

# INTEGRATED SURFACE TOPOGRAPHY CHARACTERIZATION OF VARIOUSLY POLISHED NIOBIUM FOR SUPERCONDUCTING PARTICLE ACCELERATORS\*

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## Abstract

As superconducting niobium radio-frequency (SRF) cavities approach fundamental material limits, there is increased interest in understanding the details of topographical influences on realized performance limitations. Micro-and nano-roughness are implicated in both direct geometrical field enhancements as well as complications of the composition of the 50 nm surface layer in which the super-currents flow. Interior surface chemical polishing (BCP/EP) to remove mechanical damage leaves surface topography, including pits and protrusions of varying sharpness. These may promote RF magnetic field entry, locally quenching superconductivity, so as to degrade cavity performance. A more incisive analysis of surface topography than the widely-used average roughness is needed. In this study, a power spectral density (PSD) approach based on Fourier analysis of surface topography data acquired by both stylus profilometry and atomic force microscopy (AFM) is being used to distinguish the scale-dependent smoothing effects. The topographical evolution of the Nb surface as a function of different steps of EP is reported, resulting in a novel qualitative and quantitative description of Nb surface topography.

## INTRODUCTION

Surface topography impacts performance through local loss of superconductivity (also called quenches) caused by intrusion of the rf magnetic field, which is enhanced at sharp protrusions [1]. "Sharpness" comprises both vertical and lateral aspects, requiring development of a new characterization approach beyond the familiar roughness measurements ( $R_a$ ,  $R_q$ ,  $R_z$ ), which view only the vertical component and are strongly dependent on the scan size and the particularities of the area being scanned [2]. Especially it is unknown at what scale roughness is important to SRF performance. Seeking such an analysis, the roughness of the niobium surface has been measured at a range of sizes and resolutions, which can only be accomplished using several different instruments. In this study, a systematic surface topography characterization was conducted through stylus profilometry and atomic force microscopy. In order to combine data from scales accessible to these instruments, the power spectral density

analysis technique is explored. The evolutions of the surface topography and PSD after an incremental series of electropolishing are discussed.

## EXPERIMENTAL STUDIES

### Sample Preparation

The samples were high purity polycrystalline Nb slabs with dimensions of 22 mm x 22 mm x 4 mm. All were mechanically ground using 1  $\mu$ m grit prior to any chemical treatment, as is done in the cavity production at Jefferson Lab. The BCP solution was a 1:1:2 (by volume) mixture of HNO<sub>3</sub> (69%), HF (49%) and H<sub>3</sub>PO<sub>4</sub> (85%). The EP solution was a 1:9 mixture of HF (49%) and H<sub>2</sub>SO<sub>4</sub> (96%). For electropolishing, the Nb sample and a high purity aluminium plate with the area ratio of 1:2 were inserted into the Teflon clamps with a separation of 98 mm. For each step of electropolishing, samples were subjected to 30 minutes of EP at 6V; the polarization potential is relative to a MSE reference electrode, with electrolyte temperatures of 30°C.

### Surface Roughness Measurement

Profilometry measurements were obtained with a stylus profilometer (KLA-Tencor: P-15) with a 2  $\mu$ m diameter tip. The samples were scanned in three different regions with a scan size of 200  $\mu$ m x 200  $\mu$ m and 1000  $\mu$ m x 1000  $\mu$ m. The 200  $\mu$ m x 200  $\mu$ m scan was taken as an array of 101 traces with 401 points in each trace, and the 1000  $\mu$ m x 1000  $\mu$ m scan was taken as an array of 251 traces with 2501 points. AFM measurements were performed using a commercial AFM (Digital Instruments: Nanoscope IV) in a tapping mode using silicon tips with a diameter of 10 nm. The samples were scanned in three different regions with scan sizes of 20  $\mu$ m x 20  $\mu$ m, 50  $\mu$ m x 50  $\mu$ m, and 100  $\mu$ m x 100  $\mu$ m. The AFM images were captured as arrays of height values with 256 x 256 points.

### Power Spectral Density

Power spectral density has been used as a tool to combine measurements from different scales and different instruments [3-4]. It represents the spatial-frequency spectrum of surface roughness measured in inverse-length units. The PSD can be computed as the square of the Fourier transform as equation (1) where the surface

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topography data are  $h(x, y)$  and the PSD variable

$$PSD(f_x, f_y) = \lim_{L \rightarrow \infty} \frac{1}{L^2} \left| \int_{-L/2}^{L/2} \int_{-L/2}^{L/2} h(x, y) e^{-2\pi i(f_x x + f_y y)} dx dy \right|^2 \quad (1)$$

$f_x$  and  $f_y$  are the spatial frequencies of the surface roughness and are related to the lateral dimensions of the surface features. From the above equation, we know that the PSD gives information about the relative contributions of all the possible surface spatial frequencies for an ideal measurement of an infinite surface in the limiting case from 0 frequency (an infinite surface) to an infinite frequency (infinitely small structure) [3]. Or, in the case of discrete data consisting of N points separated by  $\Delta x$ , it can be computed as in equation (2). A more complete

$$PSD(f_x)_N(m) = \left| \left( \frac{\Delta x}{N} \sum_{n=0}^{N-1} h(x)_n \exp(-i2\pi nm / N) \right) \right|^2 K(m) \quad (2)$$

mathematical description can be found in ref. [3-7]. The PSD not only represents the squared amplitude of surface features plotted against the spatial frequency of those features, but also provides information about both the lateral and vertical size of features. The range of spatial frequencies over which the PSD is valid for a certain scan size is determined by the Nyquist limits [3, 8]. In this study the power spectral density for all traces in the fast scan direction were averaged to approximate the true 2D PSD. This assumption is valid in the case of a surface with isotropic roughness [3]. In order to eliminate the measurement errors and increase the goodness of statistical representation of the whole surface, the PSD profiles measured at different locations under the same scan condition were averaged together. The PSD profiles from the profilometer data were filtered using a 4-sample moving average in order to eliminate spurious high-frequency noise [8].

## RESULTS AND DISCUSSION

Figure 1 shows the PSD of combined AFM and profilometry data from the sample with 5 minutes of BCP treatment at different scanning areas (profilometry :  $200 \mu\text{m} \times 200 \mu\text{m}$  and  $1000 \mu\text{m} \times 1000 \mu\text{m}$ ; AFM :  $20 \mu\text{m} \times 20 \mu\text{m}$ ,  $50 \mu\text{m} \times 50 \mu\text{m}$ , and  $100 \mu\text{m} \times 100 \mu\text{m}$ ). The results show that the spatial frequency ranges corresponding to the different measurements are largely overlapped and the PSD functions using the two techniques agree very closely over the range of frequencies that are common to both instruments. This shows that the PSD is a method which can combine surface profile information from different instruments in a consistent and useful manner [3, 8]. At the high-frequency end of the spectra, the slope of all the graphs shows a decrease and begins to level off. This is likely an effect of limited resolution for the AFM and the finite tip size for the profilometry [9]. Of particular interest in this figure is the change of slope that occurs at a spatial frequency approximates  $0.04 \mu\text{m}^{-1}$ . Changes in slope of the PSD are related to the inverse of the correlation length of the sample. The correlation length sets the scale at

which surface features are similar [8, 10-11]. For this data, the correlation length is approximately  $25 \mu\text{m}$ , which is on the order of the size of niobium grains in the polycrystalline material studied [12].

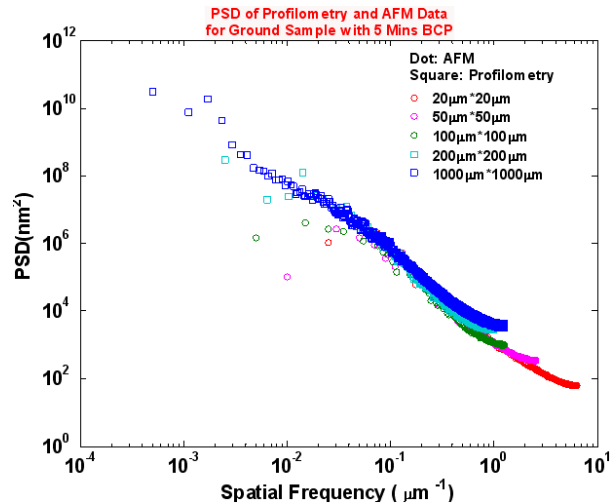


Figure 1: Combined AFM and profilometer power spectral densities from all scan sizes from the sample having undergone 5 minutes of BCP treatment.

An AFM topography and PSD comparison of the ground samples after receiving an incremental series of electropolishing treatment are shown in Figures 2, 3, and 4. The power spectra were calculated from the  $100 \mu\text{m} \times 100 \mu\text{m}$  AFM scans and  $1000 \mu\text{m} \times 1000 \mu\text{m}$  profilometry scans. We found that the amplitude of the PSD decreases dramatically after 30 minutes EP and continues to decrease less rapidly thereafter. As related to the AFM images, it indicated that the surface protrusions caused by mechanical grinding were quickly levelled out by short period of EP, the edges of grain boundaries and recessed parts of surface were continually suppressed with duration of electropolishing. Furthermore, the overlap of the PSD plots after 60 minutes EP in the high-frequency region indicates that there is no further significant surface smoothing at the small scale under the applied conditions. (This corresponds to the surface features which result in  $0.01 \mu\text{m}^{-1}$  or higher spatial frequency; the correlation length is approximately  $10 \mu\text{m}$  or smaller). The increased correlation length of the ground sample subjected to longer EP suggests that the rougher surface at larger scales has gradually been smoothed out.

## CONCLUSIONS

We have demonstrated for the first time the use of power spectral density in quantitatively characterizing the surface topography of niobium. PSD measurements allow for the combination of data taken from different instruments and at different length scales. They also allow for a detailed analysis of the surface properties of niobium in both the vertical and lateral dimensions, information otherwise inaccessible to classical methods of quantifying

roughness. We have directly compared the PSD after incremental series of well-controlled electropolishing, and noted the scale-dependent differences. Our studies show that the power spectral density provides enhanced information about the surface from the most sources, and so should be the preferred diagnostic when analyzing surface roughness. This tool will therefore allow for improved characterization and analysis in the production of SRF cavities. Use of this tool to characterize the effect of different process condition on different start materials has begun and will be reported elsewhere.

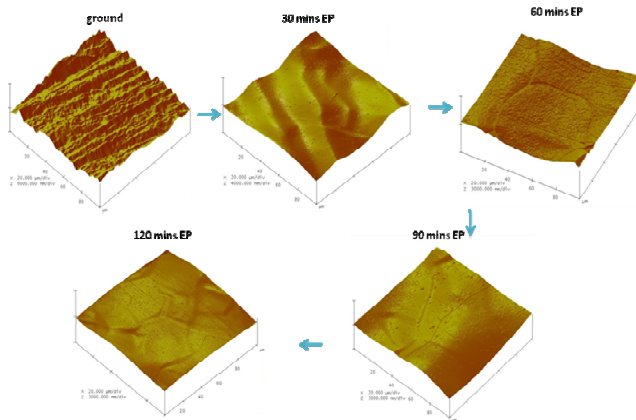


Figure 2: The AFM images ( $100\ \mu\text{m} \times 100\ \mu\text{m}$ ) for incrementally electropolished ground niobium sample.

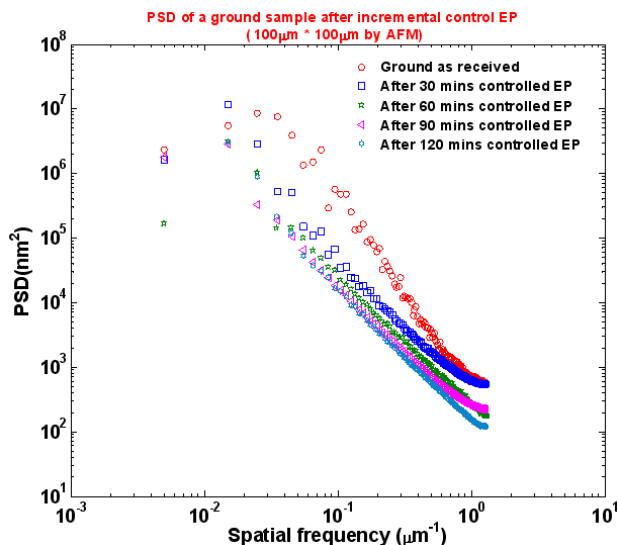


Figure 3: Power spectral density plots images ( $100\ \mu\text{m} \times 100\ \mu\text{m}$ ) for incremental electropolished ground niobium sample.

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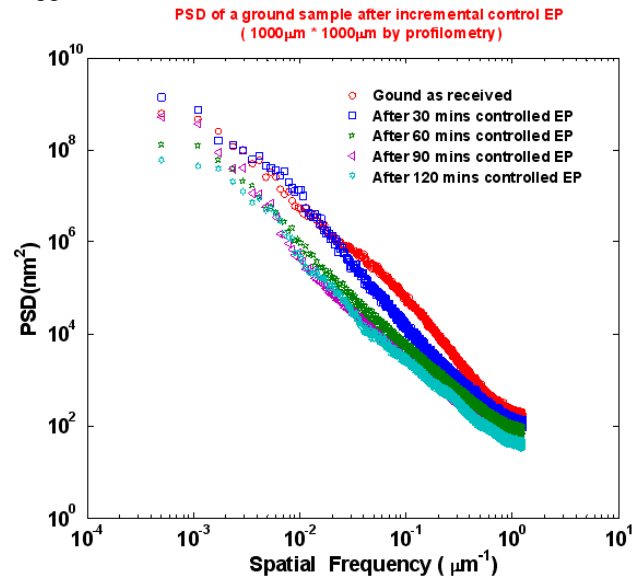


Figure 4: Power spectral density plots images ( $1000\ \mu\text{m} \times 1000\ \mu\text{m}$ ) for incremental electropolished ground niobium sample.

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