JOURNAL DE PHYSIQUE
Colloque C5, supplément au n°10, Tome 42, octobre 1981 *page* C5-85

INTERNAL FRICTION STUDY OF THE γ PEAK IN COLD-WORKED NIOBIUM

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Abstract.- The γ peak due to the double kink generation (DKG) along screw dislocations has been observed in high purity niobium. This peak appears at 270K (1 Hz) and is unstable. Some special thermomechanical treatments have been realized in order to develop and stabilize this peak.
The evolution of the internal friction spectra has been studied as a function of these treatments. The screw dislocations network created by low temperature deformation is unstable and rearranges in two stages: a W stage (180K-240K) and a X stage (280K-350K).
The transformation accomplished in the W stage and the effect of the γ peak stabilization treatment have been observed by transmission electron microscopy.

1. Introduction.- The plastic deformation of a real solid is the macroscopic consequence of dislocation motion. The mobility of these dislocations is itself connected with different microscopic processes. These processes to overcome obstacles can occur simultaneously. Some solids, due to the special characteristics of certain defects, present a few processes that occur almost in an isolated manner. It is the case of the bcc transition metals, in which the special properties of screw dislocations (1) are responsible for an increase of the critical shear stress (CSS) with decreasing temperature (2). The temperature from which the CSS becomes athermal is called transition temperature (T_c). At T_c the screw dislocations get mobile under a low applied stress through the thermally activated DKG. During internal friction measurements such DKG along screw dislocations is responsible for a relaxation phenomenon called the γ peak. This peak and its relaxation characteristics have recently been extensively studied in different bcc metals (3-6). The instability of this peak is related to the rearrangement of the screw dislocation network, which is the subject of this study.

2. *Internal friction*.- 2.1.- Sample preparation.- The niobium samples from Materials Research Co. have 100 atppm Ta as the main metallic impurity and 400 atppm of interstitial impurities (H,O,C and N). The sample purification has consisted in a decarburization treatment (3 hours at 2173K under an oxygen pressure of 10^{-6} Torr) followed by a high temperature annealing (3 hours at 2500K under a vacuum pressure of about 7×10^{-10} Torr). After this purification treatment the interstitial concentration

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should be about 100 atppm (3).

2.2. Experiment.- The internal friction and frequency measurements were carried out in an inverted torsion pendulum under a frequency of 1.3 Hz and in a temperature interval from 77K to 347K. The maximum surface strain amplitude was 10^{-6} and the heating rate was 1^{o}/min.

2.3. Results.- 2.3.1. γ, α_1 and α_2 peaks.- The γ peak associated to the thermally activated DKG along the screw dislocations have been observed recently in niobium (3). It appears at 270K at low frequency (1 Hz) according to the plastic deformation observations ($T_c \simeq 230K$).

A systematic study of the effect of deformation amplitude and deformation temperature has shown the optimum deformation conditions to observe an important relaxation. They are: $160K < T_{def} < 200K$ and $\varepsilon_{def} \simeq 5\%$.

The γ peak generally anneals out in a temperature range situated near the peak temperature. A special thermomechanical treatment has been performed in order to stabilize the dislocation network: a room temperature predeformation of 3% in traction followed by a series (about 15) of successive 5% torsion deformations at 200K with intercalated linear annealings up to room temperature (RT) and the last 5 at 347K.

The relaxation characteristics of the peak have then been determined: $E = (0.61 \pm 0.02)$ eV, $\tau^{\infty} \simeq 10^{-12}$ sec and it is 1.3 times wider than a single Debye peak.

The α_1 and α_2 peaks have been observed at 150K and 120K, respectively, at 1.3 Hz. It is generally accepted that these peaks are due to the interaction of hydrogen with dislocations (7).

2.3.2. Influence of deformation temperature.- To put in evidence the influence of the deformation temperature on the internal friction spectra, two deformation temperatures have been chosen, below and above T_c. In figure 1 are presented the curves of internal friction as a function of the measurement temperature, corresponding to different treatments reported on the figure. The fundamental difference is that the low temperature deformation develops mainly the α_2 and γ peaks (curve c), while the RT deformation is responsible for the α_1 peak. The participation of the screw dislocations in the α_2 and γ relaxations and of the 71^{o} dislocations in the α_1 peak seems clear (3). Concerning the interpretation of these peaks, some recent observations are significative. Ritchie et al.(4) in α-iron and Maul et al. (7) in niobium

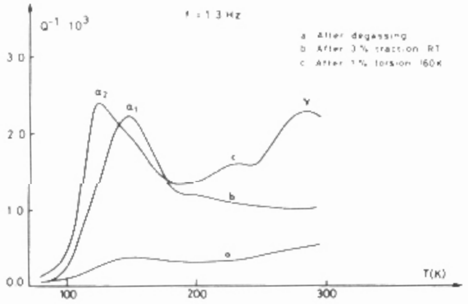

Fig. 1: Internal friction spectra of 3 degassed samples: a) after degassing; b) after cold-work 3% in traction at RT and c) after cold-work 1% in torsion at 160K

have observed an internal friction peak at 30K (0.5 Hz) and 70K (1 KHz), respectively. They have been interpreted by a DKG along 71^{o} dislocation. Moreover, Ritchie et al.(4) observed a frequency variation at 4K that is associated to the geometrical kink migration. The connection between the influence of hydrogen on the α_1 and α_2 peaks, and these last interpretations have lead us to the following conclusion: the hydrogen interacts with DKG along the 71^{o} dislocations for α_1 and with geometrical kink migration in screw dislocations for α_2. The γ peak is due to the thermally activated DKG along the screw dislocations (3).

2.3.3. Recovery stages.- The difference of mobility between 71^{o} and screw dislocations in bcc metals favours the study of the evolution of the internal friction spectrum as a function of the annealing treatments. As mentioned before, the screw dislocations get mobile under a low applied stress at temperatures near T_c, while the 71^{o} get mobile at much lower temperature (∿40K). Then it is interesting to insert screw dislocations in their "froze-in" state ($T_{def} < T_c$) and follow their evolution with annealings at temperatures near T_c. The linear annealings realized between 180K and 350K showed two recovery stages, that is, two annealing temperature ranges during which the internal friction spectrum changes: W stage (180K-240K) and X stage (280K-350K). Figure 2 shows the results obtained with a degassed sample deformed 1% in torsion at 160K (curve a) and then submitted to the linear annealings indicated on the figure. The arrows under the curves show the position and evolution of the α_2 and α_1 peaks during the W stage. The curve a of figure 3 has been obtained with a degassed sample submitted to the γ peak stabilization treatment (para.2.3.1.) and then deformed 5% in torsion at 200K. After a linear annealing until 347K, that is, in the temperature corresponding to the X stage the curve b has been obtained. The temperature intervals corresponding to the W and X stages are shown. The dashed curves marked a' and a" show the influence of the W stage while the X stage induces the transition from curve a to curve b. The inset shows the creep stage that takes place simultaneously with the W stage (curve c).

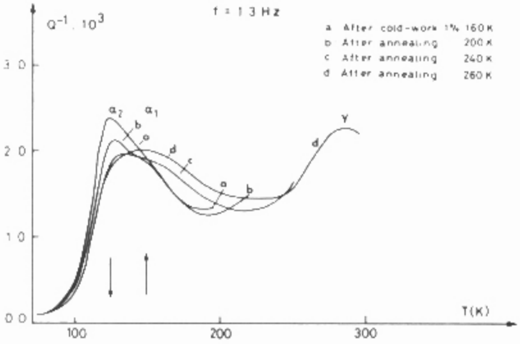

Fig. 2: Internal friction spectra of a degassed sample submitted to the treatments: a) after cold-work 1% torsion at 160K and after annealings at: b) 200K; c) 240K and d) 260K.

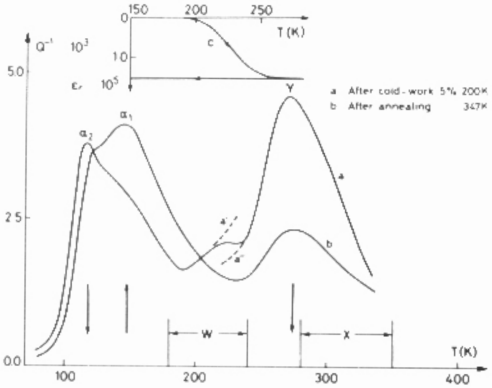

Fig. 3: Internal friction spectra of a specimen cold-worked at RT and then submitted to the γ peak stabilization treatment (para.2.3.1.). The inset shows the creep stage (ε_r = rotation deformation measured at the specimen surface) that accompanies always the W stage.

2.3.4. Conclusion.- Concerning the thermomechanical treatments these experiments have shown that the α_2 and γ peaks evolve likewise even when α_1 peak presents the opposite of this evolution, with samples predeformed at RT. Then it seems to exist one connection between the three peaks and their evolution.

Two different phenomena may be responsible for the existence of the two recovery stages, and as consequence for the γ peak elimination. The first one could be a rearrangement of the screw dislocations network and the second one, a pinning of these dislocations by the intrinsic point defects created also by the low temperature deformation. We believe that the rearrangement is the more probable process because a dislocation pinning cannot be responsible for: a) the creep stage that occurs simultaneously with the W stage, and which is due to a dislocation movement, and b) the increase of the α_1 peak amplitude with annealings in the temperature range corresponding to the X stage. A screw dislocation rearrangement may be an annihilation of screw dislocations or a screw network change with lost of the screw character (8). We will try to reply to this question by transmission electron microscopy (TEM) observations.

3. Electron microscopy.- 3.1. Objective.- The main objective of the TEM observations was the verification of the W stage interpretation: partial annihilation of the screw dislocations network. The internal friction measurements with specially treated samples (para.2.3.1.) show a screw dislocation network stability (figure 3, curve b). The second objective of the TEM observations was to verify this stability.

3.2. Sample preparation.- Cylindrical samples ($\emptyset$ = 3 mm, ℓ = 50 mm) have been degassed by magnetic induction heating in vacuum conditions mentioned in section 2.1. Some samples have been prepared after this purification treatment (A), while others were submitted to the γ peak stabilization treatment (para.2.3.1.)(B). After electron erosion cutting we have obtained some plates (7x3x0,2 mm^3) presenting a <110> normal to the surface. After thinning treatment the samples thickness was about 1500 to

2000 Å in a region situated at 3 to 6 μm away from the hole.

3.3. Results and discussion.- The experiment has consisted in the creation of a dislocation network by "in situ" deformation at low temperature in the electron microscope and in observation of its evolution during the heating run. Figure 4 shows four photographies that have been taken from a film which presents more details. This sequence has been chosen between 40K and 222K. The figure 4a shows the dislocation network at 40K after two series (deformation at 40K and linear annealing at 245K). The application of an increasing force until 1.4N creates the screw dislocation network shown on figure 4b. The heating run has been realized at constant small force (F = 0.1N) in order to keep the specimen flat and with a heating rate of 1^{0}/min. Figures 4c and 4d show the dislocation network at temperatures in the temperature range corresponding to the W stage. We can note that the long screw dislocations created by the low temperature deformation have disappeared. In the film we observe that some screw dislocations are not eliminated by the linear annealing at 245K. We believe that these dislocations are responsible for the γ peak.

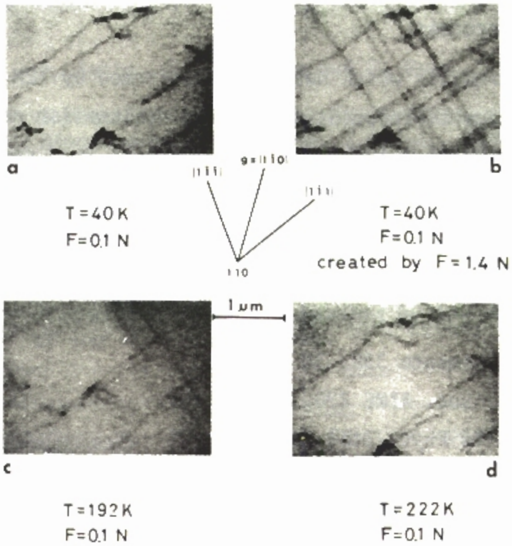

Fig. 4: Dislocation arrangements observed during low temperature "in situ" deformation in the electron microscope.

Figure 5 shows the existing dislocation network at 270K in a sample submitted to the γ peak stabilization treatment. We can observe a dislocation network composed

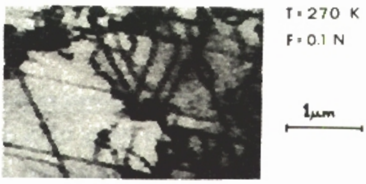

Fig. 5: Dislocation arrangement of a sample submitted to the γ peak stabilization treatment (para.2.3.1.) observed in the electron microscope at 270K.

by long and medium dislocations anchored in some dislocation agglomerations.

The TEM observations have shown that: a) some screw dislocations created by the low temperature deformation disappear at the temperature range corresponding to the W stage and, b) a stable dislocation network that contains some screw dislocations exists at 270K. The possibility of pinning may be excluded, but it is very difficult to define the rearrangement process.

4. Conclusion.- The internal friction results which have been confirmed by TEM observations are: a) the W recovery stage corresponds to a screw dislocation network rearrangement and b) the thermomechanical treatment that consists in a series (about 15) of low temperature (200K) deformation intercalated by linear annealings at RT and the last 5 at 347K, is efficient to stabilize the screw dislocation network.

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The authors want to thank Ph.Buffat, A.Steiner and R.Gotthardt for the TEM observations which have been made in "Institut Interdépartemental de Métallurgie" of the Swiss Federal Institute of Technology.

This work has been supported by the "Fonds National Suisse de la Recherche Scientifique", subsidy nr. 2.472-0.79.