

# Magnetic Investigation of High Purity Niobium for Superconducting RF Cavities

## Proposal for LCRD/UCLC 2005

### Classification (subsystem)

Material science on niobium for superconducting cavities of main linac

### Personnel and Institutions requesting funding

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### Collaborators

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### Project Overview

#### *Introduction*

Fermilab is developing superconducting RF cavities for a future high-energy electron-positron linear collider as the next international, large-scale high energy physics discovery machine [1]. More than one year ago Fermilab and the Applied Superconductivity Center (ASC) of the University of Wisconsin-Madison (UW) have joined forces to investigate high purity niobium used in the cavity fabrication. As a mission statement the work agreement between Fermilab and the ASC/UW states that the goal of the collaborative effort 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.”*

#### *Motivation*

The study of the magnetic properties of high purity niobium is extremely important for the advancement of the understanding of performance limitations of this material in the high gradient superconducting RF cavities for a future linear collider. During RF operation the high power RF fields penetrate only into a very thin layer on the surface of the cavity. This penetration layer is approximately 50 nm thick. It is currently believed that the penetration of only a few magnetic flux quanta into this layer will lead to a break-down of the superconducting state as a result of local heating due to the oscillation of the flux lines in the RF fields. The onset of flux penetration is believed to occur, in the best case, at a field close (or related) to the thermodynamic critical field (~180 mT in niobium). No niobium-based cavities have surpassed that surface field to this date. Grain boundaries and normal-conducting defects are potentially regions of suppressed superconductivity, allowing the penetration of magnetic flux lines even at low external magnetic fields. Therefore the assessment of the local shielding behavior of the superconductor in magnetic

fields is paramount for a better understanding of the performance limits of SRF cavities. The investigation of the effect of DC fields, such as with magneto-optics is the first step in this direction. The magneto-optical technique together with transport measurements can be used to quantify the possible suppression of superconductivity in grain boundaries or other surface defects. These two measurement techniques will be described next.

#### *Magneto-Optics and Transport Measurements at the ASC/UW*

We have been the first to show that the magneto-optical technique, a remarkable tool that can image local field penetration into a superconductor, can be applied to the study of high purity Nb for SRF application. The magneto-optical (MO) technique is described in detail in [3]. It uses the strong Faraday effect in YFe garnet to measure the vertical magnetic field component above a sample, in this case of superconducting material. The spatial resolution attained is ~5-10 microns. The garnet is placed on the face of the sample to retain the highest possible sensitivity. The technique is able to resolve fields of the order of 1 mT. The sample is typically a 5x5 mm<sup>2</sup> rectangle. Via indirect cooling with a cold finger containing liquid helium the sample temperature was held at temperatures between 5.6 and 7 K. An external solenoid is used to apply a vertical magnetic field on the sample.

We propose to 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 grain boundary properties. These so-called transport measurements are best performed in bi-crystal samples, that is samples which 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.

#### *ASC/UW and Fermilab Collaboration Overview*

The ASC/UW and Fermilab have agreed to collaborate on the study of the magnetic properties of bulk niobium for SRF cavities using magneto-optics, transport measurements, XPS and microscopy. According to the agreement, Fermilab will be responsible to provide samples for the studies conducted at the ASC/UW. Both parties share the efforts related to analysis and publication.

The following project goals have been 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;

To achieve the above-defined goals a series of samples need to be provided to the ASC/UW, which are optimized for the use of various measurement techniques, such as magneto-optics and

transport measurements, without compromising the chemical condition of the surface. In particular we would like to test samples of the following type:

- 1) samples representative of the e-beam weld region in the cavities (MO, SEM and XPS);
- 2) samples representative of the non-weld region in the cavities (MO, SEM and XPS);
- 3) single crystal samples to study grain boundary free material (MO, SEM and XPS);
- 4) bi-crystal samples for transport measurements of the grain boundary properties (transport, SEM and XPS);
- 5) samples obtained via sputtering of niobium on a copper substrate to study very small grain material (MO, SEM and XPS);

The samples should be prepared with different degrees of completeness (e.g. mechanically deformed and deeply etched vs mechanically deformed, deeply etched and heat treated, ... etc) such as to allow measurement of the evolution of the magnetic and transport properties during the fabrication of the cavities. The polishing variants, i.e. using chemical etching and electro-polishing, should also be differentiated. Furthermore this series of experiments should also include the low temperature bake (~120C, 50 hrs) that was recently shown to improve cavity performance significantly.

### **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.

### **Results of Prior Work**

Although we have not been previously funded under LCRD we have achieved the following with funds made available by FNAL:

- 1) SEM and optical microscopy analysis was performed at the ASC/UW on large and small grain samples provided by Fermilab. Large grain samples (produced both by welding and high temperature heat treatment) showed deep (~1  $\mu\text{m}$ ) groves at the grain boundaries. All BCP etched samples showed surface roughness associated with differing rates of etching from grain to grain resulting in localized height variations of up to 10  $\mu\text{m}$ . New, computer software based techniques were successfully employed to make the quantitative topological estimates quoted above.
- 2) XPS analysis revealed no direct evidence of sub-oxides (sputtering rate 0.3 nm/step, penetration depth of X-ray photons: ~4 nm) in the case of the samples that were surface treated in the same way as cavities. After re-oxidation in air for 5 minutes following a complete sputtering cycle, however, evidence of sub-oxides was found.
- 3) Magneto-optical measurements revealed strong variations in the magnetic shielding between large and small grain samples. A large grain weld sample as well as a sample, which was heat-treated at high temperature to grow large grains, showed evidence of flux penetration deep into the sample, indicating a breakdown of shielding in the grain boundaries (more in [2]).
- 4) Successful measurements of magnetization (Fermilab and ASC/UW) and specific heat (ASC/UW) have been performed on some samples.

Most of these results were presented at the “Pushing the Limits of RF Superconductivity” workshop at the Argonne National Laboratory in Sept. 2004 and at the Applied Superconductivity conference in Jacksonville in Oct. 2004 [2]. Our presentations at ANL can be viewed at the workshop website and a paper will be published in the proceedings. The measurements mentioned above raised several questions, which need to be resolved. Among them is the question of what caused the flux penetration into the large grain samples and whether this condition is present in e.g. the e-beam welds of SRF cavities, which similarly have very large grains. The collaboration between Fermilab and the ASC/UW has already provided new and interesting insights into the properties of chemically polished Nb for SRF cavities in magnetic fields and provides a model for University-National Laboratory collaboration in this field.

### **Facilities, Equipment and Other Resources**

At the University of Wisconsin-Madison Applied Superconductivity Center we have comprehensive facilities for superconducting materials testing and microstructural characterization (including adjacent Materials Science Center). A comprehensive list can be found at <http://www.asc.wisc.edu/facility/facility.htm>. For electromagnetic testing we have 14-16 T and 15-17 T Oxford Instruments magnets with Variable Temperature operation from 2 to 120 K, a 5.5 T Quantum Design SQUID magnetometer, an Oxford Instruments 14 T Vibrating Sample Magnetometer with Cantilever Torque magnetometer, a Quantum Design 9 T Physical Property Measurement System with DC and AC susceptibility and specific heat capability. We also have a Very-Low-Field Shielded Test Facility and extensive high-sensitivity electronics permitting nanovolt DC measurements.

### **Activities and Deliverables for this Proposed Work**

Under this proposal we plan to recruit a graduate student to:

- 1) start transport measurements to derive the grain-boundary critical currents as well as the grain boundary normal state resistivity;
- 2) support this work with SEM (OIM for grain boundary orientation), XPS and light microscopy

The list of samples to be prepared by Fermilab and submitted to tests at the ASC/UW is outlined in Table 1. The preparation and testing of all the samples discussed in this table is a multi-year task.

**Table 1: Run-plan for Fermilab/ASC-UW studies on niobium for SRF cavities. The roster of samples includes specimen prepared to different degrees of completeness. Also for each preparation condition there should be samples representative of the two different cavity areas (the weld and non-weld area) and/or the two main polishing techniques: BCP and EP (where BCP refers to “buffered chemical polishing” and EP stands for “electro-polishing”).**

Sample normal/weld/ sputtered/single-crystal	as received	deep drawn	100 $\mu$ m etch (BCP/EP)	heat treatment (750C/5hrs)	20 $\mu$ m etch (BCP/EP)	Low temp bake (120C/50hrs)
1	X					
2	X	X	X			
3	X	X	X	X		
4	X	X	X	X	X	
5	X	X	X	X	X	X

The deliverables for the next years are expected to be 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. The results of these studies should be published in a renowned journal such as Physical Review as well as in the important meetings of the SRF technology community.

### Budget Justification

The activities outlined above will involve Fermilab staff members, whose salaries are not included in the budget request below. The budget request only includes salaries for the ASC/UW staff members participating in this study. The quoted amount includes a

3% of annual time Peter Lee to co-ordinate work at UW;

3% research fraction of Anatolii Polyanskii to train student for the magneto-optical measurements;

3% research fraction of A. Squitieri to train student for the transport measurements;

The following budgetary estimate is in \$.

**Table 2: Budgetary estimate for ASC/UW-Fermilab collaboration on the study of high purity niobium for SRF cavities.**

Item	FY 2005	FY2006	FY2007
Other professionals	\$9,164	\$9,530	\$9,530
Graduate Students	\$22,253	\$22,253	\$22,253
Total Salaries (incl. fringe benefits)	\$40,319	\$41,927	\$43,608
Equipment	0	0	0
Travel	\$500	\$500	\$500
Materials and Supplies	\$1000	\$1000	\$1000
Sample preparation	\$200	\$200	\$200
Total direct costs	\$47,583	\$49,412	\$51,326
Total indirect costs	\$19,119	\$19,850	\$20,615
<b>Total direct and indirect costs</b>	<b>\$66,702</b>	<b>\$69,262</b>	<b>\$71,941</b>

## References

- [1] L. Bellantoni, N. Solyak, I. Gonin, T. Berenc, H. Edwards, M. Foley, N. Khabiboulline, D. Mitchell “*Test Results of the 3.9 GHz Cavity at Fermilab*”, presented at the LINAC 2004 Conference, Luebeck, Germany, Sept. 2004.
- [2] P. Lee, P. Bauer, A. Polyanskii, C. Boffo, L. Bellantoni, H. Edwards, A. Gurevich, M. Jewell, D. Larbalestier, G. Perkins, A. Squitieri “*An Investigation of the Properties of BCP Niobium for Superconducting RF Cavities*”, submitted to the proceedings of the “Pushing the Limits of RF Superconductivity” workshop, Argonne National Lab, Sept 2004.
- [3] 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.

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#### CURRENT POSITION

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1994 - Senior Scientist in the Superconducting Materials Research Group of the Applied Superconductivity Center at the University of Wisconsin-Madison. Focus: Microanalytical and microstructural evaluation by transmission electron microscopy, Scanning Auger, field emission SEM and electron microprobes of superconducting composites Low Temperature Superconductors. Currently co-principal investigator with Prof. David C. Larbalestier for grants from the US Dept. of Energy Division of High Energy Physics (~\$530,000 per annum), DOE-Office of Fusion Energy (~\$100,000 per annum) and Fermi National Accelerator Laboratory (\$75,000 to explore SRF Cavity work). Our analytical expertise has also attracted additional funding from industry for service work via the DOE-SBIR program.

#### COMMITTEE MEMBERSHIPS

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Conductor Advisory Committee for High Energy Physics High Field Magnets (1999 - present).  
Applied Superconductivity Conference, elected board member since 2002.  
ICMC, elected board member 2004 (term expires 2011)

#### RECENT INVITED TALKS AND SEMINARS

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"Advances in Superconducting Materials for Accelerator Magnet Application," 2003 Particle Accelerator Conference, Portland, OR, May 13<sup>th</sup> 2003.

"Microstructural and Microchemical Homogeneity for High Critical Current Density in Nb<sub>3</sub>Sn," 1st International Workshop on Progress in Nb Based Superconductors, Tsukuba, Japan February 2-3, 2004.

#### PUBLICATIONS

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**Patent:** "Artificial and Multiple Phase Micro-Electropolishing," US Patent Number 5,354,437, Oct. 11, 1994

**Books:** "*Composite Superconductors*," ed. K. Osamura, T. Matsushita, P. J. Lee, S. Ochiai, pub. Marcel Dekker, New York, 1994.

"Engineering Superconductivity, ed. Peter J. Lee, Wiley-Interscience, New York, 2001

#### **Book Contributions: 21, most recently:**

"Handbook of Superconducting Materials," Chapter 3.3: Conductor Processing of Low- $T_c$  Materials: The Alloy Nb-Ti, Lance Cooley, Peter Lee, and David Larbalestier. A Pre-publication draft is available for download from [http://128.104.186.21/asc/pdf\\_papers/630.pdf](http://128.104.186.21/asc/pdf_papers/630.pdf)

"Superconductor: WIRES AND CABLES: MATERIALS AND PROCESSES," Peter J. Lee, in the ENCYCLOPEDIA OF MATERIALS: SCIENCE AND TECHNOLOGY - Update, Edited by K. H. J. Buschow, R. W. Cahn, M. C. Flemings, P. Veysiere, E. J. Kramer, S. Mahajan. Published on-line by Elsevier at the EMSAT website in 2003. To appear in the full electronic update of EMSAT in 2004.

#### **Refereed Articles: 69 articles, most recently:**

- 1 "Development Of Point-Array APC Nano-Structures In Nb-Ti Based Superconducting Strand," P. J. Lee, L. R. Motowidlo, M. K. Rudziak and T. Wong. Advances in Cryogenic Engineering, 50B, pp. 314-321, 2004. [http://128.104.186.21/asc/pdf\\_papers/780.pdf](http://128.104.186.21/asc/pdf_papers/780.pdf)
- 2 "Flux Pinning Properties In Nb-Ti Composites Having Nb And Ti Mixed Artificial Pins," O. Miura, D. Ito, P. J. Lee, and D. C. Larbalestier, Advances in Cryogenic Engineering, 50B, pp. 307-313, 2004. [http://128.104.186.21/asc/pdf\\_papers/782.pdf](http://128.104.186.21/asc/pdf_papers/782.pdf)
- 3 "Simulations of the effects of tin composition gradients on the superconducting properties of Nb<sub>3</sub>Sn conductors," L. D. Cooley, C. M. Fischer, P. J. Lee, and D. C. Larbalestier, accepted for publication in *J. Applied Physics* (August 1st) 2004.

**Further information available at: <http://homepages.cae.wisc.edu/~plee>**