Coated Conductor Characterization Studies

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FY2002 Plans

1. Develop, evaluate, and apply effective combinations of measurement tools for the interrogation and characterization of coated conductor embodiments

- elucidate composition/structure/performance relationships
- address solutions to issues limiting long-length coated conductor performance

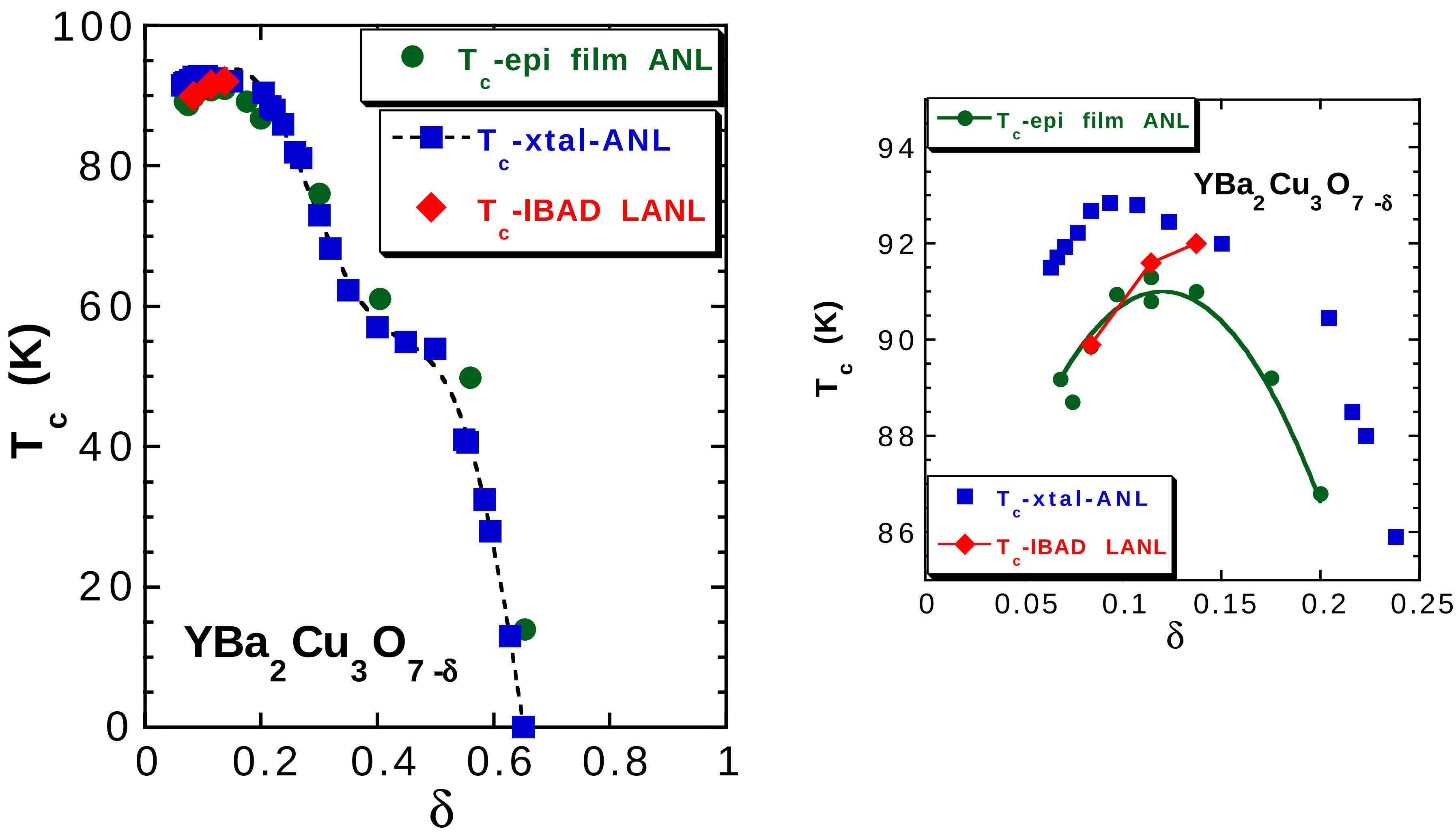
2. Extend Raman microscopy and synchrotron x-ray scattering methods to specimens obtained from the ANL coated conductor program and from, e.g., LANL and ORNL

3. Examine characterization methods that have potential utility as on-line instrumentation for process monitoring and control during coated conductor fabrication

4. Explore methods to improve J_c of coated conductors by improving grain boundary performance using samples from ANL, Ca-doped samples from Mannhart's group, and coated conductors from LANL and ORNL

5. Explore processing issues for joining coated conductors

Oxygenation of YBCO Coated Conductors



Oxygenation Protocol for YBCO Coated Conductors

Place sample in closed 'coffin' container made from YBCO - secure lid with Pt wire

Same procedure is used to adjust the 'coffin' to the right stoichiometry - this can take days to weeks

Place 'coffin' in closed Pt can and hang in a vertical tube furnace at ~ 450 ^oC

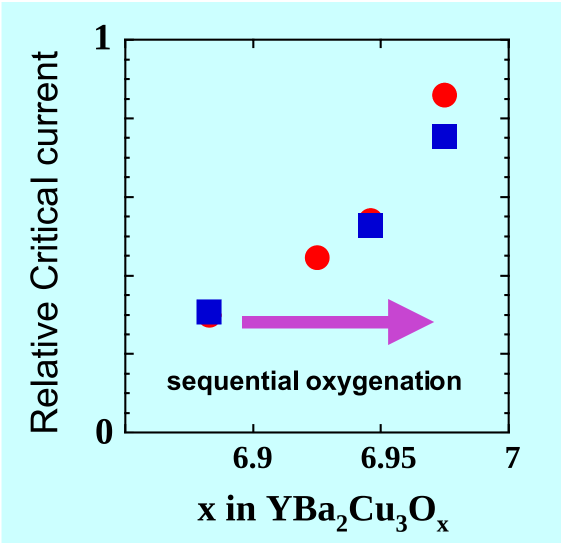
Adjust O_2 pressure with flowing gas mixture O_2/Ar or O_2/N_2 Monitor with Zr cell (Ametek)

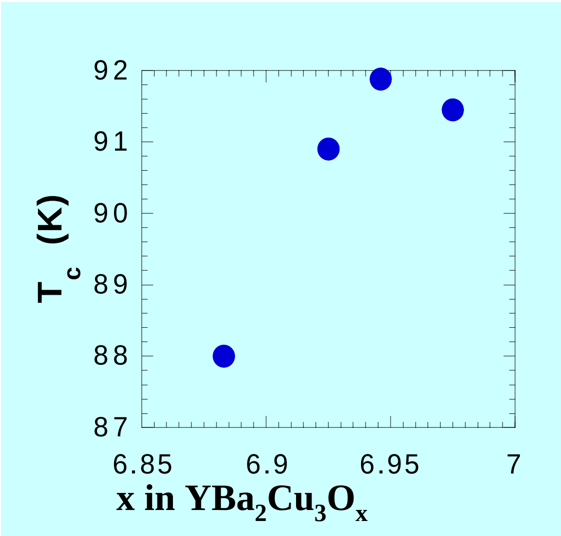
Time depends on various parameters - for films at optimum doping it can be only minutes

When equilibrated, quench Pt container, YBCP 'coffin' and sample into liquid nitrogen

Coffin acts as an O_2 reservoir to preserve stoichiometry during cooldown, in particular at the 'fictive' temperature

Oxygenation of YBCO GBs



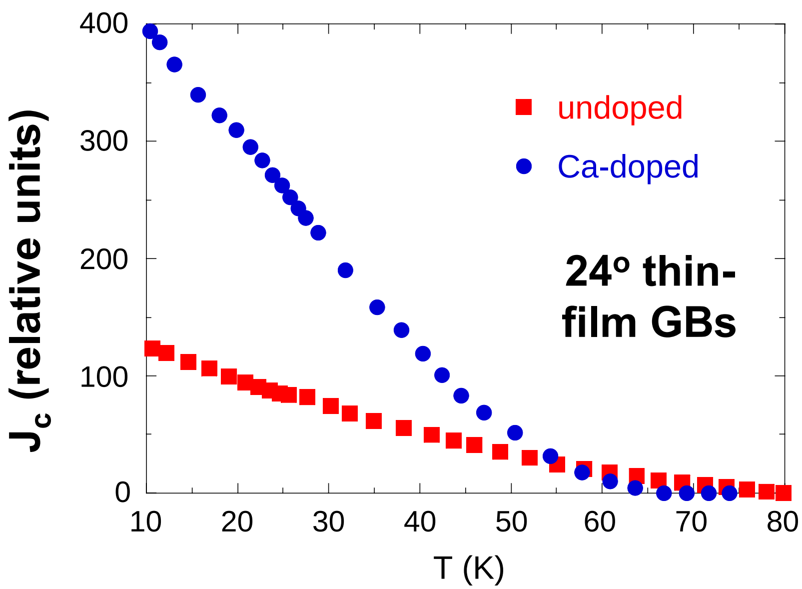


GB angles 12° and 28°

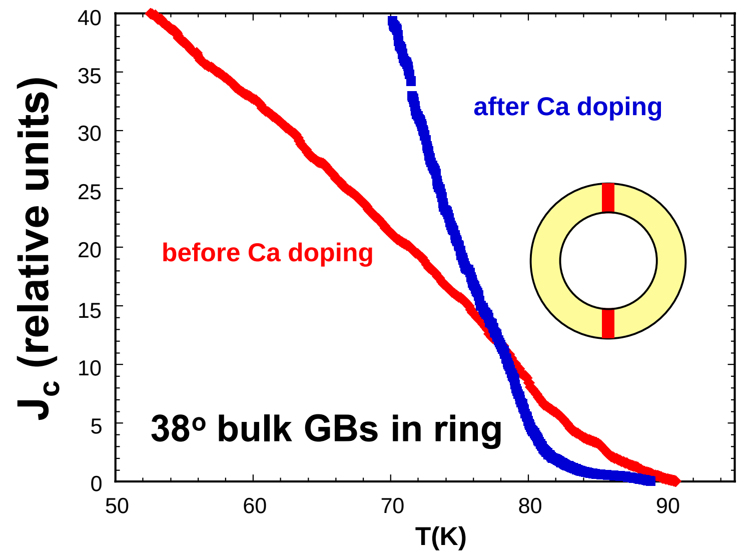
Ca^{++} doping of YBaCuO GBs

Ca^{++} substitutes for Y^{+++} in YBCO and is known to lower the bulk T_c

In collaboration with J. Mannhart, Augsburg University



Consistent results for bulk GBs and thin film GBs for Ca^{++} doping

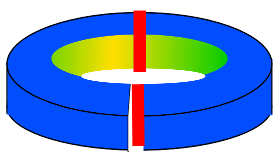


How does oxygen doping affect Ca^{++}-doped GBs *and bulk*?

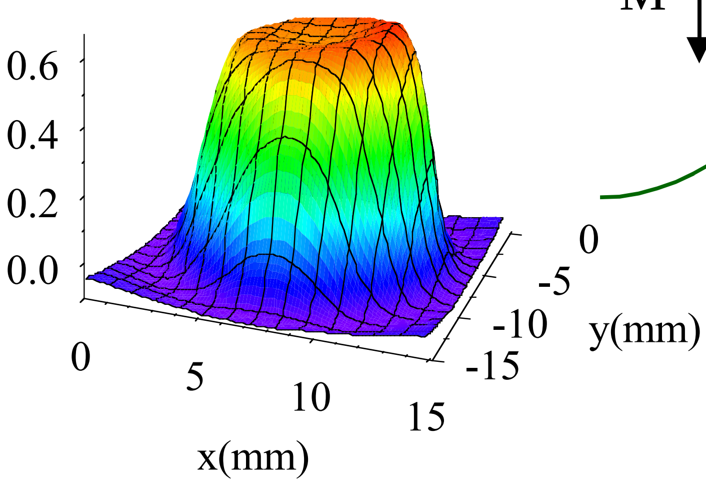
Rings Provide Independent Data on ‘Bulk’ and GBs

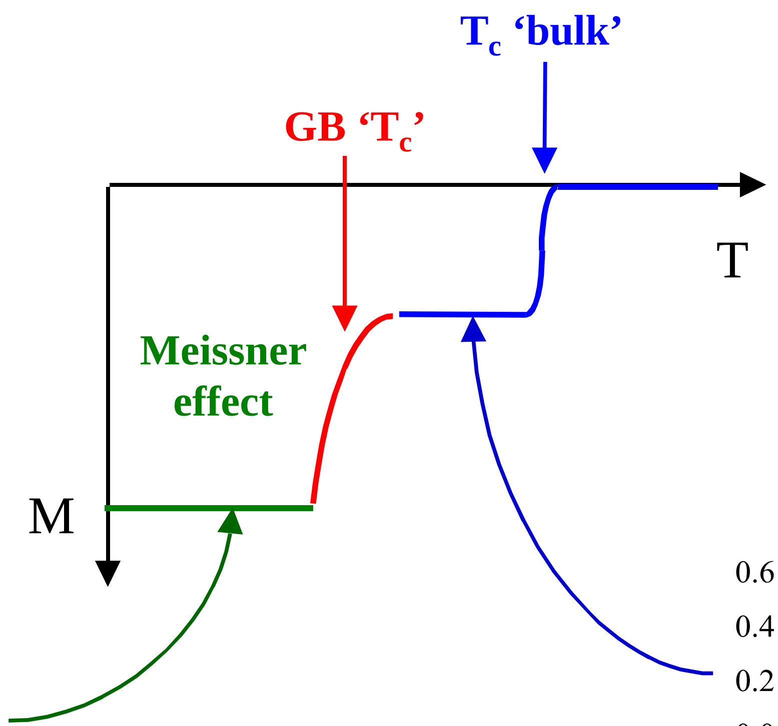
Method: cool in zero field, the apply 0.01-100 Oe, warm up in field

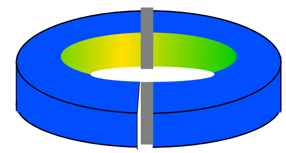
GBs superconducting at low temperature

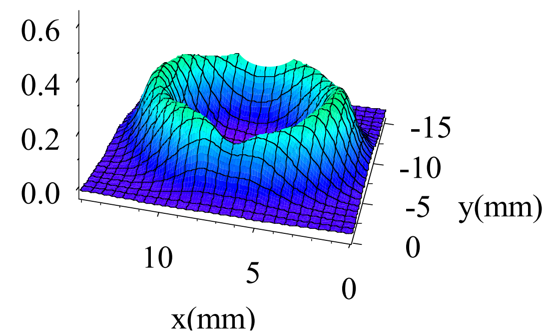


Superconducting ring with 2 GBs









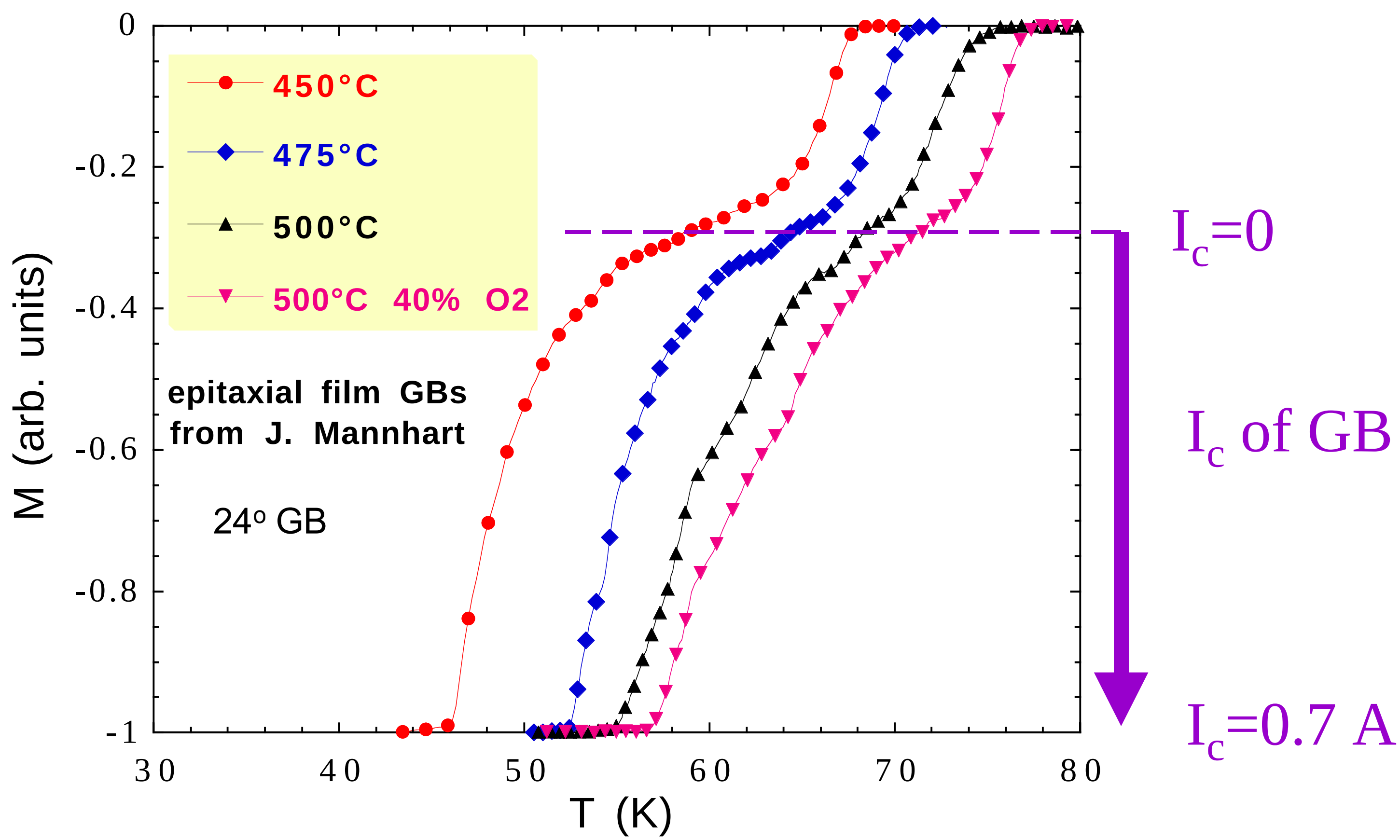
Superconducting ring with non superconducting GBs

-M ~ excluded field

Oxygen Loading of Ca-Doped GBs

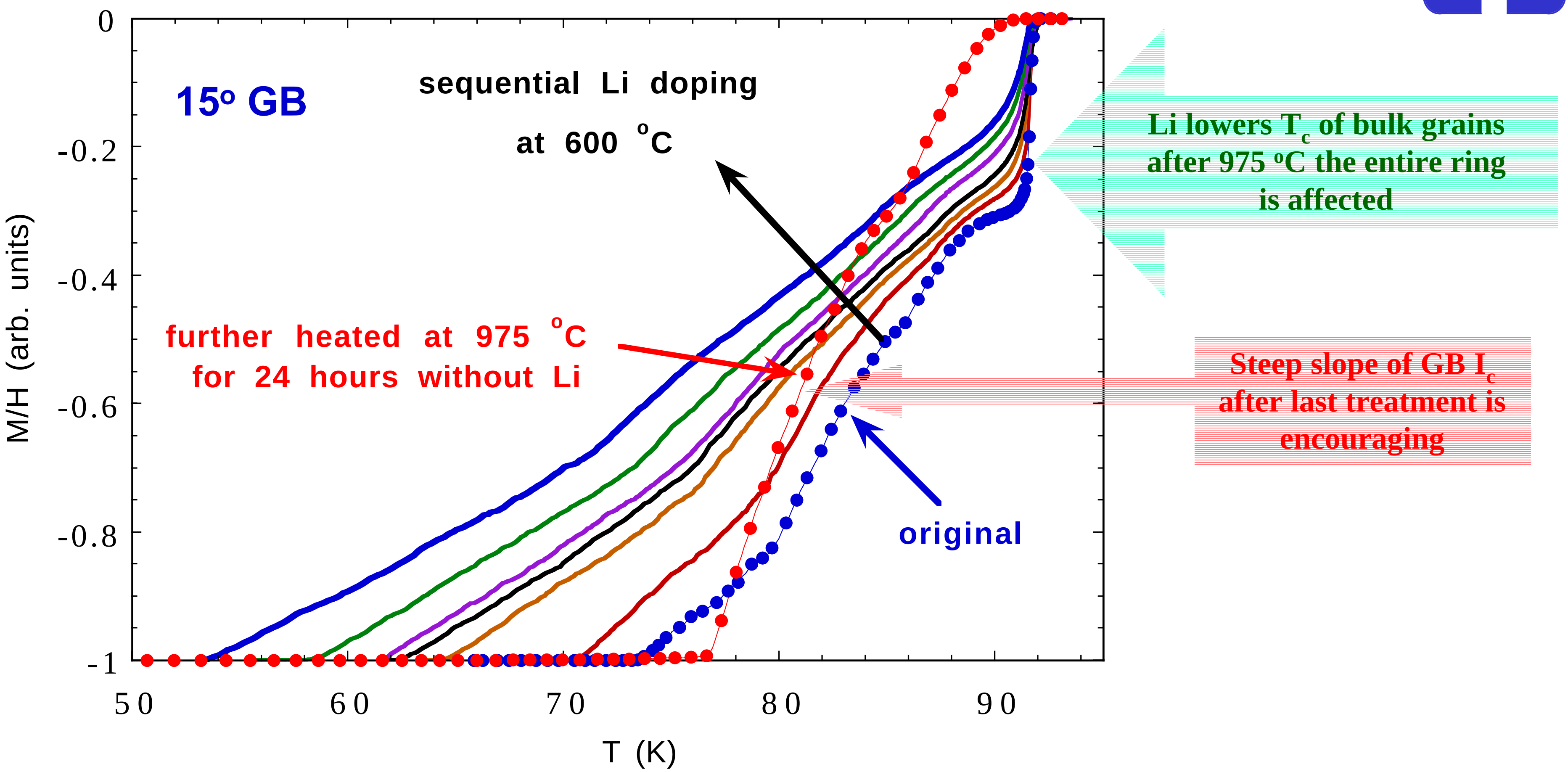
Reducing sample increases T_c and J_c

Although one can go too far, it can be recovered!



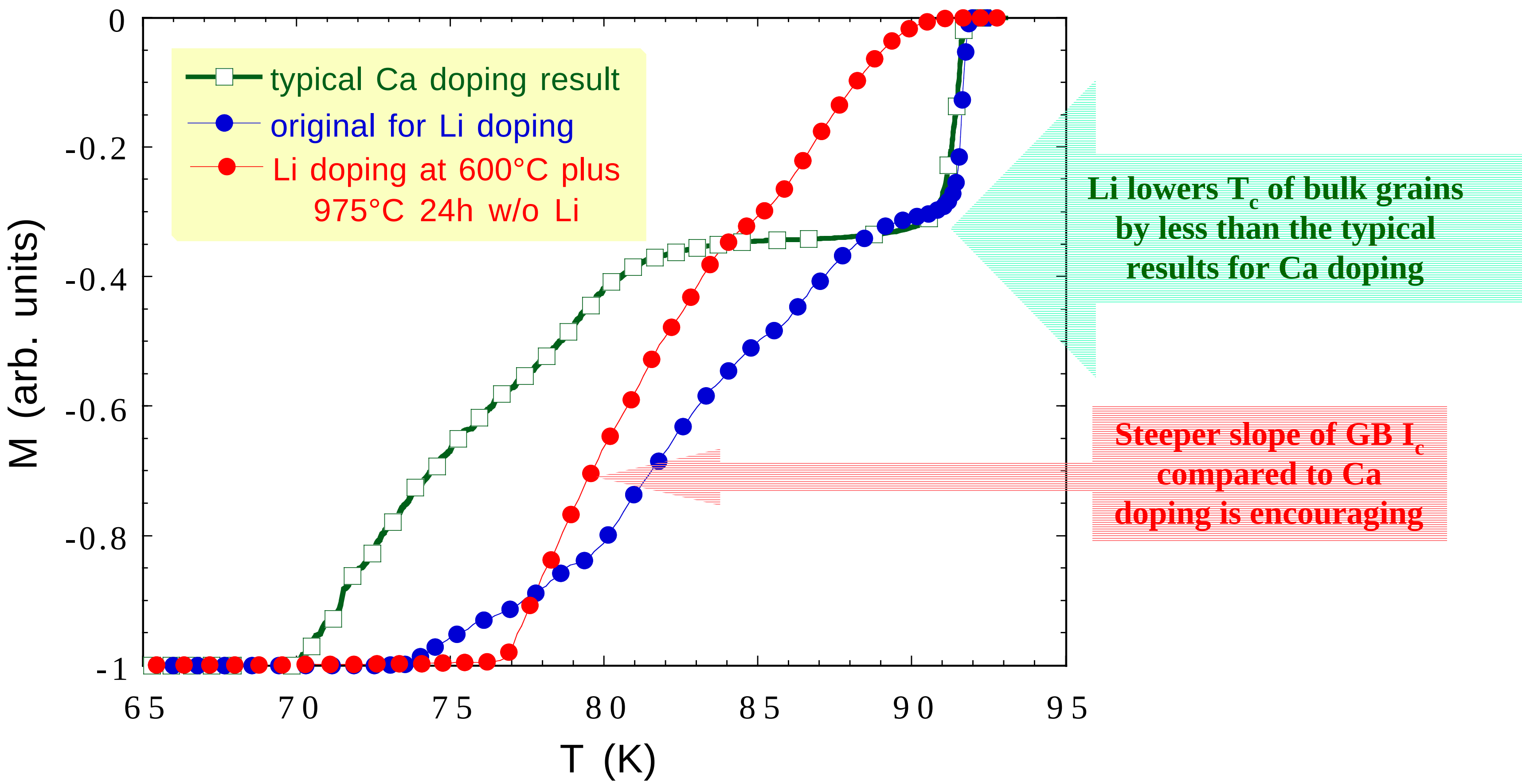
Plan: set T_c of bulk grains by O_2 doping and independently optimize GB oxygen -- may be possible because of x10 faster GB diffusion

Doping of YBCO GBs with Li



Plan: apply 975 °C treatment after less Li doping

Preliminary Comparison of Li- and Ca-Doped GBs



Results look promising and much is yet to be learned about optimum Li processing

Summary of Dopants in YBCO GBs

O has a big, beneficial effect, even when YBCO is overdoped beyond maximum T_c

Optimum O loading depends on other dopants

Alkaline earths (Mg, Ca, Sr, Ba) dope GBs at 900-950 ^oC

Ca improves low temperature I_c, lowers bulk T_c

Sr may act as a sintering aid (improves poor GBs)

Ba may be beneficial (preliminary result)

Alkalis (Li, Na, K, Rb) readily dope GBs at 600-700 ^oC

Generally, J_c is depressed,

but precise optimization of doping may improve J_c

e.g., Li doping has shown improvement

Bi - GB doping observed at 800 ^oC, but Bi attacks bulk YBCO

Zn - GB doping observed at 950 ^oC, but it depresses J_c

Rare earths (Yb, Sm, Ce) and Al, Zr, Ni, Cr

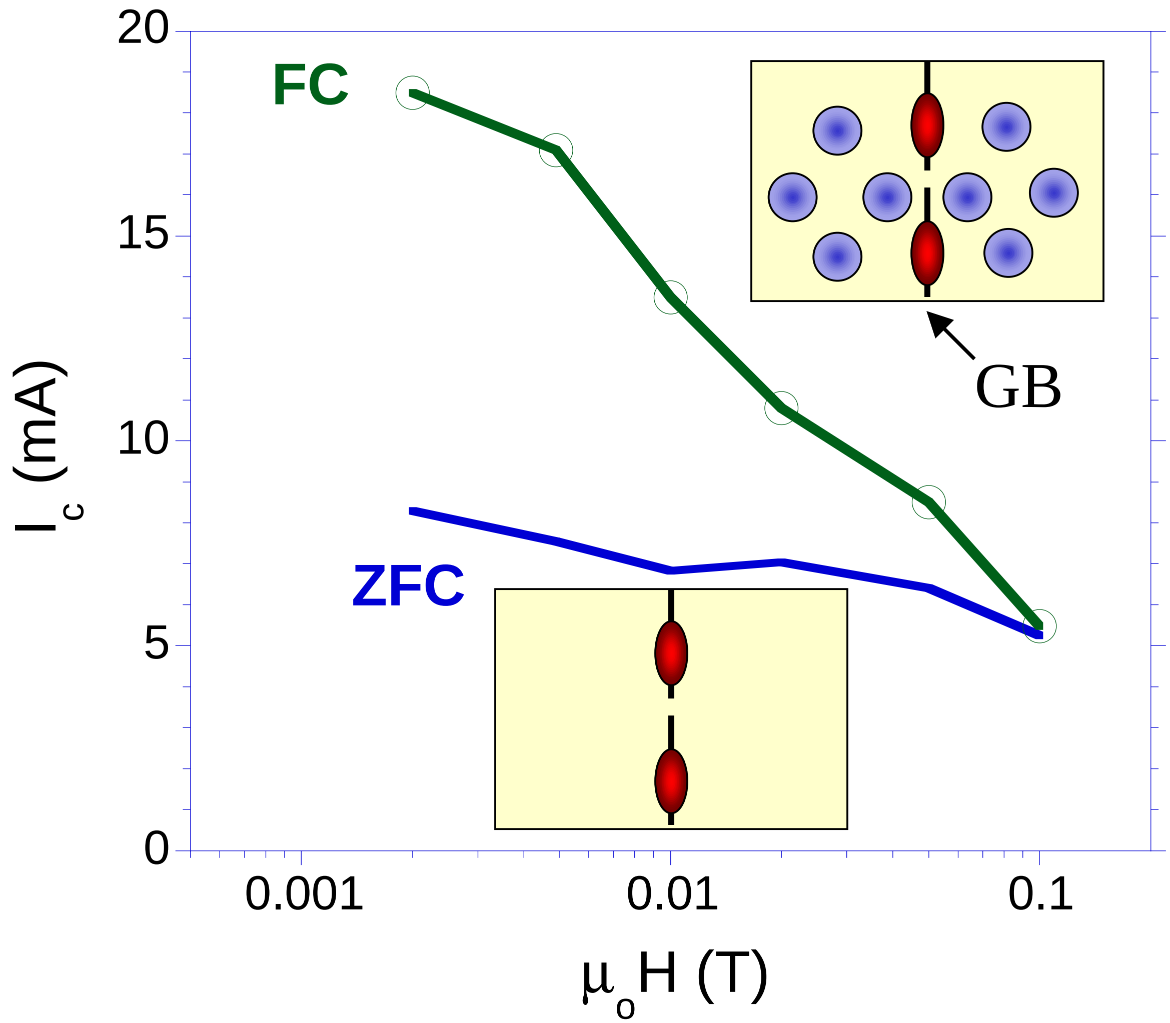
no improvement has been observed in J_c

GB doping not yet confirmed

Field History Dependence of I_c

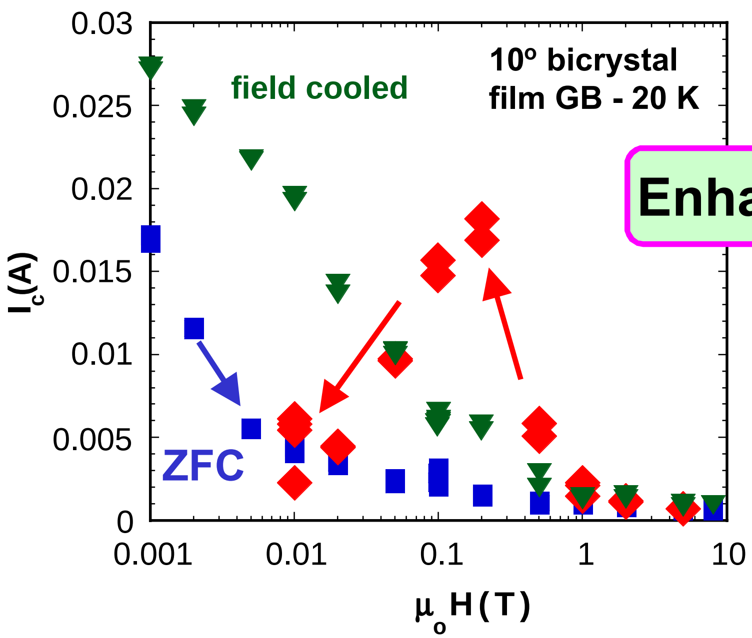
Patterned GB on RABiTS Coated Conductor

collaboration with Christen and Feenstra, ORNL

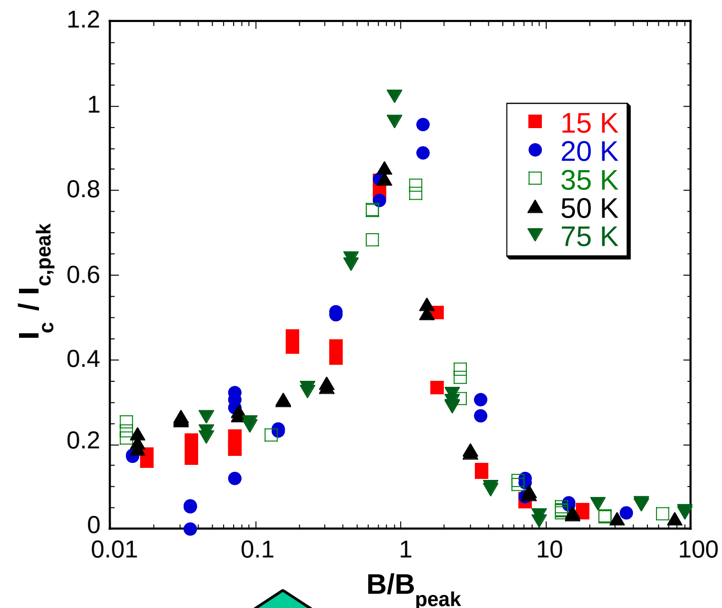


Enhanced I_c for field cooling (FC): GB vortices pinned by Abrikosov vortices in banks of GB

10º Bicrystal Thin-Film GB and ZFC Hysteresis



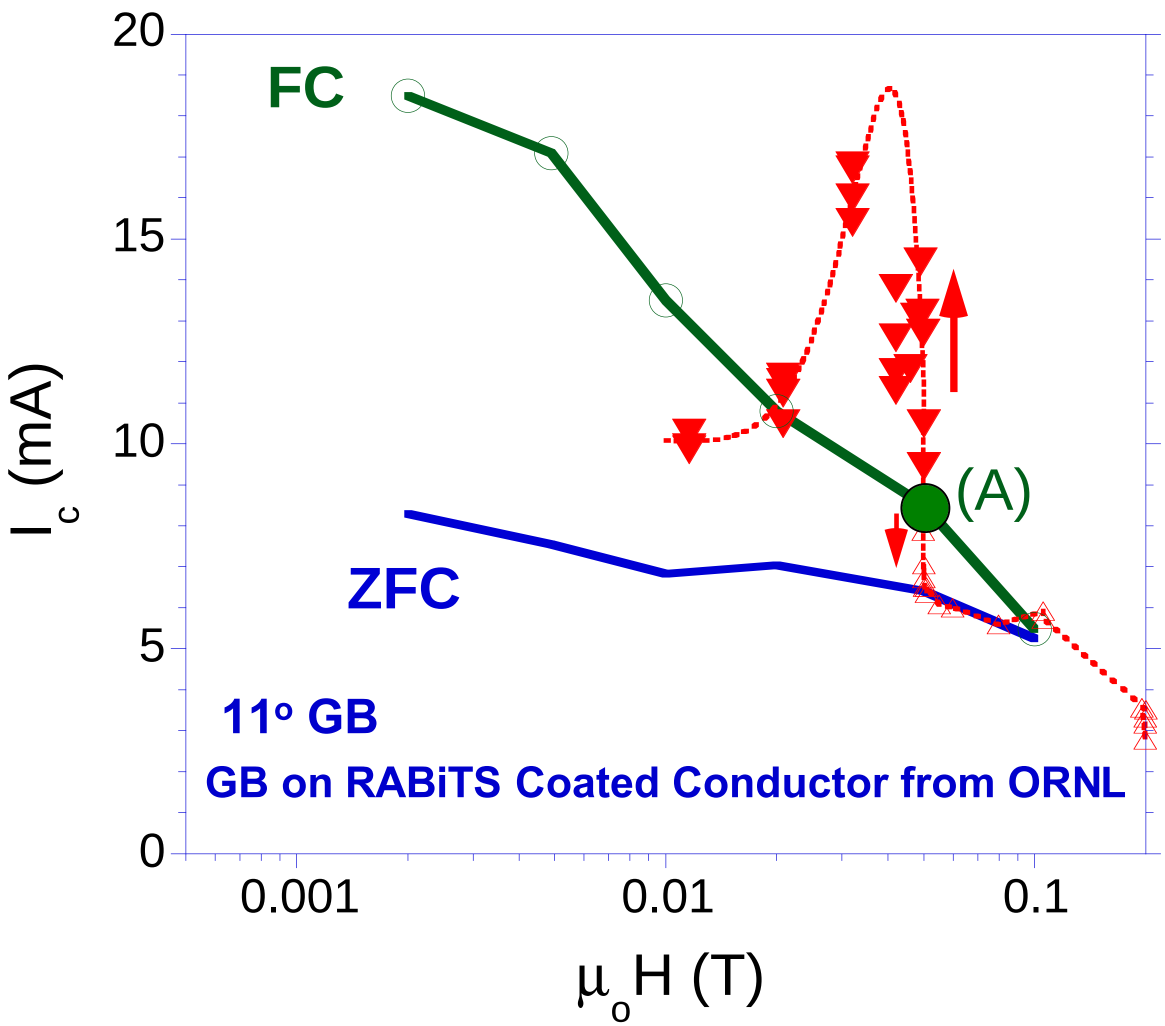
Enhancement of x5 at 0.05 T

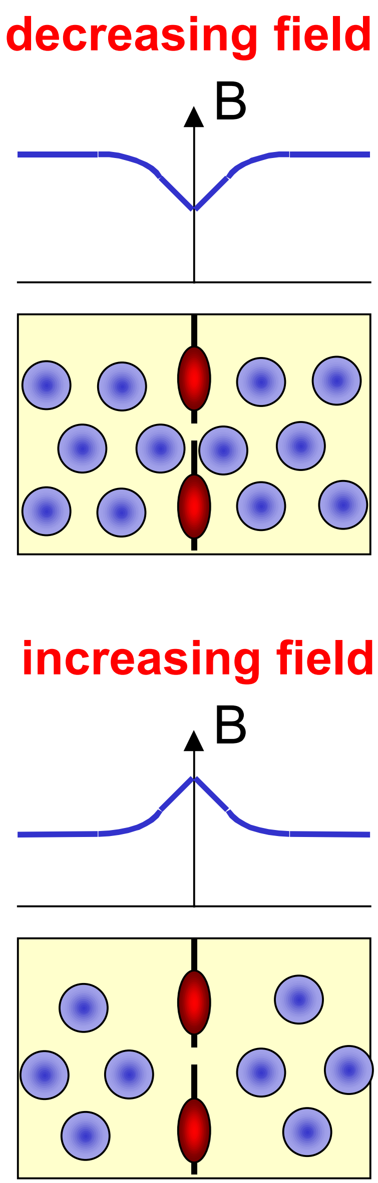


- **return path** higher than **FC data**

- **return path peak** scales with temperature

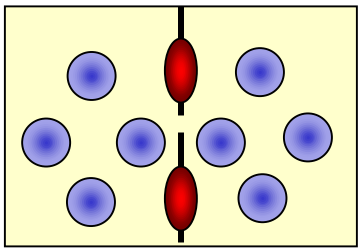
Change Field after FC in Coated Conductor GB



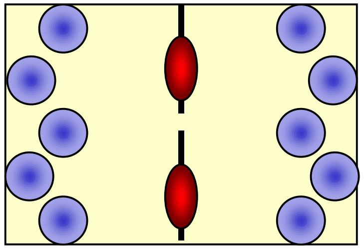


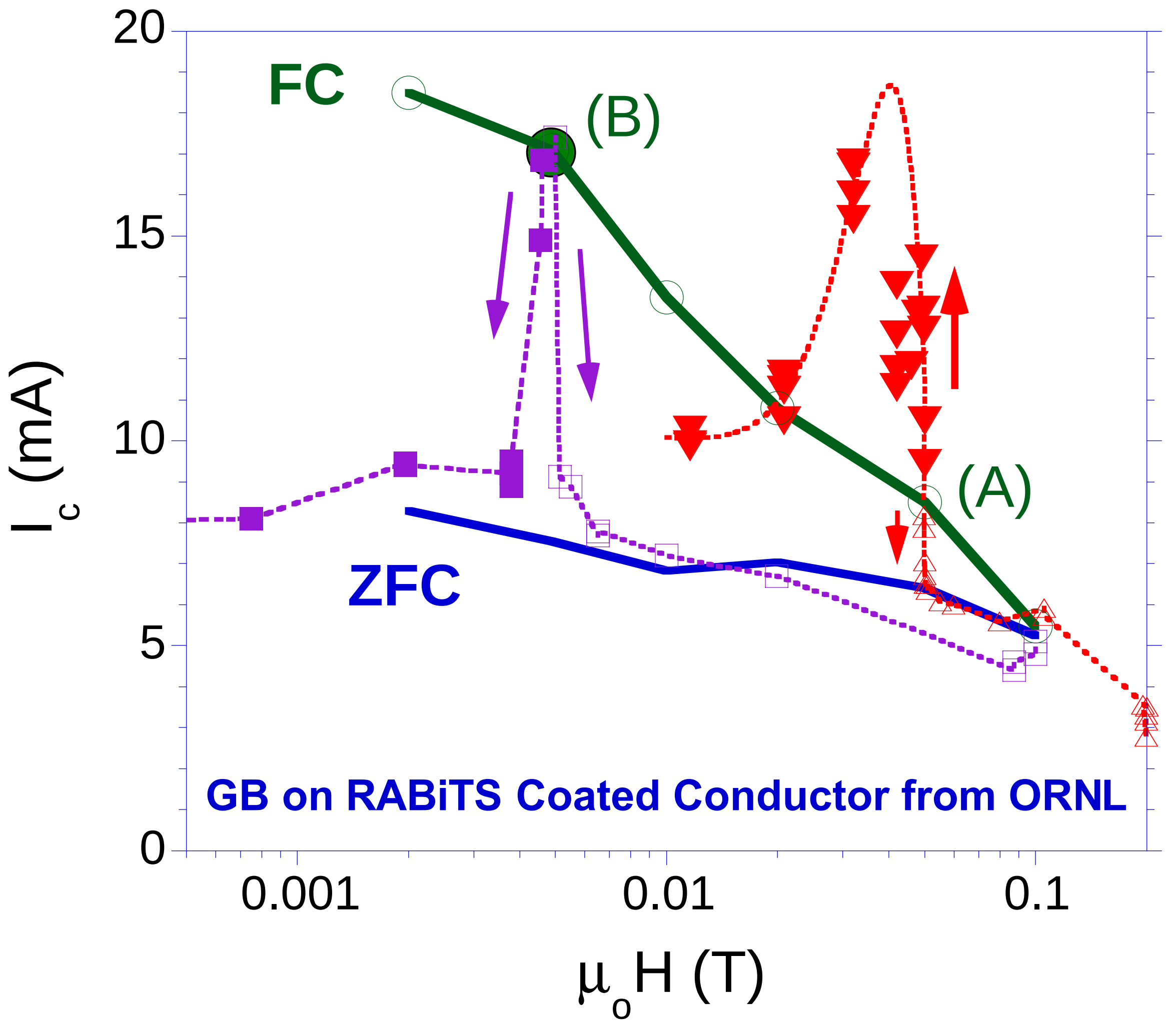
Change Field after FC in Low Fields

field cooled

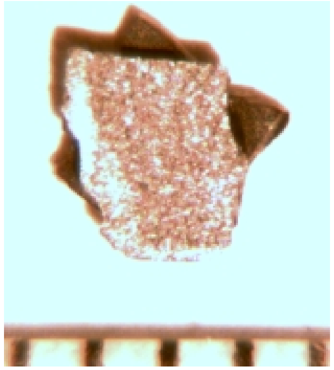


low-field vortex-free zone at edge





Joining of Coated Conductors



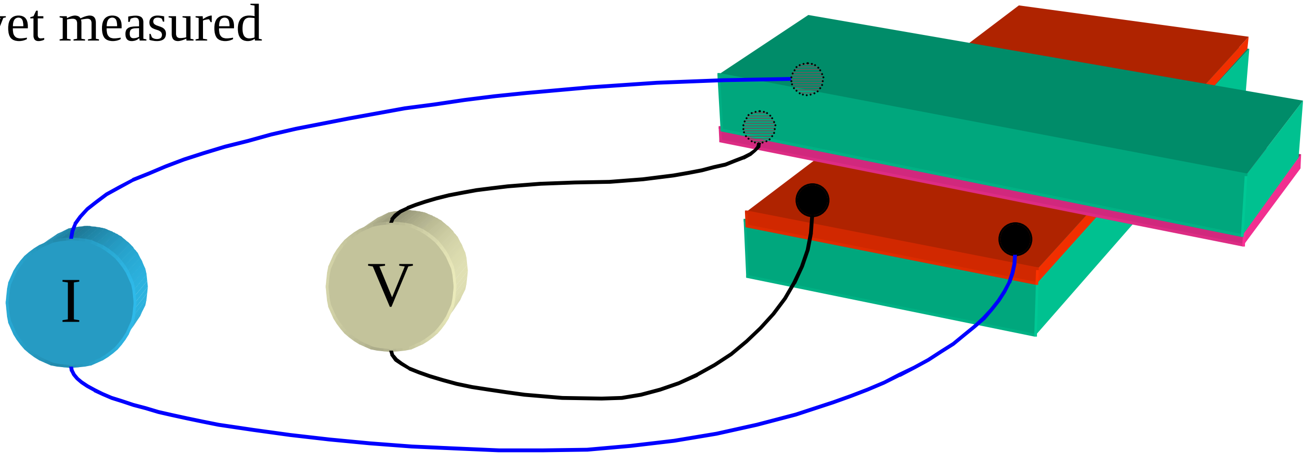
Epitaxial YBCO on SrTiO substrate

Cut into rectangles and welded w/o filler

Mechanically sound

Contact is superconducting in liquid N_2 with 4 probe transport measurement

I_c not yet measured



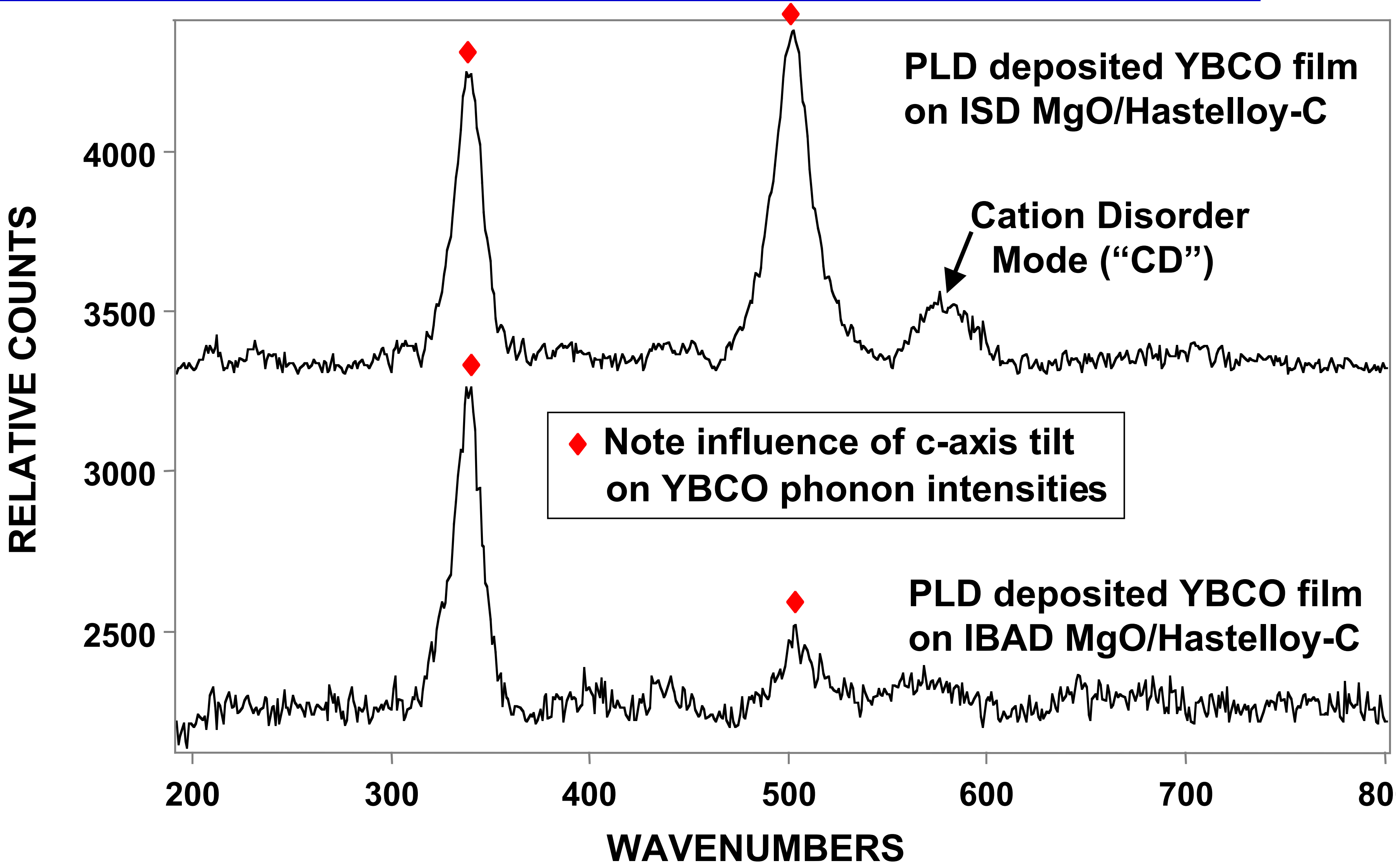
Coated Conductor Characterization by Raman Microscopy and X-ray Scattering

- **Raman Microscopy (RM)**
  - a axis grain growth in MBCO films (M = Y, Eu, Er)
  - cation disorder
  - tetragonal (T') phase separation
  - growth of PLD YBCO on silver and Ag-Cu alloys
  - excitation laser wavelength dependence
  - testing of RM configurations for in situ and on-line monitoring (with ChemIcon, Inc.)

- **X-ray Scattering at the APS**
  - texture quality of MBCO films (M = Y, Eu, Er)
  - strain effects and epitaxy transmission
  - Tc/Jc connection to twinning structures

[FY 2002 program includes substantive collaborations with LANL and ORNL, as well as ongoing work with AMSC and IGC]

Microprobe Mode Raman Spectra of YBCO on ISD and IBAD Substrates: The Effect of c-axis Tilt

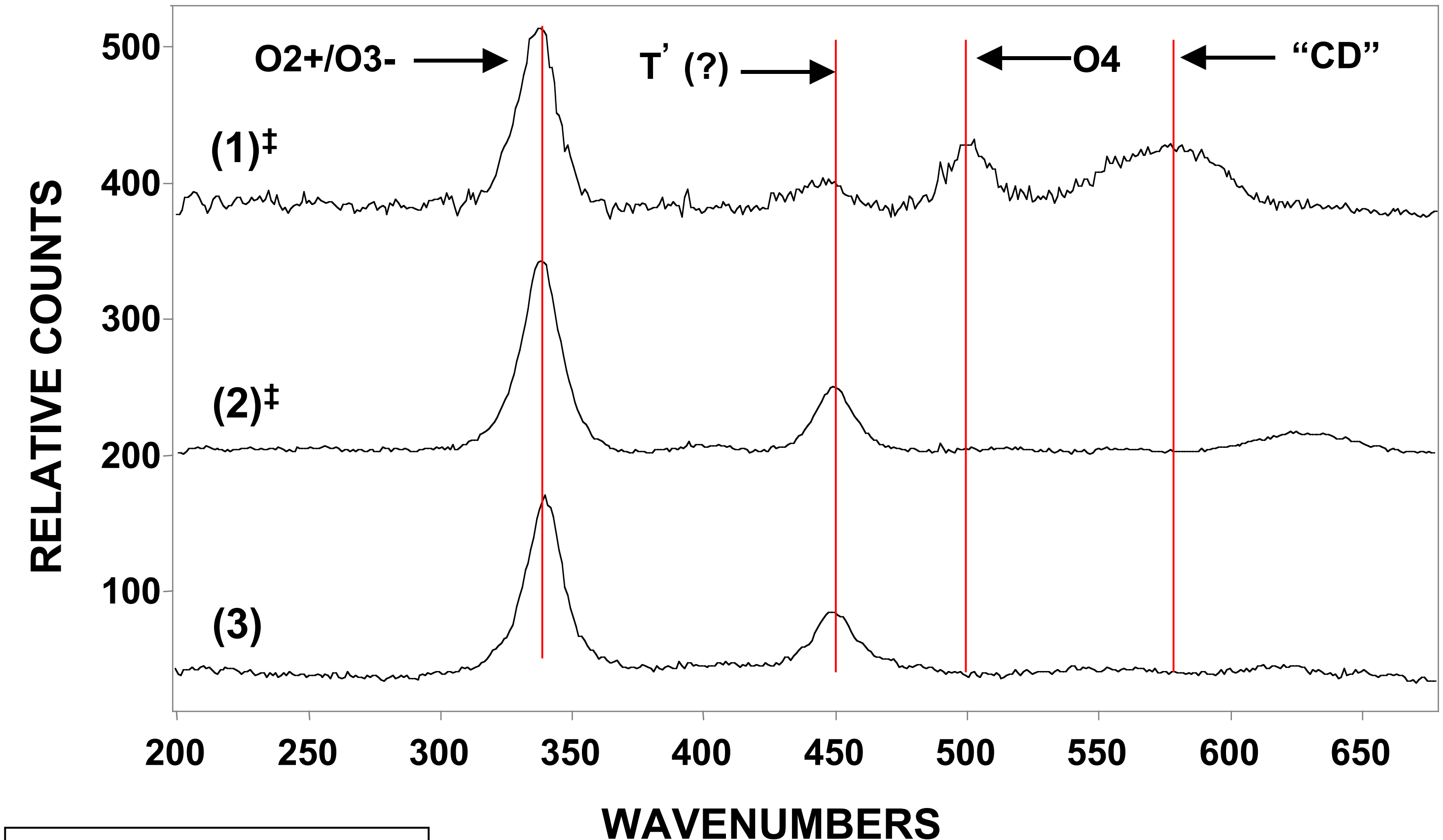


Evidence of Phase Separation in Some Coated Conductor Specimens: Is it the T' Phase?

(1) Y-123 (1.2 µm, PLD) / Sm-123 (0.1 µm, PLD) / Y-123 (1.2 µm, PLD) / CeO_2 / YSZ* / Hast. C

(2) Y-123 (ca. 1 µm, PLD) / CeO_2 / YSZ / CeO_2 / RABiTS nickel**

(3) Tetragonal Y-123 melt-processed/textured, bulk specimen***



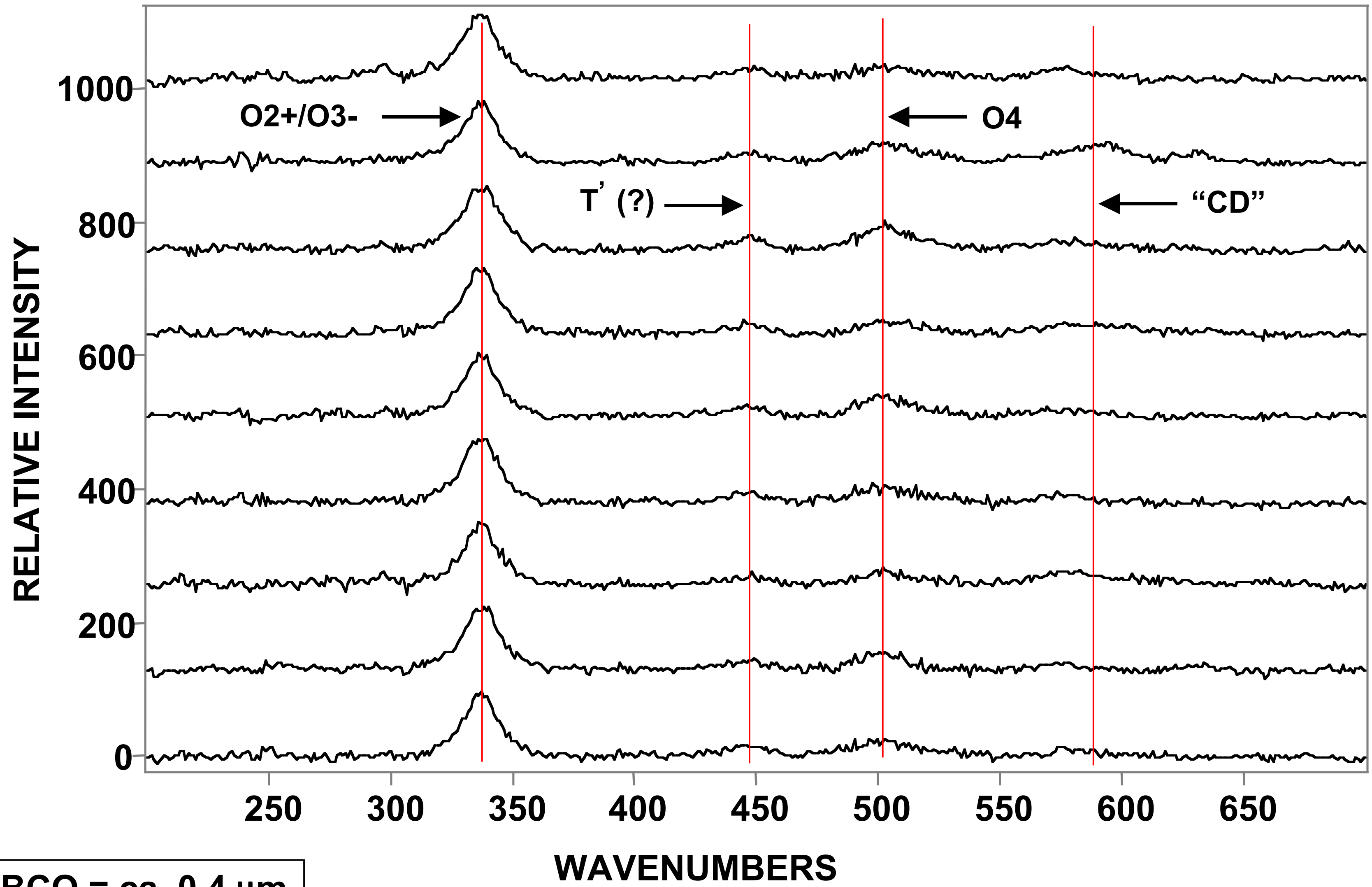
*German IBAD YSZ; PLD by S.R Foltyn; Jc (75 K, sf) = ca. 1.4 MA/cm²

**ORNL RABiTS; PLD by S.R. Foltyn; Jc (77 K, sf) <0.2 MA/cm²

***Sample obtained from Pavel Diko

‡Collaboration with S.R. Foltyn, LANL

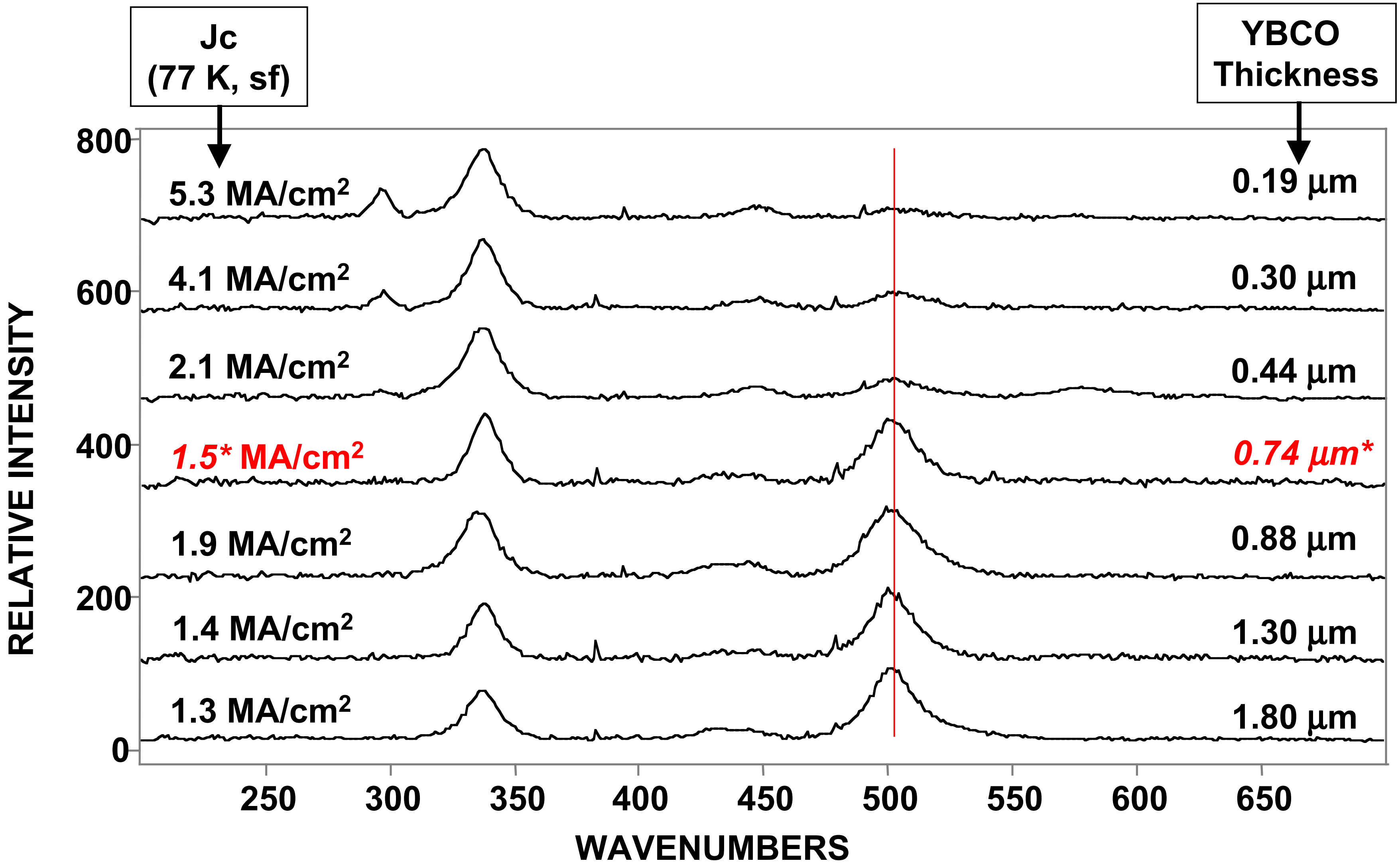
Raman Microprobe Spectra from a 3 mm by 3 mm Segment of a YBCO (PLD) Film* on $SrTiO_3$ (STO) provided by A. Goyal, ORNL



*YBCO = ca. 0.4 µm
Jc = ca. 2 MA/cm^2

Averaged Raman Microprobe Spectra of YBCO (PLD) Films on $SrTiO_3$

[Specimens provided by A. Goyal, ORNL]



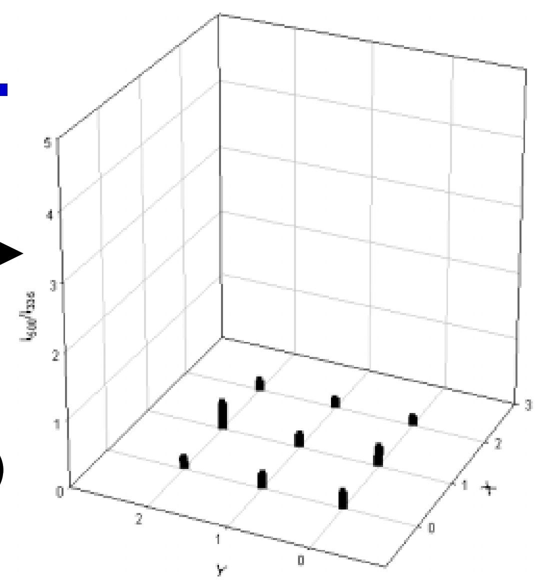
*YBCO/$LaAlO_3$

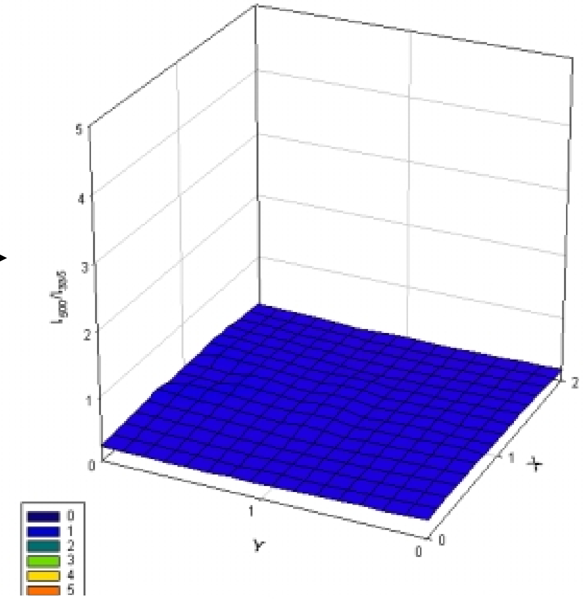
Texture Maps From I_{500}/I_{340} Raman Scans

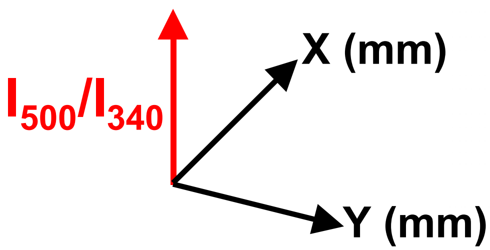
Raman Microprobe Scans | **Computed Texture Map**

YBCO (PLD)/STO*

YBCO = 0.19 µm
Jc = 5.3 MA/cm^2

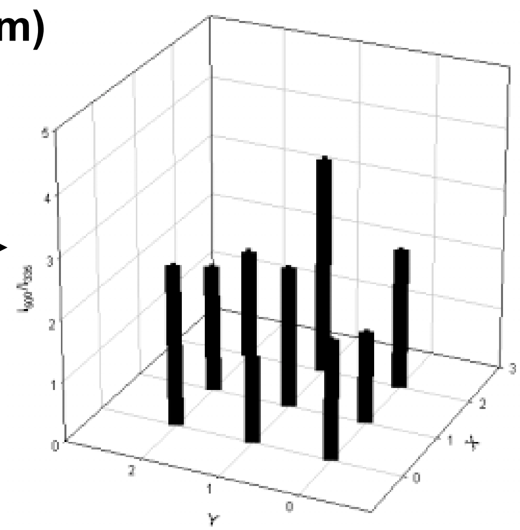


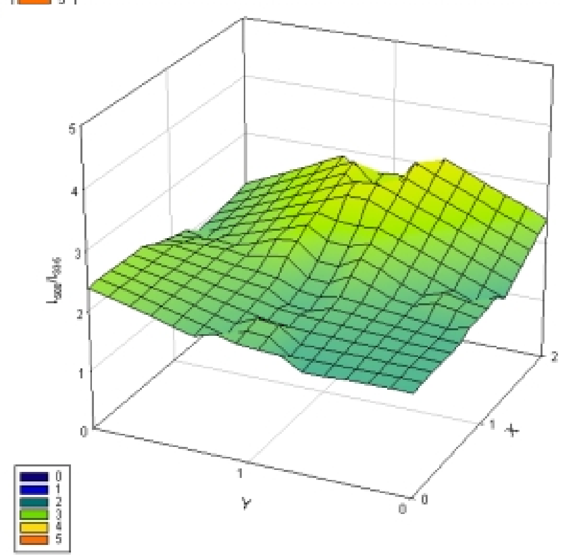




YBCO (PLD)/STO*

YBCO = 1.80 µm
Jc = 1.3 MA/cm^2

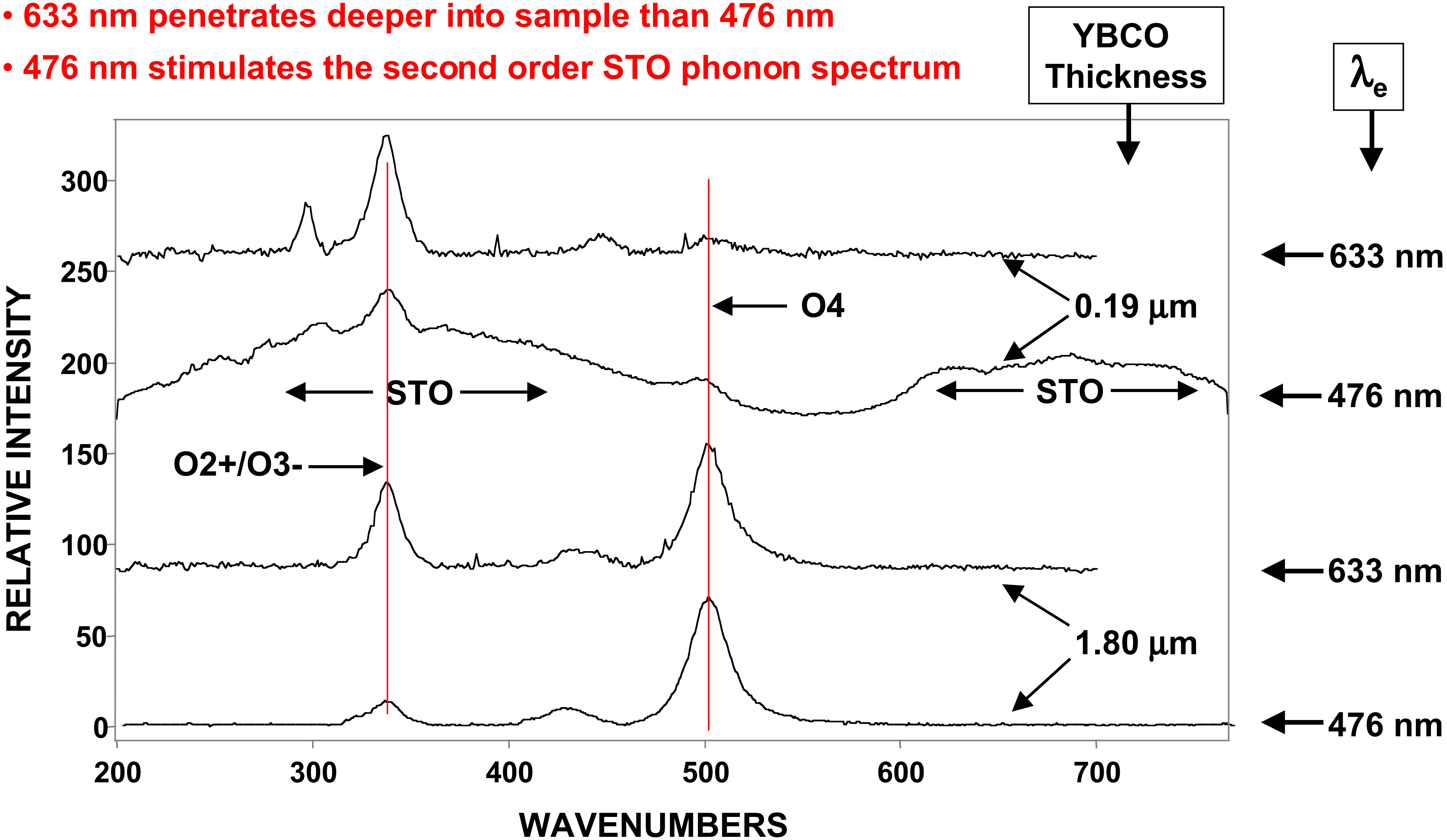




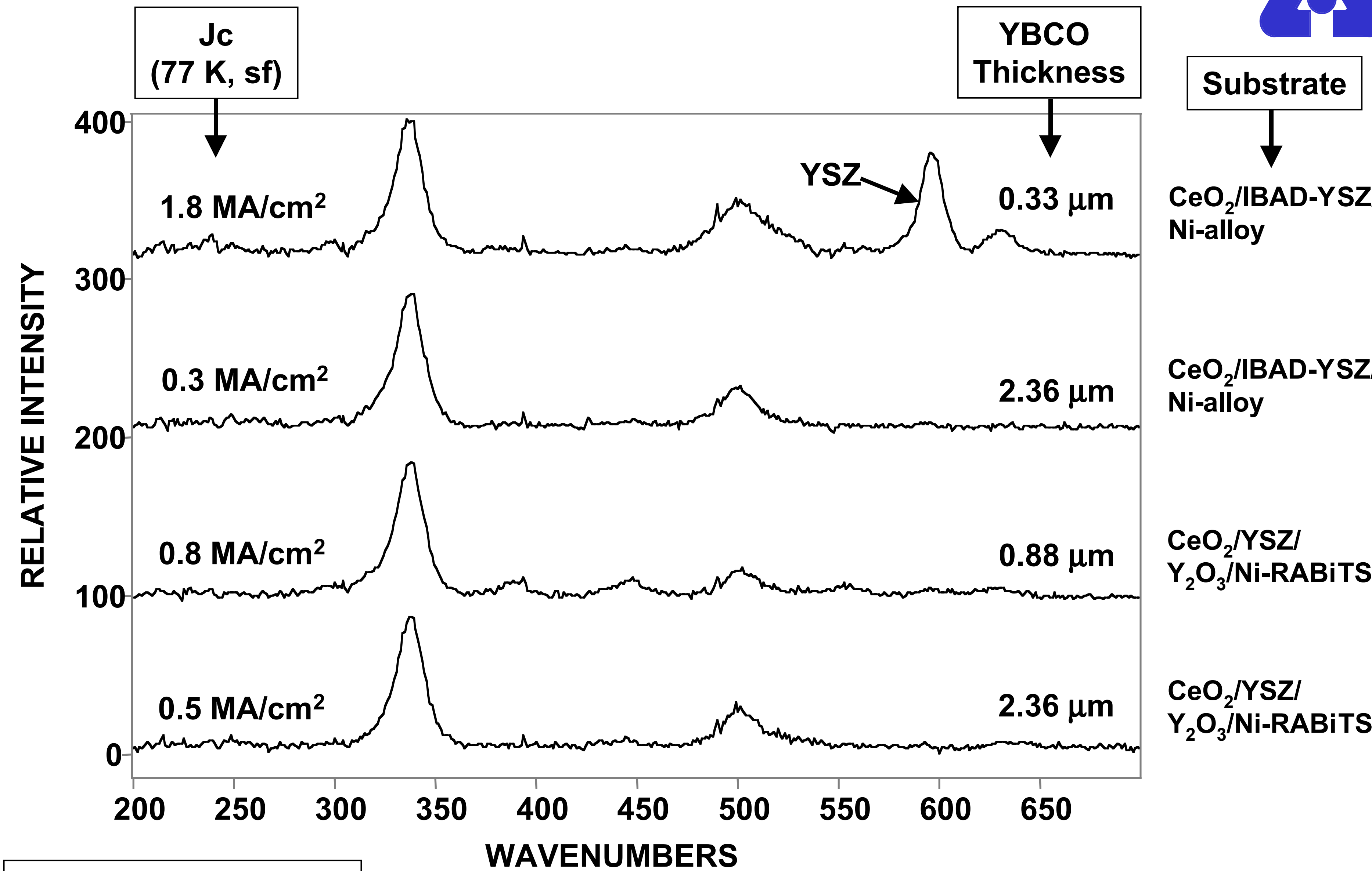
*A. Goyal, ORNL
Series Continued

Excitation Wavelength (λ_e) Dependence of Raman Spectra: YBCO (PLD) Films* on $SrTiO_3$ (STO) provided by A. Goyal, ORNL

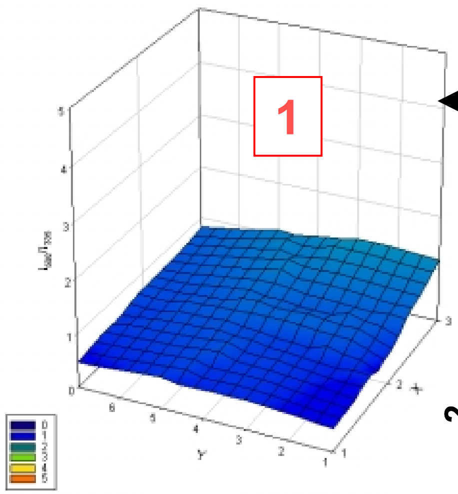
- 633 nm penetrates deeper into sample than 476 nm
- 476 nm stimulates the second order STO phonon spectrum



Ex Situ (Ba-F) YBCO Films* on ORNL RABiTS and LANL IBAD Substrates

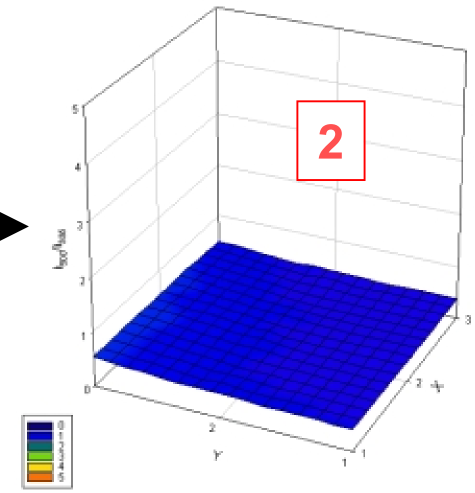


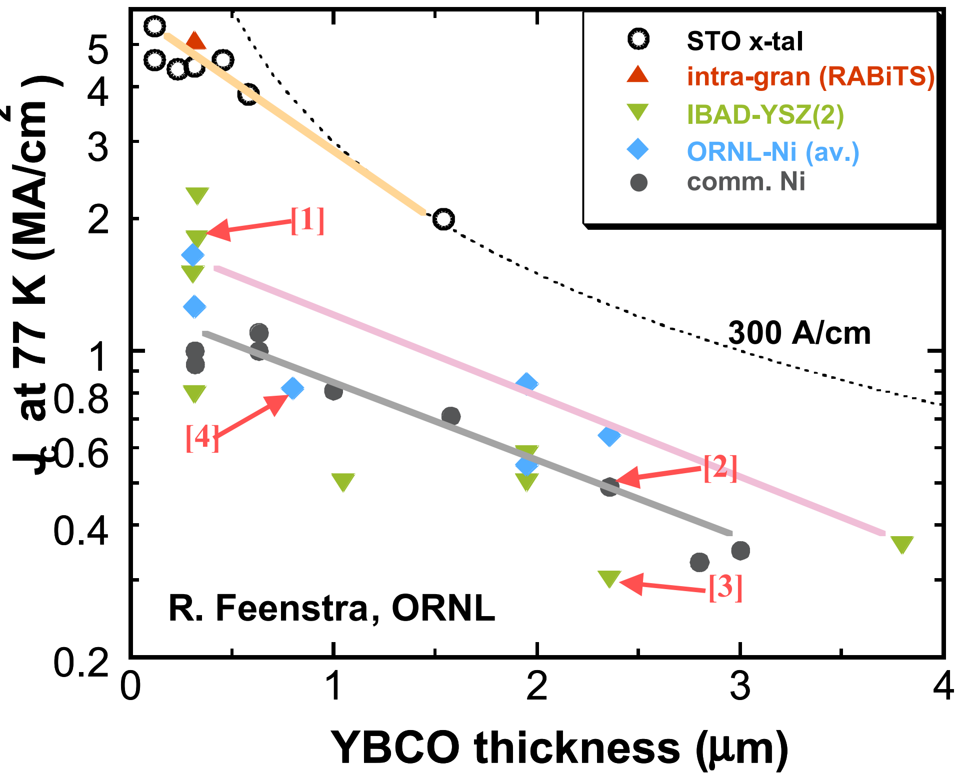
*Samples provided by R. Feenstra, ORNL



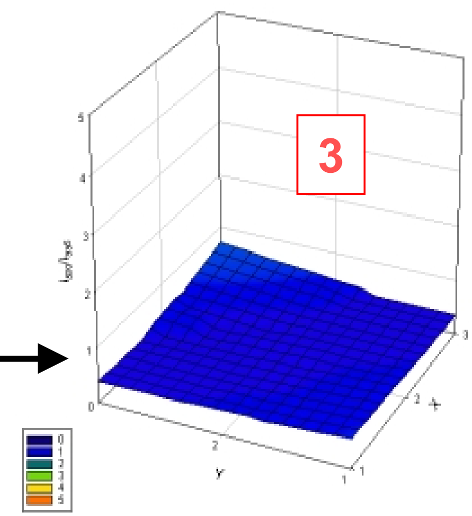
IBAD / 1.8 MA/cm² (0.3 µm)

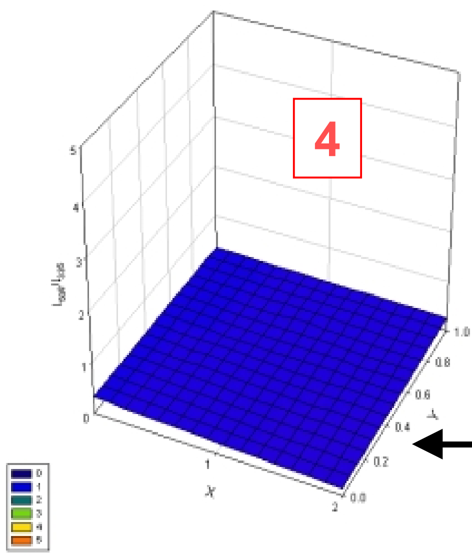
RABiTS / 0.5 MA/cm² (2.4 µm)





IBAD / 0.3 MA/cm² (2.4 µm)

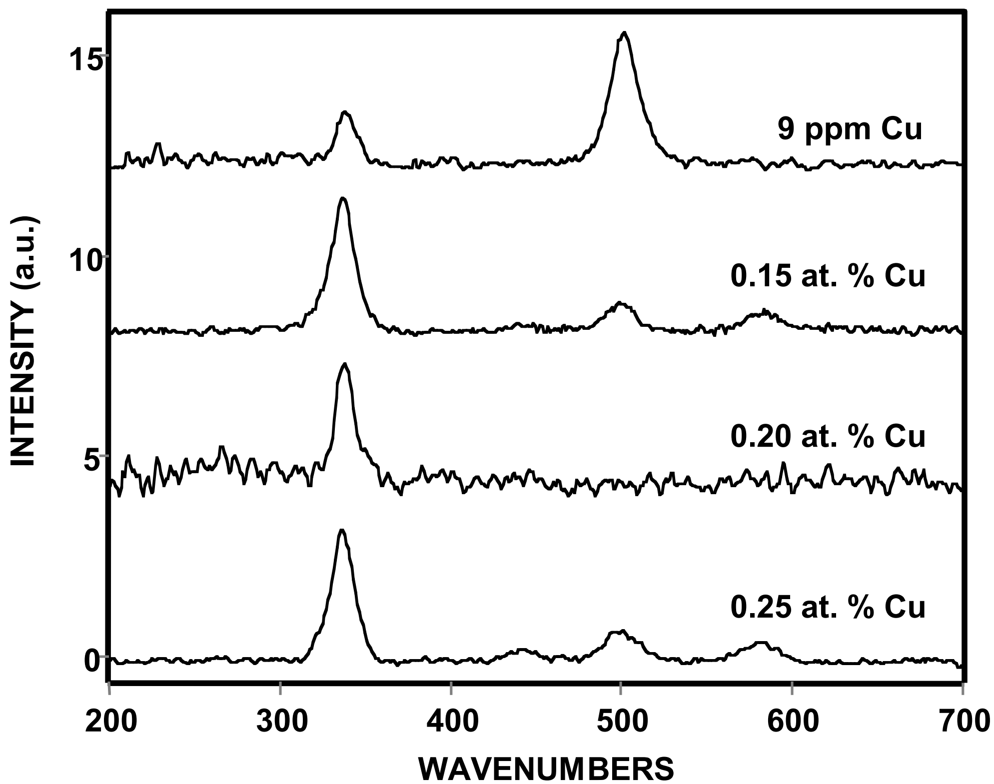


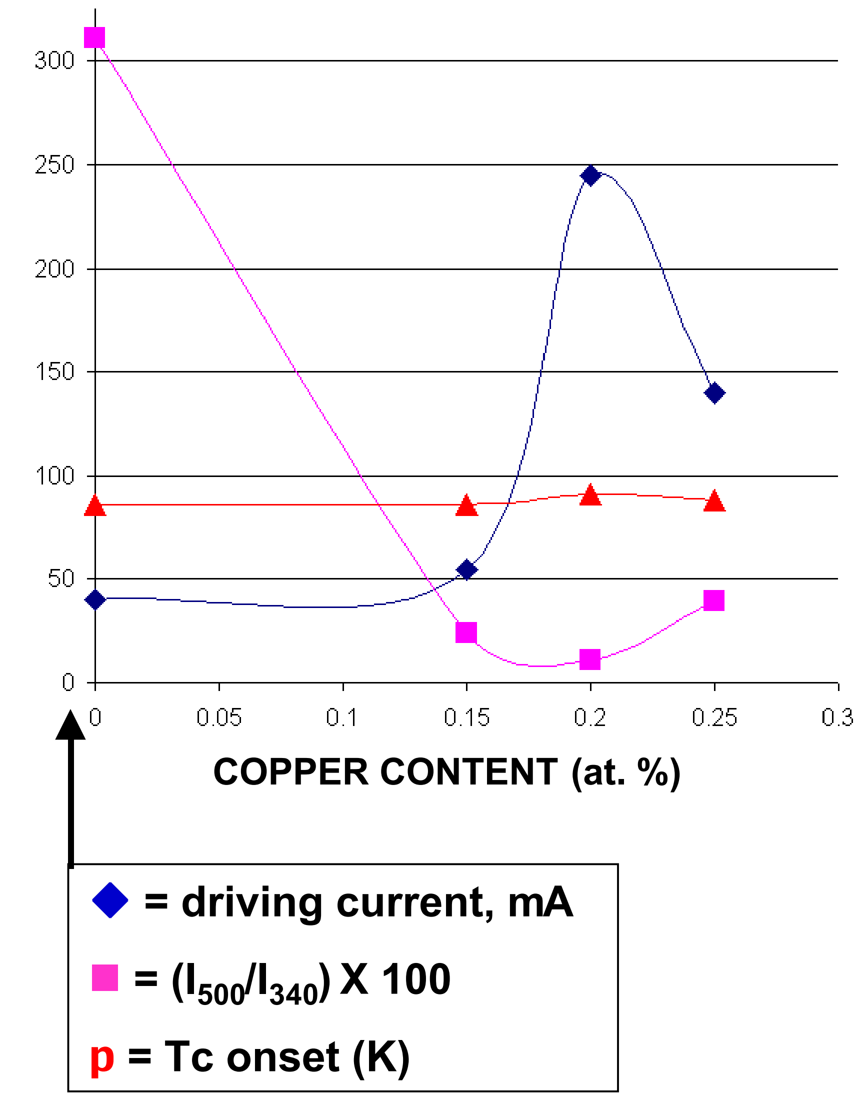


RABiTS / 0.8 MA/cm² (0.8 µm)

PLD Deposition of YBCO on Rolled Ag and Ag-Cu Substrates*

- note propensity for a axis grain growth of YBCO on pure Ag
- addition of Cu to Ag stabilizes c axis growth of YBCO
- optimum Cu content presumably connected to Cu activity at YBCO/Ag-Cu interface

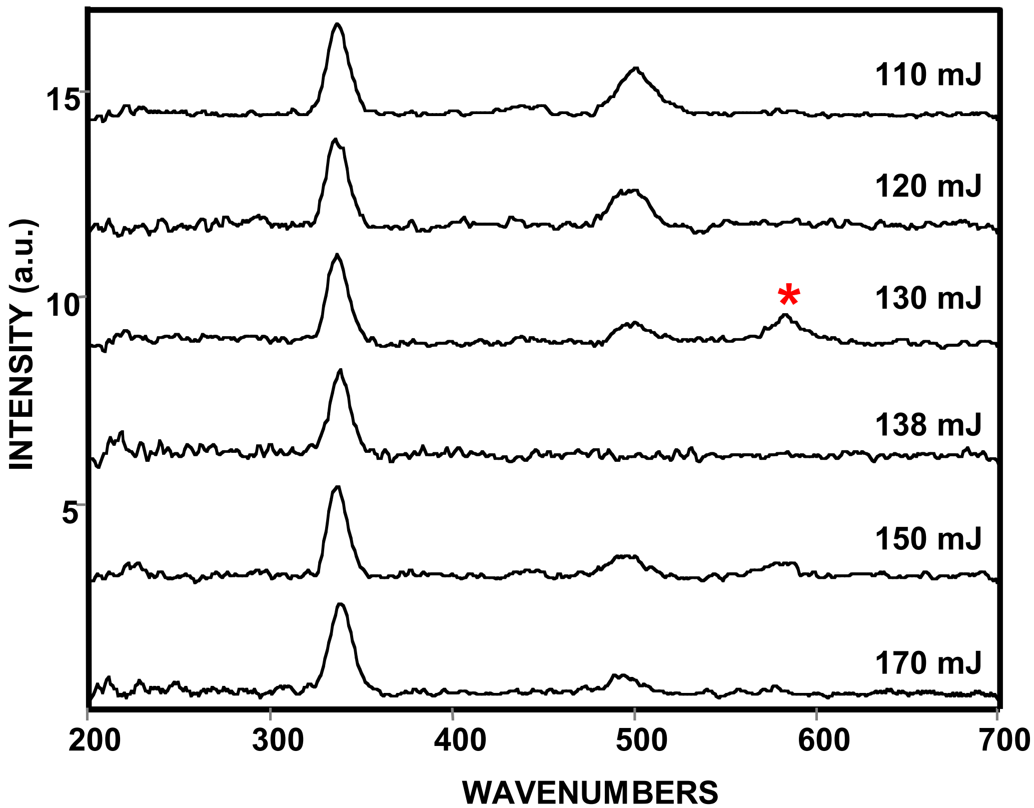


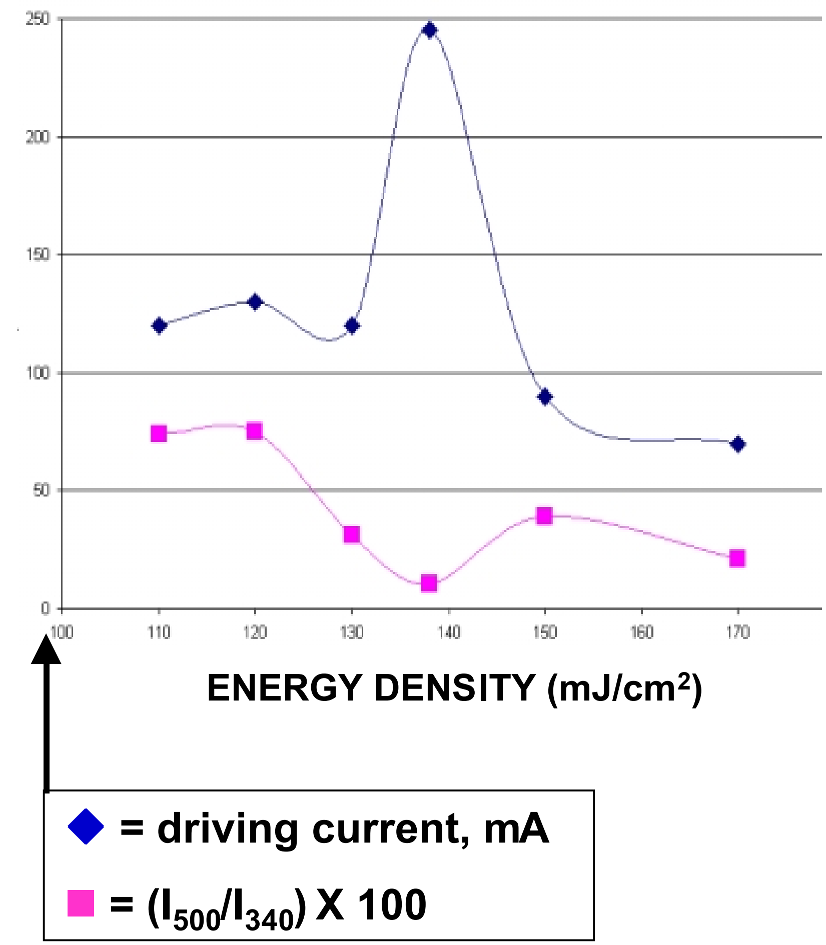


*Samples provided by R. Baurceanu, ANL

Optimization of PLD Deposition of YBCO on Ag-2 at.% Cu*

- maximum in inductive driving current corresponds to the minimum in a axis growth of YBCO grains
- this result is deposition system dependent

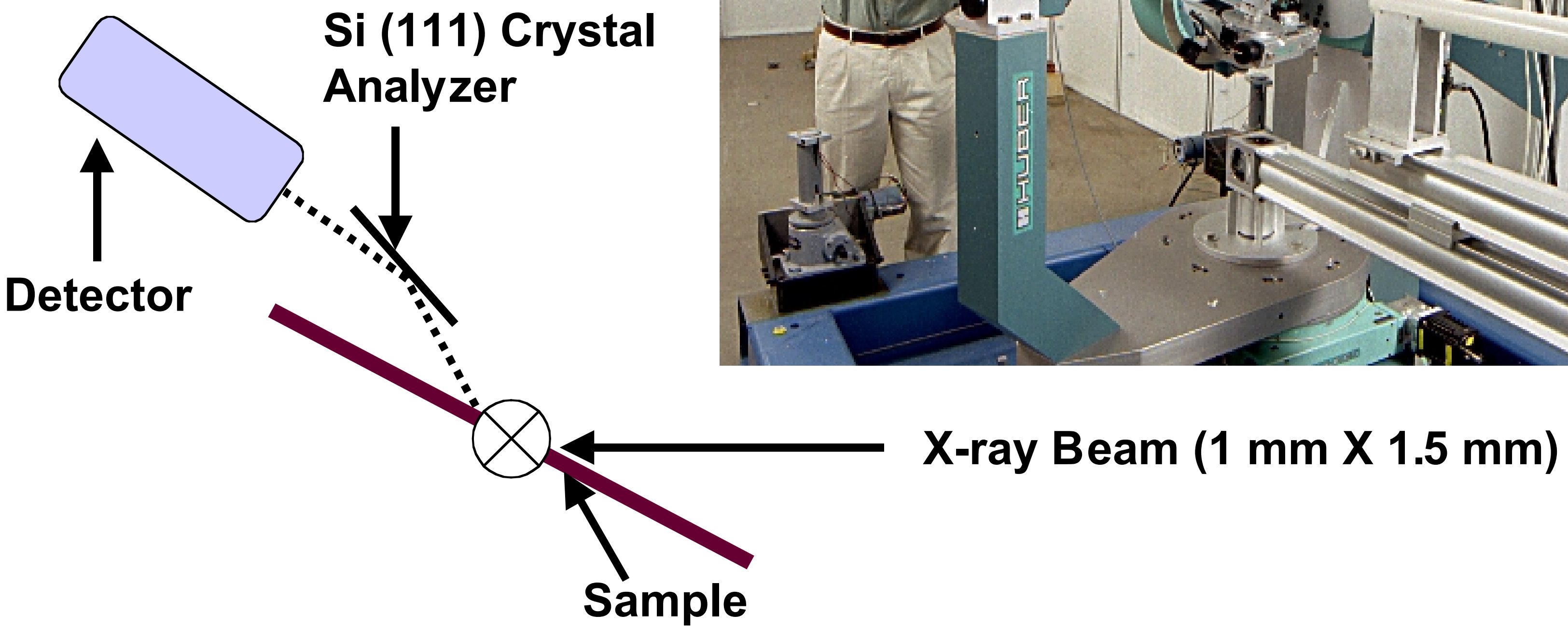




*Samples provided by R. Baurceanu, ANL

Diffraction Space Mapping (DSM) of Coated Conductors at the Advanced Photon Source (APS)

- map ω, δ (=2θ) space
- align using φ and χ
- examine asymmetric reflections using φ and χ



PLD Eu-123 Films on $SrTiO_3$ with and without Er-123 Buffer Layer [Samples provided by Quanxi Jia, LANL]

- Sample 1: Eu-123(160 nm)/$SrTiO_3$ (001) / [Tc <75K; Jc (75 K, sf) = 0 MA/cm^2]
- Sample 2: Eu-123(160 nm)/Er-123(10 nm)/$SrTiO_3$ (001) / [Tc >92 K; Jc (75 K, sf) = 2 MA/cm^2]

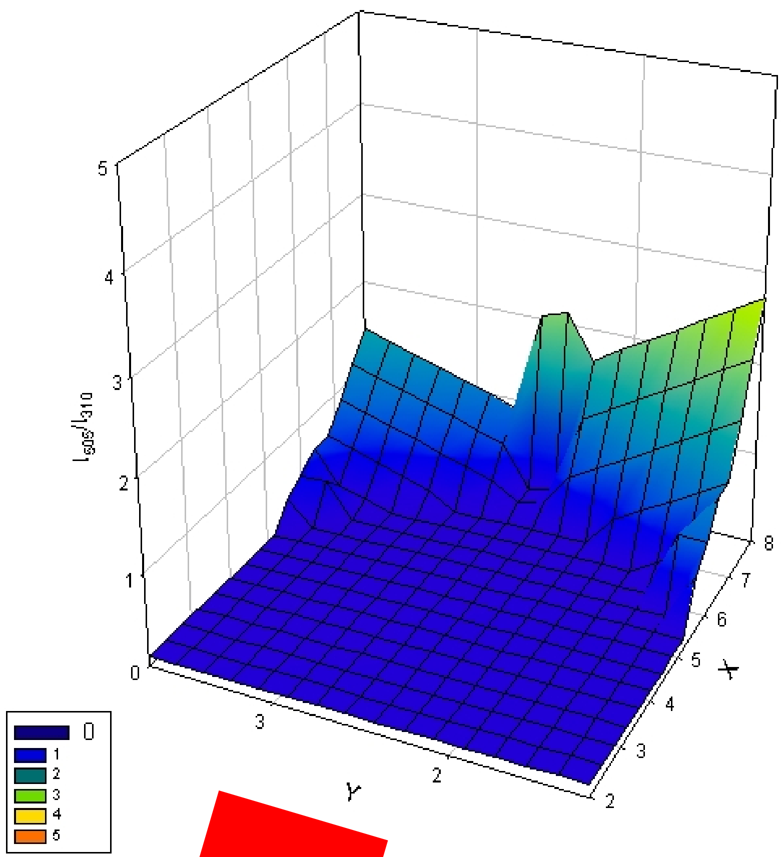
Material	a (Å)	b (Å)	c (Å)
Eu-123^O	3.84	3.90	11.71
Eu-123^T	3.88	3.88	11.81
Er-123^O	3.82	3.89	11.69
Er-123^T	3.85	3.85	11.78
$SrTiO_3$	3.90	3.90	3.90

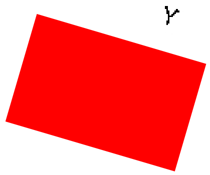
- Tetragonal $EuBa_2Cu_3O_6$ and $ErBa_2Cu_3O_6$ (Eu-123^T and Er-123^T) have a near perfect cube-on-cube lattice match with $SrTiO_3$ (001).
- If Eu-123 or Er-123 stretch in the a-b plane to match $SrTiO_3$ (001) *exactly*, then both should experience c axis compression and a concomitant increase in 2-θ (00L).
- The close match of the M-123 c axis parameter to 3-X the $SrTiO_3$ lattice parameter provides a driving force for a axis grain growth.

Texture Maps Produced by Raman Microscopy Examinations Provide a Reliable Metric for Selecting Suitable DSM Specimens

Eu-123 (160 nm)/$SrTiO_3$ (001)

Tc < 75K; Jc (75 K, sf) = 0 MA/cm²

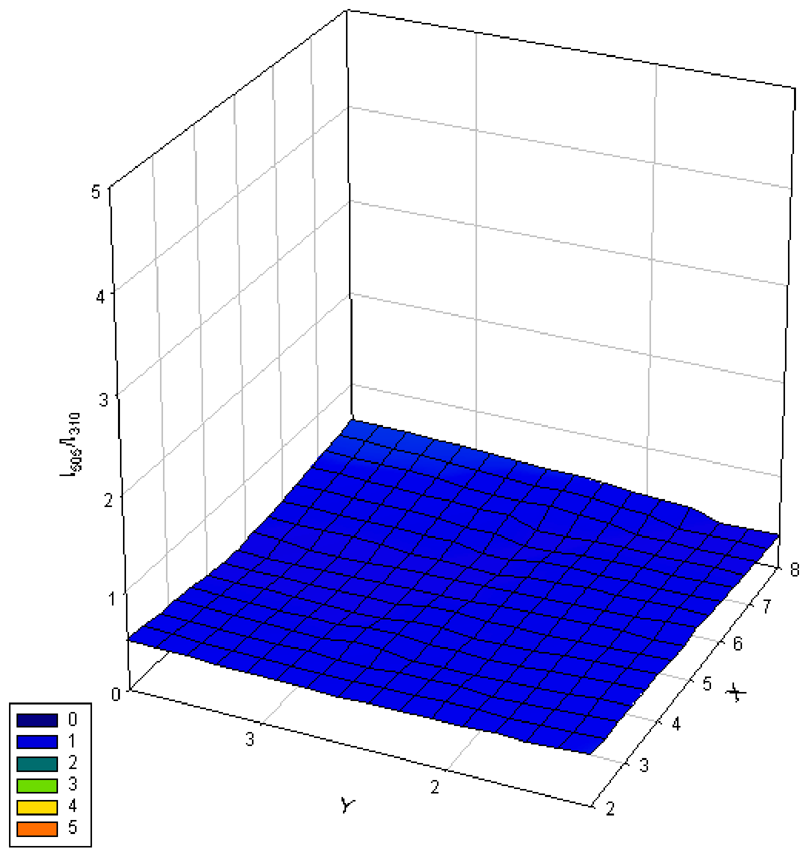




← **Footprint**

Eu-123 (160 nm)/Er-123 (10 nm)/$SrTiO_3$ (001)

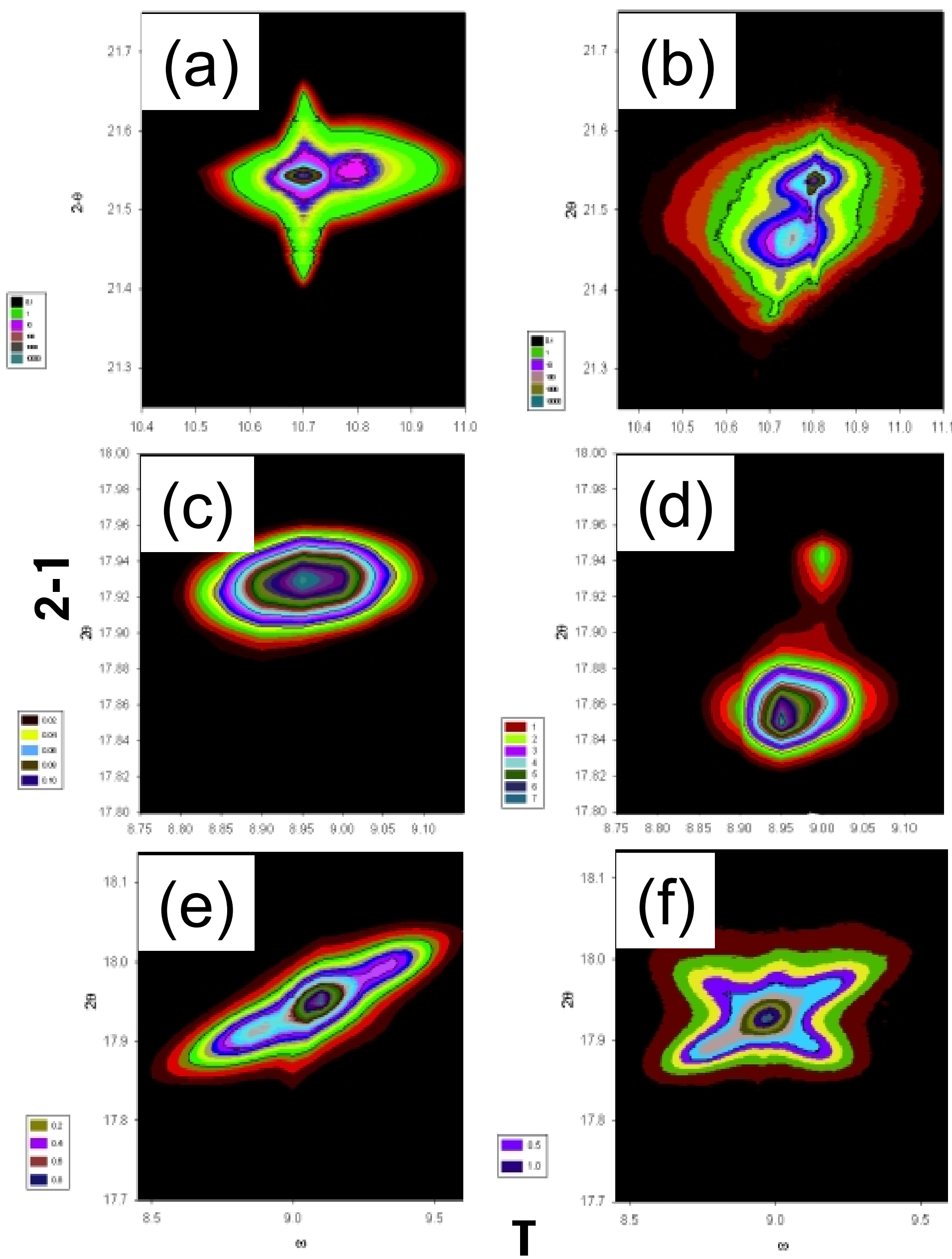
Tc >92 K; Jc (75 K, sf) = 2 MA/cm²

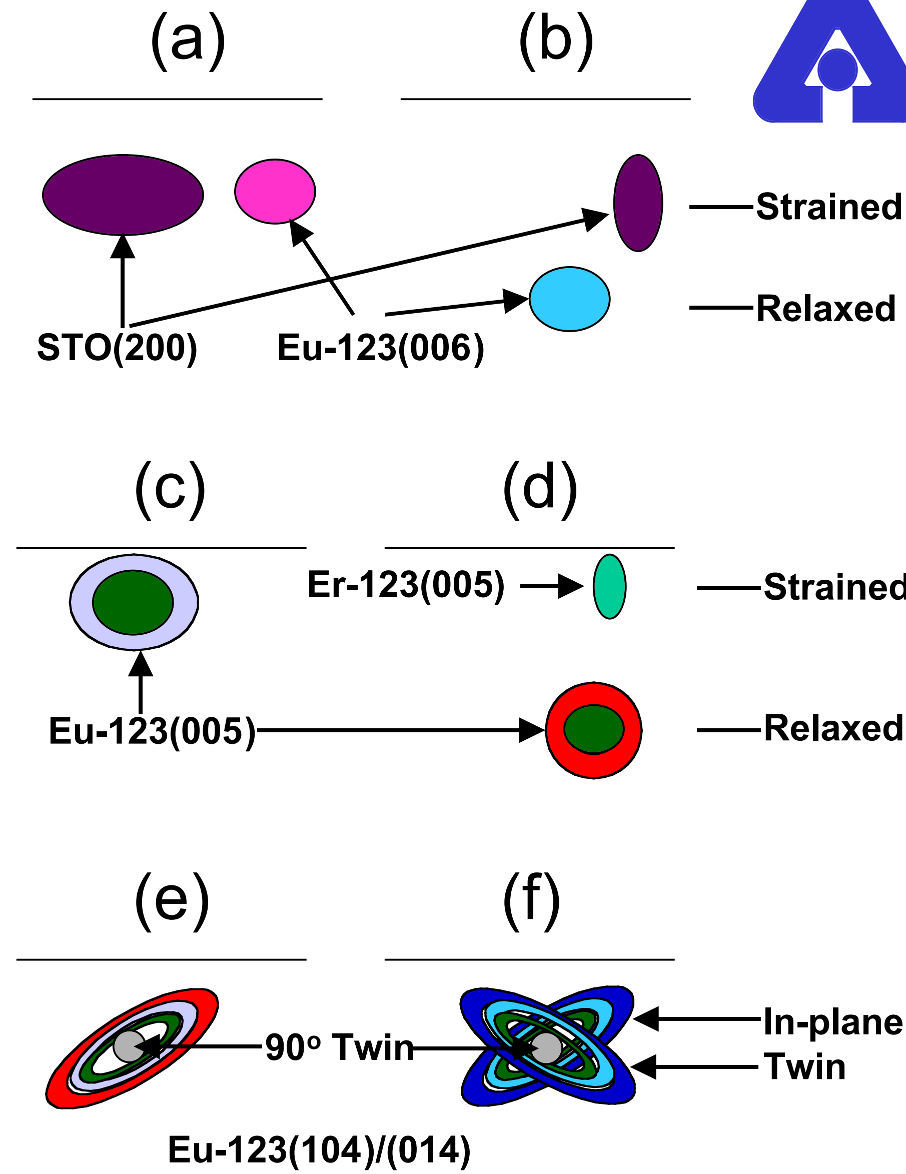


*** Samples from Q. Jia, LANL**

Eu-123 (160 nm)/STO

Eu-123/Er-123/STO



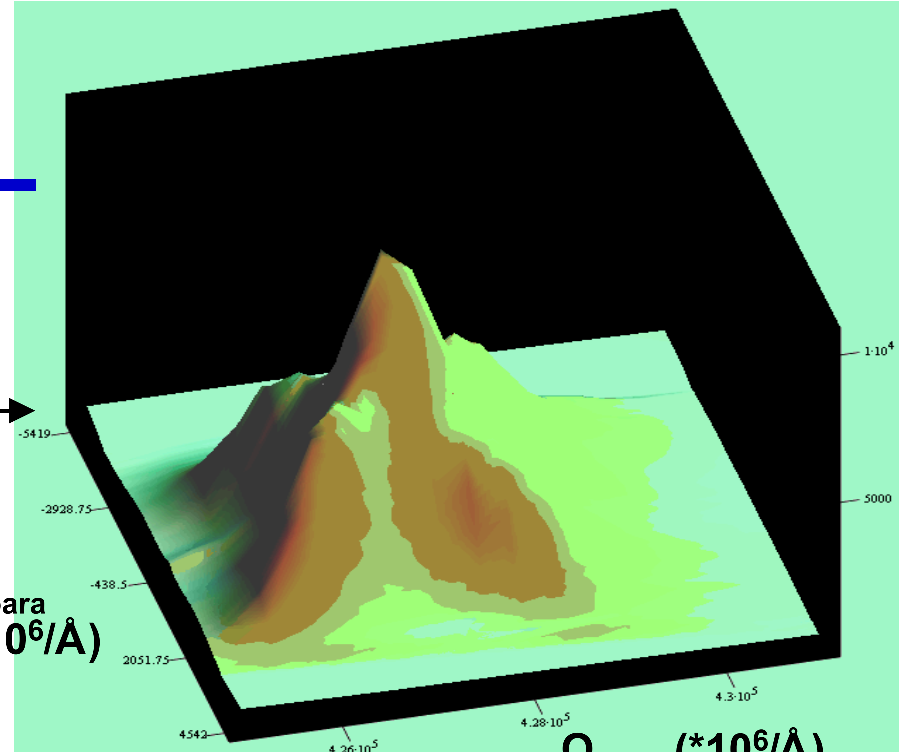


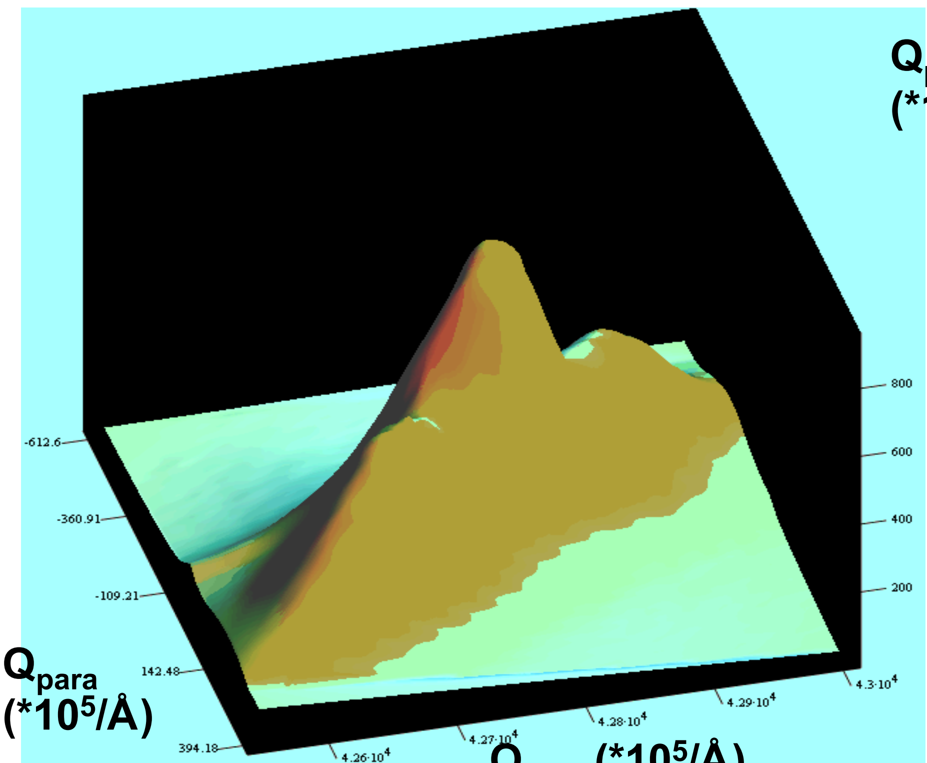
* Samples from Q. Jia, LANL

Reciprocal Space Maps for Eu-123 Films on $SrTiO_3(001)$* [(104)/(014)]

<u>Eu-123(160 nm)/Er-123(10 nm)/$SrTiO_3(001)$</u>

- Tc >92 K; Jc (75 K, sf) = 2 MA/cm^2
- In-plane and 90º twins →





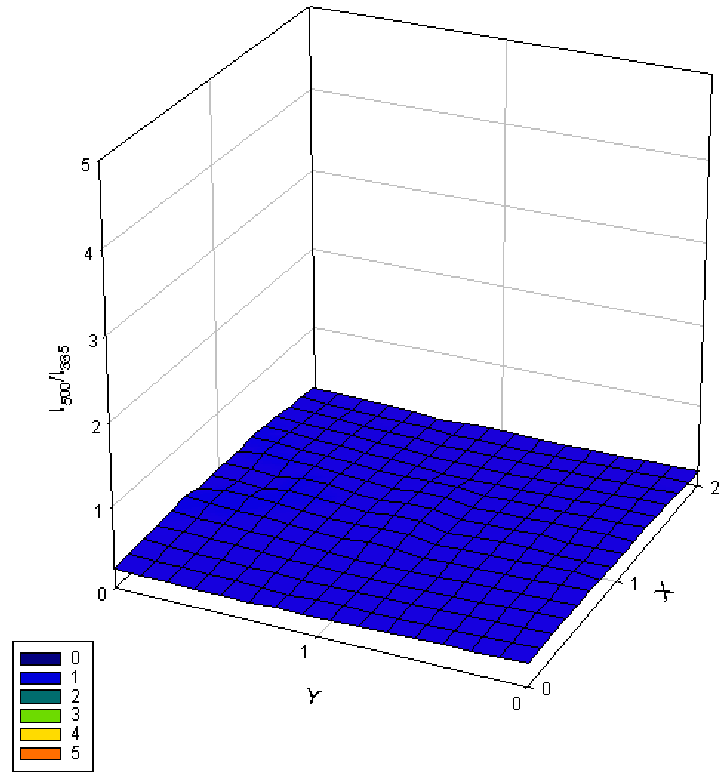
<u>Eu-123(160 nm)/$SrTiO_3(001)$</u>

- Tc < 75K; Jc (75 K, sf) = 0 MA/cm^2
- ← 90º twins only

* Samples from Q. Jia, LANL

DSM of PLD Y-123 Films on $SrTiO_3$(001) [Sample Provided by A. Goyal, ORNL]

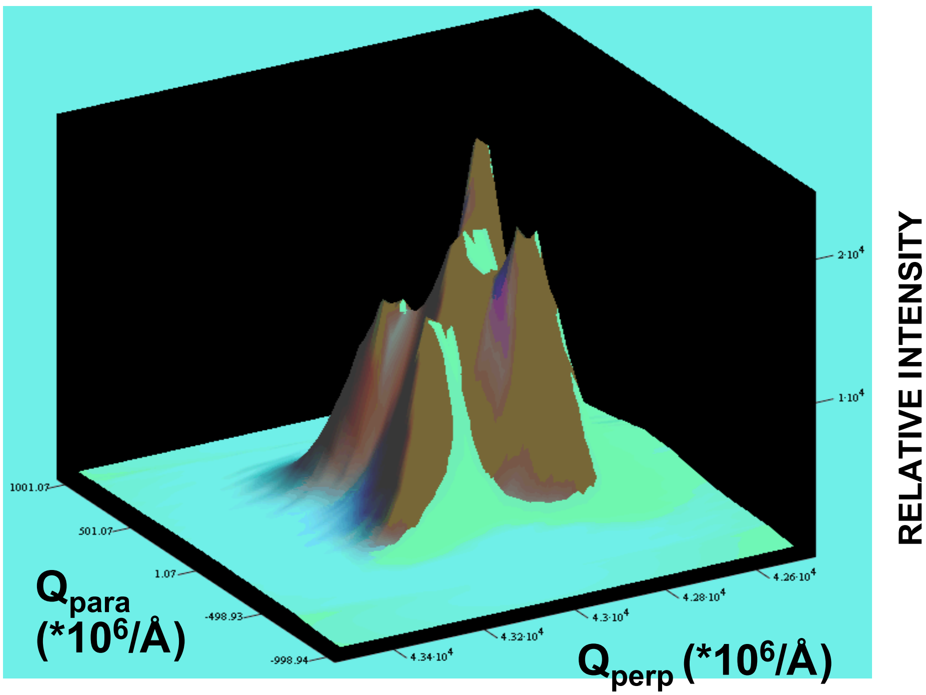
Raman Texture Map



YBCO (PLD)/$SrTiO_3$(001)

YBCO = 0.19 µm

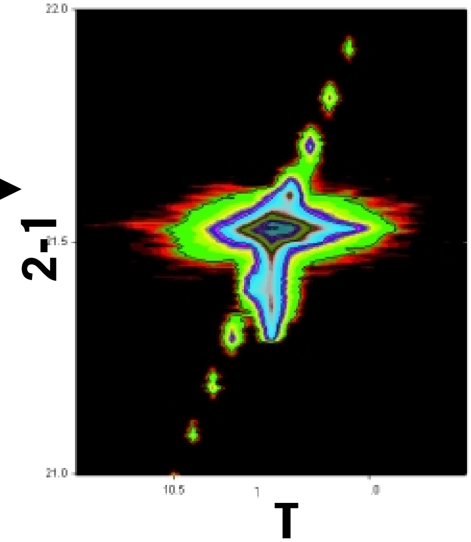
Jc = 5.3 MA/cm^2



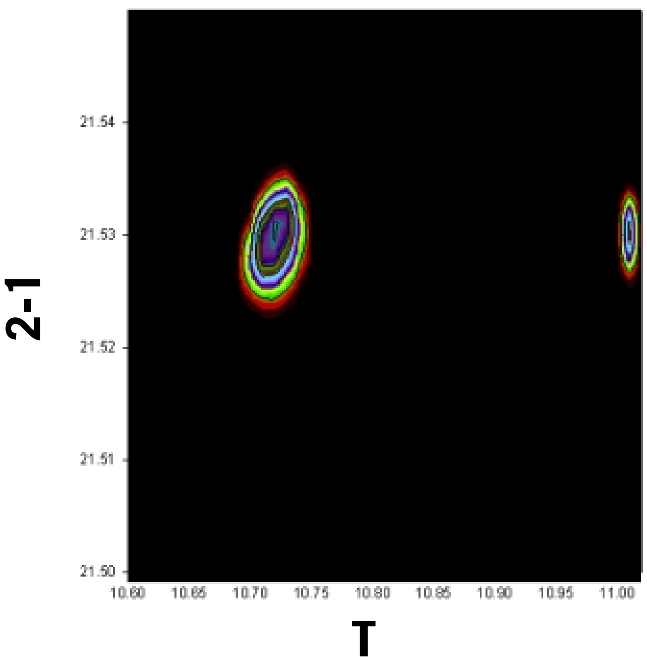
- **Reciprocal space map of the YBCO (104)/(014) reflection shows in-plane and 90° twins**

DSM of $SrTiO_3$(001) Substrates Shows Evidence of Complex Substructures

• broad scan of (200) shows evidence of surface films



• fine scan reveals presence of a twin



• reciprocal space map highlights multiplicity of structures