GRAIN BOUNDARY FLUX PENETRATION AND RESISTIVITY IN LARGE GRAIN NIOBIUM SHEET*

P. J. Lee #, A. A. Polyanskii, A. Gurevich, A. A. Squitieri, D. C. Larbalestier, Applied Superconductivity Center, University of Wisconsin-Madison, USA
P. C. Bauer, C. Boffo, H. T. Edwards, Fermilab, Batavia, IL, USA

Abstract

Kneisel, Ciovati, Myneni and co-workers at TJNAF have recently fabricated two superconducting cavities from the center of a large grain Nb billet manufactured by CBMM. Both cavities had excellent properties with one attaining an accelerating gradient of 45 MV/m (2 K) after a 48 h and 120 °C bake [1]. An investigation is underway to use Magneto Optical (MO) imaging to observe the flux penetration behavior of a sheet sliced from this billet. The large grain size (some larger than 50 mm) allowed us to isolate multiple bi-crystals and tri-crystals. In the first stage of the present study we have taken the as-received sheet (RRR ~280), which has been etched to reveal the grain structure. By magneto-optical examination we observed preferential flux penetration at some grain boundaries of a bi-crystal where the grain boundary was almost perpendicular to the sample surface and there was <1 µm surface step across the boundary. At other grain boundaries, with large steps or where the grain boundaries were not normal to the surface, we observed no preferential flux pentration. Preliminary transport measurements on a bicrystal showed greater normal state resistance and lower superconducting critical current at the grain boundary.

INTRODUCTION

An understanding of what degrades the performance of high purity Nb used for SRF cavity fabrication is vital to the success of the ILC. Using magneto-optical (MO) imaging we have recently shown that some Nb grain boundaries show preferential flux penetration below that seen in the grains.[1] This result was the first direct evidence of depressed superconductivity at a Nb grain boundary (GB), however there was also extensive surface topology associated with many grain boundaries in that study and the BCP sample had undergone an extensive heat treatment in order to enlarge the grain size. This heat treatment may also have contributed to the poisoning of the GB. In this study we examine grain boundaries in a slice of one of the CBMM extremely large-grain billets. In this first report we examine the behavior of samples from this as-received ingot slice after minimal BCP polishing and etching to reveal the grain locations.

In this first series of experiments we examine bi-crystal and tri-crystal regions of the sample in the as-cut state that is close to the as-received state of the slice. Only light BCP and GB etching was applied. Cutting has left some residual cold work in the samples. In subsequent studies we will make the same measurements at each step of a typical BCP and heat treatment procedure.

The advantages of using single crystal or very large grain sheet for cavities and recent results on such cavities manufactured by TJNF are reported elsewhere in this volume.[2]

EXPERIMENTAL TECHNIQUE

The billet slice, in the as received condition, is shown in Figure 1. The side shown was given sufficient BCP treatment at TJNAF to reveal the grain boundary locations but some residual marks from cutting can still be seen in the top left hand corner. The reverse side was in the rough-cut condition which necessitated further mechanical polishing followed by etching so that we

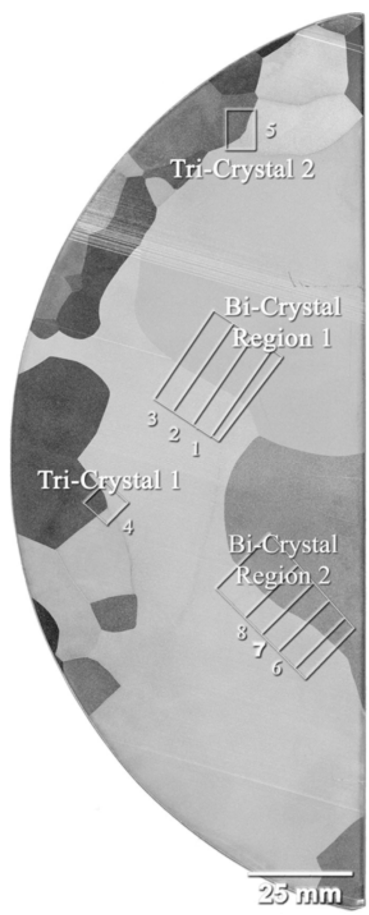


Figure 1. Overview of as-received ingot slice showing bi- and tri-crystal regions used for this series of experiments.

* The work at UW-Madison was funded by Fermilab contract #559390 and DOE-HEP under grant DE-FG02-91ER40643
#lee@engr.wisc.edu

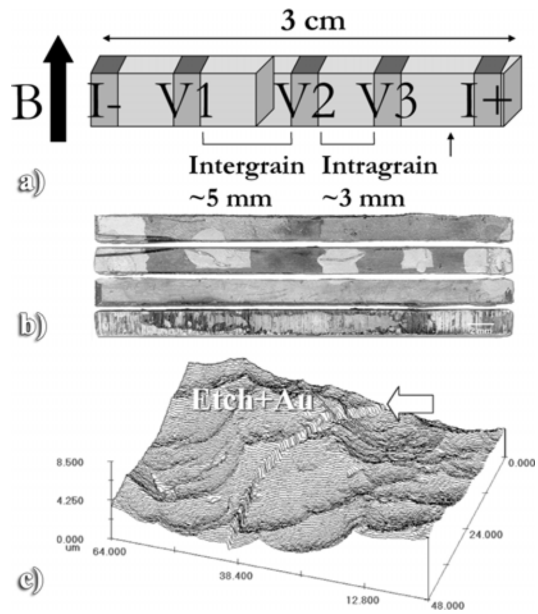


Figure 2: a) Schematic description of resistivity sample with b) images of each side of the actual sample. In c) we show a Laser Scanning Confocal Microscope image of the 1 µm thick Au contact layer.

could determine the locations of the boundaries after traversing the thickness of the slice. The 3-dimensional geometry was then determined using the "A 3D" [3] plugin for the ImageJ [4] image analysis package as well as the standalone program Reconstruct [5]. Height maps were determined using the Extended Depth of Field plugin [6] for ImageJ.

Magneto Optical Imaging

The magneto-optical (MO) technique is described in detail in [7]. It uses the strong Faraday effect in Y-Fe garnet to measure the vertical magnetic field component above a sample. The spatial resolution attained is ~5 µm when the garnet is placed directly on the face of the sample and fields of ~ 1 mT can be resolved. The sample is typically a 5x5 mm^2 rectangle (~2 mm thick). The continuous flow cryostat permitted sample temperatures down to ~6 K. An external solenoid can apply vertical fields up to ~120 mT to the sample. The field enhancement in the diamagnetic state is considerable, rising to a maximum of 2.7 at the mid-point of each edge as calculated using finite element analysis for a 5×5×2 mm^3 sample. The field enhancement pattern produces the characteristic "rooftop" field penetration pattern typified later in this paper by the behavior of the tri-crystal samples.

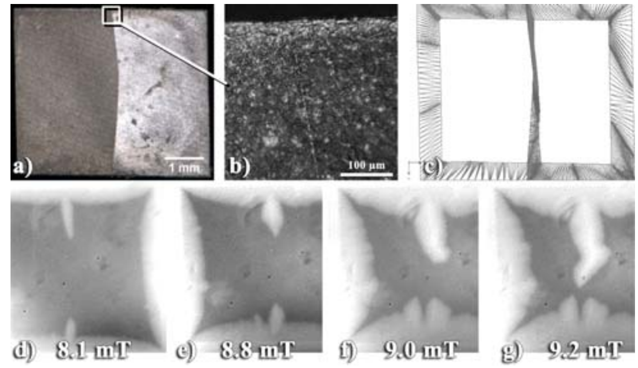


Figure 3: Bi-Crystal #1 a) with b) a height map detail from the area indicated, showing sub 5 µm topography at the grain boundary and c) a 3D model of the bi-crystal. A series of MO images, d)-g), at increasing field (T=5.6 K ZFC), shows premature flux penetration starting at a field of only 8.1 mT, << H_{c1}.

Inter-and Intra Grain Resistivity

For Inter and Intra-Grain resistivity measurements an additional bi-crystal bar was cut from Bi-Crystal#1. Three sides of the bar were cut with a diamond saw and the third was left with a heavily cold-worked band saw cut. Gold Voltage contacts were sputtered onto the surface opposite the band-saw cut after the surface had been Ar-ion etched. Regions between contacts were masked using Ag paint, which was subsequently removed (Figure 2).

RESULTS

Bi-crystals

The grain boundary in region #1 runs almost perpendicular to the surface of the slice (Figure 3b), and for the sample cut from this region there is clearly premature flux penetration along the grain boundary at fields much lower than H_{c1}. The sequence of magneto optical images in Figure 4 (ZFC, T=7.6 K) shows an initial penetration at 12 mT that is localized to the grain boundary (note the curvature matching the model surface in Figure 3b. There is virtually no topography at the grain boundary (Figure 3c).

This behavior contrasts with the second bi-crystal region, Figure 5, where the boundary is at a ~30° tilt to the surface and a classic rooftop MO image, Figure 5c, is observed up to our maximum field of 120 mT.

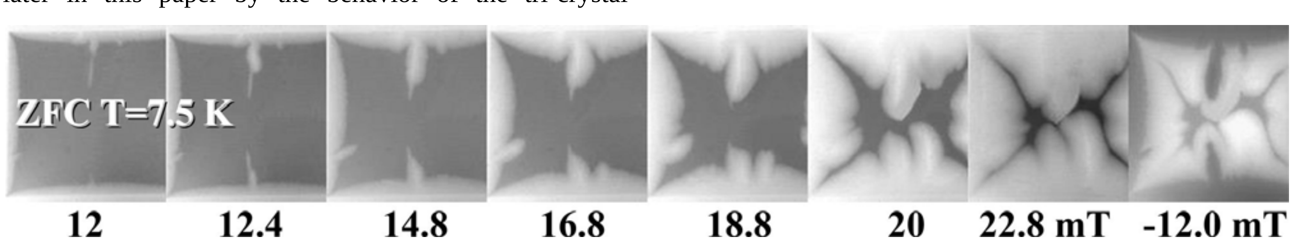
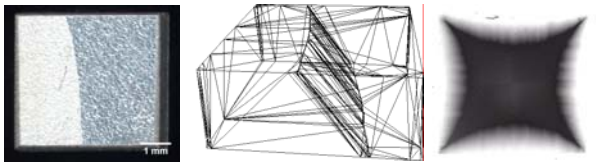


Figure 4: The reverse side of Bi-crystal #1 shown in Figure 3: Magneto Optical image sequence at 7.5 K showing flux penetration increasing along the grain boundary with increasing field.



a) b) c)

Figure 5: Bi-Crystal from region #6 a) with 3D model showing ~30° tilt to boundary b) and a classic rooftop magneto optical image for ZFC T=5.5 K and our maximum field, H=120 mT.

Tri-Crystals #4 and #5

In Tri-crystal #4 (Figure 6) there is a 13 µm step at the top left grain boundary, marked “A”, which produces a current inflection indicated by the dark lines in MO images d) and e). A small notch is observed in the 60 mT MO image where the “A” boundary meets the top edge of the sample as well as a larger notch in location “B”. Neither feature results in bulk penetration of the sample and the notches are not observed at our maximum field of 120 mT (Figure 6(e)).

For Tri-crystal region #5, Figure 7, we again see no premature flux penetration into the sample associated with the grain boundaries or topography.

Resistivity Measurements

The Au deposition technique was successful and we were able to measure a RRR across the grain boundary of 187, 11 % less than the intra-grain boundary value of 211. Superconducting state measurements showed lower critical current across the grain boundary as compared to the intragrain region.

SUMMARY

1. MO Imaging shows premature flux penetration at a perpendicular grain boundary in an as-received slice of Nb with residual cold work on surface. This behavior occurs at 8.1-20 mT (7.5 K), much lower than H_{c1}.
2. The premature flux penetration behavior observed in perpendicular boundary sample does not appear to be topologically related as much larger topological features do not to cause this behavior for the other samples.
3. Initial resistivity measurements indicate grain boundary weakness.
4. This work is continuing with the samples receiving further processing typical of cavity processing.

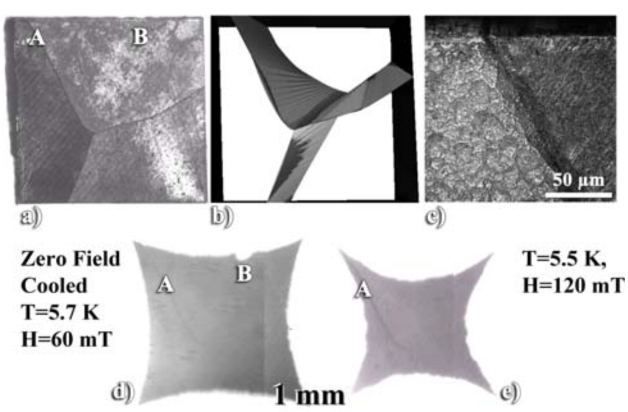


Figure 6: Tri-crystal #4 a) with 3D model b) and a height map of the top left hand grain boundary showing a 13 µm step. A typical "rooftop" magneto-optical image (d) is obtained at T=5.5 K, H=120 mT. In d) and e) a faint line, marked “A” indicating a current inflection is observed and in d) an edge notch, marked “B”, which does not extend into the sample with increasing field (e).

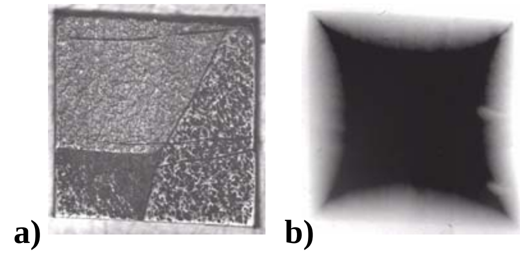


Figure 7: a) Light microscope image of Tri-crystal #5 compared with b) a magneto optical image after zero field cooling and then applying our maximum field of 120 mT at 5.3 K showing the classic rooftop pattern.

ACKNOWLEDGEMENTS

We would like to thank Peter Kneisel for providing the Nb slice, Bill Starch for metallographic preparation and Matt Feldman for sputtering the Au contacts.

REFERENCES

[1] Pierre Bauer et al., “An Investigation of the Properties of BCP Niobium for Superconducting RF Cavities,” "Pushing the Limits of RF Superconductivity" workshop, Argonne National Laboratory Report ANL-05/10, Ed. Kwang-Je Kim and Catherine Eyberger, pp 84-93, March 2005.

[2] P. Kneisel, G. Ciovati, G.R.Myneni, J. Sekutowicz, T. Carneiro, “Performance of Large Grain and Single Crystal Niobium Cavities,” presentation MoP09 at SRF05, Ithaca, NY, July 12, 2005.

[3] Albert Cardona, “A 3D editing plugin”, http://www.pensament.net/java/

[4] W.S. Rasband, ImageJ, National Institutes of Health, Bethesda, Maryland, USA, http://rsb.info.nih.gov/ij/, 1997-2005.

[5] J. C. Fiala, Reconstruct: a free editor for serial section microscopy. J Microscopy 218:52-61, 2005.

[6] Daniel Sage, “Extended Depth of Field” plugin by Biomedical Imaging Group, University of Lausanne: http://bigwww.epfl.ch/demo/edf/

[7] A. A. Polyanskii, D. M. Feldmann, and D. C. Larbalestier, "Magneto-Optical Characterization Techniques," “The Handbook on Superconducting Materials," Edited by David Cardwell and David Ginley, Institute of Physics UK , pp. 1551, 2003