Continuous Processing of YBCO Coated Conductor

OAK RIDGE NATIONAL LABORATORY

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EE/RE Funding

- **FY 2002: $1.4M ; FTE 4.0; Post-Docs 1.5**

Industrial Collaborators

- **3M Company (3M) - Myles Brostrom, Dick Ericson, Dave O'Neill, Bill Robbins, Chris Shelton, Jonathan Storer, Sheryl Vanasse, Badri Veeraraghavan, Ed Yu**
- **American Superconductor Corporation (AMSC) - S. Annavarapu, Q. Li, Marty Rupich, Bart Riley, U. Schoop, C. Thieme, Darren Verebelyi, Z. Zhang**
- **MicroCoating Technologies (MCT) - Adam King, Todd Polley, Shara S. Shoup, Marvis White**
- **Oxford Superconducting Technology (OST) - Ken Marken**
- **Neocera – K. Harshavardhan**

National Laboratory Collaborators

- **Los Alamos National Lab - Paul Arendt, Steve Foltyn**
- **NIST (Boulder) – N. Cheggur, Jack Ekin**

FY 2002 Plans

- **Long-length RABiTS development:**
  - Improve texture of metal substrates (Ni and alloys).
  - Demonstrate *uniform* epitaxial seed layers using sulfur.
  - Prepare long-length *strengthened* RABiTS.

- **"Large-area" ex-situ YBCO conversion:**
  - Determine the effectiveness of reduced pressure in aiding conversion.
  - Investigate *large-area batch* conversion parameters for YBCO precursor.
  - Compare conversion parameters for different precursors.
  - Obtain 1 MA/cm^2 on meter-length 3000Å-thick YBCO on *strengthened* RABiTS.
  - Study basic *reel-to-reel* conversion parameters for *thick* YBCO precursor.

- **External collaborations:**
  - Continue to assist partners in achieving their programmatic goals.
  - Provide collaborators with RABiTS and YBCO coated conductors.

OUTLINE

- **FY 2002 Results**
  - Status of ORNL ACCI Laboratory
  - Long-length RABiTS developments — Fred List
  - Low pressure (~200 mTorr) precursor conversion
  - Reduced pressure (~0.3 atm) precursor conversion — Dominic Lee
  - Reel-to-reel *ex-situ* YBCO conversion
- **Summary of FY 2002 Results / FY 2003 Plans**
- **Research Integration**

ORNL ACCI Laboratory: New tools and progress

ACCI Overview

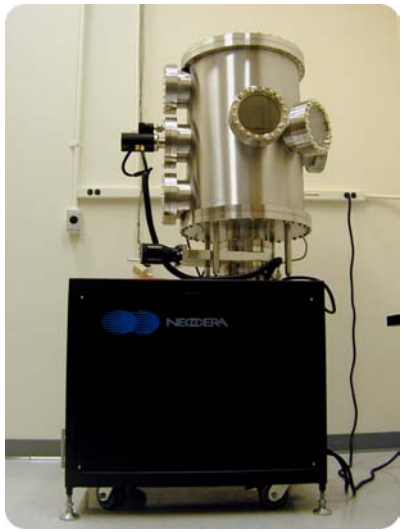
- ACCI: Accelerated Coated Conductor Initiative
- Purpose: to accelerate the development, commercialization, and application of HTS through joint efforts among DOE labs, American industry, and universities.
- Lab Space: ~2200 sq. ft. (prepared FY2001)

Some ORNL ACCI equipment is in early stages of operation.

⇐ Four-high rolling mill w/slitter for metal substrate fabrication.

Pulsed Electron Beam ⇒ Deposition (PED) system for YBCO deposition.

Neocera

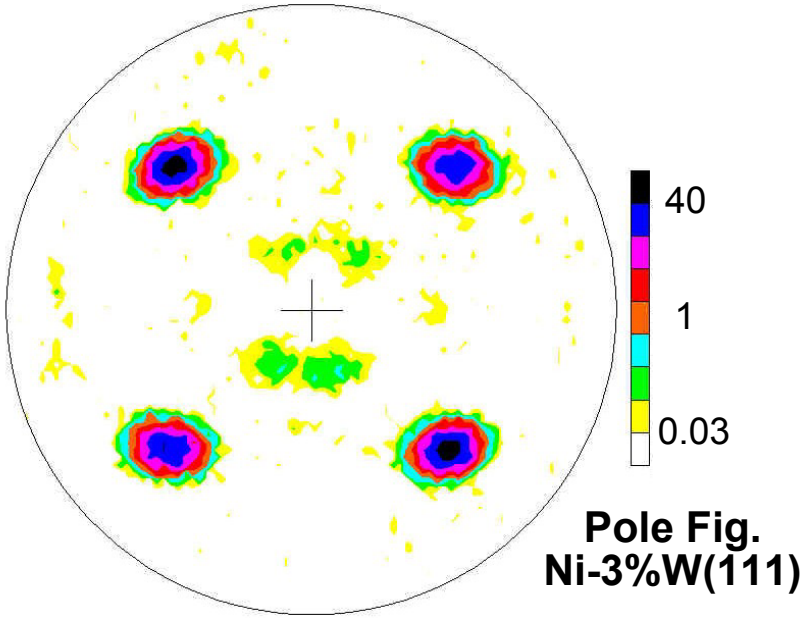


⇐ Reel-to-reel steam cleaner for metal tape cleaning.

9T refrigerated magnet ⇒ system for conductor characterization.

New XRD system reveals excellent texture for strengthen Ni-3%W alloy tape.

- Dedicated to HTS program
- Start of Operation: 03/01/02
- Total tape: **>400 m** (7/14/02)
- Users: ORNL, 3M, AMSC, MCT

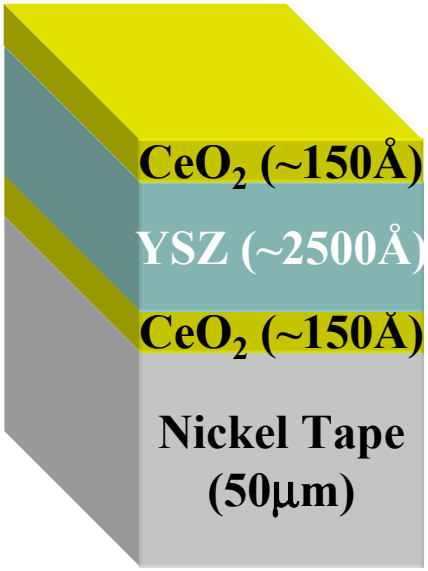


Pole Fig.
Ni-3%W(111)

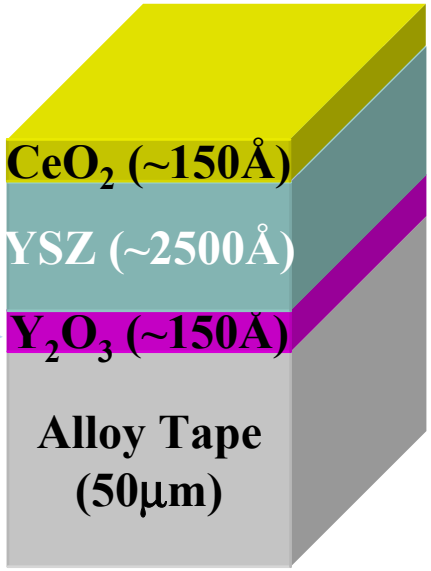
Ni-3%W (99.0% cube)
$\Delta\omega = 5.5°$ for $\phi = 0°$
$\Delta\omega = 8.7°$ for $\phi = 90°$
$\Delta\phi = 8.0°$

Y_2O_3 has replaced CeO_2 as the preferred seed layer for ORNL RABiTS.

2001 Standard RABiTS



2002 Standard RABiTS



seed layer

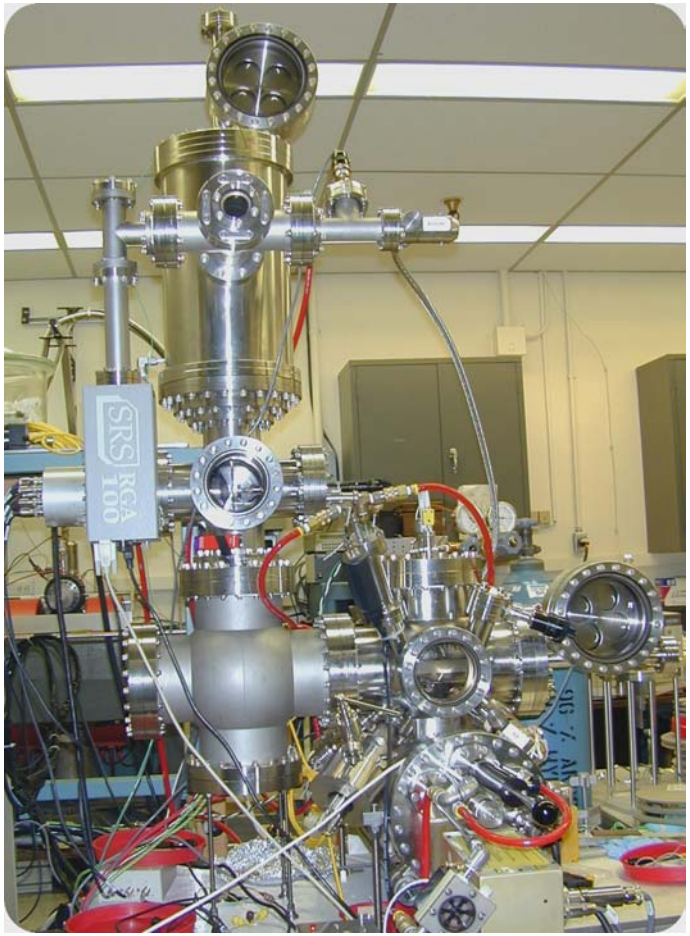
CeO_2 seed layer <u>sometimes</u> develops cracks.

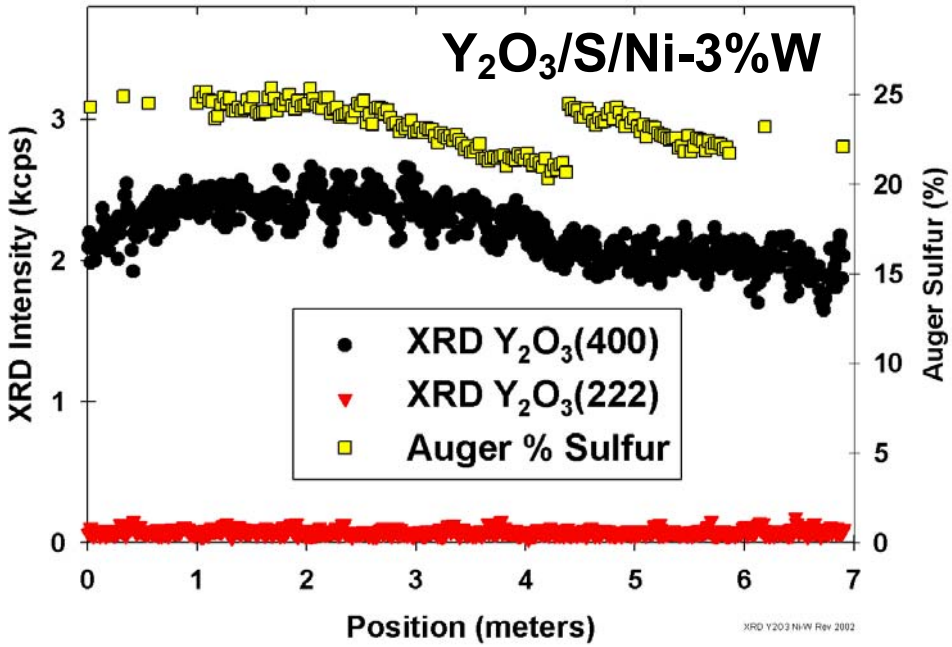
Y_2O_3 seed layer appears to be less susceptible to cracking.

Lee et al., *Jpn. J. Appl. Phys. Lett Part II.* 38 (1999) L178.

Y_2O_3 seed layers with uniform texture have been prepared using sulfur.

"Swiss Army Knife" System





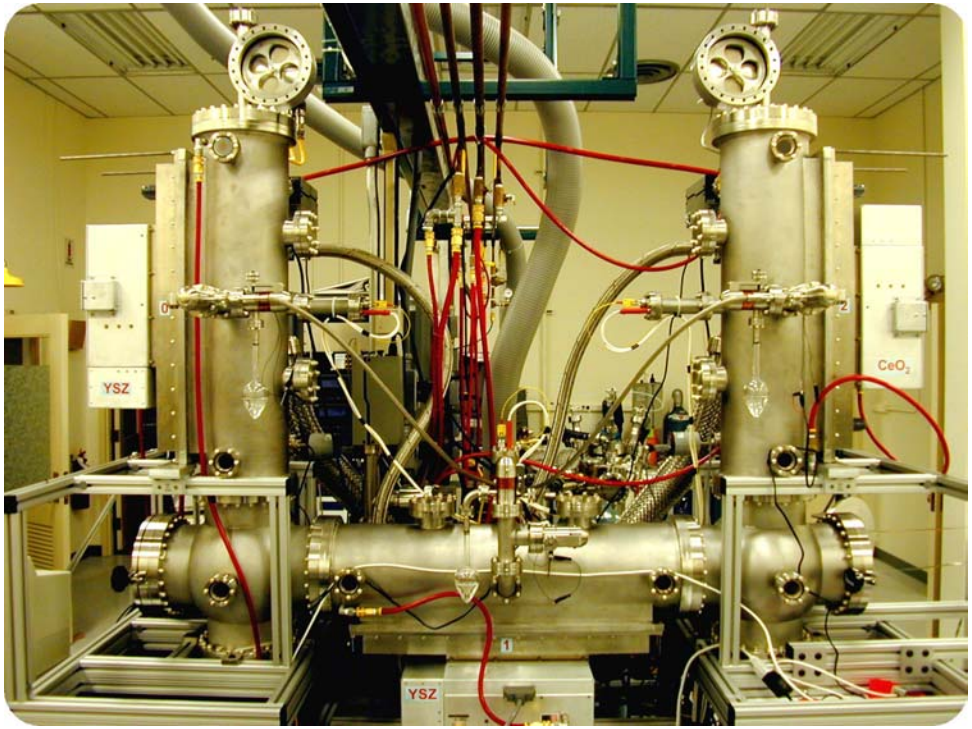
Annealing of substrates (~1300°C typical)

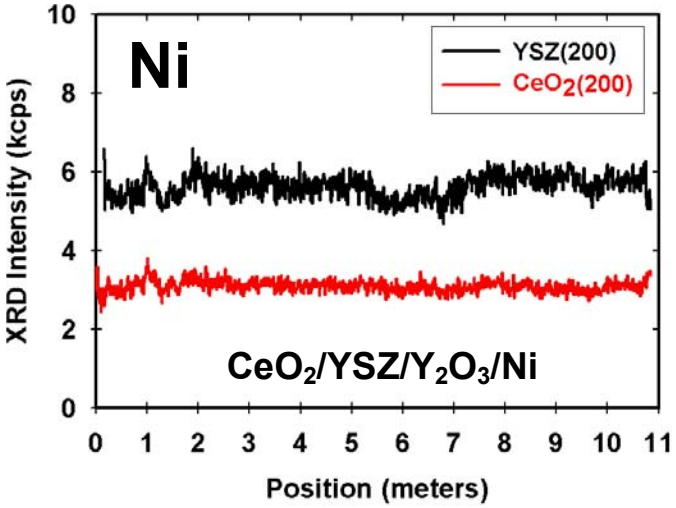
Sulfurizing of substrates (H_2S w/ *in situ* Auger)

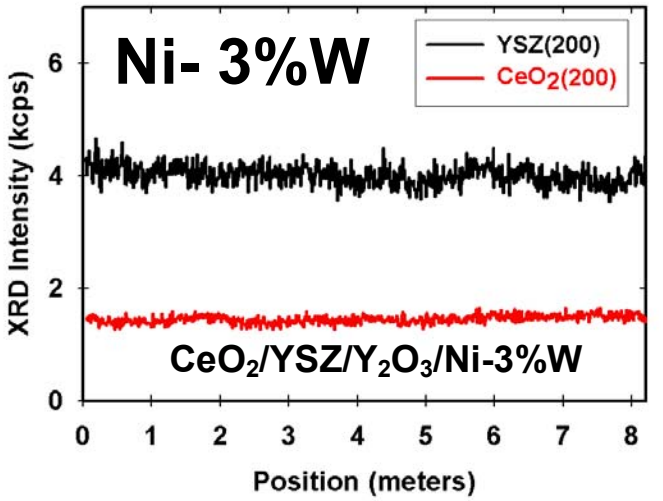
Depositing of seed layer (CeO_2, Y_2O_3)

Annual Yield: >250 meters of seeded tape

RABiTS of uniform texture is routinely produced using a new linear sputtering system.





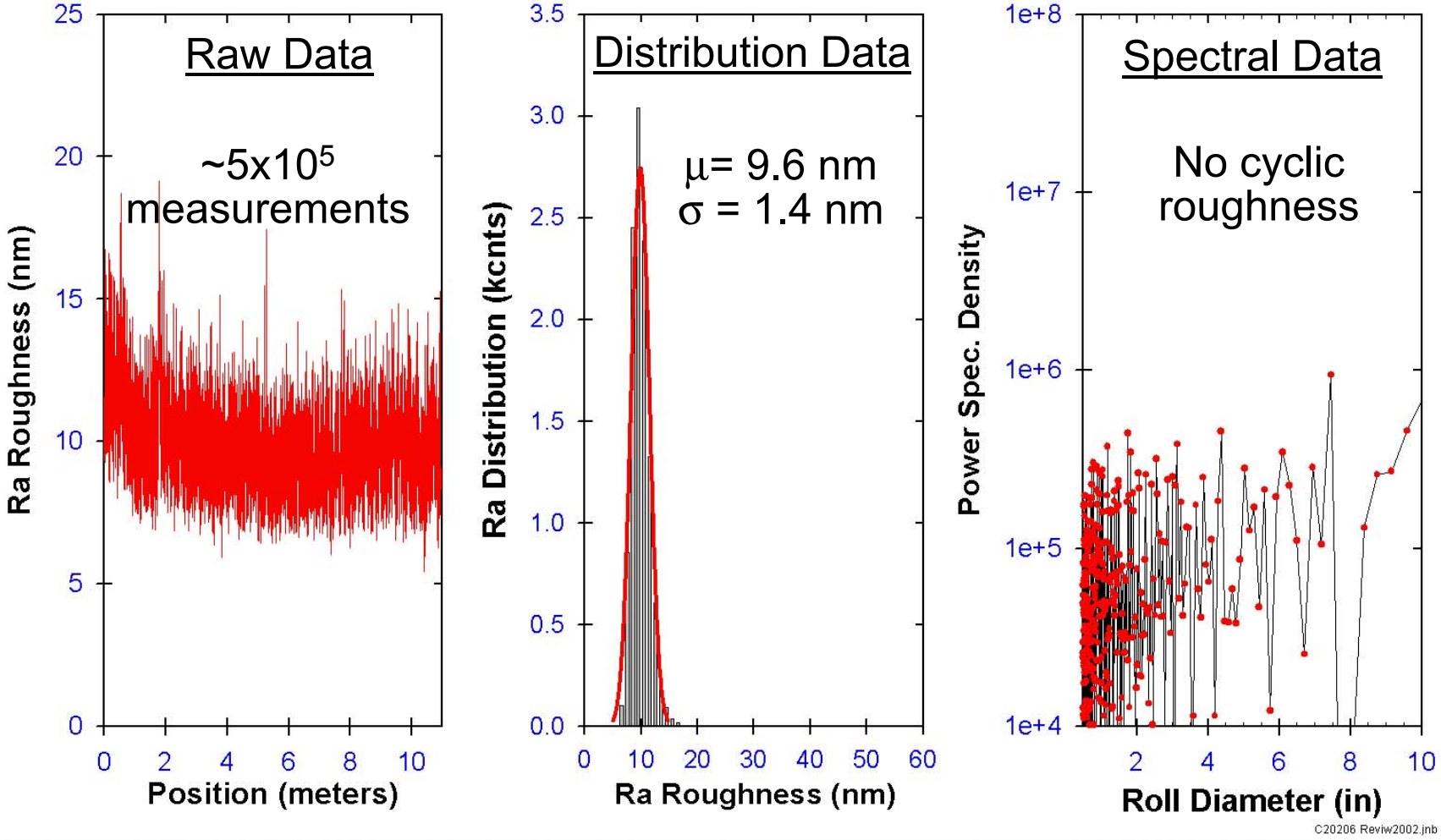


- Three 24” linear sputter sources
- Targets: Two YSZ, One CeO_2
- Throughput: ~1.0 m/hr
- Start of Production: 11/30/01
- Total product: **~170 meters** (7/2/02)

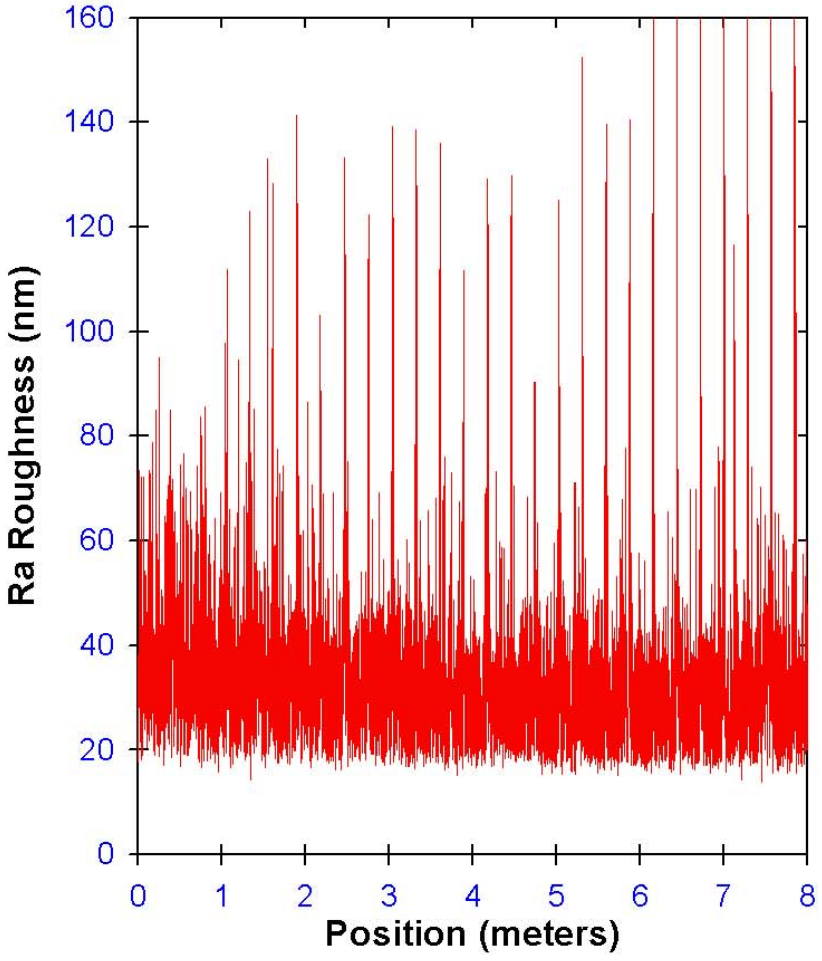
Surface roughness compliments XRD in the development of RABiTS processes.

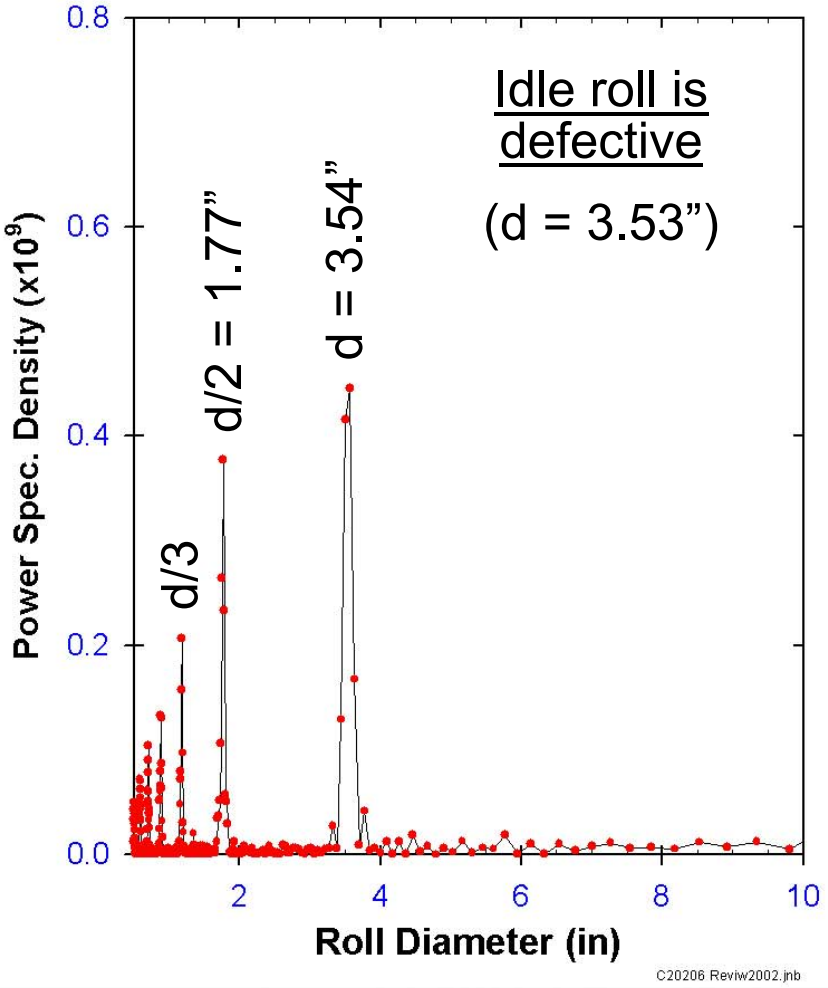
- **Method – Laser reflectance & scattering**
- **Sampling Rate – 10 readings/sec**
- **Sampling size – 1 x 5 mm**
- **Typical tape speed – 1 m/hr**
- **Capacity – 40 m of 0.002” thick tape**
- **Start of Operation – ~12/01/01**
- **Total Tape -- >300 meters**
- **Users: ORNL, 3M, MCT**

Surface roughness data routinely undergo distribution and spectral analyses.

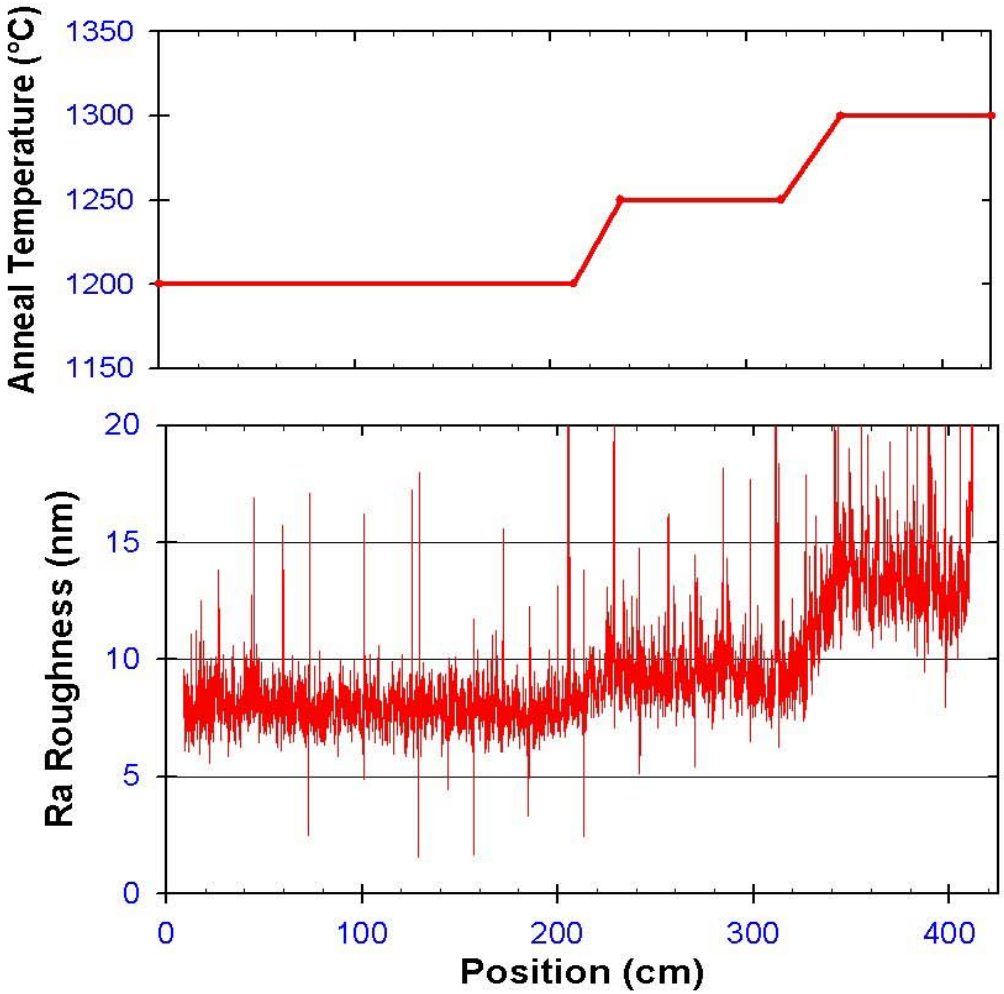


Defective processes can be identified through spectral analysis.

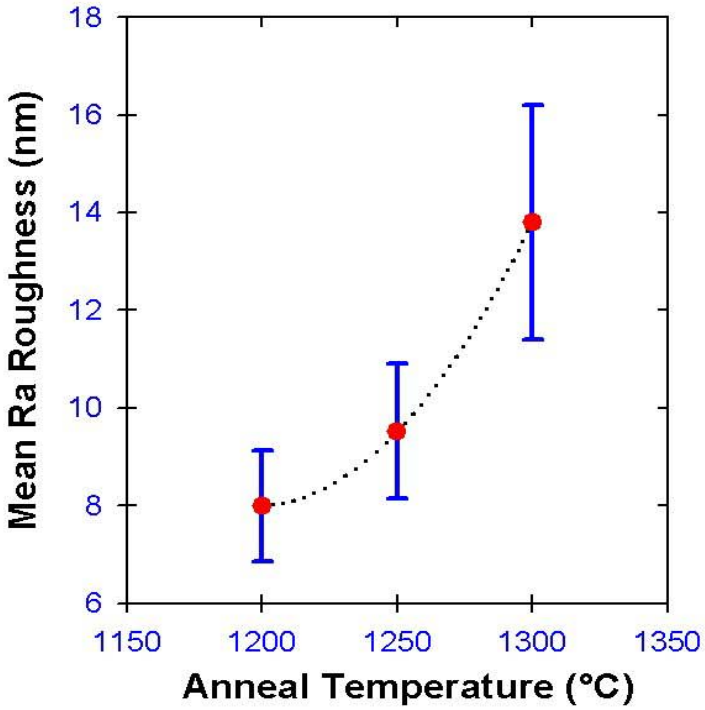




Measurements of surface roughness assist in RABiTS process design.



Tape: Ni-3%W
Pressure:1 atm 4%H_2/Ar
Hot Zone: ~12 in
Tape Speed: 60 cm/hr



R20225A Review2002.jnb

Uniform conversion of larger volumes of YBCO precursor is necessary.

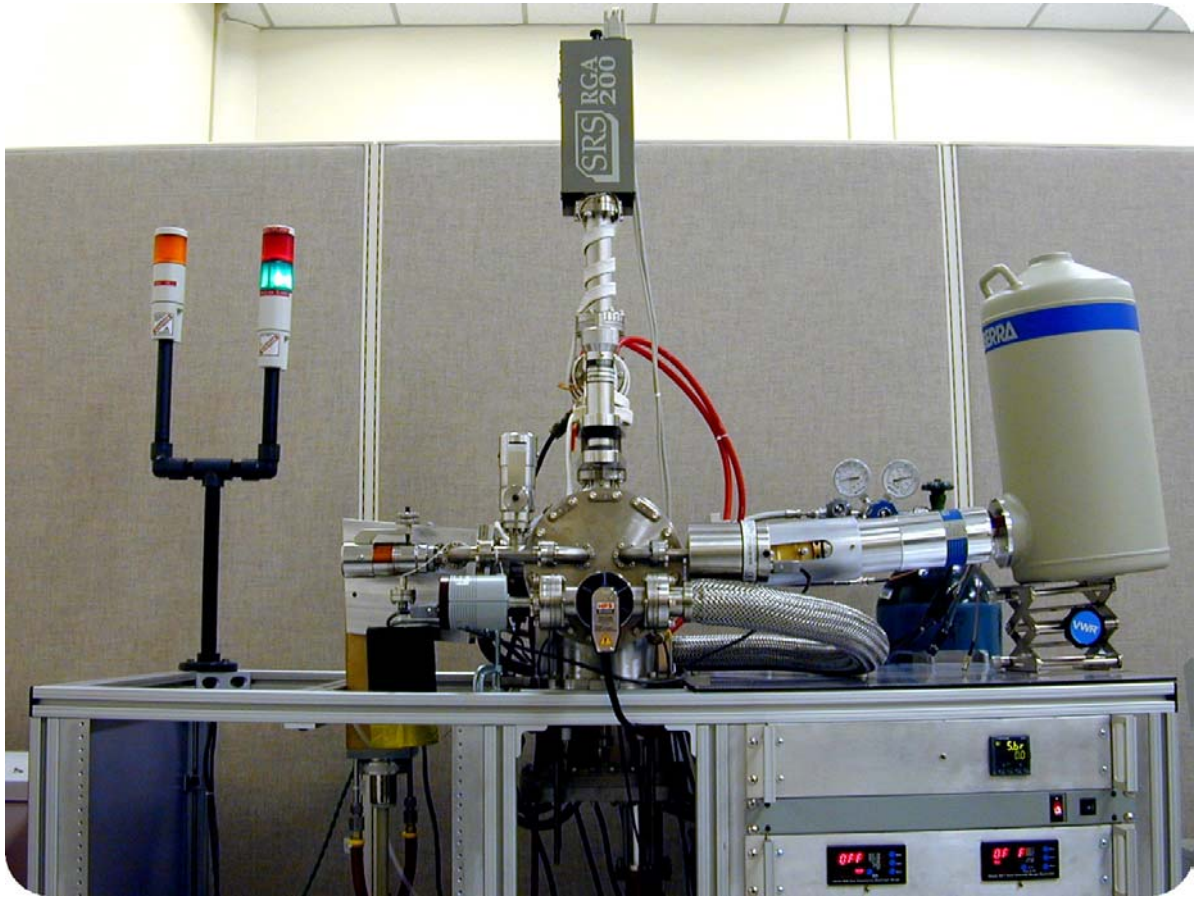
- **Scale-Up Target:**
  - **Production Rate: 100 m/hr (presently ~1 m/hr @ ORNL)**
  - **Product: 1 cm wide x 1um thick YBCO**
  - **YBCO Growth Rate: ~1Å/sec**
- **Possible reel-to-reel system:**
  - **30 cm wide tape and a 10 m long furnace**
- **Possible batch system:**
  - **½ m-diameter x 2 m long mandrel and furnace**
- **Outstanding issues:**
  - **HF removal, gas distribution & diffusion ⇒ Lower Pressure**
  - **Quantity of gas and heat to be handled ⇒ Lower Pressure**
  - **Slitting (reel-to-reel)**

For lower pressures, precursor conversion may be more uniform, rapid, and efficient.

- **Consider simply eliminating the "carrier" gas (*e.g.*, N_2):**
  - **Total pressure is decreased by ~4000x (760 Torr ⇒ 200 mTorr)**
  - **Mean free paths (λ) are increased by ~4000x (~0.4 μm ⇒ ~1.5 mm)**

- **Some direct consequences of lower pressure are:**
  - **Flow is more molecular & nozzle jetting are reduced (more uniform)**
  - **Diffusivities in gas are increased by ~4000x (more rapid)**
  - **Total gas consumption is reduced by ~4000x (more efficient)**
  - **Total heat consumption is reduced by ~4000x (more efficient)**

Low pressure precursor conversion has been studied using a new ACCI XRD system.

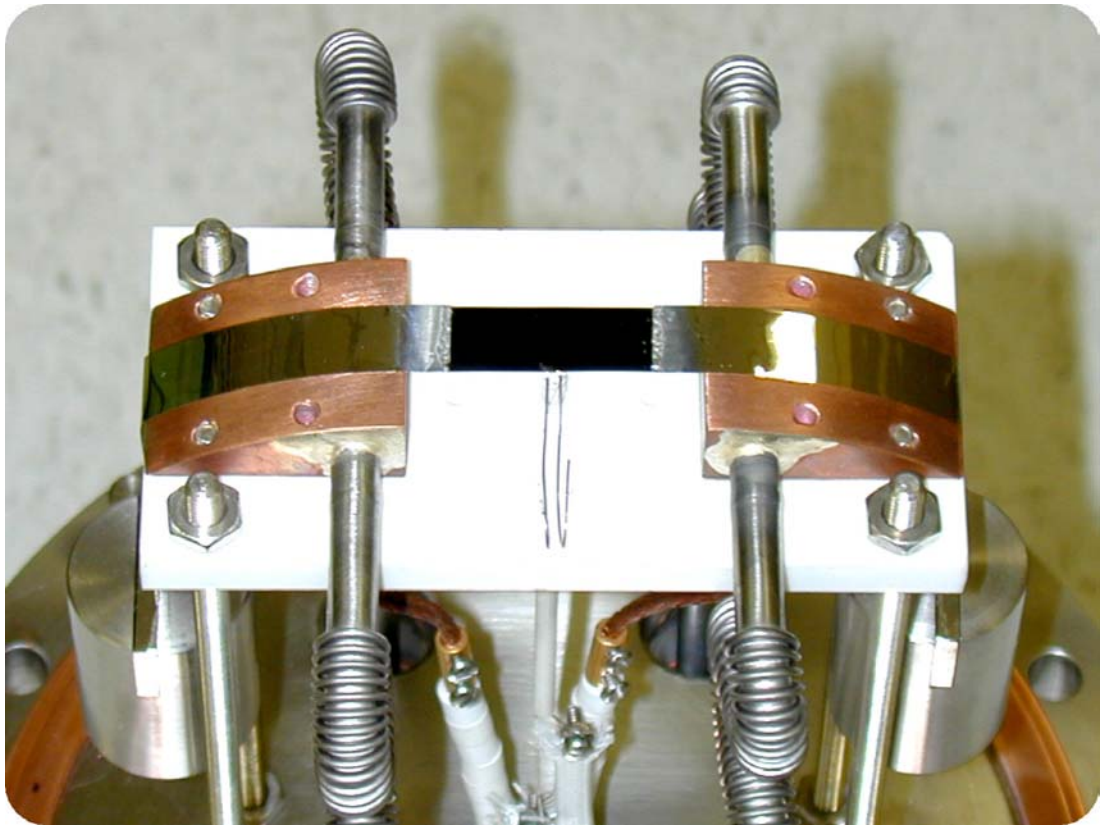


- **Energy Dispersive X-ray Diffraction (EDX)**
  - "white" W source
  - EDX detector
- **Metal-sealed vacuum chamber**
- **Resistive heating**

(<~1250°C in vacuum or reducing atm)

- Start of operation 3/1/02

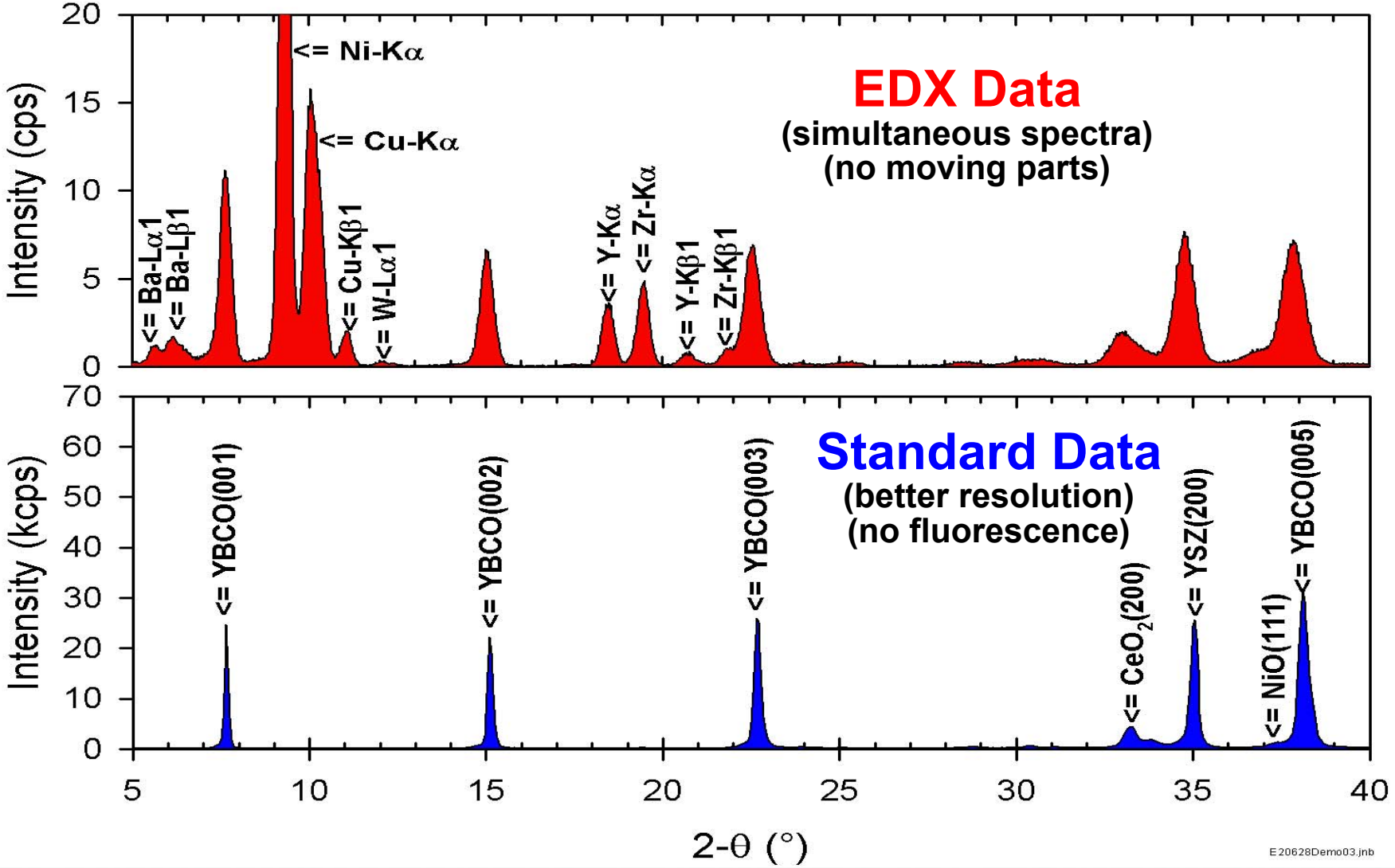
Crystalline phase development and gas phase desorption can be monitored during conversion.



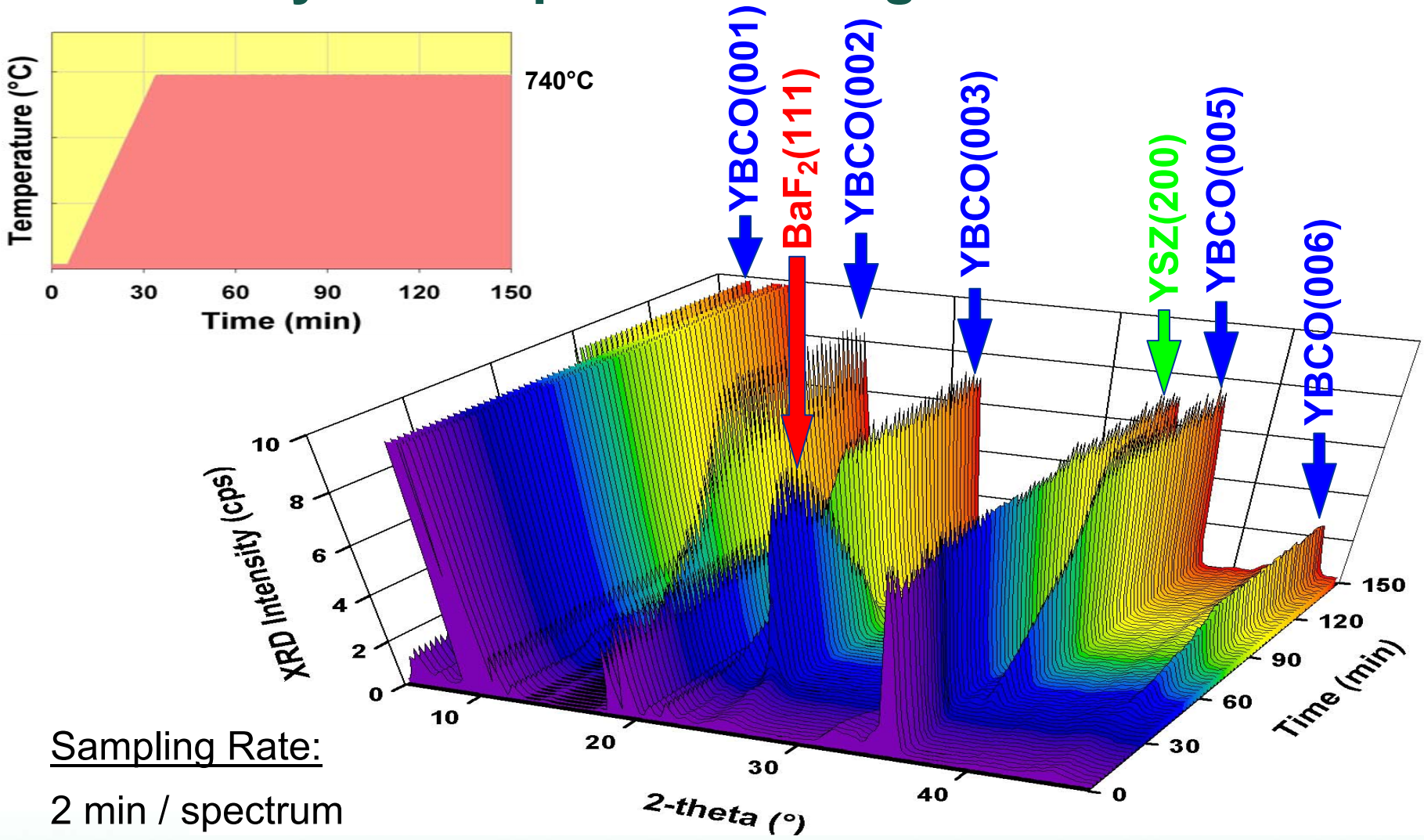
Details:

- Temperature is monitored & controlled with 3-mil K-type TC.
- An electrically conductive sample (>~1cm long) is welded to leaders and suspended between water-cooled electrodes.
- During a programmed temperature profile, EDX data is recorded every two minutes.

EDX data are similar to standard θ-2θ data.



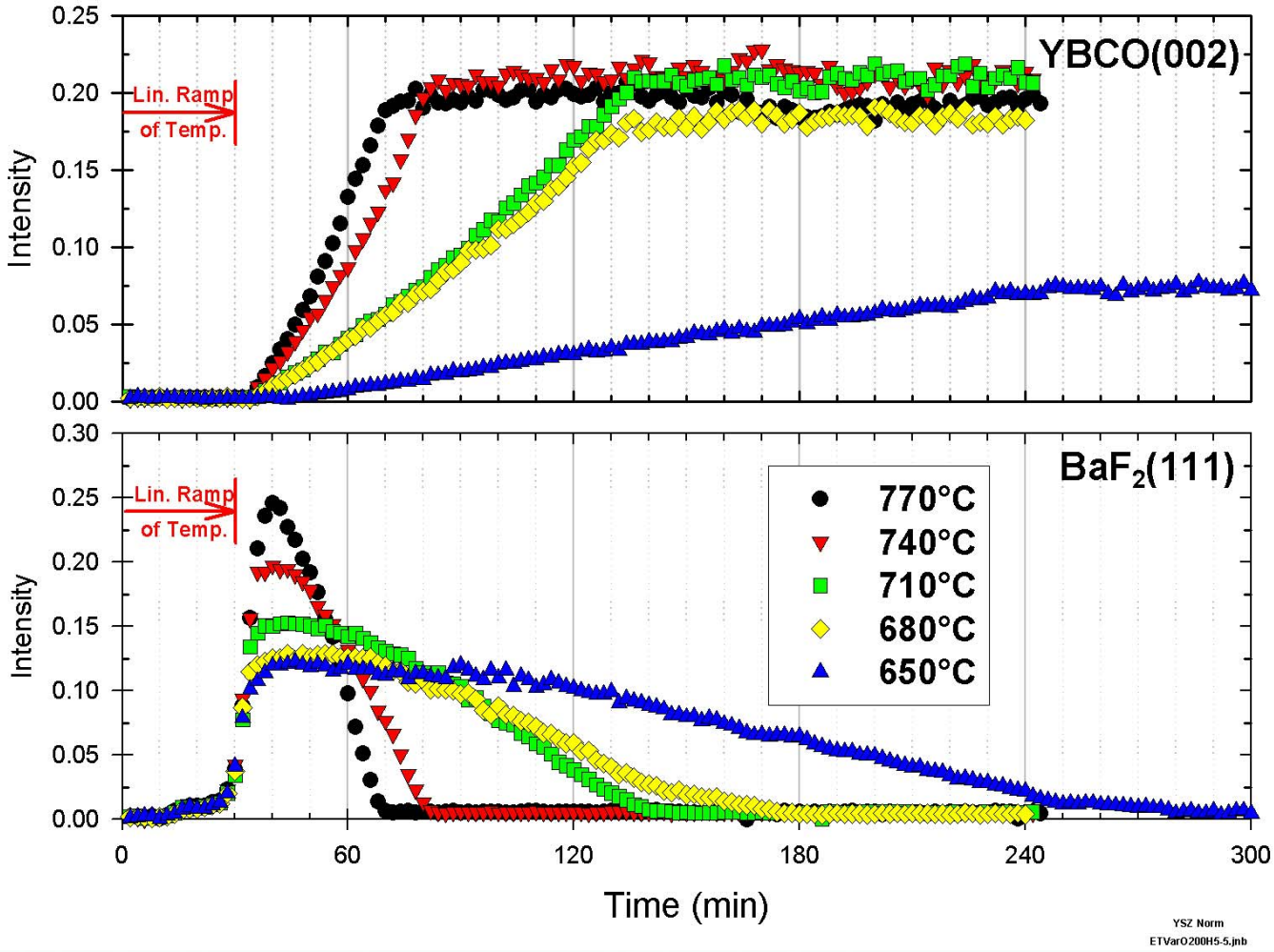
A family of x-ray spectra clearly shows evolution of crystalline phases during conversion.



Sampling Rate:

2 min / spectrum

Rate of precursor conversion increases with conversion temperature.



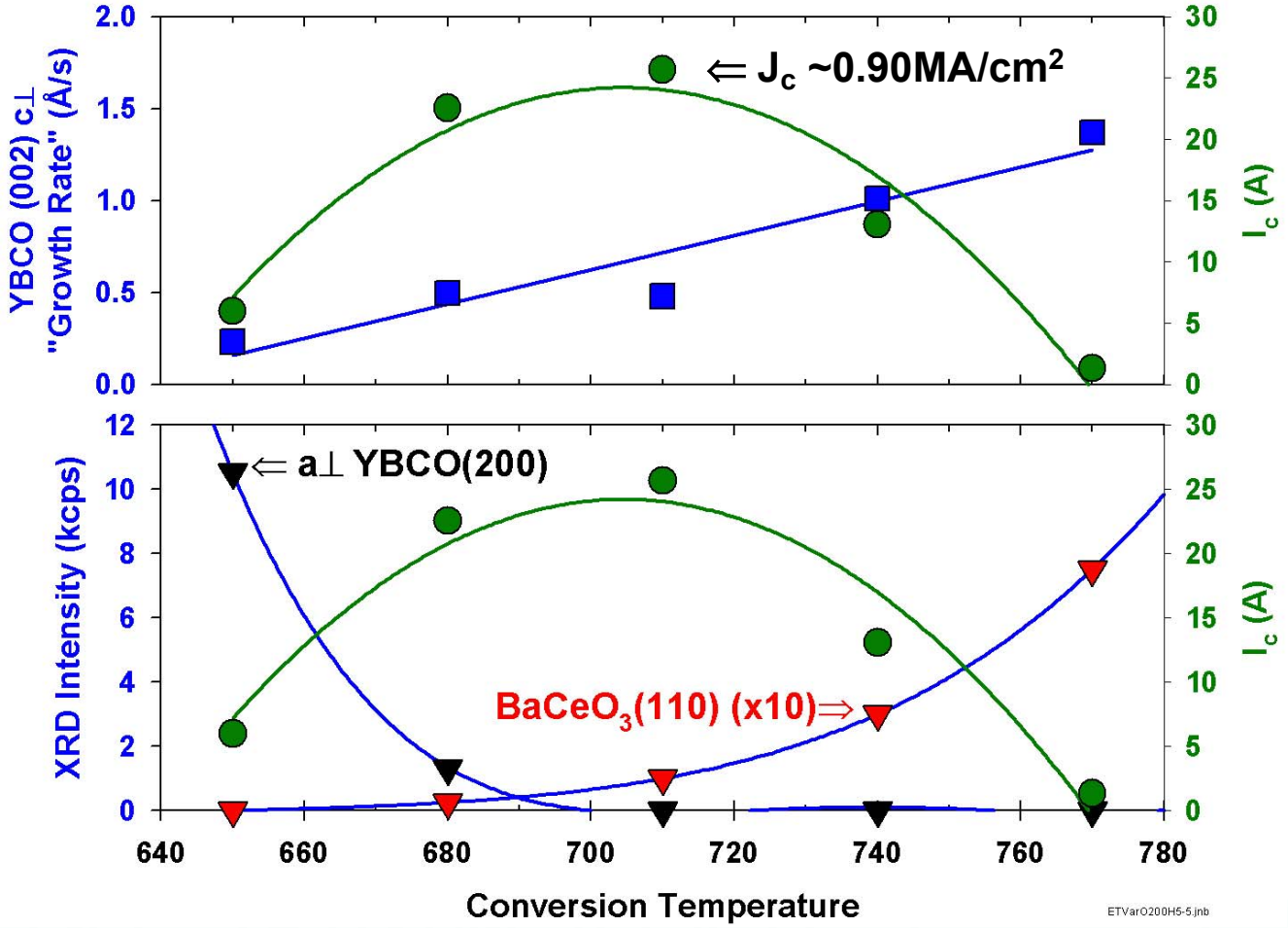
Ramp Rate = 25°C/min

P_{total} = 200 mTorr

P_{O2} = 200 mTorr

P_{H2O} = 0.05 mTorr

I_c passes through a maximum as the conversion temperature increases.



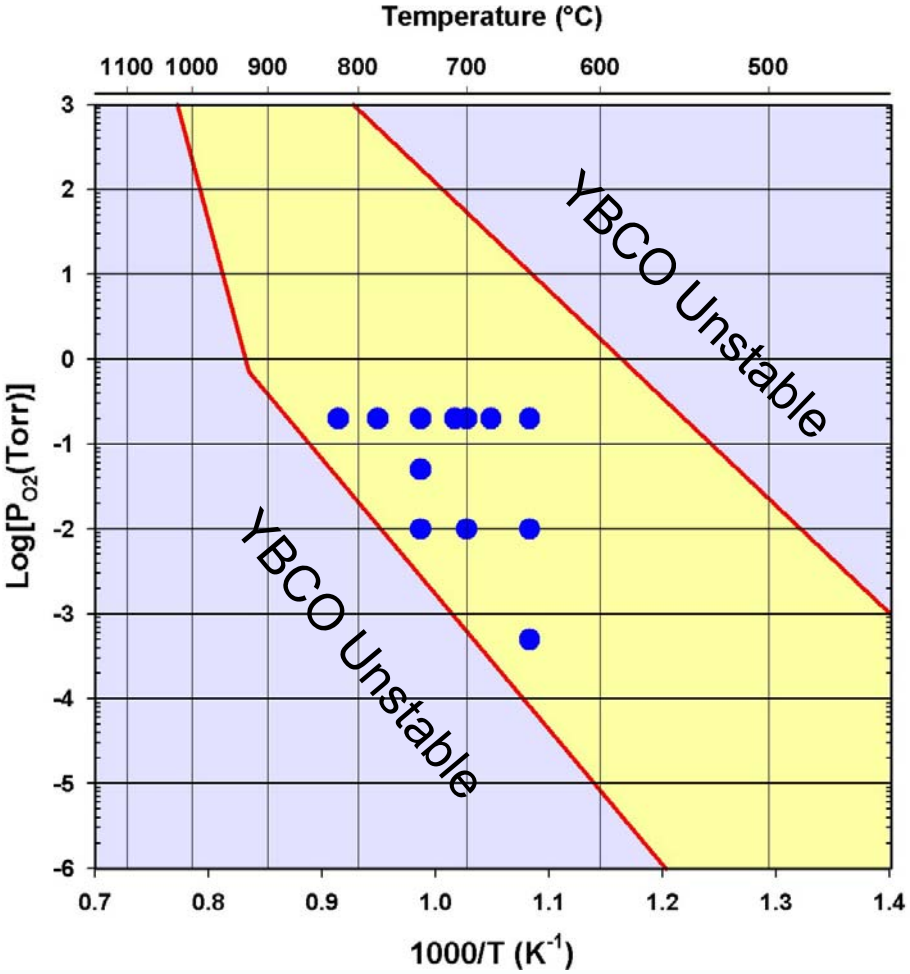
As conv. temp. ⇑

c⊥ "growth rate" ⇑

For <u>low</u> conv. temp., <u>high</u> a⊥ YBCO.

For <u>high</u> conv. temp., <u>high</u> $BaCeO_3$.

Low pressure studies are contributing to understanding of the conversion process.



Conversion Variables

- Oxygen Pressure
- Water Pressure
- Ramp Rate
- Conversion Temperature
- Conversion Time
- Precursor Thickness

1st CRADA Involvement

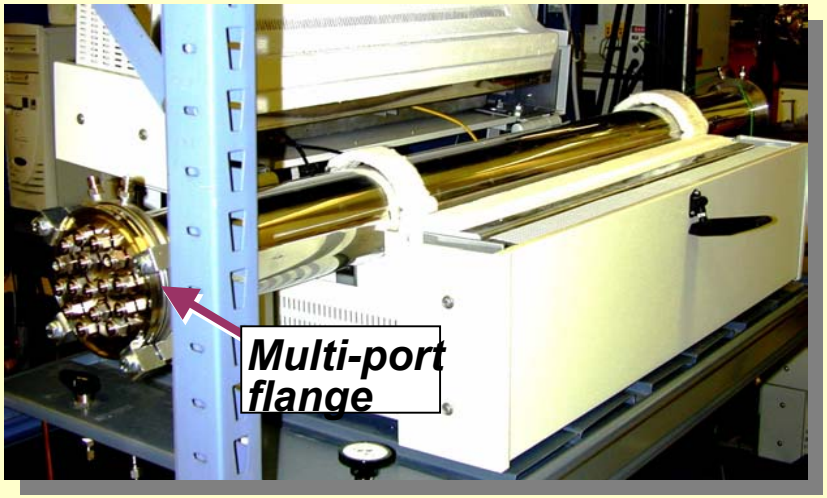
- AMSC July 29, 2002

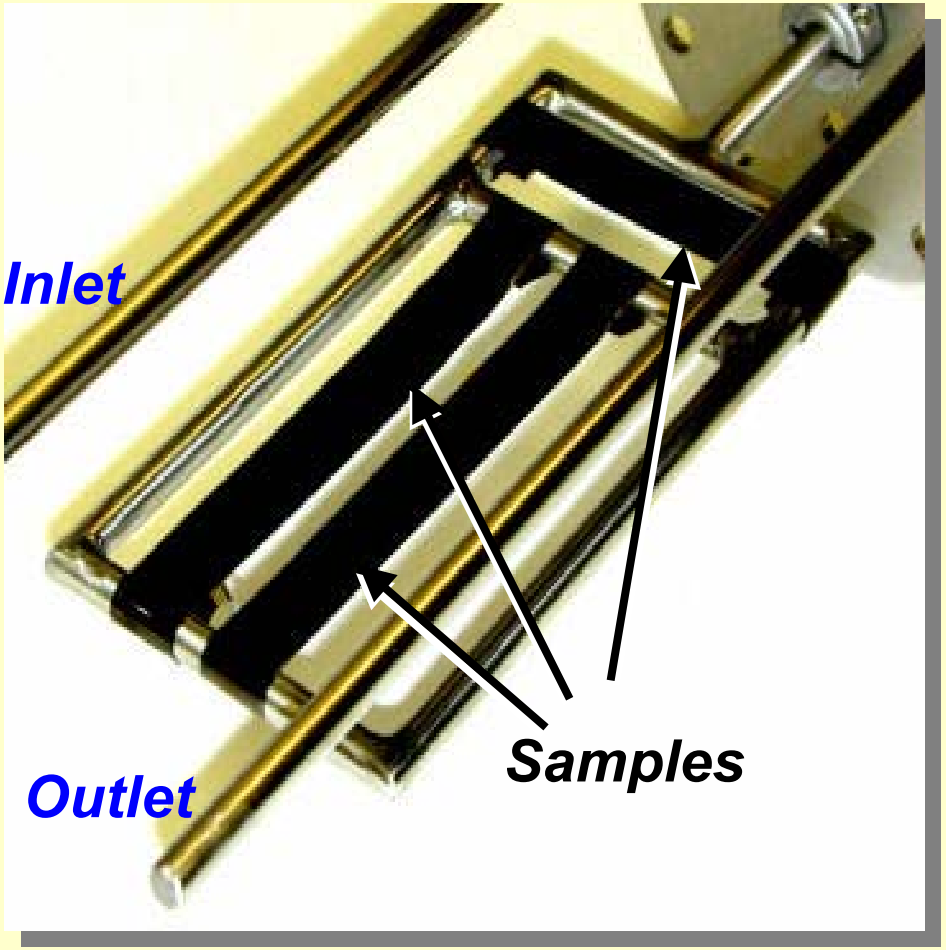
OUTLINE

- **FY 2002 Results**
  - Status of ORNL ACCI Laboratory
  - Long-length RABiTS developments — Fred List
  - Low pressure (~200 mTorr) precursor conversion
  - Reduced pressure (~0.3 atm) precursor conversion — Dominic Lee
  - Reel-to-reel *ex-situ* YBCO conversion
- **Summary of FY 2002 Results / FY 2003 Plans**
- **Research Integration**

In addition to the low-pressure system, we also utilize a furnace with reduced chamber pressure & other capabilities

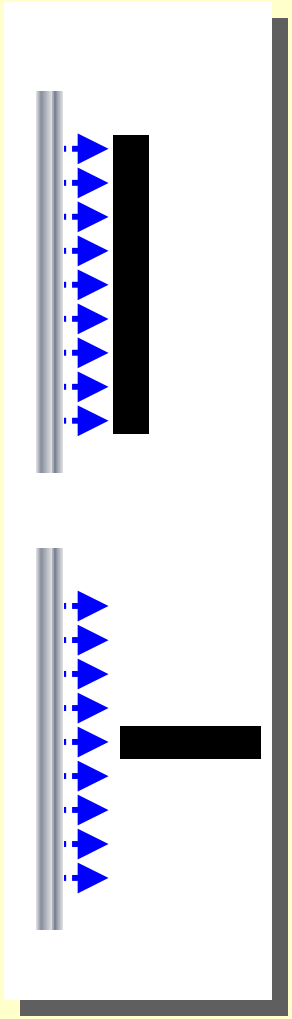
- Reduced chamber pressure by pumping on exhaust →
  ~0.3 atm at 1 l/min flow.
- Sample orientation →
  Transverse or longitudinal gas flow.

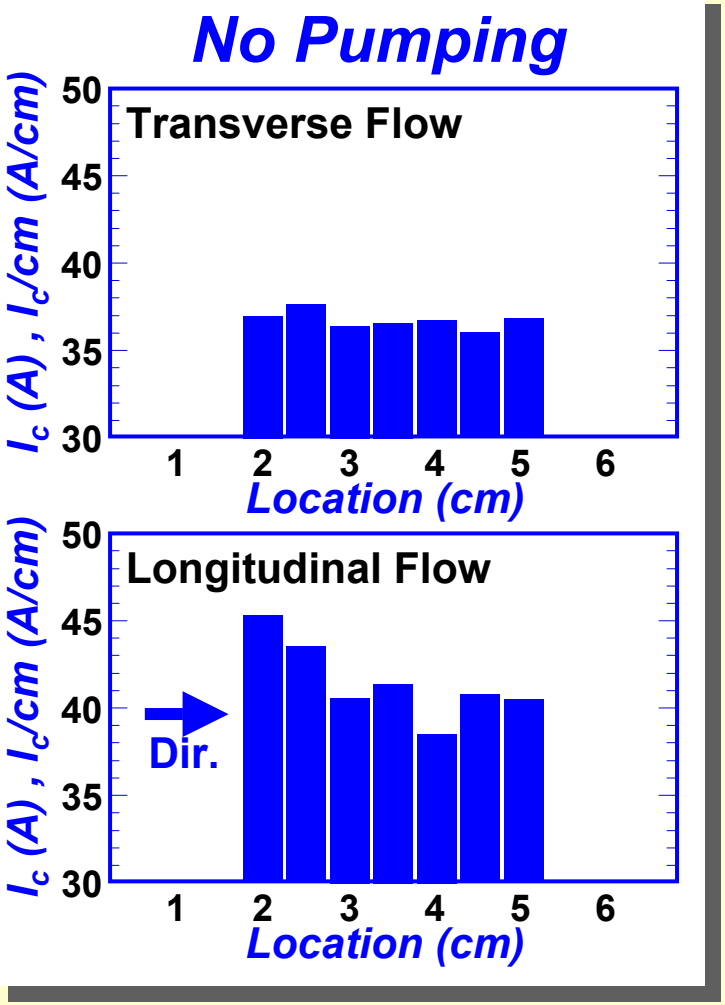


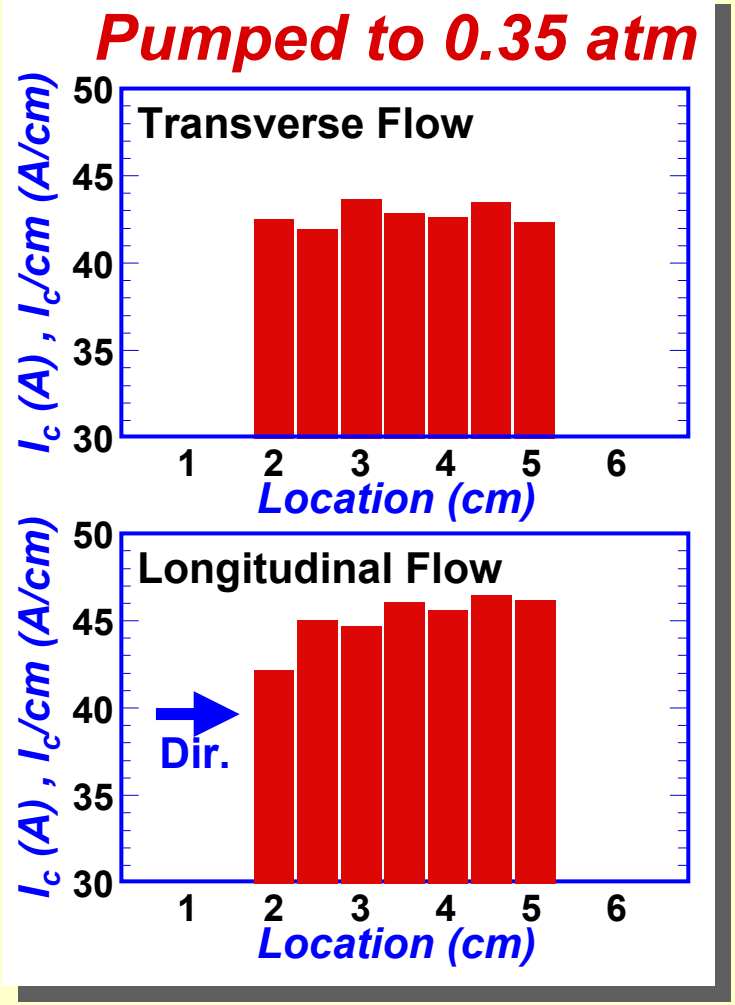


Preliminary results suggest that HTS quality and homogeneity may be improved by reducing the chamber pressure through pumping

- 3000Å precursor on Ni-W RABiTS: 740°C, 100min, 30° gas injection.







UT-BATTELLE

Reel-to-Reel Ex-Situ Conversion of "BaF_2" Precursor

This year's themes:

Better Substrate	Higher J_c
Lower P_{H_2O}	Wider Processing Window

Special acknowledgement: L. Heatherly Jr., K.J. Leonard, J. Yoo.

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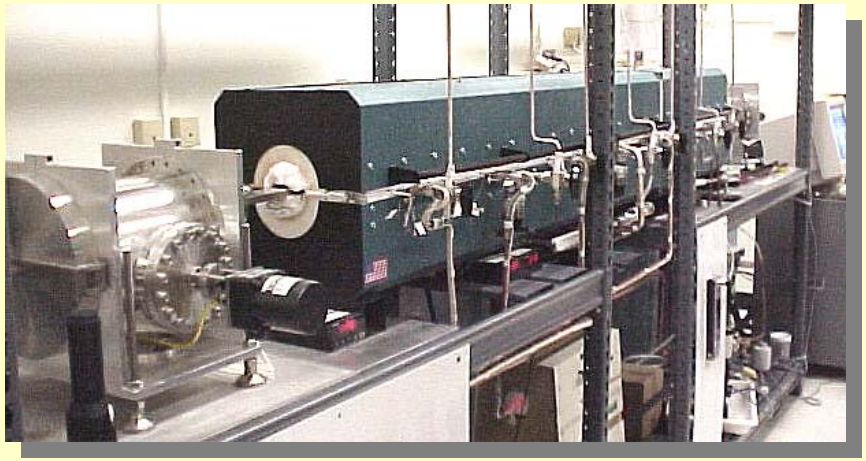
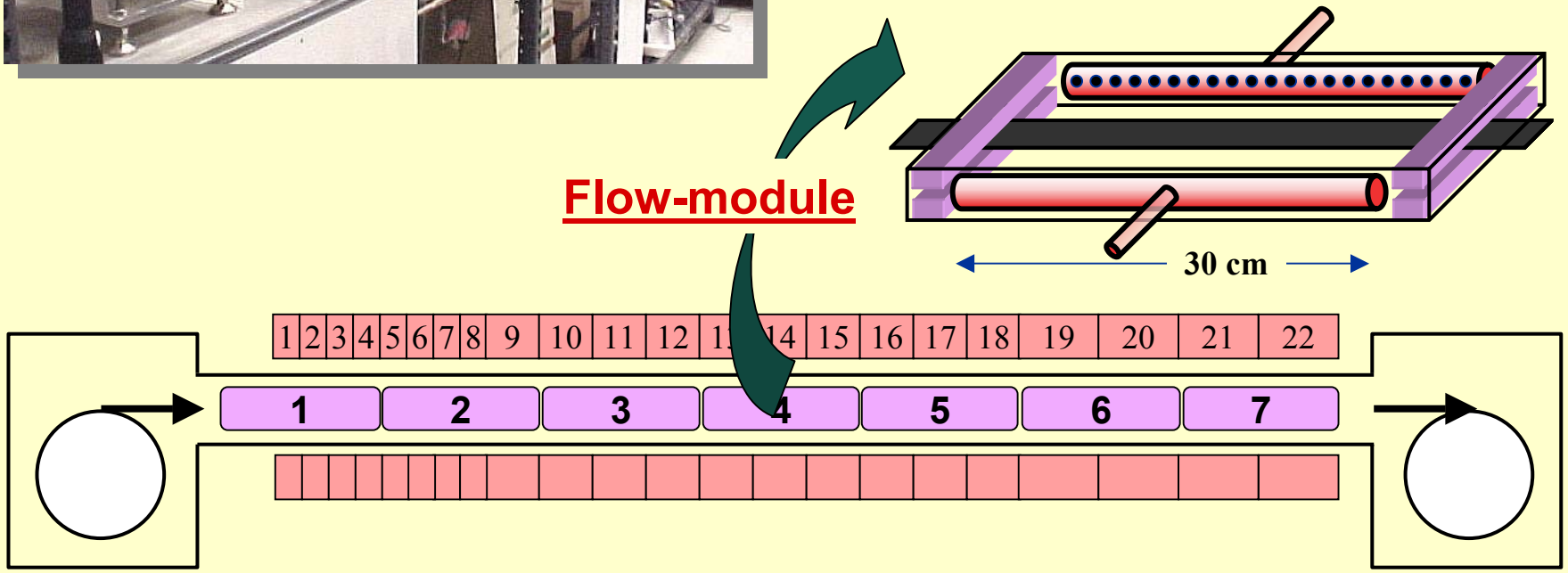
U. S. DEPARTMENT OF ENERGY

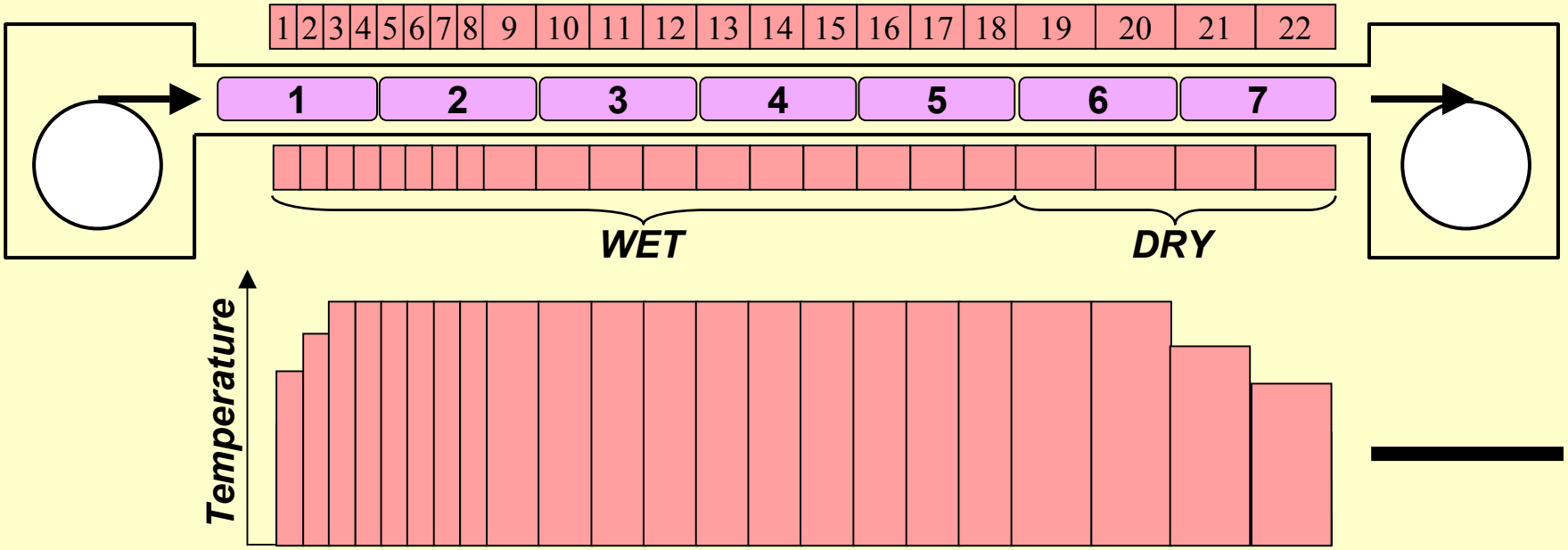
A metallic reel-to-reel chamber was constructed to study continuous conversion of ex-situ YBCO on textured substrates

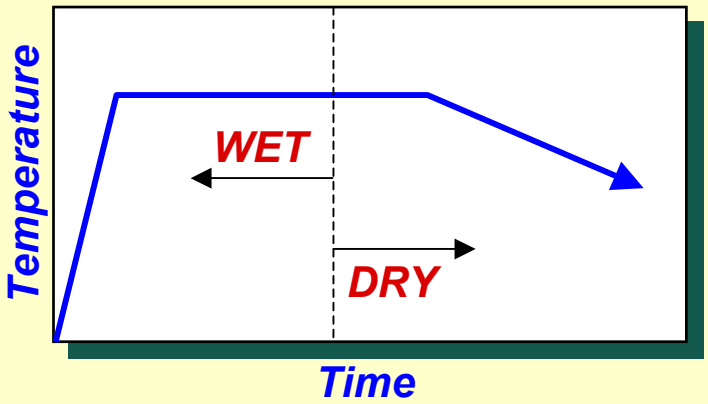


- 22 zone furnace, seven flow modules with independent rate & $P(H_2O)$ [five] controls.
- Narrow slots between modules to minimize cross-flow.



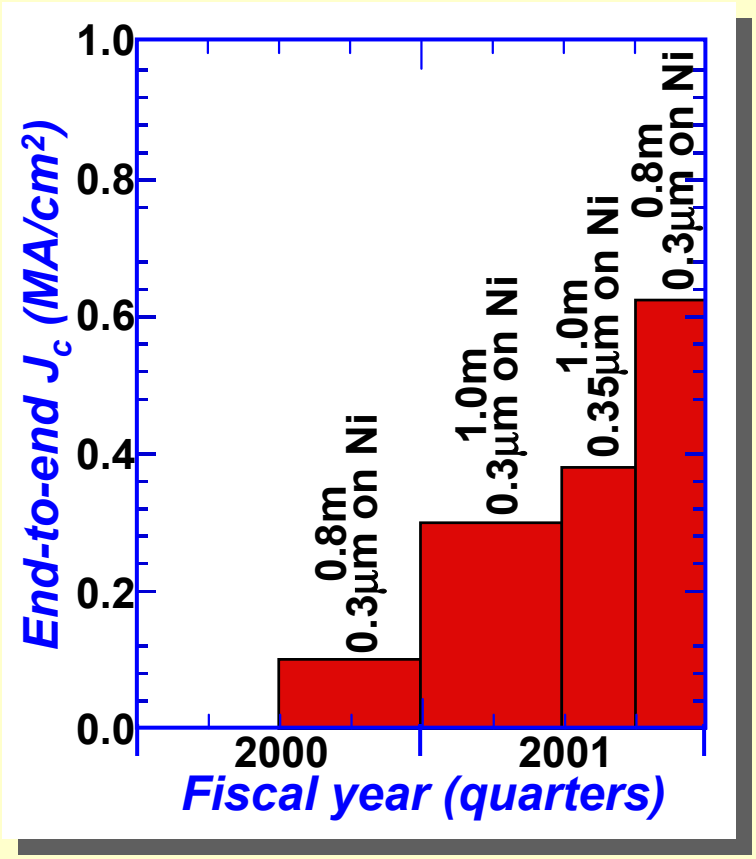
Continuous conversion is typically performed by a "Pull-Through" approach

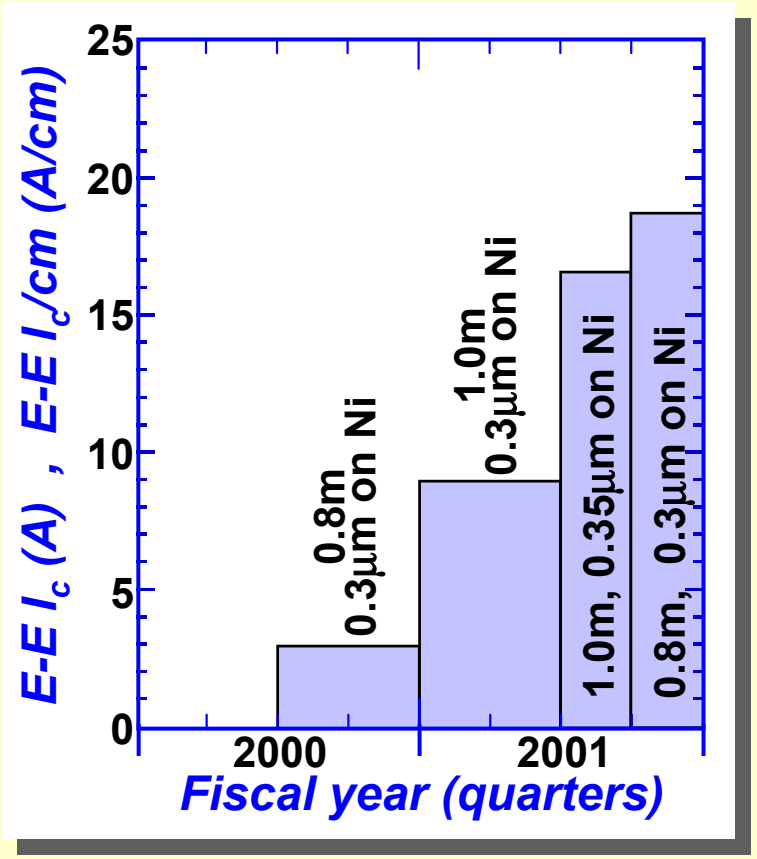




- Constant tape speed & temperature profile mimics "stationary" conversion condition.
- Enables study of variation in properties along tape length → *performance uniformity.*

Until FY2002, meter-length conversions were performed on 3000Å-thick precursors on Nickel RABiTS



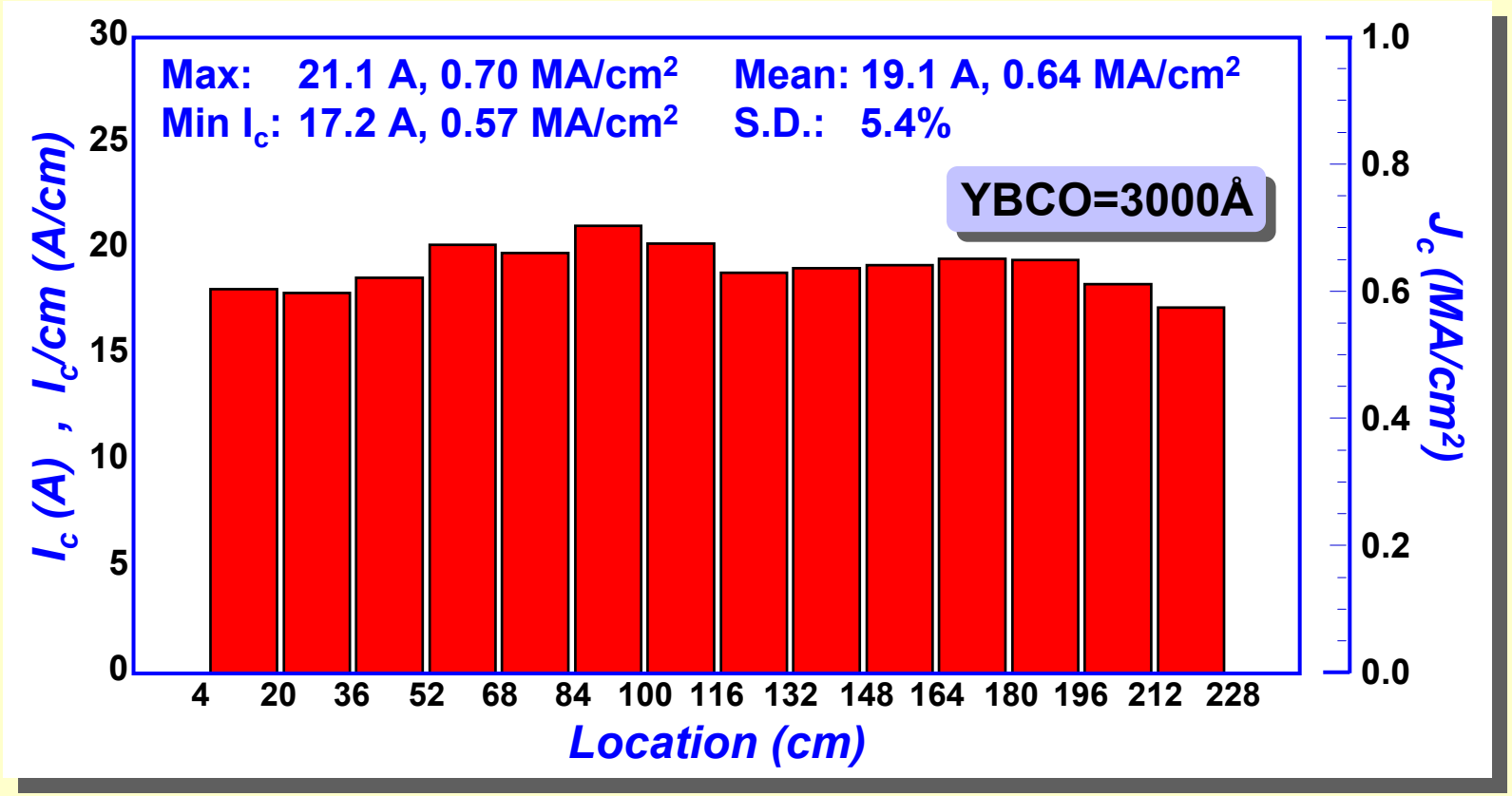


FY2002 Results:

3000Å-thick "BaF_2" Precursors

2.3 meters of 3000Å-thick precursor on Nickel RABiTS exhibits uniform properties

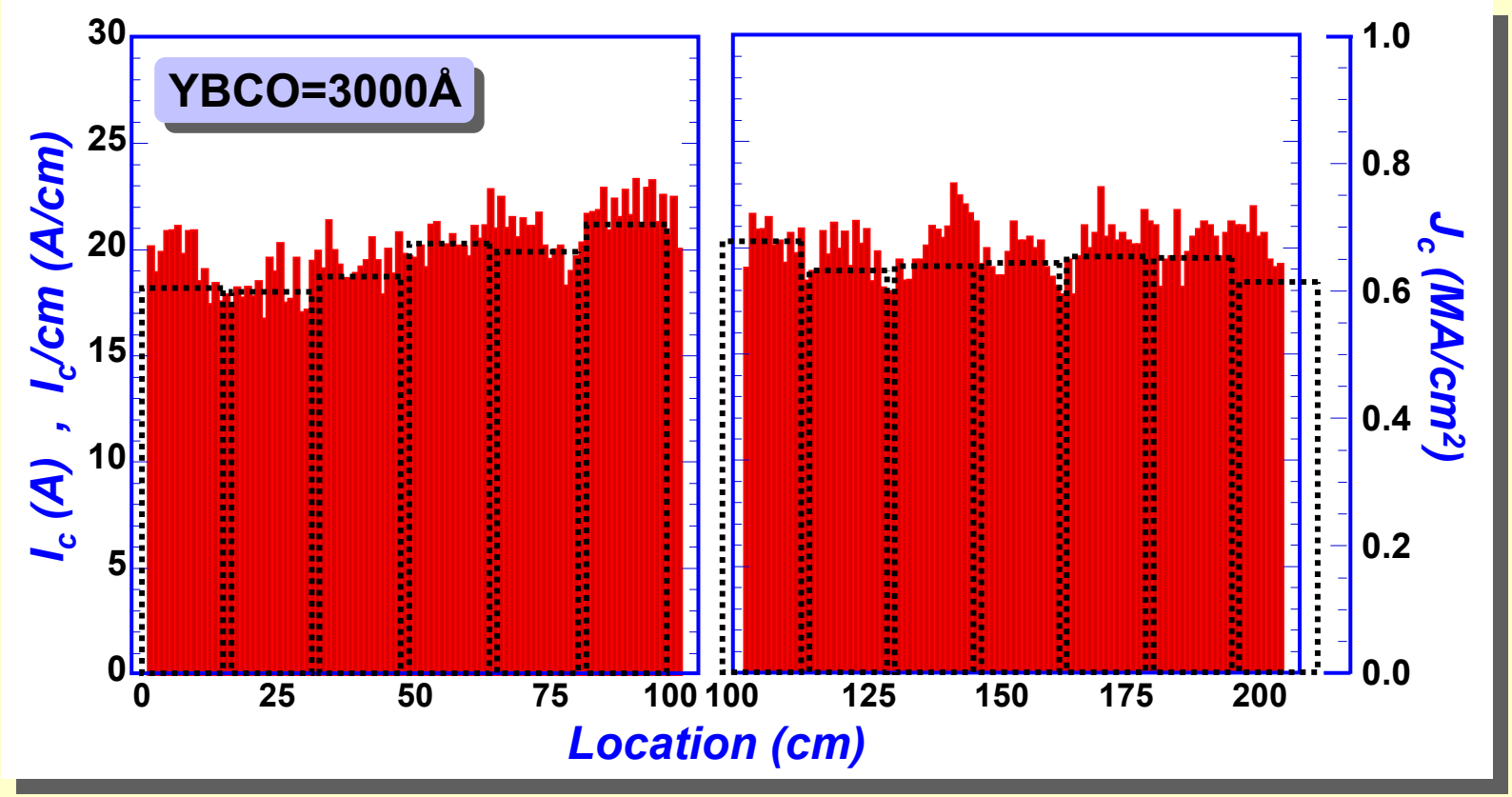
- 1cm X 232cm YBCO/CeO_2/YSZ/Y_2O_3/Ni.
- Substrate texture: $\Delta\omega$ FWHM ~ 8° $\Delta\phi$ FWHM ~ 10°.
- Converted under "high" P(H_2O) used previously.
- End-to-end I_c = 18.9 A, J_c = 0.63 MA/cm^2.



1cm sectional measurements agree well with those of every 16cm, and show that the long coated-conductor has uniform properties

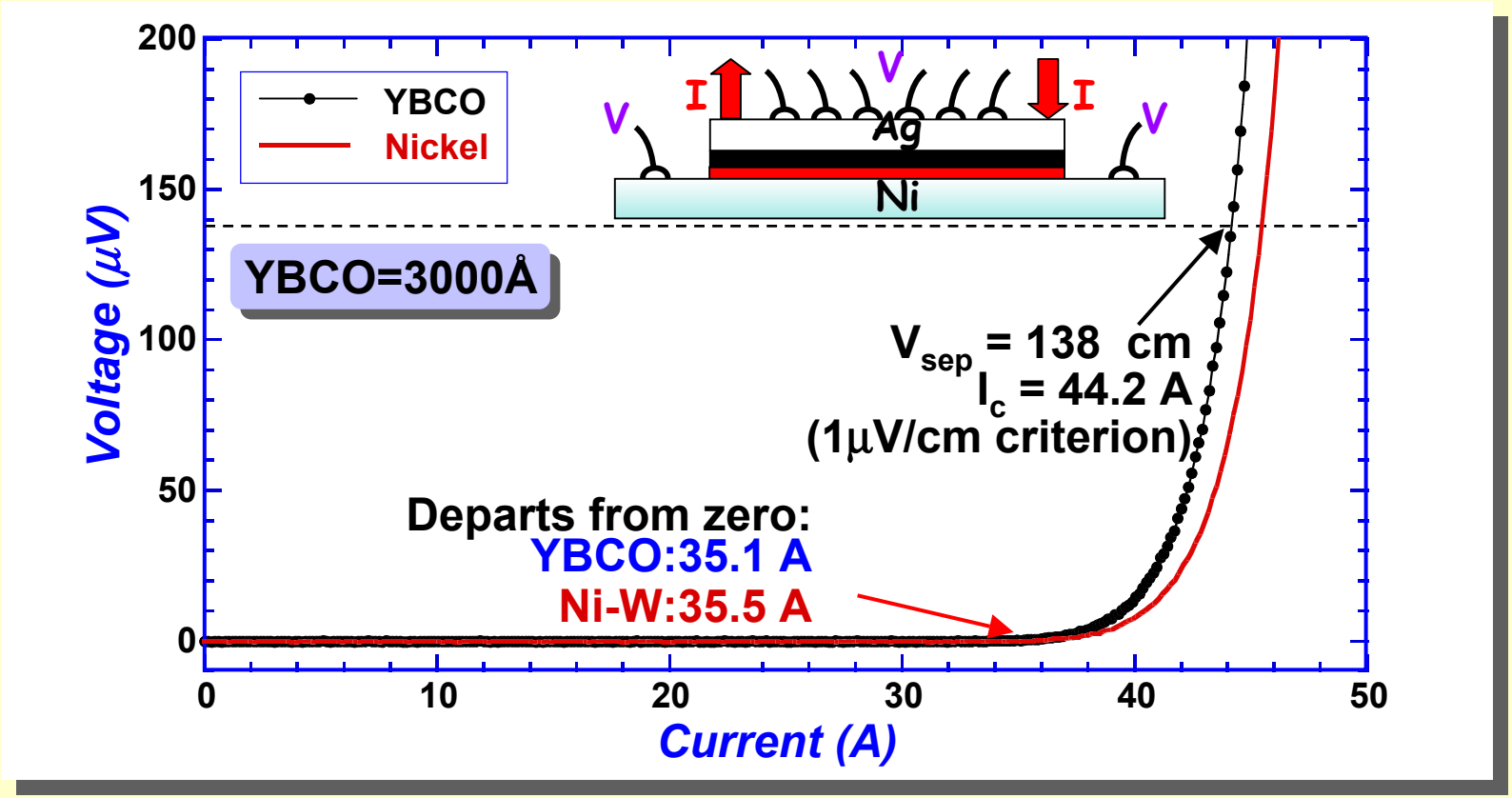
Max I_c: 23.3A
Min I_c: 16.8A
Mean I_c: 20.1A S.D.=7.7%

Max I_c: 23.0A
Min I_c: 17.8A
Mean I_c: 20.1A S.D.=5.7%



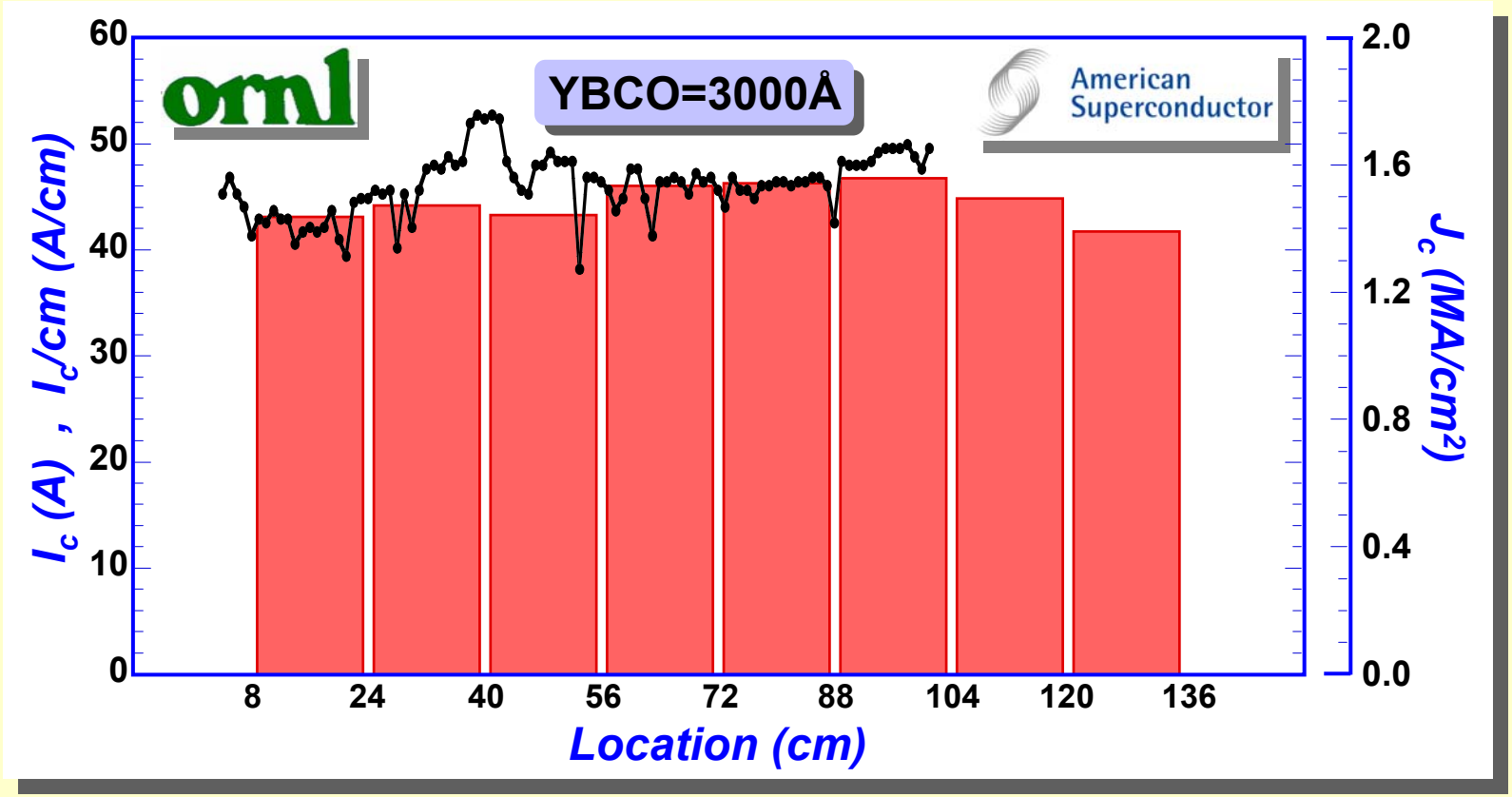
We have also converted 1.5 meter of 3000Å-thick precursor on Ni-3%W

- 1cm X 150cm ORNL YBCO/AMSC buffers/ORNL Ni-W.
- Substrate texture: $\Delta\omega$ FWHM ~ 5° $\Delta\phi$ FWHM ~ 7°.
- Also converted under "high" P(H_2O) used previously.
- End-to-end I_c = 44.2 A, J_c = 1.5 MA/cm^2.



Sectional measurements of the YBCO on NI-W RABiTS revealed that the J_c's are uniform

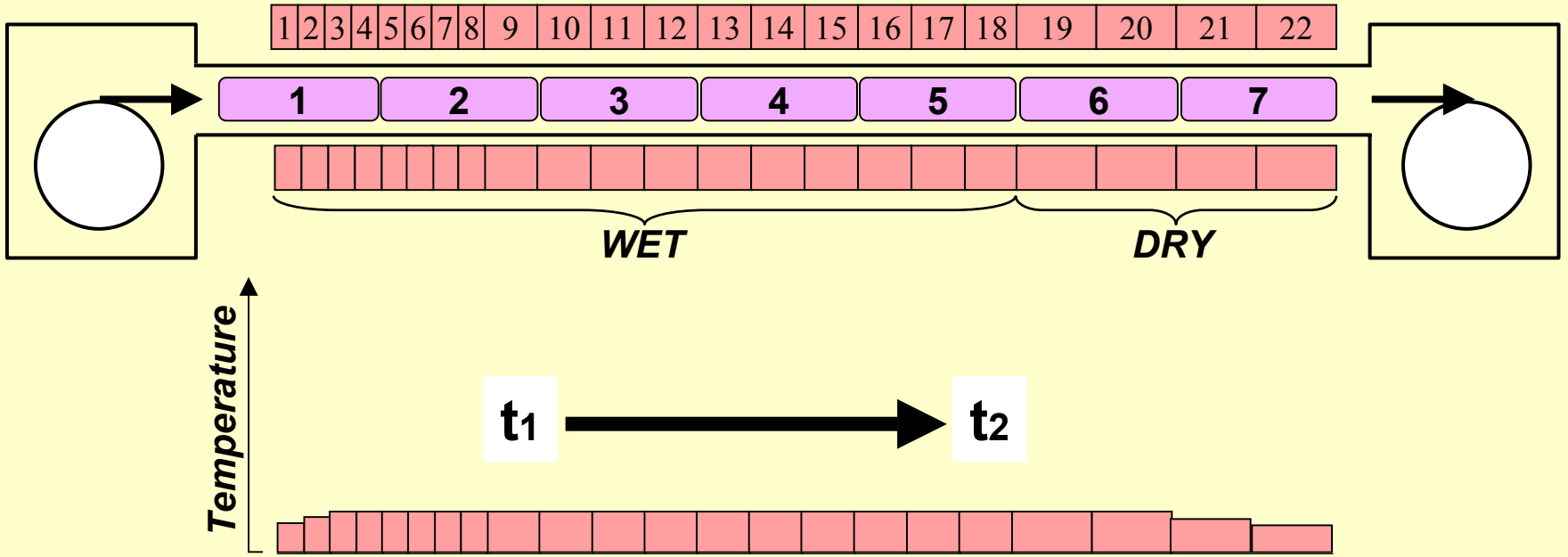
- 16cm sections: Max: 46.7A, 1.6 MA/cm^2 Min: 41.7A, 1.4 MA/cm^2
  Mean: 44.5A, 1.5 MA/cm^2 S.D.: 4.0%
- 1 cm sections: Max: 52.7A, 1.8 MA/cm^2 Min: 38.2A, 1.3 MA/cm^2
  Mean: 46.1A, 1.5 MA/cm^2 S.D.: 6.2%



FY2002 Results:

~6000Å-thick "BaF_2" Precursors

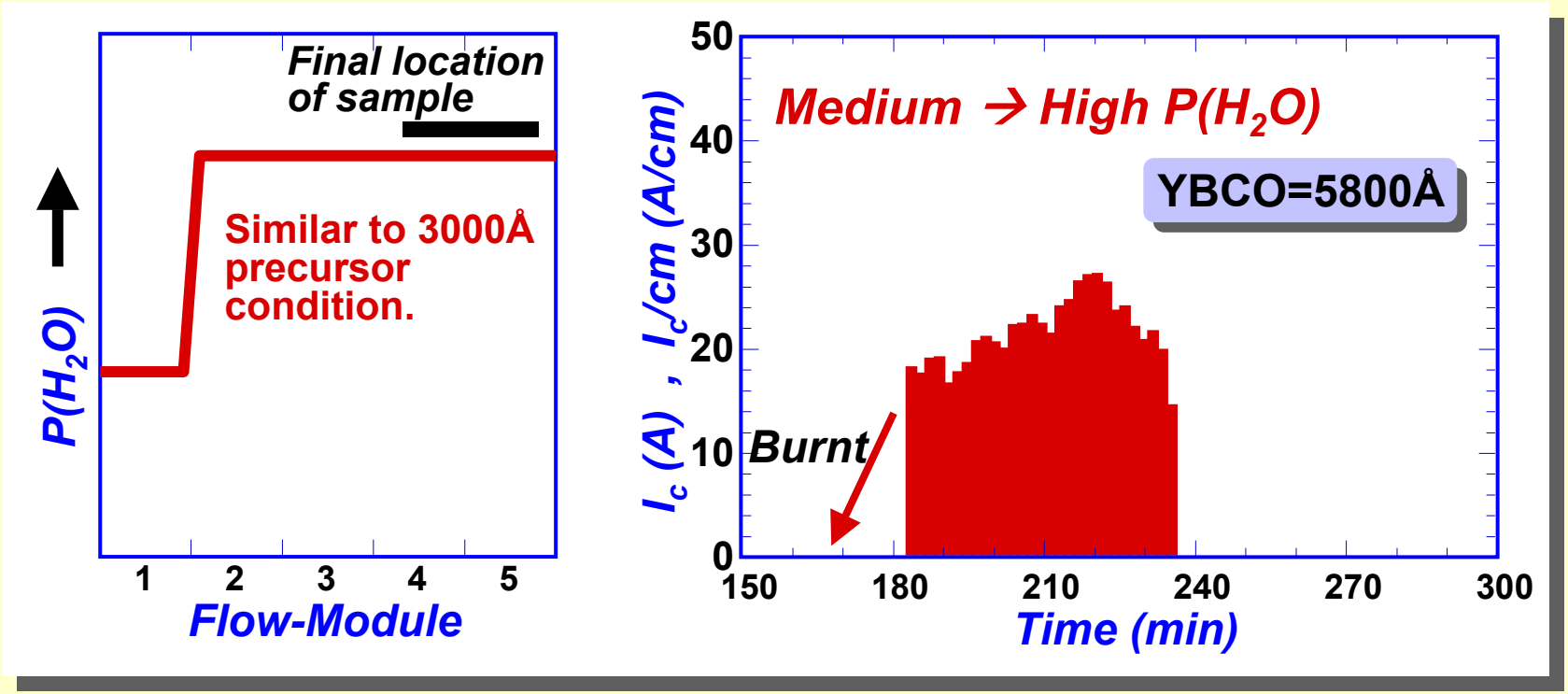
A "partial conversion" approach is more efficient in studying conversion time of coated-conductors



- **Single precursor tape** converted for various amount of time under fixed processing condition AND history.
- Enables study of variation in conversion time with phase content and properties.

"High" $P(H_2O)$ leads to a narrow processing window in thicker precursors

- ~150 cm of 5800Å precursor was deposited onto a long-length Nickel RABiTS.
- 50cm pieces were used in "partial conversion" to study processing parameters.

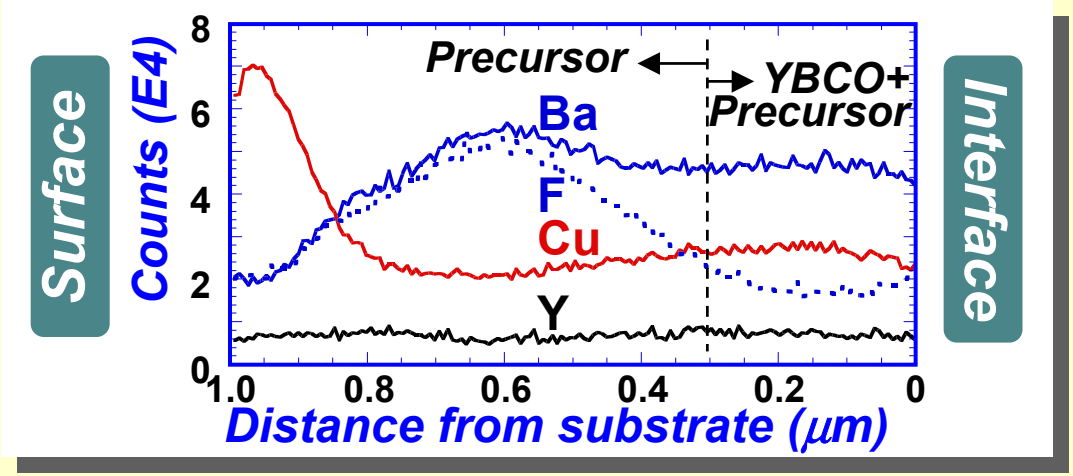


- XRD and pole figures indicate low YBCO content and poorly developed HTS cube texture for conversion time < 180 min.

Results show phase segregation in thick precursor when "high" $P(H_2O)$ is used

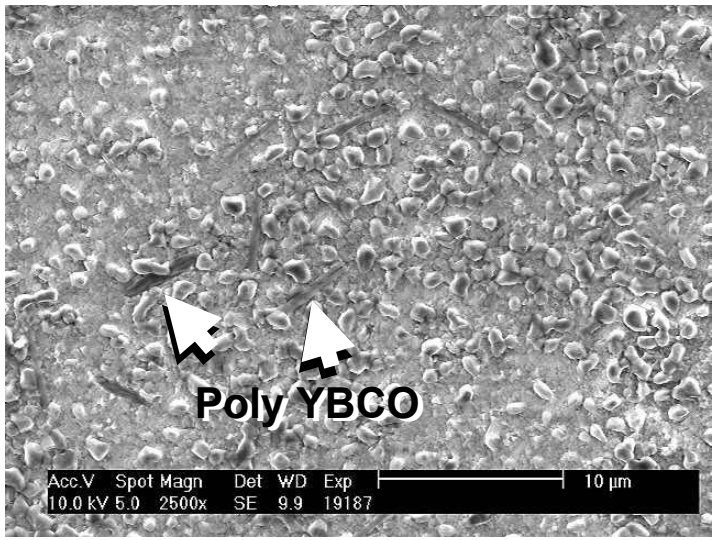
- Significant amount of grayish particles is seen on sample surface.
- MIT (Cima): Higher J_c at lower $P(H_2O)$.
  ORNL (Feenstra): [XRD] High $P(H_2O)$ → poly YBCO.
- Short-sample experiments by varying $P(H_2O)$ over almost 2 orders of magnitude:
  Gray area decreases with $P(H_2O)$ → from total coverage to none.
- Long-length thick precursor was partially converted under high $P(H_2O)$ condition and quickly quenched onto cold reel:
  - Grayish particles present on entire tape (conv. Time = 0 to 120 min).
  - R-R XRD: near constant non-zero YBCO(103) intensity.

- Depth profile Auger showed Cu segregation to surface and higher Ba concentration in center of unconverted precursor.

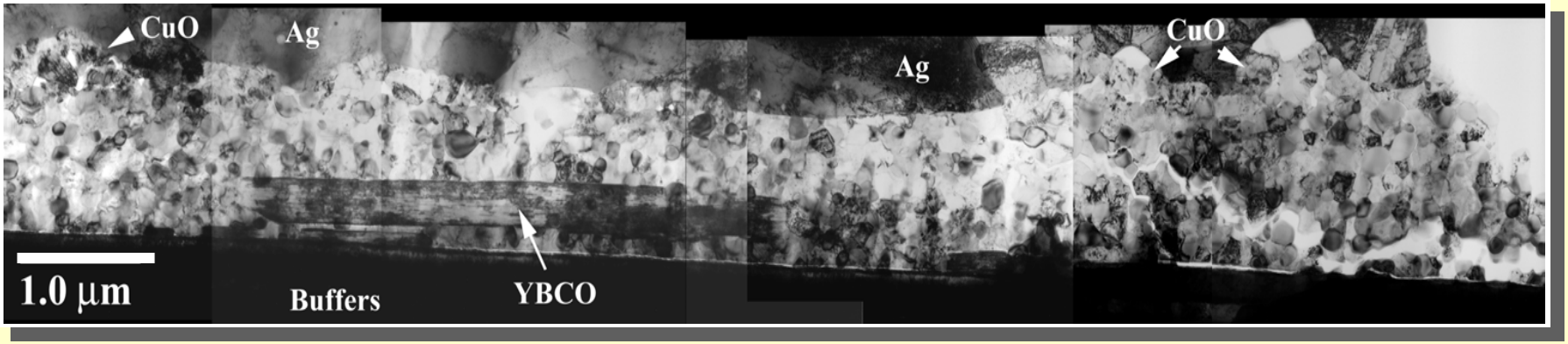


SEM & TEM confirmed that CuO particles had formed on the sample surface under high $P(H_2O)$

- In addition to CuO, randomly oriented YBCO are also seen at the surface of partially converted samples. (35 min.) →

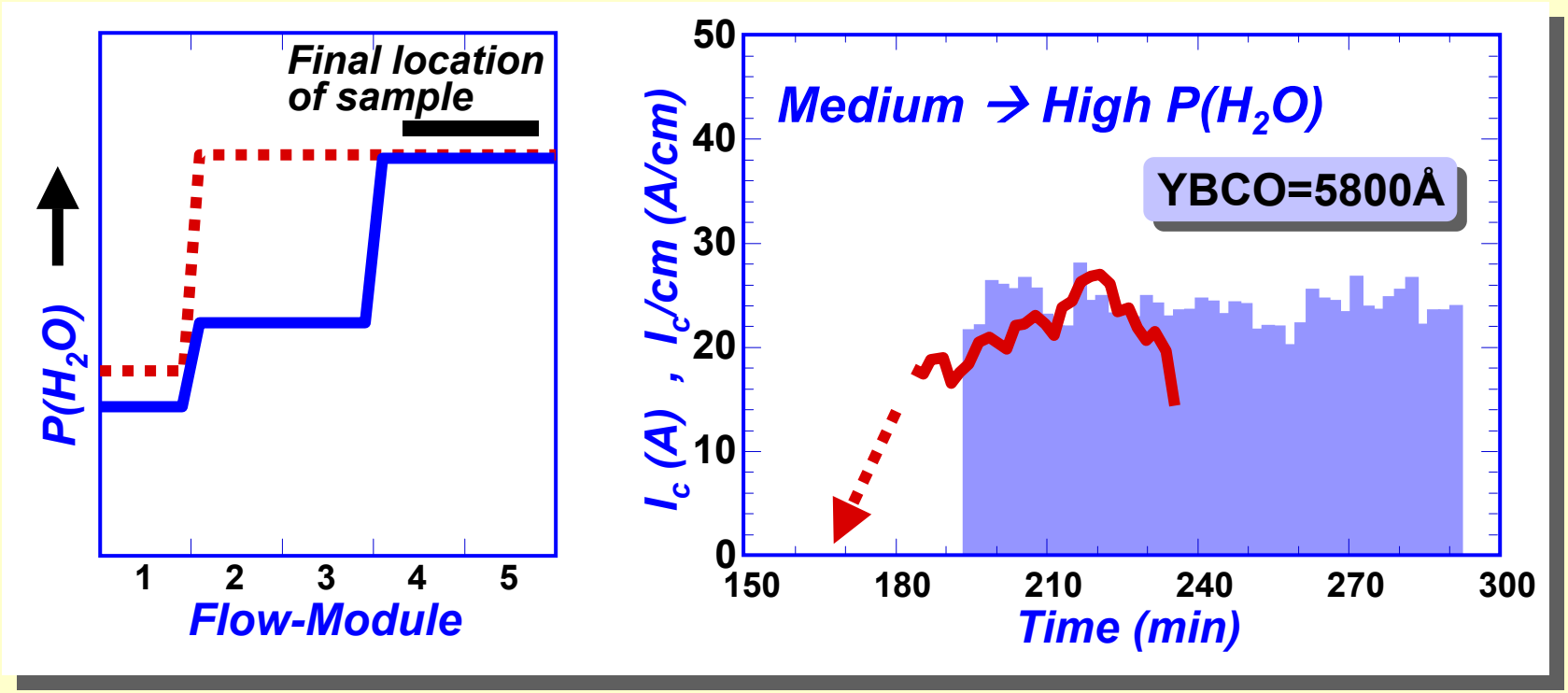


- Non-continuous cube-textured YBCO found at substrate interface.



Processing window is widened when $P(H_2O)$ is lowered

- Few small surface regions with grayish particles.

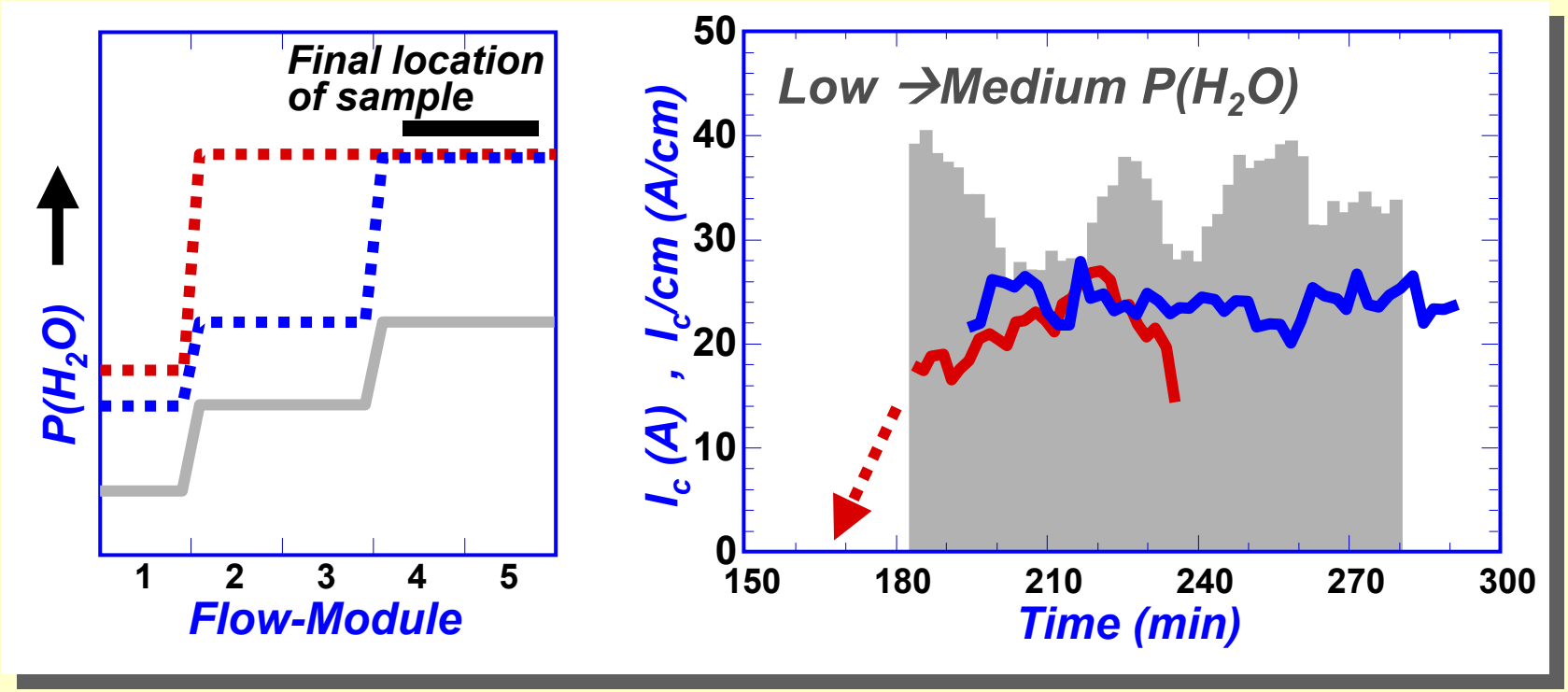


- Length of time necessary to achieve same I_c does not appear to have increased!
- Longer dwell time in high $P(H_2O)$ does not appear to have degraded the J_c!!

Higher I_c and wide processing window when $P(H_2O)$ is lowered further

- NO grayish particles seen.

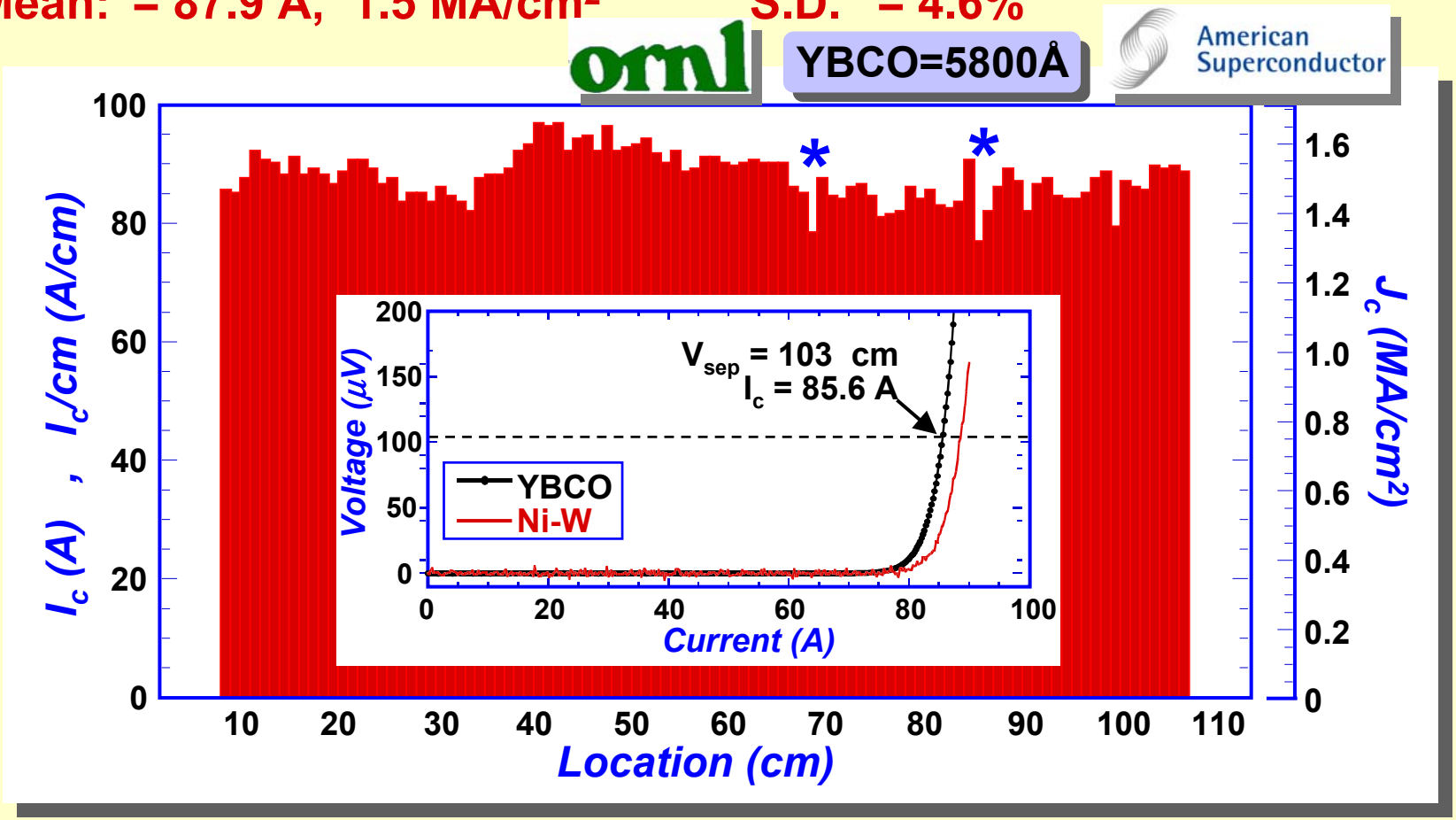
YBCO=5800Å



- Higher I_c, shorter time.
- Fluctuation in I_c mainly due to substrate texture.
- Identical processing on different precursor did not show I_c fluctuation.

Using low to medium P(H_2O), 1.15 meter of 5800Å-thick precursor on AMSC/ORNL Ni-3%W has been converted

- End-to-end I_c = 85.6 A, J_c = 1.5 MA/cm^2.
- Max: = 96.9 A, 1.7 MA/cm^2 Min: = 77.0 A, 1.3 MA/cm^2
  Mean: = 87.9 A, 1.5 MA/cm^2 S.D. = 4.6%

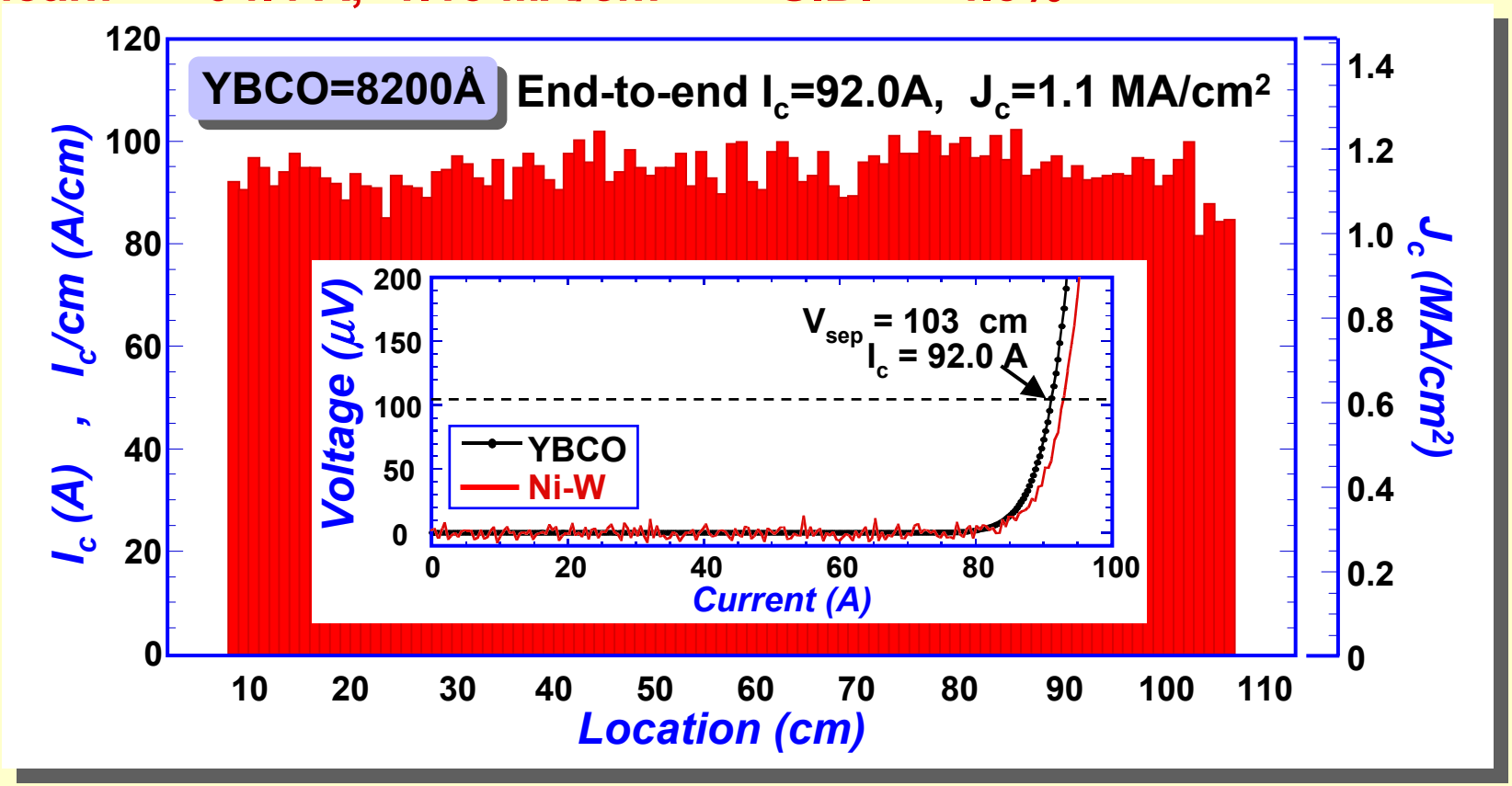


FY2002 Results:

~8000Å & Thicker Precursors

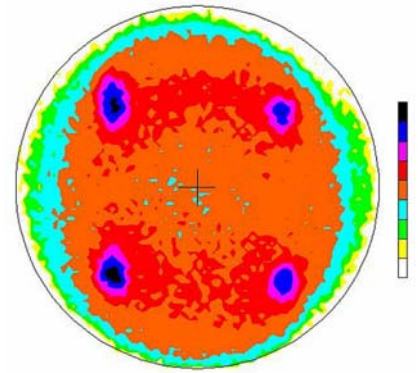
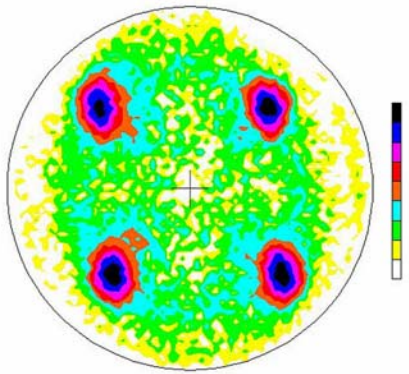
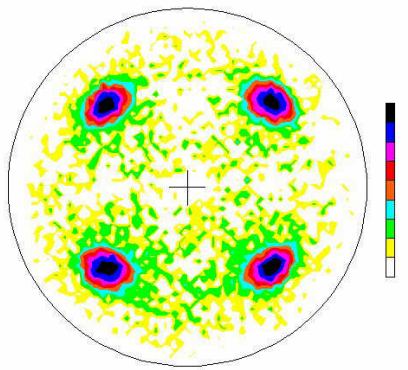
High and uniform J_c has been obtained on 8200Å precursor on all-ORNL Ni-W RABiTS processed under non-optimized conditions

- First few ORNL Ni-W RABiTS contained (111) with spikes → from Ni.
- 1cm X 115cm precursor on All-ORNL $CeO_2/YSZ/Y_2O_3/Ni/Ni\text{-}W$.
- Max: = 102.2 A, 1.25 MA/cm² Min: = 81.5 A, 0.99 MA/cm²
  Mean: = 94.4 A, 1.15 MA/cm² S.D. = 4.3%



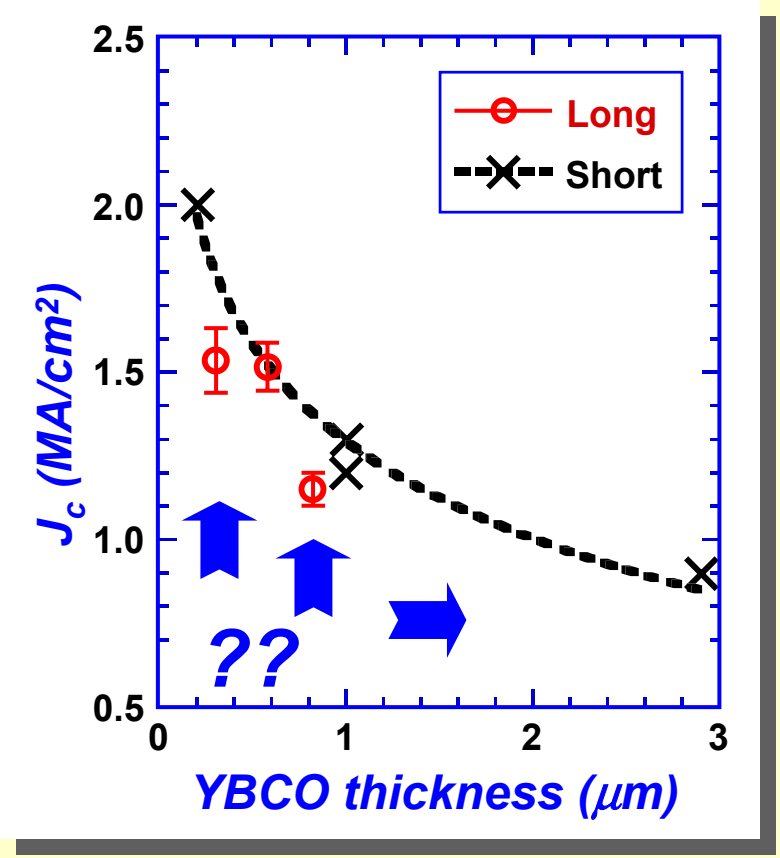
High I_c has been obtained on 1μm precursor / Nickel RABiTS in our reel-to-reel furnace

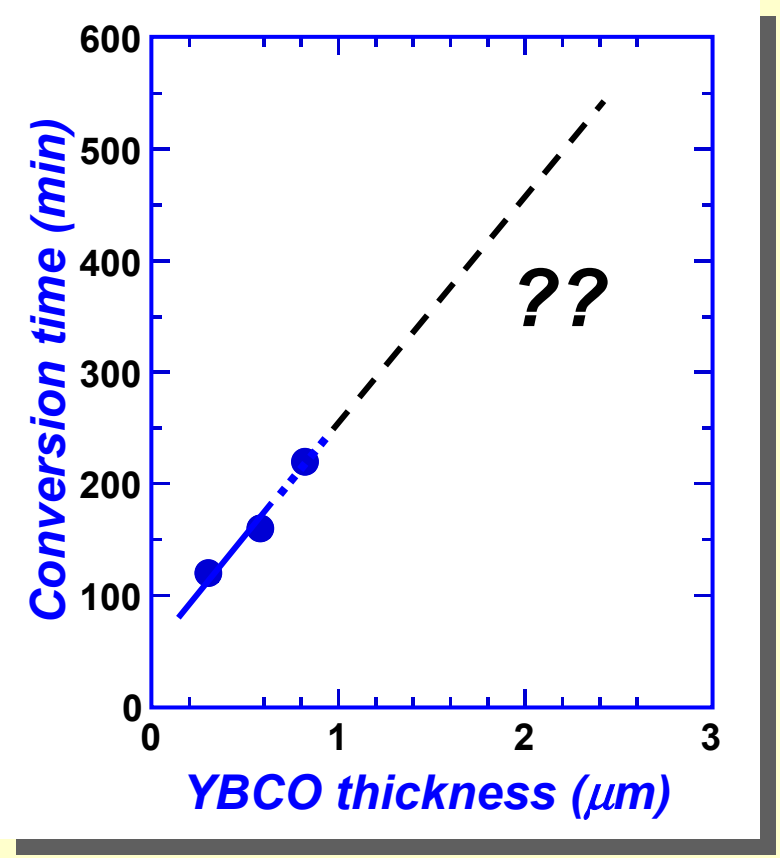
- 7cm-long samples cut from a length of 1μm precursor / CeO_2 / YSZ / Y_2O_3 / Ni RABiTS.

Very high $P(H_2O)$	*Med → high $P(H_2O)$*	*Low → Med $P(H_2O)$*
		
YBCO(005) = 5750 cps	***YBCO(005) = 18500 cps***	***YBCO(005) = 32000 cps***
64% cube	***90% cube***	***96% cube***
I_c=zero	*I_c=20A*	*I_c=70A*

- Past results show that it is possible to double the I_c on Ni-W RABiTS.
- Present development of ORNL Ni-W RABiTS → no (111) component.
- With the availability of good fully-buffered Ni-W RABiTS, we are now poised to investigate the conversion of even thicker precursors.

J_c's of meter-length ex-situ coated-conductors approach those of short PLD & ex-situ samples





- As precursor thickness increases,
  - Will the J_c's match or exceed those of short samples?
  - How long will it take to convert the precursors?

FY2002 Results Summary:
Long Length RABiTS Development

- ✓ Improve texture of metal substrates (Ni and alloys).
- ✓ Demonstrate *uniform* epitaxial seed layers using sulfur.
- ✓ Prepare long-length *strengthened* RABiTS.

- Developed highly textured long-length Ni-3%W alloy tape:
  - FY2001 Ni: $\Delta\omega$ FWHM ~ 8° $\Delta\phi$ FWHM ~ 10°
  - FY2002 Ni-W: $\Delta\omega$ FWHM ~ 5° $\Delta\phi$ FWHM ~ 7°
- Uniform in-situ reel-to-reel sulfurization of Ni and Ni alloy.
- Replacement of crack-prone CeO_2 with strong and dense Y_2O_3 seed layer.
- Produced lengths of completely buffered strengthened Ni-W RABiTS with uniform texture as determined by R-R XRD.

FY2002 Results Summary:
"Large Area" YBCO conversion- Lower Pressure

- ✔ Investigate *large-area batch* conversion parameters for YBCO precursor.
- ✔ Determine the effectiveness of reduced pressure in aiding conversion.

- **Batch furnace**: Complete YBCO conversion in length (Feenstra: strategic session).
- **Low pressure sys:** Enables in-situ monitoring of YBCO formation & phase development → High J_c achieved at very low pressure.
- **Reduced Pressure furnace**: Preliminary results show beneficial effects on J_c and homogeneity.

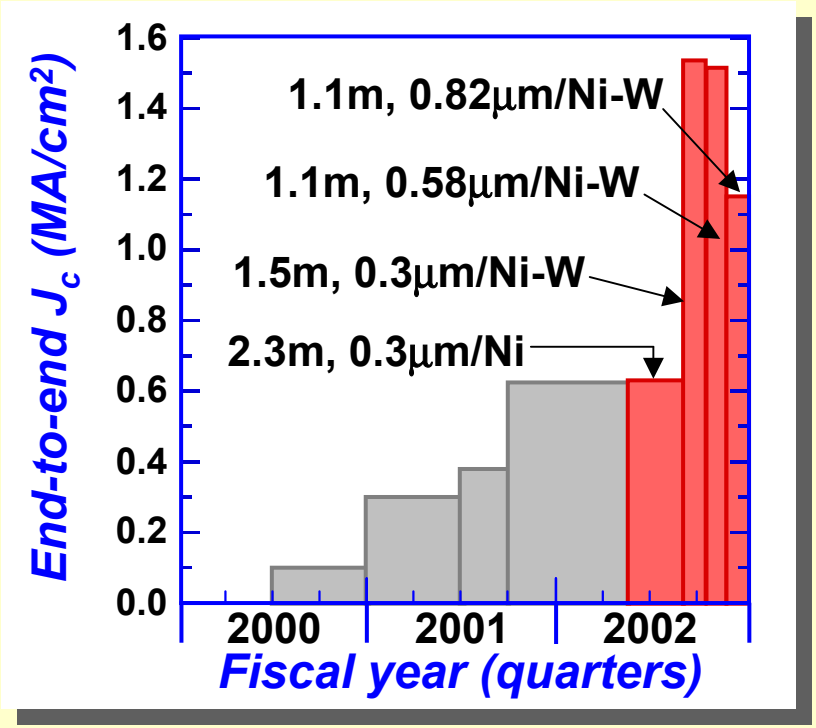
System	*P (atm)*	*J_c (MA/cm^2)*	*Gas consumption (l/min)*
Low pressure	2 E –4	1.3	LOW
Reduced pressure	0.35	1.5	1
Reel-to-reel	1.5	1.3	>5.5

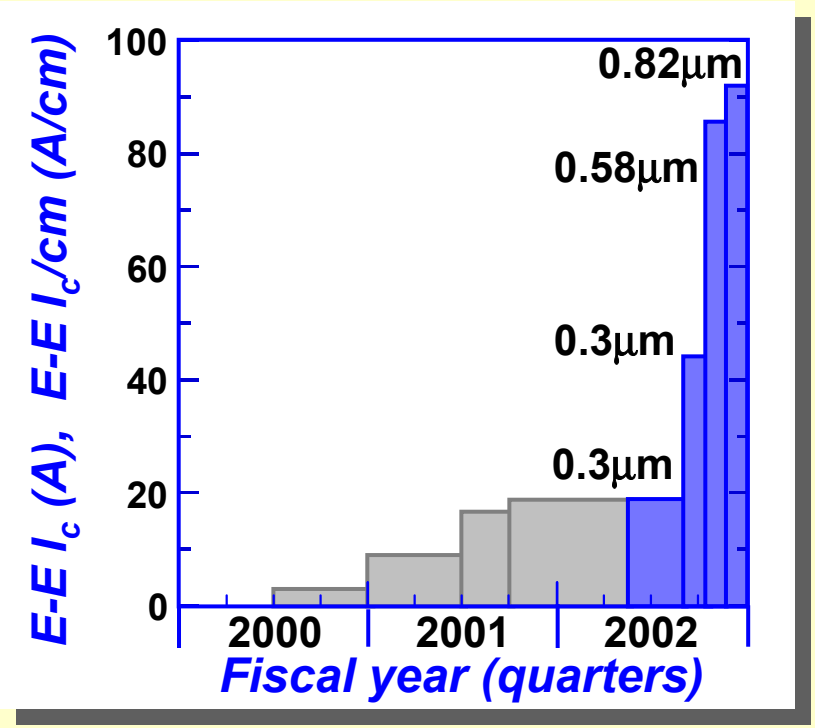
Wide range of conversion pressures gives manufacturers added flexibility.

FY2002 Results Summary:
"Large Area" YBCO conversion- Reel-to-Reel

- Compare conversion parameters for different precursors.
- Obtain 1MA/cm^2 on meter-length 3000Å-thick YBCO on *strengthened* RABiTS.
- Study basic reel-to-reel conversion parameters for *thick* YBCO precursor.

- Different precursors DO affect precursor decomposition / YBCO formation rate.





FY2003 Plans

Long-length RABiTS development:

- Utilize our rolling mill to develop and improve the texture of alloys.
- Develop and optimize buffer(s) deposition procedures on these alloys.
- Examine the scaleability of alternative or simpler buffer architectures.

Ex-situ YBCO conversion:

- Using our Low & reduced pressure systems:
  - Explore YBCO conversion characteristics over the allowable pressure range.
  - Study the effect of precursor thickness on conversion parameters.
  - Examine the effects of reduced pressure on sample homogeneity.
- Reel-to-reel conversion:
  - Continue to study the conversion characteristics of thicker precursors.
  - Explore the effects of "other" parameters → reduced pressure if possible.

External collaborations:

- Continue to assist partners in achieving their programmatic goals.
- Provide collaborators with RABiTS, YBCO and assist in ex-situ YBCO conversion on their substrates.

Research Integration

3M Innovation

- 2-way long-length samples exchange including RABiTS with and without precursor.
- R-R XRD of RABiTS & YBCO tapes.
- Weekly teleconferences, etc.

American Superconductor

- 2-way long-length RABiTS exchange.
- R-R XRD of RABiTS & YBCO tapes.
- Planned visit of ACCL by AMSC personnel.
- Weekly teleconferences, etc.

MCT Microcoating Technologies / Oxford Instruments

- R-R XRD of RABiTS and metal substrates.
- YBCO deposition on MCT RABiTS.
- ACCL visit by MCT personnel.
- Teleconferences, on-site meetings, etc.

NIST
- Provided samples from R-R furnace for strain measurements.

LANL
- Continuing collaboration through base program & 3MCRADA.

SNL
- Planning stage of precursor and substrate exchange.

FSU
- Continue to provide samples from R-R furnace for temperature and strain measurements.

U. Kansas
- Provided uniform short samples from long-length RABiTS.