
Project Title:	Ex-Situ Processing of YBCO Precursors
Organization(s):	Oak Ridge National Laboratory
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Project Purpose and FY 2004 Objectives: The purpose of this project is to understand and explore means to fabricate high performance YBCO by the ex-situ technique. Our primary objectives for FY 2004 are:

- 1) Investigate reel-to-reel (R2R) Pulsed Electron-Beam Deposition (PED) of YBCO precursors.
- 2) Develop a R2R slot die coater for solution precursors.
- 3) Explore YBCO conversion characteristics and performance of various types of precursors.
- 4) Investigate differences in solution and physical vapor deposition (PVD) precursors responsible for different reaction rates, and increase the reaction rate of PVD precursors.
- 5) Identify pinning centers and processing parameters responsible for different field dependencies in samples converted in different systems.
- 6) Add and develop both reduced- and low-pressure R2R capabilities for precursor conversion.
- 7) Process double-sided precursors.

FY 2004 Performance and FY 2005 Plans: In our quest to explore new industrially scalable options for the deposition of precursors, we have implemented R2R systems for PED and for slot-die solution deposition (April 04). In the PED process, significant initial stability issues were overcome to yield precursors exceeding 1- μ m thickness in meter lengths. In the area of solution precursors, we have developed a hybrid MOD precursor capable of sustaining high J_c 's. Meter-lengths of MOD precursors (>1- μ m thickness) were deposited using our slot die coater. Due to a budget reduction, however, we were not able to develop and upgrade our R2R conversion systems to enable reduced- and low-pressure processing.

Significant progress in increasing the conversion rate of PVD precursors has been made. Modified deposition conditions for e-beam evaporated precursors resulted in a ten-fold increase in c-axis YBCO conversion speed over the previous e-beam precursor. In addition, a similar increase in conversion rate has also been found for PED precursors. In the area of conductor performance, the strong magnetic-field dependency of I_c found in our R2R "atmospheric" system was eliminated by adjustments to the dry annealing temperature profile. This finding has significant implication on tape manufacturing where a tendency may exist to over-anneal the product to ensure complete conversion and spatial uniformity. In addition, double-sided buffers in short lengths have been fabricated, and some potential issues associated with buffer architecture and deposition have been identified. Various YBCO precursors have been converted on such substrates, and epitaxial films have been obtained.

Our FY 2005 plans focus on:

- 1) Continuing efforts to understand the effects of pressure on ex-situ conversion rate of various precursors.
- 2) Developing and installing a multi-source PED system to increase deposition rate of R2R PED precursors.
- 3) Increasing the conversion rate and performance of thicker PVD precursors.
- 4) Evaluating the feasibility of enhancing intra-granular J_c and pinning through nanoparticle inclusions using scalable ex-situ methods.
- 5) Obtaining high I_c/cm and low ac losses through conductor design such as double-sided and filamentary coated conductors

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- 6) Evaluating issues related to the conversion and performance of high volumetric large-area conductors
 - 7) Applying our ex-situ capabilities to aid in the continuing development of RABiTS, which is aimed at new alloy substrates, alternative buffers, and improved texture.
 - 8) Working collaboratively with ANL and NIST on phase development of various precursors using ANL's R2R Raman Spectroscopy, NIST's in-situ XRD expertise, and ORNL's R2R X-ray diffraction capabilities.

FY 2004 Results: In the past year, we have been able to modify the deposition conditions of e-beam "BaF₂" precursors to yield higher reaction rates. Under previous deposition conditions, typical rates ranged from 0.6 Å/s to 2.5 Å/s, depending on the conversion system, with higher rates resulting in the development of random YBCO with few cube-textured grains. In contrast, our modified precursors sustain reaction rates as high as 10 Å/s with the formation of little or no random YBCO, and J_c's >1 MA/cm² for ~1-µm-thick films.

PED is a potentially simple and stable method for PVD precursor deposition. Motivation for the exploration of this process comes from the initial observation that laser-ablated precursors on short-length samples yield J_c >1 MA/cm² (1.4-µm thickness). To cost-effectively utilize the advantage of ablation from a single source, our PED system was modified for R2R operation. Initial trials revealed two potential problems: instability in source operation (missed pulses), and substantial decrease in the deposition rate per pulse with time. By adjusting the operation parameters, the instability limit was increased from tens of thousands of successively fired pulses to several million. In addition, studies of the relationships between target mounting, growth rates, and stoichiometry of transfer showed the importance of charge redistribution during precursor deposition, and allowed us to reproducibly increase the deposition rate. Short PED precursors (~1-µm thick) have been converted under various pressures, resulting in YBCO films with J_c's greater than 1 MA/cm² (~120 A/cm-width). Furthermore, similar to the modified e-beam precursors, these PED precursors were found to be able to react at moderately fast rates.

A hybrid MOD precursor, containing both fluorine and nonfluorine containing phases has been developed. J_c's in excess of 2 MA/cm² on 0.6-µm spin-coated short samples have been achieved. Like other solution precursors, this hybrid MOD precursor was found to be capable of being converted at higher rates than PVD precursors. Precursor films >1-µm-thick have been deposited using our R2R slot die coater, and the precursors are presently being evaluated using our various conversion systems.

We have previously shown that samples converted in our R2R atmospheric-pressure furnace consistently resulted in films with strong magnetic field dependency, where the J_c drops to ~15% of its self-field value at 0.5 T. In contrast, samples processed in other systems exhibited a drop to ~23% under the same 0.5 T applied field. We have traced the cause of this strong field dependence to excessive duration of high temperature dry annealing, which is inherent to the design of the R2R system. By adjusting the thermal profile, i.e., shortening the dry annealing time, samples from this atmospheric-pressure system now consistently exhibit a field dependency that is comparable to other systems.

Double-sided YBCO samples have been processed on RABiTS with the CeO₂/YSZ/LZO/NiW architecture. To avoid scratching of the substrate or buffer from our contact heaters, the RABiTS were fabricated under stationary conditions (short length). Results on converted samples indicated that both the top and bottom sides of the precursor films are epitaxial, and are fully converted without requiring addition times compared to single-sided samples.

Technology Integration: Results on ex-situ conversion of various types are of significant interest to AMSC, and our results obtained in the base program are routinely shared with AMSC. We have also collaborated with AMSC in that a significant fraction of conversion studies were performed on AMSC RABiTS. In the area of PED precursor work, substantial knowledge concerning the stability of PED has been transferred to Neocera. Work on solution precursor/buffer continues with SNL, and in-situ XRD analysis of solution reaction kinetics has been initiated with NIST. Research collaboration with University of Cincinnati on the development of MOD precursors has been successfully concluded with the graduation of a Ph.D. student. Collaboration with ANL on phase development using Raman, which was highly rated in FY03 review, continues on a reduced scale due to the delay in alternative precursor

development owing to budget cuts. Throughout the year, our industrial CRADA partners were regularly briefed on our findings.