DETAILED NB SURFACE MORPHOLOGY EVOLUTION DURING ELECTROPOLISHING FOR SRF CAVITY PRODUCTION *

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Abstract

Electropolishing of niobium is currently an important part of attaining the best performance of SRF cavities. We endeavour to develop sufficient understanding of the process dynamics to gain predictive power over specific topographies subjected to controlled electropolishing conditions. This work examines the evolution of centrifugal barrel polished fine-grain Nb samples as this material is electropolished under different temperatures. The morphology evolution of Nb surface has been described using a combined approach of scaling analysis and predictions of the electropolishing theory. Our results suggest that centrifugal barrel polishing leaves highly strained topmost surface structure, the surface morphology evolution during following electropolishing could not be well described by a self-affine fractal structure until the plastic deformed layer is removed. The present analysis suggests electropolishing at low temperature helps to reduce the kinetics-controlled surface etching. A preliminary computational model has been developed that simulates the evolution of specific topography of a niobium surface under parameterized conditions. This work is expected to lead to the direct linking of starting surface morphology specification, specific processing protocol, and consistently attained finished surface condition.

INTRODUCTION

Electropolishing of niobium in the hydrofluoric and sulphuric acid mixers has been used for polishing Nb cavities for decades, and it has been develop empirically through cavity process practices. Recent R&D studies revealed that the standard electropolishing of niobium proceeds via anodization of niobium by the sulphuric acid moderated by the dissolution of the anodized layer (Nb_2O_5) via the available fluoride ions [1-2], higher temperature provokes another oxide dissolution process, which may be described more as etching rather than polishing[3]. Because electropolishing is becoming an important part of attaining the best performance of SRF cavities, the correlation between parameters of electropolishing process and the resulting surface smoothening needs to be established.

The scaling analysis has been used to describe the kinetic roughening of surface during the non-equilibrium deposition and erosion. In most of studies of surface morphology evolution, the observed systems were found to obey normal and anomalous scaling law [4-7]. To apply the scaling law to electropolishing, the surface morphology evolution could be carried out through the simulation using algorithms for diffusion limited model (DLA) [5]. It predicts that the corrugations of surface would diminish exponentially as a function of time, independent on the length scale of observation, and it is supported by the fundamental concept and mathematical description of an ideal electropolishing process [6].

The focus of this paper is to investigate the evolution of the Nb surface morphology during the electropolishing process. The data are analyzed using scope of both scaling analysis and predictions of the electropolishing theory. The approach has been successfully used to simulate Cu surface evolution during electropolishing [7]. A preliminary computational model has been developed that simulates the evolution of specific topography of a niobium surface under parameterized conditions.

EXPERIMENTAL

The starting Nb surfaces were produced by a series centrifugal barrel polishing under different durations at KEK. Previous statistical studies suggested that this fine centrifugal barrel polishing (CBP) treatment provided a highly reproducible starting surface [8]. Atomic Force Microscopy (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 more than five different positions with the scan size of 50 µm × 50 µm. The EP solution was a 1:10 mixture of HF (49%) and H_2SO_4 (96%). For electropolishing, two Nb disk samples were mounted to a customized sample holder subjected different durations EP at 10 volts simultaneously. The bulk electrolyte temperature was controlled by a circulated water bath at 20±1˚C and 34±1˚C respectively, the removal of Nb surface is kept the same for different temperatures by different durations EP.

The AFM images were analyzed using custom image processing software based on Matlab programming language platform. This characterization software is capable of processing AFM images of a given size represented as two dimensional matrix of the data containing the information of the surface topography as a normal distance (h) with respect to the average plain of the image. The size of the observation window in the software has been designed to be an input parameter with subsequent step of the observation window enlargement. The surface roughness ($w(l,t)$) defined as [9]:

$$w(l,t) = \sqrt{< \left(h(r,t) - \bar{h}(r,t)\right)^2 >},$$

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is obtained from this analysis as a function of the size of the observation window for a given image ($w = f(l)$). The output is functional dependence of roughness as a function of the length scale for a given surface. The second part of the software is design to execute two different scaling functions to fit the obtained w *vs.* l data. From these fitting procedures, the scaling parameters of the surface are extracted representing the characteristics of a given AFM image. The fitting function represents the normal scaling function defined as [10]

$$w(l) \sim l^{\alpha} \text{ for } l < l_c \qquad (1)$$

and

$$w(l) = w = const \text{ for } l \geq l_c \qquad (2)$$

Here, the main output of the fit are the scaling parameters of the surface defined as: saturation roughness, W_{sat}, roughness exponent α, and critical length, l_C.

RESULTS AND DISCUSSIONS

Figure 1 shows the surface morphology evolution of the fine CBP polycrystalline Nb surface with electropolishing at 20˚C and 34˚C respectively.

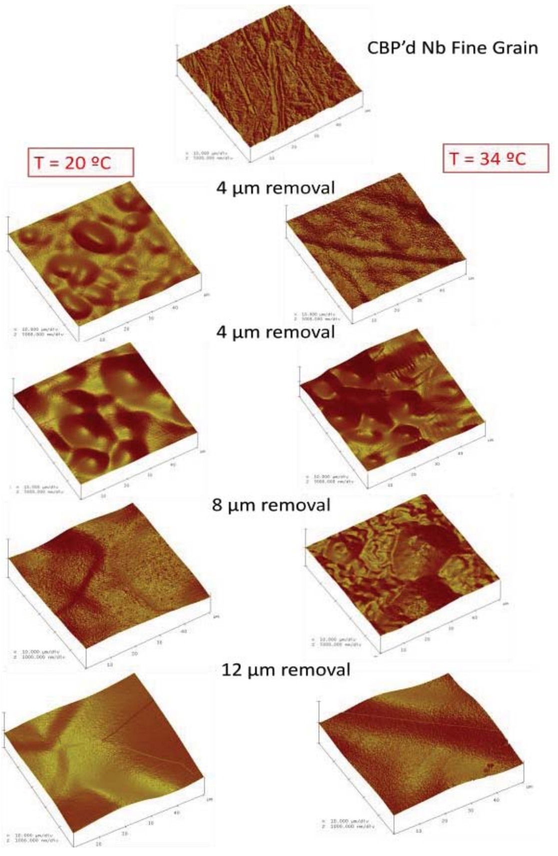

Figure 1: The surface morphology evolution of the fine CBP polycrystalline Nb surface with electropolishing at 20˚C and 34˚C respectively. The AFM was done at 50 µm × 50 µm scan area. The vertical scale of AFM is 5 µm/div for first 8 µm removals for both temperatures; 1 µm/div for 20˚C and 5 µm/div for 34˚C for the second 8 µm removals respectively; 1µm/div for last 12µm removals for both temperatures.

From these AFM images, we learn that the early stages of EP seem to roughen the fine CBP'ed surface instead of smoothing out the mechanic grooves left by the media used in the CBP. This suggests that the centrifugal barrel polishing may leave heavily strained surface structures and short duration of EP may help to reveal the underneath sub-grains [11]. A lower temperature of EP produces a better surface finish.

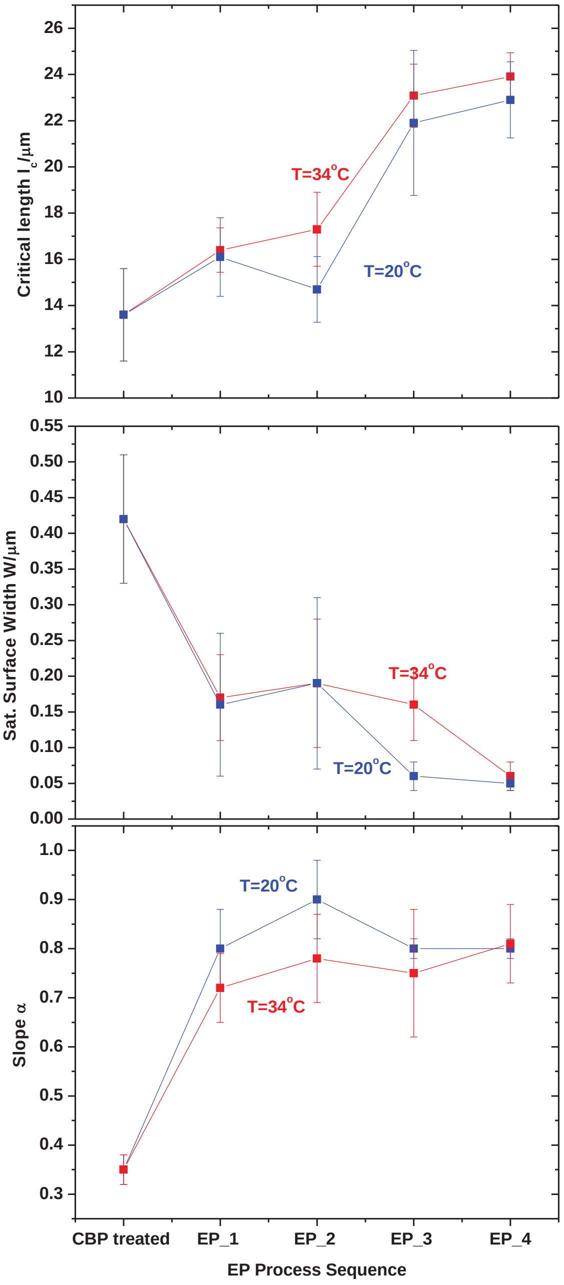

Figure 2: The critical length l_C (Fig. 2-1), saturation roughness W_{sat} (Fig 2-2), and roughness exponent α (Fig. 2-3) were derived from the fitting with different steps electropolishing. The removals are kept same for different temperatures during different EP durations: EP_1~ 4 µm; EP_2~ 4 µm; EP_3~ 8 µm; EP_4~ 12µm.

The saturation roughness W_{sat}, roughness exponent α, and critical length l_C were derived from the fitting with different steps electropolishing. We noticed that the value of critical length varies significantly from the value measured from the initial CBP surface with early stages EP, but it expresses less change after about 16μm removal by EP, see Figure 2-1. It suggests that the CBP'ed surface could not maintain the self affine fractal geometry of fine grain Nb which describes individual grain structure typically, due to plastic strain on the outmost layer. Until the plastically deformed layer is removed, the electropolishing would proceed with the virgin structure of niobium. The fitted results show the reduction of saturation roughness W_{sat} , which suggests EP smoothes out the large protrusions which fall in the scale of the diffusion layer, see Figure 2-2. However the increase of roughness exponent α with first few μm removals (~8 μm) indicates this short duration of EP exposes the rough sub-structures, instead of diminishing the corrugations of the surface, as shown in Figure 2-3.

The use of the normal and anomalous scaling law in order to evaluate the growth exponents is precluded because the surface width as a function of time is not described by the power law. Instead, this functionality is well described by the exponential decay predicted theoretically and in simulations involving DLA. In this study, we interpret the surface morphology quantified by the surface width with an analogy to the corresponding sine wave profiles [5]. Therefore, the rough surface is represented by a sine wave with amplitude $\sqrt{2}w$, where w is the value of the surface width measured experimentally.

The exponential decay for the surface corrugations of the Nb surface during the electropolishing is described as:

$$w(t) = w_0 \exp\left(-\varphi \frac{t}{\lambda}\right) \quad (3)$$

In this expression, the factor $\varphi = {}^{\pi j M_{Nb}}/_{F \rho_{Nb}}$ is a constant, could be estimated from electropolishing conditions. The term $j, M_{Nb}, F\ and\ \rho_{Nb}$ represent the polishing current density (mA/cm^2), molar mass of Nb (g mol^{-1}), Faraday's constant (C mol^{-1}), and the density of Nb (g cm^{-3}). The assumption is made that anodic current efficiency of an electropolishing process to be 100%. For electropolishing of niobium process Nb^{5+} is the only form of dissolved Nb [1~2].

The simulation of surface morphology evolution is done based on both scaling analysis and predictions of the electropolishing theory. Figure 3 present an example of the surface evolution during electropolishing for a CBP'ed Nb surface with 8 μm removal at 20°C. We noticed that the values of roughness exponent decreased linear with the polishing time. Mathematically it could be described by a linearly decreasing function of time having the form:

$$\alpha(t) = \alpha_0 - Bt \quad (4)$$

The critical length is determined through this simulation and it is about 12.5 μm. The surface width was derived from fitting and it decreases with polishing time under different length scale. From present analysis, we noticed that value of B depends on the electropolishing temperature, it increases with low temperature. This suggests that EP at the lower temperature reduces the kinetics-controlled surface etching, but further analysis is needed.

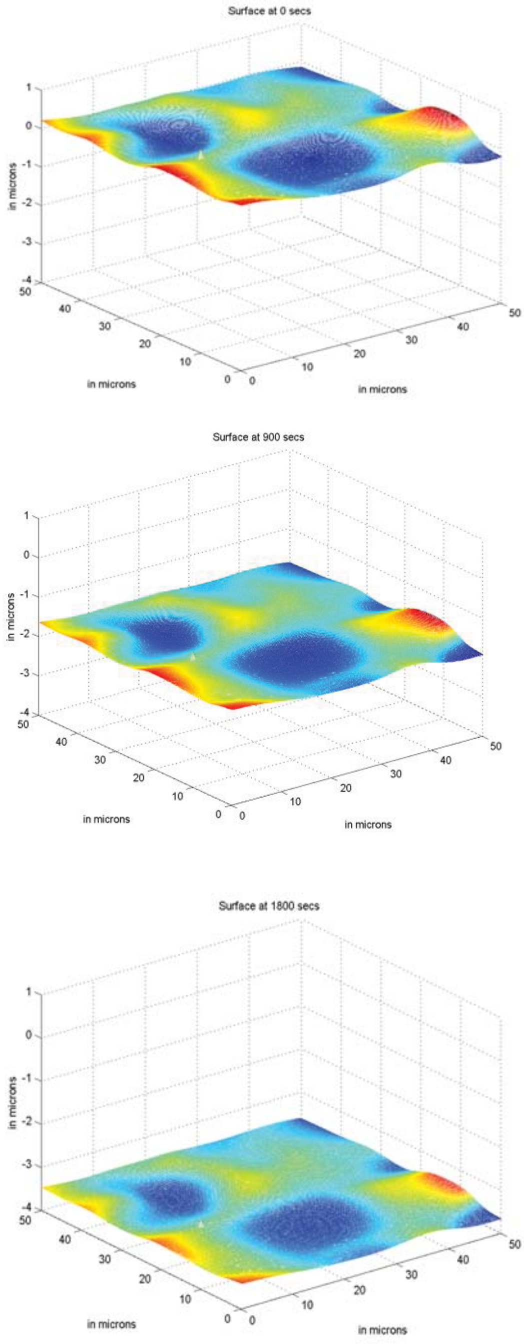

Figure 3: The simulation of CBP'ed Nb surface evolution after 8 μm removal by EP during electropolishing at 20°C.

CONCLUSION

In this study, the morphology evolution of a CBP'ed polycrystalline Nb surface during electropolishing is described by a combined approach of scaling analysis and predictions of electropolishing theory. A preliminary computational model has been developed that simulates the evolution of specific topography of a niobium surface under parameterized conditions. This work is expected to lead to the direct linking of starting surface morphology specification, specific processing protocol, and consistently attained finished surface condition.

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