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Study on the Recovery of Quenched Commercial Pure Al and Al-1wt.% Mn Alloys by Positrons

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Isochronal annealing of quenched commercial pure Al (99.5%) and Al-1wt.% Mn was performed between room temperature and 560 °C. The annealing of defects was studied using positron lifetime and Doppler broadening techniques. The behaviors can be divided into three stages: recovery, partial recrystallization, and complete recrystallization. The results suggest that a strong interaction between Mn atoms and vacancies in Al would retard the vacancy recovery from quenching. The Mn atoms enhance the nucleation of vacancy clustering in Al.

Keywords Al (99.5%), Al-1wt.% Mn, annealing, Doppler technique, positrons

1. Introduction

When metals are quenched from a high to a low temperature, most of the imperfections present in thermal equilibrium at the high temperature can be frozen. In close-packed metals the dominant imperfections are vacancies, vacancy clusters, and dislocations arising from collapsed vacancy clusters. Positron annihilation techniques are useful in studying lattice defects and their recovery behavior, and the amorphous structure of metals and alloys.[1,2] Several investigations have been performed using positron annihilation lifetime for studying defects in aluminum and aluminum alloys after quenching.[3-6] Szeles et al.[6] have studied the secondary-defect formation in quenched aluminum; they described the behavior of the annihilation parameter as a balance between the growth and dissolution of vacancy clusters and annihilation of vacancies in three-dimensional defects. At 200 K mobile vacancies either gather and build small clusters or reach various sinks; as the annealing temperature is raised, the effect of sinks becomes predominant. Annealing at higher temperatures leads to the diminution of single vacancies and small clusters, whereas more stable, larger clusters grow further. In the present work, two different types of industrial aluminum are investigated: commercial pure Al (99.5 wt.%), and Al-1wt.% Mn. Iron and silicon are common in both samples with concentrations of 0.35 wt.% and 0.14 wt.%, respectively. This work studies the recovery of lattice defects during the thermal isochronal after quenching.

2. Experimental Details

The samples were annealed at 480 °C and quenched to −196 °C, then isochronal annealed in air for 1 h in the temperature range from room temperature (RT) to 560 °C. The measurements were carried out at RT. Positron lifetime spectra were measured using fast-fast coincidence lifetime spectrometry with a time resolution of 230 picoseconds (ps) for ^{60}Co. The positron lifetime spectra were recorded with integral counts not less than 10^6 counts for each sample. By using the PATFIT program (Riso National Laboratory, Roskilde, Denmark), the lifetime spectra were analyzed into two components. From the data analysis of lifetime experiments, the average lifetime τav of the two main components $\tau_{av} = \tau_1 I_1 + \tau_2 I_2$, as well as the bulk lifetime $1/\tau_b = I_1/\tau_1 + I_2/\tau_2$ and trapping rate $k = I_2/I_1\ (1/\tau_b - 1/\tau_2)$, are deduced using a two state trapping model.[7]

The Doppler broadening annihilation line shape S-parameter was measured using a hyper germanium detector. The measured full-width at half maximum (FWHM) was established to be 1.1 keV at 662 keV of ^{137}Cs. The energy dispersion of the equipment was 40eV per channel. The number of channels included in the annihilation peak area was 320.

The total number of counts in the measured spectrum was ~10^8. The S-parameter was measured as the number of counts lying within an energy interval of 1.3 keV centered at the peak of the annihilation line. The parameter S_{nor} normalized can be determined from the ratio of S/S_{ref}; the S_{ref} was obtained by measuring the line shape distribution using annealed samples of both commercial pure Al and Al-1wt.% Mn alloys. The positron source used in this investigation was ~20μCi of ^{22}Na deposited on Kapton foil (Dupont, Wilmington, DE) and sandwiched between two layers of the sample.

3. Results and Discussion

Figure 1 shows the variation of the average lifetime τ_{av} and the line shape S_{nor} as a function of annealing temperature between RT and 560 °C for commercial pure Al (99.5%) and Al-1wt.% Mn alloys. The lifetime long component τ_2 and its intensity I_2 are shown in Fig. 2 and 3. τ_b is evaluated and compared with the lifetime short component using the two-state trapping model.[7] The results of the present work (Fig. 1) suggest some degree of Mn-vacancy interaction. This is primarily indicated by the increase in S_{nor} and τ_{av} during the recovery stages in Al (Mn) compared with results in commer-

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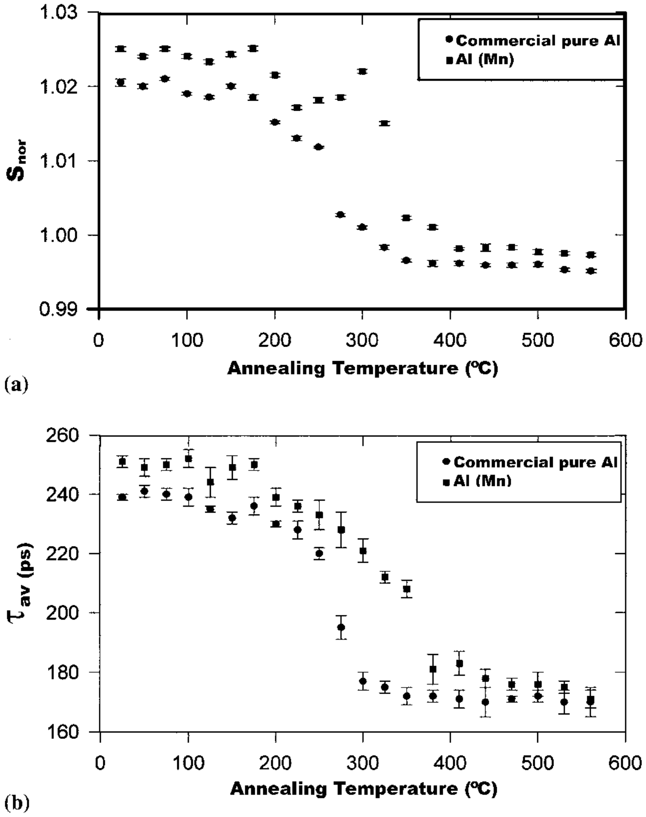

Fig. 1 The variation of **(a)** S_{nor} and **(b)** τ_{av} with annealing temperature in commercial pure Al and Al (Mn)

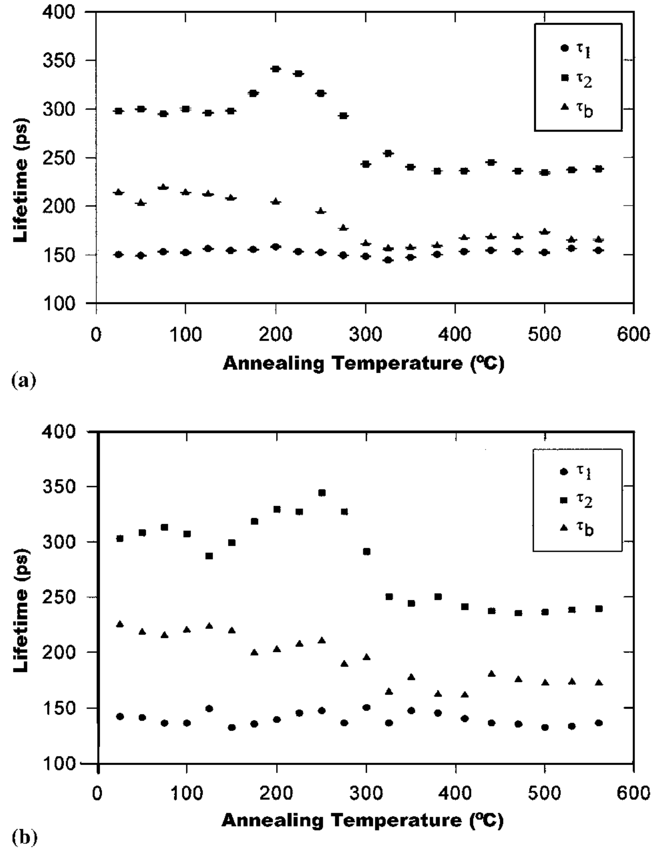

Fig. 2 The variation of lifetime **(a)** in commercial pure Al, and **(b)** in Al (Mn) alloys with the annealing temperature

cial pure Al, which are in good agreement with earlier results.[8,9]

It is apparent that the behaviors of τ_{av} and S_{nor} are similar through all the heat treatment. The behaviors can be divided into three stages: the first stage is taken from RT to 180 °C. In this stage, a little decrease in S_{nor} and τ_{av} is observed in both alloys, which can be related to the recovery process.

The initial value of S_{nor} is 1.02 in commercial pure Al and 1.025 in Al (Mn). However, the values of τ_{av} in this temperature range oscillate around 239 and 249 ps for commercial pure Al and Al (Mn), respectively, which is close to the lifetime characterizing saturation trapping of positrons at monovacancies in pure Al namely, $\tau_v = 245 \pm 10$ ps.[6]

The longer lifetime component τ_2 has a value of 300 ps in both alloys. This value allows us to detect secondary defects: small vacancy clusters, which might lead to lifetimes values, are higher than those characterizing positron annihilation in single vacancies. In this stage, the intensity of the longer component I_2 decreases from 64-50% signifying a possible reduction in the defect concentration in both alloys.

The second stage is taken from 180-300 °C in commercial pure Al and from 180-380 °C in Al (Mn). A sharp decrease is observed in S_{nor}, where S_{nor} is 1.001 in both alloys. On the other hand, τ_{av} decreases continuously to 177 ps in commercial pure Al and to 181 ps in Al (Mn); these values are higher than the lifetime found in perfect lattice ~166 ps.[6] This means that this stage is attributed to partial recrystallization for both alloys. However, τ_2 increases at the beginning of this stage to a maximum of 340 ps at 200 °C in commercial pure Al and delays to 250 °C in Al (Mn); this value of the lifetime corresponds to migration of small clusters and formation of five vacancies.[11] Above 250 °C, τ_2 decreases with annealing temperatures and saturates at a value of ~245 ps, corresponding to the characteristic lifetime for monovacancies; such behavior of τ_2 could be ascribed to the diminution of vacancy clusters, which probably collapse to dislocation loops, where the dislocations give similar saturation annihilation parameter values as single vacancies.[6]

It is apparent that I_2 shows a rapid decrease where it reaches 20% in commercial pure Al and 28% in Al (Mn), suggesting that some vacancies were lost to sinks during migration,[9] leading to the annealing of defects with different concentration in both alloys. Clearly, the second stage is shifted to higher temperatures in Al (Mn) compared with commercial pure Al. Thus, the presence of Mn in Al matrix alters the recrystallization process, which agrees with previous results[8,9,11] and can be attributed to precipitation of stable Al_6Mn particles that are known to take place in Al (Mn) alloys. The process is slow due to the low diffusion coefficient of Mn in Al, but addition of Fe and Si considerably accelerates precipitation. The nucleation and growth of some metastable particles may probably be responsible for the retardation of recrystallization in Al (Mn) alloys.[9,11]

The third stage, recognized by a saturation of S_{nor} and τ_{av}, indicates the complete recrystallization for both alloys. This

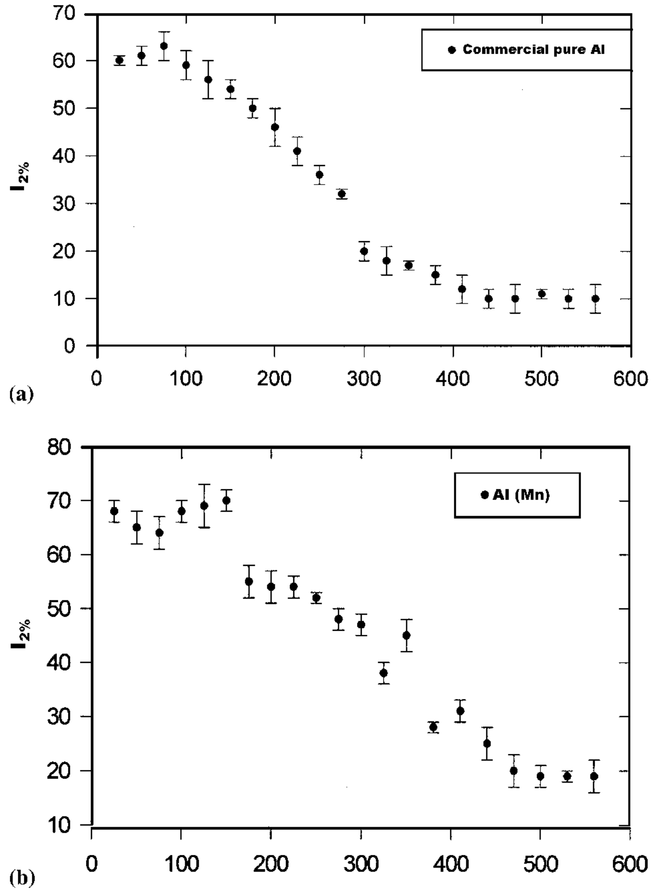

Fig. 3 The variation of I_2 **(a)** in commercial pure Al, and **(b)** in Al (Mn) alloys with the annealing temperature

stage starts at 300 °C and 380 °C in commercial pure Al and Al (Mn), respectively. At complete recrystallization, S_{nor} approaches the bulk value, which is 0.996 in commercial pure Al and 0.998 in Al (Mn). On the other hand, τ_{av} decreases slowly to the value characterizing free positron annihilation in aluminum ~166 ps. This result is in good agreement with earlier positron annihilation studies[6] and is interpreted as the annealing of dislocation loops.

However, τ_2 shows a saturation until the end of the stage at around the lifetime value and characterizes saturation trapping of positron in single vacancies, ~245 ± 10 ps.[6] The intensity of this component I_2 decreases slowly to 10% and 19% at 440 °C in commercial pure Al and Al (Mn), respectively, and remains constant till the end of this stage indicates complete recrystallization.

The shorter lifetime component τ_1 oscillates around 155 and 145 ps for commercial pure Al and Al (Mn), respectively, through all of the stages. These values of τ_1 are shorter than the lifetime found in the perfect lattice, ~166 ps.[5] The values of τ_1 do not satisfy a two-state trapping model[6] in the early stages (Fig. 3) because a strong deviation suggests the presence of more than one type of defect in the samples. However, the deviation decreases in the final annealing stage indicating the annealing of defects, which is faster in commercial pure Al and retarded to higher temperatures in Al (Mn).

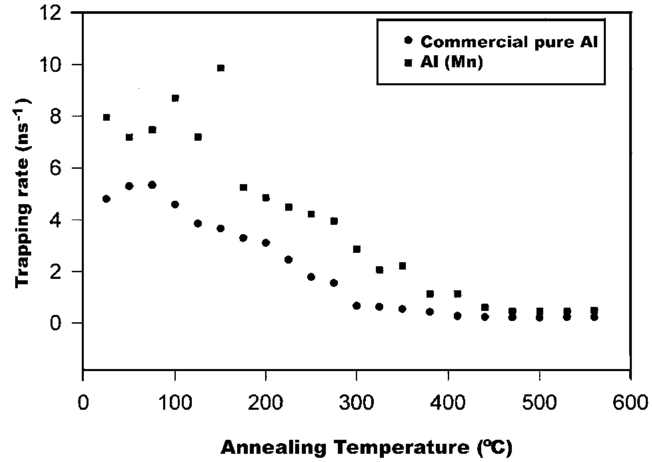

Fig. 4 The variation of trapping rate in commercial pure Al and Al (Mn) with the annealing temperature

The above results are confirmed by drawing the relationship between the trapping rate k and annealing temperature for commercial pure Al and Al (Mn) (Fig. 4). At RT, k is $4.8 \times 10^9 \cdot s^{-1}$ and $7.95 \times 10^9 \cdot s^{-1}$ in commercial pure Al and Al (Mn), respectively. Thus it may be deduced that the Mn atoms enhance the nucleation of vacancy clustering in Al in agreement with previous studies for Al (Mn).[8,9]

However, k decreases rapidly in commercial pure Al to reach $0.66 \times 10^9 \cdot s^{-1}$ at 300 °C, and almost to the same value in Al (Mn) alloy at 380 °C; then k decreases slowly in the two alloys and remains constant at the final annealing stage.

4. Conclusion

The lifetime and Doppler-broadening study of positron annihilation in quenched commercial pure Al and Al-1 wt.% Mn have suggested that:

- The behaviors of τ_{av} and S with the annealing temperature are similar.
- The results indicate the presence of three stages: recovery, partial recrystallization, and complete recrystallization.
- The recrystallization temperature in Al (Mn) is shifted to a higher temperature compared with commercial pure Al. Formation and precipitation of Al_6Mn particles may be the reason for retardation of recrystallization in Al (Mn).
- The variation of k with annealing temperature confirms the results obtained of Snor, τ_{av}, τ_2, and I_2.

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