

Influence of Li in deformed Al-Li alloys

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Positron-annihilation Doppler-broadening and lifetime measurements are used to study the influence of Li on the defect behavior in deformed Al-0.2 at. % Li and Al-9.9 at. % Li samples. Deformations were performed at low temperature (77 K) and at room temperature. For the low-temperature deformed sample the migration and subsequent clustering of vacancies is seen in a stage between 200 and 350 K. The temperature dependence of the positron trapping rate at voids is seen. Above this annealing stage the only remaining defects are dislocations that anneal out above room temperature. In the dilute alloys it is seen that these dislocations are pinned by the Li atoms. In the concentrated alloy trapping of positrons at the δ' particles is observed. In the room-temperature deformed dilute alloys vacancy-Li complexes and dislocations are formed. These defects anneal in two distinct stages.

I. INTRODUCTION

Adding impurities to pure metals can drastically alter the behavior of the material. For example, it is known that adding lithium to aluminum results in an alloy with an increased strength. Such alloys have many applications, especially in the aerospace industry.

The positron-annihilation technique (PAT) has proved to be a very sensitive measuring technique in relation to open space defects. When studying dilute alloys with the PAT technique, several supplementary effects influencing the defect behavior can occur. It is known that the addition of alloying elements can strongly affect the point defect behavior in the material.¹

In a recent work² we studied, using the positron-annihilation Doppler-broadening technique, the annealing behavior of low-temperature ($T=77$ K) deformed Al-Li alloys. From these measurements it was concluded that the presence of Li in the matrix had a strong effect on the monovacancy behavior leading to an enhanced clustering of the vacancies in the Al-Li alloy in comparison with pure aluminum. It was also seen that positrons were very sensitively detecting the growth of the δ' phase and the dissolution of this phase.

In this work we will describe some positron-annihilation lifetime studies on two types of Al-Li alloys (dilute and concentrated). For the alloys studied, the principal phases which occur in the phase diagram are α (Al-Li in solid solution), δ' (an Al_3Li coherent ordered phase), and δ (Al-Li).

Doppler-broadening and lifetime measurements have been performed to study the defect kinetics in the deformed alloys and two kinds of annealing experiments have been carried out to go deeper into the positron defect interaction: isochronal annealing and annealing measurements at the treatment temperature itself.

II. EXPERIMENT

The samples used in this work were supplied by Alcan Ltd. (U.K.). The concentrated alloy was an Al-9.9 at. % Li aged at 510 K for 4 h and finally quenched to room temperature; this thermal treatment leads to the formation of δ' particles.³ The dilute alloy was obtained by annealing for 30 h at 873 K an alloy originally containing 3.7 at. % Li; it has been found that this thermal treatment reduces the Li content in these samples to 0.2 at. %.⁴

The samples underwent several deformation processes that are summarized in Table I. The deformation degree is indicated as $\Delta D/D_0$, where D_0 is the initial sample thickness and $\Delta D = D_0 - D$ is the absolute thickness reduction. For the samples deformed at 77 K the source mounting and all further manipulations were performed under liquid nitrogen in order to prevent them from warming up. After deformation the samples were annealed from the deformation temperature up to about 600 K; the annealing behavior was followed by measuring the

TABLE I. Experimental details for the concentrated and dilute alloys studied in this work. D and L indicate, respectively, Doppler-broadening and lifetime measurements. Letters A and I refer to the annealing experiment and indicate, respectively, measuring at the annealing temperature itself and measuring at a fixed reference temperature.

Li content (at. %)	Deformation temperature	Measurement technique and deformation degree (%)	
9.9	77 K	D, A 23	$L, A/I$ 22
9.9	RT	...	L, I 20
0.2	77 K	D, A 23	...
0.2	RT	D, A 22	L, I 12

Doppler-broadening S parameter and/or the lifetime. Details about the measuring technique performed for each sample are given in Table I; further details on the measurements can be found in Sec. III. The wing Doppler parameter W has been also deduced from the Doppler data but its behavior turned out to be complementary to the S parameter data, thus giving essentially the same information. For this reason, no further mention will be made.

As a positron emitter, a conventional $^{22}\text{NaCl}$ source was used. The Doppler broadening of the annihilation line was measured with an intrinsic Ge detector having a resolution of about 1.2 keV at an energy of 511 keV, that was characterized by the conventional S parameter. The lifetime measurements were carried out with a fast-fast system having a resolution of 250 ps (full width at half maximum) (FWHM). The lifetime spectra analysis was performed by using one or two components after subtracting the source contribution.

III. RESULTS

A. Low-temperature deformed samples

Several runs have been performed in the S -parameter measurements in order to check the reversibility of the curves measured at the annealing temperature itself in different temperature ranges. With this purpose, the annealing behavior has been followed by increasing the temperature in steps of 10 K from 100 K to a certain temperature T_{r1} and then back to 100 K; after the cooling down, the temperature was again raised up to a new value T_{r2} , higher than the previous T_{r1} , and again cooled down to 100 K.

Four temperatures T_{ri} were chosen to study the Al-0.2 at. % Li alloy 23% deformed: 200, 250, 340, and 440 K. From 440 K on, the temperature was increased in steps of 20 K up to 680 K and then decreased to 100 K. The annealing up to 680 K ensures the complete recovery of the sample. The first run was made between 100 and 200 K and it has been found that the curve is reversible in this interval. These points have been omitted in the figure for the sake of clarity. Warming up the samples above 200 K changes the slope of the annealing curve but the cooling down run, after annealing at 250 K, does not coincide with the annealing curve. For each T_{ri} the cooling down path coincides with the one warming up again. It is to be noted that the qualitative features of the whole annealing curve were reproduced by a second sample deformed to 21%, but we have omitted the figure in the interests of space. The linear behavior observed between 100 and 550 K after annealing at 680 K can be attributed to thermal-expansion effects, since the slope is very close to the one previously measured at thermal equilibrium in a well-annealed dilute Al-Li alloy (1.7 at. % Li).⁵ Above 550 K the positron signal contains the contribution of thermally induced vacancies [see Fig. 1(a), cooling down run after annealing at 680 K].

For the alloy Al-9.9 at. % Li we have chosen two temperatures to perform the reversibility checks as formerly described: 250 and 340 K. After the run 100-340-100

K the temperature was further increased up to 400 K and then decreased to 100 K. The sample was afterward warmed up to 400 K (without doing any measurements) and the S parameter was measured up to 550 K. A last run was made in the temperature range 550-100-550 K. Figure 1(b) shows the results obtained for the concentrated alloy (Al-9.9 at. % Li). After annealing at 250 K, four runs have been performed in the temperature range 250-100-250 K, confirming the reversibility of the curve in this interval. However, only two runs have been plotted in the figure for the sake of clarity; for the same reason the measurements corresponding to the run 100-550 K have also been omitted in the figure. After annealing at 550 K the S parameter is reversible and linear in the temperature range 550-100 K and the slope in this interval coincides with the value obtained in a similar well-annealed alloy measured at thermal equilibrium;⁵ therefore, we attribute the temperature dependence measured between 100 and 550 K after annealing at 550 K to thermal-expansion effects. The annealing behavior

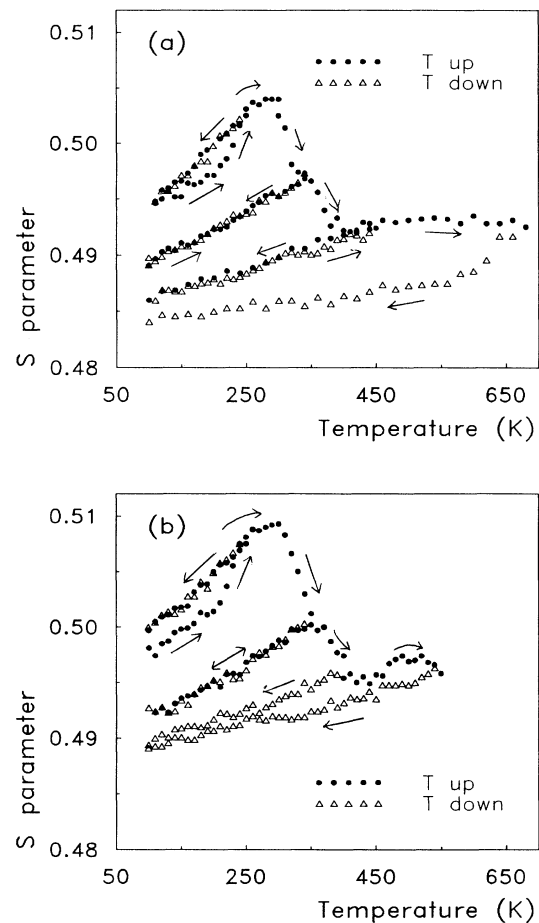


FIG. 1. The evolution of the S parameter as a function of the temperature for (a) an Al-0.2 at. % Li alloy deformed at 77 K and for (b) an Al-9.9 at. % Li alloy both deformed to 23% at 77 K. The measurements have been performed at the treatment temperature. The arrows indicate the measuring sequence.

for the as-deformed sample is qualitatively the same as in the Al-0.2 at. % Li alloy and the curve appears to be reversible in the temperature intervals 250–100–250 K and 340–100–340 K after annealing at 250 and 340 K, respectively.

In order to remove the thermal-expansion effects and additional temperature effects not originating from the deformation-induced defects, we have defined a normalized S parameter $S_n = [(S - S_a)/S_a]100$, where S is the S -parameter value at the measuring temperature and S_a the S parameter value at the same temperature after annealing at the highest temperature achieved by the sample. The data corresponding to each temperature range during the reversibility check can be satisfactorily fitted to a straight line and the slope values calculated for S_n are given in Table II.

To check the general evolution of the annealing curve and obtain some additional information concerning the type of defects involved in the annealing kinetics, lifetime measurements, once at the annealing temperature and once at a reference temperature (i.e., isochronal measurements), were performed in the Al-9.9 at. % Li alloy deformed to 22%. The measurements performed at the annealing temperature were carried out in the temperature range 100–620 K; after annealing at 620 K the sample was cooled down to 100 K and the S parameter was measured. The evolution of the lifetime parameters as a function of the temperature is shown in Fig. 2. The two-component decomposition is only shown in the range 450–620 K as the statistical scattering for lower temperatures is too high to consider the analysis reliable. The τ_2 component stays almost constant to a value of about 260 ps, whereas the associated intensity increases. However, by looking at the general behavior of the mean lifetime $\bar{\tau}$ it can be seen that the evolution between 100 and 400 K is similar to the one observed for the low- and high-Li content samples deformed to 23%: the mean lifetime increases to show a peak at 300 K and then a drastic decrease takes place.

The isochronal measurements were performed at 120 K and the recovery of the sample was followed between 100 and 620 K at temperature intervals of 20 K during heating periods of 2 h. The lifetime spectra were analyzed with only one component; more satisfactory fits were obtained with two components but since the scatter was very high, we have only plotted the mean lifetime $\bar{\tau}$. The data are also shown in Fig. 2. The most striking feature of these measurements is the constancy of the mean lifetime in the range 100–200 K, where the S parameter measured at the annealing temperature increases

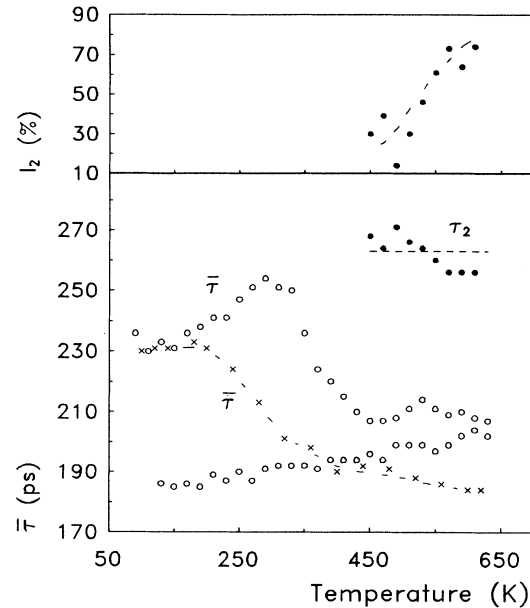


FIG. 2. The evolution of the lifetime parameters as a function of the temperature for an Al-9.9 at. % Li alloy deformed to 22% at 77 K. The open circles in the interval 100–620 K represent the measurement done at the annealing temperature, whereas in the interval 620–100 K they correspond to the data taken at the treatment temperature after the annealing at 620 K. For the same measurement sequence, the full circles correspond to the long lifetime and its intensity, respectively, for a two-component lifetime analysis between 450 and 620 K. The crosses correspond to the isochronal measurement at 120 K carried out in the same alloy deformed at 77 K to 22%. The lines have been drawn as a guide for the eye.

as a function of the temperature [see Figs. 1(a) and 1(b)]. The decrease in $\bar{\tau}$ for the isochronal measurements starts at 200 K where a noticeable increase is found in the same sample measured at the annealing temperature (see Fig. 2) and a change of slope is observed in the S parameter for the low- and high-Li content samples [see Figs. 1(a) and 1(b)].

B. Room-temperature deformed samples

The Al-0.2 at. % Li content alloy has been deformed to 22% at room temperature and the S parameter has been measured. Data were taken at the measuring temperature itself in the range 100–660–100 K. The evolution of S as a function of the temperature is shown in Fig. 3. In the cooling down run for the Al-0.2 at. % Li alloy, the data in the interval 100–550 K can be fitted to a straight line whose slope is close to the one calculated in the same temperature range for a well-annealed Al-0.2 at. % Li alloy measured at thermal equilibrium.⁵ Thus, we can conclude that this effect is due to thermal expansion. In the cooling down run above 550 K, the effect of positron trapping in thermally induced vacancies is visible (see Fig. 3). We can remove these effects by calculating the normalized S_n parameter. In the warming up

TABLE II. Values of the slope b (in units 10^{-3} K^{-1}) in the relationship $S_n = a + bT$.

Temperature interval (K)	9.9 at. % Li	0.2 at. % Li
200–100–200	5.0 ± 0.9	7.2 ± 0.7
250–100–250	7.8 ± 0.4	9.9 ± 0.5
340–100–340	4.1 ± 0.2	5.1 ± 0.3
400–100–400	1.2 ± 0.2	...
440–100–440	...	2.4 ± 0.2

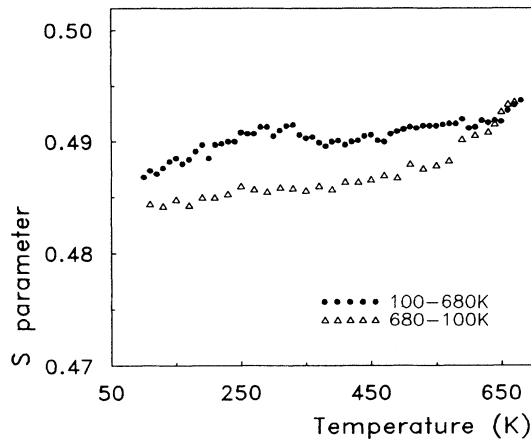


Fig. 3. The evolution of the S parameter as a function of the treatment temperature in Al-0.2 at. % Li alloy deformed at RT to 22%. After deformation, the measurements were started at 100 K.

run, the data between 100 and 300 K can be fitted to a straight line with slope $(2.7 \pm 0.3)10^{-3} \text{ K}^{-1}$, very close to the slope for the linear fit in the range 440–100–440 K for the low-content alloy deformed at 77 K (see Table II).

The Al-0.2 at. % Li alloy has been deformed at room temperature to 12% and the evolution of the lifetime parameters as a function of the temperature has been studied during an isochronal annealing experiment up to 775 K (30 min annealing time intervals, reference

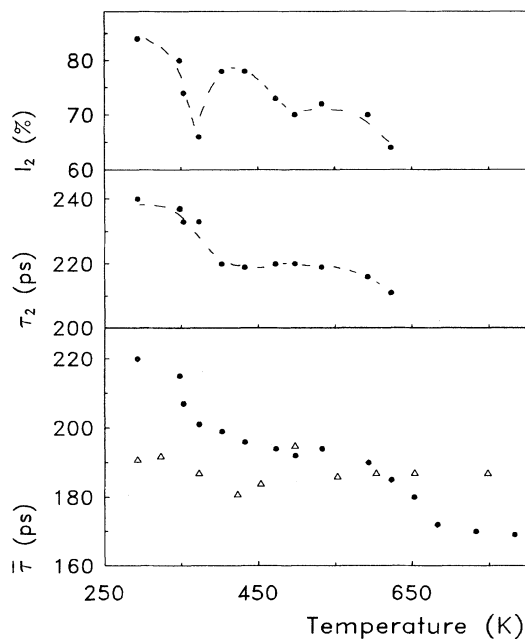


FIG. 4. Positron lifetime parameters for an isochronal annealing experiment on an Al-0.2 at. % Li alloy deformed at RT to 12% (full circles). The triangles represent the lifetime for an isochronal annealing performed in an Al-9.9 at. % Li alloy deformed at RT to 20%. The lines have been drawn as a guide for the eye.

temperature–room temperature) and the results have been plotted in Fig. 4. The spectra can be satisfactorily decomposed into two components up to 650 K. Above 650 K a single component, $\tau=165$ ps, is present in the sample; this coincides with the second annealing stage observed in the mean lifetime $\bar{\tau}$, and the value of the lifetime corresponds to the one currently attributed to bulk Al.⁶ The mean lifetime $\bar{\tau}$ displays two recovery stages: the first one is centered around 350 K and the second one around 600 K. The same stages are also present in the long lifetime τ_2 . From room temperature up to 375 K, the intensity I_2 corresponding to the long component decreases from 80 to 60 %. The τ_2 value at first stays constant at 240 ps and then drops to about 220 ps. Between 400 and 550 K the long lifetime component stays constant at this value while its intensity gradually decreases to 60%.

On the contrary, only one component could be resolved in the Al-9.9 at. % Li alloy deformed to 20% at room temperature, when the lifetime spectra were isochronally measured as a function of the temperature (30 min annealing intervals, reference temperature room temperature). The results are also shown in Fig. 4. The initial value in the as-deformed sample is 190 ps, i.e., only slightly above the lifetime attributed to free annihilation at the δ' phase⁷ and there is no significant change in the whole temperature range studied.

IV. DISCUSSION

In a deformed alloy dislocation S , vacancies and vacancy clusters are expected to be formed. According to previous positron data for pure Al, vacancies are immobile at 77 K,^{8,9} and they start migrating at around 200 K, giving rise to clusters in both quenched and heavily deformed Al,⁹ which are revealed by a drastic increase in the Doppler S parameter. Isochronal lifetime measurements performed at 120 K in quenched pure Al (Ref. 10) agree with this interpretation and they even showed the presence of small three-dimensional vacancy clusters in the temperature range 230–300 K, as revealed by the occurrence of a long component. The recovery temperature of dislocations in deformed Al, however, depends on both the deformation temperature and deformation degree.¹¹ In order to compare our results with the data in the literature for pure Al, we recall that the recovery temperatures for samples deformed at 77 K to 28 and 13 % are respectively 330 and 470 K, whereas the recovery is complete at 470 K for a sample deformed at room temperature to 24%.

Since we are concerned with the defect kinetics in the deformed alloys, we have represented the S_n parameter in Fig. 5 in order to remove any effect due to thermal expansion or to the formation of thermal vacancies. We will refer to this figure in our discussion.

A. Low-temperature deformed alloys

Inspection of Fig. 5 shows that the behavior of both the dilute and the concentrated alloys is very similar in the temperature range 100–350 K. The evolution of S_n

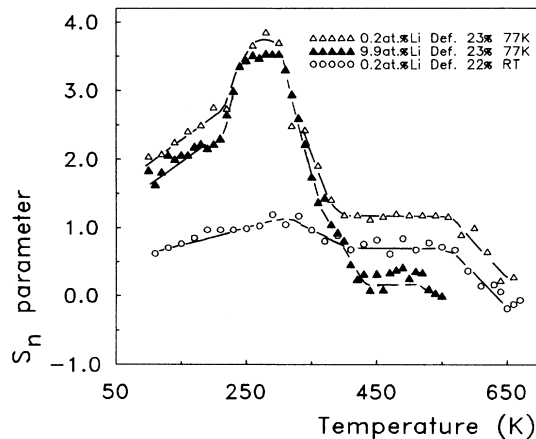


FIG. 5. The variation of the S_n parameter (see text for definition) as a function of the annealing temperature. The lines have been drawn as a guide for the eye.

as a function of the temperature is linear between 100 and 200 K and there is a clear change in the slope at about 200 K that leads to an increase in S_n , which exhibits a maximum at around 300 K. We recall that the Doppler parameter path is reversible between 100 and 200 K for the dilute sample and we can assume that it is also the case for the concentrated alloy. The presence of defects in the as-deformed samples is clearly seen in the S_n parameter value at 100 K.

Several works in the literature involving different measuring techniques and processes for the introduction of point defects in Al (Ref. 12) agree to interpret the stage observed at around 200 K as the migration of free vacancies in the lattice, thus we can interpret the change in slope in the S_n parameter observed around 200 K (see Fig. 5) as the migration of free vacancies formed in the alloy during deformation at 77 K. The increase in S_n from 200 up to 300 K can be attributed to the clustering of vacancies, as in previous works in pure Al (Ref. 9) and Al-Li alloys.^{2,13} The fact that after annealing at 250 K, the cooling down run (250–100 K) does not coincide anymore with the measured annealing curve, clearly indicates that the defect structure is not the same as in the as-deformed sample, supporting the hypothesis that vacancies migrate at 200 K, beginning to form three-dimensional clusters. The slope seen in the interval 250–100–250 K, reflects the temperature dependence of the specific trapping rate at voids.¹⁴ The decrease in the slope of the branch 340–100–340 K after annealing at 340 K, compared with the slope of the branch 250–100–250 K after annealing at 250 K (see Table II) is a consequence of a decrease in the concentration of voids, that have been almost completely removed at 340 K, in agreement with the literature.⁹ In the light of this reasoning we interpret the behavior of the S_n parameter during the annealing between 200 and 350 K as the migration and subsequent formation of large vacancy clusters that start dissolving at around 300 K.

The lifetime measurements carried out in the concen-

trated alloy (see Fig. 2) are in agreement with the previous interpretation. The occurrence of the long component $\tau_2 = 260$ ps above 450 K is interpreted as the contribution of thermally induced vacancies. According to Schaefer *et al.*,¹⁵ the drastic lifetime increase observed in pure Al at 480 K is due to the formation of thermal vacancies. By assuming the one-trap model in the temperature range 450–620 K with a trapping center characterized by a lifetime $\tau_2 = 260$ ps, the analysis of the data yields a τ bulk value of 195 ps, which is very close to the bulk value for the δ' phase.⁷

From the isochronal lifetime measurements performed at 120 K (see Fig. 2) it can be seen that the lifetime value measured in the as-deformed sample, $\tau \approx 230$ ps, stays roughly constant up to about 200 K, where it starts to decrease. It clearly indicates that the defect structure between 100 and 200 K does not change. It is to be noticed that the lifetime value in this temperature range is slightly lower than the currently attributed one to vacancies in Al, $\tau = 250$ ps.⁶ We recall that other defects characterized by a lifetime below 250 ps, i.e., dislocations, are present in the sample, giving rise to a mean lifetime $\bar{\tau}$ lower than the one corresponding to vacancies. Segers, Dorikens, and Dorikens-Vanpraet¹⁶ give a saturation value for the lifetime of 238 ps for a heavily deformed Al sample, and thus the lifetime value measured in the as-deformed state to a deformation degree of 23% (where saturation trapping does not necessarily occur) indicates an average value originating from several kind of defects. The constancy of the mean lifetime in the isochronal annealing experiment (see Fig. 2) in the temperature range 100–200 K indicates that the slope observed in the S_n parameter in the same temperature range (see Fig. 2) does not originate from a change in the trapping rate κ (trapping rate $\kappa = \mu C$) due to a change in the structure or density of defects but it rather suggests a temperature dependence of the specific trapping rate μ . It is well established that the specific trapping rate for vacancies is temperature independent¹⁷ and this is clearly seen in the work by Alam⁹ for quenched and deformed samples, where no temperature dependence is observed in the temperature range where vacancies are immobile. It is to be remarked that at such a low temperature, dislocations are present in the sample; they could also contribute to the observed temperature dependence, although the effect seems to be too large to correspond only to dislocations; in fact, the slope values in the range 200–100–200 K and in the range 440–100–440 K, where only dislocations survive, are not comparable (see Table II). From our results it is inferred that Li seems to be responsible for the temperature dependence observed in the alloy between 100 and 200 K. This effect cannot be attributed to the scattering of positrons at isolated Li atoms because it is only visible below 45 K for a concentration of 4 at. % Li, which is about the solubility limit of Li in Al.

From the above discussion we can conclude that measurements at the annealing temperature can be more useful than isochronal measurements to detect the clustering of vacancies when a broad variety of close lifetimes is present in the sample. In this case, the difficulty of decomposing the spectra in several contributions can

mask the presence of longer components of low intensity, whereas the formation of voids is accompanied by a temperature dependence of the specific trapping rate which is clearly revealed in the measurement at the annealing temperature itself.

The evolution of the S_n parameter above 350 K is clearly different for the dilute and the concentrated alloy (see Fig. 5). At 350 K, only dislocations, and perhaps some residual very large voids not affecting the trapping signal (positron diffusion length much smaller than the intervoid distance), should be present in both samples. The large plateau observed for the Al-0.2 at. % Li between 400 and 550 K can then be assigned to dislocations that anneal in the temperature interval 550–660 K. Bearing in mind the data in the literature for pure Al,¹¹ the dislocations would anneal below 470 K for the deformation process undergone by both alloys. The shift of the dislocation annealing stage, when compared with pure Al, evidences the pinning of dislocations by Li atoms in solid solution in the alloy.

The presence of δ' particles in the Al-9.9 at. % Li alloy has a significant influence in the dislocation recovery processes as observed in Fig. 5. The plateau assigned to dislocations is absent and the recovery seems to be completed at 440 K, as would be expected for pure Al. The isochronal lifetime measurements (see Fig. 2) indicate that the positrons annihilate at the δ' particles above 440 K, because the measured lifetime is very close to the bulk lifetime ($\tau = 185$ ps) for the δ' .⁷ Moreover, the bulk value obtained in the same high-content alloy measured as a function of the temperature by assuming the two-state model, support this hypothesis.

From this reasoning we can propose two alternative explanations: the dislocation pinning by Li in solid solution in the alloy is not acting when the δ' phase is present, or no significant trapping is taking place at dislocations. It is generally accepted that positrons are trapped at dislocations. Still, a controversy exists as to whether the positrons are trapped at the line itself or at the jogs.¹⁸ There is experimental evidence that deforming pure Al under very well-controlled conditions, giving rise to a low jog density, can lower the lifetime currently assigned to dislocations, $\tau = 240$ ps,¹¹ to 215 ps.¹⁹ We could interpret the low lifetime as positron trapping at dislocations exhibiting a very low jog density but we rather propose an alternative mechanism responsible for the low lifetime in the as-deformed sample. Positrons are initially trapped at dislocations associated to the δ' particles and most of them can easily reach the precipitates where they are annihilated in the free state. This would explain the presence of a lifetime close to the bulk of the δ' phase and the seemingly early dislocation recovery observed in the S_n parameter for the low-temperature deformed Al-9.9 at. % Li alloy.

B. Room-temperature deformed alloys

The lifetime value in the as-deformed state after deforming at room temperature the Al-9.9 at. % Li, $\tau = 190$ ps, is very close to the bulk lifetime in the δ' phase⁷ and thus we can interpret this result as in the previous paragraph. This lifetime value would be a conse-

quence of trapping at dislocations and subsequent free annihilation at δ' particles that are very efficient traps for positrons.

The plateau between 400 and 550 K (see Fig. 5) that we have assigned to positron trapping at dislocations in the low-temperature deformed Al-0.2 at. % Li alloy is also present when deforming at room temperature. For the room-temperature deformed Al-0.2 at. % Li alloy, it is seen that the S_n parameter decreases between 350 and 400 K (see Fig. 5). This indicates that the deformation at room temperature introduces some kind of defect besides dislocations.

The lifetime measurements performed in a 12% room-temperature deformed dilute alloy yield more precise information on the positron-defect interaction. According to our previous discussion, vacancies migrate well below room temperature. The generally accepted value for the lifetime of a positron trapped at a monovacancy in Al is 250 ps.⁶ The lifetime value measured immediately after the deformation of the dilute alloys at room temperature is very close to this value. From measurements on quenched alloys of the same type²⁰ it followed that vacancy-Li complexes were retained in the samples at room temperature. So the value of $\tau_2 = 240$ ps (see Fig. 4) measured immediately after deformation is attributed to the vacancy-Li complexes still present in the samples. The first annealing stage seen in the mean lifetime in Fig. 4 is attributed to a decrease of the vacancy-Li complexes concentration in the samples.

In the recovery curve of the room-temperature deformed samples, the positron lifetime drops to a value of 220 ps after annealing at 400 K (see τ_2 in Fig. 4). This indicates that from this temperature on, positrons are only trapped at the dislocations still present in the sample. The increase in the intensity I_2 can be attributed to an increase in the positron fraction annihilating at dislocations. In the temperature regime below 400 K vacancy-Li clusters coexist with the dislocations. From theoretical calculations of the positron defect binding energy we have to conclude that the trapping of positrons by the vacancy-Li clusters (positron binding energy is 2.7 eV for a vacancy-Li cluster with three to four vacancies)²¹ is much more efficient than the trapping of positrons by dislocations (positron binding energy at a jog at a dislocation line is 1.3 eV).²² This makes positron trapping more likely at vacancy-Li complexes in the temperature range between 300 and 400 K, even though their concentration is lower.

From this it also follows that the positron lifetime τ_2 in the temperature region between 300 and 400 K (which equals 240 ps) is a mean between the positron dislocation lifetime ($= 220$ ps) and the lifetime in the vacancy-Li complexes. From this it follows that the lifetime of the positron in the vacancy-Li complexes is certainly higher than 240 ps. This is in agreement with theoretical calculations²¹ where the positron lifetime in a vacancy-Li complex with three or four vacancies is predicted to vary between 253 to 256 ps (with the already mentioned binding energy of 2.7 eV).

In the temperature range between 400 and 600 K the intensity I_2 of the second positron lifetime decreases

gradually (see Fig. 4). It is known from the literature¹¹ that in pure Al, although the dislocation recovery depends on the degree of deformation and also on the deformation temperature, dislocations are completely removed at temperatures below 400 K. The fact that in the Al-0.2 at. % Li samples we still have trapping of the positrons in dislocations up to 600 K means that they are pinned by the Li atoms. At a certain temperature the pinning centers become unstable so that dislocations, initially decorated by Li, anneal out in a very sharp temperature stage, decreasing the lifetime of the trapped positron. This is seen in the evolution of the τ_2 at around 600 K. At that moment the second and the first lifetimes become indistinguishable from one another, giving rise to experimental one-component spectra, despite the fact that the intensity of the second lifetime component I_2 at 625 K is still around 60%. So the second recovery stage seen in the evolution of the mean lifetime in the temperature range from 575 to 700 K is due to the disappearance of the dislocations from the sample.

V. CONCLUSIONS

From this study it is seen that Li has a profound effect on the defect behavior in deformed Al-0.2 at. % Li and Al-9.9 at. % Li alloys. It also demonstrates that the positron-annihilation method is a very powerful measuring technique for the study of the defect kinetics during the annealing of deformed samples.

Two kinds of recovery measurements were performed (i) measurements at the treatment temperature itself and (ii) isochronal annealing measurements at a reference temperature well below the annealing temperature. The behavior of the obtained recovery curves is quite

different; for the measurements performed at the annealing temperature itself a maximum is seen in the annealing interval 200–350 K, whereas in the isochronal measurements a continuous decrease in the measured positron parameter ($\bar{\tau}$) is observed. This different behavior is explained by the temperature dependence of the positron trapping process at voids, demonstrating that by carefully using this temperature dependence of the positron parameters, more details can be obtained from an annealing curve when the underlying defect structure is quite complicated.

The main conclusions can be summarized as follows: (a) monovacancies become mobile at 200 K giving rise to clusters that dissolve at 350 K in both alloys; (b) in the Al-0.2 at. % Li alloy, dislocations are pinned by Li atoms postponing the complete recovery of the sample in comparison with pure Al; (c) in the Al-9.9 at. % Li alloy no positron trapping at dislocations is apparent above room temperature. In these alloys, δ' particles are present and from our measurements it follows that they are very efficient trapping centers for positrons. Positron trapping at δ' particles can also occur by an intermediate trapping state of the positron at dislocations localized by the δ' particles; (d) the defect structure in the Al-0.2 at. % Li alloy deformed at room temperature is influenced by the presence of the highly mobile vacancies during the deformation process. In these alloys, dislocations and vacancy-Li clusters are formed and positron trapping at both defects occurs. Vacancy-Li clusters dissociate at around 375 K.

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¹P. Hautojärvi, Mater. Sci. Forum **15-18**, 81 (1987).

²N. de Diego, D. Segers, J. del Río, L. Dorikens-Vanpraet, and M. Dorikens, J. Phys. Condens. Matter **3**, 5415 (1991).

³F. H. Samuel and G. Champier, J. Mater. Sci. **22**, 3851 (1987).

⁴J. del Río and N. de Diego, Phys. Status Solidi A **124**, K141 (1991).

⁵L. Dorikens-Vanpraet (private communication).

⁶A. Seeger and F. Bahnhardt, Phys. Status Solidi A **102**, 171 (1987).

⁷J. del Río, F. Plazaola, and N. de Diego, Scripta Metall. **26**, 1907 (1992).

⁸W. R. Wampler and W. B. Gauster, J. Phys. F **8**, L1 (1978).

⁹A. Alam, J. Phys. F **11**, L259 (1981).

¹⁰Cs. Szeles, Zs. Kajcsos, and A. Vértés, Phys. Rev. B **31**, 1302 (1985).

¹¹K. Petersen, in *Positron Solid State Physics*, Proceedings of the International School of Physics "Enrico Fermi," edited by W. Brandt and A. Dupasquier (Academic, New York, 1983), p. 298.

¹²R. W. Balluffi, J. Nucl. Mater. **69-70**, 240 (1978).

¹³H. P. Leighly, P. G. Coleman, and R. N. West, in *Proceedings of the Eighth International Conference on Positron Annihilation*, Gent, edited by L. Dorikens-Vanpraet, M. Dorikens, and

D. Segers (World Scientific, Singapore, 1989), p. 470.

¹⁴R. M. Nieminen, J. Laakkonen, P. Hautojärvi, and A. Vehanen, Phys. Rev. B **19**, 1397 (1979).

¹⁵H. E. Schaefer, R. Gugelmeier, M. Schmolz, and A. Seeger, Mater. Sci. Forum **15-18**, 111 (1987).

¹⁶D. Segers, M. Dorikens, and L. Dorikens-Vanpraet, in *Proceedings of the Seventh International Conference on Positron Annihilation, New Delhi*, edited by P. C. Jain, R. M. Singru, and K. P. Gopinathan (World Scientific, Singapore, 1985), p. 564.

¹⁷C. H. Hodges, J. Phys. F **4**, L230 (1974).

¹⁸L. Smedskjaer, M. Manninen, and M. J. Fluss, J. Phys. F **10**, 2237 (1980).

¹⁹C. Hidalgo, S. Linderroth, G. González-Doncel, and J. San Juan, in *Proceedings of the Eighth International Conference on Positron Annihilation, Gent* (Ref. 13), p. 371.

²⁰J. del Río and N. de Diego, Mater. Sci. Forum **105-110**, 965 (1992).

²¹J. del Río, F. Plazaola, N. de Diego, and P. Moser, Phys. Rev. B **47**, 2453 (1993).

²²H. Häkkinen, S. Mäkinen, and M. Manninen, Phys. Sci. **T33**, 216 (1990).