INTERNAL FRICTION BEHAVIOUR OF COLD-WORKED COPPER IN THE Hz-RANGE DURING STEPLIKE HEATING

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Abstract - A steplike heating after low temperature deformation of copper has been used to separate the reversible relaxation processes during the temperature step from the irreversible pinning effects during the isothermal annealing times. The substructure of pinning stage II and the behaviour of the damping peaks are discussed.

I - INTRODUCTION

At about 1 Hz and during heating after low-temperature deformation copper exhibits a superposition of the Hasiguti peaks and several pinning stages (Fig.1) /1,2/. The correlation between the damping peaks and the pinning stages has been emphasized /3-5/ and it was not excluded that the subdivision into the pinning stages II_A and II_B might only be due to the maximum in the differentiated modulus curve that represents the modulus defect of P_2 (lower part of Fig.1) /3/. Indeed, Seyed Reihani et al. /6/ found a single stage II from 100 to 200 K, measuring the modulus of deformed copper at 20 K after isochronal anneals. However, the 30 K-steps used allowed only a very rough differentiation of the modulus and details like the subdivision into stages II_A (144 K) and II_B (195 K) might have been missed. Therefore, in the present experiments 10 K-steps were used to obtain a more precise differentiation. Further a stepwise method of heating after deformation was applied to separate the temperature-dependent relaxation effects in the steps from the time-dependent pinning or depinning effects during the isothermal annealing times.

II - EXPERIMENTAL PROCEDURE

At about 2 Hz modulus and decrement were measured with an inverted torsion pendulum /7/ after 7% tensile deformation at about 100 K. The samples of 99.999% ASARCO-copper were annealed for 2 h at 1.173 K in oxidizing and reducing atmospheres /2/. For the measurement two methods of heating were used: (i) linear heating with 0.87 K/min at a strain amplitude of $2x10^{-6}$ and (ii) stepwise heating with a mean heating rate of 0.8 K/min at a strain amplitude of 1×10^{-5}. The second method is described schematically in Fig.2. The lower part depict the temperature step. During 1 minute the temperature was increased by ΔT = 10 K. Due to this rapid heating the mechanical noise inside the pendulum cryostat was increased, so that the measuring amplitude was limited to 1×10^{-5}. The temperature step was followed by an isothermal anneal for 13 minutes. Two changes of decrement δ and modulus G were observed (middle and upper part of Fig.2): $(\Delta\delta)_{step}$ and $(\Delta G)_{step}$ during the steps, $(\Delta\delta)_{isoth}$ and $(\Delta G)_{isoth}$ during the isothermal anneal. In order to reproduce the heating time of 1 minute it was only possible to obtain ΔT = 10 within $\pm$ 2 K, e.g. not all steps had the same height. Therefore it is necessary to normalize the observed changes to the height of each step. This corresponds to a rough differentiation with respect to temperature:

$$g_{total} = g_{step} + g_{isoth} = -\frac{1}{G}\left(\frac{\Delta G}{\Delta T}\right)_{step} - \frac{1}{G}\left(\frac{\Delta G}{\Delta T}\right)_{isoth} \qquad (1)$$

Similar quantities can be derived from the decrement changes.

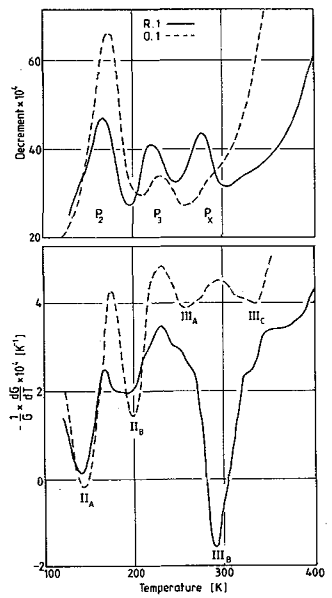

Fig.1: Decrement (upper part) and differentiated modulus (lower part) after 7% tensile deformation at about 100 K for the reduced sample R.1 (full curves) and the oxidized sample 0.1 (dotted curve) during linear heating.

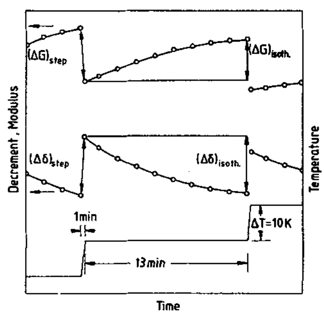

Fig.2: Schematic diagram for stepwise heating; time dependence of temperature (lower curve), of decrement (middle curve) and of modulus (upper curve).

III - EXPERIMENTAL RESULTS

Results during linear heating are pedicted in Fig.1 for the oxidized sample 0.1 (dotted curves) and the reduced sample R.1 (full curves). In the upper part the decrement curves show the Hasiguti peaks P_2 and P_3 with different height for both samples and an additional peak P_x for R.1. The differentiated modulus curves in the lower part give marked differences for stage III in both samples: small stages III_A and III_C for 0.1 and a very large stage III_B for R.1.

For about the same mean heating rate a comparison of the differentiated modulus curves during linear heating (dotted curves) and during stepwise heating (g_{total} according to Eq.(1), full curves) is given in the next figures: for the samples 0.1 and 0.2 in Fig.3 and for R.1 and R.2 in Fig.4. The curves for both methods of heating coincide, the existing differences are mainly due to the coarser method of differentiation in the case of stepwise heating. The density of measuring points used for the linear heating results is higher by a factor of 4. A similar agreement was found for the decrement curves.

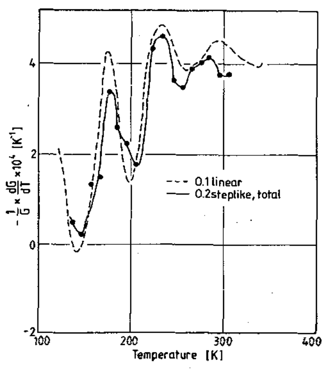

Fig.3: Differentiated modulus for linear heating (dotted curve) and stepwise heating (full curve) for the oxidized samples 0.1 and 0.2

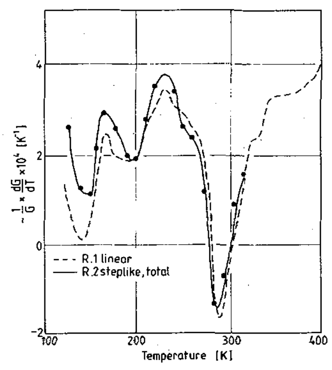

Fig.4: Differentiated modulus for linear heating (dotted curve) and stepwise heating (full curve) for the reduced samples R.1 and R.2

For the samples 0.2 (dotted curve) and R.2 (full curve) the quantity g_{step} is depicted in the upper part of Fig.5. The pinning stages of the reduced sample R.2 can be seen even during the steps. This indicates that here during the 1 minute-steps of temperature increase already changes in the state of pinning occur. The maxima in these curves correspond to the modulus defects of the damping maxima. The lower part of Fig.5 gives g_{isoth} from the isothermal anneals. It can be recognized from the comparison with the upper curves that dislocation pinning, e.g. the minima in the g-curves, occurs mainly during the isothermal annealing times. Further for R.2 over the whole temperature range pinning is found ($g_{isoth} < 0$). For 0.2, however, at 226 K and between 278 K and 288 K there exists no pinning ($g_{isoth} = 0$).

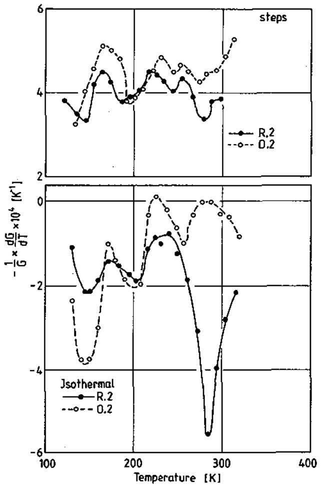

Fig.5: $g = -1/G(\Delta G/\Delta T)$ for the steps (upper part) and for the isothermal annealing times (lower part); oxidized sample 0.2 (dotted curve) and reduced sample R.2 (full curve).

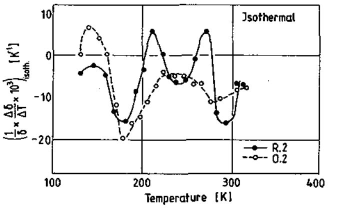

Fig.6: Isothermal decrement changes $1/\delta(\Delta\delta/\Delta T)_{isoth}$ for the oxidized sample 0.2 (dotted curve) and the reduced sample R.2 (full curve).

The isothermal changes of decrement, e.g. $1/\delta(\Delta\delta/\Delta T)_{isoth}$, are shown in Fig.6. In the dotted curve for 0.2 one can recognize for P_2 (T_{max} = 170 K) an increase of decrement up to 160 K, followed by a decrease with a maximum decreasing rate at 188 K. Above P_2 an increase and decrease of P_3 (T_{max} = 230 K) exists, but there is a general decrease of decrement ($1/\delta(\Delta\delta/\Delta T)_{isoth} < 0$). For R.2 (full curve) the changes of P_2 are smaller than in 0.2, because this sample has a smaller peak P_2 (see Fig.1). The increase of P_3 (T_{max} = 220 K) has a maximum at 213 K and the maximum decreasing rate is observed at 238 K. P_x (T_{max} = 273 K) has its maximum increase at 272 K and its maximum decreasing rate at 294 K.

IV - DISCUSSION

(i) For about the same mean heating rate the method of stepwise heating (g_{total} in Fig.3 and 4) gives the same behaviour of the differentiated modulus. Thus both methods reproduce the well-known characteristic differences of internal friction behaviour after deformation between oxidized and reduced samples /2/.

(ii) For the subdivision of the stages II_A and II_B we will consider sample 0.2, which has the larger peak P_2. In Fig.5 both stages are separated by a maximum in g_{isoth} at 171 K. This represents, at least in part, the decrease of P_2. The fact that this maximum does not coincide with the maximum decreasing rate of P_2 at 188 K in Fig.6 is certainly due to the superimposed pinning of stage II_B. The advantage of the present method is, that the isothermal changes of decrement have been measured and can be used to obtain the corresponding modulus changes. For the maximum in g_{isoth} between the stages II_A and II_B we have $g_{isoth} = g_{isoth}(pinning) + g_{isoth}(P_2)$, where the second contribution represents the modulus change due to the decrease of P_2. It is obvious that $g_{iosth}(P_2) > 0$. From Fig.6 we find for T > 160 K that $1/\delta(\Delta\delta/\Delta T)_{isoth} < 0$. Thus we get for the maximum in g_{isoth} at 171 K: $(\Delta\delta/\Delta G/G)_{isoth} > 0)$. For P_2 it will be assumed that it is caused by the interaction of stage II_A-defects with the dislocation, e.g. the relaxation is due to the diffusion of these defects with the moving dislocations /2,3,5/. Two models have been discussed for the decrease of P_2 /5/: (i) The disappearance of the stage II_A-defects from the dislocations (depinning, $(\Delta\delta/\Delta G/G)_{isoth} > 0$) and (ii) pinning by stage-II_B-defects ($(\Delta\delta/\Delta G/G)_{isoth} < 0$). The observed ratio $(\Delta\delta/\Delta G/G)_{isoth} < 0$ corresponds to model (1), which is additionally supported by the observation that after an 80 K-electron-irradiation the irradiation-induced pinning and the damping peak P_2 associated with this pinning recover by irreversible depinning at 170 K /8/. For model (i) we have /8/

$$(\Delta\delta/\Delta G/G)_{isoth} = \pi/\omega\tau \qquad (2)$$

This relation can be used to obtain $g_{isoth}(P_2)$. The peak-temperature of 171 K for the intermediate maximum between II_A and II_B corresponds to the peak temperature of P_2 (T_{max} = 170 K). Therefore for the peak value we have $\omega\tau = 1$ and $(\Delta\delta/\Delta T)_{isoth} = -1.2\text{x}10^{-4}$ gives $g_{isoth}(P_2) = 0.32\text{x}10^{-4}$ at 171 K. Thus the contribution of pinning alone, e.g. g_{isoth} (pinning) is obtained by shifting the peak value of the intermediate maximum at 171 K from $-1\text{x}10^{-4}$ to $-1.32\text{x}10^{-4}$. At 171 K then g_{isoth} (pinning) still represents an intermediate maximum between II_A and II_B, therefore, according to the present knowledge about the mechanism and decay of P_2 it must be concluded that for cold-worked copper well-separated substages II_A and II_B appear in the present experiment. At $g_{isoth} = -2\text{x}10^{-4}$ the width of the intermediate maximum is 30 K, just the temperature interval used in /6/. For this reason it is possible, that this maximum between II_A and II_B could not be recognized there.
(iii) The increase of decrement on the low temperature side of pinning stage III_B confirms the correlation between the stage III_B-defects and the relaxation peak P_x /2,9/. The decrease of P_x during pinning

stage III_B can be interpreted by the formation of multiple-defect pinning points, that do not participate in the relaxation process of P_x and thus decrease the relaxation strength of P_x.
(iv) An increase of the relaxation strength of P_2, P_3 and P_x is found from $1/\delta(\Delta\delta/\Delta T)_{isoth}$ on their low temperature flanks. A decrease begins at about the peak temperatures. Both facts indicate that all these peaks represent the superposition of pinning, relaxation and a decrease of relaxation strength.
(v) In the reduced sample besides of the pinning stages II_A, II_B and III_B a continuous pinning background is observed from 130 K to 317 K. For the oxidized sample no pinning is found at 226 K and from 278 to 288 K.

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