2003 Annual Superconductivity Peer Review

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National Renewable Energy Laboratory

03458201

Superconductivity Program at NREL for Electric Systems

Non-Vacuum Techniques for HTS Coated Conductors

Budget: $500 K

NREL Team Members: Raghu Bhattacharya (Team Leader)
Jun Chen, Priscila Spagnol, Phil Parilla, Anna Duda, Don Selmarten, Steve Rapp (Teacher Intern), Mark Wehrenberg (Student Intern)

University Cooperative Programs*: Boston College (Zhifeng Ren), University of Colorado (Allen Hermann)

Collaborations: National Laboratories: ORNL (D.K. Christen, T. Aytug, C. Cantoni, A. Gapud, A. Goyal, K-H. Kim, D.F. Lee, M. Paranthaman, E. Miller, N. Rutter, J.R. Thompson, H-Y. Zhai), LANL (Steve Ashworth), BNL (Masaki Suenaga)

Industry CRADA: Oxford Superconducting Technologies (Ken Marken, Miao Hanping)

**University Cooperative Programs are funded by NREL.*

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Program Objectives/Relevance (FY 2003)

- The U.S. Department of Energy's vision is, "Low-cost, high performance YBCO coated conductors will be available in 2005 in kilometer lengths. For applications in liquid nitrogen, the wire cost will be less than $50/KA-m, while for applications requiring cooling to temperature of 20–60 K the cost will be less than $30/kA-m."
- One of the important critical needs to achieve this goal is to develop a simple low-cost technology for producing HTS and also buffer layer for Ni-alloy substrates.
- NREL's project offers low-cost, long length, continuous non-vacuum electrodeposition and spray deposition processing technology for development of both HTS and buffer layer.

Program Objectives/Relevance (FY 2003)

Three major tasks were undertaken in FY 2003

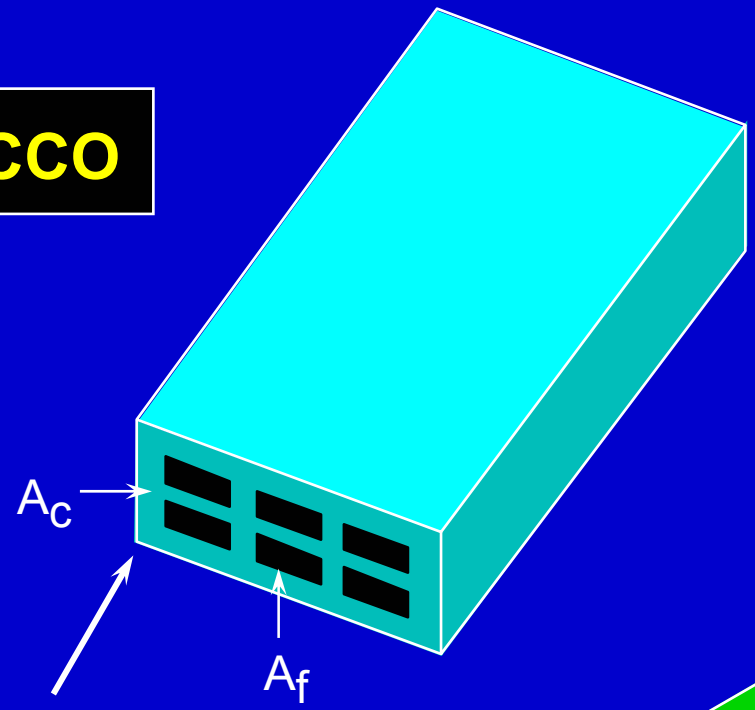
- Demonstrate current density for electrodeposited Bi-2212 film on polycrystalline Ag comparable with dip-coated films
- Demonstrate the improved superconductor properties of biaxially textured Bi-2212 films compared to uni-axial textured films
- Prepare electrodeposited buffer layer for YBCO-coated conductors

Approach

- Develop biaxially textured Bi-oxide super-conductors by non-vacuum electrodeposition and spray deposition process (NREL/OST Collaboration).
- Assist Oxford Superconductor Technology to develop the conductor for a HTS Magnetic Imaging System (NREL/OST Collaboration).

Conductor Performance Measures

BSCCO



I_c (T, B(θ), criterion)

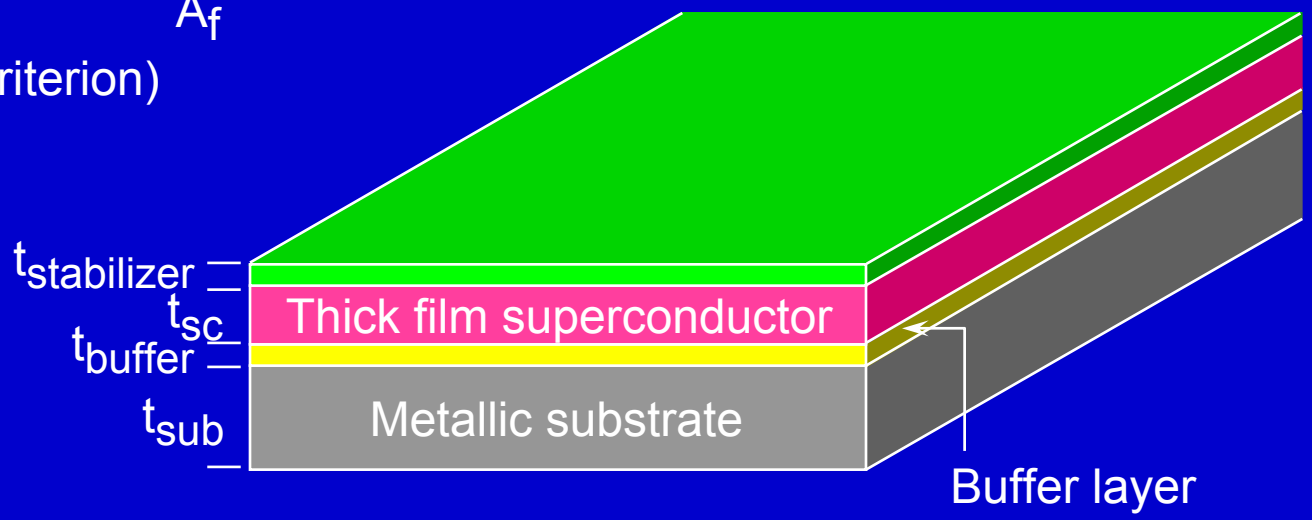
$J_c = I_c/A_f$

$J_e = I_c/A_c$

Fill factor = A_f/A_c

J_e = fill factor x J_c

Coated Conductor



$J_e = J_c(sc)\, t_{sc}/(t_{sc} + t_{sub} + t_{buffer} + t_{stab})$

NREL

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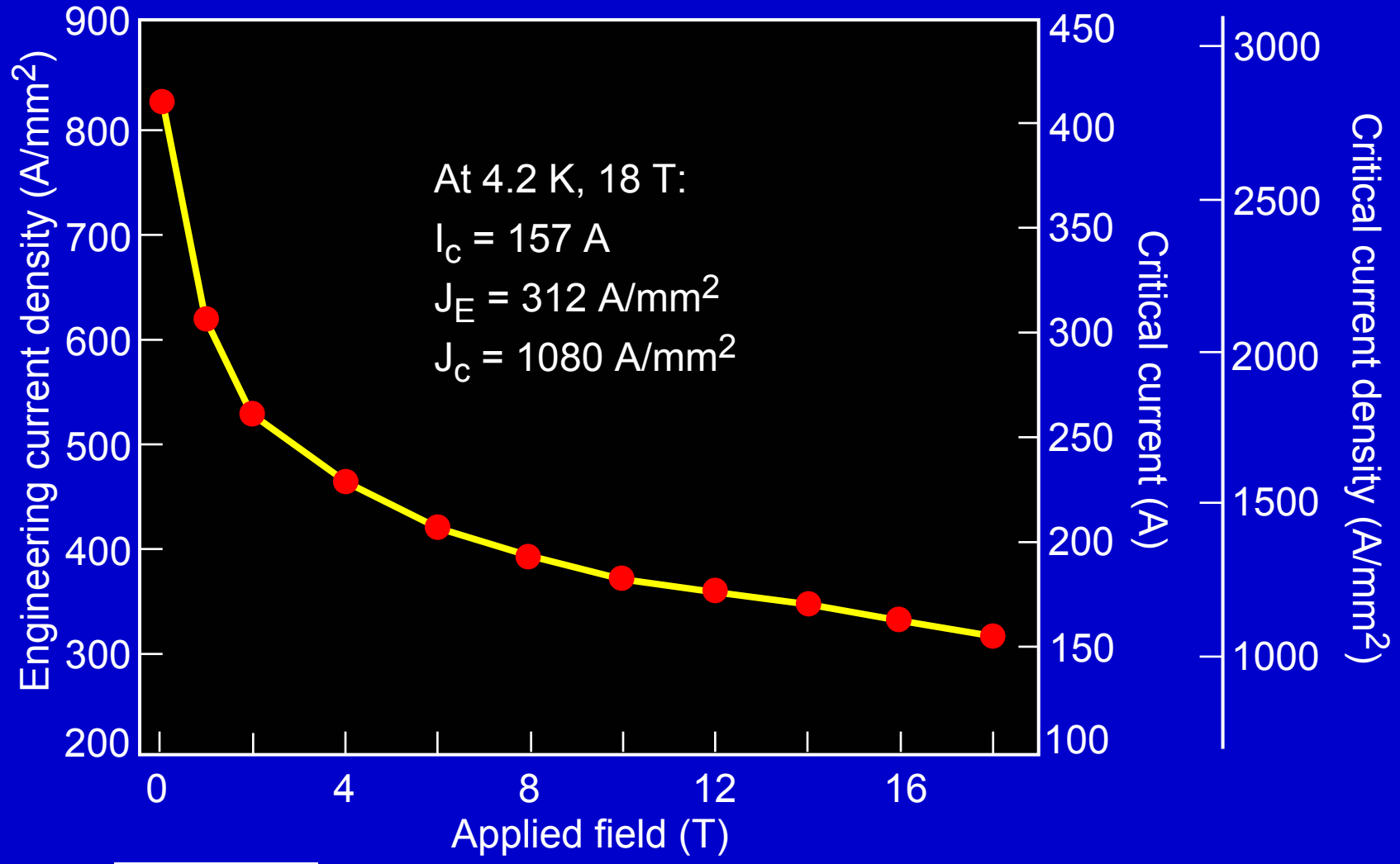
J_E, J_c, and I_c as a Function of B at 4.2 K in the 0.8 mm Bi-2212 Tape (OST Bi-2212)



Source: K.R. Marken, et al, ASC 2002, Houston, TX

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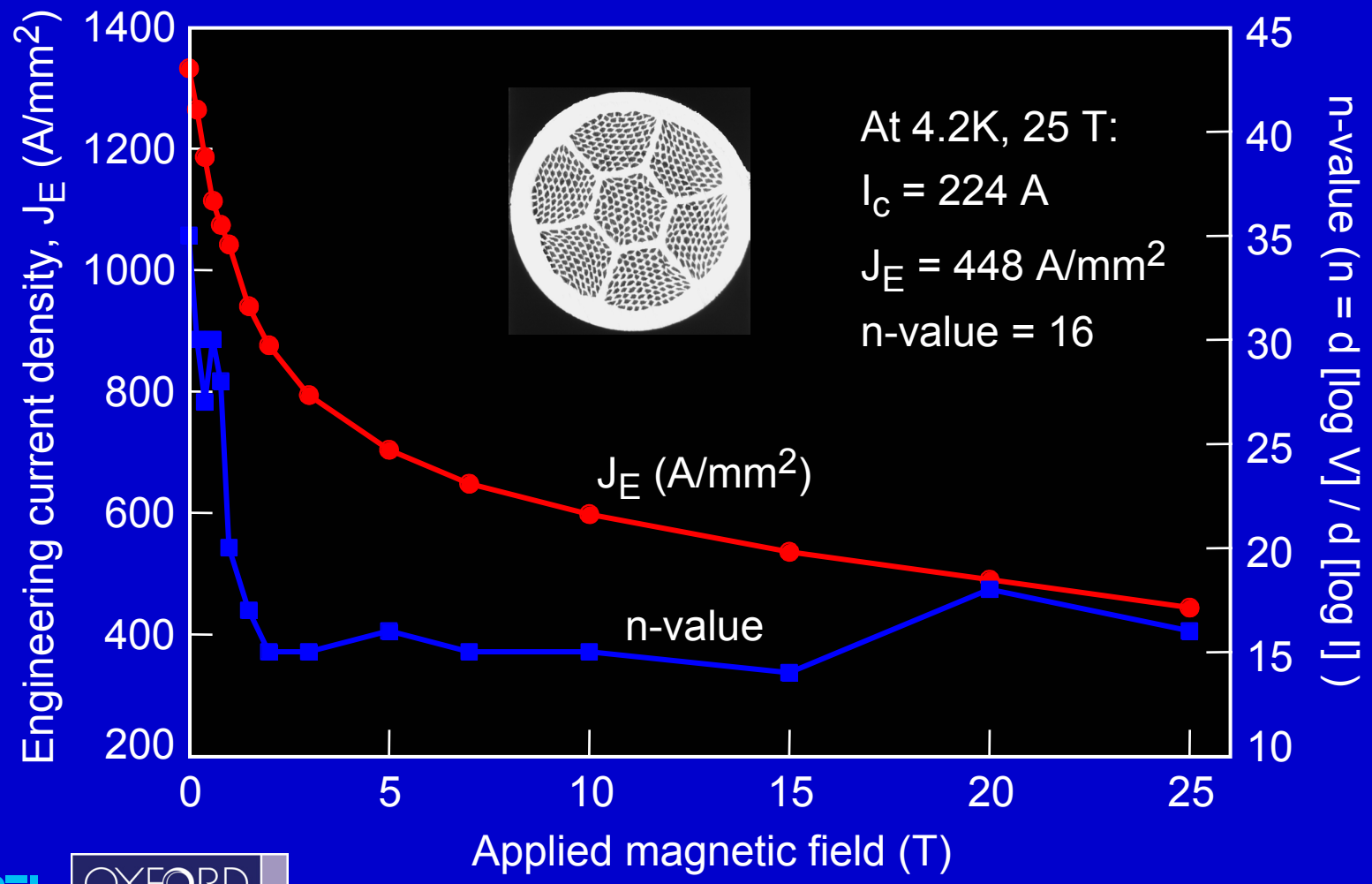
OST 2212 Wire Billet PMM030224

(0.8 mm diameter, 28% superconductor normalized to entire cross section)



Source: K.R. Marken (private communication)

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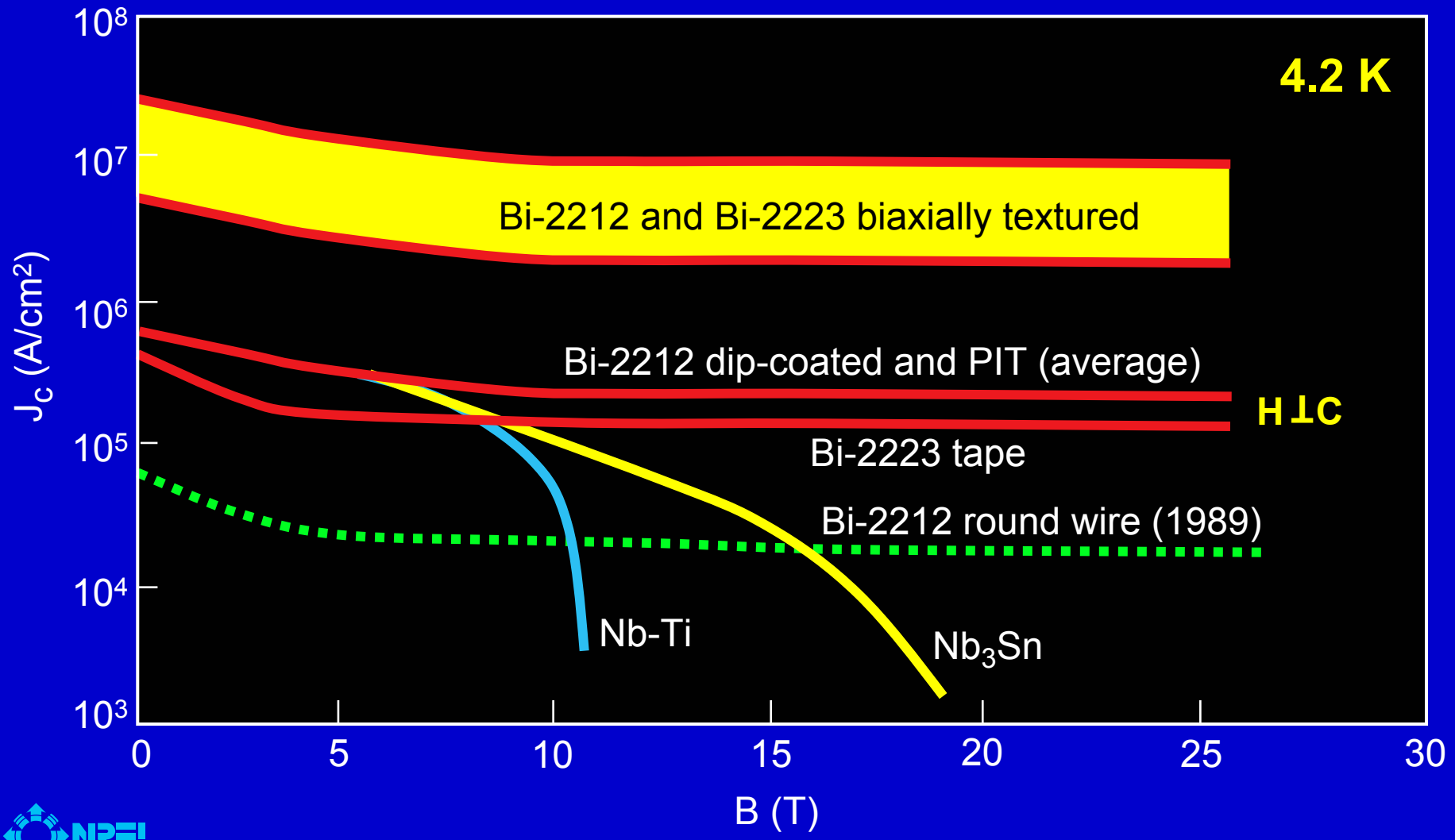
Prospect for Bi-2212 and Bi-2223 Biaxially Textured Coated Conductor



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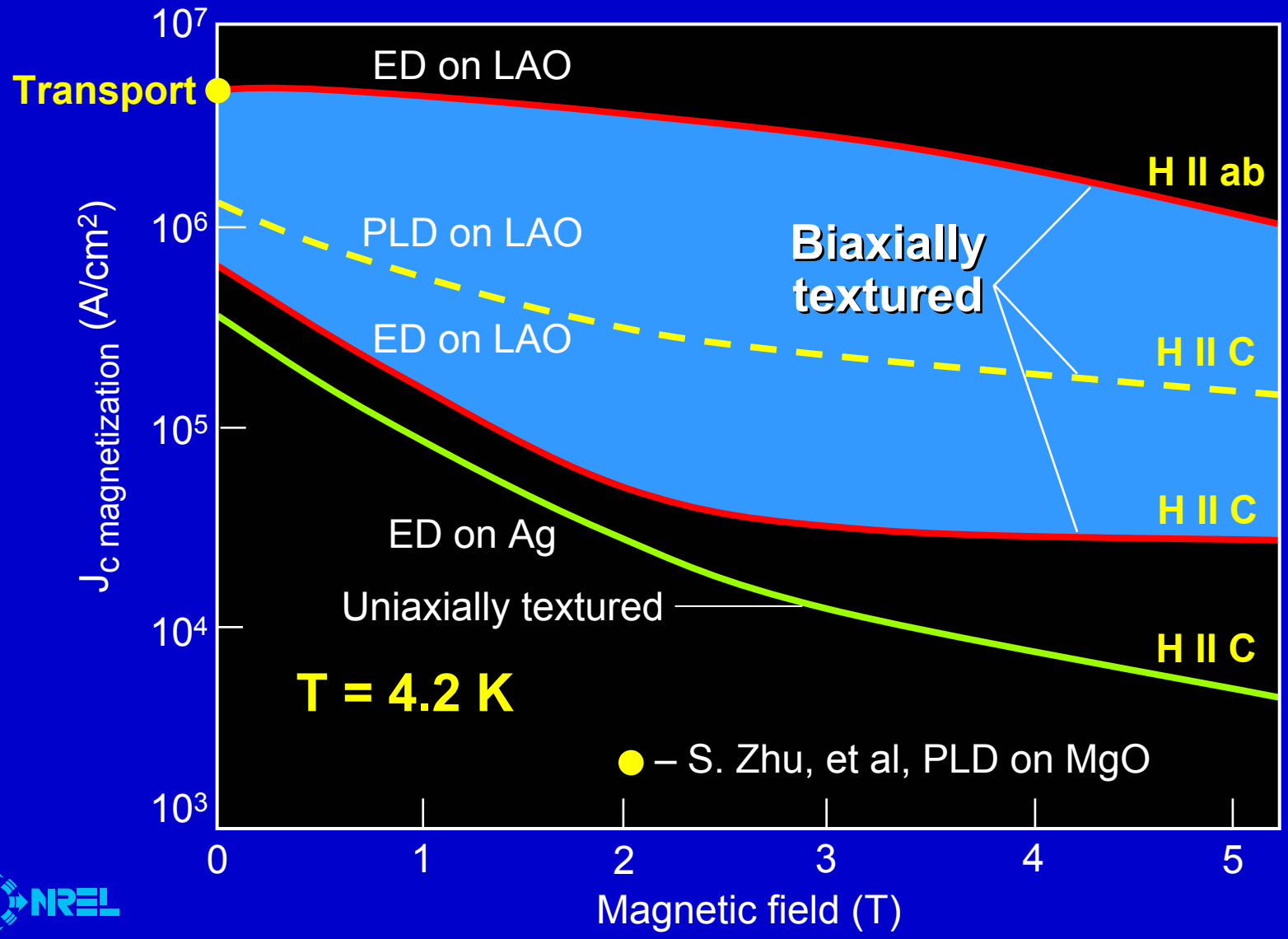
Comparison of Biaxial and Uniaxial Textured Bi-2212



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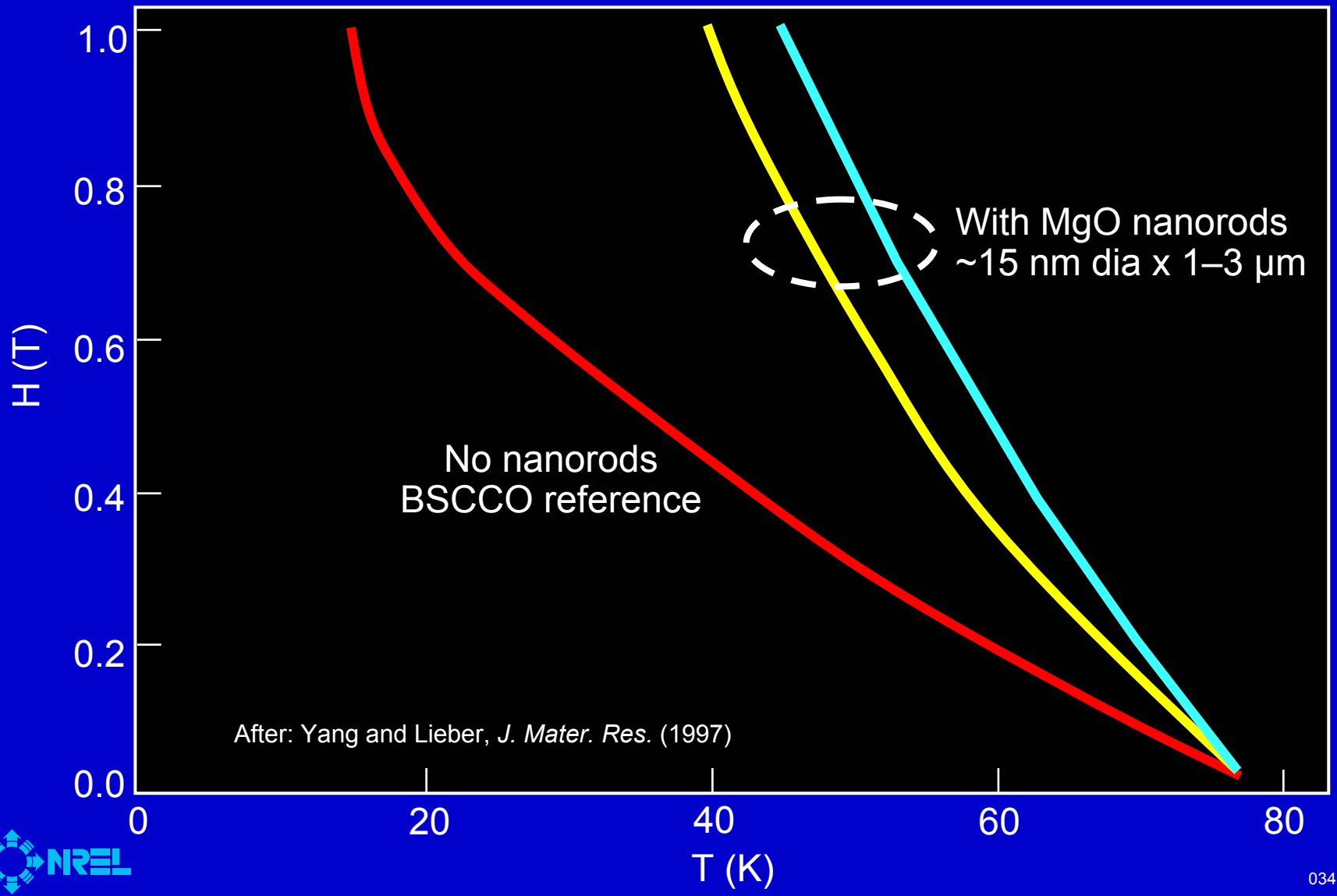
Plots of the Irreversibility Lines for Nanorod/BSCCO-2212 Composites



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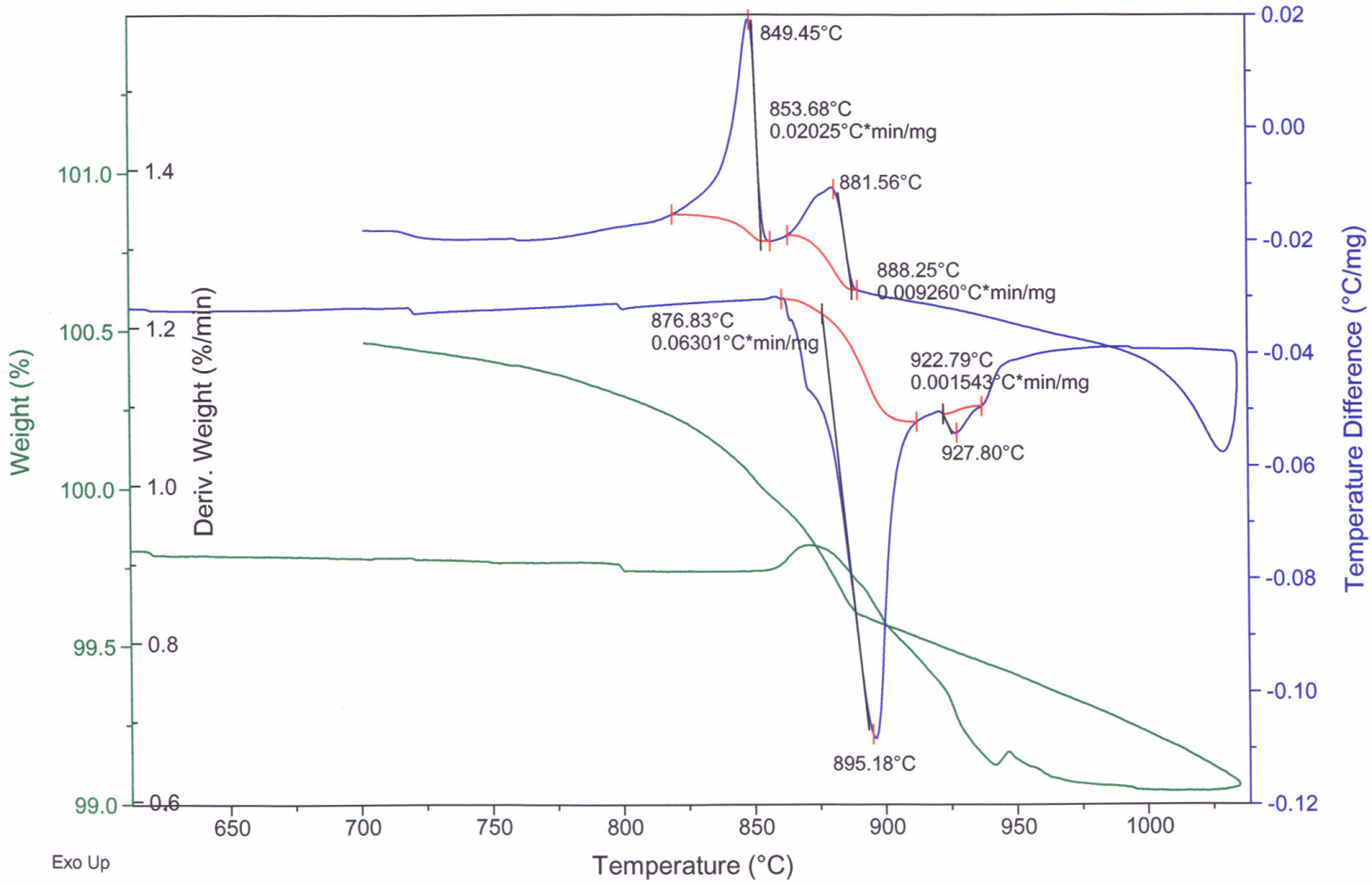
TGA-DTA Analysis Data on OST Powder



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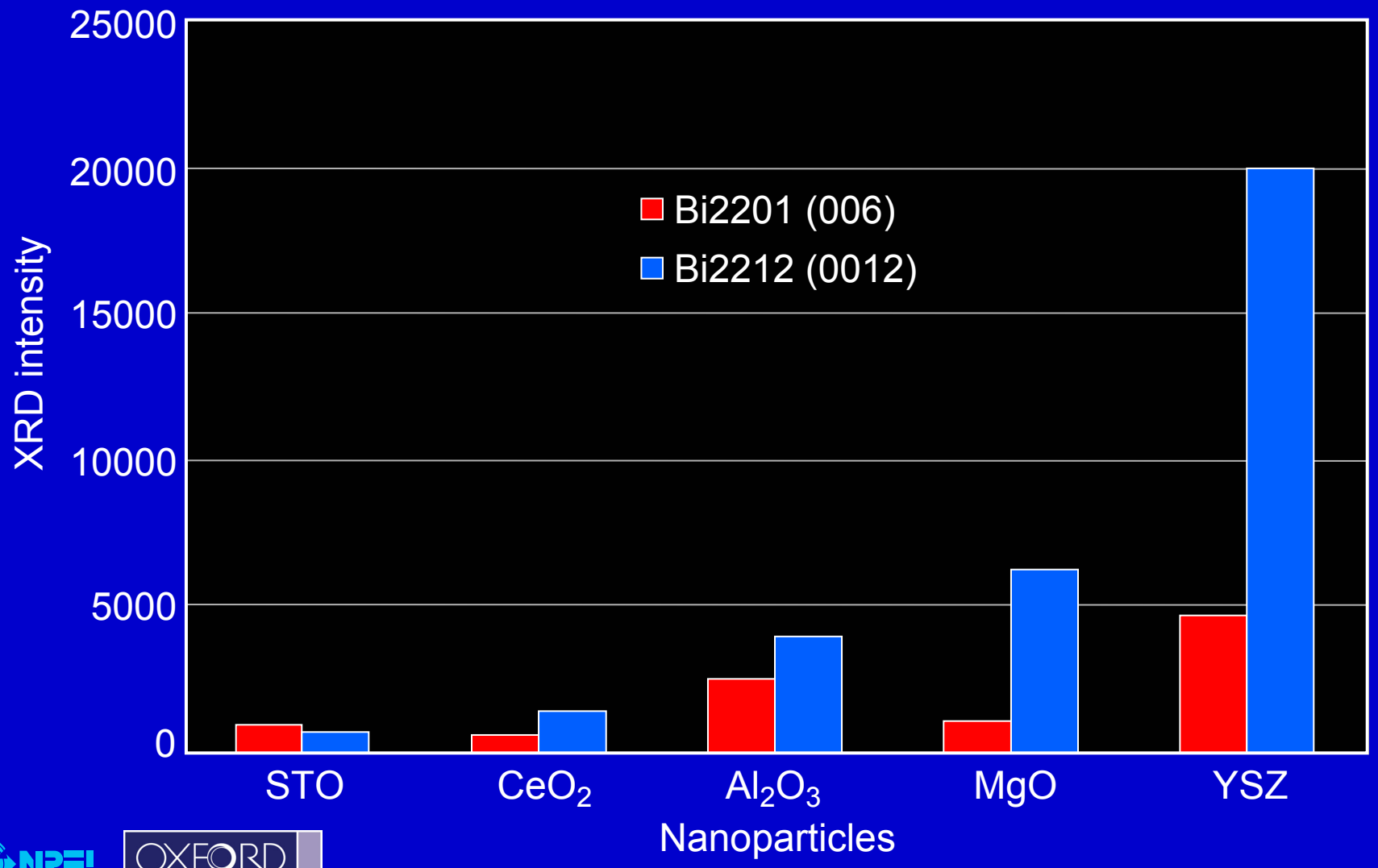
DTA/TGA Results of OST Powders

Bi-2212 Powder Composition	T_{onset} (°C)
$Bi_{2.17}Sr_2Ca_{0.97}Cu_2Ag_{0.09}$	876.83
$Bi_{2.14}Sr_{1.74}Ca_{1.18}Cu_2$	895.99
$Bi_{1.93}Sr_{2.00}Ca_{0.92}Cu_2$	897.55
$Bi_{1.88}Sr_{1.91}Ca_{0.93}Cu_2$	899.62
Powder + MgO nano-particle	888.10
Powder + YSZ nano-particle	892.62
Powder without any nano-particle	897.22

Addition of Ag and MgO lowers the onset temperature significantly.

XRD Theta/2 Theta Ratio for Bi-2212 Films with Nanoparticles



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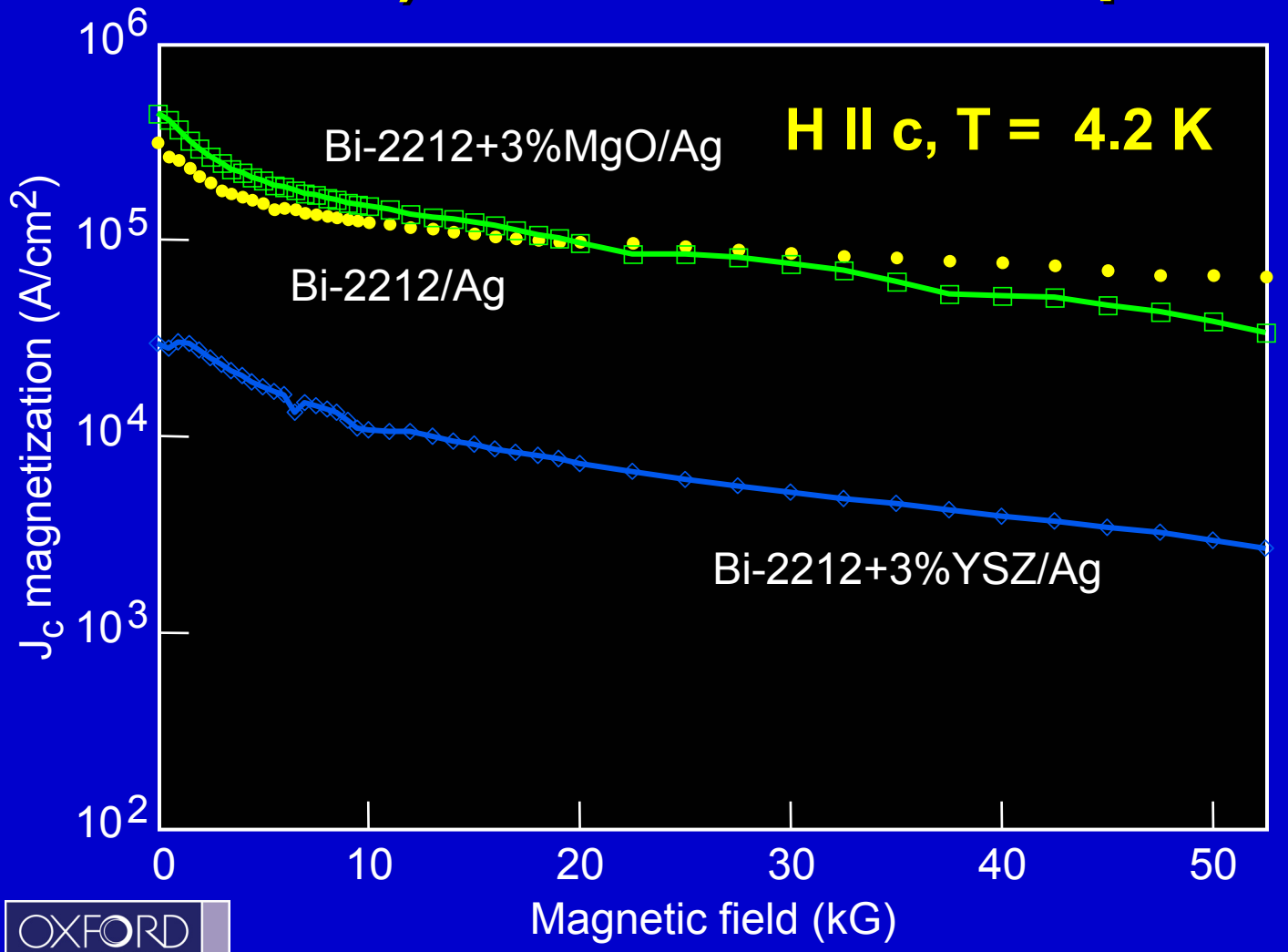
J_c vs Magnetic Field for Spray Deposited Bi-2212 on Polycrystalline Ag Foil with (MgO and YSZ) and without Nanoparticles



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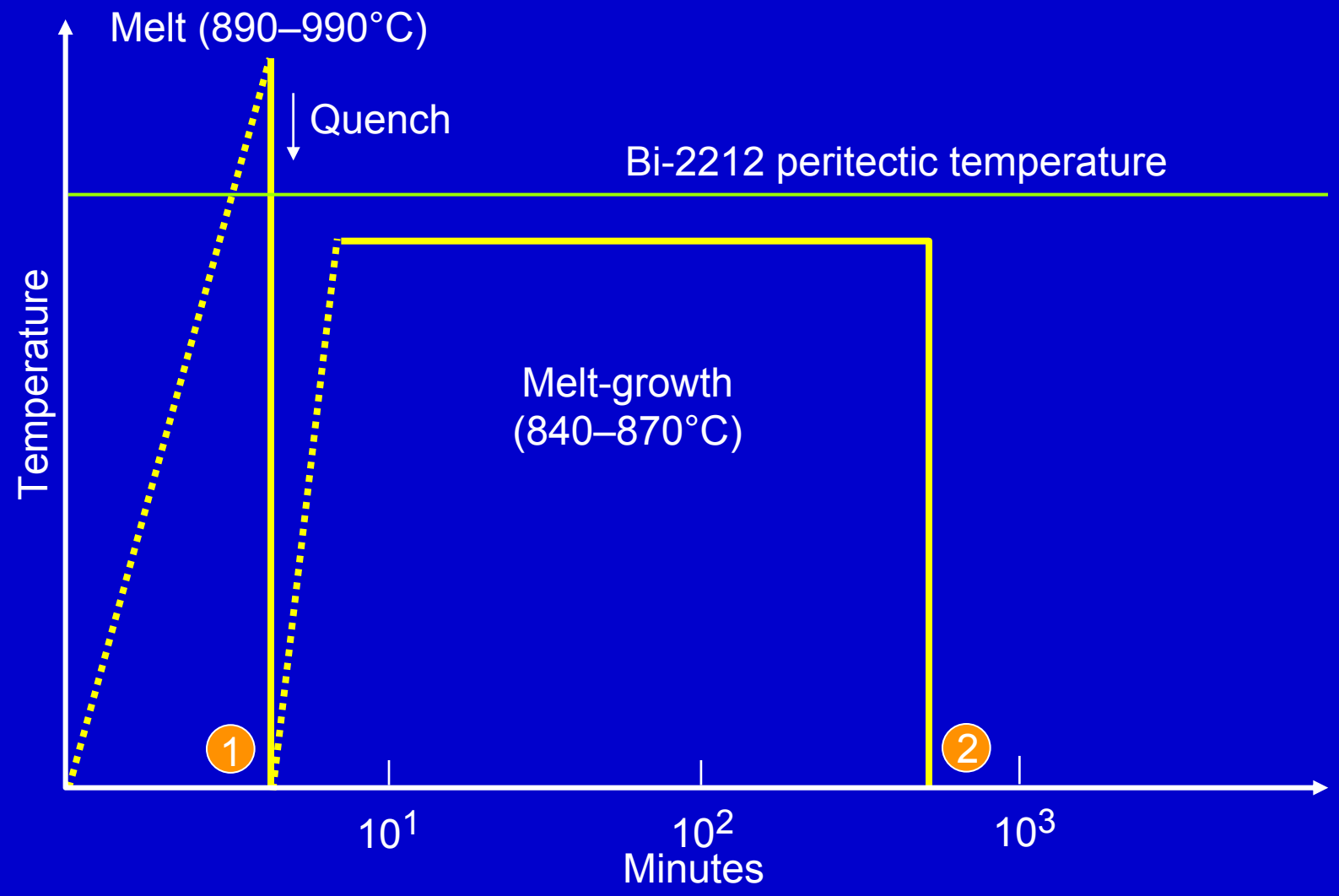
Melt-Quench/Melt-Growth Processing Schematic for Electrodeposited Bi-2212



1. Bi-2201 + Bi-2212 + (Sr, Ca)CuO_2 + Cu-free phase
2. Bi-2212 + Bi-2201 (weak) + mixed phase (weak)

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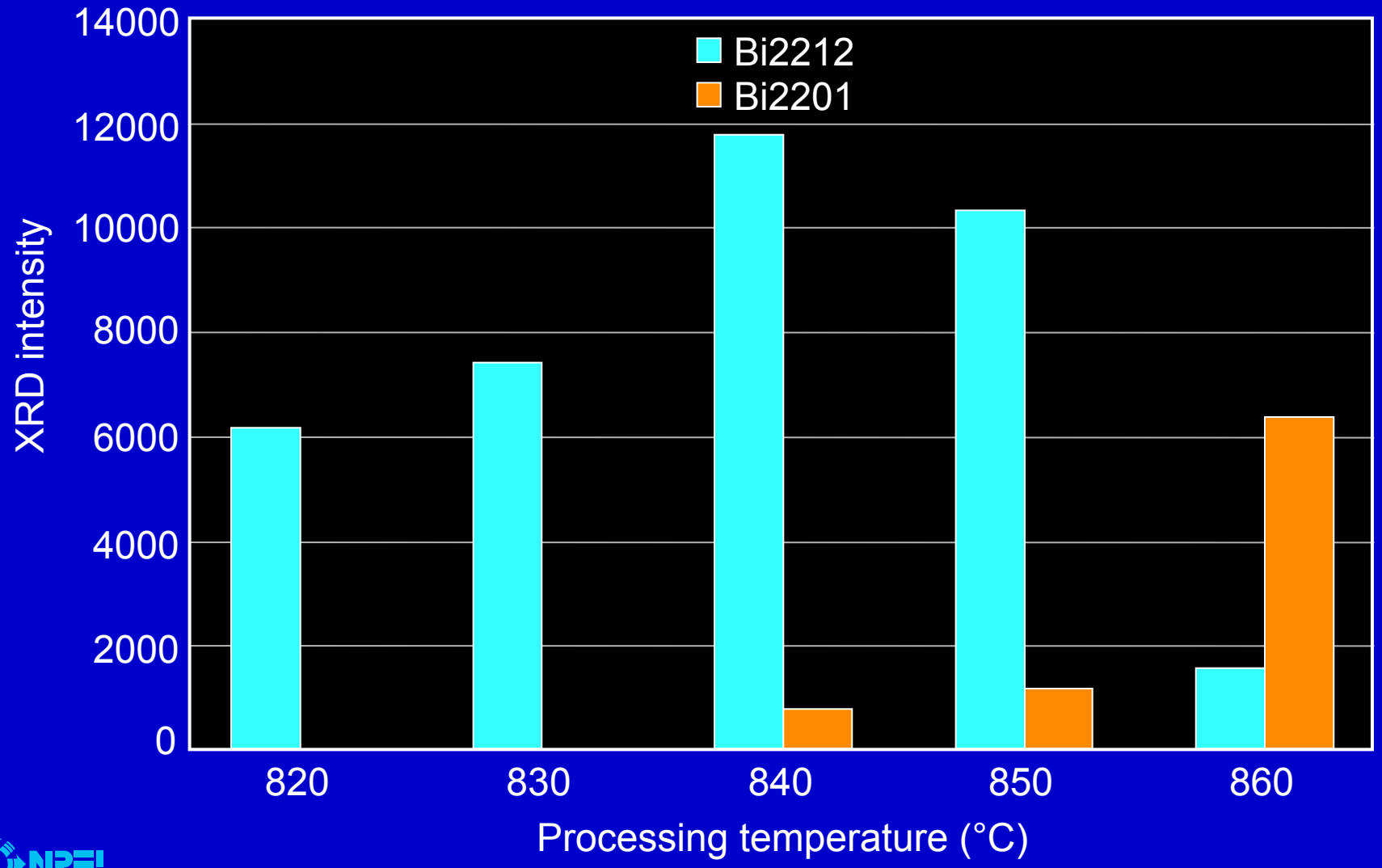
Electrodeposited Bi-2212; Melt-Quench/ Melt Growth (No $AgNo_3$) Process



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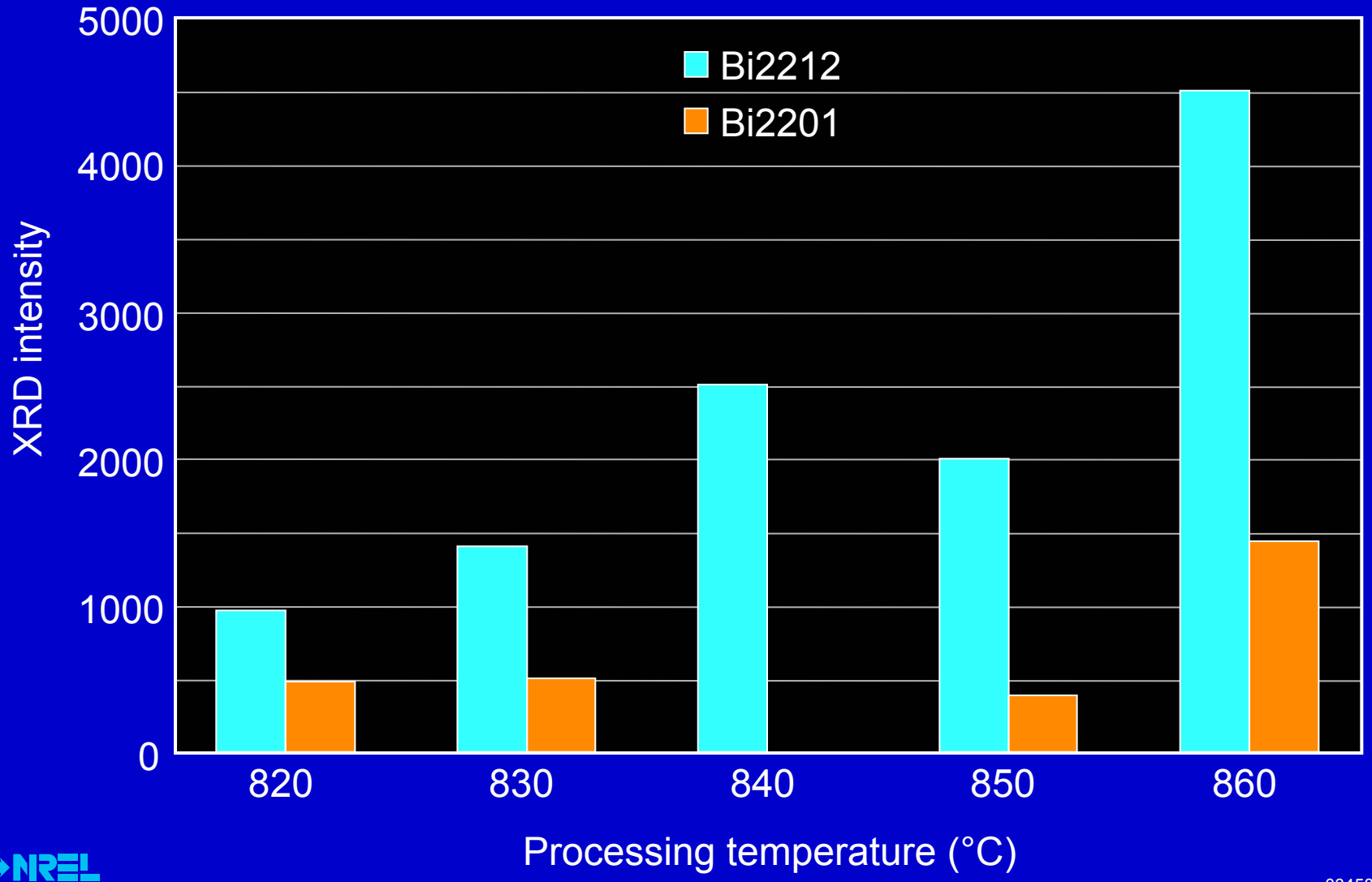
Electrodeposited Bi-2212; Melt-Quench/ Melt Growth (0.5 gm $AgNo_3$) Process



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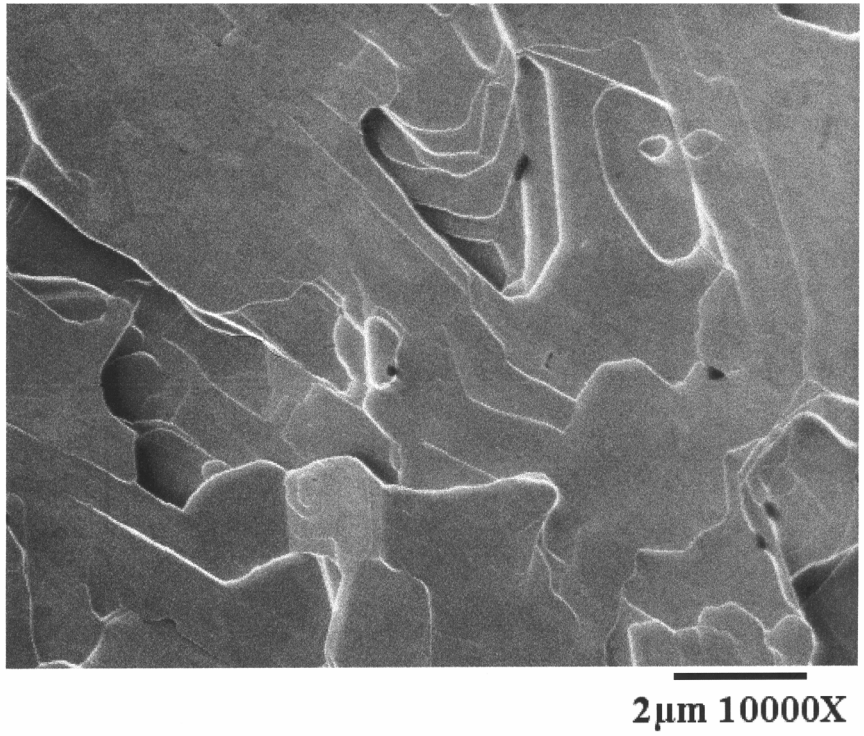
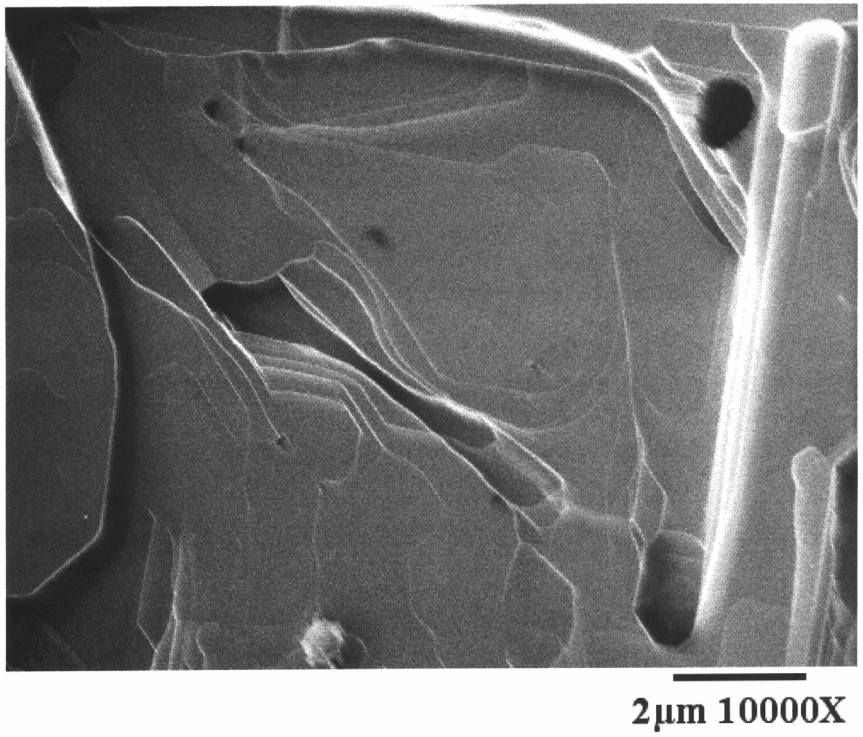
SEM of Melt-Processed ED Bi-2212

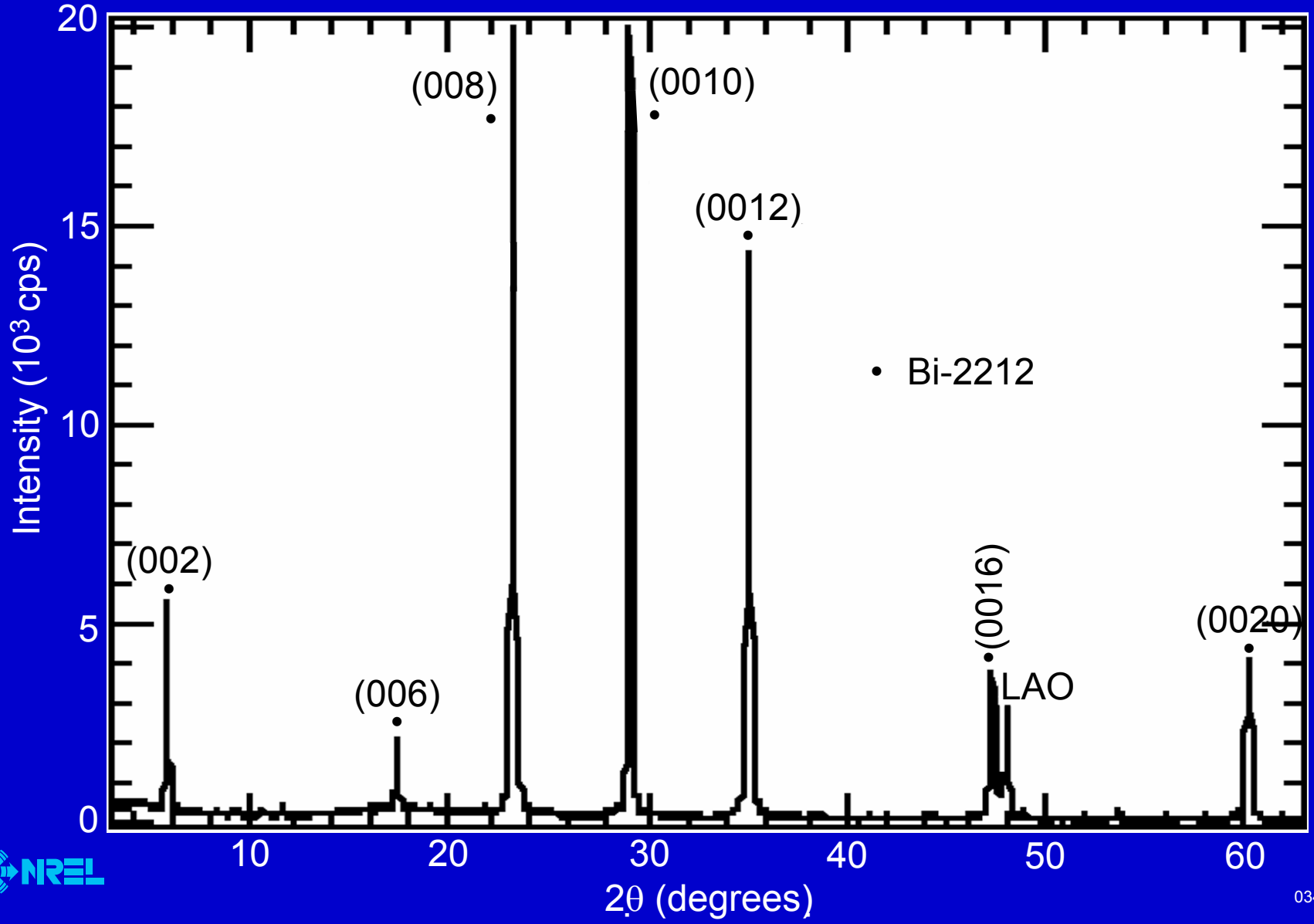
Bi-2212 on LAO



Bi-2212 on Ag



θ/2θ Scan of Electrodeposited Bi-2212 on LAO Melt-Quench/Melt-Growth



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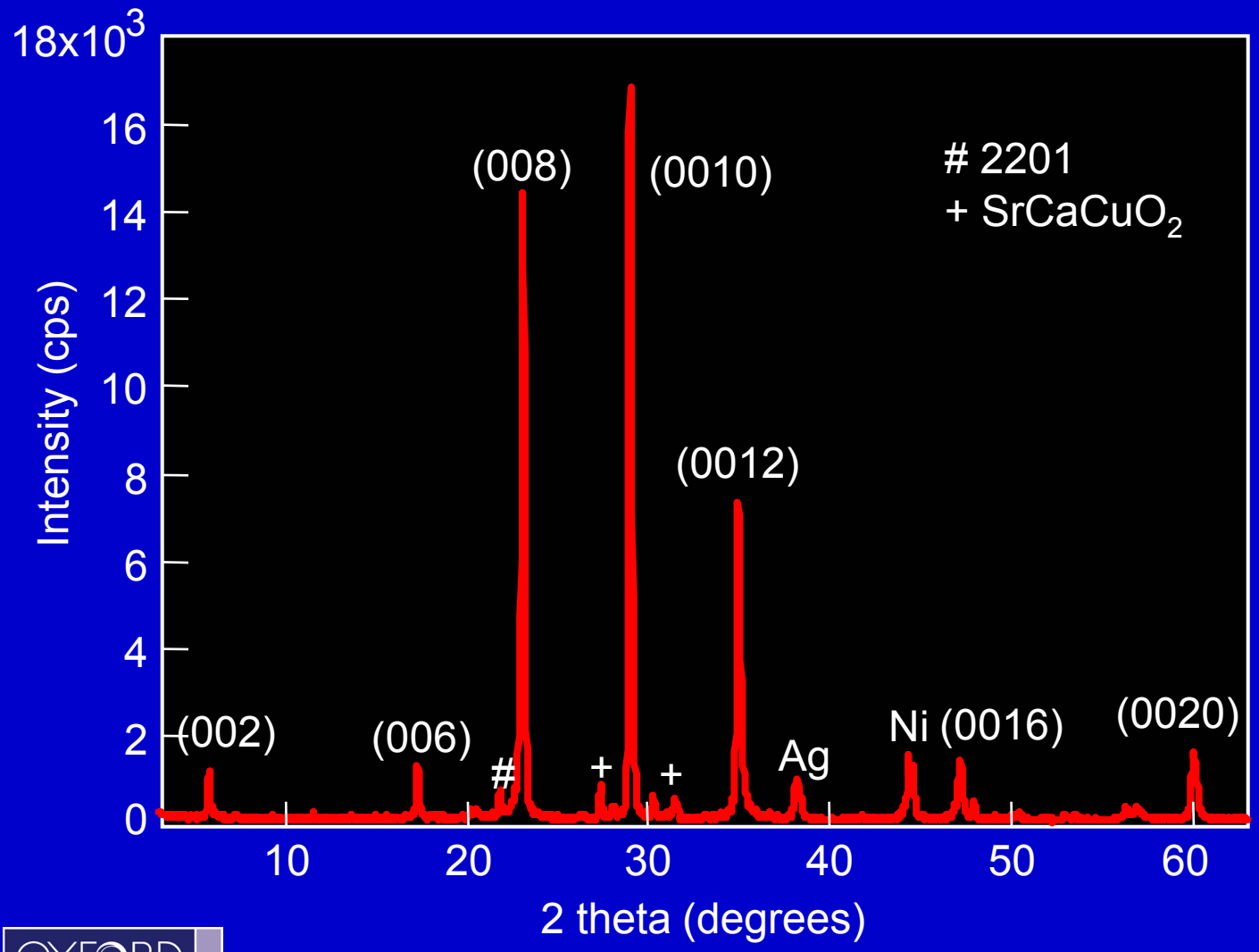
XRD of a 20 µm Thick Dip Coated Oxford Tape ($I_c \approx 700$ A at 4.2K)



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OXFORD INSTRUMENTS

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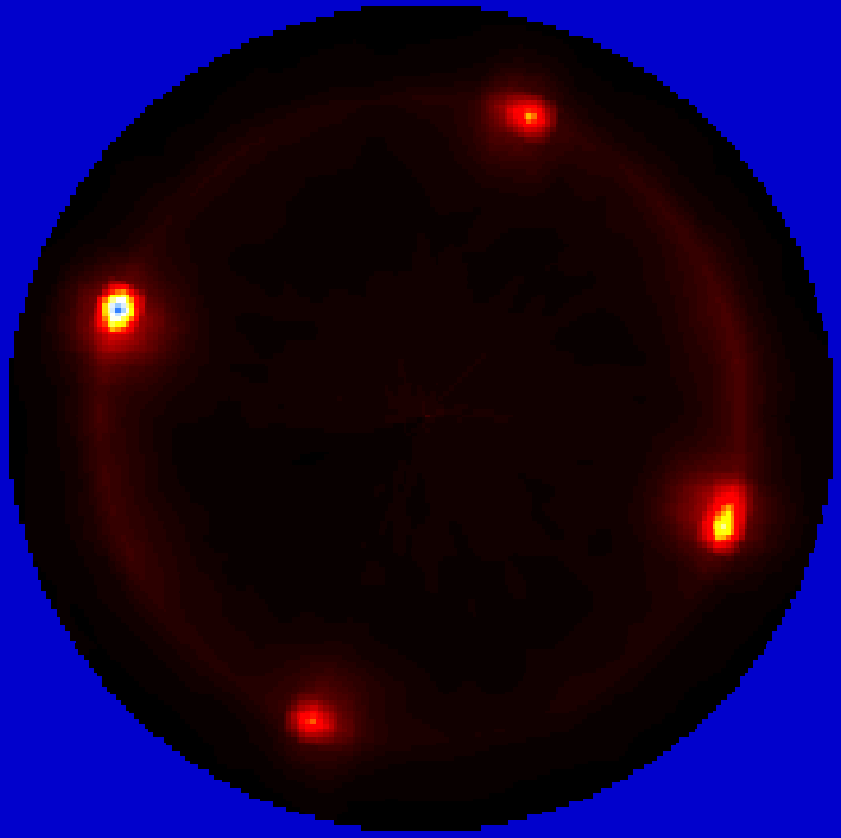
Pole-Figure Scan of Electrodeposited Bi-2212 on LAO



Description: Melt-quench/melt-growth/LAO
HKL: 105
Linear scale

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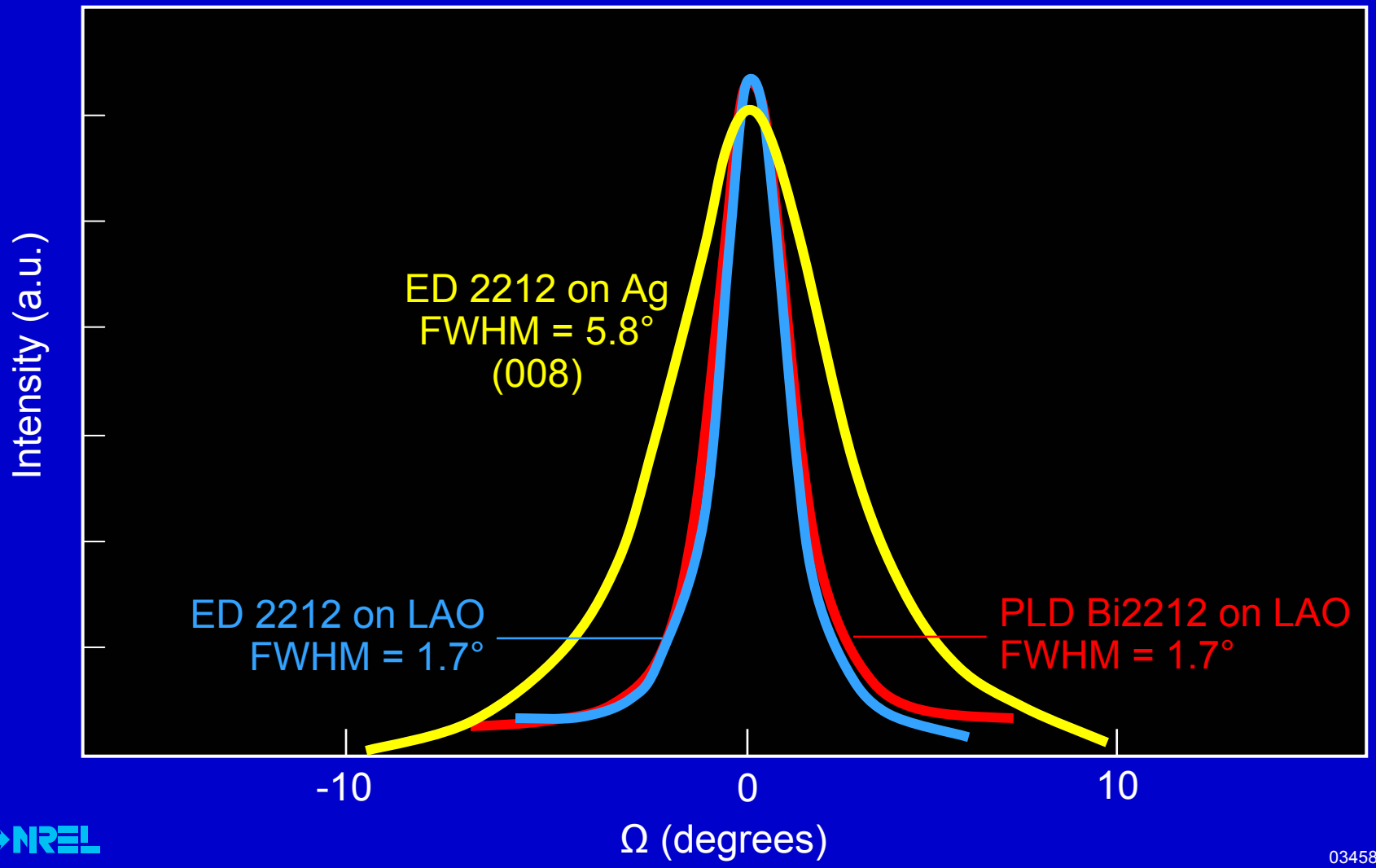
Comparison of Ω Scan for Bi-2212 Films Prepared by ED and PLD



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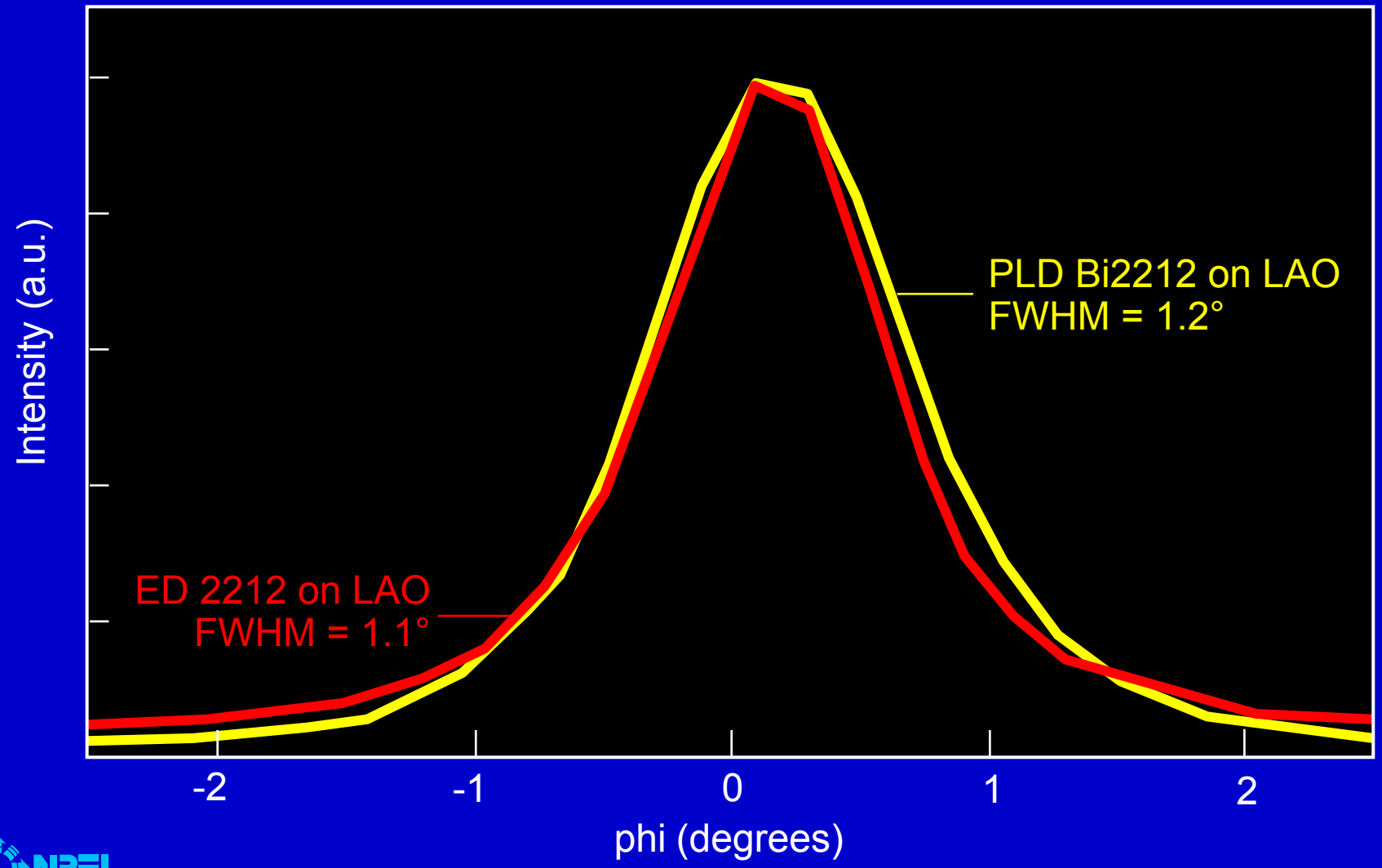
Comparison of Phi Scan for Bi-2212 Prepared by ED and PLD



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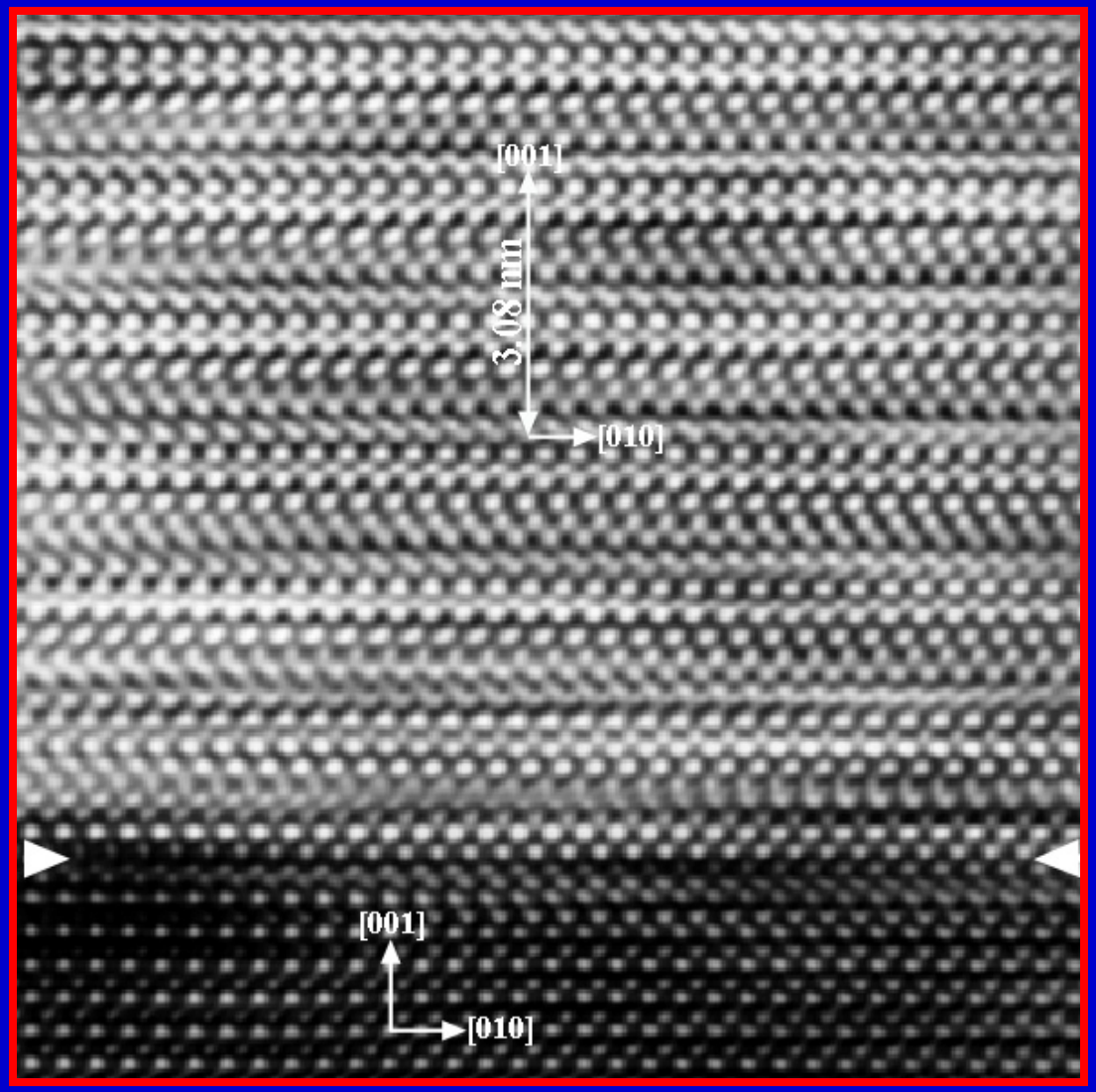
TEM Image of Electrodeposited Bi-2212 on LAO

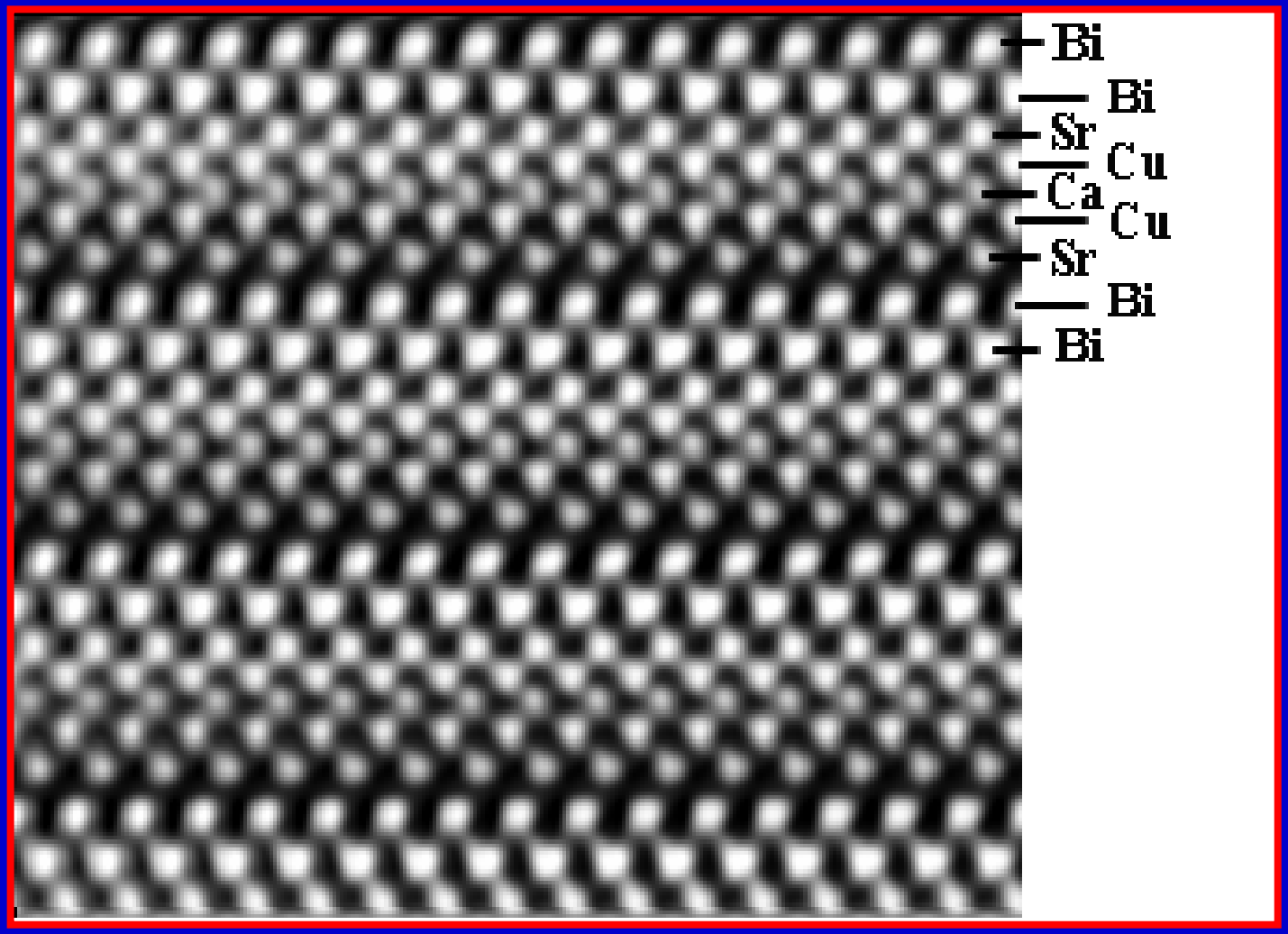


- Image was Fourier filtered to remove noise
- Arrowheads mark the interface
- Epitaxial relationship

$[001]_{LAO}//[100]_{Film}$

$(001)_{LAO}//(100)_{Film}$

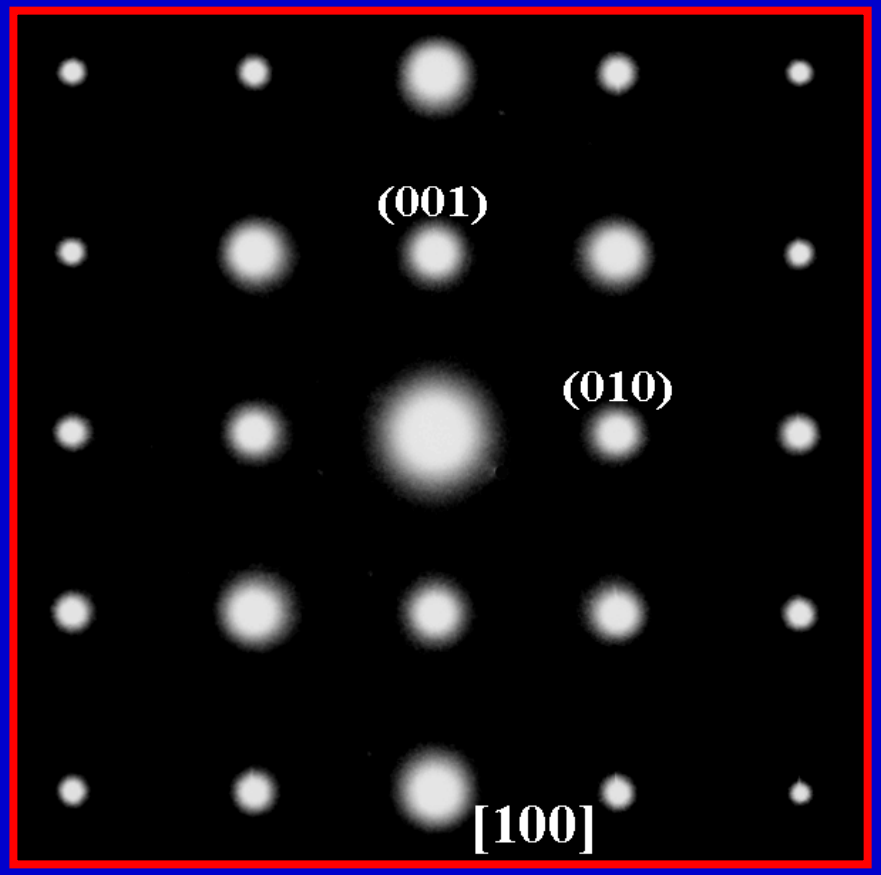
High Magnification TEM Image of Electrodeposited Bi-2212 on LAO



The bright spots correspond to the atomic columns.

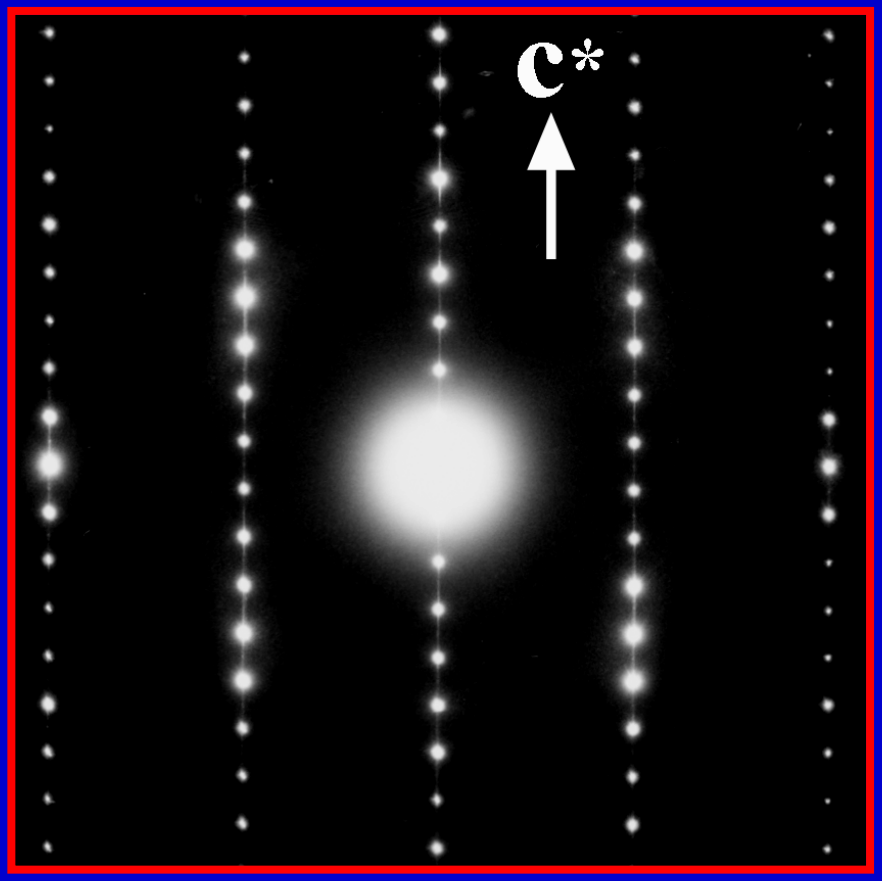
TEM Diffraction of LAO + Electrodeposited Bi-2212 on LAO

LAO [100]



Bi-2212 [100]

(The streaks along c* direction indicates stacking-faults in the c planes.)



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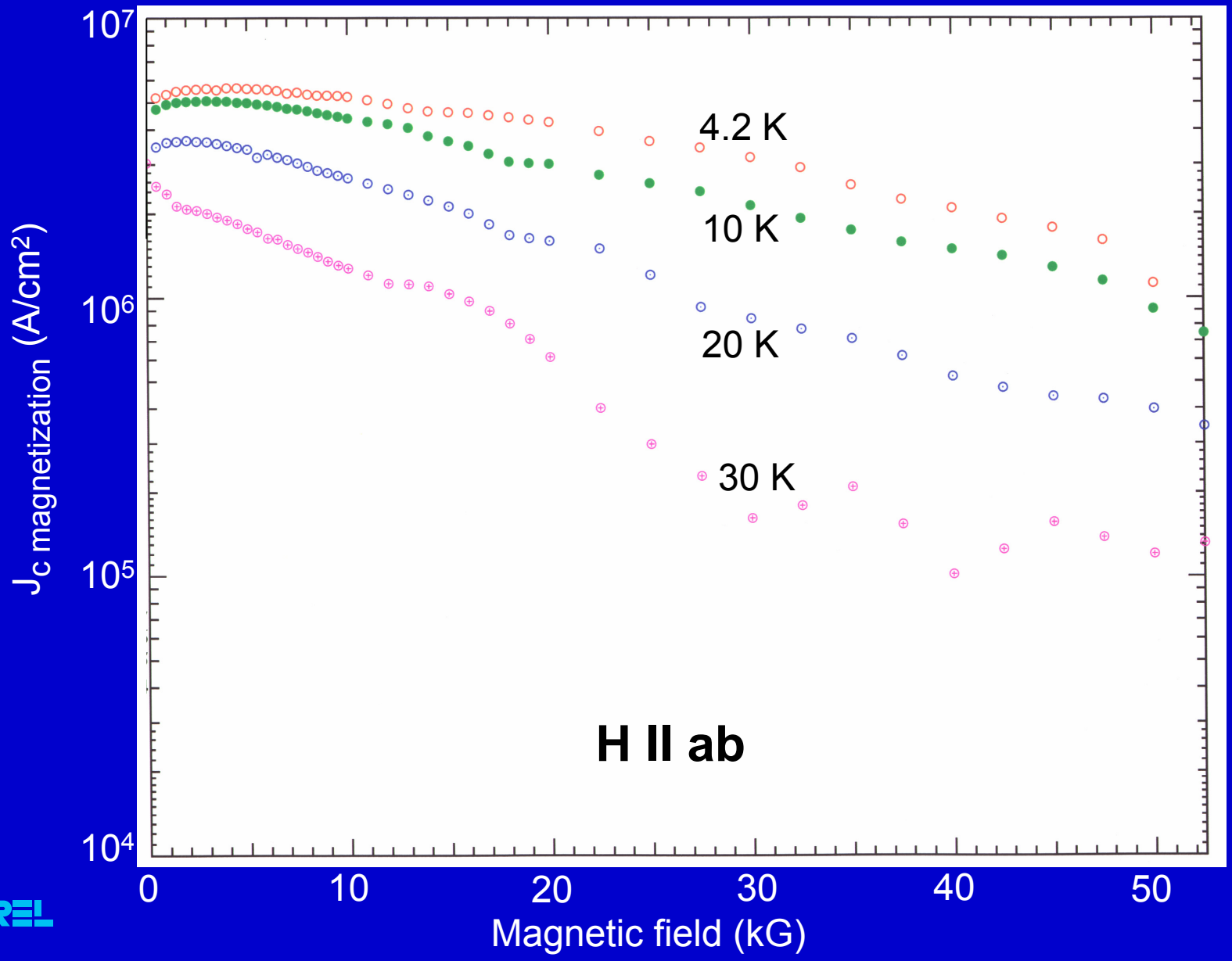
Current Density of ED Bi-2212 on LAO



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Approach

- Develop Cu stabilizer by non-vacuum electrodeposition process on YBCO coated conductor (NREL/BNL/LANL/ ORNL Collaboration).

Cu Stabilizer on YBCO Coated Conductors

- The electrodeposition of copper on electro-polished Hastelloy tapes was carried out at NREL.
- The resistivity of the copper layers were measured at BNL at room, liquid nitrogen and helium temperatures.
- Copper was deposited on Ag coated YBCO coated conductors for the determination of the contact resistance between Cu and YBCO.
- When high current conductors of sufficient lengths become available, different thickness of copper will be deposited on these conductors for the stability tests.

Electrodeposited Cu on Hastelloy Tapes

Electrodeposition Rate of Cu (μm/min)	Average Thickness (μm)	Total deposition time (minute)	RRR (N_2)	RRR (He)	Resistivity at 80 K
0.1	6	60	5.3	21.1	~3 x10^{-7}
0.25	15	60	5.7	29.1	-
0.75	45	60	6.67	53.9	~2.4x10^{-7}
1.3	69	55	7.4	50.9	~2.4x10^{-7}
1.4	57	40	7.6	52.4	~2.4x10^{-7}
2	20	10	5.64	21.2	~3 x10^{-7}
2.2	33	15	5.92	31.5	-
2.3	46	20	6.0	34.8	-
2.3	70	30	6.54	36.8	-
2.4	37	15	6.6	33.6	-
4	40	10	5.53	19.8	~3 x10^{-7}
4.5	68	15	6.02	29.9	-
4.6	23	5	5.07	14.7	-
Hastelloy Tape	NA	NA	1.05	1.07	
Cu tape	NA	NA	7	50	2.4 x 10^{-7}
Cu tape	NA	NA	5	20	3 x10^{-7}
Ag tape	NA	NA	-	100	3 x10^{-7}

NREL

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High quality Cu is defined as having RRR (He) of ~50 and RRR (N_2) ~7.

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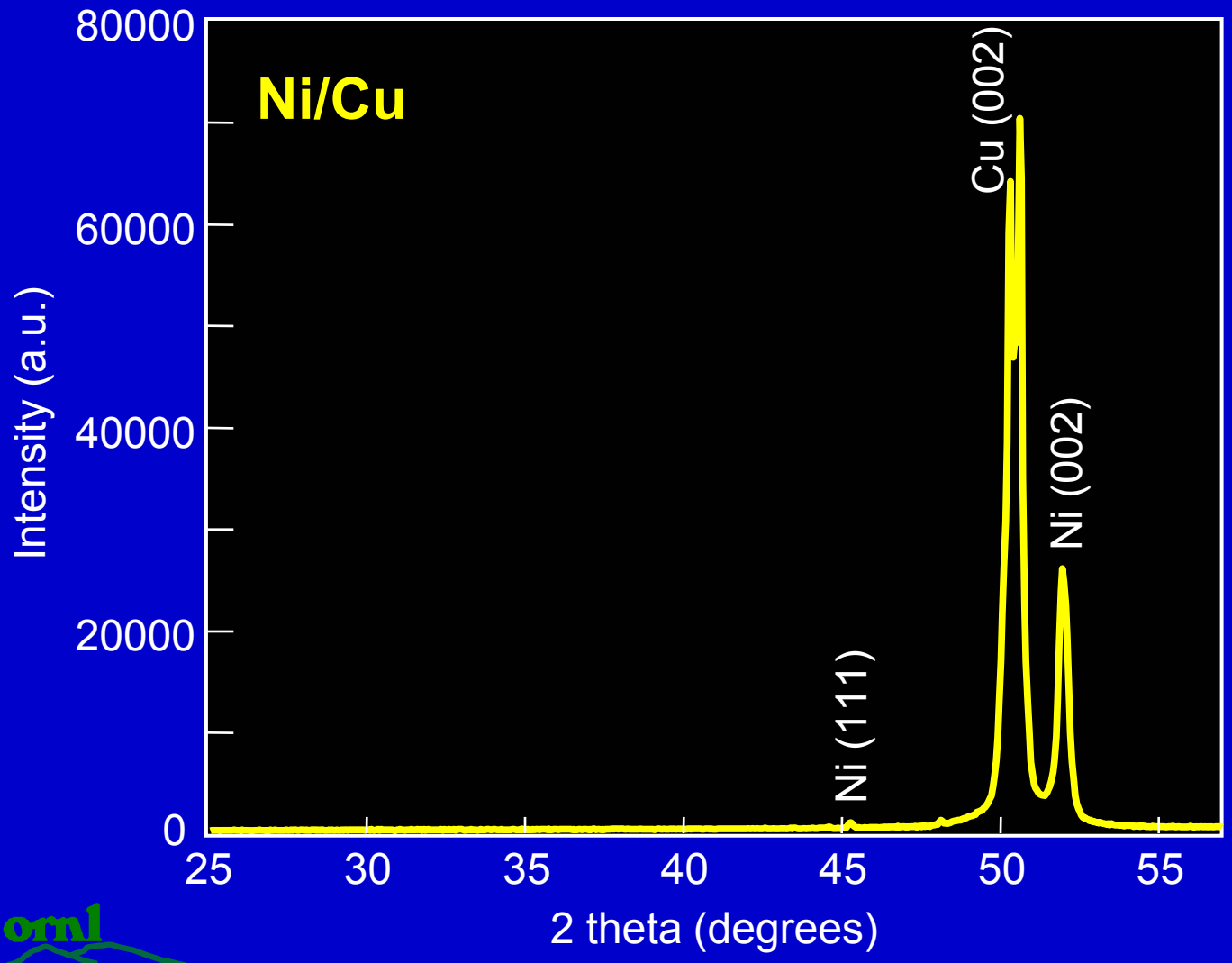
Approach

- Develop metal buffer (Ni) layer on Ni-W (paramagnetic) and Cu substrates by low-cost electrodeposition process for YBCO (NREL/ORNL Collaboration).

Theta/2 Theta Scan of as-Deposited ED-Ni on Textured Cu Tape



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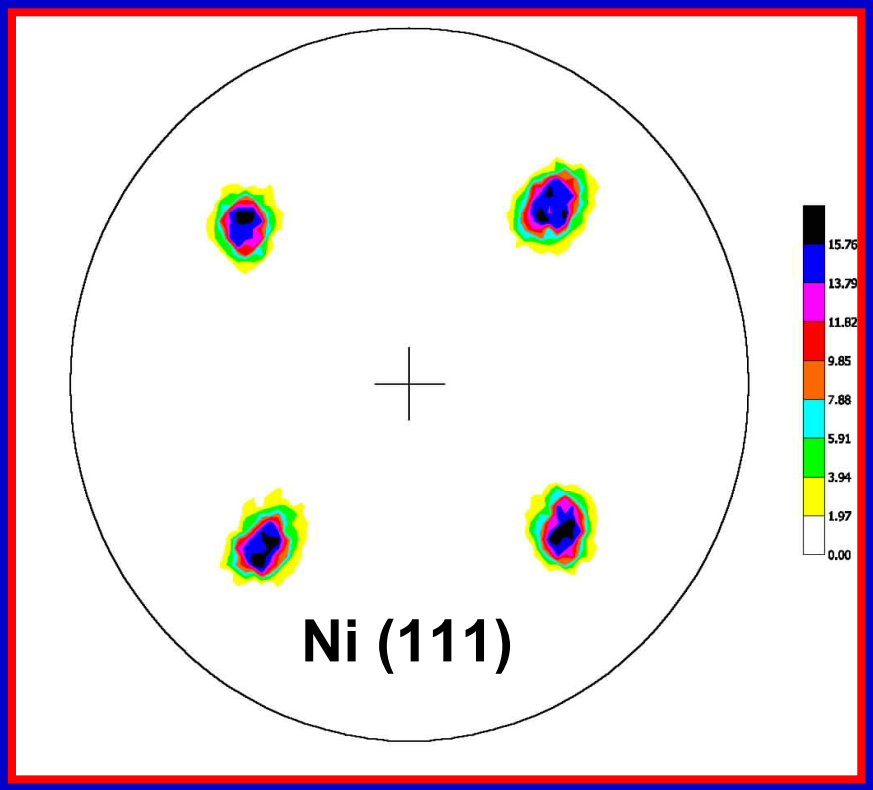
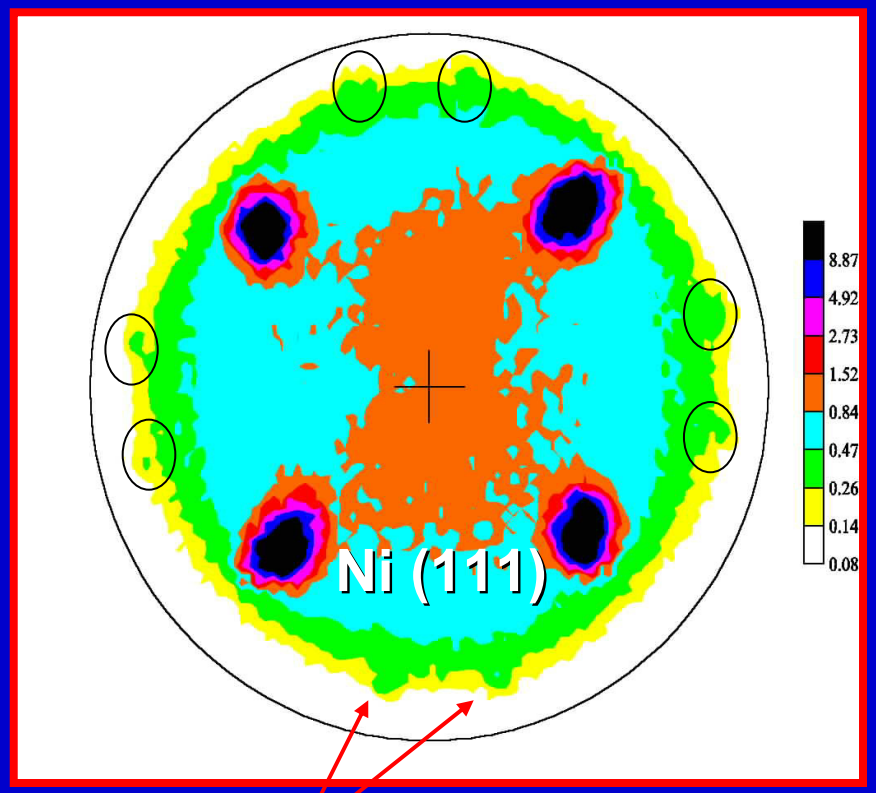
Lin and Log-scale Pole Figures of as-Deposited ED-Ni(111) on Cu Tape





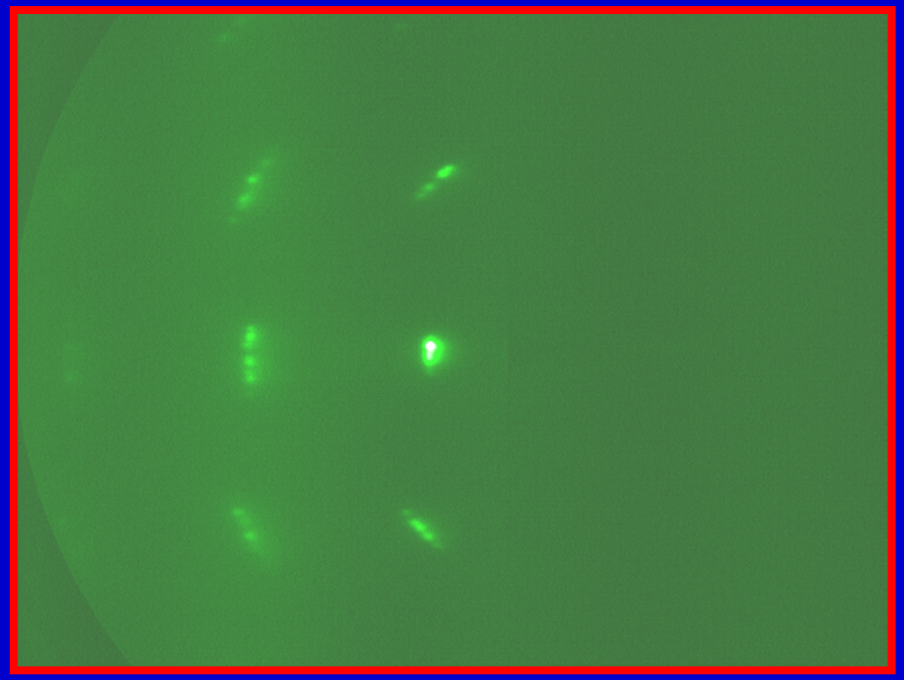
Twins in Ni

The FWHM of the Ni are very close to those of the underlying Cu tape: ϕ (111) = 7.5 degrees, Ω (out of plane) = 6.3 degrees (002).

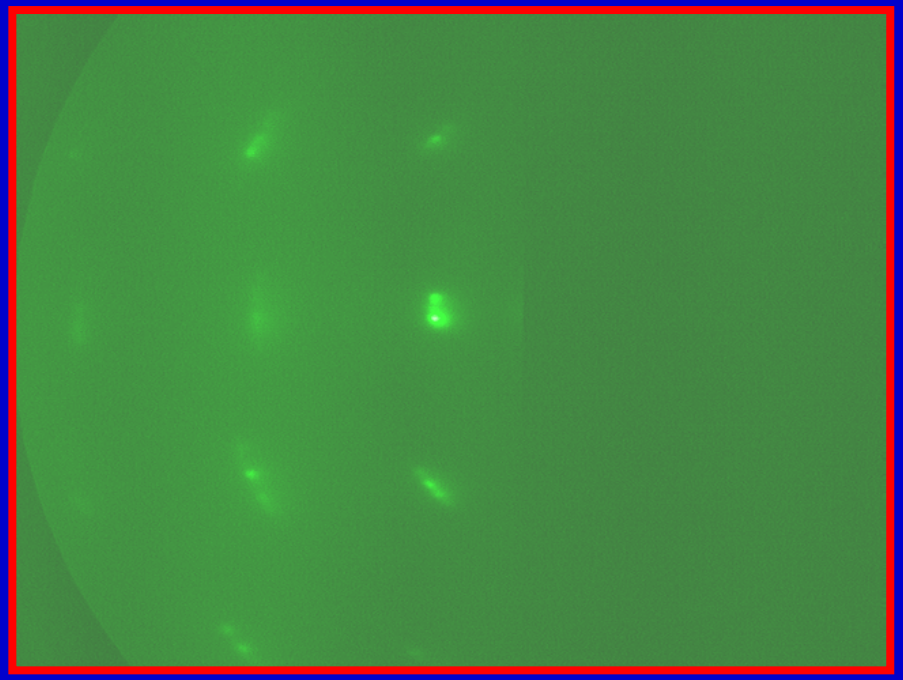
ED Nickel on Textured Copper Tape

RHEED Patterns after Holding at 355°C for 2 Hours

Smoother Sample

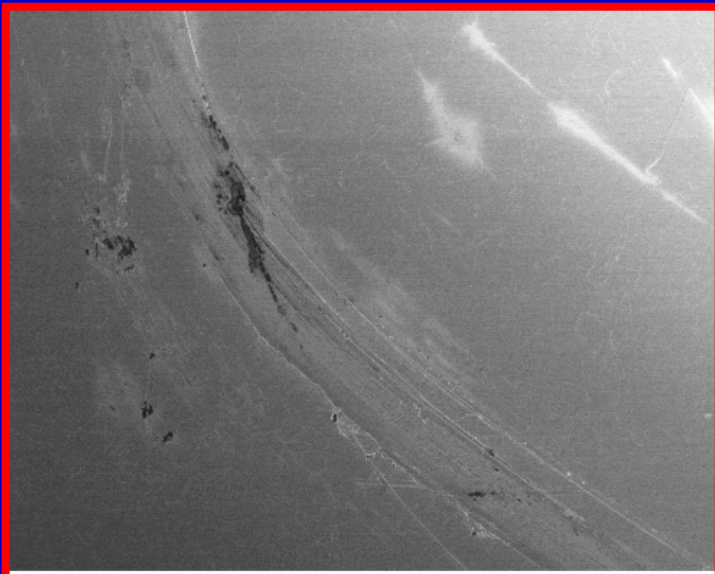


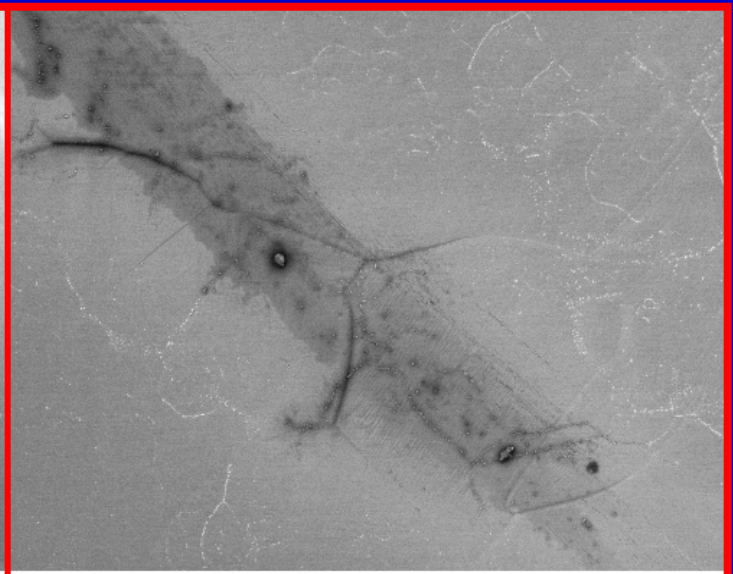
Rougher Sample

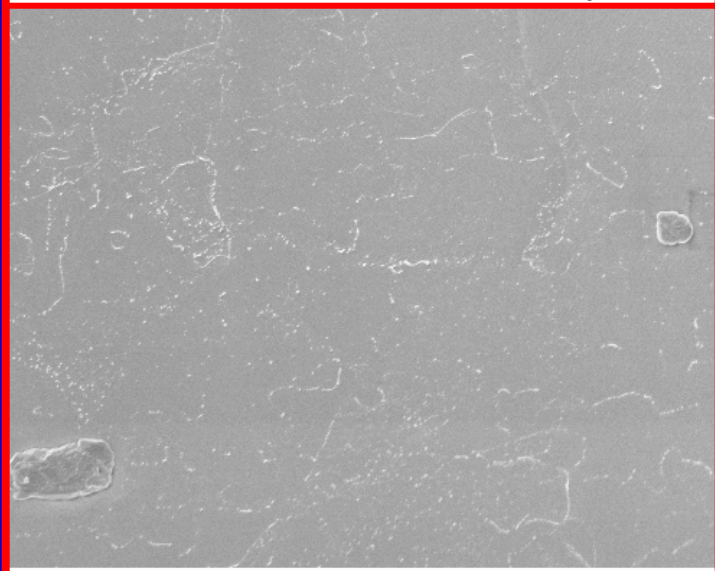


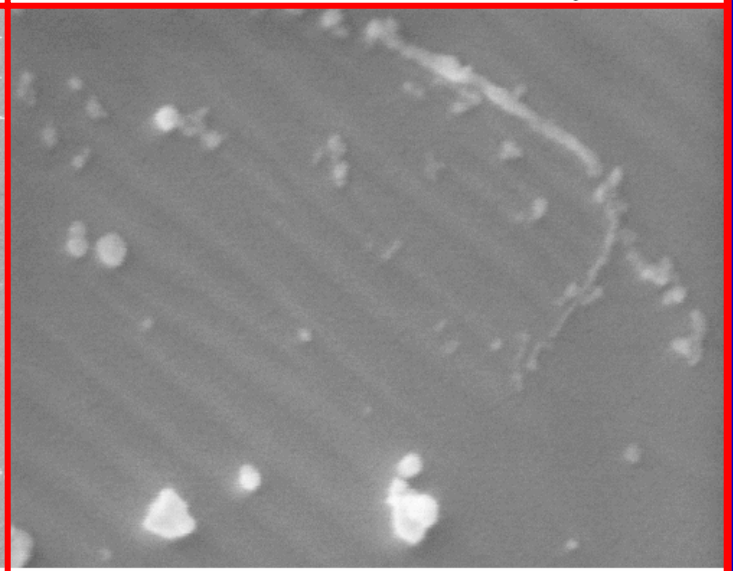
Interdiffusion begins at 400°C and is extensive at 600°C.

SEM Photograph of Ni-W Substrate









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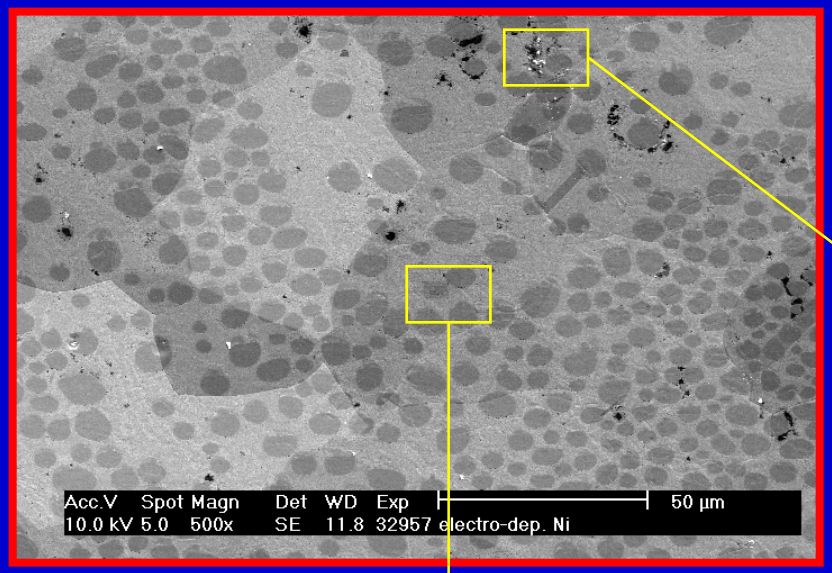
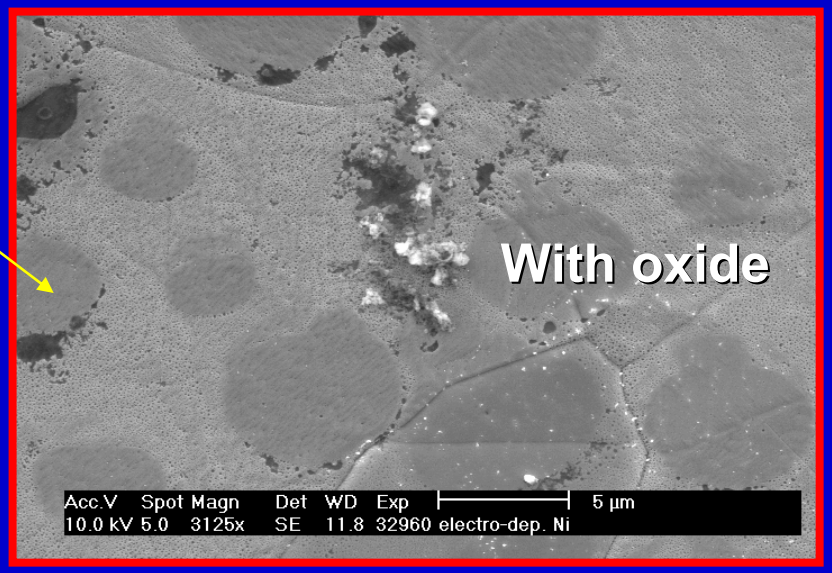
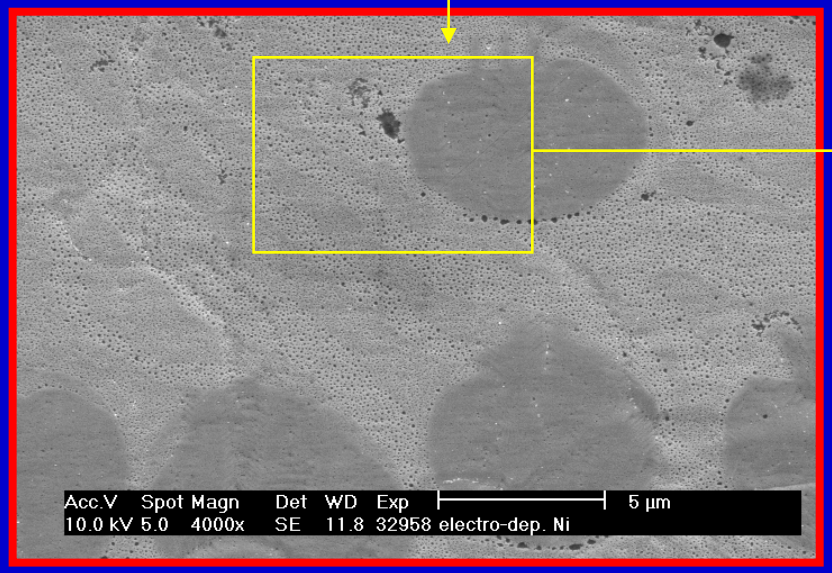
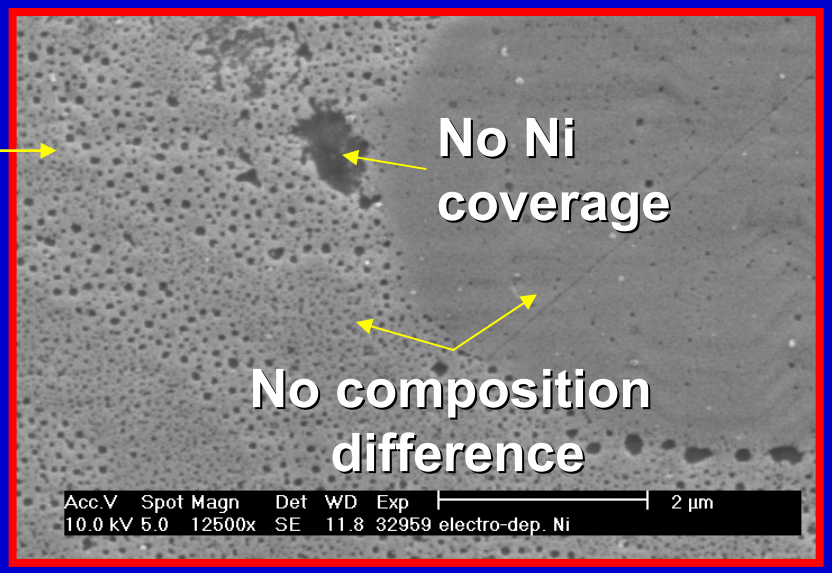
Electrodeposited Ni on Ni-W









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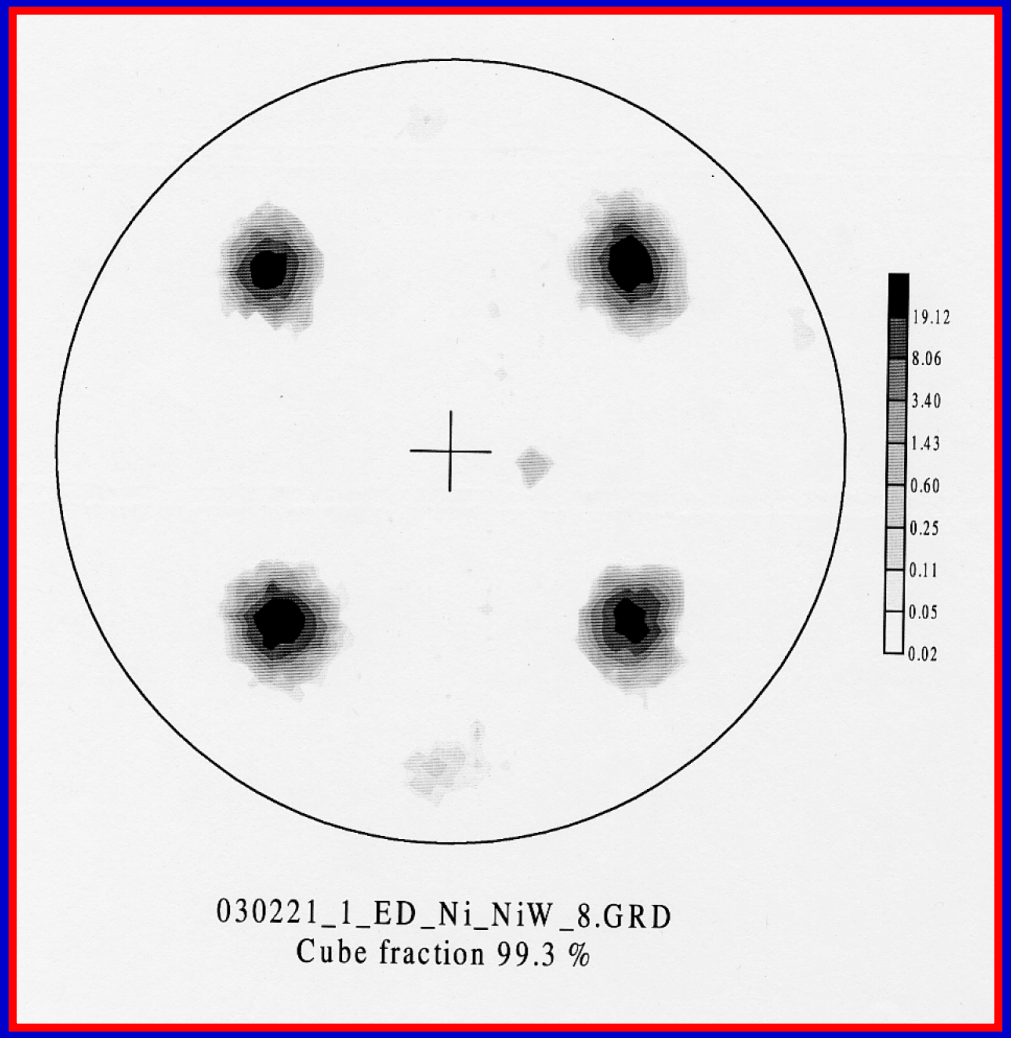
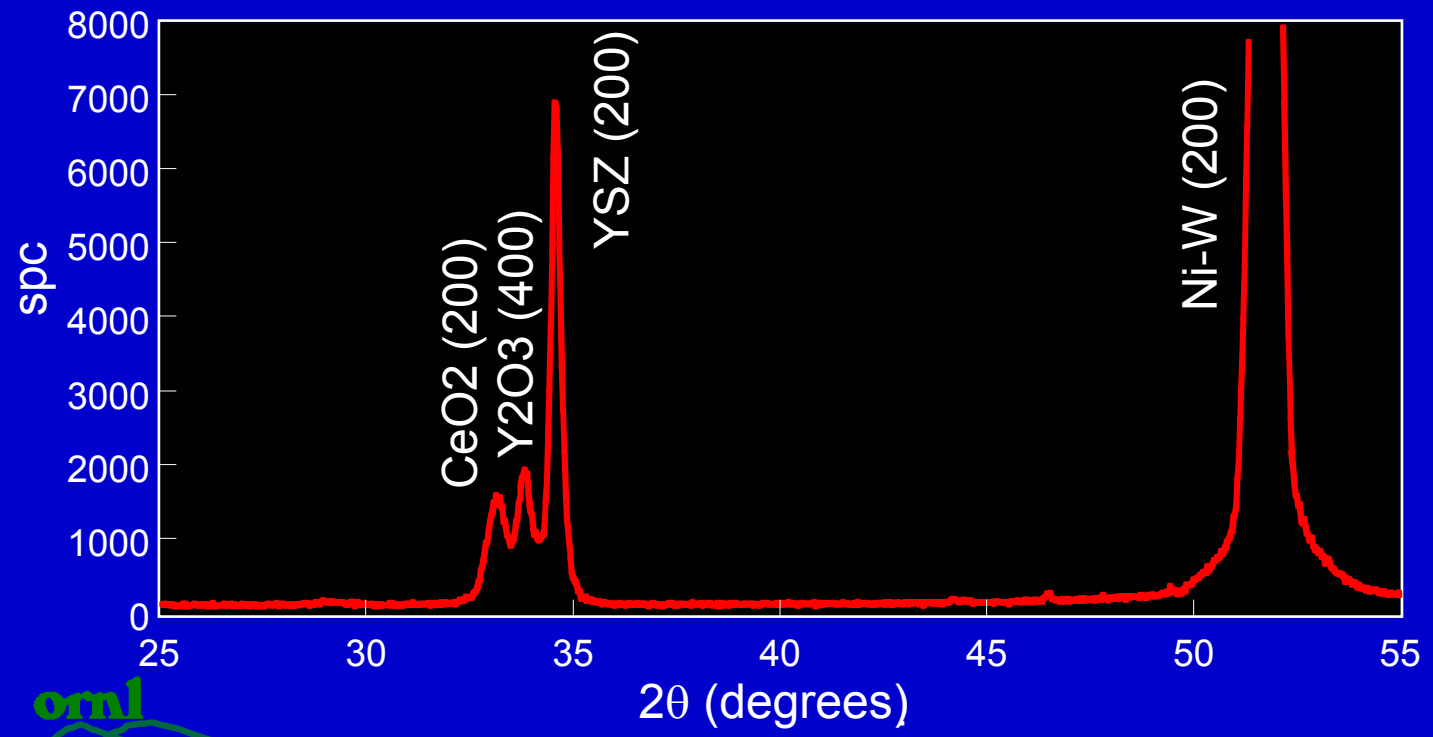
As Electrodeposited Ni on Ni-3 at.% W is 99.3% Cube-Textured



Buffer Layers were Deposited onto the ED Ni to Complete the RABiTS Architecture

- CeO_2 (~20 nm) /YSZ (~150 nm) / Y_2O_3 (~20 nm) / Ni (ED, ~1 μm) / Ni-3 at.% W (50 μm)
- Short stationary samples:
  - Y_2O_3 by e-beam evaporation
  - YSZ and CeO_2 by rf sputtering



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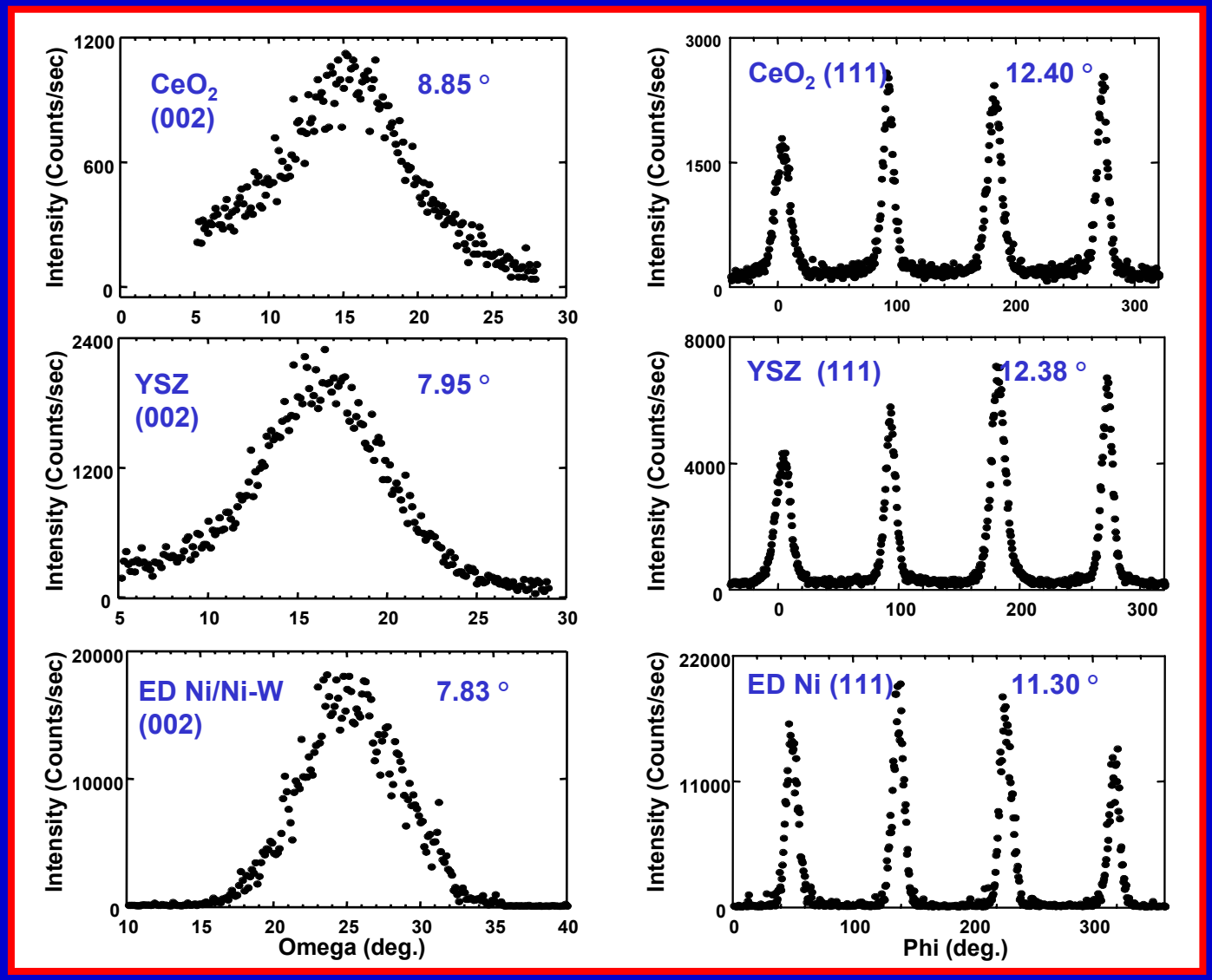
Epitaxial Buffer Layers were Obtained on the Electrodeposited Ni Surface



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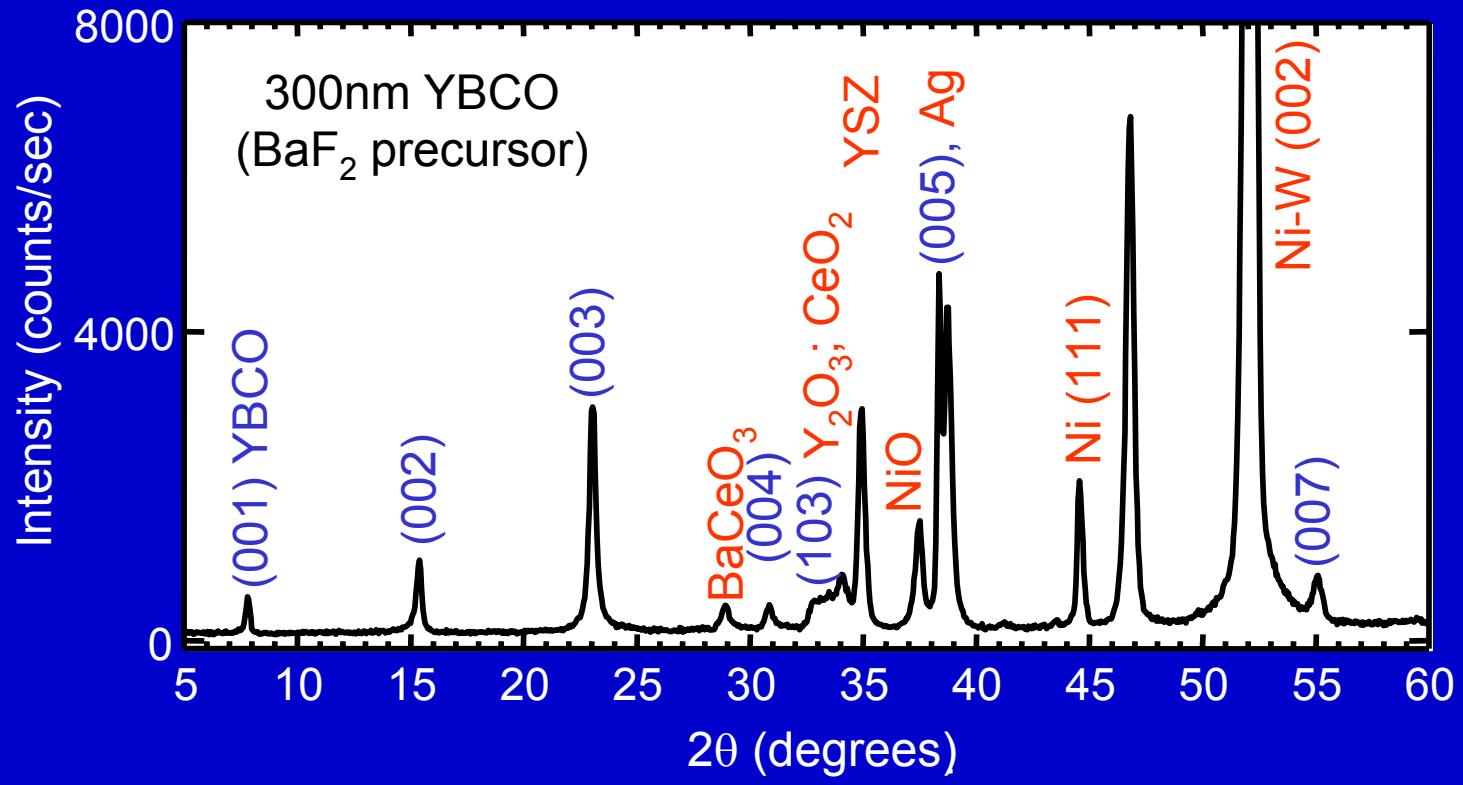
YBCO with Respectable J_c's were Obtained on these Experimental Substrates



- 200nm YBCO by PLD:
  - J_c ~200 kA/cm^2
- 300nm YBCO by "BaF_2" process:
  - J_c ~250 kA/cm^2

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2003 Annual Superconductivity Peer Review

Kenneth Marken

OXFORD INSTRUMENTS

Oxford Superconducting Technology (OST)

OST – NREL CRADA

- CRADA tasks support conductor research and development for OST's SPI project for HTS MRI.
- The SPI project is aimed at demonstrating technical and economic feasibility of HTS MRI, primarily through demonstration of lowest price 2212 conductor.
- Coated tape has the lowest labor required, and potentially the lowest silver content, of the various possible conductor configurations.
- OST's dip coated tape provides the starting point.
- NREL is tasked with performance improvements, and potential alternate coating methods.

NREL CRADA Task Summary

- NREL is working on possible methods of biaxially texturing the 2212, which has been shown to increase J_c by an order of magnitude.
- NREL is working with OST tapes, looking at alternate heat treatment methods for possible performance improvements.
- NREL is also looking at possible pinning enhancements through nanoparticle additions to existing OST tapes.
- NREL is also exploring alternatives to dip coating that may provide performance or cost advantages.

OST CRADA Task Summary

- OST is supplying precursor powders for DTA analysis, nanoparticle additions, and NREL processing.
- OST is supplying coated tapes for NREL heat treatment studies, and additional surface coating of additions.
- OST is heat treating experimental coatings done at NREL, and nanoparticle additions done at NREL.
- OST is evaluating J_c in the NREL processed tapes, including experimental coatings, experimental heat treatments, and coating modifications (nanoparticles).

OST I_c Measurements in NREL Processed Samples

Sample ID	4.2K I_c (A) 1 μV/cm	n-value	77K I_c (A) 1 μV/cm	n-value
1 (OXB-0519-2)	18.9	3.9	0.8	
2 (OXB-0521-1)	27.6	7.1	0.9	
3 (OXB-0522-1)	23	7.6	0.6	
4 (OXB-0520-2)	7.4	2.6	0	
5 (OXB-0523-1)	113.5	10.1	3.0	5.2
6 (OXB-0527-1)	27.5	7.8	0.7	
7 (OXB-0528-1)	11.8	5.0	0	
8 (OXB-0524-1)	11.4	4.5	0	

NREL OXFORD INSTRUMENTS

OST I_c Measurements in NREL Nanoparticle Added Samples

- Samples heat treated at OST
- Melt temperature not yet optimized
- DTA shows melting point changed by 5–6°C
- Optimization of heat treatment with MgO is underway

Heat Treatment Identification	Samples	4.2K Ic (A) 1 μ V/cm	n-value
HTC12-030620	1 (no additions)	334.8	8.3
T_{max} = 887.1	2 (no additions)	365.9	10.8
	3 (YSZ nanoparticle)	<1	
	4 (MgO nanoparticle)	155.5	7.8
HTC12-030707	1 (no additions)	616.7	16.7
T_{max} = 886.1	2 (no additions)	715.2	21.5
	3 (YSZ nanoparticle)	<1	
	4 (MgO nanoparticle)	484.2	12.5

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2003 Annual Superconductivity Peer Review

David Christen

ornl
UT-BATTELLE

Oak Ridge National Laboratory (ORNL)

Electrodeposited Metallic Seed Layers for Copper-based Coated Conductors

D.K. Christen, T. Aytug, C. Cantoni, A. Gapud, A. Goyal, K-H. Kim, D.F. Lee, E. Miller, M. Paranthaman, N. Rutter, J.R. Thompson, H-Y. Zhai

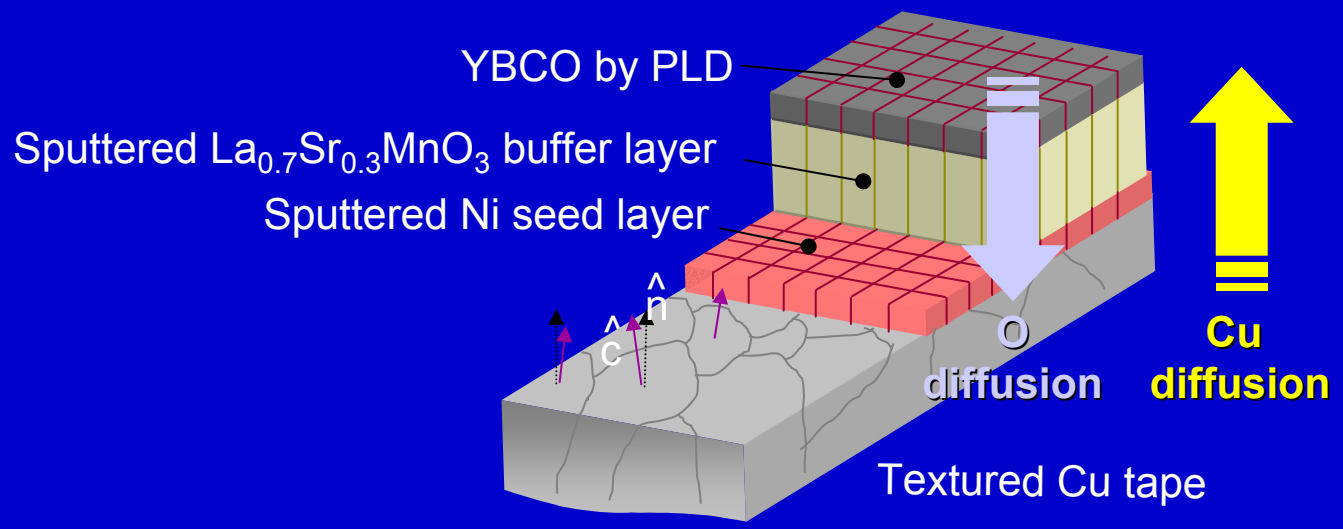
R. Bhattacharya, P. Spagnol, J. Chen

Motivation

- Epitaxial electrodeposition of metal layers has been demonstrated previously
- Copper-based tapes have potential advantages
  - Non-magnetic
  - Highly textured for RABiTS
  - Inexpensive compared to Ni-alloys
- ORNL has developed an operational buffer-layer architecture for PLD-deposited YBCO CC

Motivation

- Characteristics/properties of ORNL prototypes

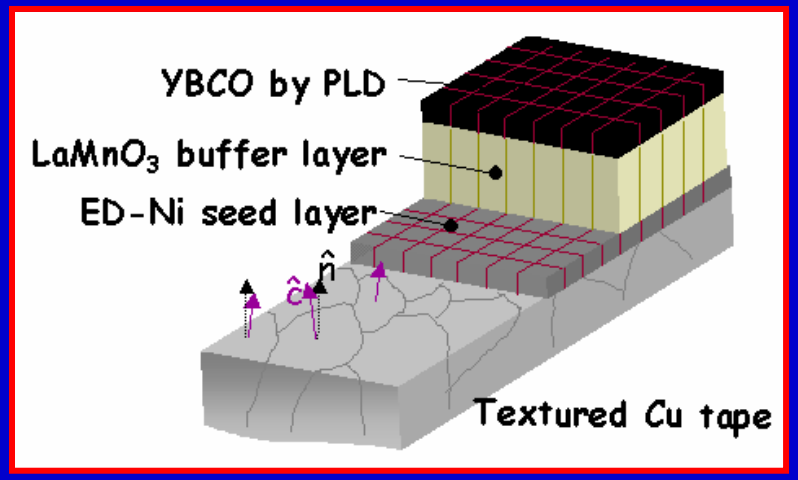


- Slow oxidation of Ni (1.5 μm thick) at Ni-buffer interface protects Cu during YBCO deposition (750°C in 100–200 mTorr O_2)
- Epitaxial LSMO (0.2 μm thick) buffer layer excellent for YBCO by PLD → J_c = 2.3 MA/cm^2 at 77K, self-field

→ *and, partial electrical coupling to Cu*

Objective

Simplify and improve efficiency by developing ED metallic seed layers



Potential Advantages

- Inexpensive, controllable source materials
- Nearly 100% utilization
- Rapid deposition
- Epitaxy at room temperature
- Easily scalable reel-to-reel

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Approach

- NREL deposits Ni or other operational metallic seed layer by ED on ORNL textured copper substrates
- ORNL completes and qualifies structure; comparison to vapor deposited CC

Milestones

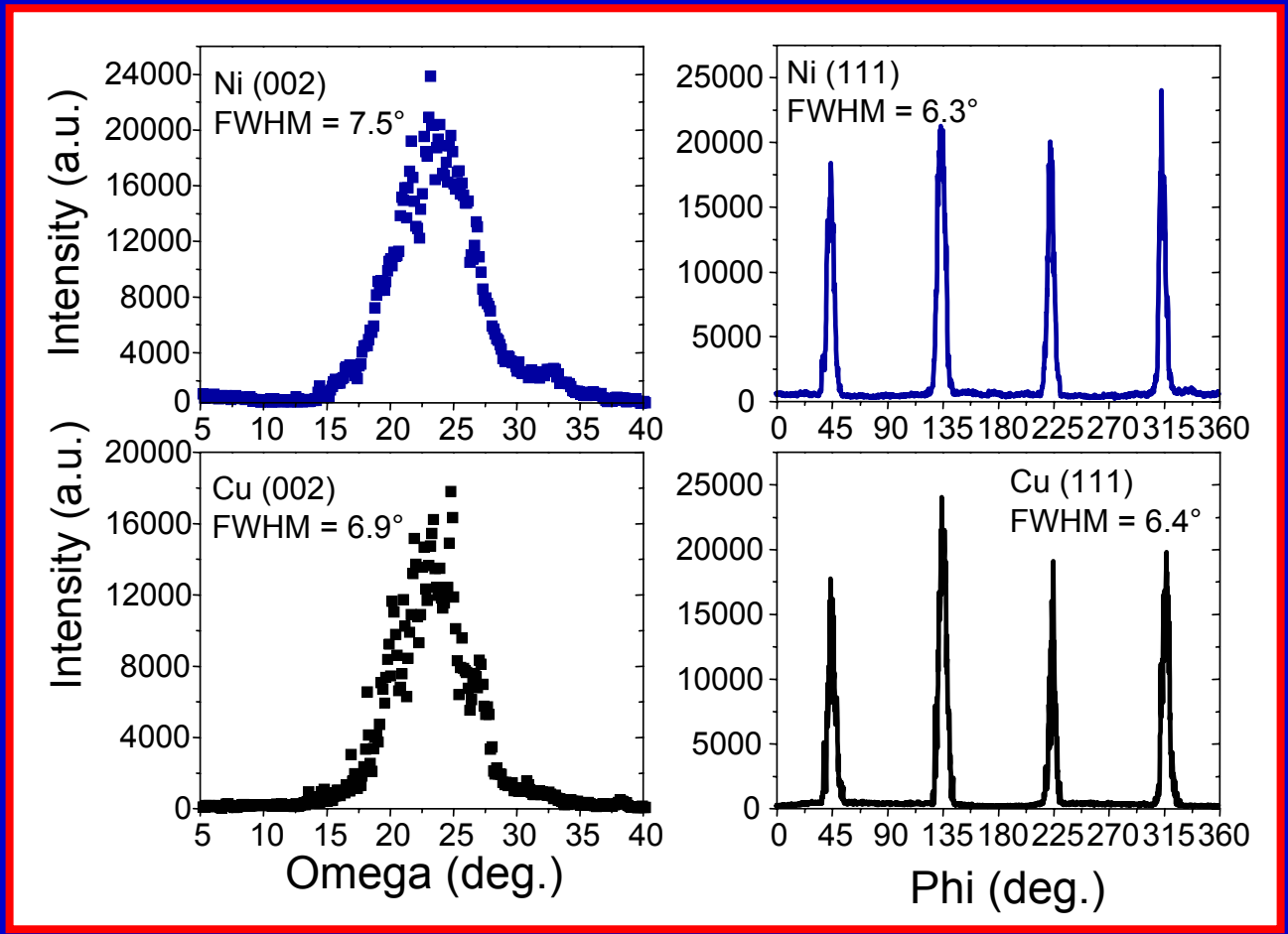
Synthesize and characterize a series of samples for:

- Epitaxy, structure, and morphology
- Magnetic and superconducting properties

Progress

- Epitaxy of NREL ED-Ni seed layers on textured Cu tapes

Cube-on-cube epitaxy; $\Delta a/a = 2.2\%$
~4.5 μm thick ED-Ni
Out-of-plane and in-plane orientation of ED-Ni on Cu comparable to Cu texture



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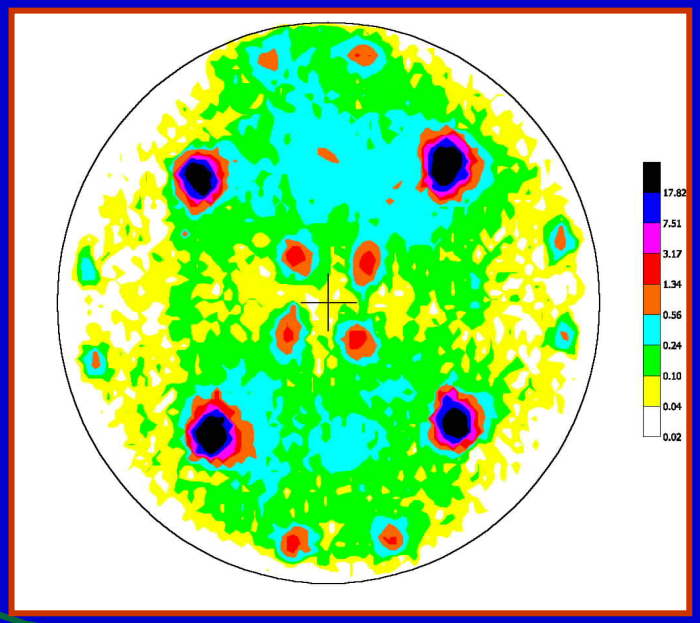
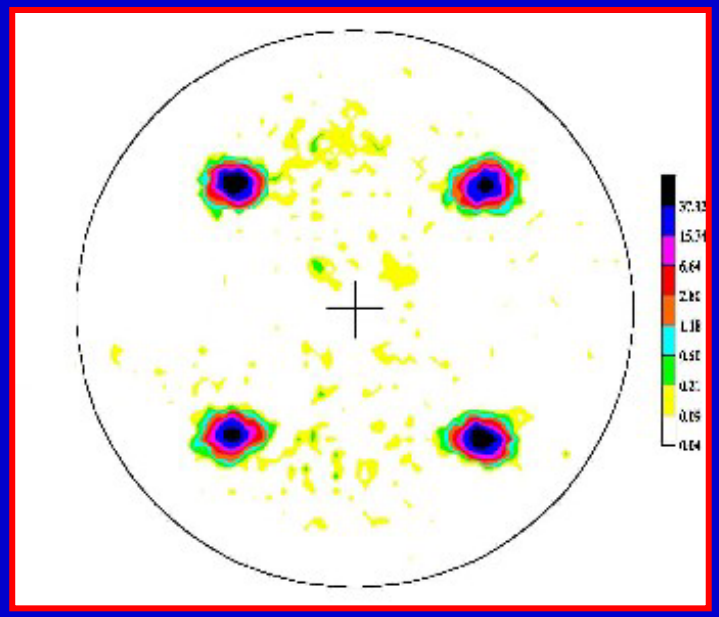
Progress

- Epitaxy of NREL ED-Ni compared to ORNL sputtered Ni seed layers on textured Cu tapes

~4.5 μm ED Ni
T_{dep} = ambient
85% cube texture

1.6 μm sputtered Ni
T_{dep} = 600°C
97% cube texture





$\Delta\omega$ = 7.5°
$\Delta\phi$ = 6.3°

(111) pole figures
As-deposited

$\Delta\omega$ = 5.8°
$\Delta\phi$ = 6.8°

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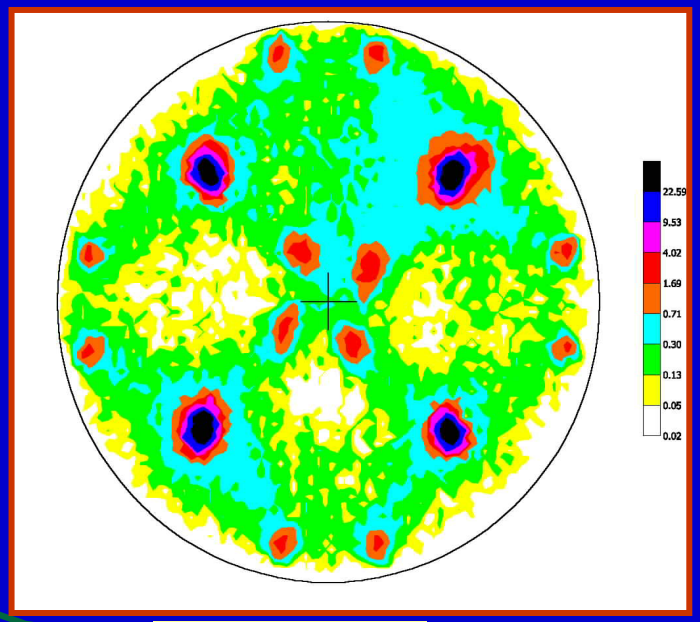
Progress

- Epitaxy of NREL ED-Ni compared to ORNL sputtered Ni seed layers on textured Cu tapes

~4.5 μm ED Ni

T_{anneal} = 600°C

85% cube texture



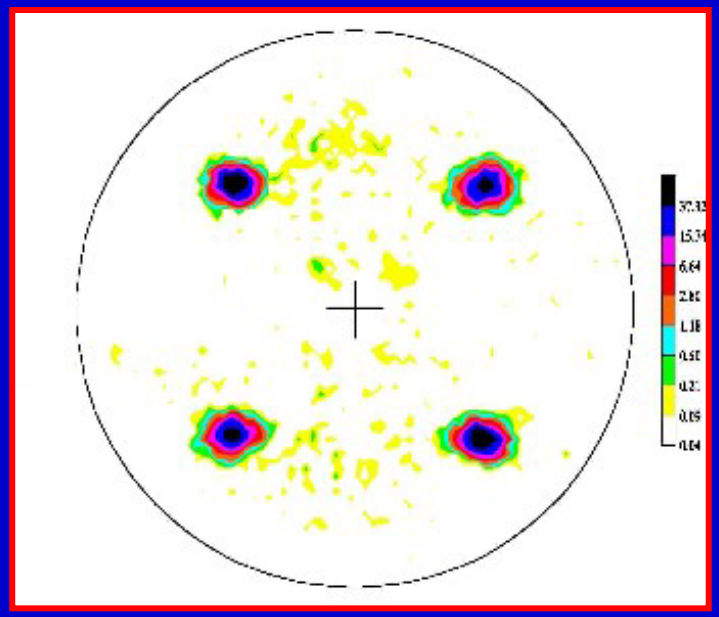
$\Delta\omega$ = 5.4°

$\Delta\phi$ = 6.4°

1.6 μm sputtered Ni

T_{dep} = 600°C

97% cube texture



(111) pole figures

$\Delta\omega$ = 5.8°

$\Delta\phi$ = 6.8°

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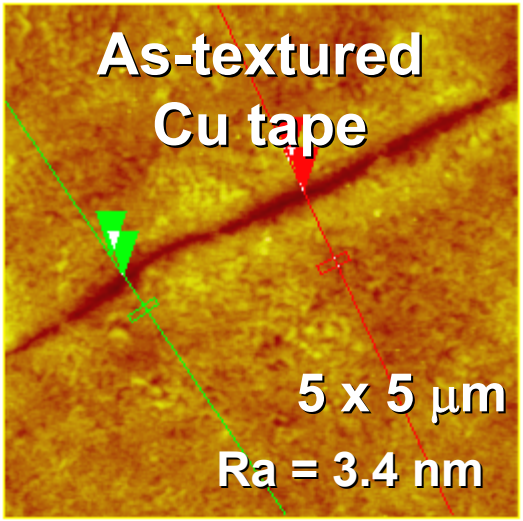
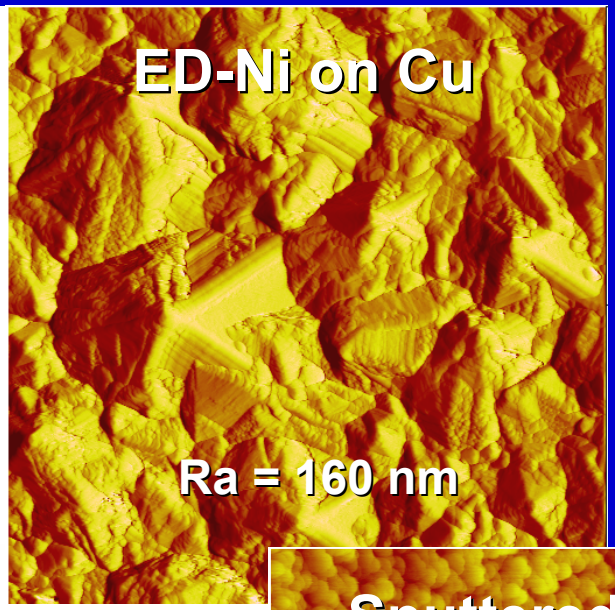
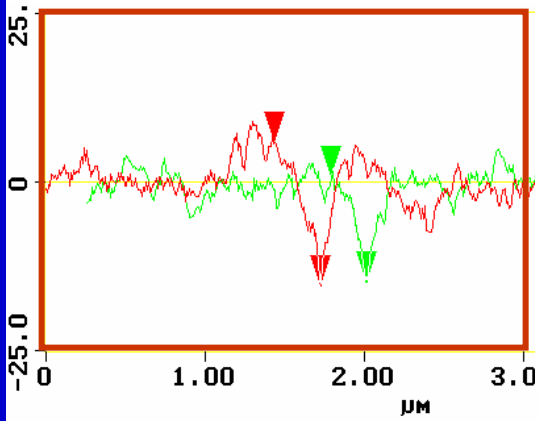
Progress

- Surface morphology/structure of 4.5 µm NREL ED-Ni seed layers

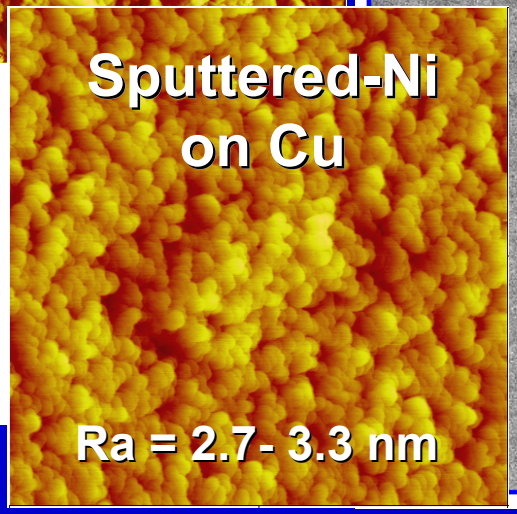
AFM Topographs

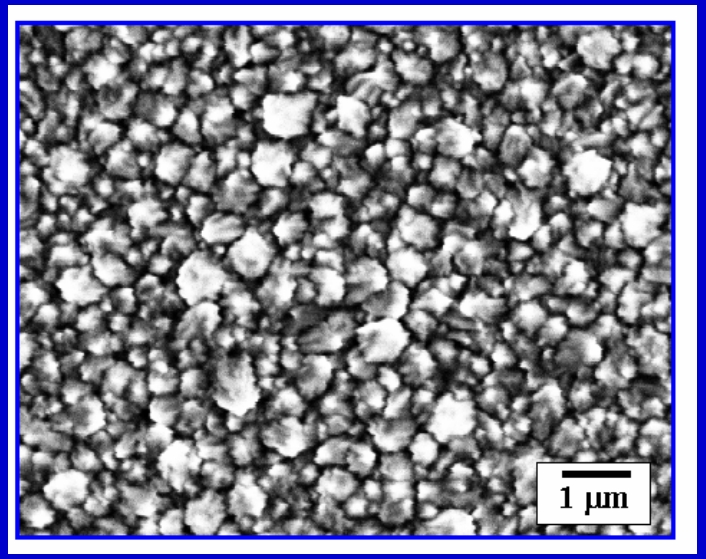




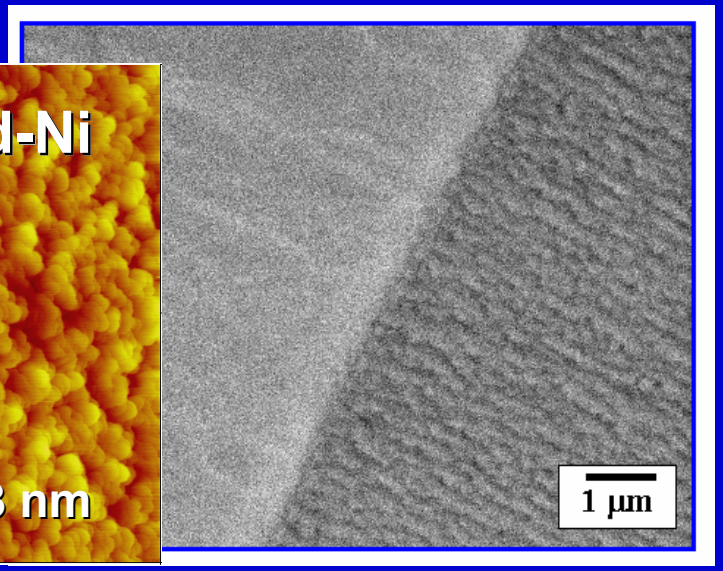


GB depth:
Red = 22 nm
Green = 16 nm





SEM Micrographs



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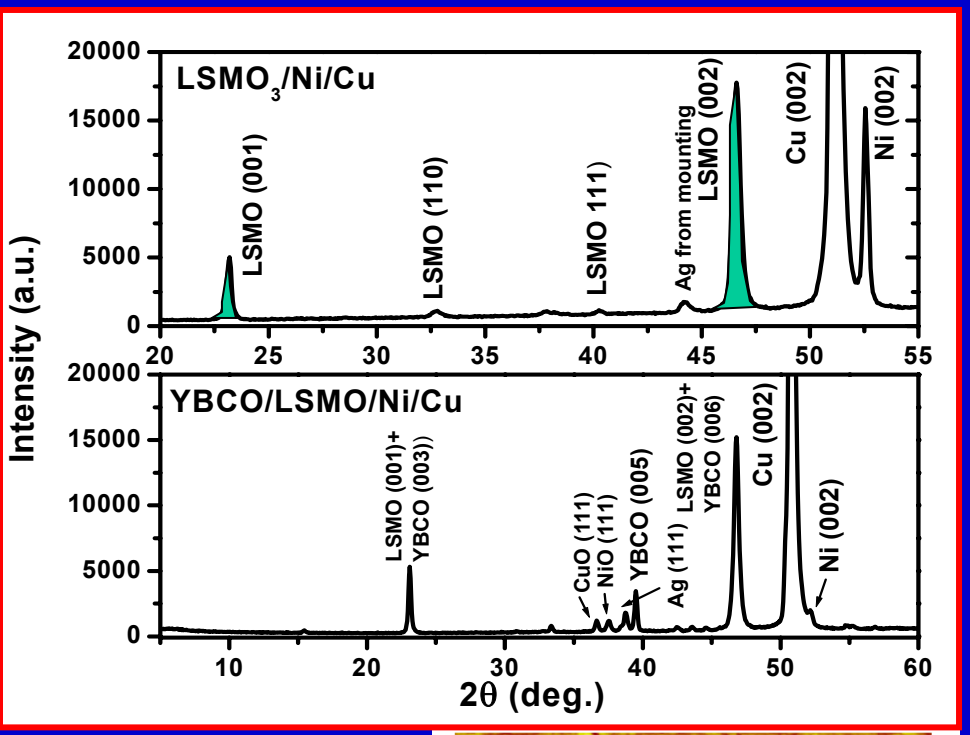
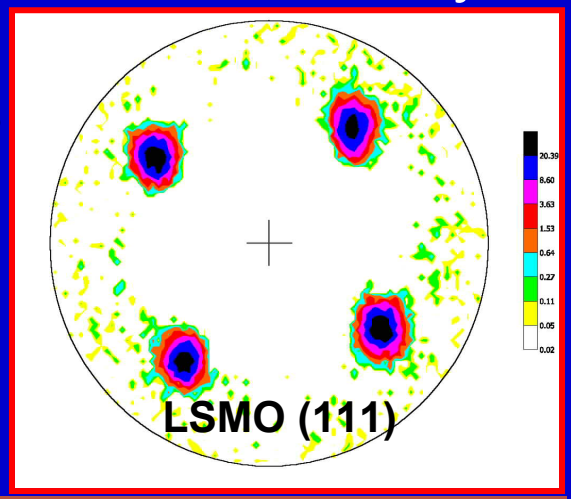
Progress

- Epitaxial orientation and properties of ORNL buffer and YBCO on 0.5 μm ED-Ni seed layer



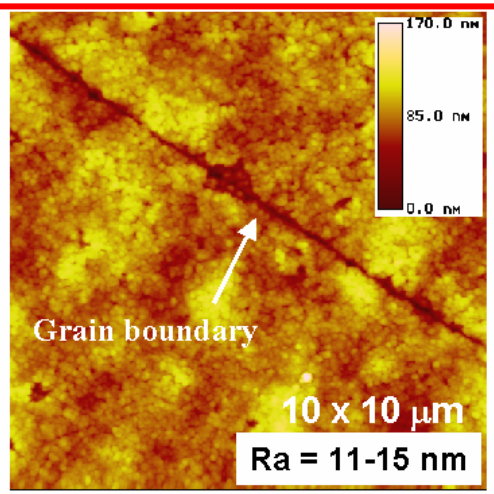
$LaSrMnO_3$
98% cube
$\Delta\omega = 10.3^\circ$
$\Delta\phi = 9.4^\circ$



ED-Ni seed layer: 0.5μm
LSMO thickness: 300 nm
YBCO thickness: 200 nm
T_c = 91 K (no contamination?!)
J_c(77K, sf) = 100 kA/cm^2

Superconducting properties

Problem: surface roughness of LSMO buffer layer



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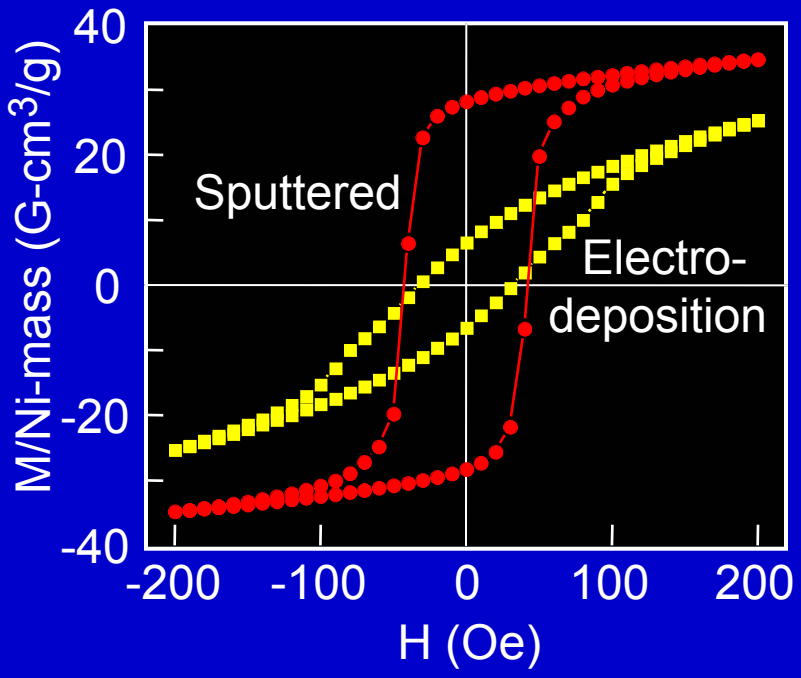
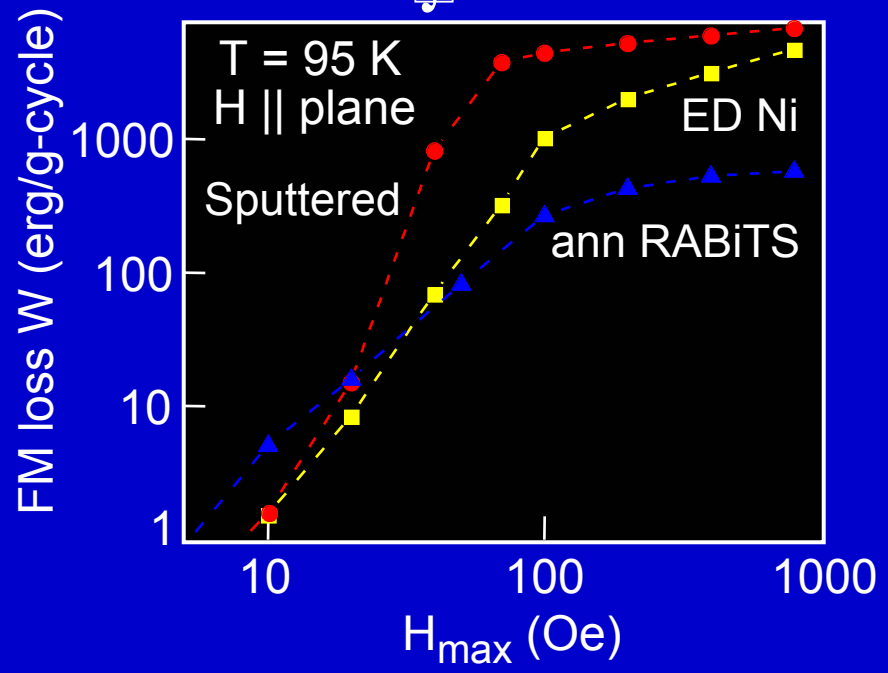
Progress

- Magnetic hysteresis of NREL 0.5 μm ED-Ni seed layers

Ferromagnetic Energy loss/cycle: $W = \oint m \cdot dH$





- Implied AC losses for envisaged operational CC tape

e.g., conductor @ 60 Hz AC operation

1.5μm ED-Ni layer; I_c = 200 A/cm-width

⇒ **max. FM tape loss: ~ 30 mW/kA-m**

NREL

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Recap

- Ni seed layers can enable high-J_c YBCO coatings on textured Cu substrate tapes (ORNL demo)
- Feasibilty of using NREL electrodeposited Ni seed layers has been assessed

Good epitaxy of ED-Ni on Cu at room temperature

ED-Ni seed layers show increased surface roughness

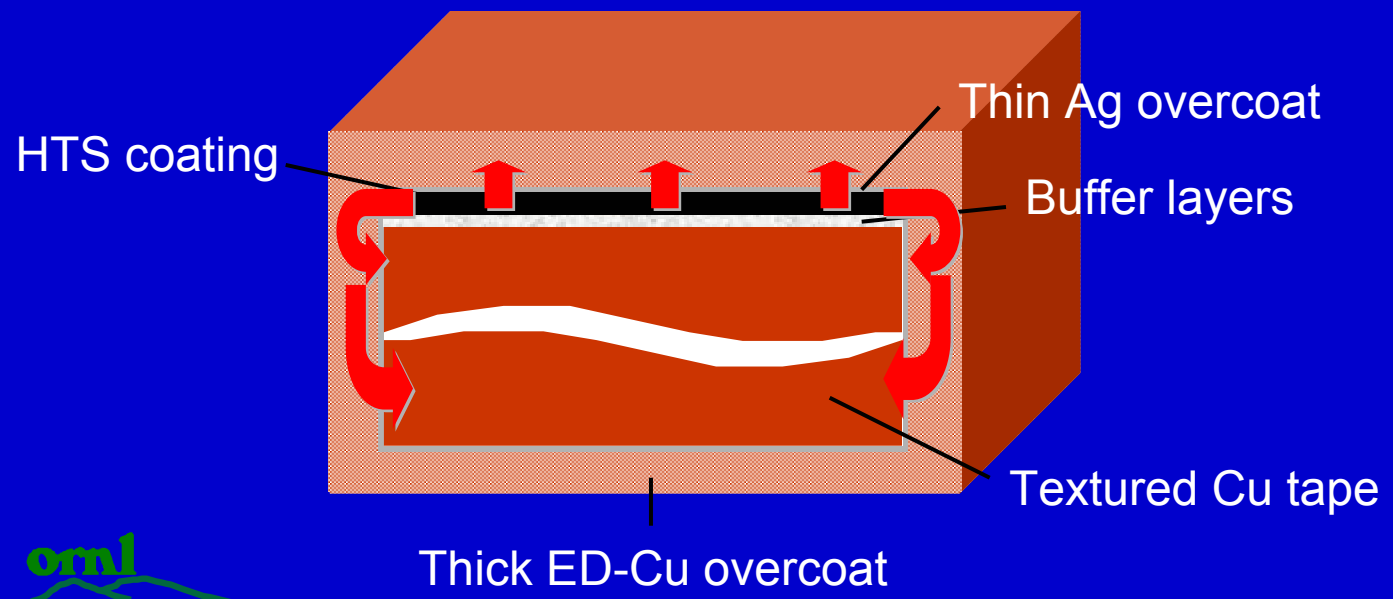
$La_{0.7}Sr_{0.3}MnO_3$ buffer layers can be grown epitaxially on ED-Ni

Superconducting properties of PLD YBCO coatings are less than optimal [J_c(77K,sf) ~ 10^5 A/cm^2, but T_c = 91 K implies no chemical contamination]

Magnetic hysteresis of ED-Ni is comparable to sputtered

FY 2004 Plans

- Identify and solve problems leading to rough ED coatings
- Develop non-magnetic ED metallic seed layers on emerging copper alloy tapes (develop solutions with appropriate cation ratios)
- Evaluate the use of ED-Cu overcoat layers for stabilization



2003 Annual Superconductivity Peer Review

Raghu Bhattacharya

National Renewable Energy Laboratory

Performance and Results

- Established new CRADA with OST and also new collaboration with ORNL, BNL and LANL.
- Developed a “melt-quench-melt-growth” process, which facilitated an open reaction and eliminated loss of material during processing.
- Bi-2212 “coated conductor” was reacted from electro-deposited precursor showing current density of 5×10^6 A/cm^2 at 4.2 K. The films are biaxially textured.
- Transmission electron microscopy (TEM) measurements on Bi-based superconducting films prepared by electrodeposition were carried out to determine the quality of the materials.
- Electrodeposited Ni seed buffer layers were developed on textured Ni, Ni-W, and Cu. Preliminary results: (a) 200 nm PLD YBCO/Ni-W; J_c ~200 kA/cm^2, (b) 300 nm “BaF_2” YBCO/Ni-W; J_c ~250 kA/cm^2, (c) 200 nm PLD YBCO/Cu; J_c ~100 kA/cm^2 .
- Prepared 70 µm thick high quality Cu layer by electrodeposition process to be used as a stabilizer on YBCO coated conductors.

FY 2004 Plans

- Work with Oxford Superconducting Technology on CRADA.
- Investigate non-vacuum techniques for producing biaxial texturing of Bi-2212 on textured metal substrates.
- Optimize Bi-2212 oxygenation heat treatment to improve transport current in the 20–40 K temperature range.
- Explore techniques for improving Bi-2212 by nanoparticle additions.
- Prepare electrodeposited biaxially textured buffer layer for YBCO HTS. Collaboration with ORNL.
- Prepare electrodeposited Cu stabilizer on YBCO-coated conductor. Collaboration with BNL/LANL/ORNL.
- Maintain the DOE Web site for the Superconductivity program.

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Research Integration

Industry: NREL and Oxford Superconducting Technology are studying the Bi-2212 films for possible high-field magnet applications. (CRADA Partner).

DOE National Laboratories: NREL established new collaborative effort with ORNL for electrodeposited buffer layer deposition; NREL also established new collaborative effort with BNL/LANL/ORNL for electro-deposition of Cu stabilizer.

University Interactions:

- Boston College and University of Colorado are working with NREL for characterization and fundamental understanding of the superconducting oxide films (Bi-2212 and YBCO). Two full time Post-Doctorate research fellow work at NREL on these two University subcontract supported by NREL.
- U. Tennessee and U. Florida students, resident at ORNL, supported by AFOSR, are working on NREL/ORNL research collaborative effort.

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DOE Distributed Energy & Electric Reliability Program

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WHAT'S NEW

An information resource for the general public and the superconductivity community. Also home to online issues of the *Superconductivity Update*.

Statistics for January–March 2003

- **More than 22,500 site visitors**
- **64% U.S visitors and 36% international**
- **More than 46,000 document views**

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www.eere.energy.gov/superconductivity

Webmaster: Susan Moon (susan_moon@nrel.gov)