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Diffusion induced grain boundary migration in the Cu-Cd system

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Abstract

The diffusion induced grain boundary migration (DIGM) has been studied in the Cu-Cd system by exposing polycrystalline copper to Cd vapor. The temperature and time dependence of the rate of migration was measured in the range 340–480 °C. A parabolic migration behavior of the grain boundaries has been observed. The diffusivity, $D_b\delta$, was calculated from the growth rates and $v/D_b\delta$ values obtained through concentration–distance profile at each temperature. It has been observed that the diffusion coefficient obtained experimentally during DIGM in the Cu-Cd system are 8 to 10 orders of magnitude higher than the corresponding volume diffusion coefficient. The activation energy for solute transport corresponds to that required for boundary diffusion in the Cu-Cd system.

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Keywords: DIGM; Diffusion; Kinetics

1. Introduction

The migration of grain boundary under the influence of preferential solute diffusion along it is termed as diffusion induced grain boundary migration (DIGM). The DIGM was first reported by den Broeder [1] while carrying out experiments on Cr-W diffusion couples. Since then the DIGM has been reported in over 40 different binary and ternary systems and ceramic systems [2–10]. Detailed metallography on the Fe-Zn system, where a polycrystalline sheet of iron was exposed to zinc vapor, was carried out by Hillert and Purdy

[2]. The grain boundaries that were in contact with the surface of the specimen were shown to migrate. In the volume of the material there was a discontinuous change in composition, i.e., only regions of the lattice swept by the migrating grain boundaries showed a change in composition. The kinetics of DIGM has been studied in the Fe-Zn [3] and Cu-Zn [4] systems.

The mechanism of grain boundary migration when solute atoms preferentially diffuse along it and the driving force for the reaction are not yet resolved. In the mean time a number of theories for the occurrence of DIGM have been proposed. Of the various theories which have been proposed the coherency strain energy theory has been advocated by Hillert [11]. Yoon et al. [12,13] have demonstrated experimentally by the addition of a third

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element to binary alloys that the driving force for DIGM emanates from the coherency strain energy. However, Ma et al. [14,15] have demonstrated DIGM when the coherency strain energy was zero. This has been achieved by the addition of 19.4 at. pct. Pd to pure Ni such that its lattice parameter became equal to that of pure Cu. The DIGM was observed when copper diffused along grain boundaries in a Ni-19.4 at. pct. Pd/Cu diffusion couple thus suggesting that the coherency strain energy need not be the driving force for DIGM. A similar view has been held by Kuo and Fournelle [16] in their work on the up-quench and down-quench experiments in an Al-Cu alloy. The coherency strain energy has been calculated to be insignificant in comparison to the free energy of mixing thus suggesting that the driving force for DIGM may be due to the free energy of mixing. The coherency strain energy is proposed to be the lower limit of the driving force for DIGM by Li and Hillert [3]. A dislocation climb mechanism has been suggested by Balluffi and Cahn [17] and Smith and King [18].

This investigation has been carried out to study the rate of migration and the diffusional transport mechanism in the Cu-Cd system during DIGM.

2. Experimental procedure

The diffusion induced grain boundary migration was studied in the Cu-Cd system with sheets of copper (99.999% purity) exposed to Cd vapor. The metal Cd was also of 99.999% purity. Copper sheets approximately 6 mm wide, 40 mm long and 0.3 mm thick were encapsulated in silica tubes under vacuum and annealed for 12 h at 1000 °C. This was done to obtain a polycrystalline structure and a large grain size such that the grain boundaries are perpendicular to the free surface. The annealed sheets were electropolished in an orthophosphoric acid solution to obtain a final thickness of approximately 250 μm . Specimens approximately 6 \times 8 mm were cut from the sheet in a diamond wafering machine fitted with a small holder. The 6 mm wide sheet was held from one end and cuts made by adjusting the position of the blade with the help of a micrometer fitted in the

diamond wafering machine. This was done to avoid the strain on the sample surface. Then the samples were individually encapsulated in silica tubes along with pieces of pure Cd under vacuum. The amount of Cd was four times the weight of the copper sheet. This ensured ample supply of Cd vapors such that the migrating grain boundaries did not recede during the long exposure time of diffusion anneal. Experiments were also carried out with the Cu-20 at. pct. Cd alloy as a source of Cd vapor. It was observed that very insignificant migration of the grain boundaries occurred even after 5 days as the alloy composition lies in the two phase field ($\alpha + \text{Cu}_2\text{Cd}$). The α phase has low solubility of Cd in Cu with a maximum value of 2.14 at. pct. Cd at 549 °C. The Cu-20 at. pct. Cd alloy was therefore not used any further for any DIGM studies in this system.

The sealed samples were diffusion annealed in a tubular furnace in the temperature range 340–480 °C. In order to study the rate of grain boundary migration a number of specimens were annealed at each temperature and subsequently quenched in an ice + water mixture. A small part of the specimen was cut from each sample. In order to study the composition–distance profile, the sample was mounted in plastic resin such that the cross-section (thickness part of the sheet) was exposed. The remaining sample, approximately 6 \times 8 mm, was also mounted to study the distance of grain boundary migration and ultimately the growth rate. Each sample was annealed only once, the growth distance was measured and it was subsequently retired.

The specimens were prepared for metallographic examination by using standard metallographic techniques. The distance of grain boundary migration was studied with the help of an optical microscope fitted with a reticule. The composition profile of the sectioned sample was obtained in a JEOL electron probe microanalyser, model 8600 MX fitted with a superprobe and standards of pure metals Cd and Cu.

3. Results and discussions

Experiments were carried out to study the migration rate during DIGM when polycrystalline

copper was exposed to Cd vapor. The concentration–distance profile of Cd diffusion was recorded on a plane perpendicular to the foil surface to facilitate the analysis.

3.1. Microscopic examination

The microstructures of the polycrystalline specimen were recorded at different stages of transformation in pure copper diffusion annealed under Cd vapor. The grain boundary migration appears to have occurred near the grain boundary–twin boundary intersection and the entire segment has moved in one direction, Fig. 1. With an increase in time of diffusion anneal, the migration distance of the grain boundary was observed to increase as shown in the photomicrographs of Figs. 2 and 3 diffusion annealed at 480 °C. Some of the boundaries did not migrate even after annealing for a

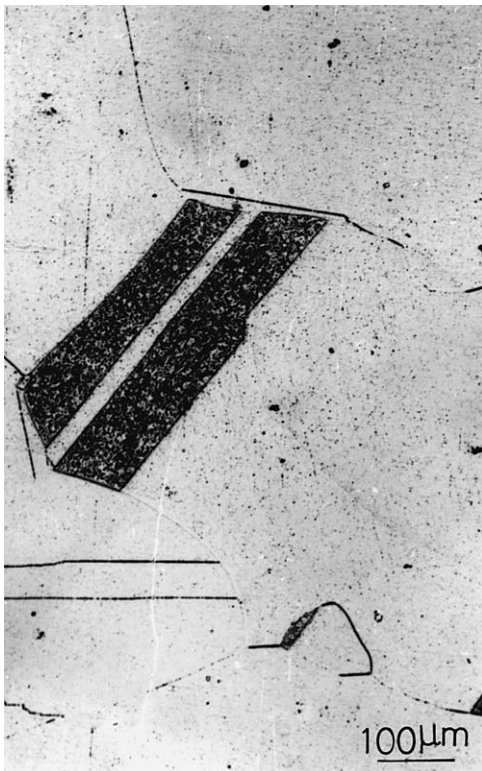


Fig. 1. Early stage of DIGM, diffusion annealed for 12 h at 480 °C.

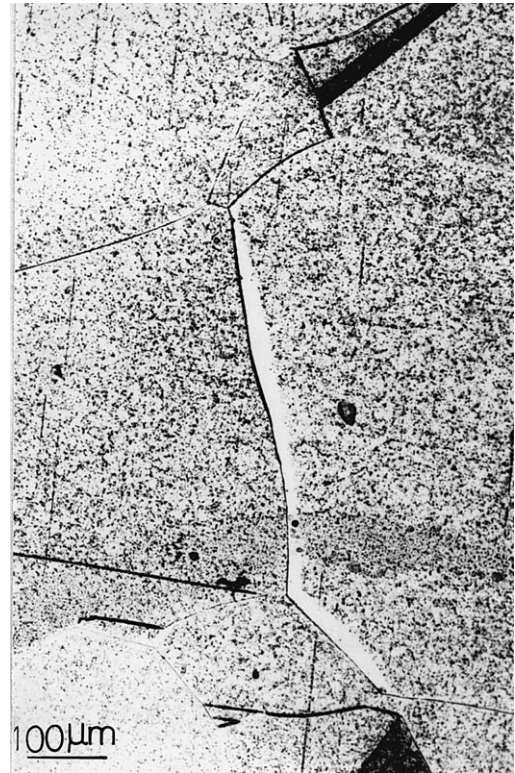


Fig. 2. The whole grain boundary segment has migrated, 96 h at 480 °C.

long time at such a high temperature. This is most likely due to the angle of misorientation of grain boundaries. Generally, low angle grain boundaries and boundaries in a special orientation such as CSL (coincidence site lattice) have low diffusivity and hence mobility. The observation is consistent with the results of DIGM in the Fe–Zn system reported by Li and Hillert [3].

The grain boundaries were observed to preferentially migrate near a grain edge as shown in the photomicrograph of Fig. 4. A small grain “A” appears to be shrinking during development of the polycrystalline structure of pure Cu. When grain boundaries were exposed to the Cd vapor, it diffused near grain edges of the small grain. The migration appears to have occurred on both sides of the grain boundary near the triple point. Examination of the geometry near the triple point at “T” indicates that the following relation will hold.

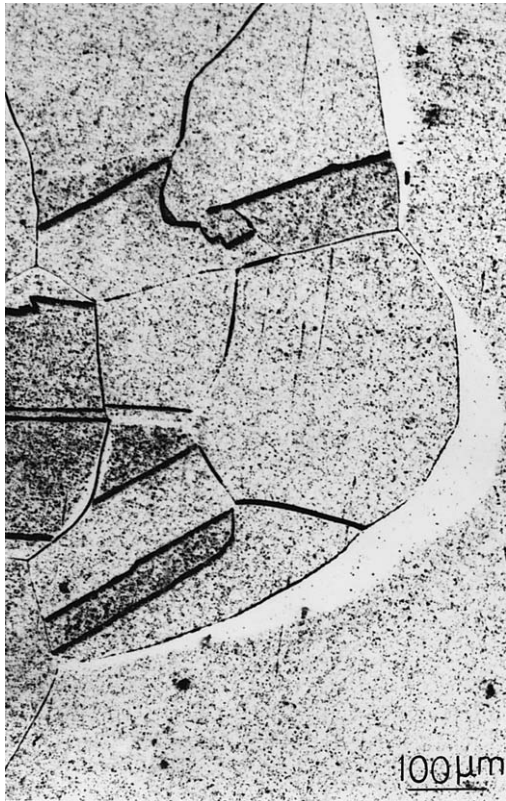


Fig. 3. Optical photomicrograph showing increased migration with increasing diffusion annealing time, 120 h at 480 °C.

$$\frac{\gamma_{g3}}{\sin\theta_3} = \frac{\gamma_{g2}}{\sin\theta_2} = \frac{\gamma_{g1}}{\sin\theta_1} \quad (1)$$

The angles θ_1 , θ_2 , and θ_3 are respectively, 150°, 145° and 65°. This would indicate that γ_{g3} has the highest value by a factor of approximately two of γ_{g1} and γ_{g2} . Thus it is obvious that if the grain boundary with interfacial energy γ_{g3} recedes, higher energy will be released which may add to the driving force for DIGM.

In many instances the DIGM was preferentially observed near grain boundary/twin boundary intersection (Fig. 5). If we examine the three interface junction at “A”, the following equality holds

$$\frac{\gamma_t}{\sin\theta_1} = \frac{\gamma_g}{\sin\theta_2} = \frac{\gamma_{gt}}{\sin\theta_3} \quad (2)$$

where γ_{gt} is the interfacial energy of the grain boundary where twin boundary terminates as

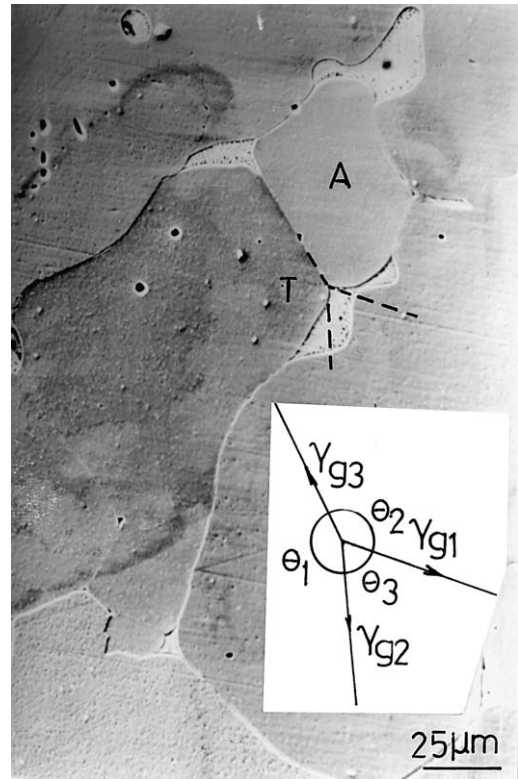


Fig. 4. DIGM has been preferentially observed near a grain edge where large grains surround a small grain, 50 h at 440 °C.

shown in the schematic of Fig. 5. Since the angle θ_2 is more than θ_3 , $\gamma_{gt} > \gamma_g$, both angles being more than 90° but less than 180°.

In some areas of the specimen, the twin boundary was observed to nucleate and grow in the wake of DIGM, Fig. 6. This has been amply illustrated for the Ag-Cd system by Lopez [19], and has been explained from the stress build up of sufficient magnitude at the migrating boundary, which generates $a/6[112]$ glissile partial dislocation in the FCC lattice.

3.2. Growth rate

The average migration rates of the grain boundaries during DIGM at different temperatures were determined from measurements of average migration distance of the boundaries as a function of time of diffusion anneal. The general scatter in the data is shown at 480 °C. The migration rates

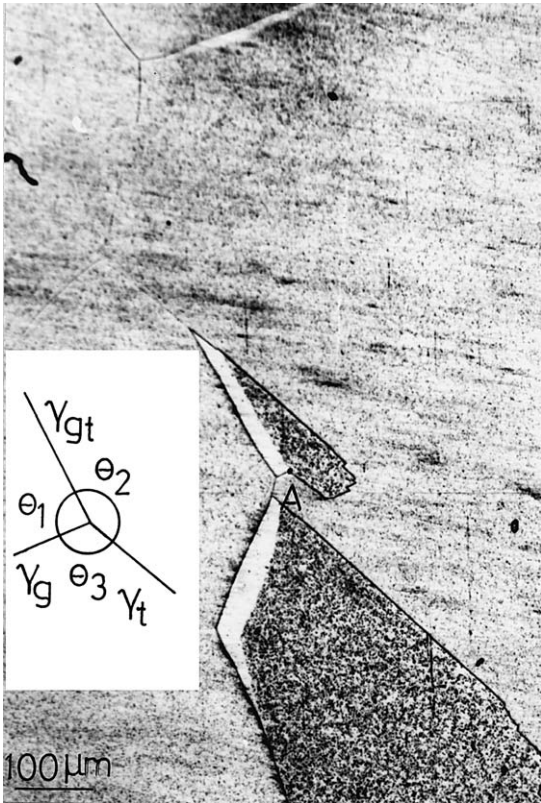


Fig. 5. DIGM on the grain boundary/twin boundary intersection where γ_{gt} is high, 96 h at 480 °C.

were determined in the temperature range 340–480 °C at 20 °C interval. The migration distance was measured in each specimen as the maximum distance from the initial position to the leading position of the boundaries. About 30 measurements were made in each specimen and boundaries which have migrated reasonably were considered for measurement.

The average migration distance has been plotted against the time to annealing, Fig. 7. The plot shows a parabolic behavior in that the growth rate decreases continuously although the growth distance increases monotonically with the time of anneal. The rate of migration is a function of time and can be determined by drawing a tangent at any point on the curve of Fig. 7. The growth distance has been observed to increase with temperature at any given time of annealing indicating that the growth rate increases with increasing diffusion temperature.

The average migration distance was plotted against the time of anneal on a log–log scale to determine the time exponent, Fig. 8. The data show some deviation but can be fitted to a straight line at each temperature and can be represented by the following equation

$$x = kt^n \tag{3}$$

where x is the average migration distance during

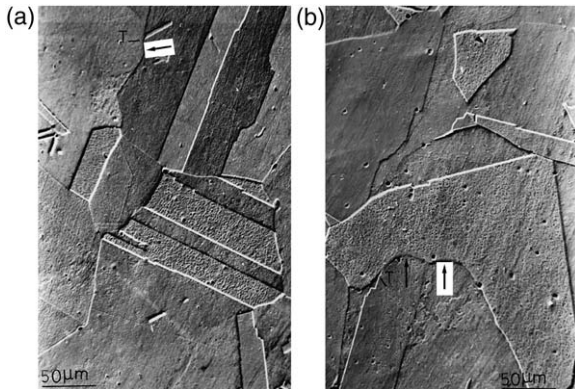


Fig. 6. Optical photomicrograph showing generation of twin boundaries in the wake of DIGM, (a) 10 h and (b) 70 h at 440 °C. The twins (T) are marked and the direction of migration of the grain boundary is shown by arrows.

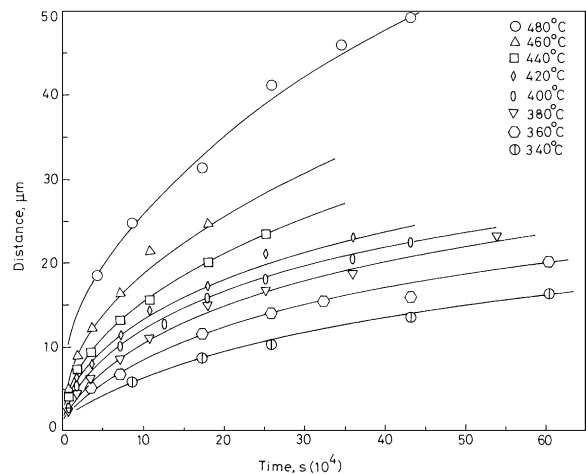


Fig. 7. Average migration distance vs time of diffusion anneal showing parabolic growth behavior at all temperatures.

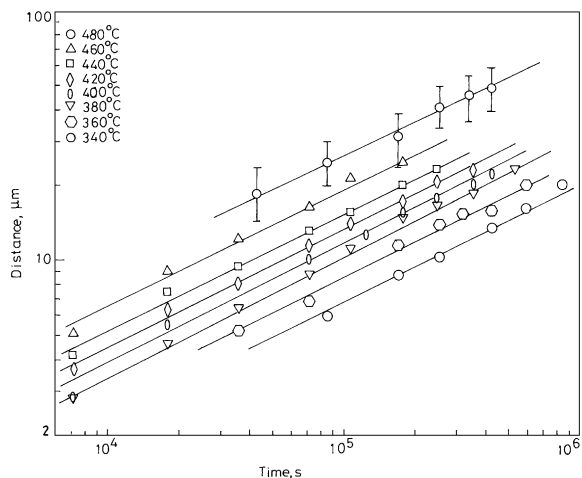


Fig. 8. Average migration distance vs time on a log–log scale with the time exponent, n , very close to 0.5.

DIGM, t is the time of annealing, n is the time exponent and k is a material constant and is dependent upon temperature. The time exponent, n , ranged from 0.47 to 0.509 in the temperature range 340–480 °C. These values are very close to 0.5 and it can be concluded that the migration of grain boundaries due to diffusion of Cd in Cu follows a parabolic growth behavior. Since the time of annealing in the experiment was not very long, there happens to be ample supply of Cd and no reversal of direction of migration was observed, as was the case in Fe–Zn and Cu–Zn systems reported by Li and Hillert [3,4].

The growth rate was calculated by differentiating x with respect to t in Eq. (3), which gives the growth rate as

$$v = xn/t \quad (4)$$

The time dependent growth rate is shown in Fig. 9 at all diffusion annealing temperatures. The growth rate is as high as $5 \times 10^{-10} \text{ ms}^{-1}$ after 2 h at 480 °C to $0.5 \times 10^{-10} \text{ ms}^{-1}$ after 100 h indicating an almost 10 times decrease in the above period. A growth rate as low as $0.173 \times 10^{-10} \text{ ms}^{-1}$ has been observed after 100 h at 340 °C.

We can make a comparison of the growth rates observed in this investigation to those reported by Li and Hillert [4] for the Cu–Zn system. They have reported a growth rate of $4.3 \times 10^{-10} \text{ ms}^{-1}$ after

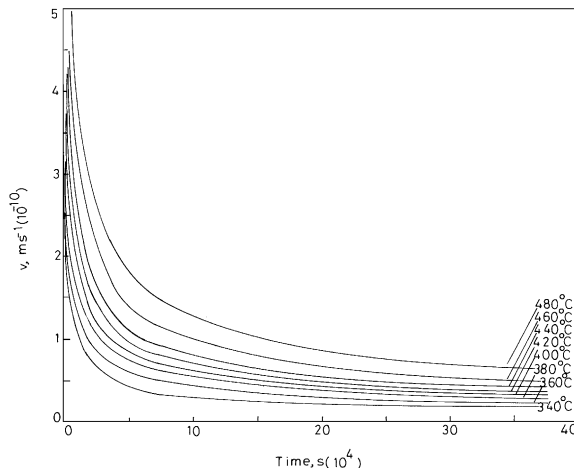


Fig. 9. The growth rate vs time of anneal.

770 min. ($4.62 \times 10^4 \text{ s}$ or 12.83 h) of anneal at 400 °C when Cu was exposed to Zn vapor with an alloy of Cu–Zn containing 30.5% Zn. This compares well with the value of $1 \times 10^{-10} \text{ ms}^{-1}$ after annealing for 10 h at 400 °C. The growth rate was reported to drop to $1.3 \times 10^{-10} \text{ ms}^{-1}$ after 48 h of annealing which in the case of the Cu–Cd system is $0.5 \times 10^{-10} \text{ ms}^{-1}$.

3.3. Concentration–distance profile

The concentration–distance profile was obtained by measuring Cd concentration as a function of distance from the surface to the center across the specimen sectioned perpendicular to the foil surface. The composition analysis has been carried out using electron probe micro analyzer (EPMA) and standards of high purity of Cu and Cd. The data are presented in Fig. 10(a–f) at some of the temperatures of the experiment. A thin layer of the Cu_2Cd phase was observed at the surface, which became the source of Cd during DIGM. There was a very steep fall in Cd concentration within just few μm of the Cu/Cu₂Cd interface. The position at which the Cd concentration corresponds to the maximum solubility limit of Cd in Cu [20] at the temperature of diffusion anneal is considered as the surface. This happened to be within a few microns of the actual surface. The position of the sectioned surface of the foil at which Cd concentration has

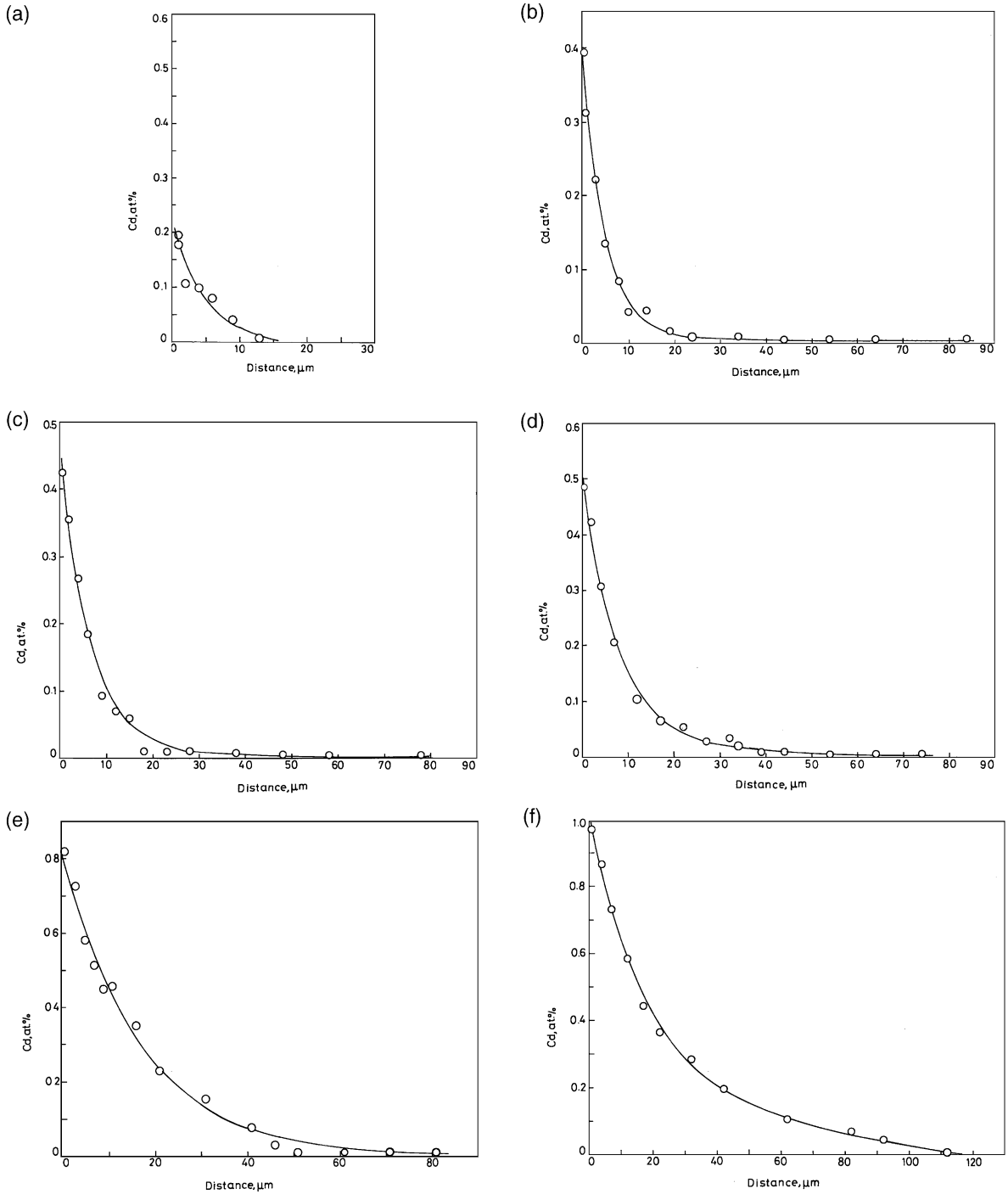


Fig. 10. Concentration–distance profile on a plane perpendicular to the foil surface. (a) 340 °C (b) 360 °C (c) 380 °C (d) 400 °C (e) 440 °C (f) 460 °C. The calculated curve, which matches the data, is shown by full line in each drawing.

reached a value zero is considered to be the center of the foil.

The concentration–distance profiles were analyzed using Cahn’s solution [21],

$$\frac{X_B}{X_B^0} = \frac{\cosh(z\sqrt{v/D_b\delta})}{\cosh(z_0\sqrt{v/D_b\delta})} \quad (5)$$

where X_B^0 is the concentration at the surface, Z is the distance from the center of the foil and Z_0 is the value of Z at the surface. In Eq. (5) D_b is the boundary diffusion coefficient and δ is the thickness of the boundary. The data obtained at each temperature was fitted to the equation by trial and error and the $v/D_b\delta$ value corresponding to the best fit was obtained. These are shown as full lines in each drawing. By combining $v/D_b\delta$ values with the rate of grain boundary migration at the same time at which the concentration distance profile was obtained, the $D_b\delta$ values were calculated. The results are shown in Fig. 11 with a model calculation, which is as follows:

At 340 °C, $v = 13.88 \times 10^{-12} \text{ ms}^{-1}$ (in a specimen annealed for 7 days)

$v/D_b\delta = 5.663 \times 10^{10} \text{ m}^{-2}$ (obtained from the best fit of the data) yields $D_b\delta = 2.47 \times 10^{-22} \text{ m}^3\text{s}^{-1}$.

The $D_b\delta$ values range from $2.47 \times 10^{-22} \text{ m}^3\text{s}^{-1}$ at 340 °C to $3.49 \times 10^{-20} \text{ m}^3\text{s}^{-1}$ at 480 °C. The $D_b\delta$

value obtained during discontinuous precipitation at 337 °C in a Cu-2 at. pct.Cd alloy [23] is $5 \times 10^{-22} \text{ m}^3\text{s}^{-1}$ which is very close to the value obtained during DIGM in this investigation. It is to be mentioned that the diffusion of solute occurs through migrating grain boundaries during discontinuous precipitation and thus there is similarity between the two phenomena.

Assuming grain boundary thickness to be 1 nm, the observed diffusivity, D_b , at 340 °C is $2.47 \times 10^{-13} \text{ m}^2\text{s}^{-1}$. The corresponding volume diffusion coefficient as calculated from Arrhenius parameters $Q_v = 191.16 \text{ kJmol}^{-1}$ and $D_0 = 0.95 \times 10^{-4} \text{ m}^2\text{s}^{-1}$ from the work of Herone et al. [22] is $D_v = 4.876 \times 10^{-21} \text{ m}^2\text{s}^{-1}$. The volume diffusion coefficient is eight orders of magnitude lower than the observed value at 340 °C. These calculations confirm that the diffusion of Cd occurred through grain boundaries during DIGM.

The ratio of volume diffusion coefficient to migration rate at 340 °C was calculated and it has been observed to have a range of 0.038 to 0.28 nm in the diffusion annealing time of 2 to 100 h. The lattice spacing of Cu is 0.361 nm, which is more than the calculated D_v/v . It can be reasoned therefore that there is negligible volume diffusion of solute in front of the migrating boundary and all of the driving force is used in boundary migration through solute transport along it.

The ratio of D_v/v was also calculated at 460 °C and it has been observed to be in the range of 5.7–45.7 nm in the time of diffusion anneal of 2 to 100 h. The D_v value at 460 °C is calculated to be $2.26 \times 10^{-18} \text{ m}^2\text{s}^{-1}$. This indicates that some volume diffusion ahead of the migrating boundary has occurred at higher temperatures of diffusion annealing.

In order to calculate the activation energy during DIGM, the $D_b\delta$ values were plotted against the reciprocal of absolute temperature in a semi-log plot, Fig. 11. The data falls on a straight line. The activation energy as calculated from the slope of the straight line is $137.5 \text{ kJ mol}^{-1}$.

A comparison of observed activation energy with those for volume diffusion of Cd in Cu ($191.16 \text{ kJmol}^{-1}$) as reported by Hirone et al. [15] indicates that the observed values is 0.7 times the volume diffusion value which is well within the

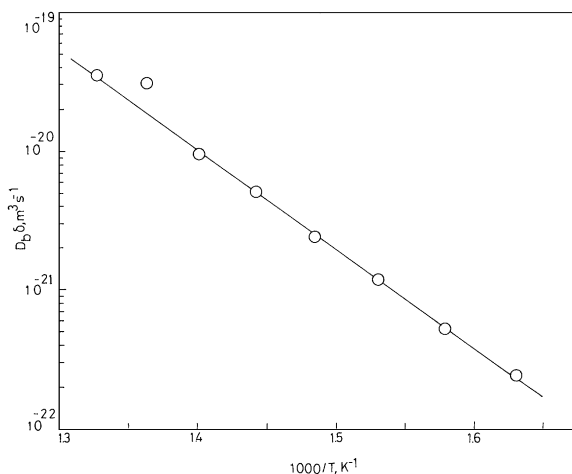


Fig. 11. $D_b\delta$ vs. $1/T$ plotted on a semi-log scale.

0.5–0.75 times observed for boundary diffusion in many metals and alloys. The activation energy during discontinuous precipitation in a Cu-2 at. pct. Cd alloy [23] has been observed to be in the range 118–125 kJmol⁻¹ which is close to 137.5 kJmol⁻¹ as observed in this investigation. From the diffusivity and activation energy values it can be concluded that the transport of Cd through grain boundaries of Cu are responsible for DIGM in the Cu-Cd system.

3.4. Driving force

An attempt has been made to calculate the driving force for the DIGM at all diffusion annealing temperatures. The quantity ΔG_m has been evaluated from the change in composition at the grain boundaries. From the procedure used by Hillert and Purdy [2], the following approximation has been used.

$$\Delta G_m = RT X_{Cd} \quad (6)$$

where X_{Cd} represents the final composition of Cd at the grain boundaries of initially pure copper specimens. The Cd concentration is taken as the solubility limit of Cd in Cu. This is consistent with the observation of Li and Hillert [3] where the Zn concentration near the original surface was close to the solubility limit of Zn in α iron in the Fe-Zn system. The concentration–distance profiles obtained in this investigation also suggest that the Cd concentration near the original surface is very close to the solubility limit of Cd in pure Cu if we disregard few microns thick layer of the Cu₂Cd phase at the surface which is the source of Cd in this system. The calculated values are shown in Table 1. The total driving force ranges from 18.5 to 75.9 Jmol⁻¹ in the temperature interval of 340–480 °C. A calculation was carried out to determine the coherency strain energy as the driving force for comparison by using the Eq. (7),

$$\Delta G_{coh} = \frac{E\eta^2 V_m (X_B^\alpha - X_B^o)^2}{1-\nu} \quad (7)$$

where E is the modulus of elasticity, η the misfit parameter, X_B^α and X_B^o are the compositions in the grain in front of the migrating grain boundary and well away from it, respectively and ν is the Pois-

son's ratio. The calculated values of ΔG_{coh} are given in the Table 1 from the value of E for different planes from the data given by Handwerker et al. [13]. A value as low as 0.8 Jmol⁻¹ has been obtained at 340 °C. The total driving force from the chemical free energy change is as high as 20 times that from coherency strain energy. The chemical free energy change may therefore acts as a driving force for DIGM.

In order to determine the force–velocity relationship, the growth rate during early stage of DIGM has been plotted against the chemical free energy change, Fig. 12. Extrapolation of the curve indicates that a minimum driving force of about 12 Jmol⁻¹ is essential to induce grain boundary migration in this system. This is consistent with the prediction of a threshold force necessary for DIGM under the influence of a chemical force suggested by Purdy [24].

4. Conclusions

The DIGM has been observed to occur in the Cu-Cd system when polycrystalline Cu was exposed to Cd vapor. The migration distance of the grain boundary increased with the time of anneal and followed a parabolic growth behavior with the time exponent in the range 0.487–0.509. The rate of migration of the grain boundaries was observed to decrease with increasing time of anneal. This may be due to the reduced driving force available as the time of transformation increases.

The diffusivity D_b has been observed to be in the range 2.47×10^{-13} to 3.49×10^{-11} m²s⁻¹ in the temperature range 340–480 °C. These values are eight to ten orders of magnitude higher than the corresponding volume diffusion coefficient in the temperature range of this study. The activation energy for solute diffusion during DIGM has been calculated to be 137.5 kJ mol⁻¹, which is half to three-fourth of the activation energy of volume diffusion of Cd in Cu. The above value is also close to the activation energy for solute diffusion during discontinuous precipitation in Cu-Cd alloys, which has been established to occur by solute transport through grain boundaries. It can therefore be concluded that DIGM in the Cu-Cd system occurs by

Table 1
Rate of growth during early stages of DIGM, chemical free energy change and coherency strain energy

| T, °C | $X_{Cd} (\times 10^{-2})$ | $v, \text{ms}^{-1} (\times 10^{-10})$ | $-\Delta G_m, \text{Jmol}^{-1}$ | $-\Delta G_{(\text{Coh})}, \text{Jmol}^{-1}$ | | |
|-------|---------------------------|---------------------------------------|---------------------------------|--|-------|-------|
| | | | | {100} | {110} | {111} |
| 340 | 0.3636 | 1.26 | 18.53 | 0.8 | 1.61 | 1.81 |
| 360 | 0.3939 | 1.5 | 20.73 | 0.93 | 1.88 | 2.12 |
| 380 | 0.4242 | 1.84 | 23.03 | 1.08 | 2.19 | 2.46 |
| 400 | 0.4848 | 2.33 | 27.12 | 1.4 | 2.85 | 3.2 |
| 420 | 0.6363 | 2.61 | 36.66 | 2.43 | 4.93 | 5.54 |
| 440 | 0.8181 | 3.11 | 48.49 | 4.02 | 8.15 | 9.17 |
| 460 | 0.9696 | 3.96 | 59.09 | 5.64 | 11.44 | 12.86 |
| 480 | 1.2121 | 4.89 | 75.88 | 8.82 | 17.87 | 20 |

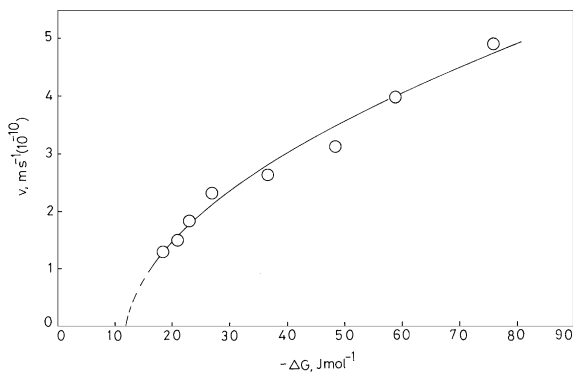


Fig. 12. Growth velocity vs driving force (chemical)

solute transport through the grain boundaries. The chemical free energy change acts as a driving force during boundary migration.

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