Coherent X-ray Diffraction at the Advanced Photon Source

Ian Robinson
Garth Williams
Mark Pfeifer
Ivan Vartaniants
Franz Pfeiffer
Wei Zhang
Yang Da

University of Illinois
Sector 34-ID-C

DLS Coherence Workshop
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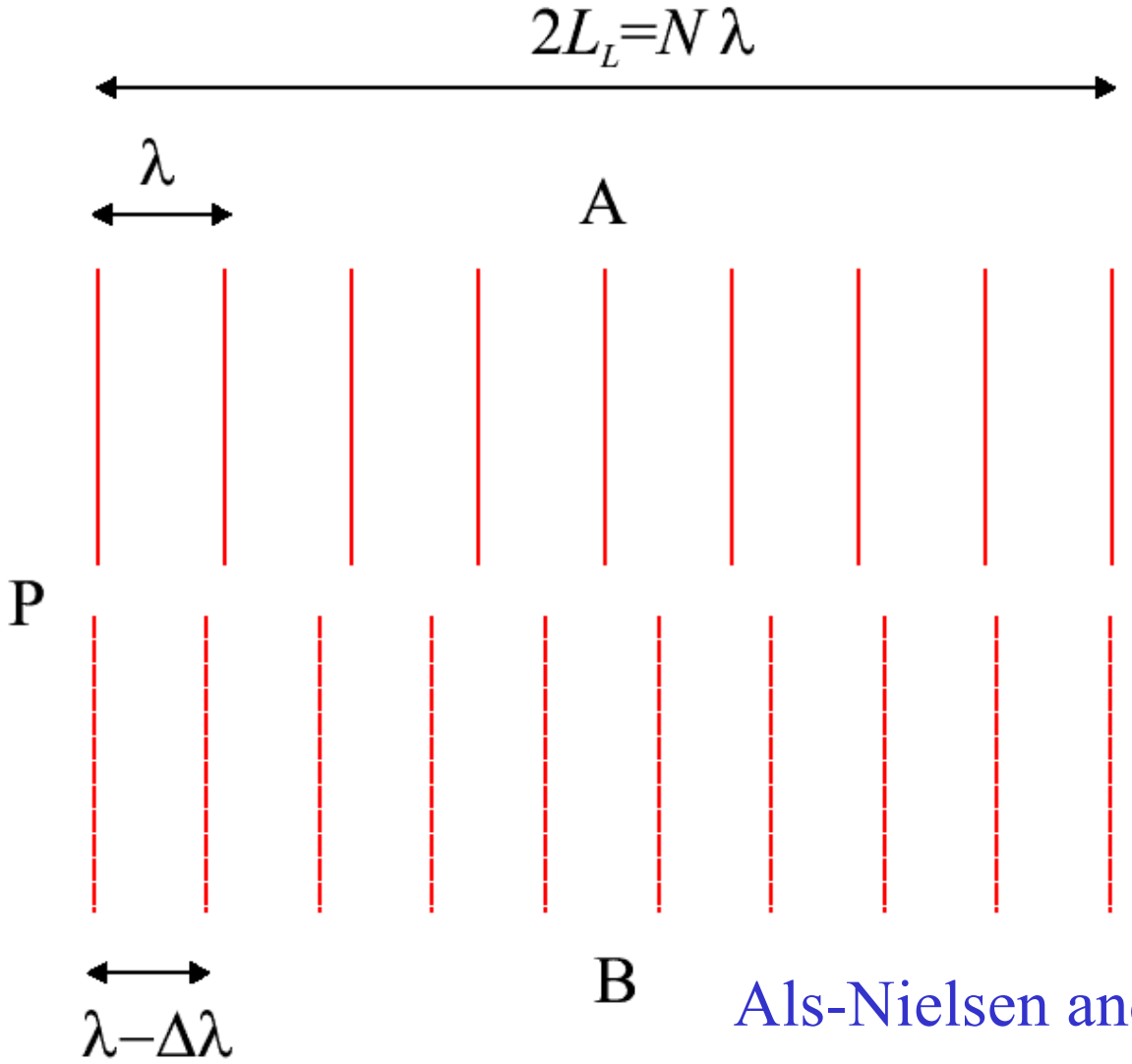
Outline

- Coherence in Diffraction
- CXD Beamline at APS
- How to Solve the Phase Problem
- Nanocrystal Shapes
- Use of Optics in CXD

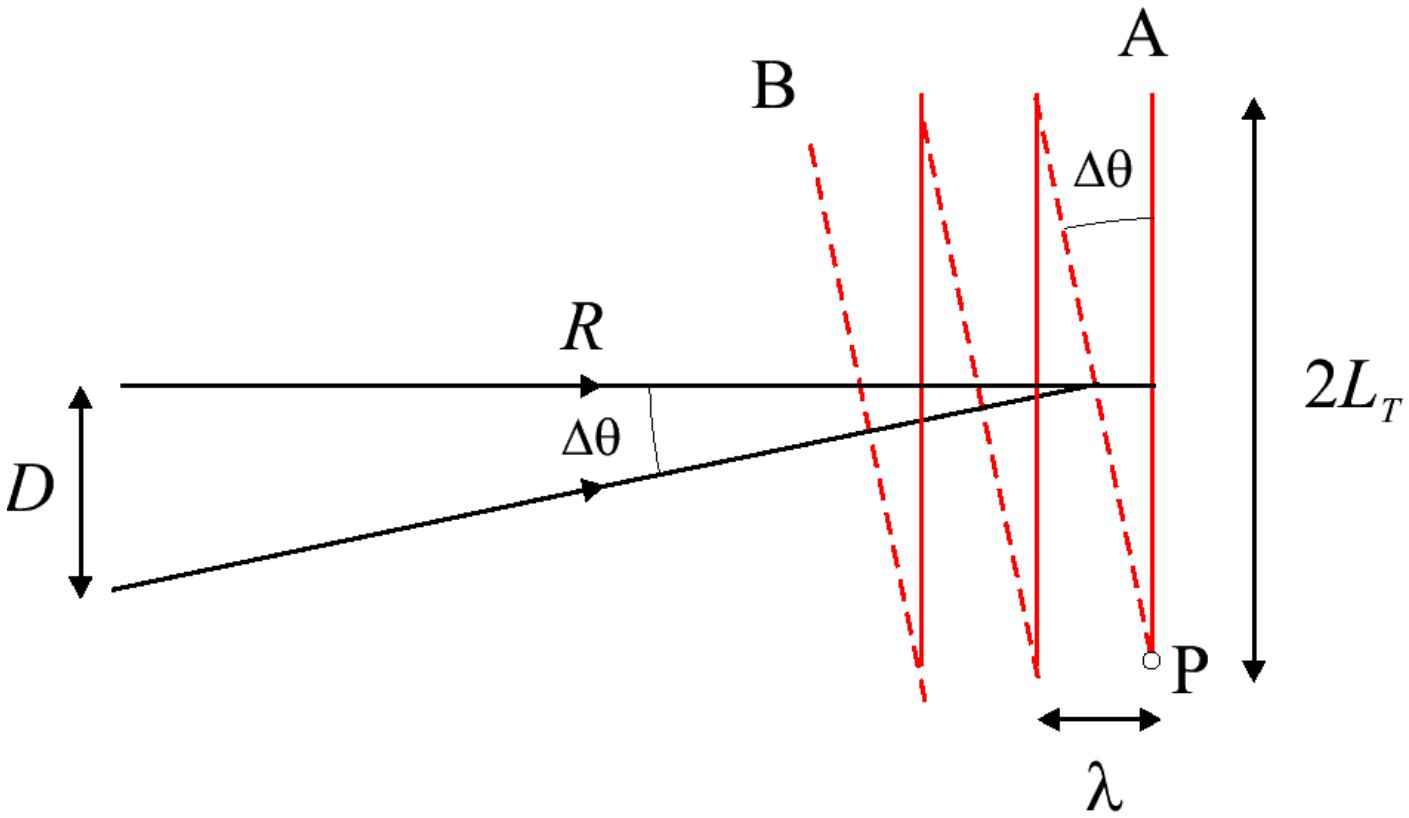
Longitudinal Coherence



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

Als-Nielsen and McMorrow (2001)

Lateral (Transverse) Coherence



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

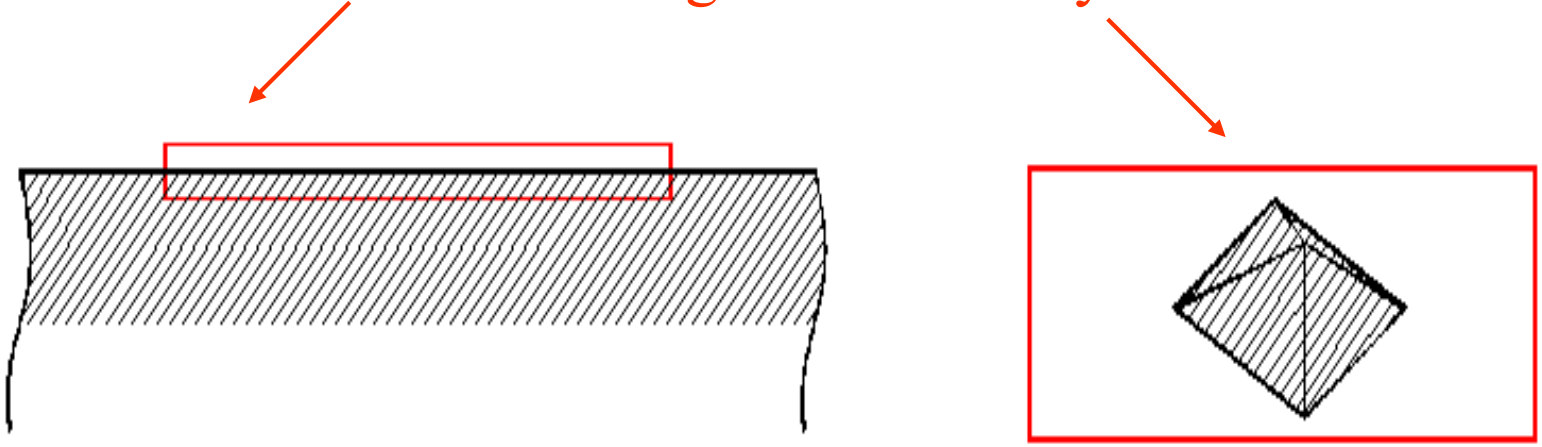
Als-Nielsen and McMorrow (2001)

Coherence at the APS or ESRF

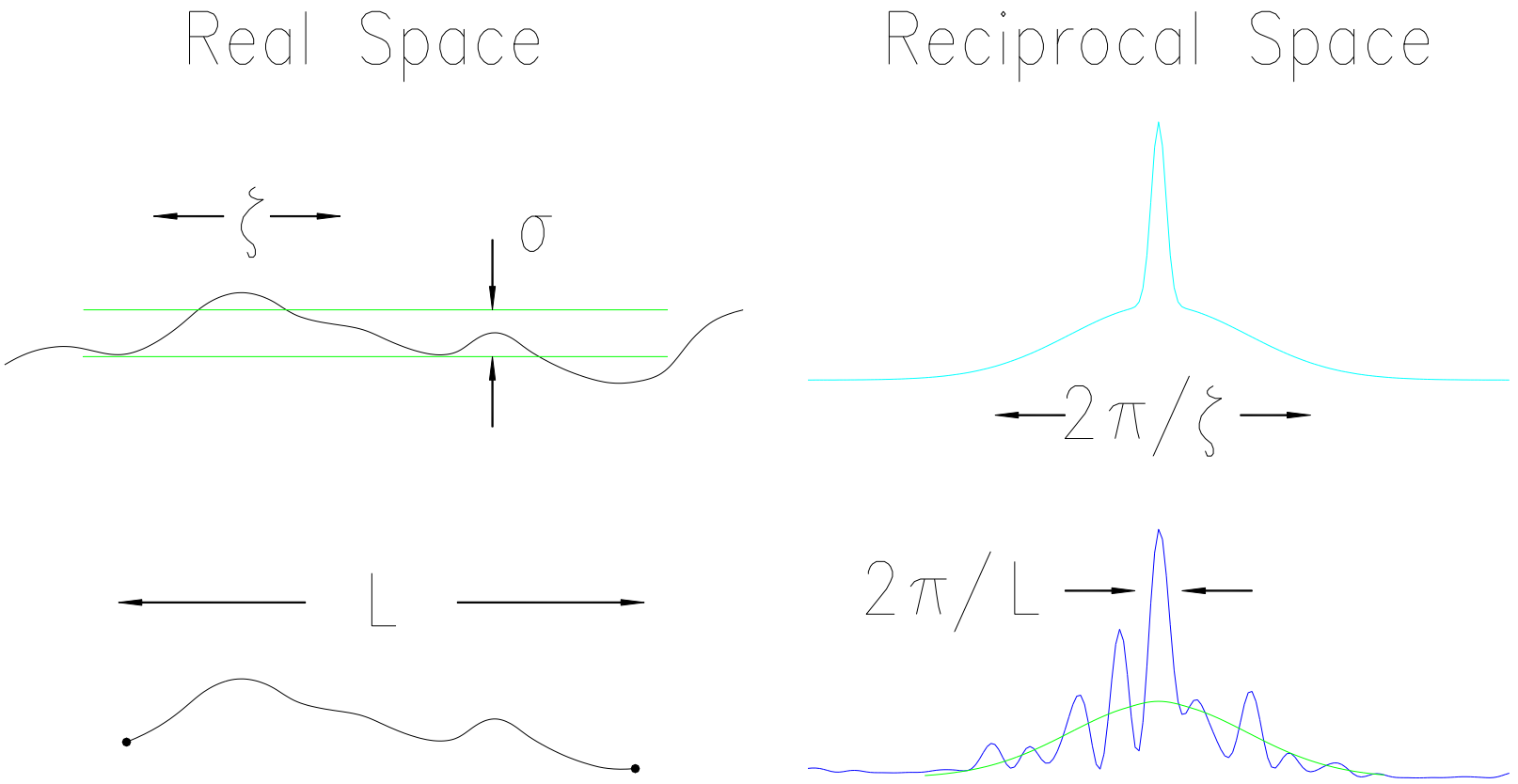
Typical of 3rd Generation (undulator) Synchrotron Source

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

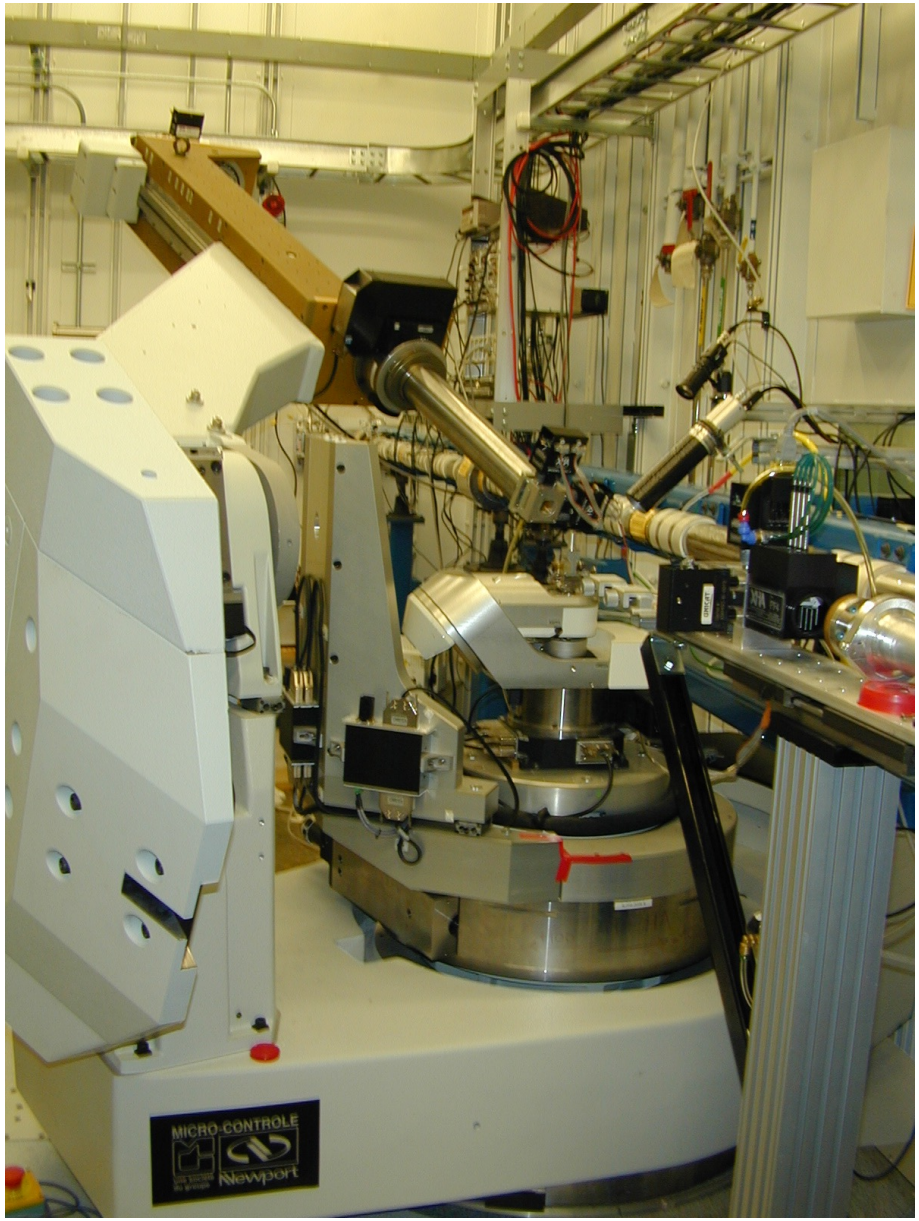


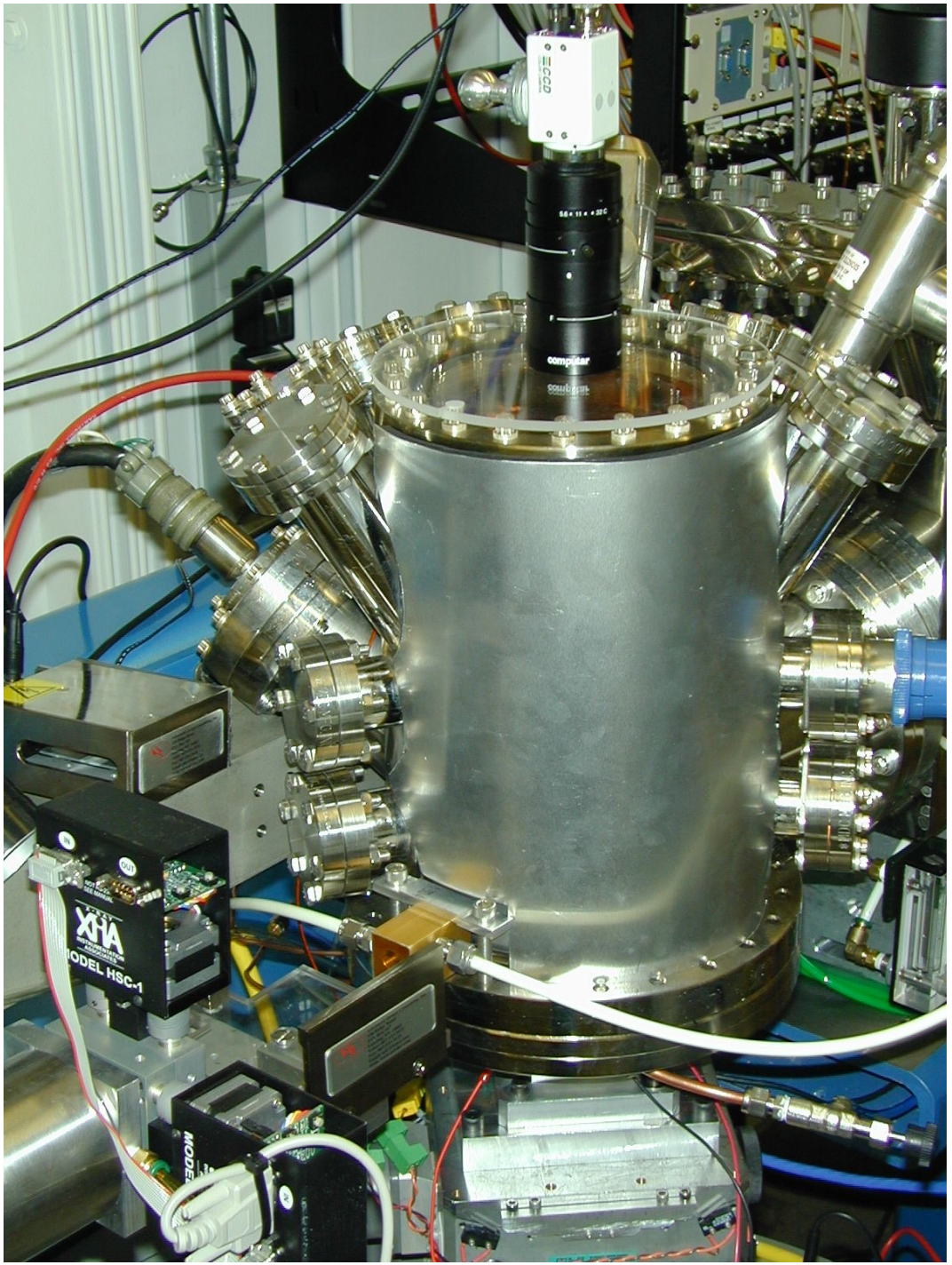
Diffuse Scattering acquires Structure using CXD

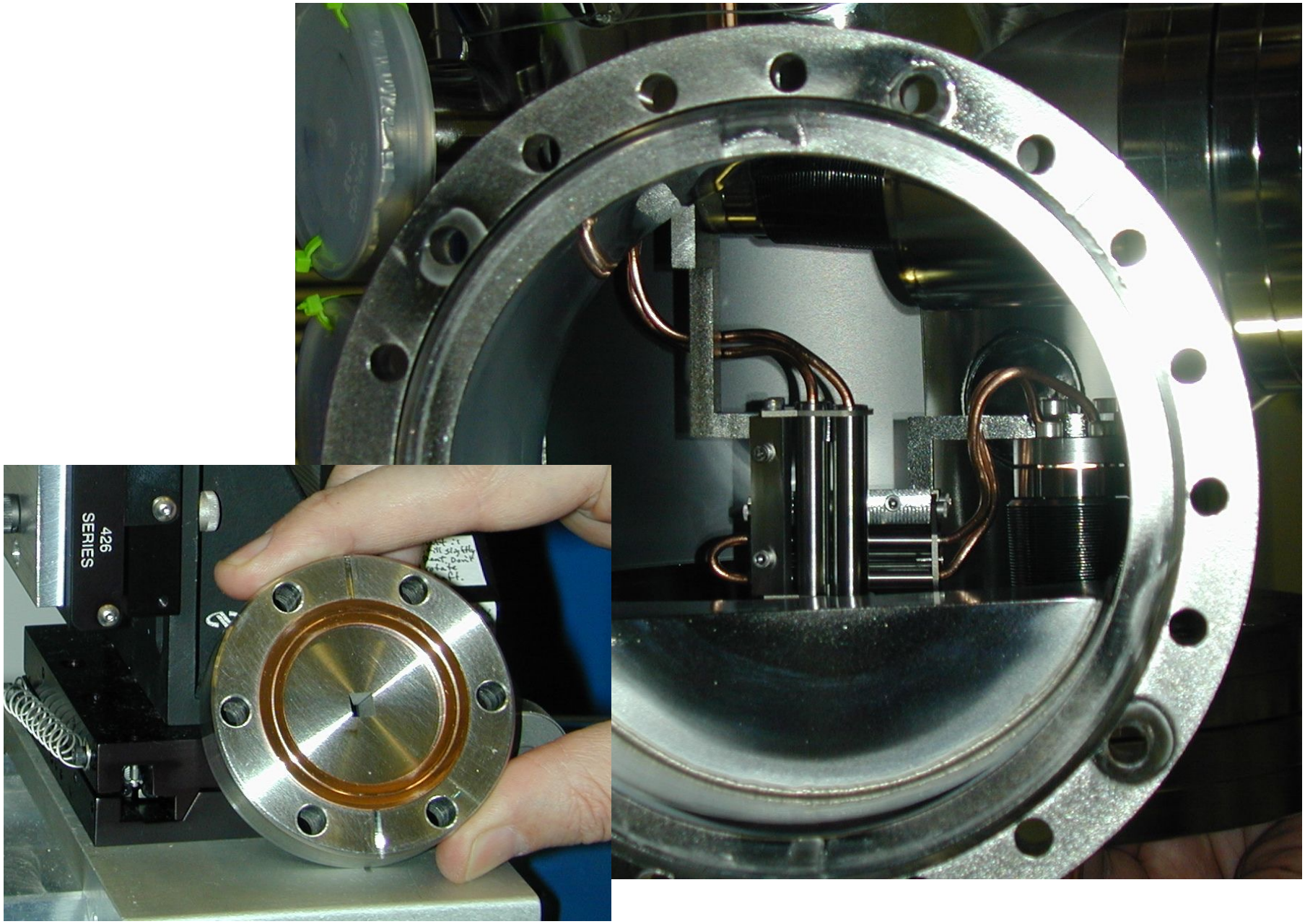


34-ID-C Capabilities

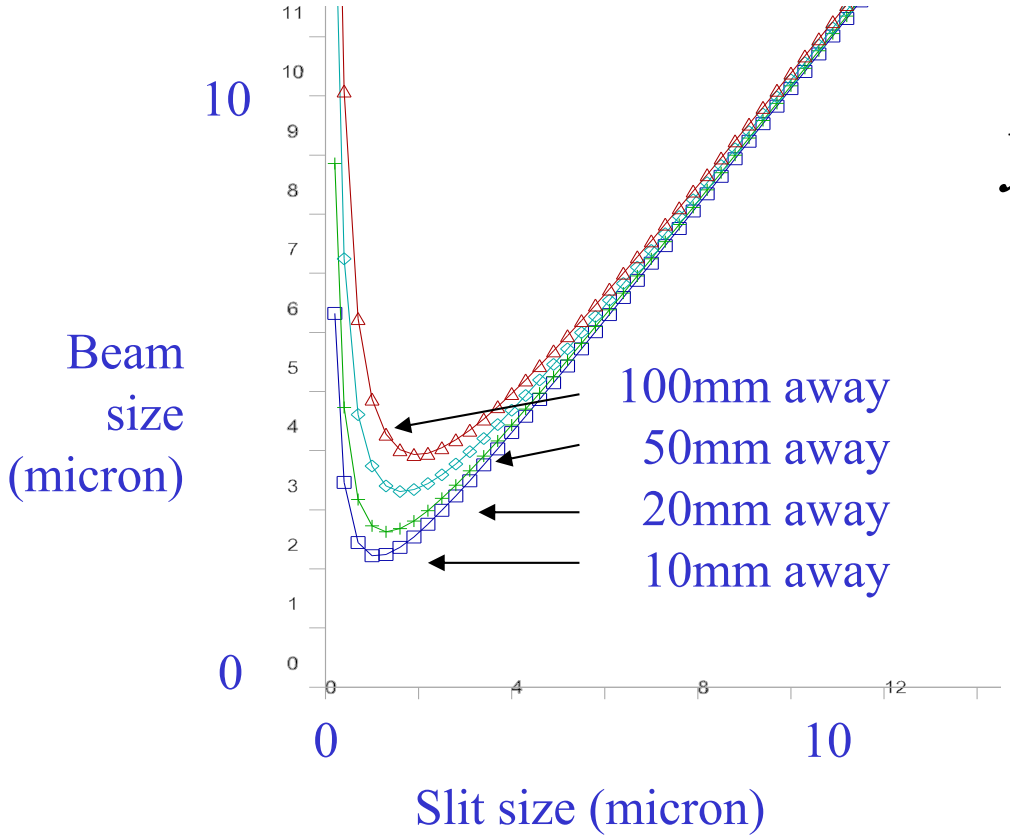
- Beam-splitting mirror
  - cuts out half of central cone
- Double Crystal Monochromator
  - Diamond, Si(111) and Si(220)
- Coarse and Fine coherence slits
  - UHV Roller-blade design
- Long-arm Microcontrole Diffractometer


MICRO-CONTROLE
Newport

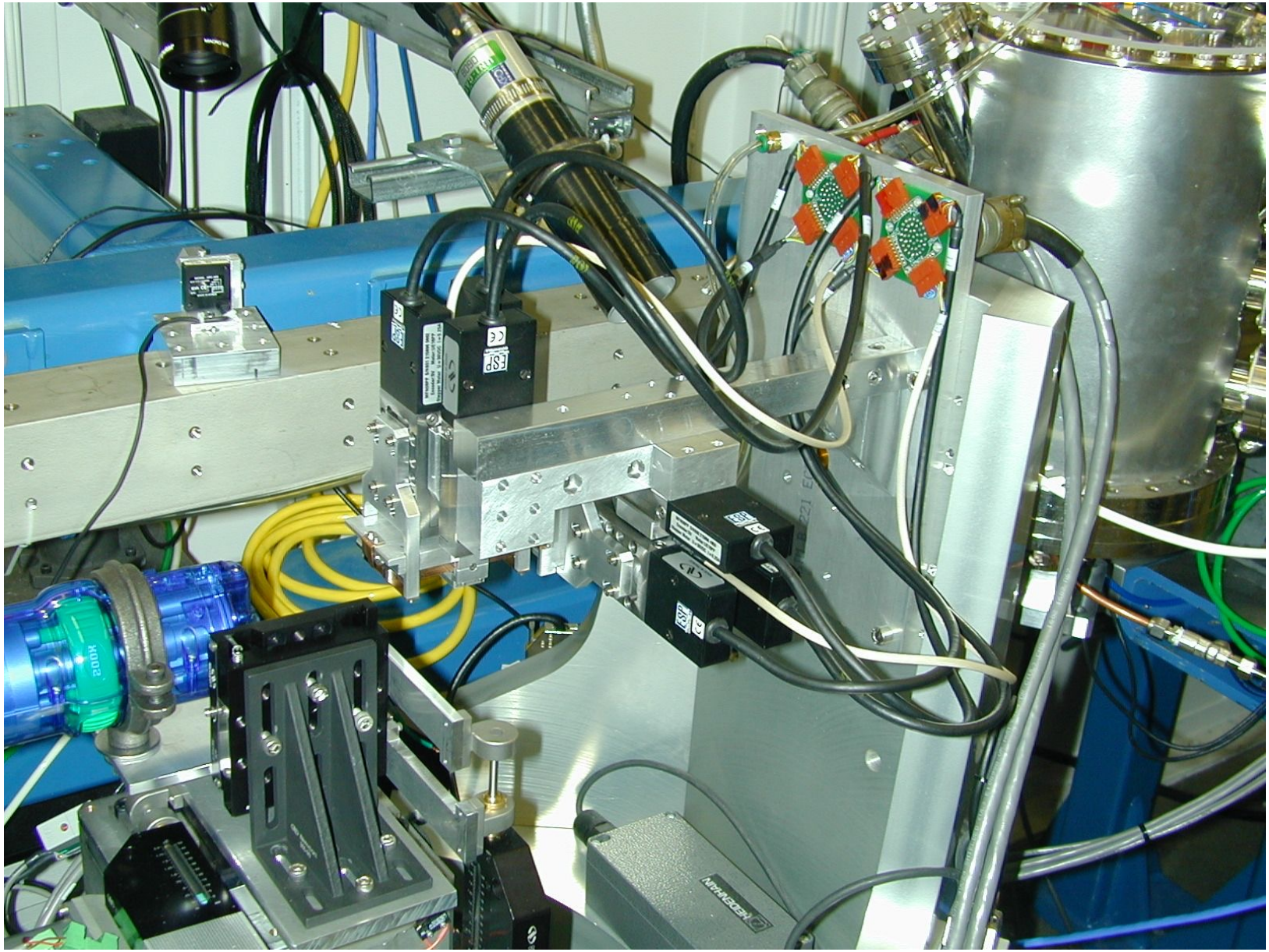

computar
MODEL HSC-1


426
SERIES

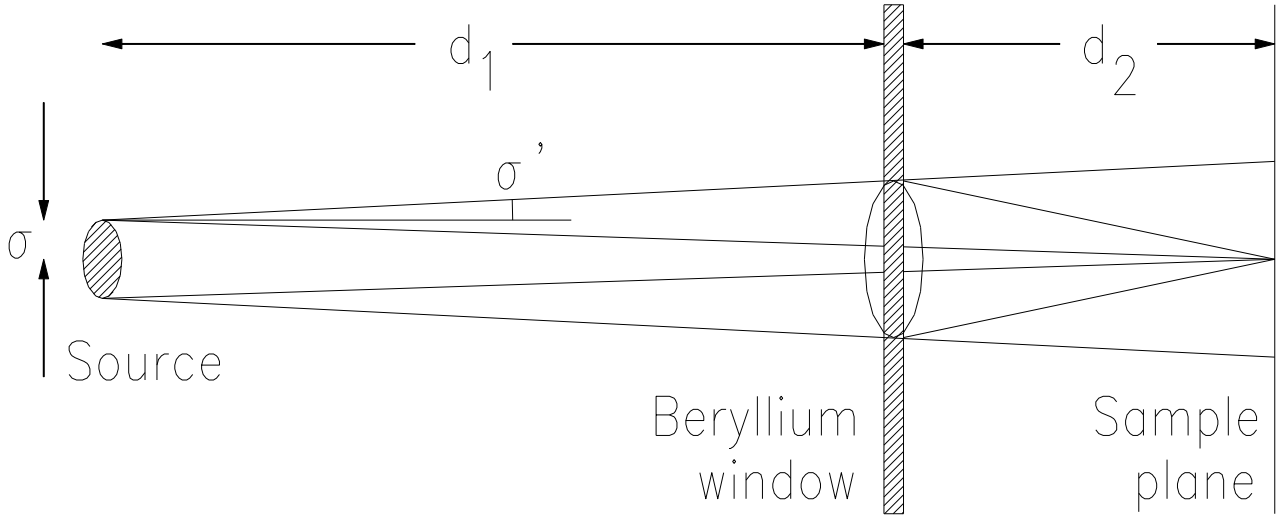
Smallest Beam using Slits (9keV)



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



Window as a Secondary Source

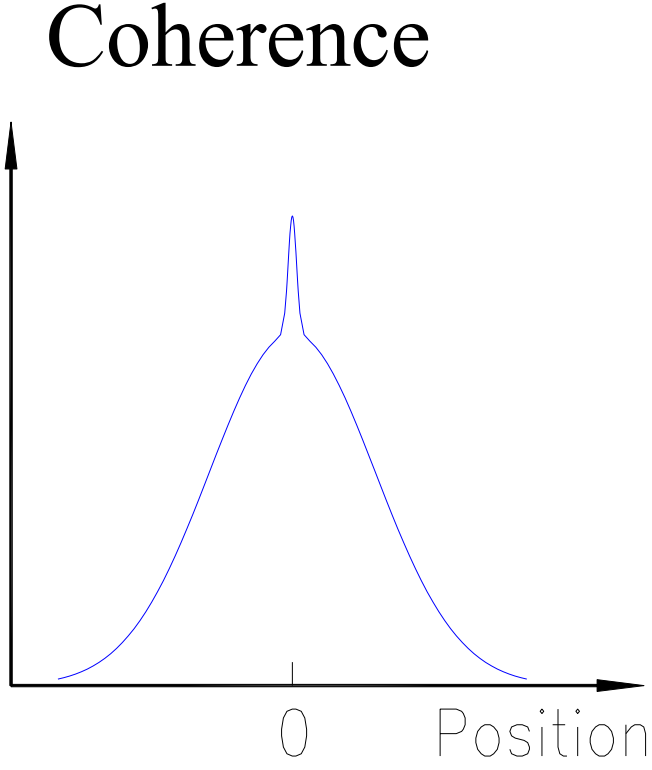


$$L_T = \frac{\lambda D}{2\sigma}$$

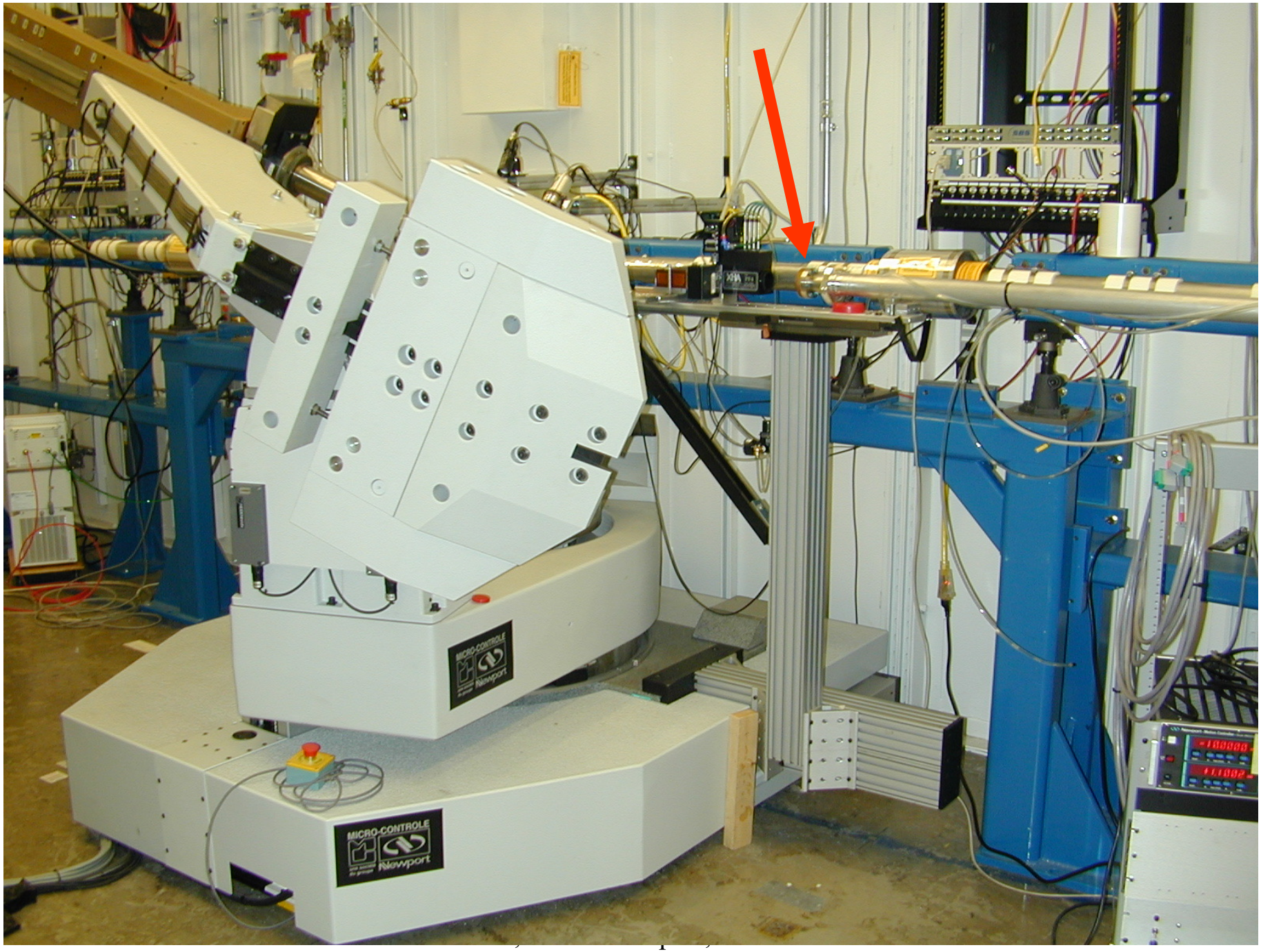
$$L_{T2} = \frac{\lambda D}{2\sigma} \frac{(1-\frac{d_1}{D})}{(1+\frac{\sigma' D}{\sigma}\frac{d_1}{D})}$$

Vert ~50
Horiz ~3

Two Components to MCF

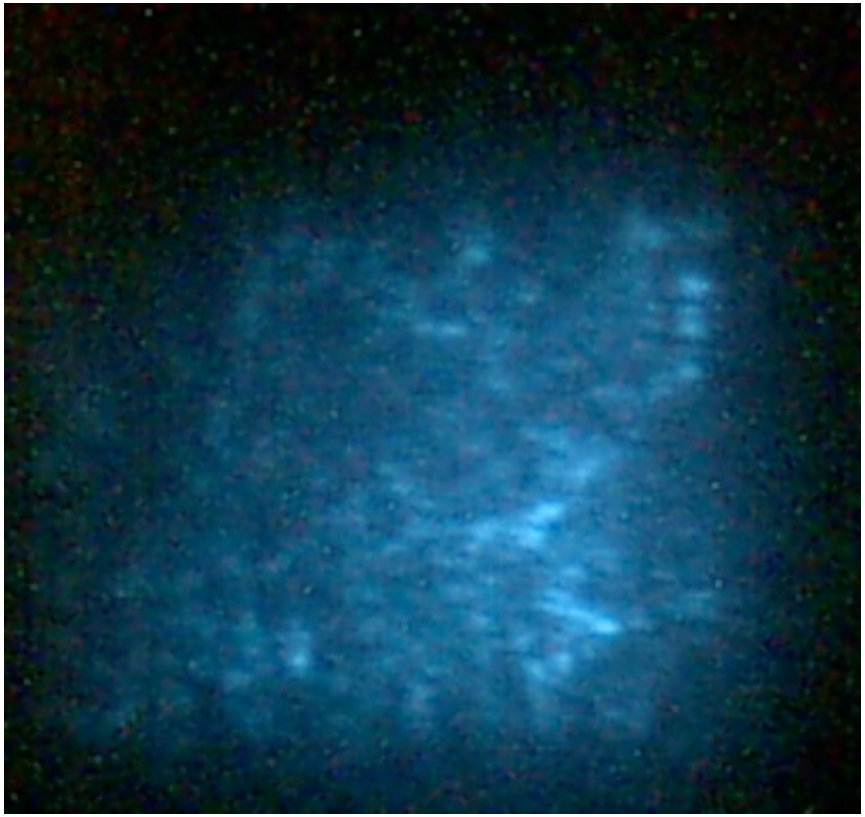


- Width of narrow component is very sensitive to location of window
- Amplitude of narrow component depends on level of diffuse scattering from window
- Amplitude increases with distance (for phase object)

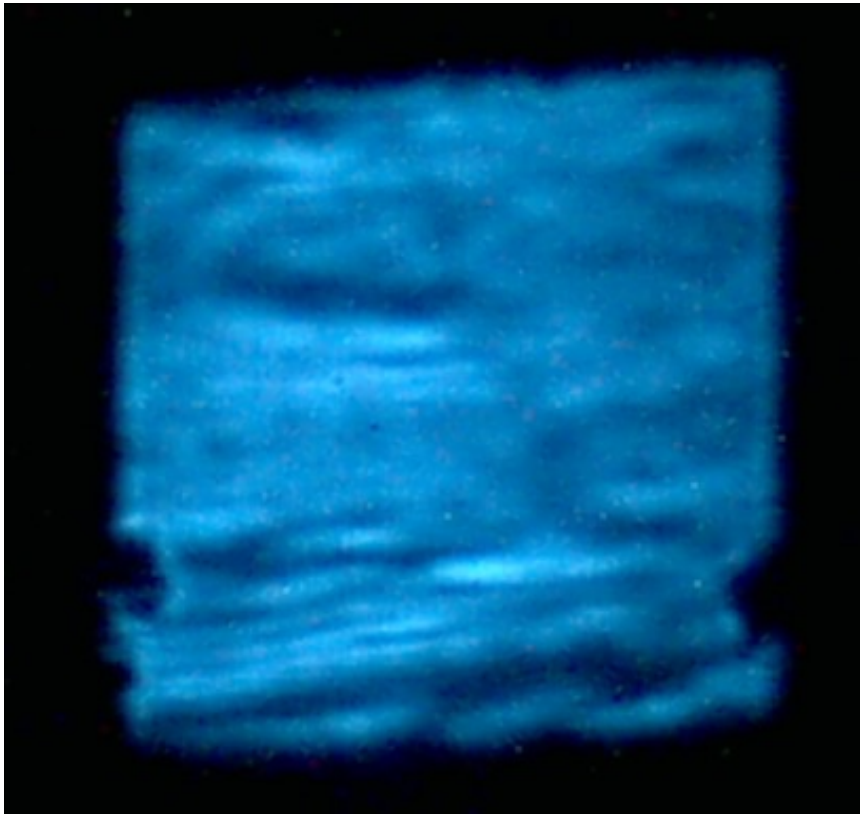

MICRO-CONTROLE
Newport
MICRO-CONTROLE
Newport

BN Coating on Sector 34 Window

protects against corrosion of Window by pink beam

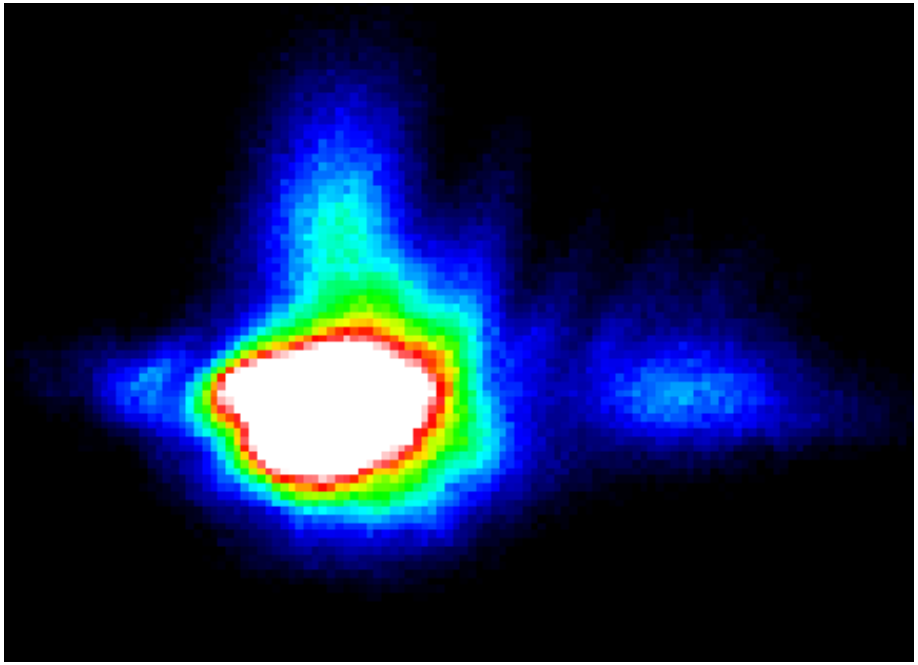


BN spray coating

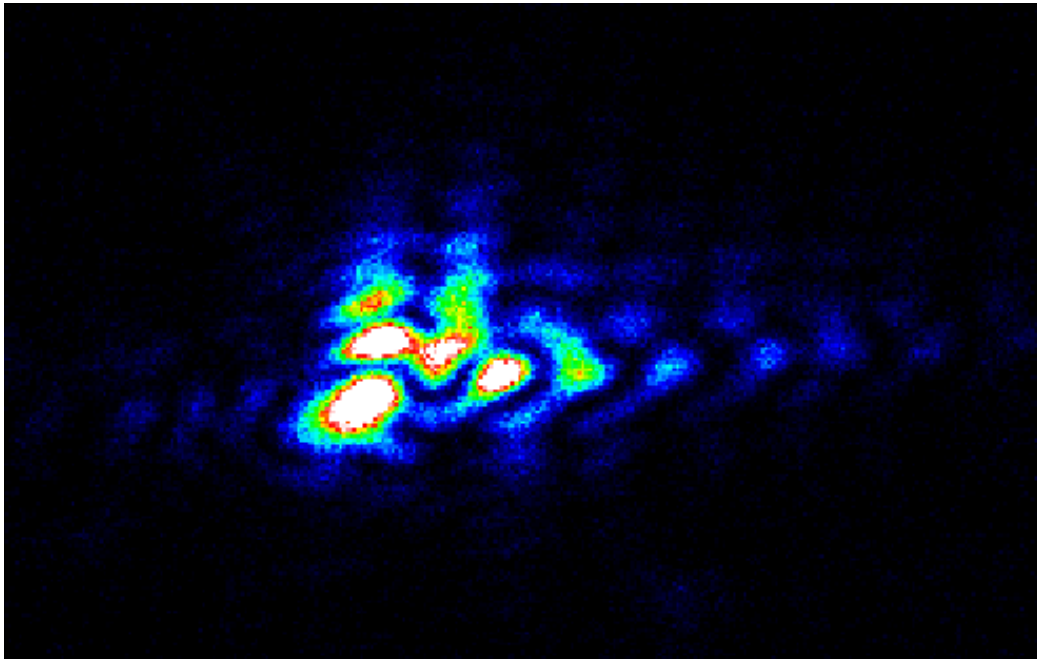


Coating removed

Au Nanocrystal CXD at Sector 34



BN-coated window

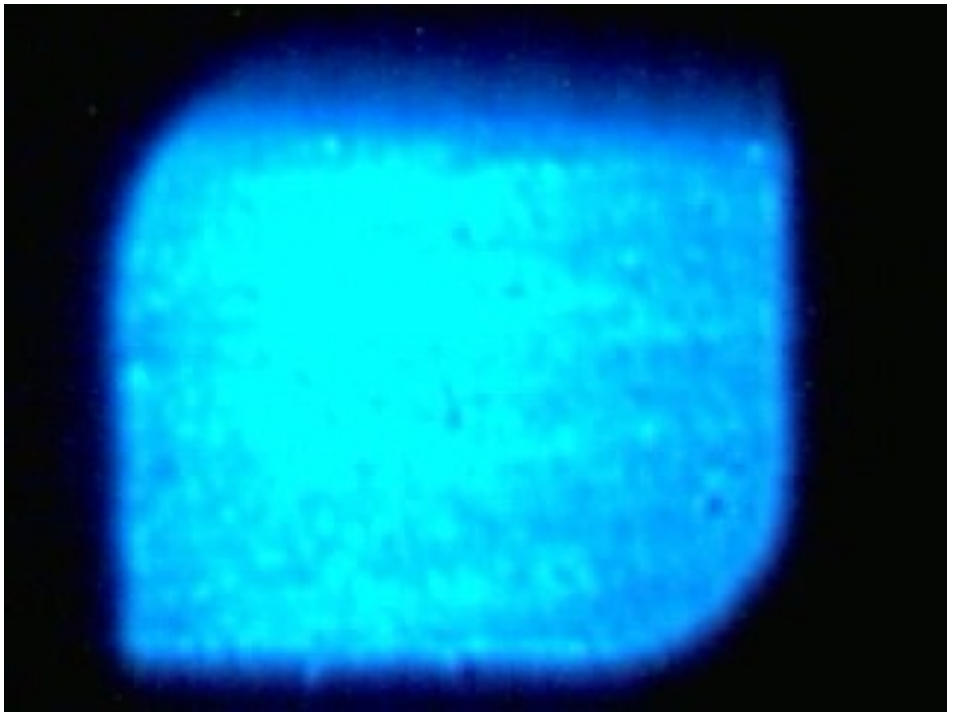


BN removed Sept 2002

Switch to Si(111) monochromator

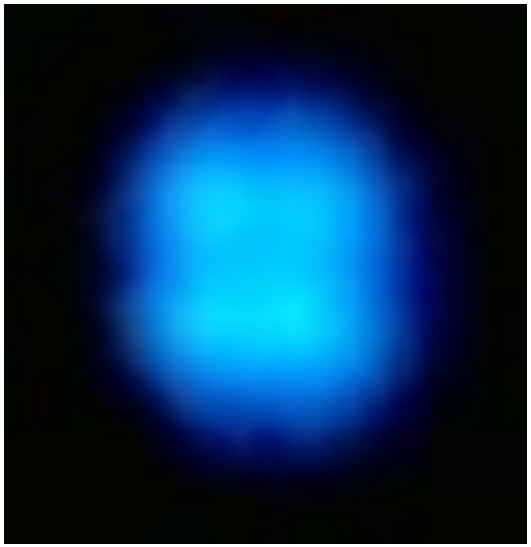
Scan table to watch topography

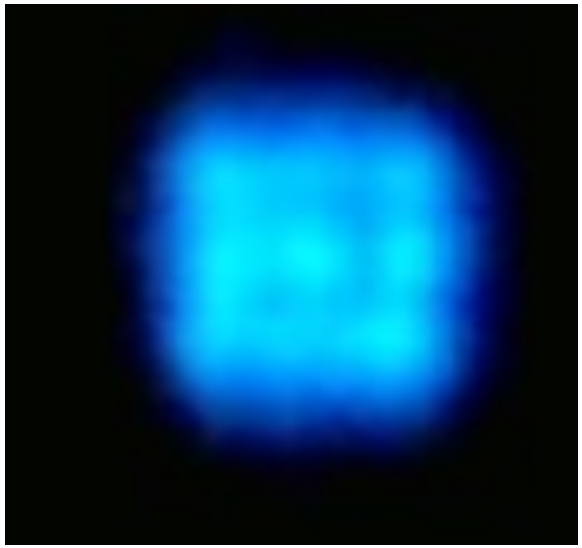
Diamond



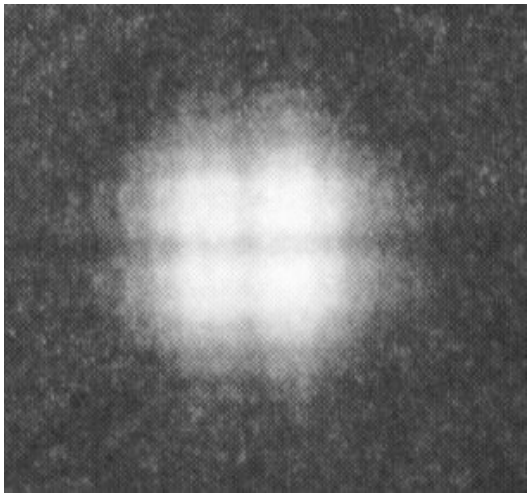
Silicon

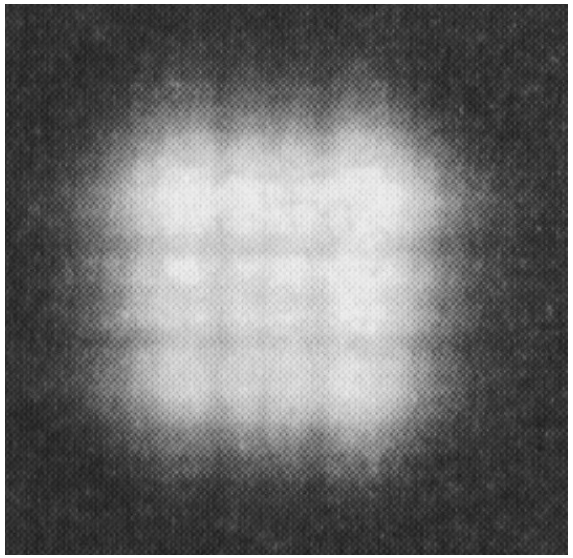
Fresnel Diffraction when $d^2 \sim \lambda D$





X-ray beam defined by RB slits

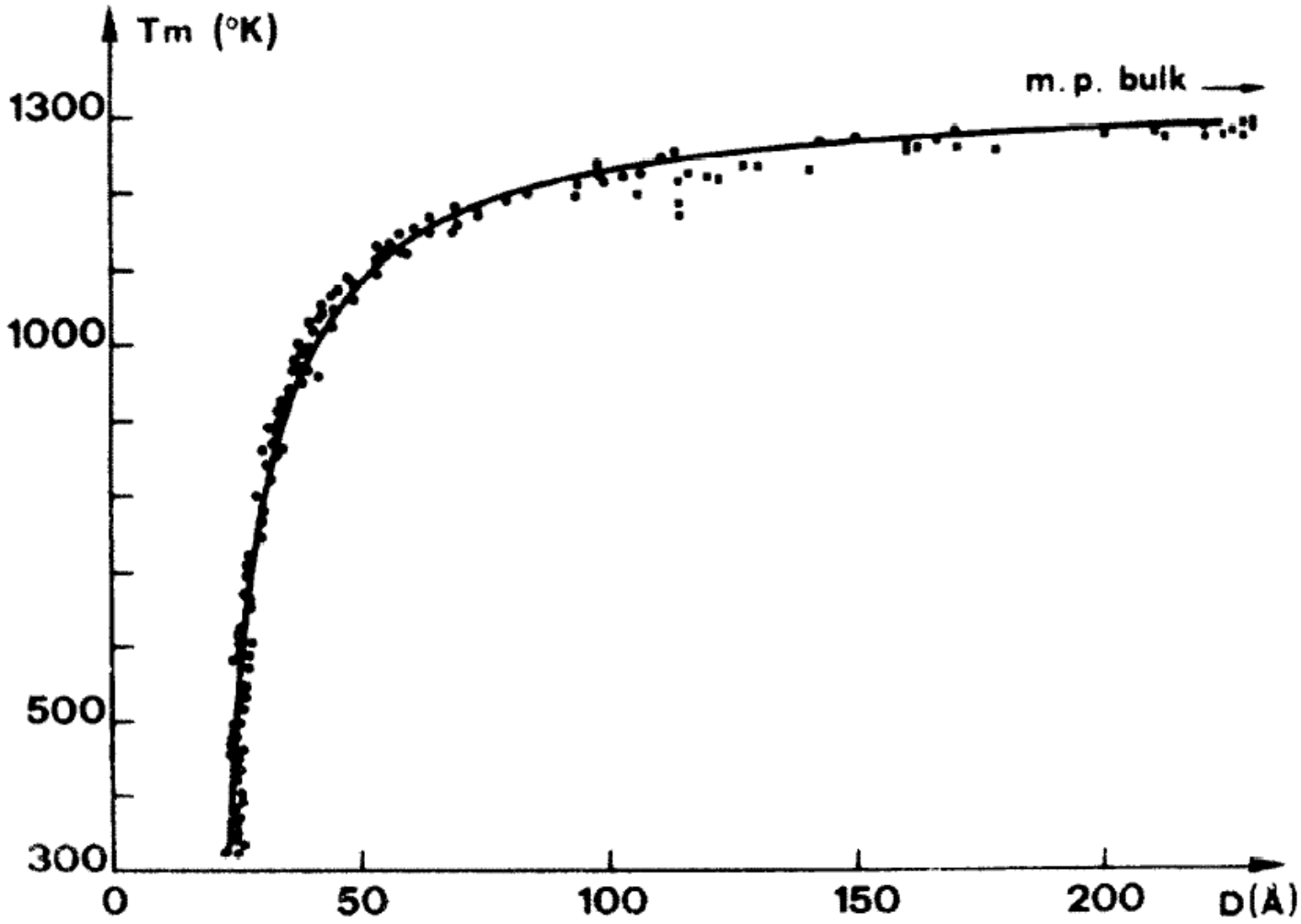




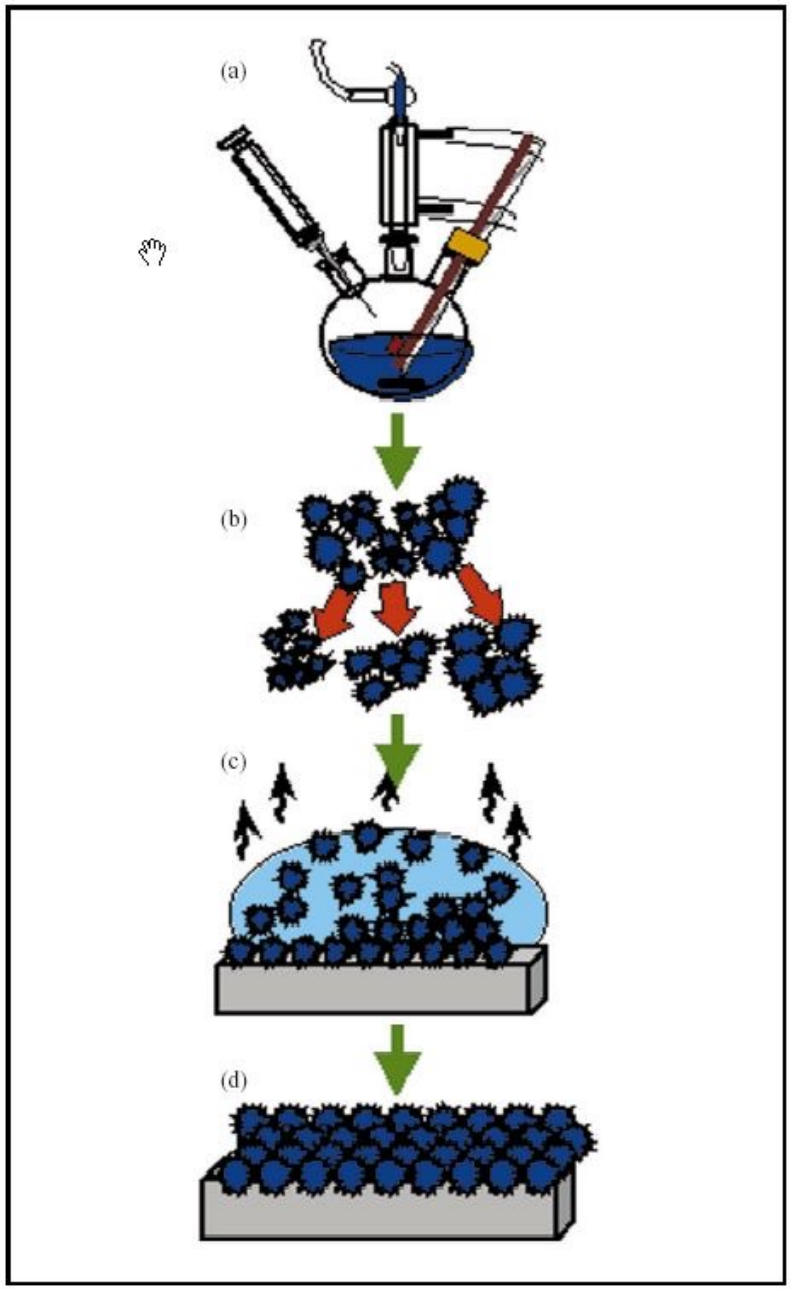
Visible Fresnel diffraction from Hecht "Optics"

Size-dependent Melting of Au Particles

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

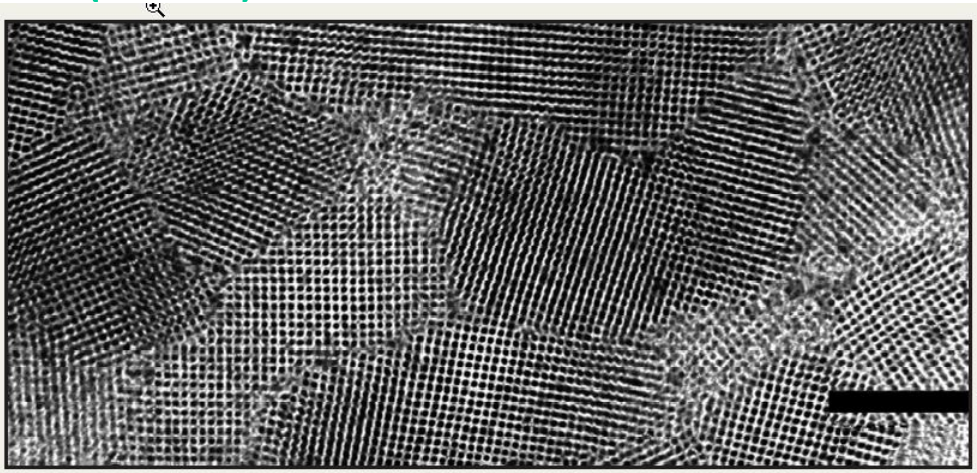


Chemical Synthesis of Nanocrystals



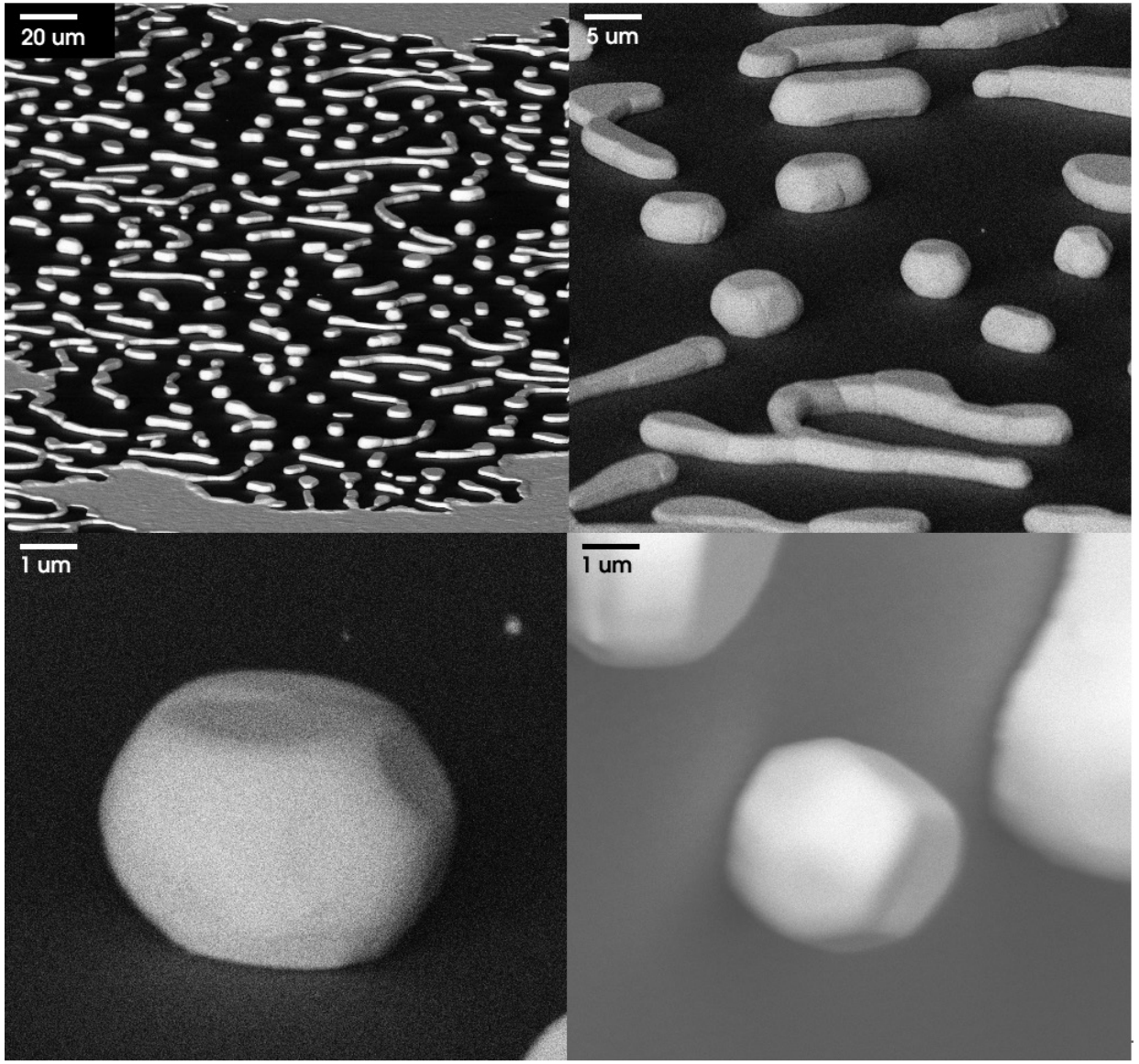
- Reactants introduced rapidly
- High temperature solvent
- Surfactant/organic capping agent
- Square superlattice (200nm scale)

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

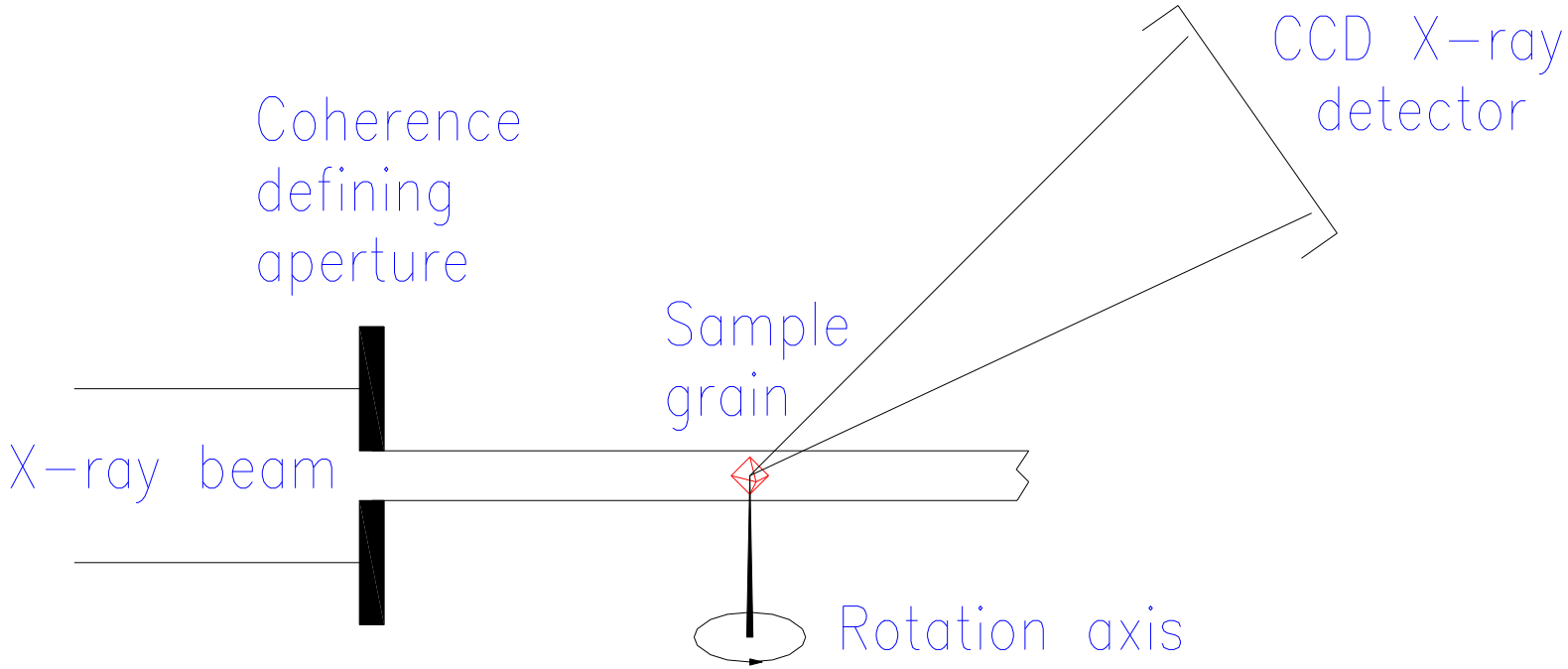


SEMS

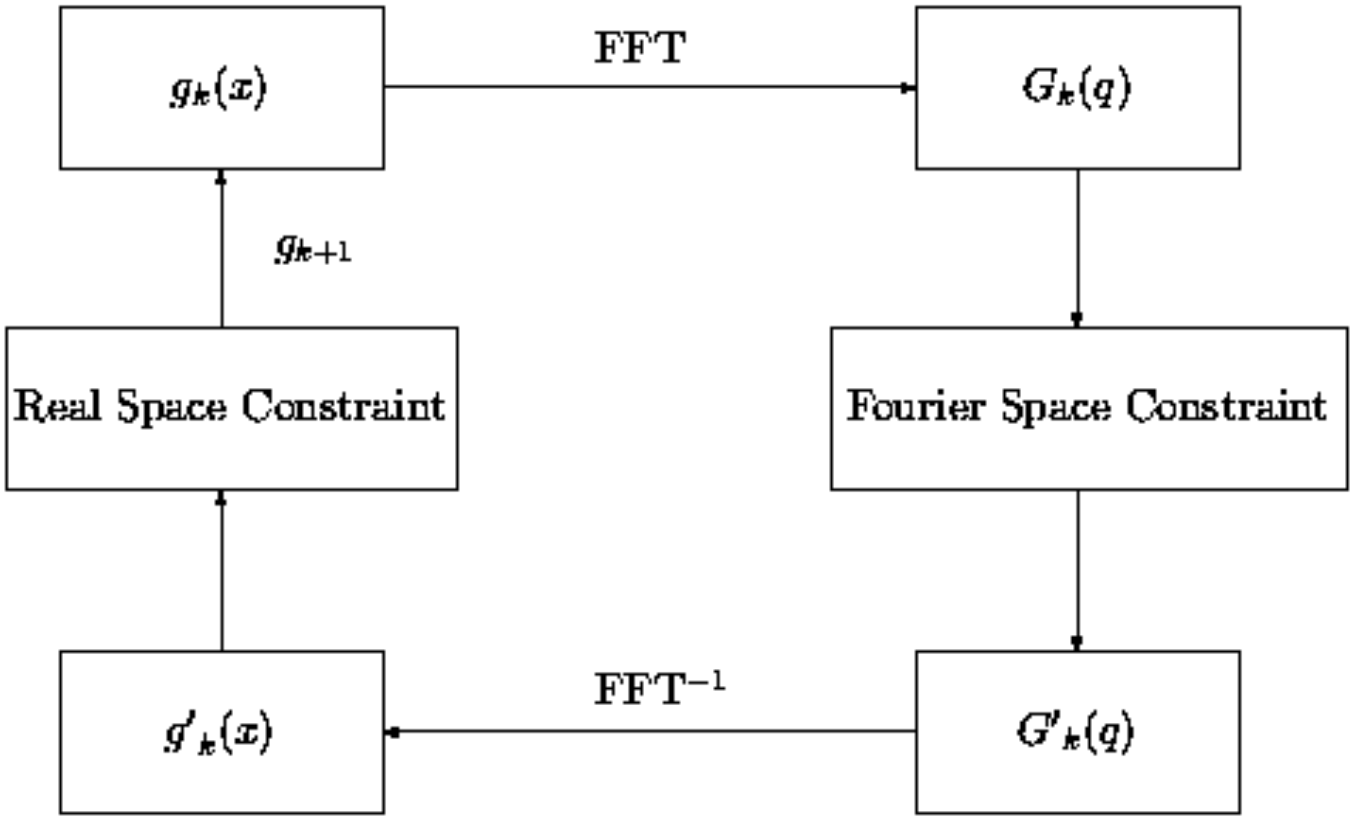
- Au blanket film
- Quartz substrate
- Annealed at 950°C for 70 hrs.



Lensless X-ray Microscope



Generic "Error Reduction" method



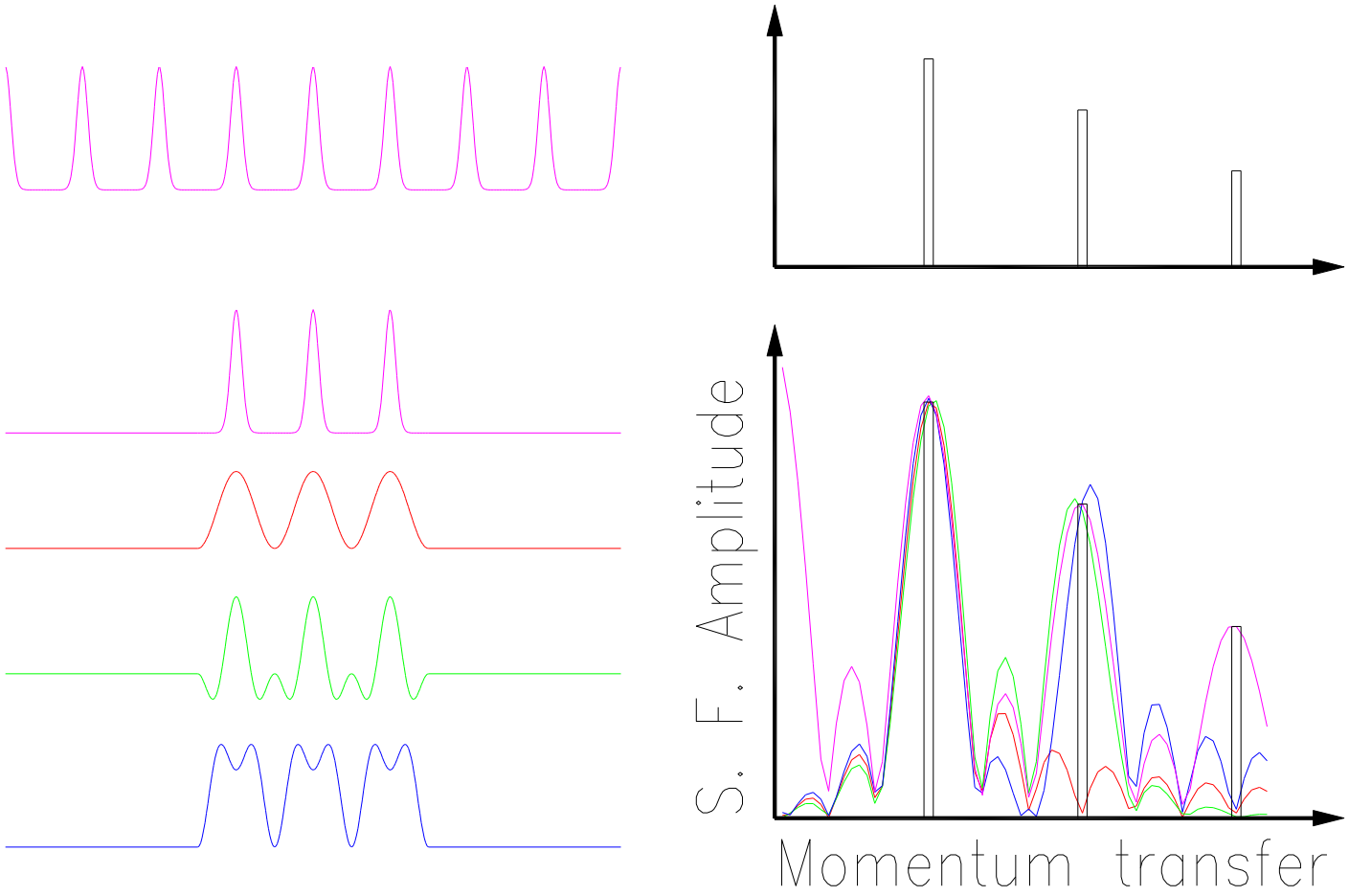
J. R. Fienup Appl. Opt. 21 2758 (1982)
R. W. Gerchberg and W. O. Saxton Optik 35 237 (1972)

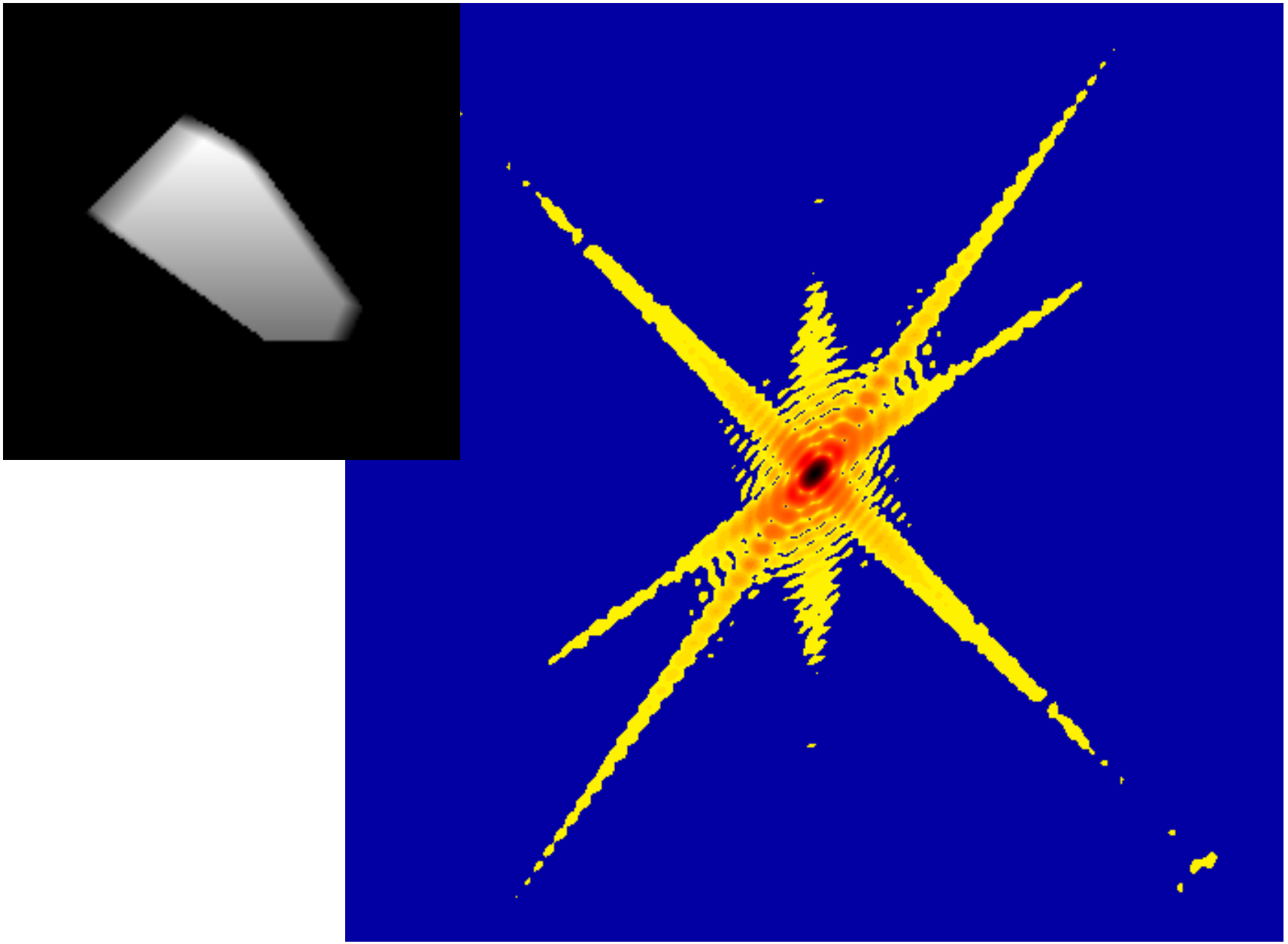
Real-space Constraints in Crystallograhy

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)

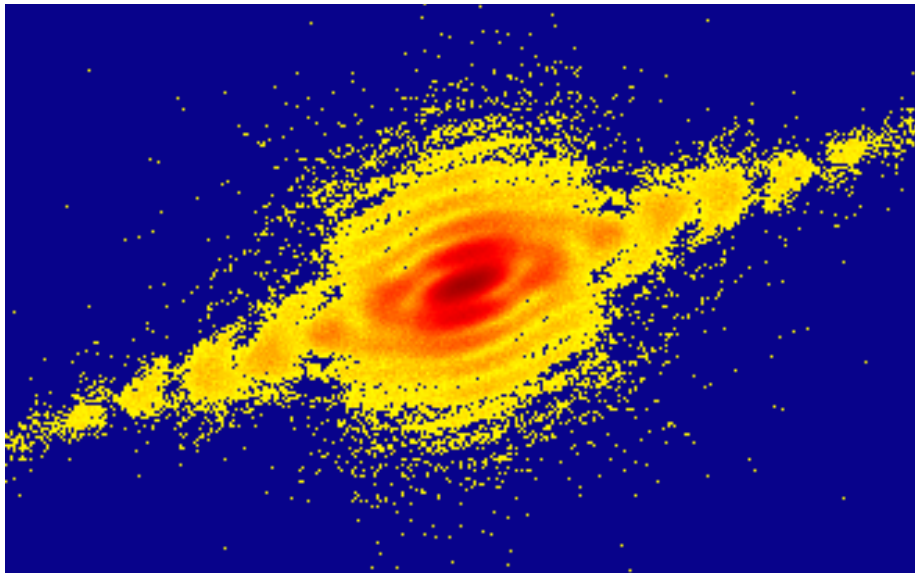
Phase Problem: Finite-size Effect

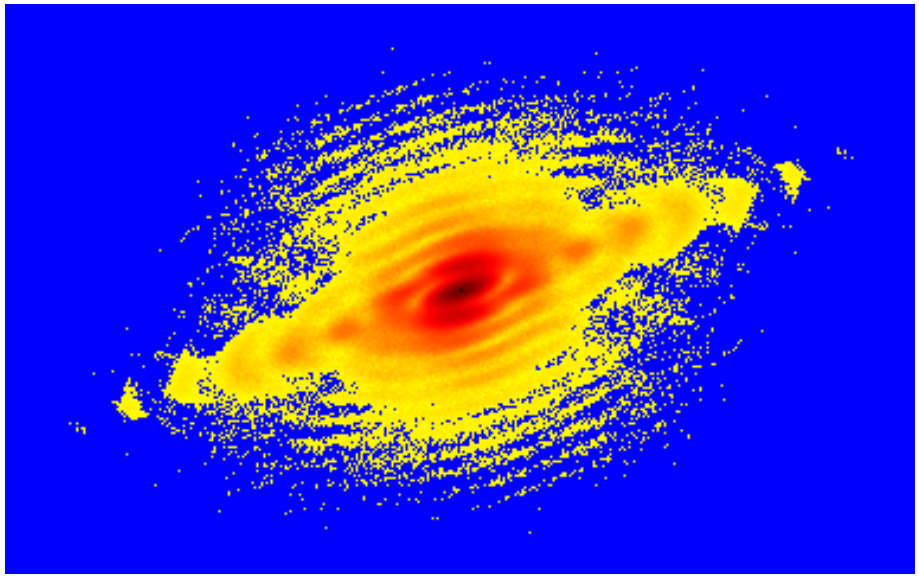


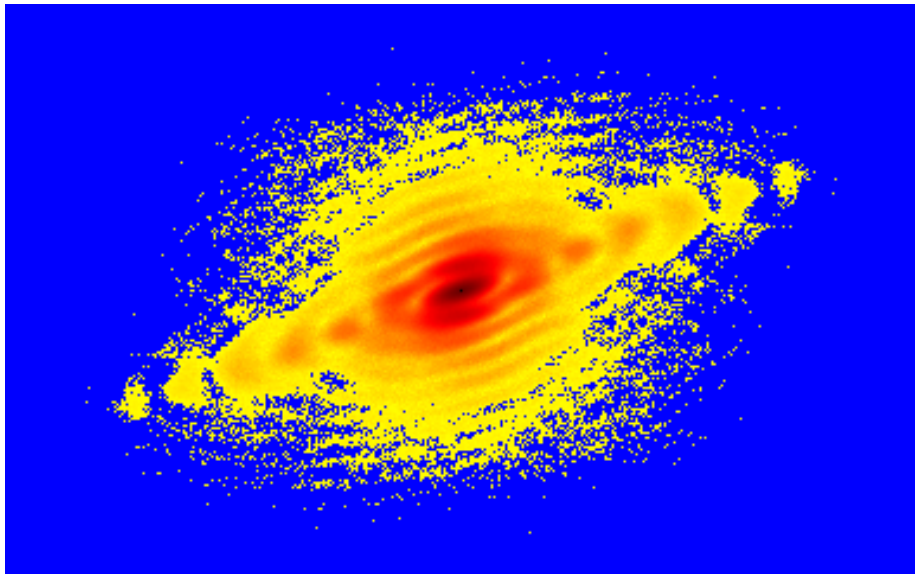


Symmetrized Data and two best fits

Chisq=0.0005

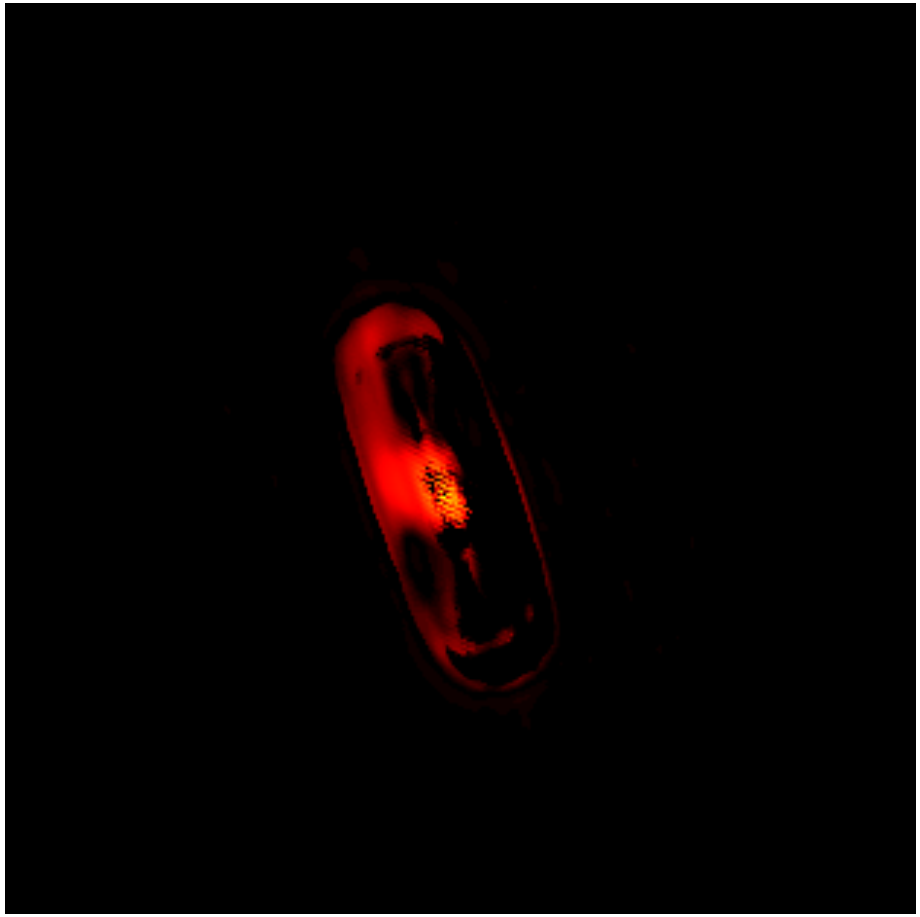


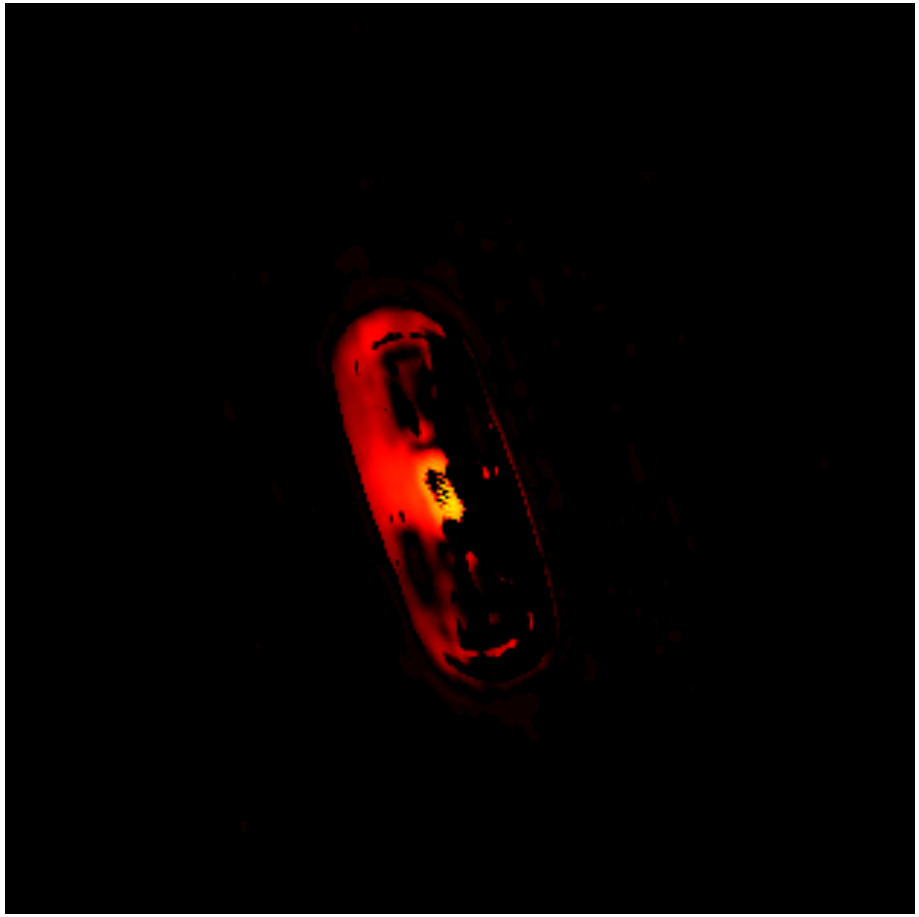




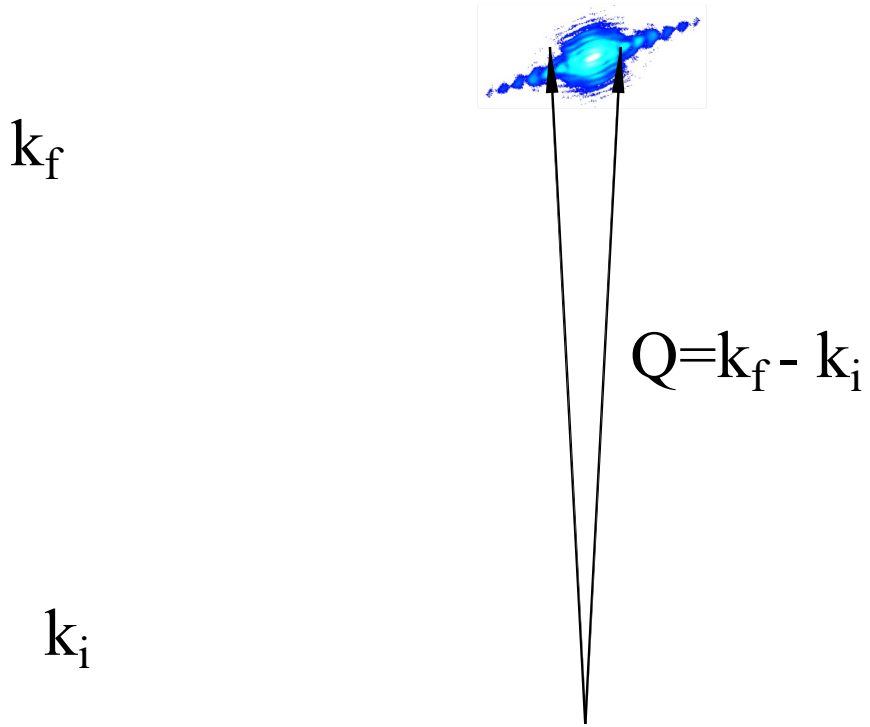
2D Reconstructions

chisquare = 0.0005

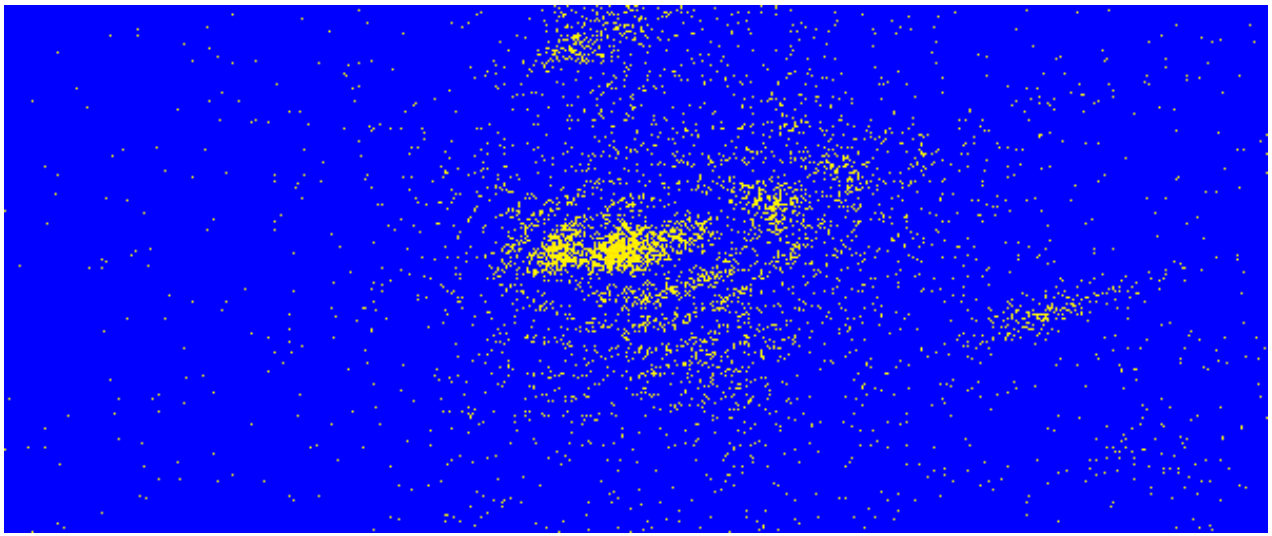


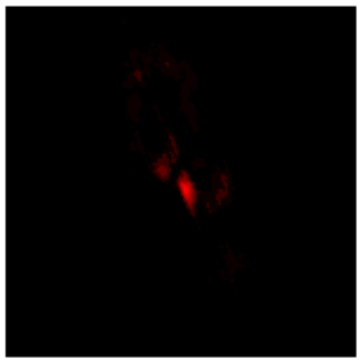
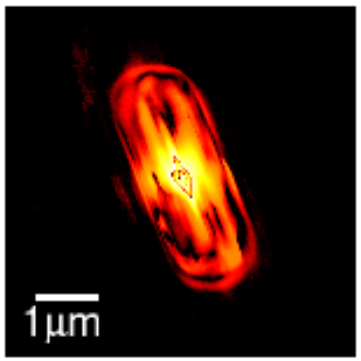


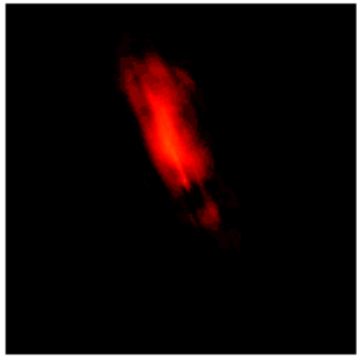
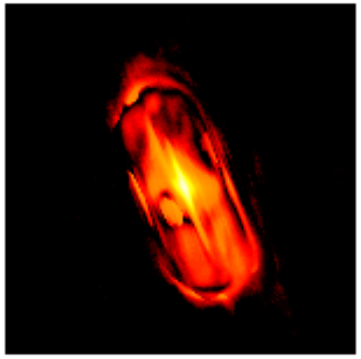
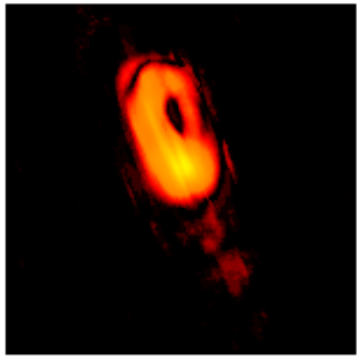
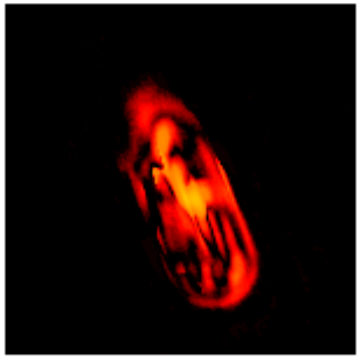
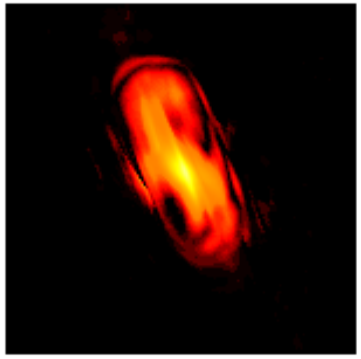
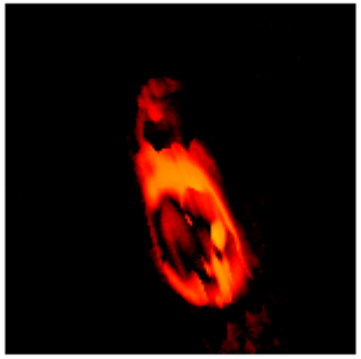
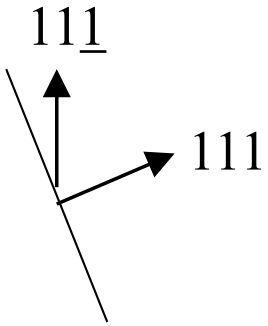
3D Diffraction Method

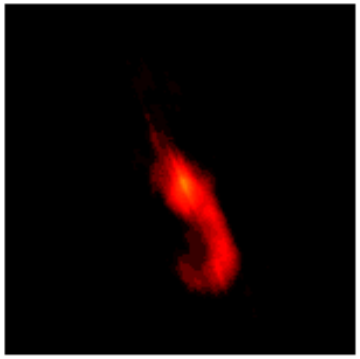
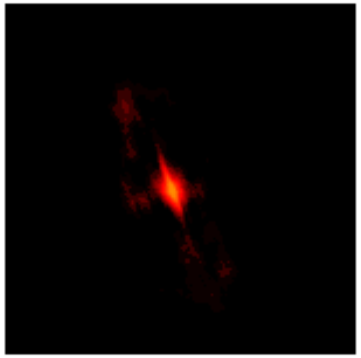


3D Diffraction Data
1 micron Au crystal

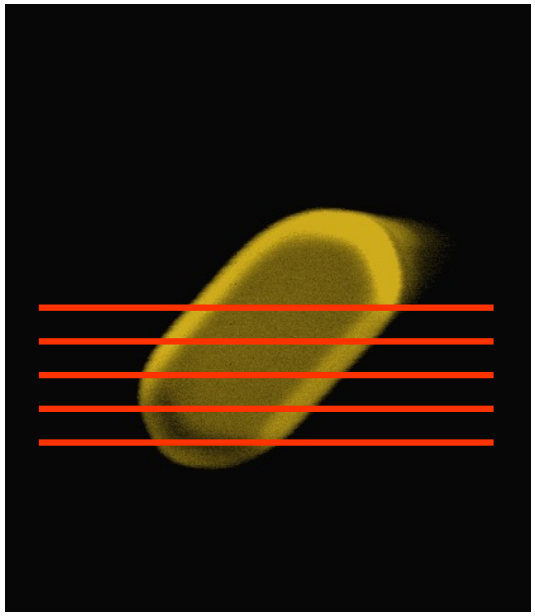


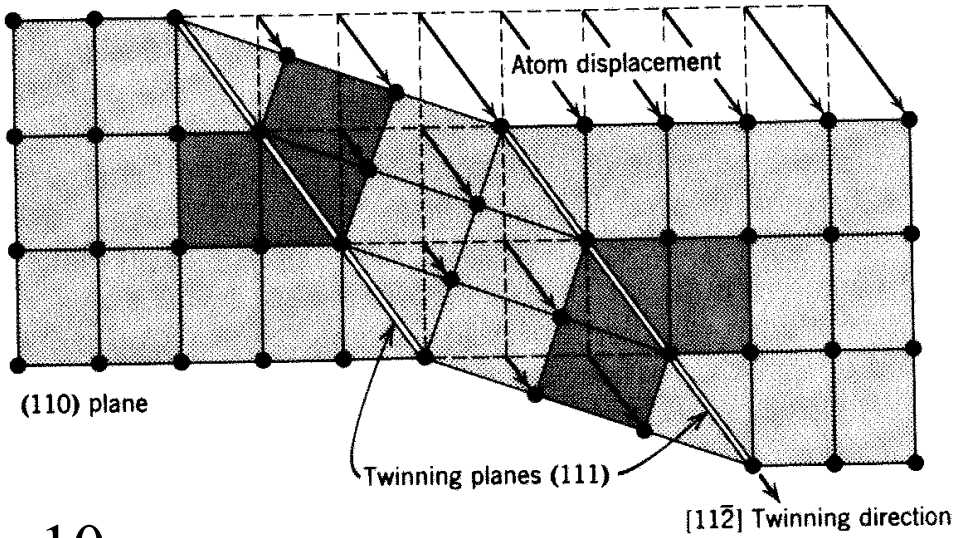



Slices through plan view SEM:



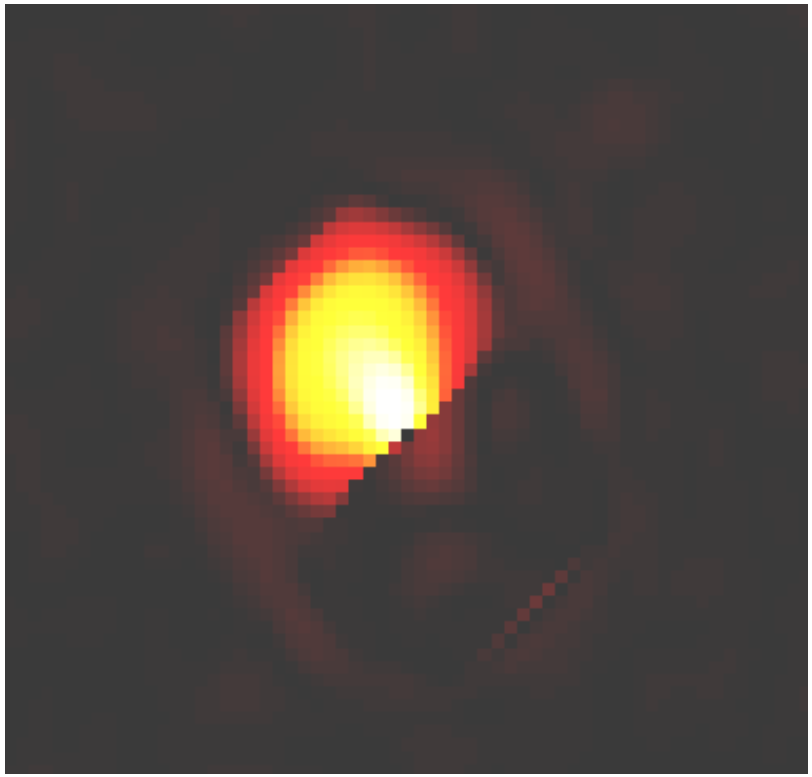
Twinning in deformed FCC metals

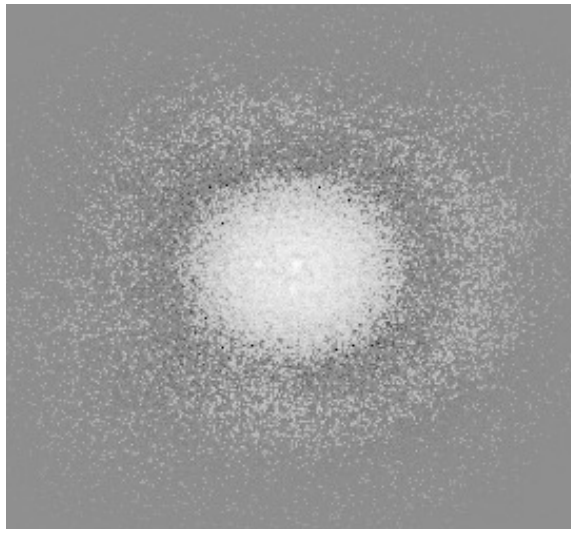
J. Wulff, "Structure and Property of Materials III" (1965)

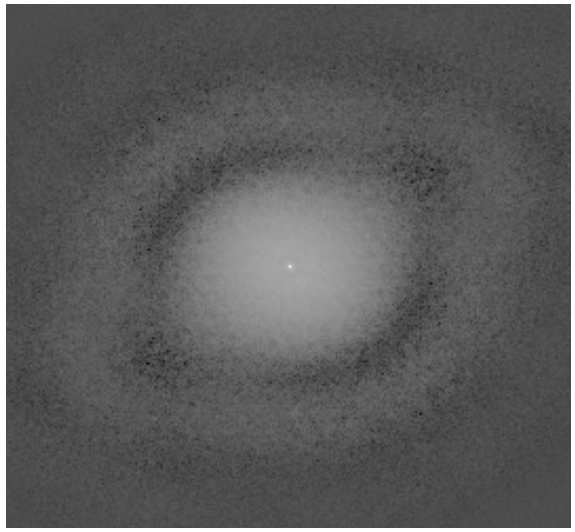


~10μm
Cu

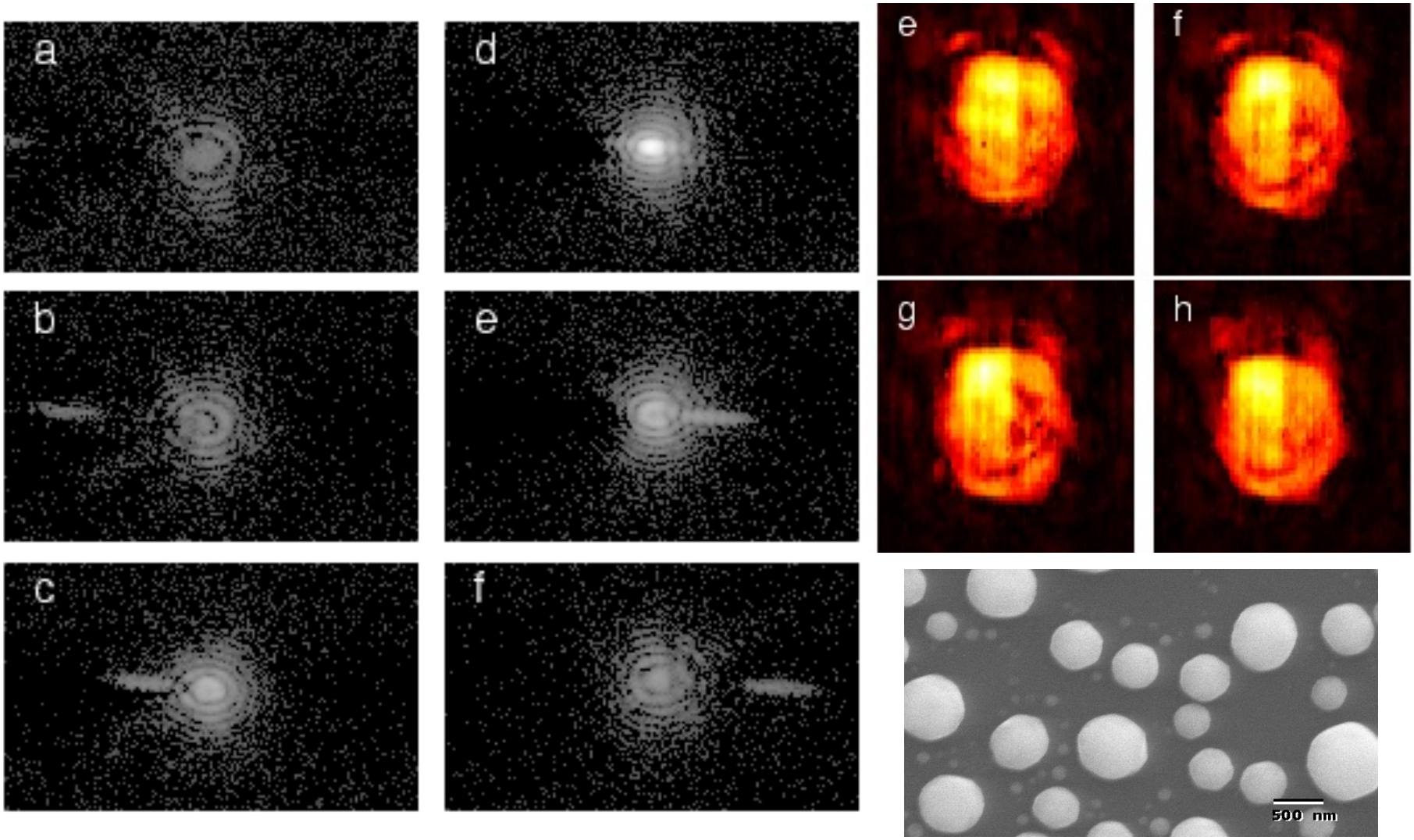
Reconstruction of Pb Nanocrystal



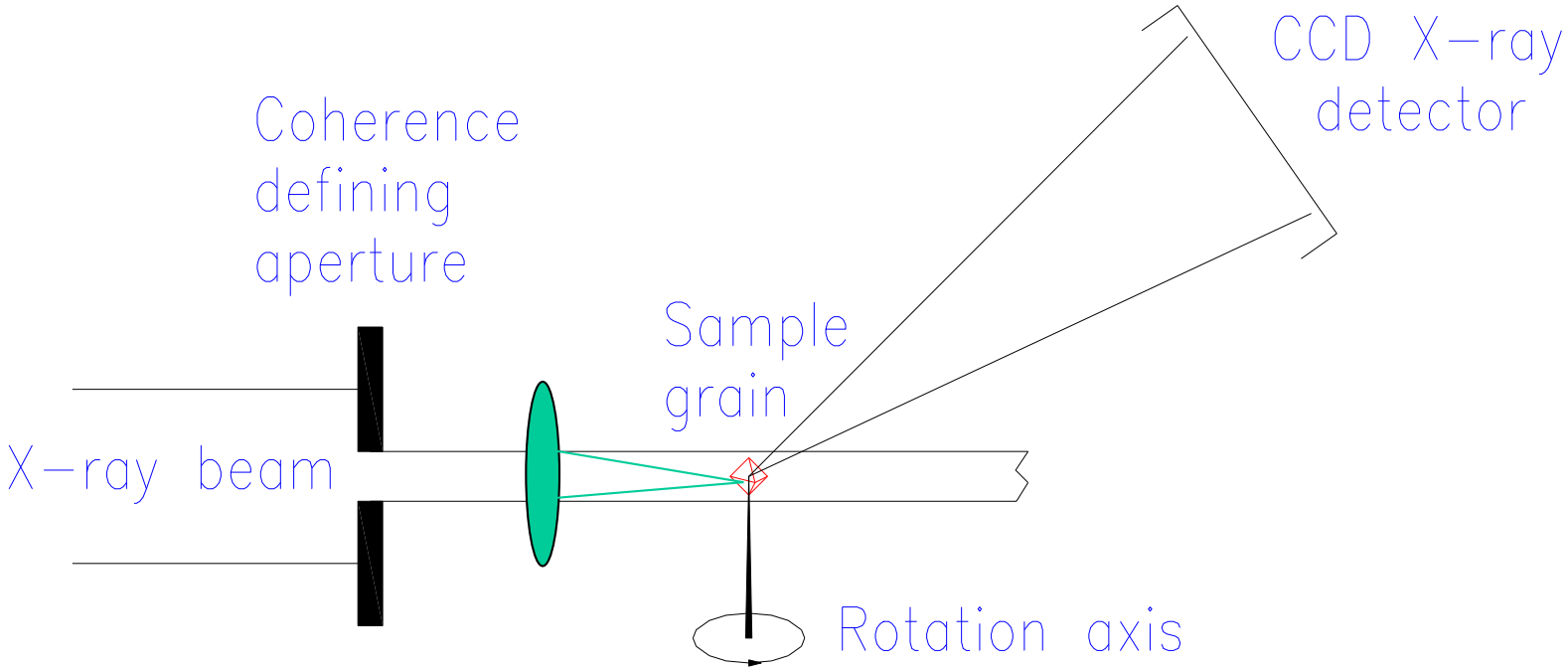




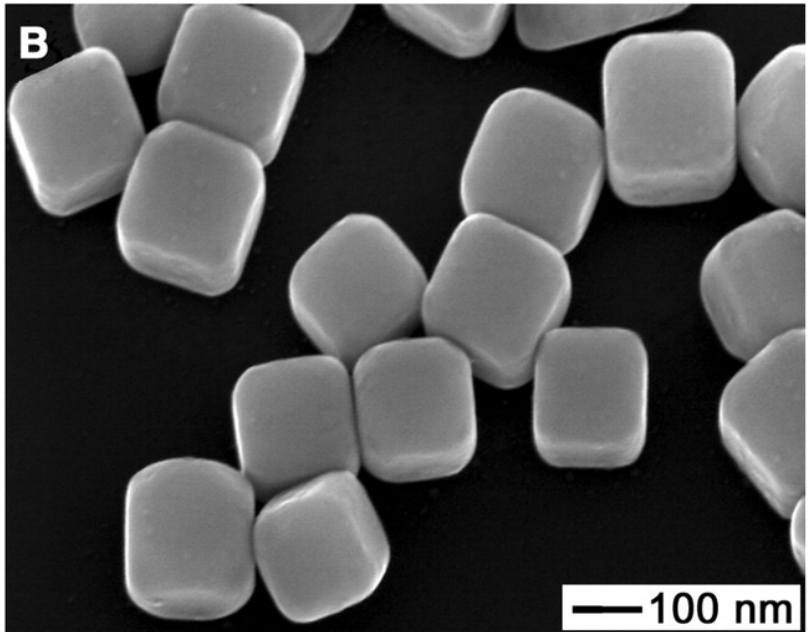
3D reconstruction of Pb nanocrystals

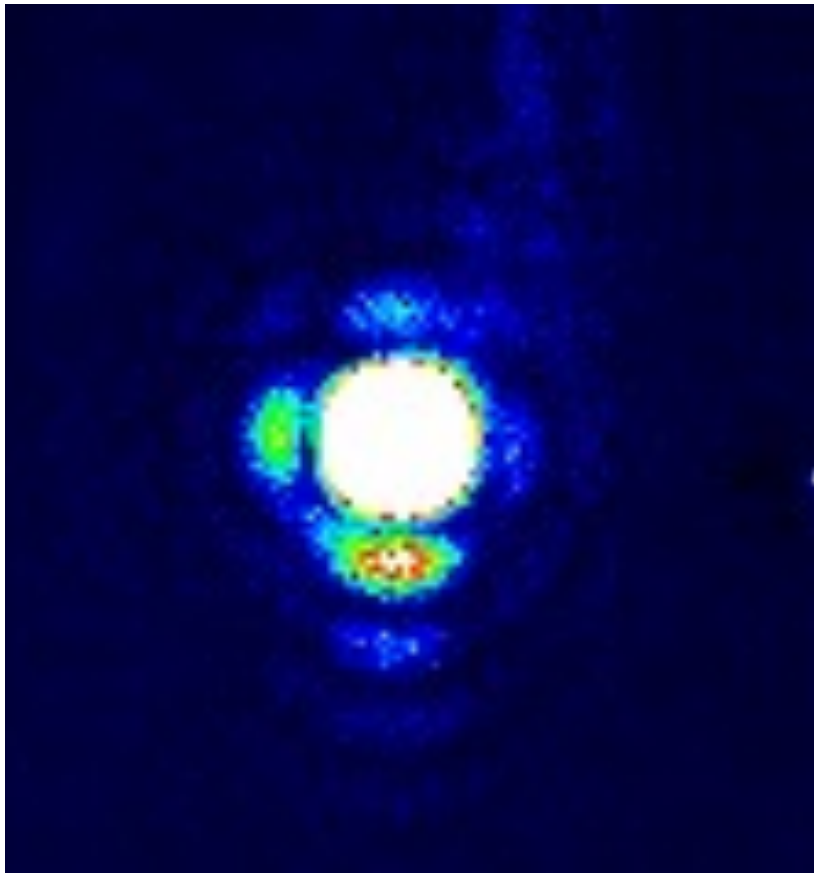


Lensless X-ray Microscope



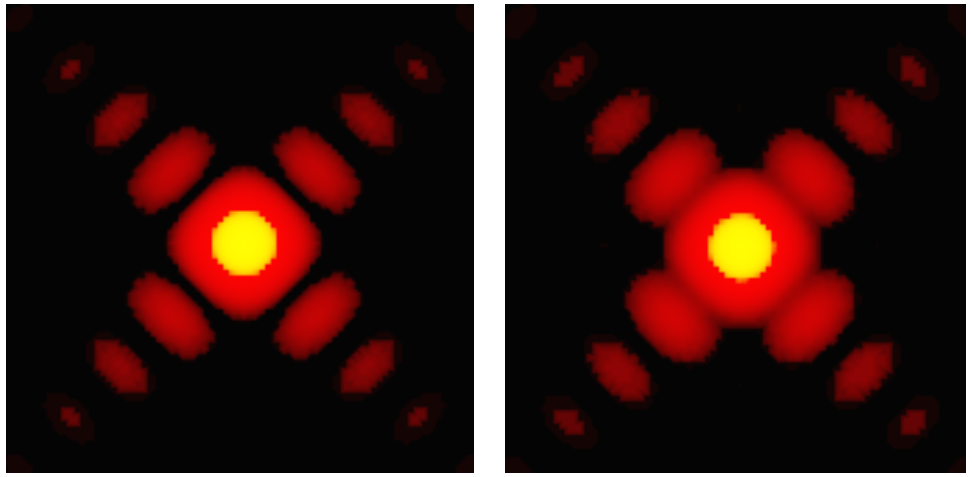
Chemically Synthesized Silver Nanocubes



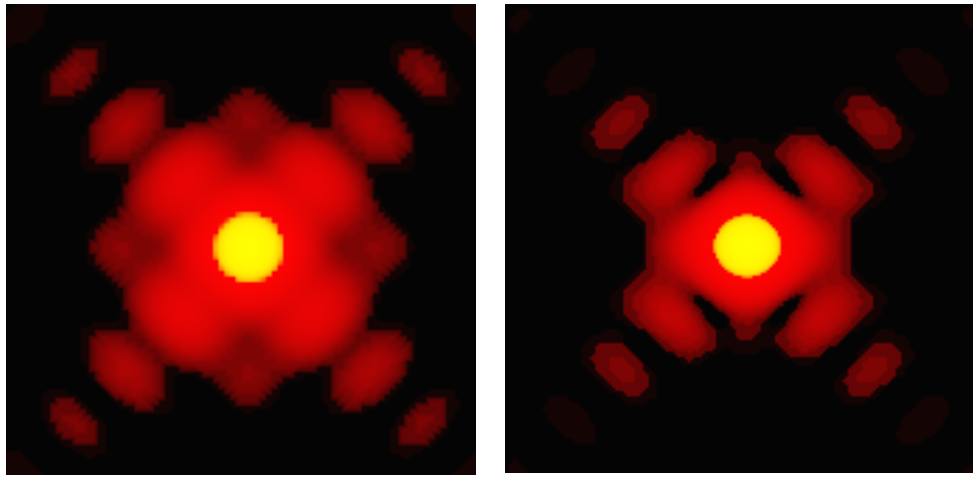


Yugang Sun and Younan Xia,
Science 298 2177 (2003)

Spherical and cylindrical waves



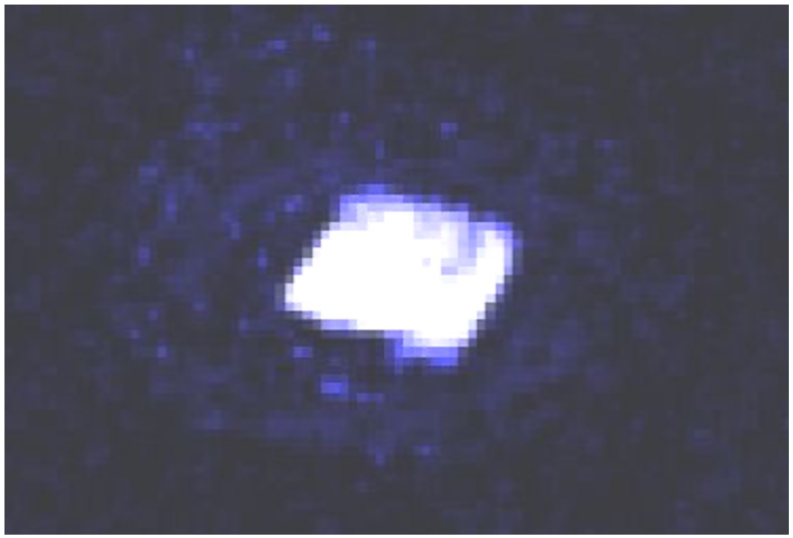
π/4



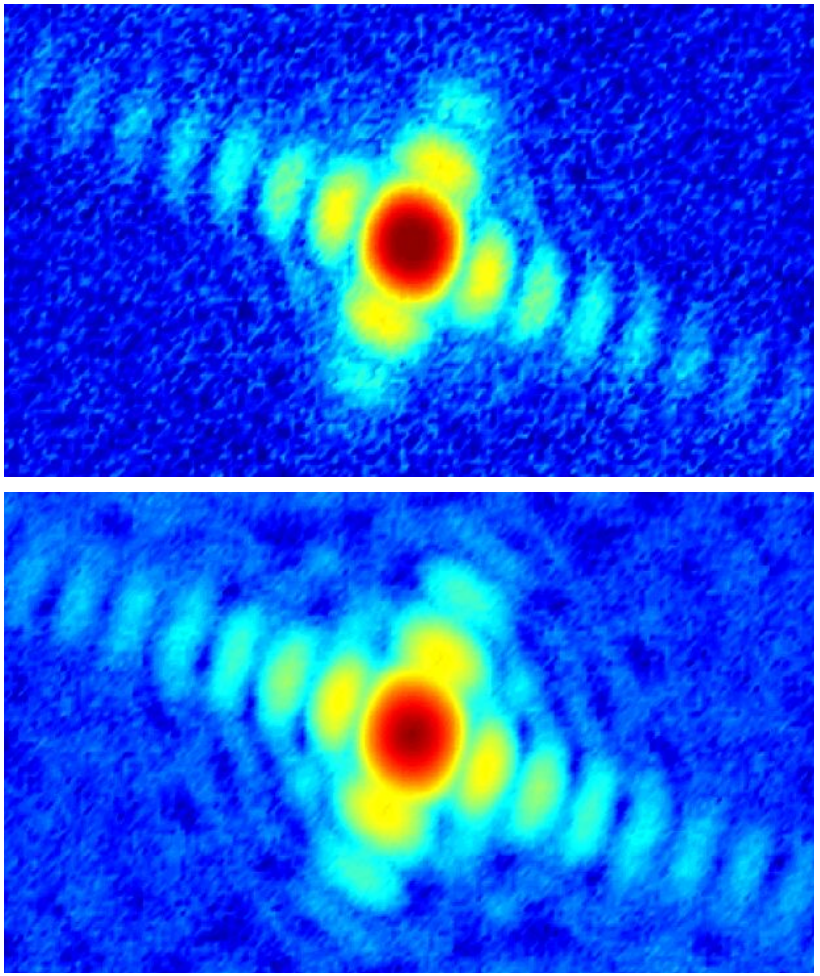
π/2

π/4 (x)

Reconstruction of Ag Nanocrystal

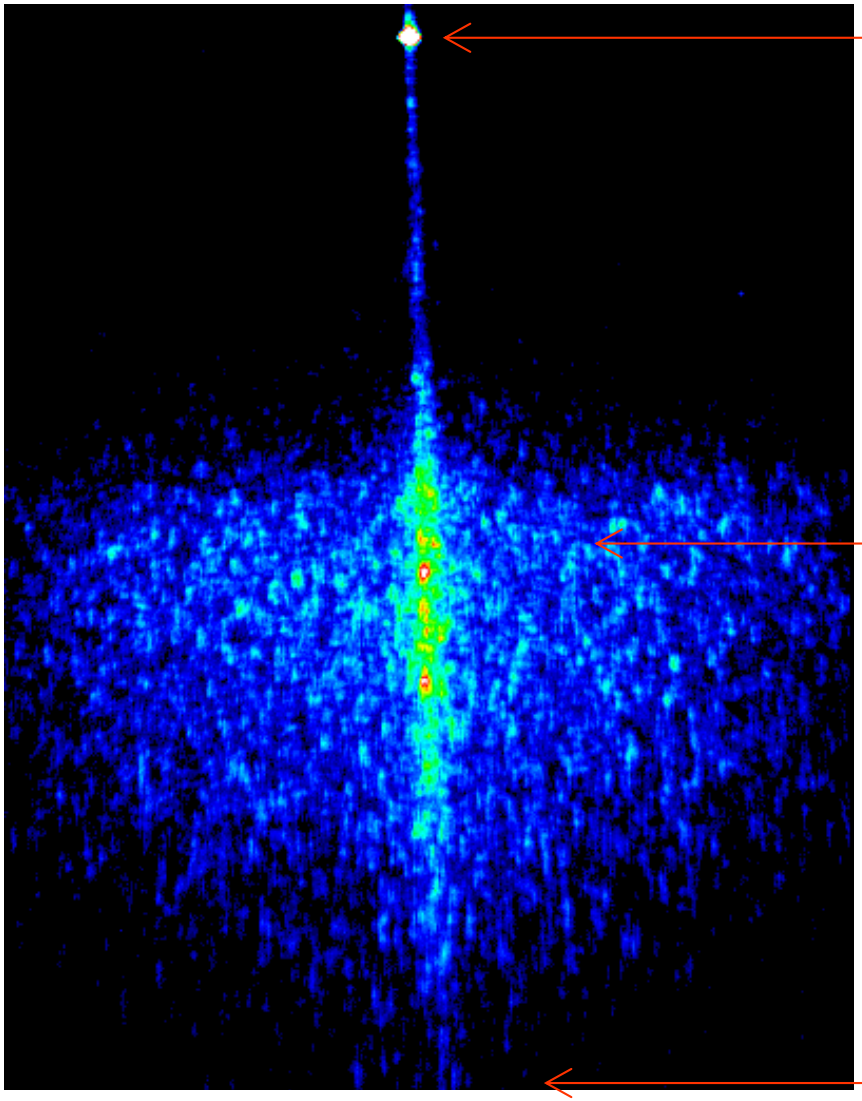


200nm



Coherent Structure in "Yoneda" Peak

Grazing-exit SAXS from a 1000A Au polycrystalline film



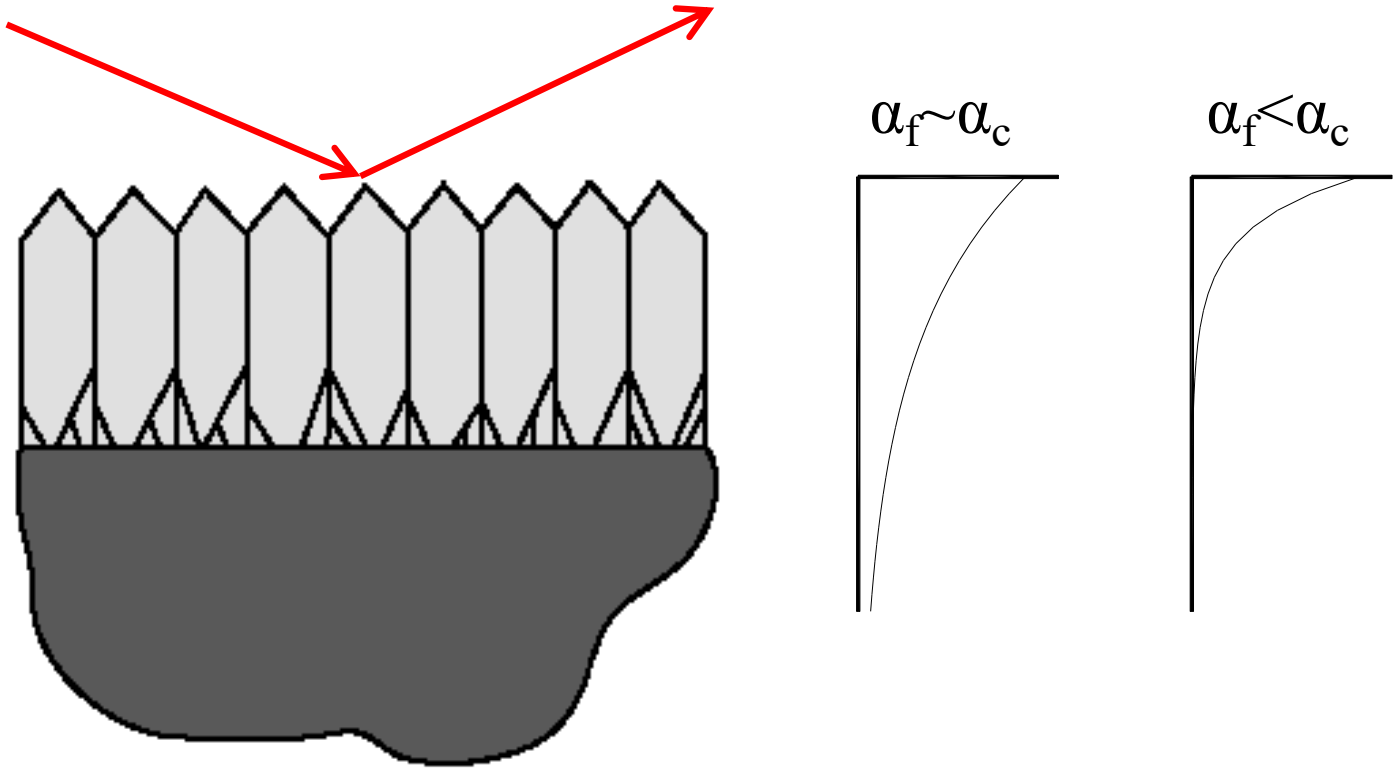
Specular ($\alpha_f \sim \alpha_i$)

$\alpha_f \sim \alpha_c$

$\alpha_f \sim 0$

Competitive Grain Growth

C. V. Thompson, Ann. Rev. Mat. Sci. **30** 159 (2000)

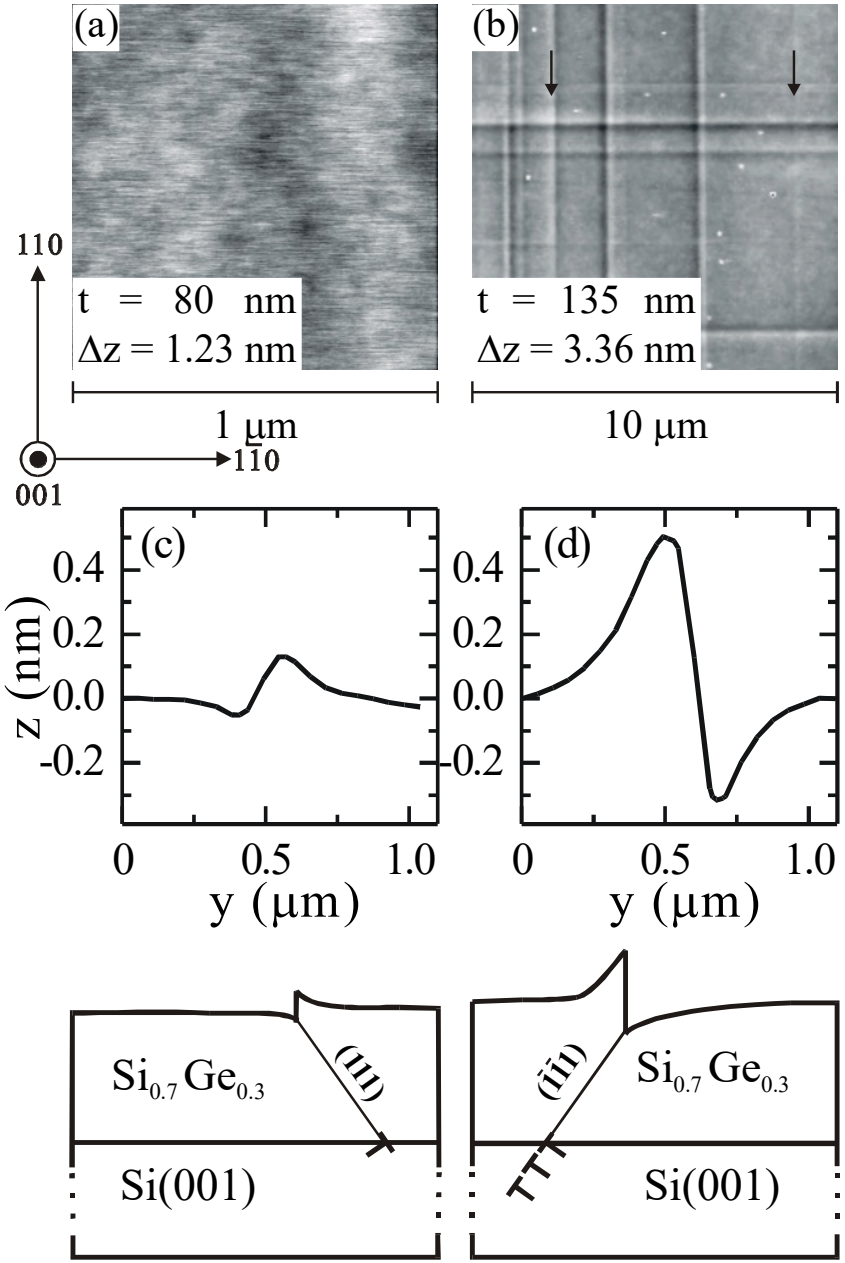


Low dislocation density GeSi films

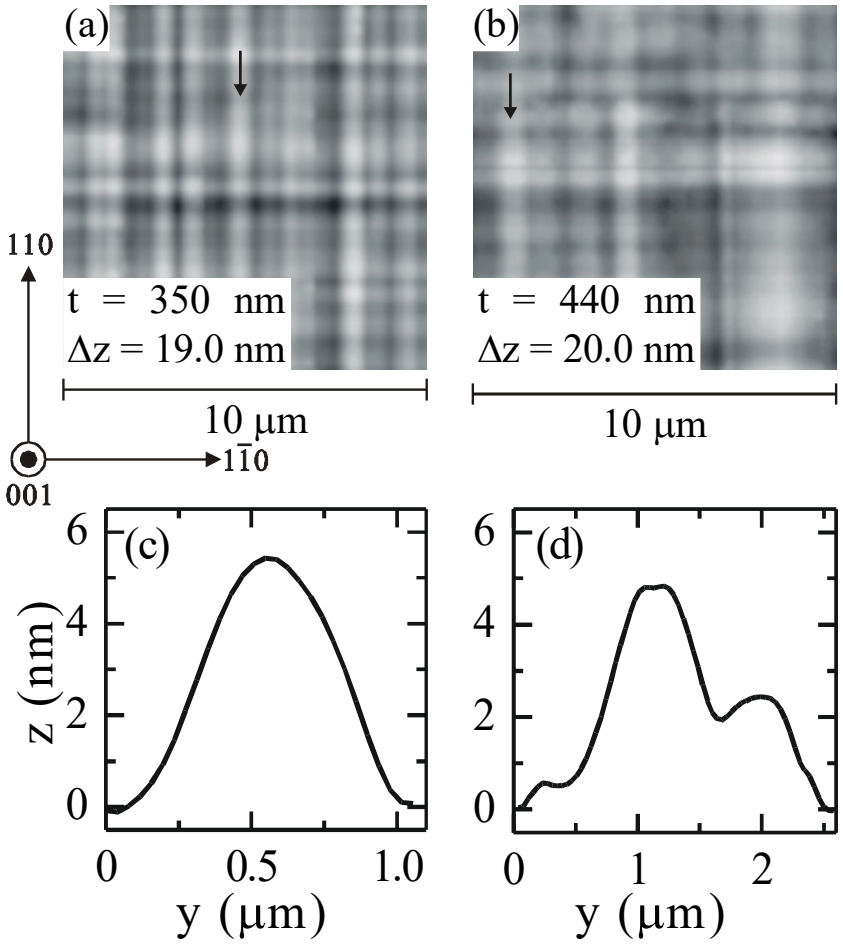
Thickness close to critical thickness

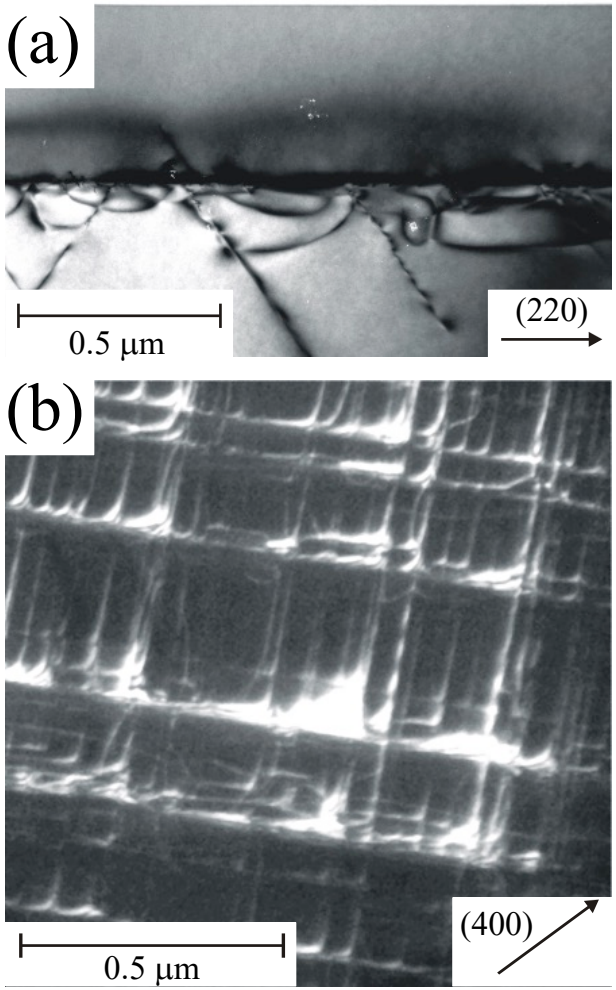
Dislocations aggregate at interface and glide to surface along {111}

T. Spila, UIUC Thesis



Higher Dislocation Density $Ge_{0.3}Si_{0.7}$

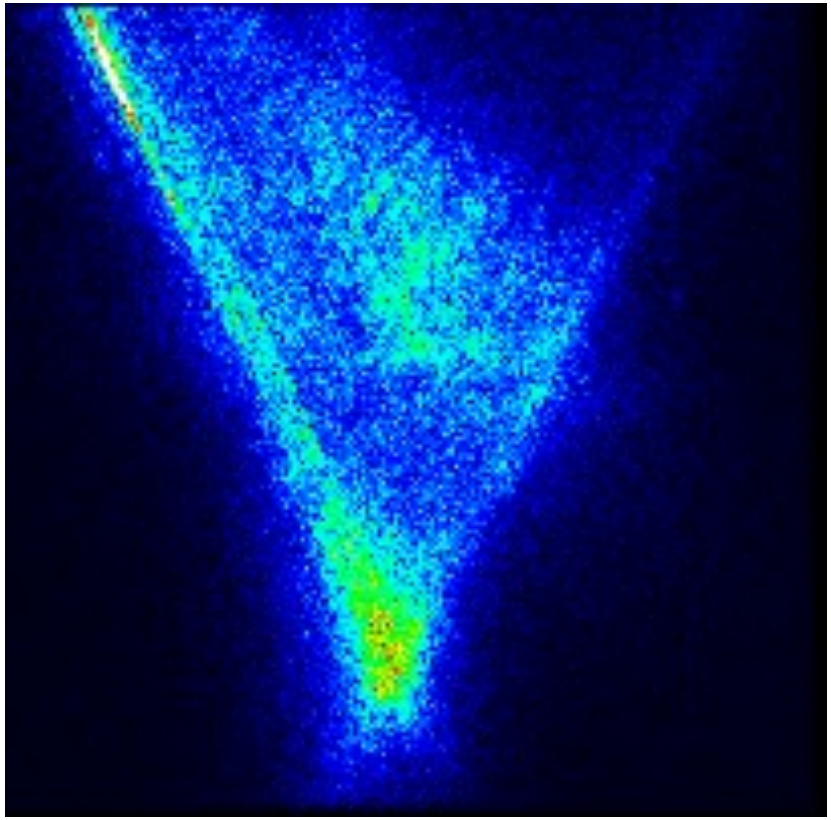




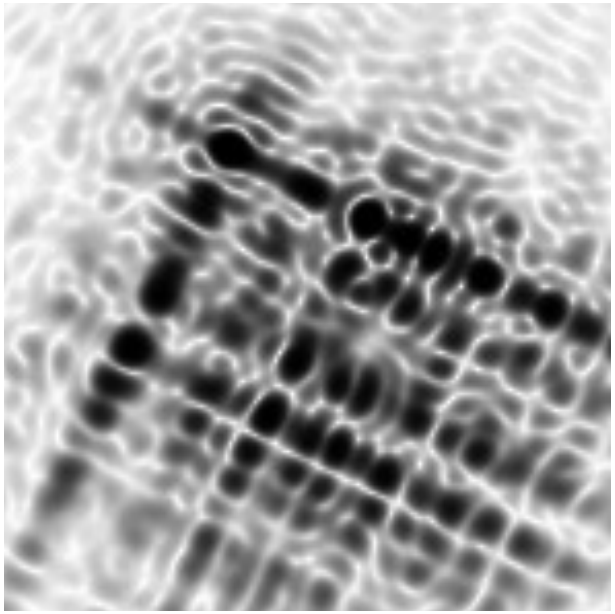
t=440nm

Use CCD and scan sample position

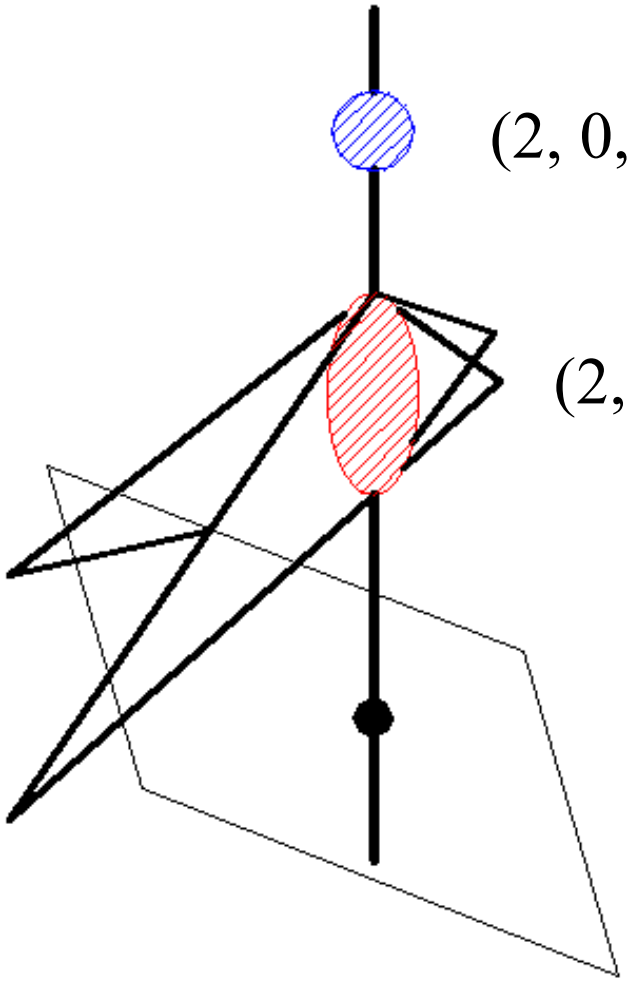
(2,0,1.92) 280nm thick $Ge_{0.3}Si_{0.7}$



Dislocation Diffraction



Dislocations in Reciprocal Space



Conclusions and Outlook

- Inversion of CXD demonstrated
- Internal structure of Au Nanocrystals
- Phasing by computation instead of lens
- Preservation of coherence upon focussing
- Dislocations give asymmetric patterns
- Single molecules one day