Project Title:	**Coordinated Characterization of Coated Conductors**
Organization(s):	**Argonne National Laboratory**
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FY 2004 Funding:	$520 K

Project Purpose and FY 2004 Objectives: The purpose of this task is to develop, integrate, and implement a coordinated set of characterization methods to evaluate superconductivity in coated conductors and pinpoint the causes of degraded performance. Some methods examine intermediate stages of fabrication in a manner compatible with further high-temperature processing. These characterizations include measurements of local supercurrent transport, phase composition, microstructure, and epitaxy quality for YBCO coated conductors that range in size up to multi-meter-length tapes and that embrace the entire tape embodiment (substrate through cap layer). Our FY 2004 objectives included: (1) the establishment of Raman spectroscopy as an on-line process monitoring tool, (2) the integration of magneto-optical imaging (MOI) and Raman microscopy with transmission electron microscopy (TEM) assisted by focused ion beam (FIB) sample sectioning, (3) the extension of this integrated characterization package to long-length coated conductors supplied by our industrial partners, and (4) the development of seamless methods to monitor properties during/after sequential high-temperature treatments to improve superconductivity, e.g., optimizing oxygenation of coated conductors.

FY 2004 Performance and FY 2005 Plans: Performance in FY 2004: In collaboration with SuperPower, Inc. and Kaiser Optical Systems, Inc., we conducted design, concept development, and qualification testing of a fiber optic-based Raman microprobe embodiment that will soon be installed on one of SuperPower's coated conductor processing stands. We performed coordinated MOI, Raman microscopy, and electron microscopy analyses of selected high performance and low performance regions of meter-long YBCO CC tapes supplied by SuperPower. The insight gained from these complementary techniques is significantly enhanced by our use of FIB-based TEM specimen preparation. This integration elucidated several defects that could inhibit performance. In addition, we have made progress in our coordinated characterization methodology by developing new data collection and specimen preparation techniques. In collaboration with American Superconductor (AMSC), we used Raman microscopy to probe the details of phase evolution in the early stages of TFA precursor transformation. Using a novel scheme to reversibly change the oxygen concentration, we demonstrate that, below 77 K, the highest J_c values in coated conductors from SuperPower as well as ISD from ANL occur at the highest (overdoped) oxygen concentration.

Plans for FY 2005: Our primary focus of effort in FY 2005 will be to apply our coordinated characterization of coated conductor approach to state-of-the-art coated conductor segments supplied by our CRADA partners—AMSC and SuperPower. A restoration of some of or the entire FY2004 funding cut is needed to fulfill our commitments to these CRADA partners. To the maximum extent possible we will conduct these analyses either *in situ* (e.g., using Raman spectroscopy) or in a manner that is compatible with further high-temperature processing (e.g., by "nondestructive" MOI and FIB-based excision of "micro specimens"). In the Superpower sample, we identified a defect that has a significant impact on performance. However, our coordinated characterization also reveals several other types of defects that we need to further evaluate by high-resolution SEM and TEM. We will also isolate specific defect structures to evaluate the local J_c by magnetization methods. To improve J_c, we will attempt greater oxygenation of coated conductors using, e.g., ozone.

We will continue to work with SuperPower to follow through on the implementation of the fiber-optic Raman microprobe being installed at their facilities in Schenectady and with AMSC on the optimization of their precursor conversion process. We want to carry out more comprehensive TEM studies of precursor conversion of YBCO evolution to complement on-going Raman studies. Hopefully, FY 2005 funding levels will also permit us to reengage with ORNL on the BaF_2 process optimization work that we started in FY 2003.

FY2004 Results: Reduced funding in FY 2004 had a significant impact on the breadth of our characterization activities and on the number of specimens we could meaningfully examine. In particular, our productive and rewarding collaborations with national laboratory partners were greatly curtailed. We concentrated our efforts on meeting the characterization needs of our two principal industrial partners, AMSC and SuperPower. Segments of CC tapes supplied by SuperPower, along with comprehensive local J_c data, were subjected to the integrated MOI/Raman/FIB-SEM/TEM methodology. MOI was used to explore the differences in high-, mid-, and lower-I_c regions of a long-length tape, showing that local defects influence current flow in each case. Raman confirmed that the quality of the superconductor was degraded in the defected regions identified by MOI. TEM in those regions, aided by FIB-based specimen preparation, revealed that interactions at the buffer layer play a key role in performance. Detailed studies using energy-filtered TEM and STEM-based spectroscopy show that these interactions can be very subtle, reinforcing the need for coordinated characterization to identify such regions. We studied the effects of oxygen concentration on the temperature and magnetic field dependence of J_C in coated conductors from SuperPower as well as ISD from ANL using a novel oxygenation scheme. We found that the most over-doped state always gave the highest J_c. In studies carried out in conjunction with AMSC, we performed Raman microscopy measurements on TFA precursor (and compositionally modified TFA precursor) specimens that were quenched during ramp up to the precursor conversion temperature. We were also able to detect the buffer layers during the early stages of precursor transformation.

Technology Integration: In FY04 we enhanced our interaction with Superpower and with the Electron Microscopy Center at Argonne to address a significant performance issue for long-length conductors. We implemented our complete coordinated characterization methodology, utilizing MOI, Raman, FIB-SEM, and TEM, to identify one specific defect that influences superconducting performance. The detailed microstructural information we provided also yielded new insight into the YBCO growth process on this SuperPower coated conductor architecture.

During FY 2004 our efforts to establish Raman spectroscopy as a viable/useful on-line diagnostic procedure for monitoring the progress of YBCO phase formation and the quality of the CC films were taken to yet another level with the design/development/testing and placement of a Raman probe on one of SuperPower's YBCO CC tape production lines. This accomplishment involved working closely with both SuperPower and Kaiser Optical Systems, and built upon our FY 2003 accomplishments which showed that meaningful Raman interrogations could be performed on moving tapes and that commercially available Raman hardware is fully adaptable for application at long-length coated conductor manufacturing facilities. We also continued to work with AMSC on the optimization of their TFA precursor conversion process, but at a reduced level of effort compared to FY 2003. Other Raman microscopy-based research on phase evolution in meter-length BaF_2-precursor tapes that was being performed in collaboration with ORNL, had to be curtailed due to the large funding reductions experienced in the Argonne program in FY 2004.