EXAMINATION OF CUTOUTS INNER SURFACES FROM Nb_3Sn COATED CAVITY*

Uttar Pudasaini[1], G. Eremeev[2], Charles E. Reece[2], J. Tuggle[3], M. J. Kelley[1,2,3]
[1]Applied Science Department, The College of William and Mary, Williamsburg, VA 23185, USA
[2]Thomas Jefferson National Accelerator Facility, Newport News, VA 23606, USA
[3]Virginia Polytechnic Institute and State University, Blacksburg, VA 24061, USA

Abstract

The potential for higher operating temperature and higher gradient has motivated SRF cavity researchers to pursue Nb_3Sn as an alternative to niobium for nearly fifty years. Far and away the most common embodiment has been a few micron thick Nb_3Sn layer on the cavity interior surface obtained by vapor diffusion coating, with one or another set of parameters. While many cavities have been made and RF tested, reports of dissecting a cavity in detail to examine the coating and relate it to RF measurements are rare. We coated a BCP-treated single cell cavity in a typical process of tin/tin chloride activation at 500 ºC followed by tin vapor deposition at 1200 ºC. After RF testing, we cut and examined sections from several locations to learn composition, thickness topography of the interior surface. The effect of process variables, such as surface preparation, process temperature and duration, and vapor chemistry needs to be explored.

INTRODUCTION

Nb_3Sn is an intermetallic alloy with superconducting properties to operate at higher temperature of about 4.2 K compared to standard niobium cavities which operate at 2 K. The estimated superheating field of Nb_3Sn also promises higher accelerating gradient than niobium [1]. Researchers had already realized the potential of Nb_3Sn more than 40 years ago, and attempted to fabricate Nb_3Sn coated cavities [2-4]. The vapor diffusion technique was readily successful to deposit a few micron thick Nb_3Sn coating on niobium cavity. These cavities were successful to attain quality factors $>10^{10}$ at few MeV/m gradients, but suffered with precipitous drop with increased gradient [5]. The reason for such degradation is not established so far. We encountered a sharp drop of quality factor in the cavity, C3C4, that was coated with Nb_3Sn during coating system commissioning at Jefferson Lab as shown in Fig. 1 [6]. C3C4 was a BCP treated 1.5 GHz single cell cavity, and coated in a typical process of tin/tin chloride activation at 500 ºC followed by tin vapour deposition at 1200 ºC. Detailed description of coating system and coating process is available in [7].

The cavity was dissected following RF test to pursue material studies of coated interior surface using the cavity cutouts. We aim to correlate the material studies results of cavity cutouts with their RF properties. First results from surface studies of cavity cutouts are presented in here.

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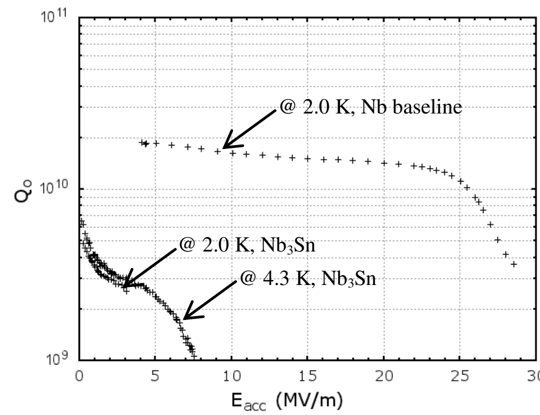

Figure 1: Q_o vs E_{acc} for C3C4 cavity before and after coating. Note the sharp drop of Q_o after coating.

CUTOUT PREPARATION

Temperature mapping of the cavity during RF test was used to choose areas from which to extract cutout samples. They were milled out at machine shop using 3/32 two flute OSG Exomini TiN coated end mill running at 3200 rpm. During the milling process, attempt was made to not cut through, but to leave a thin layer of material in order to reduce contamination and damage to the internal coating surface. This was not always possible due to cavity curvature, but some material was left when the milling was finished. The samples were then removed with pliers, and edges were de-burred with a scalpel or scissor before acetone rinse. The cutouts were again rinsed with methanol, and dried with ionized nitrogen. These cutouts further received ultrasonic cleaning with 2% detergent followed by low pressure ultra pure water rinsing. After drying with ionized nitrogen, these samples received second round of acetone rinse and methanol rinse subsequently. Finally, the cutouts were dried with ionized nitrogen. Several cutouts were prepared from cavity as well as beam pipes for analysis.

EXAMINATIONS AND RESULTS

Prepared cutout samples were examined with a field emission scanning electron microscope (FESEM) with energy dispersive X-ray spectroscopy (EDS) for microstructure and local composition. Atomic force microscope (AFM) was used to investigate surface topography. Electron backscattered diffraction (EBSD) was used to examine the cross-section of cutouts for crystallographic characterization.

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Microstructure and Local Composition

SEM images were captured from multiple locations of each sample. Uniform coverage of coating was observed in each sample as shown in Fig 2. Observed SEM images were very similar when the cutouts extracted from cavity region were examined. Composition was measured at multiple locations of each sample with EDS. Average compositions of each samples were found very similar. Average tin content of ~(24±2) atomic % was observed in each sample.

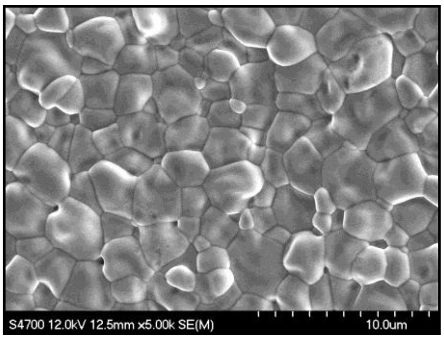

Figure 2: SEM image of Nb_3Sn coated surface on CVT10.

On the other hand these samples revealed microscopic pits in the coating. These pits were found normally at vertices where multiple grain boundaries meet as shown in Fig. 3. Presence of these pits with sharp edges is understood to be harmful for RF performance of cavity due to current and magnetic field enhancement [8,9].

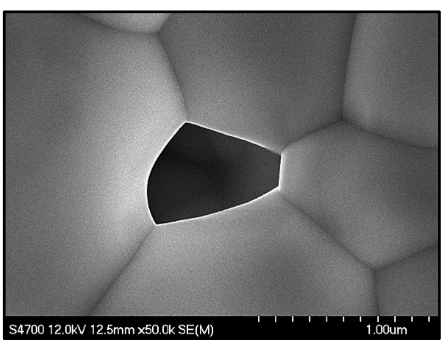

Figure 3: An example of pit found on CVT11. Dark area with bright outlines at the center is the pit. Bright outlines refer to sharp edges. Note that the size of the pit is ~0.5 μm.

Microstructure of cutout samples from top and bottom beam pipe were very similar to each other, but they were a little different than those obtained from cavity region. Average grain size of coatings on beam pipe was < 2 μm compared to average grain size of coating on cavity region, that is >2 μm. Also the difference in surface morphology is evident as shown in Fig. 4. No pits were observed during examination of beam pipe cutouts. Local composition of coatings from beam pipe cutouts were similar to that of cutouts from cavity region. Note that the cavity material had higher RRR (~300), while beam pipes were made out of reactor grade niobium (RRR~40).

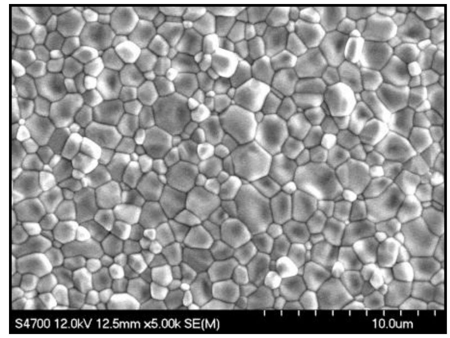

Figure 4: SEM image obtained from beam pipe cutout. Note that the structure of coating is different than cavity cutout shown in Fig. 2.

Patches with irregular grain structures were encountered in some cutout samples. Findings of similar areas were also reported by other researchers [10]. Most of those areas had the size that was equivalent to the area covered by few regular grains. Larger patches with size of few hundred square microns were encountered occasionally in specific samples. An example of such area is shown in Fig. 5. EDS measurement on these smooth areas shows less tin than regular areas, a probable indication of thin coating.

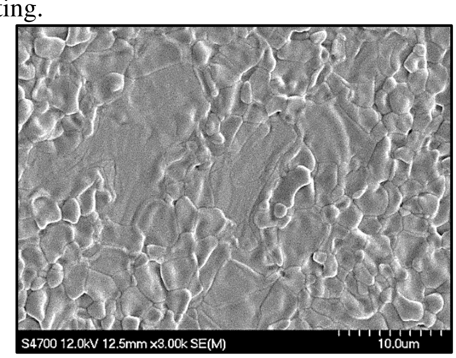

Figure 5: SEM image of patchy area obtained on CVT14.

Topography

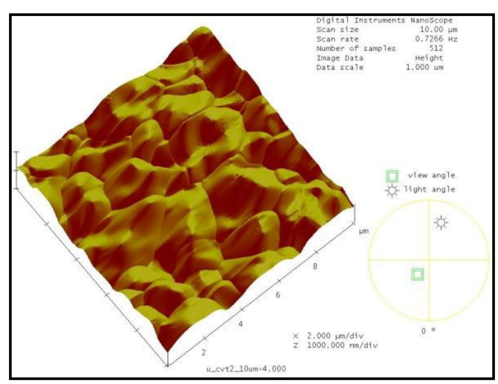

Figure 6: AFM image of regular Nb_3Sn coatings on cavity cutout. Roughness is evident with curved facets.

AFM was successfully used only on few samples due to the curvature of cutouts. AFM results were consistent with SEM/EDS results to locate the pits and irregular grain structure in the coating. A typical illustration of coating topography on cavity is shown in Fig. 6. Roughness was also measured from 5 μm x 5 μm and 50 μm x

50 μm scans. Four samples were scanned and expressed in terms of average power spectral densities (PSD) for comparison. Note that the area under PSD curve corresponds directly to root-mean-square (RMS) roughness. Difference of roughness was not found very significant as shown in Fig. 7.

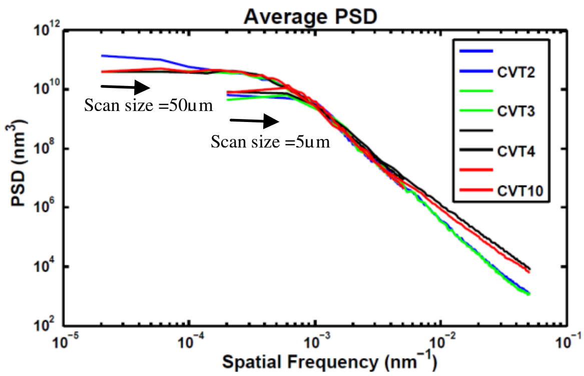

Figure 7: Comparison of average PSDs of cutouts calculated using AFM data. CVT4 and CVT10 found rougher in high frequency end (lateral scale 0.1 µm and less) indicating the presence of more microscopic features than CVT2 and CVT3.

Cross-section Measurement

Cross-sections were prepared using focused ion beam (FIB) after applying protective layer of platinum onto the coating surface. Obtained cross section was further polished using ion mill. SEM images of cross-sections were then captured to measure the coating thickness as shown in Fig. 8. Coating thickness was measured at several locations of cutout samples from cavity region and beam pipes. Coating thickness varied between 2-3 µm in cavity region. Only 1.5-2.5 µm thick coating was observed in beam pipes. Coating thickness was not found to vary between top and bottom beam pipes. Thickness of coating was found as thin as 400 nm in the patchy area, see Fig. 9, conforming thin coating on such areas.

EBSD images were captured from cutout cross-sections to insight the structure of coating. Representative orientation image maps (OIM) obtained from cutout samples are shown in Figure 10. Columnar grains were normally observed going from top to bottom. We also observed the formation of non columnar small grains.

Conclusion and Future Work

We examined the inner surface of Nb_3Sn coated niobium cavity using cutouts obtained by cavity dissection. Uniform distribution of coating was observed throughout the surface with some patchy areas containing irregular grains with thinner coating. Microscopic pits were observed in the coating which could have potentially harmed the cavity performance. The surface was found to be rough, but variation of roughness between examined samples was not very significant. Coatings thickness, grain size and grain growth were found to vary between cavity region and beam pipes. We are in process of using other characterization tools to examine these samples. We will finally attempt to compare the material properties of coating from different areas to corresponding RF performance obtained using temperature map.

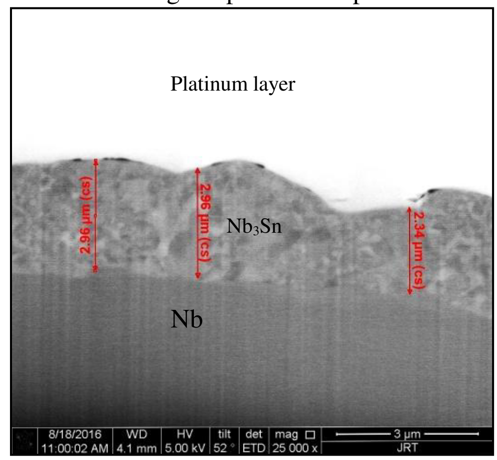

Figure 8: SEM image of a FIB cross-section obtained from CVT 10. Thickness varied from 2.34-2.96 µm.

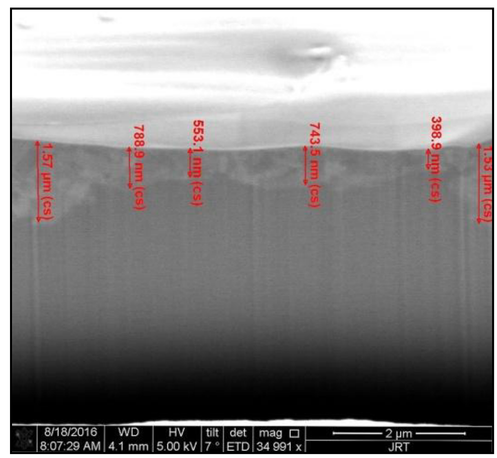

Figure 9: SEM image of a FIB cross-section of patchy area similar to Figure 5. Coating is significantly thin (400-800 nm) compared to neighbouring area (>1.5 µm).

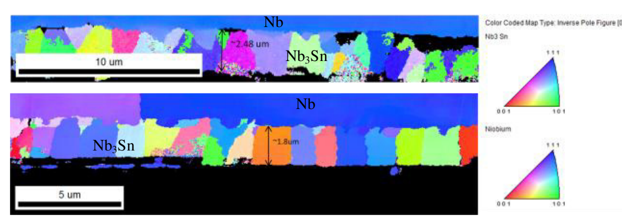

Figure 10: EBSD images obtained from sample CVT 10 at top which is obtained from cavity region. Image at bottom is from beam pipe cutout. Beam pipe has thinner coating with more columnar grains than CVT10. Some instrumental artifacts (dark area in between Nb/Nb_3Sn grains) are present.

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