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GENESIS OF TOPOGRAPHY IN BUFFERED CHEMICAL POLISHING OF NIOBIUM FOR APPLICATION TO SUPERCONDUCTING RADIOFREQUENCY ACCELERATOR CAVITIES*

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Abstract

Topography arising from the final etch step in preparing niobium superconducting radiofrequency (SRF) accelerator cavities is understood to significantly impact cavity performance at high field levels. This study investigated the effect of process temperature and time on the etch rate and topography arising from the widely-used buffered chemical polishing (BCP). This study aims to understand more thoroughly the genesis of topography in BCP of polycrystalline niobium, with the ultimate aim of finding a path to surface smoothness comparable to that obtained by electro-polishing (EP). It was found that the etch process is controlled by the surface reaction; and that the etch rate varies with crystallographic orientation. The familiar micron-scale roughening necessarily results. Gas evolution has an impact, but is secondary. The major outcome is that surface smoothness comparable to EP appears to be inherently unachievable for polycrystalline niobium using BCP, setting an upper limit to the gradient for which it is useful.

BACKGROUND

BCP is still an essential step in SRF cavity fabrication, from welding surface preparation, to bulk removal, to final surface "flash" polishing, despite the rapid development of alternative polishing methods. The attractive advantage of BCP lies in its simple setup and fast process. The disadvantage of BCP, however, is that the "polished" niobium surface displays a characteristic roughness. Instead of discarding BCP due to this roughness, it is more efficient to use it wisely where suitable [1-2], which will eventually optimize the cavity fabrication process. To achieve that, it is necessary to understand the forming mechanism of this characteristic topography. This study provides evidence to support that crystal orientation dependent etching plays a major role in the formation of BCP niobium topography. It adds to existing understanding about the niobium BCP reaction and resulting surfaces [3-6].

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EXPERIMENT

Material and Preparation

The polycrystalline (fine grain) niobium samples were electric discharge machined (EDM) from high RRR sheet material used for cavity fabrication. The bi-crystal and single crystal samples were cut from a large grain sheet. The sample dimensions were all 10 mm × 10 mm × 3.2 mm. After EDM, the samples were etched in 1:1:2 BCP solution for 1 min to remove machining contaminants. They were then rinsed with de-ionized water and air dried.

For polishing rates and topography study, various polishing conditions were performed and they are summarized in Table 1.

Table 1: Niobium Sample Types and Polishing Conditions

Sample type	Temperature	Duration	Orientation
Fine grain	0-30°C	1-20minutes	Face up, down, and sideway
Bi-crystal	Room Temperature (20~22°C)	12minutes	Face up
Single crystal	Room Temperature (20~22°C)	Accumulated 90minutes	Face up

Polishing rates were determined using polycrystalline samples. Samples were immersed in fresh 1:1:2 BCP solution for designated duration from 1 to 10 minutes, taken out of solution, rinsed with de-ionized water, and air dried. No stirring was applied during polishing. Polishing rates were calculated from the weight difference before and after polishing divided by polishing time and polished surface area.

Characterization

The topography of the niobium samples was examined by scanning electron microscopy (SEM), optical microscopy, and atomic force microscopy (AFM). The crystal orientation of the niobium samples was examined by electron backscatter diffraction (EBSD).

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RESULTS AND DISCUSSION

Figure 1 shows the polishing rates of fine grain samples at temperatures from 0°C to 30°C, for durations from 1-10 minutes. Each data point is the average of three measurements. The polishing rate started highest at 1 minute and dropped gradually to a steadier rate. Plotting the natural logarithm of polishing rate against inverse temperature (polishing rates at 6 minutes were used here), gives an activation energy and temperature constants that suggest the BCP reaction is a chemical reaction controlled process rather than a diffusion controlled process within the 0-20°C range.

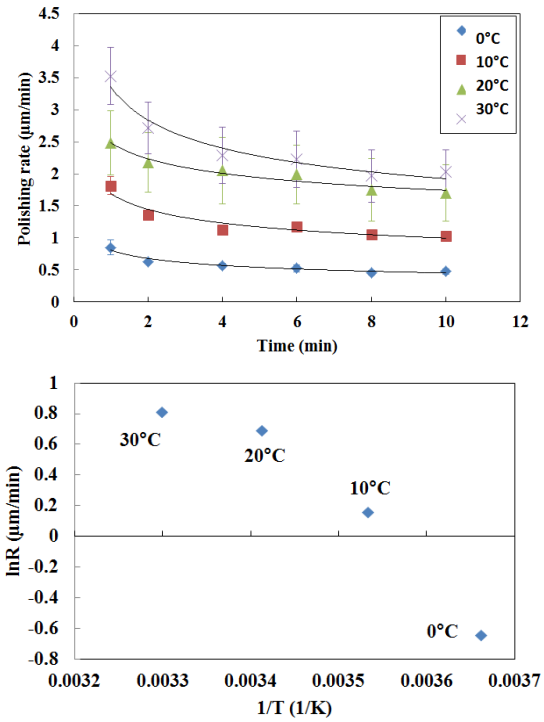

Figure 1: Nb BCP polishing rate as a function of time at 0-30°C. The natural logarithm of polishing rate as a function of inverse temperature (polishing rates at 6 minutes were used here).

Figure 2 is the SEM images of fine grain niobium polished at 10°C and 22°C. The BCP duration varied from 1 to 20 minutes. The secondary electron (SE) images shows better topography information, while the backscattered electron (BSE) images provides better picture of crystal grains. Along the polishing duration sequence, the small pitting features faded, and the topography developed towards more similar to that of the crystal grains. This process happens faster at 22°C than at 10°C.

Figure 3 shows the SEM images of fine grain niobium polished at room temperature (about 20°C) for 6 minutes, about 15 μm removal. The surfaces to be polished were oriented in three directions during BCP: face up, face down, and face horizontally (side way). Note that these samples were mechanically polished to mirror finish, which helped better reveal the effect of crystal grains

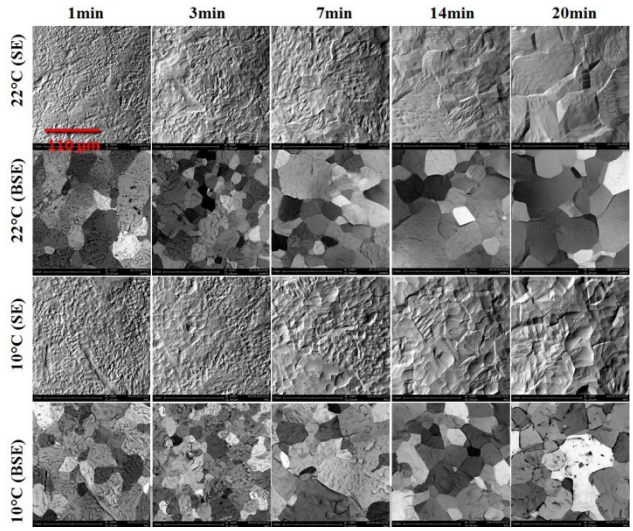

Figure 2: SEM images of fine grain niobium polished for 1-20 minutes, at 10°C and 22°C. For each sample, the upper image is from secondary electron and the lower image is from backscattered electron.

on topography after BCP. Facing down and facing horizontally showed a polishing rate of 3 μm/min, twice of the rate for facing up, which was 1.5 μm/min. The removal rate difference due to orientation is also observed during cavity BCP; therefore flipping of cavity is performed to achieve even removal. More bubble burst prints were observed on samples that were facing down.

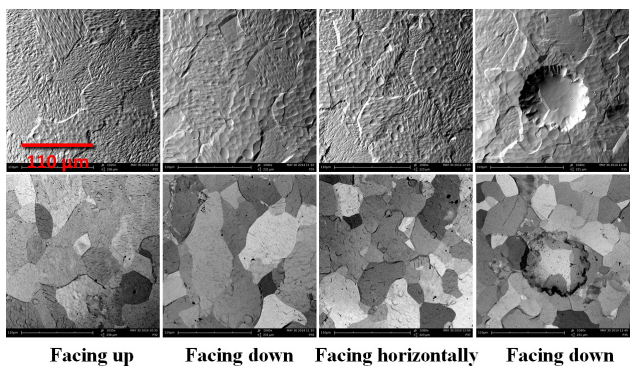

Figure 3: SEM images of fine grain niobium polished for 6 minutes at room temperature. For each sample, the upper image is from secondary electron and the lower image is from backscattered electron. The orientation of the sample was: facing up, facing down, facing horizontally, and facing down in the order of the images.

Combining EBSD with the profile of the surface, the correlation of height variance after BCP and crystal orientation was established. Figure 4 shows the SEM images of four single crystal niobium samples that accumulated 90 minutes BCP at 22°C, equivalent to 225 μm removal. After such a heavy BCP, fine grain niobium would develop a very rough surface. But the single crystal samples still showed rather smooth surface finish. Figure 5 shows the optical image of a bi-crystal niobium after 12 minutes BCP at 20°C, equivalent to 30 μm removal. AFM profil-

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ing showed a 0.68 µm step at the border between the two grains. The surface within each grain remained smooth. Figure 6 shows the SEM image of a fine grain niobium sample after 6 minutes BCP at room temperature, about 15 µm removal. EBSD of the center grid tells the crystal orientation of each grain within this grid. The same grid was examined with optical microscopy, and 3D surface profile was used to determine height difference between (100), (101), and (111) surfaces. Similar process was performed at several other locations and a statistics of height sequence was found. The order from highest to lowest after 15 µm removal is: (111), (001), (101). That means (101) removal is the fastest among the three and (111) the slowest. Height difference between (111) and (101) is 2±0.7 µm.

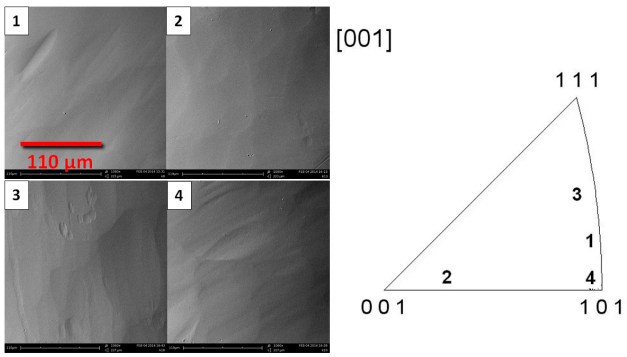

Figure 4: SEM images of four single crystal niobium samples received accumulated 90 minutes of BCP at 22°C. The orientation of the four single crystals is marked in the inverse pole figure.

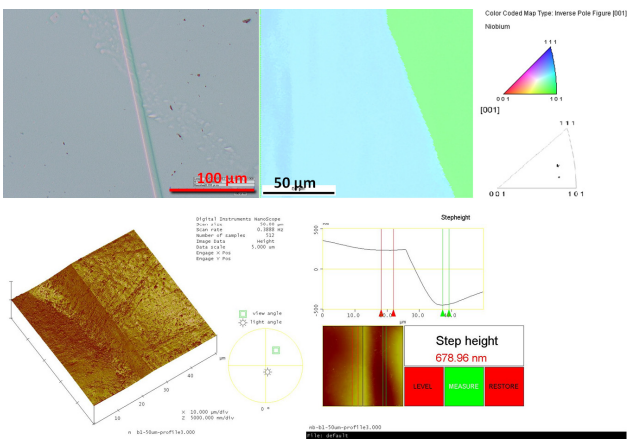

Figure 5: Optical image of a bi-crystal niobium sample after 12 minutes of BCP at 20°C. EBSD and AFM scan on the bi-crystal shows the orientation of the two grain is close to each other and the step at the grain boundary is about 0.68 µm.

Gas evolution on the polishing surface is a unique character of BCP compared with other polishing methods. Features related to bubbles are found on single crystal and bi-crystal samples, even when the sample was facing up during BCP. Bubble prints are less obvious on fine grain samples due to the dominating feature resulted from differential etching of different crystal orientations. Typical bubble prints have diameters from 50 to 100 µm and depths of about 1 µm, as shown in Fig. 7.

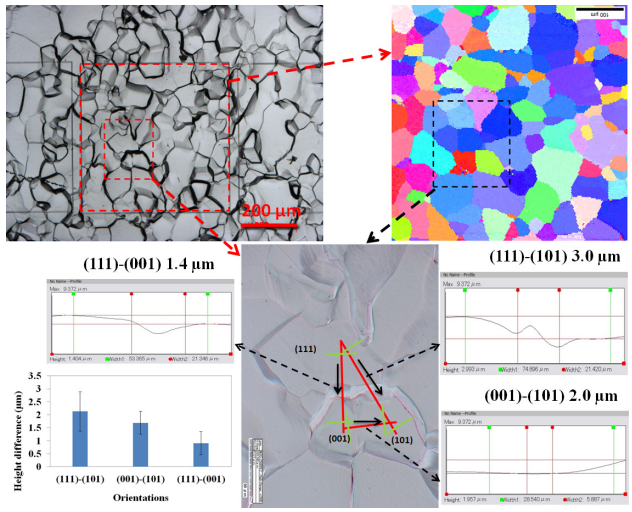

Figure 6: SEM image of fine grain niobium received 6 minutes of BCP at room temperature. EBSD corresponds to a 500 µm × 500 µm square are at the center of the SEM image. Height difference between the (111), (100), and (101) grains at the center was measured by optical microscope 3D profile function.

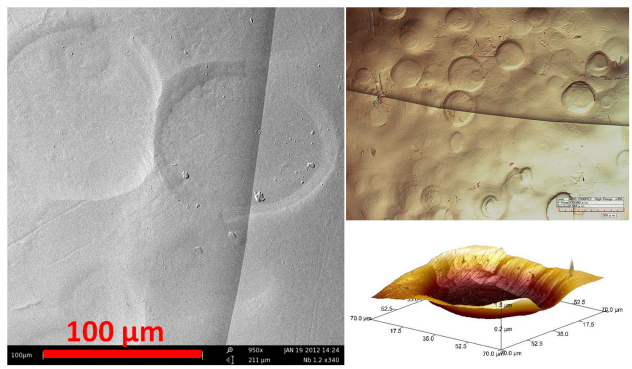

Figure 7: SEM, optical microscope, and AFM image of bubble prints on bi-crystal niobium received 80 µm removal at room temperature.

CONCLUSION

- Under static conditions, the topography of BCP'd fine grain niobium is primarily determined by grain orientation dependent differential etching. Step size at grain boundaries depends on the crystal orientation of the two grains. Bubble print from gas evolution is a secondary contributor.
- Polishing rate is temperature and sample orientation dependent.
- Topography is decided by total removal, which is a combined result of time, temperature and orientation.

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