CHARACTERIZATION OF SCALE-DEPENDENT ROUGHNESS OF NIOBIUM SURFACES AS A FUNCTION OF SURFACE TREATMENT PROCESSES*

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Abstract

Microscopic topography is attributed to be a critical issue for realizing optimum performance of superconducting radio frequency (SRF) cavities. Several surface processing methods such as chemical, mechanical and plasma are used to obtain relatively smooth surfaces. Among those process methods, buffered chemical polish (BCP) and electropolishing (EP) are most commonly used in current niobium SRF cavity production. The power spectral density (PSD) of surface height data provides a thorough description to the topography measurement and reveals useful information including fractal and superstructure contributions.

Polishing duration and temperature can have predictable effects on the evolution of such features at different scale regions in PSD spectrum. In this study, one dimensional average PSD functions derived from the topography of niobium surfaces treated by BCP and EP with different controlled starting conditions and durations have been fitted with a combination of power law, K-correlation and shifted Gaussian models, to extract characteristic parameters at different spatial harmonic scales.

INTRODUCTION

Particle accelerators play a steadily increasing role in a steadily expanding range of scientific research. Their greater capabilities and superior cost for performance in many instances results in even more rapid growth for accelerators using superconducting radio frequency (SRF) cavities to power the beam. Topography of the cavity internal surface can be a major issue for cavity RF performance, thus different treatments are developed and applied to achieve high accelerating gradient.

To correlate topography and performance, a means of acquiring and analyzing topography data is needed. Data acquisition so far has been by stylus profilometer (SP) or atomic force microscopy (AFM). The data set is the vertical position of the probe at the sequence of lateral positions comprising the scan. Data analysis may be as simple as the average displacement from the mean vertical position (R_a, roughness), which has proven insufficiently incisive for SRF purposes. The next level of analysis is to condition and Fourier transform the scan data to display the contribution at each lateral dimension, the power spectral density (PSD).[1] The approach can be extended by separating the PSD into contributions related to families of surface features, as is done in the optics community.[2] These and related analyses probe the average characteristics of the surface. They are relatively insensitive to a small number of singular features, though a single major protrusion or pit may cause poor cavity performance. For these, a kind of approach that views singularities is needed.[3]

We report here two sets of experiments relevant to SRF cavity processing to show what can be revealed by the more extensive data processing approaches.

EXPERIMENT

Materials

Two fine grain samples were polished as for metallography. (nanopolished, NP), then subjected to BCP etching with very short duration to study the initial steps of BCP polishing.

Four other fine grain samples (FG) were subjected to BCP etching sufficient to remove mass equal to 100 μm thickness from the interior surface. All fine grain specimens used here were cut from untreated sheet as 10 mm × 10 mm squares. [2]

Treatments

Genesis of topography in BCP etching: Initial effect of etching

Two nanopolished NP specimens were examined as-received, after BCP with solution 1:1:2 for a time expected to remove 3 μm and the same BCP treatment applied to another two NP fine grain samples after a time expected to remove a further 3 μm.

Smoothing in EP: Initial smoothing

Four fine grain specimens, FG, subjected 100 μm BCP, were then subjected to EP treatments at 20 °C expected to remove 5 μm, 10 μm, or 15 μm one each, respectively. These conditions were chosen to explore the early short duration of EP such as is used in the latest final production etch practice of the CEBAF 12 GeV Upgrade. [3,4]

Characterization

The topography was examined by optical microscopy and atomic force microscopy (AFM) Digital Instruments

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Nanoscope IV using tips with diameter <10 nm, eigenfrequency 75 kHz and Young's modulus 7.5 nanoN/m. A typical AFM dataset consists of a (512 × 512) matrix of data points. The lateral resolution of the AFM is determined by scan length/sampling points. In this experiment, the resolution for scan of 100 μm is 195 nm. Two areas were scanned on all as received Nb samples materials and at least four on all that were treated.

RESULTS

Case 1: NP Fine Frain Polycrystalline Niobium with Initial BCP Treatment

The development of sharp stepped features even at this early stage of etching is noticeable. Presumably the steps are grain boundaries, but specific evidence is needed. A particularly significant aspect is that they appear to be elevation changes, not grooves. The observed topography appears to be more consistent with differential etching of adjacent grains than with grain boundary attack. Table 1 shows the steady increase of RMS roughness R_q and local angle D_a as material is progressively removed. Note with increasing duration of BCP, the roughness increases around 20nm/μm, while the local angle increases around 0.25°/μm.

Table 1: Roughness and local angle of "NP"

Initial BCP(nm)	Nanopolished	+3μm BCP	+6μm BCP
R_q(nm)	41	79	127
D_a (°)	0.7	1.9	3.2

The averaged PSD of each sample at each sample at each stage is presented in Figure 1. From inspection of Fig.1, we observe that the PSD's of the nanopolished NP samples as-received show a straight line character at mid and high frequencies with curvature at low frequencies, nominally, a power law and a shelf. Presuming that nanopolishing is some variant of metallographic polishing, this topography could reflect random roughening at a very low level together with the signature of the polishing process (e.g., wheel vibration).

The impact of BCP is conditioned by the presence of grain boundaries, a candidate for selective attack. The PSD shows continuing evolution toward the power law character reflecting a fractal or stepped surface structure.

The features of most concern for SRF performance are the pronounced sharp edges at the apparent grain boundaries.

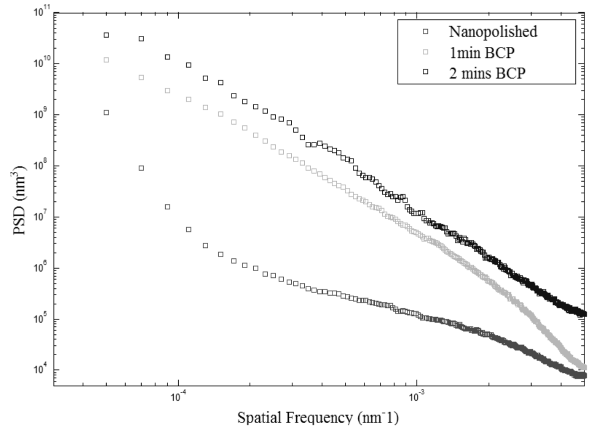

Fig. 1: Average 1D PSD on nanopolished FG samples between nanopolished, 3 μm and 6 μm BCP removal PSD.

As noted above, 1:1:2 fresh BCP solution removes mass at a rate equivalent to a surface recession velocity of 3 μm/min at 25°C.[5] Two minutes BCP then results in about 6 μm average thickness removal, implying a nominal height difference no more than several microns. In this two minutes BCP treatment, however, the step height difference increases from 22nm→379nm→414nm (Fig.4). A large step develops quickly and maintains prominence with further etching. The limited scan length of AFM (100 μm) vs. grain size (50–100 μm) impedes obtaining a statistically significant assessment of the distribution of step spacing and height. The application of other approaches is needed.

Case 2: Initial EP Conversion of Heavily BCP Treated FG Samples

Taking the view that process determines topography, it is important to learn how rapidly a change of process (BCP to EP) reaches steady state. Four fine grained Nb samples FG were subjected together to 100 μm removal by BCP. One was kept as a BCP record and each of others was electropolished at 20°C to remove 5μm, 10μm, and 15μm, respectively. Roughness and local angle are given in Table 2 with AFM scan 50 μm × 50 μm.

Note that with increasing duration of EP, the roughness and local angle change little (Table 2). Interestingly, optical microscopy (Fig. 2) shows step-like features at 10 μm EP stage that are not evident after longer EP.

Table 2: Roughness and local angle of "FG"

Initial EP(nm)	100 μm BCP only	+5 μm EP	+10 μm EP	+15 μm EP
R_q (nm)	169	163	162	138
D_a (°)	2.8	3.5	3.3	3.3

Fig. 2: Optical microscopy; probable steps are circled.

After 5μm removal by EP, the PSD has significantly transformed from power law structure into the shelf structure (Fig. 3). The effect is far more dramatic in the PSD plot than in the roughness data. Net removal of a few microns cannot be expected to significantly change the height variation, which underlies the roughness values. However, localized attenuation of sharp projections can have a major impact on the power law contribution. Further, the PSD intensity in the frequency range 4×10^{-4} – 4×10^{-3} nm^{-1} (few tenths to few micron lateral scale) actually increases as material is removed by EP.

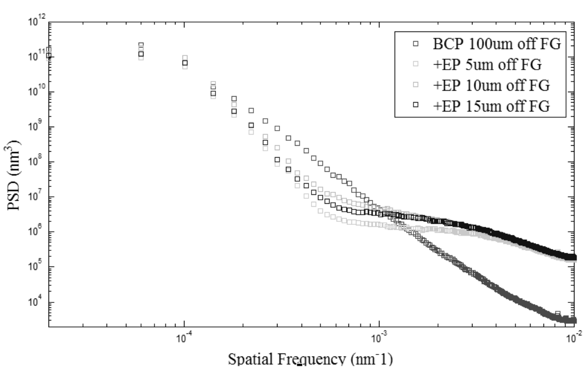

Fig. 3: PSD after different short electropolishing durations with initial state of buffered chemical polishing (AFM scan size 50 μm × 50 μm) at 20^{o}C.

DISCUSSION

The major change in PSD experiments occurs over the frequency range from 10 μm-1 to 1 μm-1 in both studies. The inverse Fourier transform over this range yields a profile of the surface features that contribute to this change in the PSD spectrum (Fig. 4).

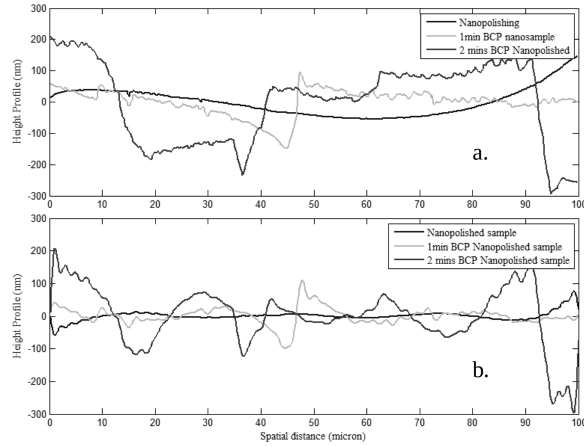

Fig. 4: Line scan (a) and surface inverse transform from 1 μm to 10 μm (b).

With this inverse transform, one can tell both BCP and EP at early stage are sensitive to the grain boundary features whose lateral range are 1 μm to 10 μm.

Another feature is the crossover of PSD's for EP and BCP in the neighborhood of 1 μm^{-1}. The apparent meaning is that at small length scale, BCP is smoother and the reverse at longer scale. The EP process is understood to accomplish levelling by depletion of the active fluoride ion species in a layer adjacent to the surface having a thickness of a few tens of microns, depending on specifics.

CONCLUSIONS

Detailed analysis of the power spectral distribution sheds additional light for understanding of Nb surface morphology under cavities production and raises further questions, potentially having practical significance. Significantly more information is needed.

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