THE ROLE OF THE AGING TEMPERATURE ON THE SELF HEALING KINETICS IN AN UNDERAGED AA2024 ALUMINIUM ALLOY

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Positron lifetime spectroscopy has been applied to quantify the restoration of free volume defects (vacancies, dislocations and nano-cracks) formed during plastic deformation of a commercial aluminium AA2024 alloy in an underaged (UA) state. Plastic deformation of an underaged aluminium alloy increases the average positron lifetime from an initial value of 182 ps to 209 ps. After deformation the positron lifetime value decreases with time and approaches its initial value at a rate depending on the aging temperature. The results show that the nano-structure of an underaged material after deformation and aging at 65-70 °C has the same positron annihilation (PA) characteristics as the material before deformation. From this we conclude that the solute in the matrix is capable of closing the open volume defects caused by plastic deformation. The healing process seems to be a thermally activated process with an activation energy of 0.91eV, which corresponds to that of Cu and Mg pipe diffusion in Al matrix.

Keywords: Positron annihilation, vacancy, precipitation, solute, aluminium

1 Introduction

Aluminium alloys are a subject of great interest in applications where low weight and high strength are required. They are used in aircraft and spacecraft industry and modern transport industry in general and for this reason Al alloys have been studied extensively. One of the biggest interests has been to characterize the precipitation mechanism of solute atoms. Past and current research on precipitation behaviour has generally focused on obtaining the highest strength or highest combined strength-toughness values [1-3]. In the present work we demonstrate the first attempt to reveal the effect of temperature on the self healing characteristics of the well known age hardenable aluminium alloy AA2024.

In solid metals and alloys self healing will take place only with some difficulties as, in general, the atoms are insufficiently mobile to travel substantial distances to do their repairing action. However, there are two metal systems conceivable which may be more prone to show self healing: supersaturated aluminium alloys, which age harden naturally at room temperature, and high temperature metals operating close to their melting point. In this work we concentrate on a hardenable aluminium alloy.

In this work we use a positron annihilation method in order to study the native defects in Al alloy. A positron is the antiparticle of an electron, and it can get trapped by defects, i.e. by vacancies or other open volume defects. Positrons can also get trapped by negative ions or by solute aggregates with a higher positron affinity than the Al matrix. A measurable positron signal is arising from the annihilation of positron-electron pair where the mass of both particles is converted into two 511 keV-quanta [4].

The purpose of this work is to study with positron annihilation spectroscopy how solute atoms drift to the nano damage formed during plastic deformation and fill the damage sites by cluster formation or precipitation. We present the damage development in AA2024 in an under aged (UA) condition and its restoration by an appropriate thermal treatment.

2 Experimental

For our research we used commercial 1 mm thick AA2024 sheet in the hardened (T3) state. The AA 2024 alloy nominally contains Cu (3.8-4.9 at.%), Mg (1.2-1.8 at.%), Mn (0.3-0.9 at.%), Fe (0.5 at.% max.), Si (0.5 at.%), Zn (0.25 at.%) and other constituents with concentrations of 0.15 at. % at maximum. Out of the starting material we machined tensile test samples with dimensions in accordance to the ASTM B557 standard.

To generate a suitable under-aged condition we performed the following heat treatment: samples were first solutionised in a furnace with circulating N_2 gas (495 $^\circ$C for 30 min) and then quenched into ice water (0 $^\circ$C). The solutionising treatment was followed by a heat treatment at 195 $^\circ$C for 5 min. All samples were stored in LN_2 before straining and in-situ PA tests.

A ^{22}Na positron source was sealed in between two Kapton foils with a thickness of 8 μm and sandwiched between two Al specimens to be strained simultaneously. To prevent slippage of the two samples with respect to each other during straining sandpaper was placed in between the samples before fixing them in the clamps of a spindle driven by Instron tensile testing machine. The samples were strained stepwise to a maximum level of 2% at a strain rate of 1.67×10^{-3} min^{-1}. To avoid precipitation reactions taking place during the PA measurements the samples were cooled to - 15 °C using Peltier elements attached close to the central section of the sample. The same Peltier elements were used to heat the samples to the desired ageing temperature (25, 40 or 65 °C). Samples were kept in the tensile testing machine at all times.

Lifetime detectors were placed as closely as possible to the sample surface to maximize the counting rate. For lifetime measurements we used plastic scintillators and photomultiplier tubes, which were coupled to a fast digitizer (2 GS/s). The triggering of the digitizer was achieved by an external circuit, which was fed with the split signals from the anodes of the start and stop detectors [5]. The time window for the coincidence detection of the start and stop signal in the external triggering box was established by a simple cable delay (~ 5 m) [5]. The activity of the ^{22}Na source was 3 MBq with ~ 150 counts per second for lifetime measurements.

After background reduction and source correction, the spectra were analyzed with the posfit-program [6] using a single Gaussian resolution function with a FWHM of 260 ps. The collected data exceeded 1 million counts in order to obtain a statistically meaningful sample.

3 Results and Discussion

Fig. 1. shows the average positron lifetime as a function of (post-deformation) time for the underaged material both in the undeformed and the deformed state. The positron lifetime in the undeformed UA Al sample (Fig. 1., filled squares) is initially 182 ps and increases during aging at room temperature. The positron is attracted by open volume defects, like vacancies and dislocations, and it can be found in the regions where the positive charge from ion cores is locally reduced. A lifetime of 182 ps in Al alloy is often attributed to positron trapping at vacancies with increased Cu content because it is close to the vacancy lifetime in pure Cu (see e.g. [8]). As the positron lifetime in the undeformed UA sample increases during RT aging this indicates that fewer annihilations occur with high momentum electrons, i.e. the local average electron density at the annihilation site decreases with time. It is believed that artificial aging at 195 °C promoted the solute clustering, mainly of Cu, including vacancies leading to the low initial lifetime value of 182 ps. The following natural aging slowly releases the vacancies from Cu rich areas enabling the secondary precipitation of Mg and Cu [9].

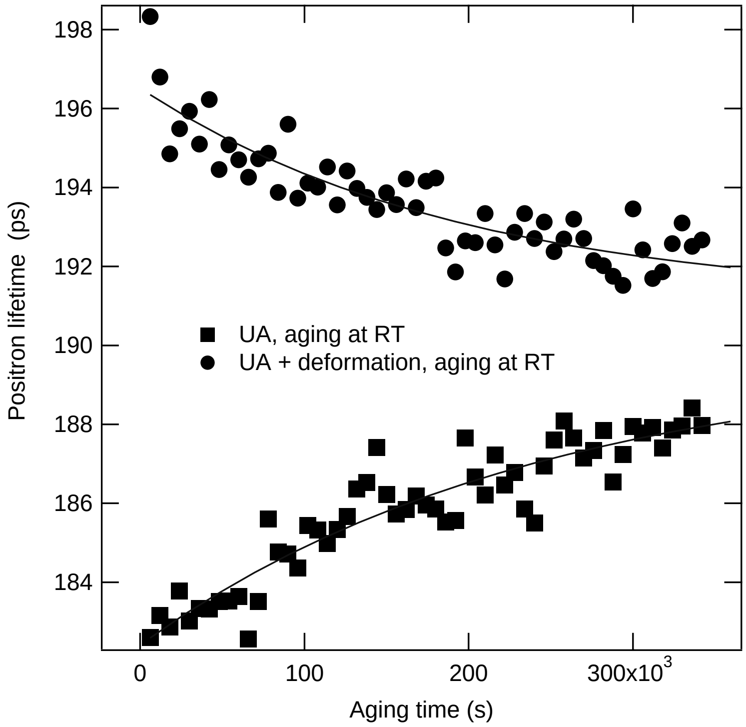

Figure 1: Average positron lifetime as a function of RT aging time. The solid lines are fits calculated on the basic of the positron trapping model. Data is obtained from [7]

By deforming the UA sample the initial lifetime is increased from 182 ps to 210 ps (see Fig 2.), which is a well known consequence of the increased dislocation density. Unlike the undeformed material the average lifetime subsequently decreases as a function of aging time, indicating free volume annihilation during aging at room temperature. The shorter lifetime corresponds to the increased average electron density at the trapping site.

The aging behaviour of the deformed underaged material differs significantly from that of the deformed material in the original T3 state (Figure 2). Clearly the difference between the initial and final aged state in the T3 material is not as pronounced as in the UA material. Compared to the lifetime before deformation ($\tau \approx 192$ ps [10]) this is an indication that the open volume generated with applied mechanical stress is maintained in the T3 material regardless of aging. The solute in T3 material is already aggregated and hence cannot provide any further precipitation (and demise of free volume) in the Al alloy.

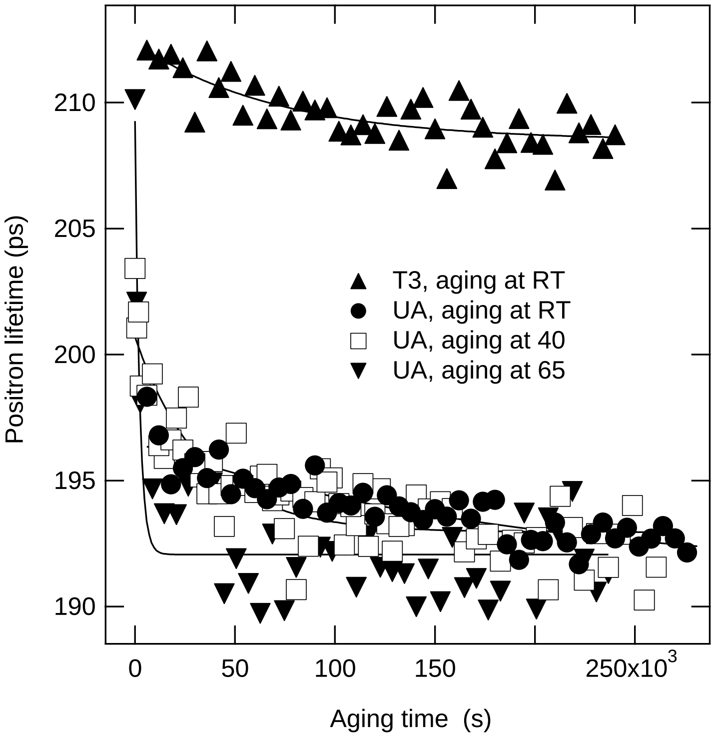

Figure 2.: The average positron lifetime as a function of aging time. The solid lines are the best fit curves based on positron trapping model. The data for room temperature aging is obtained from [7]

In order to study the kinetics of solute precipitation in deformed UA material the average positron lifetime is measured while aging at and above RT (Fig. 3). The figure 3 clearly shows that the rate of decay of the positron lifetime decreases strongly with ageing temperature. The initial value (about 210 ps) and the final value (191 ps) are independent of the ageing temperature. The initial value reflects the state directly after plastic straining while the final value reflects a quasi equilibrium state.

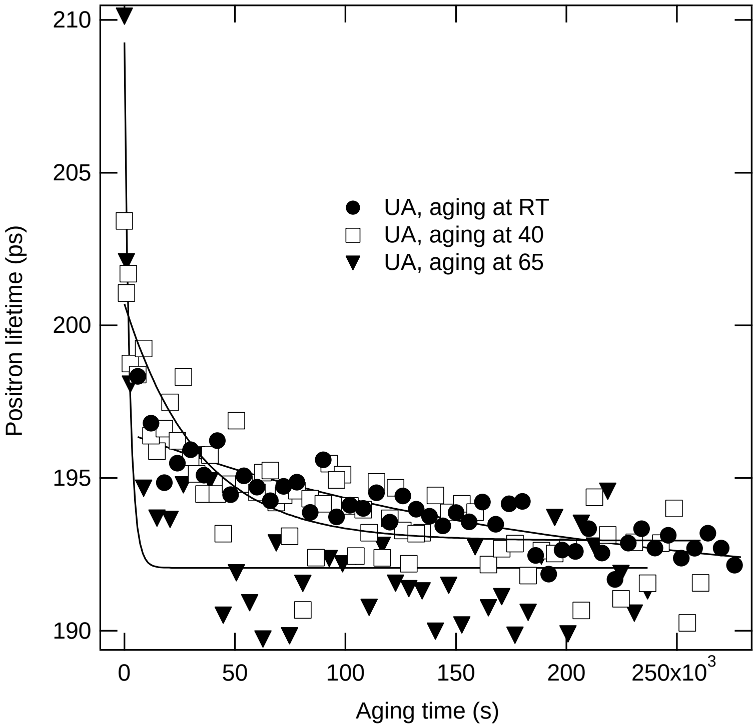

Figure 3: The evolvement of positron lifetime during isothermal annealing. Solid lines are fitted curves to the positron trapping model (see Eq. 1.1 and Table 1). RT data is obtained from [7]

We can now attempt to make a quantitative analysis of the rate of decay by assuming a decay law of the following form

$$\tau_{ave} = \tau_{\infty} + \left(\tau_0 - \tau_{\infty}\right)\exp\left(-t/t_T\right) \qquad (1.1)$$

where τ_0 and τ_{∞} are the initial and final lifetimes, respectively, and t_T is the time constant at given temperature. Results of such a fitting procedure are given in the Table 1 and are also plotted in figure 3 (solid lines).

Table 1: Results of the fit parameters of the initial lifetime τ_0, the final lifetime τ_{∞} and the time constant t_T for the deformed under-aged Al samples

Sample ($^{\circ}$C)	τ_0 (ps)	τ_{∞} (ps)	t_T ($\times 10^3$ s)
UA (RT)	196.5 ± 0.3	191.1 ± 0.2	190 ± 20
UA (40)	200.8 ± 0.8	193.0 ± 0.3	36 ± 5
UA (65)	209 ± 2	192.1 ± 0.3	2.3 ± 0.6
UA (RT,no deform.)	182.4 ± 0.4	189.7 ± 0.1	240 ± 20

The basic way of deriving the activation energy for solute diffusion from the measured positron data is to plot $ln\ t_T$ as a function of $1/k_BT$ (for details see [11] and references therein). The data are adequately described by the following imposed equation for thermally activated processes

$$\ln\left(t_T\right) = Q_A / k_B T + const. \qquad (1.2)$$

where Q_A is an activation energy for solute movement, T temperature and k_B Boltzmann factor. Usually Q_A is a sum of vacancy formation energy E_V^f and solute migration energy $E_M^{Mg/Cu}$, but if the migration is assisted by extrinsic vacancies formed by irradiation or deformation, for example, the activation energy is given by $Q_A = E_M^{Mg/Cu}$. The fit to the plot gives an activation energy of Q_A=0.91(6) eV. The vacancy formation energy E_V^f in Al is 0.61 eV and the activation energy for self diffusion for Al atoms is 1.3 eV [12,13]. Computational studies show that the activation energies for Mg and Cu (bulk) diffusions are 1.23 eV [12] and 1.22 eV [13], respectively. From this context it is clear that the value of 0.91 eV measures neither migration barrier E_M for Al/Cu/Mg atoms nor the total activation energy Q_A for the bulk/self diffusion. However, taking into account that the material is plastically deformed and full of dislocations, it is interesting to compare the experimentally determined activation energy with that of pipe diffusion along the core of a dislocation. For pipe diffusion the activation energy is generally 0.6-0.7 times that of the bulk diffusion [12]. Given the close fit in activation energies for such a mechanism, it is acceptable to conclude that in the underaged material the restoration of the deformation damage takes place via pipe diffusion of Cu or Mg along the core of the dislocation. It has to be pointed out that in the analysis above the positron trapping rate at open volume defects is assumed to be temperature independent. Nothwithstanding this simplification, one can conclude that during aging of deformed underaged material the solute atoms migrate with the help of strain-induced vacancies and dislocations to the site of open volume defects and close them during aging.

4 Conclusions

We have studied underaged commercial Al alloy AA2024 with positron annihilation technique in order to probe deformation induced open volume defects during aging at elevated temperatures. The results indicate that the open volume formed by straining of the under aged material becomes undetectable with positrons after long term aging at RT or above. The dynamic solute precipitation is discussed in terms of self healing, where the solute is autonomously repairing the defects formed by applied mechanical stress. The study shows that the self healing potential of this well known aluminium alloy is an area yet to be explored and optimized.

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