LANL-SuperPower CRADA: Development and Multi-Scale Characterization of IBAD MgO/MOCVD YBCO Coated Conductors

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SuperPower’s 2G wire is based on high throughput processes & superior substrate

- High throughput is critical for low-cost 2G wire and to minimize capital investment
- SuperPower’s 2G wire is based on high throughput IBAD MgO and MOCVD processes
- Use of IBAD as buffer template provides the choice of any substrate
- Advantages of IBAD are high strength, low ac loss (non-magnetic, high resistive substrates) and high engineering current density (ultra-thin substrates)
LANL-SuperPower Collaboration on IBAD-MgO based 2G Wire since 2003

- LANL-SuperPower CRADA in the ninth year. This collaboration has been an integrated part in our 2G wire program
- Applied research at LANL scaled up successfully at SuperPower.
  - Substrate electropolishing:
    - 2003 - Technology transferred from LANL to SuperPower
    - 2007 - SuperPower reports electropolishing of 1400m long tape
  - IBAD-MgO buffer:
    - 2003 -2006 - Technology transferred from LANL to SuperPower
    - End of 2006 - SuperPower made the first largest delivery of 2G wire, IBAD-MgO based MOCVD wire, ~10,000 m delivered to Albany Cable project
    - Now: routine production of 1000+m long buffered tapes at high throughout at SuperPower
- Support also on Testing and microstructure analysis
  - In-field Ic test at 1 cm resolution and over ~1 m length helped identify issues with MOCVD process since 2004
Long-length, high-current 2G Wire produced in SuperPower manufacturing line

- Minimum current ($I_c$) = 282 A/cm over 1065 m
- $I_c \times \text{Length} = 300,330$ A-m (new world record!)

77 K, $I_c$ measured every 5 m using continuous dc currents over entire tape width of 12 mm (not slit)
2G Program relevance to DOE-OE mission

• Strongly tied to the DOE-OE mission through the development of 2G HTS wires that meet all the necessary requirements (piece length, performance, cost, and manufacturing capacity) of systems that are being constructed to modernize the electric power grid and enhance the reliability and security of the energy infrastructure.

• The program has a direct impact on OE sub program goals namely, development of prototype wire achieving 1,000,000 A-m and production of HTS coil operating in applied magnetic fields up to 5 T at 65 K.
  – **Very strong emphasis on Length $\times I_c$ performance and high-field coil demonstration in this program**

• FY09 objectives established to focus on high current and in-field performance wire, high efficiency processes and advanced wire architecture. Scope of LANL-SuperPower collaboration consistent with new program focus.
Substrate planarization will improve process efficiency

- Simultaneous deposition of alumina & yttria layers sequentially using two chambers available in Pilot Buffer system - combined two process steps into one, eliminating one set-up time.
- Speed of 750 m/h* of 4 mm wide tape to deposit both alumina & yttria; deposition time is only 6 hours!
- Simultaneous deposition of homo-epi MgO & LMO processes
- 4 buffer layers deposited on 1,400 m tapes routinely with one pilot buffer system. Weekly production*: 8,400 m.
- Substrate planarization enables elimination of vacuum deposition of alumina and yttria; buffer throughput will double to 16,800 m/week.
The LANL – SuperPower collaboration is focused on advancing the development of commercially viable HTS wires based on IBAD MgO templates and MOCVD YBCO film deposition.

HTS wire is an important part of DOE’s mission to develop a more reliable and secure electricity transmission grid (Cables, Fault Current Limiters) in the U.S.

FY 2009 funding $ 700K

Outline:

• Electropolishing
• Solution Deposition Planarization
• Long length studies at LANL
• Reel-to-reel in-field measurement capability at SuperPower
• Pinning studies on short samples
Electropolishing

IBAD-MgO requires a smooth starting substrate for good final texture

- IBAD texture degrades rapidly with increasing roughness
- Need smooth surface to 1-2 nm RMS roughness (5 x 5 µm)
Electropolishing is a key enabling technology that allows production of substrates of smooth Hastelloy tapes in long lengths.

**LANL – SuperPower Electropolishing Timeline**

- **1996:** LANL starts electropolishing short tapes (for IBAD YSZ)
- **2002:** LANL starts electropolishing long tapes in reel-to-reel system
- **2003:** LANL transferred technology to SuperPower
- **2007:** SuperPower reports electropolishing of 1400m long tape
- **2008:** LANL successfully electropolished a 1000m long, 12 mm wide, Superpower Hastelloy tape (10-fold increase in length from previous LANL record)
- **2008:** LANL continued electropolishing 50 to 100 m long tapes as needed for internal use
Goal: architecture simplification

By replacing Hastelloy by a template with similar mechanical properties but chemically simpler, the requirements for the barrier layer can be reduced.

Raw tape Roughness measured at SP by AFM

- $Ra = 10.5 \text{ nm}$
- Peak to valley: $30 \sim 139 \text{ nm}$

Electropolished

A different electropolishing solution was made and the tape was polished at LANL

Tape was returned to SuperPower for evaluation

- $Ra = 6.9 \text{ nm}$
- $PV: 120 \text{ nm}$

Short portion at LANL: IBAD-MgO/homo MgO:

- $\Delta \phi = 4.0^\circ$
- $\Delta \Omega = 2.6^\circ$

- $Ra = 1.1 \text{ nm}$
- $PV: 3 \text{ nm}$

Appropriate for use
Solution Deposition Planarization (SDP)
SDP research was performed in collaboration with P. Clem, SNL

Solution Deposition Planarization can be a low-cost alternative to electropolishing.
LANL developed a SDP capability, results presented at PR 2008 (V. Matias)

Additional benefits of SDP include:
• 100x reduction in chemical waste
• Potential architecture simplification by elimination of $Y_2O_3 / Al_2O_3$ layers
• Compatible with a broad group of metal templates
• Covers inclusions, completely encapsulates metal tape

**SuperPower defined the transfer of the SDP technology as a high priority within the CRADA**

Since late 2008 intensive activity and collaboration between LANL and SuperPower have produced significant progress in short time. We pursued 2 parallel approaches:

- LANL continued to develop the SDP technology and adapt it to SP needs
- With LANL support, SP started to design and build an in-house SDP capability
Solution Deposition Planarization is effective by overcoating surface asperities with a flat surface.

SDP films display ~85% shrinkage, so each layer only removes 15% of existing roughness:

\[
R_0 \times (1-t_1/t_0)^n = R_n
\]

\[30 \text{ nm} \times (0.85)^n = 2 \text{ nm}, \quad n \sim 16\]

Number of layers can be adjusted as needed.

SDP has been demonstrated at LANL to work well for IBAD.

![Graph showing the relationship between magnetic field (H) and current density (Jc) for Y2O3 SDP solution on PLD-YBCO on IBAD MgO.]

PLD YBCO

STO

MgO

SDP

Y2O3

Hastelloy

250 nm

Particle
**First path: SDP at LANL**

**Approach:**
- LANL received Hastelloy tape from SP
- Sent back several 3m long pieces after SDP with $\text{Y}_2\text{O}_3$ solution under various conditions, for completion of the rest of the architecture at SP
- SP applied a combinatorial research approach, testing different architectures and conditions on portions of each piece
- A 0.9$\mu$m thick REBCO layer was grown by MOCVD on all the samples

**Result:**
- Superconducting films with $I_c$ up to 130A/12 mm ($J_c \approx 1.2$ MA/cm$^2$) at 77K, self field were obtained

**Significance:**
- SDP is compatible with SuperPower CC technology, including MOCVD REBCO
Second path: a SDP capability was designed and built at SuperPower. It is currently operational, in a testing stage.

- Start: December 2008
- SP personnel trained at LANL: January 2009
- System operational at SP: June 2009

Reel-to-reel SDP system at SuperPower
Smooth Y₂O₃ films were directly coated on unpolished Hastelloy tapes by reel-to-reel planarization.

- **Raw tape**
  - 20X20µm
  - Ra = 9.8nm
  - Peak to Valley: 20 ~ 41 nm
  - Slope: 1~6 degree
  - 5X5µm
  - Ra = 2.5nm
  - Peak to Valley: 8 ~ 23 nm
  - Slope: 1~5 degree

- **Planarized**
  - 20X20µm
  - Ra = 2.4nm
  - PV: 9nm
  - Slope: 0.7 degree
  - 5X5µm
  - Ra = 0.9nm
  - PV: 4.5nm
  - Slope: 1 degree
Evolution of the roughness through the 4 layers of SDP Y$_2$O$_3$ coating at SuperPower on Hastelloy tape
Hastelloy tape planarized at SuperPower was sent back to LANL for deposition of the MgO layers and texture characterization.

**Result:**
Texture of the MgO
\[ \Delta \phi = 6.4^\circ \quad \Delta \Omega = 3.5^\circ \]

**Significance:**
The SDP facility recently installed at SuperPower is compatible with IBAD MgO CC technology.
SuperPower produced the complete CC architecture on SuperPower’s planarized tape

Result:
Superconducting films were obtained:

- Thickness = 0.9 μm
- $I_c(77K, sf) = 168A/12mm$ (140A/cm)
- $J_c(77K, sf) = 1.55 \text{ MA/cm}^2$

Significance:
The SDP facility at SuperPower is compatible with 100% Superpower IBAD-MgO / MOCVD REBCO CC technology
High quality buffers and HTS films demonstrated on planarized substrates

- 20 m long planarized yttria tape made on unpolished Hastelloy.
- IBAD MgO, Homo-epi MgO, LMO in pilot systems
- Out-of-plane texture = 4.5° FWHM, In-plane texture = 7° FWHM
- $I_c = 140 \text{ A/cm}$ achieved in MOCVD film with no alumina and only 3 vapor deposited layers

Planarization integrated in the complete SuperPower wire fabrication processes
Several years ago at LANL we started to develop a non-destructive capability for position-dependent measurements of the performance of long CC, that allows us to cope with and anticipate the rapidly-evolving and increasingly challenging requirements of our industrial partners.

**Basic concept:**

In-field measurements: reduced $I_c$ avoids sample damage and defines the position.
This “timeline” includes both LANL IP, hardware and software development, and support to SuperPower through the CRADA.

PR 03: 1 m long, ~1 cm wide tapes. Single H//c, T=75K

PR 05: 4 cm wide tapes. Currents exceeding 350 A.

PR 06: Modular design allowed use of 2 stages simultaneously (different H or orientations, higher throughput)

PR 06: 4 mm wide stabilized “final product”

PR 07: 25m long, improved reliability & tracking. First SuperPower tape measured.

PR 08: First hardware ready to be delivered to SuperPower.

LANL Long Length Characterization Timeline
Since the last PR we made several improvements to our long length measurements system, that were applied to investigate SuperPower tapes.

(\textit{the development of these new capabilities was not part of the CRADA})

We increased the current capability in our system from 125A to \(I>150A\) enabling higher \(I_c(75K, sf)\) on industry’s 4mm wide tape to be measured.

By controlling the electromagnet with a bipolar power supply we can apply negative fields, to compensate the remanence. This is necessary to accurately measure \(I_c\) at self field.

With these improved capabilities we made position dependent in-field measurements on several long SuperPower tapes.
We measured 3 pieces (lengths ~ 25m, 25m & 50m) of SuperPower 2009 production tape with Zr-doping.

• Angular dependence measured at selected positions.
• The tilted “ab-peak” and asymmetric shape are frequently observed in IBAD tapes.
• The shape, values, and angles of the peaks are very uniform along the length.
• $I_c(X)$ measurements at the angle of the peak, not at 90°

$B = 0.52T$  

- $X$ (cm)

- $0$
- $255$
- $623$
- $1273$
- $1854$
- $2327$

- $I_c(A)$

- $\Theta = 0^\circ$ ($\sim H//c$)
- $\Theta = 96^\circ$ ($\sim H//ab$)

• $I_c(X)$ measured every 15 cm over a range of ~ 2 cm
• Both orientations measured simultaneously
Comparison of the 3 pieces of SuperPower 2009 production tape with Zr-doping: The angular dependences are very similar.

Rotations over more than 360° can be used to check the measurement system accuracy and reproducibility.

TEM studies show that the tilt is due to sheets of second phase particles tilted relative to the YBCO basal plane.
The 50m piece was measured at 3 orientations: $\Theta=0^\circ$ ($\sim H//c$), $\Theta=97^\circ$ ($\sim H//ab$) and at the lowest $I_c$ orientation $\Theta=60^\circ$

We measured the field dependence of $I_c$ at selected positions.
New magnet setup allows measurements from self field to ~1.3T. The field dependence of the Zr-doped tape is very uniform.
LANL provided a magnet stage (0.52 Tesla over 7.5 cm) for in-field measurement on long tapes at all field orientations (360°) & associated testing know-how. SuperPower constructed a reel-to-reel system with sensors, controllers and software using this magnet stage.
• Detailed angular dependence at the 1st meter was measured first to determine the field angles to be used at the reel-to-reel in-field measurement.
• Seven field angles were selected: 0°, 40°, 70°, 88°, 89°, 90°, 110° for reel-to-reel in-field measurement. Tested 100 m long section. Data at three field angles are shown. 40° is the field angle at which a minimum $I_c$ was observed.
A ~30 m long segment from the new production tape was measured. Detailed angular dependence at the 1st meter shows dramatically improved performance.

Although the enhanced performance is accompanied with an asymmetry in the angular dependence, both $I_{c_{\text{min}}}$ values are at least twice as that in 2008 undoped tape.
• Measurements at SP and LANL on the same tape (different portions) are consistent
Vortex pinning studies on short samples at LANL

We complemented the long length measurements with studies of short pieces of SP production tape and research samples, by using both transport and magnetization tools.

Transport studies on Zr-doped 2009 production tape

Good consistency between reel-to-reel and short samples results
Vortex pinning studies on short samples at LANL

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Transport studies on Zr-doped 2009 production tape

Good consistency between reel-to-reel and short samples results

The “ab peak” is shifted ~12° at 0.2T and monotonically approaches $\Theta=90^\circ$ (the “true” ab) as H increases.

Combined effect of pinning by tilted planar defects (layers of $Y_2O_3$ particles) and the misalignment between applied and internal magnetic field due to YBCO anisotropy.

Maiorov et al. APL 2005.
Holesinger et al. SuST 2009.
We use the angular dependence of the N value of the I-V curves as a tool to discriminate pinning from planar defects and “intrinsic pinning”

Example from another 4mm wide SuperPower sample (without Zr)

Normally N grows with $J_c$ for all types of pinning centers.

Exception: “intrinsic pinning” by crystallographic ab planes, which is periodic pinning, where N decreases as $J_c$ increases.

In these SP tapes both the crystallographic ab planes and the tilted planar defects produce pinning. The result is a N(Θ) with sharp minimum and maximum.

Analogous studies in the Zr-doped samples are in progress
By combining transport and magnetization tools we investigate vortex pinning over a wide range of temperatures and fields.

This extensive set of tools have shown over the past several years to be very effective at identifying structure/properties correlations, and at providing feedback for processing optimization.
Performance Against AOP Milestones for FY2009

- LANL will adapt the recently developed 3-layer simplified architecture for compatibility with SuperPower’s MOCVD-RE123 technology.

- LANL will provide SP multi-meter sol-gel smoothed metal tape for deposition of IBAD template layers.

- LANL will deposit Y-Al-O composite on a multi-meter long metal tape and send it to SuperPower for deposition of the remaining of the 4-layer simplified architecture and MOCVD-RE123. (Stretch goal)

The key concept is architecture simplification. Specifics were modified by mutual agreement to focus on the SDP technology

- Ni-Cr 80/20 tape from SP electropolished at LANL.

- Solution Deposition Planarization technology successfully transferred to from LANL to SuperPower. System designed, built and operational at SuperPower.
Performance Against AOP Milestones for FY2009

- LANL will measure and analyze the $I_c(H,\Theta,T)$ of SuperPower’s long tapes and short samples by transport and magnetization studies.

- LANL will explore the processing/structure/properties correlations in SuperPower coated conductors by the combination of transport and magnetization studies with AEM and other structural tools.

- Improved positional dependent long length characterization set of tools at LANL used to extensively characterize $I_c(H,\Theta,X)$ in several SP production tapes
  - $I_c(X)$ at several orientations (maximum length ~50m).
  - $I_c(\Theta)$ at selected positions: asymmetry due to tilted planar defects
  - $I_c(H)$ from self-field to ~1.3 T. Determination of $\alpha$ values.

- Investigation of $I_c(H,\Theta,T)$ and $N(H,\Theta,T)$ in short pieces of production and research samples by transport and magnetization. Dip in $N(\Theta)$ fingerprints intrinsic pinning.
Performance Against AOP Milestones for FY2009

- LANL will develop and deliver hardware to SuperPower for long-length in-field characterization, compatible with their existing 5m length self-field system.

SP incorporated the magnet stage designed, built and tested at LANL into reel-to-reel transport $I_c$ rig for measurement of production tapes over long lengths. System is fully operational and in use.

![Graph showing field angle vs. $I_c$]
Performance Against AOP Milestones for FY2009

• Test AC losses of SuperPower’s long striated tapes. Confirm / disprove loss reduction effect of ‘filaments and cross cuts’ in IBAD coils.

This activity was put on hold during FY 2009.
Technology Transfer

The activities in the LANL-SuperPower CRADA during 2009 supported various aspects of the path to commercialization of SuperPower’s coated conductors.

The CRADA activities included site visits, conference calls, interchange of samples and data, discussions of results and plans, and joint publications.

SDP research was performed in collaboration with P. Clem, SNL

Publication:
Plans for FY 2010

- LANL will continue supporting the development of the Solution Deposition Planarization capability at SuperPower.
- We will jointly explore SDP process on simpler substrates such as Ni-Cr.
- LANL will continue helping SuperPower in the setup of reel-to-reel in-field measurement tools, through hardware delivery, transfer of know-how, and training, as required.
- LANL will continue measuring and analyzing the $I_c(H, \Theta, T)$ of SuperPower’s long tapes and short samples by transport and magnetization studies, as needed.
- LANL will support SuperPower on studies of ac losses, as needed.

Specific milestones will be continuously revised and redefined in consultation, based on previous results and evolving needs.