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To cite this version:

J. Lauzier, C. Minier. A PROPOSAL TO IDENTIFY THE DISLOCATION CLASSES GIVING RISE TO DIFFERENT BORDONI RELAXATION PROCESSES IN COPPER. Journal de Physique Colloques, 1981, 42 (C5), pp.C5-133-C5-138. <10.1051/jphyscol:1981520>. <jpa-00221062>

HAL Id: jpa-00221062
https://hal.archives-ouvertes.fr/jpa-00221062
Submitted on 1 Jan 1981

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A PROPOSAL TO IDENTIFY THE DISLOCATION CLASSES GIVING RISE TO DIFFERENT BORDONI RELAXATION PROCESSES IN COPPER

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Abstract.—The variation of the Bordoni peaks during electron bombardment of copper samples has shown a substructure of the peaks which is different in annealed and cold worked specimens. A comparison with electron microscope observations allows a tentative identification of B2 to <110> dislocations, of screw type for the high temperature component and with a Burger's vector at 60° for the low temperature component. B1 is associated to dislocations along <112>.

1. Introduction.—In copper, Bordoni [1] and Niblett and Wilks [2] have found two internal friction peaks which are often labelled the two Bordoni peaks B2 and B1. They are usually attributed to the thermally activated double kink formation on the dislocation lines lying along the Peierl's valleys [3]. In the f.c.c. metals it has been proposed to associate B2 with the screw dislocations and B1 at lower temperature with the non screw dislocations [4].

In fact B2 is always found larger than the theoretical predictions and it is suspected that this peak is the sum of several components [5]. For a further clarification we have studied copper samples submitted to different cold work amounts.

- Electron irradiations have been performed at a temperature for which the vacancies are immobile and the mobile interstitials are expected to pin the dislocations. If B2 is due to a single dislocation class (screw dislocations) a continuous decrease of the peak's height and a small decrease of the temperature of the maximum are expected during irradiation, but if B2 is connected to several dislocation classes which interact differently with the interstitials, the variation of the height, width and temperature of the peak will be more complex.

- Electron microscope observations have been realized on the same samples, the density and the nature of the dislocations have been determined.

2. Experimental procedure.—The samples in the form of a dual cantilever foil are prepared from polycrystalline plates of 99.9999 % Cominco copper of 0.2 mm thickness, they are first annealed at 850° C under vacuum, then after mounting into the cryostat each specimen can be annealed in situ at 450° C and/or cold worked at 8 K to two different amounts of approximately 0.5 % and 3 %. The resonant frequency f of the sample (v 400 Hz) and the internal friction are measured with a strain amplitude of 10^-7-10^-6.

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The cryostat working between 8 K and 750 K and the irradiation facilities have been described previously [6] and also the apparatus for automatic and continuous measurements [7].

Electron microscopy is performed on samples prepared in the same way, in high voltage microscope (1 MeV) which allows the observation of specimens with a thickness of 2000 A to 3000 A.

3. Results.-
3.1. Internal friction spectra for different cold work amounts.- Figure 1 shows the results.

Fig. 1. Internal friction versus temperature of copper samples
a) annealed at 450°C
b) cold worked by 0.5 % and annealed at 110 K
c) cold worked by 3 % and annealed at 110 K
d) cold worked by 3 % and annealed at 300 K.

As it has been discussed previously by Stadelmann and Benoit [8] the experiment confirms that a large Bordoni peak B₂ can be observed in well annealed samples of very pure metals. The temperature of B₂ in annealed samples decreases when the annealing temperature decreases, it is 90 K after an annealing at 1100 K and 72 K after a treatment at 700 K.

Peak B₁ appears around 30 K in cold worked samples and in these specimens the temperature of B₂ is around 65 K.

3.2. Variation of the damping during irradiation.- On the sample of figure 1C cold worked by 3 % at low temperature, we have performed successive electron bombardments at 110 K with a 1.2 MeV energy, after each one a linear heating has been performed.
Fig. 2. Internal friction versus temperature after several irradiations at 110 K with the following doses $\Phi$ (el/cm$^2$): (a) before irradiation, (b) 0.45 $10^{15}$, (c) 1.7 $10^{15}$, (d) 7.5 $10^{15}$, (e) 14 $10^{15}$, (f) 32 $10^{15}$, (g) 81 $10^{15}$, (h) 0.28 $10^{18}$, (i) 0.68 $10^{18}$, (j) 1.5 $10^{18}$, (k) 2.3 $10^{18}$, (l) 4.2 $10^{18}$, (m) 8 $10^{18}$.

Figure 2 shows the internal friction variation; one can see that $B_2$ decreases continuously during the irradiation, but the temperature of the maximum begins to increase (65 K to 77 K) up to curve i ($\Phi = 7 \times 10^{17}$ el/cm$^2$) and then decreases.

Figure 3 shows that the width of $B_2$ increases first with the dose and then decreases for $\Phi > 7 \times 10^{17}$ el/cm$^2$.

For a quantitative analysis of the variation of the height of $B_2$ with the irradiation dose $\Phi$, we have assumed that the loop length varies as $L = \frac{L_0}{1 + n}$, $L_0$ is the initial length and $n$ the number of additional pinning points arriving during the irradiation has been taken as $n = \sigma \Phi$. The different theories expect a variation $q^{-1} = k L^{\alpha}$ with $\alpha$ varying between 0 and 2 [9] [10] [11]. An analysis of the variation of the height of $B_2$ over all the dose range ($10^{14}$ to $10^{18}$ el/cm$^2$) shows that two domains must be considered: below $\Phi = 10^{17}$ el/cm$^2$ a relation $q^{-1} = k L^{0.15}$ is quite good as shown on figure 4a and above, figure 4b shows that a relation $q^{-1} = k L$ gives a good fit.
All these unusual behaviours of a Bordoni peak can be understood if we assume that several relaxation processes take place in the \( B_2 \) range of this cold worked sample and if the dislocations contributing to the low temperature part of \( B_2 \) which are preponderant before irradiation are pinned more easily than those contributing to the higher side. In fact for a dose \( \Phi = 10^{17} \text{ el/cm}^2 \) the lower temperature component of \( B_2 \) has decreased to a value of the same order as the high temperature component and the spectrum of \( B_2 \) is broader than initially, when the dose increases further, the high temperature component becomes preponderant, the width and the temperature of the peak decrease as would be expected for a single relaxation process.

The same experiment has been performed on the well annealed sample of figure 1a [12] and the results are quite different. A relation \( q^{-1} = k L \) is very good on the whole dose range and the temperature of the peak is not shifted towards high temperature during irradiation. The assumption that in this sample only the high temperature component of \( B_2 \) exists is confirmed by microscopic observations.

3.3. Electron microscopy observations.- The observation of a well annealed sample reveals a very small density of dislocations (\( \sim 10^6 \text{ cm}^{-2} \)) the few which are seen are long (\( \sim 10 \mu\text{m} \)) nearly straight in the \( <110> \) direction and an analysis has shown that they are all of screw type. The samples cold worked at 77 K present a higher dislocation density (\( 10^8 \) to \( 10^9 \text{ cm}^{-2} \) after a 0.5 % amount and \( 10^9 \) to \( 10^{10} \text{ cm}^{-2} \) after a 3 %
cold work) with a non homogeneous distribution (figures 5 and 6). Some straight segments are seen which correspond to non screw dislocations, a majority of them are dislocations lying in the $<110>$ direction with a Burger's vector at $60^\circ$ but one observes also some segments lying in the $<112>$ direction (figure 5).

**Fig. 5.** Electron microscopy observation of a sample cold worked at 77 K to an amount of 0.5%. Dislocations a lie along $[110]$ and $[01\bar{1}]$ with a Burger's vector at $60^\circ$. Dislocations b lie along $[12\bar{1}]$ with a Burger's vector at $30^\circ$.

**Fig. 6.** Electron microscopy observation of a sample cold worked at 77 K to an amount of 3%.

4. **Discussion.**—All our internal friction results and microscopic observations can be explained with an interpretation proposed earlier by Thompson and Holmes [13]: the Bordoni relaxation is the sum of four contributions.

1. edge dislocations along $<112>$ direction
2. dislocations along $<112>$ direction with a Burger's vector at $30^\circ$
3. dislocations along $<110>$ direction with a Burger's vector at $60^\circ$
4. screw dislocations along $<110>$

1 and 2 would contribute to $B_1$ and 3 and 4 to $B_2$, the screw dislocations corresponding to a higher temperature.

In well annealed samples we have observed only screw dislocations and the damping spectrum presents mainly a peak $B_2$ situated around 75 to 90 K.

In cold worked samples the majority of the straight dislocations are along $<110>$ with a Burger's vector at $60^\circ$, they are responsible of the large relaxation effect observed around 65 K. The few dislocations along $<112>$ would be responsible of $B_1$.

During the electron bombardment of the cold worked samples the low temperature component of $B_2$ associated to dislocations with an edge character decreases more
rapidly than the high temperature component corresponding to the screw dislocations which interact less strongly with the interstitials.

References