FABRICATION OF YBCO-COATED CONDUCTORS BY INCLINED SUBSTRATE DEPOSITION TECHNIQUE*

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GOALS

* Conduct research to establish the processing science and technology for making practical coated conductors.
* Establish and maintain relationships with partners (industries, universities, and national labs.) to facilitate rapid commercialization of HTS technology.

APPROACH

* Address issues related to the important steps of the ISD process – growth of biaxially textured MgO on metallic substrate, deposition of buffer and superconductor layers.

ANL Coated Conductor Team

Energy Technology Division: Balu, Baurceanu**, Cheon*, Dickerson*, Dorris, Erck, Fisher, Koritala, Li**, Ma, Singh, Weber*, Williams*

Chemical Technology Division: Maroni, Kropf, Venkataraman*

Materials Science Division: Berghuis, Chen**, Claus, Gray, Miller, Paulikas, Tang**, Veal, Vlasko-Vlasov, Welp, You

Funding leverage: DOE-SC; AF/OSR; industry cost-share

* Graduate students (5)
** Post-docs (4)

Collaborators

Industry

- American Superconductor
  - Malozemoff, Rupich, Riley, Annavarapu
- IGC-SuperPower
  - Selva, Reeves, Lee, Li
- Universal Energy Systems, Inc. (UES)
  - Bhattacharya

National Labs.

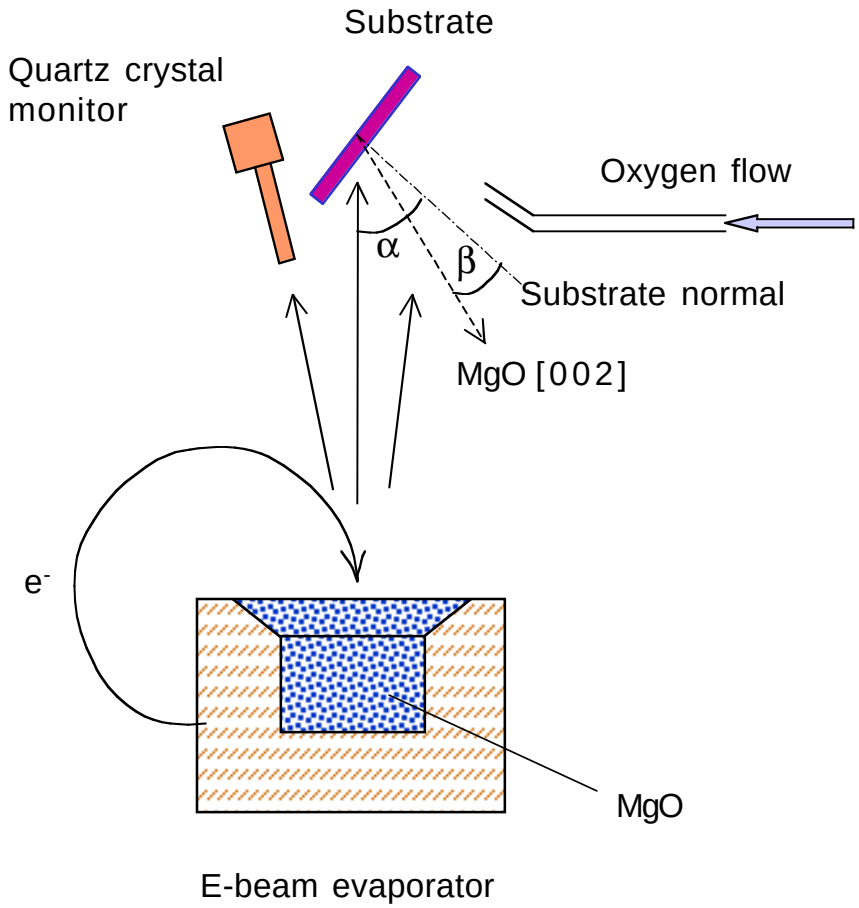
- LANL
  - Arendt, Foltyn
- ORNL
  - Feenstra, Christen, Parans, Goyal

Collaborators (cont.)

Universities

- University of Illinois
  - McNallan, Indacochea
- Ohio State University
  - Snyder
- Illinois Inst. Technology
  - Segre, Nash
- Augsburg University
  - Mannhart
- Cali University
  - Prieto
- SUNY/Albany
  - Efstathiadis, Haldar
- University of Wisconsin
  - Larbalestier, Gurevich

Inclined Substrate Deposition of MgO

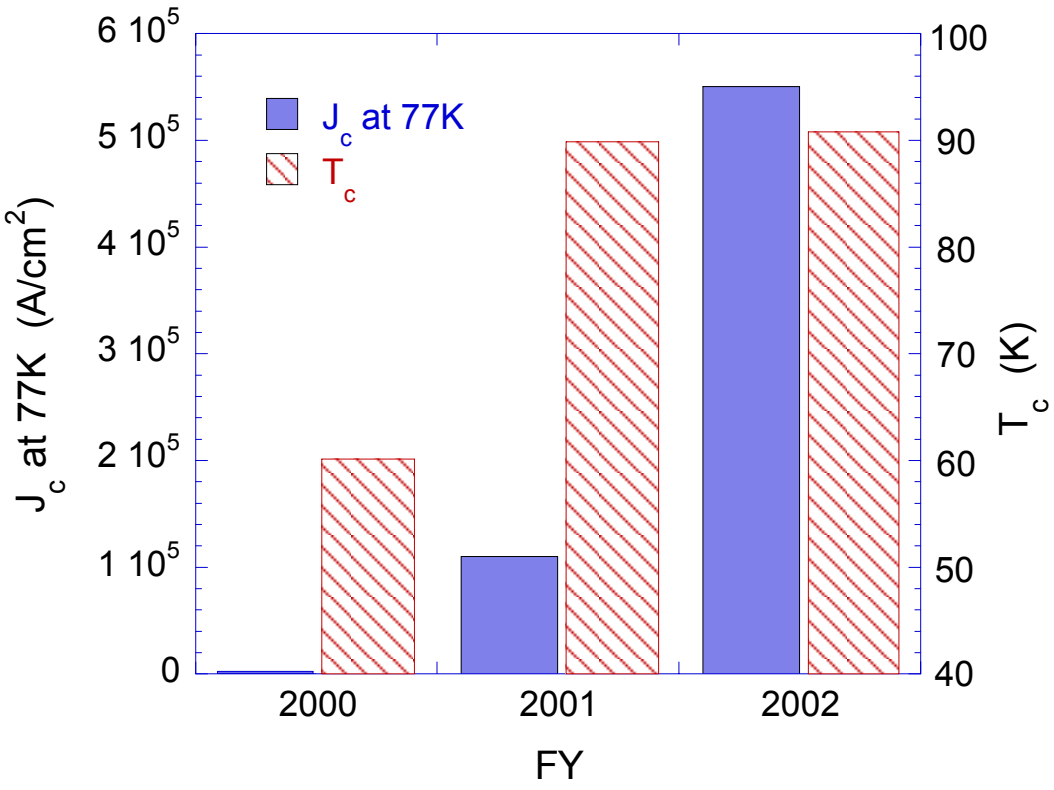


α: inclination angle

β: tilt angle

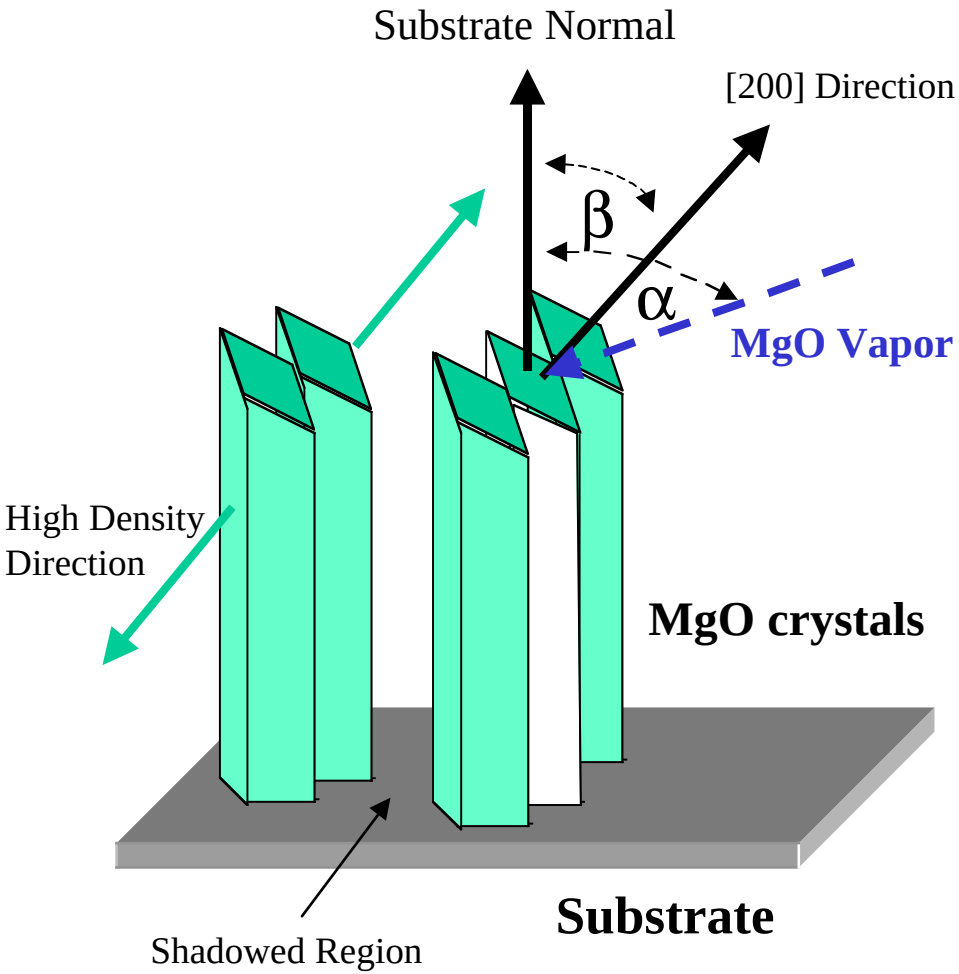
- Simple and fast
- Room temperature process
- Independent of substrate
- Amenable to scale-up
- Produces highly textured MgO films

Progress in the Development of Coated Conductors by ISD



FY	T_c (K)	J_c@77K (A/cm^2)
2000	60	0
2001	90	1.1 x 10^5
2002	91	5.5 x 10^5

Texture Development by ISD



- **Growth anisotropy**

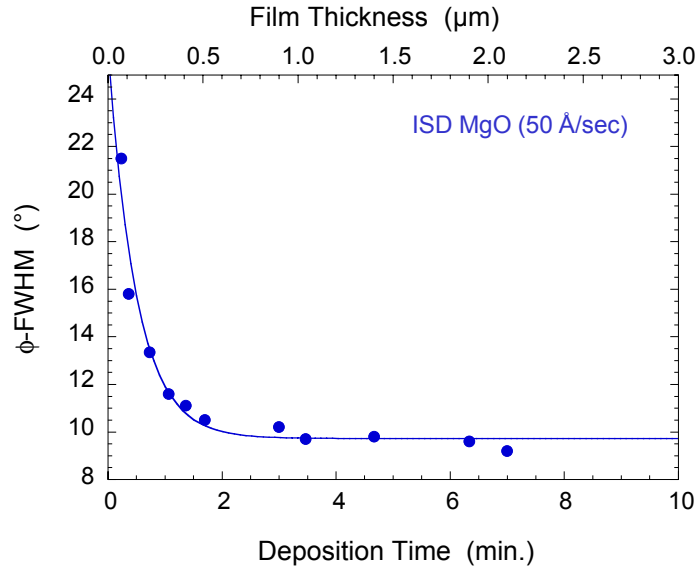
MgO grows in columns truncated with (200) faces.

- **Self Shadowing**

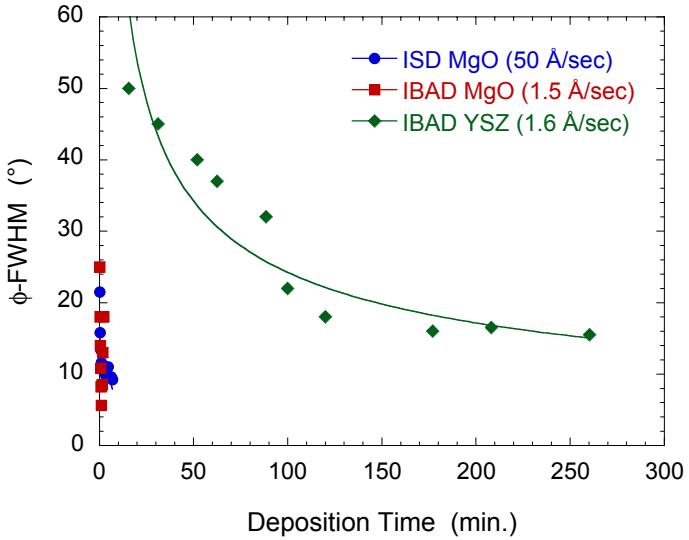
Grains that have the greatest "capture cross-section" aligned with the "fast" growth direction outgrow grains that do not have this alignment.

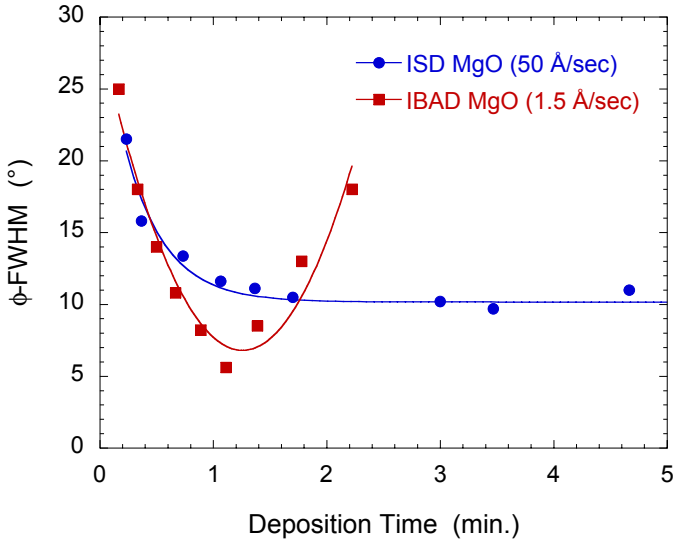
β = Tilt angle
(degree to which [200] is inclined towards vapor flux)

Deposition of Biaxially Textured Films



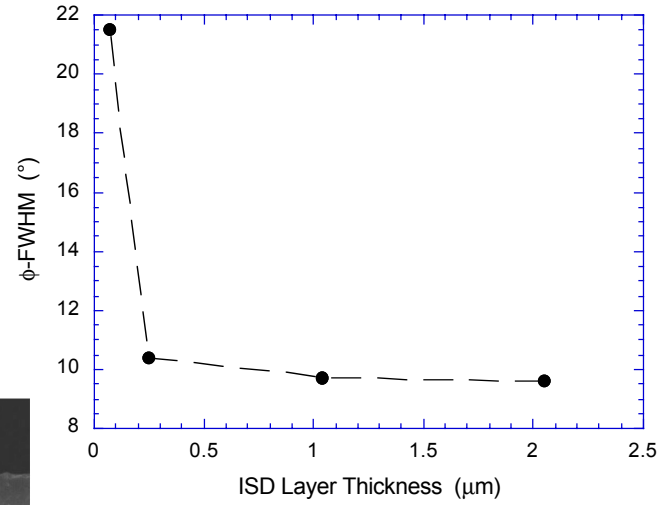
- Texture develops in the first 0.5 μm thick film
- Independent of deposition rate (up to 100 Å/sec)
- Good tolerance to substrate surface roughness

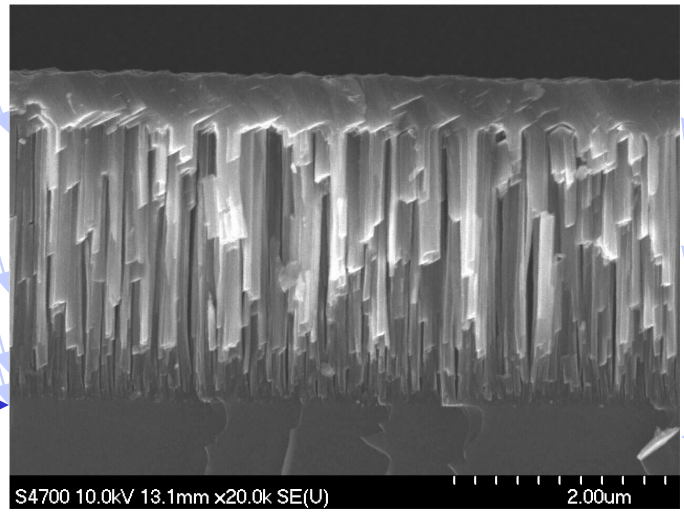




Thickness Dependence of ISD MgO Texture

ISD MgO Layer Thickness (µm)	Homo-epi MgO Thickness (µm)	MgO (200) ϕ-scan FWHM (°)
2.05	0.48	9.6
1.04	0.48	9.7
0.25	0.48	10.4
0.07	0.48	21.5





Homo-epi MgO Layer

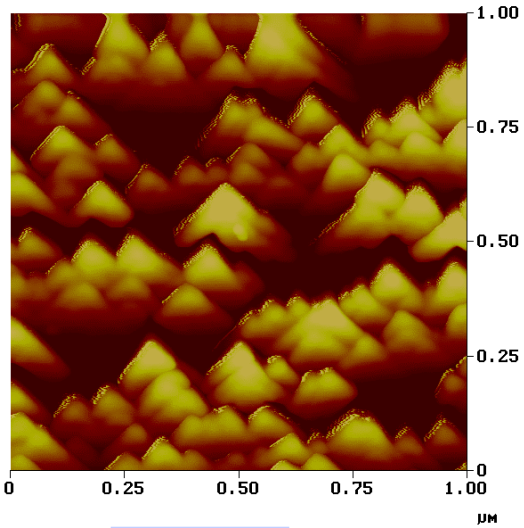
ISD MgO Layer

Substrate

Film/Substrate interface

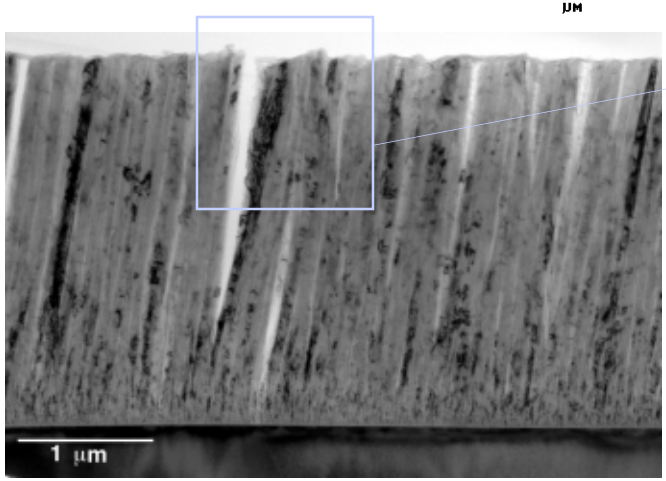
MgO Template Film Grown by ISD

AFM

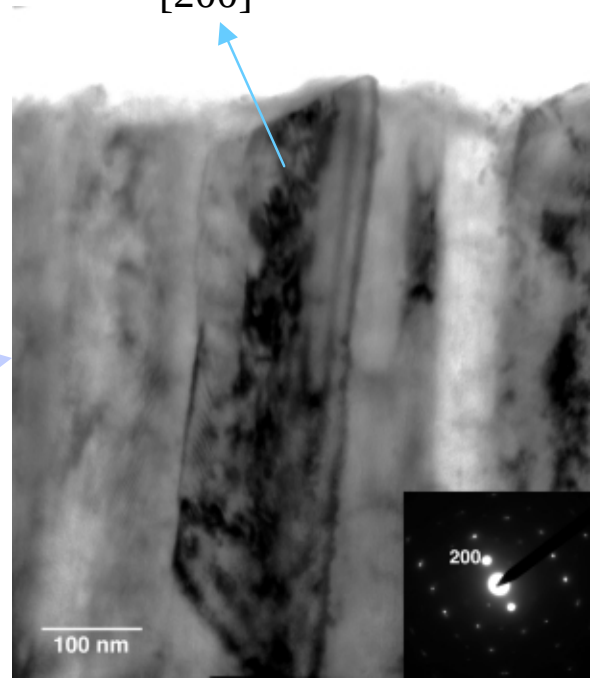


RMS roughness = 28.9 nm

TEM



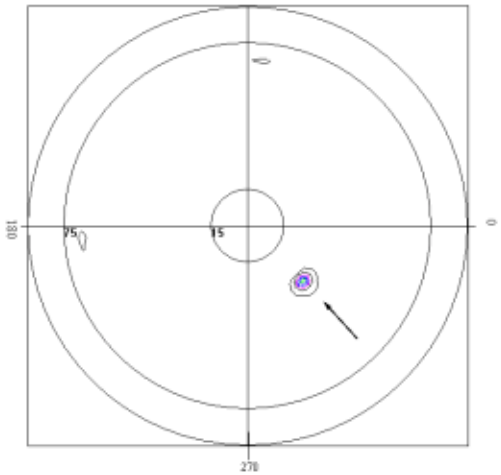
[200]



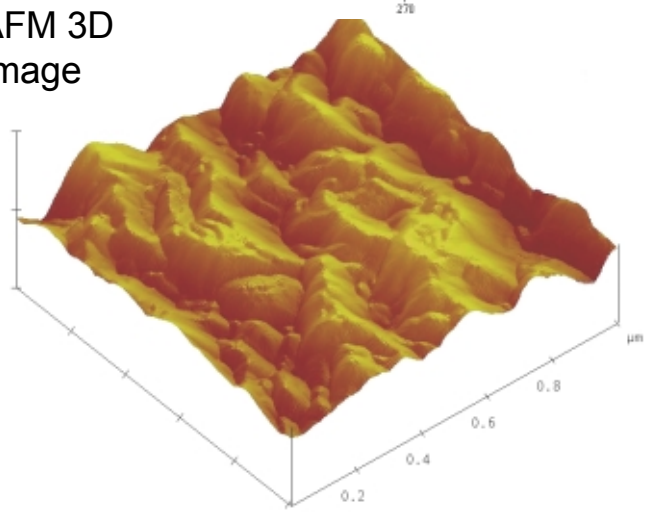
Column face is a (200) plane.

ISD MgO with Homo-epi MgO Layer

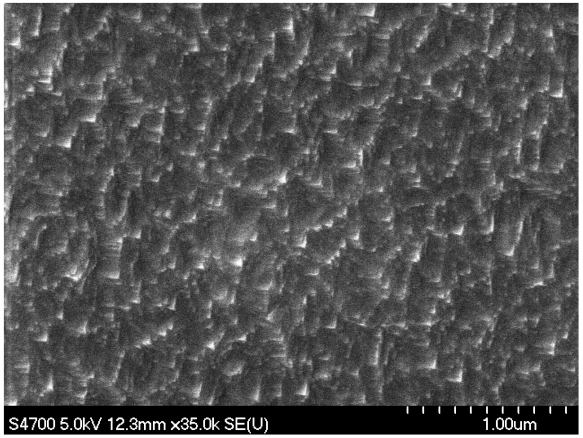
MgO (002)
Pole figure



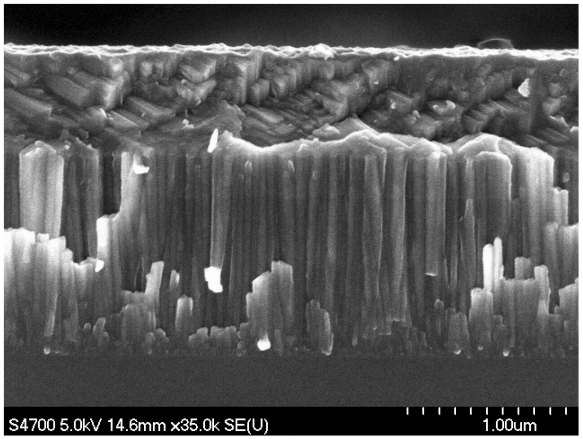
AFM 3D
Image



RMS roughness = 8.3 nm

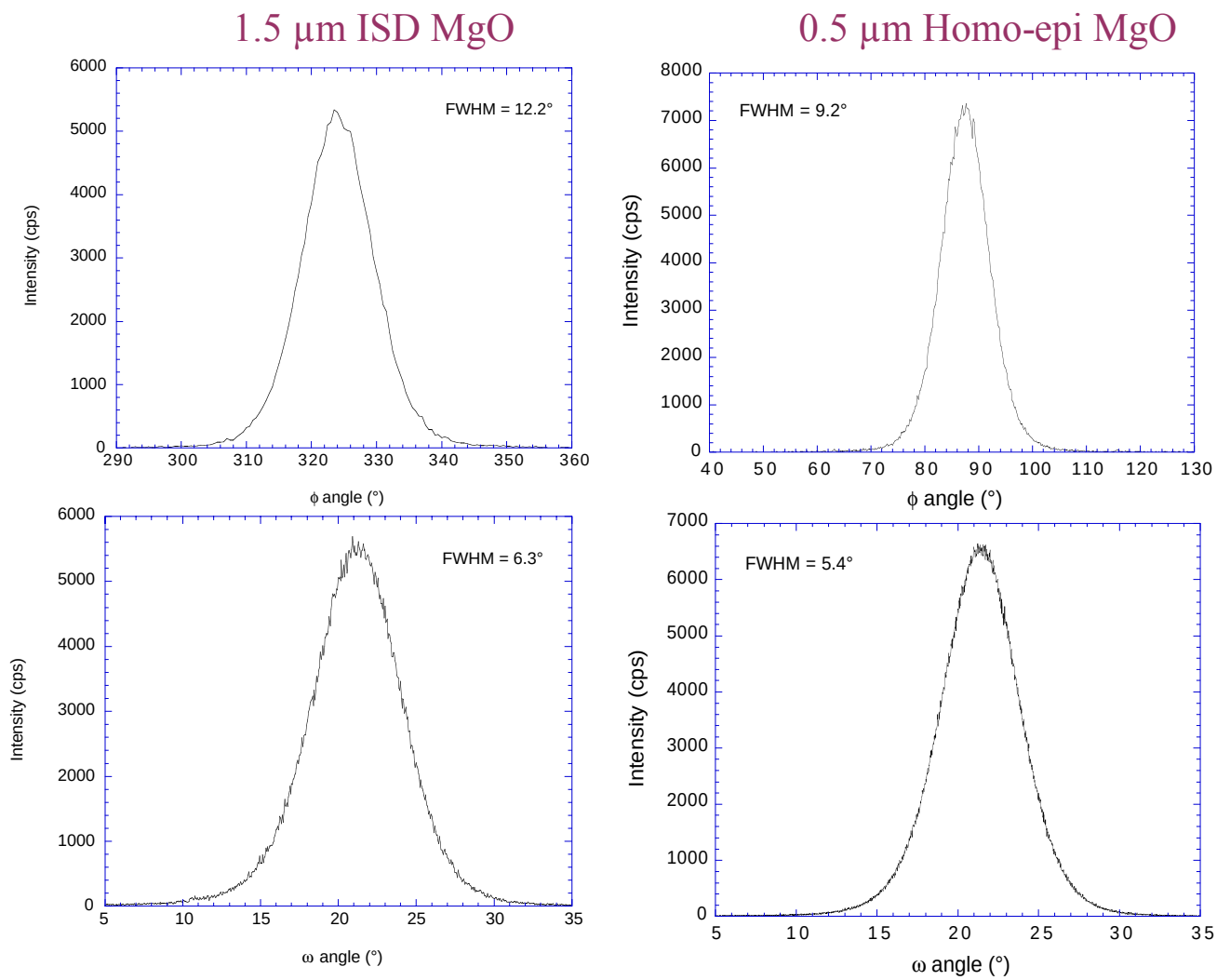


Plan-View
SEM

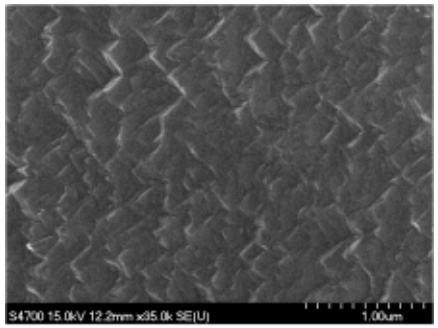


Cross-sectional
SEM

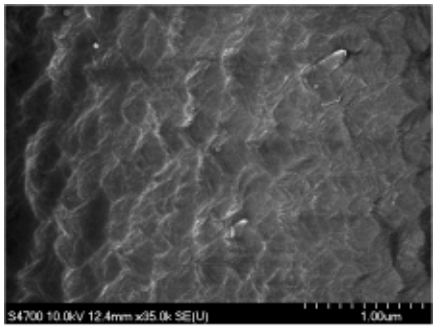
Texture Alignment in MgO Films



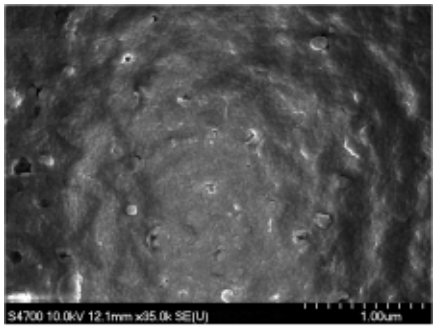
Surface Morphology of Various Layers in ISD MgO Architecture



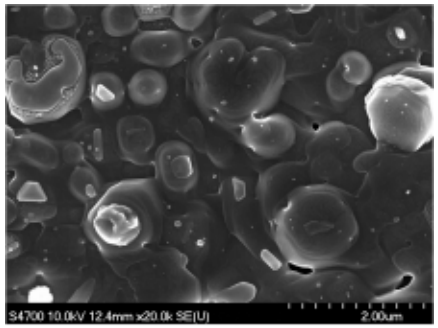
Home-epi MgO surface
S.R. ≈9 nm



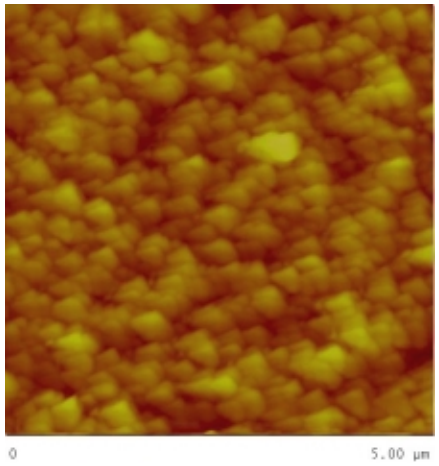
YSZ surface
S.R. ≈8 nm

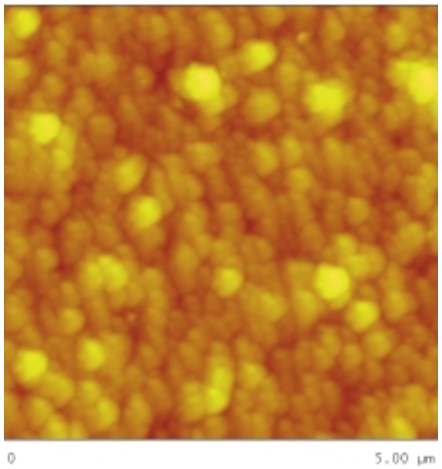


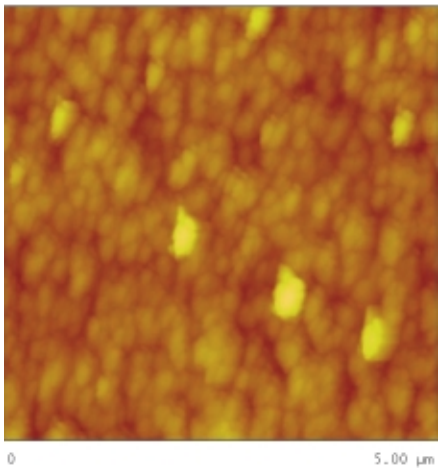
CeO_2 surface
S.R. ≈8 nm

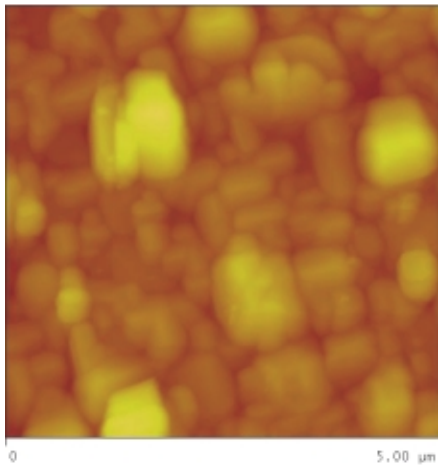


YBCO surface
S.R. ≈25 nm

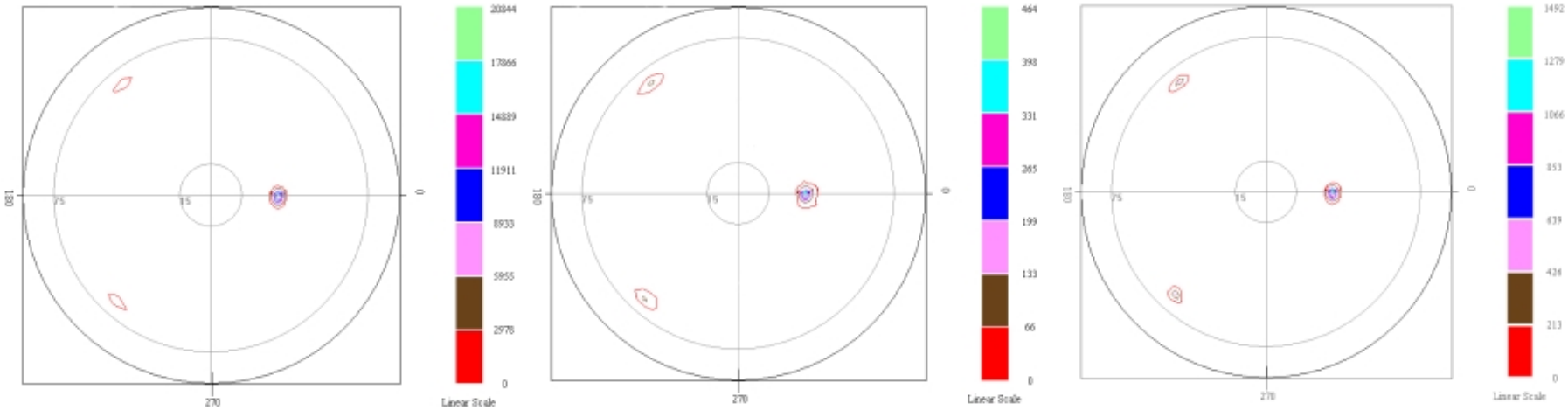








CeO_2/YSZ Buffer Layers on ISD MgO

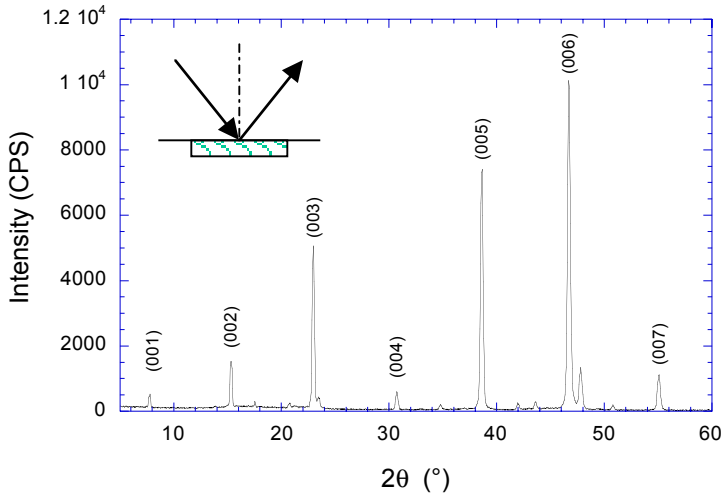


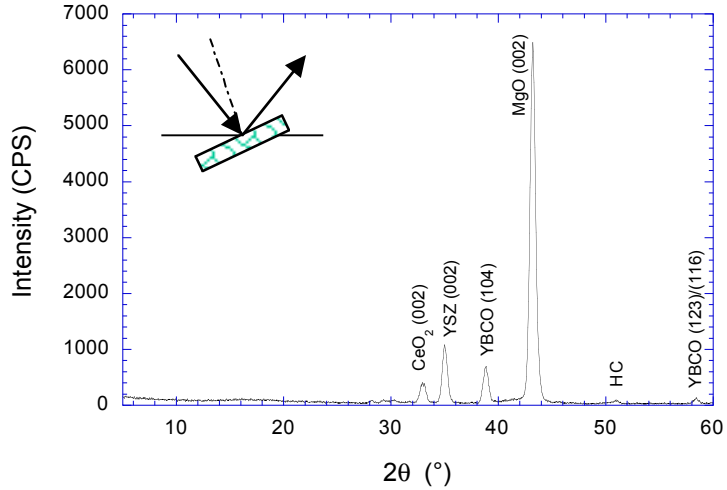
MgO (002) YSZ (002) CeO_2 (002)

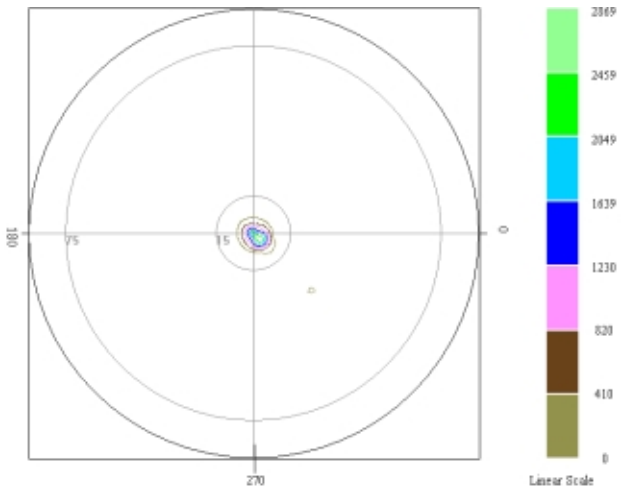
"Cube-on-cube" epitaxy relationship:

CeO_2[001] // YSZ[001] // MgO[001] and
CeO_2[110] // YSZ[110] // MgO[110]

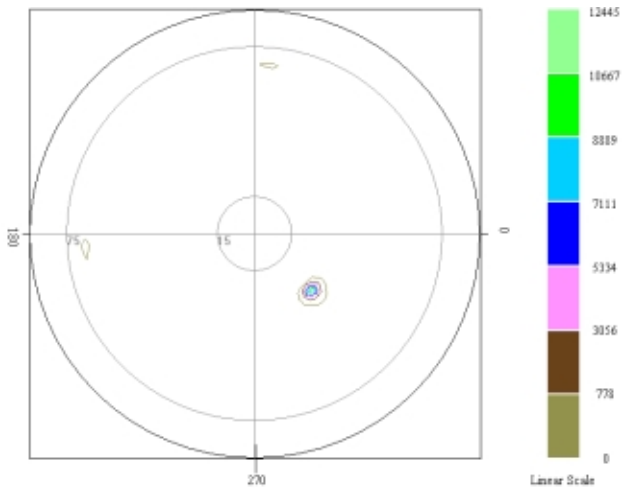
YBCO Grown on CeO_2/YSZ-Buffered ISD MgO Substrate





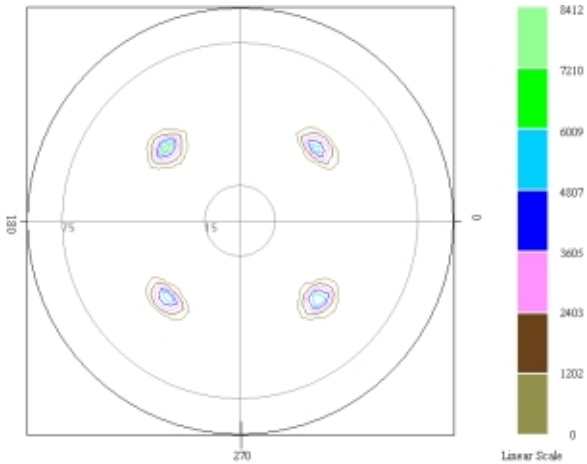


YBCO (005)

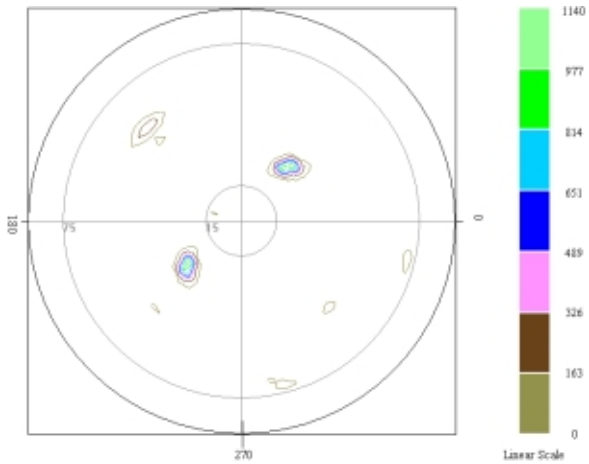


MgO (002)

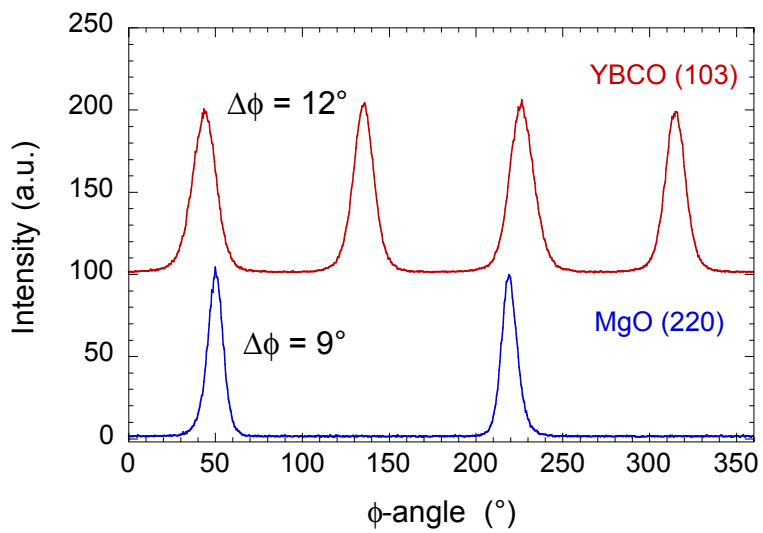
YBCO Grown on CeO_2/YSZ-Buffered ISD MgO Substrate



YBCO (103)



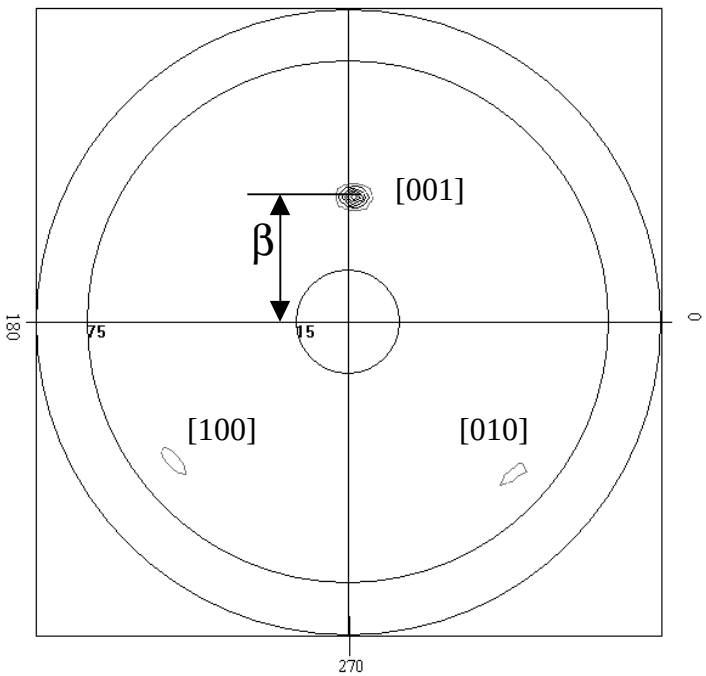
MgO (220)



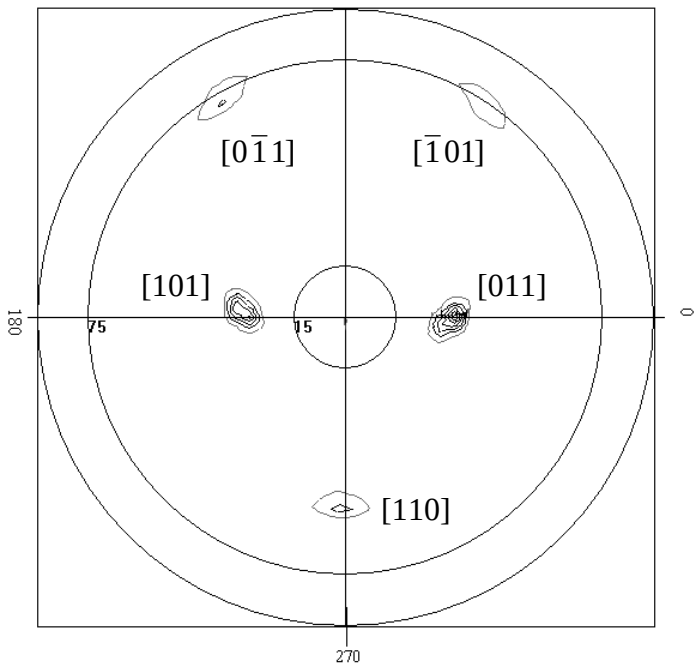
Orientation relationship:

YBCO[001] // MgO[112] and
YBCO[100] // MgO[111] or
YBCO[010] // MgO[110]

XRD Pole Figures of ISD MgO

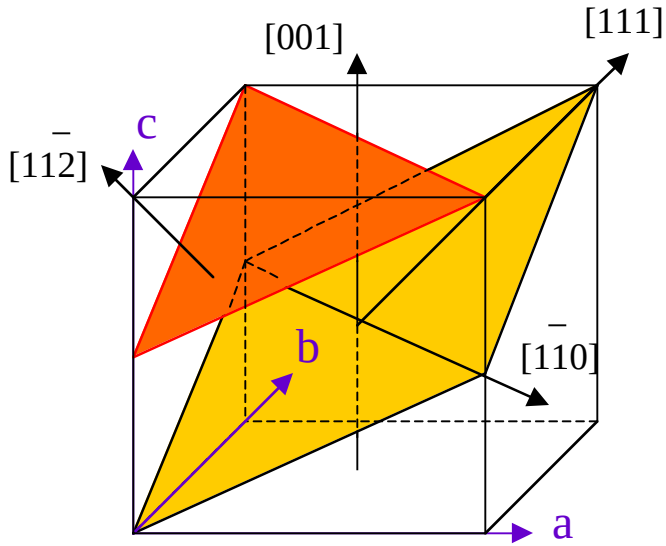


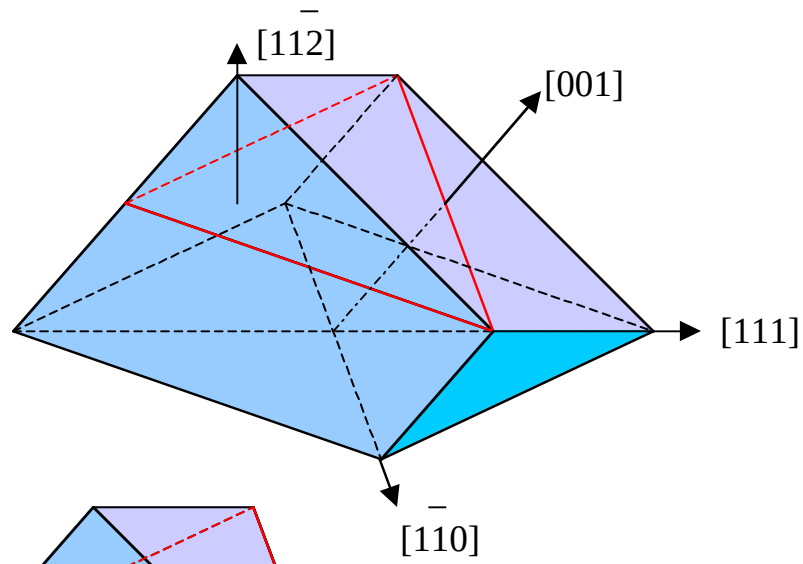
MgO (002)

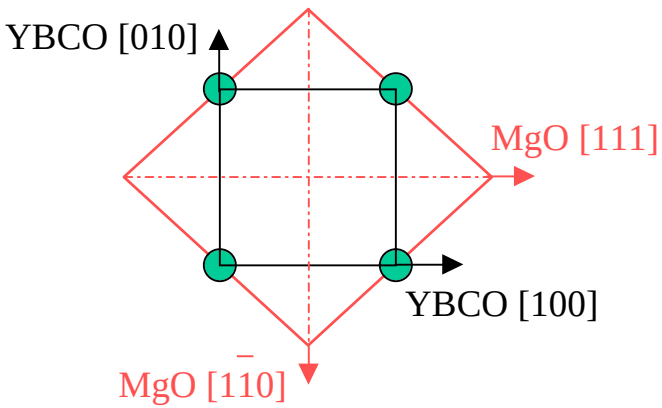


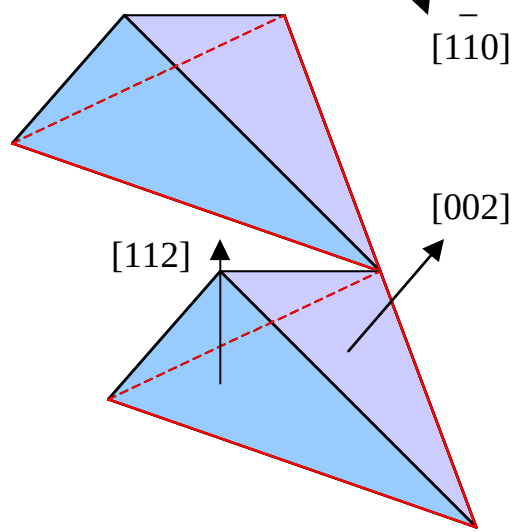
MgO (220)

Crystallographic Orientation of ISD MgO





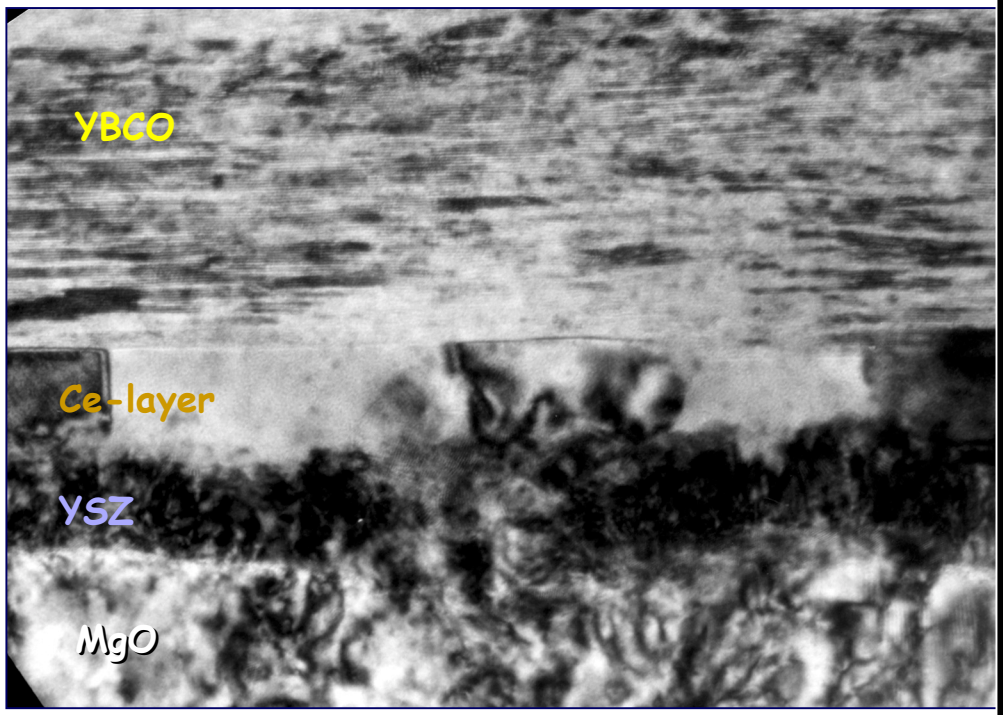




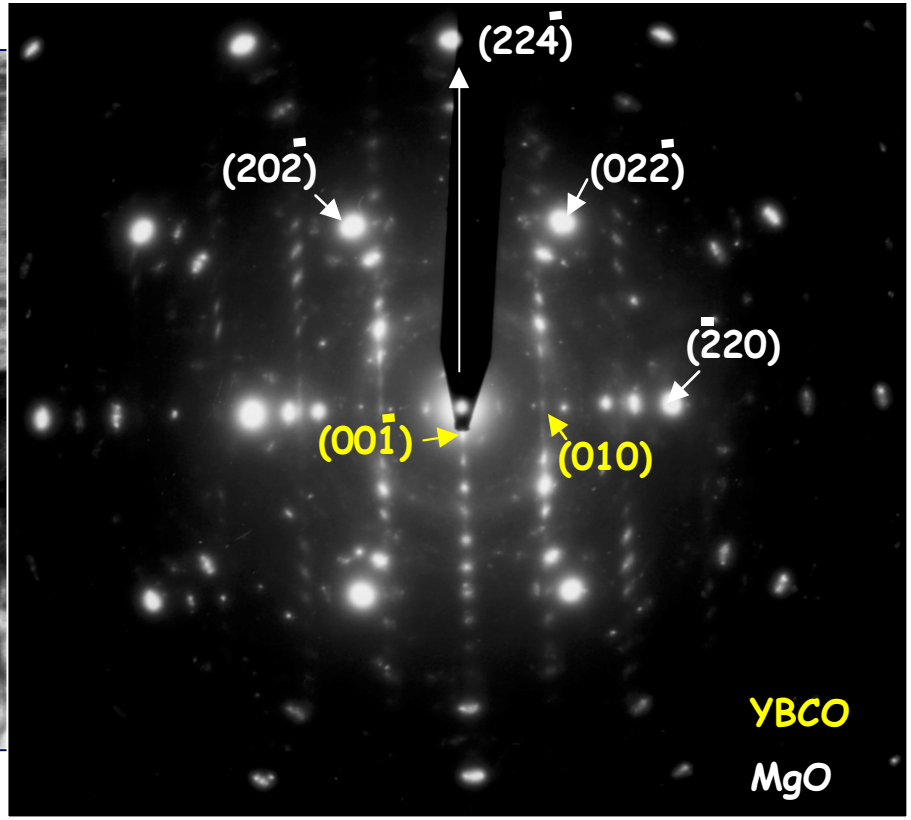
In-Plane Textured YBCO on ISD MgO

Selected Area Diffraction reveals orientation relationship:

$[111]_{MgO}$ // $[100]_{YBCO}$; $[220]_{MgO}$ // $[010]_{YBCO}$

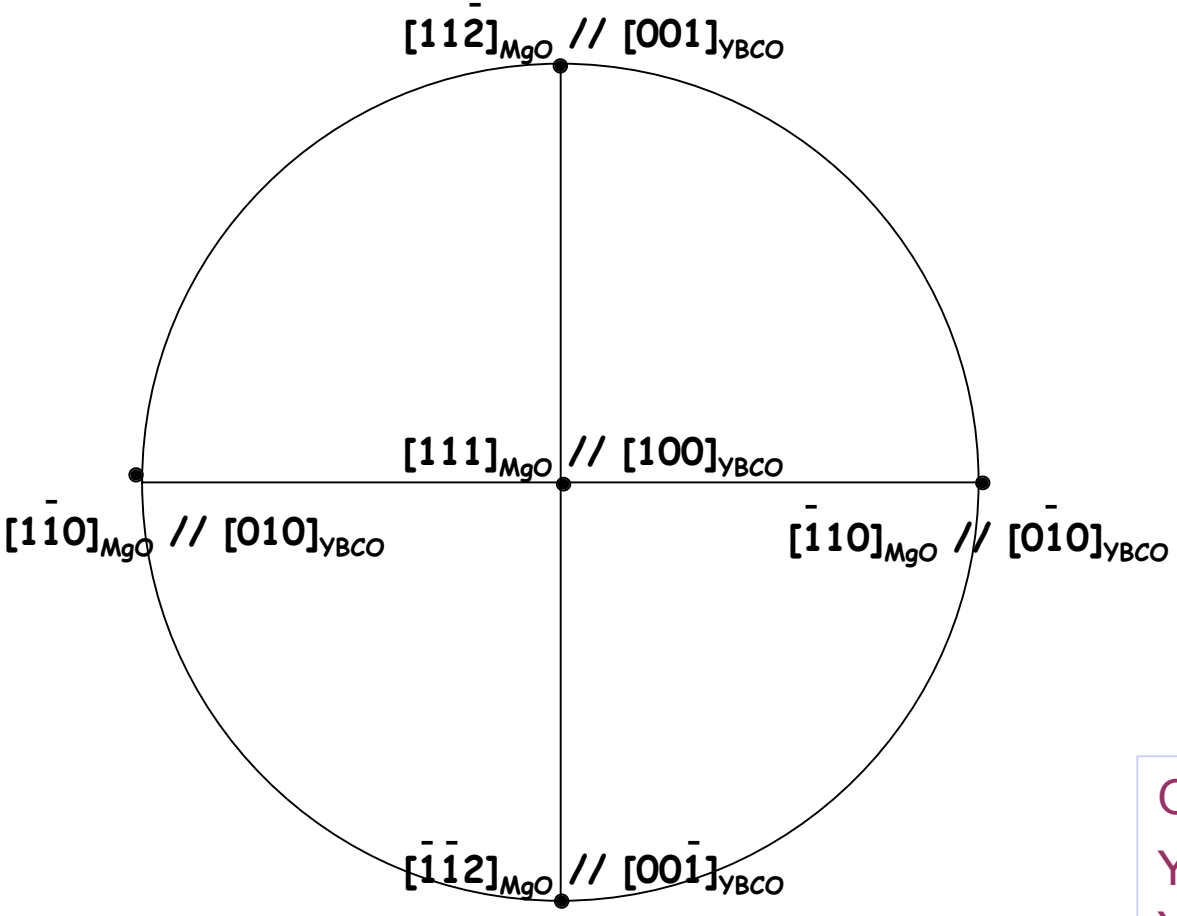


50 nm

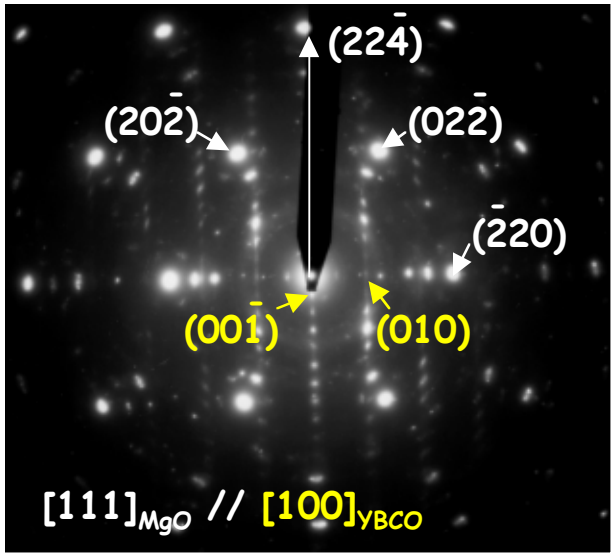


zone axes: $[111]_{MgO}$ // $[111]_{YSZ}$ // $[100]_{YBCO}$

Orientation Relationship: YBCO & MgO



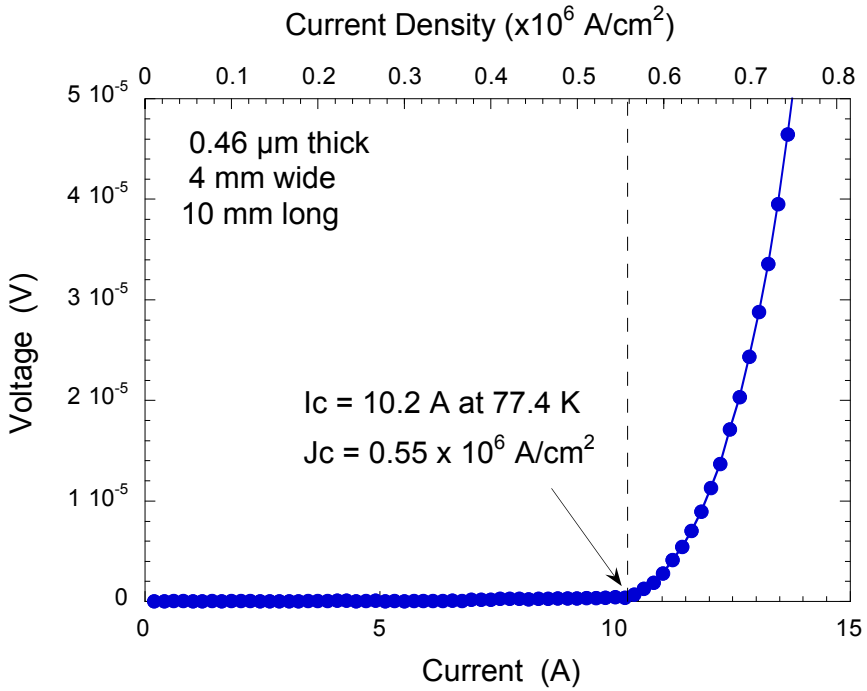
Stereograph projection

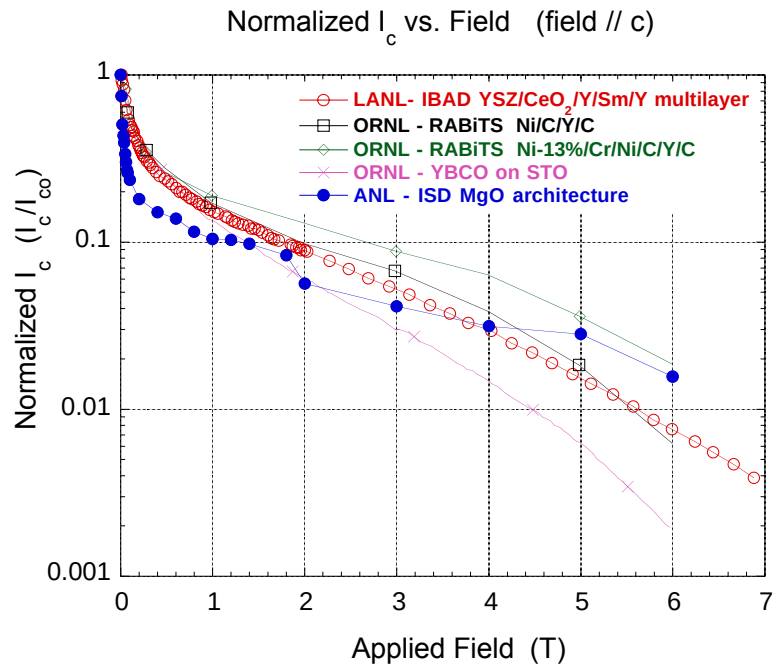


Orientation relationship:

YBCO[001] // MgO[112] and
YBCO[100] // MgO[111] or
YBCO[010] // MgO[110]

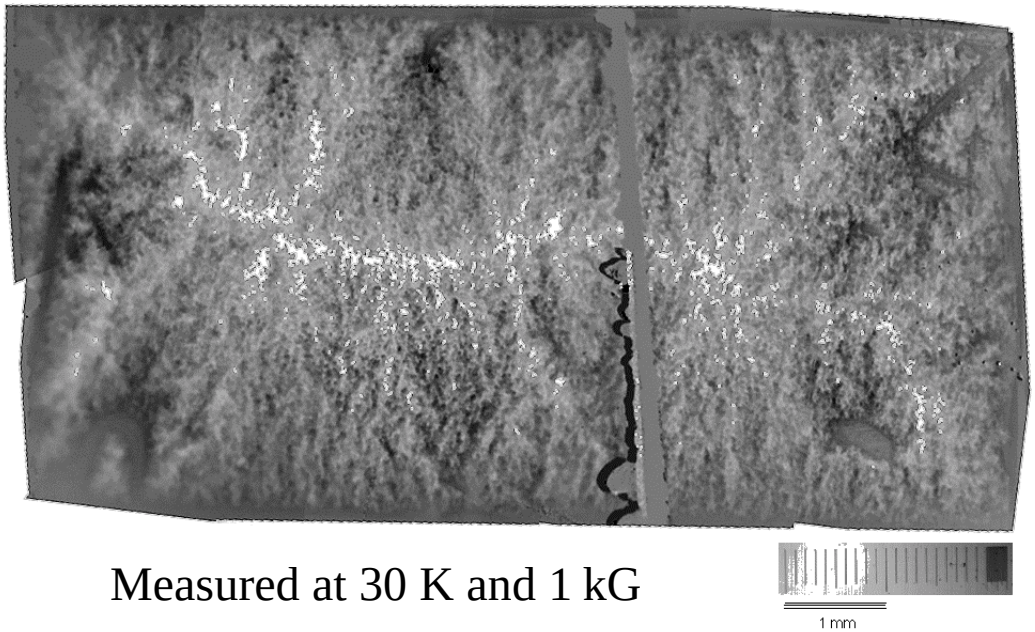
Properties of YBCO on Buffered ISD MgO





Ref: LANL and ORNL data are from 2001 Peer Review reports.

MOI of YBCO on ISD MgO



Measured at 30 K and 1 kG

- c-axis verticality
- observable defects

YBCO grown on ISD-MgO-buffered HC
Film thickness = 0.46 µm.

Research Integration

Program funding was beneficially leveraged by forming partnerships with other organizations and using external expertise/facilities.

Industry	National Labs.	Universities
• American Superconductor	• LANL	• Ohio State
• IGC-SuperPower	• ORNL	• Illinois Inst. Technology
• UES, Inc.		• U. of Illinois
		• SUNY/Albany

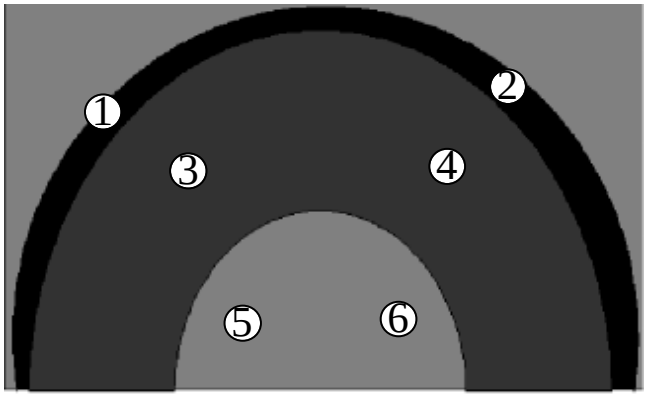
Funding leverage: DOE - Office of Science
AF/OSR; industry cost-share

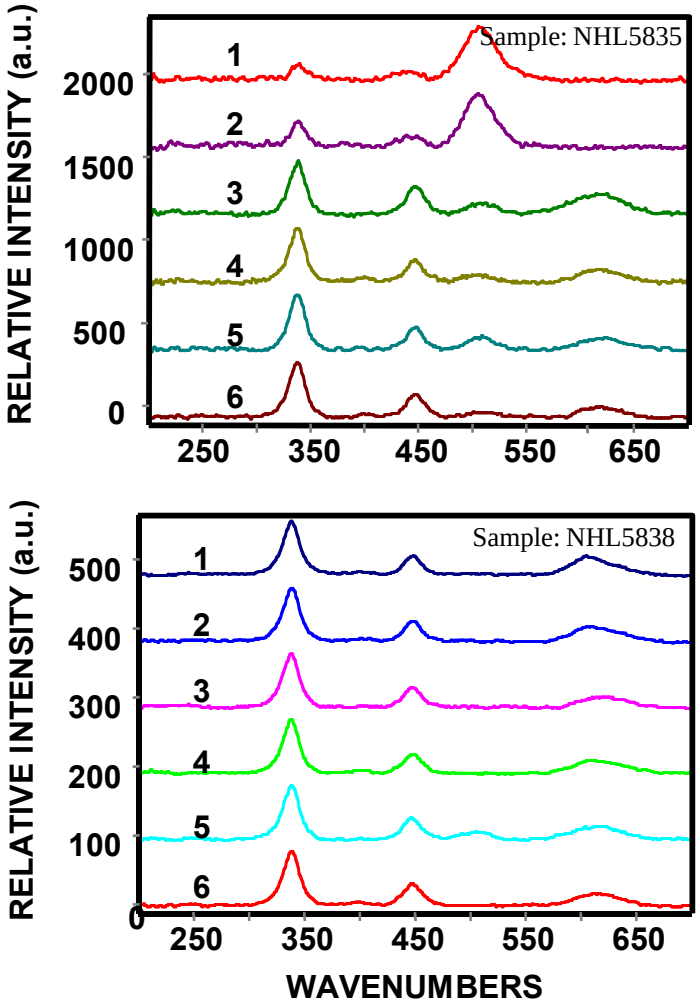
Research Integration (cont.)

- **Collaborated through two CRADAs.**
  - Characterization of buffer & YBCO layers
  - MOCVD

Raman spectroscopic characterization of coated conductors

Position of scans on surface of CC





- **O4 phonon (ca. 500 cm^{-1}) indicates a-axis grain growth.**
- **O2+/O3- phonon (ca. 340 cm^{-1}) is indicative of well-textured YBCO.**
- **Broad band @ 620 cm^{-1} is due to trace amount of $BaCuO_2$.**

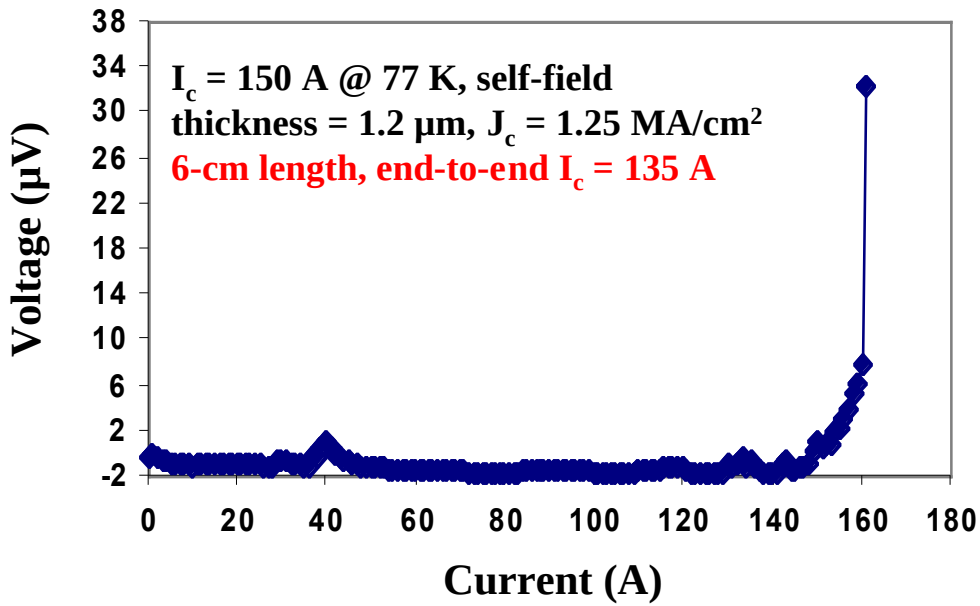
Research Integration (cont.)

IGC–SuperPower:

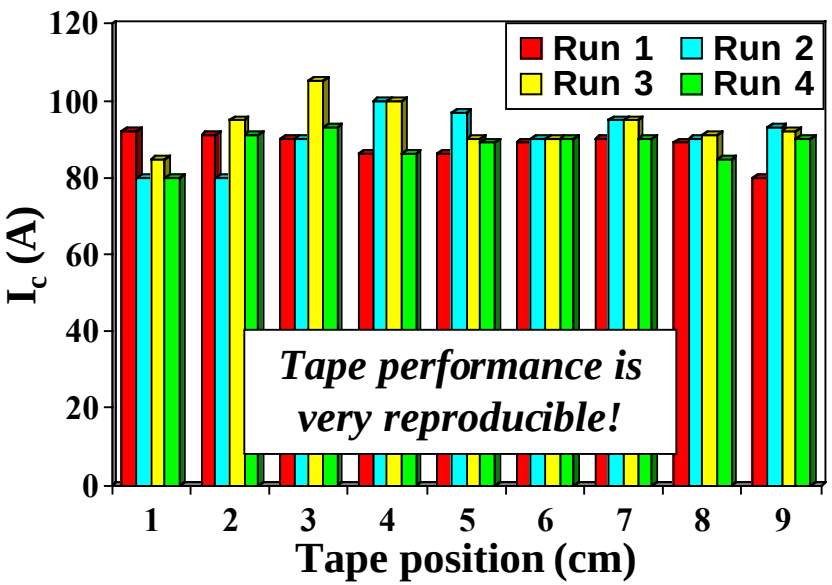
- Collaborated through two CRADAs on coated conductor development.
  - YBCO deposition by MOCVD
  - characterization of buffer & YBCO layers

MOCVD

- High throughput
- Low capital cost
- Low precursor cost in bulk quantities
- Long uninterrupted deposition runs
- Unlimited deposition zone length

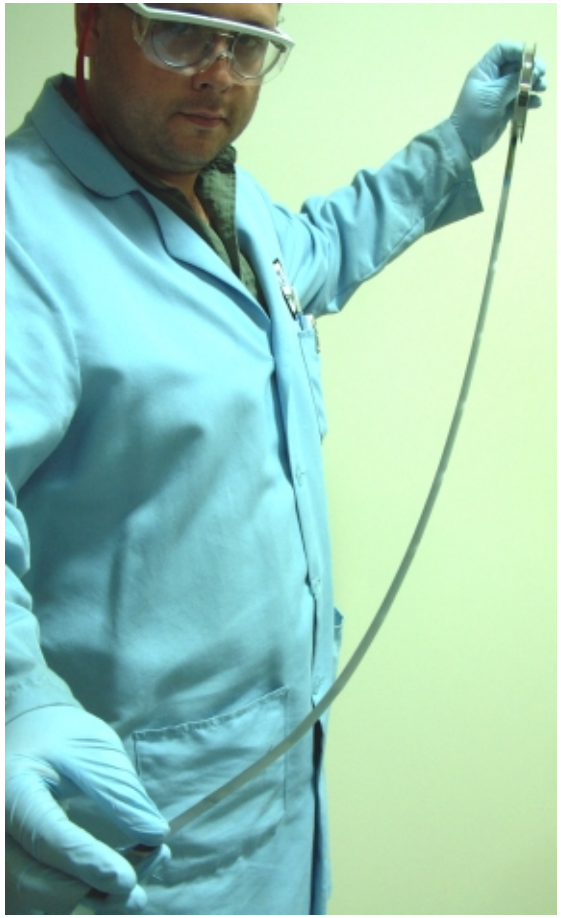


High I_cs by MOCVD in moving tapes

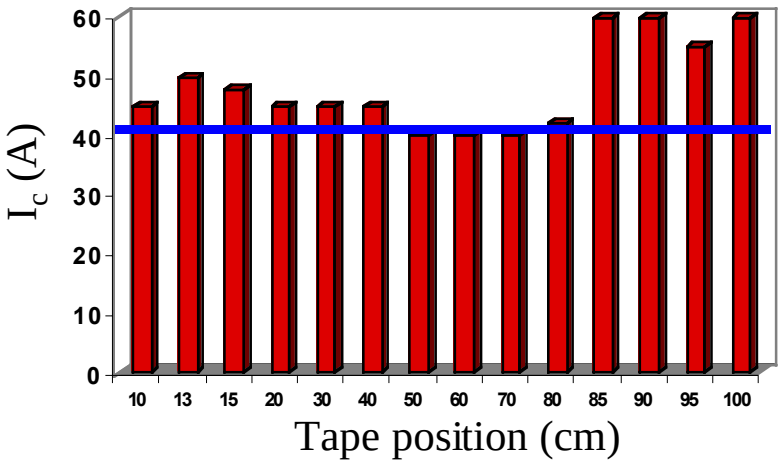


Research Integration (cont.)

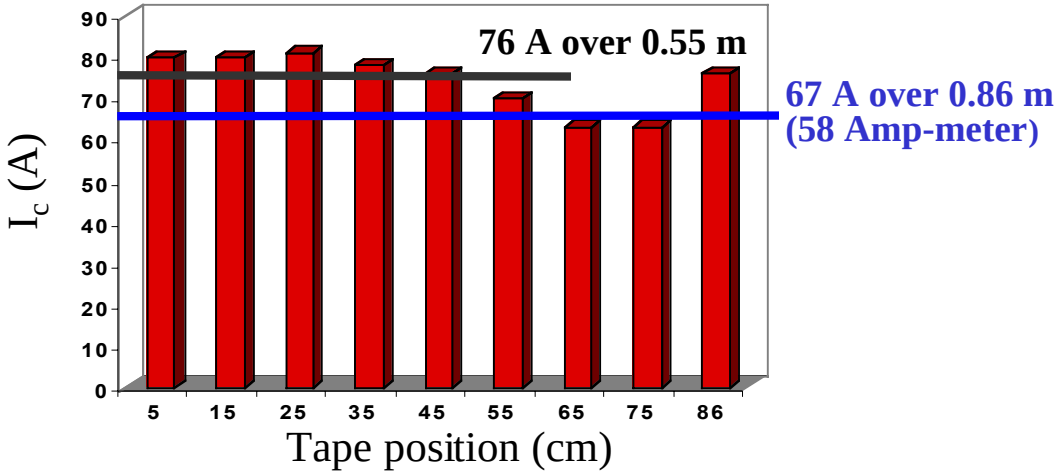
IGC has begun scaling up MOCVD to long-length tapes.



40 A achieved over 1 m



Performance improved with altered process.

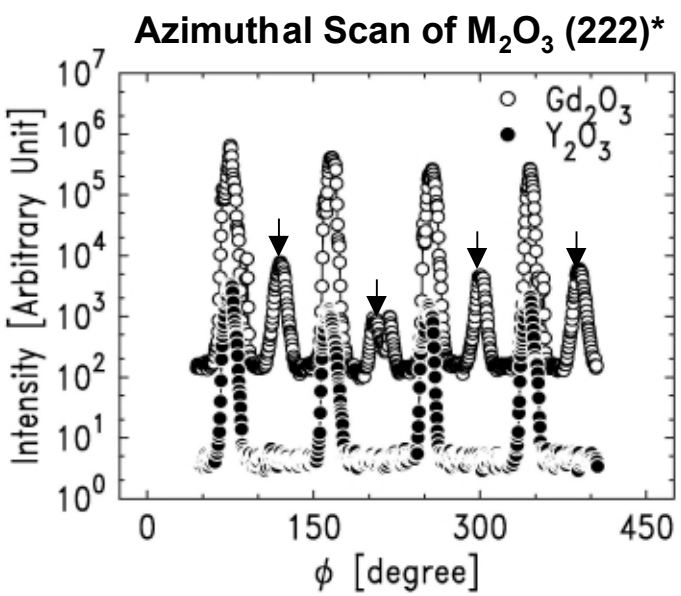


Research Integration (cont.)

American Superconductor:

- Collaboration under a CRADA on coated conductors to:
  - Characterize YBCO films, buffer/seed layers, and precursors (texture, structure, Raman spectroscopy, synchrotron x-ray)
  - ISD substrates
  - TFA precursor conversion

Synchrotron X-ray Diffraction Measurements Provide Information about Buffered Textured Metal Substrates



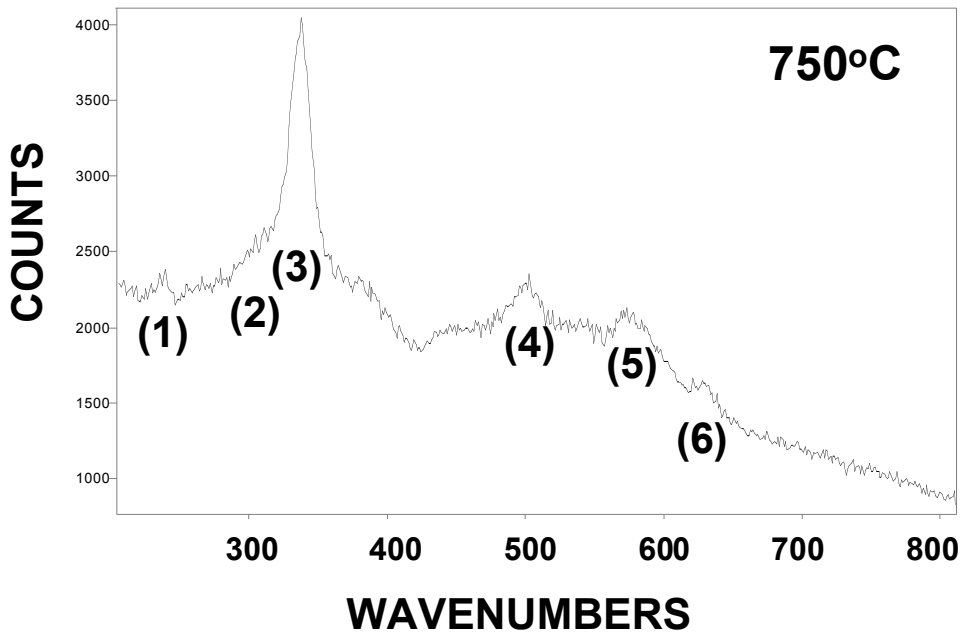
- Gd_2O_3 layer is applied by MOD method.
- Y_2O_3 layer is applied by e-beam evaporation.
- Gd_2O_3 layer is rougher than Y_2O_3 layer (from reflectivity measurements).
- Gd_2O_3 and Y_2O_3 layers are very well ordered.
- Domain sizes in c-direction: 22 nm for Gd_2O_3 and 25 nm for Y_2O_3.

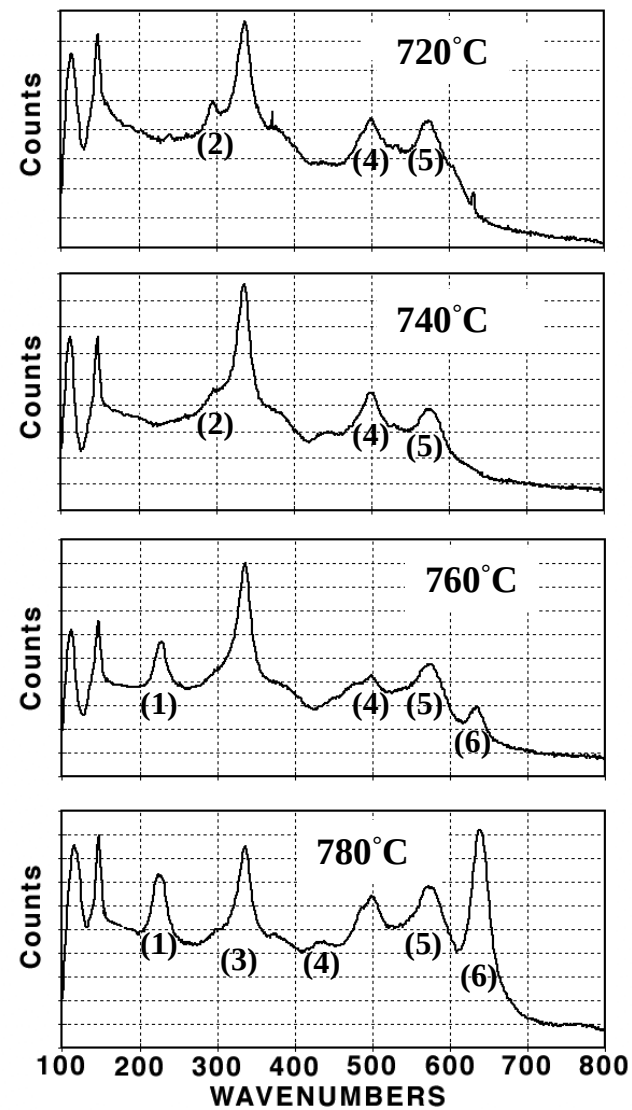
Research Integration (cont.)

AMSC CRADA: Characterization of YBCO Films

Raman Spectra of YBCO Films Prepared Using a Trifluoroacetate (TFA) Precursor

300-nm-thick YBCO films on $LaAlO_3$ (sc)





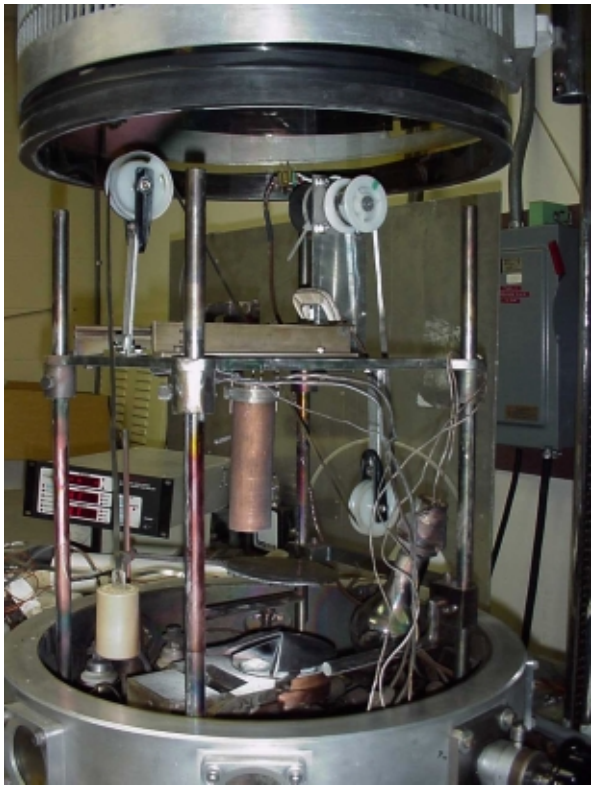
(1) "O" defect
(2) CuO
(3) vs. (4): c-axis verticality
(3) vs. (5): cation disorder
(4) oxygen stoichiometry
(6) $BaCuO_2$

Research Integration (cont.)

Universal Energy Systems, Inc:

- Collaboration under AFOSR STTR Program "Coated High-T_c for Power Systems."
  - development of textured MgO by ISD
  - characterization of buffer layers
  - YBCO deposition & characterization

- ≈35-cm-long Hastelloy C tape coated with ISD-MgO
- ϕ-scan FWHM = 12-18°

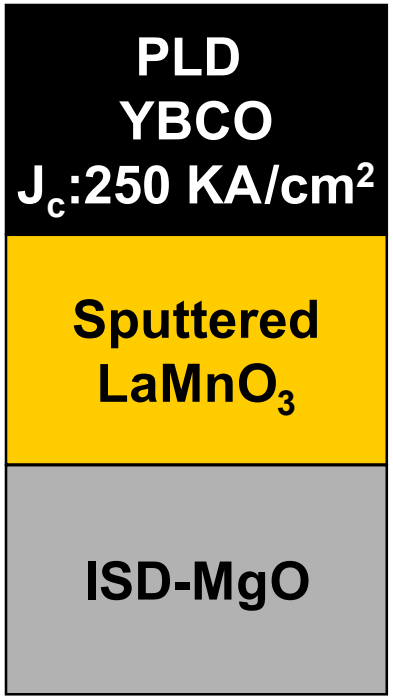


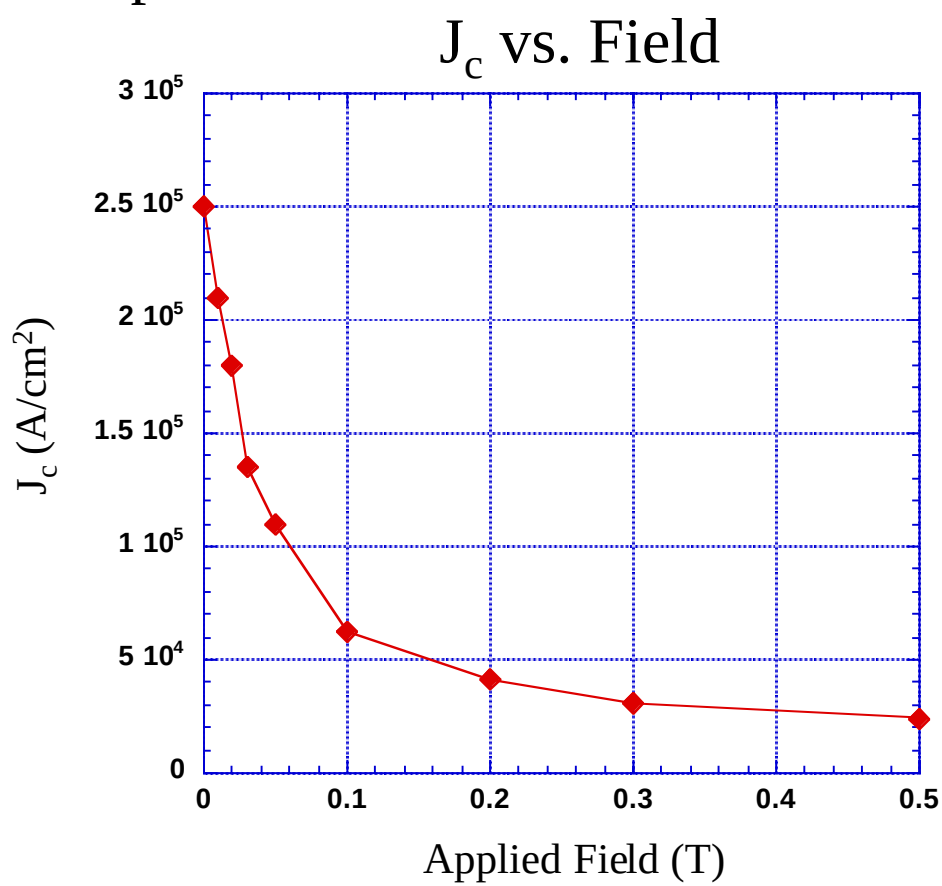
ISD-reel-to-reel setup

Research Integration (cont.)

ORNL-ANL:

- Alternate buffer layer development

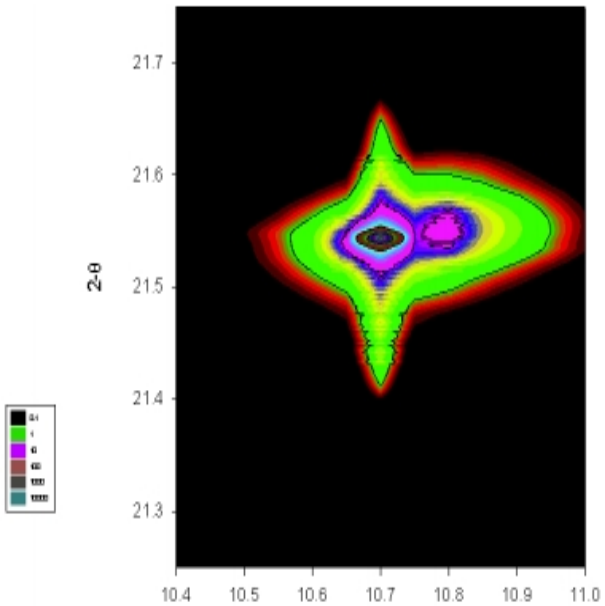




Research Integration (cont.)

LANL-ANL:

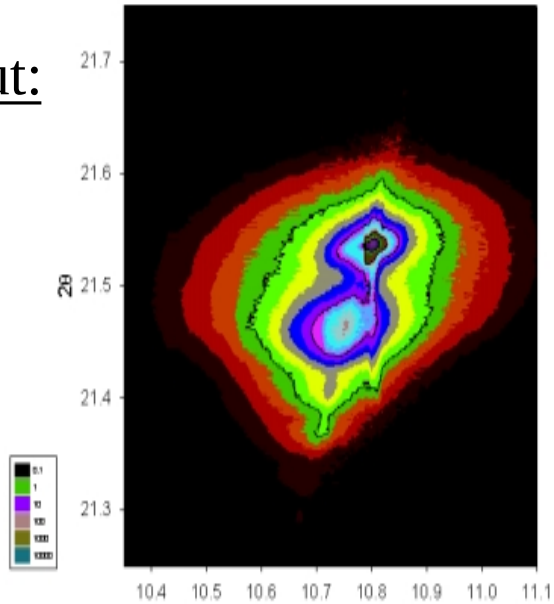
- Synchrotron-based measurements of coated conductors (ANL's Advanced Photon Source)



Eu-123/STO

Provides information about:

- Composition gradients
- Lattice disorder
- Strain
- Texture
- Interplanar tilt



Eu-123/Er-123/STO

- PLD protocol for depositing ceria and YBCO on ISD substrates
- Proton-irradiation of coated conductors to improve performance

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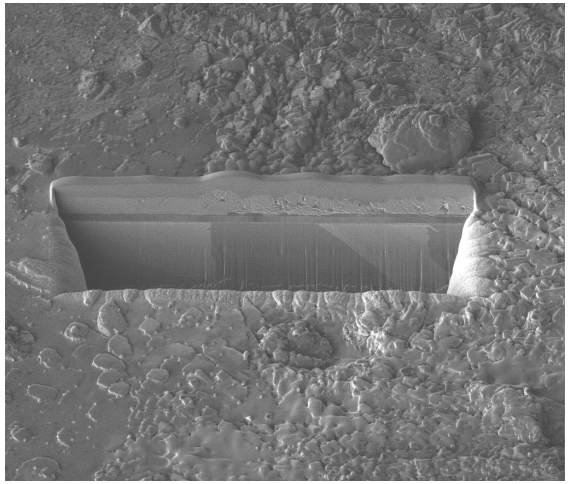
STATE UNIVERSITY OF NEW YORK

Research Integration (cont.)

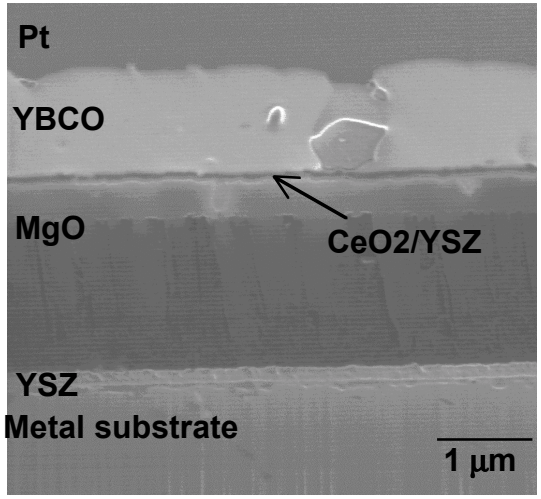
- Analytical techniques to analyze coated conductors
  - EDS, AFM, FIB, AES

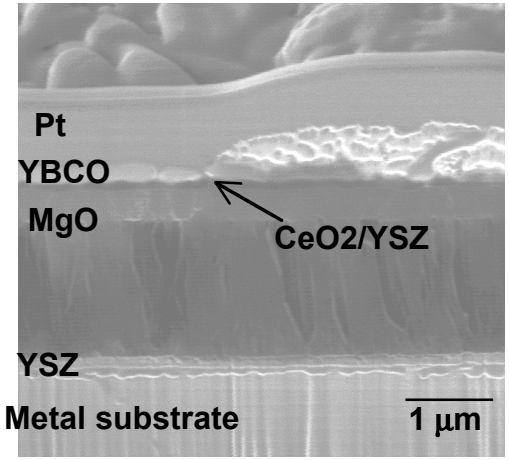
Focused Ion Beam (FIB) study of coated conductors

- **A 30 kV beam of Ga ions is focused on the sample surface.**
- **The Ga ions sputter off material in an user-defined region.**
- **The sample is tilted 45° to observe the cross-section using secondary electrons.**



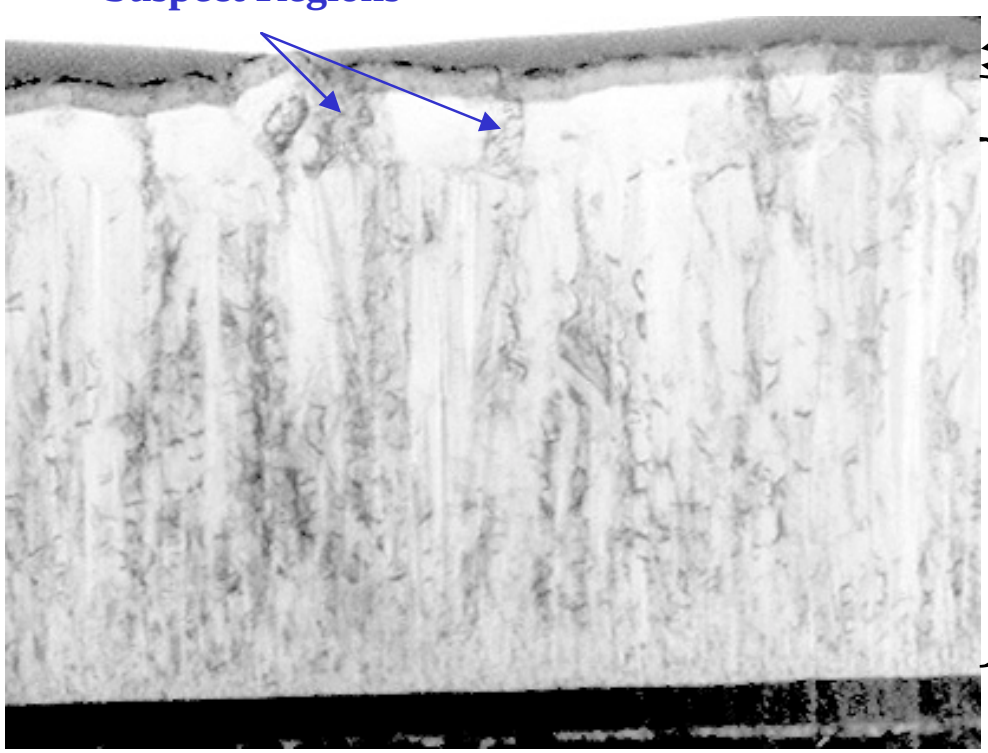
Cross-section view of ISD YBCO





Research Integration (cont.)

- Identify phases and monitor texture using 2D X-ray detector system.
  - reaction of buffer layers during deposition of YBCO
  - better statistics for phase identification
- Characterize layers by FIB/SEM/TEM.



Summary:

- Using ISD MgO architecture, YBCO coated conductors were fabricated with $J_c = 5.5 \times 10^5$ A/cm^2, a factor of 5 improvement over last year.
- ISD is a fast and robust method to produce sharp biaxial texture in MgO on nontextured metallic substrates.
- Homo-epi MgO and YSZ buffer layers reduce surface roughness and improve biaxial alignment.
- YBCO on ISD MgO is biaxially aligned, YBCO(103) ϕ-scan FWHM ≈12°, and shows an unique orientation relationship with MgO.
- High-quality YBCO-coated conductors were reproducibly fabricated by the ISD MgO process.