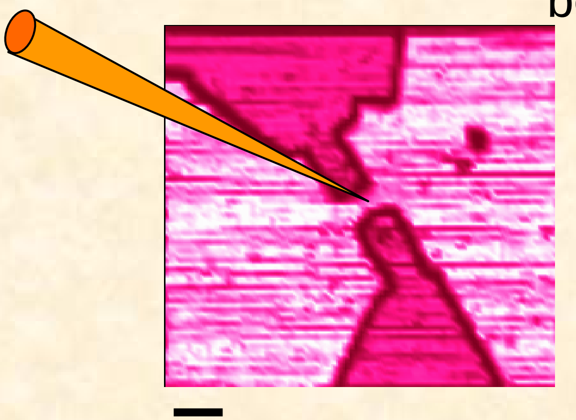
KB Mirrors and Nanobeam Materials Science

Gene Ice

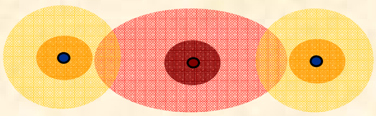
Oak Ridge National Laboratory

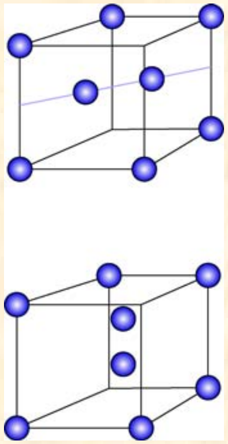
Tiny achromatic/
polychromatic
beams



10µm

Composition
chemistry
bonding





Atomic
structure and
defects

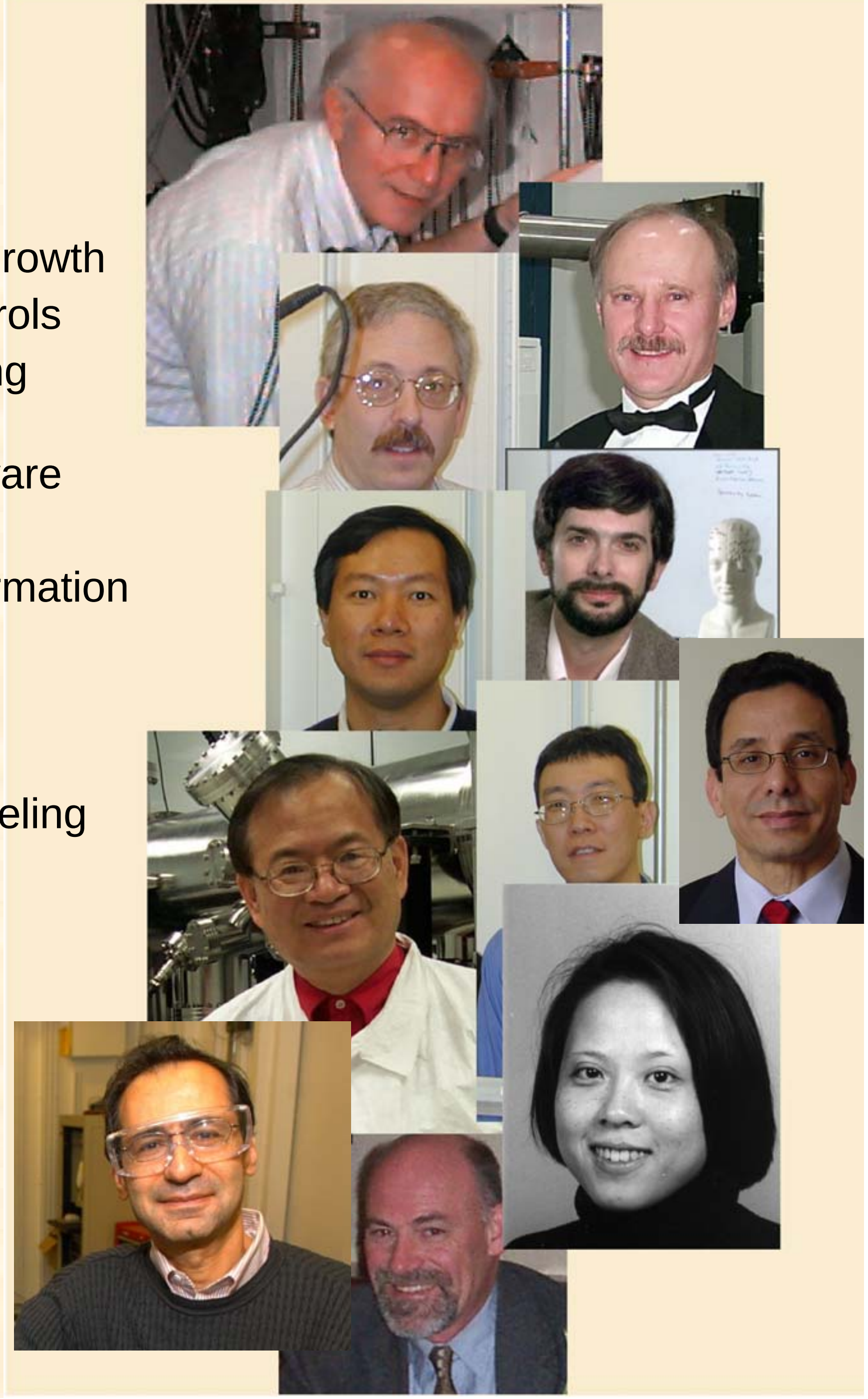
New Science Opportunities with Nanometer-Sized ERL X-ray Beams

Team of ORNL/APS scientists

- **ORNL**
  - **Bennett Larson-** Co-principle DAXM/3D
  - **John Budai-**Epitaxial films and 3D grain growth
  - **Jonathan Tischler-** nanobeams and controls
  - **Wenge Yang-**Mesoscale deformation using nanoindentation/software
  - **Eliot Specht** -automated orientation software
  - **Wenjun Liu-**Grain boundary networks
  - **Judy Pang**-in-situ 3D polycrystalline deformation

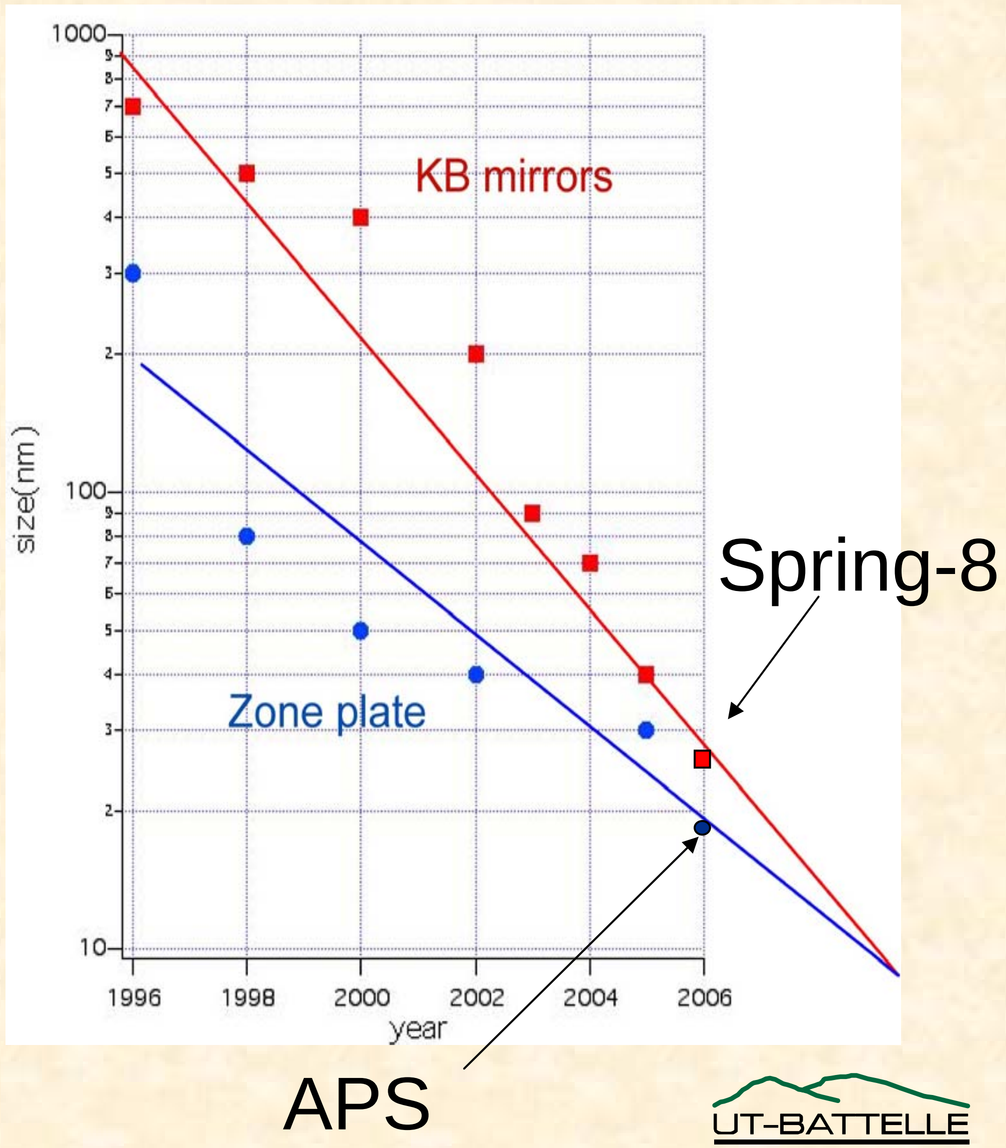
- **APS**
  - **C. Liu-** profile coating
  - **A. Macrander-** advanced techniques/modeling
  - **A. Khounsary -** advanced focusing optics
  - **L. Assoufid-** metrology

- **Canadian/Australian collaborators**
  - **S. McIntyre**
  - **A. Gerson**
  - **R. Feng**



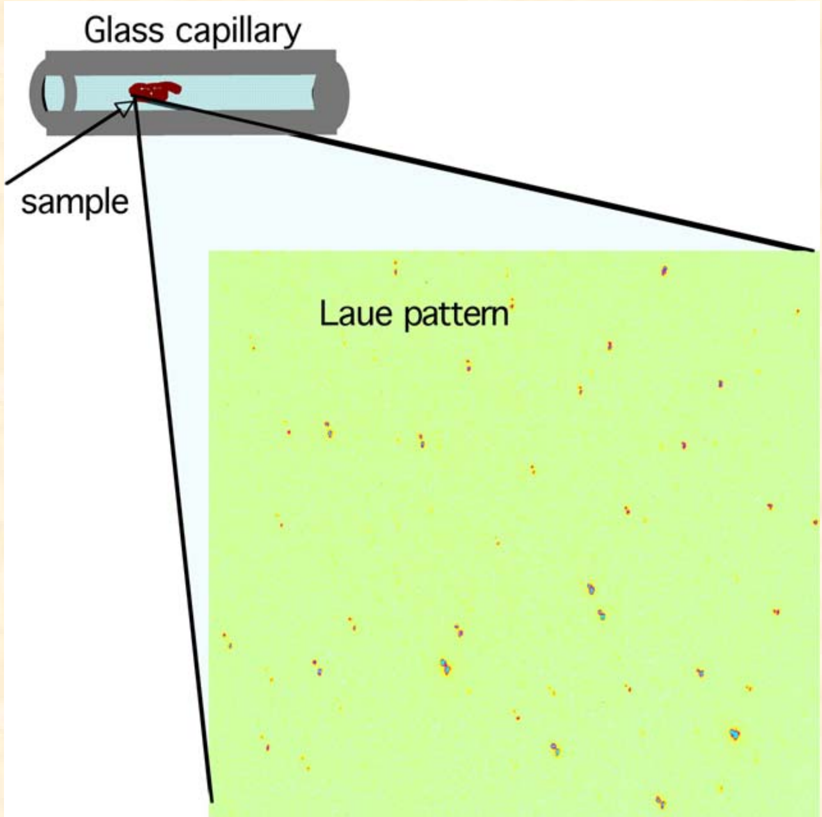
Small beams change everything!

- **Mapping!**
  - **Chemistry**
  - **Local structure**
  - **Heterogeneity**
  - **Defects/ correlations**
- **Combinatorial/ ease of sample preparation**
- **ESRF/APS fielding ever more microbeam capability**
- **International race to make small hard x-ray beams**



Polychromatic/ achromatic *small* beams are particularly important

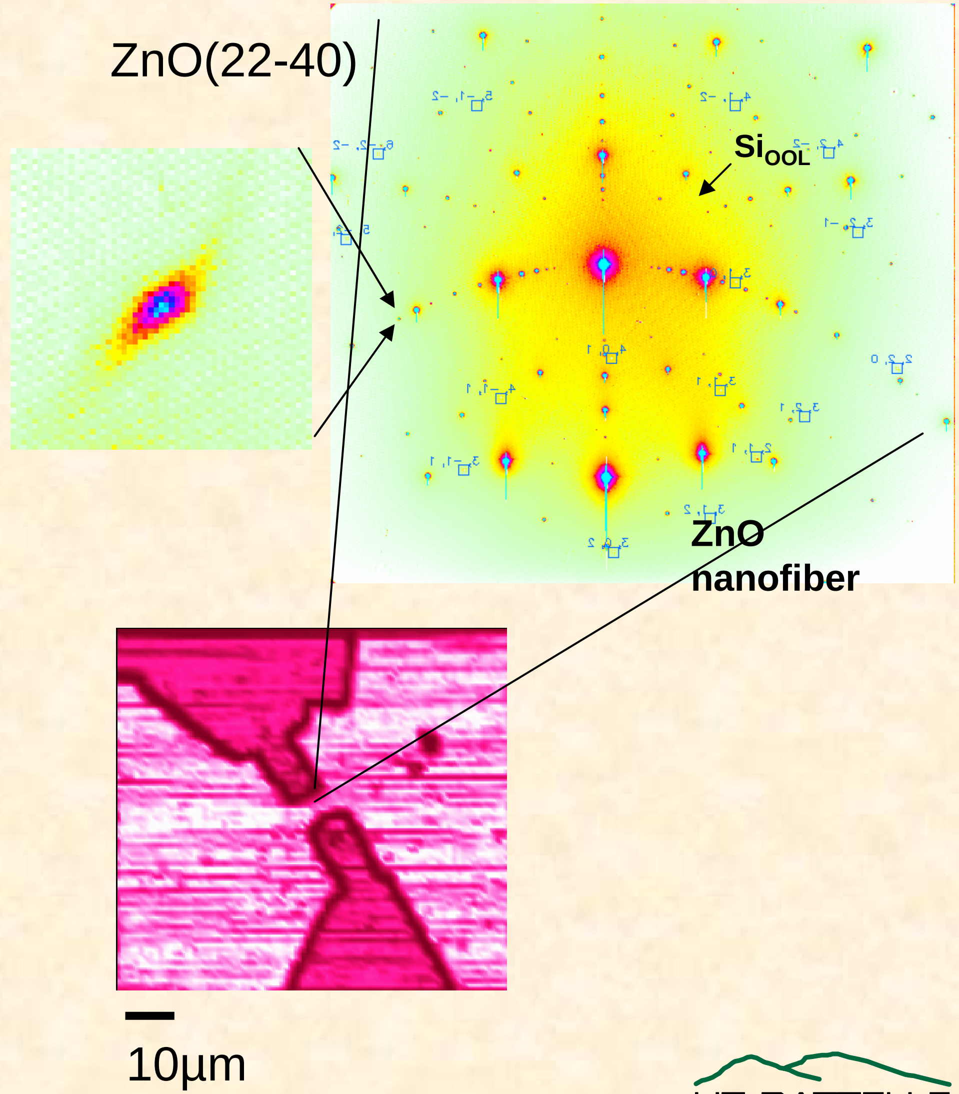
- **Spectroscopy**
  - **Adjustable bandpass**
  - **Rapid scans**
- **Diffraction**
  - ***No* sample rotations!**



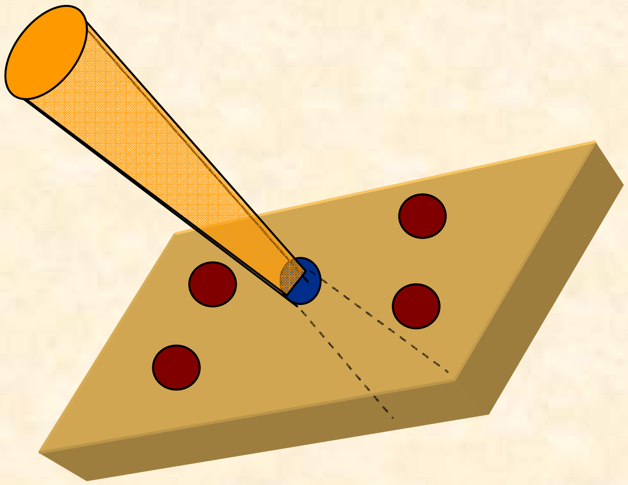
At the nanoscale -*really* important!

Goal of nanobeams is <u>*new*</u> science

- **Big beams *can* study small samples**
- **Electrons *can* penetrate nanometers into samples!**
- **Electrons only penetrate so far-**
- **Signal-to-noise poor**

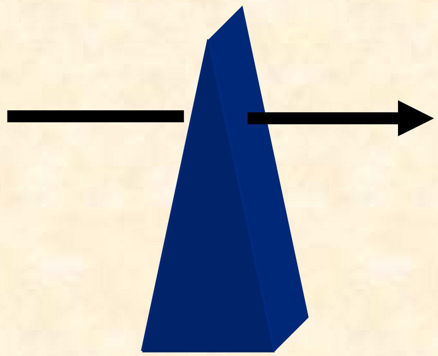


3 motivations for 1-nm probe



- **Improve signal-to-background for individual nanoparticle**
  - **Isolate from matrix/ surrounding particles**

- **Resolve nano regions**

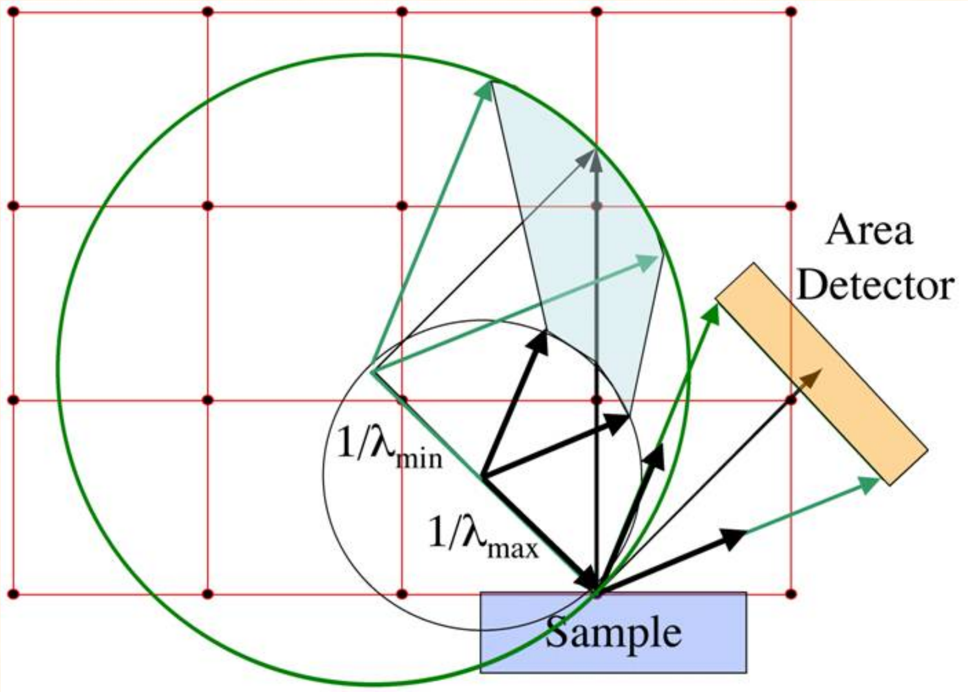


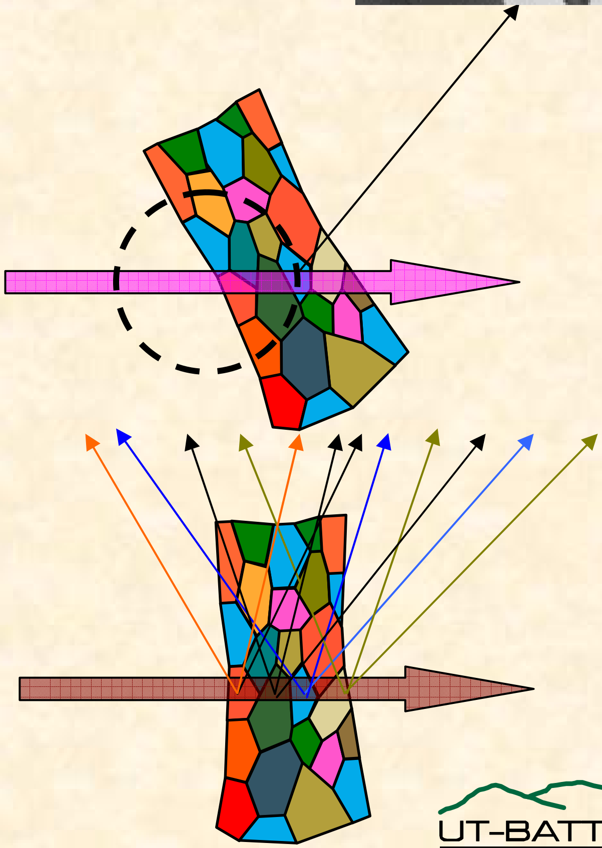
- **Look at small volumes below surface along boundaries etc./ correlations**

Probably essential for science!

Spirit of Laue Diffraction embedded in thinking about advanced nanoprobe

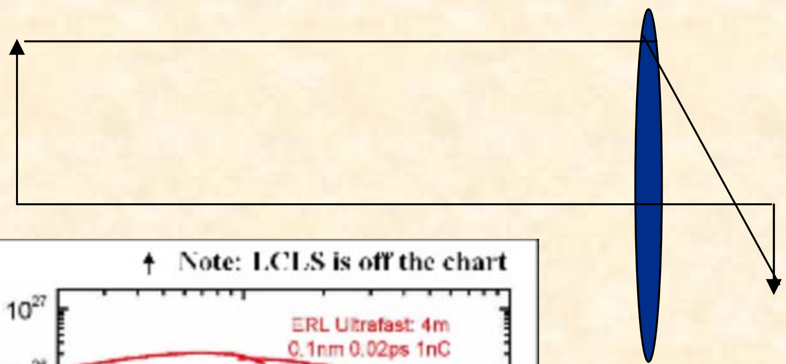
- Polychromatic beam solves intrinsic diffraction problem; no sample rotations!

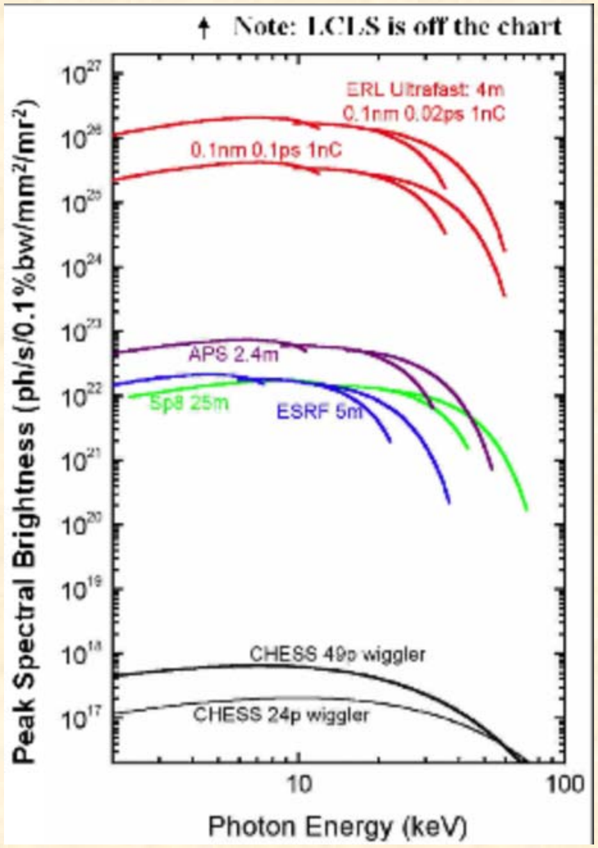




Proposed ERL provides 2 huge advantages for KB mirrors

- **Small source size simplifies x-ray optics**
  - **Coherent beams**
  - **Improved geometrical demagnification**
- **High brilliance**
  - **Figure of merit for nanodiffraction & nanospectroscopy**
  - **Reasonable count rate for small beams!**





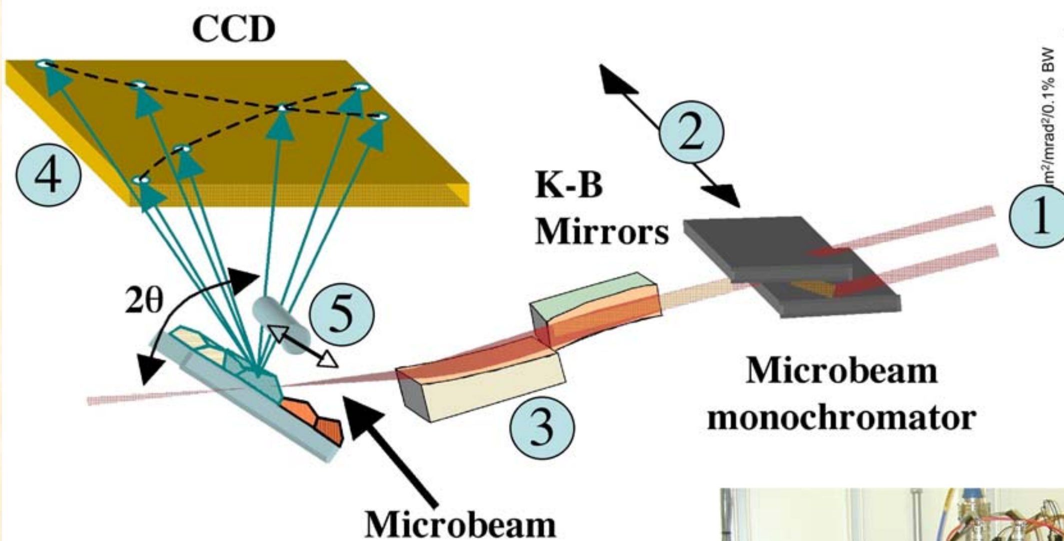
New science opportunities with micron-sized source

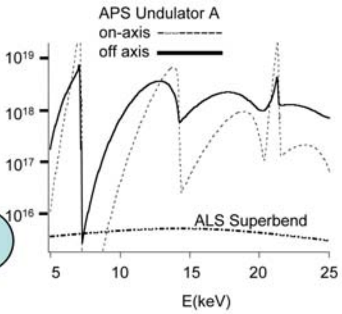
- **Extend Polychromatic Diffraction**
  - **20nm probe will extend current science**
  - **μs Laue images$\rightarrow$3D images with TV resolution in few minutes**

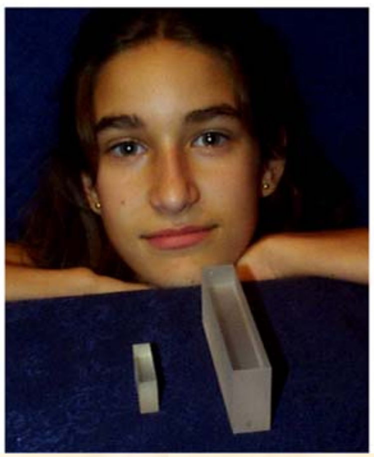
- **Nondispersive scanned probe**
  - **Structure identification of tiny volumes inside materials**
  - **Grain-boundary measurements**
  - **Defect structure**

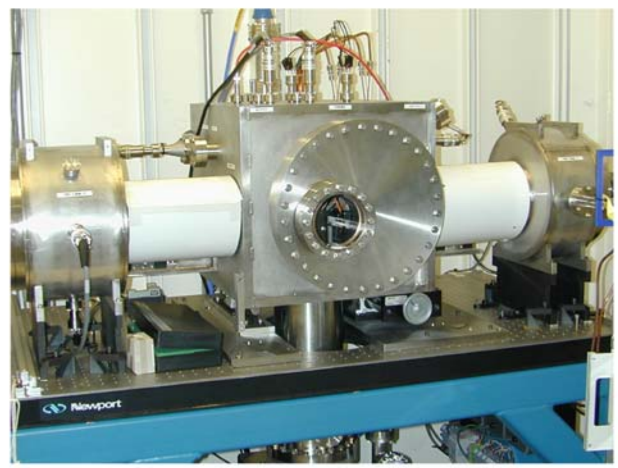
- **Single atom spectroscopy?**
  - **Chemistry, bonding of single or few atoms**

3-D X-ray Crystal Microscope has 5 key Elements



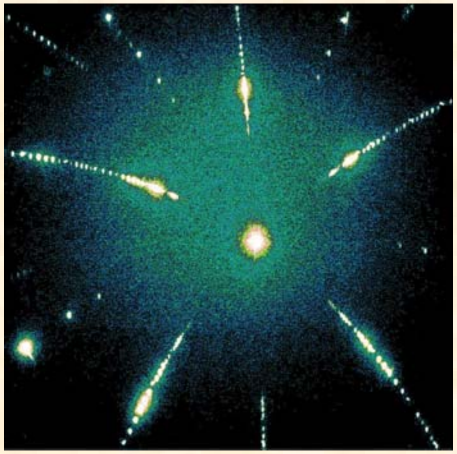




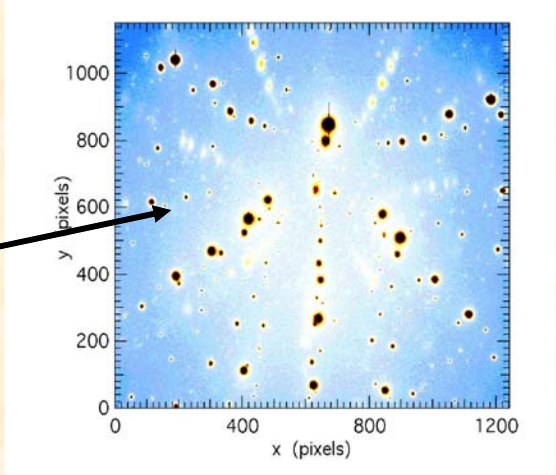


<0.3 x 0.4 x 0.7 μm^3
strain~10^{-4}-10^{-5}

Polychromatic microdiffraction characterizes 3D materials distributions

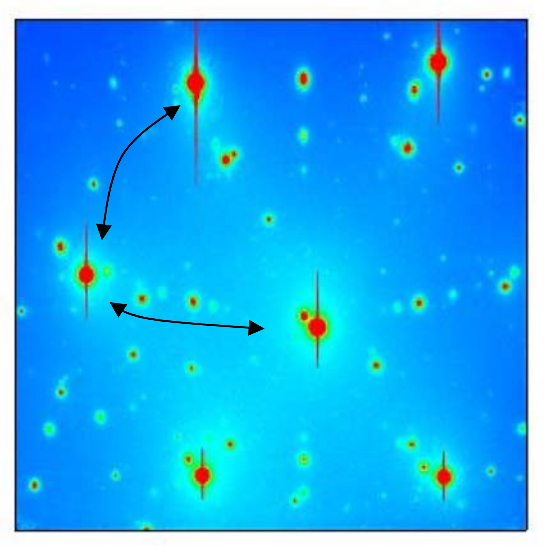


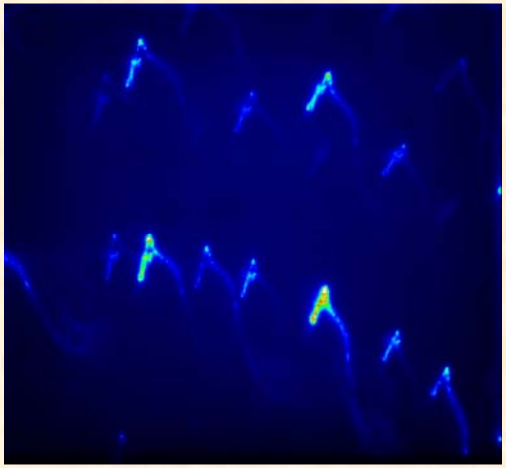
Phase/phase boundaries



Texture (0.01º)/grain boundaries

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





Dislocation Tensor

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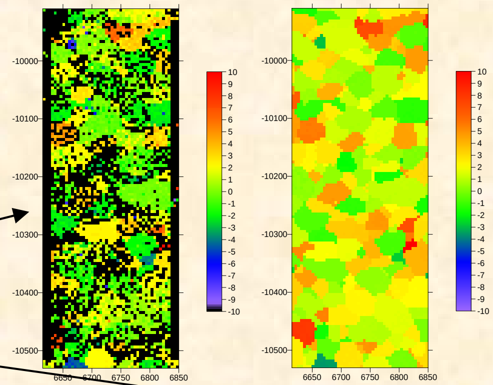
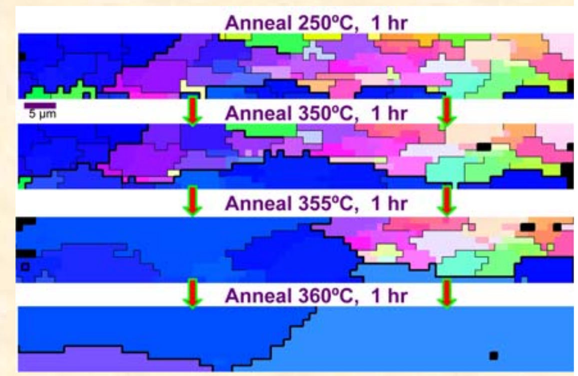
U. S. DEPARTMENT OF ENERGY

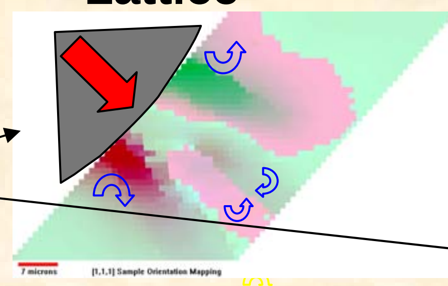
Science of 1st generation 34-ID-E compelling



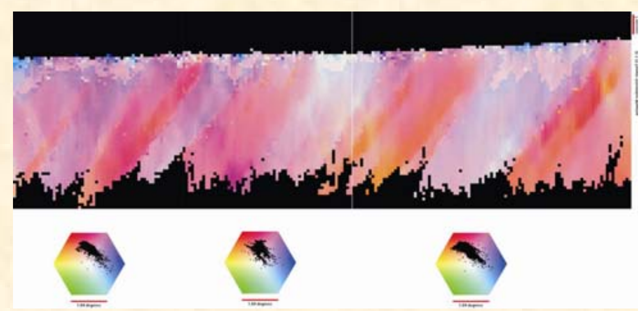
- **Grain growth**
  - **Thin films/ Surfaces**
  - **3D**
  - **Anomalous**
  - **CSL**



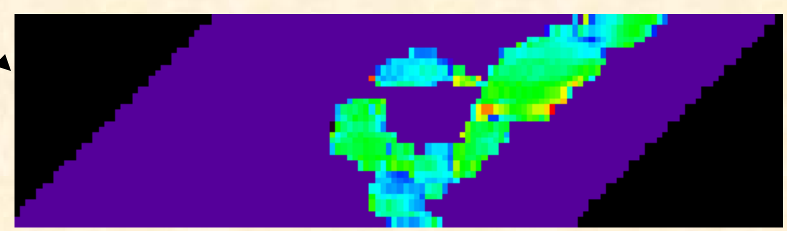
Lattice



- **Deformation/strain**
  - **Single crystal**
  - **Polycrystal**
  - **Near surfaces**



- **Cracks**
  - **Mesoscale field**

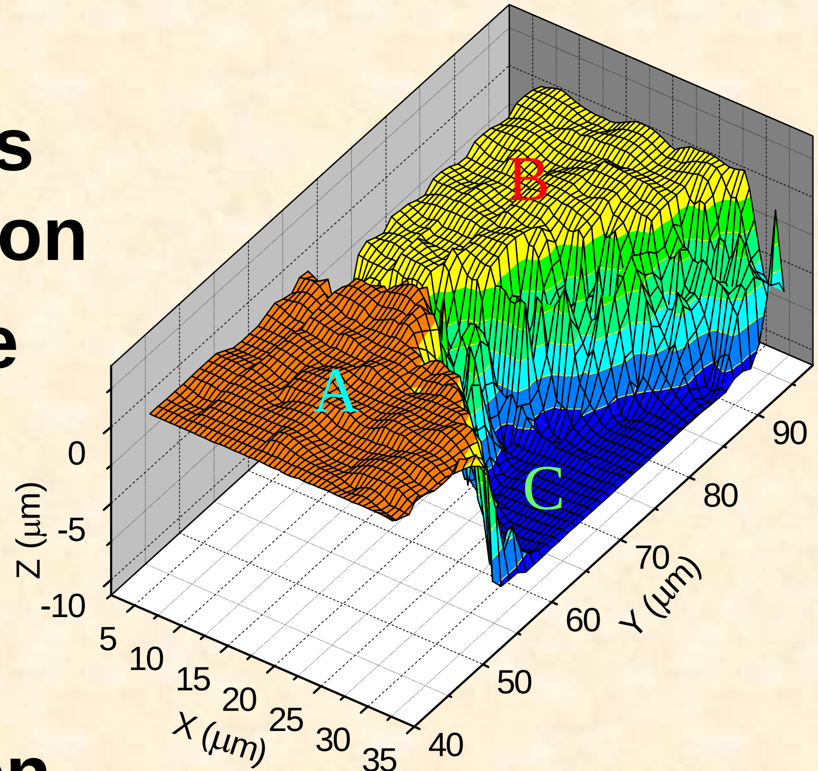


Faster 3D imaging & better spatial resolution will revolutionize science

- **Volume measurements with essential resolution -plasticity length scale**

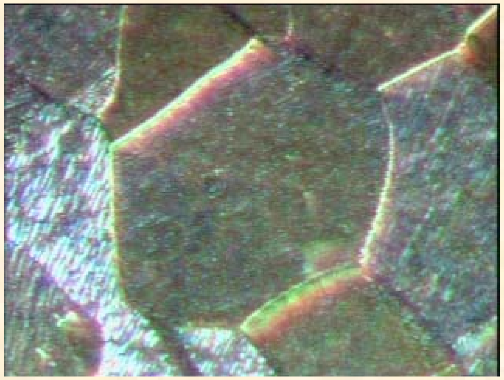
- **In-situ dynamics**

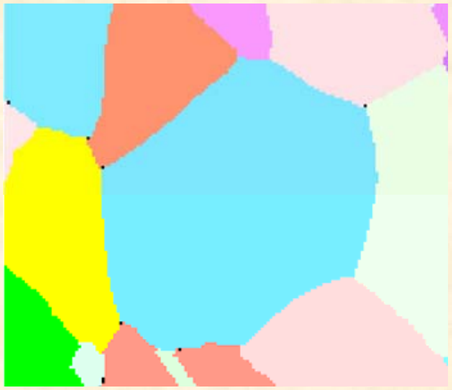
- **User driven exploration**



Higher brilliance *and* Rapid readout needed for 3D imaging

- **ALS/APS developing 100 hz 16 bit CCD**
- **~1000x faster than current CCD**
- **80x 80 x 80=5.1x10^5 =10^4 sec=2.7 hrs**
- **ERL has potential for μs measurements! 1000 x1000 x1000 in seconds!**

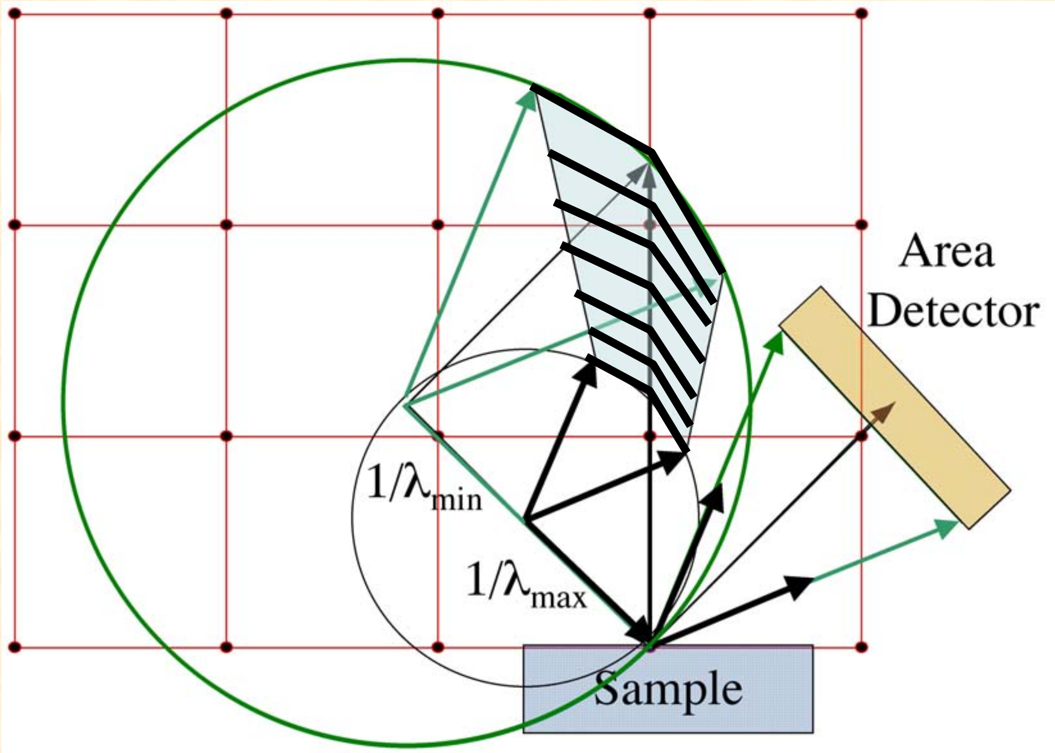




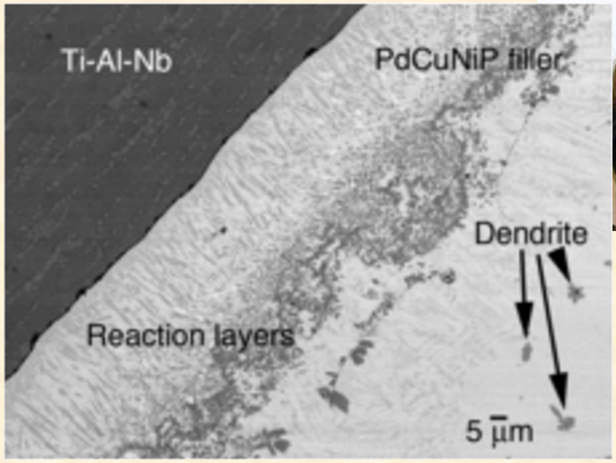
80 x 80 surface image

Energy scan provides single-crystal sensitivity without sample rotations

- **No sample rotations**
  - **Best spatial resolution**
- **Best signal/noise**
  - **Diffuse x-ray scattering**
  - **Grain boundaries**
- **Combinatorial**
  - **Defect distributions**

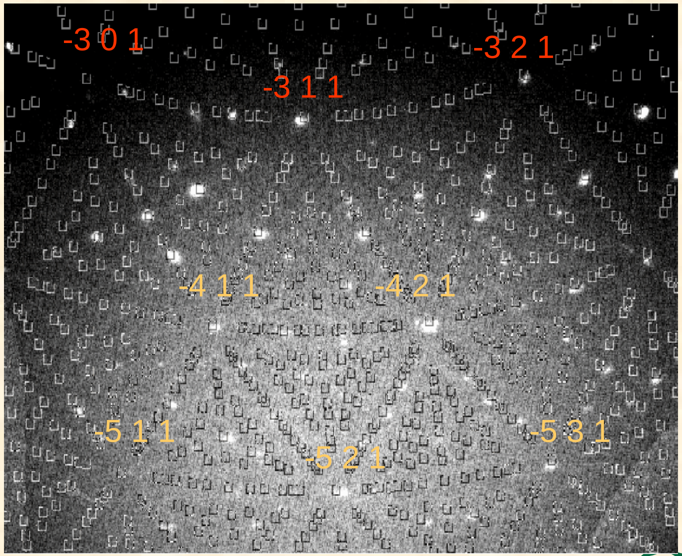


Unknown phase identification



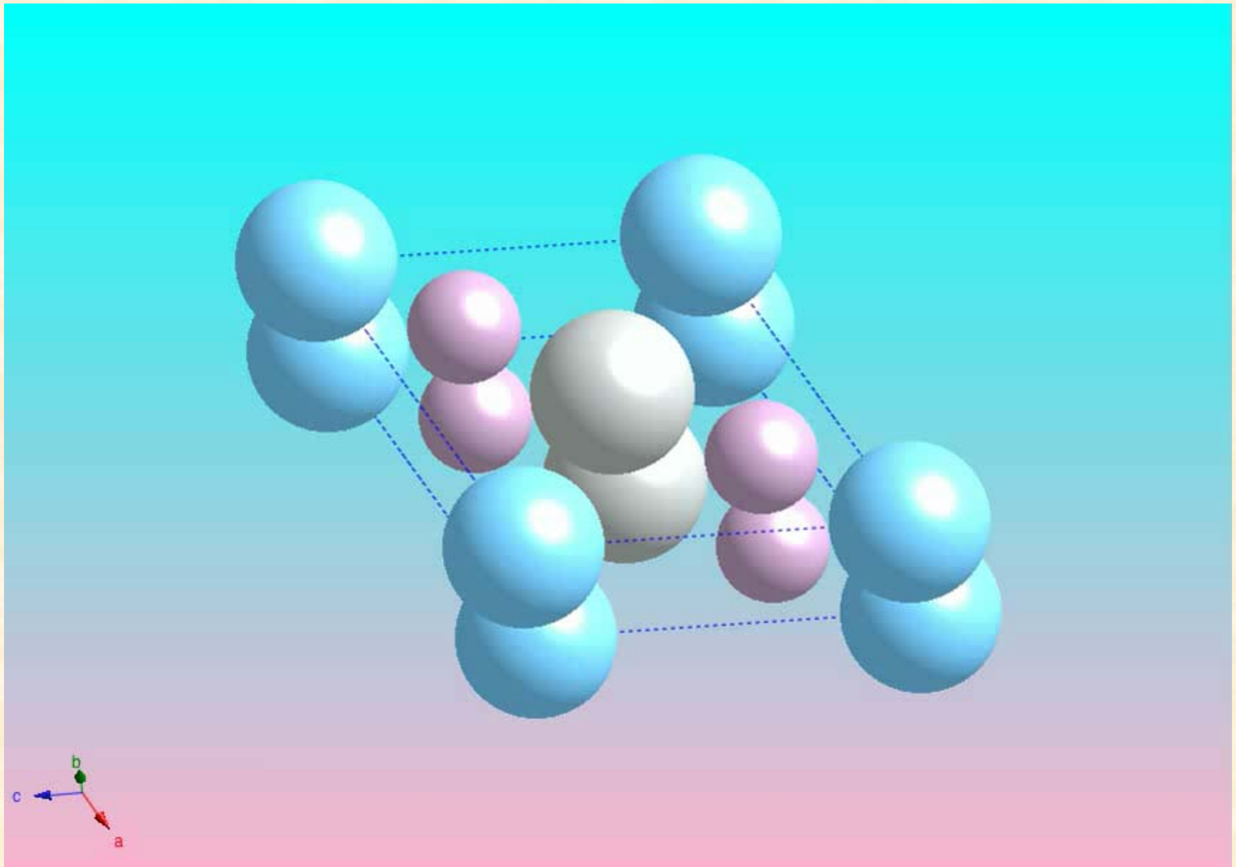
- **Generalization of orientation software can identify phases**
- **Energy scans provide integrated reflectivities.**
- **Identified two minor crystal phases tetragonal/hexagonal**

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



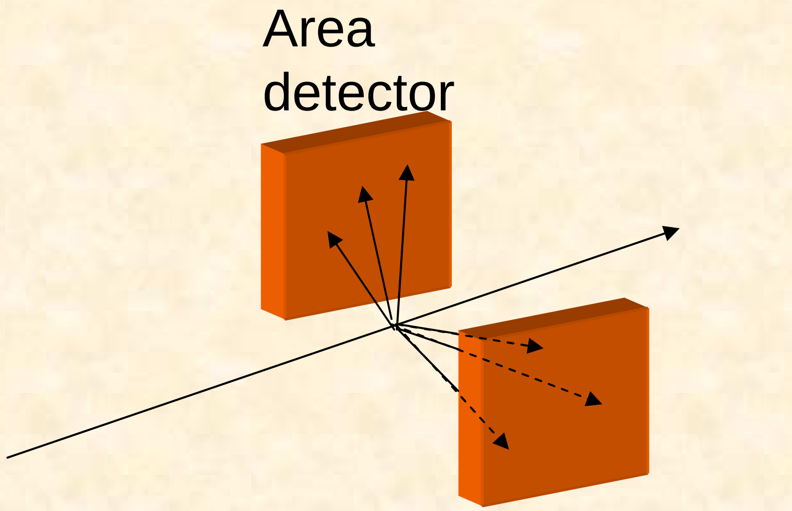
Cannot be found by powder

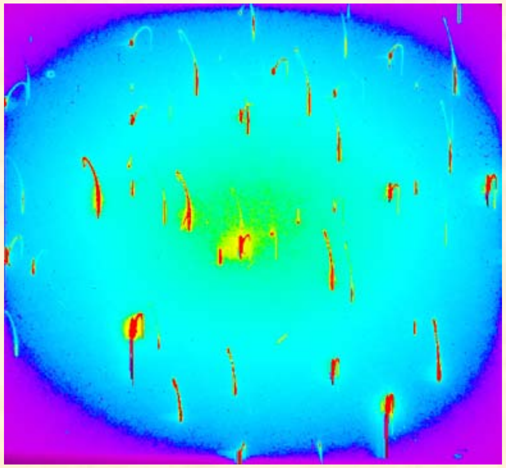
Structure refinement underway



Similar to High pressure advantages of polychromatic methods

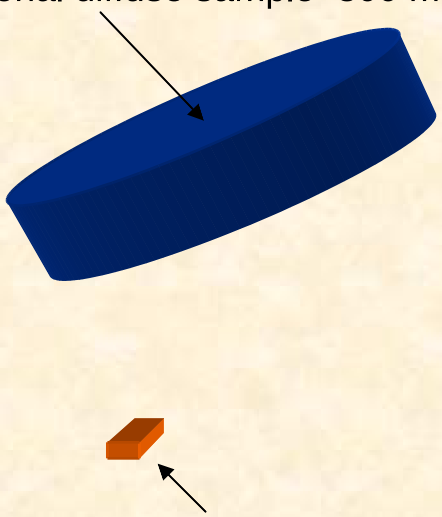
- **Small sample volumes**
- **Difficult to rotate sample chamber**
- **Pressure gradients/polycrystals complicate interpretation**



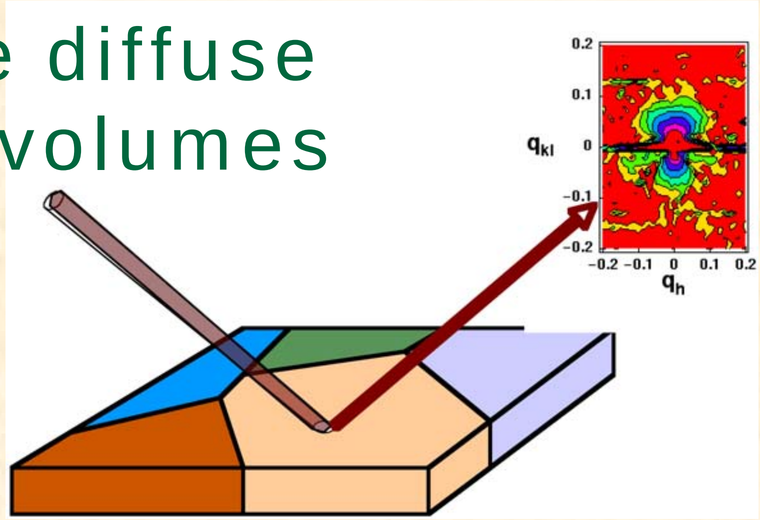


Small irradiated volumes simplify handling/preparation

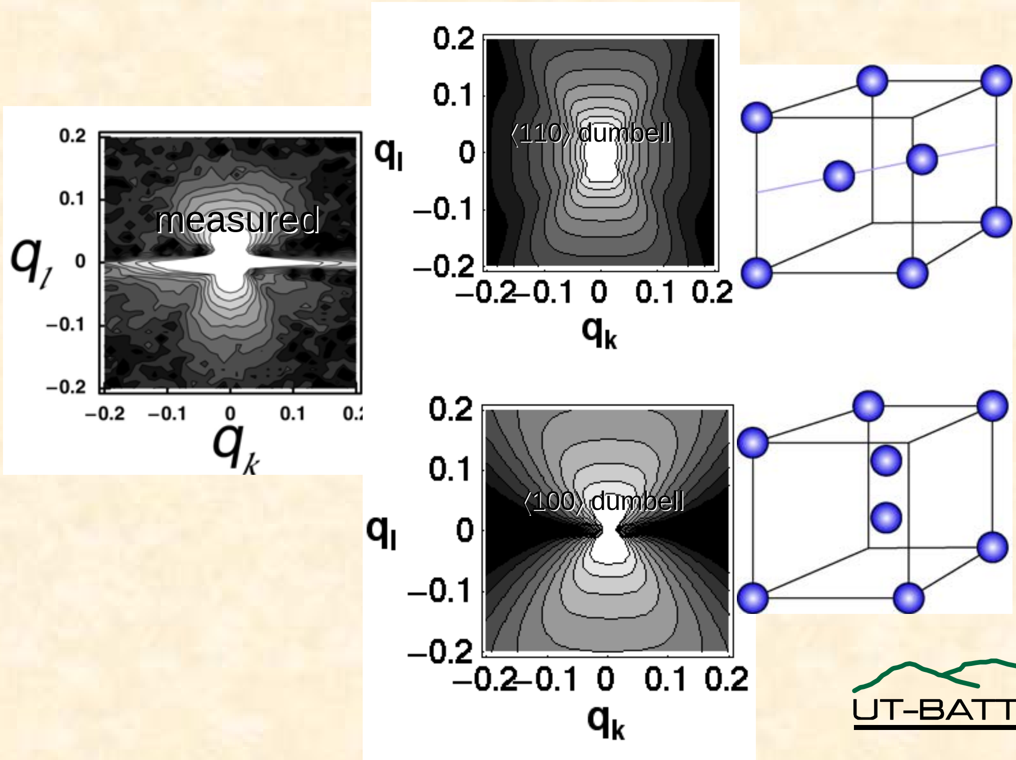
- **Activity ~volume (10^{-5})**
- **Much less waste (10^{-7})**
- **Polycrystalline samples easier obtain- *closer to real materials***



Energy scans measure diffuse scattering from small volumes

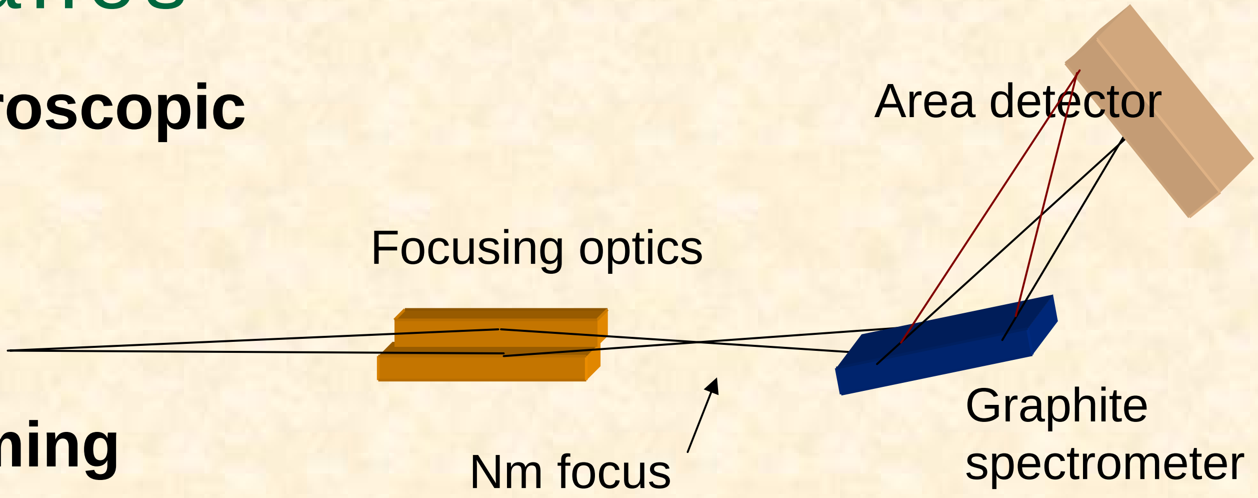


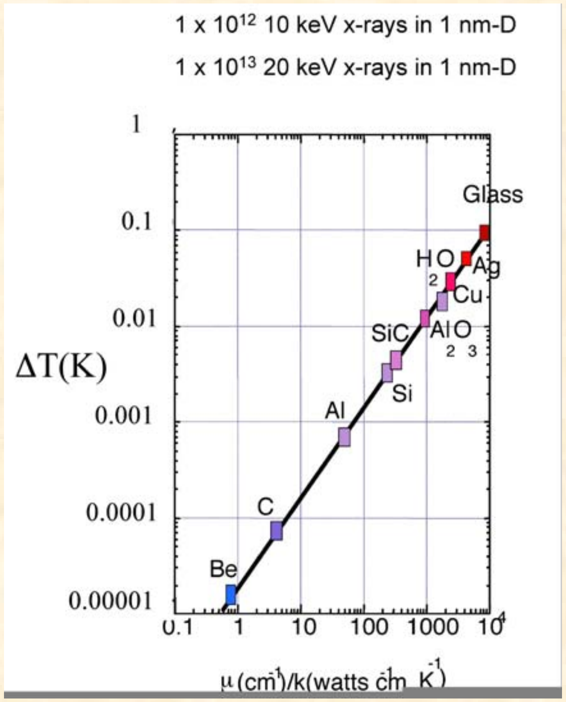
- **Small volumes simplify sample prep**
- **Combinatorial**
- **Relate defects to mesoscale features**



Femptoxanes

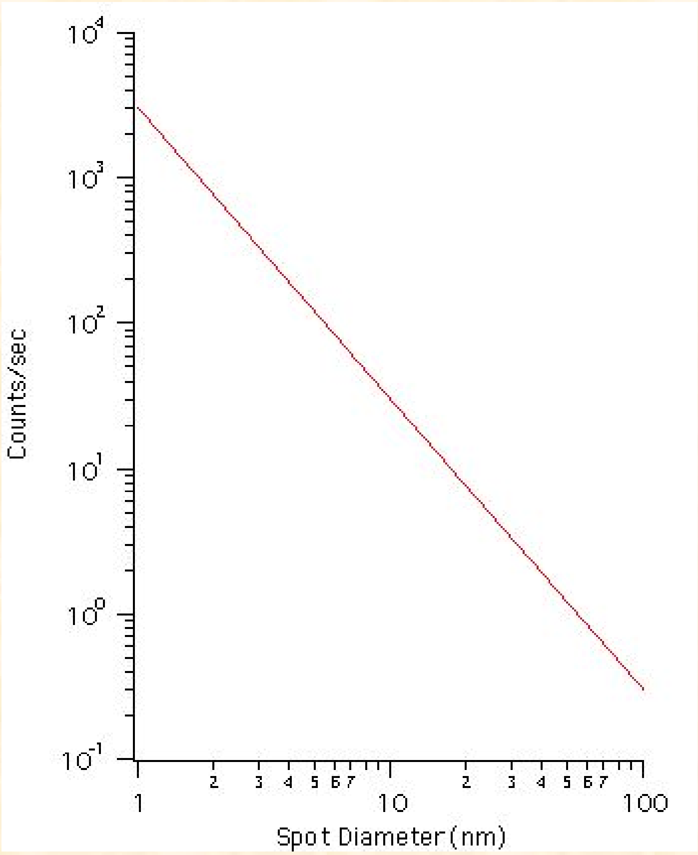
- **Ultrafast spectroscopic mapping**
  - **Real areas!**
- **Single pulse timing**
  - **Dynamics of photo-induced reactions**

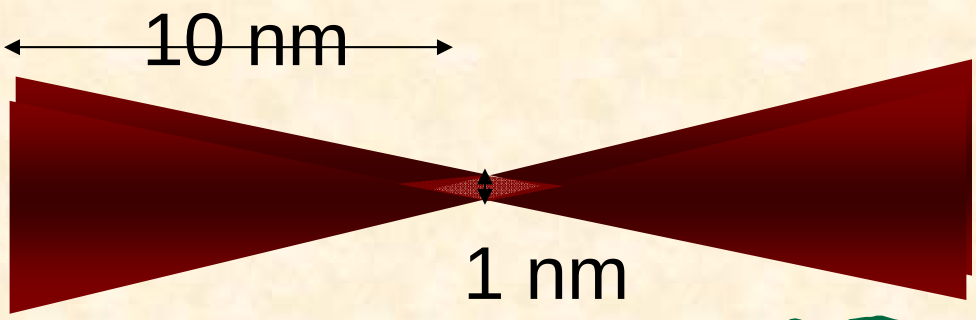




Single atom spectroscopy

- **Signal sufficient to detect single atom**
- **Background must be controlled with crystal optics**
- **Position localized in 3D**



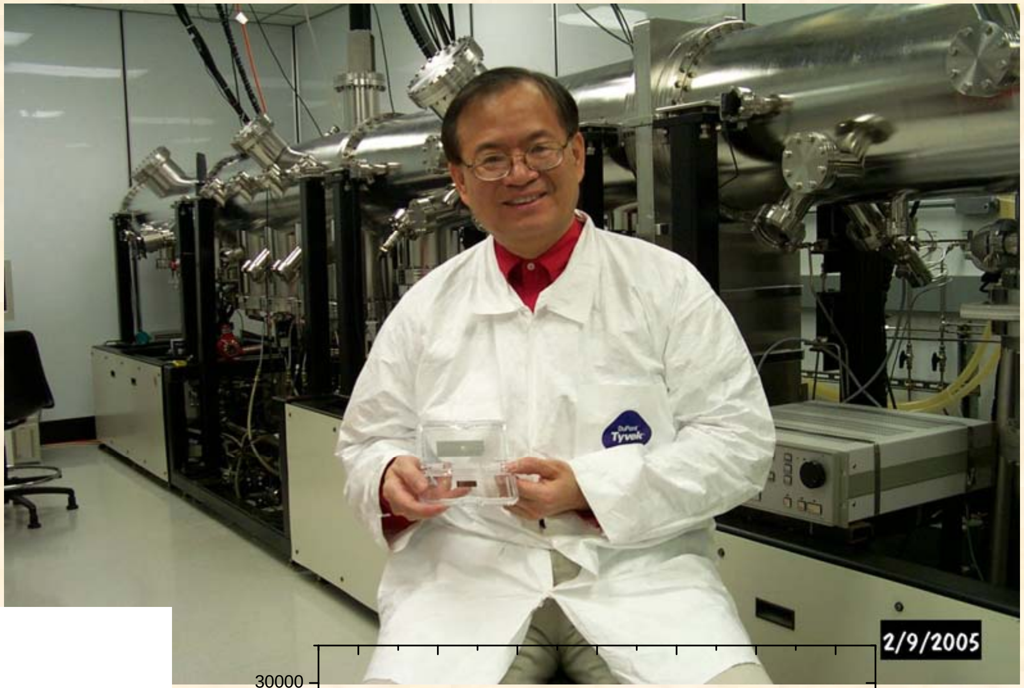


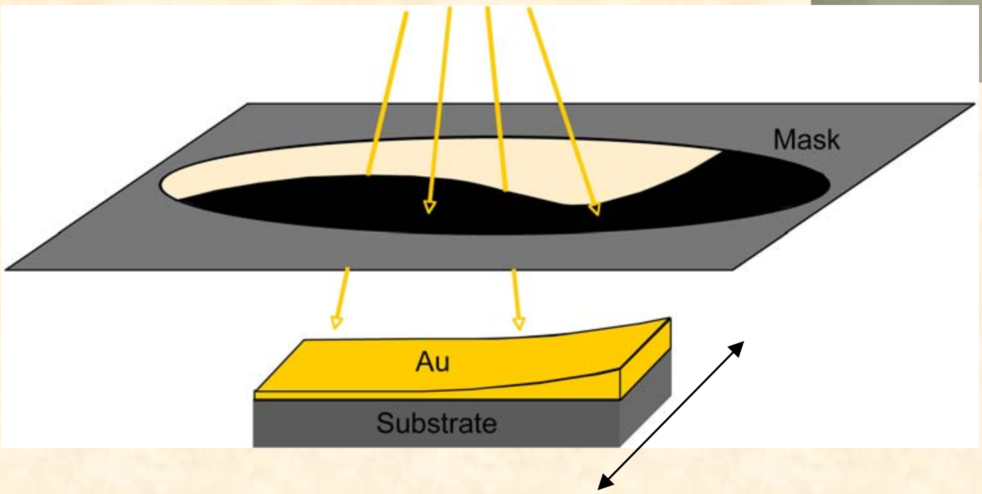
Better optics essential to exploit ERL properties

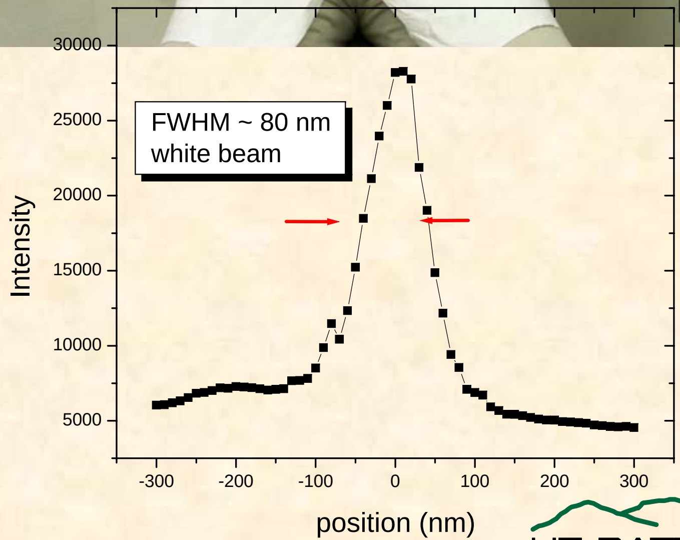
- **More perfect mirrors**
- ***Much* faster area detectors**
  - **Phosphors/ parallel readout/pipelined analysis**
- **New technical approaches/software**
  - **Spatial deconvolution**

APS (C. Liu) pioneering precision profile coating

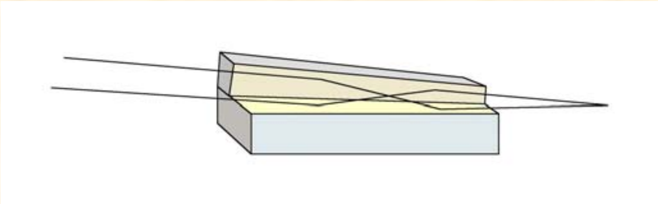
- **Flexible path to x-ray quality aspherical optics**
- **Corrects fabrication imperfections**
- **Physically/thermally stable**



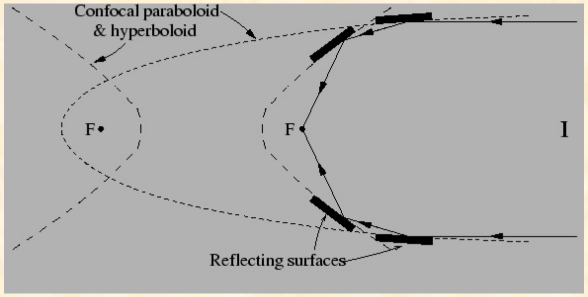




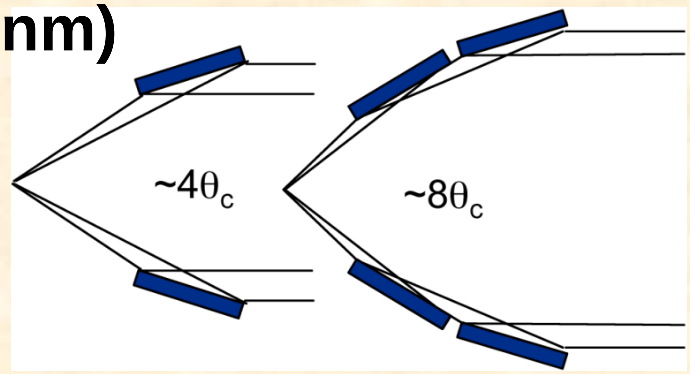
New approaches needed for nondispersive <25 nm beams



- **Nested (20 nm)**
- **Multilayers (6-8 nm)**
- **~~Wolter (6-8 nm)~~**



- **Deflected beam (4-8 nm)**



Summary: ERL + advanced KB optics offers new science opportunities

- **Single atom spectroscopy**
- **Single pulse fs XANES**
- **Unknown phase identification from nanomaterials**
- **Grain boundary structure**
- **3D mapping**
- **Diffuse scattering from ultra-small volumes**