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The positron trapping efficiency of dislocations in deformed dilute aluminium alloys

E Hashimoto, M Iwami and Y Ueda

Laboratory of Crystal Physics, Faculty of Science, Hiroshima University, Higashi-Hiroshima 724, Japan

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Abstract. Positron-lifetime measurements have been made on deformed Al–0.01 at.% Mg, Al–0.01 at.% Si and Al–0.01 at.% Cu alloys at room temperature. The specific trapping rate μ_d of positrons into dislocations depends strongly on alloying elements. Mg and Cu atoms can reduce μ_d to a great extent, while Si atoms are not so effective. These results are closely related to the pinning of dislocations by solute atoms.

1. Introduction

In positron studies of defects in metals, the specific trapping rate for unit defect concentration has an important bearing on the understanding of the trapping mechanism as well as the determination of defect concentrations [1]. Therefore, considerable experimental and theoretical effort has been made to determine the correct value of the specific trapping rate for various types of defect. However, there is a scatter in the experimental values, especially for dislocations. For example, a large spread, $(0.066\text{–}2) \times 10^{-4}$ m^2 s^{-1}, exists for Al [2]. This spread exceeds uncertainties in the experimental determination of dislocation density.

In our recent experiment on deformed Al [3], it has been found that impurities can reduce the positron trappings efficiency of dislocations even in trace amounts. Besides, in high-purity samples, the trapping efficiency decreases with increasing dislocation density.

In this paper, we present a study of the positron trapping efficiency of dislocations in deformed dilute Al–Mg, Al–Si and Al–Cu alloys. In these alloy systems, the dislocation–solute atom interaction has been measured by amplitude-dependent internal friction. The systematic study of identical impurity elements can make significant advance in our understanding of the origin of the effect of impurities described above.

2. Experimental procedure

The materials used in this study were Al–0.01 at.% Mg, Al–0.01 at.% Si and Al–0.01 at.% Cu alloys. Polycrystalline samples were annealed at 773 K for Al–Mg alloy and at 823 K for Al–Si and Al–Cu alloys, and they were cooled down to room temperature in the furnace. The samples were then chemically etched in aqua regia and rinsed in distilled water. Several of them were used as annealed samples, and others were deformed prior to positron annihilation measurements.

The deformation was accomplished at room temperature by rolling to various extents of thickness reduction, 0.9–11%. After deformation, the samples were cut into a square of

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side 15 mm, and were chemically etched again. The deformed samples thus prepared were 1–1.3 mm in thickness.

Positron-lifetime measurements were performed at room temperature with a fast–fast timing spectrometer with a time resolution of 217 ps (FWHM). The positron source and sample configuration used was a conventional sandwich type. The source was prepared by deposition of about 1.5×10^6 Bq of ^{22}NaCl on Kapton foil. The component due to annihilations in the salt and foil was obtained from positron-lifetime spectra for well annealed Al (99.999% purity). The spectra could be decomposed into two lifetime components: the shorter ($\tau \simeq 160$ ps) due to the free annihilation in the bulk and the longer (400 ps) with intensity $\simeq 10\%$ associated with the source effect. The source contribution was subtracted from the spectra in the analyses. The analyses were made by the POSITRONFIT program [4].

Doppler-broadening measurements were also made on the 9% deformed Al–Cu sample with a Ge detector system, with a resolution of 1.69 keV (FWHM) at 1.33 MeV. The sample was measured at temperatures in the range 4.2–250 K with a temperature stability better than ±1 K. The shape of the Doppler-broadened photopeaks was characterized by a conventional peak-height (S) parameter.

The dislocation density N_{d} in the deformed sample actually used in the positron measurement was determined from the residual resistivities at liquid-He temperature: $N_{\mathrm{d}} = (\rho - \rho_0)/\rho_{\mathrm{d}}$, where ρ and ρ_0 are the residual resistivities of the sample before and after annealing at 773 K for 2 h, respectively, and ρ_{d} is the resistivity per unit density of dislocations at liquid-He temperature. The residual resistivity was measured by the standard four-terminal method with a superconducting chopper amplifier, with a voltage sensitivity better than 5 pV [5]. The value of $1.2 \times 10^{-25}\,\Omega$ m^3 [6], obtained for dislocations in pure Al, has been used for ρ_{d}, since it is little known how ρ_{d} depends on the alloying element and its content.

3. Results and discussion

The positron-lifetime spectra for the annealed samples could be described with only one lifetime component $\tau = 158$–160 ps, independent of alloying elements. The τ value is in agreement with those quoted for the bulk lifetime τ_{f} in pure Al.

A two-component analysis gave satisfactory fits for all the deformed samples studied. Figure 1 shows the two components obtained, τ_1 and τ_2, and the longer-component intensity I_2, plotted as a function of the dislocation density N_{d}. In the figure, the positron-lifetime results for deformed Al with different impurity content are also plotted for comparison.

The longer component τ_2 seems to be, within its statistical accuracy, independent of the alloying element, the impurity content and the dislocation density, with a mean value $\tau_2 \simeq 235$ ps. The shorter component τ_1 agrees, within about 10 ps, with the value deduced from the simple trapping model assuming that only one kind of positron trap, characterized by a lifetime τ_2 is present in the sample. The positron lifetime of about 235 ps is close to the lifetime found for annihilation in dislocations in Al; hence we conclude that the positron trapping component originates from trapping at dislocations, and that the active trapping sites of the dislocation remain unchanged in all the deformed samples studied.

The specific trapping rate μ_{d} into dislocations was obtained from the following formula: $N_{\mathrm{d}}\mu_{\mathrm{d}} = I_2(\lambda_1 - \lambda_2)$, where λ_1 ($\equiv 1/\tau_1$) and λ_2 ($\equiv 1/\tau_2$) are the experimentally measured decay rates.

Figure 2 shows μ_{d} plotted as a function of N_{d} for deformed Al and dilute Al alloys. In deformed Al, as already reported [3], impurities even in trace amounts can reduce μ_{d},

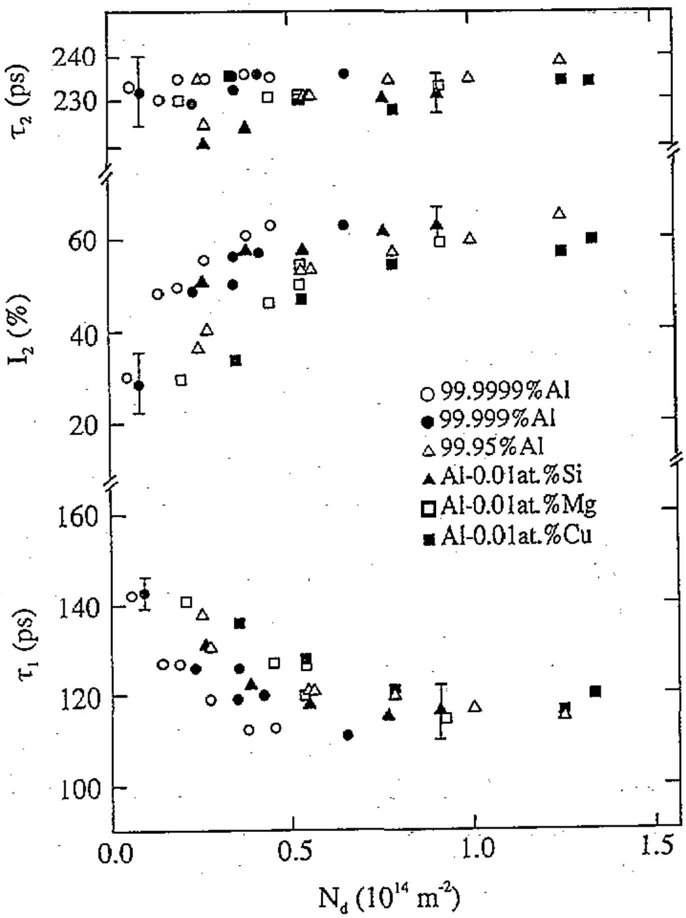

Figure 1. Positron-lifetime parameters plotted as a function of dislocation density N_d for deformed Al and dilute Al alloys.

namely, the effective trapping efficiency of dislocations. Besides, the μ_d in high-purity Al decreases rapidly with increasing dislocation density N_d.

The main features of the specific trapping rate μ_d in deformed dilute Al alloys are as follows: (1) in Al–0.01 at.% Si alloy, μ_d and its dependence on N_d are almost the same as those observed in deformed Al of 99.999% purity; (2) Mg and Cu atoms can reduce μ_d to a great extent; (3) the μ_d in Al–Mg and Al–Cu alloys is only weakly dependent on N_d. To interpret these features, details of the interaction between a dislocation and a solute atom are necessary.

Recent measurements of amplitude-dependent internal friction in dilute Al alloys [7, 8] provide information very useful to the present study. (A) Mg and Cu atoms interact strongly with dislocations, and segregate on the dislocation line to high concentrations. The binding energies between a dislocation and a pinning solute atom are determined as 0.19 and 0.195 eV for Mg and Cu solute atoms, respectively. (B) Si atoms precipitate into small clusters on the dislocation line because of their low solubility even in Al–0.01 at.% Si alloy. Thus the pinning concentration on the dislocation line in Al–0.01 at.% Si will decrease in inverse proportion to the number of atoms in a cluster, and becomes lower

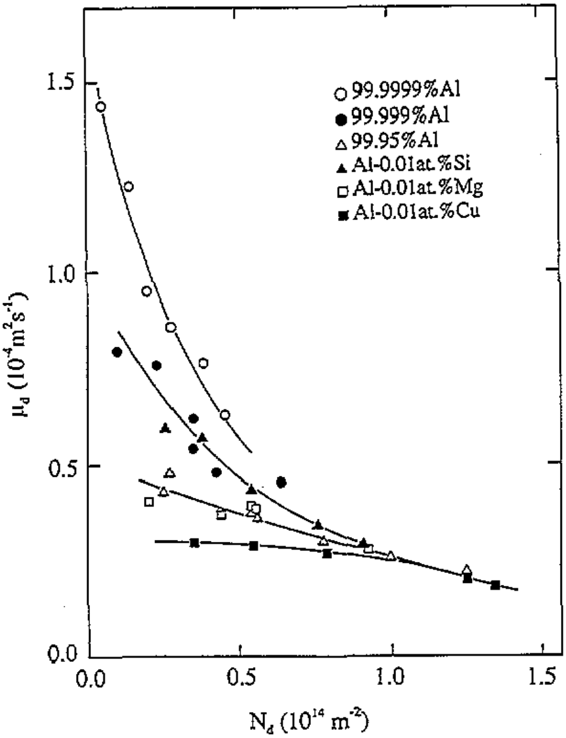

Figure 2. Specific trapping rate μ_d of positrons into dislocations plotted as a function of dislocation density N_d for deformed Al and dilute Al alloys. The lines are drawn to guide the eye.

than in Al–0.01 at.% Mg and Al–0.01 at.% Cu alloys. Incidentally, the value of 0.12 eV has been obtained as the binding energy between a dislocation and a single Si atom in neutron-irradiated high-purity Al [9].

The features of μ_d in deformed dilute alloys, (1)-(3), in connection with the dislocation–solute atom interaction quoted above, suggest that the effective positron trapping efficiency of dislocations is closely related to the pinning concentration on the dislocation line. The trapping efficiency decreases with increasing impurity pinning points.

By analogy of the effect of impurity pinning points, the trapping efficiency may be also influenced by the formation of pinning points (e.g. junctions) resulting from dislocation interaction either during deformation or during the subsequent recovery. In fact, a rapid decrease in μ_d with increasing N_d is clearly observed in deformed pure Al and Al–0.01 at.% Si alloy, where the effect of impurities is less pronounced. Besides, μ_d becomes independent of the sample purity or alloying elements for a large density of dislocations, $N_d > 10^{14}$ m^{-2}. However, to clarify the nature of such pinning points, further studies are required.

The temperature dependence of the positron annihilation parameters in deformed dilute alloys is useful to understand the effect of dislocation pinning described above. Figure 3 shows the lineshape parameter S plotted as a function of temperature for the 9% deformed Al–0.01 at.% Cu sample. S decreases with increasing temperature up to about 100 K, showing two different temperature regions. This behaviour of S can be well described in terms of a generalized trapping model that includes positron trapping at dislocation lines (shallow traps) from which positrons can either be transferred into associated deep traps (e.g. jogs) or be thermally detrapped. The temperature dependence of S below about 30 K is essentially due to the temperature dependence of the trapping rate to the dislocation line, while above 30 K the temperature dependence is due to thermal detrapping of positrons from dislocation lines. An analysis based on a model proposed by Smedskjaer *et al* [10] yields a value of about 10 meV for the positron–dislocation-line binding energy E_b.

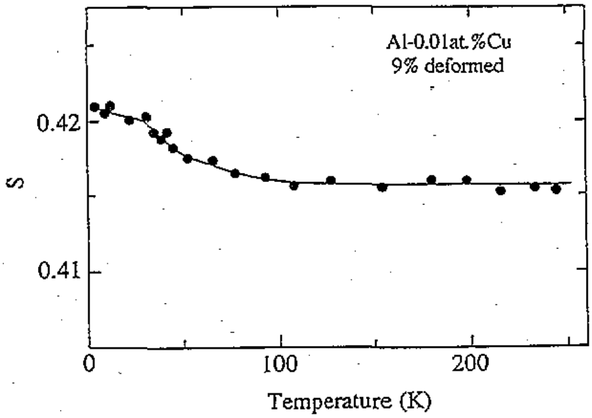

Figure 3. Peak-height lineshape parameter S plotted as a function of temperature for deformed Al–0.01 at.% Cu alloy. The line is drawn to guide the eye.

The dislocation in Al–0.01 at.% Cu alloy, as found in deformed pure Al [11], can also provide shallow traps for positrons, and can act as a stepping-stone to deeper traps. Besides, the value deduced for E_b is almost the same as that found for dislocations in deformed Al of 99.9999% purity [11], despite a reduction in μ_d to a great extent. According to the trapping model mentioned above, the effective positron trapping efficiency of dislocations depends strongly on the rate of transition to jogs via dislocation lines. Thus it is presumable that pinning obstacles impede the positron's free motion along the dislocation line, retarding the transition rate to jogs. This must necessarily lead to a reduction in μ_d. A situation where only some of the dislocations can be a precursor state for deeper traps will be established when the pinning concentration becomes as high as in, for example, Al–0.01 at.% Cu alloy.

In this study, positron-lifetime measurements have been made on deformed dilute Al–Si, Al–Mg and Al–Cu alloys at room temperature. It has been found that the positron trapping efficiency of dislocations is closely related to the pinning concentration on the dislocation line. Pinning obstacles presumably impede the positron's free motion along the dislocation line and retard the rate of transition to jogs (deep traps) via dislocation lines (shallow traps), thereby resulting in a reduction in the effective trapping efficiency of dislocations for positrons.

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