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Effect of isothermal annealing on the Bordoni relaxation in cold worked copper

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The Bordoni relaxation in ultra-purity cold worked copper subsequent to isothermal annealing in the temperature range 298–423 K at 700 Hz frequency is investigated using a set up based on the electrostatic system. The Bordoni peak is observed at 79 ± 2 K. The annealing characteristics of the Bordoni peak are found to be of complex nature. The decay and subsequent regrowth of the peak following annealing above room temperature are interpreted in terms of defects diffusing to dislocations and their removal from dislocations, respectively. The calculated activation energy values for the first decrease (0.55 eV), first increase (0.24 eV) and first minimum (0.27 eV) are in good agreement with the activation energies for interstitial and divacancy diffusion estimated by different workers. The activation energy for divacancy diffusion is computed to be 0.49 eV.

1. Introduction

The relaxation peak first discovered by Bordoni [1] in the internal friction spectra of cold-worked face-centred cubic (fcc) metals at low temperatures continues to be the subject of a considerable amount of experimental and theoretical investigations. In recent decades the major characteristics of the Bordoni peak have been reviewed [2–8].

Much interest has been focused on the Bordoni relaxation process mainly because it has been assumed that the peak represents a special form of an elemental dislocation motion which does not implicate other types of lattice defects. The fact that the relaxation peak depends on cold work while the temperature at which it occurs is irresponsive to the concentration of point defects, is taken to mean that the Bordoni peak accrues from the inherent characteristic of dislocations. The change of the Bordoni peak temperature with frequency has been interpreted in terms of a thermally activated relaxation process.

The most prominent features of the experimental data have been explained convincingly by Seeger's [9] hypothesis of double-kink generation on dislocations lying parallel to one of the close packed crystallographic directions in the lattice. At a finite temperature the dislocation line is assumed not to lie in a single potential valley only but to contain kinks as it passes from one valley to another. If a stress is applied which is less than that required to move the dislocation line by one inter-atomic distance (Peierls stress), then the dislocation line can be displaced by a mechanism involving the formation of additional pairs of kinks. This would require thermal energy and could occur with a temperature-dependent frequency. Seeger proposed that a relaxation peak could result if the frequency of the applied stress is comparable to the frequency with which a pair of kinks is formed. In attempts to elucidate the dependence, to certain extent, of the temperature of the peak on the degree of prior cold work and the impurity concentration of the metal; and also to account for the fact that the peak is appreciably broader than a single relaxation peak, Seeger's original theory has undergone several modifications [10–14]. Comparatively more recent modifications have been proposed by Schlipf and Schindlmayr [15] and Esnouf and Fantozzi [16] to explain the latter feature of the peak.

The Bordoni relaxation, which appears after plastic deformation in fcc metals, has been generally attributed to the thermally activated nucleation of double kinks on dislocations. Several workers have employed the dislocation double kink model to explain various experimental observations [17].

The Bordoni relaxation in copper has been studied extensively in the kHz to MHz frequency range. The present research was carried out in an attempt to

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identify the effect of isothermal annealing on the Bordoni relaxation characteristics in high-purity polycrystalline copper at a frequency less than a kHz.

2. Experimental procedure

The specimens tested in this investigation were of spectroscopically pure copper with a total detectable impurity content not exceeding 7 ppm. The material was supplied by Johnson Matthey Company Limited in the form of cylindrical rods 15 cm long and 5 mm in diameter. The test pieces had the final dimensions of 3 × 0.9 × 0.1 cm. All the specimens were given a standard vacuum anneal at 873 K for 3 hours prior to 10% plastic deformation by cold rolling. Since the annealing characteristics of the Bordoni peak tend to be of a complex nature and depend markedly on the amount of deformation and grain size, care was taken for all the specimens to undergo identical thermomechanical treatments.

Rectangular copper specimens of the dimensions given above were excited into vibration electrostatically by applying a high frequency potential to an electrode placed a few thousandths of an inch from one end of the test piece. The vibrational frequency of the specimen was twice that of the applied voltage, so that it was possible to eliminate the initial frequency, which could increase the background friction and would also lead to spurious results, by a selective amplifier.

The internal friction apparatus (Fig. 1) consisted essentially of a cylindrical brass chamber of 0.36 cm wall thickness, 10 cm internal diameter, 9.9 cm height and a 1.3 cm wide flange at the top. A brass lid about 0.65 cm thick and 12.6 cm diameter could be screwed onto the flange with a teflon O-ring for vacuum sealing.

The specimen was gripped in a holder by a screw to lay it parallel to two brass electrodes. The specimen holder was held by a pillar fixed perpendicularly to the lid of the chamber. The electrode assemblies consisted of teflon cylinders into one end of which was screwed a brass section to act as the fixed plate of the condenser. The other end of the assembly was connected to a micrometer situated outside the chamber to permit vertical motion of the electrodes. One electrode was used as an exciter while the other was employed as a detector to enable internal friction measurements to be made. The distance between the specimen and either electrode could be controlled accurately to a thousandth of an inch. The chamber was evacuated with a rotary pump.

All electrical connections to the electrodes were made through the brass vacuum tube. The driving electrode was connected to a Muirhead D-650-B oscillator which has a maximum output of 150 volts. The detecting electrode, which is biased by 300 volts D.C. to improve its sensitivity, was connected via a condenser to a frequency analyser and amplifier. This enables the signal which is of the order of 0.1 MV to be selectively amplified in the presence of harmonics and other undesirable tones of fundamental frequency. The selectively amplified signal was fed to a monitoring oscilloscope and to an electronic gate and counter assembly, by means of which the logarithmic decrement of the decay of the vibration can be measured. Fig. 2 shows a block diagram of the electrostatic internal friction experimental set-up.

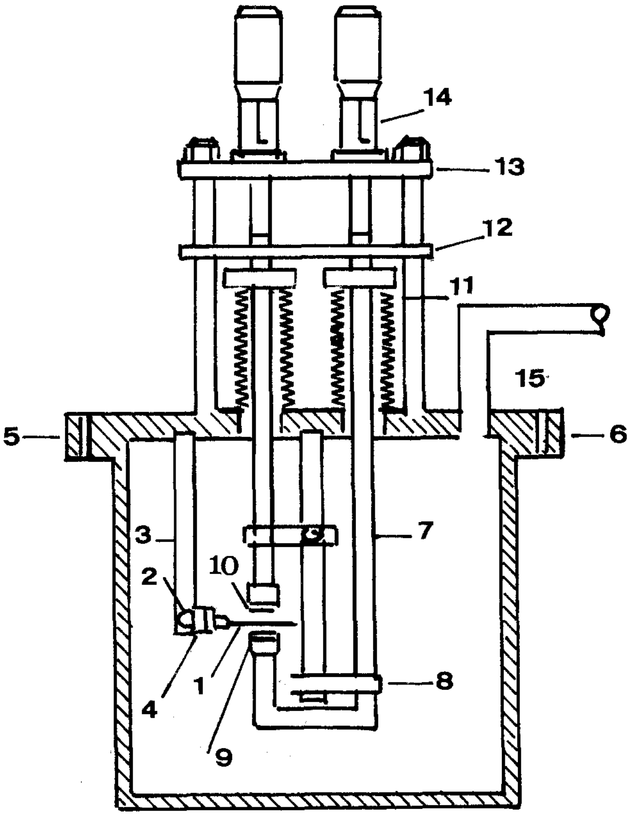

Figure 1 The internal friction apparatus (1) specimen (2) specimen holder (3) supporting pillar (4) screw for specimen holder (5, 6) teflon O-ring (7) stainless steel bar (8) movable support (9) teflon cylinder (10) brass electrode (11) bellows (12) fixed stage for electrode/specimen alignment (13) fixed stage for the micrometer (14) micrometer (15) brass tube to vacuum.

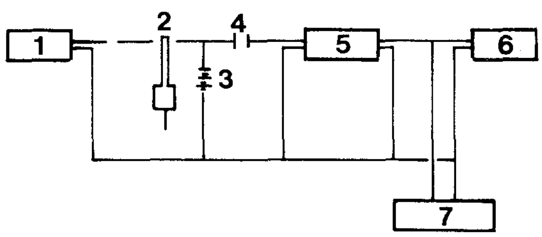

Figure 2 Block diagram of the electrostatic internal friction experimental set-up: (1) decade oscillation (2) specimen (3) 300 V D.C. batteries (4) condenser (5) frequency analyser (6) electronic counter (7) cathode ray oscilloscope.

After the specimen plate is appropriately mounted, the chamber is evacuated to less than 0.1 mm Hg and immersed in liquid N_2 (or He) contained in a Dewar flask. The electrodes are brought to within a few thousandths of an inch of the specimen. The oscillator is tuned to half the resonant frequency, w, of the specimen which can be determined approximately from the equation

$$W^2 = \left(\frac{1.9}{l}\right)^4 \left(\frac{Ea^2}{12\rho}\right) \tag{1}$$

where l and a are the specimen length and thickness, respectively; E is Young's modulus and ρ is the density of the material.

The drive voltage was switched off and the number of cycles occurring between two preset voltage levels in the decay oscillation of the cantilever specimen was measured by a counter unit. The internal friction is then computed from the logarithmic decrement.

3. Discussion and results

The characteristic features of the Bordoni peak subsequent to isothermal annealing at constant temperatures of 298, 323, 373 and 423 K are reported in the present paper. Results pertaining to the influence of isothermal annealing on the internal friction behaviour of high-purity polycrystalline copper are presented in Figs 3–23.

(i) Annealing at 298 K

Figs 3–5 depict the effect of isothermal annealing at 298 K. The frequency of vibration was 580 Hz. Annealing for 16.5 hours reduces the Bordoni peak height from a deformed value, Q, of 13.7×10^{-4} to 11×10^{-4}. Further annealing causes the peak height to increase sharply, reaching the level of 15.6×10^{-4} after 33 hours (Fig. 3). This is appreciably higher than the deformed value. Annealing for 67.5 hours causes the peak to fall to a minimum of 11.17×10^{-4} (Fig. 4). The peak height goes through a maximum and a minimum after 131.5 and 147.5 hours respectively (Figs 4–5). Annealing was not pursued beyond 165 hours.

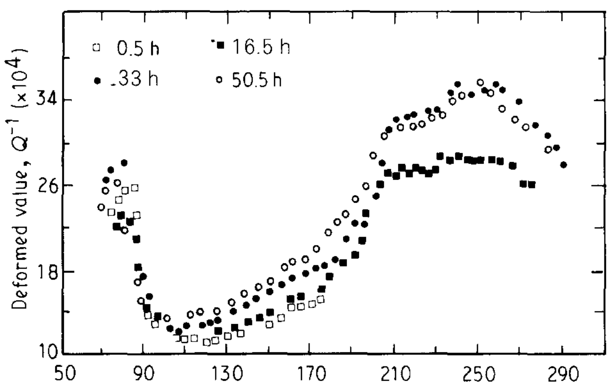

Figure 3 The effect of isothermal annealing time (0.5–50.5 h) at 298 K.

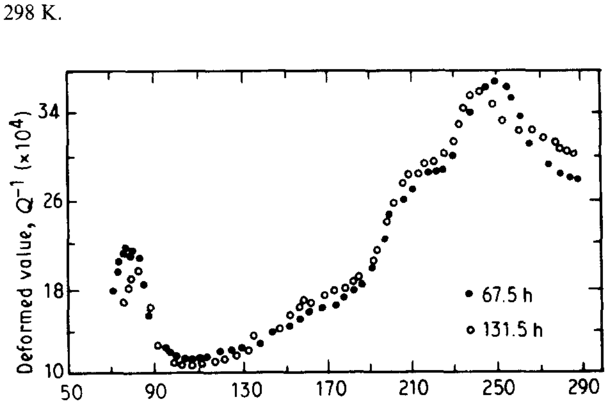

Figure 4 The effect of isothermal annealing time (67.5 h, 131.5 h) at 298 K.

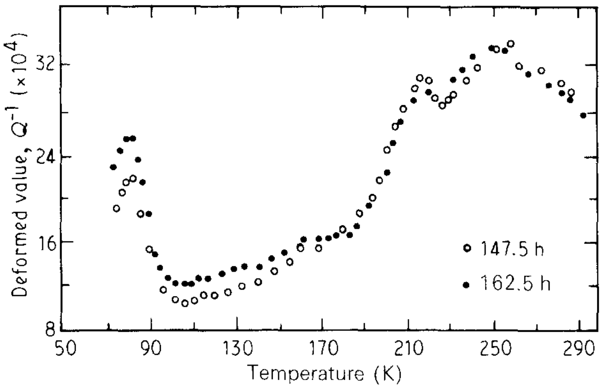

Figure 5 The effect of isothermal annealing time (147.5 h, 162.5 h) at 298 K.

The peak temperature remains constant (82 K) in the first 33 hours of annealing but experiences a measurable decrease to 79 K for an extended annealing period of 162 hours.

The general background level of internal friction at 113 K does not alter markedly with annealing. However, the total internal friction at higher temperatures tends to increase with increasing annealing time. Peaks are observed at 213 and 243 K. Both peaks grow with room temperature annealing up to 67.5 hours. On further annealing the height of the first peak undergoes a decrease which corresponds to the second regrowth of the Bordoni peak.

(ii) Annealing at 323 K

A second specimen of the same material, vacuum annealed at 873 K for 3 hours and deformed 10.65% was annealed at 323 K in argon atmosphere. The specimen was removed from the furnace after certain annealing periods to measure the internal friction in the temperature interval 77 to 170 K at a frequency of 690 Hz. The results are presented in Figs 6–11.

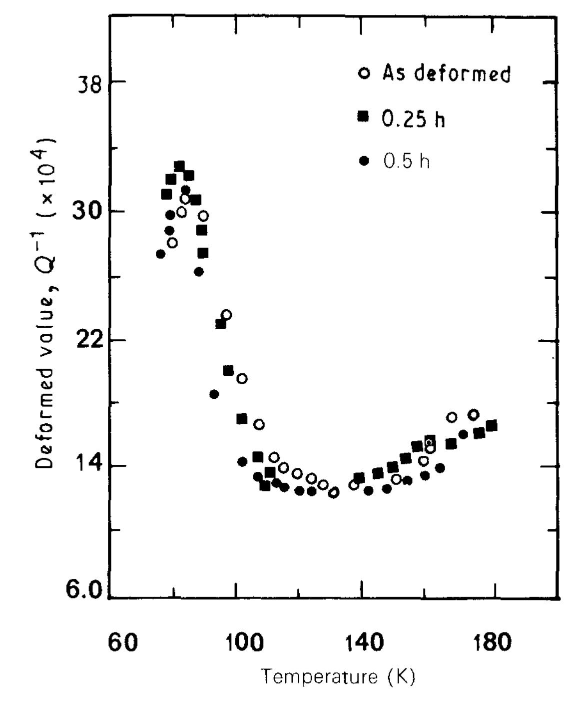

Figure 6 The effect of isothermal annealing time (0.25 h, 0.5 h) at 323 K.

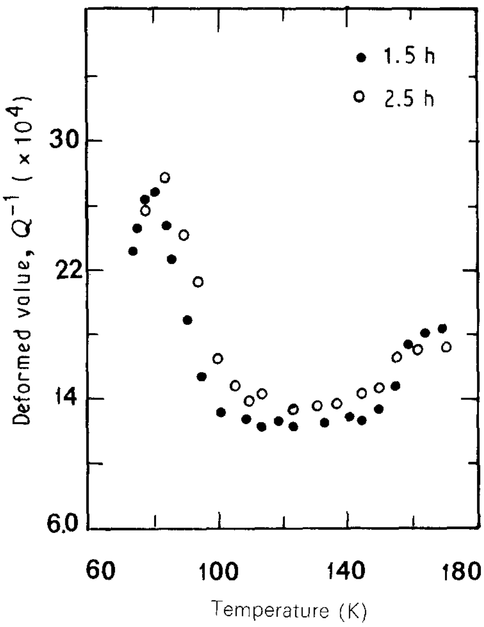

Figure 7 The effect of isothermal annealing time (1.5 h, 2.5 h) at 323 K.

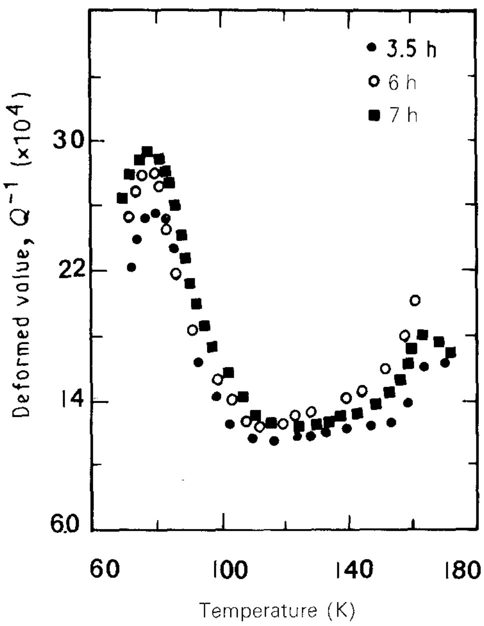

Figure 8 The effect of isothermal annealing time (3.5 h, 6 h, 7 h) at 323 K.

The peak height of the specimen deformed by 10.65% (18×10^{-4}) was appreciably greater than those of other specimens. The Bordoni peak height initially increases with annealing at 323 K attaining a value of 19.6×10^{-4} after an anneal time of 0.25 hours. Thereafter, further annealing causes a reduction in the peak height to a value of 13.68×10^{-4} after 3.5 hours. However, extended annealing up to 16 hours increases the peak height to a maximum value of 23×10^{-4}.

The general behaviour of the Bordoni peak in copper annealed at 323 K and 298 K is similar. Annealing subsequent to deformation results in a small increment of the peak; the higher annealing temperature producing the greater effect. The peak height

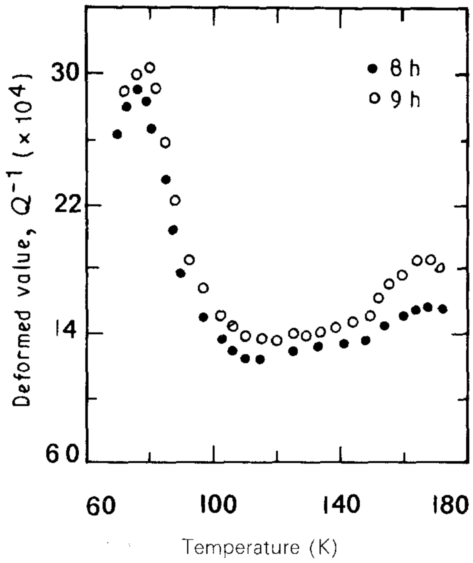

Figure 9 The effect of isothermal annealing time (8 h, 9 h) at 323 K.

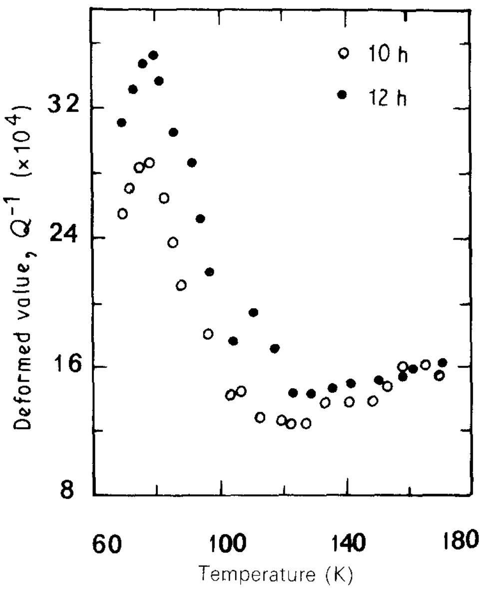

Figure 10 The effect of isothermal annealing time (10 h, 12 h) at 323 K.

decreases after about half an hour at either temperature. The rate of decrease is greater for the 323 K anneal; nonetheless both treatments decrease the height by about 30%. The general effect of an increase in annealing temperature on the position of the maxima and minima is to reduce the times at which they occur.

(iii) Annealing at 373 K

A specimen subjected to the same primary annealing treatment and deformation conditions as described in the previous instances was isothermally annealed at 373 K in an argon atmosphere for certain time intervals. The internal friction was measured in the range 77 K and 170 K at a frequency of 470 Hz. The experimental data obtained is presented in Figs 12–18.

Immediately after deformation the peak height decreased to its first minimum of 8.85×10^{-4} following an anneal of one hour. The peak then regrew to attain

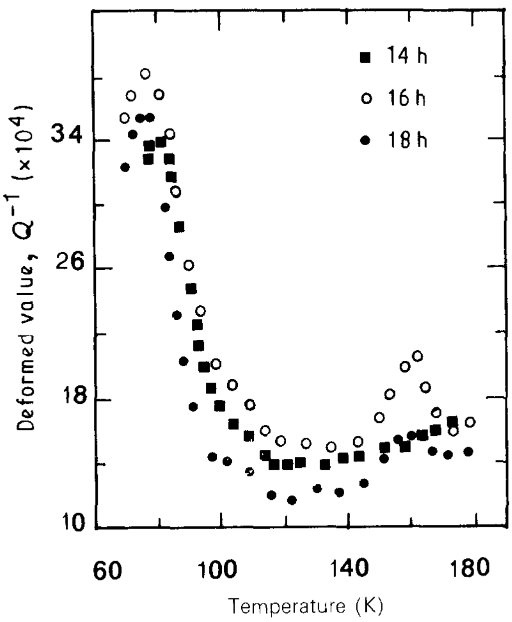

Figure 11 The effect of isothermal annealing time (14 h, 16 h, 18 h) at 323 K.

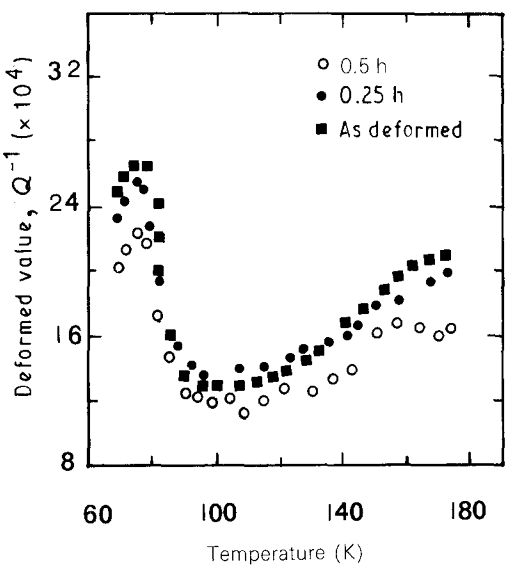

Figure 12 The effect of isothermal annealing time (0.25 h, 0.5 h) at 373 K.

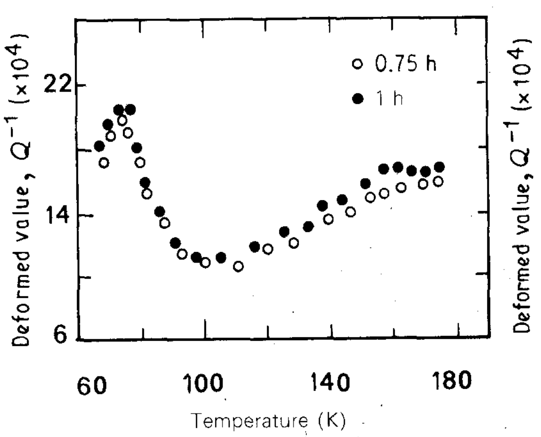

Figure 13 The effect of isothermal annealing time (0.75 h, 1 h) at 373 K.

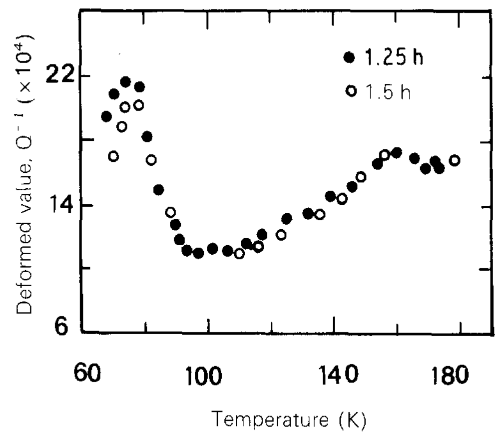

Figure 14 The effect of isothermal annealing time (1.25 h, 1.5 h) at 373 K.

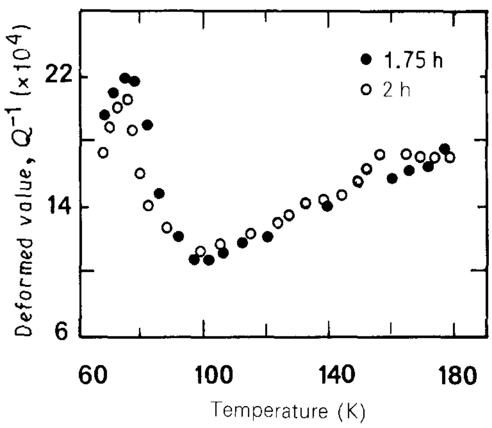

Figure 15 The effect of isothermal annealing time (1.75 h, 2 h) at 373 K.

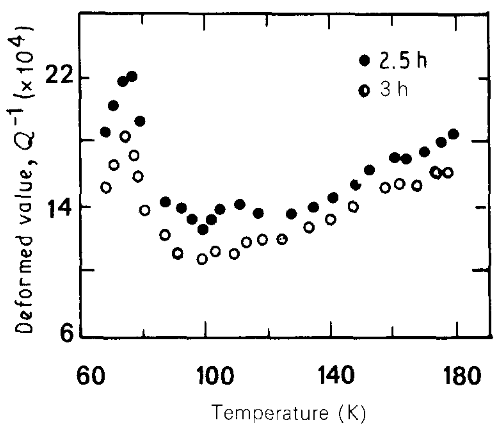

Figure 16 The effect of isothermal annealing time (2.5 h, 3 h) at 373 K.

a height of 10.8×10^{-4} after 1.75 hours at 373 K. Upon further annealing the peak height passes through a minimum (7.55×10^{-4}) and then a maximum (9.2×10^{-4}) value after 3 and 5.5 hours respectively.

It is interesting to note that annealing at 373 K does not bring about an immediate growth of the peak but instead causes a decrease. Apart from this observation the internal friction behaviour at all three annealing temperatures is strikingly similar. Increasing the annealing temperature decreases the time at which the

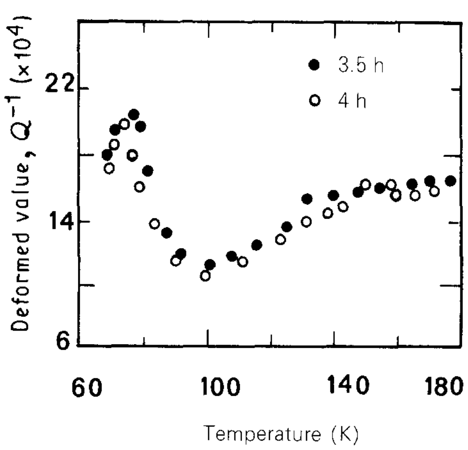

Figure 17 The effect of isothermal annealing time (3.5 h, 4 h) at 373 K.

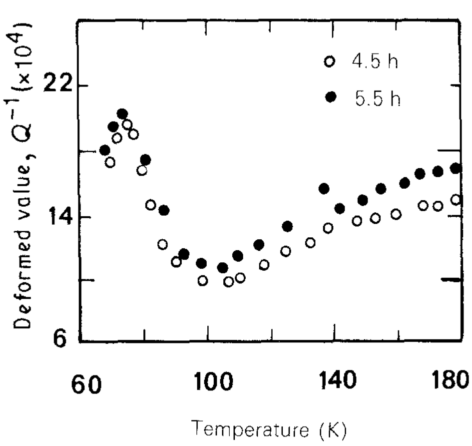

Figure 18 The effect of isothermal annealing time (4.5 h, 5.5 h) at 373 K.

maxima and minima occur. The initial decrease, once more, is of the order of 30%.

(iv) Annealing at 423 K

A fourth specimen was similarly annealed in a vacuum at 873 K for 3 hours and then deformed 10.2% by cold rolling. Subsequent isothermal annealing was carried out in argon at 423 K for 6 hours. The specimen was withdrawn from the furnace at various times during annealing and its internal friction was measured in the temperature range 77 K and 170 K. The frequency of vibration was 550 Hz. The results are shown in Figs 19–23.

The peak height experiences a sharp decrease from a deformed value of 13.9×10^{-4} to 5.7×10^{-4} following annealing for 0.5 hours. However, the peak regrows and attains a maximum height of 13.78×10^{-4} after annealing for 1 hour. Upon further annealing, the peak height decreases once again and passes through a minimum of 10.75×10^{-4} after 2.75 hours. Annealing beyond this point causes a small increase in the peak height after 4.25 hours. This is followed by a steady decrease.

The results of this treatment are consistent with those obtained earlier as the higher annealing temperature shortens the time to attain the maxima and minima.

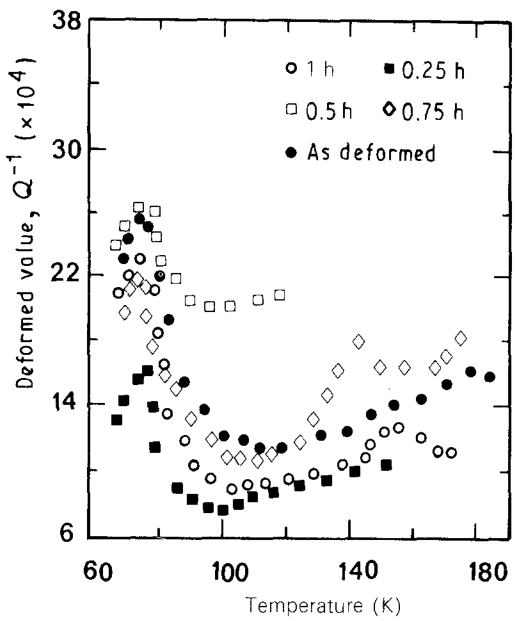

Figure 19 The effect of isothermal annealing time (0.25 h, 0.5 h, 0.75 h, 1 h) at 423 K.

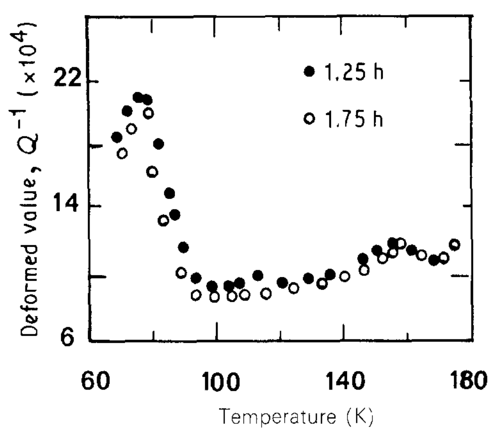

Figure 20 The effect of isothermal annealing time (1.25 h, 1.75 h) at 423 K.

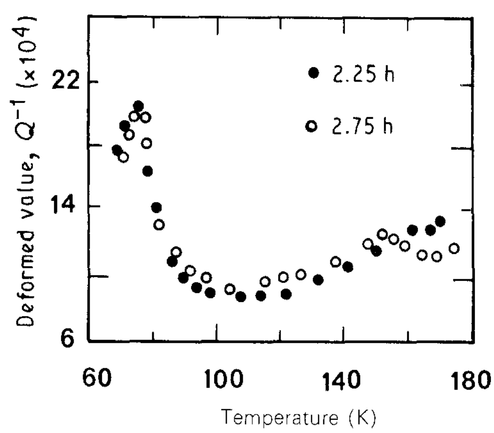

Figure 21 The effect of isothermal annealing time (2.25 h, 2.75 h) at 423 K.

A summary of the isothermal annealing behaviour of the Bordoni peak in copper between 298 and 423 K is presented in Fig. 24. Isothermal annealing reveals two distinct features:

(a) The peak height initially decreases and then increases except for the specimen annealed at 323 K

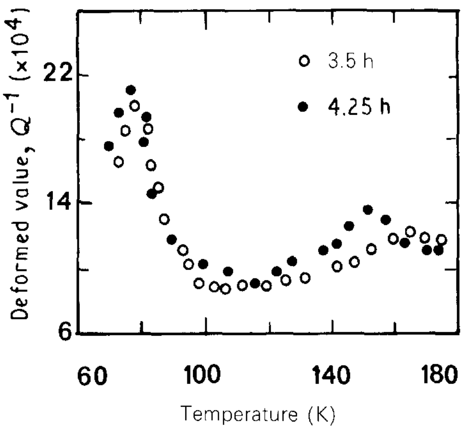

Figure 22 The effect of isothermal annealing time (3.5 h, 4.25 h) at 423 K.

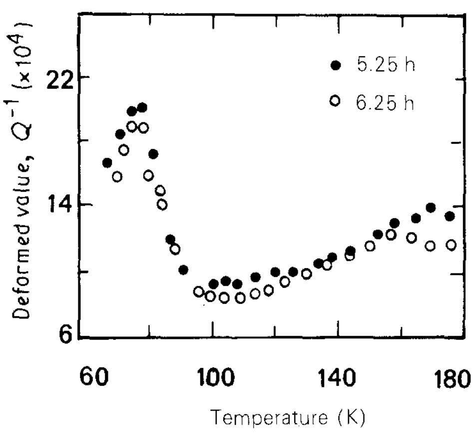

Figure 23 The effect of isothermal annealing time (5.25 h, 6.25 h) at 423 K.

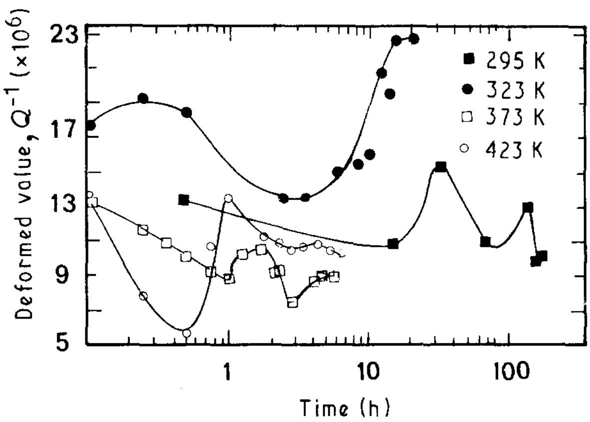

Figure 24 Isothermal annealing of the Bordoni peak in copper in the range 295–423 K.

(b) The general effect of an increase in the annealing temperature is to reduce the times at which the maxima and minima occur.

Many investigators have shown the height of the Bordoni peak to decrease upon annealing [18–21]. The increase in Bordoni peak height before it decreases as observed in the specimen annealed at 323 K is in agreement with the findings of Niblett and Wilks [3, 22] who showed that there is a range of annealing temperatures in which the peak increases in height before it decreases. The magnitude of this increase depends quite critically on the grain size of the specimen. Bruner and Mecs [23, 24] also observed an increase in Bordoni peak height when annealed above and below room temperature, respectively, after deformation at 4.2 K.

The initial decrease in the peak height is followed by an increase reaching a maximum before a second decrease. A similar increase in peak height has also been observed by Okuda [21] and Bruner and Mecs [24] during isochronal annealing of polycrystalline copper after deformation at 4.2 K.

Further isothermal annealing of copper causes the Bordoni peak height to decay reaching a second minimum before increasing again. Isothermal annealing at 323 K was not pursued beyond 18 hours, hence the second minimum was not observed. Thus, the present results suggest the annealing behaviour to be complex.

As pointed out earlier increasing the annealing temperature tends to produce a systematic decrease in the time at which the maxima and minima occur, which in turn implies a thermally activated process to be at work. The peak temperature too is influenced by the annealing process. The present results indicate that annealing moves the Bordoni peak to a lower temperature thus confirming the findings of Niblett and Wilks [3] and Okuda [21]. Bruner and Mecs [23] and Okuda [21], however, showed that the Bordoni peak moves to higher temperatures during low temperature annealing. This seemingly contradictory behaviour is thought to arise from the changing proportion of dislocations of different types which may affect the strength of individual relaxations in the relaxation spectrum.

Speculative interpretations of the reduction and regrowth of the Bordoni peak height during annealing have been suggested by many workers. Bruner and Mecs [24] proposed a model for the Bordoni peak based upon the thermally activated motion of paired partial dislocations between vacancy pinning points and attributed the reduction in the Bordoni peak to the migration of defects other than vacancies to dislocations; thus attenuation would result from the reduction of the average length of the free vacancy pins. The regrowth of the Bordoni peak height could be due to the migration of more vacancies to dislocation lines.

Okuda [21] attempted to explain the reduction in peak height during low temperature annealing in terms of deformation-induced point defects migrating to and pinning the dislocation lines. This increase in the concentration of the pinning points suppresses kink pair formations and therefore the peak height decreases. Regrowth of the Bordoni peak on annealing is considered to be associated with a decrease in density of pinning points on the dislocation lines or disappearance of the pinning points. Plastic deformation generates various kinds of crystal defects such as dislocations, interstitials, vacancies and their clusters. Depending on their nature, these defects may anneal at different temperatures, the more mobile one annealing at lower temperatures. During annealing, these defects would migrate to dislocation lines and pin them down. As the annealing temperature increases, defects would evaporate, become annihilated or cluster together.

There is some disagreement among investigators concerning the annealing temperature range of point defects and interstitials in particular. Okuda [21] maintains that annealing up to 180 K causes a decay and regrowth of the Bordoni peak in copper and gold due to pinning and unpinning of dislocations by interstitials. He further suggests that annealing above 220 K causes a different kind of pinning process, which may be ascribed to divacancies and later to single vacancies.

Mecs and Nowick [25] consider the decay of the Bordoni peak in copper to be due to a shortening of the effective length of dislocation loops taking part in the Bordoni relaxation.

3.1. A dislocation model

If it is considered that the decay and subsequent regrowth of the Bordoni peak after annealing at temperatures above room temperature are due to the diffusion of defects to dislocations and their subsequent removal from dislocations, respectively, then a simplified model can be formulated. At low temperatures removal of defects occurs by diffusion along dislocations to jogs [26]. If the movement of defects to dislocations is greater than the movement of defects along dislocations then the height of the Bordoni peak will decrease. Conversely, if the movement of defects to dislocations is less than the movement of defects moving along dislocations the height of the Bordoni peak will increase. A minimum should occur when the rate of defect diffusion to dislocations is equal to the rate of disappearance of defects along dislocations.

We now proceed to present the mathematical treatment of the model as follows: The energy of interaction, E, of a defect at a distance r from a dislocation may be written as

$$E = E_{\mathrm{m}} \left(\frac{b}{r} \right)^{n} \tag{2}$$

where E_{m} is the binding energy and b is the lattice parameter. In the stress field of the dislocation the defect experiences a force

$$F = -\mathrm{d}E/\mathrm{d}r = -n\, b^{-1}\, E_{\mathrm{m}}\, (b/r)^{n+1} \tag{3}$$

If the diffusion coefficient for the defect is D its mobility will be $D/k\mathrm{T}$ (k is Boltzmann's constant and T is the absolute temperature), and its drift velocity will be

$$\mathrm{d}r/\mathrm{d}t = v = D\,F/k\mathrm{T} = -(n\,E_{\mathrm{m}}\,D/bk\mathrm{T})\,(b/r)^{n+1} \tag{4}$$

Integration and taking $r = 0$ when time $t = 0$ yields

$$(r/b)^{n+2} = -n(n+2)\,E_{\mathrm{m}}\,Dt/b^2\,k\mathrm{T} \tag{5}$$

If the migration time started at $-t$, the defects which arrive at dislocation when $t = 0$ are those which were originally at a distance less than r given by equation 5. Let $C + C_{\mathrm{o}}$ be the initial concentration of defects, and C_{o} be the equilibrium concentration of defects. Assuming that the defects at a dislocation are confined to a cylinder of cross-sectional area b^2 then the concentration, C_{I}, of defects which have arrived at the dislocation will be given by the well known Cottrell-Bilby relation

$$C_{\mathrm{I}} = \pi\, r^2 C/b^2 = C[n(n+2)E_{\mathrm{m}}\,Dt/b^2\,k\mathrm{T}]^{2/(n+2)} \tag{6}$$

The rate, R, at which defects reach unit length of dislocation may be written as

$$R = \left(\frac{1}{b}\right)\left(\frac{\mathrm{d}C_{\mathrm{I}}}{\mathrm{d}t}\right) \quad \text{or}$$

$$R = \frac{2\,\pi C}{b(n+2)t}\,[n(n+2)E_{\mathrm{m}}\,Dt(b^2 k\mathrm{T})^{-1}]^{2/(n+2)} \tag{7}$$

The value of n will depend on the relative magnitude of the size effect or elastic constant effect. Friedel has demonstrated the value of n to equal unity [27]. In that event equation 7 reduces to

$$R = \frac{2}{3}\left(\frac{\pi C}{bt}\right)[3E_{\mathrm{m}}\,Dt(b^2\,k\mathrm{T})^{-1}]^{2/3} \tag{8}$$

The rate of disappearance of the defects can be obtained by a similar model. Let L be the average distance between jogs on a dislocation. Assuming the equilibrium concentration of defects near the jogs to be C_{o} a concentration gradient of the order C_{I}/L would be set up along the dislocation. If the diffusion coefficient for the migration of defects along the dislocation is D_1 then the velocity V_1 of defects would be given by $V_1 = D_1 C_{\mathrm{I}}/Lb$. The rate R_1 of defect removal from a unit length of the dislocation is

$$R_1 = D_1 C_{\mathrm{I}}/L^2 b \tag{9}$$

The annealing behaviour of the Bordoni peak in the present study may now be elucidated as follows. According to the currently accepted theories the Bordoni peak height is proportional to the dislocation loop length between pinning defects. Hence as the rate of defect diffusion to dislocations or the rate of defect migration along dislocation becomes preponderant the Bordoni peak height would experience a decrease or regrowth, respectively. Fig. 24 illustrates a fall in the peak height upon annealing in the range 298–423 K and a subsequent regrowth. This produces the first minimum in the annealing curves, which occurs when $R = R_1$. Thus equating (8) and (9) yields the following equation for the minimum time

$$t_{\min} = \frac{2}{3}\left(\frac{L^2}{D_1}\right) \tag{10}$$

$$D_1 = D_{\mathrm{o}1}\,\mathrm{e}^{-Q_1(k\mathrm{T})^{-1}} \tag{11}$$

where Q_1 is the activation energy and $D_{\mathrm{o}1}$ is the equilibrium concentration value of D_1. Substituting for D_1 in equation (10) gives

$$t_{\min} = \frac{2}{3}\left(\frac{L^2}{D_{\mathrm{o}1}}\,\mathrm{e}^{Q_1(k\mathrm{T})^{-1}}\right) \tag{12}$$

A plot of log $t_{\min}$ against $1/\mathrm{T}$ is a straight line as shown in Fig. 25. From measurement of the slope Q_1/k the value of Q_1 is found to be 0.27 eV.

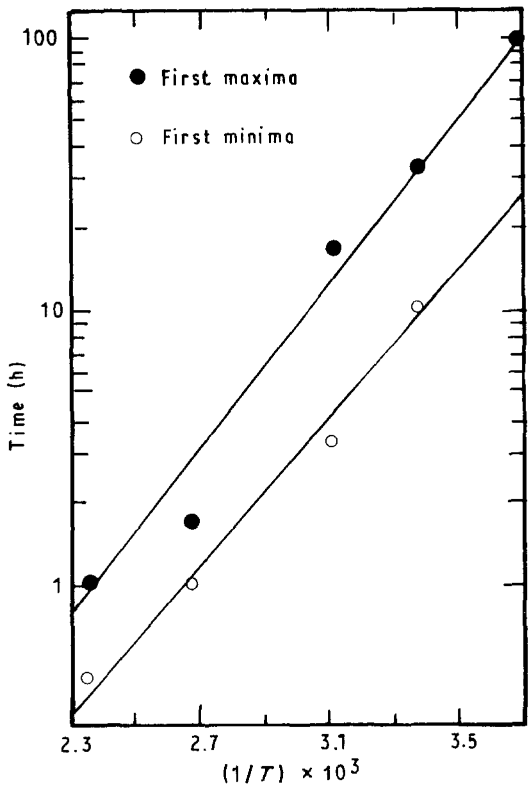

Figure 25 First maxima and first minima versus $1/T$.

It is also possible to analyse the decay and the subsequent regrowth of the Bordoni peak height by employing the same model.

The net rate of increase R_N of defects at a dislocation is given by

$$R_N = R - R_1 \quad (13)$$

$$R_N = \frac{1}{b}\left(\frac{dC_N}{dt}\right) \quad (14)$$

where C_N is the net concentration of defects. The concentration of defects at the dislocation after time t is therefore

$$C_N = \int_0^t \left\{\left(\frac{2}{3}\right)\left(\frac{\pi C}{t}\right)[3E_m Dt/b^2 kT]^{2/3} - \frac{D_1 \pi C}{L^2}(3E_m Dt/b^2 kT)^{2/3}\right\} dt,$$

or

$$C_N = \pi C(3E_m Dt/b^2 kT)^{2/3} - \frac{3D_1\pi C}{5L^2}(3E_m Dt/b^2 kT)^{2/3} t \quad (15)$$

During the initial period of the decrease in peak height, the second term will be small and

$$C_N = \pi C(3E_m Dt/b^2 kT)^{2/3} \quad (16)$$

The height of the Bordoni peak Q_B as given by the theories of both Seeger *et al.* [28] and Brailsford [29, 30] is dependent on the third power of the dislocation loop length, L_a. That is $Q_B^{-1} \propto L_a^3$ or

$$(Q_B^{-1})^{1/3} \propto L_a \quad (17)$$

During the pinning process the average length, L_a, of dislocation can be taken as

$$\frac{1}{L_a} = \frac{1}{L_o} + \frac{C_N}{b} \quad (18)$$

$$L_a = L_o/(1 + L_o C_N/b) \quad (19)$$

where L_o is the average initial length of loops. Substituting for L_a in (17) gives

$$(Q_B^{-1})^{1/3} \propto L_o/(1 + L_o C_N/b) \quad \text{or}$$

$$(Q_B^{-1})^{1/3} \propto L_o\left(1 - \frac{L_o}{b}C_N + \ldots\ldots\right) \quad (20)$$

substituting equation (16) in (20) yields

$$(Q_B^{-1})^{1/3} \propto L_o\left[1 - \frac{L_o \pi C}{b}(3E_m Dt/b^2 kT)^{2/3}\right] \quad (21)$$

Hence a plot of $(Q_B^{-1})^{1/3}$ versus $t^{2/3}$ should be a straight line with a slope proportional to

$$\frac{L_o^2 \pi C}{b}(3E_m D/b^2 kT)^{2/3} \quad (22)$$

Fig. 26 illustrates the linear relationship between $(Q_B^{-1})^{1/3}$ and $t^{2/3}$ at the annealing temperatures 298, 323, 373, and 423 K.

Now $D = D_o\, e^{-Q/kT}$ where Q is the activation energy for the diffusion of defects to dislocations. From equation (22)

$$\text{slope} \times T^{2/3} \propto (e^{-Q/kT})^{2/3} \quad (23)$$

The slopes are computed from Fig. 26. A plot of log (slope $\times\, T^{2/3}$) versus 1/T should be linear with a slope given by $-2Q/3k$ (Fig. 27). The results yield a linear plot of the slope which gives an activation energy of 0.55 eV.

After the first minimum the peak height increases. According to the simple model presented here this means that a greater number of defects leave than arrive at dislocations. In this instance defect concentration at dislocations is controlled by the second term in equation (15), that is

$$C_N = -\frac{3D_1 \pi C}{5L^2}\left(\frac{3E_m Dt}{b^2 kT}\right)^{2/3} t \quad (24)$$

Substituting for C_N in (20) yields

$$(Q_B^{-1})^{1/3} \propto L_o\left[1 + \frac{3L_o D_1 \pi C}{5bL^2}\left(\frac{3E_m Dt}{b^2 kT}\right)^{2/3} t\right] \quad (25)$$

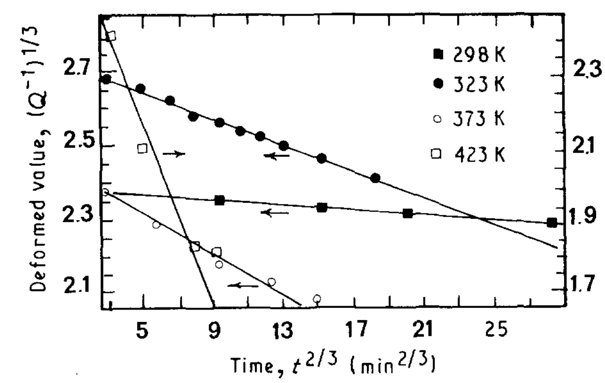

Figure 26 First decrease of the Bordoni peak $(Q^{-1})^{1/3}$ versus $(t)^{2/3}$.

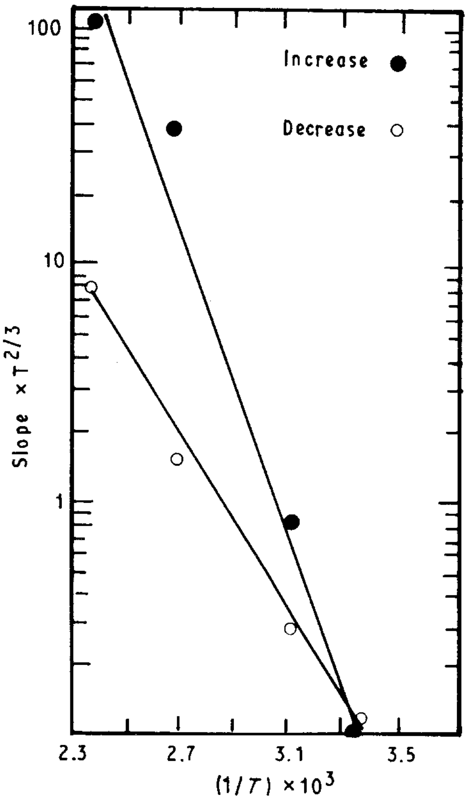

Figure 27 Slope of the first increase $\times\,T^{2/3}$ and slope of the first decrease $\times\,T^{2/3}$ versus $1/T$.

Hence a plot of $(Q_B^{-1})^{1/3}$ versus $t^{5/3}$ should be linear with a slope equal to

$$\frac{3L_o^2 D_1 \pi C}{5bL^2}\left(\frac{3E_m D}{b^2 kT}\right)^{2/3} \tag{26}$$

Fig. 28 shows such plots for the annealing temperatures 298, 323, 373 and 423 K. Now from equation (25)

$$\text{slope} \times T^{2/3} = \frac{3L_o^2 D_1 \pi C}{5bL^2}\left(\frac{3E_m D}{b^2 k}\right)^{2/3}$$

and (11)

$$D = D_o e^{-Q/kT} \quad \text{and} \quad D_1 = D_{01}\, e^{-Q_1(kT)^{-1}}$$

$$\log(\text{slope} \times T^{2/3}) =$$

$$\log\left[\frac{3L_o^2 \pi C D_{01}}{5bL^2}\left(\frac{3E_m D_o}{b^2 k}\right)^{2/3} - \frac{Q_1}{kT} - \frac{2Q}{3kT}\right] \tag{27}$$

A plot of log (slope $\times\,T^{2/3}$) against $1/T$ should be a straight line with a slope equal to $-1/k(Q_1 + 2/3Q)$. Fig. 27 shows that the results fit the equation and a slope of 2.9 is obtained. Hence the calculated activation energy, Q_1 is 0.24 eV.

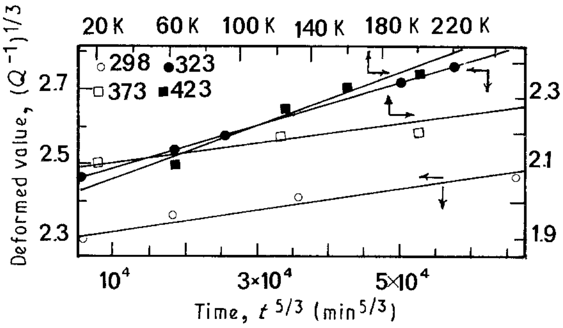

Figure 28 First increase of the Bordoni peak $(Q^{-1})^{1/3}$ versus $(t)^{5/3}$.

A similar analysis of the first maximum in the annealing curves of Fig. 24 may be carried in a similar way. If it is assumed that a second type of defect migrates to dislocations, then the first maximum should occur when the rate R of arrival of the second type of defects to dislocations is given by

$$R = \frac{2\pi C}{3bt}\left(\frac{3E_{m2} D_2 t}{b^2 kT}\right)^{2/3} \tag{28}$$

subscript 2, i.e. D_2, denotes quantities for the second type of defects. The rate R_1 of disappearance of the first defect along dislocation lines is described by equation (9)

$$R_1 = \frac{D_1 \pi C}{L^2 b}\left(\frac{3E_m Dt}{b^2 kT}\right)^{2/3} \tag{29}$$

The maximum occurs when $R = R_1$. Hence,

$$\left(\frac{2}{3}\right)\left(\frac{\pi C}{bt}\right)\left[\frac{3E_{m2} D_2 t}{b^2 kT}\right]^{2/3} =$$

$$\frac{D_1 \pi C}{L^2 b}\left(\frac{3E_m Dt}{b^2 kT}\right)^{2/3} \quad \text{or}$$

$$\frac{2}{3t}(E_{m2} D_2)^{2/3} = \frac{D_1}{L^2}(E_m D)^{2/3}. \quad \text{Hence}$$

$$t = \frac{2L^2}{3D_1}\left(\frac{E_{m2}}{E_m}\right)^{2/3} \times \left(\frac{D_2}{D}\right)^{2/3} \tag{30}$$

$$D_1 = D_{01}\, e^{-Q_1/kT}$$

$$D = D_o\, e^{-Q/kT}$$

$$D_2 = D_{02}\, e^{-Q_2/kT}$$

Substituting for D_1, D and D_2 in equation (30) gives

$$t = \frac{2L^2}{3D_{01}}\left(\frac{E_{m2}}{E_m}\right)^{2/3}\left(\frac{D_{02}}{D_o}\right)^{2/3}$$

$$\times\, e^{Q_1/kT}\, e^{(2/3)Q(kT)^{-1}}\, e^{-(2/3)Q_2(kT)^{-1}}$$

Hence

$$\log t = \log\left[\frac{2L^2}{3D_{01}}\left(\frac{E_{m2}}{E_m}\right)^{2/3}\left(\frac{D_{02}}{D_o}\right)^{2/3}\right]$$

$$-\frac{1}{kT}\left[\frac{2Q_2}{3} - \frac{2Q}{3} - Q_1\right] \tag{31}$$

Hence a plot of the log of the time for the first maximum against the reciprocal of the annealing temperature should be a straight line with a slope of

$$-\frac{1}{k}\left(\frac{2Q_2}{3} - \frac{2Q}{3} - Q_1\right)$$

The above plot is illustrated in Fig. 25. The measured slope is 1.52. Hence $Q_2 = 0.49$ eV.

The activation energy calculated from experimental data for the diffusion of type I defects to dislocations and for the diffusion of type I defects along dislocations in copper are thus shown to be 0.55 eV and 0.25 eV, respectively. Similarly, the activation energy deduced for the second type of defects to diffuse to dislocations is 0.49 eV. Theoretical activation energies for the migration of defects have been computed by many workers. The activation energy values for the diffusion of vacancies, divacancies and interstitials vary between 0.97–1.3 eV, 0.35–0.7 eV and 0.05–0.5 eV, respectively [31]. Theoretical and experimental values computed for the migration energy of a divacancy are 0.35 eV [32] and 0.68 eV [31], respectively. Bowen *et al.* [32] calculated the migration energy to be 0.52 ∓ 0.1 eV. De Batist [8] suggests that the activation enthalpy for the migration of divacancies is comparable to that of an interstitial.

Thus experimental evidence suggests that the defects responsible for the decrease and increase in the height of the Bordoni peak in copper are interstitials and divacancies. It is not possible at this stage to identify the type of defects which gives rise to the initial and second decrease in the Bordoni peak height since information on the activation energies for the diffusion of defects to and along dislocations is limited.

4. Conclusions

Isothermal annealing of the Bordoni peak at different temperatures can be accounted for by the migration of defects to and along dislocations. When the rate of defect migration to dislocations is greater than that of defect migration along dislocations, the density of pinning points on dislocations increases resulting in a decrease in the Bordoni peak height. Likewise, when the rate of diffusion of defects to dislocations is exceeded by the rate of defect migration along dislocations the density of pinning points on dislocations decreases, i.e. depinning occurs resulting in an increase in the height of the Bordoni peak. Due to this increase or decrease in the density of pinning defects on dislocations the Bordoni peak height decreases or increases, respectively. Thus the defect-dislocation interaction phenomenon manifests itself in the occurrence of minima and maxima in the internal friction spectrum of the material.

The activation energies for the first decrease and first increase in the Bordoni peak height are calculated to be 0.55 eV and 0.24 eV, respectively. The value for the activation energy of the first minimum is 0.27 eV. These figures are in agreement with the activation energy values for interstitial and divacancy diffusion estimated by different investigators. An activation energy of 0.49 eV for the second type of defect was computed from the first maximum. Hence, the second type of defect was identified as divacancy.

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