Uses of X-ray Coherence for Exploring Structure on the Nanoscale

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NSLS Users Meeting
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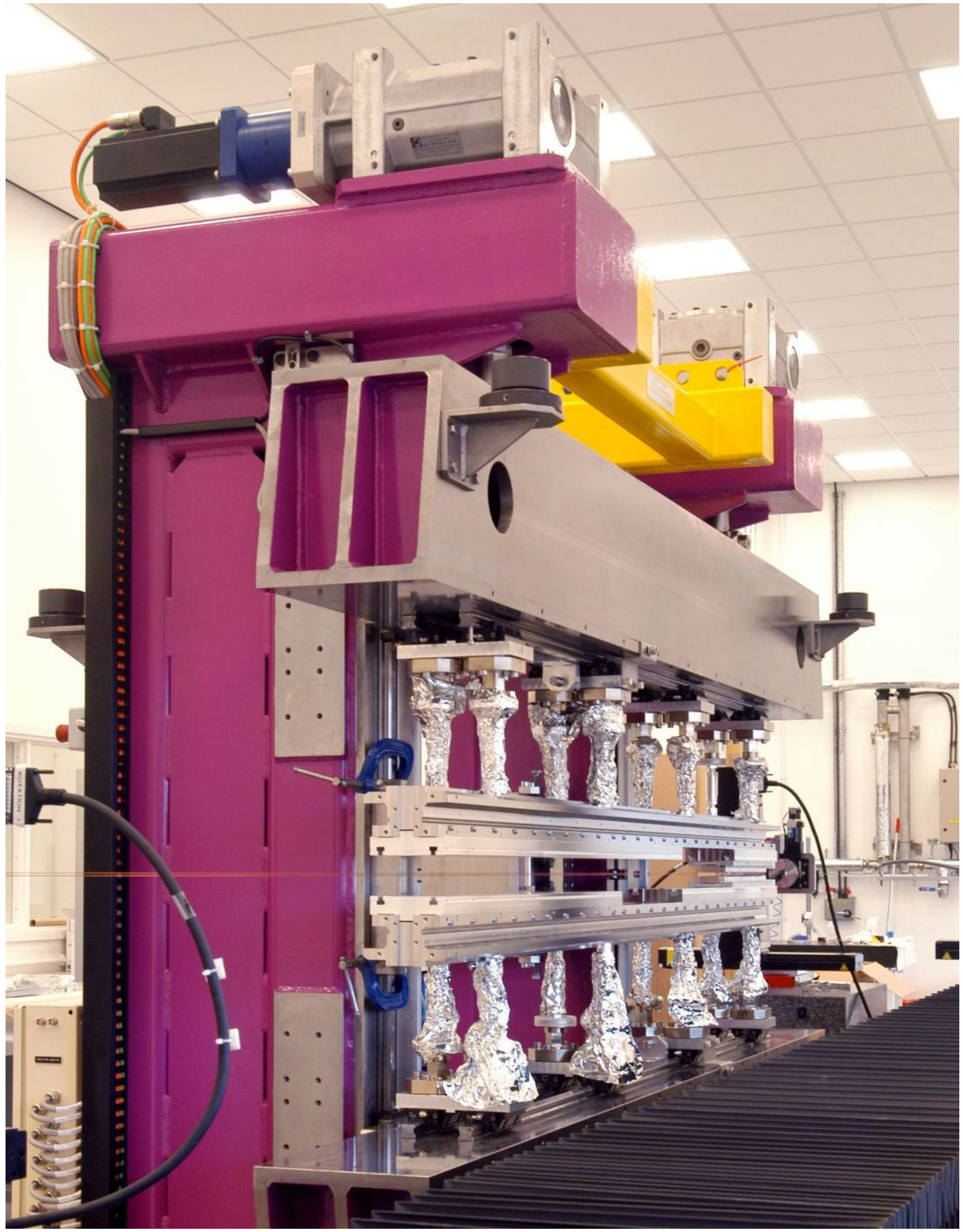
Outline

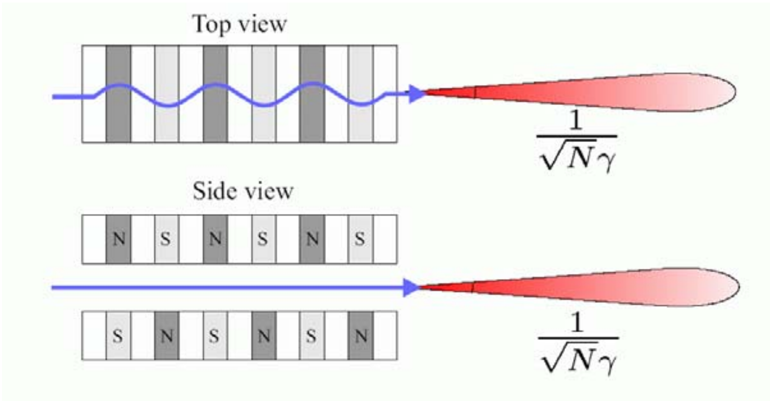
- Coherent x-ray diffraction
- CXD can solve the phase problem
- Nanocrystal structures
- Extension to phase objects
- Exploration of crystal strain
- Nanowire structures
- Biological applications of coherence

Diamond in-vacuum X-ray Undulator

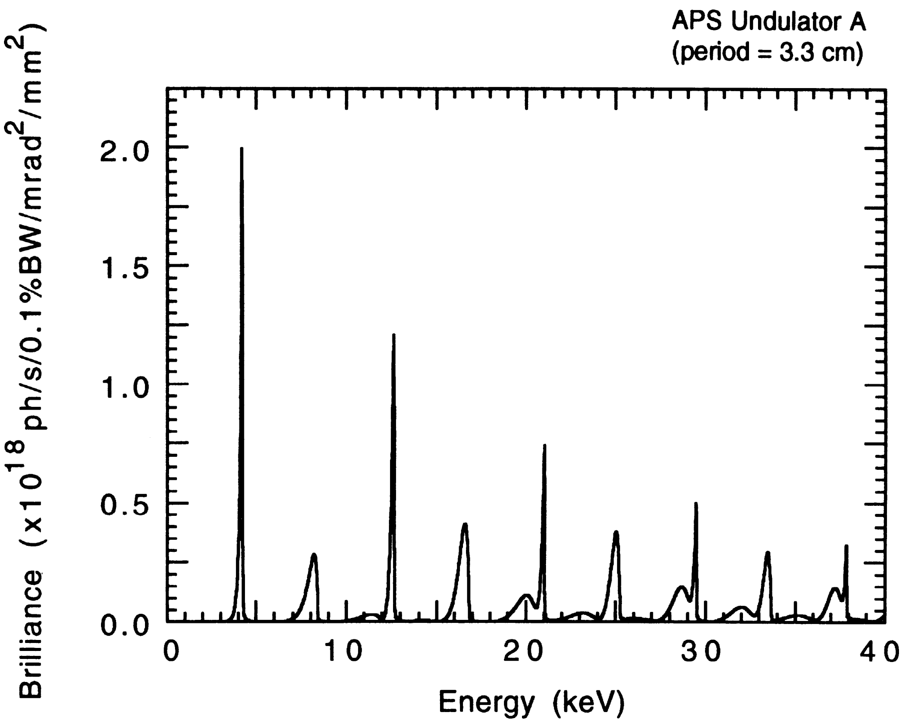


I. K.

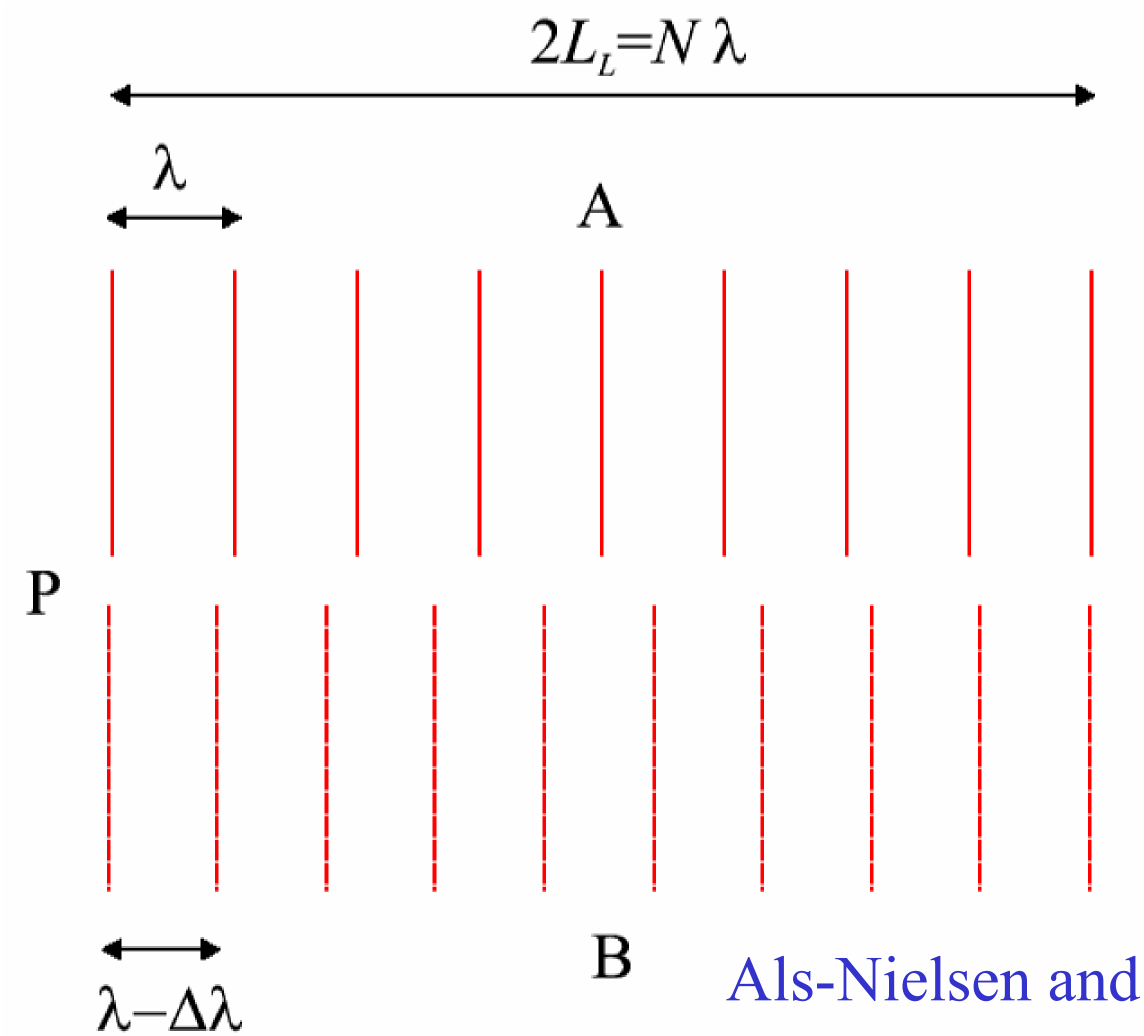
X-ray Undulator Principle



$$\lambda_X = \frac{\lambda_U}{2\gamma^2}\left\{1 + \frac{K^2}{2} + (\gamma\theta)^2\right\}$$



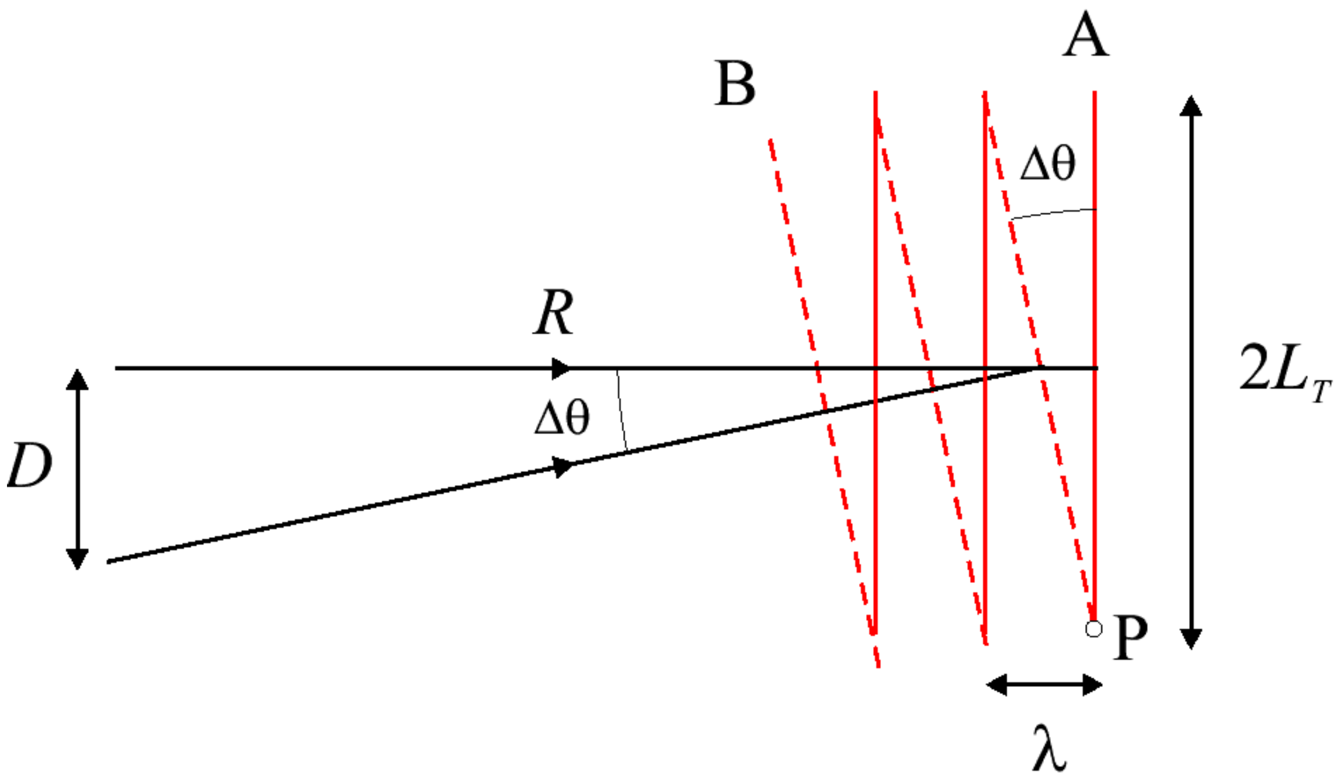
Longitudinal Coherence



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

Als-Nielsen and McMorrow (2001)

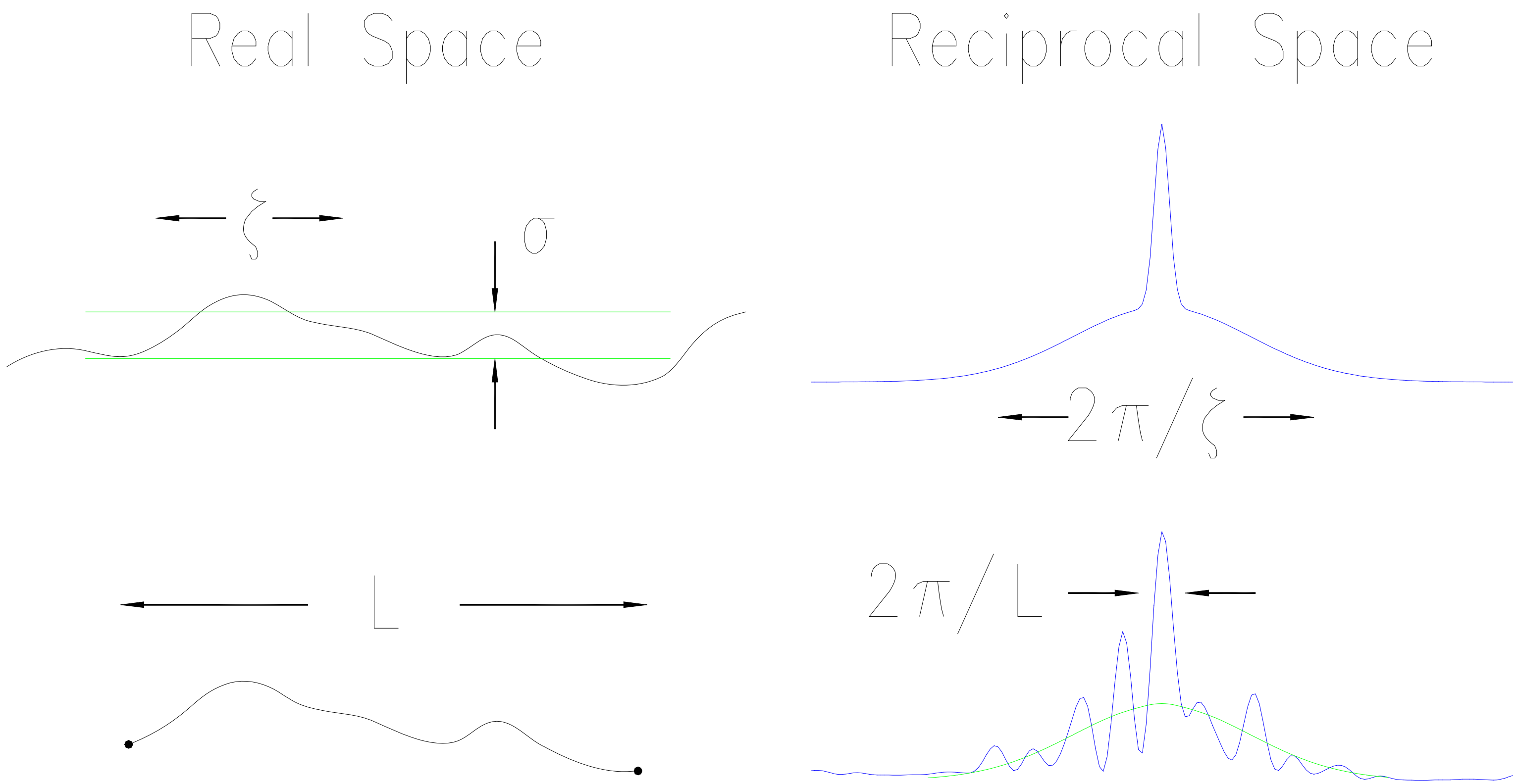
Lateral (Transverse) Coherence



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

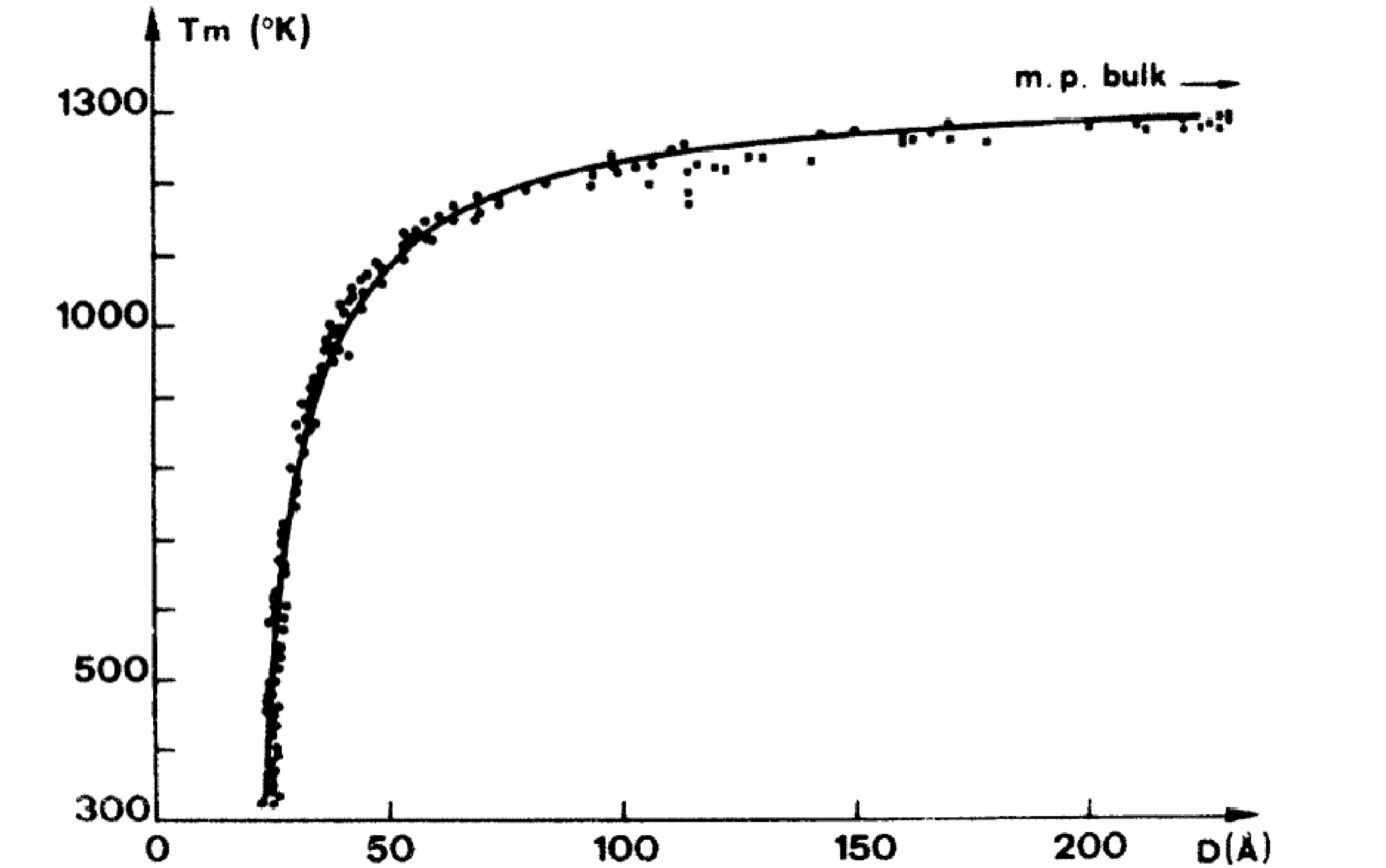
Als-Nielsen and McMorrow (2001)

Diffuse Scattering acquires fine structure with a Coherent Beam

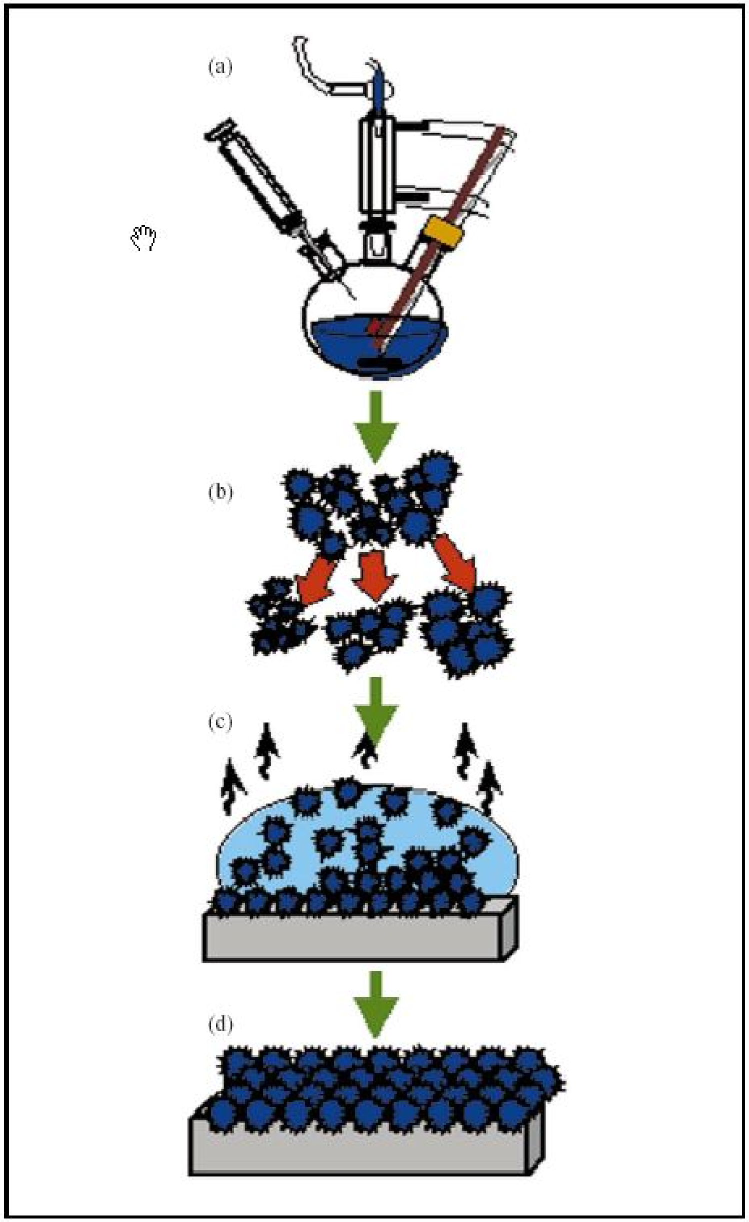


Size-dependent Melting of Au Particles

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

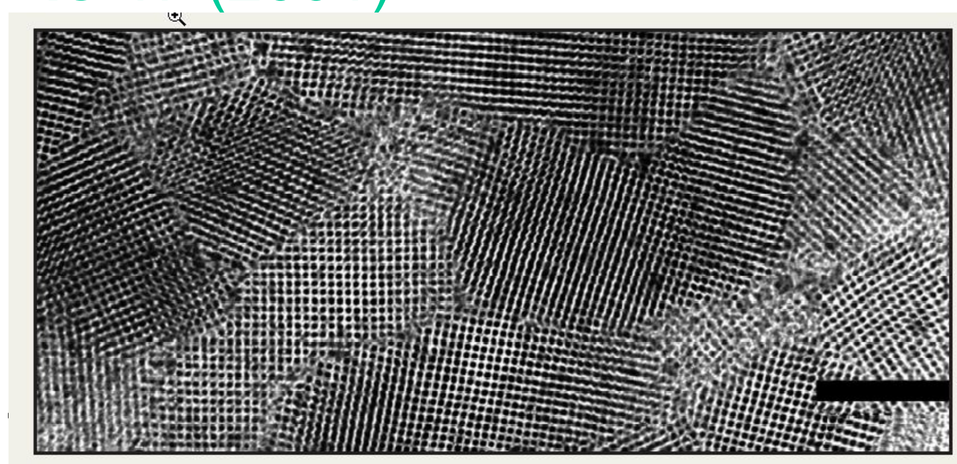


Chemical Synthesis of Nanocrystals



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

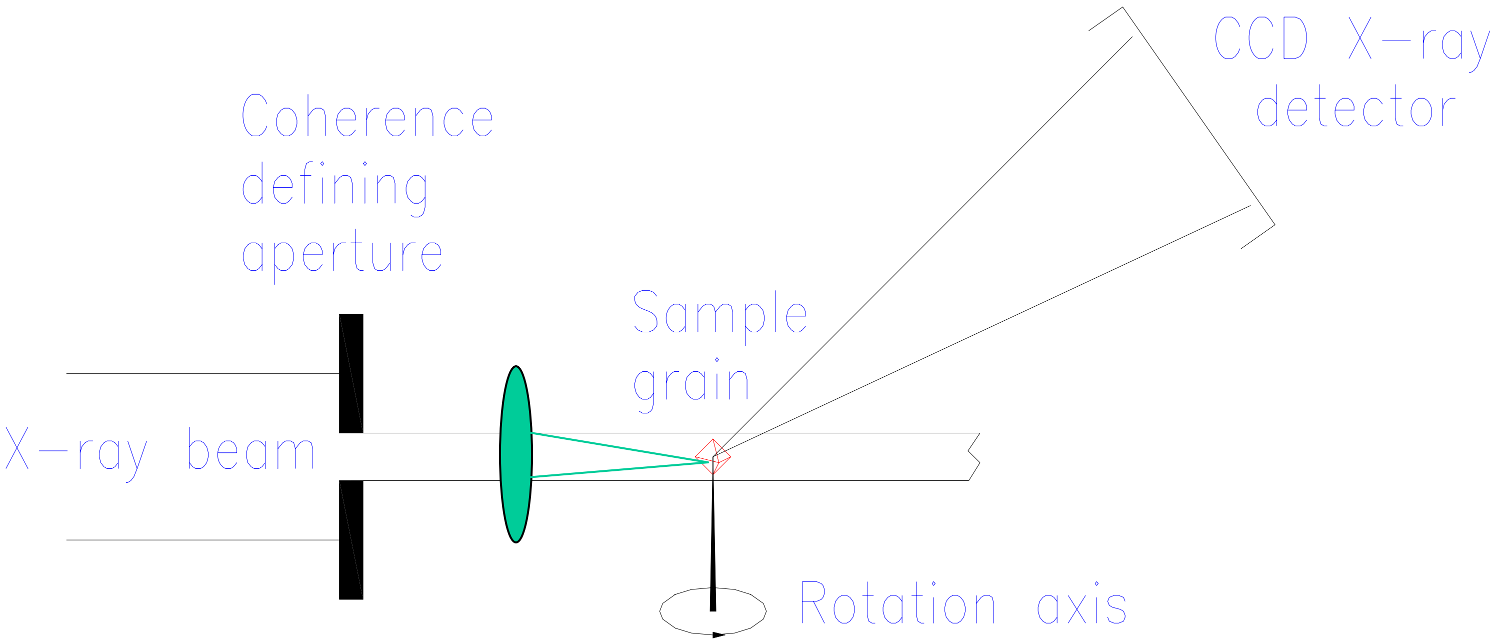
C. B. Murray, IBM J. Res. & Dev. **45** 47 (2001)



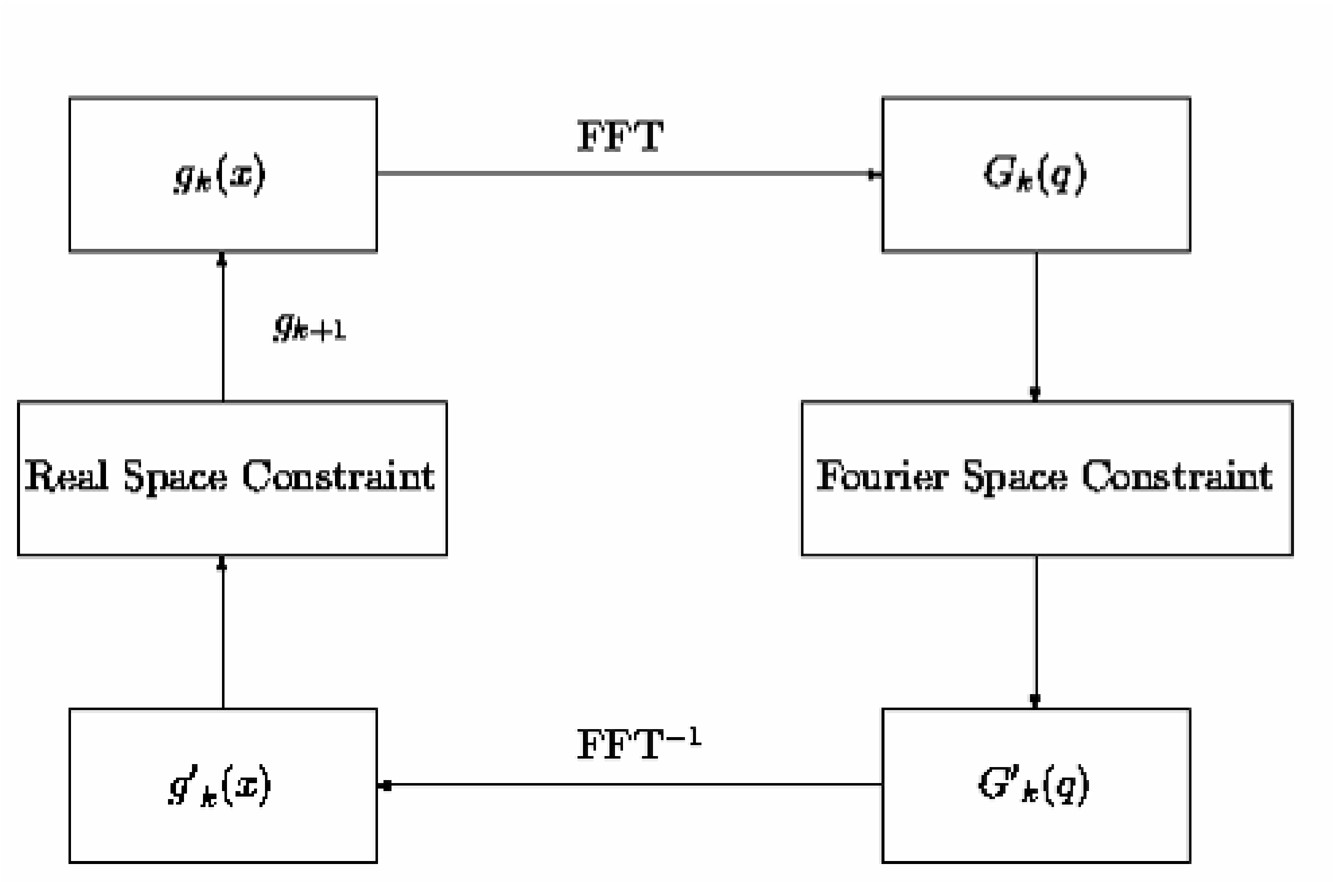
Lensless X-ray "Microscope"

APS ξ_{HOR}= 20µm, focus to 1µm

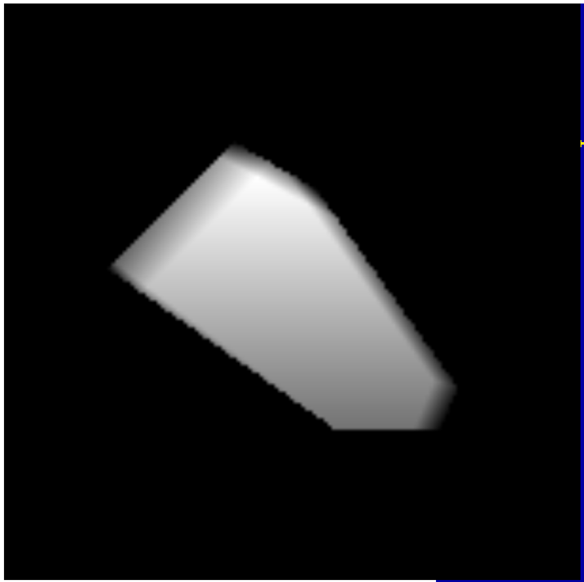
NSLS-II ξ_{HOR}= 500µm, focus to 0.05µm

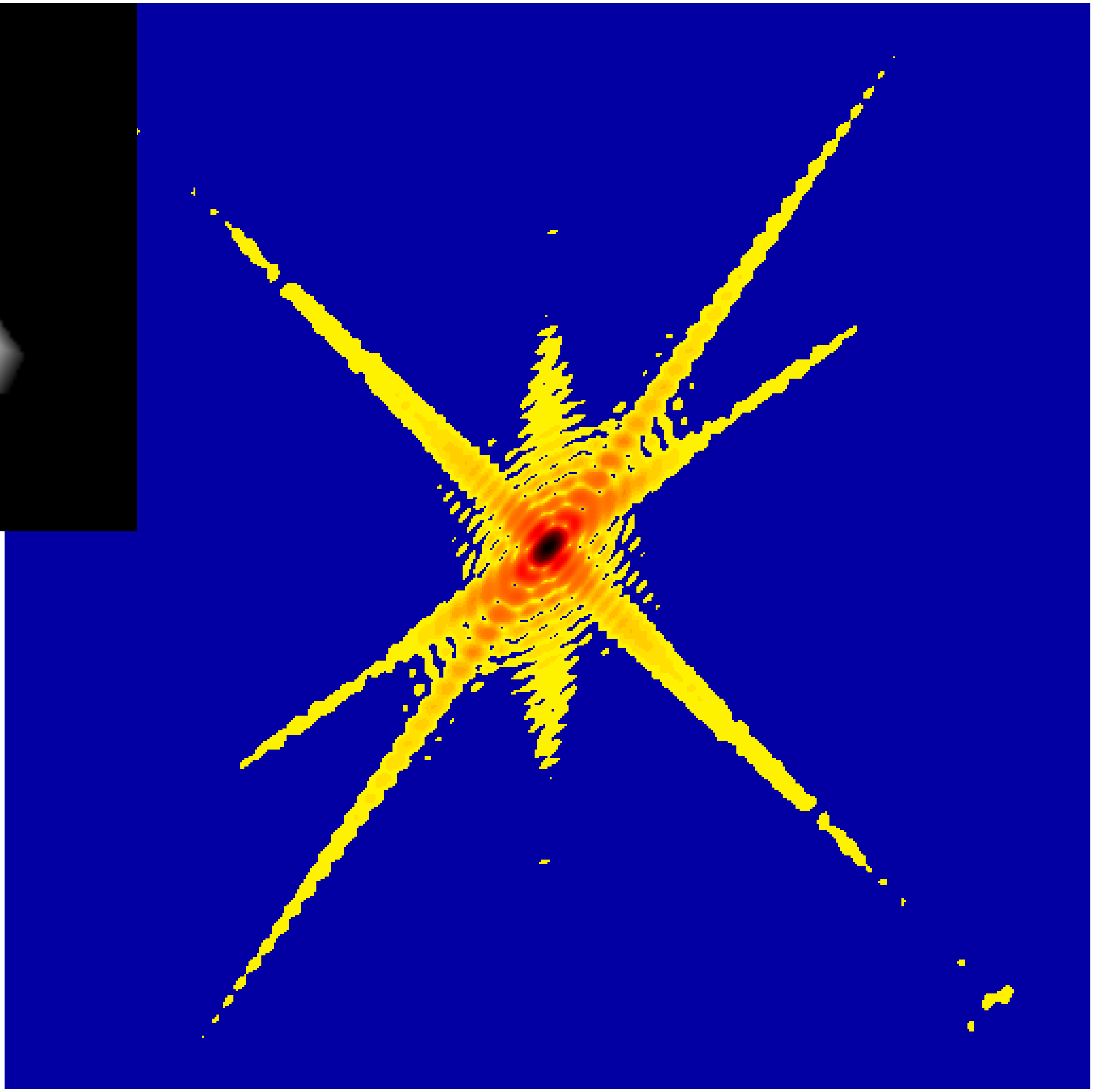


Generic "Error Reduction" method

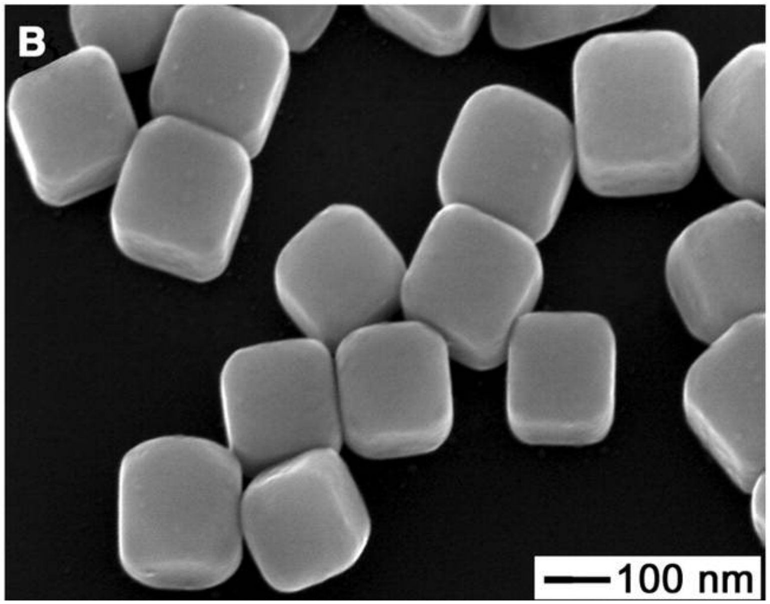


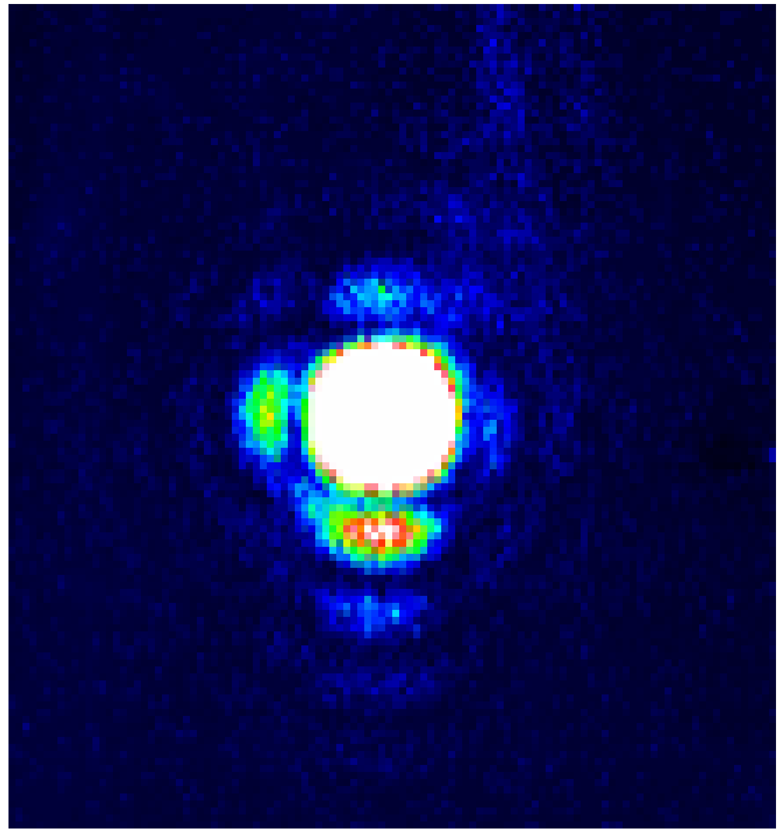
J. R. Fienup Appl. Opt. 21 2758 (1982)
R. W. Gerchberg and W. O. Saxton Optik 35 237 (1972)





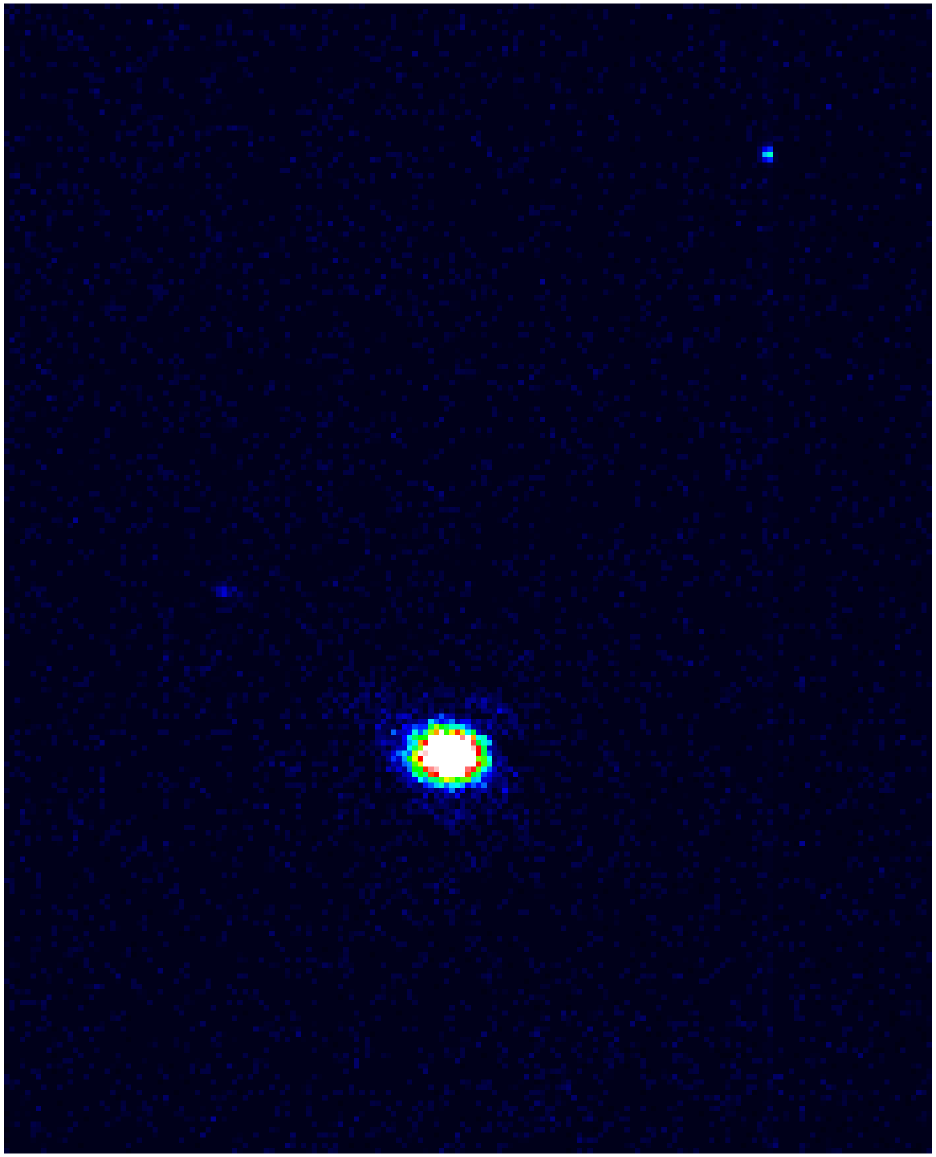
Chemically Synthesized Silver Nanocubes

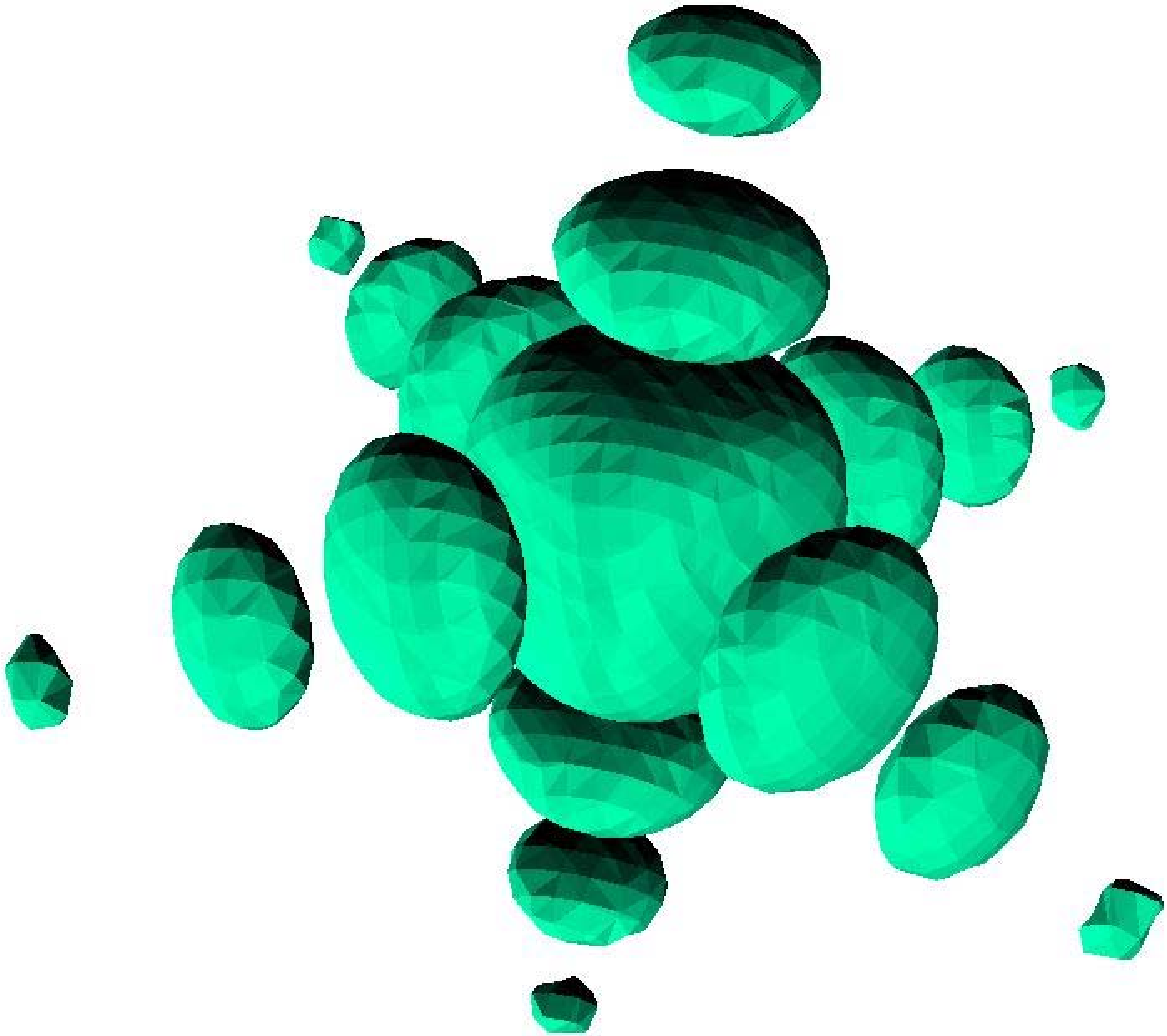


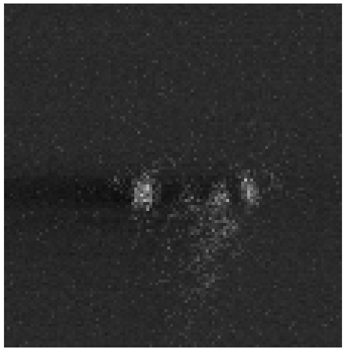


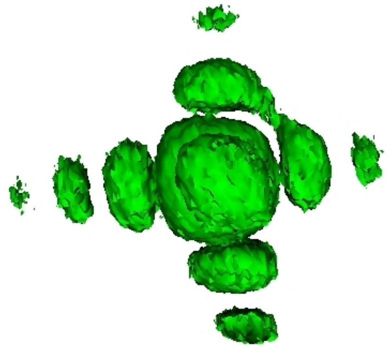
Yugang Sun and Younan Xia,
Science 298 2177 (2003)

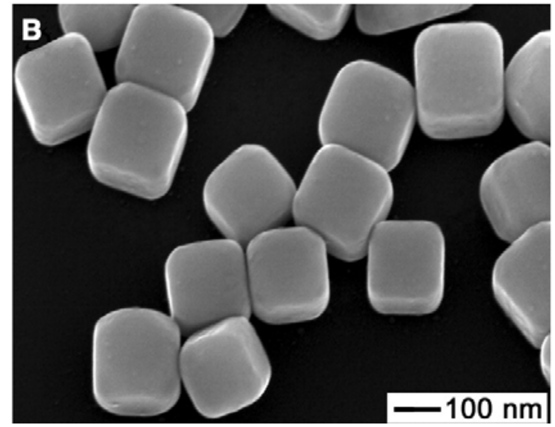
Rocking scan of Ag cubes with 0.01° steps

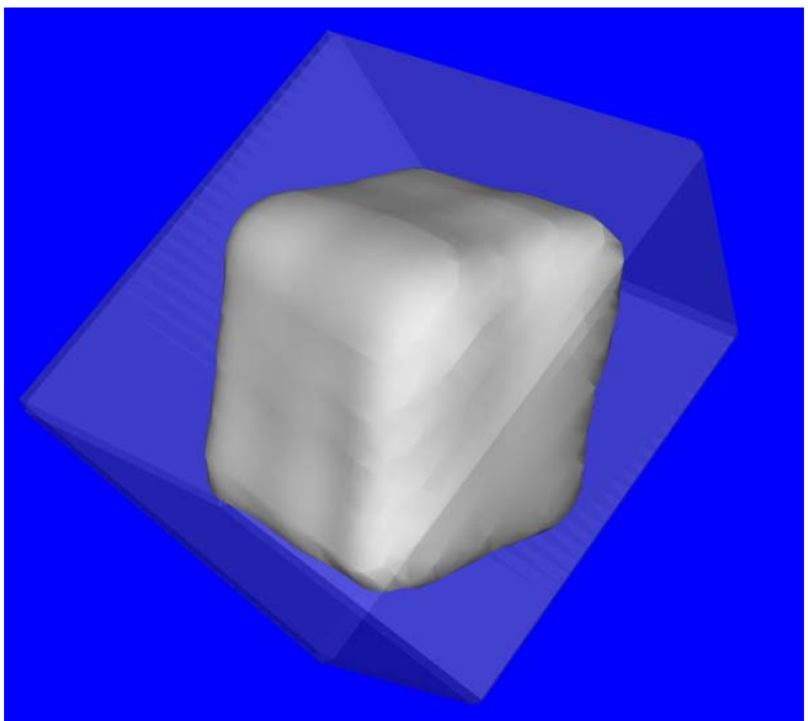


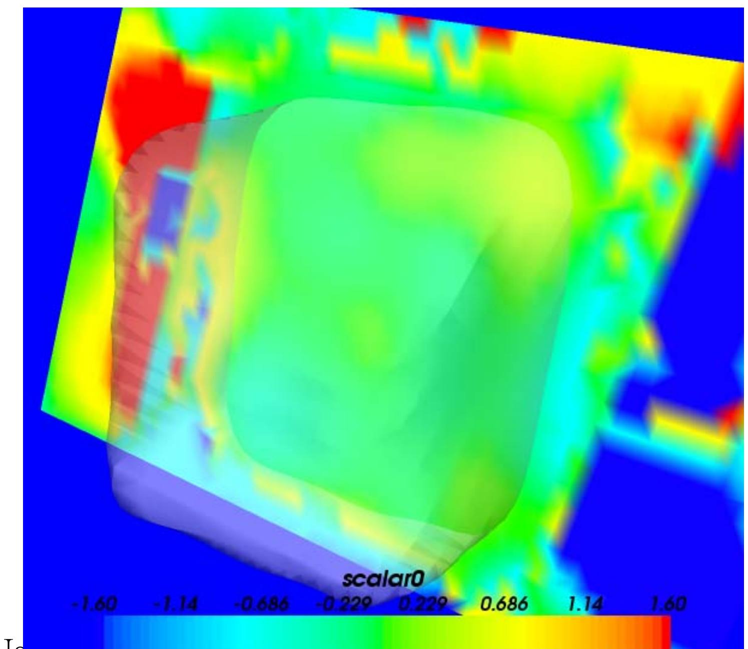






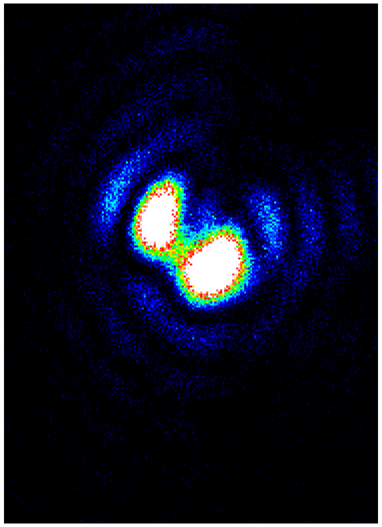

B
100 nm

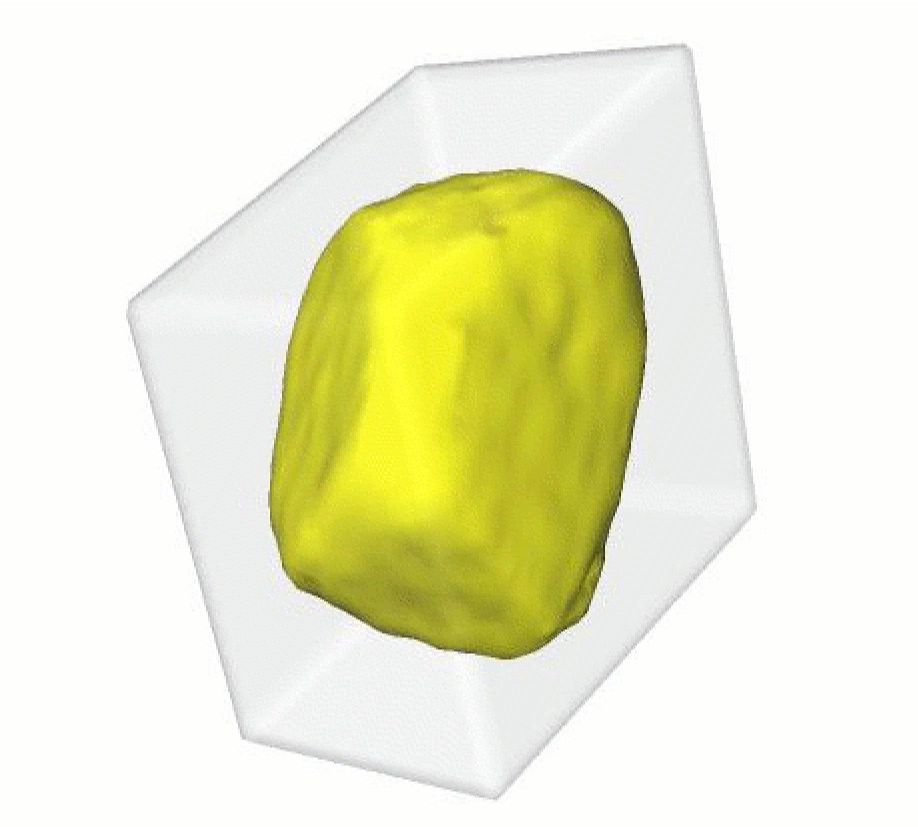



scalar0
-1.60
-1.14
-0.686
-0.229
0.229
0.686
1.14
1.60

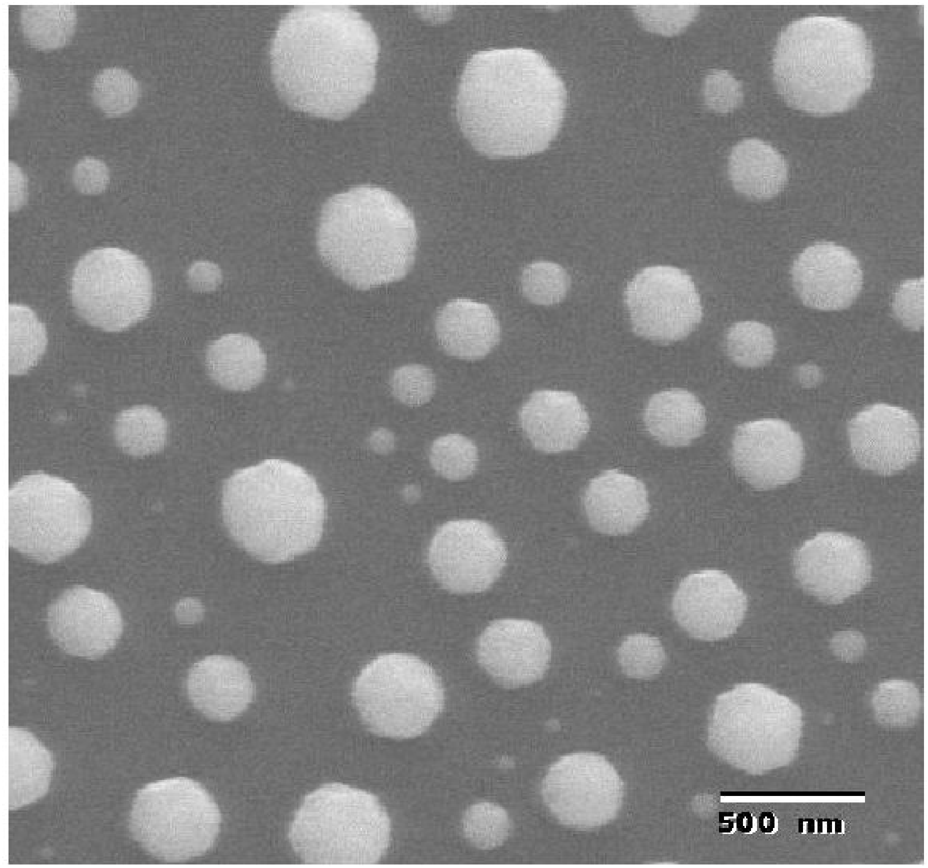
Gold nanocrystal reconstruction

showing support used for 20 HIO followed by 10 ER

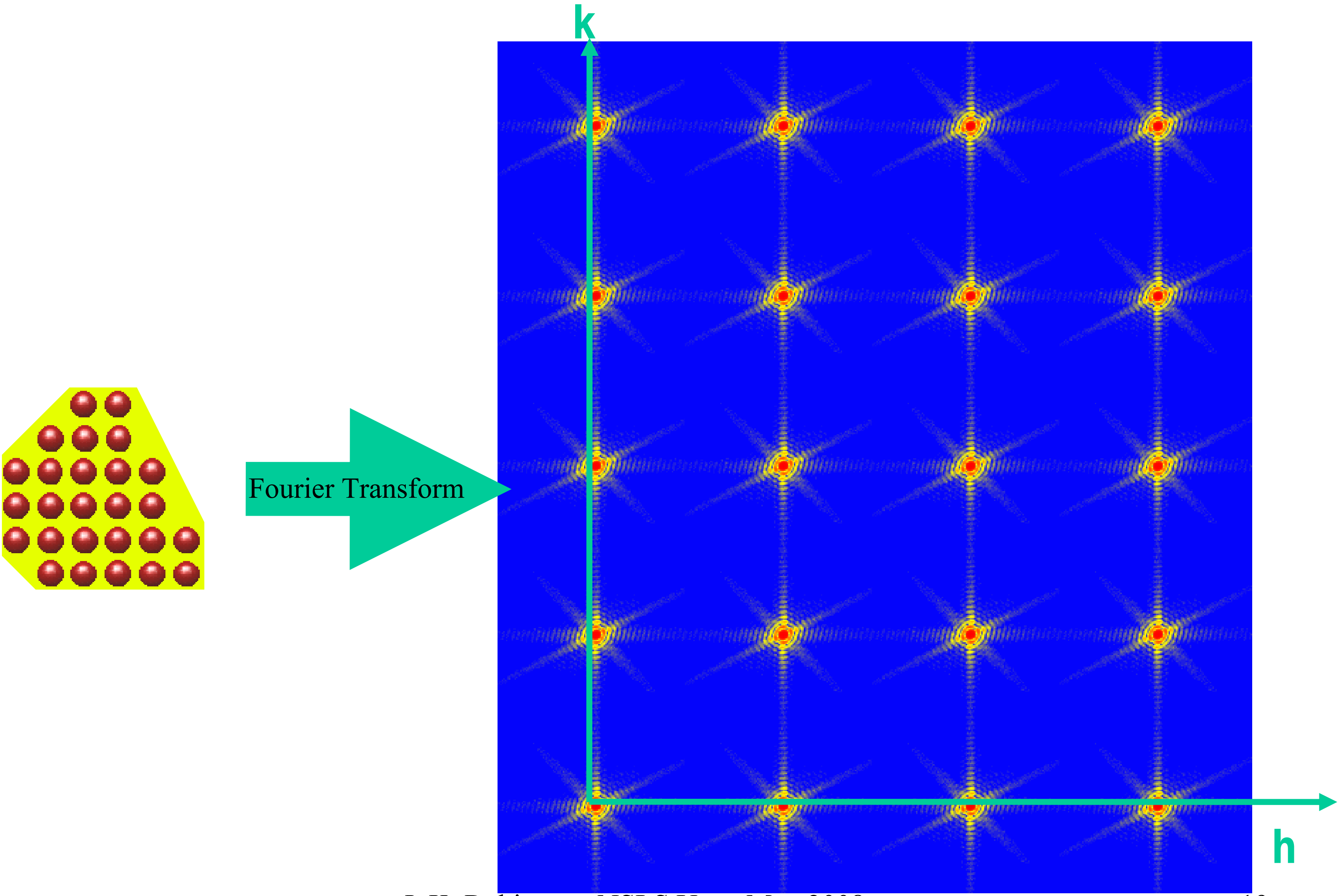




In situ growth of Pb crystals



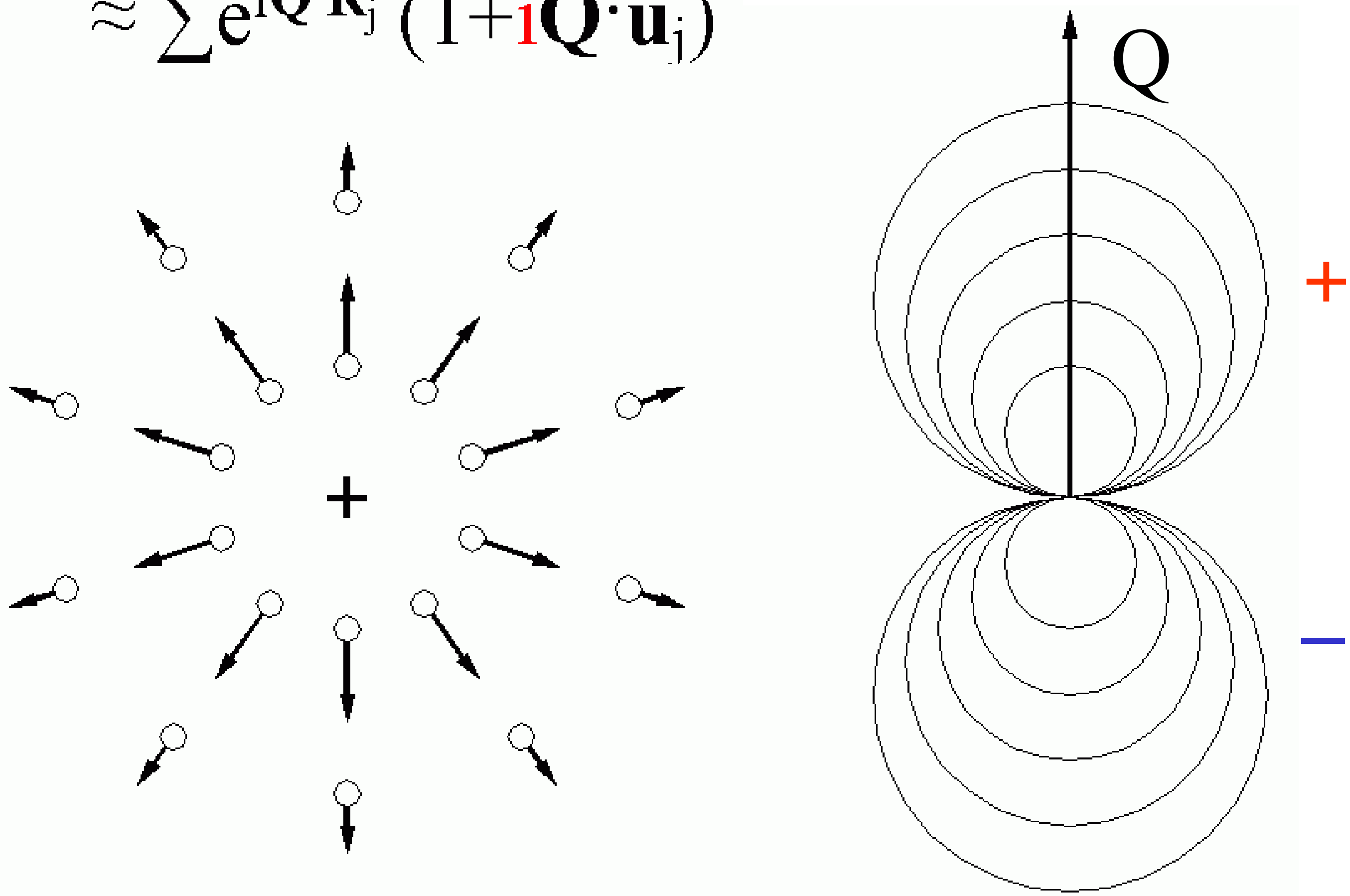
Coherent Diffraction from Crystals



Diffraction by Strain of Point Defect

$$A \sim \sum e^{i\mathbf{Q}\cdot(\mathbf{R}_j+\mathbf{u}_j)}$$
$$\approx \sum e^{i\mathbf{Q}\cdot\mathbf{R}_j}\,(1+i\mathbf{Q}\cdot\mathbf{u}_j)$$

Imaginary density



Good statistics, 3D diffraction data

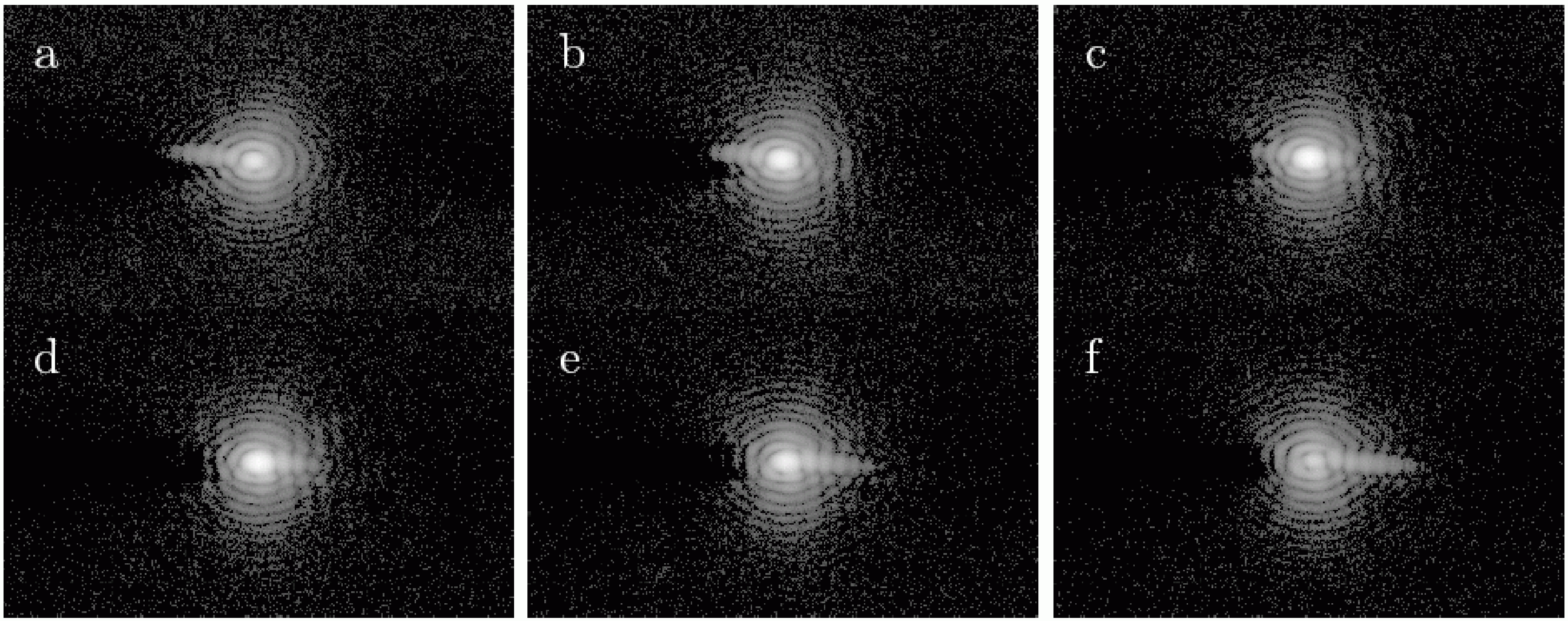
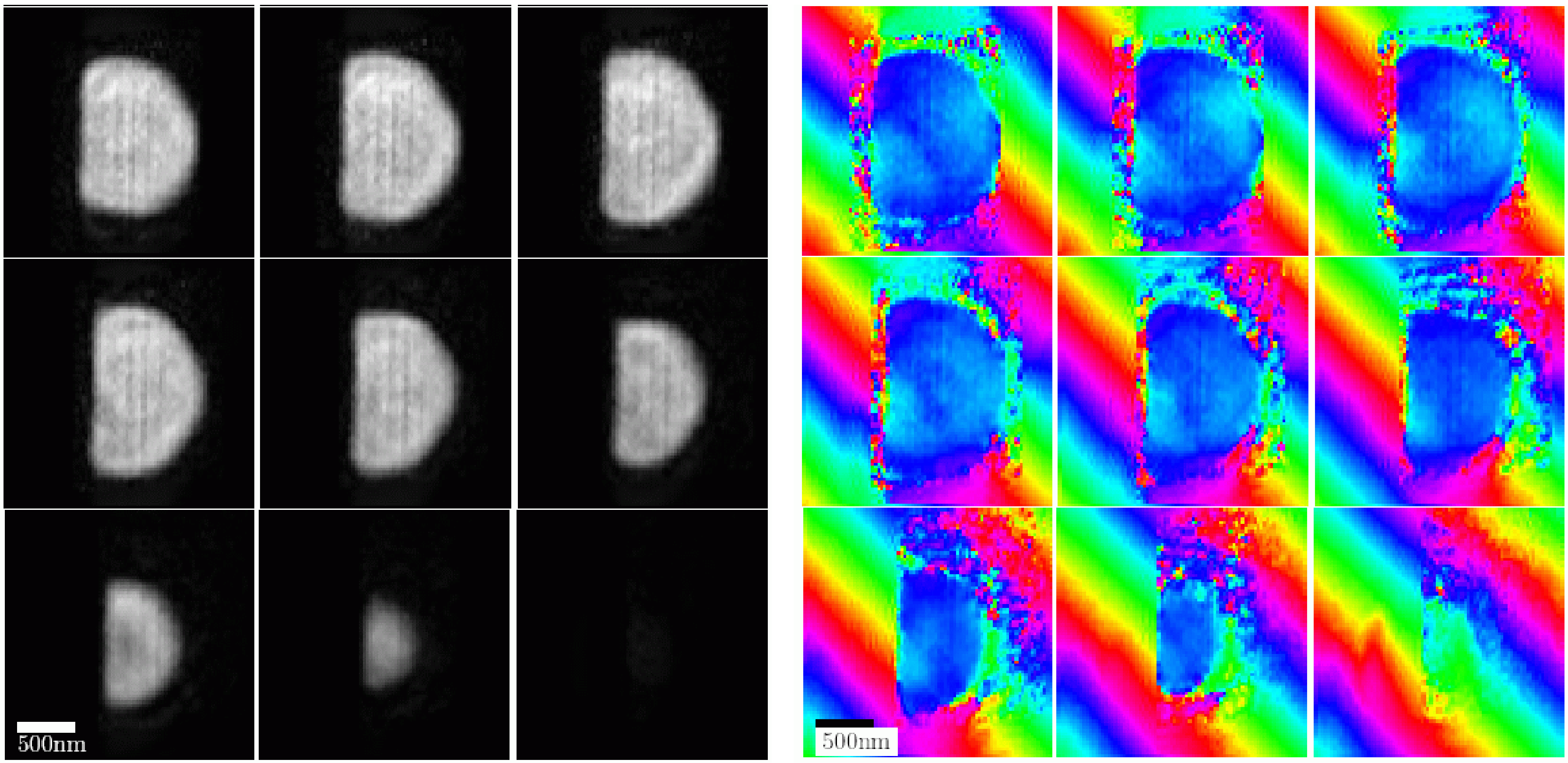
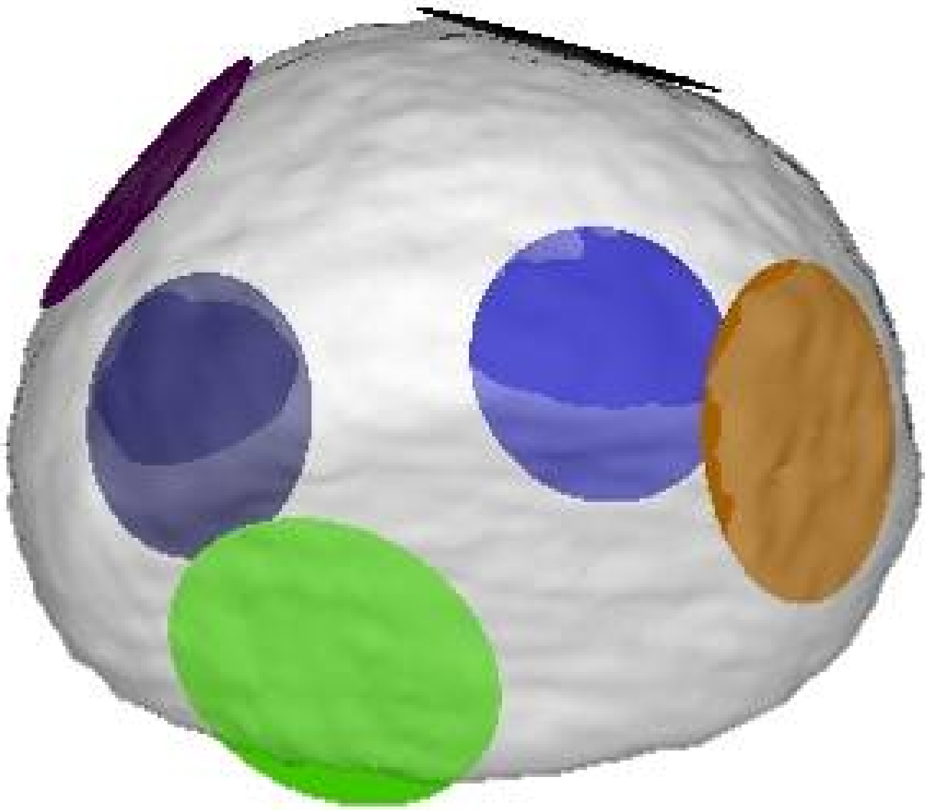


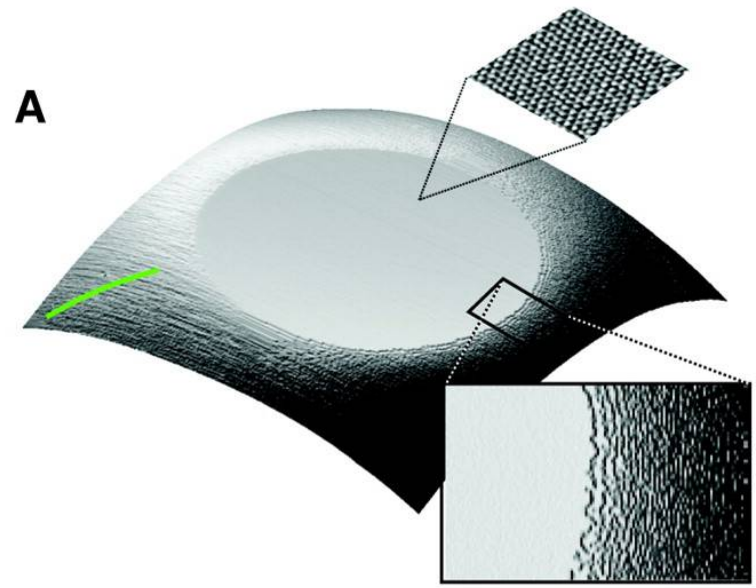
Figure 4.12: Center slices from 3D CXD pattern from Pb sample, on a log scale. Data file 296 from 10/03.

Complex Density (amplitude *and* phase)



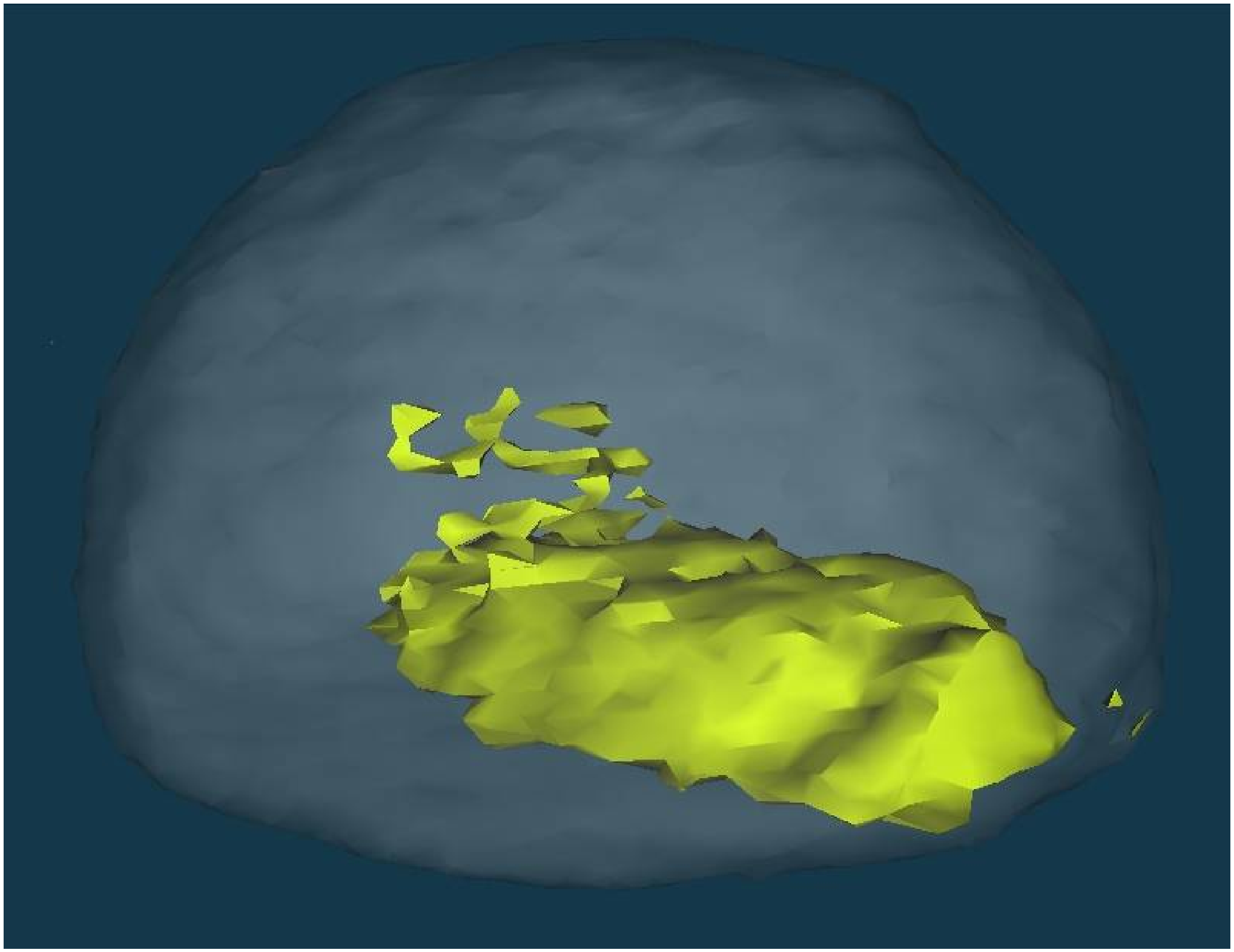
Facets of Equilibrium Crystal Shape



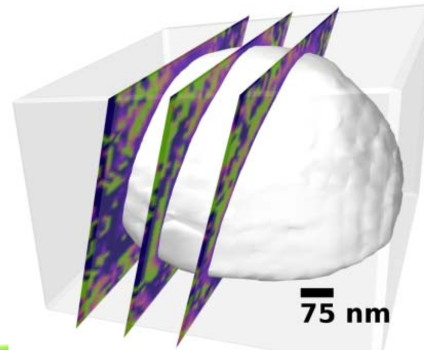


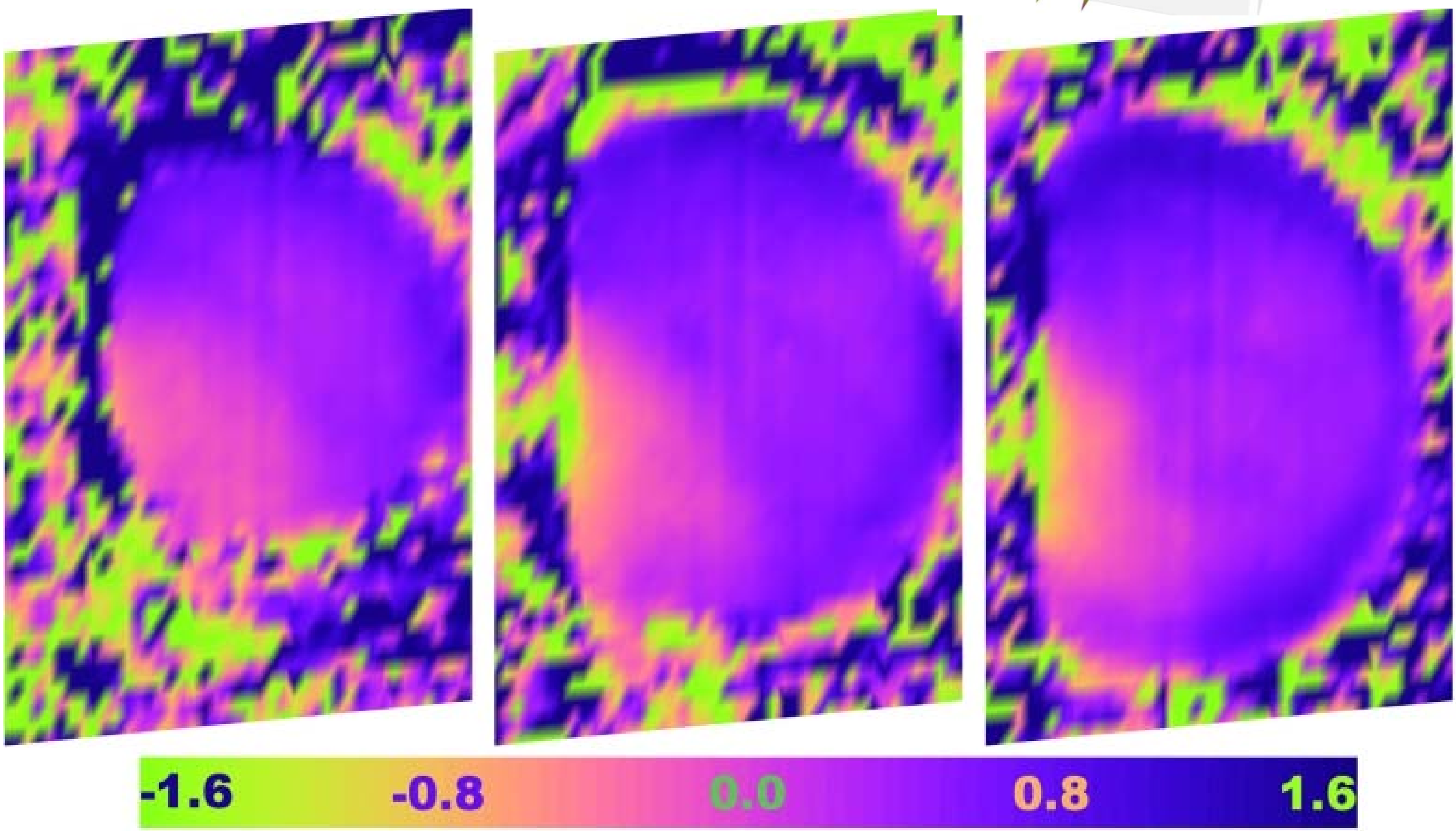
Thurmer K, Williams E, Reutt-Robey J
Science **297** 2033 (2002)

Modeling of 3D Phase Bump

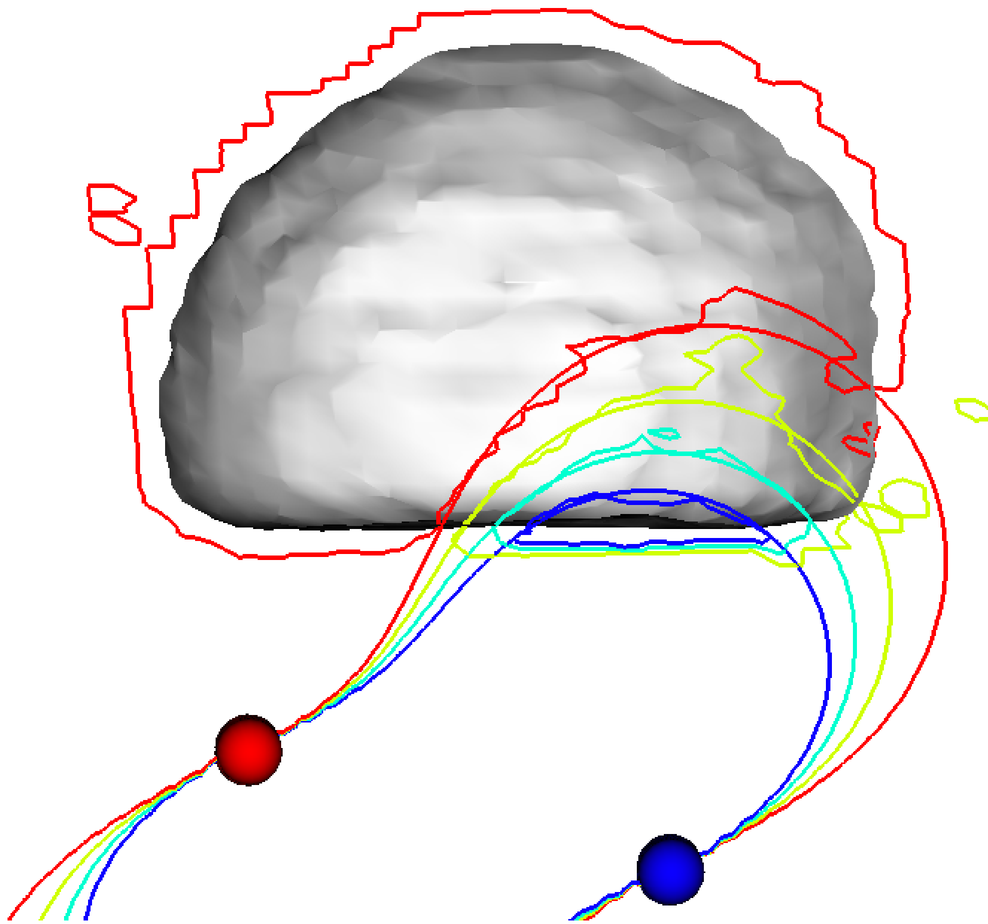


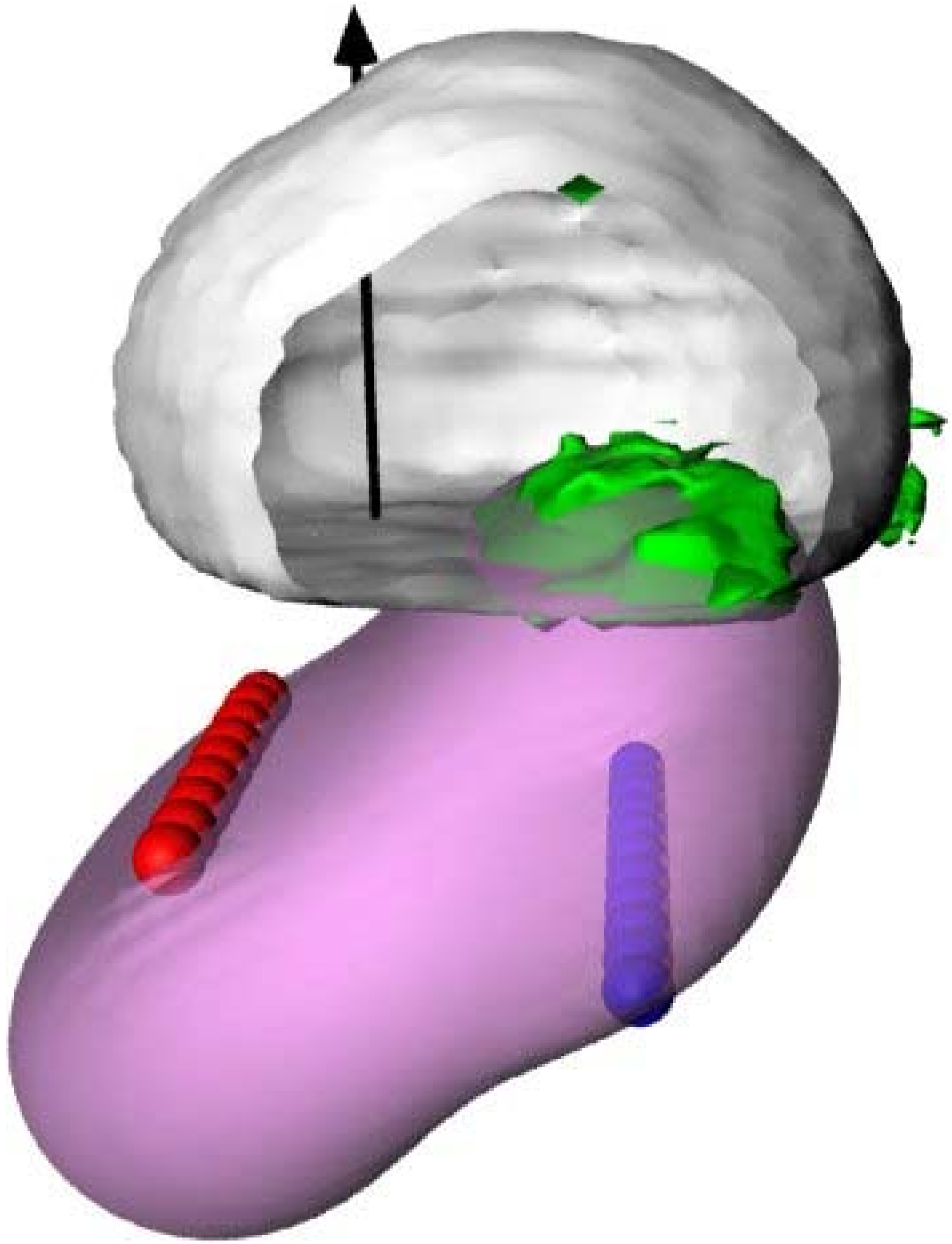
3D phase map sections





Field lines of "Point Charges"





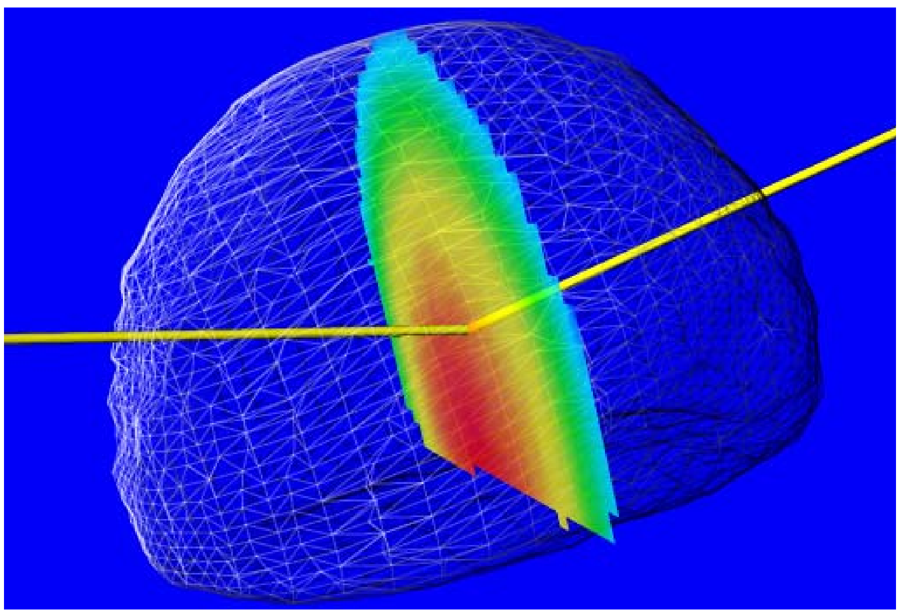
Refraction effects in Lead at 8.9keV

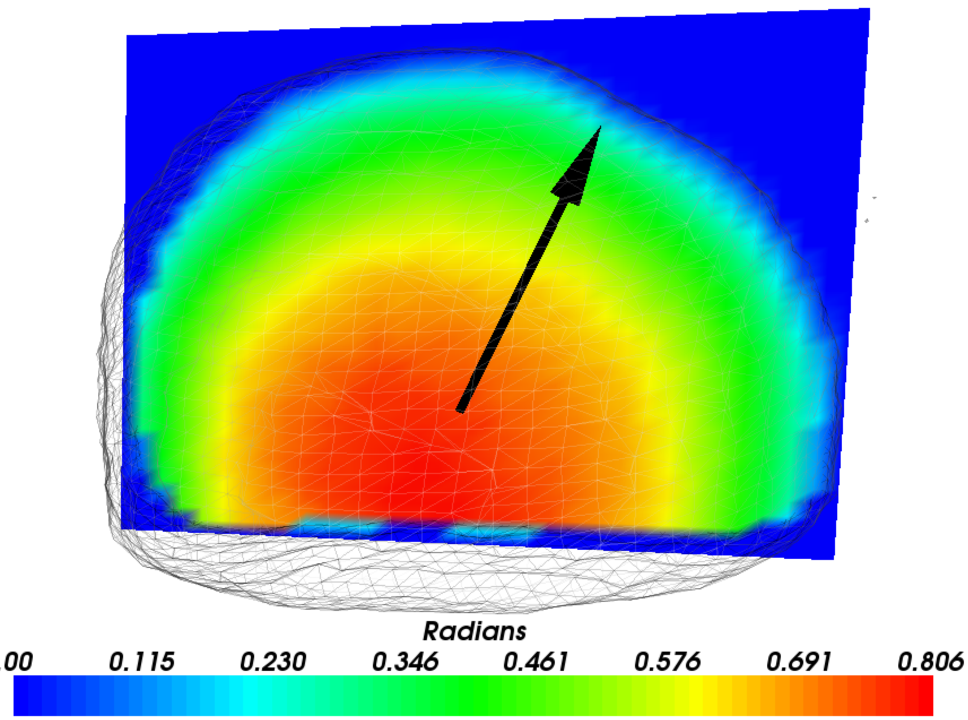
Phase accumulation due to refraction along scattering path

d=750nm: $kd\delta = 0.76$rad

$kd\beta = 0.07$

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





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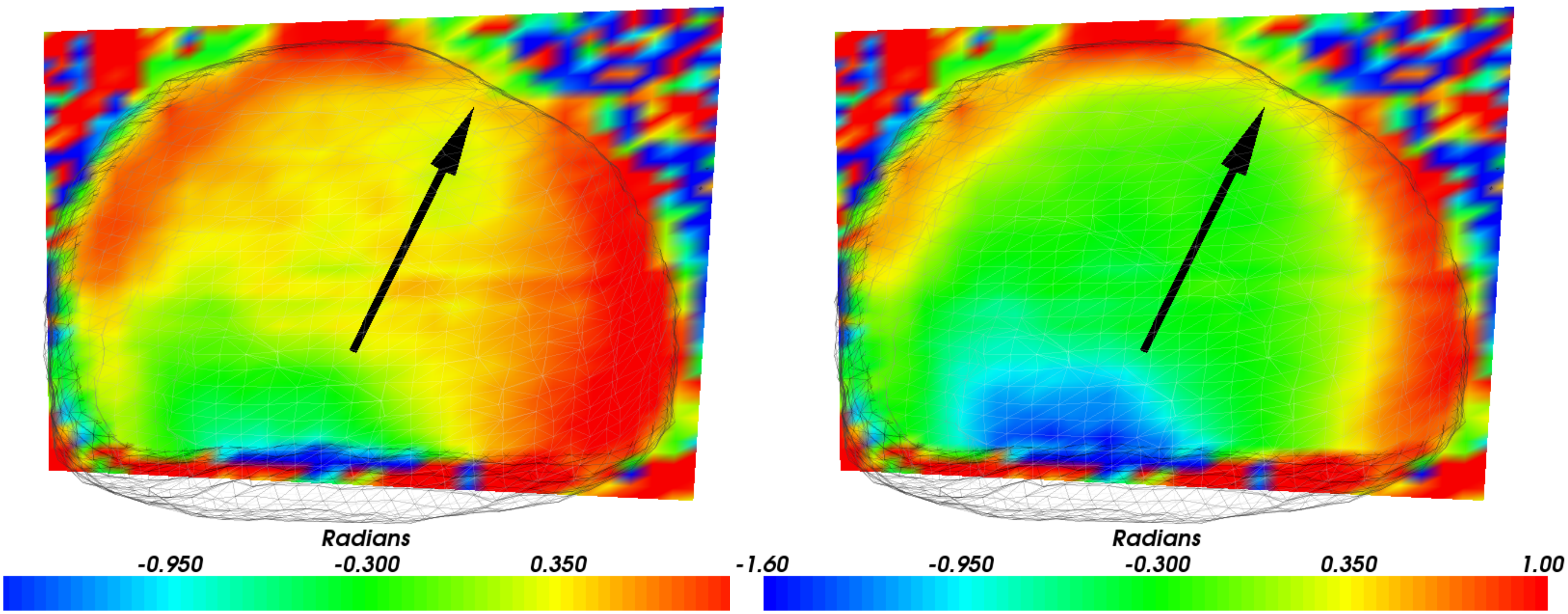
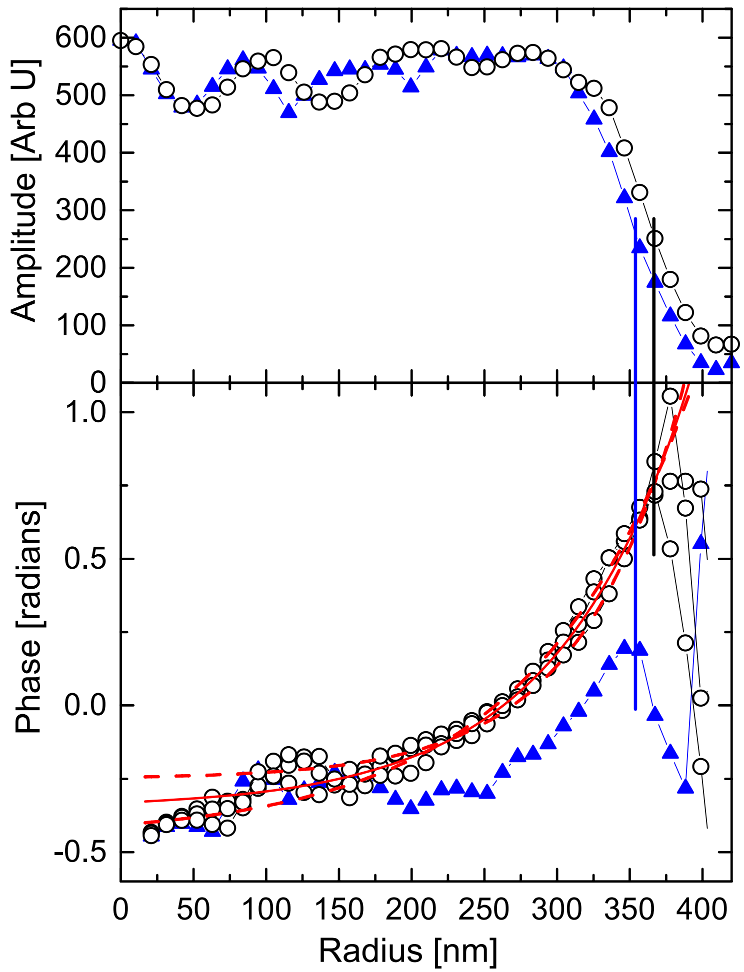
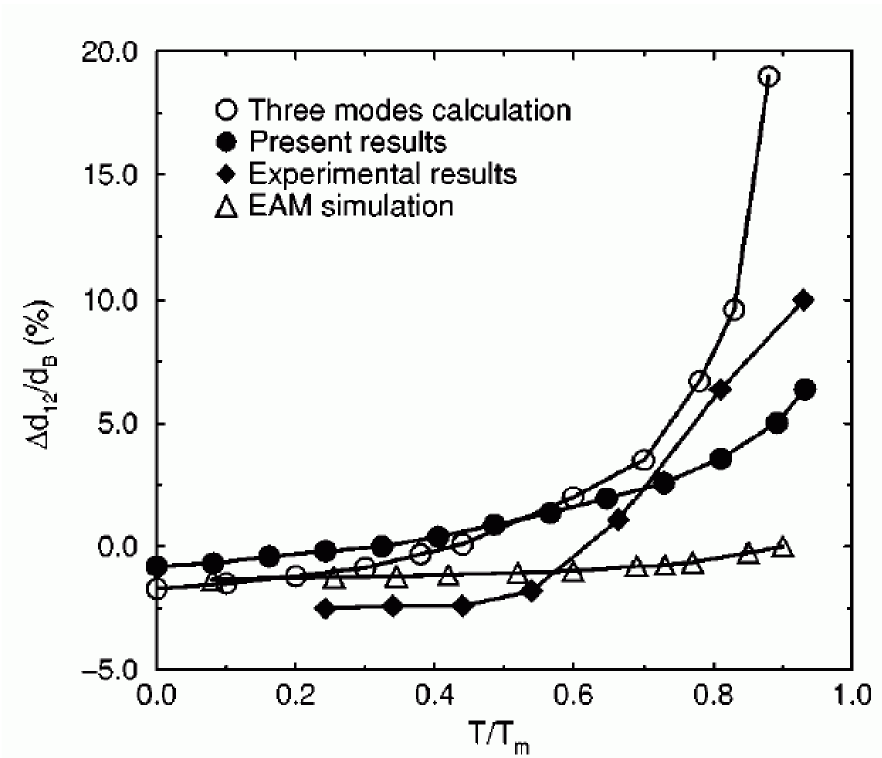
Phase Maps before and after Correction



Surface Thermal Expansion

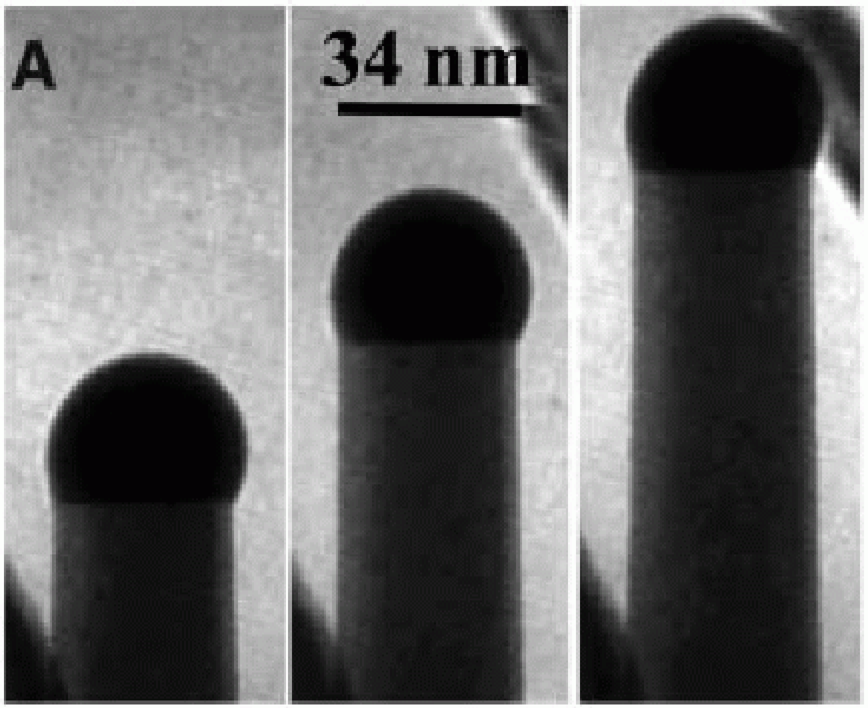
Ag(111): Scheffler et al PRB <u>59</u> 970 (1999)

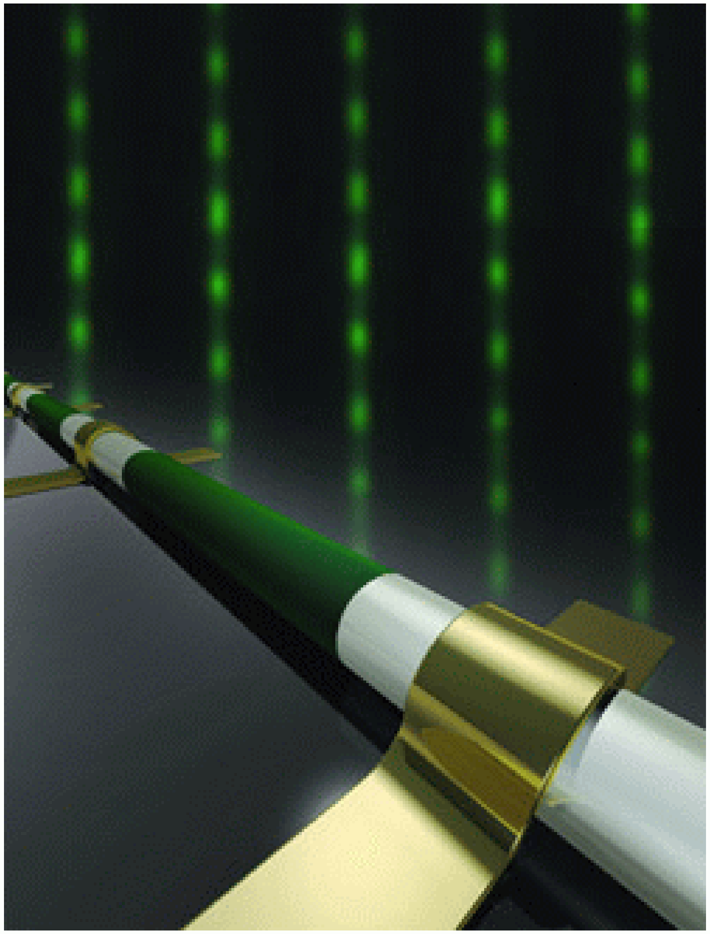




VLS growth of nanowires

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

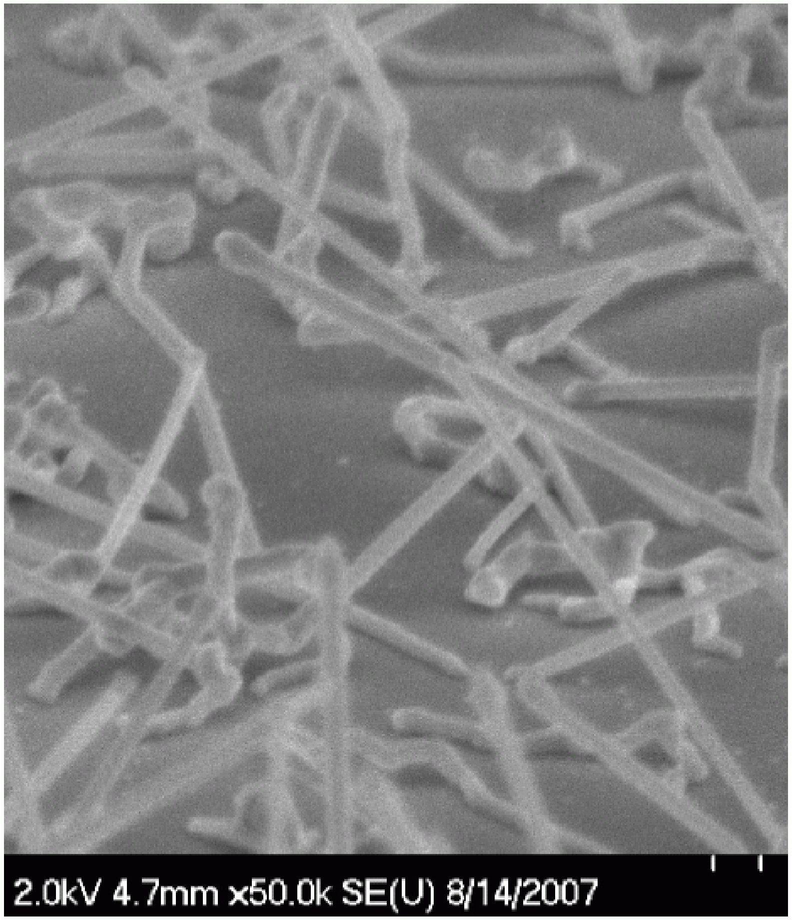




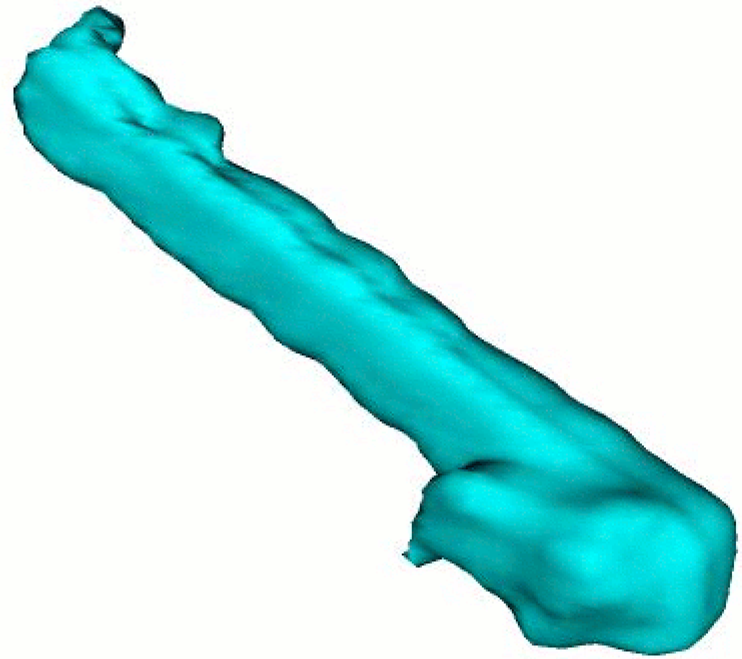
NiSi/Si nanowire heterostructure devices. *Nature* **430**, 61 (2004).

Reconstruction of InP nanowire

CVD on Si, Suneel Kodambaka, UCLA

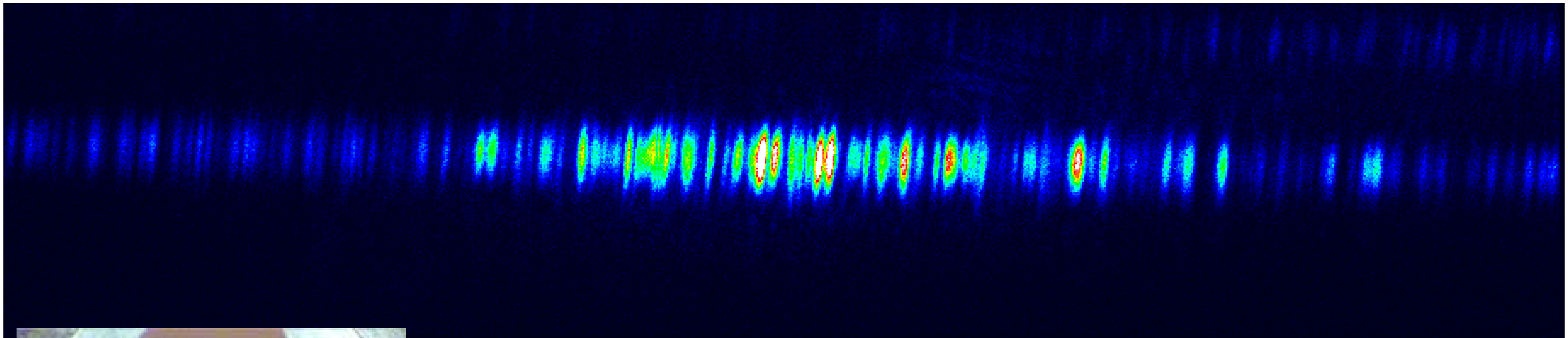


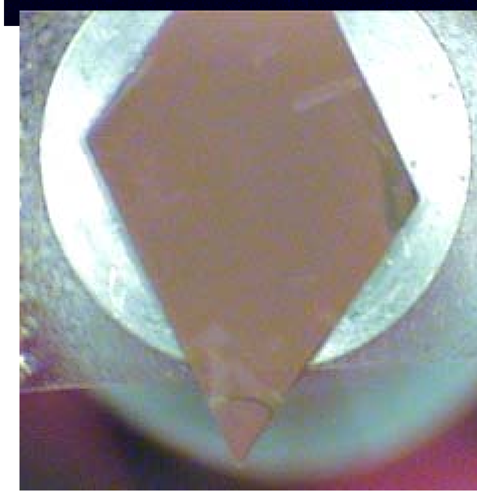
InP nanowires grown on Si (111)

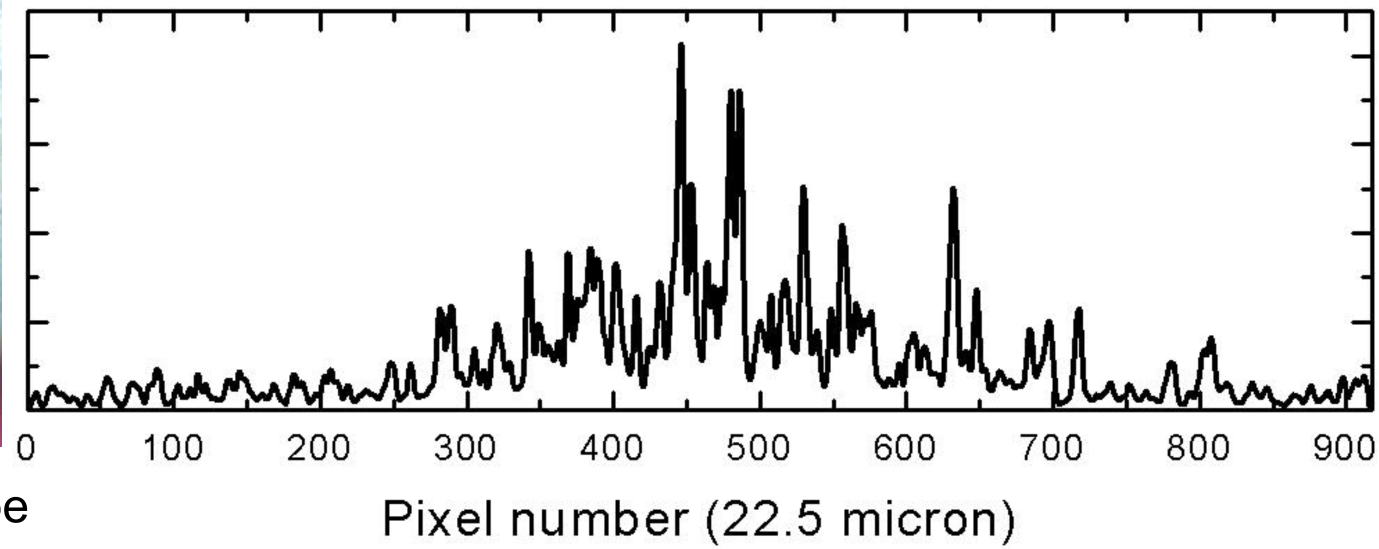


GaAs Nanowire “Barcode”

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



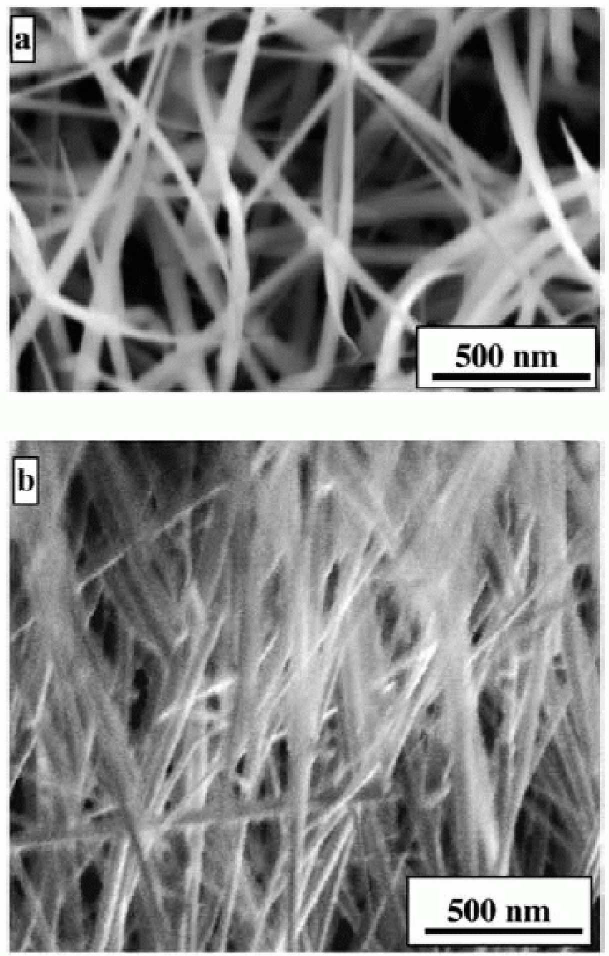


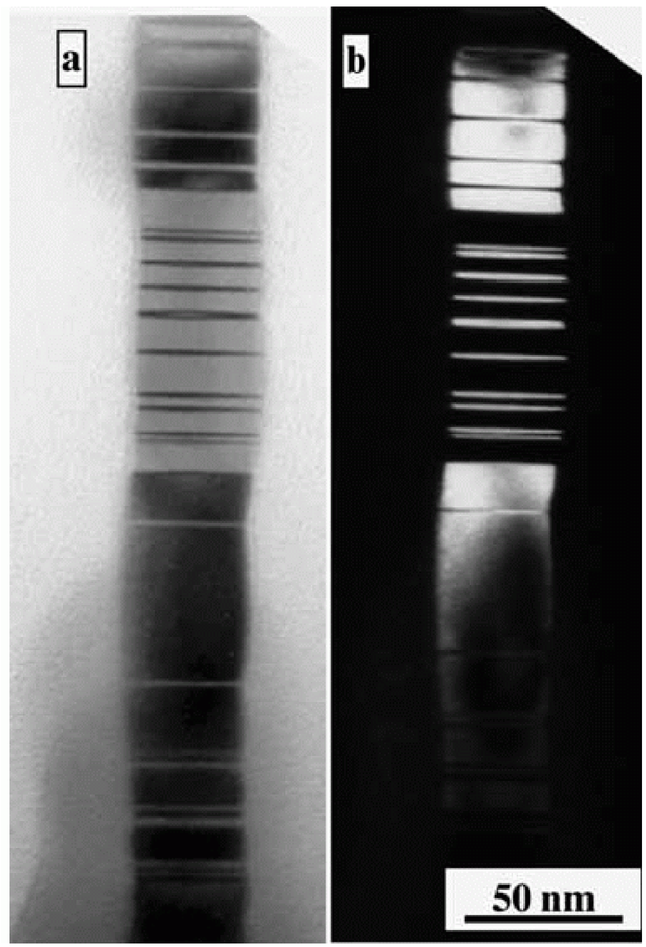


GaAsNW1106-22.spe
B9348 from Philips

Dark Field TEM of GaAs Nanowires

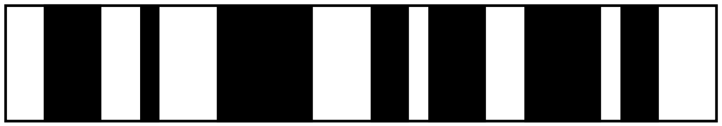
R. Banerjee et al, Phil. Mag. Lett. 86 807 (2006)



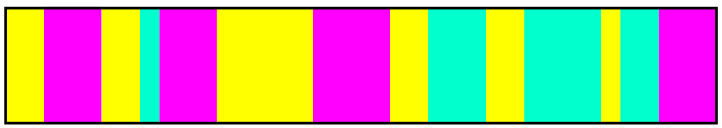


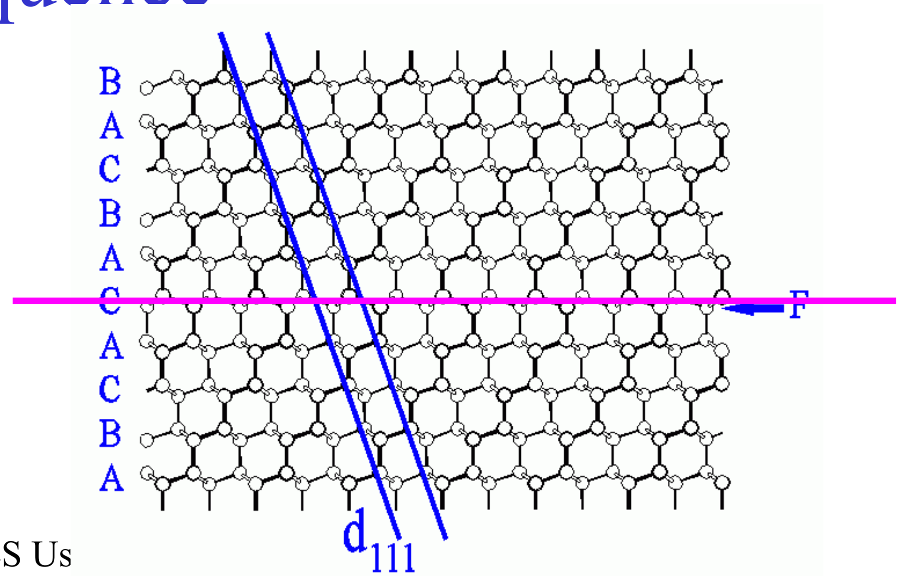
Models of Barcode Diffraction

(111) wires at (11-1) reflection



- Twinned stacking sequence

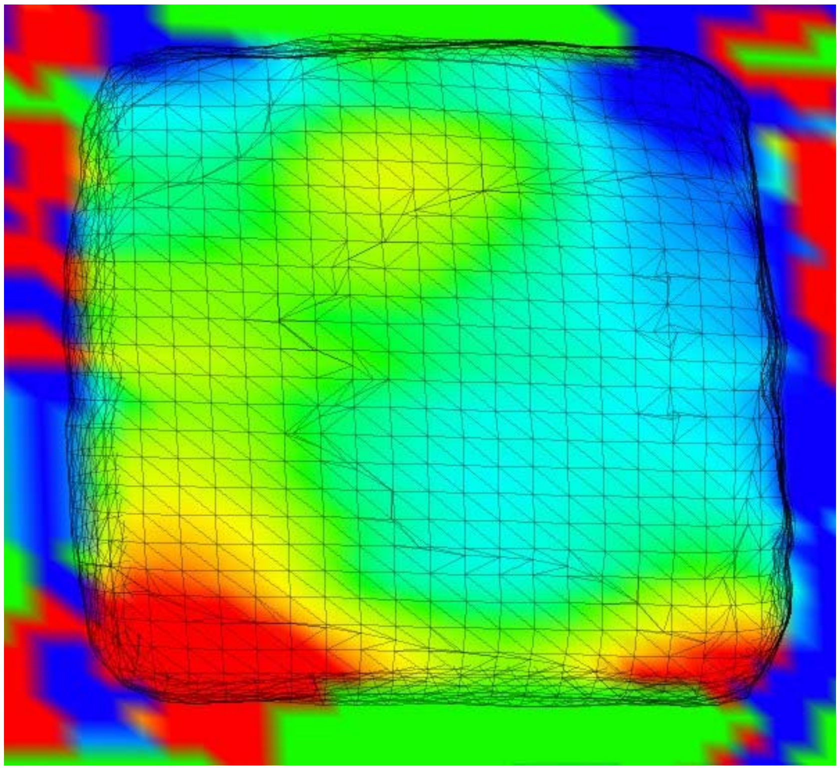


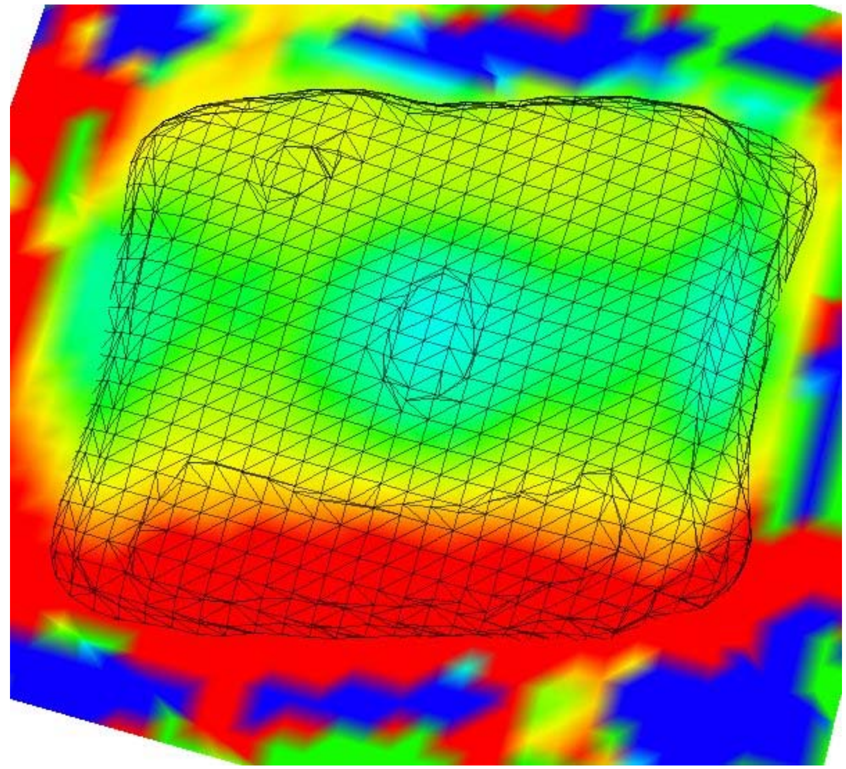


- Deformation faults

Contact strain of Zeolite ZSM-5

with Hyunjung Kim and K. B. Yoon at Sogang University



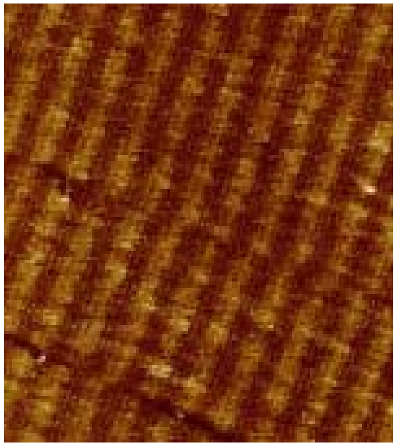


CXD applied to collagen
Felisa Berenguer, LCN

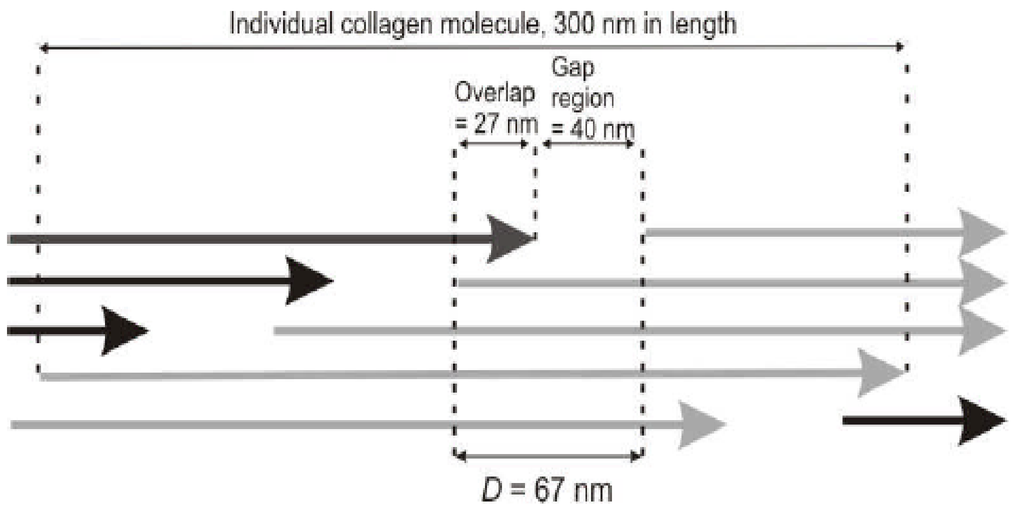
• Collagen is the most common protein in animal tissue (bones, teeth, tendon, cartilage, connective tissue)

• Potential applications in medicine (artificial bone, skin diseases)

• Collagen packing to built-up fibres is not completely understood
Different proposed models by Orgel 2007, Wess 2006, Bozec 2007 …

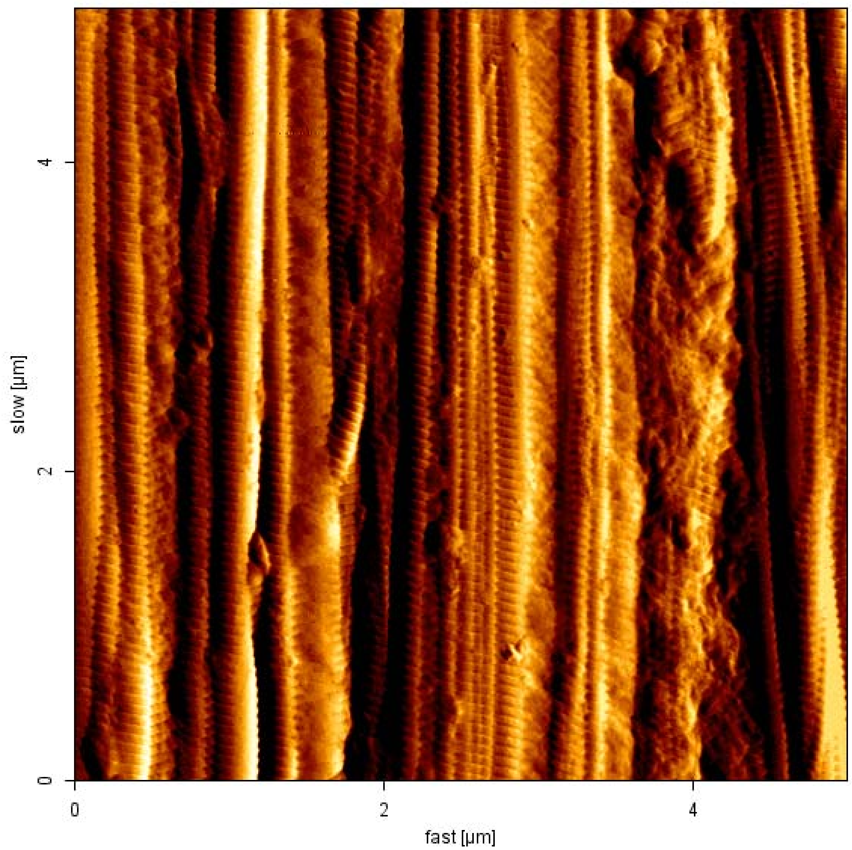


SEM [Cisneros, 2006]

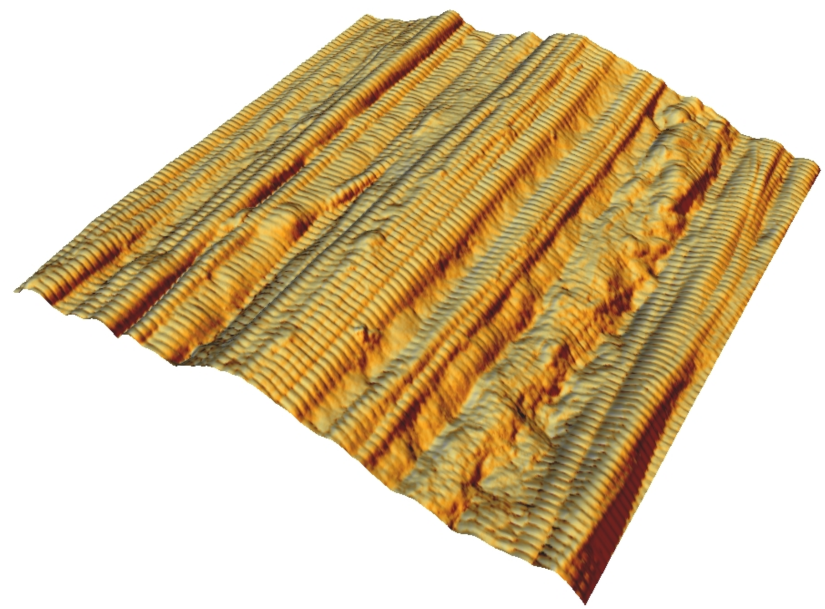


[Hodge and Petruska, 1976]

AFM imaging of rat-tail collagen



Dehydrated rat tail tendon tissue



Strong D-banding with 67 nm periodicity:

→Diffraction pattern with strong meridional maxima at multiples of 1/67 nm^{-1}

I. K. Robinson, NSLS User, May 2008

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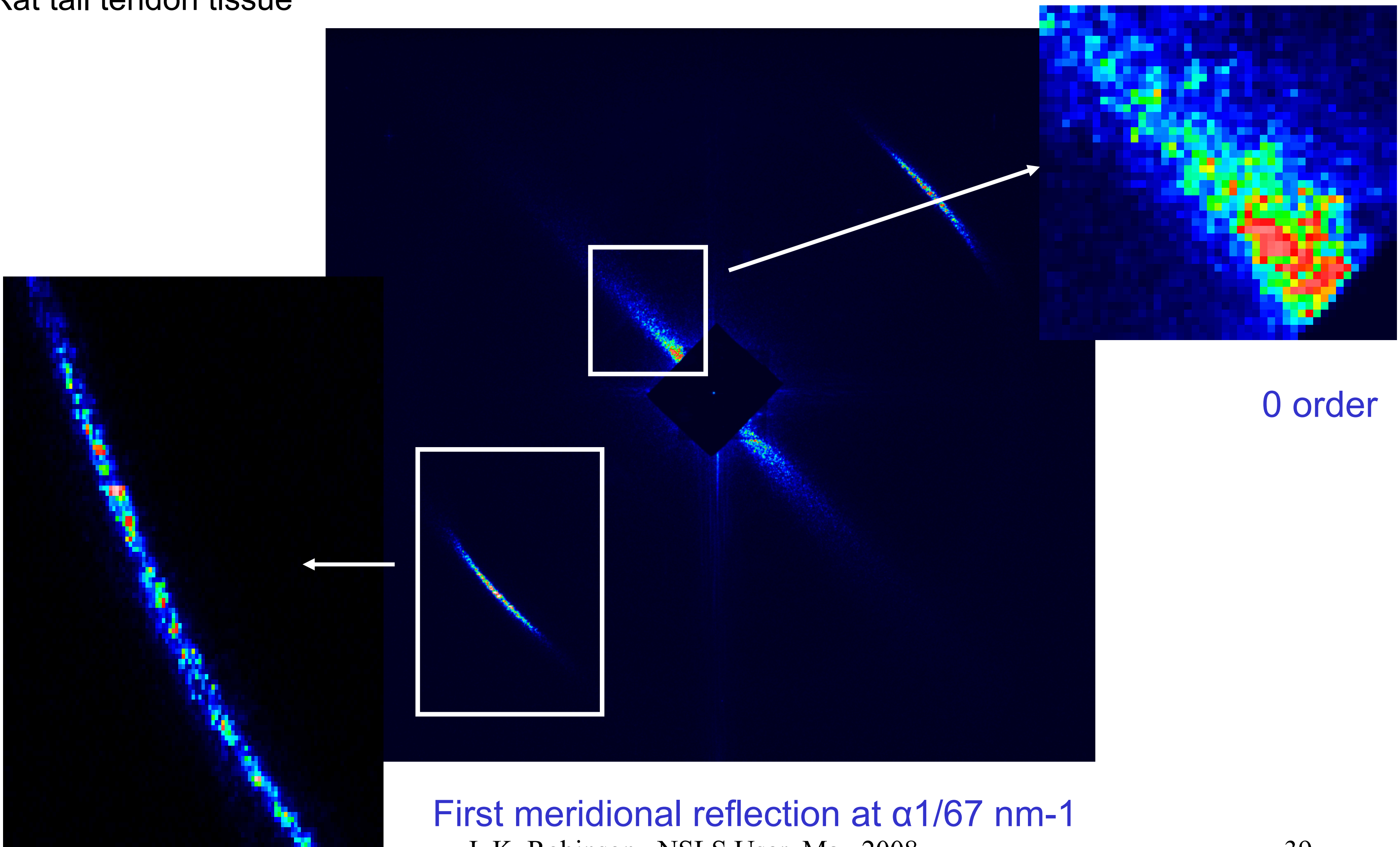
First experimental results

Rat tail tendon tissue

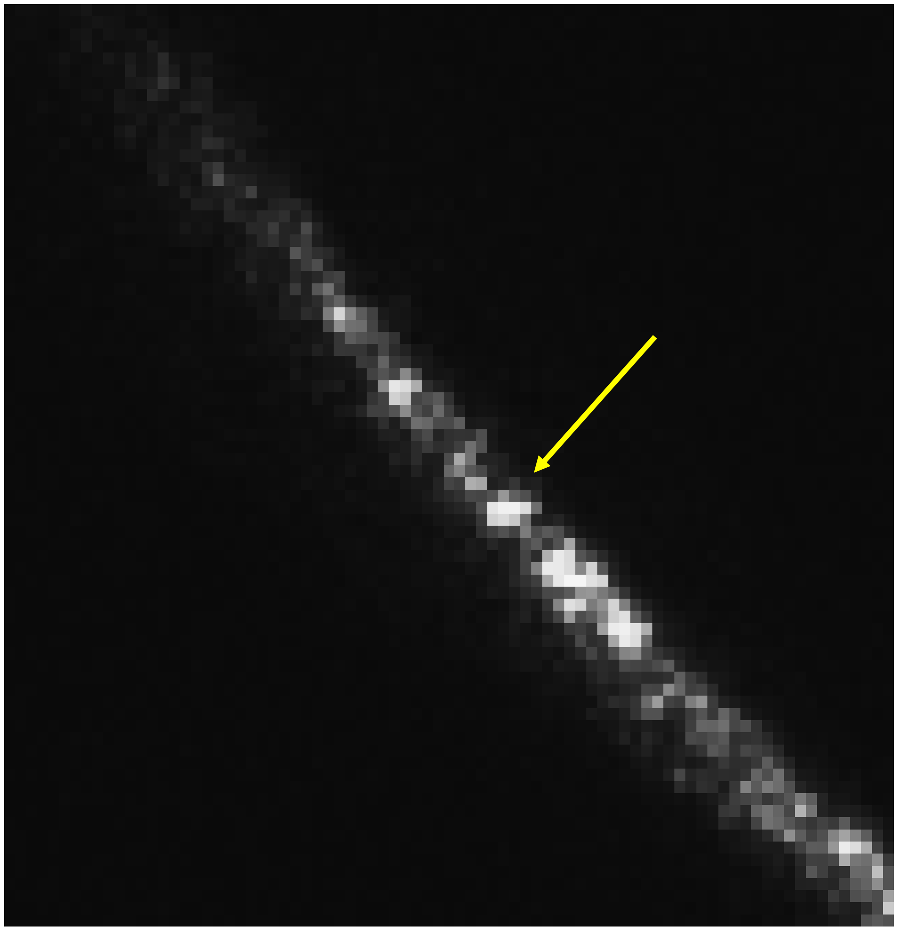


0 order

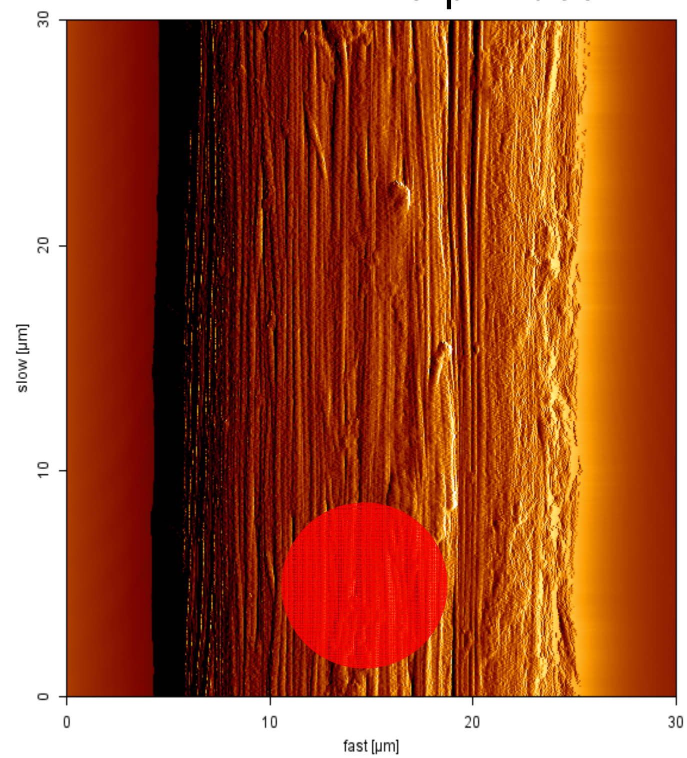
First meridional reflection at α1/67 nm-1

X-ray Ptychography

First meridional reflection



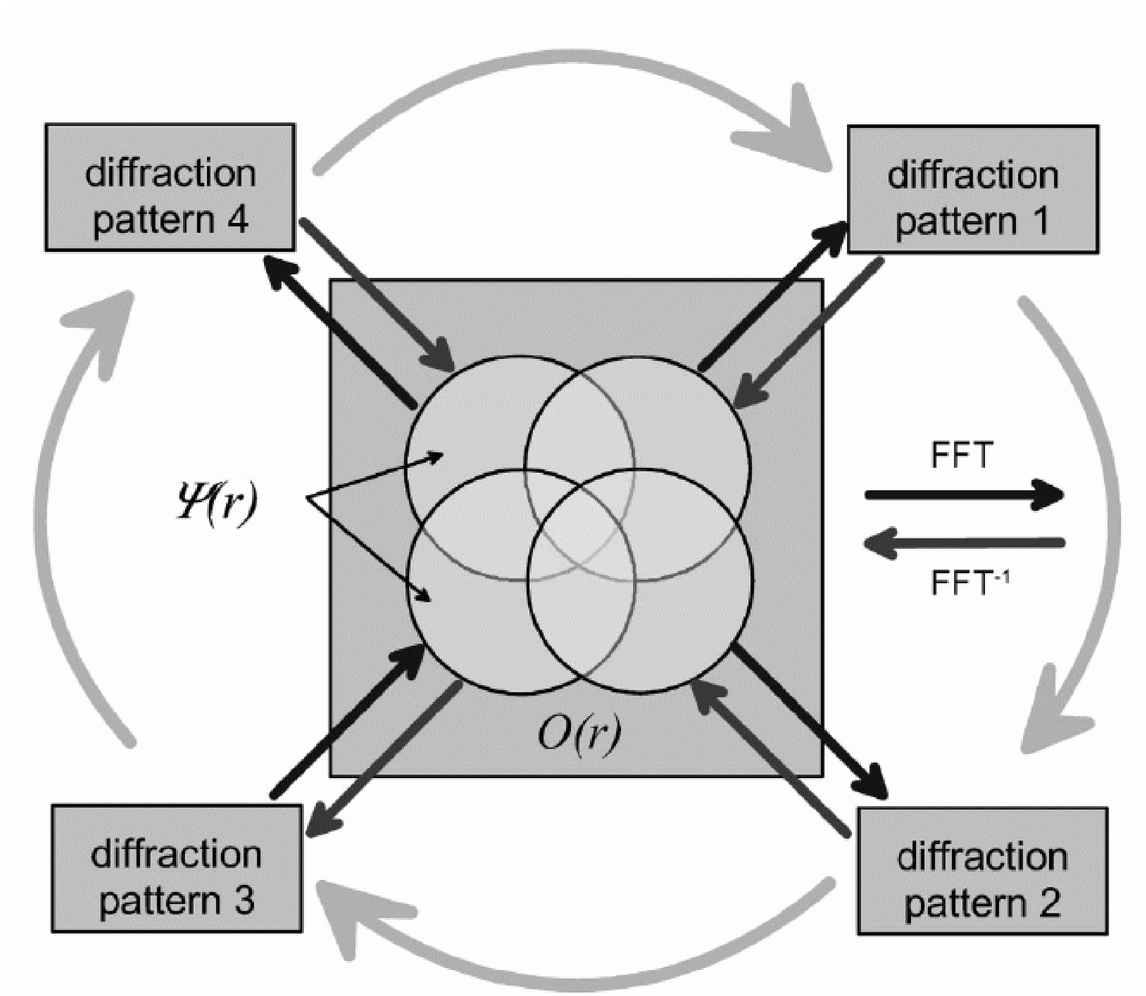
10 µm beam

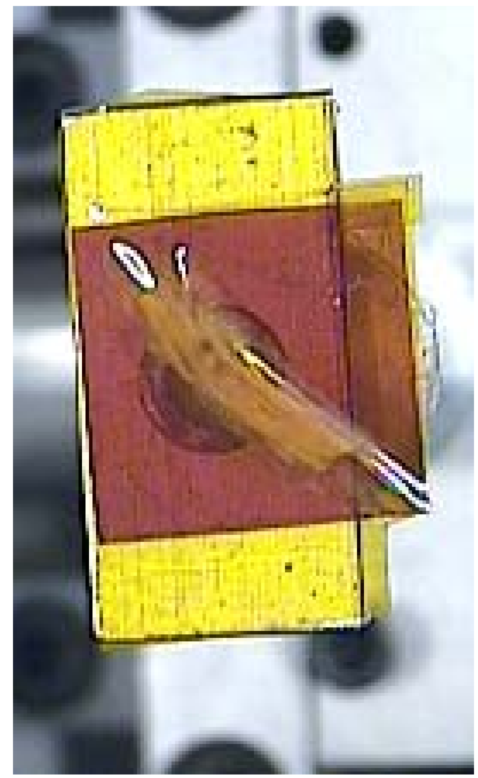


Dark field imaging:

collagen distribution in different tissues

X-ray Ptychography





J. M. Rodenburg et al, Phys. Rev. Lett. 98 034801 (2007)

Conclusions

- Internal structure of Ag, Au and Pb Nanocrystals
- 3D imaging practical for nanocrystals
- Phasing by HIO computation instead of lens
- Strain fields imaged from asymmetric patterns
- Contact Forces and Surface Strain
- Nanowires have domain structure
- Application to biological tissue by ptychography