# LANL/STI CRADA: Progress in Reactive Co-Evaporation on IBAD

**Vladimir Matias**, Chris Sheehan, Yates Coulter

*Superconductivity Technology Center*

*Los Alamos National Laboratory*

**Brian Moeckly,** Viktor Glyantsev, Chris Yung

*Superconductor Technologies Inc.*

LANL FY09 Funding: $400K, 1 FTE

*Project Goal: Reduce cost of coated conductor manufacturing*

*Our project addresses the OE HTS wire goals through significant improvements in price performance ratio for HTS wire.*

# Superconducting power applications require low-cost HTS wire: ≤ \$10/kA•m

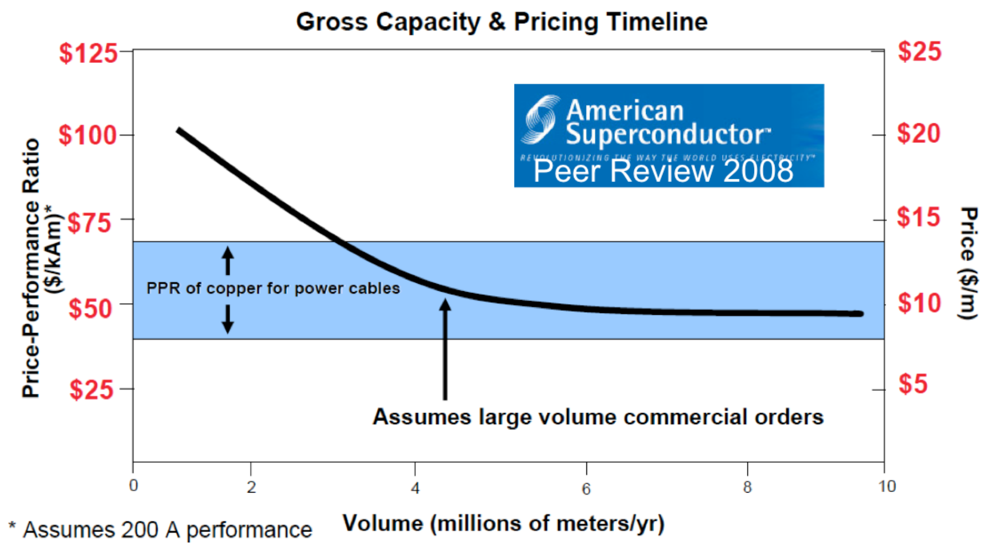
NAVIGANT CONSULTING

- Navigant Consulting market study (2006)
- In the near-term, Navigant says cost of HTS wire is a barrier to commercialization of superconducting grid applications
- For 2012 and beyond, Navigant study states DOE CC goals should be \$10/kA•m, 77 K, SF, and \$20/kA•m, 65 K, 2 T

- We believe that if the U.S. doesn't get there in a short time, foreign competition will be there before us

# Current trends in CC cost are not encouraging for achieving $10/kA•m

- Present price is $300-400/kA•m, $LN_2$, and projected future price (in volume) is $50/kA•m
- HTS deposition is the largest part of the cost
- Need a high-yield, high-quality, high-throughput, low materials and labor cost HTS process
- => Reactive co-evaporation (RCE) is a proven process in HTS film manufacturing for over 15 years; highest yield and lowest cost




SuperPower Inc — Subsdiary of PHILIPS Royal Philips Electronics N.V.

SuperPower (CCA '08): $50/kA•m may be attainable at 1000 km/yr

# A complete low-cost CC manufacturing strategy is required

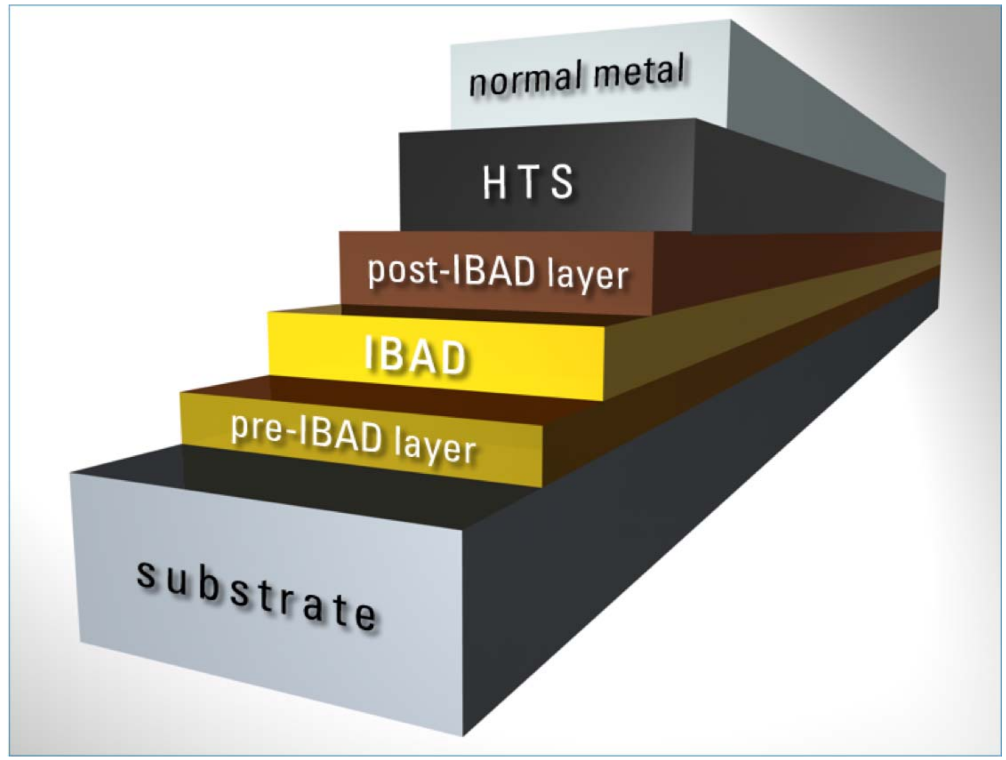
- ✓ Inexpensive substrate: *stainless steel with low-cost finish*
- ✓ Universal and inexpensive finishing process: *SDP*
- ✓ Low-cost template formation process: *fast IBAD*
- ✓ Simple buffer layer architecture: *single textured layer*
- ✓ Fast, large-area and high-quality HTS deposition process with low-cost materials: *RCE-CDR*
- ✓ Simple and fast normal metal deposition

# Goals of LANL/STI CRADA tasks are to develop low-cost and high-performance CC's

- Understanding the limits of the oscillatory environment RCE process (RCE-CDR, *Cyclic Deposition and Reaction*)
- Develop IBAD template for STI RCE-CDR process:
  - inexpensive substrates
  - simple IBAD architecture
- Develop and demonstrate high-$I_c$ HTS layers, in self-field and applied magnetic field, and characterize them
- STI will develop in parallel, with help from LANL, manufacturing processes for coated conductors

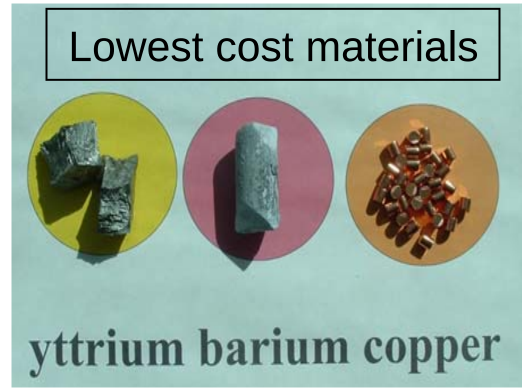
# Presentation Outline

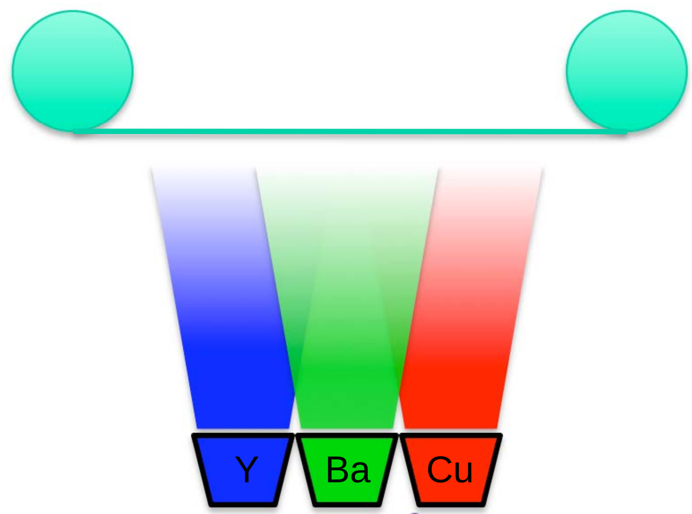
- RCE-CDR process and LANL results (VM)
- IBAD Template (VM)
- STI HTS results (BM)
- FY09 Milestones Status/Plans (VM)



# Reactive Co-Evaporation as a key enabler for low-cost coated conductors

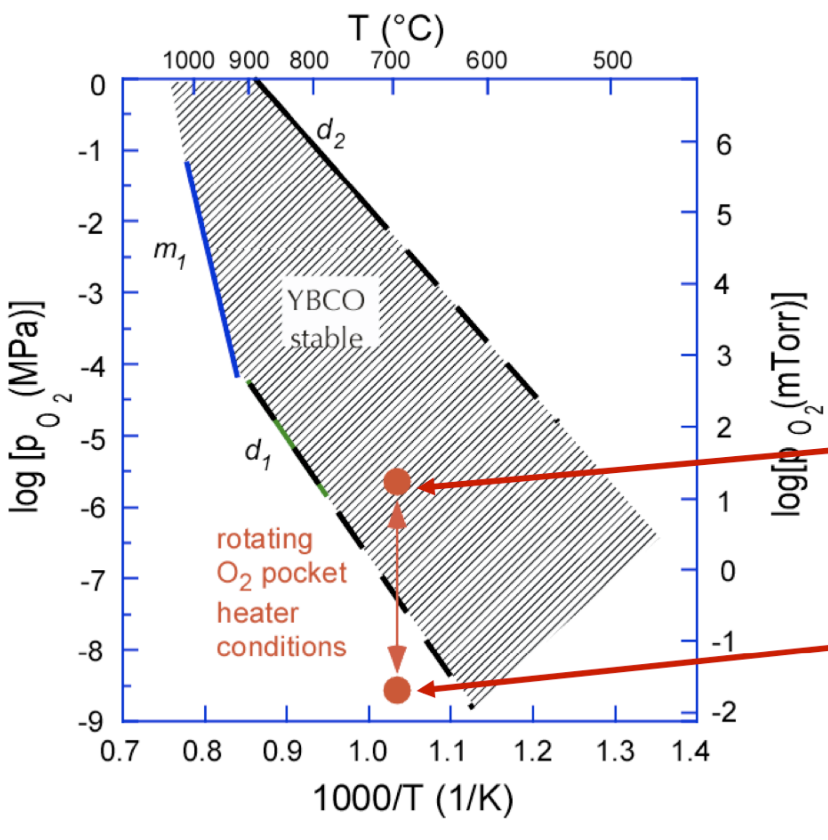
- Reactive Co-Evaporation (RCE) is an *in situ* growth process
- Coevaporation of individual elements
  - Elemental sources inherently least expensive
  - High deposition rate can be used
  - Scalable to large deposition area
  - Adjustable composition
  - Can self-dope using phases in the ternary phase diagram
- Low oxygen pressure during synthesis (10 - 20 mTorr)
  - Thermodynamic conditions are different compared to *ex situ* or even PLD
  - Lower deposition temperature



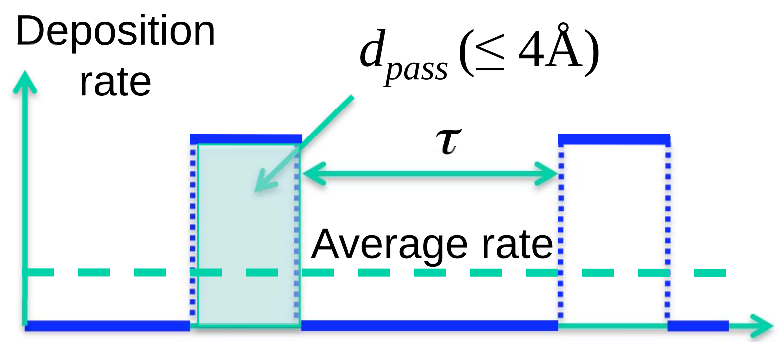



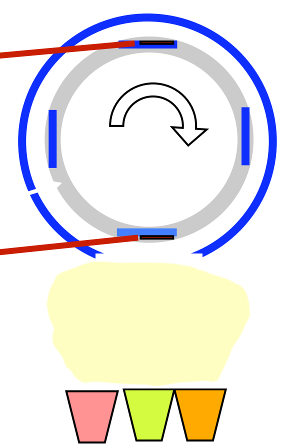
# RCE-CDR: Reactive Co-Evaporation by Cyclic Deposition and Reaction

Stability of YBCO in the *p-T* diagram




Matias et al *IEEE Trans Appl. Super.* 19, 3172 (2009)



- Low cost economics dictates high deposition rate
- Kinetics for growth of high-quality films dictates low dep rate
- Solved by mechanically rotating samples and thereby parallel processing many samples simultaneously

STI

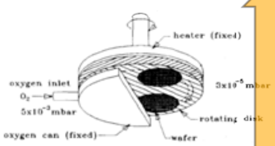


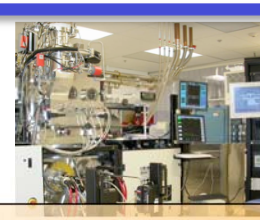
Los Alamos

# RCE-CDR process development historical timeline

**Wafer Deposition**

1990 1995 2000 2005 2010



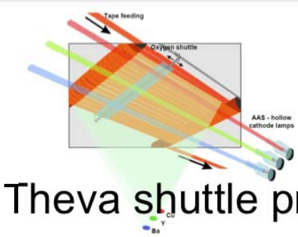



Conductus/STI process

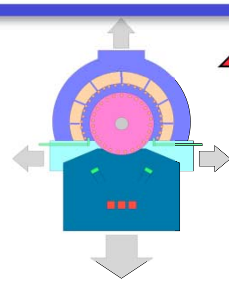
Kinder, TU Munich develops process;
Continued by Theva GmbH THEVA

**Coated Conductors**

1995 2000 2005 2010




Theva shuttle process

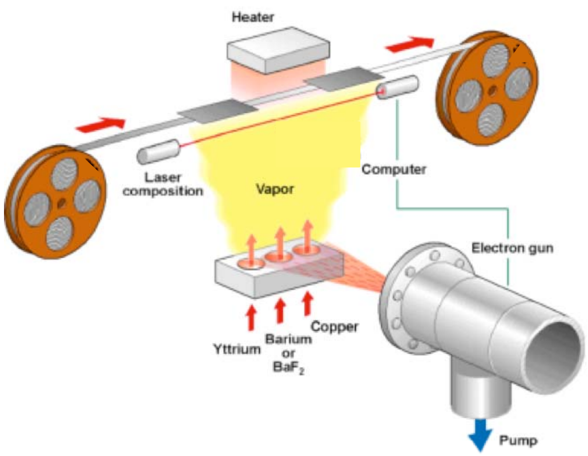


Korean EDDC process

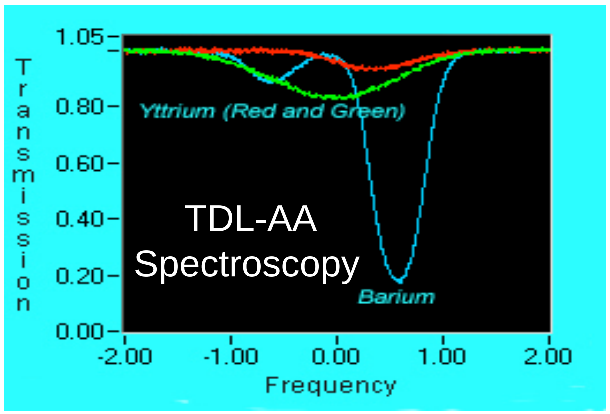
LANL reports results on RCE PR07

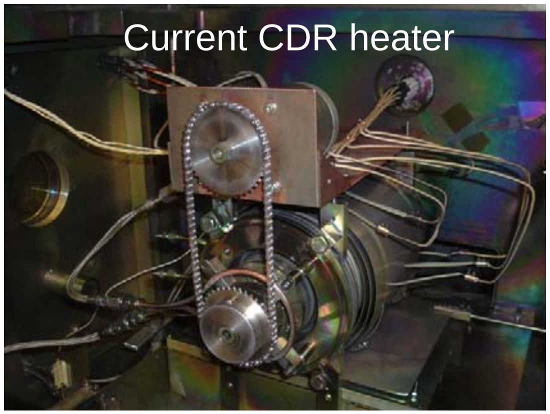
SuNAM, Korea

# LANL RCE-CDR Setup for Coated Conductors




- RCE-CDR approach
- Differentially pumped Pierce electron gun for evaporation
- Diode laser atomic absorption (AA) rate control
- Deposition rate 6 – 10 nm/s

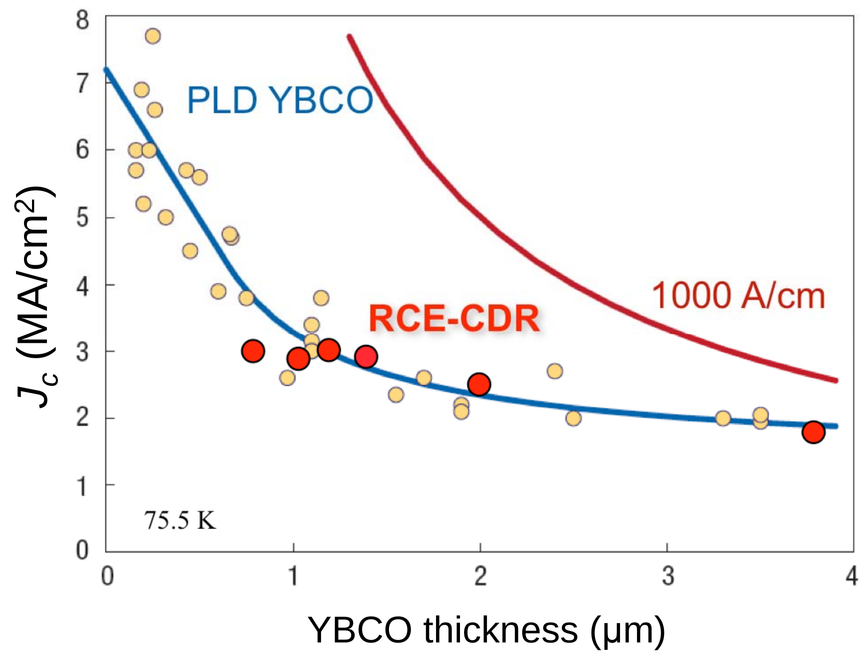



# Best RCE Critical Current Results at LANL

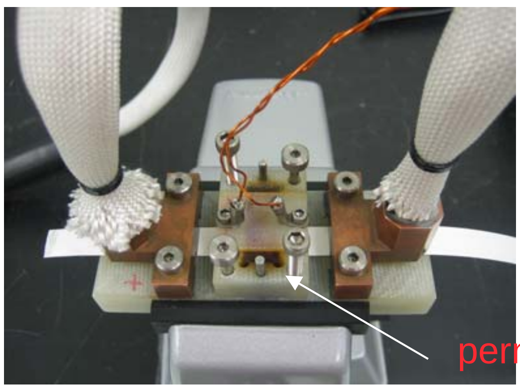
- Best Self Field results to date:
  - 3.0 $MA/cm^2$ in a 1.2 μm film
  - 2.5 $MA/cm^2$ in a 2.0 μm film
  - **2.9 $MA/cm^2$ in 1.4 μm**
  - **950 A/cm-width in 6 μm**

**New results since PR08**




PLD data from Foltyn et al., *Nature Materials* **6**, 631 (2007)

Matias et al *IEEE Trans Appl. Super.* 19, 3172 (2009)



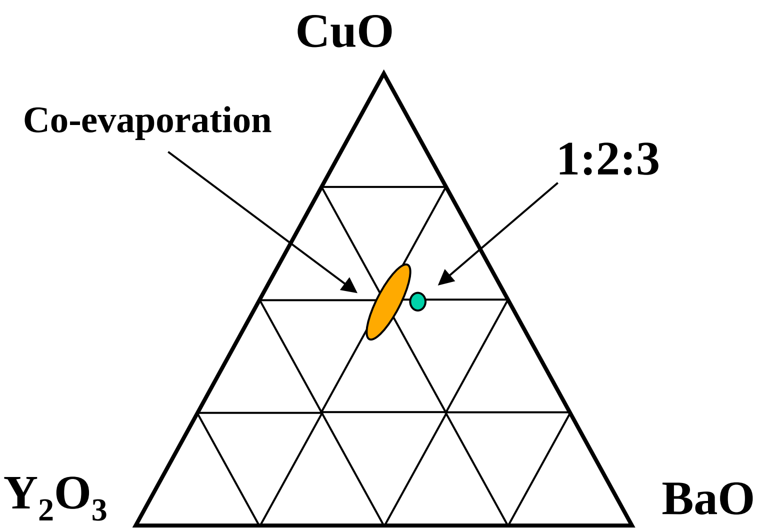
permanent magnets

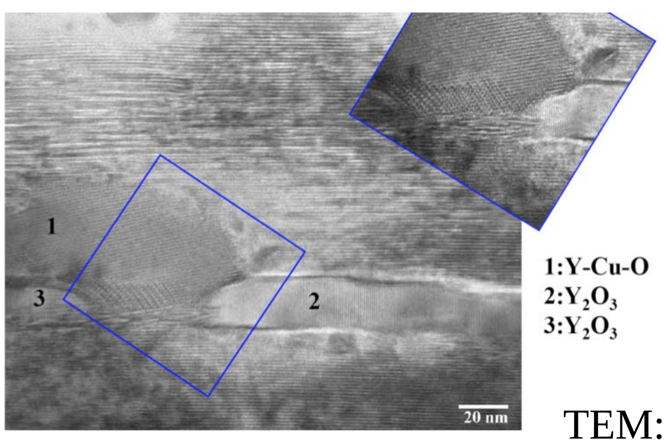
- Measure full 1-cm width coated conductor in magnetic field
- Scale SF result from bridge



Los Alamos

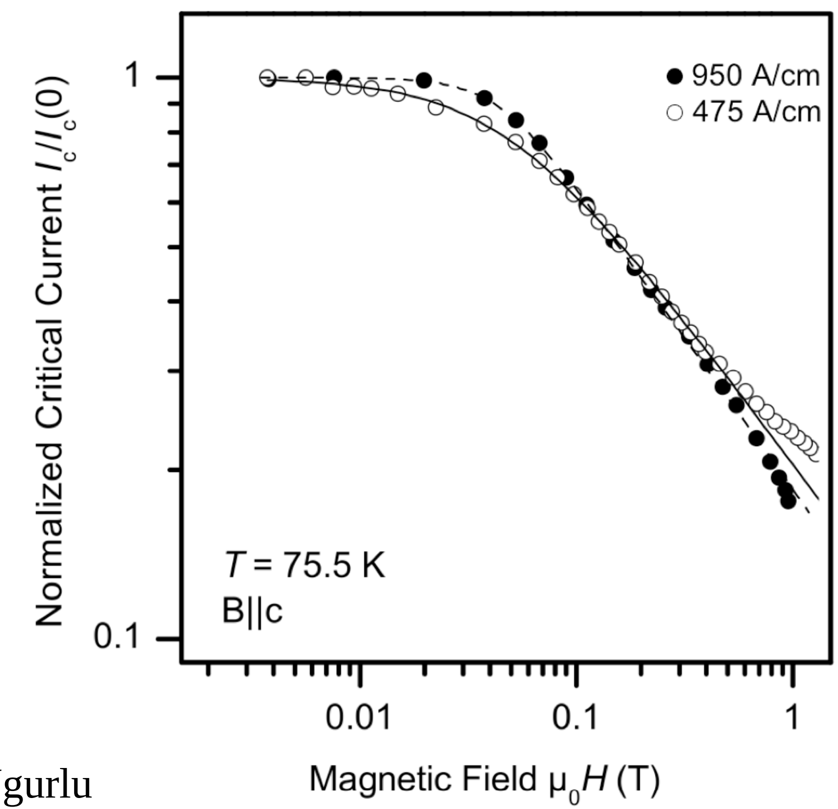
# Magnetic field dependence of critical current can be adjusted by adding pinning centers




TEM: Ozan Ugurlu

- Composition can be adjusted to optimize pinning centers from ternary phase diagram



# Cost projection for RCE-CDR process in volume yields \$4/kA•m for HTS layer deposition

- Conservative cost model uses present performance values ($J_c$ of 2.5 MA/cm$^2$ at 2 μm)
- Scales up to 5000 km/yr production
- Assumes only 10% utilization and 70% yield; included are capex depreciation and indirect (facility) cost
- Cost model predicts \$4.50/kA•m, $LN_2$ (for HTS layer only)
- Still possible to reduce cost further by: materials recycling and process automation

Matias et al. *J. Korea Inst. Appl. Super. Cryo.* **9**, 1 (2007)

# In FY09 LANL explored two simplified IBAD templates with STI : both successful

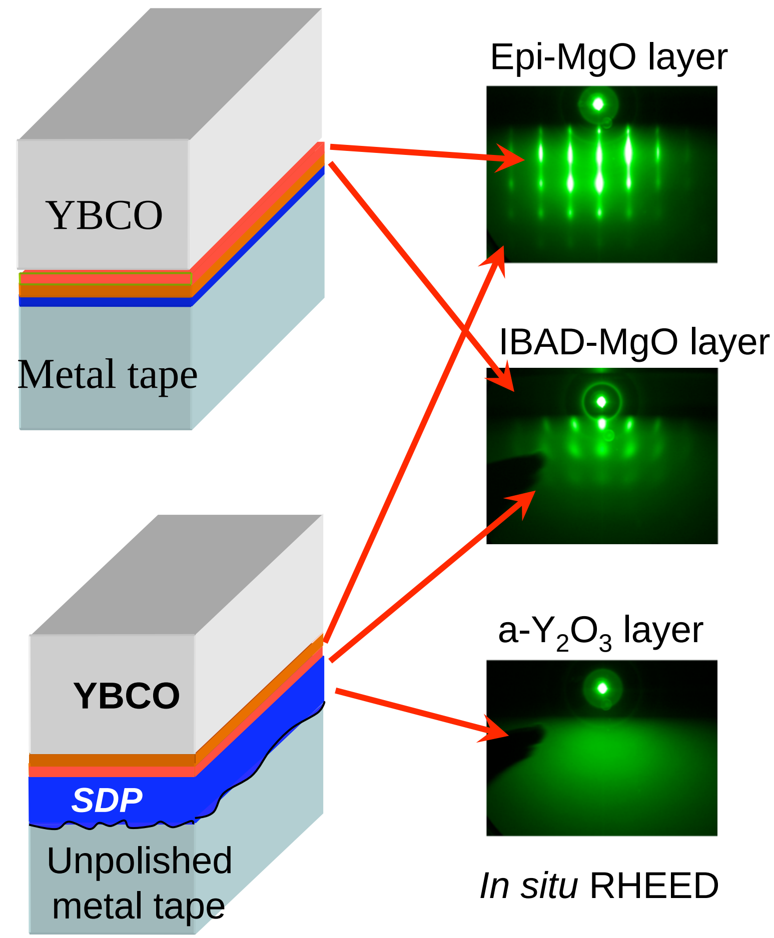
- IBAD Template A:

  Electropolished Hastelloy / $Y_2O_3$ (6 nm) / IBAD-MgO / epi-MgO (25 nm)

| |
|---|
| TOTAL IBAD Layered Template Thickness ~ 35 nm! |

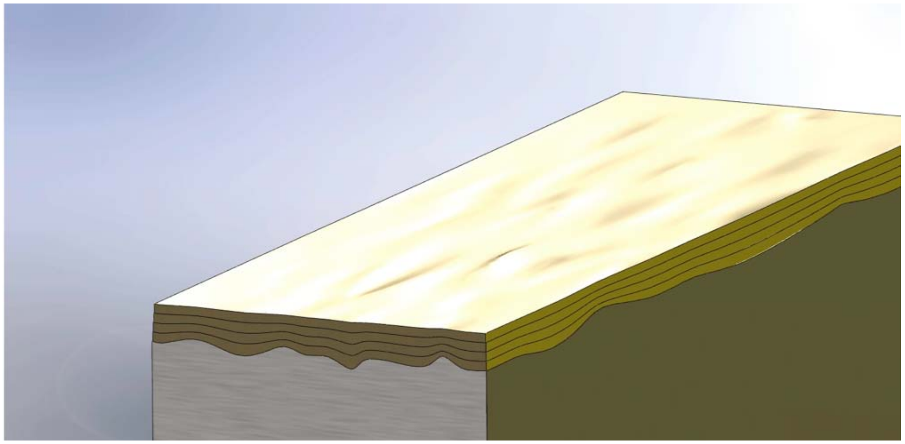
- IBAD Template B:

  Unpolished metal / SDP $Y_2O_3$ (1 μm) / IBAD-MgO / epi-MgO (25 nm)



# Solution deposition planarization (SDP) is a promising method for substrate finishing and eliminating defects

- Solution planarization is an effective means of overcoating surface asperities with a flat surface
- Solution deposited layer encapsulates metal tape and prevents metal interdiffusion
- Process developed by Los Alamos and Sandia National Laboratories




TEM: Terry Holesinger

After solution deposition substrate is IBAD-ready with an $Y_2O_3$ bed layer

See LANL presentation this afternoon in the strategic research session.

# Corporate Overview

- Founded 1987
- Focus: HTS thin films for RF & microwave applications
- Develops and manufactures all key technologies
  - HTS thin films
  - Stirling cryocoolers
  - Cryogenic filter/LNA systems
- Regional sales & customer service offices throughout U.S.
- Solid customer base
  - Wireless Carriers:
    - AT&T, Verizon Wireless, Sprint, T-Mobile, Alltel, US Cellular
  - U.S. Government:
    - Air Force, DARPA, Navy, Army, Dept. of Defense, NASA, LANL, ORNL


STI Superlink HTS RF filter receive system

# Cryocooler expertise

- 17 years of cryocooler and cryopackaging experience
- Free-piston integral Stirling cycle cryocooler technology
- Proven high-volume, low-cost manufacturability
- High reliability, long life, and high performance
  - Zero maintenance required
  - Over 6,000 cryocoolers deployed
  - Many cryocoolers have been in continuous 24/7 operation without maintenance in remote sites for >10 years
  - No signs of wear-out mechanisms
  - Run-time > 200 Million hours
  - MTBF >> 1 Million hours
  - Typically achieves 20% Carnot efficiency at 77 K in the field
- Scalable


- 5 W lift at 77 K;100 W input at 35°C heat reject
- 6.2 lbs.
- 3.6 in. OD x 11.8 in. Length
- -40°C to 60°C ambient
- 60 Hz, 120 W maximum
- Any orientation
- Vibration level with passive balancer: <10 N

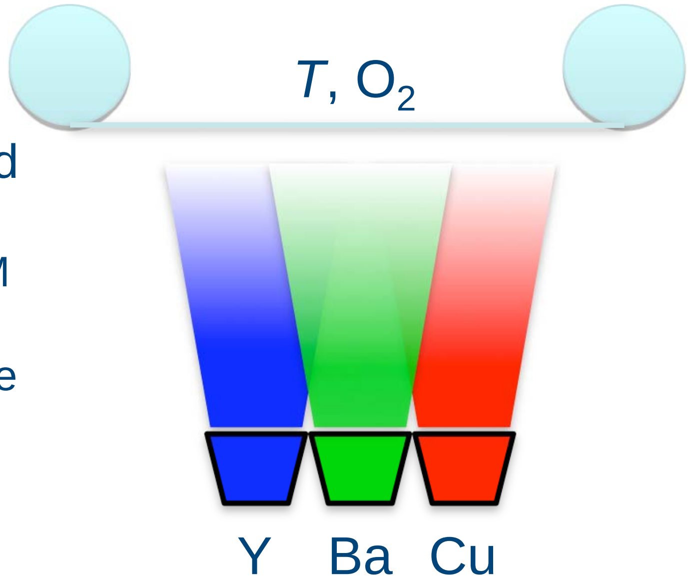
# High-throughput RCE-CDR HTS deposition system

- *HTS film production is inexpensive*
- *Bulk expense of completely fabricated HTS filter is due to substrate cost*

- Comprised of STI proprietary technology
- Load-locked wafer transfer
- Multiple sub-chambers
- Semi-automated operation
  - Extensive software interfacing
  - All operations controlled and operated from GUI
  - Run by operators (not Ph.D.s)
- Very stable rate monitoring and control
- Highly reliable: yield > 95%
- Current production capacity ~100 $m^2$/yr (@2 shifts/day) for 1-$\mu$m-thick films in *batch* mode
- Multiple sources for thicker film growth

# Other advantages of STI RCE-CDR HTS for CC

- Produces the highest quality HTS films
- *In situ*, single-step growth process
- Strict control of film composition over large areas and thicknesses
  - Uses well-established PVD composition controls (QCM and AA)
  - Maintains YBCO composition to within 2 at% over large areas and from run to run
  - Instantaneous composition control
- Superb film crystallinity is maintained at all times
- Single-coat process: sub-μm to several μm
- Quasi-blackbody heater ensures uniform temperature control over large areas
- Allows use of lower-cost, reduced-layer-architecture textured templates
  - Can deposit directly onto very thin MgO layers
- Can transfer innovations developed using other PVD techniques (e.g., BZO additions via PLD)



# Low-cost 2G HTS wires

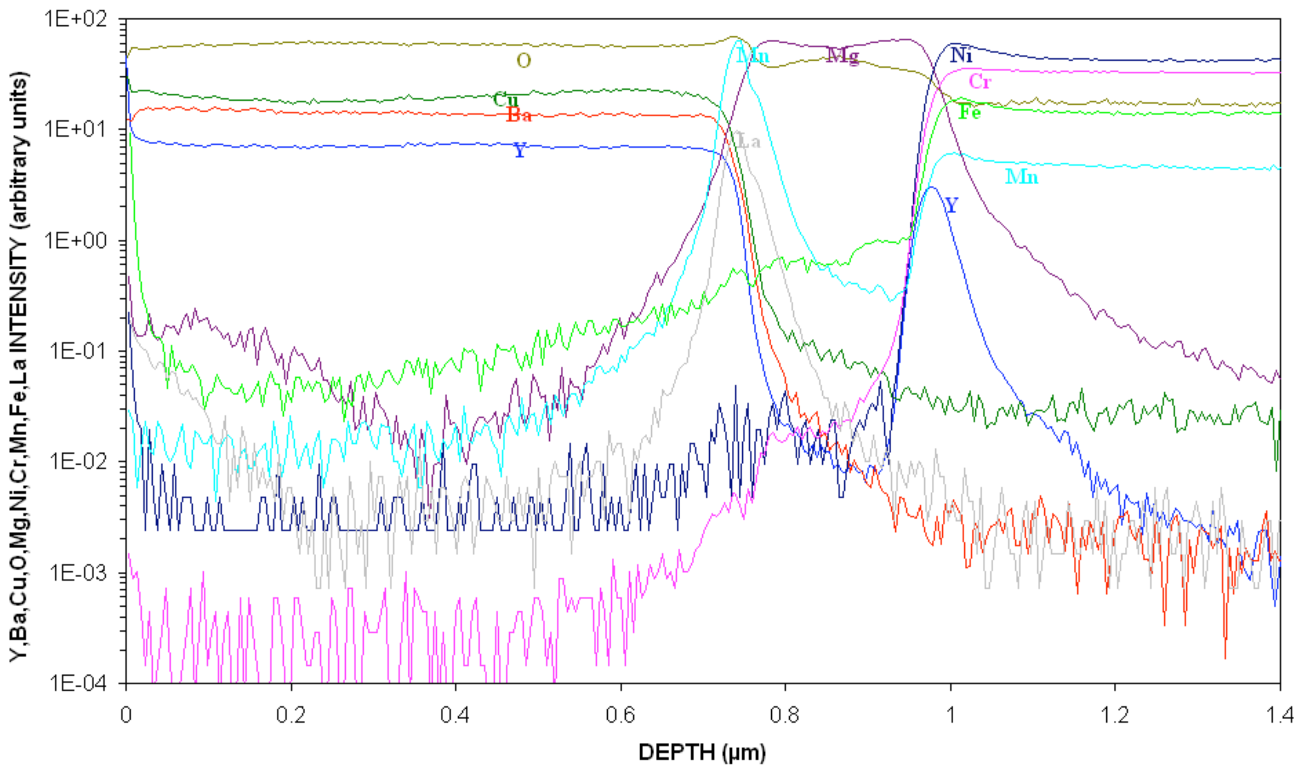
- Major components of cost
  - Capital equipment
  - Materials
  - Yield / throughput
- Reduced capital equipment costs
  - Single-step processes, semiconductor-like equipment
  - No metal tape rolling or polishing equipment required: tape directly from vendor
  - SDP $Y_2O_3$: Non-vacuum process equipment
  - IBAD/epi MgO: Single deposition system required
  - RCE-CDR: Single deposition system required

  } *Key tools needed for 800 km/yr*
- STI has focused on HTS thin film manufacturing for 20 years
  - RCE for 15 years
- HTS thin film production costs well understood
- Remains to transfer our batch wafer process to a continuous reel-to-reel process
- Similar high-volume, large-area PVD-based web-coating techniques exist in industry
- *Will allow 2G HTS wire cost to reach the commodity level*

# YBCO by RCE-CDR on standard IBAD template

- 700-nm YBCO grown by RCE-CDR

- Template:
  - Hastelloy C276
  - 5 nm $Y_2O_3$
  - 10 nm IBAD MgO
  - 200 nm epi MgO
  - 100 nm LMO

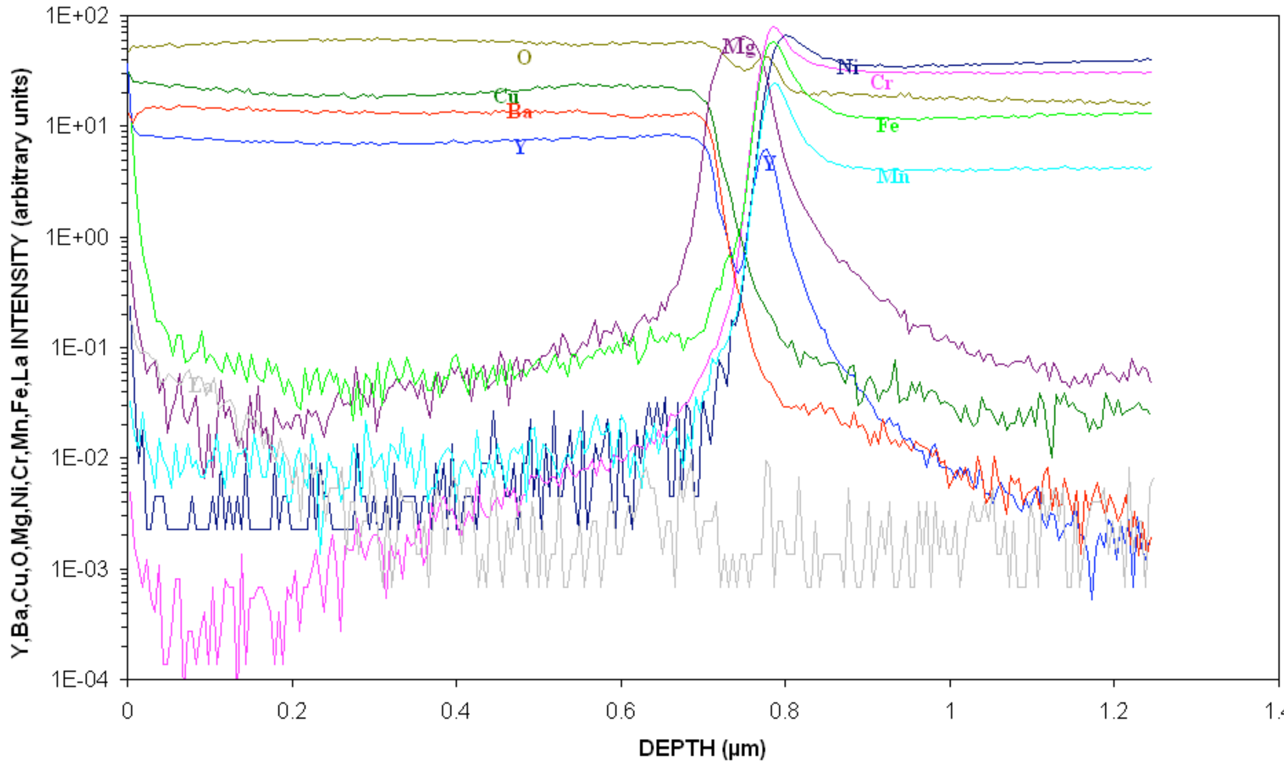



- YBCO composition is uniform throughout thickness
- No interdiffusion from Hastelloy substrate

B8032B

# YBCO grown on simplified IBAD template

- » 700-nm YBCO grown by RCE-CDR
- » Template:
  - » Hastelloy C276
  - » 5 nm $Y_2O_3$
  - » 10 nm IBAD MgO
  - » 25 nm epi MgO

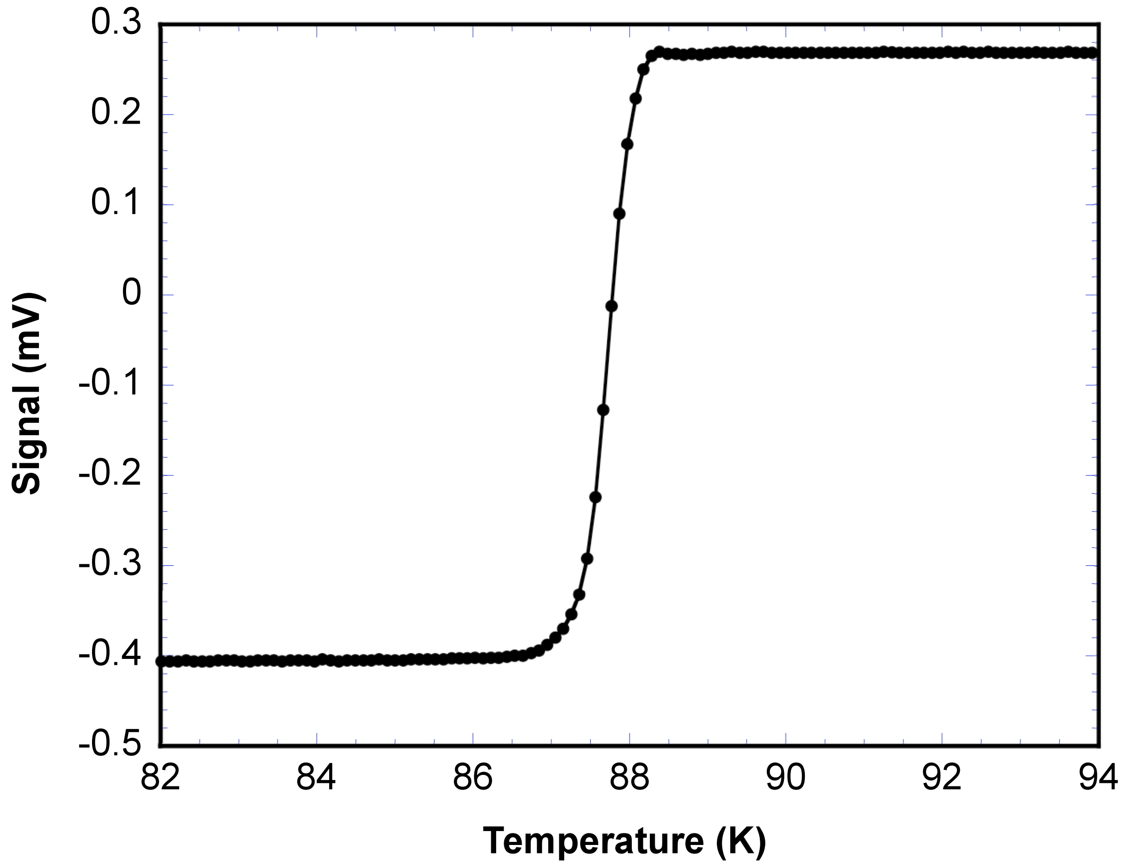



- » YBCO composition is uniform throughout thickness
- » No interdiffusion from Hastelloy substrate

B09005c

# Inductive $T_c$ measurements

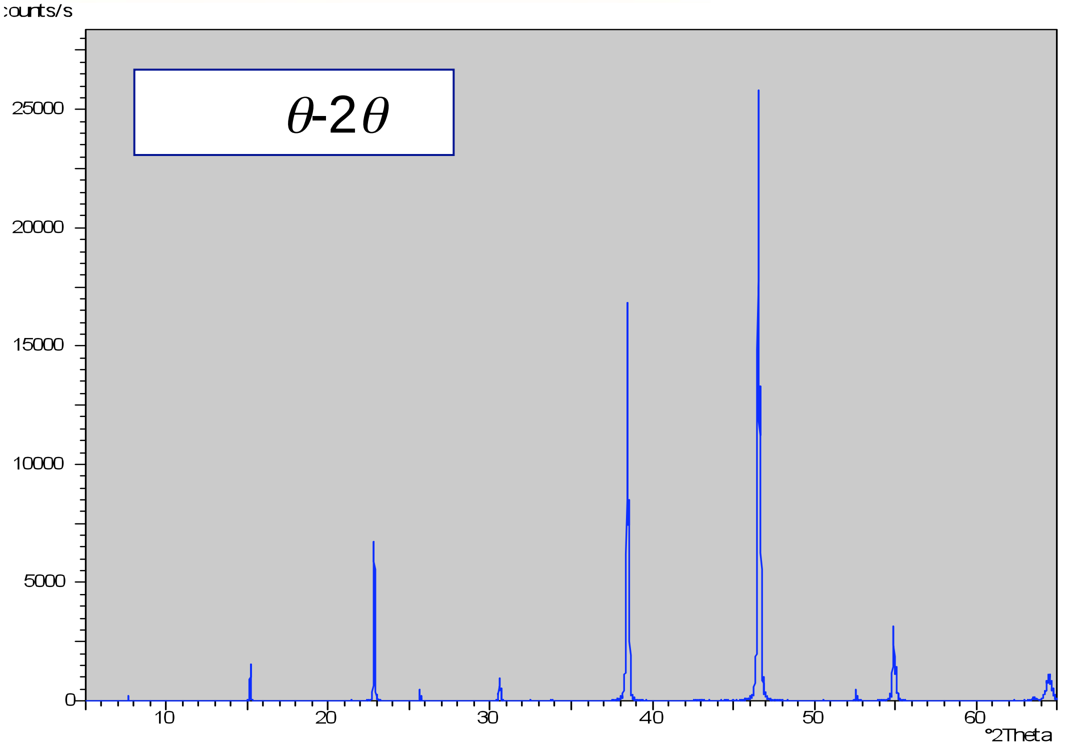
- 700-nm YBCO grown by RCE-CDR
- Template:
  - Hastelloy C276
  - 5 nm $Y_2O_3$
  - 10 nm IBAD MgO
  - 25 nm epi MgO
- Sharp transition




B09005c

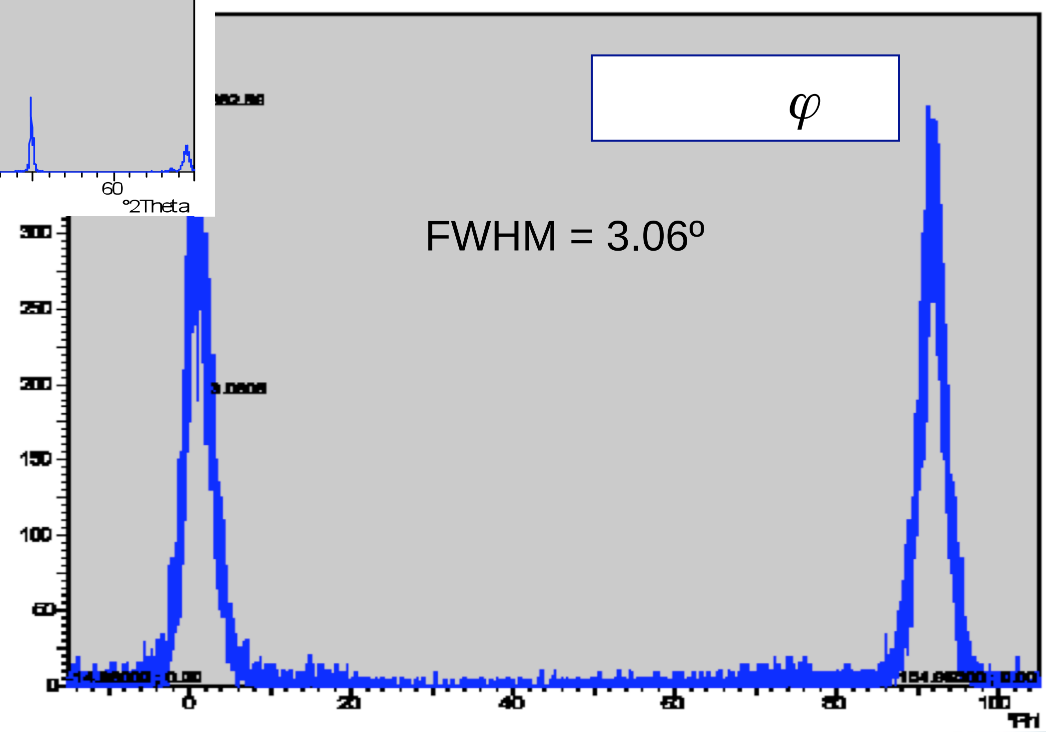
# YBCO directly on thin MgO / $Y_2O_3$ / Hastelloy

- 10 nm IBAD MgO + 20 nm epi MgO
- 1.5 μm YBCO film




Out-of-plane alignment

In-plane alignment

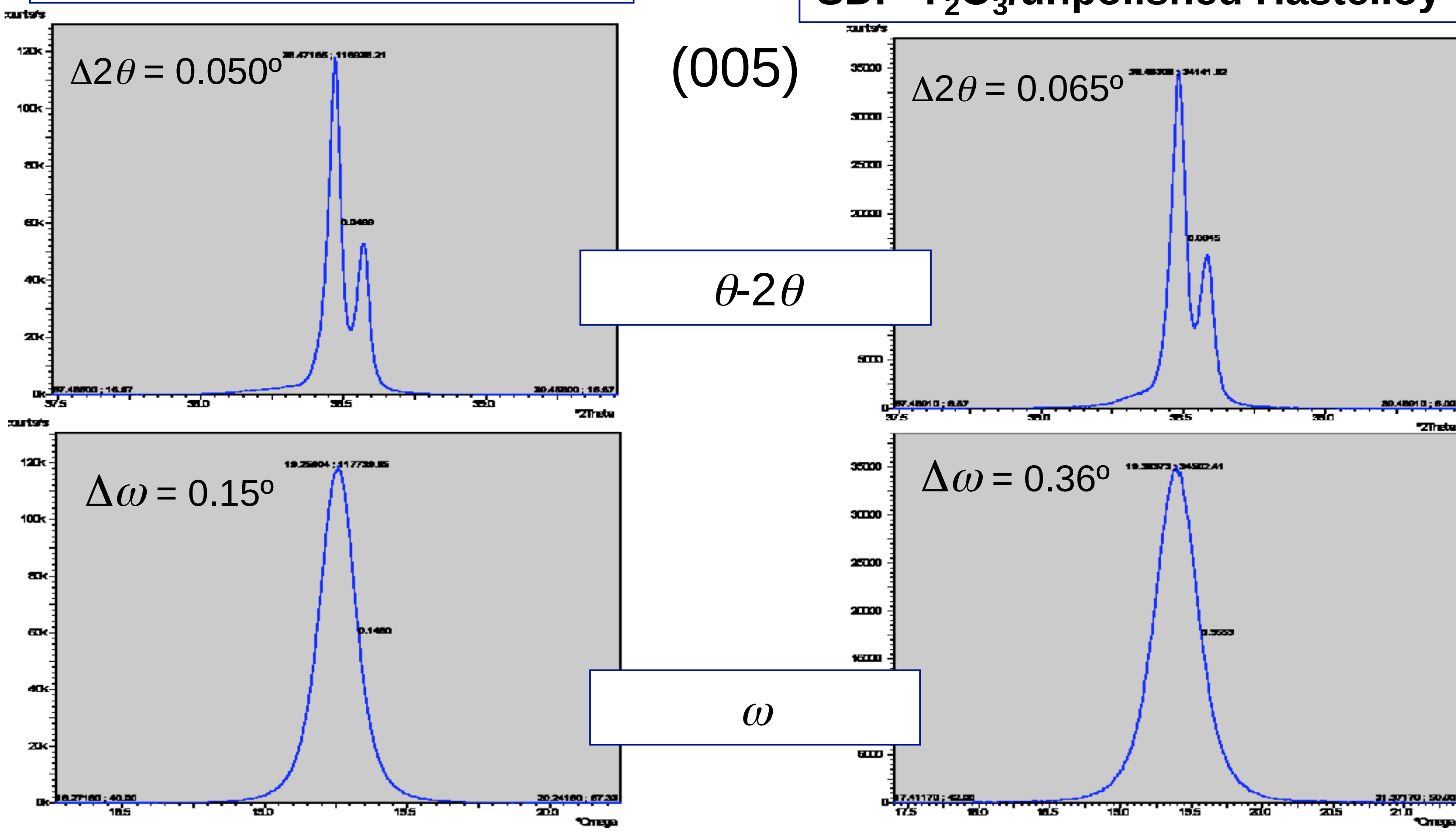


# Crystallinity

- Crystallinity is superb
- Compares favorably to our standard films on single-crystal MgO
- 5.3-μm films grown in same run:

**YBCO on MgO single crystal**

**YBCO on *30 nm* IBAD/epi MgO on SDP-$Y_2O_3$/unpolished Hastelloy**



# Selected $I_c$ measurements

- YBCO by RCE-CDR on simplified IBAD textured templates
- Measured on 1 cm x 6 cm tapes

| Sample | Template | YBCO *t* (nm) | $I_c$ (A/cm-width) At 75 K, SF | $I_c$ (A/cm-width) At 77 K, SF |
|---|---|---|---|---|
| X09010A | Template A<br>• Polished Hastelloy<br>• 6 nm $Y_2O_3$<br>• 5 nm IBAD MgO<br>• 25 nm epi MgO | 3000 | 354 | |
| X09058C | Template B<br>• Unpolished Hastelloy<br>• 1400 nm SDP $Y_2O_3$<br>• 5 nm IBAD MgO<br>• 25 nm epi MgO | 5300 | **590** | 524 |
| X09048A | Template C<br>• Polished Hastelloy<br>• 5 nm $Y_2O_3$<br>• 5 nm IBAD MgO<br>• 400 nm epi MgO | 5700 | | **623** |

# Development and production plans

- New pilot RCE-CDR deposition system for 2G HTS tapes
  - Phase I
    - Deposition on 1 cm x 6 m IBAD tape
    - Builds directly on our existing HTS wafer production system (deposition rates and areas)
    - System construction complete, in process development
  - Phase II
    - Continuous deposition on 50 m of 10-cm-wide tape/run
    - Equivalent 1.25 km of 4-mm-wide tape per run

- Transfer LANL template technology to STI
  - Pilot systems to produce 50 m of 10-cm-wide tape/run

- Scale-up to large-scale production to follow
  - Wider tape widths
  - 1-km+ lengths


Pilot RCE-CDR CC system

# FY09 Milestones

- Establish a CRADA between LANL and STI
  - ✓ Umbrella CRADA established in November 2008
- LANL will supply STI with sufficient quantity of IBAD templates for their RCE development work
  - ✓ LANL supplied a variety of IBAD samples up to 3 m in length
- Demonstrate high $J_c$ (> 2 MA/cm$^2$) YBCO deposited at 15 nm/s
  - ❖ Goal modified to adapt to STI priorities
  - ❖ LANL deposited by RCE directly on MgO layers (2.9 MA/cm$^2$ in a 1.4 µm thick film, $LN_2$ SF)
- STI will demonstrate YBCO films on IBAD templates with $I_c \geq 500$ A/cm at $LN_2$ in self field
  - ✓ STI demonstrated 620 A/cm, $LN_2$ SF, on LANL IBAD template

# FY09 Milestones Cont'd

- With LANL assistance, STI will reduce the number of layers in the IBAD template
  - ✓ STI demonstrated YBCO directly on MgO layers; 620 A/cm at $LN_2$
- STI will demonstrate CC fabrication in a reel-to-reel method – stretch goal (September 2009)
  - STI is in the process of developing a reel-to-reel tape transport deposition system

# Plans for FY10

- LANL will supply the required IBAD template in quantity to STI
  - Goal: enable STI to make intermediate length of coated conductors
- LANL will characterize STI coated conductors structurally and electrically
  - Goal: TEM and in-field electrical transport analysis of STI samples
- Continue with SDP-based templates to transition to stainless steel (SS)
  - Goal: demonstrate 500 A/cm on SS
- Develop artificial pinning centers in RCE-CDR for high $J_c$ in a magnetic field
  - Goal: demonstrate 200 A/cm at 2 T, 65 K, using STI CC process
- Develop a long length process for RCE-CDR at STI
  - Goal: Demonstrate intermediate length (> 5 m) capability and uniformity with end-to-end > 400 A  (stretch goal)

# Summary

- LANL and STI believe that RCE-CDR process is lowest cost HTS deposition process and can ultimately achieve < $10/kAm Coated Conductor cost (in volume production)
- LANL demonstrated 950 A/cm, $LN_2$ SF, in 6 µm film by RCE-CDR, 400 A/cm in a 1.4 µm thick film
- STI and LANL have achieved results on a simplified IBAD template process, requiring only $Y_2O_3$ and MgO layers
- STI demonstrated 620 A/cm, $LN_2$ SF; 3.5 $MA/cm^2$ in a 0.7 µm film, $LN_2$ SF, on LANL IBAD-MgO templates
- STI is scaling up for longer length CC production