

Confocal Microscope Alignment of Nanocrystals for Coherent Diffraction Imaging

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Diamond Light Source

Synchrotron Radiation

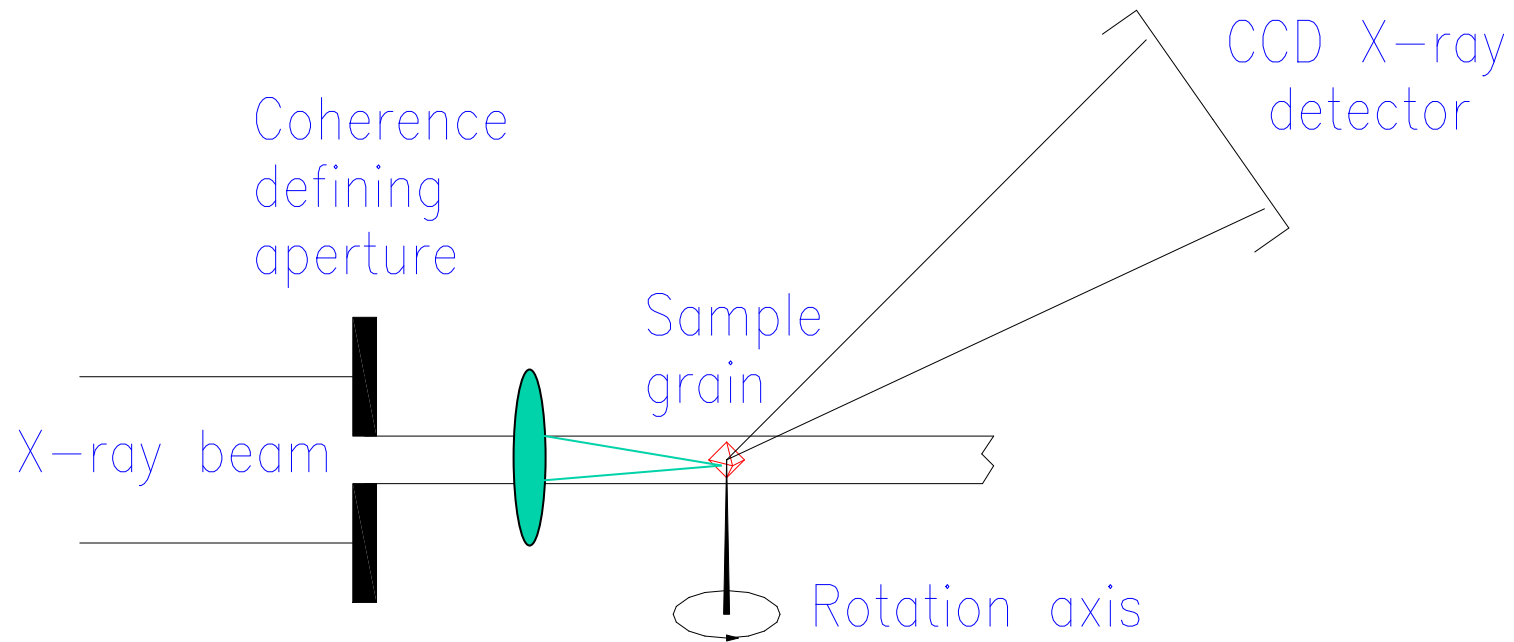
Instrumentation Conference

Melbourne, September 2009

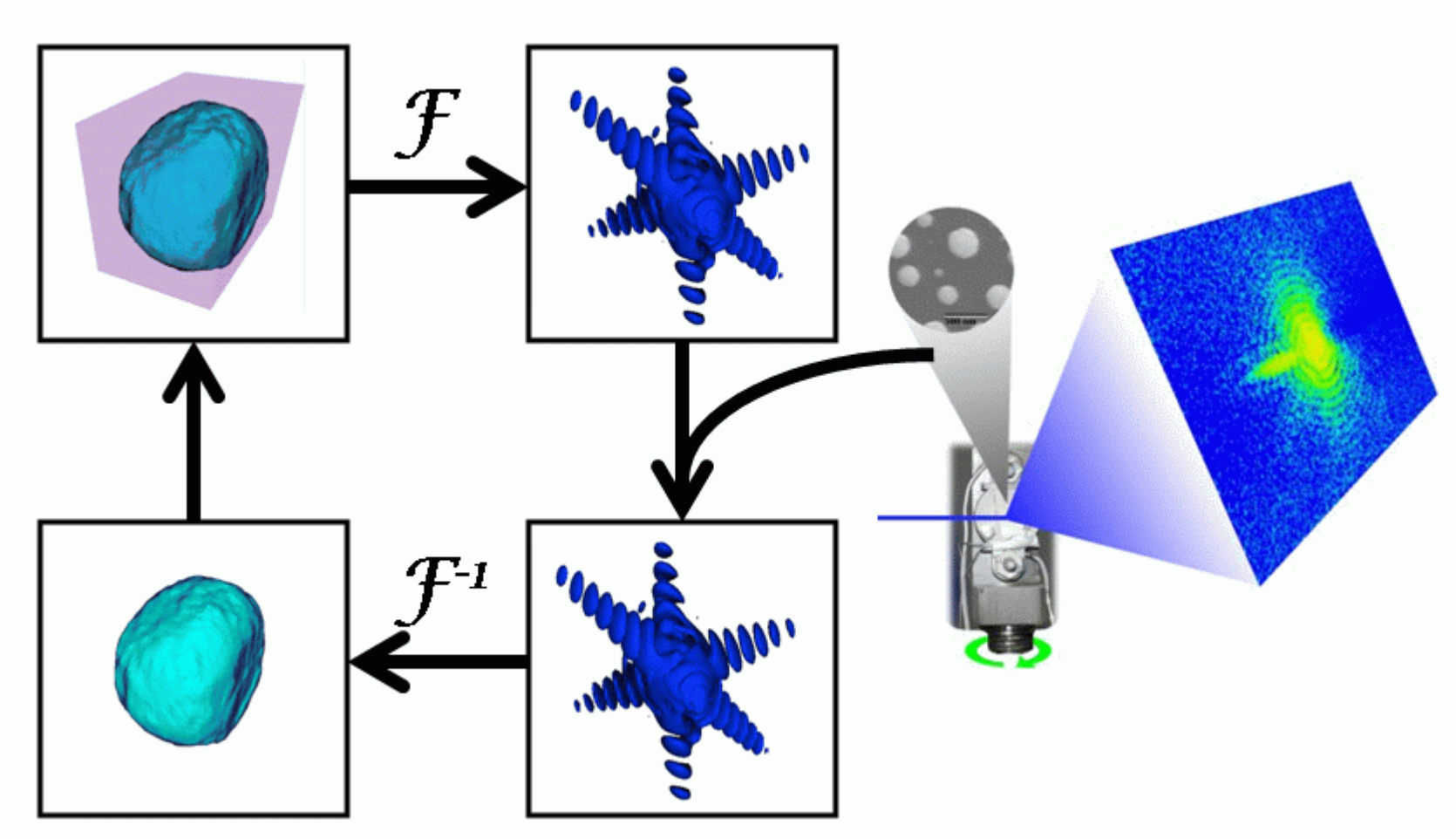
Outline

- Coherent x-ray diffraction
- Coherence solves the **phase** problem
- Exploration of crystal strain in ZnO
- Effects of longitudinal coherence
- Confocal microscope at 34-ID-C
- Twin domains in Au nanocrystals

Lensless X-ray Microscope, 2003



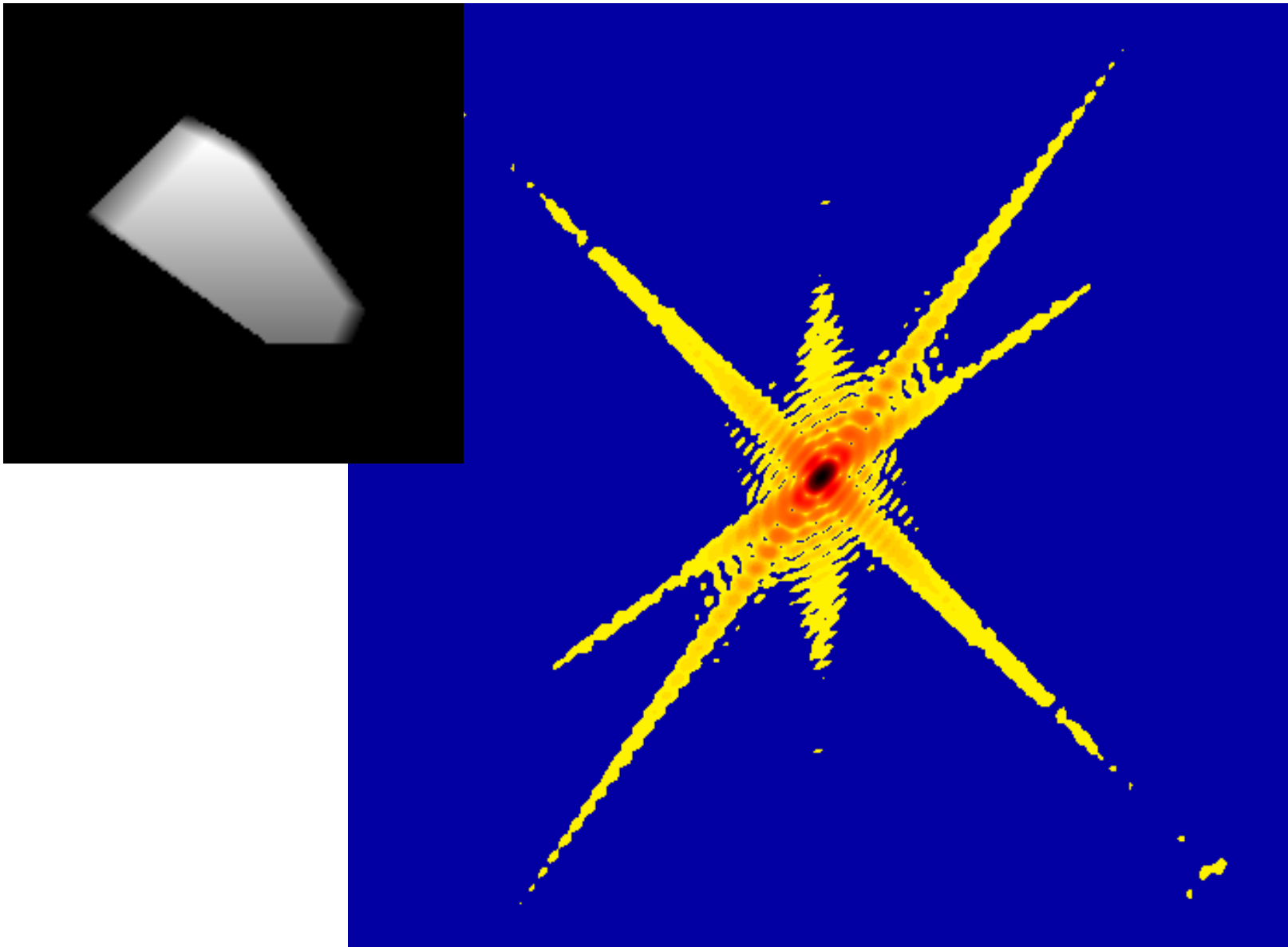
Generic “Error Reduction” method

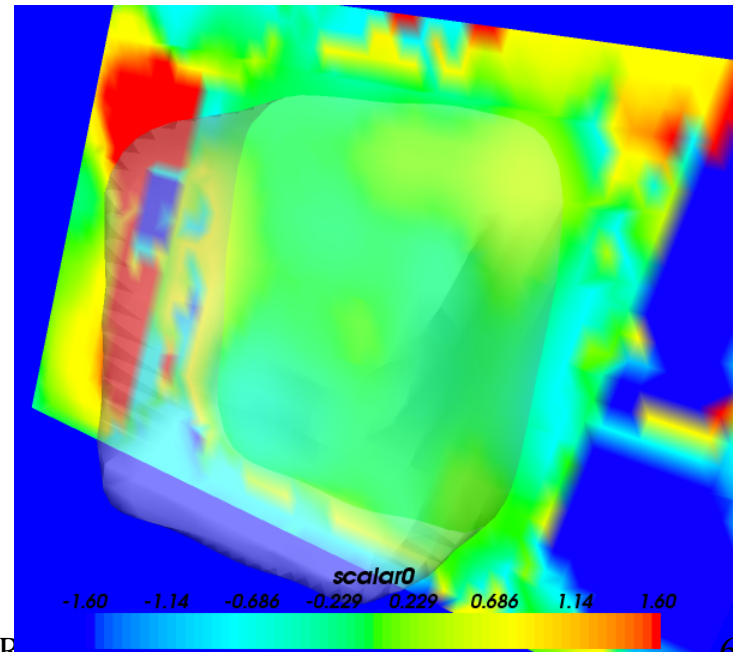
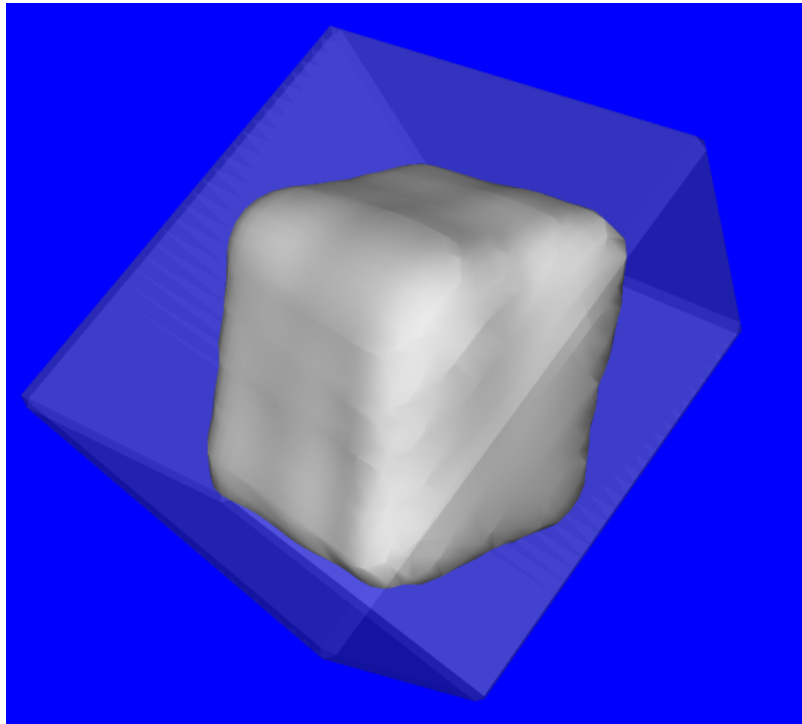
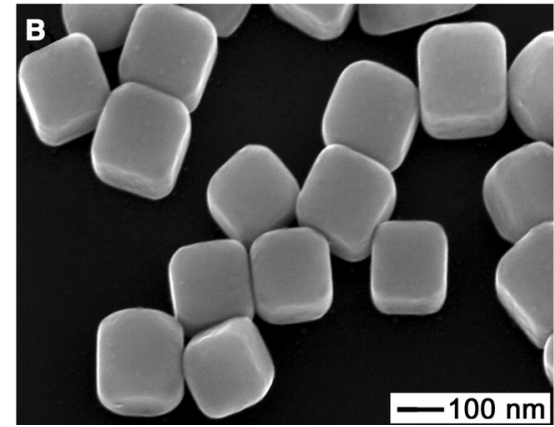
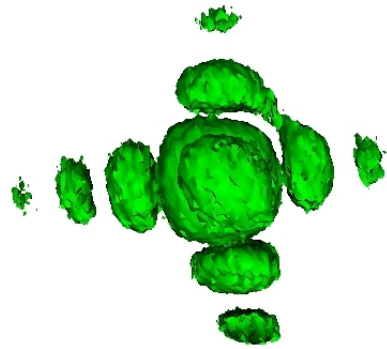
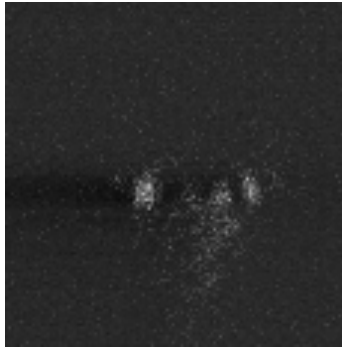


J. R. Fienup *Appl. Opt.* 21 2758 (1982)

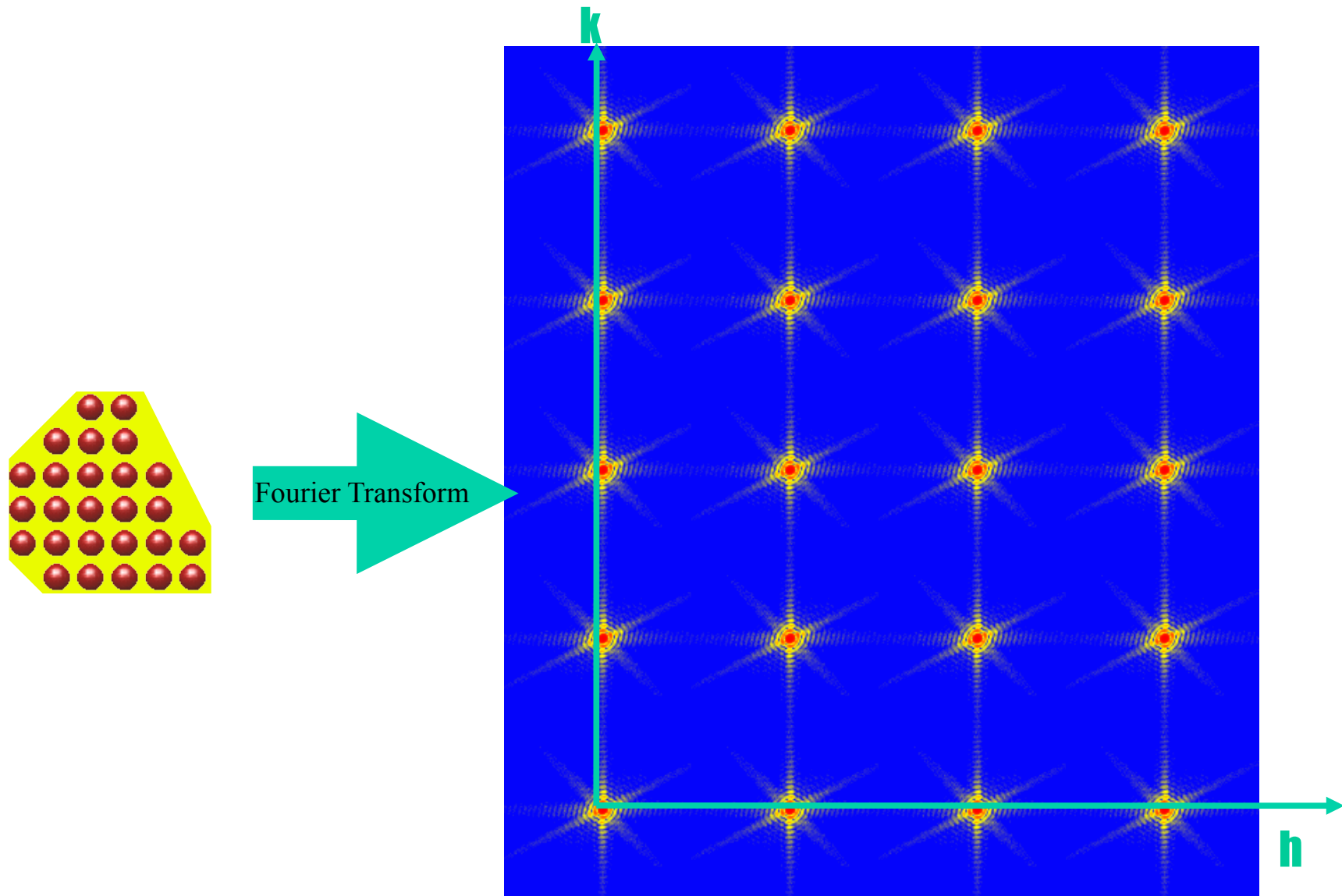
R. W. Gerchberg and W. O. Saxton *Optik* 35 237 (1972)

I. K. Robinson, SRI 2009



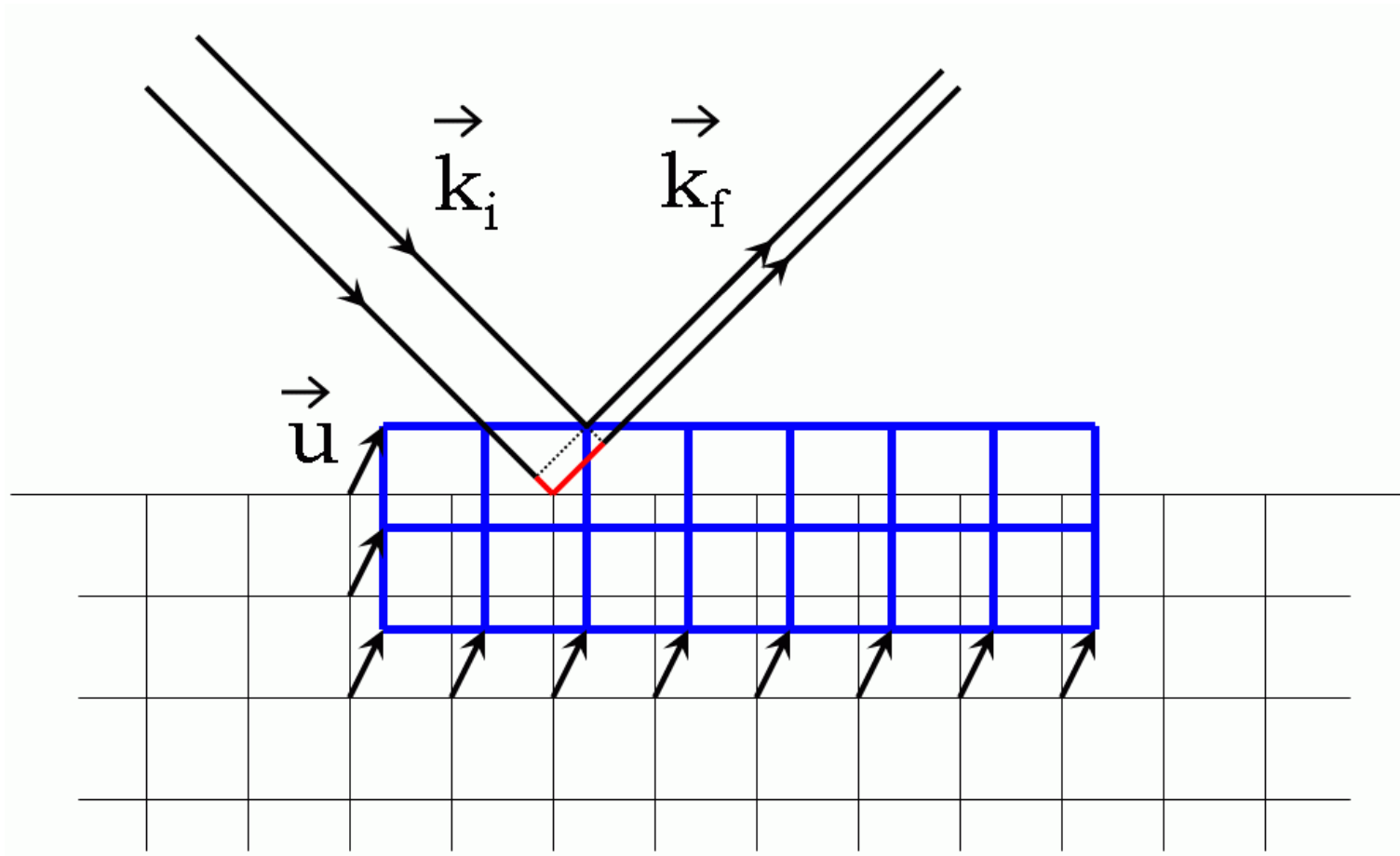


Coherent Diffraction from Crystals



Sensitivity to strain

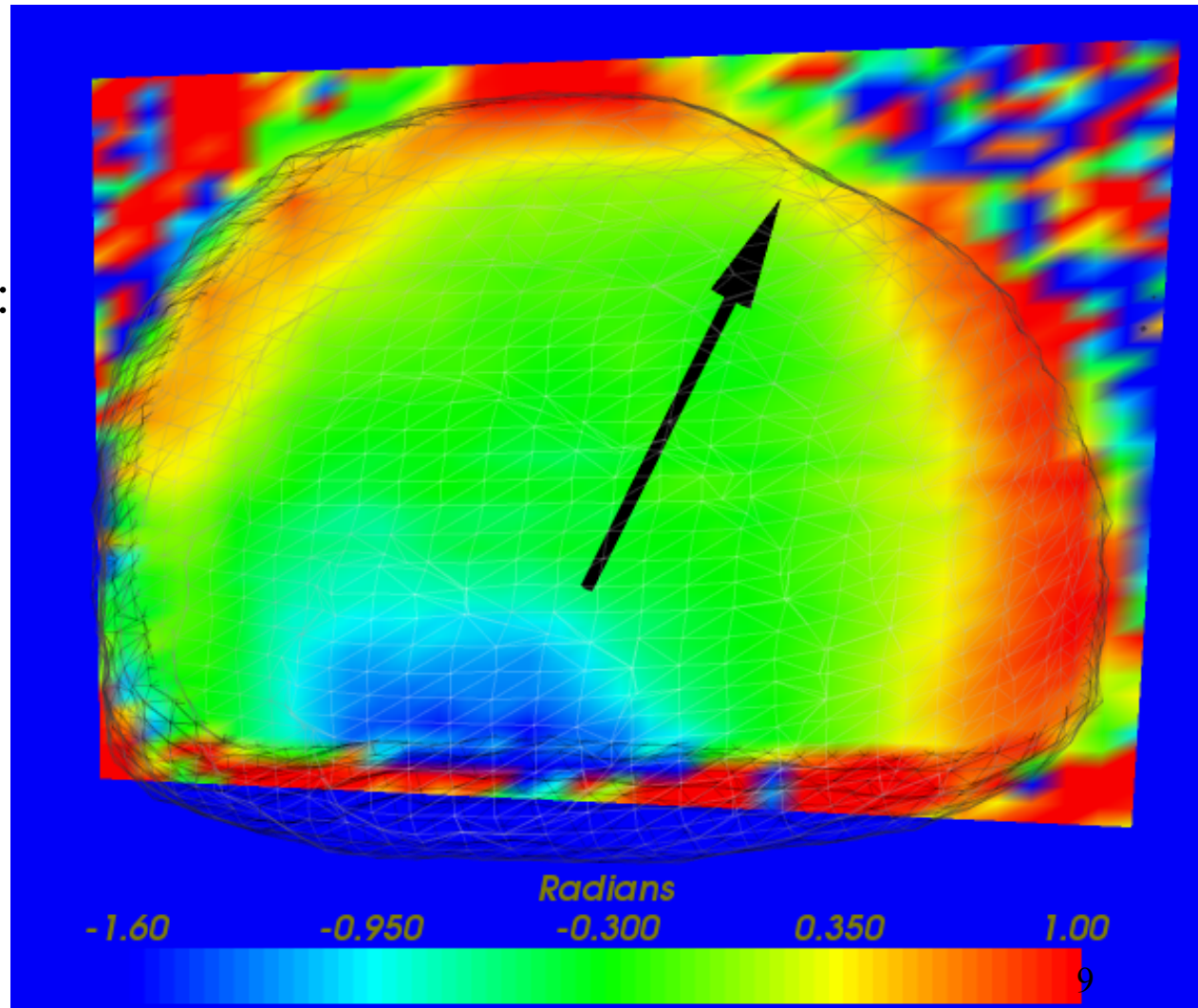
$$\Delta\varphi = \mathbf{k}_f \cdot \mathbf{u} - \mathbf{k}_i \cdot \mathbf{u} = \mathbf{Q} \cdot \mathbf{u}$$



Refraction corrected phase map

Max phase = 1.15rad
= 0.052nm

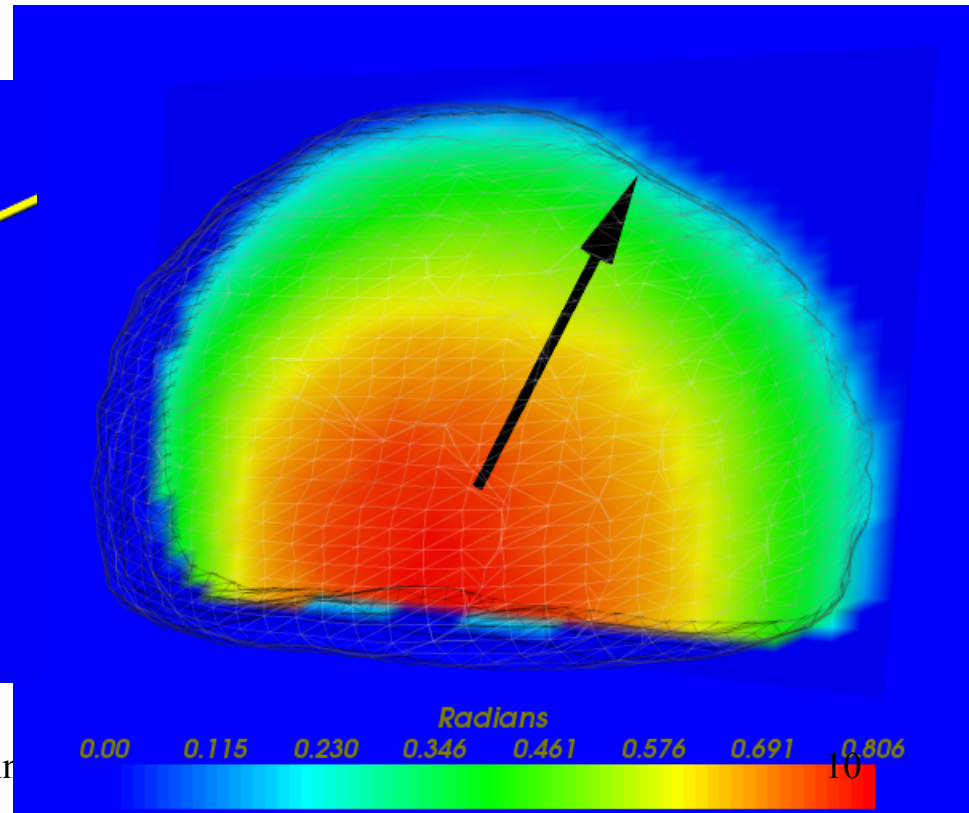
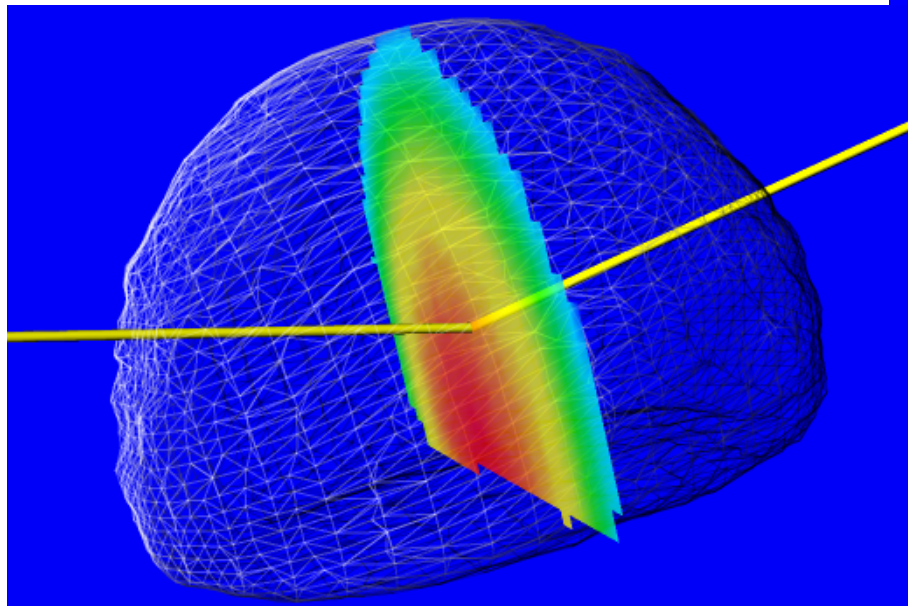
Phase on the (111) facet:
= 0.47 rad
= 0.02nm



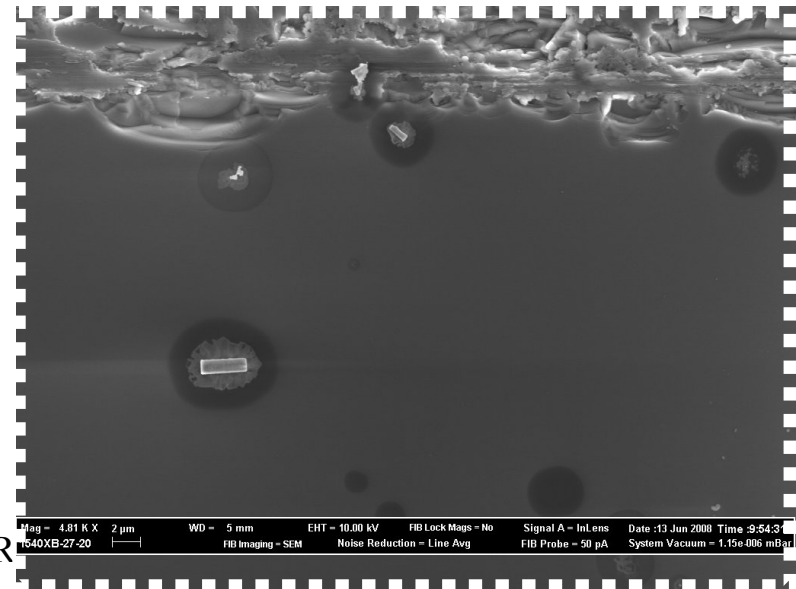
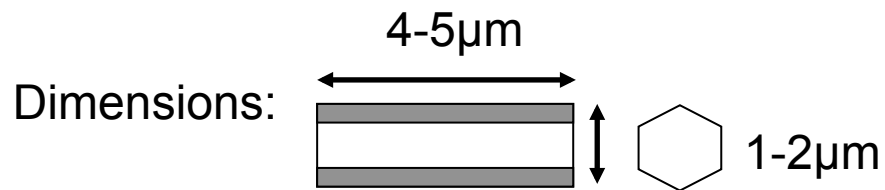
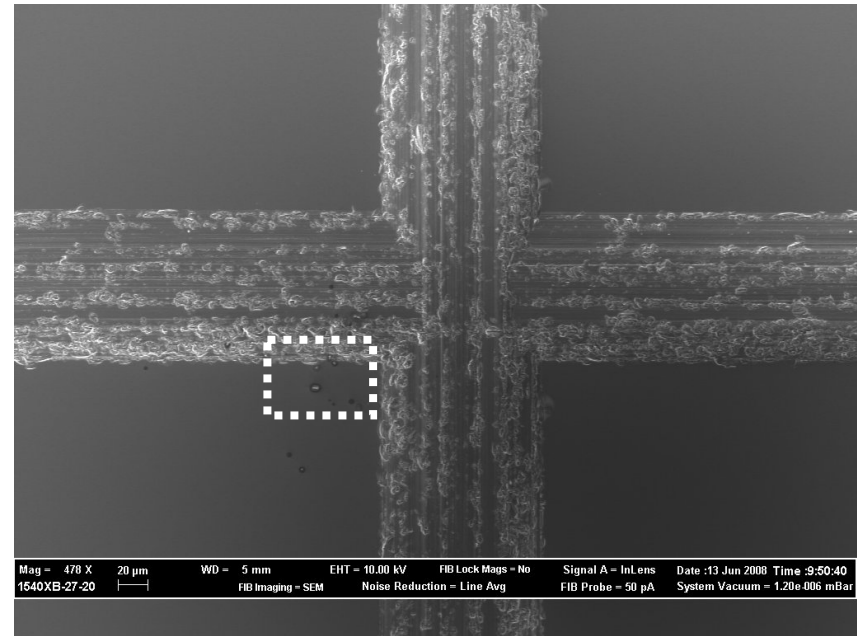
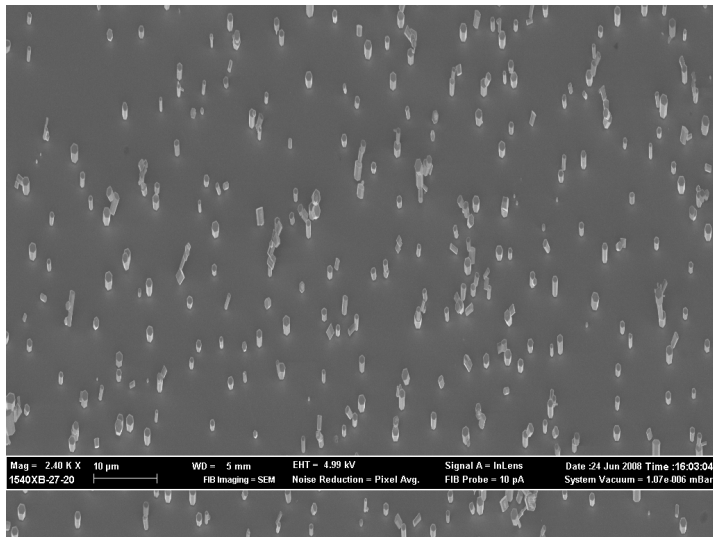
Refraction effects in Lead at 8.9keV

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

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



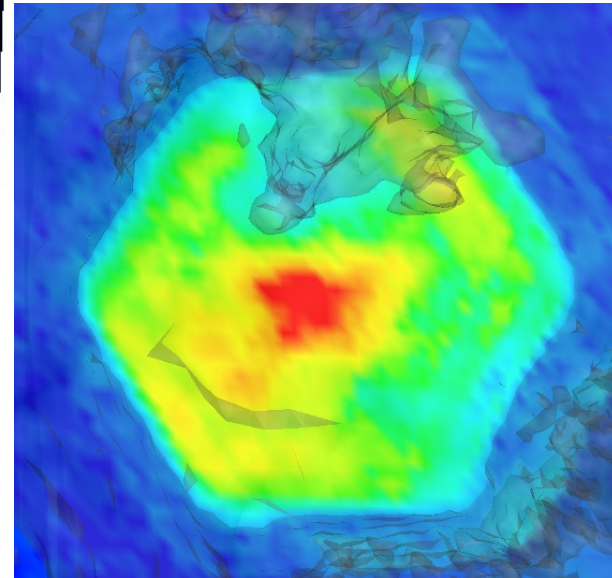
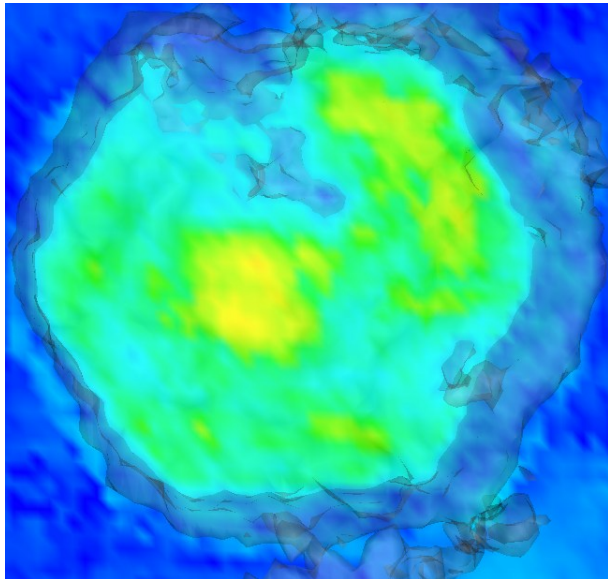
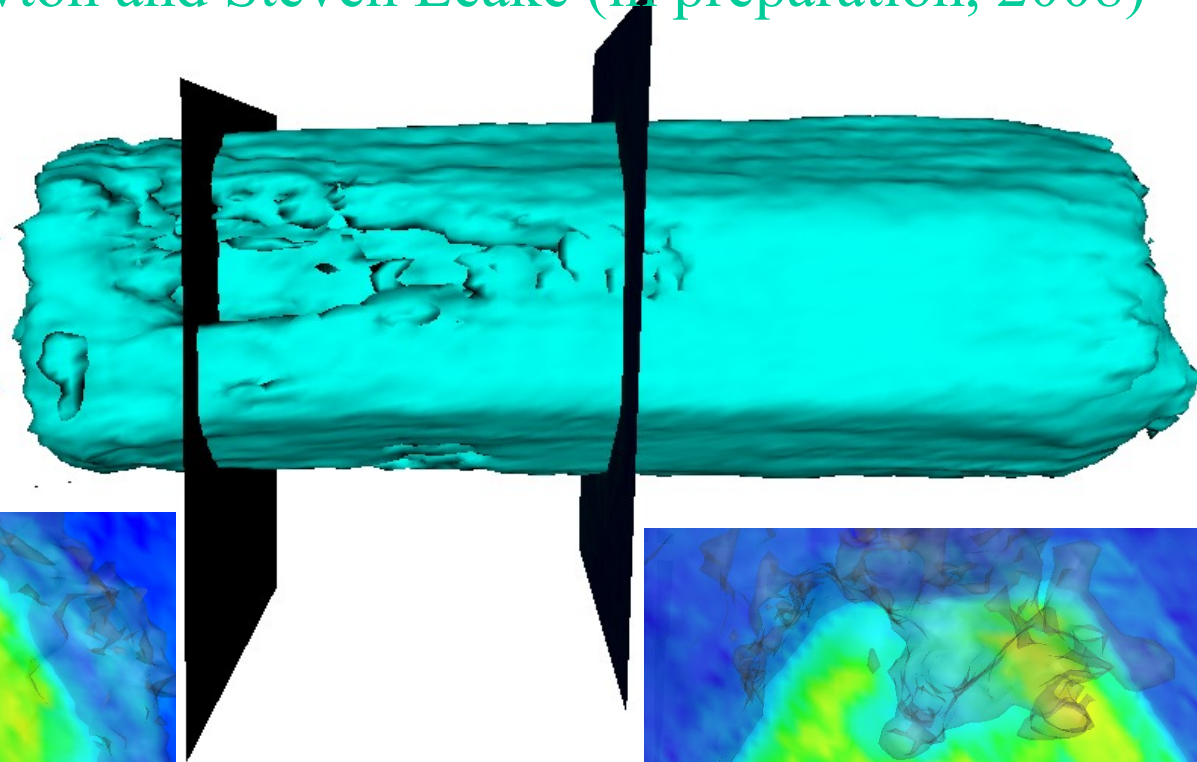
ZnO Sample Preparation



I. K. Robinson, SR

Density sections ZnO-39 (010)

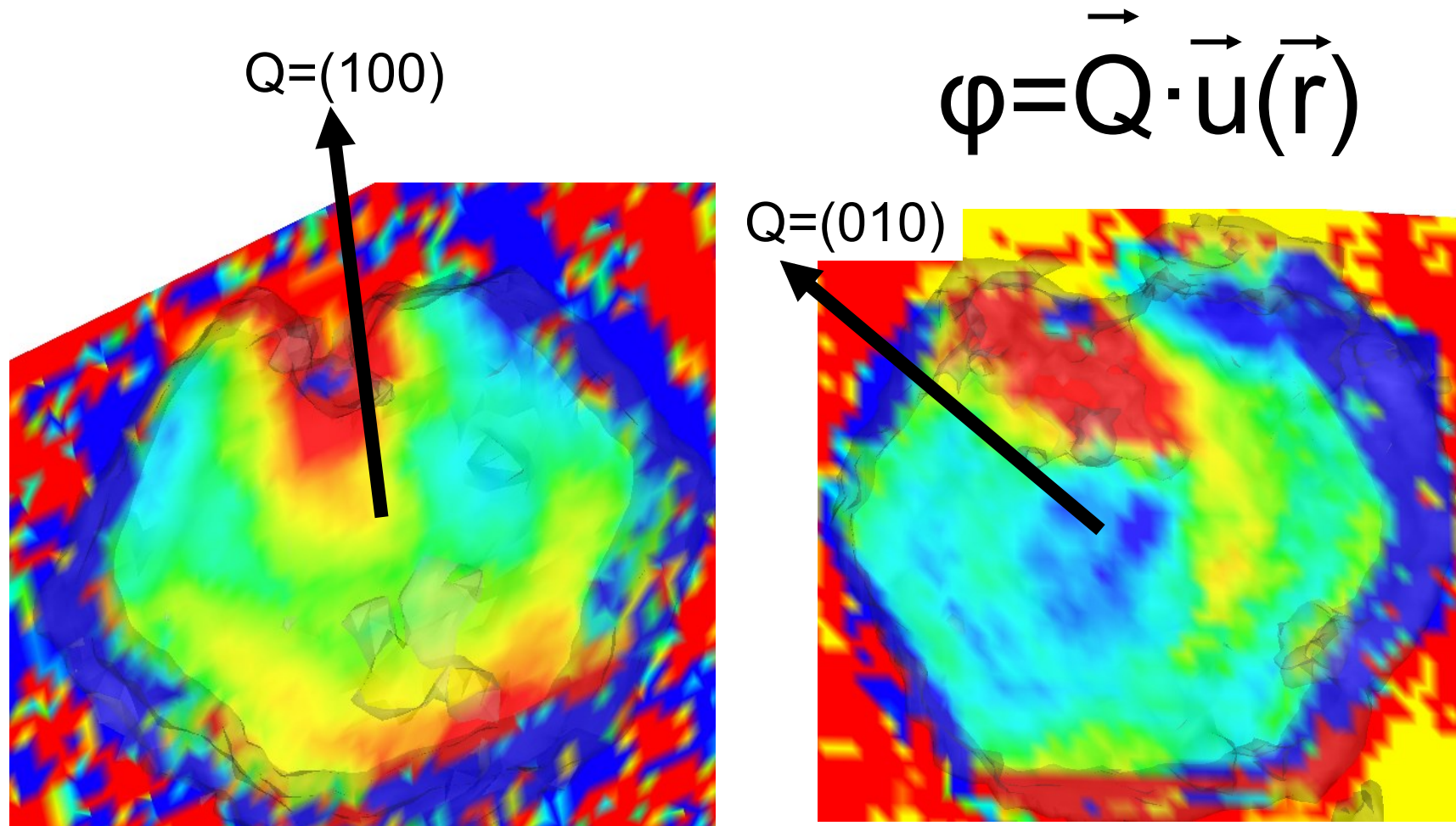
Marcus Newton and Steven Leake (in preparation, 2008)



I. K. Robinson, SRI 2009

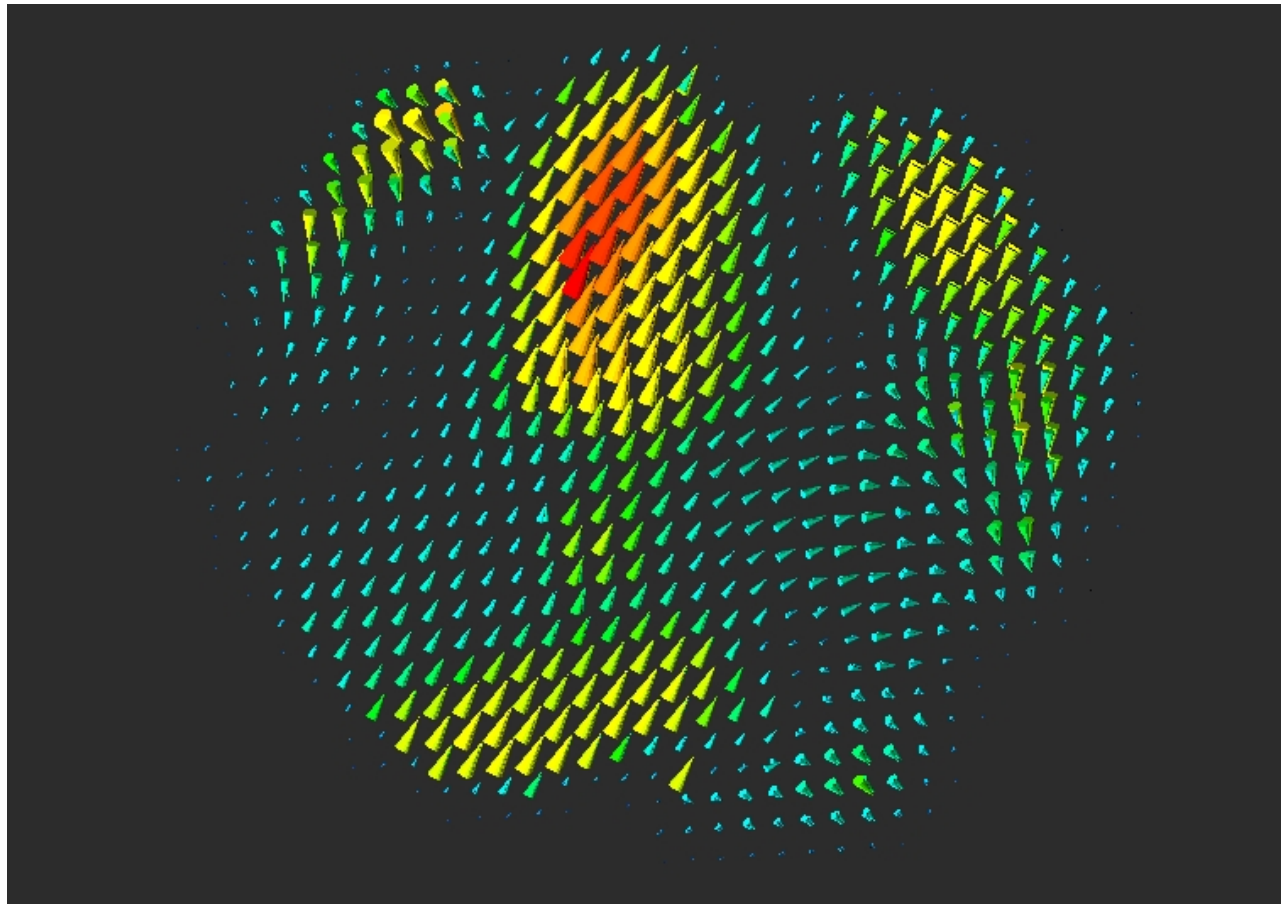
Phase maps from 2 Bragg peaks

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

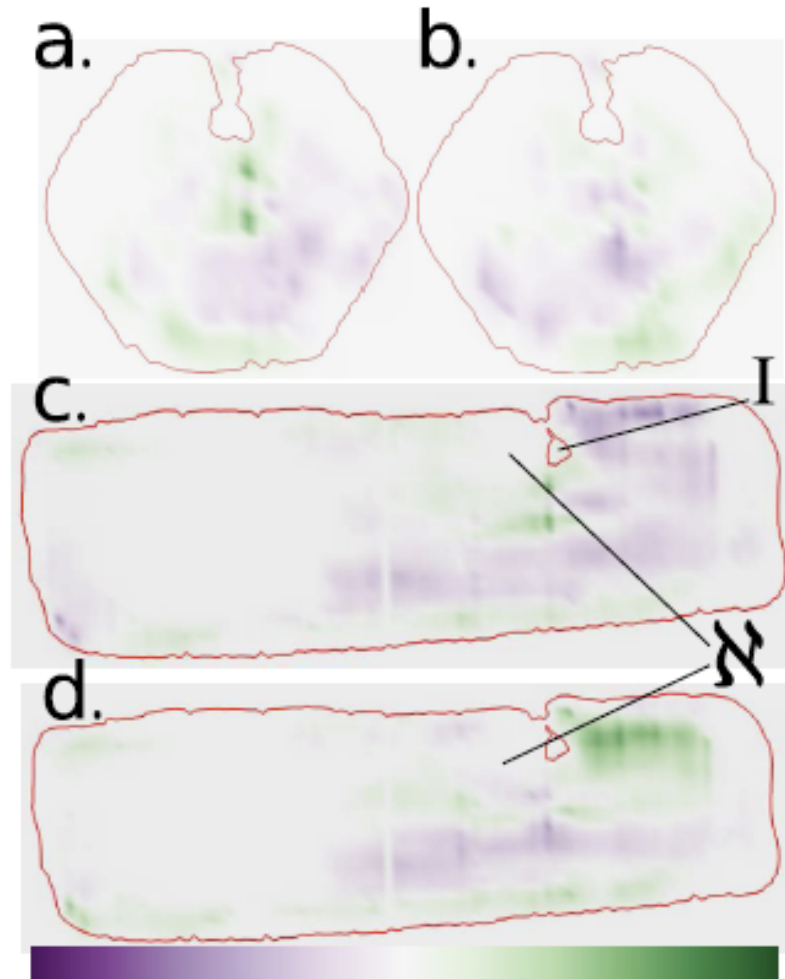
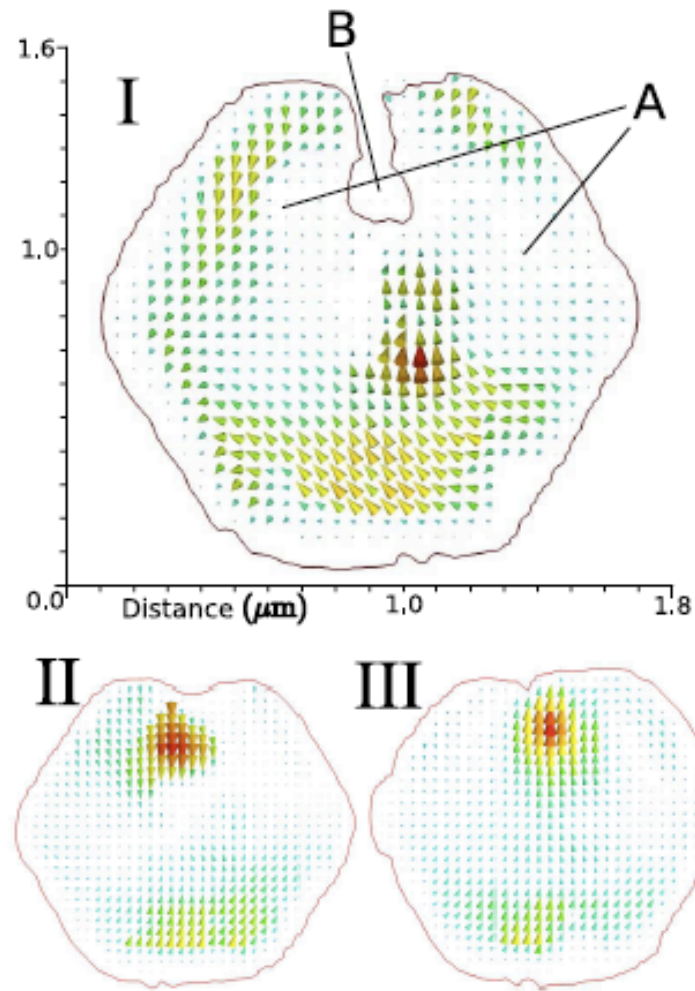


2D vector field of displacements

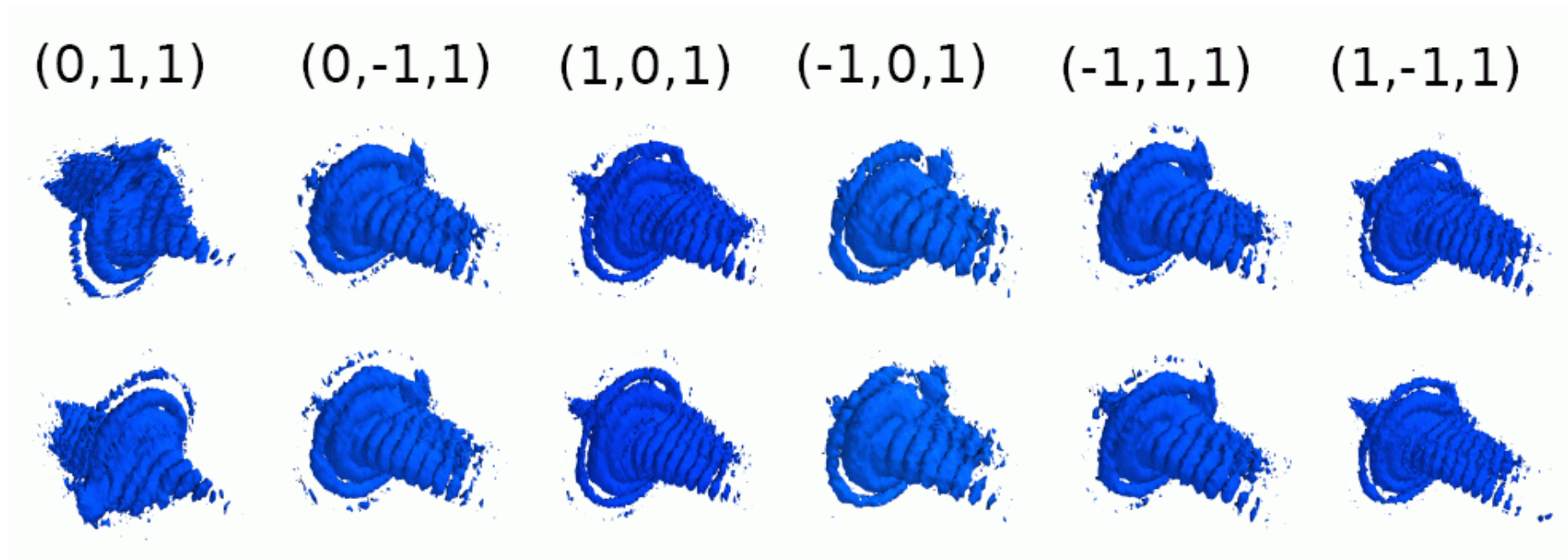
Marcus Newton, UCL, Jan 2009



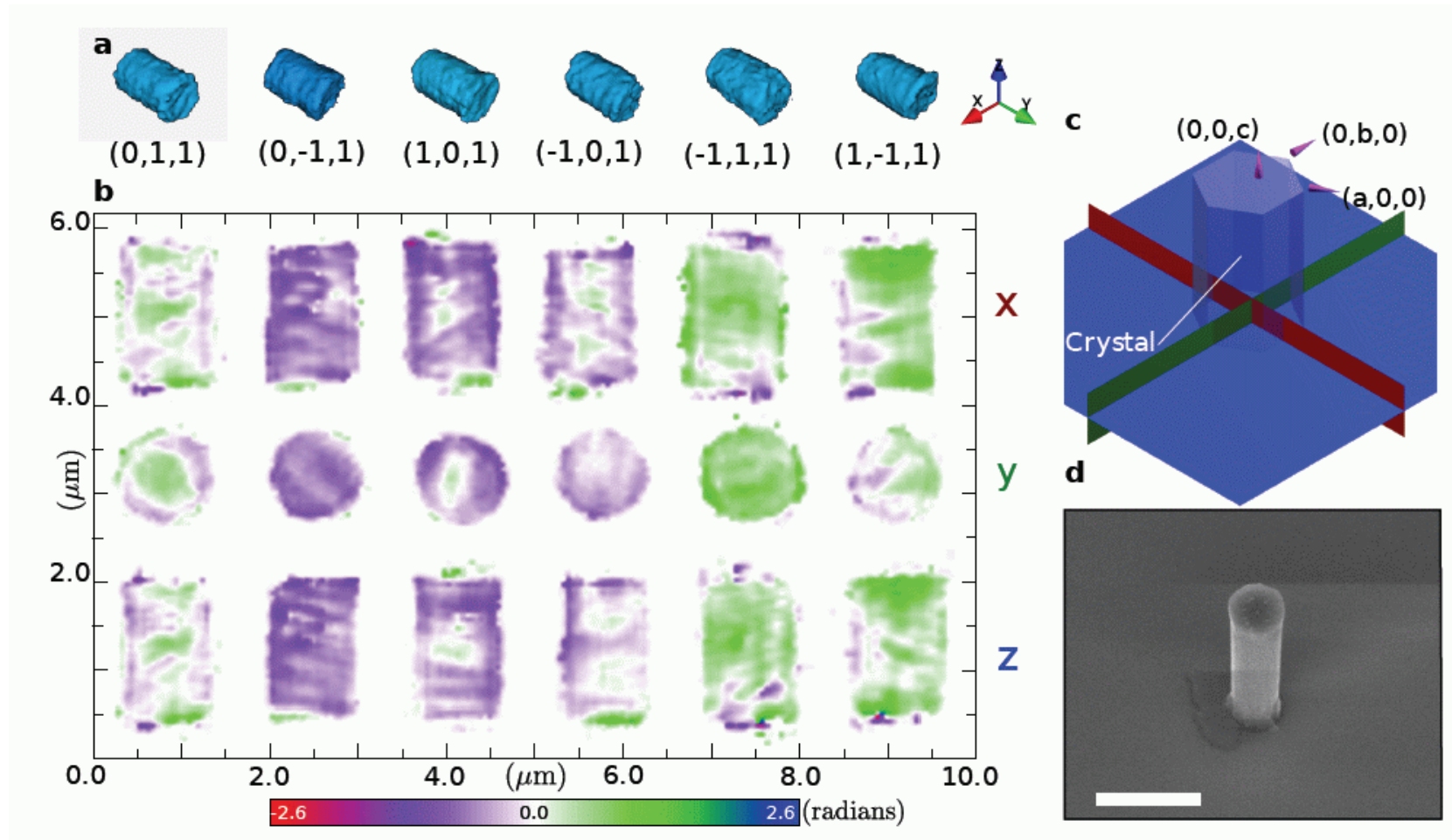
Strain and Rotation fields



Extension to 6 Bragg Peaks

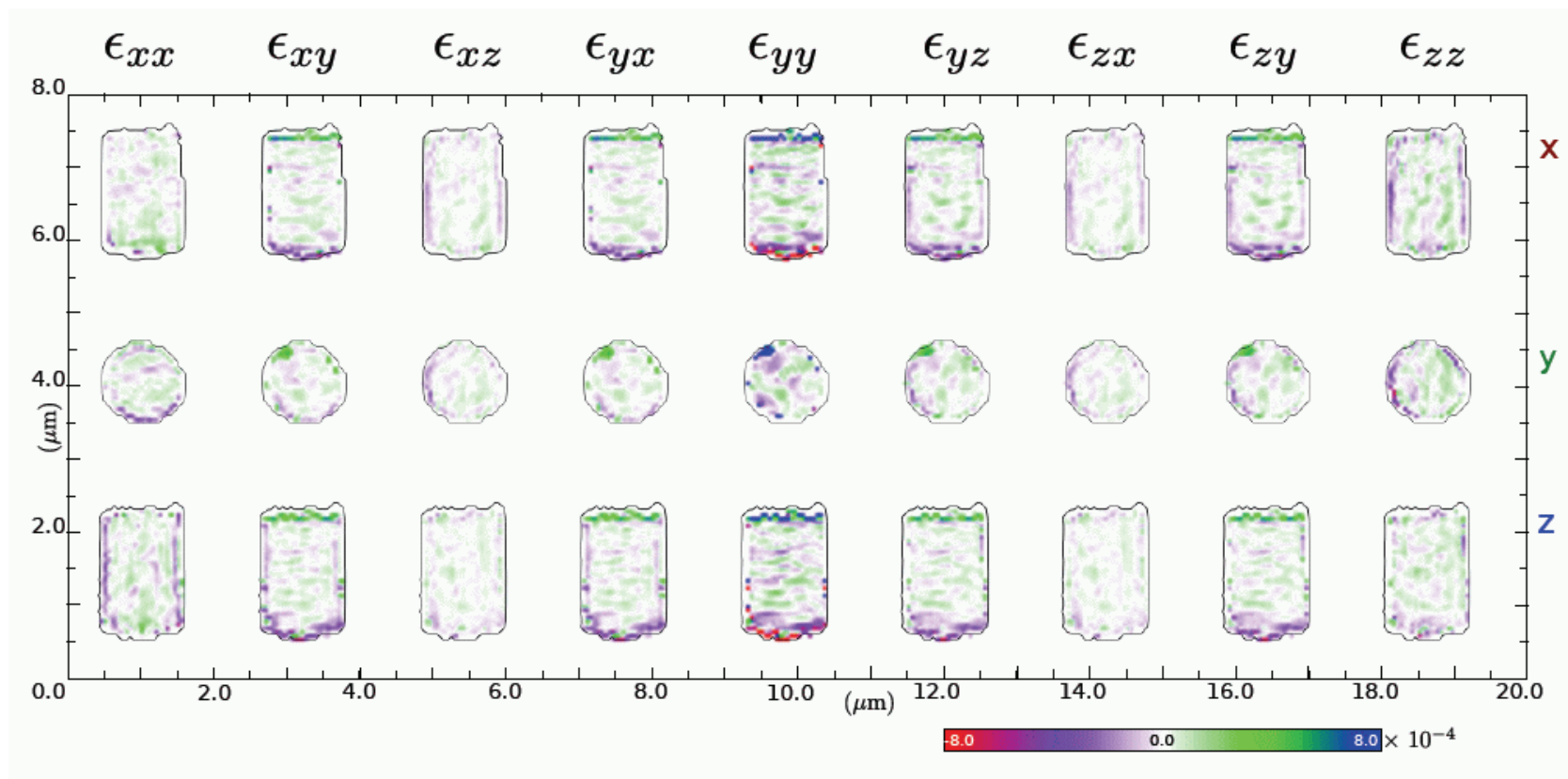


Extension to 6 Bragg Peaks

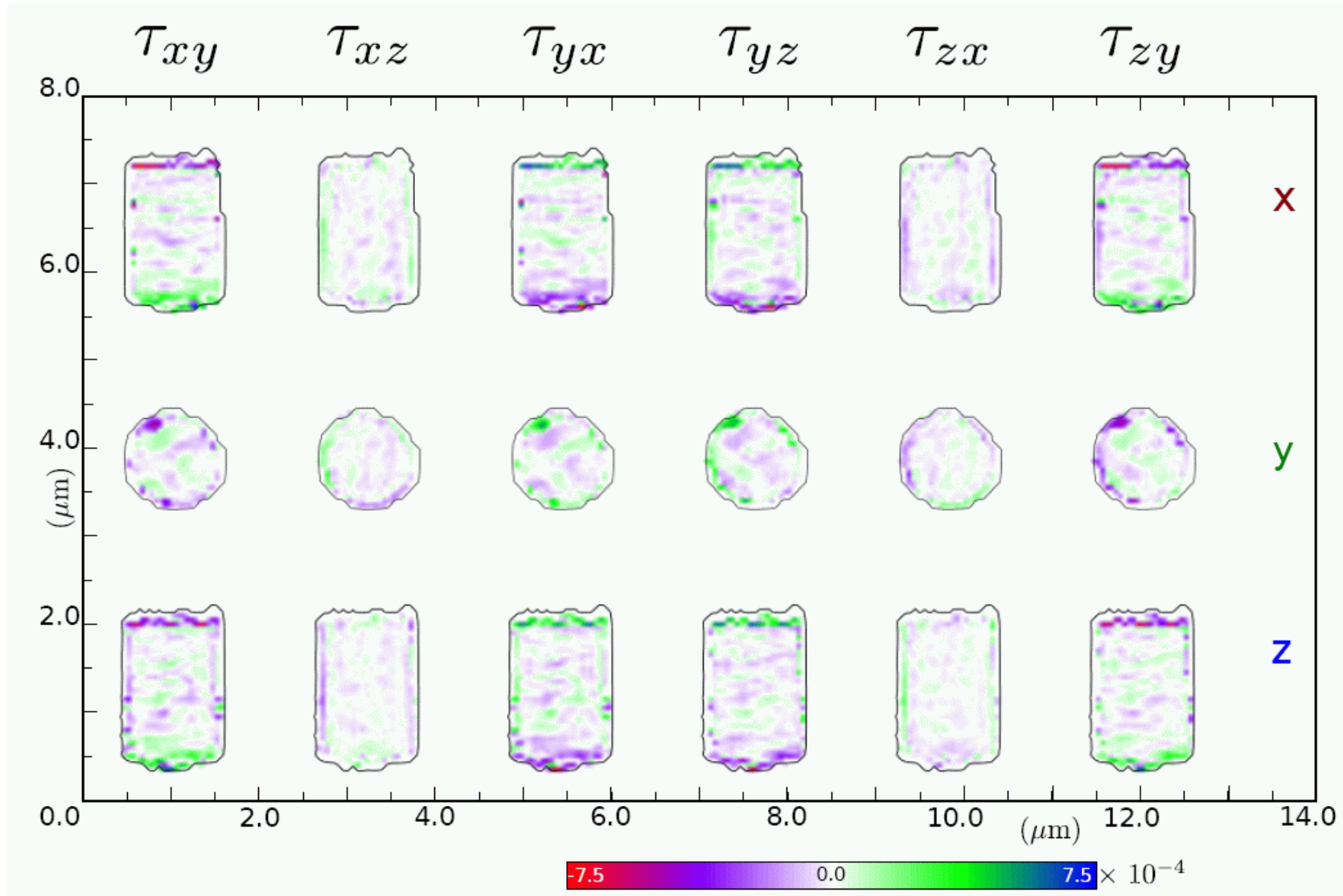


Full Strain Tensor

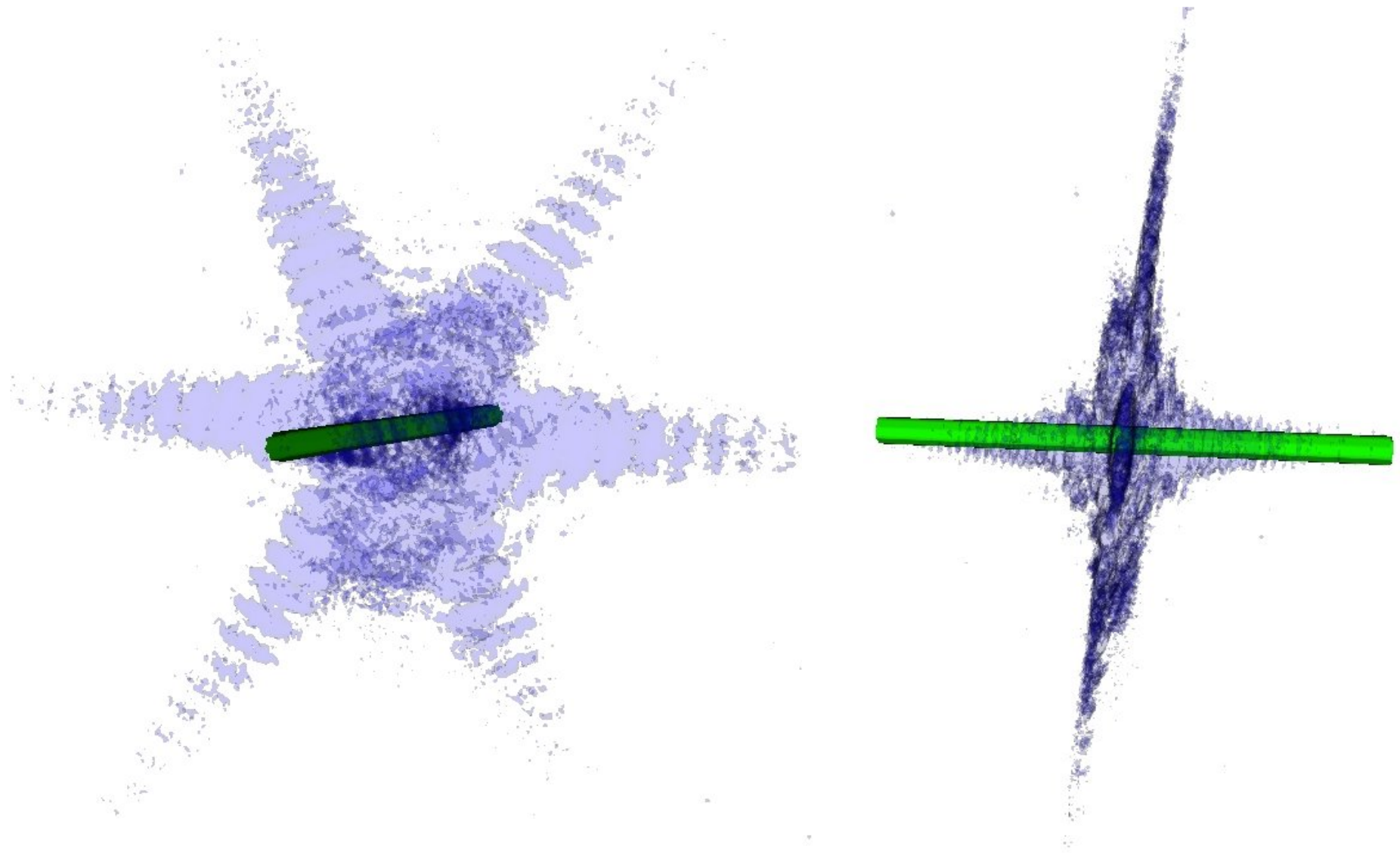
$$\epsilon_{ij} = \frac{1}{2} \left(\frac{\partial u_j}{\partial x_i} + \frac{\partial u_i}{\partial x_j} \right), \quad \tau_{ij} = \left(\frac{\partial u_j}{\partial x_i} - \frac{\partial u_i}{\partial x_j} \right)$$



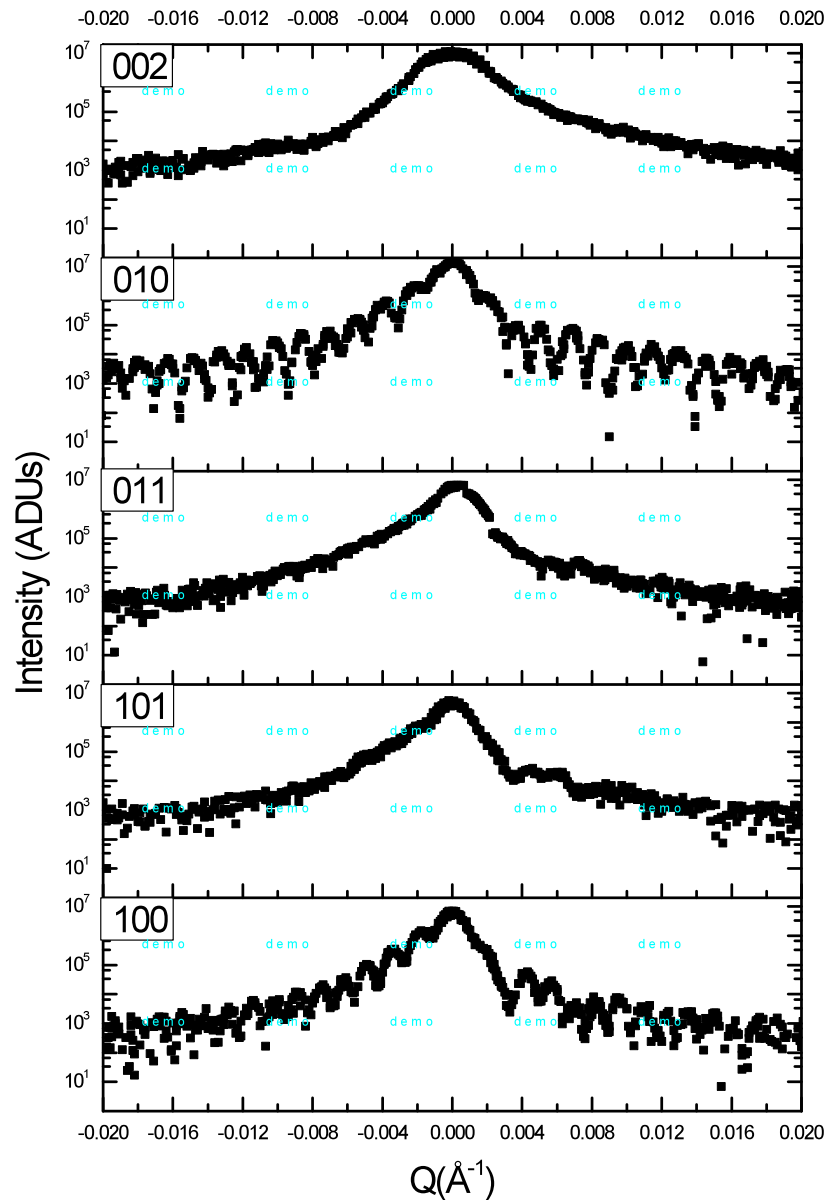
ZnO Rigid-body Rotations



How to extract the contrast data



Five Bragg peaks



002 no fringe visibility

010 & 100 good fringe visibility

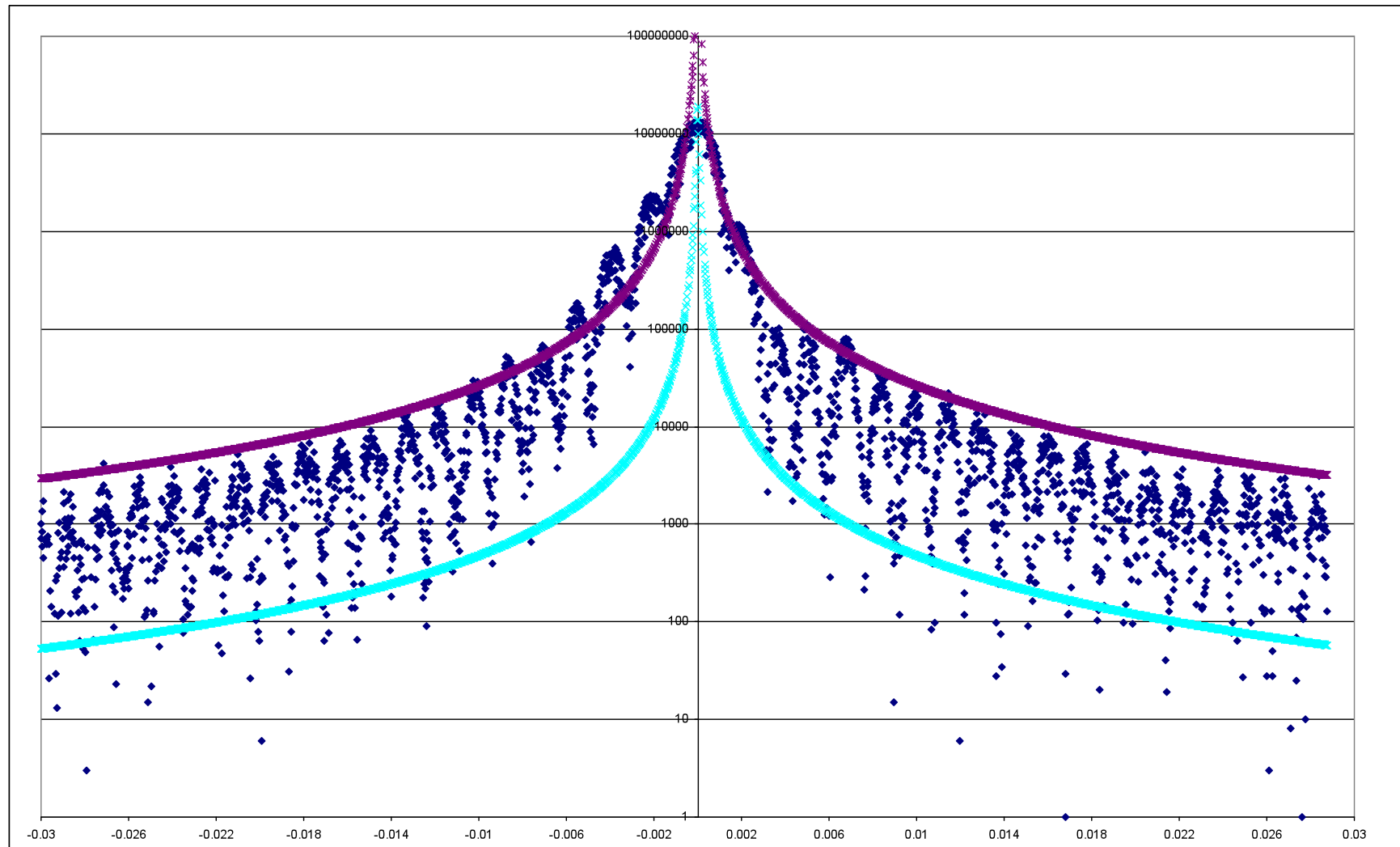
101 & 011 diminished fringe visibility
but fringes still evident

010:011 & 101:100
show complementary fringes
but not between each other

Error in coordinate transform ruled out

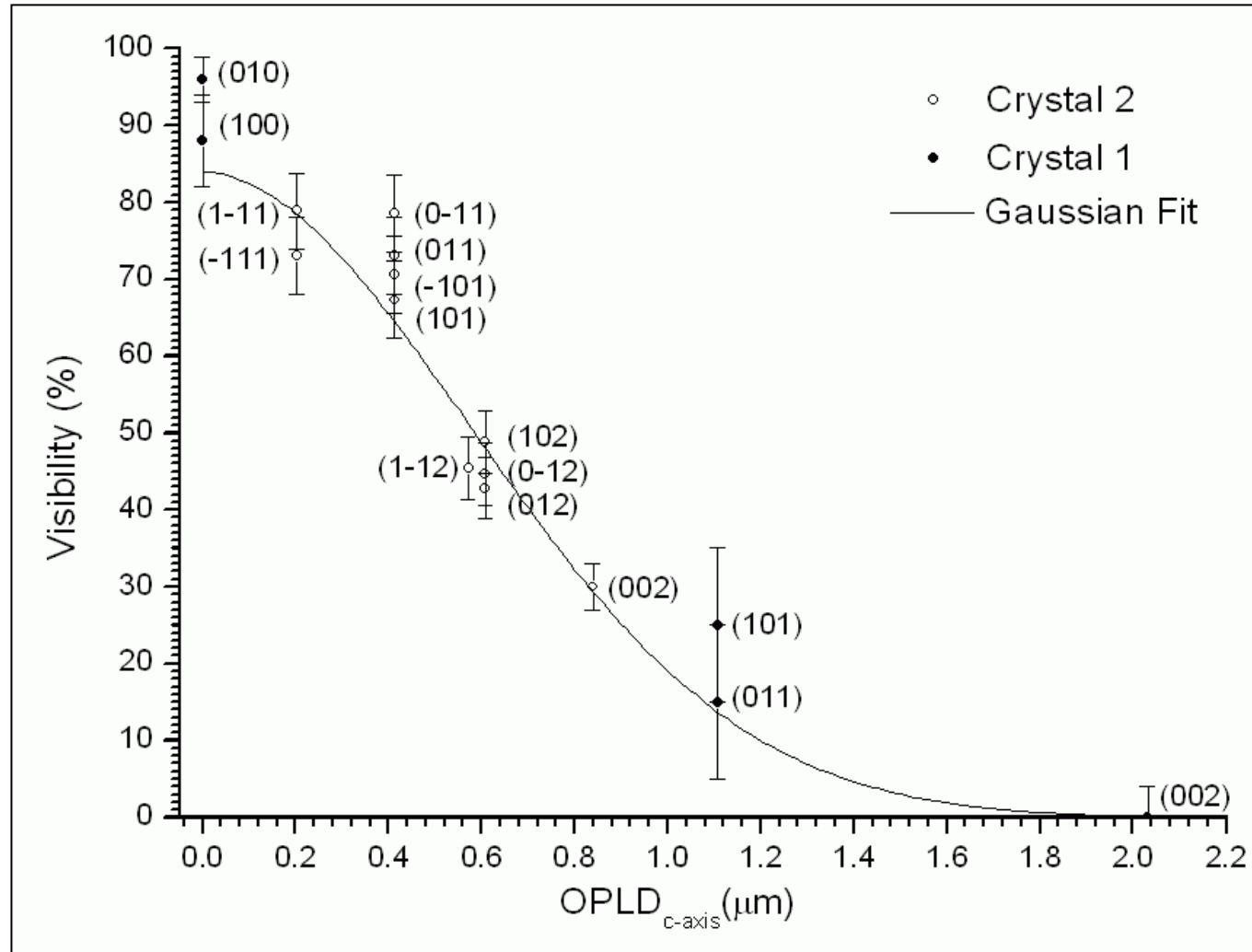
010 and 100 reflections fringe spacing
difference $\sim 20\%$

Fringe Visibility $96\pm 2\%$ @010



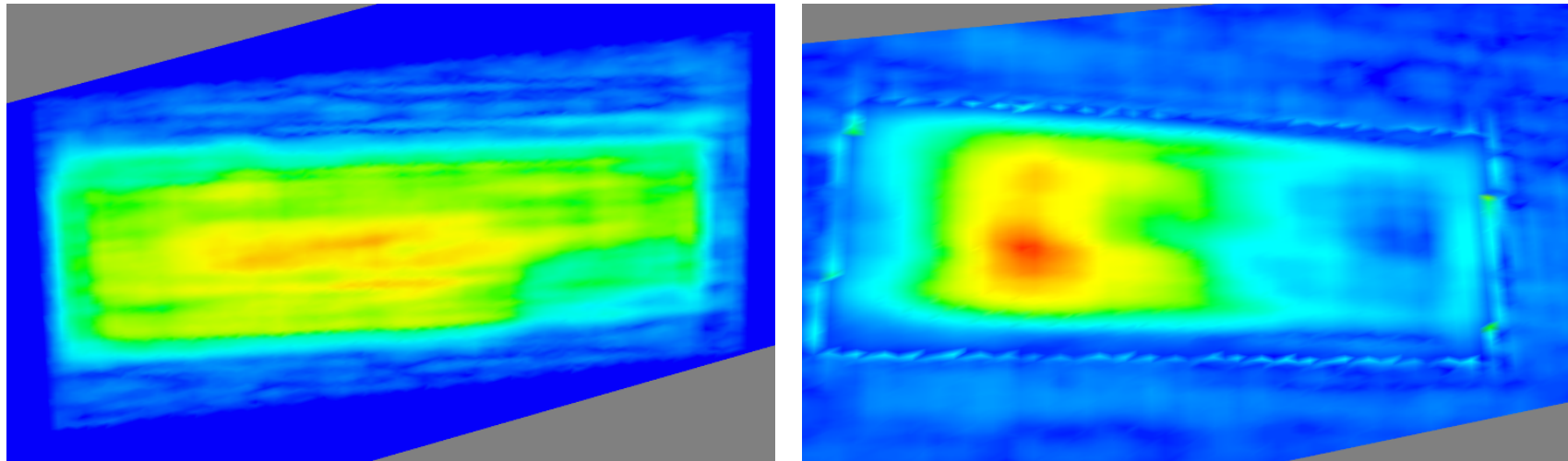
Measured Longitudinal Coherence

S. Leake, M. Newton, R. Harder and I. Robinson,
Optics Express 17 15853 (2009)

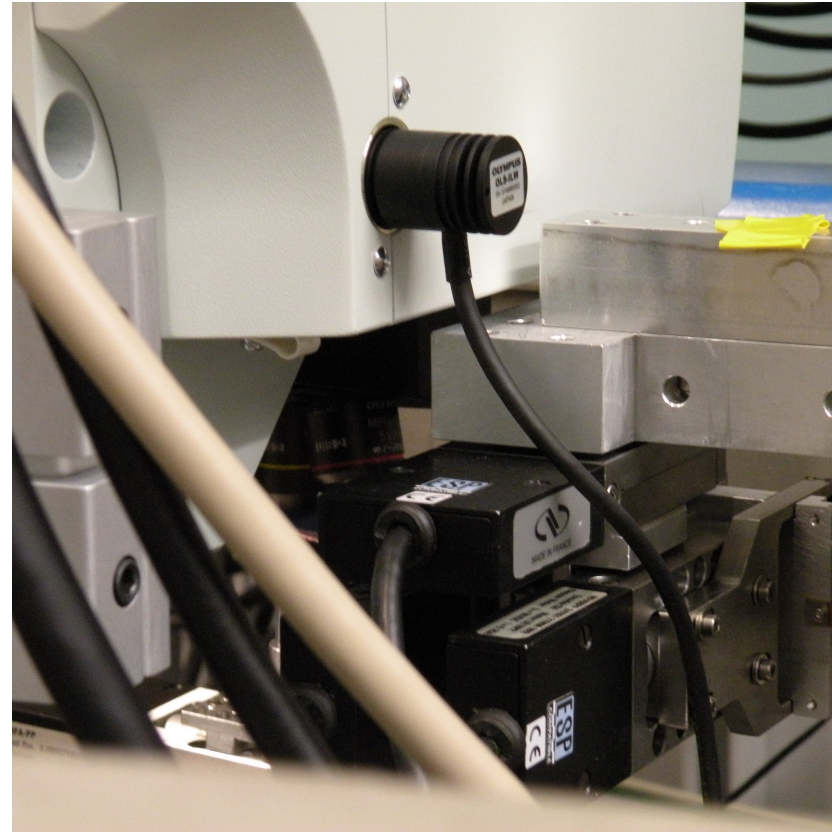
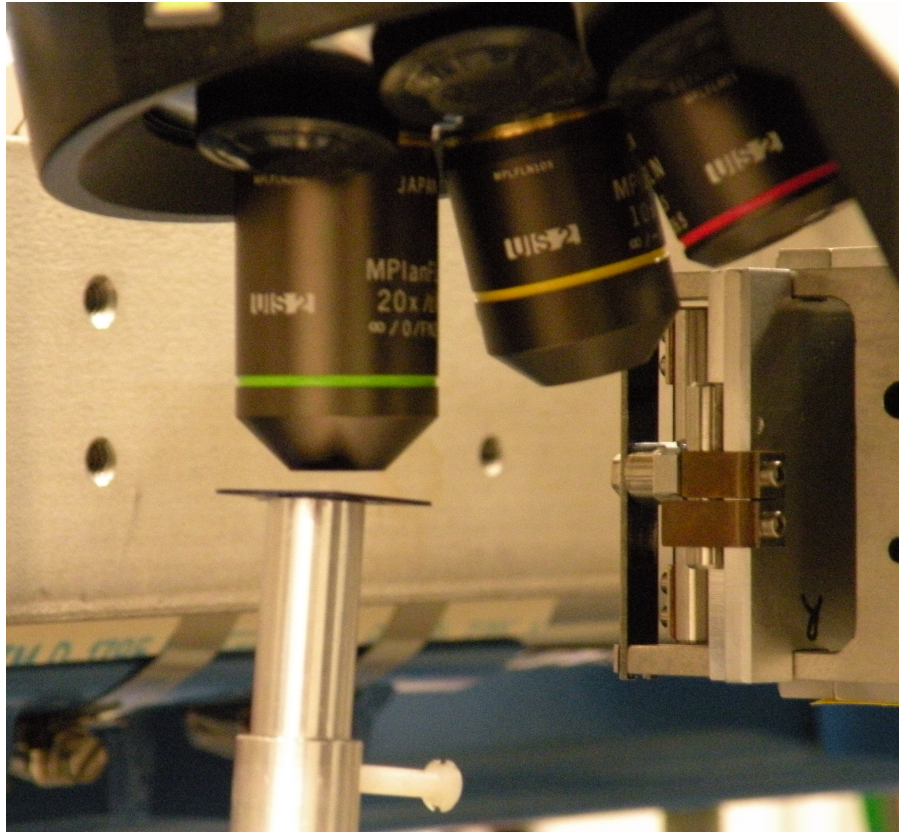


Density Section of Reconstruction using 100 and 101 reflections

“hot spot” more developed for limited coherence
S. Leake, M. Newton, R. Harder and I. Robinson,
Optics Express 17 15853 (2009)

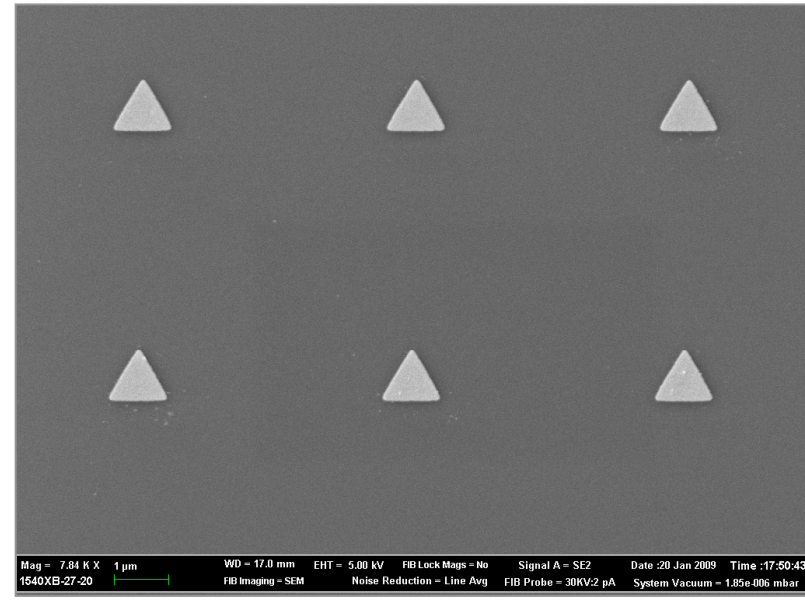
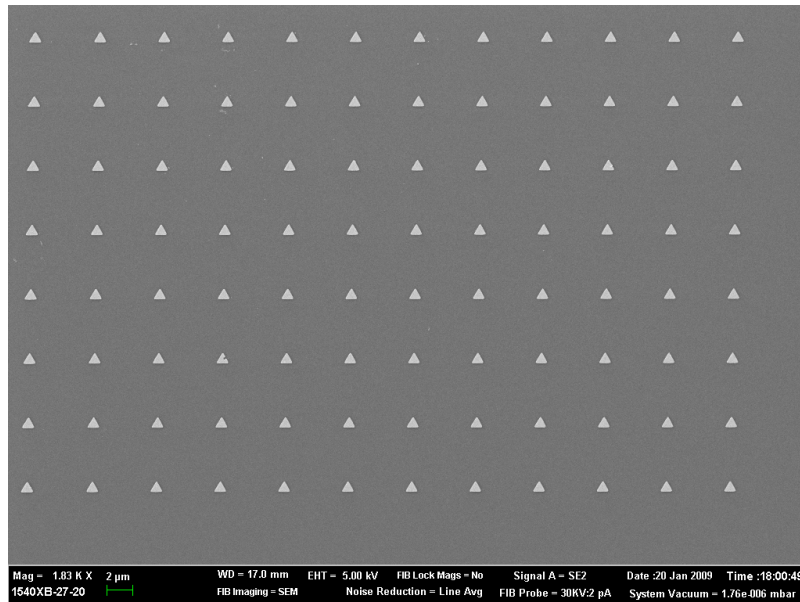


Confocal Alignment Microscope

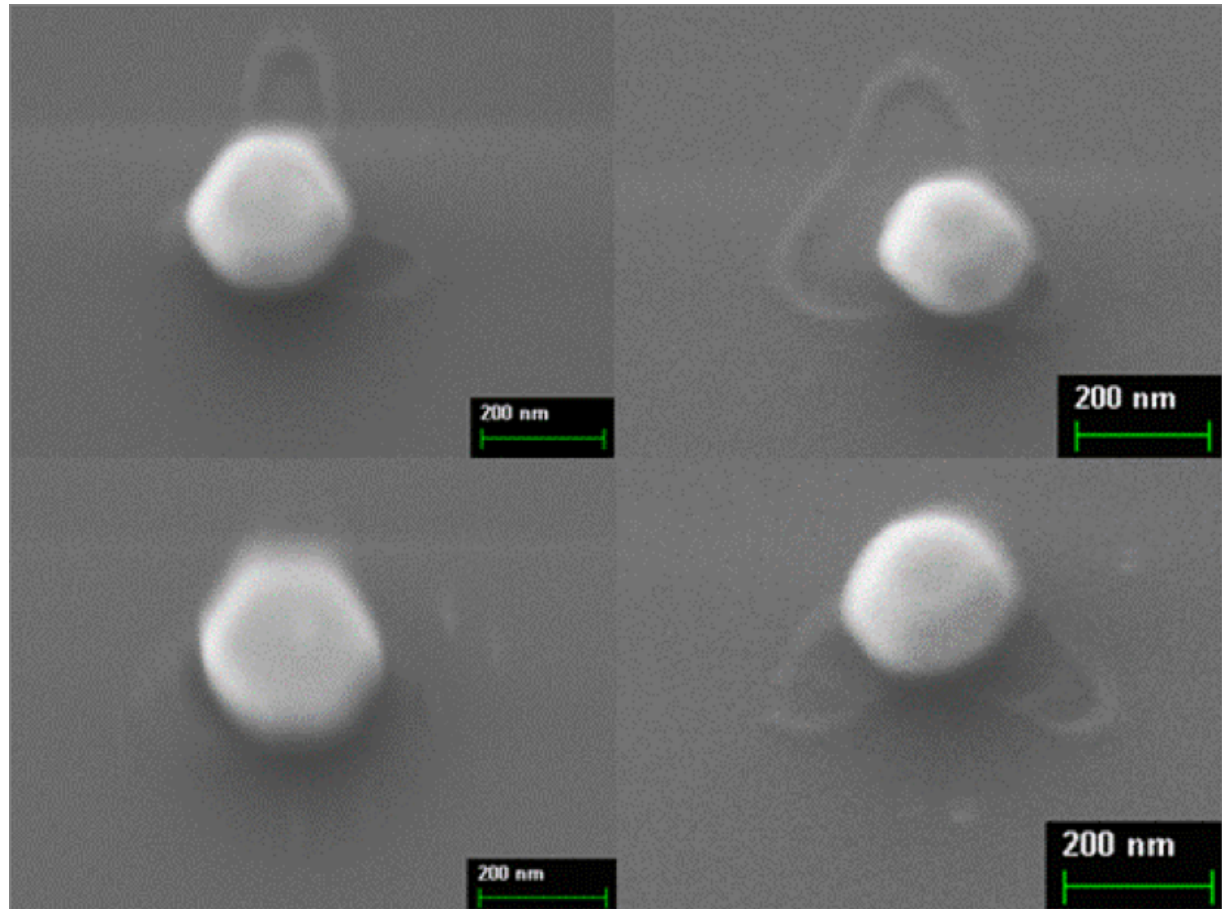
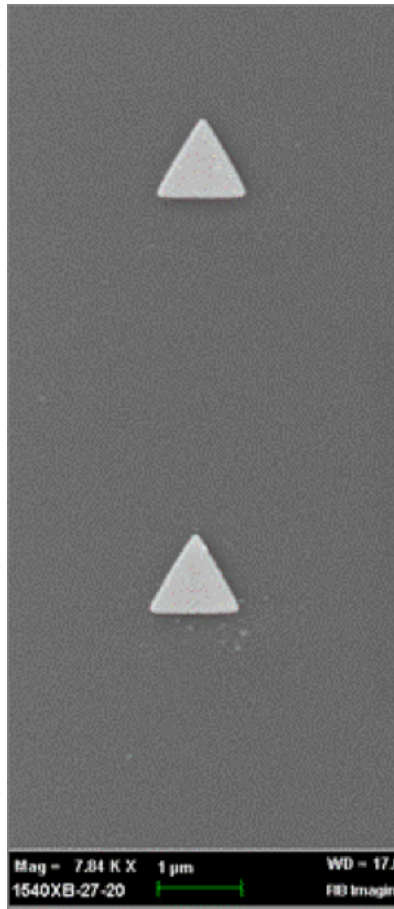


Patterned Au nanocrystal samples

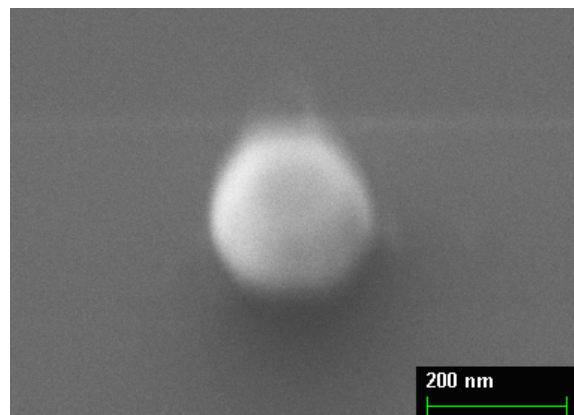
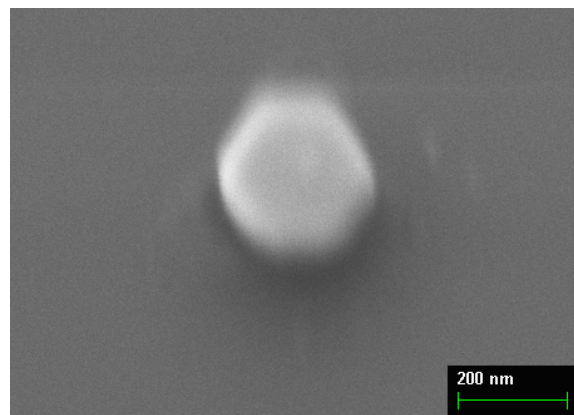
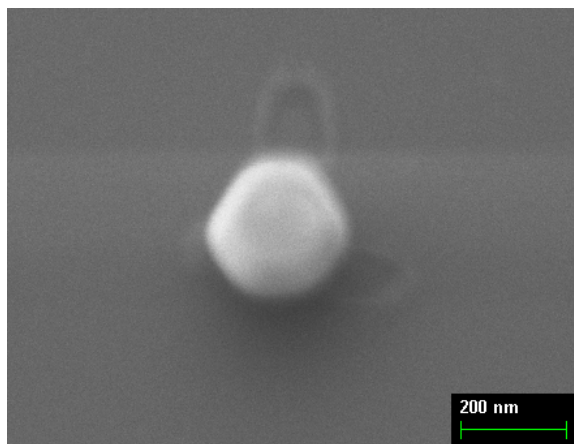
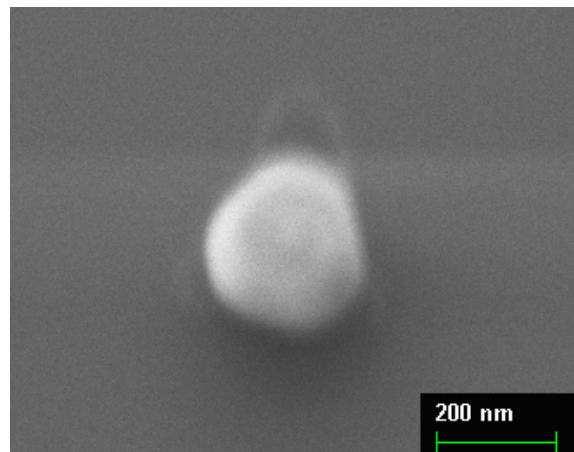
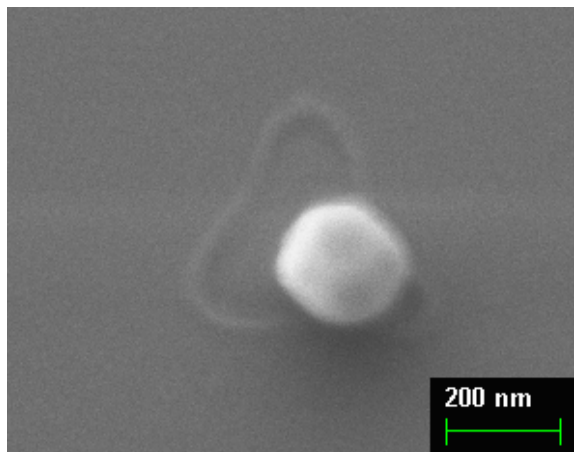
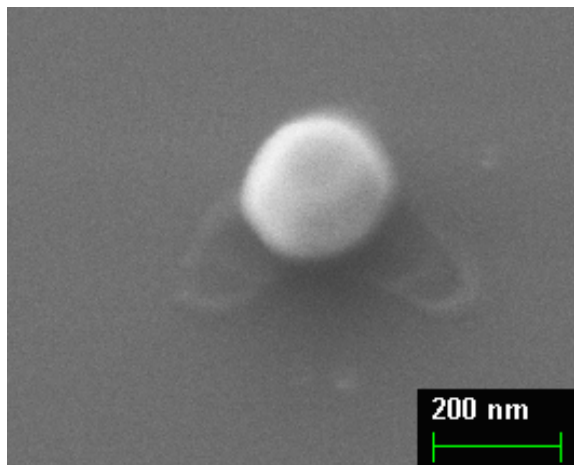
N. Shimamoto, Waseda University, Japan

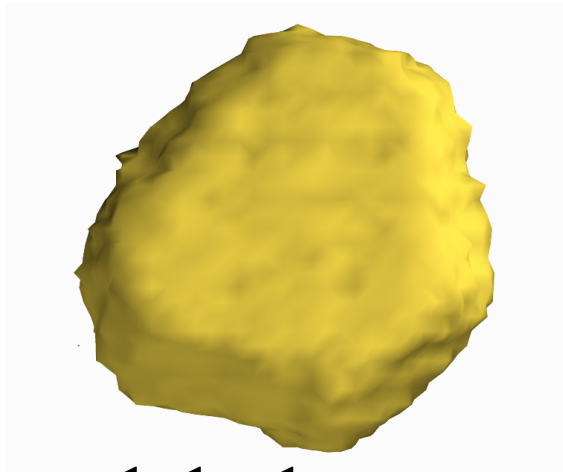


Single Au nanocrystal synthesis

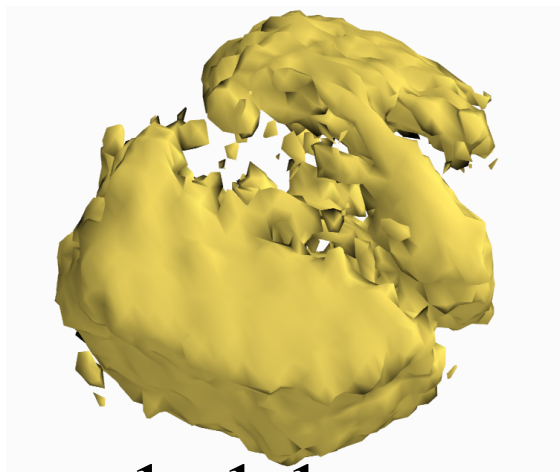


SEM images of Crystals after 950C anneal for 8hrs

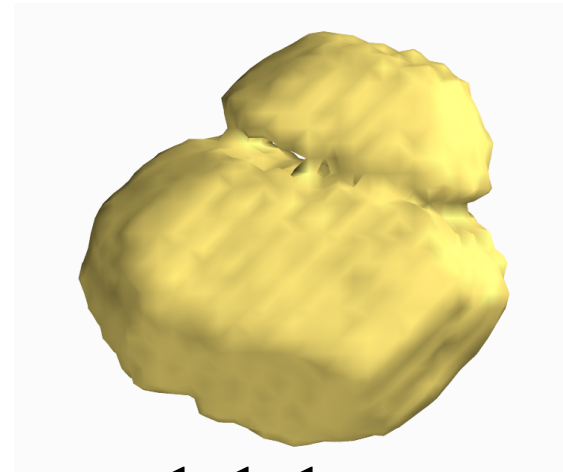




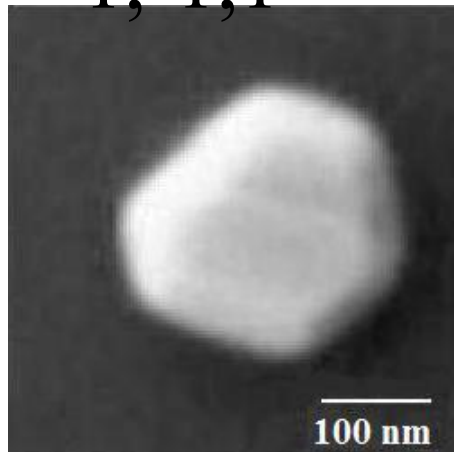
1,1,-1



1,-1,1

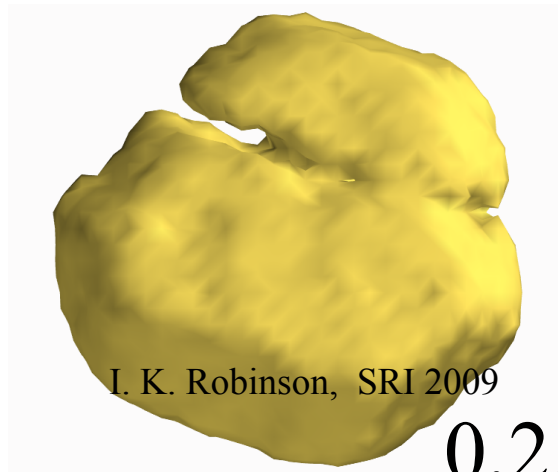
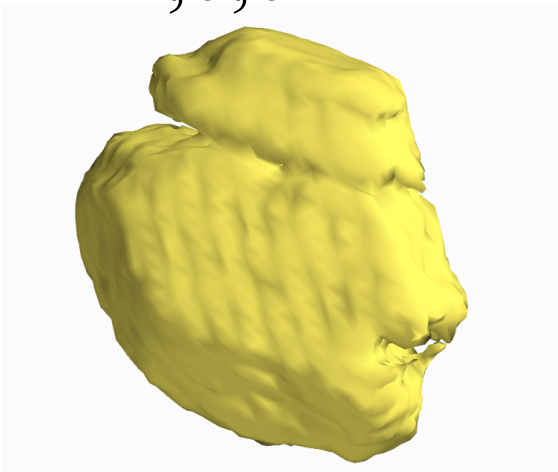


-1,1,1

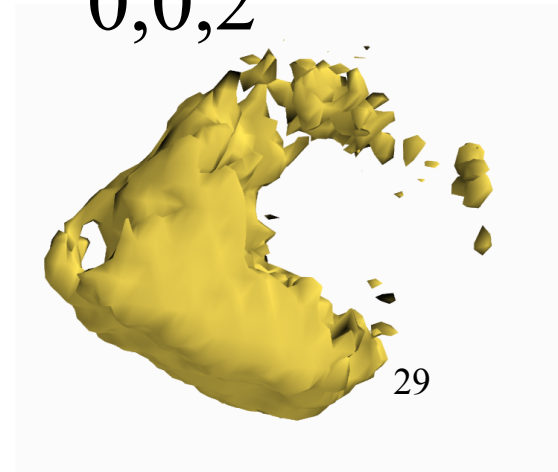


100 nm

2,0,0



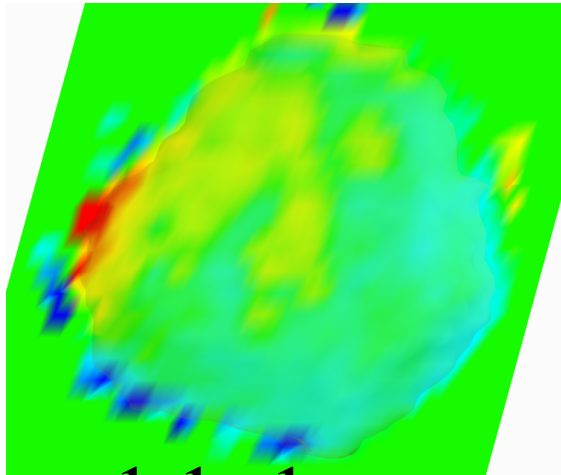
0,2,0



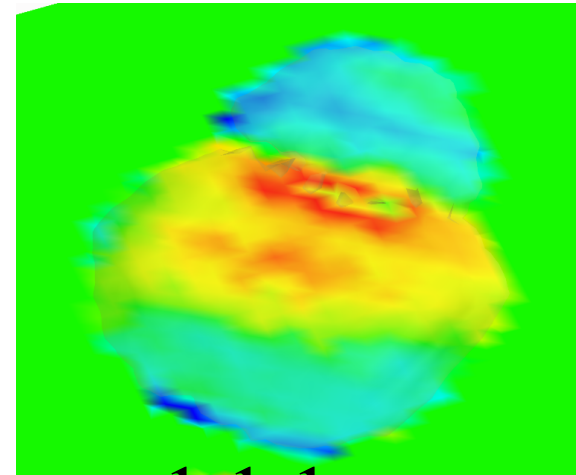
0,0,2

I. K. Robinson, SRI 2009

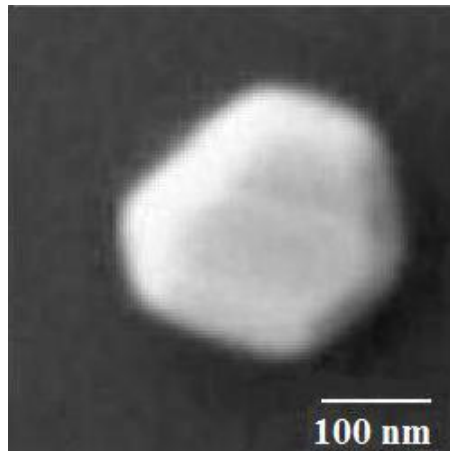
29



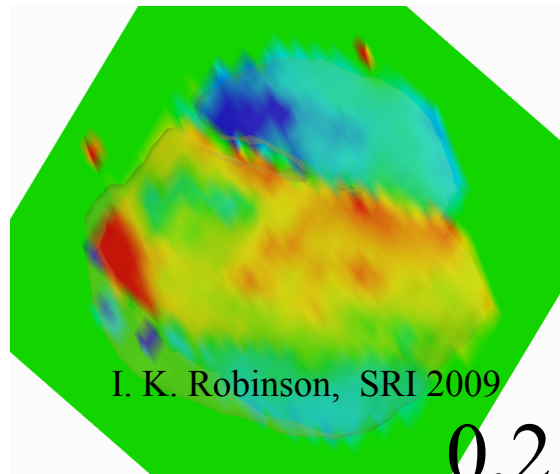
1,1,-1



-1,1,1



100 nm

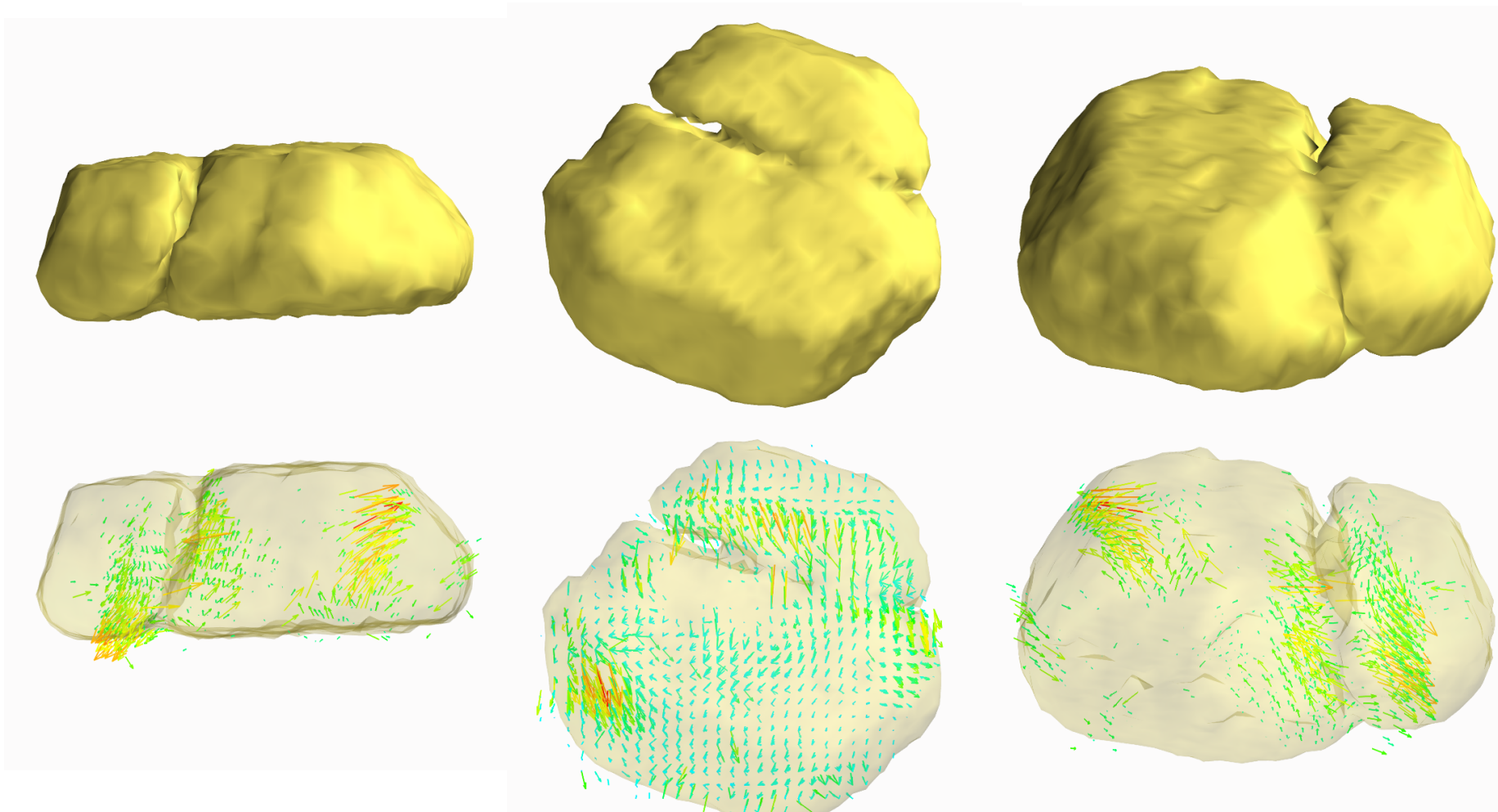


0,2,0

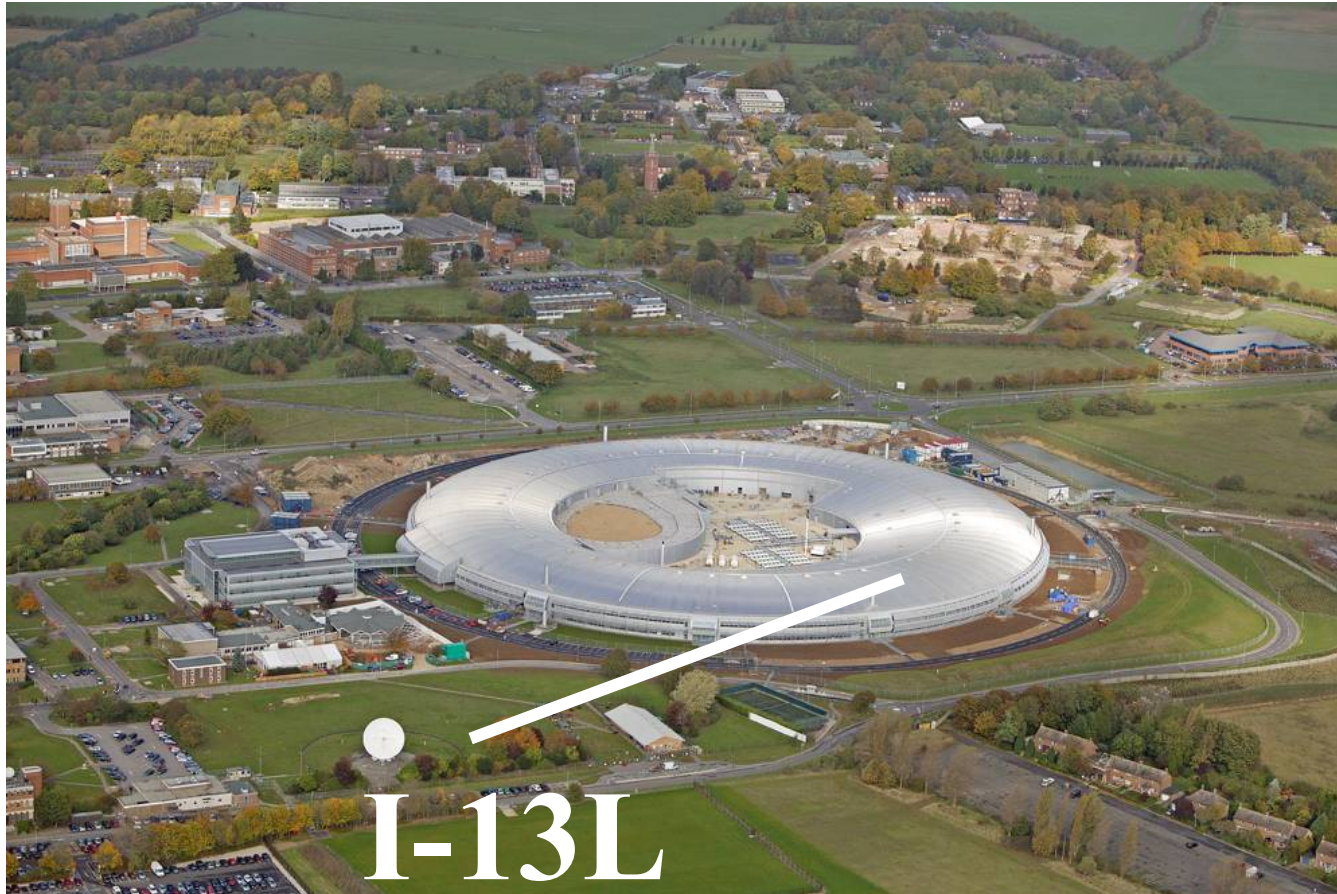
I. K. Robinson, SRI 2009

Vector displacement field

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



Diamond Light Source (RAL)



I-13L

I. K. Robinson, SRI 2009

Conclusions

- Internal structure of Au and ZnO Nanocrystals
- Phasing by computation instead of lens
- Strain fields imaged from asymmetric patterns
- Coherence mapped with multiple Bragg peaks
- Full strain tensor requires multiple Bragg peaks
- Confocal microscope solves sphere of confusion