MICROSTRUCTURAL EVOLUTION OF 7012 ALLOY DURING THE EARLY STAGES OF ARTIFICIAL AGEING

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(Received 17 February 1999; accepted 2 August 1999)

Abstract—a study of the microstructural evolution of a commercial 7012 (Al–Zn–Mg–Cu) age-hardenable alloy following artificial ageing by high resolution and conventional transmission electron microscopy and positron annihilation lifetime spectroscopy is presented. At the early stages of decomposition, the microstructure included precipitation of either pre-precipitate solute clusters or Guinier–Preston zones and semi-coherent \( \eta' \) precipitates, with typical sizes between 1 and 10 nm. Quantitative information on the size, number density and morphology of the particles present in the microstructure was obtained. The results were correlated with those obtained using positron annihilation lifetime spectroscopy.

1 INTRODUCTION

The Al–Zn–Mg-based alloys have important technological applications due to their high response to age-hardening, low density and high strength. The precipitation in this ternary alloy system has been the subject of many investigations (e.g. Refs [1–4]). The precipitation sequence usually proposed for alloys with Zn and Mg contents similar to 7012 alloy is \[ a \text{SSS} \rightarrow \text{spherical GP zones} \rightarrow \eta' \text{ precipitates} \rightarrow \eta \text{ precipitates}. \]

The supersaturated solid solution is obtained by a solution treatment followed by quenching to a lower temperature. The decomposition process is highly dependent on the excess vacancy concentration and is thereby sensitive to the quenching condition [6]. The decomposition commences with the formation of GP zones. Two types of GP zones have been proposed to form: solute rich GPI zones [3] which dominate in Al–Zn alloys and Al–Zn–Mg alloys with low Mg content; and solute/vacancy-rich GPII zones [3, 7]. The \( \eta' \) transition phase is semi-coherent, which is generally considered to be hexagonal with lattice parameters \( a_\eta \approx 5.15 \text{ Å} \) and \( c_\eta \approx 8.6 \text{ Å} \) [8]. Although this phase is incoherent with the matrix lattice, it can exhibit numerous orientations [2, 8, 9] and may nucleate directly from the solid solution [1].

Recent studies leading to the direct determination of composition and improved understanding of the microstructure of GP zones and precipitates in Al-based alloys by atom probe field ion microscopy (APFIM) and high resolution transmission electron microscopy (HREM) are described in Refs [10–13]. Good examples of state-of-the-art HREM studies of GP zones in other Al-based systems are given in Refs [14, 15].

The continuing progress with advanced imaging techniques, however, does not suppress the interest for other well-established experimental methods: small angle X-ray scattering (SAXS), small angle neutron scattering (SANS), differential scanning calorimetry (DSC) and mechanical property measurements. Examples of such studies on Al–Zn and Al–Zn–Mg alloys are detailed in Refs [16–18].

Positron annihilation spectroscopy (PAS) has been satisfactorily used in the last two decades to study decomposition phenomena in age-hardenable alloys and this approach has recently been reviewed [19]. Positron annihilation lifetime spectroscopy (PALS) technique has been used to study the precipitation kinetics in the Al–Zn–Mg system both in experimental alloys [7, 20, 21] and in commercial alloys [21–26].
Transmission electron microscopy (TEM) and PAS, especially PALS, show complementary aspects [26]. Whilst conventional TEM and HREM provide direct information on the microstructural changes induced by thermal treatments in the alloy, PALS allows us to characterize the physical processes responsible for these changes. Recently, nucleation and growth of GP zones in Al–Zn–Mg-based commercial alloys has been studied using PALS, and the typical activation energies for solute migration have been determined [22, 25]. In addition, during the first minutes of artificial ageing, a fall in the positron lifetime was correlated with a decrease of the material hardness in 7005 and 7012 alloys [22]. Although the softening effect was already known in these alloys [3, 27], the positron lifetime results have linked this softening with the dissolution of small GPII zones. From the joint use of PALS and Vickers microhardness techniques it was possible to investigate the time evolution of formation, dissolution and recovery of GP zones.

In this work, the microstructural evolution of 7012 alloy during the early stages of artificial ageing was examined by means of TEM and HREM. Dimensional and morphological changes of pre-precipitate solute clusters or GP zones and \( \eta' \) precipitates were obtained. The results are discussed critically in comparison with those obtained previously with PALS [22, 25].

## 2. EXPERIMENTAL PROCEDURES

The 7012 alloy commercially known as Zergal 4 has composition Al–6.0Zn–2.0 Mg–1.0Cu (wt%) [Al–2.58Zn–2.31 Mg–0.44Cu (at.%)], and contains minority elements 0.12Zr–0.10Mn–0.06Ti–<0.25Fe–0.15Si–<0.04Cr (wt%) [28].

For the positron lifetime measurements, disc-shaped samples of 1.5 mm thickness and 10 mm diameter were used. The surfaces were polished metallographically with 3 \( \mu \)m diamond paste. Specimens for TEM with 3 mm diameter were cut from the heat treated 10 mm diameter discs and the thickness reduced to 0.2 mm. They were thinned by double-jet electropolishing with a 30% HNO\(_3\) solution in methanol at –35°C and 10 V.

The samples were thermally treated by: (i) solution treating for 2 h at 475°C in an electric air circulating furnace; (ii) quenching to water at 20°C; (iii) pre-ageing at room temperature (RT) \( \approx 20°C \); (iv) isothermal ageing at 150°C for various times in a glycerin bath; (v) quenching to alcohol at 20°C; and in some specific cases, (vi) re-ageing at RT. Assuming that the GP zone metastable phase boundary for 7012 alloys is similar to that of Al–Zn–Mg alloys, in spite of the presence of secondary alloying elements, particularly Cu, the ageing temperature used in the present work (150°C) is above the GP zone metastable phase boundary (see Ref. [3]). A similar assumption was used in a recent study on Al–Zn–Mg–(Ag, Cu) [29].

The lifetime spectrometer was a fast–fast timing coincidence system with a time resolution (FWHM) of 255 ps. The total number of counts in each spectrum was about \( 10^6 \), accumulated in approximately 14,000 s. The lifetime spectra were analysed using the POSITRONFIT program [30]. A 20 \( \mu \)Ci source of \( ^{22}\text{NaCl} \) deposited on a thin Kapton foil (1.1 mg/cm\(^2\)) was sandwiched between two identical alloy specimens so that the source contributes to the spectra with only one component (\( t_S = 382 \) ps, \( I_S = 10.6\% \)). Further experimental details are given elsewhere [25].

The specimens were observed with a Philips CM200UT transmission electron microscope, operating at 200 kV. Bright-field and HREM images were obtained along the (011) zone axis. Due to the relatively limited tilt allowed by the UT objective lens, grains with the appropriate orientation had to be carefully localized.

Approximately 240 GP zones were manually measured from bright-field micrographs after enlargement by means of image processing methods so as to plot frequency histograms of the zone radii. In order to evaluate the precipitate number density, the foil thickness was estimated using two-beam images from the (111) and (002) reflections. The images showed thickness fringes, which corresponded to extinction distances of \( \bar{x}_{111} = 71.4 \) nm and \( \bar{x}_{002} = 86.9 \) nm. The same region was then imaged in bright field along the (011) zone axis at the same magnification, showing thickness fringes with an unknown effective extinction distance. By comparison with the two-beam images, the effective extinction distance was estimated with an error of about 25%. The thickness fringes were then used to estimate the specimen thickness. The overlap effect was considered for calculation of particle densities, using the corrections described by Hilliard [31]. It was found that neglecting the overlap correction the particle density is underestimated by less than 15%.

## 3. RESULTS

Figure 1 provides a summary of the positron lifetime \( \tau \) behaviour in the 7012 alloy for the different ageing thermal treatments (see Ref. [22]). For comparison, the microhardness evolution is also shown as an inset in the top left-hand corner of this figure. To present the lifetime evolution during artificial isothermal ageing at 150°C, a time scale of \( \tau^{1/3} \) was chosen. This represents the structural evolution that occurs during ageing in a coalescence regime [23]. Furthermore, the lifetime evolution during re-ageing at RT for samples heat treated until reaching the minimum value of \( \tau \) in the artificial ageing is included. The left-hand curve of Fig. 1 shows that the positron lifetime varies very little during pre-ageing at RT. In this situation, the microhardness
has increased to a plateau as recently described elsewhere [25]. The artificial ageing curve shows that the initial effect of heat treatment is a rapid decrease of the positron lifetime and microhardness. A similar softening was already observed in other age-hardenable Al-based alloys and was assigned to a reversion process [3, 27]. The PALS results show evidence of reduced trapping in GP zones. It was noted that this phenomenon is not permanent and can be detected only if PALS measurements are performed shortly after the interruption of the corresponding heat treatment. When the data are recorded several weeks later, \( t \) recovers to the initial value (labelled R, Fig. 1). This was also noted in previous work on alloy 7012 [25]. For longer ageing times, the decrease of both parameters is followed by an increase which eventually brings both \( t \) and \( H_V \) to a new maximum. In previous works [22, 25], these increases were mainly attributed to the irreversible transformation of GP zones to \( Z' \).

In Fig. 1, the points labelled 1–5 represent those states of the microstructure investigated using TEM techniques.

Figure 2(a) shows a (011) BF image of a sample after 5 days pre-ageing at RT. Small dark spherical particles with a mean diameter of 1 nm were observed. This contrast is assumed to correspond either to solute clusters or to GP zones and arises from the different structure factor of these regions compared to that of the matrix.

In a recent study on Al–Cu–Mg–(Ag) alloys it was proposed that a distinction between pre-precipitate solute clusters and classical coherent GP zones could be made on the basis of size, shape, composition, degree of order, orientation and structure [14]. At the present stage, after pre-ageing, it is not possible to distinguish between solute clusters or GP zones. This point will be reconsidered in Section 4.

The same microstructure as described above was also observed after 5.5 months pre-ageing. The measured values of the mean particle diameter after 5 days and 5.5 months of natural ageing at RT are given in Table 1. The mean diameter did not vary, indicating that the size of the observed particles stabilizes at about 1 nm. In Fig. 2(b), a HREM image of a sample pre-aged for 5.5 months at RT is shown in which spherical regions with diameters <2 nm can be seen, which are assumed to correspond to the particles observed in Fig. 2(a). No distortion of the matrix lattice planes is observed across these zones, indicating a high degree of coherence. The mean size estimated from HREM micrographs is larger than that estimated from BF images, which is probably due to the low contrast of the zones, which makes the smaller particles difficult to observe.

In Figs 2(c)–(e) the microstructure corresponding to 8 min (2 min\(^{1/3}\)) of isothermal artificial ageing at 150°C is shown (labelled 3, Fig. 1). At this ageing time the positron lifetime reaches a minimum value. The (011) BF image in Fig. 2(c) shows particles with three distinct types of contrast which we have classified as follows:

1. Plate-shaped particles (referred to as type A, hereafter) typically 1 nm \( \times \) 5 nm, aligned parallel to the (111) and (111) planes in the [011] zone axis, forming an angle of \( \sim 71° \) between them. These particles were considered as edge-on \( \eta' \) platelets, with (111) and (111) habit planes.
2. Ellipsoidal particles (referred to as type B, hereafter) with sizes somewhat smaller than the type A particles.

3. A fine distribution of spherical particles with similar contrast to that observed in pre-aged specimens [Fig. 2(a)] which was attributed to solute

Fig. 2. (a) Bright-field micrograph for a pre-aged sample for 5 days at room temperature after solubilization and quenching (labelled 1, Fig. 1). (b) HREM micrograph for a pre-aged sample for 5.5 months at room temperature. (c) Bright-field micrograph of an artificially aged sample for 8 min at 150°C (labelled 3, Fig. 1). (d), and (e) HREM micrographs with the same thermal treatment as (c). All micrographs were obtained along the (011) zone axis.
clusters or GP zones (a detailed discussion about these particles is given below).

Figures 2(d) and (e) show HREM images of specimens aged artificially for 8 min. Edge-on \( \eta' \) particles \( \sim 7 \) nm long (type A), rounded particles (type B) and solute clusters or spherical GP zones can be distinguished. Distortions of lattice planes that cross the type A and B particles were observed, indicating a small mismatch of the lattice parameters. The interplanar distance of the close packed planes is slightly larger in the \( \eta' \) particles. The \( \eta' \) plates have a thickness of about seven close packed planes.

Figure 3(a) shows a typical BF image of a specimen aged 1 min at 150\( ^\circ \)C, which corresponds to the maximum softening of the alloy (labelled 2, Fig. 1). The same types of particles as those observed in samples aged 8 min were found, although the density of the type A and B particles was much lower and the size somewhat smaller. The mean diameter of the solute clusters or GP zones has a value intermediate between that observed in the naturally aged samples and those aged artificially for 8 min at 150\( ^\circ \)C. In Fig. 3(b), a HREM micrograph of a specimen aged 1 min at 150\( ^\circ \)C is shown. A small edge-on \( \eta' \) particle is arrowed, illustrating that the formation of \( \eta' \) precipitates begins at a very early stage.

In Table 1, data of the size, density and volume fraction of the observed particles are summarized for naturally aged samples and samples artificially aged for times up to 8 min at 150\( ^\circ \)C. The mean diameter of solute clusters or GP zones is found to decrease during artificial ageing, while the density does not vary significantly. This effect is illustrated in more detail in Figs 4(a) and (b), in which the size distribution of solute clusters or GP zones corresponding to samples naturally pre-aged for 5 days and samples with an additional artificial ageing of 8 min at 150\( ^\circ \)C, respectively, is shown. The size of type B (ellipsoidal) particles and of type A (edge-on \( \eta' \) particles) increases with ageing time. The density of these types of particles is similar, although that of type B is slightly larger. The maximum diameter of type B particles is also smaller than that of type A particles. The density of both types of particles is approximately one order of magnitude smaller than that of solute clusters or GP zones, while the volume fraction is of the same order of magnitude.

After reaching the minimum value of \( r \) during artificial ageing (point 3, Fig. 1), samples were re-aged at RT for a few months (\( > 3.5 \) months) and it was observed that the positron lifetime increased to the same value as the samples naturally pre-aged for 5 days (\( \sim 212 \) ps, labelled R, Fig. 1). The observed contrast in the TEM micrographs (not included here) was similar to that shown in Fig.
2(c), although the sizes of particles of type A and B were slightly larger.

The BF and HREM images obtained along the $h011i$ zone axis are shown in Figs 5(a) and (b), respectively, and correspond to the state labelled 4 in Fig. 1 ($\tau$ = 3 h or 5.37 min$^{1/3}$ of artificial ageing at 150°C). Here the positron lifetime reaches the RT pre-ageing value of 12.5 ps. Particles of type A and B with sizes between 3 and 10 nm are observed in the micrograph of Fig. 5(a). Contrast arising from solute clusters or GP zones is no longer visible. The size and density of $\eta'$ plates clearly increase with longer ageing times. In addition, type B particles show facets as from 2.5 h ageing. The traces of the longer facets are along $\{111\}$ planes and some shorter facets with $\{002\}$ traces can also be observed.

The microstructure corresponding to the state labelled 5 in Fig. 1 ($\tau$ = 8 h of artificial ageing at 150°C), in which $\tau$ reaches its maximum value, is shown in Fig. 5(c). The microstructure is similar to that observed in Fig. 5(a), but with higher number density and size of type A and B particles. Faceting of the precipitates is also more pronounced.

The mean $\eta'$ precipitate radius as a function of the ageing time at 150°C is plotted in Fig. 6. Each point represents the mean value of 25 measurements. Only particles in the thin regions of the specimens were considered, in order to avoid corrections due to overlap. For comparison, the mean radius of spherical particles with hexagonal structure obtained in the alloy Al–4.84Zn–2.06 Mg (wt%) by Lyman and Vander Sande [32], is also included in this figure. In the top inset, the evolution of $r^3$ against ageing time for the early stages of artificial ageing is shown.

4. DISCUSSION

4.1. Natural ageing

In the samples aged only at RT the contrast observed in the TEM micrographs [Figs 2(a) and (b)] corresponds either to solute clusters or to GP zones. There is no evidence for the formation of $\eta'$ precipitates in accordance with the results of Mukhopadhyay et al. [10]. The solute clusters/GP zones density obtained in the present work, is of the same order of magnitude as that obtained for naturally aged samples of experimental Al–Zn–Mg alloys with a different composition than that of 7012 alloys. This disagreement could be attributed to composition differences and the presence of secondary alloying elements in the 7012 alloy and/or to the higher sensitivity of the microscopic techniques used nowadays.

As mentioned in Section 1, it has been proposed that two types of GP zones form in Al–Zn–Mg alloys. The mechanism of their formation is related to the relative solute–vacancy interaction [4].
Fig. 5. Micrographs of artificially aged samples at 150°C obtained along the (011) zone axis. (a) Bright-field image for 2.5 h (labelled 4, Fig. 1). (b) HREM image with the same thermal treatment as (a). (c) Bright-field image for 21 h (labelled 5, Fig. 1).
would then be expected to be the same as that of with an oval shape. The density of type B particles correspond to edge-on disc-shaped have been observed with TEM. Type A particles kinds of morphology, named type A and B particles stage of artificial ageing, precipitates showing two by a significant loss of vacancies. This process of partial dissolution is accomplished the above interpretation, and indicates that clusters/GP zones illustrated in Fig. 4, which sup-

Fig. 6. Variation of the cube of the average precipitates \( \eta' \) radius as a function of ageing time at 150°C (full symbols). Open symbols correspond to the results of Lyman and Vander Sande [31] in Al–4.84 Zn–2.06 Mg (wt%).

Despite the rapidity of migration of quenched-in vacancies at RT in Al, it is proposed that a portion of them become trapped by Mg atoms. This process has been associated with the decrease of the positron lifetime during pre-ageing shown in the middle left-hand part of Fig. 1 (see also Ref. [22]). Recently, some of the authors of the present paper have suggested that the dominant diffusing species during pre-ageing treatments in the 7012 alloy were Mg–vacancy-rich GPII zones [25], which evolve to form solute/vacancy-rich GPII zones and which provide deep traps for positrons, exhibiting a characteristic lifetime of about \( \sim 212 \) ps [20, 25] (labelled 1, Fig. 1).

4.2. Artificial ageing

During the first minutes of artificial ageing, positron lifetime has a rapid fall which correlates with a softening of the material. This effect has been attributed to partial dissolution of the smallest GPII zones in which positrons annihilate [25]. Due to this dissolution, an increasing fraction of positrons are annihilated in the Al matrix, whose positron lifetime is shorter (\( \sim 164 \) ps [20]), producing a decreasing average lifetime. The decrease in \( \tau \) is consistent with the decrease in size of the solute clusters/GP zones illustrated in Fig. 4, which supports the above interpretation, and indicates that this process of partial dissolution is accomplished by a significant loss of vacancies.

In addition to the decrease of \( \tau \), during the initial stage of artificial ageing, precipitates showing two kinds of morphology, named type A and B particles have been observed with TEM. Type A particles correspond to edge-on disc-shaped \( \eta' \) precipitates [2, 33] that have \{111\} habit planes parallel to the \( \langle 011 \rangle \) zone axis. The B particles are assumed to be \( \eta' \) precipitates with habit planes inclined to the \( \langle 011 \rangle \) zone axis, which show up in the micrographs with an oval shape. The density of type B particles would then be expected to be the same as that of type A particles. Since the density of type B particles was found to be slightly higher, and the maximum diameter smaller than that of type A particles, a fraction of the type B particles may be a second type of precipitate, probably the hexagonal precipitates observed by Lyman and Vander Sande [32]. The densities of \( \eta' \) precipitates show a good agreement with those found by other authors (see Refs [13, 33]).

The micrographs of Figs 2(c)–(e) show that \( \eta' \) precipitates are formed already in the very early stages of artificial ageing, and coexist with dissolving solute clusters or GP zones.

After reaching a minimum value labelled 3 in Fig. 1, the positron lifetime increases with increasing ageing time. It has been proposed that positrons can annihilate in the free volume of the partially coherent \( \eta'/\text{Al-matrix} \) interfaces [20]. This interpretation is supported by the present results, which show that the \( \eta' \) precipitates produce distortions in the matrix, as shown in the HREM image of Fig. 5(b) (labelled 4, Fig. 1). The increase in positron lifetime is therefore attributed to the increase in the \( \eta' \) number density, illustrated in the micrographs of Figs 5(a) and (c). The maximum of the \( \tau \) vs ageing time curve (labelled 5, Fig. 1) indicates that, at this stage, positron annihilation would be produced by a saturation regime dominated by trapping at the interface of \( \eta' \) precipitates [23, 26].

The present results given in Table 1 and Fig. 6 show that during the early stages of artificial ageing the \( \eta' \) precipitates grow. A good correlation exists between the \( \eta' \) precipitates’ mean radius (open symbols) and Vickers microhardness (full symbols) during ageing at 150°C shown in Fig. 1 (top left-hand corner), which confirms that the \( \eta' \) precipitates are responsible for the increase in hardness.

Our data are shown in Fig. 6 together with those obtained by Lyman and Vander Sande [32]. Both sets of data show that during the first minutes of artificial ageing the cube of the average precipitate radius grows linearly with the ageing time. Although more experimental information on the ageing time between \( \sim 30 \) min and \( \sim 150 \) min is necessary, the present results suggest that \( r^3 \) saturates for higher ageing times, in agreement with the results of Lyman and Vander Sande. From the TEM observations it was found that the aspect ratio \( (D/\bar{a}_v) \) maintains a value of \( \sim 5.5 \) indicating that, as a first approximation, \( r^3 \) represents the mean volume of the precipitates. Further work is in progress to clarify these arguments.

Gueffroy and Löfler [16] investigated the reversion behaviour of an Al–Zn–Mg alloy, during artificial ageing at 160, 180 and 200°C using SAXS. The specimens had been pre-aged at 100°C for different times in order to produce GP zones with start radii between 1.2 and 2.7 nm. They observed a decrease of the integral intensity \( Q_s \) during the early stages of artificial ageing, and attributed this effect to the
Dissolution of unstable GP zones. They also found that the mean size of the particles did not change, which is in disagreement with our results. This discrepancy may be attributed to the fact that the measurements of Guéfroy and Lööfker represent the mean size of all particles, which include GP zones, and also the larger \( \eta \) precipitates and ellipsoidal zones observed in this work.

4.3. Room temperature re-ageing

In samples re-aged at RT for over 3.5 months after the minimum of the positron lifetime during artificial ageing was reached, \( \tau \) recovers to the initial value (see solid line up to point labelled R in Fig. 1). Since the density of \( \eta \) precipitates in the matrix was relatively low, the probability that positrons annihilate in traps associated with these precipitates is expected to be small. Therefore, the increase of \( \tau \) is attributed mainly to the growth of solute clusters or GP zones during the reconstruction process [22]. The solute diffusion mechanism would be the same as that proposed for GP zone formation, although the kinetics is slower due to the absence of excess vacancies [25].

4.4. Final remarks

Based on the previous discussion, the particles formed during pre-ageing at RT may be identified as either pre-precipitate solute clusters or GP zones. Because the artificial ageing temperature (150°C) is above the metastable phase boundary for GP zones, both solute clusters and GP zones are expected to dissolve during artificial ageing. It is therefore not possible to distinguish between both types of particles.

During the early stage of artificial ageing the solute clusters, which are generally regarded sub-critical\(^\dagger\), and/or the smallest GP zones formed at RT gradually dissolve simultaneously with the formation of \( \eta \) precipitates. This phenomenon has been previously named ‘partial dissolution’ or ‘partial reversion’ [3]. The nucleation of \( \eta \) precipitates is likely to occur at large GP zones as observed by Mukhopadhyay et al. [10]. The present results show that the \( \eta \) precipitates are formed already in the very early stages of artificial ageing and that the number density of \( \eta \) precipitates is one order of magnitude smaller than that of solute clusters or GP zones. These observations suggest that a small fraction of the latter may indeed serve as nucleation sites for the \( \eta \) precipitates.

5. CONCLUSIONS

Due to the difficulty of imaging precipitates smaller than 5 nm, most previous TEM work has concentrated on the later stages of precipitation. In this work quantitative information on the size, density and morphology of solute clusters/GP zones and metastable precipitates formed during the early stages of artificial ageing was obtained. This information was correlated with PALS and microhardness data. On the basis of the present results the following conclusions are drawn.

1. After quenching and during natural ageing, spherical particles with sizes ~1 nm are formed. They are identified as pre-precipitate solute clusters or GP zones.
2. There was no evidence of \( \eta \) precipitation in pre-aged samples.
3. During the early stages of artificial ageing at 150°C the solute clusters and/or GP zones dissolve partially and \( \eta \) precipitates are simultaneously formed. A third type of precipitate may also be present. The morphology, size and density of the precipitates were studied in detail.
4. The partial dissolution of solute clusters/GP zones is accomplished with a significant loss of vacancies; therefore, these point defects could have an important role on the \( \eta \) precipitation kinetics.
5. During artificial ageing the increase in Vickers microhardness and positron lifetime is related to the growth of \( \eta \) precipitates. At the early stages the growth of \( \eta \) precipitates follows a law such as \( r^\gamma \propto \tau_{\text{aging}} \).
6. During re-ageing, the growth of the \( \eta \) precipitates as well as the other identified particles in the microstructure was observed.

The microstructural description given in the present study partially confirms some hypotheses of previous works. In addition, quantitative information on the precipitates and their precursors has been obtained using transmission electron microscopy. Further characterization using the higher resolution of the APFIM technique would allow the determination of the composition of the precipitates.

Acknowledgements—Discussions of TEM results with Francisco Lovey (Centro Atómico Bariloche) are gratefully appreciated. This work was supported by the Consejo Nacional de Investigaciones Científicas y Técnicas (PIP/BID No. 4318/97), Agencia Nacional de Promoción Científica y Tecnológica (PICT No. 0192/98), Comisión de Investigaciones Científicas de la Provincia de Buenos Aires and Secretaría de Ciencia y Técnica (UNCentro), Argentina.

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\^\dagger\) Note that in this type of alloy there exists a critical size \( D^* \) below which the particles are unstable against the dissolution.