X-ray and Neutron Microdiffraction

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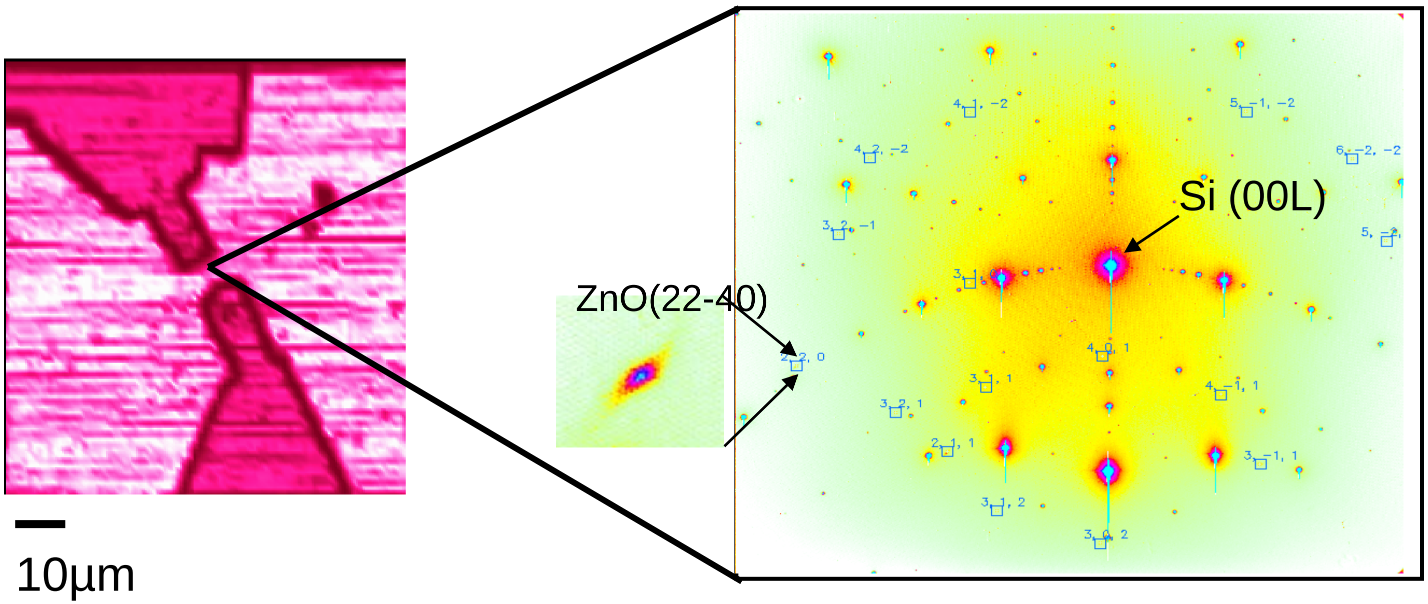


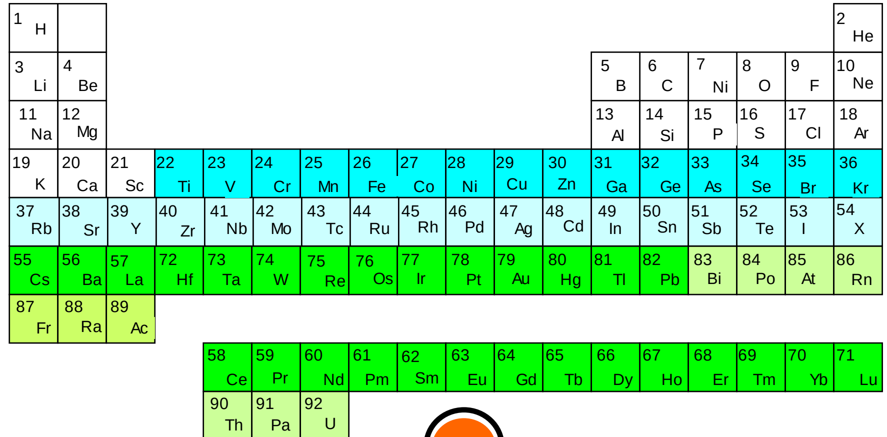
2010 Neutron X-ray Summer School

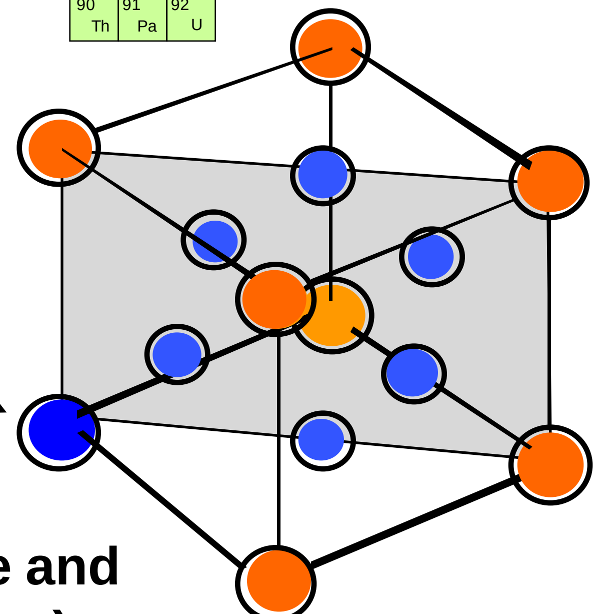
Two words

Spatial Resolution

Materials characterization begins 3 questions

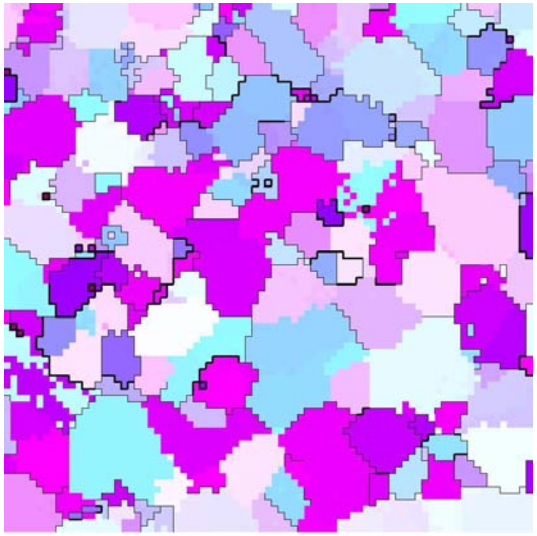
- **What is the elemental composition?**
- **What is the crystal/local structure?**
- **What are the defects?**



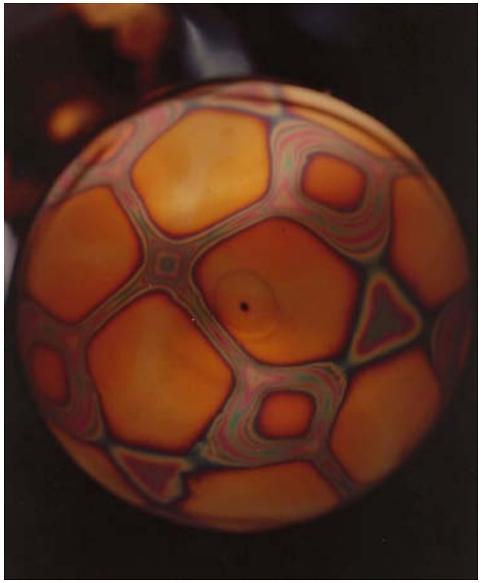


X-rays and neutrons probe structure and defects through scattering (diffraction)

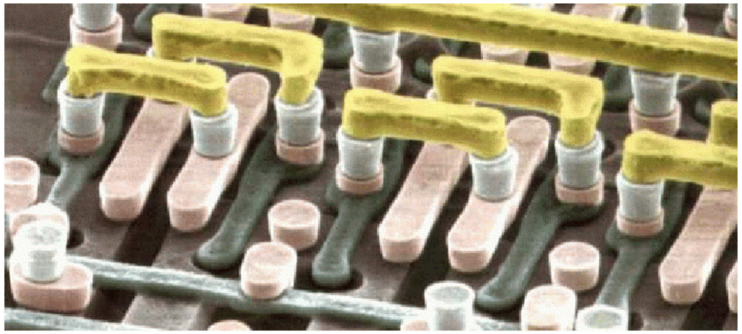
Spatial resolution essential!



- **Most materials** *polycrystalline* **(0.1-50 μm)**
  - **Anisotropic**
  - **Heterogeneous**
  - **Plastic/elastic deformation/ diffusion/ oxidation/**



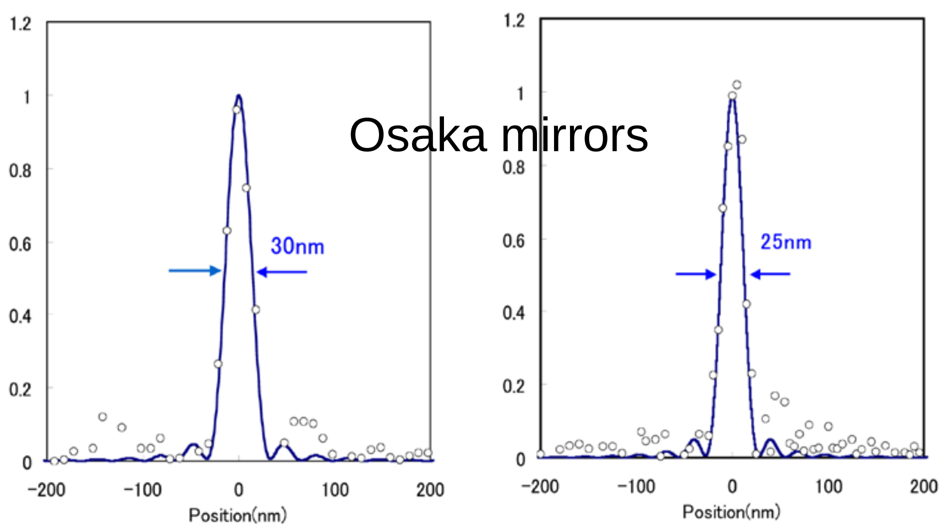
- **Even** *within* **single and "perfect" crystal:**
  - **Strain**
  - **Defects**
  - **Spontaneously organize to reduce energy**



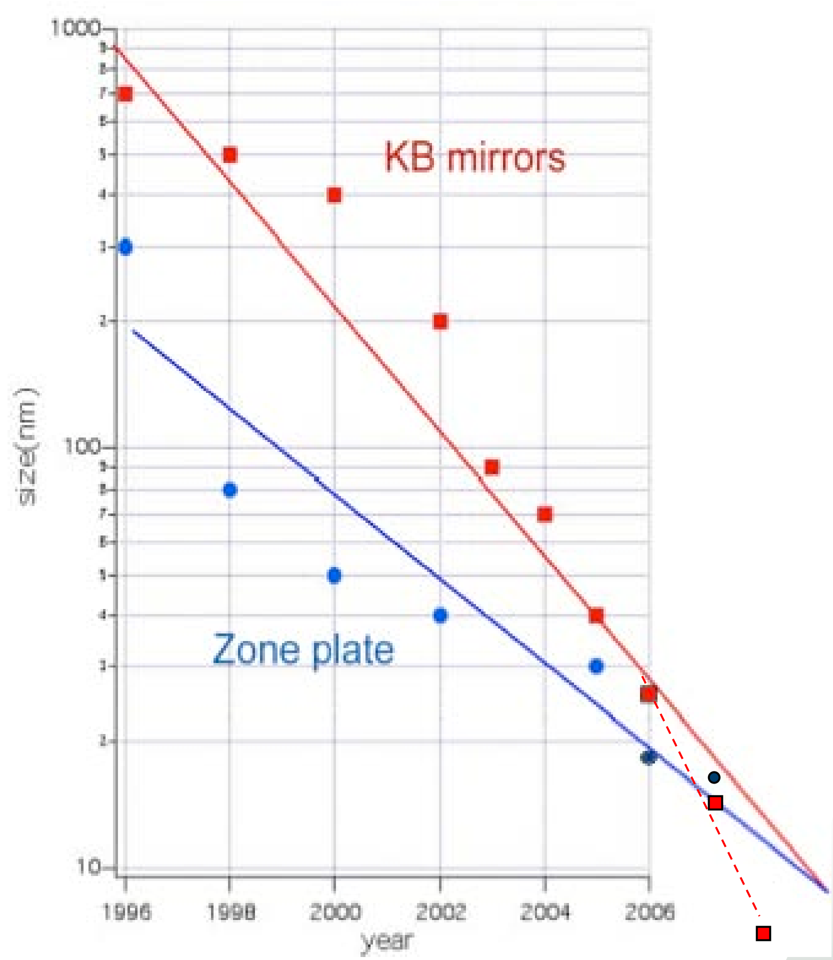
Spatial resolution essential for most advanced energy systems

Optics improving rapidly

- **Both chromatic and achromatic <20 nm**
- **New choices**
  - **Mirrors**
  - **Zone plates**
  - **Laue zone plates**
  - **Compound refractive optics**

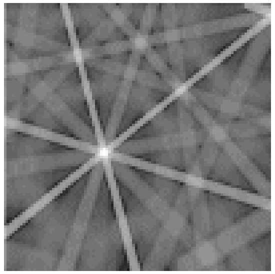


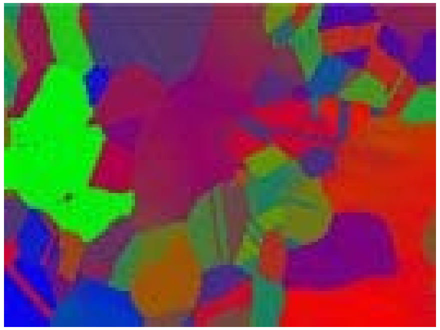
Osaka mirrors



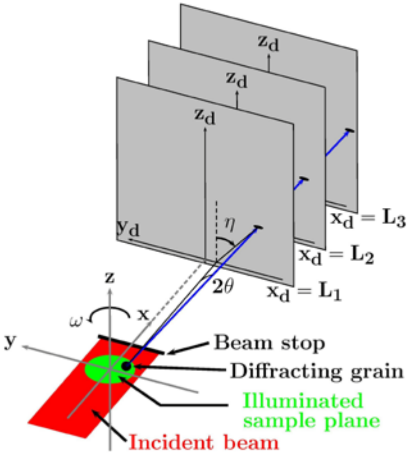
Diffraction mapping emerging area in electron and x-ray microscopy

- EBSD-transformed study of polycrystals
  - Surface phase
  - Surface orientation
  - FiB-3D mesoscale structure

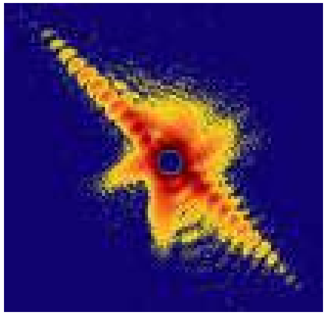




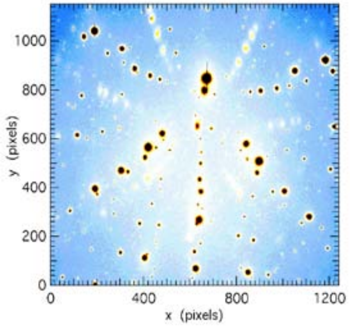
- 4D X-ray microscopy Lienert et al.
  - Time resolved
  - Deep penetration

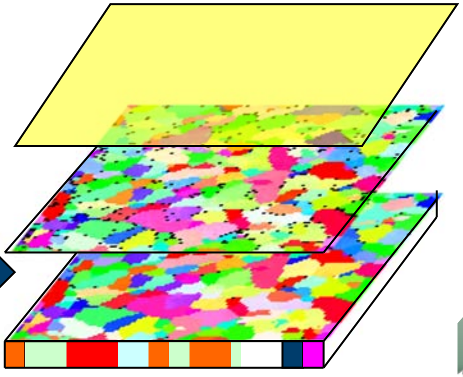


- Coherent X-ray Diffraction (Robinson et al.)
  - Simple structures

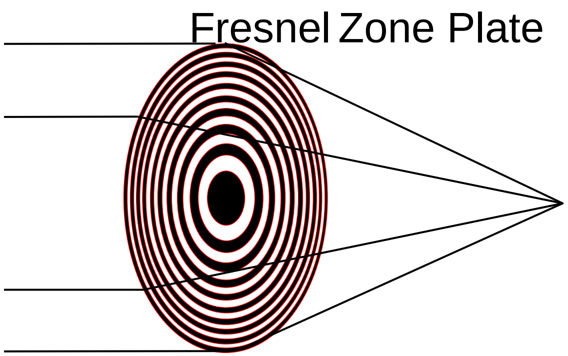


- Polychromatic X-ray microdiffraction
  - Phase/texture/***strain***/
  - ***Nondestructive***
  - ***Submicron***

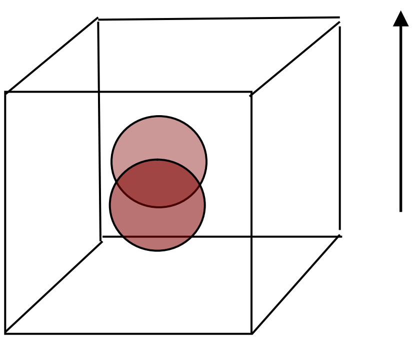




Monochromatic micro crystallography probes simple crystal systems

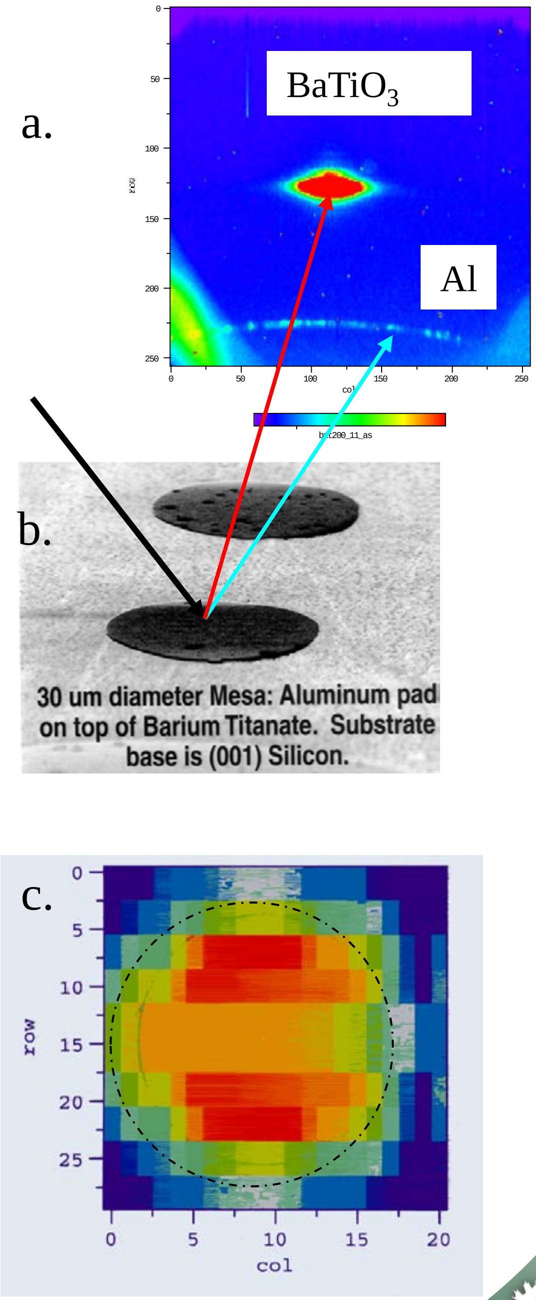


Wide-range of focusing choices

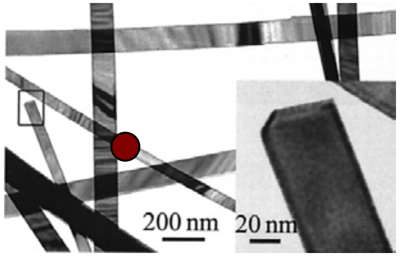


Ferroelectrics ideal samples

Thompson et al. Study domain growth/switching etc.

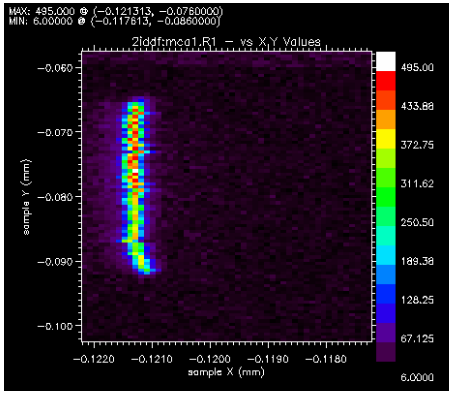


Cai et al. and others study ultra-small nanocrystalline volumes with existing microbeams

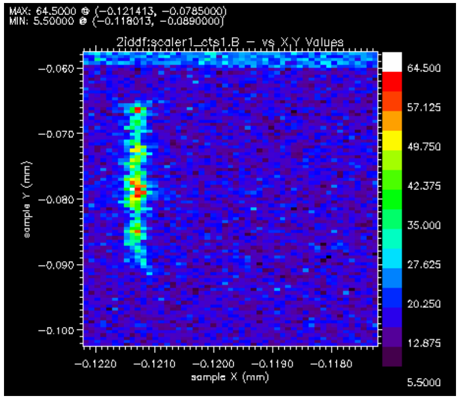


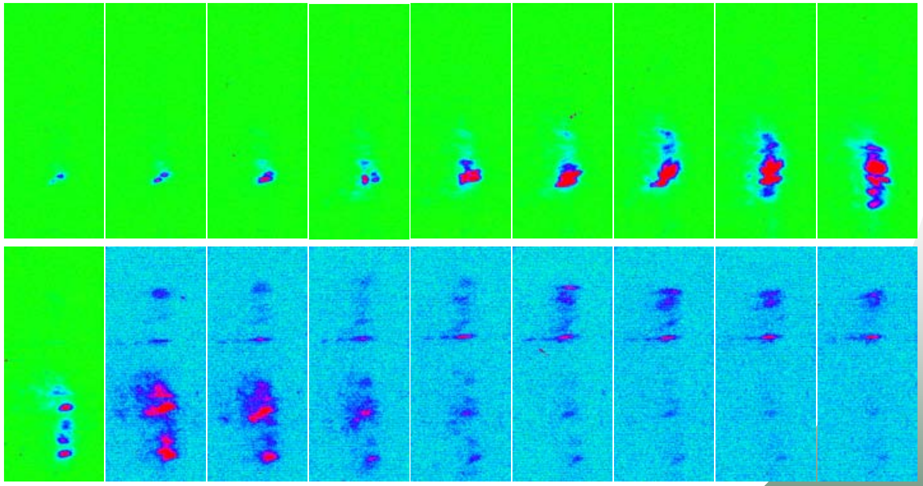
•**150 nm beam resolves crystalline substructure in individual Sn_2O_3 nanobelts**

Fluorescence map

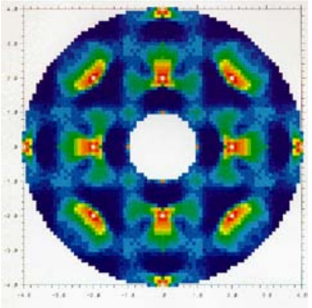


Diffraction map

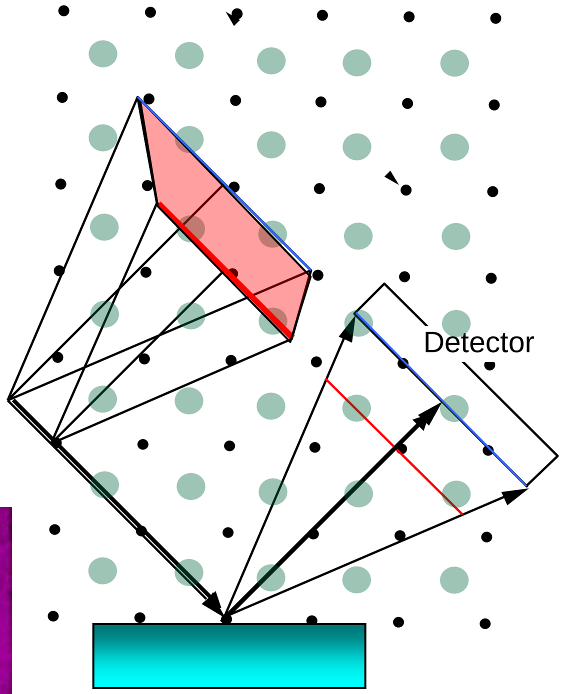


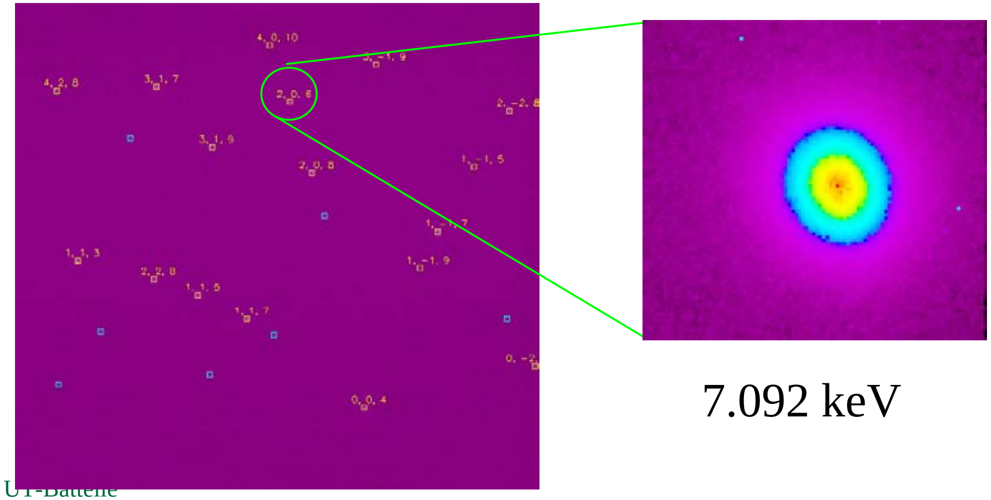


Short-range chemical order heterogeneity magnetically annealed crystal now accessible



- **6 orders of magnitude weaker than Bragg scattering**
- **Area detector integrates over plane in reciprocal space**





7.092 keV

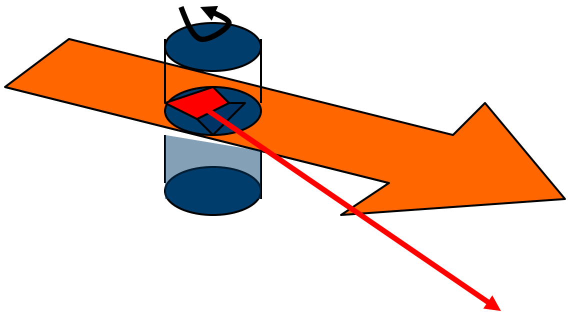
APS *Nanoprobe*- opens new opportunities for spatially resolved

- **Diffraction proposals compelling**
- **Physics of small**
- **Integrated circuit materials**

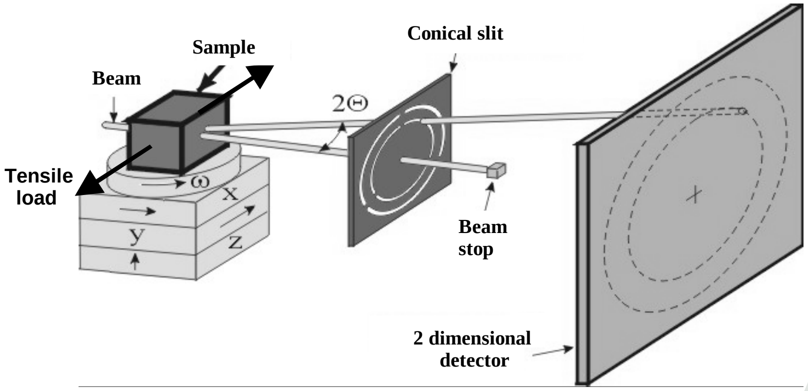
~30 nm target/ <10 nm possible in near future/ NSLSII ~1 nm!

4DXRD Microscope emerging tool for studying mesoscale dynamics-single rotations

- **Singly focused monobeam illuminates numerous grains**
  - **Bragg condition satisfied by single rotation**
  - ***Time resolution! (4D)***



- **Grain outline determined**
  - **Ray tracing**
  - **conical slit**
  - **Back-projection tomography**

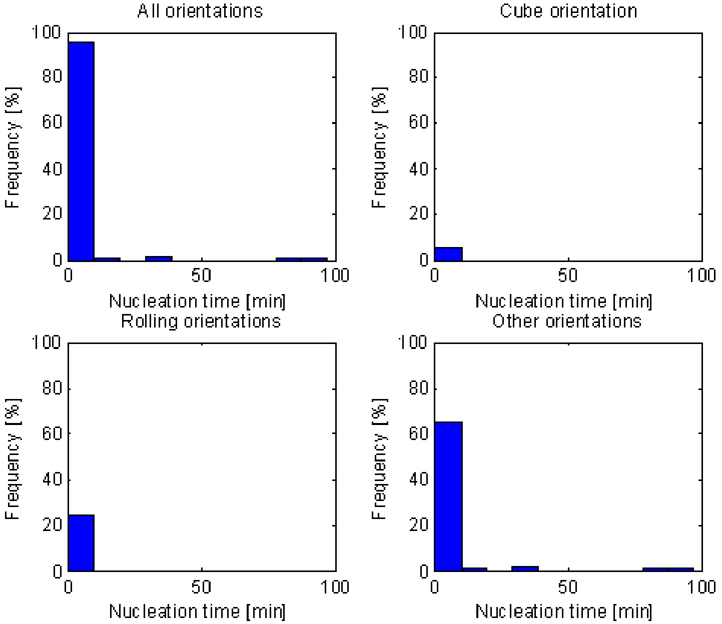


- **E>50 keV allows deep measurements**

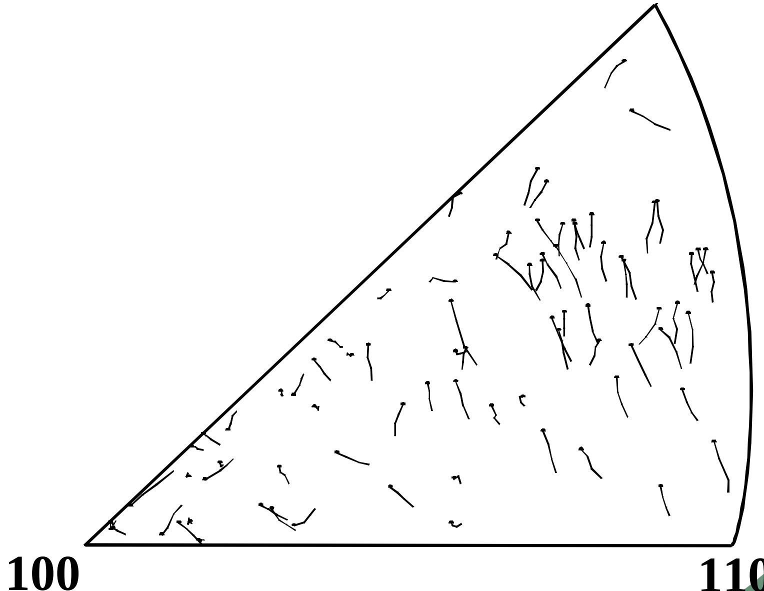
Best with high-energy beams/Beamline 1 at APS

4DXRD Microscope powerful dynamics probe

- **Recrystallization growth individual grains-deep**
  - **E. M. Lauridsen, D. Juul Jensen, U. Lienert and H.F. Poulsen (2000). *Scripta Mater.,* 43, *561-566***

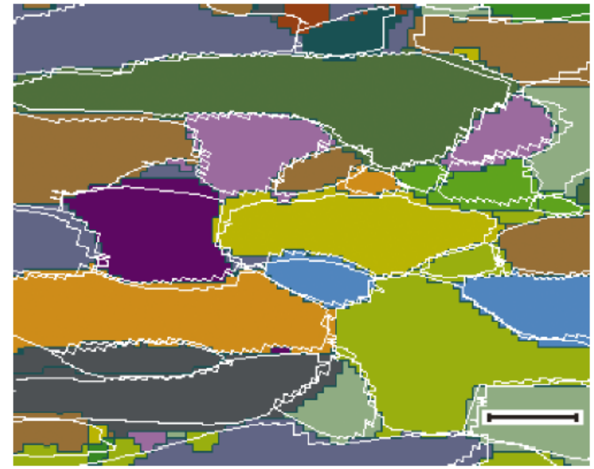


- **Rotations/texture evolution individual grains during deformation**
  - **Tests deformation models**
  - **L. Margulies, G. Winther and H.F. Poulsen, Science** 291, **2392-2394 (2001).**

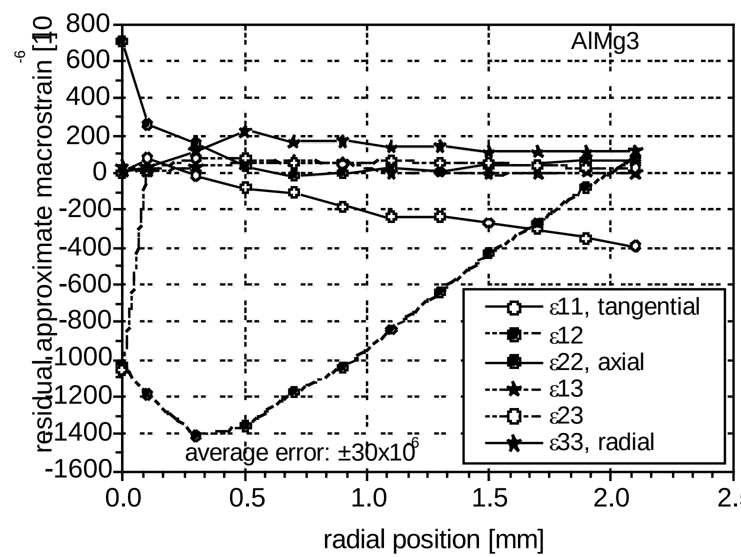


4DXRD Microscope provides additional powerful capabilities

- **Grain boundary mapping in coarse grained materials-5μm**
  - **Poulsen et al. J. Appl. Cryst. 34 751-756 (2001)**



- **Single crystal refinement for polycrystals**
- **Macro/microstrain**



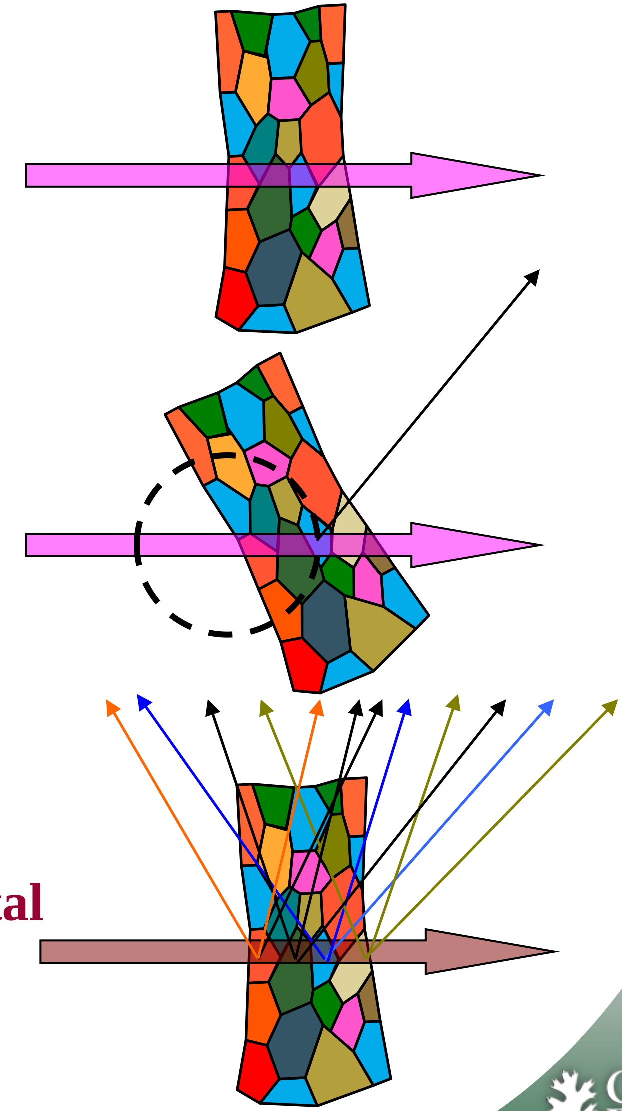
Strain tensor elements in torsion sample

Ideal for neutrons! But needs high-resolution detectors!

Polychromatic simplifies microdiffraction

Solves intrinsic problem with conventional microdiffraction-

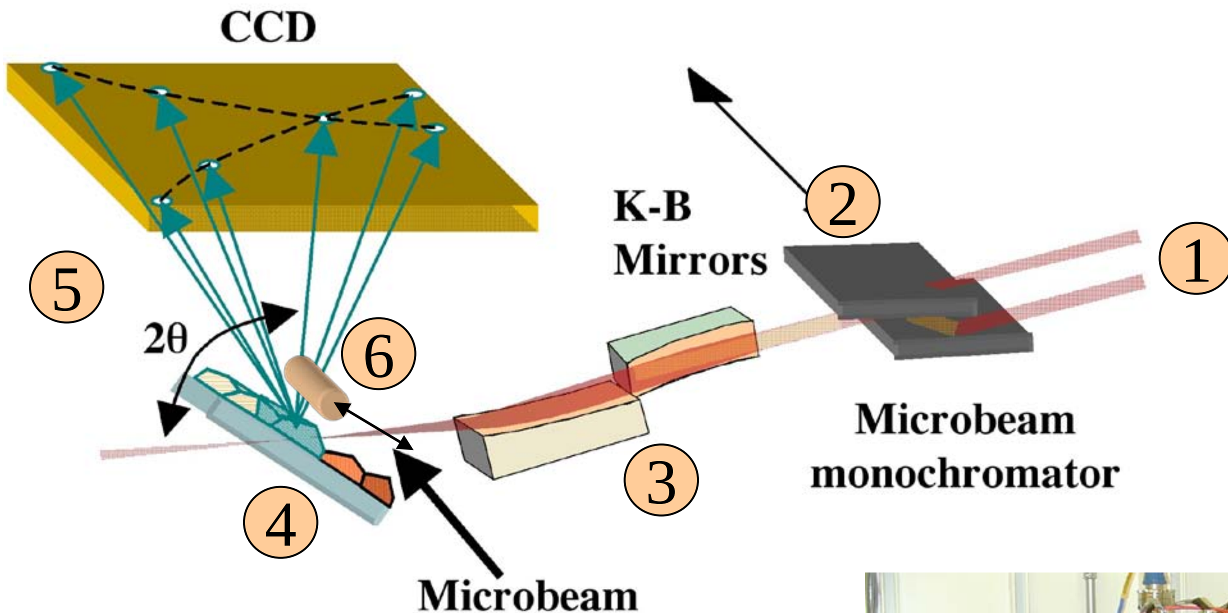
-Sample does not need to be rotated!

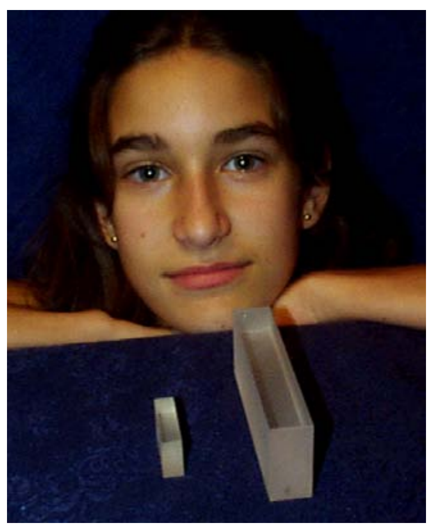


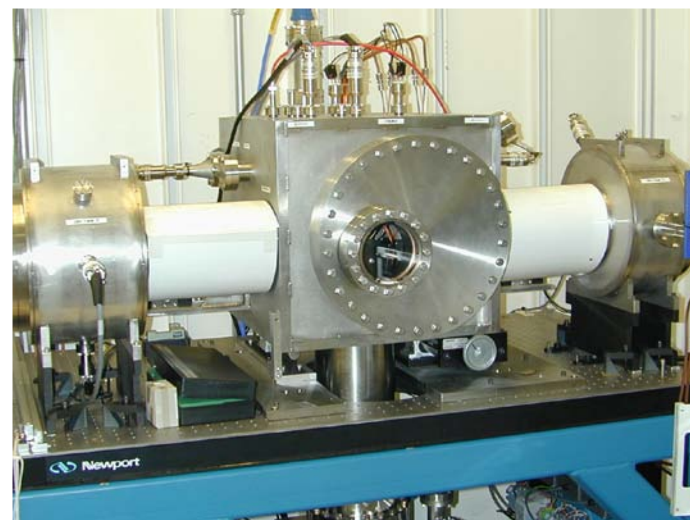
Special software required- Can index polycrystalline samples

3D nondestructive probe of stress/strain/crystal structure!

3-D X-ray Crystal Microscope has 6 key Elements

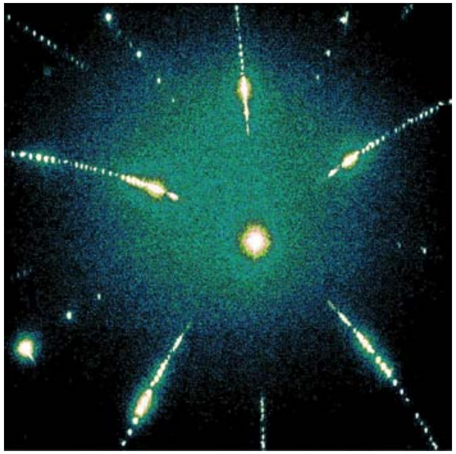




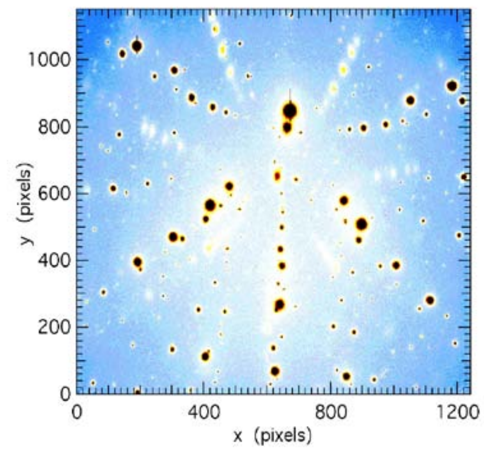


<0.3 x 0.3 x 0.5 μm^3
strain~10^{-4}-10^{-5}

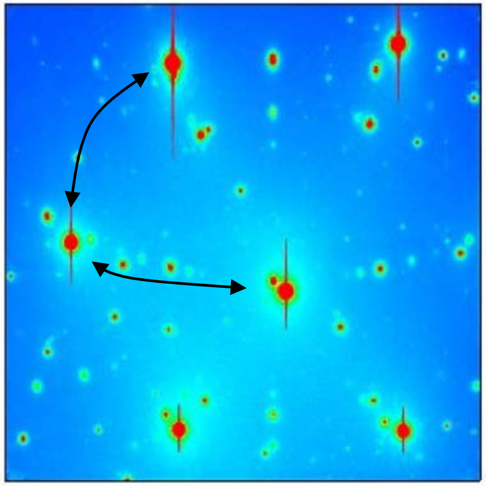
MicroLaue patterns depend on internal structure/orientation



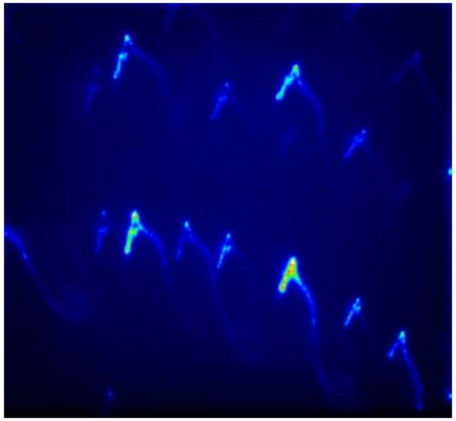
Phase/phase boundaries



Texture (0.01º)/grain boundaries

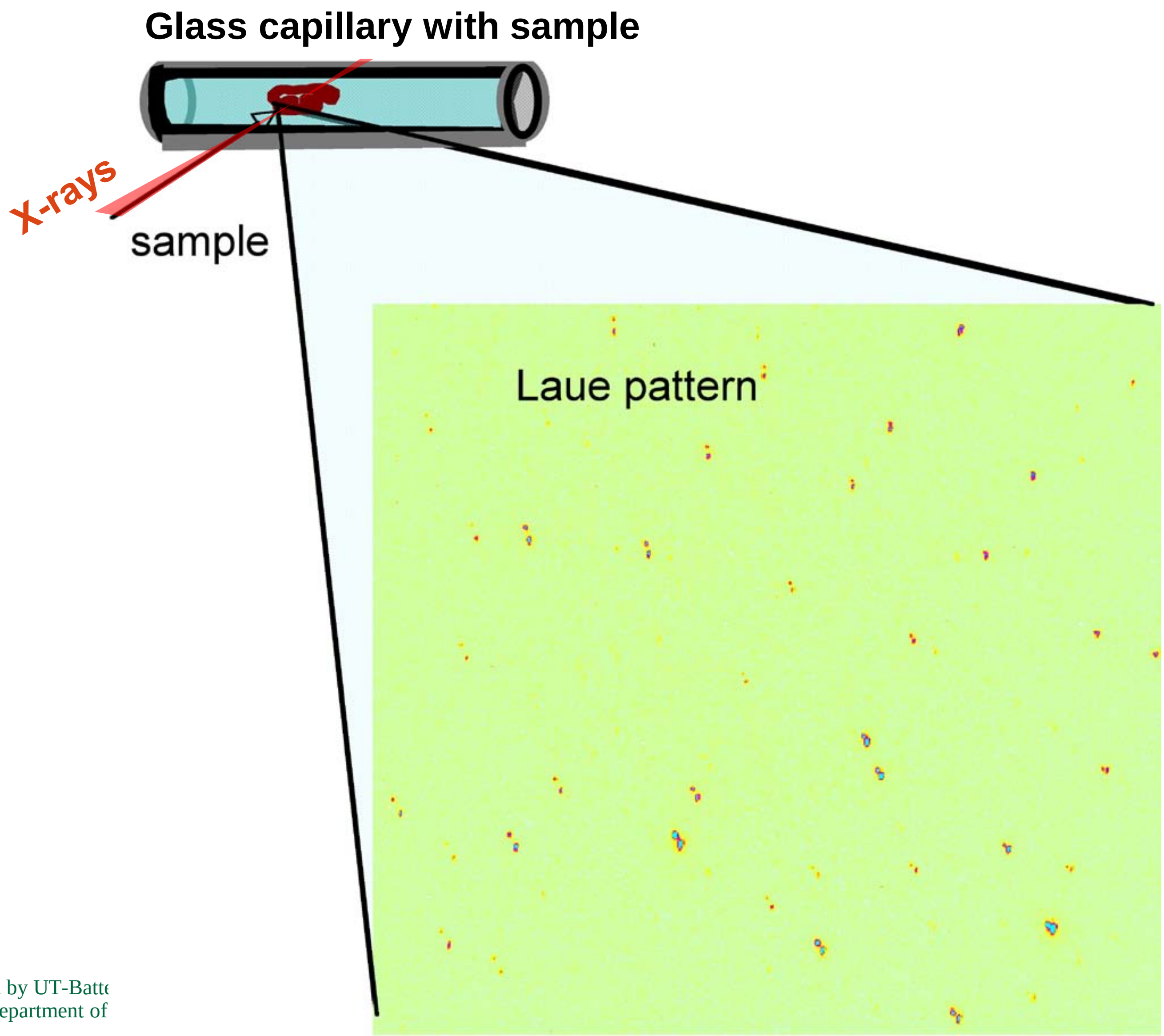


Elastic strain tensor(1 x 10^{-4})



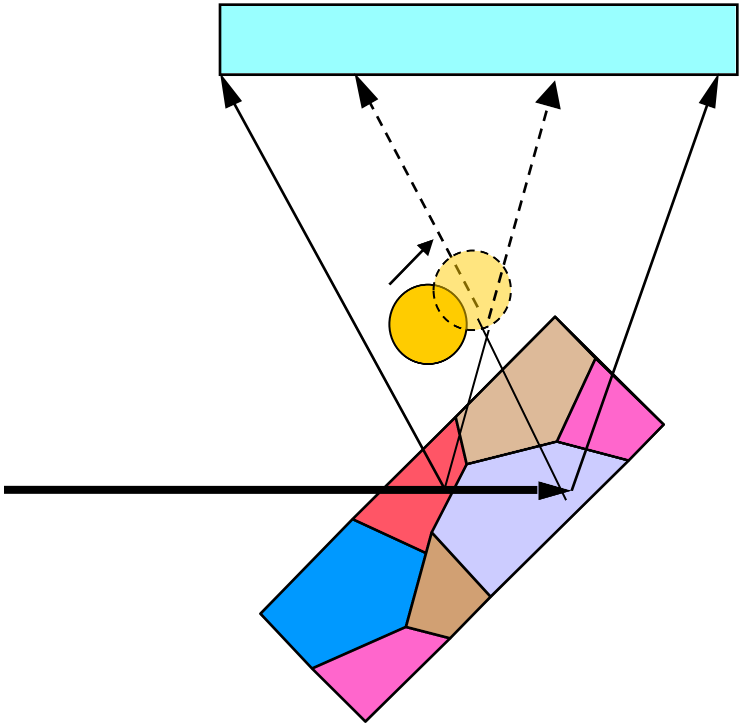
Dislocation Tensor

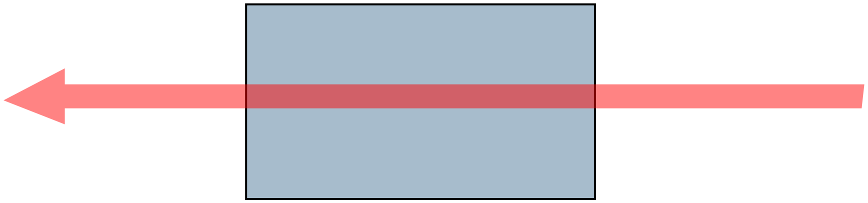
Laue methods essential for some samples



Differential aperture microscopy resolves submicron along incident beam!

- Simplifies data interpretation
- Submicron Z resolution
- Isolates weak diffraction from strong
- First demonstration by Larson et al. on deformed Cu -

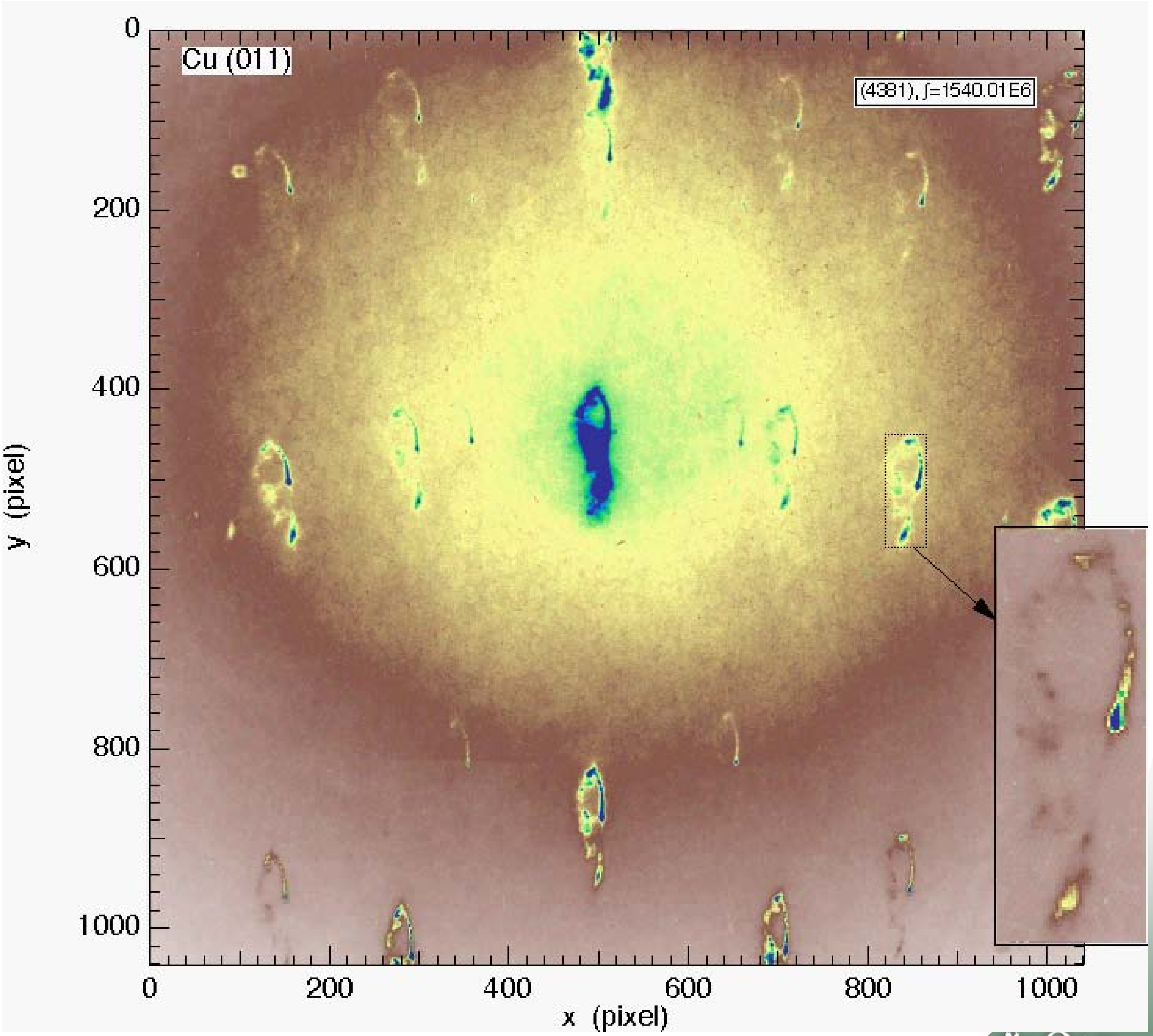




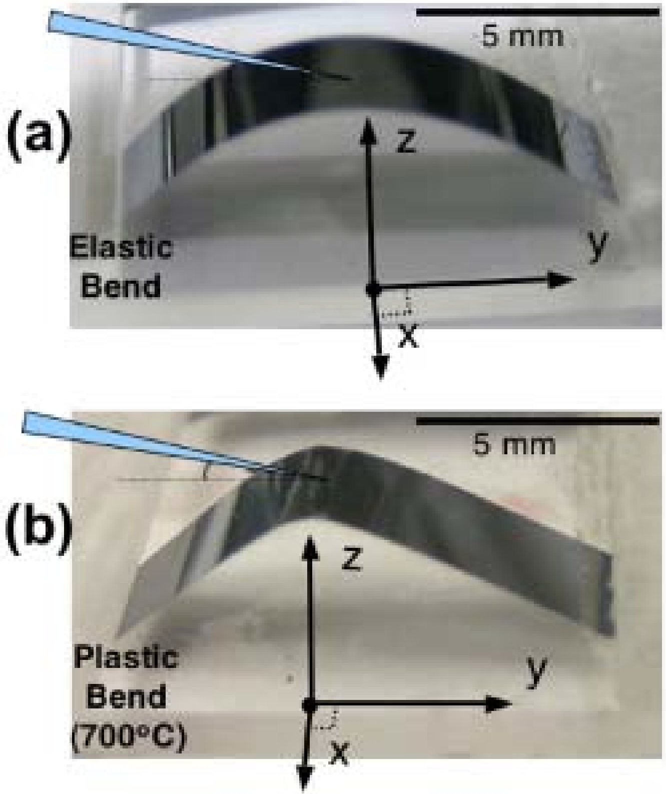
As wire moves its edge cuts through Laue spots

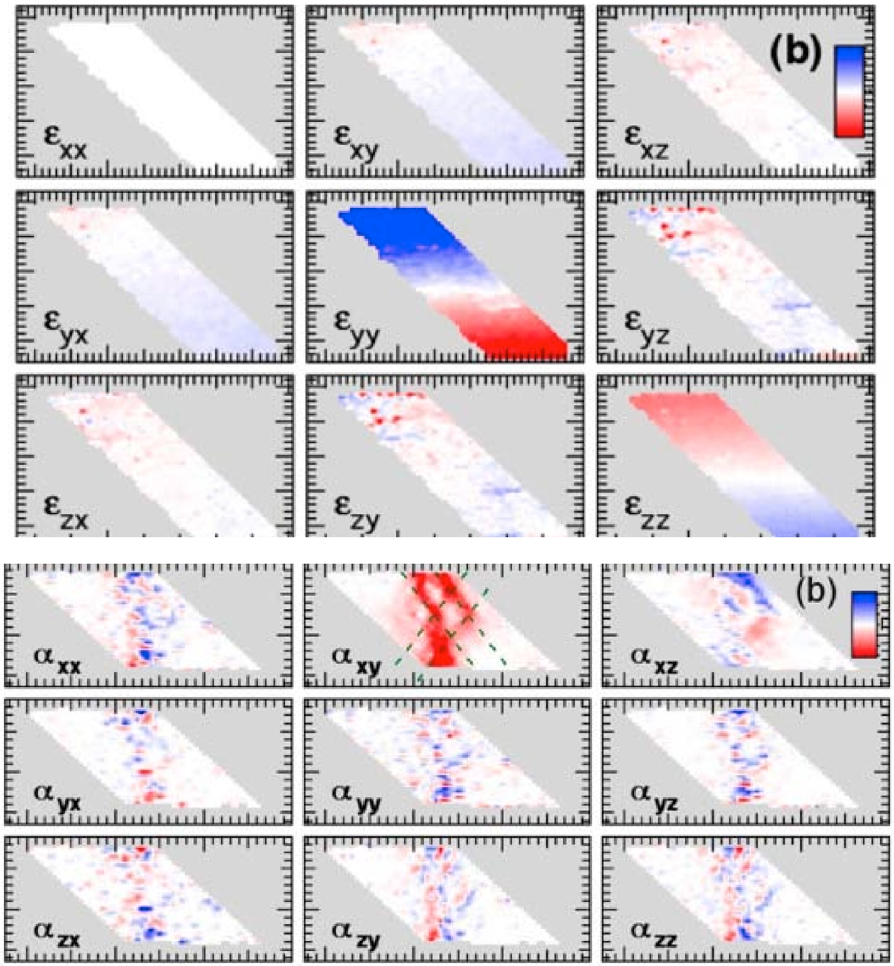
- **Near-surface fluorescence provides moving shadow**

- **Long scans needed for deep penetration**



Measurements of elastic strain tensor *inside* bent single crystal Si illustrate power of DAXM



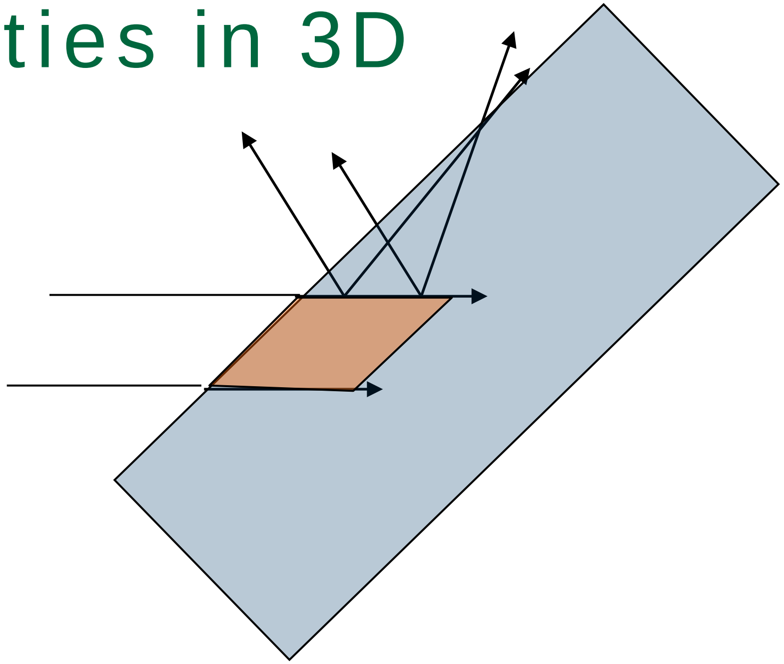


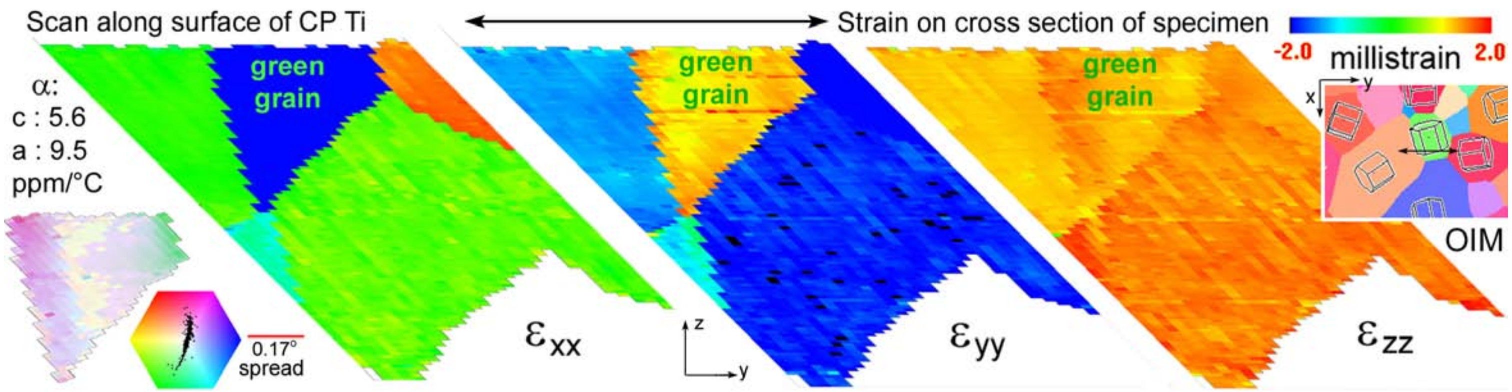
Orientations to 0.001°

Larson et al. J. Eng. Mat. and Tech. 130 021024 (2008) ORR award

Maps crystal properties in 3D

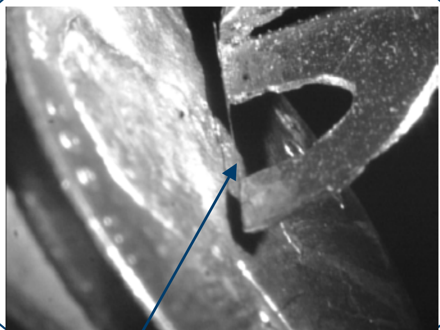
- **Phase**
- **Texture (orientation)**
- **Elastic strain tensor**
- **Nye tensor (deformation)**





T. Bieler et al.

Experimental Hutch 34ID-E at UNICAT, Advance Photon Source

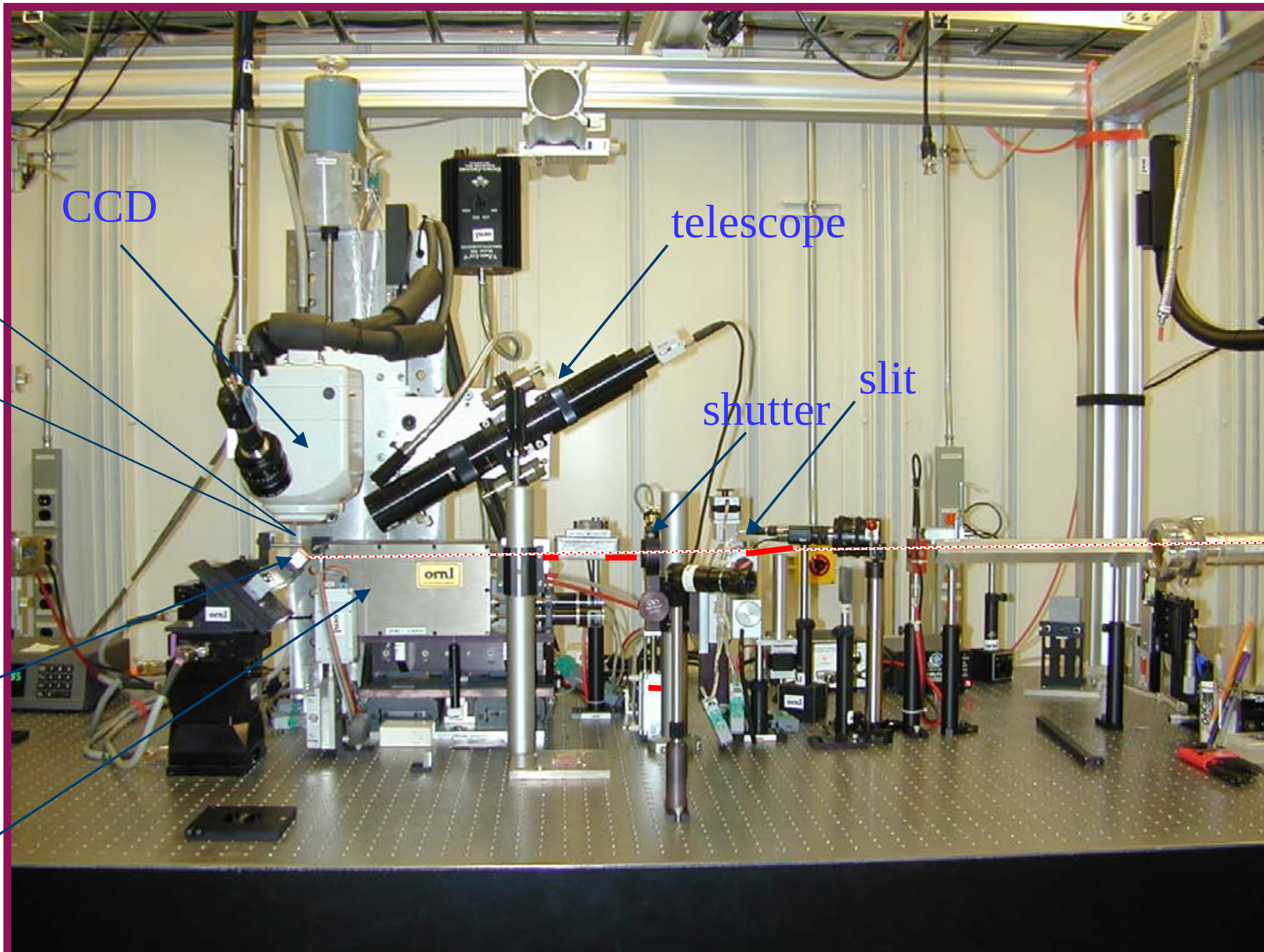


differential aperture

(wire scan, ~ 200 μm above sample surface)

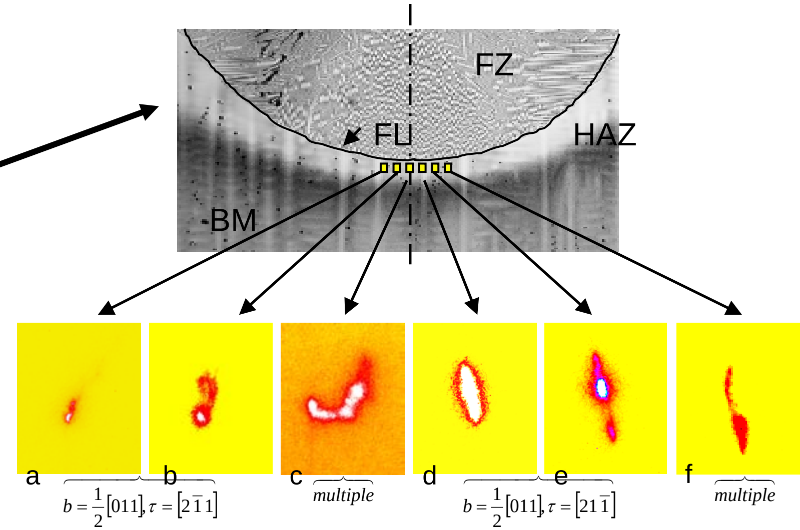
Sample stage

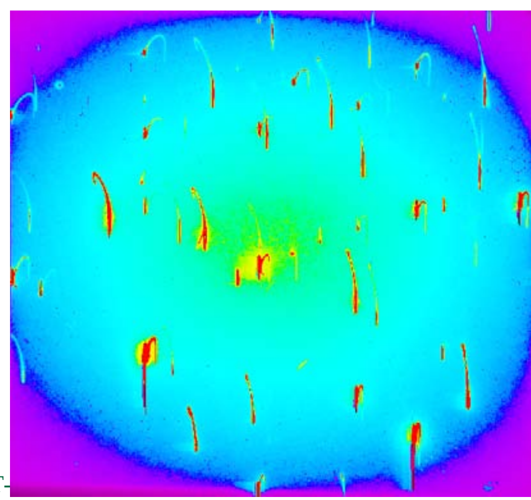
K-B Focusing mirrors

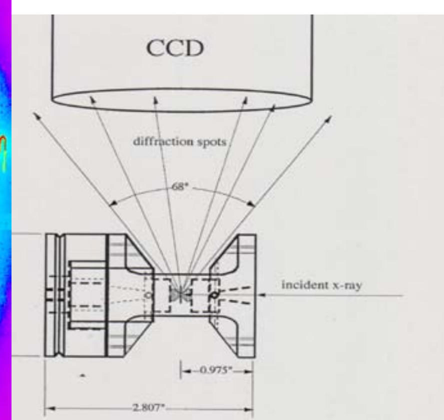


Ongoing research too extensive to cover

- **Fracture/stress localization in thin films**
- **Residual stresses/ deformation/ grain boundary network near welds**
- **Complex phase patterned materials**
- **Extreme environmental chambers**

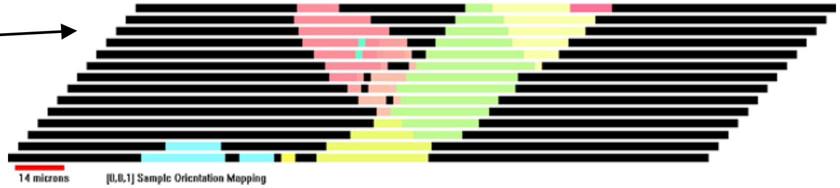




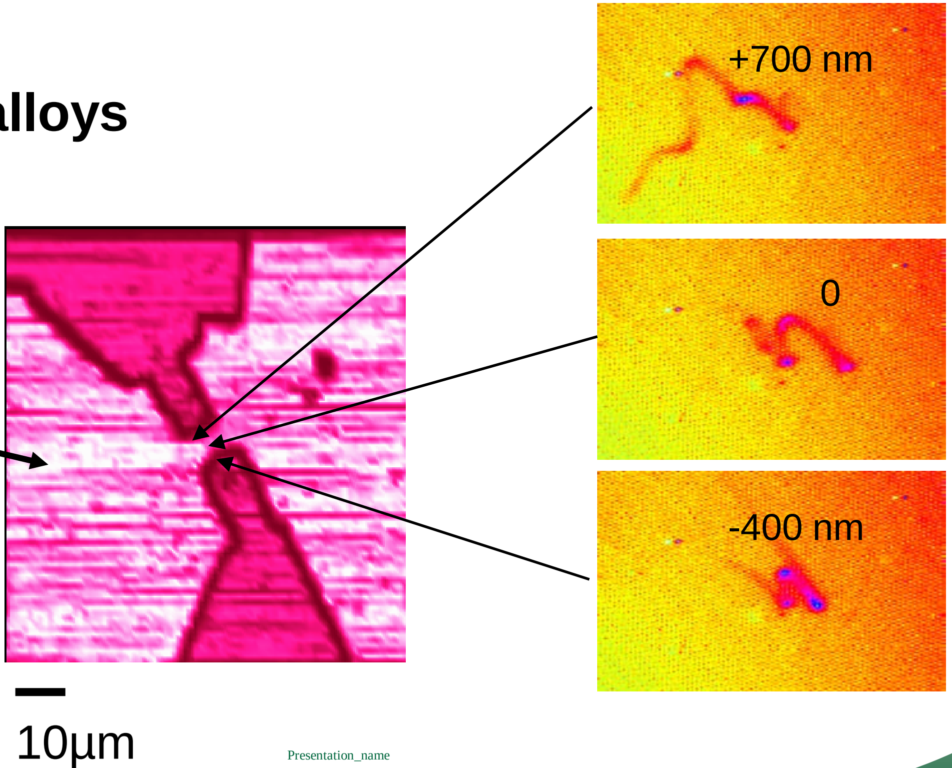


Ongoing too extensive continued..

- **Domain wall structure measurements**
- **Sn whisker growth**



- **High-performance alloys**
- **Nanomaterials**



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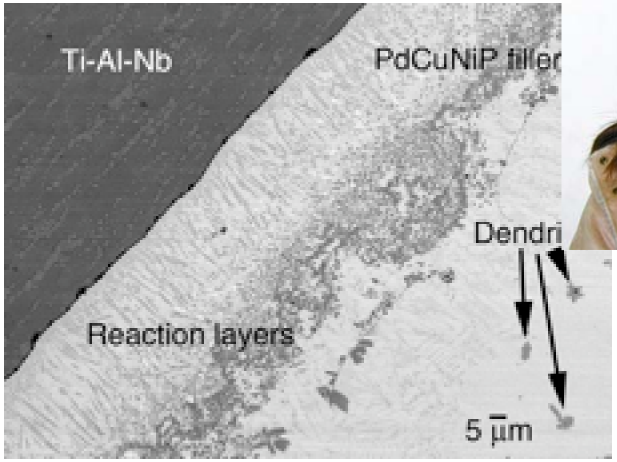
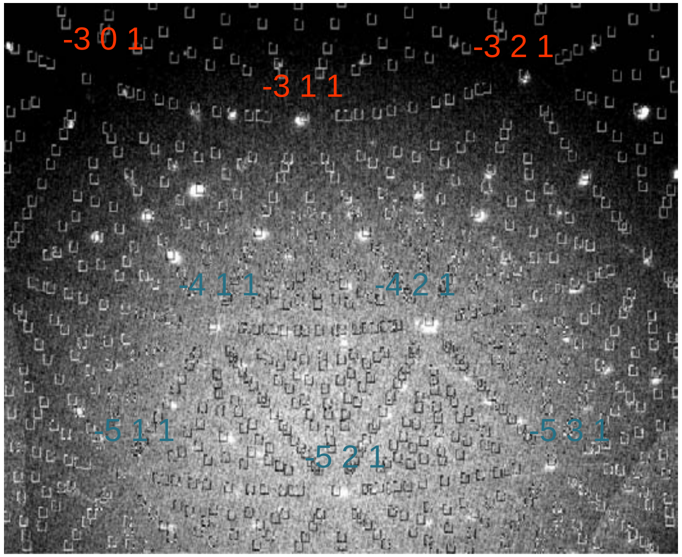
Energy scans allow structure determination

- **Generalization of orientation software can identify phases**
- **Energy scans provide integrated reflectivities.**
- **Identified two minor crystal phases tetragonal/hexagonal**

Cannot be found by powder

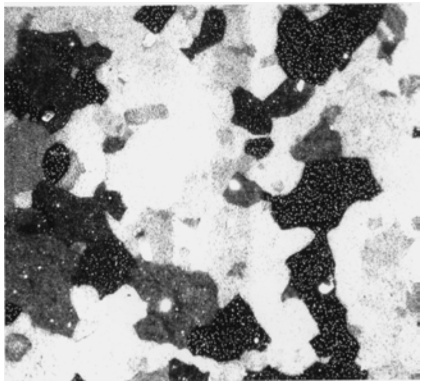


BAM braze $Pd_{40}Cu_{30}Ni_{10}P_{20}$

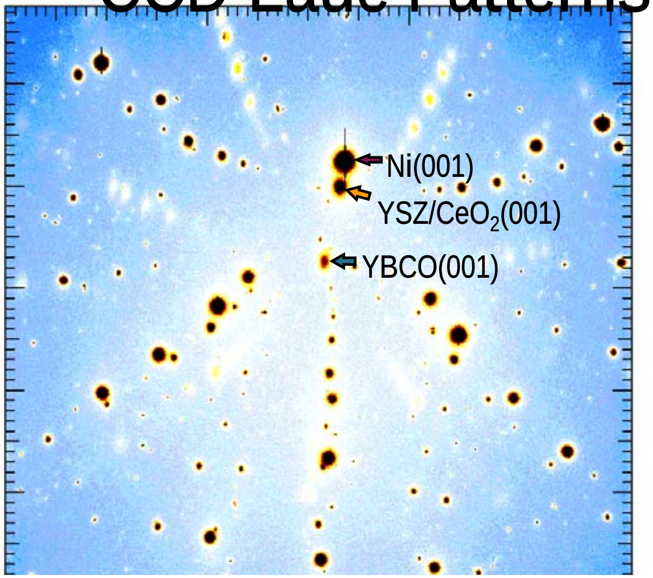


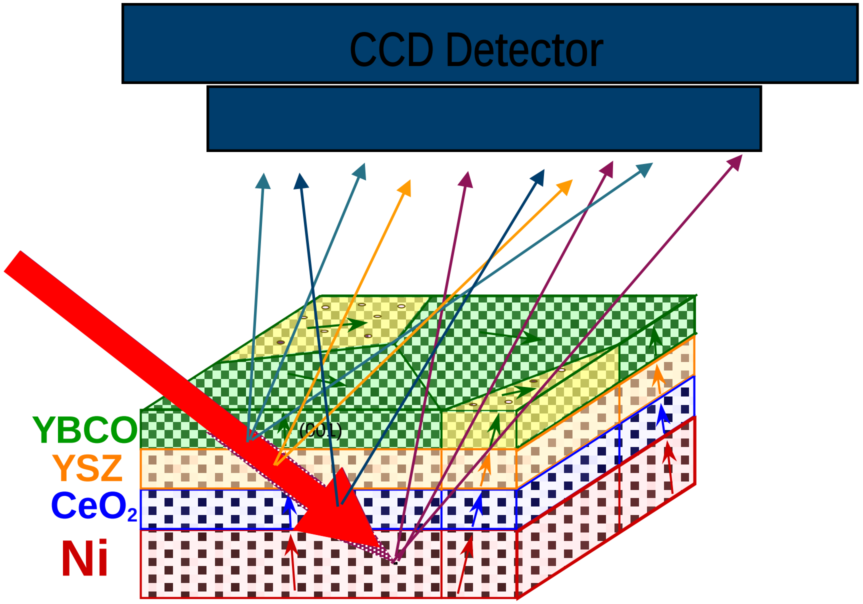
Grain-growth/ Budai et al. characterized epitaxial growth RABiTS

Optical: ~50μm grains



CCD Laue Patterns

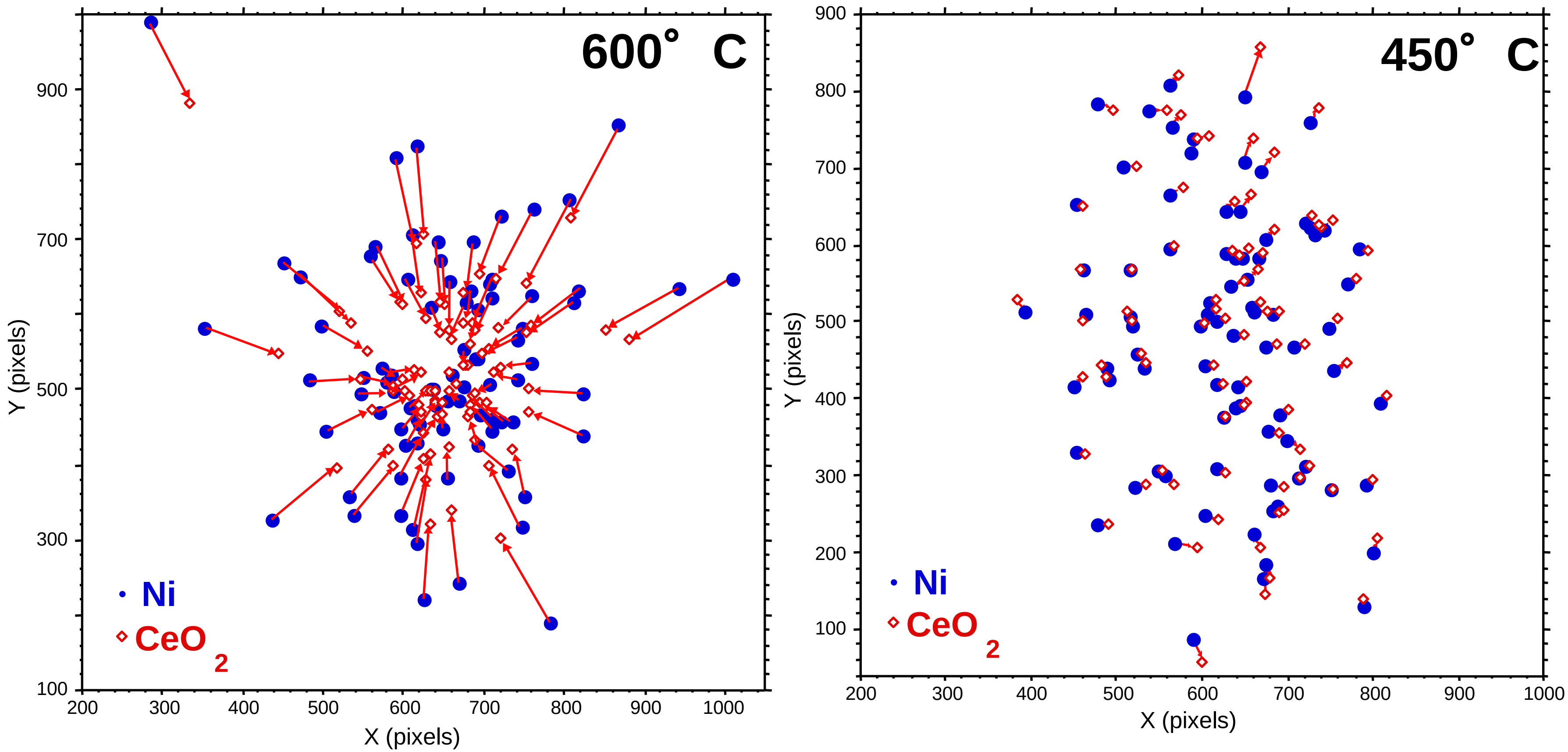




CeO_2 Observation:

Exact epitaxy for growth at low T; lattice tilts at high T

Relative CeO_2 orientation depends deposition temperature



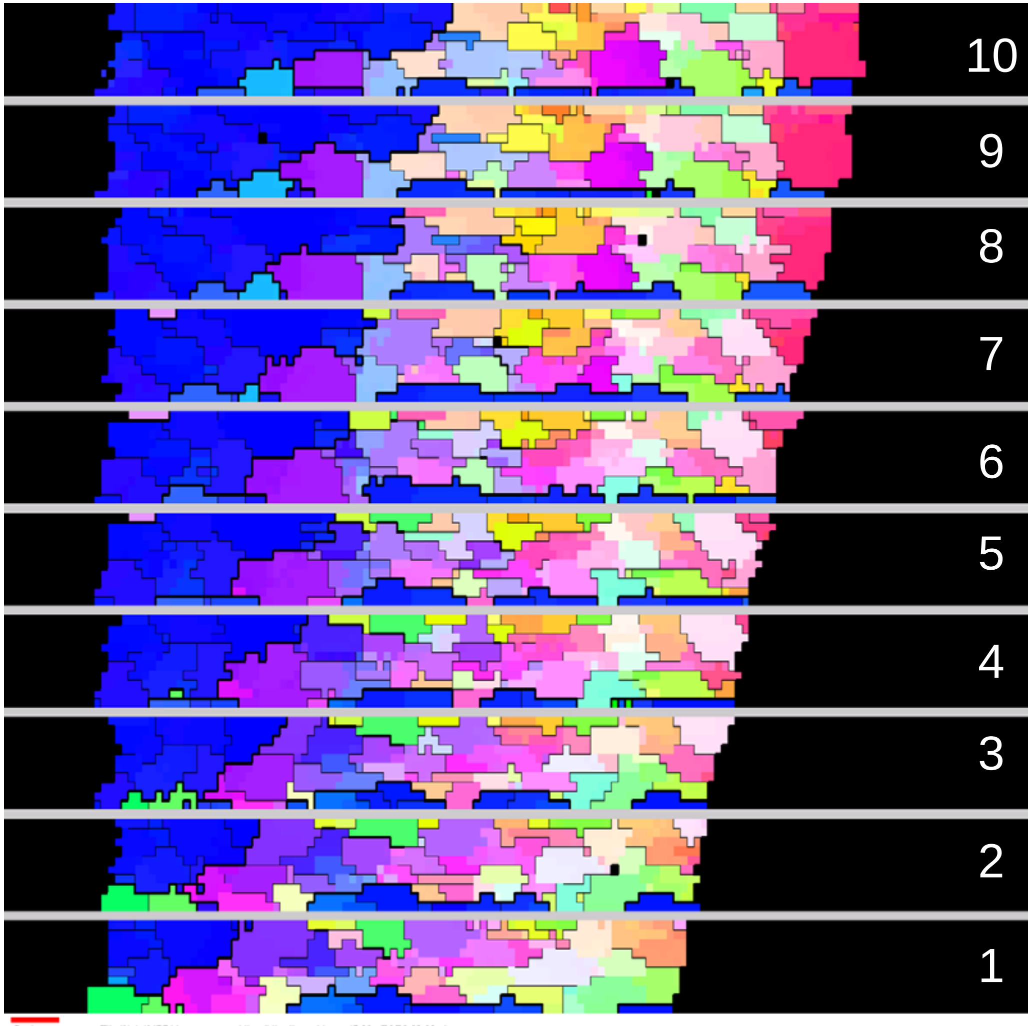
Step edge growth- good:

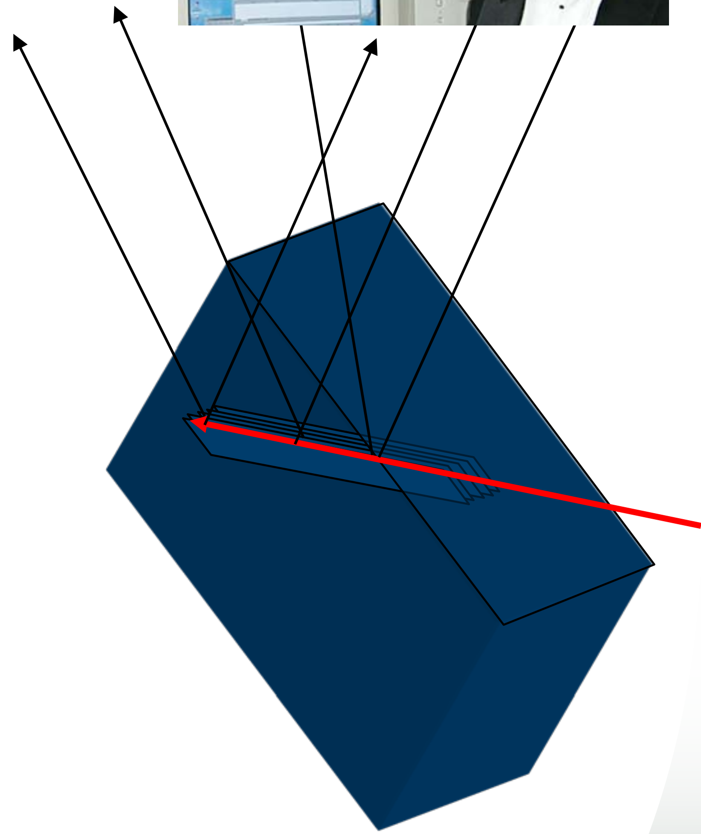
Crystallographic tilt towards ⊥

Tilt increases monotonically with miscut

Island growth-bad:

In-situ observations of 3D Grain Growth

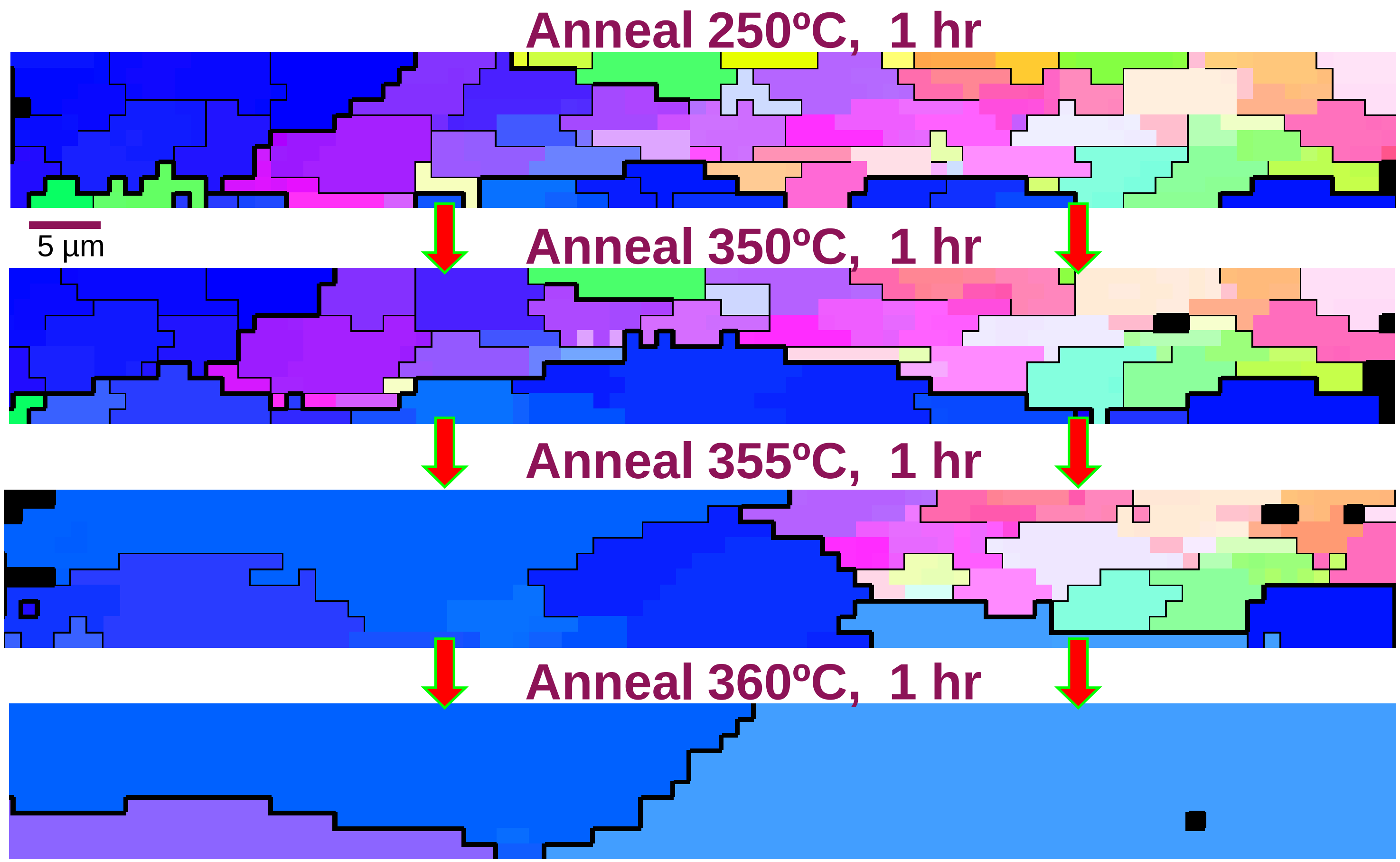




Hot-rolled (200˚ C
1xxx Al(~1%Fe,Si)
Alcoa Polycrystal

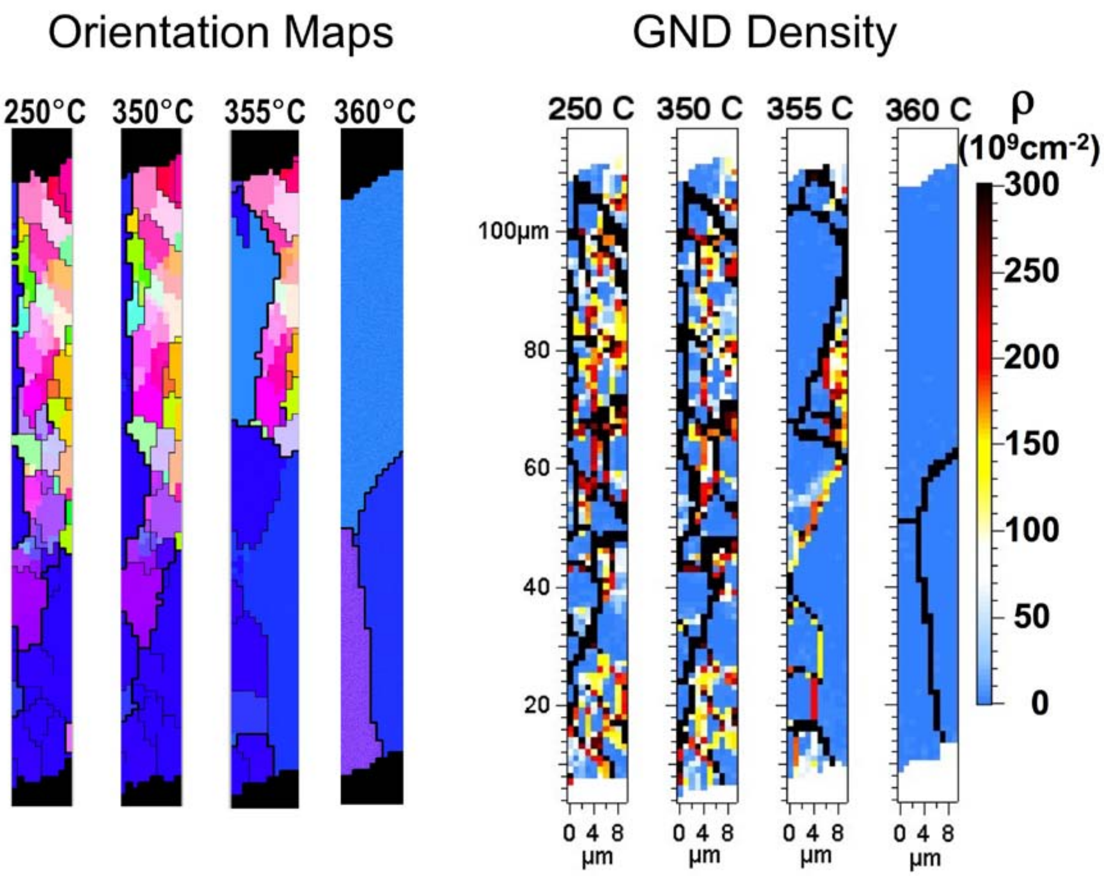
Thermal Grain Growth in Hot-Rolled Aluminum

1 µm pixels, Boundaries: 5° & 20°



- **GB motions include both high-angle and low-angle boundaries**
- **Complete and detailed 3D evolution needed for validation of theories.**

Thermal Grain-Growth And Microstructure Refinement in Polycrystalline Al



- **3D X-ray Microscopy Measurements of Dislocation Density Finds Microstructure Refinement to Be Important**

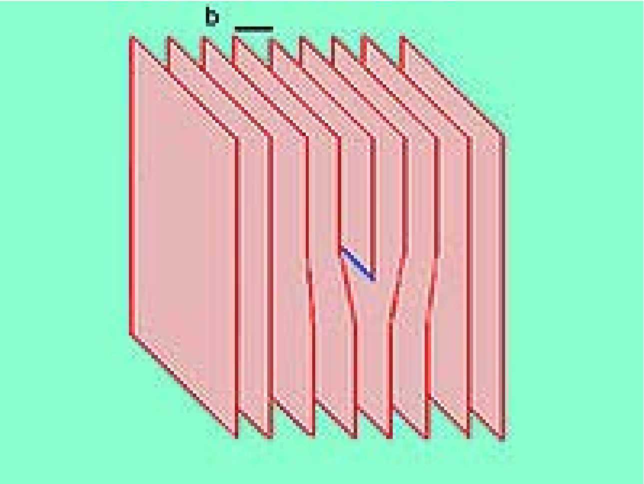
Deformation mediated by "dislocations"

- **Individual dislocations can be seen with TEM-but…**

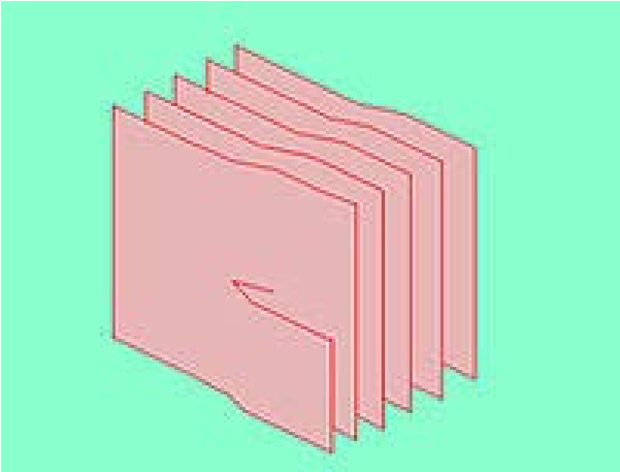
X-ray people complain thin electron samples Fundamentally different

Neutron people complain thin X-ray samples Fundamentally different

What is "thin" and "bulk"?



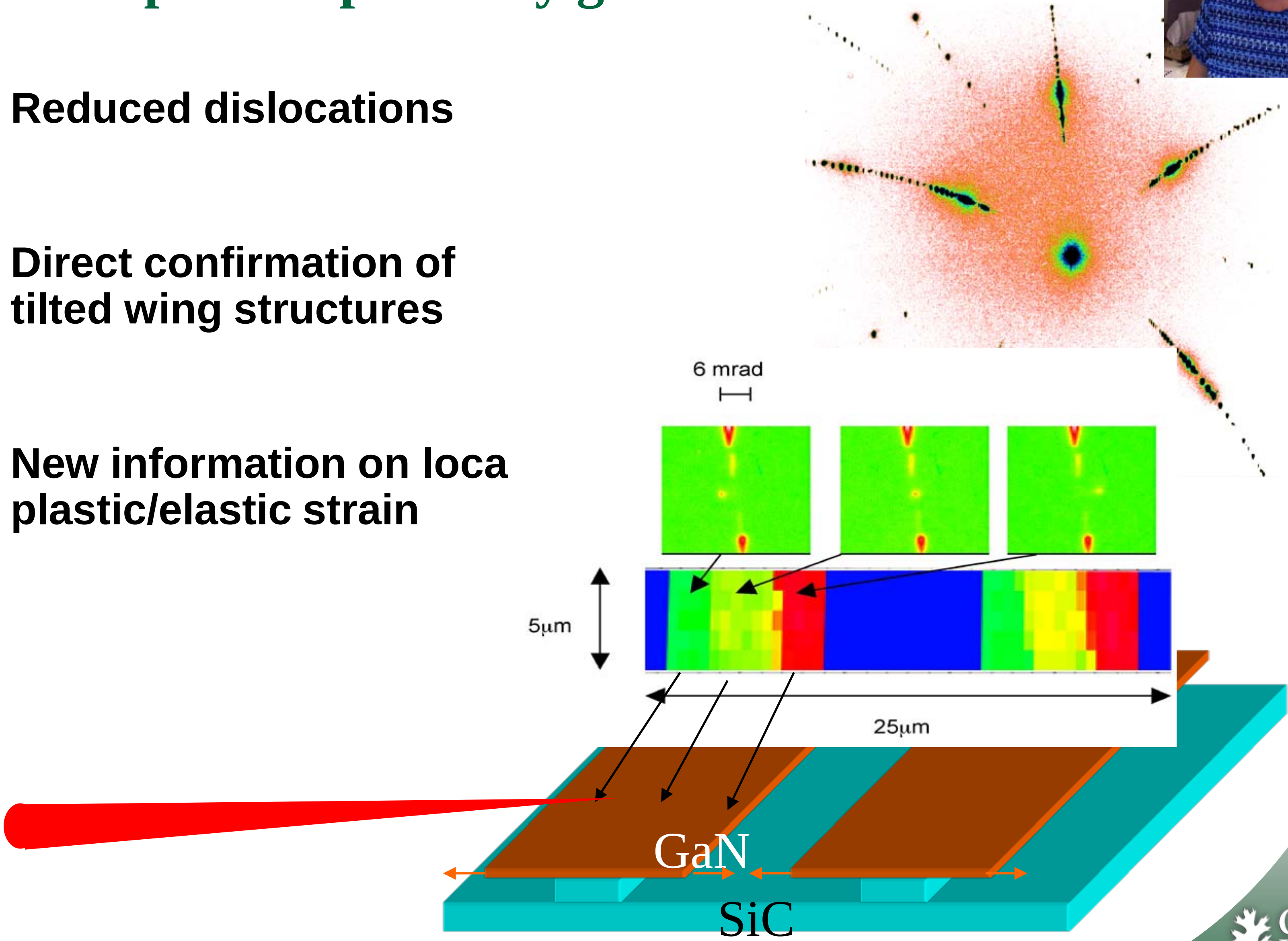
Edge dislocation



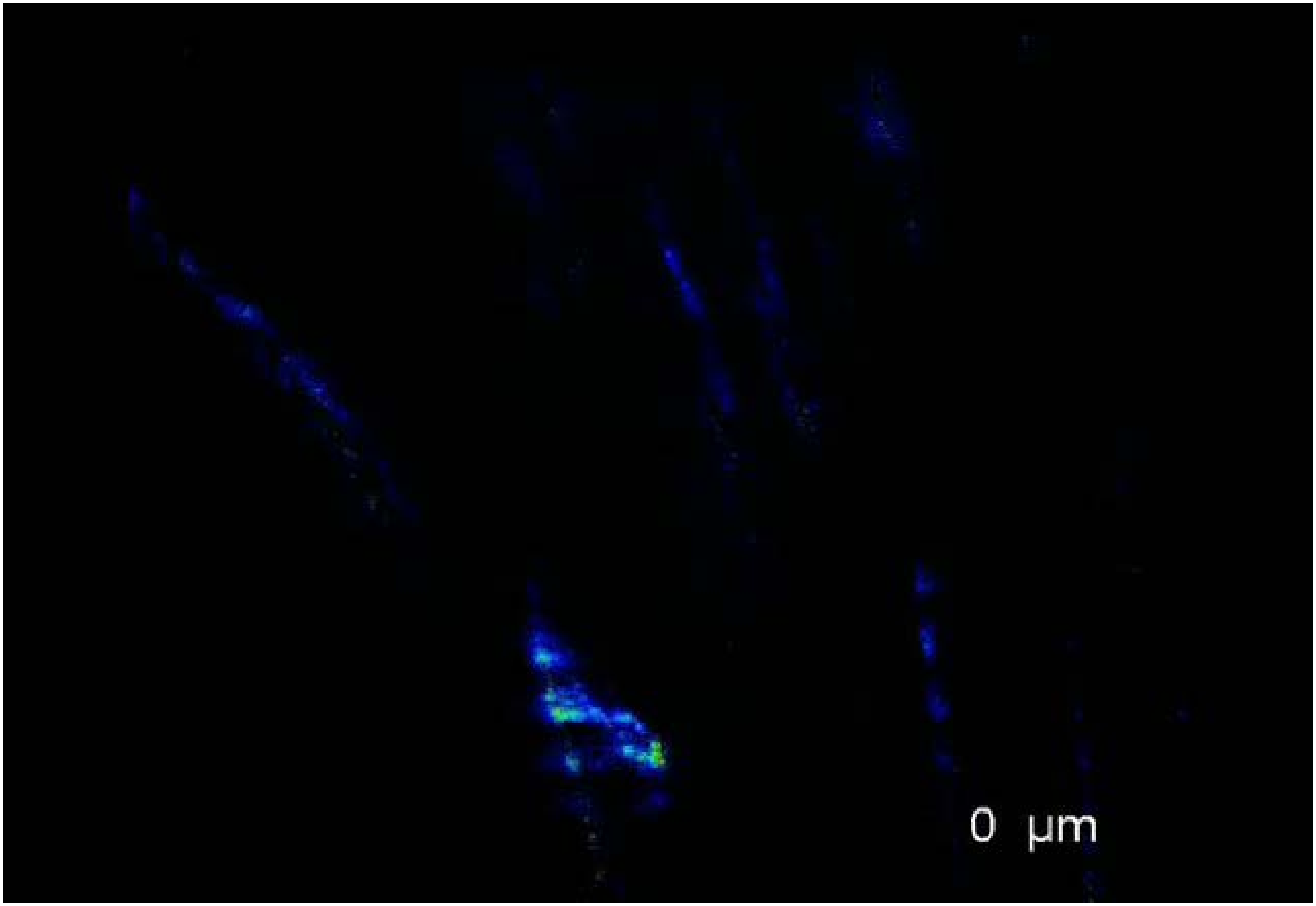
Screw Dislocation

Local orientation and plastic/elastic deformation mapped in pendeoepitaxially grown GaN

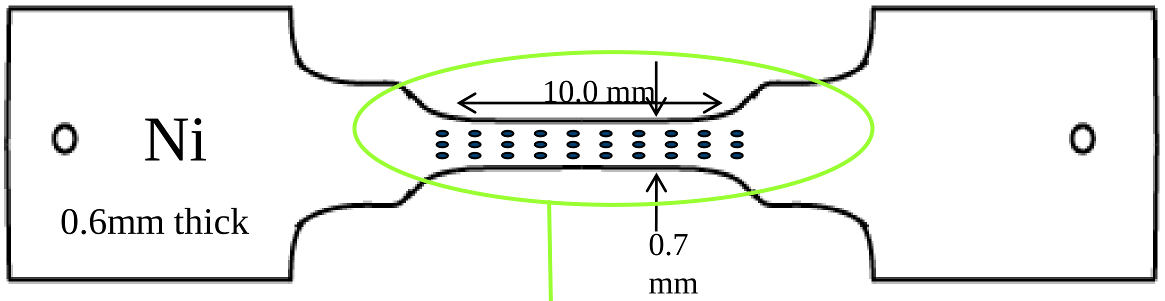
- **Reduced dislocations**
- **Direct confirmation of tilted wing structures**
- **New information on loca plastic/elastic strain**

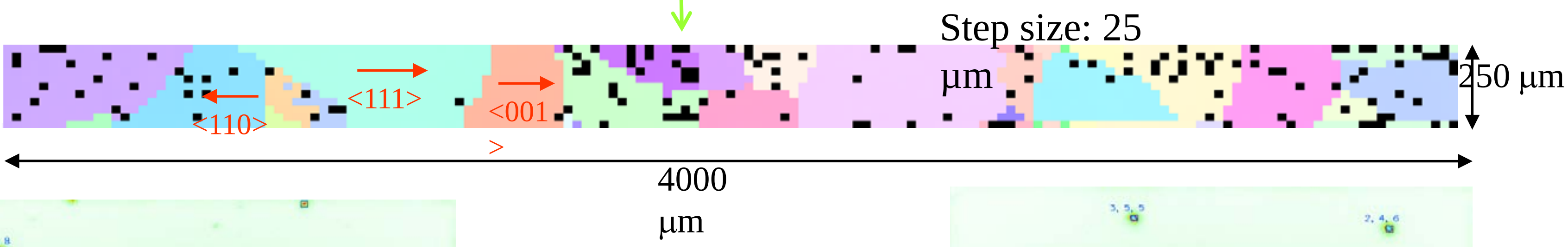


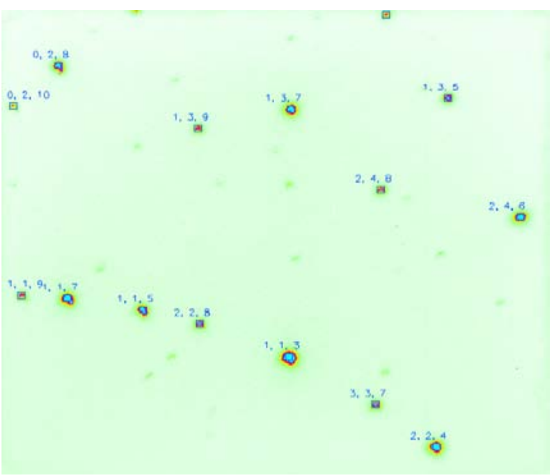
Deformation typically larger near surfaces/grain boundaries



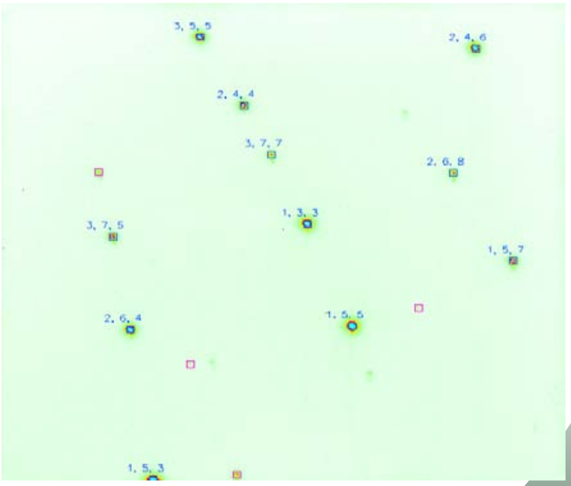
Deformation in polycrystals illustrates grain boundary behavior



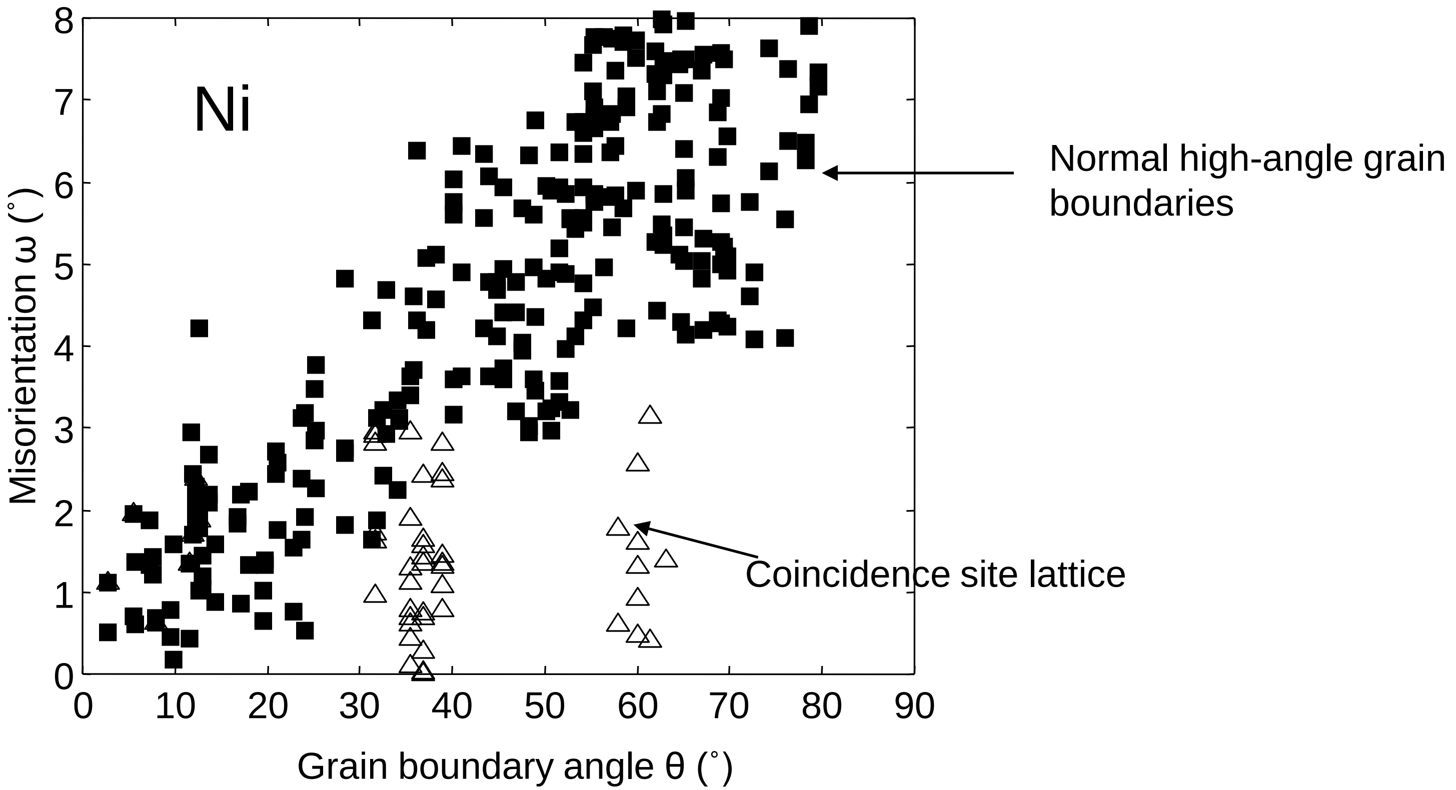




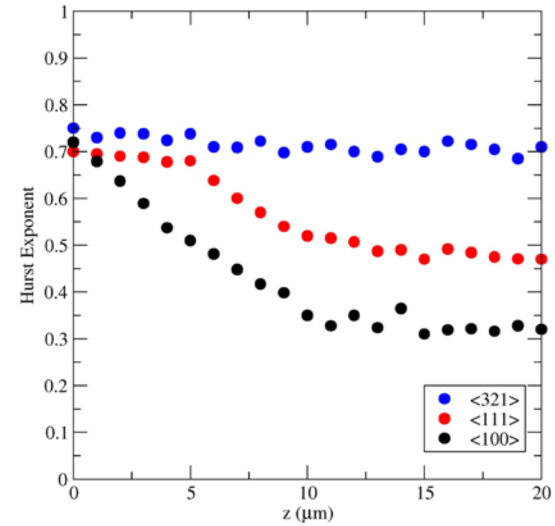
Indexation



Deformation induced rotations across grain boundaries sensitive to boundary type

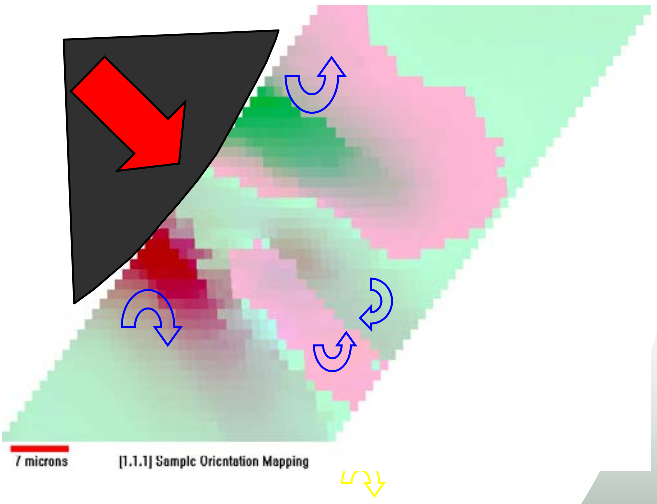


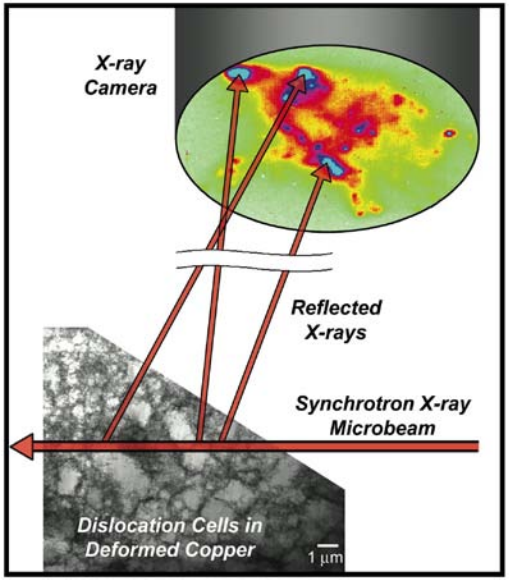
New information about deformation



- Near surfaces and interfaces
- In single and polycrystals
- Organization dynamics of defects (Krivoglaz defects of 2nd kind)

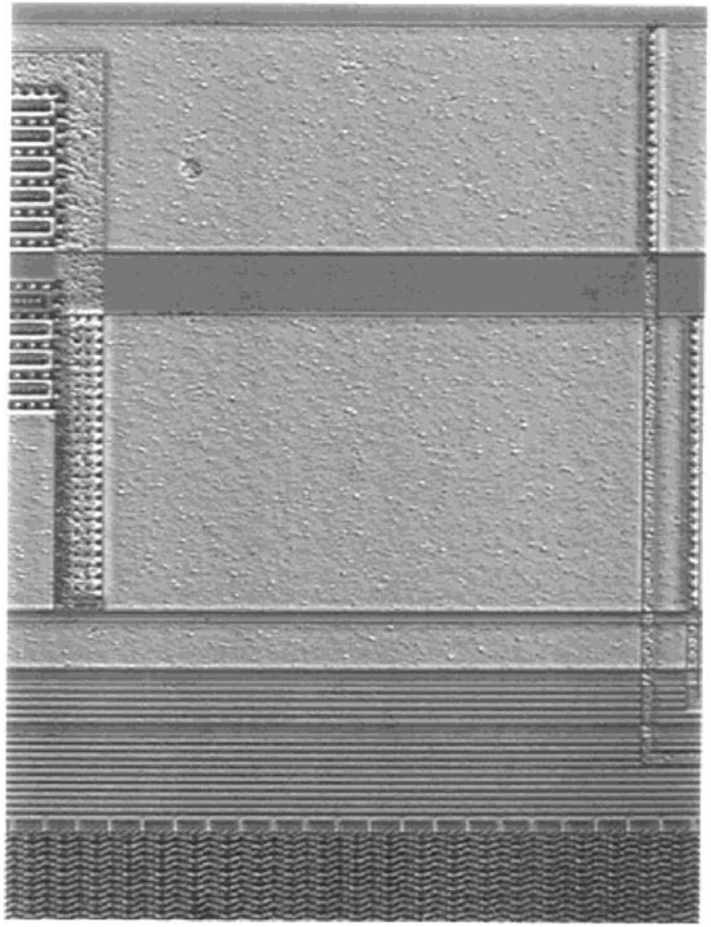
Lattice Rotation Map





Deformation found during electromigration

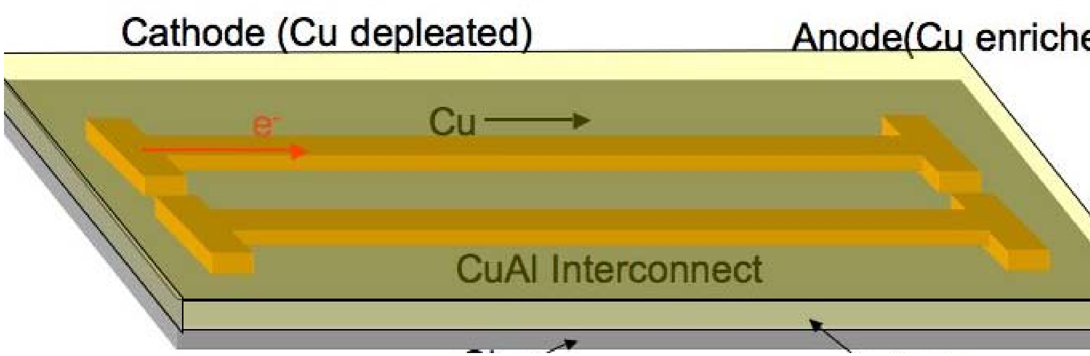
- **Device reliability linked to electromigration-MA/cm^2**
- **Theories assume stress development opposses mass transport**
- **Need to measure stress drove development of polychromatic microdiffraction**

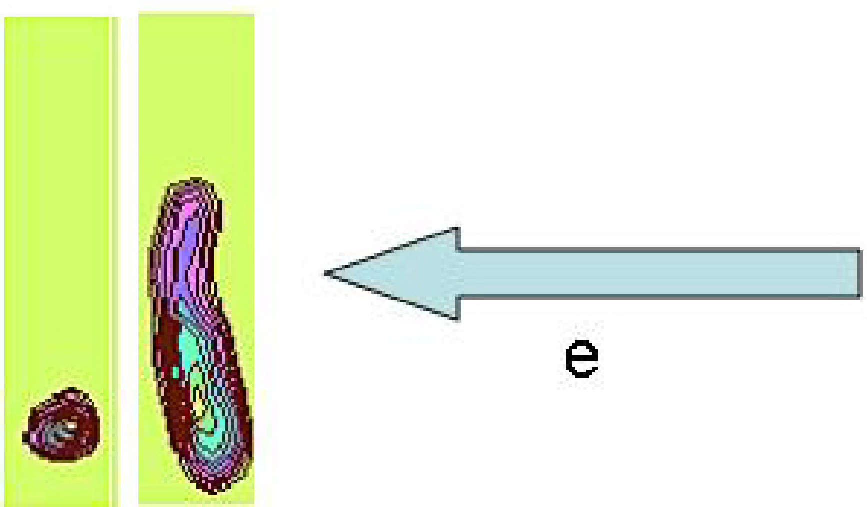


R. I Barabash et al. *J. Appl. Phys* **93** 5701 (2003).

Al (Cu) interconnects studied

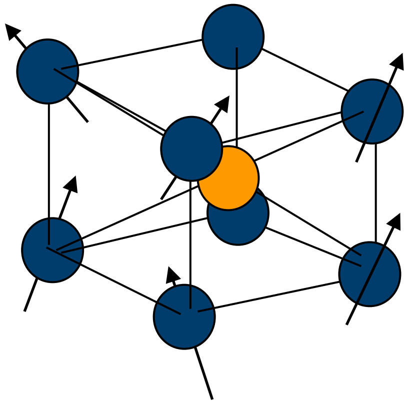
- **Deformation big grains.**
- **Small grains rotate.**
- **Deformation preceded Cu diffusion**
- **Unpaired dislocations develop parallel to the electrical field.**
- **Little dislocation activity near the anode end of the wire (Cu enhanced).**



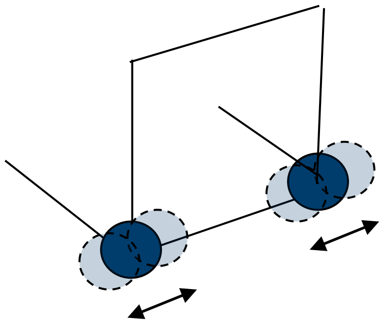


Neutron analog additional opportunities

Magnetism



Atomic motions



Low Z materials

Nobel prize to Shull and Brockhouse

Even the most intense neutron source must be used efficiently

Neutron sources 10^{12} lower brilliance than advanced x-ray

Neutrons expensive 10^{13} more expensive!

10^{-16}\$ /x-ray

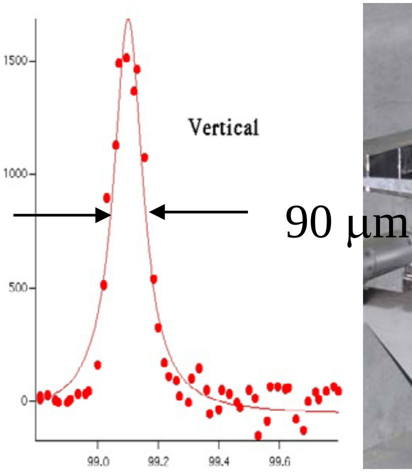
10^{-3}\$ /neutron

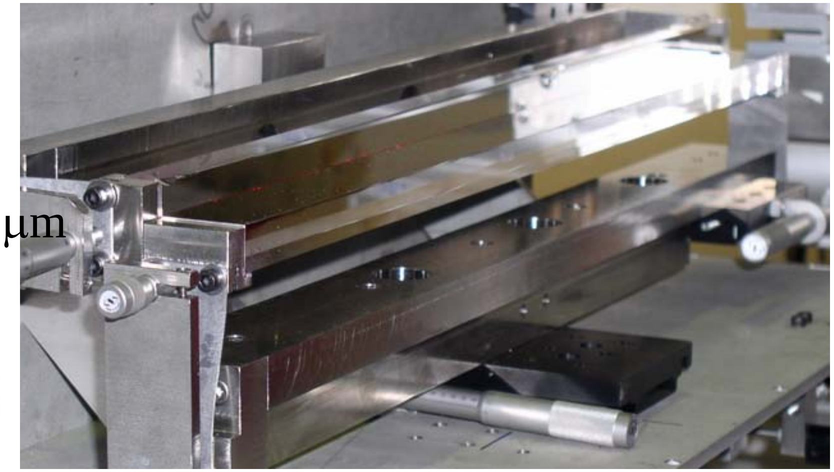
Increase divergence/bandpass

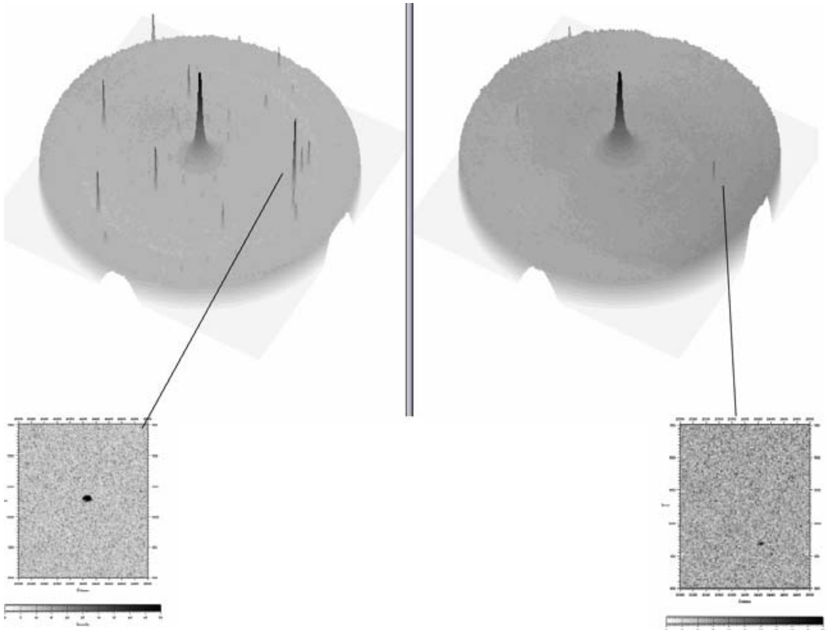
10^{-9}\$/ neutron

Neutron mirrors produce microbeams

- **Better signal-to-noise**
- **Resolve inhomogeneities**
- **Map crystal distributions**



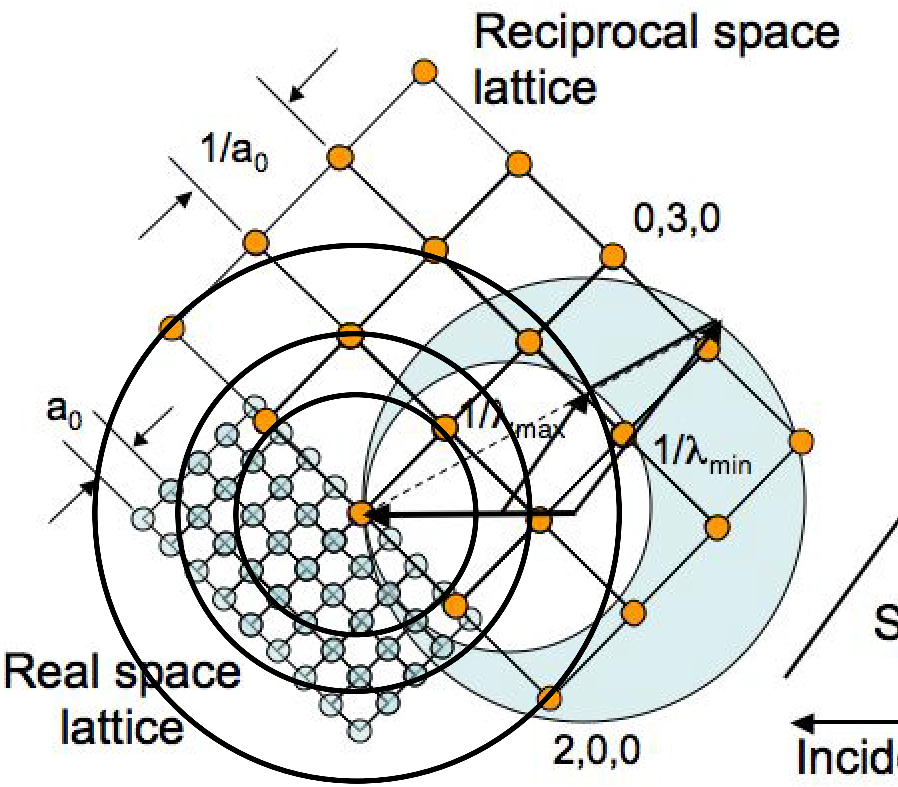




Useable 25 μm beams?

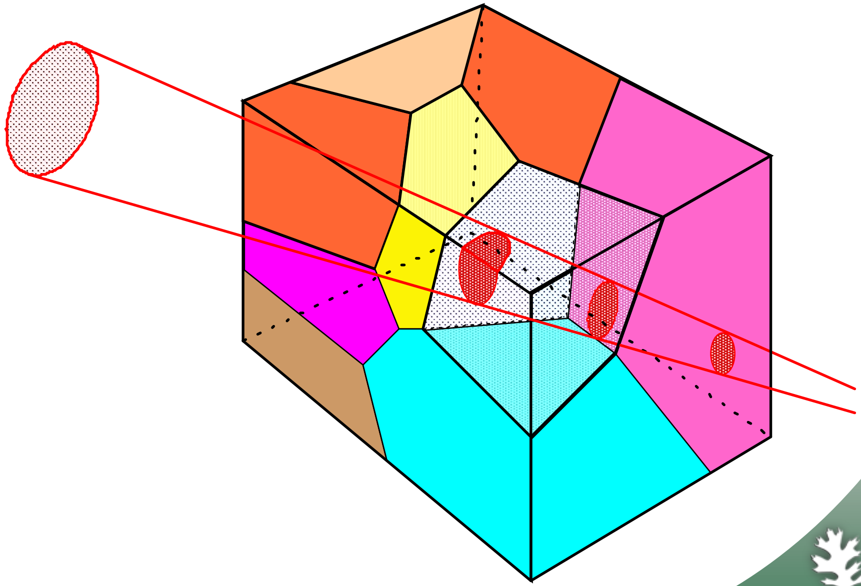
Spallation neutron science intrinsically polychromatic

- **Analogous to polychromatic X-ray microdiffraction-*but includes* energy**
- **Allows for structure determination**
- **Absolute strain measurements**



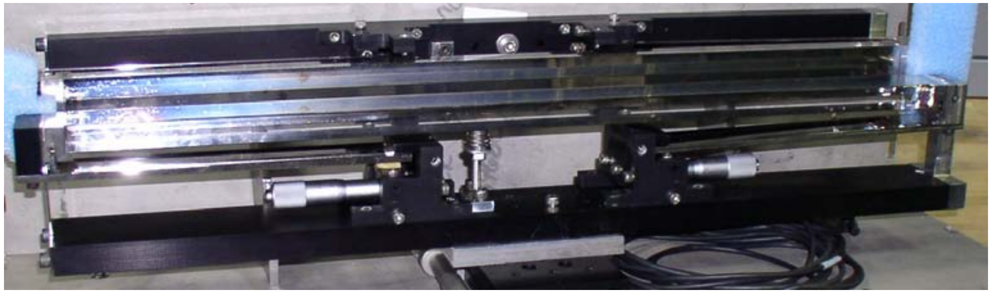
Will extend neutron science

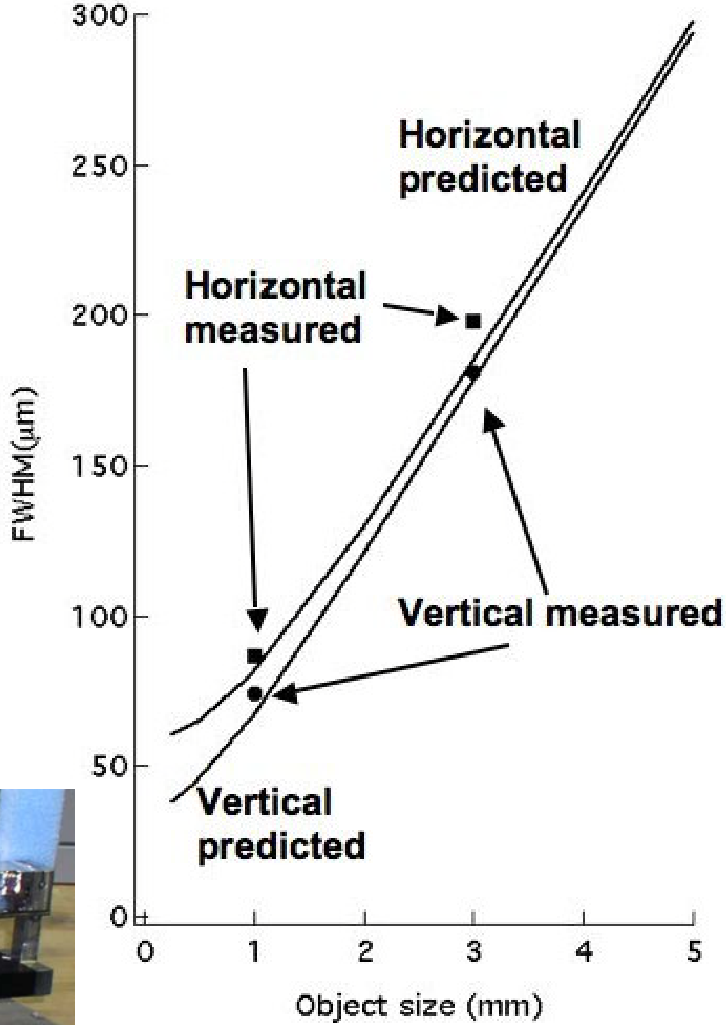
- **Inhomogeneous samples**
- **Small samples in environmental chambers**
- **Spatial resolved distributions deep in samples**



SNAP experiments diffraction high pressure cells

- **Focusing optics work near theoretical limit**
- **Minor improvements should enable 25 micron measurements**





Conclusion: Microdiffraction

- **Addresses long-standing issues materials physics**
- **Techniques and instrumentation rapidly evolving**
- **Answers specific questions about materials systems (Energy materials)**
- **Extend x-ray and neutron characterization to new classes of samples.**
  - **Dangerous**
  - **Inhomogeneous**
  - **Samples in extreme environments**

Advanced materials structures- intrinsically 3D
And spatial resolution- is needed urgently
X-ray and neutron beams- can penetrate to see
Materials crystal structures- and heterogeneity

CHORUS

Nondispersive - optics change what we can see
Mesostructure- resolved by crystallography
Atomic defects quantified - so that we can surmise
Emergent structures origins- at the mesoscopic size

New optics and new methods- extend what we can do
With spatial resolution- time resolution too
Nondestructive lets us watch- materials deep inside
Chambers or complex system - where once they could hide

Emerging applications- I've tried to show a few
Energy materials- have challenges quite new
With x-ray and neutron beams- we now are freed
To study these materials- on the scale that we need