CONTINUOUS PROCESSING OF COATED CONDUCTORS

OAK RIDGE NATIONAL LABORATORY

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Oak Ridge Team

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Funding

- **FY 2001 EE/RE Funding: $1500K**
  - **5 FTE Staff, 4 FTE Post-Doctoral Fellow**

Industrial Collaborators

- **3M Company (3M)**
  - **Myles Brostrom, Dick Ericson, Dave O'Neill, Bill Robbins, Jonathan Storer, Ed Yu**
- **American Superconductor Corporation (ASC)**
  - **S. Annavarapu, Les Fritzemeier, Q. Li, Marty Rupich, Bart Riley, U. Schoop, C. Thieme, Z. Zhang**
- **MicroCoating Technologies (MCT)**
  - **Adam King, Totd Polley, Shara S. Shoup, Marvis White**
- **Oxford Superconducting Technology**
  - **Ken Marken**

COATED CONDUCTOR DEVELOPMENT AT OAK RIDGE NATIONAL LABORATORY

GOAL: Develop, with industry, technology enabling the manufacture of REBCO conductor

APPROACH: Scalable methods were selected to

- Make textured substrates—RABiTS
- Form epitaxial superconductor deposit
  --Ex-situ conversion of BaF_2-based precursor for continuous processing of lengths

High Tape Production Rates Appear Possible With Ex-situ Conversion of BaF_2 Precursor

YBCO Growth rates in the range of 1-3Å/s appear achievable.

Reel-to-reel system

For conversion of a 1μ thick layer in a 10m long by 30cm wide reaction zone

- **Tape speed=3.5-10.5 m/hr**
- **Production rate=105-315 m/hr of 1cm wide tape**

Batch process

A 32 cm dia. by 1 m long mandrel holds ~100m of 1 cm wide tape. At 1 Å/s growth rate and 1μ thickness the conversion time ~3 hr.

Issues include

- HF concentration, distribution over the tape-- reduced pressure
- Quantity of gas to be handled—reduced pressure
- Slitting

ORNL Has a Broad Coated Conductor Effort Aimed at Understanding As Well As Process Development

- **Thermo-mechanical processes for texturing metals**
  - **Alloys**
  - **Industrial "supply chain"**
- **Epitaxy of oxides on metals**
  - **Effects of surface chemistry and structure**
    - **Sulfur superstructure**
    - **Alloys**
  - **Non-vacuum deposition processes**
    - **Sol-gel, MOD**
  - **Reel-to-reel deposition**
  - **Conductive buffer layers**

ORNL Effort (continued)

- **Ex-situ approaches to superconductor formation**
  - **Co-evaporation and TFA**
  - **Studies of the conversion process**
  - **Thickness dependence of J_c**
  - **Reel-to-reel conversion of lengths**
  - **Large area conversion; batch system, low pressure process**
- **Grain boundary effects on J_c**
  - **Ca-doping**
  - **Bi-crystals**
- **Conductor stability and normal zone propagation**
- **Characterization of lengths**
  - **Reel-to-reel XRD**
  - **I_c measurements**

The YBCO Deposition Effort at ORNL Is Being Strengthened

Short sample research

- **PLD**
- **Ex-situ**
  - **co-evaporation of BaF2 precursor**
  - **TFA**

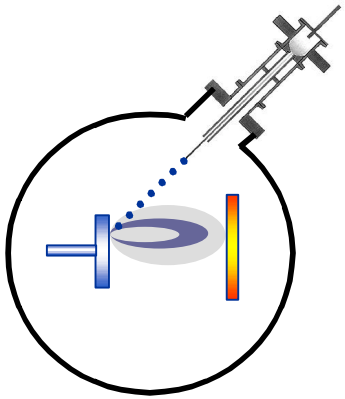
Continuous processing

- **Ex-situ conversion of e-beam deposited precursor-3M and ORNL**
- **TFA, precursor from AMSC**

New cooperative agreement with Neocera, Inc. is focused on new process: Pulsed Electron Deposition

YBCO Growth by Pulsed Electron Deposition (Cooperative Agreement with Neocera, Inc.)

- A unique **physical vapor deposition** technique using a self-pinched, pulsed electron beam to ablate from a solid target
- Comparable to PLD, but a flange mounted unit plus 20 kV power supply **replaces 100 W excimer laser** and optics
- ***Cooperative Agreement:***
  1. Optimization of YBCO on $SrTiO_3$ (growth at Neocera, characterization at ORNL)
  2. YBCO on RABiTS (growth at Neocera, RABiTS and characterization provided by ORNL)
  3. Installation of PED unit at ORNL
  4. Discussion of issues related to scalability



Accelerated Coated Conductor Initiative (ACCI) Facilities

- **Reel-to-reel systems:**
  - **High-rate RF sputtering for buffer layer deposition (new)**
  - **E-beam BaF_2 precursor deposition (relocated)**
  - **Four-circle XRD characterization (new)**
  - **Transport and inductive J_c characterization (new)**
  - **Tape cleaning and handling equipment (relocated)**
  - **Reel-to-reel laser surface roughness measurement (new)**
- **Batch and short sample systems:**
  - **6-m batch reactor for BaF_2 precursor conversion (new)**
  - **Low pressure batch reactor with EDX and RGA (new)**
  - **Warm-bore, 9T magnet for J_c characterization (new)**

Important Results Were Obtained on Buffer Layer Properties and Surface Chemistry

- **In our reel-to-reel rf-annealing/CeO_2 deposition system, poor reproducibility of seed buffer layer texture was found to be a serious problem.**
- **A sulfur superstructure on the metal surface, formed by surface segregation, was found to promote epitaxy of the desired orientation in the seed layer.**
- **On-line surface analysis in the R-R system indicated**
  - **Incomplete superstructure causes non-reproducibility,**
  - **Amount of sulfur varied with**
    - **Texturing anneal**
    - **Source of substrate**
- **A complete superstructure can be obtained by a simple in-line process, leading to improved seed layer texture.**

FY2001 Plans

- **Improve the structure, processing and throughput of buffer layers:**
  - **Use Design of Experiment in conjunction with reel-to-reel equipment.**
  - **Develop equipment and procedures to improve tape handling to reduce defects.**
  - **Studies of oxide growth on metal surfaces.**

- **Further develop *Ex-situ* process to convert YBCO in 1 meter lengths:**
  - **Improve homogeneity of precursor and YBCO conversion.**
  - **Identify and optimize dominant processing parameters.**
  - **Target J_c of 1 MA/cm^2.**
  - **Collaborate with LANL to process YBCO on meter-length IBAD.**

- **Continue to assist collaborators with equipment design and conductor characterization:**
  - **Continue technology transfer of equipment design and ideas.**

Outline:
Continuous Processing of Coated Conductors

- **FY 2001 Results:**

  Progress on buffer layer deposition

  - **Background research on the effects of sulfur—Claudia Cantoni**
  - A sulfur template for buffer layers on lengths—Fred List
  - 3M results for long length, high rate buffer deposition—Fred List

  Conversion of YBCO(F) precursor in lengths—Dominic Lee

- **Summary of FY2001 Results/FY2002 Plans-**Dominic Lee

- **Research Integration-**Dominic Lee

Issues related to seed buffer layer deposition

- Introduction of continuous processing of coated conductors revealed unsuspected problems in seed layer fabrication, motivating careful strategic investigation

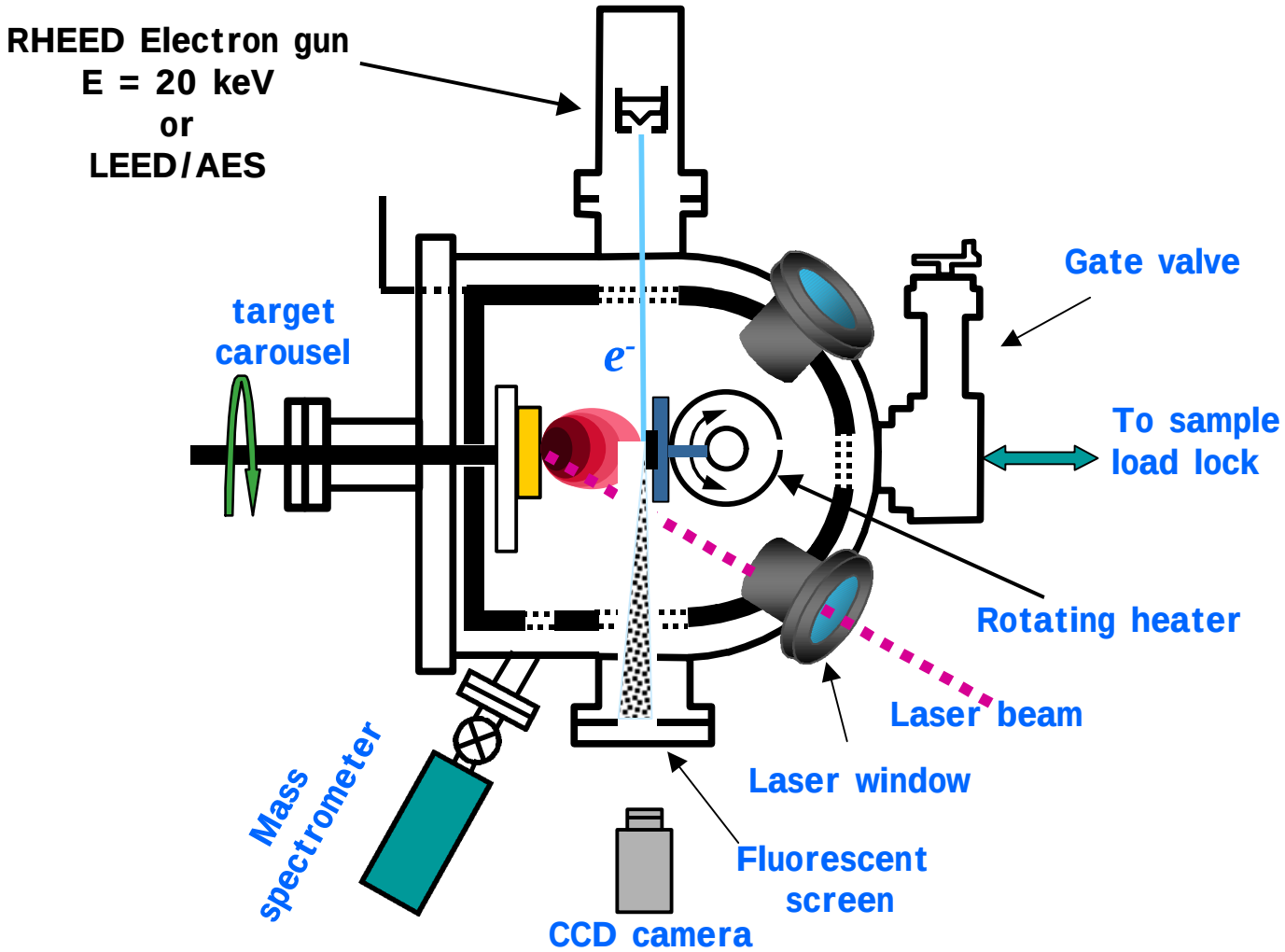
- **Understanding seed layer nucleation on Ni surface pointed out problem sources and suggested equipment refining**

- **A key step for controlling and optimizing seed layer texture has been introduced**

The nucleation of oxide buffer layers on metal surfaces is poorly understood

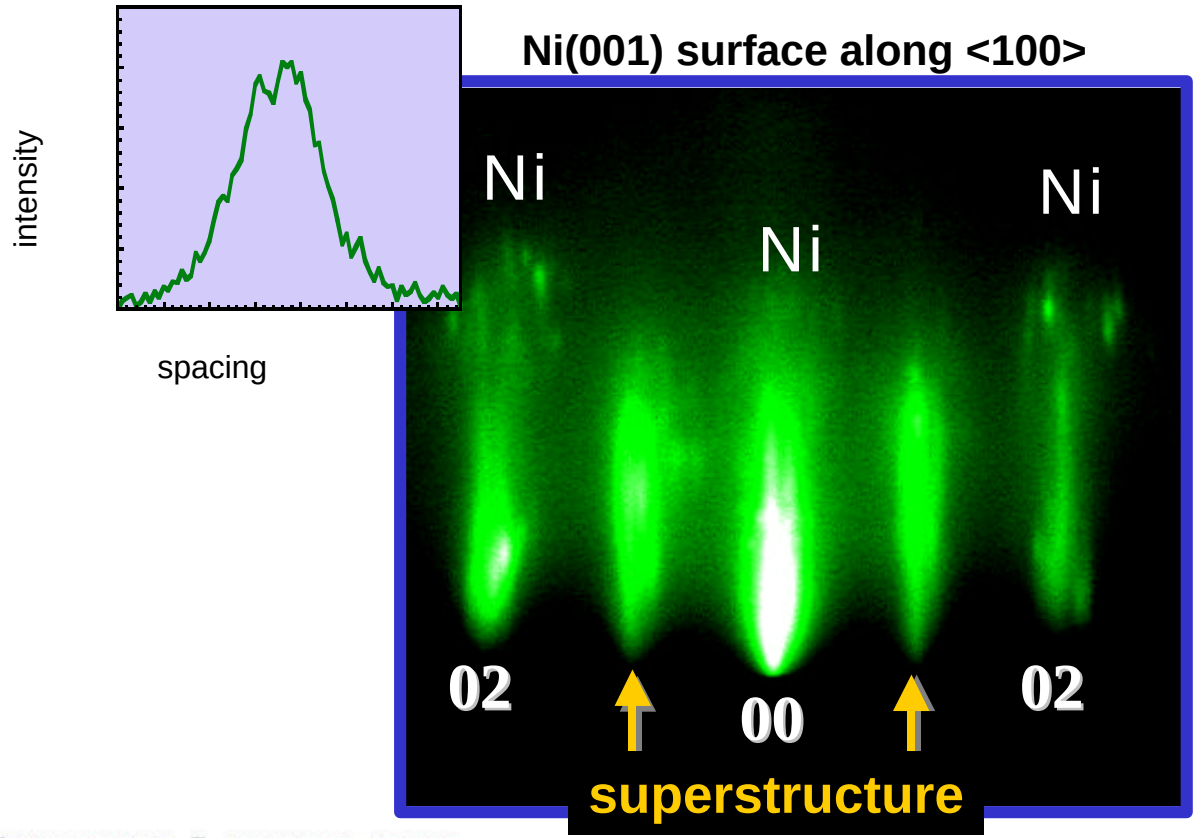
- What is the Ni/Ni-alloy surface structure/chemistry at the beginning of the oxide layer deposition?
- Does NiO or a Ni sub-oxide form and affect the oxide buffer layer growth?
- Do species adsorbed on the Ni surface affect oxide nucleation?

In situ RHEED was used to monitor oxides buffer layer growth processes



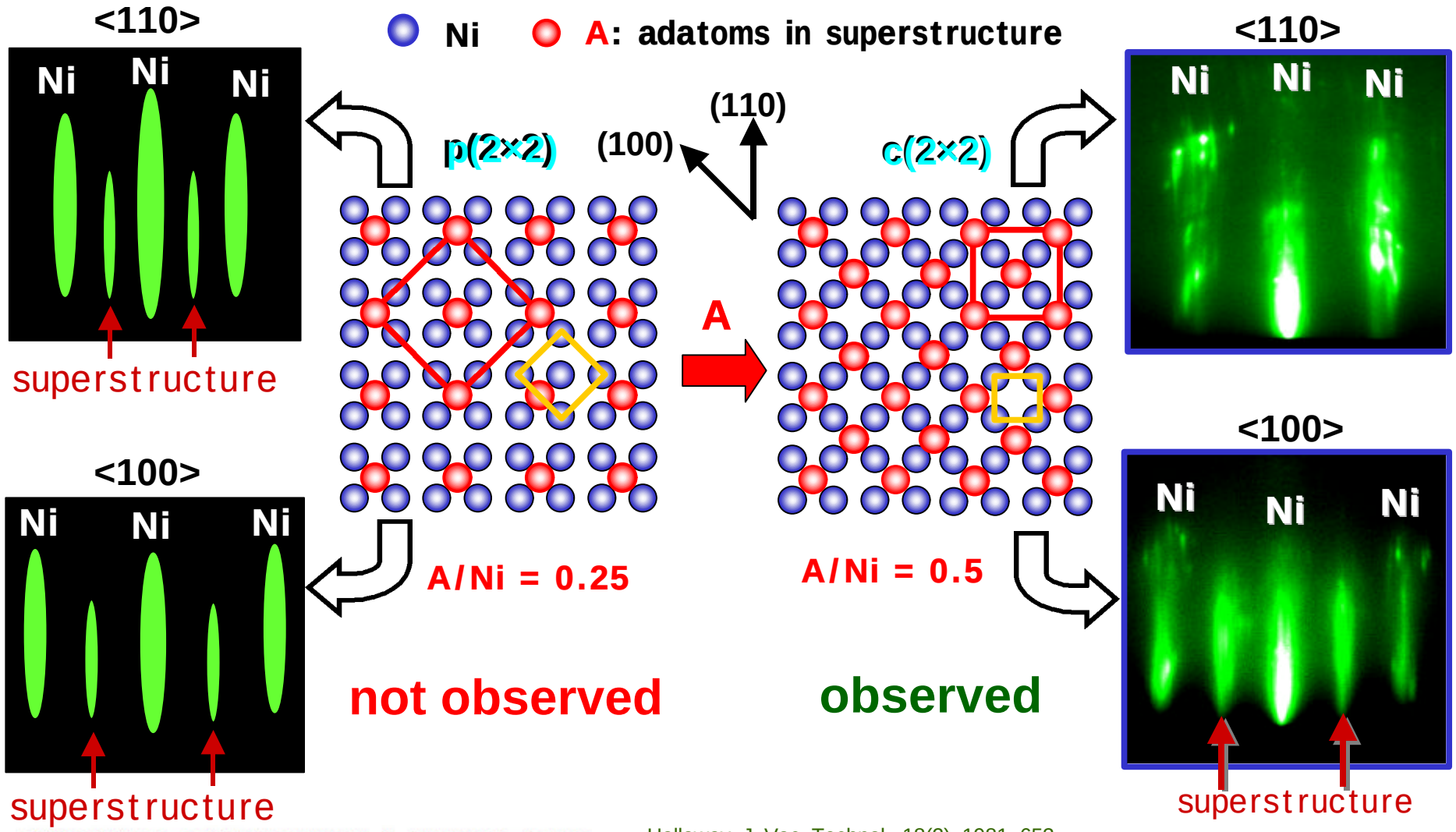
Textured Ni shows a complex surface

- Patterns are broadened due to the orientation distribution of the grains
- A superstructure is observed on most clean Ni surfaces
- Superstructure cannot be removed by annealing in 4%Ar/H_2



Two types of superstructures can be observed on Ni

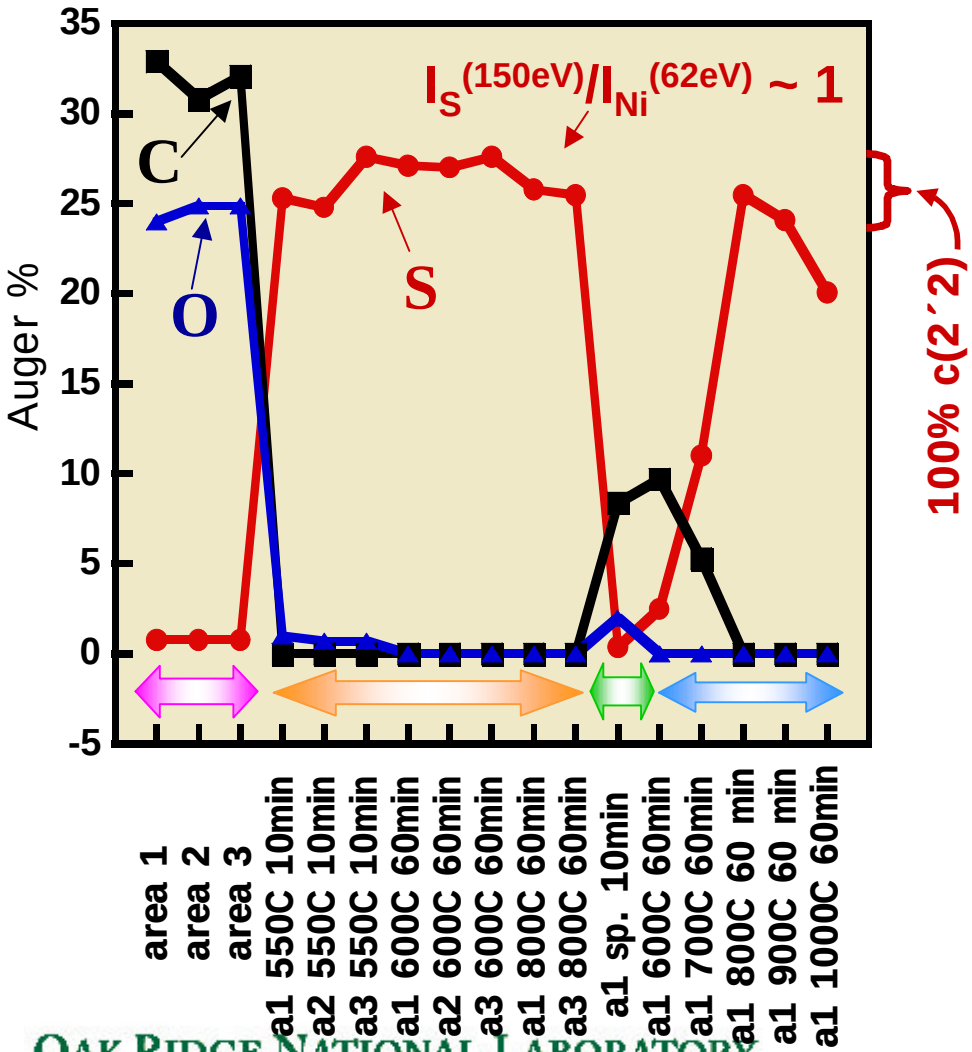
Possible species: C, CO, O, S…

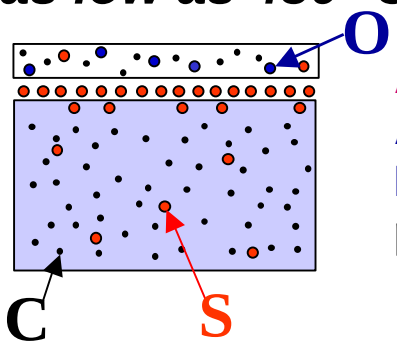


Holloway, J. Vac. Technol., 18(2), 1981, 653
Mitchell et al., Surface Sci. 61 (1976) 355
Fleisch et al. Surface sci. 78 (1978) 141

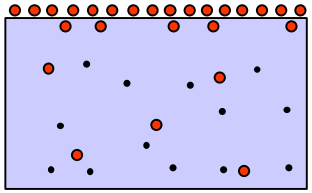
Summary of AES experiments

- *A stable S surface layer forms during the initial fabrication anneal*
- *S segregates to the surface at T as low as 430° C*

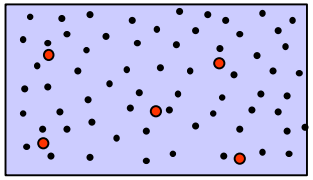




AS INSERTED
AES: S, C, O
RHEED: amorphous pattern

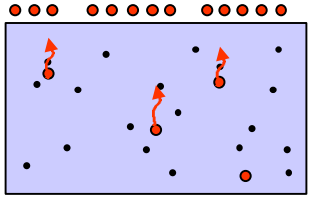


AFTER ANNEALING AT 550°C
AES: only S peak
RHEED: superstructure



AFTER ION SPUTTERING

AES: only C peak



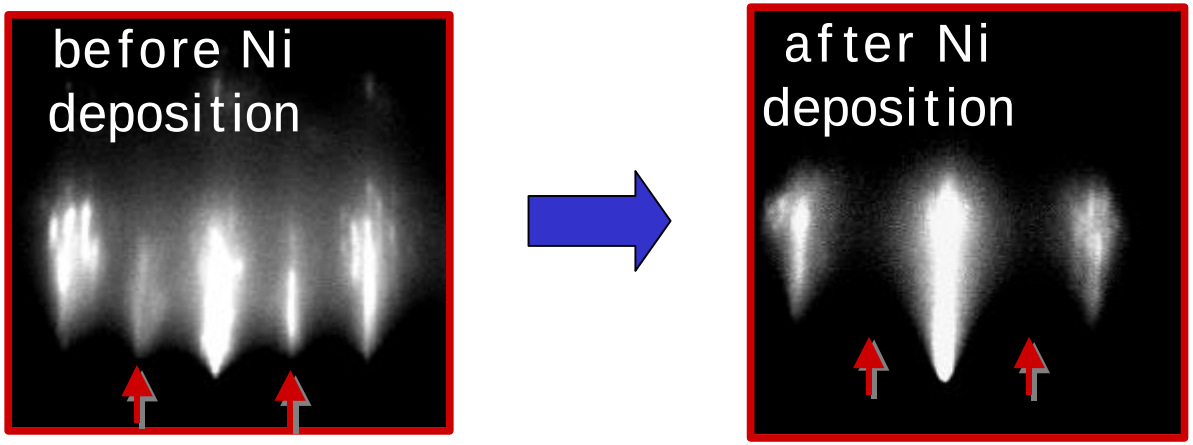
AFTER ANNEALING

AES: increasing S peak
decreasing C peak

PLD Ni overlayers do not show S superstructure

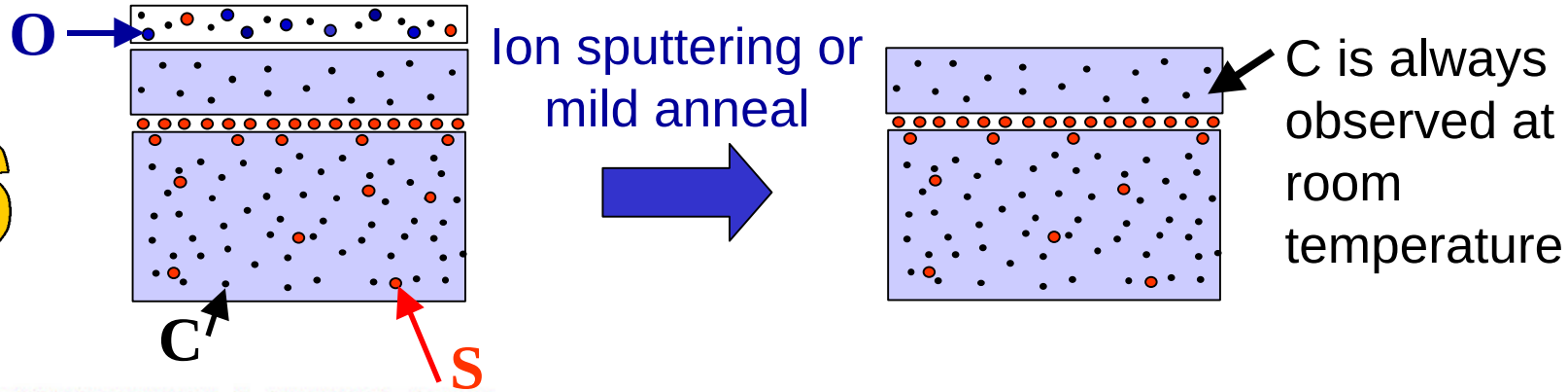
- ***500 – 1000 Å Ni film is deposited in situ on textured Ni***

RHEED



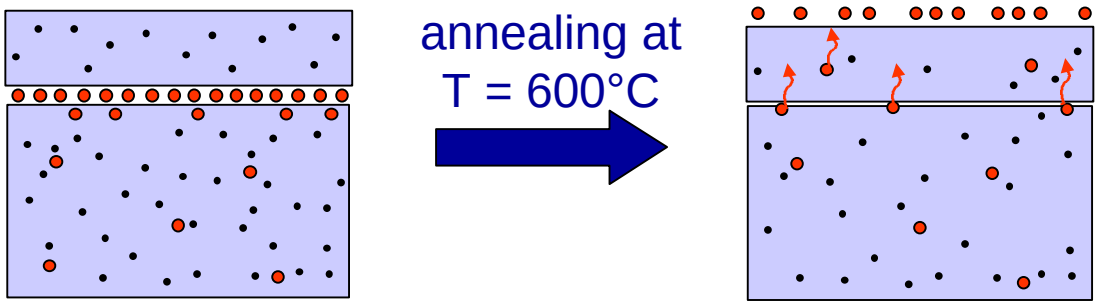
- ***AES data show that the Ni film contains no detectable S***

AES



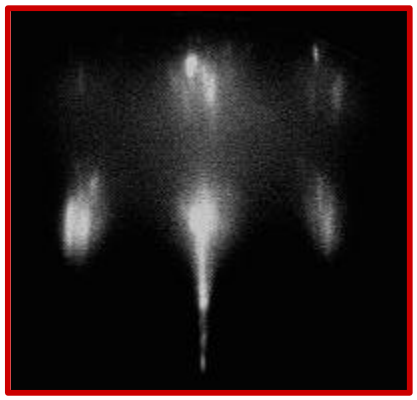
Sulfur diffuses to the surface during annealing

- ***S is detected only after anneals at T≥ 600◦C***
- ***Heating at T=750◦C produces a visible c(2×2) superstructure on a previously superstructure-free Ni overlayer***

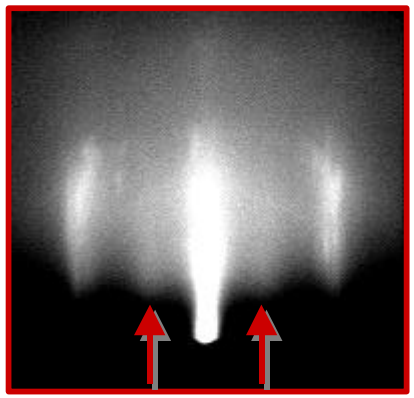


increasing S peak,
decreasing C peak

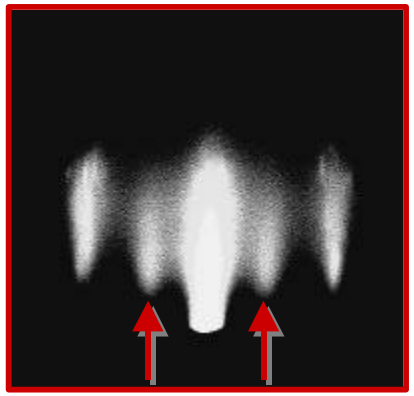
no c(2×2) | c(2×2) starts to form | c(2×2) continues to grow



T=350◦C



T=750°C, 1h

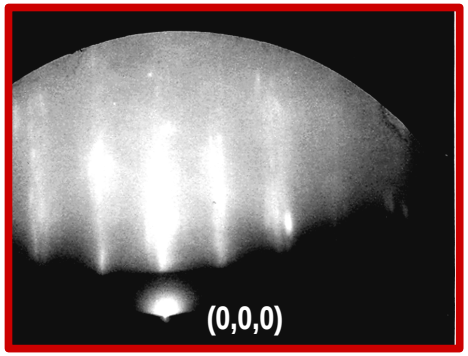


T=750°C, 2h

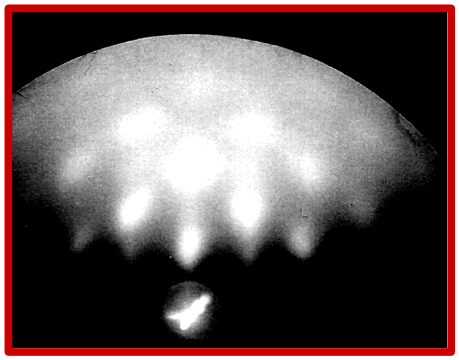
J. M. Grimal, Surface Science, 249 (1991) 171
A. Patrige et al., Scripta Met. Et Materalia 26 (1992) 847

Growth of YSZ on C(2´2) surface

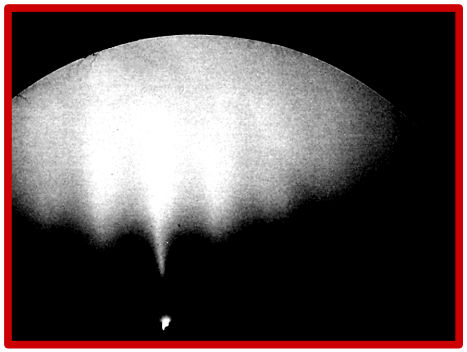
- YSZ nucleates (200) on the Ni superstructure
- A J_c of 1.15 MA/cm^2 was obtained for *ex situ* YBCO (BaF_2 method) using a thin CeO_2 cap layer (200 Å)



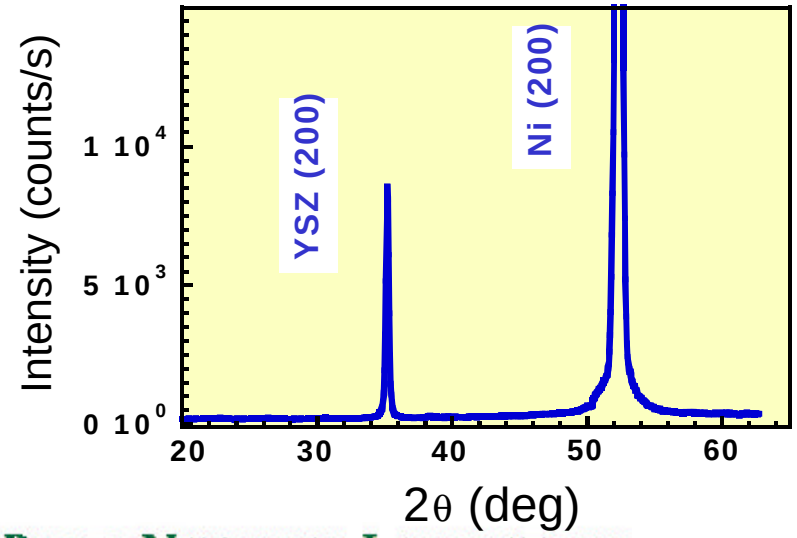
Ni + superstructure
T=800°C, P_b= 5×10^{-8} Torr



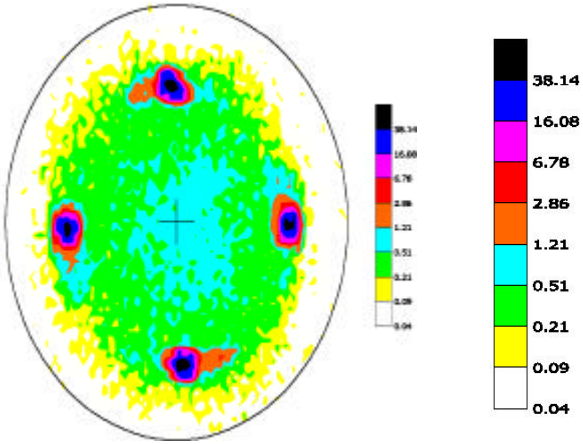
Nucleation of YSZ
first ~ 100 Å in vacuum



Final pattern
T=800°C, P(O_2) = 1×10^{-5} Torr

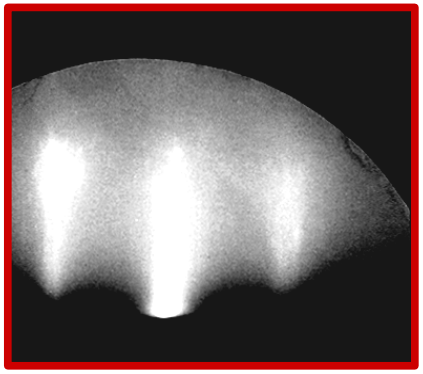


thickness=120 nm

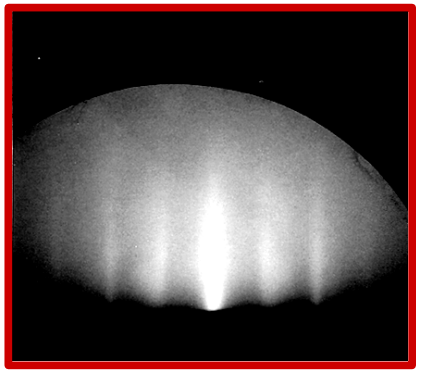


YSZ (111) log pole figure

Growth of YSZ on clean Ni surface without superstructure results in (111) epitaxy

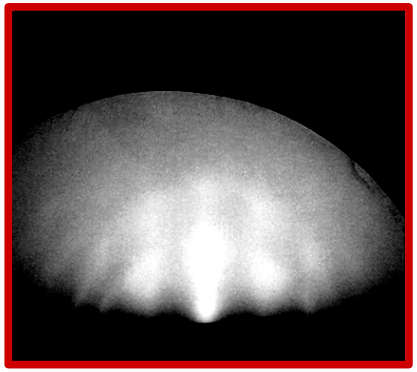


Clean Ni surface



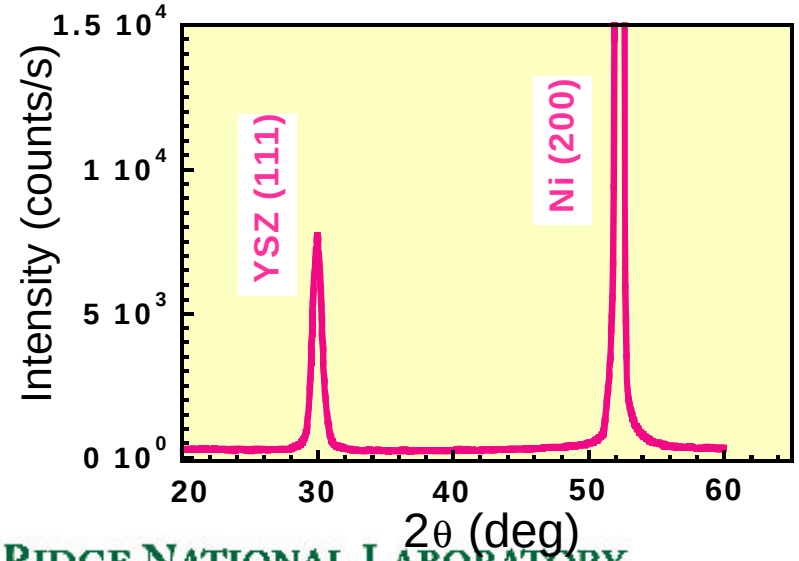
Nucleation of YSZ

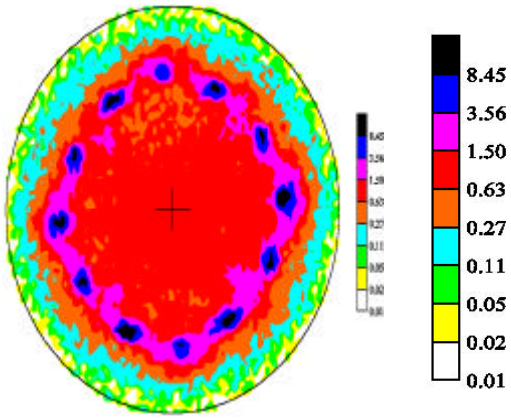
T=800 °C, P_b=5×10^{-8} Torr



Final pattern

T=800 °C, P(O_2)=1×10^{-5} Torr



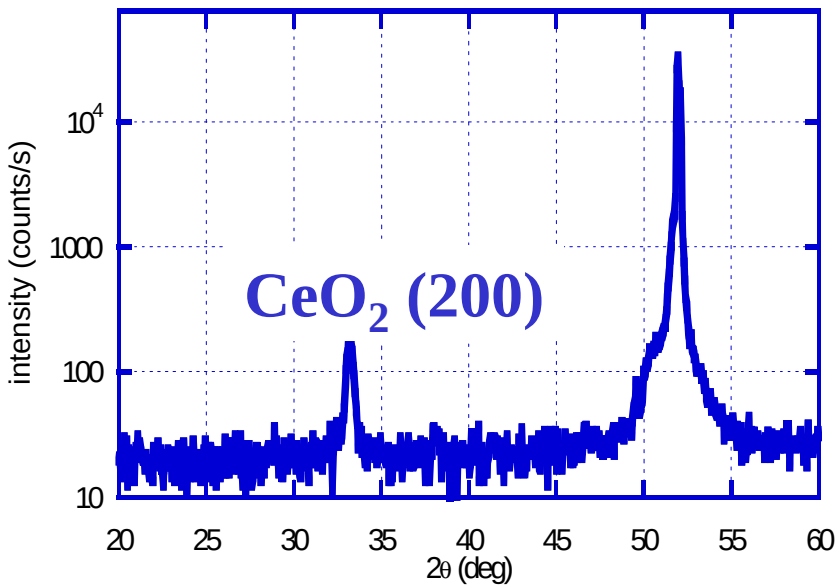


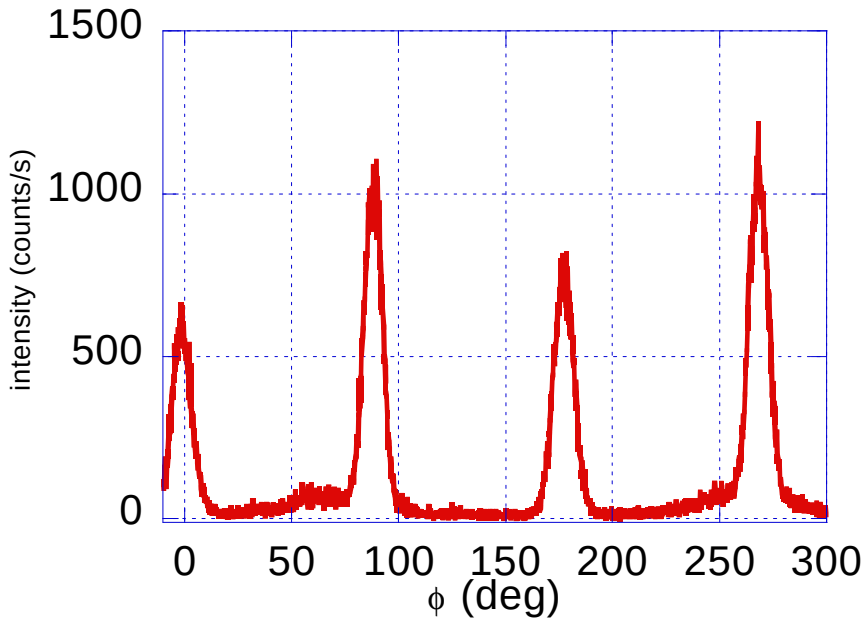
YSZ (200) log pole figure

Nucleation of other seed layers with fluorite structure: CeO_2

2 conditions are necessary to nucleate CeO_2 (001):

- ***c(2× 2)-S superstructure***
- ***H_2O background pressure ~ 1 × 10^{-5} Torr***

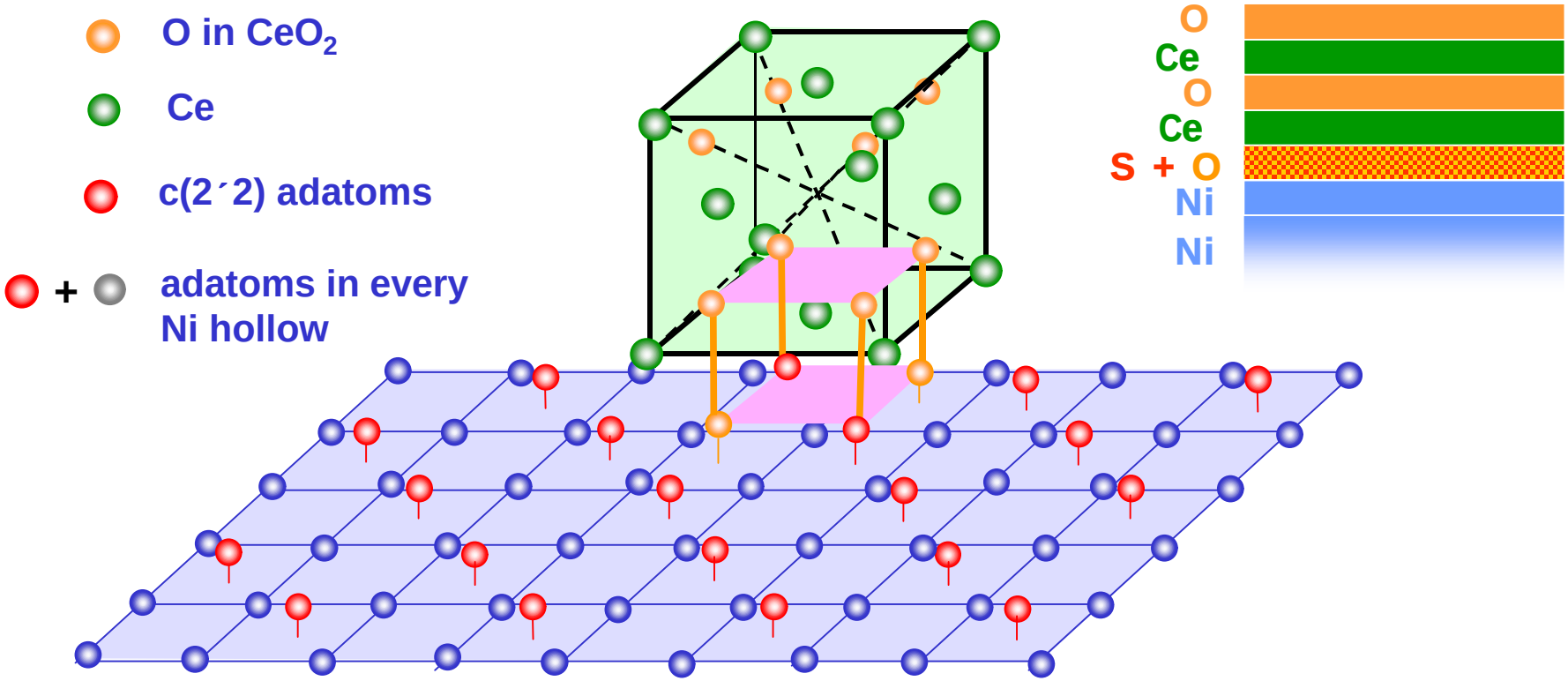




- ***$P[H_2O]=2.5 \times 10^{-5}$ Torr $P[H_2O]/P[H_2]=10^2$ T= 750°C***

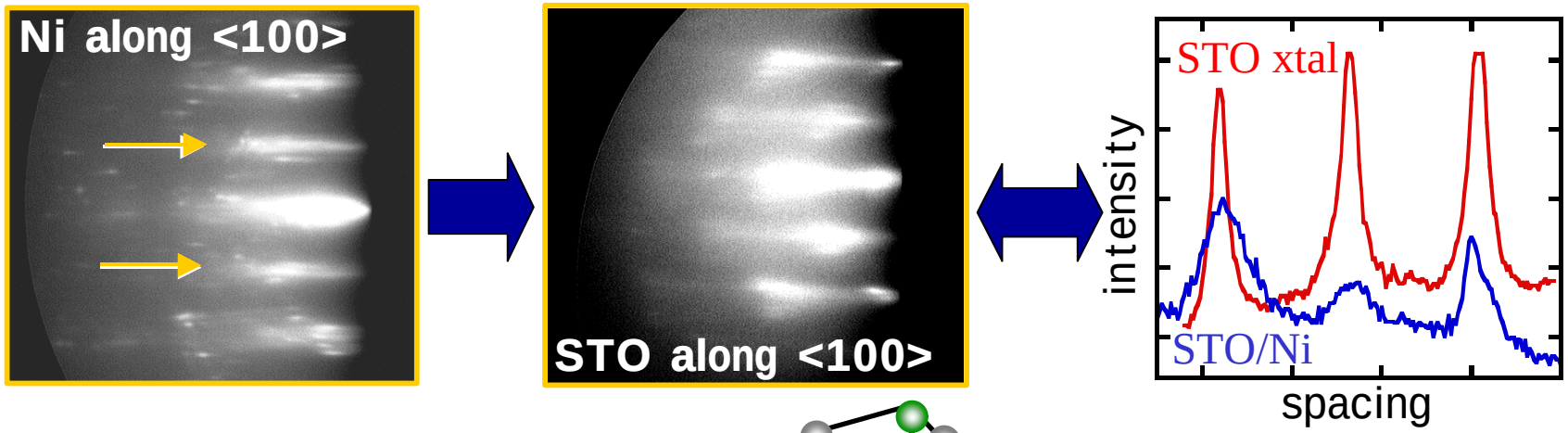
Proposed c(2×2) mediated epitaxial growth of YSZ and CeO_2 on (001) Ni surface

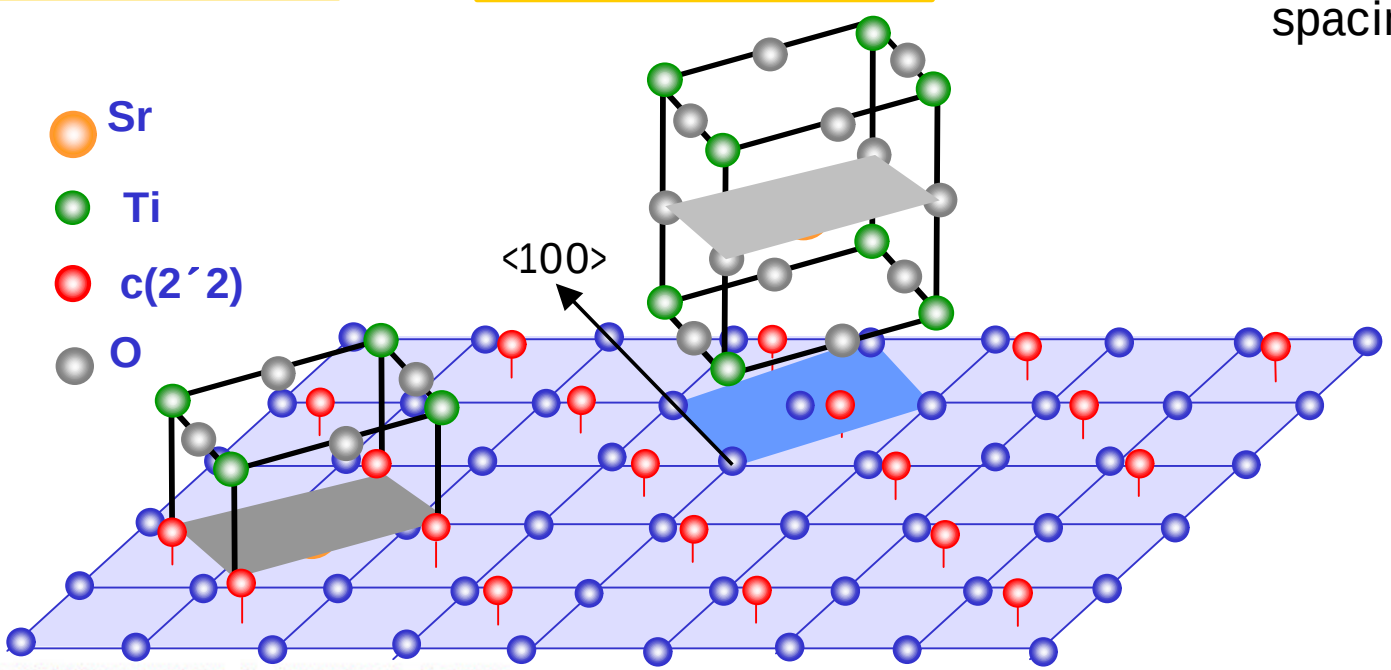
- Good lattice match between adatoms and CeO_2 (YSZ) oxygen sublattice
- c(2×2) adatoms become constituents of the interface layer between Ni and CeO_2 (YSZ)



Growth of Epitaxial STO on c(2 ´ 2)/Ni

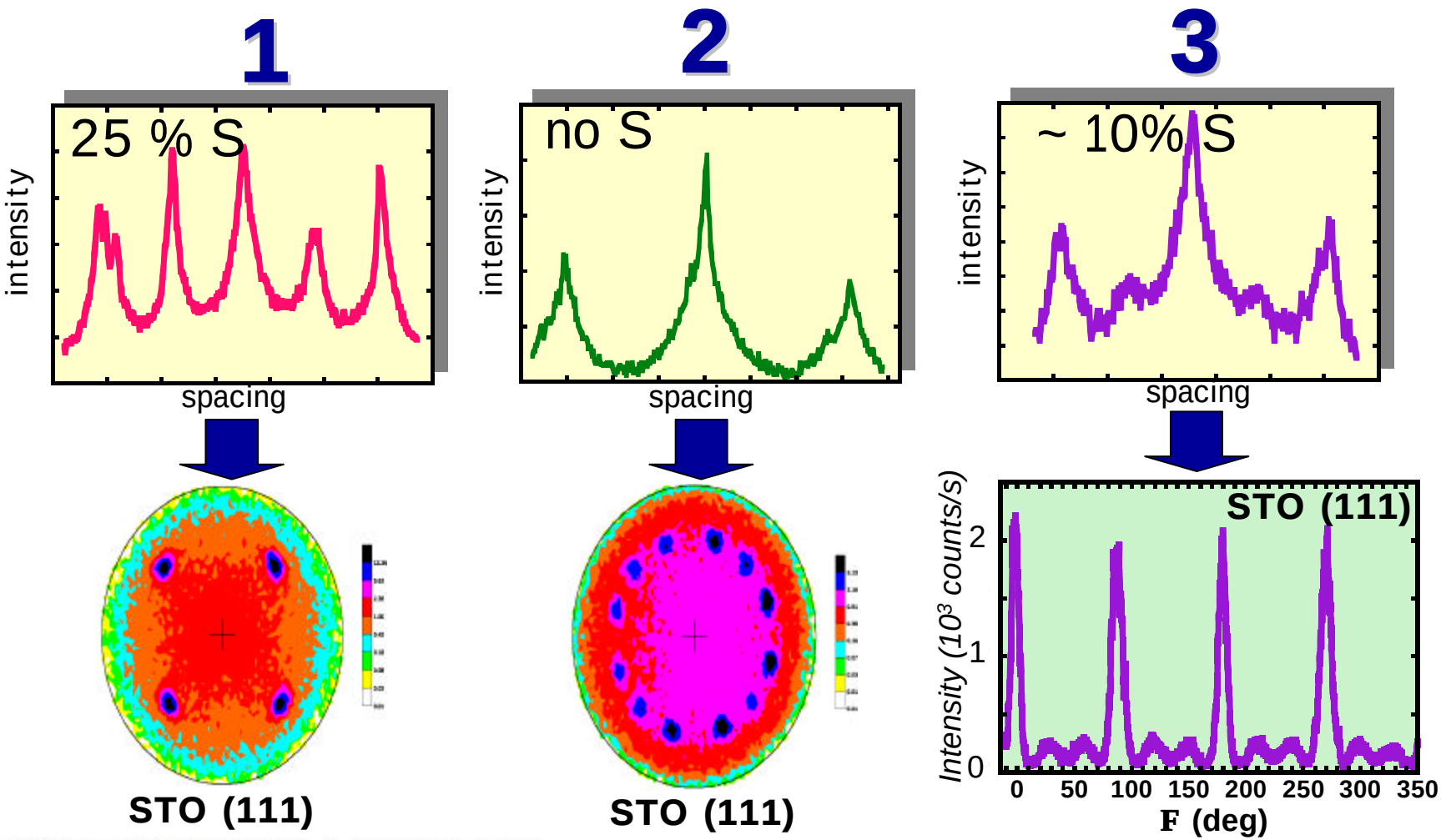
- *STO grows cube on cube on Ni with S superstructure T= 600 – 750° C, nucleation in vacuum*





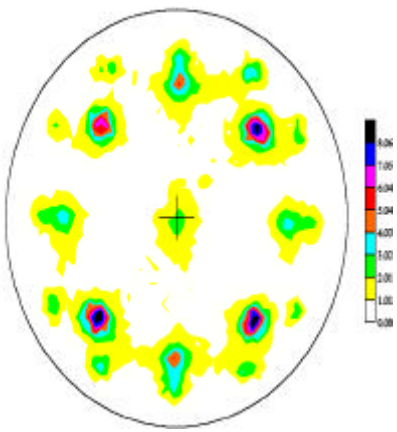
Nucleation of perovskite seed layers: STO

- *Texture degrades with decreasing S content of Ni surface*

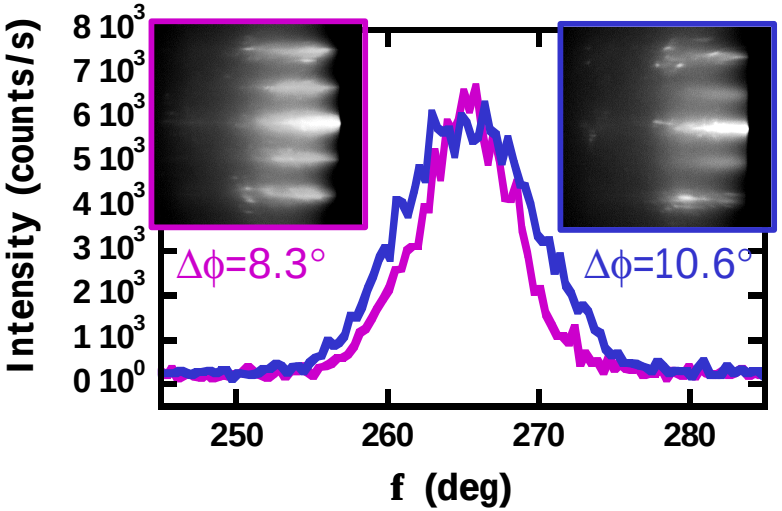


Effect of variations in S content

- Particular annealing processes (e.g.: high temperature, long dwell time, presence of O_2) can deplete the metal of S
- S concentration below 10 % [~43% of c(2×2)] results in multiple orientation
- Seed in-plane alignment correlates with c(2x2) coverage



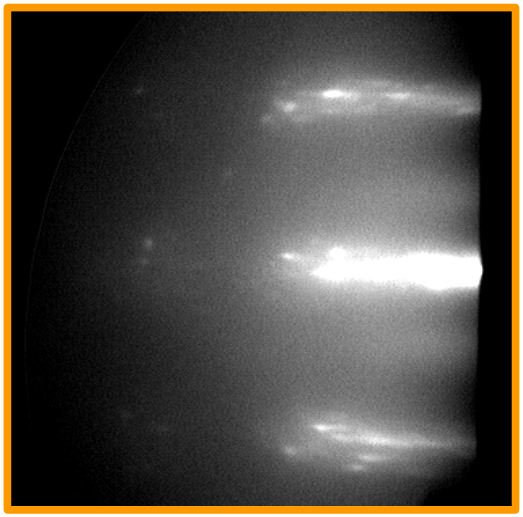
➢YSZ (111)



Need for an in situ process able to control and reproduce the S superstructure

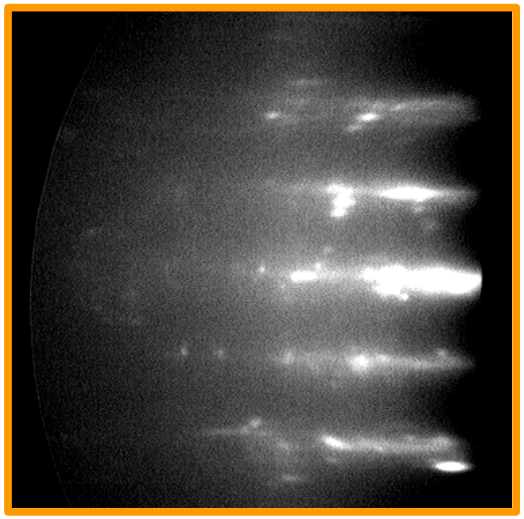
H_2S treatment provides the c(2´2)-S needed for buffer layer epitaxy

- A low H_2S partial pressure causes rapid dissociative chemisorption of S on the Ni surface
- A stable superstructure is grown at T=800°C and $P[H_2S] = 5.0\times10^{-7}$ Torr
- No evidence of surface degradation during treatments as long as 30 min



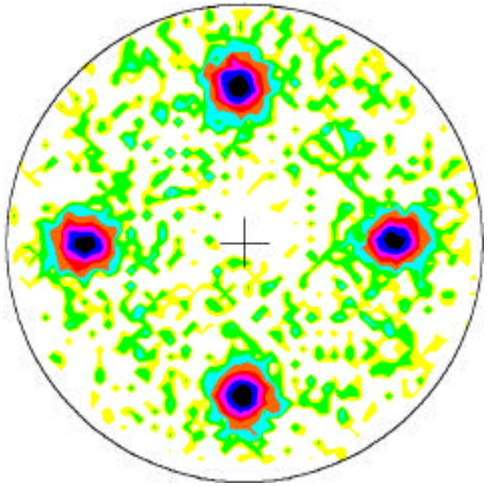
Ni surface with
38% of c(2x2)

H_2S



Ni surface after H_2S
C_S = 100% c(2x2)

YSZ

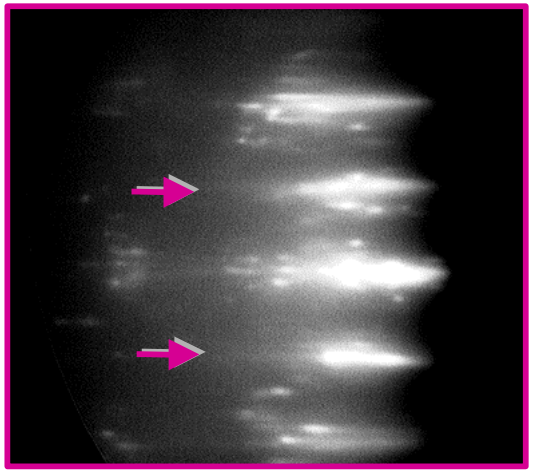


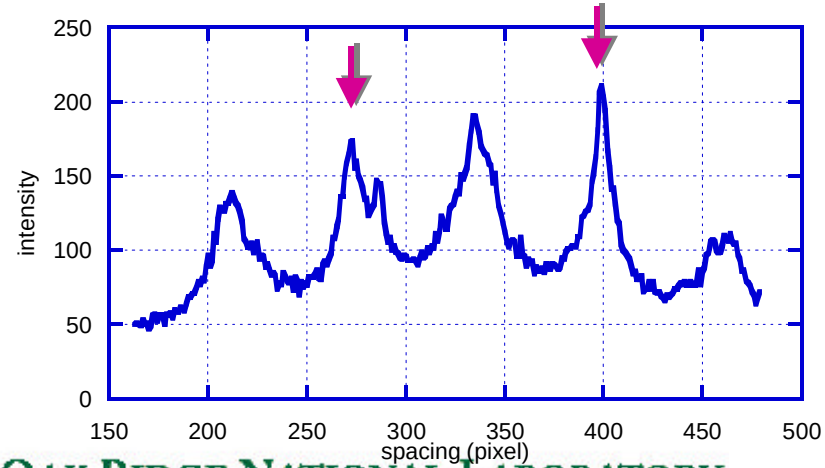
YSZ (111)
$\Delta\phi = 7.0°$ $\Delta\omega = 6.8°$
99.96% cube

RHEED observations on Ni alloys

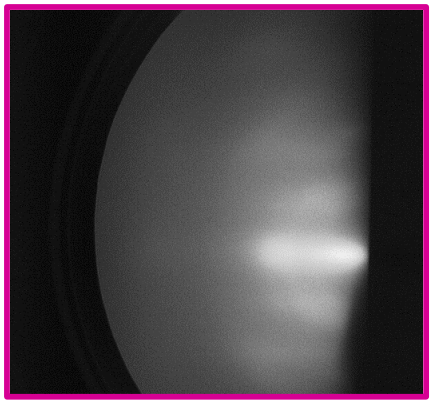
Ni- 3% W

500°C, 1h,
$P_b=5\times10^{-8}$ Torr

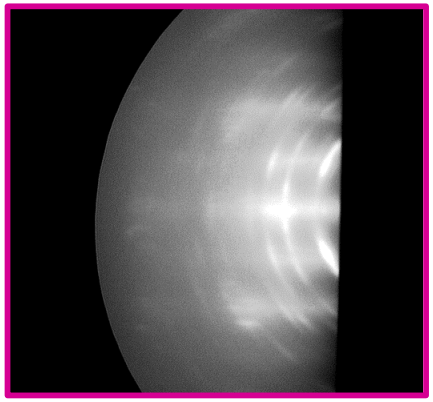




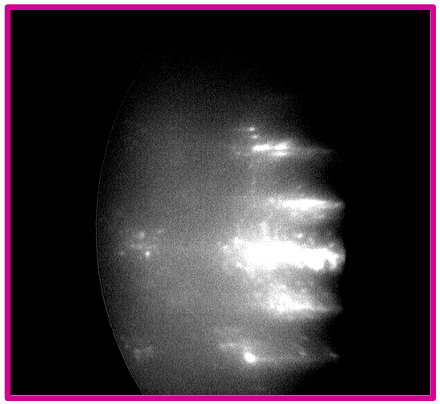
Ni-13% Cr



500°C, 2h,
$P_b=1\times10^{-8}$ Torr
$P[O_2]=8\times10^{-11}$ Torr



600°C
$P_b=3\times10^{-8}$ Torr
$P[O_2]=2\times10^{-10}$ Torr



after
700°C, 1.5 h
$P[4\%H_2/Ar]$ =
200 mTorr

Summary

- **Sulfur atoms diffuse and segregate to the free surface of Ni and Ni-alloys, forming a c(2´2) superstructure that promotes epitaxial growth of YSZ, CeO_2 and STO**

- **A stable S superstructure can be formed on S depleted Ni surfaces by in situ H_2S exposure. YSZ grows with full (200) texture on such H_2S-treated surface**

High quality oxide seed layers can be grown on Ni surfaces containing 23-27 AES % of S

A sulfur free surface can be formed by depositing Ni *in situ*

Ni-13% Cr surface can be cleaned by annealing in 200 mTorr Ar-4%H_2 at 700°C, revealing a c(2×2) superstructure

Outline:
Continuous Processing of Coated Conductors

- **FY 2001 Results:**

  Progress on buffer layer deposition
  - Background research on the effects of sulfur—Claudia Cantoni
  - **A sulfur template for buffer layers on lengths —Fred List**
  - **3M results for long length, high rate buffer deposition—Fred List**

  Conversion of YBCO(F) precursor in lengths—Dominic Lee

- **Summary of FY2001 Results/FY2002 Plans-**Dominic Lee

- **Research Integration-**Dominic Lee

A Sulfur Template for Buffer Layers on Lengths

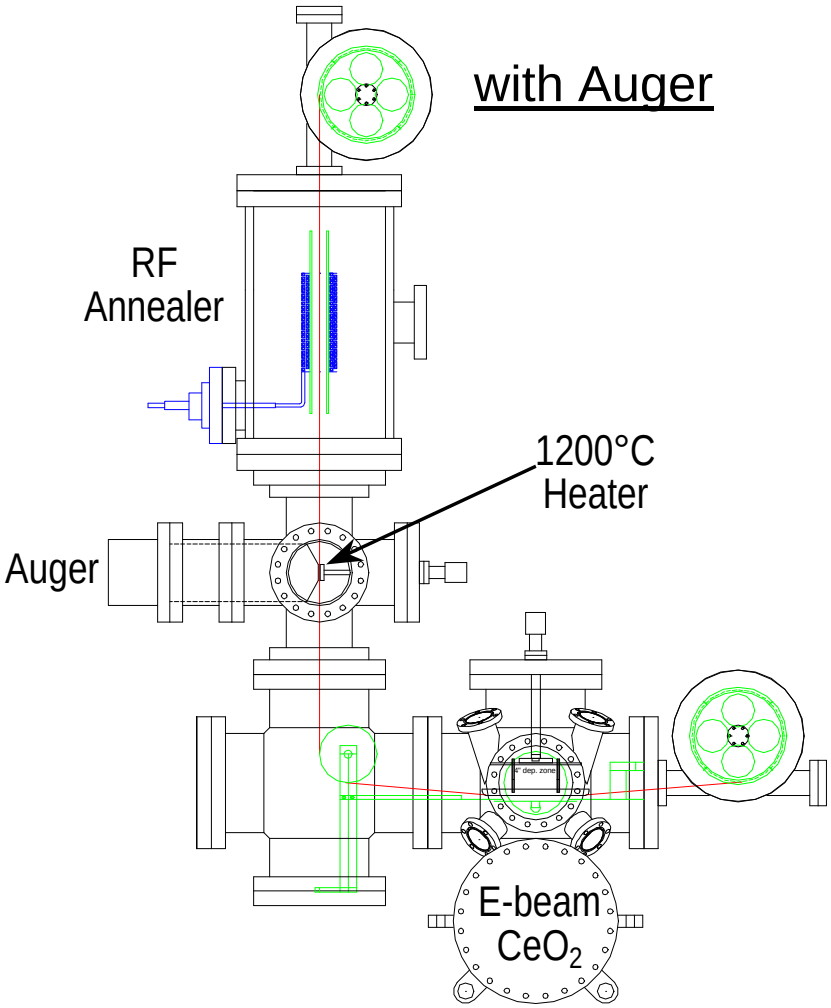
Motivation

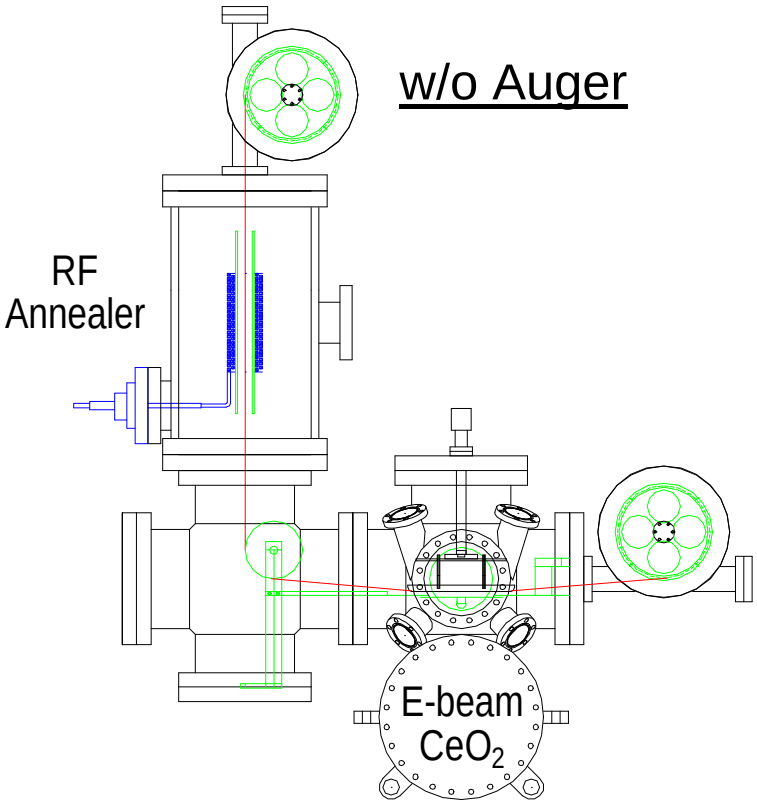
- High quality CeO_2 on lengths has been difficult to reproduce reliably.
- Process conditions for obtaining good CeO_2 vary with the nickel source and nickel annealing conditions.

Goals

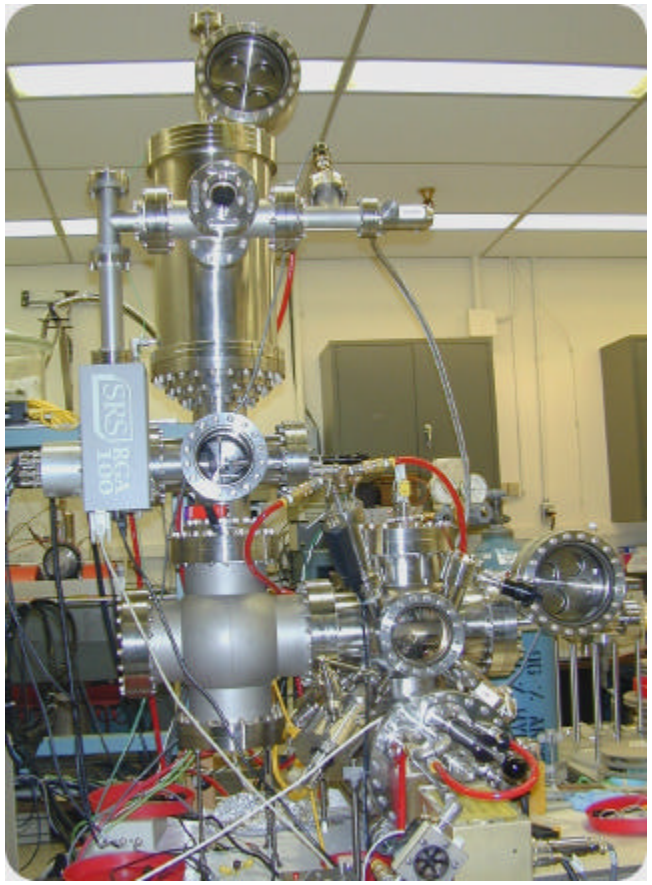
- To understand the role sulfur can play for epitaxial growth of CeO_2 on lengths of nickel.
- To use this understanding to develop a more robust process for buffer layer growth on lengths.

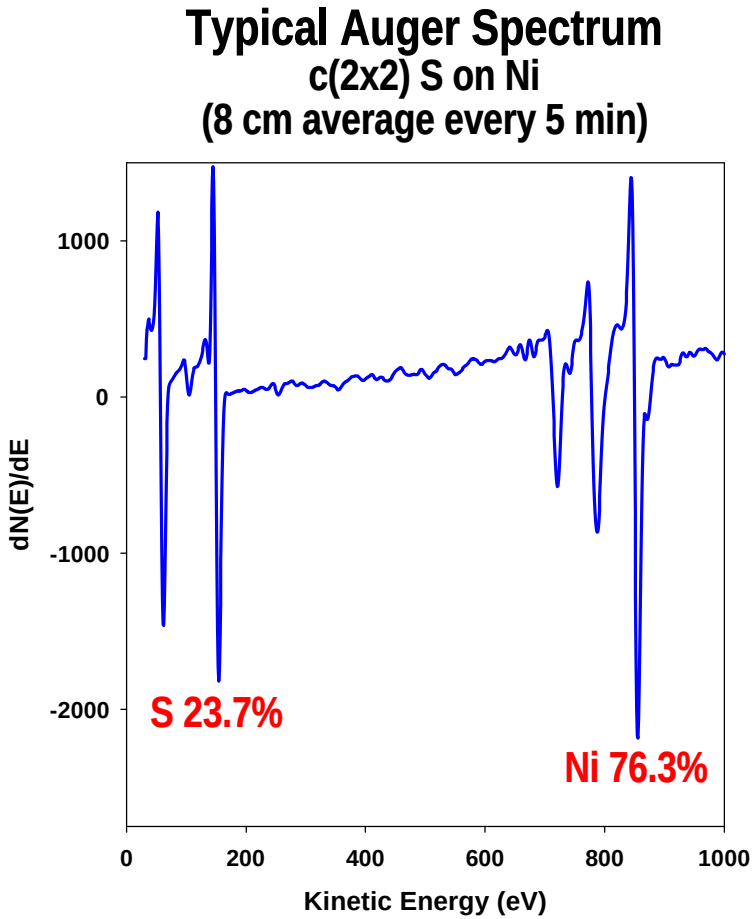
To measure surface sulfur, an Auger analyzer has been added to the reel-to-reel anneal/seed chamber.





In situ Auger characterization of moving tape facilitates epitaxial buffer layer development.



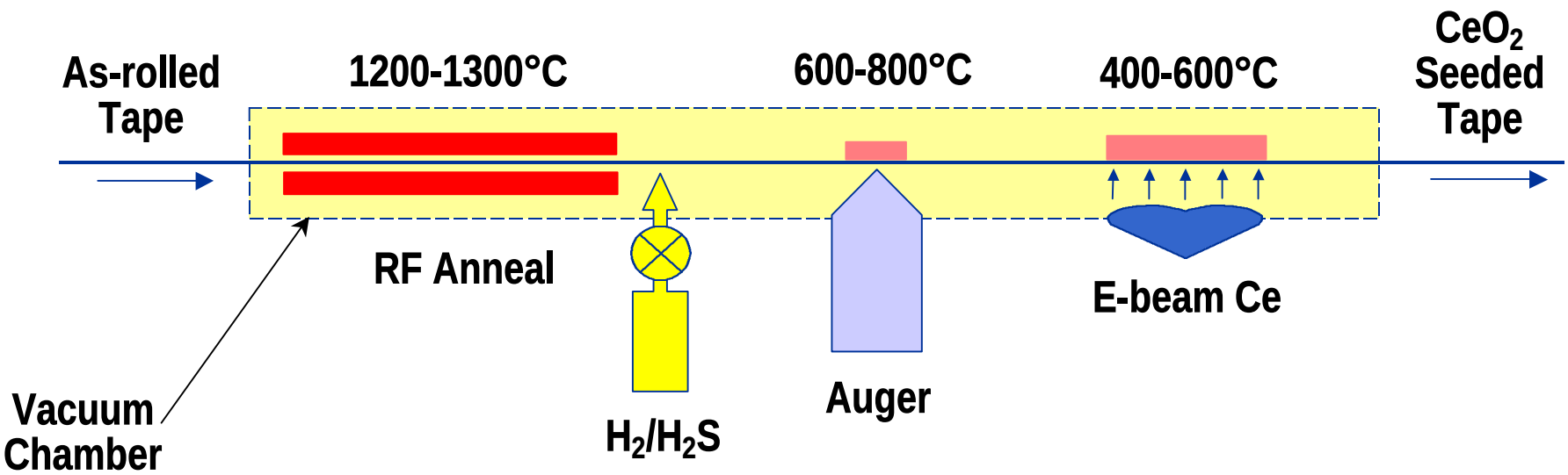


Two approaches to control surface sulfur have been explored for lengths of tape.

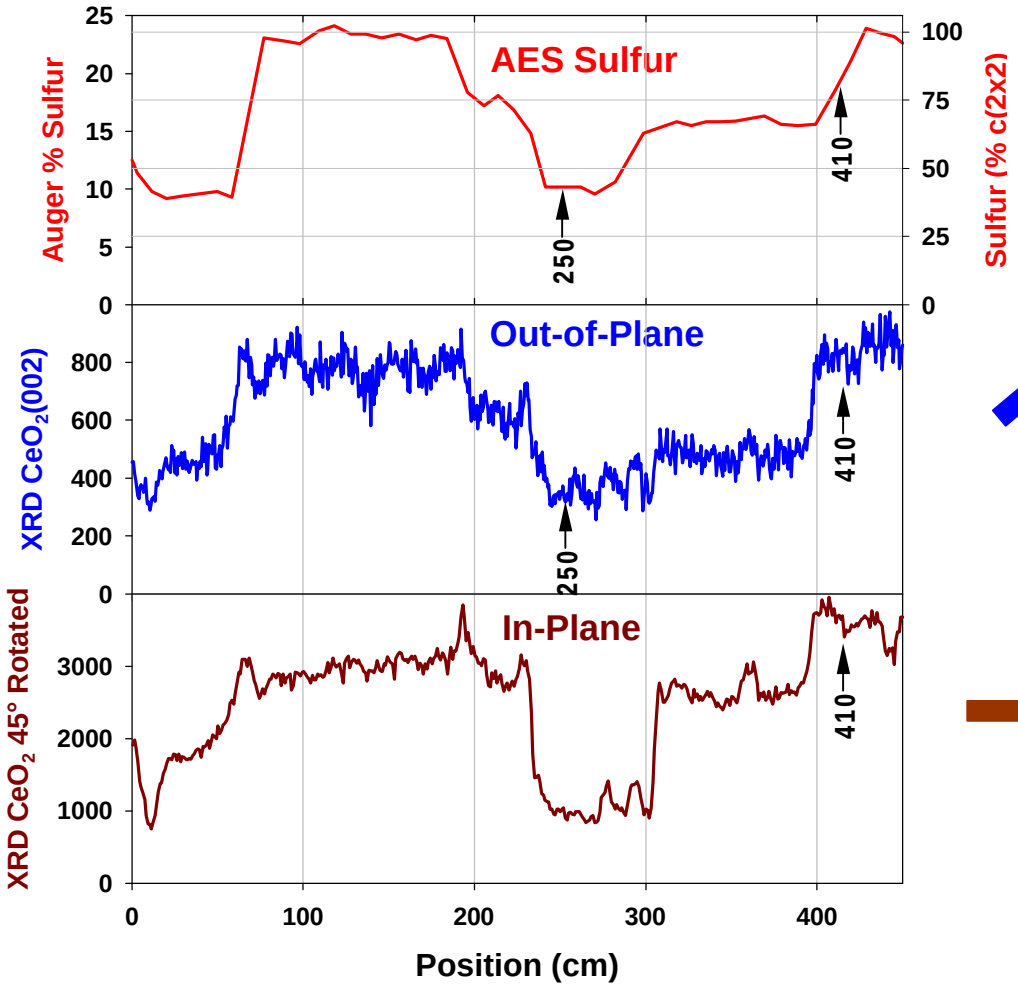
- **Segregation of sulfur to the tape surface from the bulk**
  - Diffusion kinetics depend on temperature, time, and bulk sulfur concentration.
  - Time to segregate sulfur is typically long (~>1 hr) and seems to vary with the bulk sulfur concentration. ⇒ **process irreproducibility** ☹
  - The fully developed c(2x2) sulfur template is difficult to develop for many tapes, perhaps because of too little bulk sulfur. ☹

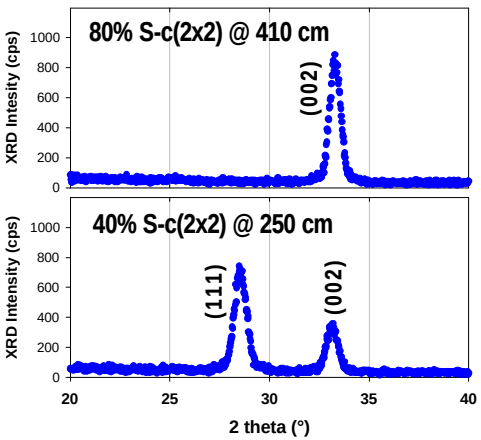
- **Adsorption of sulfur on the tape surface from the gas**
  - H_2S and H_2/H_2S mixtures provide the gaseous sulfur source.
  - The development of nickel texture is decoupled from the development of the sulfur template. ⇒ **greater process flexibility** ☺

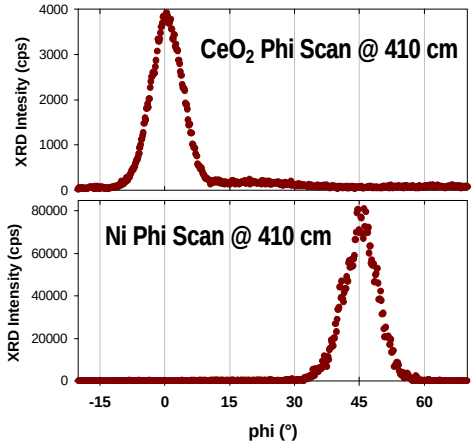
Annealing, sulfur adsorption, and CeO_2 deposition all occur in one-pass for a tape speed of 1 m/hr.



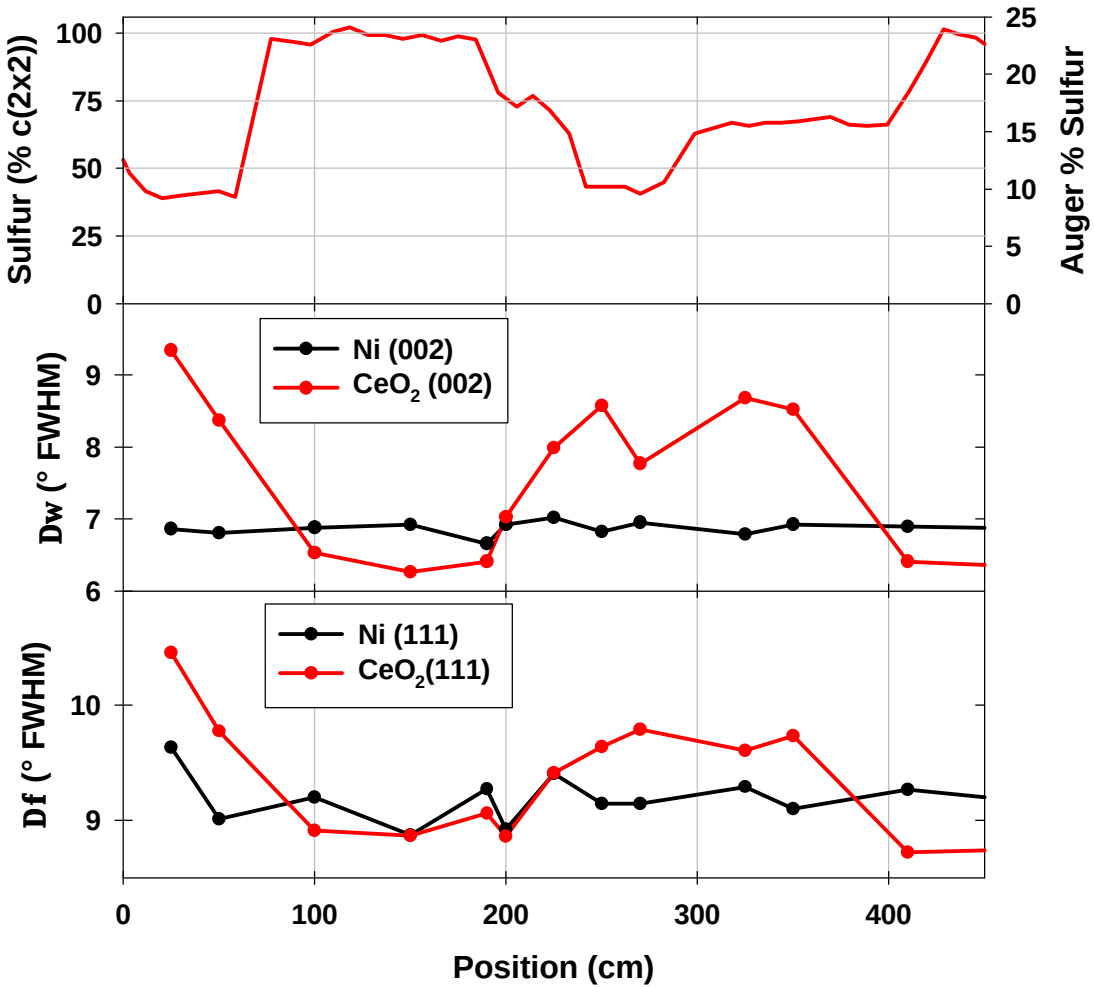
The x-ray intensity of CeO_2 on Ni increases as the surface concentration of S on Ni increases.







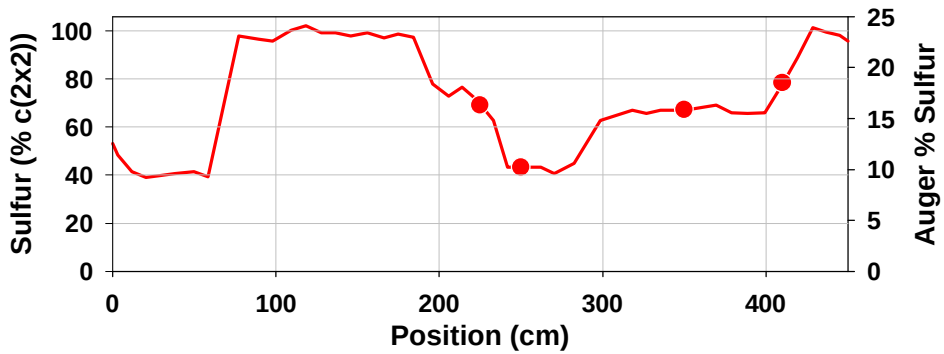
The epitaxial quality of CeO_2 on Ni increases as the surface concentration of S on Ni increases.

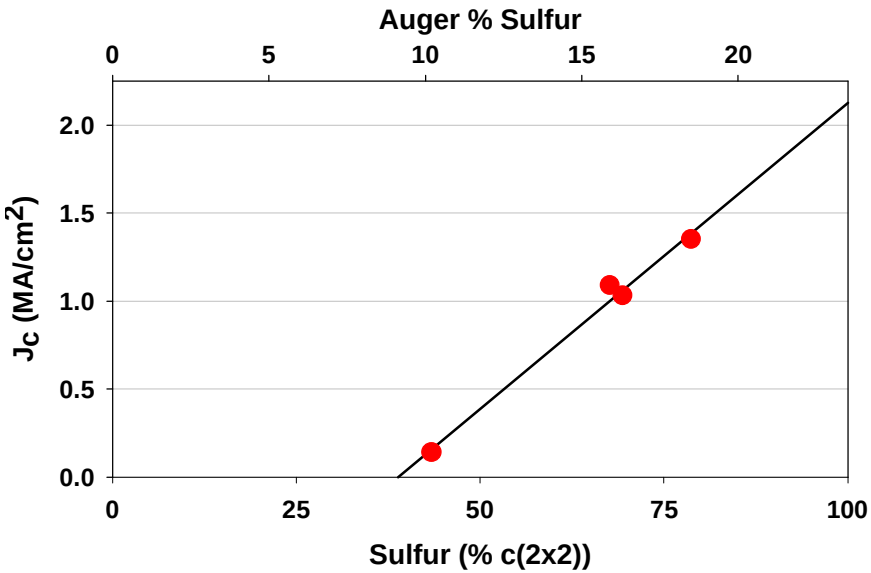


CeO_2 on ~80% c(2x2) sulfur:

Dw FWHM:	**6.4°(CeO_2)**
	6.9° (Ni)
Df FWHM:	**8.8° (CeO_2)**
	9.2° (Ni)

The J_c for PLD YBCO on RABiTS increases as the surface concentration of S increases.

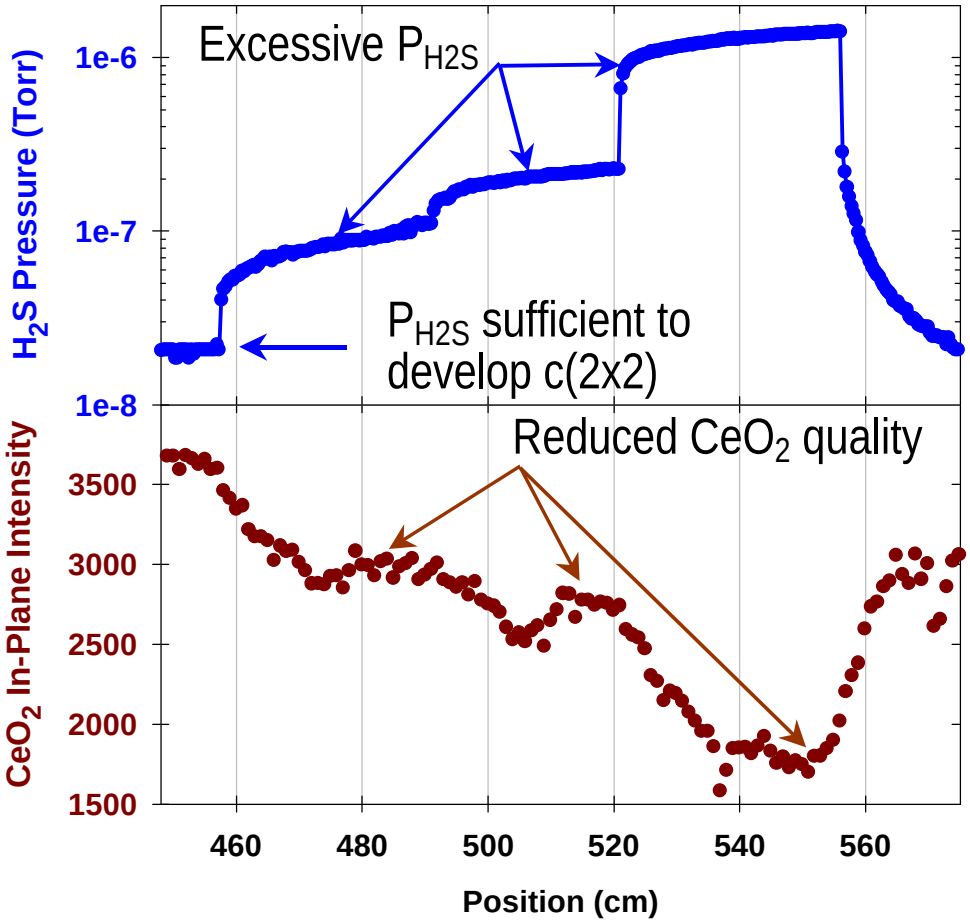




- Four, 3 cm lengths of CeO_2/Ni:
  - Sputtered with YSZ & CeO_2 cap
  - PLD ~2000Å YBCO

- <u>As surface sulfur increases</u>:
  - J_c increases
  - CeO_2 seed quality increases
    - Dw ß
    - Df ß

For simultaneous processing, excessive H_2S pressure is deleterious to CeO_2 quality.



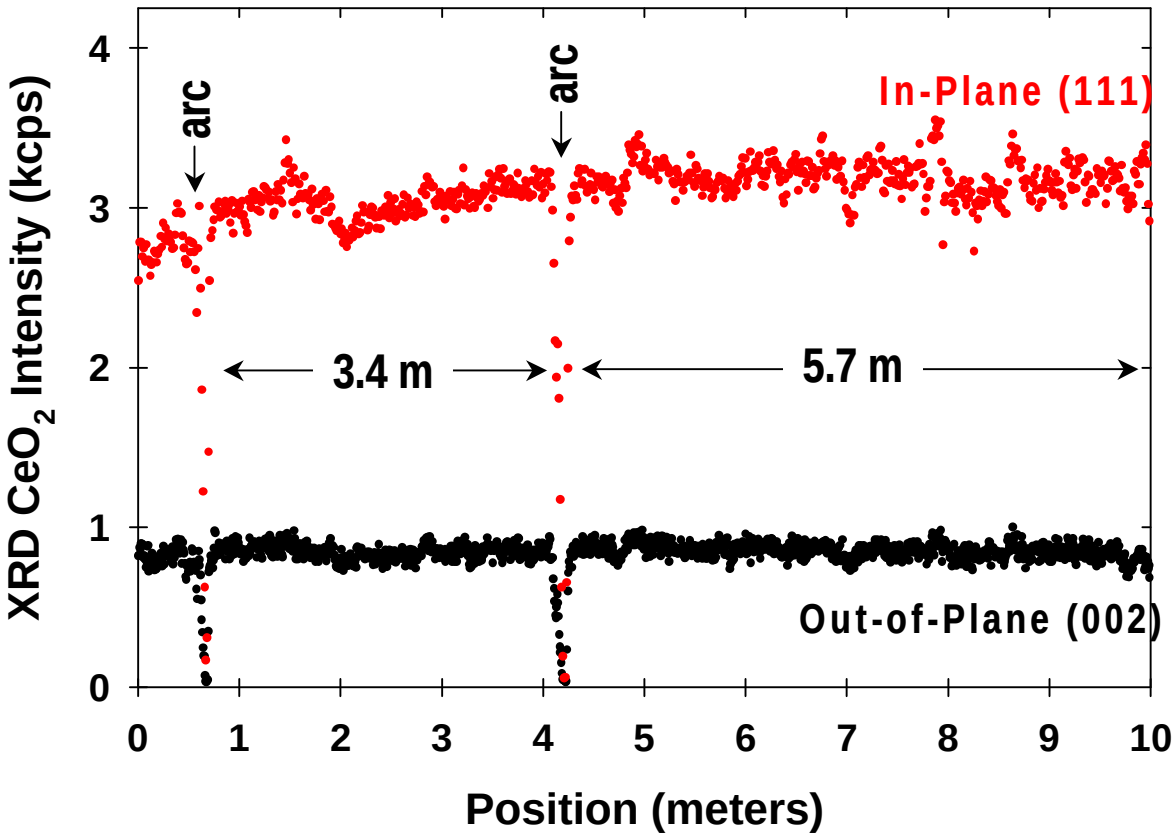
Sulfur on Ni : 100% c(2x2) throughout

P_{H2S} : ~1x10^{-8} to 1.5x10^{-6} Torr

- **<u>Two-pass process is now being evaluated</u>**

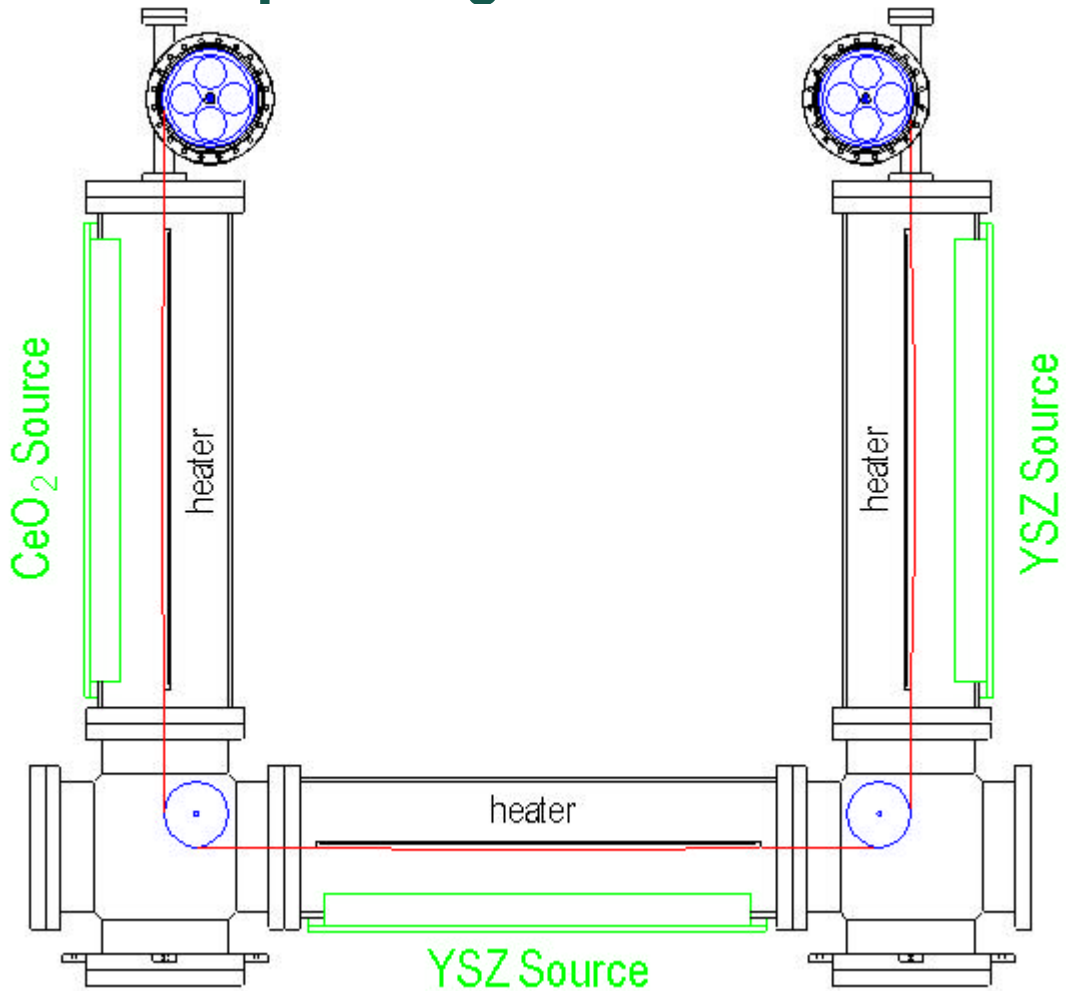
- **1st pass – Ni anneal & H_2S**
- **2nd pass – CeO_2 seed**
- **greater process flexibility**

Long lengths of tape with high quality CeO_2 have been prepared in a single-pass using H_2S.



- Tape Speed : 1 m/hr
- ~10 meter length total
- c(2x2) sulfur
- >99% (002) Out-of-Plane
- >99% 45° rotated In-Plane
- Tape awaits further processing with YSZ,CeO2,YBCO

A new, high rate, buffer deposition system for completing RABiTS is being assembled.



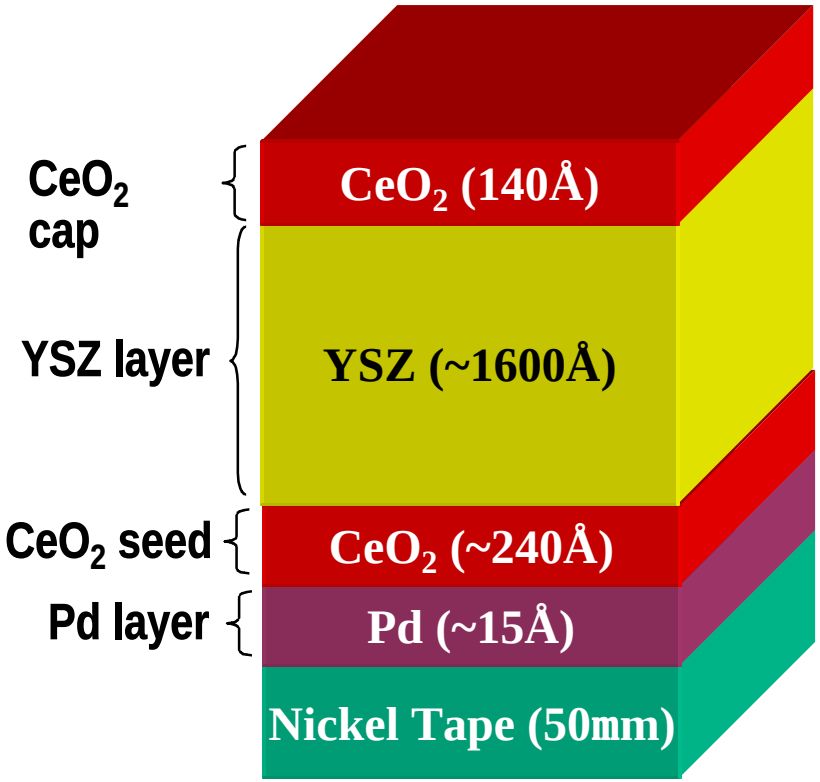
Key Hardware

- Three 3.5 x 24” linear sources
- Three 1.5 x 28” 2.8kW heaters

Expected Throughput

1.0 m/hr (to match throughput of nneal/seed deposition system)

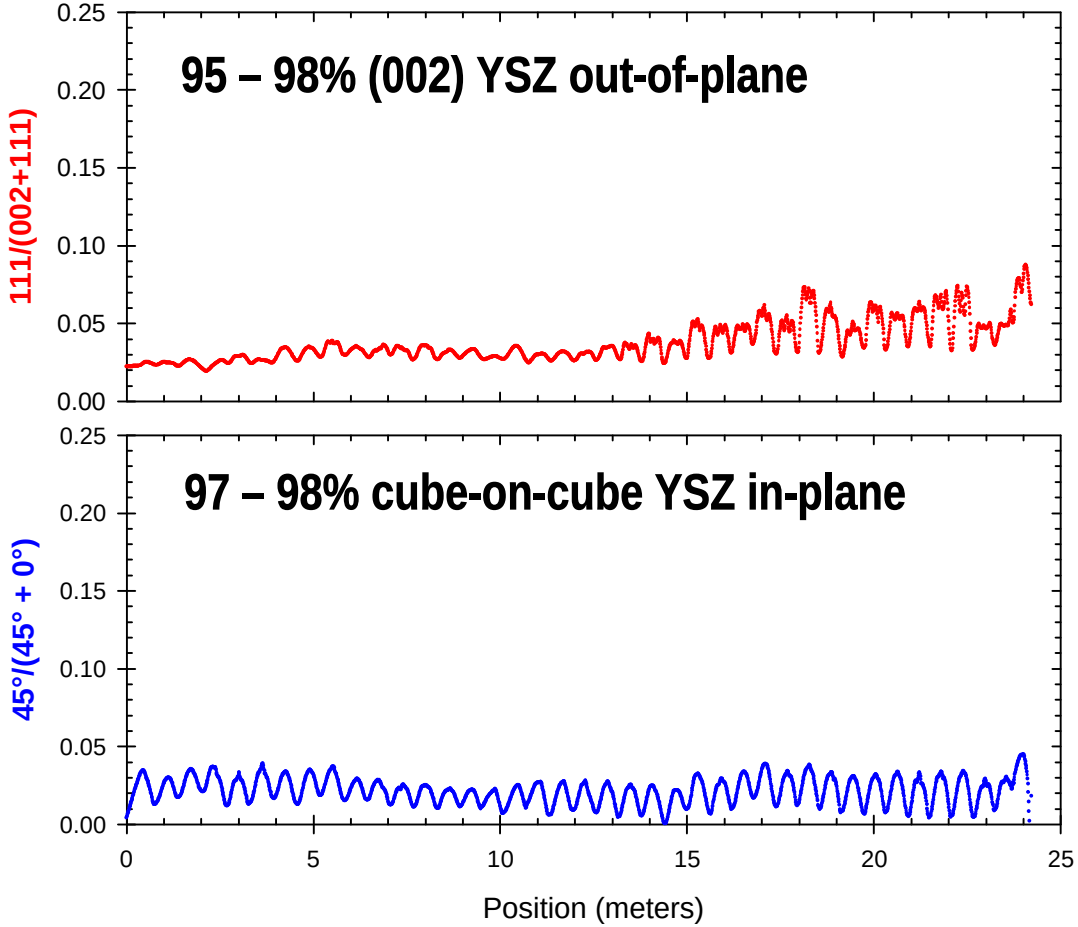
Lengths of 3M RABiTS are routinely Prepared in a single-pass at 18 m/hr.



Length:	~20 meters typical
	~500 meters max.
Web Speed:	~30 cm/min
Web Width:	1 cm typical
	3.1 cm max.
Web Heating:	Resistive
CeO_2:	Reactively Sputtered
YSZ:	Reactively Sputtered
Pd:	DC Sputtered

3M Innovation

The x-ray crystalline quality of 3M RABiTS is good over 24 meter lengths.



- Single – Pass Process
- Web Speed - 30 cm/min
- XRD :

  Dw ~10° FWHM CeO_2 & YSZ

  Df ~12° FWHM CeO_2 & YSZ

3M Innovation

A Sulfur Template for Buffer Layers on Lengths: Summary

- The c(2x2) sulfur template improves buffer layer quality on lengths.
- J_c of PLD YBCO increases with increasing sulfur coverage.
- The sulfur template develops rapidly for a wide range of H_2S exposure.
- Lengths of good quality CeO_2 seed have been grown at 1 m/hr on tape with the sulfur template.

Outline:
Continuous Processing of Coated Conductors

- **FY 2001 Results:**

  Progress on buffer layer deposition
  - Background research on the effects of sulfur—Claudia Cantoni
  - A sulfur template for buffer layers on lengths—Fred List
  - 3M results for long length, high rate buffer deposition—Fred List

  Conversion of YBCO(F) precursor in lengths—Dominic Lee

- **Summary of FY2001 Results/FY2002 Plans-Dominic Lee**

- **Research Integration-Dominic Lee**

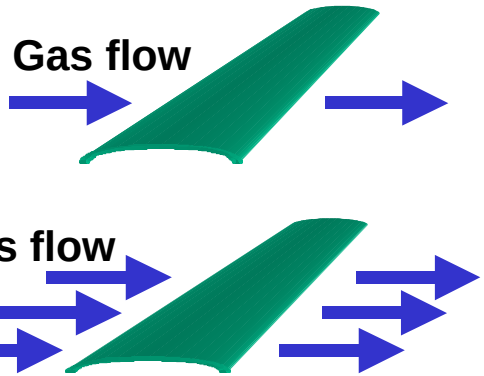
Continuous Ex-Situ Conversion of YBCO

- **In FY2000, we reported that:**
  - **Efficiency of HF removal is a major concern in *"large-scale"* YBCO conversion -**
    - **Higher HF concentration downstream of gas flow (I.e. trailing end of sample).**
    - **HF concentration decreases with increasing gas flow rate.**
  - **Various issues combine to limit both the overall J_c *value* and *uniformity* of ex-situ long-length YBCO on RABiTS.**

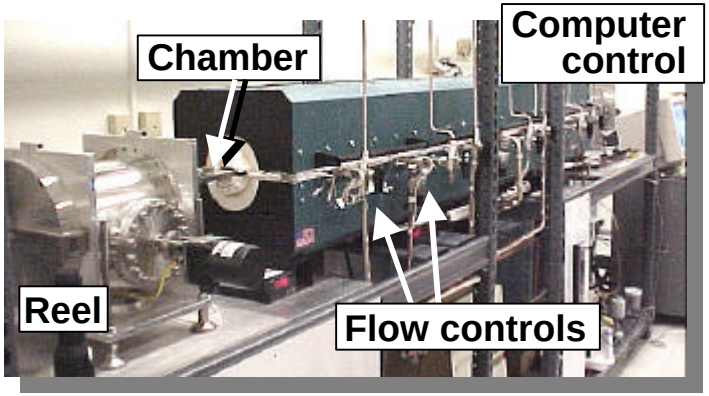
- **FY2001 plan:**
  - **To pinpoint the causes and address the J_c limiting issues.**
  - **YBCO conversion -**
    - **Improve sample homogeneity.**
    - **Optimization of dominant processing parameters.**
    - **J_c of 1 MA/cm^2.**

A Transverse-Flow Extended-Zone Approach was Chosen to Maximize the Conversion Area

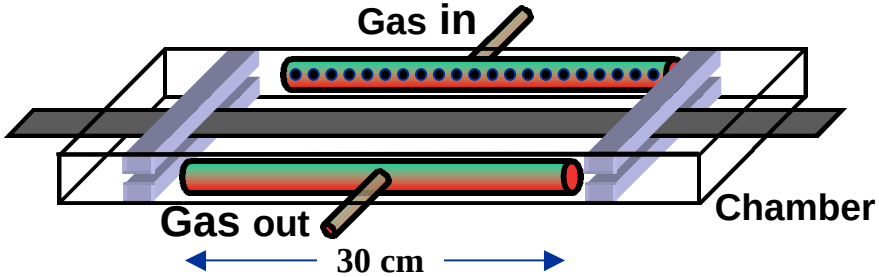
- **Approach:**
  - **Transverse-flow -**
    - **Instead of length, sample width is now the effective dimension.**
  - **Extended-zone -**
    - **By distributing the gas flow along sample length, much larger area can be converted at same time.**



A 7 Flow-Module 22-Zone Reel-to-Reel Furnace was Built to Study Long-Length YBCO Conversion



- **Furnace:** 2 meter long, Inconel 601.
  Gas: Seven flow modules, independent gas controls.
- First attempts on long-length conversion yielded samples with very *non-uniform* J_c's

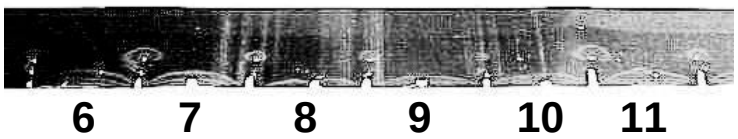


What Limits Us from Getting *High* and *Uniform* J_c's in Long-Length Ex-Situ YBCO on RABiTS?

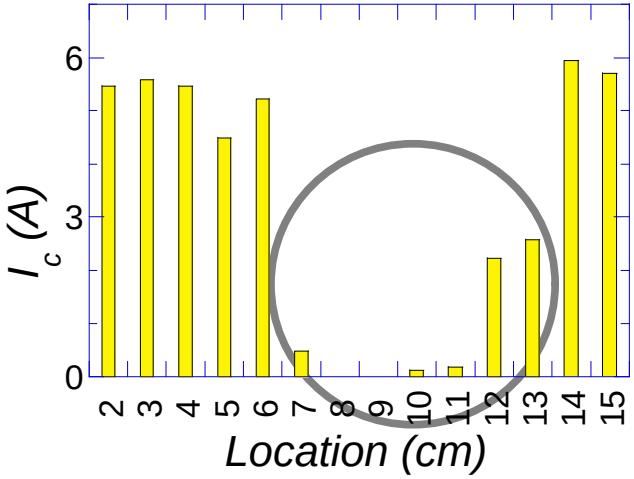
- **To achieve high J_c's in long lengths, *every* processing step has to be successful on the *same* sample.**
- **Some J_c limiting issues are:**
  - **1. Uniformity and reproducibility of RABiTS -**
    - ***overall value, uniformity.***
  - **2. Non-stoichiometric precursor -**
    - ***end-to-end value, uniformity.***
  - **3. Sample–gas orientation -**
    - ***uniformity.***
  - **4. Dimensional flaws -**
    - ***uniformity.***
- **We have either resolved or implementing solutions to these issues.**
- **1. RABiTS texture -**
  - **Solution: Addition of H_2S source to ensure complete coverage of superstructure.**
  - **Long-length results could not take advantage of the improved H_2S solution, but we expect to have these RABiTS for YBCO conversion soon.**

2. Linear Features are Frequently Seen in Precursor and Converted YBCO

- **Linear features:**
  - **These lines run across the width of the sample…**



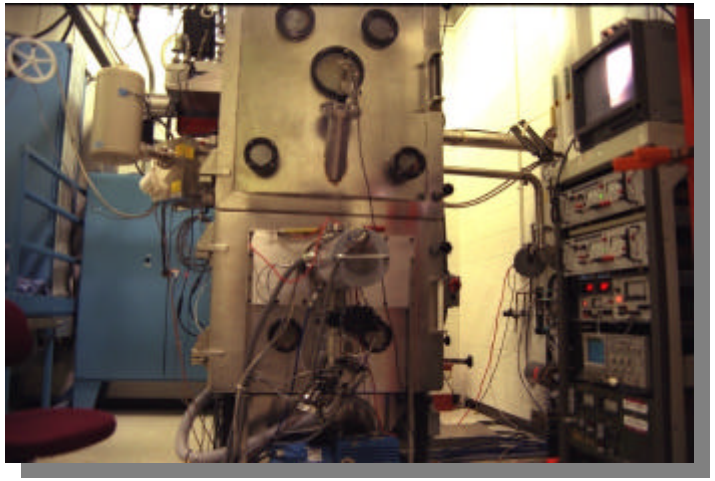
… and resulted in zero or low sectional J_c's.



- **These features are the result of precursor non-stoichiometry from our *bench-top* system:**
  - **Fluctuation during deposition.**
  - **Shut-down due to discharge.**

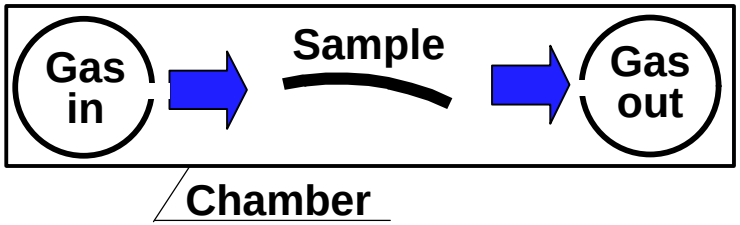
- **Solutions:**
  - **Replacement of e-beam with effusion cells.**
  - **Precursor deposition at 3M.**

3M Innovation

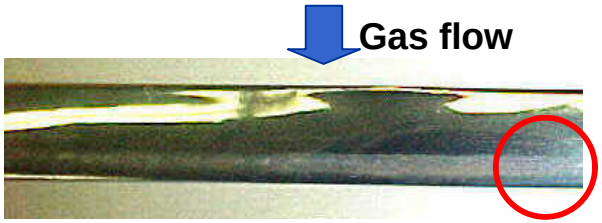


3. Edge to Edge YBCO Non-Uniformity is Worsened by Sample Curvature and/or Tilting

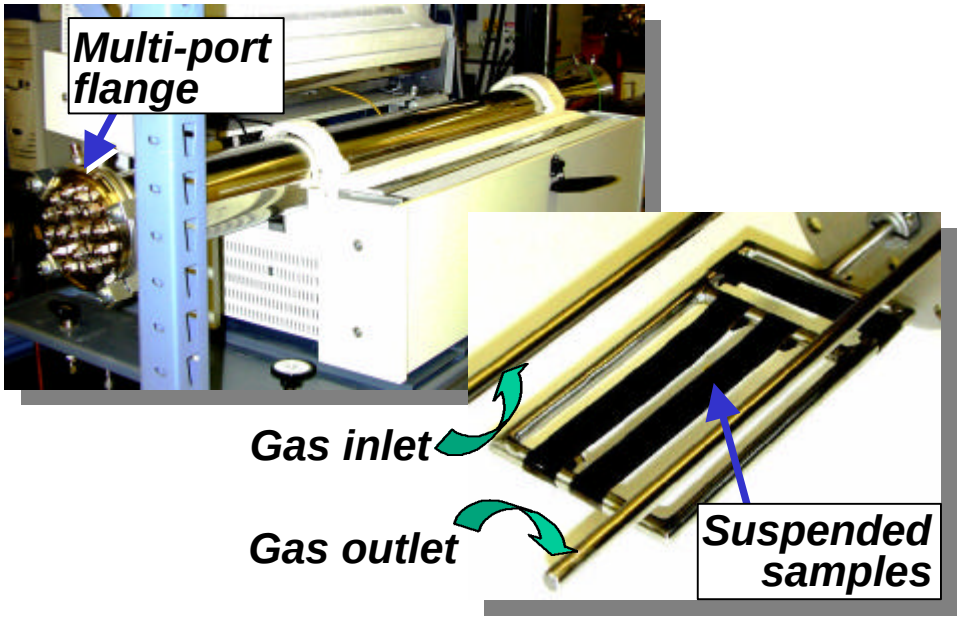
- Cross-sectional view of reaction chamber



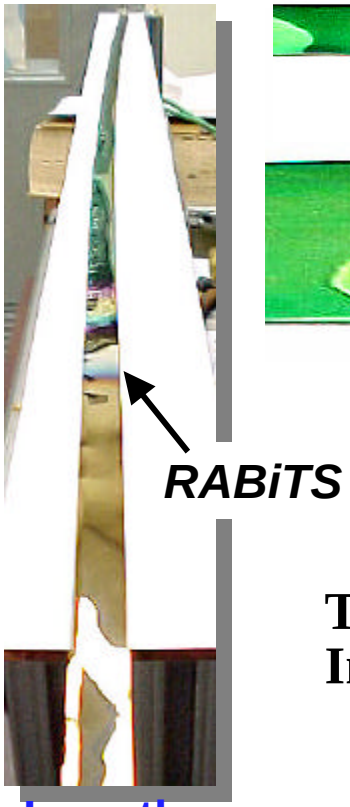
- YBCO show a region of incomplete conversion at the downstream edge



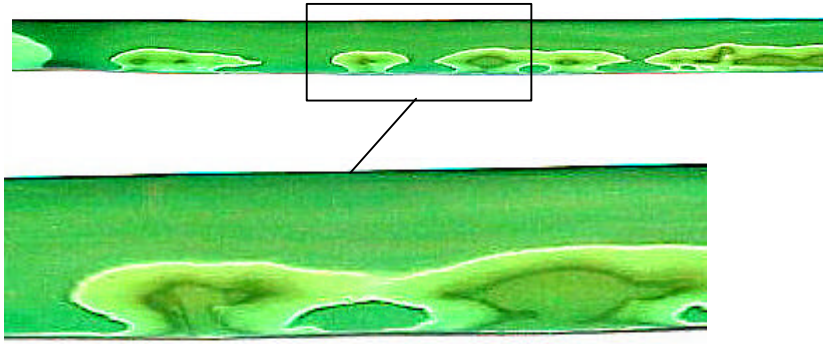
- **Solution:**
  - We are in the process of designing a chamber with *alternating* gas injection.
  - We are also investigating the effects of various nozzle designs and angle-of-attack (to be incorporated into the alternating flow chamber).



4. Kinks were Common Flaws Found in RABiTS

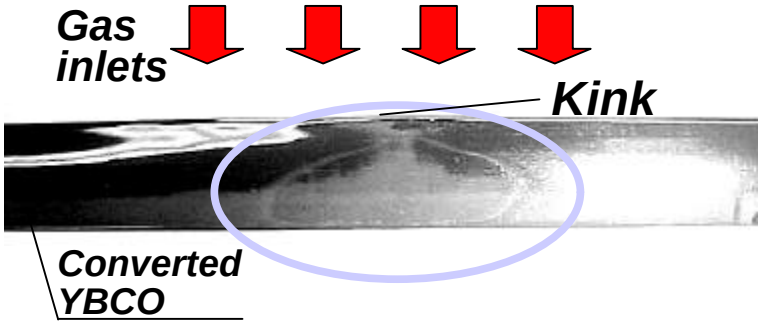


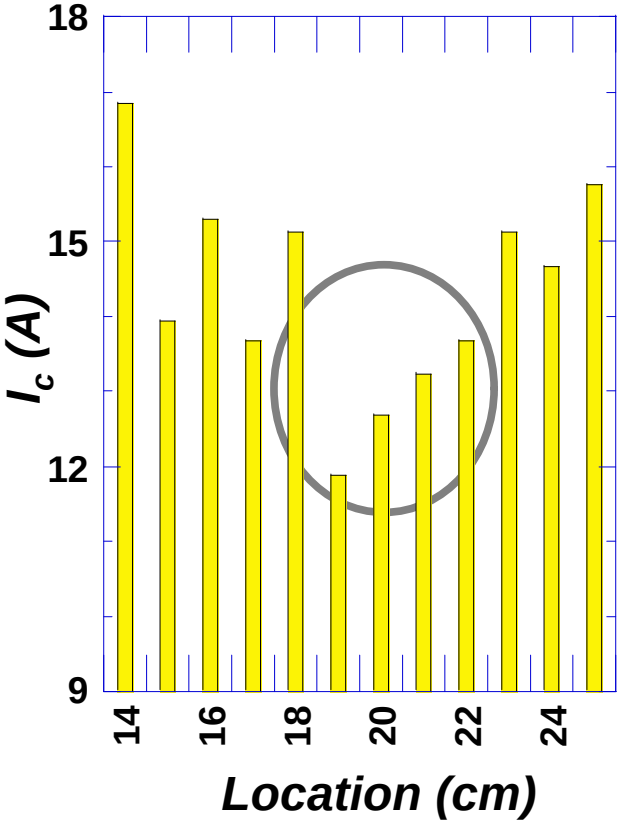
Length view



Close-up view

These dimensional flaws resulted In non-uniform conversion…

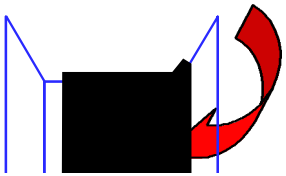




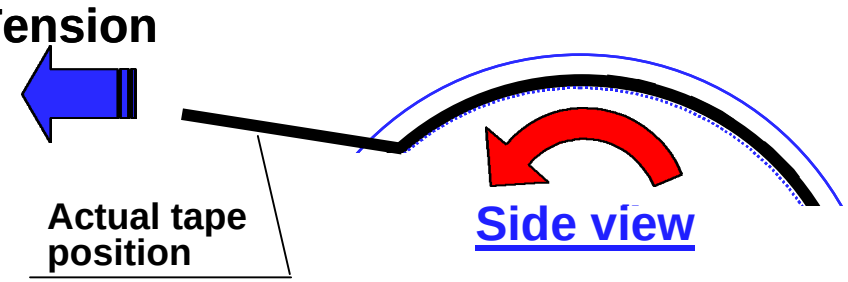
… and low local J_c's.

These Flaws have been Eliminated by Using Wider Reels & Modifying the Tape Transfer Procedure

- **Origin of kinks:**
  - Tapes that are not straight and uniform in width would be in contact with the side-wall during *wind-up*…



Edge view



… and kinks will result during *pay-out* of the tape.

- **Alloy RABiTS will not encounter this issue.**

- **Straight and dimensionally uniform Ni RABiTS can now be obtained routinely.**

Close-up view

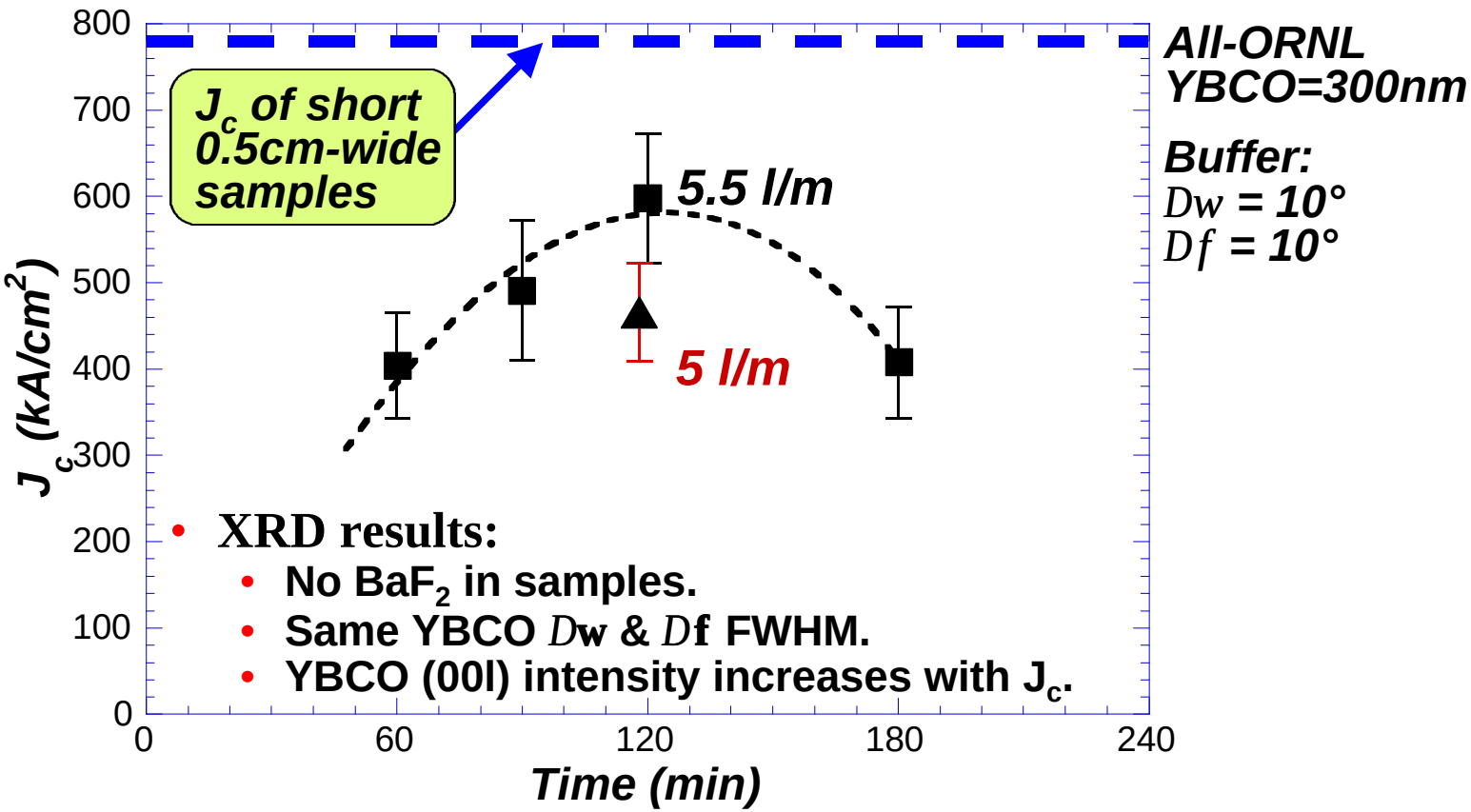
1 meter-length view

- ***Elimination of kinks resulted in immediate improvement in J_c uniformity →***

  S.D. dropped from ~24% to ~10%.

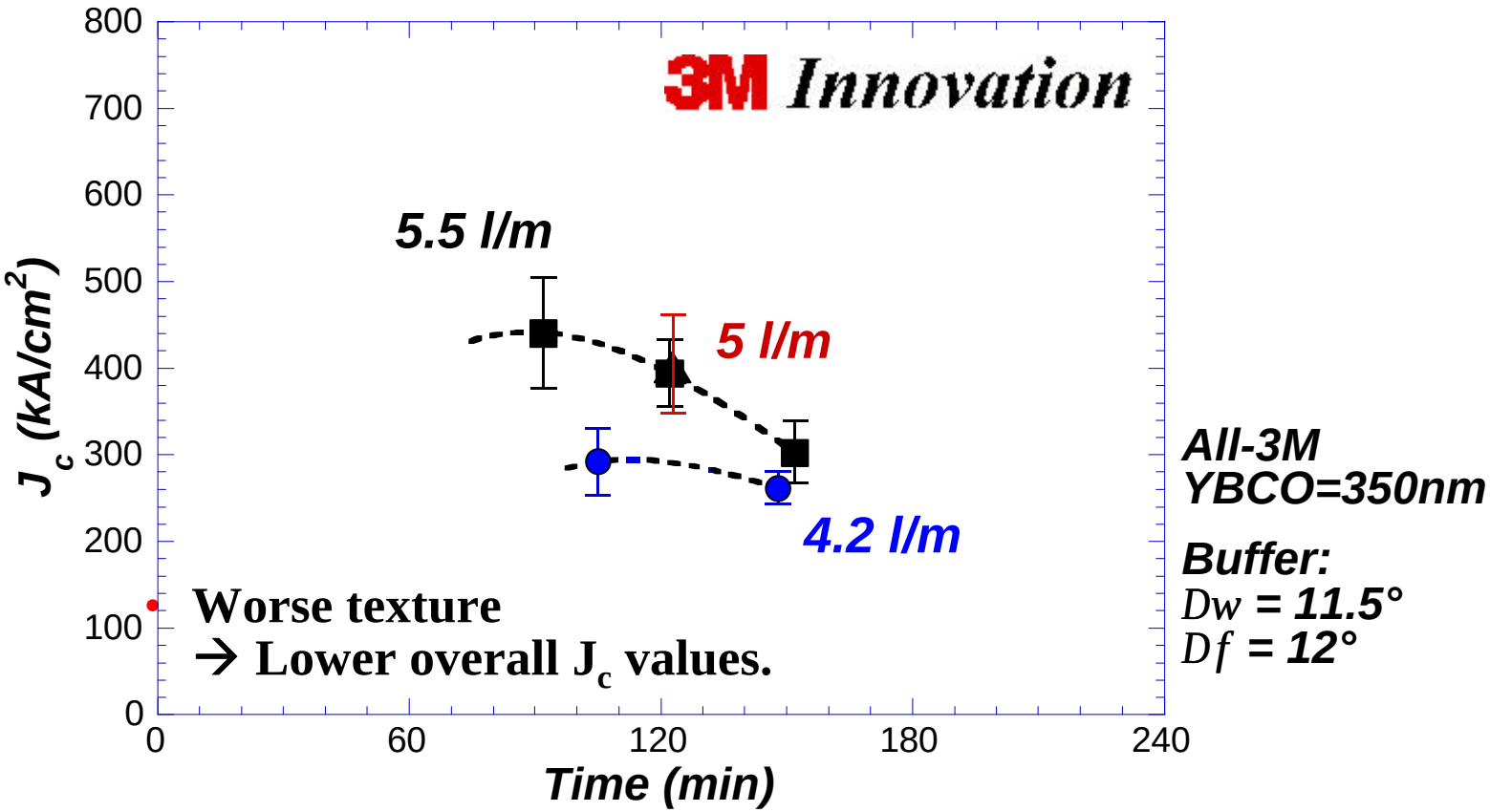
We Proceeded to Study the Flow Rate Dependency of J_c in Long-Length Conversion

- **"Optimum" conversion time of long samples is not the same as that of short samples (~ 7cm) processed in *both* single- and 7-module furnaces.**
- **0.6 to 1 meter-long samples were cut from a *single length* of RABiTS to minimize the effects of substrate texture variation and cross-talk between modules.**



Flow Dependency of J_c has also been Seen in Long-Length All-3M Tapes Converted at ORNL

- **0.6 to 1 meter-long samples were cut from a *single length* of All-3M Tape.**



Flow Dependency of J_c has also been Seen in Long-Length All-3M Tapes Converted at ORNL

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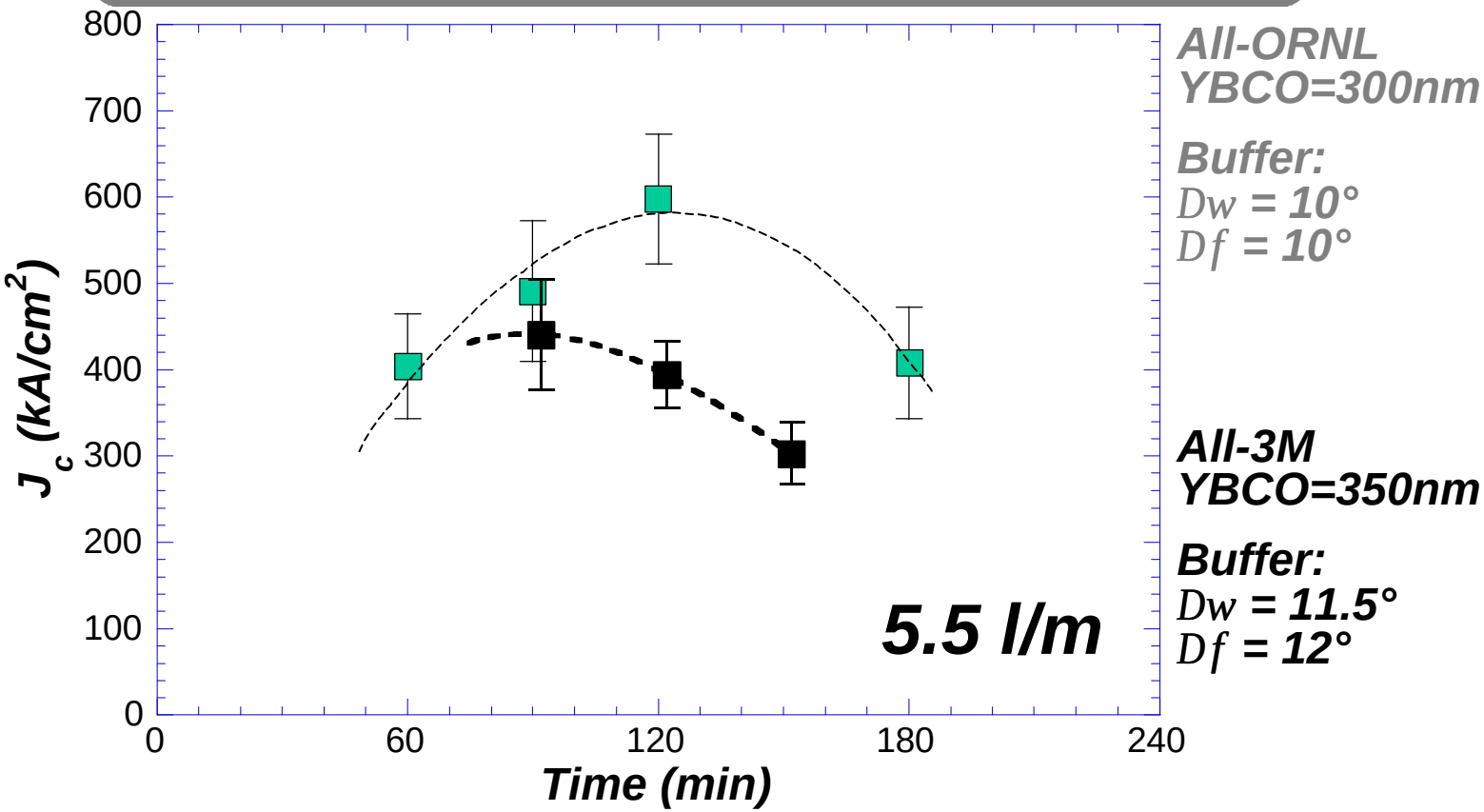
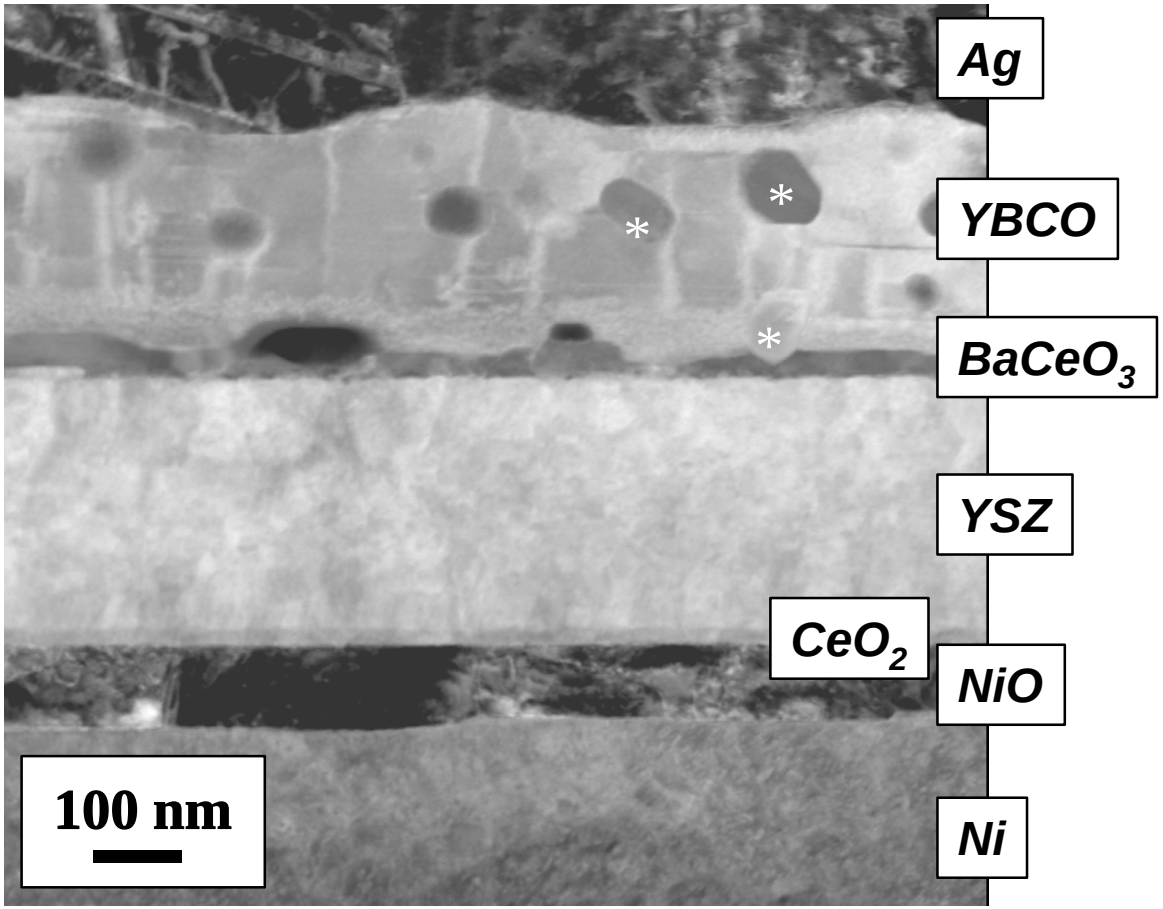
"Performance-based" optimum conversion time appears to be different between precursors.

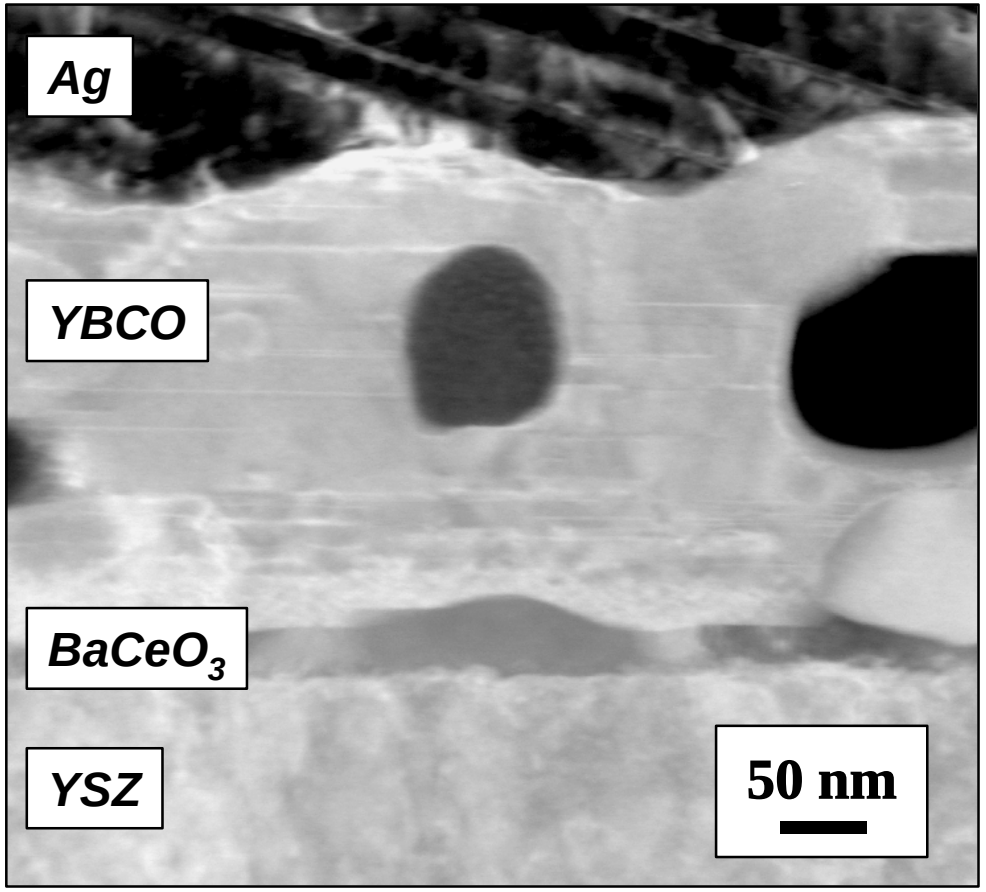


TEM Result Shows that CeO_2 Cap-Layer had Reacted with Ba in Precursor to Form $BaCeO_3$



K.L. More (ORNL)

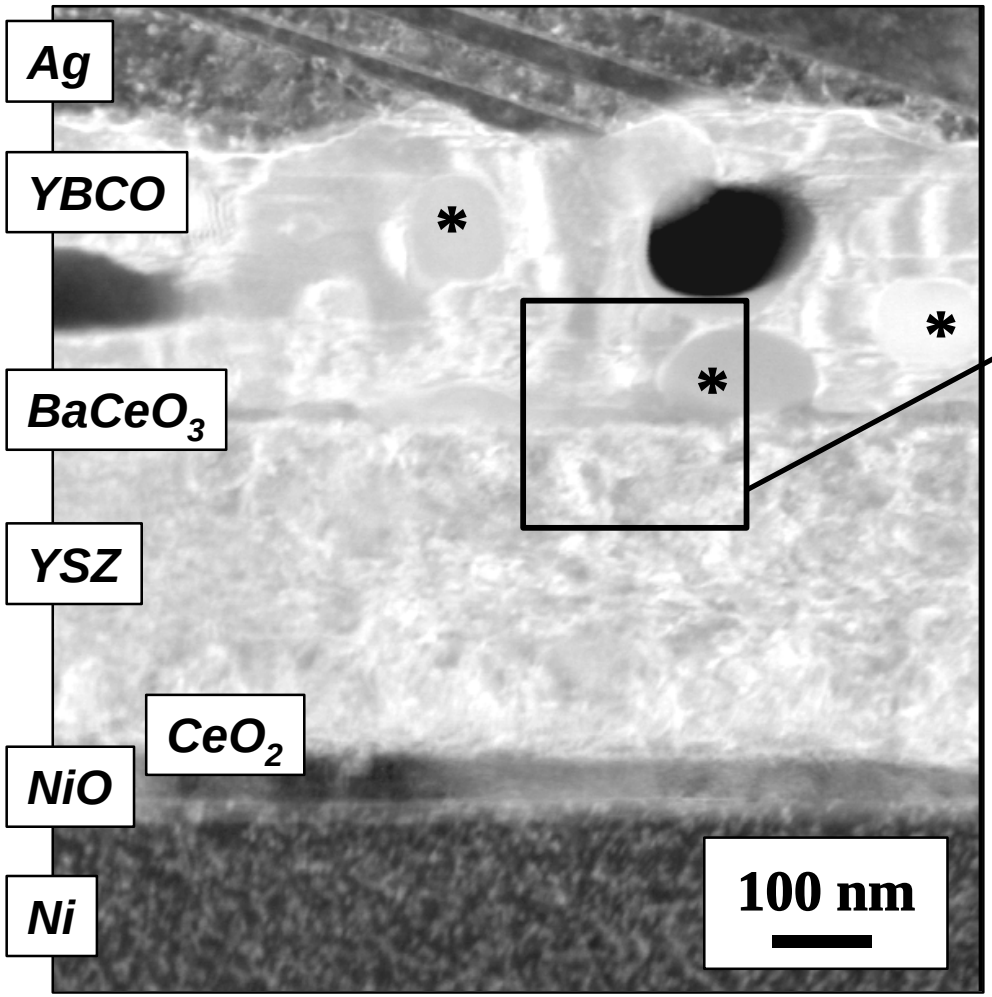
Significant Number of Second-Phase Particles as Large as 100nm can be Seen Throughout the YBCO Film

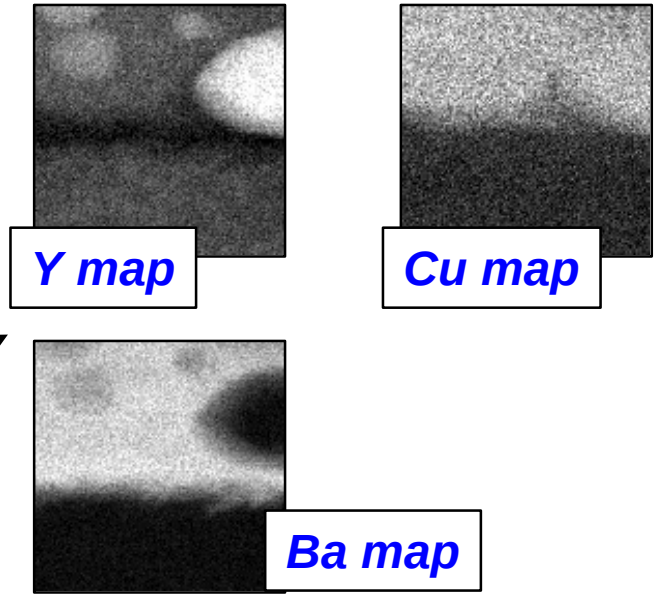


K.L. More (ORNL)

- **Nicely c-axis aligned YBCO can also be seen.**

These Second-Phase Particles are Y- & Cu-Rich but Ba-Deficient



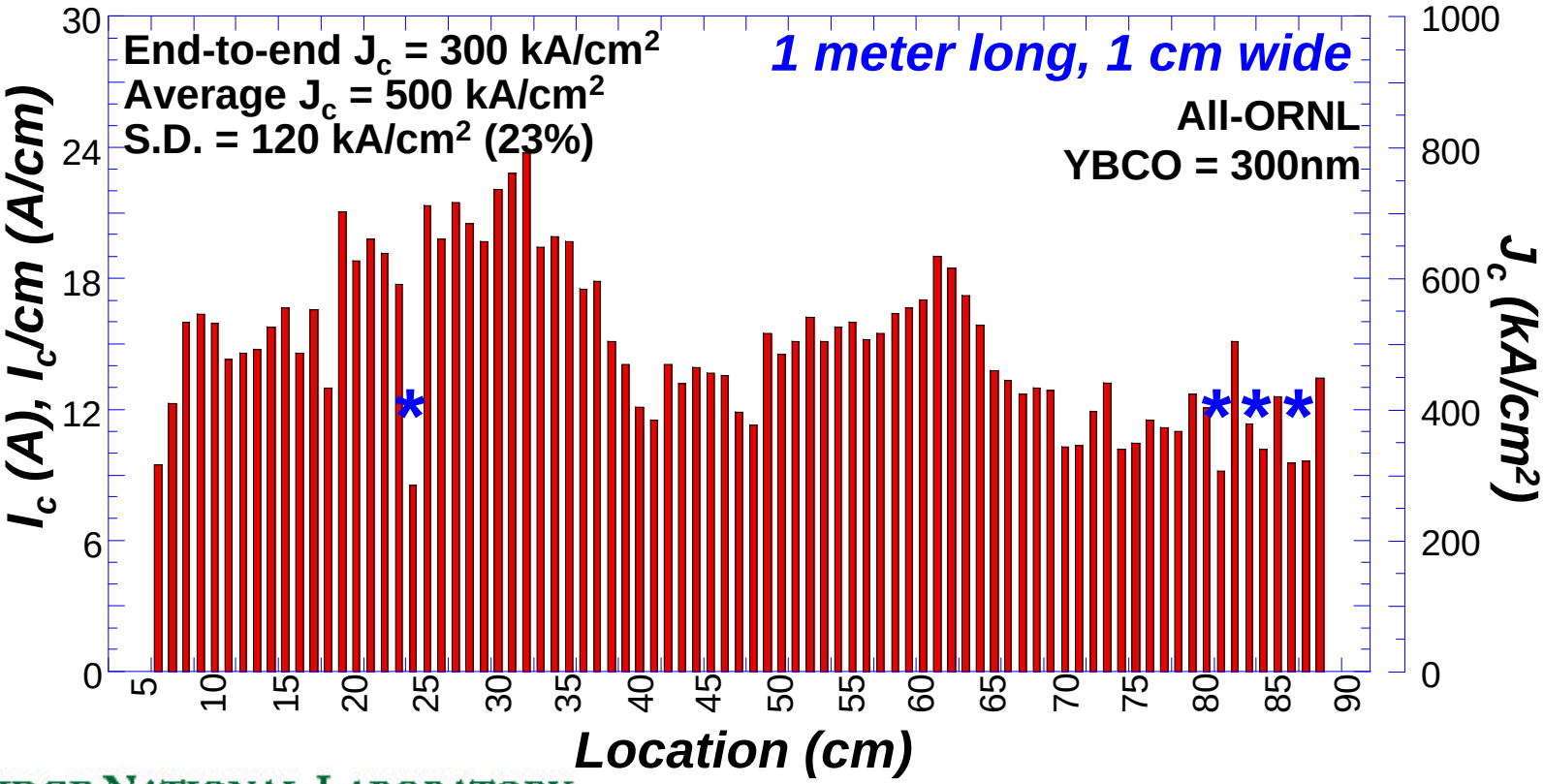


- **Precursor composition may need to be adjusted depending on the thickness of the CeO_2 cap-layer**

K.L. More (ORNL)

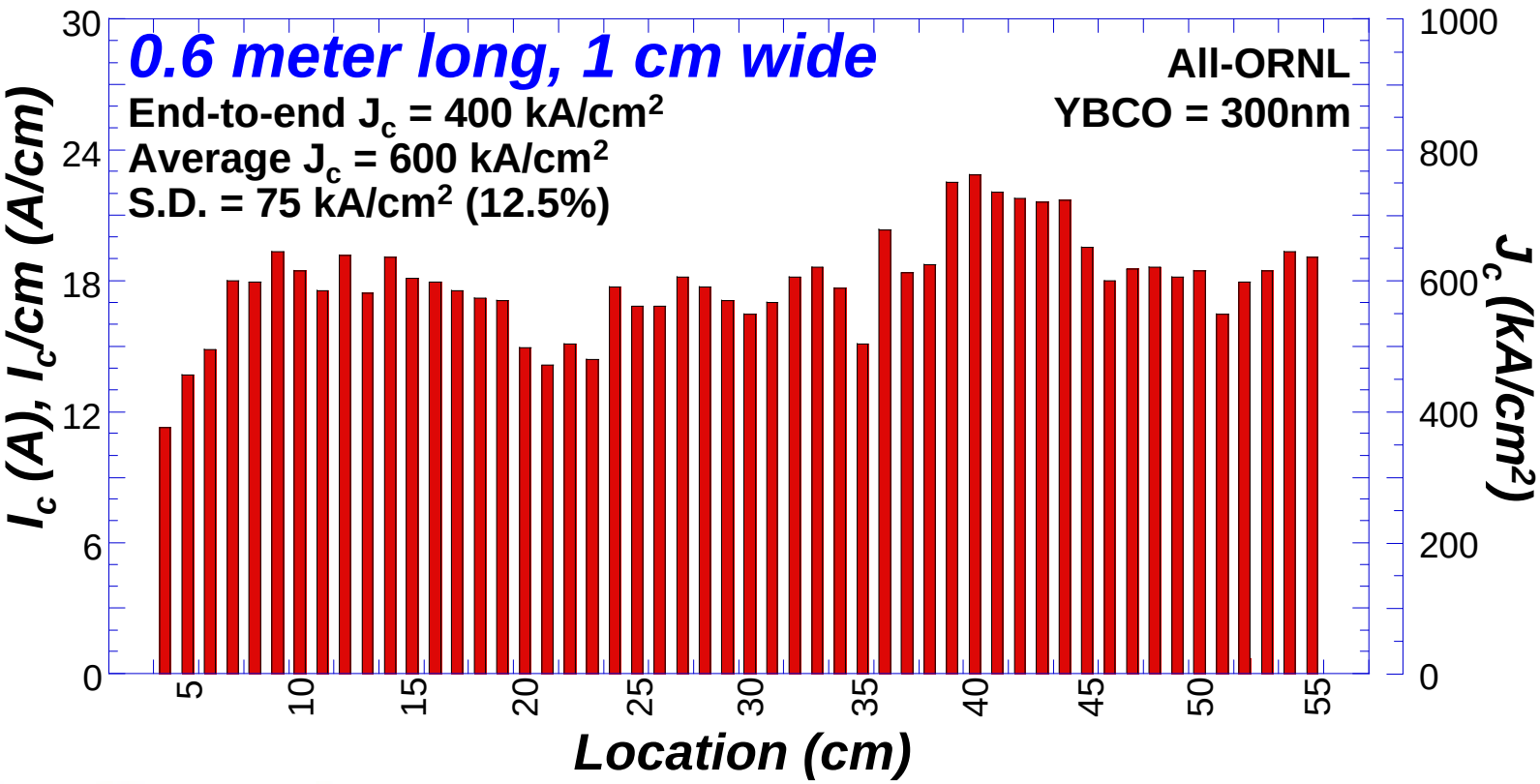
With Dimensional Flaws & Precursor Discharges, Our Meter-Length YBCO has Good but Non-Uniform J_c

- **Best 1 meter on all-PVD ORNL RABiTS with ORNL precursor (kinks in RABiTS, visible line features marked by *):**
- **Buffer texture -**
  - **Dw FWHM: 13.8º (f =0), 8º (f =90)**
  - **Df FWHM: 11.3º, No 45º component**
- **YBCO texture:**
  - **Dw FWHM: 10.4º (f =0), 8º (f =90)**
  - **Df FWHM: 9.4º (113), 10.5º (103), 8.6º (102), No 45º**



Once the Issue with Dimensional Flaws has been Resolved, More Uniform J_c was Obtained in Lengths

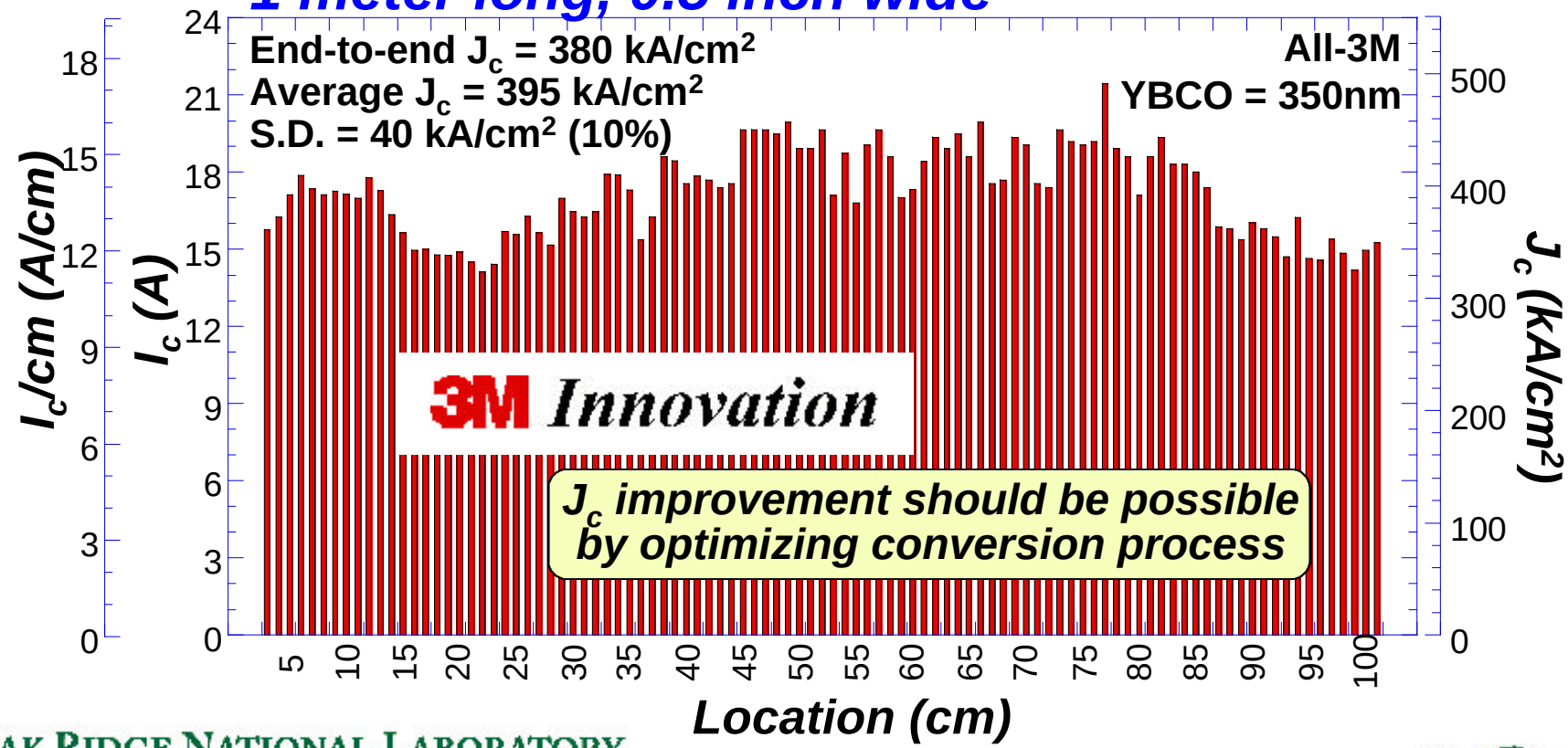
- **Best 0.6 meter on all-PVD ORNL RABiTS with ORNL precursor**
- **Buffer texture -**
  - **Dw FWHM: 12.3º (f =0), 9.8º (f =90)**
  - **Df FWHM: 9.7º, No 45º component**
- **YBCO texture:**
  - **Dw FWHM: 11.6º (f =0), 9.6º (f =90)**
  - **Df FWHM: 9.8º (113), 11.4º (103), 9.5º (102), No 45º**



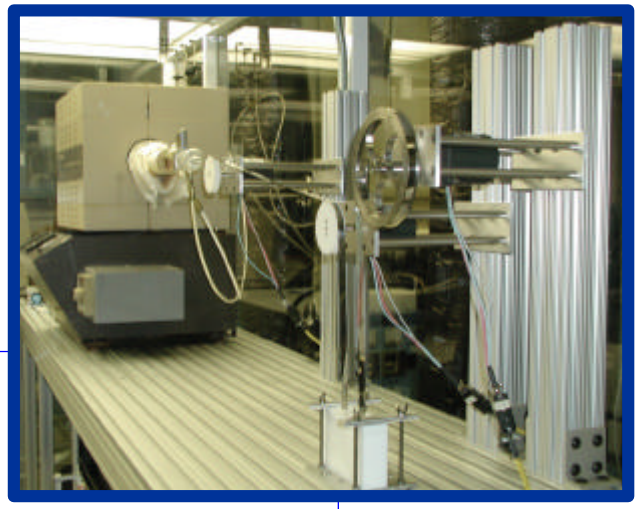
Exciting Results have been Obtained on Meter-length of All-3M Tapes

- **Best 1 meter on all-PVD 3M RABiTS with 3M precursor**
- **Buffer texture -**
  - **Dw FWHM: 14.6º (f =0), 11.5º (f =90)**
  - **Df FWHM: 11.9º, 6.6% 45º rotated**
- **YBCO texture:**
  - **Dw FWHM: 7.9º (f =0), 5.8º (f =90)**
  - **Df FWHM: 9.6º (113), 10.9º (103), 9.7º (102), No 45º**

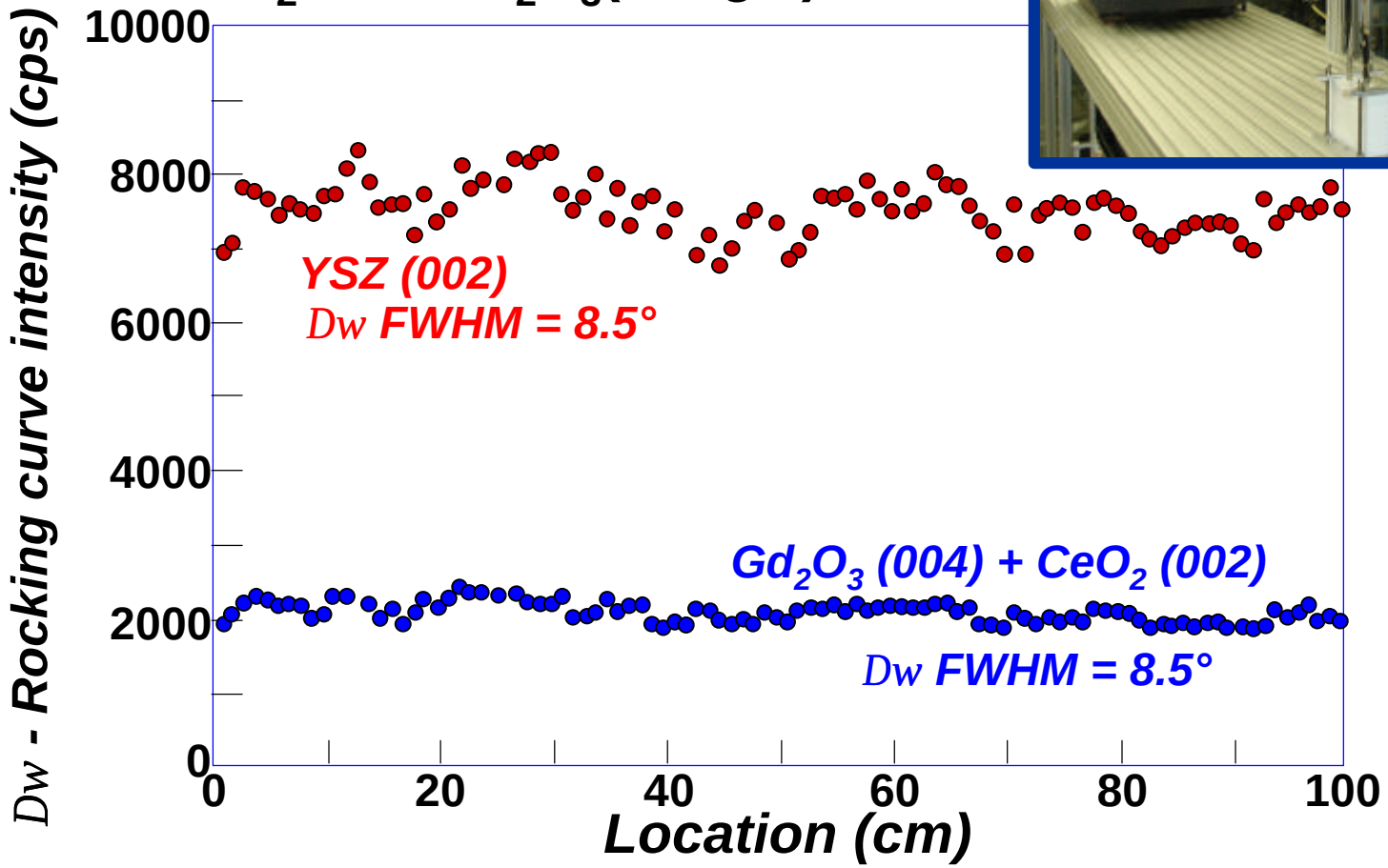
1 meter long, 0.5 inch wide



We have Recent Success in Continuous Dip-Coating of Sol-Gel Seed Layer on Ni

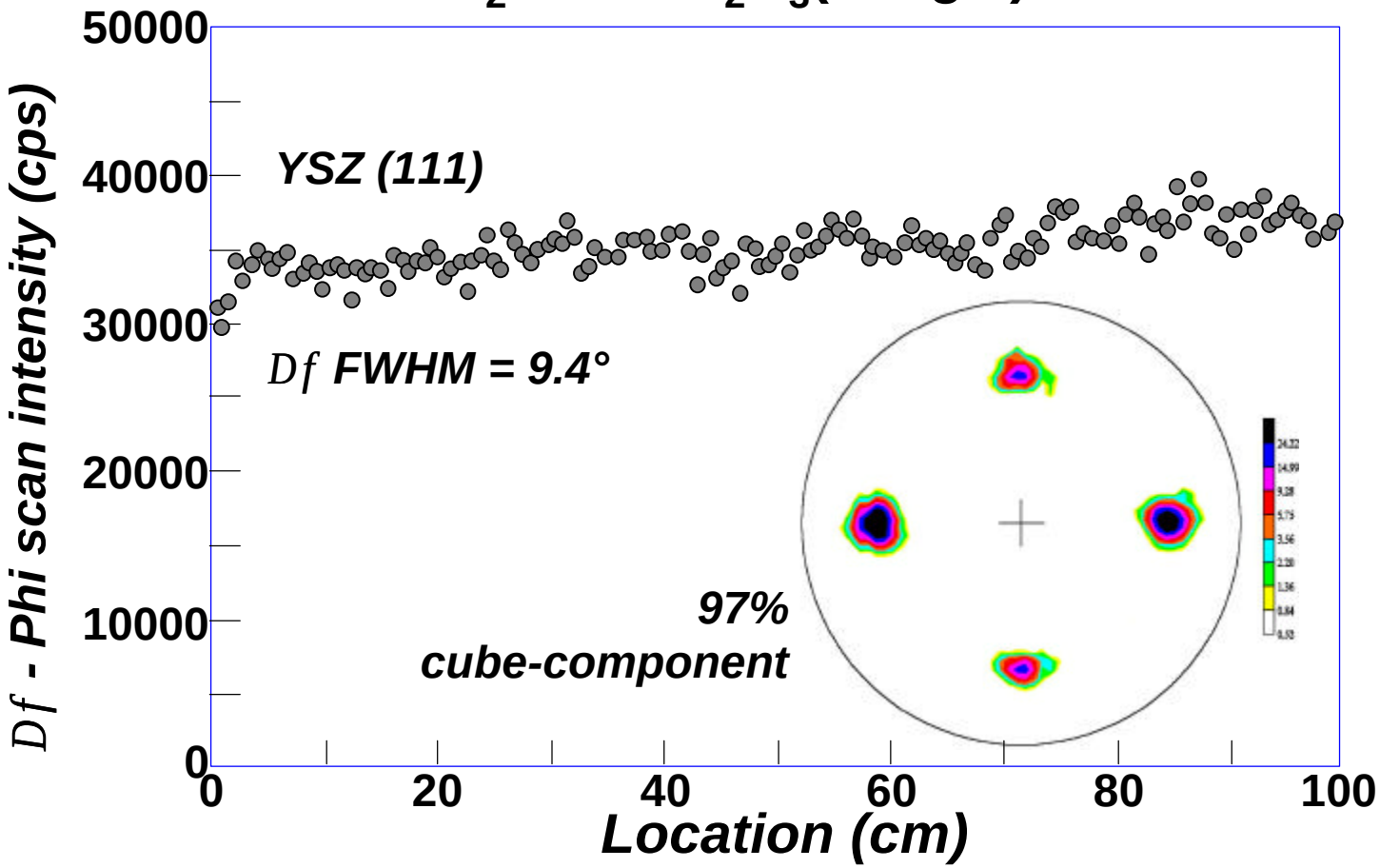


- CeO_2/YSZ/Gd_2O_3(sol-gel)/Ni:



Both YSZ Barrier Layer and CeO_2 Cap Layer Deposited by RF Sputtering Retain the Good Texture of the Gd_2O_3 Seed

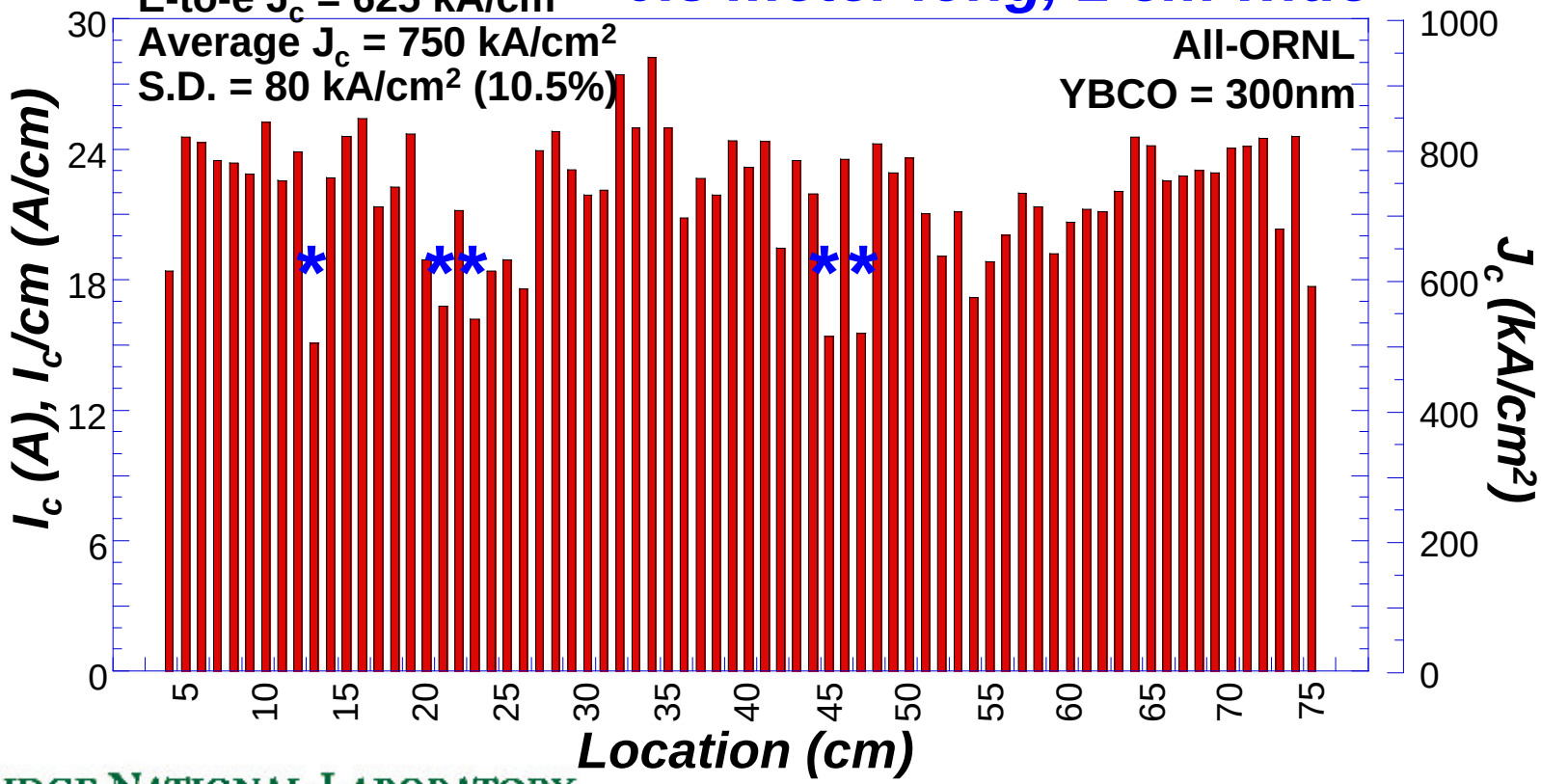
- CeO_2/YSZ/Gd_2O_3(sol-gel)/Ni:



High J_c has been Obtained on Long-Length RABiTS that was Fabricated by Non-Vacuum and Vacuum Methods

- **0.8 meter on all-ORNL RABiTS with Sol-Gel Gd_2O_3 seed & ORNL precursor (visible line features marked by *):**
- **Buffer texture -**
  - **Dw FWHM: 11.2º (f =0), 8.5º (f =90)**
  - **Df FWHM: 9.4º, small 45º component**
- **YBCO texture:**
  - **Dw FWHM: 9.9º (f =0), 7.5º (f =90)**
  - **Df FWHM: 9.1º (113), 10.3º (103), 8.6º (102), No 45º**

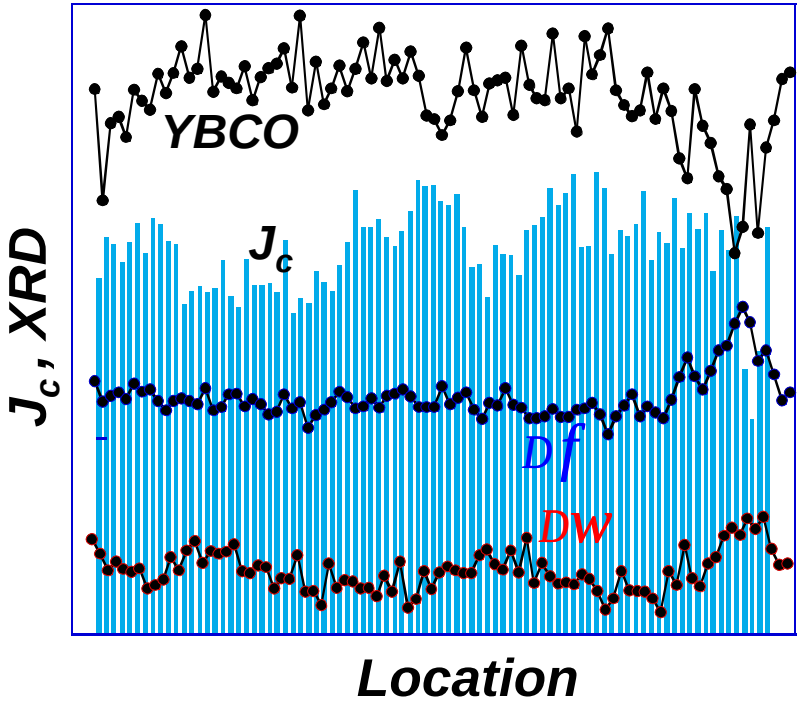
0.8 meter long, 1 cm wide



We are in the Process of Collecting a Growing Database, and Have Begun to Conduct Detailed Statistical Analysis

- **Detailed J_c and XRD data is being collected along 1 cm increments of long tapes.**
- **Multi-variate regressions are being performed between J_c and relevant XRD data**
  - **YBCO: (00l) Df (113) Dw (f = 0) Dw (f = 90)**

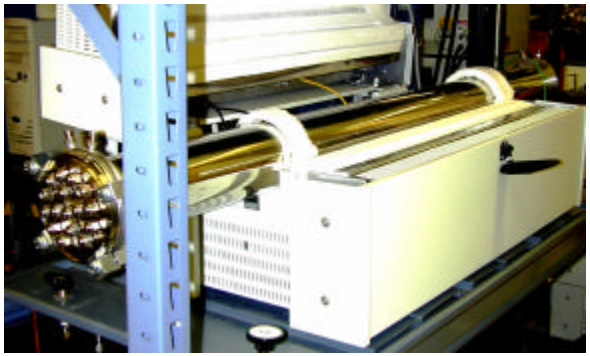
Analyses showed that ~20% of J_c variation can be attributed to texture variation in the substrate → Significant component of J_c non-uniformity is due to extrinsic factors



Substantial headroom for improvement in J_c uniformity

Can Large-Area Conversion be Performed in a Cost-Effective Manner?

- **Outstanding questions:**
  - Can "very large" areas, especially along the width, be converted uniformly?
  - Will the required processing time increase with "volume"?
  - *Reduction in chamber pressure may aid in HF removal, reduce gas expenditure, decrease the boundary layer and increase the diffusion rate.*

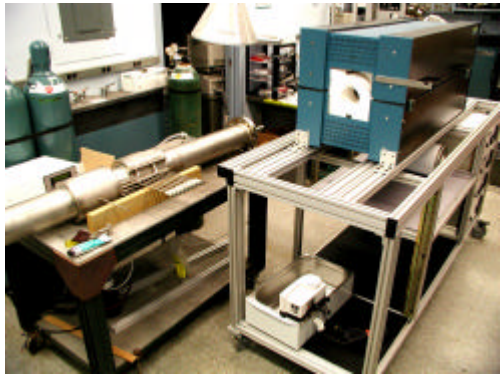


Variable gas injection furnace

- 12cm x 7cm samples capability
- Variable gas inlet-outlet configurations
- 0.1 to 2 atm chamber pressure

Low-pressure conversion unit

- 5 cm samples
- In-situ energy dispersive XRD
- In-situ mass spectroscopy (1E-5 to 1E-2 atm)



Batch conversion furnace

- >6 m tape capability
- 0.05 to 1 atm chamber pressure

FY 2001 Plans and Performance

To improve the structure, processing and throughput of buffer layers:

- Determined that sulfur superstructure on metal surface is responsible for cube texture in seed layer.
- Succeeded in incorporating H_2S source to increase the surface sulfur content in our textured metal (stationary and reel-to-reel)
  → promotes seed-layer epitaxy.
- Succeeded in long-length reel-to-reel dip coating of sol-gel seed layers with good texture and structural characteristics.
- Ready to take delivery of a reel-to-reel RF sputtering system with linear guns (expected to increase our YSZ deposition speed by 20 times).

FY 2001 Plans and Performance (Cont.)

☑ Further develop ex-situ process to convert YBCO in 1 meter lengths:

- Identified and devised solutions for major J_c limiting issues.
- Improved tape handling that resulted in immediate improvement in J_c uniformity.
- Began systematic study of long-length conversion furnace.
- Determined that overall J_c is sensitive to gas flow rate.
- Encountered indications that processing conditions may be different for different precursors.
- Designed and constructed various systems to investigate effects of gas delivery configuration, large area conversion, and reduced processing pressure.

FY 2001 Plans and Performance (Cont.)

1 MA/cm^2 in 1 meter length:

- RABiTS texture limited the overall J_c value.
- Precursor non-stoichiometry limited the J_c uniformity → end-to-end J_c.

Target
E-to-e J_c= 1 MA/cm^2

FY2001

All-ORNL, 0.6meter
E-to-e J_c= 400 kA/cm^2
Highest J_c= 800 kA/cm^2
S.D.=12.5%

All-ORNL, 0.8meter
E-to-e J_c= 625 kA/cm^2
Highest J_c= 950 kA/cm^2
S.D.=10.5%

All-ORNL, 1 meter
E-to-e J_c= 300 kA/cm^2
Highest J_c= 800 kA/cm^2
S.D.=23%

All-3M, 1 meter
E-to-e J_c= 380 kA/cm^2
Highest J_c= 500 kA/cm^2
S.D.=10%

FY2000

All-ORNL, 1 meter
E-to-e J_c= 100 kA/cm^2
Highest J_c= 750 kA/cm^2
S.D.=24%

- Collaboration with LANL on conversion of YBCO on IBAD. Is planned

FY 2002 Plans

- **Long-length RABiTS development:**
  - Improve texture of metal substrates: Ni and Alloys.
  - Optimize sulfur treatment to demonstrate *consistent* epitaxial seed layer deposition.
  - Long-length *strengthened* RABiTS by vacuum and non-vacuum methods.

- **"Large-area" ex-situ YBCO conversion:**
  - Verify that conversion parameters vary with precursor, and the cause thereof.
  - Refine reel-to-reel conversion technique to obtain 1 MA/cm^2 on meter-length 3000Å-thick YBCO on *strengthened* RABiTS.
  - Establish basic reel-to-reel conversion parameters of thick YBCO precursor.
  - Establish basic processing parameters of Large-Area Batch furnace.
  - Determine the effectiveness of reduced pressure in aiding conversion.

- **External collaborations:**
  - Continue to assist partners in achieving their programmatic goals.
  - Collaboration with LANL to convert YBCO on IBAD.
  - Provide collaborators with RABiTS and YBCO coated conductors for innovative research.

Research Integration: Strong and Close

3M Innovation

- Converted total of ~10 meters all-3M tapes.
- 3-M precursor deposition on all-ORNL RABiTS.
- Joint fabrication of RABiTS.
- Regular reel-to-reel XRD of all-3M RABiTS.
- Technology transfer to company.
- Private briefings & quarterly meetings.
- Weekly teleconferences.
- Joint publications.

- "High-rate" fabrication of long-length RABiTS.
- Long-length precursor deposition with uniform stoichiometry.

American Superconductor

- Provided RABiTS with seed-layers to ASC.
- Joint fabrication and evaluation of RABiTS.
- Reel-to-reel XRD of ASC RABiTS.
- Technology transfer to company.
- Private briefings & on-site meetings.
- Bi-weekly teleconferences.
- Joint publications.

- All reel-to-reel TFA-based YBCO on RABiTS (>10cm) with J_c of 1 MA/cm^2 & I_c of 42A.

Research Integration (Cont.)

- Deposited PLD YBCO on MCT CCVD RABiTS produced by company.
- Reel-to-reel XRD of long-length MCT CCVD RABiTS.
- Joint investigation on textured metals with Oxford.
- Technology transfer to companies.
- Private briefings, on-site meetings & teleconferences.

- 1 MA/cm^2 J_c on MCT CCVD RABiTS.

LANL

- Samples exchange.
- Quarterly meetings and weekly telephone conferences via 3M CRADA.

FSU / MIT

- Provided short and long YBCO coated conductors for DOE sponsored research.

Dissemination of Technical Information

- Over 50 publications and presentations.
- 5 US patents issued.