Project Title:	**Coordinated Characterization of Coated Conductors**
Organization(s):	**Argonne National Laboratory**
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FY 2003 Funding:	$750 K

Project Purpose and FY 2003 Objectives: The purpose of this task is to develop and implement a coordinated set of characterizations for coated conductors that can be done in a manner compatible with further high-temperature processing. These characterizations include measurements of supercurrent transport, phase composition, microstructure, and epitaxy quality for YBCO coated conductors that range in size up to multi-meter-length tapes. Our FY 2003 objectives included: (1) extension of Raman microscopy to meter-length samples and to on-line applications and (2) integration of Raman with transmission electron microscopy (TEM). During the year we added several new objectives: (3) extension of magneto-optical imaging (MOI) to commercial coated conductor lengths, (4) integration of MOI with transport, and (5) development of seamless methods to monitor properties during/after sequential high-temperature treatments to improve superconductivity, e.g., optimizing oxygenation of coated conductors. The prior task involving tape joining was dropped.

FY 2003 Performance and FY 2004 Plans: Performance in FY 2003: We expanded our Raman microscopy capabilities to include examinations of meter-length coated conductor tapes (including moving tapes) by adaptation of reel-to-reel (R2R) techniques. We performed electron microscopy on selected regions of meter-long tapes to complement the Raman results. The integration of these techniques provides new insight into reaction sequences during YBCO formation. We extended our MOI capability to handle coated conductor segments up to 10 cm long. We developed a contact-free magnetization method to obtain transport data on coated conductors by patterning them into rings--the accuracy of J_c was confirmed by direct current-voltage data for the *same* samples. Coupling these with MOI on rings containing a 10^o artificial grain boundary (GB), we determined *both* the GB and epitaxial film J_c. By oxygenating GBs in YBCO suitcases, we were able to confirm the improvement in J_c after oxygen *overdoping* of "artificial" *thin-film* GBs. We used a variant of position resolved electron diffraction to track the evolution of texture in template layers produced by physical vapor deposition. These studies play an important role in process optimization.

Plans for FY 2004: Our major theme will be to develop and implement coordinated characterizations of coated conductors that can be done *in situ* and/or in a manner that is compatible with further high-temperature processing. We plan to construct a transportable fiber-optic-based Raman microprobe, suitable for testing at coated conductor fabrication facilities. For testing the effects of oxygen overdoping on coated conductors patterned into rings, we will develop dry-etching methods to avoid degradation during subsequent high-temperature treatment. Our combination of techniques (Raman, MOI, and transport) will be used to identify weak regions for subsequent comprehensive TEM studies aimed at pinpointing deleterious microstructural features. A goal for microstructure characterization is the incorporation of site-specific specimen preparation utilizing focused ion beam techniques that will allow exceptional integration of these characterization approaches. Significantly, this approach improves compatibility of TEM studies with further processing of conductors by minimizing the destructive nature of specimen preparation and increases our capacity to apply this integrated approach to characterization to a wider range of issues in the wire development program. In a further coordination of characterization, we will develop new approaches for local measurement of properties utilizing the same site-selectivity as well.

FY2003 Results: In FY 2003 we extended the Raman microscopy examination methodology to meter-plus-length CC tapes. A detailed R2R Raman investigation, coupled with TEM examinations of key regions, was performed on a tape provided by American Superconductor (AMSC). The provided tape was processed using a "quenched tape" methodology developed by Lee et al. at ORNL, wherein the tape is slowly rolled into a preheated furnace and then rapidly reeled back out of the furnace to create a graded Y-Ba-Cu-O phase assemblage along the length of the tape. The Raman results track YBCO phase evolution from the amorphous precursor stage, through early YBCO phase formation, optimum YBCO development, and the over-heat-treated state. The TEM results confirm the presence of intermediate and impurity phases, such as CuO, BaF_2, Ba-Cu-O compounds, and $BaCeO_3$ detected in the Raman scans, as well as important information about their location and morphology. The findings of other Raman microscopy examinations on samples provided by IGC-SuperPower (including a 12-meter CC tape), AMSC, and Brookhaven National Laboratory will be summarized as part of the review presentation. We reversibly changed the oxygen concentration of $YBa_2Cu_3O_x$ thin films from under- to over-doped by varying the oxygenation temperature between 500°C and 300°C in a suitcase of suitably prepared YBCO bulk material. We applied this procedure to artificial bi-crystal grain boundaries in YBCO films and used our contact-free method to show that J_c continues to increase even past optimum doping. Enhancements up to ten-fold are seen for higher angle boundaries, e.g., 24°. In competition with the enhancement of J_c by oxygen overdoping, we find that the YBCO film can deteriorate after multiple processing steps, presumable due to chemical reaction at high temperatures with residues from the photolithography. This competition is more important for low angle boundaries, because the GB J_c is close to that of 'bulk' YBCO. We plan to avoid contamination by ion milling the thin-film rings. We have coordinated X-ray measurements of texture development with localized measurement using position resolved electron diffraction in selected area mode. Such localized measurements yield new insight into texture evolution and surface texture. In order to address these issues in thin layers, we have begun to develop a STEM-based approach.

Technology Integration: During FY 2003 we continued our efforts to establish Raman spectroscopy as a viable/useful on-line diagnostic procedure for monitoring the progress of the YBCO phase formation process and the quality of the resulting film. Working with Kaiser Optical, Inc. we developed a design for an on-line Raman probe and conducted some preliminary tests of the concept that were highly encouraging. In particular, we showed that Raman interrogation could be performed on moving tapes and that commercially available remote detection hardware is fully adaptable for application at a long-length coated conductor manufacturing facility. [Other Raman microscopy analyses on meter-length BaF_2-precursor tapes, performed in collaboration with ORNL, will be described in a separate Peer Review presentation.] The combined characterizations (transport and MOI) have begun looking at sections of meter-long tapes from IGC-SuperPower. Through a close interaction with the Electron Microscopy Center at Argonne, we have augmented our capabilities for the coordination of microstructure characterization with other crucial characterizations. This effort is intended to address the perennial need for more microstructure characterization by all partners in the wire development program, with initial studies directed towards texture development with IGC-SuperPower and phase evolution with AMSC.