Exploring New Directions in the Science and Technology of IBAD Texturing

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Acknowledgements

Coworkers:

- Brady J. Gibbons (staff): PLD
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- Chris Sheehan (tech)
- John Rowley (tech)
- Konrad Güth (undergrad)
- Ruud Steenwelle (undergrad)

Collaborators:

- LANL
  - Terry Holesinger
  - Paul Arendt, Liliana Stan
- Sandia National Lab
  - Paul Clem
- Stanford University
  - Bob Hammond, Randy Groves

Outline

- Introduction/motivation
- Experimental technology: tools and methodology
- Scaling of texture evolution
- Speed of texture formation
- Best results on MgO texture
- New IBAD materials
- Substrate preparation
- Summary slides: performance, results, research integration, future plans

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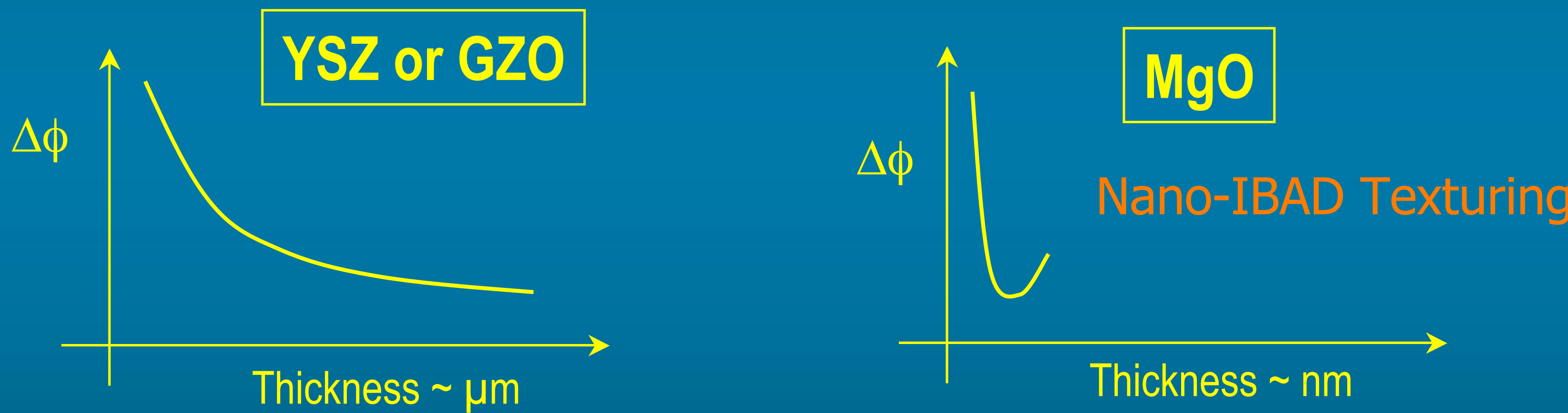
Introduction: IBAD-YSZ vs IBAD-MgO

- Ion-Beam Assisted Deposition (IBAD) for Texturing of Films
- IBAD-YSZ: Y. Iijima et al., Fujikura, 1991
- IBAD-MgO: R.H. Hammond et al., Stanford University, 1995

In-Plane Texture Evolution



- Phase space for exploration is enormous:

  IBAD material, substrate, substrate preparation, ion energy, ion-to-atom ratio, etc.

⇨ Combinatorial approach to research

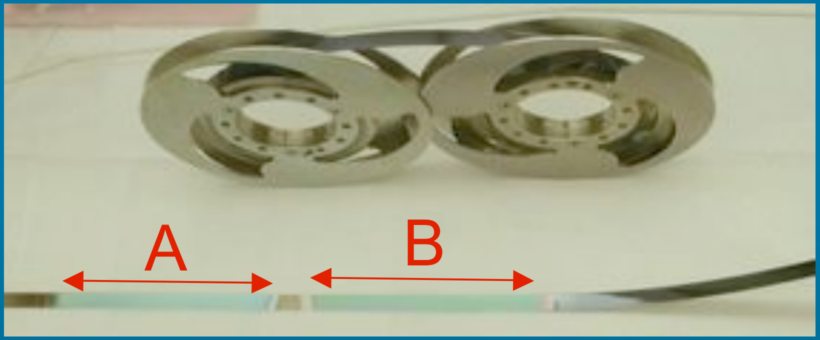
Motivation for Basic and Applied Research

- Fundamental questions remain:
  - What are exactly the mechanisms for texture formation (esp. for nano-IBAD)?
  - Which materials can be used in texture formation?
    - Fluorite and pyrochlore structures exhibit the slow texture evolution (55°, <111>, 300 eV)
    - Rocksalt structures exhibit the fast texture evolution (45°, <110>, 800 eV)
- Applied questions:
  - What is the best alignment that can be attained?
  - Can it be further improved?
  - How fast can the texture be formed?
  - Can we engineer the crystal: tune the lattice parameter? Adjust the vicinal angle? Use arbitrary substrate?

Combinatorial approach to IBAD exploration

- High-Throughput Experimentation through a Linear Combinatorial approach
- Available to us thanks to reel-to-reel processing

A and B have different layer depositions



- Hundreds of meters of tape available through long length CC preparation: clean and polished metal tape
- *In situ* monitoring is key

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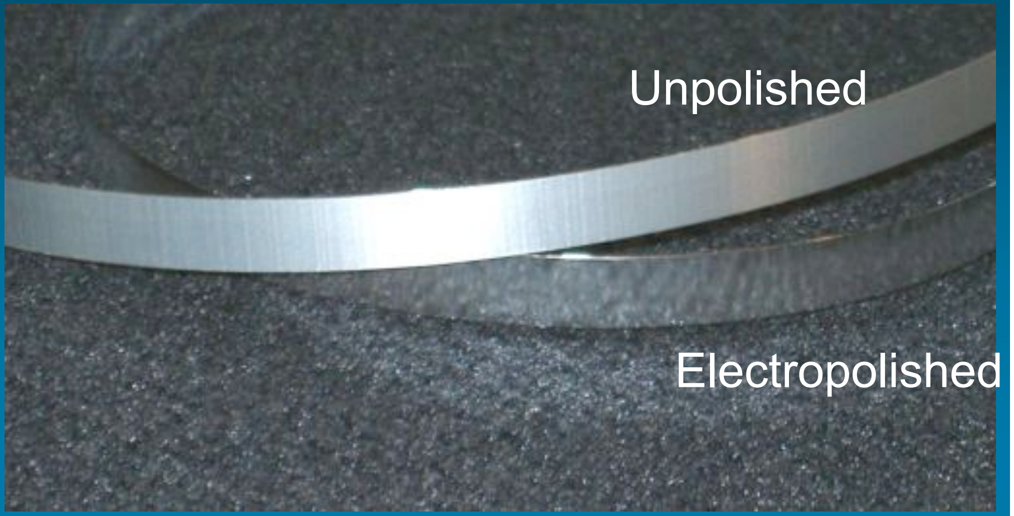
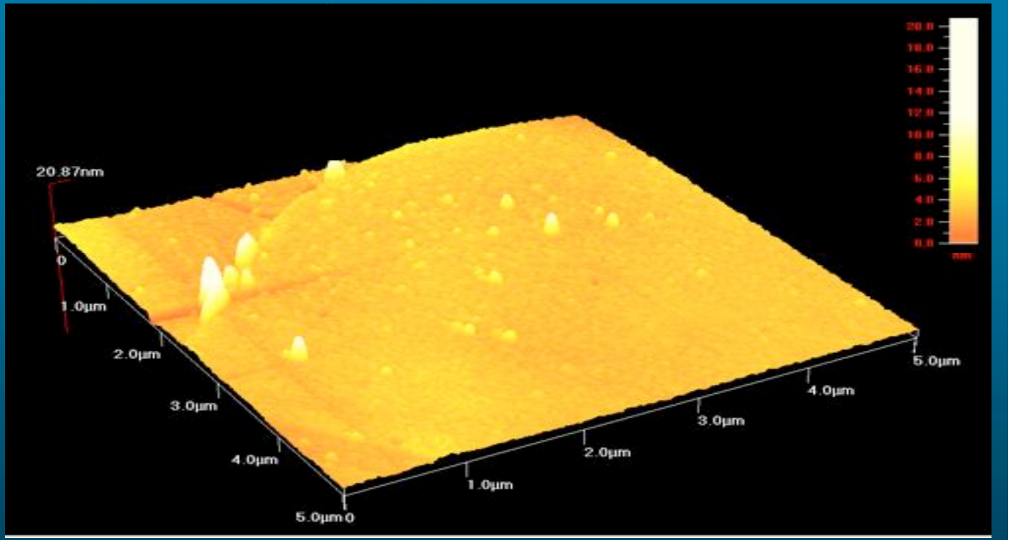
Key enabler: smooth tape in long lengths

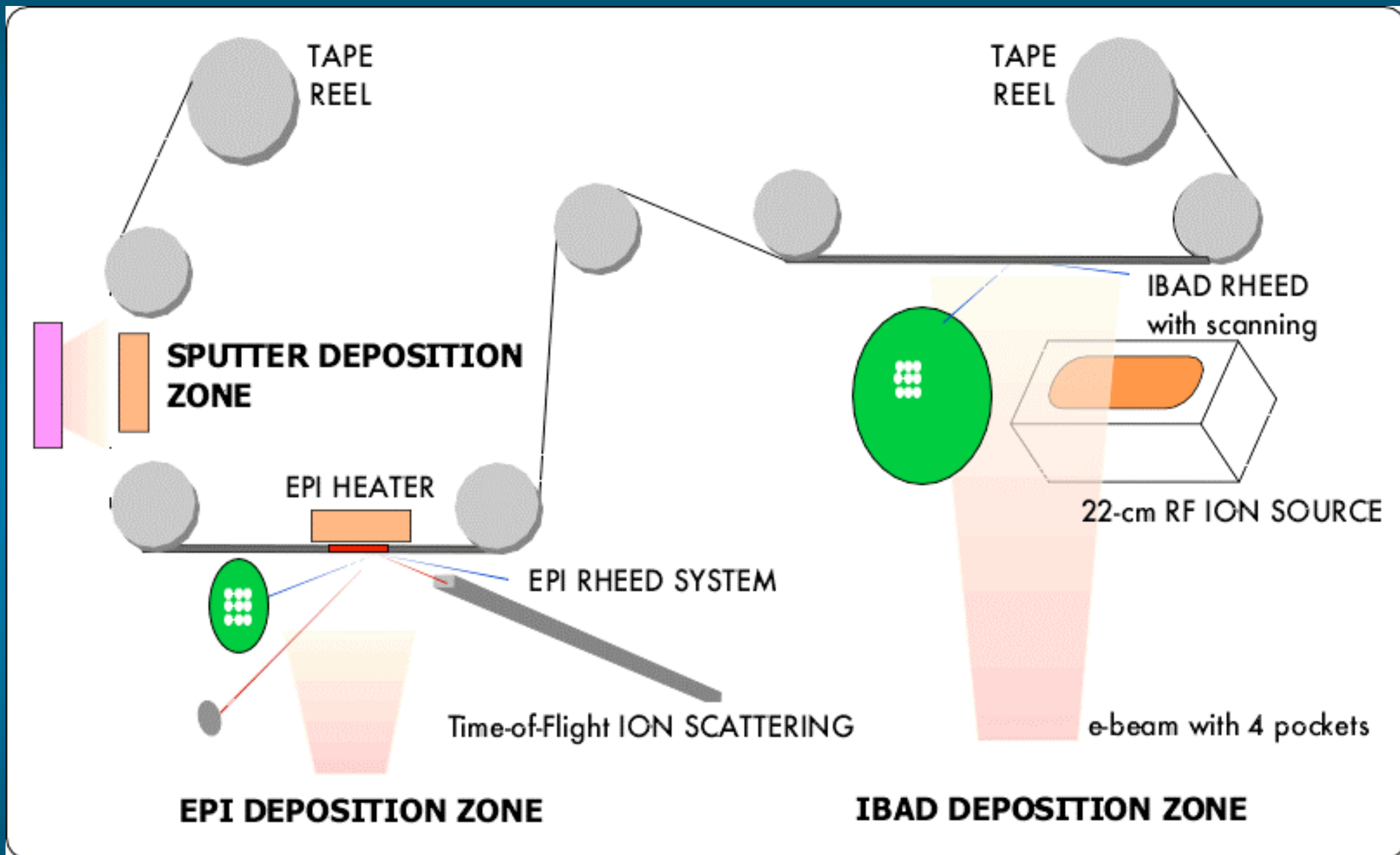
Electropolishing





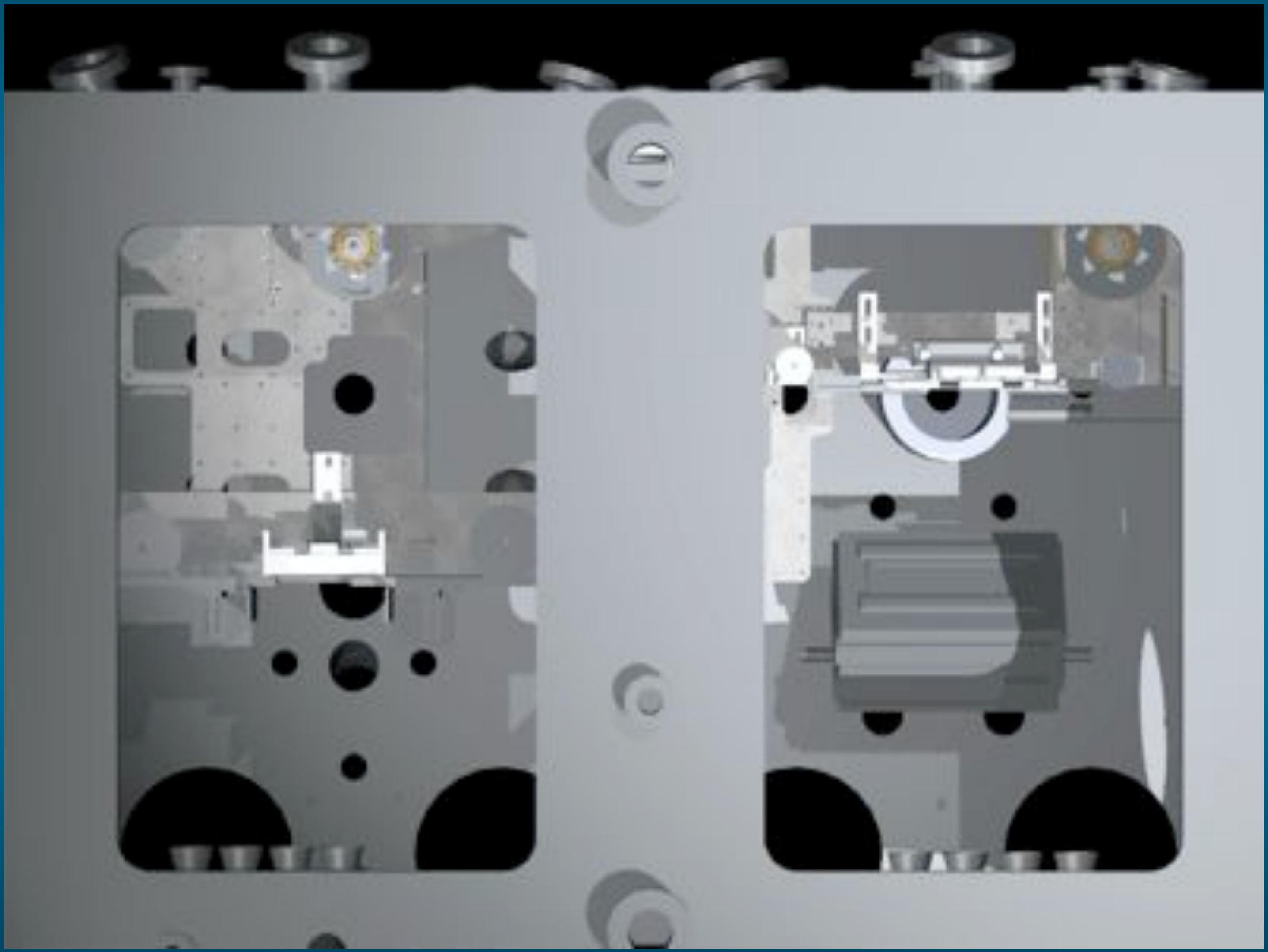
- Clean smooth tape in 100 meter lengths
- RMS roughness ~0.5 nm on 5 x 5 µm scale

Schematic of LANL IBAD reel-to-reel system



- Flexible for R&D of various materials systems

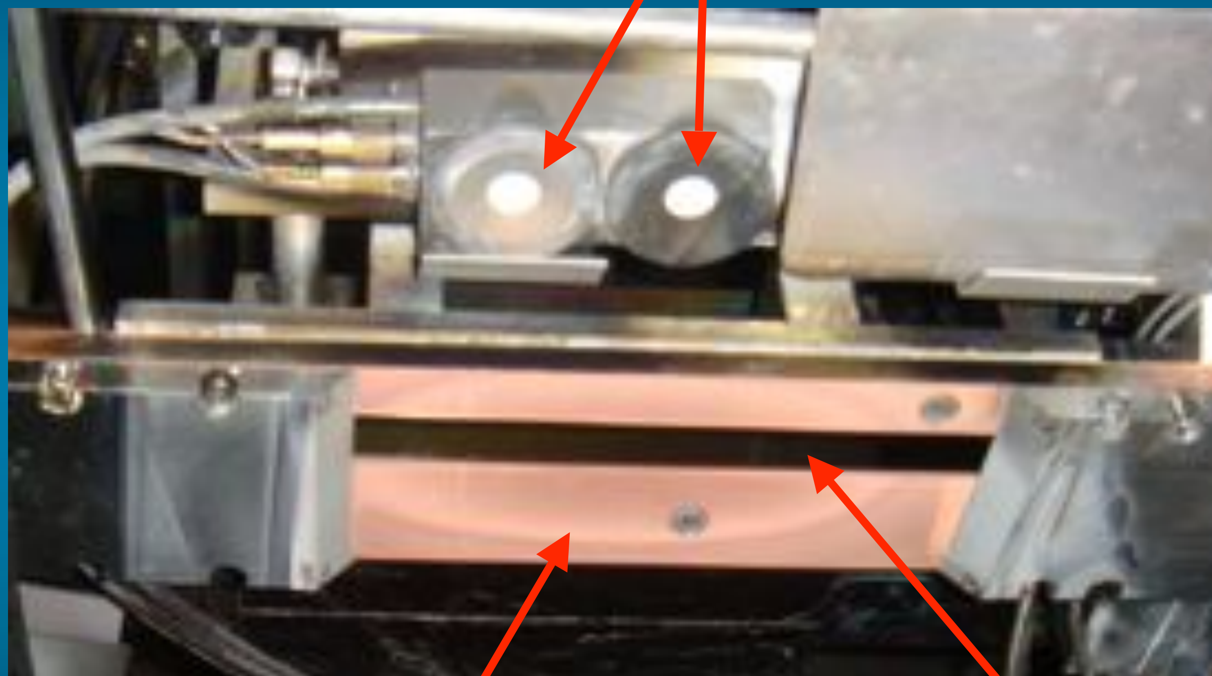
IBAD reel-to-reel system



IBAD deposition zone setup

QCM's

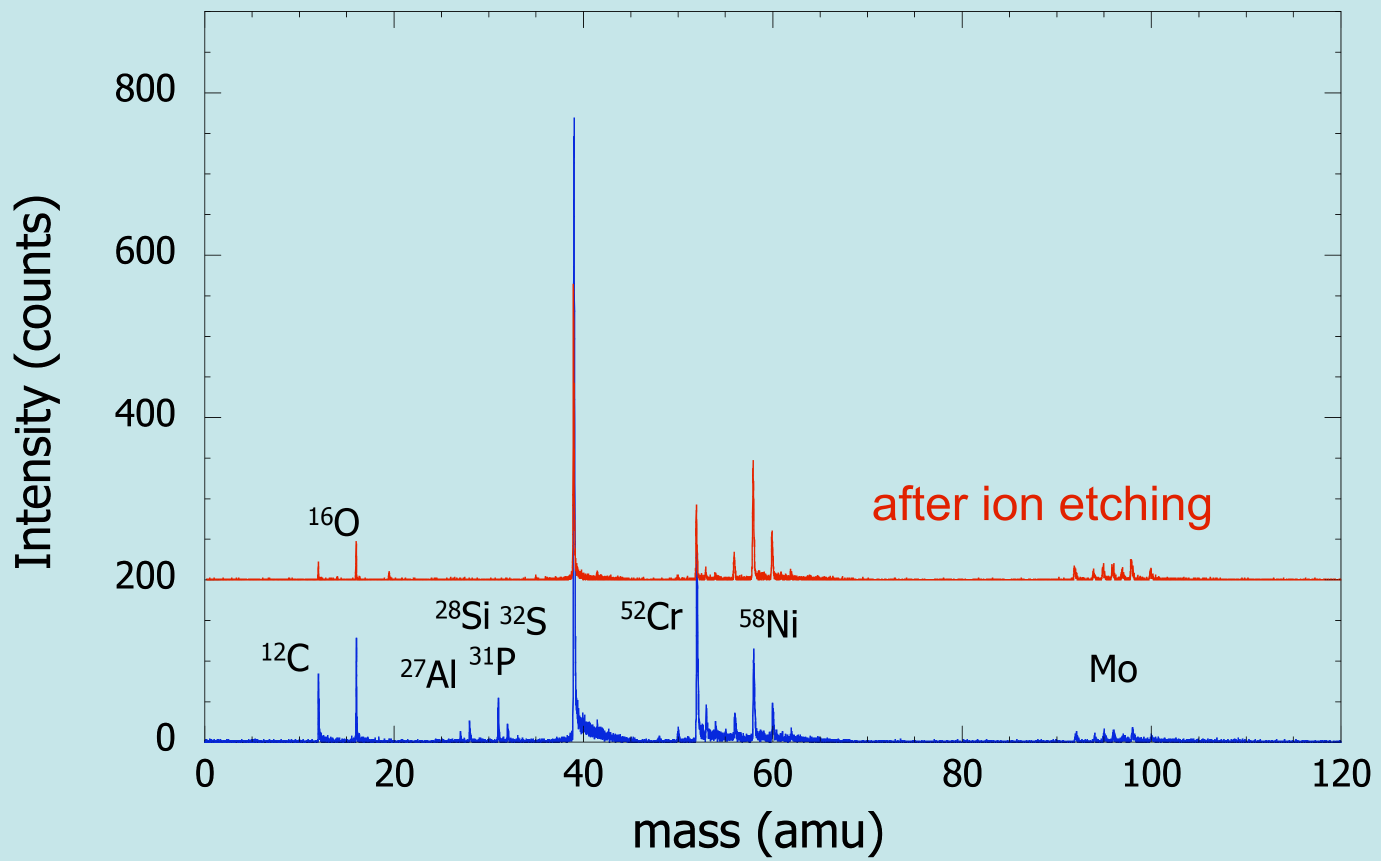
Cu block tilting



Water cooled copper block

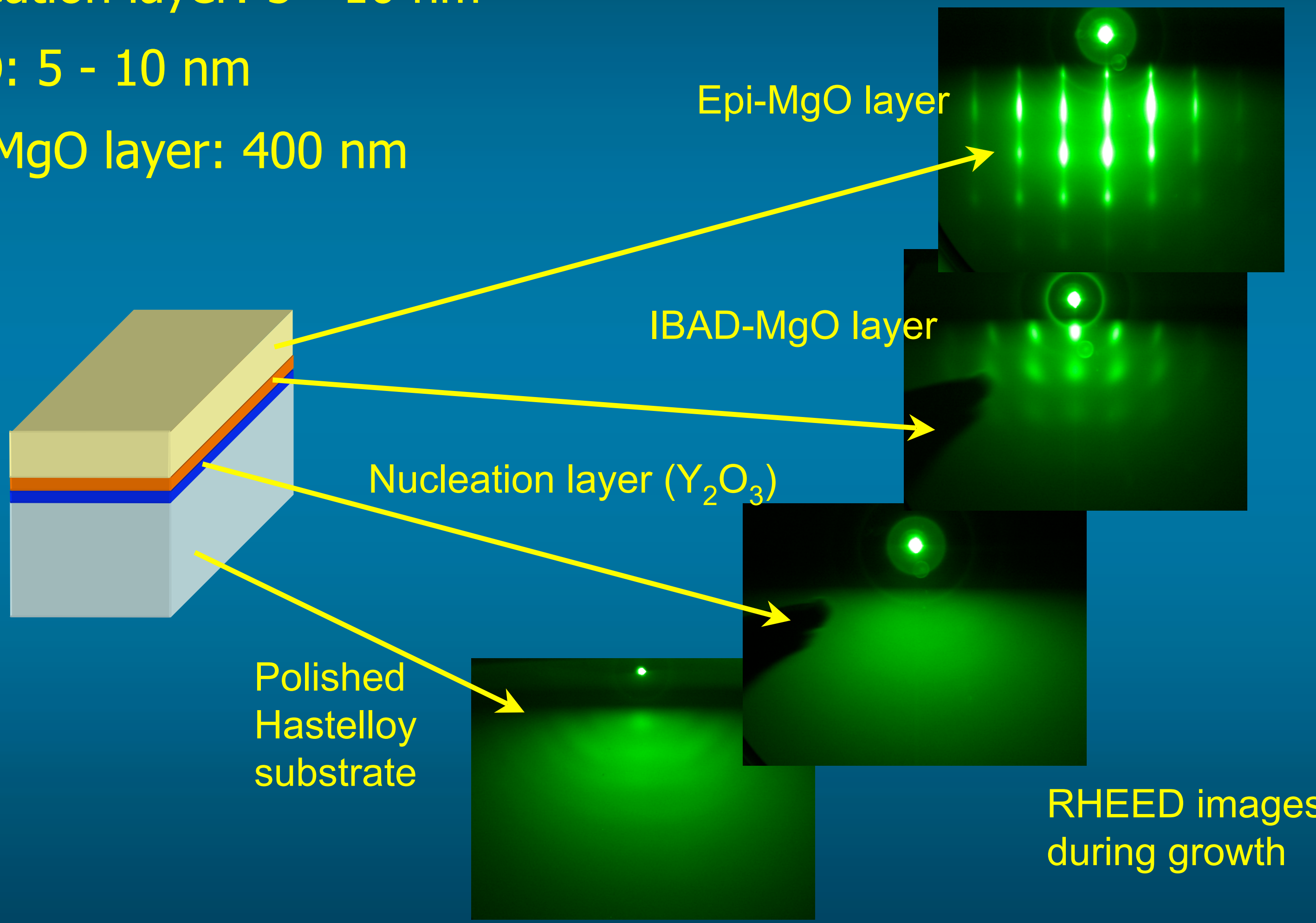
TAPE

Direct recoil mass spectroscopy of bare tape

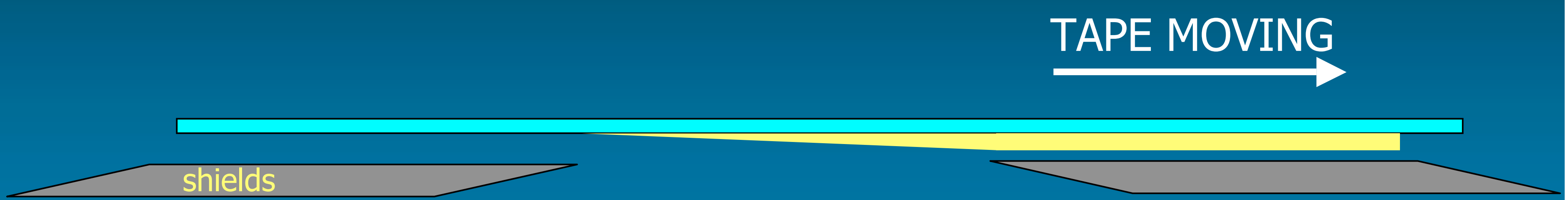


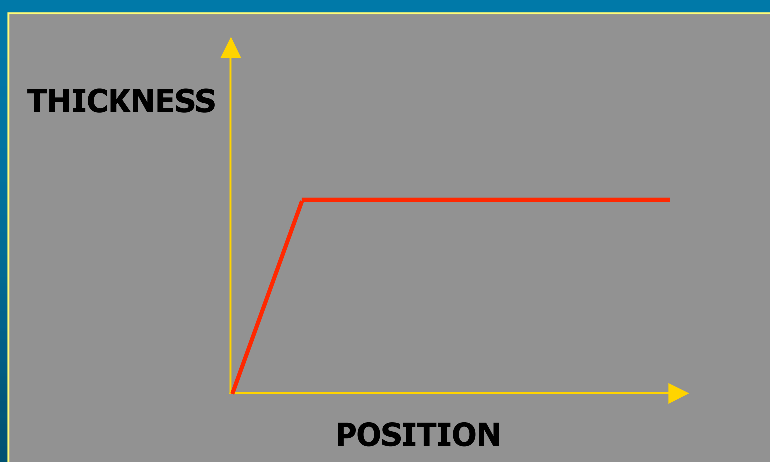
IBAD template architecture

- Y_2O_3 nucleation layer: 5 - 10 nm
- IBAD-MgO: 5 - 10 nm
- Thick epi-MgO layer: 400 nm



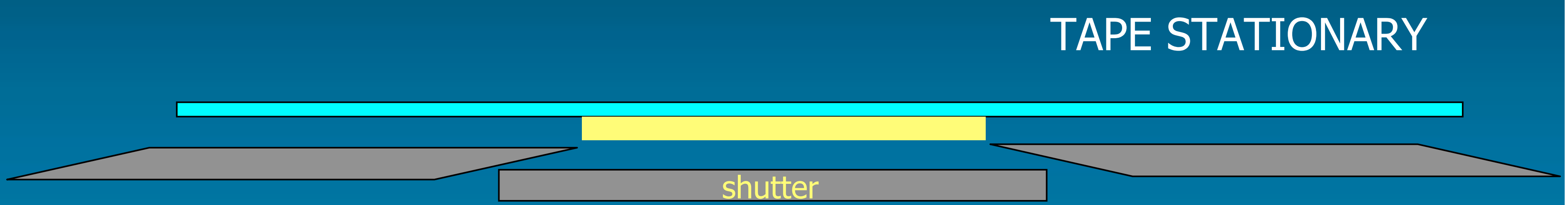
Combi experiment type I: moving tape

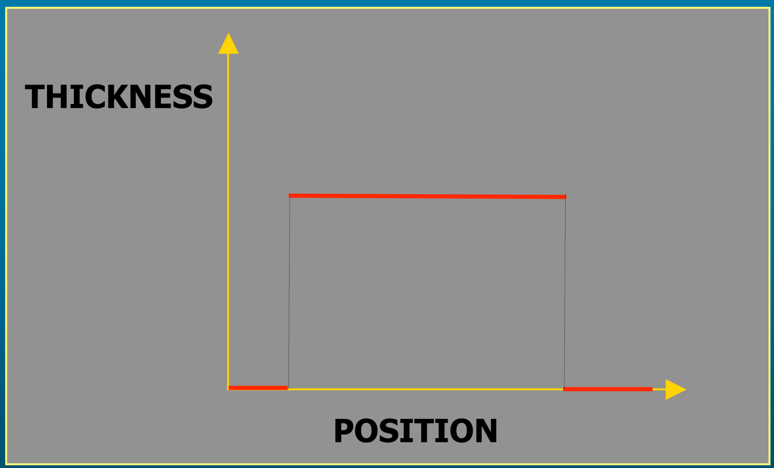




- Good for making long uniform samples
- Base for other combi experiments

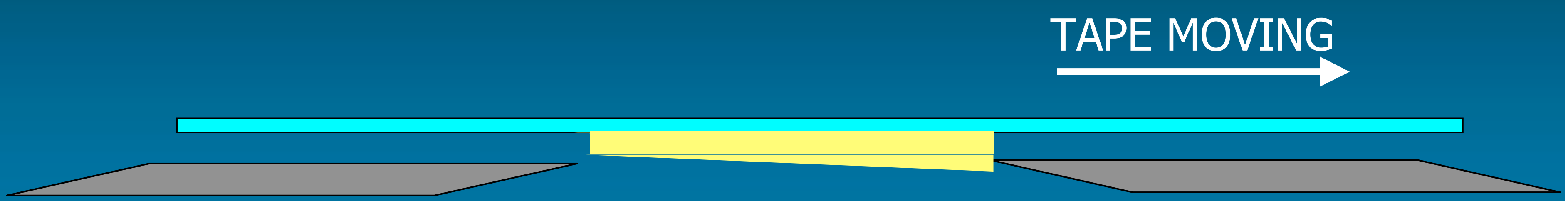
Combi experiment type II: stationary tape

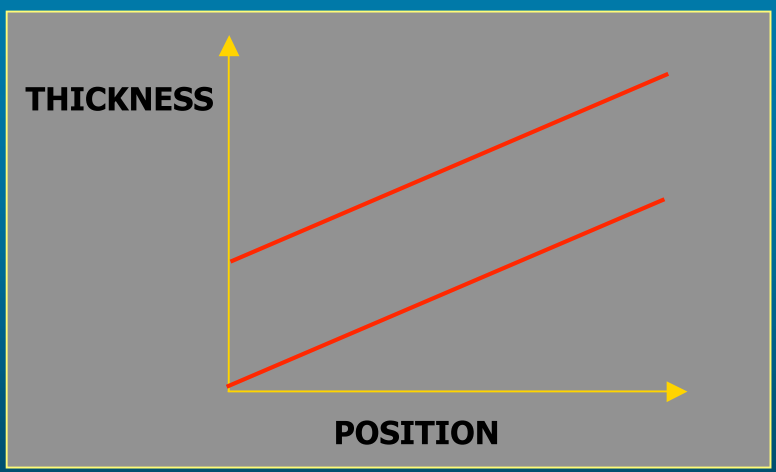




- Obtain uniform 10 cm long sample
- Good for control experiments to determine uniformity
- Doesn't use much tape

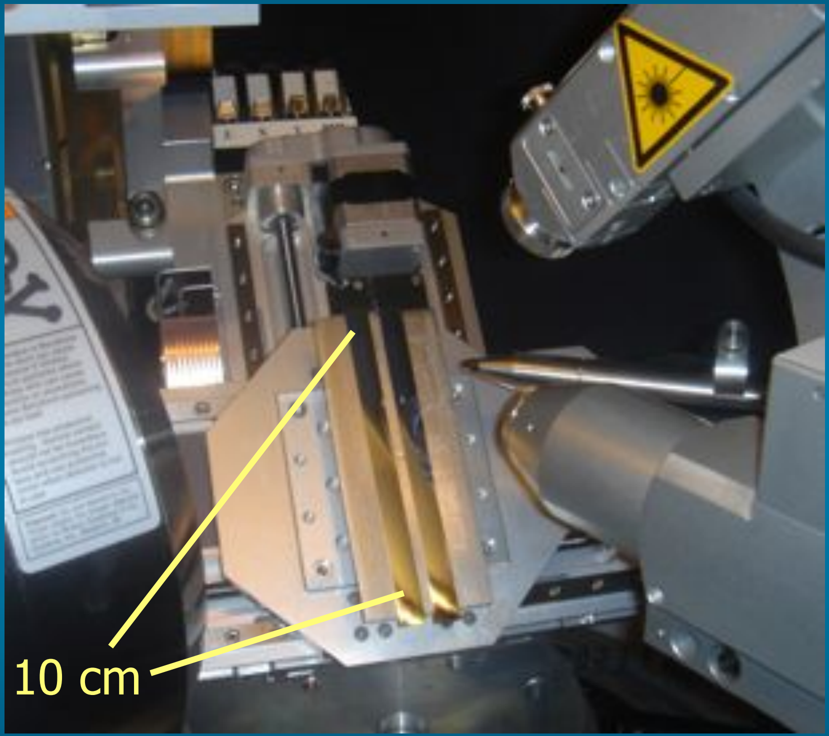
Combi experiment type III: moving and stationary

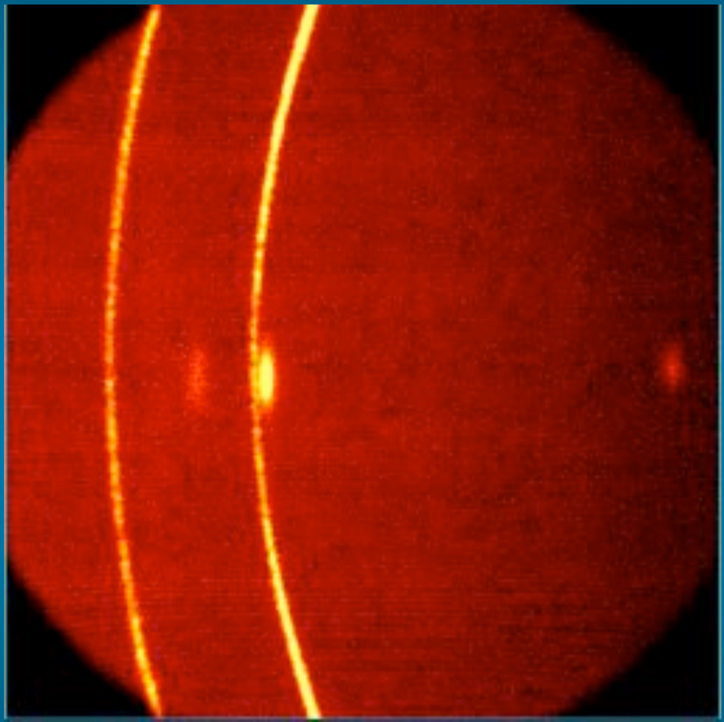




- Obtain a thickness profile over 10 cm length
- Predetermined thickness range
- Good for experiments on interaction with other layers

Positional characterization





Combi YBCO PLD sample made by Brady Gibbons

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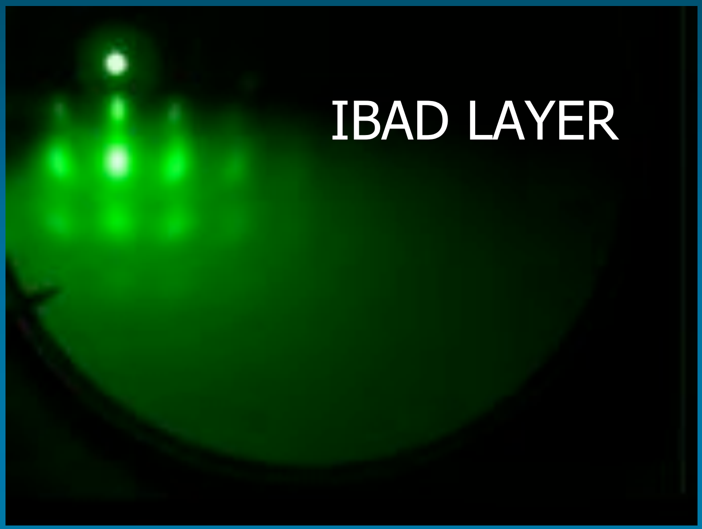
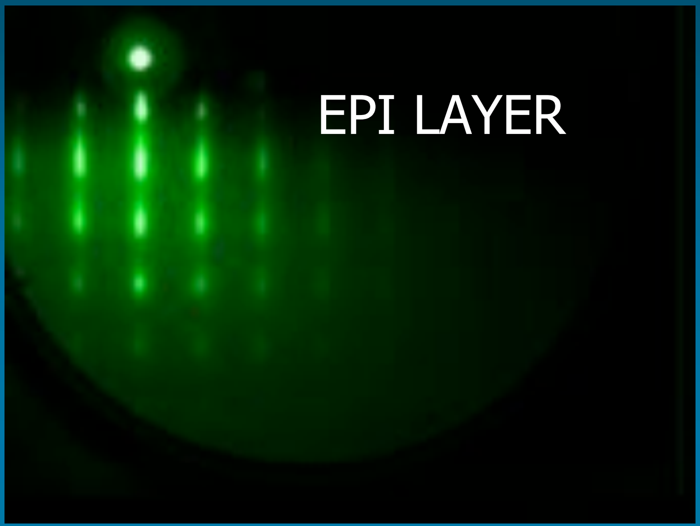
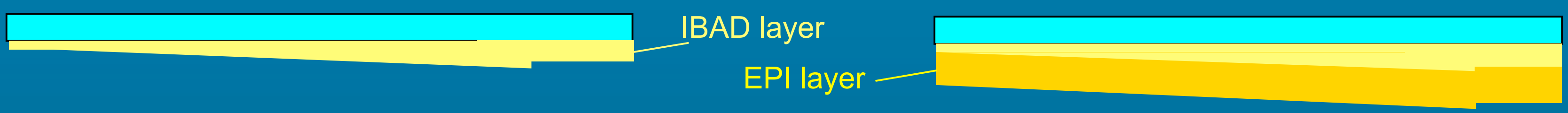
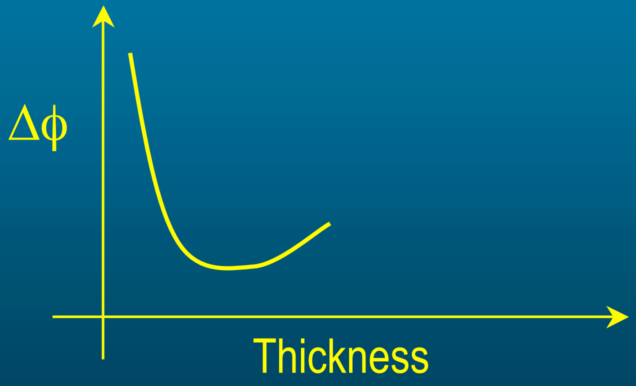
IBAD-MgO thickness evolution

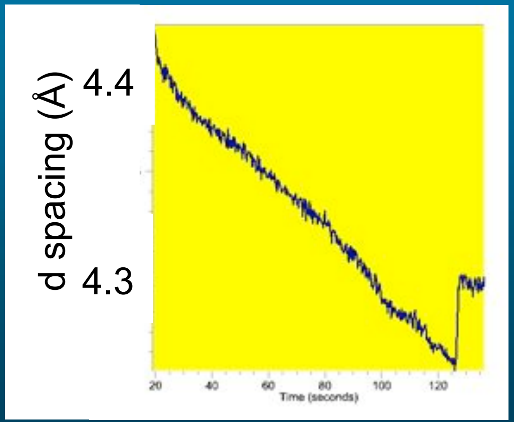


RHEED images taken after growth with moving tape









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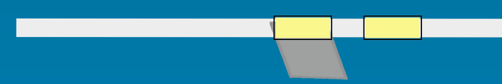
IBAD ion-to-atom ratio

- IBAD depositions are usually characterized by the ratio of incoming ions and atoms or molecules, *r*
- We define ρ which is proportional to *r:*

$$\rho = 1 - t_{QCM1}/t_{QCM2}$$

QCM2 exposed only to MgO beam

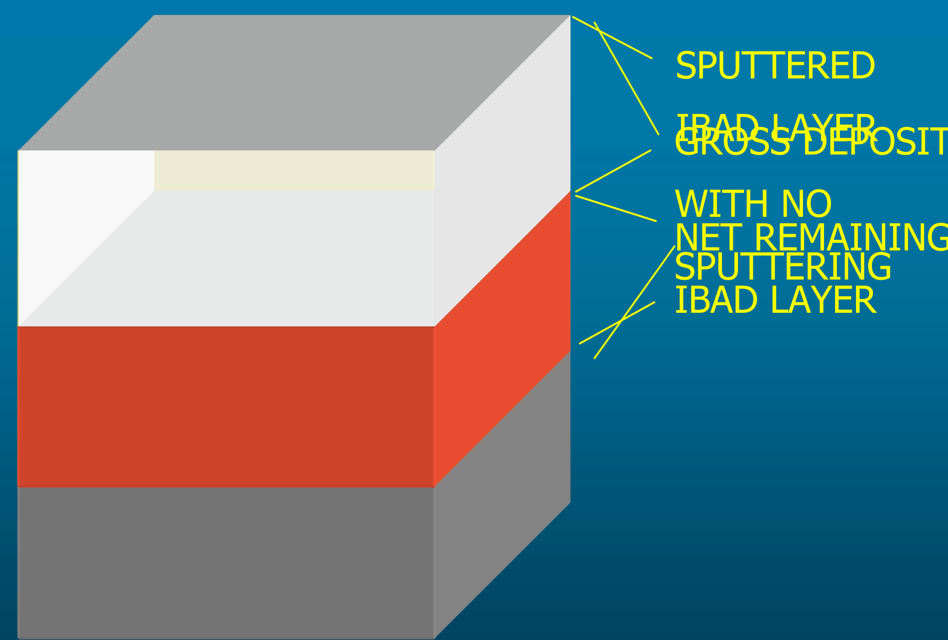
QCM1 exposed to MgO and ion beams



$$\rho = \frac{\text{SPUTTERED THICKNESS}}{\text{GROSS DEPOSIT WITHOUT ETCHING}}$$

$$\rho_{\text{measured}} \sim \rho_{\text{sample}}$$

(not equal since conditions are not the same on the QCM as the sample)



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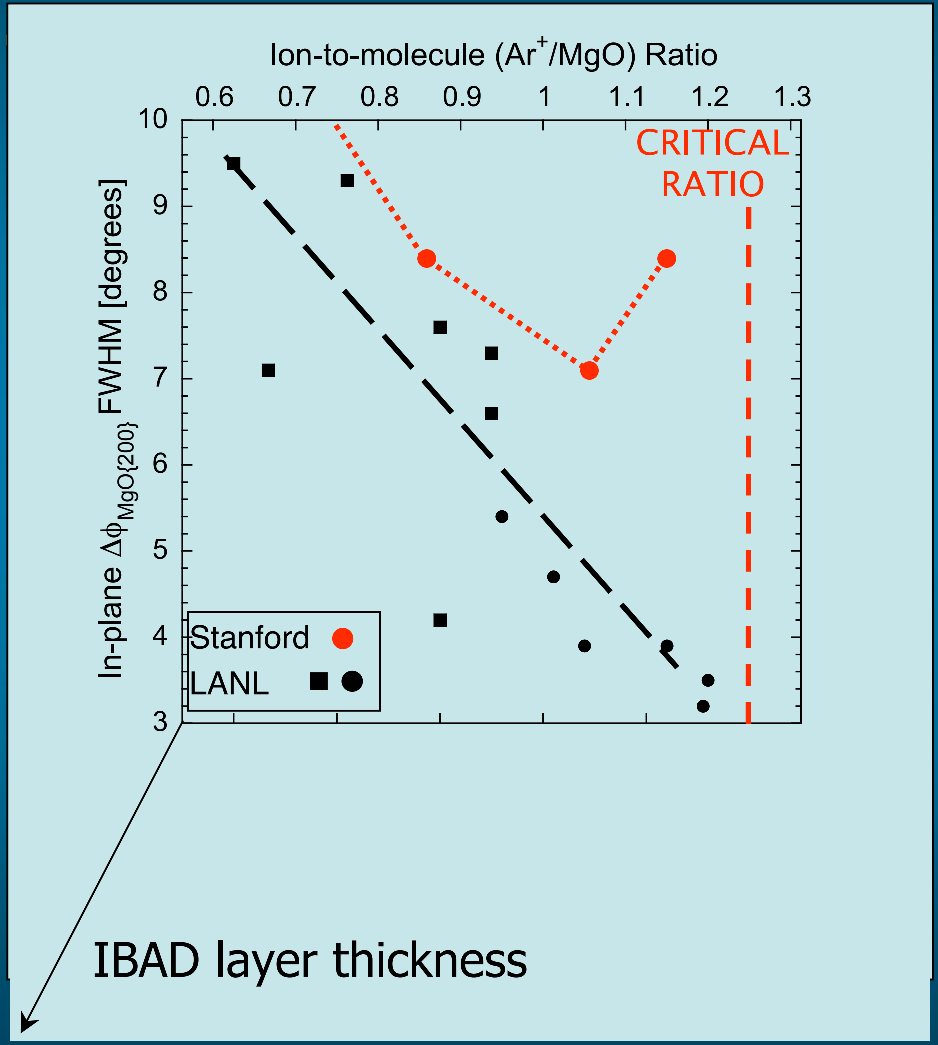
Ion-to-atom ratio during IBAD texturing

- Many IBAD parameters influence final texture:
  - temperature
  - beam energy
  - nucleation surface
- Early data indicated that texture improves as the ion-to-atom ratio, *r* increases; *r* is proportional to $\rho = 1 - t_{QCM1}/t_{QCM2}$

A.T. Findikoglu et al J. Mater. Res. **19**, 501 (2004)



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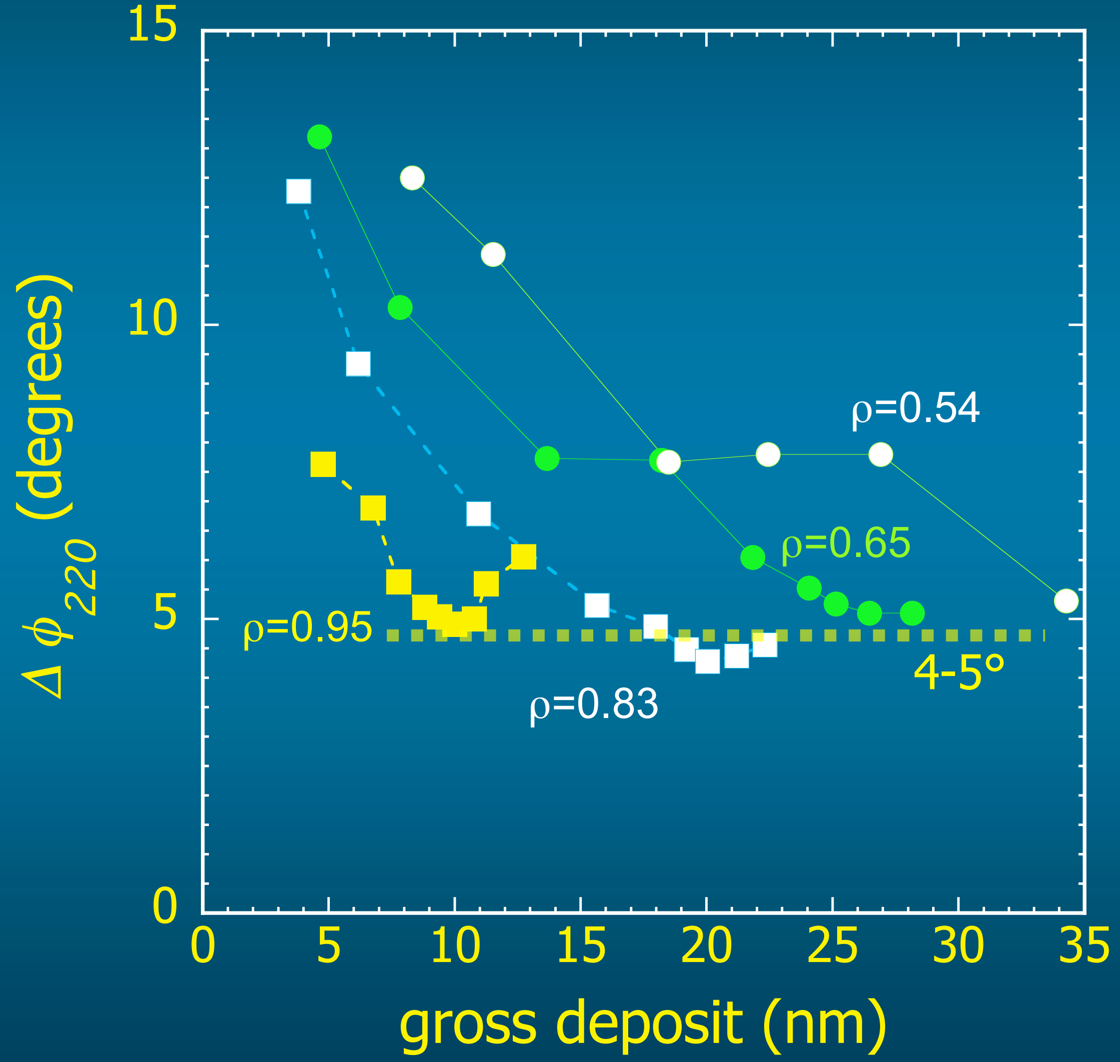
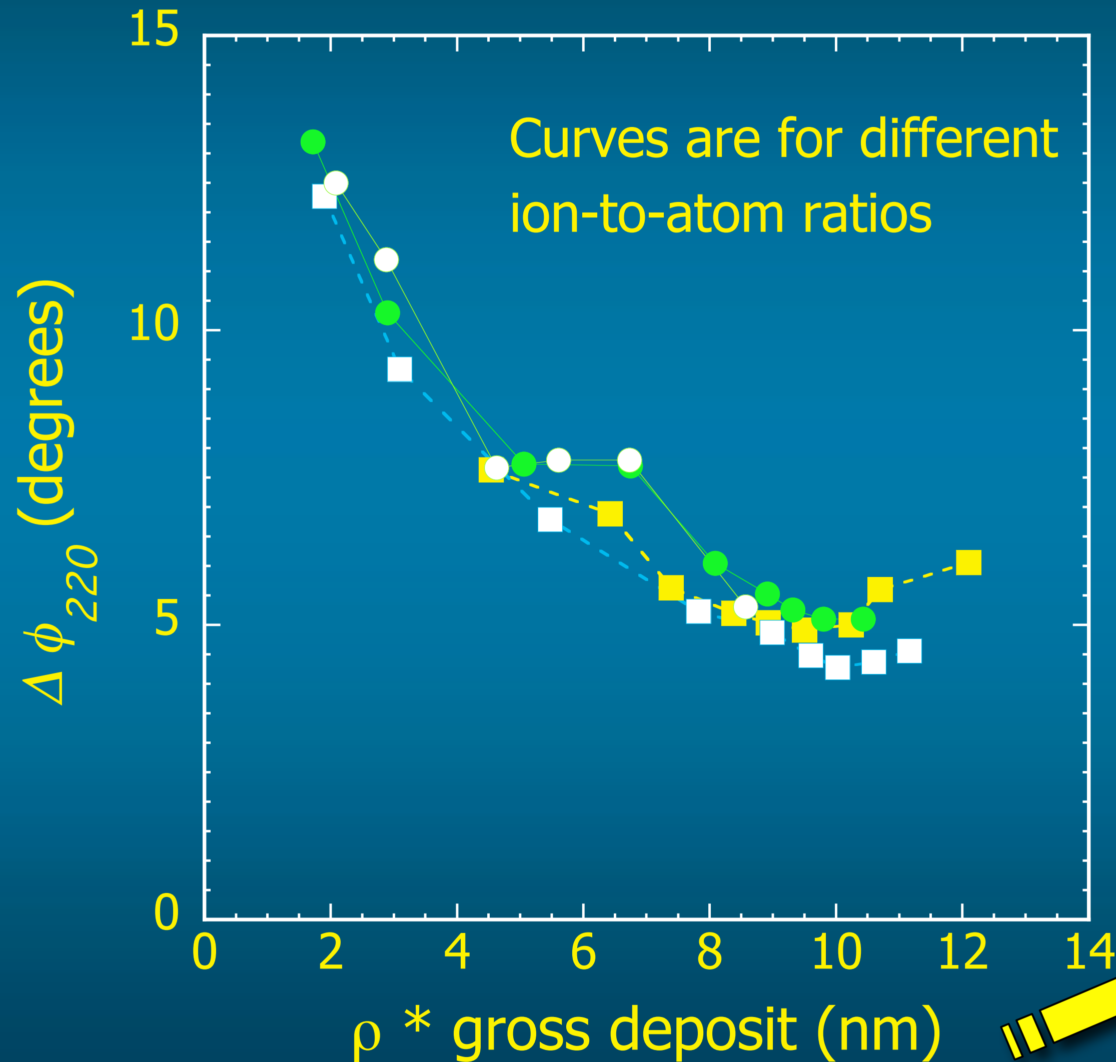
IBAD-MgO texture depends on ratio *and* total deposit thickness



- Data shown for IBAD-MgO on metal tape with Y_2O_3 nucleation layer
- XRD measures the epi layer on top
- Best texture can be attained in a wide window for the ion-to-molecule ratio

IBAD-MgO texture evolution scales with total ion beam fluence

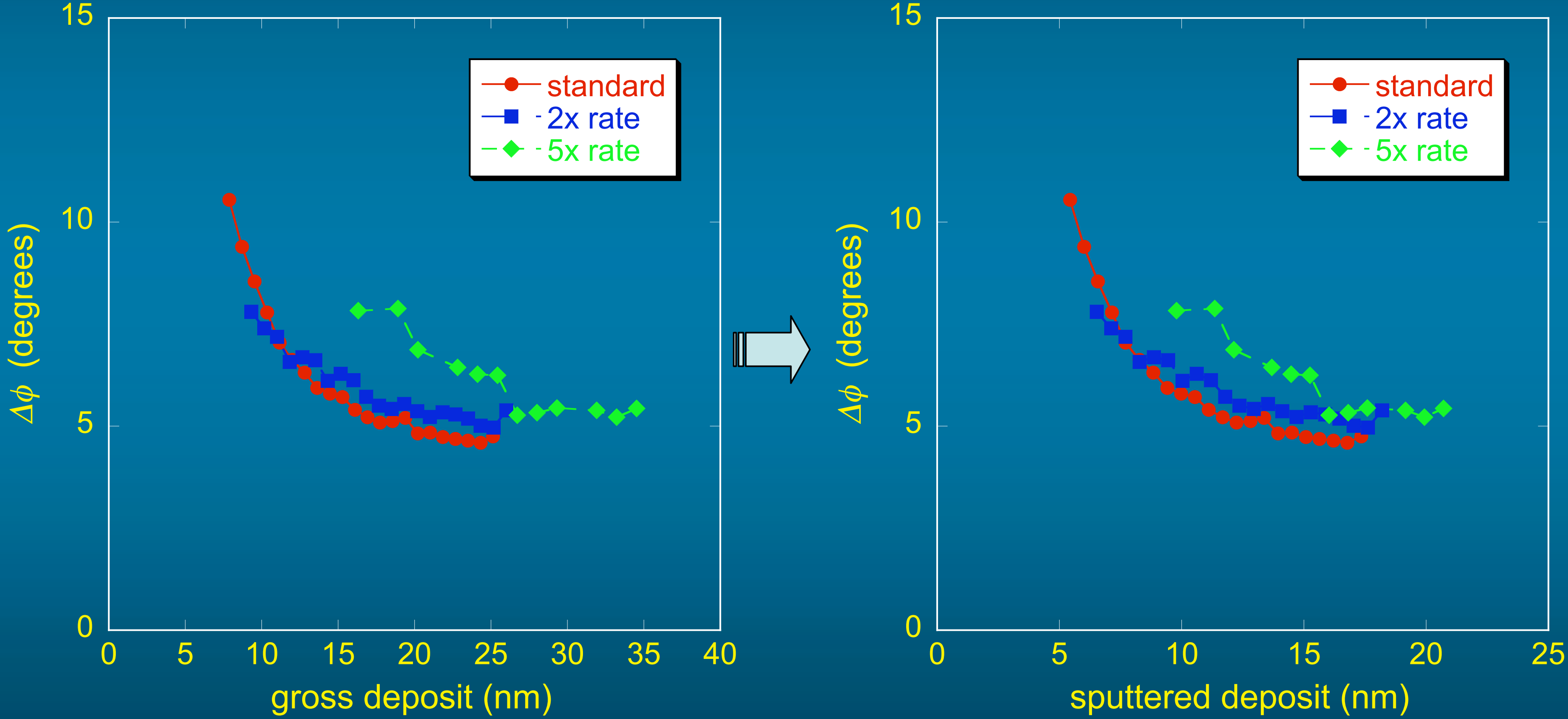


Speed of texture evolution is determined by ion beam fluence

Amount of ion sputtered material

Scaling vs Deposition rate and ion beam current

- Texture evolution also scales with deposition rate
- Increased both deposition rate and ion current; similar *r*



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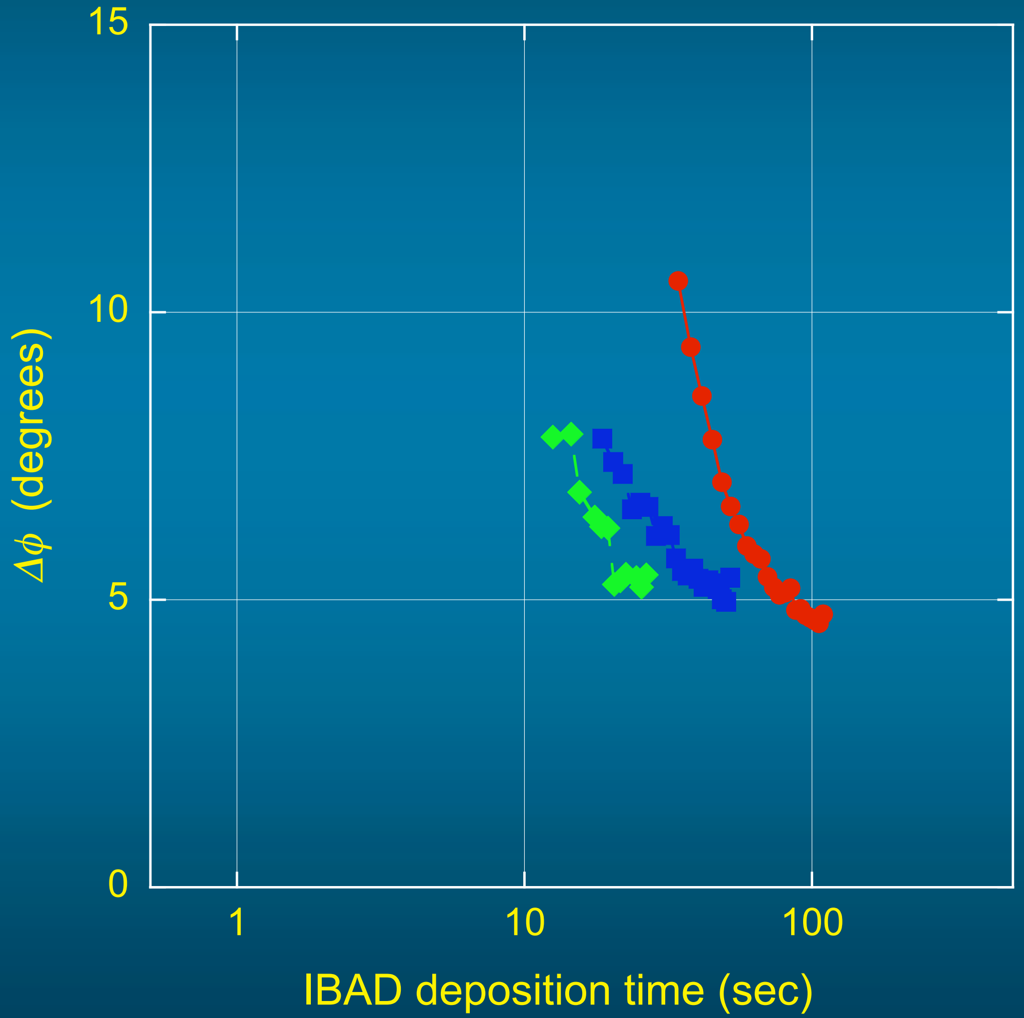
How fast can one texture MgO?



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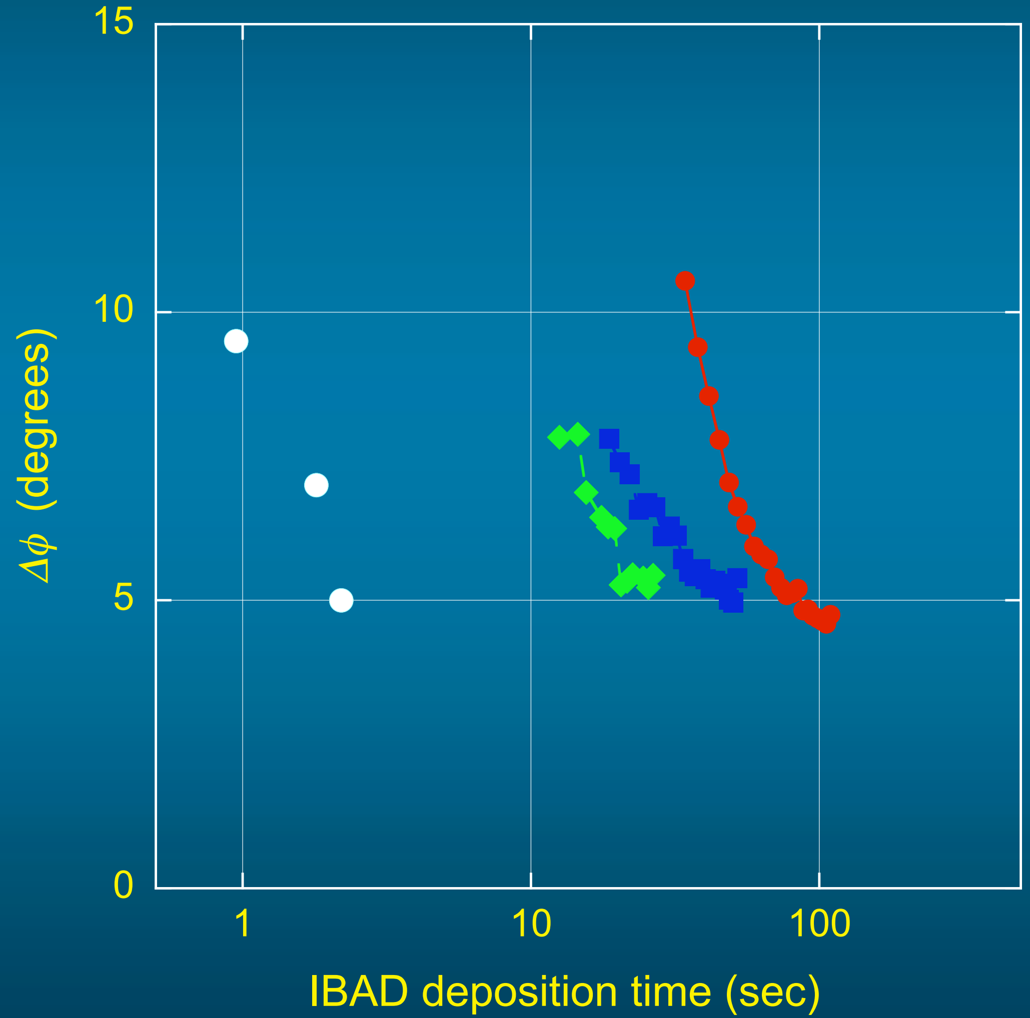

- **Demonstrated high-rate IBAD:**

Time (sec)	Δφ (degrees)
2.2	5.0
1.8	7.1
0.95	9.5

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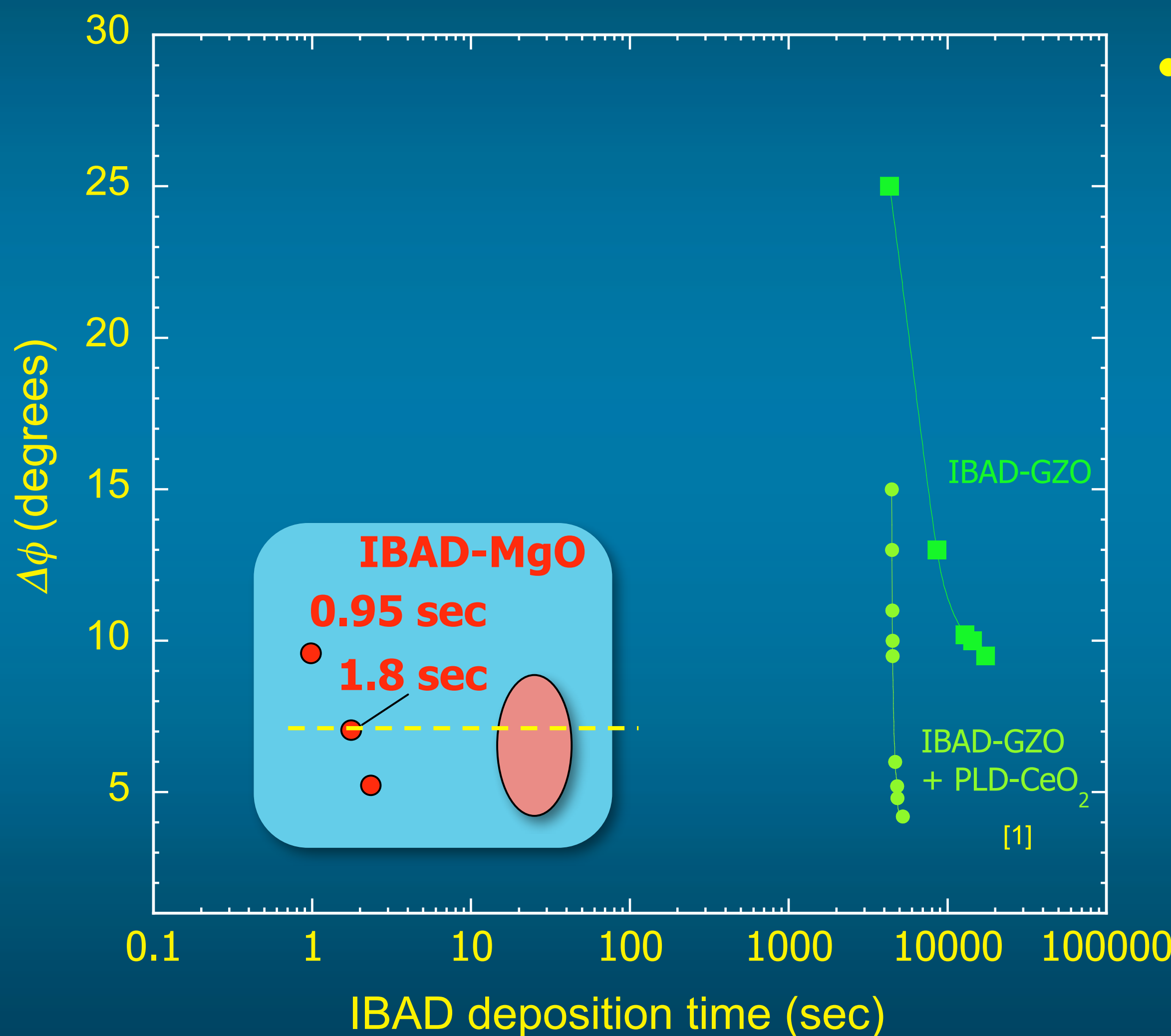
IBAD-MgO and IBAD-GZO comparison



- **540 meters/hr** with 9.5° texture in IBAD-MgO:
  - Single pass (14 cm dep zone)
  - Single ion source
  - 1500 V, 500 mA

[1] *Physica C*, **392**, 777, 2003

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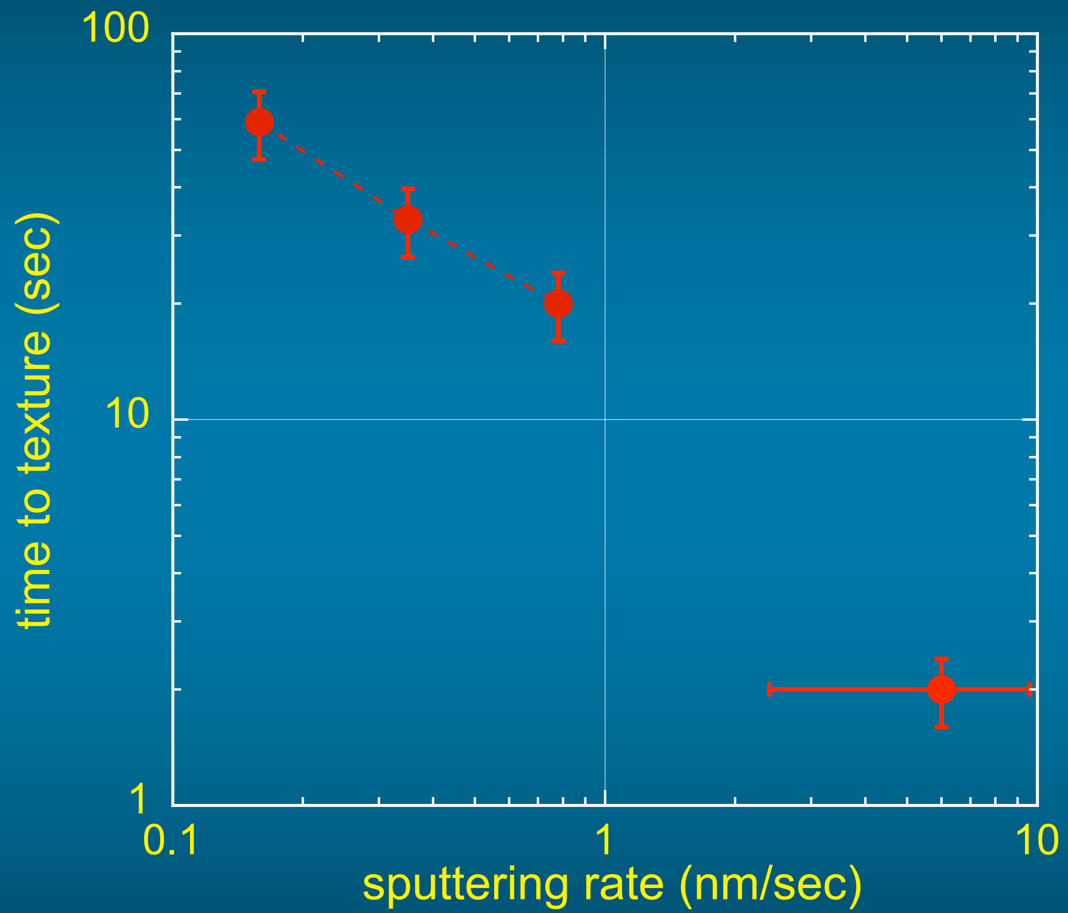
Scaling results



- Data scales well with the total amount of ion beam sputtering or damage

Ion beam current/voltage

750V/56mA **1000V/250mA** **1500V/500mA**

750V/119mA

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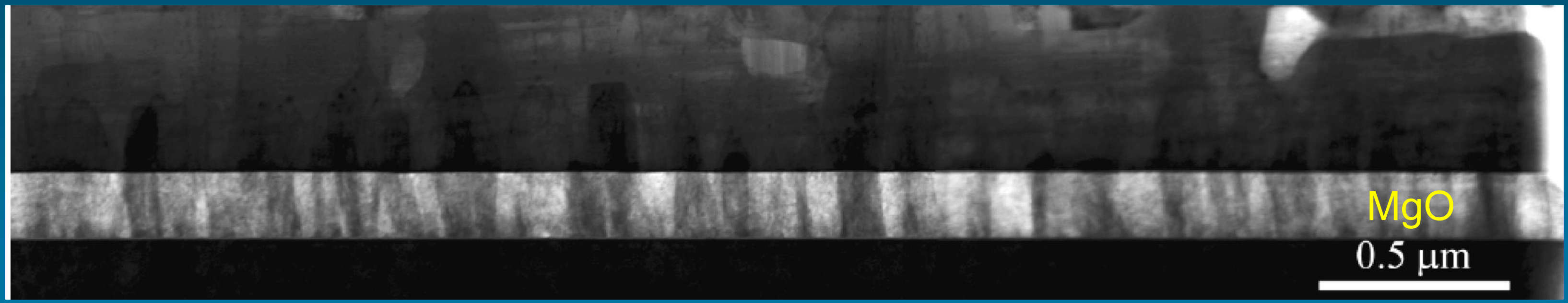
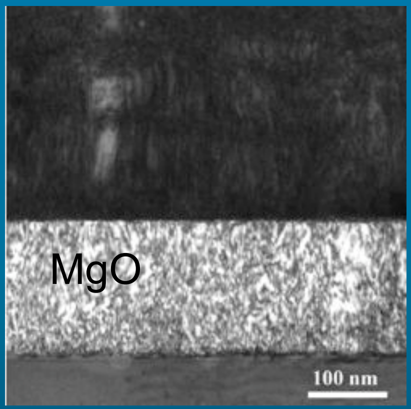
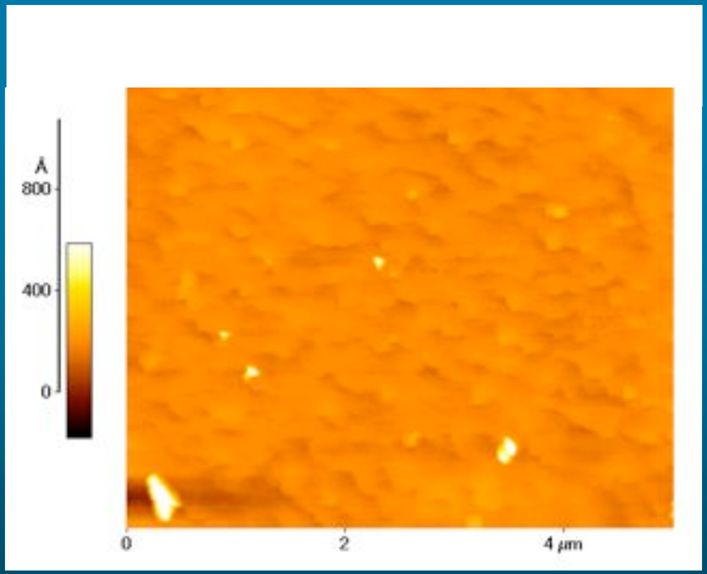
IBAD-MgO Template from LANL



TEM: T. Holesinger



- Thick homoepi MgO (up to 0.5 micron)
- MgO lattice tilted by a few degrees
- Control of tilting exhibited by changing deposition conditions



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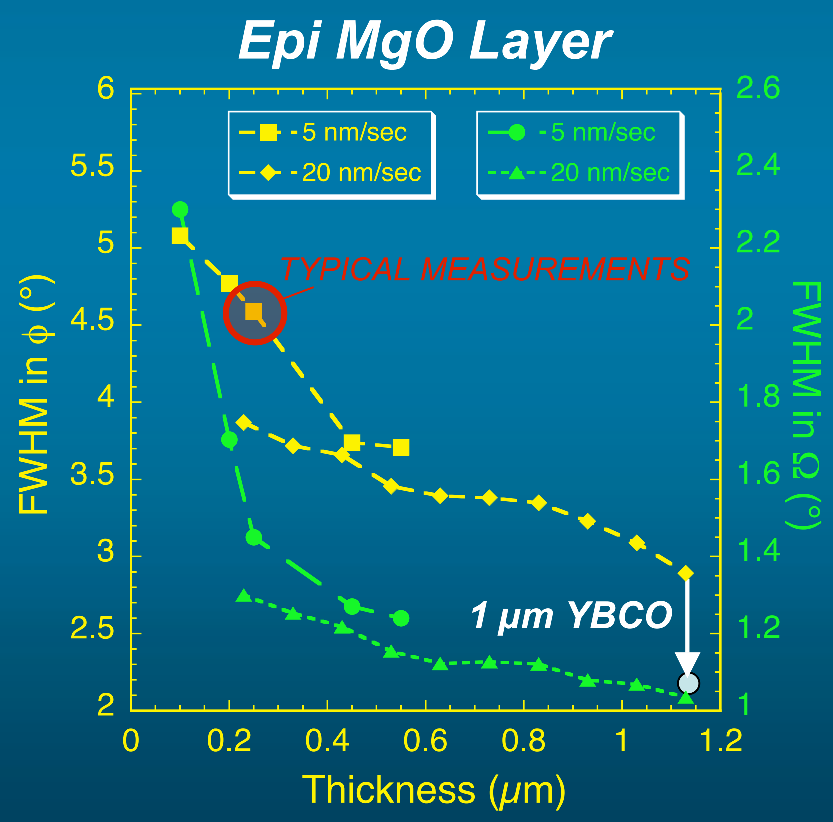
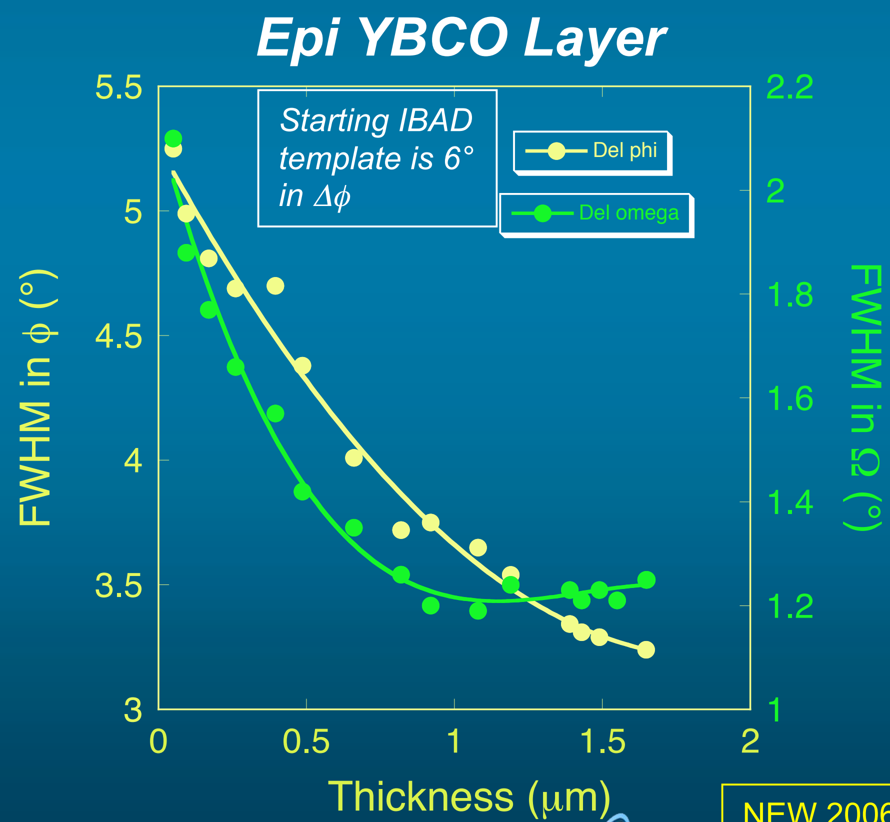
Improvement in texture with thickness

- In-plane and out-of-plane texture improves with thickness of the film and for deposition of subsequent layers
- For thick (1 μm) homoepi MgO layers $\Delta\phi$ can be as low as 3° and YBCO typically improves further by 1–3°; best measured YBCO 1.9° in $\Delta\phi$





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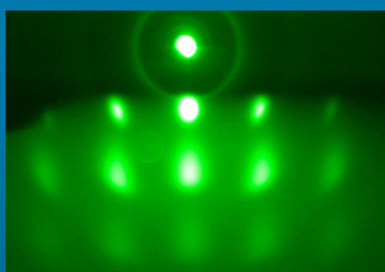
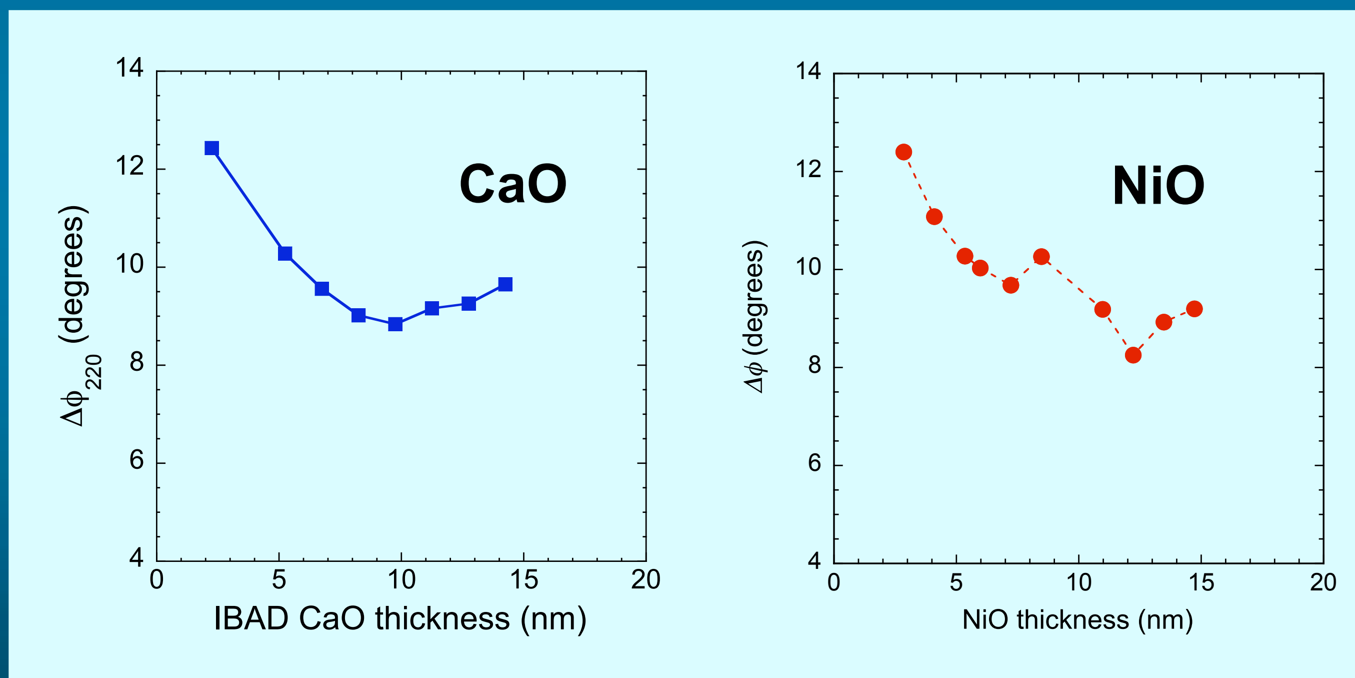
Some other rocksalt materials have similar texture evolution in IBAD

- Demonstrated other IBAD materials: TiN, CaO, NiO, etc.
  - Layers deposited reactively
  - Appear to have similar type of fast texture evolution as MgO





TiN previously reported also by Hühne et al, APL 2004

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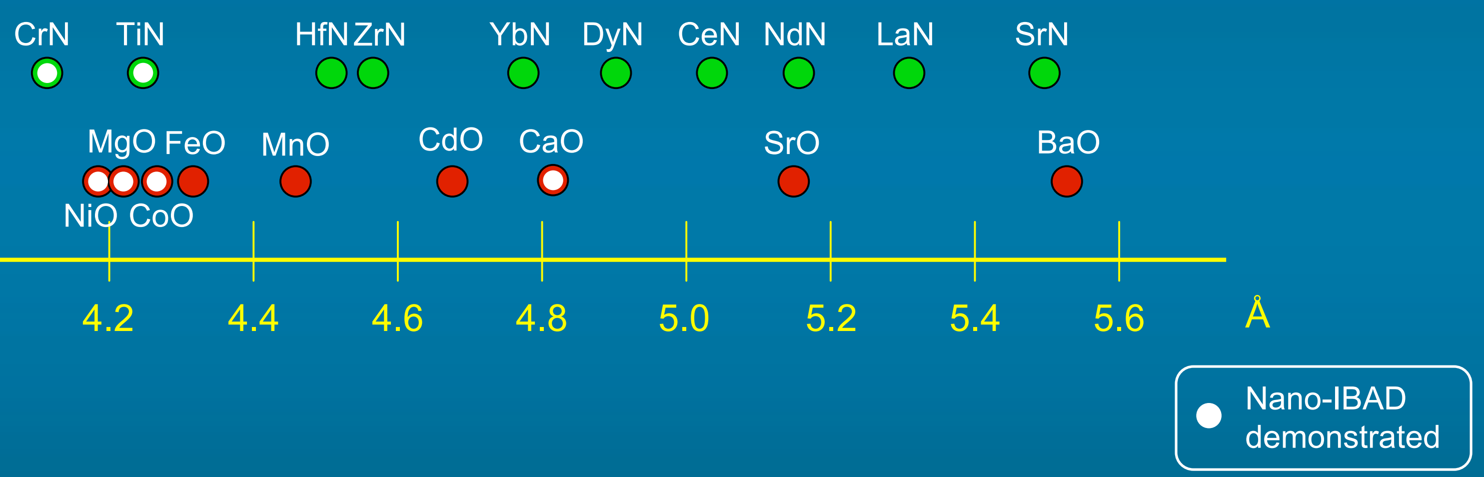
Rocksalt materials for IBAD texturing

- What would make a difference (for Coated Conductors)?
  - If the texture or other structural property is better
  - Lattice parameter tuning



  - Desired physical property: resistivity, dielectric constant
    - Many of the nitrides are conducting

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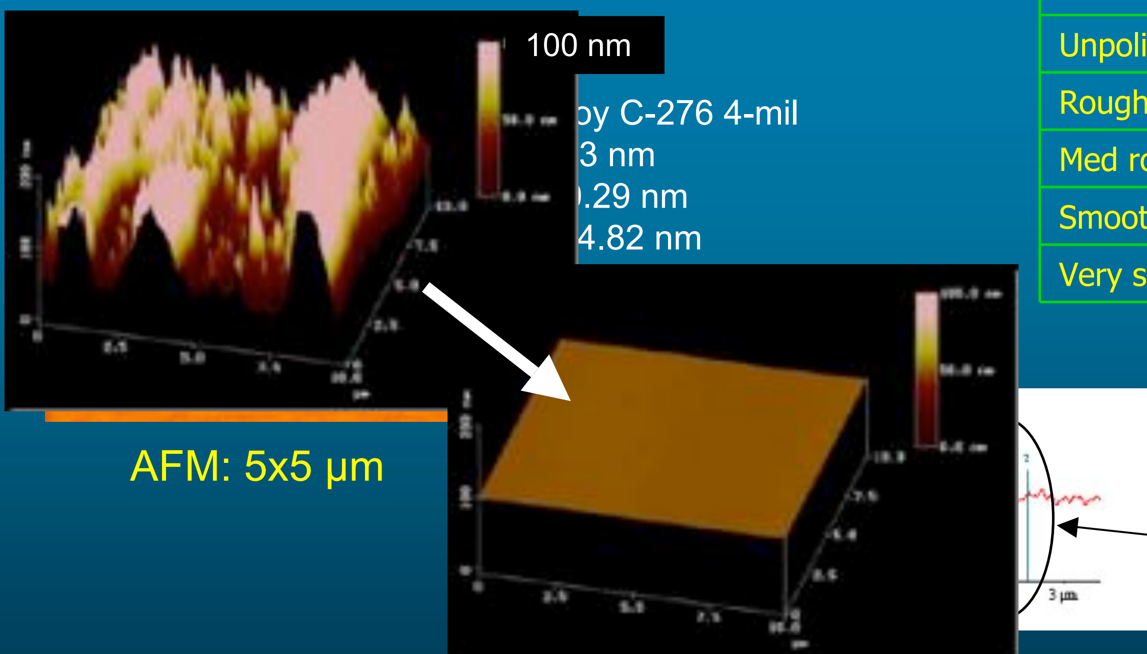
Substrate Preparation Development

- LANL developed the electropolishing process for Hastelloy C-276; achieved atomic-level smoothness
- EP process works well for a specific alloy; not easily transferable to many other alloys
- We are exploring other methods for IBAD tape finishing:
  - Sol-gel deposition to smooth and deposit first nucleation layer for IBAD simultaneously
  - Mechanical polishing of alloys

Mechanical polishing for substrate preparation

- MIPOX Nihon Micro Coating specializes in leading edge technology for surface control
- MIPOX performs high-end mechanical polishing with sub–nanometer surface smoothness
- MIPOX polished LANL’s substrate alloy tapes down to < 0.5 nm RMS roughness in 10 cm long samples (tape not moving)

Hastelloy C-276	Ra	RMS
Unpolished	36 nm	48 nm
Rough polish	3.5 nm	4.9 nm
Med rough polish	1.8 nm	2.4 nm
Smooth polish	0.9 nm	1.3 nm
Very smooth polish	0.2 nm	0.3 nm



AFM: 5x5 µm

2 Å groove

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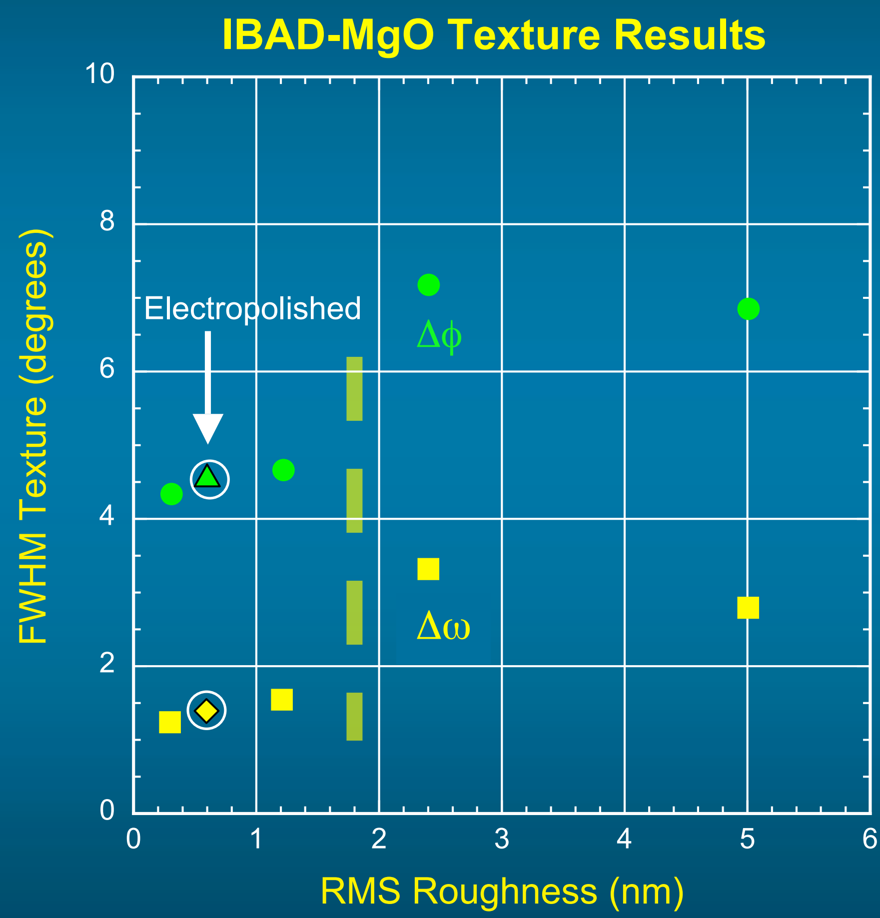
IBAD results on mechanically polished tapes

MIPOX

- IBAD-MgO textured layers were grown at LANL on a series of polished samples (all under the same conditions, 250 nm epi)
- Samples mechanically polished by MIPOX with RMS roughness less than 1.5 nm had similar texture results to the LANL electropolished tapes
- There appears to be a degradation in texture above ~2 nm RMS surface roughness for the Hastelloy



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Substrate Preparation Comparison

	Electropolishing	Mechanical Polishing	Sol-gel nucleation layer
RMS Roughness demonstrated	0.5 nm (from 20 nm)	0.3 nm (any substrate)	0.5 nm (from 2 nm)
Starting material	Hastelloy (major tweak)	Any tape (minor tweak)	Any tape
Cost	low	?	low
IBAD ready	Needs nucleation layer	Needs nucleation layer	✔

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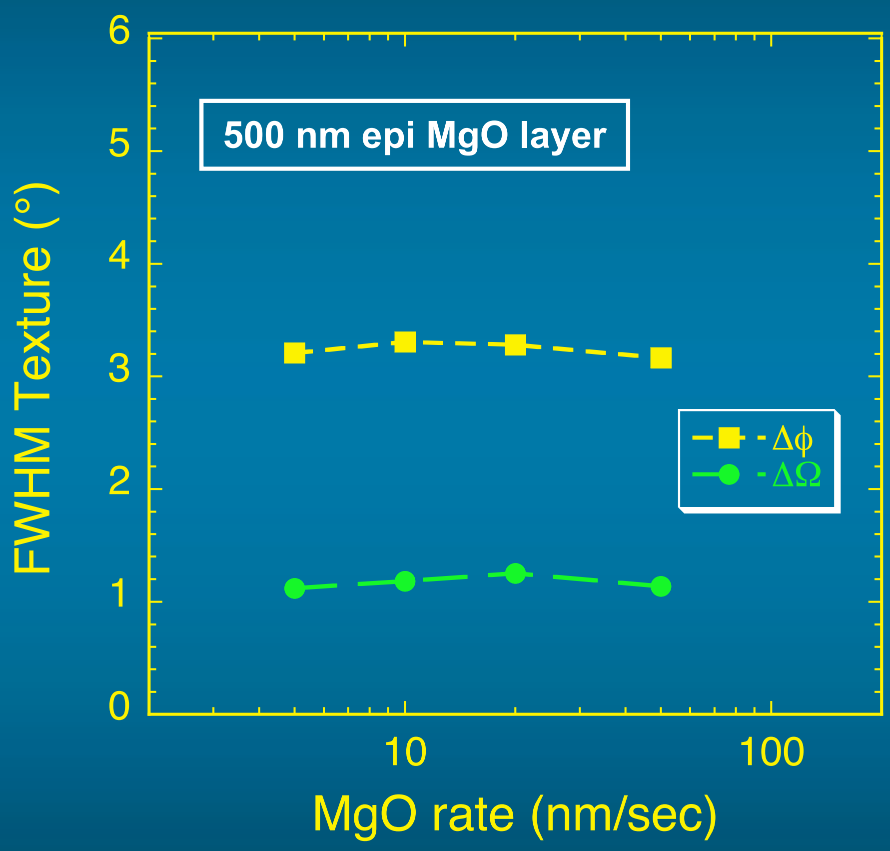
Performance - 2006 Goals for IBAD

- **Goal:** Increase the speed of rate limiting step in template production by one order of magnitude
  - ✔ Deposition of epi-MgO rate increased from 20 Å/sec to 500 Å/sec
- **Goal:** Evaluate Y_2O_3 sol-gel nucleation layer further
  - Success with nucleation layers from Sandia
  - Implemented our own process using reel-to-reel

Epitaxial MgO Deposition Rate

- Increased epi-MgO rate from 2 nm/sec (last year) to 50 nm/sec with no degradation of texture
- Even much higher rates seem possible, but difficult to implement in laboratory setup



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Speed of template production

For realistic manufacturing costs need about 1 km/hr cm-equivalent

	Electro-polishing	Nucl. Layer (YO)	IBAD-MgO	Epi-MgO
Demonstrated speed (LANL) km/hr	0.04	0.015	0.27	0.004
Lab capable speed (extrapolated) km/hr	0.2	0.2	1	0.015
IndustriaL scale up to 1 km/hr	✔	✔	✔	?

Speed of template production

For realistic manufacturing costs need about 1 km/hr cm-equivalent

	Electro-polishing	Nucl. Layer (YO)	IBAD-MgO	Epi-MgO
Demonstrated speed (LANL) km/hr	0.04	0.015	0.54	0.05
Lab capable speed (extrapolated) km/hr	0.2	0.2	1	0.2
IndustriaL scale up to 1 km/hr	✔	✔	✔	✔

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- **Goal:** Increase the speed of rate limiting step in template production by one order of magnitude
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LANL reel-to-reel sol-gel nucleation layer deposition

Process developed in collaboration with Paul Clem at Sandia

- Optimizing process for film smoothness and best IBAD texture
- Achieved: 4.8° in-plane and 1.3° out-of-plane in IBAD-MgO (250 nm epi)
- RMS roughness (5x5 μm):

Hastelloy C-276	RMS bare	RMS with sol-gel Y_2O_3
Unpolished	15 nm	3 - 8 nm
Electropolished	0.8 nm	0.4 nm

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Results

- IBAD processes scalable to high rates (and high throughputs)
  - Demonstrated 1 – 2 sec deposition time (500 m/hr)
  - Epi-MgO deposition scalable to > 50 nm/sec
- Thicker epi-MgO templates have better texture: in-plane ~3° and out-of-plane ~1°
- Demonstrated other rocksalt structure materials with similar IBAD texturing: including NiO, CaO
- IBAD templates have been applied to coated conductors with:
  - 4° in-plane texture on the MgO
  - 2° in-plane texture on the YBCO (PLD)
  - ≤ 1° out-of-plane on the YBCO (PLD)
  - Over 500 A/cm-width demonstrated by PLD-YBCO (LANL), 250 A/cm (MetOx), 200 A/cm (AMSC)

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Research Integration

- Substrate preparation
  - Sandia National Laboratories: sol-gel Y_2O_3
  - NREL: electrodeposited Y_2O_3
  - MIPOX: mechanically polished metal tapes
  - SuperPower and AMSC: electropolishing of metal tapes
- IBAD templates for Coated Conductor fabrication
  - This year provided meters of IBAD template tape to AMSC and MetOx; new CRADA with MetOx
  - Provided template tape internally at LANL and to Stanford University

Plans for FY2007

- Experimental technology improvements
  - Develop software automation for Design of Experiments
  - Study quantification of RHEED for texture
- Explore new materials combinations
  - Identify new IBAD Materials
  - Study new nucleation layers for IBAD
  - Study IBAD texturing on YBCO layers
- Develop IBAD templates
  - Examine further the mechanical polish with MIPOX
  - Develop sol-gel nucleation layer further with Sandia

Conclusions

- What are the mechanisms for texture formation?
  - Clues: Texture scales with cumulative radiation damage; but limited at some point
- Which materials can be used in texture formation?
  - Rocksalt materials; perhaps others?
- What is the best IBAD alignment that can be attained?
  - Empirical evidence suggests ~4°
- Can it be further improved?
  - Texture improves with thicker layers (so far achieved 1 – 2°)
- How fast can the texture be formed?
  - IBAD texture can be achieved in 1 sec (demonstrated 540 m/hr)
- Can we engineer the crystal: tune the lattice parameter? Adjust the vicinal angle? Use arbitrary substrate?
  - Yes ^{3}