

Positron Annihilation Lifetime (PAL) Studies of the Variations of Microstructure During Isochronal Annealing of Cu-Zn (60/40) Alloy

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The microstructure variations during isochronal annealing of the as-received and quenched Cu-Zn (60/40) alloy were studied in the temperature range from room temperature range (RT)-1173 K. The experimental information was basically obtained by means of positron annihilation lifetime spectroscopy and Vickers microhardness. The microstructure was studied by optical and scanning electron microscopy observations. The Arrhenius plot of the data produced by the analysis of the positron annihilation spectra according to the simple trapping model is valid only at certain temperatures. Two regions were selected. The first region was at lower temperatures and its activation energy was 0.55 ± 0.02 eV, which may be attributed to the energy of vacancy formation. The second region (at higher temperatures) was characterized by an activation energy (1.23 ± 0.03 eV) that suggested it was related to the activation energy of recrystallization in the bulk material. The analysis of positron lifetime indicated clear evidence of the existence of three-dimensional defects in addition to one-dimensional defects, thus it needs a modification to the simple trapping model to consider the inhomogeneous trapping.

Keywords Cu-Zn alloys, phase transformation, positron annihilation lifetime, recrystallization, three-dimensional defects, Vickers microhardness

1. Introduction

The typical application of ($\alpha + \beta$) brasses is for the manufacture of chips screws and marine fittings, owing to the high mechanical properties and excellent corrosion resistance of brass.^[1] The heat treatment changes the microstructure of the alloy either by recovery and recrystallization processes or by some part of phase transformation.^[2] The phase transformation, precipitation, and defects are related to the formation and migration of vacancies in the material.^[3] In general, in Cu-based alloys, the β -phase is only stable at high temperatures, but it can be retained in a metastable stage at lower temperatures by means of a fast quench. The formation of α_1 -plates in β -Cu-Zn alloys exhibited a dual nature, i.e., displacement and diffusional characteristics.^[4,5] Wu^[6] concluded that the combined effects of solute depletion and increasing elastic anisotropy lead to the formation of the solute-depleted region for the α_1 shear growth.

In recent years, positron annihilation has become a valuable nuclear method to study the microscopic structure of solids.^[7] It has proved to be a very useful and effective technique for studying defects in Cu-based alloys.^[8] The annihilation characteristics depend on the type of defect in which the positron is

trapped. Therefore, it is possible in many cases to differentiate between different types of defects.

The use of positron annihilation spectroscopy (PAS) for studies of defects in solids is mainly based on two facts.^[9] First, positrons are antiparticles to electrons, so that a positron may annihilate with an electron. An amount of energy, equivalent to the mass of the two particles, will then be emitted simultaneously with the annihilation (normally as gamma radiation) that carries information about the state of the positron-electron pair before annihilation. Second, positrons injected into a material may become trapped in defects and annihilate there (rather than in the bulk material).

Normally, regions of lower-than-average atomic density also have lower-than-average electron density. The lifetime of positrons trapped in defects will depend on the average electron density in the defects (the lower the electron density, the longer the lifetime). Thus, in principle, each type of defect gives rise to a characteristic positron lifetime, τ_i .^[7] If several types of defects are present in a sample, this will therefore result in a lifetime spectrum for the sample that consists of several lifetime components. Each component is a decaying exponential, the slope of which equals the annihilation rate (τ_i^{-1}). The purpose of the data analysis is to separate the various components and to determine their lifetimes, τ_i , and intensities, I_i (i.e., the relative areas under each component in a spectrum). Usually, the lifetimes are numbered from the shorter to the longer lifetimes (τ_1, τ_2, τ_3 , etc.). The intensities are measures of the fractions of positrons that annihilate with the equivalent lifetimes. In practice, it is possible to resolve only two or three lifetime components in a spectrum for a metal. A certain lifetime value is then characteristic for positrons that annihilate either in bulk material, τ_1 (with intensity I_1), or trapped in a certain type of defect, τ_2 (with intensity I_2). The third component, τ_3 (with intensity I_3), represents the formation of orthopositronium, that can be neglected in metal alloys.

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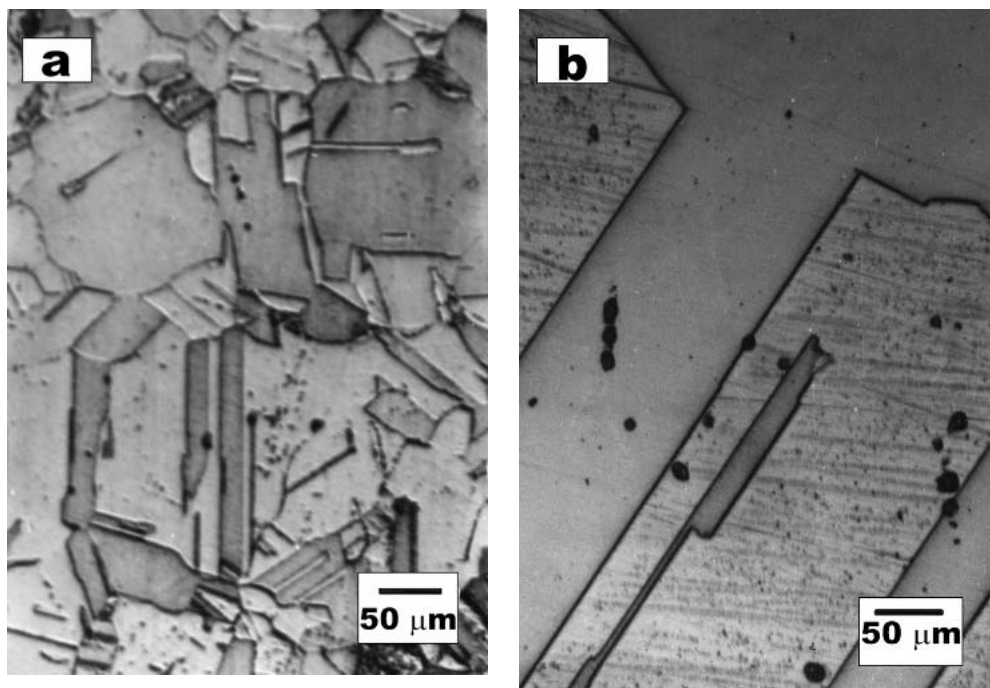


Fig. 1 The microstructure of Cu-Zn (60/40) at RT (optical microscopy): (a) as received, (b) quenched

The purpose of this work was to use the positron annihilation lifetime (PAL) spectroscopy technique to study the microstructure variations with the isochronal annealing of as-received and quenched Cu-Zn (60/40) alloy, in addition to the traditional means (microhardness, and optical and scanning electron microscopy).

2. Experimental

2.1 Materials

The chemical composition of the impurities in Cu-Zn 60/40 alloy used in this investigation (in wt.%) was 0.2 Si, 0.1 Al, 0.25 Sn, 0.2 Mn, 0.3 Ni, 0.3 Fe, and 1.5 to 2 Pb. The as-received material resembles a hot-rolled alloy, whereas the quenched materials were also annealed at 1073 K for 6 h, then rapidly cooled in ice water at 1 °C. The microstructure of the as-received and quenched Cu-Zn 60/40 at room temperature (RT) is shown in Fig. 1. Both were isochronal annealed for 1 h in the temperature range 423-1173 K.

After the specimens were polished, they were etched in a solution of 10 g FeCl₃, 10 cm³ HCl, and 100 cm³ water. The microstructure features were investigated using optical and scanning electron microscopy (SEM).

2.2 Microhardness Measurements

Microhardness measurements were performed for all samples using a microhardness tester (Vickers, Shimadzu, Japan) with applied load of 50 g for 10 s. Ten readings were taken for each sample, and the standard deviations were calculated.

2.3 Positron Annihilation Lifetime Spectroscopy (PAL)

In this technique, the time delay between the annihilation photon (511 keV) and a photon that is simultaneously emitted

with the positron is measured by means of a fast-fast time delay coincidence system. A time resolution of 200 picosecond (ps) full width at half maximum (FWHM) for ⁶⁰Co was obtained. Spectra were recorded at 550 counts per second (cps) with 20 microcuries (μCi) ²²Na source deposited on Kapton foil, then sandwiched between two similar samples, each 1 mm thick. The complex lifetime spectrum consists of a sum of decaying exponentials convoluted with the resolution function $R(t)$ of the spectrometer and a constant background β , such that the measured spectrum is represented by the general formula

$$y(t) = R(t) \sum_i \left[\frac{I_i}{\tau_i} \exp\left(-\frac{t}{\tau_i}\right) + \beta \right] \quad (\text{Eq 1})$$

where i is the number of lifetimes and τ_i the mean lifetime of the i th component with intensity I_i . To solve this equation, the lifetime spectra were analyzed into three components using a computer program PATFIT-88 (Risø National Laboratory, Roskilde, Denmark). The spectra were corrected for 3% contribution from the source and the variance of fit ranged from 0.94 to 1.25. From the PATFIT program analysis, the lifetimes of positrons in the free and trapped state, τ_1 and τ_2 , and their intensities, I_1 and I_2 , respectively, were obtained and were equal to

$$\tau_1 = (\lambda_b + K)^{-1}, \tau_2 = \lambda_d^{-1} \quad (\text{Eq 2})$$

where K is the trapping rate and λ_b and λ_d are the annihilation rates in the bulk ($\lambda_b = [1/\tau_b]$) and the defect, respectively. The relative intensities are

$$I_1 = 1 - I_2, I_2 = K (\lambda_b - \lambda_d + K)^{-1} \quad (\text{Eq 3})$$

From the lifetimes and intensities, the average (τ_{av}) and bulk (τ_b) lifetimes were calculated:

$$\tau_{av} = I_1\tau_1 + I_2\tau_2, \tau_b = \left(\frac{I_1}{\tau_1} + \frac{I_2}{\tau_2}\right)^{-1} \quad (\text{Eq 4})$$

The average lifetime is insensitive to uncertainties in the decomposition and should coincide with the center of mass of the spectrum.

In the simple trapping model,^[10] where only one type of defect is present and a trapped positron does not escape from the defect, the lifetime spectrum is two-exponential. The positron trapping rate K is given by

$$K = \frac{I_2}{I_1} (\lambda_b - \lambda_d) \quad (\text{Eq 5})$$

If there is more than one type of positron trap present in the lattice, the analysis with more than two decay components is often not possible because of the high number of free parameters. However, in a two-component fit, the longest lifetime τ_2 is usually clearly separated, whereas the shorter lifetime τ_1 becomes a superposition of the other decay components. In this case, an equation of the simple trapping model can be used to check whether the analysis is compatible with one defect type only. If only one type of trap is present and no detrapping occurs, the test lifetime τ_1 is calculated from equations of the simple trapping model using the experimental values of τ_{av} and τ_1 . If the experimental values are higher, then τ_1 is due to annihilations of both free positrons and those trapped at additional defects in the lattice.

According to the simple trapping model, the trapping rate K is proportional to the concentration of the defects C_d , such that

$$K = \mu_d C_d \quad (\text{Eq 6})$$

where μ_d is the specific trapping rate (constant). From an analysis of the positron trapping rate at vacancies in thermal equilibrium, K is given by

$$K = \mu_v C_v = \mu_v \exp(S_v^f/k) \exp(-Q_v^f/kT) \quad (\text{Eq 7})$$

where S_v^f denotes the vacancy formation entropy, Q_v^f is the value of the vacancy formation enthalpy, k is Boltzmann constant, and T is the absolute temperature. Because $\mu_v \exp(S_v^f/k)$ is constant, Eq 7 can be reduced to the following formula:

$$K = A \exp(-Q_v^f/kT) \quad (\text{Eq 8})$$

According to the Arrhenius plot ($\ln K - 1/\tau$), this will give a straight line, the slope of it will give Q/k . Thus the activation energy of the process can be calculated.

3. Results and Discussion

The variations of microhardness measurements (H_v) and the average positron lifetimes (τ_{av}) during isochronal annealing of Cu-Zn (60/40) (as received and quenched) are shown in Fig. 2.

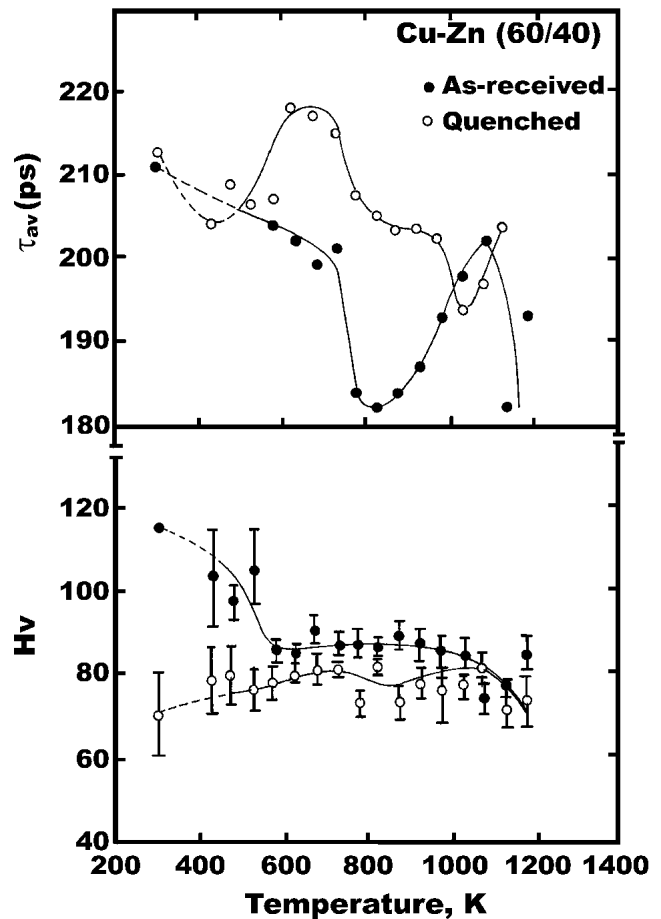


Fig. 2 Influence of isochronal annealing of Cu-Zn (60/40) (as received and quenched) on Vickers microhardness (H_v) and positron average lifetime (τ_{av}) (The error bars of τ_{av} are too small to show the curve.)

It appears that the trend of H_v with temperature is not similar to τ_{av} ; also, the behavior of the two cases is different. The analysis of the results can be divided into four different regions of temperatures: (a) from 423-573 K, (b) from 623-773 K, (c) from 823-1023 K, and (d) from 1073-1173 K.

The first region, which is characterized by a decrease of both H_v and τ_{av} for the as-received specimens and an increase of H_v and τ_{av} for the quenched specimens may be attributed to recovery and vacancy formation, respectively. For the as-received specimens (hot rolled [$\alpha + \beta$]), a recovery stage is expected at temperatures at which the vacancies become mobile. However, for the quenched specimens, little climb and rearrangement of dislocations can take place.^[2] Moreover, if a metal is cooled rapidly from a high quenching temperature (T_Q) to a low temperature, the vacancies at T_Q are quenched in and can thus be observed at the lower temperature.

The second region indicates more or less a stability of H_v and a decrease of τ_{av} for both specimens, which means the same type of activation process (may be the beginning of recrystallization). In the third region, a tendency of decrease in H_v and rapid increase of τ_{av} is noted for the as-received specimens, whereas a tendency of increase in H_v and a decrease of

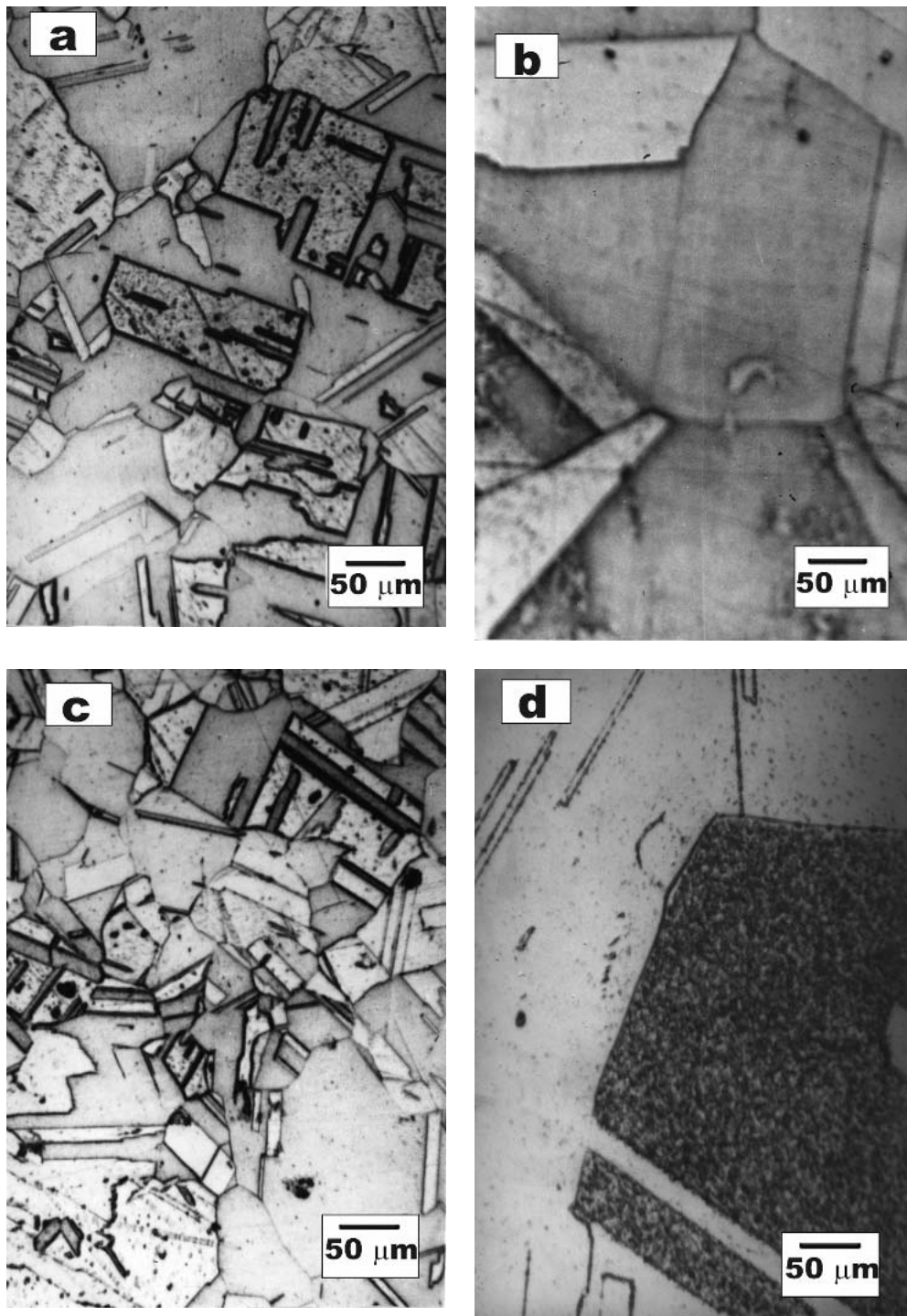


Fig. 3 The microstructure of Cu-Zn (60/40) at different temperatures (optical microscopy): (a) 523 K (as received), (b) 523 K (quenched), (c) 923 K (as received), and (d) 923 K (quenched)

τ_{av} is observed for the quenched specimens. The fourth region shows an increase of H_v and slight increase of τ_{av} followed by a sudden decrease of H_v and increase of τ_{av} for the quenched specimens, whereas a decrease of H_v and increase of τ_{av} for the as-received specimens is observed.

It was found before that for 20.2 wt.% beta-Cu-6.6 wt.%

Al-0.7 wt.% Co (Cu-Zn Al Co alloy) aged at different temperatures and times, the presence of the martensite or the β -phase on aging involves a decrease in the hardness, until it reaches nearly the original value.^[11] It appears that in the third and fourth regions, two processes are opposing each other in the two specimens (recrystallization and phase transformation).

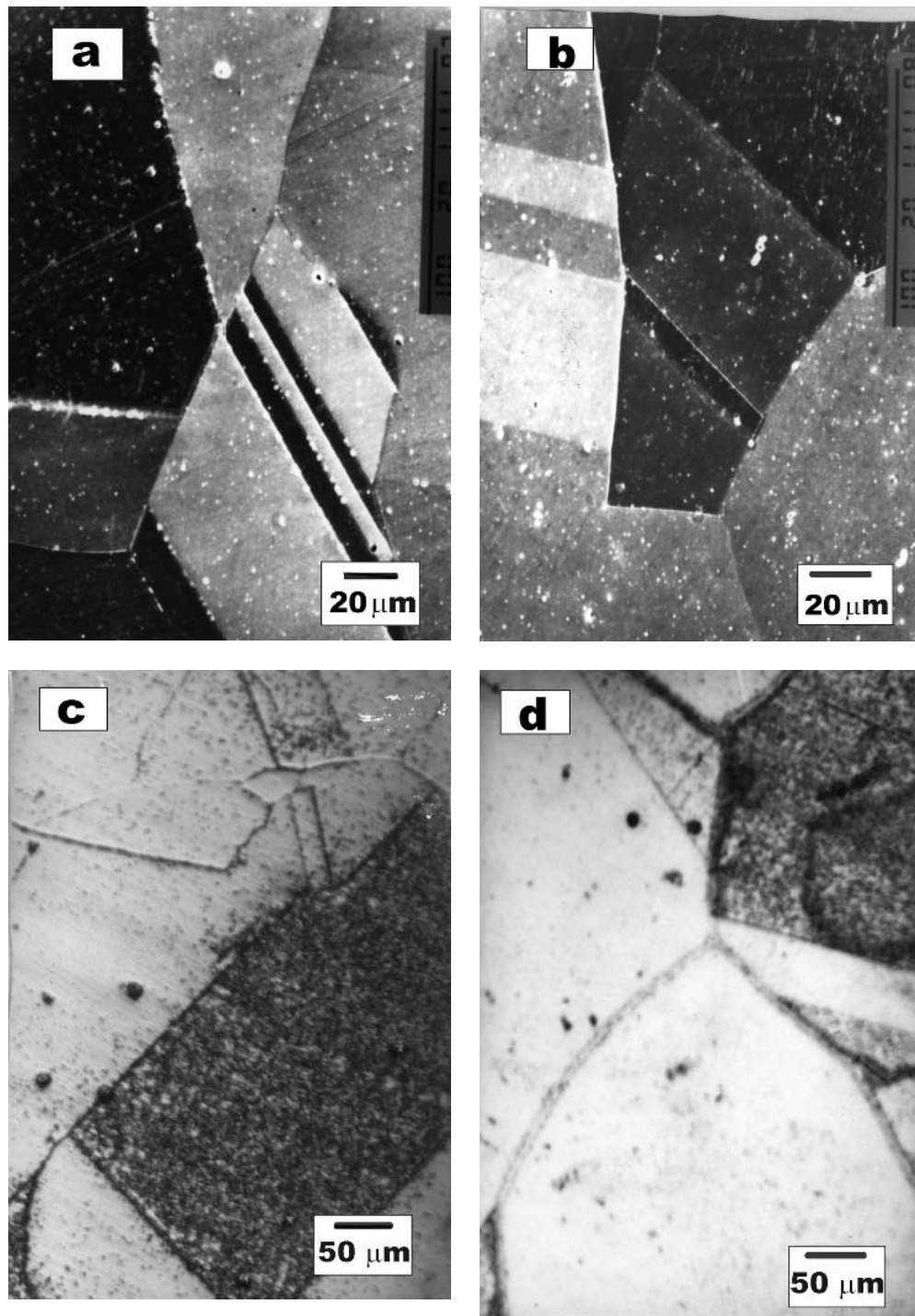


Fig. 4 Scanning electron (a, b) and optical microscopy (c, d) observations of Cu-Zn (60/40): (a) (as received) at 1073 K, (b) (quenched) at 1073 K, (c) (as received) at 1123 K, and (d) (quenched) at 1123 K

In addition, the decrease of H_v observed for the as-received specimens in the third region and in the fourth region for the quenched specimens may be due to the presence of martensite or the β -phase, as observed before for β Cu-Zn Al Co alloy.^[11]

Referring to the phase diagram of Cu-Zn alloy,^[11] we can conclude that the microstructure variations with temperature for the as-received Cu-Zn (60/40) is as follows: (a) from RT

(Fig. 1a)-723 K ($\alpha + \beta$), (b) from 723-1073 K ($\alpha + \beta$), and (c) from 1073-1173 K (β). The sequence features of the quenched Cu-Zn (60/40) are (a) β at RT (Fig. 1b) and partial transformation from β to α phase up to 723 K, (b) from 723-1073 K ($\alpha + \beta$), and (c) from 1073-1173 K (β). Our results on the microstructure of both specimens, using optical and SEM, are shown in Fig. 3 and 4.

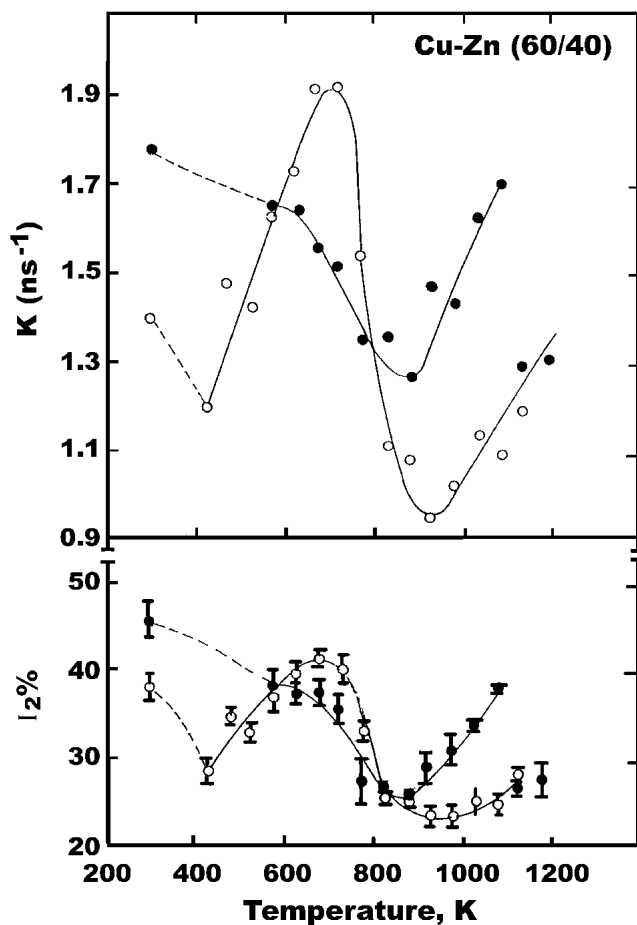


Fig. 5 Influence of isochronal annealing of Cu-Zn (60/40) on the trapping rate (K) and intensity ($I_2\%$) of the long lifetime (τ_2). (The error bars of K are too small to show in the curve.)

Figure 5 shows the influence of isochronal annealing on the trapping rate (K) and the intensity ($I_2\%$) of the long lifetime (τ_2) for Cu-Zn (60/40) specimens. The trend of K and I_2 with temperature is the same for both specimens. It appears that the lines of the two curves are crossed at certain temperatures, and the majority of the K and I_2 values are higher for the quenched specimens in the temperature range 573–773 K, whereas K and I_2 values of the as-received specimens are higher at temperatures greater than 773 K. The significant increase in K and I_2 produced at a temperature near the long range order-disorder temperature could be assigned to the formation of divacancies^[12] as was suggested by Kim and Burgers^[13] in their study on β Cu-Zn with Doppler broadening (DB) of positron annihilation measurement. The experimental study by Zemtsova^[14] with Cu₃ Au alloys adopted with 8 and 12 at.% Pd revealed a high density of dislocations in the regions where a diffusion phase transformation (atomic ordering) took place as a result of a discontinuous ordering, which is a complex phenomenon comprising two or three elementary reactions.

The Arrhenius plot of our results ($\ln K - 1/T$) as shown in Fig. 6 does not exhibit the usual linear behavior, and this has been observed before in β Cu-Zn-Al.^[12] We can distinguish three regions at which the Arrhenius plot can be applied; two

and three are parallel to each other. The activation energies deduced at lower and higher temperatures are 0.55 ± 0.02 eV and 1.23 ± 0.03 eV, respectively. In β Cu-based alloys, the vacancy formation energy is estimated to be about 0.5 eV.^[15] The vacancy formation energy obtained for β Cu-Zn-Al alloy by Somoza et al.^[12] is 0.57 ± 0.02 eV, whereas the value determined by Chabik and Rozenfeld^[16] for the same material is 0.56 eV. Thus, the activation energy at lower temperatures obtained in this investigation (0.55 ± 0.02 eV) may be attributed to the vacancy formation energy of Cu-Zn (60/40) alloy.

The activation energy at higher temperatures obtained in this study (1.23 ± 0.03 eV) may be attributed to recrystallization in the bulk of Cu-Zn (60/40) alloy, which is lower than that obtained for Cu.^[17] Kluin and Hehenkamp^[18] deduced the vacancy formation enthalpies extracted from positron annihilation experiments by using the trap model for Cu, Cu-0.9 at.% Ge and Cu-2.7 at.% Ge; the values are 1.28 ± 0.04 , 1.16 ± 0.04 , and 0.97 ± 0.02 eV, respectively.

In ($\alpha + \beta$) brass, it is possible to observe the growth of separate vacancy populations in the two coexisting phases. Saturation in the α -phase requires that the system reach a temperature of ~ 973 K.^[19] The growth of the population of the bcc domains (β -phase) begins near 473 K, while that in the fcc domains (α -phase) begins at ~ 673 K.^[19] The decomposition of β in ($\alpha + \beta$) Cu-Zn alloys indicates that two distinct types of α -phase were formed: a rod-like type and a plate-like type.^[20] The rod-type α precipitate formed at higher temperatures (773 to 973 K) and precipitated in a Widmanstätten pattern.^[20]

From this study, it appears impossible to deduce positron diffusivity from lifetime measurements of polycrystals if the analysis is restricted to less than three lifetime components.^[21] Seeger^[21] gives an exact treatment of a model that allows fully for both the positron diffusion inside the grains and the trapping-detraping reactions at the grain boundaries. This model is also useful for the analysis of positron experiments on alloys containing precipitates.

Aldi et al.^[22] attributed the deviations from the simple trapping model of the experimental results of cold-worked Al with different thickness measured by positron lifetime spectra to the nonuniform distribution of positron traps over distances larger than the positron diffusion length. Dupasquier^[23] has reviewed and analyzed the experimental evidence of inhomogeneous trapping based on the assumption that the samples contain two types of domains with different positron trap densities. Xiong et al.^[24] considered that the domains with very high positron trap densities represented a three-dimensional (3-D) defect in many cases. In their case, grain boundaries are considered a 3-D defect. Wu^[6] found that the α rod-like 3-D form in the decomposition of β in ($\alpha + \beta$) Cu-Zn alloys has fcc crystal structure.

The relationship between the mean disappearance rate (Γ) [$\Gamma = (I_1/\tau_1 + I_2/\tau_2)$], which is the inverse of lifetime of positron in bulk (τ_b) ($\tau_b = 1/\Gamma$) as a function of isochronal annealing of Cu-Zn 60/40 (as received and quenched), is shown in Fig. 7. It shows two saturation stages for the quenched specimens and only one saturation stage for the as-received specimens. Two points of the as-received specimens coincide with that of the first stage of the quenched specimens; the only difference is that Γ decreases for the quenched specimens before reaching the second stage, whereas the as-received specimens continue

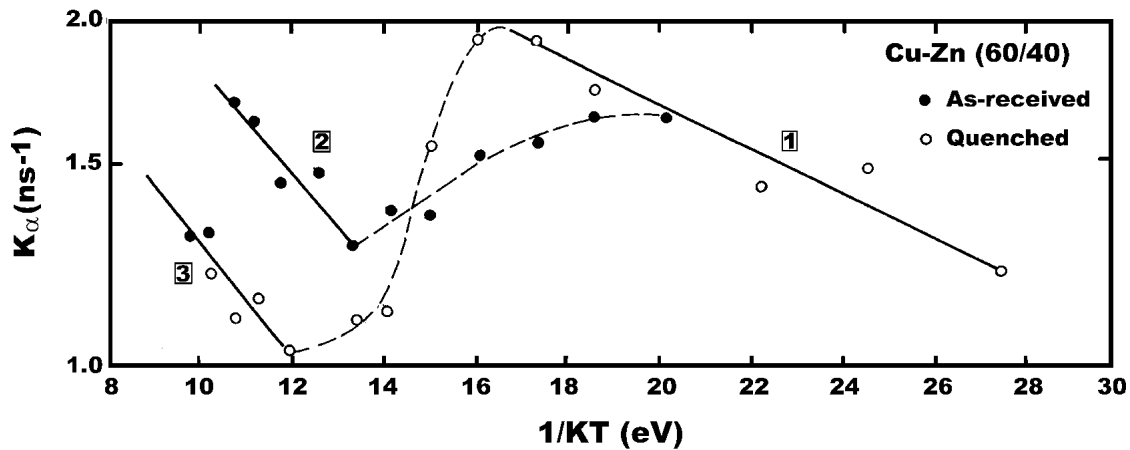


Fig. 6 Arrhenius plot for the as-received and quenched Cu-Zn (60/40). (1) Activation energy = 0.55 ± 0.02 eV. (2, 3) Activation energy = 1.23 ± 0.03 eV

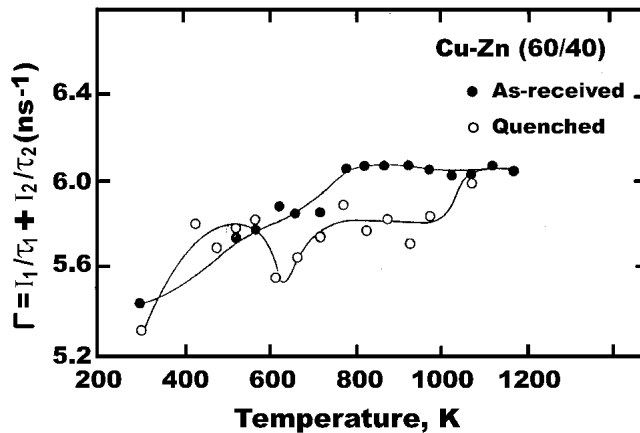


Fig. 7 The mean disappearance rate (Γ) as a function of isochronal annealing of Cu-Zn (60/40) as received and quenched (The error bars of Γ are too small to show in the curve.)

to increase. We can observe from Fig. 7 that Γ values (at saturation stages) are smaller than the annihilation rate of positrons in the free state, which gives a clear indication of the existence of 3-D defects in Cu-Zn 60/40, as concluded by Xiong et al.^[24] to interpret their experimental results of Zn-22 wt.% Al.

Xiong et al.^[24] discussed the effect of a volume fraction α occupied by 3-D defects [$\alpha = (1/\tau_1 - \Gamma)/(1/\tau_1 - K/\tau_2)$] on the classical positron trapping model. They concluded that for fine grains ($<0.28 \mu\text{m}$), positrons were trapped and annihilated in the grain boundaries, whereas for larger grain size, the volume fraction α decreased. In our investigation, the relationship between α and isochronal annealing of Cu-Zn 60/40 indicates comparable values at temperatures in the range of 573-873 K (Fig. 8). The variation of the trapping rate K with $\alpha\%$ is shown in Fig. 9 which indicates a linear relation for the two specimens, but the slope of the quenched specimens is higher. The trapping rate (K) reaches maximum at $\alpha\%$, and is equal to 43% and 48% for the quenched and as-received specimens, respectively.

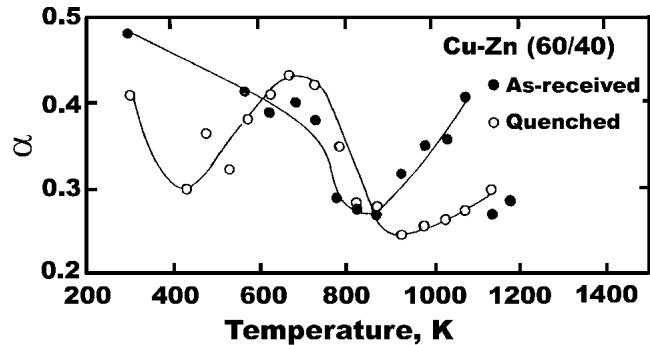


Fig. 8 The volume fraction (α) occupied by 3-D defects as a function of isochronal annealing of as-received and quenched Cu-Zn (60/40)

4. Conclusions

The conclusions are summarized as follows:

- The analysis of H_v and τ_{av} results of the isochronal annealing of both as-received and quenched Cu-Zn (60/40) indicates four regions of temperature behavior. The first region (423-573 K) is assumed to be attributed to recovery of the as-received specimens and vacancy formation for the quenched specimens. The second region (623-773 K) is characterized by near-stability of H_v and τ_{av} for both specimens, which may be due to the beginning of recrystallization. In the third and fourth regions (823-1023 K and 1073-1173 K, respectively) it appears that two opposing processes (recrystallization and phase transformation) exist.
- The Arrhenius plot, according to results of the trapping model of positron annihilation, does not exhibit the usual linear behavior. Two limited regions can be distinguished in which the activation energies deduced at lower and higher temperatures are 0.55 ± 0.02 and 1.23 ± 0.03 eV, respectively. They are assumed to be attributed to vacancy formation and recrystallization in the bulk materials, respectively.

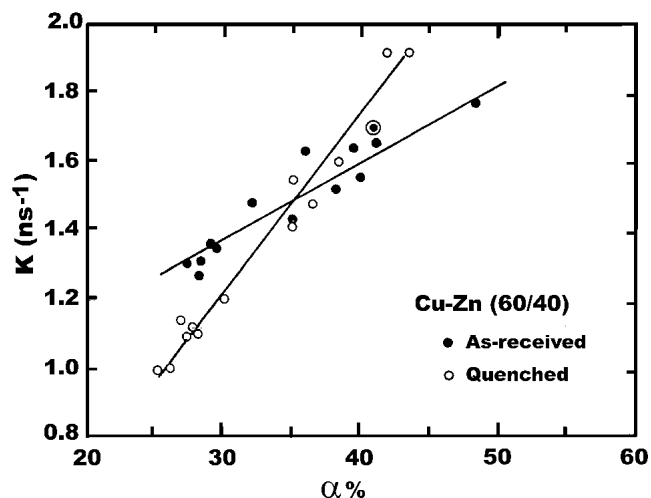


Fig. 9 The trapping rate (K) as a function of the volume fraction (α %) occupied by 3-D defects of as-received and quenched Cu-Zn (60/40)

- A detailed analysis of positron lifetime annihilation indicates clear evidence of the existence of 3-D defects in addition to the one-dimensional defect, thus the simple trapping model must be modified to consider the inhomogeneous trapping.

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