

Coherent X-ray Diffraction Imaging from nanocrystals

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Mark Pfeifer

Ivan Vartanians

London Centre for Nanotechnology

Diamond Light Source

2nd school and workshop on x-ray
micro and nanoprobes

Palinuro, June 2009

Outline

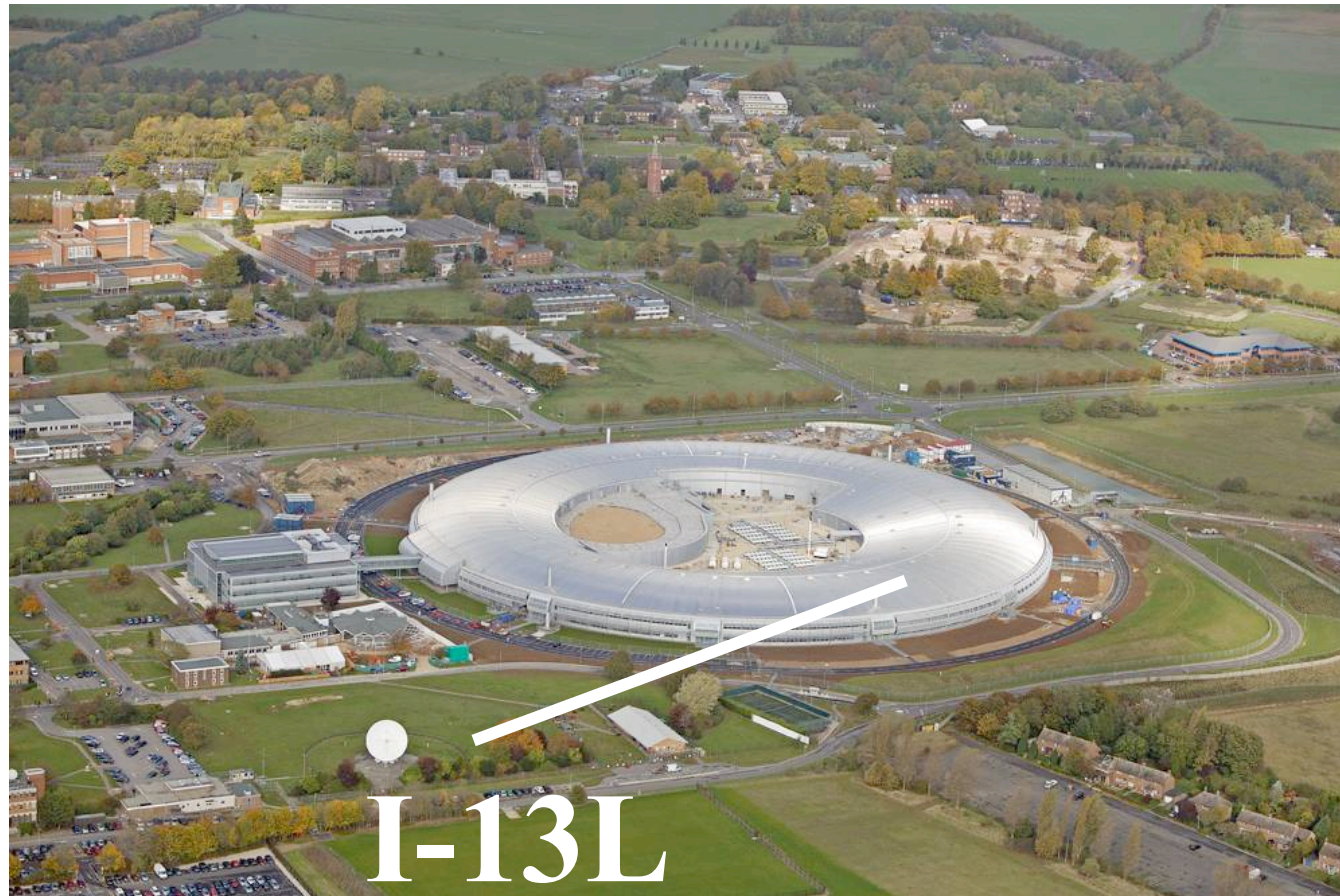
- Coherent x-ray diffraction
- CXD can solve the **phase** problem
- Nanocrystal structures
- Extension to **phase** objects
- Exploration of crystal strain
- Nanowire and newer structures

London Centre for Nanotechnology

- Clean Rooms
- Low-T STM
- Lithography
- 3-beam FIB
- Visualisation
- CLS?

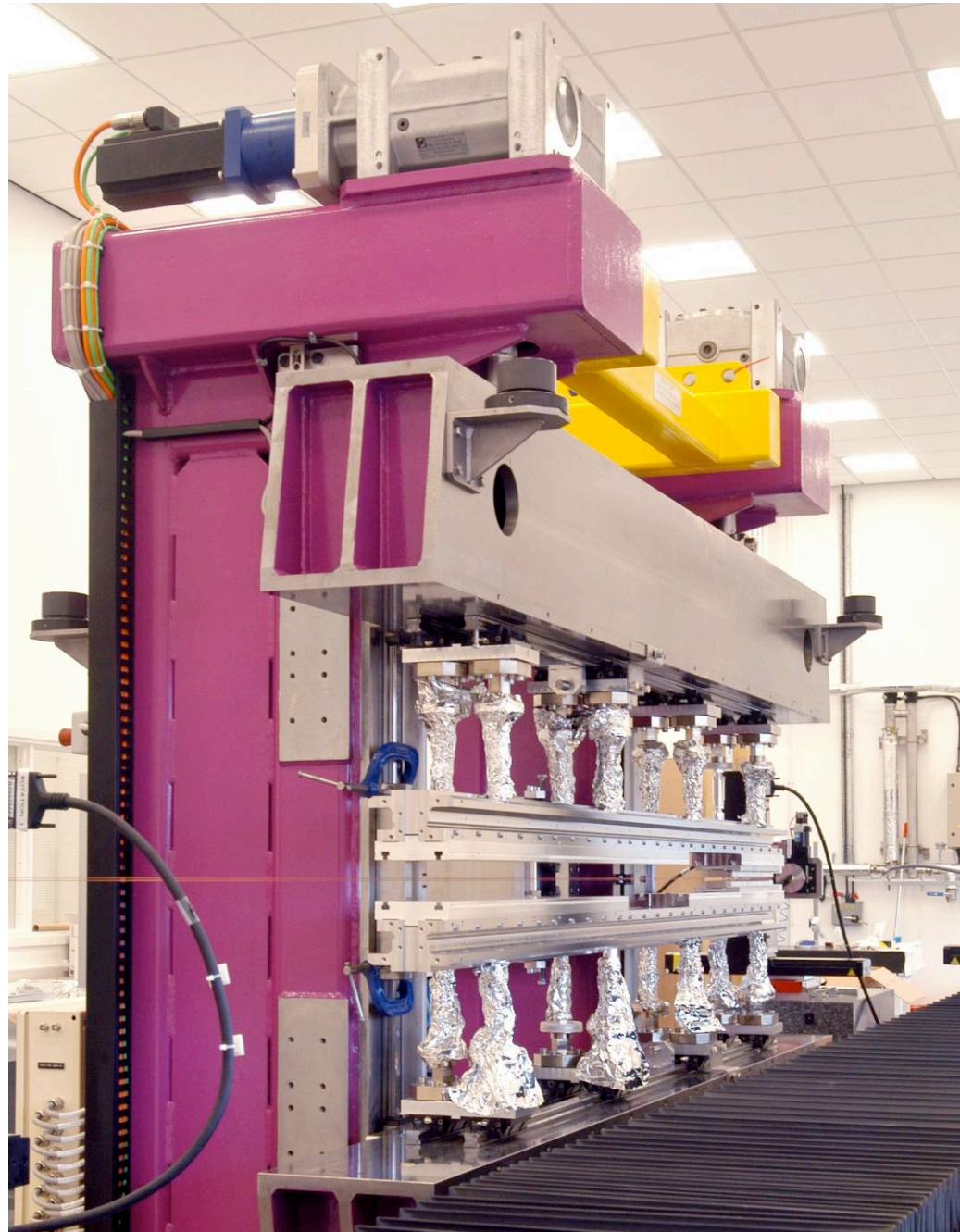


Diamond Light Source (RAL)

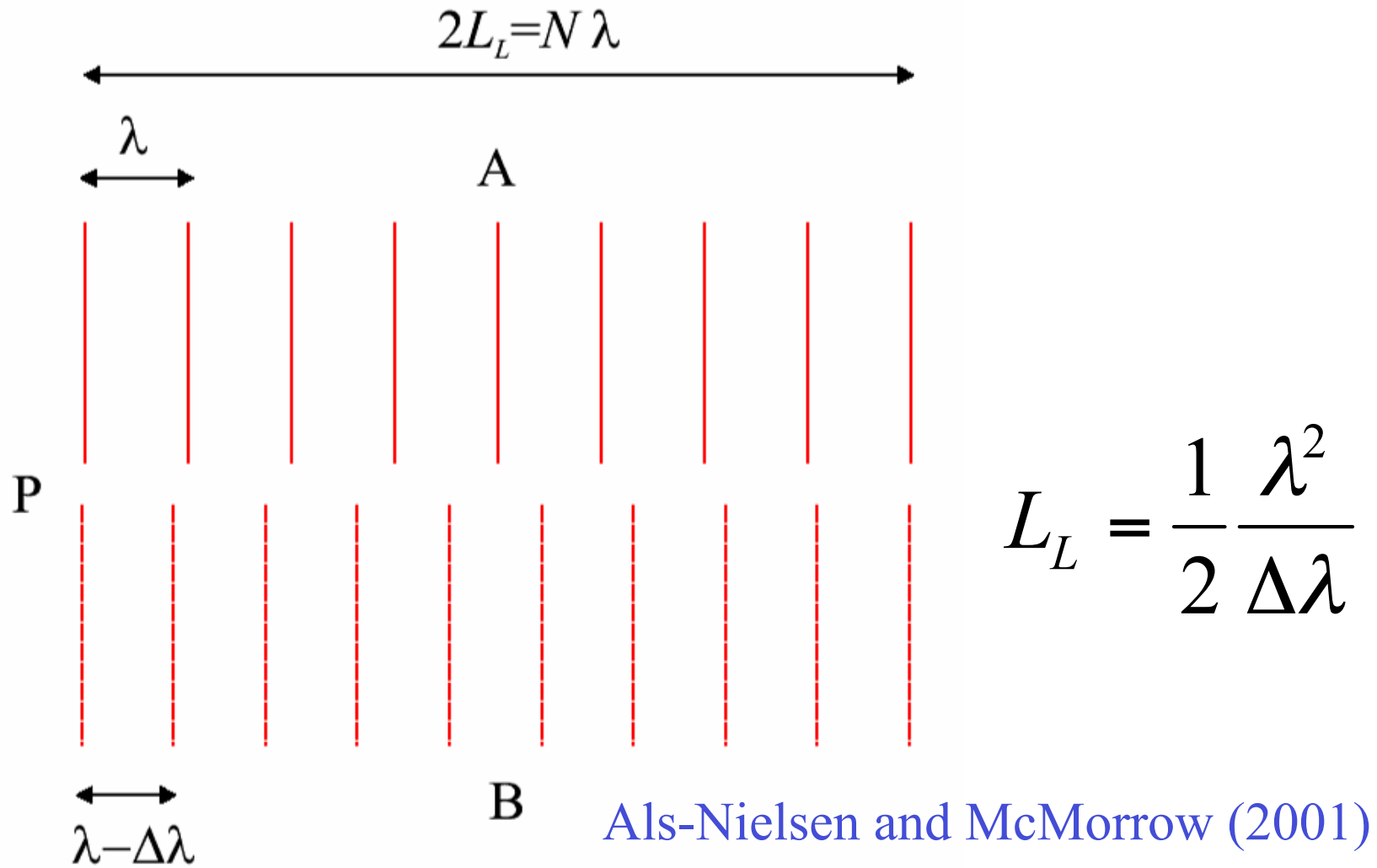


I-13L

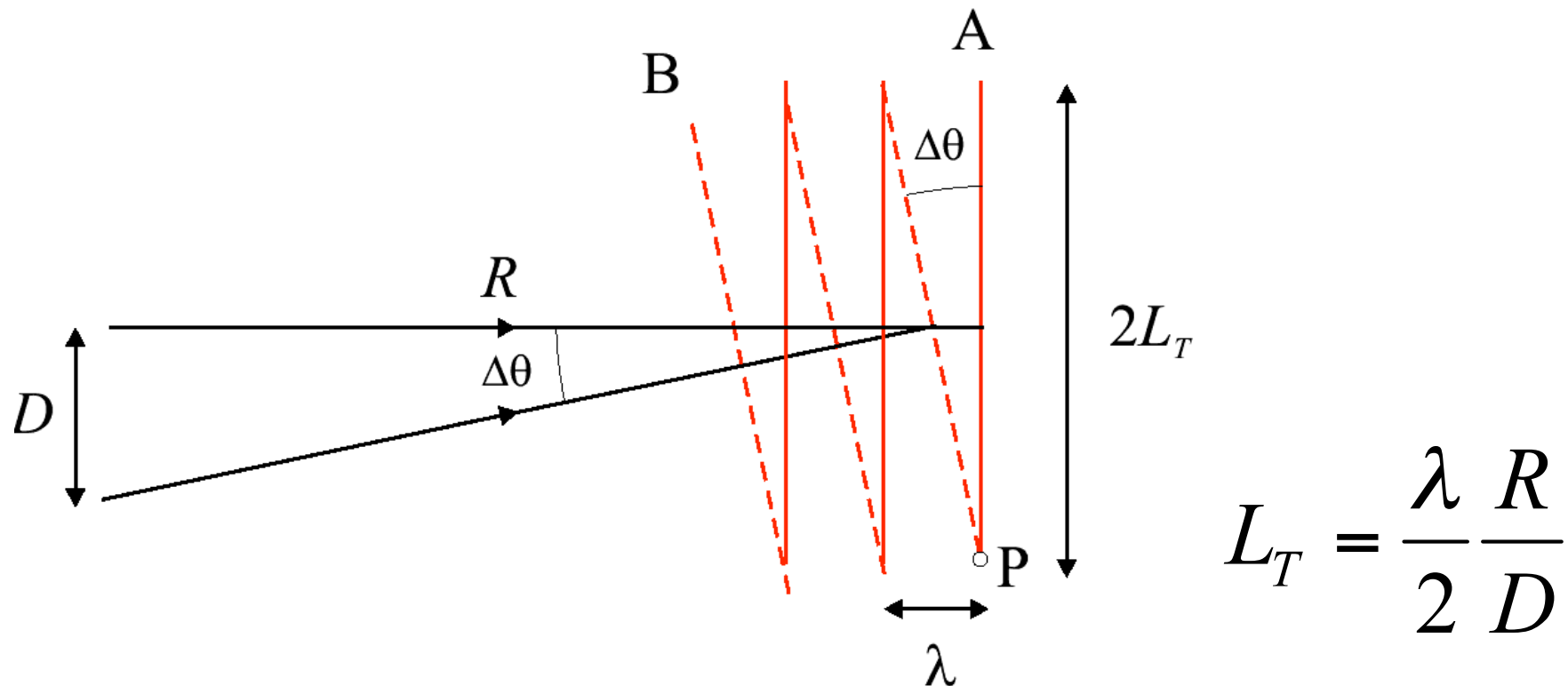
Diamond
in-vacuum
X-ray
Undulator



Longitudinal Coherence



Lateral (Transverse) Coherence

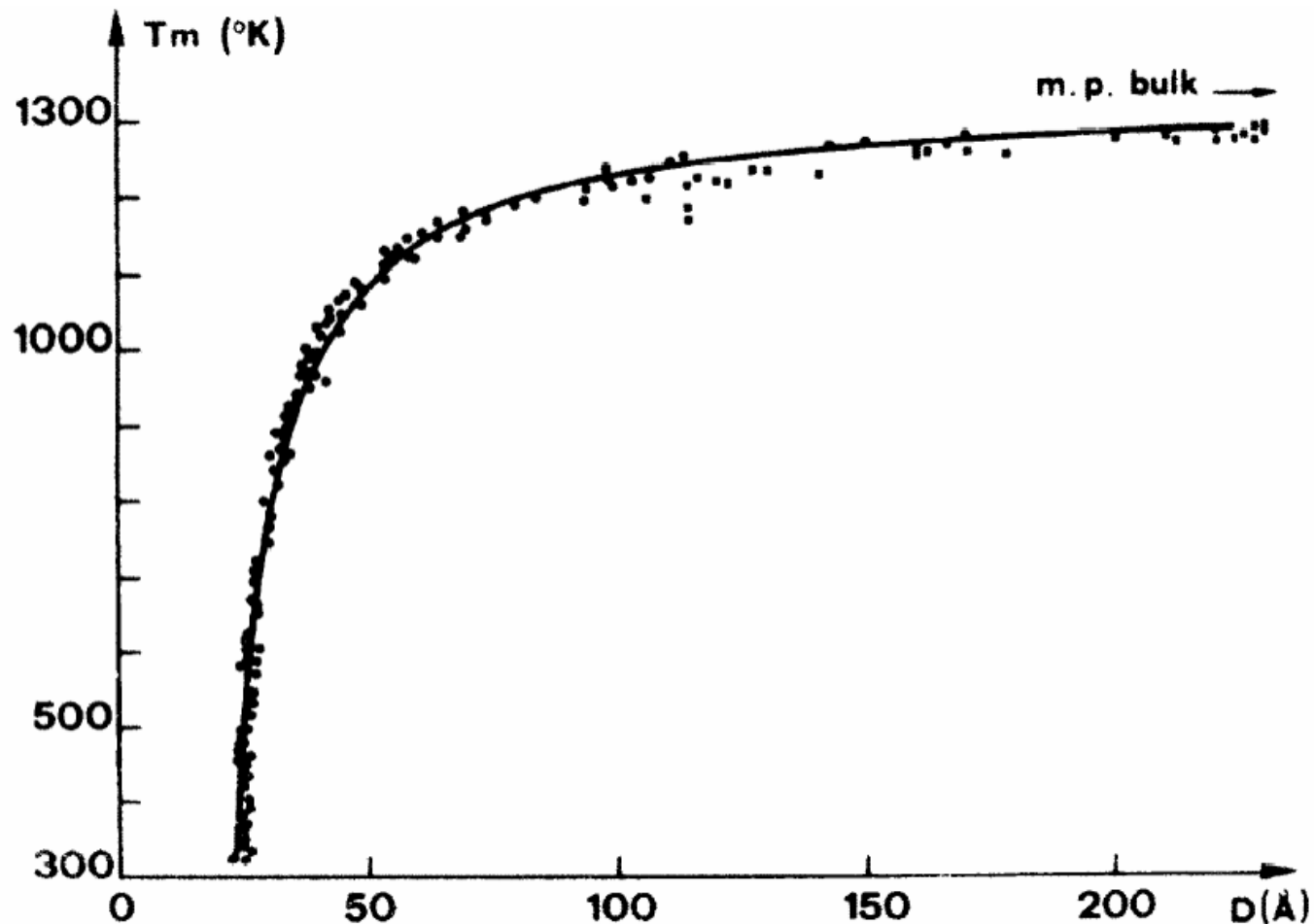


$$L_T = \frac{\lambda R}{2 D}$$

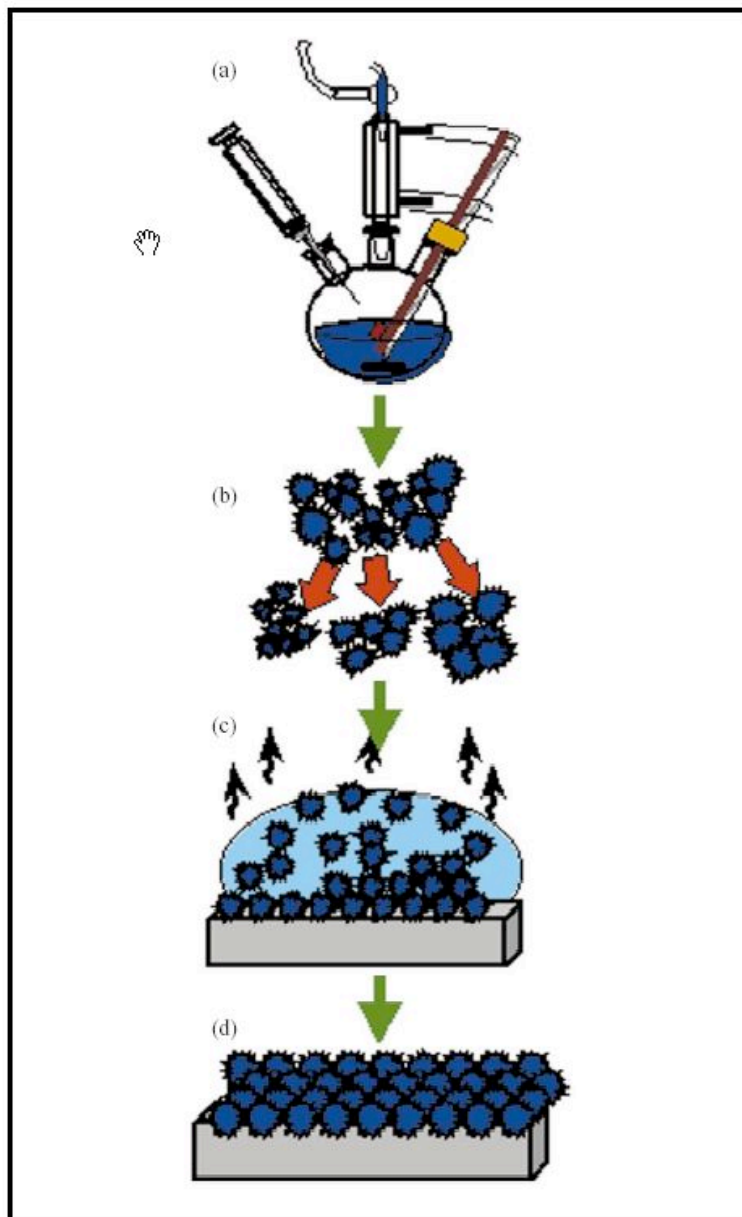
Als-Nielsen and McMorrow (2001)

Size-dependent Melting of Au Particles

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

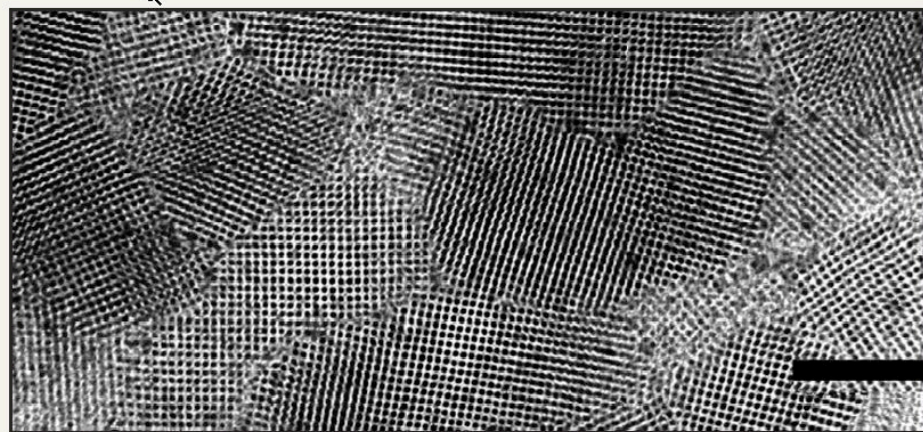


Chemical Synthesis of Nanocrystals



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

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



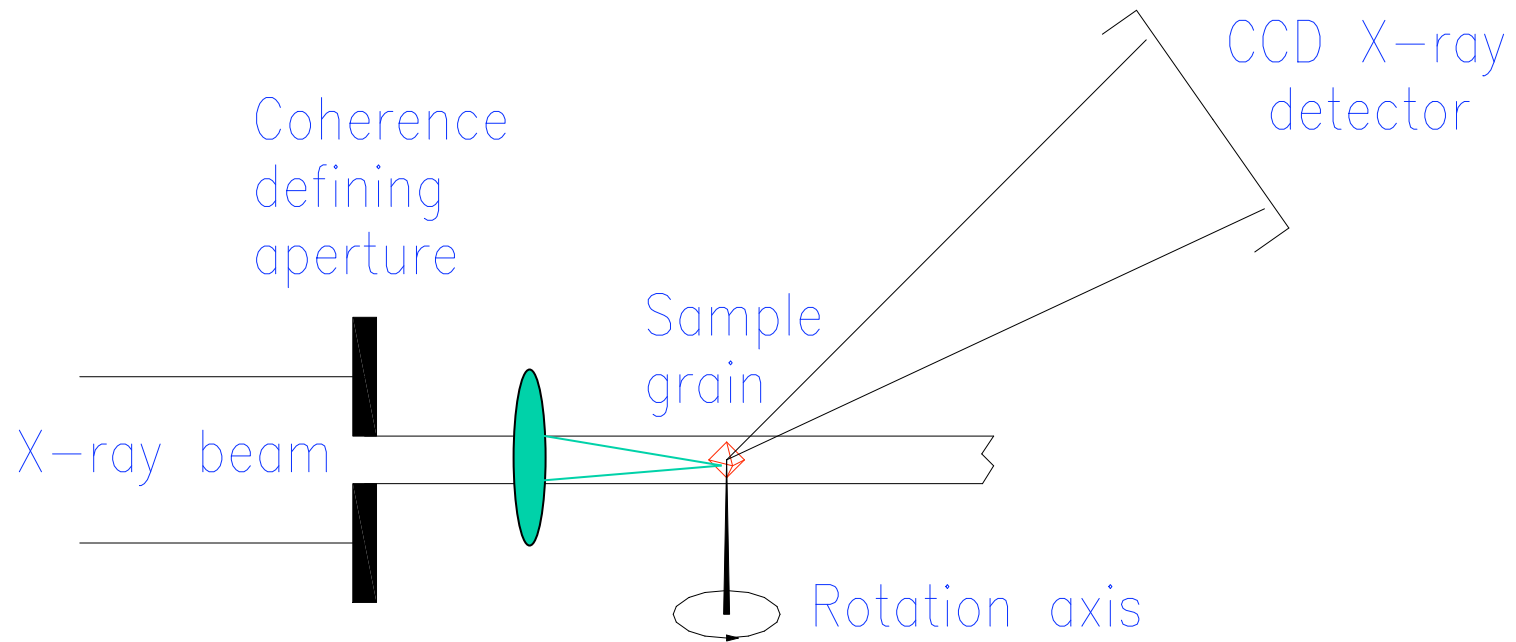
Robert Hooke's microscope, 1665

National Museum of Health
and Medicine, Washington
DC

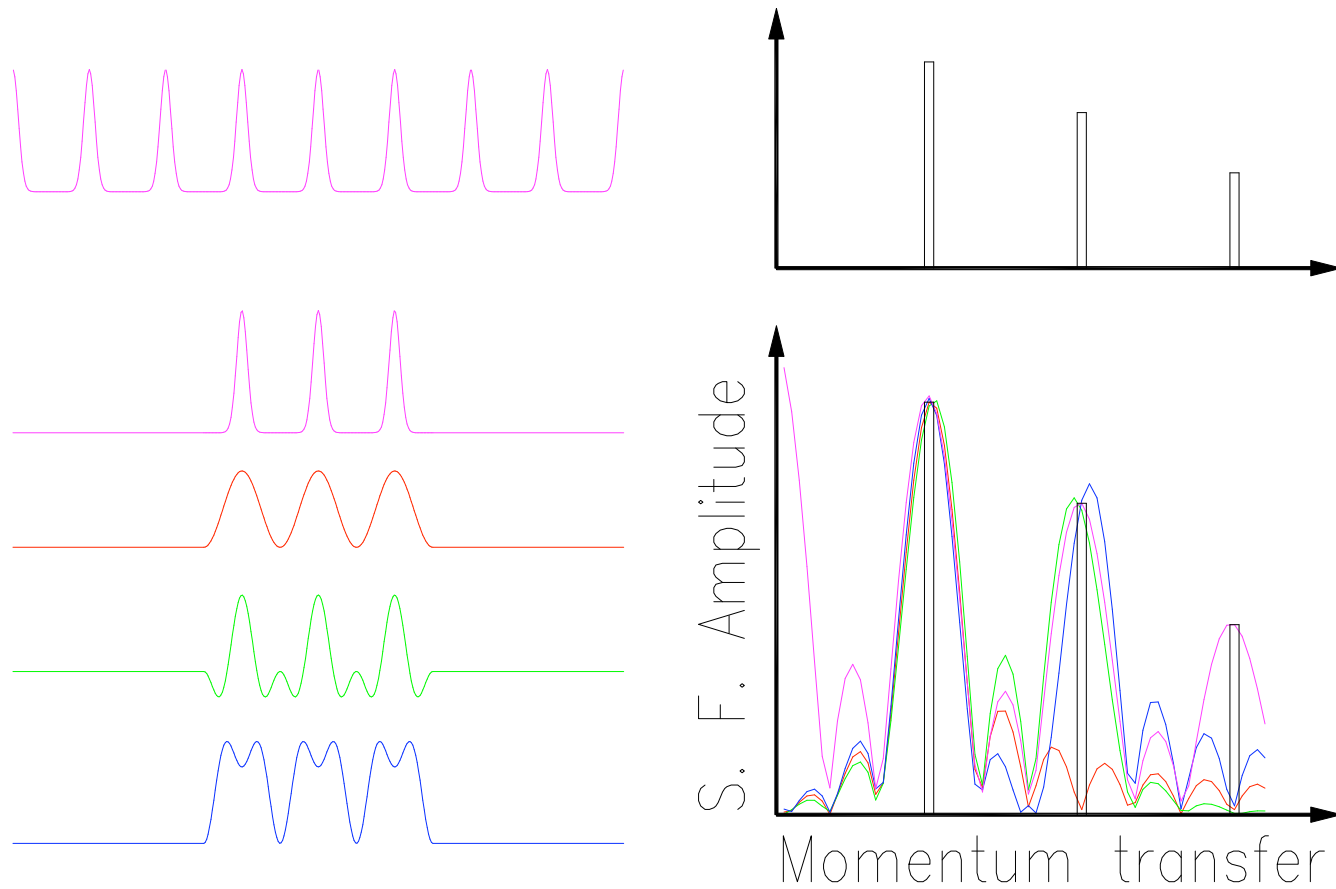




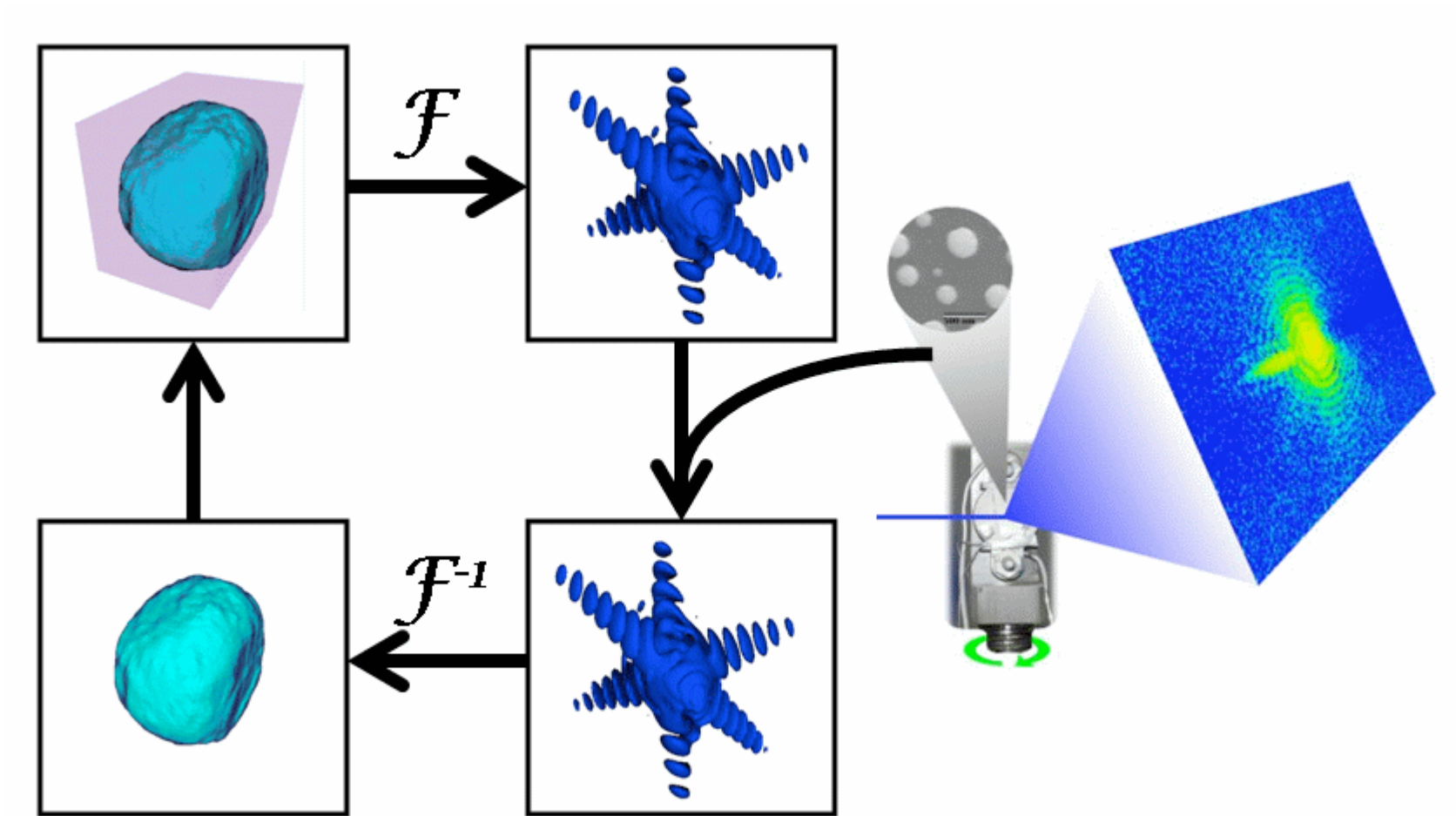
Lensless X-ray Microscope, 2003



Oversampling solves Phase Problem

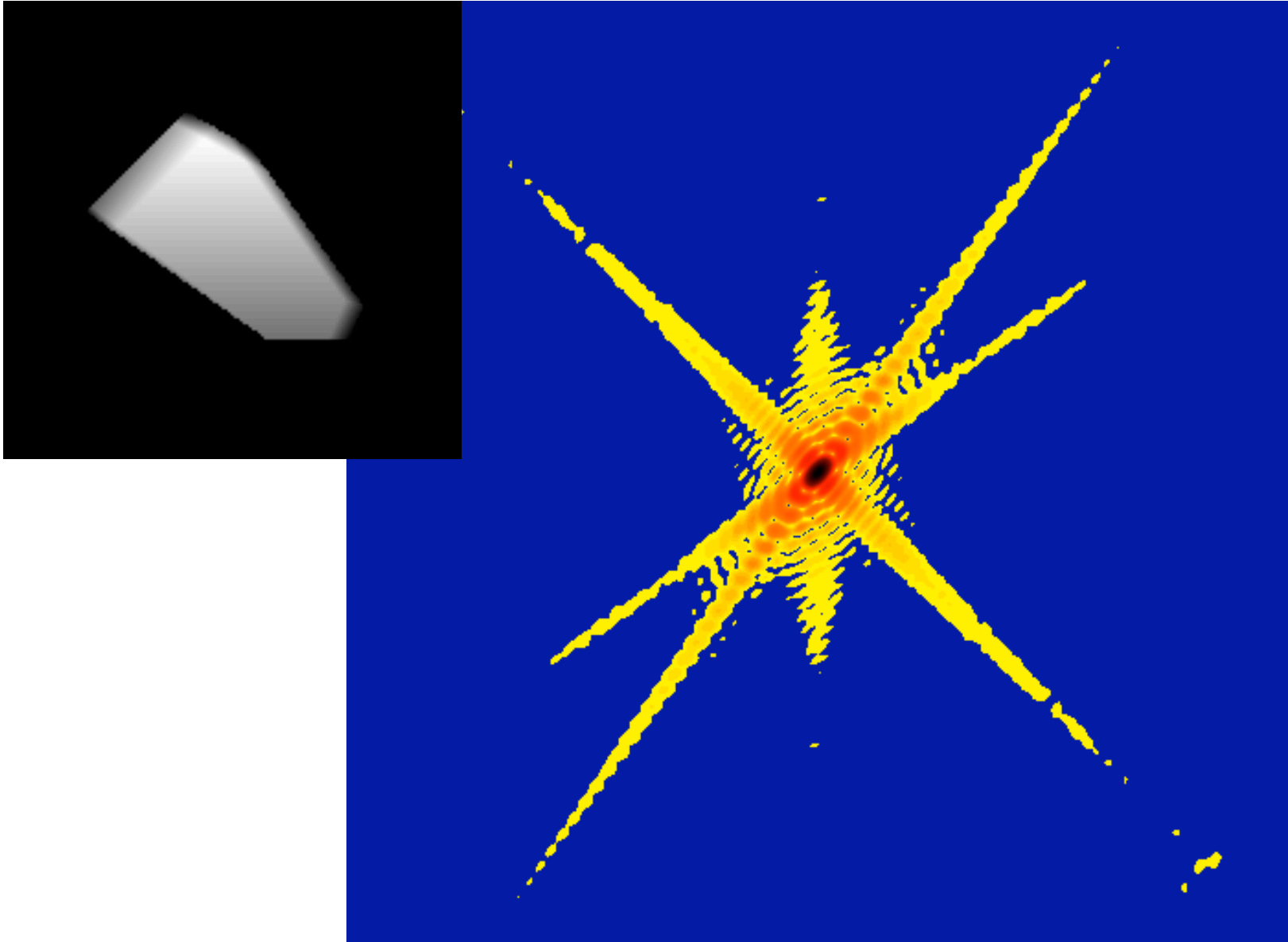


Generic “Error Reduction” method

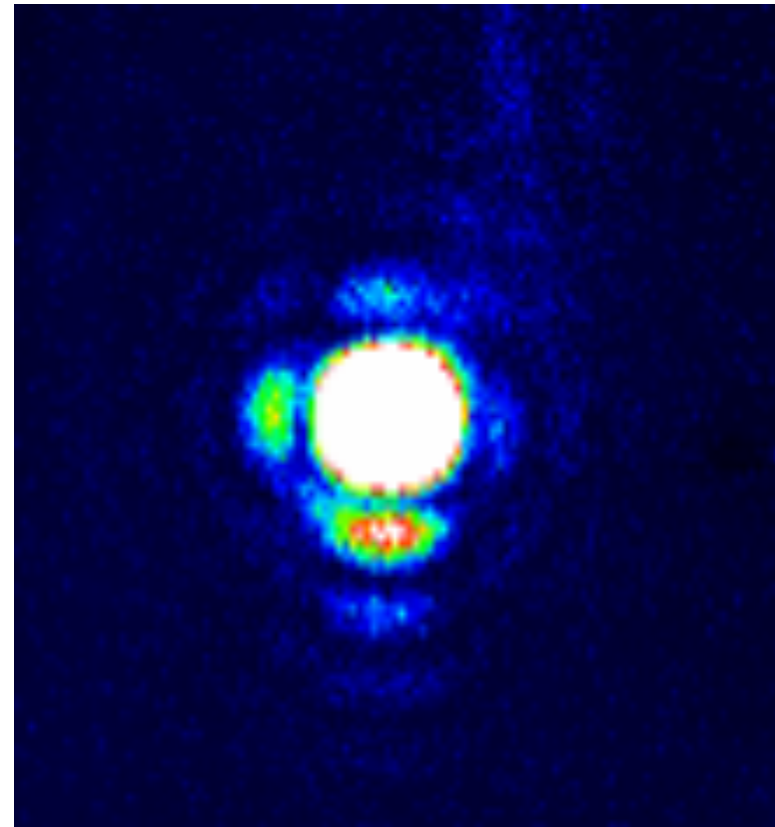
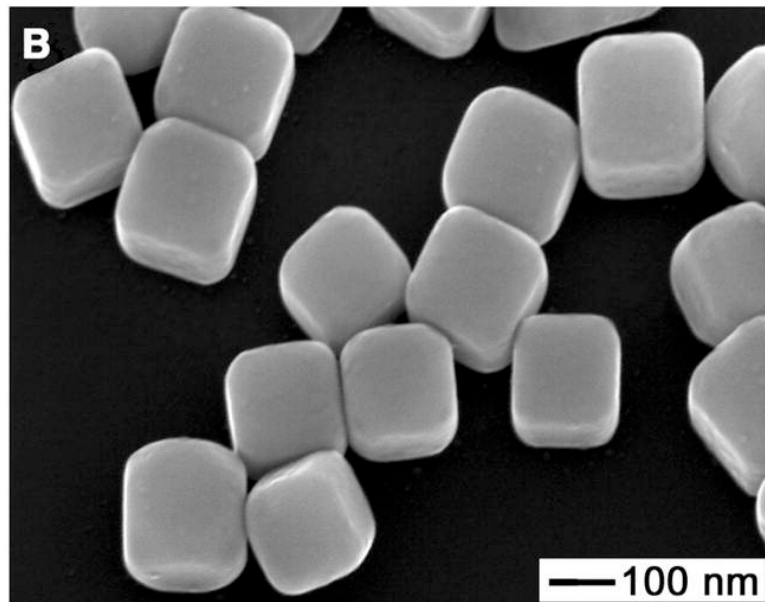


J. R. Fienup *Appl. Opt.* 21 2758 (1982)

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



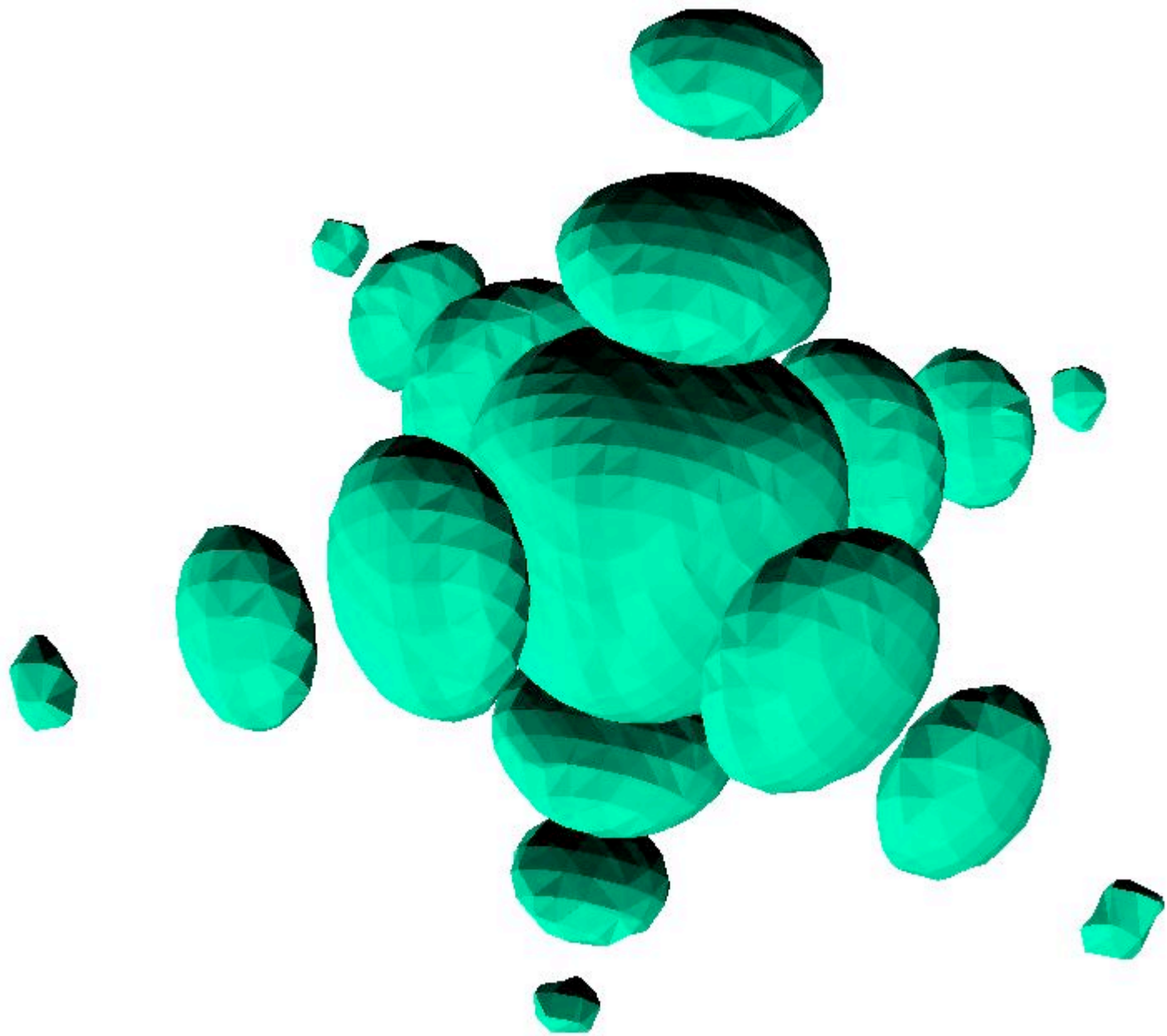
Chemically Synthesized Silver Nanocubes

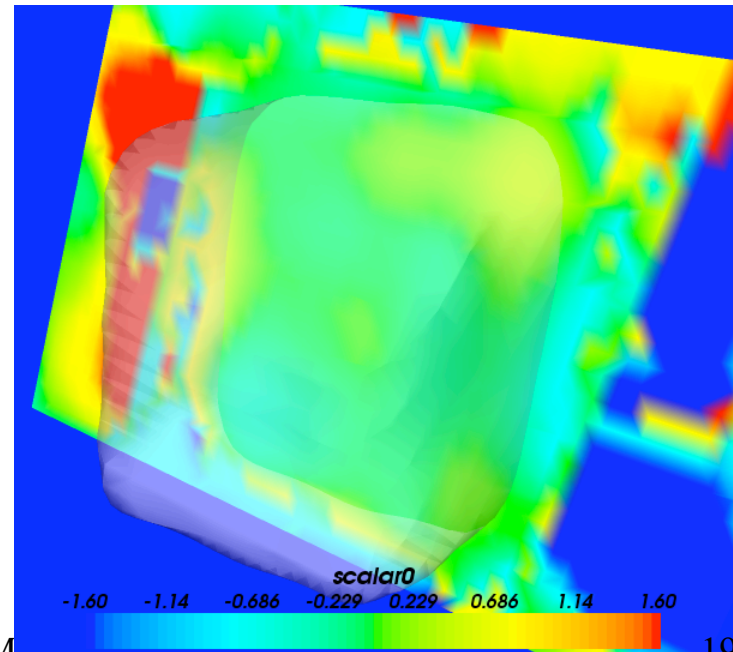
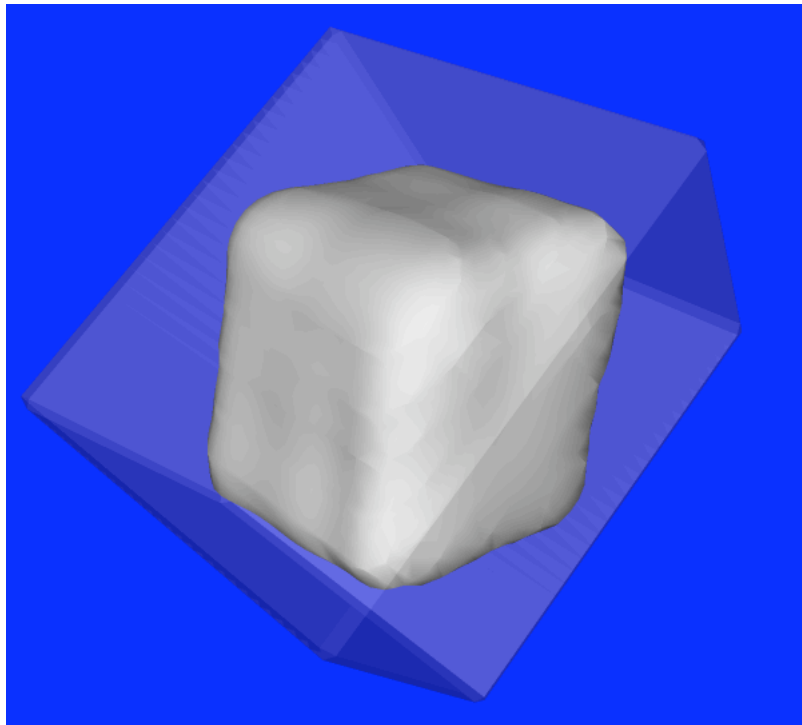
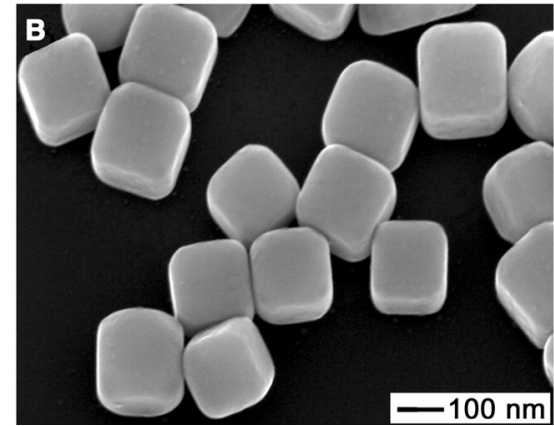
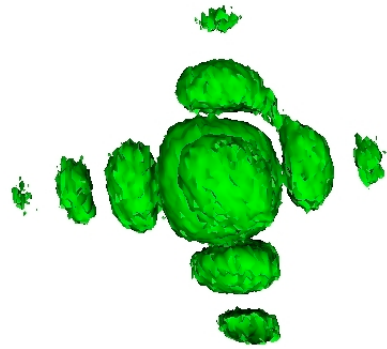
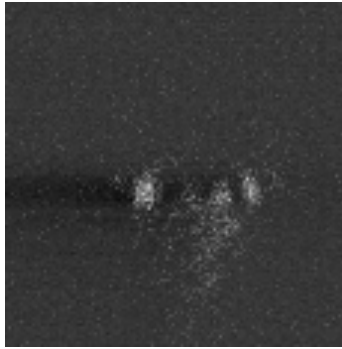


Yugang Sun and Younan Xia,
Science 298 2177 (2003)

Rocking
scan of Ag
cubes with
 0.01° steps

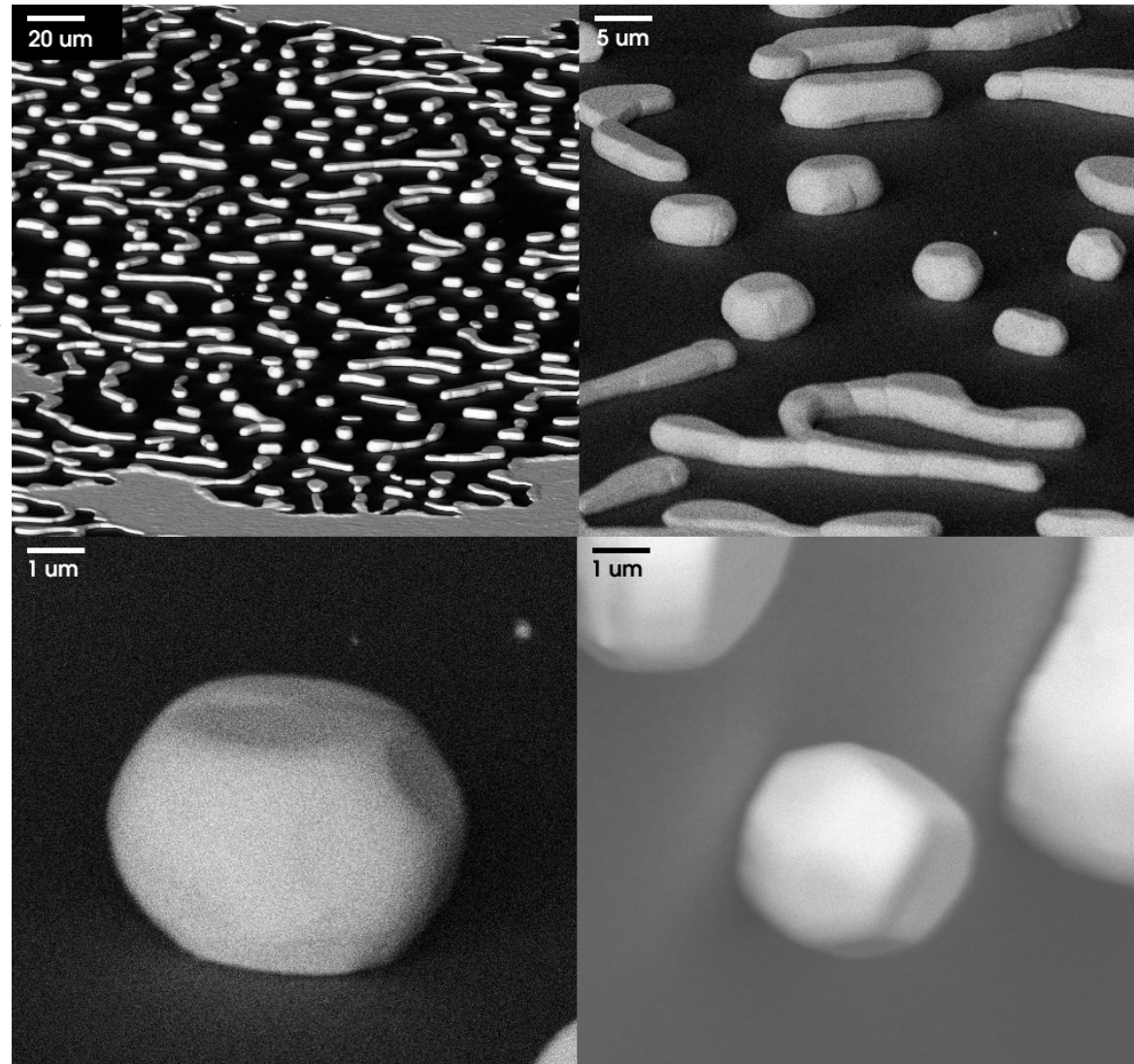






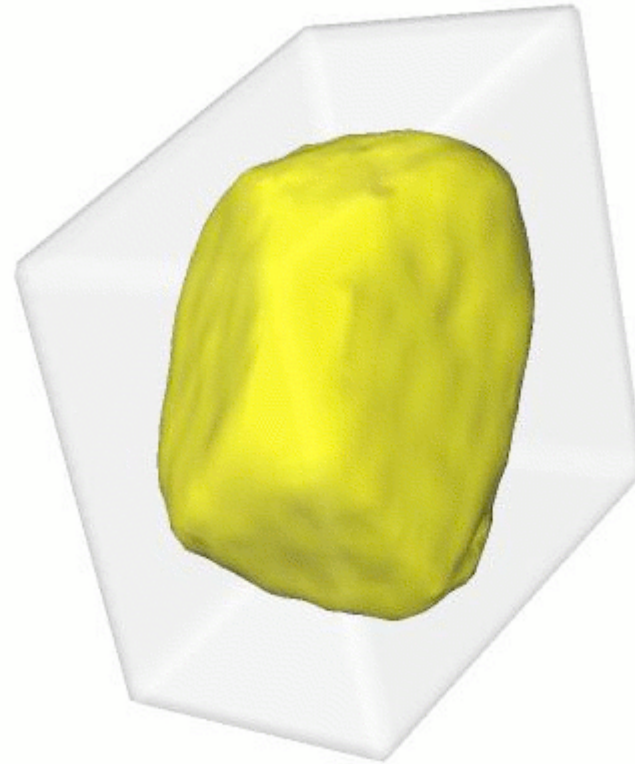
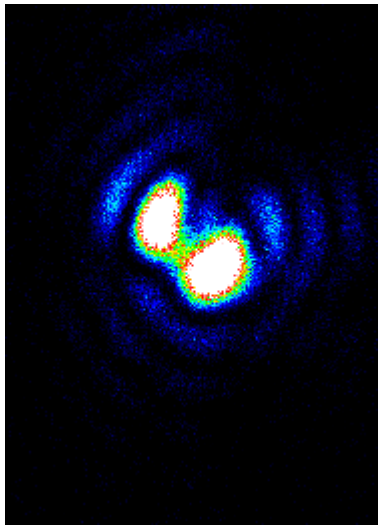
SEMS

- Au blanket film
- Quartz substrate
- Annealed at 950°C for 70 hrs.

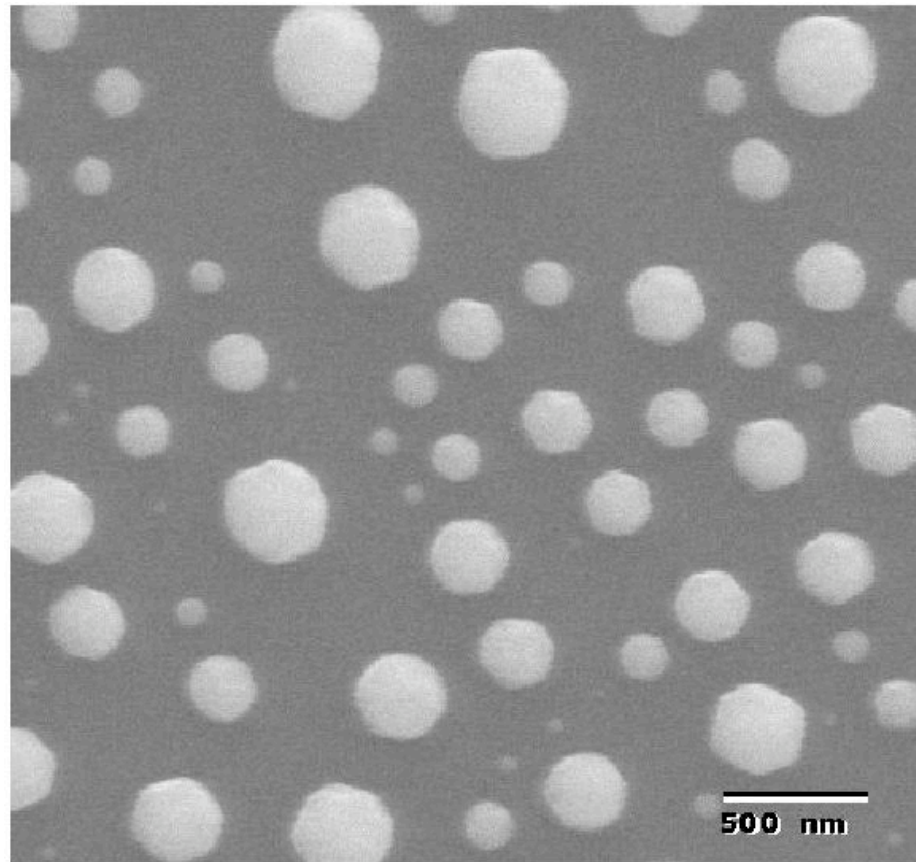
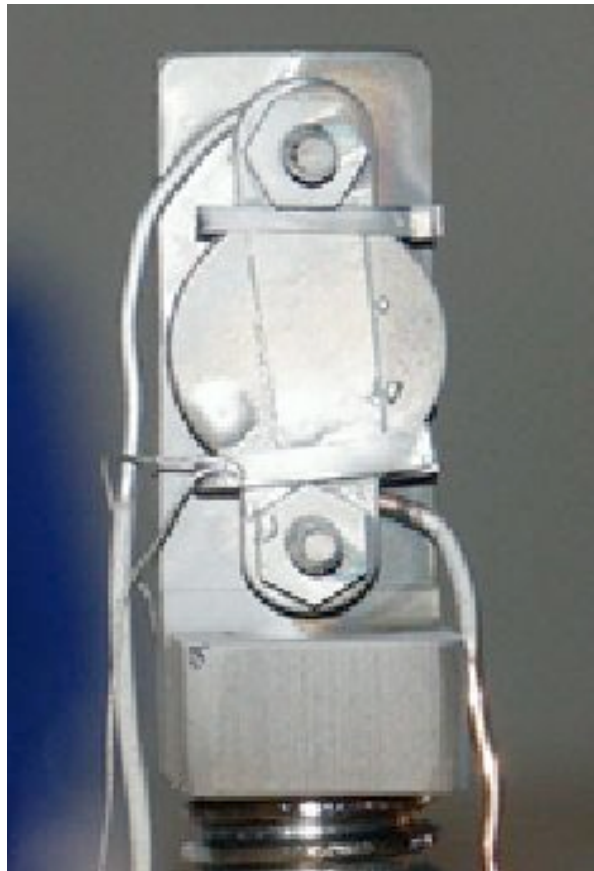


Gold nanocrystal reconstruction

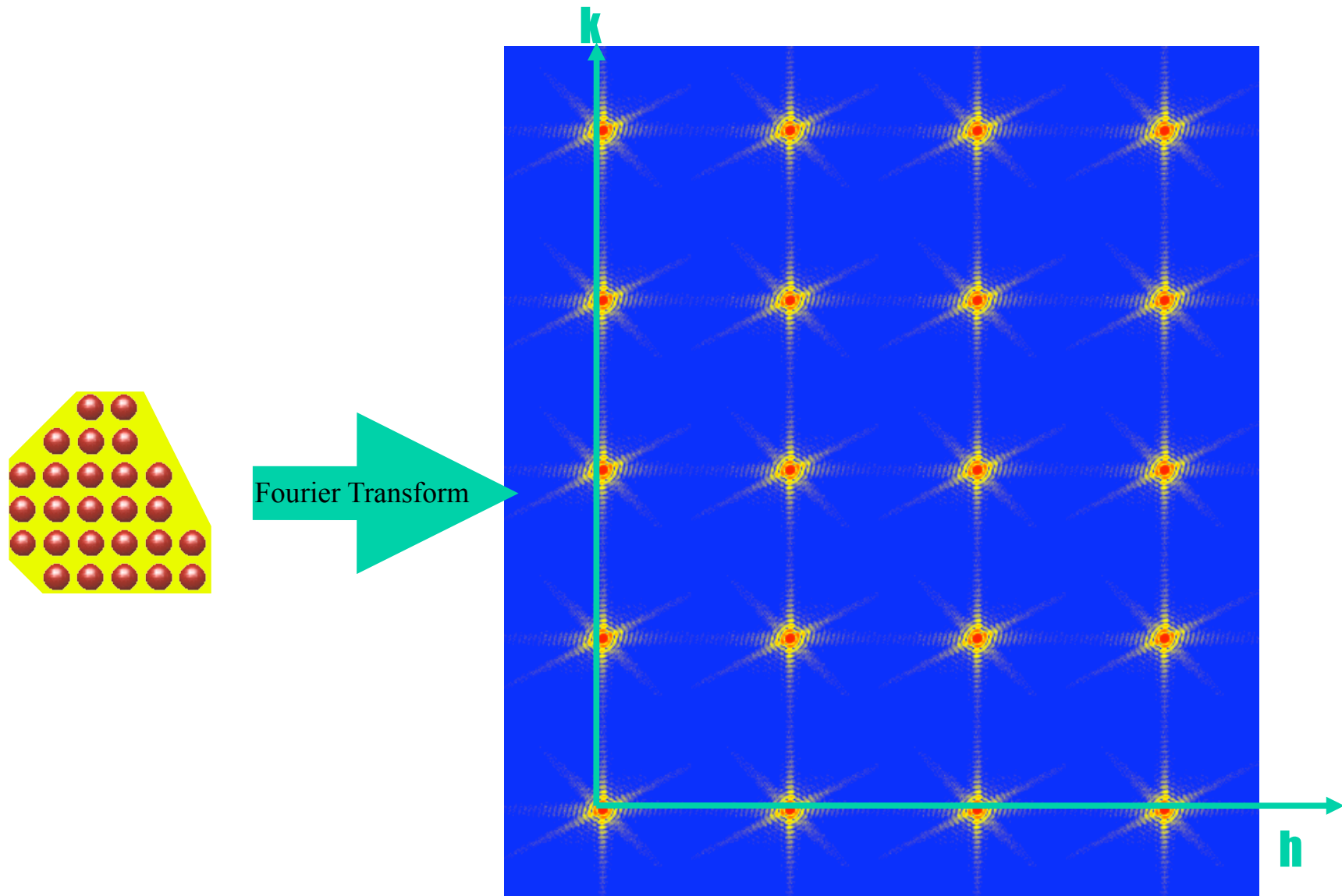
showing support used for 20 HIO followed by 10 ER



In situ growth of Pb crystals

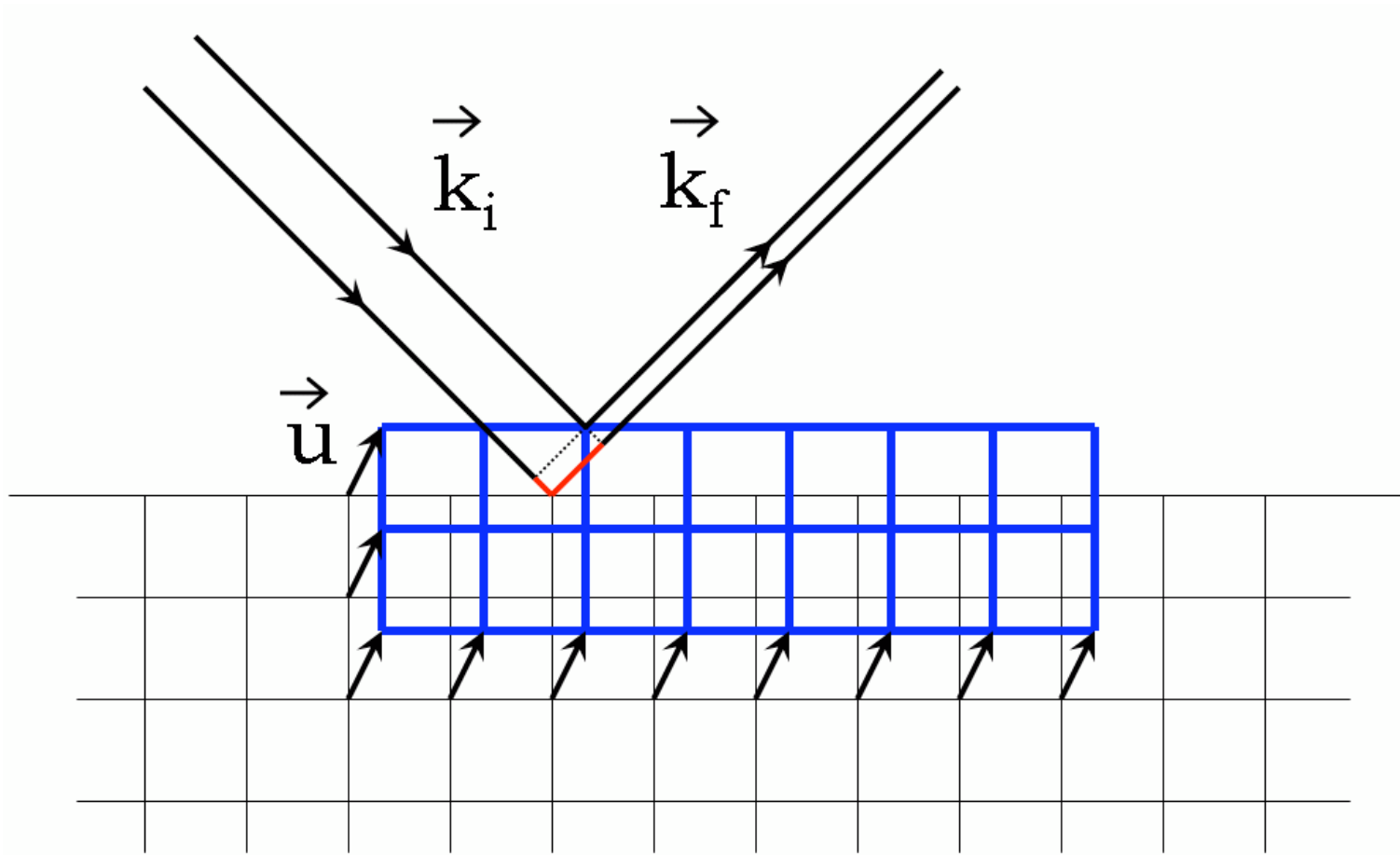


Coherent Diffraction from Crystals



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

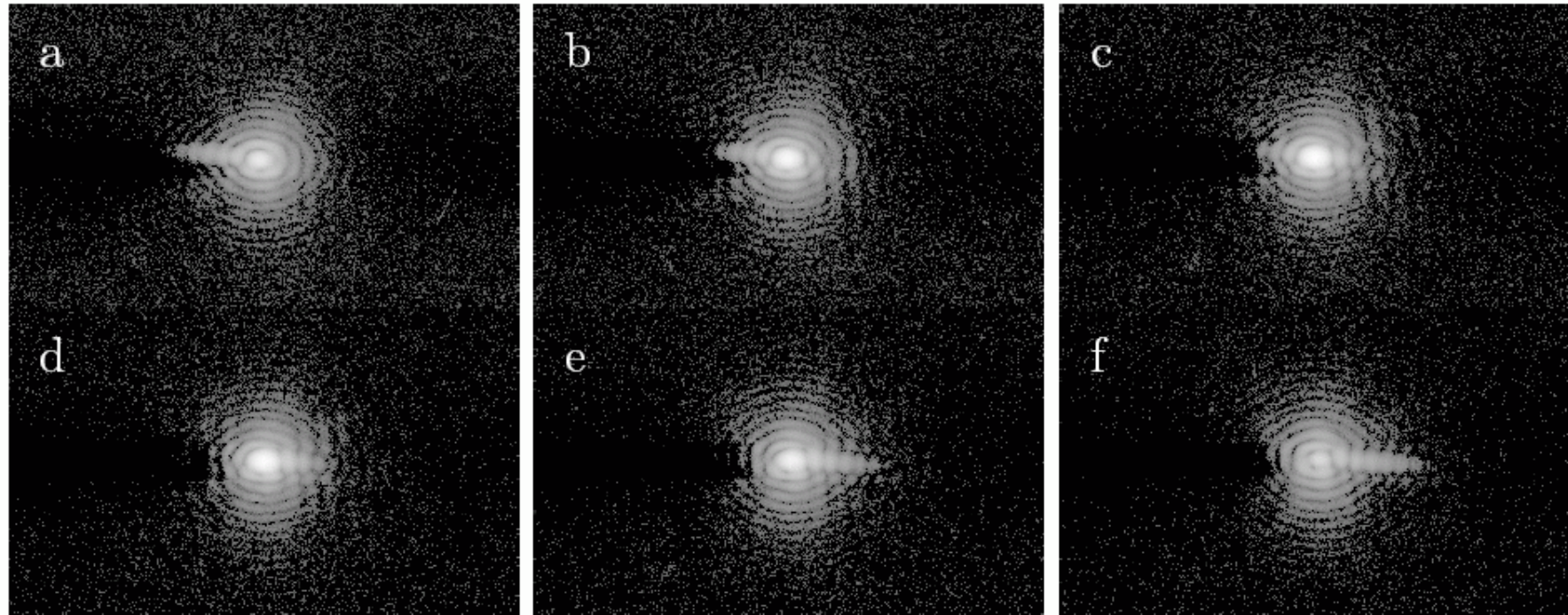
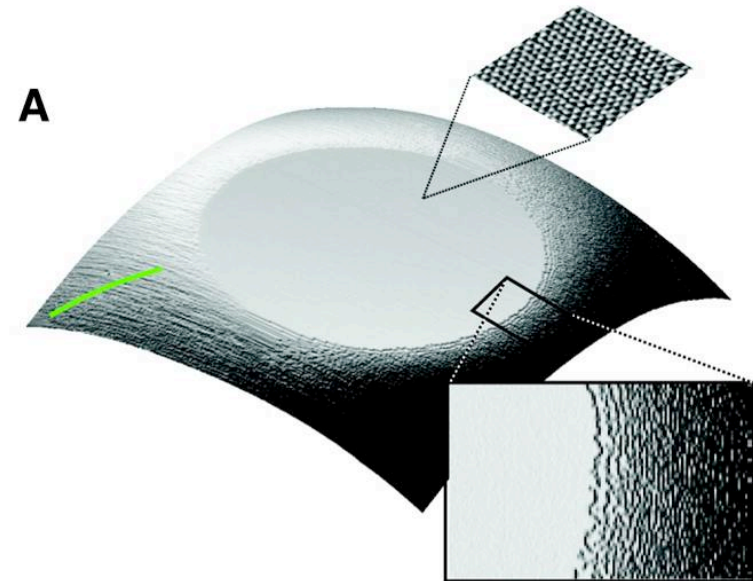


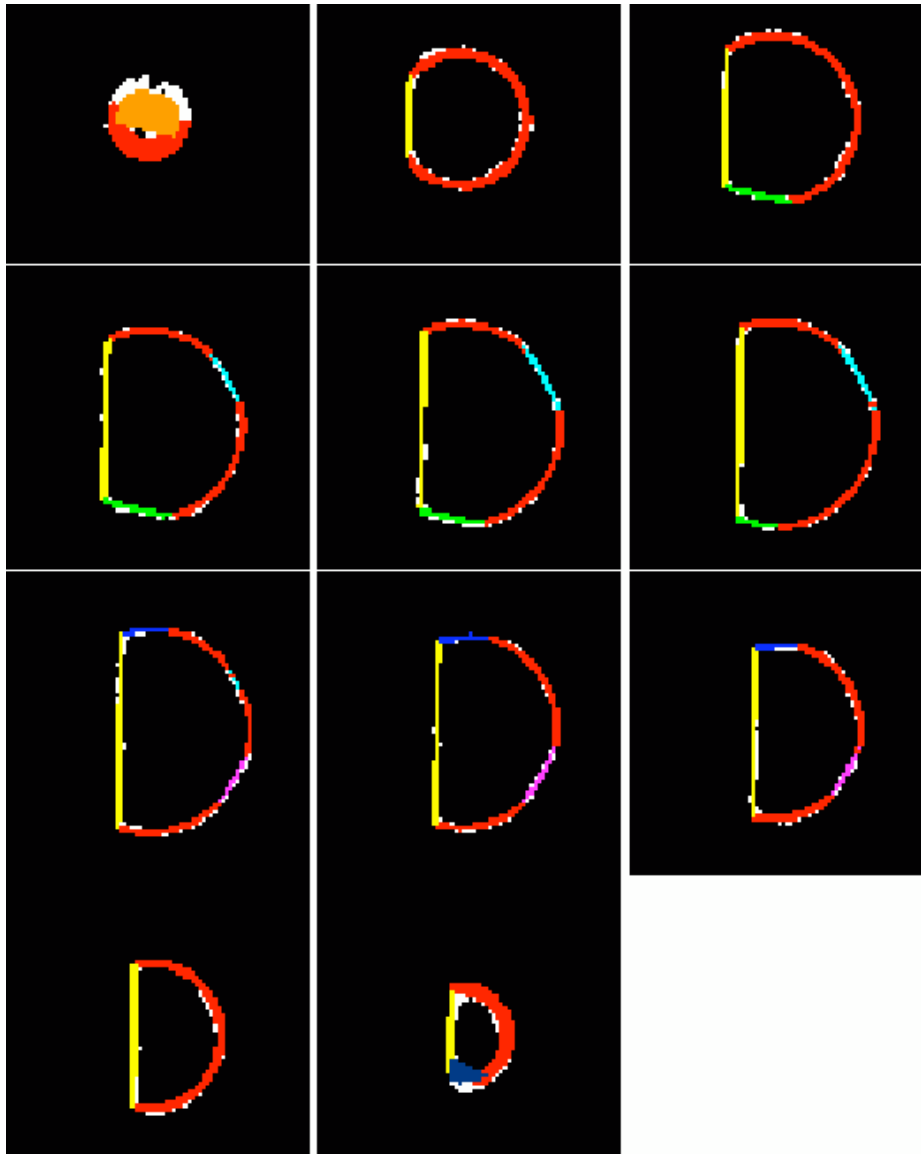
Figure 4.12: Center slices from 3D CXD pattern from Pb sample, on a log scale. Data file 296 from 10/03.

Facets of Equilibrium Crystal Shape



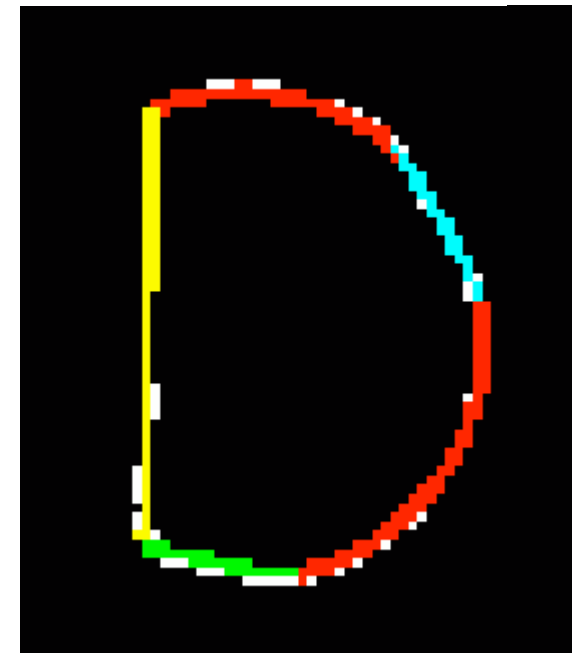
Thurmer K, Williams E, Reutt-Robey J
Science **297** 2033 (2002)

Fitting to faceted shape

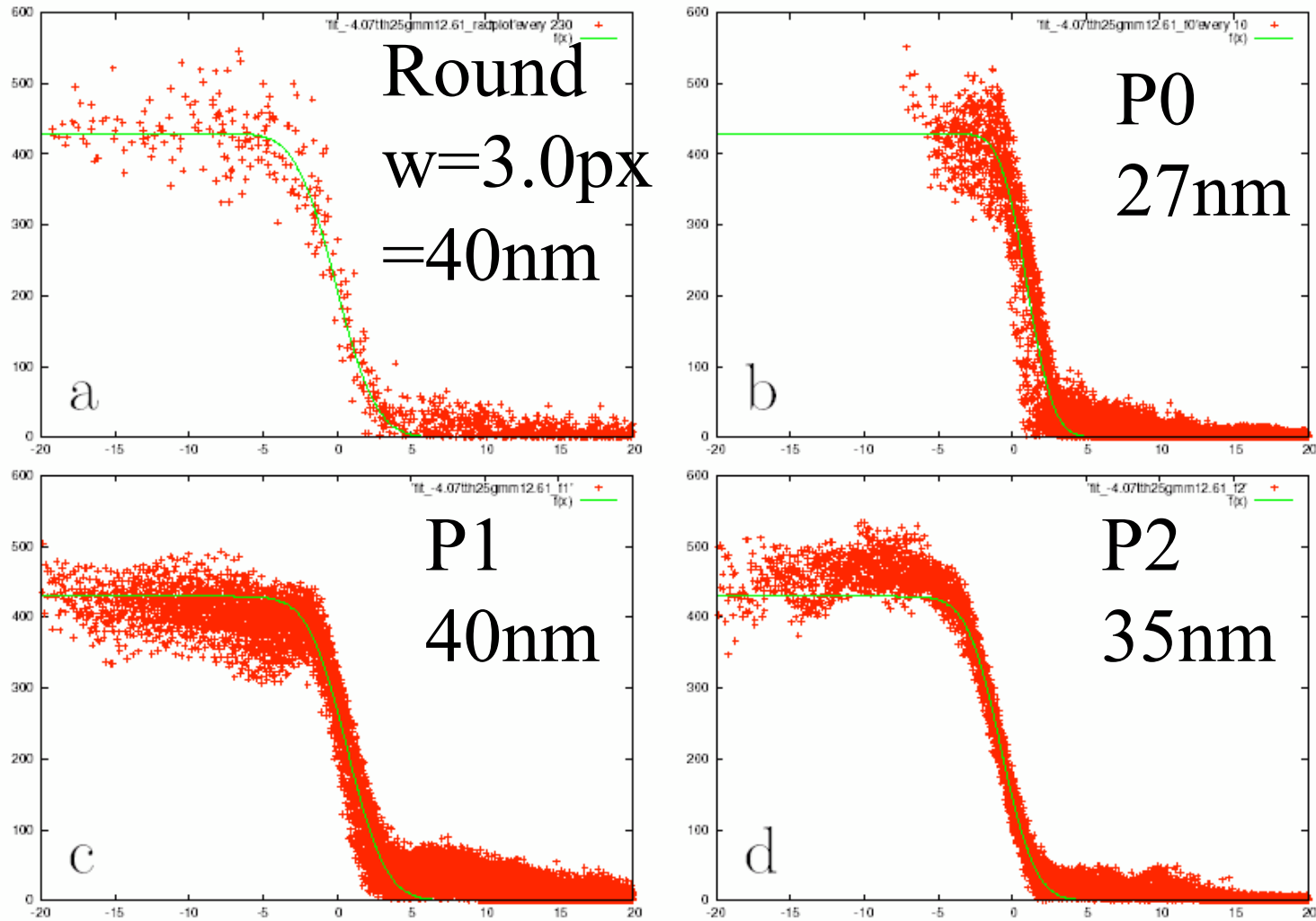


	$ R $		P0	P1	P2	P3	P4	P5	P6
	9.4	P0	0	85	149	79	134	106	71
	25.7	P1		0	123	164	83	76	102
	25.1	P2			0	72	67	74	110
	25.9	P3				0	111	106	76
	25.4	P4					0	113	68
	25.4	P5						0	176
	26.0	P6							0 0

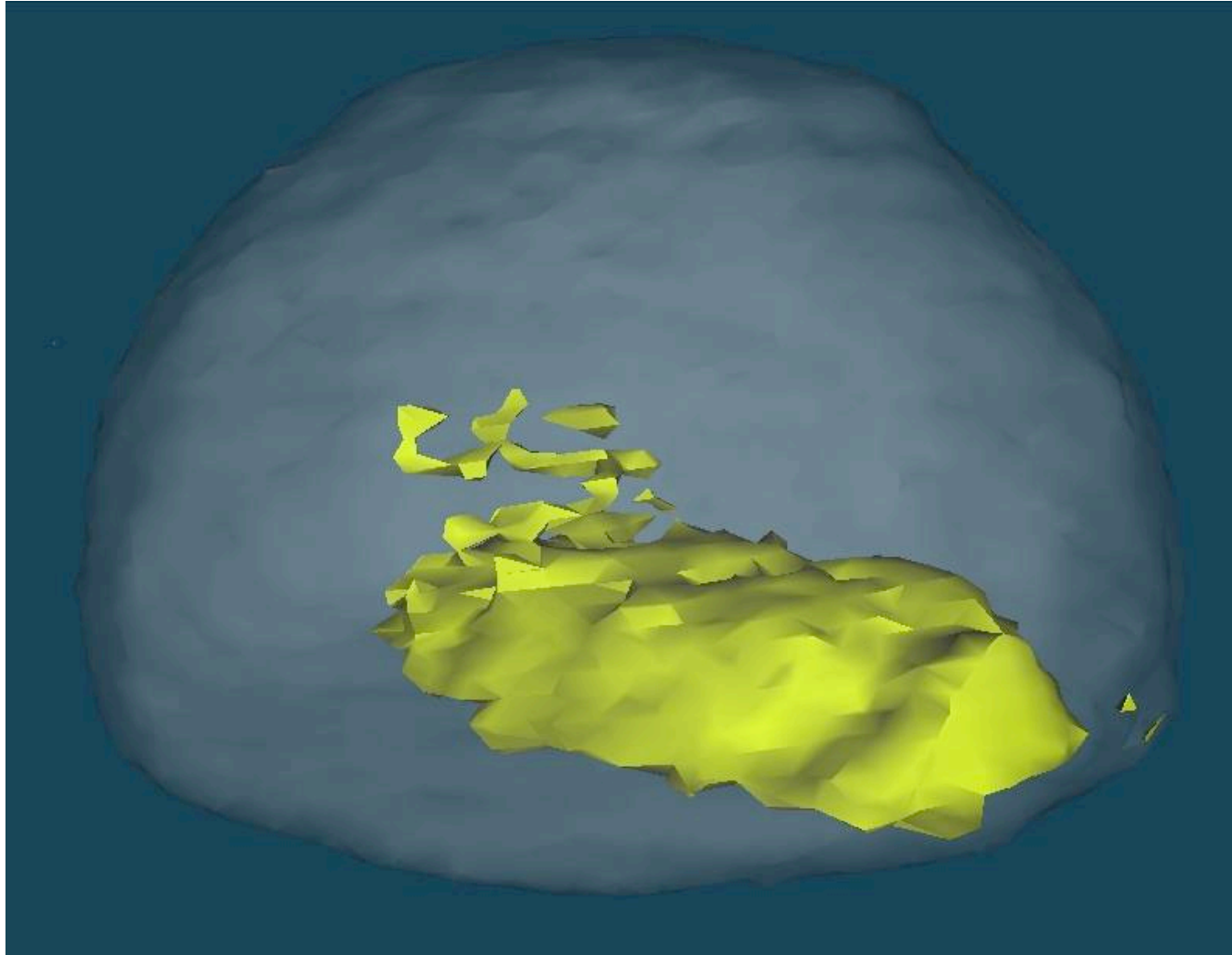
Angles between facets



Density distribution across surface

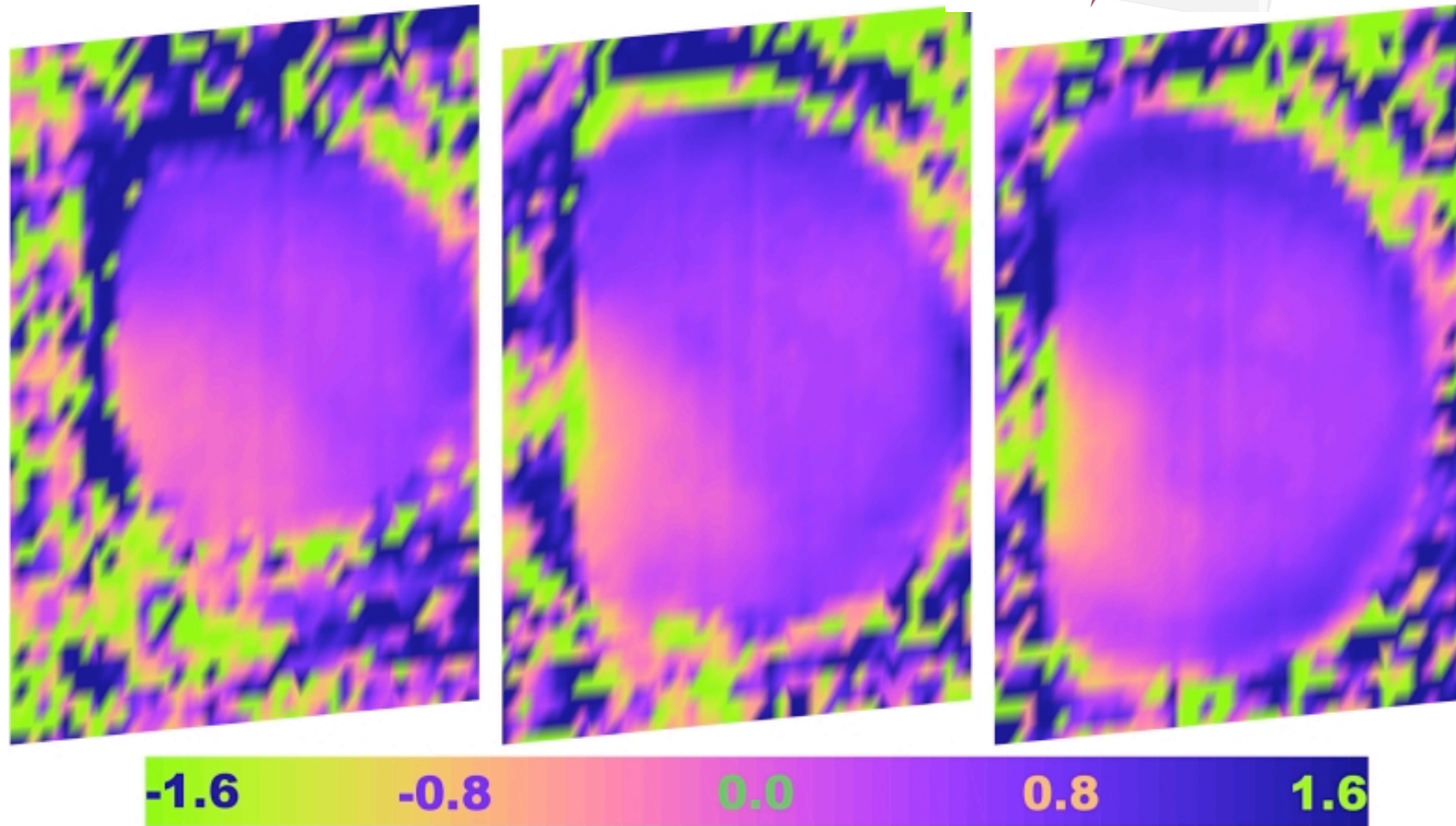
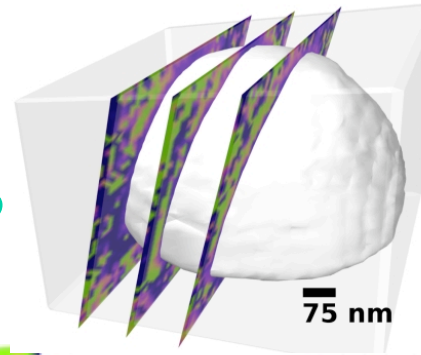


Modeling of 3D Phase Bump

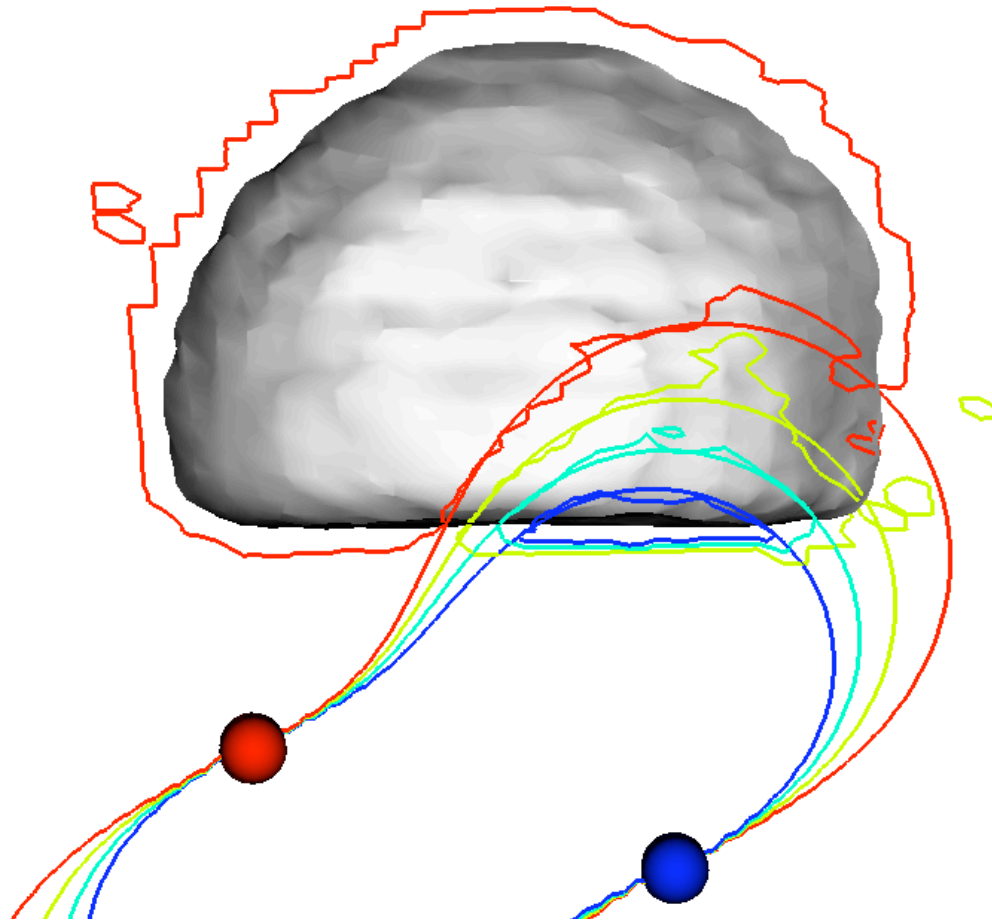


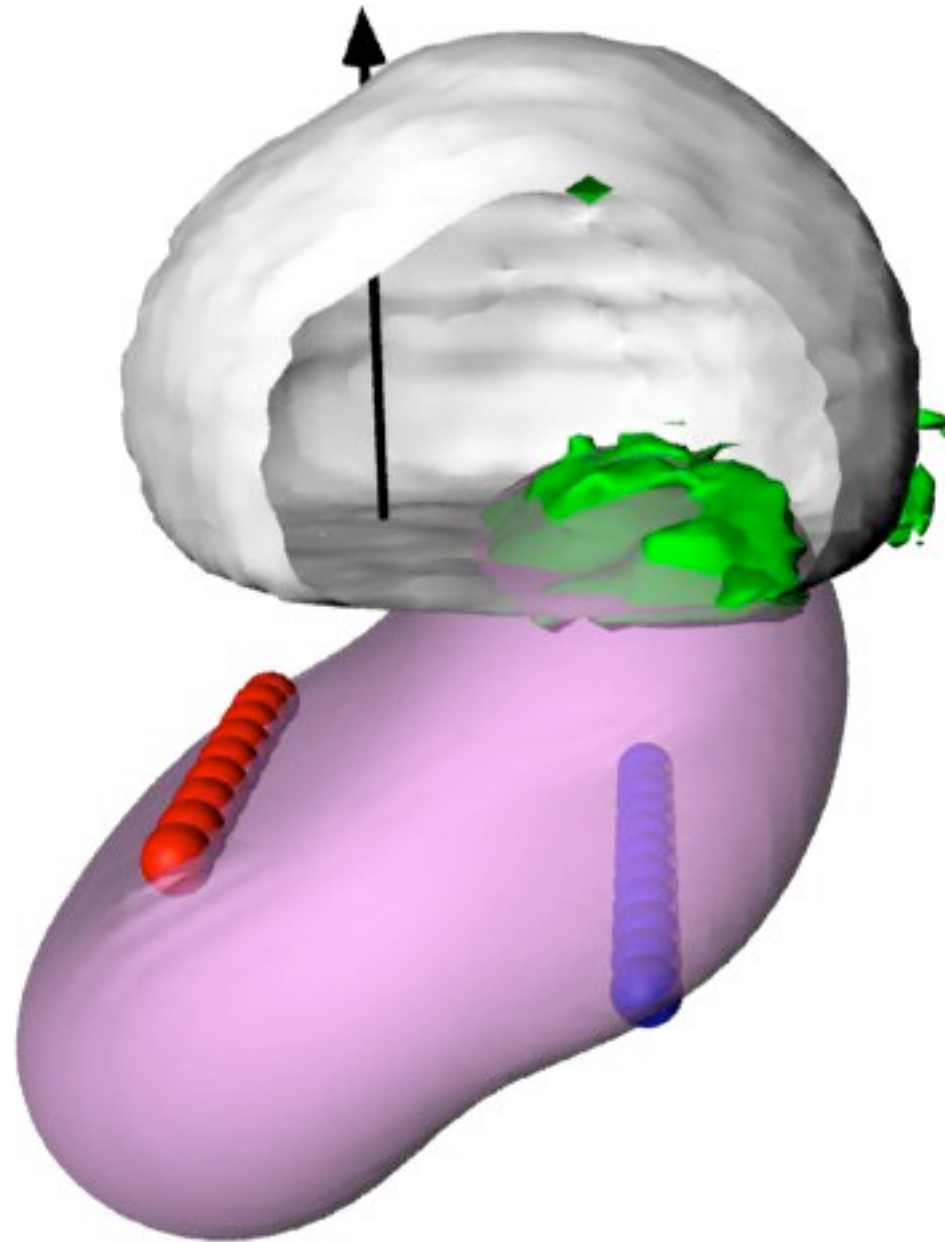
I. K. Robinson, XMNP2009

3D phase map sections



Field lines of “Point Charges”

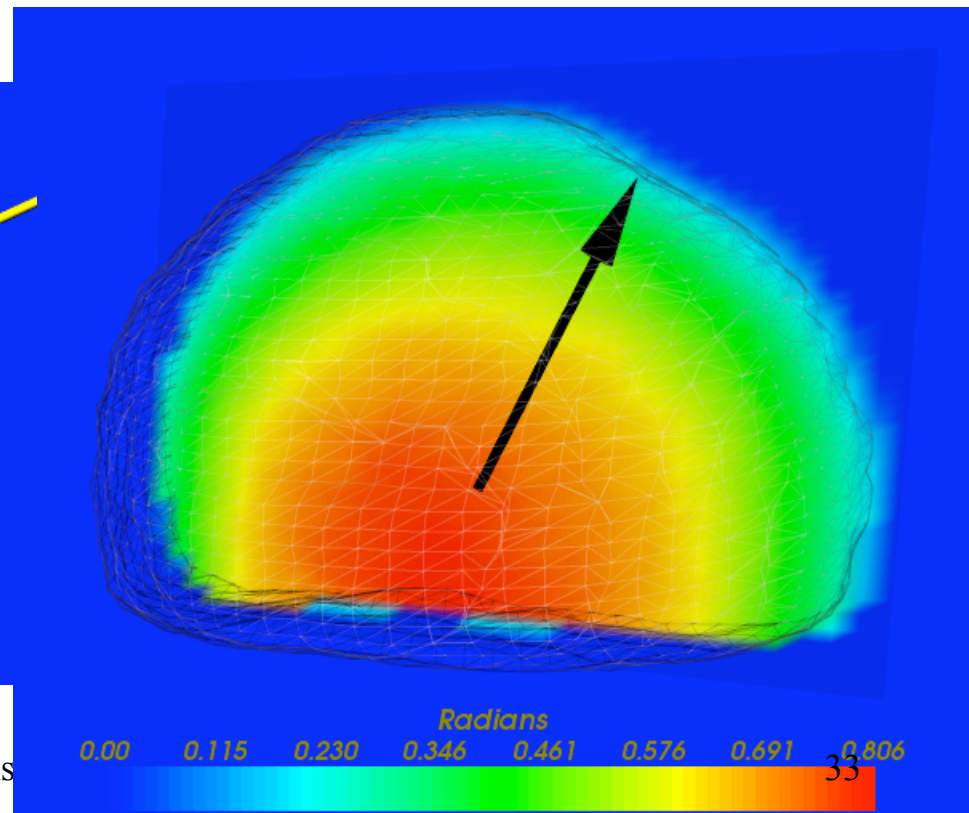
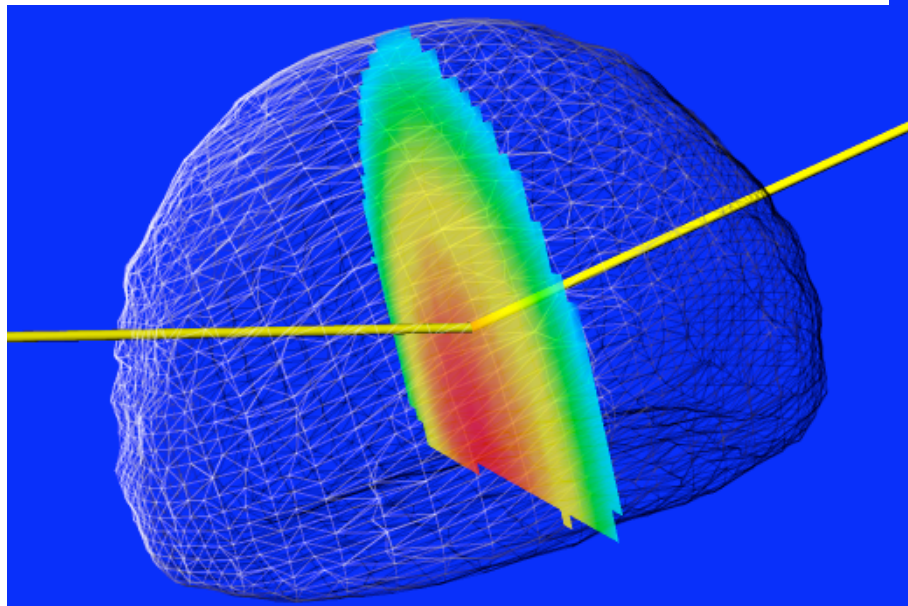




Refraction effects in Lead at 8.9keV

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

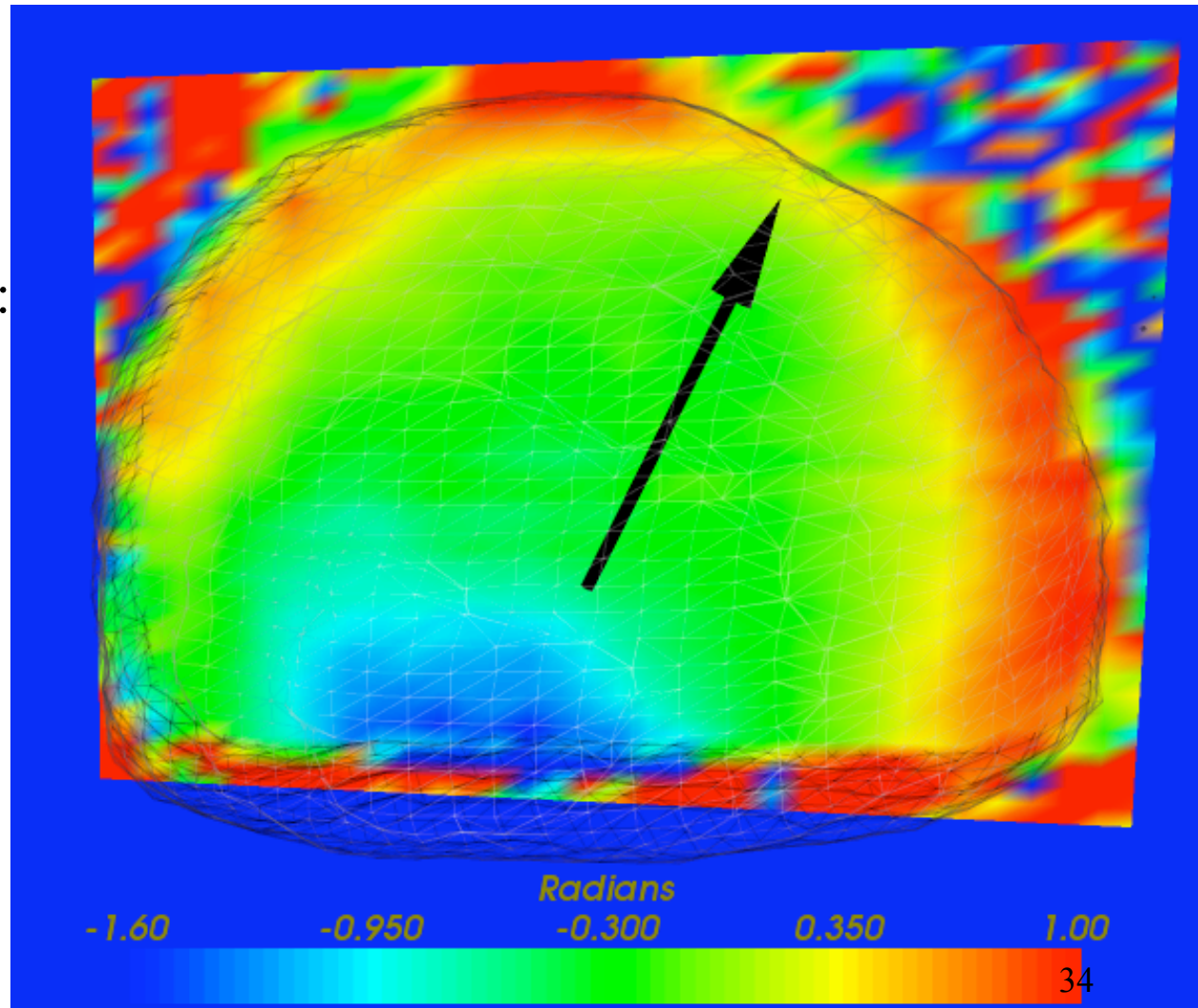
$$\delta=2.23\times 10^{-5}$$
$$\beta=2.19\times 10^{-6}$$



Refraction corrected phase map

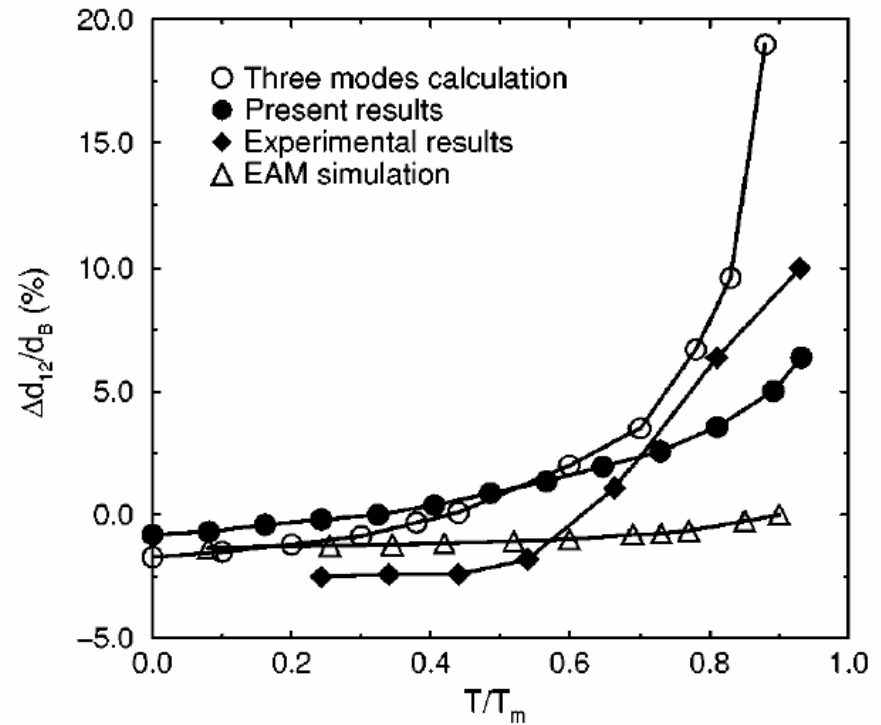
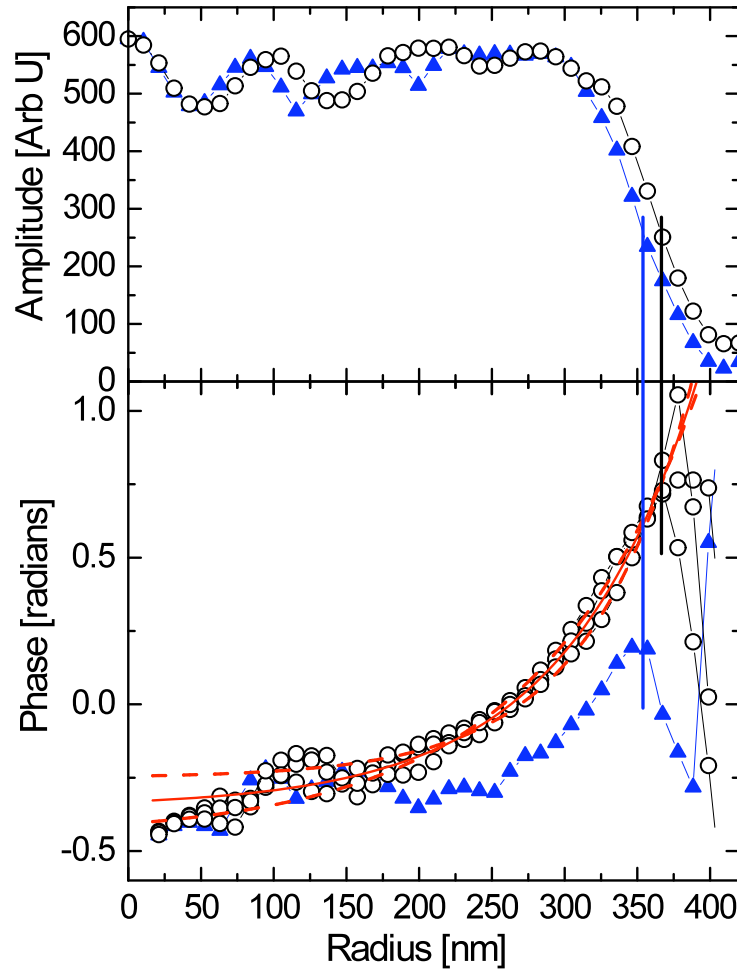
Max phase = 1.15rad
= 0.052nm

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



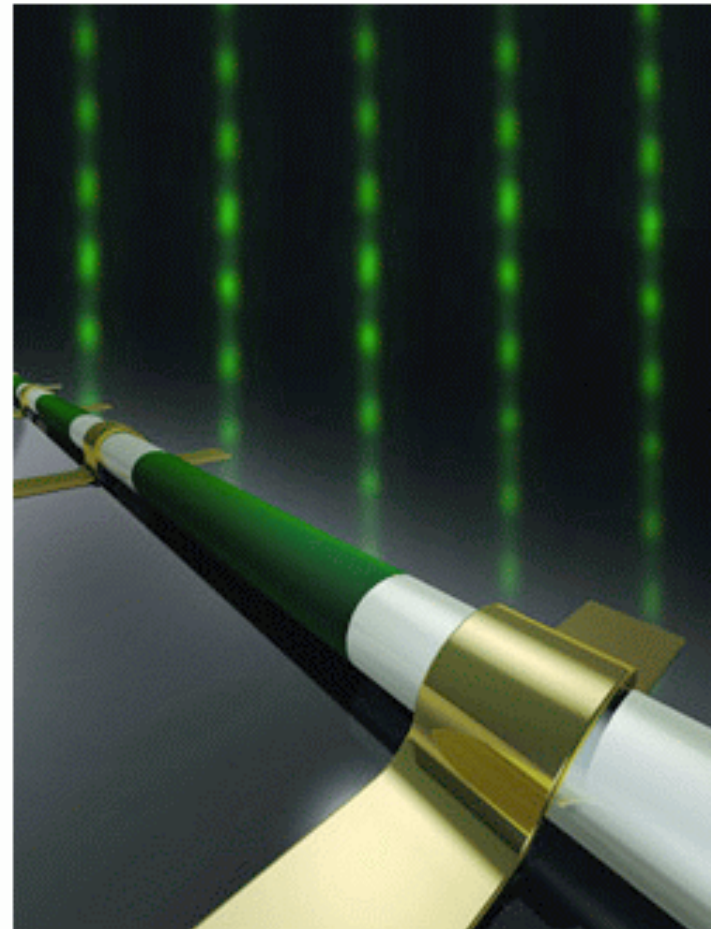
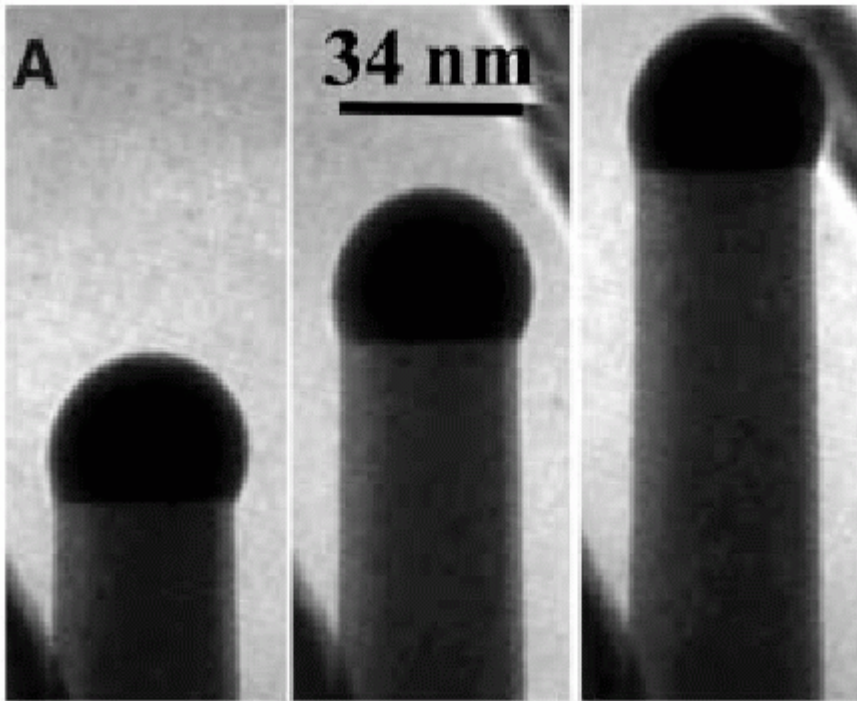
Surface Thermal Expansion

Ag(111): Scheffler et al PRB 59 970 (1999)



VLS growth of nanowires

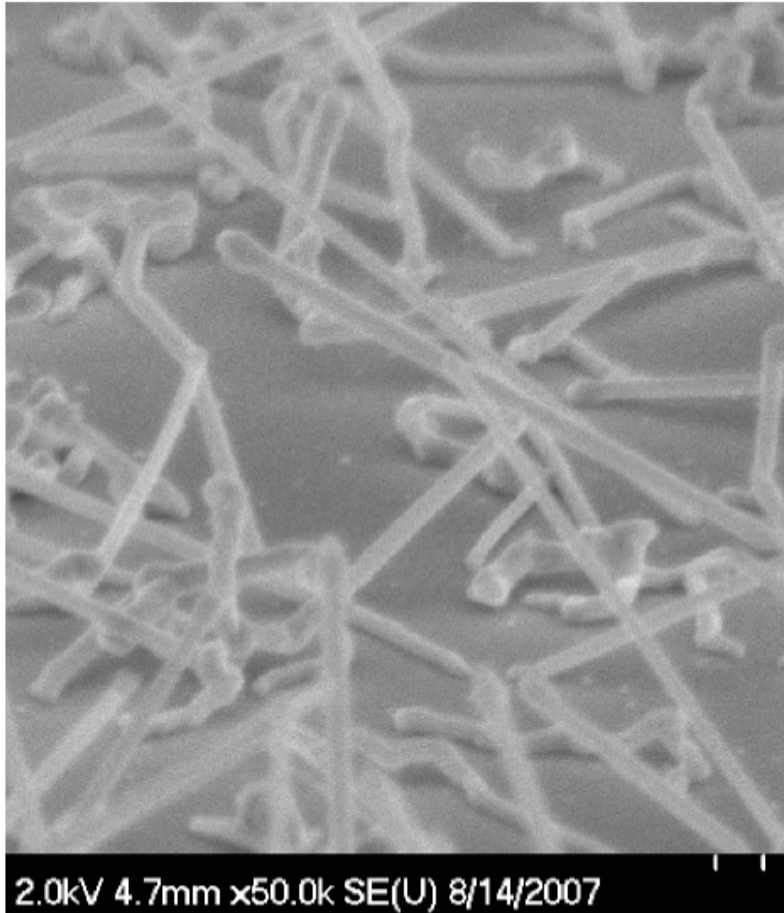
S. Kodambaka et al., *Science* 316 729 (2007)



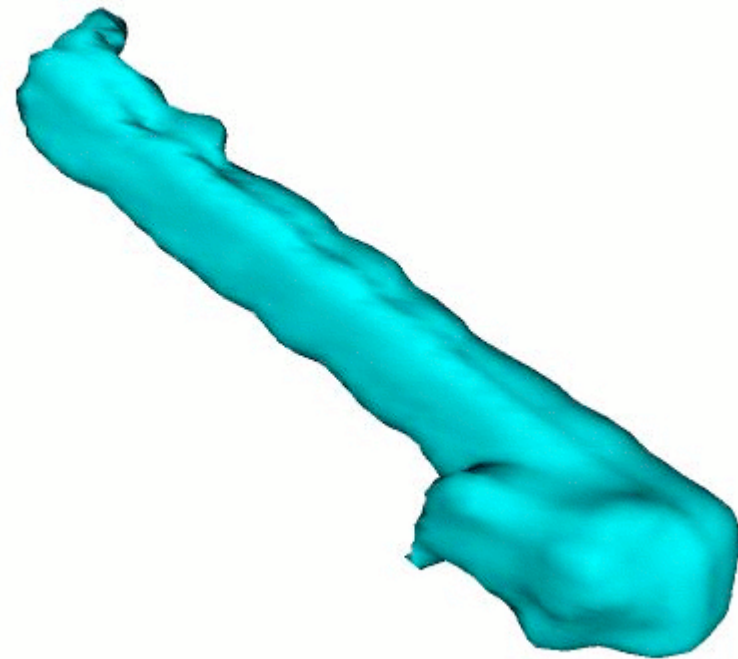
I. K. Robinson, XMN NiSi/Si nanowire heterostructure devices. *Nature* **430**, 61 (2004).

Reconstruction of InP nanowire

CVD on Si, Suneel Kodambaka, UCLA

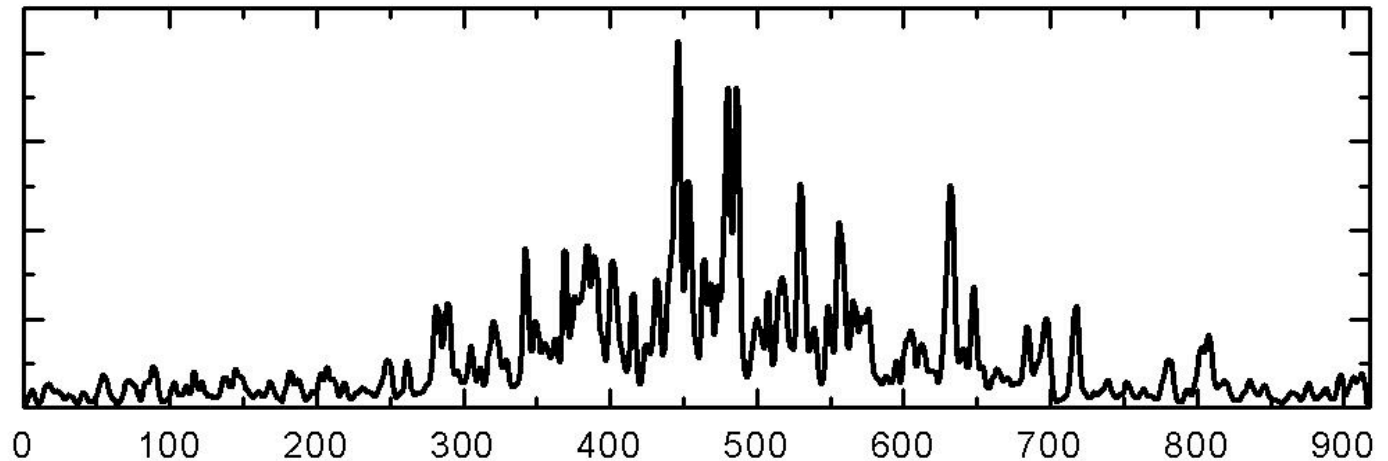
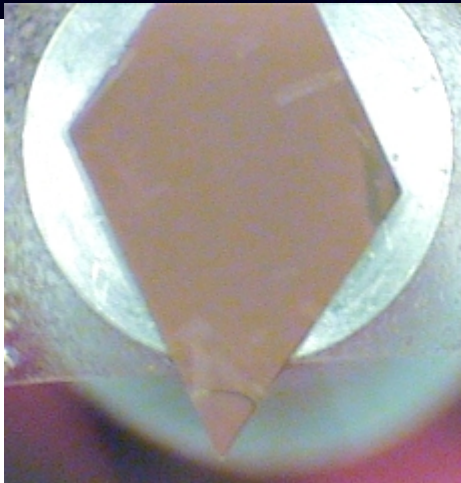
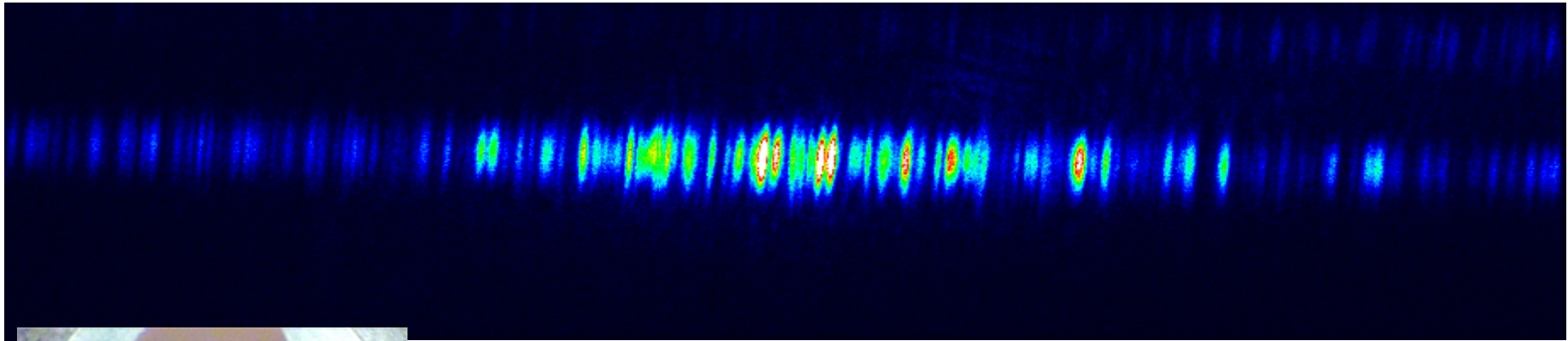


InP nanowires grown on Si (111)



GaAs Nanowire “Barcode”

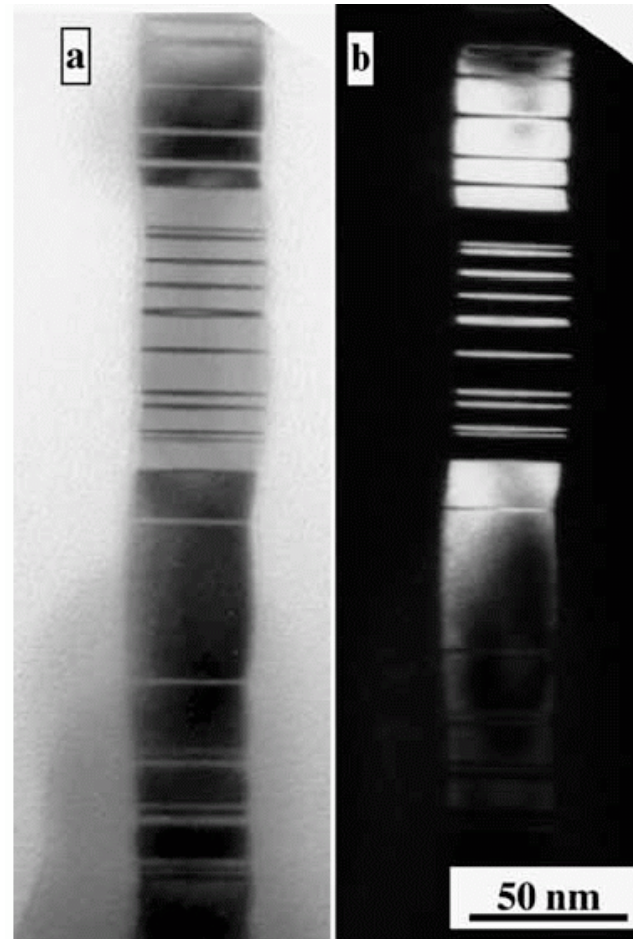
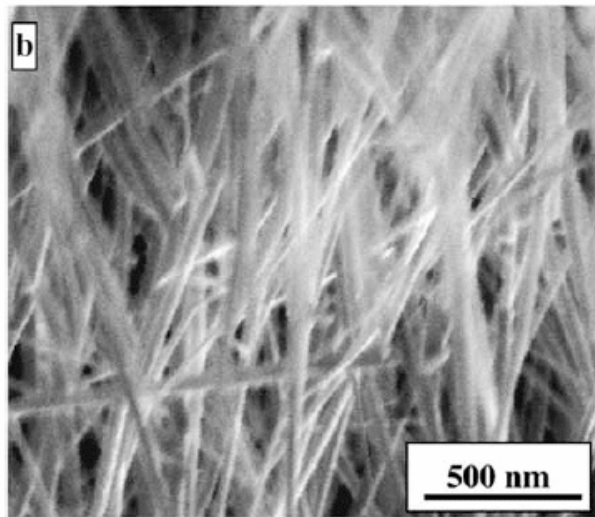
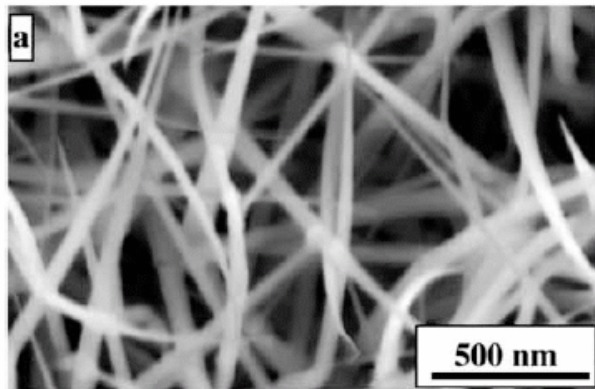
Vincent Favre-Nicolin, Joel Eymery (CEA),
Rienk Algra (Philips), Ross Harder



GaAsNW1106-22.spe
B9348 from Philips

Dark Field TEM of GaAs Nanowires

R. Banerjee et al, Phil. Mag. Lett. 86 807 (2006)

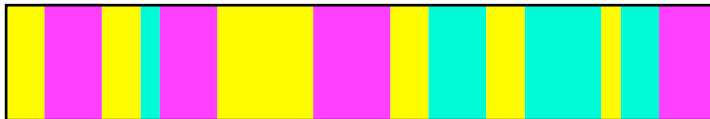


Models of Barcode Diffraction

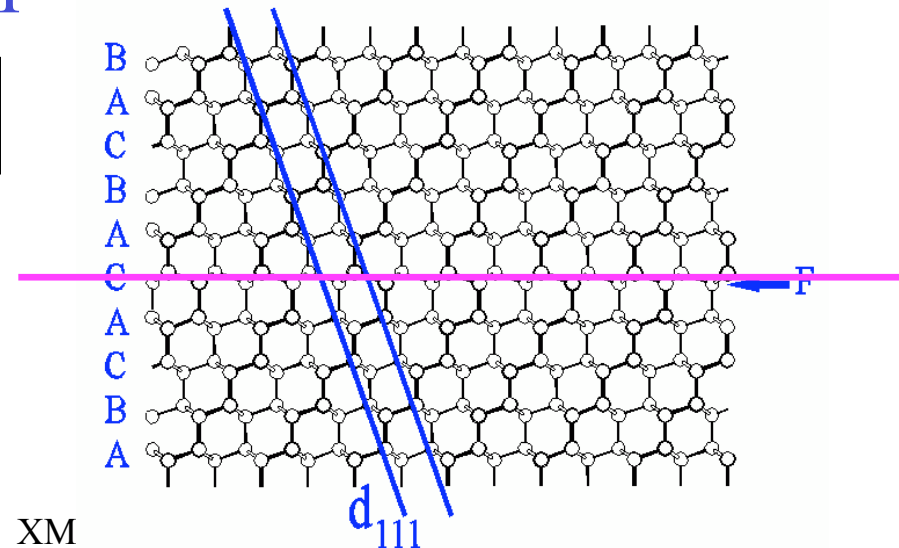
(111) wires at (11-1) reflection



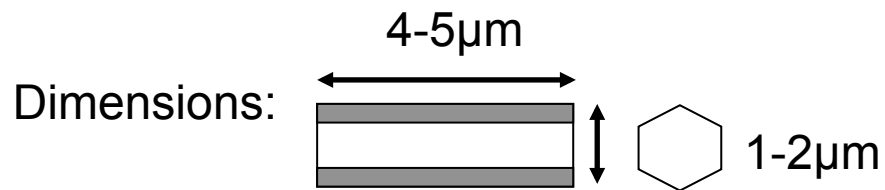
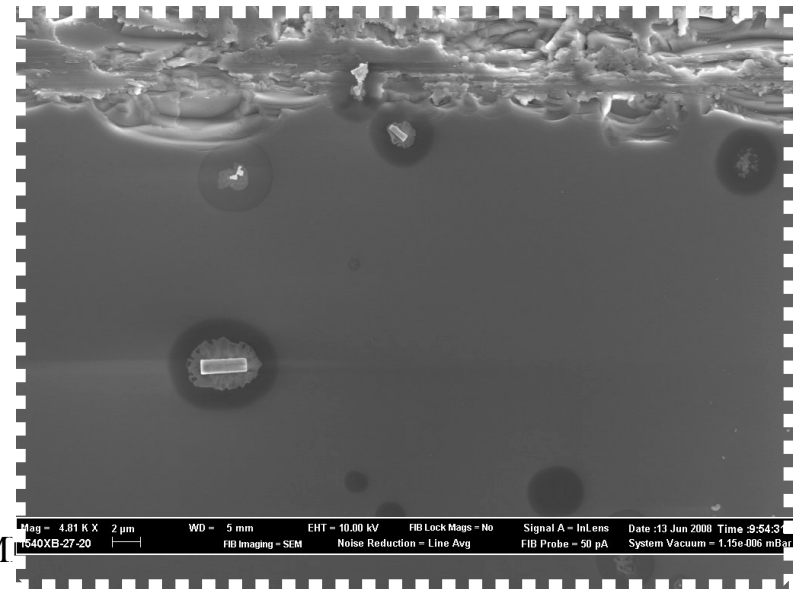
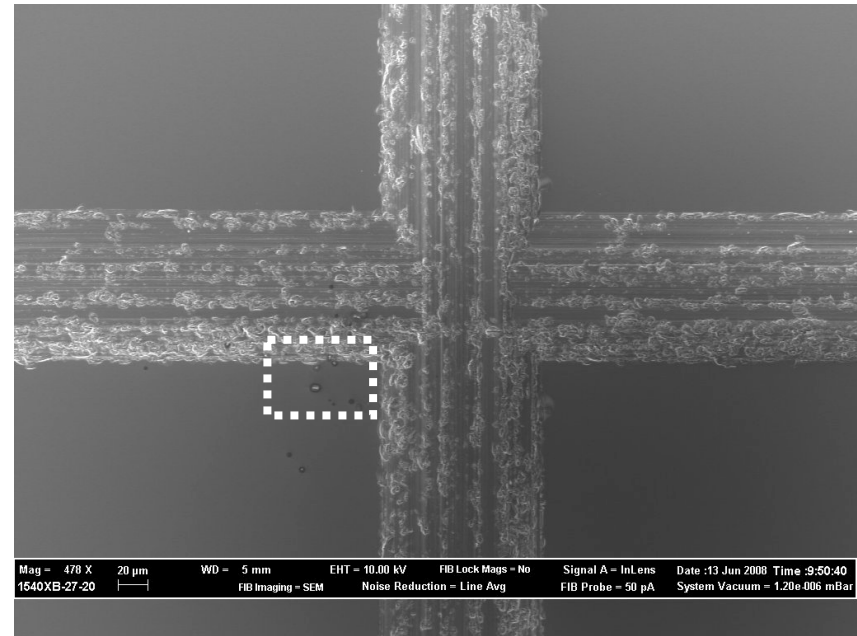
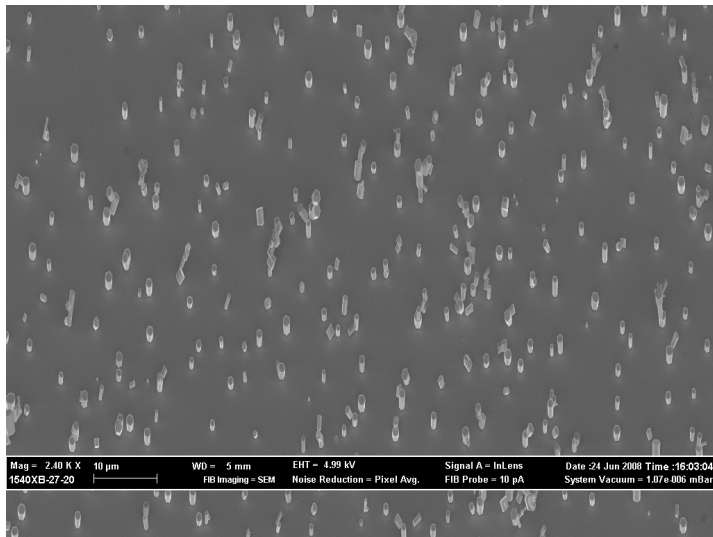
- Twinned stacking sequence



- Deformation faults

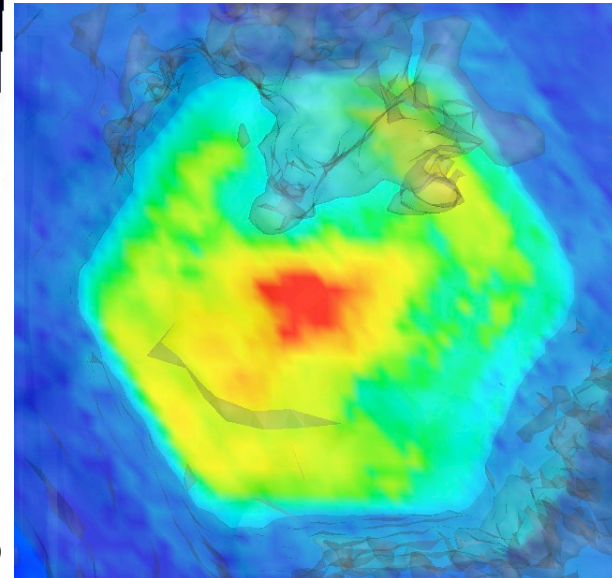
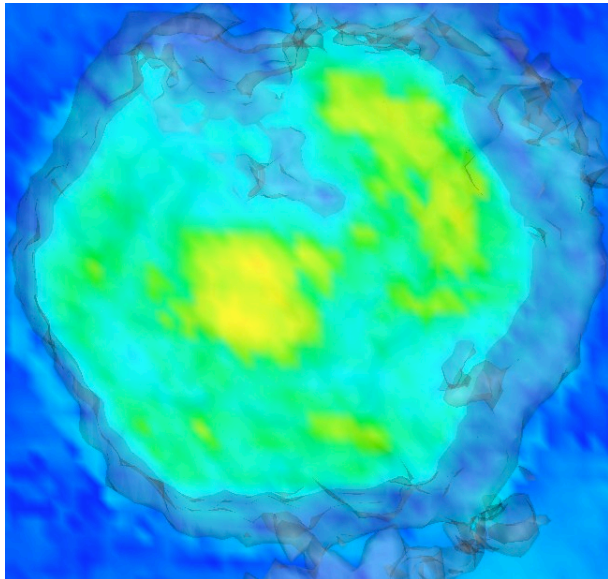
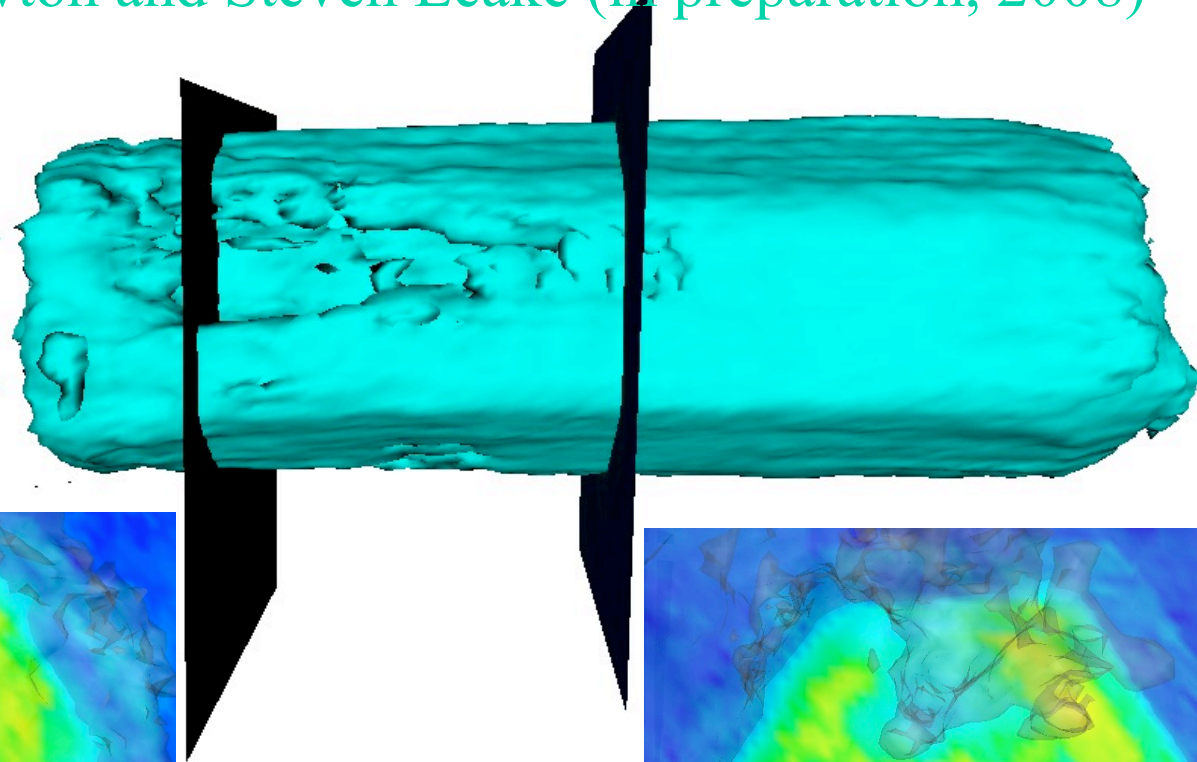


ZnO Sample Preparation



Density sections ZnO-39 (010)

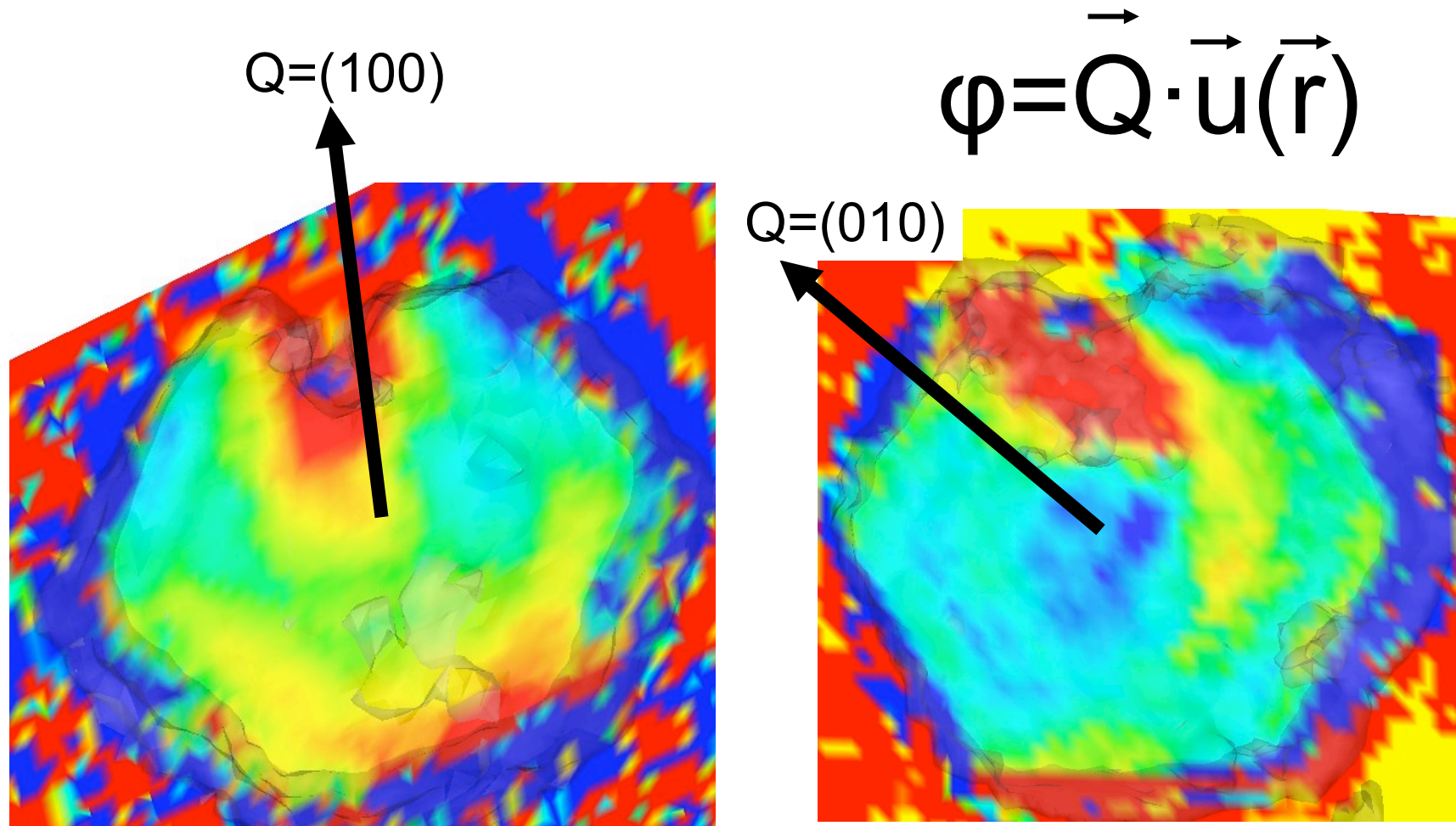
Marcus Newton and Steven Leake (in preparation, 2008)



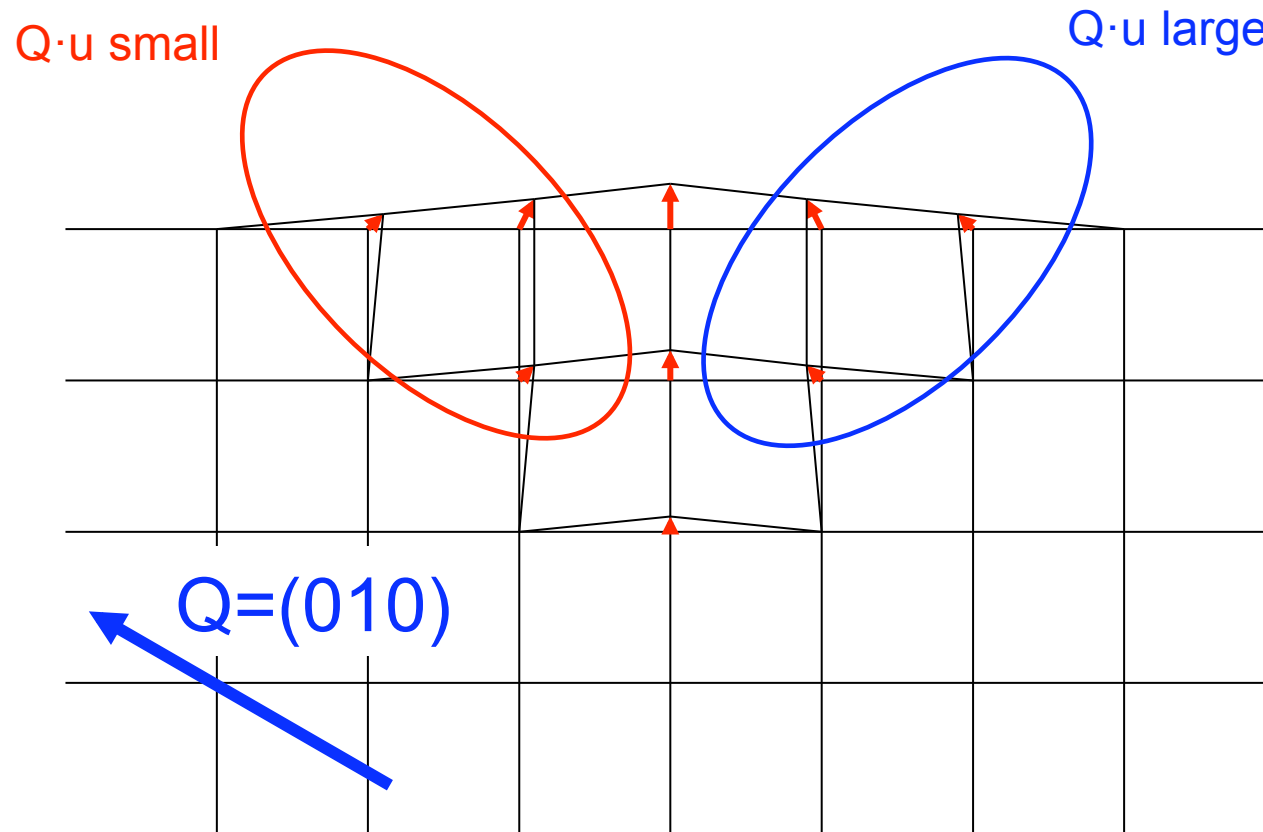
I. K. Robinson, XMNP2009

Phase maps from 2 Bragg peaks

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

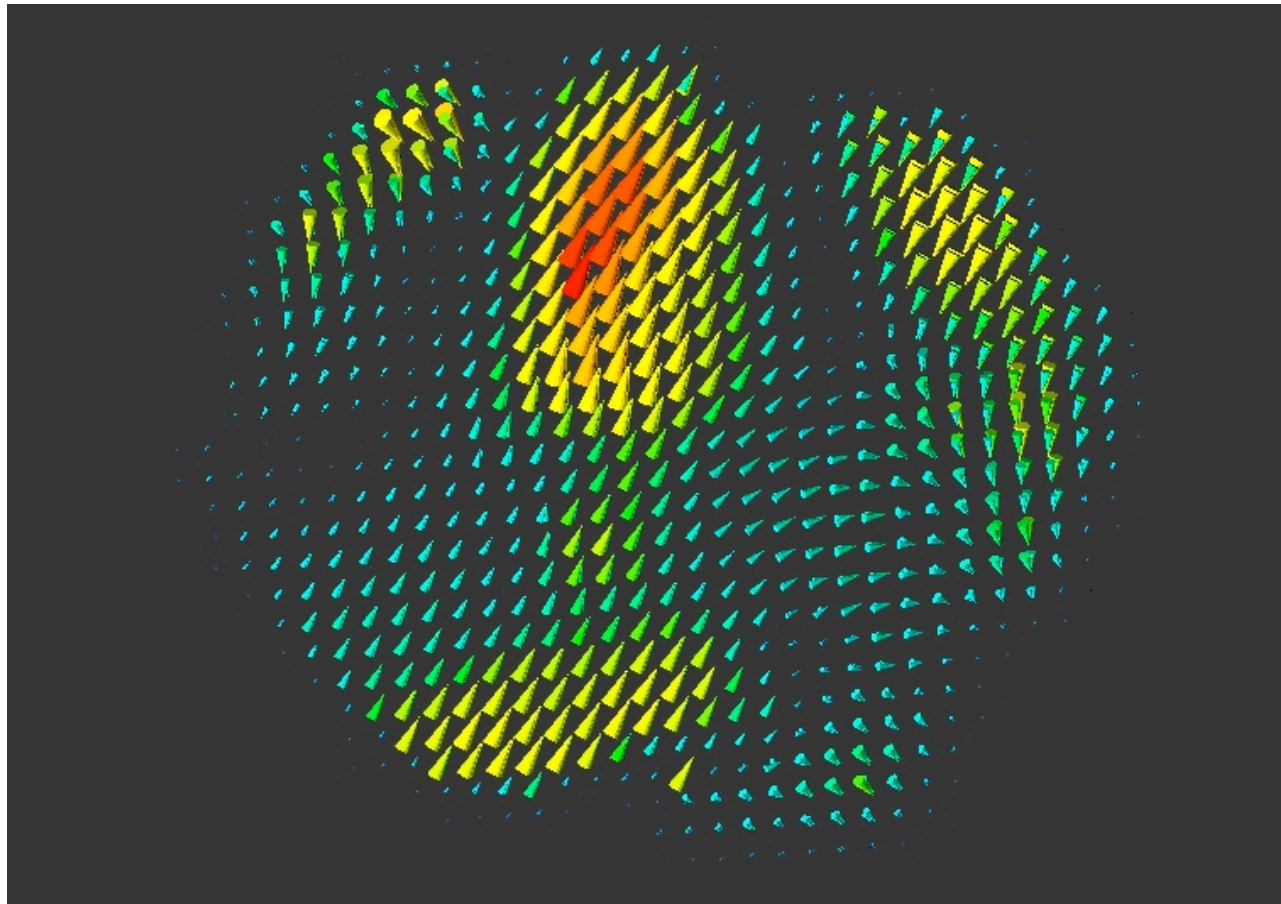


Typical displacement field

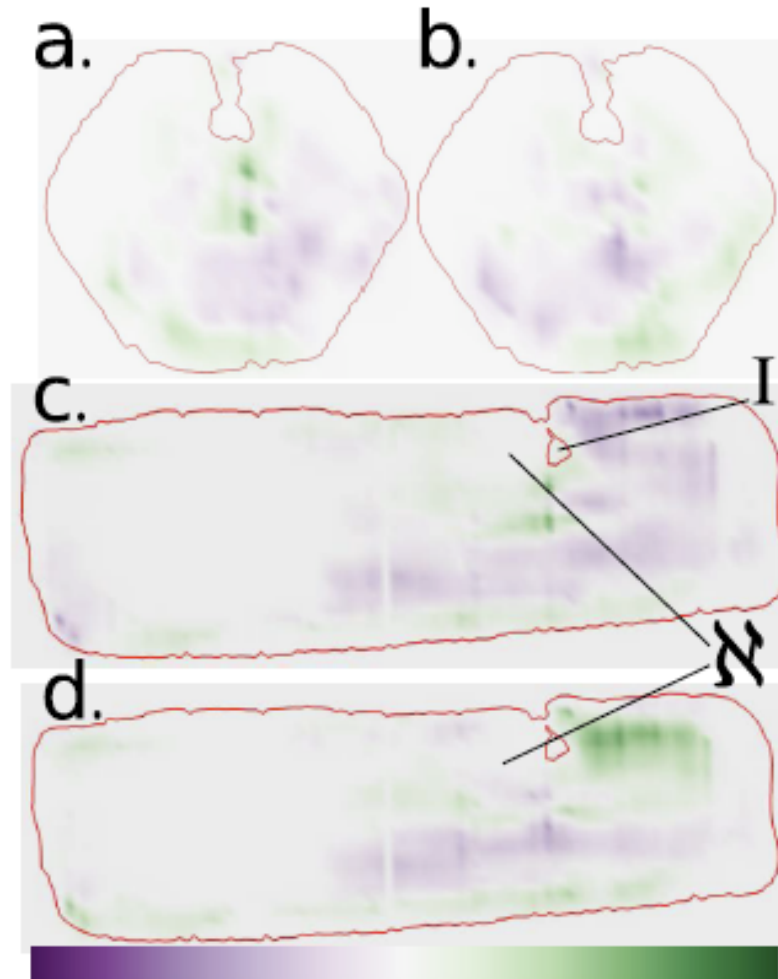
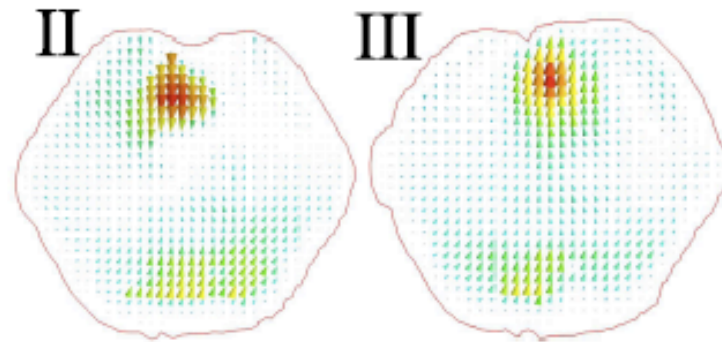
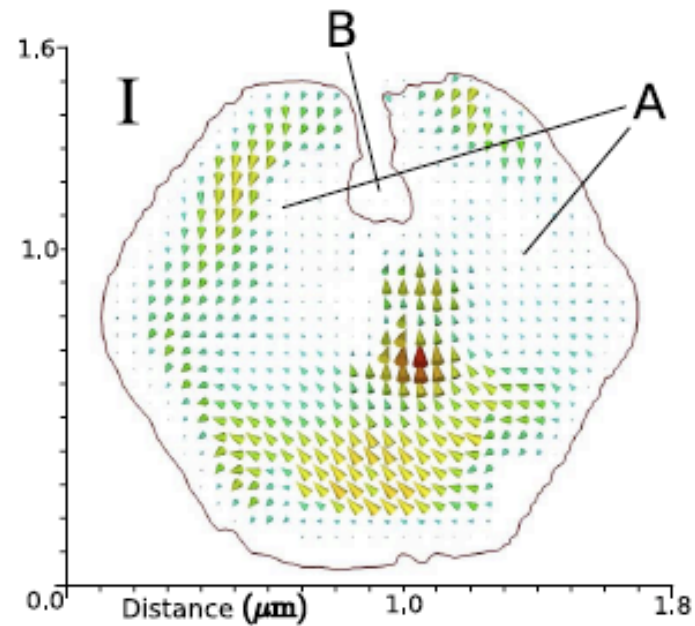


2D vector field of displacements

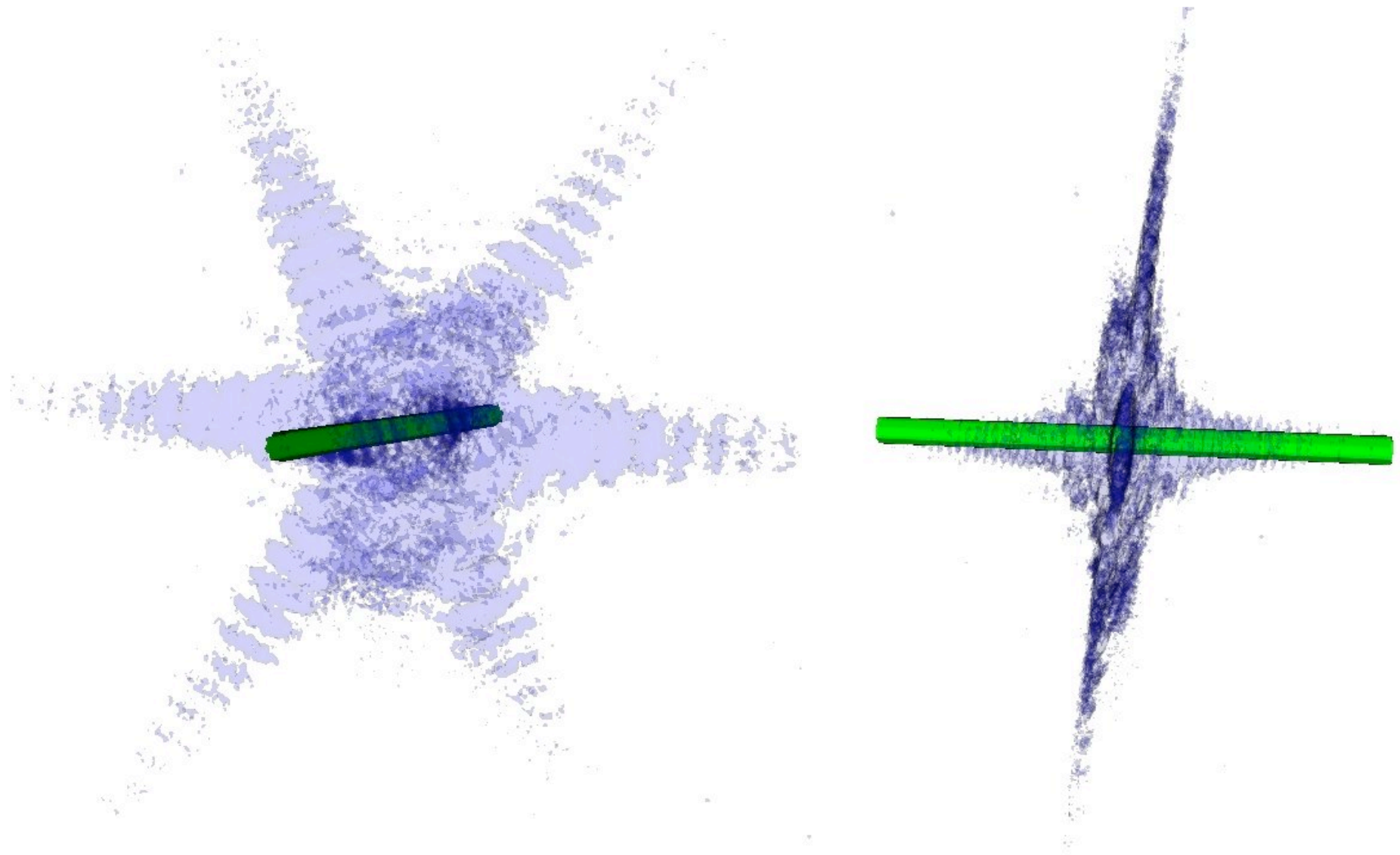
Marcus Newton, UCL, Jan 2009



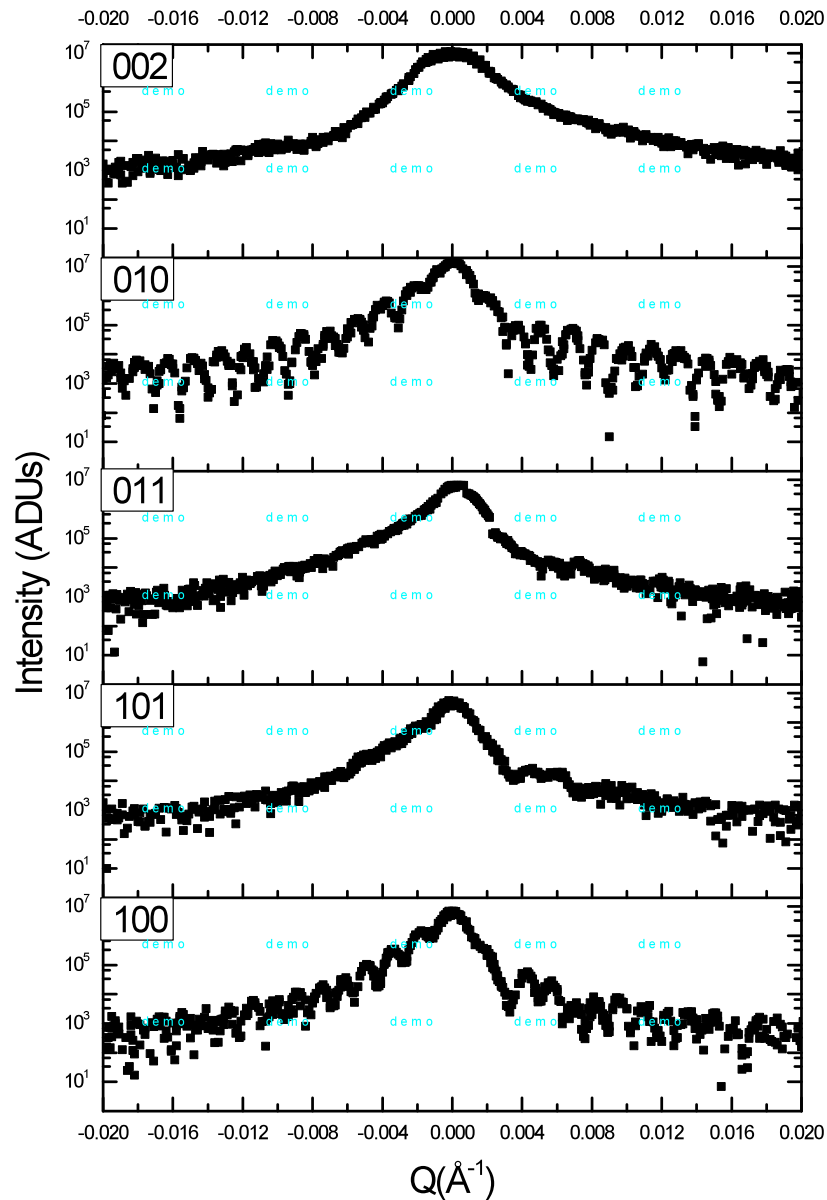
Strain and Rotation fields



How to extract the contrast data



Five Bragg peaks



002 no fringe visibility

010 & 100 good fringe visibility

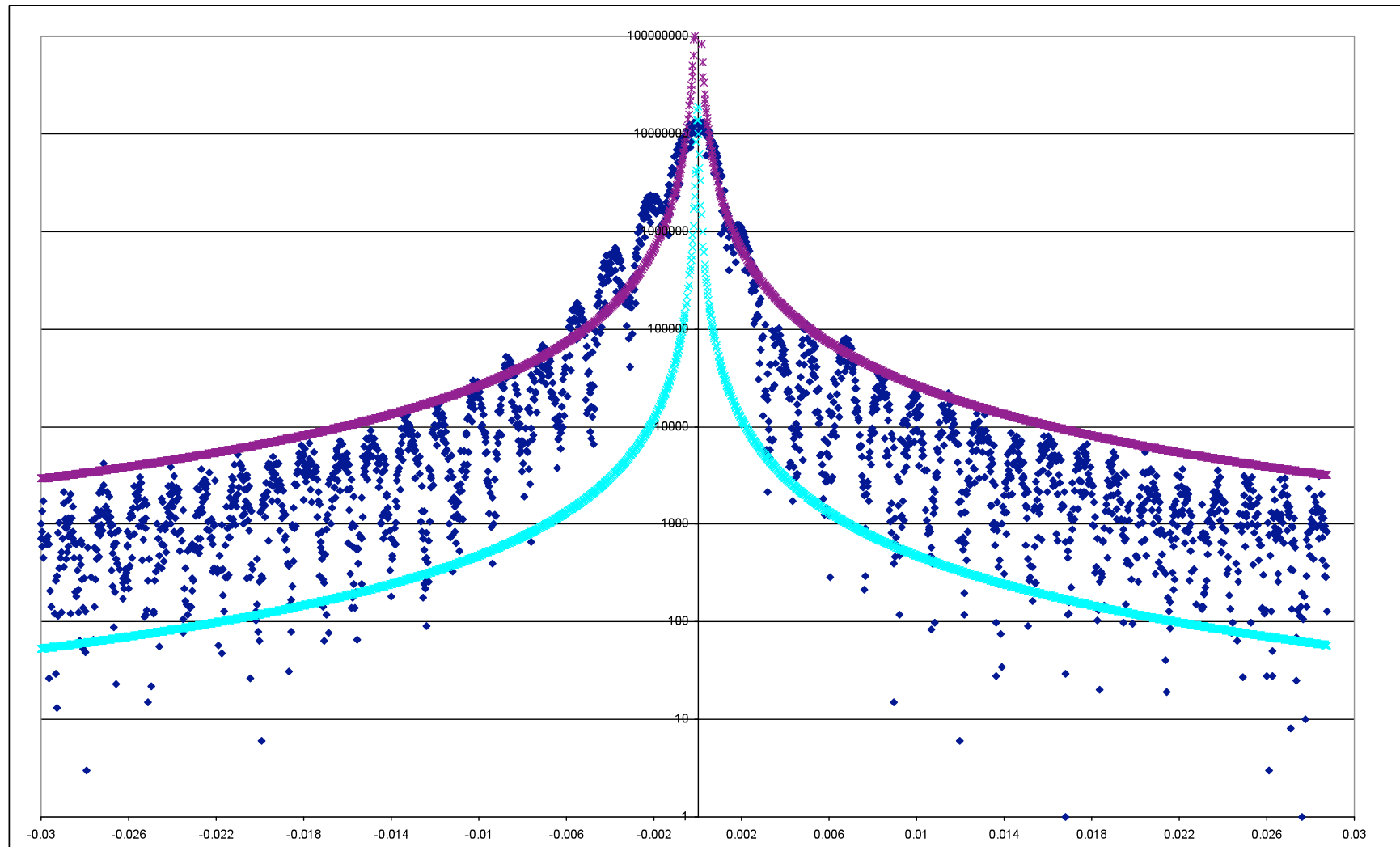
101 & 011 diminished fringe visibility
but fringes still evident

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

Error in coordinate transform ruled out

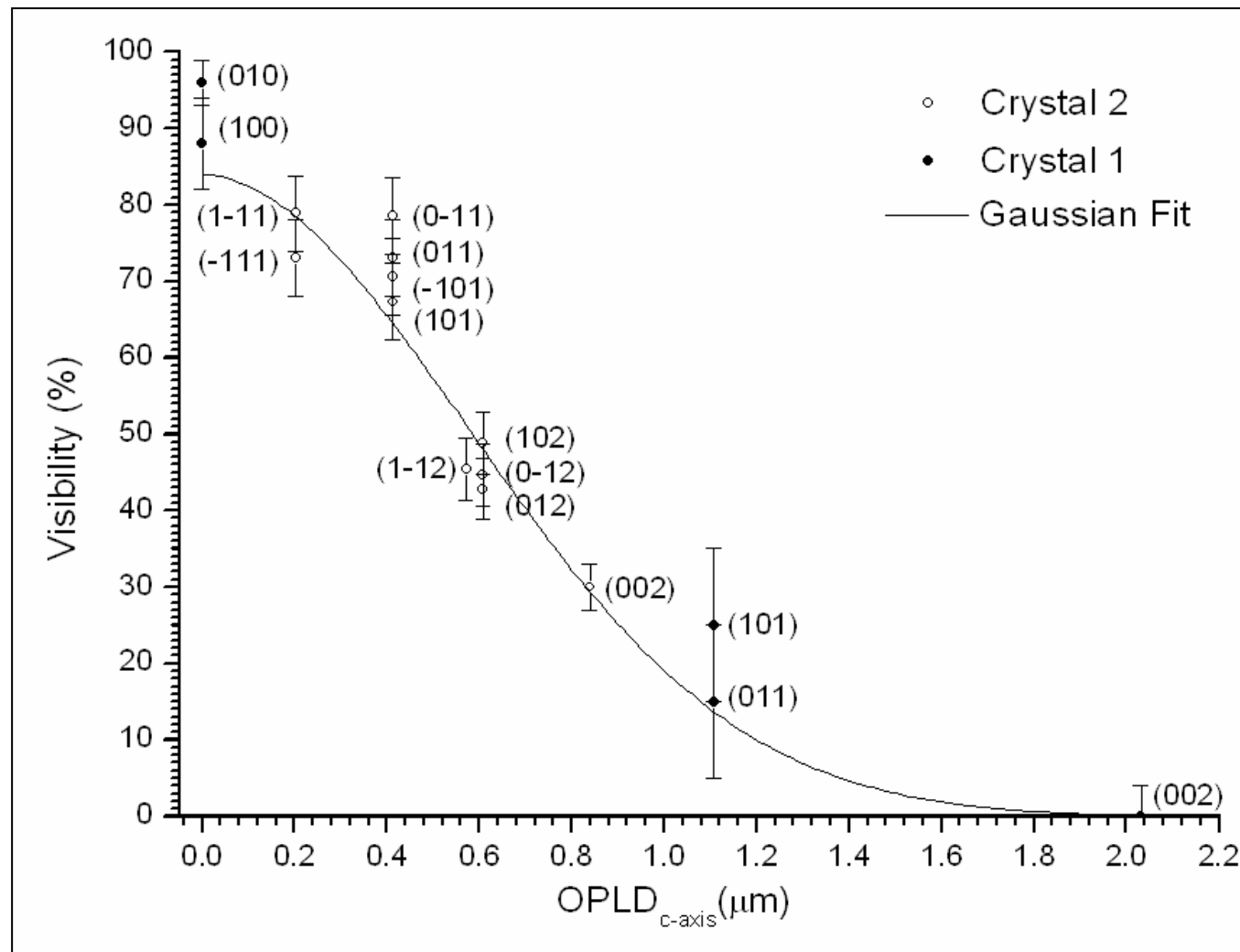
010 and 100 reflections fringe spacing
difference $\sim 20\%$

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



Measured Longitudinal Coherence

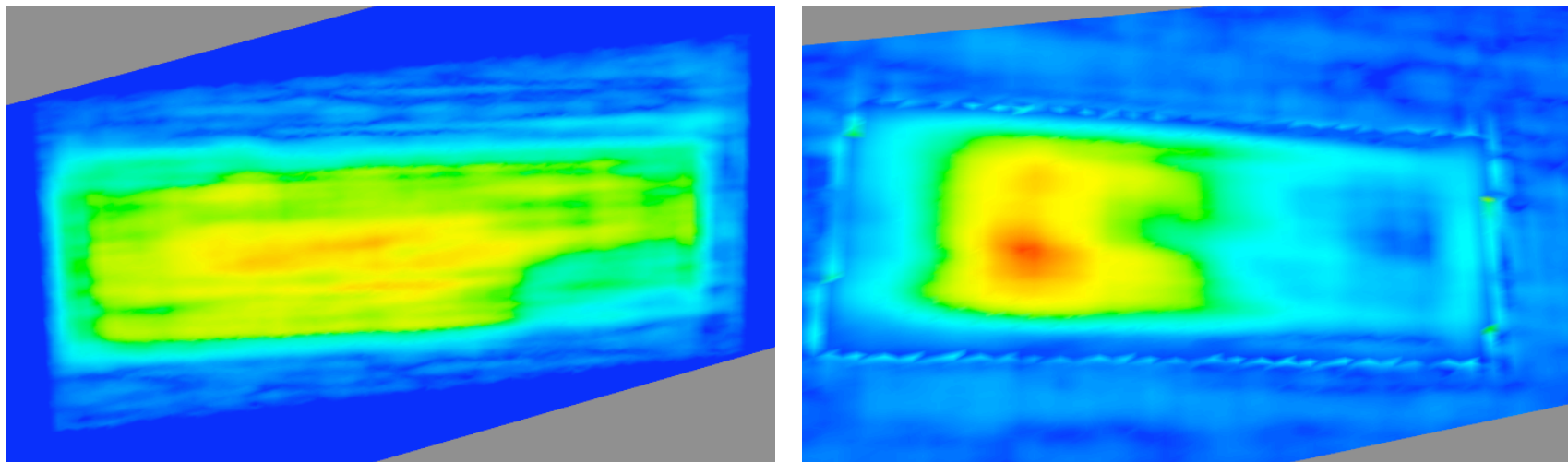
Steven Leake, unpublished, Dec 2008



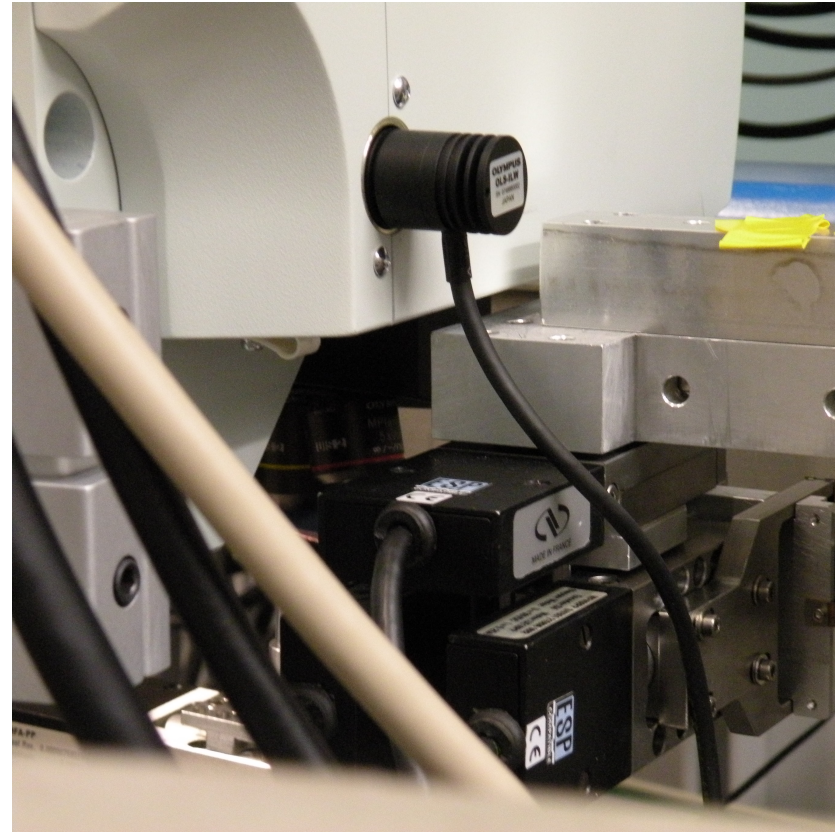
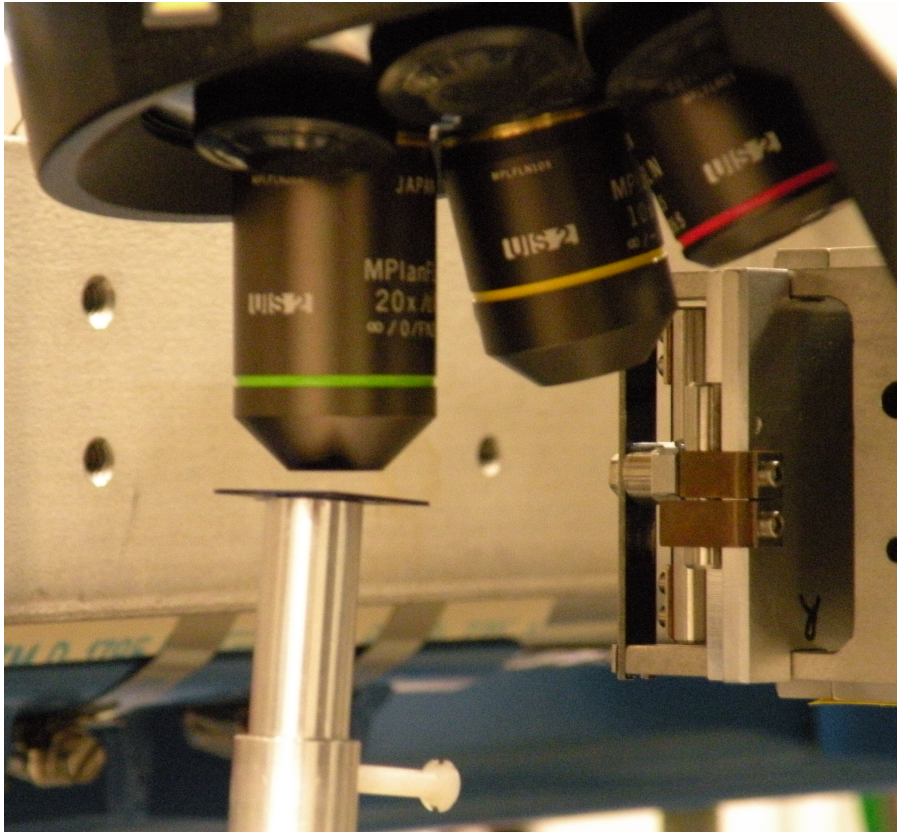
I. K. Robinson, CFEL 2009

Density Section of Reconstruction using 100 and 101 reflections

“hot spot” more developed for limited coherence
Steven Leake, unpublished, Dec 2008



Confocal Alignment Microscope



Conclusions

- Internal structure of Ag and Pb Nanocrystals
- 3D imaging practical for nanocrystals
- Phasing by computation instead of lens
- Strain fields imaged from asymmetric patterns
- Contact Forces cause strain inside crystal
- Surface strain has orientation dependence
- Measurement of coherence function