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Li clustering in Al–Li alloys studied using positrons

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Abstract. The behaviour of two Al–Li dilute alloys having two different Li contents (1.7 and 3.7 at.%Li) has been studied using positron annihilation. The lifetime value is about 20 ps higher than that measured for pure Al and is independent of the Li content. When the specimens are cold rolled the lifetime increases to a value that is lower than the one currently associated with dislocations in Al and does not depend on the Li concentration. After isochronal annealing the lifetime achieves the initial value $\tau = 185$ ps. The results are interpreted in terms of Li clustering.

1. Introduction

The positron–defect interaction in pure metals is now rather well understood; however, to date very little information has been obtained on the behaviour of positrons in dilute and concentrated alloys.

When dealing with alloys several effects must be considered where positron trapping is concerned. Besides the well known trapping centres, such as vacancies and dislocations, in a material containing second-phase particles the positrons can be trapped and annihilated at the interface of the matrix and precipitate or by defects present in the precipitates themselves. Since the positron is sensitive to the local core potential, in an alloy it can show a tendency towards being localised and annihilated in zones where the Coulomb repulsion is lower. The positron affinity for one of the components in a metallic alloy has been treated both theoretically and experimentally (Lock and West 1974, Stott and Kubica 1975, Kubica *et al* 1975, Koenig 1978). Dlubek *et al* (1979, 1981, 1986) have also proved experimentally that in Al–Zn alloys the positrons tend to be localised in Zn-rich zones. These extra effects that affect the behaviour of positrons in an alloy can thus provide useful information on the very early stages of precipitation and can help with the detection of very small impurity clusters that cannot be detected using other techniques.

In recent years positrons have been used satisfactorily to follow precipitation processes (Dlubek *et al* 1979, 1981, 1986, Panchanadeeswaran *et al* 1984, Lühr-Tank *et al* 1987) and it has also been demonstrated that the positron method can provide information on the composition of Guinier–Preston zones in Al–Zn alloys (Dlubek *et al* 1986).

The Al–Li alloys have received a great deal of attention because of their importance in the aerospace industry and it is well established that their properties are strongly

Table 1. Positron lifetimes in Al–Li alloys.

Sample	Thermal treatment	τ (ps)
Al–1.7 at.% Li	Quenched from 820 K	189 ± 2
Al–1.7 at.% Li	Annealed at 820 K for 1 h	184 ± 2
Al–3.74 at.% Li	Quenched from 820 K	186 ± 2
Al–3.74 at.% Li	Annealed at 520 K for 1 h	191 ± 2
Al–3.74 at.% Li	Annealed at 520 K for 6 h	190 ± 2
6N purity Al	Annealed at 800 K for 3 h	167 ± 2

influenced by the formation of second-phase particles. According to theoretical predictions, positrons show a high affinity for Li-rich zones in an Al matrix (Stott and Kubica 1975). Thus the positron annihilation technique can be a useful tool for investigating the microstructure of these alloys.

Very recently, Leighly *et al* (1989) have measured the Doppler-broadening S-parameter for Al–Li alloys and they have interpreted their results in terms of vacancy–Li interactions and Li clustering.

We have carried out positron annihilation measurements on Al–Li alloys with the aim of contributing to the understanding of positron localisation in alloys by providing experimental data. In this work we report on the positron response in two Al–Li alloys and on the effect of Li on the recovery of the alloys after plastic deformation.

2. Experimental details

Two Al–Li alloys containing 1.7 and 3.74 at.%Li have been studied. The latter was supplied by Alcan Ltd while the Al–1.7 at.%Li alloy was prepared by ourselves in the following way: to prevent oxidation, the lithium (90% enriched ^{6}Li) was handled under paraffin oil. The paraffin was then replaced by n-pentane, combining a protective function and high volatility. Then the sample was transferred inside a crucible of the host material into a high-vacuum induction furnace. After evaporation of the protective liquid, the melting process was performed under an atmosphere of purified argon. The samples were homogenised at 820 K for 10 min and then quenched to 273 K. Several thermal treatments to be specified later were performed in an Ar atmosphere. The samples were also plastically deformed by cold rolling at room temperature to thickness reductions of 20 and 48% for the Al–1.7 at.%Li and the Al–3.74 at.%Li alloys, respectively. The isochronal annealing measurements were carried out at room temperature in 50 K steps for heating periods each of 1 h. The positron lifetime spectra were recorded at room temperature using a fast system. About 15 μCi of ^{22}NaCl aqueous solution deposited onto a thin nickel foil (0.45 mg cm^{-2}) was used as a positron source. In all cases the positron spectra could be decomposed into one component, after subtracting the source contributions.

3. Results and discussion

The results for the undeformed samples are summarised in table 1, where the value for well annealed 6N purity Al has also been given, for comparison. As can be seen, the

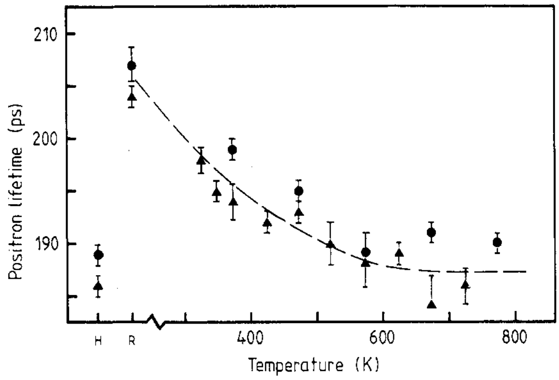

Figure 1. The evolution of the average lifetime as a function of the temperature in two Al–Li alloys rolled to produce two different degrees of deformation. The lifetime values for the two alloys in the homogenised and as-rolled state have been marked in the figure as the points H and R, respectively. ●, Al–1.7 at.% Li, 20% thickness reduction; ▲, Al–3.7 at.% Li, 48% thickness reduction.

positron lifetime is independent of the Li content; furthermore, the thermal treatments do not noticeably affect the lifetime value. These results indicate that the nature of the trapping centres is the same for the two alloys in the as-quenched state, and that the defects responsible for the observed trapping signal are retained during the annealings.

In figure 1 the average lifetime is plotted as a function of the temperature for the deformed samples. The lifetime value is higher than that for the as-quenched and annealed specimens; the recovery is very smooth up to approximately 620 K, where the average lifetime achieves the value characteristic of the undeformed samples. No substantial differences are observed between the two sets of samples studied. Since the measured lifetime value for well annealed 6N purity Al is $\tau = 167$ ps, the lifetime value $\tau \simeq 188$ ps obtained for the as-quenched alloys indicates some kind of trapping phenomenon associated with the presence of lithium. According to the literature the lithium is in solid solution for both alloys (Williams and Edington 1975); thus, the interaction of positrons with second-phase particles can be ruled out. It might be suggested that the observed trapping signal is due to vacancies retained during quenching. Vacancies in Al are known to migrate below room temperature (Petersen 1983). It is also a well established fact that the addition of impurities to a pure metal can shift the vacancy migration temperature to higher values (Hautojärvi *et al* 1985, Moser *et al* 1987) and that impurities can decorate vacancies, giving rise to a decrease in the lifetime value characteristic of this defect (Hautojärvi *et al* 1985).

The current reported positron lifetime value for vacancies in pure Al is $\tau_v = 240$ ps (Petersen 1983), thus, a reduction of about 50 ps for the decorated vacancy seems too high in comparison with other results extracted from the literature. Recent calculations by Puska and Manninen (1987) show that the effect of decoration of vacancies in Al by light impurities such as Li is weak. On the other hand, the observed trapping signal is maintained even after annealing at high temperature for long periods, indicating that the trapping centre responsible for the measured lifetime is very stable. According to Ceresara *et al* (1977), vacancy–impurity pairs retained during quenching migrate below room temperature in Al–Li dilute alloys. All the above mentioned arguments lead us to conclude that Li-decorated vacancies are not the defects giving rise to the observed lifetime. The theoretical calculations (Stott and Kubica 1975) predict that positrons tend to be localised in Li-rich zones in an Al matrix; thus, the present results can be interpreted instead in terms of positrons localised and annihilated in Li-rich zones. It is to be noted that the spectra are satisfactorily decomposed into just one component, suggesting that the measured saturation signal can be ascribed to annihilation in Li clusters.

The value $\tau \simeq 188$ ps is closer to the lifetime value characteristic of pure Al than to that for Li, which has been reported to be $\tau = 300$ ps (Lock and West 1974, Kubica *et al* 1975). Recent theoretical results on the Zn clustering in Al–Zn alloys predict a dependence of the positron lifetime as a function of the cluster size ranging from the Al bulk value, when no Zn clustering takes place, to the Zn bulk value when the cluster contains about 12 or more Zn atoms (Bharathi and Chakraborty 1988). Since the value we have measured is closer to the Al bulk value we believe that the Li cluster consists of very few atoms. Furthermore, the cluster size seems to be independent of the Li content, at least in the concentration range studied.

When the alloys are plastically deformed the trapping signal increases to $\tau \simeq 205$ ps in both cases, regardless of the degree of deformation. In room-temperature plastically deformed pure Al it is mainly dislocations that are to be expected; positron experiments performed on pure Al deformed at room temperature have demonstrated that the trapping signal increases with the degree of deformation and that a high defect concentration favours recovery (Petersen 1983).

The increase observed in the lifetime for the as-deformed samples has been interpreted as arising from the trapping of positrons at the defects created during rolling. The measured lifetime value $\tau \simeq 205$ ps represents an average of the values for positrons trapped in Li clusters and defects created by deformation. It is to be noted that the contribution of Li clusters to the lifetime is the same for both the alloys studied, and the value after deformation is independent of the reduction in thickness, whereas in the case of pure Al the higher the degree of deformation, the higher the trapping signal.

These results suggest that the defects formed during deformation are mainly asociated with Li clusters. The presence of the clusters causes the degree of deformation at which saturation trapping is observed to be less than that for pure Al. This effect has also been observed in an Al–4 at.% Cu containing precipitates (Panchanadeeswaran and Byrne 1981).

4. Conclusions

Two Al–Li alloys containing 1.7 and 3.74 at.% Li have been studied by positron annihilation. The positron lifetime is independent of the Li content and is longer than that for pure Al, suggesting that positrons are localised and annihilated in small Li clusters. The defects formed during deformation in the cold-rolled alloys are mainly associated with Li clusters.

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