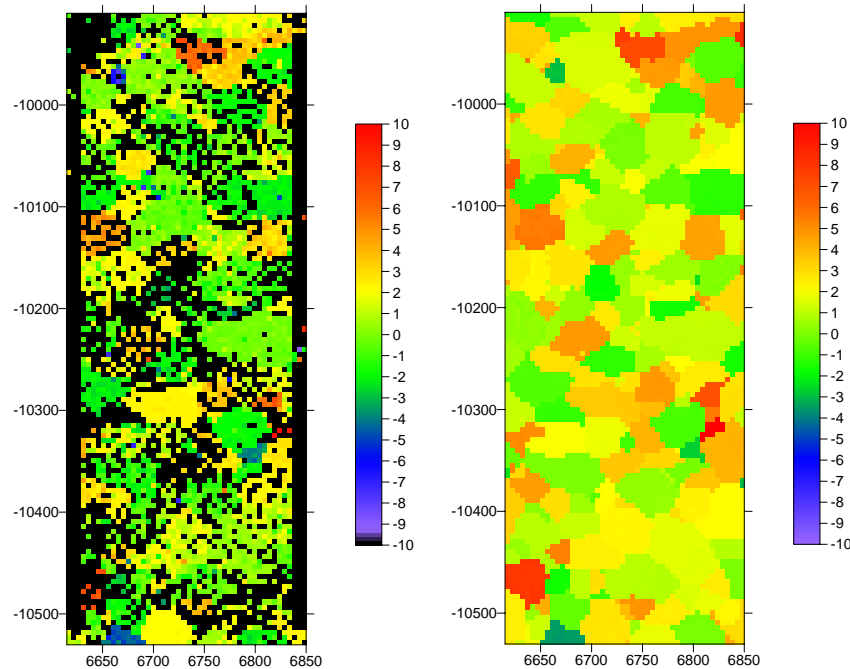


Optics and Techniques for Diffraction and Contrast Imaging

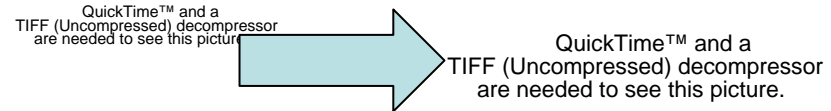
Gene E. Ice

*Materials Science and Technology Division
ORNL*

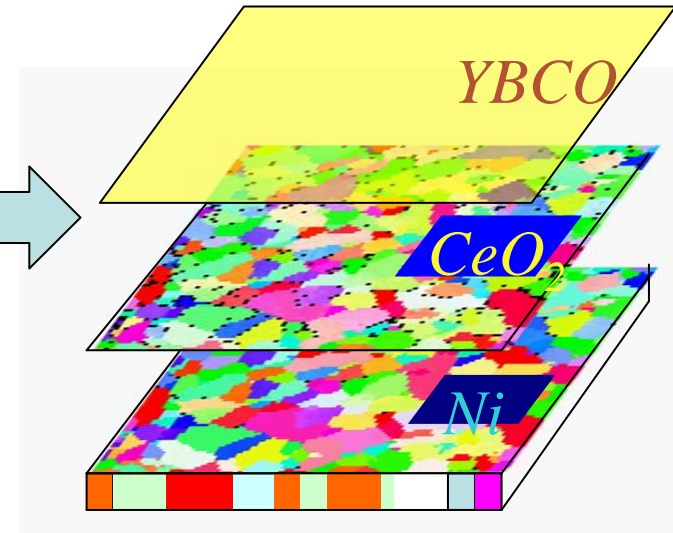
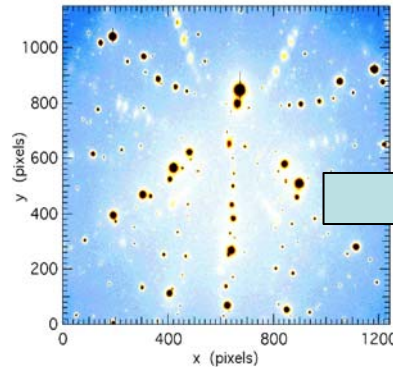


Diffraction mapping emerging area in electron and x-ray microscopy

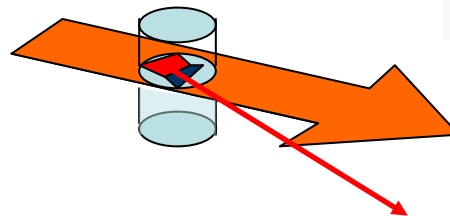
- **EBSD**
 - Surface grain structure
 - Grain curvature
 - Phase



- **Polychromatic X-ray microdiffraction**
 - Phase/texture/strain, deformation

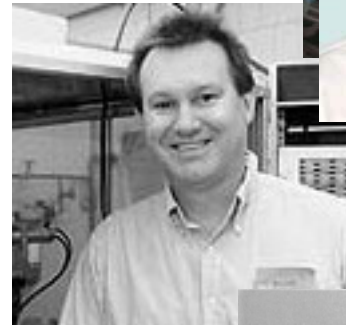
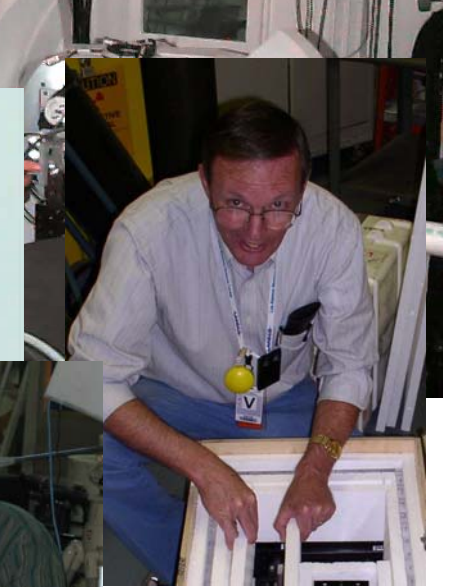
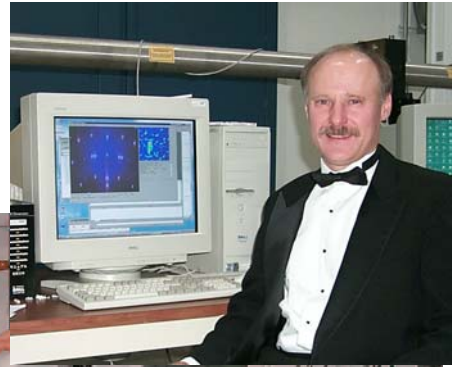


- **4D X-ray microscopy (RISØ)**
 - Time resolved
 - Deep penetration



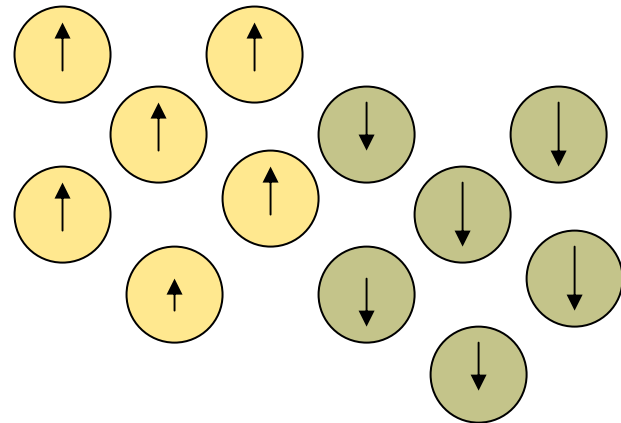
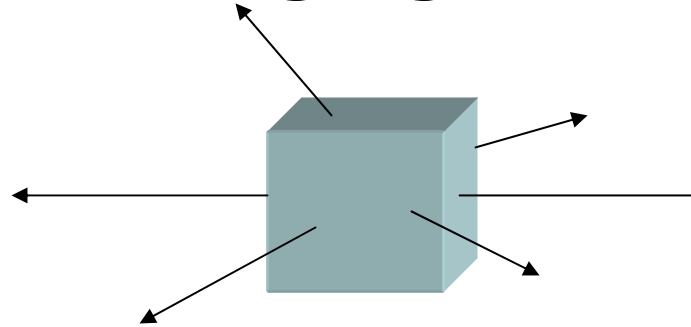
Lots of people involved

- Ben Larson/ John Budai/Jon Tischler -IPNS polychromatic microdiffraction
- Sven Vogel, Camden Hubbard - LANSE tests of focusing optics
- Ron Roggie, Judy Pang, Steve Spooner -Chalk River tests of focusing with polychromatic microdiffraction)
- Chris Tulk, John Parise etc.



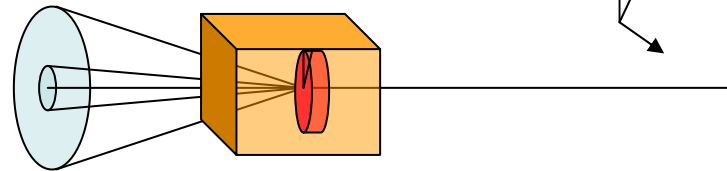
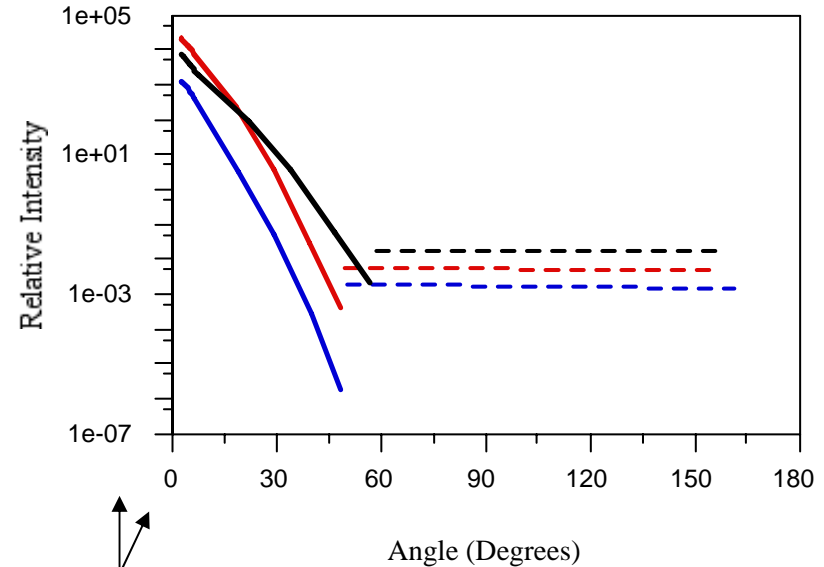
Neutrons unique advantages for diffraction imaging

- Penetrating
- Scatter at high Q
- Low damage (lowest theoretical limit)
- Low Z atoms (precipitates etc).
- Large magnetic cross section

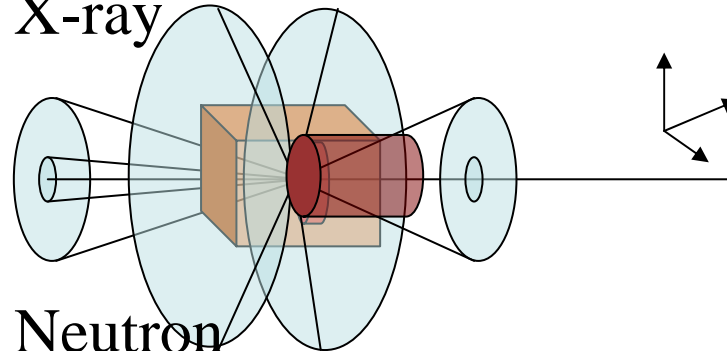


Neutrons probe bulk materials good Q resolution

- 12 orders of magnitude less brilliant than 100 keV x-rays
- But scatter into all directions
- Compliment high spatial resolution of x-rays at low energies (20 keV)



X-ray



Neutron

Polychromatic diffraction solves intrinsic problem of microdiffraction

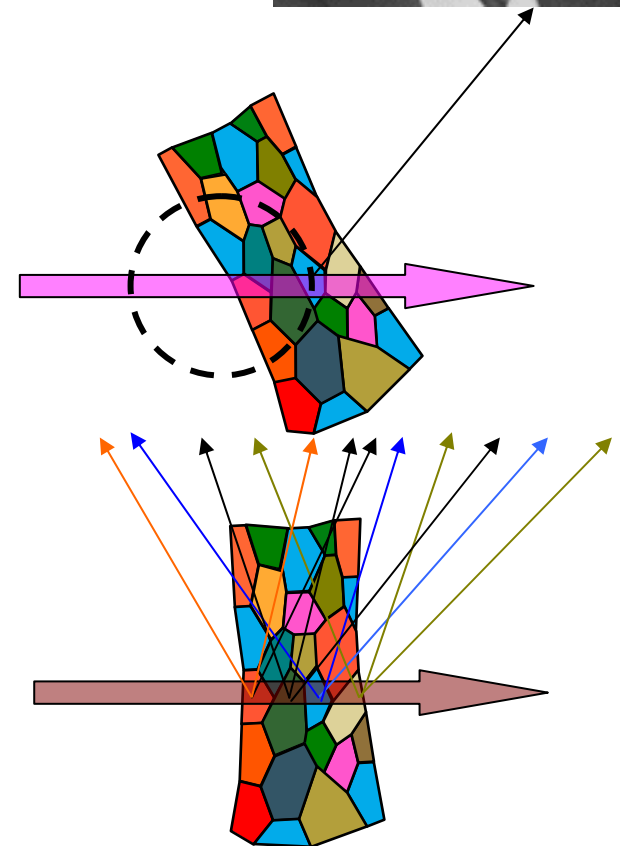


X-rays -Sample does not need to be rotated!

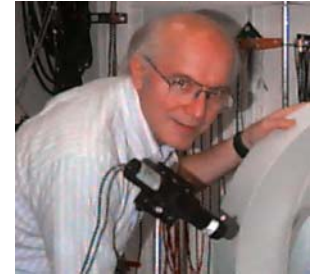
Neutrons- use every neutron!

Special software required- Can index polycrystalline samples

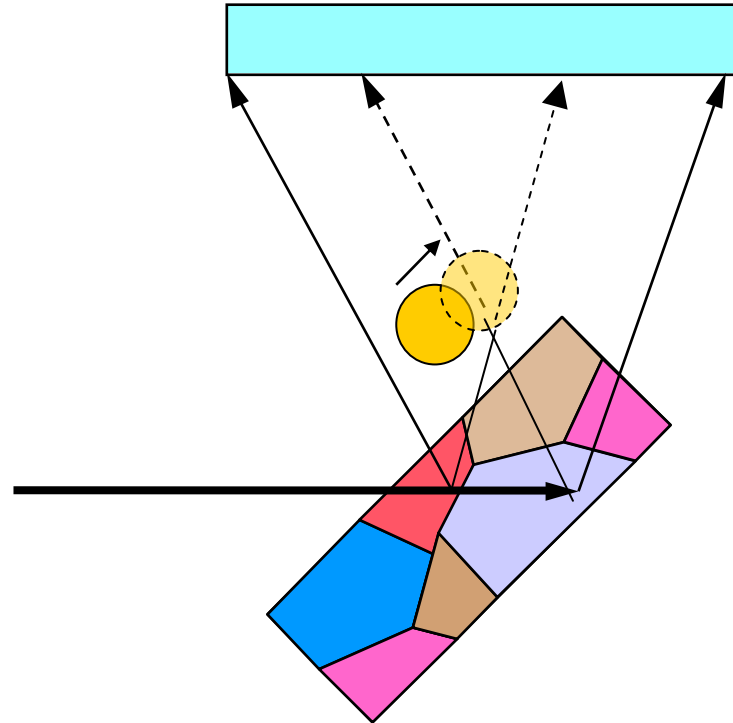
3D nondestructive probe of stress/strain/crystal structure!



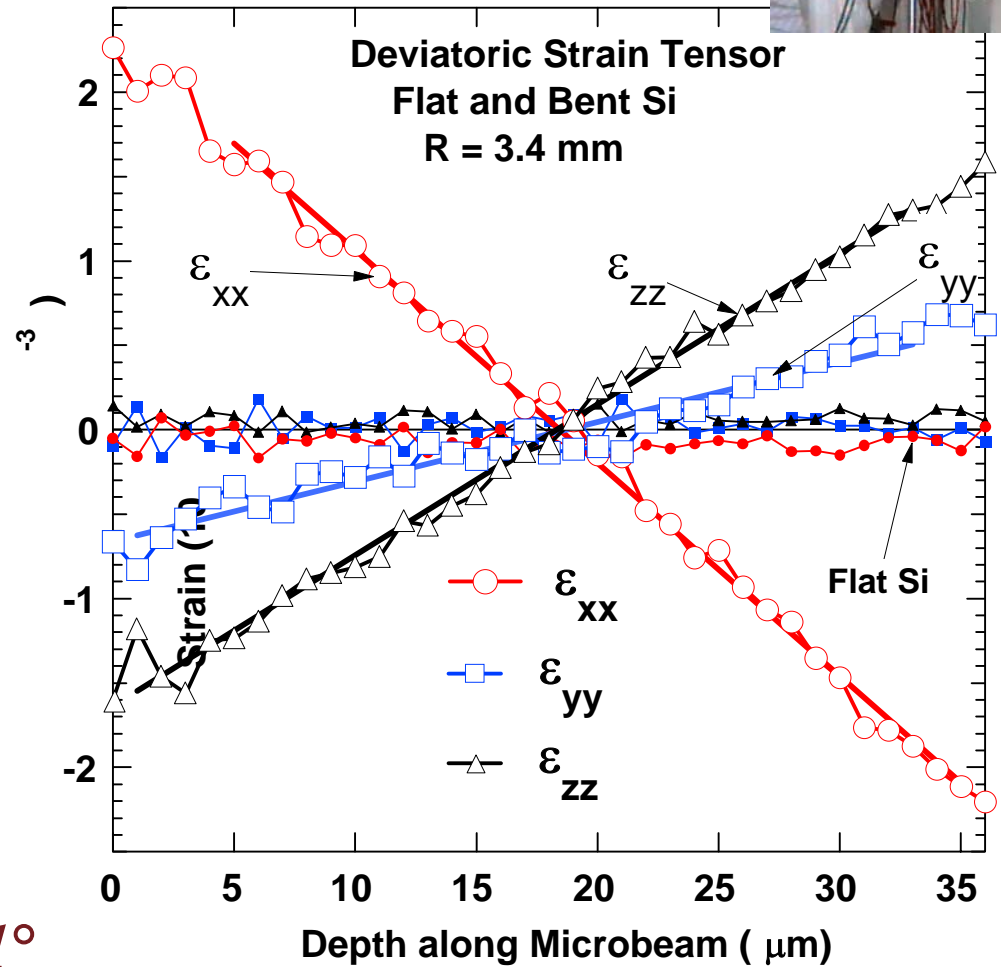
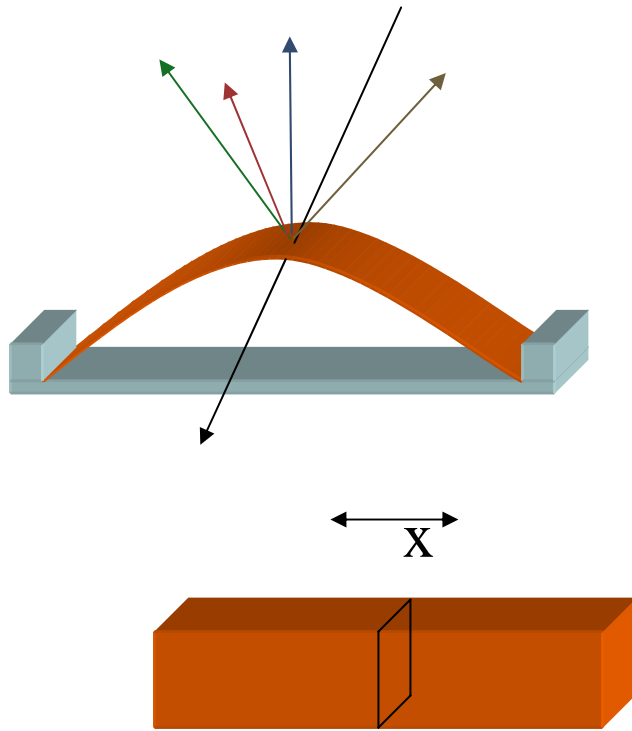
Differential aperture x-ray microscopy yields submicron information in all three directions!



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



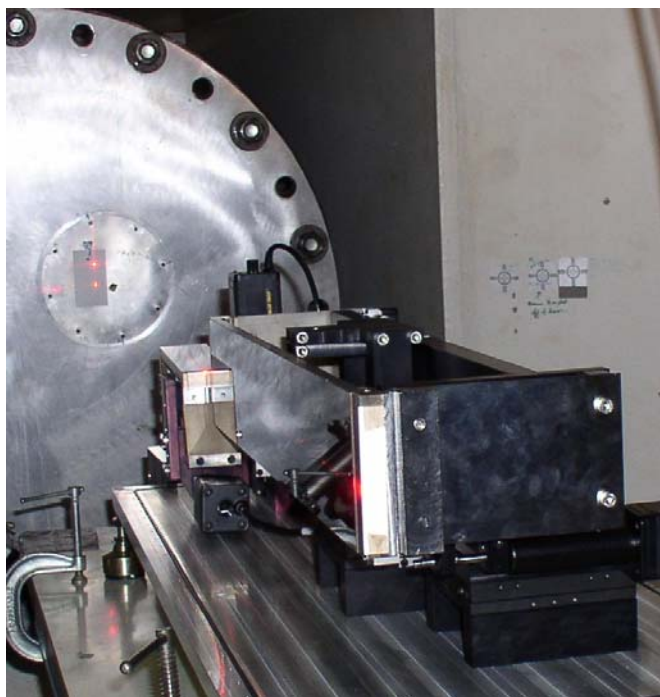
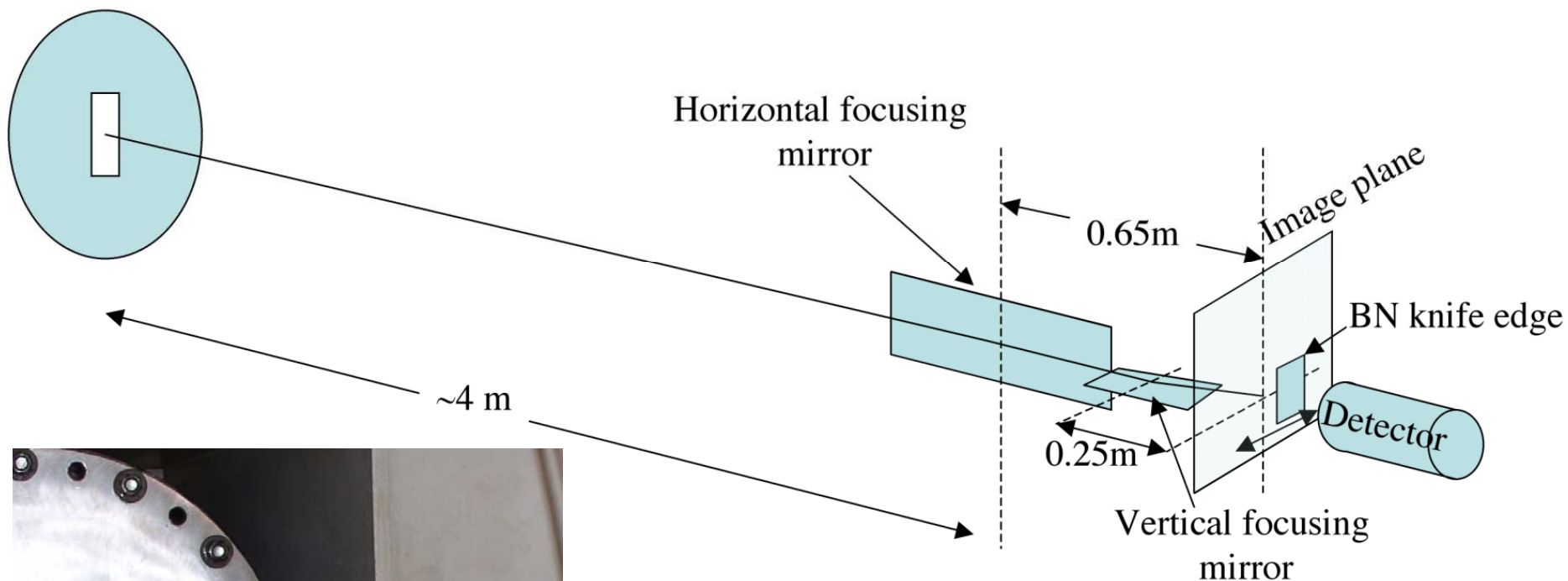
Precision measurements of strain tensor *inside* bent single crystal Si illustrate power of DAXM



Orientations to 0.001°

Elements for neutron microdiffraction tested

1 x 3 mm BN slit ~ 4 m from target



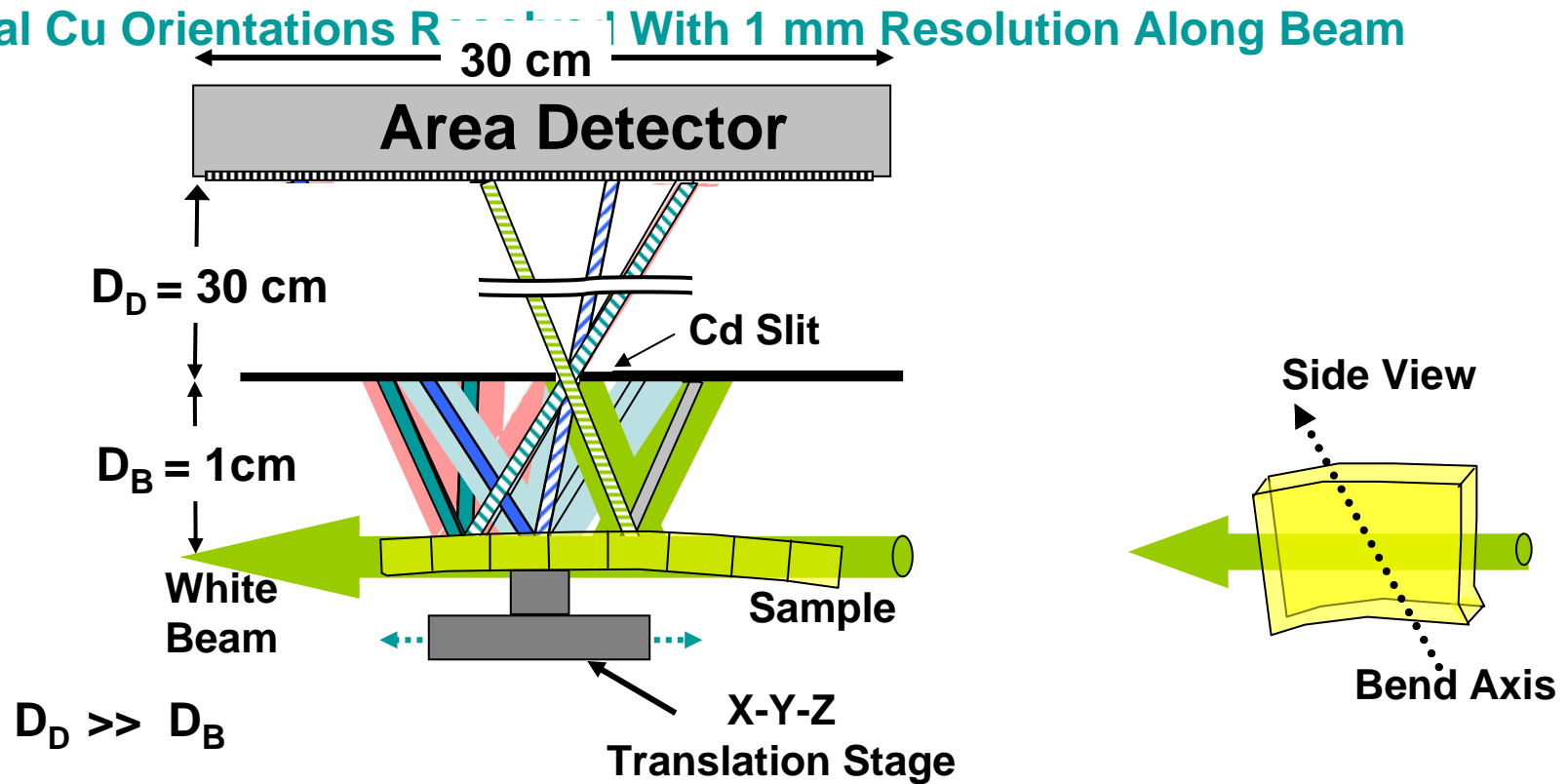
Small polychromatic beams

3D aperture-resolved diffraction

Negligible gravitational dispersion

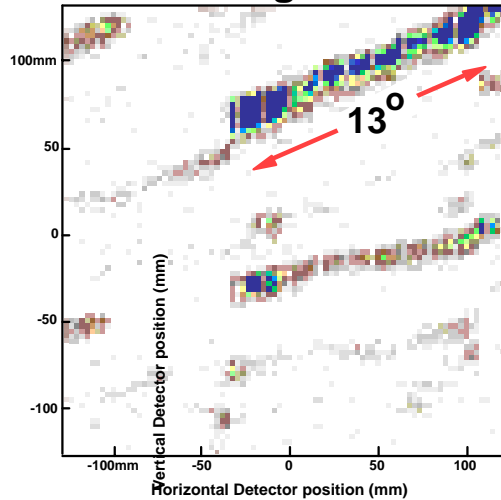
3D Neutron Structural Microscopy Has Been Demonstrated at IPNS

- Measurements Made On IPNS Single Crystal Diffraction (SCD) Beamline
- Cylindrically Bent, $\langle 001 \rangle$ Oriented Cu Crystal Plate ($2 \times 9 \times 10 \text{ mm}^3$)
- Slit System Fabricated And Depth-Resolution Analysis Software Developed
- Local Cu Orientations Resolved With 1 mm Resolution Along Beam

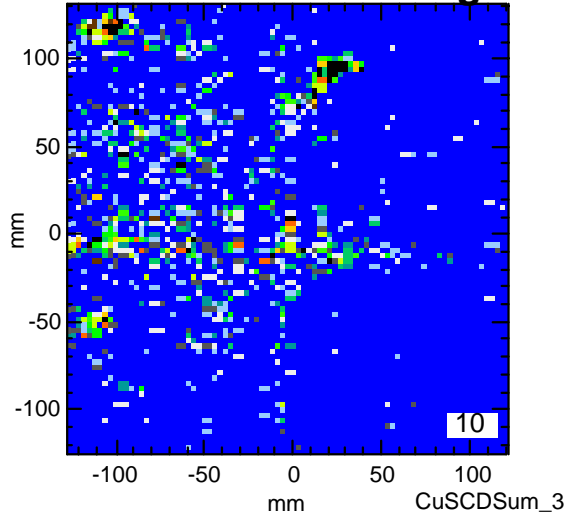


Pinhole-Camera Depth Resolution Process

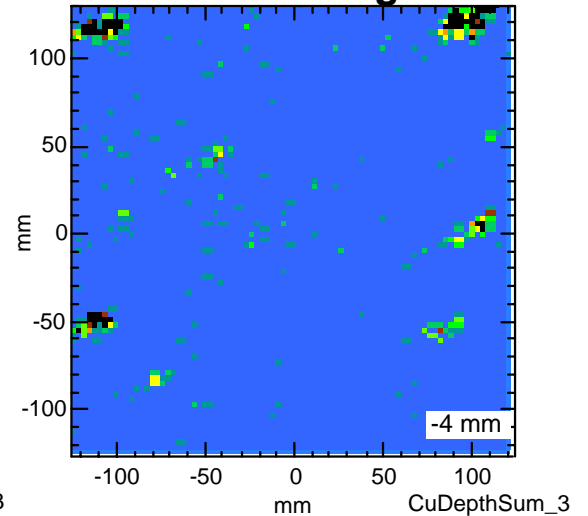
Laue Image - No Slit



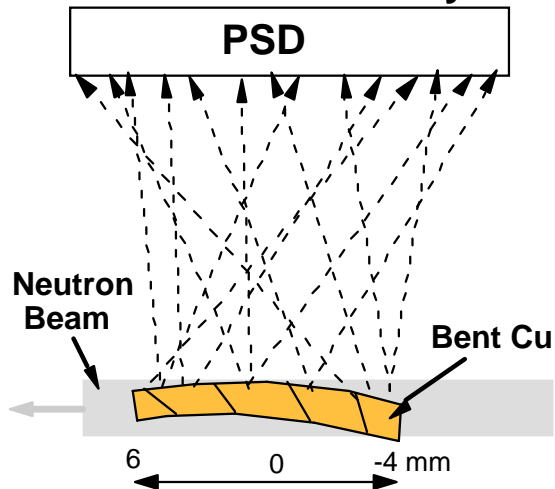
Pinhole Camera Image



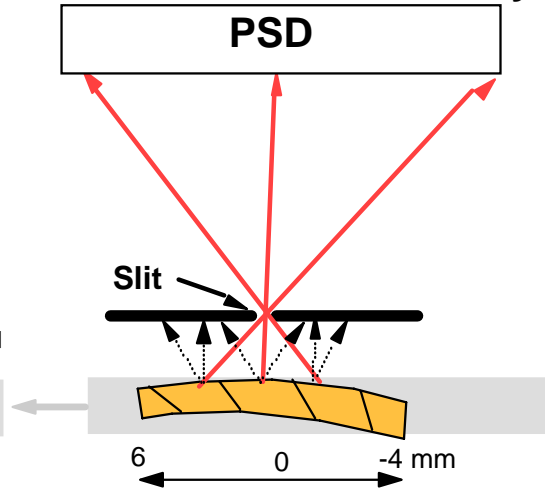
Reconstructed Image -4 mm



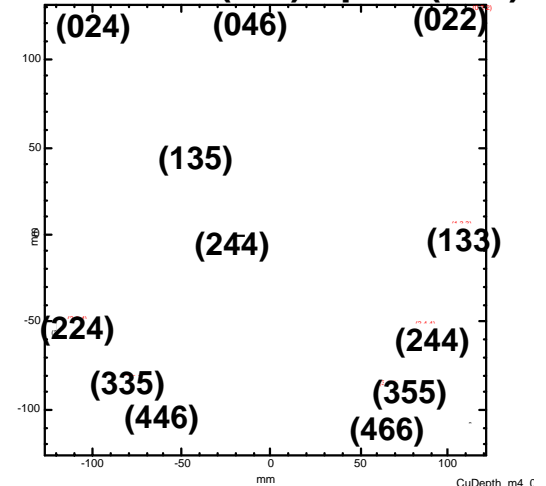
No Slit Geometry



Pinhole-Camera Geometry

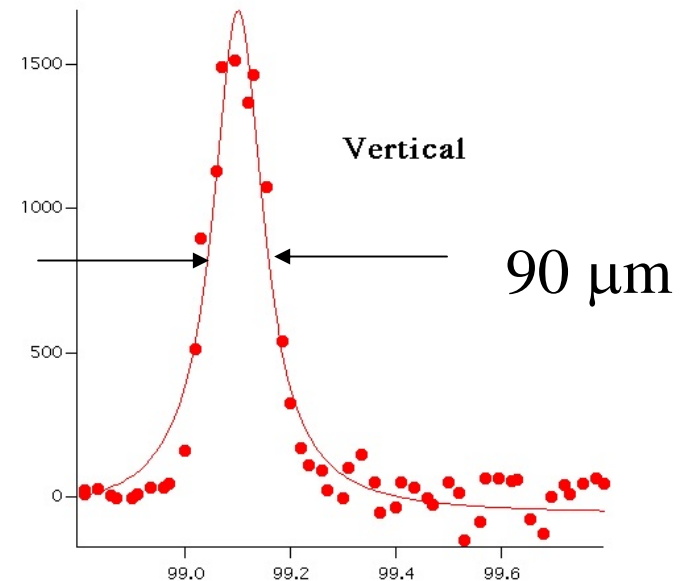
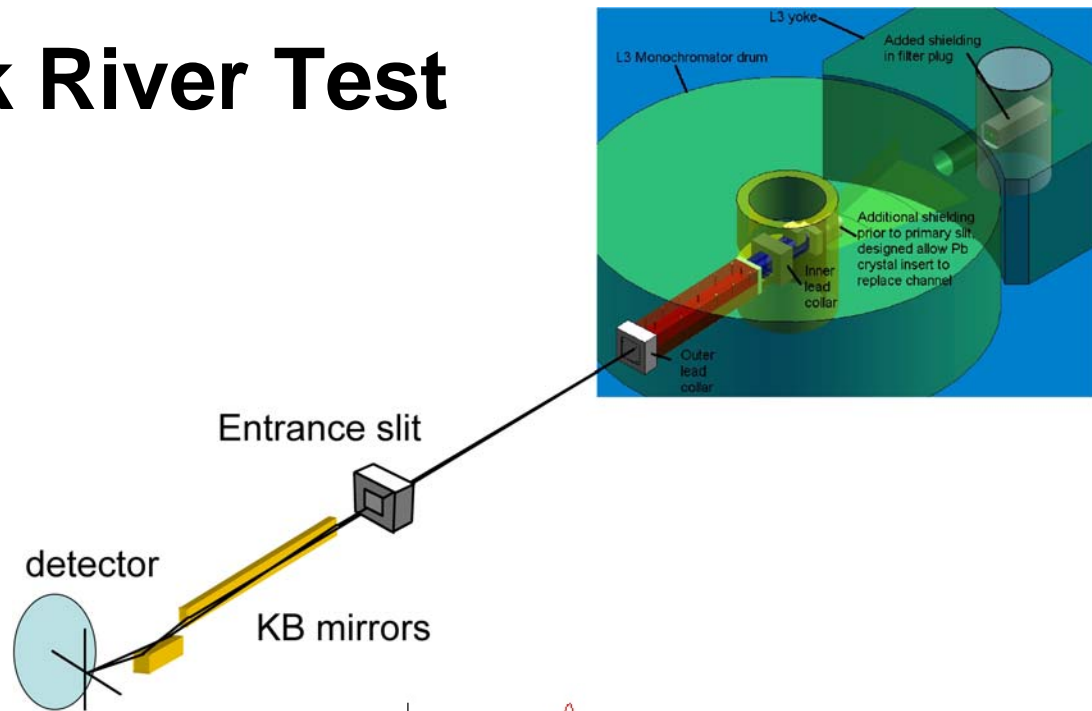


Predicted (hkl) up to (666)



Integrated Chalk River Test

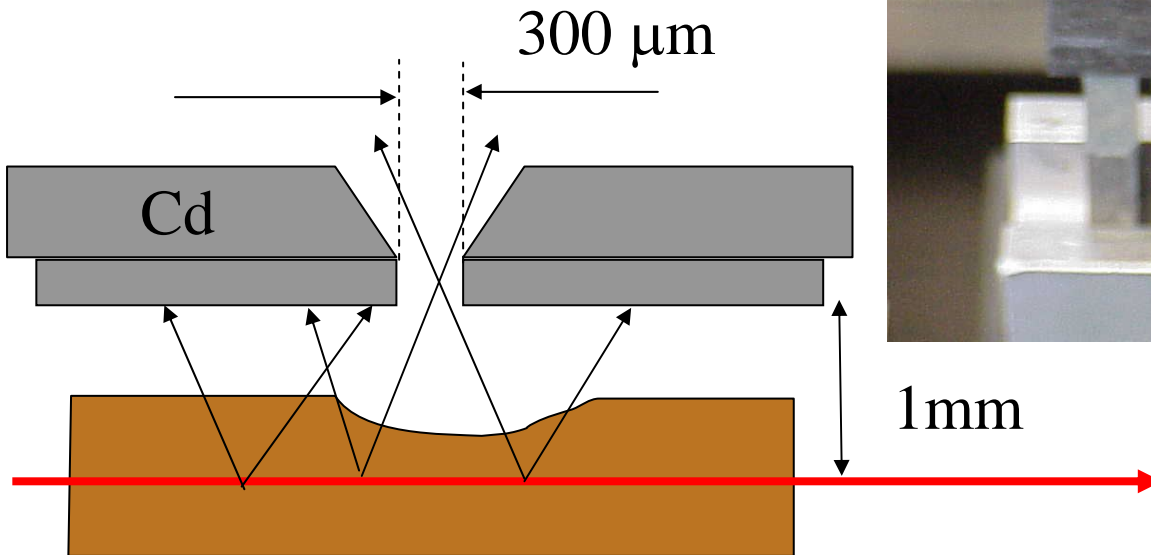
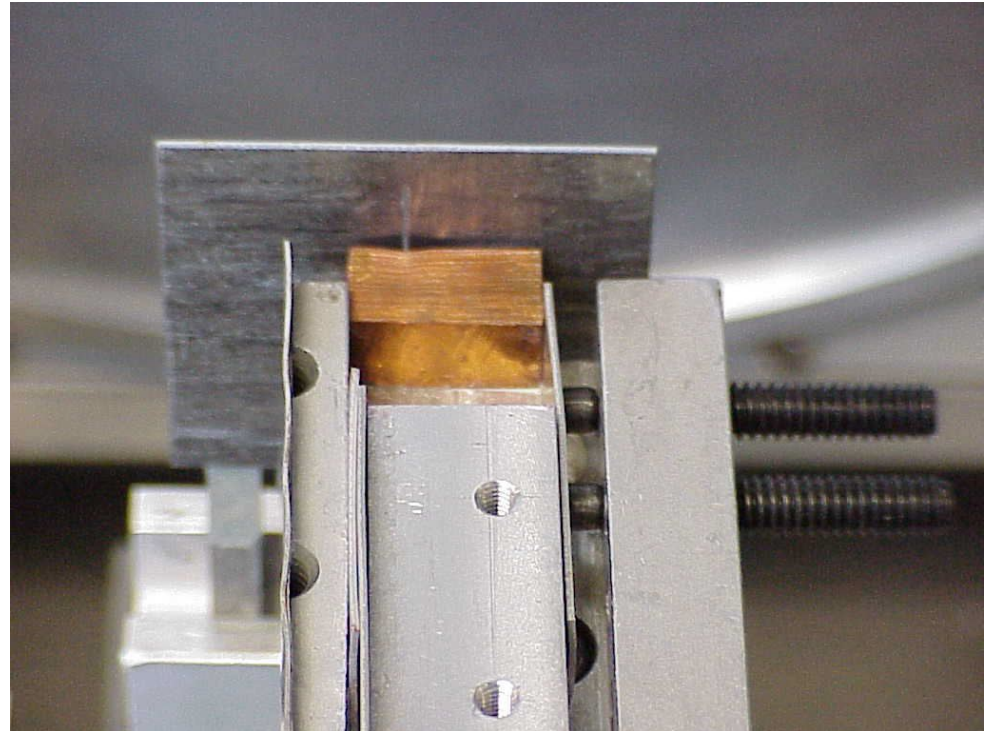
- Deflecting mirror in mono tank suppresses background
- Upstream slits provide source
- Image plate allows microLaue
- Focus spot $89 \times 90 \mu\text{m}$
- “Gain” measured ~ 20
 - Estimated “actual” $\sim 100\text{-}180$
 - “Theoretical” focused” ~ 150
 - “Theoretical” small beam ~ 100



Near theoretical performance

Deformed Cu sample measured with much higher spatial resolution

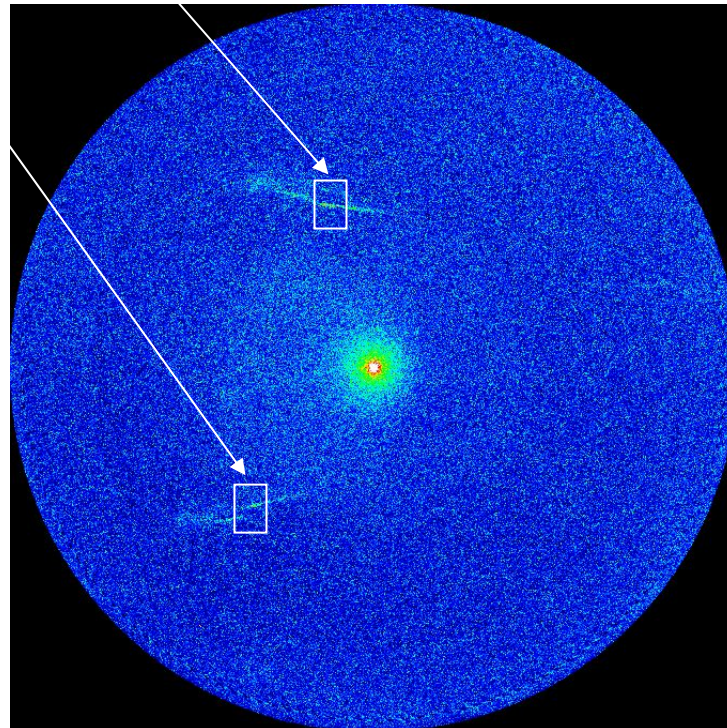
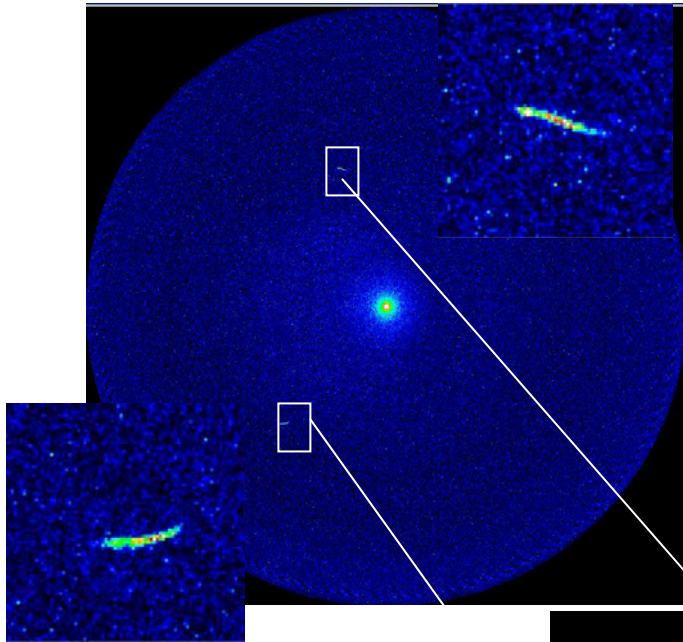
- 200 x smaller area
- 3 x smaller slit



Cu: (2x2binning)

Beam Size: 100 μ m x 100 μ m

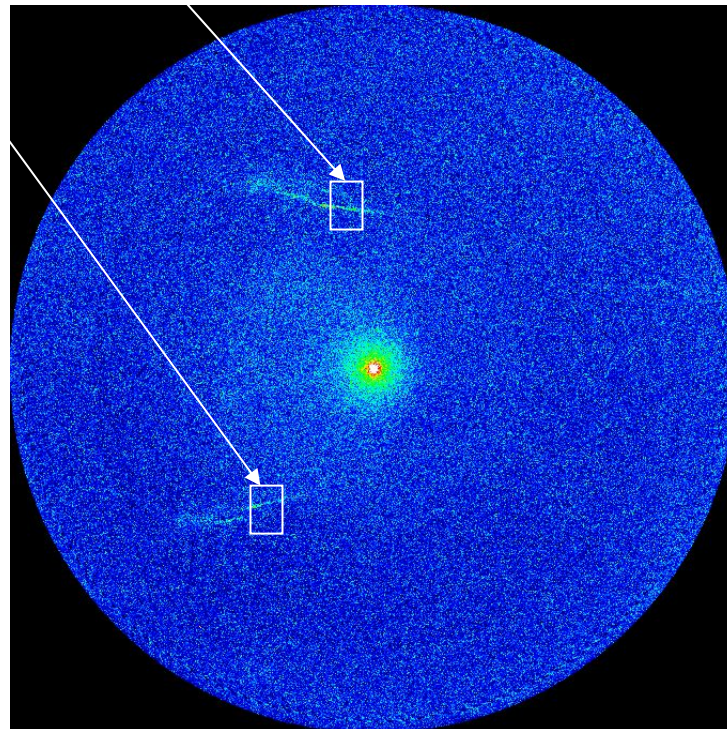
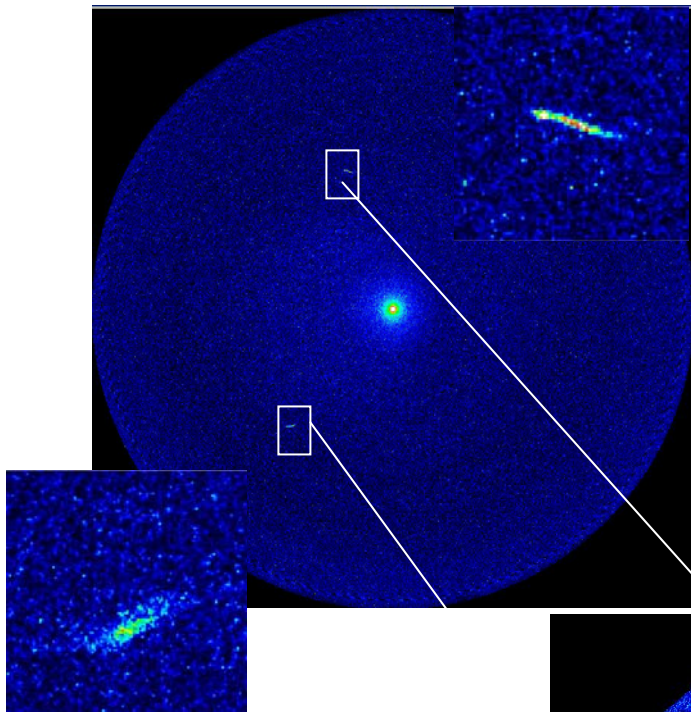
Slit: 0.3 mm



Cu: (2x2binning)

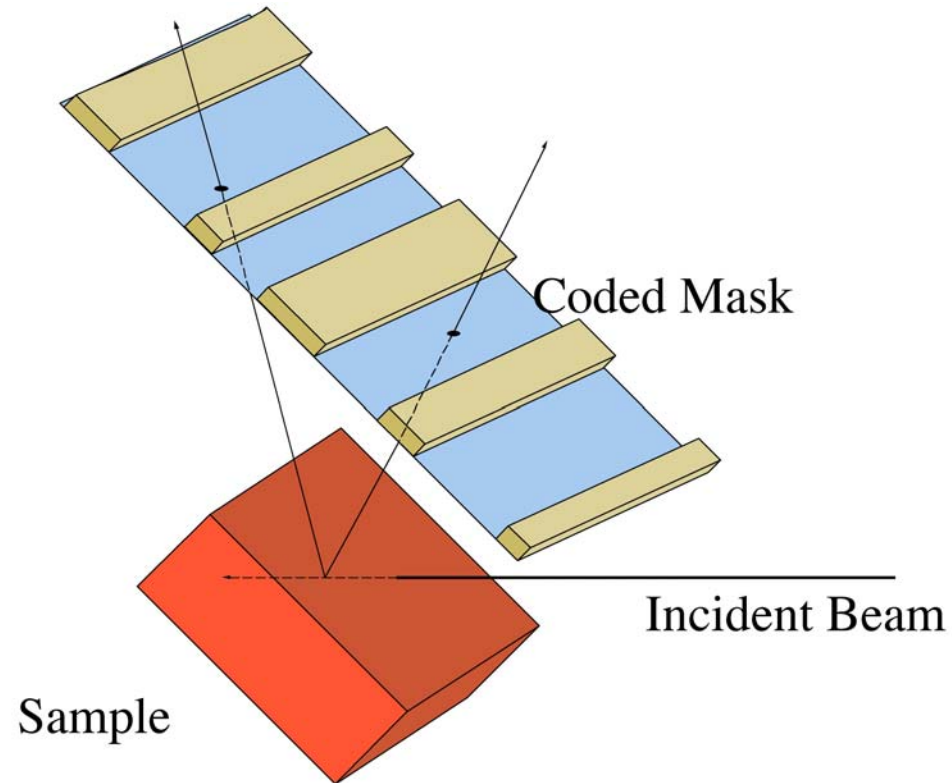
Beam Size: 100 μ m x 100 μ m

Slit: 0.3 mm



Coded aperture methods can further accelerate experiments

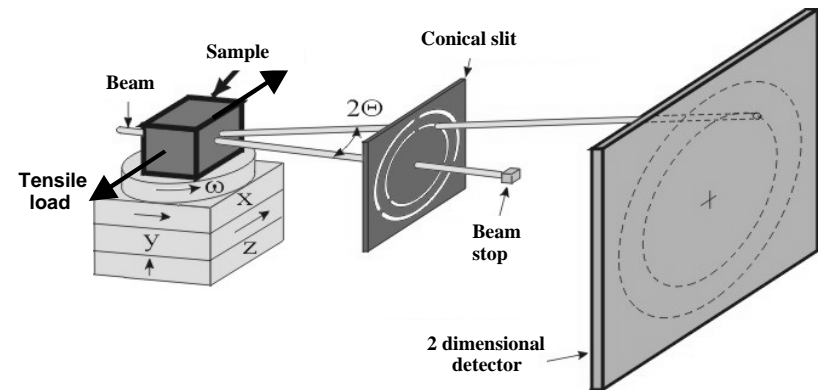
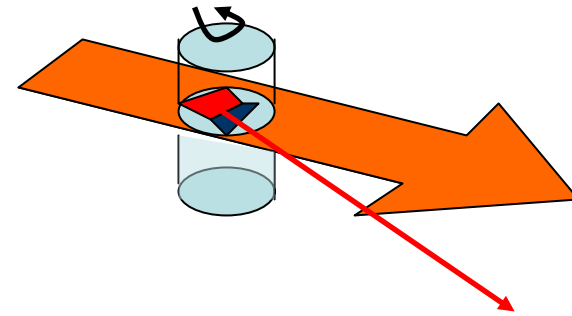
- Additional factor of 10 possible!
- 3D information along line minutes
- 25 μm ?
 - 3 x faster with nested mirrors
 - 10 x faster with SNS/HFIR
 - 10 x with coded



Optimized system 5 x faster than test system with 4 x better spatial resolution in all 3 dimensions!

4D Microscope offers advantages for neutrons

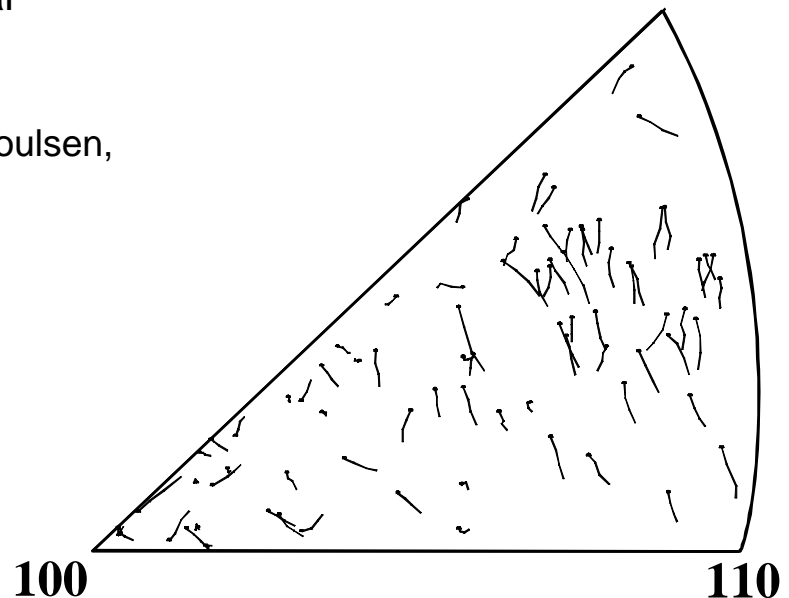
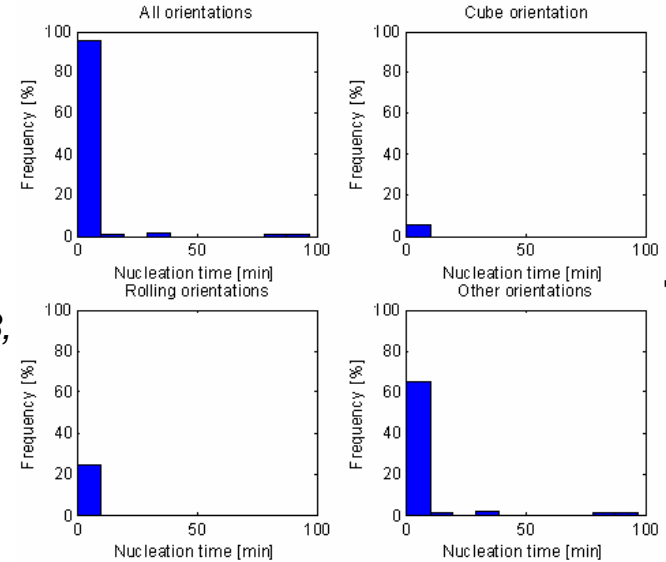
- Singly focused monobeam illuminates numerous grains
 - Bragg condition satisfied by single rotation
- Grain outline determined
 - Ray tracing
 - conical slit
 - Back-projection tomography
- $E > 50$ keV allows deep measurements



FAST!!

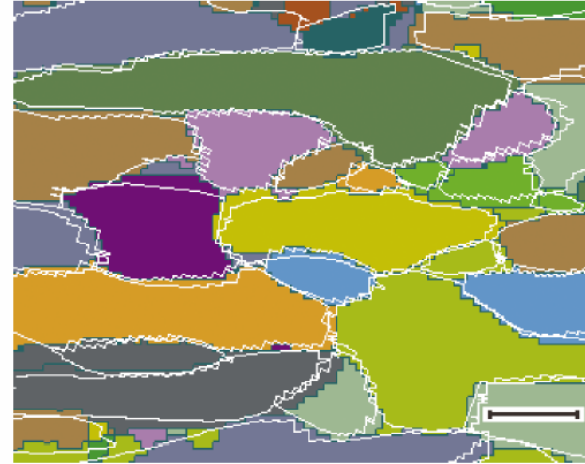
Particularly important for dynamics

- 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).

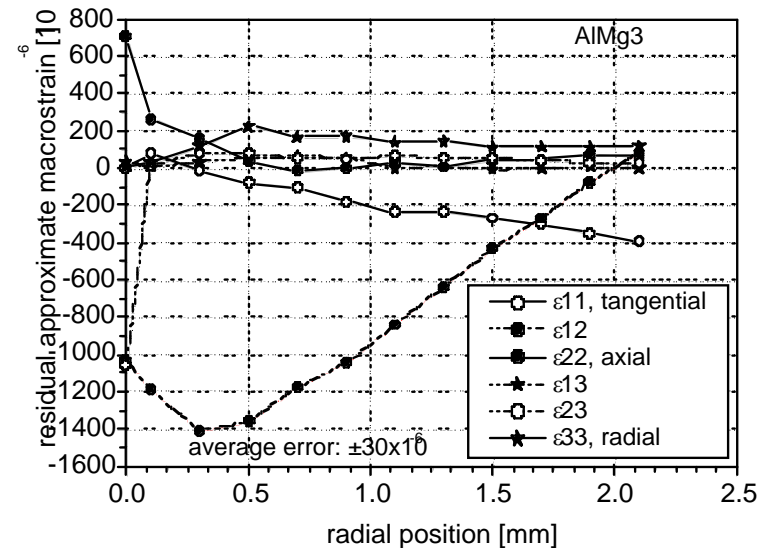


3DXRD Microscope provides additional powerful capabilities

- Grain boundary mapping in coarse grained materials- $5\mu\text{m}$
 - Poulsen et al. J. Appl. Cryst. 34 751-756 (2001)



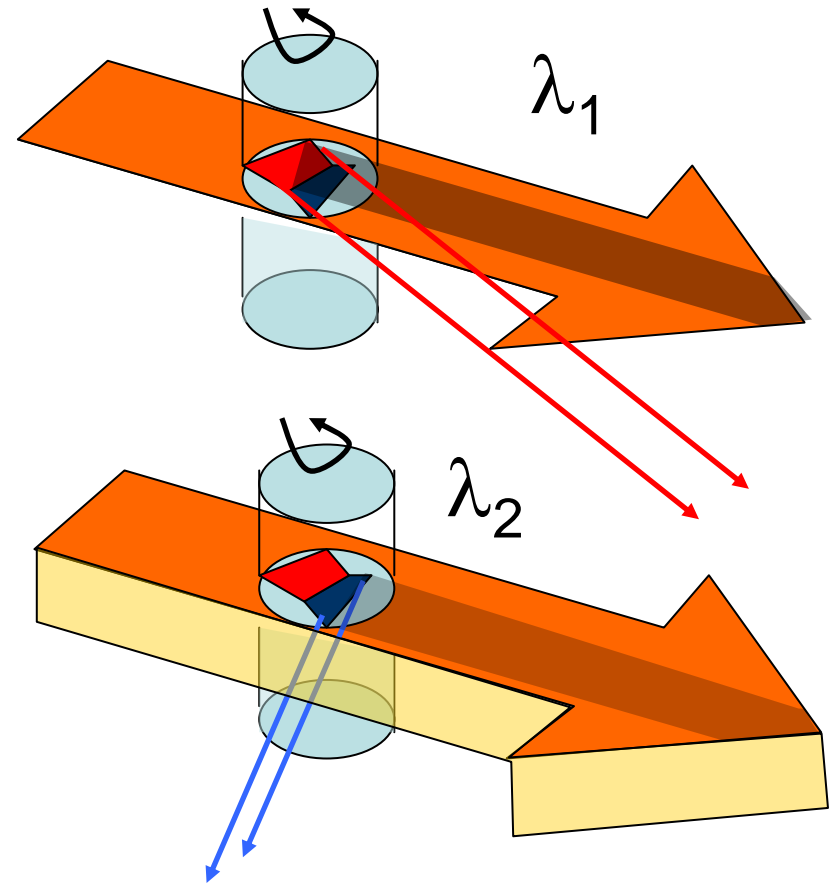
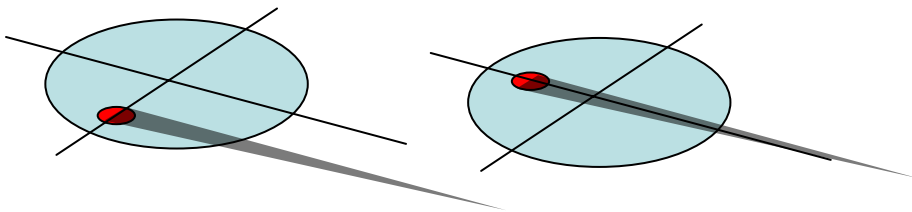
- Single crystal refinement for polycrystals
- Macro/microstrain



Strain tensor elements in torsion sample

Pulsed neutrons offer major advantages

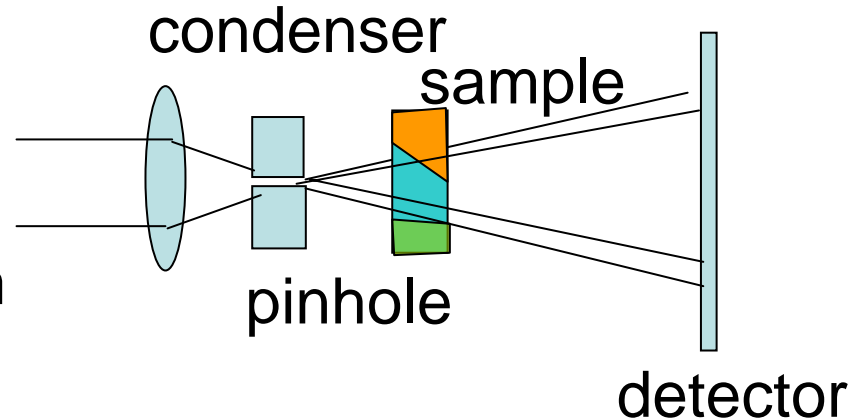
- Simultaneous multiple λ 's combines polychromatic with 4D
- Best S/N
- Large diffraction contrast for direct beam



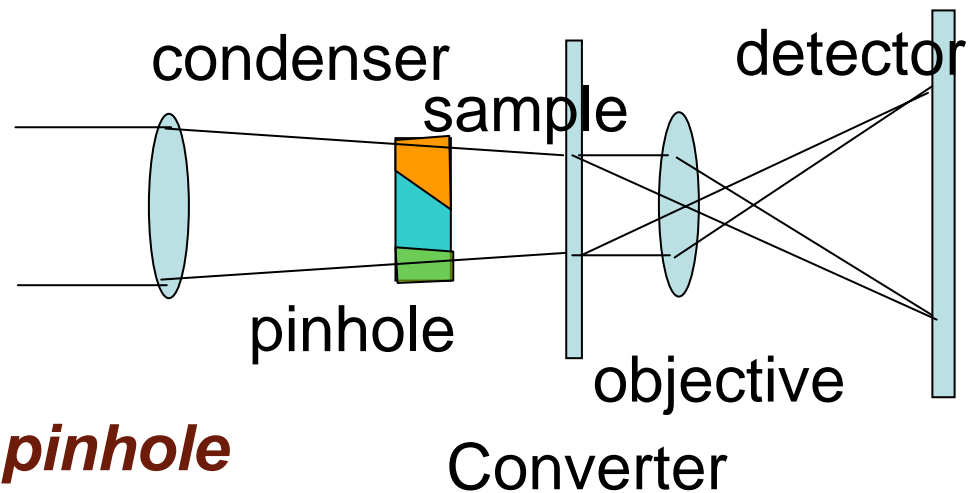
Two images defines source point- detector is the key!

Two approaches

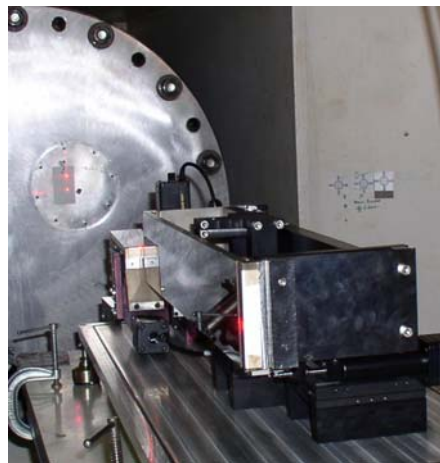
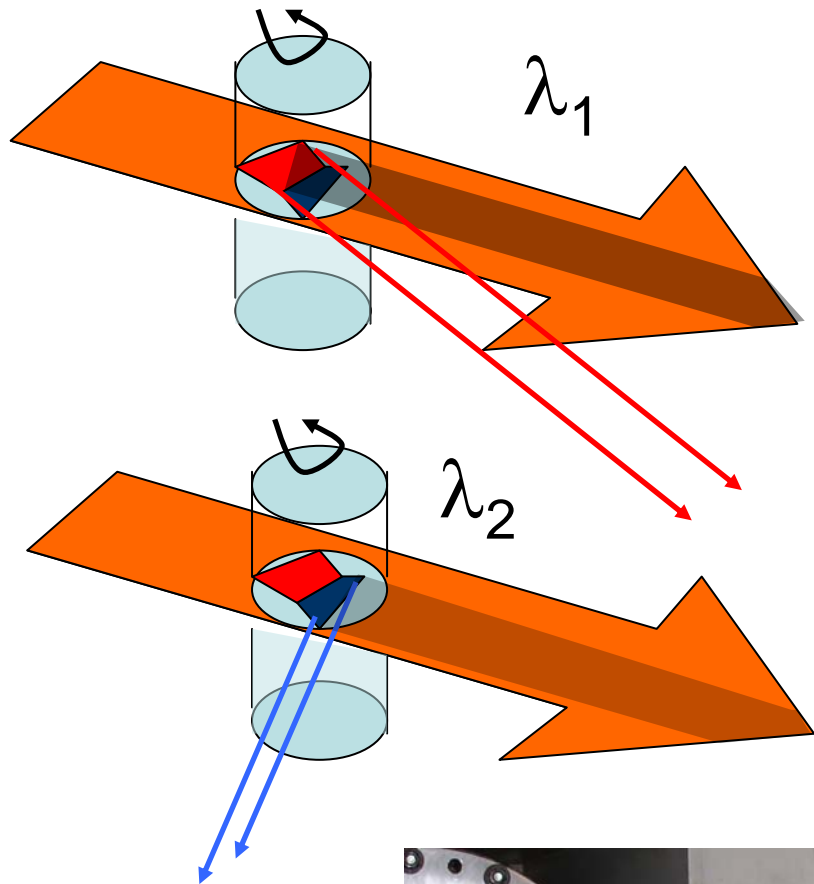
- Projection imaging
 - Simpler optics
 - Collimation a problem



- Magnified imaging
 - Already in use
 - Needs timing!



Can possibly use multiple pinhole methods to increase signal!



Neutrons very precious, not a single one to spare
 But ask the average person- they really couldn't care
 Imaging essential- with crystallography
 We visualize distorted grains-see what we need to see

CHORUS

*Neutron optics- they increase the density
 In a volume -resolved three dimensionally
 Resolve & define- identify- the way that crystals grow
 To guide and investigate- how much theorists know.*

Scanning probes essential-they focus on one grain
 But Imaging much faster- with major temporal gains
 Combining many wavelengths - rotate the sample too
 Will let us see inside samples-give science a new clue

CHORUS

New neutron detectors can change what we can do
 Imaging position- with neutron wavelength too
 And with this new development & it clear as you can see
 Imaging contrast -through crystallography