Final Scientific/Technical Report GRANT NO. DE-FG02-06ER41450

1. DOE award number: GRANT NO. DE-FG02-06ER41450
Name of recipient: Florida State University
Office of Sponsored Research
97 South Woodward Avenue, 3rd Floor 6.
Tallahassee FL 32306-4166
Project title: Magnetic Investigation of High Purity Niobium for
Superconducting RF Cavities
Principal investigators: Peter J. Lee and David C. Larbalestier

2. Distribution limitation notices: None

3. Executive summary (layman one paragraph)

Remarkable progress towards understanding of the behavior of Nb grain boundaries in high purity cavity sheet has been accomplished. An excellent graduate student has been trained in superconductor characterization and he has developed new techniques for the preparation of high quality electron transparent samples for transmission electron microscopy so that electrical and magneto optical measurements can be made on the same local areas. Nb grain boundaries have been characterized for surface topography, superconducting flux penetration, transport current measurement and microstructure. An excellent framework for continued studies in this area has been established. The work has been well received in the SRF community and has been presented at international workshops.

4. Comparison of the actual accomplishments with the goals and objectives of the project.

The following project goals were defined:

- 1) *to improve the understanding of the effect of the chemical composition and topology of the surface, in general, and the grain boundaries, in particular, of state of the art high purity niobium for SRF cavities, including e-beam weld regions, on the capability of the material to shield magnetic flux;*
- 2) *to provide quantitative estimates of the grain-boundary critical currents and grain boundary resistance (in the normal state) such as further characterize grain-boundaries;*
- 3) *to correlate surface and grain-boundary chemical composition with the RF performance of SRF cavities, such as for instance for the case of the observed improvement of performance following a low temperature, in situ bake-out;*

As summarized below we successfully trained an excellent graduate student, Zu Hawn Sung, who has made excellent grain boundary electrical and microchemical measurements of the grain boundaries in high purity Nb for SRF cavities (objectives 1 and 2). The techniques developed in this proposal can now be applied to additional samples that have undergone different processing routes (objective 3), such measurements have been initiated in collaboration with FNAL and will continue under funding via FNAL now that this program has ended.

5. Summary

The effect of GBs on the superconducting properties of an SRF niobium cavity is still in an open question. GBs are certainly one defect that can be responsible for depressing the field at which flux vortex penetration occurs by locally reducing the superconducting gap, the GB depairing current density J_b and enhancing the excess resistance R_f . Therefore, in order to better understand whether or how grain boundary weakness can affect intrinsic limit of an SRF cavity performance, we have investigated DC flux penetration at GBs by correlating the local superconducting properties (as determined by magneto-optical imaging and atomic-scale structure analysis by TEM) with the global superconducting properties (as determined by transport measurement) of BCP-treated bi-crystals isolated from as-received, large grain, high purity niobium sheet.

ZFC (Zero Field Cooled) Magneto-optical (MO) images in Fig.1., show that flux penetration that did not occur on $\sim 25^\circ$ tilted GB, from top to bottom of sample, can appears when the plane of GB is aligned to be parallel to external field by rotating the sample. The comparison of MO images on mechanically flattened surface with BCP'ed one suggests that the topological features introduced by BCP might be not places of nucleation of magnetic flux, even though FC (Field Cooled) mode shows no bulk current perturbation at GB.

Figure 1. Surface optical images and Magneto Optical (MO) images before/after mechanical surface flattening

No enhanced indentation of Nb-O layer at the GB was observed by our TEM analysis after FIB sample cross-sectioning. One of our thin TEM lamellae, fabricated by *state of the art, in situ* FIB technique, is shown in Fig 2. For this sample prep, double protective caps (Au-Pd & C) were coated in PECS (Gatan) system in order to protect top Nb oxide layer from the focused Ga ion beam. Bright Field (BF) TEM images at the vicinity of the grain boundary are shown in Fig. 3, and clearly indicate that there is no preferential oxidization at the grain boundary during BCP surface treatment in these samples (even though the surface energy

Figure 2. Fabrication of TEM sample across Nb grain boundary using focused Ga ion beam.

for preferential etching at the grain boundary increases as the mis-orientation between grains increases).

Figure 3. BF TEM images of cross sectional view at the vicinity of the grain boundary according to misorientation across the grain boundary a) 19°, b) 25°, and c) 32°.

Nevertheless, as shown in Fig. 4, the ohmic behavior of the V-I curve from transport measurement as a function of perpendicular magnetic field indicates the perturbation of the transport current due to preferential etching of grain boundaries during optimized cavity surface fabrication. Our sample preparation technique for this transport measurement is well demonstrated in the light and 3D surface topology images in Fig. 4. After BCP treatment on the I-shaped bi-xtal sample, followed by mechanical thinning, the center of the bridge of the I-shape sample was further reduced by BCP etching to minimize transport current. Although the extracted $R(H)$ for linear V-I response for the Nb grain boundary does not follow the behavior of a grain boundary weak link by the flow of Abrikosov Josephson (AJ) vortices along the grain boundary before Abrikosov (A) vortices pinning in grains, it have been shown that the deterioration of superconducting properties at the grain boundary by critical SRF cavity inner surface treatment like BCP (Buffer Chemical Polishing), EP (Electro Polishing) or OP (Oxipolishing) is inevitable.

Figure 4. Contrasting VI curves from single and bi-crystal samples show clear evidence of suppressed superconductivity at the GB.

In conjunction with the investigation of intrinsic limits for SRF properties of Nb cavity, since surface topology also produces significant local field enhancement, we have made considerable effort to assess the value of non-contact topological measurement techniques for chemically treated or e-beam welds from single cell cavity so as to quantify the local topology. In this study we compared surface height measurements from a scanning (violet) laser confocal microscope, SLCM, that provides spatial resolution in the Z-direction by eliminating out-of-focus information in object planes of the optical system, with a commercial software package [Mex 5.0 Alicona Imaging] that extracts height information from sets of 2 or 3 tilted Eucentric images, in this case obtained using a high resolution FESEM (Zeiss 1540 EsB).

The surface topological data produced by the LSCM confocal microscope is illustrated in Fig. 5 (a) and (b) as a texture image, 2D intensity image, and 3D imaging. Together they show the characteristic topology in the vicinity of the GB created by the optimized BCP cavity surface treatment. The topology was automatically obtained with a height resolution of up to ~ 10 nm as part of the standard multi-plane LSCM image acquisition technique. Also Digital stage control allowed us to make volumetric analyses of our entire specimens which are useful for our electrical measurements like shown in the top right image of Fig. 4; 3D surface topology.

Because of the high depth of field of SEM we used the software package to interpret height information from the geometric movement of features when the sample is tilted. For our samples the optimum tilt angles and working distance were found to be $\pm 7^\circ$ and 5 mm respectively. Fig. 5 shows (c) the 2D contour image determined from 3D imaging processing by combining three captured SE images along with eucentric tilting from -7° , 0° , to $+7^\circ$. Both results provide comprehensive depth information for the GB. However, compared to SLCM, the results from the SEM imaging indicate additional sub-micron height variations that do correspond to visible surface features observed in the secondary electron image. The magnitude of the grain boundary height is in good agreement with that from the same area from the SLCM.

Figure 5. (a) Surface intensity image, and (b) 3D height map with height profile by Olympus LEXT OLS 3100, (c) 2D contour image obtained from Mex 5.0 digital imaging process in Carl Zeiss 1540 EsB-FESEM, and (d) Comparison of height

Current publications of the Applied Superconductivity Center are publicly available from:

<http://www.magnet.fsu.edu/magnettechnology/research/asc/publications.aspx>

Peer-Reviewed Publications

1. Lee, P.J.; Polyanskii, A.A.; Sung, Z.; Larbalestier, D.C.; Antoine, C.; Bauer, P.; Boffo, C. and Edwards, H., Flux Penetration Into Grain Boundaries Large Grain Niobium Sheet For SRF Cavities: Angular Sensitivity, Proc. Single Crystal Niobium Technology Workshop, Araxá, Brazil, Oct. 30-Nov. 1 (2006), AIP Conference Proceedings 927, ed. G. Myneni, T. Carneiro and A. Hutton, 927, 113-120 (2007)
2. Sung Z., Lee, P.J., and Larbalestier, D.C., Non-Contact 3D Surface Metrology of Large Grain High Purity Niobium, Proc. Microscopy and Microanalysis Meeting, Albuquerque, NM, August 3-7 (2008).

Presentations, Posters & Abstracts

1. Sung Z., Lee, P.J., and Larbalestier, D.C., Non-Contact 3D Surface Metrology of Large Grain High Purity Niobium, Microscopy and Microanalysis Meeting, Albuquerque, NM, August 3-7 (2008).
2. Sung Z., Lee, P.J., Polyanskii, A.A., Gurevich, A., and Larbalestier, D.C., The effect of grain boundaries on large grain high purity niobium for an SRF cavity, Applied Superconductivity Conference, Chicago, IL, August 17-22 (2008)
3. Sung Z., Lee, P.J., Polyanskii, A.A., Gurevich, A., and Larbalestier, D.C., An Investigation of the influence of grain boundaries on flux penetration in high purity large grain niobium for particle accelerators, 13th Int. SRF Workshop, Beijing, China, October 11-19 (2007)
4. Sung Z., Lee, P.J., Polyanskii, A.A., Gurevich, A., and Larbalestier, D.C., Analytical electron microscopy studies and transport characteristics of large grain niobium for SRF cavity, SRF material workshop, Fermi National Laboratory, May 23-24 (2007)
5. Lee, P.J.; Polyanskii, A.A.; Squitieri, A.A.; Gurevich A.; Sung, Z. and Larbalestier, D.C., Nb for SRF Cavities: Bi-Crystal and Tri-Crystal Studies, Midwest SCRF Materials Workshop, National Superconducting Cyclotron Center, Michigan State University, East Lansing, MI, March 17 (2006) [read online]
6. Lee, P.J.; Polyanskii, A.A.; Sung, Z.; Larbalestier, D.C.; Antoine, C.; Bauer, P.C.; Boffo, C. and Edwards, H.T., Flux Penetration Into Grain Boundaries Large Grain Niobium Sheet For SRF Cavities: Angular Sensitivity, Proceedings of the Single Crystal Niobium Technology Workshop, Araxa, Brazil, October 30-November 1 (2006) [read online]
7. Lee, P.J.; Polyanskii, A.A.; Sung, Z.; Larbalestier, D.C.; Antoine, C.; Bauer, P.C.; Boffo, C. and Edwards, H.T., Grain Boundaries Studies at the Applied Superconductivity Center, Midwest SCRF Materials Meeting, Argonne National Laboratory, November 14 (2006) [read online]
8. Lee, P.J.; Polyanskii, A.A.; Sung, Z.H.; Gurevich A.; Larbalestier, D.C.; Bauer, P.C.; Boffo, C.; Antoine, C. and Edwards, H.T., Magneto Optical and Microstructural Investigation of Grain Boundaries in Large Grain High Purity Niobium for Superconducting RF Cavities, Applied Superconductivity Conference, Seattle, WA, August 27-July 1 (2006) [read online]
9. Polyanskii, A.A.; Lee, P.J.; Gurevich, A.; Sung, Z.; Larbalestier, D.C.; Antoine, C.; Bauer, P.C.; Boffo, C. and Edwards, H.T., Review of Magneto-Optical Resolution on High-Purity NB for Superconducting RF Application, Proceeding of The International Workshop on Thin Films and New Ideas for Pushing the Limits of RF Superconductivity, Padua, Italy, October 9-12 (2006)

- c. Networks or collaborations fostered;

As illustrated above this work enhanced our collaboration with FNAL and was an integral part of the Midwest Cavity working group that has now become .

The techniques developed in this work are being used by us to study sample coupons prepared at FNAL and multilayers produced by JLab.

A SHARE proposal was awarded at ORNL to use EELS to study the microchemistry and the Nb grain boundaries. This aspect of the work is also continuing.

- d. Technologies/Techniques;

See *Publications*

- e. Inventions/Patent Applications, licensing agreements;

None

- f. Other products, such as data or databases, physical collections, audio or video, software or netware, models, educational aid or curricula, instruments or equipment.

NA

7.

Electronic Submission. The final scientific/technical report must be submitted electronically via the DOE Energy Link System (E-Link) at <http://www.osti.gov/mlink-2413>.

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