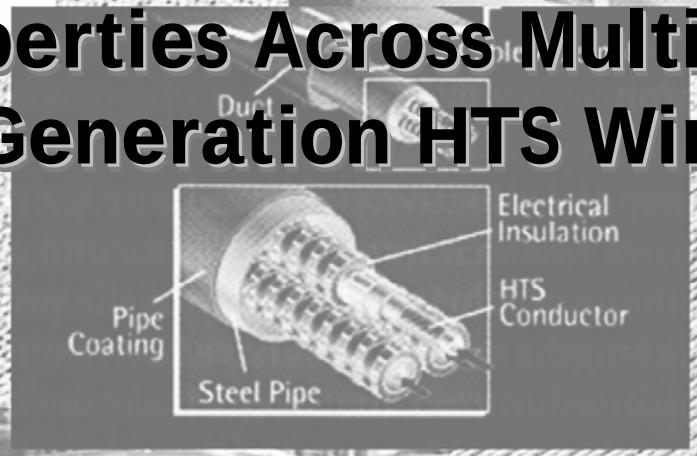
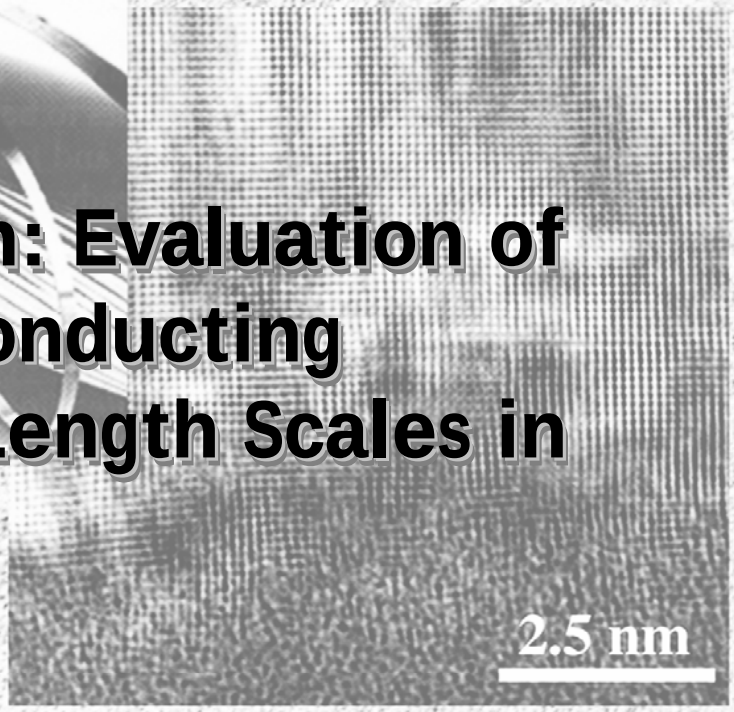
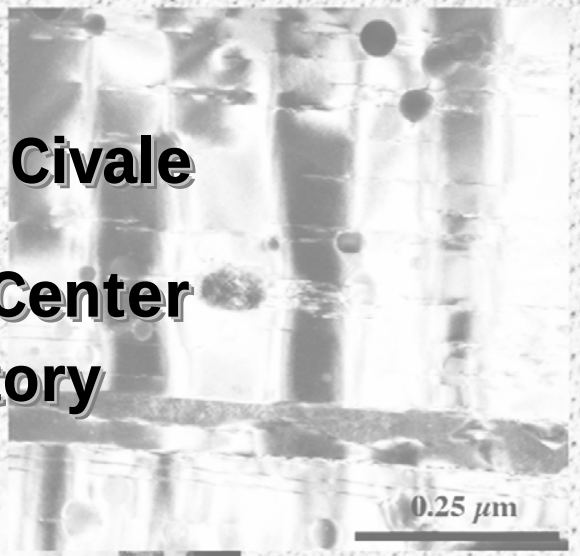
Multi-Scale Characterization: Evaluation of Microstructural and Superconducting Properties Across Multiple Length Scales in 2nd Generation HTS Wire.







Terry Holesinger and Leonardo Civale

Superconductivity Technology Center
Los Alamos National Laboratory

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Outline

- Perspective

American Superconductor™

- Understanding the microstructure development and conversion process for thick, high-I_c MOD YBCO films.

SuperPower Inc.

- Trouble-shooting a wire production issue.
- Characterizing the microstructural development of high-I_c MOCVD YBCO films.

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- Targeted Wire Development
  - $Sm_{1-x}Zr_xO_y$ buffer layers for IBAD MgO
  - Ultrafine Multilayer Structures

- Summary – Results section summarized by Leonardo Civale

Los Alamos has a substantial set of unique capabilities for characterizing HTS wires over a wide range of length scales.

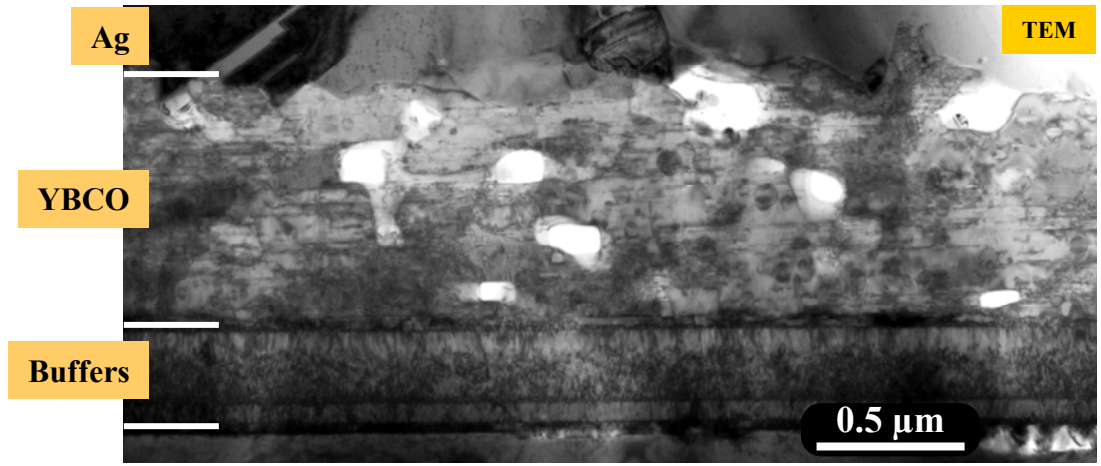
- LANL has always had a strong characterization effort to support industrial, national laboratory, university, and internal HTS development programs. This year, the characterization effort was formally emphasized as a new talk in the 2nd generation wire session because of its importance in wire development.
- We have developed and maintained capabilities and expertise that do not exist in the U.S. superconductor industry.
  - Electron microscopy facilities
  - Extensive electrical characterization facilities
- Long length wire manufacturing is now clearly in the hands of U.S. Industry.
- It is now more important to ensure U.S. industry success via long-length wire characterization and trouble-shooting.
- Continuous wire development capabilities at LANL are used to target advances in new materials, wire processing, and strategic research. (It is important to keep this capability)

Many deposition processes have been adapted for depositing high-J_c YBCO films for real wire applications.

- Starting to get control of these very complex systems!
- Different microstructures have "enough" pinning to support high self-field J_c, but they can have different responses in applied magnetic fields and different thickness dependencies for J_c.
- Requirements for nano-engineering of tunable superconducting wires.
  - Uniform microstructure
  - Uniform defect density
  - Continuously changeable defect landscape.

Laminar

MOD-BaF_2 YBCO (AMSC) 0.8 µm YBCO film



Columnar

PLD YBCO/ IBAD YSZ (LANL) 1.5 µm YBCO film

Understanding the microstructure-chemistry-property relationships in these complex systems requires a dedicated characterization effort.

Understanding the microstructure and conversion process for thick, high-I_c MOD films.

Questions to Answer

1. What is the microstructure of the precursors?
2. How do the thick films nucleate and grow?
3. What is the chemistry and microstructure through the thickness of the film as a function of time and final product?

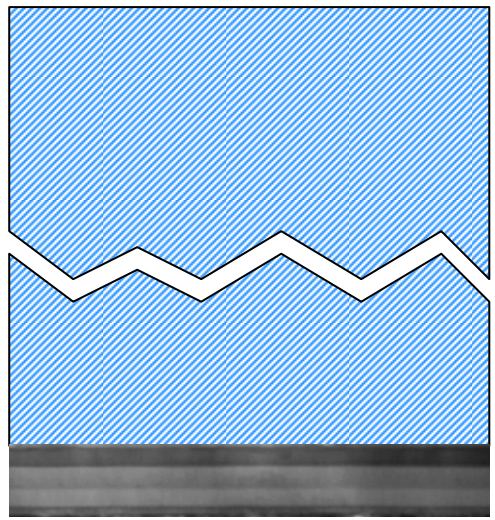
It is a significant materials challenge to find an optimized process for thick (>1 μm), high-I_c MOD YBCO films with uniform, through-thickness properties.

- Thick precursors with the standard high-J_c MOD chemistry developed cracks during the decomposition step.
- Precursor chemistry modification allowed for films > 1.5 μm; however, J_c was too low.
- **Alternative:** Deposit and decompose standard MOD precursors in two or more steps to produce a crack-free precursor that will convert to a thick, high-J_c film.

Goal: Optimize I_c and $J_c(H,\Theta,T)$ while minimizing the number of layers.

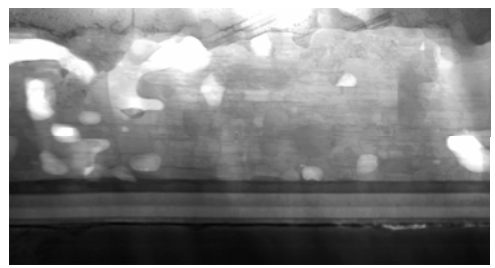
Stages of a 0.9 μm film

MOD Precursor (Liquid) **20 μm**



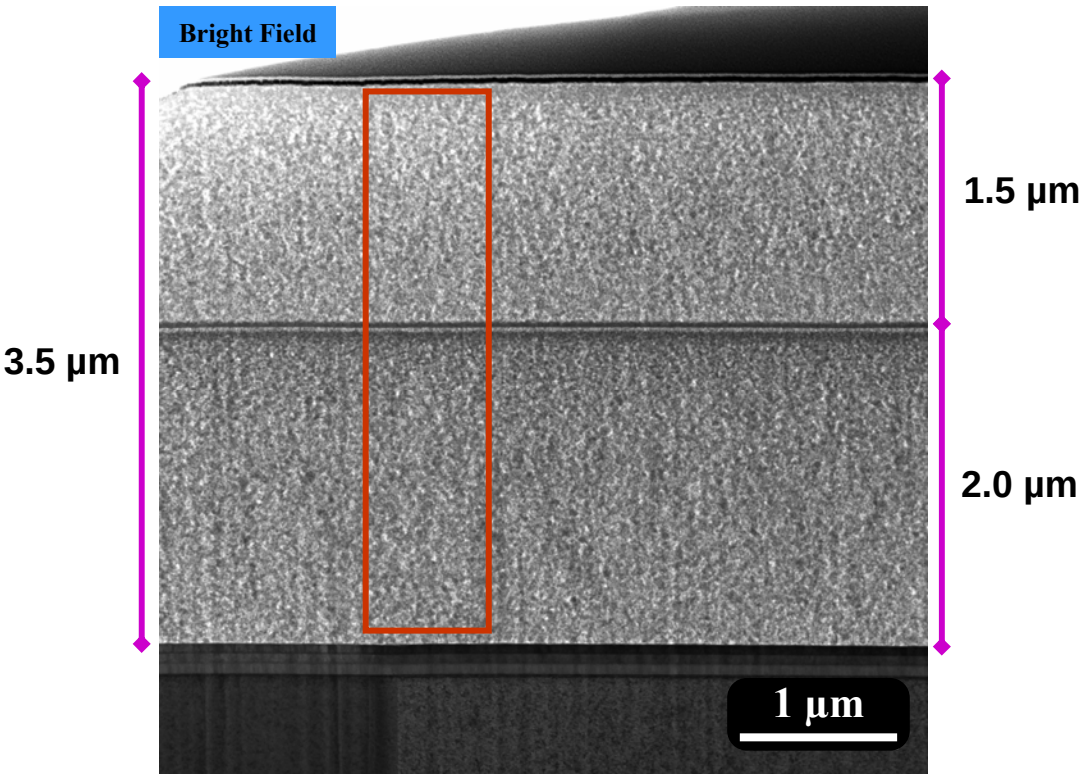
Decomposed Precursor **2.5 μm**

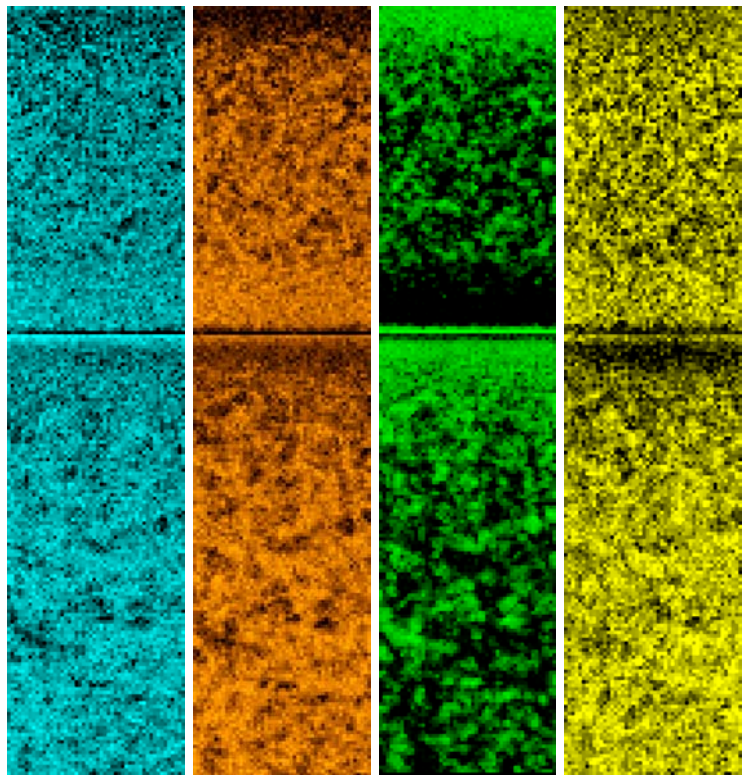
Superconducting Film **0.9 μm**



The conversion of thick MOD films via a multi-coat process is hampered by phase segregation that occurs during the decomposition process.

- First YBCO layer is decomposed before the second YBCO layer is added.
  - **Target**: 1.4 µm fully converted film
  - **Design:** 0.6 µm MOD film on top of a 0.8 µm MOD film.
- The precursor is a nano-crystalline, highly porous mixture of oxides and fluorides.
- During the decomposition process, Cu(O) segregates to the top of each layer; BaF_2 to the bottom.

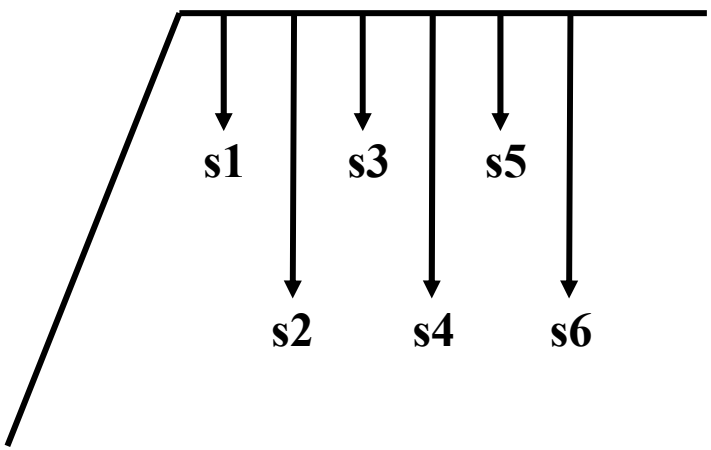




Y Ba Cu F

A quenching study was set up to examine the through-process conversion of YBCO double-coat films (no rare-earth additions).

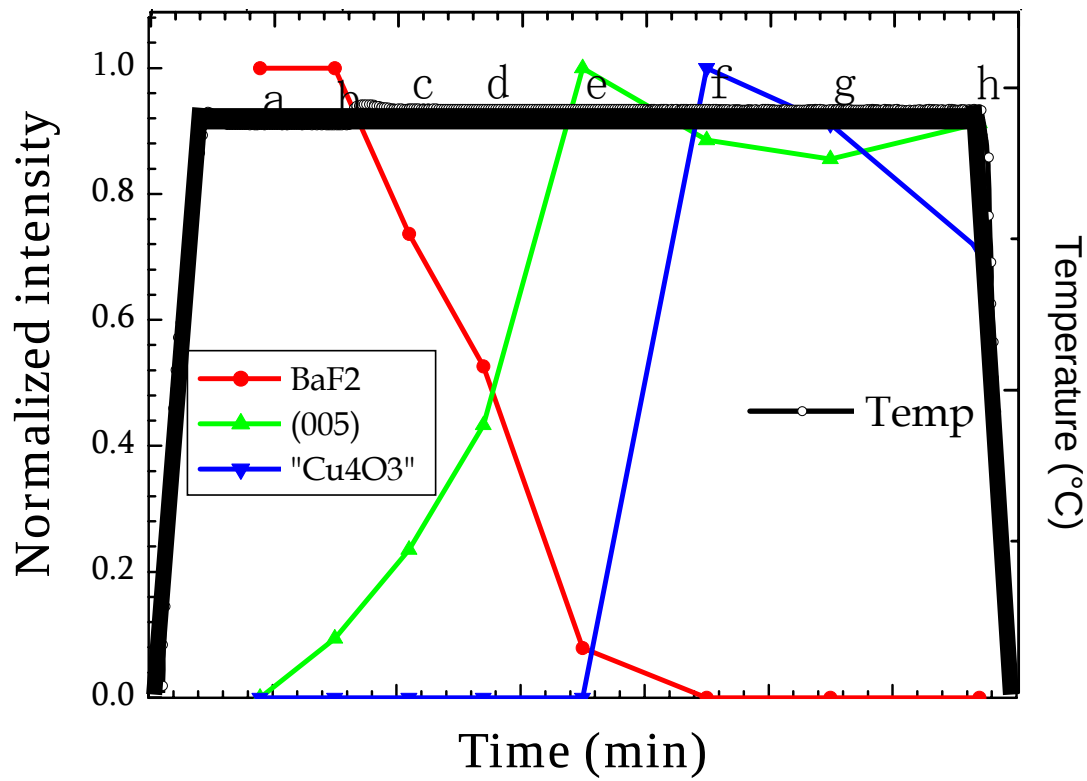
- Samples were quenched after spending specific amounts of time at the conversion temperature. Quench rate estimated at > 300°C/s.



- X-ray diffraction data from AMSC showed three distinct regimes for the conversion process.

- Small samples: complication in that the edges convert faster than the middle.

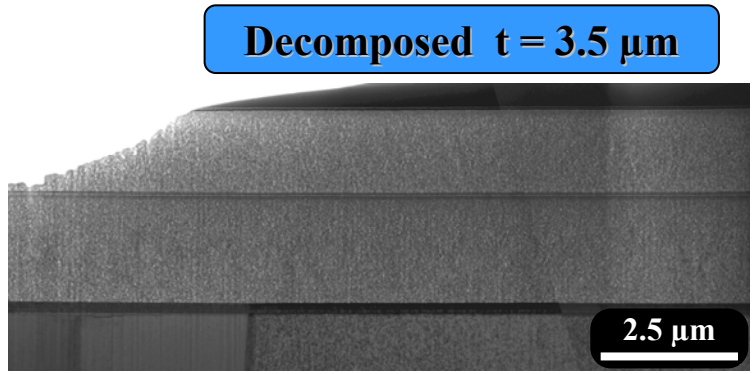
Reaction temperature profile and XRD phase evolution

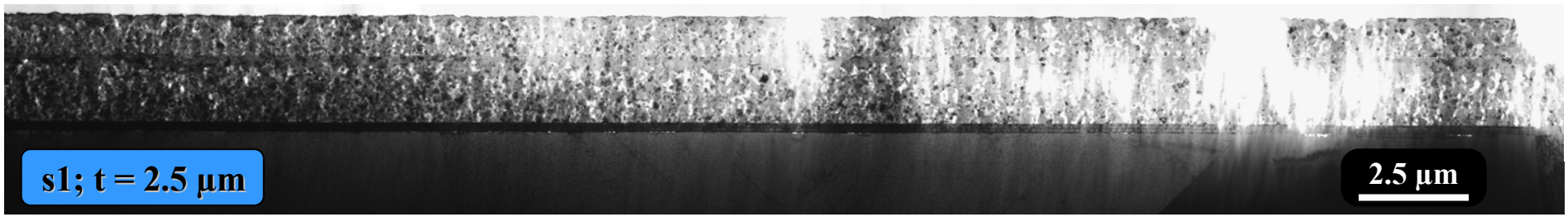


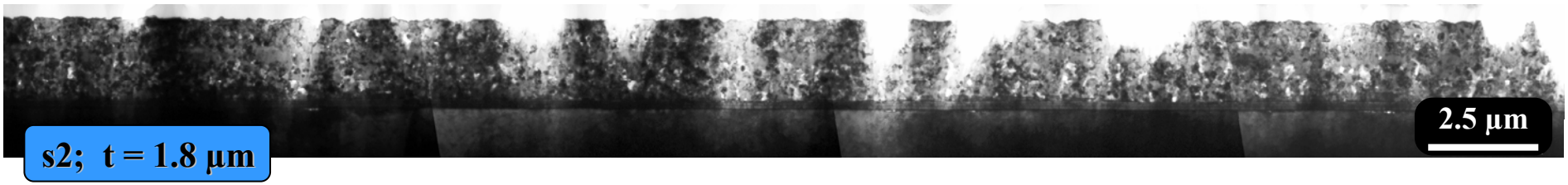
XRD data courtesy Xiaoping Li, AMSC

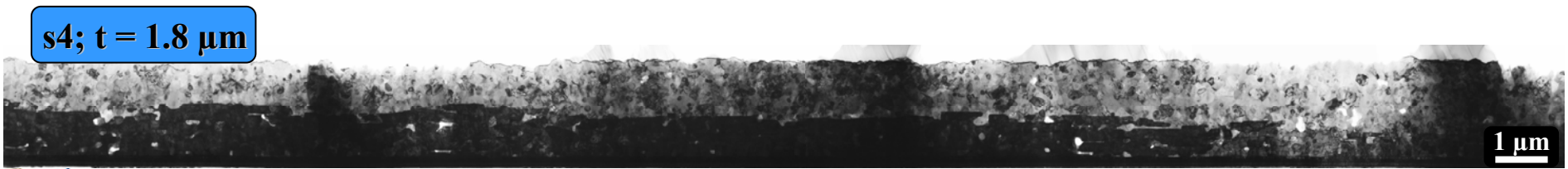
There is a substantial change in the precursor morphology as the conversion process moves beyond the decomposition stage.

- Nearly a factor of two reduction in the film thickness.
- Substantial coarsening of the microstructure.
- The film reaches its greatest density just after the YBCO starts to form at the buffer layer interface.



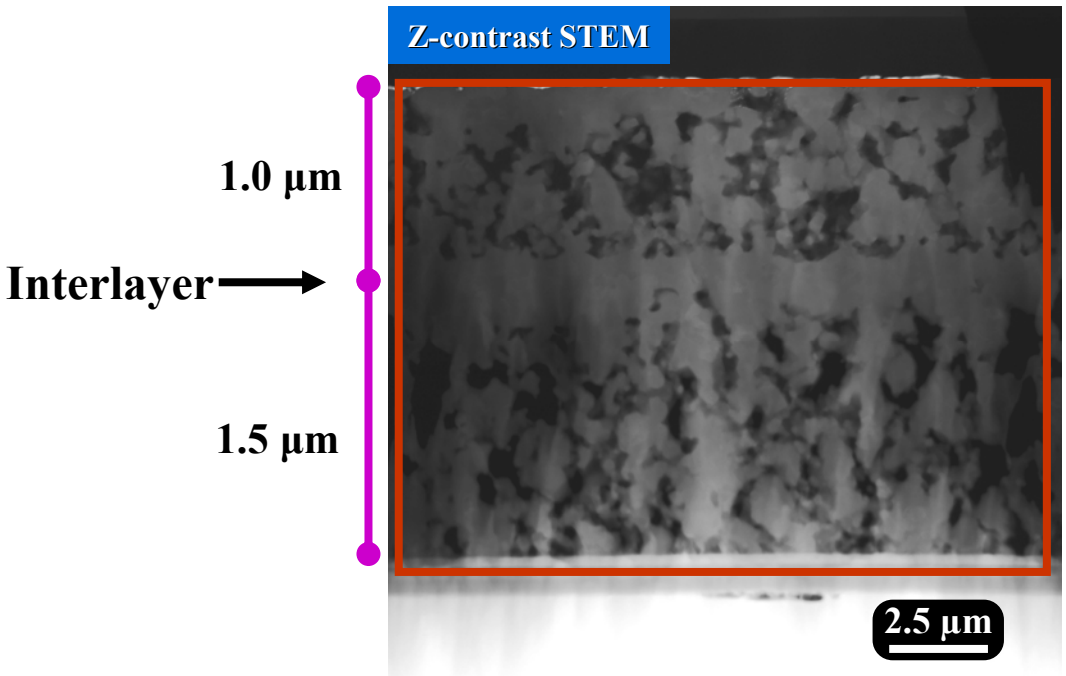


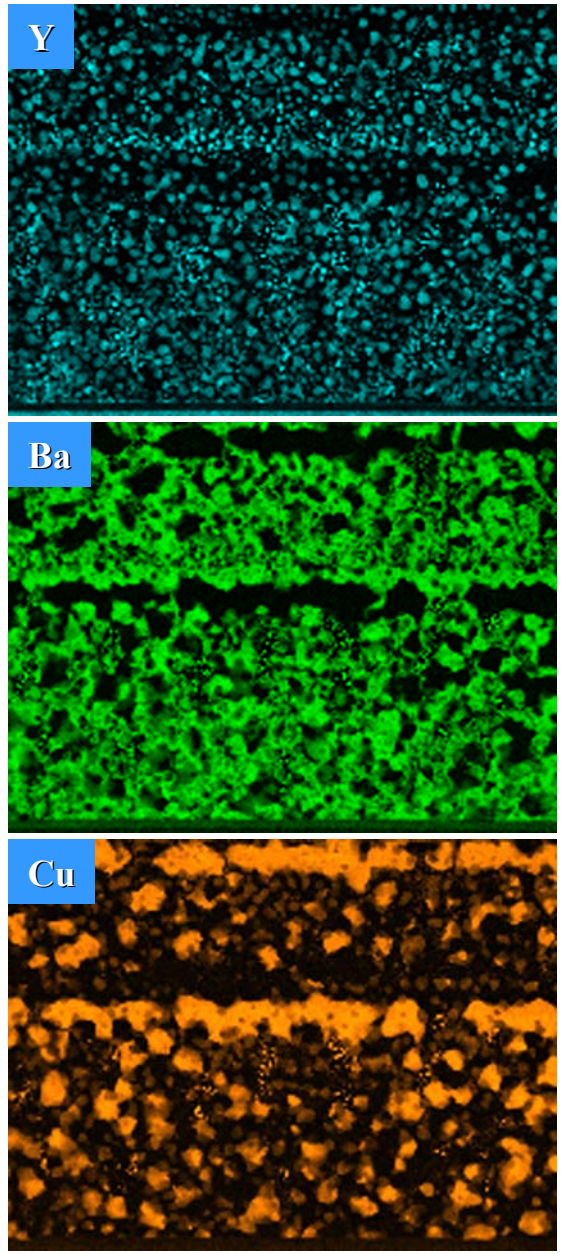




Grain coarsening, thickness reduction, and porosity consolidation are evident at point s1 in the conversion process.

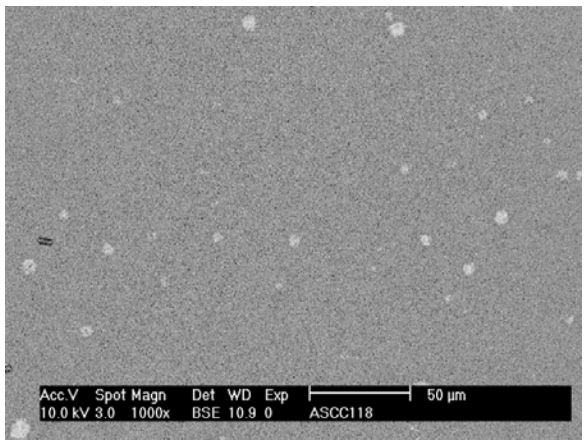
- No YBCO nucleation along the buffer layer interface at this point.
- Precursor shrinkage: 3.5 µm to 2.5 µm.
- Significant coarsening of the precursor, though no surface spots observed yet in the SEM.
- Phase segregation is still present.
- An interlayer structure, defined by the boundary between the two precursors develops further during the high temperature conversion.



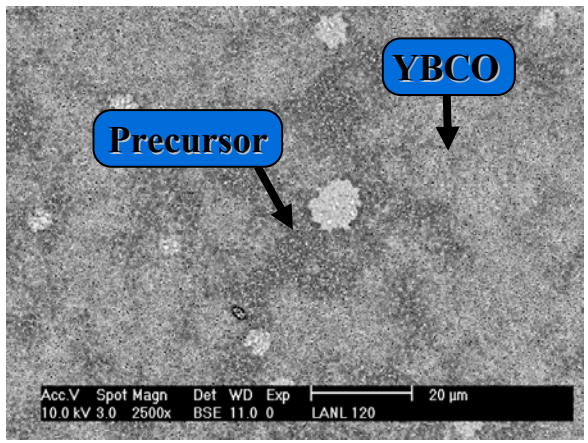


With further time at the conversion temperature, spot structures on the surface of the films were sometimes observed to form.

- Spots are not associated with YBCO nucleation.
- Observed to hinder YBCO formation as the area directly around them is the last to convert.

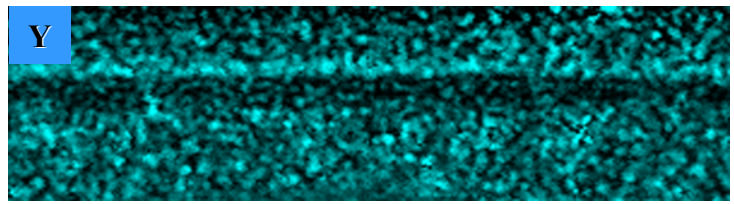


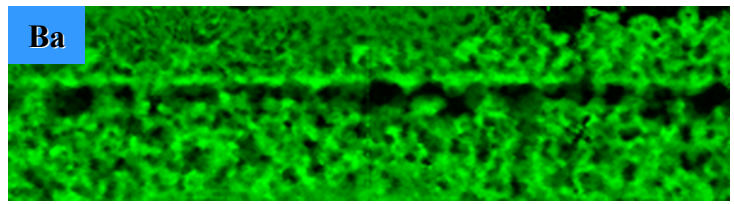
time s3

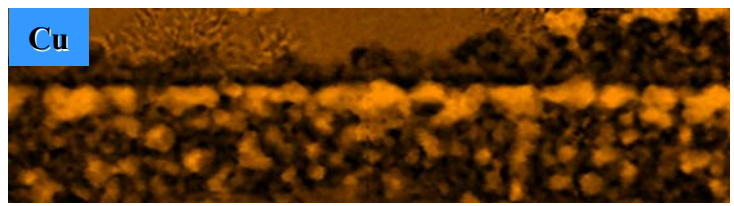


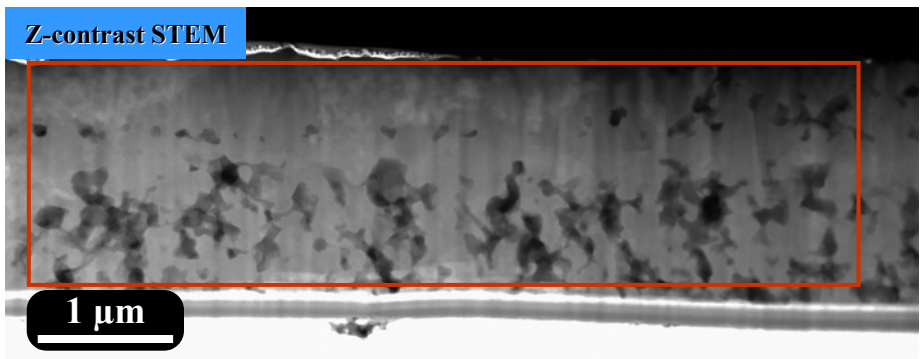
time s5

- Spectral imaging showed the spots to be comprised of Ba-Cu-O-F and Y_2O_3.
- Their formation appears to be associated with Cu-O segregation and they are primarily confined to the top precursor layer.





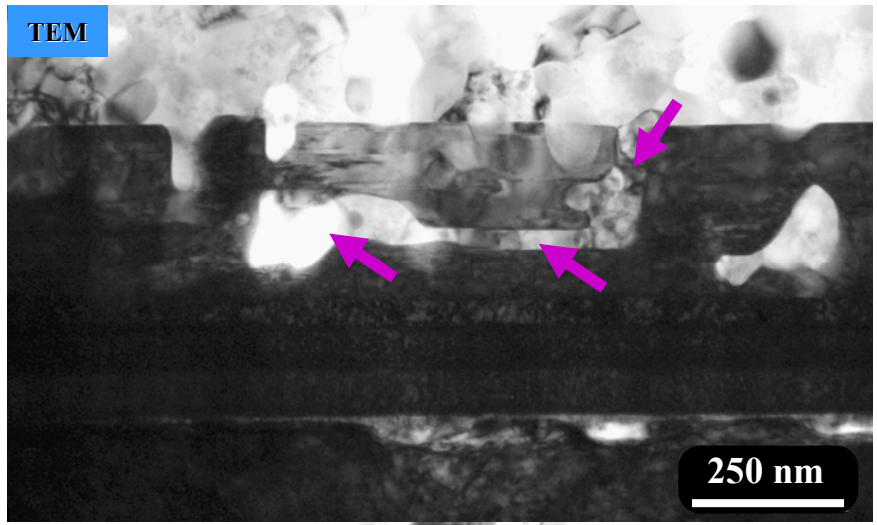


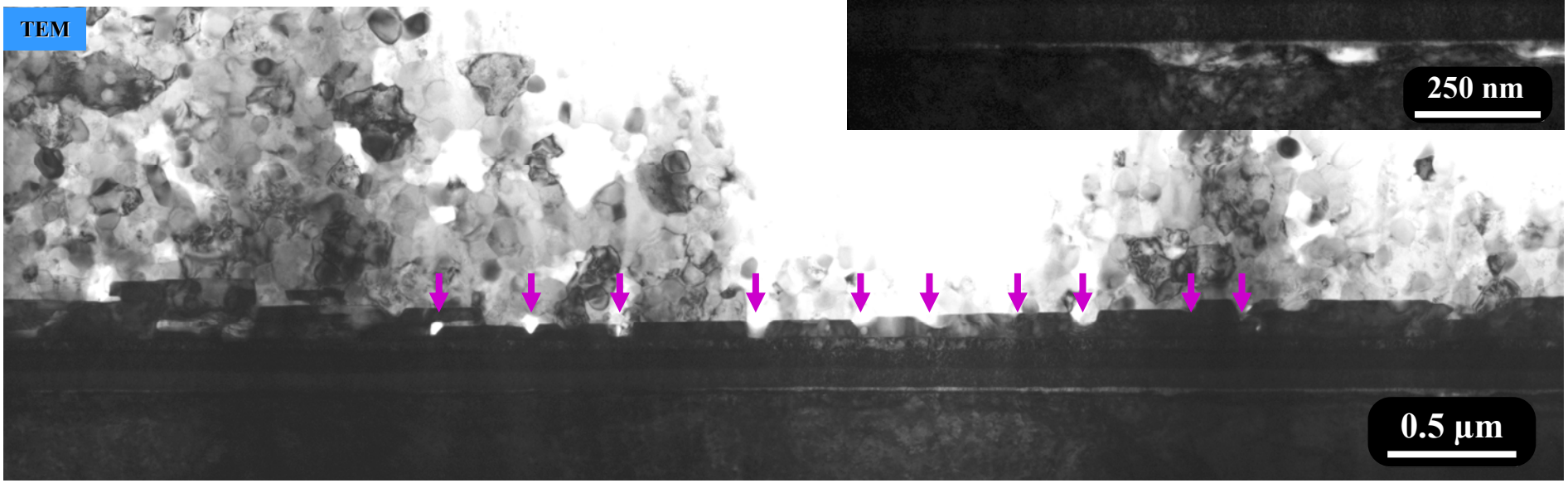


The nucleation of the YBCO layer is a well-ordered event along the buffer layer interface.

- A single-layer of YBCO formed after s2.
- The film is the densest at this point.
- Individual YBCO grains defined by facets
- Space around facets is filled by a Ba-O-F (liquid phase).
- Assume that the nuclei are defined by the facets marked by arrows.
  - grain size: 0.35 µm
  - Nucleation density: 8.2 x 10^8 cm^{-2}

- Mechanism for pore formation is already present as illustrated by the overgrowth of the "liquid" phase by YBCO.



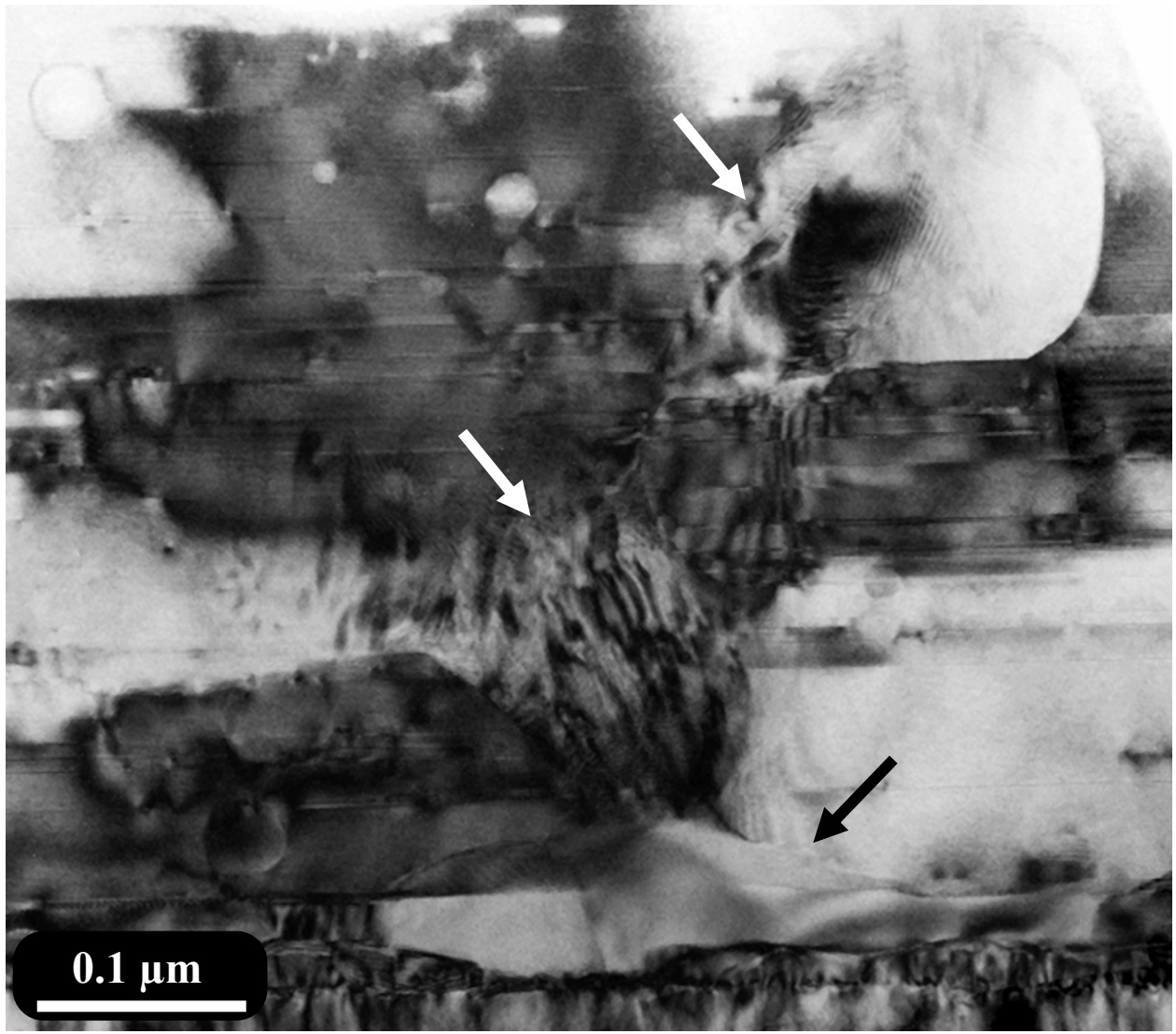


The overgrowth of Ba-O-F liquids during the conversion process can trap these liquids at the interface leading to $BaCeO_3$ formation.

- $BaCeO_3$ formation occurs after the nucleation and alignment of the YBCO film.

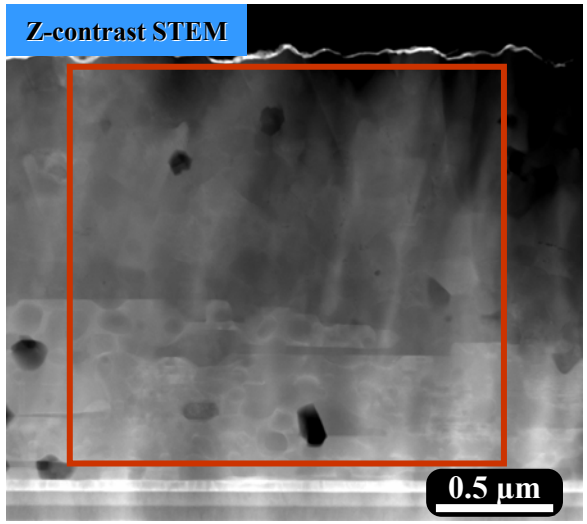
White arrows: Ba-Cu-O

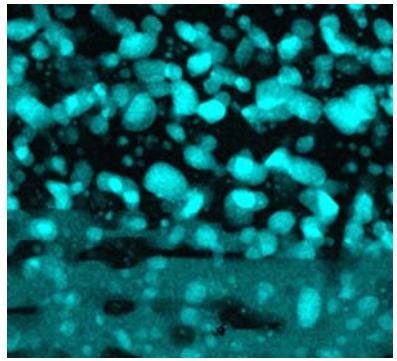
Black arrow: $BaCeO_3$.

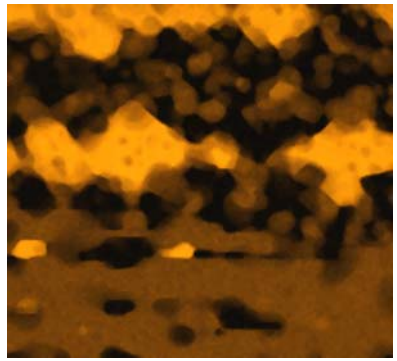


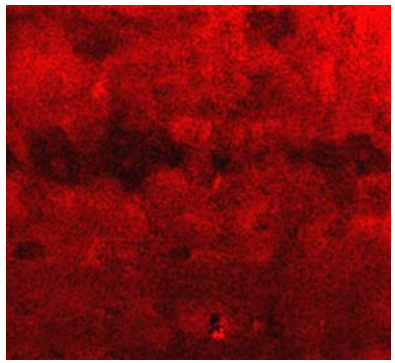
At point s4 in the conversion process, ≈ 0.75 µm of YBCO has formed in spite of the persistent phase segregation.

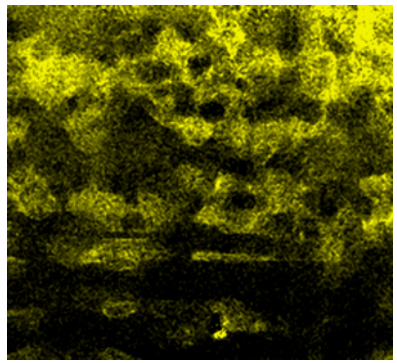
- Layers of Ba-O-F / Cu-O in between the YBCO grains (porosity formation).
- TEM composite image shows a locally variable thickness to the converted portion of the precursor film.
  - $t_{(Ave)}$ = 0.77 µm
  - $t_{(Max, Min)}$ = 1.0 / 0.44 µm
  - σ = 0.14 µm

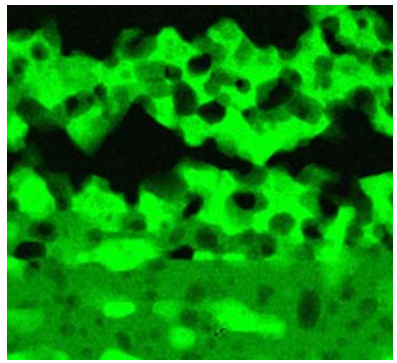


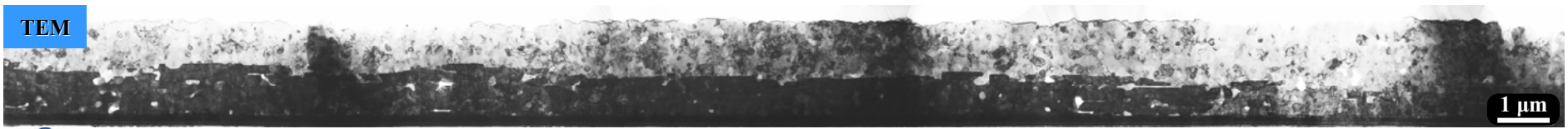






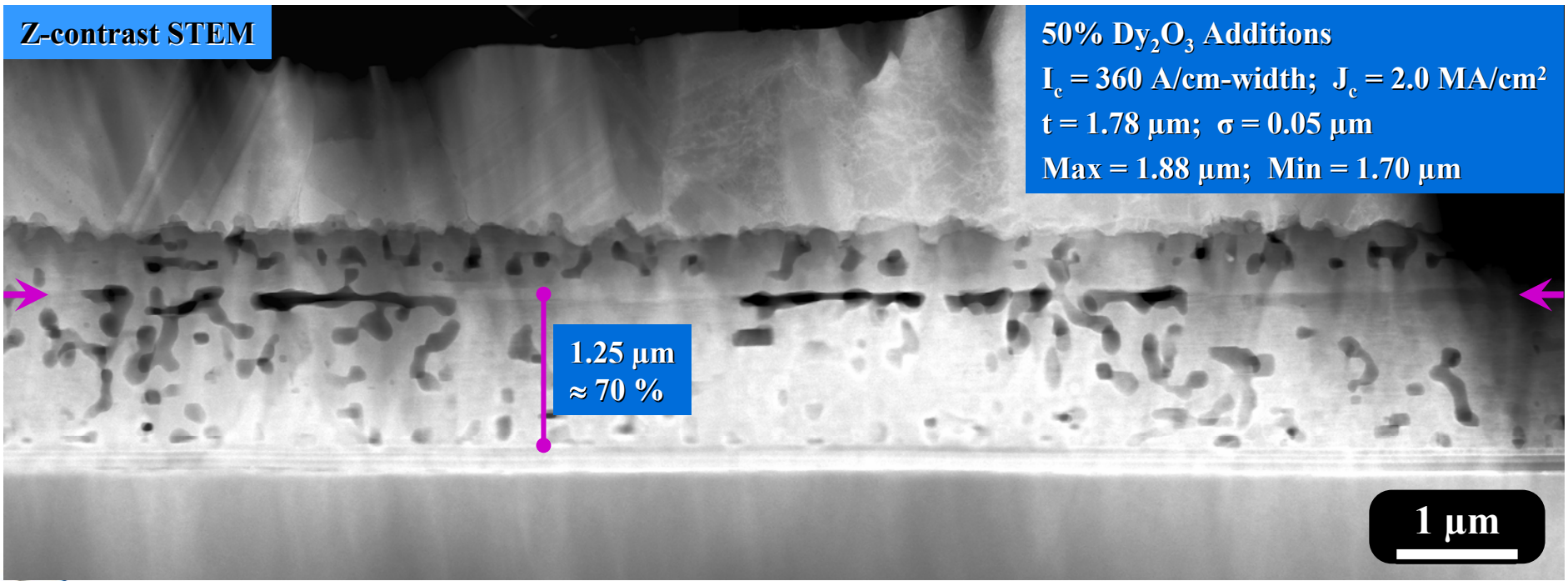






The persistence of phase segregation through the conversion process leads to the formation of a discontinuous or "interlayer" structure in the double-coat films.

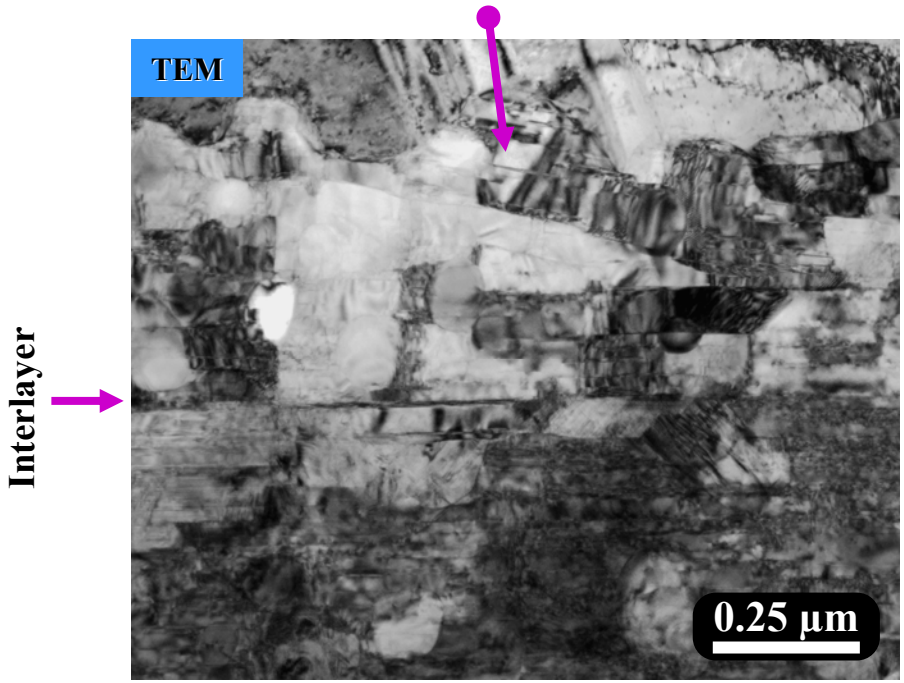
- All films have been found to be thicker than the intended 1.4 µm.
- The interlayer structure is composed of inclusion free YBCO, porosity, and secondary phases (Ba-Cu-O and Cu-O).
- The interlayer position does not correspond directly to the starting internal precursor interface position.
  - In the converted film, the amount of film below the interlayer is ≈ 70-75%.
  - The amount of film below the interface of the two layers in the precursor film is ≈ 55%.



Optimization of I_c in double-coat films necessitates the minimization of misorientations in the remainder of the YBCO film above the interlayer.

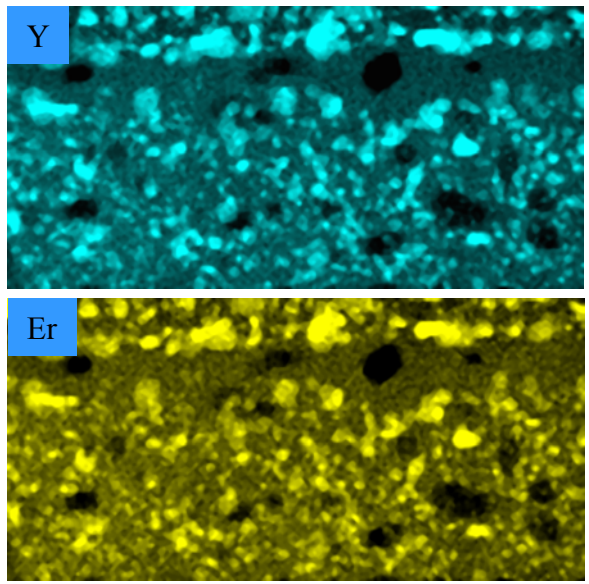
- TEM observation was that the YBCO / YBCO double coat films had more misorientations in the top layer relative to rare-earth addition samples.

Misoriented YBCO



- Howerver, rare-earth additions to both layers resulted in many large secondary phases in the top layer.

Y(Er50%)BCO / Y(Er50%) BCO

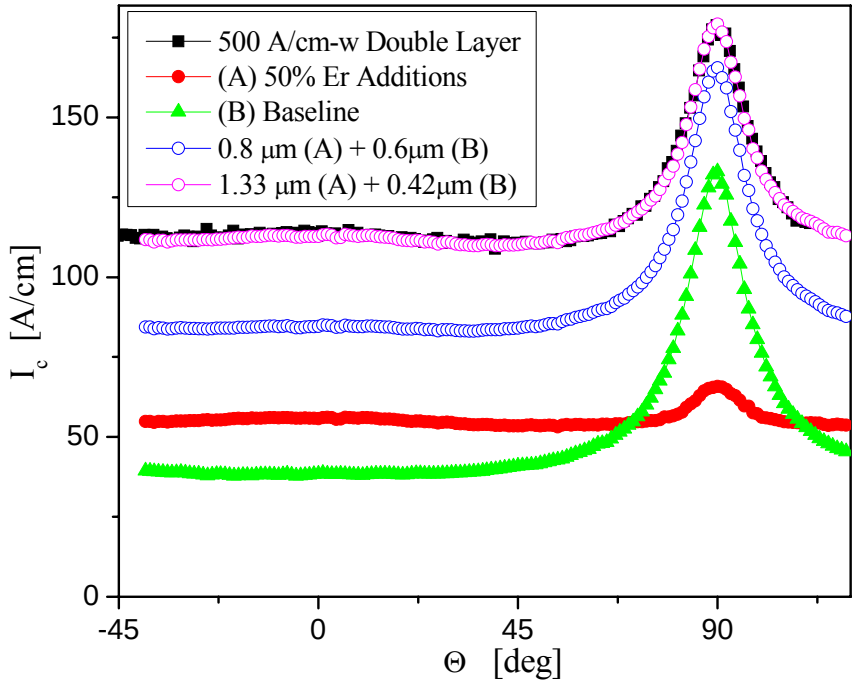


STEM spectral images

The solution, based on these observations, was to develop a hybrid multi-coat film where the bottom layer contained 50% rare-earth additions and the top layer was YBCO.

The culmination of this work was the development of a hybrid double-coat film that met the 500 A/cm-w goal set by AMSC.

- As shown in the WDG presentation, a hybrid double-layer MOD film (0.6 µm YBCO on 0.8 µm Y(Dy)BCO (50% additions)) was developed to meet a 500 A/cm-w goal.



LANL showed that the performance of these hybrid films can be modeled with 0.9 µm MOD film data.

1.33µm (A) + 0.42µm (B)

0.8µm (A) + 0.6µm (B)

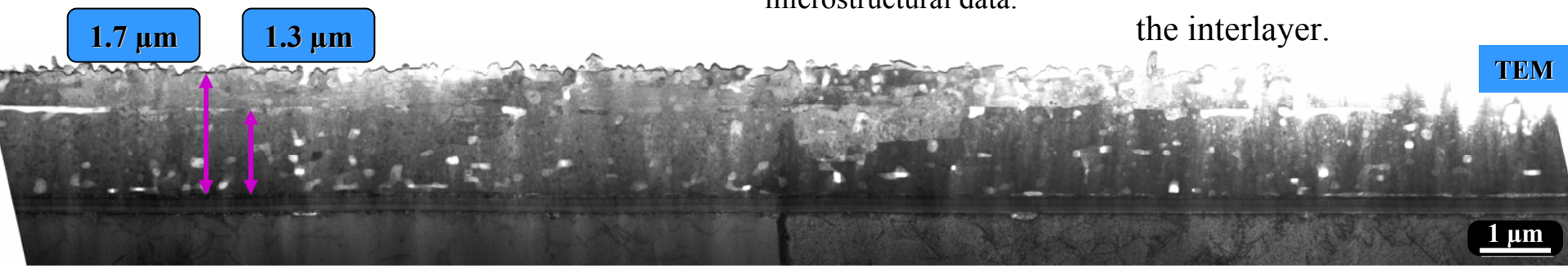
The best fit agrees very closely with the actual microstructural data.

Modeling of the double layer film with the single-layer 0.9 µm MOD film data implies a weak J_c dependence.

Bottom 1.3 µm: nano-dot structure obtained with rare-earth additions to YBCO.

Top 0.4 µm: Y-124 planar intergrowth structure obtained with undoped YBCO.

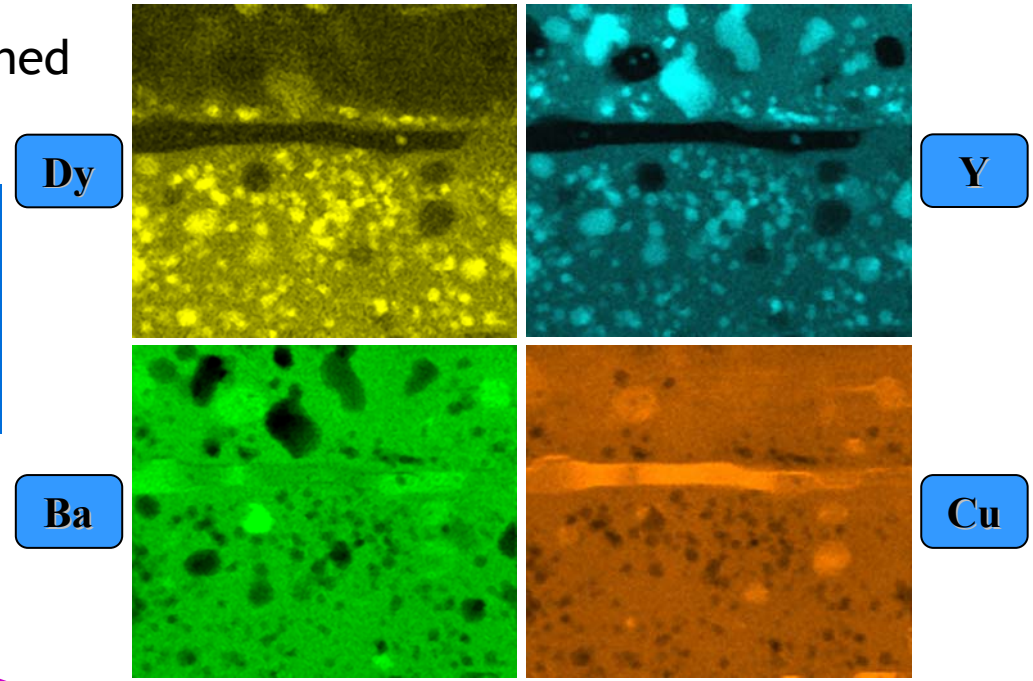
≈ 75% of the film lies below the interlayer.



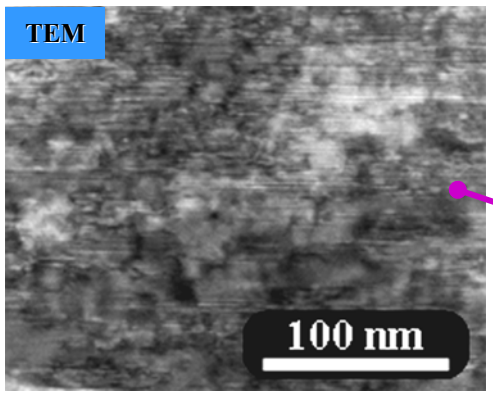
Substrate Grain Boundary

Spectral imaging showed that the different layer chemistries (Dy additions) of the hybrid double-coat films were maintained through the full conversion process.

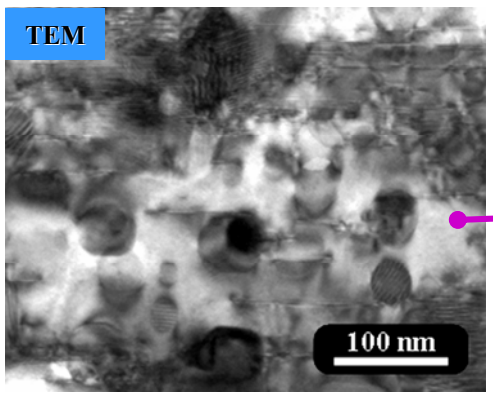
Maintaining these different chemistries through processing opens up a powerful new way to tune the in-field properties of thick, high-I_c MOD films.



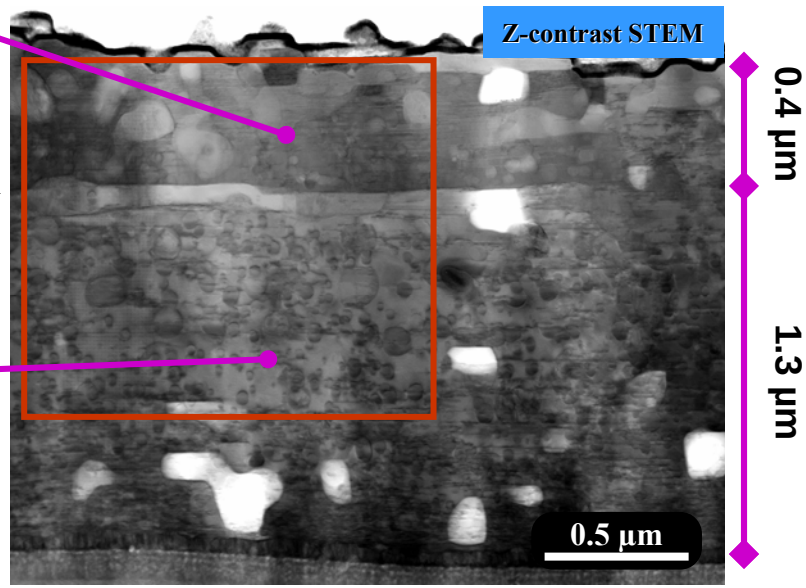
Y-124 planar defect pinning microstructure



Nanodot pinning microstructure



Interlayer



(1) Trouble-shooting a wire production issue.
(2) Characterizing the microstructure of high-I_c MOCVD films.

Questions to Answer

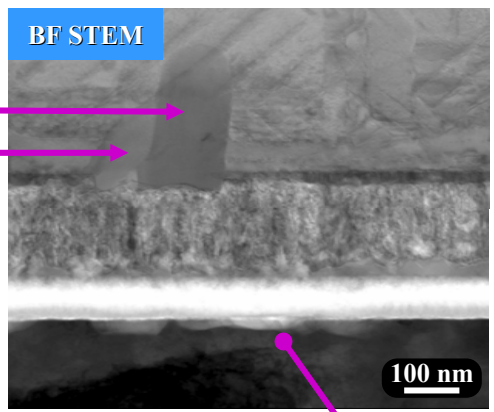
1. What is the structure of the high-performance MOCVD films?
2. Can specific microstructural changes be associated with the optimization of the MOCVD process?

In the past year, LANL personnel traveled to SuperPower to finish the transfer of the IBAD MgO technology for scale-up.

- Ray DePaula and Liliana Stan traveled to SuperPower in Oct. 2005 to help with installation of the IBAD MgO production chamber.
  - Shared their operating experience with RHEED in monitoring IBAD MgO development in-situ.
  - Shared their insights and information on what to look for in high-quality IBAD MgO.
- By Nov.-Dec. time frame, SuperPower was producing 100 m piece-lengths of IBAD MgO on a regular basis.
- Shortly thereafter, delamination and low-I_c problems arose in the production process.
  - LANL also observed this during IBAD MgO development.
  - Typically traced the problem to a contamination issue during one of the processing steps.
- Also, SuperPower made a change in the IBAD MgO process, substituting an alternative buffer layer for the homoeptiaxial MgO step with good reasons:
  - Faster process
  - Very good results in short samples.
- What was the cause of the delamination process?

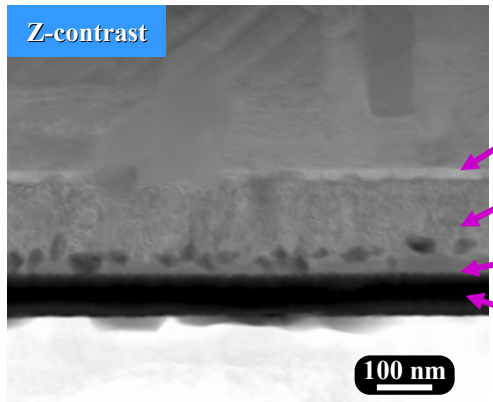
LANL found by STEM/TEM that during continuous processing, an element from the alternative buffer layer diffused into the IBAD MgO layer leaving a layer of porosity along that interface.

- At some points, the LMO buffer layer could not prevent reactions between the alternative buffer and YBCO, which led to misoriented YBCO.

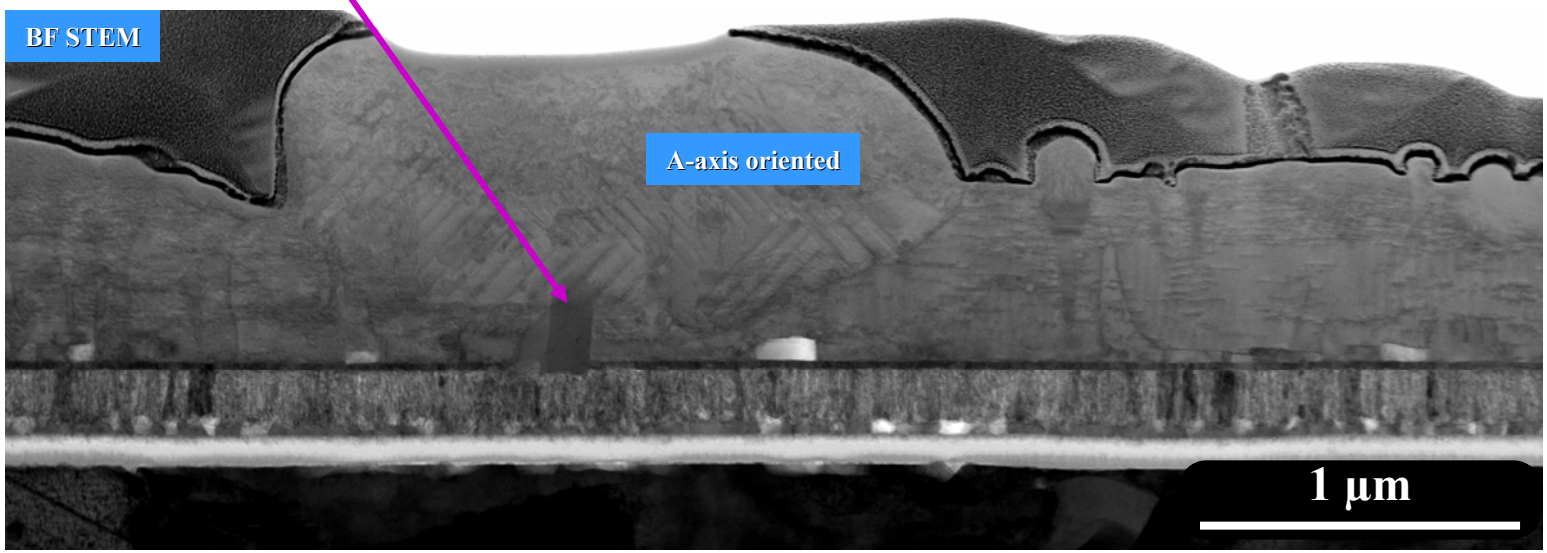


Interfacial reaction phases

Porosity Layer



YBCO
LMO
Alt. buffer
IBAD MgO
Y_2O_3
alumina substrate



Porosity Layer

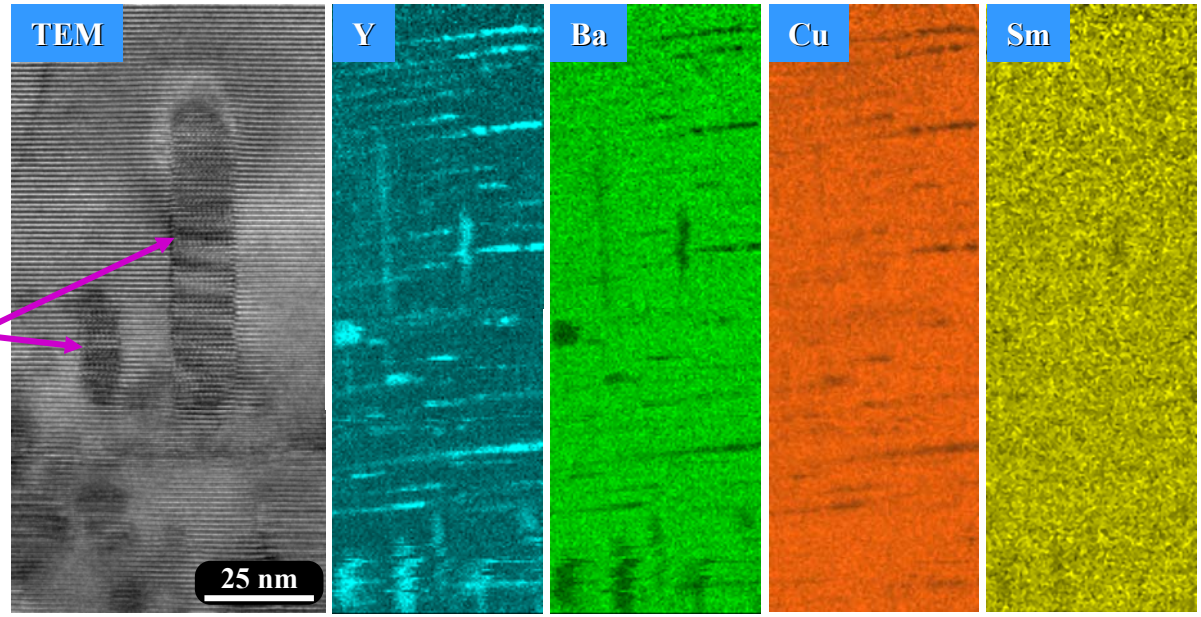
By reverting back to the homoepitaxial MgO layer and modifying the processing conditions (both were needed), SuperPower was able to solve the delamination problem.

- In May 2006, ≈ 7 months after the setup of the IBAD MgO production chamber with help from LANL, SuperPower reported record results for 2G wire.
  - 219 A/cm-w over 322 m.
  - 557 A/cm-w in short R&D sample.

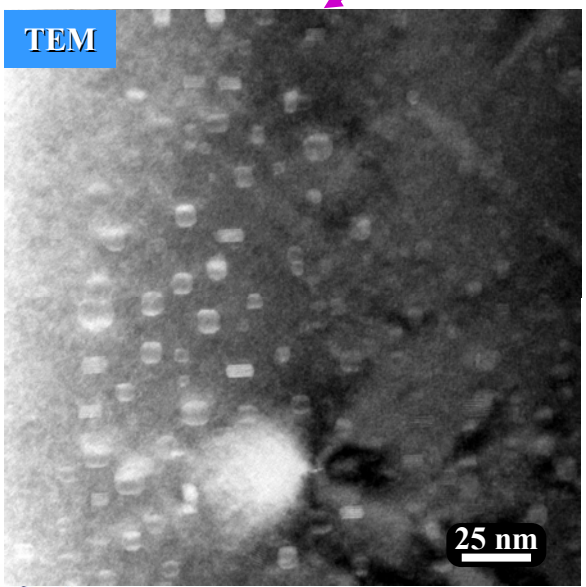
- Finding ways to simplify the IBAD architecture with new materials that have faster deposition rates is still an active area of research which we will discuss later with the $RE_xZr_{1-x}O_y$(RE = rare eath) work at LANL.

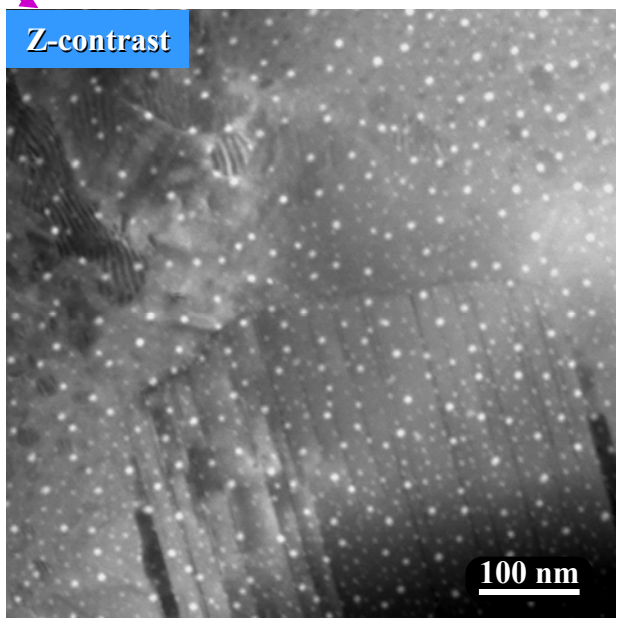
High-I_c SuperPower MOCVD films have a clean microstructure with a uniform distribution of $(Y,Sm)_2O_3$ and (Y,Sm)-Cu-O precipitates.

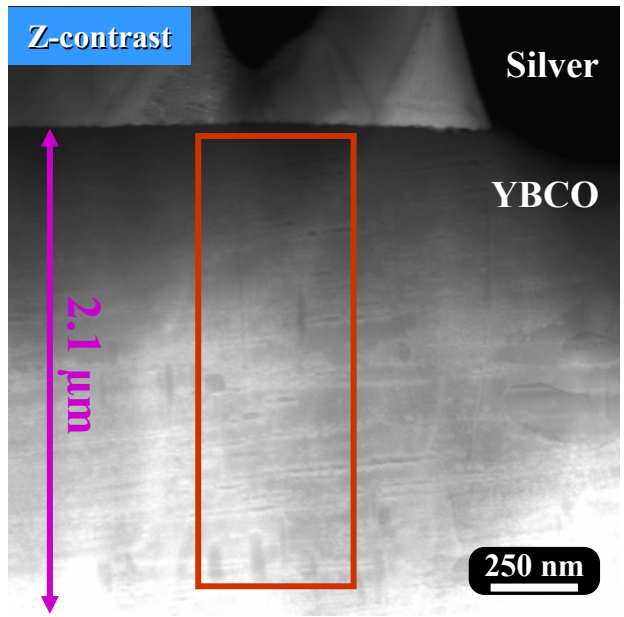
- MOCVD $Y_{0.9}Sm_{0.1}Ba_2Cu_3O_y$
- I_c(77K, SF) = 668 A (557 A/cm-w)
- J_c(77K, SF) = 2.65 MA/cm^2
- Planes or sheets of (Y,Sm)-oxide particles.
- The (Y,Sm)-Cu-O particles are oriented along orthogonal directions in the YBCO.



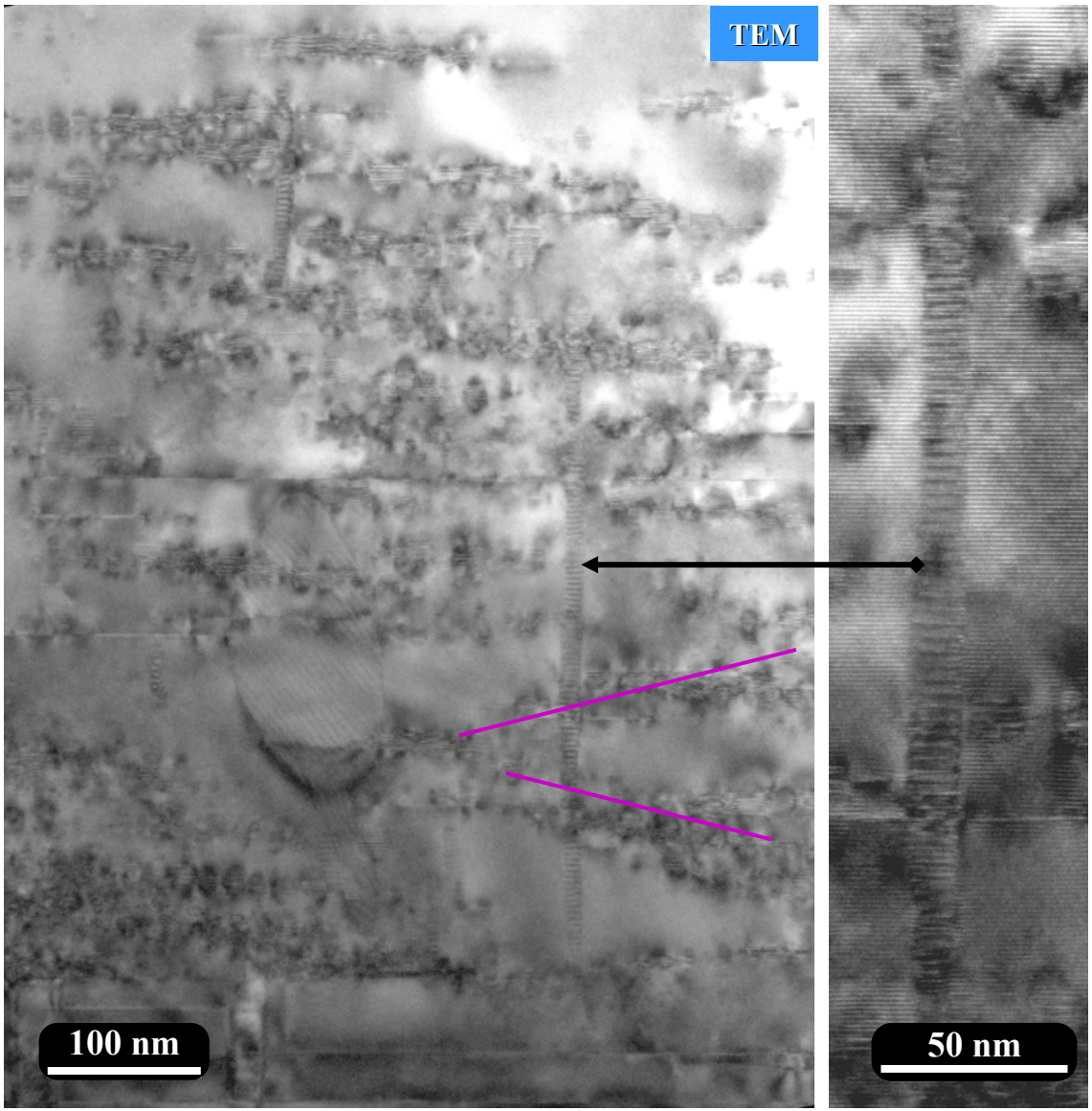
Plan view







The sheets of $(Y,Sm)_2O_3$ oxide particles are actually misaligned to the YBCO by a few degrees.

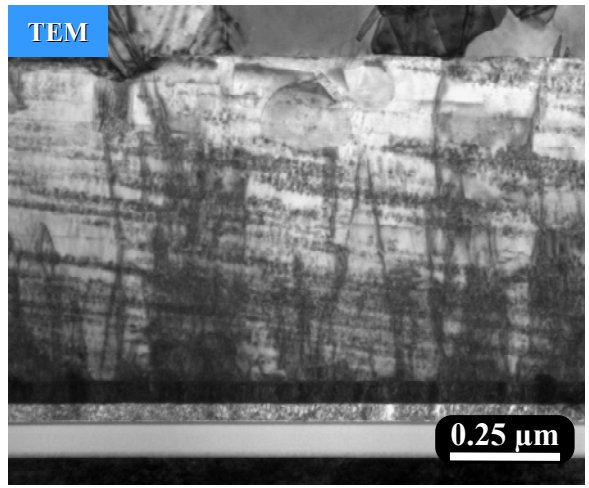


YBCO basal plane orientation

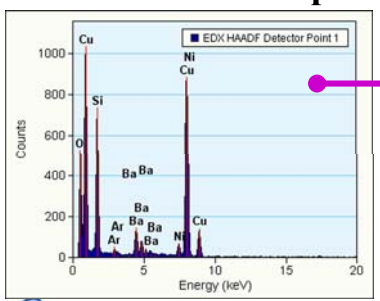
- Diffraction suggests a tilt of 2.15° for the YBCO structure, relative to the buffer layer interface.
- Layers of Y_2O_3 particles are at an angle of 3-7° to the basal plane of the YBCO film.
- The (Y,Sm)-Cu-O can grow into long rods like the one shown at right. (Bamboo structure)
- The (Y,Sm)-Cu-O rods may provide another avenue for improving the pinning in these already high-quality films. (need a higher density)

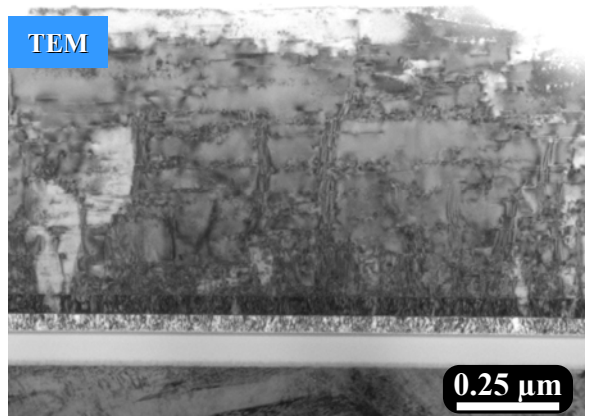
The recent advances in high-I_c SuperPower YBCO films have come about through the control of secondary phase development during deposition during their multi-pass process.

- After the first pass to put down a 0.7 µm MOCVD YBCO film, the structure resembles that of the thick, high-I_c champion film and there are very few secondary phases (CuO) on the surface.
  - $J_c = 4.2$ MA/cm^2; $I_c = 295$ A/cm-w
- In contrast, the previous conditions for MOCVD resulted in large Ba-Cu-O / Cu-O surface particles and a less uniform distribution of internal secondary phases.
  - $J_c = 3.3$ MA/cm^2; $I_c = 230$ A/cm-w



Low current first pass microstructure





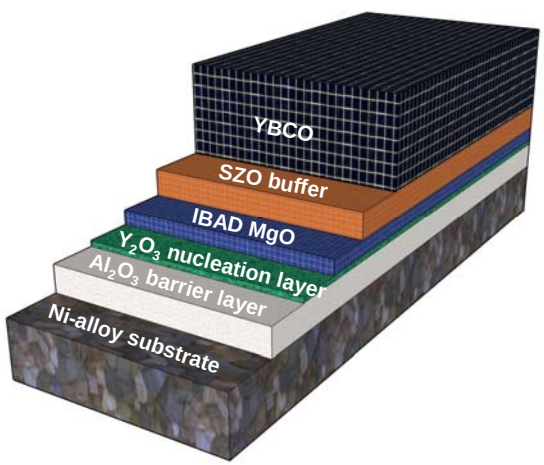
Targeted Wire Development

(1) $Sm_xZr_{1-x}O_y$ buffer layers for IBAD MgO

(2) Ultrafine multilayer structures in wires

Work has continued this year at LANL on a new class of buffer layers, rare earth zirconium oxides ($RE_xZr_{1-x}O_y$), that can be used to replace both the homoepitaxial MgO and intermediate ($SrTiO_3$, $LaMnO_3$, $SrRuO_3$) buffer layers.

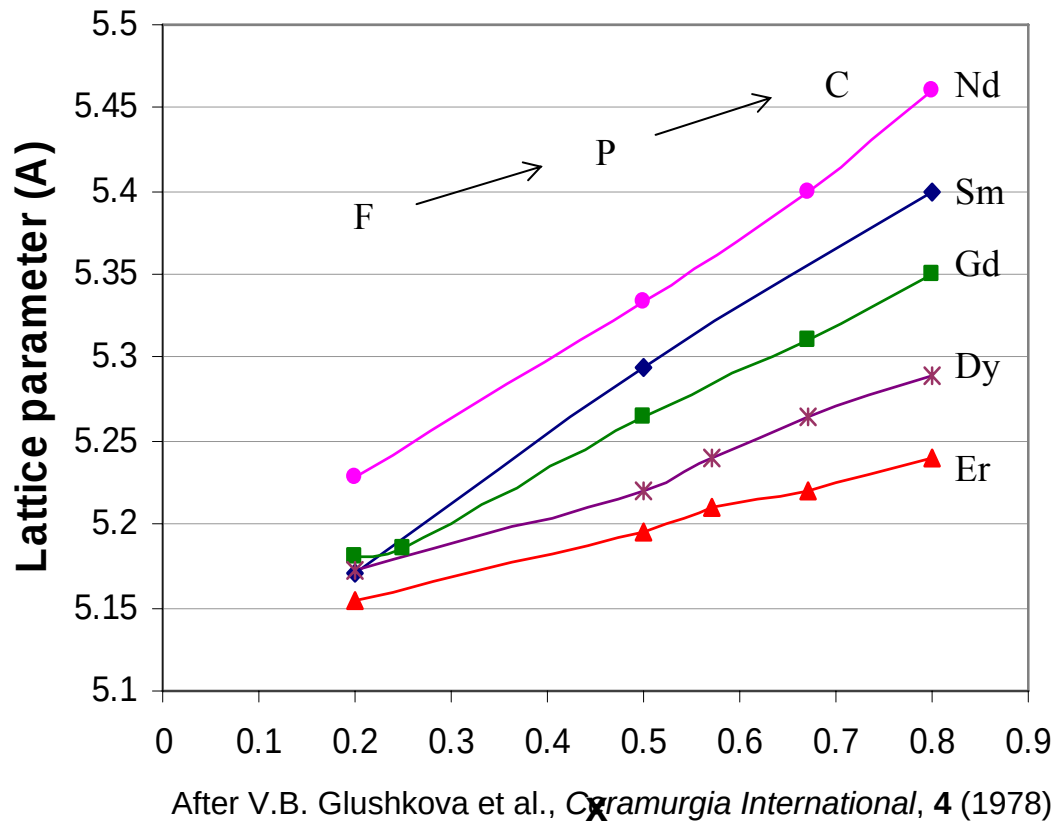
- The $RE_xZr_{1-x}O_y$ materials have high-melting points and are compatible with YBCO.
- Exist in cubic phases over a wide range of stoichiometries.
- Simplify the IBAD MgO architecture.
- Fast deposition by high-rate reactive sputtering from metal targets.



F - defect fluorite structure

P - pyrochlore structure

C - cubic C-type structure

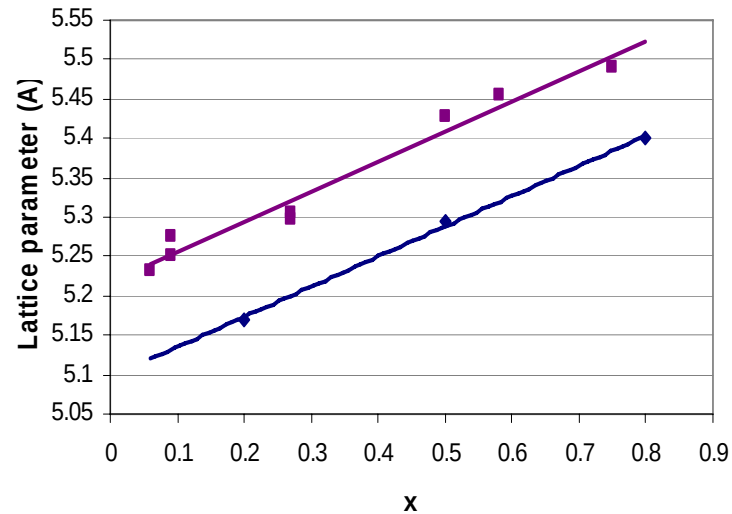


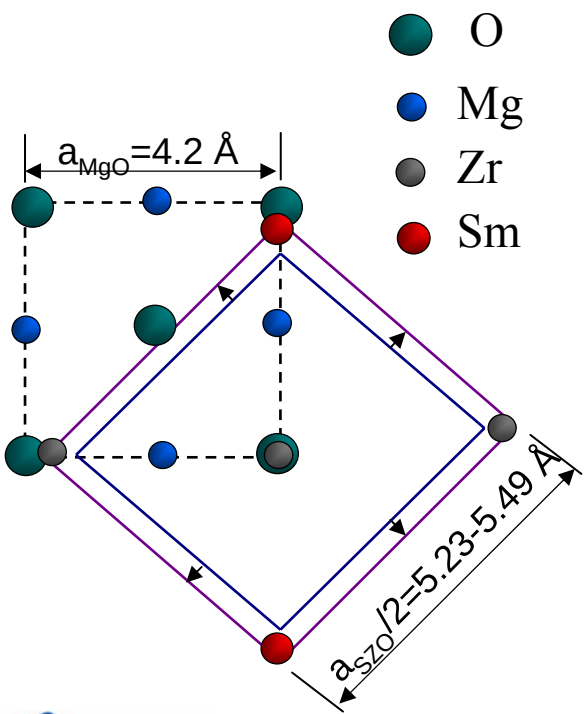
After V.B. Glushkova et al., *Ceramurgia International*, **4** (1978)

Initial studies with $Sm_xZr_{1-x}O_y$ (SZO) showed that epitaxial films could be obtained for a wide range of buffer layer compositions.

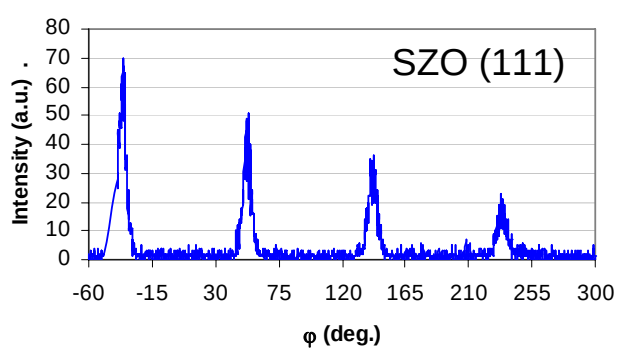
- $(0.06 \leq x \leq 0.75)$
- Lattice mismatch between SZO and MgO is large (7.2% – 12%)
- Lattice constant increases with Sm content.
- Epitaxy could be explained by a domain matching mechanism.

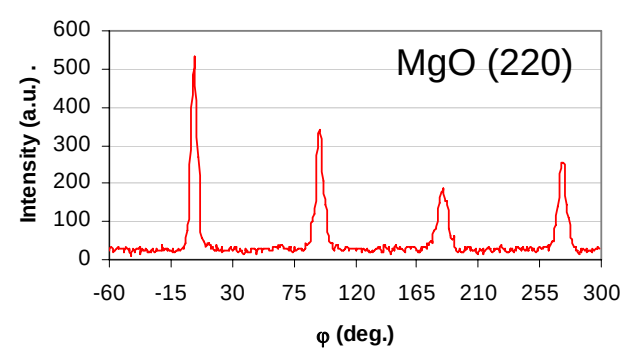
- Lattice constant for SZO on IBAD MgO was found to be larger than the bulk values.





Epitaxial relationship:
$(001)_{SZO}||(001)_{MgO}$
$<110>_{SZO}||<100>_{MgO}$

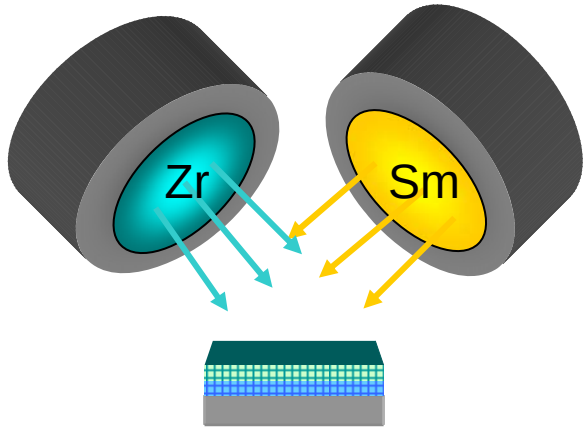




High J_c values (> 2 MA/cm^2) were found for PLD YBCO films deposited on reactively sputtered SZO buffers.

- J_c > 2 MA/cm^2 for the whole range of $Sm_xZr_{1-x}O_y$ compositions that form cubic phases.
  - x = 0.09; J_c = 2.2 MA/cm^2; $t_{(YBCO)}$ = 0.7 µm
  - x = 0.50; J_c = 2.9 MA/cm^2; $t_{(YBCO)}$ = 1.0 µm
  - x = 0.75; J_c = 2.8 MA/cm^2; $t_{(YBCO)}$ = 0.9 µm
- In-plane texture of YBCO was 2.5-3° for x > 0.25.
- Ba was found to react with the SZO, in some cases resulting in the formation of CuO particles at the interface.

RF magnetron sputter guns



Heated substrate

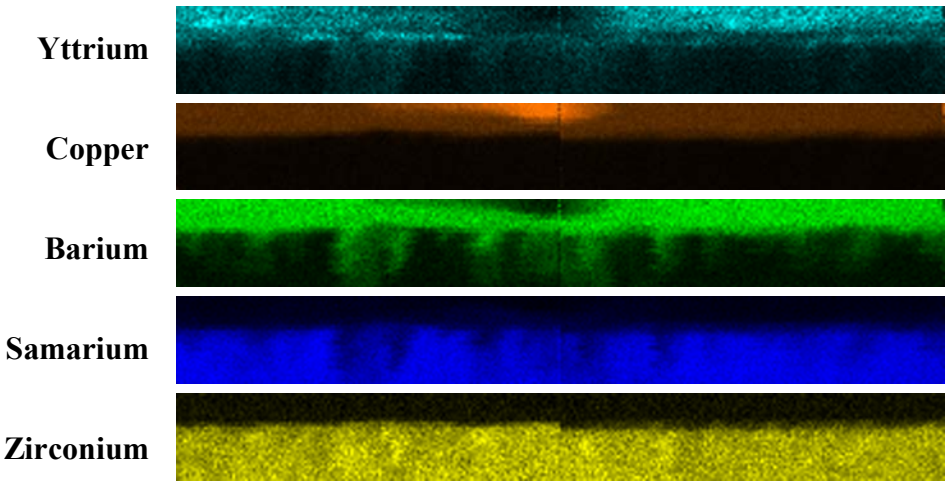
Substrate: IBAD MgO/Y_2O_3/Al_2O_3/Hastelloy

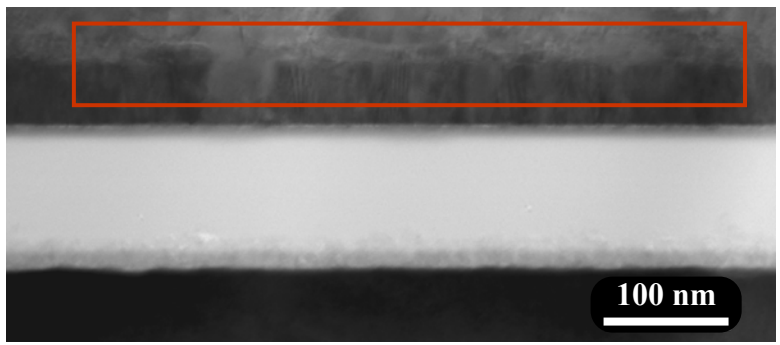
Substrate temperature: 745-760 ºC

rate = 1.5 nm/min.; t = 50 nm

MgO in-plane texture: ~6º FWHM

$Sm_{0.75}Zr_{0.25}O_y$





YBCO

SZO

IBAD MgO

Y_2O_3

***a*-alumina**

The formation of CuO at the interface as a result of the interfacial reaction caused the formation of isolated, misoriented YBCO grains.

- Macroscopically, the YBCO texture is quite good (< 3°)
- Locally, isolated misaligned grains.

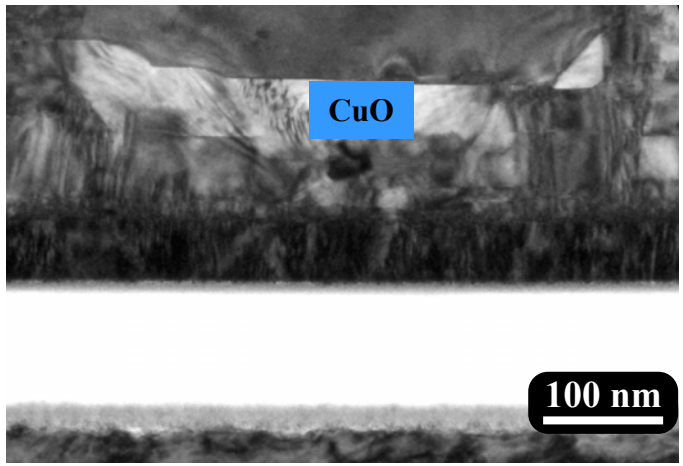
YBCO

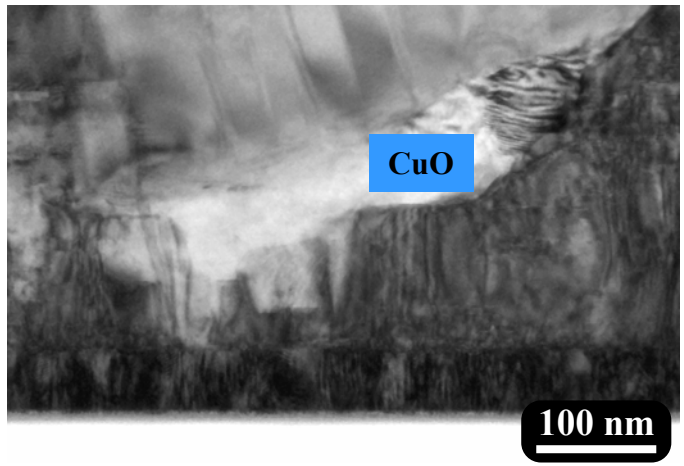
$Sm_{0.75}Zr_{0.25}O_y$

SZO

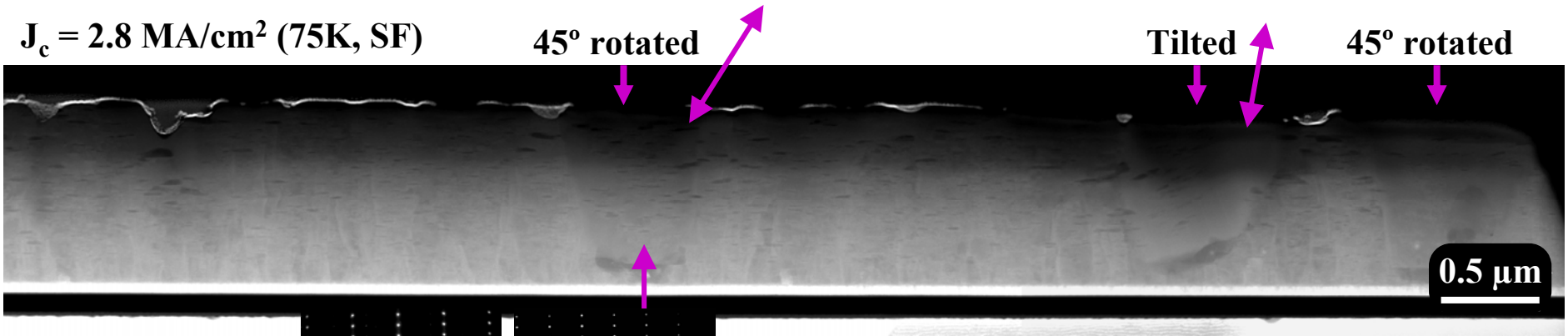
IBAD MgO
Y_2O_3

a-Al_2O_3

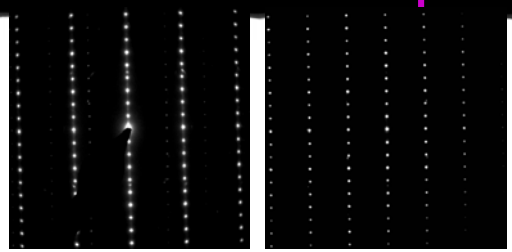




J_c = 2.8 MA/cm^2 (75K, SF)



Main Orientation
[1$\bar{1}$0]

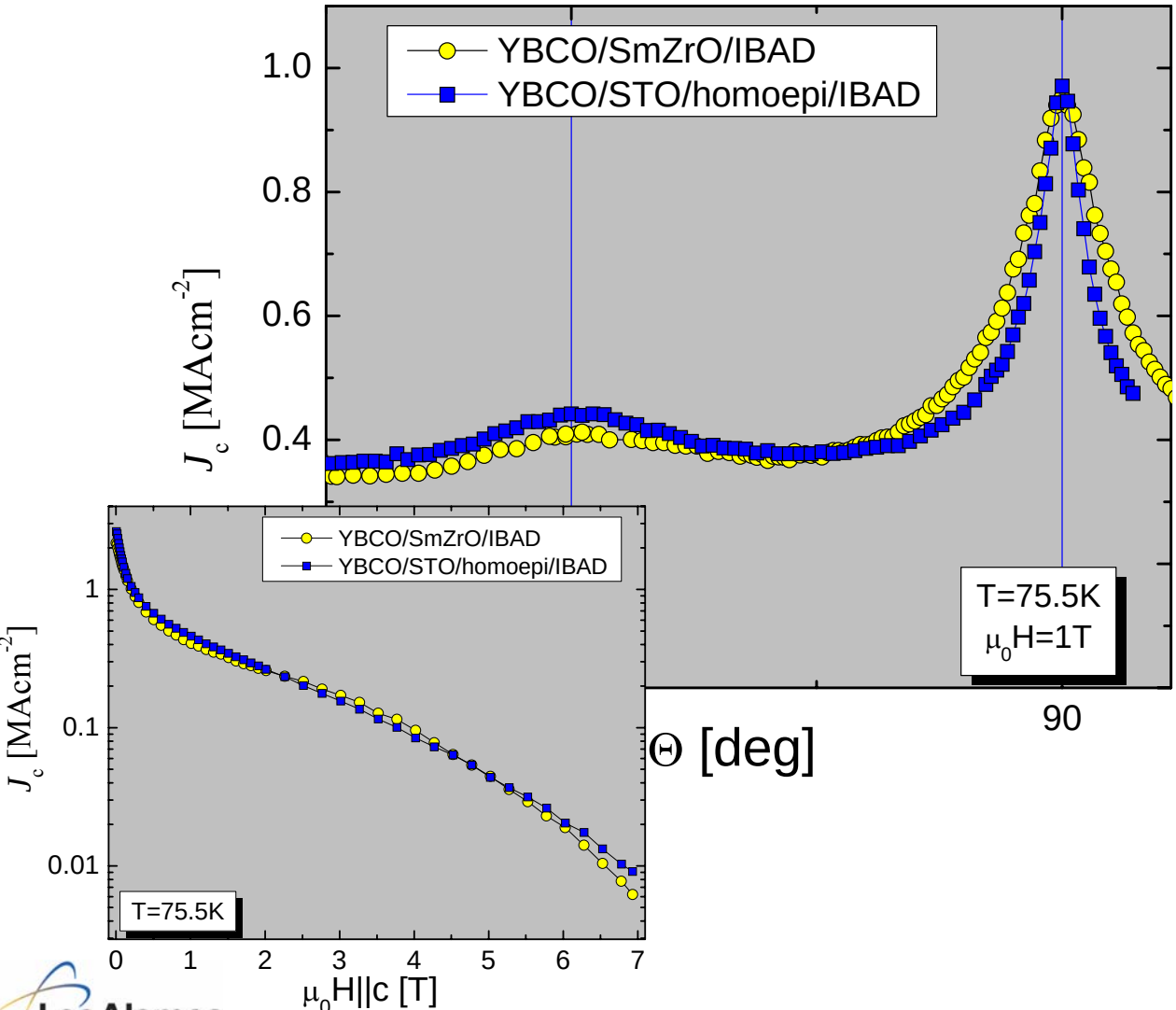


45° rotated
[0$\bar{1}$0]

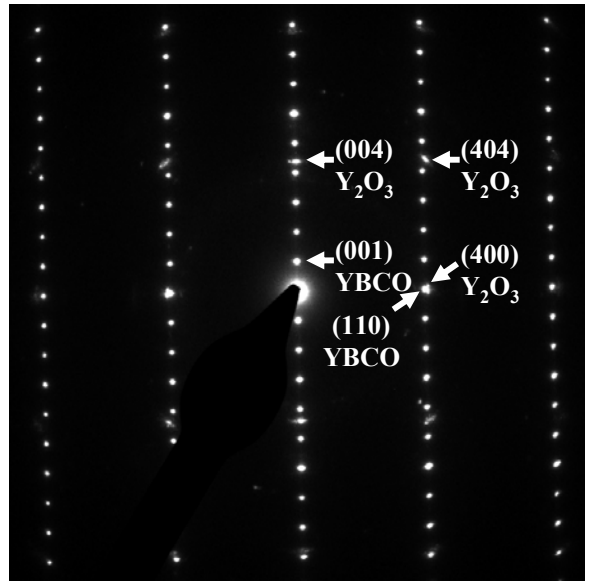
- Localized interfacial reactions are not a limiting factor for SZO use.
- Wide range of compositions with which to minimize reactions.

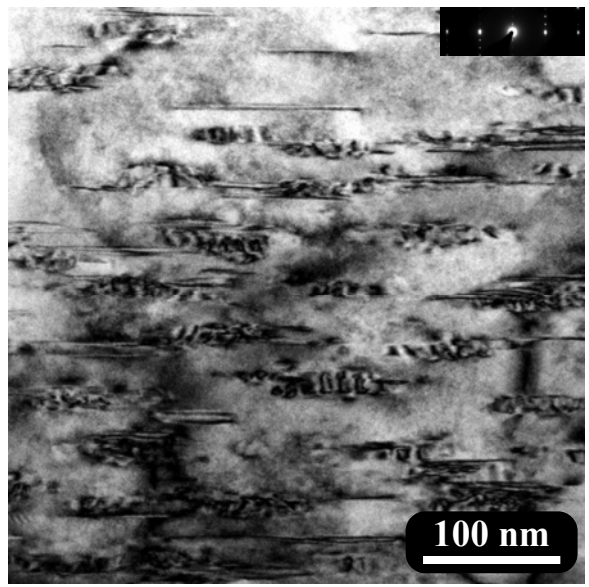
The YBCO films on SZO buffer layers were characterized by a high density of coherent Y_2O_3 particles.

- Field and angular dependence of YBCO on $Sm_{0.5}Zr_{0.5}Oy$ buffer layer is very similar to YBCO on STO buffer layer.



$[0\bar{1}0]$ Y_2O_3 || $[1\bar{1}0]$ YBCO
(400) Y_2O_3 || (110) YBCO





g = <110>

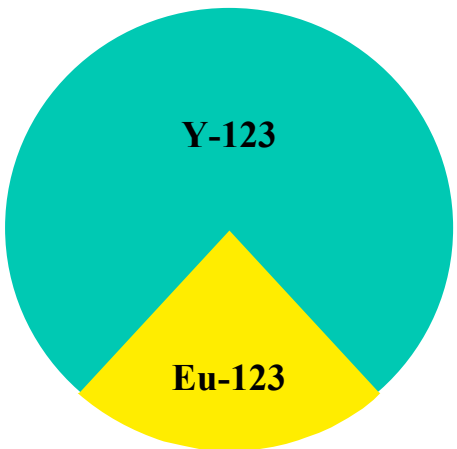
The development of an ultra-fine multilayer process for continuously processed tapes offers a new level of study in the optimization of defect structures and arrangements of inclusions in YBCO wires.

- Both AMSC and SuperPower rely on inclusions within their films to provide extra pinning for application specific needs.
- Not clear what an optimized defect structure would be in either case.
- LANL will conduct sample development with continuous processing.
  - More uniform sample microstructures with moving tapes.
  - Produce a number of identical samples for post-deposition processing and multiple characterization studies.
- Ultimate goal is the ability to control the size, density, and spacing of inclusions in YBCO films.
  - Try to optimize flux pinning (microstructure) to the application needs.
  - In reality, need to optimize flux pinning structure to each point in the H,Θ,T regime.
  - Need to develop a predictive capability for flux pinning microstructures.

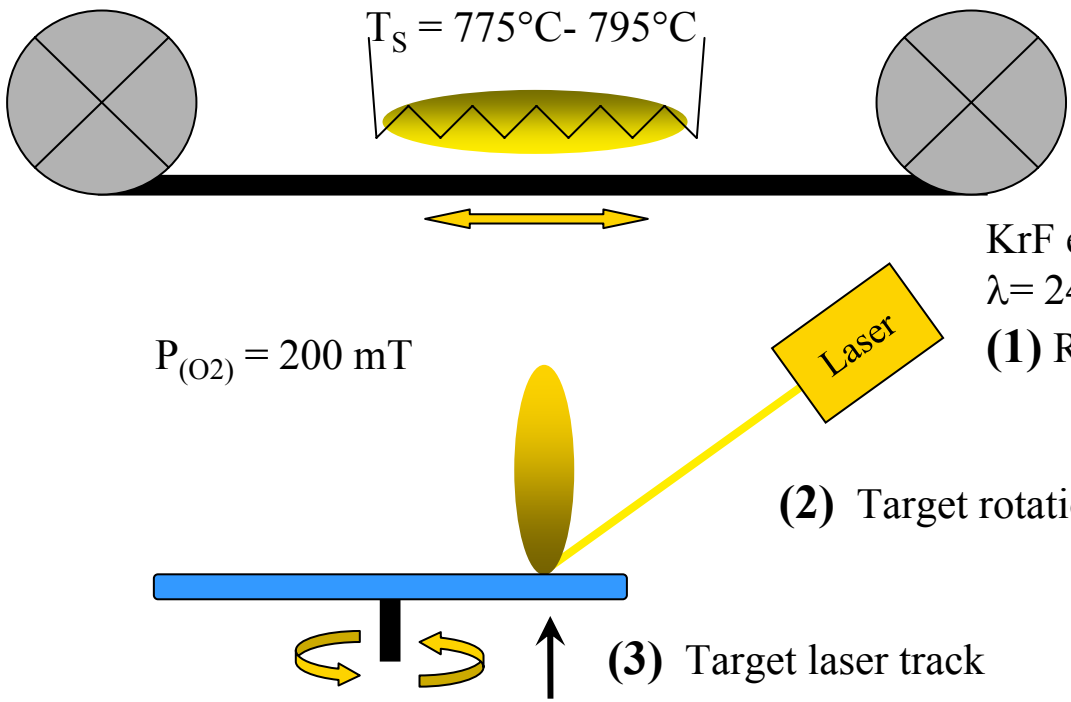
Sectored, monolithic targets allow continuous PLD deposition of ultra-fine multilayer films on stationary or moving substrates.

- PLD is a point-source deposition process; hence the composition of the plume at any one time is spatially correlated to its point of origin on the target.
- Y-123 / Eu-123 sectored targets were developed for multilayer deposition.
  - Eu-123: a=3.897Å b=3.838Å c=11.710Å
  - Y-123: a=3.886Å b=3.818Å c=11.680Å

High Purity oxides and carbonates.

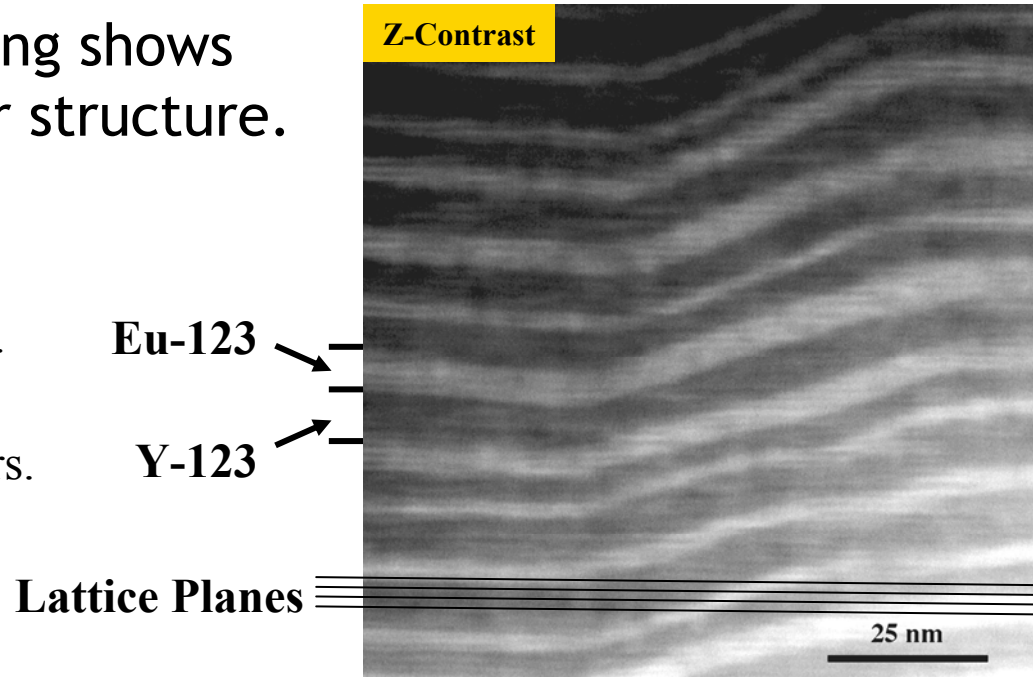


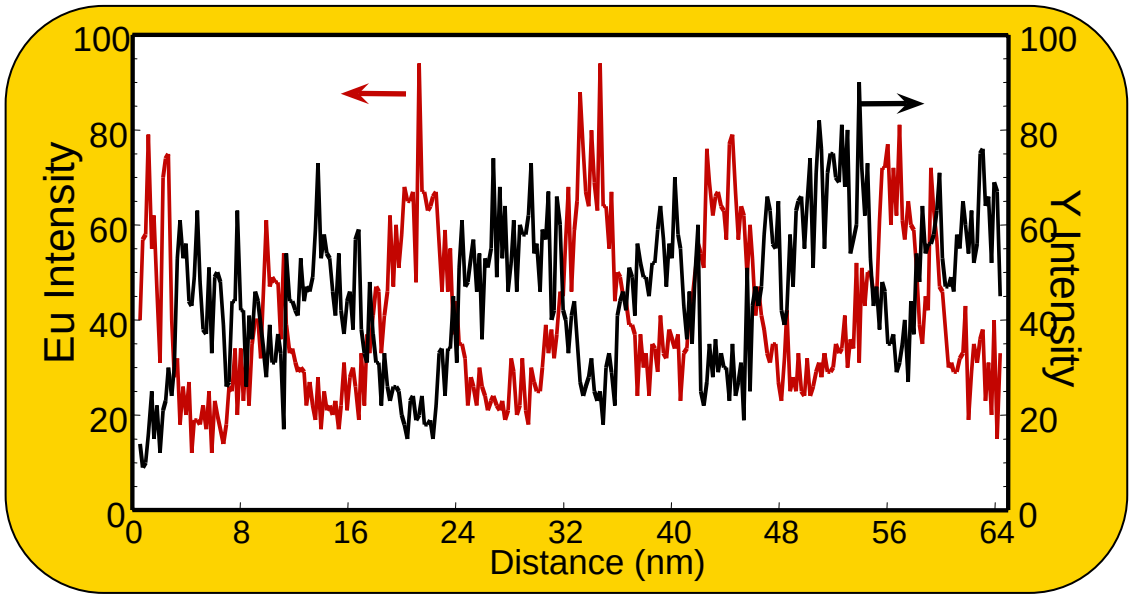
Eu-123 comprised ≈ 1/3-1/4 the target area.

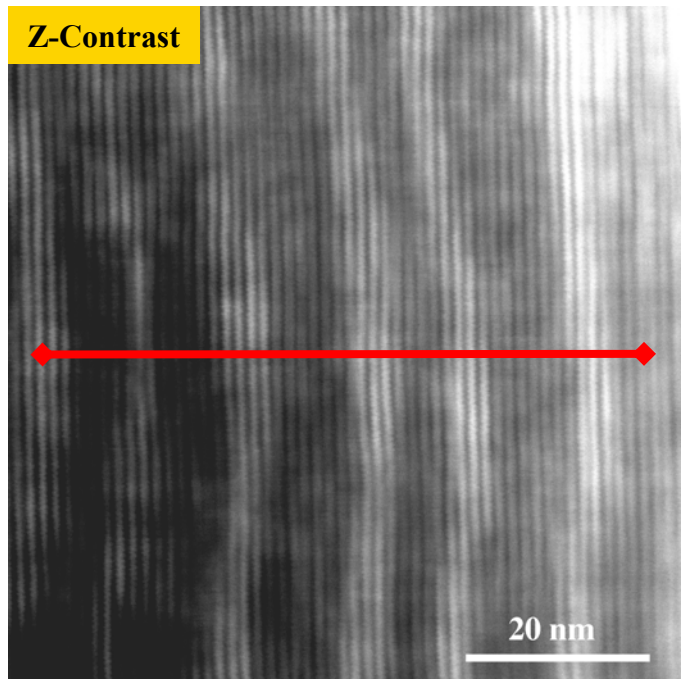


High resolution, Z-contrast imaging shows the meandering of the multilayer structure.

- Meandering caused by the Stranski-Krastanov growth mode of YBCO.
- Chemical and crystalline microstructures.
- Z-contrast imaging and EDS line scans suggest little interdiffusion between layers.
- This stationary sample shows some non-uniformity in the relative spacing of the layers.





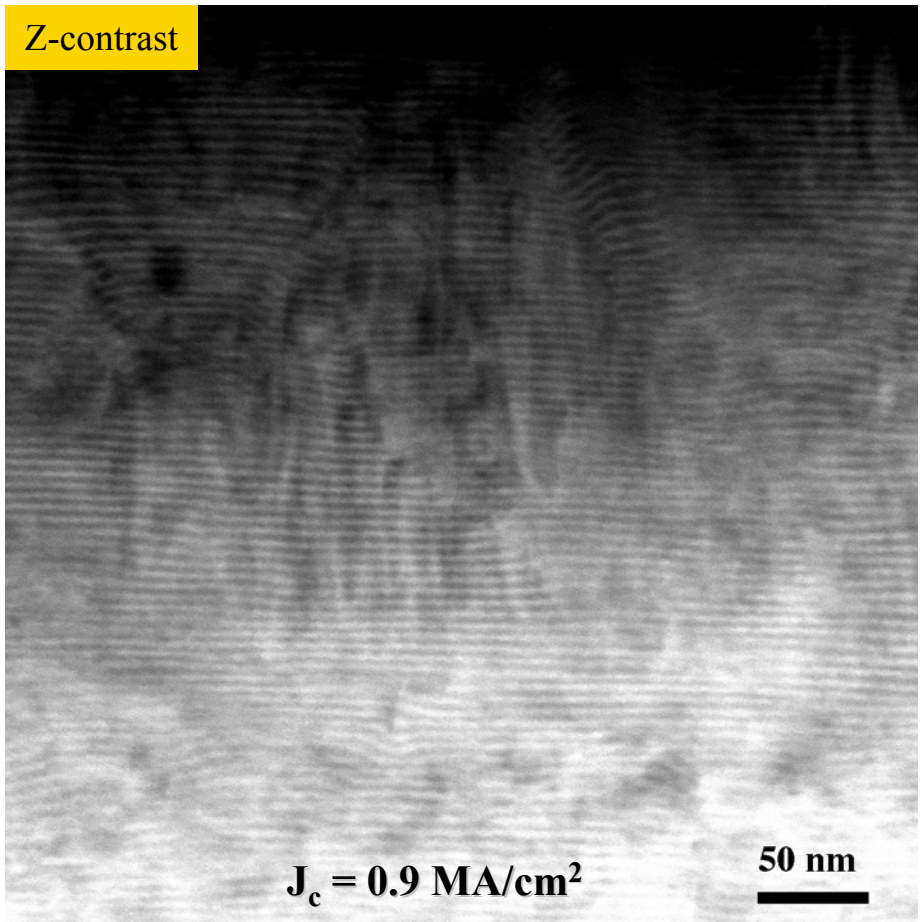


A uniform multilayer structure is obtained via PLD deposition using the sectored target and a linearly translated substrate.

- Λ = 5.5 nm: Eu-123 ≈ 1.8 nm and Y-123 ≈ 3.7 nm
- t = 1.84 µm (335 multilayer pairs).
- Very few secondary phases; some dislocations near the interface.

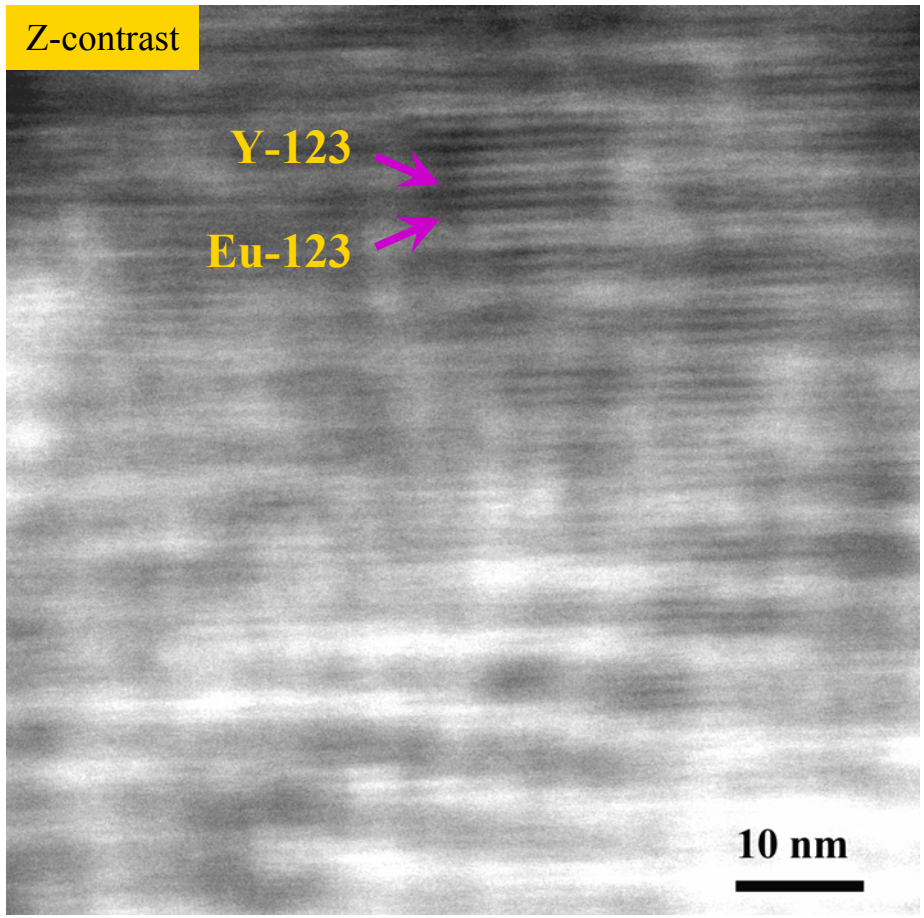
Tape speed ≈ 0.75 m/hr

Target rotation ≈ 15 rpm



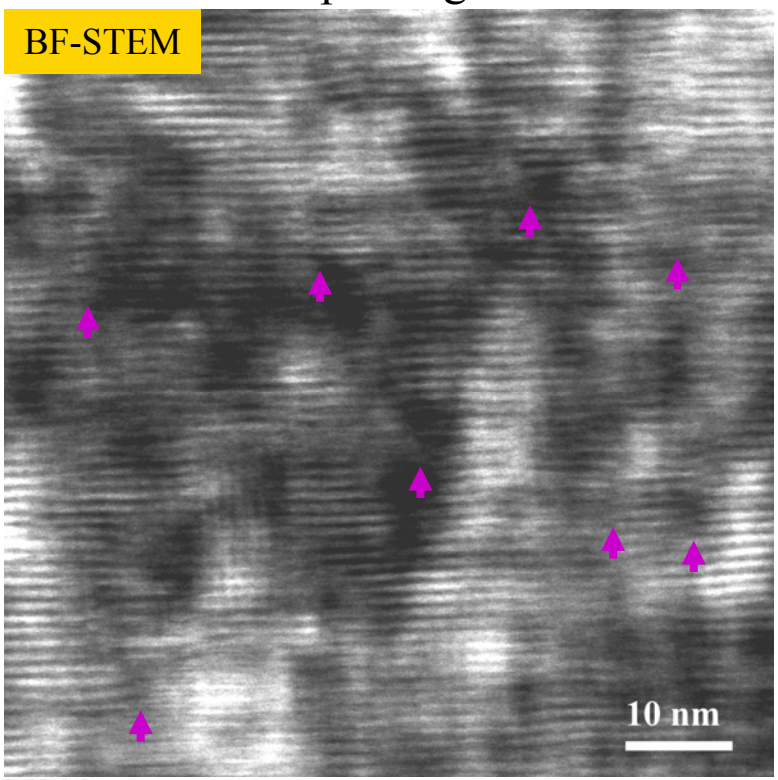
J_c = 0.9 MA/cm^2

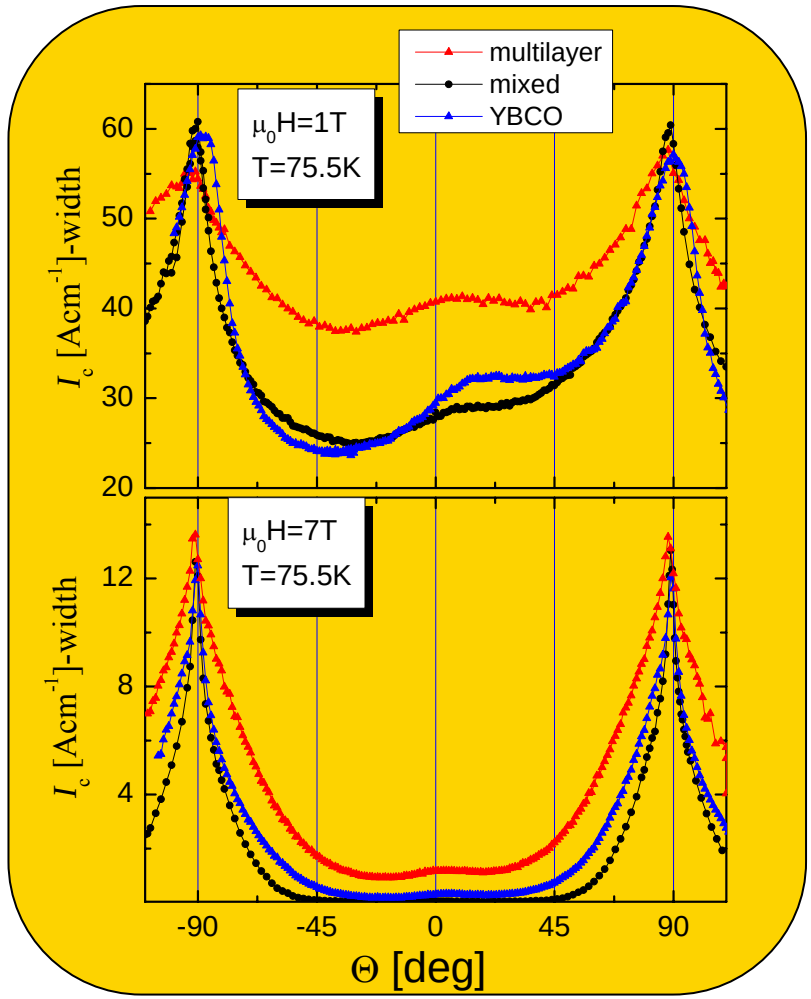
75K, SF



Z-contrast imaging shows the Eu-123 and Y-123 layers to be ≈ 2 and 4 unit cells thick, respectively.

- Interplay between the chemical and crystalline microstructures to form a number of antiphase boundaries in the film.
- Significantly improved in-field performance for the ultra-fine multilayer sample.
- No inclusions in this sample that could be associated with the pinning.





The tilted IBAD MgO template used in these experiments gives rise to asymmetric structure around H || c.

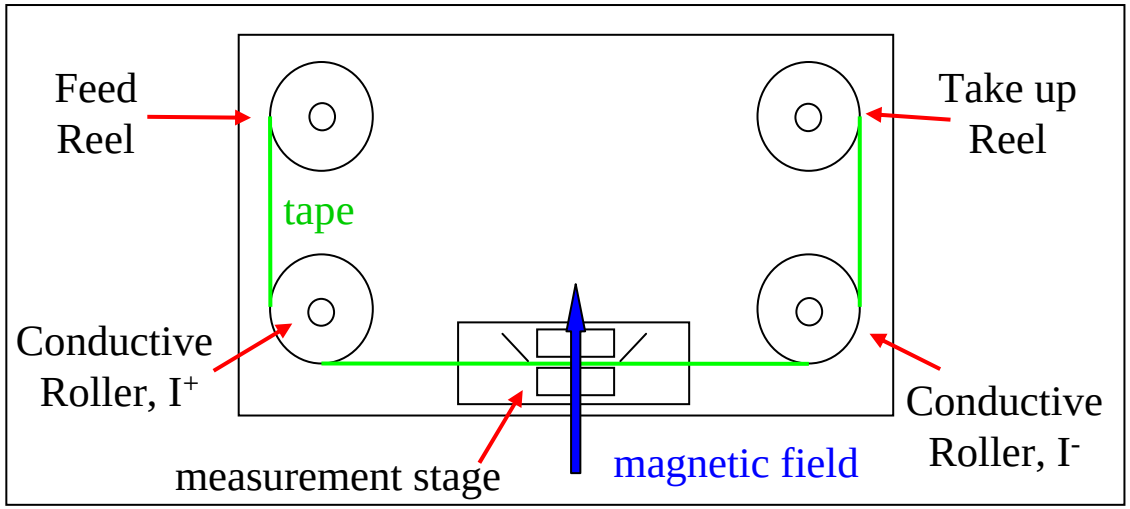
B. Maiorov, Appl. Phys. Lett. (2005).

Studies of superconducting properties of 2nd generation wires

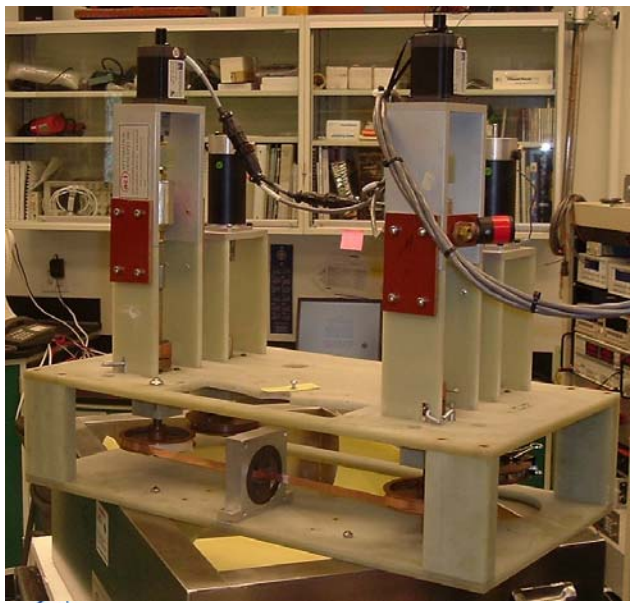
Outline

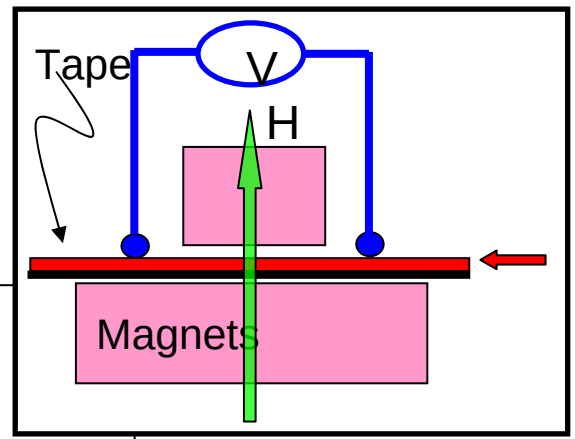
- Position dependent studies of long lengths of CC
  - Perspective and goals
  - Development of new capabilities
  - Characterization and troubleshooting of American Superconductor tapes
  - From characterization of tapes to qualification of commercial products
- Pinning studies of champion MOCVD film from SuperPower

In anticipation to the scale up of CC and the need to characterize them, several years ago LANL developed a capability for position-dependent, continuous critical current measurements of long tapes.



- In-field measurements: reduced I_c avoids sample damage and defines the position.
- Interchangeable permanent magnets setup for flexibility in selection of field profile
- H//c up to ~0.75 T

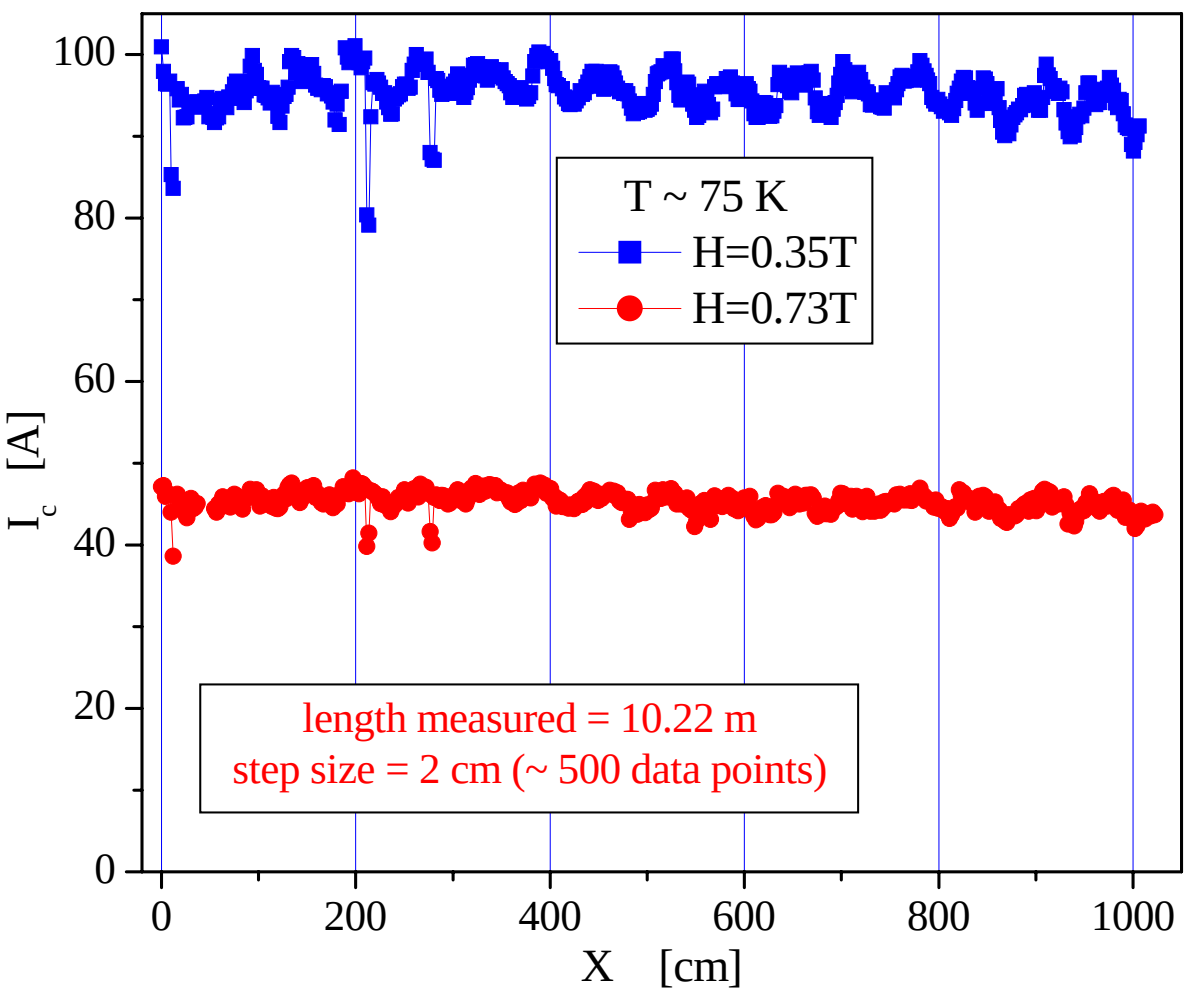


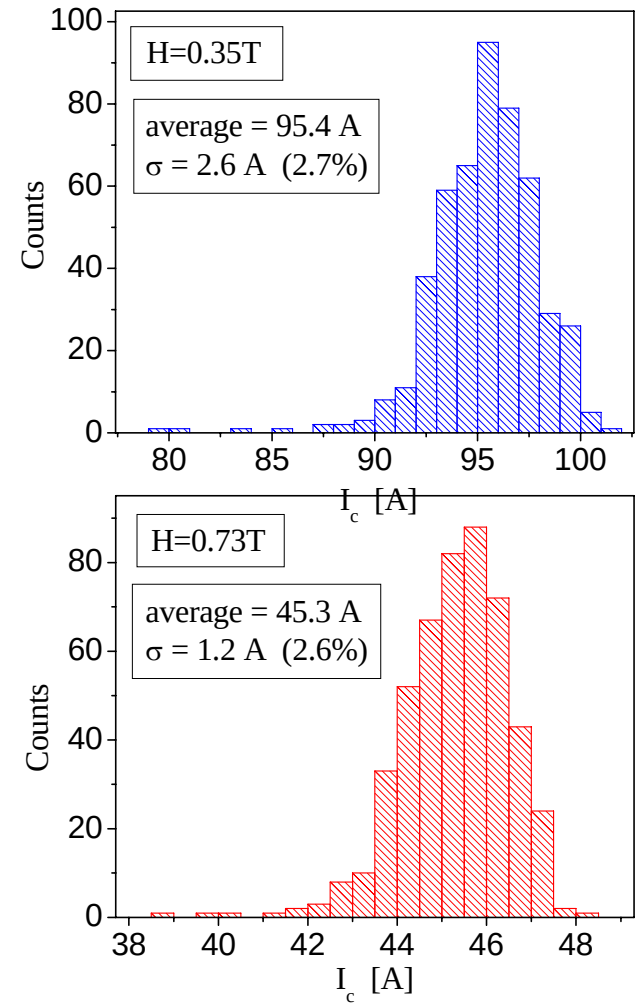


Some initial characteristics:

- Maximum current ~ 80 A
- Designed for 1 cm-wide tapes
- Maximum length 10's of meters
- Positional resolution ~ 0.1 mm
- Measurement rate: ~ 1 curve/minute

Positional dependence of J_c for a 10 m long MOD/RABiTs tape using the LANL continuous measurement system



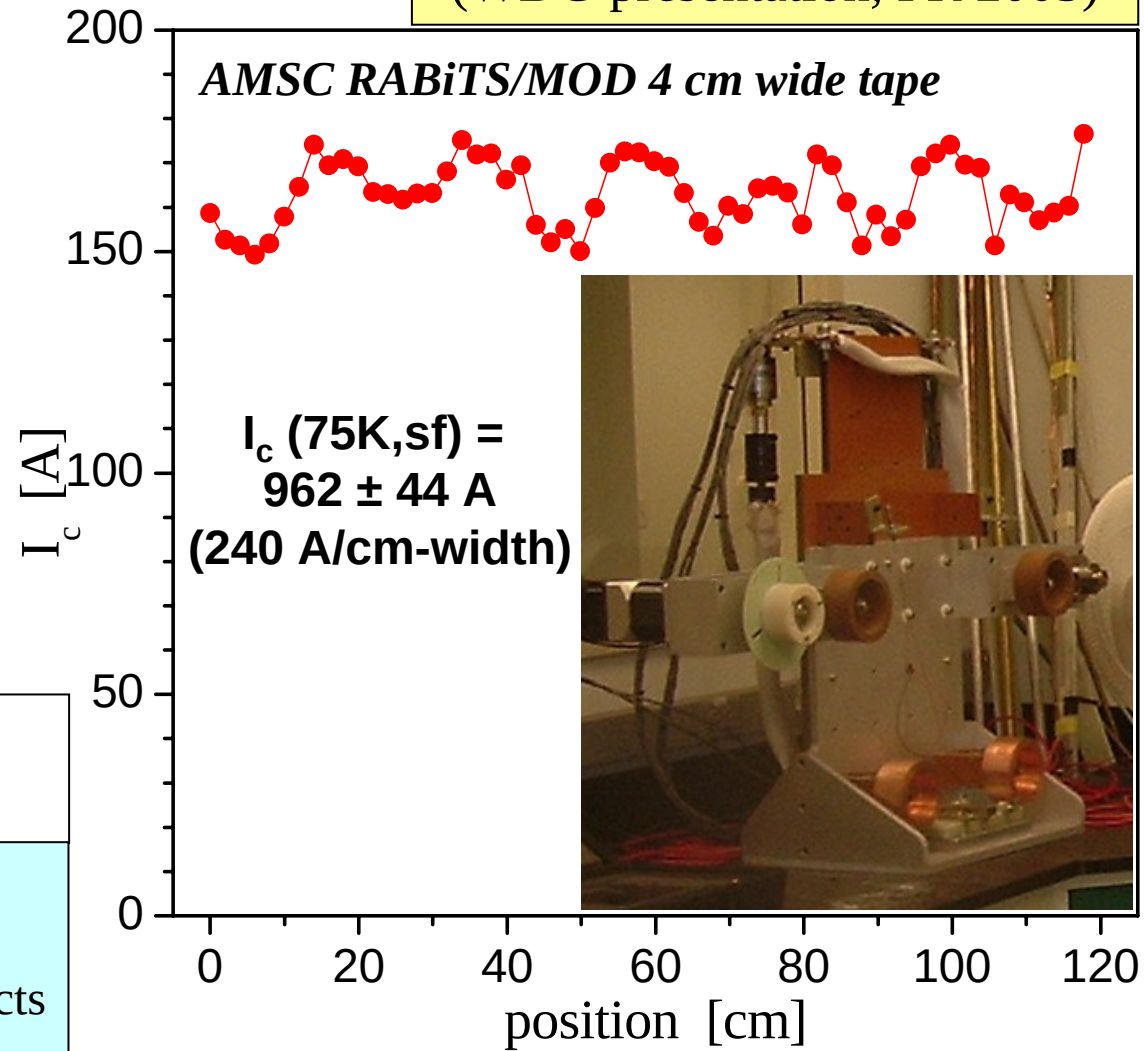


AMSC tape, measurement presented in the WDG talk at PR 2004

As the scale up of our industrial partners progressed, we developed a new system with tape load/unload at room temperature

The original goal (still valid) was to have a system that could be inserted in a production line

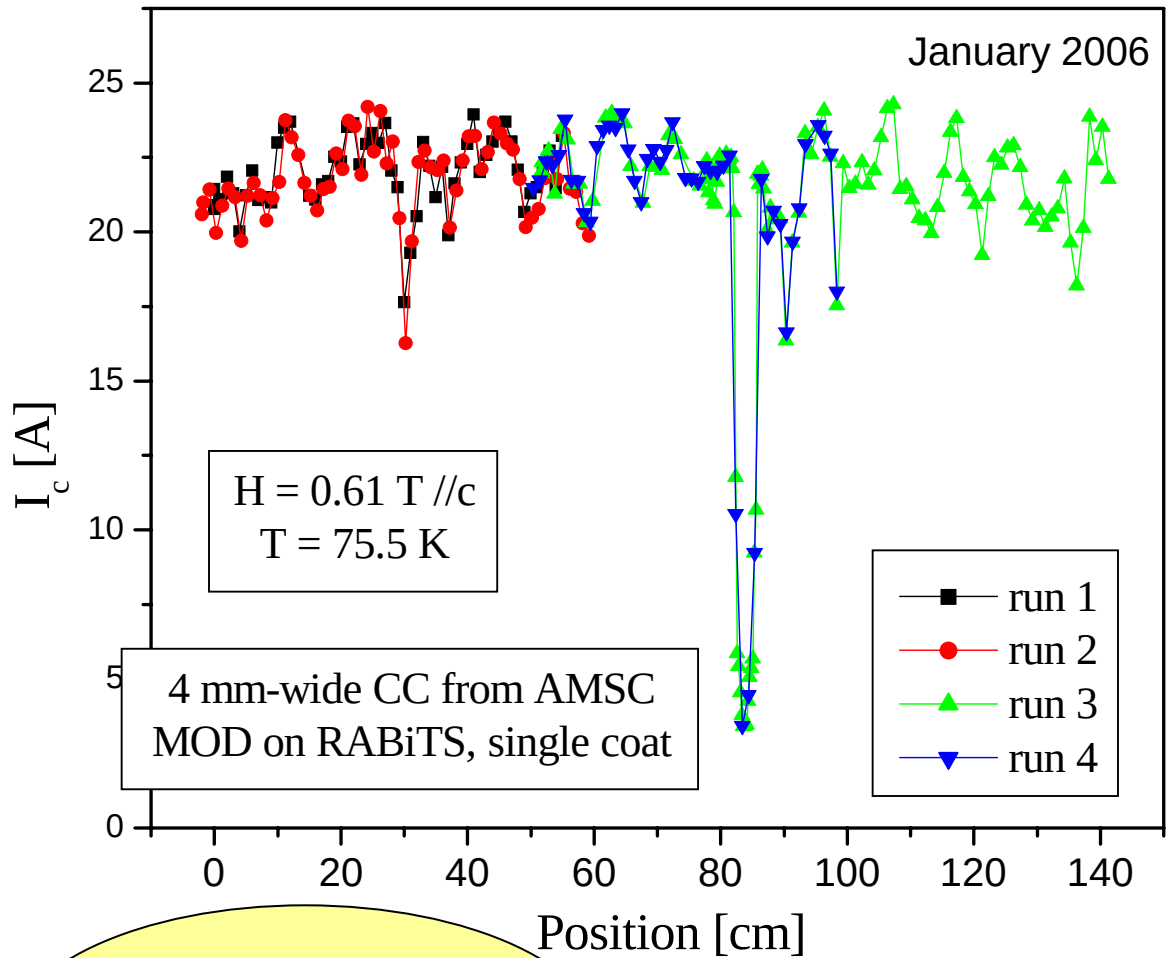
(WDG presentation, PR 2005)

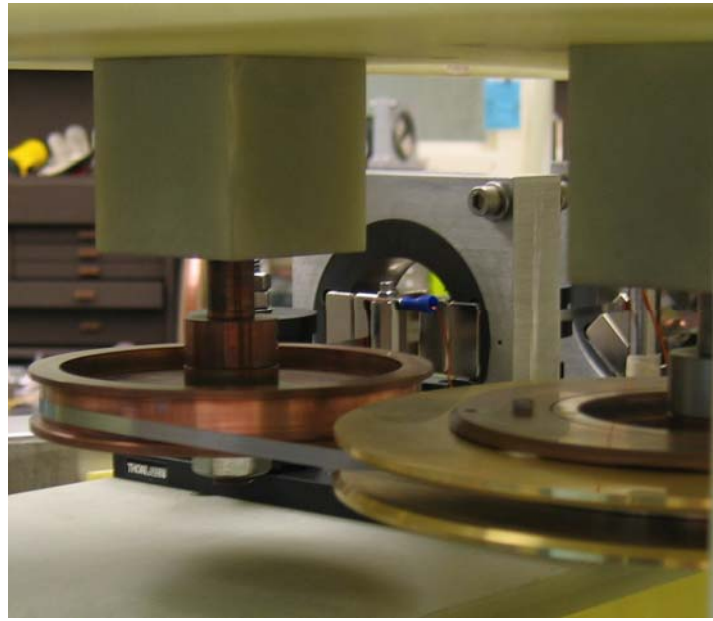


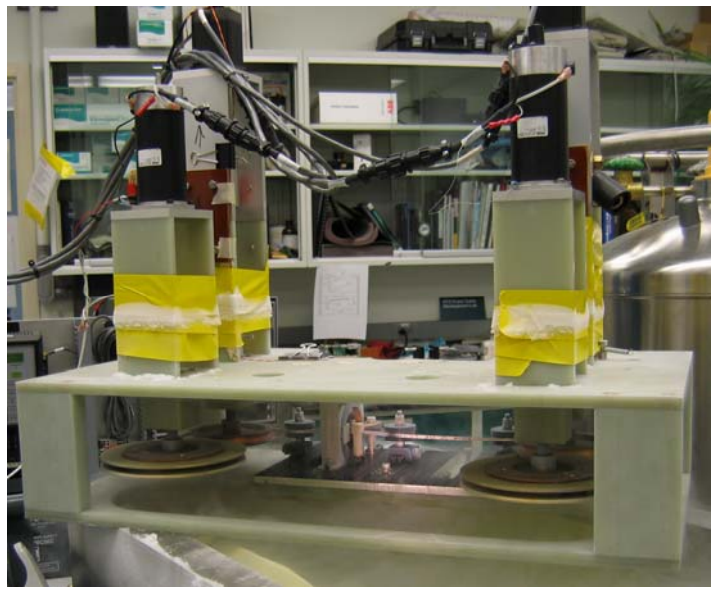
Eventually, it evolved into a facility to measure 4 cm-wide tapes

Challenge:
To apply currents exceeding 350 A through the rotating Cu current contacts (horizontal axes)

We adapted the original system to measure 4 mm wide tapes (retaining the 1 cm-wide tapes capability)

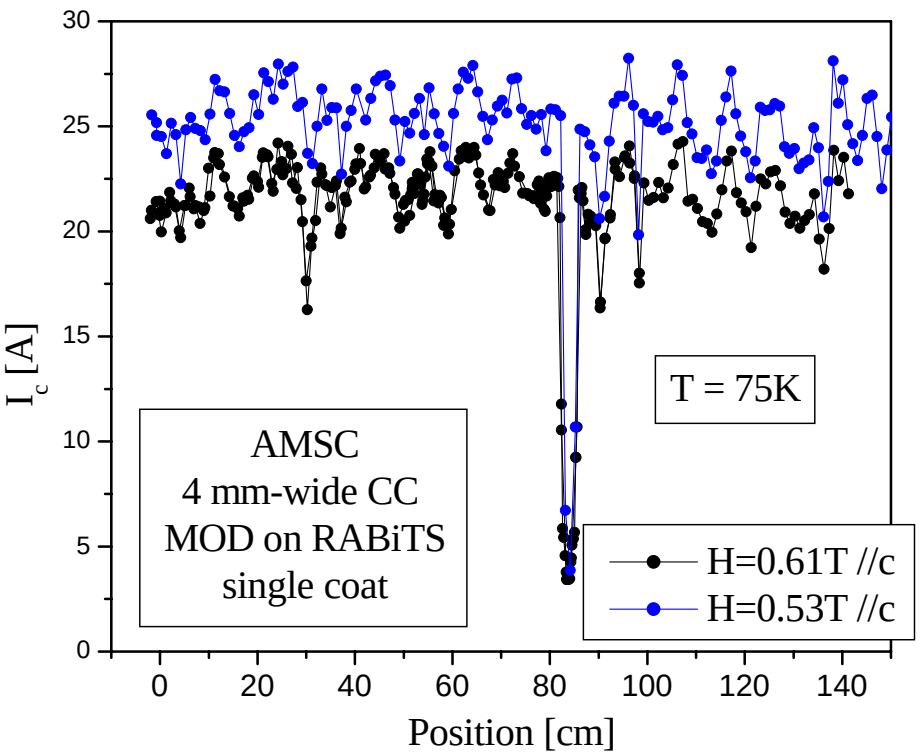


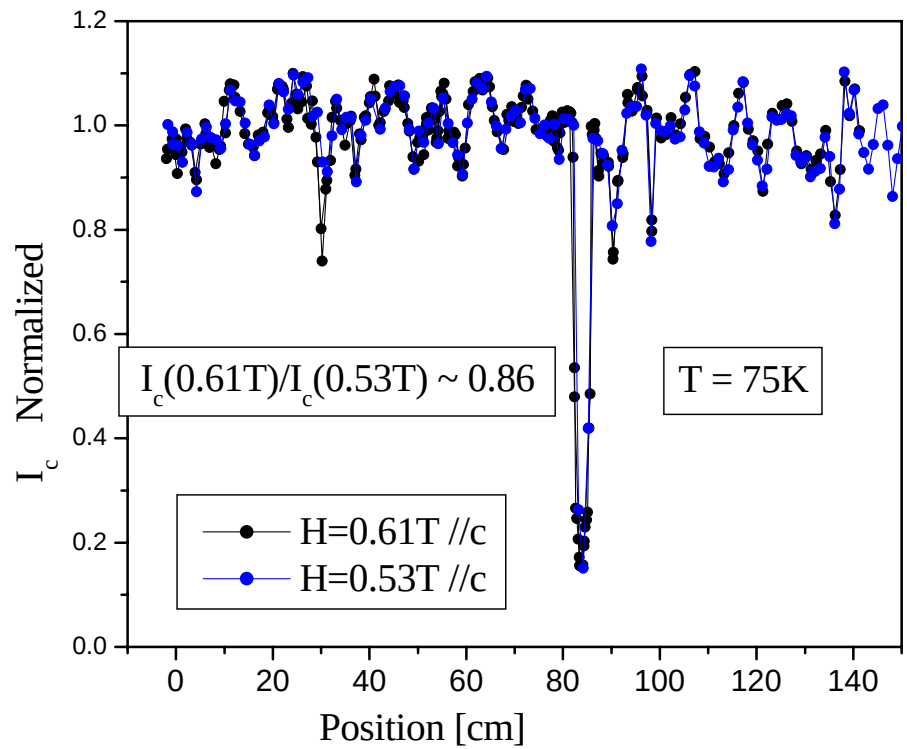




Good reproducibility:
- no tape damage
- good tracking

Measurements at different fields are useful to identify the source of the positional variations of I_c

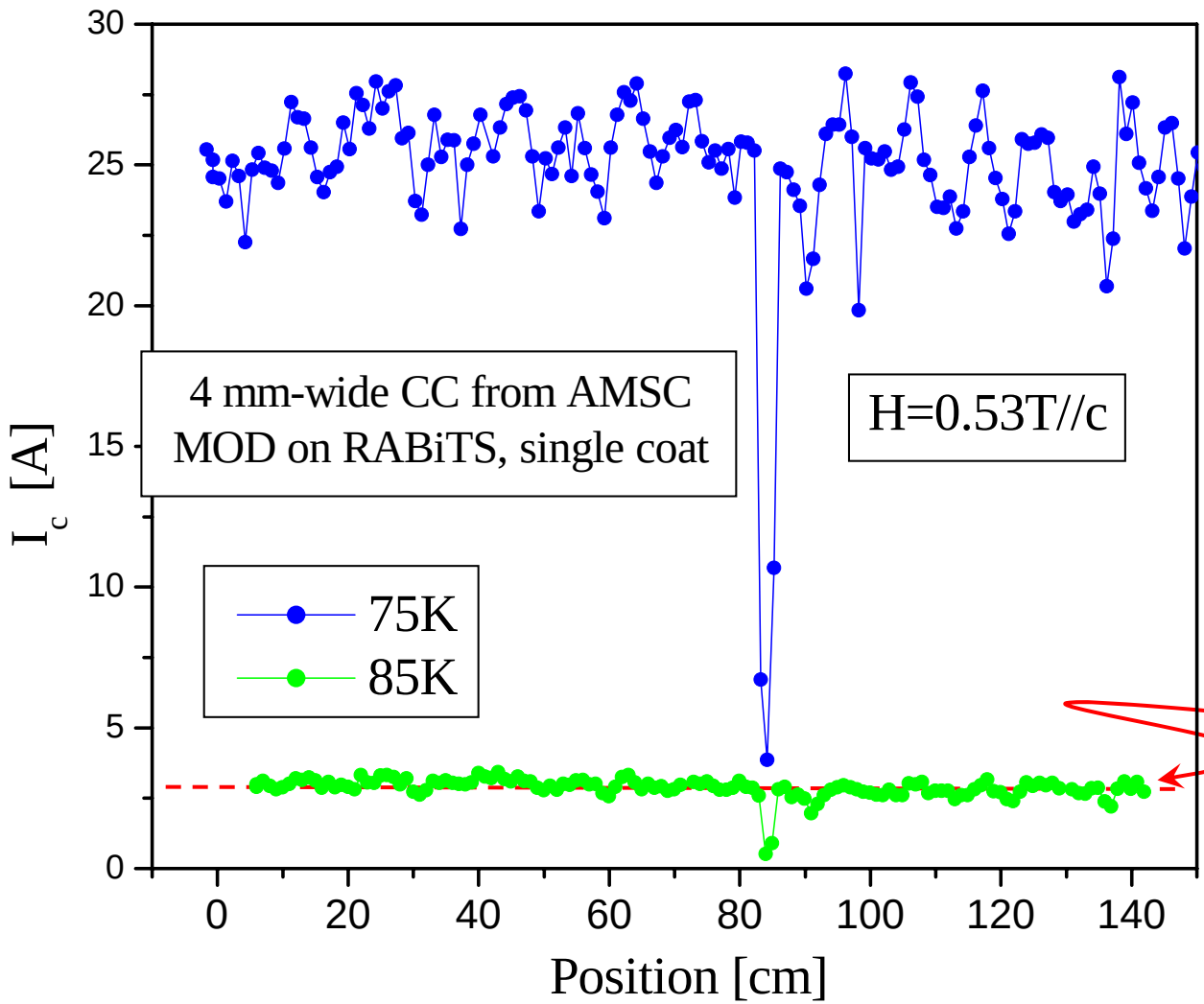




The superposition of the normalized curves indicates that, to a good approximation, $I_c(H,x) = f(H)*g(x)$

This suggests cross sectional fluctuations rather than variations in the field-dependent pinning properties

The most basic characterization parameter of a superconductor is its transition temperature T_c. However, the position dependence of T_c is hard to measure in a long tape

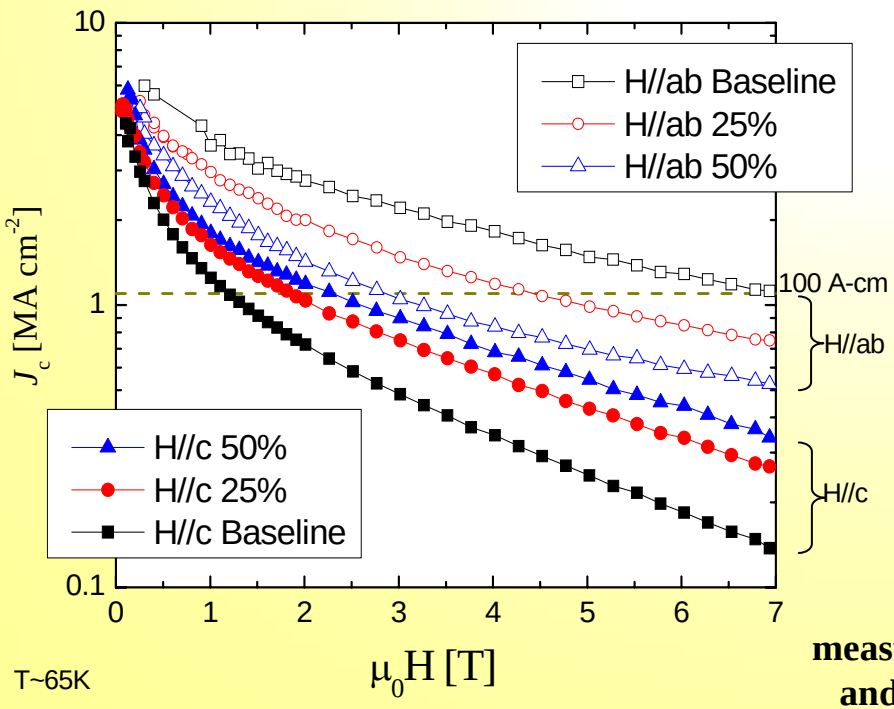


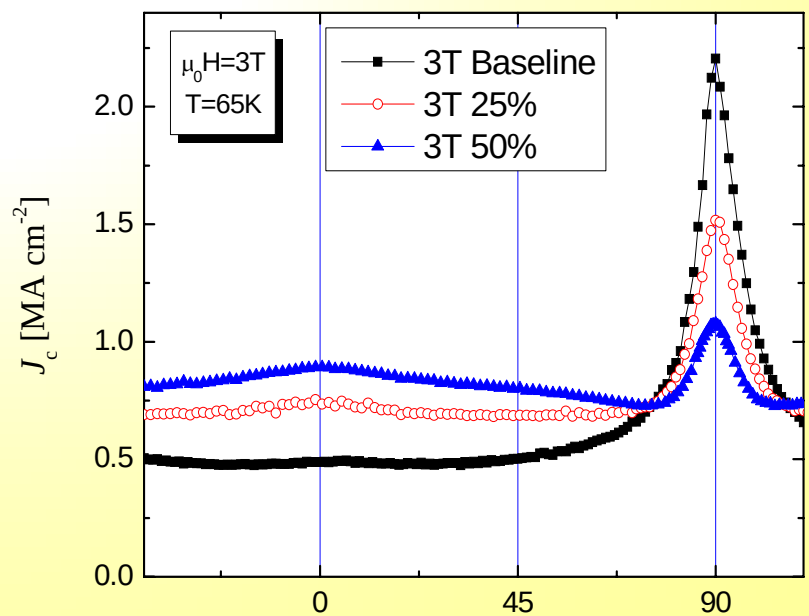
A better option is to measure the in-field I_c at a temperature very close to T_c, using liquid Ar (85K)

Good I_c uniformity indicates T_c uniformity

The different field and angular dependence of J_c produced by pinning enhancement methods (now being incorporated into production lines) imply that measurements for H//c alone are not sufficient

Effects of Er additions in MOD CC from AMCS (WDG presentation, PR 2005)

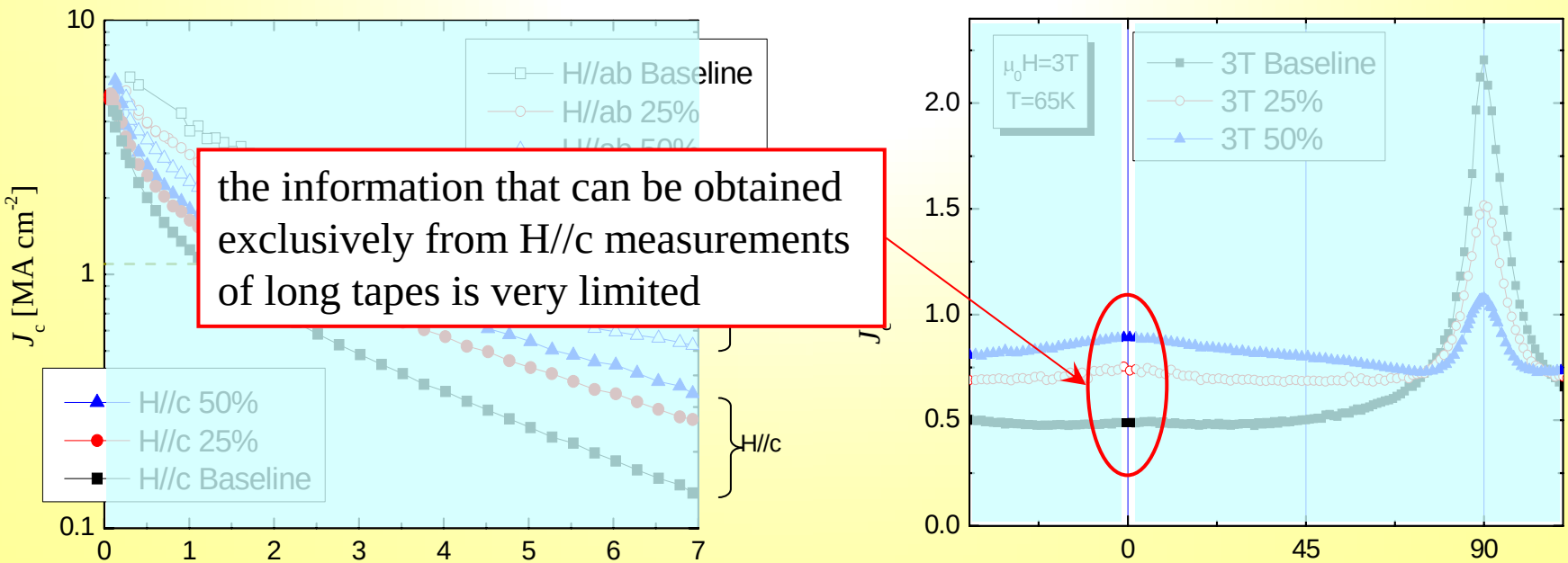




measurements by L. Civale and B. Maiorov (LANL)

The different field and angular dependence of J_c produced by pinning enhancement methods (now being incorporated into production lines) imply that measurements for H//c alone are not sufficient

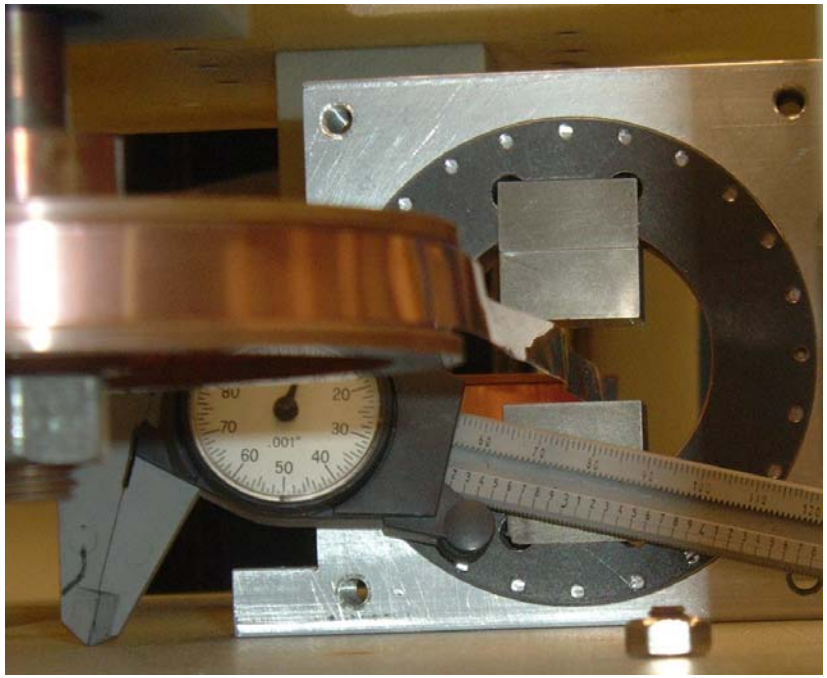
Effects of Er additions in MOD CC from AMCS (WDG presentation, PR 2005)

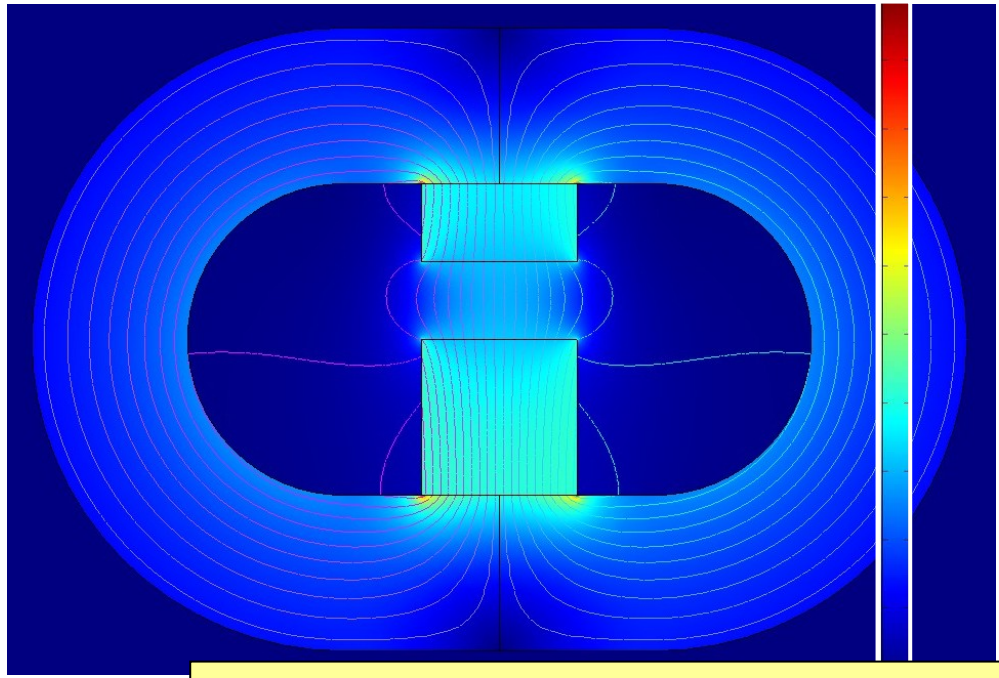


measurements by L. Civale and B. Maiorov (LANL)

The rapidly evolving scale up process of CC now requires the capability to perform position-dependent characterization of tapes at field orientations other than H//c…..

We adapted our system to be able to perform position-dependent measurements of I_c for H//ab





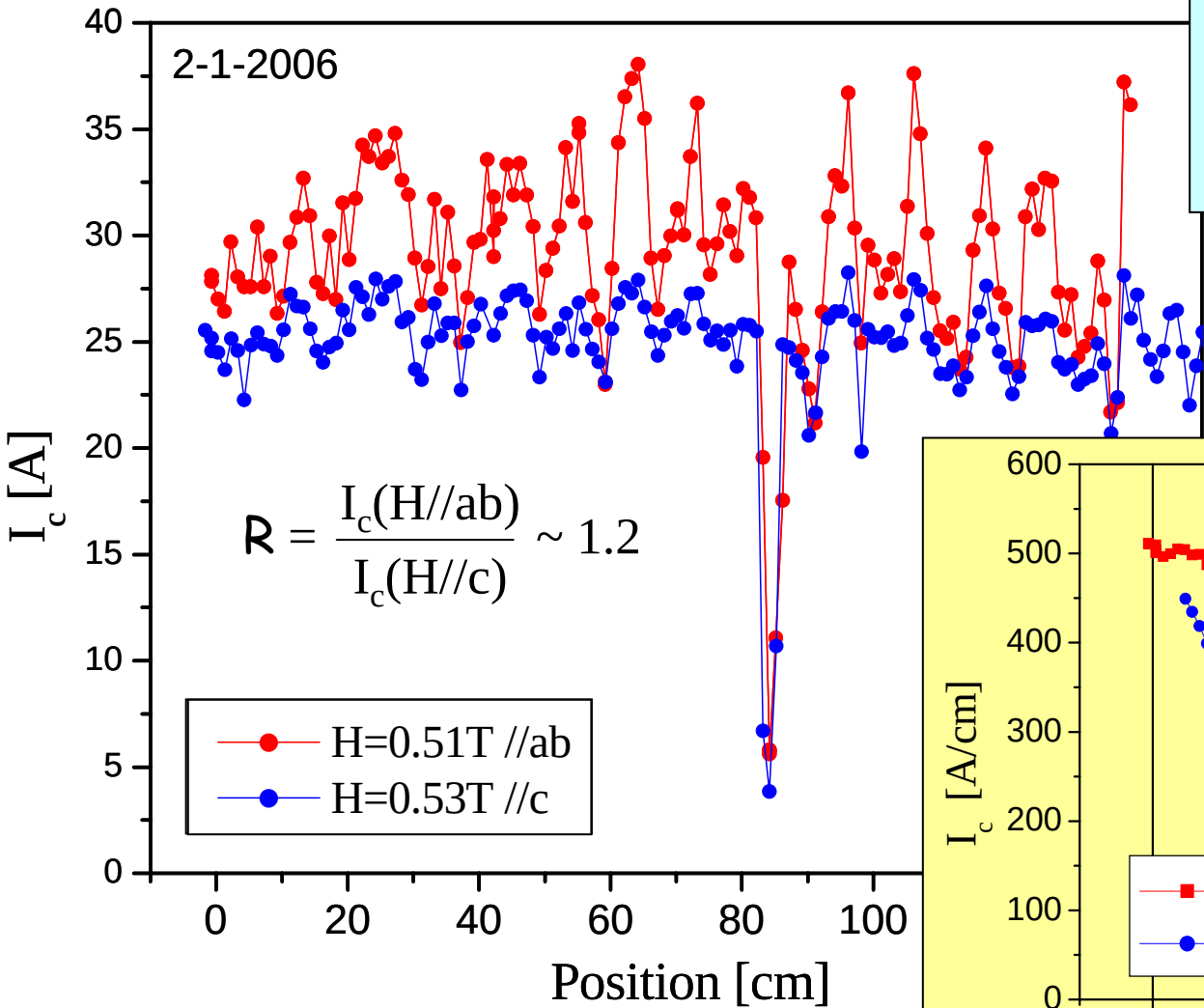
numerical simulation by Francesco Grilli

Issues:

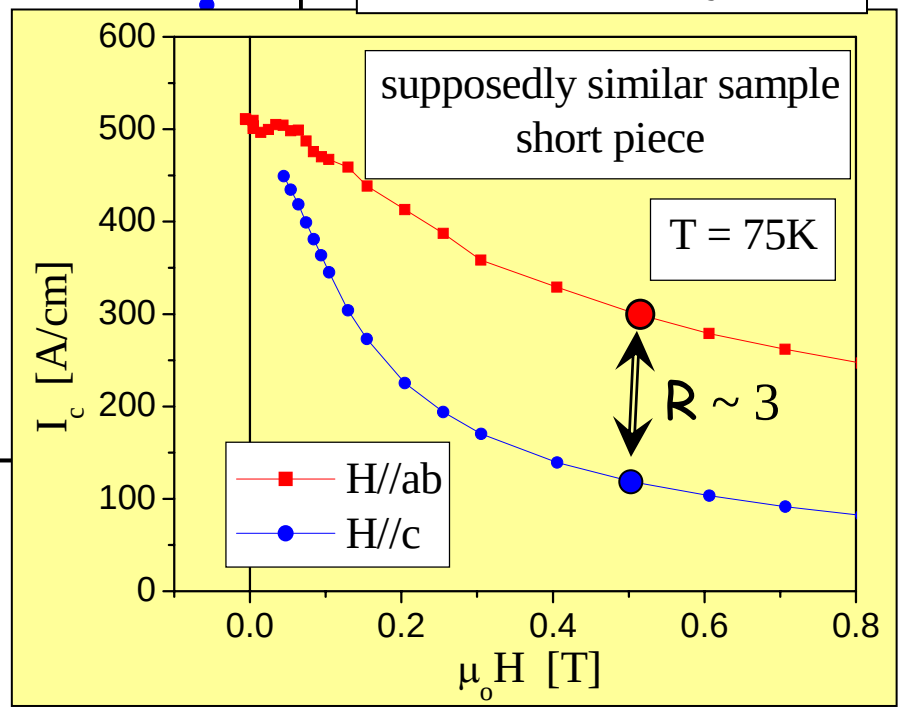
- Field uniformity (estimated by numerical modeling and experimentally confirmed)
- H decreases and becomes less uniform as the gap between magnets increases ⇒ easier for narrower tapes (4 mm) – but system is designed for 1 cm-wide tapes also
- Tape-field alignment is more critical
- Does the available field produce enough reduction in I_c for H//ab to allow measurements and avoid sample damage? YES

We have successfully performed position-dependent measurements of I_c for H//ab in several 4 mm-wide long tapes

The anisotropy in this tape differs significantly from the expectation (more later…)



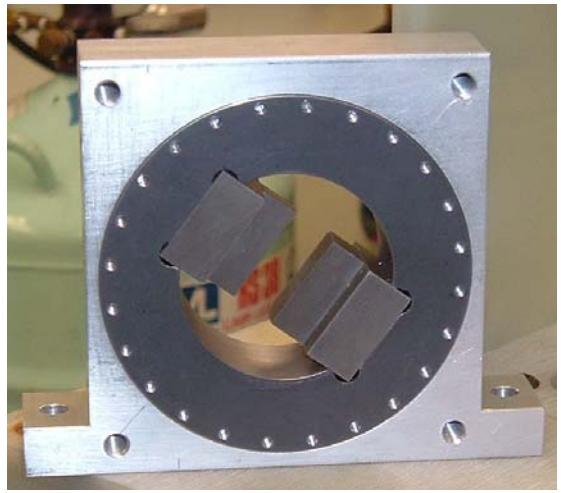
Comparison with results from our 7T static measurements system



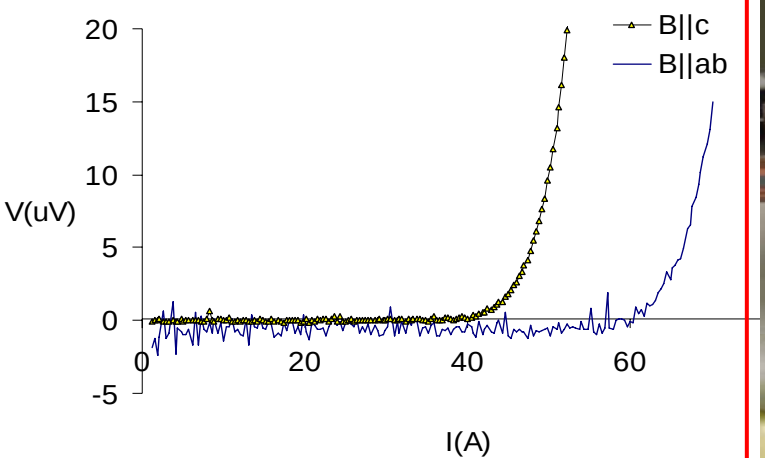
The new measurement stage allows us to measure at any H orientation Moreover, due to the modular design of the system we can use up to 3 stages simultaneously

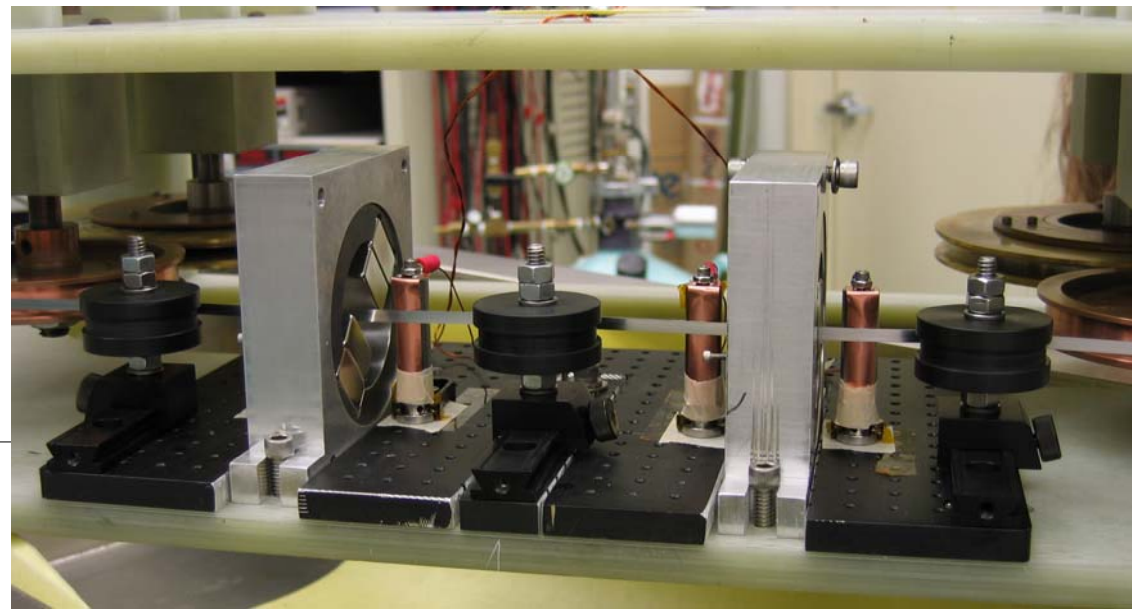
This opens up a number of possibilities. We can simultaneously measure at:

- 3 orientations, same field (anisotropy)
- 3 fields, same orientation (field dependence, α)
- all 3 stages at same field and orientation (3 times faster)
- cost increase: 1 nanovoltmeter per stage

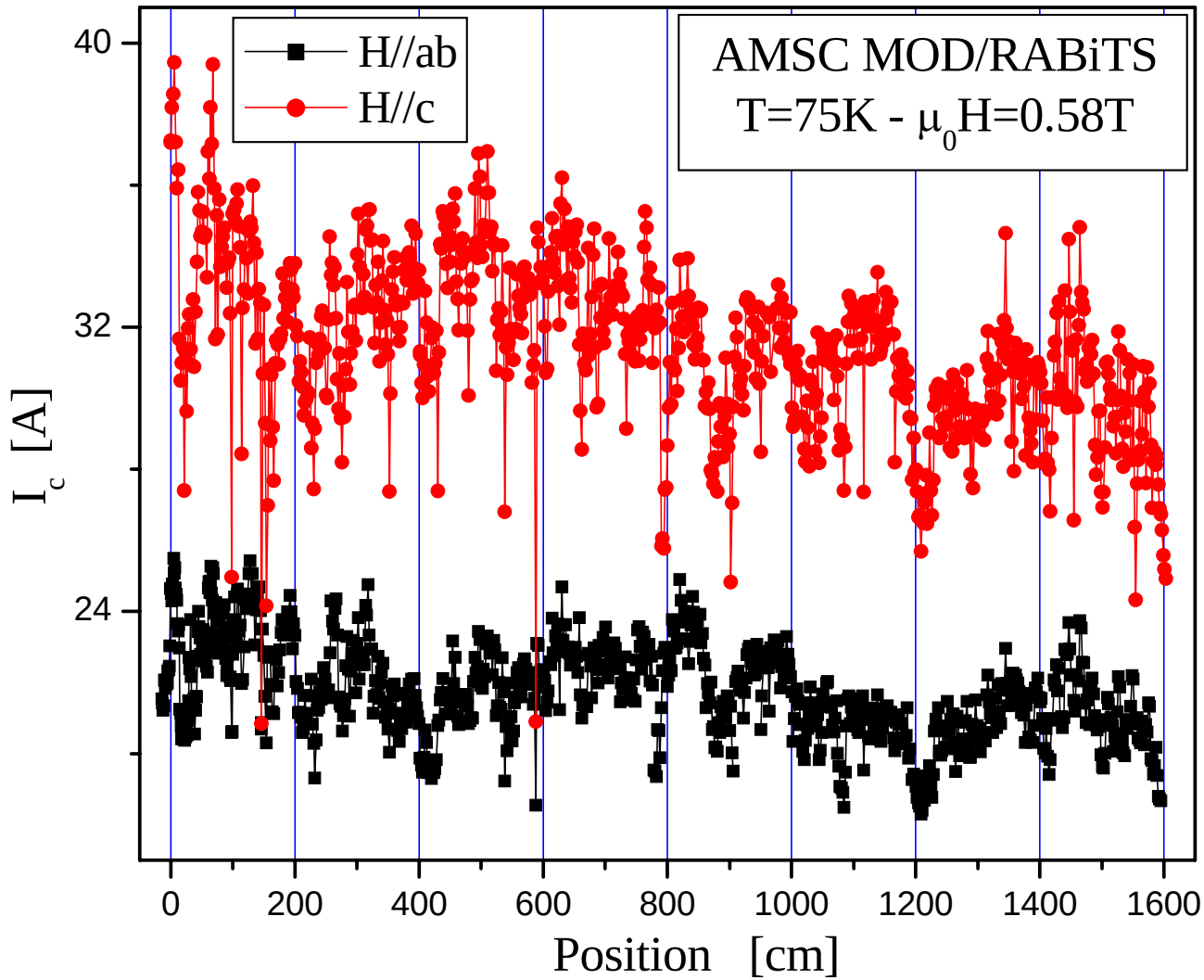


Question: what happens when the current locally exceeds I_c at one of the stages?
Answer: no problem, the Cu stabilizer can handle it!





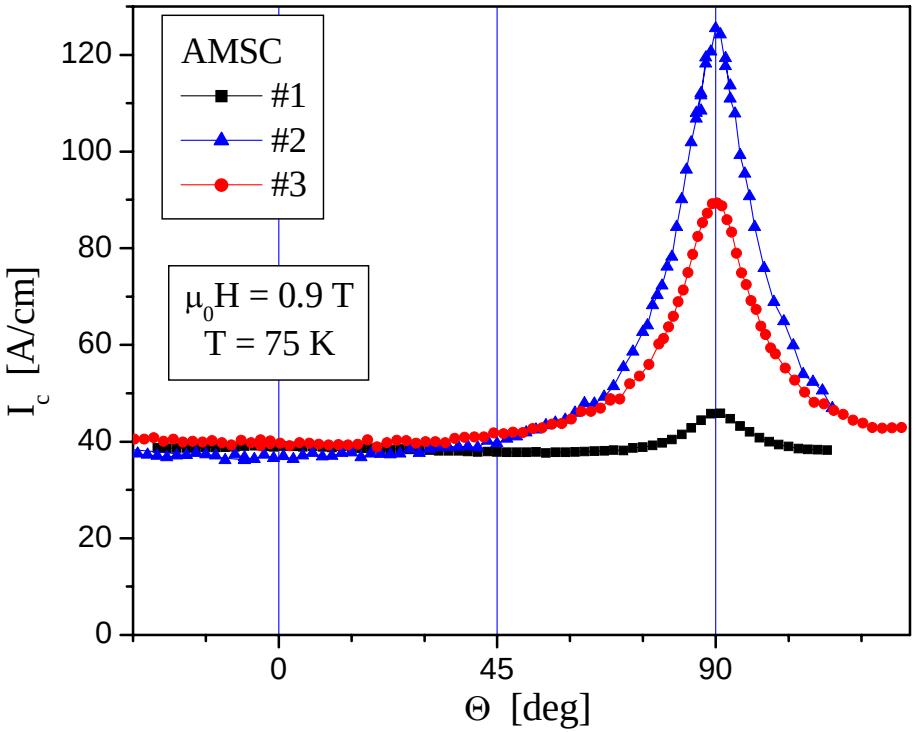
With the new capabilities we measured I_c for H//c and H//ab in a 16 m long, 4 mm wide tape from AMSC

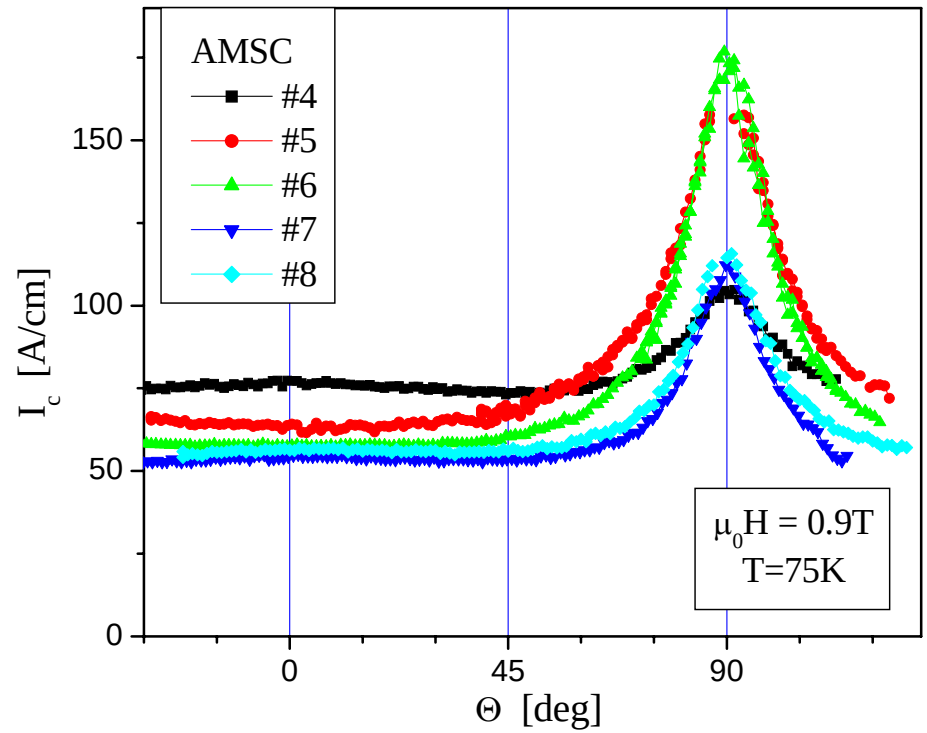


- 1600 I-V curves (fully automated)
- Now the applied field is the same in both orientations
- Clear positional correlation between H//c and H//ab

The problems with I_c for H//ab persisted. Note the reversed ratio of I_c for H//c and H//ab.

To help AMSC to troubleshoot this scale up problem, we performed fast angular dependence studies of I_c on a large number of short samples from their production line using our 1 T iron-magnet capability





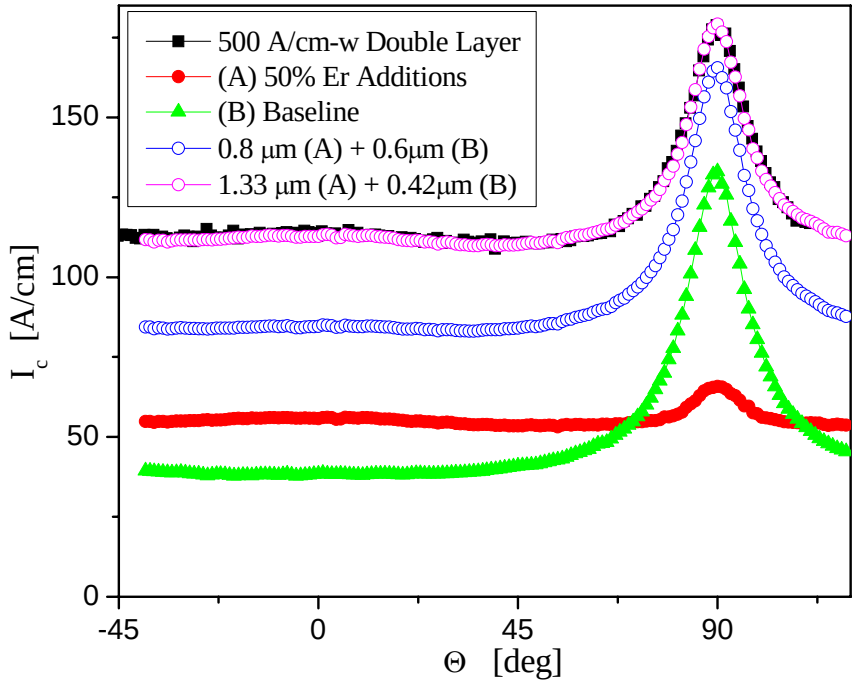
Thanks in part to our help, AMSC identified and solved this scale up problem.

At AMSC request, we have started to build a similar system for them, so they will have this diagnosis capability in house. Advantages: fast, no liquid He required, much lower cost than a high-field SC magnet system

The successful end of this story was already shown earlier in this talk by T. Holesinger

The culmination of this work was the development of a hybrid double-coat film that met the 500 A/cm-w goal set by AMSC.

- As shown in the WDG presentation, a hybrid double-layer MOD film (0.6 µm YBCO on 0.8 µm Y(Dy)BCO (50% additions)) was developed to meet a 500 A/cm-w goal.



LANL showed that the performance of these hybrid films can be modeled with 0.9 µm MOD film data.

1.33µm (A) + 0.42µm (B)

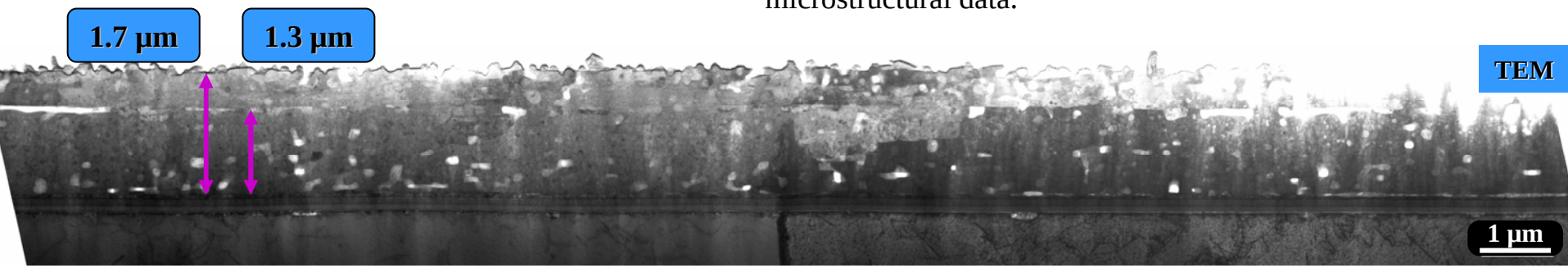
0.8µm (A) + 0.6µm (B)

The best fit agrees very closely with the actual microstructural data.

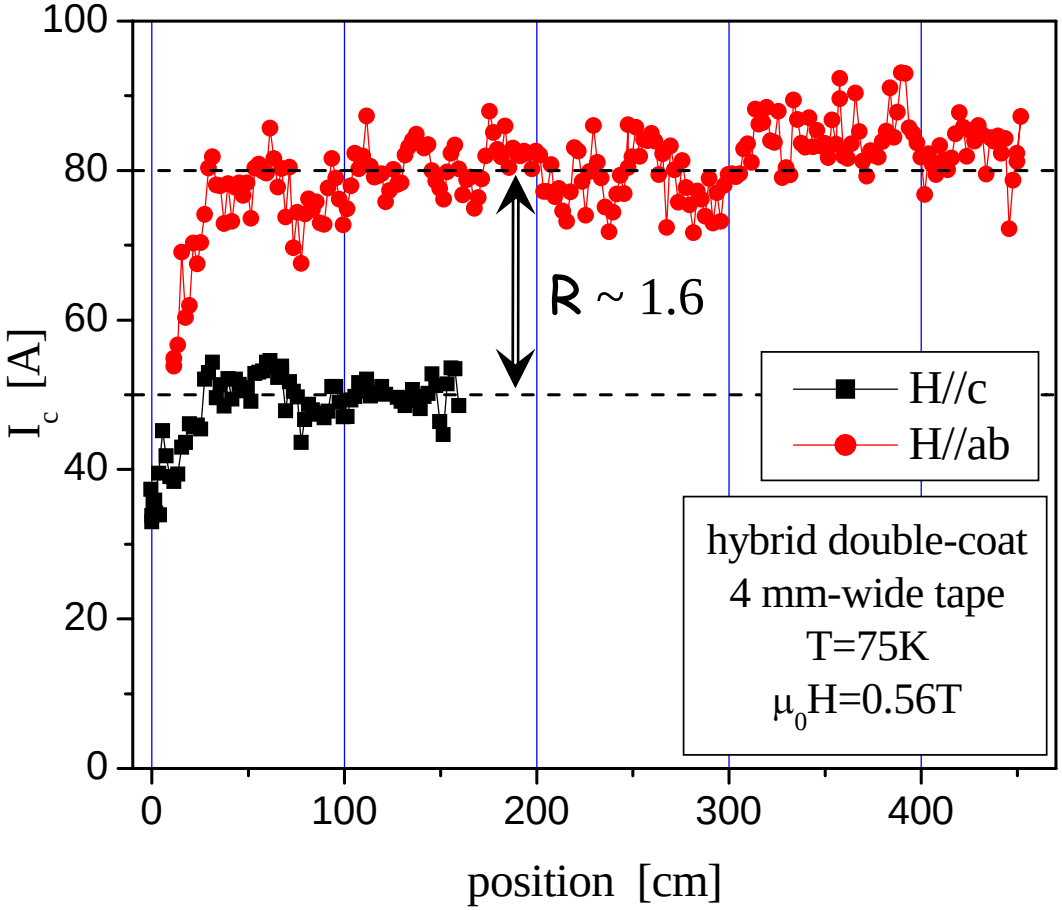
Modeling of the double layer film with the single-layer 0.9 µm MOD film data implies a weak J_c dependence.

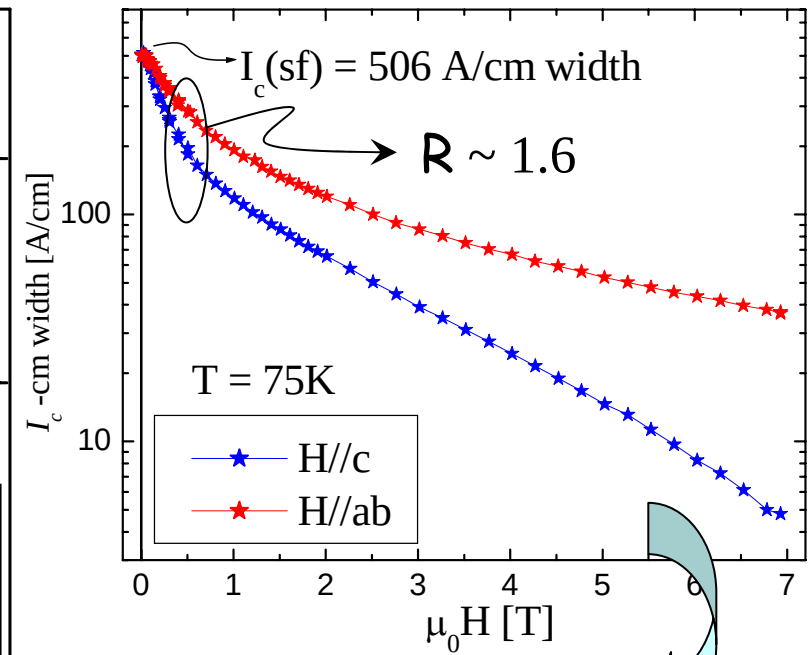
Bottom 1.3 µm: nano-dot structure obtained with rare-earth additions to YBCO.

Top 0.4 µm: Y-123 planar intergrowth structure obtained with undoped YBCO.



The solution of this scale up problem lead to the fabrication of long lengths of hybrid double-coat 4 mm wide tapes. We studied a 4 m long portion of that CC in our continuous measuring system





I_c(0.56T//ab) ~ 0.55 * I_c(sf)

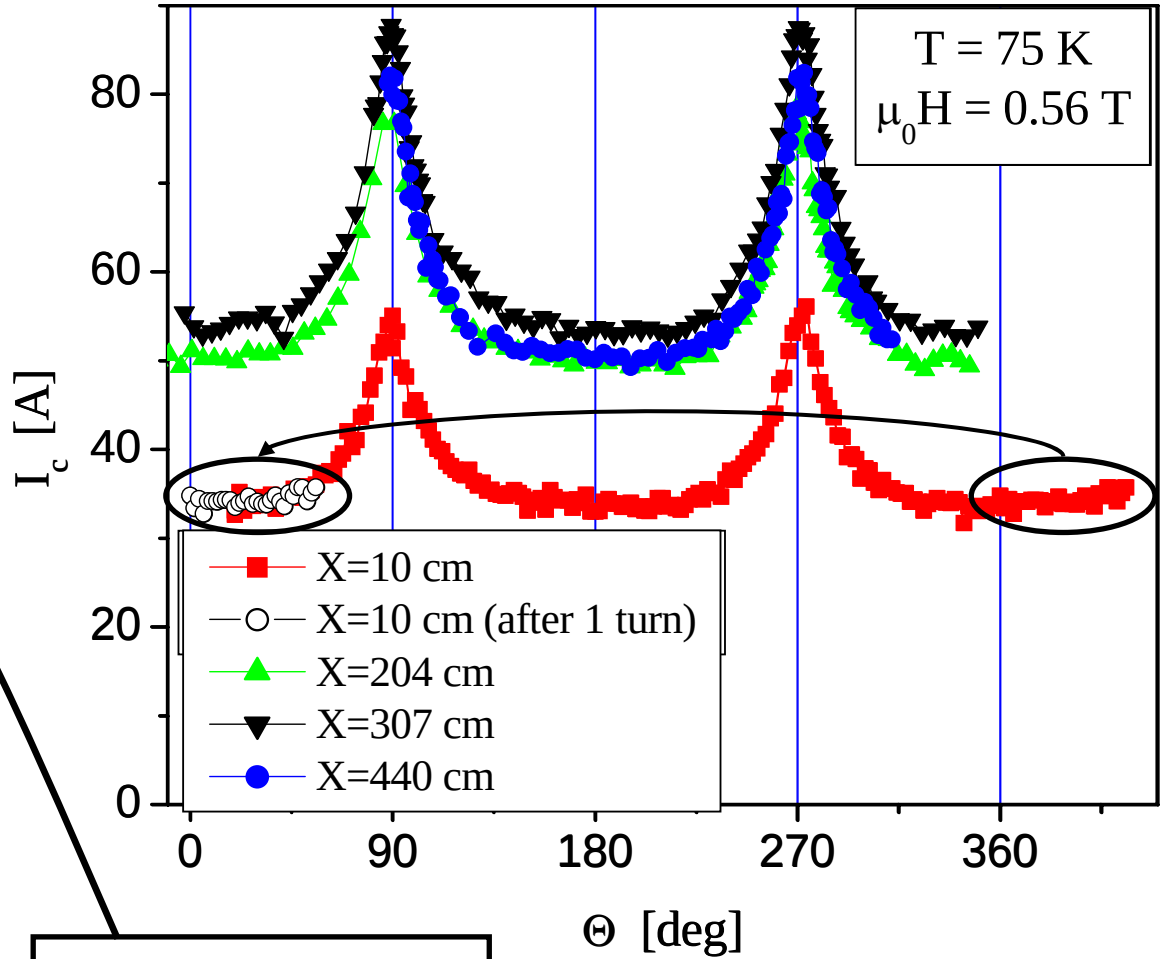
⇓

Extrapolation for the long tape gives I_c(sf) ~ 145 A (⇒ 360 A/cm width)

Of course the next step would be to be able to measure the full angular dependence of Ic in the continuous tape……

Anticipating the need of the U.S. Superconductor Industry for characterization of the angular dependence of long CC, we developed the capability to measure $I_c(\Theta)$ in the continuous measurement system

The rotating measuring stage is incorporated to our continuous system and controlled by same software. We can stop the $I_c(X)$ run and check $I_c(\Theta)$ at any point. (routinely and for troubleshooting)



Rotation axis. Driven by stepper motor

The next step: From characterization of tapes to qualification of commercial products

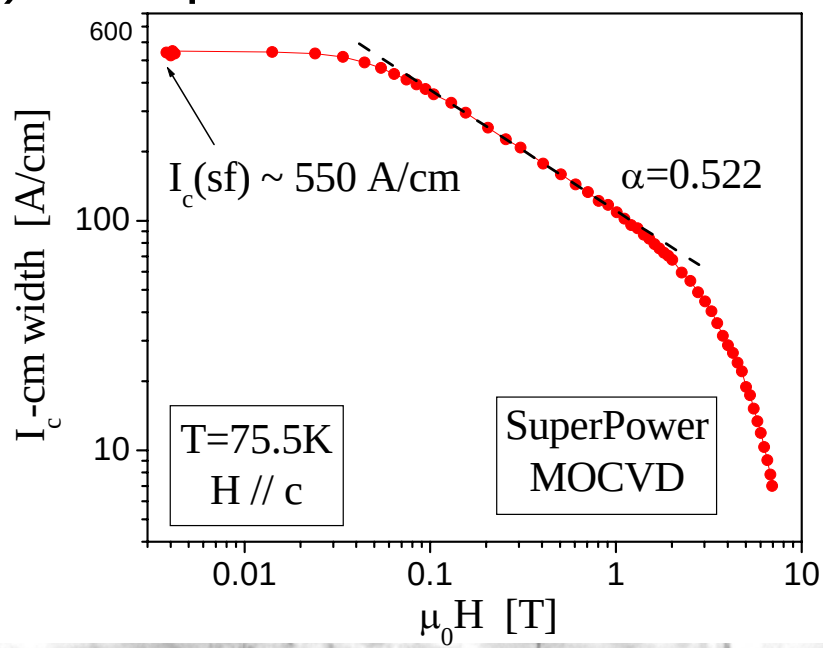
What nondestructive characterization measurements should be incorporated to the production lines?

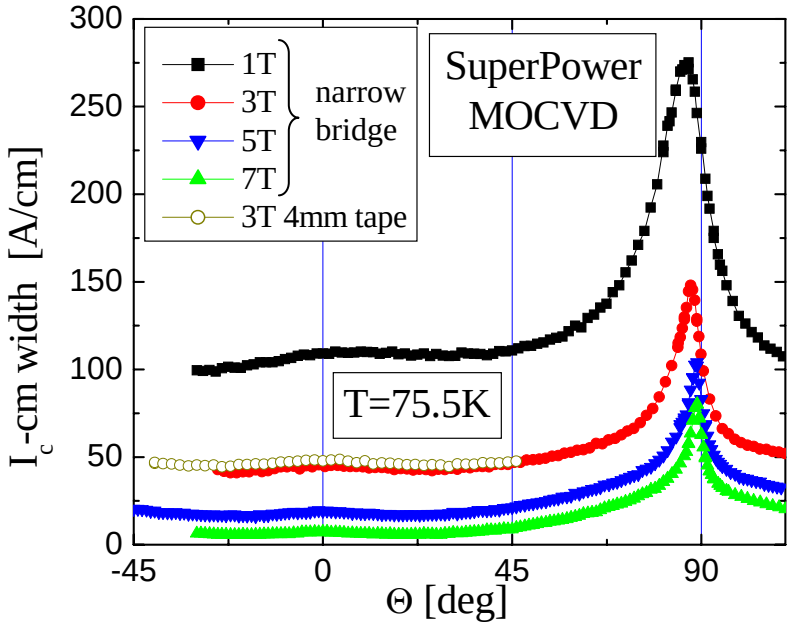
- I_c(H//c) in wide tapes before slitting?
- 4 mm wide final product:
  - I_c(X) at how many fields and orientations?
  - I_c(Θ)?
  - Ic(X) at 85K (liquid Ar) to test T_c uniformity?
  - ????
  - What distance between points? (related to speed)
  - Very fixed parameters (simple, easy to use and automatize) or flexible (more complex operation)

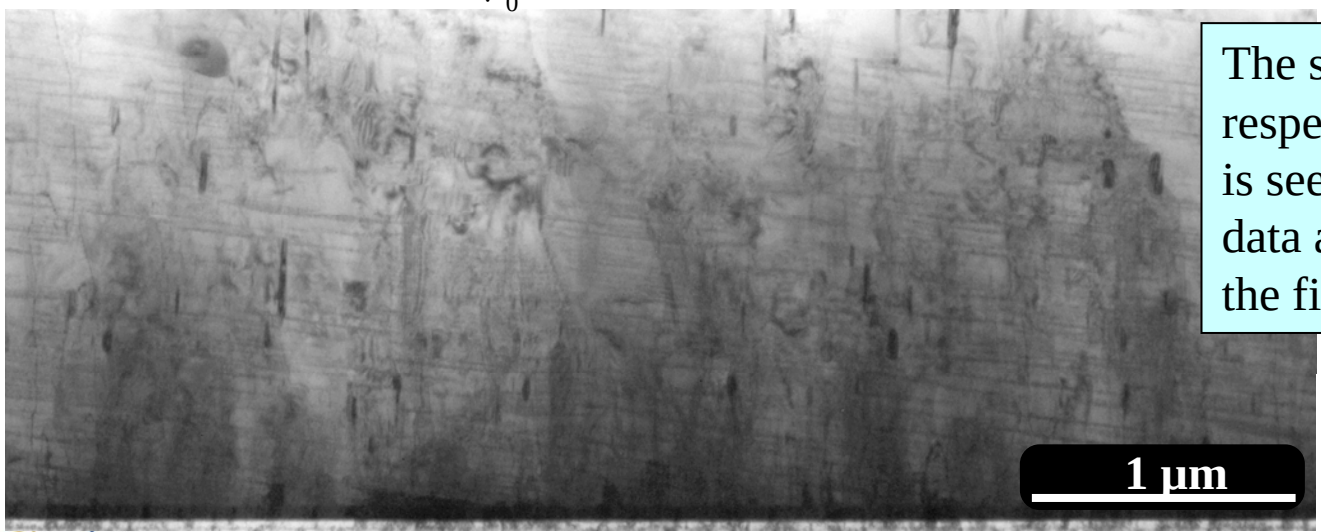
It is now the time to discuss this step with the companies

We have measured the pinning properties of a short piece of the SuperPower continuously processed, MOCVD champion sample.

(1) Transport, 75K





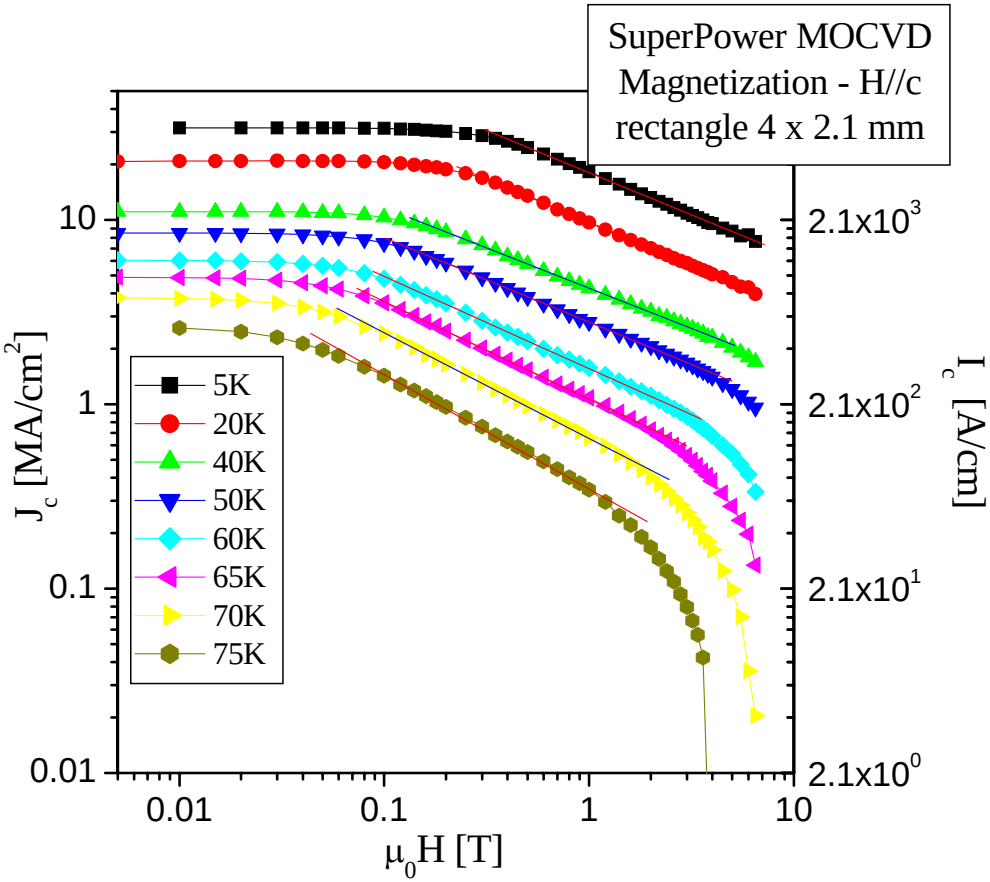


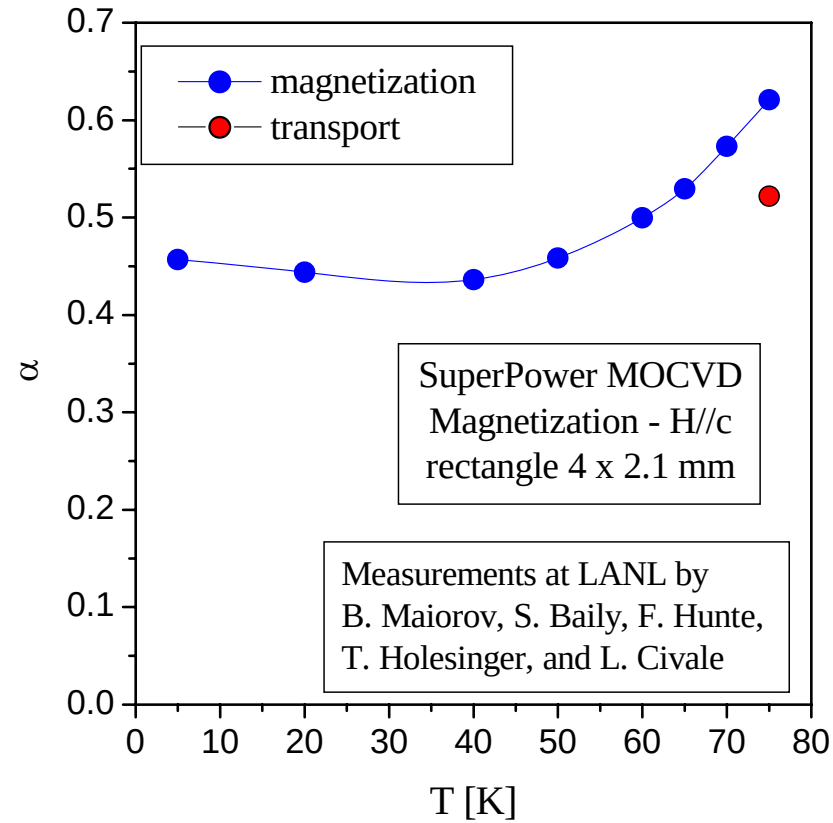
The small tilt of the defects with respect to the substrate plane, ~2°, is seen in the angular anisotropy J_c data as a shift of the ab peak from the film plane.

(see B. Maiorov *et al.*, APL 2005)

We have measured the pinning properties of a short piece of the SuperPower continuously processed, MOCVD champion sample.

(1) Magnetization (H//c)





I_c(5K,sf) = 6600 A/cm-width

I_c(5K,6.5T) = 1600 A/cm-width

The difference in a between magnetization and transport is due to creep effects (different voltage criterion), see Foltyn-Civale presentation, PR 2005

Results: LANL - American Superconductor Collaboration

- Developed an understanding of the microstructural development in multicoat MOD films.
  - Discovered the development of a discontinuous microstructure (interlayers) that would prevent a uniform through-thickness microstructure.
  - Showed that distinct chemical and structural microstructures can be maintained through processing in a multi-coat film with different precursor layer chemistries.
- Validated a powerful new strategy for tuning the $J_c(H,\Theta,T)$ properties via chemistry modification of individual precursor layers.
  - Individual layers can be tuned like standard 0.9 µm single-layer films.
  - Substantial cost savings for multi-coat MOD films. Can use well-established 0.9 µm MOD precursor chemistries to build up multi-coat films.
  - The modeling of the double-coat data with single-layer MOD film data implies a weak $J_c(t)$ dependence.
- Measured position dependence of $I_c(H,\Theta,T)$ in several long length tapes via new LANL measurement systems.
- Isolated a production issue that was affecting the in-field angular properties of the wires.
- Measured $J_c(H,\Theta,T)$ in short pieces of production line samples, including 500 A/cm-w class double-coat hybrids.

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Results: LANL - SuperPower Collaboration

- LANL successfully helped SuperPower bring their IBAD MgO production chamber on line (last visit of 2 LANL members to SuperPower on Oct 2005).
- Helped solve a serious delamination issue that was affecting production.
- 7 months after startup (May 2005), SuperPower reported record results on IBAD MgO.
  - 219 A/cm-w over 322 m.
  - 557 A/cm-w in short R&D sample.
- Showed that the microstructure of their champion MOCVD $Y_{0.9}Sm_{0.1}BCO$ films contains a uniform, high-density distribution of sheets of $(Y,Sm)_2O_3$ particles and a sparse array orthogonally oriented (Y,Sm)-Cu-O rods.
- Showed that the recent improvements in the MOCVD process to obtain the record results came from both reducing surface secondary phases during the first pass in the MOCVD chamber and developing a more uniform distribution of secondary phases within the film.
- Investigated pinning of champion ~550 A/cm-w film as a function of temperature and magnetic field strength and orientation, by transport and magnetization.

Results: LANL Wire Development

- Showed that high quality YBCO films could be obtained on reactively sputtered $Sm_xZr_{1-x}O_y$ buffer layers which will allow for a simplified IBAD MgO architecture and faster processing rates.
- Ultra-fine multilayer films were produced by PLD with periodicities less than 15 nm on stationary and moving substrates from a single target.
  - 5.5 nm periodicity for a multilayer film on a moving IBAD MgO template.
  - Distinct Eu-123 layers that are two unit cells thick.
  - Improved superconducting properties in applied magnetic fields.
- Process can be used for continuous processing of HTS coated conductors to produce a controlled and uniform nano-structured multilayer or second phase distribution.

Performance

- LANL has always had a strong characterization effort to support industry, institutional, and internal HTS development programs. This year, the characterization effort was formally emphasized as a new talk in the 2nd generation wire session because of its importance in wire development.
- To accomplish our goals we have pursued several parallel paths.
  - ✓ We integrated our resources with the wire manufacturers and other outside collaborators to compare and contrast superconducting properties and defect structures for different film processes.
  - ✓ We used several techniques, such as TEM, HRTEM, FIB, elemental spectral maps, SEM and XRD, to determine the structural properties.
  - ✓ We developed facilities that allow us to measure nondestructively the position-dependent electrical response (I-V curves) of long lengths of tape at different fields and temperatures. The modular design of our devices provides versatility and unique capabilities such as field-orientation dependence studies of long tapes.
  - ✓ We have compared the results with those obtained by static methods on short samples.
  - ✓ We applied these tools and knowledge to characterize state-of-the-art tapes and to troubleshoot scale up problems and provide feedback to our industrial partners.
  - ✓ Simultaneously, we produced and characterized our own high quality PLD films with carefully designed defect structures.

Research Integration

- Close ties with US Industry via CRADA and multi-institutional collaborations.
  - American Superconductor
  - SuperPower
- Started new CRADAs with
  - Metox
  - Oxford
- Close collaborative ties with Universities and National Laboratories
  - Argonne, Brookhaven, Oak Ridge, and Sandia National Laboratories
  - Air Force Research Laboratory
  - Universities of Wisconsin, Stanford, Houston, Florida
- Detailed information on characterization issues are communicated with partners through regularly scheduled meetings, teleconferences, and proprietary reports.
- Joint presentations and publications with partners and collaborators.
- Our characterization effort at LANL has provided data to 12 of the 21 talks in the 2nd generation wire and strategic research sessions.

FY2007 Plans

- In FY 2007 we will continue supporting our industrial collaborators in their scale up efforts. Based on the data obtained with our various tools and our accumulated knowledge of the structure-chemistry-property relationships in coated conductors, we will
  - provide our collaborators with information and guide that will help them to optimize their wires.
  - As needed, we will continue using our tools to troubleshoot problems in the scale up process.
  - We will work with industry to improve our long-length characterization tools and adapt them to their evolving needs.
  - We will also continue designing, fabricating and installing property measurement tools for our industrial partners.
- 2nd generation wire technology is reaching the level where we must be ready to switch from *characterization* of tapes to *qualification* of commercial products. To that end, in consultation with our partners, we will determine what is the minimum set of properties that must be monitored as part of a production line.
- Demonstrate a simplified IBAD MgO architecture with continuous processing using high-rate, reactively-sputtered $Sm_{1-x}Zr_xO_y$ films with YBCO deposition processes.
- YBCO films with controlled defect structures will be made by continuous processing using PLD and sectored targets. We will design and demonstrate a 2nd generation wire with flux pinning tuned for specific operation conditions.