Determination of Grain Size Distribution in Niobium Using an Image Analysis Routine

Jefferson F.C. Lins [1]; Hugo R.Z. Sandim[1]; Rosinei B. Ribeiro[1] and André L. Pinto[2]*
[1] FAENQUIL – Faculdade de Engenharia Química de Lorena - Departamento de Engenharia de Materiais – DEMAR, Address: Pólo Urbo-Industrial Gleba AI-06, Lorena - SP, 12600-000, Brazil, Phone: +55 (12)31599916, Fax: +55 (12)31533006; [2] IME -Instituto Militar de Engenharia – Departamento de Engenharia Mecânica e de Materiais, Address: Praça General Tibúrcio 80, Praia Vermelha, Rio de Janeiro - RJ, 22290-270, Brazil, Phone: +55 (21) 2546-7080.
[*]E-mail: fabricio.lins@ppgem.faenquil.br

Abstract

Niobium ingots obtained by electron beam melting display a typical oligocrystalline microstructure. This kind of microstructure leads to a very heterogeneous microstructure regarding final grain size and texture after deformation and subsequent annealing. Longitudinal sections of 150-mm diameter niobium ingot were cold rolled up to 80% in multiple passes and then vacuum annealed at 1200°C for 1 hour. After recrystallization annealing the average grain size was determined by two distinct methods: an image analysis routine developed for optical microscopy and by automated EBSD – Electron Backscattered Diffraction data. The results determined by both techniques were rather similar.

Keywords: niobium, grain size, EBSD, quantitative metallography, image analysis.

Introduction

It is well known that grain size determination in materials is complicated by a number of factors [1]. The difficult arises from complicate shapes, diversity in size of the elements of interest and mainly by the presence of artifacts during metallographic preparation. Furthermore, the most difficult process for correct determination of average grain size of certain metals (for instance, refractory metals and low-carbon steels) is the etching procedure necessary to reveal the grain boundaries.

Niobium ingots obtained by electron beam melting display an oligocrystalline microstructure. Noticeable differences in the deformation microstructure are observed from one grain to another indicating orientation effects. This microstructural heterogeneity exerts a strong influence on recrystallization since the stored energy varies from grain to grain. After annealing, recrystallization takes place in most of the grains, although with distinct kinetics. These features lead to a very inhomogeneous microstructure in terms of grain size and texture.

The aim of this paper is determining the grain size distribution in annealed sheets of unalloyed niobium. This can be performed by means of a routine for optical microscope using a semi-automatic image analysis system. The results obtained with this routine were compared with those determined using the EBSD – Electron Backscattered Diffraction technique.

Materials and Methods

A high-purity coarse-grained niobium ingot was obtained by means of multiple electron-beam melting. In the initial state the ingot consists of large columnar grains in the cm-range. Chemical composition of niobium is in agreement with ASTM B392-89 standard. The chemical composition of the material is shown in Table 1. A thick slab (50-mm in thick x 50-mm wide x 150-mm long) was cut out from the center of this ingot and then rolled at room temperature without intermediary annealing to a reduction in thickness of 80%. Two consecutive grains were cut out from the rolled plate. The prior grain boundary (referred to the as-cast condition) was aligned

nearly parallel to the rolling direction (RD). This sample consists of grains (A and B) and has been investigated in the cold-worked state in a previous work [2]. Vacuum annealing was carried out at 1200°C for 1 h.

Metallographic preparation of sections was carried out using conventional techniques and a final polish using colloidal silica to get good surface quality. For the chemical etching it was used an acid solution ($5HF:5HNO_3:50H_2O$) held at 273 K. The samples were etched for around 20 s to remove the surface deformation and reveal details of the microstructure. Hence, the best conditions for etching of grain boundaries were achieved when the etching time increased to about 4 min using another acid solution. For the final chemical etching was used an aggressive acid solution ($1HNO_3:1HF:2HCl$) held at 298 K. This solution requires a corrosion-resistant resin for mounting. A mixture of these acids was necessary because niobium and niobium-base alloys have good corrosion resistance.

Table 1: Chemical composition of the unalloyed niobium (wt-ppm).

Elements	W	Fe	Al	Ta	O	N
Ingot	-	-	<200	< 2000	< 50	< 30

Microstructural characterization was carried out in a LEO 1450-VP scanning electron microscope (SEM) with W-filament operating at 8 kV in the backscattered electrons mode (BSE) with electron channeling contrast (Figure 1). This technique allows identifying the misorientation between two or more grains and subgrains with good accuracy [3]. The EBSD scans were carried out in an area of about 300 x 800 μm^2 (Figure 2).

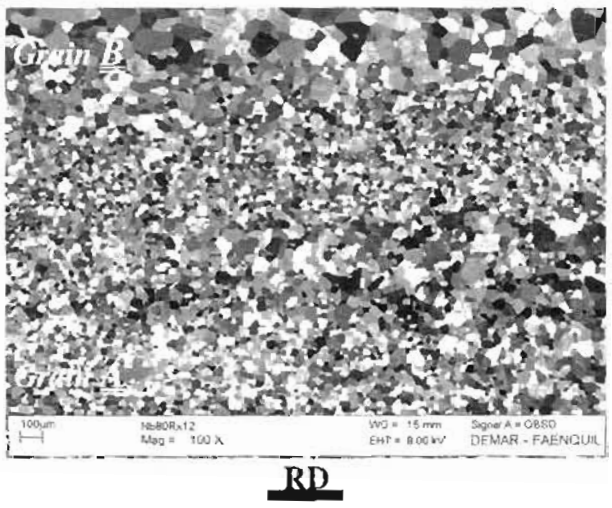


Figure 1: Electron channeling contrast image of grains A and B annealed at 1200°C for 1h (SEM-BSE, Magnification: 100x).

Grain size evaluation was determined by means of automatic indexing of Kikuchi patterns after suitable image processing in a TSL system interfaced to a JEOL JSM-5800LV SEM operating at 20 kV with W-filament. Two adjacent regions of each grain in the vicinity of the prior grain boundary were mapped.

The semi-automatic routine for optical microscope was developed with Leica Q-Win 2.3 software. Light optical microscopy images were acquired in a Leica DM IRM microscope with normal illumination (bright field). The image resolution was 640 x 480 pixels. The measurements were made in 20 fields in regions corresponding to the former grains A and B.

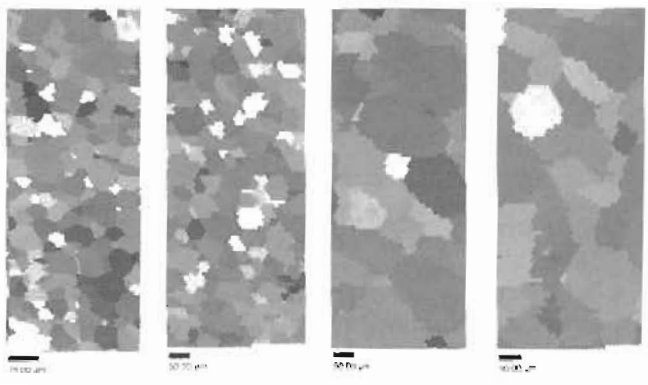
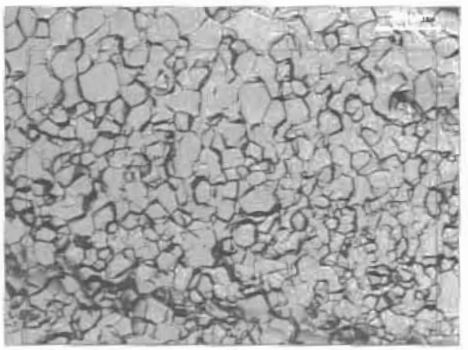


Figure 2: EBSD orientation maps showing the true grain size distribution in former: a) grain A; b) grain B.

Results and Discussion

The EBSD technique provides very accurate results, however, it has some limitations mainly due to the difficulties for suitable specimen preparation and resolution of the system [4-5]. The inaccuracy of EBSD results decreases when the magnification increases [6]. This is especially valid when fine-grained materials are being mapped, however, this cannot be satisfied during mapping of coarse-grained materials. The magnification of each region is limited to 350 times in the present work. Depending on the mean grain size (e.g. coarse grains), only a small population of grains could be mapped within each scan. The traditional quantitative metallography using optical microscopy has the advantage of providing faster results than others techniques, including EBSD image analysis. The comparative results of the mean grain size are shown in Table 2. The routine steps are displayed in Figure 3.



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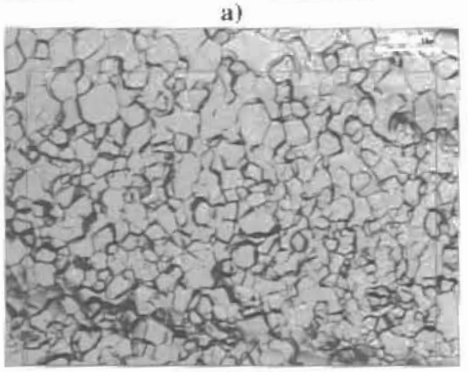

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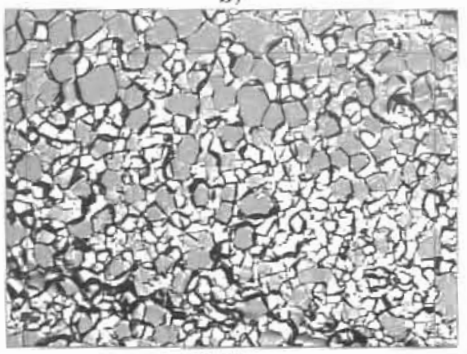

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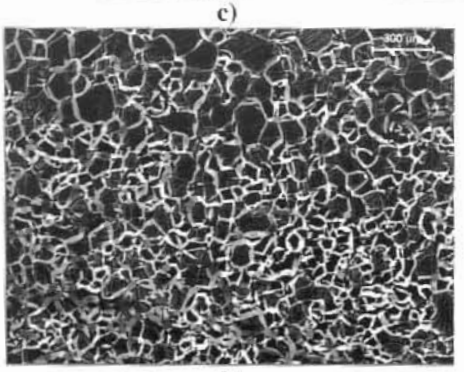

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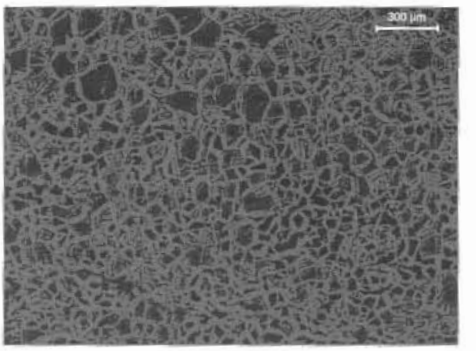

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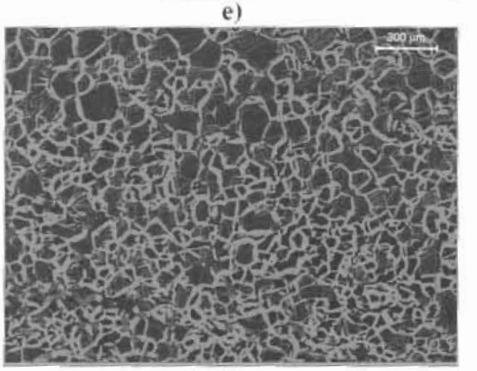

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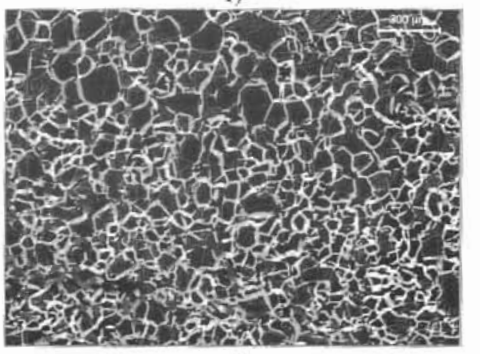

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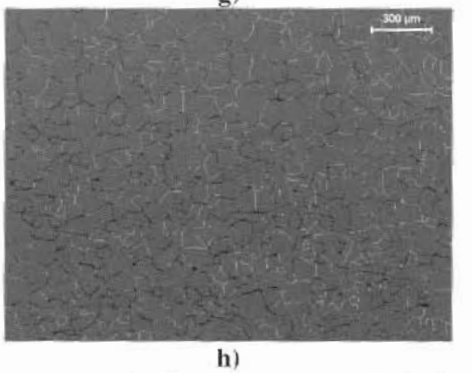

h)

Figure 3: Routine developed for light optical microscopy showing the steps for the determination of the mean grain size in a random field: a) image acquisition (Magnification: 50x); b) image equalization with median filter; c) contrast stretching with *Enhance* filter; d) image processing with high-pass filter (*BlackTopHat*); e) grey detection; f) binary image; g) skeleton from binary image; h) final result .

Table 2: Results of the mean grain size determined by the EBSD technique and by the optical routine proposed in this work.

Former Grain	EBSD technique			Optical routine		
	μm		Number of sampled grains	μm	Fields	Number of sampled grains
	Scan 1	Scan 2				
A	32	37	330	32 ± 11	20	1094
B	74	76	80	71 ± 8	20	432

The results determined using the routine developed in this work are in agreement with those provided by the EBSD image analysis software. Humphreys [4] suggests a minimum number around 200 grains for determination of true grain size with the EBSD maps. In the case of the prior grain A this was observed. However, only 80 grains could be sampled in grain B but the results found using EBSD for grain size were similar to the values obtained by our routine.

It is worth mentioning that the success of proposed routine depends on careful metallographic preparation and is very dependent on the extent of the chemical etching (etching time, temperature and concentration).

Conclusion

A comparison between the EBSD technique and a traditional standard method of quantitative metallography (optical microscopy) was performed to evaluate the routine developed to determine average grain size in niobium sheets. The results show similar values for average grain size in both grains A and B.

Acknowledgments

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