Instrumental Issues concerning Surface Diffraction that exploits Coherence

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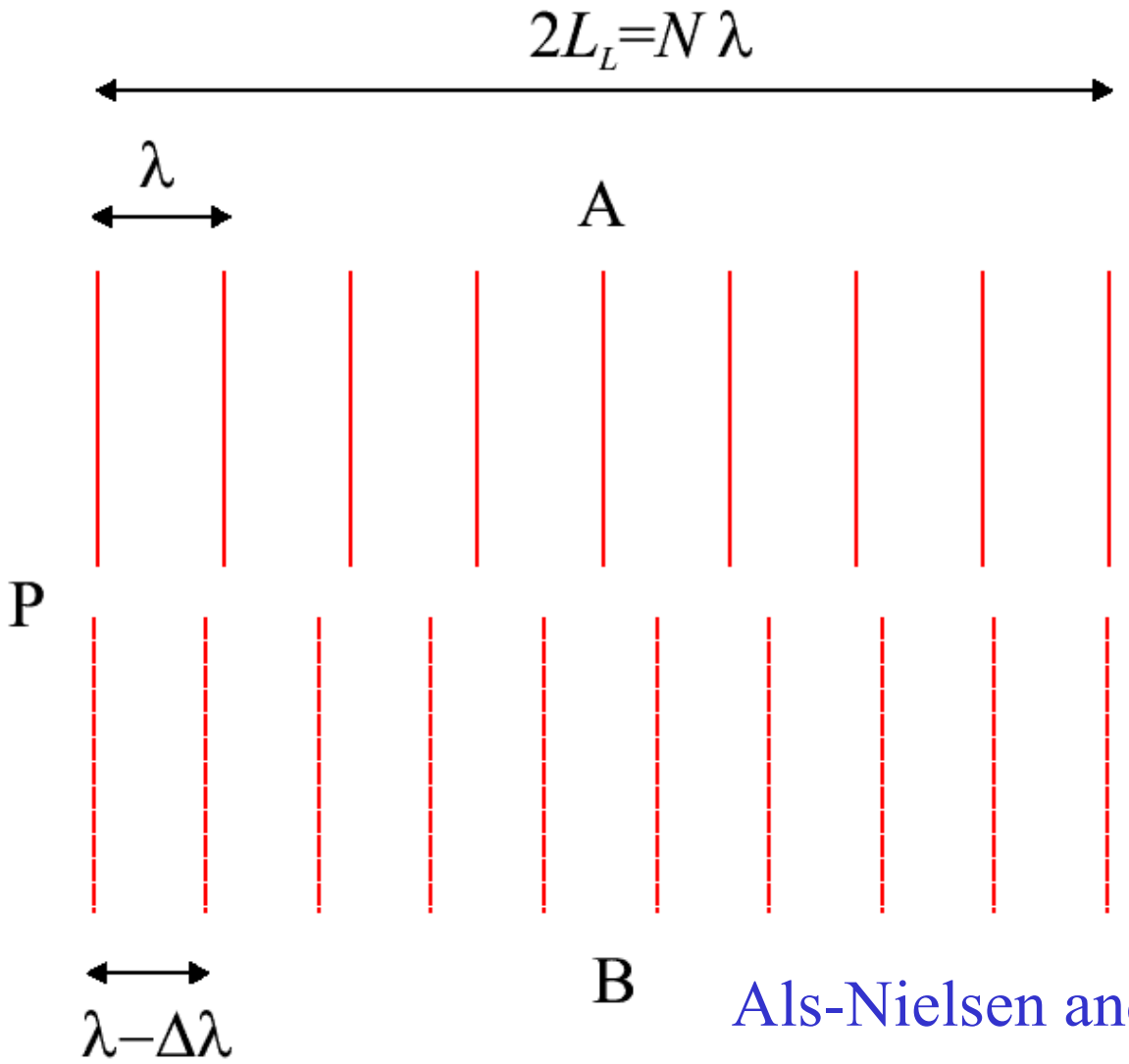
Workshop on Surface and Interface Science at the ESRF

Outline

- Coherence in Diffraction
- How to Solve the Phase Problem
- Nanocrystal Shapes
- Real space Phase objects
- Instrumental issues at Sector 34
- Use of Optics in CXD

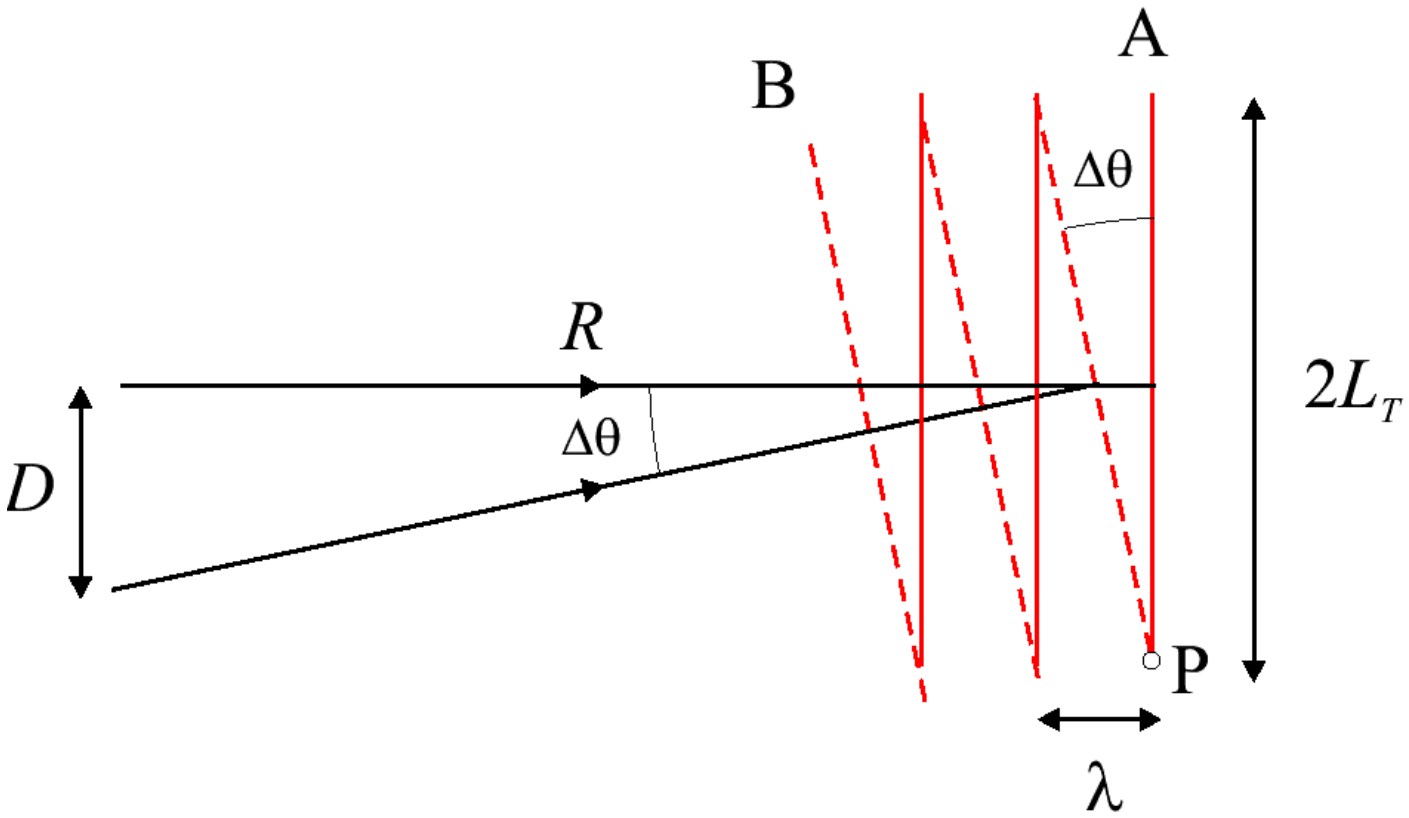
Longitudinal Coherence



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

Als-Nielsen and McMorrow (2001)

Lateral (Transverse) Coherence



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

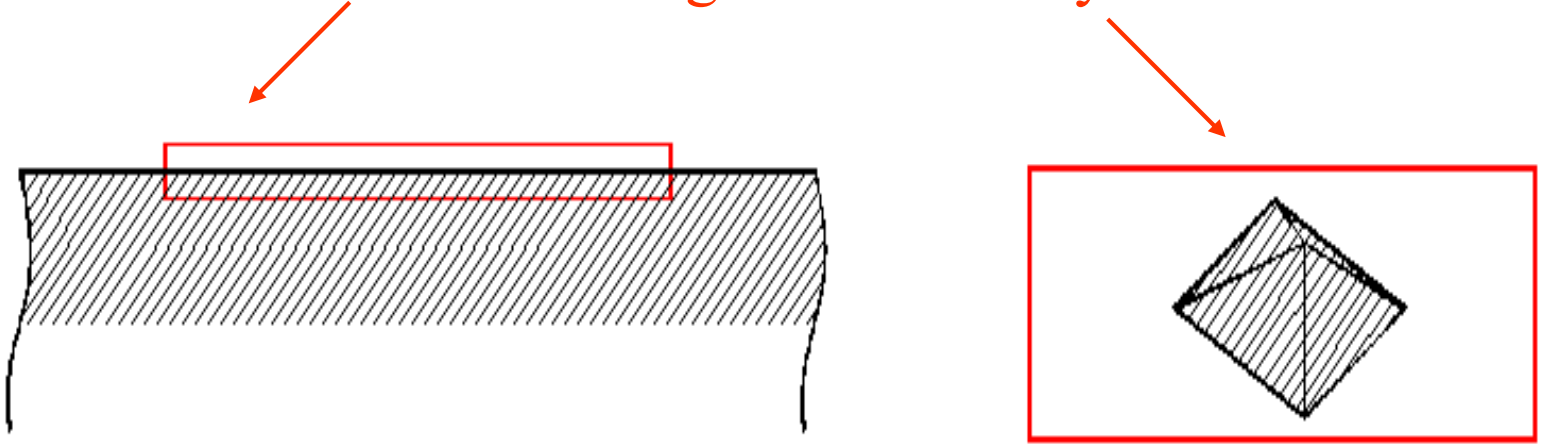
Als-Nielsen and McMorrow (2001)

Coherence at the APS or ESRF

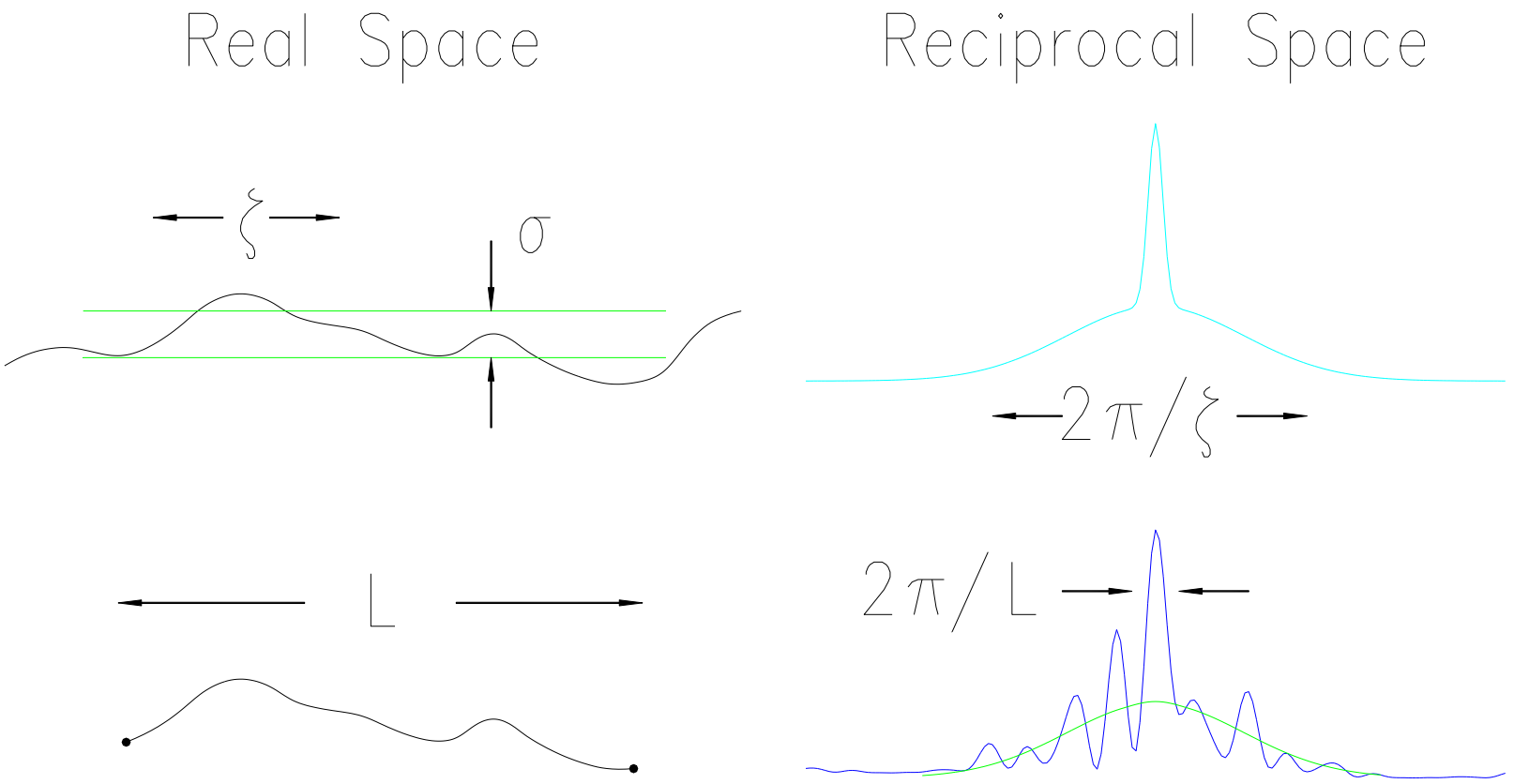
Typical of 3rd Generation (undulator) Synchrotron Source

Coherence of	ξ_{VER}	ξ_{HORIZ}	ξ_{LONG}	Flux
Raw Undulator	35μm	9μm	0.004μm	2×10^{12}
Si(111) Monochromator	35μm	9μm	1μm	1×10^{10}
C(111) Monochromator	35μm	9μm	3μm	3×10^{9}

Coherent region defined by slits

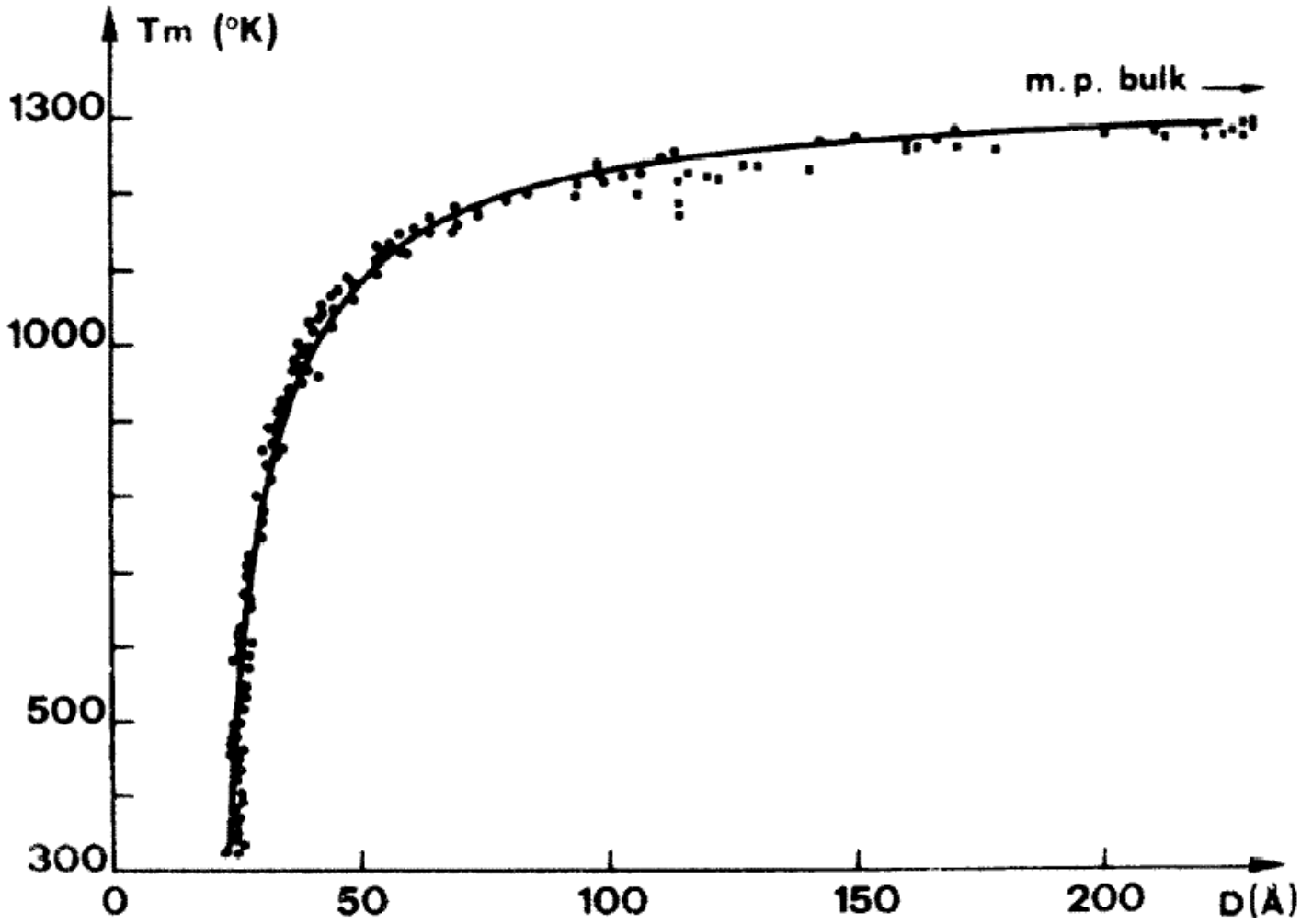


Diffuse Scattering acquires Structure using CXD

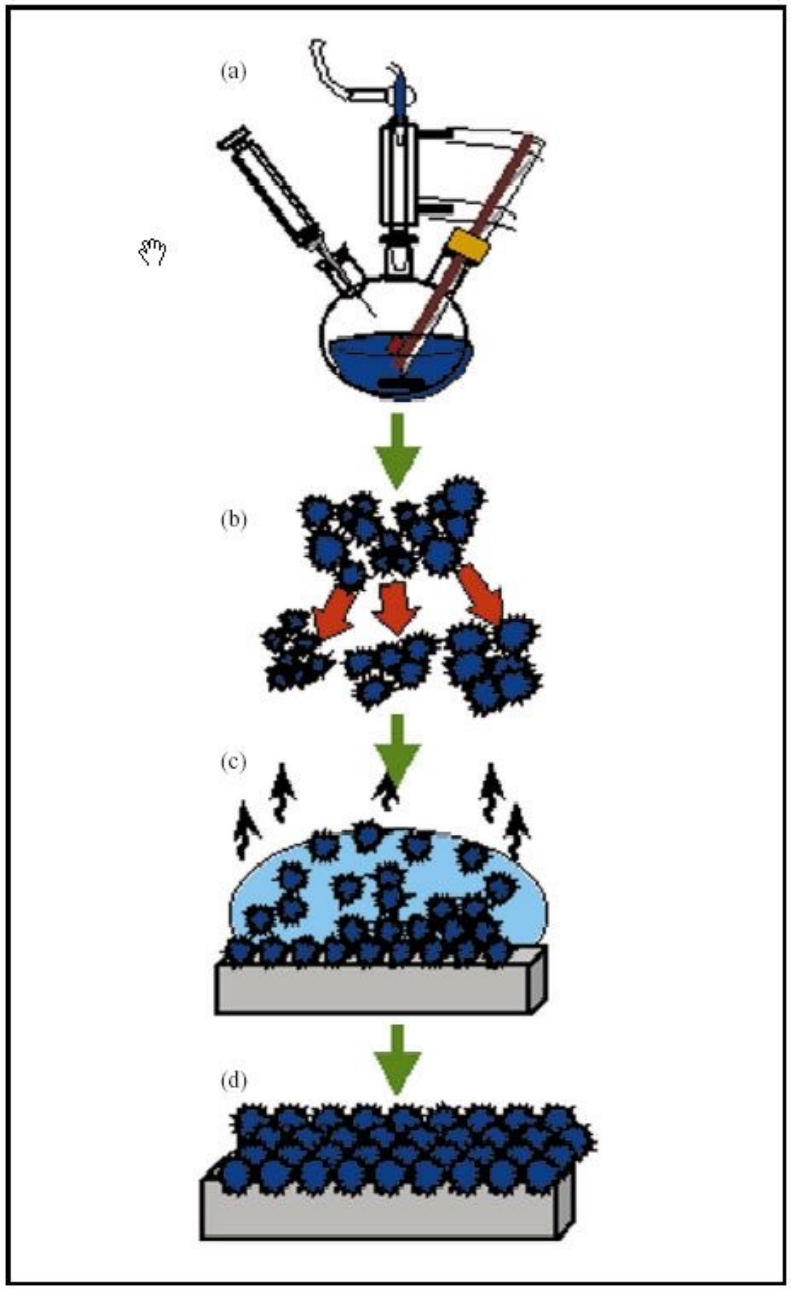


Size-dependent Melting of Au Particles

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

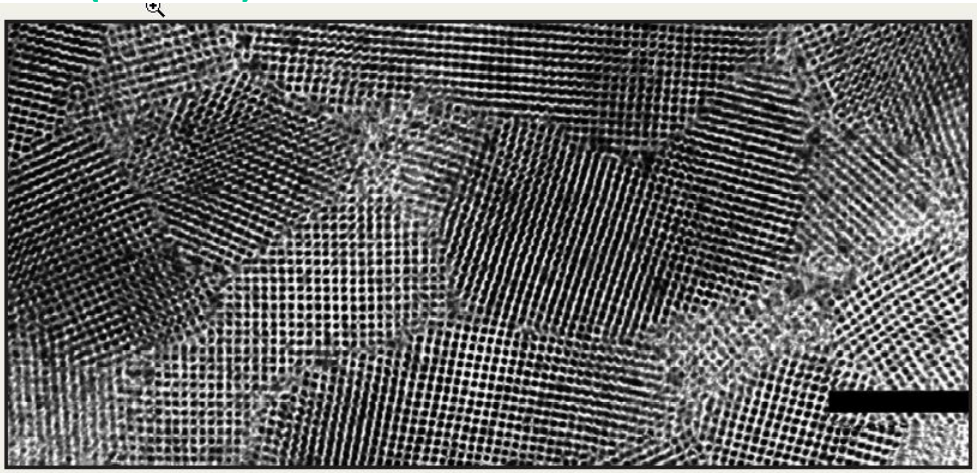


Chemical Synthesis of Nanocrystals



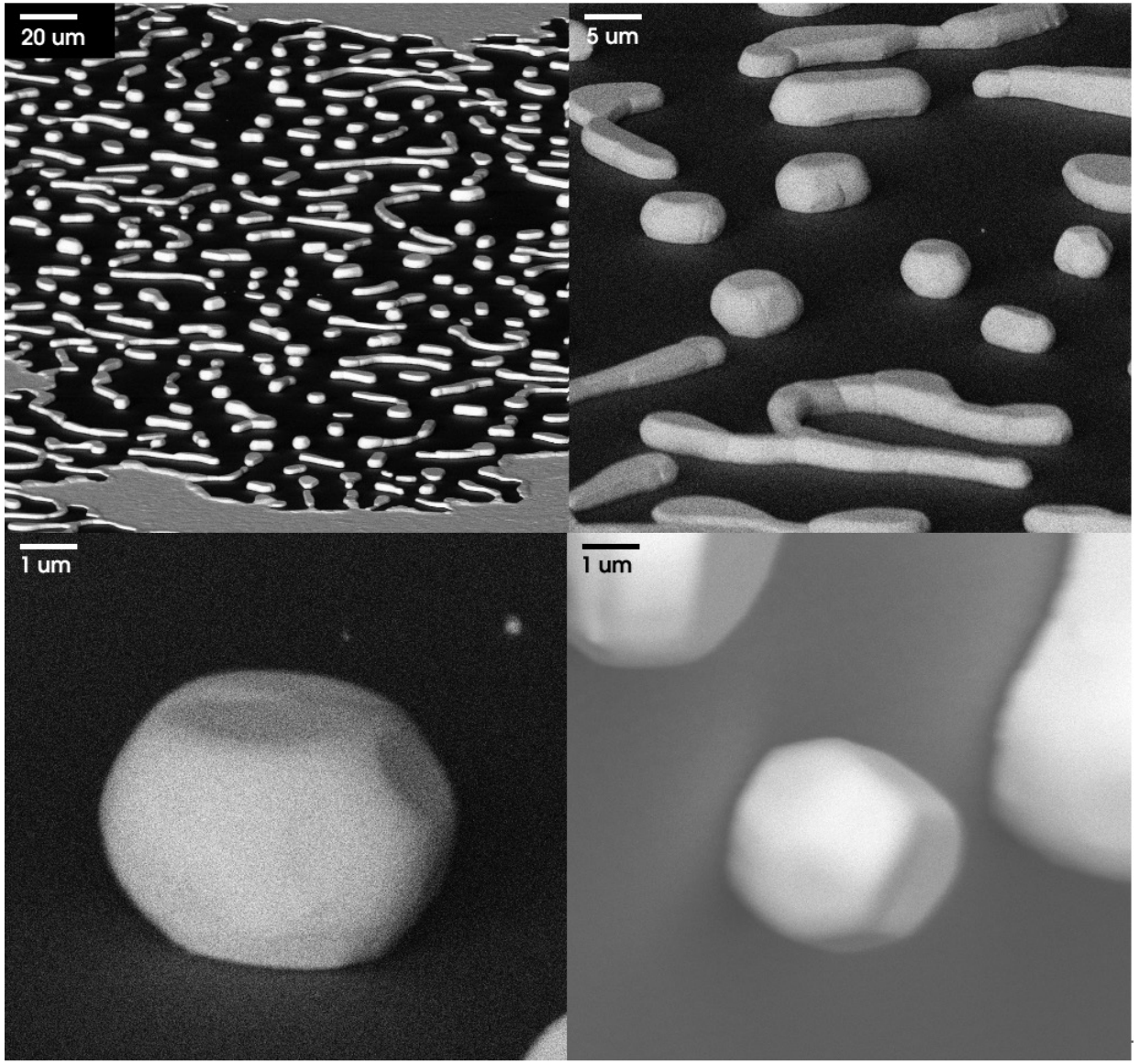
- Reactants introduced rapidly
- High temperature solvent
- Surfactant/organic capping agent
- Square superlattice (200nm scale)

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

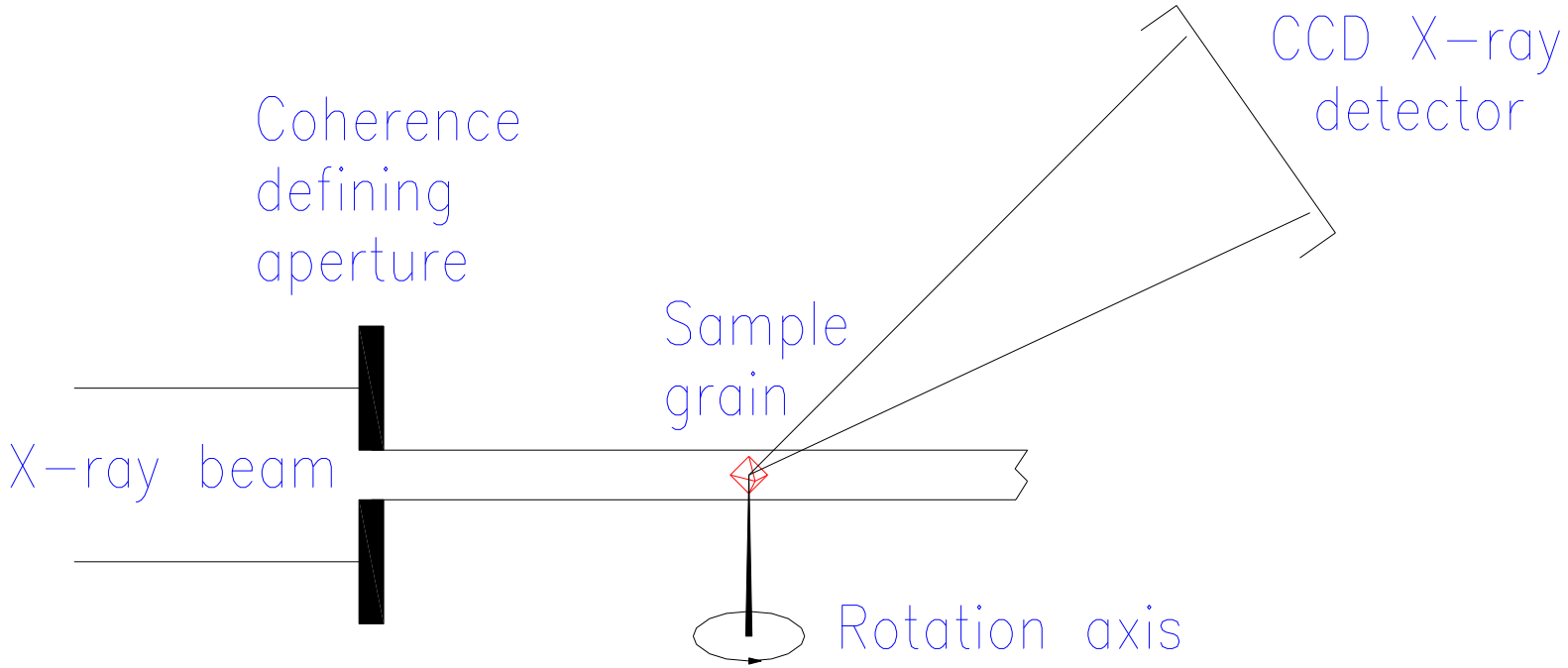


SEMS

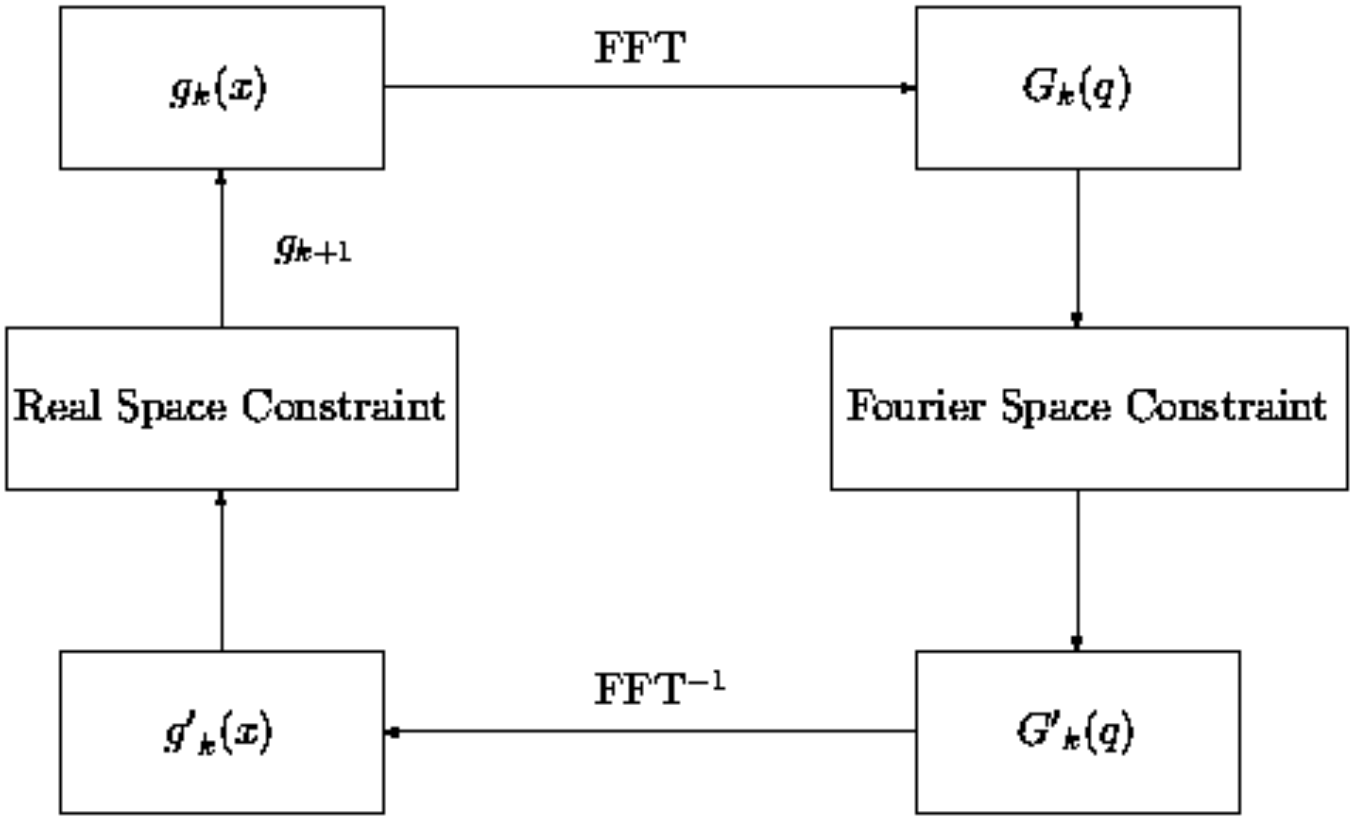
- Au blanket film
- Quartz substrate
- Annealed at 950°C for 70 hrs.



Lensless X-ray Microscope



Generic "Error Reduction" method

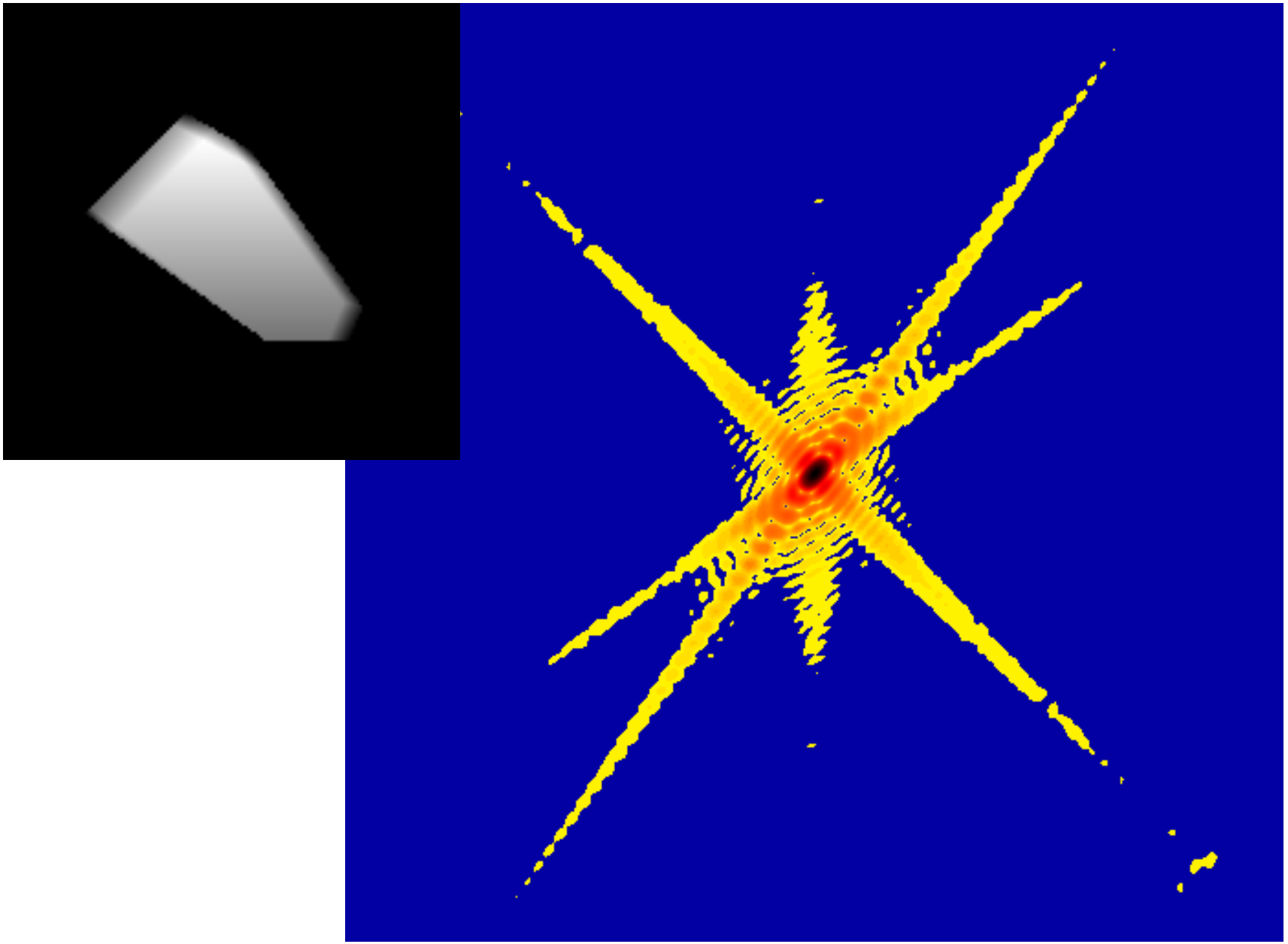


J. R. Fienup Appl. Opt. 21 2758 (1982)
R. W. Gerchberg and W. O. Saxton Optik 35 237 (1972)

Real-space Constraints in Crystallograhy

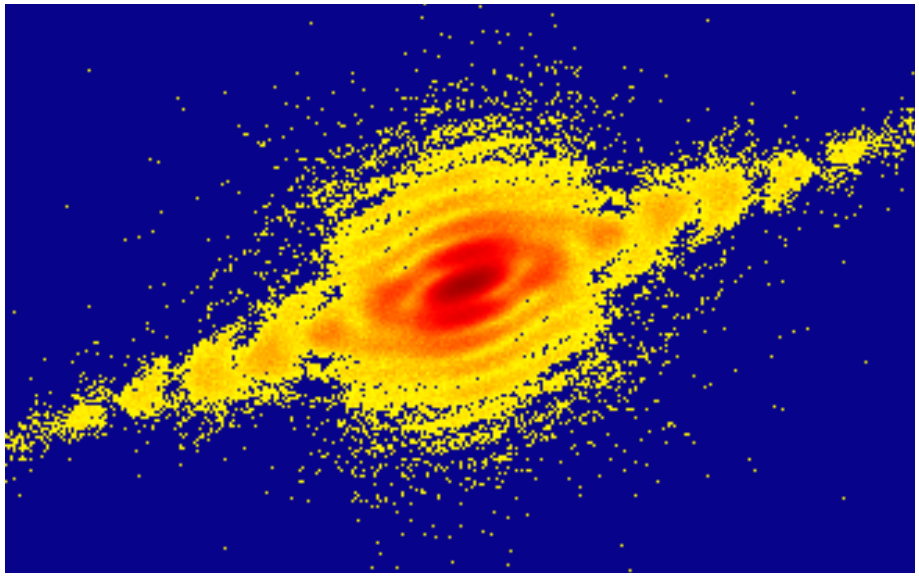
R. P. Millane, J. Opt. Soc Am. A **13** 725 (1996)

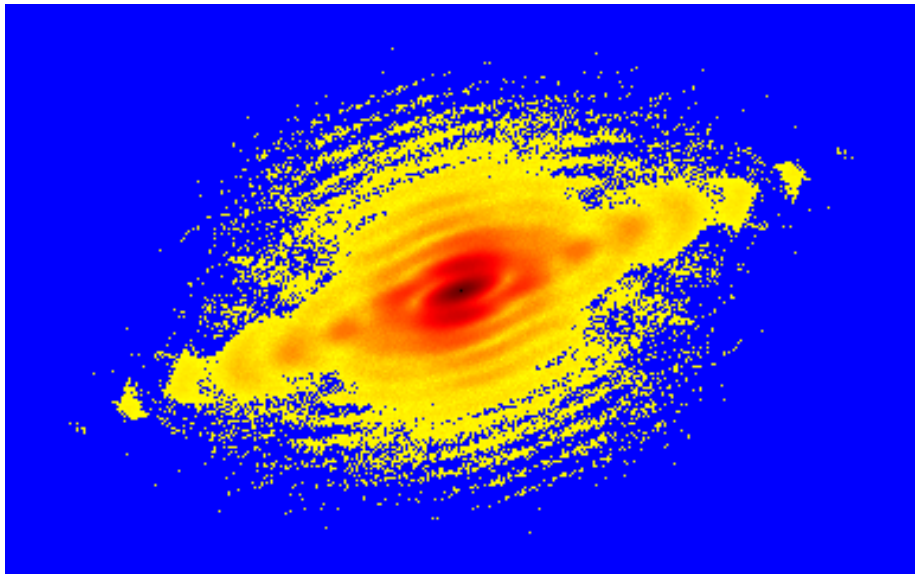
- 'Positivity' and 'Atomicity' constraints (Sayre)
- Finite support, molecular envelope
- Solvent flattening/Molecular replacement
- Non-crystallographic symmetry
- Non-uniqueness is 'pathologically rare' (d>1)
- Uses memory to avoid stagnation (Fienup HIO)

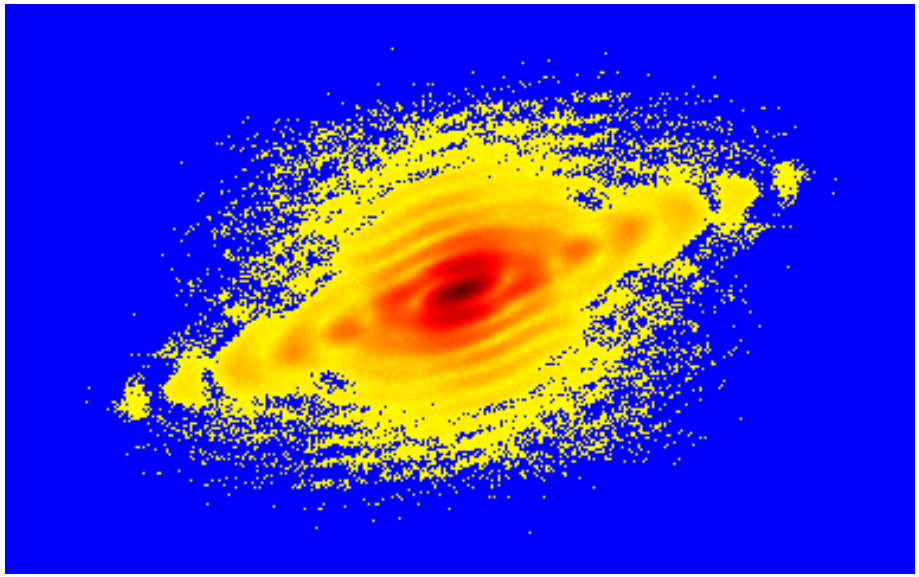


Symmetrized Data and two best fits

Chisq=0.0005

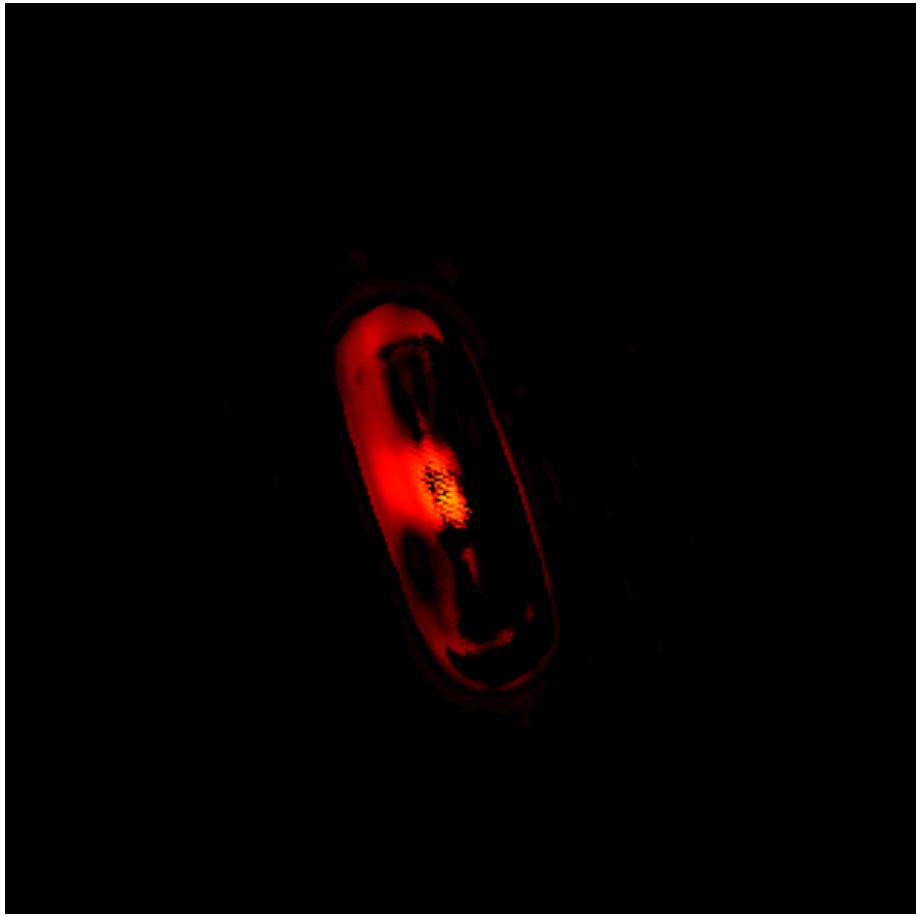


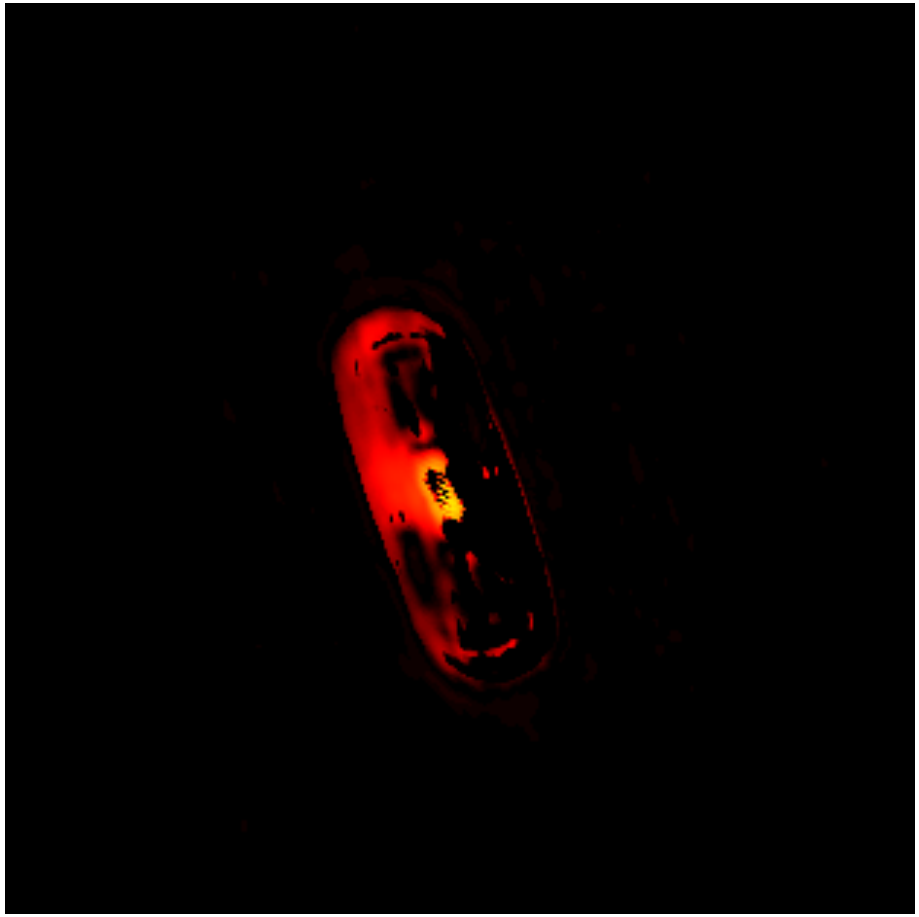




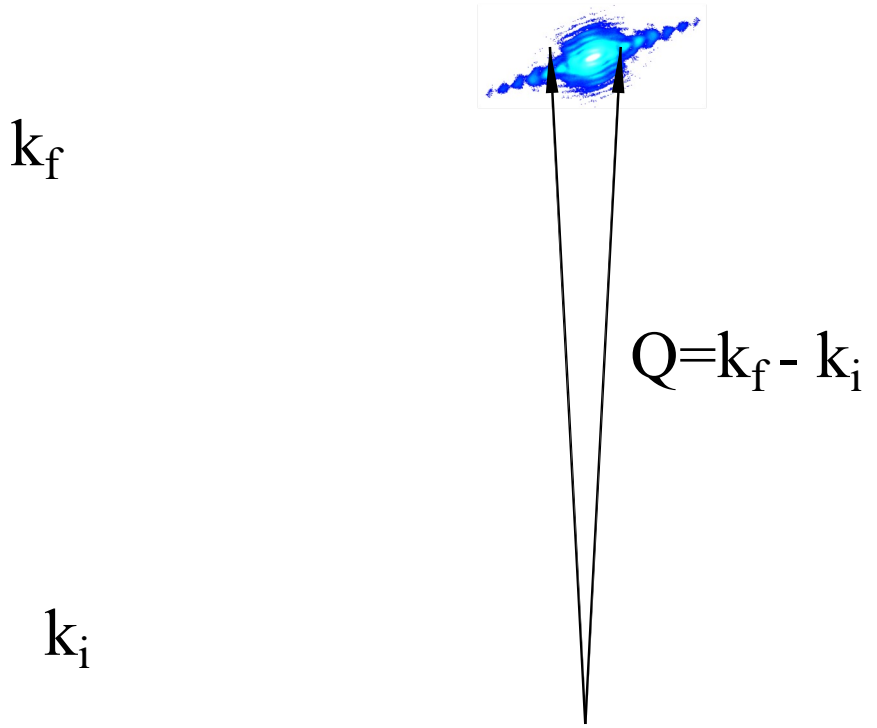
2D Reconstructions

chisquare = 0.0005

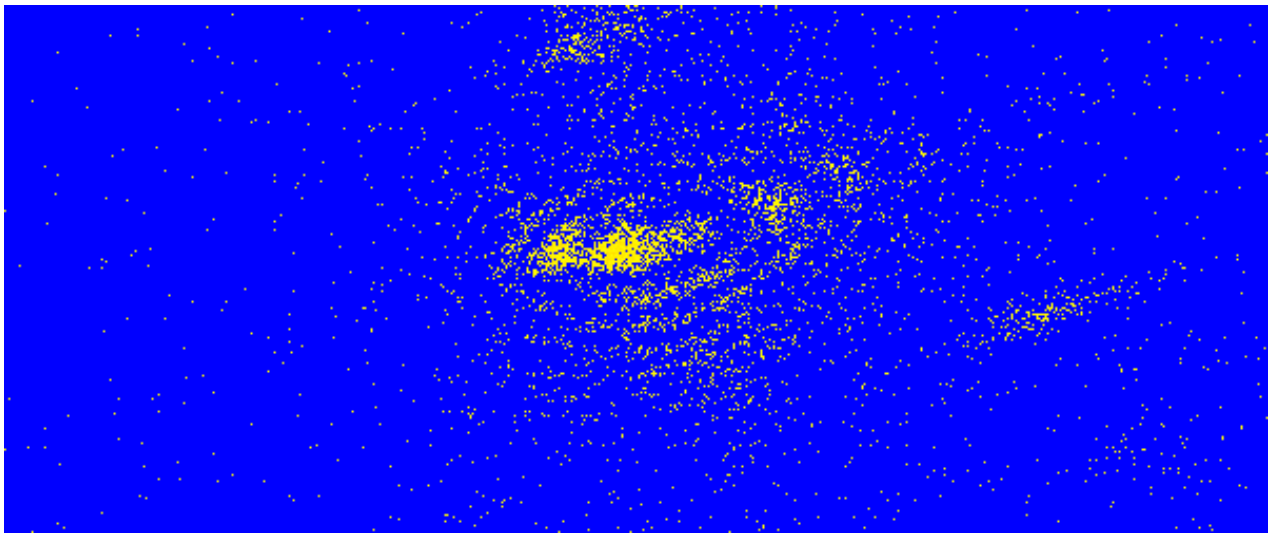


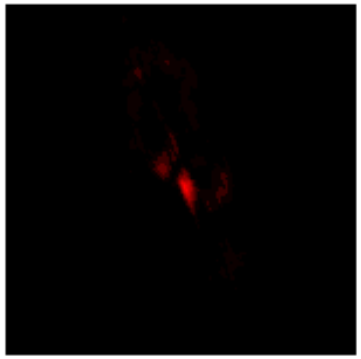
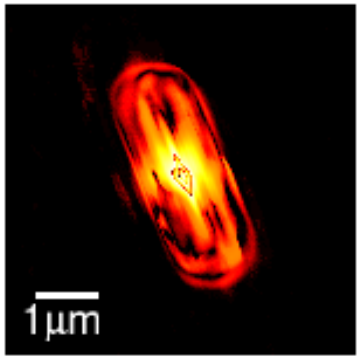


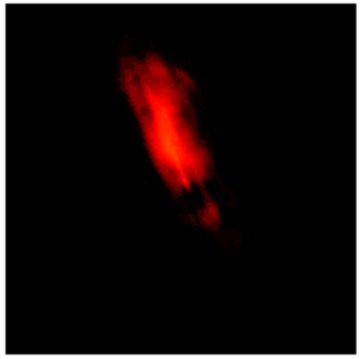
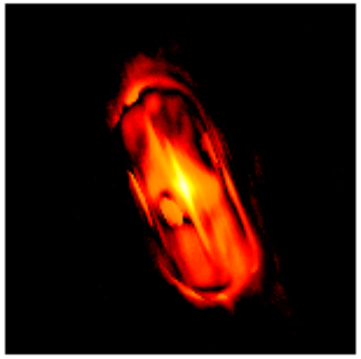
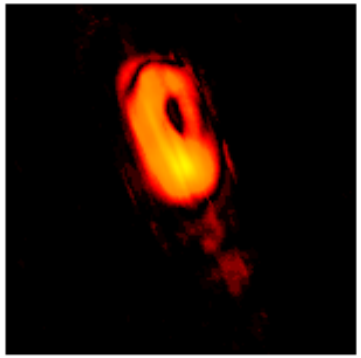
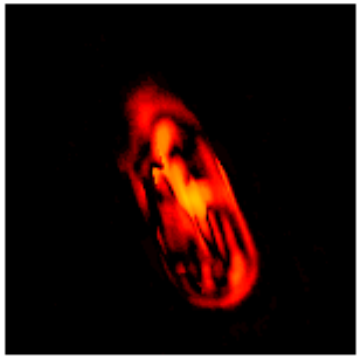
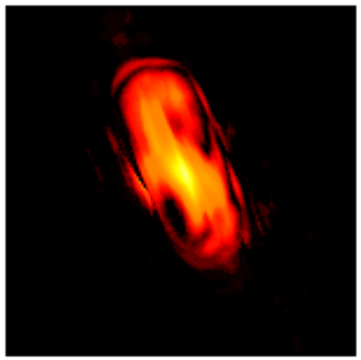
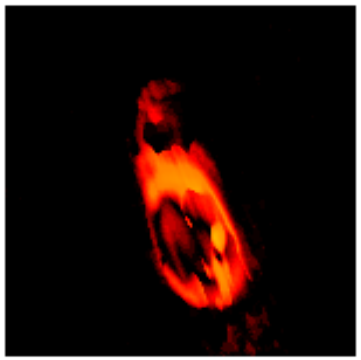
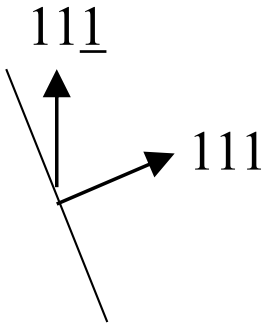
3D Diffraction Method

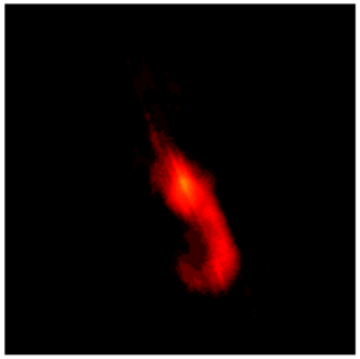
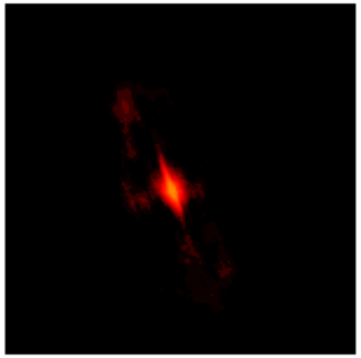


3D Diffraction Data
1 micron Au crystal

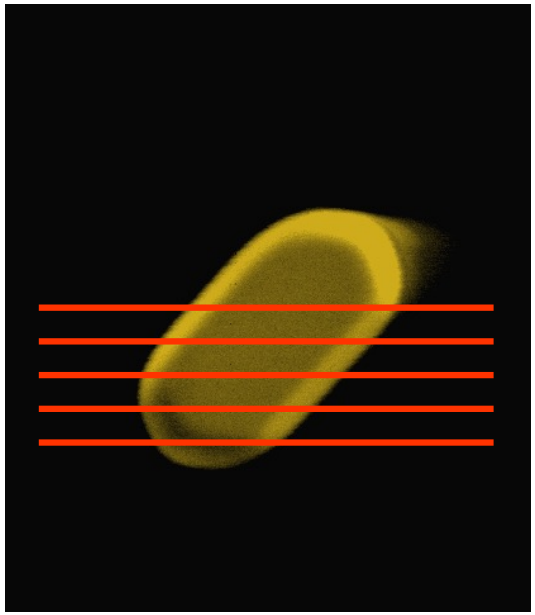


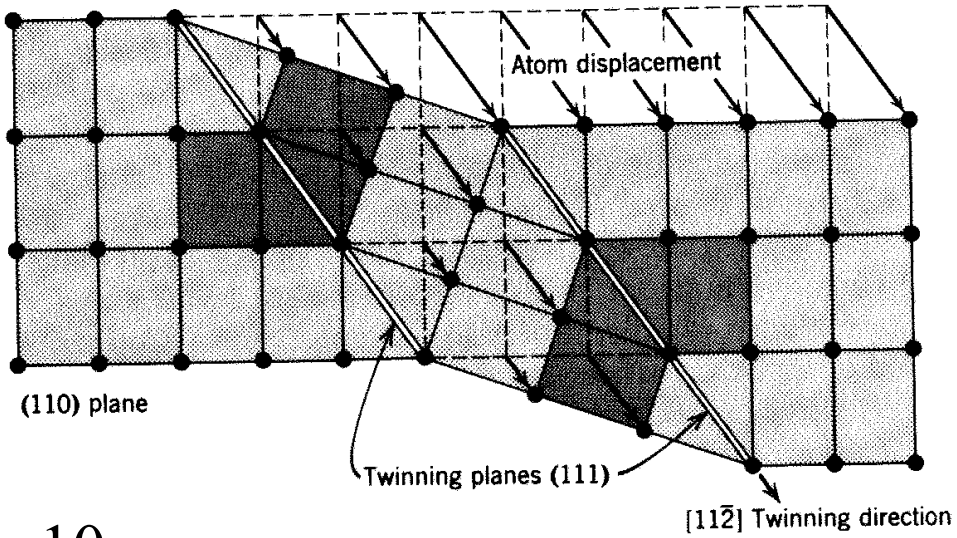



Slices through plan view SEM:



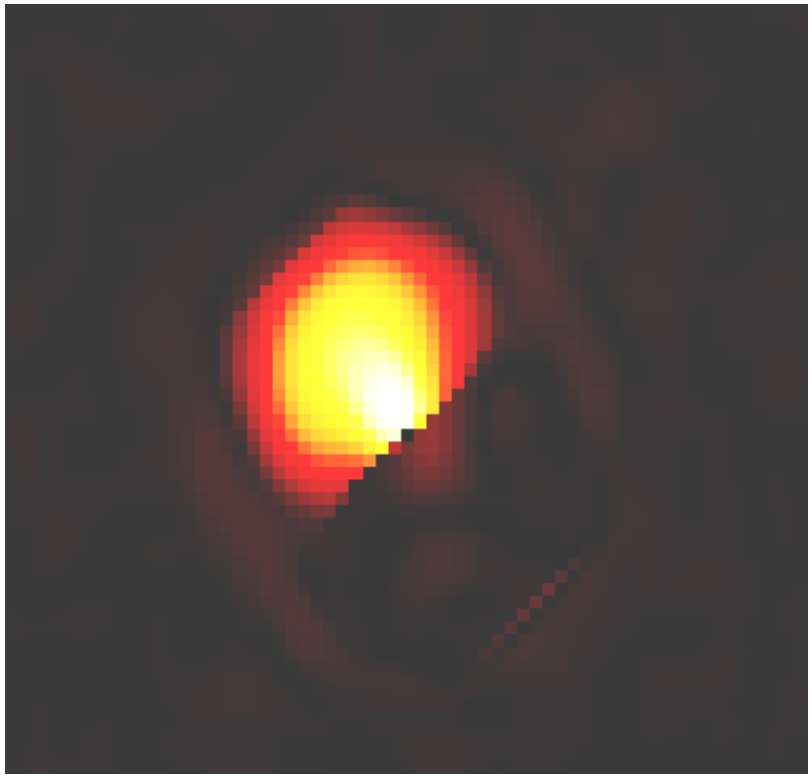
Twinning in deformed FCC metals

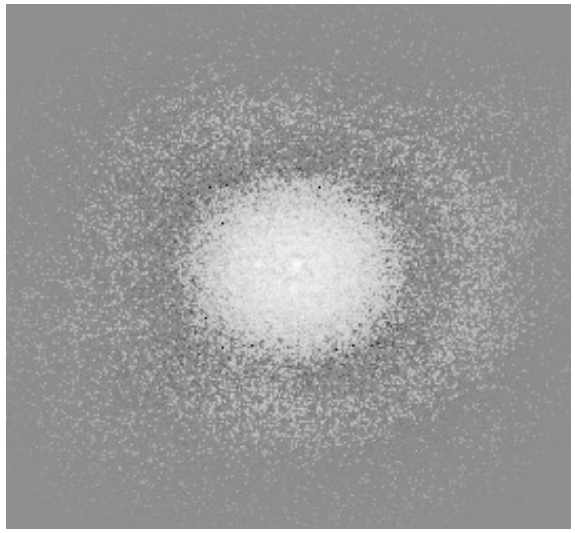
J. Wulff, "Structure and Property of Materials III" (1965)

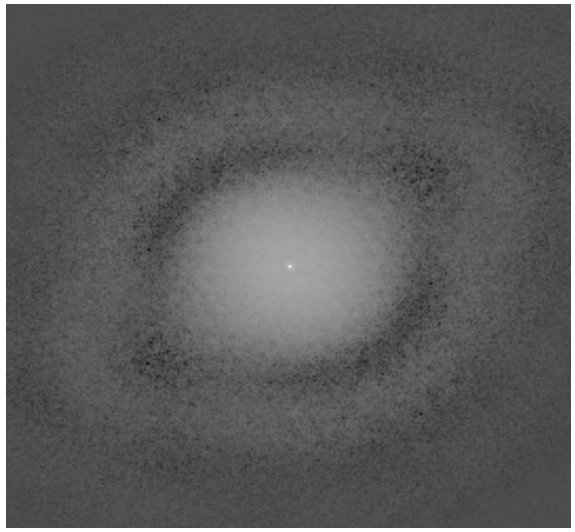


~10μm
Cu

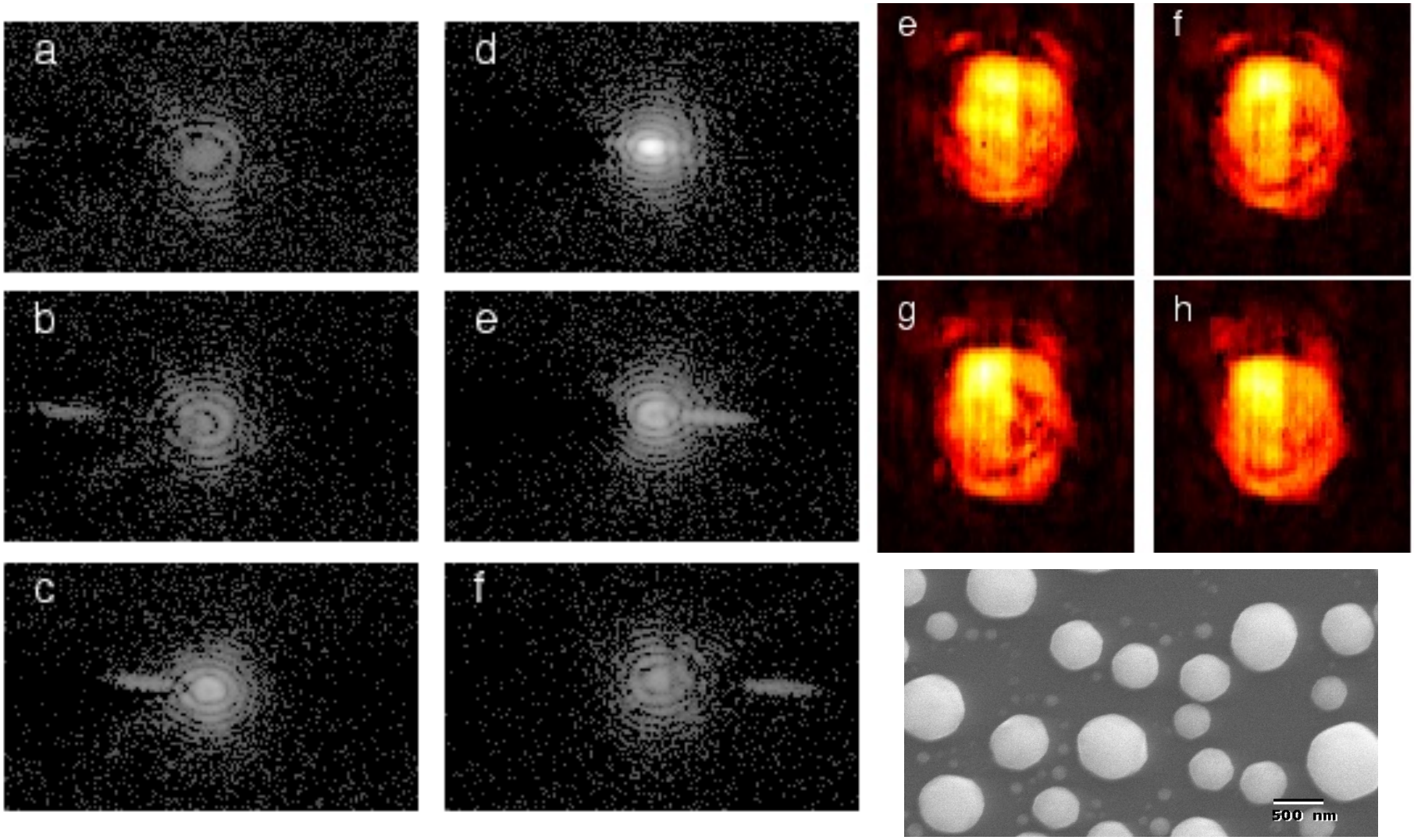
Reconstruction of Pb Nanocrystal



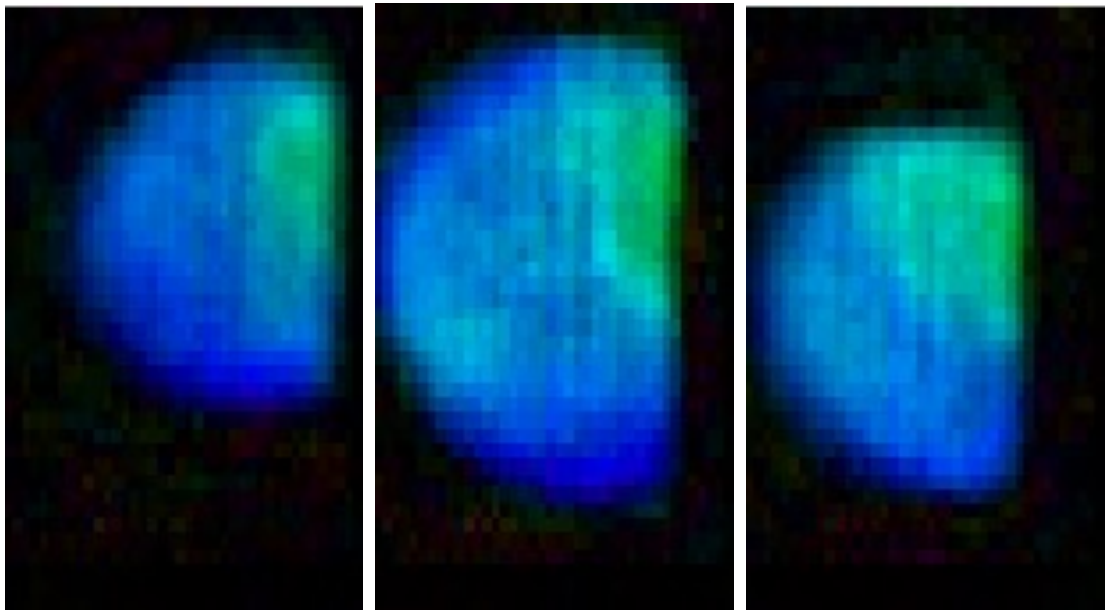




3D reconstruction of Pb nanocrystals

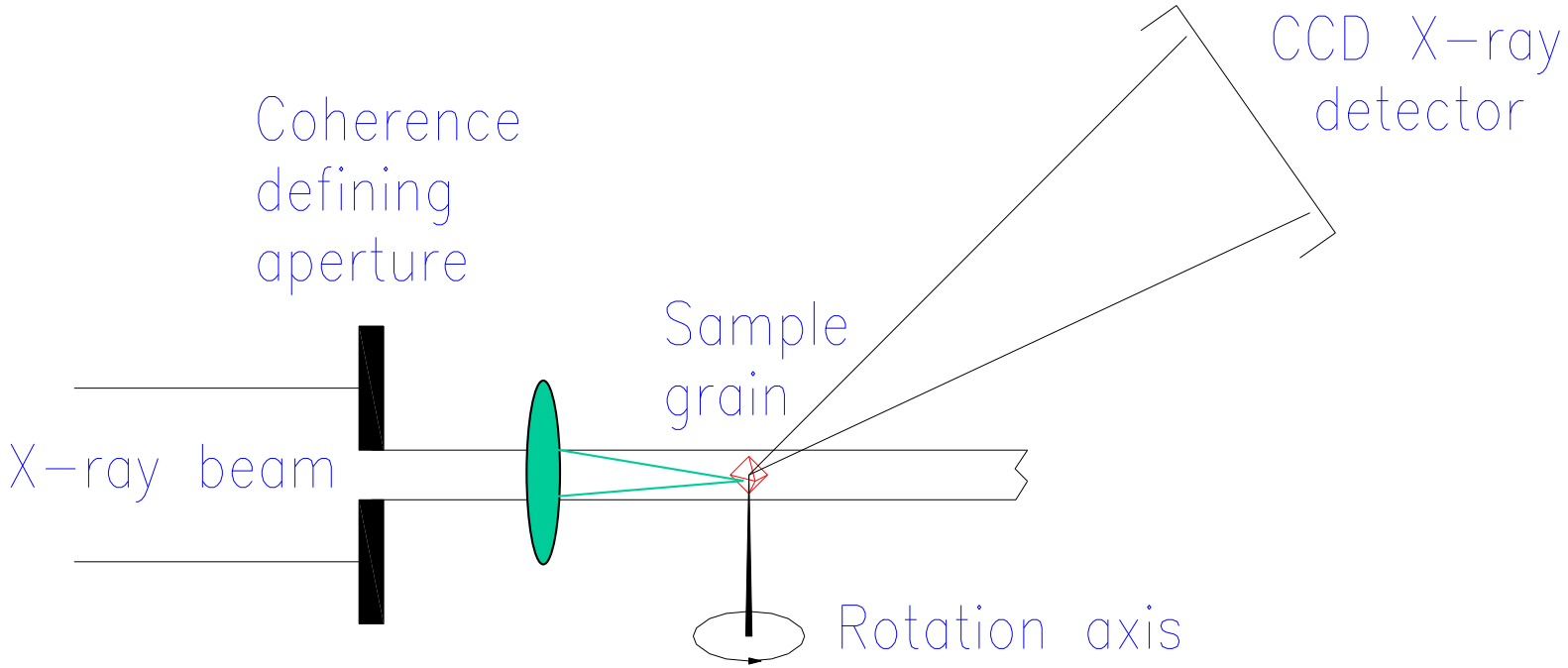


3D Phase Images of Lead Nanocrystals

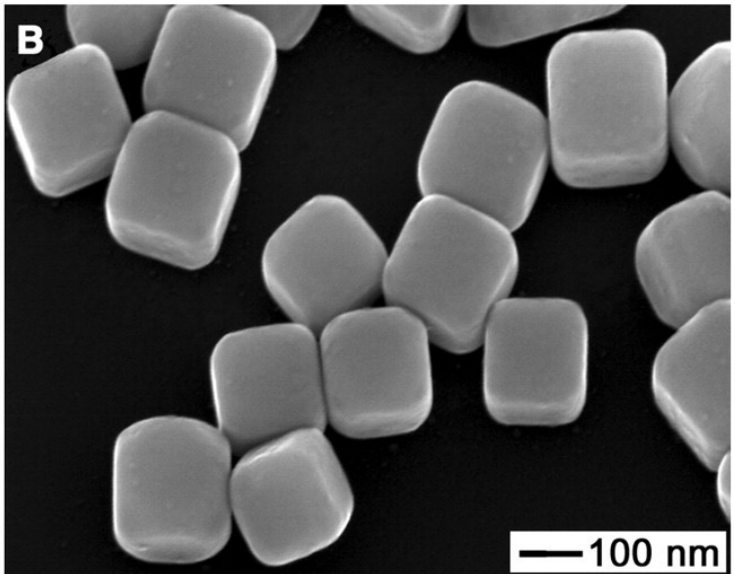


- 500nm Pb crystals grown in-situ in UHV system at APS (Argonne) on a SiO_2 support (vertical).
- 3D coherent X-ray diffraction data is inverted into hemi-spherical cross-sectional slices.
- These images are colored according to the phase of the density, which changes abruptly at an internal boundary.
- This plane is identified as a single deformation fault in the crystal.

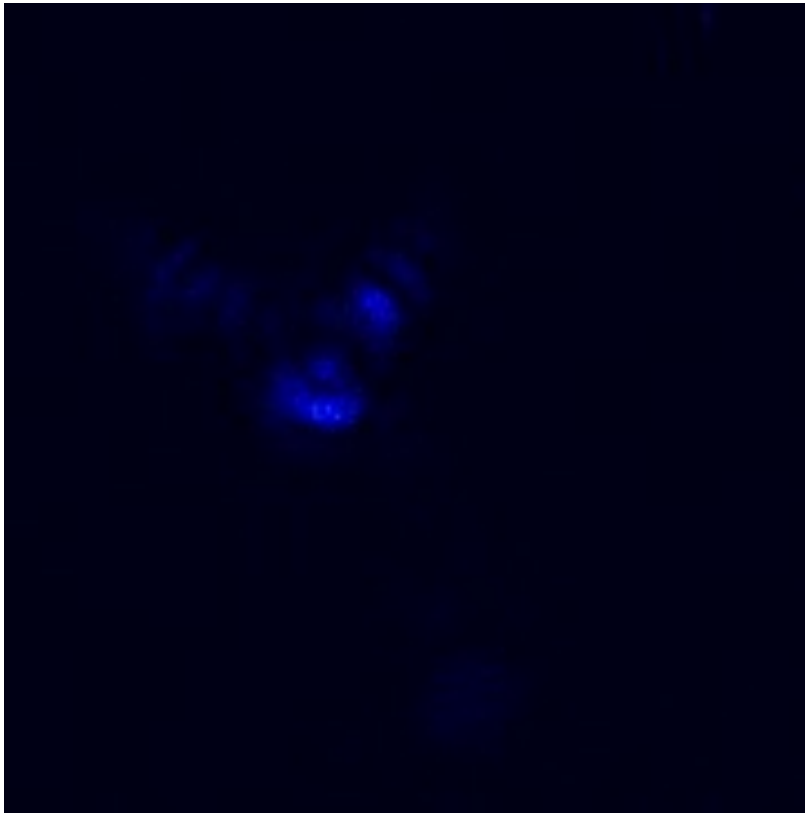
Lensless X-ray Microscope



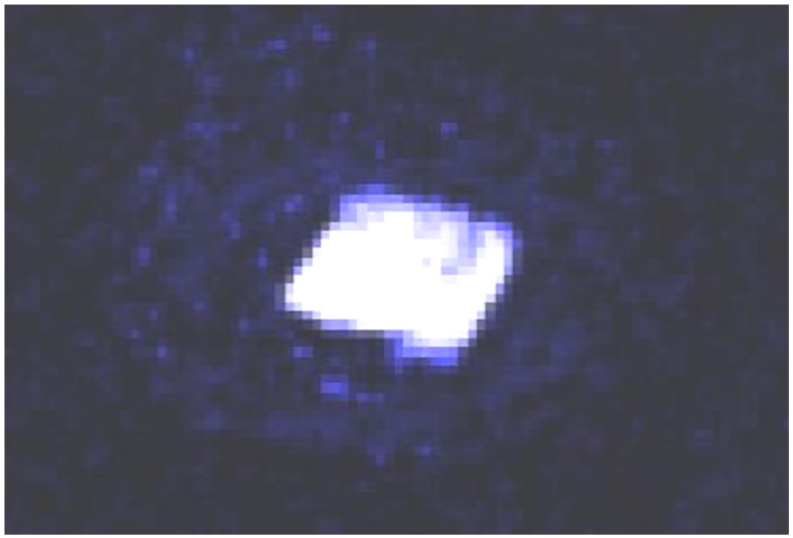
Chemically Synthesized Silver Nanocubes



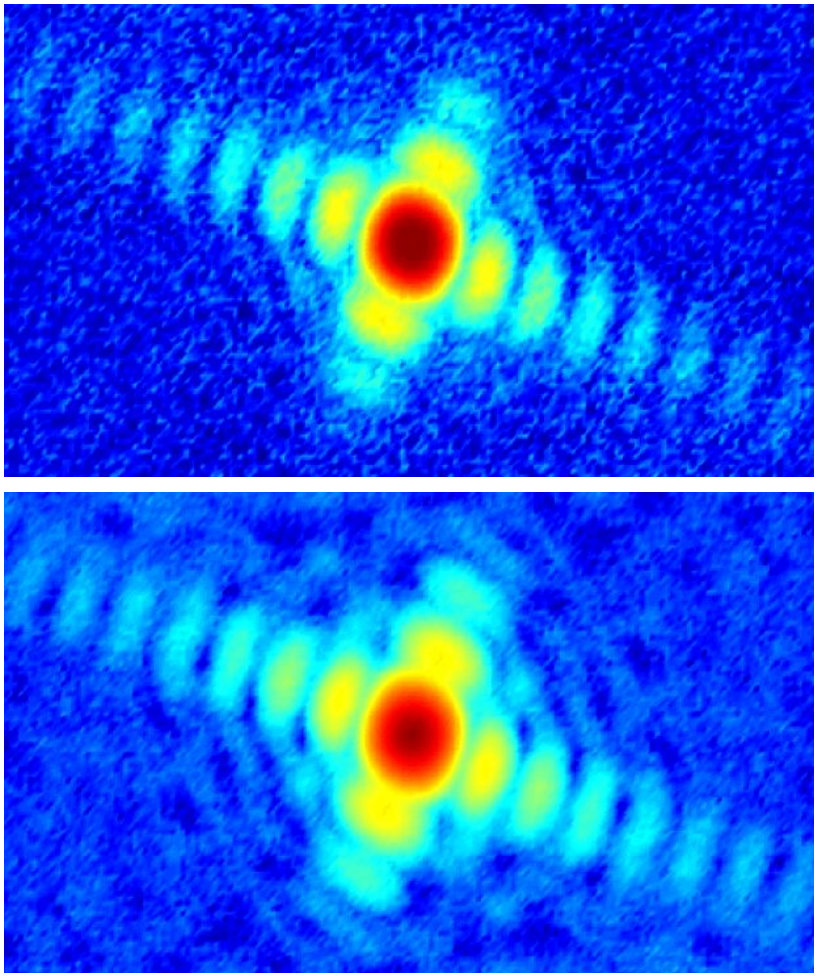
Yugang Sun and Younan Xia,
Science 298 2177 (2003)



Reconstruction of 2-fold View

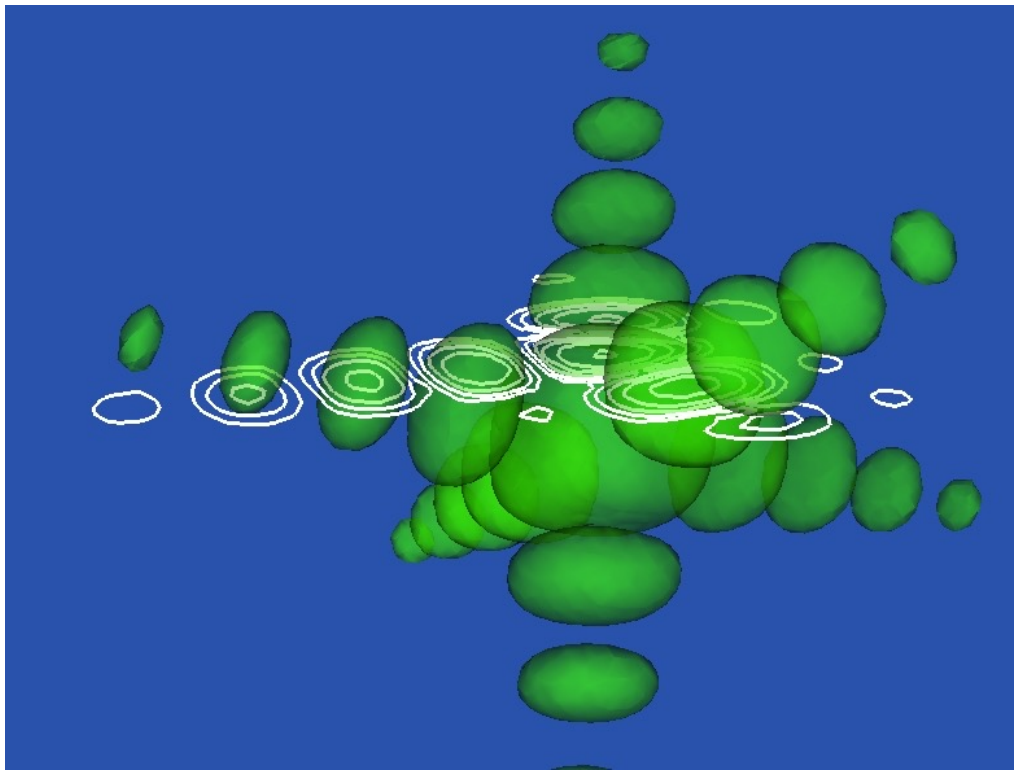


200nm



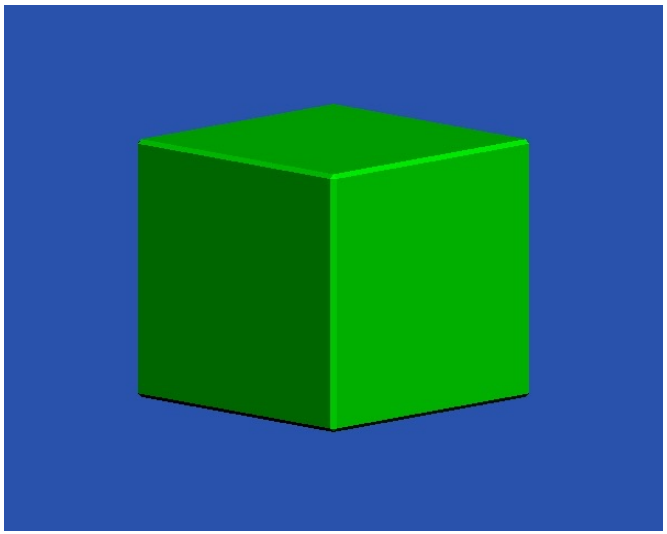
Miscentered Non-Centrosymmetric Reciprocal Space Slice

$$A_q \neq A_{\bar{q}}^*$$

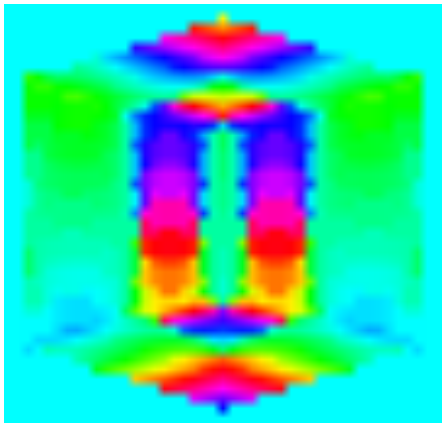


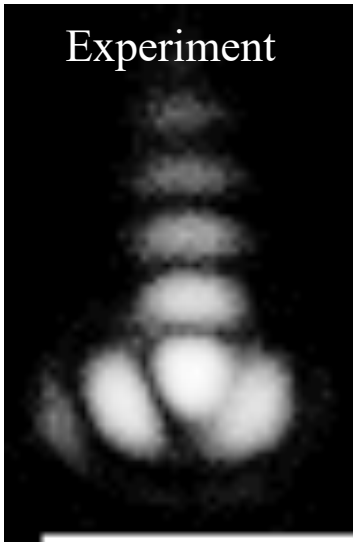
Real-space phase.

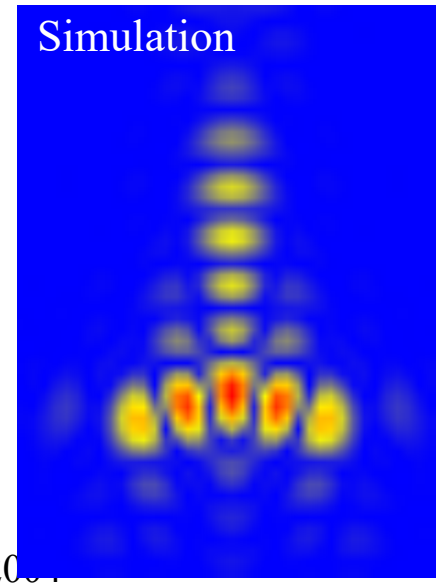
What can we expect to see?



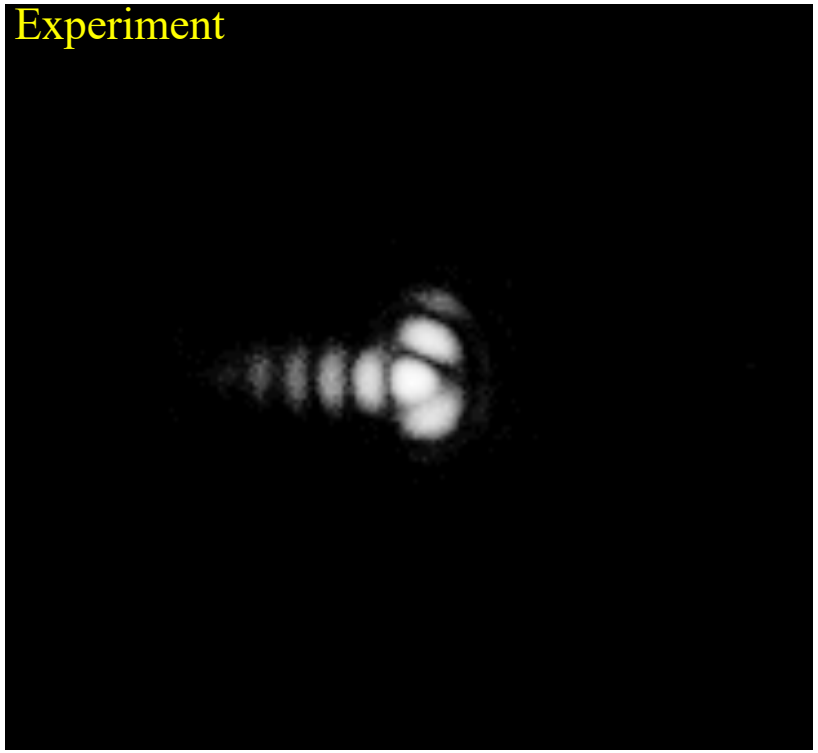
$$A(\bar{q}_o+\bar{q}_z)=\sum_i \sum_z \underbrace{\rho(x,y,z)}_{u_i\{x,y\}} e^{2\pi i \bar{q}_z\cdot\bar{z}_i} e^{2\pi i\{\bar{q}_o\}\cdot\bar{r}_i}$$

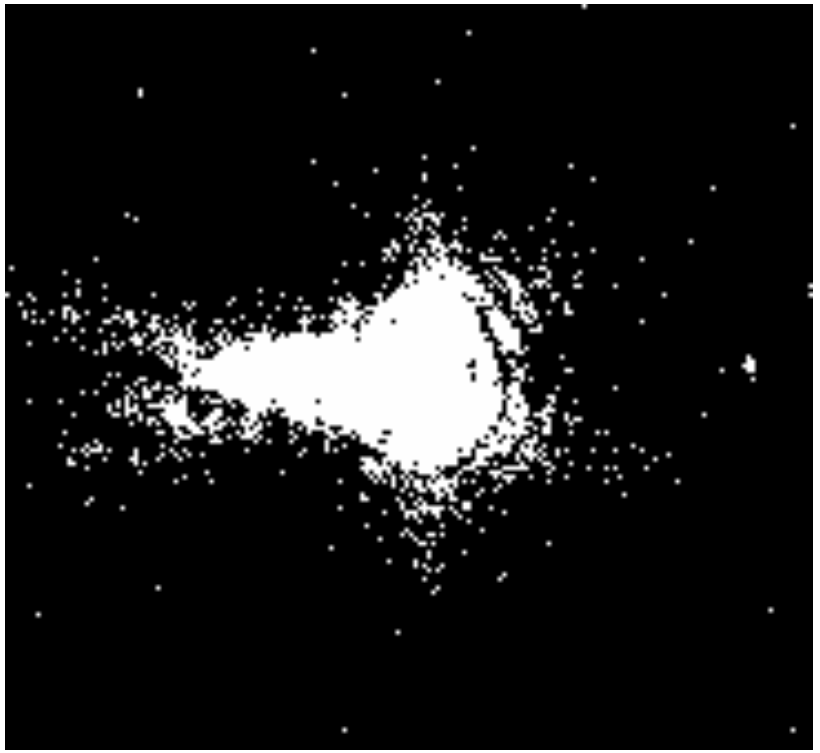




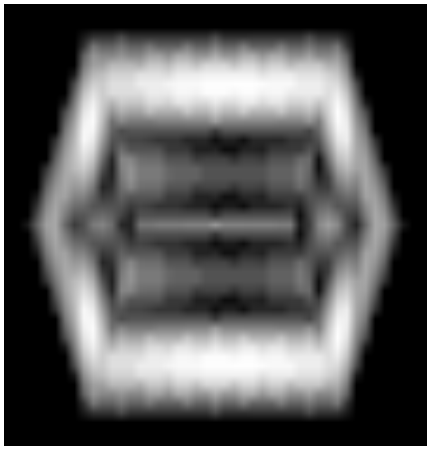


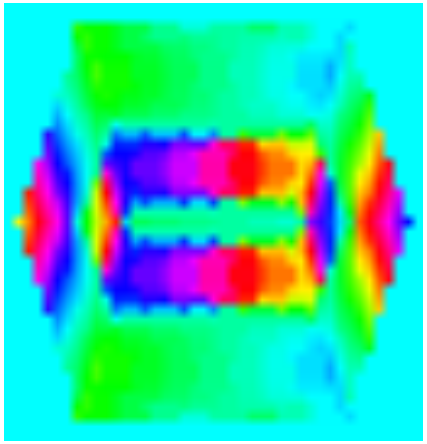
Data and Data Mask (>7% Max)

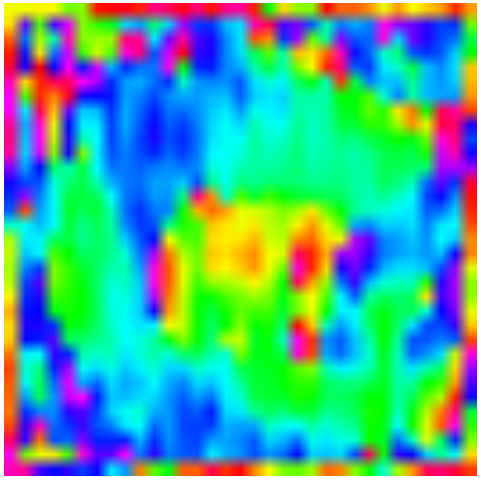


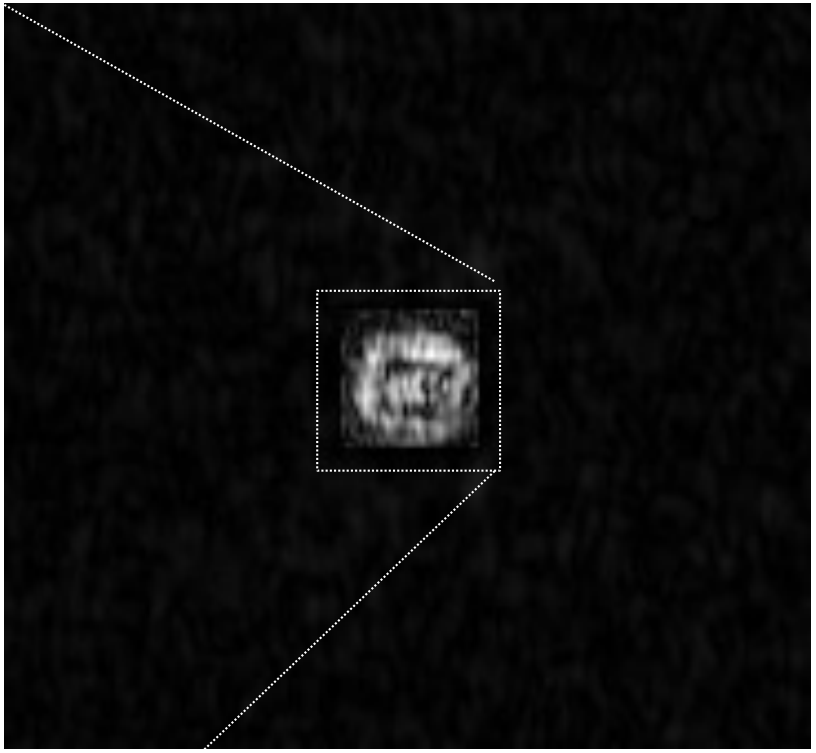


Direct Space Result (1000 Iterations)

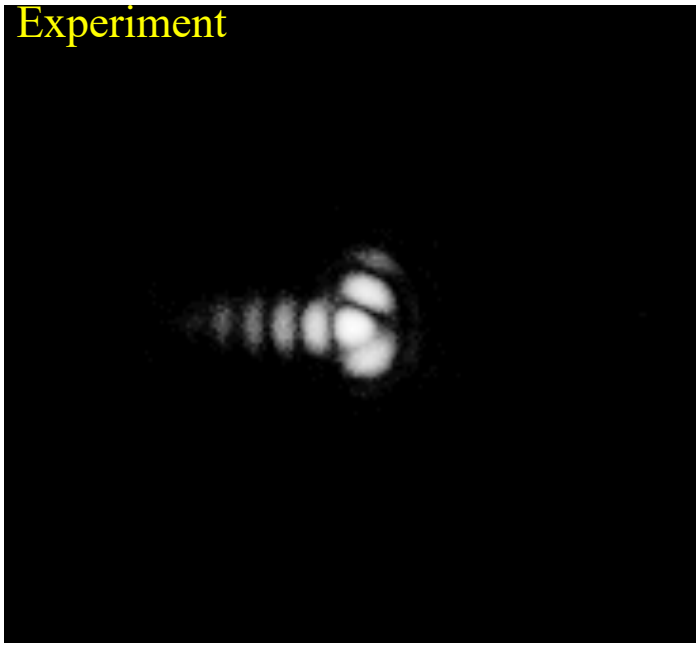


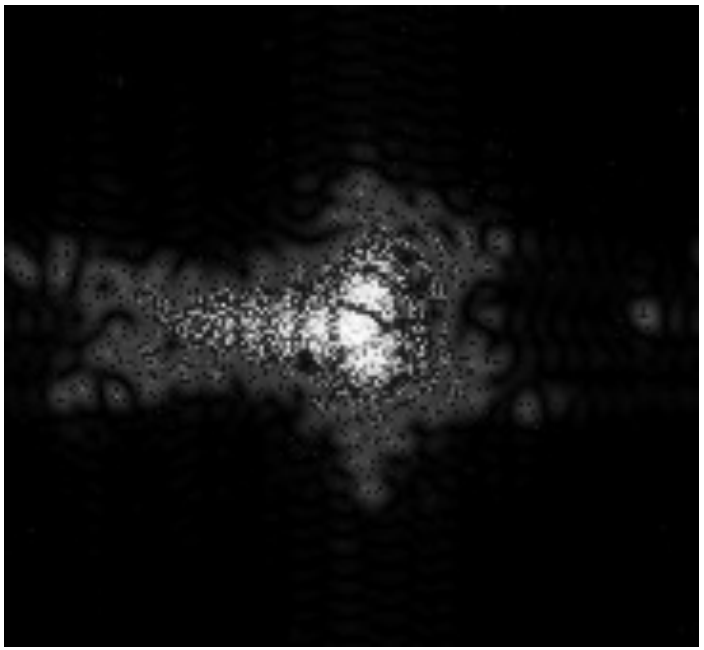


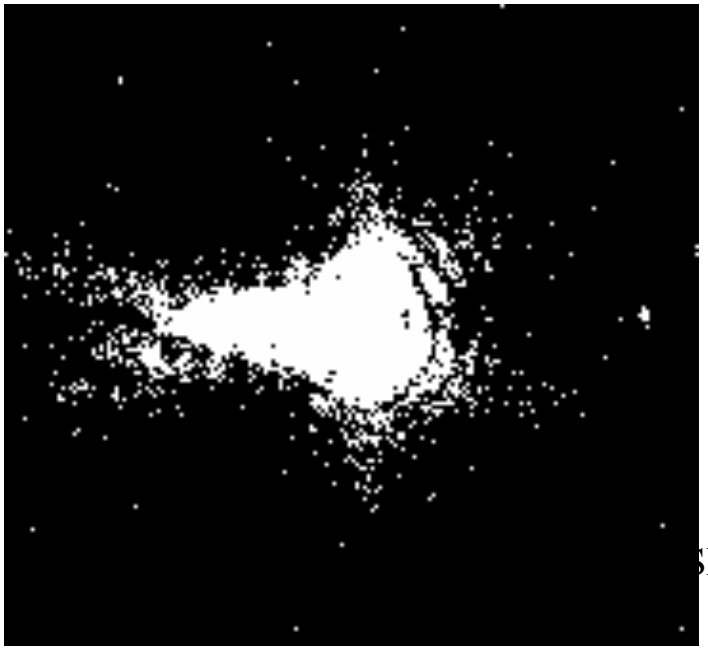


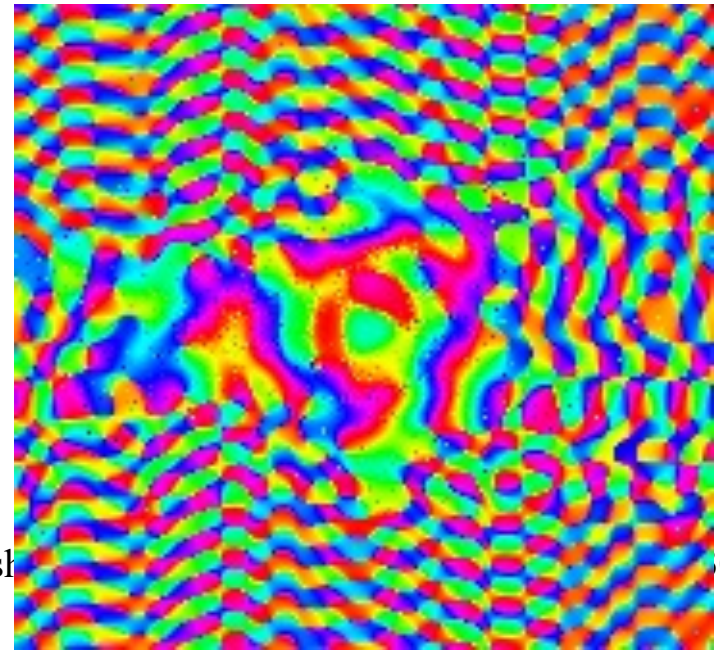


Reciprocal Space Result (1000 iterations)



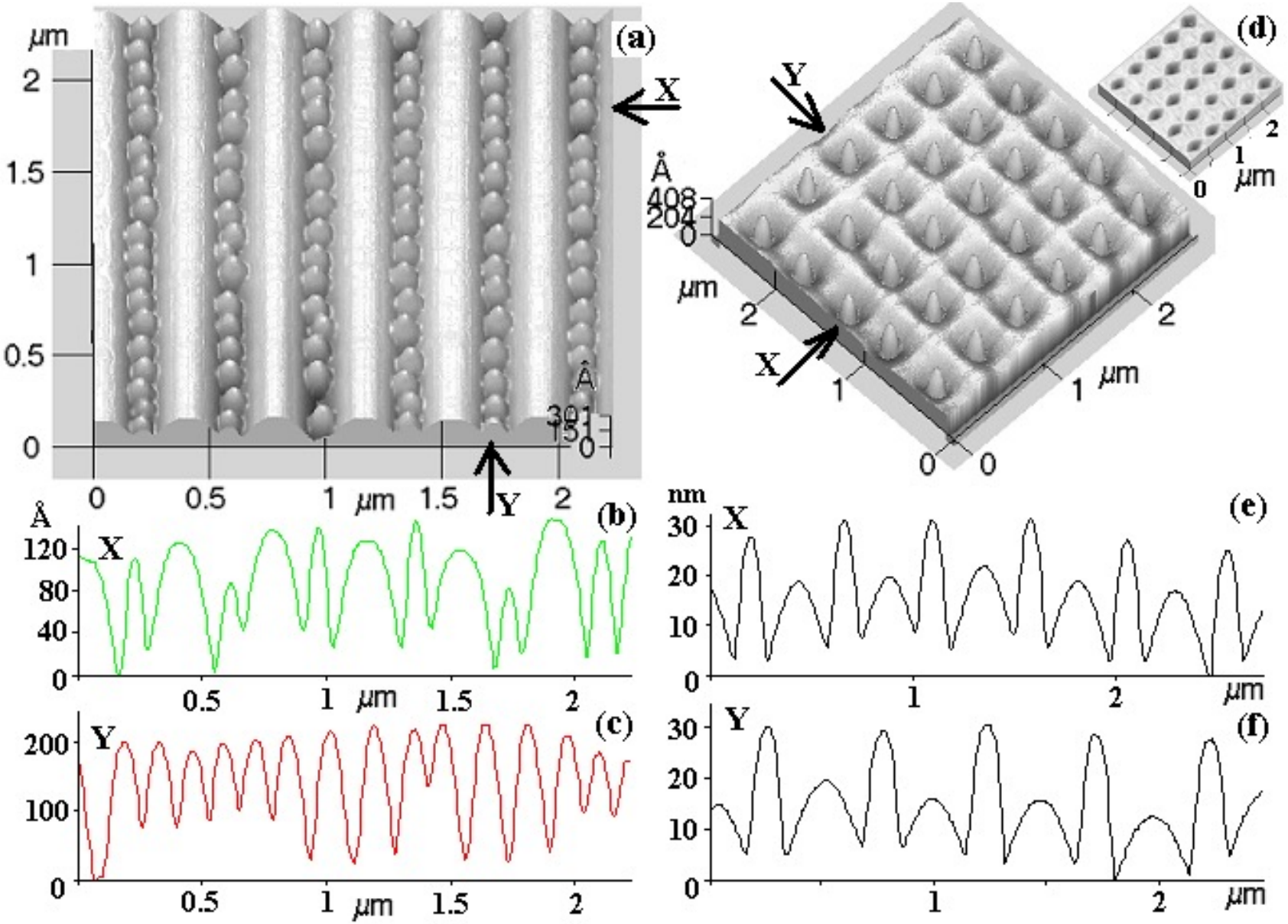






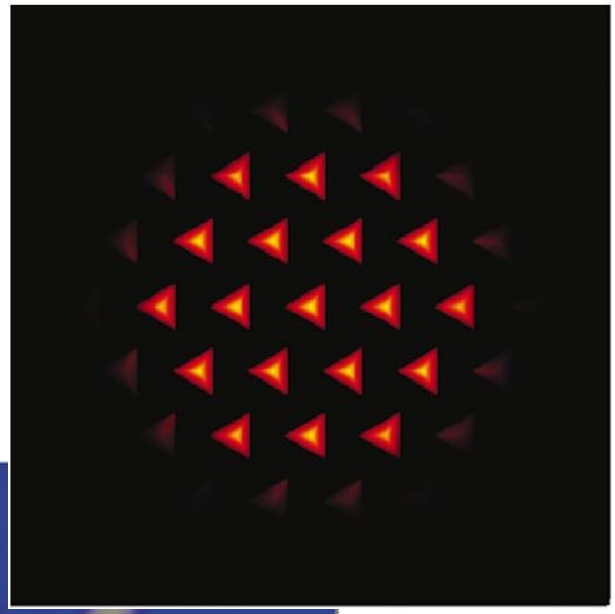
1D and 2D Quantum Dot Arrays

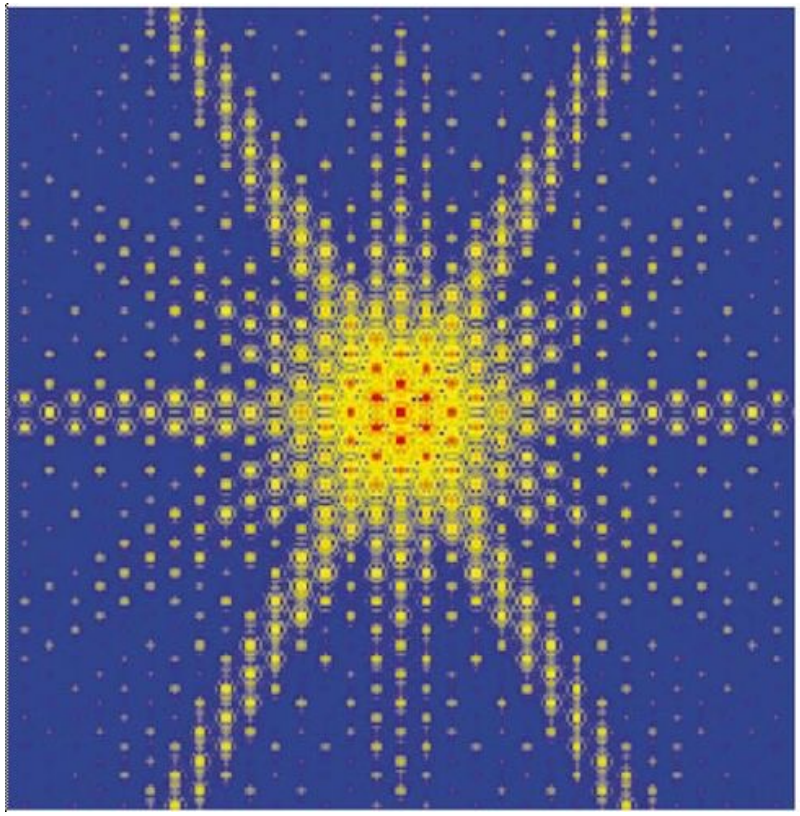
Zhenyang Zhong, G. Bauer, Johannes Kepler Universität Linz

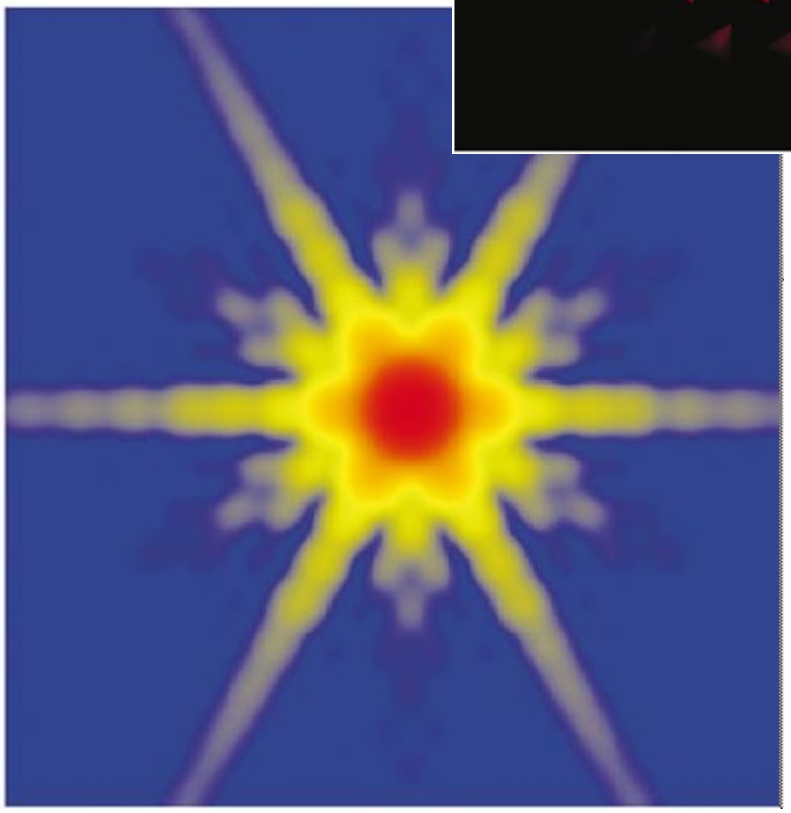


Vary Coherence (model)

I. A. Vartanyants and I. K. Robinson,
J. Sync. Rad. **10** 409 (2003)

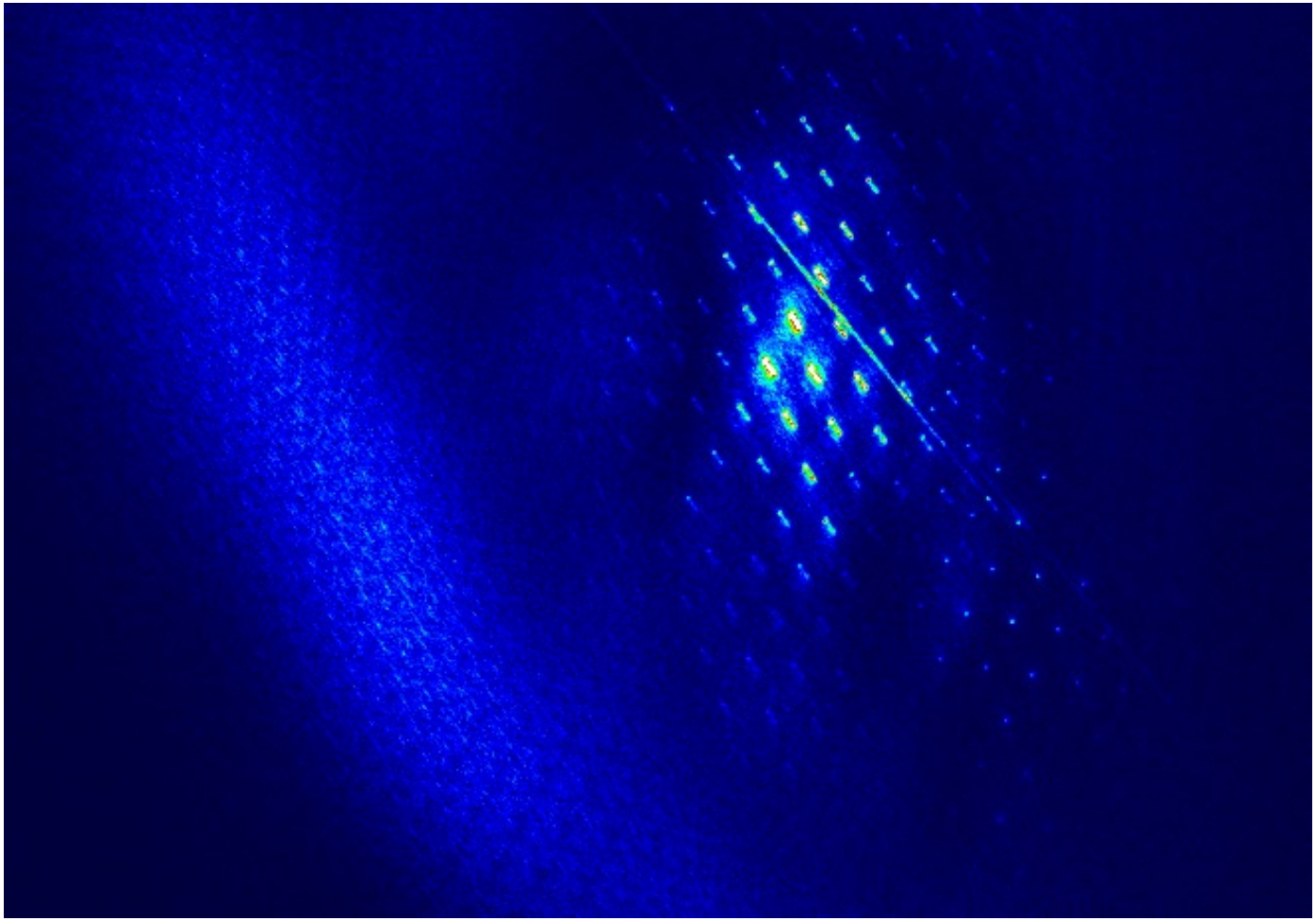




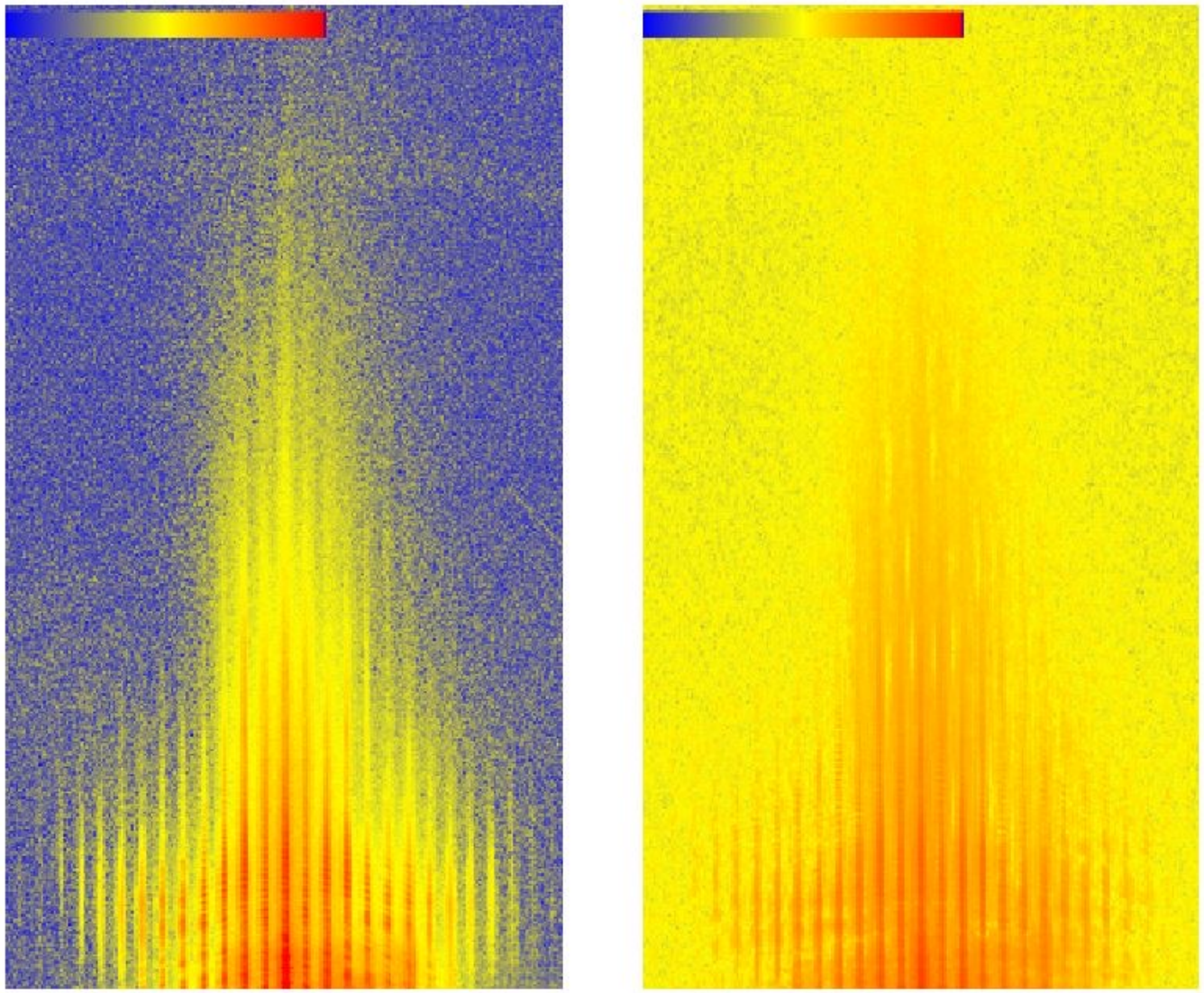


202 Diffraction from Ge QD array

with G. Bauer, Z. Zhong and T. H. Metzger

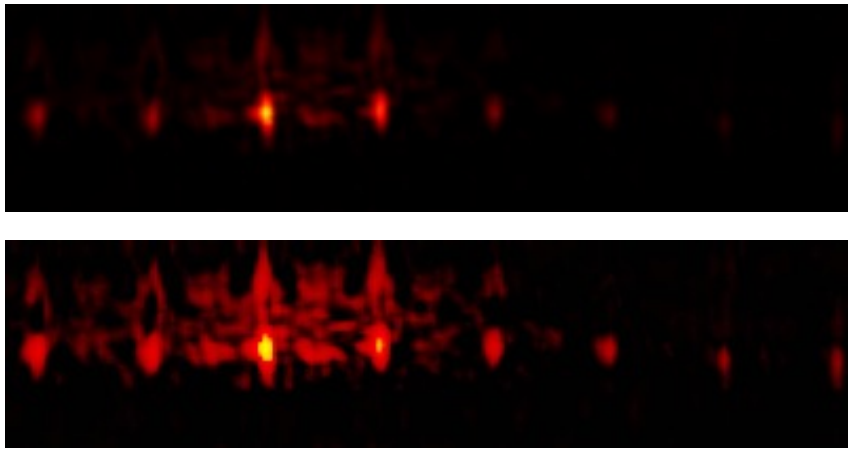


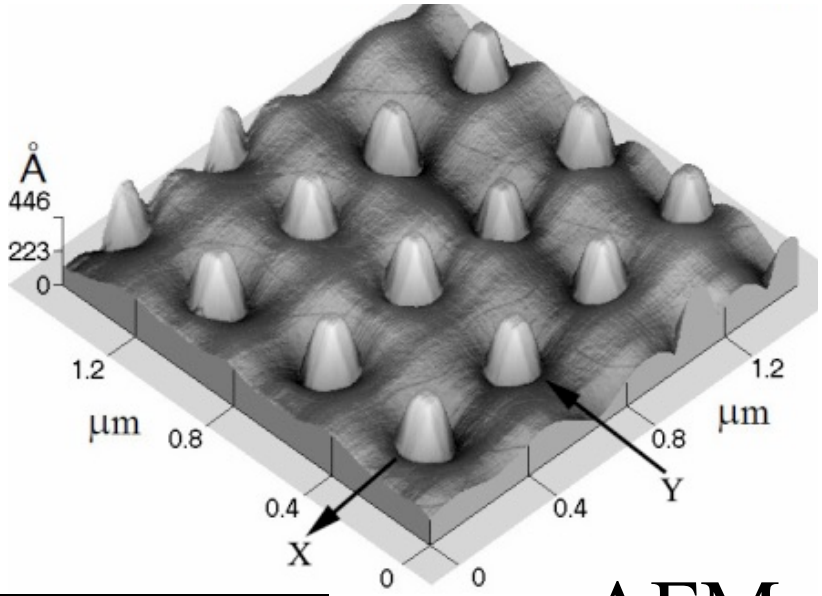
Result of fitting GISAXS data



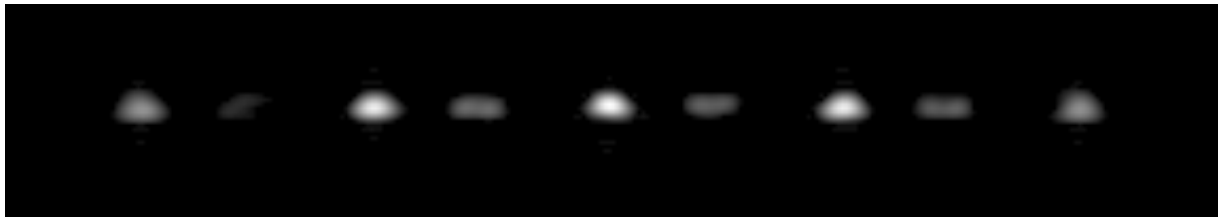
Images from Fitting GISAXS

with Jeff Onken



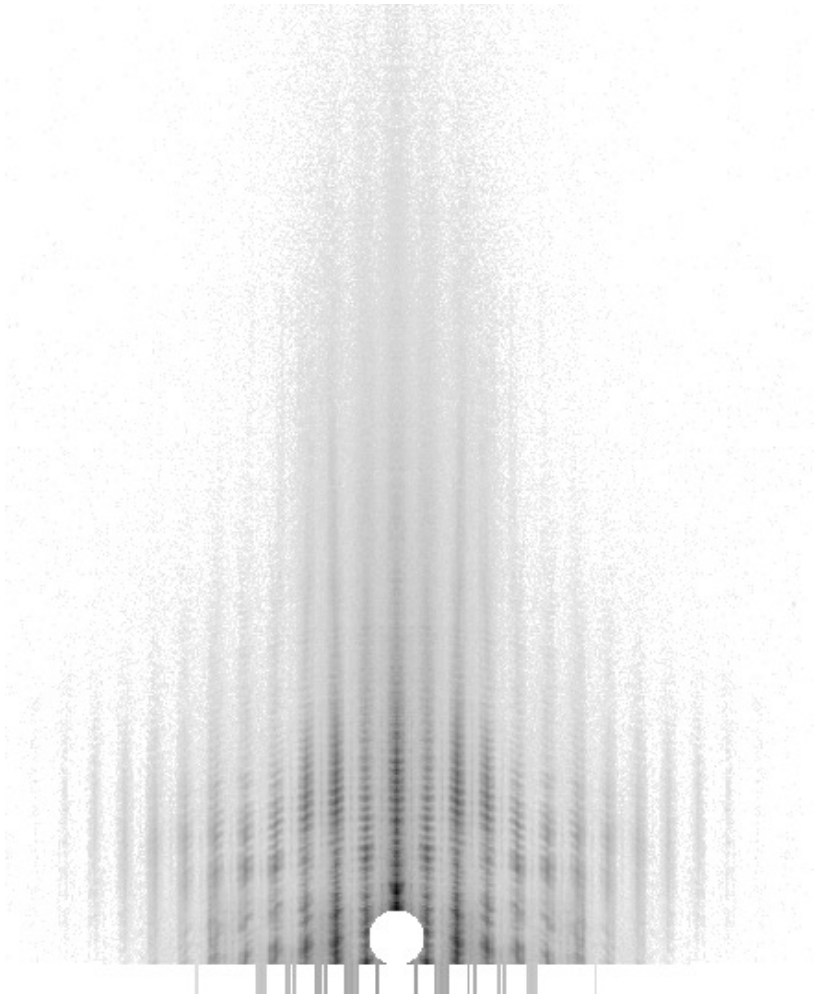


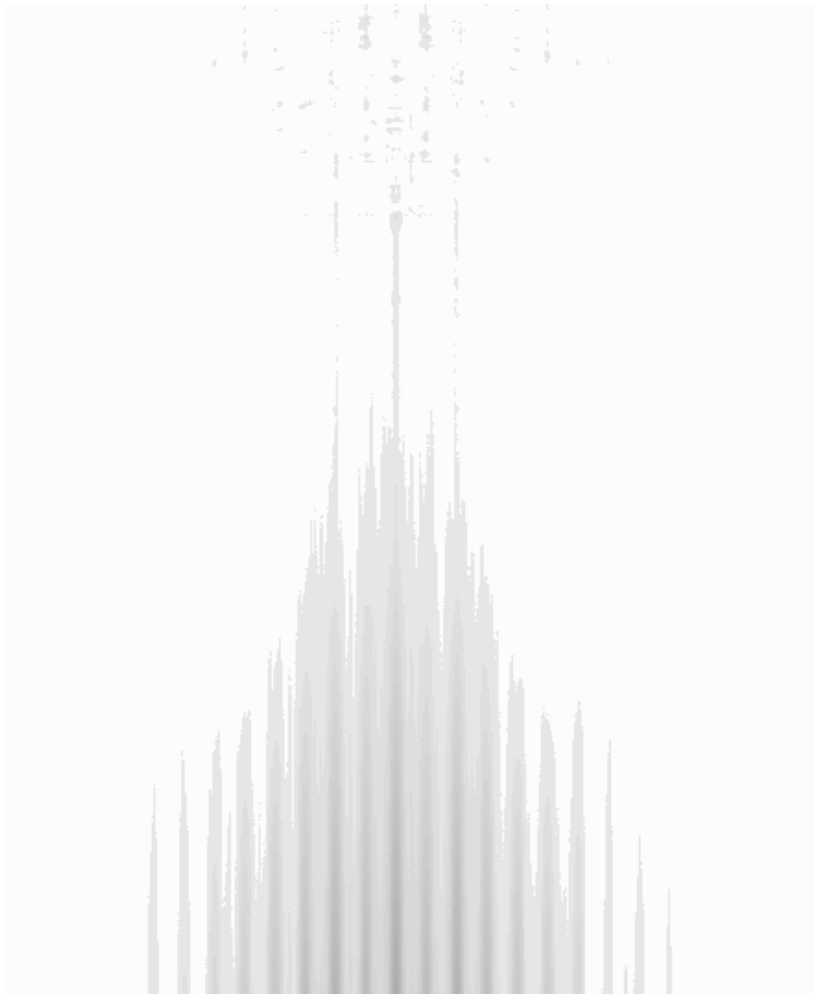
AFM



Mask used as support

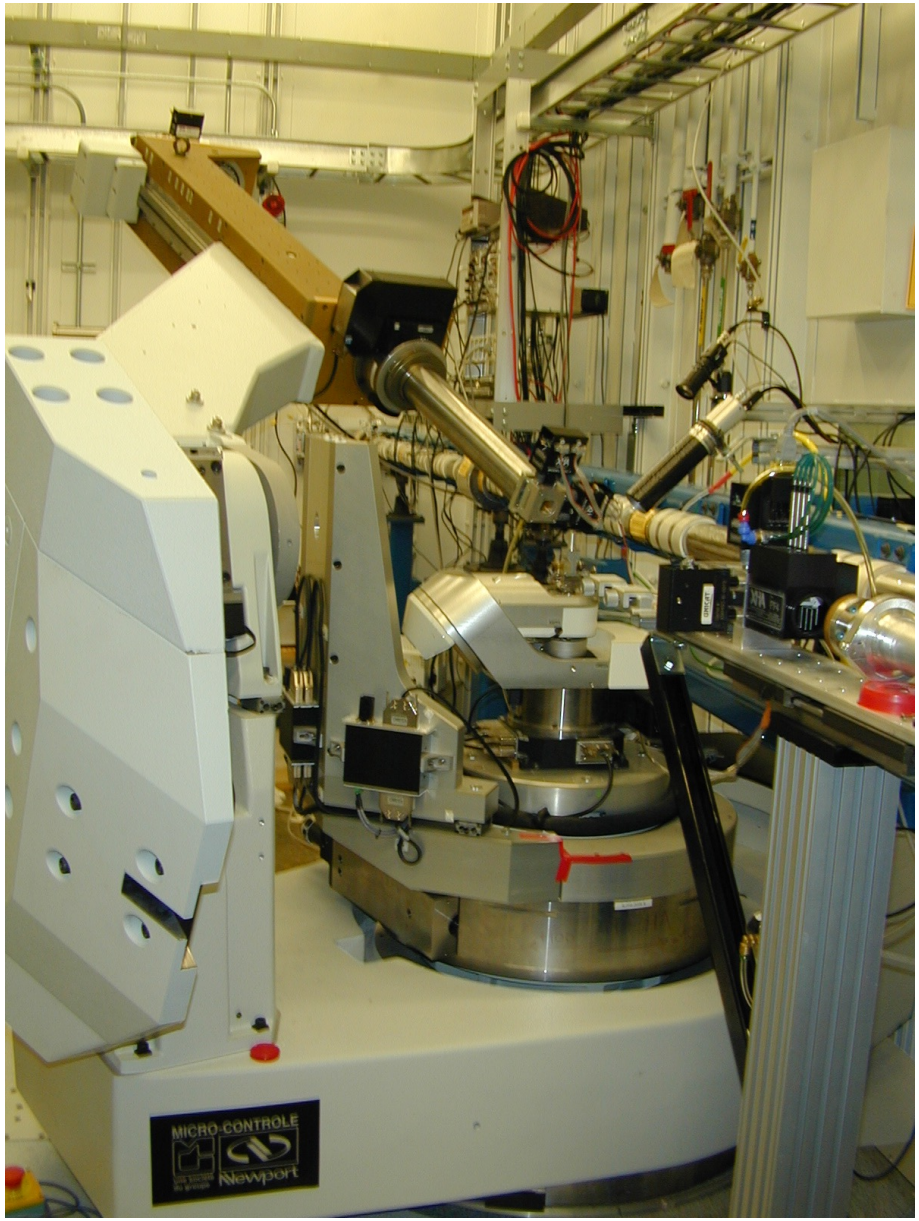
Diffraction pattern under constraint

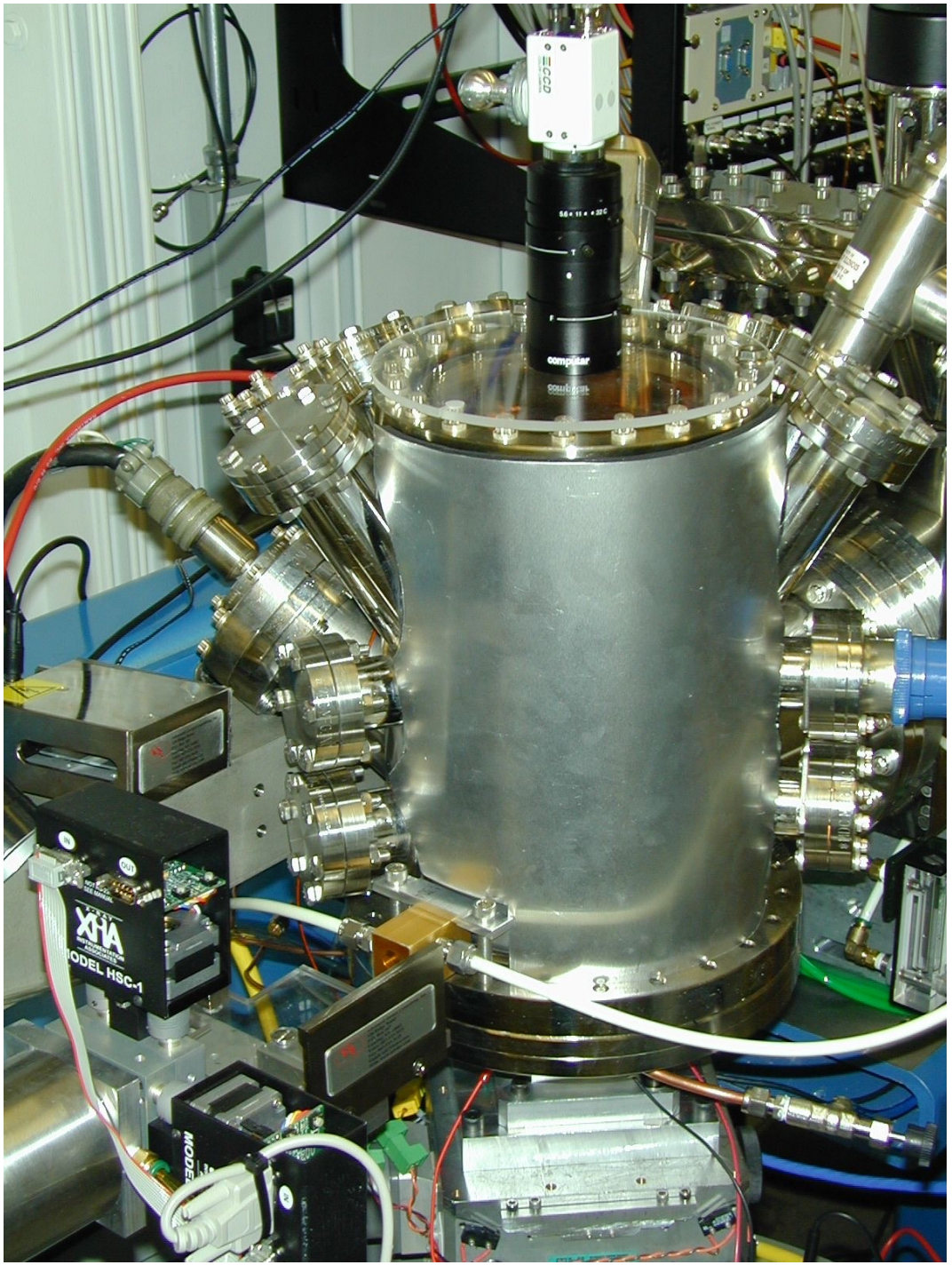


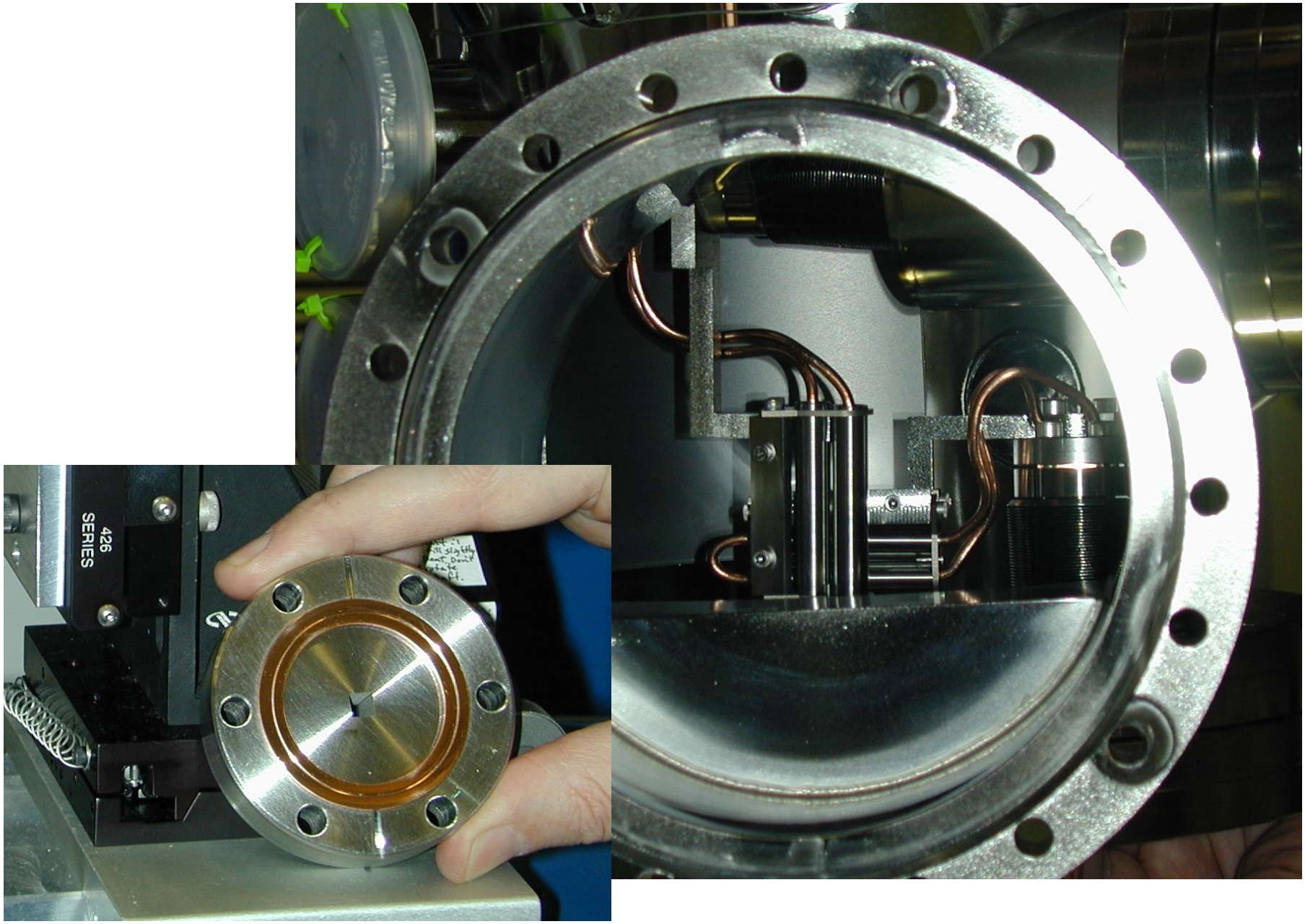


34-ID-C Capabilities

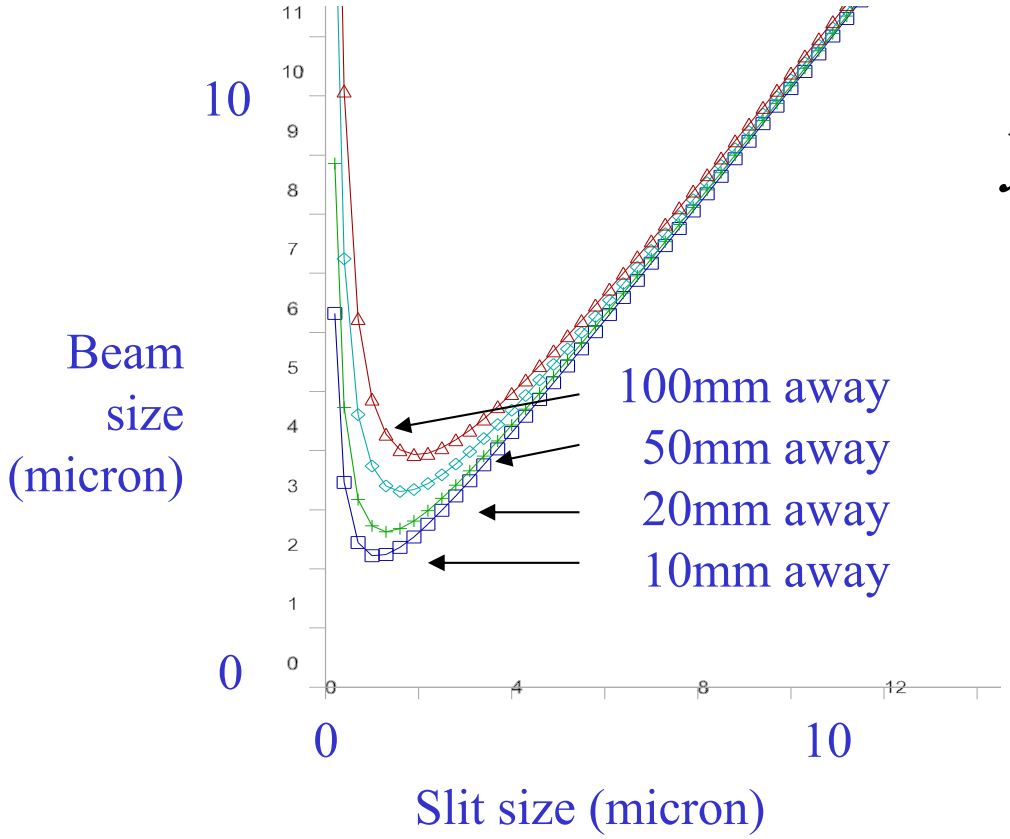
- Beam-splitting mirror
  - cuts out half of central cone
- Double Crystal Monochromator
  - Diamond, Si(111) and Si(220)
- Coarse and Fine coherence slits
  - UHV Roller-blade design
- Long-arm Microcontrole Diffractometer


MICRO-CONTROLE
Newport

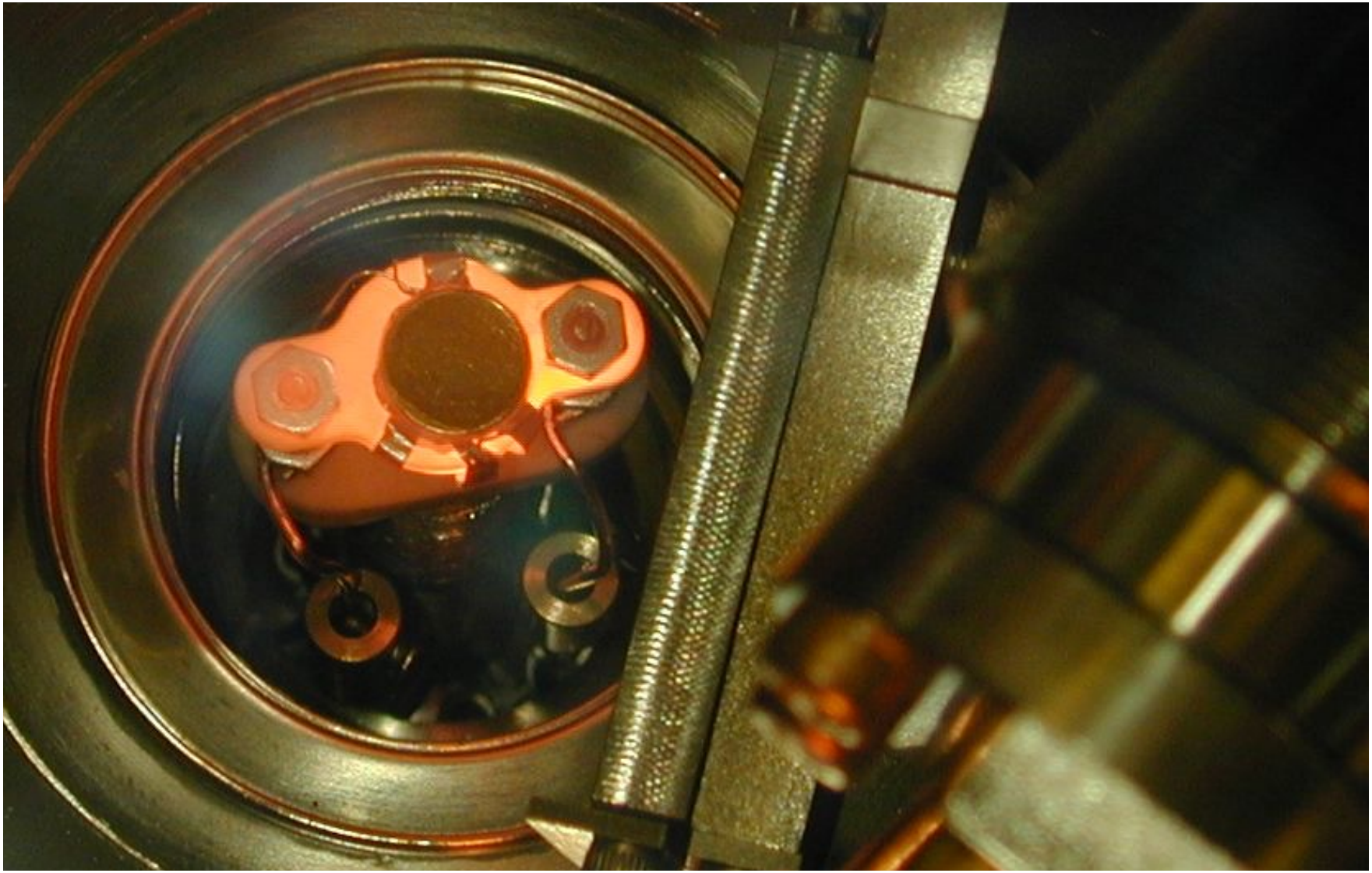

computar
MODEL HSC-1

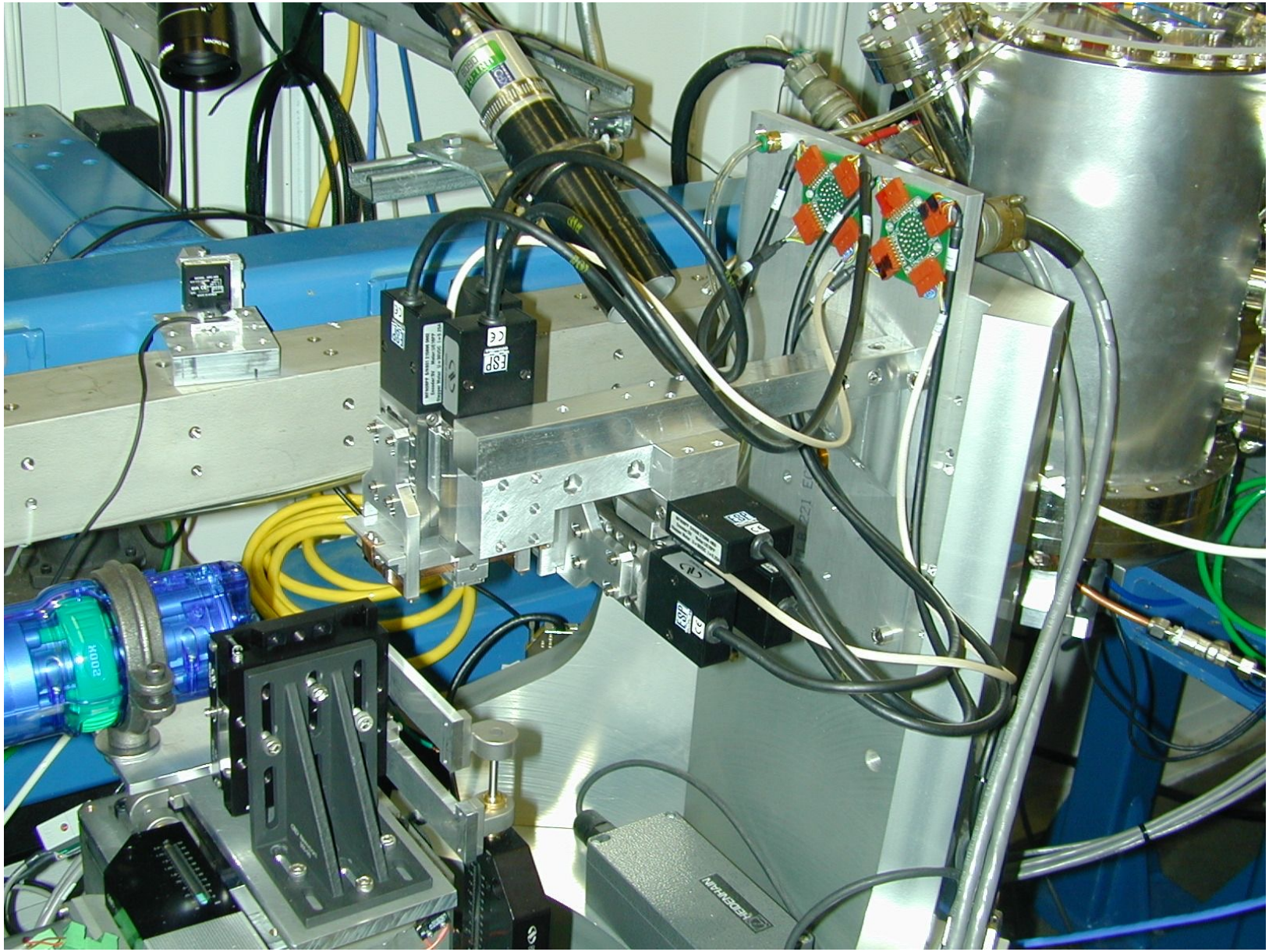

426
SERIES

Smallest Beam using Slits (9keV)

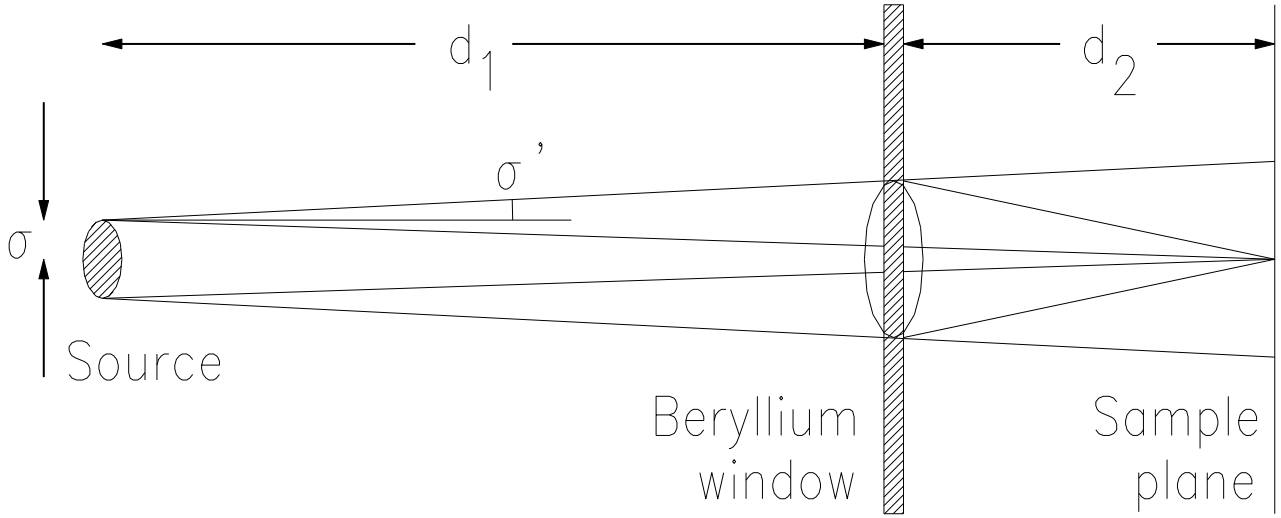


$$y(x) = x + \frac{\lambda d}{x}$$





Window as a Secondary Source

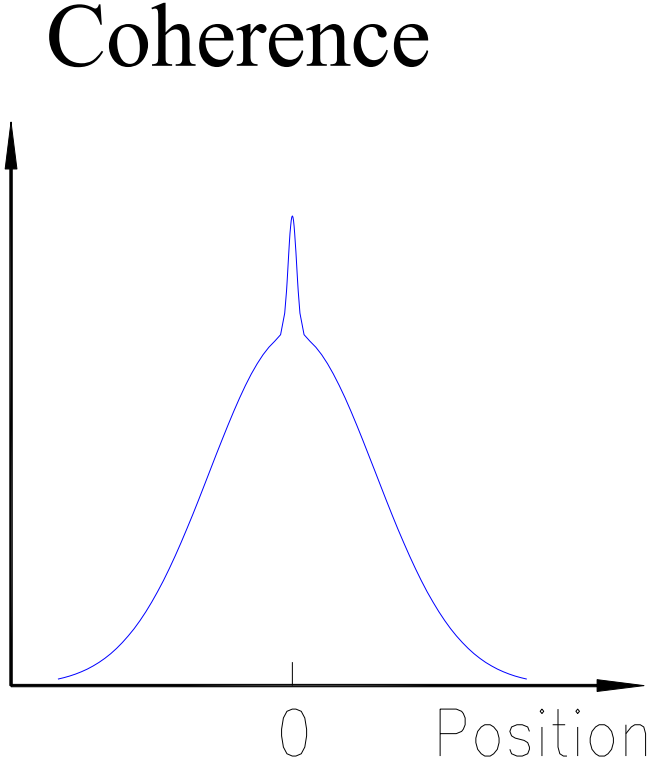


$$L_T = \frac{\lambda D}{2\sigma}$$

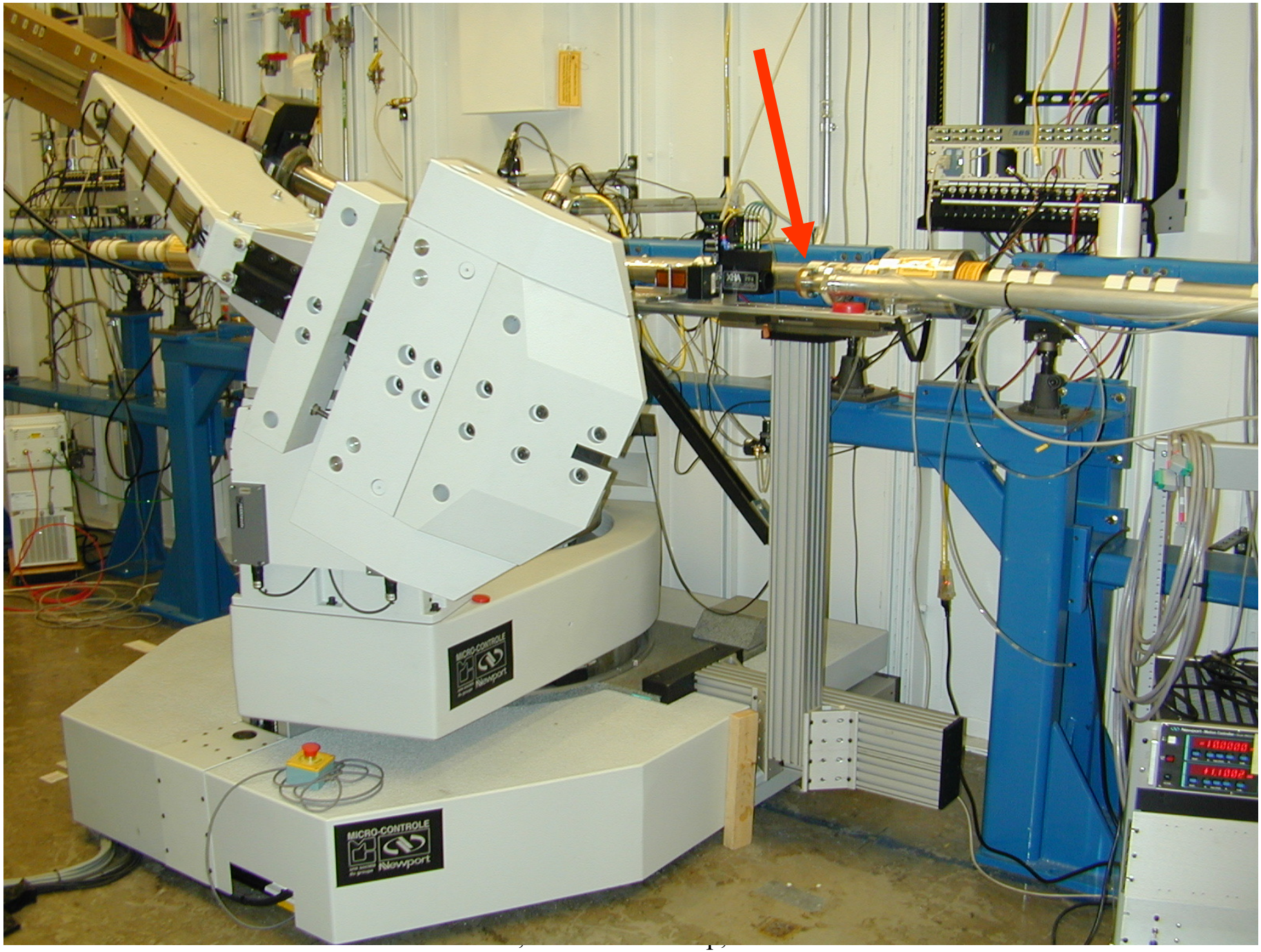
$$L_{T2} = \frac{\lambda D}{2\sigma} \frac{(1-\frac{d_1}{D})}{(1+\frac{\sigma' D}{\sigma}\frac{d_1}{D})}$$

Vert ~50
Horiz ~3

Two Components to MCF

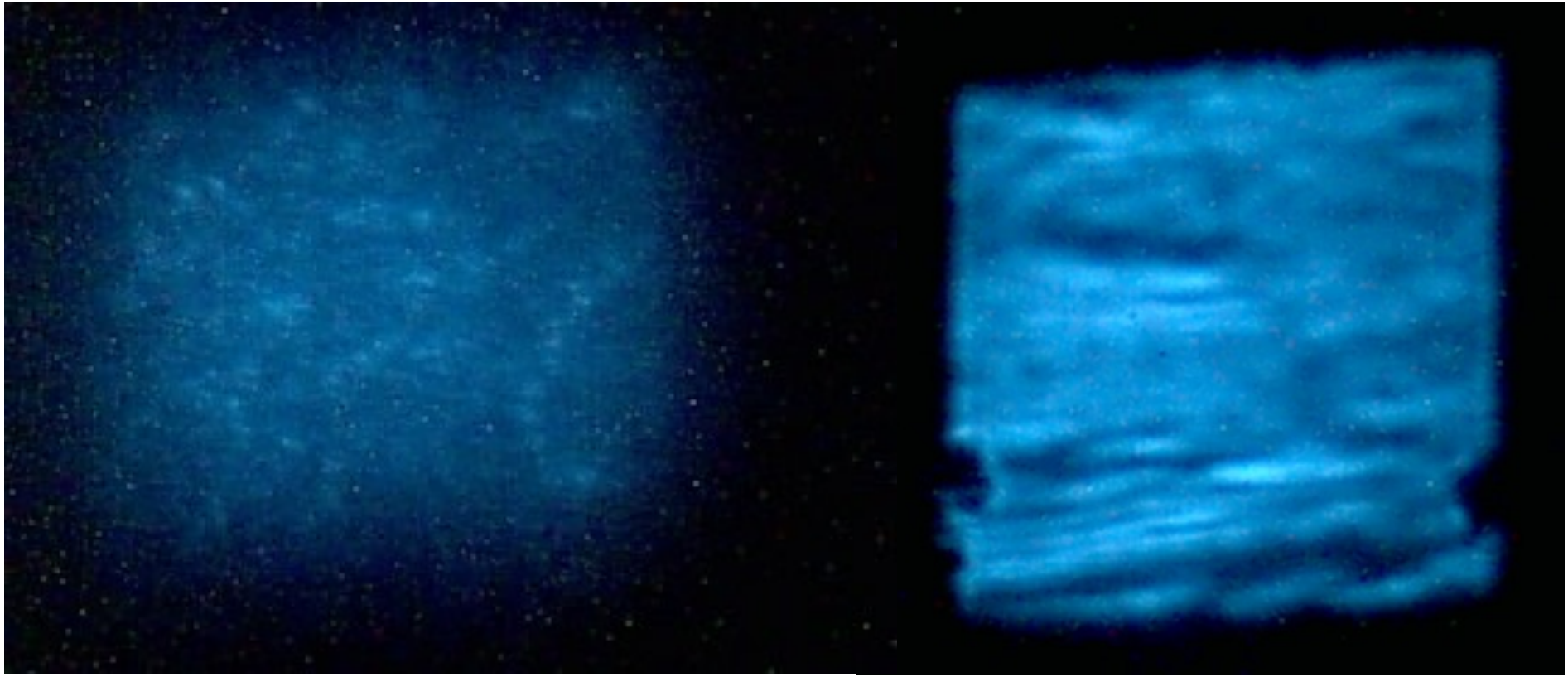


- Width of narrow component is very sensitive to location of window
- Amplitude of narrow component depends on level of diffuse scattering from window
- Amplitude increases with distance (for phase object)


MICRO-CONTROLE
Newport
MICRO-CONTROLE
Newport

BN Coating on Sector 34 Window

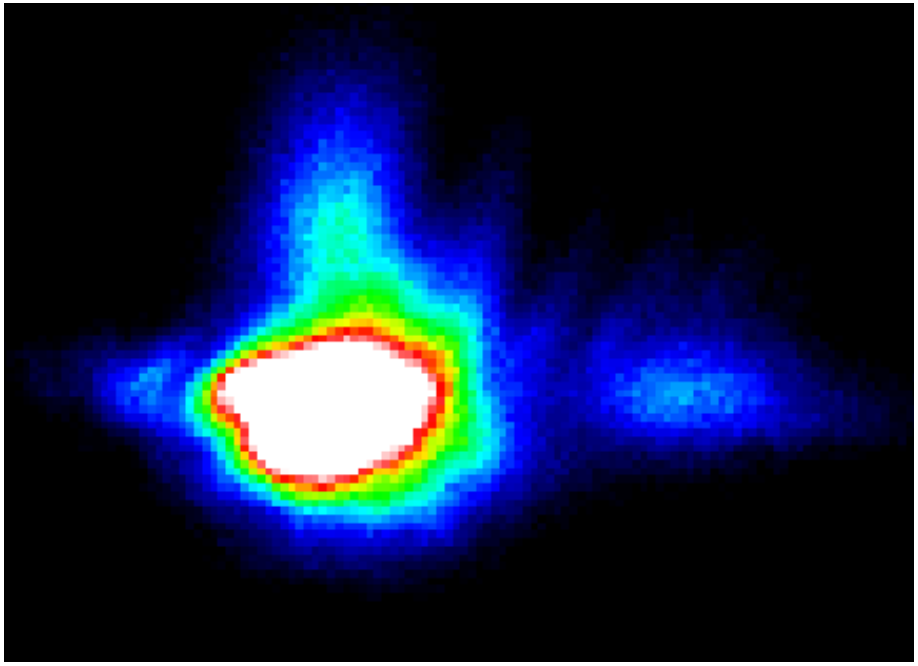
protects against corrosion of Window by pink beam



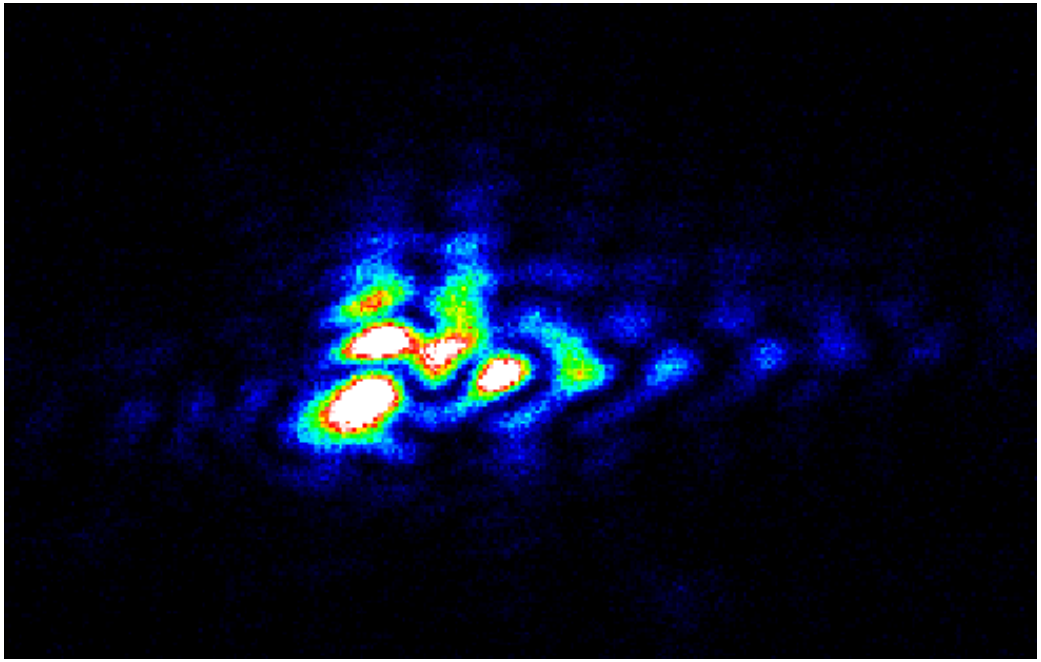
BN spray coating

Coating removed

Au Nanocrystal CXD at Sector 34



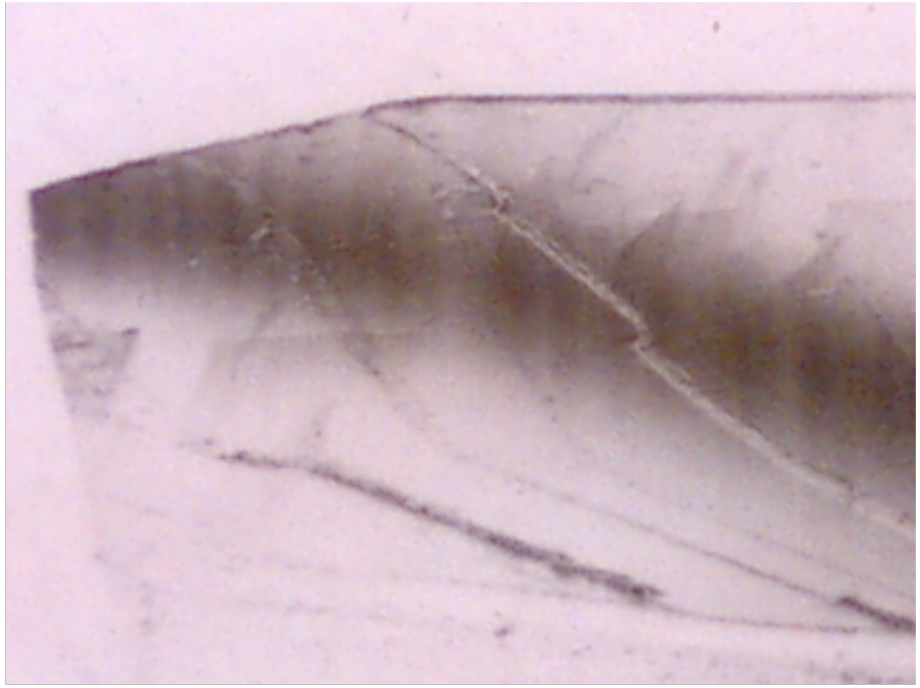
BN-coated window



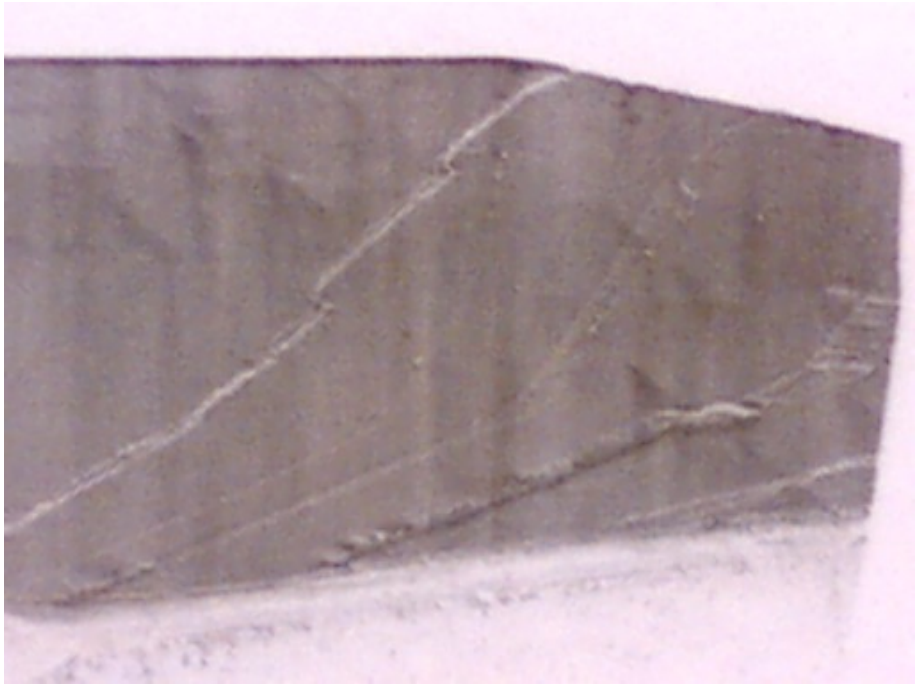
BN removed Sept 2002

Topography of Diamonds

Measured at 33-BM on Kodak dental film



Second crystal, as mounted

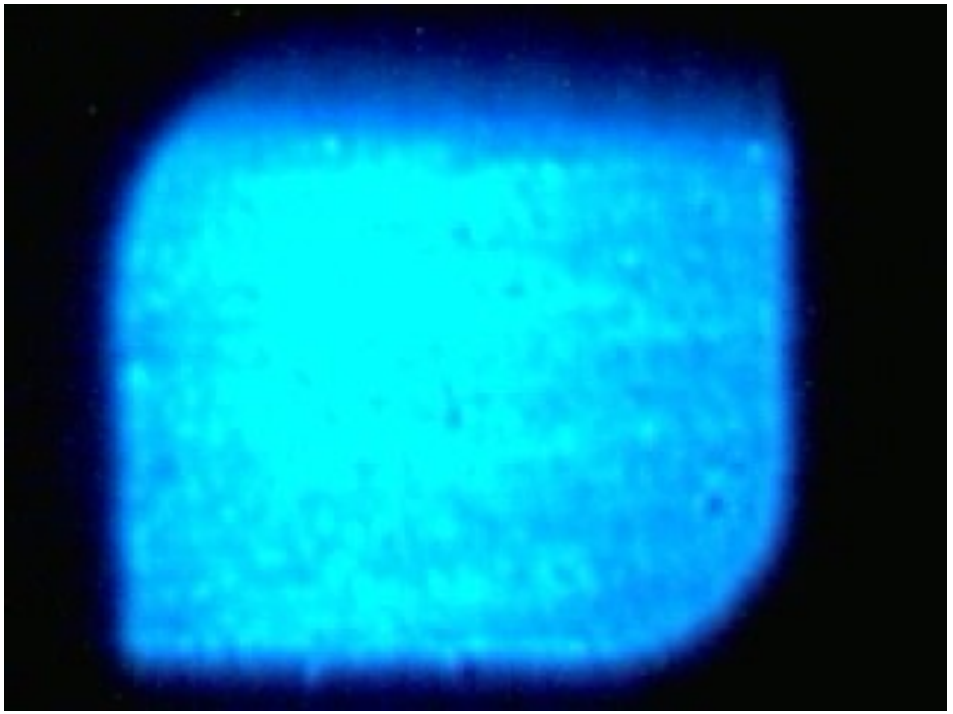


After losening clamping screws

Switch to Si(111) monochromator

Scan table to watch topography

Diamond



Silicon

Conclusions and Outlook

- Inversion of CXD to spatial images
- Internal structure of Au Nanocrystals in 3D
- Phasing by computation instead of lens
- Possible to obtain complex images
- Images of Quantum Dots in an array
- Intimate relationship of samples and beamline