INSTITUTO DE ENERGIA ATÔMICA - SÃO PAULO

PREFERRED ORIENTATIONS IN NIOBIUM DETERMINED BY NEUTRON DIFFRACTION

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PREFERRED ORIENTATIONS IN NIOBIUM DETERMINED BY NEUTRON DIFFRACTION

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ABSTRACT

Neutron diffraction has been utilized to study textures developed in polycrystalline niobium, cold rolled to 60% and 80% in thickness.

The pole figures obtained were analysed in terms of the following ideal orientations $(112)[\bar{1}10]$, $(001)[110]$, $(113)[\bar{1}10]$, $(111)[11\bar{2}]$, $(111)[1\bar{1}0]$.

A comparison of these, with textures in b.c.c metals shows a close similarity.

INTRODUCTION

Niobium has long been considered as an important high temperature nuclear structural material for both fission as well as fusion reactor applications. Niobium combines a high melting point, low density and low thermal neutron capture cross section, relatively high thermal expansion coefficient, high thermal conductivity chemical inactivity and good resistance to liquid metal corrosion(4,5).

It is well known that some of the world's major deposits of niobium ore (Phyrochlore $(NaCa_2)$ Cb_2O_6 (O,OH,F) are located in Brasil, and presently Brasil is the world's largest producer of Niobium oxide concentrates(6). The technology to produce high purity niobium is difficult, however, a major effort is under way at the University of Campinas in São Paulo to produce high purity niobium by electron beam melting and zone refining(9). The characterization of various physical properties of niobium, for nuclear applications, has been undertaken by the Coordenadoria de Ciências e Tecnologia dos Materiais in this Institute(8).

The material used in the production of fuel element cladding or as first wall blanket for fission or fusion reactors respectively, must in general characterize highly isotropic mechanical and thermal properties, thereby ensuring utilization of simpler and economical fabrication processes. The anisotropy of physical properties of polycrystalline metals is closely connected with texture its presence being governed by the non random distribution of grain orientations. The commonly used technique of texture determination is based on X-ray diffraction. However in some cases this technique does not give accurate results, for example, the presence of large grain size precludes the use of conventional X-ray technique, as an analysis by Laue method becomes immensely tedius.

On the other hand a low absorption coefficient of neutrons in the majority of chemical elements, as well as large cross section of neutron beam makes neutron diffraction an ideal technique for texture determination in metal specimen consisting of coarse grains(13). In order to obtain a

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reproducible pole figure one has to know the orientations of at least 10^4 grains (Haessner Criterion). The volume of irradiated sample using neutron diffraction technique is $10^5 - 10^6$ times greater than the usual X-ray technique.

The overall objective of the present work is to correlate preferred orientations in niobium with its physical properties. As a first step neutron diffraction technique has been utilized in this study to investigate textures produced by cold work in coarse grained electron beam refined niobium.

EXPERIMENTAL

Niobium samples in the form of approximately 4 mm thick plates, previously cut from a 99,8% pure electron beam melted ingot, were obtained from the Solid State Physics Department of the University of Campinas (UNICAMP) São Paulo. These plates were cold rolled to 60% and 80% reduction in thickness.

In order to increase the irradiated volume of niobium, the samples were prepared as follows: two sheets each 2.0 mm thick, of cold rolled material were superposed over each other. The 80% cold rolled niobium consisted of three superposed sheets each having a thickness of 1.03 mm. The rolling direction was always maintained, necessary geometrical conditions for the experiments were satisfied by collimating neutron beam having a circular cross section, and by exposing the central round portion of the specimen surface to irradiation ($\phi = 35$ mm).

The neutron diffractometer was set at a Bragg angle corresponding to the (110) reflection. In order to facilitate neutron reflections from crystallites prossessing all possible space orientations the samples were rotated about three mutually perpendicular axes. The specimen rotation was achieved by means of a special goniometer[2]. The registered changes in neutron intensity are due to specimen texture. The angles introduced are defined as follows:

α = angle between the scattering vector and the specimen surface.

β = angle between the rolling direction (R.D.) and the projection of scattering vector on the specimen surface. All possible values of angles can be obtained by utilizing both transmission and reflection methods. At small α-angles ($0 - 45^\circ$) transmission method and at large α-angles ($45^\circ - 90^\circ$) reflection method were used. Data obtained from both methods were normalized at an overlapping angle of $\alpha = 45^\circ$.

RESULTS AND DISCUSSION

Figure 1 – shows neutron diffraction patterns for the as received, 60% and 80% cold worked niobium sheets. The registered irregularity in the neutron intensity for the as received samples is due to coarse grains. For 60% rolled niobium two additional intensity peaks are observed, whose positions are asymmetrical with respect to the peak observed at $\beta = 90^\circ$. This is probably connected with the presence of recrystallization texture.

Figure 2 – Shows a plot of neutron intensity vs α angle corrected for changes in irradiated sample volume ($\beta = 100^\circ$).

The absolute units of pole density were determined according to the equation:

$$I\, S = \sum_i I_i\, \Delta S_i$$

where

I S is pole density of theoritical isotropic specimens with similar absorption and scattering capacity as the investigated specimen, integrated over the whole range of density orientations.

I_i is the mean pole density measured at the elemental sphere surface ΔS_i.

Σ is the summation over all elemental sphere surfaces corresponding to the whole range of density orientations.

Figures 3 – and 4 show pole figures for 60% and 80% cold worked niobium respectively. Absolute units are used in these pole figures, this assures comparison, of the degree of preferred orientation developed in the various specimens. The maximum pole density in Figures 3 and 4 is equivalent to 8 absolute units.

Following are the preferred orientations obtained from Figures 3 and 4.

a) Niobium 60% cold worked	b) Niobium 80% cold worked
$(112)\ [\bar{1}10]$	$(112)\ [110]$
$(001)\ [110]$	$(001)\ [110]$
$(111)\ [11\bar{2}]$	$(111)\ [11\bar{2}]$
$(113)\ [\bar{1}10]$	$(113)\ [\bar{1}10]$
$(111)\ [1\bar{1}0]$	$(111)\ [1\bar{1}0]$

and an additional $(111)\ [1\bar{2}1]$at an angle of 20° to T.D.

These preferred orientations are all typical for b. c. c metals(1,7,10-12) for example those determined for niobium(1), vanadium(7), tantalum(11) and molybdenum(12). In addition weakly developed preferred orientation $(111)\ [1\bar{1}0]$ has also been reported for some b. c. c metals(3,14). In figure 3, the orientation $(111)\ [1\bar{2}1]$ is observed to be at an angle of 20° to T.D, suggesting that this particular orientation is not connected with the rolling process, reather it could be due to recrystallization texture(1), produced by electron beam melting, used in the production of niobium samples. This arguement is reinforced by the fact that in Figure 4; for samples subjected to 80% cold work, this orientation does not seem to be important.

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RESUMO

Texturas produzidas por trabalho a frio em placas de ~~N~~nióbio foram investigadas.

Figuras de polos, para o metal laminado a frio, foram interpretadas em termos das seguintes orientações ideais:

$(112)\ [\bar{1}10]$, $(001)\ [110]$, $(113)\ [\bar{1}10]$, $(111)\ [11\bar{2}]$, $(111)\ [1\bar{1}0]$

Semelhança com texturas de outros metais cúbicos de corpo centrado foi observada.

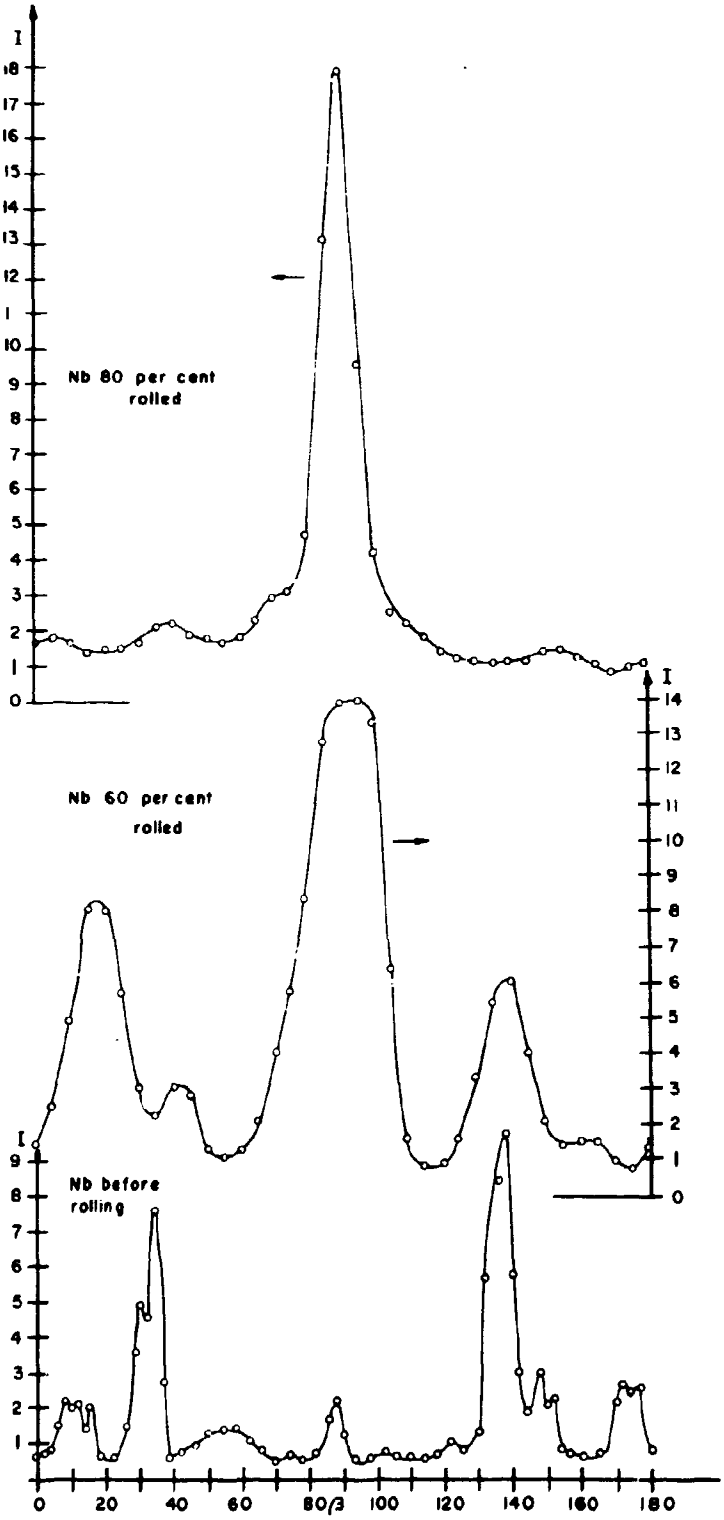

Figure 1 – Intensity versus β angle for $\alpha = 0$ in niobium as cast, Nb 60 per cent cold rolled and Nb 80 per cent cold rolled.

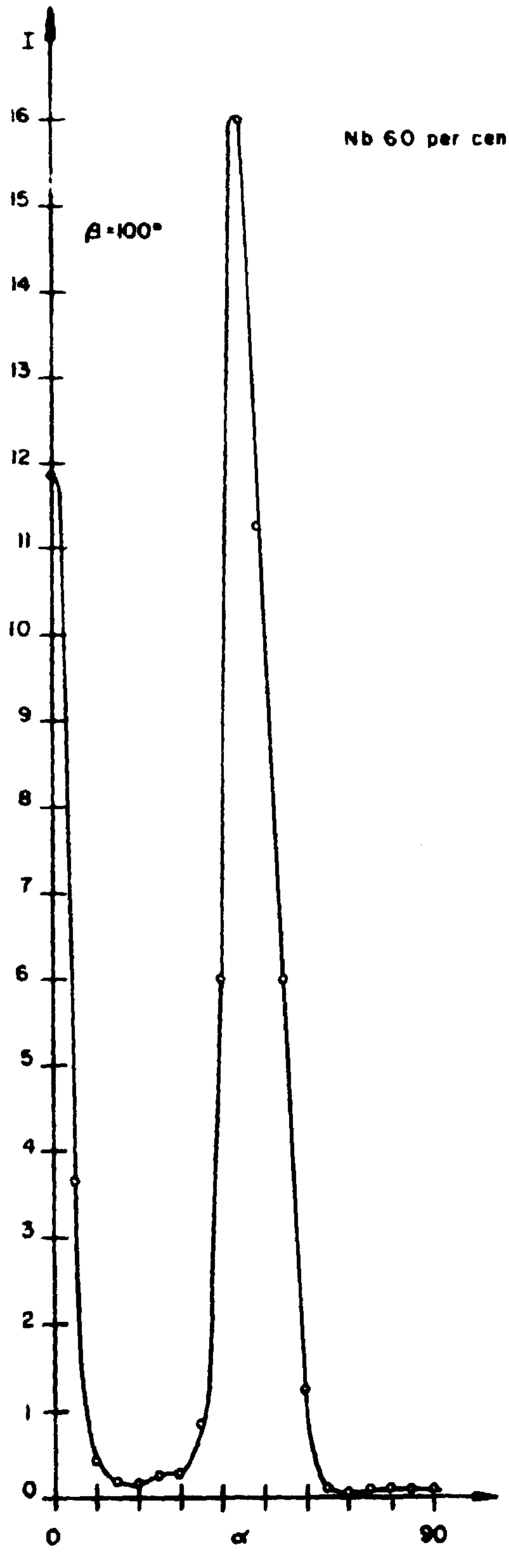

Figure 2 – Intensity versus α angle for β = 100° for Nb 60 per cent cold rolled.

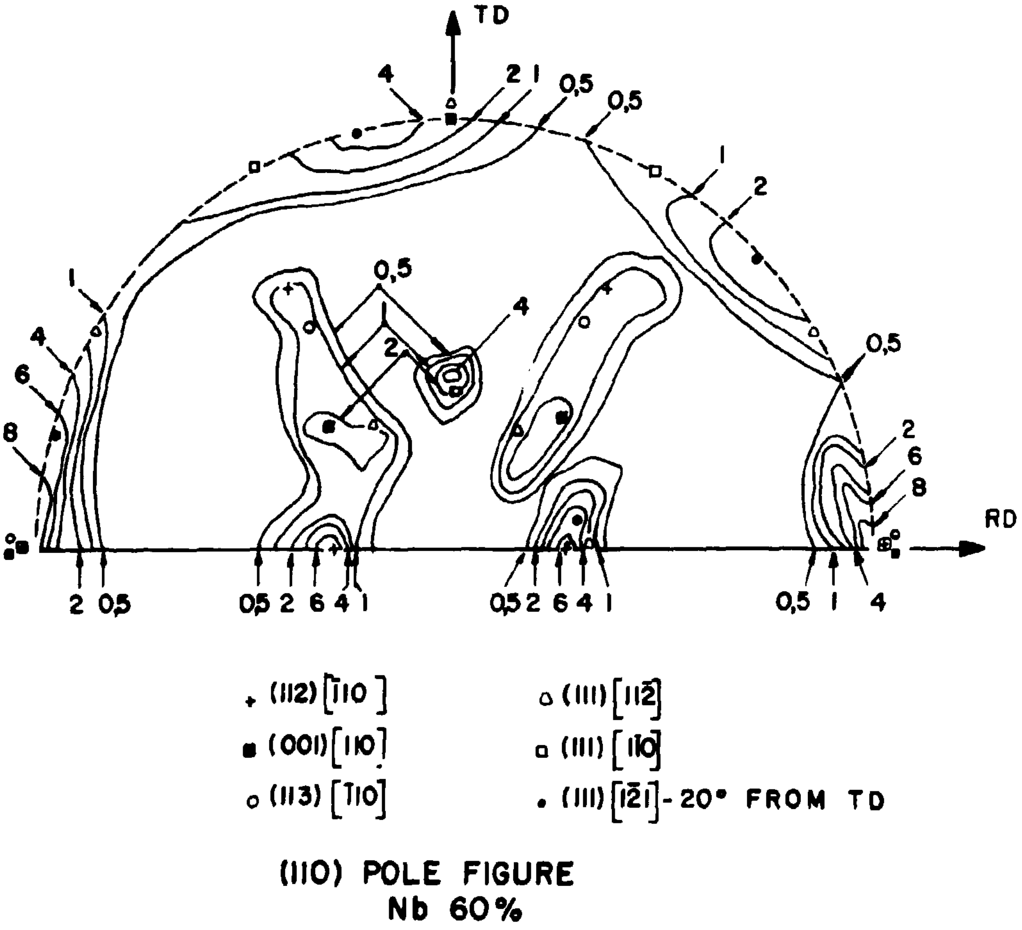

Figure 3 — Pole figure for Nb 60 per cent cold rolled.

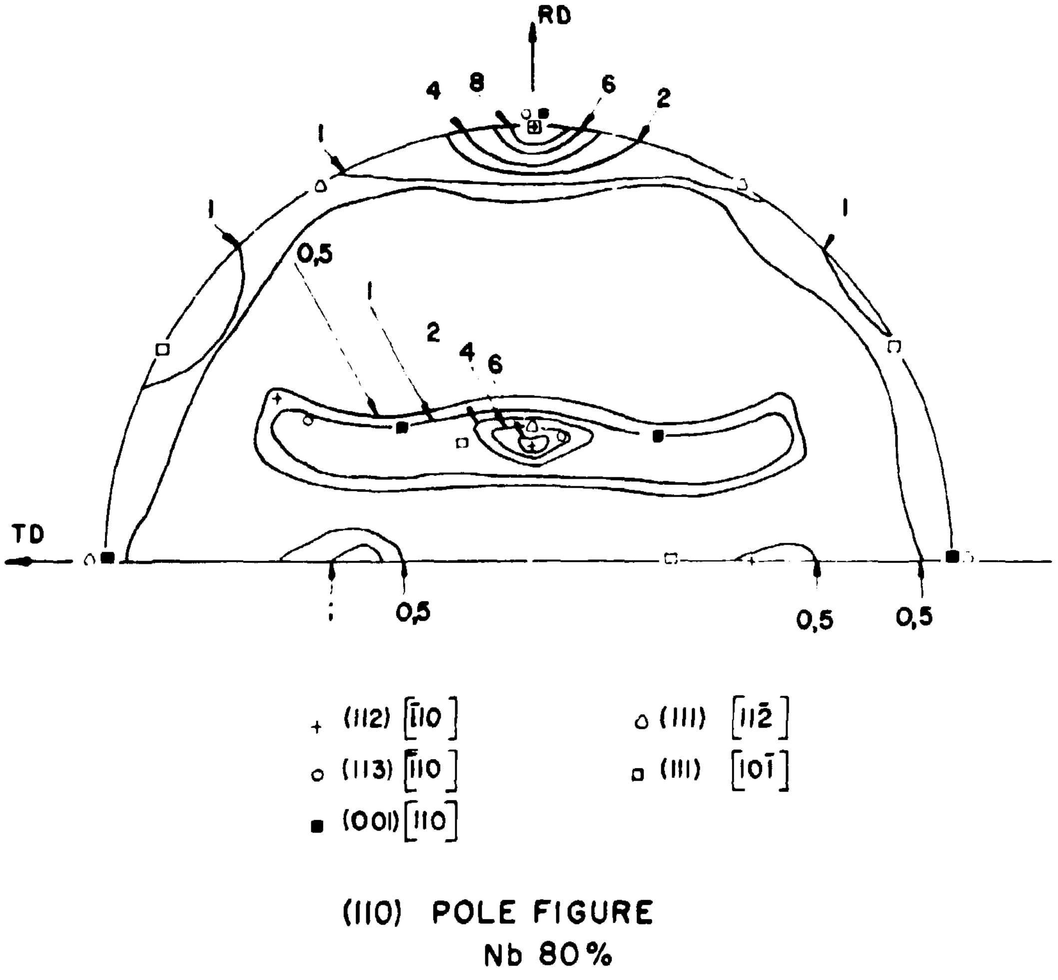

Figure 4 – Pole figure for Nb 80 per cent cold rolled.

REFERENCES

1. ANDERS JR, F. J. & POLLOCK, W. I. Tensile properties and rolling texture of niobium sheet. In: GONSER, B. W. & SHERWOOD, E. W., eds. *Technology of columbium (niobium): papers presented at the symposium on ..., of the Electrothermics and Metallurgy Division of the Electrochemical Society, May 15 and 16, 1958, Washington, D. C.* New York, Wiley, 1958. p.60-5.

2. AZEVEDO, A. M. V. et alii. *Texture determination of a MTR reactor fuel element by neutron diffraction.* (A ser publicado na série *Publicações IEA*).

3. DILLAMORE, I. L. & ROBERTS, W. T. Rolling textures in F. c. c. and B. c. c. metals. *Acta metall.*, Toronto, 12:281-93, 1964.

4. FRANK, R. G. Recent advances in niobium alloys. In: MACHLIN, I., ed. *Refractory metal alloys: metallurgy and technology.* New York, Plenum Press, 1968. p.325-72. (CONF-680448).

5. HEAL, T. J. Properties of magnesium, niobium and zirconium. In: FINNISTON, H. M. & HOWE, J. P., eds. *Metallurgy and fuels, v.4: metallurgy of nuclear reactor components.* New York, Pergamon, 1961. p.765-834.

6. KRISHNADEV, M. R. & GALIBOIS, A. An evaluation of the potentialities of columbium and suggestions for improving its market. In: SOUTHWEST RESEARCH INSTITUTE, San Antonio, Texas. *3rd interamerican conference on materials technology, August 14-17, 1972, Rio de Janeiro, Brasil.* Mexico, Centro Regional de Ayuda Tecnica, s.d. p.581-7.

7. McHARGUE, C. J. & HAMMAND, J. P. Preferred orientation in vanadium. *Trans. Am. Inst. Min. metall. Petrol. Engrs*, New York, 194:745-6, 1952.

8. MONTEIRO, W. A. & FOTEDAR, H. L. Influência da irradiação de nêutrons rápidos na recuperação e recristalização do nióbio. In: PROCEEDINGS of the 2nd international conference on materials technology, August 1976, Boston. (A ser publicado na série *Publicações IEA*).

9. PINATTI, D. G. & DAINESI, C. R. Purificação do nióbio por feixe eletrônico. [Trabalho apresentado ao 2º congresso de engenharia e ciências de materiais, São Carlos, julho 1976].

10. PUGH, J. W. The temperature dependence of preferred orientation in rolled tungsten. *Trans. Am. Inst. Min. metall. Petrol. Engrs*, New York, 212:637-42, 1958.

11. ______ & HIBBARD, W. R. Rolling textures in tantalum. *Trans. Am. Soc. Metals*, Chicago, 48:526-39, 1950.

12. SEMCHYSHEN, M. & TIMMONS, G. A. Preferred orientation of arc cast: molybdenum sheet. *Trans. Am. Inst. Min. metall. Petrol. Engrs*, New York, 194:279-86, 1952.

13. SZPUNAR, J. et alii. Texture studies of coarse-grain metal sheets by means of neutron diffraction. *Nukleonika*, Warsawa, 13:1111-8, 1968.

14. WASSERMAN, G. & GREWEN, J. *Textures metallisder Werkstoffe.* Berlin, Springer-Verlag, 1962.