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Temperature dependence of positron trapping at defects in an Al–Li alloy

J del Río†, N de Diego† and F Plazaola‡

† Departamento de Física de Materiales, Facultad de Ciencias Físicas, Universidad Complutense, 28040 Madrid, Spain

‡ Elektrika eta Elektronika Saila, Zientzi Fakultatea, Euskal Herriko Unibertsitatea, 48080 Bilbo, Spain

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Abstract. The Al–3.7% Li alloy has been thermally treated in order to obtain samples containing vacancy–Li clusters and samples with both clusters and dislocation loops. The positron lifetime parameters have been measured in both cases as a function of the temperature in the interval 10–295 K. For the samples containing only vacancy–Li complexes two lifetimes are observed in the whole temperature range. The average lifetime, the long component and its associated intensity stay roughly constant from 100 up to 295 K and decrease in the interval 100–10 K. In the samples containing two kinds of trap for positrons, two lifetimes are observed only in the temperature region 100–295 K, where the presence of loops leads to a drastic decrease of the long lifetime and an increase in its associated intensity. In the temperature range 10–100 K the only resolved lifetime decreases as observed in the sample containing vacancy–Li complexes. The results are interpreted in terms of a strong temperature dependence of the trapping rate into loops and a reversible 'pinning' effect of the Li atoms on the open-volume defects.

1. Introduction

Al–Li based alloys are especially attractive materials to be studied with positrons, since their affinity to Li [1, 2] helps to detect small Li-rich zones in addition to dislocation loops and vacancy-like defects to which positrons are sensitive. In fact, the strong affinity of positrons to Li in Al–Li based alloys makes positron annihilation techniques very powerful to tackle a great variety of problems in these alloys such as precipitation processes [3] or lithium losses [4]. Positron affinity appears to play a very important role in the trapping processes in Al–Li based alloys. The results obtained in quenched Al–Li alloys [5] indicate a large enhancement of positron trapping into dislocation loops decorated by Li, that makes them competitive positron traps relative to vacancy clusters. The obtained value for an upper limit of the specific trapping rate into loops decorated with Li is 3.6 cm^2 s^{-1} [5], almost an order of magnitude larger than the specific trapping rate at dislocations in pure Al [6]. From these results it is inferred that the trapping characteristics in alloys where the positrons feel a strong affinity for one of the alloy elements are different to the characteristics in pure metals. However, in order to understand the complex positron behaviour in these materials and to be able to use the technique satisfactorily to identify defects, further studies need to be carried out.

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lt is well established that the positron lifetime in a defect-free metal exhibits a temperature dependence that is originated from thermal lattice expansion or contraction. Moreover, a characteristic temperature dependence has been also observed in the positron trapping at certain types of defect such as large voids [7] or dislocations [8]. However, to the authors' knowledge no studies have been carried out up to now to determine the temperature dependence of defects decorated with atoms exhibiting a high positron affinity. Apart from the interest of such studies from a basic point of view, the knowledge of the annihilation parameters as a function of the temperature can aid in identifying the type of defect present in a crystal and can be especially useful in the case of complex materials such as the mentioned alloys.

With the aim of deepening the study of the positron trapping characteristics in alloys where one of the components exhibits strong positron affinity, we have performed positron lifetime measurements as a function of the temperature from 10 to 300 K in two Al–Li alloys having different defect structure. We present the experimental details in section 2; section 3 is devoted to discussing the results and in section 4 we briefly outline the main conclusions.

2. Experiment

Two samples of the Al–3.74 at.% Li alloy supplied by Alcan Ltd (UK) were aged at 873 K under flowing argon for 31 and 9 h and subsequently quenched to 273 K. According to the literature Li is in solid solution for this concentration. Hereinafter the samples aged for 31 and 9 h will be denoted as A and B respectively. After the thermal treatment, sample B contains two kinds of positron trap, i.e., dislocation loops and vacancy–Li atom complexes [5]. However, in sample A the longer ageing time produces larger loops separated by a mean distance larger than the positron diffusion length. Therefore, in sample A only the vacancy–Li complexes can act as traps for positrons [5].

The samples were carefully chemically polished prior to the positron measurements in order to remove the Li-depleted zone close to the surface generated after the thermal treatment. The positron lifetime was measured for both samples A and B at 295 K and then the samples were cooled down to 10 K. Positron lifetime spectra were recorded as a function of the temperature from 10 to 295 K. Moreover, a run of measurements from 160 to 10 K was also carried out for sample A. The lifetime spectrometer was a fast–fast system having a resolution of 260 ps (FWHM). The positron emitter was a conventional ^{22}NaCl source deposited onto a Ni foil. The spectra were analysed with one or two components after subtracting the source contribution, that was extracted from the spectrum analysis of a well annealed 6N purity Al sample, whose surface has undergone the same treatment as the specimens to be measured.

3. Results

Figures 1 and 2 show the evolution of the lifetime parameters as a function of the temperature for samples A and B, respectively. In the case of sample A (see figure l), the spectra were in all cases satisfactorily decomposed into two lifetimes. Between 10 and 130 K, only one component could be resolved in the spectra corresponding to sample B (see figure 2), whereas from 150 up to 295 K two components were present in the sample. The positron lifetime parameters measured at 295 K are identical, within the experimental error, before and after the thermal run 295–10–295 K, indicating that the measurements are reversible in the temperature range 10–295 K. They are also in good agreement with previous results in similar samples [5].

Figure 1 shows the data obtained for the sample A (aged for 31 h at 873 K), where only one trap for positrons, i.e. vacancy–Li complexes, is present [5], Two regions are clearly observed. The first one corresponds to the interval 100–295 K, where the lifetime parameters remain roughly constant and close to the values measured at 295 K. The second region is placed between 100 and 10 K, where the long lifetime component τ_2 decreases from 275 ps down to 250 ps and the average lifetime $\bar{\tau}$ from 240 to 220 ps. The intensity I_2 corresponding to the long component diminishes slightly in the second region. In figure 1 the points corresponding to the run 293–10 K have been also plotted, clearly demonstrating the reversibility of the measurements in this temperature interval.

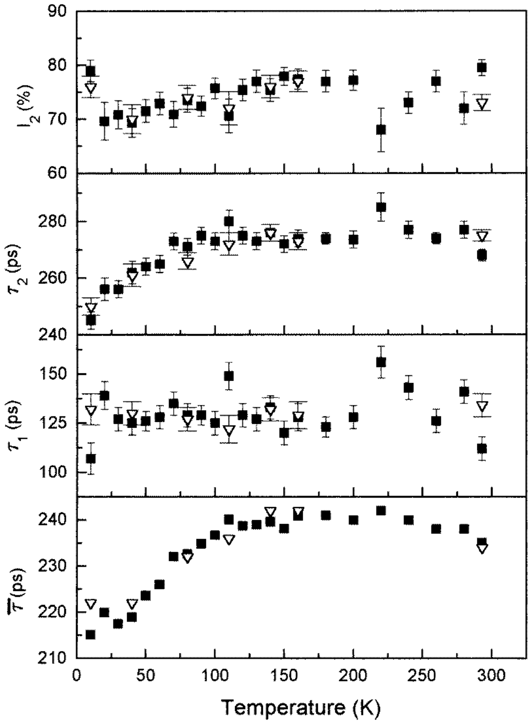

Figure 1. The evolution of the positron parameters as a function of the temperature in the Al–3.74 at.% Li alloy (A sample, see text for explanation). ■, 10–293 K run; ▽, 293–10 K run.

Figure 2 shows the results for the sample B (aged for 9 h at 873 K), which contains two different types of trap for positrons, i.e., dislocation loops and vacancy–Li complexes [5]. As in the previous case, two regions are clearly observed. In the temperature region between 10 and 140 K, only one component is present whereas in the interval 150–295 K the average lifetime splits into two components. The lifetime value τ_2 and its associated intensity I_2 are roughly constant between 240 and 295 K. However, from 240 down to 150 K, a monotonic decrease of τ_2 and an increase of the intensity I_2 are observed. In the temperature region 10–140 K, the lifetime decreases from 195 to 176 ps. It is to be remarked that the lifetime value measured at 10 K, $\tau = 176$ ps, is identical to the lifetime value measured in defect free Al–3.74 at.% Li alloy at room temperature [9].

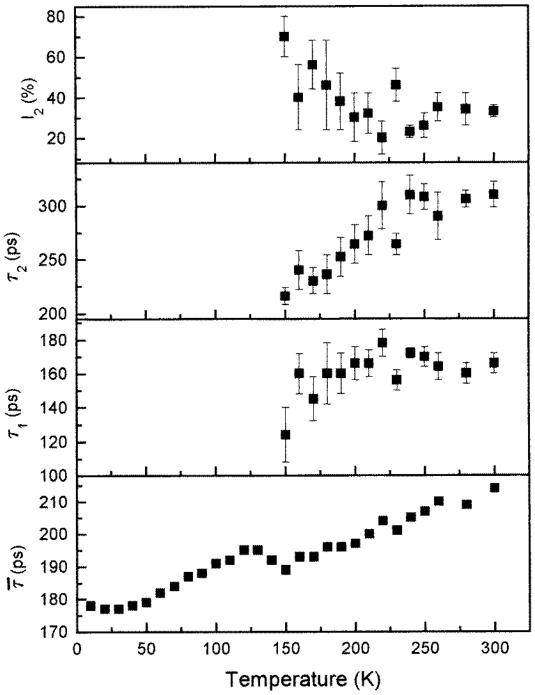

Figure 2. The evolution of the positron parameters as a function of the temperature in the Al–3.74 at.% Li alloy (B sample, see text for explanation).

The positron trapping behaviour at room temperature of the Al–3.74 at.% Li alloy has been discussed in detail in a previous work [5]. According to the results presented in that paper, the samples labelled A contain small vacancy clusters of different sizes stabilized by Li atoms whereas the samples labelled B contain both vacancy–Li complexes and dislocation loops formed by the collapse of larger vacancy–Li clusters. The loops, strongly decorated with Li, are more effective traps in comparison with the vacancy–Li complexes and therefore there is an enhanced specific trapping rate into loops [5].

Recalling that sample A contains only one type of trap for positrons, the measured lifetime τ_2 and its associated intensity correspond to trapping at vacancy–Li complexes. From the data displayed in figure 1 it is seen that the long lifetime stays constant above 120 K. Its mean value between 130 and 295 K is $\tau_2 = (275 \pm 3)$ ps and represents very probably an average of the lifetime values associated to complexes of different sizes. The intensity I_2 associated with this lifetime stays also roughly constant, within the experimental error, in the same temperature range. These results indicate that both the type and the concentration of defects do not change between 295 and 120 K. From 120 down to 10 K the lifetime τ_2 drops linearly from 275 to 250 ps and its associated intensity undergoes a slight decrease of approximately 8%. The observed changes cannot be interpreted as a change in the type and the density of the traps because the curve is reversible in this region. Therefore, we rather explain the decrease in the long component as a consequence of the volume contraction of the vacancy–Li clusters when the temperature decreases. Aina *et al* [10] have also reported a linear decrease with the temperature of the long component in an Al–Ca–Zn alloy containing vacancy-like traps associated with grain boundaries and have explained this result in terms of the presence of traps affecting the lattice thermal contraction. These authors have measured a temperature coefficient for the lifetime associated with vacancy-like defects present at grain boundaries which is about four times higher than the coefficient of volume expansion in pure Al and Al alloys. For the present measurements the temperature coefficient, $C = (\mathrm{d}\tau_2/\mathrm{d}T)/\tau_{2(ref)}$, has been calculated in the interval 10–110 K, taking $\tau_{2(ref)}$ as the mean value for τ_2 between 130 and 295 K, and it yields a value equal to $(1.02 \pm 0.08) \times 10^{-3}$ K^{-1}, which is higher by an order of magnitude than the corresponding value for Al and most Al alloys. However, it is very remarkable that this effect is only present from 120 K downwards, whereas Aina *et al* [10] have observed this regular decrease of the lifetime with the temperature in the whole measuring range from room temperature down to 10 K. This behaviour can be understood by the Li decoration of clusters, which prevents cluster shrinkage when the temperature decreases. This ‘pinning’ effect is apparent in the constancy of the long lifetime τ_2. When the lattice stress overcomes a critical value and the Li atoms are no longer able to hold the initial cluster volume, the free volume associated with the defect starts to decrease, producing a decrease in τ_2, as shown in figure 1. At the same time, the contraction of clusters having a very small free volume can be so high as to hinder positron trapping at them; thus, we can tentatively attribute the slight decrease in the intensity I_2 to a reversible diminution of positron traps.

In the sample labelled B (see figure 2) two kinds of trap for positrons are present, i.e., vacancy–Li atom complexes and dislocation loops; thus, the value of the intensity I_2 associated with the longer component represents the sum of the intensities I_{cl} and I_{dl}, corresponding to the vacancy–Li clusters and to the dislocation loops, respectively. The concentration of clusters is the same as in sample A [5] and therefore the temperature dependence of I_{cl} in sample B must reflect the same behaviour as in sample A. Under this reasoning, we cannot attribute the change of the intensity I_2 in the interval 150–250 K to a variation of I_{cl}, since it stays constant in this temperature range (see figure l). lt is to be recalled that the annihilation parameters measured at 295 K are identical before and after cooling the sample down to 10 K. Thus, this reversible change in I_2 can only be explained by a temperature dependence of the trapping rate into dislocation loops that increases with decreasing temperature, as has been previously observed in metals containing dislocation loops [11] and more particularly in pure Al [12], where the increase in the intensity corresponding to loops reflects the enhancement of the trapping rate into dislocation loops. However, it is to be remarked that in pure Al the intensity undergoes a change of approximately 3% between 200 and 150 K [12] whereas in our case the effect is much more pronounced with an intensity increase of 40% in the same temperature interval. It seems that the presence of Li atoms decorating the dislocation loops is responsible for the anomalous increase of the intensity associated with dislocation loops as compared with pure Al.

As far as the I_2 component is concerned, inspection of figure 2 demonstrates that τ_2 stays roughly constant from 295 up to 230 K and drops drastically from this temperature on. The observed decrease in the lifetime reflects also the enhancement of the trapping rate into dislocation loops and is due to the competition trapping between loops and clusters. To ensure that our hypothesis does not come from artifacts derived from spectra analyses, we reanalysed our data by fixing the long component τ_2 to 310 ps, which is the value corresponding to clusters. Figure 3 shows the constrained analyses for sample B. It clearly shows the monotonic decrease of I_2 and increase of τ_1 when the temperature diminishes up to 170 K. This behaviour indicates the decrease of positron trapping at vacancy clusters, even though the vacancy-cluster concentration does not change, as indicated by the reversibility of the measurements exhibited by sample A. Therefore, the plots shown in figure 3 reinforce

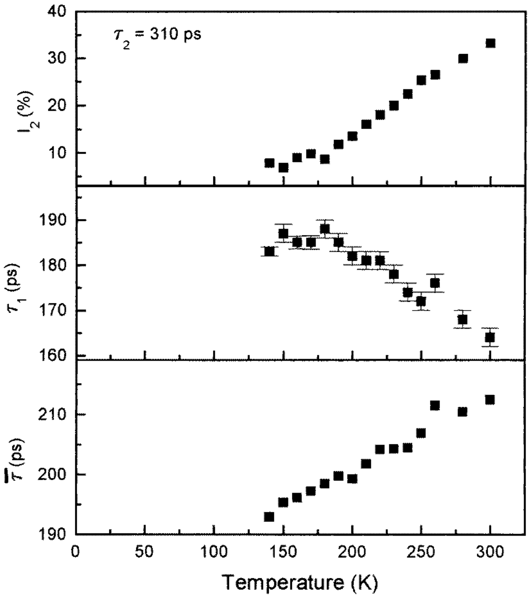

Figure 3. The evolution of the positron parameters as a function of the temperature in the B sample (constrained analyses by fixing the long lifetime component).

the previous hypothesis of a remarkable increase of positron trapping at dislocation loops when the temperature decreases.

For a temperature close to and lower than 150 K the specific trapping rate into dislocation loops is so high that positrons are trapped in saturation at these defects and as a consequence one only component is found (see figure 2). The temperature at which saturation trapping at loops occurs is probably around 120 K, where the lifetime is equal to 192 ps, which is close to the theoretical value [13].

If saturation trapping takes place at loops, no temperature dependence of the lifetime should presumably be observed below 120 K. However, the lifetime decreases regularly with the temperature up to 40 K, where the lifetime value characteristic of the defect-free alloy is attained. We have fitted the lifetime values to a linear dependence with the temperature and have extracted a temperature coefficient from it in the same way as explained for the A sample. It is defined in this case as $C = (\mathrm{d}\tau_2/\mathrm{d}T)/\tau_{(ref)}$, with $\tau_{(ref)} = 192$ ps, which we attribute to the lifetime associated with loops. From the experimental data we obtain a value for C equal to $(1.12 \pm 0.04) \times 10^{-3}$ K^{-1}, which coincides, within the experimental error, with the temperature coefficient extracted for the sample containing vacancy–Li complexes. It is also remarkable that the lifetime decrease starts at about 120 K, which is approximately the critical value where the lattice stress due to contraction overcomes the 'pinning' effect of the Li atoms that decorate the loops. Therefore, as in the preceding case, we can interpret the lifetime decrease down to 40 K as an effect of the lattice contraction when the temperature decreases. As explained before for the vacancy–Li complexes, the shrinkage of the dislocation loops can lead to a noticeable decrease of the open volume associated with them hindering positron trapping. In the case of dislocation loops this effect seems to be very strong and at very low temperatures positrons annihilate with the lifetime of the defect-free alloy.

4. Conclusions

The Li atoms present in the studied diluted Al–Li alloy play an important role in the trapping processes. The temperature dependence of the trapping rate into loops is confirmed; however, the presence of Li leads to a very pronounced enhancement of the positron trapping into dislocation loops.

The results suggest that the Li decoration of defects prevents the lattice from contraction in the defect region when the temperature decreases. The critical temperature where this ‘pinning’ effect disappears is approximately 100 K.

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