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Positron-annihilation study of the aging kinetics of AlCu-based alloys. II. Ag microalloying

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Positron-annihilation spectroscopy has been applied for studying the effects of microalloying additions of Ag to Al-4%Cu and Al-4%Cu-0.3%Mg (wt %) alloys on the aging kinetics and on the association of vacancies to solute elements. The results show that: (i) in Al-Cu, the addition of Ag increases (from 0.32 to 0.61 eV) the effective activation energy that controls the formation of solute aggregates; (ii) on the contrary, in the Al-Cu-Mg alloy, the activation energy is decreased (from 0.65 to 0.22 eV); (iii) in Al-Cu and in Al-4%Cu-0.3%Ag (wt %) alloys the solute aggregates (clusters or GP zones) formed after long aging at temperatures below 70 °C do not contain vacancies; (iv) in Al-Cu-Mg, the addition of Ag enhances the formation of co-clusters (or GPB zones) containing Mg and vacancies; (v) the formation of Mg-rich aggregates is enhanced by Ag also at a high aging temperature (180 °C), and this leads to a better stabilization of the structure produced by artificial aging. The above effects are interpreted as due to the tendency of Ag atoms to bind vacancies in the Al-Cu and vacancy-Mg complexes in Al-Cu-Mg.

I. INTRODUCTION

The effect of microalloying elements on the behavior of age-hardenable alloys is an interesting physical problem, addressing the mechanisms of transport and aggregation of the solute, which has important technological applications (optimization of the thermal treatments, nucleation of precipitates, stability of the microstructure, etc.). Special attention has been given in the literature to the effect of Ag on Al-Cu-based alloys. Enhanced age hardening is stimulated by microalloying additions ($\sim$0.1 at. %) of Ag in a wide range of ternary Al-Cu-Mg alloys.[1,2] In Al-Cu-Mg alloys having high Cu:Mg ratios, Ag promotes the nucleation of a finely dispersed, metastable precipitate (designated Ω), that forms as thin plates of the $\{111\}_\alpha$ planes (e.g., Refs. 3–5). Atom probe field ion microscopy (APFIM) studies have shown that the microalloying addition of Ag promotes clustering of solute atoms[6,7] and it is proposed that these clusters provide sites at which the Ω phase is nucleated probably via the formation of GP zones.[8] Although indirect observations suggest that the effect of Ag may originate in a preferred interaction of Ag atoms, Mg atoms and vacant lattice sites during, or immediately after, quenching from the solution treatment temperature,[1,2] the precise role of vacancies in the nucleation of Ω and of other precipitates remains unknown.

One technique that is capable of detecting and identifying vacancy-type defects is positron annihilation spectroscopy (PAS) (e.g., Refs. 9 and 10). In the present work, an extensive positron annihilation study of age hardening in Al-Cu-based alloys has been performed, with special attention to the association of vacancies to solute atoms. The results are reported in two parts. Paper I, to be found elsewhere in the same issue of this journal, is specifically focused on the ternary alloy Al-4%Cu-0.3%Mg (wt %). It gives the methodological basis for the analysis of the results and establishes the main terms of comparison for attacking the problem of the effect of microalloying additions on the decomposition kinetics. This paper, which is the second part of the report, describes the application of PAS to the study of other compositions (Al-Cu-Ag, Al-Cu-Mg-Ag), where Ag is contained as the minor addition (0.1–0.2 at. %). The binary system Al-Cu is also included in this investigation to provide more comparative data.

II. EXPERIMENT

In this section results are presented regarding the binary system Al-4%Cu (wt %) [corresponding to Al-1.74%Cu

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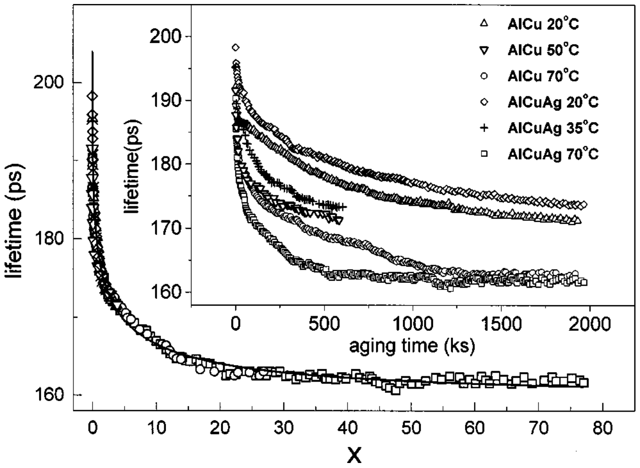

FIG. 1. Positron lifetime in Al-Cu and Al-Cu-Ag during aging at different temperatures versus the aging time (inset) and versus the scaled time variable $x = t/t^*$.

(at. %)], the ternary system Al-4%Cu-0.3%Ag (wt %) [corresponding to Al-1.74%Cu-0.08%Ag (at. %)], and the quaternary system Al-4.5%Cu-0.3%Mg-0.7Ag (wt %) [corresponding to Al-1.97%Cu-0.35%Mg-0.18%Ag (at. %)].

The alloys were solution treated for 25 min at 520 °C in an argon atmosphere and then quenched and aged at 20 to 70 °C in air and 180 °C in a silicone oil bath. The quenching at the end of each thermal treatment above room temperature was made by dropping the sample in an ice-water mixture. Positron lifetime spectra were measured with a time spectrometer with a resolution of 250 ps (full width at half maximum) at a counting rate of 200 coincidences per second. The lifetime spectra were always analyzed as a single exponential decay plus a Kapton source contribution. Although this kind of analysis was always satisfactory from a statistical point of view, nevertheless the mean life of the exponential may have the meaning of an average over an unresolved ensemble of similar components. The standard statistical error of our lifetime measurements is ~0.5 ps. A negative bias (about −2 ps), due to a spurious contribution of prompt coincidences to the spectrum, may affect the absolute lifetime values. More details of the preparation and the thermal treatments of the samples, as well as those regarding the measurements and the analysis of the data, are given in paper I.

A. Experiment 1: One-stage aging at moderate temperatures

The purpose of this experiment was to observe the evolution of the alloy at temperatures low enough to avoid formation of semicoherent or incoherent precipitates. After solution treatment and quenching, the samples were left to age at selected temperatures. The lifetime spectra were measured *in situ* during aging.

1. Al-Cu and Al-Cu-Ag

The evolution of the average positron lifetime τ with the aging time is depicted in the inset of Fig. 1 for Al-Cu and for Al-Cu-Ag. The lower part of Fig. 1 reports the same lifetime data plotted against the reduced time variable $x = t/t^*$. The scaling time constants t^* are reported in Table I. The possibility of scaling the decay curves taken at different temperatures onto a temperature-independent master curve was already demonstrated in paper I for the ternary system Al-Cu-Mg. In that case, the scaling was made in terms of the ''degree of freshness,'' defined by the equation $\Phi = (\tau - \tau_\infty)/(\tau_0 - \tau_\infty)$. The difference here is that the scaling can be obtained in an even simpler form, with no need to subtract the asymptotic lifetime value τ_∞, because all the curves not only begin at the same point, but also converge at the same asymptote. The best-fit curve through the points of the scaled curve corresponds to a Kohlrausch function $\Phi = \exp(-x^\beta)$, with $\beta = 0.320$.

2. Al-Cu-Mg-Ag

Figure 2 shows the evolution of the positron mean life versus the aging time for the quaternary system Al-Cu-Mg-Ag. The best-fit curves through the points are calculated according to the positron-trapping model as described in the next section. As a comparative basis for the visual evaluation of the effect of adding Ag as a quaternary element, Fig. 2 also contains a curve (broken line) that represents the aging at 20 °C of the ternary alloy Al-Cu-Mg (from paper I). Differences between the ternary and the quaternary alloy are evident in this kind of presentation, however, they disappear if the data are scaled in terms of the degree of freshness Φ and of the reduced time variable $x = t/t^*$, with appropriate values of t^* (see Table I). This is shown in Fig. 3, where Φ, calculated with the lifetime data of Fig. 2 and the parameters

TABLE I. Scaling time constants t^* and other parameters used for the calculation of the degree of freshness Φ at various aging temperatures.

Alloy	T (°C)	τ_0 (ps)	τ_∞ (ps)	t^* (ks)
Al-Cu	20	204	161	398 ± 40
Al-Cu	50	204	162	145 ± 15
Al-Cu	70	204	161	71 ± 7.5
Al-Cu-Ag	20	204	161	892 ± 90
Al-Cu-Ag	40	204	161	198 ± 21
Al-Cu-Ag	70	204	161	25 ± 2.5
Al-Cu-Mg-Ag	20	214	198	38 ± 3
Al-Cu-Mg-Ag	35	214	196	24 ± 2
Al-Cu-Mg-Ag	42	212	195	19 ± 2
Al-Cu-Mg-Ag	50	211	195	17 ± 1
Al-Cu-Mg-Ag	65	212	194	12 ± 1

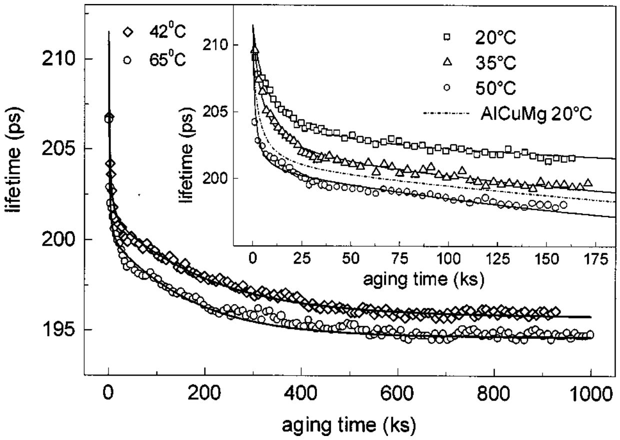

FIG. 2. Positron lifetime in Al-Cu-Mg-Ag during aging at different temperatures versus the aging time; the broken line in the inset shows, for comparison, the curve obtained at 20 °C for Al-Cu-Mg (smoothed experimental data from paper I).

τ_0 and τ_∞ that are given in Table I, is plotted against $x^{0.272}$. The special axis scales adopted for Fig. 3 (a logarithmic scale for Φ and power $x^{0.272}$ for the horizontal axis) help the visual evaluation of the quality of the fitting of the scaled lifetime data with the master curve that was obtained for the ternary alloy (a Kohlrausch function with exponent $\beta = 0.272$). The dashed lines represent the limits of the 1-sigma region of confidence. It is worth commenting briefly on the meaning of the exponent. When $\beta = 1$, the Kohlrausch function is just an exponential decay, with decay rate scaled to 1. This behavior is expected for a single thermally activated atomistic process. For $\beta < 1$, the Kohlrausch function represents a complex decay with a spectrum of decay rates; the width of the spectrum increases when β becomes smaller. The present results ($\beta = 0.320$ for Al-Cu and for Al-Cu-Ag, $\beta = 0.272$ for Al-Cu-Mg and for Al-Cu-Mg-Ag) thus indicate that the width of the spectrum is independent of the temperature and has only a weak dependence on the composition of the alloy, with a tendency to become broader in the presence of Mg.

3. *Effective activation energies*

The temperature dependence of the kinetics of the decomposition process, as observed by PAS, is completely described, in a quantitative way, by the scaling time constants t^*. Although the nonatomistic character of a decay that follows a Kohlrausch law prevents the definition of the activation energy, the observed dependence of t^* on the temperature (shown in Fig. 4) nevertheless makes clear that decomposition is a thermally activated process, and that an effective activation energy can be obtained from the slope of an Arrhenius plot. The best-fit lines reported in this figure correspond to the following effective activation energies: 0.32±0.02 eV for Al-Cu, 0.61±0.07 eV for Al-Cu-Ag, and 0.22±0.03 eV for Al-Cu-Mg-Ag. For comparison, Fig. 4 also shows the data for Al-Cu-Mg (from paper I), that correspond to an effective activation energy of 0.65±0.05.

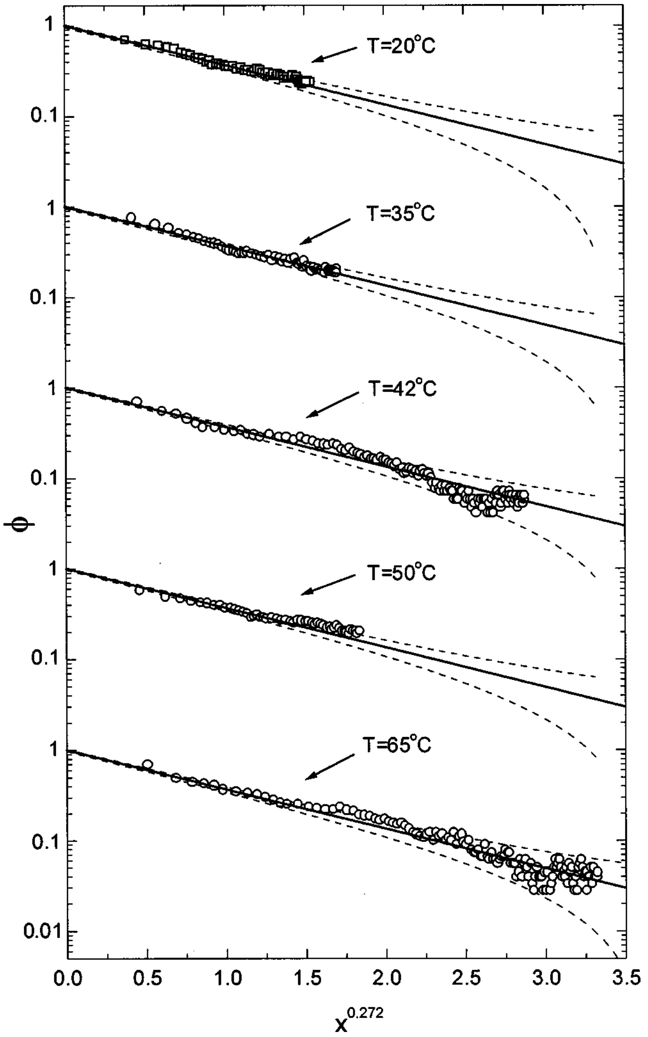

FIG. 3. Degree of freshness $\Phi = (\tau - \tau_\infty)/(\tau_0 - \tau_\infty)$ of Al-Cu-Mg-Ag versus $x = t/t^*$ in a nonlinear scale. The straight lines represent the Kohlrausch function with $\beta = 0.272$ and the broken lines delimit the $1-\sigma$ region of confidence corresponding to a lifetime standard error of 0.5 ps.

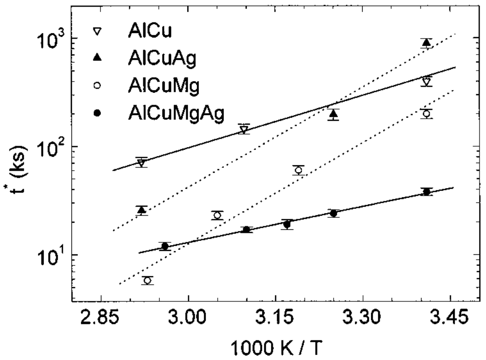

FIG. 4. Arrhenius plots of the scaling time constants t^*.

B. Experiment 2: Artificial aging at 180 °C, followed by natural aging at room temperature

This experiment was performed for the quaternary alloy Al-Cu-Mg-Ag. Positron lifetime measurements were taken at 20 °C after artificial aging at 180 °C for times from 15 s to 10 h and quenching at 0 °C. The purpose of the experiment was twofold: (i) to obtain information on the association of vacancies to solute aggregates at the end of treatments of different duration at a temperature above the solvus of GP zones, (ii) to observe the further evolution at room tempera-

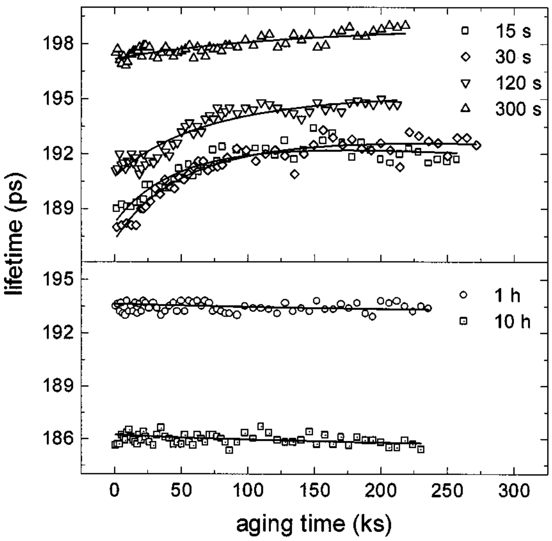

FIG. 5. Positron lifetime in Al-Cu-Mg-Ag during the second aging stage at room temperature versus the aging time. The first aging stage was at 180 °C. The duration of the first stage is given by the curve labels.

ture, in case aging was incomplete during the first stage of the treatment. Our study, addressing vacancies, is in a sense complementary to the three-dimensional atom probe (3D-AP) study,[8] which gives information on solute-solute associations promoted by the same thermal treatment in the same alloy analyzed in the present work. The PAS results are displayed in Fig. 5, where the mean positron lifetime τ is plotted versus the aging time at room temperature. The origin of the time axis corresponds to the quenching at 0 °C at the end of the treatment at 180 °C. The curves through the experimental points of Fig. 5 are from a best-fit with the positron trapping model, as discussed in the next section.

The data of Fig. 5 show a double effect of adding trace amounts of Ag to the system Al-Cu-Mg: (i) the positron lifetime at the end of the artificial aging becomes a function of the artificial aging, whereas it is practically a constant for the ternary alloy; (ii) the lifetime evolution taking place at room temperature is much less evident than for the ternary alloy. A more specific comparison between the ternary and the quaternary system is presented in Fig. 6, where the positron lifetimes at the end of the artificial aging at 180 °C (τ_{aa}, obtained by extrapolation to $t=0$ of the curves in Fig. 5) and at the end of the second stage of aging (τ_{fin}, obtained by averaging the last three points of each curve of Fig. 5), are plotted against the artificial aging time at 180 °C.

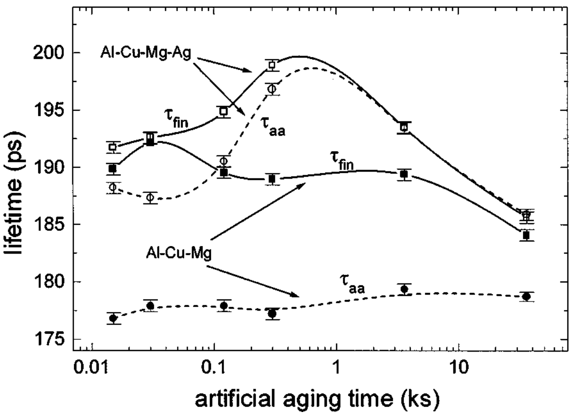

FIG. 6. Positron lifetimes in Al-Cu-Mg and in Al-Cu-Mg-Ag immediately after artificial aging (τ_{aa}) and after further aging at room temperature (τ_{fin}), versus the duration of the artificial aging.

III. DISCUSSION

The results presented in the previous section shed light onto the influence of microlloying elements on the vacancy-solute interactions at various stages of the aging of the alloy, and on the aging kinetics. The two aspects are strictly related, but a separate discussion might be easier to follow.

A. Vacancy-solute interaction

For all the alloys studied in the present work, the extrapolation at $t=0$ of our lifetime curves for one-stage aging (τ_0) leads to values well below the value expected for trapping at monovacancies in pure Al.[11,12] As already mentioned in paper I, isolated vacancies may have disappeared too quickly to be observed by PAS.[13,14] Thus we obtain lifetimes that are essentially characteristics of positron trapped at vacancies associated to isolated solute atoms or to very small solute clusters. The value of τ_0 observed for the ternary and the quaternary systems containing Mg (from 211 to 214 ps) is higher than that obtained for Al-Cu and Al-Cu-Ag (204 ps). The remarkable effect of a small percentage of Mg (only 0.3 wt %) clearly shows a strong tendency of Mg to bind vacancies more effectively than Cu. The vacancy-solute binding energies are 0.25 eV for Mg and less than 0.23 for Cu.[15] Also Ag is known to bind vacancies in Al (binding energy <0.23 eV),[15] but no effect was visible at the small concentrations present in our samples.

The asymptotic value $\tau_\infty = 161$ ps obtained for Al-Cu and Al-Cu-Ag after aging at moderate temperatures (below 70 °C) is the same as for bulk Al, 16–18 ps below that expected for vacancies in a pure Cu environment. The obvious conclusion here is that, after long aging, the residual concentration of vacancies is fallen below the limit of sensitivity of PAS (about 1 ppm in Al). This conclusion contradicts the idea that GP zones in Al-Cu alloys are surrounded by an atmosphere of vacancies.[16] Previous PAS studies on binary Al-Cu alloys give no information on the situation occurring after long aging at moderate temperatures, but show that no positron trapping at vacancies is observed after isochronal aging up to 500 °C.[17] A similar situation occurs for the binary Al-Ag alloy.[18,19]

In the presence of Mg, the situation is entirely different. The survival of vacancies after long aging at moderate temperatures is demonstrated by asymptotic lifetimes τ_∞ between 184.5 and 191 ps for Al-Cu-Mg (paper I, Table I) and between 194 and 198 ps for Al-Cu-Mg-Ag (paper II, Table I). In paper I, the dependence of τ_∞ on the aging temperature has been interpreted as being due to a change in the chemical environment of the sites where the positrons are trapped. Following the same line of interpretation, and considering that the presence of Mg contributes to the increase of τ_∞, the effect of the addition of Ag in trace amounts must be seen as a stabilization of Mg-rich regions. This is in accordance with the tendency of Ag and Mg to form co-clusters, as revealed by atom probe field ion microscopy[6] and by 3D-AP.[8] A

TABLE II. Results of the trapping model analysis for experiment 1.

T (°C)	K_1^∞/K_3^∞	K_2^∞/K_3^∞	t_1 (ks)	t_2 (ks)	t_3 (ks)	χ^2
20	54×10^{-2}	4.35	57	75	660	0.52
35	30×10^{-2}	3.10	31	68	320	0.34
42	9×10^{-2}	2.70	29	55	200	0.17
50	7×10^{-2}	2.58	24	53	180	0.70
65	4×10^{-2}	2.15	15	40	150	0.41

quantitative evaluation of the effect, expressed in terms of the ratio of positron trapping rates in different environments, can be obtained by fitting the lifetime data with the positron-trapping model. The best-fit curves, obtained with the same hypotheses discussed in paper I (i.e., saturated positron trapping in three different families of traps), are shown in Fig. 2. The corresponding model parameters are reported in Table II (t_i are the time constants that govern the temporal evolution of the trapping rates, K_i^0 and K_i^∞ are initial and final values of the trapping rates).

The tendency of Ag to enhance the formation of Mg-rich regions also becomes evident after artificial aging at 180 °C through the general increase of the positron lifetime. In particular, the increase of τ_{aa} due to the addition of Ag (see Fig. 6) indicates the association of vacancies to Mg during the treatment at 180 °C, which was not observable by PAS for the ternary alloy Al-Cu-Mg. The anticipated aggregation of Mg strongly suppresses residual positron lifetime variations occurring at room temperature after the interruption of the heat treatment at 180 °C. Indeed, the data of Fig. 6 show that, for the quaternary alloy, the difference between τ_{aa} and $\tau_{\rm fin}$ quickly disappears whereas, for the ternary alloy, the difference persists even after 10 h at 180 °C. When residual aging at room temperature after quenching from 180 °C can still be observed (i.e., aging time at 180 °C up to 300 s), the kinetics can be reproduced with the help of the positron trapping model in the version described in paper I. The only difference is that the trapping rate K_2^0, which refers to trapping at vacancies in a mixed Cu-Mg environment, cannot be fixed at 0 but must be kept as an adjustable parameter. The best-fit curves shown in Fig. 5 correspond to $t_2=95$ ks, $t_3=700$ ks, and to the other parameters listed in Table III (see above for the definition of the symbols).

B. Kinetics

The decomposition of the alloy at 180 °C is too fast to be followed in real time by PAS. We must therefore limit ourselves to comment the data of Figs. 5 and 6 by observing that 15 s are already enough for observing an important aging effect, and that Ag helps the formation of Mg-rich aggregates.

TABLE III. Results of the model analysis for experiment 2.

Time	K_2^0/K_3^∞	K_2^∞/K_3^∞	K_3^0/K_3^∞	χ^2
15 s	0.59	1.30	0.70	1.13
30 s	0.58	1.49	0.86	0.82
120 s	0.97	2.18	0.89	0.80
300 s	3.19	5.34	1	0.62
1 h	1.71	1.73	1	0.50
10 h	0.52	0.49	1	0.50

More information comes from the study of aging at temperatures below 70 °C. The results of the present work show that the decomposition kinetics at moderate temperatures, as observed by PAS, can be described by a Kohlrausch function. This is reminiscent of other transport-related phenomena occurring in disordered systems as, for instance, the shear stress relaxation in a glass.[20] The similarity might not be accidental. It could be the result of transport phenomena leading to a subcritical structure that does not correspond to a minimum of the free energy of the system (a stable or metastable equilibrium) but is determined by the different evolution rates of competing processes. To be more specific, we are suggesting that the solute aggregates formed at moderate temperatures in ternary and quaternary alloys never reach an equilibrium condition, but freeze with the structure that was progressively built by the aggregation at different rates of the different species of solute.

In a global way, the kinetics is governed by the effective activation energies E_a obtained from the Arrhenius plots of Fig. 4. This energy is in general related to the transport and aggregation of the different migrating species, with greater sensitivity for the species that is/are responsible for the aging effect at times near to t^*. In Al-Cu, an activation energy of only 0.32 eV indicates that the transport of Cu is controlled by the migration of vacancy-Cu pairs surviving after quenching. A contribution of vacancies formed at thermal equilibrium would require an activation energy greater than the formation energy of vacancies in Al ($E_{1v}=0.68$ eV).[12]

The addition of Mg or Ag as a ternary minor element has the effect of increasing the activation energy from 0.32 eV to 0.61–0.65 eV. If the main diffusing species is still the vacancy-Cu pair, a simple explanation of the effect is that the vacancies that survive after quenching are preferentially bound to the ternary element and become available for forming pairs with Cu only after thermal dissociation of the complexes formed with Mg or Ag. In accordance with this explanation, the value of E_a would represent the sum of the binding energy of the donor complex with the migration energy of the vacancy-Cu pair.

The lowest activation energy (0.22 eV) was obtained for Al-Cu-Mg-Ag. It is not easy to explain this result if one assumes that the kinetics is still controlled by Cu atom transport. If one maintains this hypothesis, the conclusion would be that after quenching from 520 °C, the vacancies are preferentially bound to Cu atoms rather than to Mg-Ag atom pairs. There are no structural or geometrical reasons that might support this idea. Moreover, it is in contrast with two of our observations: (i) the positron lifetime measured in as-quenched samples after the solution treatment is about 10 ps longer in Al-Cu-Mg-Ag than in Al-Cu, which is not compatible with a preferential vacancy-Cu atom binding; (ii) a strong association of vacancies to Mg-rich structures is observed at the end of aging at moderate (20–70 °C) and at high temperature (180 °C). Observation (ii), however, suggests an alternative explanation: since aging effects observed by PAS in the Al-Cu-Mg-Ag alloy are strongly affected by the formation of Mg-Ag aggregates, the kinetics at times around t^* could essentially be related to the diffusion of the complex vacancy-Mg-Ag formed during, or immediately after, the quenching at the end of the solution treatment. In this case, $E_a = 0.22$ eV would only be the migration energy of this complex.

IV. CONCLUSIONS

The action of microalloying additions of Ag on the kinetics and on the association of vacancies to the solute, as observed in the present work, can be summarized in the following points:

(i) There is no effect of Ag on the content of vacancies in the solute aggregates formed by aging at temperatures below 70 °C in Al-Cu; this content is zero within the limits of accuracy of PAS in Al (i.e., atomic concentration below 10^{-6}).

(ii) Ag produces a marked increase (from 0.32 to 0.61 eV) in the effective activation energy that controls the kinetics of aging of Al-Cu below 70 °C. Assuming that the diffusion of the main alloying element (Cu) is assisted by association with a vacancy, this result can be interpreted as a vacancy scavenging action of isolated Ag atoms. In this respect, Ag behaves in the same way as Mg does (the addition of Mg in small amounts to Al-Cu raises the effective activation energy up to 0.65 eV). The difference between Ag and Mg is that, in the case of Ag, the vacancies are released after aggregation of the solute (Cu and Ag), while in the presence of Mg the vacancies survive in the solute aggregates.

(iii) Ag greatly enhances the formation of solute aggregates containing Mg and vacancies. This effect is clearly seen both at moderate temperatures (below 70 °C) and at high temperature (180 °C). In the case of artificial aging at 180 °C, the effect of Ag in promoting formation of Mg-rich regions rapidly suppresses subsequent residual aging that, in the case of the ternary system Al-Cu-Mg, was observed to persist after the cooling of the sample at room temperature.

(iv) The addition of Ag to Al-Cu-Mg alloy decreases the effective activation energy governing the kinetics of aging below 70 °C from 0.65 to 0.22 eV. The effect can be explained by assuming that the diffusing species that dominates the kinetics (as seen by PAS, i.e., filtered through a special sensitivity to the vacancies) is the complex vacancy-Mg-Ag aggregates.

The above points (iii) and (iv), taken together, are in accordance with the observations by APFIM (Ref. 6) and by 3D-AP (Ref. 8) of early co-clustering of Mg and Ag, and give an independent support to the hypothesis that these co-clusters may act as precursors for the precipitation of the Ω phase.

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