

Study of formation and reversion of Guinier-Preston zones in Al-4.5 at %Zn-x at %Mg alloys by positrons

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Measurements of the positron lifetime, τ , and the shape parameter, S , of the 2γ -angular correlation curve are reported for the Al-4.5 at %Zn-x at %Mg alloys ($0 \leq x \leq 2.5$). In the alloys aged at room temperature after quenching two types of Guinier-Preston (GP) zones coexist both of which are able to trap positrons. The first type of zone is rich in zinc and completely free of any vacancy-type defects. The second type contains vacancies surrounded by zinc and magnesium atoms. From the comparison of τ and S , the composition of the surroundings of vacancies as a function of the magnesium concentration of the alloys is concluded. By increasing the magnesium content and the ageing time of the alloys the second type of GP zone increasingly dominates over the first one. The stability of both types of zone are studied in a reversion experiment. In an alloy with 2.5 at %Mg the formation of the η' and η -phases are observed. The positron annihilation results are compared with the results obtained by calorimetry, small angle X-ray scattering and electron microscopy on the same alloy series.

1. Introduction

The precipitation process in the ternary Al-Zn-Mg system has been the subject of several investigations, (see [1, 2] and references therein). Among them there are several papers dealing with the formation of Guinier-Preston (GP) zones. The coexistence of two types of GP zones in this alloy system was concluded by various authors [3-5]. Recently differential scanning calorimetry (DSC), transmission electron microscope (TEM) and small angle X-ray scattering (SAXS) investigations have been performed on a series of Al-4.5 at %-x at %Mg ($0.05 \leq x \leq 3$) alloys, post-quench aged for different times at room temperature [2]. The authors concluded that in alloys with less than 0.5 at %Mg two types of GP zone are formed at the beginning. During long-term ageing the less stable zones transform into the more stable ones.

Positron annihilation is a method that provides information about the concentration, configuration and internal structure of lattice defects [6-8]. The investigation of precipitation processes in age-hardenable alloys is a relatively new application of this method. The role of quenched-in vacancies on the formation and dissolution processes of GP zones and precipitates was studied by various authors on Al-Zn [9-12] and Al-Zn-Mg [13, 14] alloys. Recently two types of GP zones were identified with the aid of positrons in Al-3 at %Zn-x at %Mg and 6 at %Zn-x at %Mg alloys [15].

The purpose of the present work is to study the

Al-4.5 at %Zn-x at %Mg alloy series with the aid of the positron annihilation technique (positron lifetime as well as momentum distribution measurements) in order to obtain further information on the properties and behaviour of GP zones. Our results are compared with the ones obtained by DSC, SAXS and TEM [2] and also with the positron results on the Al-3 at % Zn-x at %Mg and 6 at %Zn-x at %Mg series [15]. Early results of positron investigations on a limited series of Al-4.5 at %Zn-x at %Mg alloys have been published in [13] and [14].

2. Experimental details

The positron annihilation measurements were performed for a series of Al-Zn-Mg alloys with a constant zinc concentration of 4.5 at % and with magnesium concentrations of 0.05, 0.1, 0.2, 0.5, 1 and 2.5 at %, respectively. The samples were cut from the same stock as those used in [2]. After solution annealing for 30 min at 480°C, the samples were quenched in room temperature water. Thereafter the samples were aged at room temperature for 1 h, 10 days, 3 months, 6 months, 1 year and 2 years. In addition to the Al-4.5 at %Zn-x at %Mg alloys, the series Al-3 at %Zn-x at %Mg ($x = 0.5, 0.8$ and 1.5) and Al-6 at %Zn-x at %Mg ($x = 0.1, 0.5$ and 1.4) studied already in [15] were measured after 10 days ageing.

Positron lifetime measurements were performed using a conventional fast-slow coincidence system (see, for example [6]) with a time resolution of 260 psec. Two

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identical samples were sandwiched with a $10\ \mu\text{Ci}$ positron source which was made by evaporating a carrier-free $^{22}\text{NaCl}$ solution onto a thin ($1.1\ \text{mg cm}^{-2}$) nickel foil. Each spectrum was measured for 4 h, during which time about 2×10^6 pulses were accumulated. After subtracting the background and source components the lifetime spectra were analysed as a sum of exponential decay components. One or, in some cases, two components enabled a satisfactory fit. From the analysed lifetimes τ_i and the relative intensities I_i , the average positron lifetime $\bar{\tau} = \sum \tau_i I_i$ was calculated. The statistical error of $\bar{\tau}$ was about ± 2 psec.

Momentum distribution measurements were performed by measuring the 2γ -angular correlation curve with a conventional long-slit device (see, for example [6]) having a resolution of $5\ \text{mrad} \times 230\ \text{mrad}$. Positrons of a $120\ \text{mCi}$ ^{22}Na source were focused onto the sample by a magnetic field of 0.5 T. Each angular correlation curve was measured for 6 h resulting in a total number of 4×10^6 counts. From the curve the shape parameter S was calculated as the relative area of a fixed central region (-3 to $3\ \text{mrad}$), which was chosen to give an S value about 0.5 for well annealed, pure aluminium. The statistical error of S was approximately $\pm 0.15\%$.

For Al-4.5 at % Zn with 0, 0.05 and 2.5 at % Mg respectively, post-quenched aged for 3 months, positron annihilation measurements were performed as a function of the sample temperature on the range between 30 and 400°C . Positron lifetime measurements were performed at a given sample temperature during a period of 6 h. After each measuring period, the next temperature was reached within 10 min and the measurement was started again. The S parameter measurements were performed in an analogous way.

3. Results and discussion

3.1. The effect of alloy composition and ageing time on the GP zone properties

Figs. 1a and b present the results of the positron lifetime and S parameter measurements on the series of Al-4.5 at % Zn- x at % Mg alloys aged for different times at room temperature after quenching from 480°C . The results for the samples aged for 0.5 and 1

year are identical to those for the 2 years aged samples and therefore not shown.

In the binary Al-4.5 at % Zn alloy the positron lifetime spectrum has only one component with a lifetime value of 155 psec. The S parameter amounts to $0.911 S_{\text{Al}}$. Both, τ and S , are independent of the ageing time. This behaviour is due to the saturated positron trapping by completely coherent Zn-rich GP zones free of any vacancy-type or dislocation-type defect [15]. We denote this type of GP zones by GPZ1. The reason of the positron trapping by these zones is connected with the positron affinity to zinc atoms in an Al matrix (see discussion in [9]).

Even in the Al-4.5 at % Zn-2.5 at % Mg alloy a single lifetime component was measured independently of the ageing time, but the lifetime value is in this case of 220 psec. This value indicates that all positrons annihilate from a state localized at a vacancy. The S parameter amounts to $1.010 S_{\text{Al}}$ reflecting that the vacancy surroundings are enriched by zinc (see Section 3.2). Such positron traps are not observed in Al-Zn alloys without magnesium. Therefore we conclude that the presence of magnesium stabilizes these vacancies. Obviously positrons are trapped at first by GP zones containing zinc and magnesium. The positrons entering such a zone are then quickly trapped by a vacancy before they annihilate. This type of GP zones we denote by GPZ2.

In Al-Zn-Mg alloys with a magnesium content of x at % ($0 < x < 2.5$) the parameters $\bar{\tau}$ and S are situated in between the limits determined by the GPZ1 and GPZ2 values. Increasing the magnesium content of the alloy $\bar{\tau}$ and S exceed the characteristic value of GPZ1 and approach the GPZ2 value above 1 at % Mg. The decomposition of the lifetime spectrum provides two components with characteristic lifetime values of $\tau_1 = \tau_{\text{GPZ1}} = 155$ psec and $\tau_2 = \tau_{\text{GPZ2}} = 220$ psec. This clearly indicates that all positrons are trapped by both types of GP zones. A certain fraction of positrons are trapped by GPZ2, the rest annihilates inside GPZ1. In the limit of saturation positron trapping the intensity of the second component, I_2 , is identical to the fraction, η_{GPZ2} , of positrons trapped by GPZ2, which can be estimated from the relative change of the

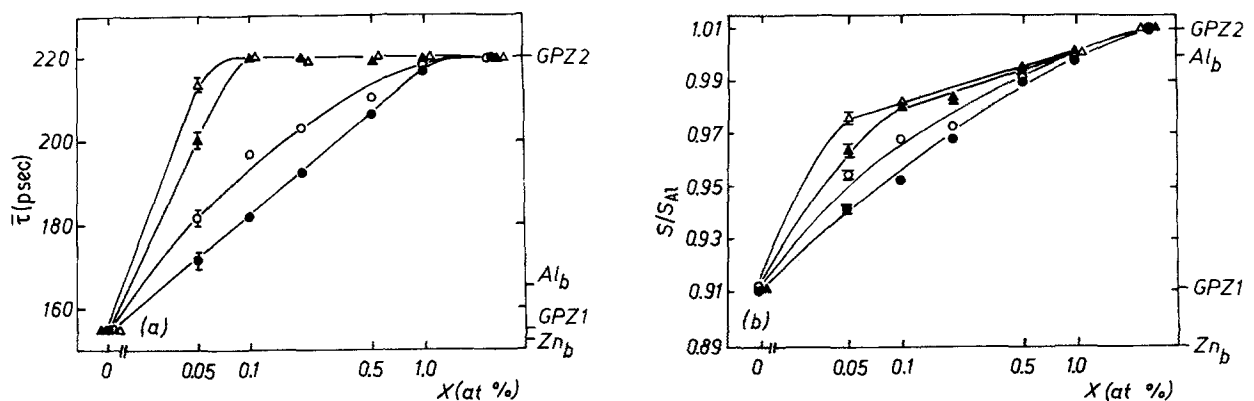


Figure 1 (a) The average positron lifetime $\bar{\tau}$ and (b) the normalized shape parameter S/S_{Al} in Al-4.5 at % Zn- x at % Mg alloys quenched from 480°C into room temperature water and aged at this temperature for (●) 1 h, (○) 10 days, (▲) 3 months and (△) 2 years as a function of the magnesium concentration x . The levels denoted by Al_b , Zn_b , GPZ1 and GPZ2 indicate the characteristic parameters of aluminium and zinc bulk and of the two types of GP zones.

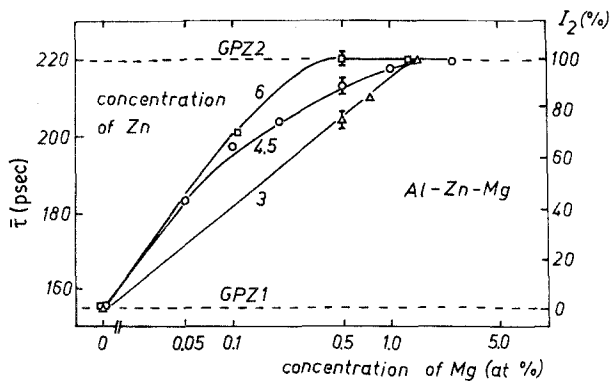


Figure 2 The positron average lifetime and the intensity of the second lifetime component I_2 , respectively, in Al-Zn-Mg alloys quenched from 480°C into room temperature water and aged for 10 days at room temperature as a function of the magnesium concentration x . (Δ) Al-3 at %Zn- x at %Mg, (\circ) Al-4.5 at %Zn- x at %Mg, (\square) Al-6 at %Zn- x at %Mg.

lifetime values

$$I_2 = \eta_{\text{GPZ2}} = \frac{\bar{\tau} - \tau_{\text{GPZ1}}}{\tau_{\text{GPZ2}} - \tau_{\text{GPZ1}}} = \frac{\kappa_{\text{GPZ2}}}{\kappa_{\text{GPZ1}} + \kappa_{\text{GPZ2}}} \\ = \frac{r_{\text{GPZ2}} C_{\text{GPZ2}}}{r_{\text{GPZ1}} C_{\text{GPZ1}} + r_{\text{GPZ2}} C_{\text{GPZ2}}} \quad (1)$$

where $\kappa = 4\pi D_+^{\text{app}} r_i C_i$ ($i = \text{GPZ1}$ or GPZ2 , $D_+^{\text{app}} =$ apparent positron diffusion constant, see [9]) denotes the positron trapping rate into the GP zones, which is proportional to the radius, r_i , and to the volume concentration, C_i , of the GP zones.

Assuming that the radii of both types of zones do not strongly differ, the parameters I_2 and $\bar{\tau}$ are determined by the relative number of the second type of GP zones, $C_{\text{GPZ2}}/(C_{\text{GPZ1}} + C_{\text{GPZ2}})$. As Fig. 1a shows the fraction of GPZ2 increases strongly with increasing the magnesium content of the alloy as well as increasing the ageing time at room temperature. After the long-term ageing (3 months or more) of the alloys with a magnesium content $x \geq 0.1$ at %, only GPZ2 positron traps are found. This behaviour can be explained in the following way. After quenching the alloys to room temperature Zn-rich GP zones (GPZ1) are quickly formed by the migration of well-mobile vacancy-Zn pairs. The strain field of these GP zones induces the precipitation of magnesium atoms into the zones with the subsequent formation of GPZ2, this is the transformation of GPZ1 into GPZ2 (note that in binary Al-Mg alloys 2.3 at %Mg is dissolved in Al at room temperature). Due to the low mobility of vacancy-Mg pairs at room temperature the process takes some time. This explains the fractional increase of GPZ2 with increasing ageing time as well as with increasing the magnesium content of the alloy (see also discussion in [1], p. 2220).

Fig. 2 shows the influence of zinc content (3, 4.5 and 6 at %) on the GP zone properties of the Al-Zn-Mg alloy. The rise of $\bar{\tau}$ and I_2 with increasing magnesium content becomes steeper for those series of alloys having the highest zinc content. This indicates that for a given magnesium content and a given ageing time the relative number of GPZ2 increases with increasing the zinc content of the Al-Zn-Mg alloy. This

observation supports the picture of strain-induced diffusion of magnesium to GP zones and its incorporation into the zones. The larger the size (i.e. the strain field) of the GP zones, the stronger the magnesium precipitation into the zones. For a given ageing temperature and time the size of the GP zones increases with increasing the zinc content of the alloy. Therefore in alloys with a higher zinc content more magnesium precipitates and a larger fraction of GPZ2 are formed.

3.2. Composition of the GP zones

If positrons are completely trapped by one type of GP zones, the annihilation parameters, $\bar{\tau}$ and S , reflect directly the type of positron localization and the chemical composition of the region where the positron annihilates [15]. The chemical composition can be estimated with quite a high accuracy, relating linearly the GP zones annihilation parameters with the corresponding parameters of the pure metals. In the case of completely coherent and defect free GP zones (GPZ1) positrons annihilate from a state spread out over the whole GP zone. The S parameter of the GPZ1 will be given by

$$S_{\text{GPZ1}}^{\text{Al}} = (1 - C_{\text{Zn}})S_{\text{b}}^{\text{Al}} + C_{\text{Zn}}S_{\text{b}}^{\text{Zn}} \quad (2)$$

where S_{b}^{Al} and S_{b}^{Zn} denote the bulk positron annihilation parameters of the pure metals aluminium and zinc respectively [15]. With the experimental values $S_{\text{GPZ1}}^{\text{Al}} = 0.911 S_{\text{Al}}$, $S_{\text{b}}^{\text{Zn}} = 0.890 S_{\text{Al}}$ and $S_{\text{b}}^{\text{Al}} = S_{\text{Al}}$, we estimate a zinc content of 81 ± 2 at %. Applying the correction which takes into account the positron affinity to zinc atoms (see [9]) we obtain a better estimation of 70 ± 5 at % in agreement with SAXS results [16, 17].

In the case of GPZ2 positrons annihilate from a state strongly localized at a vacancy, inside the GP zones containing zinc and magnesium atoms. Relating the S parameter of the GPZ2 with the characteristic parameters of vacancies in the pure metals Al ($S_{\text{v}}^{\text{Al}} = 1.05 S_{\text{Al}}$), Zn ($S_{\text{v}}^{\text{Zn}} = 0.966 S_{\text{Al}}$), and Mg ($S_{\text{v}}^{\text{Mg}} = 1.13 S_{\text{Al}}$) one obtains a rough estimate for the composition of the surroundings of the vacancies [15].

$$S_{\text{GPZ2}}^{\text{Al}} = (1 - C_{\text{Zn}} - C_{\text{Mg}})S_{\text{v}}^{\text{Al}} + C_{\text{Zn}}S_{\text{v}}^{\text{Zn}} \\ + C_{\text{Mg}}S_{\text{v}}^{\text{Mg}} \quad (3)$$

The value $S_{\text{GPZ2}}^{\text{Al}} = 1.010 S_{\text{Al}}$, found for GPZ2 in Al-4.5 at %Zn-2.5 at %Mg, is compatible with a number of 1, 2 or 3 magnesium atoms and 7, 8 or 9 zinc atoms among the twelve near-neighbour sites of a vacancy.

In alloys with a low magnesium concentration the second type of GP zones can be expected to contain less magnesium atoms than in Al-4.5 at %Zn-2.5 at %Mg. The variation in the composition of the GPZ2 could affect the positron annihilation parameters. In the positron lifetime spectra we have not observed such an effect, $\tau_2 = \tau_{\text{GPZ2}} = 220$ psec is practically independent of the alloy composition and ageing time. The reason is that the positron lifetime reflects mainly the open volume at the site of positron localization i.e. the vacancy volume, which does not

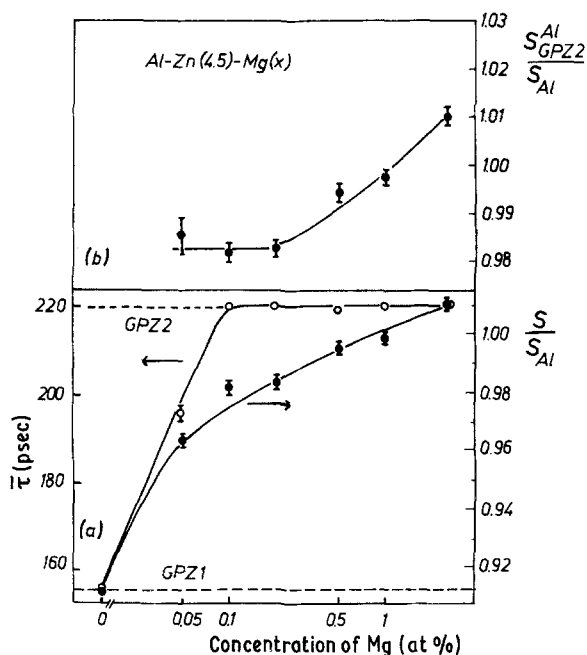


Figure 3 (a) (○) The average positron lifetime $\bar{\tau}$ and (●) the normalized shape parameter S/S_{Al} in Al-4.5 at % Zn-x at % Mg alloys quenched from 480° C into room temperature water and aged for 3 months at that temperature. The scales of $\bar{\tau}$ and S/S_{Al} data are adjusted to obtain the same minimum and maximum values, denoted by GPZ1 and GPZ2. (b) The characteristic shape parameter of the second type of GP zones, S_{GPZ2}^{Al}/S_{Al} as a function of the magnesium content of the alloys, estimated from the comparison of the $\bar{\tau}$ and S data.

change. However, the S -parameter is more sensitively affected by the chemical surroundings of the positron trap [15]. As an example of this, Fig. 3a shows that for the alloy aged for 3 months, S and $\bar{\tau}$ do not behave in the same way. The S values fall below the ones corresponding to $\bar{\tau}$. Assuming that τ_{GPZ2} is independent of the GPZ2 composition, this behaviour has to be attributed to a composition dependence of the characteristic S parameter of GPZ2, S_{GPZ2}^{Al} . From the comparison of $\bar{\tau}$ (or I_2) and S we have estimated the S_{GPZ2}^{Al} value. As Fig. 3b shows, S_{GPZ2}^{Al} decreases with the decreasing of magnesium content in the alloy, from 1.010 towards a level of 0.983 S_{Al} . The lowest S_{GPZ2}^{Al} value (0.983 S_{Al}) corresponds to a maximum number of not more than one magnesium atom, whereas eleven zinc atoms

should occupy the nearest neighbour places of a vacancy. However, the decrease of S_{GPZ2}^{Al} with decreasing the nominal magnesium concentration of the alloy reflects clearly the decrease in magnesium content and/or the increase in the zinc content of the surroundings of vacancies existing inside GPZ2. The positron annihilation results correlate qualitatively with SAXS estimations of the GP zone composition which provide values of 20 to 55 at % Mg and 40 at % Zn [1, 18, 19] and showed an increase of the magnesium content of the GP zones with increasing the magnesium concentration of the alloy [19]. In comparison we have to take into account that the positron annihilation parameters reflect the composition of the surroundings of the vacancies which trap positrons inside the zones rather than the zone composition itself.

3.3. Reversion of Guinier-Preston zones

The behaviour of the average positron lifetime $\bar{\tau}$ and the S parameter in Al-4.5 at % Zn alloys with 0, 0.05 and 2.5 at % Mg, post-quench aged for 3 months at room temperature, as a function of the sample temperature is shown in Fig. 4. In the binary Al-4.5 at % Zn alloy positrons are completely trapped by GPZ1 resulting in the lowering of $\bar{\tau}$ and more strongly of S compared with the aluminium bulk values. The rapid increase of both $\bar{\tau}$ and S at 120° C reflects the dissolution of GPZ1. The annihilation parameters then nearly correspond to the aluminium bulk values which show that the large majority of positrons annihilate from a delocalized state in the Al-4.5 at % Zn alloy. No indications of positron traps such as lattice defects or GP zones are observed. The slight increase of $\bar{\tau}$ and S below 220° C can be attributed to the thermal expansion of the sample. Above 220° C the annihilation parameters exhibit a steeper rise due to positron trapping by the increasing number of thermally generated vacancies.

In the case of Al-4.5 at % Zn-0.05 at % Mg all positrons are trapped by GPZ1 and GPZ2, in the post-quenched 2 months aged state. The lifetime spectrum consist of two components with lifetime values $\tau_1 = \tau_{GPZ1} = 155$ psec and $\tau_2 = \tau_{GPZ2} = 220$ psec and with the intensity $I_2 = 60\%$. The positron trap-

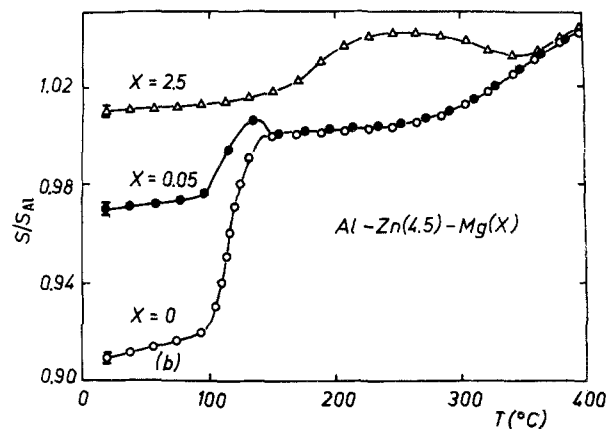
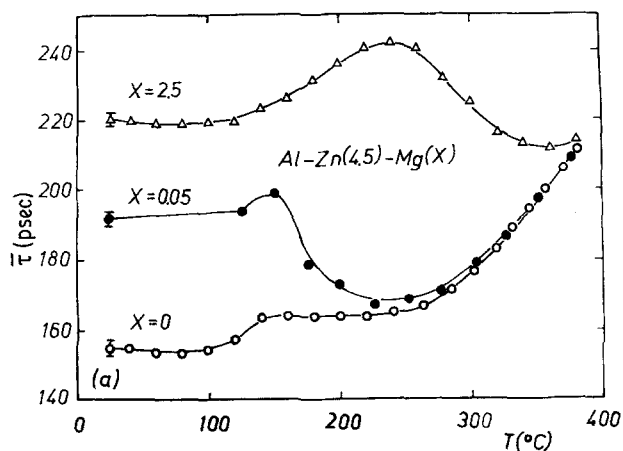


Figure 4 (a) The average positron lifetime $\bar{\tau}$ and (b) the normalized shape parameter S/S_{Al} as a function of the sample temperature (6 h measuring time per point) in Al-4.5 at % Zn-x at % Mg with (○) $x = 0$, (●) 0.05 and (Δ) 2.5 post-quench aged for 3 months.

ping by GPZ1 leads to the fractional decrease of S (compared with Al–4.5 at %Zn), whereas the positron trapping by GPZ2 leads to the fractional increase of $\bar{\tau}$ and I_2 (compared to Al–4.5 at %Zn–2.5 at %Mg). The S parameter shows more sensitivity to the presence of GPZ1, however, the positron average lifetime, $\bar{\tau}$, reflects more clearly the presence of GPZ2. Above 100°C, S increases rapidly. This is caused by the dissolution of GPZ1 which leads also to a weak increase in $\bar{\tau}$. Due to the dissolution of GPZ1, more positrons are trapped by GPZ2. Above 180°C the value of τ_1 corresponds to the one predicted by the one trap model $\tau_1 = 1/(1/\tau_b^{Al} + \kappa_{GPZ2})$ [6]. This indicates that GPZ1 is completely dissolved and positrons annihilate either from the matrix or from the GPZ2 trap. The positron trapping rate into the GPZ2 positron trap, can be estimated by the equation

$$I_2 = \kappa_{GPZ2} / \left(\frac{1}{\tau_b^{Al}} + \frac{1}{\tau_{GPZ2}^{Al}} + \kappa_{GPZ2} \right) \quad (4)$$

With the experimental value $I_2 = 53\%$ at 180°C we obtain $\kappa_{GPZ2} = 1.7 \times 10^9 \text{ sec}^{-1}$. Assuming a GPZ2 radius of 3 nm this trapping rate corresponds to a concentration of $1.1 \times 10^{15} \text{ GP zones cm}^{-3}$. The decrease in $\bar{\tau}$ (and in I_2) above 150°C can be attributed to the dissolution of GPZ2. At 240°C the annihilation parameters corresponds almost completely to the aluminium bulk values indicating the total dissolution of GPZ2. Above the temperature $\bar{\tau}$ and S rise again due to the increasing positron trapping by thermally generated vacancies and vacancy-alloying atom complexes.

In Al–4.5 at %Zn–2.5 at %Mg all positrons annihilate from GPZ2 positron traps with a characteristic lifetime of 220 psec and a characteristic S parameter of 1.010 S_{Al} . $\bar{\tau}$ as well as S are constant up to 120°C, thereafter a smooth increase follows. This indicates that despite of the partial dissolution of GP zones observed by SAXS [2] there are enough GPZ2 to trap all positrons. The increase of $\bar{\tau}$ and S above 120°C can be attributed to the formation of new types of positron traps. These new positron traps can be identified as the semicoherent η' -particles and the incoherent η -particles [2, 13, 14]. Positrons are expected to become localized in the incoherent parts of the particle–matrix interface, resulting in an increase of both $\bar{\tau}$ and S parameter [14].

3.4. Comparison with DSC, SAXS and TEM results

The positron annihilation results of the present study correlate very well with the results of the investigation of the same series of alloys performed by DSC, SAXS and TEM techniques [2]. In alloys with low magnesium content ($x \leq 0.5$ at %) two exothermic peaks were observed in the DSC thermograms and they were attributed to the dissolution of two types of GP zones. In the alloys aged for a longer period or with a richer magnesium content, only one dissolution peak was observed indicating that only the more stable GP zones exist. Obviously both types of GP zones observed by DSC are identical to the ones detected by positrons (and denoted above by GPZ1 and GPZ2). As observed

by DSC, magnesium stabilizes the zones on the one hand and is connected with vacancies on the other, as shown by positrons. The results show that already a small magnesium content in the zones is sufficient to cause both effects. Comparing the heating results, the different heating rates and the different sensitivity of the various methods have to be taken into account. By positron annihilation GP zones dissolution temperatures occur at about 120 and 180°C, whereas the DSC thermograms exhibit two dissolution peaks at about 80 and 160°C. The origin of this discrepancy has to be attributed to the high and nonlinear dependence of the fraction of trapped positron on the trapping rate (see [6]). Therefore, the positron annihilation shows mainly the late stage of the GP zone dissolution. The same reason e.g. the high and nonlinear sensitivity and the slow effective heating rate of 0.1 K min^{-1} used in the positron study leads to indications about the formation of the η' -phase already above 100°C. The DSC thermograms taken with a heating rate of 40 K min^{-1} showed exothermic peaks at about 205, 240 and 290°C attributed on the basis of TEM results to the homogeneous and heterogeneous nucleation of the η' -phase and to the formation of the η -phase. The η -phase is clearly separated in the positron lifetime spectra above 300°C. The general behaviour of the positron parameters correlates well also with the behaviour of the SAXS total scattered intensity which shows that in alloys with low magnesium content (≤ 0.5 at %) no other phase formation process is observed after complete dissolution of GP zones at about 210°C, whereas in alloys with more than 1 at %Mg the formation of more stable η' and η precipitates starts before the total dissolution of GP zones.

4. Conclusions

In the investigated Al–Zn–Mg alloys post-quench aged at room temperature, two different types of GP zones exist which trap positrons. The first type of GP zones (GPZ1) is completely free of any defect (vacancy or dislocation) and contains about 70 at %Zn. The type of this GP zone is the same as the one formed in the binary Al–Zn alloys. The second type of GP zones (GPZ2) contains vacancies having zinc and magnesium atoms in their surroundings. Increasing the magnesium content of the alloy the number of magnesium atoms in the surroundings of the vacancies increases from 1 to 3.

By increasing the magnesium content and the ageing time of the alloys the fraction of GPZ2 increasingly dominates over that of GPZ1. The behaviour is understood assuming strain induced precipitation of magnesium into zinc-rich GP zones. The dissolution of GP zones 1 and 2 occurs at 120 and 180°C, respectively. Positron annihilation is insensitive to the partial dissolution of GP zones, in the Al–4.5 at %–2.5 at %Mg alloy, but on the other hand it shows the formation of the semicoherent η' -phase, already above 100°C and of the incoherent η -phase.

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