Coherent X-ray Diffraction imaging of strain fields in Nanocrystals

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East Midlands Materials Society,
Leicester, February 3, 2011

Outline

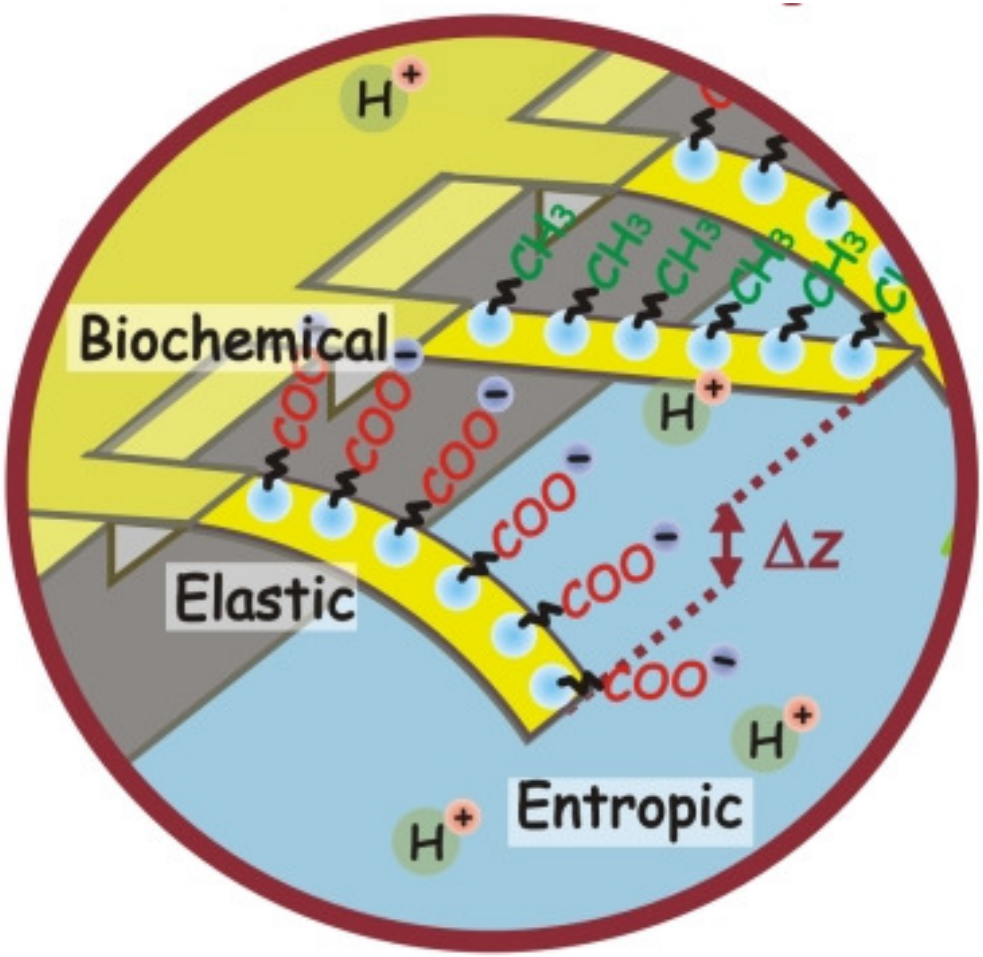
- Coherent x-ray diffraction
- CXD can solve the phase problem
- Nanocrystal structures
- Crystal strain as complex density
- Nanowire structures
- Full strain tensor

London Centre for Nanotechnology

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

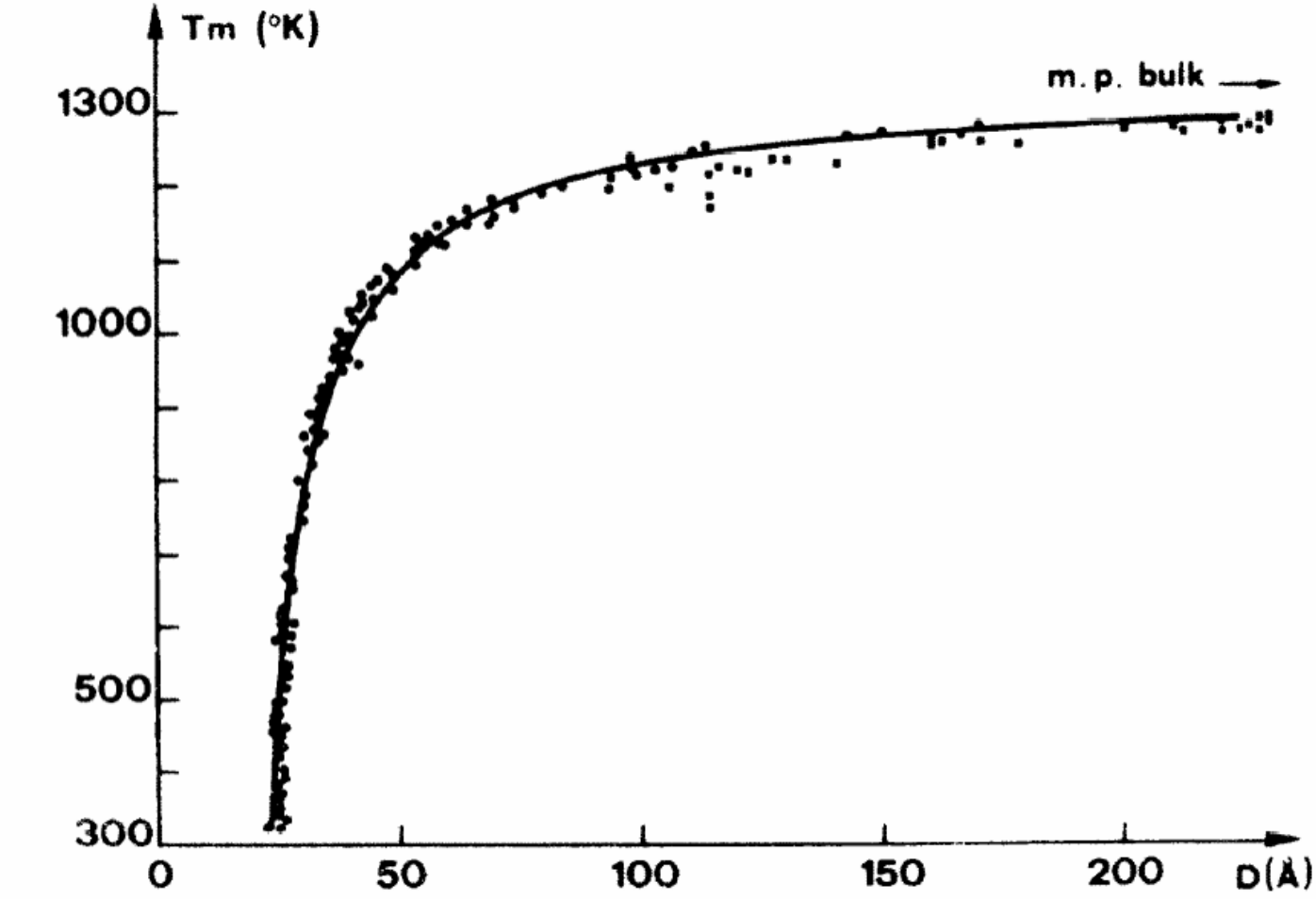
Nanocantilevers

Dr Rachel McKendrie

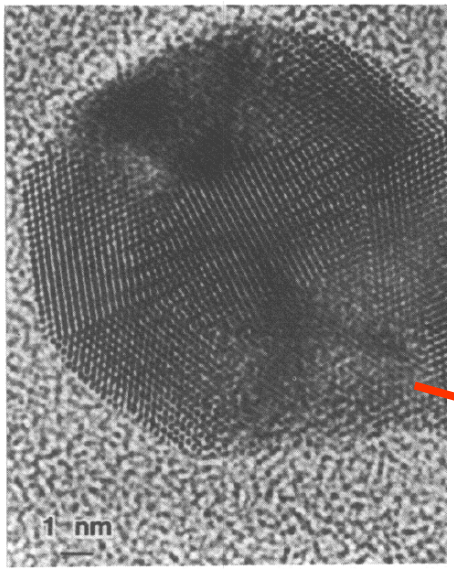


Size-dependent Melting of Au Particles

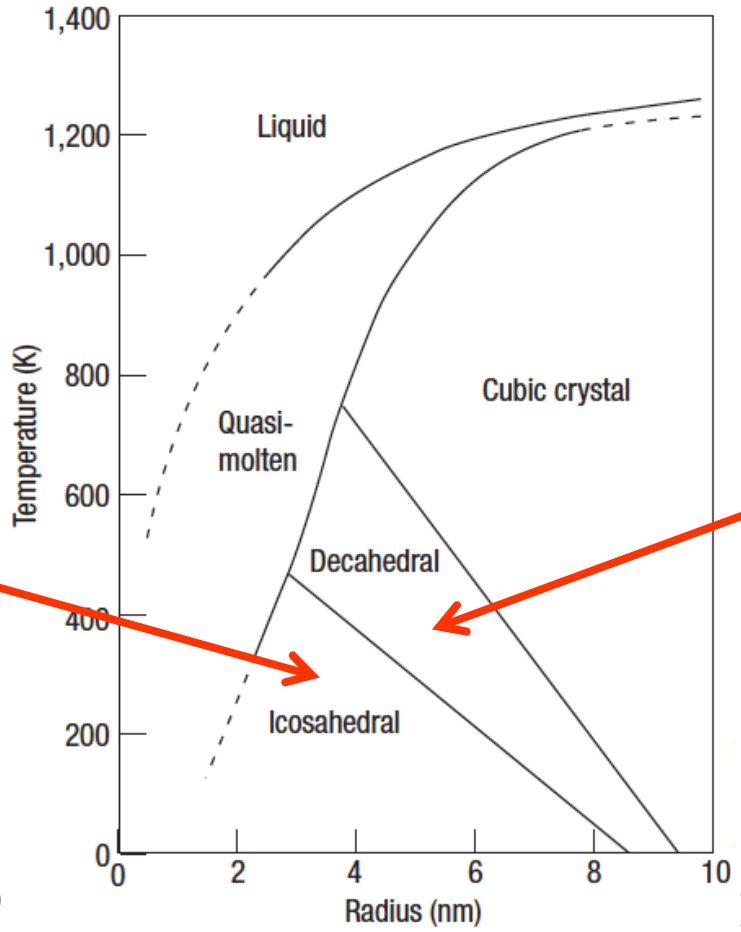
P. Buffat and J-P. Borel, Phys. Rev. A 2287-97 (1975)

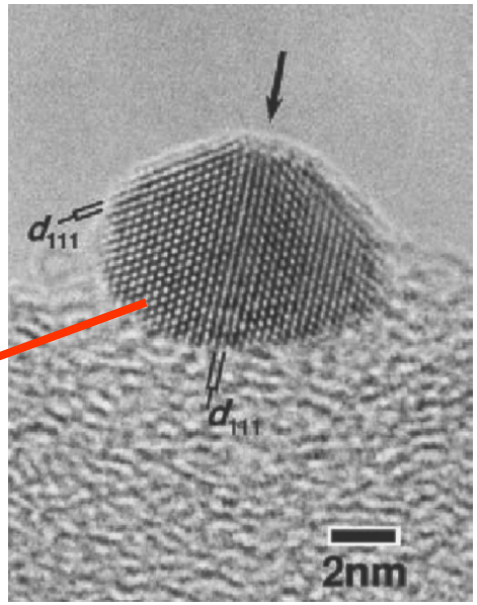


Structure of Gold vs Size



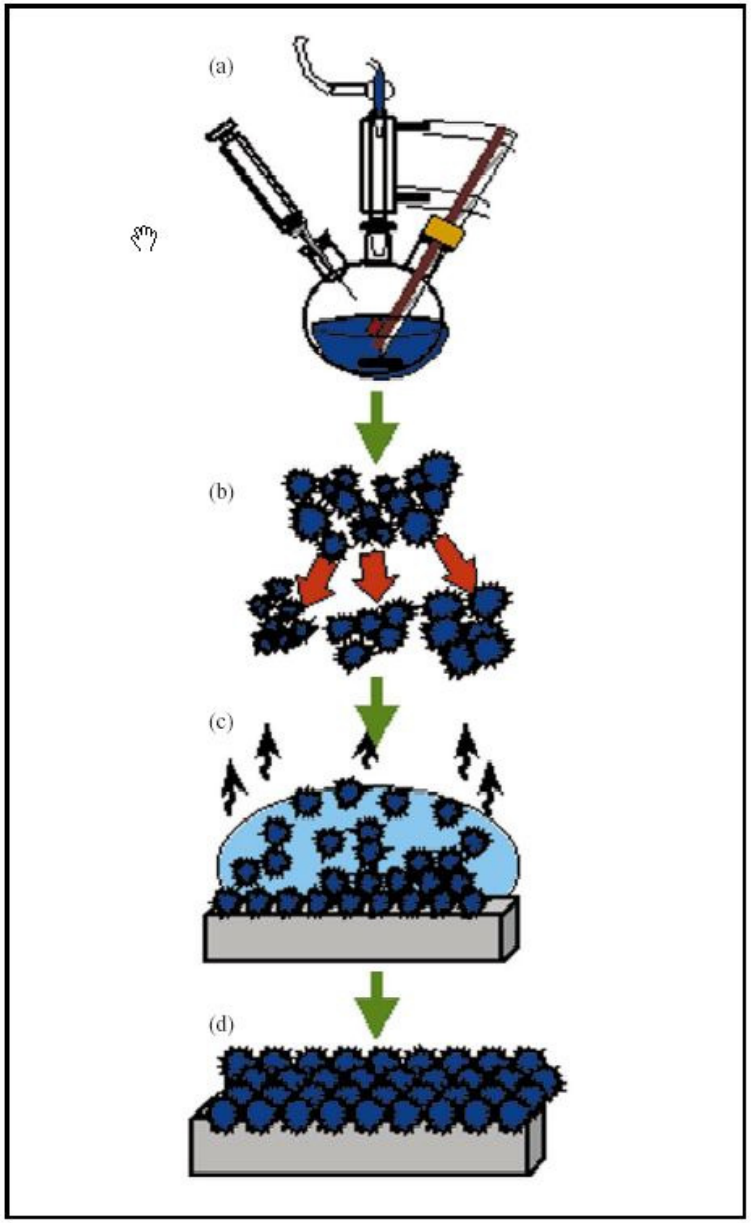
L. D. Marks, RPP (1994)





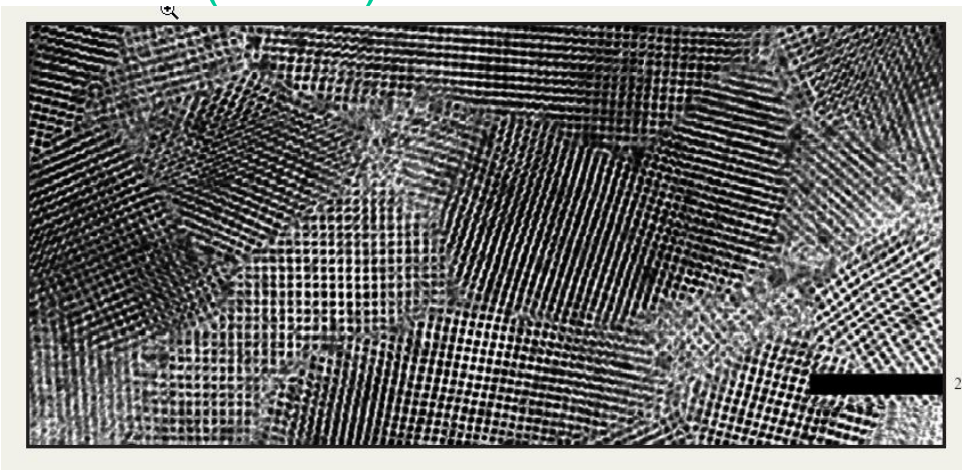
Koga and Sugawara (2003)

Chemical Synthesis of Nanocrystals



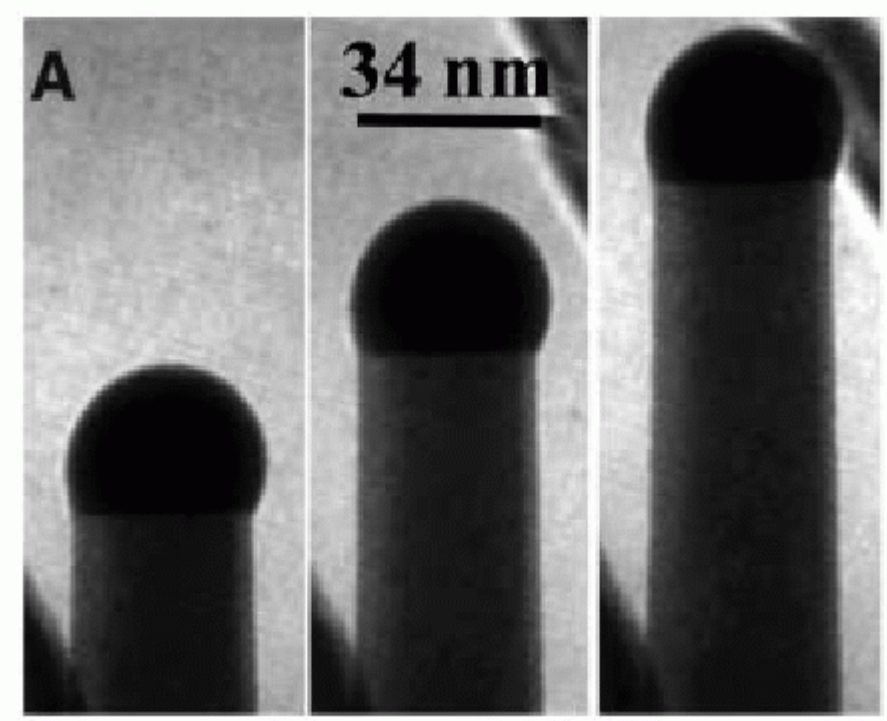
- Reactants introduced rapidly
- High temperature solvent
- Surfactant/organic capping agent
- Square superlattice (200nm scale)

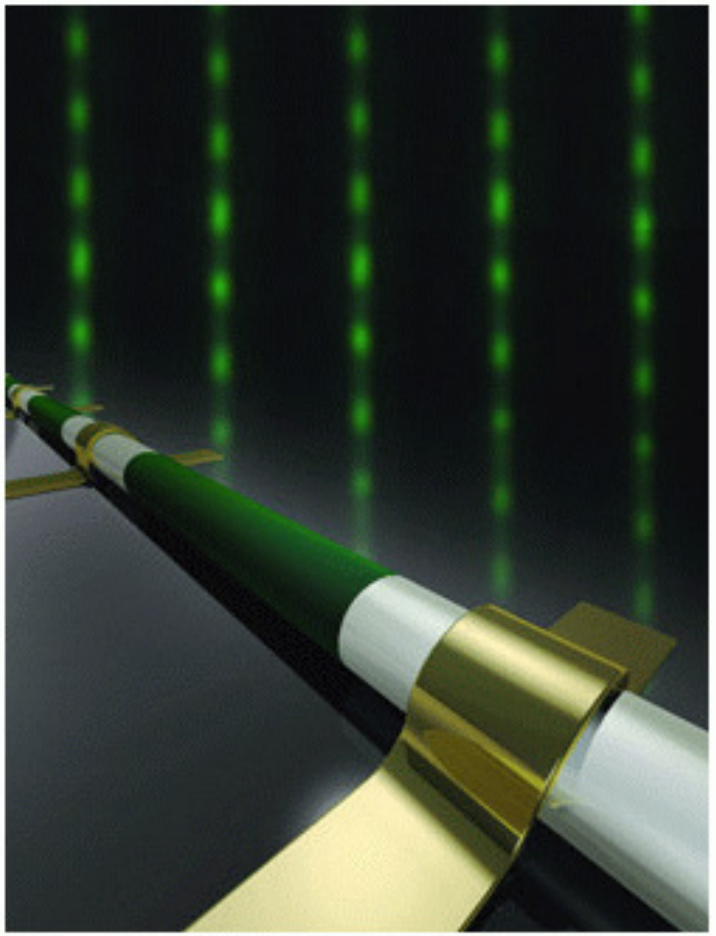
C. B. Murray, IBM J. Res. & Dev. **45** 47 (2001)



VLS growth of nanowires

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

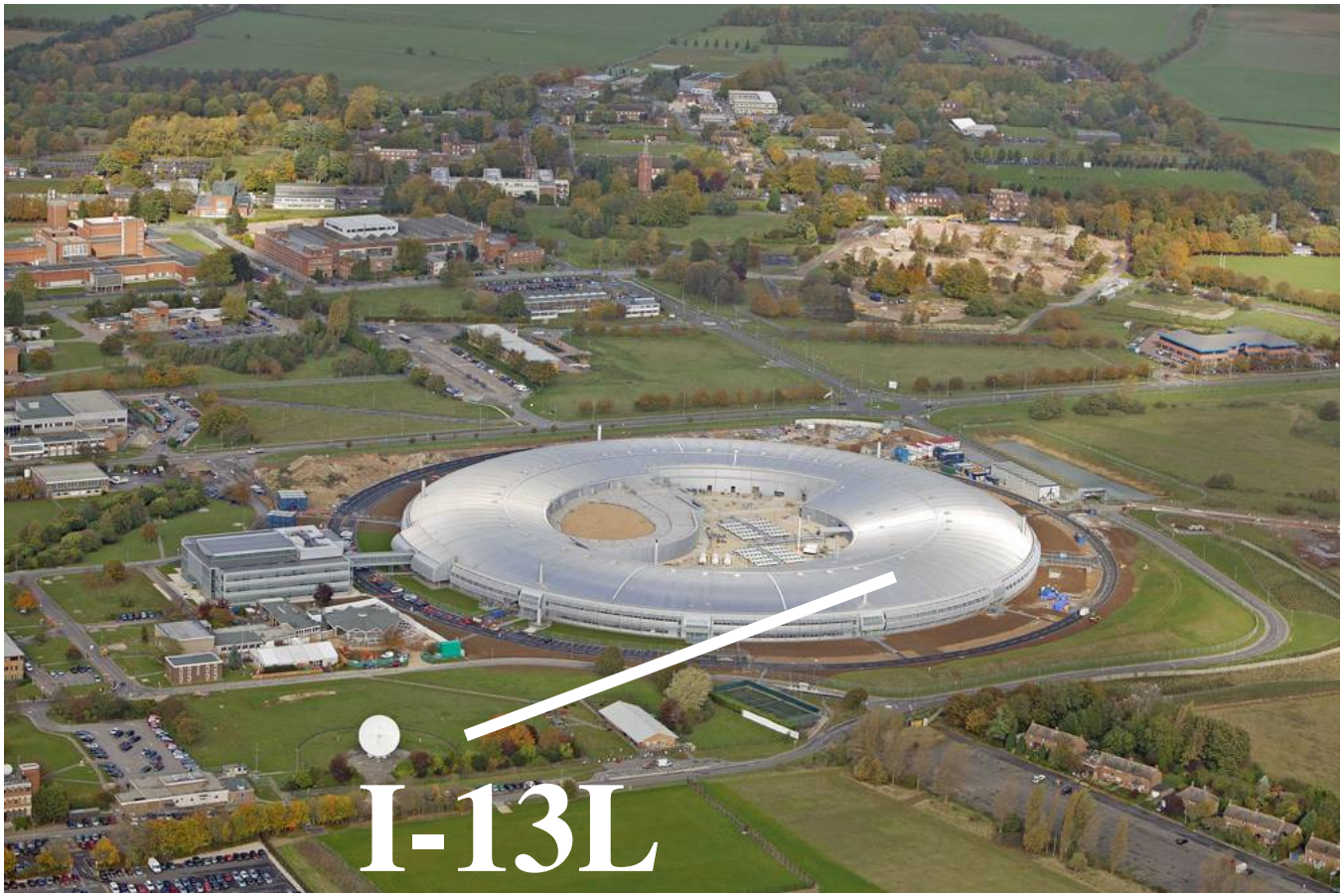




I. K. Robinson, EMM

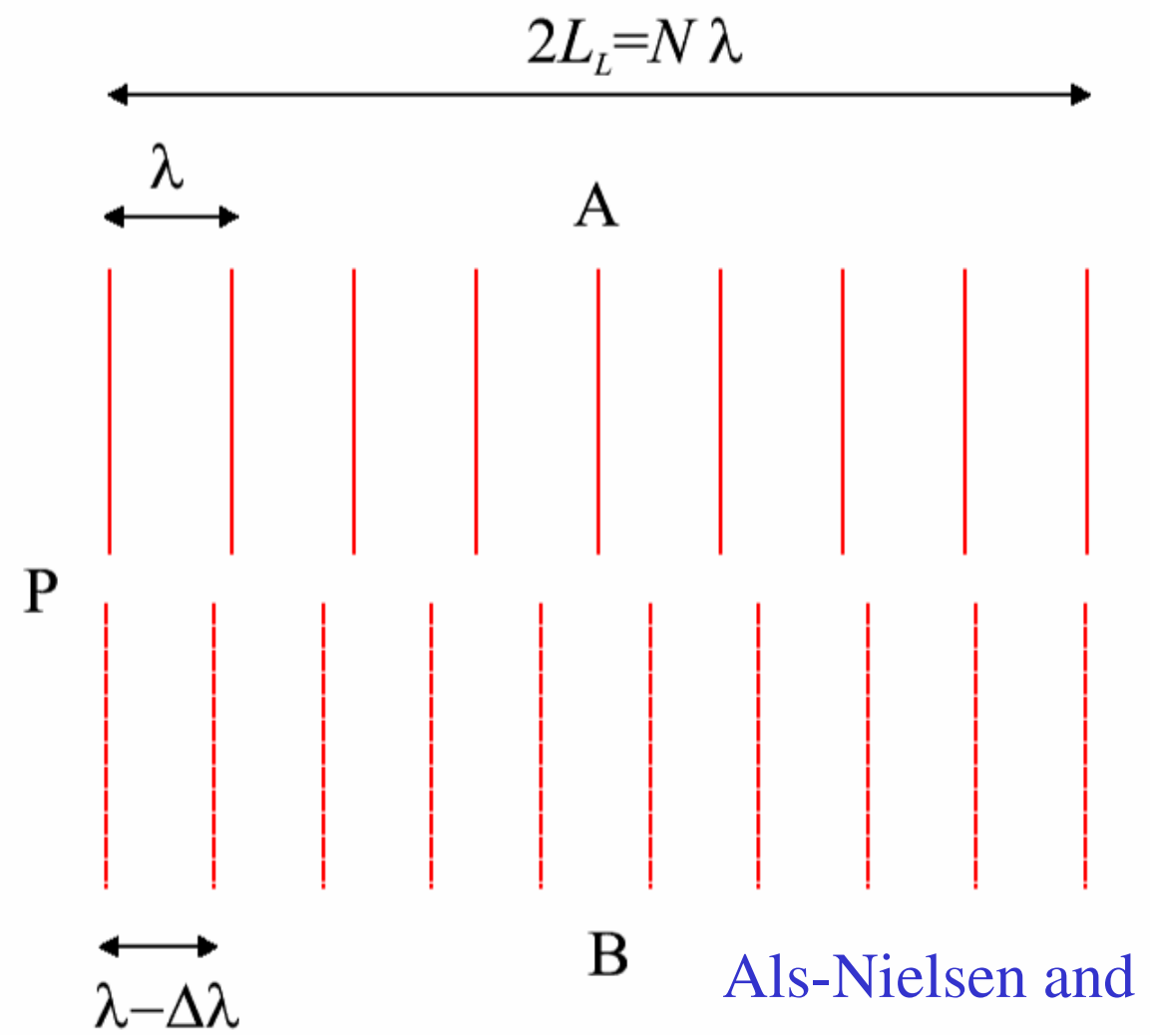
NiSi/Si nanowire heterostructure devices. *Nature* **430**, 61 (2004).

Diamond Light Source (RAL)



Diamond in-vacuum X-ray Undulator

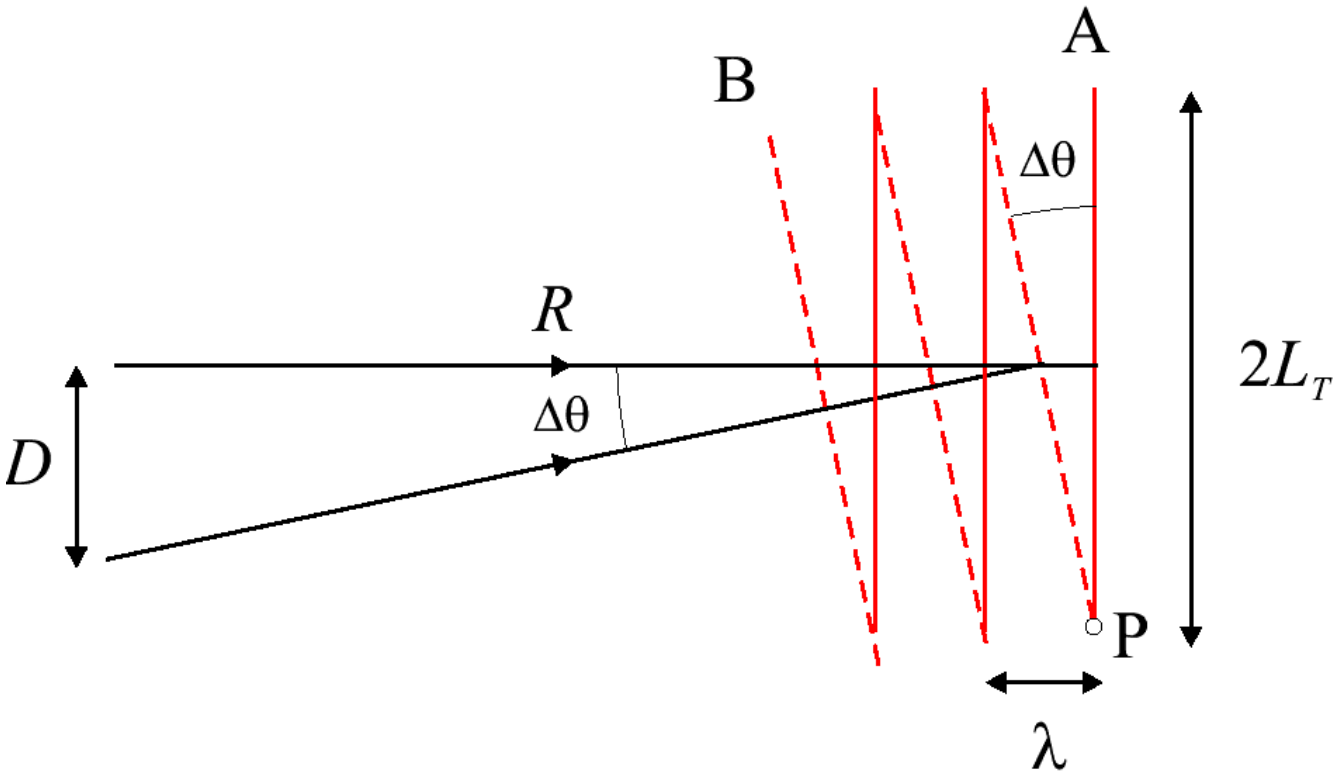
Longitudinal Coherence



$$L_L = \frac{1}{2}\frac{\lambda^2}{\Delta\lambda}$$

Als-Nielsen and McMorrow (2001)

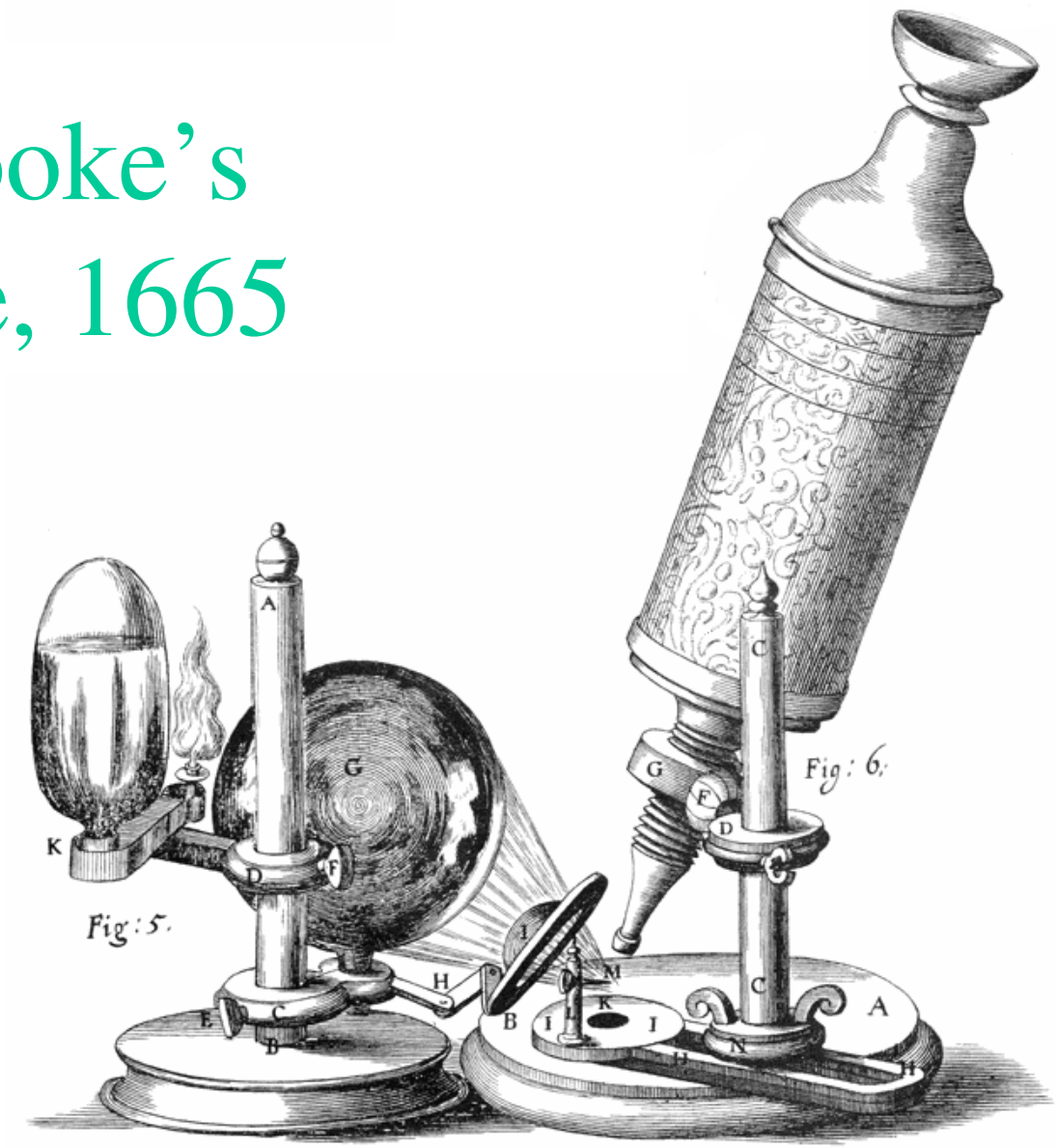
Lateral (Transverse) Coherence



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

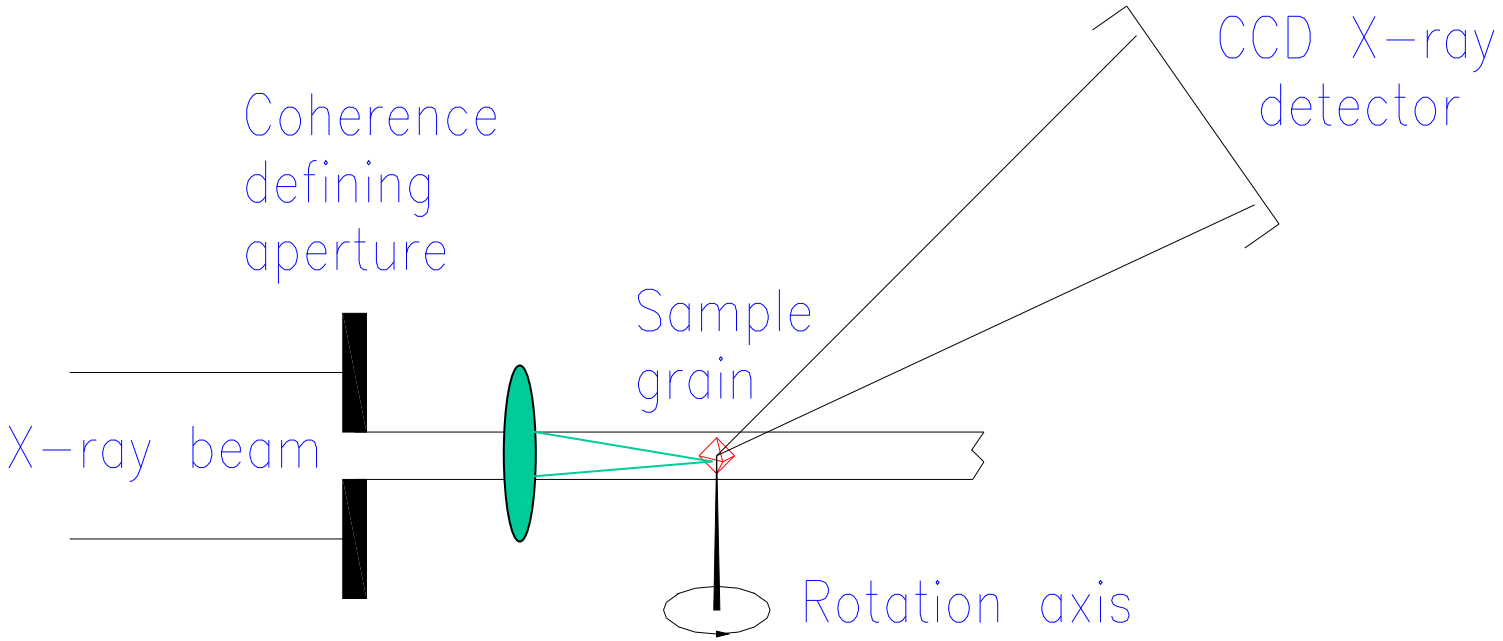
Als-Nielsen and McMorrow (2001)

Robert Hooke's microscope, 1665

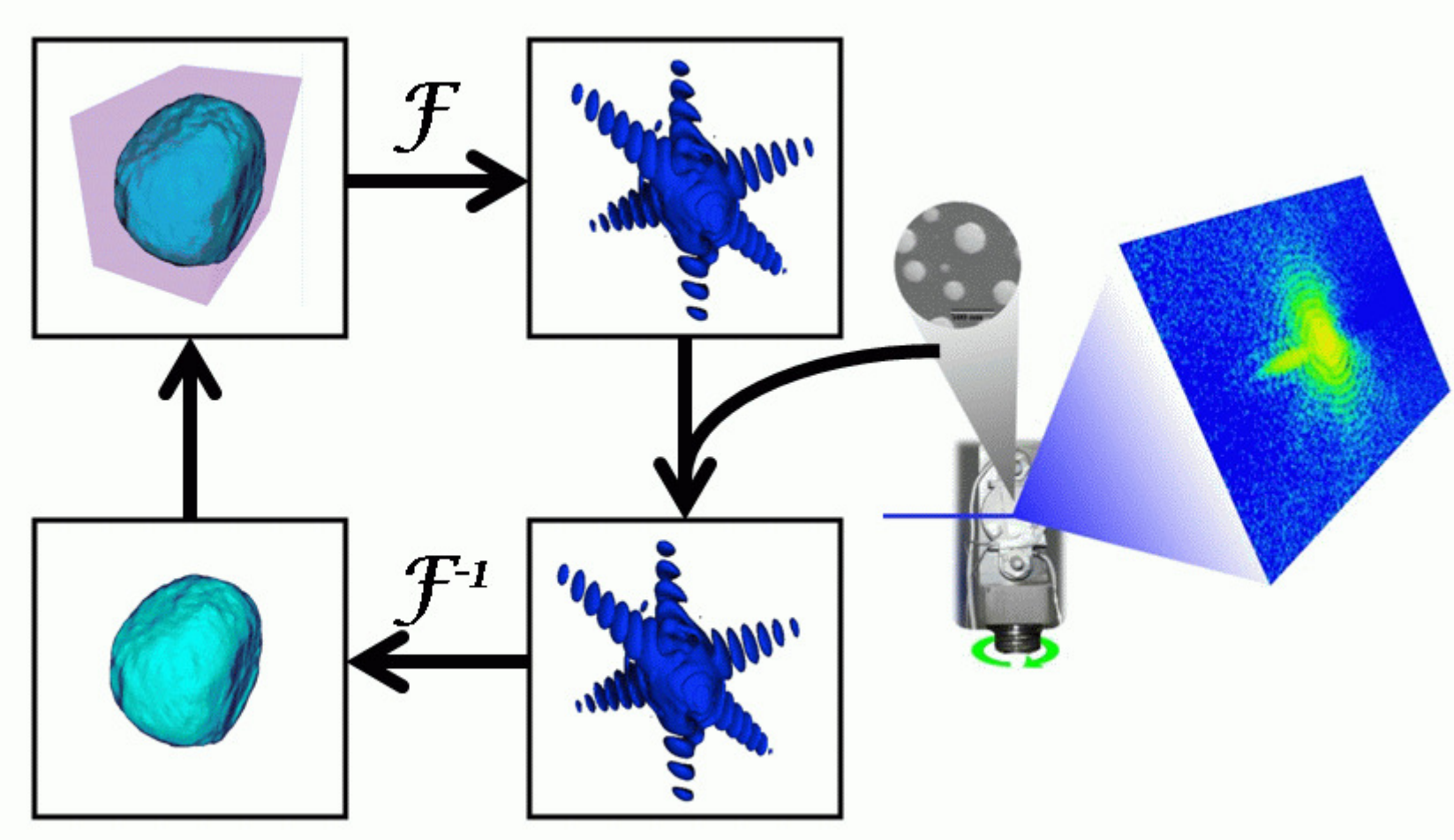


National Museum of Health and Medicine, Washington DC

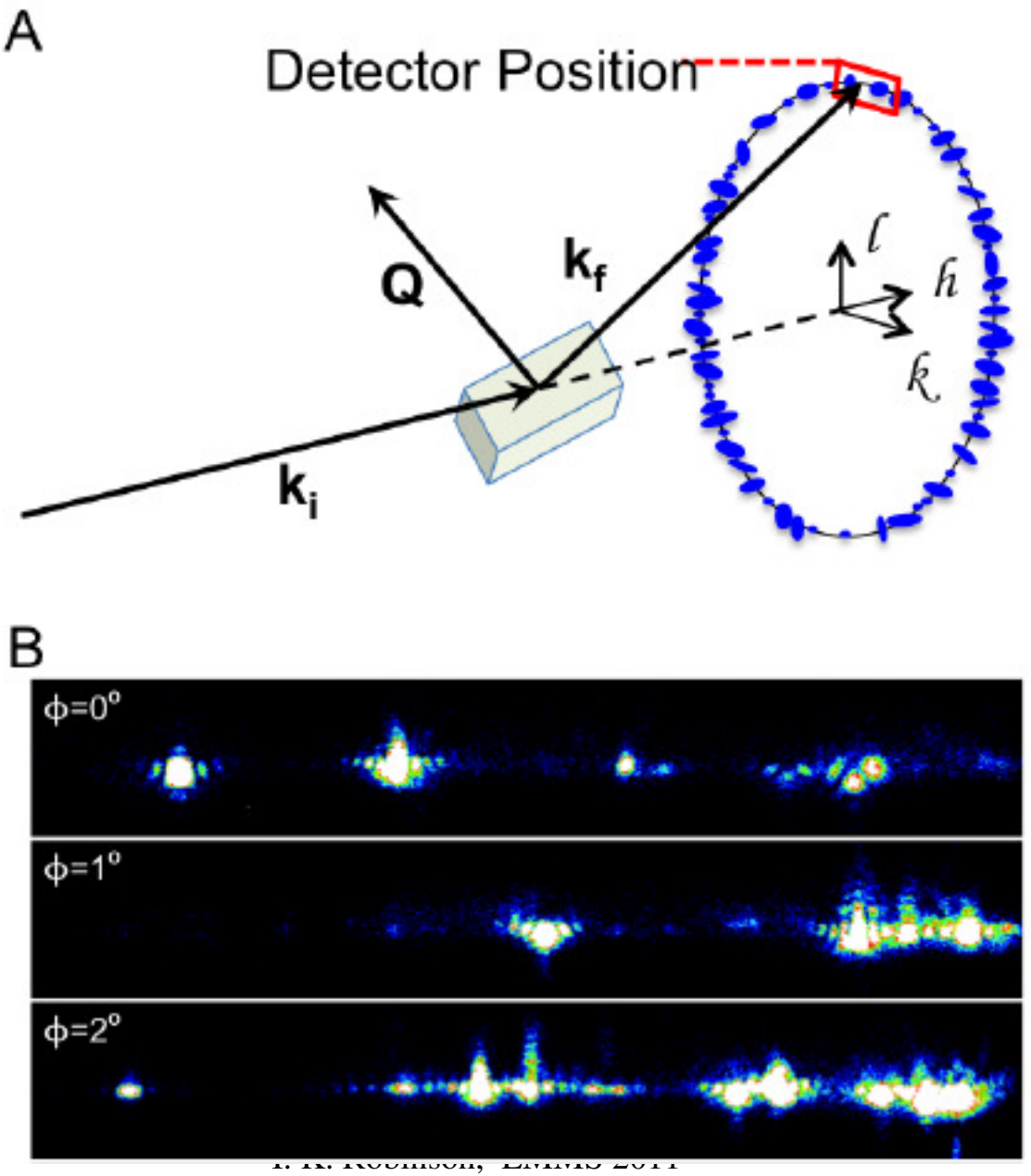
Lensless X-ray Microscope, 2003



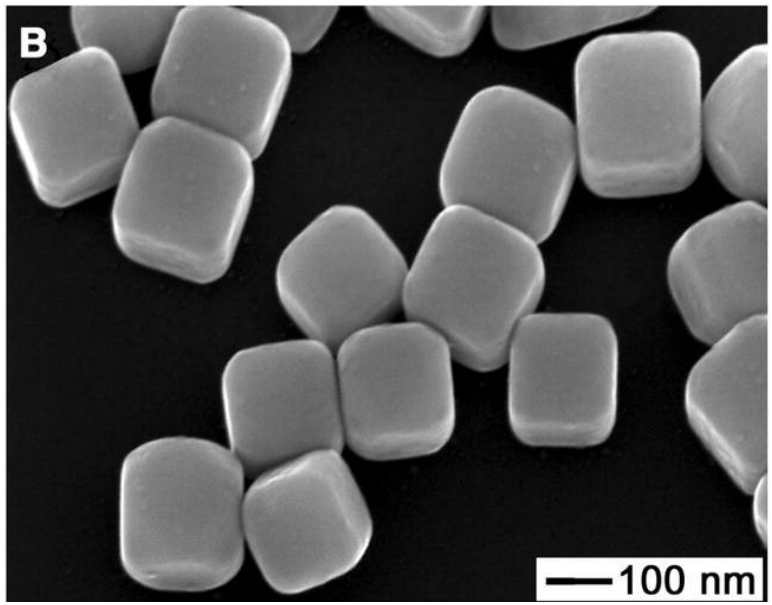
Generic "Error Reduction" method

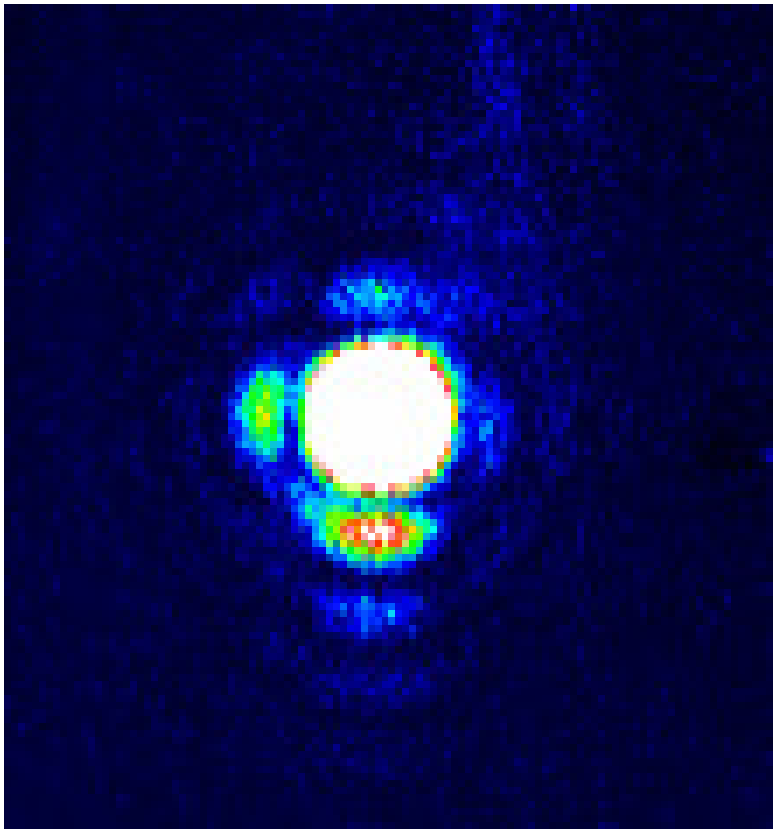


J. R. Fienup Appl. Opt. 21 2758 (1982)
R. W. Gerchberg and W. O. Saxton Optik 35 237 (1972)


A
Detector Position
Q
k_f
k_i
l
h
k
B
ϕ=0°
ϕ=1°
ϕ=2°

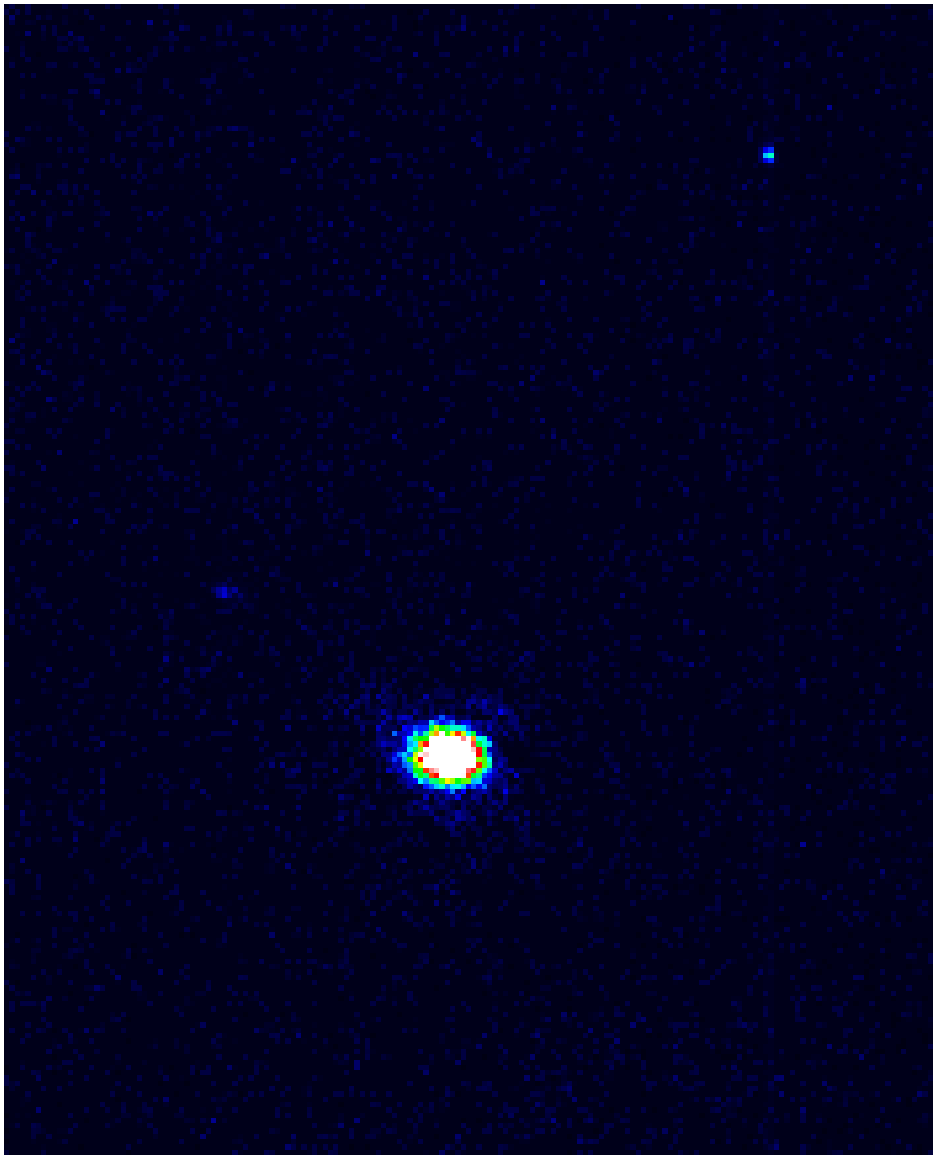
Chemically Synthesized Silver Nanocubes

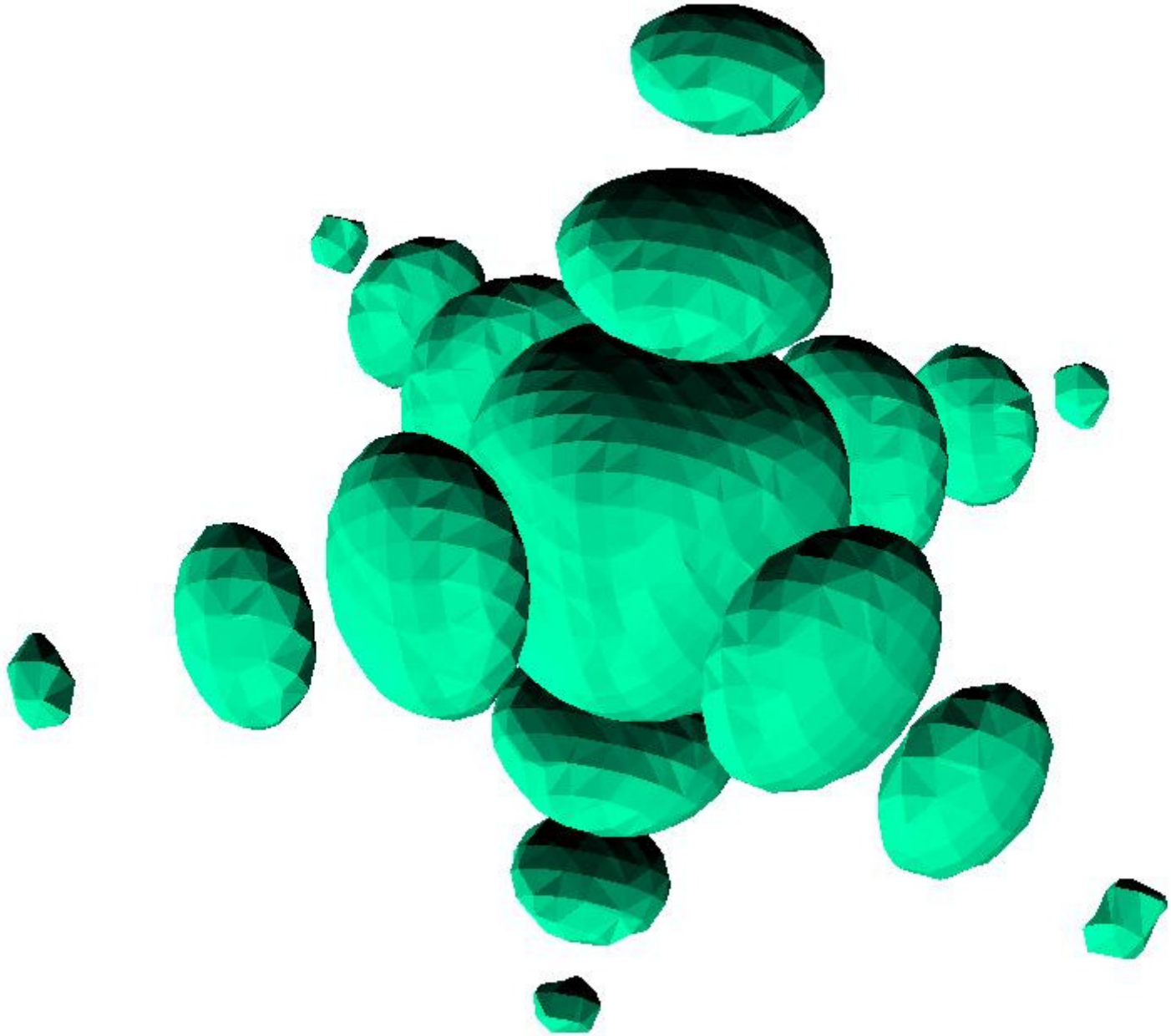


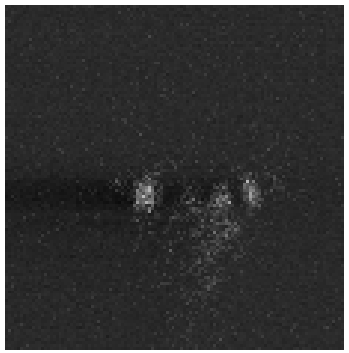


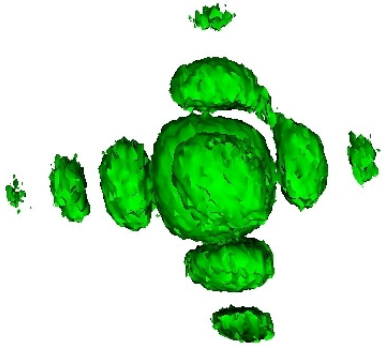
Yugang Sun and Younan Xia,
Science 298 2177 (2003)

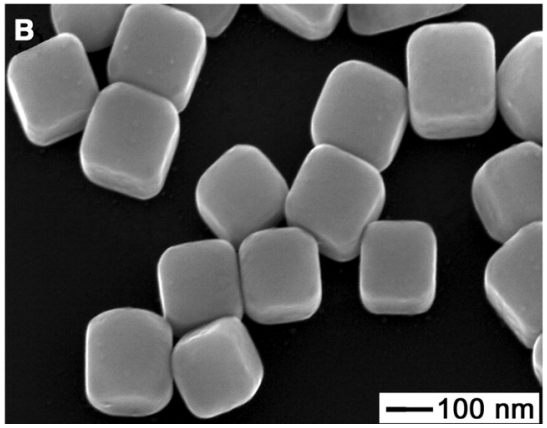
Rocking scan of Ag cubes with 0.01° steps

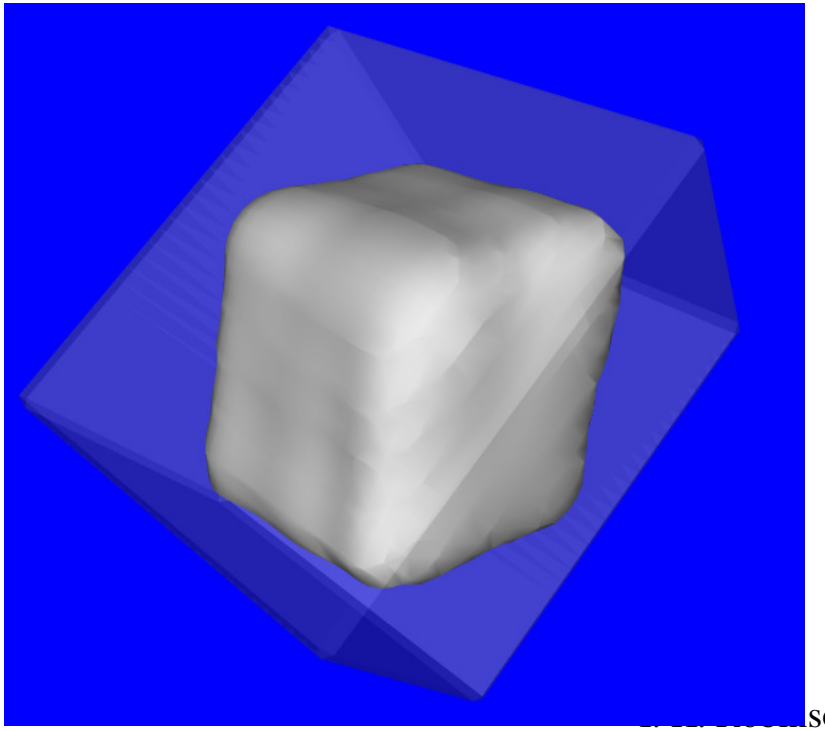


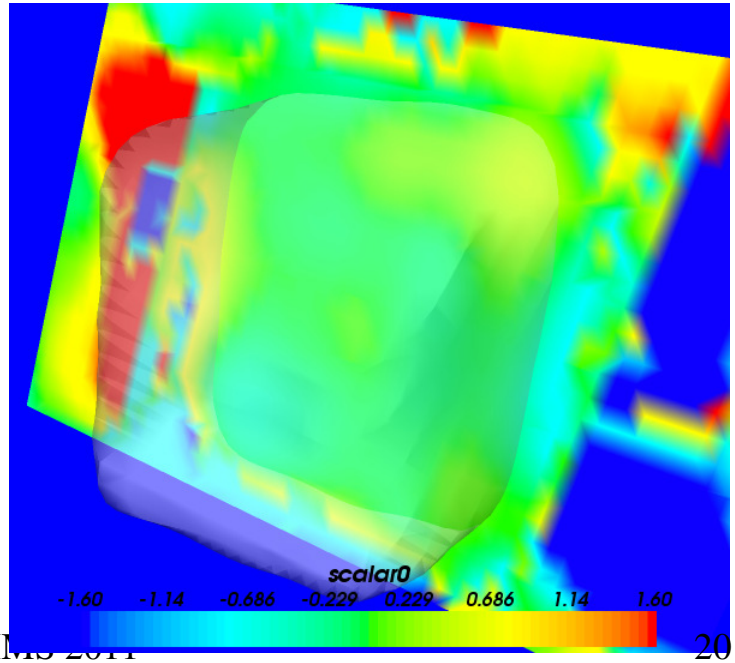




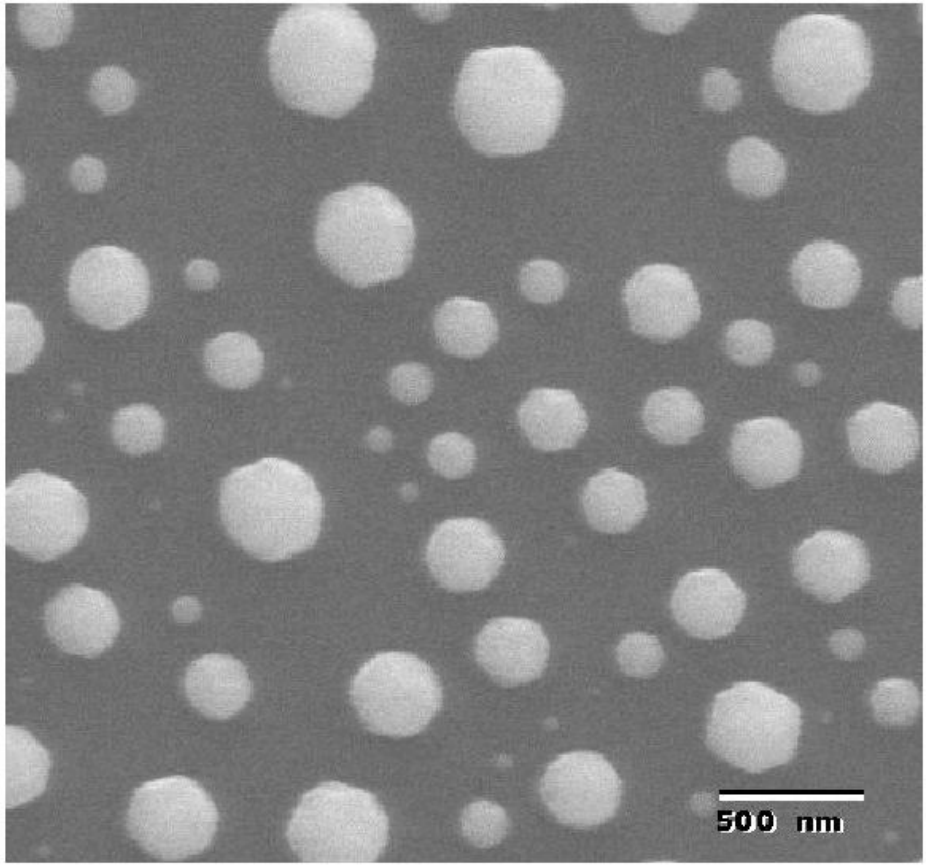



B
100 nm

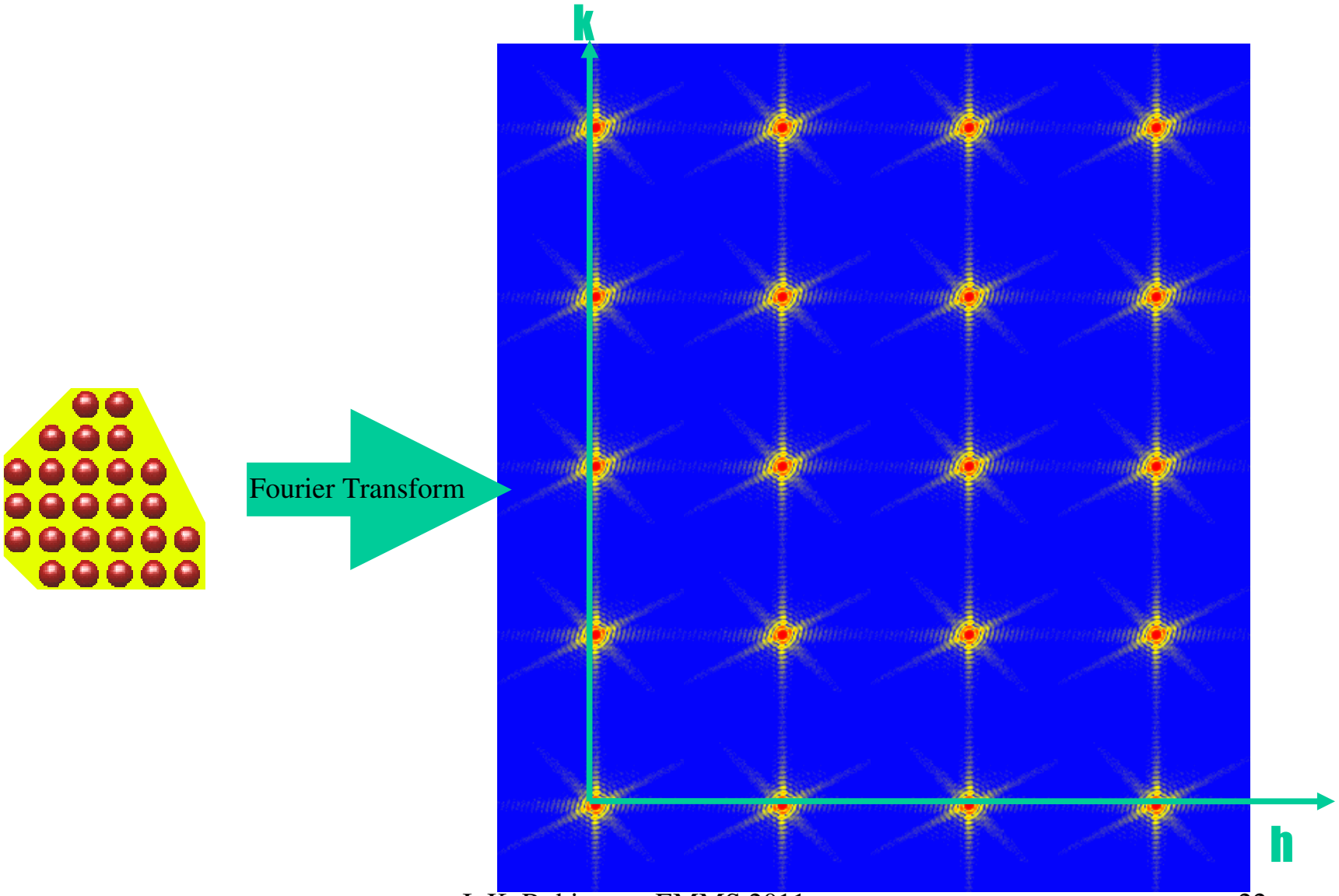



scalar0
-1.60 -1.14 -0.686 -0.229 0.229 0.686 1.14 1.60

In situ growth of Pb crystals

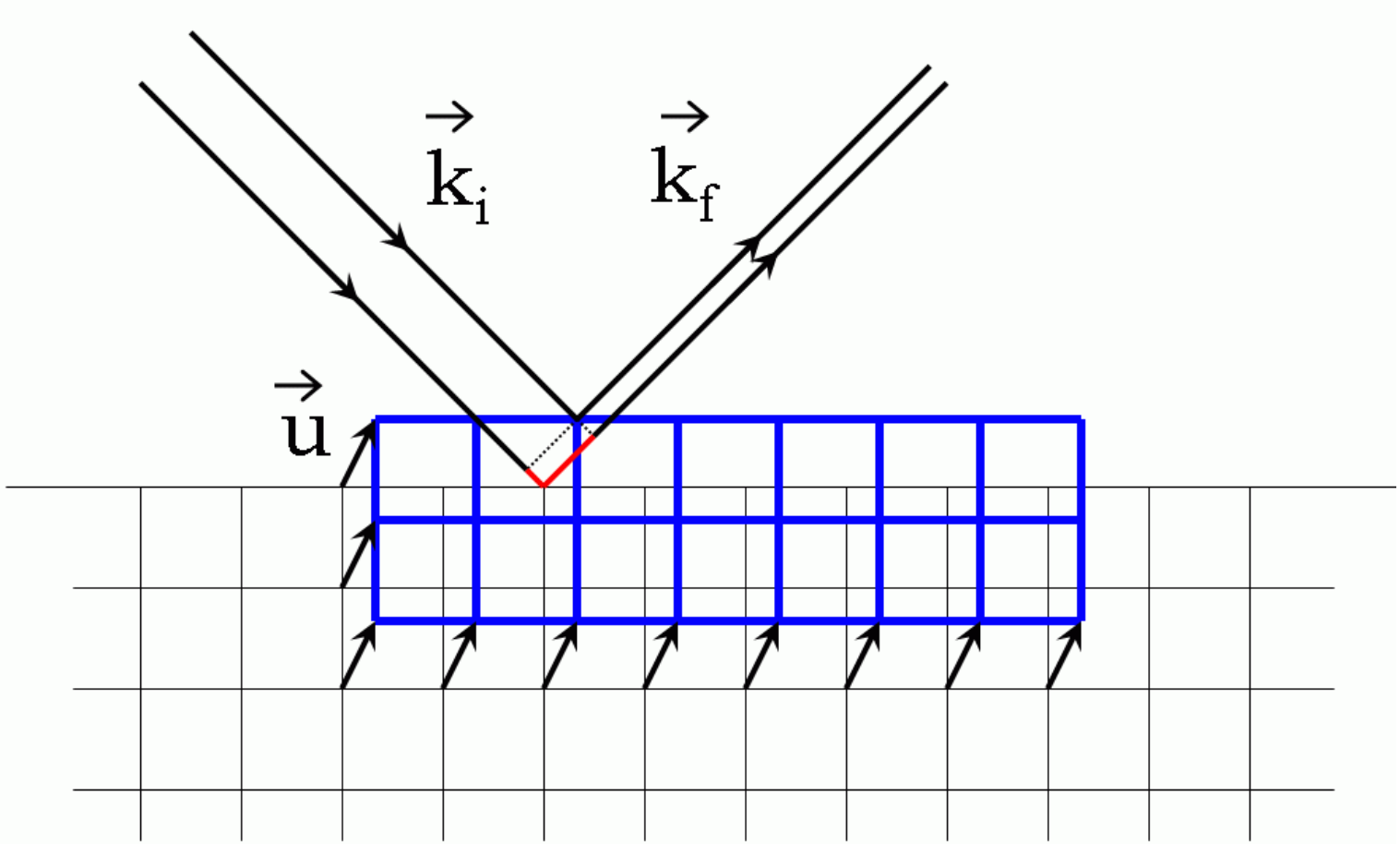


Coherent Diffraction from Crystals



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

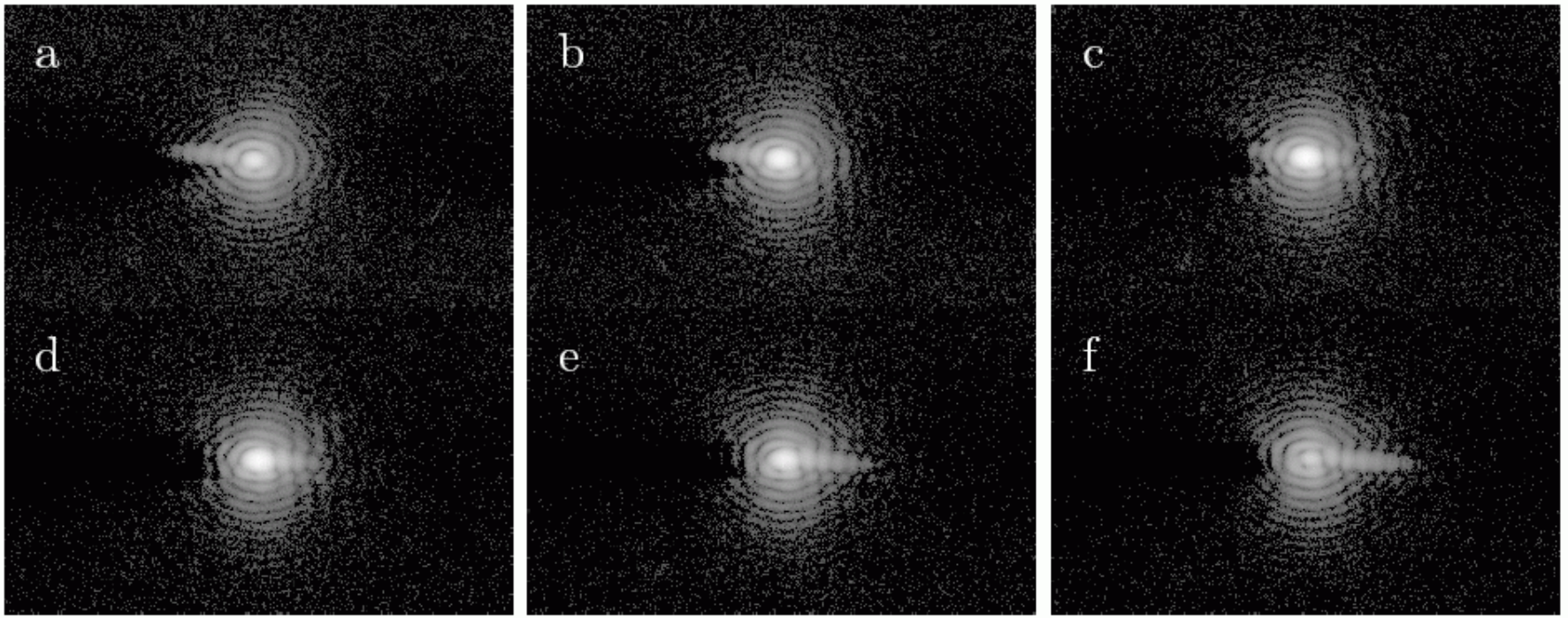
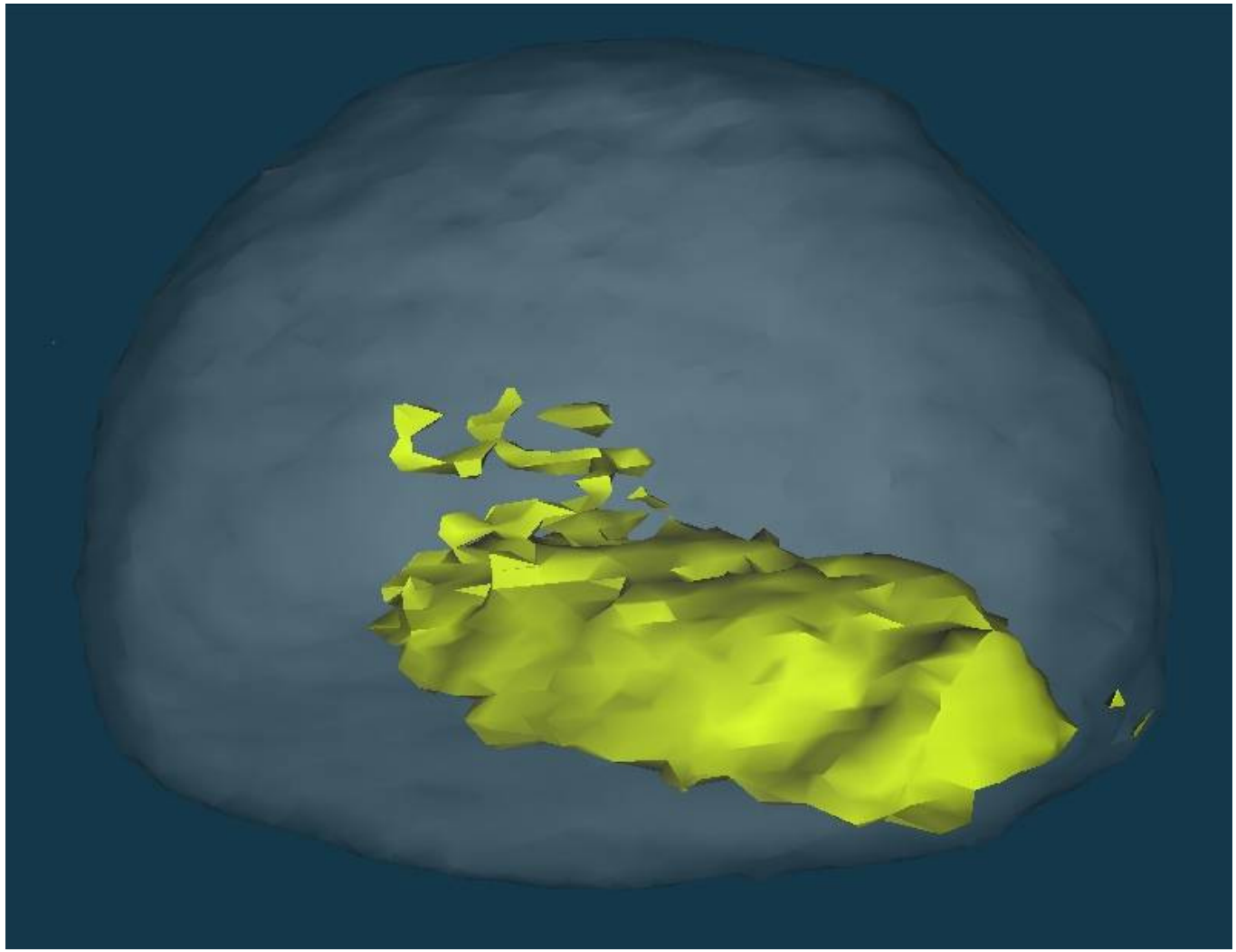
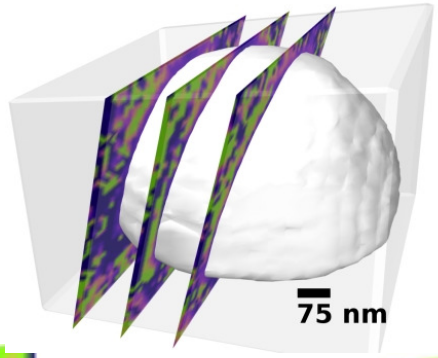


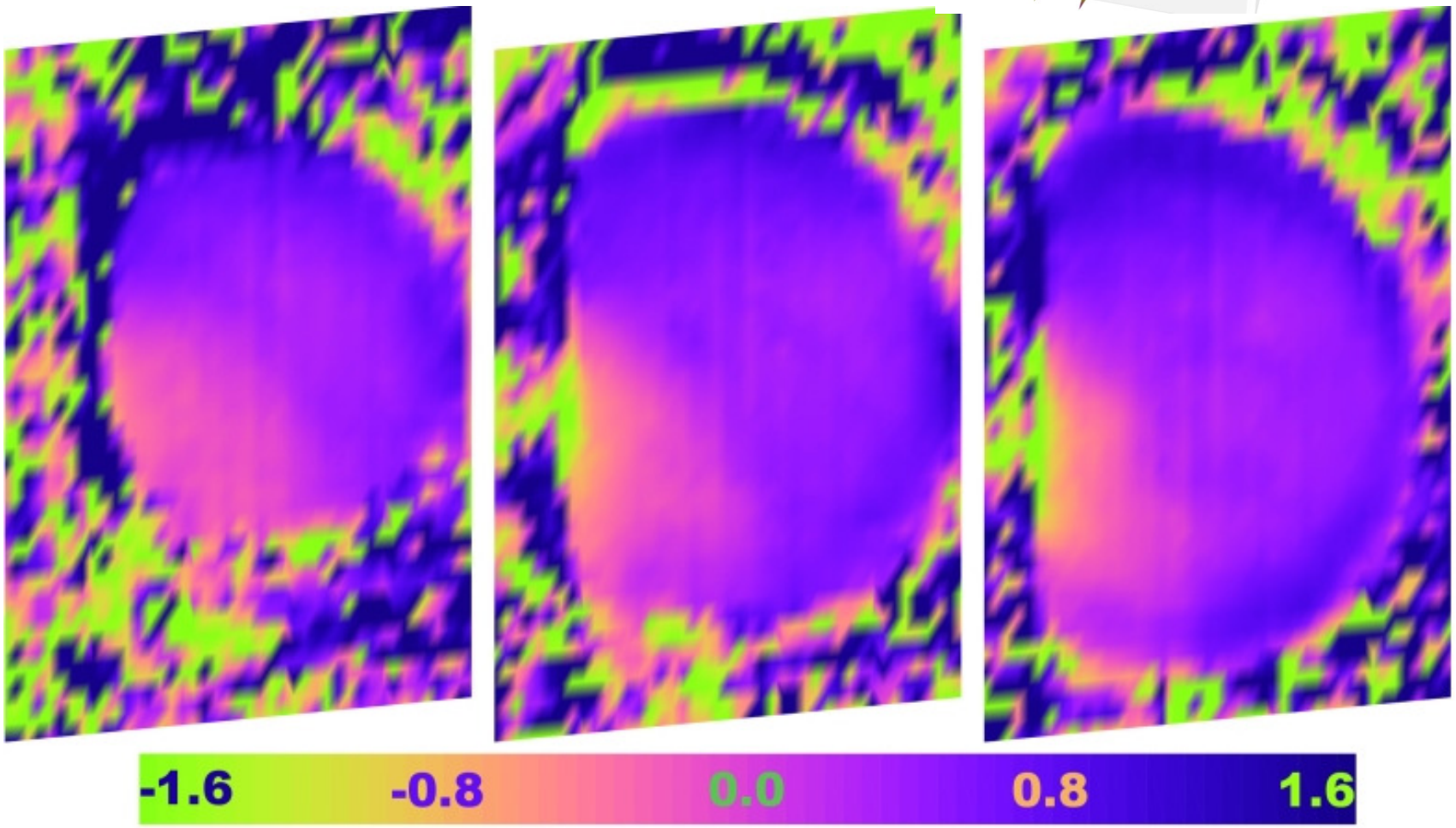
Figure 4.12: Center slices from 3D CXD pattern from Pb sample, on a log scale. Data file 296 from 10/03.

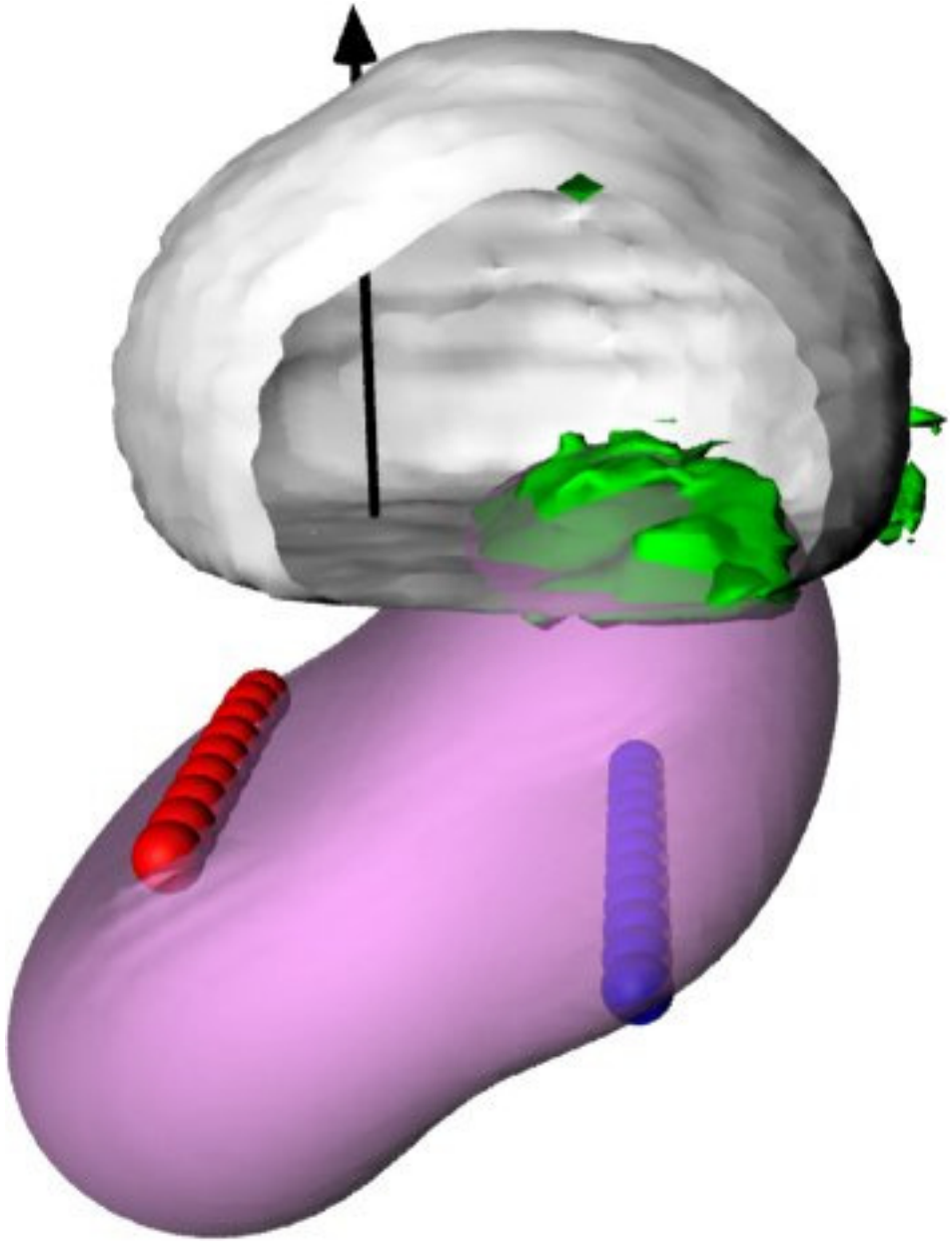
Modeling of 3D Phase Bump



3D phase map sections





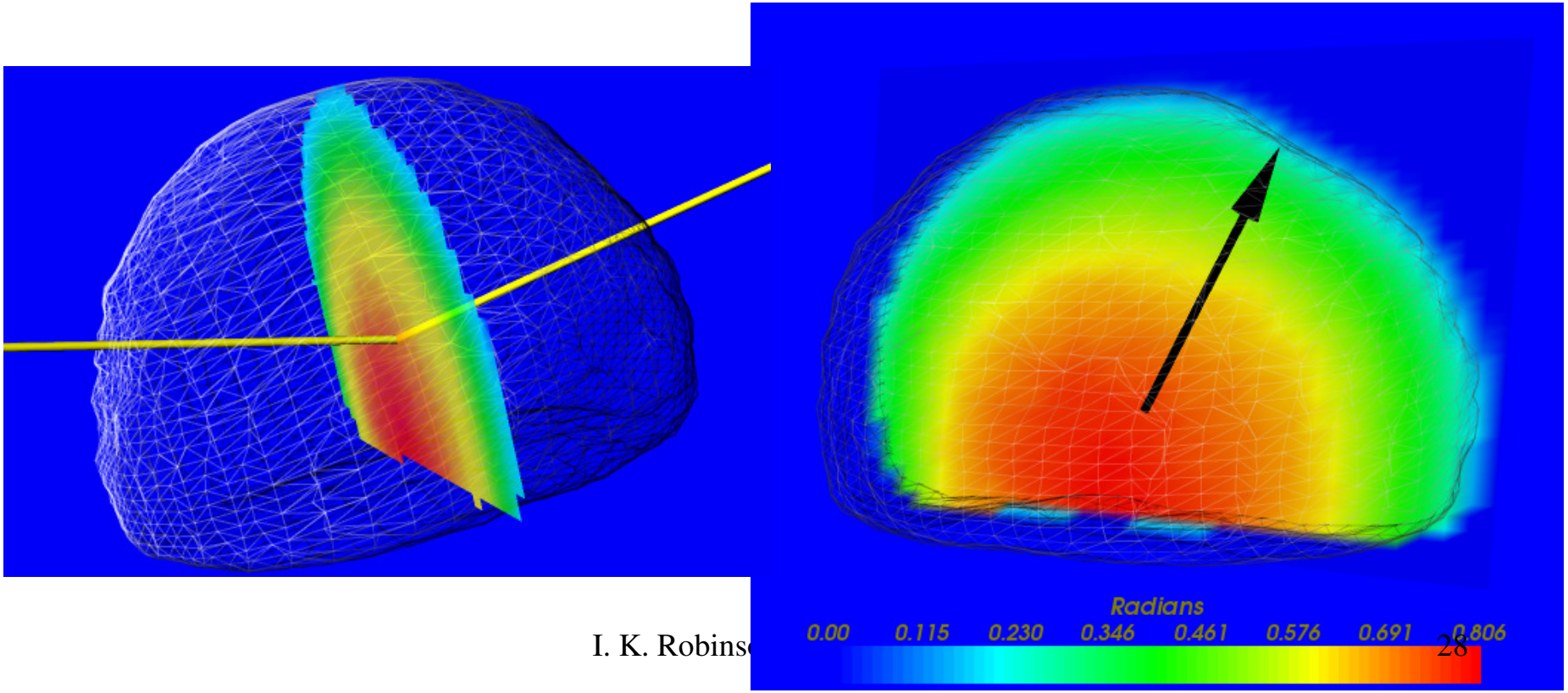


Refraction effects in Lead at 8.9keV

Phase accumulation due to refraction along scattering path

$$n=1-\delta+i\beta$$

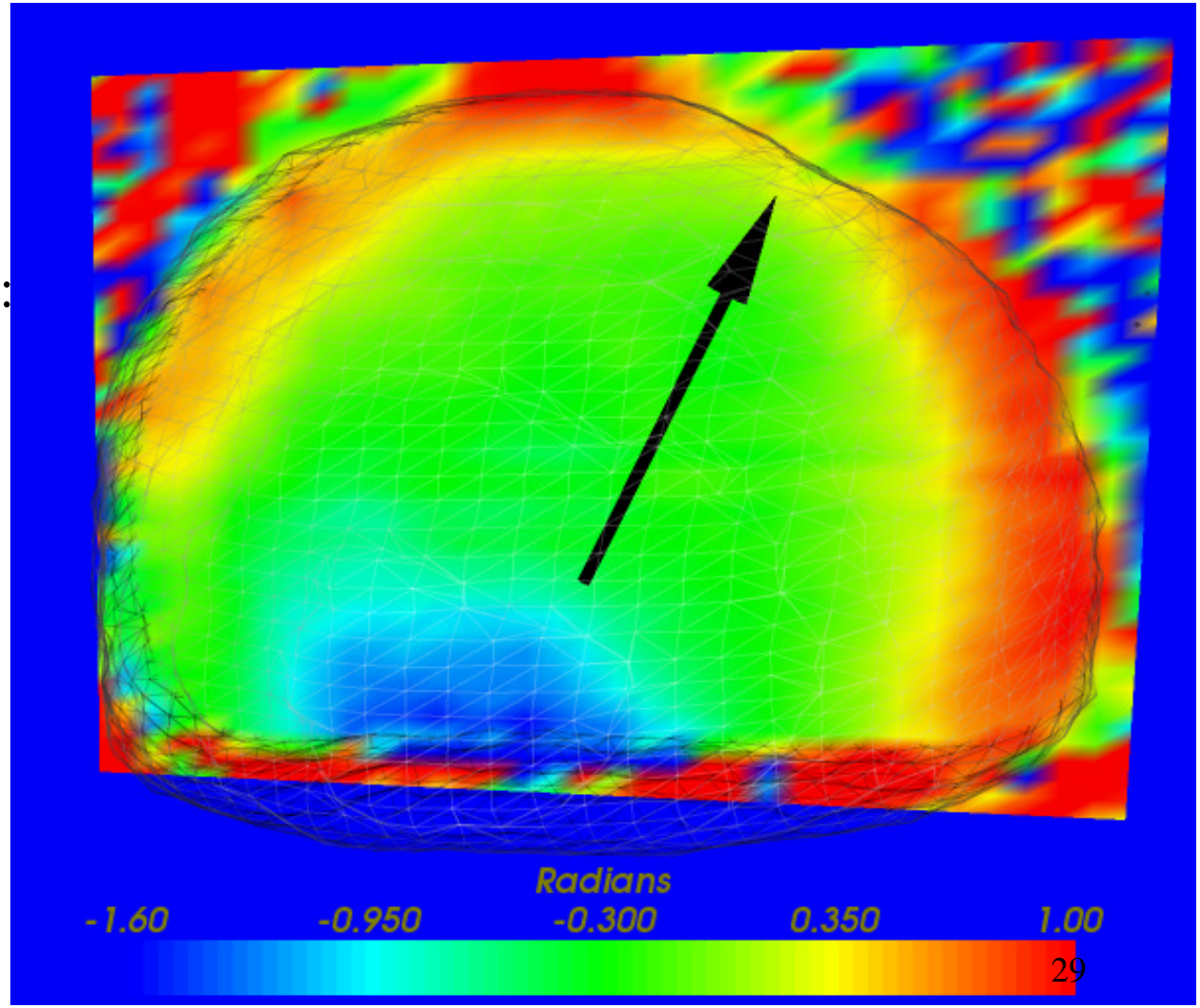
$$\delta=2.23\text{x}10^{-5}$$
$$\beta=2.19\text{x}10^{-6}$$



Refraction corrected phase map

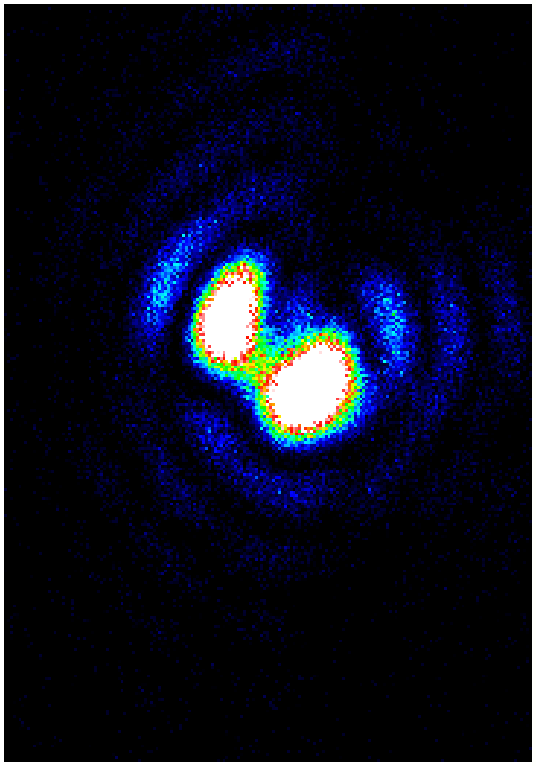
Max phase = 1.15rad
= 0.052nm

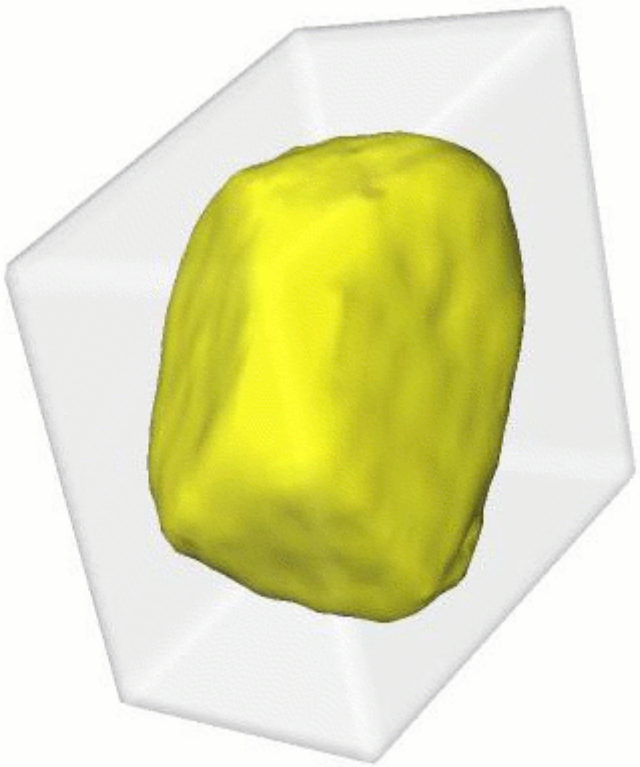
Phase on the (111) facet:
= 0.47 rad
= 0.02nm



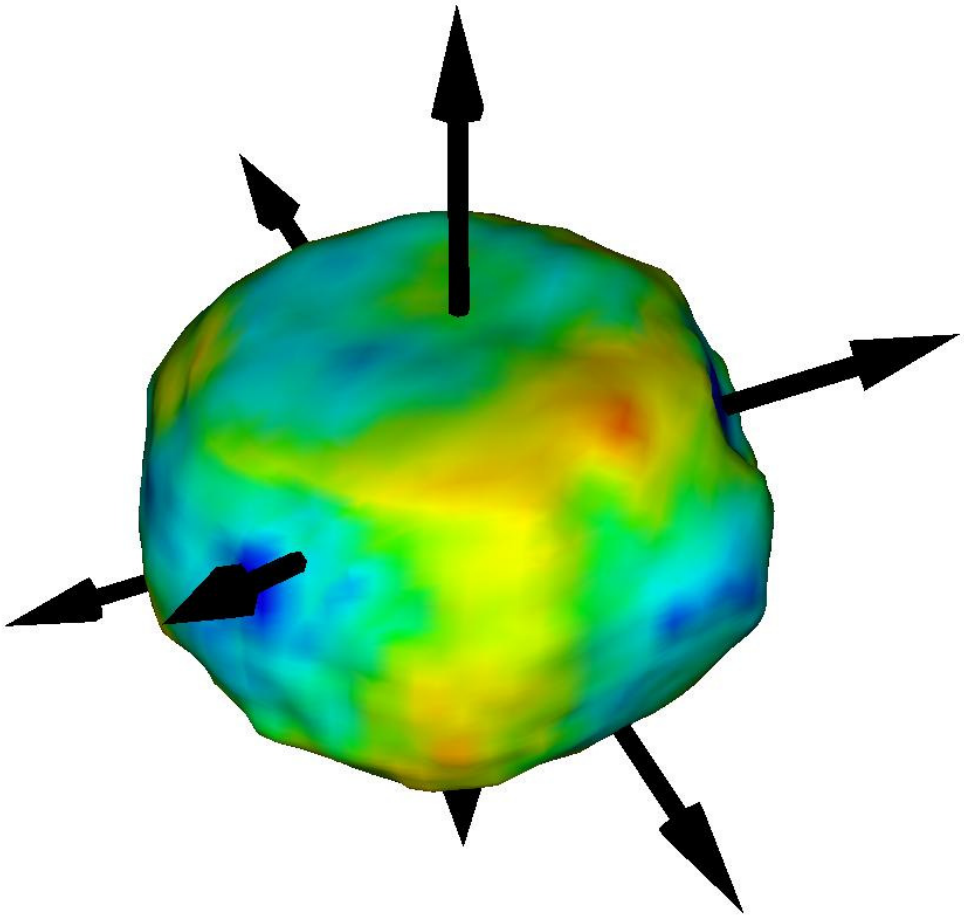
Gold nanocrystal reconstruction

showing support used for 20 HIO followed by 10 ER

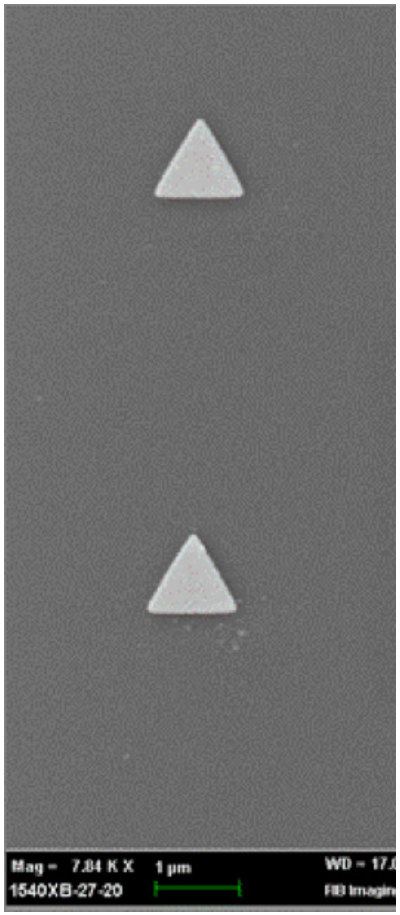


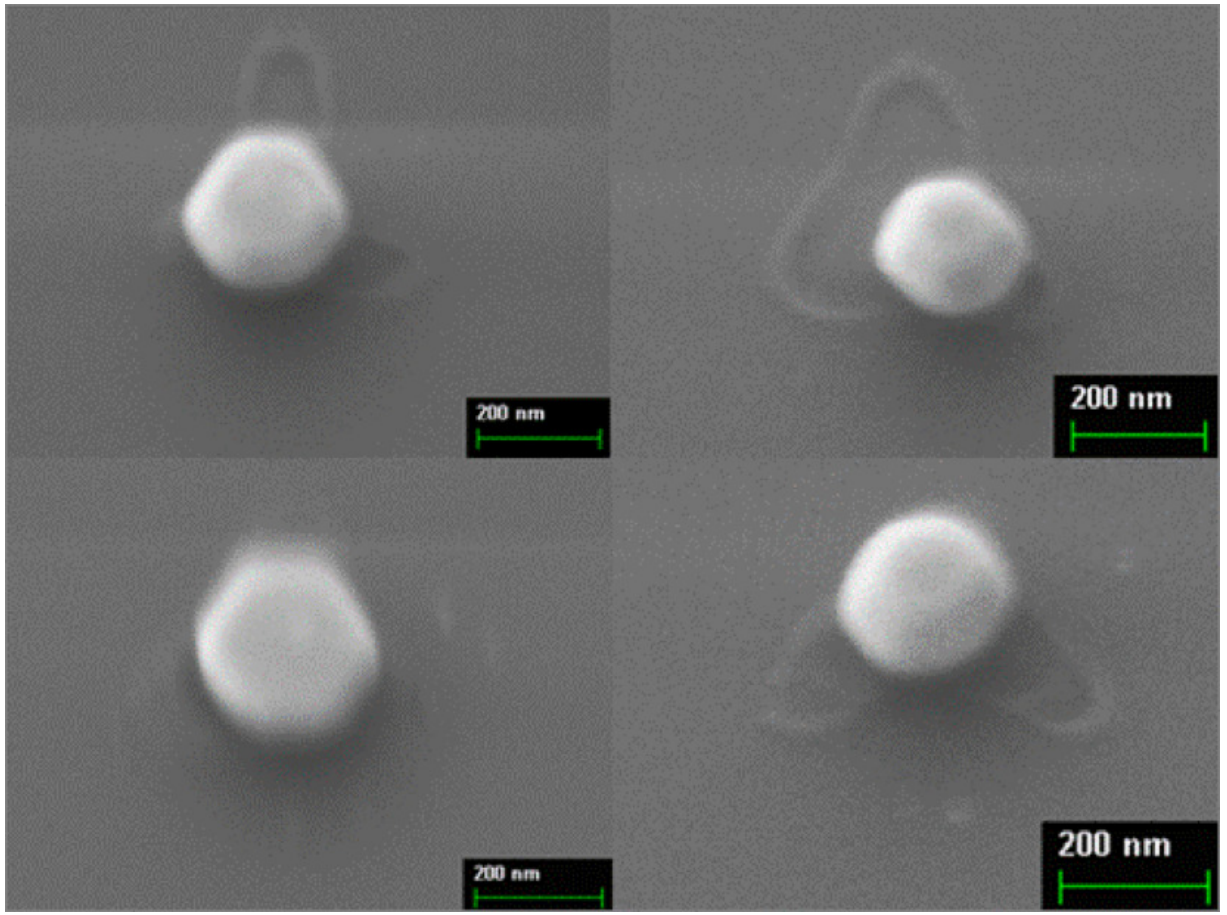


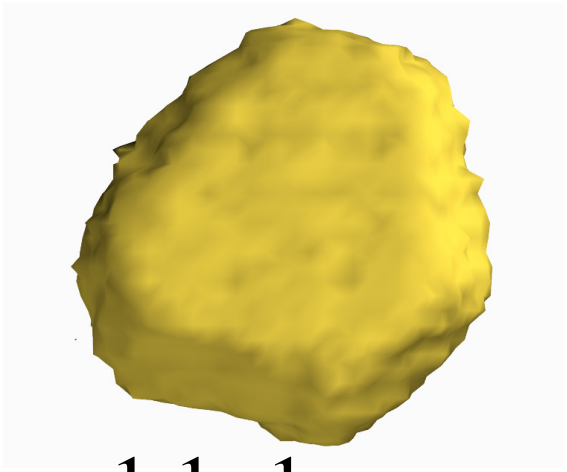
Phase isosurface of residual strain



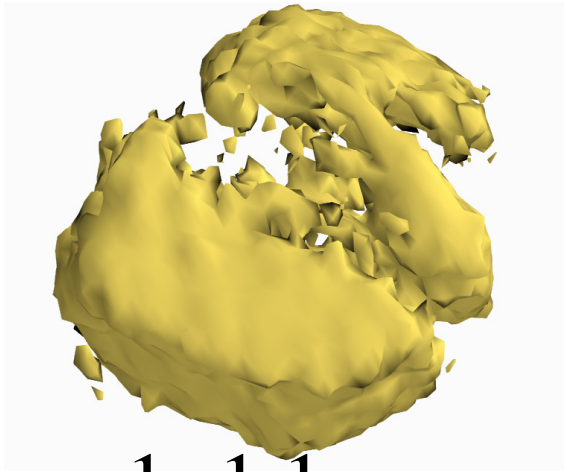
Single Au nanocrystal synthesis



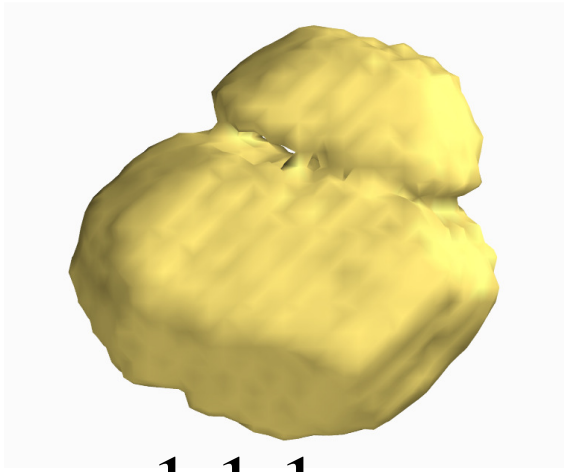




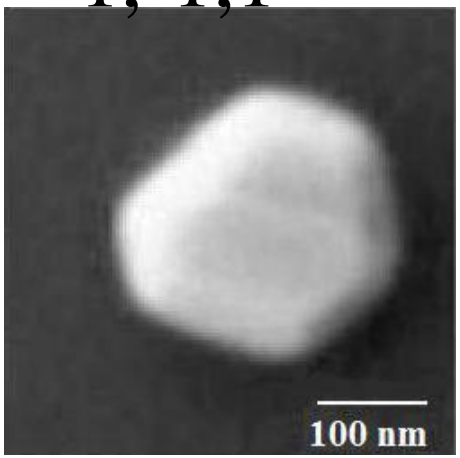
1,1,-1



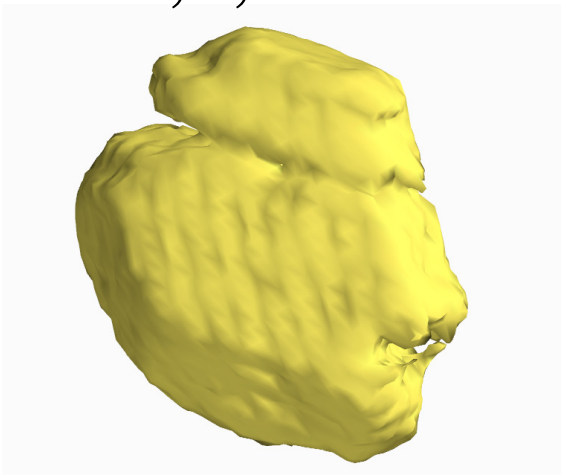
1,-1,1

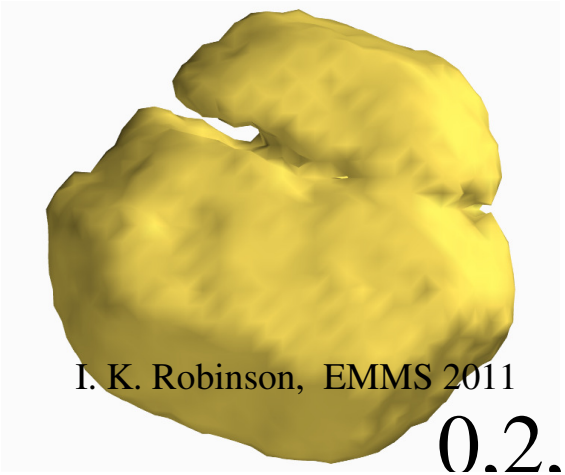


-1,1,1



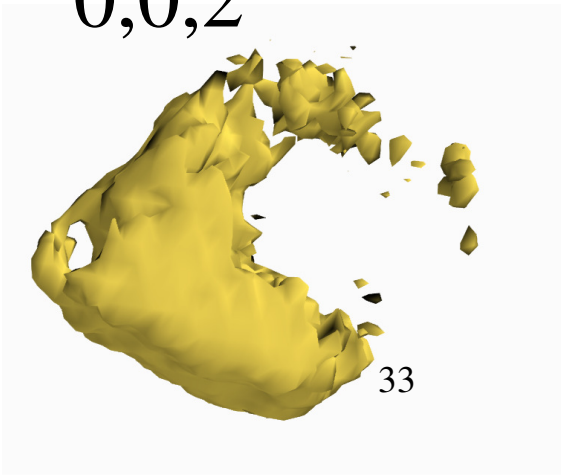
2,0,0





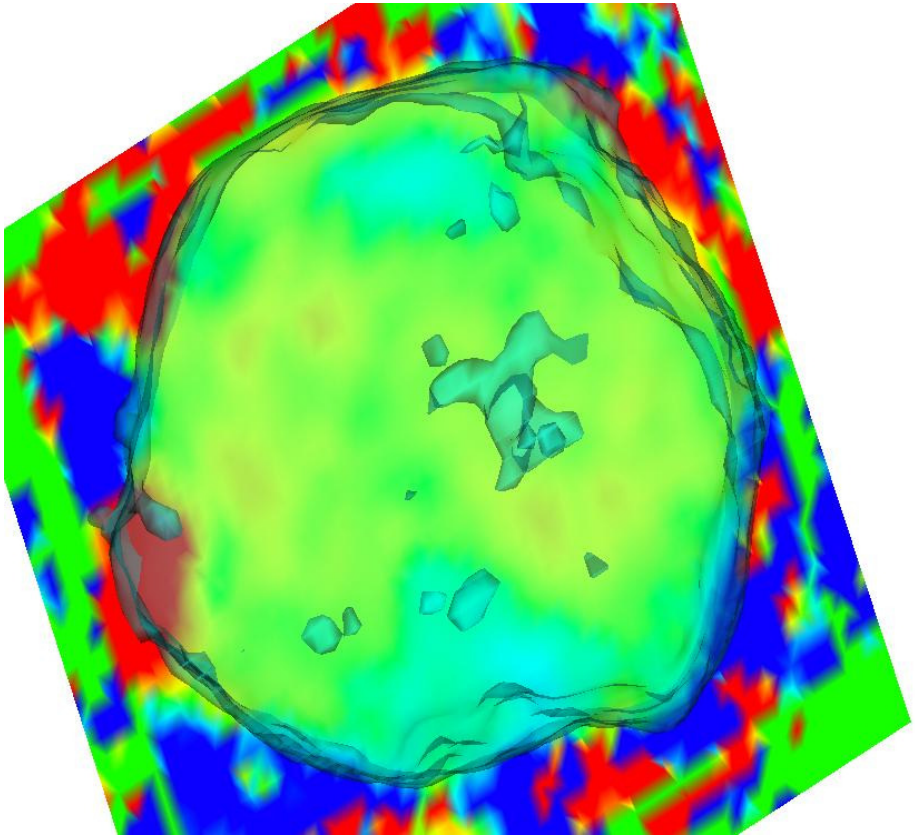
0,2,0

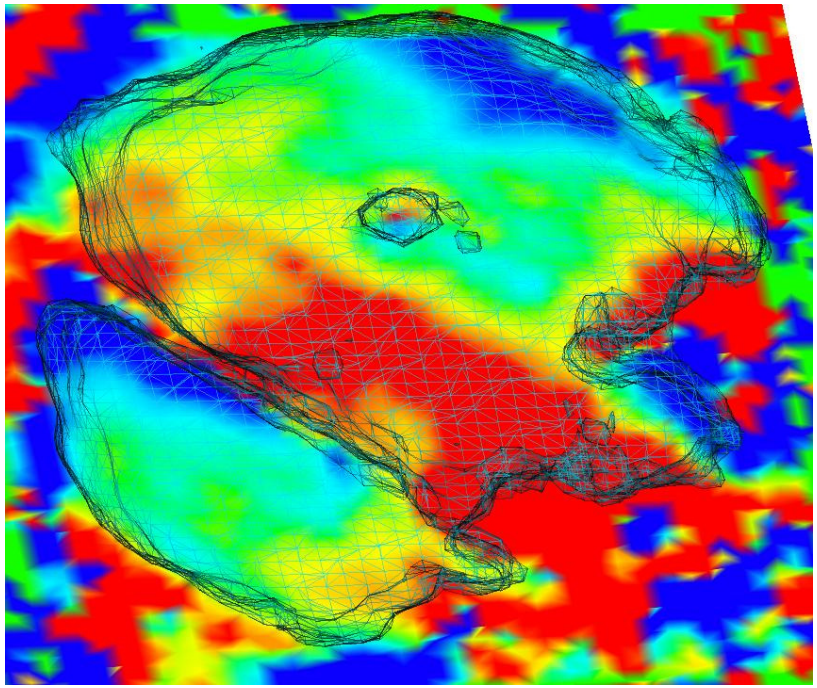
0,0,2



Two views of strain in Au NC

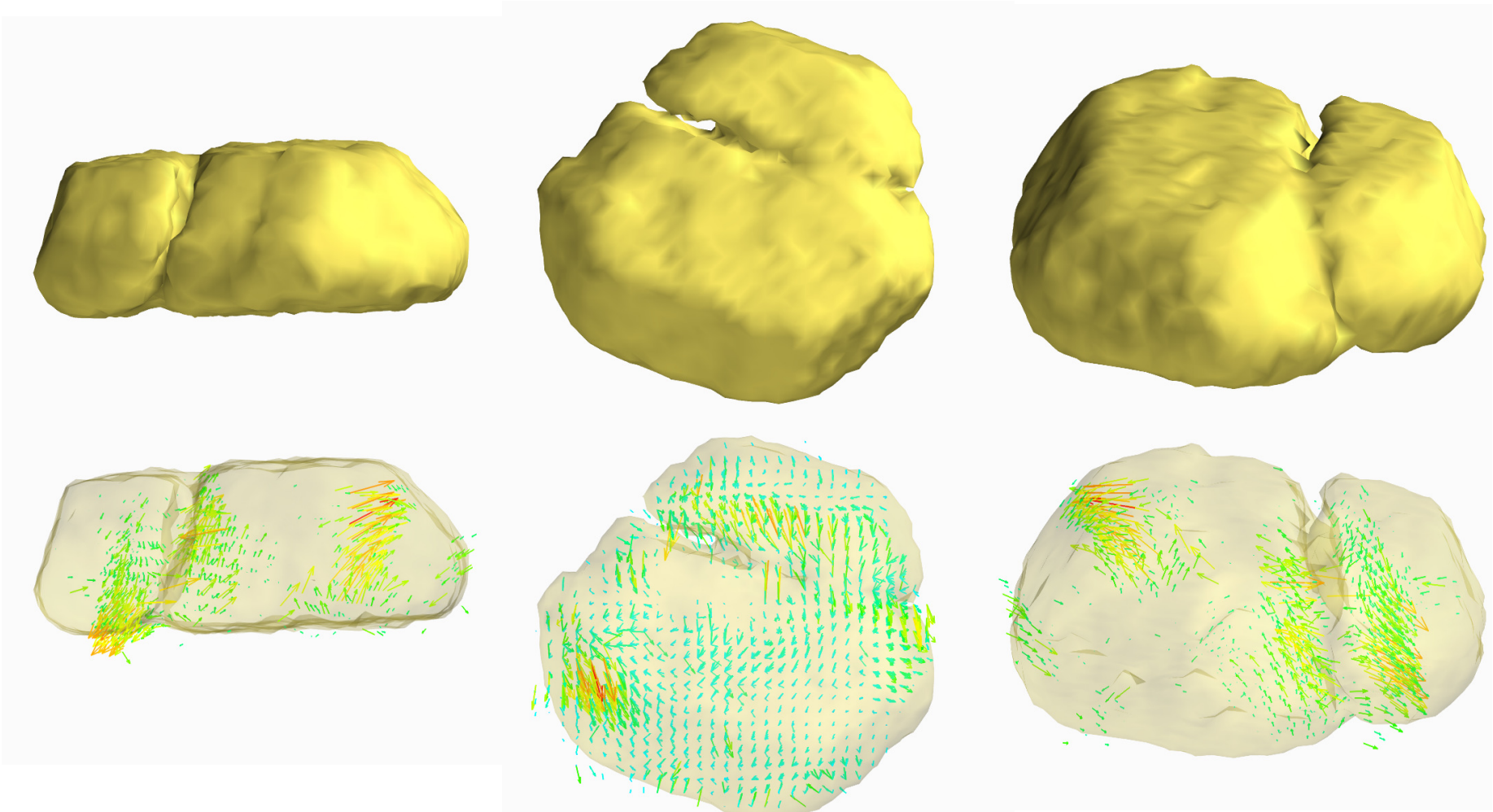
Au409B-52 (11-1) and Au409B-60 (200)



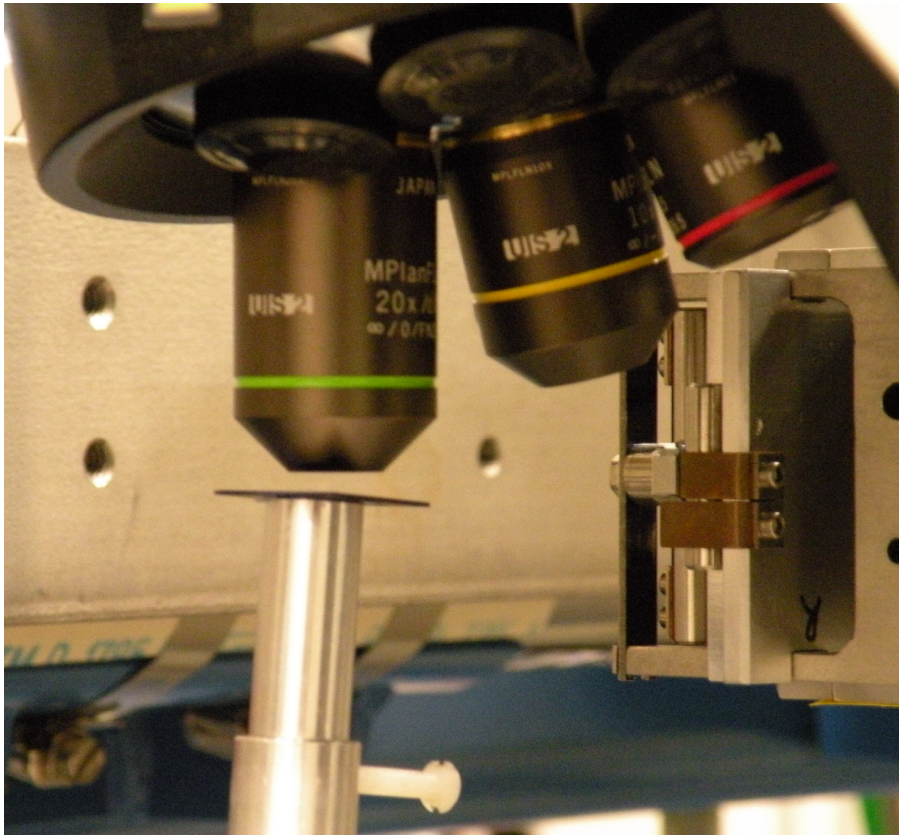


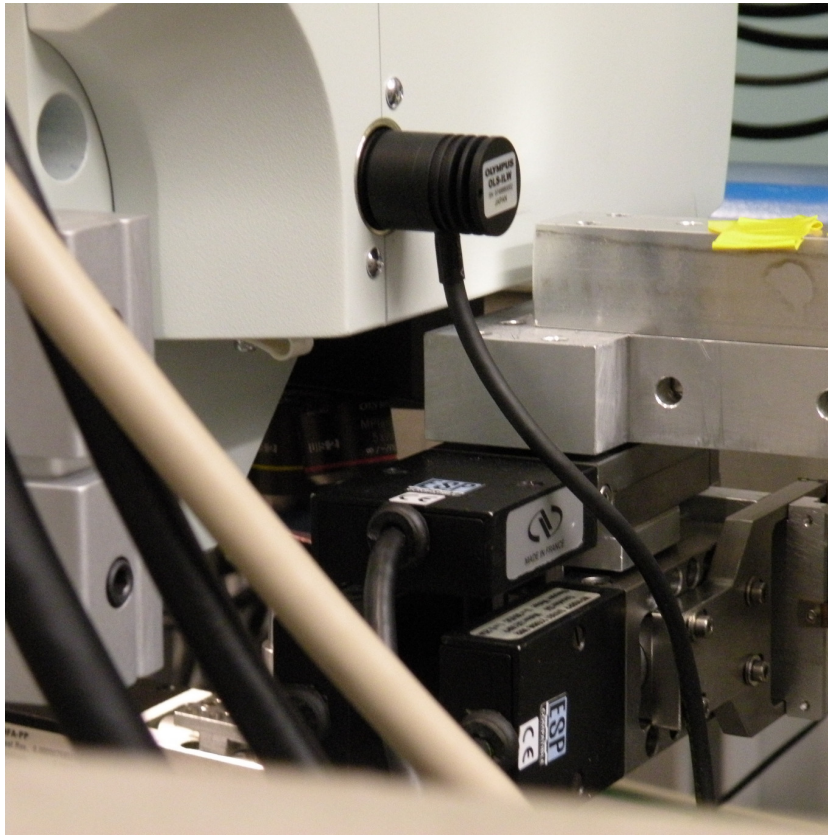
Vector displacement field

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



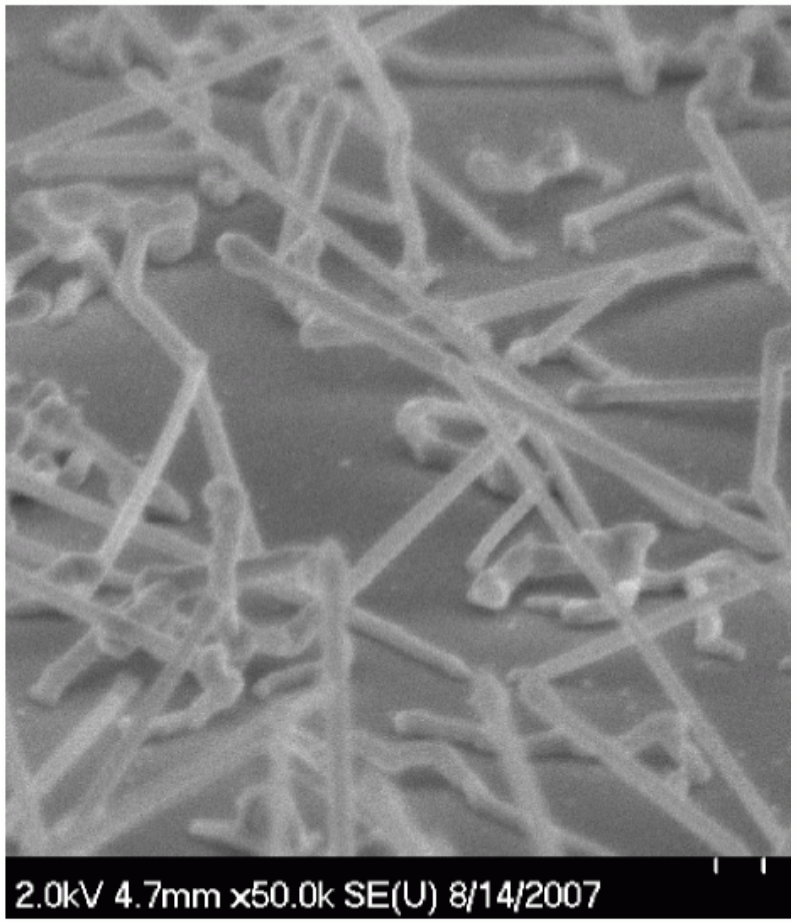
Confocal Alignment Microscope



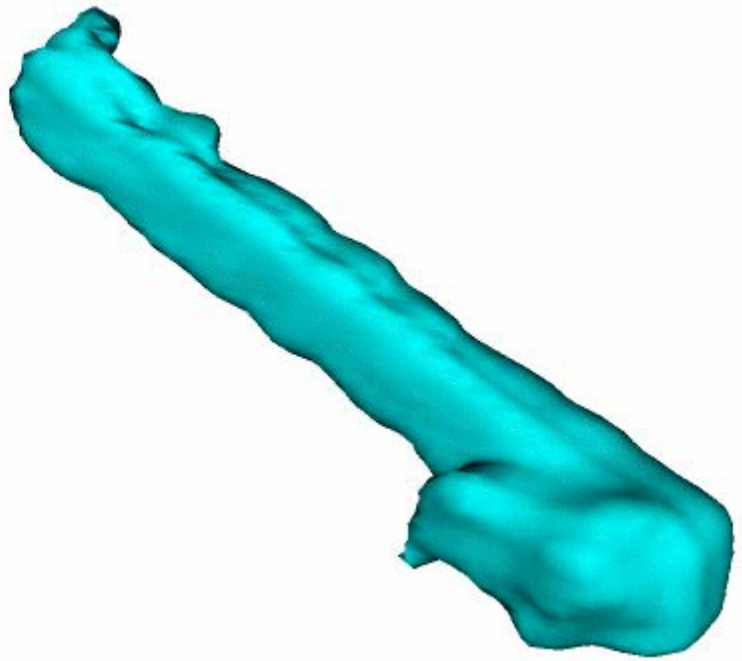


Reconstruction of InP nanowire

CVD on Si, Suneel Kodambaka, UCLA

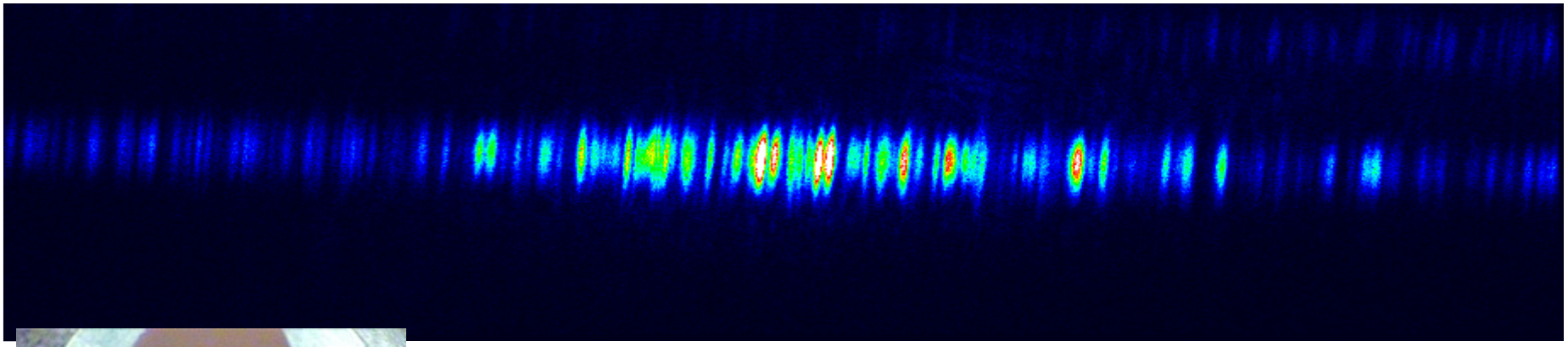


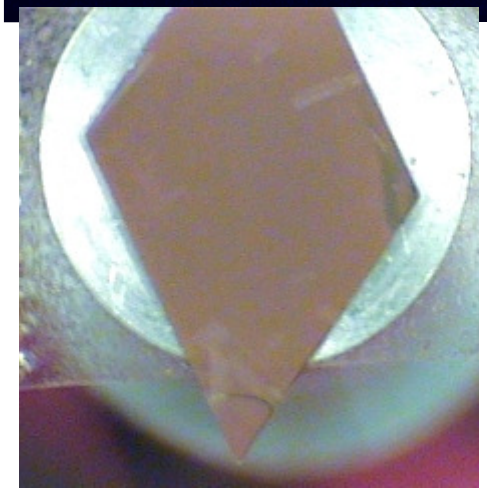
InP nanowires grown on Si (111)

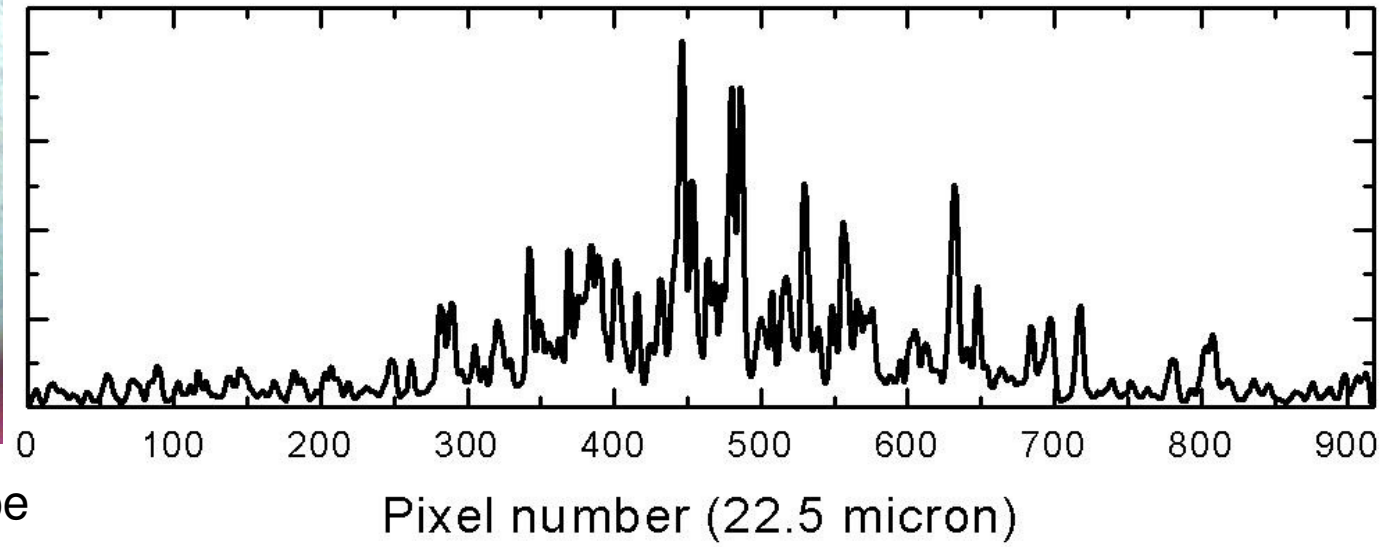


GaAs Nanowire "Barcode"

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

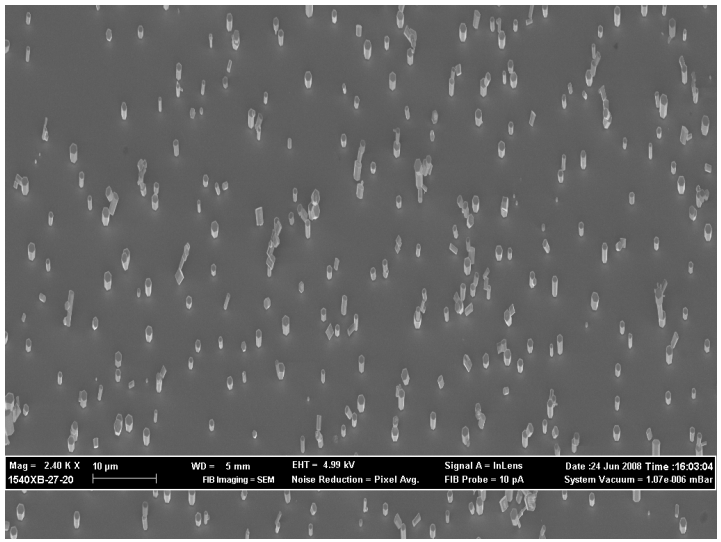


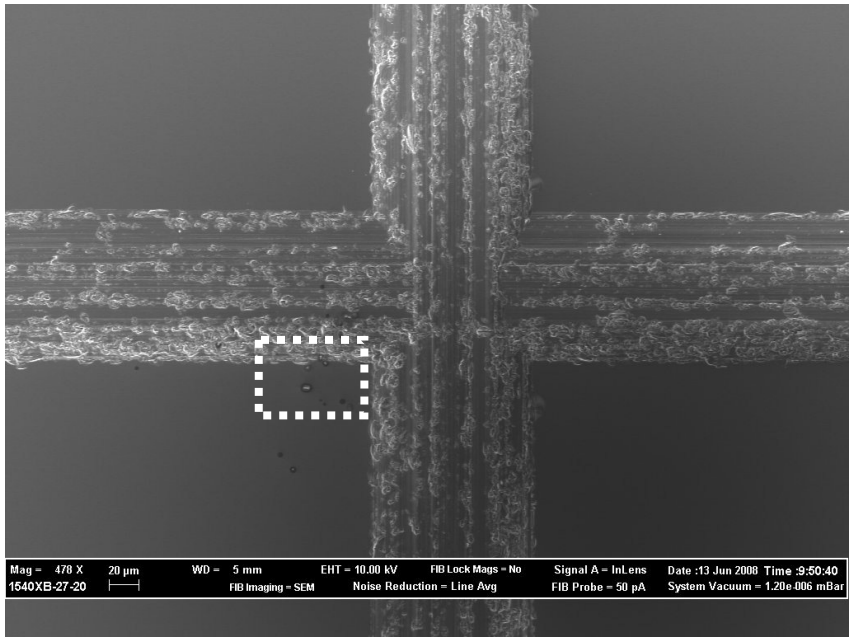


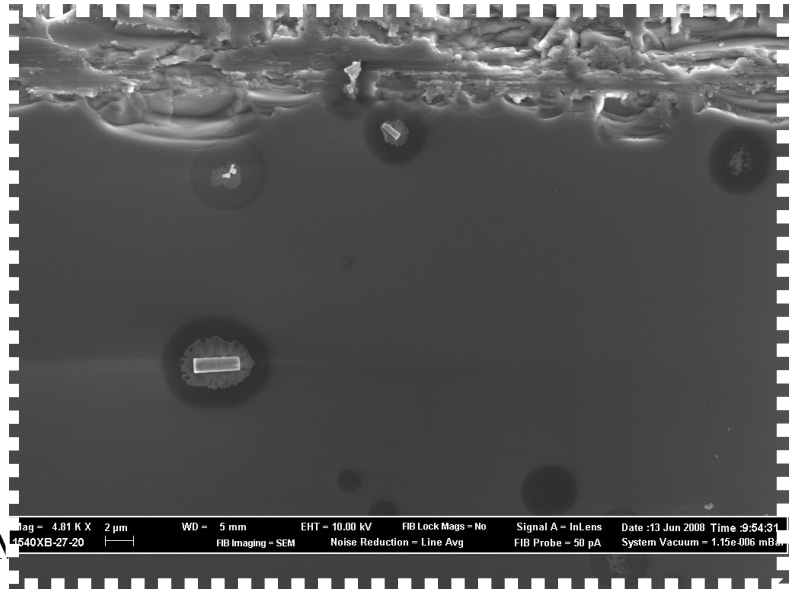


GaAsNW1106-22.spe
B9348 from Philips

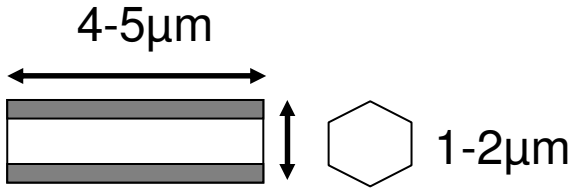
ZnO Sample Preparation





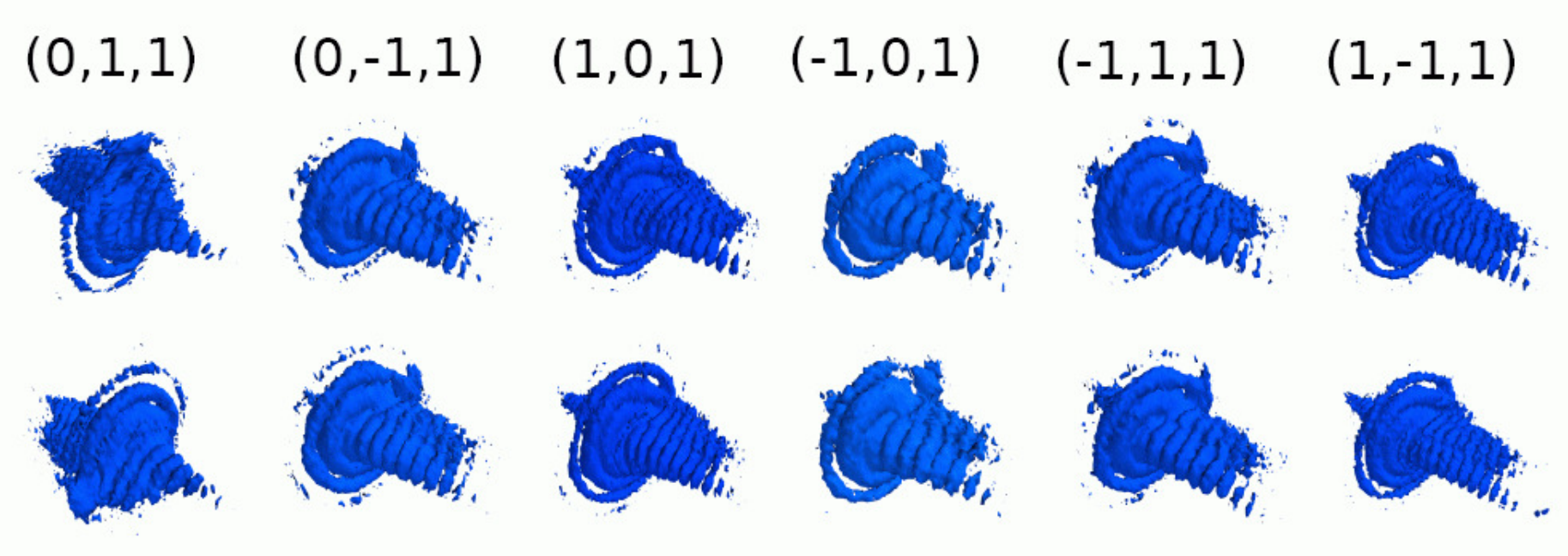


Dimensions:

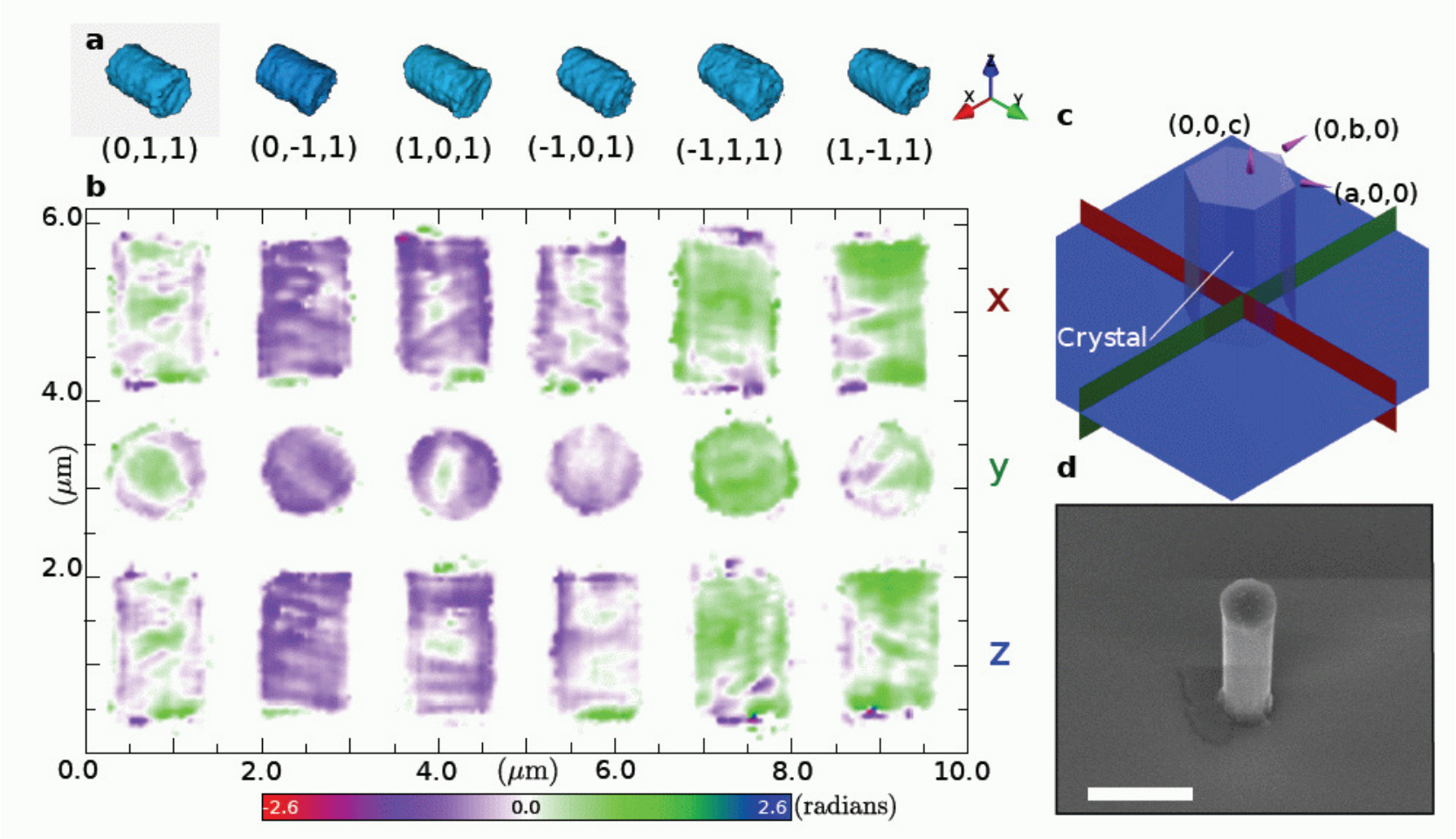


I. K. Robinson, EMM

Six Independent Bragg Peaks

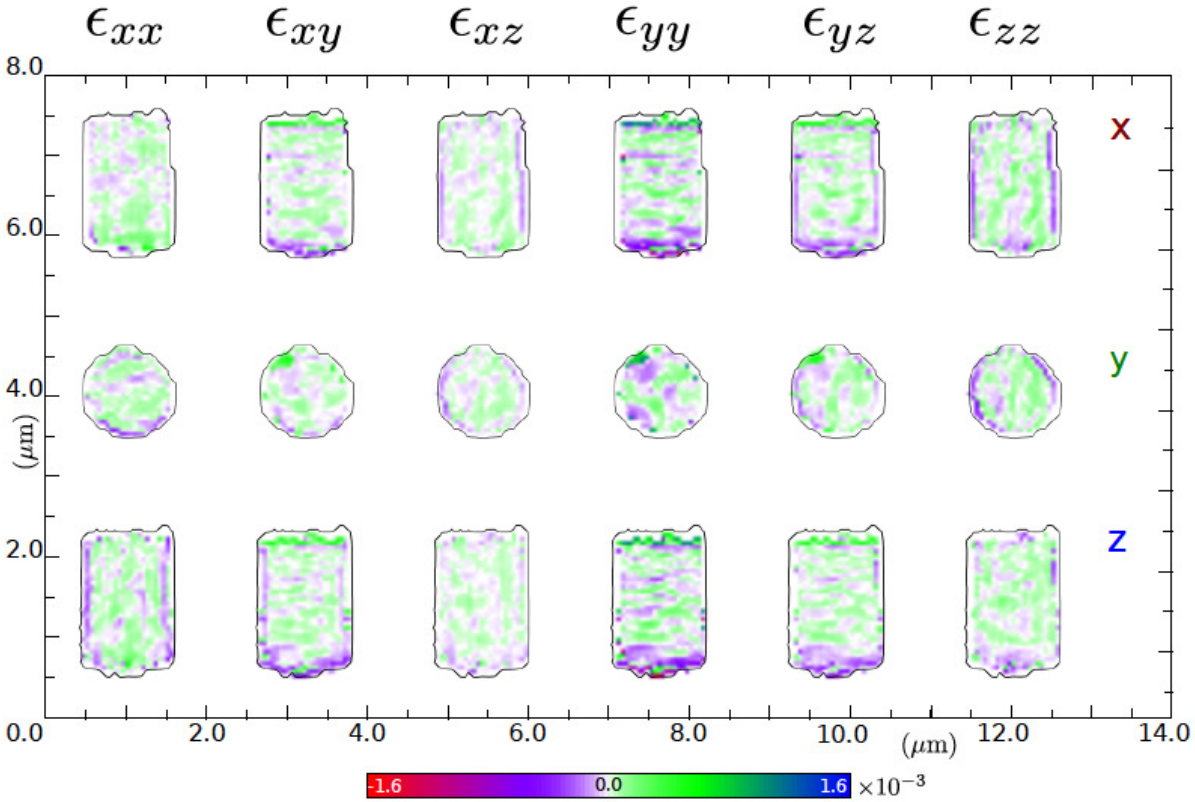


Separate images 6 Bragg Peaks

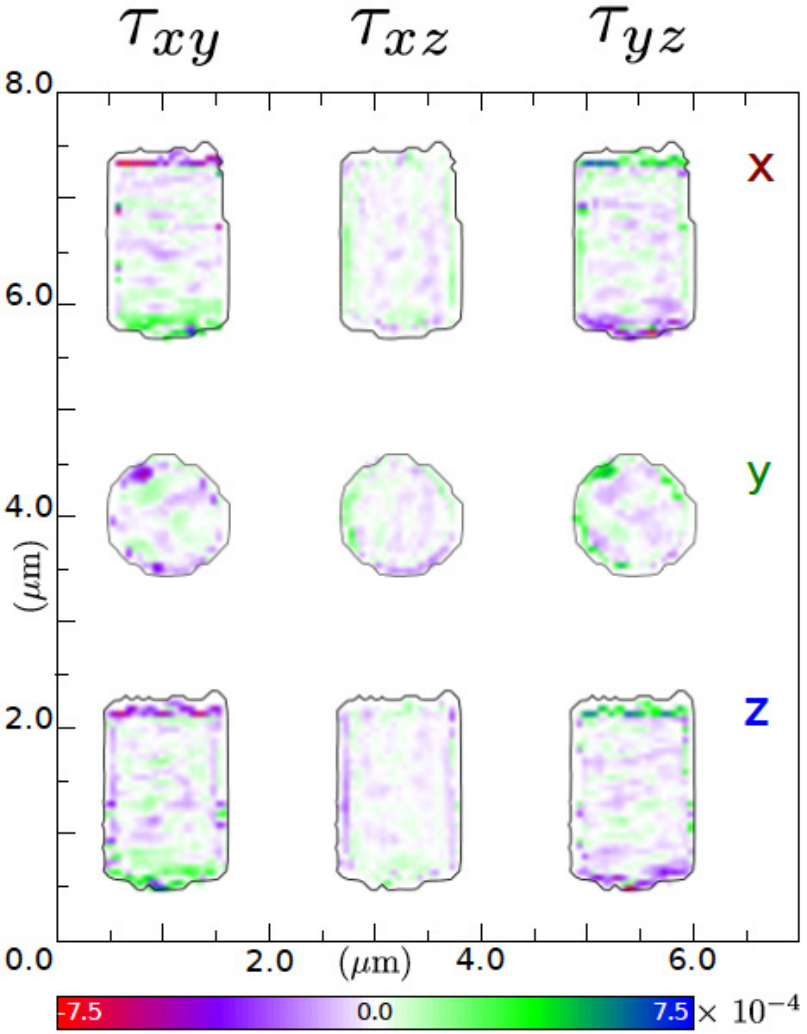


Full Strain Tensor

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

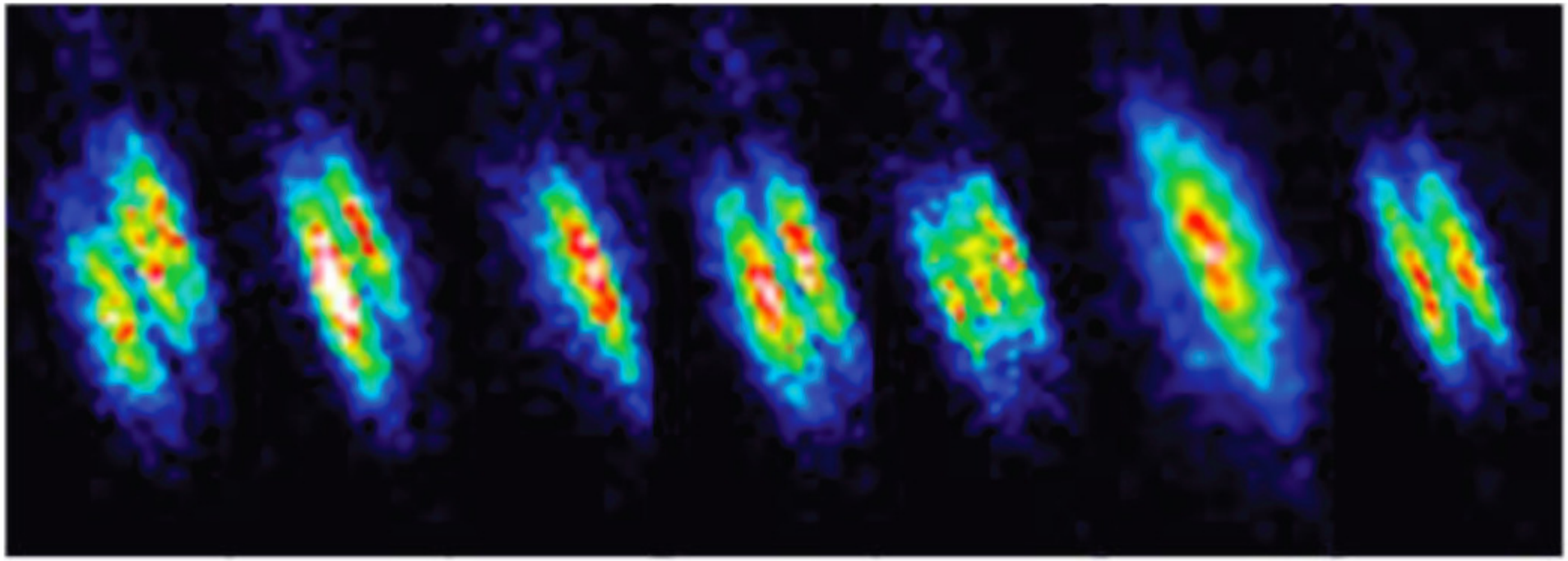


ZnO Rigid-body Rotations

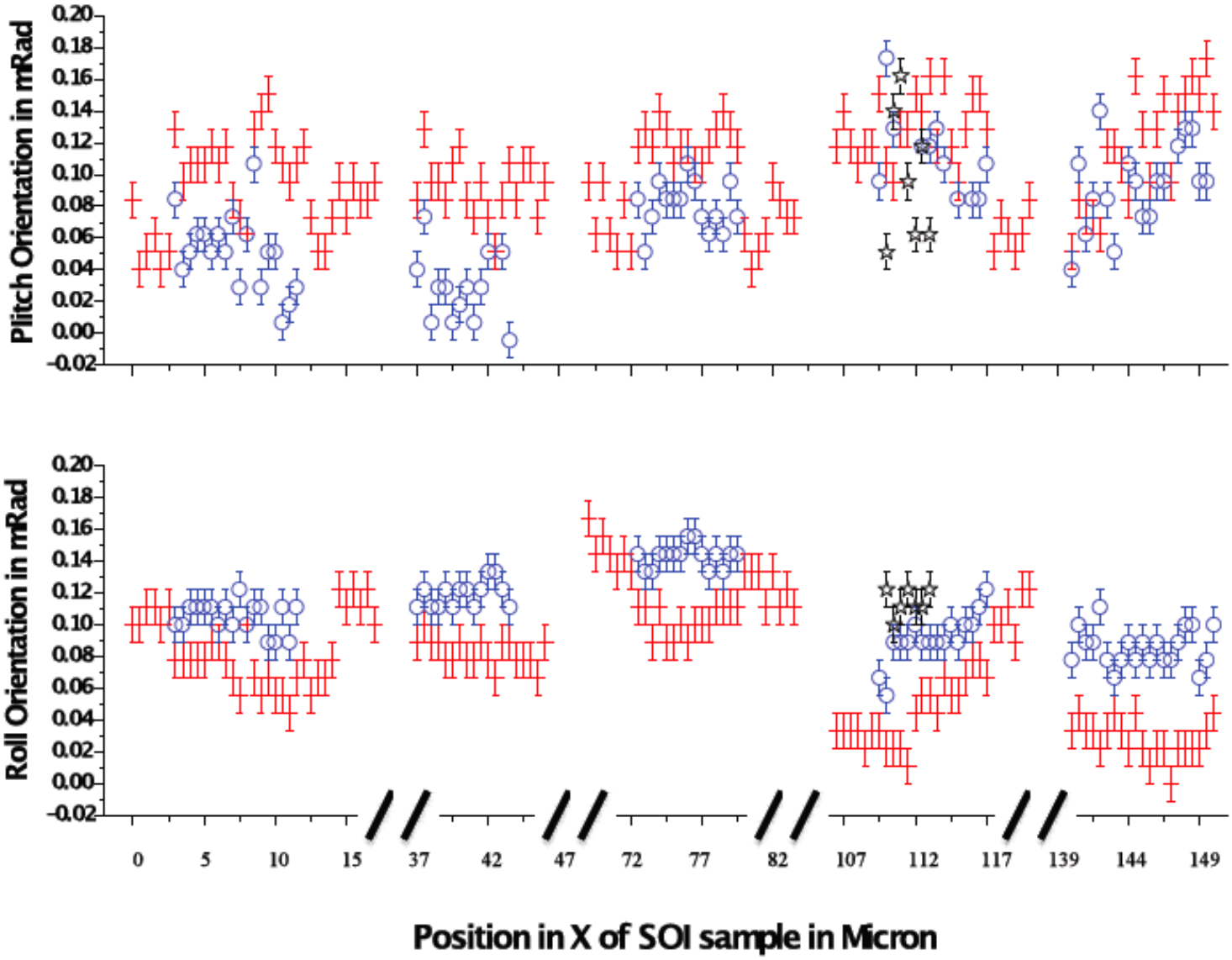


Silicon on Insulator

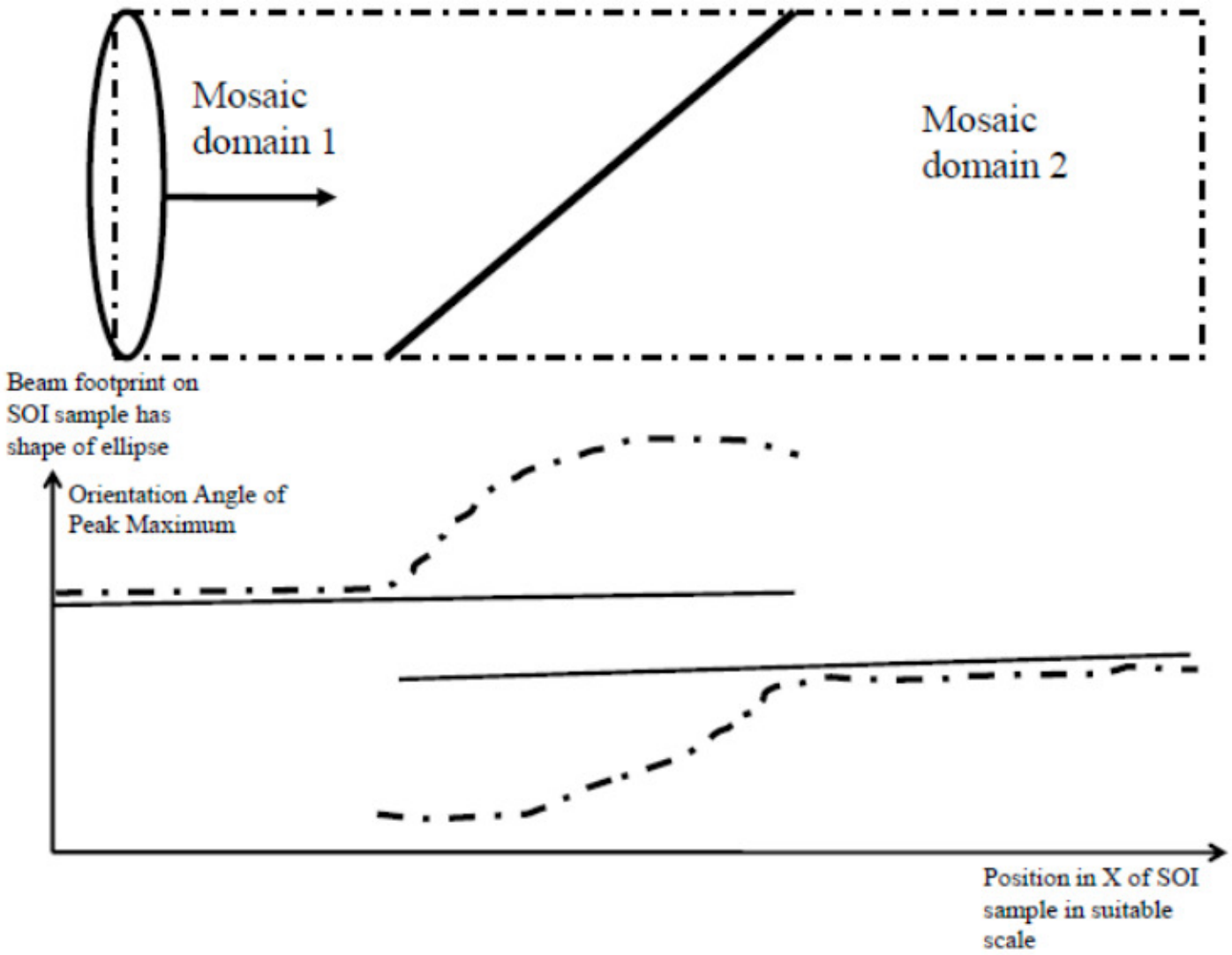
Microdiffraction, 1.6um beam, 9um steps
Xiaowen Shi et al, Z. Krist (2010)



Mosaic domains on SOI film



Coherent or incoherent effect?



Conclusions

- CXD is a new branch of Crystallography
- 3D imaging practical for nanocrystals
- Phasing by computation instead of lens
- Strain fields imaged from asymmetric patterns
- Applications to semiconductors and metals