked into a polyethylene container for irradiation. The samples were stacked together with evenly spaced U-doped glass shards (IRMM-540; De Corte et al., 1998) and Durango (DUR) and Fish Canyon Tuff (FCT) age standards. Thermal neutron irradiation of the package took place in the well-thermalized channel X26 of the BR1 nuclear reactor in Mol (Belgium, SCK-CEN; De Grave et al., 2010). We estimate neutron flux gradients by linear interpolation over the induced track density of the U-doped glasses. After radioactive cool-down, the external detectors were punctured at three positions to create mount-mica reference points, after which the micas were removed from the mount. The micas were then etched for 40 minutes in a 40%<sub>w</sub> hydrofluoric acid solution at ~21°C. The mounts and mica external detectors were then fixed on standard microscope object glasses, using transparent nail polish.

We measure track densities on a fully motorized Nikon Eclipse Ni-E microscope, equipped with a Nikon DS-Ri2 colour camera. The microscope and camera are linked to a computer with Nikon NIS Elements Advanced Research software, complemented with an in-house macro-enabled Microsoft Excel sheet (TRACKflow β). Surface track densities were measured on-screen, on images captured using a 100x plan apochromat class objective. The spontaneous track area in the apatite grain and the induced track area in the mica external detector are digitally matched using micro-georeferencing in TRACKflow (Helmert transformation and Z-surface interpolation) on a minimum of three coarse and five fine reference points. The digital images are further matched using an image flip and rotation. Where possible, a minimum of 1000 spontaneous tracks (s.e. ≈ 3%) were counted. All analyses were performed by one observer, and ages have been calculated based on one overall mean weighted zeta (OMWZ). The OMWZ is based on both DUR and FCT age standards, from different irradiations, following the recommendations of Hurford (1990). This OMWZ was verified using the Z-approach (Enkelmann et al., 2005) with standards that were not used to calculate the OMWZ. Data processing was performed in the TRACKflow Excel sheet, which is based on definitions, formulae and algorithms from Galbraith (2005). For data visualisation, radial plots were generated using IsoplotR (Vermeesch, 2018).

In contrast to surface tracks, confined tracks are not cut by the surface and retain their full etched length. They are thus etched via surface tracks (TINT; Bhandari et al., 1971) or via cracks or cleavages (TINCLE; McDougall et al., 1971). If cracks/cleavages were pre-existing (not due to mechanical sample crushing), TINCLEs might be naturally etched by circulating fluids, and thus rendered insensitive for later temperature history recording (Jonckheere and Wagner, 2000). TINCLE measurements are thus unreliable and were avoided for this study. This makes it, especially when disregarding TINCLEs, tedious and sometimes impossible to find a statistically adequate number of confined tracks in samples with a low surface track density. A way to overcome this problem is found in creating artificial etchant conduits with either a <sup>252</sup>Cf fission fragment source (Donelick and Miller, 1991) or accelerator-ion

irradiation (Jonckheere et al., 2007). The ion accelerator gives the advantages that the created sub-parallel tracks can easily be distinguished from natural tracks and that beam parameters (energy, direction and fluence) can be set. The ion used for bombardment is not at choice of the fission track user, however different ions (Fe, Ni, Cu, Kr, Ag, Xe, Au, Pb, Bi and U) yield similar results concerning the number of obtained tracks (Jonckheere et al., 2007). Considering these advantages, we applied heavy ion bombardment at GSI Helmholtz on a subset of samples. A second set could not be treated in this facility, due to maintenance works at that time.

Confined track lengths and angles to the crystallographic c-axis were measured using a 100x plan apochromat class objective and a 2x secondary optical magnification (Nikon DSC zooming port). Additional digital magnification leads to a total effective magnification of ~8800. Projected tracks were corrected to 3D lengths in TRACKflow using the precise z-readout of the microscope (0.025  $\mu\text{m}$  linear encoder) of the track's end-points and a correction for the optical media transitions ( $n_{\text{air}} = 1.0028$ ;  $n_{\text{Ap}} = 1.6455$ ). We aimed at a minimum of 100 confined tracks for each sample. For each sample, anisotropy was estimated by calculating the  $l_a$  and  $l_c$  intercepts and plotting the track length versus the angle.

The etch pit diameter parallel to the crystallographic c-axis ( $D_{\text{par}}$ ; Donelick, 1993) was measured using automated and manual measurement. We used intensity-based image separation in Nikon NIS Elements, with a filter based on area, elongation, roughness and orientation, to automatically measure  $D_{\text{par}}$  (as the mean maximum Feret distance). This approach was verified by manual length measurements and each analysed grain image was checked for false etch pit identification. A consequence of this approach is that the number of  $D_{\text{par}}$ s measured will be higher for samples with a high track density. Samples that were unsuitable for automatic  $D_{\text{par}}$  measurements, due to impurities or polishing defects, were measured manually, with five  $D_{\text{par}}$  measurements per grain.

Within apatite a certain degree of anisotropy might occur concerning the track length (Donelick, 1991, 1999; Ketcham et al., 2007a). Confined tracks tend to be longer in directions parallel to the crystallographic c-axis and shorter perpendicular to the c-axis. This anisotropy is often corrected for by using a c-axis projection (Ketcham et al., 2007a). A plot of  $l_a$  vs.  $l_c$  however shows that the anisotropy of our samples deviates from the line used for c-axis projection (Donelick, 1991) (Appendix B, Fig. 2). Indeed, when plotting angle to c-axis vs. track length for all tracks of all samples, for both uncorrected and c-axis projected samples, it becomes clear that often the c-axis projection overcorrects. The same can be observed on the  $l_a$ - $l_c$  plot. For our samples, a minority is indeed corrected, while the majority is converted to inverse anisotropy, with tracks perpendicular to the c-axis becoming longer than tracks parallel to the c-axis. As the result of c-axis projection is based on anisotropies different from our samples, and the effect is different for each of our samples, we decided not to apply c-axis projection. Interesting to note is the weak correlation ( $R^2 = 0.22$ ) between AFT central age and the degree of anisotropy, samples become more isotropic with increasing age (Appendix B, Fig. 2).

## A.2 Apatite (U-Th)/He method and procedure

Suitable grains for AHe analysis were picked under a Leica MZ 16 FA with diascopic, polarized light. Samples were withheld for AHe analysis based on the quality of the separation. Primary grain selection was based on clarity, idiomorphy and the absence of visible inclusions, following recommendations of Flowers and Kelley (2011). A secondary selection was performed under a Nikon Eclipse Ni-E microscope with epi- and diascopic light, at 200x magnification. Two horizontal axes (length, width)

were measured on the live image produced by the mounted Nikon DS-Ri2 high-resolution camera. The third, vertical axis (thickness), was measured using the microscope z-drive registration (0.025  $\mu\text{m}$ ). We made multiple (10–15) single-grain aliquots, with each grain packed into a 99.99% pure platinum crucible (Refining Systems Inc.). The platinum tubes used for packing the apatite were first cleaned using 65%<sub>w</sub> nitric acid and then rinsed until neutral using ultrapure (Merck Millipore) water. We conducted the AHe analysis at the Geochronology Centre of University College London (United Kingdom).

Samples were outgassed under ultra-high vacuum at  $\sim 900^\circ\text{C}$  for 5 min using a fibre-optically coupled diode laser with a 808 nm wavelength, then spiked with  $^3\text{He}$  and gas volumes determined using a Pfeiffer plasma quadrupole mass analyser. Molar abundances of U and Th were determined by isotope dilution using a mixed  $^{235}\text{U}$ - $^{230}\text{Th}$  spike. The Sm abundance was determined by comparison with a standard solution of known U/Sm ratio. U-Th-Sm analyses were carried out by ICP-MS, using an Agilent 7700x quadrupole mass spectrometer. Apparent AHe ages were calculated and corrected for  $\alpha$ -emission following the approach of Ketcham et al. (2011). Durango apatite was run as an external standard with each batch of samples as an additional check of the analytical accuracy.

Data reduction and error propagation were performed in HelioCalc (<http://www.ucl.ac.uk/~ucfbpve/heliocalc/>). Outliers were detected following the modified Chauvenet criterion embedded in IsoplotR (Vermeesch, 2018), which was also used to calculate the central ages (Vermeesch, 2008).

### A.3 Inverse t-T modelling

Samples that yielded sufficient confined track length measurements were withheld for quantitative thermal history modelling. The T-t proposed models were generated using the QTQt software package (version 5.6.3) of which the working is explained in detail by Gallagher (2012). This application performs inverse thermal modelling based on the Bayesian transdimensional Markov chain Monte Carlo (MCMC) approach (Gallagher et al., 2009; Gallagher, 2012). The proposed models result from multiple random sampling (Monte Carlo) through the model space, proposing a new model in each step, conditional on the previous model (Markov chain). This generates a number of discrete time-temperature points, between which linear interpolation is performed. Whether a new T-t point is accepted or not, is dependent on the likelihood (fit to the data). The Bayesian approach furthermore favours simpler models, i.e. models with fewer points that fit the data adequately. In this way it is thus mostly the data deciding the outcome. The allowed step size of each perturbation on a previous model towards a new model is determined by the search parameters, as set by the user. In this process, QTQt generates an output with a range of withheld accepted models. For this study we will report the ‘expected model’, which is a 1 Ma interval weighted mean of the temperature probability distribution. The weighing is done based on the posterior probability for each individual model. This way common features of all accepted models will be retained, while features occurring in only a small number of models are dampened through averaging. This further allows to generate 95% credible intervals, to provide an uncertainty estimation on the expected model (Gallagher, 2012; KG *pers. comm.*).

In order for QTQt to calculate proposed time-temperature paths, we provide the program with spontaneous and induced track density data, length-frequency distributions and  $D_{\text{par}}$ . Where adequate AHe data were available, a second T-t path was calculated including these AHe results, corrected using

the RDAAM model (Flowers et al., 2009). We use the multikinetic fission track annealing model of Ketcham et al. (2007), with  $D_{\text{par}}$  serving as kinetic parameter (Ketcham et al., 1999).

Since all samples are from outcrops, we constrain the present-day temperature to  $20 \pm 20^\circ\text{C}$ , which seems realistic for recent climate conditions in eastern Brazil. The prior for temperature was set at  $70 \pm 70^\circ\text{C}$ , which envelopes the maximum temperature interval of which the data contain information. The prior for time is set at  $t_0 \pm t_0$ , for which  $t_0$  is the oldest AFT age, which also theoretically is the oldest age of each sample. We first ran the algorithm for 10 000 burn-in and 50 000 post-burn iterations in order to find the appropriate search parameters. Acceptance rates should be between 0.2 and 0.6 (QTQt documentation). When the appropriate parameters were found, we ran 200 000 iterations, with a burn-in of 50 000 for independent sample inversion. Adjacent samples were considered for joint inversion if (i) we don't have evidence of major structural separation, and (ii) they show a normal age-elevation relationship, i.e. age becoming older with elevation. After a first run of 10 000 burn-in and 50 000 post-burn iterations to find adequate search parameters, the model was run again for  $1\text{E}^6$  burn-in and  $2\text{E}^5$  post-burn iterations.

#### A.4 Sources of uncertainty and 'detection limit'

Although it is beyond the scope of this writing to provide a summary of statistics, in this section we briefly discuss sources of uncertainty as they are important for the interpretation and sometimes neglected in literature.

Same as for all sorts of analysis, AFT dating has a standard error, which is in essence a result of the number of tracks, and thus of the combination of U concentration and age (Wagner and Van den haute, 1992; Galbraith, 2005). The uncertainty produced by natural variance is well treated by the use of the central age (Galbraith and Laslett, 1993; Galbraith, 2005). This produces the mainly published quantified uncertainty that is displayed by the error bar, e.g. on an age-elevation plot. An unquantified error that adds to this statistical error is the observer error. Part of this semi-systematic human error (who accepts what as a track) is addressed within the multi-parameter  $\zeta$  approach (Hurford and Green, 1983; Hurford, 1990). Nonetheless, a sample-specific human error still exists due to differences in defects and uranium content; i.e. tracks in samples with very high track density are more difficult to distinguish, resulting in a larger human error. Thus, with increasing track density, the statistical error becomes smaller, as, starting from a certain threshold, the total error will start to rise until the point where a certain user decides that a track density is too high to count. Naturally, the human error in samples containing a high number of defects will be higher than in clean samples.

For length measurements, the main source of error is the systematic error inherent to the system and applied calibrations and corrections, which is more than often ignored. A second source of error is also introduced through the observer. The precision of an observer can however be verified by measuring standard packages. As is already addressed by Sobel and Seward (2010), deviation of the etching conditions can also induce error in length measurements. This error is also not quantified.

This summary on errors aims to indicate that by the time data from a sample is used for inversion, there is already a degree of error present within both the statistical and the unquantified error on both the density and length distribution. The expected T-t path predicted by the models depends on the software used (Vermeesch and Tian, 2014) and also contains a credibility envelope. However, to the best of our knowledge, errors on the annealing models themselves are not included into this envelope. We

furthermore draw attention to the ‘prior-effect’, induced by setting the modelling space, with T-t paths becoming increasingly simple to the degree of constant cooling, with a larger modelling space.

To conclude, we emphasize that expected T-t models contain a large degree of accumulated uncertainty which is often ignored. These predictions should thus be treated with care and always be verified with the data. This brings us to the role of the missing section. Since apatite fission tracks accumulate at full length above the partial annealing zone, further cooling or heating to less than 60°C cannot be registered. In the time interval between when a model predicts that a sample cooled to below 60°C and today, it is uncertain when the last section was removed.

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