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Precipitation behavior investigated through positron annihilation in Sc-doped Al-6Mg followed by the effects of Zr-addition

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Abstract

Phase decomposition in Al–6Mg alloy doped with Sc up to 0.6 wt.% was first investigated through positron lifetime and coincidence Doppler broadening spectroscopic (CDBS) measurements. The results varied significantly with the degrees of doping and heat treatment conditions due to the entrapment and annihilation of positrons at vacancies and lattice irregularities like coherent and semi-coherent precipitate-matrix interfaces. Sc-vacancy complexes helped in fine scale precipitation of Al_3Sc during the annealing. The substructure stabilization is effected more at low annealing temperature and shorter annealing times. The precipitation behaviour in 0.2 wt.% Zr-doped Al-6Mg-0.4Sc alloy under different annealing conditions was also studied. Although Sc has better diffusivity in Al-6Mg than Zr, the latter appeared to be an ideal additive to generate new precipitates of the form $Al_3Sc_{1-x}Zr_x$ and the differences are reflected in the positron lifetimes and CDBS ratio curves. Transmission electron microscopy showed spheroidal precipitates with complete absence of facets, implying the modification of the surface morphology of the precipitates.

Keywords : aluminium alloys, defects, phase decomposition, positron annihilation, precipitation

1. Introduction

In a previous work, we had shown that the phase decomposition in Al–6Mg can be varied significantly with the degrees of doping and heat treatment conditions [1]. This happened due to the entrapment and annihilation of lattice irregularities like impurities and vacancies and coherent and semi-coherent precipitate-matrix interfaces. Heat treatments at different temperatures and for various durations help in understanding the kinetics of precipitate formation and growth and the techniques of positron annihilation are highly suitable to look at the formation, migration and growth of precipitates in solid matrices. In this work, we report the results of positron annihilation

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studies on Sc-doped Al-6Mg alloy and the effects of addition of Zr that is well found to be an additive material for enhanced precipitation and growth.

2. Experimental Details

For the preparation of the samples, elemental aluminium and the aluminium-scandium master alloy (2 wt.% Sc) were melted in a clay-graphite crucible. Required quantity of magnesium ribbons of high purity were then added into the melt. Melting was carried out in a resistance-heating pot furnace under suitable flux cover. Several cycles of solidification and melting were done to achieve uniform composition of binary Al-Mg and ternary Al-Mg-Sc alloys of varying Sc content. The molten alloys were allowed to cool at the rate $20Ks^{-1}$ in a metal mould and solidified and then cut into suitable pieces using a low speed diamond wheel saw for subsequent characterization.

For positron lifetime measurements, we used a slow-fast gamma-gamma coincidence set-up and the spectra were analysed using PALSfit computer program [2]. A single Gaussian fit with a resolution about 170 ps full width at half the maximum gave satisfactory results with essentially two positron lifetimes. For the CDBS experiments, two high pure Ge detectors of resolution 1.29 and 1.33 keV at 511 keV were used. The ^{22}Na source used had a strength of about 10 μCi and was in the deposited form on a thin nickel foil. The necessary source corrections had been incorporated while analyzing the positron lifetime spectra.

3. Results and Discussion

Figure 1 illustrates the changes in positron lifetimes and the intensity versus Sc-doping and at the final addition of Zr. Sc reduces the positron lifetimes, indicating its active role in modifying the electronic environment around the positron trapping centres. In other words, Sc-vacancy complexes are being formed, which later during the annealing helps in fine scale precipitation as Al_3Sc, a fact verified from selected area diffraction patterns. A point worth noting is that the substructure stabilization is effected more at low annealing temperature and shorter annealing times. The precipitation of Al_3Sc starts from the onset of annealing and increases in number due to the enhanced dissociation of Sc-vacancy complexes at high annealing times.

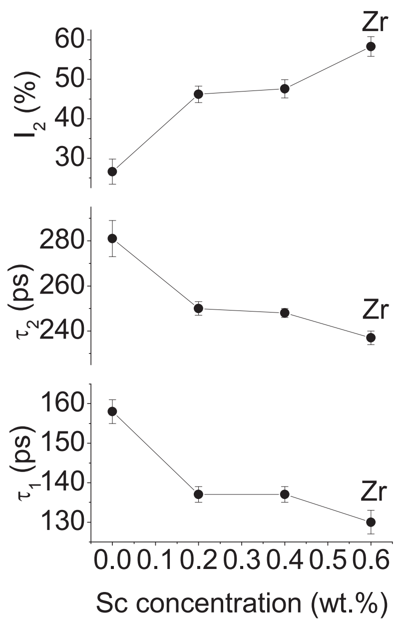


Figure 1. The positron lifetimes τ_1 and τ_2 and intensity I_2 in the Al-6Mg, Al-6Mg-0.2Sc, Al-6Mg-0.4Sc and Al-6Mg-0.4Sc-0.2Zr (shown against x = 0.6) samples.

The vacancy type defects aided the formation of precipitates. In the undoped basic alloy Al-6Mg, positrons were entrapped at interfaces of Mg_5Al_8 precipitates. The concentration of these precipitates increased considerably upon annealing the alloy at higher temperatures. This is indicated by a substantial increase of the positron lifetime τ_2 after the annealing at 673K for longer durations (Figure 2). The precipitation of Al_3Sc in the Sc-doped alloys is influenced by the annealing time and temperature which determine the degree of dissociation of Sc atom-vacancy complexes (Figure 3). Sc atoms are therefore found highly efficient in the formation of new phases of precipitation and growth, thereby implying to affect the hardening of the alloy after different concentrations of doping and annealing at different temperatures. The above findings are supported from transmission electron microscopy (Figures 4(a) and 4(b)). The images indicate the formation and coarsening of the precipitates at different stages.

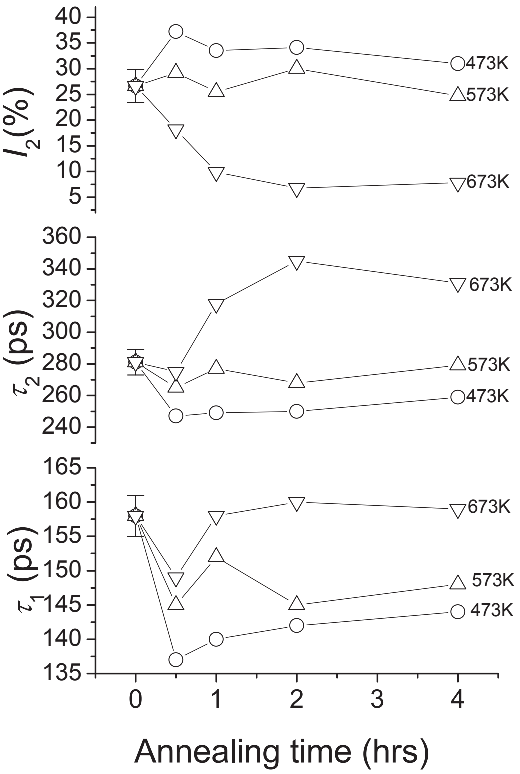


Figure 2. The isothermal variation of the positron lifetimes τ_1 and τ_2 and relative intensity I_2 of the alloy Al-6Mg at three different temperatures 473K, 573K and 673K.

While the positron lifetimes and intensities indicated the magnitude of the precipitate size and density, the CDBS curves were helpful in monitoring the changes of predominant trapping sites for positrons at the different stages of doping and alloying. The proximity and final overlap of the CDBS quotient curves with the line representing Al as shown in Figure 5 indicate that Sc atoms basically interacted with Al for the formation of precipitates whereas Mg atoms remained out of their influence.

The addition of Zr in trace amounts to the alloy Al-6Mg-0.4Sc gave additional insight into the precipitation aspects through distinct changes in the positron annihilation parameters. On the basis of the changes in variations of the positron annihilation parameters of the two alloys Al-6Mg-0.4Sc (Figure 3) and Al-6Mg-0.4Sc-0.2Zr (Figure 6), it can be seen that the addition of Zr in Al-6Mg-0.4Sc leads to the formation of precipitates of the form $Al_3Sc_{1-x}Zr_x$. These precipitates are resistant to annealing and stabilize dislocation substructure by way of pinning them.

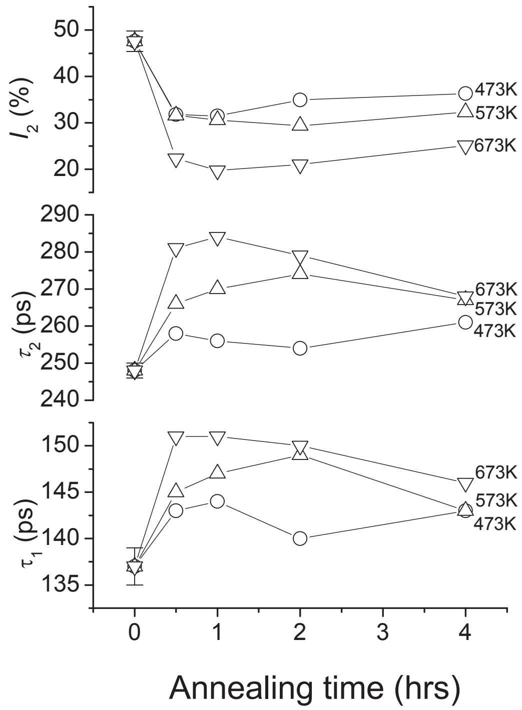
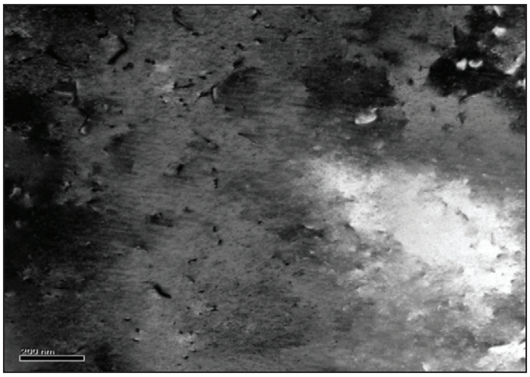


Figure 3. The isothermal variation of the positron lifetimes τ_1 and τ_2 and relative intensity I_2 of the alloy Al-6Mg-0.4Sc at three different temperatures 473K, 573K and 673K.



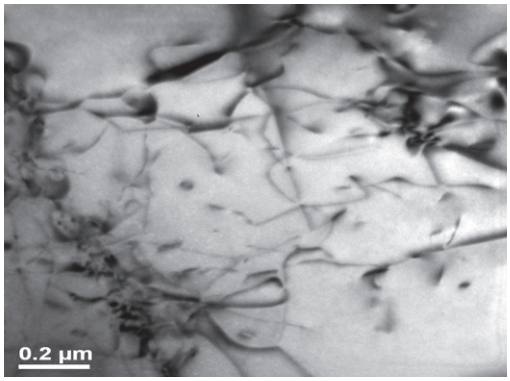


Figure 4. Transmission electron microscopic images of (a) Al-6Mg after annealing at 673K and (b) Al-6Mg-0.4Sc after the annealing at 473K.

The annealing of the Zr-doped alloy at high temperatures leads to the segregation of free Zr atoms at the surfaces of precipitates. Further, these atoms are responsible for the spherical morphology of precipitates in contrast with faceted morphology of binary Al_3Sc precipitates (Figures 7(a) and 7(b)). It is thus demonstrated that Zr is an ideal choice for aiding the formation of such precipitates and may help in the modification of the mechanical properties of such alloys. As for example, the ultimate formation of Zr-vacancy complexes can help to adequately compensate for the removal of vacancies otherwise possible at higher annealing temperatures. TEM observations also revealed the defect evolution and interaction stages and indicated the formation of non-faceted precipitates in the alloys.

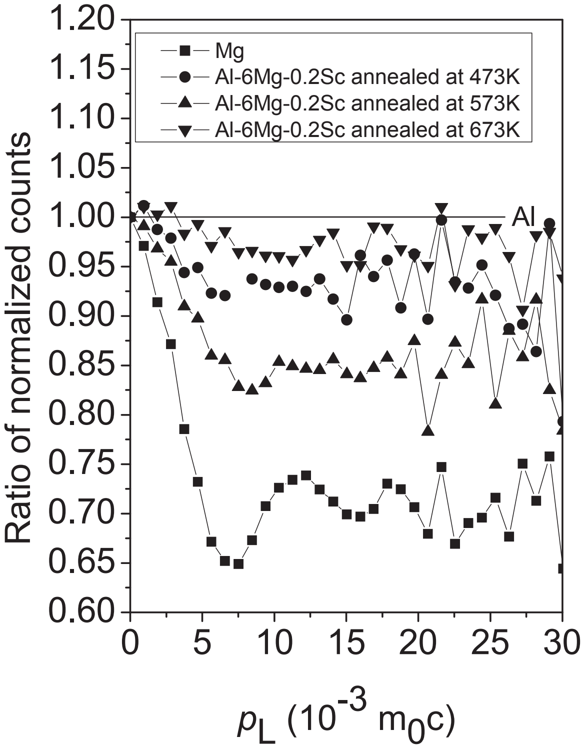


Figure 5. The CDB spectra of the Al-6Mg-0.2Sc alloy after the final (4 hrs) annealing at 473K, 573K and 673K. The curve of Mg is also shown. The spectra are normalized with that of Al, shown by the horizontal line at *y* = 0.

4. Conclusions

This study demonstrates the influence of addition of Sc and further of Zr on the formation and evolution of binary and ternary precipitates in Al-6Mg alloy due to isochronal and isothermal annealing at different temperatures and durations. The positron lifetimes and CDBS curves were found rather sensitive to these changes and hence their variation could reflect the growth of such precipitates in the alloy. Initially we found a gradual decrease of the positron lifetimes due to the incorporation of Sc and later of Zr. This is expected due to the enhanced electron density created by the presence of these atoms. The subsequent changes are brought in while the alloys were subjected to heat treatments. Mg_5Al_8 precipitates are found to influence the course of variation of the positron lifetimes and intensities in the basic ally Al-6Mg while Al_3Sc did it in the Sc-doped alloy. With the addition of Zr, a new kind of precipitates of the composition $Al_3Sc_{1-x}Zr_x$ took the major rule in defining the trend of variation of the positron annihilation parameters. The study proved to be useful in understanding the effects of addition of such additive species like Sc and Zr on the precipitation and segregation behaviour in solid systems such as Al-6Mg which have got important technological applications.

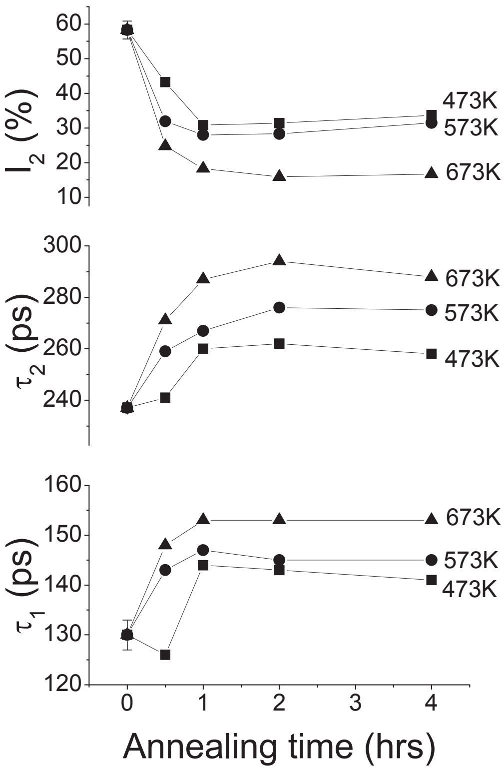


Figure 6. The isothermal variation of the positron lifetimes τ_1 and τ_2 and relative intensity I_2 of the alloy Al-6Mg-0.4Sc-0.2Zr at three different temperatures 473K, 573K and 673K.

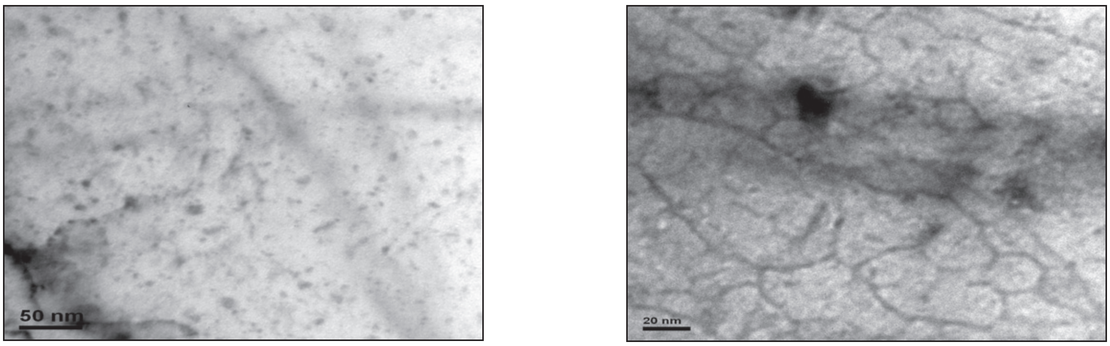


Figure 7. Transmission electron microscopic images of (a) Al-6Mg-0.4Sc as-cast alloy and (b) Al-6Mg-0.4Sc-0.2Zr after the annealing at 673K.

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