

ELECTRON IRRADIATION EFFECT ON SINGLE  
CRYSTAL OF NIOBIUM.

M.P.OTERO

G.LUCKI

Instituto de Pesquisas Energéticas e Nucleares  
IPEN-CNEN/SP.

ABSTRACT.

The effect of electron irradiation (900 keV) on gliding dislocations of single crystal Nb with its tensile axe in the [941] orientation was observed for the "in-situ" deformation in a high voltage electron microscope (HVEM) at Argonne National Laboratory. The experiment was carried out by the lh-electron irradiation with no stress applied. Straight dislocations actuating as sinks for the electron-produced defects became helicoidal as the irradiation proceeded. Frenkel pairs were created in Nb for electron energies  $\geq 650$  keV and, as the single vacancies do not undergo long-range migration in Nb at temperatures much below 620 K, the defects that are entrapped by the dislocations are self-interstitials produced by electron displacement.

Applying the stress it was possible to observe that the modified dislocations did not glide while the dislocations not affected by the irradiation are visibly in movement. This important result explains the neutron and electron-irradiation induced work-hardening effect for Nb that was previously observed.

## 1. INTRODUCTION.

One of the most important characteristics to be considered in the nuclear reactor design is the behaviour of nuclear materials to the mechanical stresses. These mechanical stresses can be produced by the expansion of the materials due to the environments at high temperatures and pressures. Adding to these physical parameters there is the radiation damage effect that can improve or worsen the qualities of the materials under irradiation. Besides, one has to consider the neutron absorption properties for the materials to be employed as the fuel cladding and as the internal protection of the walls of nuclear reactors. The former must have a low cross section for the neutron absorption and the later a high one.

Another energy source in development is the nuclear fusion design. Researches are been carried out in order to make possible the practical use of the resultant energy that comes from nuclear fusion processes. As the necessary temperature that turns on the thermonuclear reactions is extremely high, the best option, for the present, is to hold the plasma in flotation by magnetic fields. The "first wall" of such a nuclear fusion reactor is the closest one to the plasma. Therefore the material for the first wall needs to have a very high melting point in order to keep up with the extremely high temperature and with the very high mechanical stresses that it experiences.

Among many metals and alloys under studies for that purpose there is a refractory metal called Niobium (Nb). It has a high melting point temperature ( $2,458^{\circ}\text{C}$ ), a good resistance to chemical attacks and a relatively low cross section for neutron absorption.

The objective of this work is to give a contribution to the understanding of the neutron and electron-irradiation effects on the mechanical properties of Nb. To accomplish this objective, the interaction of dislocation loops with gliding dislocations during "in-situ" electron-irradiation and deformation was studied in the high voltage electron microscope (HVEM) at Argonne National Laboratory - ANL.

## 2. EXPERIMENTAL PROCEDURE.

Single-crystalline rods (6.3 mm diameter) with an axial orientation of the [941] crystallographic direction were prepared by electron-beam zone melting and seeding with an oriented crystal. This orientation was chosen because of its respective work-hardening and work-softening response to an applied stress on tensile testing at 77 K<sup>(1)</sup>.

The oriented rods were sectioned to yield sheet tensile specimens with the wider surface of the specimens containing the primary Burgers vector, i.e.,  $a/2[11\bar{1}]$ , Fig.1. The [941] orientation is near to the center of the [100] - [110] - [111] triangle orientation

The sheet specimens that were to be deformed in an Instron tensile machine for a determination of their stress-strain curve were ~0.2 mm thick and 1.6 mm wide within a gauge length of 5.5 mm

The specimens that were intended for simultaneous "in-situ" tensile deformation and TEM observation in the HVEM were 0.2 mm thick and 0.5 mm wide within a gauge length of 2.5 mm. The surfaces of the sheet tensile specimens were mechanically polished to give a surface finish with less than 0.3  $\mu\text{m}$  depth undulations. The sheet specimens were subsequently degassed with respect to the O, N and C impurities by ultra-high vacuum ( $\sim 10^{-9}$  torr) annealing at 2,600 K. For TEM observation in the HVEM, the specimens were

thinned to an electron-transparent thickness (0.1 - 0.15 $\mu$ m) by electrolytic polishing in a 12%HF - 88%HNO<sub>3</sub> solution at 275 K. The stress-strain curves for the bulk specimens were obtained by the use of an Instron tensile machine with a strain rate of 5X10<sup>-4</sup> mm/s. The "in-situ" deformation of the tensile specimens were carried out in the ANL-HVEM using a double-tilt straining stage with a straining rate of 2X10<sup>-4</sup> mm/s. Neutron irradiation at room temperature of some Instron specimens were performed at IPNS-intense pulsed neutron source at ANL to a fluence of 5X10<sup>21</sup> neutrons/m<sup>2</sup>.

### 3. RESULTS AND DISCUSSION.

#### 3.1. Instron Stress-Strain Curves.

The stress-strain curves for tensile specimens in the unirradiated and irradiated (5X10<sup>21</sup> neutrons/m<sup>2</sup>) conditions obtained on deformation in an Instron-machine are shown in Fig.2. It can be seen that the irradiation increases the yield stress from 3.5 to 4.0 kgf/mm<sup>2</sup>. This same increase in the yield stress due to the neutron irradiation was observed for Nb + 200 ppm O<sub>2</sub> with the orientation [941] and [441] <sup>(2)</sup>. For the present experiment Nb - [941] degassed (~10ppmO<sub>2</sub>) it can be seen that the irradiated specimens there is a work-softening in the range 0.2-4% strain followed by a work-hardening effect beyond 4% and for the unirradiated specimens a persistent work-softening was observed until the fracture after a very pronounced necking in the gauge region of the specimen.

#### 3.2. "In-Situ" Electron-Irradiation and Deformation.

The effect of irradiation at HVEM with 900 keV electrons at room temperature can be seen in a series of micrographs in Fig.3.

The irradiation was performed during 1h, followed by an "in-situ" deformation. The first micrograph shows the initial conditions of the specimen with  $10^{17}$  dislocations/cm<sup>2</sup>. As the irradiation proceeds the dislocation density increases by the creation of a very large number of small dislocation loops. The observed growing of the dislocations loops and the changes in the shape of the extended dislocations are clearly due to the electron-irradiation. After 1h of irradiation, the irradiated area became extremely damaged.

Followed the 1h irradiation a stress was applied on the specimen during 12 minutes and the resulted deformation was observed. The gliding of extended dislocations is showed by the last micrograph in Fig.2. One can observe that the extended dislocations exposed to electron irradiation cannot glide during "in-situ" deformation. The differences between the behavior of the dislocations belonging to the irradiated area and the dislocations that were out of that area explain quite well the increase in the yield stress as observed in Fig.1, i.e., the irradiation harden the Nb when it is deformed in the [941] direction. The work-hardening of a [941] Nb single crystal irradiated to  $3.1 \times 10^{17}$  electrons/cm<sup>2</sup> was fully reported by Nagakawa and Meshii<sup>(1)</sup>. This work-hardening phenomena induced by irradiation is generally regarded as a consequence of dislocation channeling, in which irradiation-induced defects are removed upon post-irradiation plastic deformation<sup>(2,3,4)</sup>

The growth rate of loops A, B and C, observed in the micrographs of Fig.2, was determined to be 2 nm/min. For Nb [441]-orientation it was found to be 3 nm/min<sup>(3)</sup>. The growth of loops and the effect on the extended dislocations can be attributed to the diffusion of self-interstitial atoms produced by electron displacement. The threshold energy for atomic displacement in Nb by an

electron is 24 eV. Thus Frenkel pairs are created in Nb for electron energies ~650 keV. Single vacancies do not undergo long range migration in Nb at temperatures much below 620 K.

#### 4. CONCLUSION.

This experimental study confirmed the work-hardening effect induced by neutron- and electron-irradiation on Nb [941] - oriented single crystal. It was possible to follow the production of the defects and the effect on the grown-in line dislocations. Also the existent loops was determined to be 2 nm/min. The existent loops and dislocations are of interstitial nature, because the defects produced by electron-irradiation are self-interstitials that are added to those existent, causing their growing-up instead of their shrinkage.

#### 5. REFERENCES.

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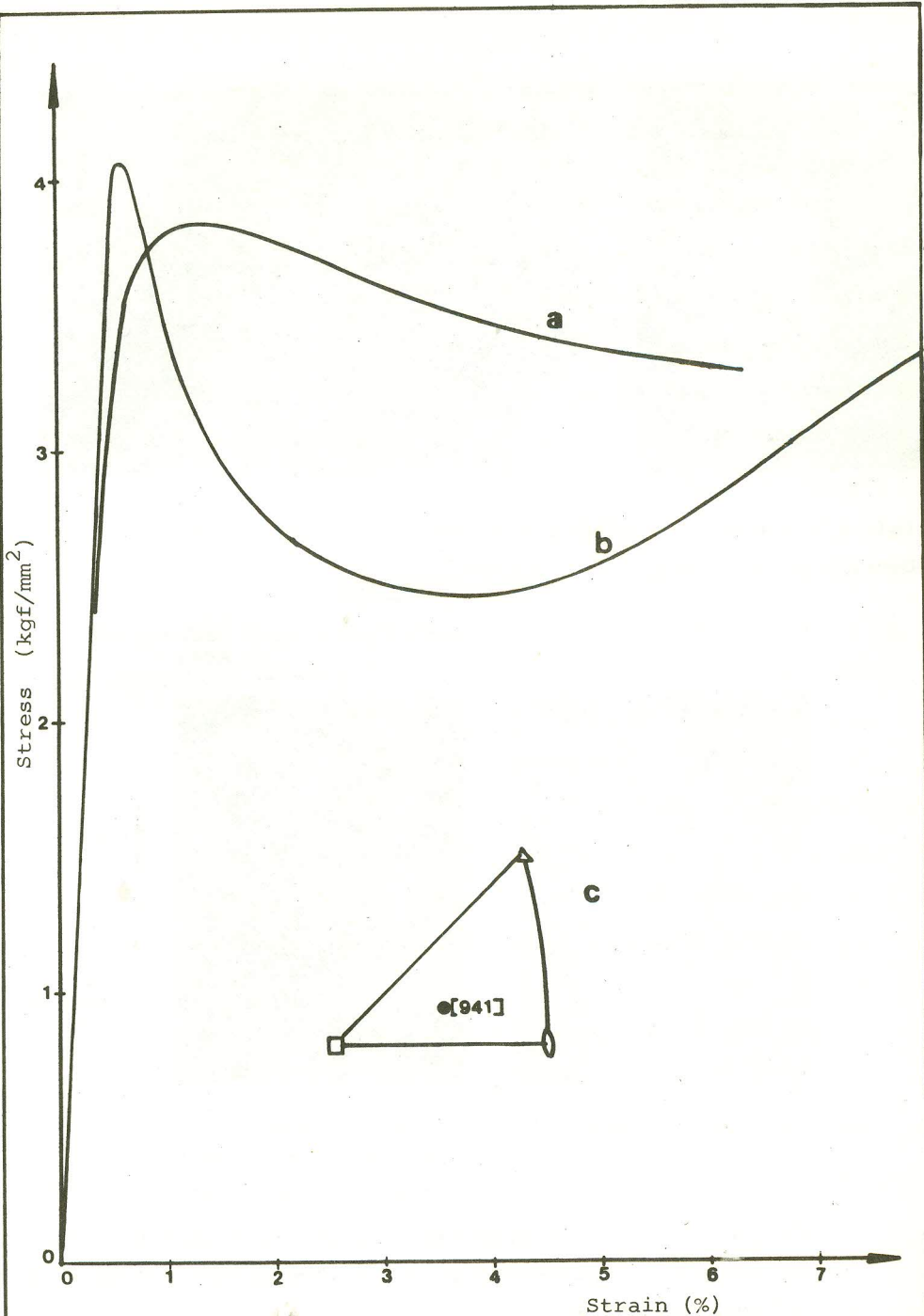


Fig.1 - Stress-Strain Curves for Nb Single Crystals.  
 (a) - Unirradiated, (b) Neutron Irradiated at 325 K with  $5 \times 10^{21}$  neutrons/m<sup>2</sup>, ( $E > 0.1$  MeV), (c) Position of the tensile axis in the stereographic triangle.

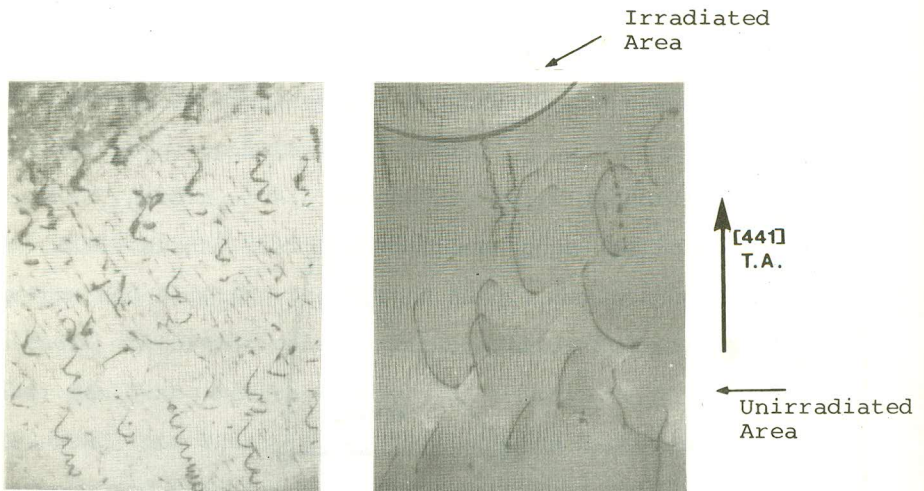


(a)  $t = 0$  min.

(b)  $t = 23$  min.

(c)  $t = 40$  min.

Density =  $10^{17} \frac{\text{disl.}}{\text{cm}^2}$



(d)  $t = 59$  min.

(e)  $t = 72$  min.

This area is below the irradiated area (d).

Fig. 2 - Effect of "in-situ" electron-irradiation ( $E=900$  keV) on Nb [941] - oriented single crystal. The stress was applied after  $t = 59$  min.