

1015
Meeting on fundamental aspects of radiation damage
in metals. Gatlinburg (USA), FR7600686
5-10 October 1975

CEA-CONF--3332

EFFECT OF IRRADIATION POINT DEFECTS ON THE INTERNAL
FRICTION PEAKS OF COLD WORKED COPPER

P. Moser, C. Minier, J. Lauzier

Département de Recherche Fondamentale, Section de Physique du Solide
CEM-Grenoble, BP 85 Centre de Tri, 38041 GRENOBLE CEDEX (France)

ABSTRACT

In cold worked copper the internal friction is decreased by electron irradiation between 20-140 K and increased between 140-280 K. In a copper specimen containing a convenient dislocation network, it is possible to develop Hasiguti type peaks.

INTRODUCTION

Below room temperature, the internal friction spectrum of cold worked copper presents five peaks. Two of them are "lattice-dislocation interaction" peaks (Bordoni B_1 and B_2) explained in terms of double kink generation on dislocation loops of special orientations^{1,2}. Three of them are "cold work point defect-dislocation interaction" peaks (Hasiguti P_1 , P_2 and P_3) attributed to thermomechanical depinning of dislocations³.

By using successively cold work and irradiation on copper samples, we compare the nature and the pinning effect of both cold work defects and irradiation defects.

MATERIAL AND METHODS

High purity copper (99,999 %) was annealed for 5 hours at 800°C under a 10^{-6} Torr vacuum, then defects were introduced by 300 K cold work followed by 20-30 K electron irradiation. Two apparatus, in line with a Van de Graaf accelerator were used, both allowing "in situ" cold work, irradiation and internal friction measurements, the first one for B_1 referred samples (1 % torsional cold work at 300 K; 20 K, 3 MeV, irradiation of $0.3 - 25 \times 10^{18}$ el.cm⁻²; torsional oscillations of $f = 2$ Hz creating a maximum sample strain of $\epsilon = 10^{-5}$) (Fig. 1, 4, 5, 6)⁴, the second one for B_2 referred samples (smaller 300 K cold work; < 0,1 %; smaller irradiation doses;

28 K, 2 MeV, 4×10^{15} el.cm⁻²; higher frequency: 630 Hz and higher sensitivity $\epsilon = 10^{-7}$ in bending mode) (Fig. 2, 3)⁵.

EXPERIMENTAL RESULTS

Cold work

Fig. 1 shows the internal friction of the 300 K cold worked sample S_1 during successive warming-up. We observe Bordoni B_2 , Hasiguti P_1 and P_2 peaks⁶. During annealing the Hasiguti peak P_1 increases and then decreases.

The dotted curve on Fig. 2 corresponds to the sample S_2 , having a smaller amount of cold work, just enough for giving a nice B_2 peak but no P_1 peak. Fig. 3a, obtained in comparable conditions shows the amplitude

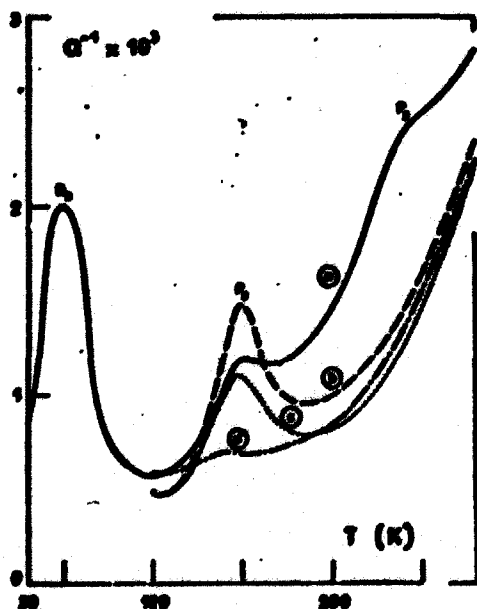


Fig. 1: Internal friction during warming-up of the 1% cold worked copper sample S_1 (a: after 300 K cold work, b, c and d: after warming-up to 340 K, 380 K and 420 K respectively). Measurement frequency $f = 2$ Hz.

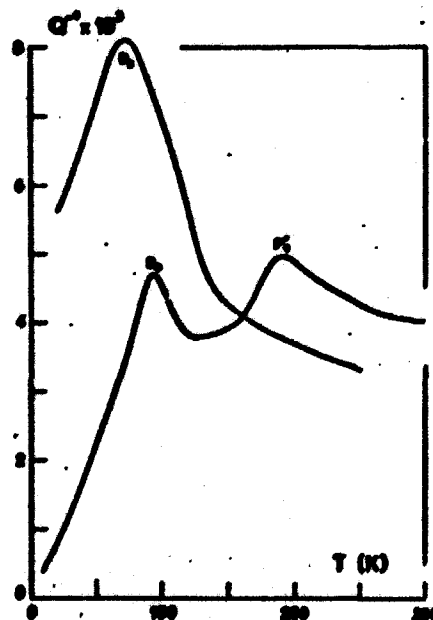


Fig. 2: Internal friction of a lightly cold worked sample S_2 before (...) and after (—) electron irradiation (28 K, 2 MeV, 4×10^{15} el.cm⁻² and 300 K annealing, $f = 630$ Hz).

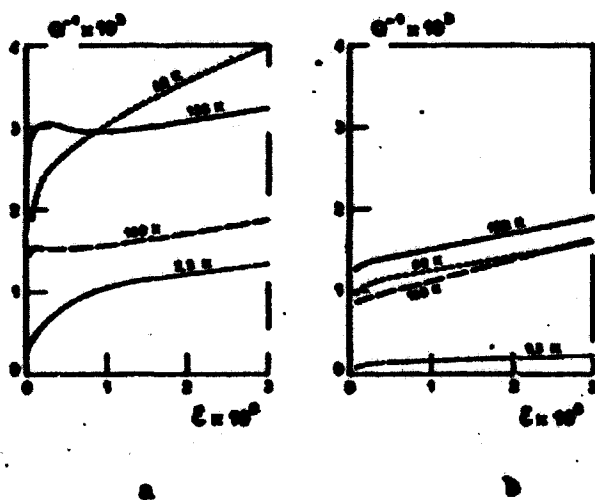


Fig. 3 : Amplitude dependence of internal friction at different measurement temperatures before and after 20 K irradiation followed by 300 K annealing for the sample S_2 ($f = 630$ Hz).

dependence of the internal friction. A rapid variation is observed at low strains ($\epsilon < 10^{-6}$) and low temperature which was attributed to thermally activated motion of preexisting geometrical kinks⁷.

Cold work followed by irradiation

For observing the interaction of irradiation defects with preexisting dislocations, the samples S_1 (Fig. 1a) and S_2 (Fig. 2 dotted curve) are of particular interest : the B_2 peak is well developed indicating the presence of a dislocation network made of long dislocation loops free from interacting defects². The absence of Hasiguti peaks shows the absence of an appreciable concentration of cold work point defects in a form which can interact with dislocations.

After a 27 K irradiation (2 MeV, 4×10^{15} el.cm⁻²) followed by 300 K annealing the sample S_2 shows a small B_2 peak and a new P_1' peak (Fig. 2 full line curve). This peak P_1' presents the same characteristics as those of the P_1 Hasiguti peak appearing after cold work. The amplitude dependence mentioned above is practically suppressed by the irradiation (Fig. 3b).

Fig. 4 shows the results obtained with a sample S_1 . Before irradiation, a small Hasiguti peak P_1 is present (curve 1). During the warming-up, immediately after a heavy 20 K irradiation (3 MeV, 1.2×10^{16} el.cm⁻²) the internal friction is drastically reduced by a pinning stage occurring at 50 K⁸ and observed on the modulus curve simultaneously obtained (curve 4). On the contrary, between 150 and 275 K, the internal friction is

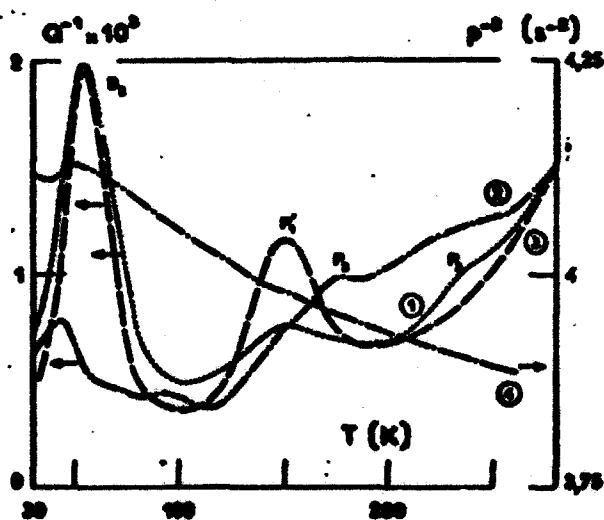


Fig. 4 : Internal friction spectra of a S_1 sample after 1 % cold work at 300 K (curve 1), an irradiation at 20 K with a dose of 1.2×10^{18} el.cm $^{-2}$ (curve 2) and an annealing at 300 K (curve 3). Curve 4 shows the frequency measurement corresponding to curve 2.

increased with broad maxima at 180 and 240 K (curve 2). After 300 K annealing, the peak B_2 is partly restored and a new peak P_1' appears as in Fig.2. The temperature shift due to the difference in measurement frequencies gives, for P_1' , an activation energy of 0.3 eV.

Fig. 5 presents the irradiation dose effect on internal friction spectrum of sample S_1 , after 300 K annealing. With increasing doses, the background decreases. The Bordoni B_2 peak decreases in size and width. As shown on Fig. 6 the peak P_1' increases first, then goes to a maximum for 1.2×10^{18} el.cm $^{-2}$ and finally decreases. The slope of the decay of the B_2 peak, and the dose giving the highest P_1' peak depend strongly on the amount of the initial cold work. In aluminium and gold, a similar beha-

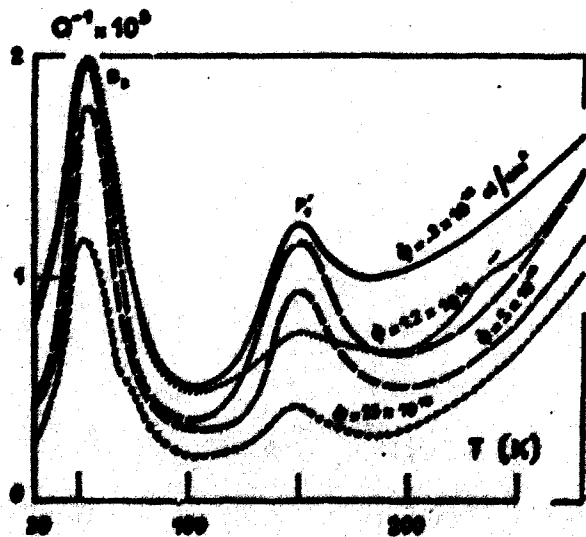


Fig. 5 : Internal friction of S_1 samples, after 1 % cold work and 20 K irradiation to different doses irradiation to different doses followed by 300 K annealing ($f = 2$ Hz). Before irradiation (...).

viour was observed^{9, 10}.

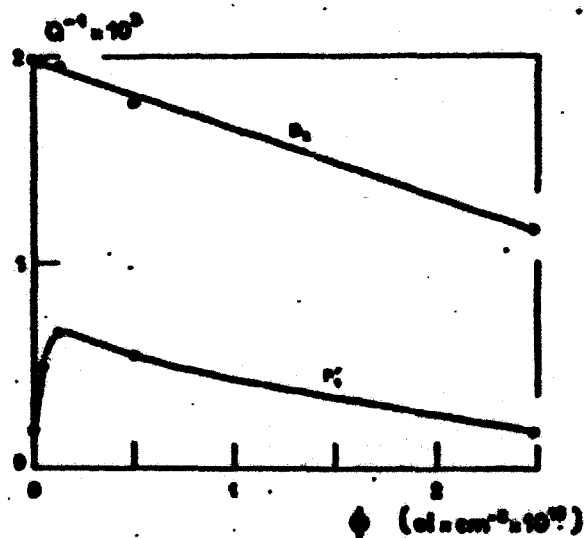


Fig. 6: Irradiation dose dependence of the Bordoni B_2 peak and Hasiguti type P_1' peak.

DISCUSSION

The significant reduction of the internal friction, and the disappearance of the amplitude dependence observed at low temperatures and low strains on Fig. 3 mean that even a low concentration of irradiation point defects ($< 10^{-7}$) is sufficient to hinder the motion of the preexisting geometrical kinks.

The 80 % decrease of the Bordoni peak B_2 and its asymmetrical shape (Fig. 4 curve 2) result from the length reduction of the dislocation loops by pinning attributed to the long range interstitial migration⁶. The incomplete restoration of the B_2 peak after room temperature annealing means that the immobile pinning interstitials are annihilated by combination with migrating vacancy type defects, allowing the dislocation loops to return to their approximate original lengths.

The complete analogy between the P_1' irradiation peak and the P_1 cold work peak shows that the same mechanism, involving the same point defect, occurs for both peaks. The conditions for the appearance of these peaks suggest that the most probable mechanism is the thermomechanical depinning of dislocations from vacancy type defects¹¹. Nevertheless we cannot exclude an interpretation in which the point defects are impurity atoms¹², but dragged to dislocations by a vacancy mechanism. The variation of the P_1' peak amplitude with the dose is correctly interpreted by Shiller's model¹³.

The small maxima observed at 180 K and 250 K (fig. 4 curve 2) anneal

out similarly to the well known Hasiguti peaks P_2 and P_3 created by cold work and situated at the same temperature⁶. This behaviour suggests that the 150-275 K internal friction is a continuum of Hasiguti peaks resulting from the interaction between preexisting dislocations and more and more complex irradiation defects.

CONCLUSION

The internal friction spectrum of cold worked copper is strongly changed by introducing point defects :

- The stage I₂ defect (free migrating interstitial) decreases the internal friction in the range of Bordoni peak B_2 through a pinning mechanism.
- Stage II defect (interstitial agglomerates) increases the internal friction in the range of P_2 and P_3 peaks through a Hasiguti mechanism.
- The stage III defect (vacancy or divacancy) annihilates the previous effects and develop a Hasiguti type peak P_1' observable when the sample contains a convenient dislocation network.

REFERENCES

1. A. Seeger, J. de Phys. 32 C2, 193 (1971).
2. W. Benoit, G. Fantozzi, Second European Conference on Internal Friction and Ultrasonic Attenuation in Solids (Rome, 1975).
3. J. Perez, P. Peguin, G. Fantozzi, P. Gobin, Ann. Phys. 5, 303 (1970).
4. R. Pichon, P. Vancopi, P. Bichon, G. de Keating-Hart, P. Moser, Rev. Phys. Appl. 5, 427 (1970).
5. J.C. Soulié, J.P. Jodeau, Y. Depierre, C. Minier, Rad. Eff. 11, 221 (1971).
6. J. Lauzier, M. Ory, G. Fantozzi, C. Minier, P. Moser, Phys. Stat. Sol. to be published.
7. M. Ory, J. Lauzier, C. Minier, G. Fantozzi, P. Moser, Second European Conference on Int. Frict. and Ultras. Atten. in Solids (Rome, 1975).
8. D. Keefer, J.C. Robinson, A. Sosin, Acta Met. 13, 1135 (1965).
9. G. Fantozzi, J. Perez, W. Benoit, P. Moser, Z. Kabsch, Rad. Eff. 11, 277 (1973).
10. O. Mercier, W. Benoit, P. Moser, G. Fantozzi, P. Perez, P. Gobin, V. Internal Conference on IFUA, Aachen (1973).
11. B. Bays, W. Benoit, P.A. Grandchamp, J. de Phys. 32 C2, 153 (1971).
12. G. Sokolowski, K. Lücke, V. Int. Conf. on IFUA, Aachen (1973).
13. F. Schiller, Phys. Stat. Sol. 5, 391 (1964).