POSITRON-LIFETIME INVESTIGATION OF PRECIPITATION EFFECTS IN AI-Cu ALLOY

O. Melikhova¹, J. Čížek¹, I. Procházka¹, J. Kuriplach¹, I. Stulíková¹ and J. Faltus²

¹Department of Low-Temperature Physics, Faculty of Mathematics and Physics, Charles University, V Holešovičkách 2, CZ-180 00 Prague 8, Czech Republic.
²Research Institute of Metals, Panenské Břežany 50, CZ-250 70 Odolená Voda, Czech Republic.

Abstract

Precipitation effects in Al-2.0 at.% Cu alloy were investigated by means of positron-lifetime (PL) spectroscopy. PL spectra were well fitted by two exponential components (lifetimes τ_1 , τ_2) for all isochronal annealing temperatures. The lifetime of trapped positrons lies between the lifetime 180 ps for Cu monovacancy and 250 ps for Al monovacancy. Up to 240 °C increasing of number of Cu atoms in vicinity of vacancy takes place. Formation of semicoherent θ° precipitates begins from 260 °C. Phase transformation of θ° to θ starts at temperature 320 °C. Above 400 °C dissolution of θ precipitates occurs. Results are discussed within LMTO calculations of positron lifetimes and affinities for θ° and θ precipitates.

Keywords

positron-lifetime spectroscopy, Al-Cu alloy, precipitation effects

1. Introduction

The development of new workable aluminium-based alloys is one of the topics in current materials science of light alloys. Most of wrought aluminium alloys are based on Al-Cu alloy. Partially due to the above reason there is, in the current state-of-art, detailed knowledge about phase transitions which take place in Al-Cu alloy (see e.g. [1-3]). Although the alloy is composed of only two elements, its microstructure evolution is relatively complex. On the basis of many studies [2-9], it has been proposed that the decomposition sequence in this system contains the following processes:

Supersaturated Solid Solution (SSS) \Rightarrow GP1 zones \Rightarrow GP2 zones (or θ '') $\Rightarrow \theta$ ' $\Rightarrow \theta$

Although the decomposition sequence is well established by number of studies, several open questions still remain.

The main purposes of investigation of Al-Cu alloy performed in the present work can be summarized as follows:

(i) to correlate positron-lifetime (PL) results, obtained at various annealing temperatures, with TEM and electrical resistivity results available in literature in order to attribute unambiguously lifetime components of trapped positrons resolved in PL spectra to corresponding defects in specimen,

(ii) to clarify behavior of the specimen annealed at high temperatures (above 300°C),

(iii) to perform theoretical calculations of positron lifetimes and affinities for precipitates, formed during decomposition, in order to decide whether positron trapping inside these precipitates is possible, and

(iv) to get quantitative information about stability of microstructure of Al-Cu alloy quenched from variety of annealing temperatures to room temperature, at which the PL measurement is performed.

2. Experimental Details

The PL spectrometer used in the present work was equipped with coincidence system representing a modification of our fast-slow configuration described in detail in [10]. Its detector part consisted of two BaF₂ cylinder scintillators (Ø 25x10 mm) coupled to PHILIPS XP2020/Q photomultipliers (PMT). The dynode timing signal from 10th dynode of PMT passes through the capacitor to the input of the constant-fraction differential discriminator (CFDD), which provides also the coarse energy selection of events. Output timing signals from CFDDs activate the start and stop inputs of the time-toamplitude converter (TAC). The energy signals from 9th dynode of PMT are first shaped by a passive RC-integration circuit and then passively summed. The sum of signals is amplified by the spectroscopy amplifier whose output is connected with timing single-channel analyzer (TSCA) adjusted at the sum of energies 511 keV $(\text{stop photon}) + 1274 \text{ keV} (\text{start photon}) \text{ in the case of }^{22}\text{Na}$ positron source. The output pulses from TSCA strobe the signal from the TAC. The TAC output signal amplitude is digitized by the analog-to-digital converter (ADC) and is stored in the computer. The complete electronics is built from commercially available modules produced by ORTEC, CANBERRA and C.E.S. The more detailed description of spectrometer was published elsewhere [11].

Face-to-face detector geometry with common symmetry axis was used. A carrier-free ²²NaCl (\approx 1.3 MBq) deposited and sealed between 2 µm thick mylar foils was used as a positron source. At least 9x10⁶ counts were collected in each spectrum. The lifetime spectra were decomposed into the individual components by means a fitting procedure [12] based on the maximum-likelihood principle. Parameters of the time resolution function, composed of three Gaussians, were found in simultaneous fit, with total FWHM being approximately 150 ps.

The investigated Al-2 at.% Cu alloy was prepared from Al 99.9995 at.% and Cu 99.995 at.% in Research Institute of Metals, Panenské Břežany. The resulting concentration of Cu in the alloy was determined as 2.05 at.% (4.7 wt.%) by the mass spectroscopy and chemical analysis. The disk-shaped (diameter 10mm) polycrystalline specimens for PL measurements were prepared from the 1mm thick sheet.

The specimens were solution heat treated at 525 °C for 30 min in a vertical furnace with argon protective atmosphere and fast cooled in water. The quenched specimens were stored into liquid nitrogen till the PL measurement.

Subsequently, the specimens were subjected to isochronal annealing with effective heating rate of 1 K/min in steps 20 $^{\circ}$ C / 20 min. The heat treatment was performed in silicon oil base from 80 to 250 $^{\circ}$ C and in vertical furnace with argon atmosphere above 250 $^{\circ}$ C. The quenched specimens were stored into liquid nitrogen till the PL measurement.

All PL measurements were performed at room temperature.

3. **Results and Discussion**

Mean positron lifetime $\bar{\tau} = \sum I_i \tau_i$ represents a robust parameter, which is not affected by the number of components and correlation among the fitted parameters constraints considered in individual fits. Thus, firstly we used $\bar{\tau}$ as integral characteristics of PL spectra. Its dependence on annealing temperature is shown in Fig. 1. One can see in the Fig. 1 that the mean lifetime rapidly decreases firstly from initial value of 215 ps to minimum value of about 164 ps at 230 °C. Subsequently $\bar{\tau}$ exhibits increase to a local maximum of about 180 ps at 360 °C. Above 360 °C $\bar{\tau}$ decreases slightly, approximately by 10 ps. Finally above 490 °C $\bar{\tau}$ abruptly increases back to the initial value.

Firstly it should be pointed out that the last annealing temperature was 525 °C, i.e. the same as the temperature of solution heat treatment. As also heating times were the same, it is evident that the mean lifetime of specimen annealed at 525 °C (the last annealing temperature) has *principally* to be the same as that for initial as-quenched (AQ) specimen. Indeed, both these lifetimes are the same in the range of statistical uncertainties, see Fig. 1.

As-quenched specimen

Two components with lifetimes $\tau_2 = 204.9 \pm 0.4$ ps and $\tau_3 = 570 \pm 20$ ps were found in PL spectrum of AQ specimen, i.e. specimen quenched from solution heat treatment temperature of 525 °C. Corresponding intensities are $I_2 = 97.3 \pm 0.2$ % and $I_3 = 2.7 \pm 0.1$ %. Lifetimes of both the components are remarkably higher both than Al and Cu bulk lifetimes (161 ps [13] and 114.5 ps [14], respectively). Thus, all positrons in the AQ specimen annihilate from trapped state in defects.

Lifetime τ_2 of the dominant component is significantly lower than lifetime of positrons trapped in Al monovacancy (250 ps [15]). It means that positrons are not trapped in single Al monovacancies. It is consistent with the fact that single vacancies are not stable in Al at room temperature [16, 17]. There is positive binding energy of

Figure 1. Dependence of the mean lifetime on annealing temperature

about 0.2 eV for vacancies in Al to Cu atoms [18]. Thus, there are the vacancies bound to Cu atoms in AQ specimen, which are stable at room temperature. The lifetime τ_2 lies between the lifetime 180 ps for Cu monovacancy [15] and 250 ps for Al monovacancy [15]. Hence, natural explanation of the component with lifetime τ_2 is contribution of positrons trapped in vacancies bound to Cu atoms. It means that at least one of the twelve nearest neighbour sites of vacancy is occupied by Cu atom. The same conclusion was made also by Krause et al. [19] and recently by Somoza et al. [20]. Krause et al. [19] found lifetime 205 ps, while Somoza et al. [20] 203 ps on AQ Al-Cu specimen. Similarly Čada et al.[21] reported $\tau_2 = 207 \pm 2$ ps. Clearly, all these reported values exhibit good agreement with present investigation.

In addition to the main component with lifetime τ_2 , another component with relatively small intensity 2.5 % was resolved in the AQ specimen. High lifetime $\tau_3 = 570 \pm$ 20 ps of this component indicates that it represents contribution of positrons trapped in larger defect (with open volume higher than a single vacancy), most probably vacancy clusters, which are formed from migrating quenched-in single vacancies unstable at room temperature. It is not clear, whether these clusters are stabilized by Cu atom. However, as this component disappeared for higher annealing temperatures, it seems to be caused by vacancy clusters in Al matrix, which are completely annealed out at 80 °C, see following text. The lifetime 570 ps corresponds to upper limit lifetime of trapped positrons in clusters [22]. Hence, it can be attributed to very large clusters (voids) with diameter above 1 nm.

No contribution of free positrons was found for the as-quenched specimen. Thus, it is not possible to determine concentration of vacancies bound to Cu atoms in the specimen. However, a lower limit of vacancy concentration above which only signal from trapped positrons is found in PL spectrum, i.e. lower limit of actual vacancy concentration, can be estimated. In this simple estimation, the weak component with lifetime τ_3 , is neglected. Using simple trapping model (STM) [23] relative intensity of the vacancy component is



$$I_2 = \frac{K_v}{\lambda_B + K_v - \lambda_v},\tag{1}$$

where λ_B , λ_v represent the bulk annihilation rate and the annihilation rate of positrons trapped in vacancies (bound to Cu atoms, i.e. $\lambda_v = 1 / 205 \text{ ps}^{-1}$), respectively, and K_v is positron trapping rate to vacancies. One can consider $I_2 = 95$ % as a reasonable limit above which no free-positron contribution is resolved in PL spectrum. For our case this limit value corresponds to the trapping rate $K_v = 1.3 \times 10^{11} \text{ s}^{-1}$. Concentration of vacancies c_v is directly connected with the trapping rate

$$c_{v} = K_{v} / v_{v}, \qquad (2)$$

where v_{ν} is the specific positron trapping rate for vacancy. For Al monovacancy $v_v = 2.5 \times 10^{14}$ at. s⁻¹ [23], while for Cu monovacancy $v_v = 1.2 \times 10^{14}$ at. s⁻¹ was reported [23]. Positrons in the studied specimen are trapped in vacancies bound to Cu, which clearly does not correspond to single Al monovacancy nor Cu one. As rough estimation of actual specific trapping rate for vacancies bound to Cu atoms we used weighted average of the two values given above. As weights we took the values η and $l-\eta$, which tune the lifetime of Al and Cu monovacancy to the observed lifetime τ_2 , i.e. $\tau_2 = 250\eta + 180(1-\eta)$. From the latter equation $\eta = 0.6$ was obtained, which leads to estimated specific trapping rate $v_v \approx 1.7 \times 10^{14}$ at. s⁻¹ for Al vacancies bound to Cu atoms. Hence, the estimation of lower limit of concentration of vacancies bound to Cu atoms in the AQ specimen is $c_v \approx 1.5 \times 10^{-4}$ at ⁻¹. Note that concentration of thermal equilibrium vacancies at solution heat treatment temperature of 525 °C is 4.3×10^{-4} at.⁻¹. It means that at least 25% of thermal equilibrium vacancies at solution heat treatment temperature remain quenched in the specimen. Similarly to our case, saturation trapping of positrons were found in AQ (solution heat treatment temperature 510 °C) Al-2at.%Cu specimen by Čada et al. [21]. On the other hand, Krause et al. [19] reported substantially lower concentration of 0.7×10^{-4} at.⁻¹ in AQ (solution heat treatment temperature 540 °C) Al-1.9%Cu specimen. It is probably a consequence of lower quenching rate in [19].

No evidence of decomposition was found in AQ (solution heat treatment temperature 540 °C) Al-1.7at.%Cu specimen by strong two-beam bright field microscopy as well as beam dark field microscopy [24]. The diffraction patterns consisted only of circular fundamental reflections. No satellites or streaks were detected [24]. Strain modulations, which are present because of composition modulations with wavelength varying from 4 to 8 nm were found after 5 hour aging at room temperature [24]. After aging for one week at room temperature 3.0 nm GP zones were observed [24].

On the basis of the results mentioned above we can conclude that no GP zones are present in our AQ specimen.

Stability of the AQ microstructure at room temperature, i.e. temperature of PL measurement, is of high importance, as one measurement of PL spectrum takes



Figure 2. Time dependence of the lifetime for the Al-Cu aged at room temperature. The PL spectra were recorded in 30 min steps. PL spectrum for each step was fitted independently.

usually two or three days. Therefore, we performed test of stability of the AQ microstructure at time scale of a few days. The AQ specimens were measured (immediately after quenching) by PL spectroscopy at room temperature. The obtained PL spectrum was recorded after each 30 min. Total time of measurement was 100 hours. Each partial PL spectrum (i.e. spectrum of each 30 min step) was subsequently decomposed independently. Due to limited statistics the spectra were characterized only by one lifetime component. Dependence of the lifetime of this component on time for the AQ specimen is shown in Fig. 2. Clearly, the lifetime exhibits no changes except of statistical fluctuations. Hence, we conclude that microstructure of the AQ specimen is stable at time scale at least 50 hours.

In order to test influence of rapid quenching rate to microstructure of AQ specimen, another specimen was homogenized 30 min at 525 °C and then slowly cooled in furnace. Subsequent PL measurement of this specimen revealed single component spectrum with lifetime 165 ± 2 ps, which corresponds to the Al bulk lifetime (161 ps [13]). Thus, all positrons annihilate from free state and no defects detectable by PL spectroscopy are present. It means that large precipitates of equilibrium θ phase were most probably formed during the slow cooling, distance between them is too large to obtain any measurable contribution in PL spectrum.

Isochronal annealing

All PL spectra of isochronaly annealed specimens were well fitted by two exponential components (except of the source contribution). The lifetimes τ_1 and τ_2 of the spectral components are plotted in Fig. 3 as a function of annealing temperature.

One can see in the Fig. 3 that the lifetime τ_1 of the shorter component lies for all annealing temperatures below the Al bulk lifetime (161 ps [13]). Hence, the first component with lifetime τ_1 represents contribution of free positrons, while the second component comes from positrons trapped at defects. Temperature dependence of



Figure 3. Temperature dependence of lifetimes τ_1 and τ_2



Figure 4. Temperature dependence of relative intensity of I_2 of trapped positrons component

relative intensity I_2 of the defect component is shown in Fig. 4.

The component with lifetime $\tau_3 = 570$ ps present in the AQ specimen was not found at higher annealing temperatures except of the last annealing temperature 525 °C, which equals to the temperature of solution heat treatment.

Both lifetime τ_2 and relative intensity I_2 decrease with annealing temperature in the temperature region 20 -240 °C. The decrease of τ_2 is an indication of increasing number of Cu atoms surrounding the quenched vacancies. At the same time decrease of relative intensity I_2 reflects diminishing concentration of vacancies. Clearly, when a vacancy is surrounded only by Cu atoms, i.e. all twelve nearest-neighbour sites are occupied by Cu atoms, then lifetime of trapped positrons is 180 ps (lifetime of positrons trapped at Cu monovacancy, indicated by dotted line in Fig.3). In our case τ_2 goes to value 170 \pm 4 ps, which is close to 180 ps, see Fig.3. Thus, we conclude that Cu atoms form small clusters with increasing annealing temperature. As a result quenched vacancies are at temperature 240 °C surrounded exclusively by Cu atoms. Similar conclusion was done also by Krause et al. [19] and Somoza et al. [20].

a)





Figure 5. Diffraction pattern a) and bright field image b) at [001] zone axis orientation obtained by TEM on Al-2 at. % Cu annealed at 170 °C. Reprinted from [26].

It is possible to calculate concentration of quenched vacancies as a function of annealing temperature by using of STM [23]. We assume that the vacancies are distributed homogeneously throughout the specimen. The concentration of vacancies can be then expressed as

$$c_{v} = \frac{1}{v_{v}} \frac{I_{2}}{I_{1}} (\lambda_{B} - \lambda_{2}),$$
 (3)

where the specific trapping rate v_{ν} was estimated by the same way as described above. At 160 °C c_{ν} decreases to 1.3 × 10⁻⁵ at.⁻¹, i.e. roughly by factor of 10. Krause et al. [19] found also decrease of vacancy concentration by the same factor from AQ state to 150 °C.

The Cu clusters formed during isochronal annealing represent embryonic regions for further formation of GP zones [25]. PL spectroscopy is sensitive to local chemical surrounding of vacancies, however it has no sensitivity to crystallography, i.e. it is not possible to determine when (at which annealing temperature) GP zones are formed. Thus, it is necessary to correlate PL results with other techniques, preferentially TEM, which can unambiguously detect presence of GP zones in specimen.

© Krystalografická společnost

a)







Figure 6. TEM diffraction pattern a) and dark field image b) at [001] zone axis orientation on Al-2 at.% Cu annealed at 200 °C. Reprinted from [26]

Therefore, we used TEM and electrical resistivity results obtained on Al-Cu of the same composition by Čada [26]. Formation of GP1 zones was observed by TEM at temperature 170 °C [26]. It is indicated by streaks in diffraction pattern for [001] zone axis, which is shown in Fig. 5. A mixture of GP1 and thicker GP2 zones was observed by TEM in specimen annealed at 200 °C [26]. Presence of GP2 zones is confirmed also by appearance of central reflections in diffraction pattern. Bright field TEM image of specimen annealed at 200 °C together with diffraction pattern for [001] zone axis reprinted from [26] are shown in Fig. 6. On the basis of the TEM results we conclude that vacancies bound to Cu atoms are incorporated to GP zones at higher annealing temperatures (above 170 °C). Continuously with clustering of Cu atoms and subsequent formation of GP zones, number of vacancies gradually decreases, which is reflected by decrease of relative intensity I_2 . Vacancies bound to Cu atoms enhance their mobility and thereupon allow formation of GP zones [18]. Vacancies incorporated to GP zones represent, however, non-equilibrium defects, which





Figure 7. TEM diffraction pattern a) and dark field image b) at [001] zone axis orientation on Al-2 at.% Cu annealed at 260 °C. Reprinted from [26]

are gradually annealed out even at room temperature. It was experimentally observed by Somoza et al. [20] during ageing of Al-1.7at.%Cu at various temperatures. Concentration of vacancies decreased with ageing time and finally it fell below sensitivity limit of PL spectroscopy (about 4×10^{-7} at.⁻¹ for Al) and the aged specimen exhibited only single PL component, which comes from free positrons. It means that after long ageing times, vacancies are released from GP zones and GP zones free of vacancies are present in specimen. Formation of semicoherent θ ' precipitates was observed by TEM at temperature 260 °C [26]. Bright field TEM image for [001] zone axis of specimen annealed at 260 °C is shown in Fig. 7. Formation of θ ' precipitates is reflected also by substantial decrease of electrical resistivity [26]. Formation of θ ' precipitates causes slight increase of lifetime τ_2 , see Fig. 3, as well as noticeable decrease of relative intensity I_2 in temperature region 240-350 °C, see Fig. 4. This slight change of lifetime τ_2 suggests change of type of positron trapping sites. Vacancies are likely completely annealed out at the annealing temperature of 240 °C and positrons are trapped at misfit defects at θ ' precipitate-matrix interface. Clearly,

the lifetime of positrons trapped at the misfit defects is very close to lifetime of positrons trapped in Cu vacancy (180 ps [15]). Phase transformation of θ ' to θ starts at temperature 320 °C [26]. In this temperature interval incoherent spherical precipitates of equilibrium θ phase (Al₂Cu) are formed. It is reflected by remarkable increase of lifetime τ_2 , see Fig. 3. As θ precipitates are incoherent with Al matrix, positrons are trapped at precipitate-matrix interface. One can see in Fig. 3 that lifetime τ_2 of positrons trapped at θ precipitate-matrix interface is remarkable higher then lifetime of positrons trapped at misfit defects around θ ' ones.

Rapid increase of electrical resistivity starts at temperature 400 °C [26]. It gives strong evidence of the onset of dissolution of θ precipitates. The dissolution of θ precipitates is reflected by radical decrease of relative intensity I_2 in our PL spectra. Clearly, it is due to decreasing number of positron traps at θ precipitate-matrix interfaces. On the other hand, lifetime τ_2 of trapped positrons remains unchanged because there is no change of type of traps. Thus in temperature region 360-490 °C dissolution of θ precipitates takes place.

We have assumed that positrons are trapped at interfaces between precipitates and matrix. It is indicated by lifetime τ_2 , which is substantially higher than Cu and Al bulk lifetimes and simultaneously lower than the lifetime of positrons trapped in Al vacancy. In order to check the possibility of positron trapping also *inside* the precipitates, we performed theoretical calculations, which are described in the next section.

Theoretical Calculations

It is well known that positrons may be trapped inside a precipitate of different phase [23]. The trapping of positrons inside a precipitate is possible if the following two conditions are fulfilled.

Absolute value of positron affinity A_p^+ ($A_p^+ < 0$) for a precipitate has to be higher than absolute value of the positron affinity A_m^+ ($A_m^+ < 0$) for matrix, i.e. $|A_p^+| > |A_m^+|$. It is convenient to introduce difference between positron affinity for matrix and precipitate $\Delta A^+ = A_m^+ - A_p^+$. The affinity difference ΔA^+ corresponds to difference between positron ground state energy in matrix and inside precipitate.

If we assume a spherical-shape precipitate, which can be considered as a spherical potential well, then minimum precipitate radius r_p^{\min} for possibility of positron trapped state [23]

$$r_p^{\min} = 3.1/\sqrt{\Delta A^+},\tag{4}$$

where the affinity difference ΔA^+ ($\Delta A^+ > 0$) is expressed in eV and the minimum precipitate radius in angstroms.

It should be noted that positrons can be trapped also at precipitate-matrix interface (in the case of incoherent or semicoherent precipitates, see previous section) or at open volume defects inside precipitates independently whether the above conditions are satisfied or not. In the present work we have calculated positron lifetimes and affinities for θ ' and θ precipitates. So-called zero positron density limit [27, 28] was adopted in the calculations of positron properties.

The results of calculations, i.e. positron affinity A and bulk lifetime τ_B for Al, θ ' and θ phase, are shown in Tab.1. The results obtained using Boronski-Nieminen (BN) approach [29] are given on the left side of Tab. 1, while results calculated in the frame of generalized gradient approximation (GGA) [30] are shown on the right side. One can see that except of the bulk lifetime for Al (difference 10 ps between BN and GGA approach) the results given by BN and GGA are very similar.

It is clear from Tab. 1 that positron affinity for θ phase precipitates is the same as positron affinity for Al matrix. It means that θ phase precipitates do not represent attractive sites for positrons. Hence, positron trapping occurs only at θ precipitate-matrix interface and not inside the θ precipitates. It is indicated also by lifetime of positrons inside θ precipitates being substantially lower than that observed in experiment, see Fig. 3.

Table 1.

Positron affinities A^+ and bulk lifetimes τ_B calculated for Al as well as θ' and θ phase using linear muffin-tin orbital (LMTO) technique within atomic-sphere approxiamtion (ASA) [31]. The enhancement factor due to electron-positron correlation was approximated using formula introduced by Boronski and Nieminen (BN) as well as using generalised gradient approximation (CGA).

| System | A ⁺ [ev] (BN) | τ _B [ps] (BN) | A ⁺ [ev] (GGA) | τ _B [ps] (GGA) |
|--------|-----------------------------|-----------------------------|------------------------------|------------------------------|
| Al | -4.8 | 163 | -4.6 | 153 |
| θ' | -6.1 | 174 | -5.7 | 174 |
| θ | -4.8 | 145 | -4.6 | 143 |

On the other hand, θ' precipitates exhibit remarkably higher absolute value of affinity and represent, therefore, attractive sites for positrons. The minimum radius of θ' precipitate necessary for creation of trapped state is $r_p^{\min} =$ 3.4 Å. Lifetime of positrons inside θ' precipitates is 174 ps, which is close to that experimentally observed between annealing temperature 250 and 300 °C, where θ' phase is present in specimen. Thus, we conclude that positrons are trapped at misfit defects on incoherent boundary of θ' phase and/or directly inside the θ' precipitates.

4. Conclusions

The results obtained in investigations of decomposition of Al-Cu alloy performed in the present work can be summarized into following items:

(i) Vacancies bound to Cu atoms are present in specimen rapidly quenched from solution heat treatment temperature. Small fraction of very large vacancy clusters was also found.



67

(ii) No noticeable change of microstructure of asquenched specimen occurs at time scale of 50 hours.

(iii) Cu atoms tend to cluster with increasing annealing temperature. In such way number of Cu atoms surrounding the quenched-in vacancies increases. Simultaneously concentration of the quenched-in vacancies gradually decreases due to thermally activated migration to sinks.

(iv) Formation of semicoherent θ ' precipitates occurs at temperature 240 °C. Positrons are trapped at misfit defects introduced into specimen due to formation of θ ' precipitates and/or directly inside the θ ' precipitates. The θ ' precipitates represent attractive sites for positrons, lifetime of positrons inside them is 174 ps. Experimental lifetime of trapped positrons in temperature region, in which θ ' phase is present in specimen lies between 170 and 180 ps.

(v) At 350 °C incoherent spherical precipitates of equilibrium θ phase are formed. Positron trapping does not occur inside the θ precipitates. Positrons are trapped at precipitate-matrix interface and annihilate with lifetime ≈ 220 ps. Above 400 °C dissolution of θ phase precipitates takes place.

(vi) Finally the specimen annealed at $525 \,^{\circ}$ C (solution heat treatment temperature) exhibits the same microstructure as the initial as-quenched specimen.

Acknowledgements

This work was supported by Grant Agency of the Czech Republic (contract No.106/00/1047) and Ministry of Education, Youth and Sports of Czech Republic within European program COST (project OC 523.70).

References

- R. Schülbe & U. Schmidt, *phys. stat.sol. (a)* **103**, (1987) 29.
- J.M. Silcock, T.J. Heal, & H.K. Hardy, J. Inst. Met. 82, (1954) 239.
- 3. V.A. Phillips, Acta metall. 21, (1973) 219.
- 4. A. Guinier, Solid State Phys. 9, (1959) 293.
- 5. T. Abe, K. Miyazaki & K. Hirano, *Acta Metall.* **30**, (1982) 357.
- R.B. Nicholson, G. Thomas & J. Nutting, J. Inst. Met. 87, (1958) 429.
- 7. M. Wada, H. Kita & T. Mori, Acta Metall. 33, (1985) 1631.
- 8. K. Hono, T. Satoh & K. Hirano, *Philos. Mag.* **53A**, (1986) 495.
- 9. J.L. Murray, Int. Met. Rev. 30, (1985) 211.

- F. Bečvář, L. Lešt'ák, I. Novotný, I. Procházka, F. Šebesta & J. Vrzal, *Mater. Sci. Forum* **175-178**, (1995) 947.
- F. Bečvář, J. Čížek & I. Procházka, Acta Phys. Pol. A 95, (1999) 448.
- I. Procházka, I. Novotný & F. Bečvář, *Mater. Sci. Forum* 255-257, (1997) 772.
- J. Čížek, I. Procházka, T. Kmječ & P. Vostrý, *phys. stat.* sol. (a) 180, (2000) 439.
- J. Čížek, I. Procházka, P. Vostrý, F. Chmelík & R.K. Islamgaliev, *Acta Physica Polonica A* 95, (1999) 487.
- 15. A. Seeger & F. Banhart, phys. stat. sol. (a) 102, (1987) 171.
- T. Federoghi, S. Ceresara & F. Pieragostiny, *Philos. Mag.* 12, (1965) 1093.
- 17. S. Mantl & W. Triftshäuser, Phys. Rev. B 17, (1978) 1645.
- 18. H. Kimura & R.R. Hasiguti, Acta Mettal. 9, (1961) 1076.
- R. Krause, G. Dlubek, G. Wendrock & O. Brümmer, in: *Proc. of European Meeting on Positron Studies of Defects, Wernigerode 1987*, eds. G. Dlubek, O. Brümmer, G. Brauer, K. Hennig, Martin-Luther-Universität, Halle-Wittenberg, p. F1.
- 20. A. Somoza, A. Dupasquier, I.J. Polmear, P. Folegati & R. Ferragut, *Phys. Rev. B* **61**, (2000) 14464.
- P. Čada, M. Cieslar, P. Vostrý, F. Bečvář, I. Novotný & I. Procházka, *Acta Physica Polonica A* 88, (1995) 111.
- 22. M.J. Puska & R.M. Nieminen, J. Phys. F: Met Phys. 13, (1983) 333.
- P. Hautojarvi & C. Corbel in: *Proceedings of the International School of Physics "Enrico Fermi"*, Course CXXV, Ed. A. Dupasquer, A.P. Mills, IOS Press, Amsterdam 1995, 491.
- 24. R.J. Rioja & D.E. Laughlin, Met. Trans. 8, (1977) 1257.
- 25. E. Matsubara & J.B. Cohen, Acta Metall. 31, (1983) 2129.
- P. Čada, Studium fázových transformací ve slitině Al-Cu elektrickou rezistometrií a anihilací pozitronů (diploma thesis), MFF UK, Praha (1994) in Czech.
- R.M. Nieminen, in: Proceedings of The International School of Physics 'Enrico Fermi' Course CXXV, eds. A. Dupasquier, A.P. Mills, IOS Press, Amsterdam 1995, p. 443.
- M.J. Puska & R.M. Nieminen, *Rev. Mod. Phys.* 66, (1994) 841.
- E. Boronski & R.M. Nieminen, *Phys. Rev. B* 34, (1986) 3820.
- B. Barbiellini, M.J. Puska, T. Torsti & R.M. Nieminen, *Phys. Rev. B* 51, (1995) 7341.
- O.K. Andersen, O. Jepsen & M. Šob, in: *Electronic Band* Structure and Its Applications, ed. M. Yussouff, Springer Verlag, Heidelberg, 1987, p.1

