ISBN: 978-3-95450-211-0 doi:10.18429/JACoW-SRF2019-MOP013

REDUCING SURFACE ROUGHNESS OF Nb_3Sn THROUGH CHEMICAL POLISHING TREATMENTS*

H. Hu†, M. Liepe, R. D. Porter
Cornell Laboratory for Accelerator-Based Sciences and Education (CLASSE),
Ithaca, NY 14853, USA

Abstract

Niobium-3 tin (Nb_3Sn) is a promising alternative material for SRF cavities, with theoretical limits for critical temperatures and superheating fields reaching twice that of conventional Nb cavities. However, currently achievable accelerating gradients in Nb_3Sn cavities are much lower than their theoretical limit. One limitation to the maximum accelerating gradient is surface magnetic field enhancement caused by the surface roughness of Nb_3Sn. However, there are currently no standard techniques used to reduce Nb_3Sn surface roughness. Since Nb_3Sn is only 2-3 microns thick, it is difficult to selectively polish Nb_3Sn without removing the entire layer. Here, we investigate reducing the surface roughness of Nb_3Sn through applying chemical polishing treatments, including modified versions of standard techniques such as Buffered Chemical Polishing (BCP) and Electropolishing (EP). Through data acquired from Atomic Force Microscope (AFM) scans, SEM scans, and SEM-EDS analysis, we show the effects of these chemical treatments in reducing surface roughness and consider the changes in the chemical composition of Nb_3Sn that may occur through the etching process. We find that BCP with a 1:1:8 solution is ineffective while EP halves the surface roughness of Nb_3Sn.

INTRODUCTION

Niobium-3 tin cavities have been shown to reach high quality factors on the order of 10^{10}, enabling cavities to achieve accelerating gradients of over 16 MV/m at 4.2 K [1–3]. Abrupt bumps in the surface result in significant magnetic field enhancements. Thus, surface roughness of Nb_3Sn limits the current maximum achievable accelerating gradients. While there are standard techniques to treat Niobium cavities, there are currently no standard techniques to reduce the surface roughness of Nb_3Sn. This is due to the fact that Nb_3Sn cavities only contain a thin layer of Nb_3Sn coated on the surface through tin diffusion that is 2-3 microns thick. Polishing techniques that require removal of large quantities of material would strip away the Nb_3Sn that has been coated on. We consider the effects of using modified versions of standard techniques of Buffered Chemical Polishing (BCP) and Electropolishing (EP) to polish Nb_3Sn.

Standard BCP treatments involve a 1:1:1 or 1:1:2 solution consisting of HF (49%), HNO_3 (65%), and H_3PO_4 (85%). Prior testing at Cornell has demonstrated that BCP using a 1:1:2 solution strips away material from the surface faster than can be controlled [4]. The initial minute of BCP rapidly attacks the surface and destroys the Nb_3Sn layer [5]. In this paper, we test a modified treatment of BCP polishing using a 1:1:8 solution that is more controllable.

EP treatments use a 1:9 volume ratio of HF (48%) to H_2SO_4(96%) solution. For Niobium cavities, EP is used to create a smoother finish on the surface [6]. Initial testing has not yielded significant evidence of surface roughness reduction through EP treatments for Nb_3Sn [4]. This paper focuses on the effects of BCP and EP treatments on the surface roughness of Nb_3Sn. Through height data obtained with an Atomic Force Microscope (AFM), we conduct Amplitude Spectral Density calculations to characterize the changes in surface roughness as a result of these treatments.

METHOD

Sample Preparation

1 cm x 1 cm samples of bulk niobium are first cleaned using BCP and anodized at 30 V to create an oxide of approximately 100 µm on the surface. Following the Standard Coating procedure used at Cornell, the sample is then coated with tin in an ultra-high vacuum furnace, forming a 2-3 µm thick layer of Nb_3Sn on the surface of the sample [7]. The samples were then polished using either BCP with a 1:1:8 solution of HF, HNO_3 and H_3PO_4 or EP. The 1:1:8 BCP samples etched for 75 s and 150 s and removed approximately 1 µm and 2 µm respectively.

For the sample treated with EP, we first mapped out an I-V curve to determine the region that corresponds to polishing. Figure 1 shows the I-V curve of the EP treatment, including the etching, polishing and oscillating regions. The polishing region of the I-V curve occurs between 1.3 V and 2.2 V. The etching rate of EP was determined through dimensional analysis of the oxidation and reduction reactions occurring at the cathodes and anodes for both niobium and tin [8]. Taking into account the density of Nb_3Sn, we were able to estimate that Nb_3Sn etches at a rate of 13.5 nm/min per mA/cm^3. Approximately 1.43 µm of Nb_3Sn was removed from the surface during this process.

Sample Analysis

After each of the treatments, the samples were scanned using an AFM at both 20 µm x 20 µm and 90 µm x 90 µm scan sizes at 3 random locations for each size. The height maps obtained were used to characterize the roughness of each sample through an Amplitude Spectral Density (ASD)

* This work was supported by the U.S. National Science Foundation under Award PHY-1549132, the Center for Bright Beams. This work made use of the Cornell Center for Materials Research Shared Facilities which are supported through the NSF MRSEC program (DMR-1719875).
† hh539@cornell.edu

ISBN: 978-3-95450-211-0 doi:10.18429/JACoW-SRF2019-MOP013

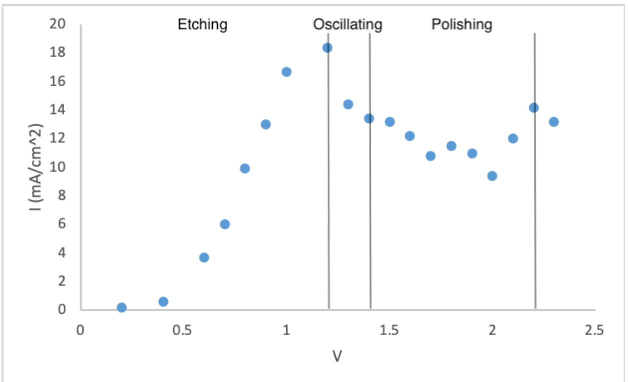

Figure 1: I-V Curve for electropolishing.

approach, a process which takes a Fast Fourier Transformation of the height data at each spatial frequency [4]. The ASD data from scans from the same sample were then averaged and plotted along with the sample standard deviation for each of the samples.

Finally, the samples were SEM scanned and EDS-SEM analysis was performed on the surface to determine Sn content. The EDS-SEM analysis was conducted using 15 keV accelerating voltage in order to determine Sn concentration.

RESULTS AND DISCUSSION

AFM Analysis and Surface Roughness

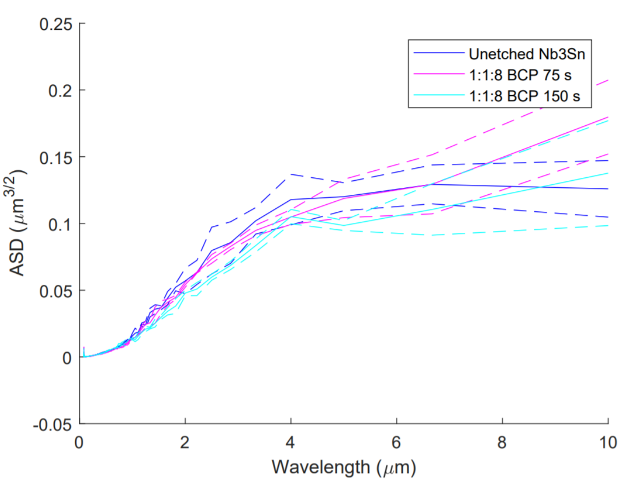

Figure 2: ASD plots for BCP treated samples.

Buffered Chemical Polishing has very minimal effects on reducing the surface roughness of Nb_3Sn. As shown in Fig. 2 the sample that etched for 150 s showed slight improvements in roughness. However, the effects are not statistically significant enough to indicate that a 1:1:8 solution of BCP etched for 150 s is an effective treatment for reducing the surface roughness of Nb_3Sn.

Electropolishing shows a not statistically insignificant effect on reducing the surface roughness of Nb_3Sn . The roughness appears to be halved at particular frequency ranges (see Fig. 3). The apparent reduction in roughness, however, may

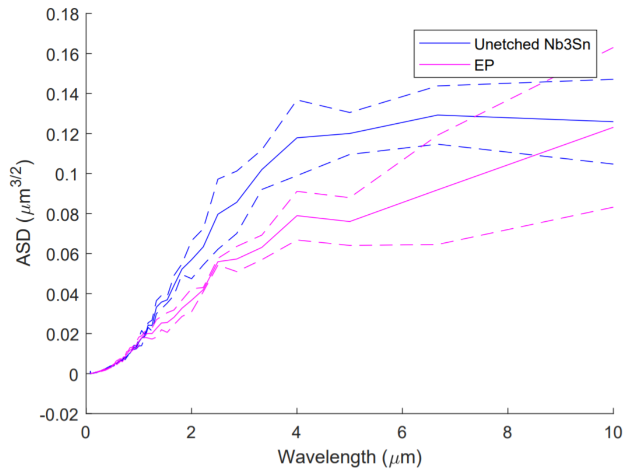

Figure 3: ASD plots for EP treated samples.

be due to noise from the scans. Nonetheless, the effect of EP on Nb_3Sn is promising and could potentially be improved on to reduce the surface roughness of Nb_3Sn from a factor of two to a factor of four [9].

SEM and SEM-EDS Analysis

SEM scans of the electropolished samples are shown in Fig. 4. Nb_3Sn grain structure is still visible suggesting that the Nb_3Sn layer was not completely removed. The grain boundaries are less sharp compared to the unetched Nb_3Sn as would be expected for polishing. In the zoomed-out SEM scan we can see surface inhomogeneity. “White” regions are visible in the 100 µm SEM scan. These may be thicker regions of Nb_3Sn that did not etch and have increased roughness (discussed below).

EDS acquires a Sn to Nb ratio of 4 at % Sn in the dark regions and 23.5 at % Sn in the light regions. The low Sn regions could in part be explained by the penetration of the e-beam and changes in layer thickness. In thin regions the beam is penetrating through Nb_3Sn layer and detecting the bulk Nb, decreasing the relative Sn to Nb signal ratio. For a 15 keV electron beam the penetration is ≈ 1 µm in Nb_3Sn, so layers thinner than this will begin to show lower Sn to Nb ratios. Nb_3Sn layers grown at Cornell are typically 2 – 3 µm thick, so with 1.2 µm of removal it is possible that this could occur. However, 4 at % Sn requires a thinner Nb_3Sn layer (≈ 200 nm) than we expect. It is possible that more than 1.2 µm were removed, Sn is being preferentially removed, or the film is unusually thin. EDS of cross-sections is the easiest way of determining this, but they have not been done at this time. The Sn concentration is the light region is consistent with regular Cornell Nb_3Sn coatings.

SEM scans of the BCP samples are shown in Fig. 5. Almost all of the Nb_3Sn layer had been removed. The 1 min 15 s BCP 1:1:8 etch appears to still show Nb3Sn grain boundaries, suggesting the Nb_3Sn layer has not been completely removed. Curiously, there appears to be a small structure in the middle of many of the grains. In the 2 min 30 s etch the structures in the grain are more visible.

ISBN: 978-3-95450-211-0 doi:10.18429/JACoW-SRF2019-MOP013

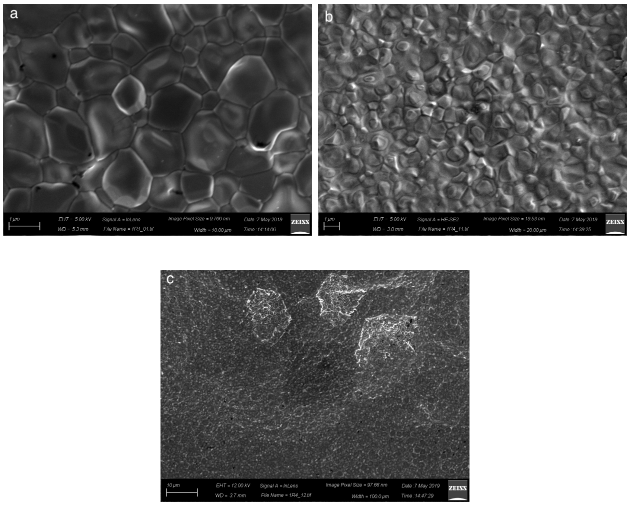

Figure 4: SEM scans of Nb_3Sn samples showing changes from EP treatment for (a) untreated surface, (b) ≈ 1.2 µm of EP, and (c) ≈ 1.2 µm of EP, zoomed out.

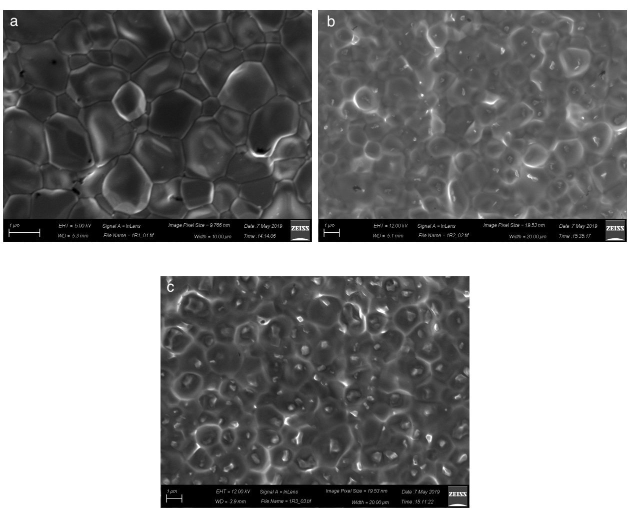

Figure 5: SEM scans of Nb_3Sn samples showing changes from BCP treatment for (a) untreated surface, (b) 75 s 1:1:8 BCP, and (c) 150 s 1:1:8 BCP.

EDS analysis of the BCP sample found the 75 s etch to have stoichiometric Nb_3Sn, but the 150 s etch to have decreased Sn concentration. Similar to the EP results, we suspect this means the 150 s etch has removed most of the Nb_3Sn layer. Since most of the layer has been removed without reducing surface roughness it is likely that increasing the etching time will only remove the Nb_3Sn layer. This implies the BCP solution needs further modification in order to polish Nb_3Sn.

CONCLUSION

Buffered Chemical Polishing with a 1:1:8 volume ratio solution is not a useful polishing technique for reducing the surface roughness of Nb_3Sn. The BCP recipe did not reduce surface roughness even when removing most of the Nb_3Sn layer. Our tests with Electropolishing indicates a reduction in surface roughness by almost a factor of two in the relevant length scale for surface magnetic field enhancement. This makes EP promising for continued development. Further testing with EP is needed to affirm the effectiveness of this treatment, improve polishing uniformity, and improve surface roughness reduction. Further work may take into consideration other polishing treatments such as Bipolar EP, Buffered EP, and Laserpolishing.

REFERENCES

[1] S. Posen, D. L. Hall, M. Liepe, and Th. Proslier, "High Q0 at Medium Fields in Nb3Sn SRF Cavities at 4.2 K", in *Proc. IPAC'15*, Richmond, VA, USA, May 2015, pp. 3467–3470. doi:10.18429/JACoW-IPAC2015-WEPTY078

[2] S. Posen, M. Liepe, and D. Hall, "Proof-of-principle demonstration of Nb3Sn superconducting RF cavities for high Q0 applications", *Applied Physics Letters*, vol. 106, Issue 8, p. 082601, 2015.

[3] S. Posen, M. Liepe, "Advances in development of Nb3Sn superconducting radio-frequency cavities", *Phys. Rev. ST Accel. Beams*, vol. 17, p. 112001, 2014.

[4] R. D. Porter, F. Furuta, D. L. Hall, M. Liepe, and J. T. Maniscalco, "Effectiveness of Chemical Treatments for Reducing the Surface Roughness of Nb3Sn", in *Proc. IPAC'17*, Copenhagen, Denmark, May 2017, pp. 1145–1148. doi:10.18429/JACoW-IPAC2017-MOPVA124

[5] U. Pudasaini, G. V. Eremeev, M. J. Kelley, C. E. Reece, and J. Tuggle, "Post-Processing of Nb3Sn Coated Nb", in *Proc. IPAC'17*, Copenhagen, Denmark, May 2017, pp. 1190–1193. doi:10.18429/JACoW-IPAC2017-MOPVA144

[6] C. Boffo, P. Bauer, T. Reid and R. Geng, "Electropolishing on Small Samples at Fermilab", in *Proceedings of the 12th International Workshop on RF Superconductivity*, Ithaca, New York, July 2005.

[7] D. L. Hall, M. Liepe, and J. T. Maniscalco, "RF Measurements on High Performance Nb3Sn Cavities", in *Proc. IPAC'16*, Busan, Korea, May 2016, pp. 2320–2322. doi:10.18429/JACoW-IPAC2016-WEPMR024

[8] P. Kneisel, "Surface Preparation for Niobium", *First Workshop on RF Superconductivity*, Karlsruhe, Germany, 1980, pp. 27-40.

[9] Wagner, Carl, "Contribution to the Theory of Electropolishing", *Journal of The Electrochemical Society*, vol. 101, no. 5, pp. 225-228, 1954.