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Change in the positron-trapping efficiency of dislocations in Al on heating after deformation

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Abstract. Positron-lifetime measurements have been made in order to study how the positron-trapping efficiency of dislocations, μ_d, in Al (99.9999% purity) changes during isochronal annealing after deformation at room temperature. It has been shown that μ_d goes on increasing during the anneal, while the lifetime τ_d for trapped positrons decreases at first but then becomes a constant, $\simeq$210 ps. The highest value of μ_d obtained was about 4.5×10^{-4} m^2 s^{-1}, much larger than previous values, $(0.066–2) \times 10^{-4}$ m^2 s^{-1}. After heating at a higher temperature, it has also been shown that a small applied stress can yield a marked decrease in μ_d without any significant change in τ_d. These results suggest that μ_d depends strongly on dislocation configurations. The increase in μ_d during the anneal may be due to a development of long straight dislocation-line segments.

1. Introduction

Since the first realization of the sensitivity of positrons to lattice defects introduced by deformation [1, 2], considerable effort, both experimental and theoretical, has been made to understand the interaction of positrons with dislocations in metals. In particular, much attention has been focused on the temperature dependence of the positron-trapping rate at dislocations, since it can provide useful information about the nature of the positron trap associated with dislocations and the corresponding trapping mechanism. A reasonable amount of experimental data have been satisfactorily interpreted in terms of a model that allows positrons initially trapped at dislocation lines (shallow traps) to get trapped in dislocation-associated deep traps (e.g. jogs) or to become thermally detrapped [3].

According to the trapping model mentioned above, a number of factors can influence the positron-trapping efficiency μ_d (i.e. the trapping rate per unit density) of dislocations. In fact, there is a large spread in the experimental μ_d-values, exceeding uncertainties in the determination of the dislocation density. For example, $\mu_d = (0.066–2) \times 10^{-4}$ m^2 s^{-1} for Al [4]. However, the complexity of the trapping mechanism at dislocations makes it difficult to assess which factor is dominant in a given situation. This is a serious problem in attempts to understand the positron–dislocation interaction.

Our recent experiments carried out on deformed Al and dilute Al alloys [5, 6] have suggested that the trapping efficiency μ_d depends strongly on dislocation configurations formed under interactions among aggregates of dislocations and those with impurity atoms. These interactions undoubtedly contribute to the formation of complex configurations during deformation, thereby resulting in a large spread in μ_d as quoted above. Consequently, the

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systematic study of the configuration dependence of μ_d should help us to delve deeper into the problem of the positron–dislocation interaction.

In this paper, we first present a study of how the trapping efficiency μ_d changes on heating deformed 99.9999% Al. A notable effect of heating after deformation is to drive tangled dislocations toward a straight configuration. Next, we present a preliminary experiment on how μ_d changes under a small applied stress after a development of relatively long straight dislocation-line segments.

2. Experimental procedure

The material used in this study was zone-refined Al of 99.9999% purity. Two kinds of specimen were prepared: one was a polycrystal and the other a single crystal oriented for single slip.

Polycrystalline specimens (1 × 15 × 15 mm) were cut and chemically etched with aqua regia. Then they were annealed in vacuum ($\sim 1 \times 10^{-4}$ Pa) at 600 °C for 6 h and furnace cooled to room temperature. These specimens were deformed at room temperature by rolling to a thickness reduced by 5%.

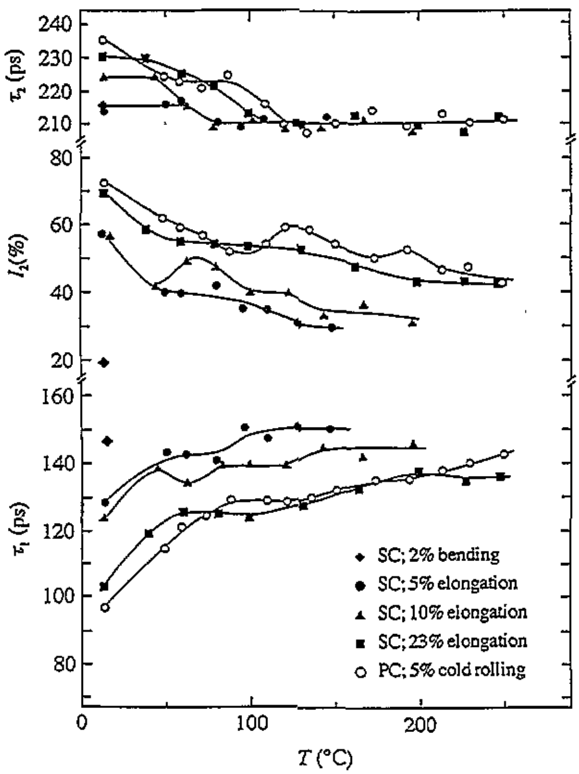

Figure 1. The change in the positron-lifetime parameters on heating deformed polycrystalline Al (PC) and single-crystal Al (SC) specimens of 99.9999% purity. The annealing was for 1 h at each temperature T. The lines are drawn to guide the eye.

Single-crystal specimens (0.9 × 12 × 70 mm) were prepared from a zone-refined Al rod by spark erosion. The main surface of the specimens was cut parallel to the $(14\bar{2})$ crystallographic plane, and the specimen axis was parallel to the orientation [213]. The axis orientation is chosen to be near the middle of a stereographic triangle (i.e. as far as possible from the principal symmetry direction), so that under deformation the resolved shear stress is greatest on a single-glide system. Both the surface normal and axis orientation were determined by the transmission Laue method to an accuracy $\pm 2°$.

Four single-crystal specimens were electropolished and cyclically annealed between 190 and 300 °C six times in vacuum for 48 h to decrease the dislocation density. These specimens were deformed at room temperature: one of them was strained by 2% by three-point bending around an axis parallel to $[\bar{2}11]$, and the others were strained by 5%, 10% and 23%, respectively, by elongation in the direction of the specimen axis. Specimens for positron-annihilation measurements were cut from the central part of each deformed single crystal by spark erosion.

For the deformed specimens, isochronal anneals were taken in vacuum for 1 h every 15–30 °C between 40 and 250 °C, followed by positron-lifetime measurements performed at room temperature. The time resolution of the apparatus was 230 ps (FWHM). The positron source was prepared by deposition of about 1.5×10^6 Bq of ^{22}NaCl on Kapton foil. The component due to annihilation in the salt and foil was obtained from positron-lifetime spectra for well annealed Al specimens (99.9999% purity). The spectra could be decomposed into two lifetime components: the shorter ($\tau = 158$–160 ps) due to the free annihilation in the bulk and the longer (400 ps) with intensity of about 10% associated with the source effect. The source contribution was subtracted from the spectra in the analyses. The analyses were made using the POSITRONFIT program [7].

The average dislocation density N_d in the specimen actually used in the positron-annihilation measurement was estimated at several stages in the annealing procedure from the residual resistivities at liquid-He temperature: $N_d = (\rho - \rho_0)/\rho_d$, where ρ and ρ_0 are the residual resistivities of the specimen before and after the final anneal at 300 °C, respectively, and ρ_d is the resistivity per unit density of dislocations at liquid-He temperature. The resistivity ρ_d should be independent of dislocation configurations, since the wavelength of conduction electrons on the Fermi surface in Al is only about 4 Å. Thus the generally accepted value, $1.2 \times 10^{-25}\ \Omega\ \mathrm{m}^3$ [8], was used. The residual resistivity was measured with a superconducting chopper amplifier, with a voltage sensitivity of 2 pV [9].

3. Results and discussion

The positron-lifetime spectra were satisfactorily fitted with two exponential decay components. Figure 1 shows the two components obtained, τ_1 and τ_2, and the longer-component intensity I_2, plotted as functions of the annealing temperature.

For the polycrystalline Al specimens, the positron lifetime of the trapped component, τ_2, is about 235 ps after deformation by cold rolling (multiple slip). This value is in agreement with those previously quoted for dislocations in Al. This value also is close to but a little smaller than the positron lifetime in monovacancies in Al, $\simeq$240 ps [10].

For the single-crystal specimens, on the other hand, τ_2 in the deformed state increases from 215 to 230 ps with increasing elongation. This is consistent with recent experiments carried out on deformed Al by Hidalgo *et al* [11]. These authors found that the positron lifetime associated with dislocations, τ_d, increases with an increasing number of slip systems activated: τ_d is 215, 220 and 240 ps when single-, double-, and multiple-slip planes are activated, respectively. To examine this effect, in figure 2 we show a stereographic triangle indicating the axis orientations of our deformed single-crystal specimens. Obviously, for elongations in excess of 10%, the axis orientations become more distant from those predicted for an ideal single slip on the primary system $(1\bar{1}1)[011]$, implying that at least two slip

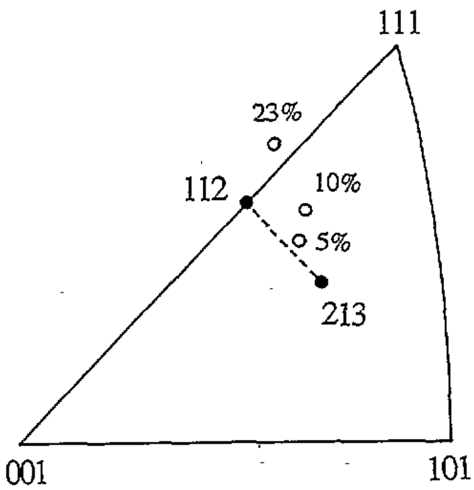

Figure 2. A stereographic triangle indicating the axis orientations of the single-crystal Al specimens strained by 5%, 10% and 23%, respectively, by elongation. The original axis orientation of the specimen is parallel to [213]. The dotted line shows the change in the axis orientation predicted for specimen elongation via an ideal single slip on the primary system $(1\bar{1}1)[011]$.

systems are activated. Hidalgo *et al* also suggested that the different lifetime τ_d is to be attributed to different structures of jogs [11].

The lifetime τ_2 (i.e. τ_d), decreases at first during the isochronal anneal, but becomes a constant, $\simeq$210 ps, for all the specimens studied. This may be caused by the forming of simple jog configurations with a development of straight dislocation configurations during recovery after deformation, such as the growth of three-dimensional dislocation networks.

The network growth is a typical phenomenon most frequently observed during recovery, and is thought to be important in the present study. In the network growth, dislocations reduce their length by straightening with the help of slip and climb. The underlying driving force is, of course, the extra energy present in the deformed state in the form of dislocations. Consequently, it might reasonably be expected that the lifetime of 210 ps is due to positron annihilation in well separated single jogs on relatively long straight dislocation-line segments. After the final anneal at 300 °C for 3 h, the lifetime spectra could be described with only one component, τ = 158–160 ps, corresponding to the positron lifetime in the perfect lattice, τ_f.

The two-component trapping model was found to give satisfactory fits of the lifetime spectra. The effective positron-trapping rate κ_{eff} may, therefore, be obtained from $\kappa_{eff} = (1 - \bar{\tau}\lambda_f)/(\bar{\tau} - \tau_2)$, where λ_f ($\equiv 1/\tau_f$) is the positron-annihilation rate in the perfect lattice, and $\bar{\tau}$ is the mean lifetime deduced from $\bar{\tau} = I_1\tau_1 + I_2\tau_2$. Here, I_1 is the intensity of the shorter component τ_1 and satisfies a relation $I_1 + I_2 = 1$.

Figure 3 shows κ_{eff} plotted as a function of annealing temperature. The trapping rate κ_{eff} basically decreases with increasing temperature, but exhibits a small hump or a plateau after an initial rapid decrease. The dislocation density N_d, on the other hand, falls monotonically as shown in the inset. Thus the behaviour of κ_{eff} above will be explained in terms of a competition between the elimination of dislocations during the anneal and the increase in the positron-trapping efficiency of the remaining dislocations.

The trapping efficiency μ_d was estimated from $\mu_d = \kappa_{eff}/N_d$. Figure 4 shows μ_d plotted as a function of N_d. The main result is that μ_d increases significantly with a reduction in N_d

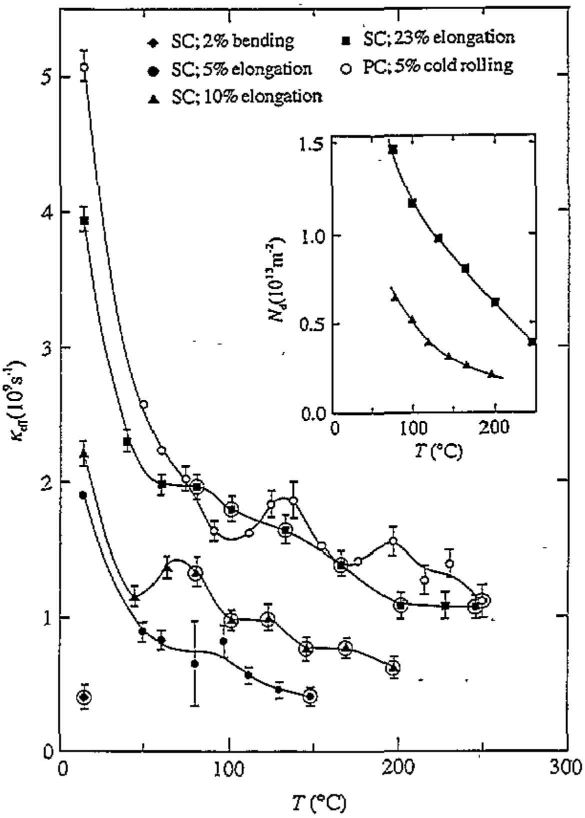

Figure 3. The change in the effective positron-trapping rate κ_{eff} on heating deformed polycrystalline Al (PC) and single-crystal Al (SC) specimens of 99.9999% purity. Encircled symbols show the points where the residual resistivity was measured after annealing treatment. The inset shows the change in dislocation density N_d during annealing. The lines are drawn to guide the eye.

during recovery after deformation. As already noted, three-dimensional dislocation networks grow coarser and coarser as recovery proceeds, thereby reducing their dislocation content. Straightening movement of dislocations plays the leading part here. Therefore, it is clear that μ_d depends strongly on the length of straight dislocation-line segments: the longer the straight-line segment the larger the value of μ_d. In fact, μ_d reaches about 4.5×10^{-4} m^2 s^{-2} at a value of N_d of less than 3×10^{12} m^{-2}, after the straight configurations have become well developed. This value for μ_d is much larger than previous values, $\sim(0.066\text{–}2) \times 10^{-4}$ m^2 s^{-1}. In the deformed state, on the other hand, a relatively small μ_d is found even at a value of N_d of 2×10^{12} m^{-2}, introduced by bending (single slip), since the straightening movement is insufficient at room temperature. The configuration dependence of μ_d must necessarily lead to a large spread in its experimental values.

The way μ_d increases during the anneal should depend on the ease with which the network growth occurs. As is well known, the growth of a dislocation network is liable to be stopped by a locking point, such as a dislocation junction, where the available movement is insufficient to break it [12]. The network is then in a metastable state—in other words, there is an energy barrier which has to be overcome to permit further growth. Thus it is reasonable to expect that a higher temperature or a longer time is necessary to overcome

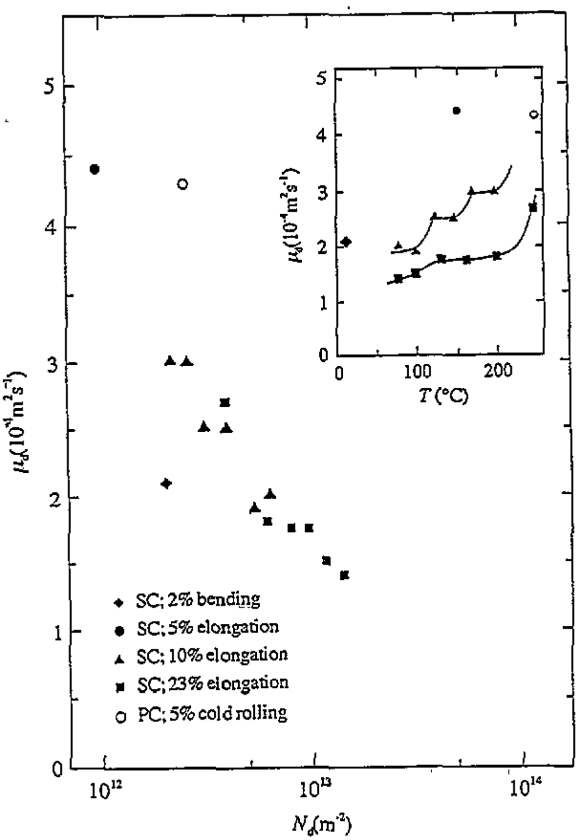

Figure 4. The positron-trapping efficiency of dislocations μ_d plotted as a function of the dislocation density N_d remaining during annealing for deformed polycrystalline Al (PC) and single-crystal Al (SC) specimens of 99.9999% purity. The inset shows μ_d plotted as a function of annealing temperature T. The lines are drawn to guide the eye.

the energy barrier when an initial deformation structure becomes more complicated—that is when more than two slip systems are activated. This feature is reflected in the temperature variation of μ_d as shown in the inset in figure 4.

In the preceding discussion, it has been concluded that the trapping efficiency μ_d depends strongly on the length of straight dislocation-line segments. To confirm our conclusion, we finally describe a preliminary experiment on how μ_d changes when dislocations bow out under a small applied stress.

The Al specimens (99.9999% purity) used were: polycrystalline specimens (1 × 13 × 50 mm) heated at 108 °C for 1 h after 8% cold rolling; and single-crystal specimens (0.9 × 12 × 70 mm) heated at 72 °C for 2 h after 6% elongation. Positron-lifetime measurements were performed at room temperature in the sequence of three experimental steps: in step 1, no stress is applied to the specimen; in step 2, a stress is applied in the direction of the specimen axis; in step 3, the stress is removed.

Figure 5 shows the positron lifetime τ_2 and the effective positron-trapping rate κ_{eff} in a repetition of the sequence of experimental steps 1–3 for different applied stresses. The salient feature of the results is that κ_{eff} is decreased markedly even by a small applied stress. This clearly indicates that the positron-trapping efficiency μ_d is very sensitive to dislocation configurations, as already described.

A possible configurational change under a small applied stress is the bow-out of relatively long straight dislocation segments via slip, the ends of which are pinned, corresponding to nodes in a network, etc. This situation is probably realized in the single-crystal specimen under the applied stress σ of 0.03 MPa. Such a small stress can yield only small bow-out even if the length of dislocation segment lying on the primary slip plane is as large as 1 μm. In fact, as shown in figure 5, κ_{eff} returns to the initial value in step 1 immediately after the stress is removed, and also τ_2 remains unchanged ($\simeq$210 ps) in any experimental step. Similar results are also found for the polycrystalline specimen when the applied stress σ is 0.09 MPa. The difference in σ above should be attributed to the length of straight dislocation segments developed by heating.

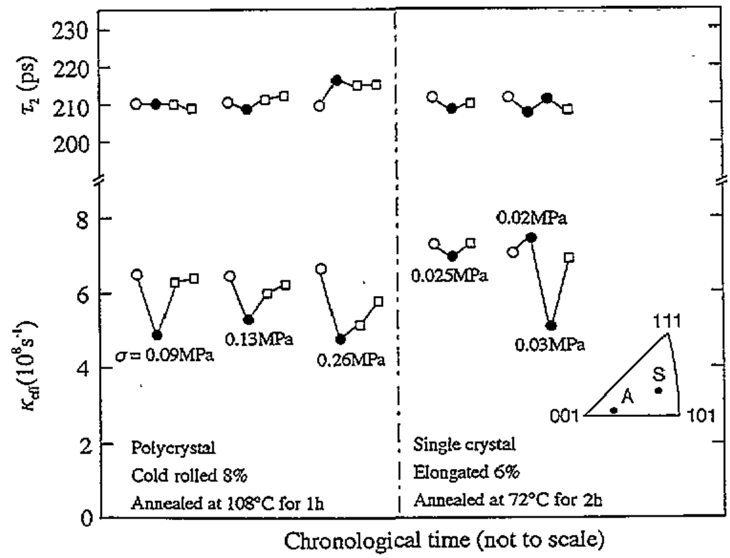

Figure 5. The positron lifetime τ_2 and effective positron-trapping rate κ_{eff} in the sequence of three experimental steps for deformed polycrystalline Al and single-crystal Al specimens (99.9999% purity) after heating: in step 1 (○), no stress is applied to the specimen; in step 2 (●), a stress σ is applied; in step 3 (□), the stress is removed. Both the surface normal (S) and axis orientation (A) of the single-crystal specimen are indicated in the inset.

On increasing applied stress, dislocations and nodes in a network are found to migrate by slip and climb, and finally plastic deformation occurs. This situation is likely in the polycrystalline specimen under an applied stress larger than 0.1 MPa. In fact, the specimen was found to be strained by 0.08% after applying a stress σ of 0.26 MPa. In this case, as expected, κ_{eff} remains less than the initial value even after removing the stress, and τ_2 also becomes larger than the initial lifetime, $\simeq$210 ps.

Consequently, it is clear that even slightly bowed-out or skewed dislocations markedly reduce their positron-trapping efficiency. This confirms our conclusion as already described that the positron-trapping efficiency of dislocations depends strongly on the length of straight-line segments. Such configurational dependence of μ_{d} may suggest that the positron-trapping potential at the dislocation line is so narrow and shallow that the bound state can no longer be realized even in slightly bowed-out configurations.

4. Summary

In this work, positron-lifetime measurements have been made to study how the positron-trapping efficiency of dislocations, μ_d, changes on heating deformed Al (99.9999% purity). It has been shown that μ_d increases with a development of straight dislocation configurations—for example, the growth of three-dimensional dislocation networks during recovery after deformation. Furthermore, it has been shown that even slightly bowed-out or skewed dislocations under a small applied have markedly reduced values of μ_d. These results clearly indicate that μ_d depends strongly on the length of straight dislocation-line segments. The highest value of μ_d obtained reaches about 4.5×10^{-4} m^2 s^{-1}. This value is one order of magnitude larger than the theoretical estimates for sufficiently long straight dislocations [13]. To clarify the configuration dependence of μ_d, further studies, both experimental and theoretical, are required.

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