Study for Observation Conditions for α and γ Relaxation in Niobium

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ABSTRACT

The main objective of the present work is the determination of the ideal conditions for the observation and study of the relaxation processes due to dislocations in high purity niobium. This work is divided into two parts. In the first part, the arrival of point defects on dislocations during the course of thermal treatment is used to study the prior purification and subsequent contamination of the sample. In the second part a study is made of the evolution of the α-peak during the same treatment.

The niobium samples from Materials Research Co. were degassed in a vacuum of 4×10^{-9} Torr at a temperature of 2500 K. The internal friction and excitation frequency spectra as a function of temperature were obtained with degassed and coldworked samples at 77 or 295 K. After various thermal treatments up to 700 K the internal friction spectra shows the existence of at least two stages of defect migration and also a diminution of the height of the α-peak.

KEYWORDS

Internal friction ; b.c.c. ; dislocations ; impurity ; double-kink generation ; cold-work ; Bordoni peak ; defect migration .

INTRODUCTION

Deformed b.c.c. refractory metals present at low frequency an internal friction spectrum composed of three peaks, figure 1 (curve 1). According to Chambers (1966) these peaks are called α, β and γ. The α and γ-peaks are attributed by Seeger and Wüthrich (1976) to double kink generation in non-screw and screw dislocations, respectively ; and the β-peak by Ritchie, Dufresne and Moser (1978) to the unpinning of dislocations from immobile point defects. Linear annealings at 500 and 700 K, have modified substantially the internal friction spectra, figure 1, curves 2 and 3 respectively. The latter annealing has caused oxygen penetration and the appearance of the corresponding Snoek peak. This change of the spectra with annealing shows that a study of the peaks is impeded by the arrival of point defects on dislocations, causing pinning, and by the rearrangement of the

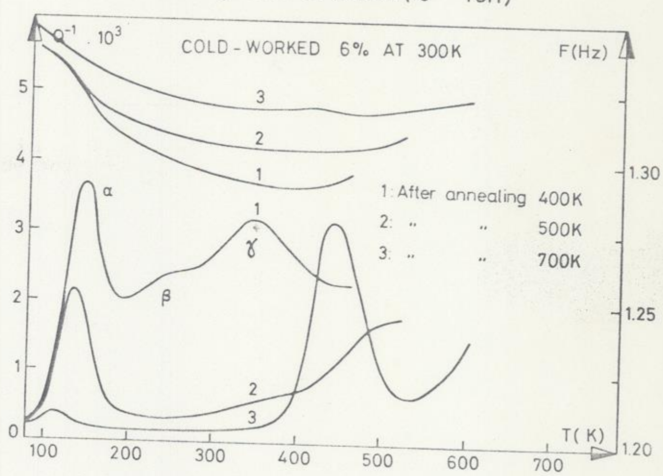

Fig. 1. Internal friction and excitation frequency after subsequent annealing of a cold-worked sample 3 % at 300 K.

dislocation network. Thus, a preliminary study is necessary, which can be divided into two parts. In the first part, the various stages of annealing must be found ; and in the second part, their interpretation must be given.

EXPERIMENTAL PROCEDURE

The niobium samples from Materials Research Co. (main metallic impurity 200 at. ppm Ta) were degassed under a vacuum of about 4×10^{-9} Torr (5×10^{-12} bar) at a temperature of 2500 K. After this treatment a bamboo structure with grains about 4 mm long was observed. The internal friction and frequency measurements were carried out in cylindrical samples (3 mm in diameter and 40 mm in length) under longitudinal vibration for resonant frequencies near 40 kHz. The internal friction and excitation frequency spectra have been obtained with one sample after degassing alone and also with samples deformed in compression at 295 and/or 77 K following the degassing. The thermal treatments consisted in linear annealings in which the maximum temperature attained in each measurement is considered as the annealing temperature for the next measurement. For the temperature range above room temperature we have employed different pressures, from 1 Torr of helium to 2×10^{-8} Torr. In the ultra high vacuum installation we cannot measure below room temperature.

EXPERIMENTAL RESULTS

The internal friction is measuring strain amplitude independent in the amplitude range used (2.5×10^{-6}).

Figure 2 shows the internal friction and the excitation frequency spectra as a function of measurement temperature for a deformed sample (initially .8% at room temperature and then 2% at 77 K). The measurement pressure was 1 Torr

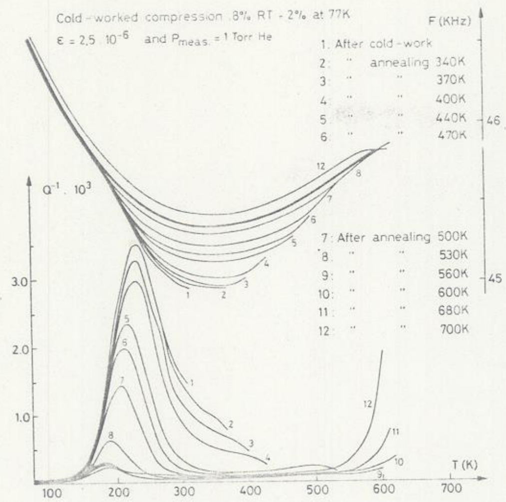

Fig. 2. Internal friction and excitation frequency after deformation and subsequent linear annealings. P = 1 Torr He.

of helium. The α-peak appears at about 240 K with the high temperature side well developed. After annealings above 340 K an internal friction decrease and an irreversible frequency increase are observed. Internal friction drops appear for measurement temperatures just above 400 K and again just above 500 K. The parallel decrease of the α-peak with annealing up to 400 K may be interpreted as being due to the background decrease, with the real peak intensity constant. After annealings above 400 K, both the peak intensity and temperature decrease. Above 600 K annealings there is an increase of the oxygen content in the matrix, associated with the increase in Snoek peak height.

In figure 3 are shown the spectra obtained with a sample first after degassing and then after cold-work 3 % at 77 K. The pressure was 1 Torr of helium for the experiments at temperatures below room temperature and 2×10^{-8} Torr above room temperature. The spectra evolution is analogous to that shown in figure 2. One observes three stages that contribute to the internal friction decrease. The first is situated between 300 and 400 K, the second just above 400 K and the third between 500 and 600 K. The oxygen Snoek peak that appears at about 600 K corresponds to about 10 at. ppm.

This behaviour was also observed with a sample that was degassed without subsequent deformation.

DISCUSSION

To interpret the observed stages we will reconsider the above evolution of the α-peak during annealing and also consider the work of Faber and Schultz (1977). In figure 4 the upper part shows schematically the resistivity recovery as a function of annealing temperature, for three low temperature electron irradiated niobium samples ; and the lower part presents the spectra taken from figure 3.

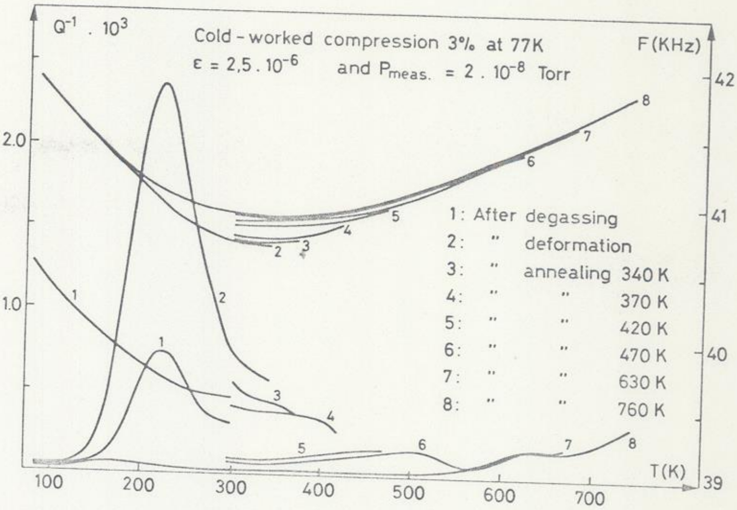

Fig. 3. Internal friction and excitation frequency after degassing, deformation and subsequent linear annealings. P = 2 x 10^{-8} Torr.

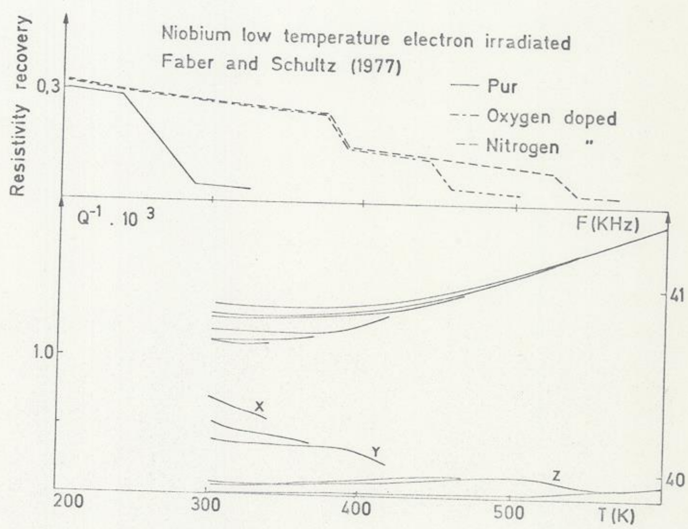

Fig. 4. Resistivity recovery as a function of annealing temperature (upper part) and internal friction and excitation frequency taken from figure 3.

One observes that stage X lies between the single recovery stage observed in the high purity sample and the common stage of the doped samples. The Y stage is found at lower temperature than the oxygen migration stage and the Z stage corresponds well to the nitrogen migration stage. As previously observed, the real change of the α-peak height and position occurs for annealing temperatures that correspond to the Y and Z stages and does not occur for temperatures of the X stage. This behaviour supports the association of the Y stage with oxygen migration and the Z stage with carbon and/or nitrogen migration, where these impurities pin the non-screw dislocations thus decreasing the peak height and temperature. The X stage is very important, because it is during this stage that a modification in the γ spectrum takes place. This modification may be due to the migration of point defects to the screw dislocations or to the rearrangement of its network. However, some facts make the second possibility more reasonable. Firstly, the α-peak temperature is constant during this stage. Secondly there is no corresponding resistivity stage ; and finally, this stage was also observed in the non deformed sample. These facts are all arguments against point defect migration.

CONCLUSION

To study the γ-peak it was found necessary to know the effect of the point defect restoration on dislocation mobility.
Numerous stages have been observed probably due to point defect migration. These stages may then be eliminated with better niobium purification.
One stage (X stage) does not seem directly connected with impurities or intrinsic point defects. Its appearance could be due to the dislocation restoration (directly connected with the γ-peak) in which case ways must be found to ensure the network stability.

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