EFFECTIVENESS OF CHEMICAL TREATMENTS FOR REDUCING THE SURFACE ROUGHNESS OF Nb_3Sn*

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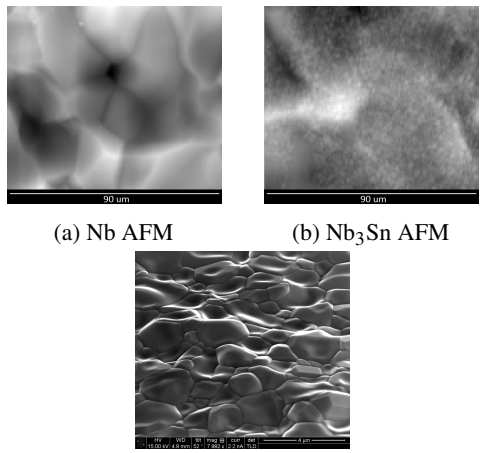
Abstract

Current Niobium-3 Tin (Nb_3Sn) superconducting radio-frequency (SRF) accelerator cavities have rougher surfaces than conventional electropolished Niobium accelerator cavities. The surface roughness can cause enhancement of the surface magnetic field, pushing it beyond the critical field. If this occurs over a large enough area it can cause the cavity to quench. The surface roughness may cause other effects that negatively impact cavity quality factor (Q) performance. Reducing surface roughness of Nb_3Sn cavities may be necessary to achieve higher gradient with high Q. Current chemical treatments for reducing the surface roughness of Niobium are challenging for Nb_3Sn: the Nb_3Sn layer is only ≈ 2 µm thick while it is difficult to remove less than 1 µm uniformly with most chemical treatments. This paper presents measurements of the surface roughness before and after Buffered Chemical Polish, Electropolishing and oxipolishing.

INTRODUCTION

Nb_3Sn cavities produced at Cornell University [1–5] have rougher surfaces than conventional electropolished Niobium cavities (see Fig. 1). Previous simulations have shown that the Nb_3Sn roughness can significantly enhance the surface magnetic (1% of the surface has magnetic fields enhanced by at least 45%) [6], possibly lowering the maximum achievable quench field or causing other deleterious effects. This is also supported by Klystron pulsed power measurements near Tc (see Fig. 2), which suggest the superheating field in our Nb_3Sn cavities is ≈ 230 mT (at 0 K) [7]. This is significantly lower than theoretical calculations that predict superheating fields of ≈ 400 mT (at 0 K) [8]. In these results the surface quench field is calculated based on the energy within the cavity, assuming a smooth geometry. A 45% surface magnetic field enhancement caused by roughness would increase the quench field measurement to ≈ 330 mT. Developing chemical treatments to reduce the surface roughness is therefore important for the development of Nb_3Sn cavities and will also allow for the removal undiscovered surface defects that impact performance



(a) Nb AFM (b) Nb_3Sn AFM

(c) Nb_3Sn SEM image.

Figure 1: Surface images of Nb and Nb_3Sn. Notice that the grain sizes of Nb_3Sn are on the order of microns, much smaller than Nb.

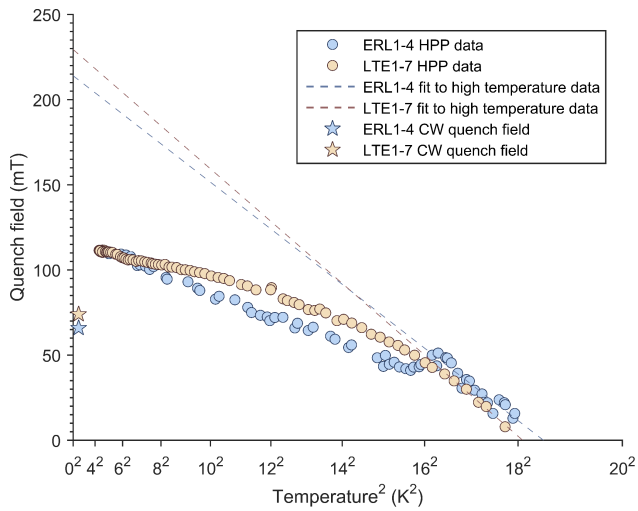


Figure 2: Plot of quench field (calculated from internal energy) versus T^2 from klystron high pulsed power measurements of Cornell Nb_3Sn cavities [7].

There are chemical treatments for reducing the roughness of niobium; however, they may be unreliable for etching thin (2 – 3 µm thick) layers of the Nb_3Sn. Here we present measurement of the impact of three common chemical treatments—Buffered Chemical Polishing (BCP), Electropolishing (EP) and oxipolishing—on the surface roughness of Nb_3Sn coated samples when used to etch less than two microns.

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METHOD

Niobium samples received a 100 µm BCP and were coated with Nb_3Sn using the standard Cornell recipe [9]. One sample received a 40 µm EP prior to coating. Some of the samples were scanned using an Asylum MFP-3D Atomic Force Microscope (AFM) at least 3 random locations using both 20 µm × 20 µm and 90 µm × 90 µm scan sizes. Not every sample was scanned due to insignificant differences between roughness measurements.

After Nb_3Sn coating, the samples were etched repeatedly using various etchants: 12 V, 0.03 A electropolish (10 s, 10 s, 20 s, 45 s and 75 s treatments); 30 V oxipolish (6, 9 and 9 passes); 1:1:2 buffered chemical polish (30 s); and 1:1:8 buffered chemical polish (15 s, 15 s). After each treatment, the samples were scanned as described above and height map was analyzed using an Amplitude Spectral Density (ASD) method detailed below. For the remainder of this paper the sample treatment will be described by the cumulative amount of treatment received.

AMPLITUDE SPECTRAL DENSITY

The magnetic field enhancement of a bump (roughness) depends on it's height, thickness and width [10] making single number characterization of the roughness (such as the root mean square roughness, R_a) inappropriate. This is because a large amplitude bump that rises slowly can contribute far less to the surface magnetic field enhancement than a smaller amplitude bump that rises sharply. In addition to this, chemical etches may preferentially favor certain spatial-frequency (one over length units) bands when reducing roughness.

To better characterize the surface roughness, we have adopted an "Amplitude Spectral Density" (ASD) approach based off of Power Spectral Density characterizations of surface topography. This characterization has been used heavily in optics and microelectronics and was applied to niobium surfaces by Chen Xu et. al. [11] on which we based our approach.

In this approach, we take the Fast Fourier Transform (FFT) of each line of our AFM scan and take the average of the absolute value for each spatial-frequency. If we had an infinite scan size and sampling rate, we would receive an amplitude density. Instead we receive discrete frequencies and amplitudes. We assume that the spectrum is continuous between points and rescale the values to be a density (in frequency) by multiplying by the square root of the scan size. The ASD is related to the root mean square roughness by $R_a = \sqrt{\int df\ ASD^2} = \sqrt{\int df\ PSD}$.

There are still several corrections that need to be applied to get the true ASD. Any roughness components with wavelengths larger than the scan size will be spread out over all the frequencies in the FFT, increasing the measured roughness and possibly skewing the plot. To correct for this and remove the low frequency components, the heightmaps are deskewed [11]. In this process a 3rd order polynomial is fit to, then subtracted from each line in the AFM scan (see Fig. 3). After this process, it is assumed that the largest wavelength component is compromised and it is ignored. The second issue is that frequency components exist in that height map that are in between FFT frequencies. Again, these components will be spread over the FFT frequencies, skewing the data. To correct for this we apply a Tukey window function (see Fig. 4) [11]. This smoothly reduces the heightmap at the edges to remove the mismatch at the ends caused by frequency components that do not perfectly divide the scan size.

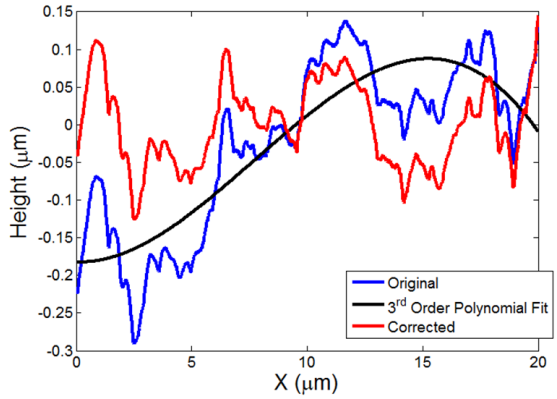

Figure 3: The polynomial correction applied to a height profile. The polynomial is fit to the height map, then subtracted from the height map.

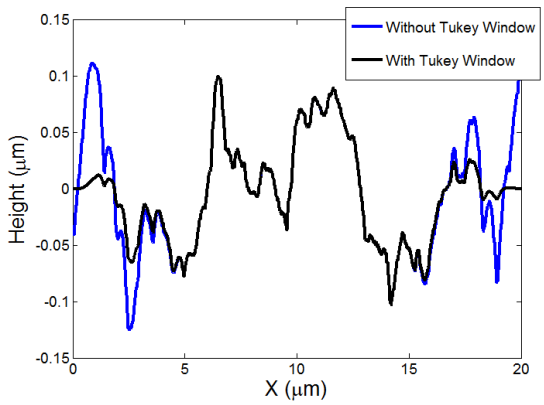

Figure 4: The Tukey window function applied to a height profile (already polynomial corrected). Note that the profile falls to zero at the edges after the Tukey window function is applied.

One correction that we do no apply is anti-aliasing. We have ignored this because the AFM already applies anti-aliasing and that high frequency components have very small amplitudes and are unlikely to skew the data.

Finally, the ASD from scans of the same sample were averaged and the standard deviation was calculated based on the three (or more) scans.

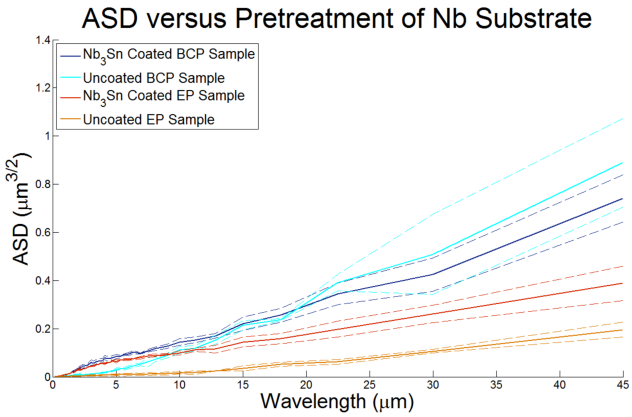

Figure 5: ADS of samples before and after coating for different pre-treatments.

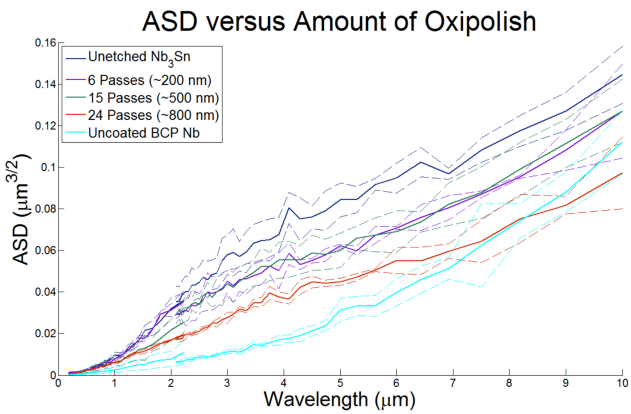

Figure 6: ADS of samples receiving different amounts of oxipolish. Approximate amount of removal listed.

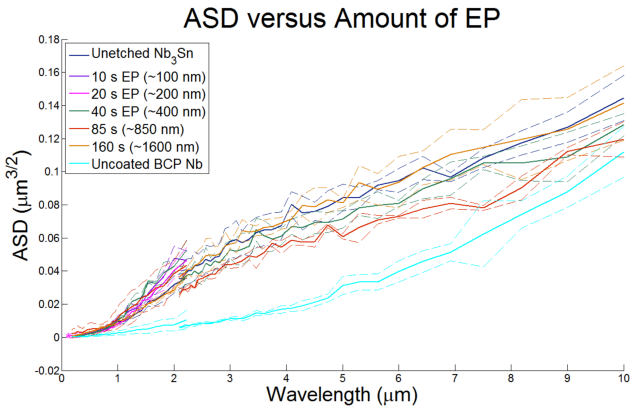

Figure 7: ADS of samples receiving different amounts of EP. Approximate amount of removal listed.

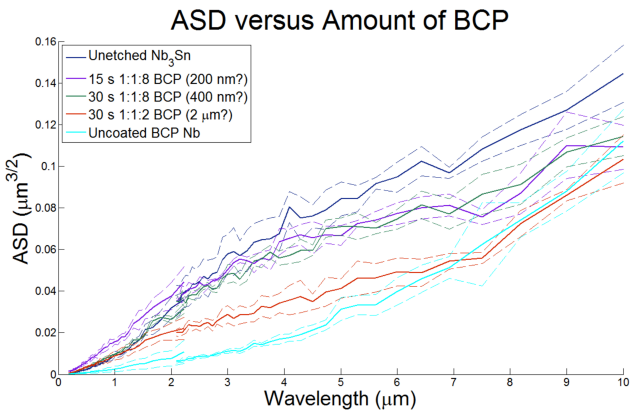

Figure 8: ADS of samples receiving different amounts of BCP. The amount of removal is unknown, but rough estimates are given.

RESULTS AND DISCUSSION

Figure 5 shows the ASD before and after coating of a sample that was BCP'd before coating and one that was EP'd before coating. At short length scales the roughness is determined primarily by the coating process than by the substrate roughness. On longer scales substrate roughness and coating process determine roughness. A simplified enhancement model suggests the most important region for magnetic field enhancement is the 1 to 10 micron range and will display all remaining plots only up to 10 μm. Additionally, above 10 μm all ASD profiles were consistent with each other (except for the samples EP'd before coating).

Oxipolishing shows a significant reduction in the surface roughness of the Nb_3Sn, with less than 1 μm etching (see Fig. 6). Electropolishing, however, did not reduce the surface roughness even when most of the surface layer was removed (see Fig. 7).

The initial BCP test was done with a 1:1:2 solution for 30 s at room temperature (typical etch rate of 1 μm/s). Though the treatment reduced the roughness (see Fig. 8), after the etching there appeared to be spots where bare niobium was showing, suggesting most of the Nb_3Sn layer was destroyed. The destruction of the surface layer was confirmed through SEM-EDS measurements showing little tin signal on the surface. In addition to this, a 1:1:8 BCP mixture was created and used to etch a sample. This sample was weighed before and after etching, revealing that after 30 cumulative seconds less than 500 nm was removed. Some roughness reduction was measured (see Fig. 8), but more treatments are needed to measure its effectiveness.

CONCLUSION

We have developed a way to characterize the surface roughness of our Nb_3Sn coatings and measured the change in roughness from BCP, EP and oxipolish chemistry. oxipolishing significantly reduced the surface roughness. EP did not reduce the surface roughness. Room temperature 1:1:2 BCP removes Nb_3Sn too quickly (during the first minute) to be easily controlled, but 1:1:8 BCP mixture etches at a slow enough rate to be controlled. Further work is needed to determine the magnetic field enhancement of the etched Nb_3Sn.

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