MATERIALS CHARACTERIZATION FOR SRF CAVITIES: GAINING INSIGHT INTO Nb_3Sn

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Abstract

Although SRF accelerators are an invaluable research tool they can be painfully expensive to construct and operate at the current level of SRF technology. This cost is significantly due to the necessity to operate at a temperature of only 2K. Considerable research is currently underway into next generation SRF cavity technologies such as N-doping and Nb_3Sn coating. Both of these technologies will lower the cryogenic load of accelerators, correspondingly lowering both construction and operating costs. However, current understanding of either technology is incomplete and in order to elucidate the underlying mechanisms there is a need to push current characterization methods forward. In this work, ion beam techniques (e.g. focused ion beam (FIB)), and electron backscatter diffraction (EBSD) were applied to help understand Nb_3Sn coating mechanisms. This presentation will focus on characterization, providing examples of EBSD work, along with discussion of some of the issues encountered while trying to produce high quality EBSD data.

INTRODUCTION

EBSD is a technique applicable to crystalline samples, which utilizes electron diffraction to determine and map crystalline orientation within a sample. Lateral resolution of ~10 nm is possible with an information depth of ~50 nm [1]. EBSD hardware is usually mounted as an addition on an SEM/EDS instrument allowing a materials microstructure to be characterized quite thoroughly with a single instrument. Grain size and shape, misorientation between and within grains, phase content, and texture results such as inverse pole figures can be obtained. In addition, data can be combined with elemental mapping if installed as an integrated EDS/EBSD as the instrument utilized here.

EXPERIMENTAL

Well prepared samples are essential for EBSD analysis and sample preparation makes up the vast majority of the method development and work required. Any significant amount of amorphous surface layer, such as deformation from mechanical preparation or surface oxides can prevent EBSD measurement. Metal specimens prepared with normal polishing techniques require further polishing to reduce the deformation layer. Normally colloidal silica suspensions in the size range of 0.02 µm are sufficient for a final polish. In the case of Nb/Nb_3Sn, due to the soft nature of Nb, hardness difference between Nb and Nb_3Sn, and edge retention needed at both the Nb/Nb_3Sn interface and Nb_3Sn surface it was found impractical to achieve quality results by mechanical only polishing. As a final polish, ion beam techniques were employed and found to perform well, giving high quality surfaces excellent for EBSD analysis. Both diffuse ion beam and focused ion beam (FIB) techniques were utilized with great success.

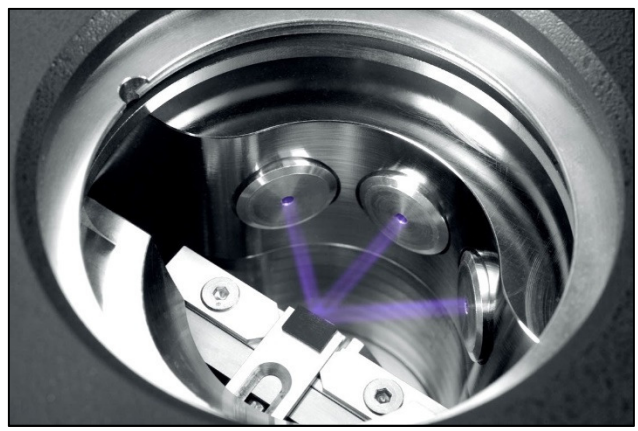

Figure 1: Leica TIC 3x sample chamber with triple argon ion beams. Sample location is at beam convergence point.

Diffuse Beam Preparation

Diffuse ion beam polishing was accomplished via a Leica EM TIC 3x milling system. The TIC 3x utilizes three loosely focused argon ion guns in order to polish a relatively large area. The sample chamber with three argon ion beams can be seen in Figure 1. Utilizing the TIC 3x we have successfully prepared areas of Nb_3Sn coated Nb up to several hundred microns in length. Since the ion beams utilized are diffuse in nature, care must be taken mounting and masking the sample in a way to protect the surface and uniformly sputter material away over the area of interest. With ion beam techniques the main regulators for damage depth are accelerating voltage and incidence angle. Best results were found with incidence angles $<10^{o}$ and by working stepwise from 10kV down to ≤3kV. Stepping down accelerating voltage has the same effect as moving from lower to higher grit sandpaper, successively removing less material and causing less damage with each step. Eventually only a few nm's of damage remain; less than ~3 nm under the above conditions [2].

While diffuse ion polishing via the TIC creates EBSD quality surfaces, sample size and shape are somewhat limited. In addition, special care has to be taken in sample mounting for regularly shaped coupons. Sample mounting becomes quite difficult with anything outside of the simplest geometries.

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ISBN 978-3-95450-182-3

Focused Ion Beam Sample Preparation

Focused ion beam polishing was accomplished utilizing the FEI NanoLab 600 dual beam. The NanoLab makes use of a FIB with Ga source capable of 1-30 kV, with a maximum of 21 nA of current. While FIB preparation of cross sections yields smaller cross sections and is more complicated in nature than preparation by the TIC, the quality of analysis surface is unsurpassed, and quite complicated geometries, such as SRF cavity cutouts, can be prepared.

For FIB preparation, desirably, a cross section should be initially mechanically polished. Starting with a well-polished cross section makes FIB polishing much more efficient and practical as FIB removes relatively small amounts of material at low rates. In order to preserve the surface of the Nb_3Sn and create an intact cross section a protective layer of Pt is deposited on the sample surface over the cross sections area of interest. Once the surface is protected a similar approach is taken as with TIC polishing stepping down through accelerating voltages and beam currents, removing smaller and smaller amounts of material, while subsequently causing less damage. Initial material removal steps are performed at the highest removal rate of 30 kV / 21 nA and stepped down to 2 kV / ~50 pA. With a low incidence angle (~1°) and 2 kV accelerating voltage, the damage will again be limited to the first ~3 nm of the polished surface [2]. Optimal patterns have only been collected after a 2 kV final polish.

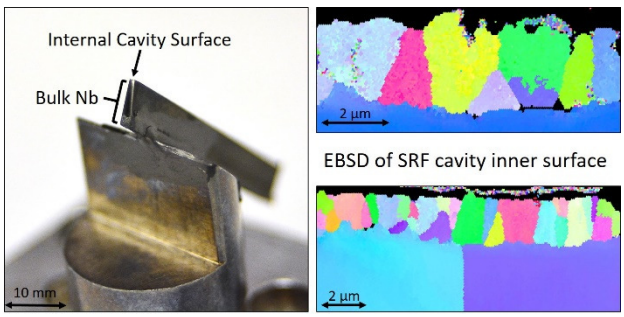

Figure 2: Cut-out sample from a coated and tested SRF cavity mounted for FIB sample preparation and orientation maps.

Where FIB sample preparation excels beyond other sample preparation techniques is in its ability to deal with small specimens or samples with complicated geometries. Cavity cutouts (segments cut from a wall of an SRF Cavity) are an example with compound curved or concave surfaces and often small in size. Figure 2 shows an example of a small cavity cut out with less than 1 mm of cavity surface available for analysis. The cross section was polished to a 2 kV surface finish via FIB. Grain orientation maps collected from the cut-out can also be seen in Figure 2.

EBSD Collection

EBSD was performed via the FEI NanoLab 600 dual beam, which is equipped with an integrated EDS/EBSD collection system, including an EDAX TSL EBSD camera and Octane Elite EDS with 25 mm^2 detector. Typical EBSD conditions range in voltages from 10-30 kV and beam currents of 1-50 nA at 20° incidence angle [1][3]. Depending on material and surface conditions voltages and currents should be adjusted for best results. Optimal conditions for Nb_3Sn with 2 kV surface polish were found to be 30 kV and 5.5 nA with an incidence angle of ~14°. This lower incidence angle increases interaction volume while decreasing depth, which reduces lateral resolution, but helps to increase the signal to noise ratio.

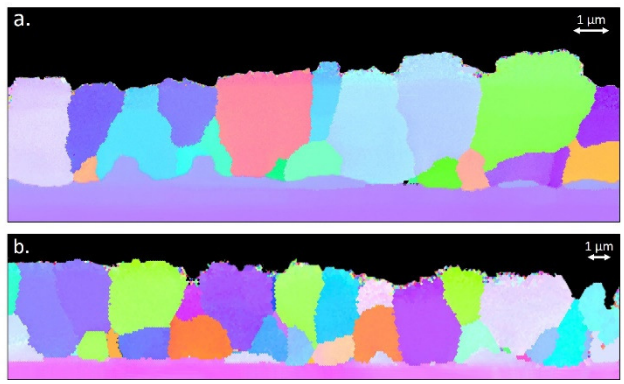

Figure 3: EBSD of overcoat samples showing additional Nb_3Sn growth at Nb interface.

RESULTS AND DISCUSSION

Provided here are several examples and brief discussion of how the EBSD technique is helping to elucidate the Nb3Sn coating process. For further experimental details and discussion regarding formation of Nb3Sn please see U. Pudasaini, et al, in the upcoming SRF 2017 proceedings [4].

The Overcoat Experiment

In order to gain insight into the formation and growth of Nb_3Sn films previously coated coupons were subjected to additional coating and characterized. These coupons were referred to as “overcoat” samples. The overcoat samples were characterized by EBSD in cross section. (Figure 3) EBSD shows the formation of new grains at the Nb_3Sn/Nb interface. This indicates a formation mechanism which includes additional Sn diffusing to the interface and initiating additional grain growth, as opposed to the formation of a new surface layer. In addition, grain formation appears to occur more times than not at the intersection of a Nb_3Sn grain boundary and the Nb interface, indicating grain boundary diffusion as the primary mode for Sn movement to the interface. Further evidence of this can be seen in the “cupping” at the base of many Nb_3Sn grains (see Figure 3a), showing grain growth faster around the edges of the grain. With grain boundary diffusion as the primary mode of transport of Sn to the growth interface, it follows that growth rate is proportional to grain size.

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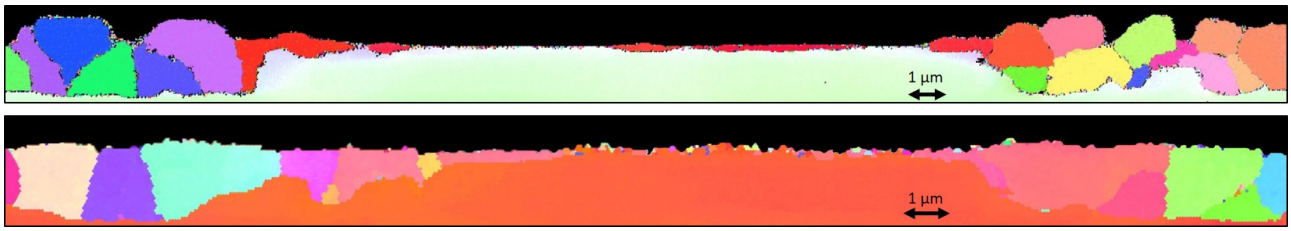

Figure 4: EBSD orientation maps for FIB polished cross sections of Nb_3Sn patches. Patches were seen as a continuous layer in SEM imaging, however possibly due to polishing defects or the low incidence angle utilized for analysis, the thinnest parts of the patch were not completely resolved in the EBSD maps.

Patch Defects

Patches are defects which form during the Nb_3Sn coating process and are seemingly universal to varying degree across many bulk material, cavity, and coating conditions. EDS surface analysis was reported by others, which shows varying Nb/Sn ratio with changing accelerating voltage [5]. This variation is indicative of a thin surface layer with thickness less than the escape depth of characteristic X-rays detected in EDS. The expected escape depth for Nb/Sn at the low end of the reported accelerating voltages is still several hundred nanometers, making it difficult to judge actual thickness. Here, FIB cross section analysis was utilized to quantify thickness of several patches from a single coupon and patch thickness was found to be 194(±59) nm as compared to Nb_3Sn thickness of 1.6(±0.1) µm for the coupon.

EBSD results from FIB prepared patches in cross section can be found in Figure 4. In agreement with the previous overcoat EBSD it is apparent from both Figure 4 maps that the Nb_3Sn layer forms at the Nb interface and progresses downward using up Nb as Sn is made available at the interface. A combination of relatively high beam current, low incidence angle, and damage from polishing leads to the thinnest parts of the patch being unresolved in the EBSD maps shown. However, in all patches observed by SEM the Nb_3Sn layer appeared to be thin, but continuous across the area of the patch. Figure 5 shows an example patch cross sectioned via FIB.

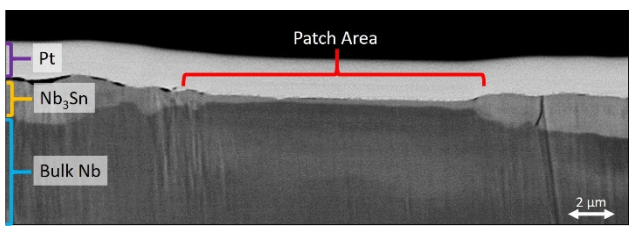

Figure 5: SEM image of FIB cross section from a patch showing a continuous Nb_3Sn layer ~200 nm thick.

Surface EBSD analysis of Nb_3Sn is made difficult by the prominent topography of the surface. The combination of surface topography and low incidence angle (~20°) of the electron beam required for analysis forms many "shadowed" (black areas in Fig. 6) regions where no signal is produced. While this produces a noisy surface map the large patch areas are relatively smooth and index well. Figure 6 shows an SEM image with corresponding EBSD map. In agreement with the cross sections, this map shows the patches are large single grains of Nb_3Sn.

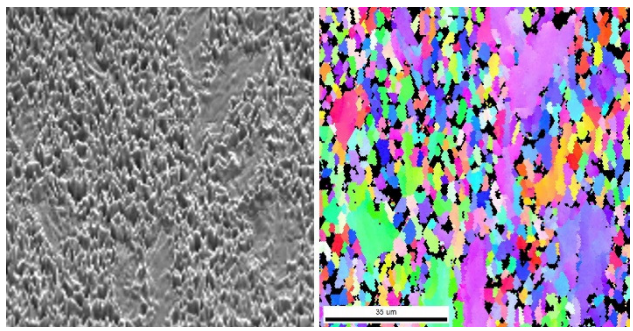

Figure 6: SEM image and corresponding orientation map of Nb_3Sn surface area containing patches. Patches index as single crystal Nb_3Sn.

CONCLUSION

As seen in both the overcoat experiment and the Nb_3Sn patch characterization, EBSD will play a vital role in the quest to understand the next generation of SRF materials. The example work shown here indicates Nb_3Sn forms at the Nb_3Sn/Nb interface, progressing "downward", consuming bulk Nb as Sn is made available at the interface through grain boundary diffusion. This implies patches arise from the formation of large single crystal grains early in the coating process and with a lack of grain boundaries the interface is starved of Sn inhibiting further growth of Nb_3Sn in the patch area.

For further discussion of Nb_3Sn formation please see U. Pudasaini, et al, in the upcoming SRF 2017 proceedings [4].

ACKNOWLEDGMENTS

- Co-Authored by Jefferson Science Associates, LLC under U.S. DOE Contract No. DE-AC05-06OR23177
- We are grateful for support from the Office of High Energy Physics, U.S. Department of Energy under grant DE-SC0014475 to the College of William & Mary.
- The Institute for Critical Technology and Applied Science (ICTAS), and Nanoscale Characterization and Fabrication Laboratory (NCFL) at Virginia Tech
- Travel to IPAC'17 supported by the Division of Physics of the United States National Science Foundation (Accelerator Science Program) and the Division of Beam Physics of the American Physical Society

ISBN 978-3-95450-182-3

REFERENCES

[1] A.J. Schwartz, M. Kumar, B.L. Adams, "Electron Backscatter Diffraction in Materials Science", Springer, 2009

[2] Stopping Range of Ions In Matter [Software], ver: SRIM-2013.00, www.SRIM.org

[3] T. Maitland, S. Sitzman, Chapter exert from "Scanning Microscopy for Nanotechnology Techniques and Applications", Springer, 2007

[4] U. Pudasaini, G. Eremeev, C.E. Reece, J. Tuggle, M.J. Kelley, "Insights into formation of Nb_3Sn Film during the vapor diffusion process" Upcoming SRF 2017 Proceedings, Lanzhou, China 2017

[5] Daniel Leslie Hall, J.J. Kaufman, M. Liepe, J.T. Maniscalco, "Surface Analysis Studies of Nb_3Sn Thin Films", IPAC 2016 Proceedings, Busan, Korea 2016