

# AN INVESTIGATION OF CORRELATIONS BETWEEN MECHANICAL AND MICROSTRUCTURAL PROPERTIES OF HIGH PURITY POLYCRYSTALLINE NIOBIUM\*

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## Abstract

Superconducting radiofrequency (SRF) cavities made from high purity niobium are commonly used in particle accelerators. An understanding of the relationship between mechanical and functional properties and the processing history is essential in order to manufacture cavities with uniform performance. The crystallographic texture and microstructure in polycrystalline sheet varies considerably, and identifying its influence on properties is needed to achieve a better understanding of how to control properties of high purity niobium. Texture (preferred crystal orientations) strongly affects mechanical properties and formability of metals and alloys. Samples received from many lots of material from two suppliers, A and B, for building cavities for the Facility for Rare Isotopes were examined to identify relationships between these two properties. Texture of the undeformed niobium samples through the thickness was measured using Orientation Imaging Microscopy<sup>TM</sup> (OIM). Texture is identified with pole figures (PF), orientation distribution functions (ODF) and grain misorientation relationships. Stress-strain testing was done to identify ultimate tensile stress (UTS), elongation, 0.2% yield strength, and the hardening rate. From tests on many lots, there is no clear trend between the mechanical and material properties in high purity niobium. Correlations between various microstructural and mechanical properties show significant scatter and few apparent correlations.

## INTRODUCTION

In order to manufacture cavities with uniform performance, understanding of the relationship between mechanical and microstructural properties is necessary. Niobium sheets can be ordered from many suppliers that meet established cavity specifications. However, the properties of the niobium sheets often show a large range of variability and exhibit random variations in properties.

It is well established that crystallographic texture is an inherent characteristic of metals produced by deformation processing and heat treatment, and has a significant influence on mechanical, physical as well as chemical properties of materials [1]. In general, there are relationships between the mechanical and material properties, which can provide useful information for

improving a manufacturing process. For high purity niobium, these correlations are still not understood. As mechanical properties change throughout the fabrication process, the correlation between mechanical properties and texture is useful to predict the mechanical properties of the final product. Understanding of these relationships should enable manufacturing cavities with similar mechanical properties. Moreover, the large data set obtained can support additional research, so that when problems arise in manufactured cavities, there is a database to consult.

During the rolling process, different strain conditions take place in the surface and center, because the shear on the surface activates slip systems that rotate crystals differently from the plane-strain compression in the center [2]. Due to these differences, the plane-strain compression typically causes  $\{111\}$  to be approximately parallel to the sheet normal direction (ND) in the middle, which is blue orientation in the default color scale of OIM maps [2]. Shear on the surface normally causes  $\{001\} \parallel$  ND, which is a red orientation [2].

## MATERIALS AND METHODS

To study the mechanical and microstructural properties of high purity niobium, samples were cut from unused material area, as shown as Figures. 1 and 2 [3]. An Instron 4302 universal testing machine was used to measure tensile behavior systematically in experiments. SEM-EBSD measurements were made on a Camscan

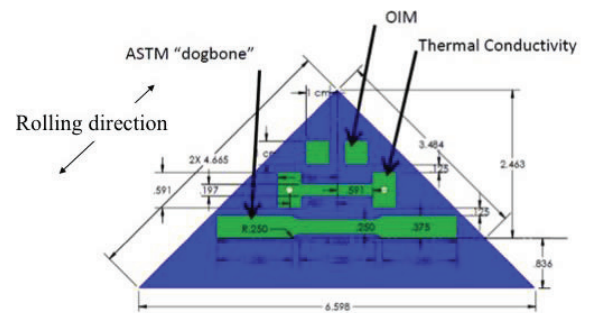


Figure 1: Layout of supplier A test samples [3].

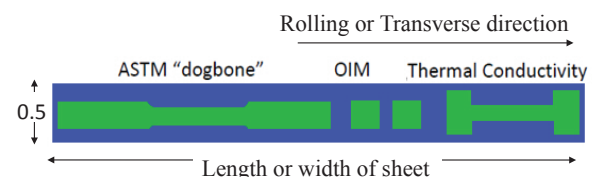


Figure 2: Layout of supplier B test samples.

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44FE field emission scanning electron microscope. Additional detailed measurements were made to investigate the repeatability of measurements made on the same lot in different locations.

Sections of the orientation distribution function (ODF) were made with  $\Phi_2$  equals  $0^\circ$  and  $45^\circ$  which illustrate fiber texture components commonly observed in BCC metals. From the exported ODF, a Matlab code was written to extract fiber data from the ODF. The  $\Phi_2 = 45^\circ$  section was used to find the maximum value, average and standard deviation along the  $\gamma$ -fiber, where  $\Phi$  is in the range of  $45^\circ$  to  $65^\circ$ .

To compute a hard-soft contrast factor related to grain boundaries, a Matlab code was written to take a ratio of [100] (soft) and [111] (hard) crystal direction components with respect to sample X, Y, Z directions for each pair of grains along each boundary. The product of  $\max[100]_i * \max[111]_j$  components in the tensile axis Y direction for  $i, j = \text{grains A, B}$ , provides a measure of how well aligned two  $\langle 111 \rangle$  and  $\langle 100 \rangle$  crystal directions are for each grain pair; the higher value of two possible outcomes was chosen. Each value was weighted by the length of the boundary. If there are more grain pairs with hard and soft orientations adjacent to each other, the contrast factor will be high. The local average misorientation (LAM), kernel average misorientation (KAM) and coincident site lattice (CSL) boundary fractions were also calculated using the TSL analysis software.

## RESULTS

To assess the consistency of the material, measurements made in three places several cm apart from each other at an edge of an OIM square and the two ends of the dog-bone sample illustrated in Fig. 1 are shown in

Fig. 3, with a set of inverse pole figure (IPF) maps (left), pole figures (PFs) (right top) and ODF plots (right bottom). The IPF maps of the three locations share strong similarity but there are some modest differences to each other. The PFs are similar and the peak positions on  $\gamma$ -fiber (high intensity bands in the lower ODF plots) have peaks in different orientations, but are otherwise similar.

From the IPF maps, the texture of niobium contains mostly preferred  $[111] \parallel \text{ND}$  orientations, which is blue in the color scale and a small fraction of  $[001] \parallel \text{ND}$ , which is red in the color scale. The differences are most apparent in the IPF maps, where some samples have distinct bands of grains with specific colors, or a mixture of colors.

The average and standard deviation of material and mechanical properties are shown in Table 1. The average UTS, elongation and average ODF intensity of all the tested samples from supplier A are slightly higher, where the supplier B lots show a higher average grain size and 0.2% yield strength. Other properties, such as the contrast factor and fraction at the peak in the LAM distribution show small differences.

## DISCUSSION

By comparing results of dog-bone and OIM square measurement illustrated for one sample in Fig. 3, as well as in a couple of other samples, the OIM square is reasonably representative of the properties of the tensile sample that was near it, so that correlations between microstructure and properties can be considered meaningful.

Formability in BCC metals is known to be good when the texture has a strong  $\langle 111 \rangle \parallel \text{ND}$  texture component (high fraction of 'blue' grains). Thus a negative correlation between elongation and the average ODF

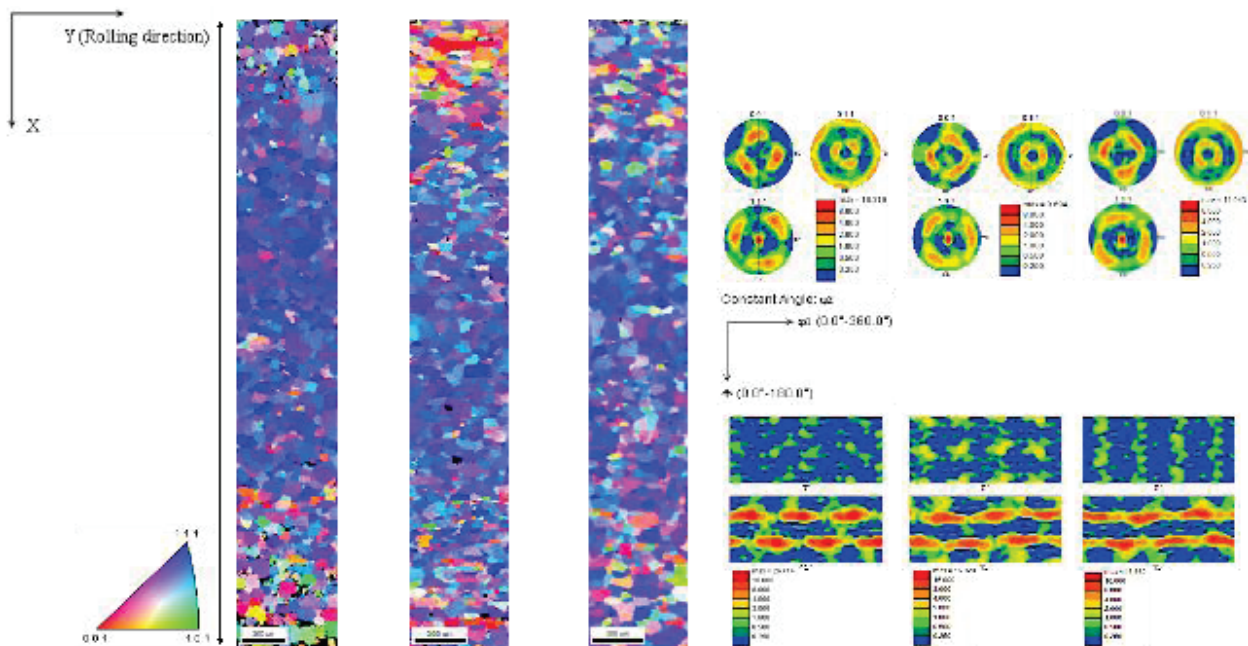


Figure 3: IPF maps, PFs and ODF slice at  $\Phi_2 = 45^\circ$  for a) OIM square, b) left dog-bone and c) right dog-bone measurements of supplier A 4499 #74.

Table 1: Summary of Average\Standard Deviation of Material and Mechanical Properties from Suppliers A and B

Supplier	Average ODF	Grain Size (Microns)	Contrast Factor Y100111	LAM Fraction	CSL Boundaries Fraction
A	3.83\1.10	35.80\6.44	0.767\0.008	0.296\0.071	0.094\0.008
B	3.27\1.46	38.29\9.15	0.789\0.011	0.305\0.104	0.092\0.011
	UTS	Elongation	0.2% Yield Strength	10% Hardening Rate	25%
A	190.28\4.98	65.32\5.12	60.52\12.14	5.18\0.49	1.73\0.37
B	181.65\6.33	61.32\4.00	70.83\10.62	4.15\0.47	0.80\0.19

value on the  $\gamma$ -fiber (in the  $\Phi_2 = 45^\circ$  section), is expected, but there is no apparent correlation for the supplier A samples in Fig. 4. There is a modest correlation for the supplier B samples deformed in the rolling direction ( $0^\circ$ ) in Fig. 4. The presence of the correlation in the rolling direction is most easily rationalized because the deformation dominant in rolling, of stretching in the rolling direction is also what occurred in the tensile test, so the grains have an orientation that reflects this

directionality. The supplier A and  $90^\circ$  supplier B samples had the majority of grains deform in a direction that differed from extension in the rolling direction.

A similar correlation is apparent in Fig. 5 where the relationship between the UTS and average grain size is shown. There is a similar modest relationship between the UTS and the grain size for samples cut parallel to the rolling direction in supplier B samples. Since the  $\gamma$ -fiber is fairly strong in these samples due to the rolling process,

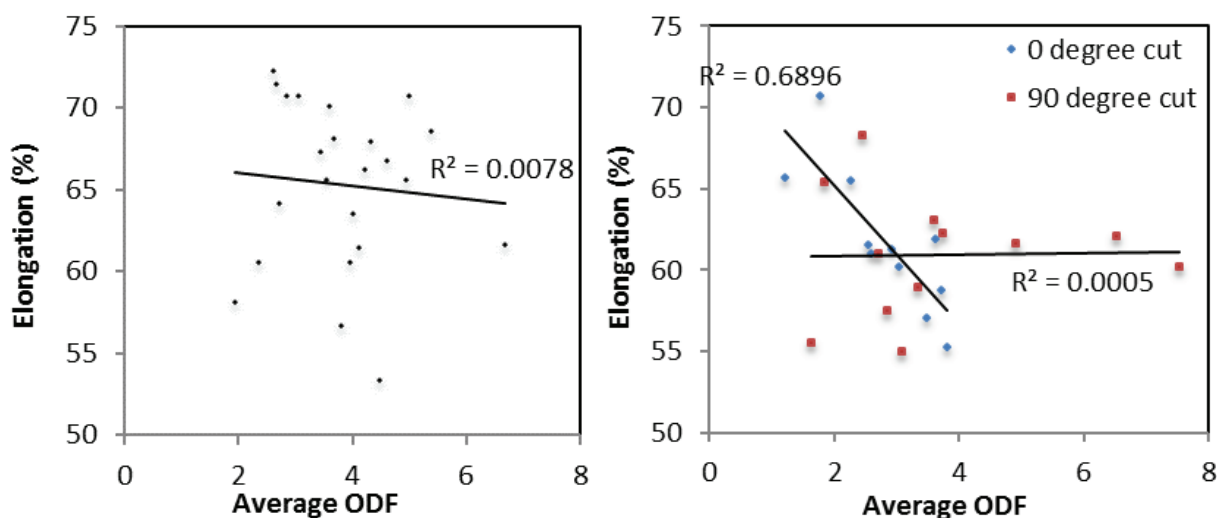


Figure 4: Correlation between elongation (%) vs.  $\gamma$ -fiber average ODF intensity for suppliers A (left) and B (right).

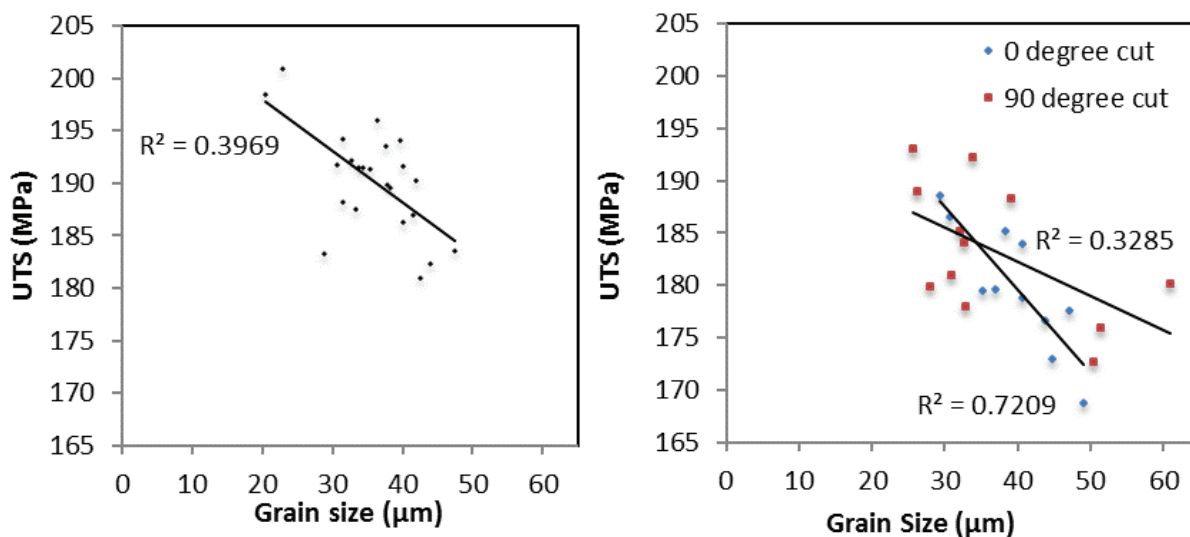


Figure 5: Correlation between UTS (MPa) vs. grain size in diameter ( $\mu\text{m}$ ) for suppliers A (left) and B (right).

continued deformation in the same direction is sensitive to the evolved microstructure, but when grains are deformed in directions differing from the prior rolling direction, there is a weaker influence of the microstructure on the flow stress. These plots taken together show that in the rolling direction, the stronger the material, the smaller the elongation, and with a smaller grain size, there will be more grain boundaries, and the resulting UTS will be larger. As formability in steels is improved with a strong  $\gamma$ -fiber, this result for niobium differs from steel.

These plots show the strongest evidence of correlation for all of the microstructural quantities plotted, so the lack of a clear relationship between microstructure and properties indicates that deformation processing niobium are not easily predicted. All the results suggest that niobium could have unique properties compared to other metals. Future characterization work with more consistency in cutting test samples with respect to the rolling direction may help elucidate other relationships, or further demonstrate the lack of correlations observed here.

Even though few trends can be drawn on the correlation plots, all of the lots met qualifications specified for acceptance criteria, so no reasons for concern are expected in manufacturing SRF cavities. It is expected that, except for implications of cost, SRF cavities should be made from samples that having similar properties. This assessment provides a measure of the range of properties that result from existing acceptance criteria, and provide a database for comparing the quality of cavities that are manufactured for the FRIB. Thus, this information may enable new correlations between performance and microstructure to be identified in the future, and thus may provide a basis for making improvements in specifications for niobium sheet metal for SRF cavities.

## CONCLUSIONS

The correlation between mechanical and microstructural properties of high purity niobium was examined. Instead of a clear trend, weak correlations were identified between grain size, strength, and the strength of the  $\gamma$ -fiber in samples deformed in tension in the rolling direction; otherwise, all scatter plots provided no appreciable correlation between microstructural metrics and mechanical properties. To achieve the goal of making uniform grains and texture, further analysis on different ways to quantify texture are needed and more sampling for consistency in measurements for each lot should be used. Moreover, samples having significantly different properties to make different cavities should be tracked to examine whether there is a difference in the performance, to provide further improve understanding of the relationship between performance, microstructural properties, and mechanical properties.

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