

Synchrotron-based Coherent X-ray Diffraction for Imaging ZnO Nanomaterials

Ian Robinson

Ross Harder

Steven Leake

Marcus Newton

London Centre for Nanotechnology

Diamond Light Source

CMMP 09

Warwick University

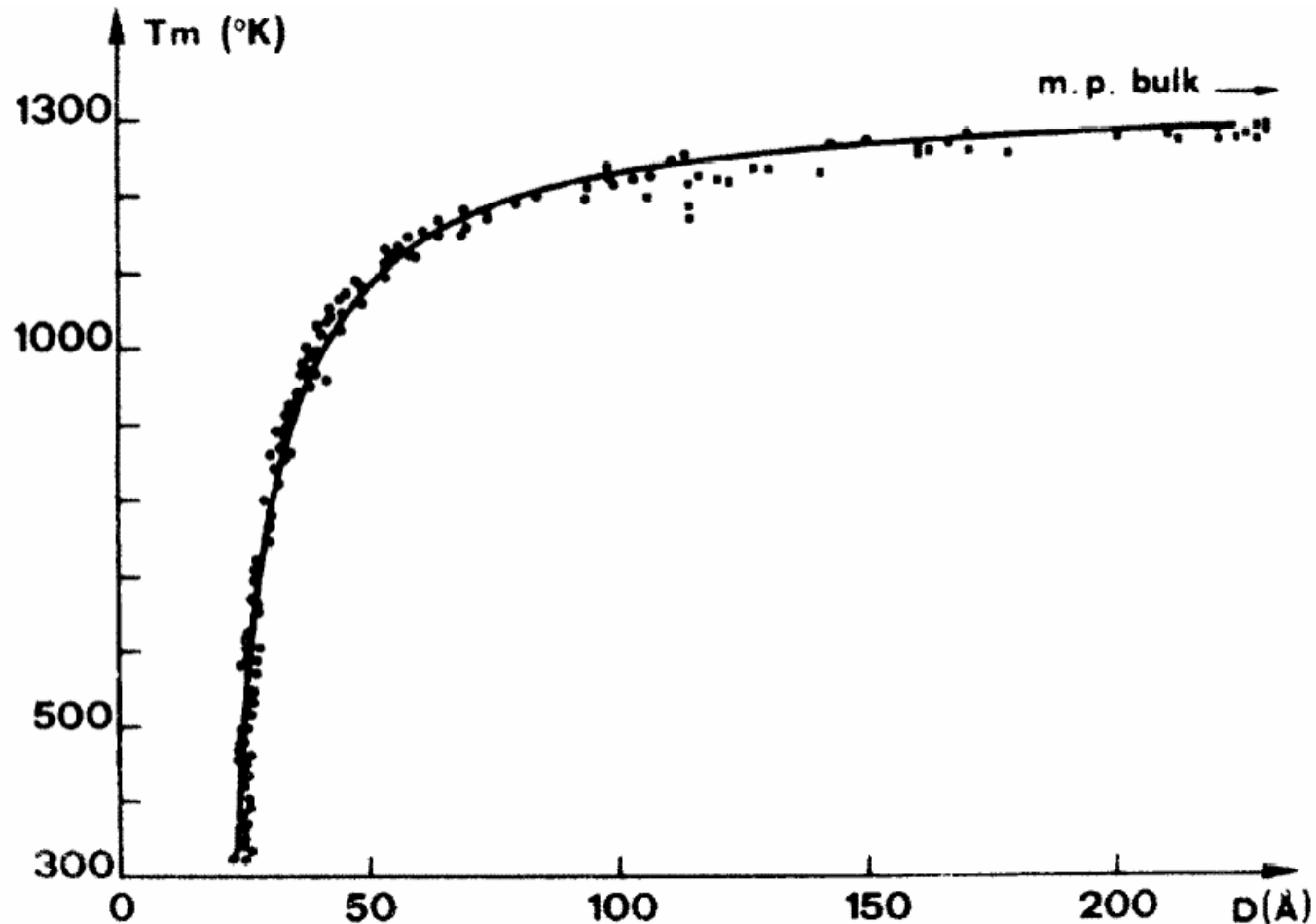
December 2009

Outline

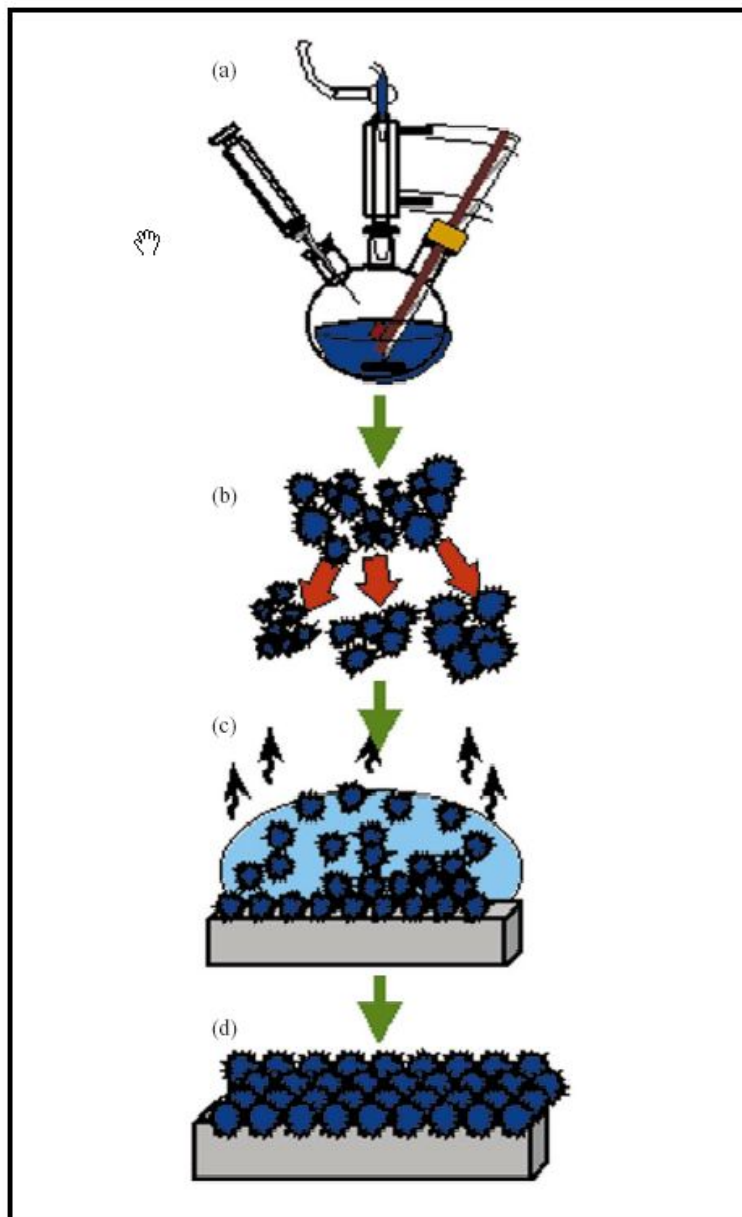
- Coherent x-ray diffraction
- CXD can solve the **phase** problem
- Nanocrystal structures
- Extension to **phase** objects
- Exploration of crystal strain
- Full Strain Tensor is accessible

Size-dependent Melting of Au Particles

P. Buffat and J-P. Borel, Phys. Rev. A 2287-97 (1975)

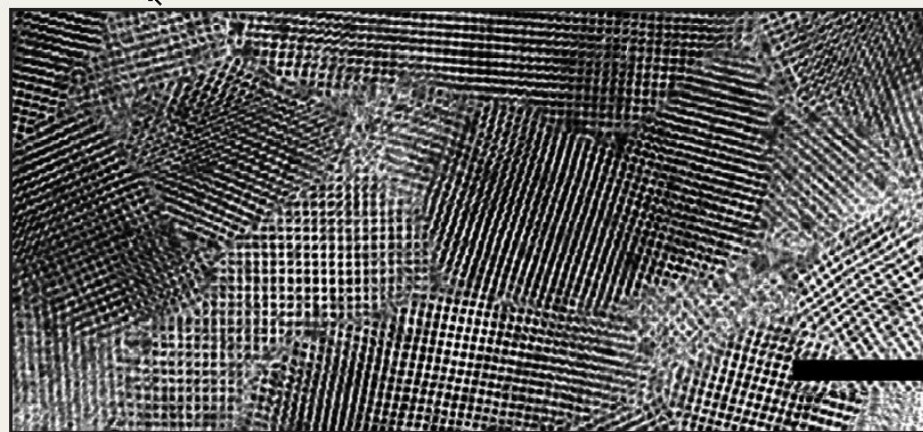


Chemical Synthesis of Nanocrystals

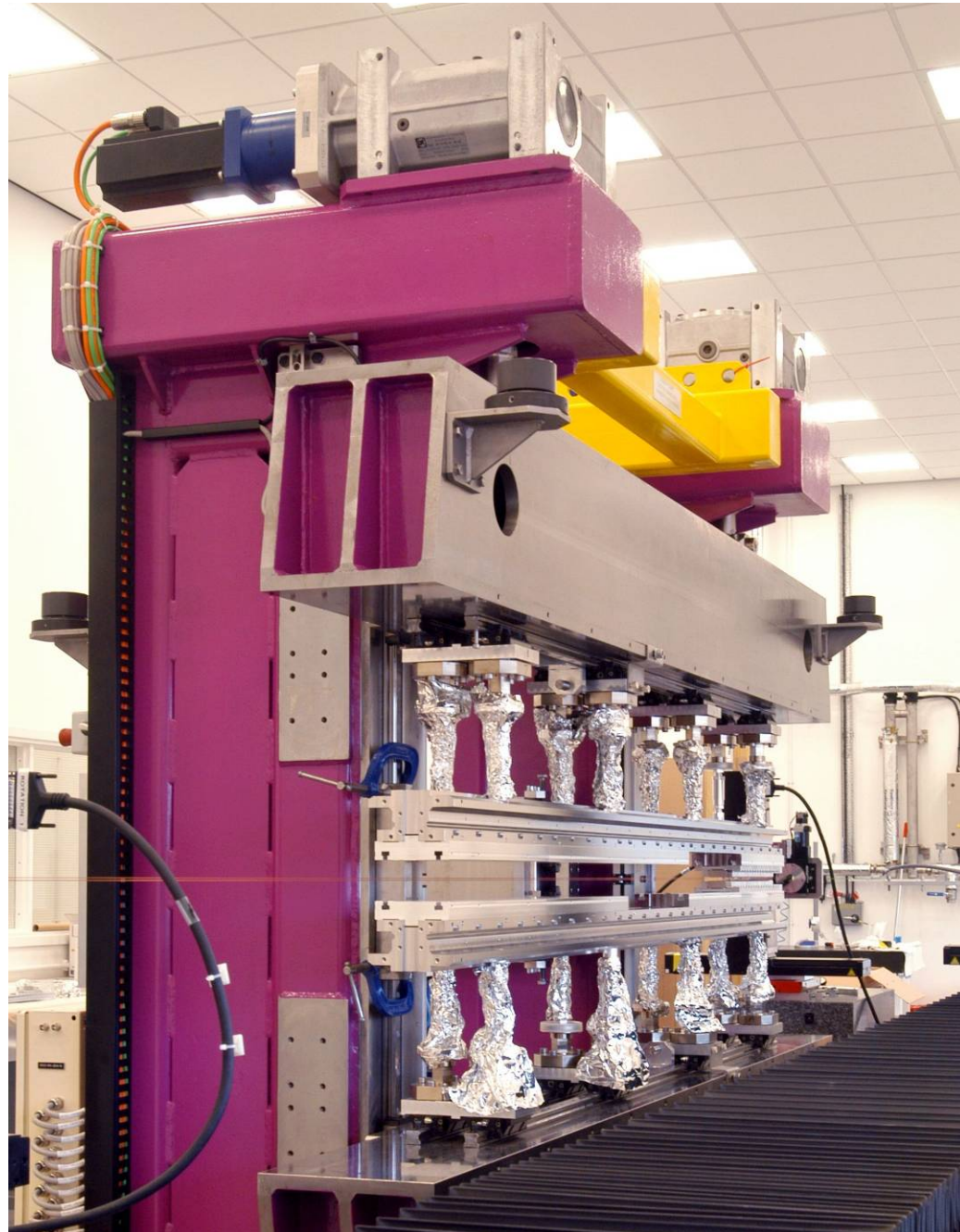


- Reactants introduced rapidly
- High temperature solvent
- Surfactant/organic capping agent
- Square superlattice (200nm scale)

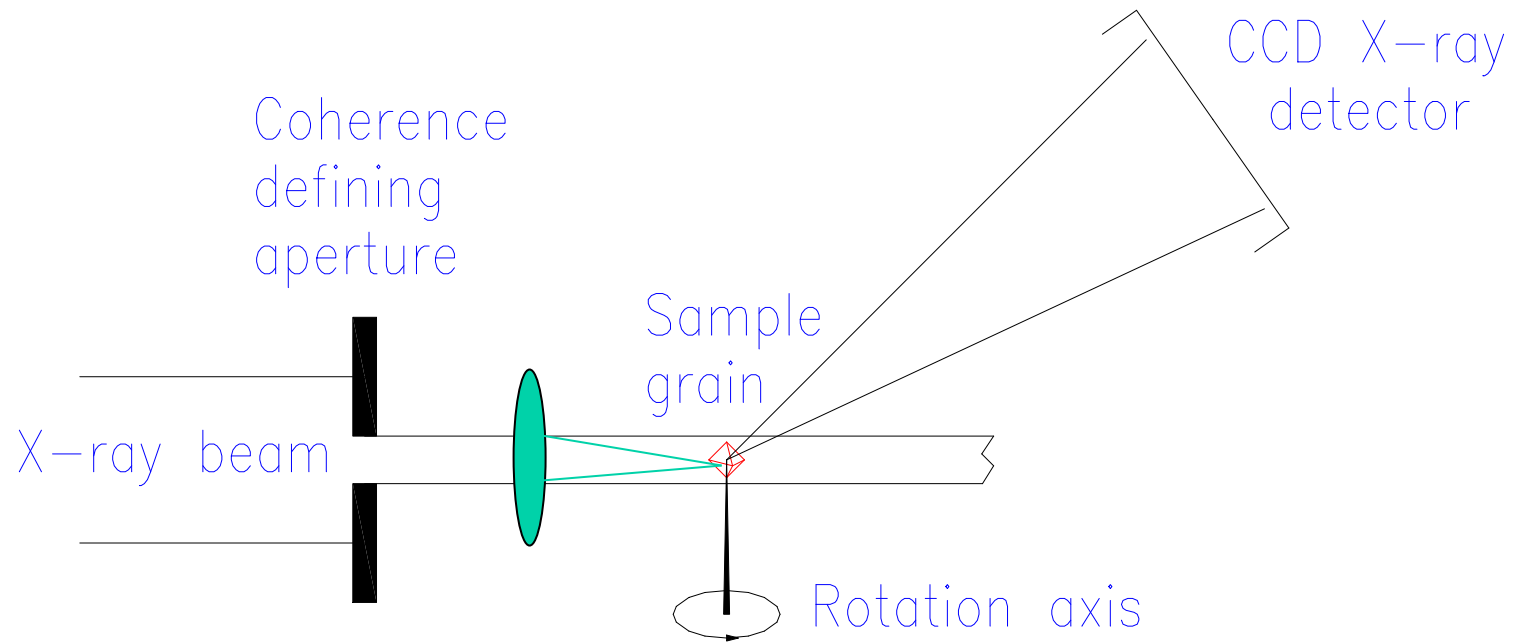
C. B. Murray, *IBM J. Res. & Dev.*
45 47 (2001)



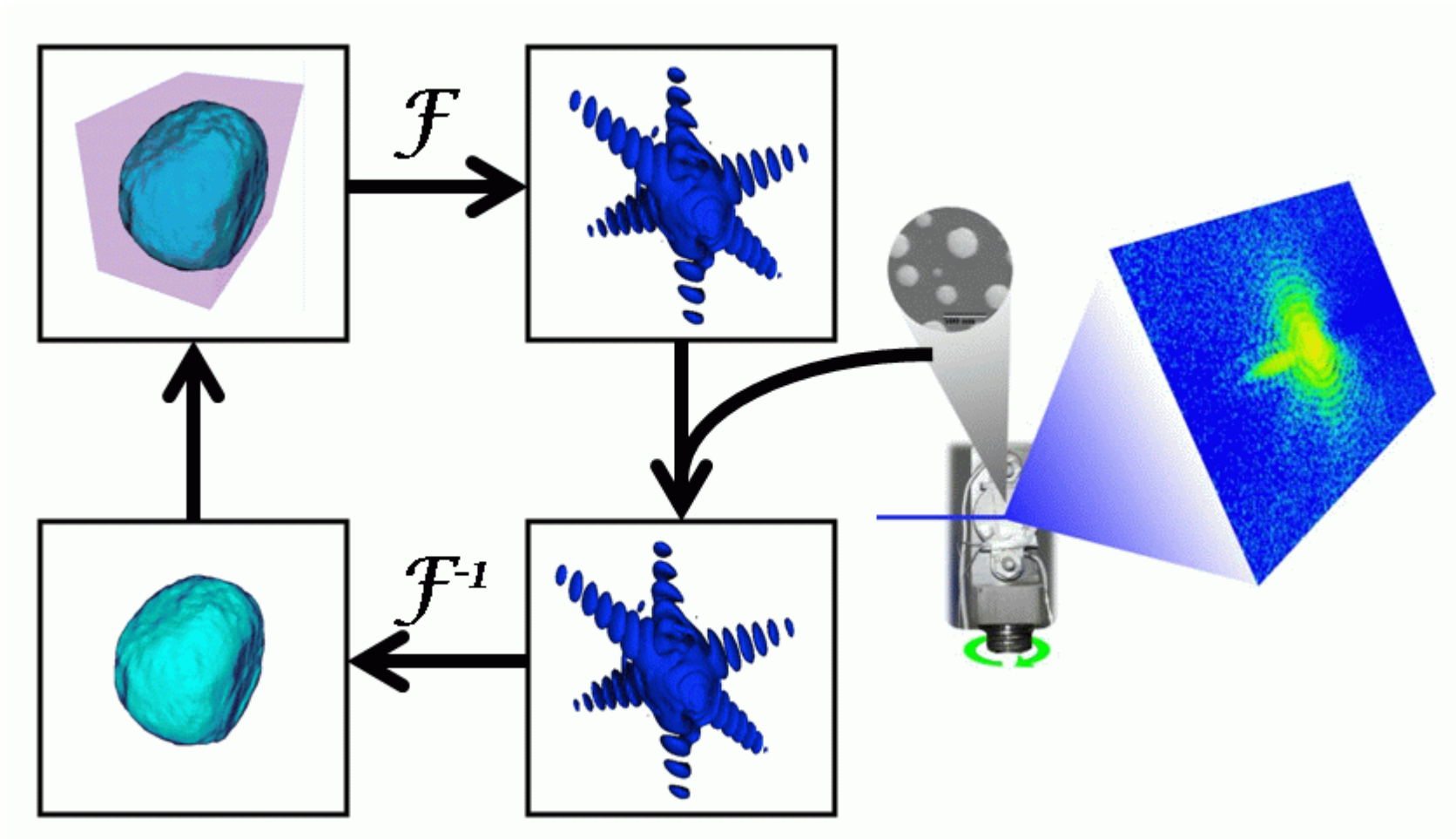
Diamond
in-vacuum
X-ray
Undulator



Lensless X-ray Microscope, 2003



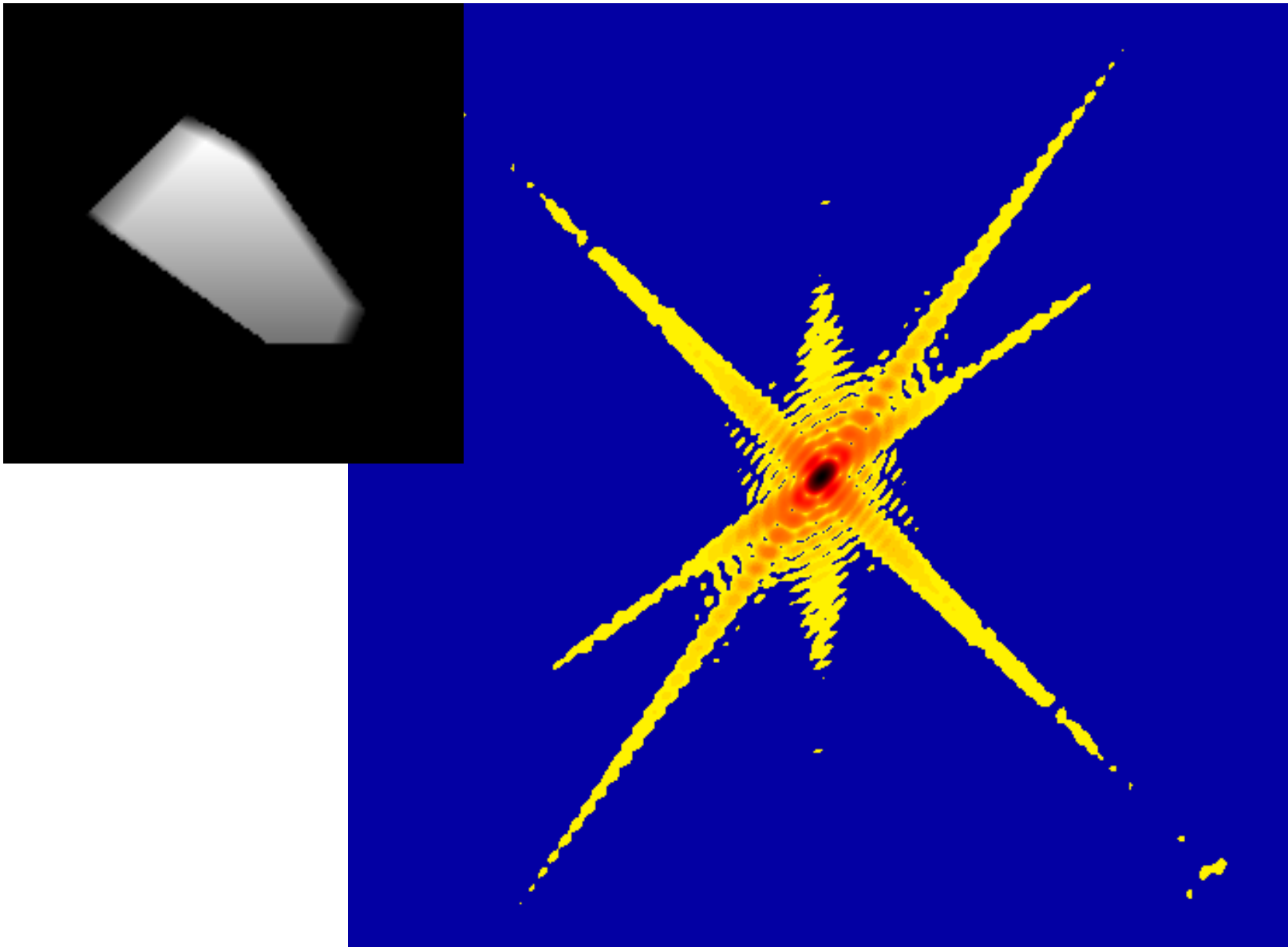
Generic “Error Reduction” method



J. R. Fienup *Appl. Opt.* 21 2758 (1982)

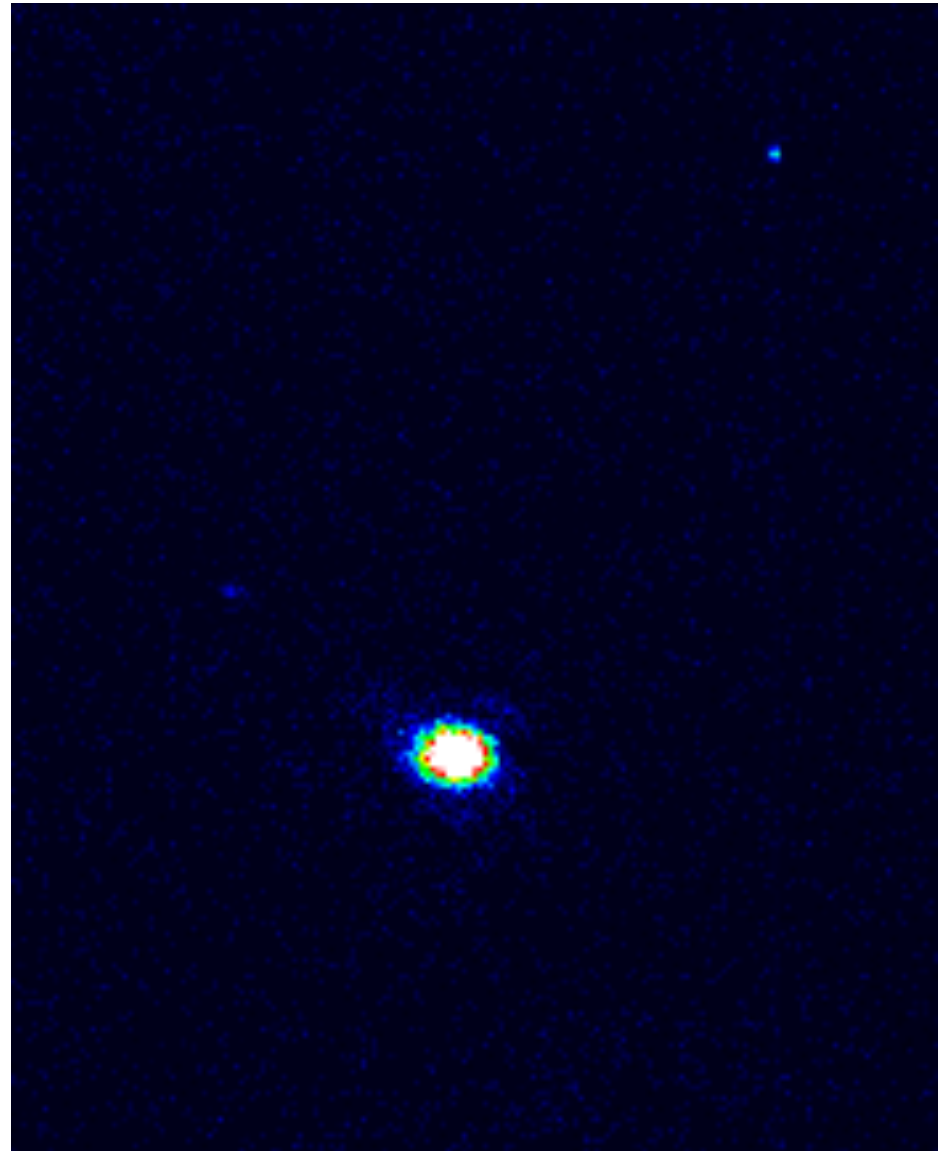
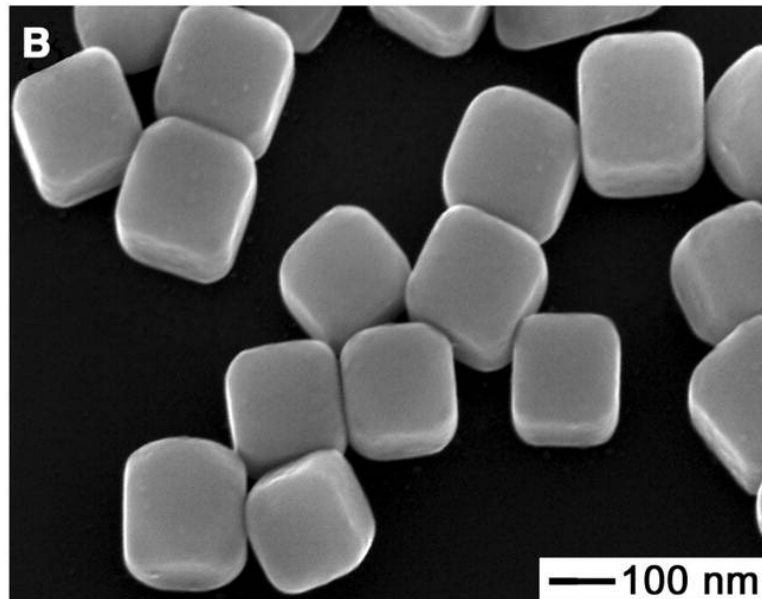
R. W. Gerchberg and W. O. Saxton *Optik* 35 237 (1972)

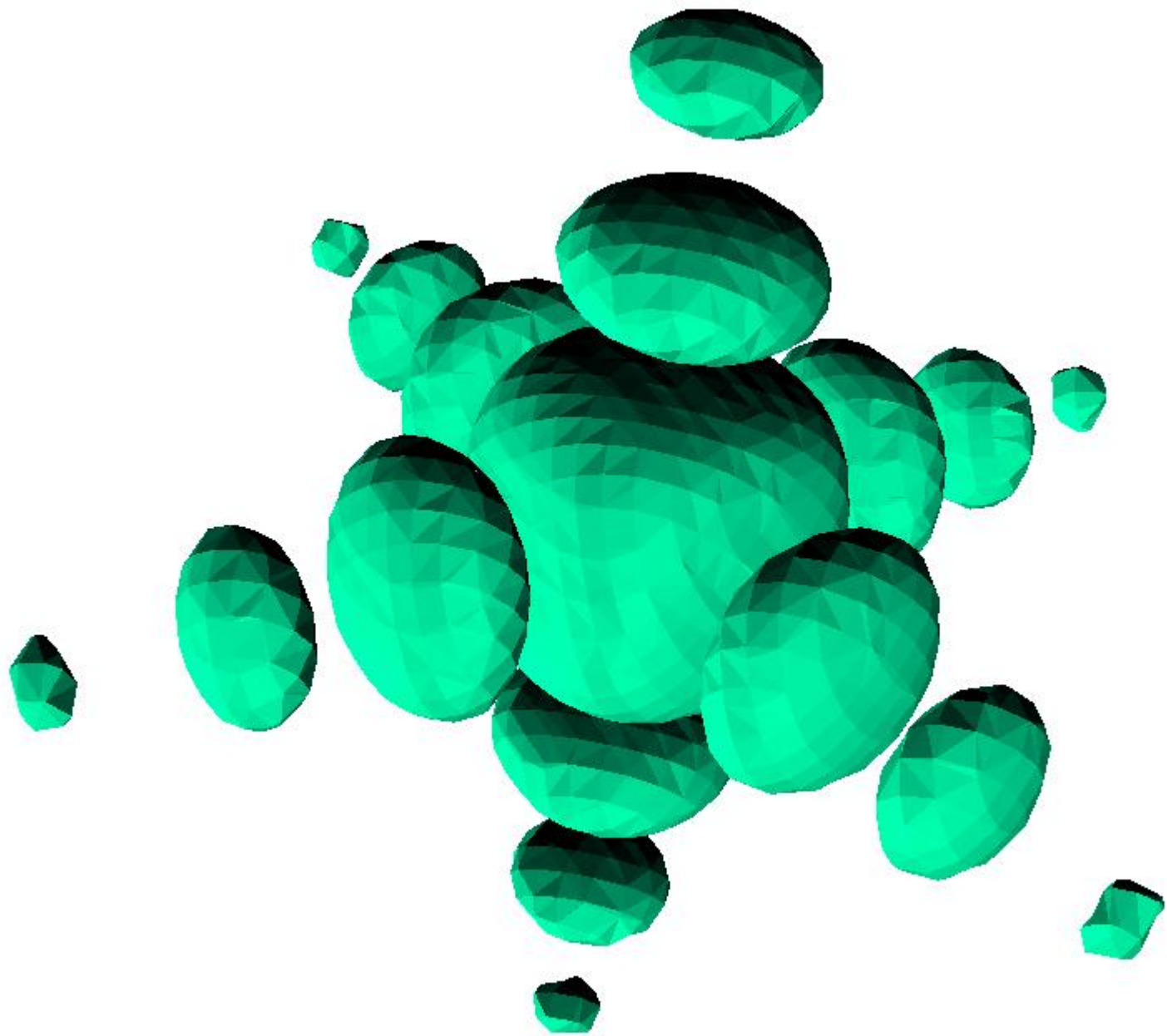
I. K. Robinson, CMMP 2009

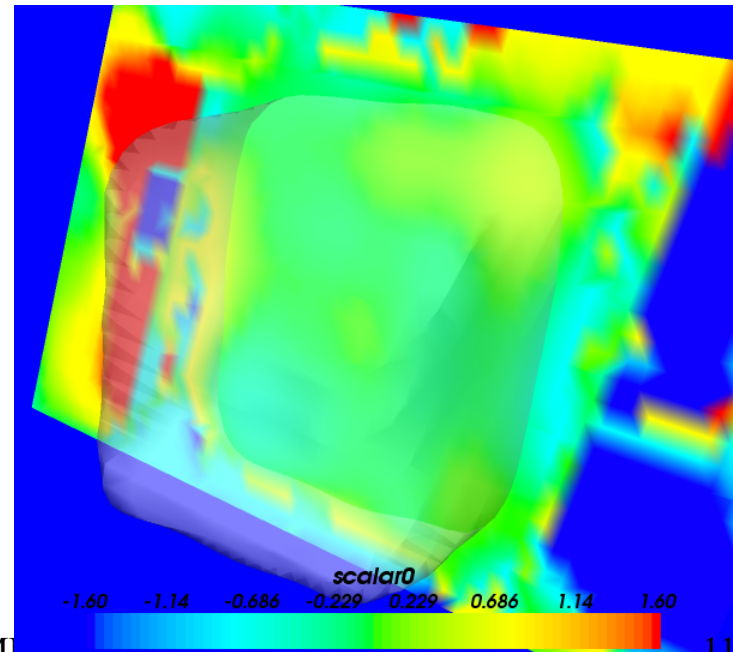
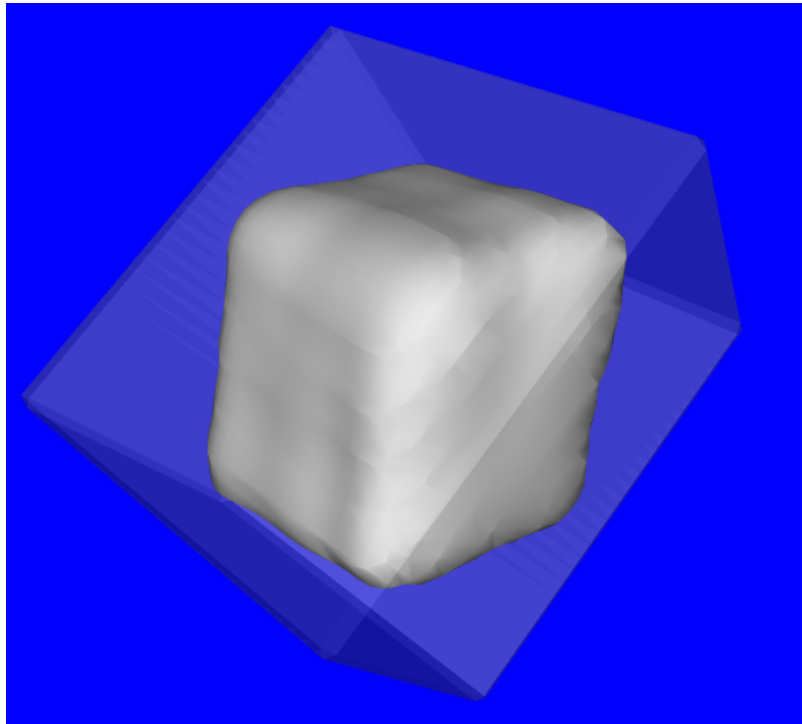
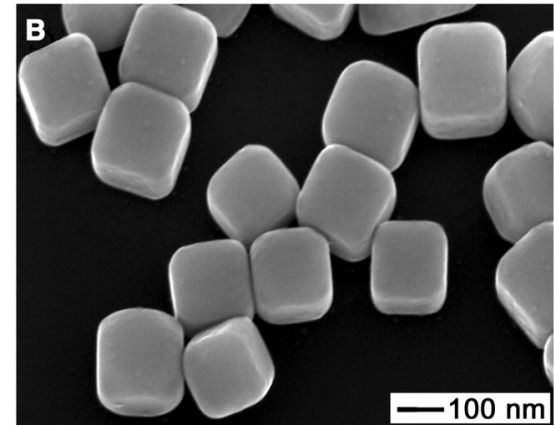
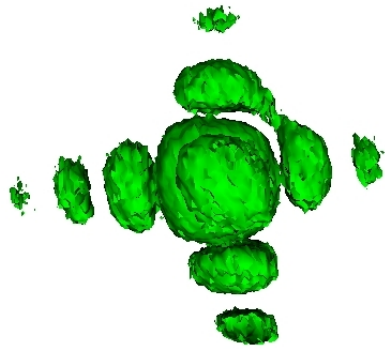
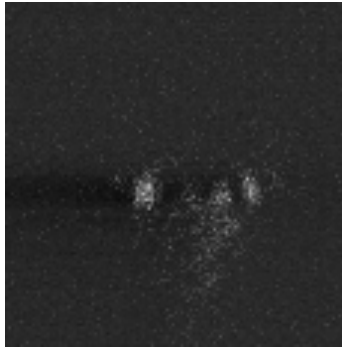


Chemically Synthesized Silver Nanocube Rock with 0.01° steps

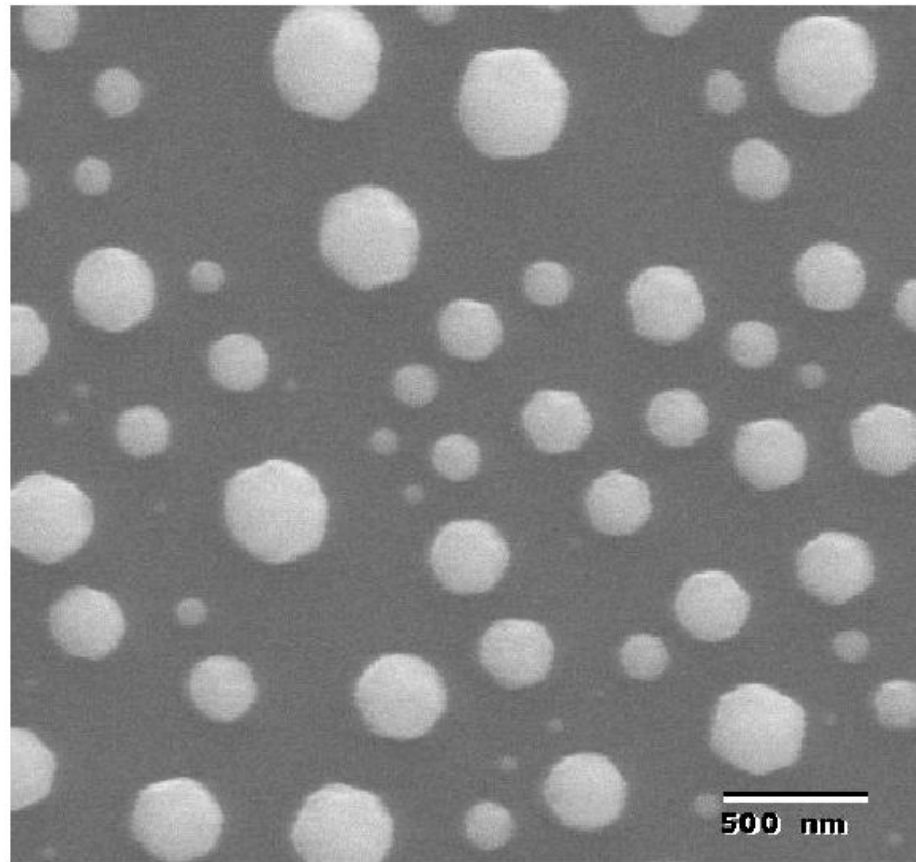
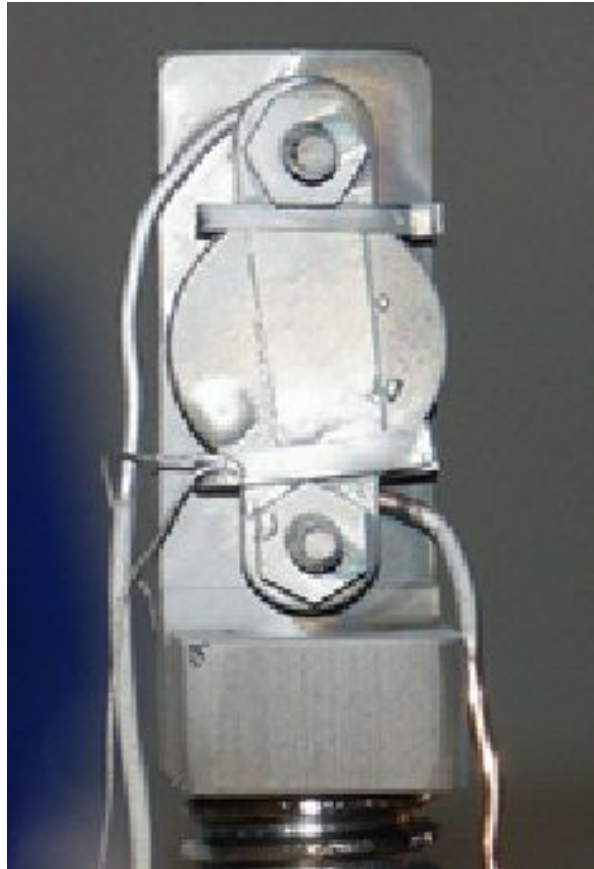
Yugang Sun and Younan Xia,
Science 298 2177 (2003)



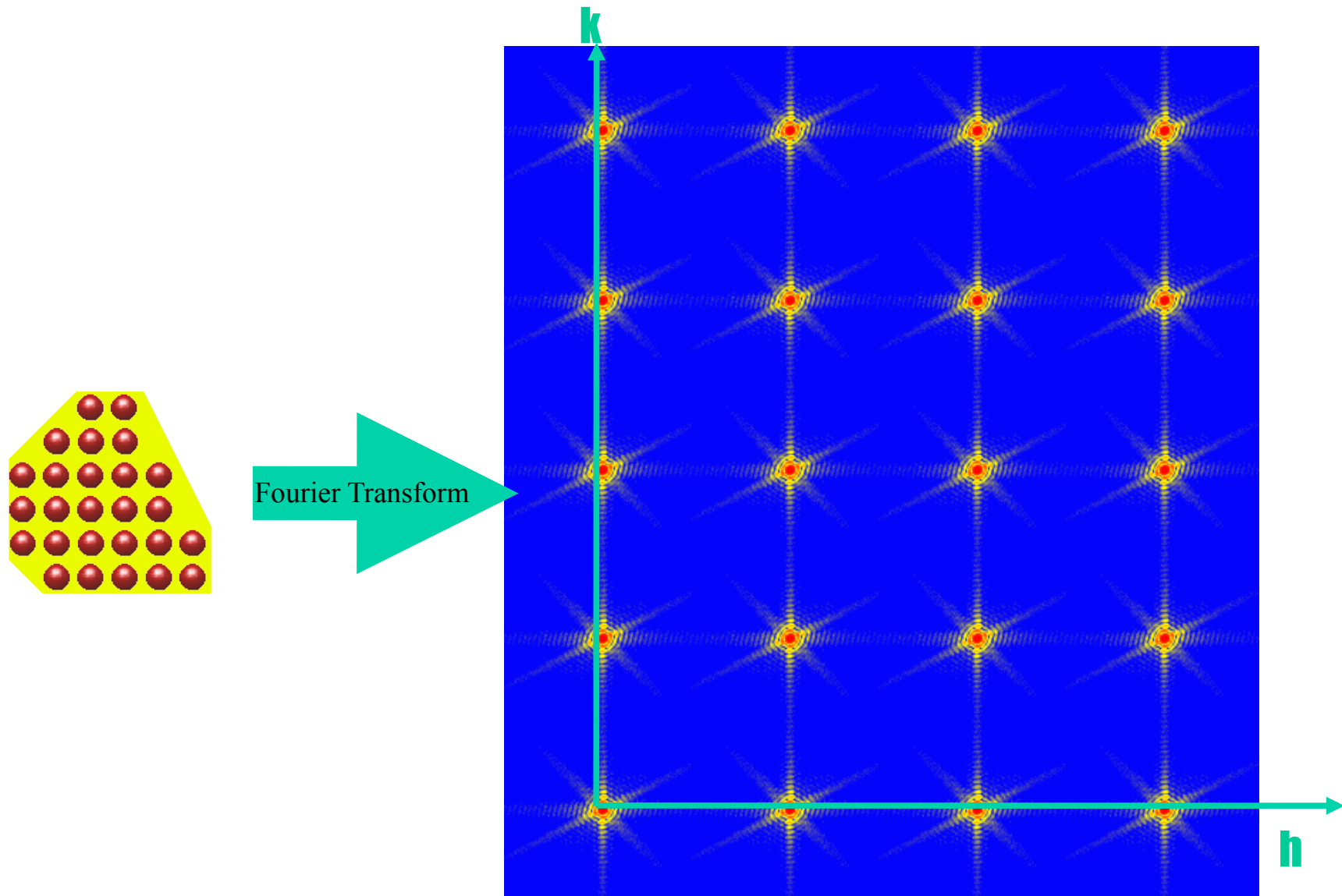




In situ growth of Pb crystals

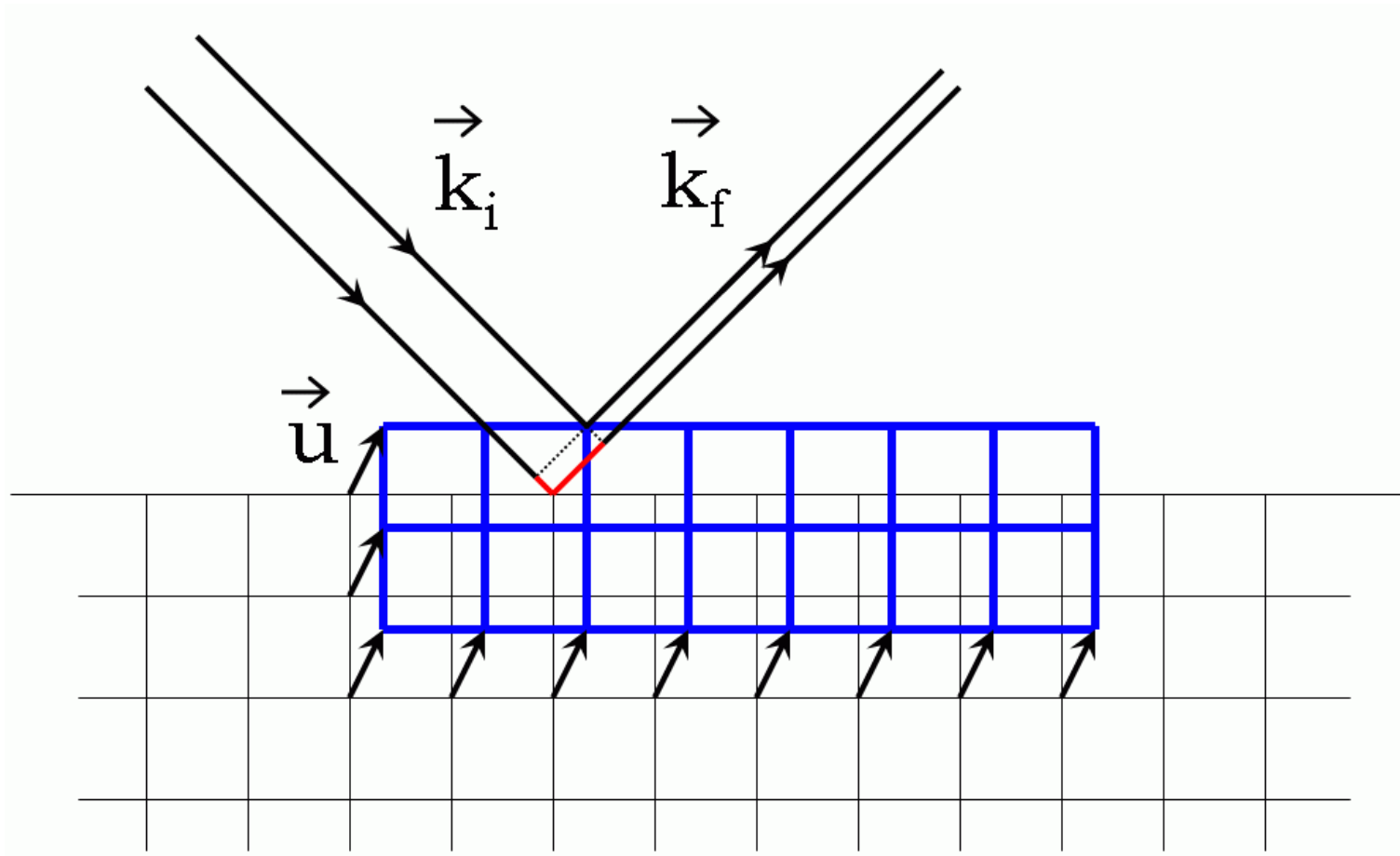


Coherent Diffraction from Crystals



Sensitivity to strain

$$\Delta\varphi = \mathbf{k}_f \cdot \mathbf{u} - \mathbf{k}_i \cdot \mathbf{u} = \mathbf{Q} \cdot \mathbf{u}$$



Good statistics, 3D diffraction data

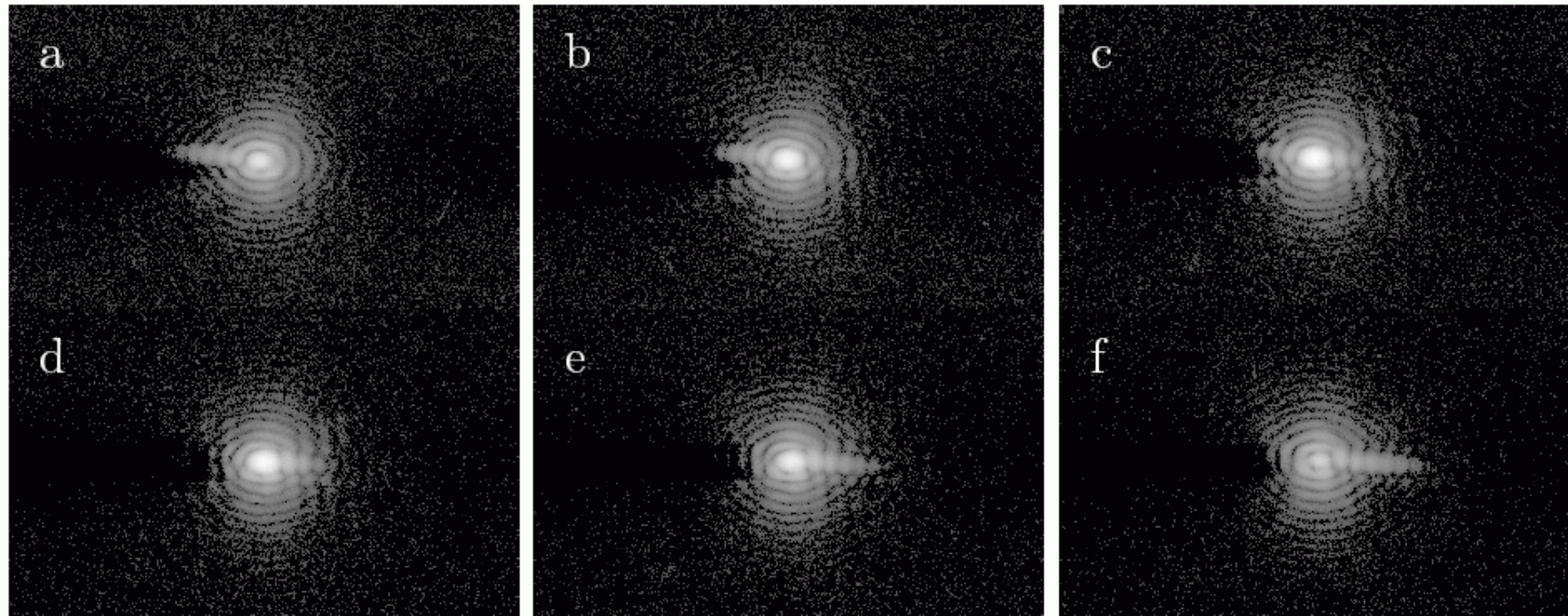
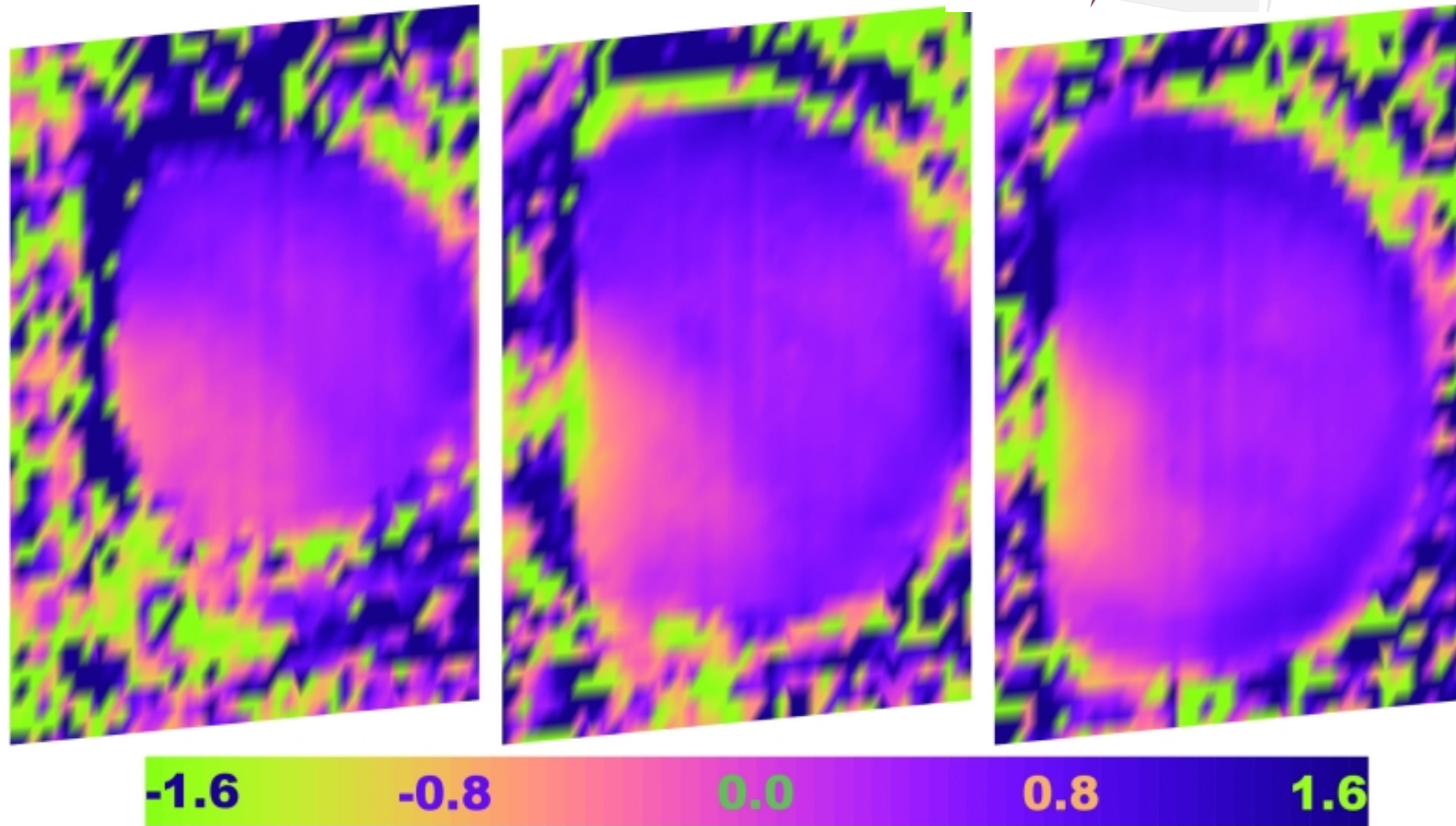
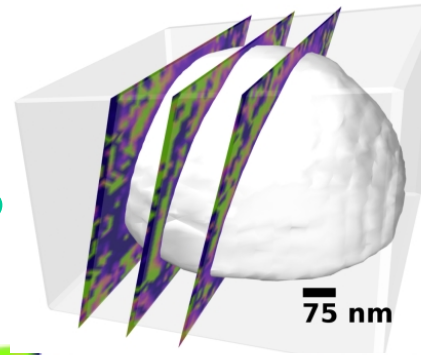
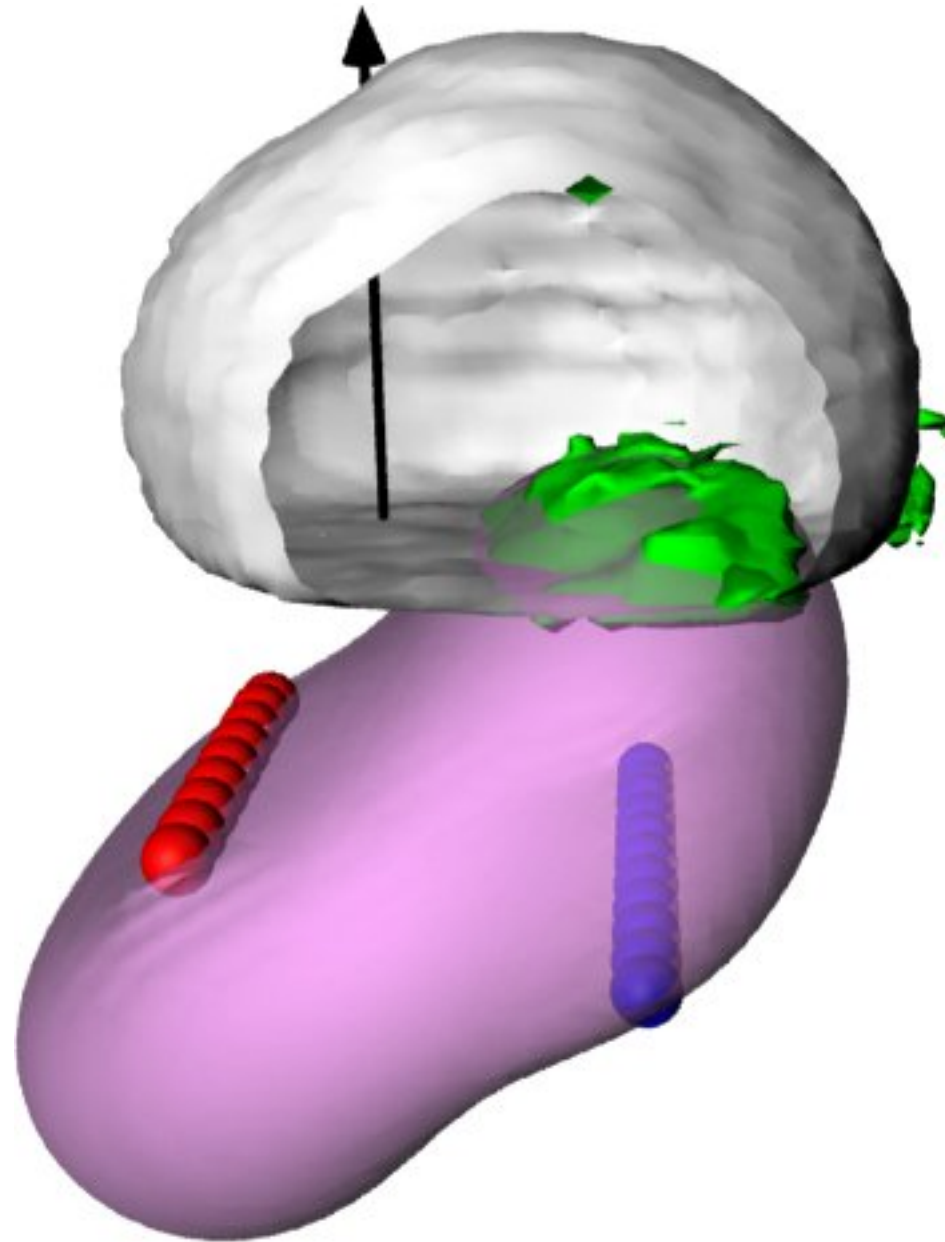


Figure 4.12: Center slices from 3D CXD pattern from Pb sample, on a log scale. Data file 296 from 10/03.

3D phase map sections

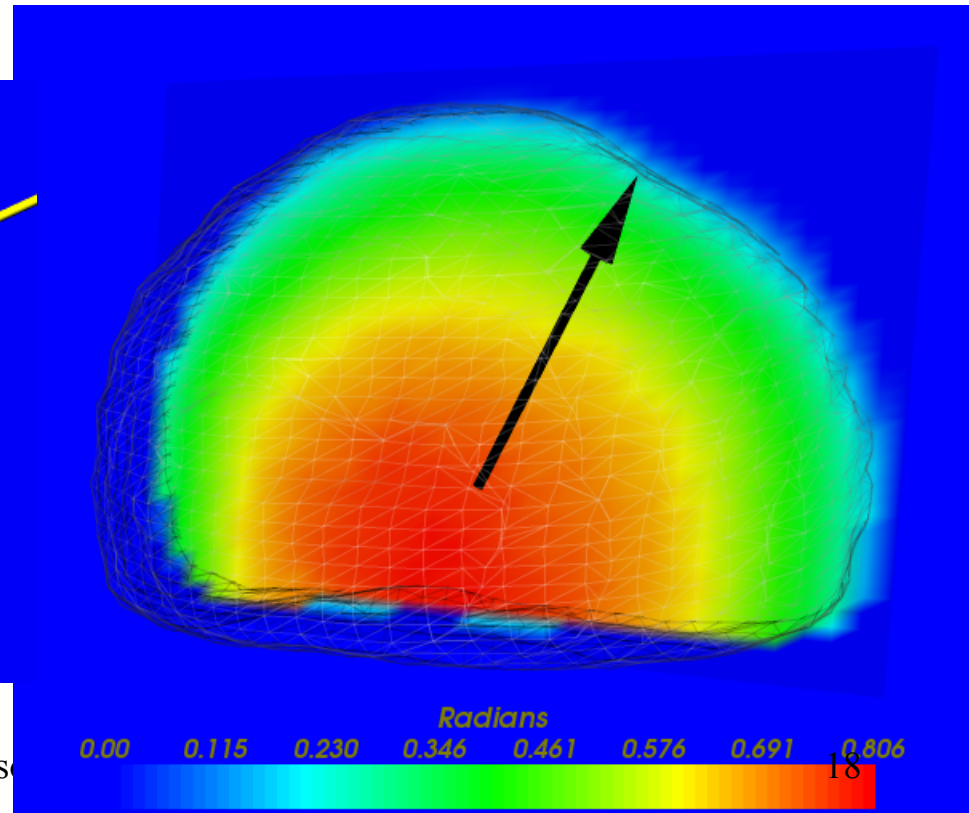
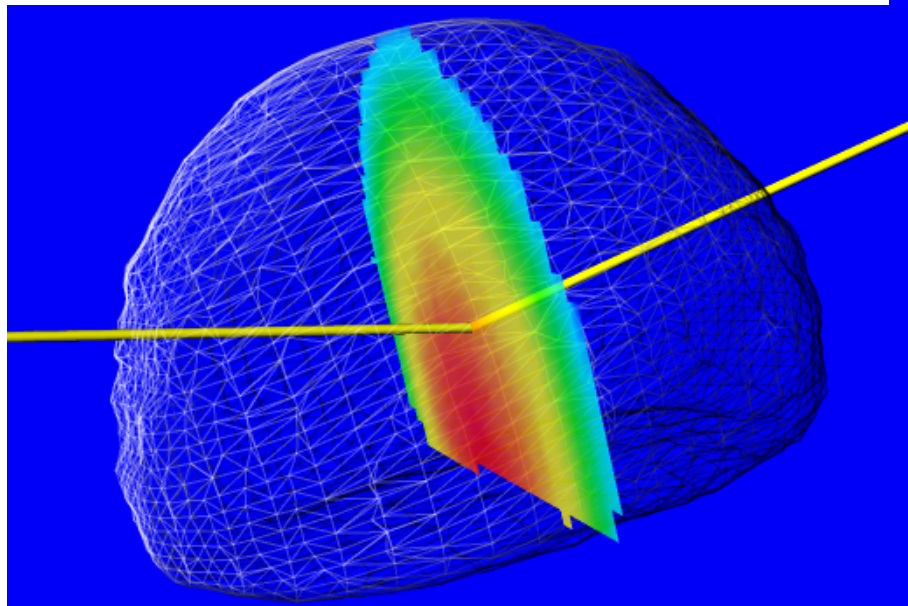




Refraction effects in Lead at 8.9keV

Phase accumulation due to refraction along scattering path
 $n=1-\delta+i\beta$

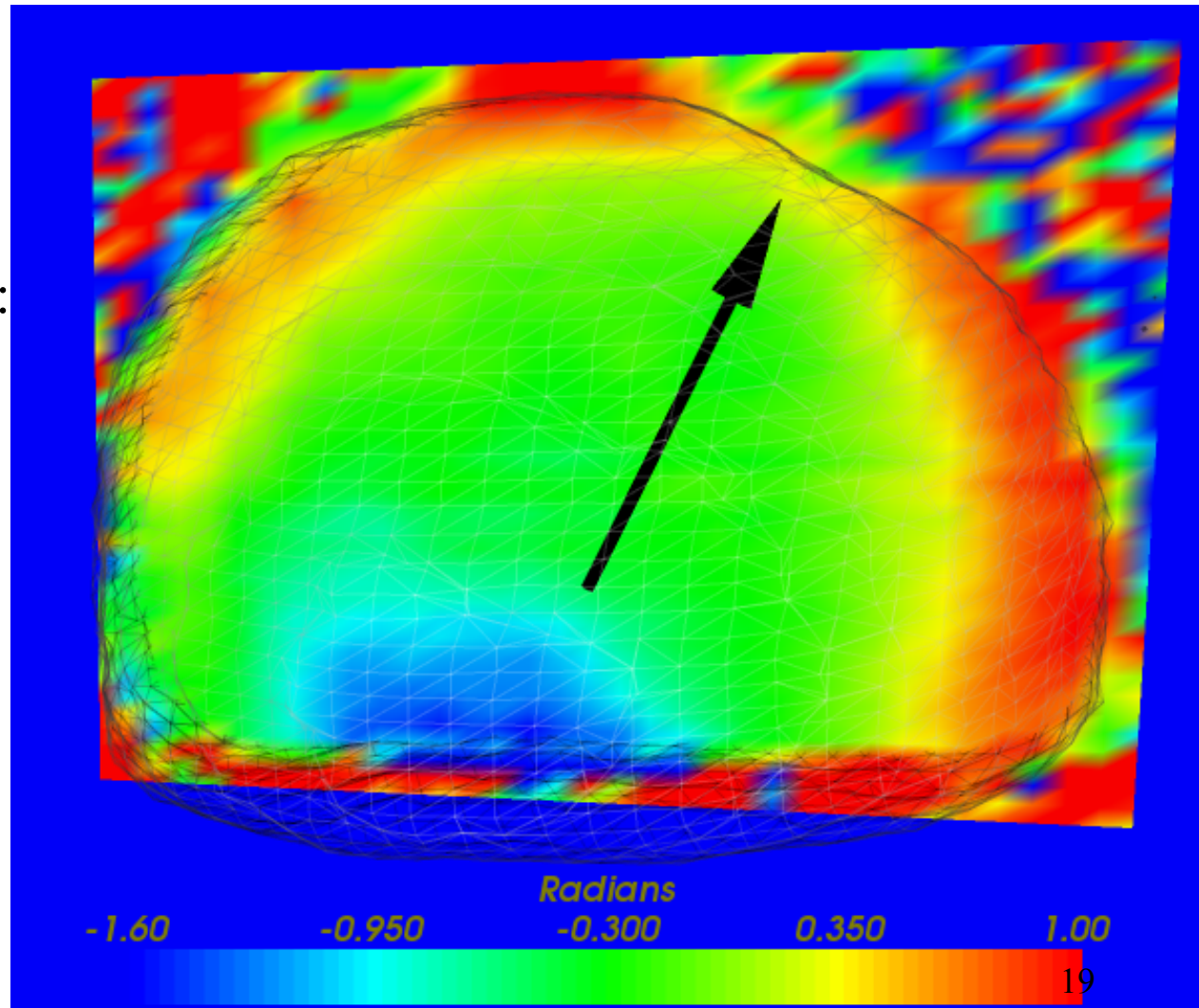
$$\delta=2.23\times 10^{-5}$$
$$\beta=2.19\times 10^{-6}$$



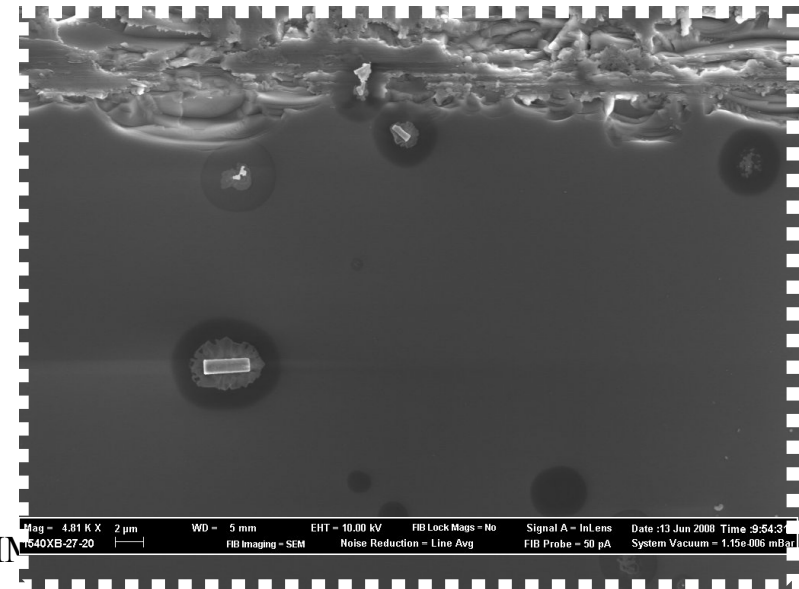
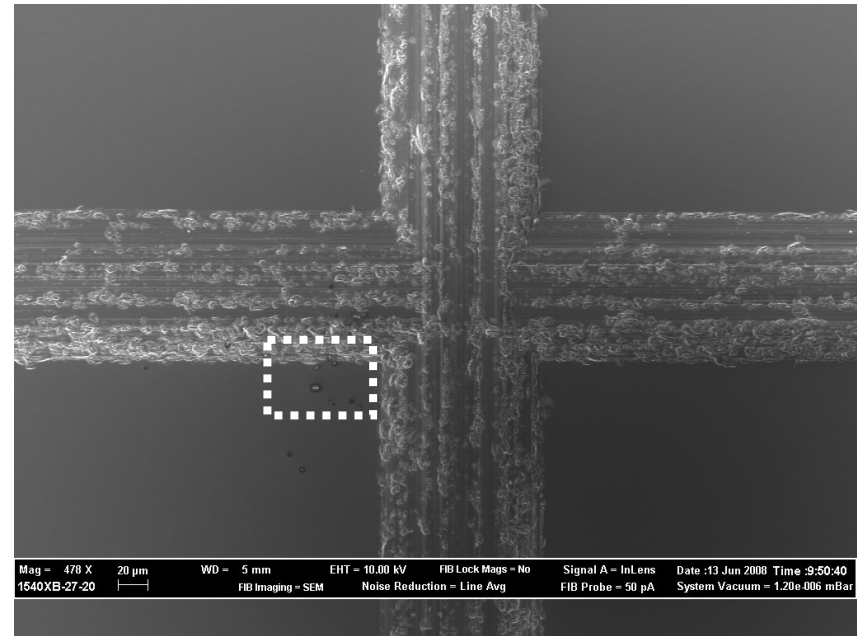
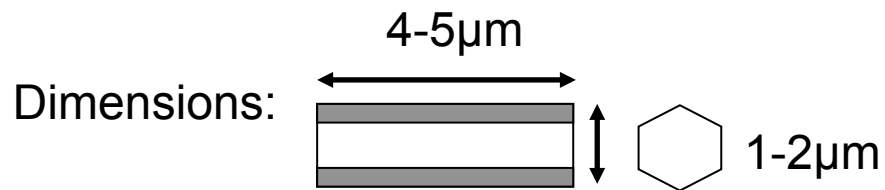
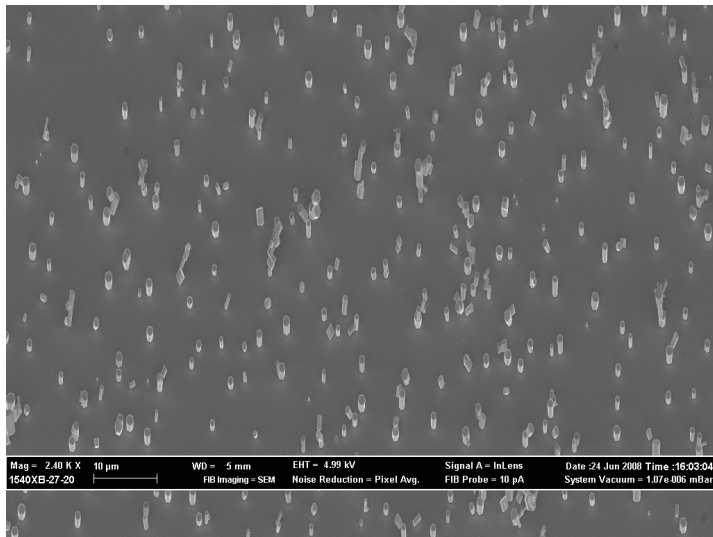
Refraction corrected phase map

Max phase = 1.15rad
= 0.052nm

Phase on the (111) facet:
= 0.47 rad
= 0.02nm

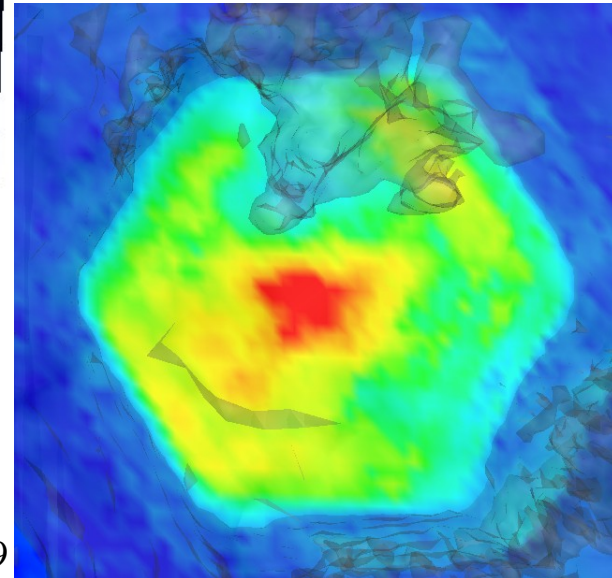
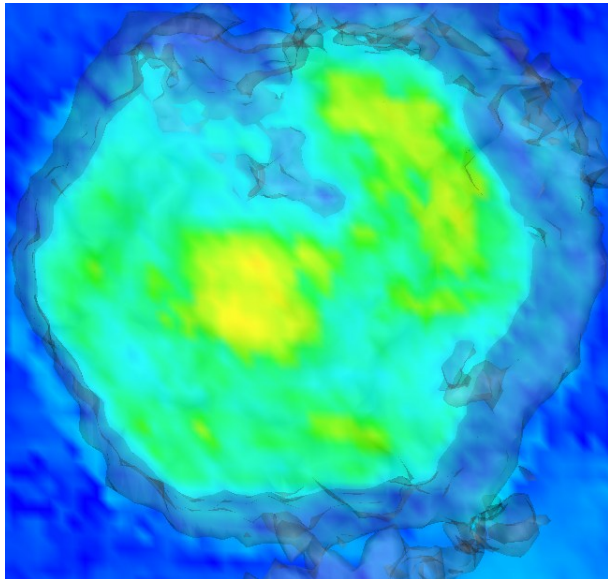
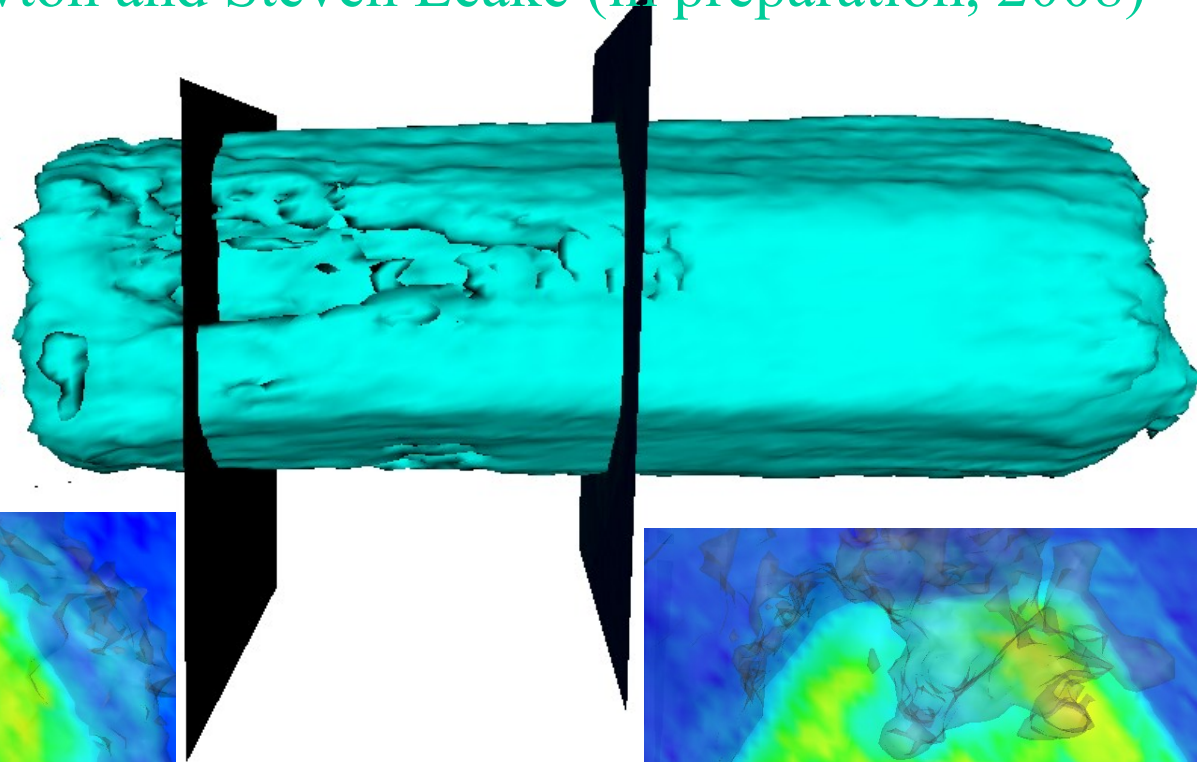


ZnO Sample Preparation



Density sections ZnO-39 (010)

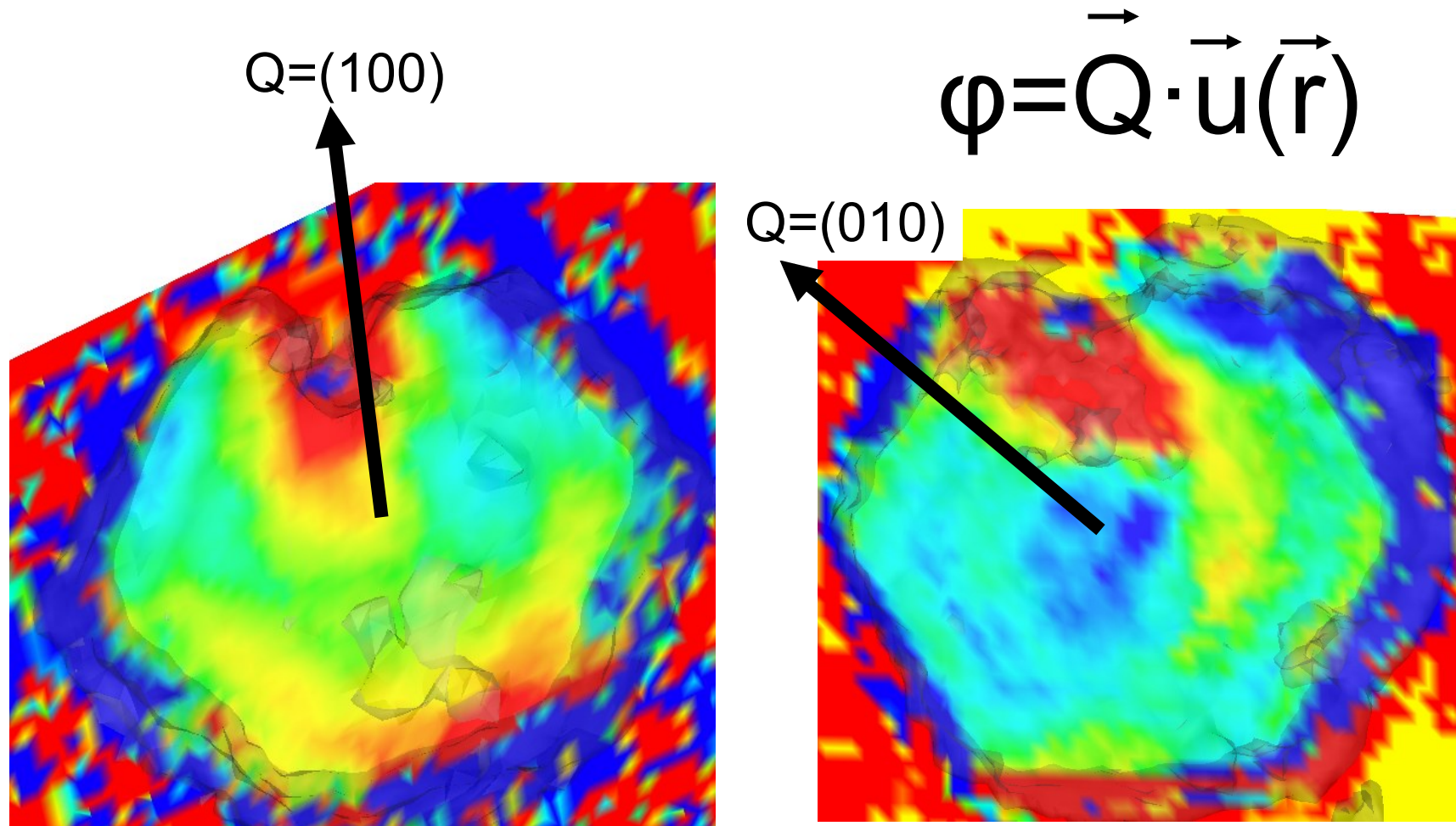
Marcus Newton and Steven Leake (in preparation, 2008)



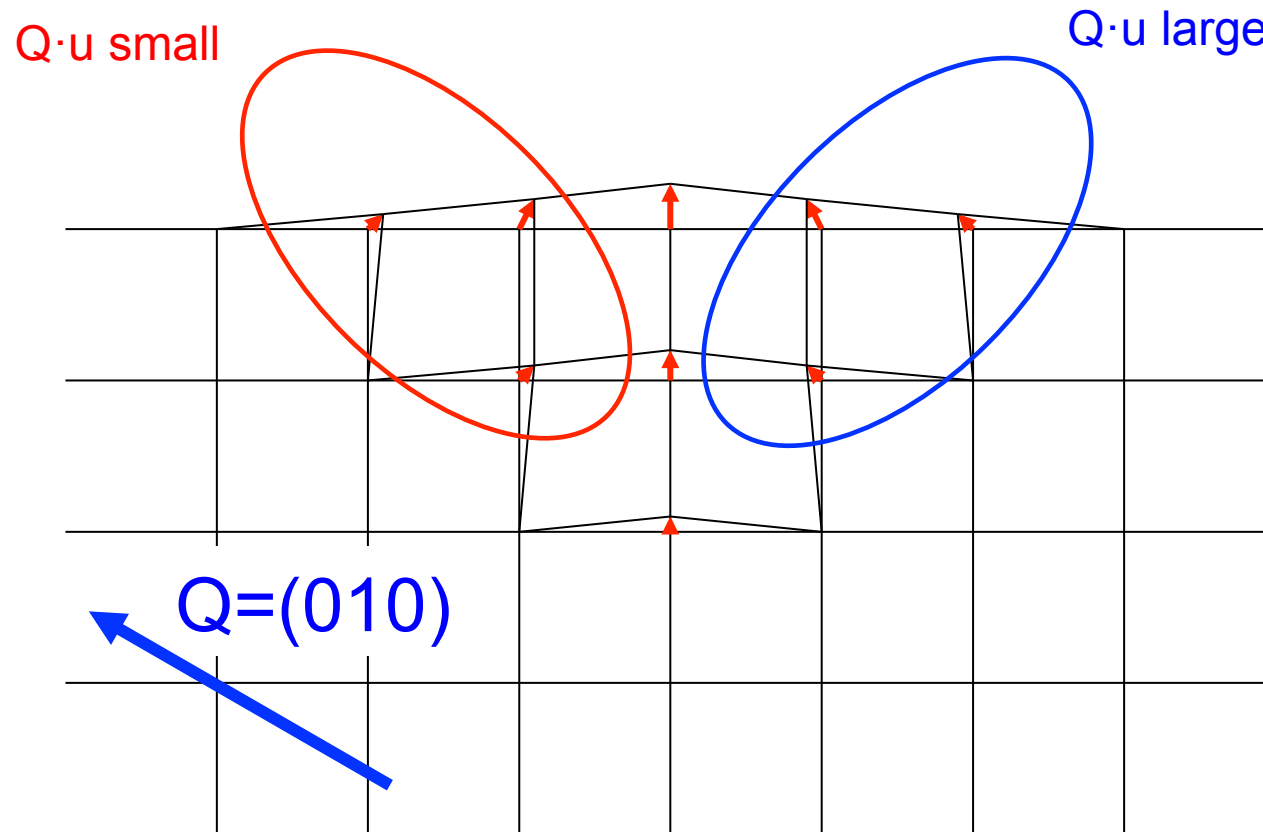
I. K. Robinson, CMMP 2009

Phase maps from 2 Bragg peaks

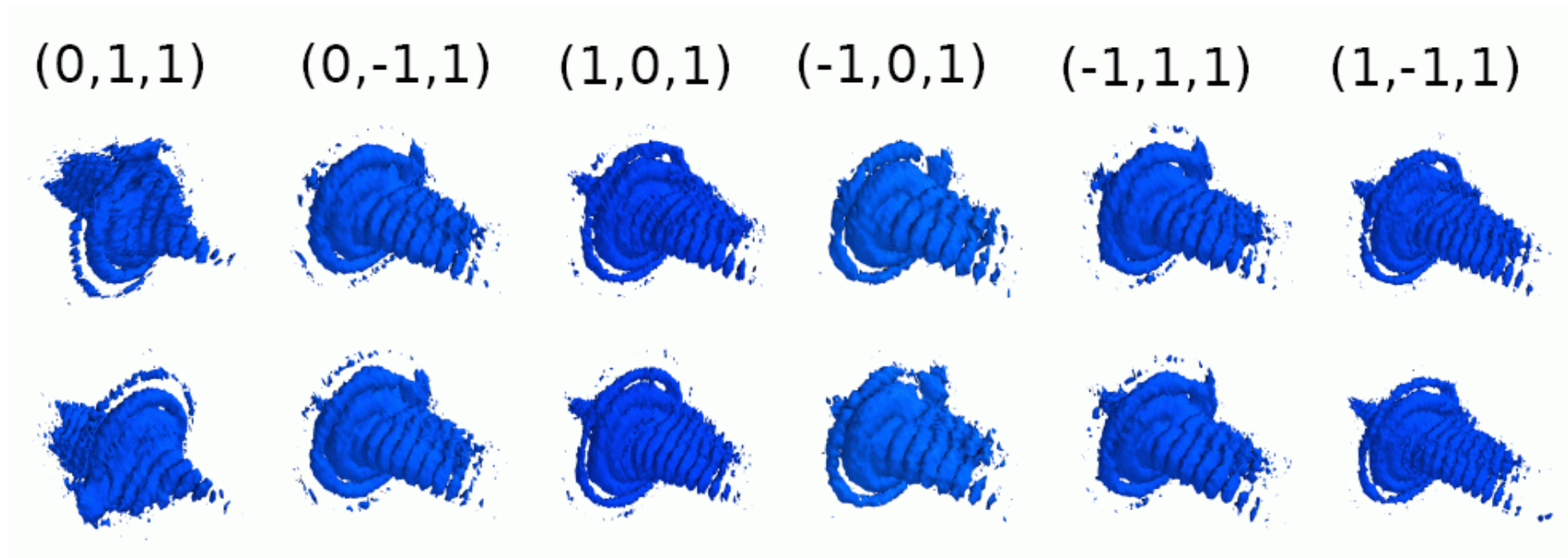
Blue-Red is +2 radians. Slice at -1500nm from centre ZnO-5 -39



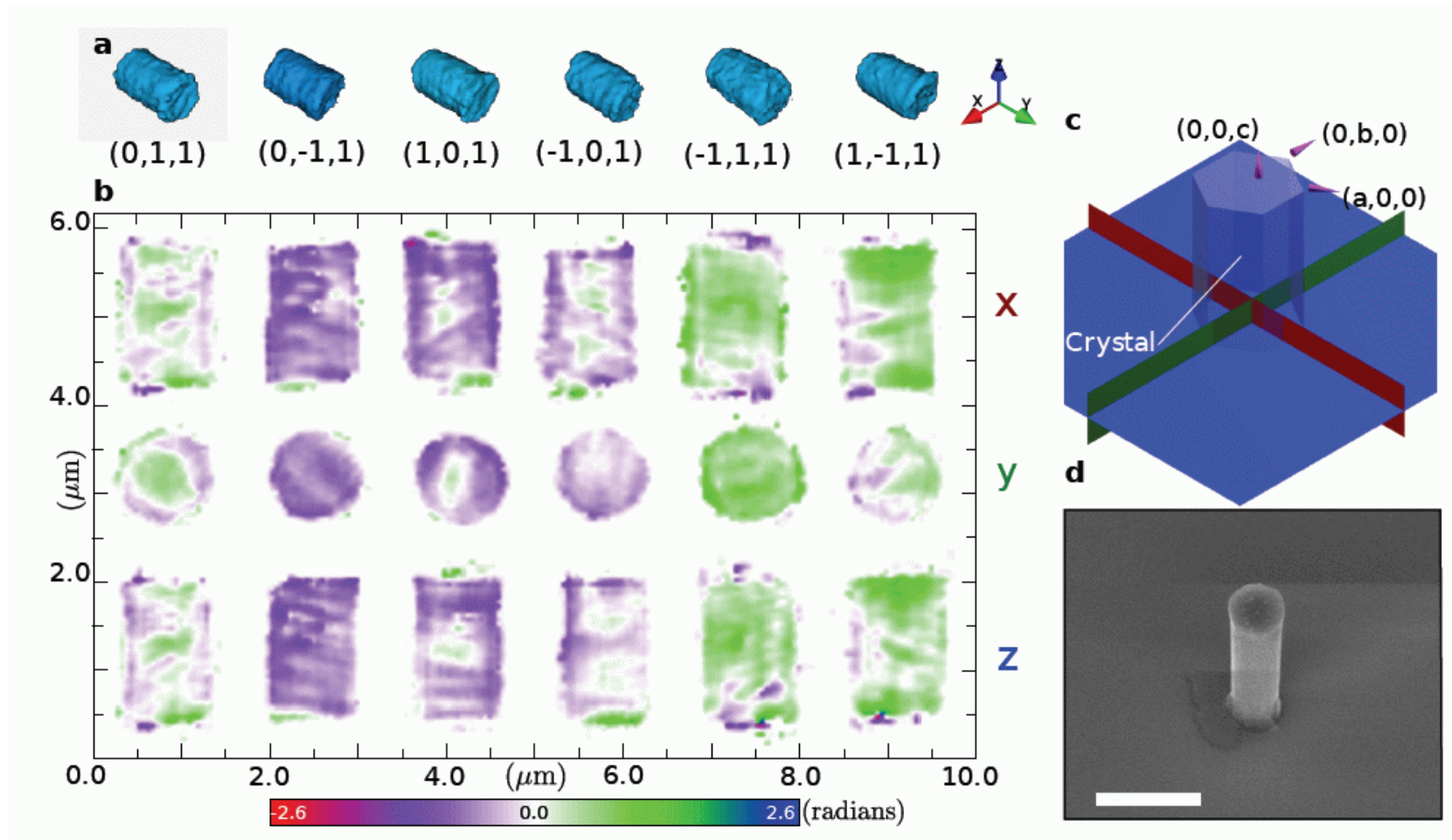
Typical displacement field



Extension to 6 Bragg Peaks

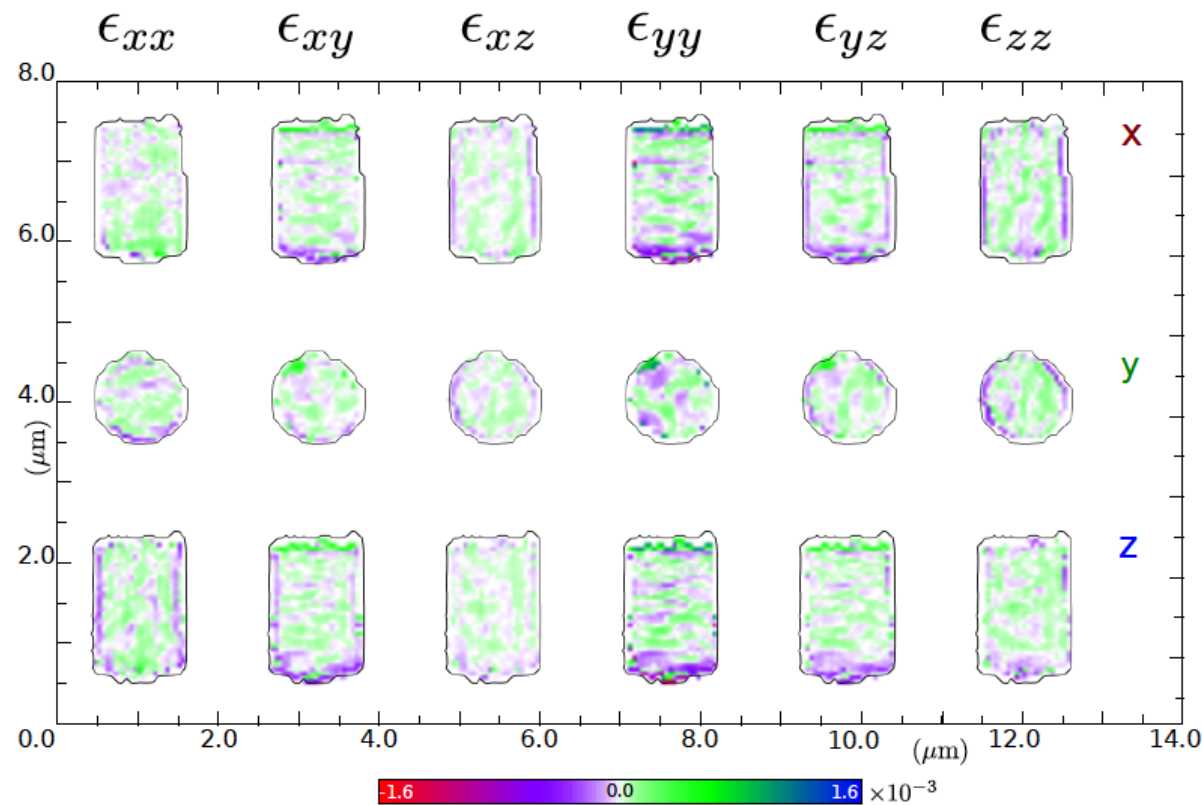


Extension to 6 Bragg Peaks

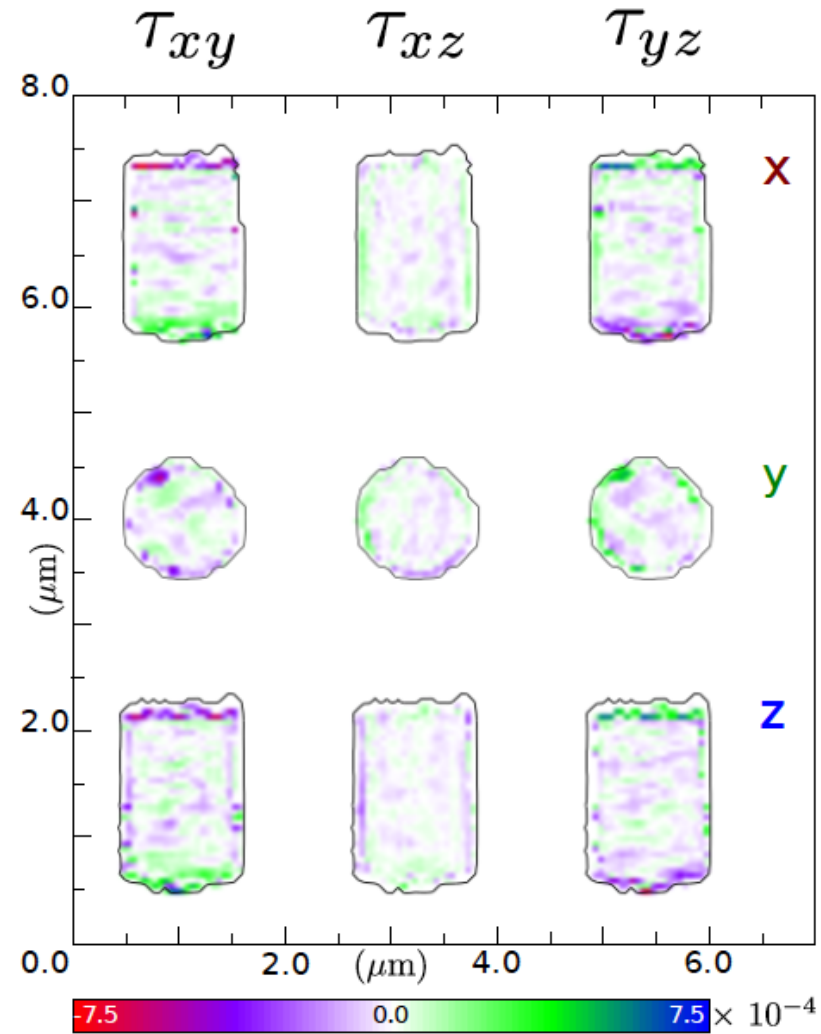


Full Strain Tensor

$$\epsilon_{ij} = \frac{1}{2} \left(\frac{\partial u_j}{\partial x_i} + \frac{\partial u_i}{\partial x_j} \right), \quad \tau_{ij} = \left(\frac{\partial u_j}{\partial x_i} - \frac{\partial u_i}{\partial x_j} \right)$$



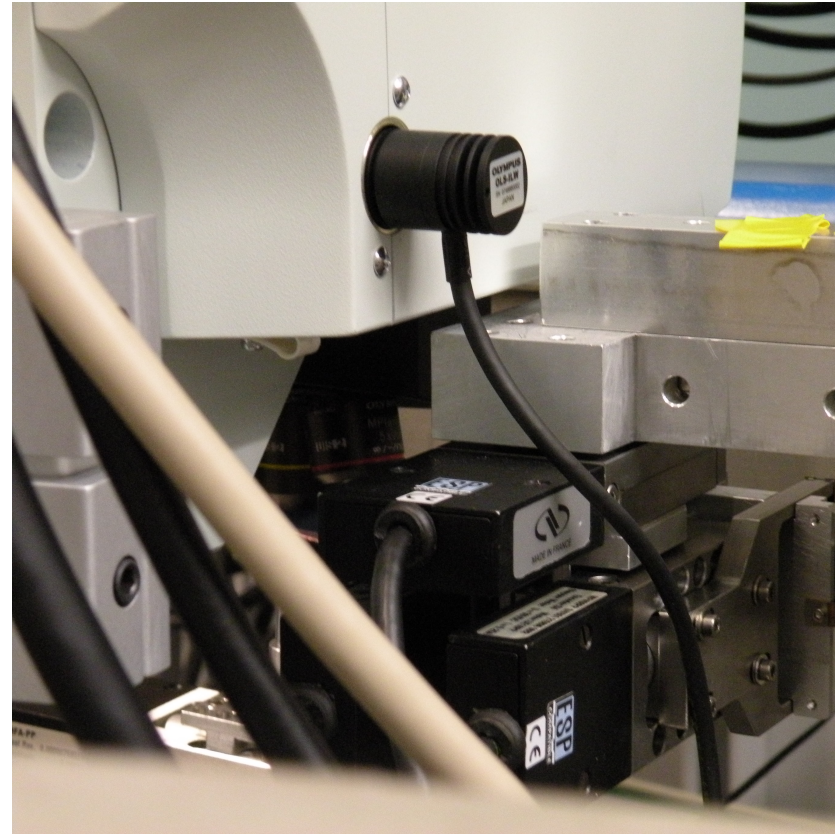
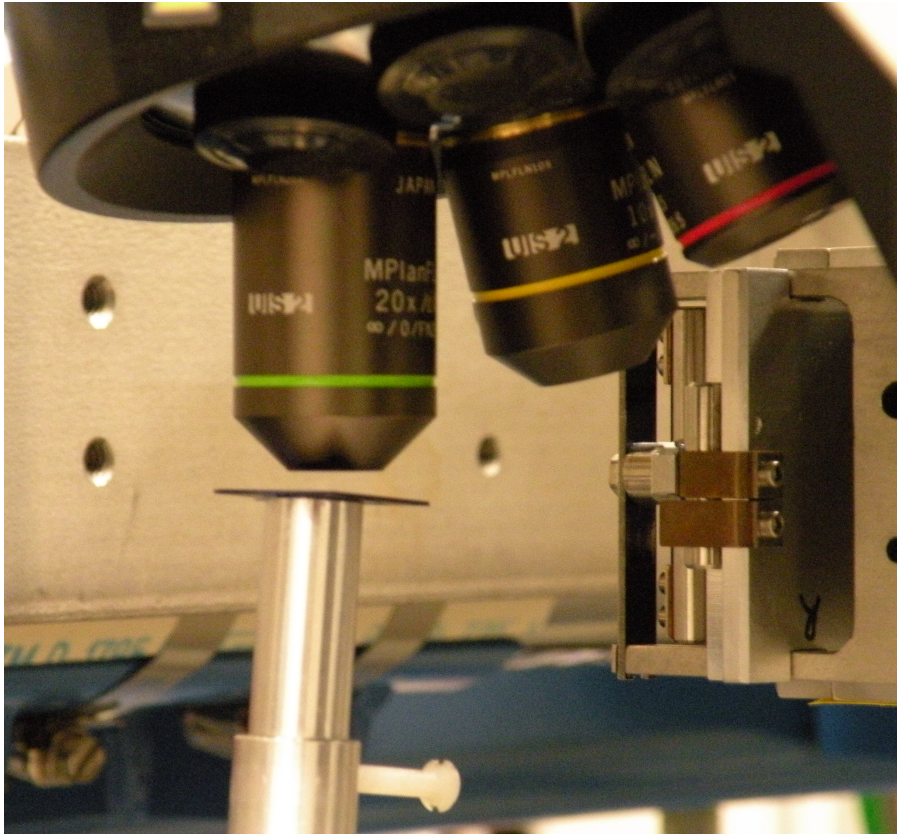
ZnO Rigid-body Rotations



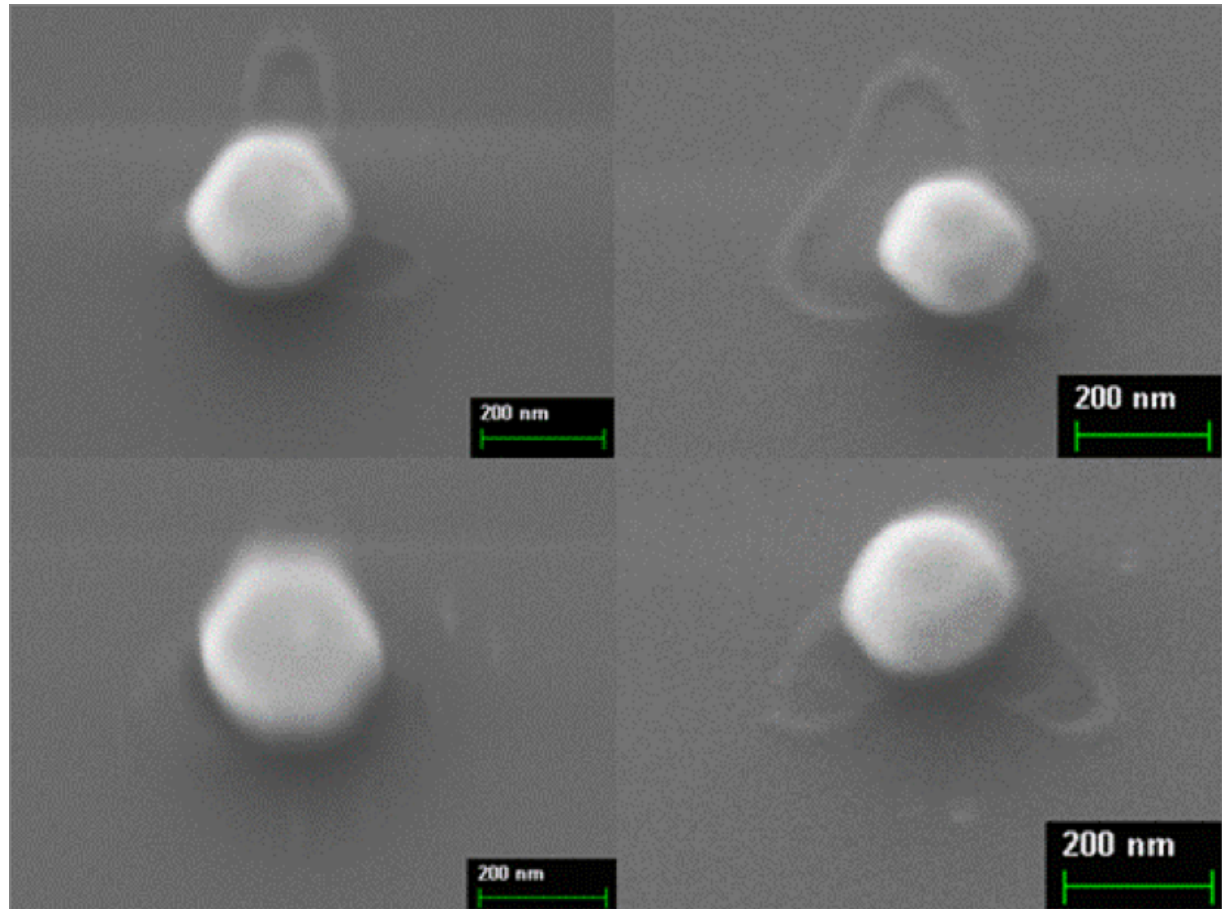
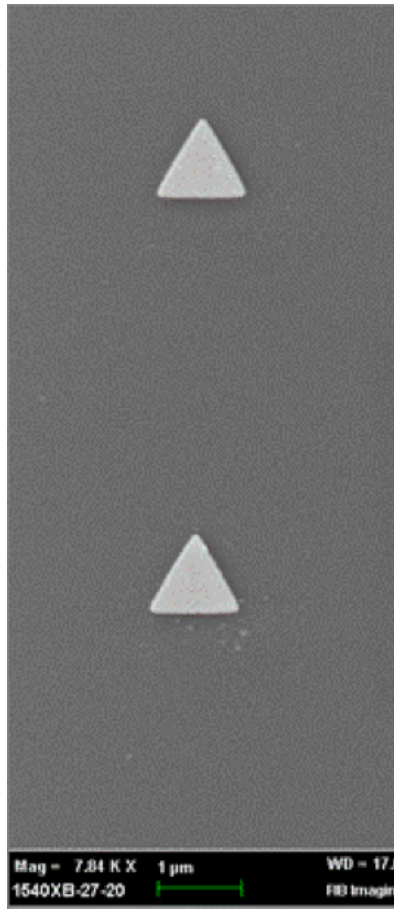
Conclusions

- Internal structure of Nanocrystals
- Phasing by computation instead of lens
- Strain fields imaged from asymmetric patterns
- Full Strain Tensor accessible

Confocal Alignment Microscope

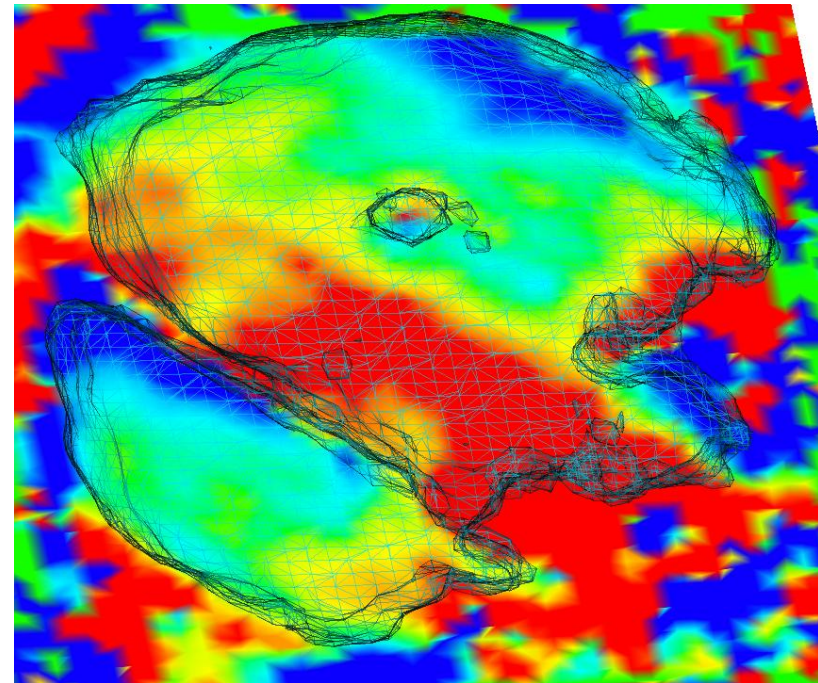
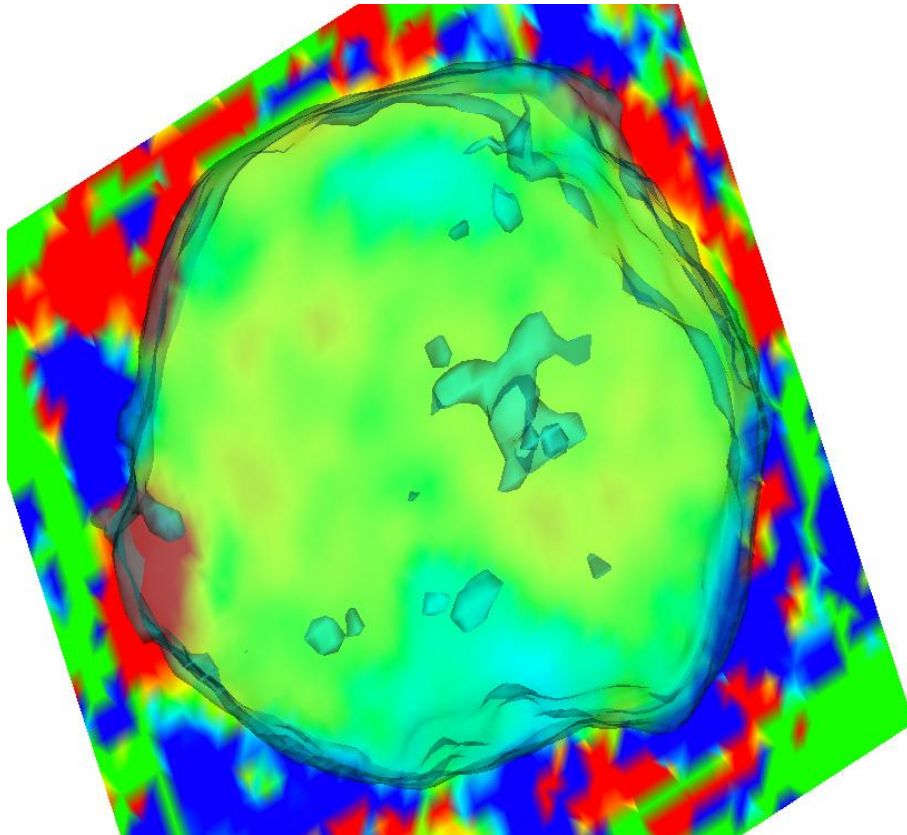


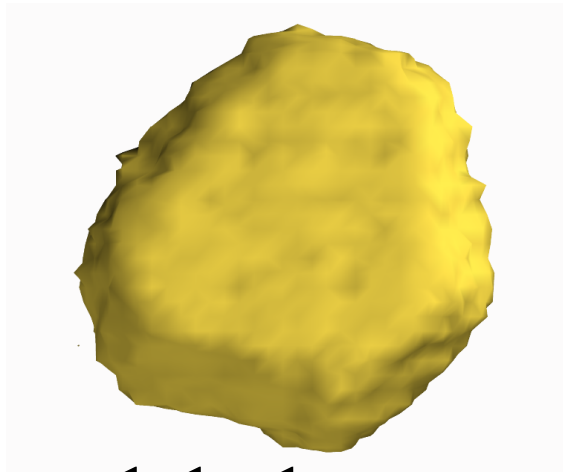
Single Au nanocrystal synthesis



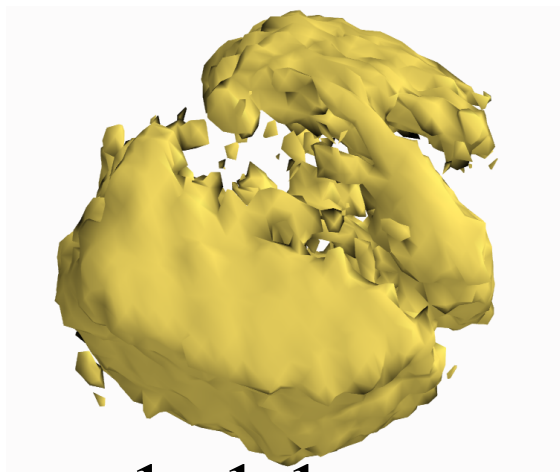
Two views of strain in Au NC

Au409B-52 (11-1) and Au409B-60 (200)

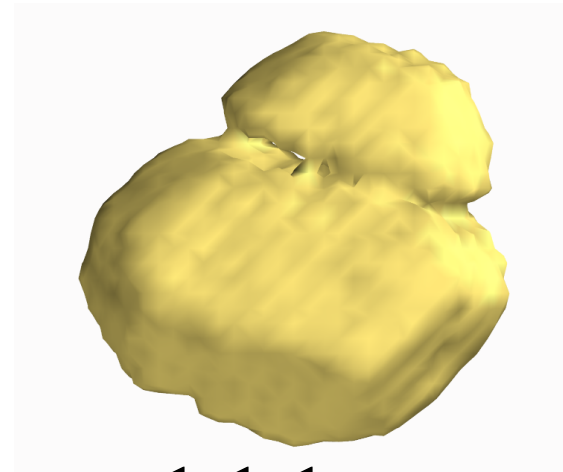




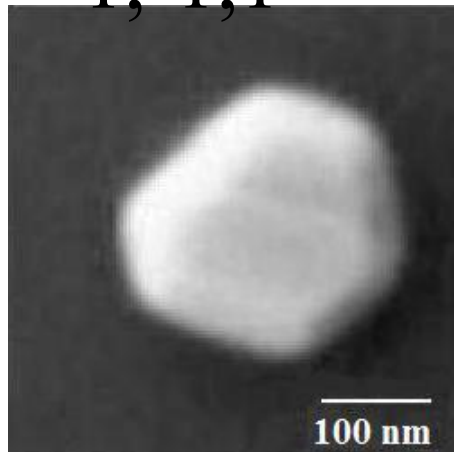
1,1,-1



1,-1,1

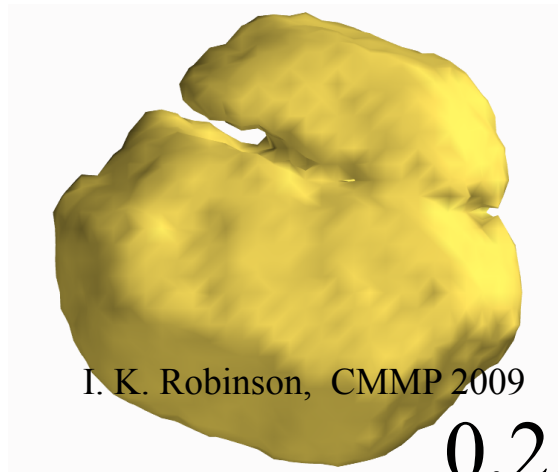
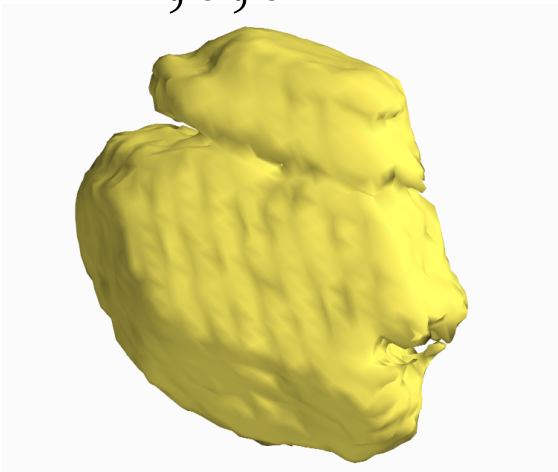


-1,1,1

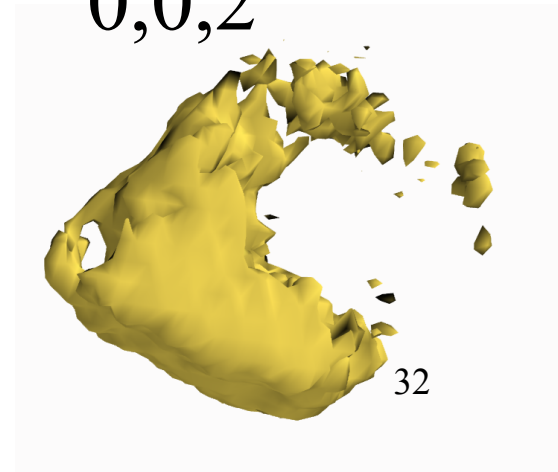


100 nm

2,0,0



0,2,0



0,0,2

I. K. Robinson, CMMP 2009

Vector displacement field

Merged reconstructions from (11-1) (020) and (-111)

