Progress in *ex-situ* PVD-BaF_2 Processes

Oak Ridge National Laboratory

C. Cantoni, H. M. Christen, S. W. Cook, D. K. Christen, R. Feenstra, A. A. Gapud, A. Goyal, L. Heatherly, A. Ijaduola, D. F. Lee, J. Li, F. A. List, M. Paranthaman, P. M. Martin, C. M. Rouleau, E. D. Specht, J. R. Thompson, S.-H. Wee, Y. Zhang

Presented at the
2005 DOE Superconductivity Program Annual Peer Review
Washington DC, Aug. 4, 2005

FY2005 Funding: $ 800,000

UT-BATTELLE

OAK RIDGE NATIONAL LABORATORY
U. S. DEPARTMENT OF ENERGY

Project goal:

Development of *ex-situ* PVD-BaF_2 processes for coated conductor applications

- thick YBCO coatings (several µm) with $I_c \cong 1000$ A/cm at 77 K (FY2010)
- technological know-how for small sample → reel-to-reel processing

- **Base program research in support of:**
  - Coated Conductor Development Roadmap
  - Wire Development Group
  - CRADAs
  - Core RABiTS substrate and buffer development

- **Activities concentrate on:**
  - e-beam deposition of precursor films (stationary and reel-to-reel)
  - Pulsed Electron Deposition as alternative precursor source
  - improving I_c performance of thick YBCO coatings on RABiTS
  - improving flux pinning (guided by WDG objectives)

Significant results towards FY2005 goals include:

- improved understanding of the role of precursor preparation history in enabling fast conversion into high-J_c YBCO
- record high $J_c \cong 3.5\text{-}4$ MA/cm^2 (77 K) on RABiTS in thin (0.12 µm) YBCO
- identification of dominant pinning mechanism and defects in PVD-BaF_2 *ex-situ* films
- installation and implementation of a low-pressure reel-to-reel furnace
  - achieved high-J_c values from e-beam precursors (1-2 µm)
- identification of different conversion behavior from e-beam vs. PED precursors
  - impetus for program redirection in FY2006

Presentation outline

Ron Feenstra	Development of high-I_c *ex-situ* YBCO conductors
Dominic Lee	Reel-to-reel low pressure furnace processing
Hans Christen	Pulsed Electron Deposition of BaF_2 precursors
Bob Hawsey	FY2005 Performance FY2006 Plans Research Integration

Presentation outline

Ron Feenstra	Development of high-I_c *ex-situ* YBCO conductors
Dominic Lee	Reel-to-reel low pressure furnace processing
Hans Christen	Pulsed Electron Deposition of BaF_2 precursors
Bob Hawsey	FY2005 Performance FY2006 Plans Research Integration

Overview

- improving the thickness dependence of J_c (review)

- role of intermediate, low-T precursor anneal
  - enabling high J_c in fast-converted films
  - quenching studies
  - techniques used: FIB-SEM, XPS, RAMAN (with ion milling)

- vortex pinning
  - general characteristics for H||c
  - temperature dependence of J_c(H)

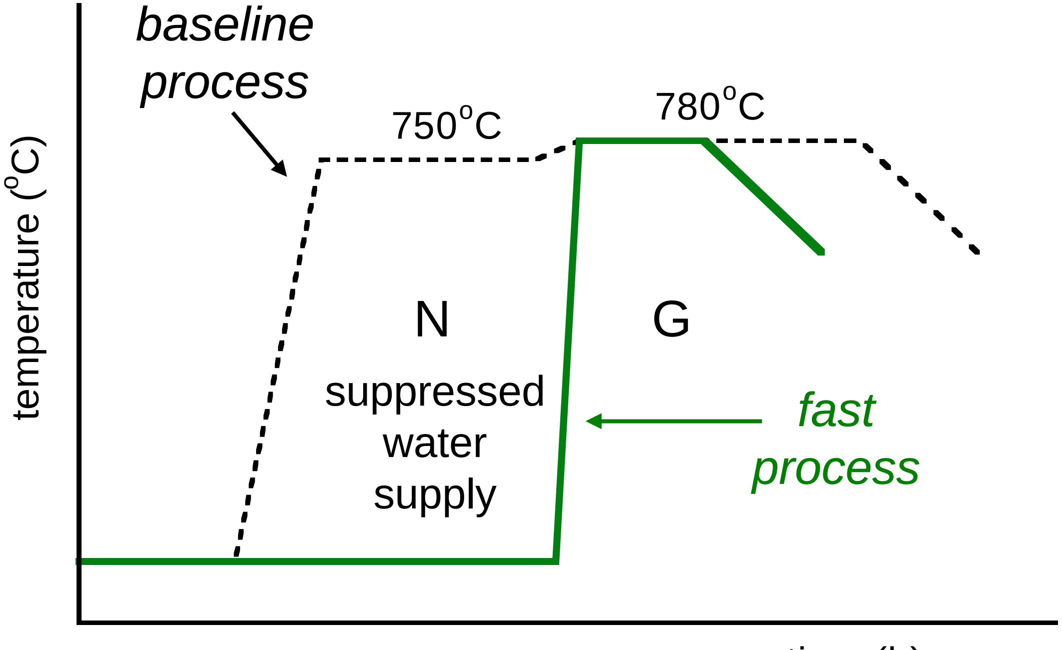
- precursors deposited by e-beam evaporation (Y, BaF_2, Cu sources)
  - arbitrary thickness in range 30 nm – 3 µm

- *ex-situ* conversion in flowing gases at 1.0 atm total pressure
  - rates up to 14 Å/s demonstrated

FY04: A fast conversion process was developed for PVD-BaF_2 precursors

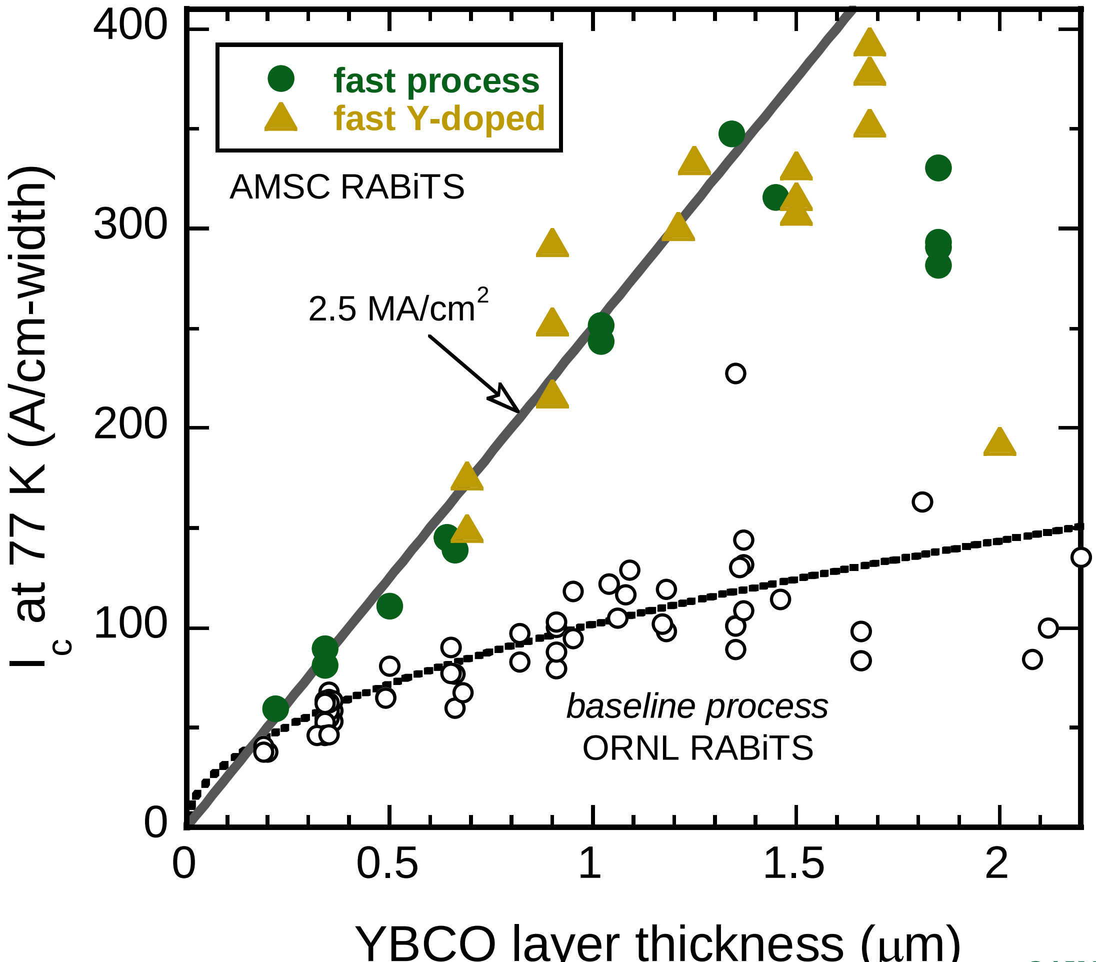
- New processing scheme ties together precursor preparation history and the *ex-situ* conversion
  - insertion of low-T oxidation/modification anneal (400ºC)
  - "aggressive" conversion conditions (gas flow, T, $p(H_2O)$)

    standard 1 atm & low-pressure conversion systems



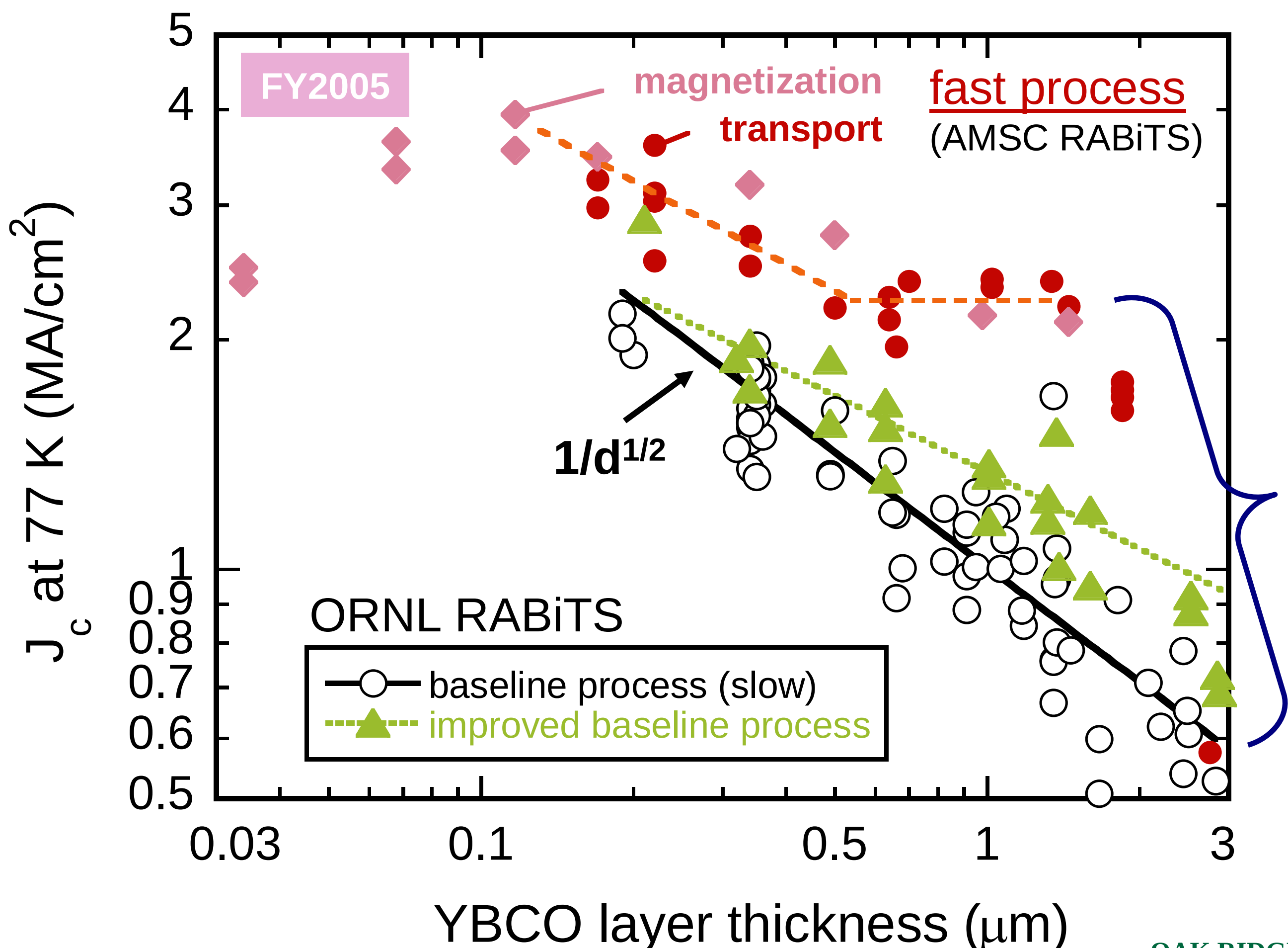
FY2004: I_c of fast processed films increases linearly with the YBCO thickness (d < 1.5 μm)

- **best I_c ≅ 400 A/cm for 1.7 μm Y-doped YBCO**



Processing iterations have reduced J_c fall-off with YBCO thickness

Baseline process → Improved baseline process → Fast process
(FY2002) (FY2003) (FY2004)



fast process not optimized for thick YBCO

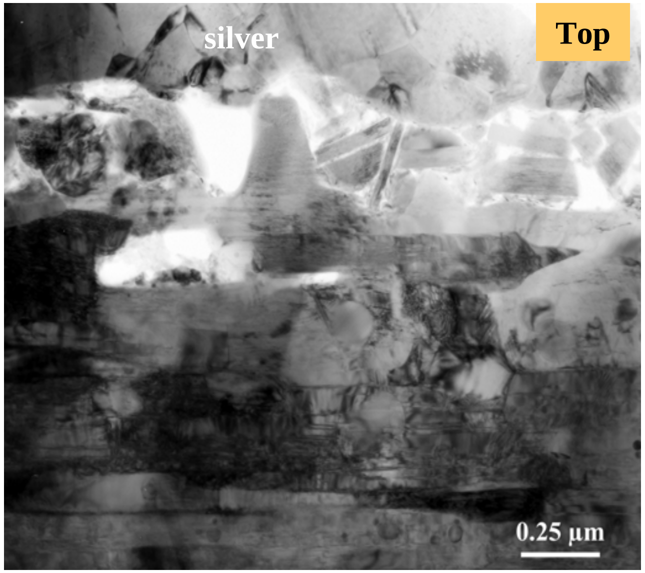
Structural changes enabling improved performance of thick YBCO have been identified by TEM

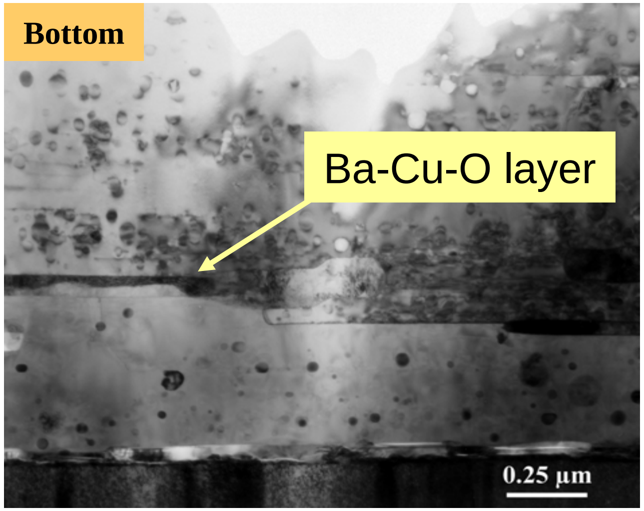
- Terry Holesinger (LANL)
- **<u>bimodal structure</u>** in thick films (baseline process)
  - large, well formed YBCO grains in bottom half of film
  - smaller, faulted YBCO grains in the top half

➢ different growth modes through thickness

Holesinger et al., JMR **20**, p.1216-1233 (2005)

➢ process iterations have enabled elimination or modification of the bimodal structure → **improved J_c performance**





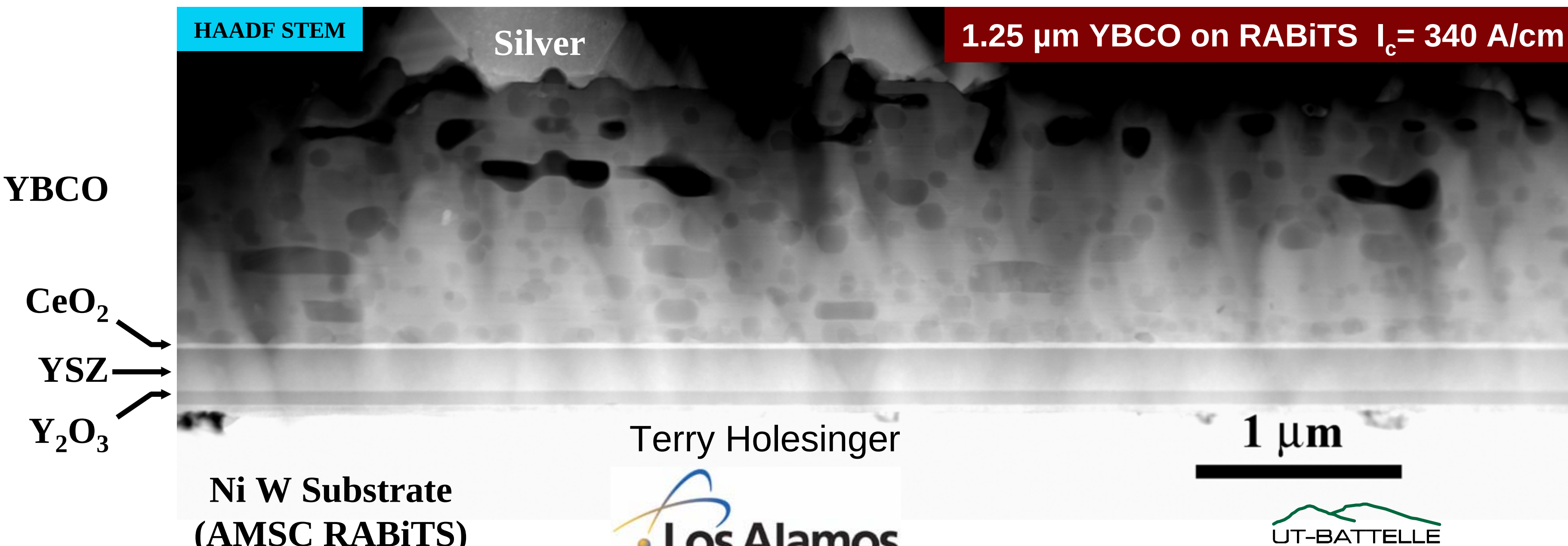
Los Alamos NATIONAL LABORATORY EST. 1943

UT-BATTELLE

OAK RIDGE NATIONAL LABORATORY
U. S. DEPARTMENT OF ENERGY

Fast-processed film (1.25 μm) exhibits uniform through-thickness microstructure

- non “bimodal”—no intercalated second phase layers
- porosity is present
- uniform distribution of planar defects and small secondary phases
- increased similarity with MOD *ex-situ* YBCO films



Terry Holesinger

Los Alamos NATIONAL LABORATORY EST.1943

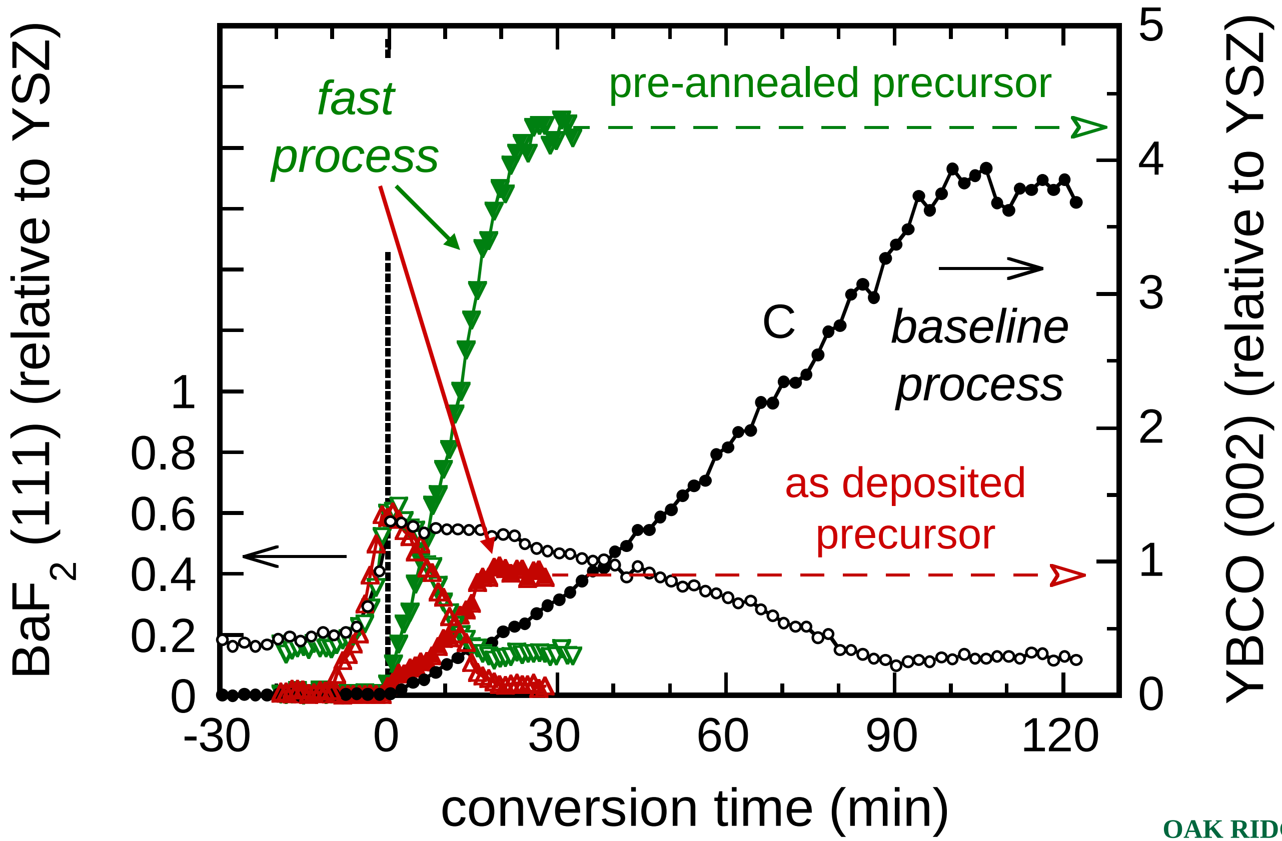
UT-BATTELLE

OAK RIDGE NATIONAL LABORATORY
U. S. DEPARTMENT OF ENERGY

Low-temperature pre-anneal is essential for successful application of FAST conversion process

- <u>conversion rate</u> controlled by processing parameters
- <u>YBCO quality</u> controlled by precursor microstructure/chemistry

- *in-situ* XRD monitors conversion in real time (low-pressure system)



I_c = 189 A/cm

1 μm YBCO
RABiTS

I_c < 1 A/cm

Insertion of low-T modification anneal resembles the calcination anneal in MOD BaF_2 processing

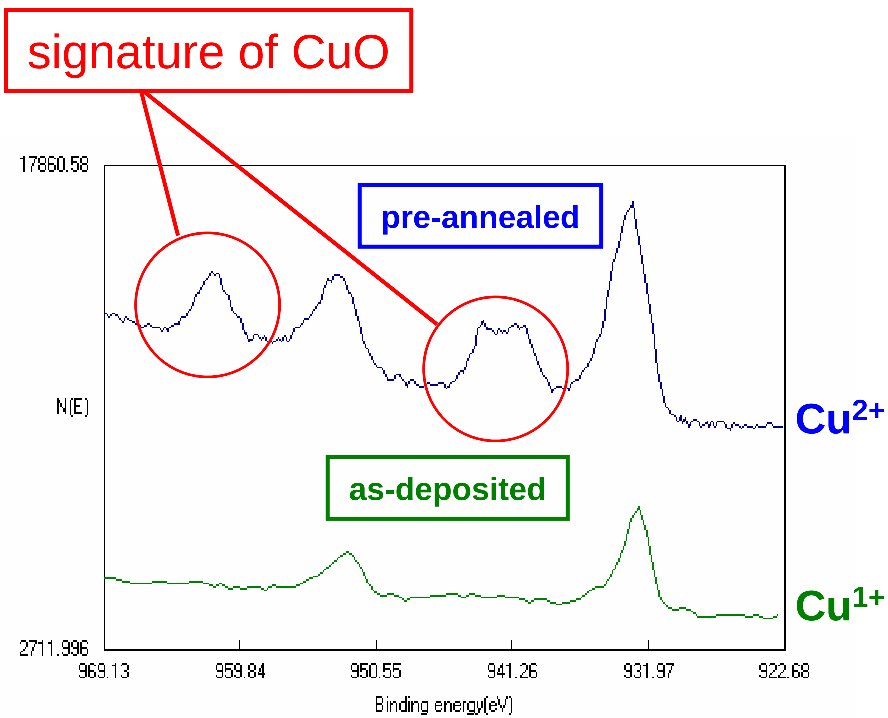
- modification anneal is expected to increase the oxidation level, induce structural development on the nano-scale
  - FY2004: benefits of these changes were speculated

➢ In FY2005, a new line of research was initiated to elucidate the role of precursor characteristics on the *ex-situ* conversion process
  - research is leveraged by external collaborations: Albany NanoTech, Univ. of Tennessee, ANL

New angle towards better understanding / control of thick film conversion

XPS confirms increased Cu oxidation level in pre-annealed precursor

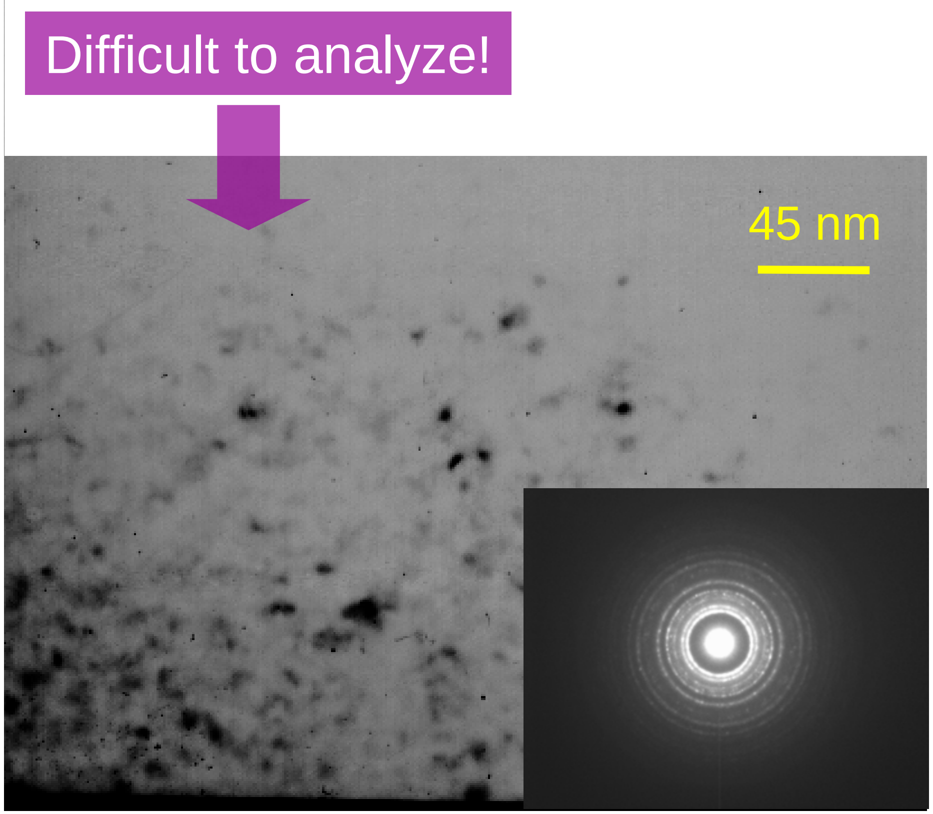
(500ºC, 1 atm. O_2)



Yifei Zhang

TEM shows structural development on nano-scale

- as-deposited precursor is amorphous

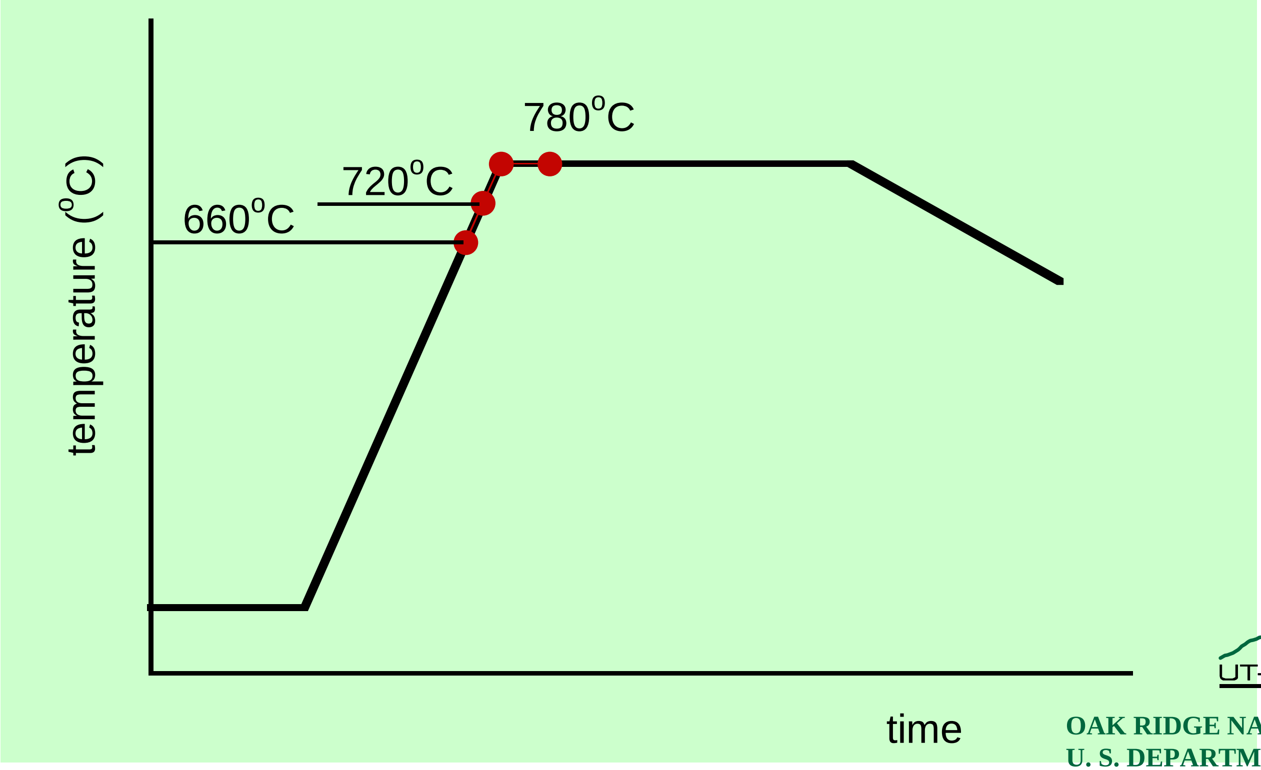


Claudia Cantoni

UT-BATTELLE
OAK RIDGE NATIONAL LABORATORY
U. S. DEPARTMENT OF ENERGY

Strategy for characterizing effects of low-T pre-anneal: Track phase development into early nucleation of YBCO

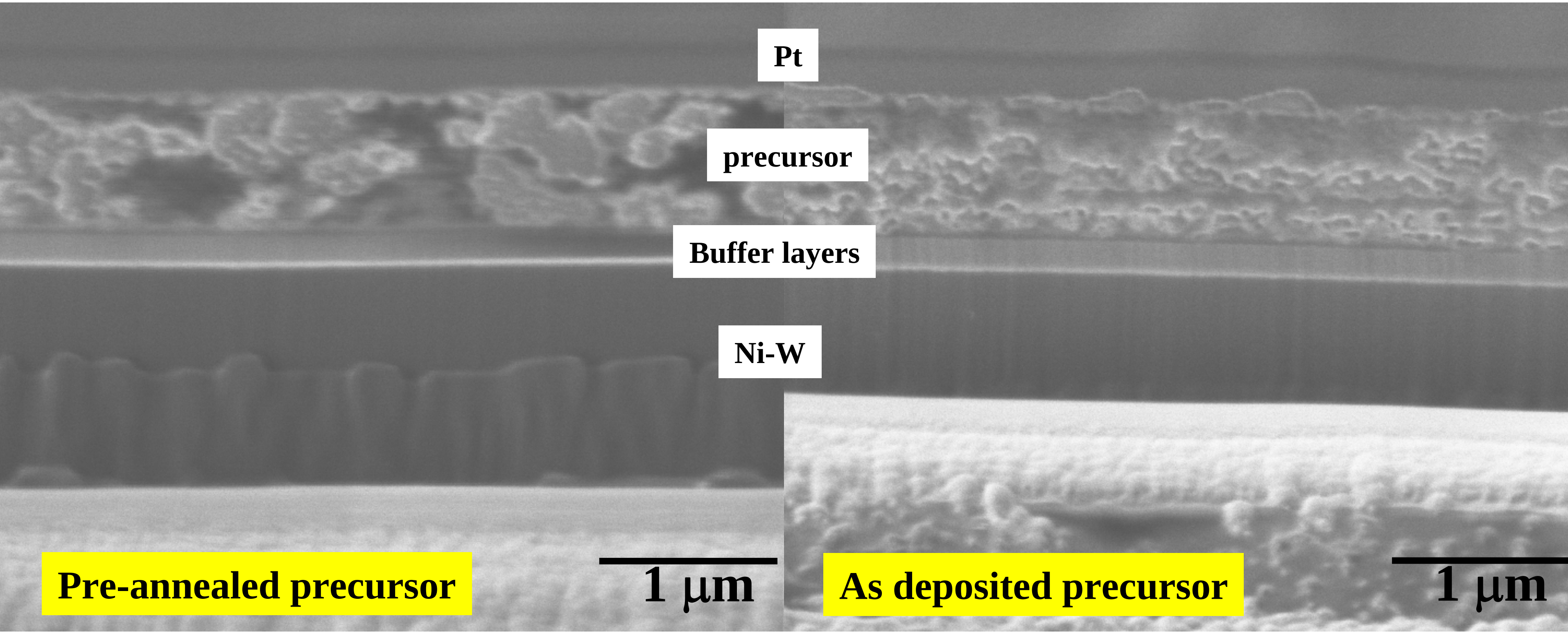
- side-by-side processing / quenching of ***as-grown*** and ***pre-annealed*** precursors
  - as-grown precursor ("bad genes") → poor YBCO performance
  - pre-annealed precursor ("good genes") → high I_c YBCO
- characterization performed by FIB-SEM, XPS, ion backscattering, RAMAN, …
  - establish which technique provides relevant information most efficiently



FIB-SEM visualizes different microstructures depending on precursor pretreatment

- precursors for 1 µm YBCO (ORNL RABiTS)
- samples quenched from 780ºC (at the end of the ramp; t_{ann}= 0)

granular microstructure | granular, layered microstructure



Manisha Rane

Comparison of FIB Microstructures at 50 kX

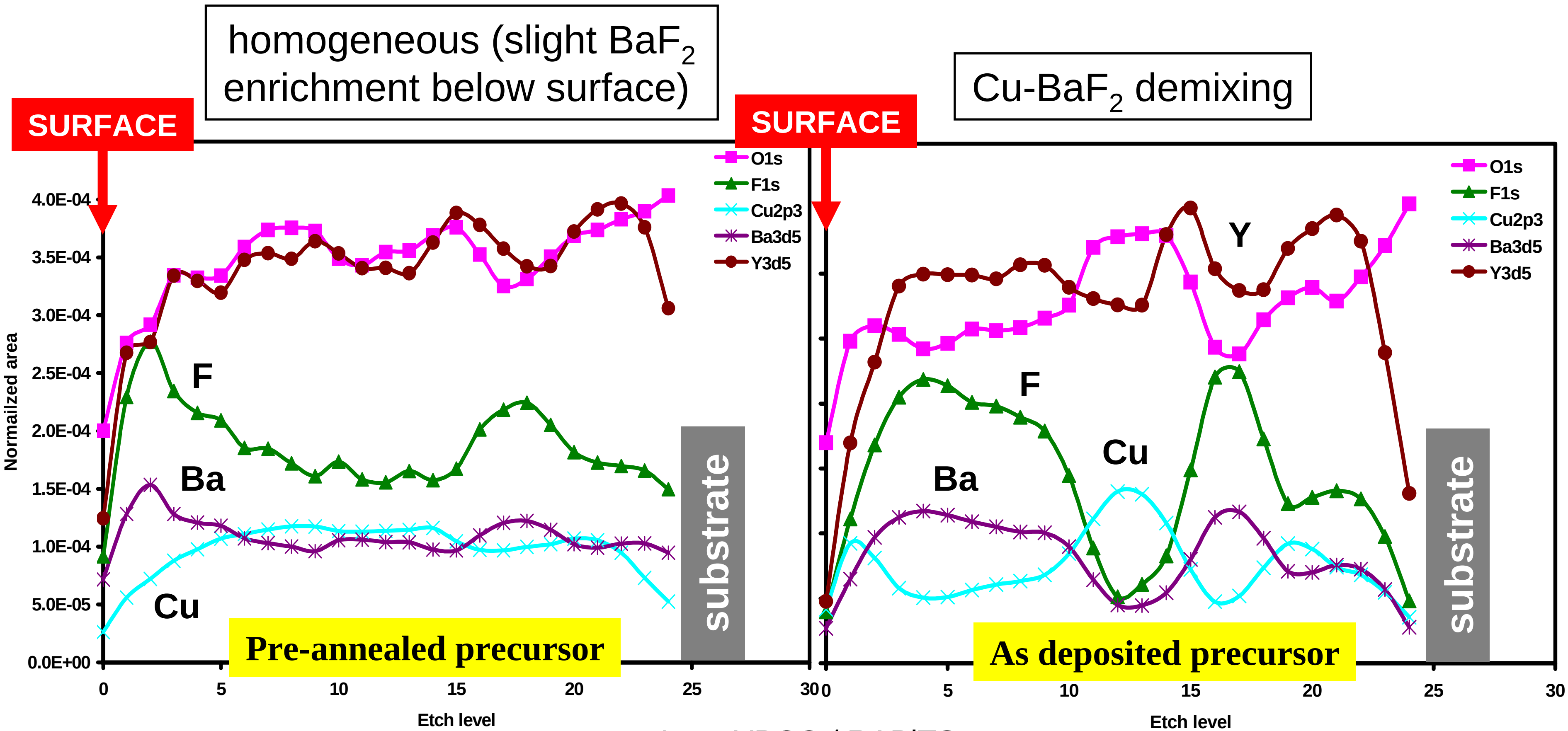
UNIVERSITY AT ALBANY
State University of New York

UT-BATTELLE

OAK RIDGE NATIONAL LABORATORY
U. S. DEPARTMENT OF ENERGY

XPS elemental depth profiles show a layered segregation in **as-deposited** precursor after ramp to conversion temperature

- **layered segregation may interfere with homogeneous conversion**



1 µm YBCO / RABiTS

Manisha Rane

UNIVERSITY AT ALBANY
State University of New York

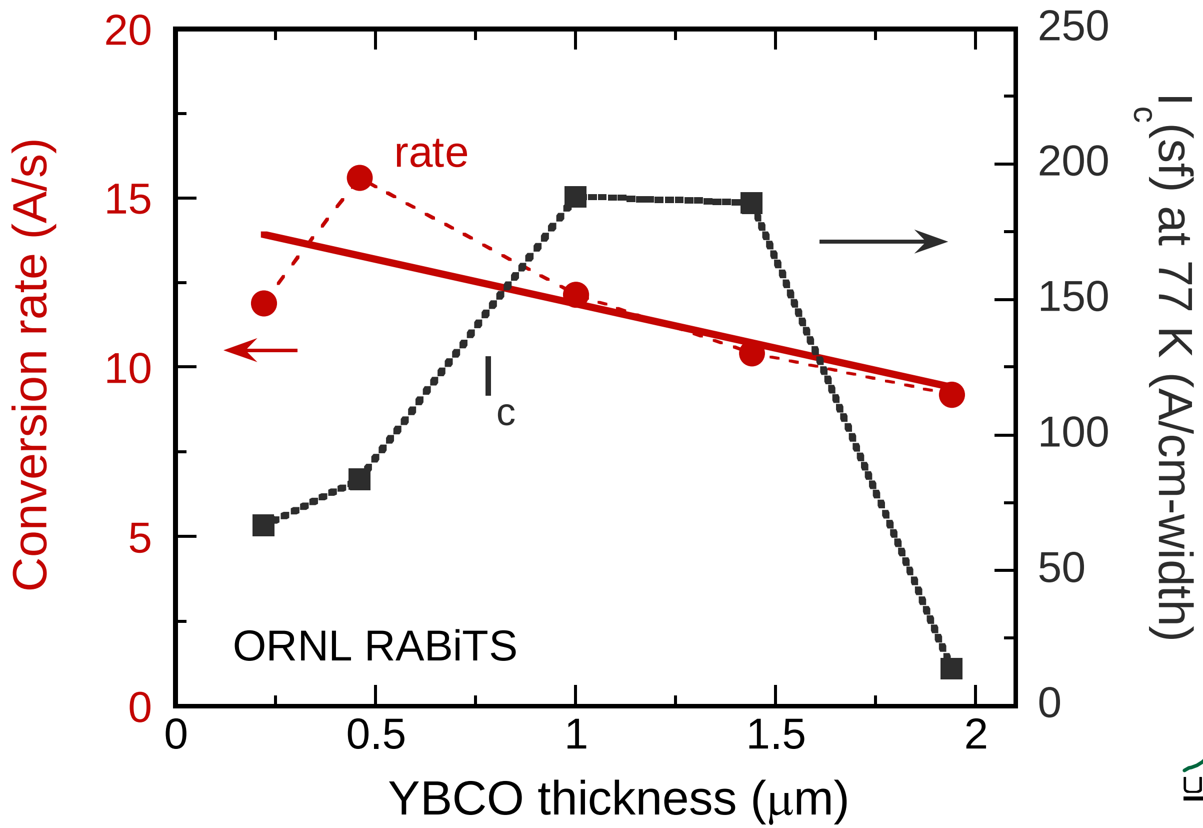
UT-BATTELLE
OAK RIDGE NATIONAL LABORATORY
U. S. DEPARTMENT OF ENERGY

Conversion rate of e-beam BaF_2 precursors exhibits minor dependence on the precursor thickness

- constant precursor preparation history, FAST conversion conditions
- low-pressure conversion system with real-time XRD

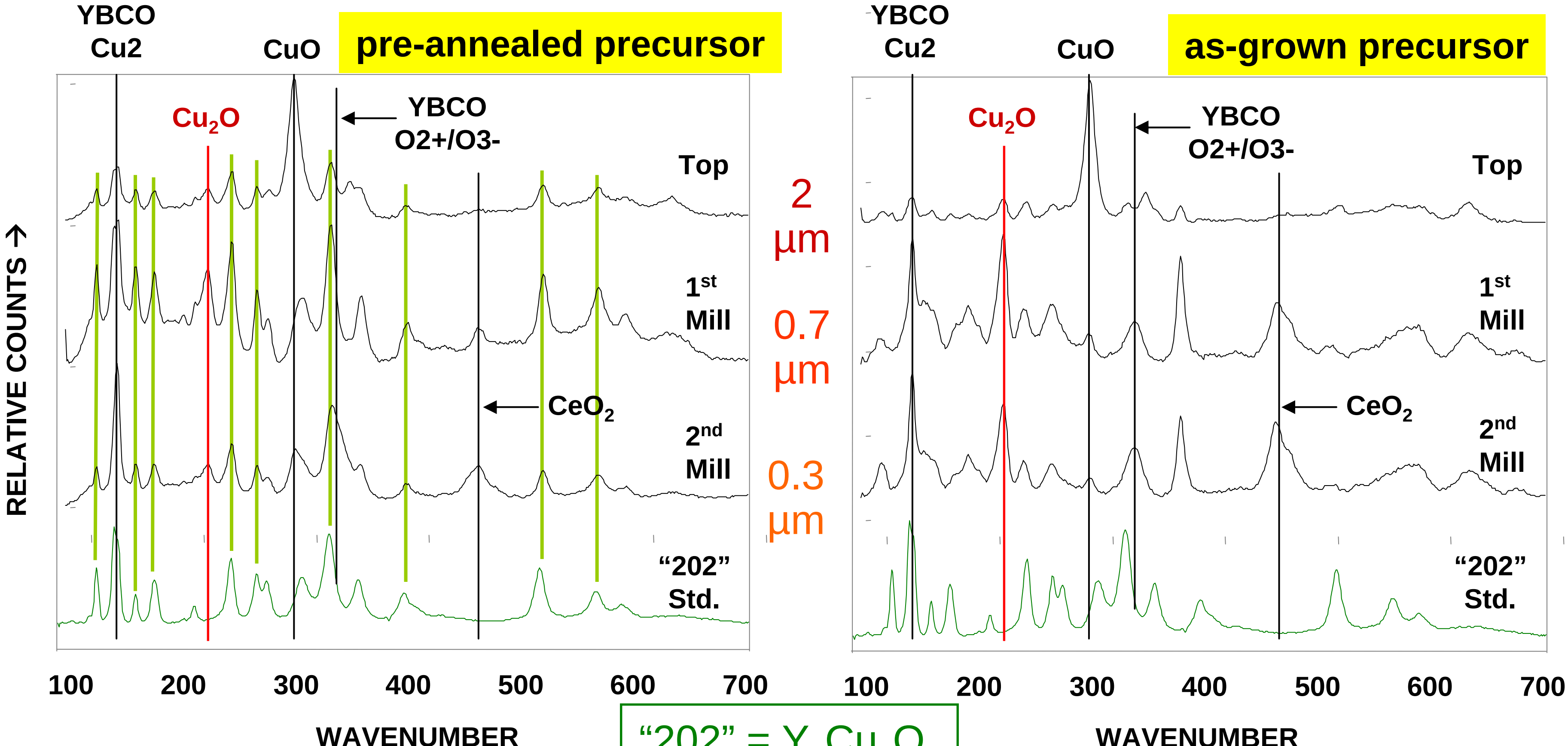
➢ poor c-axis YBCO growth, low I_c of 2 µm film suggest <u>precursor origin</u>

- similar to problems in fast conversion of 1 µm thick as-grown precursors



Raman spectroscopy at different levels in quenched **2 µm thick** precursors reveals history-dependent reaction paths

- quenched after 4 min. into the conversion anneal at 780ºC
- reduced "202" (intermediary phase) development in <u>as-grown</u> precursor



"202" = $Y_2Cu_2O_5$

Vic Maroni

ARGONNE NATIONAL LABORATORY

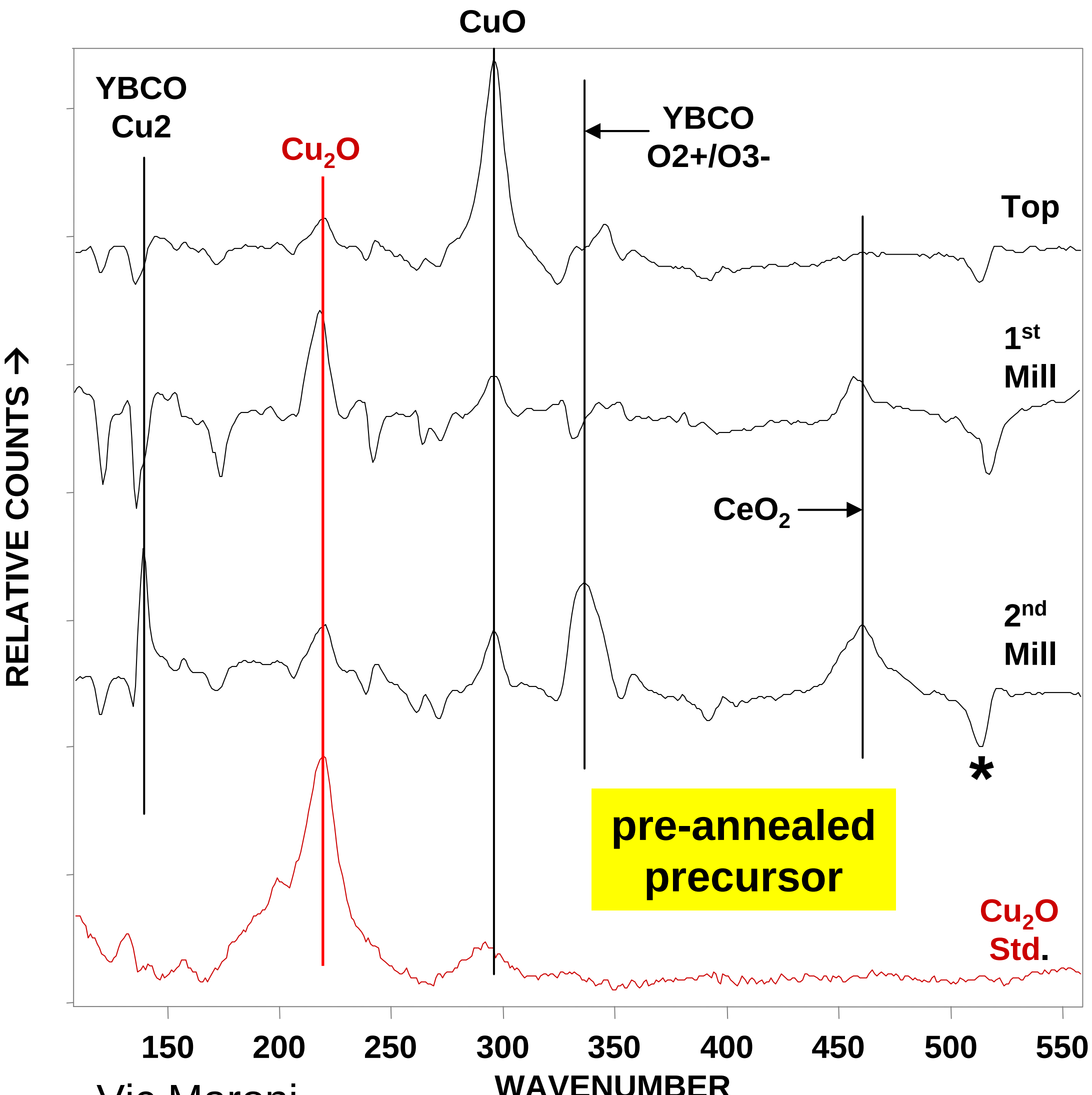
THE UNIVERSITY of WISCONSIN MADISON

Matt Feldmann

UT-BATTELLE

OAK RIDGE NATIONAL LABORATORY
U. S. DEPARTMENT OF ENERGY

Subtraction of the "202" spectrum provides evidence for varying amounts of Cu_2O, CuO at different depths into the film



2 µm

0.7 µm

0.3 µm

- reduced amount of O near substrate interface may reduce YBCO nucleation probability
- possible cause of large grains in bottom part of thick films ("bimodal" structure)

***Note: the series of negative-going blips are due to over-compensated "202" bands**

Vic Maroni

ARGONNE NATIONAL LABORATORY

THE UNIVERSITY of WISCONSIN MADISON

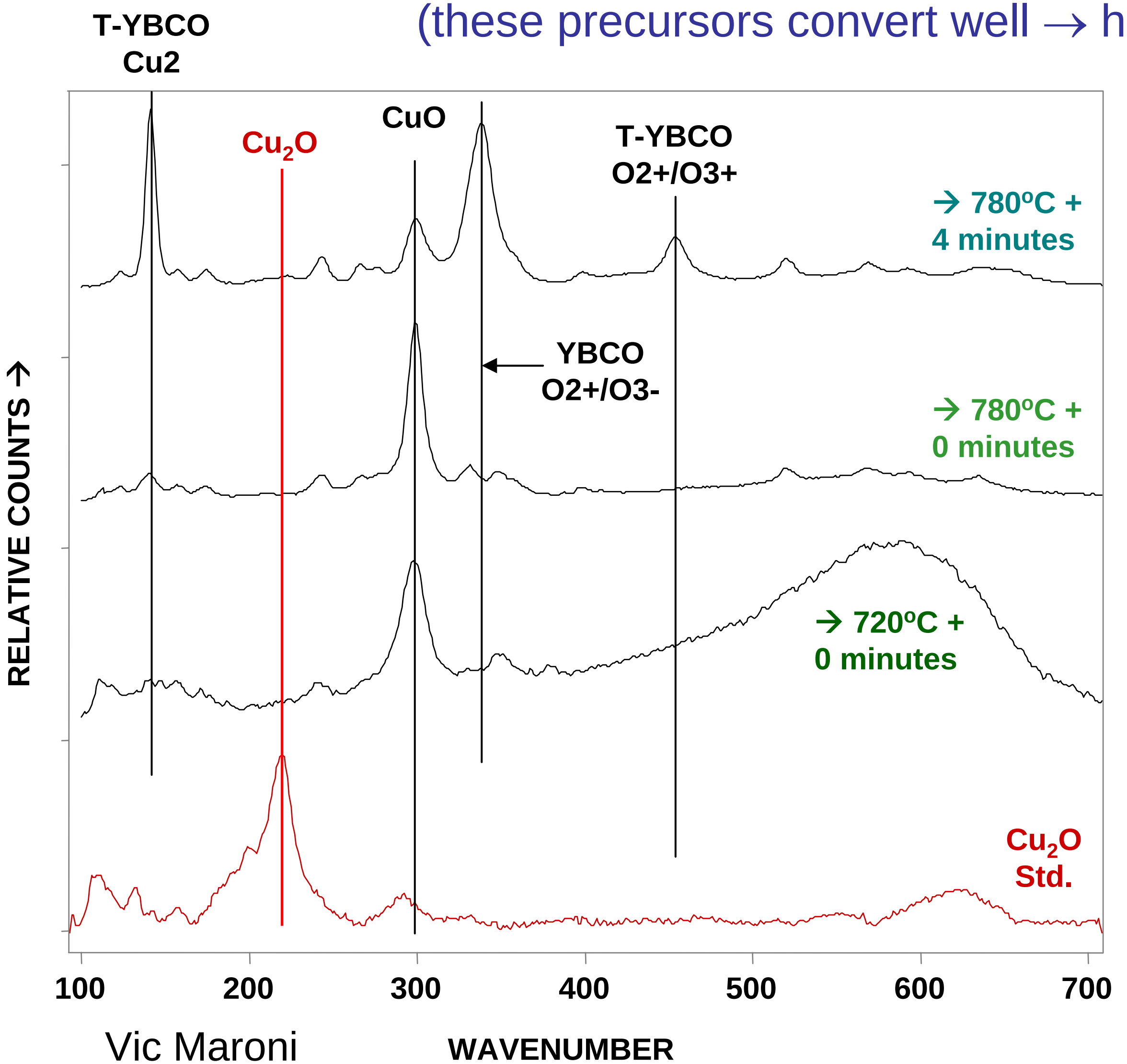
Matt Feldmann

UT-BATTELLE

OAK RIDGE NATIONAL LABORATORY
U. S. DEPARTMENT OF ENERGY

By contrast, **pre-annealed 1 µm** thick precursors appear adequately oxidized prior to YBCO nucleation

(these precursors convert well → high I_c)



Vic Maroni

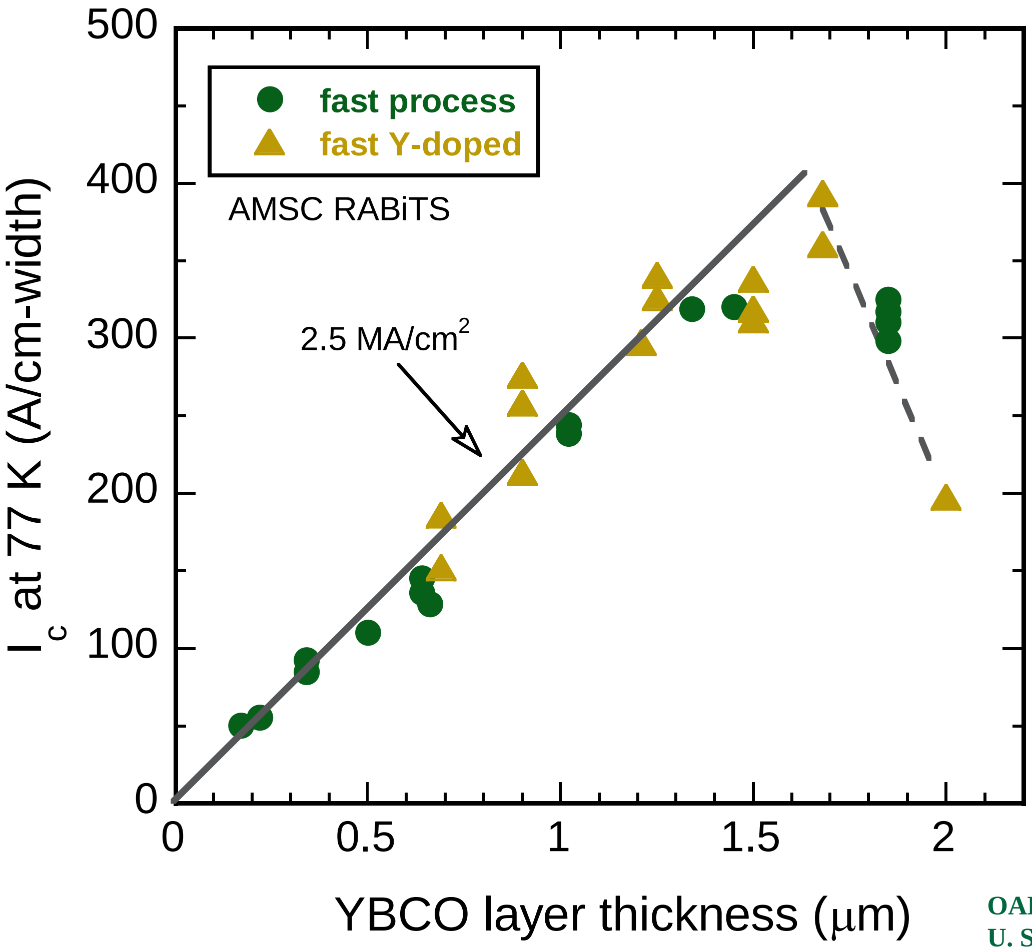
- kinetics of precursor (Cu) oxidation introduce a thickness dependence in the processing
  - ➢ opportunities for improvement

ARGONNE NATIONAL LABORATORY

UT-BATTELLE

OAK RIDGE NATIONAL LABORATORY
U. S. DEPARTMENT OF ENERGY

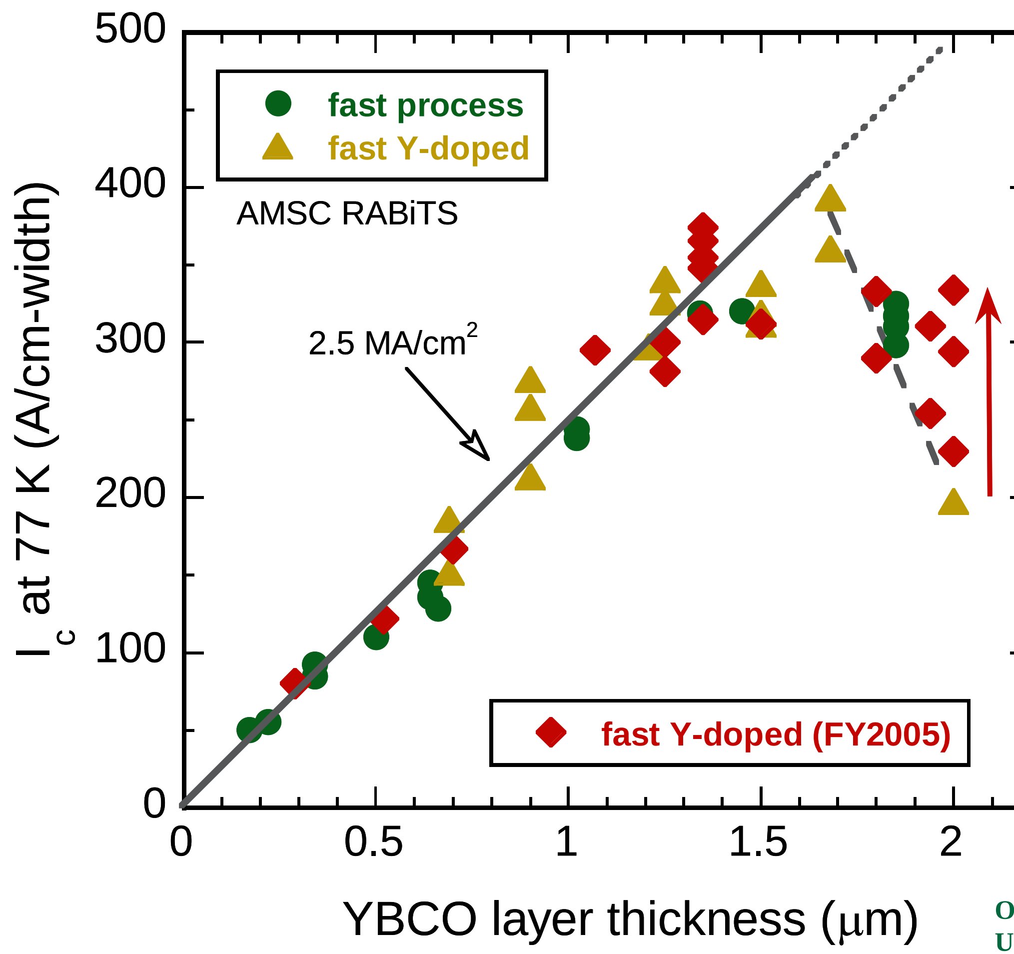
Sudden drop in I_c for d > 1.7 μm appears to result from inadequate precursor conditioning



UT-BATTELLE

Sudden drop in I_c for d > 1.7 µm appears to result from inadequate precursor conditioning

- FY05: improved performance at 2 µm achieved from "Edisonian" approach
- FY06: I_c > 400 A/cm expected from implementation of characterization feedback
- ➢ processing focus will be on thicknesses of 1.5-2 µm



UT-BATTELLE

OAK RIDGE NATIONAL LABORATORY
U. S. DEPARTMENT OF ENERGY

Vortex pinning in PVD-BaF_2 *ex-situ* films
general characteristics

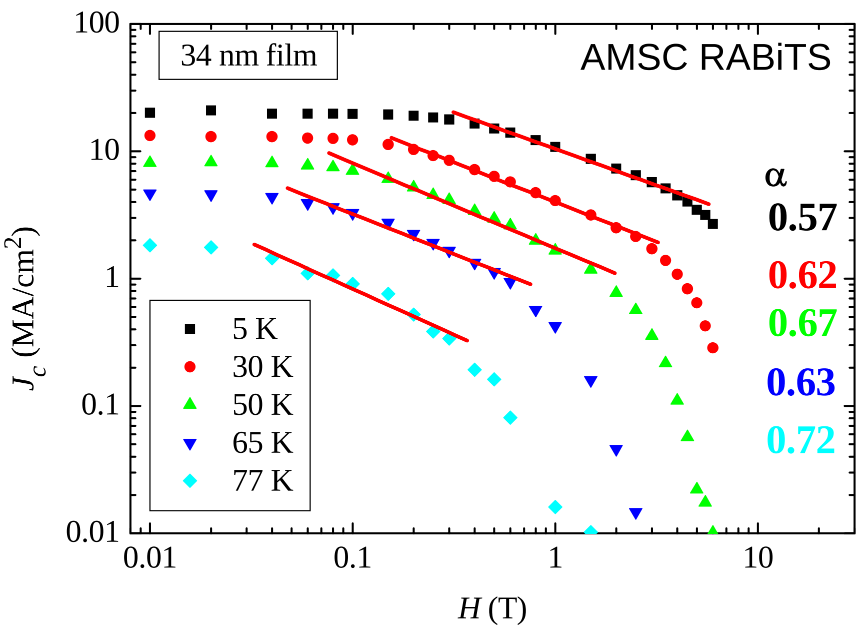
- power law dependence $J_c \propto H^{-\alpha}$ in intermediate H || c fields

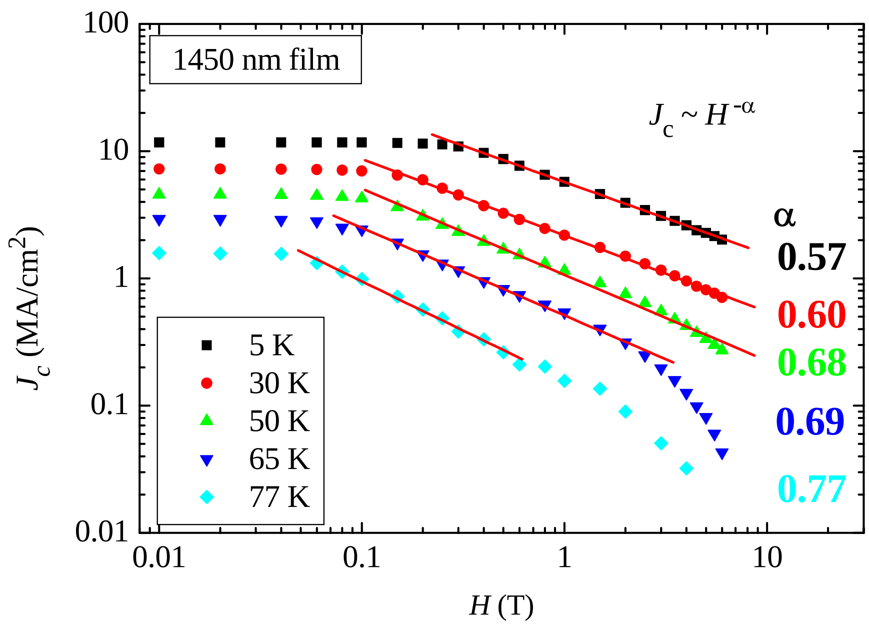
- scaling of low-temperature J_c in applied fields of several Tesla to J_c(sf) at 77 K
  - rotating machinery applications (T= 40 K)

$J_c(H,T)$ exhibits many features characteristic of pinning by strong, sparse defects (Y_2O_3, $Y_2Cu_2O_5$, nano-pores,…)

- magnetization study in thickness range 34 nm – 1.4 µm (J_c > 2 MA/cm^2 at 77 K)
- power law $J_c \sim H^{-\alpha}$ with α = 0.57–0.69 for all thicknesses and T = 5 – 65 K
- compares well with theoretical value α = 5/8 for pinning by strong, sparse defects

  [Ovchinnikov and Ivlev (PRB, 1991) and van der Beek et al (PRB, 2002)]

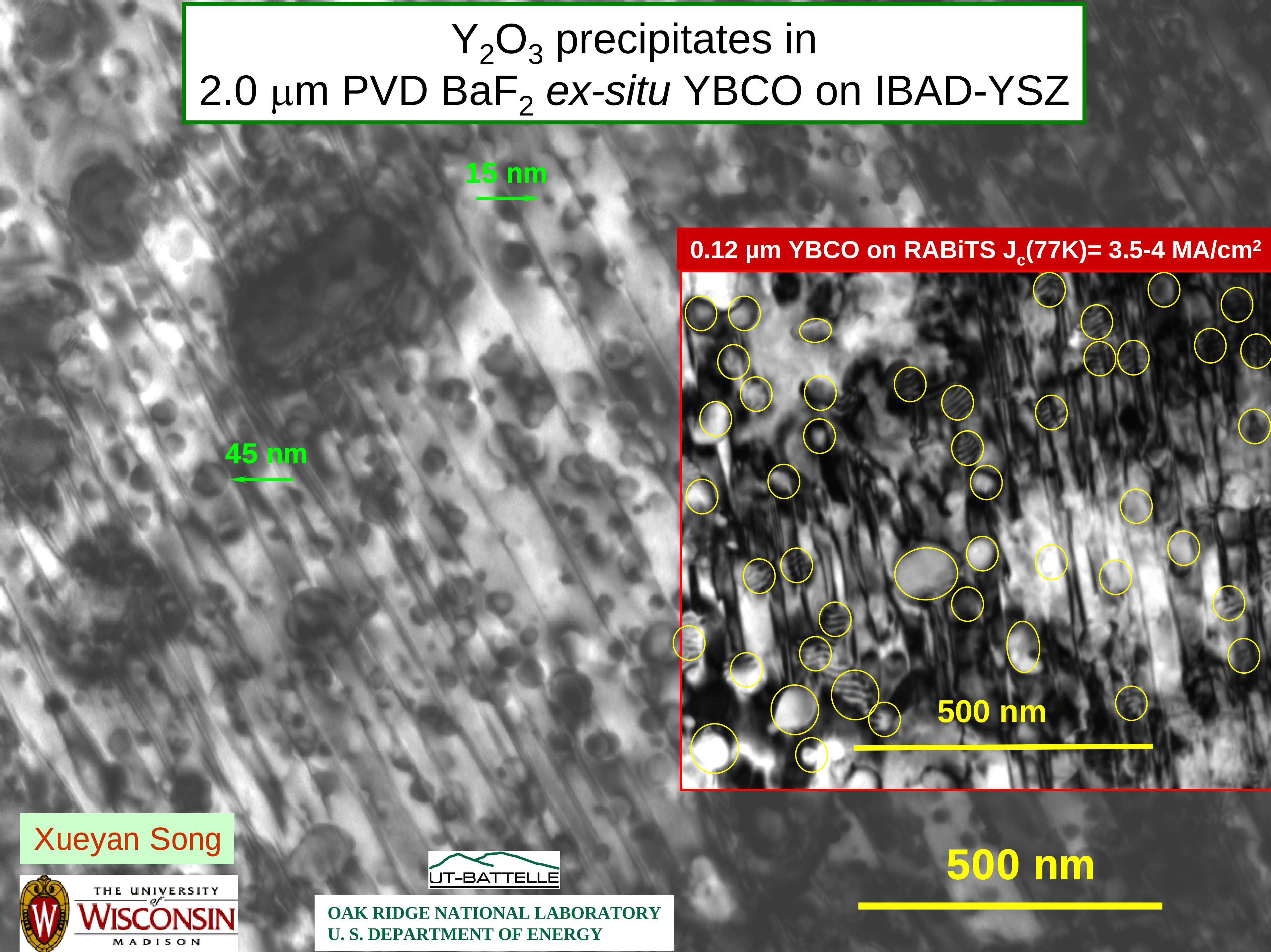




Anota Ijaduola
Jim Thompson

UT-BATTELLE
OAK RIDGE NATIONAL LABORATORY
U. S. DEPARTMENT OF ENERGY

Y_2O_3 precipitates in 2.0 μm PVD BaF_2 *ex-situ* YBCO on IBAD-YSZ



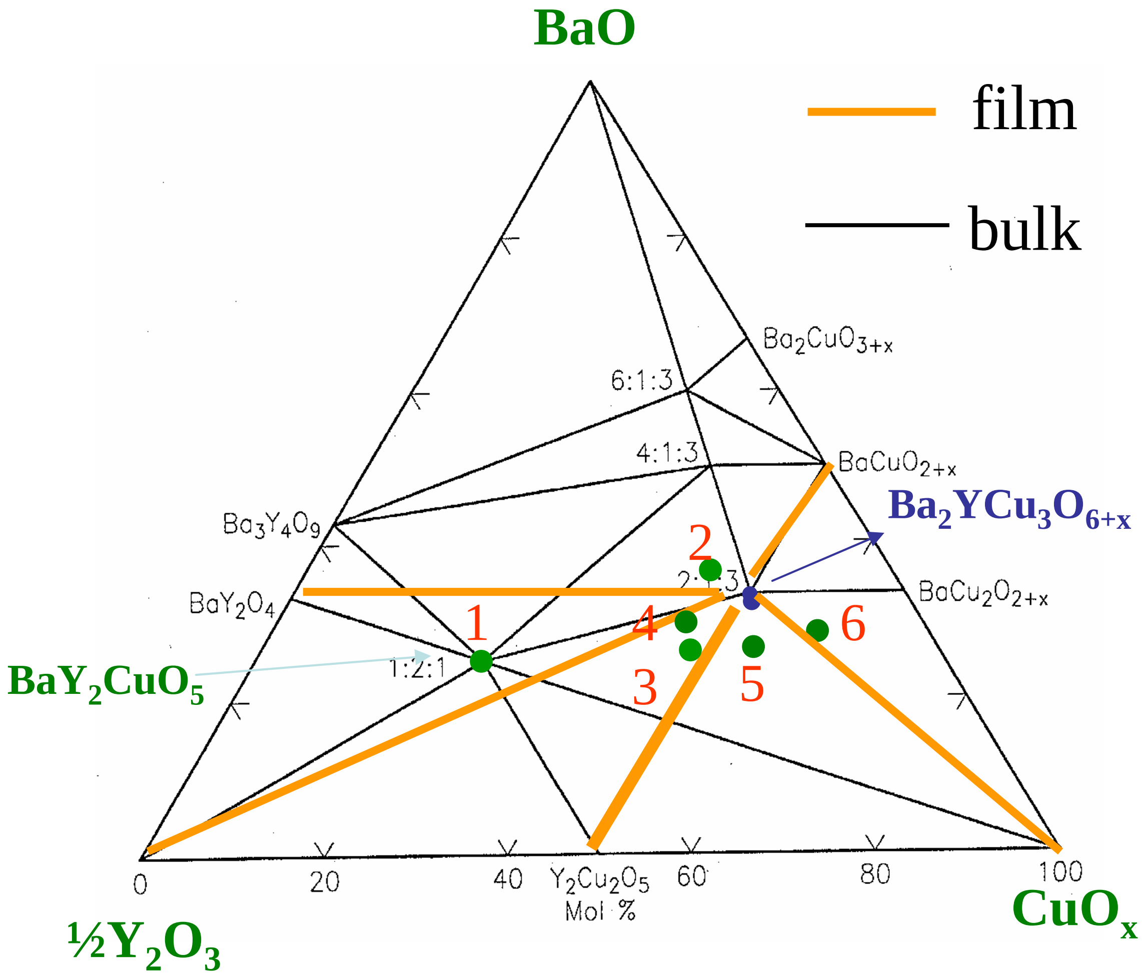
Xueyan Song

THE UNIVERSITY of WISCONSIN MADISON

UT-BATTELLE

OAK RIDGE NATIONAL LABORATORY
U. S. DEPARTMENT OF ENERGY

Phase Relationships of the BaO-½Y_2O_3-CuO system <u>in *ex-situ* films</u> confirm origin of Y_2O_3 precipitates



- **Different phase relations in the vicinity of Y-213 for bulk and film (*ex-situ*)**
- BaY_2CuO_5 does not exist in film; X-ray pattern shows Y_2O_3, $Ba_2YCu_3O_{6+x}$, and BaY_2O_4.
- $Ba_2YCu_3O_{6+x}$ is compatible with Y_2O_3 $Y_2Cu_2O_5$, BaY_2O_4, CuO, $BaCuO_2$ etc., but not with BaY_2CuO_5 (211-phase)

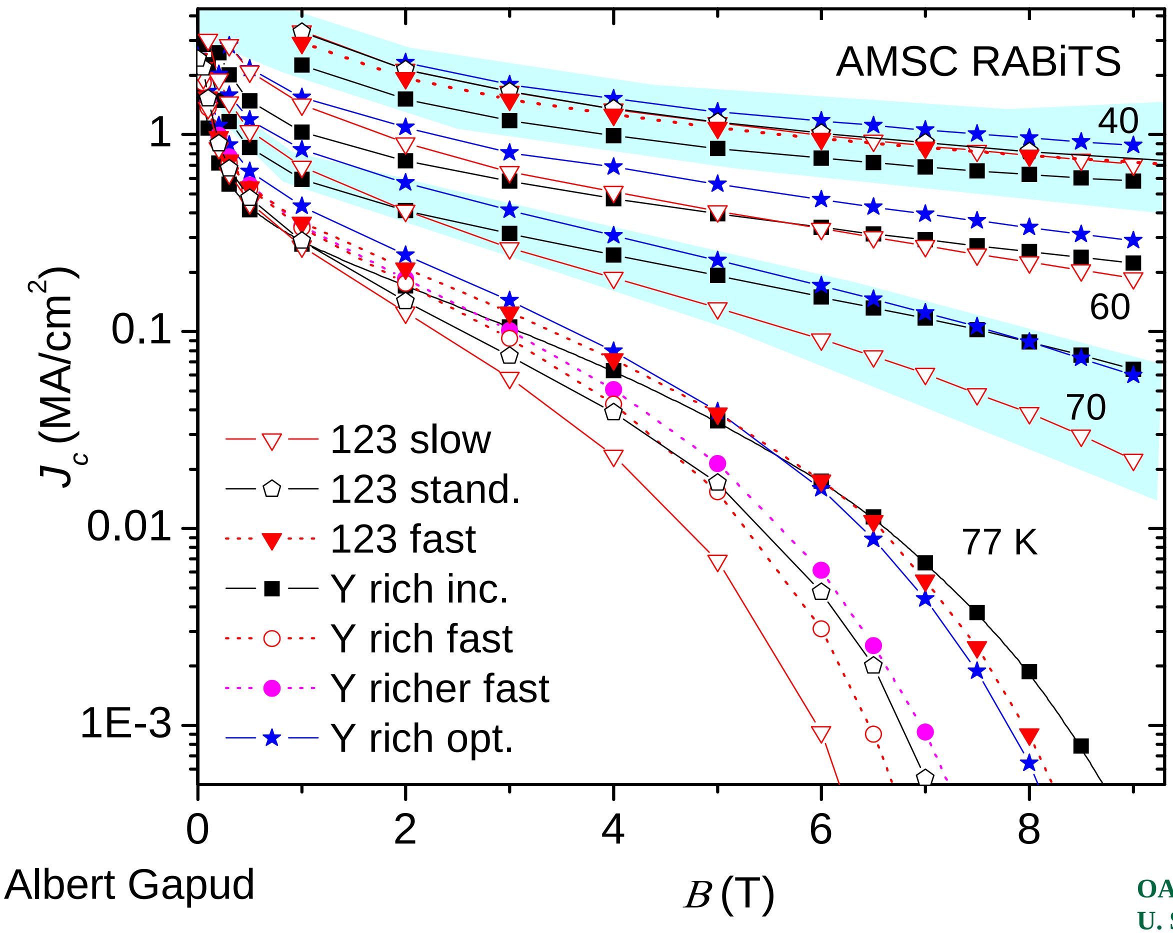
Winnie Wong-Ng
Igor Levin
Larry Cook

NIST

UT-BATTELLE
OAK RIDGE NATIONAL LABORATORY
U. S. DEPARTMENT OF ENERGY

Operating range of rotating machinery (motors) involves H fields of 2-3 T—need low T for adequate I_c

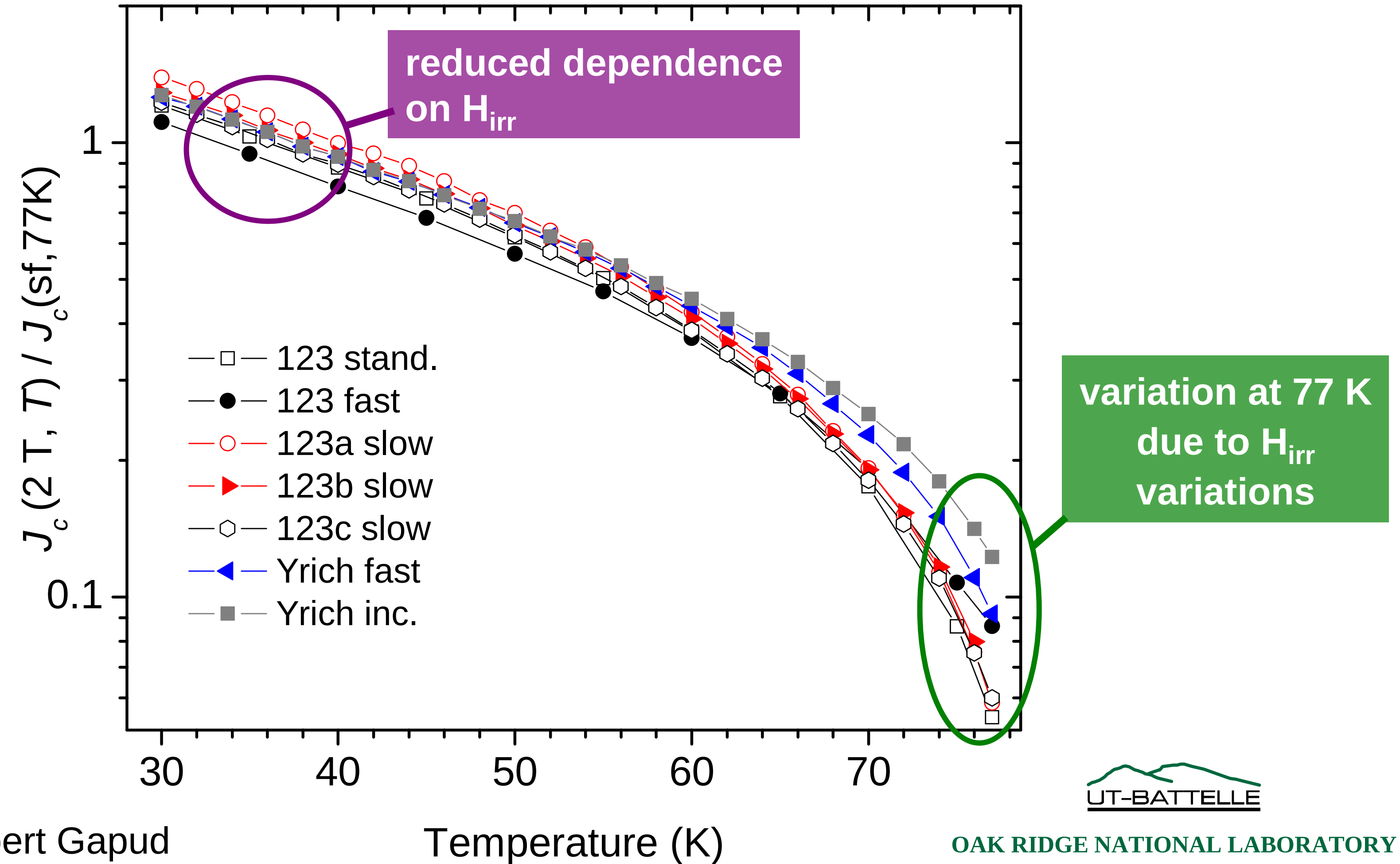
- YBCO films with variable preparation history, angular dependence of J_c
- irreversibility field variations at 77 K—correlate with T_c (89-94 K)
- low T performance does not track H_{irr}(77 K) variations



Albert Gapud

Low-temperature, in-field performance scales with J_c(sf) at 77 K

- J_c(H||c = 2T) at 30-40 K ≅ J_c(sf, 77 K)
- proportionality constant increases with decreasing power law exponent α



Albert Gapud

Summary (FY2005 Results)

- J_c dependence on YBCO thickness is processing dependent
  - process iterations have reduced the J_c drop-off with thickness
- precursor preparation history has a strong effect on I_c performance
  - layered phase separation and reduced $Y_2Cu_2O_5$ formation are undesirable
  - intermediate low T pre-anneal (300-500ºC) improves Cu oxidation
- conversion rate is ~ thickness independent in range 0.2-2 µm
  - kinetic effects associated with O transport play a role in early YBCO nucleation
- FIB-SEM and Raman spectroscopy (with ion milling) identified as efficient techniques towards process optimization

Summary (FY2005 Results)

- <u>Performance improvements</u>:
  - J_c= 3.5-4 MA/cm^2 (77 K) in 0.12 µm YBCO on RABiTS (AMSC)
  - I_c = 300 A/cm in 1.0 µm Y-doped YBCO on RABiTS
  - I_c = 370 A/cm in 1.4 µm Y-doped YBCO on RABiTS
  - I_c of fast-processed 2 µm films has been increased from 200 $\rightarrow$ 330 A/cm
  - I_c > 400 A/cm appears feasible with tuning of precursor-conversion processing
- <u>Flux pinning</u>:
  - thickness and temperature independent power law $J_c \propto H^{-\alpha}$ exponent α is consistent with pinning by strong, sparse defects (α = 5/8)
  - TEM $\rightarrow$ nano-scale secondary phase inclusions, voids
  - J_c in application regime for rotating machinery (2-3T, 30-40 K) scales with self-field J_c at 77 K, not improved by increases in H_{irr}

Presentation outline

Ron Feenstra — Development of high-I_c *ex-situ* YBCO conductors

Dominic Lee — Reel-to-reel low pressure furnace development and processing

Hans Christen — Pulsed Electron Deposition of BaF_2 precursors

Bob Hawsey — FY2005 Performance
FY2006 Plans
Research Integration

Why use low pressure for precursor conversion?

- **By simply eliminating the "carrier" gas (*e.g.,* N_2):**
  - **Total pressure is decreased, and mean free paths are increased.**

- **Some expected consequences of lower pressure are:**
  - **Flow is more molecular & nozzle jetting is reduced (more uniform)**
  - **Diffusivities in gas are increased (more rapid)**
  - **Total gas consumption is reduced (more efficient)**
  - **Total energy consumption is reduced (more efficient)**

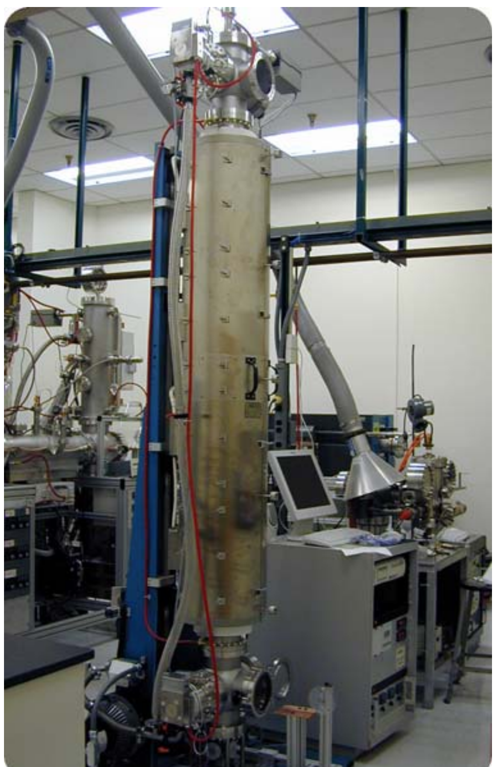
Because low-pressure conditions facilitate more rapid, uniform, and efficient precursor conversion.

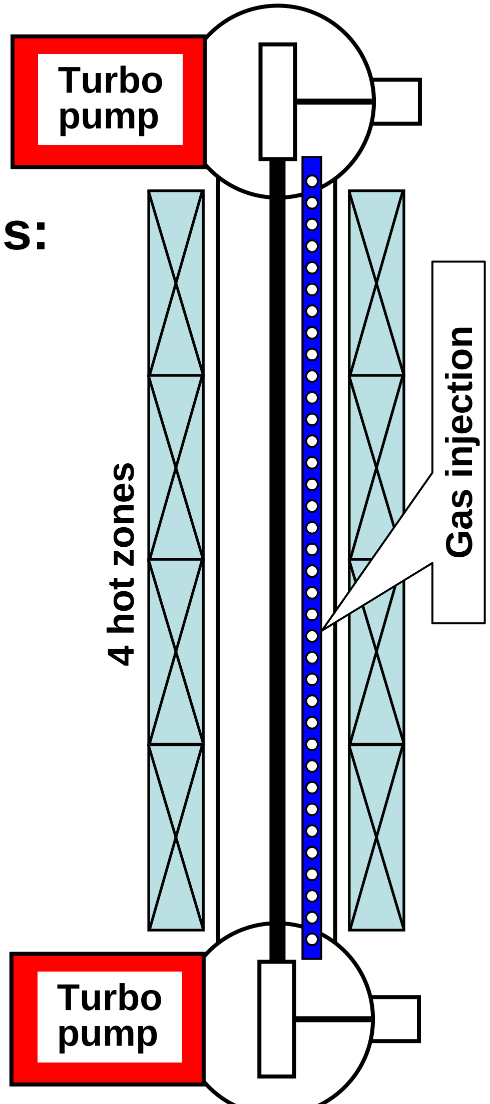
*** LP work also performed at MIT, BNL, CRIEPI (Japan), etc.**

A R2R low pressure conversion system was developed to examine the efficacy of this approach

- **Need to determine if benefits seen in small idealized unit can be replicated in larger system, as well as issues involved in scale-up.**

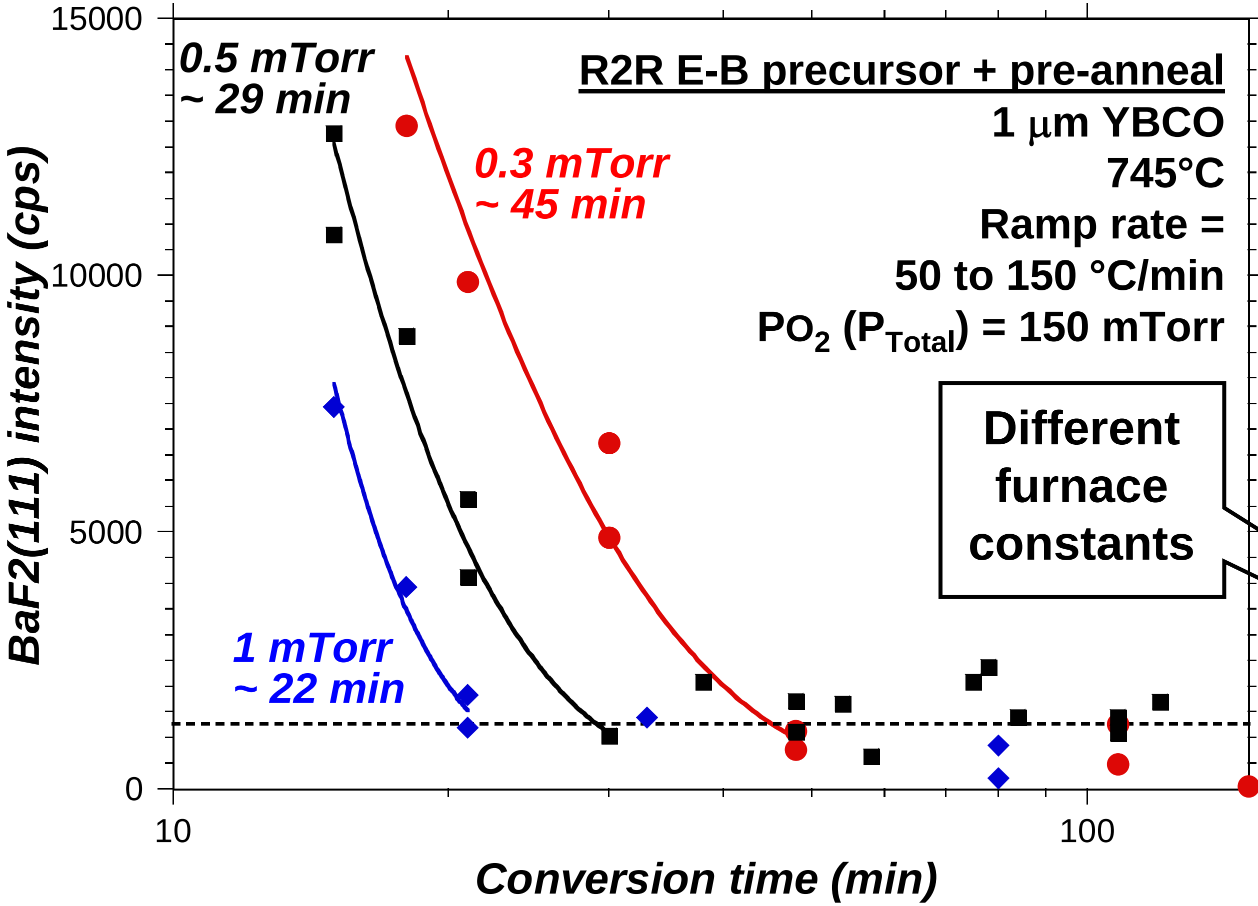
- **Design of R2R system based on lessons learned from our R2R atm and stationary low pressure systems:**





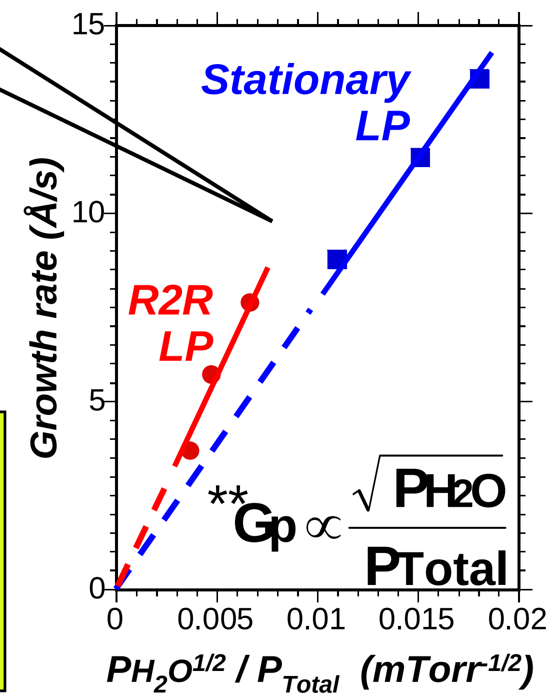
- **4-zone, 2-meter furnace.**
- **Turbo pumps on either end.**
- **Single gas injection tube along length.**
- **P_{base} ~ 1 x 10^{-6} Torr**
- **P_{total} < 1 Torr (O_2 and H_2O).**
- **Aggressiveness increased by higher T, higher P_{H_2O} lower P_{Total}.**

G_p sensitivity to PH_2O is higher in the R2R furnace



	PH_2O (mTorr)	G_p (Å/s)
R2R LP	0.3	3.7
	0.5	5.7
	1.0	7.6
Stationary LP	5	8.8
	10	11.5
	15	13.6

Different furnace constants

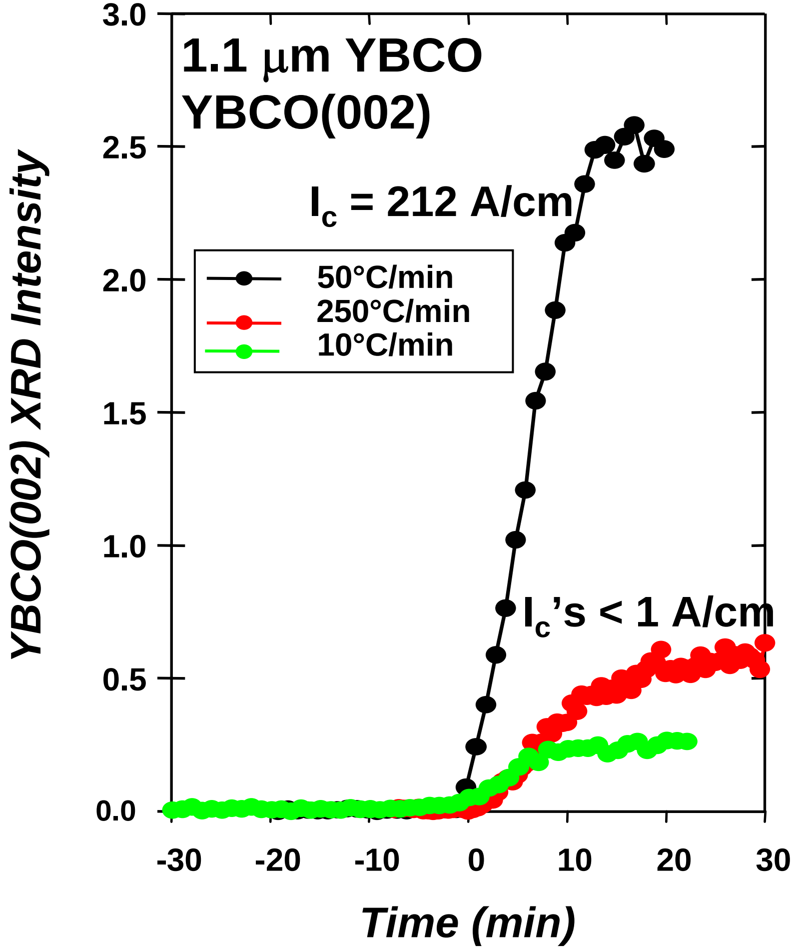


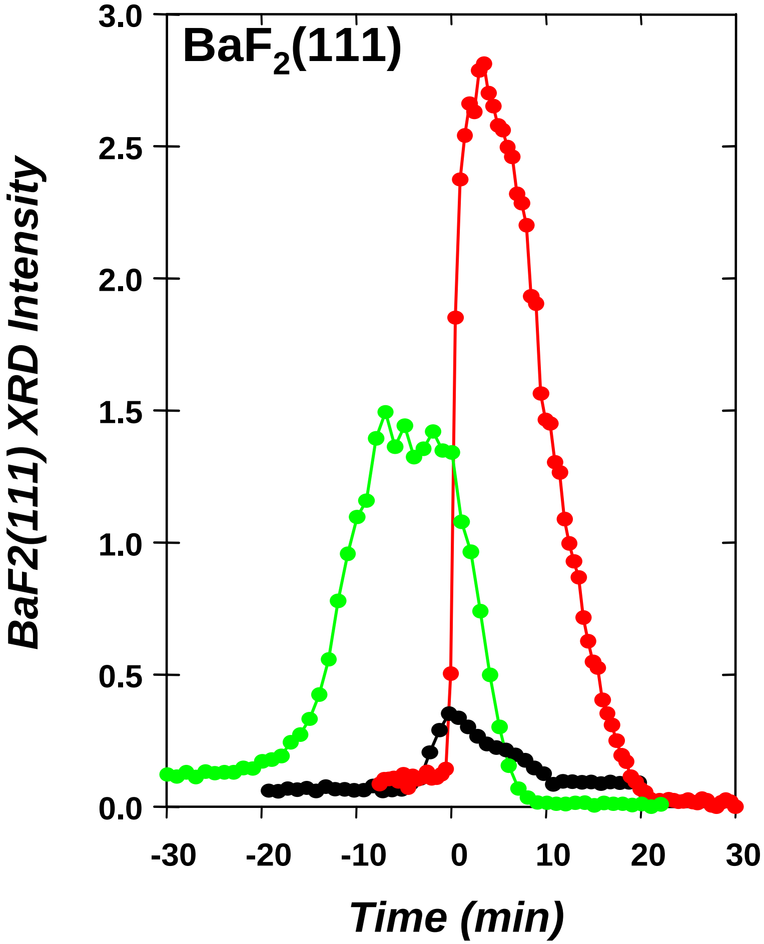
Much lower gas consumption: Decreased by ~1500x compared to our R2R atmospheric system.

May be advantageous to increase P_{Total} to reduce G_p variations associated with PH_2O fluctuations.

** V. F. Solovyov et al., IEEE Trans. Appl. Supercond., 11, 2939 (2001). (BNL group)

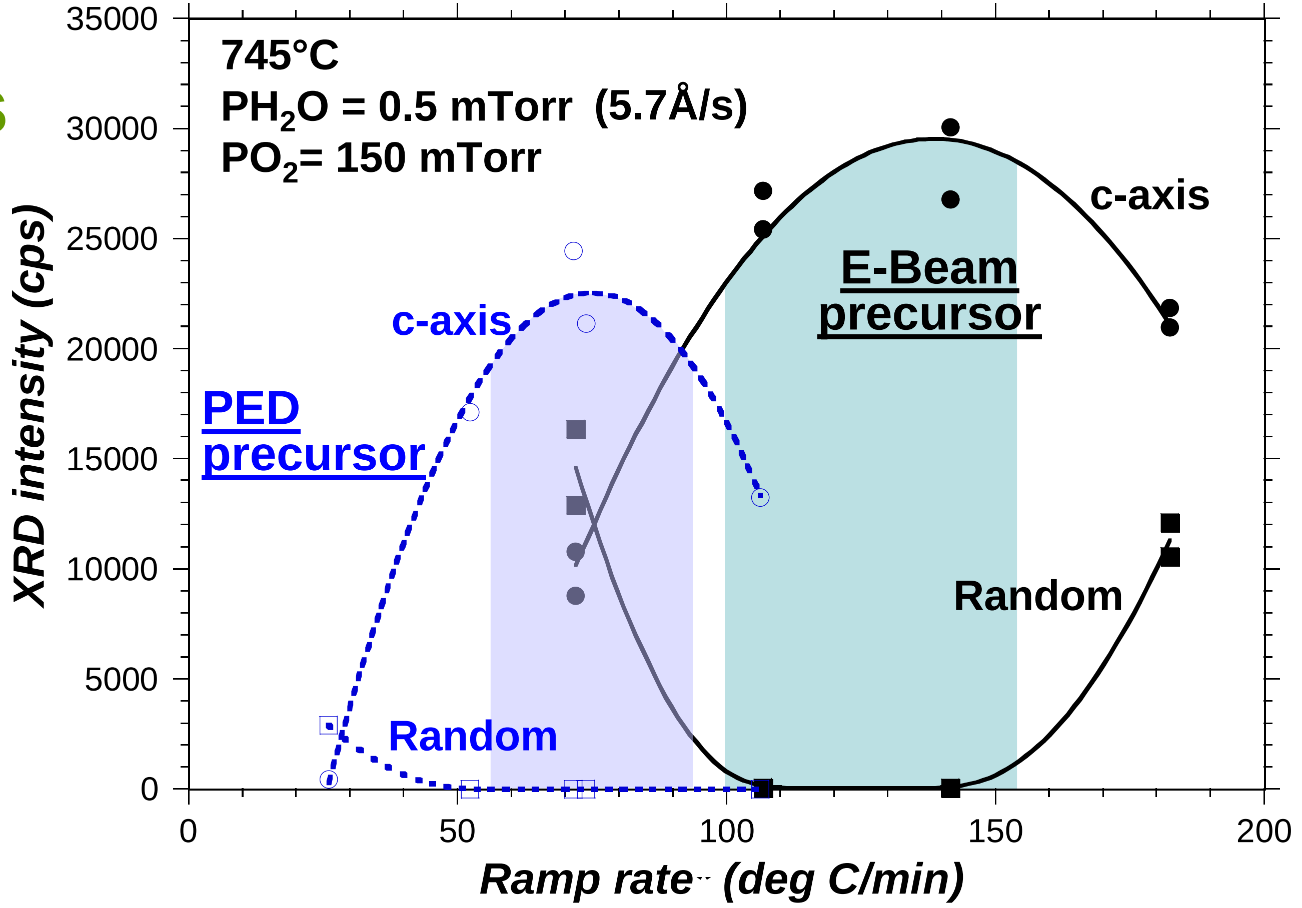
Samples converted in the stationary low pressure unit exhibited strong ramp rate dependency





- This dependency has also been reported for MOD precursors at various pressure ranges by MIT.

Different ramp rate dependencies are found for different precursors

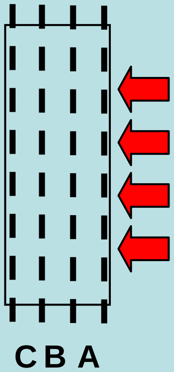


R2R E-B precursor I_c:

1 μm
- **131 A/cm (745°C, 107°C/min, 5.7 Å/s, 38 min)**
- **140 A/cm (745°C, 142°C/min, 5.7 Å/s, 38 min)**
- **148 A/cm (780°C, 107°C/min, 7.6 Å/s, 28 min)**

2 μm → 200 A/cm.

Excellent transverse homogeneity is seen in 1 cm-wide films processed in R2R low pressure furnace



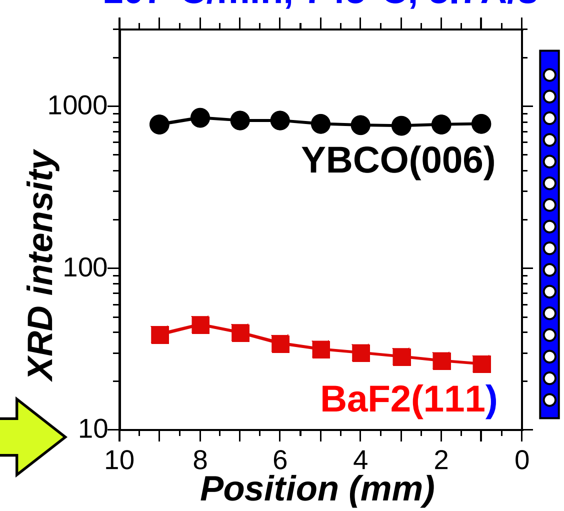
- Non-uniform I_c across the width of samples processed in our <u>R2R atmospheric furnace (>1.5 atm).</u>

R2R atmospheric

Sample	Full 1 cm	A	B	C
I_c/cm (A/cm)	110.0	111.4	95.1	88.6

A 27% variation across tape

R2R Low Pressure

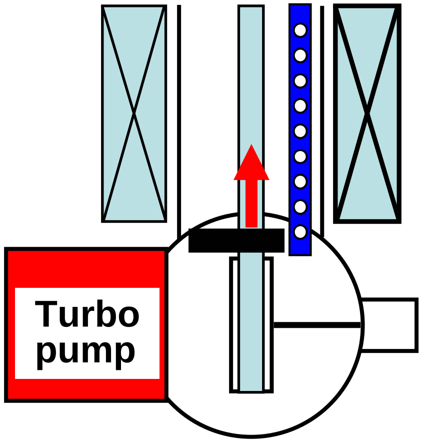


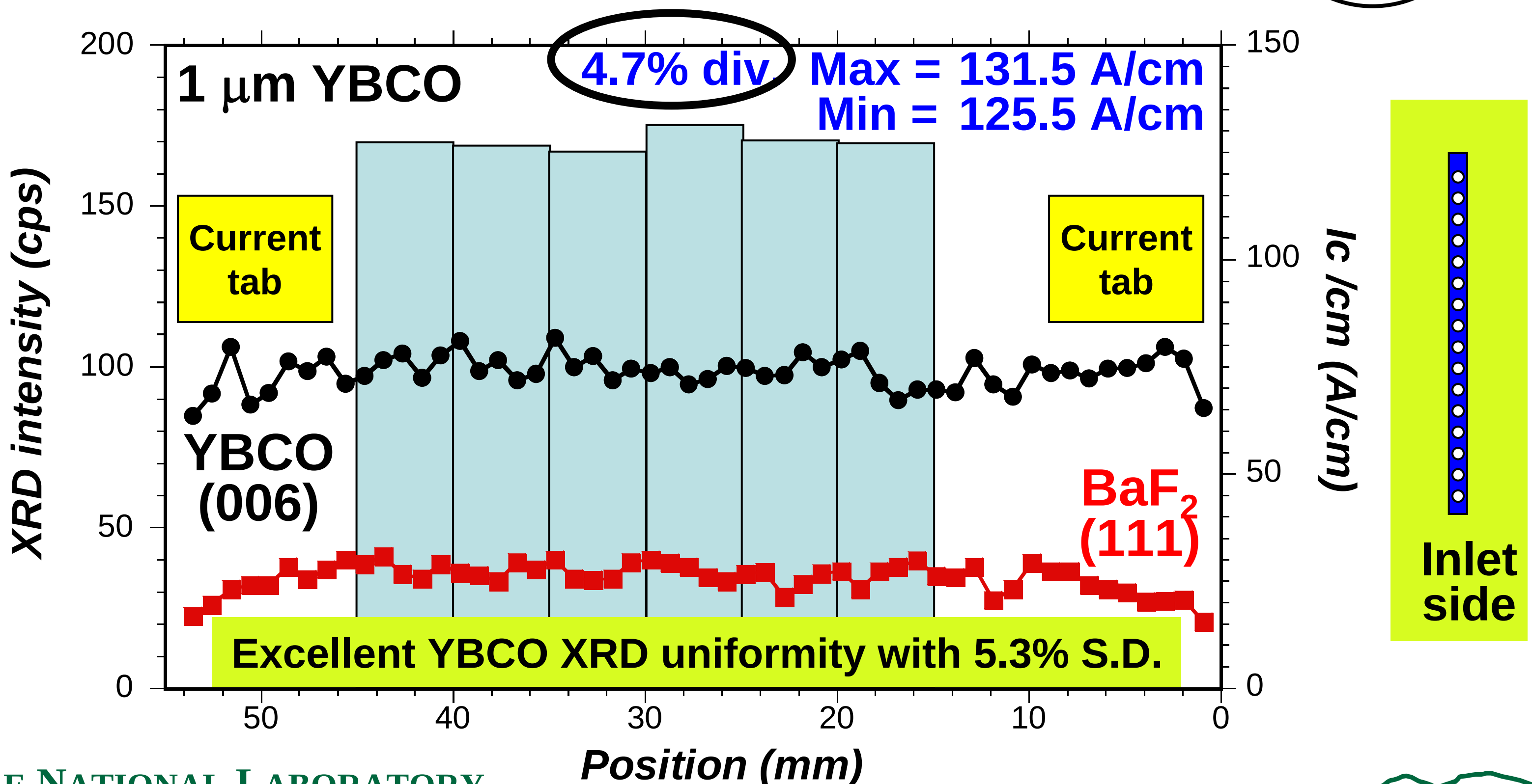
Excellent YBCO(006) uniformity with only 3.9% S.D.

T (°C)	Ramp (°C/min)	G_p (Å/s)	I_c/cm (A/cm) 1-cm	A	B	C	Variations
745	107	5.7	130.9	123.8	124.6	123.0	1.3%
745	<u>142</u>	5.7	139.6	142.3	139.2	138.0	3.1%
<u>780</u>	107	<u>7.6</u>	147.8	147.5	150.6	148.0	2.1%

Same excellent transverse homogeneity is found for even "wider" tape

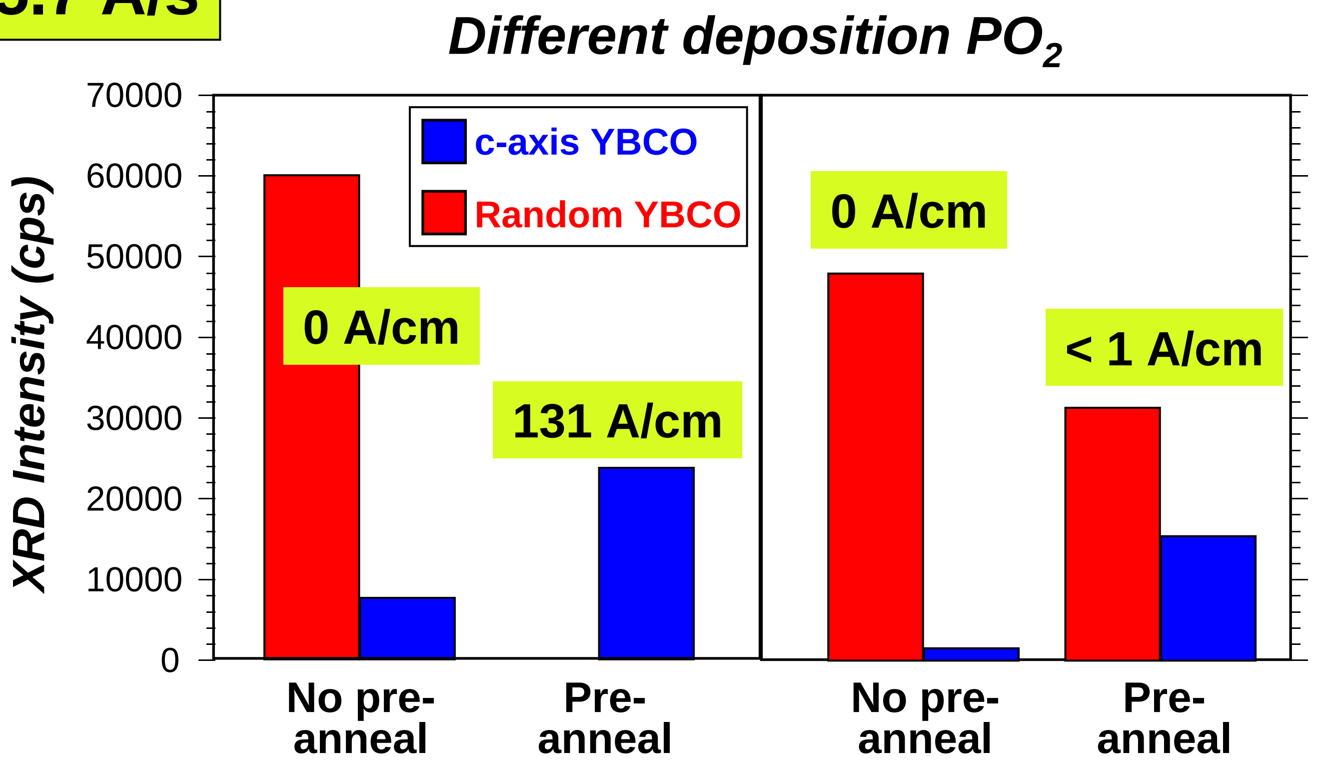
- A 5.5 cm 1 μm sample was welded crosswise to examine transverse characteristic of conversion.
- Sample was converted at 5.7 Å/s.





E-beam precursors deposited under different conditions can exhibit different nucleation and growth behaviors.

G_p ~ 5.7 Å/s



- **With better deposition control and pre-anneal, R2R E-B precursors can tolerate aggressive conversion conditions …**

… However, subtle variations can have consequences on precursor convertibility.

Summary

- **Fast growth rate E-B precursors have been obtained using our R2R deposition system.**
- **A low pressure R2R conversion system has been put in operation.**
  For <u>1 μm</u> YBCO, I_c/cm as high as <u>148 A</u> at G_p of <u>7.6 A/s</u>,
  <u>2 μm</u> YBCO, I_c/cm as high as <u>200 A</u> at G_p of <u>5.7 A/s</u>.
  Performance comparable to stationary low pressure system.
- **Excellent homogeneity was found for samples converted in this system: <u>< 5% I_c variation</u> in transverse direction for 5.5 cm tape.**
- **Significant variations in conversion characteristics are exhibited by different precursors.**

Presentation outline

Ron Feenstra	Development of high-I_c *ex-situ* YBCO conductors
Dominic Lee	Reel-to-reel low pressure furnace development and processing
Hans Christen	Pulsed Electron Deposition of BaF_2 precursors
Bob Hawsey	FY2005 Performance FY2006 Plans Research Integration

In FY04, Pulsed Electron Deposition (PED) was added as a potentially simpler ex-situ precursor deposition technique

Advantages of PED:

- Single target → run-to-run stoichiometry control
- Materials utilization, instrument cost
- Scalability (multiple sources, cost-effective)
- Flexibility (RE-substitutions, etc.)

Three-fold motivation for ORNL:

- Explore a *commercial approach to precursor deposition*
- Develop an *in-house source of precursors* for program needs
- Gain an *understanding of the PED process*

Neocera prototype (2004) integrated with ORNL's R2R tape handling

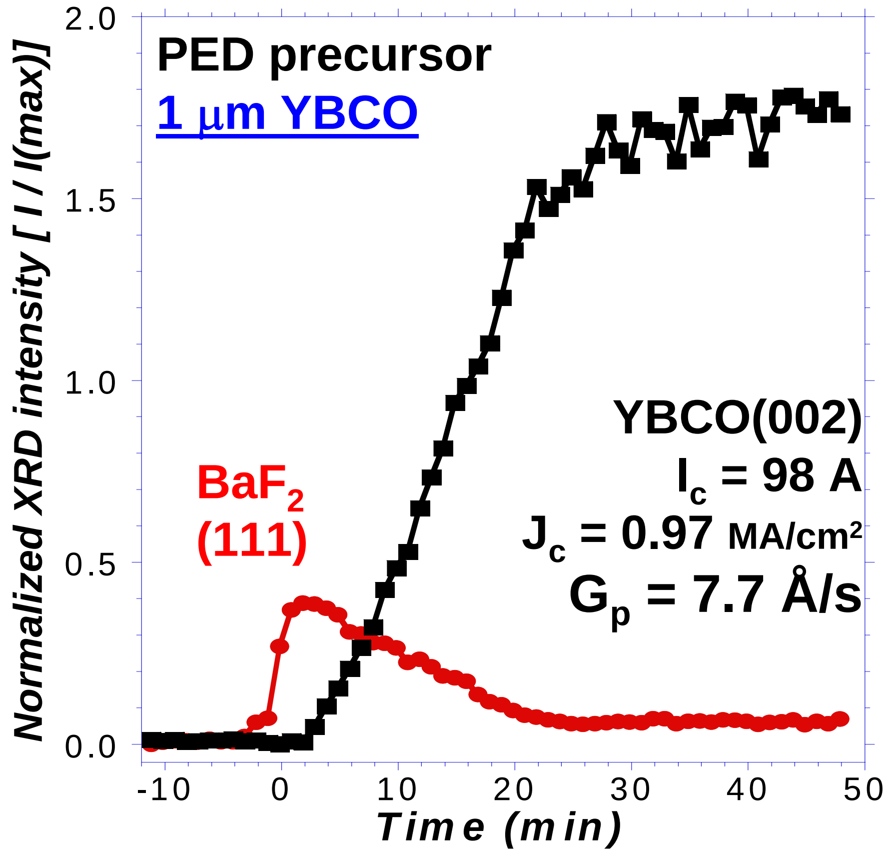
In FY04, we obtained encouraging results on reel-to-reel PED precursors

At slow conversion rates (~ 1Å/s):

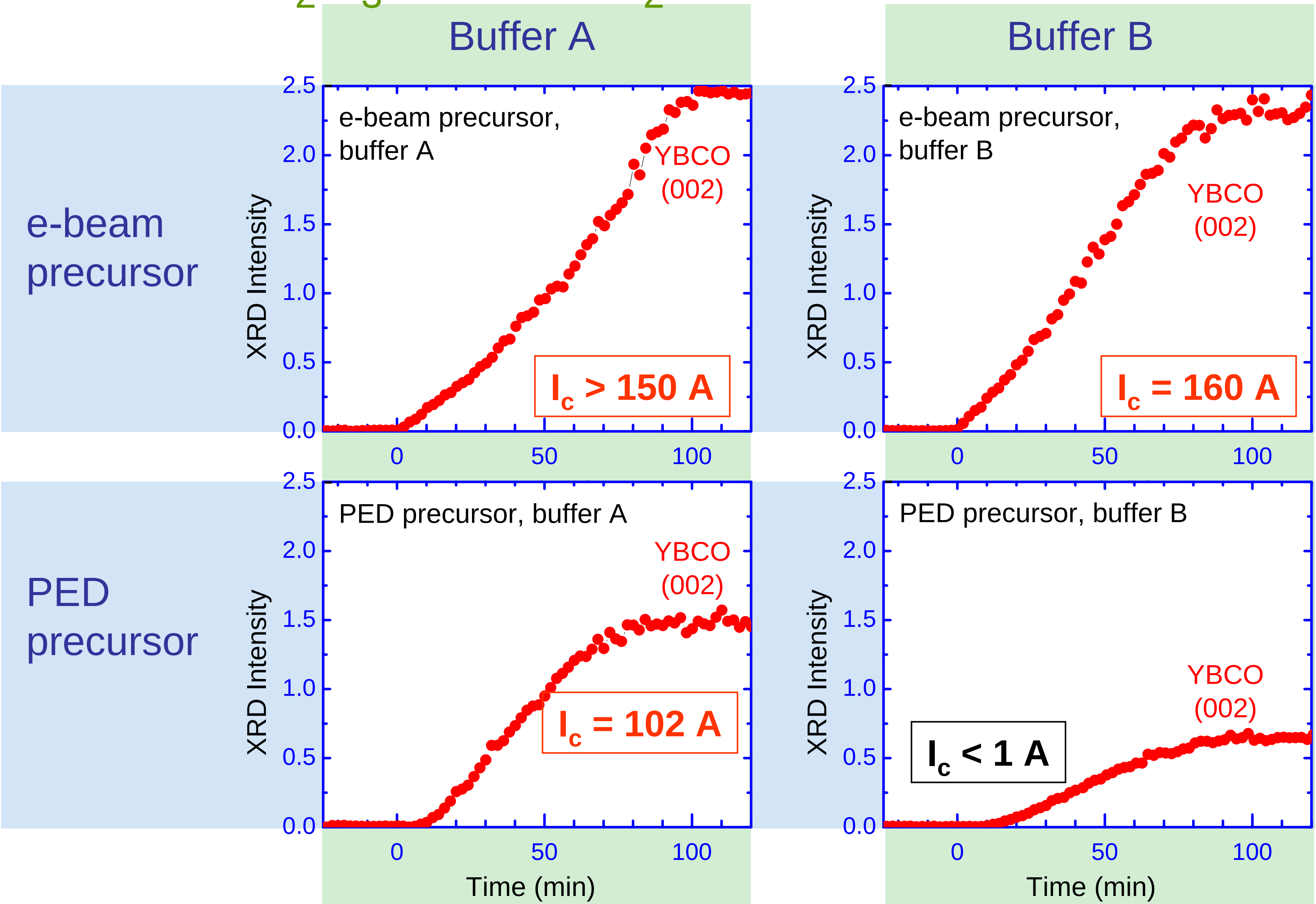
- J_c = 1.6 MA/cm^2 for 0.72 μm YBCO
- I_c = 159 A for 1.3 μm YBCO

Conversion rates of 7.7 Å/s demonstrated (I_c = 98 A for 1 μm YBCO)

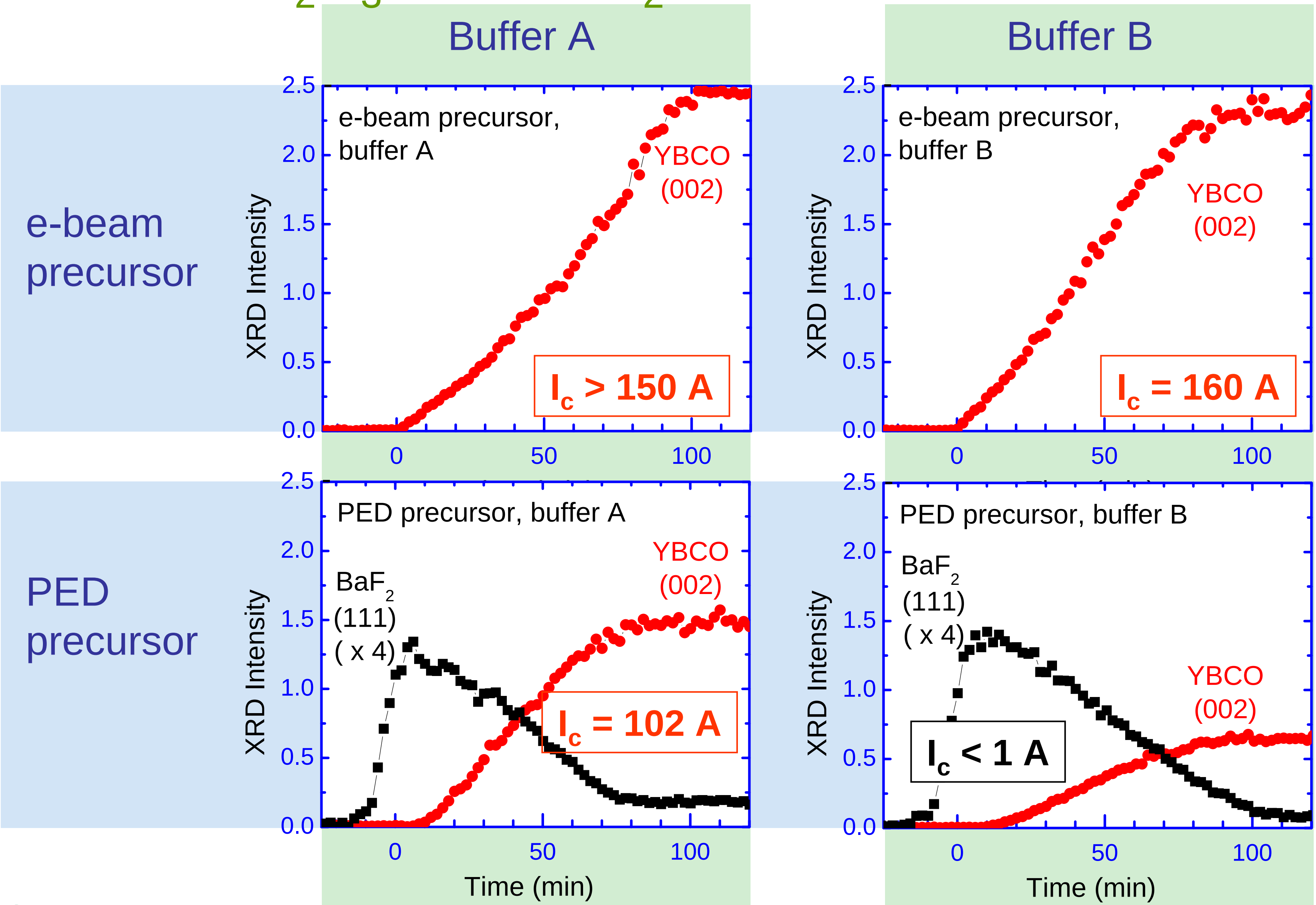
In FY05, we observed an unexpectedly high sensitivity to buffer-layer characteristics.



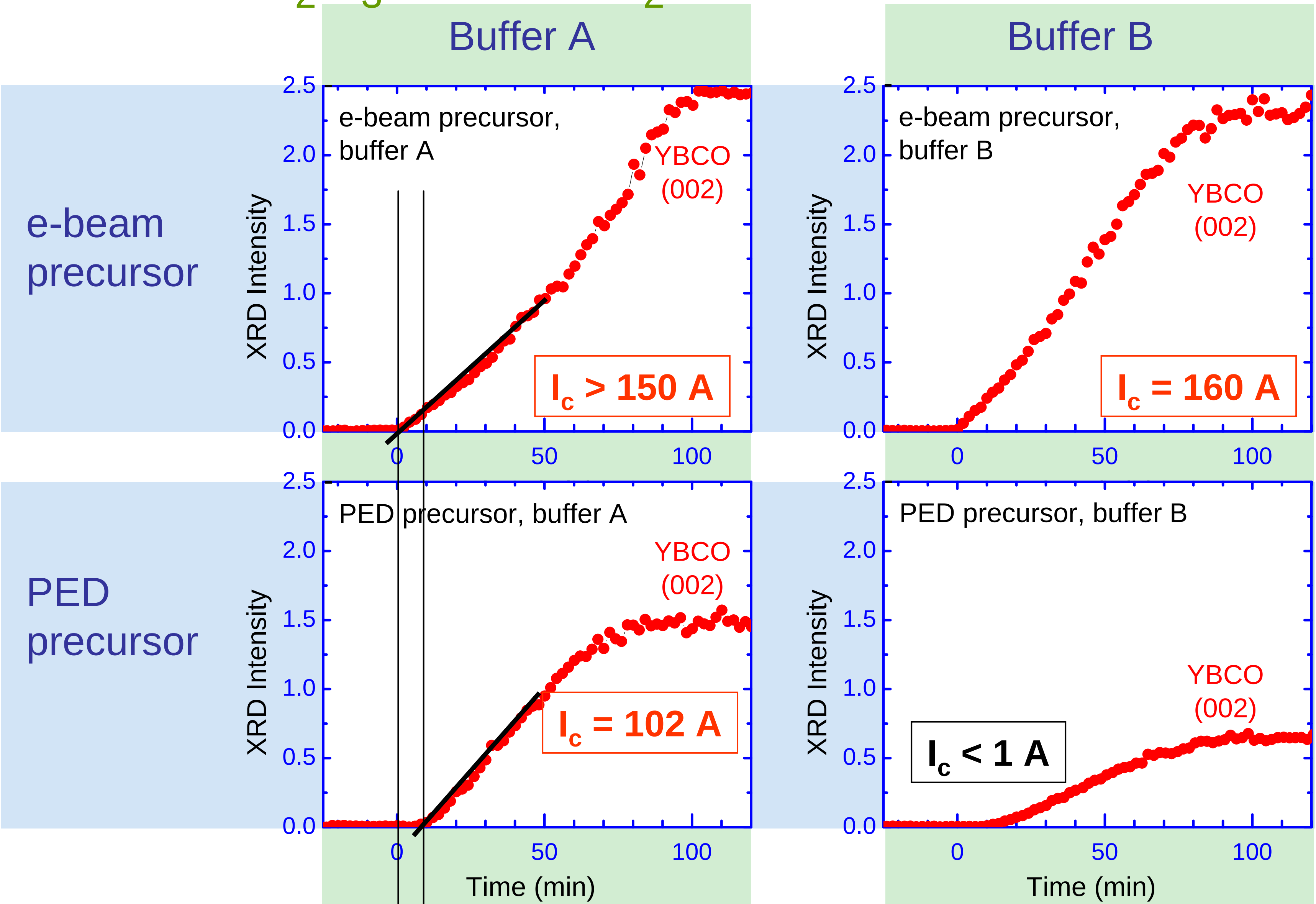
Sensitivity of PED precursors to Y_2O_3/YSZ/CeO_2 buffer characteristics



Sensitivity of PED precursors to Y_2O_3/YSZ/CeO_2 buffer characteristics



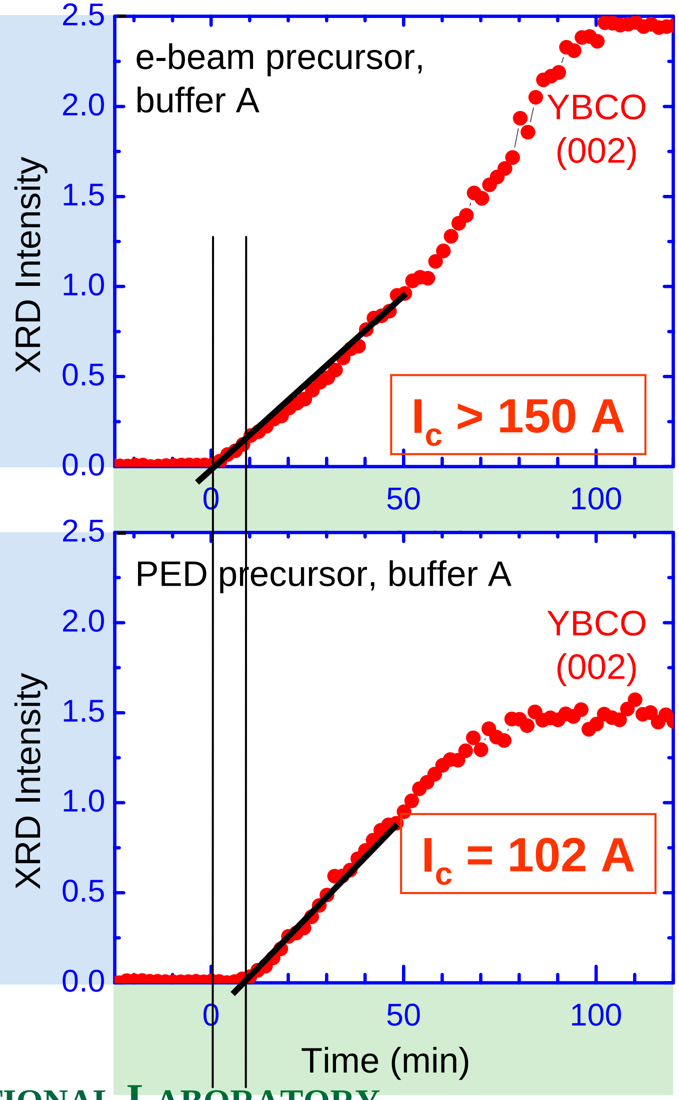
Sensitivity of PED precursors to Y_2O_3/YSZ/CeO_2 buffer characteristics



An "incubation time" (5-15 min) is typical for PED precursors, but not for e-beam precursors

e-beam precursor

PED precursor



Why are PED results highly sensitive to buffer layer characteristics?

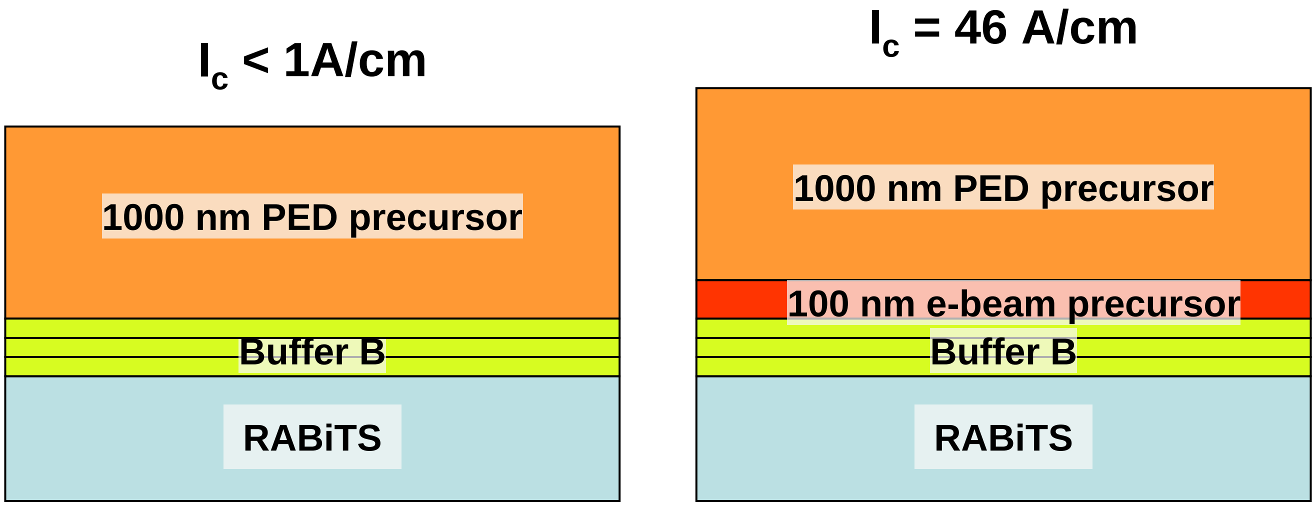
- Incubation:

  Does buffer B not tolerate incubation?
- PED particulates (SEM):

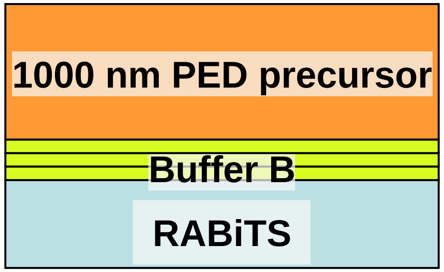
  Does buffer B suffer damage during PED process?

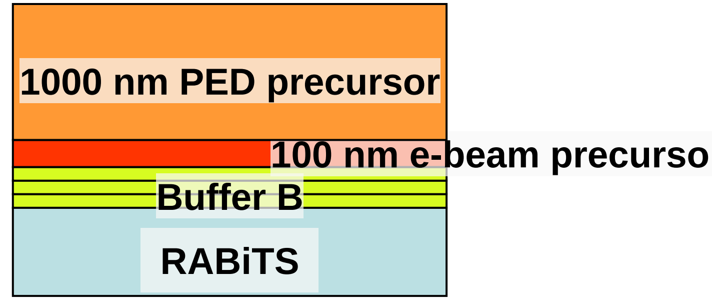
Why are PED results highly sensitive to buffer layer characteristics?

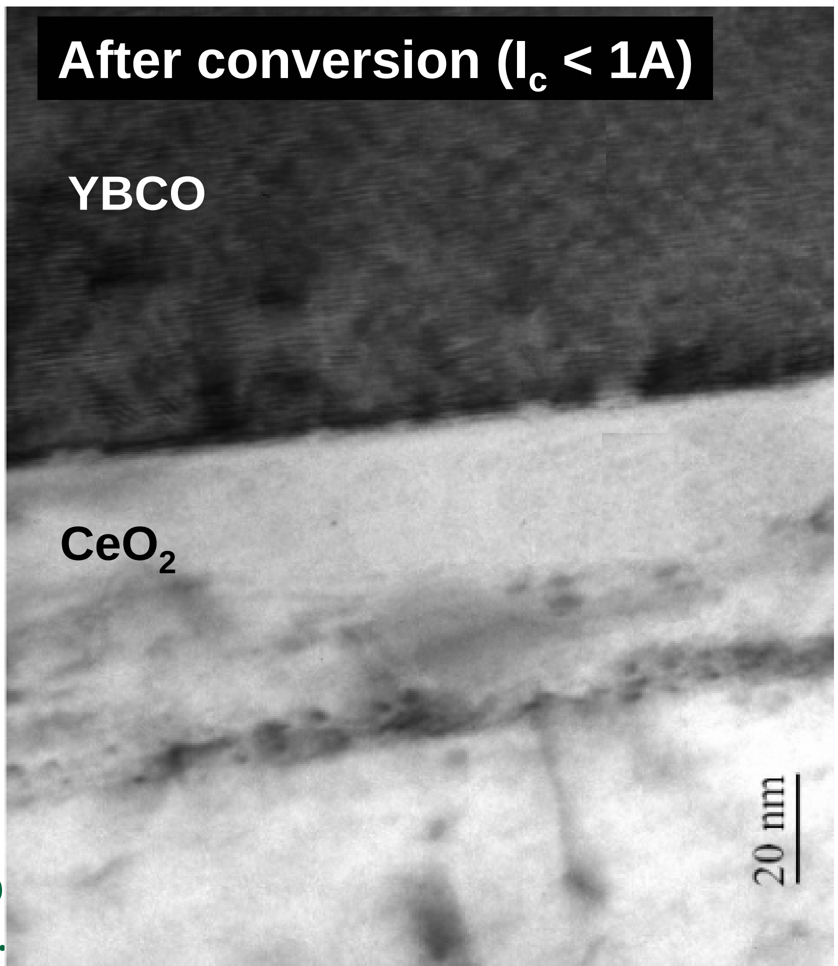
Test structures:

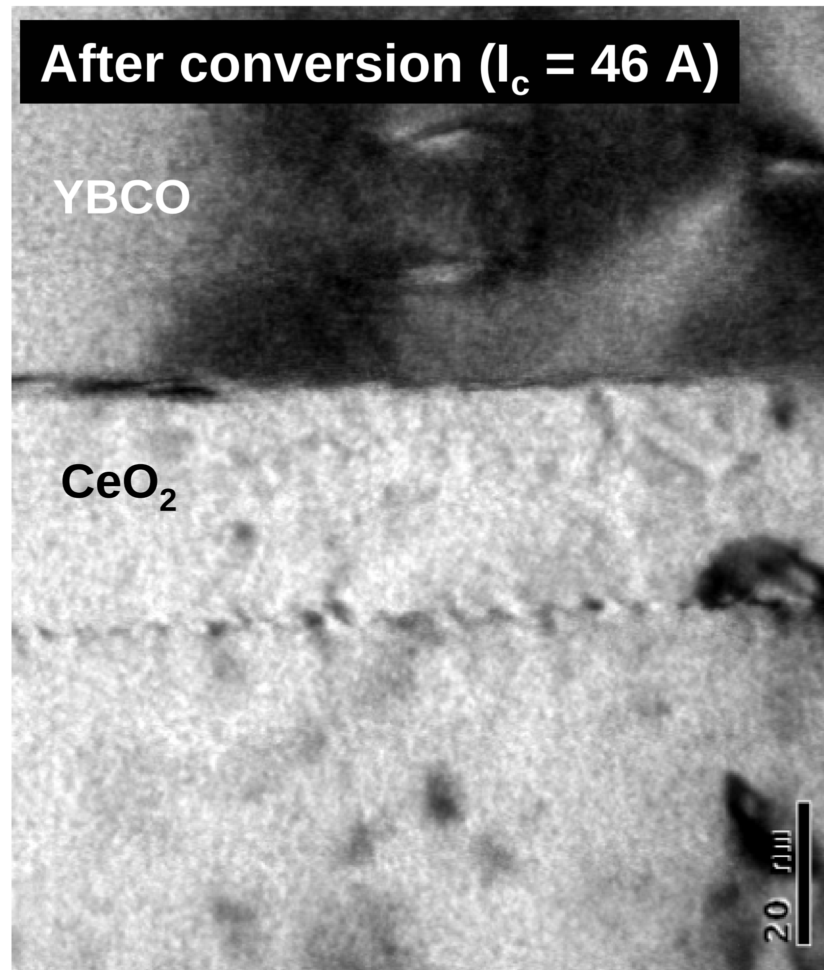


No detectable damage at PED precursor / CeO_2 interface

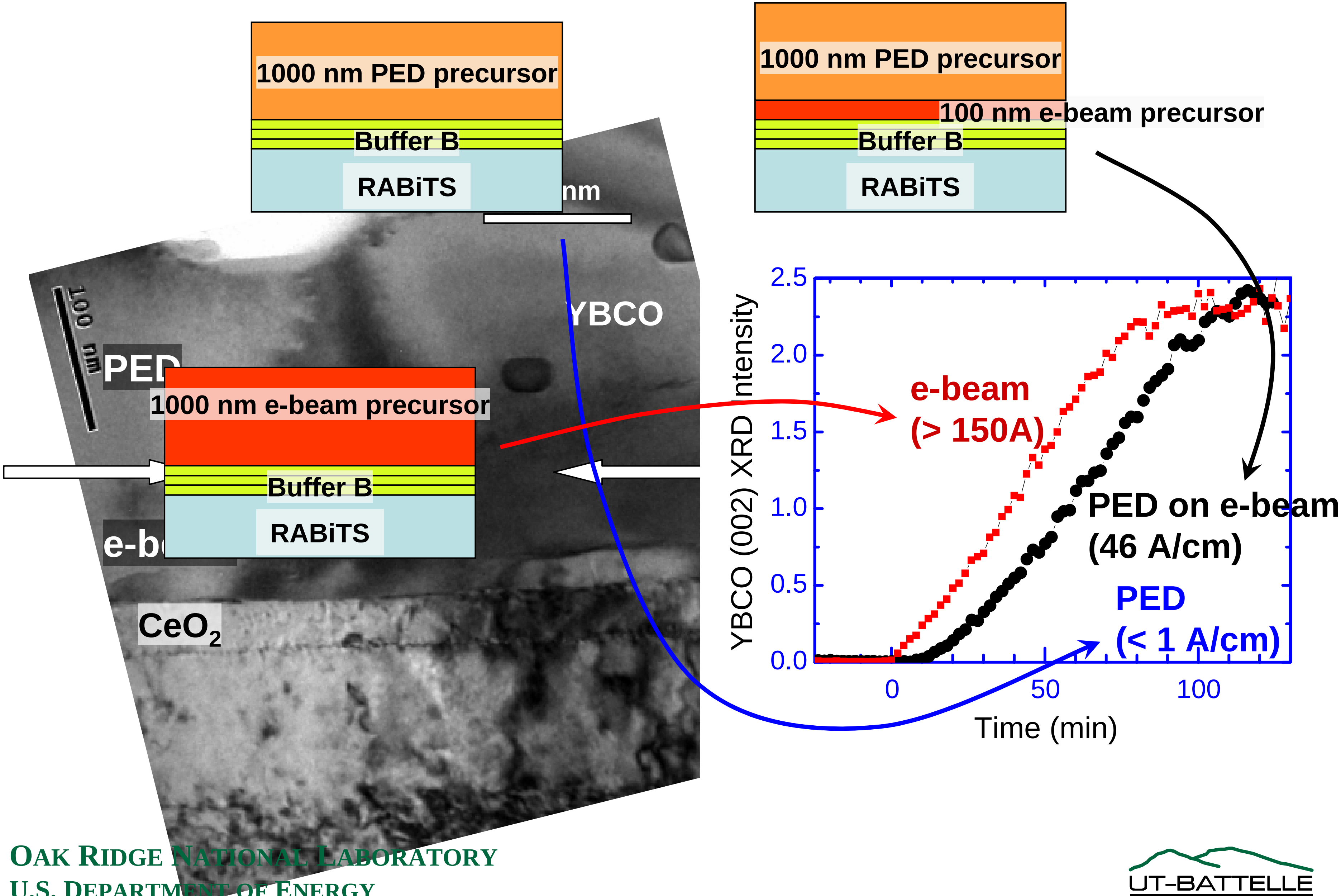








Also: no visible interface between converted e-beam and PED YBCO



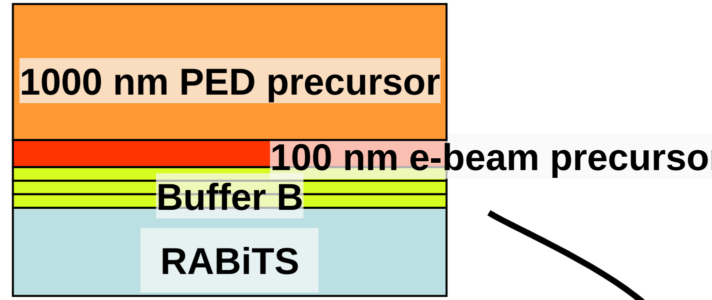
UT-BATTELLE

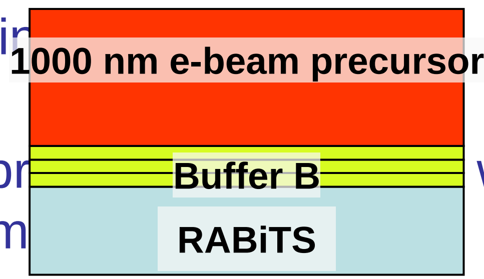
Incubation time remains even with e-beam “seed” layer

Incubation is not related to nucleation, but possibly to diffusion

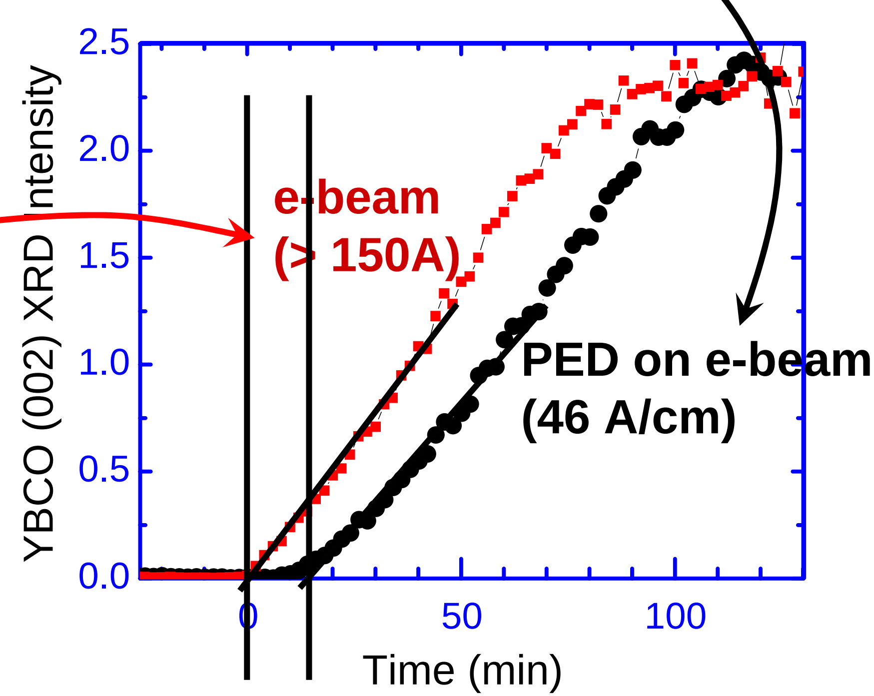
46-A/cm sample shows same incubation time as <1-A/cm sample;

therefore, the incubation time does not appear to be the dominating factor leadi





PED pr well on e-beam

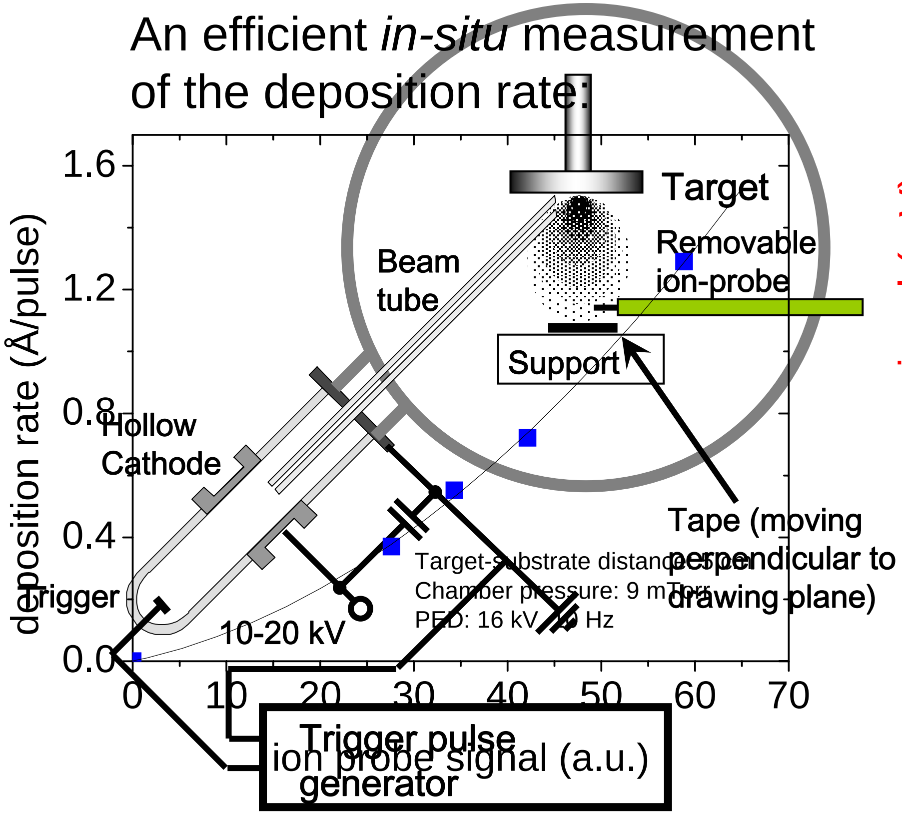


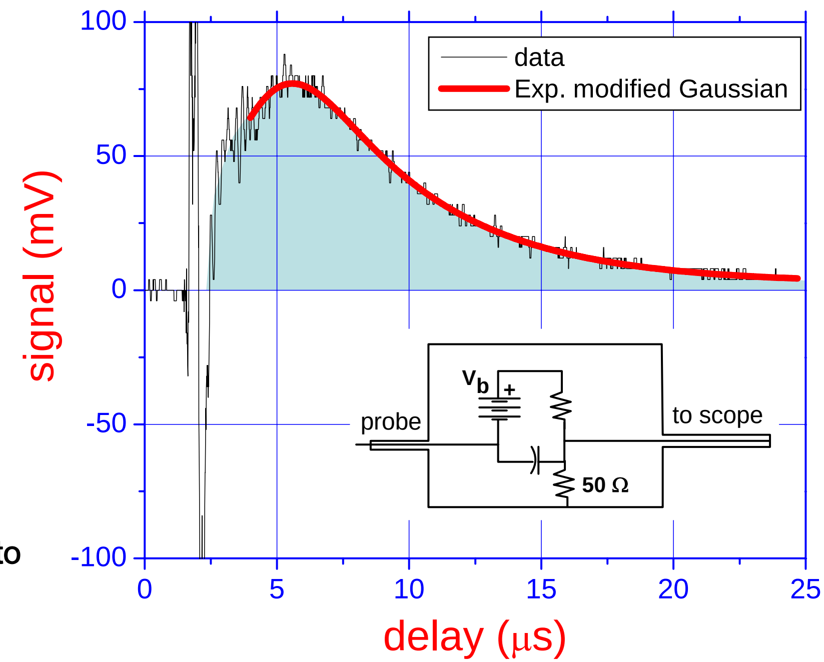
therefore: poor crystallization of PED precursor (without “seed”) appears related to poor nucleation

A closer look at PED energetics

An ion probe can be used to measure the energy of impinging species:

An efficient *in-situ* measurement of the deposition rate:



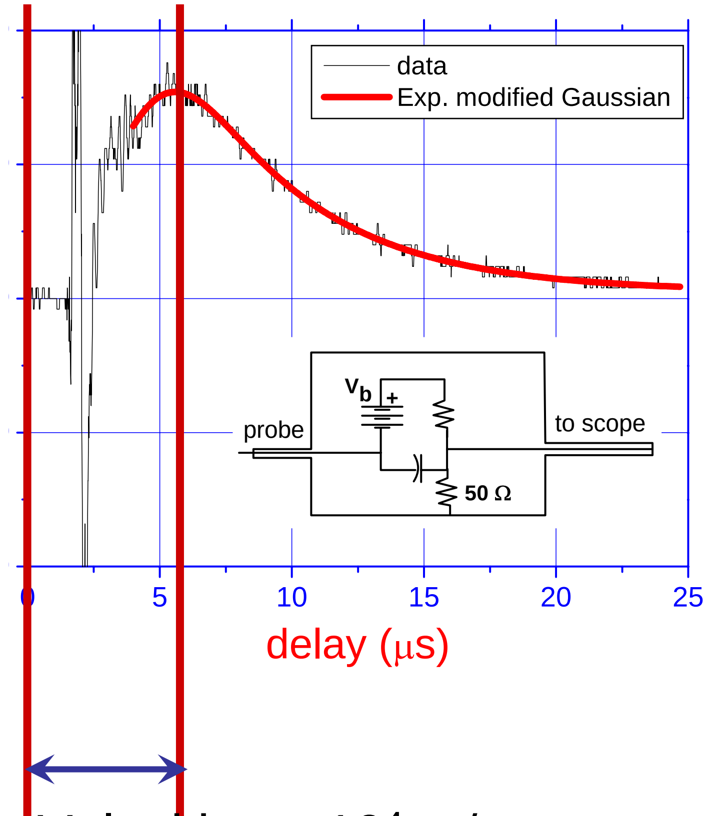


Monitors the *combined* effect of target wear and source performance

A closer look at PED energetics

An ion probe can be used to measure the energy of impinging species:

- **High energy of incident species**
  Comparison: in pulsed *laser* deposition, re-sputtering and poor crystallization are often observed at these energies.
- **Reduction of energy requires**
  - **increased background gas pressure (impossible in PED)**
    or
  - **increased target-substrate distance (results in deviation from stoichiometry for PED)**
- **There is no easy way to address this issue.**



Velocities ≈ 10^4 m/s
→ energies 10 – 80 eV

Summary of PED results

- PED: Sensitive to buffer characteristics
- "Incubation time" during conversion of PED precursors
    - Possibly related to diffusion
- E-beam "seed" layer improves crystallization of PED precursors
    - Poor crystallization of PED precursor (on some CeO_2 layers) seems related to nucleation
- No TEM-detectable damage to the CeO_2 layer from PED process
- PED: impinging species have high kinetic energies (10 – 80 eV)
    - Can't use pressure or distance to reduce these energies

Presentation outline

Ron Feenstra	Development of high-I_c *ex-situ* YBCO conductors
Dominic Lee	Reel-to-reel low pressure furnace development and processing
Hans Christen	Pulsed Electron Deposition of BaF_2 precursors
Bob Hawsey	FY2005 Performance FY2006 Plans Research Integration

FY2005 Performance (Scoring Criterion)

High-I_c *ex-situ* YBCO conductors

Plan:

☑ Study and improve the thickness dependence of J_c in PVD-BaF_2 *ex-situ* films on RABiTS

Performance:

- Performed magnetometry J_c study in the thickness range 30 nm to 1.5 µm for *ex-situ* YBCO on RABiTS
  - observed maximum in J_c for thicknesses between 0.1-0.2 µm with record high J_c value of 3.5-4 MA/cm^2 (77 K)
- improved performance of 1-1.5 µm thick films on RABiTS by Y-doping
  - I_c = 300 A/cm in 1.0 µm YBCO
  - I_c = 370 A/cm in 1.4 µm YBCO
- improved performance of 2 µm YBCO from 200 $\rightarrow$ 330 A/cm

FY2005 Performance (Scoring Criterion)

High-I_c *ex-situ* YBCO conductors (continued)

Plan:

☑ Added objective: Elucidate role of precursor preparation history on the *ex-situ* conversion process

Performance:

- Performed quenching study of YBCO nucleation during temperature ramp and early parts of the *ex-situ* conversion anneal
- Initiated external collaborations to enable characterization with a variety of techniques
- Identified perturbing effects on the growth during fast conversion of thick as-grown precursors:
  - layered phase separation (Cu $\leftrightarrow$ BaF_2)
  - incomplete Cu oxidation
- Observed minor dependence of conversion rate on precursor thickness

FY2005 Performance (Scoring Criterion)

High-I_c *ex-situ* YBCO conductors (continued)

Plan:

- [x] Study and improve flux pinning properties of PVD-BaF_2 *ex-situ* YBCO

Performance:

- Performed study as a function of YBCO thickness and temperature
- Identified general characteristics for pinning with H||c:
  - constant $J_c \propto H^{-\alpha}$ power law exponent α with values of ~ 0.6, consistent with pinning by strong, sparse defects
  - scaling between self-field J_c at 77 K and in-field performance at reduced T (30-40 K)
- Achieved improvements in pinning for H||c and H||(ab) by processing modifications and Y-doping (WDG presentation)
- *Plan to study RE-BCO was deferred to FY2006 in lieu of expanded compositional studies in the YBCO system*

FY2005 Performance (Scoring Criterion)

R2R e-beam precursor deposition

Plan:

- [x] Obtain precursors that can tolerate fast G_p.

Performance:

- Combined deposition control with pre-anneal treatment.
- Increased G_p with high I_c.

FY2005 Performance (Scoring Criterion)

Develop a R2R low-pressure furnace

Plan:	Performance:
☑ Develop a R2R low-pressure furnace	R2R low pressure furnace was put in operation (~1500x reduction in gas consumption compared to our R2R atm).
☑ Process ex-situ precursors with high I_c and G_p.	For 1 mm YBCO, 150 A/cm at G_p = 7.6 Å/s For 2 mm YBCO, 200 A/cm at G_p = 5.7 Å/s (previous R2R G_p from 0.7 to 1.5 Å/s).
☑ Demonstrate improvement in YBCO homogeneity.	Excellent transverse Ic homogeneity was found: I_c/cm variation ~ 2 to 3 % for 1 cm-wide < 5% for 5.5 cm-wide (Previous R2R: I_c/cm variation of up to 27%).

FY2005 Performance (Scoring Criterion)

PED precursor development

Plan:

- ☒ Further develop R2R PED as a routine precursor deposition tool.
- ☒ Optimize precursor to enable fast G_p with high I_c.

Performance:

- PED vs. e-beam: Significant differences in conversion characteristics observed.
- Sensitivity in YBCO nucleation to buffer condition.
- Substantial incubation period
  - regardless of buffer condition,
  - also observed in samples with an e-beam evaporated precursor seed
- Measured high energies of deposited species, which may result in damage to certain types of CeO_2 surfaces.

Ex-situ YBCO Strategic Research: Taking Stock

The ex-situ processing approach has historically been instrumental at ORNL

– In demonstrating feasibility of ex-situ conversion as a YBCO method.

– In demonstrating that high I_c RABiTS-based 2G wires can be obtained in reel-to-reel processed lengths.

– As a method to qualify alternative RABiTS architectures.

– And ORNL's CRADA partners (past and present) selected the R2R conversion approach and have used ORNL's facilities.

Ex-situ precursors are useful for strategic research

- US companies have made tremendous progress;
  reel-to-reel processed, world-class 2G wires in long lengths were announced at this peer review.
- In particular, 4 cm-wide long-length 2G wires processed into 4-mm wide tapes with high uniform I_c have been obtained using an ex-situ approach (MOD precursor) by American Superconductor.
- Ex-situ films can be made thicker and with high I_c.
- We no longer need to pursue other YBCO growth methods as an alternative to processes being pursued by industry.
- PVD precursors are a flexible research tool, with processing and microstructural features that may be compared to MOD such that progress can be accelerated.
- Resources are required by new and expanded strategic CRADA commitments.

These factors prompted us to reprioritize our new and existing strategic research portfolio.

FY2006 Plans (Scoring Criterion)

R2R PED:

- Motivated by PED's potential as low-cost alternative to MOD.
- Pursued as in-house source of precursor material.
- Discovered an unexpected sensitivity of nucleation to buffer condition
- Stop work.

R2R e-beam evaporation:

R2R e-beam evaporation is no longer essential to the ORNL mission.

We plan to substantially reduce our effort to a level that is consistent with our ongoing commitments.

- *R2R e-beam evaporation* → Stop work.
- *Stationary*: Focused efforts in support of WDG and CRADA

FY2006 Plans (Scoring Criterion)

High-I_c ex-situ YBCO conductors:

- Implement results from continued precursor studies to improve Ic in 2 µm thick YBCO on RABiTS to values > 500 A/cm (77 K, sf)

  - processing focus will shift from 1 → 1.5-2 µm

- Study effects from additions to YBCO to improve transport properties of 1.5-2 µm thick films using a 4th evaporation source

- Perform exploratory studies on alternative RE-BCO film growth

  - compare difficulty of processing with YBCO

Research Integration (scoring criterion) (1 of 2)

- Wire Development Group (AMSC, UW, LANL, ANL, ORNL)
  - vortex pinning, current limiting mechanisms, characterization
- American Superconductor Corporation
  - Conductor fabrication (CRADA)
- Neocera, Inc.
  - Pulsed Electron Deposition: Source development
- University of Tennessee
  - PVD-BaF_2 *ex-situ* process, J_c characterization, pinning studies
- Albany NanoTech
  - multi-technique characterization of quenched precursors
- NIST-Gaithersburg
  - phase relations of BaF_2 *ex-situ* process
- NIST-Boulder
  - mechanical properties of thick YBCO-RABiTS conductors

Research Integration (scoring criterion) (2 of 2)

- ANL
  - Raman studies of quenched precursors
- BNL
  - information exchange, site visit
- ICMAB, Barcelona, Spain
  - magnetometry of high-Jc films on RABiTS
- Univ. Cambridge, UK
  - GB studies
- Embry-Riddle Aeronautical University
  - PED precursor characterization

- 15 peer reviewed scientific publications
- National and international conference contributions

OAK RIDGE NATIONAL LABORATORY
U. S. DEPARTMENT OF ENERGY

Available quality of coated conductors enables *and* necessitates conductor research with industry:

- Multi-filamentary HTS fabrication and characterization
- Stabilizer geometry suitable for low ac loss applications
- Low aspect-ratio 2G wires for fully transposed reduced ac loss cables and coils