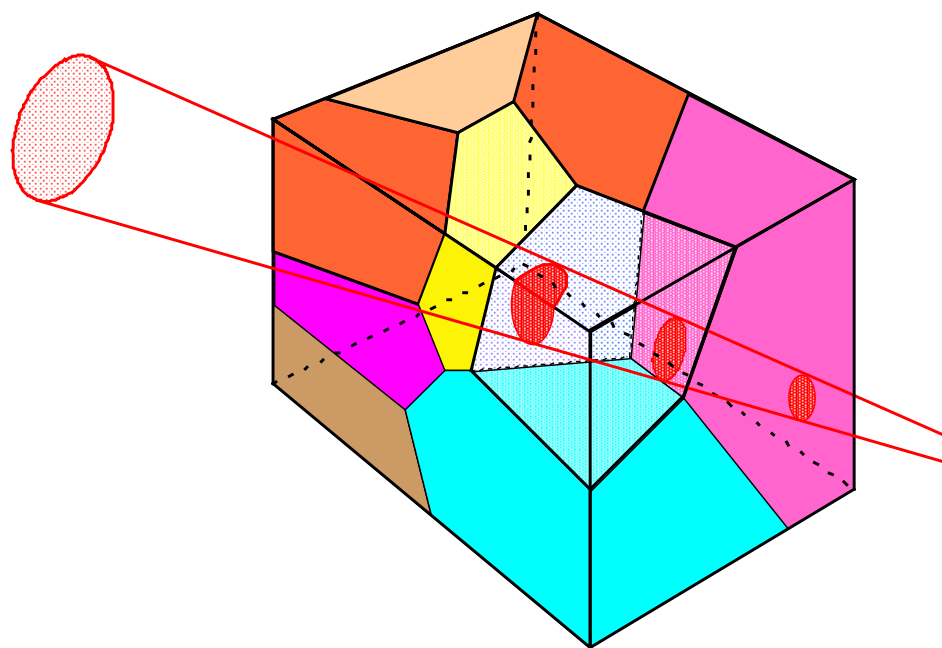


# Frontiers of X-ray Microdiffraction

Gene Ice, Bennett Larson, Jin-Seok Chung, Wenge Yang, Jon  
Tischler, John Budai, Fred Walker  
*Oak Ridge National Laboratory*



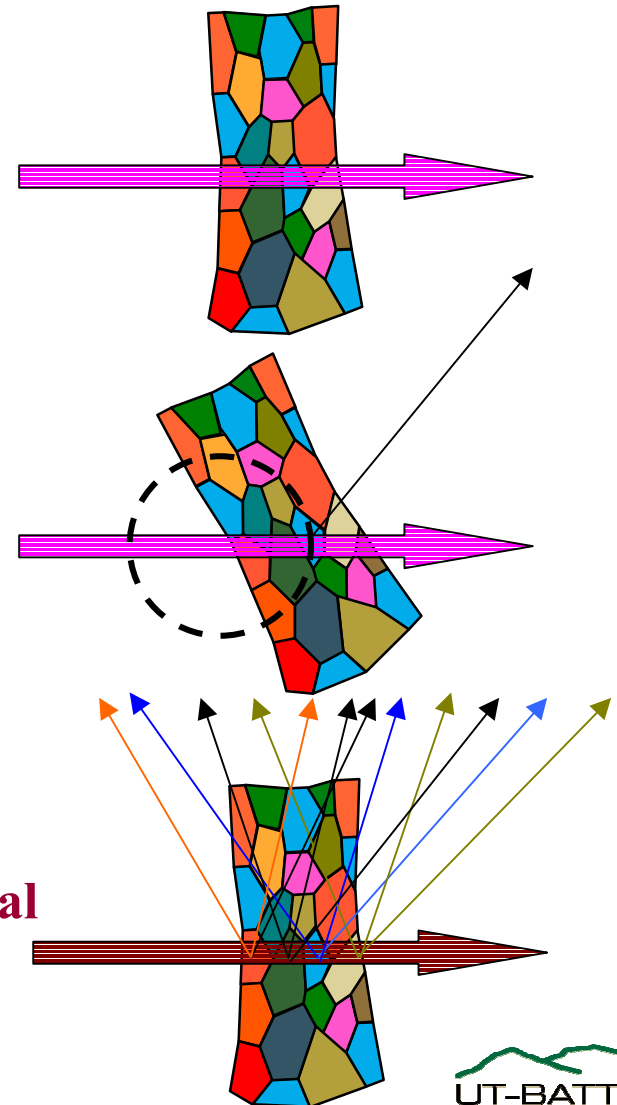
# ORNL has demonstrated a new class of microdiffraction instrumentation based on polychromatic x-ray beams

Solves intrinsic problem with conventional microdiffraction-

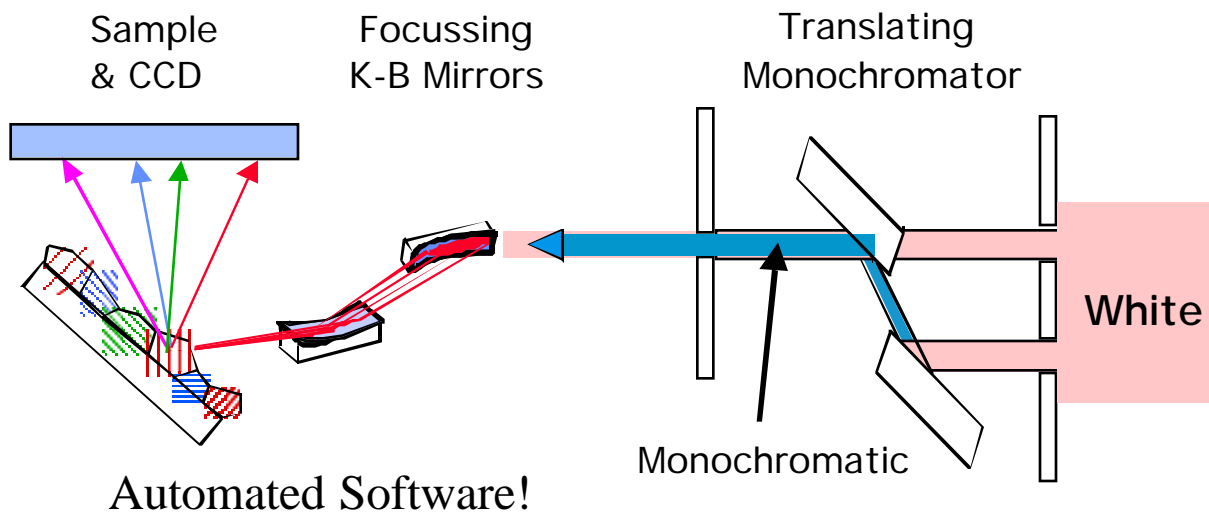
-Sample does not need to be rotated!

Special software required- Can index polycrystalline samples

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



# Polycrystalline microdiffraction station has four key elements

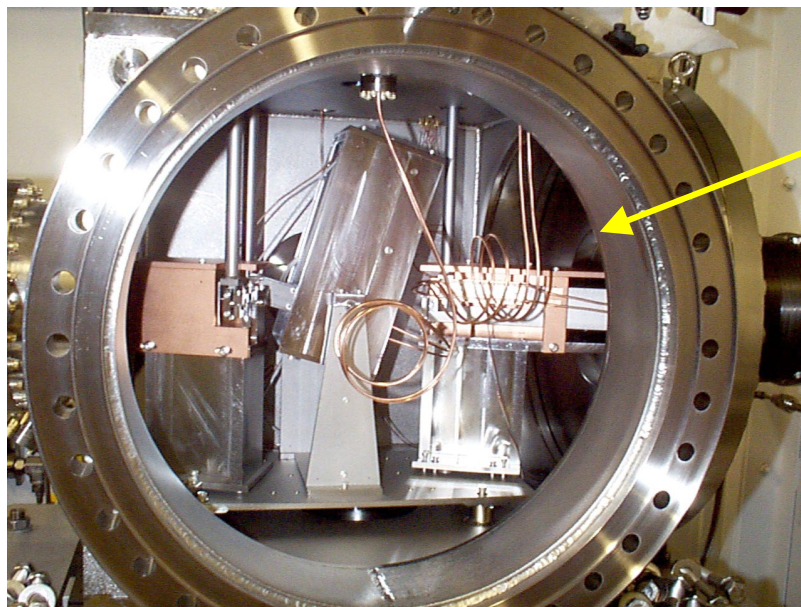
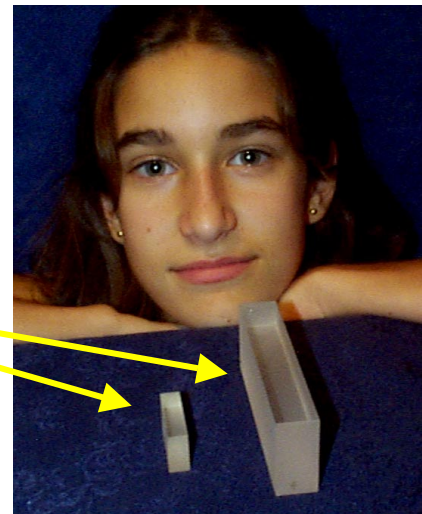


Current 3-D resolution:  $<0.4 \times 0.5 \times 0.7 \mu\text{m}^3$

Current strain tensor resolution:  $\sim 10^{-4}-10^{-5}$

# Technical challenges addressed to make microscope practical

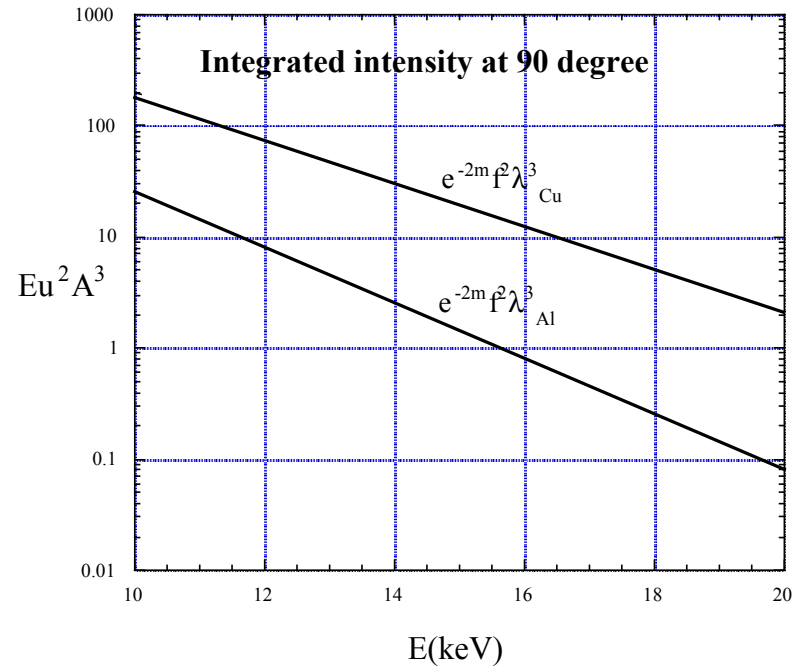
- R&D 100 award winning mirrors



- Unique micro-monochromator
  - Ultra-stable/nondispersive
  - Full strain tensor
- ORNL aquisition/analysis software-
- Differential triangulation microscopy

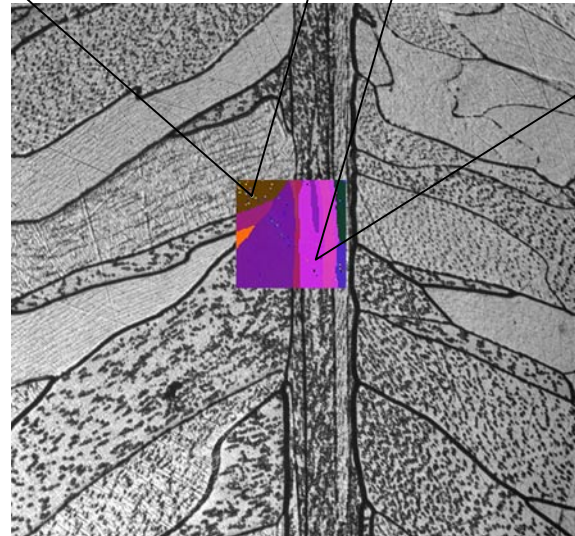
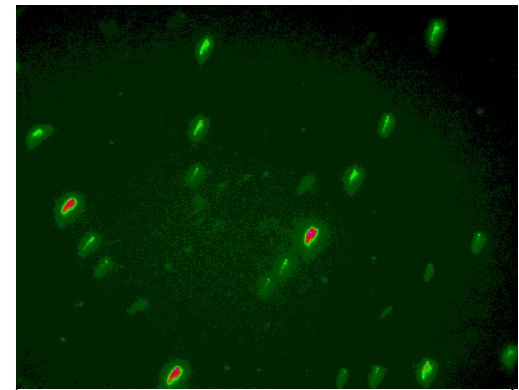
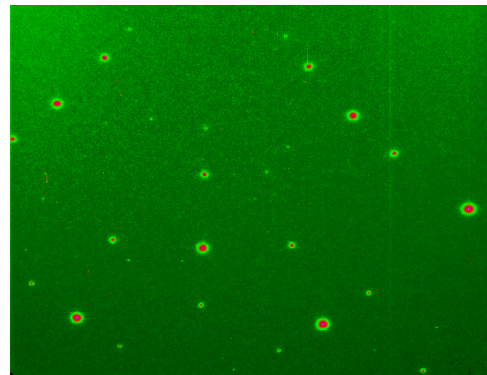
# Optimization complex and can depend on particular experiment

- In  $2\theta=90^\circ$  geometry signal  $\propto E^{-7}$
- Number of reflections  $\propto E^3$
- Absorption length  $\propto E^3$
- Thermal load  $\propto E^{-2}$
- Diffraction limit  $\propto E^{-1}$



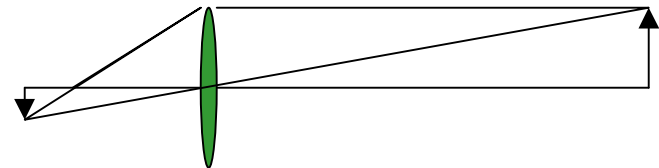
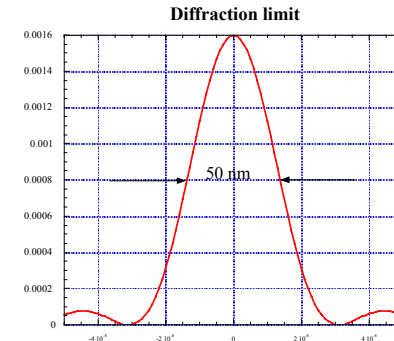
# **Nondestructive in-situ characterization of materials properties.**

- **Penetrating 3-D**
- **Grain orientation/  
morphology**
- **Grain phase**
- **Gain strain**
- **Plastic deformation**



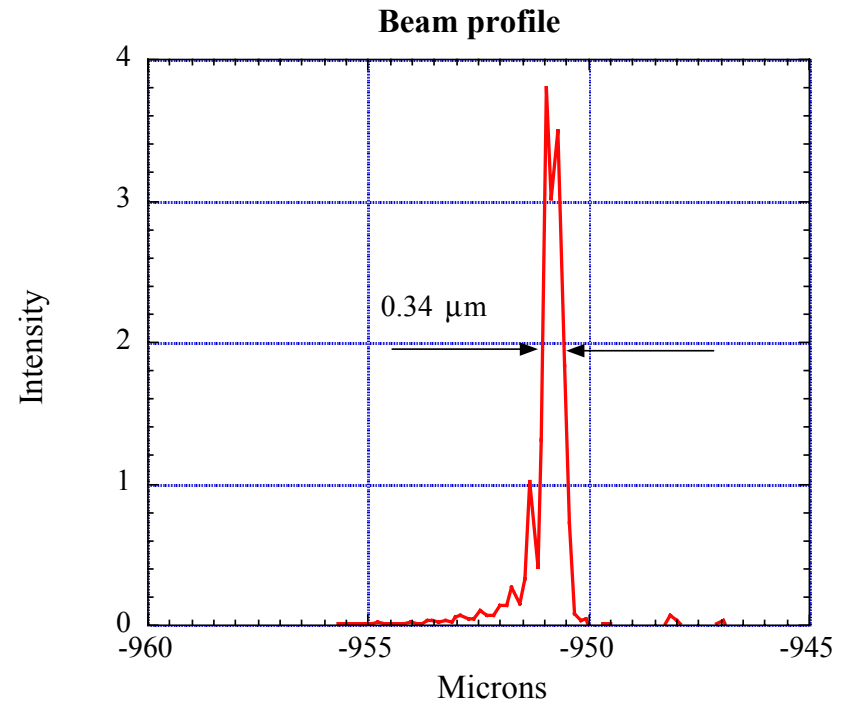
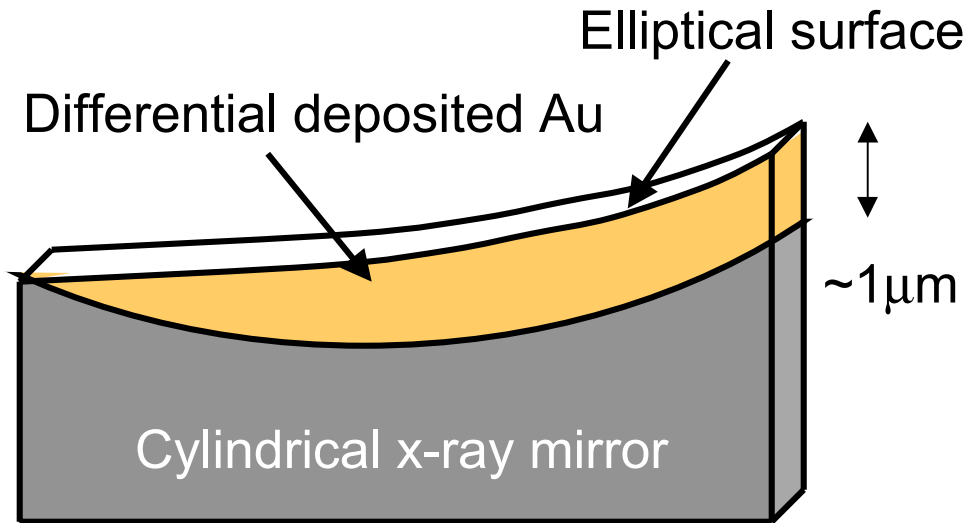
# Current performance is limited by geometrical demagnification/ mirror perfection/windows

- **Diffraction limit ~50 nm**
  - 20 keV x-rays
  - 1-2 mrad convergence
- **Geometrical demagnification ~500 nm**
- **Mirror figure errors ~250 nm**
  - Emerging technologies for superior figure/roughness



# Differential deposition profile transforms cylindrical to elliptical

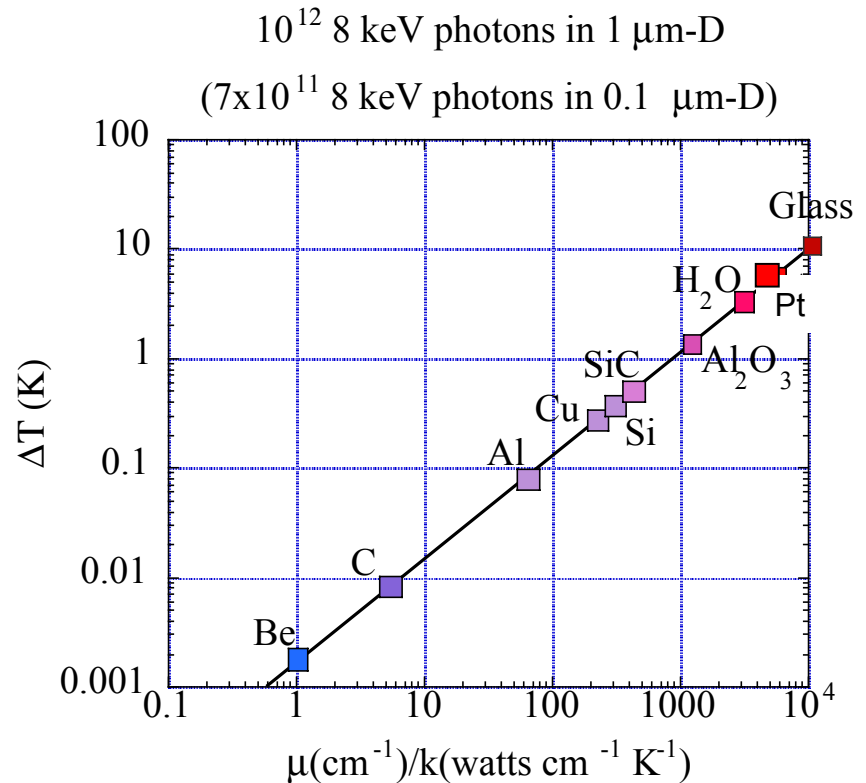
- Can simultaneously correct small imperfections
- Fit final mirror to optimize focal length and tilt





# Thermal loading will become important with ERL and advanced optics

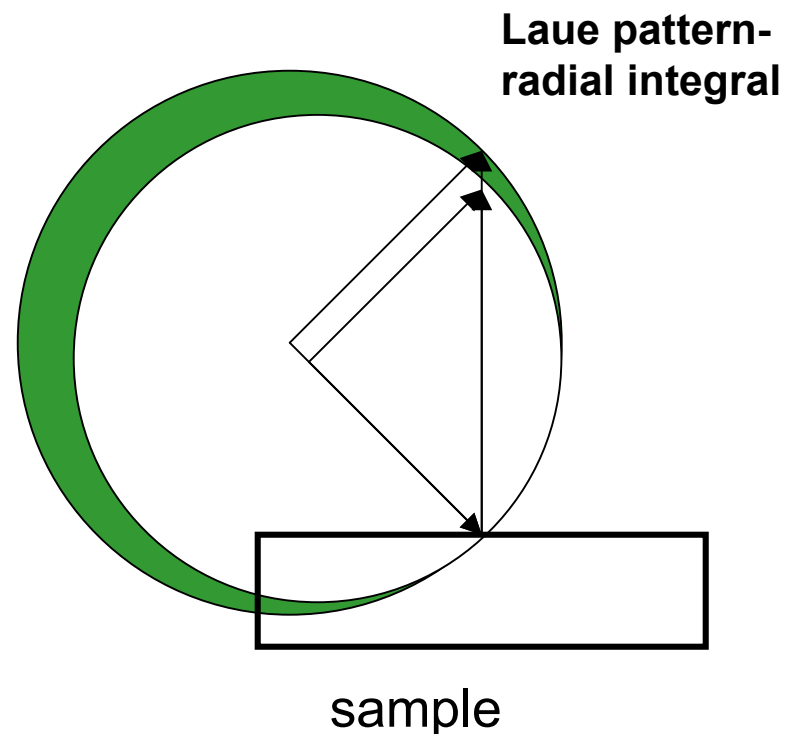
- Surprisingly *benign* focused white beams
  - Linear with power
  - Weakly with focal spot size
- Simple extension of current techniques impractical - less efficient approaches offer new opportunities!



$$\Delta T \sim (P_T)(\mu/k) [1/2 + \ln R_2/R_1] / 2\pi$$

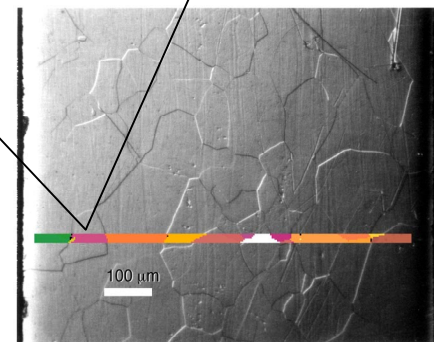
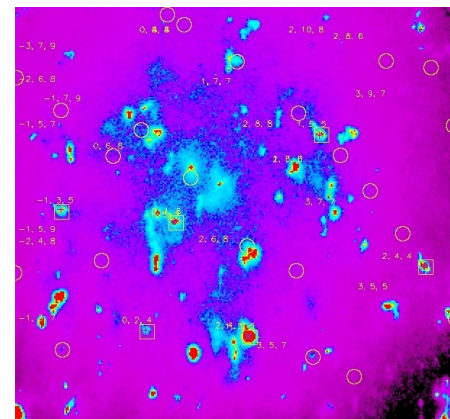
## High performance of ERL will extend technique

- **Energy scan mode**
  - Much better signal-to-noise
  - Complete reciprocal space
- **Diffraction limited focus**
  - ~50 nm radius will reduce thermal load by 100
- **High energy**
  - Deep 3-D measurements



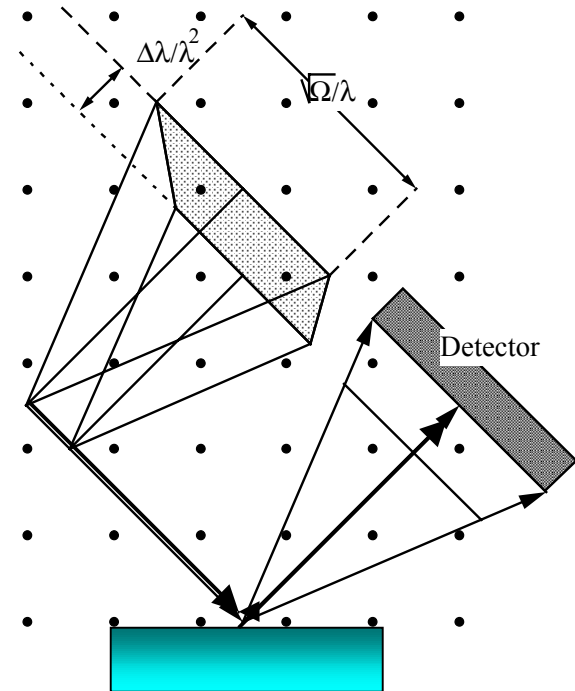
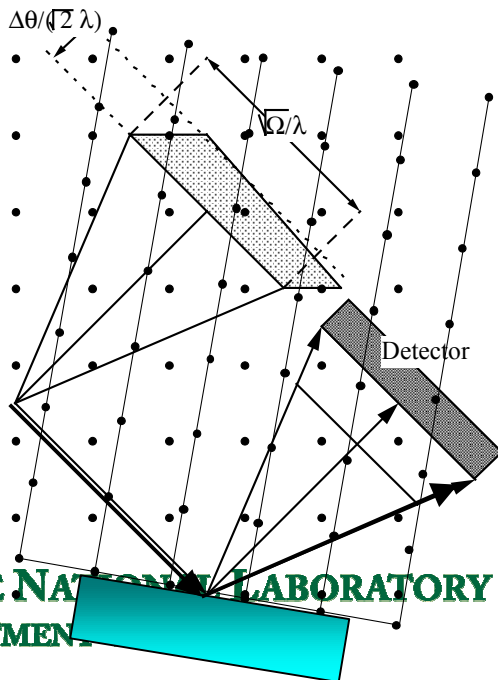
# High performance of ERL will open up new regimes

- **Better spatial resolution**
  - Nanoscale materials
  - Fracture (local environment)
  - Nucleation and growth
  - Fine grain mosaic materials
- **Better signal-to-noise**
  - Fluorescing materials
  - Highly deformed/mosaic materials
- **High Energy**
  - 3D in high Z materials



## Number of reflections detected can be controlled

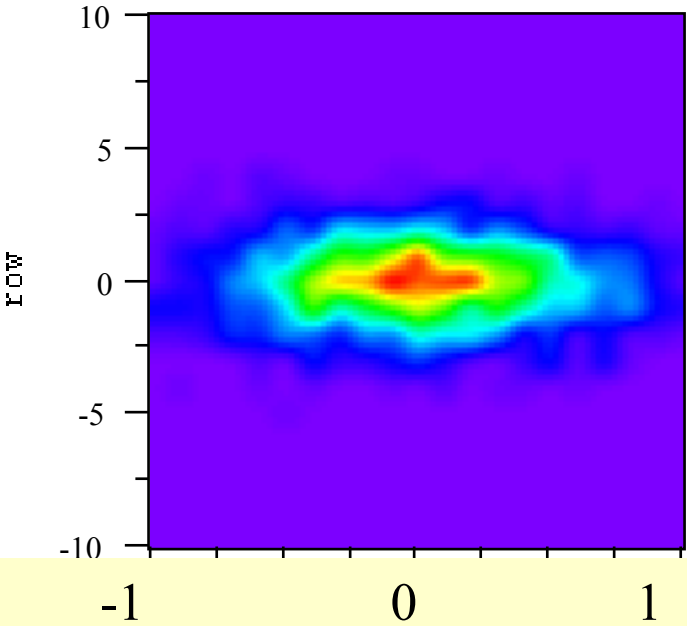
- Increase detector solid angle  $\sim 1$  steradian
- 1 mrad  $\rightarrow$  1/3 reflection
- 10% bandpass @ 20 keV  $\rightarrow$  4-5



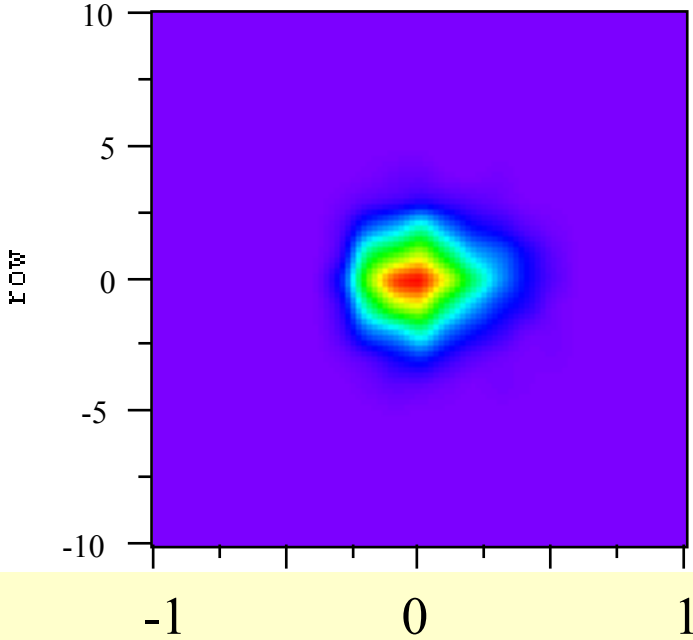
8° rotation = 10% bandpass but blurs resolution for 4μm thick sample.

# Large horizontal beam size can be mitigated with intermediate aperture

1 mm aperture

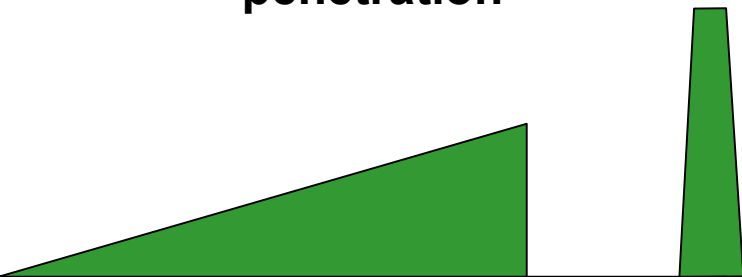


0.1 mm aperture

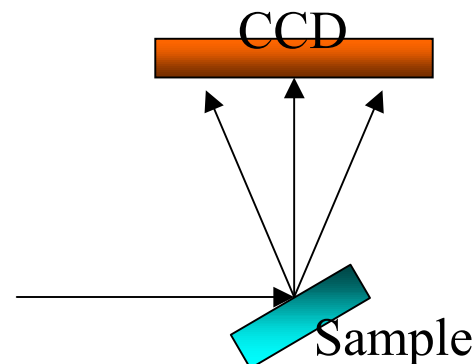
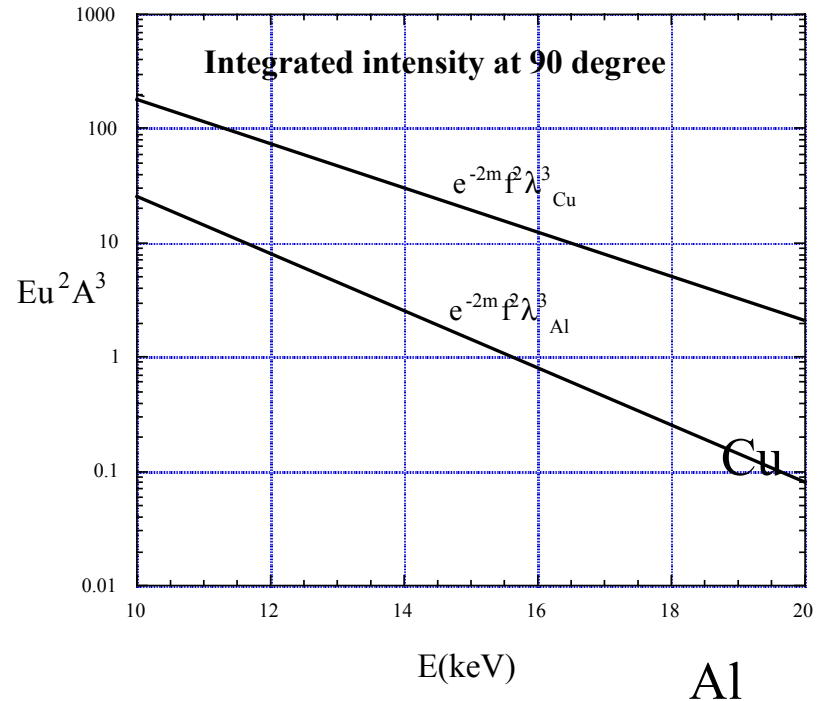


## Ideal operating energy/conditions is a compromise

- High energy  
→ many reflections
- Low energy → better reflectivity
- High E → lower thermal load
- High E → deeper penetration



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