Magnetic Investigation of High Purity Niobium for Superconducting RF Cavities

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Project Overview

How this project addresses one or more of the critical R&D needs of the ILC.

The goal of this project is to improve the understanding of the effects of chemical composition and surface topology (grain size, grain boundaries) in Niobium for superconducting RF applications and possibly to explain the so-called Q-slope observed in superconducting resonators.

Project Description

Background to these studies.

We have been the first to show that magneto-optical imaging, a tool that can image local field penetration into a superconductor, can be applied to the study of high purity Nb for SRF application. Magneto-optical (MO) imaging can be a valuable tool in the study of local perturbations in the superconducting behavior of Nb [1] Using MO we have previously shown that some Nb grain boundaries show preferential flux penetration at applied fields below penetration within the grains [2] This result was the first direct evidence of depressed superconductivity at a Nb grain boundary (GB). In that study we used large bi-crystal and tri-crystal samples cut from a transverse cross-sectional slice (RRR ~ 280, Ta content ~800 ppm) of one of two extremely large-grain billets fabricated for Jefferson Laboratory (TJNAF) by CBMM (Figure 1). TJNAF built 2 cavities from the center large grain of the ingot and both cavities performed well, one close to the theoretical critical field (~185 mT) while the other cavity approached an accelerating gradient of 45 MV/m, close to the highest ever measured [3]. This work indicated that:

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 that H_{c1} (Figure 1).
2. The premature flux penetration behavior observed in perpendicular boundary sample (Figure 2: Bi-Crystal Region 1) 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 for Bi-Crystal Region 1.

Work Being Performed In This Study

Large grain Nb slices provided by TJNAF continue to provide excellent source material for our grain boundary studies. The large grains provide opportunities for multiple samples of the same well characterized grain boundaries to be cut and prepared under different conditions. Our primary techniques are magneto optical imaging, light microscopy (we have been evaluating scanning laser confocal microscopy as a means of quickly and accurately characterizing surface topology) and transmission electron microscopy. We will also use grain-boundary critical current and grain boundary resistance (in the normal state) measurements to further support the magneto-optical measurements of the penetration of RF magnetic flux. The grain-boundary critical current measurements (as a function of external field) give an independent assessment of flux penetration into the grain boundaries. Normal state grain boundary resistivity measurements measure the electron scattering at the grain boundaries and therefore give further information about the

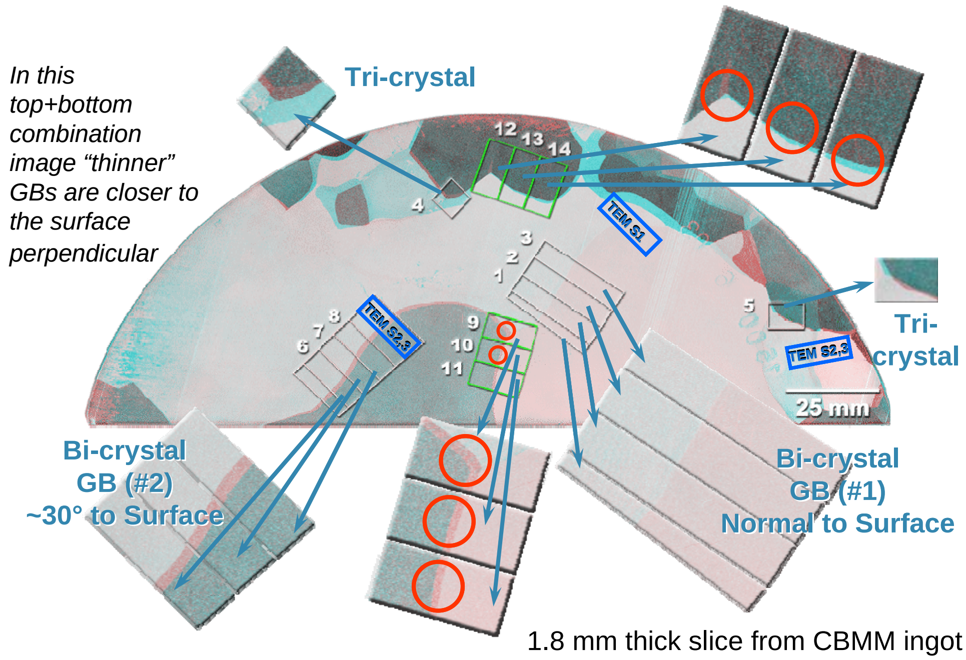


Figure 1: Sample Locations within TJNAF large-grain slice for Phases 1 (#1-8) and 2 (#9-14) as well as additional location for TEM samples.

grain boundary properties. These so-called transport measurements are best performed in bi-crystal samples (samples that consist of two single crystals separated by a grain boundary). The experiments then consist of the measurement of the current/voltage characteristic of each grain and the grain boundary.

Broader Impact

One of the primary goals of this work will be to introduce a graduate student, trained in metallurgy/materials science, to the field of superconducting cavities. The Applied Superconductivity Center has been very successful in training students who have gone on to make valuable contributions to superconducting materials fabrication in both industry and laboratories.

Progress Report

Describe the progress to date on your project, since April, 2006, or since the start of your project, whichever is earlier. This project was started 9/01/05

1.) Bi-crystal samples characterized in the as-received state were sent to FNAL for BCP removal of surface layers. 6 new samples cut from bi- and tri- crystal regions. These samples sent to FNAL for complete processing through standard schedule. Quantification of the surface topology has been carried out in conjunction with demonstrations on non-contact profilometers by Zygo Corporation (Figure 2) and ADE Corporation (Figure 3) as well as using scanning laser confocal imaging. The interferometry shows the dominant topological feature in these two samples to be the grain boundaries. A remarkable feature of both these instruments is how clear the small ~1-2 μm deep boundary groove is despite the low magnification and broad field of view (3 mm). In

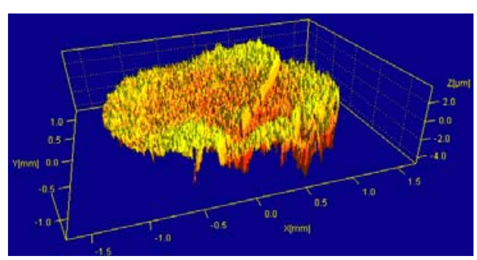


Figure 2: Low magnification interferometry produced height map of bi-crystal 3 mm EDM'ed disk. The height drop at the grain boundary is measured at 1.36 μm.

figure 3 we show a higher magnification image of a grain boundary in a fully processed sample. At this greater magnification additional topological detail is observed.

2.) MO imaging performed on samples following BCP procedure at FNAL (no heat treatment or secondary BCP)

In Figure 4 we compare the perpendicular boundary sample for Bi-Crystal Region 1 before BCP and after BCP. The light microscope images of the top surfaces (Figure 6 left) indicate a deeper grain boundary groove after BCP. Premature flux penetration is observed at the grain boundary in both conditions but the penetration is diverted in the BCP sample. No surface features were observed that correlate with the location of this diversion. This work was reported on in more detail at SRF'05 [4] and can be compared with processed sheet collaboration with FNAL reported in [5].

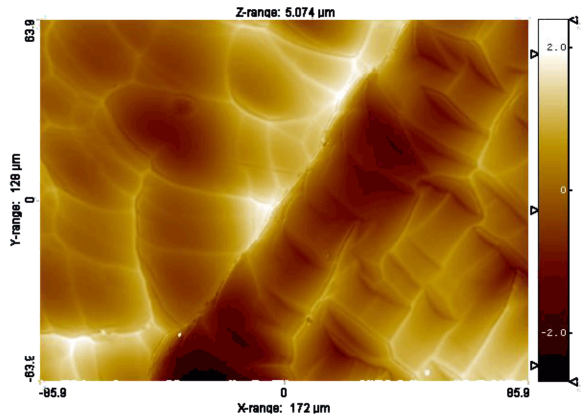


Figure 3: High magnification interferometry produced height map of grain boundary bi-crystal in 3 mm EDM'ed and BCP'ed disk.

Prior to the beginning of this work we had performed all our observation under perpendicular magnetic field, which is the conventional mode for this type of observation. We have now extended our MO investigations to in-plane applied field – which is closer to the conditions in a SRF cavity. In Figure 6 we show images obtained in this condition for the perpendicular bi-crystal sample. Now the feature that causes the flux diversion is again apparent – and yet this feature is not visible under conventional microscopy. Additional in-plane work is reported in ref. [6].

None of the tri-crystals and none of the bi-crystals with GBs oriented away from the perpendicular showed premature flux penetration. The bi-crystal sample from [4] with a grain boundary at 35° to surface, that did not show flux penetration at the grain boundary in our earlier MO examination and rotated and resliced so that the GB was now perpendicular to the top surface and parallel to the applied field. When the rotated GB was retested by MO and now the flux clearly penetrates at the grain boundary first. However the penetration in the 1.89 mm thick sample was asymmetric and not as clear as the earlier study. It appeared that there was sufficient twisting of the grain boundary through the specimen that the sensitivity was reduced. In order to reduce the magnitude of the geometric twist through the sample the sample was thinned to 0.3 mm thickness using a diamond saw. The results shown in Figure 5 now

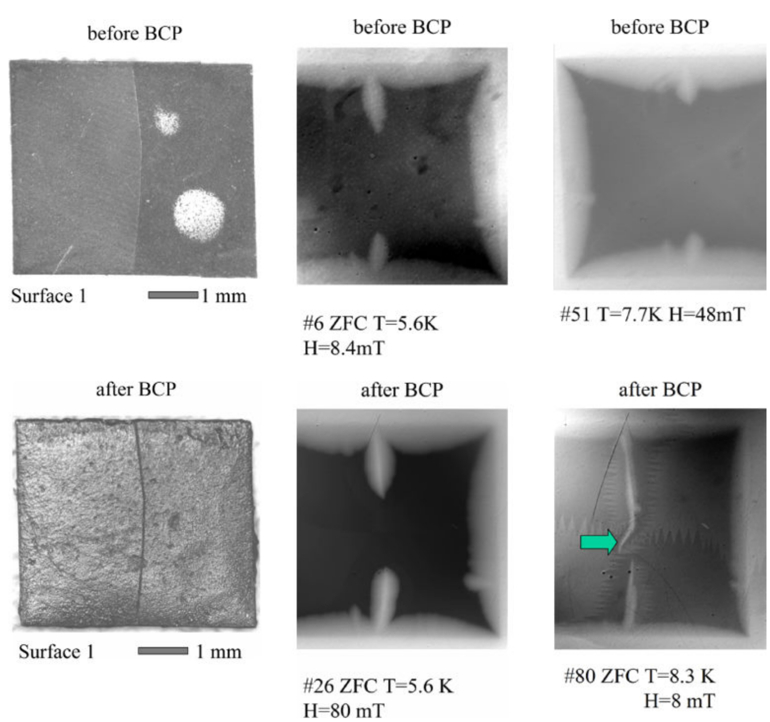


Figure 4: Perpendicular boundary sample for Bi-Crystal Region 1 (top) before BCP and (bottom) after BCP. The light microscope images of the top surfaces (left) indicate a deeper GB groove after BCP. Premature flux penetration is observed at the grain boundary in both conditions but the penetration is diverted in the BCP sample.

clearly show both flux penetration across the diagonal GB and a sharp perturbation in the circulating critical currents in the trapped field condition. This work is reported at the Single Crystal Niobium Technology Workshop in Araxá, Brazil, October 30-November 1 (2006).[7]

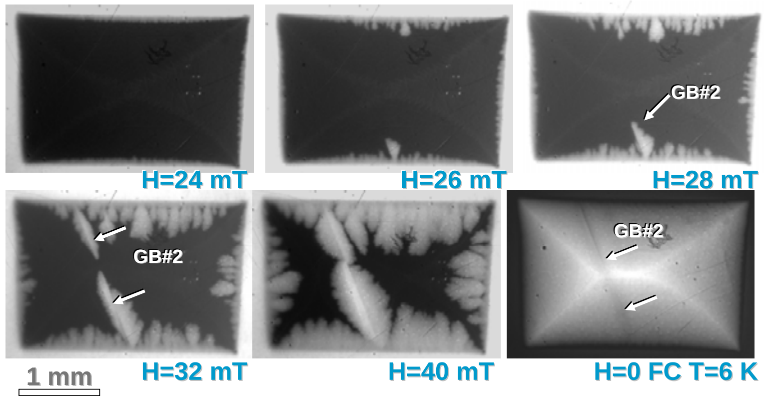


Figure 5: Summary of the MO investigation at 6 K after rotating the non-flux-penetrated GB oriented at 35° to the perpendicular and thinning it to 0.3 mm thickness. The flux clearly penetrates at the grain boundary across the entire sample width. A sharp perturbation in the circulating critical currents in the trapped field condition (FC) is also apparent.

3) TEM specimen preparation for investigation of grain boundary chemistry was initiated. SRF-grade Nb was found to be too soft for conventional grinding to thin sheet. A diamond saw was successfully used to cut 2 mm perpendicular bi-crystal slice samples into two ~1 mm thick samples., while EDM at FNAL was used to create 3 mm TEM disks for final polishing. Transmission Electron Microscopy, TEM, samples have been successfully prepared by a combination of careful mechanical polishing and final BCP. The final grit used for mechanical polishing is 0.3 µm alumina, which is continued until all visible scratches are removed. As can be seen from the TEM micrograph in Figure 7a) the depth of residual microstructural damage must still be at least ~5 µm at this stage as damage is still clearly visible if mechanical polishing is continued to a 10 µm foil thickness. If mechanical polishing is stopped at 20 µm, however, the mechanically damaged layer can be removed by BCP as shown in Figure 7b.

Importantly, the TEM specimen preparation work shows that a perfectly flat, mirror-like surface can be produced by this combination of careful mechanical polishing and BCP. EELS microchemical analysis was initiated but these studies indicated the need for high signal and higher resolution than was possible on the equipment available to us at the UW. We have applied to use the facilities of a much better analytical TEM at ORNL under a SHARE proposal to continue this work.

4) Gold contacts strips were sputtered onto to a BCP prepared bicrystal bar for resistivity measurements. However this set of Au contact had too high a level contact resistance. Work is currently under way to lower the contact resistance. A new design of transport sample is also under investigation, using the TEM specimen preparation technique to create a multi-use sample that can be used for MO, I_{ct} and TEM.

Final year Project Activities and Deliverables

1) Further develop TEM specimen preparation procedures and investigation of grain boundaries in these Nb samples.
2) Support this work with FESEM (OIM for grain boundary orientation) and light microscopy
3) Develop pogo-pin method of transport measurement for these samples, that uses a new specimen design that will also allow MO imaging and subsequent TEM imaging of the sample.
4) Quantitative estimates of the DC flux penetration field in the various samples as well as the grain boundary critical currents and the grain boundary normal state resistance.

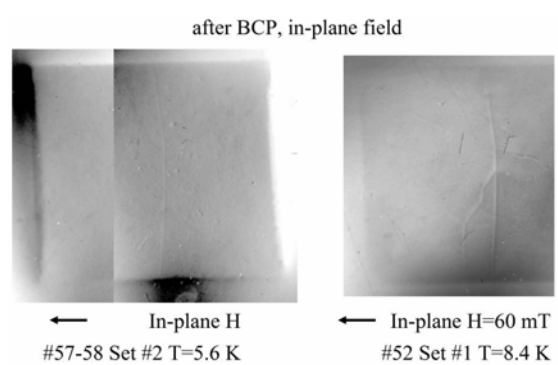
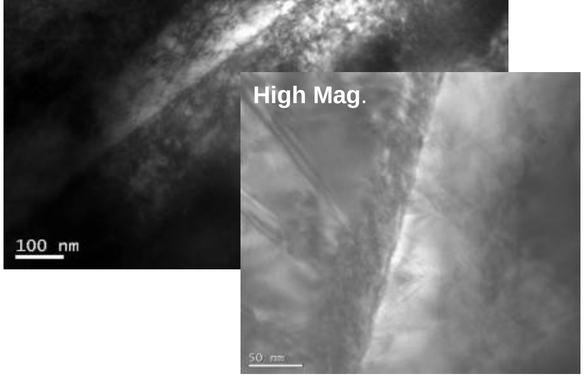
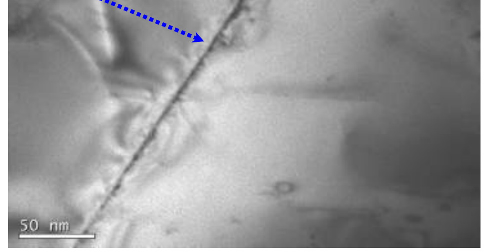


Figure 6: MO images of the perpendicular grain boundary sample with in-plane applied field. In this mode the flux diverting feature (not visible in conventional microscopy) is again apparent under H = 60 mT at 8.4 K.





TEM Bright Field Image

Figure 7: TEM Sample Preparation: TEM images of grain boundary on BCP'ed Nb from large grain TJNAF slice. In a) the sample had been ground to ~10 µm thick before BCP'ing to transparency. Dense dislocation networks from cold work of mechanical polishing can be seen. In b) the mechanical polishing was stopped at ~20 µm and the sample was BCP'ed to TEM transparency.

5) The results of these studies should be submitted for journal publication as well as presented to the SRF technology community.

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[7] P. J. Lee, et al., "Flux Penetration Into Grain Boundaries Large Grain Niobium Sheet For SRF Cavities: Angular Sensitivity," submitted for publication in Proc. Single Crystal Niobium Technology Workshop, Araxá, Brazil, Oct. 30-Nov. 1 (2006), AIP Conference Proceedings.

Additional Supplemental Reporting of Results

A more complete compilation of some of the data from this work than can be included here was reported at the Midwestern SRF Materials Research Meeting, held at the National Superconducting Cyclotron Laboratory, Lansing MI, March 17th 2006. This report and the other presentations can be found at:

http://ilc-dms.fnal.gov/Workgroups/SRFMaterials/SRFMaterialMeetings/SRFMat-2006-03-17/

And from the Midwest SRF Materials Meeting on Nov 14th 2006 at Argonne:

http://ilc-dms.fnal.gov/Workgroups/SRFMaterials/SRFMaterialMeetings/MidwestSRFMatMeetNov06/Presentations/folder_contents