ANALYSIS OF THE TOPOGRAPHIC TRANSFORMATION OF NIOBIUM SURFACES UNDER CONTROLLED EP CONDITIONS*

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Abstract

As the field requirements of niobium SRF cavities approach fundamental material limits, there is increased interest in understanding the details of topographical influences on realized performance limitations. In this study, a set of samples representing 24 different starting conditions used in cavity processing has been assembled. This set includes fine grain, large grain, and single crystal Nb samples under electron beam welding (EBW), hand grinding, and centrifugal barrel polishing (CBP) with a variety of stones, the latter provided by KEK colleagues. Sample topography has been carefully characterized in both the initial condition and after removal of 30 microns via well-controlled EP. A power spectral density (PSD) approach based on Fourier analysis of surface topography, stylus profilometry (SP) and atomic force microscopy (AFM) is used to distinguish the scale-dependent smoothing effects. The detailed topographic transformation of Nb surface with the varied starting state of the Nb surface is reported. This study will help to identify optimum EP parameter sets for controlled and reproducible surface levelling of Nb for cavity production.

INTRODUCTION

The theoretical and experimental evidence suggests that the ideal surface for the interior of an SRF cavity would be atomically smooth except for the curvature needed to accommodate the cavity shape. Since the rf super-current flows in the top 50nm of the surface, topographic structure approaching this scale is expected to matter as SRF cavity performance approaches theoretical working limit. In order to provide a systematic characterization, R_a-mean roughness and R_q-mean square root roughness are widely employed metrics to describe Nb surface roughness and is proved useful. However, their values are dependent on the scan size and the particularities of the area being scanned, and the results are generally different because of the spatial-frequency bandwidth limits. We have recently completed the application of power spectral density method to Nb surface topography [1]. It is a method of not only combining the measurements from different diagnostic instruments, but also is less dependent on instrumental effects when one measures parameters such as surface roughness and correlation length [2, 3]. Briefly, a series of AFM and SP scans are obtained from several surface locations of the specimens according to the protocol developed, and the squares of the Fourier transforms of the measured surface heights are combined as a plot of roughness PSD versus spatial frequency (inverse of wavelength).

Prior to application of the final electropolishing step for niobium SRF cavities, various methods have been used to remove the so-called surface damage layer. Examples include 20μm removal by BCP for TESLA Test Facility (TTF) cavities [4], a proposed 45~80μm removal by EP for the X-ray free-electron laser (XFEL) in Europe and International Linear Collider (ILC) cavities [5], 80~100μm removal by BCP used for Jefferson Lab cavities [6], and 25~200μm removal by centrifugal barrel polishing (CBP) for some of High Energy Accelerator Research Organization (KEK) cavities [4-6]. However, the effect of the starting surface condition before electropolishing has not been systematically investigated. As a result, a study of surface variation with different treatments under well-controlled EP would be very helpful in the overall improvement of surface finishing and the practical processing costs.

In this study, a full representative set of samples representing 24 different starting processing conditions has been assembled and treated under well controlled EP parameters. The topographical transformation of Nb surface has been systematically characterized by optical microscopy, AFM, SP and analyzed by roughness PSD. The study is expected to provide statistic evidence for indentifying the optimum process conditions for controlled and reproducible surface finishing of Nb for cavity production.

EXPERIMENTAL STUDIES

Sample Preparations

The samples were high purity polycrystalline, large grain and single crystal Nb. The detailed treatments for this set of samples are listed in the Table 1. The BCP solution was a 1:1:2 (by volume) mixture of HNO_3 (69%), HF (49%) and H_3PO_4 (85%). The EP solution was a 1:10 mixture of HF (49%) and H_2SO_4 (96%). For electropolishing, three Nb disk samples were mounted to a customized sample holder subjected to 100 minutes of EP at 10 volts simultaneously. The bulk electrolyte temperature was controlled by a circulated water bath at 30±1˚C, and the removal for each of sample is about 30±3μm for each of samples measured by SP.

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Table 1: The Detailed Treatments of 24 Different Starting Conditions Samples Used in this Study [7]

				CBP Details			
No.	Type	EBW	Treatment	Rough stone	Middle stone-1	Middle stone-2	Fine stone
KEK-1	FG	No	CBP(rough only)+degreasing	6hrs			
KEK-2	FG	No	CBP(fine)+degreasing	6hrs	6hrs	6hrs	6hrs
KEK-5	FG	Yes	CBP(rough only)+degreasing	6hrs			
KEK-6	FG	Yes	CBP(fine)+degreasing	6hrs	6hrs	6hrs	6hrs
KEK-8	LG	No	CBP(rough only)+degreasing	6hrs			
KEK-9	LG	No	CBP(fine)+degreasing	6hrs	6hrs	6hrs	6hrs
KEK-14	LG	Yes	CBP(rough only)+degreasing	6hrs			
KEK-15	LG	Yes	CBP(fine)+degreasing	12hrs*	6hrs	6hrs	6hrs
KEK-12	SC	No	CBP(rough only)+degreasing	6hrs			
KEK-13	SC	No	CBP(fine)+degreasing	6hrs	6hrs	6hrs	6hrs
KEK-17	SC	Yes	CBP(fine)+degreasing	18hrs*	6hrs	6hrs	6hrs
KEK-18	SC	Yes	CBP(rough only)+degreasing	6hrs			
(*) renew stone each 6 hrs							

No.	Type	EBW	Treatment Details
Jlab-1	FG	No	Degreasing + UPW ultrasonic rinse + light BCP (~ 5 μm removal) + UPW ultrasonic rinse
Jlab-2	LG	No	Same as above
Jlab-3	SC	No	Same as above
Jlab-4	LG	Yes	Degreasing + UPW ultrasonic rinse + Iris EBW + Degreasing + UPW ultrasonic rinse+0.1μm grit grinding + Degreasing + UPW ultrasonic rinse
Jlab-5	FG	Yes	Same as above
Jlab-6	SC	Yes	Same as above
Jlab-7	LG	Yes	Degreasing + UPW ultrasonic rinse + Equator EBW + Degreasing + UPW ultrasonic rinse
Jlab-8	LG	Yes	Same as above
Jlab-9	LG	Yes	Same as above
Jlab-10	FG	Yes	Same as above
Jlab-11	FG	Yes	Same as above
Jlab-12	FG	Yes	Same as above

Surface Topographical Measurements

Optical microscopic images were taken from a HIROX KH-3000VD High Resolution Digital-Video Microscopy System**.** SP measurements were obtained with a stylus profilometer (KLA-Tencor: P-15) with a 2μm diameter tip. The samples were scanned in three different regions with a scan size of 1000μm×1000μm, and the 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 each scanned in five different regions with scan sizes of 50μm×50μm. The AFM images were captured as arrays of height values with 512x512 points. In order to eliminate measurement errors and increase the goodness of statistic 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 profilometry data were filtered using a Savitzky-Golay smoothing method in order to eliminate spurious high-frequency noise [8].

EXPERIMENTAL STUDIES AND DISCUSSIONS

Surface Topography-Optical Microscopy

All electron beam welded CBP Nb samples have shown localized defects which are clearly different from the typical surface character after EP. Some of them were not observable by optical microscopy before EP. The detailed mechanism for the appearance of those defects is under study. Figure 1a is an example of defect observed on a fine CBP EBW single crystal sample (KEK-17) before and after EP. The deepest depth of this defect after CBP is about 118μm. After 100 minutes EP (~30μm removal), the depth decreased to about 90μm, and the change of geometric shape was observed. The images shown in Figure 1a and 1b were captured from 2 and 3 frames before (Figure 1a) and after EP (Figure 1b) respectively

due to the limitation of spot size of Hirox microscope at 350X magnification. The depth of defect is estimated by the multi-focus function of the Hirox microscope. No such defects were observed from hand ground EBW Nb samples before or after EP.

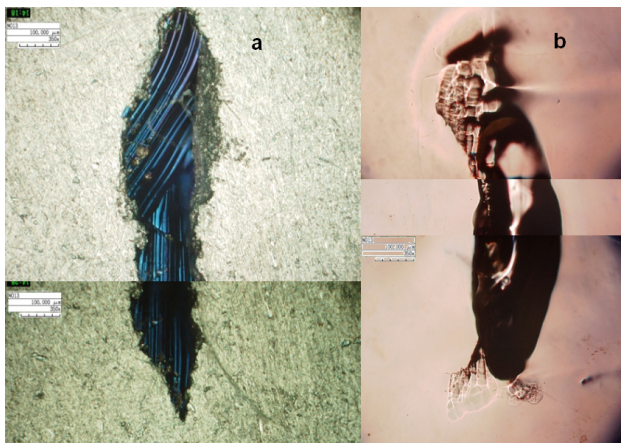

Figure 1: The optical microcopy image of a defect on the fine CBP EBW single crystal Nb sample (KEK-17) before and after EP.

Surface Topography-AFM and SP

The results of AFM and SP measurements for this set of 24 samples (as received and after EP) are summarized in the Figure 2. The data shows that the fine CBP provides a reproducible smooth surface as comparing with other treatments. After 100 minutes EP, the fine CBP surface achieves the best surface finishing.

Surface Topography –Power Spectral Density

Figure 3 shows the PSD of combined AFM and profilometry data from the sample (Jlab-1) with 5 minutes of BCP treatment at different scanning areas by SP and AFM. The results illustrate that the spatial frequency ranges corresponding to the different measurements largely overlapped [9]. It describes the characteristic isotropic roughness produced by applied processes at a lateral scale from few nm to few hundred µm, which provides a scale-dependent metric to directly view the effect of surface processing in terms of the feature that is understood to matter for performance**.**

Figure 4 is a PSD analysis of rough CBP, light CBP, hand grinding and light BCP fine grain Nb samples after 30µm removal by EP. The transformation of surface roughness after EP at a lateral scale from 1µm to 1mm shows that fine CBP produces the best surface finishing after a short duration EP.

The PSD analysis in Figure 5 shows that fine CBP produces a very reproducible surface for polycrystalline, large grain and single crystal Nb. However, after the 100 minutes EP at 30±1˚C, a noticeable difference was observed from the different crystalline Nb samples. Compared with fine grain Nb samples, the single crystal samples show the smallest amplitude of PSD and better correlation length. We explain the difference as perhaps

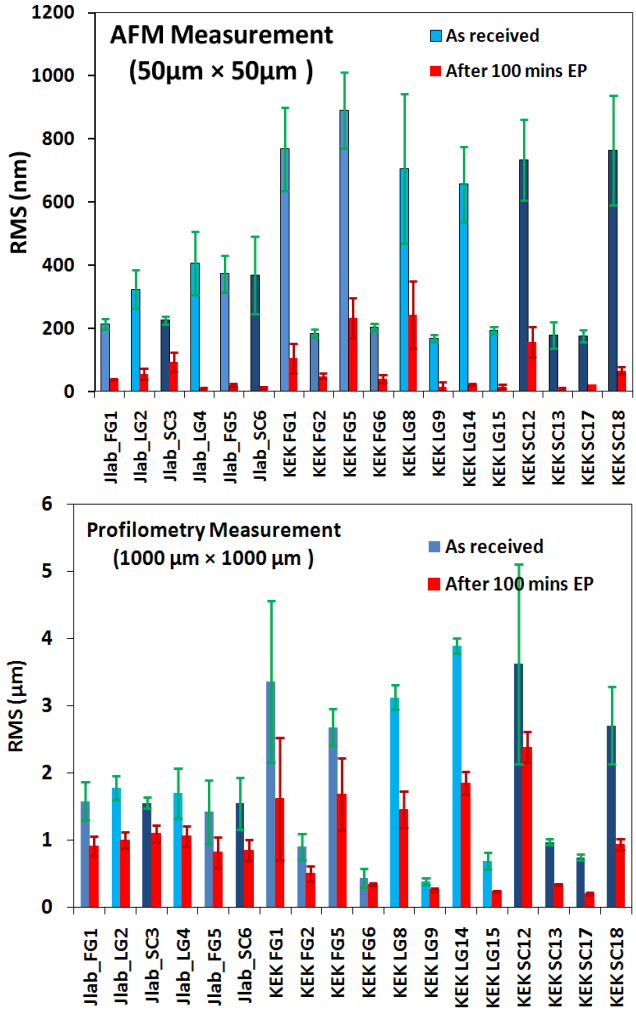

Figure 2: The statistical results of AFM (above) and SP measurements (below) for 24 samples studied before and after EP.

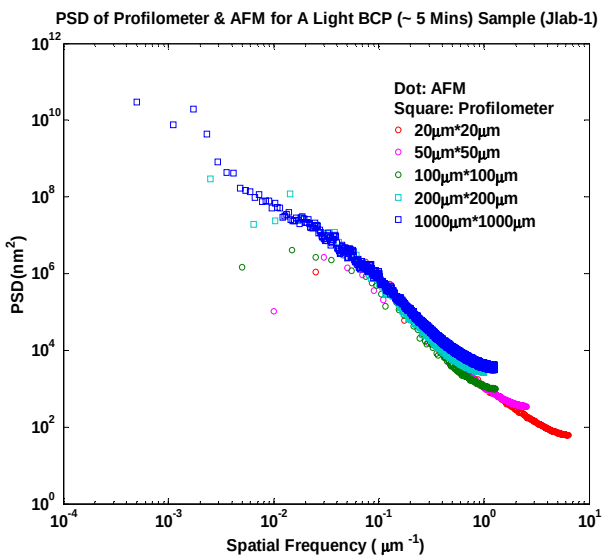

Figure 3: Combined AFM and profilometer power spectral densities from all scan sizes from the sample having undergone 5 minutes of BCP treatment.

caused by chemical etching occurring in parallel with the desired EP brightening process at the regulated 30˚C condition [10], however further studies are needed.

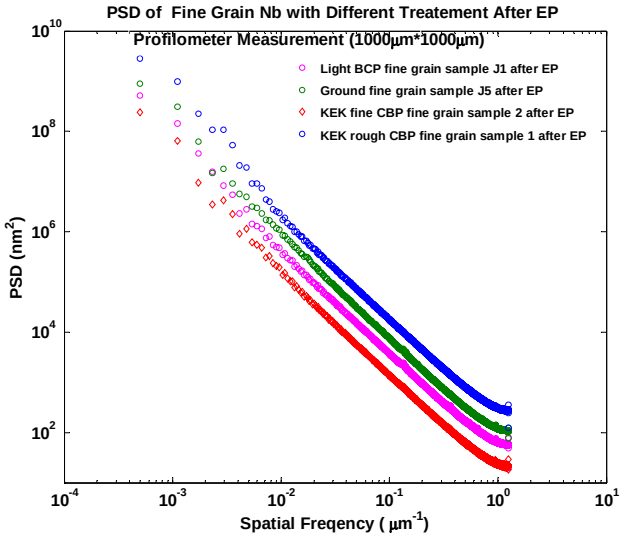

Figure 4: The PSD of rough CBP, fine CBP, hand grinding and light BCP treated polycrystalline Nb sample after 30μm removal by EP at 30±1˚C.

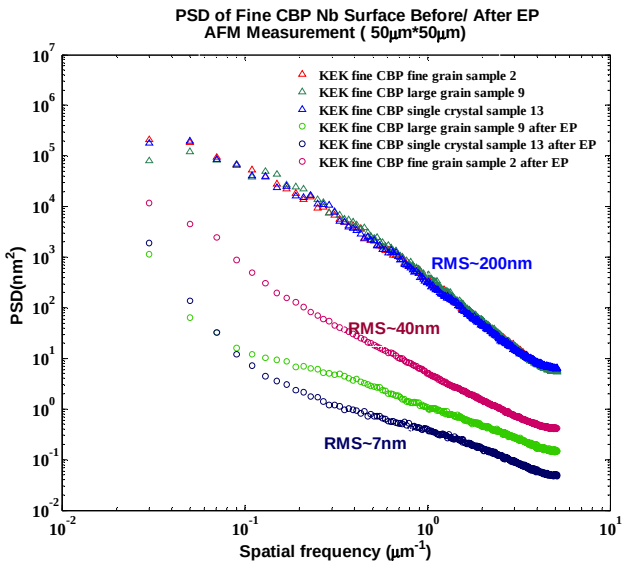

Figure 5: The PSD of fine CBP polycrystalline, large gain and single crystal Nb samples after 30 μm removal by EP at 30±1˚C.

CONCLUSION

In this study, the topographical transformation of 24 Nb samples with different starting processing conditions has been systematically characterized by optical microscopy, AFM, SP and analyzed by PSD before and after a well-controlled EP. Some localized defects produced by particular conditions on electron beam welded samples treated by CBP have been observed by optical microscopy. Such defects were not removed by 30um EP. The characteristic isotropic roughness produced by the applied processes, such as CBP, light BCP, hand grinding and EP, for all samples was analyzed by PSD, together with statistical measurements of AFM and SP. The results show that the fine CBP may provide a reproducible starting surface and could be transformed to a nano-smooth surface finishing by only a 30μm removal by EP. These studies provide useful data for the optimization of the EP process which can be applied to the processing of single and multi-cell cavities.

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