Variation of defects in deformed $L1_2$ $Al_{67}Mn_8Ti_{25}$ ordered intermetallic alloy during annealing

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The isochronal recovery of deformation-induced defects in $L1_2$ $Al_{67}Mn_8Ti_{25}$ ordered alloy was investigated by positron lifetime measurement and *in situ* electron transmission microscopy. Three recovery stages of the positron lifetime were observed for the deformed samples during isochronal anneals. Vacancy migration was proposed to explain the first stage from room temperature to 200°C; The second stage, between 200 and 400°C, was assigned to be the dissociation of superdislocations with superlattice intrinsic stacking fault (SISF) or antiphase boundary (APB); The third stage, occurring up to 800°C, was attributed to the only dissociation scheme of dislocation with antiphase boundary (APB) accompanied by a relaxation of antiphase domain.

1. Introduction

Among the intermetallic compounds Ti_3Al, TiAl and $TiAl_3$ in Ti-Al system, Al_3Ti has the lowest density and best oxidation resistance. However Al_3Ti is very brittle at room temperature [1]. To improve its ductility, a ternary element (e.g. Fe, Cr or Mn) has been added to convert its tetragonal DO_{22} structure to the cubic $L1_2$ structure [2]. The Mn- or Cr-containing $L1_2$ trialuminde has been found to show not only compressive ductility but also a little of tensile ductility [3]. To explore the deformation mechanism, transmission electron microscopy (TEM) studies on the dislocation characteristic and dissociation scheme in $L1_2$ Al_3Ti alloys had been reported by a number of researchers, though the results are not consistent [4–10].

Positron lifetime spectroscopy has been successfully used to study defects in intermetallic compounds [11–14]. Positrons are sensitively trapped by defects such as vacancies and dislocations. Shirai *et al.* had studied point defects in electron-irradiated $L1_2$ $Al_{67}Mn_8Ti_{25}$ by using positron annihilation lifetime spectroscopy [15]. They found that all constitutional point defects in electron-irradiated $L1_2$ $Al_{67}Mn_8Ti_{25}$ recovered to the fully annealed state below 277°C. Based on the finding that positron lifetime increases almost linearly with increasing magnitude of Burgers vector of dislocation in metals, it has been demonstrated that positron lifetimes can differentiate dislocations [16, 17].

In the present work, the recovery of defects in the deformed $L1_2$ Al_3Ti alloy had been studied by means of position lifetime spectrum and *in situ* TEM observation during annealing.

2. Experimental

The intermetallic alloy with nominal composition $Al_{67}Mn_8Ti_{25}$ (at.%) was prepared by arc melting in argon atmosphere on a water cooled copper hearth. The button ingots were homogenized at 1100°C for 60 hours and a uniform $L1_2$ single phase was attained. In order to introduce defects the samples were deformed to 2% and 6% strain in compression at room temperature at an applied strain rate of 1×10^{-3}/s. Then the samples were isochronal annealed for 20 min. successively at every 50°C interval from room temperature to 800°C. In order to have little relevance to positron detraped by thermal vacancies, the specimens were cooled down in the furnace. After each annealing treatment, $15 \times 15 \times 1$ mm^3 specimen was prepared. Positron lifetime spectra were measured at room temperature using a positron lifetime spectrometer with a time resolution of 240 ps full width at half maximum (FWHM). A ^{22}Na source deposited on an aluminum foil was sandwiched between two identical $Al_{67}Mn_8Ti_{25}$ pieces. Measured lifetime spectra were analyzed by the computer programs Resolution [18] and Positronfit Extended [19]. The mean positron lifetime τ_m is defined as:

$$\tau_m = \Sigma I_i \tau_i \qquad \text{with } \Sigma I_i = 1$$

where τ_i is the ith component and I_i its intensity. Three lifetime model was used when i took 1, 2 and 3. A shorter lifetime τ_1 related to the annihilation of free positron in the lattice (matrix), a longer lifetime τ_2 resulting from the annihilation of positrons trapped at lattice defects and τ_3 related to the annihilation of positrons trapped at positron source. The intensity I_2 of this longer component is a measure of the relative number of positrons which are trapped and annihilated at defects, and hence I_2 is related to the concentration of the defects. Since the τ_3 was less than 1%, the $\tau_3 I_3$ was fixed and did not be considered in our analysis.

TEM specimens were cut from the compression deformed samples and mechanically ground, and then accomplished by ion polishing. *In situ* TEM observations were carried out using a single-tilt heating stage for temperatures up to 700°C in a JEOL 200CX microscope.

3. Results

3.1. Positron lifetime

The positron lifetime of $Al_{67}Mn_8Ti_{25}$ before annealing was measured in the three different deformation conditions. The mean positron lifetime τ_m was 177 ps in the non-deformed sample and a value of 190 ps was obtained after 2% compressive deformation. An increase in τ_m, leading to 210 ps, was shown in 6% deformed sample (Table I).

Figs 1 and 2 show the positron lifetime of two deformed samples during subsequent isochronal annealing. Ignoring the different compressive strains, three major recovery stages are revealed in both samples. Firstly, τ_m decreased slightly during annealing in the low temperature region about 200°C (stage I); then τ_m began to decrease as the temperature increased and reached 165 ps for 2% deformed sample or 170 ps for 6% deformed sample around 400°C (stage II), and then both kept almost constant value about 170 ps up to 800°C (stage III).

The results of analysis for two samples are shown in Figs 1 and 2, respectively. It can be seen that shorter lifetimes τ_1 were almost constant during whole annealing experiments. In stage I, the longer lifetime τ_2 decreased and intensity I_2 did not change; In stage II, both τ_2 and I_2 decreased, and in stage III, both τ_2 and I_2 kept constant up to 800°C.

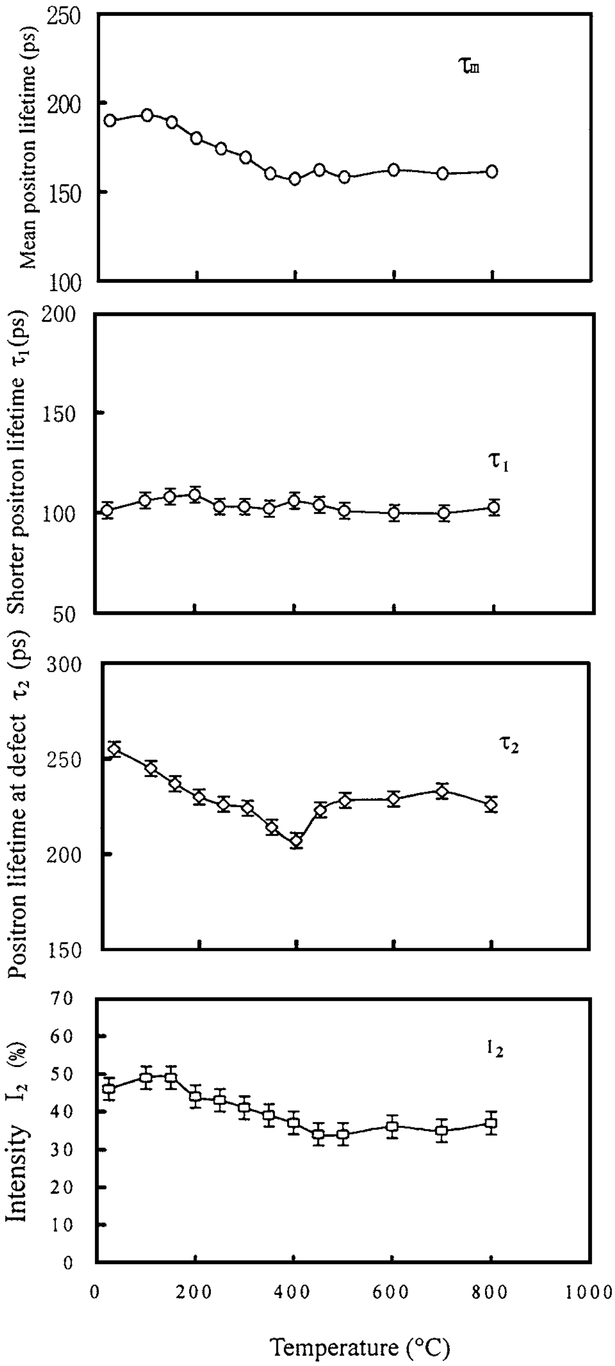

Figure 1 Changes in mean positron lifetime (τ_m), positron lifetime at defect (τ_2), the relative intensity of the defect component (I_2) and shorter positron lifetime (τ_1) on isochronal annealing of $Al_{67}Mn_8Ti_{25}$ after 2% compression.

3.2. TEM observation

TEM observations were performed on deformed specimens prior to annealing. TEM micrographs of the deformed structure are shown in Fig. 3, the single microstructure was identified as ordered $L1_2$ structure. Long and straight dislocations were found in 2% deformation sample (Fig. 3a) and higher density of shorter dislocations were induced in the sample of 6% deformation (Fig. 3b).

In situ weak beam electron microscopy was used to inspect the dissociation of superdislocations in 2% deformed sample during annealing. Before annealing only a few dislocations were dissociated. For example, in Fig. 4a, dissociation of dislocations 1, 2 and 4 could not be detected with any foil orientation. But dislocation 3 was found to be dissociated. It has not so far been possible to unambiguously identify the nature of the partial dislocations (i.e., whether a superlattice intrinsic stacking fault (SISF) separated superdislocation

TABLE I Changes in positron lifetime and intensity of $Al_{67}Mn_8Ti_{25}$ after compression at room temperature

Deformation state	Mean positron lifetime τ_m (ps)	Short positron lifetime τ_1 (ps)	Positron lifetime at defect τ_2 (ps)	Intensity I_2 (%)
Not	177	109 ± 4	243 ± 4	38 ± 3
2%	190	101	255	46
6%	210	109	250	56

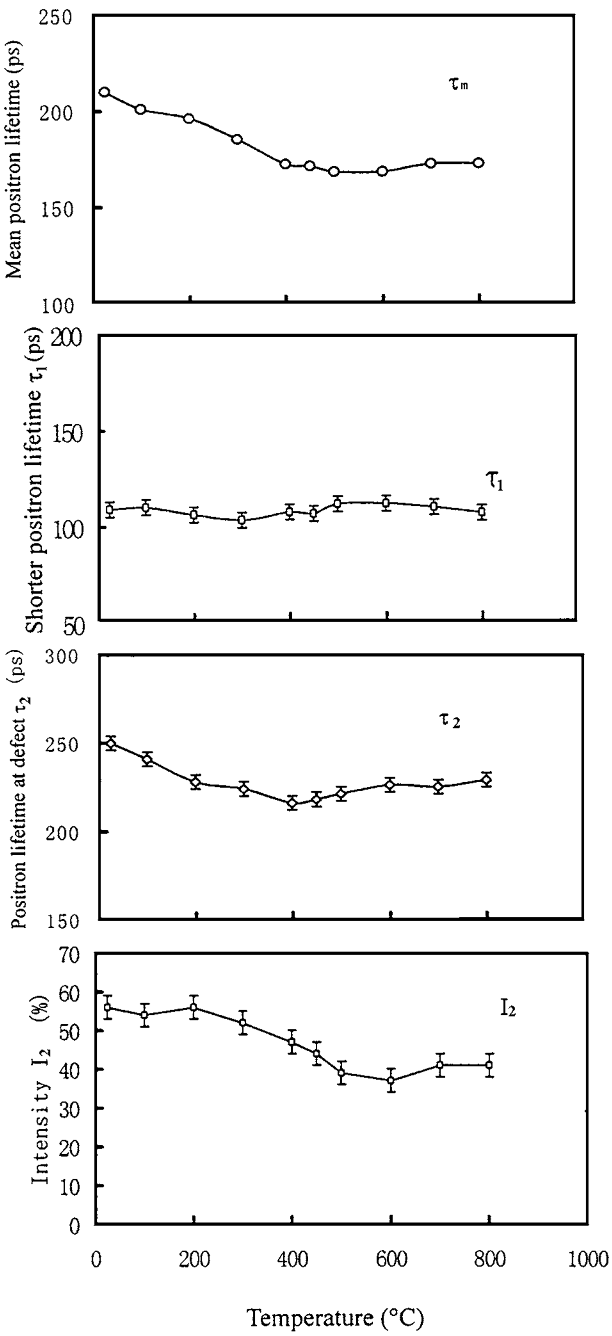

Figure 2 Changes in mean positron lifetime (τ_m), positron lifetime at defect (τ_2), the relative intensity of the defect component (I_2) and shorter positron lifetime (τ_1) on isochronal annealing of $Al_{67}Mn_8Ti_{25}$ after 6% compression.

with two $a/3\langle 211\rangle$ partial dislocations or an antiphase boundary (APB) separated with two $a/2\langle 110\rangle$ partial dislocations) in view of the very small separation of the two partials and the tendency to overlapping image contrast.

On annealing at elevated temperature, a visible change in dislocation separation occurs. Fig. 4 illustrates some of the changes observed by *in situ* TEM. After annealing for a few minutes at 500°C, the separation of the dissociated dislocations increases to 10–12 nm. It should also be noted that the increase took place very rapidly, essentially within the first minute as the thin foil sample heated to the annealing temperature, and no further change took place thereafter. It is also of interest to note that some of the previously undissociated dislocations, began to dissociate at 500°C, but dislocation 1 kept undissociated at this annealing temperature (Fig. 4b). When annealing at 600°C, the dislocation spacings further increased and even the undissociated dislocations separated as superdislocation pairs, see Fig. 4c. On annealing at 700°C the dislocation spacings increased greatly (Fig. 4d). Analysis of dislocations in the 700°C sample after cooled down to the room temperature showed that they all lie on {111} planes and dissociated into two $a/2\langle 110\rangle$ partial dislocations bounding with an antiphase boundary. The separation of partial dislocations was measured to be 30–40 nm. Fig. 5 summarizes the separations of super-partials in 2% deformed sample after heated 5 min at different temperatures, the increasing of separation distance with increasing annealing temperature is quite obvious.

4. Discussion

The mean positron lifetime of $L1_2$ $Al_{67}Mn_8Ti_{25}$ before deformation, $\tau_m = 177$ ps, should correspond to the positron annihilation in the residual vacancies. This value is close to the positron lifetime measured by Shirai *et al.* in electron irradiated $L1_2$ $Al_{67}Mn_8Ti_{25}$ [15]. The increment of positron lifetime from 177 ps to 190 ps after 2% deformation and to 210 ps after 6% deformation suggests that the majority of positrons annihilated after being trapped by deformation-induced defects. The results of TEM observation on the deformed samples showed clearly that the dislocations have been greatly generated. Therefore the increase in positron lifetime should be related to the trapping of positron by the deformation-induced superdislocations.

The recovery process of deformed $Al_{67}Mn_8Ti_{25}$ during isochronal annealing displays three stages according to the measured positron lifetime. The first stage was characterized by a decrease of τ_2. Shirai *et al.* [15] suggested that the species of point detect migrating in this stage must be vacancies rather than interstitials, because only vacancy migration can change the positron lifetime at defects (τ_2). Interstitial migration can only cause a decrease in the relative intensity of the vacancy component (I_2) as a result of annihilation with vacancies. Thus, the results shown in Figs 1 and 2 demonstrate that in the first stage (i.e., from room temperature to around 200°C), there happened the migration of vacancies, it is worthy to notice that the relative intensity of the defect component does not decrease despite the vacancy migration (Figs 1 and 2). This fact indicates that vacancies do not annihilate at permanent sinks, such as grain boundaries and surfaces, but annihilate at dislocations that were induced by deformation.

Stage II (200–400°C) displays a great drop of τ_m, τ_2 and I_2. Since the vacancies in $Al_{67}Mn_8Ti_{25}$ exist only up to 277°C [15], the mainly active traps present in the samples annealed at the second stage are dislocations. The tendency that τ_m, τ_2 and I_2 decreased as the temperature increased indicates that dislocations acted as weaker traps at high temperature than those at low temperature. In fact such a temperature dependence of τ_2 suggests that a thermal detrapping of positrons occurs around 400°C. At the third stage, positron lifetime

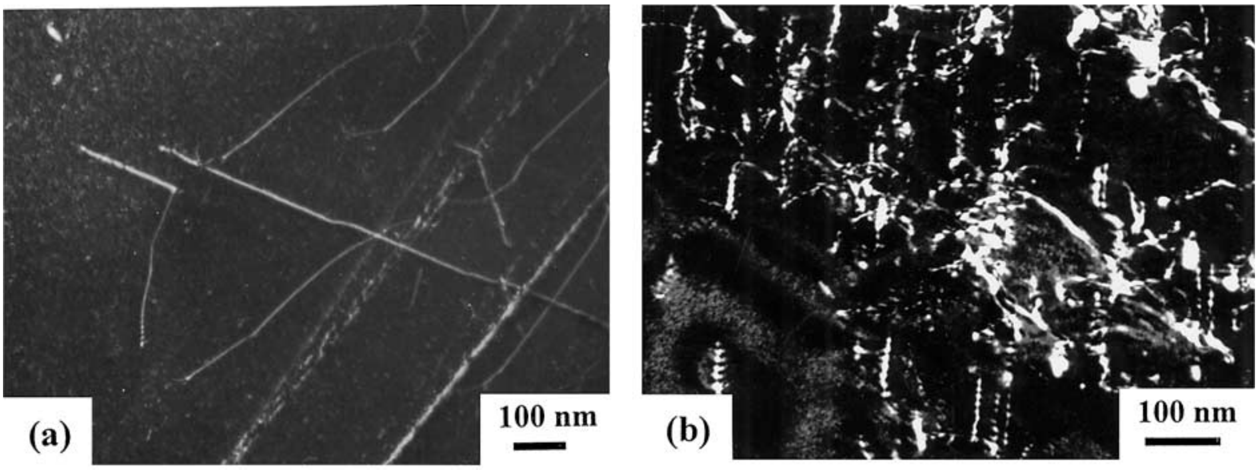

Figure 3 Dislocation structure in $Al_{67}Mn_8Ti_{25}$ compression deformed at room temperature. (a) deformed 2% (b) deformed 6%.

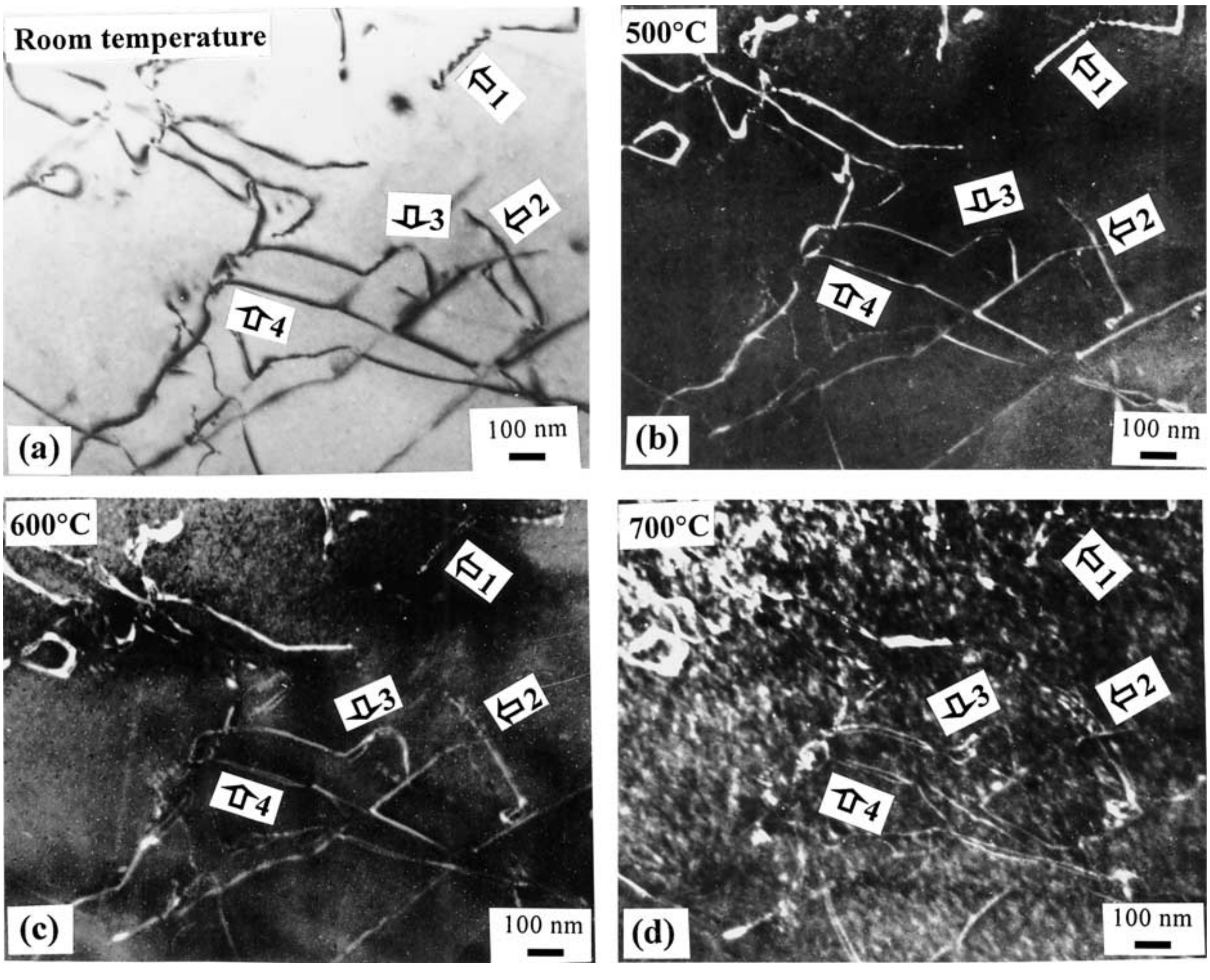

Figure 4 TEM micrographs of dislocations in $Al_{67}Mn_8Ti_{25}$ deformed by 2% at room temperature (a) and after *in situ* annealing at 500°C (b), at 600°C (c) and at 700°C (d), **g** vector = $\bar{1}\bar{1}1$.

measurements showed a constant value of τ_m, τ_2 and I_2 during annealing. The difference between the temperature dependence of the positron lifetime for the second stage and the third stage (Figs 1 and 2) indicates that there were different types of dislocations to act as positron traps.

TEM results of some investigators on dislocation structures in $L1_2$ $Al_{67}Mn_8Ti_{25}$ alloy showed that the $\langle 110\rangle$ superdislocation dissociated into $1/2\langle 110\rangle$ superpartials on $\{111\}$ plane bounding an antiphase boundary (APB) after deformation at room temperature, 400°C or 700°C [4–7]. While others found that there is a change of dissociation mode with the change of temperature, i.e., the dissociated dislocations of the alloy deformed at room temperature or 400°C are of $1/3\langle 112\rangle$-type bounding with the superlattice intrinsic stacking

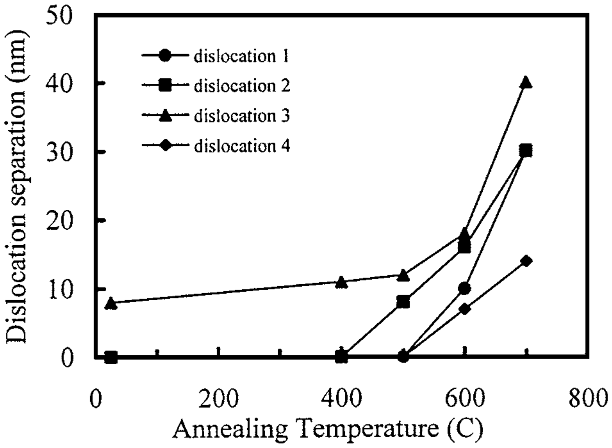

Figure 5 Separation of partial dislocations in the as deformed state and after annealing at each temperature.

fault (SISF), but in the 600°C deformed sample APB-bounding dislocations were observed [8–10]. The arguments arose from the fact that the TEM identification of superpartials may be confused by some side effects such as anisotropic elasticity of the $L1_2$ Al_3Ti alloy, the interaction of strain fields of closely spaced partials and the influence of residual contrast from the edge components, asymmetrical image intensities could be observed in both APB-coupled pairs and SISF pairs. Therefore some researchers suggested that both types of dissociated dislocation, SISF and APB, might simultaneously exist at low temperature. To relate the change of positron lifetime with the dislocation character it is believed that at the second annealing stage, some dislocations dissociated into $a/3\langle 112\rangle$ partials where the positron showed a thermal detrapping [20], while some others dissociated into $a/2\langle 110\rangle$ partials. At the third stage, all the dislocations were APB dissociated. And the *in situ* TEM showed that the dynamic relaxation of dislocations took place. Since the increasing of the space distance between two superpartials was so rapid which can hardly be controlled by long range diffusional processes, it is suggested that this change was dominated by local change of chemistry or order at the APB, where the positron showed a non-thermal detrapping [20].

5. Conclusion

The recovery of the deformation-induced defects in the $Al_{67}Mn_8Ti_{25}$ intermetallic alloy was investigated by positron lifetime spectroscopy and *in situ* transmission electron microscopy. The positron lifetime increased from 177 ps in no deformation sample to 190 ps in 2% deformation sample then to 210 ps in 6% deformation sample. This positron lifetime increase is attributed to the trapping of positrons by deformation-induced dislocations.

During isochronal annealing, three recovery stages of change in mean lifetime τ_m were observed by positron lifetime measurements. Vacancy migration was proposed to explain the first stage from room temperature to 200°C; the second stage, between 200 and 400°C, was assigned to a variation of the type of dislocations resulting from the dissociation of superdislocations with superlattice intrinsic stacking fault (SISF) or antiphase boundary (APB); the third stage, up to 800 °C, was attributed to the APB-type dissociation scheme of dislocations accompanying with a relaxation of antiphase domain.

Acknowledgements

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