Limits of Coherent X-ray Diffraction for Imaging Small Crystals

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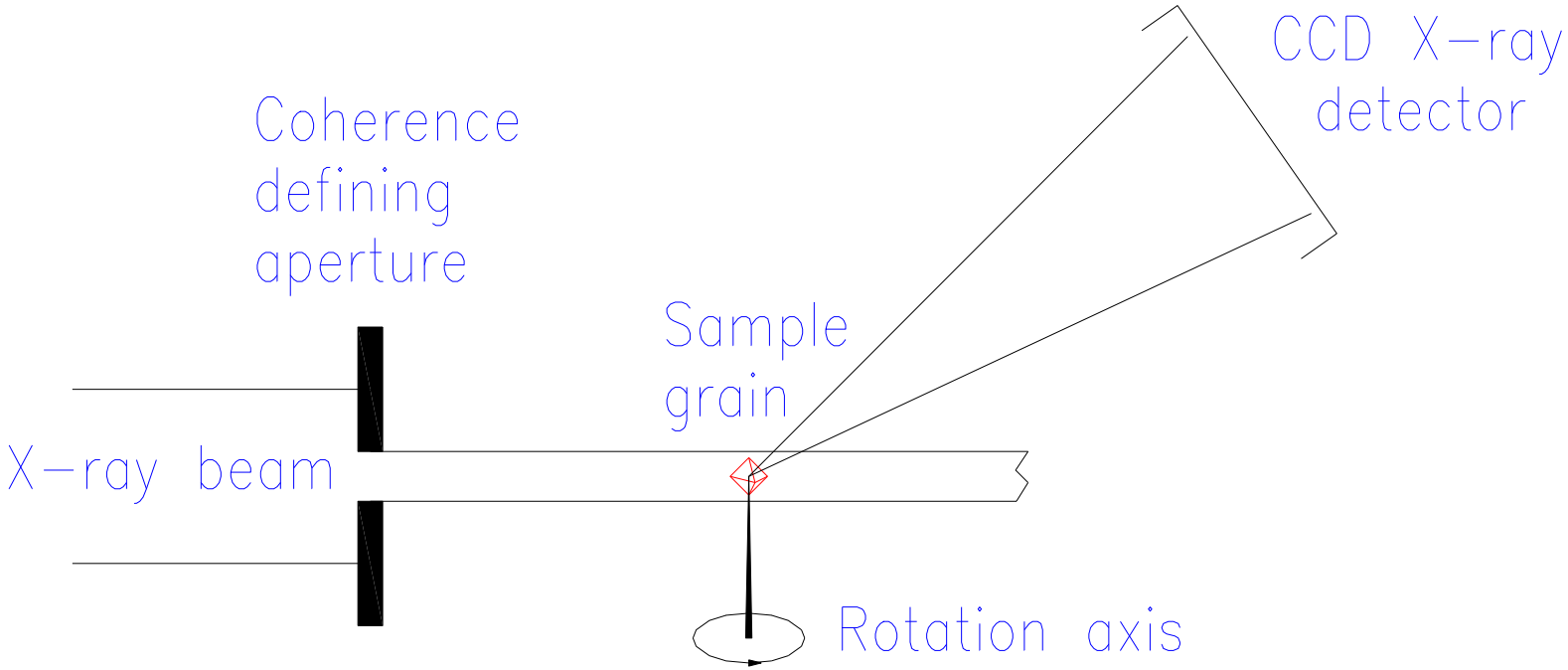
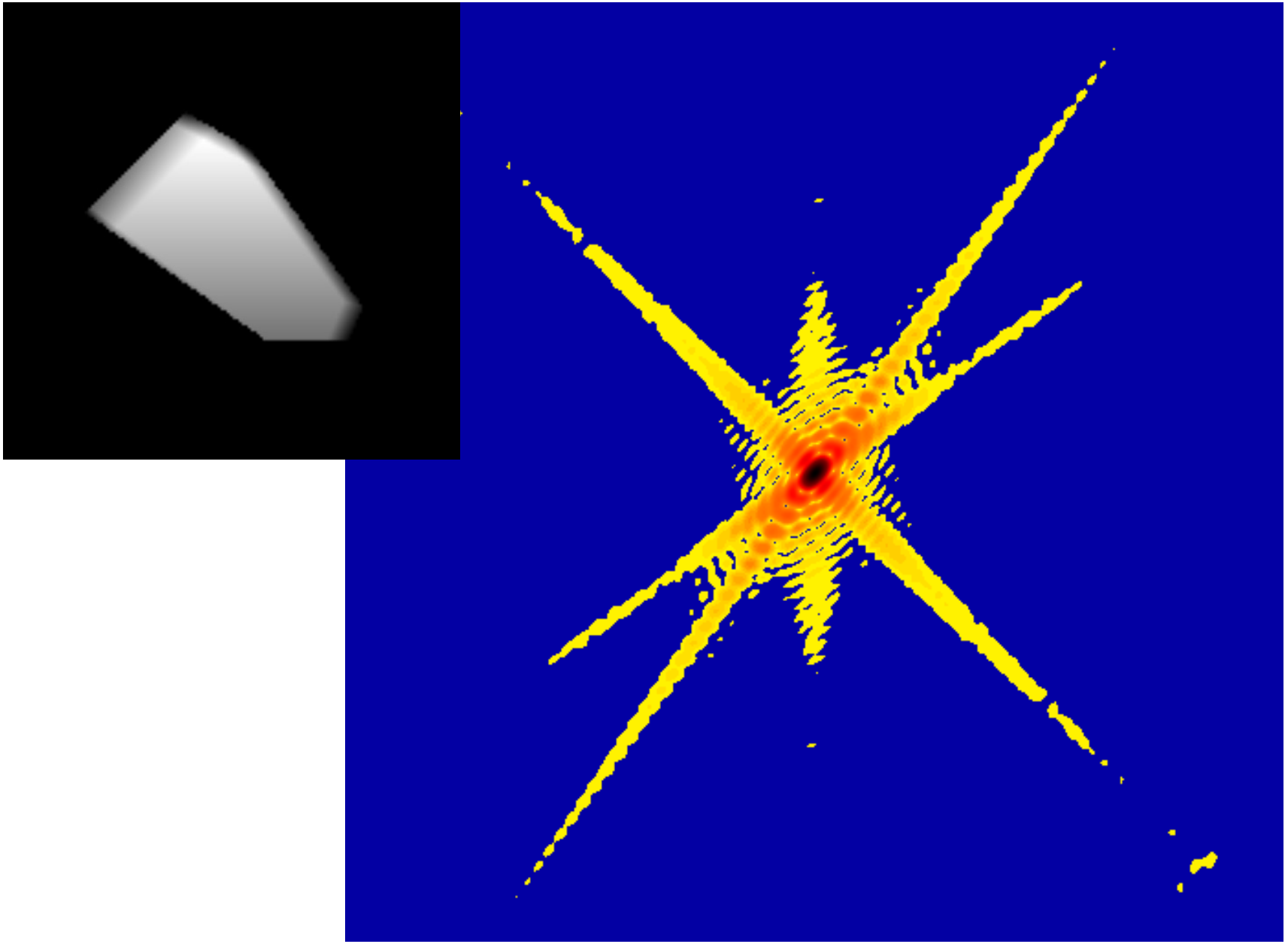
Second International Workshop on Noncrystallographic Phase Retrieval

Outline

- Nanocrystal Shapes
- Vortices During Phasing
- How small can we go?
- Future Directions of CXD

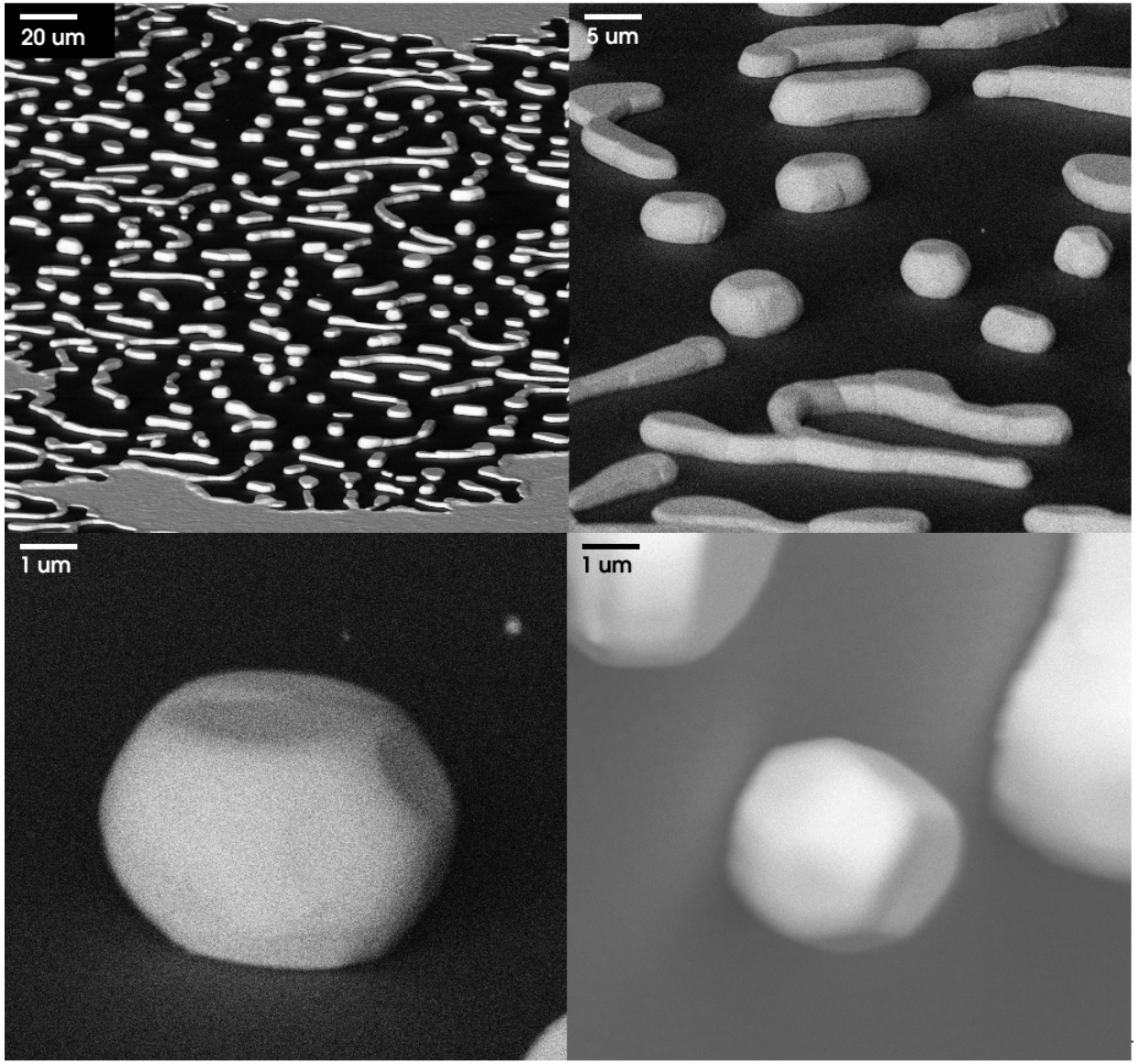
Lensless X-ray Microscope



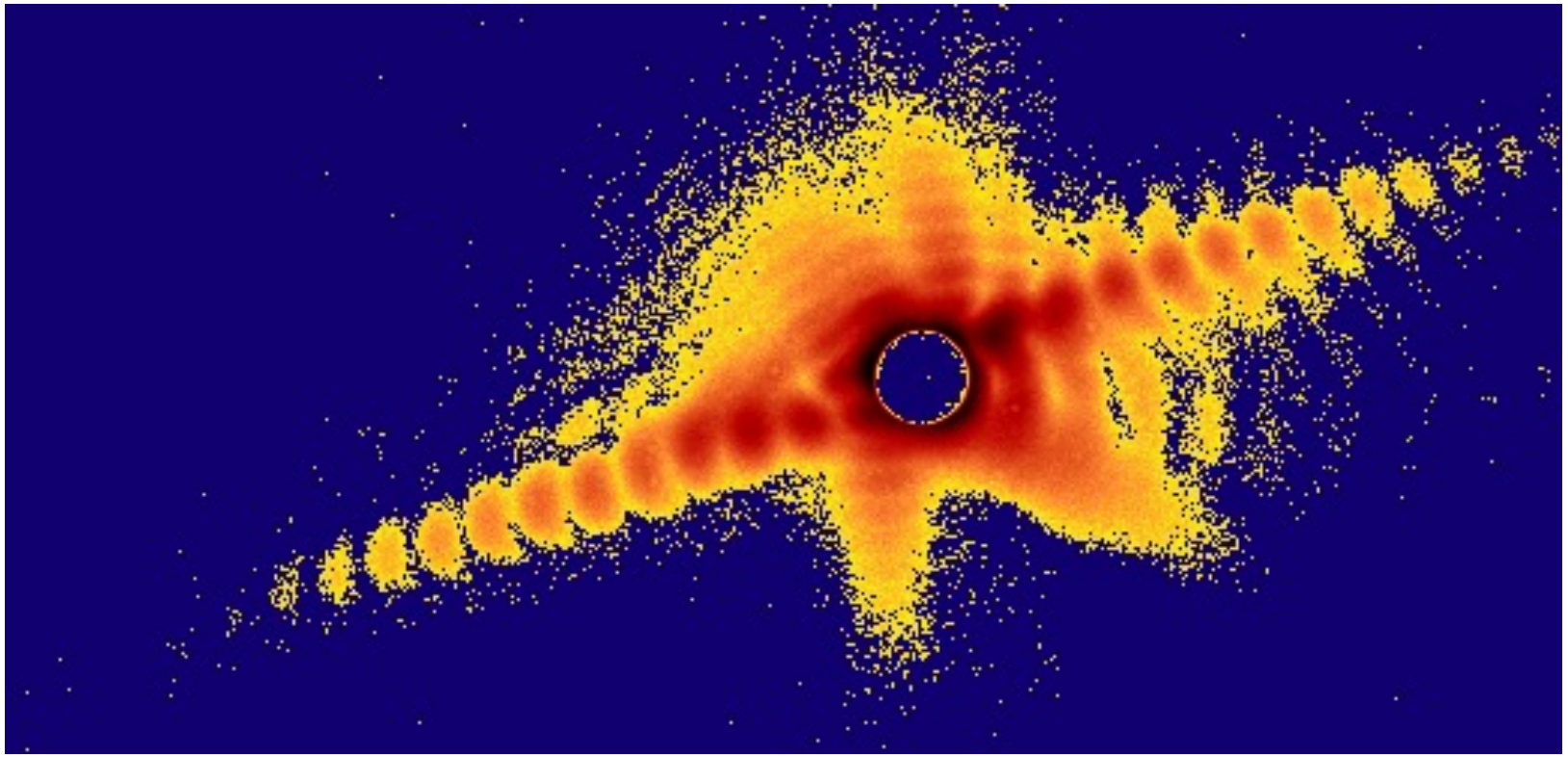


SEMS

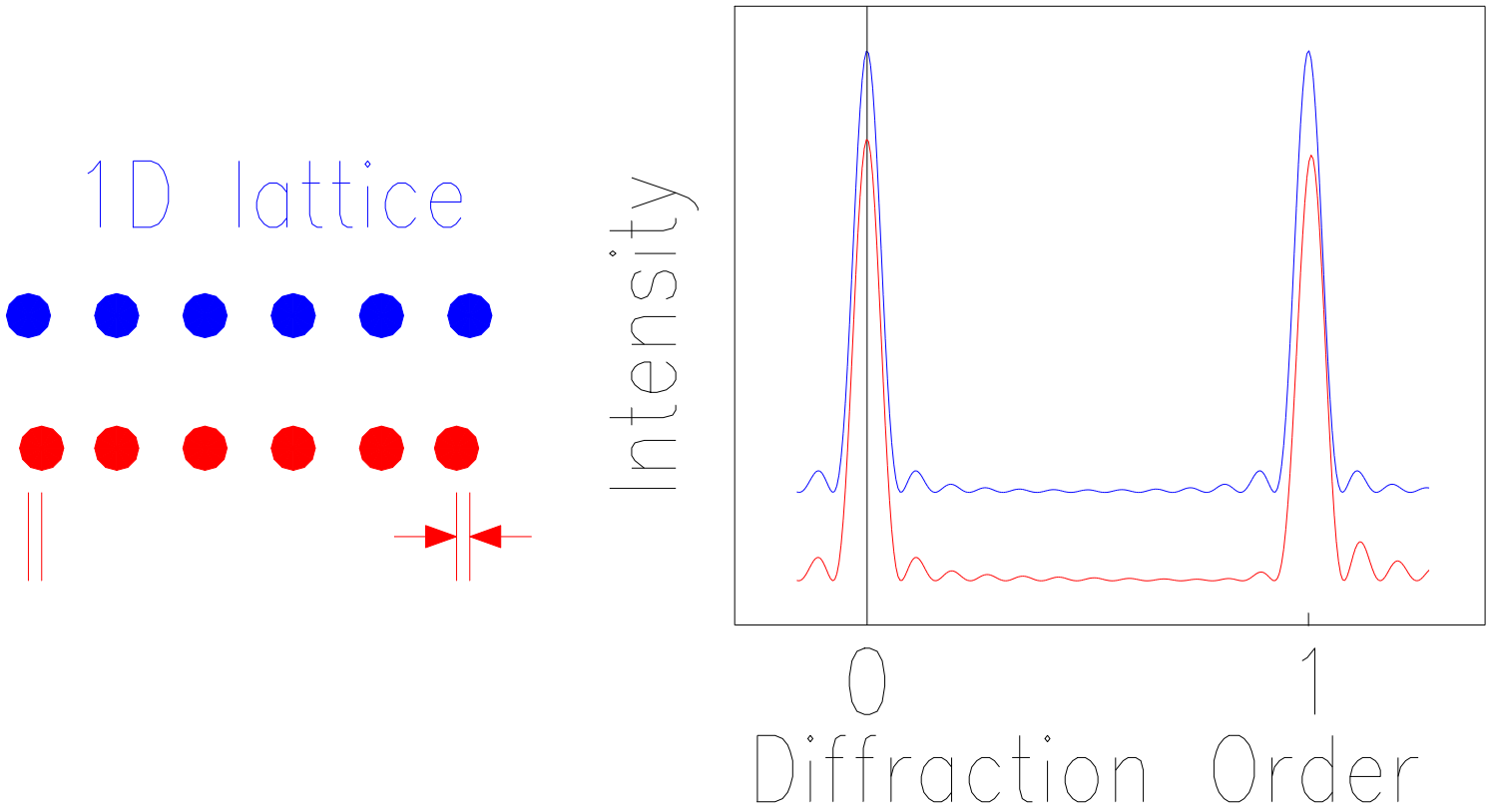
- Au blanket film
- Quartz substrate
- Annealed at 950°C for 70 hrs.



Micron-sized gold crystal: (111) Bragg reflection

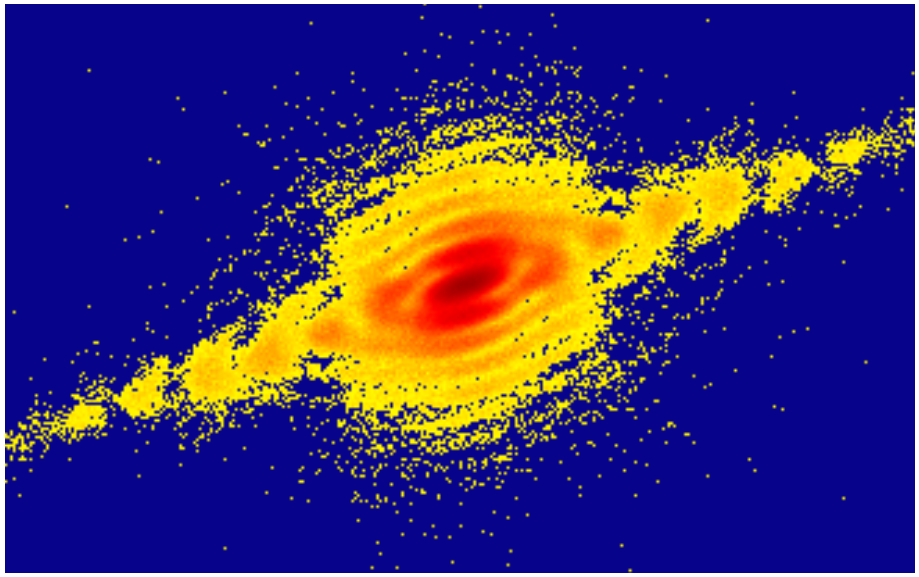


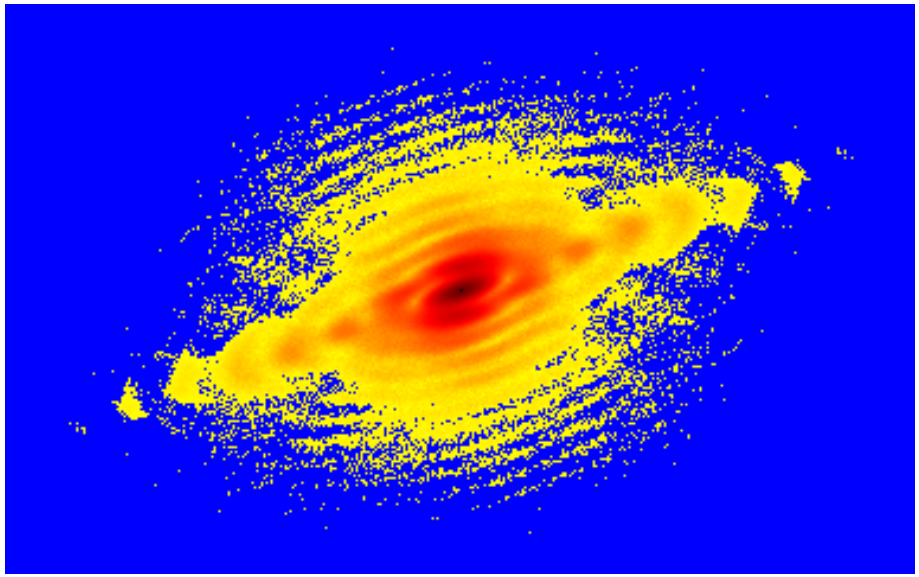
Imaging of Lattice Strains

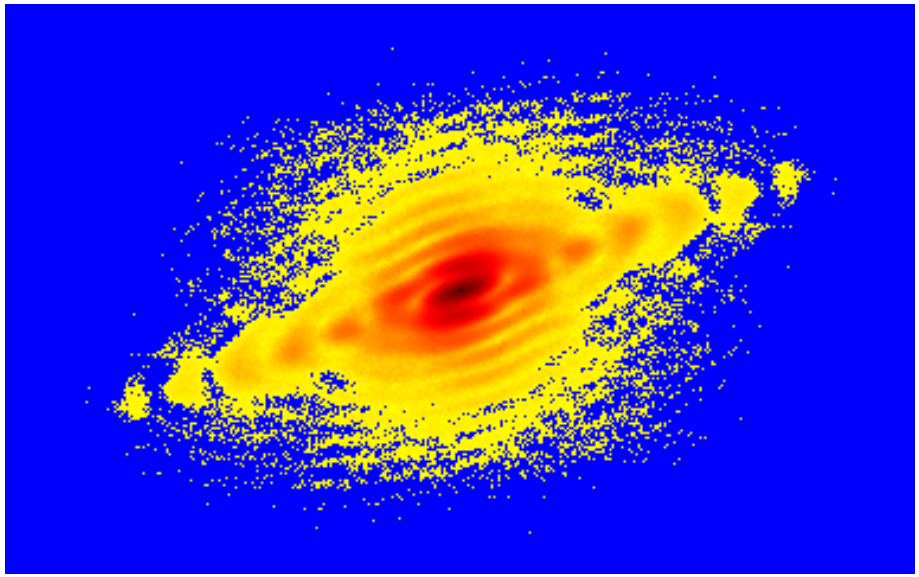


Symmetrized Data and two best fits

Chisq=0.0005

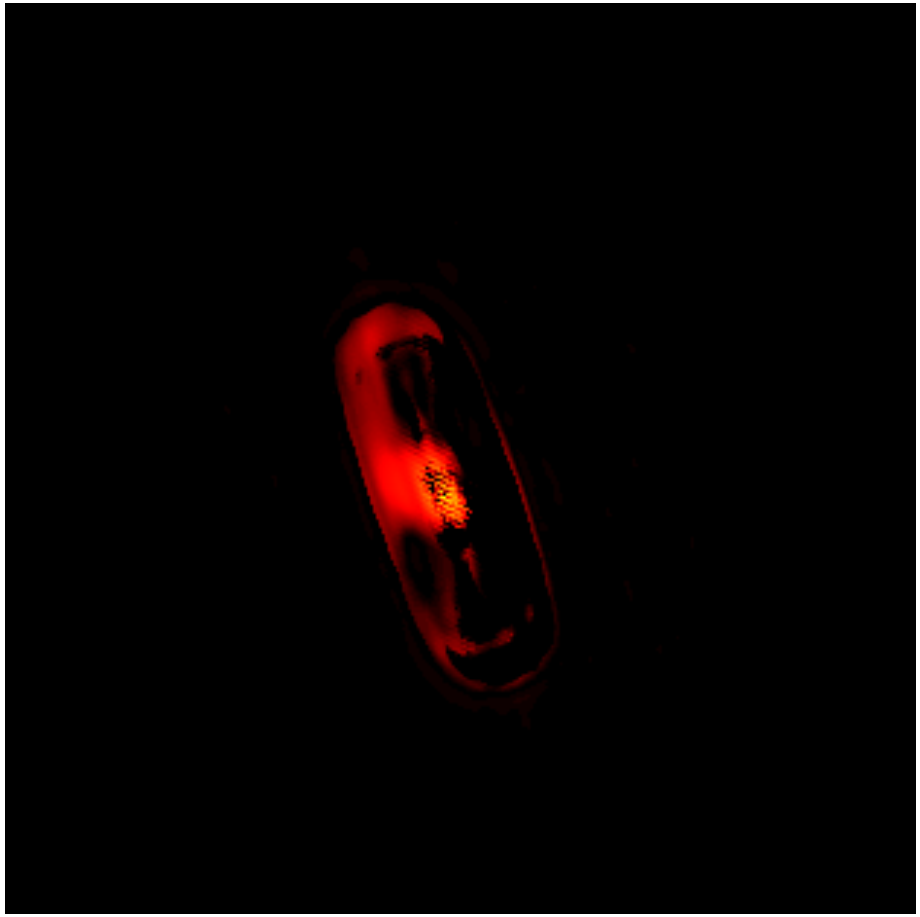


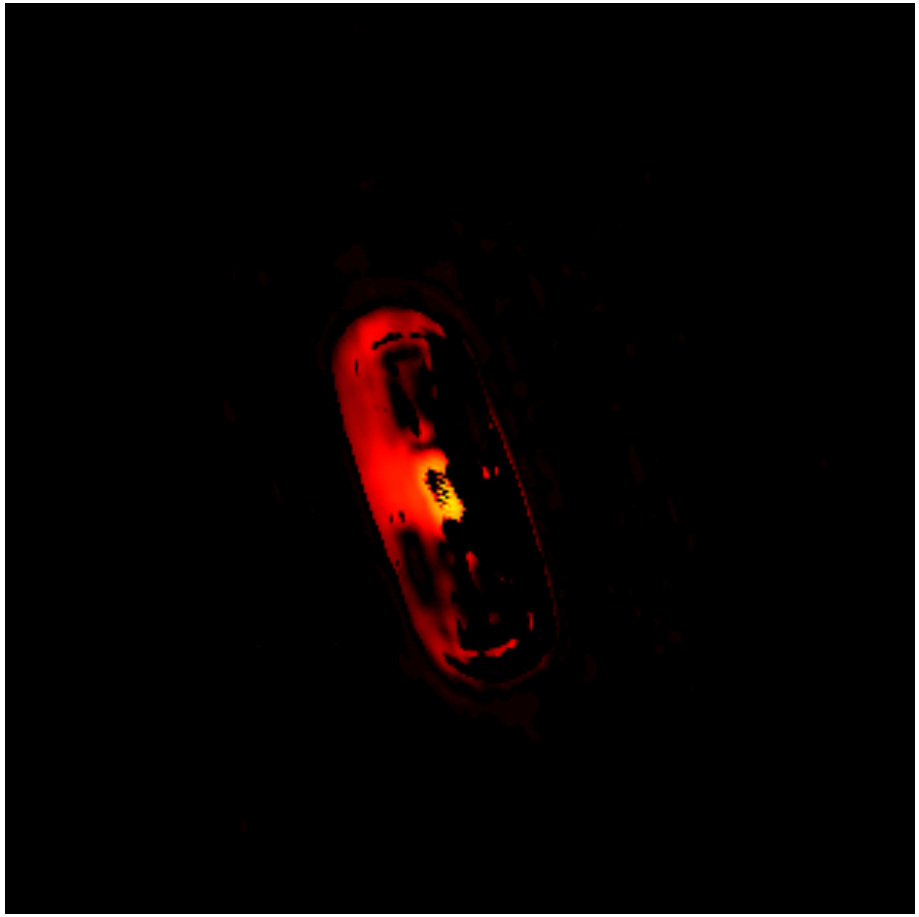




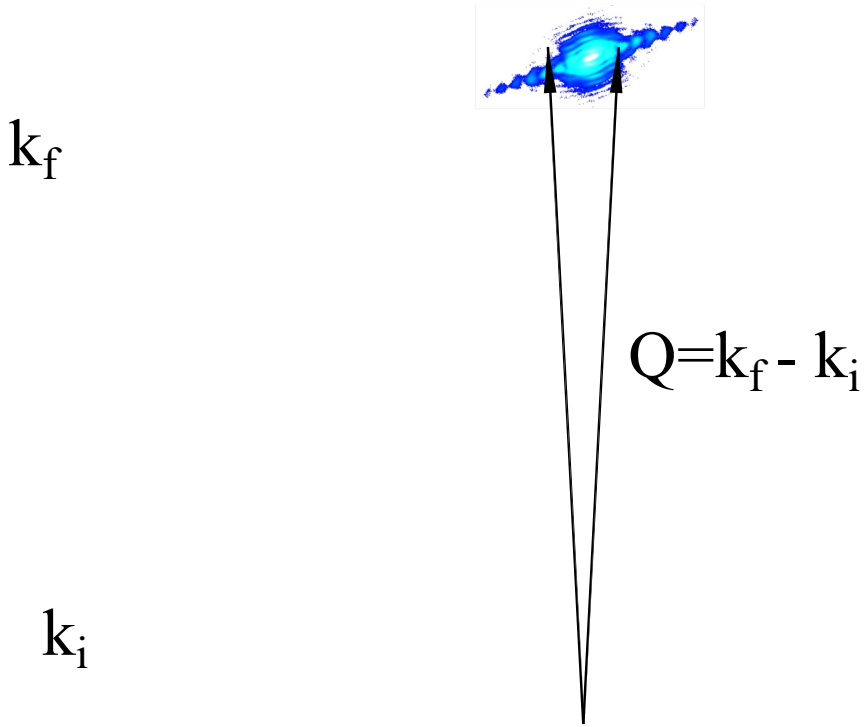
2D Reconstructions

chisquare = 0.0005

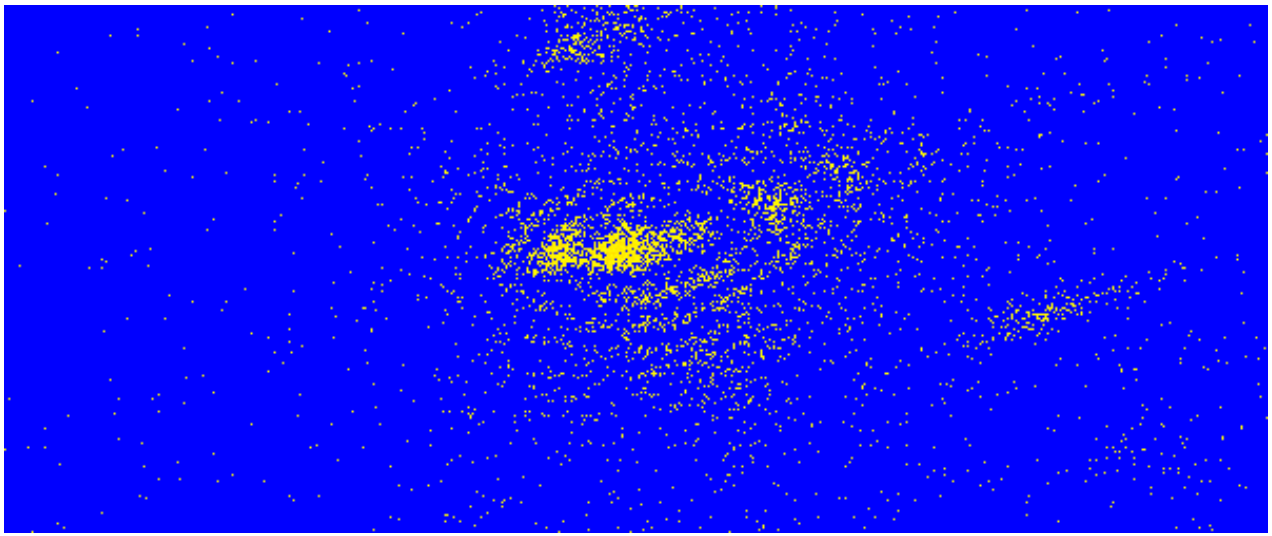




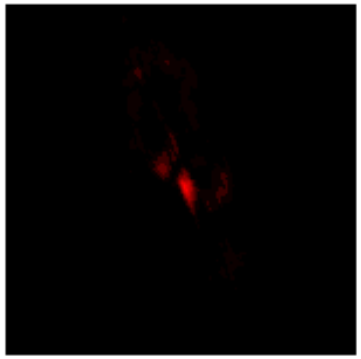
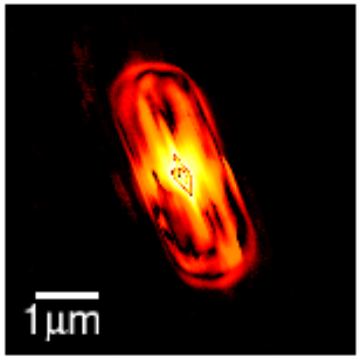
3D Diffraction Method

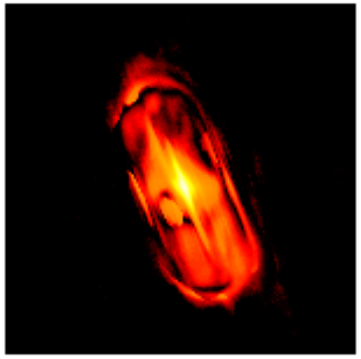
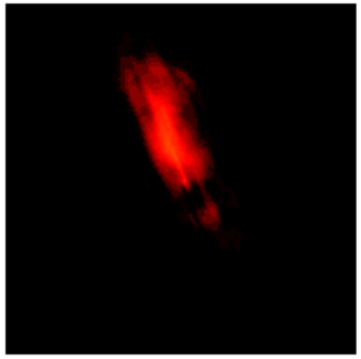
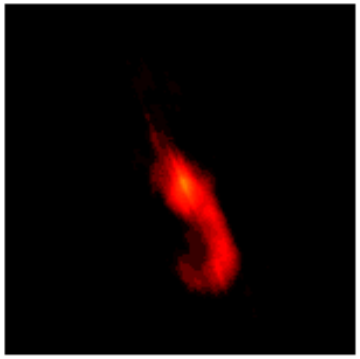
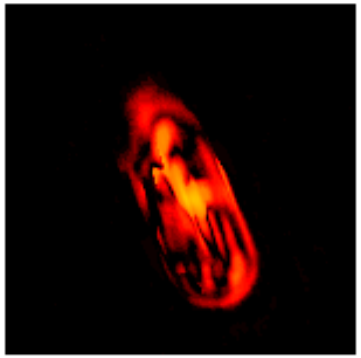
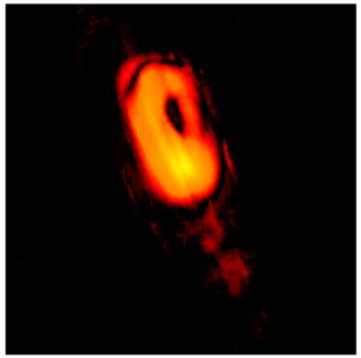
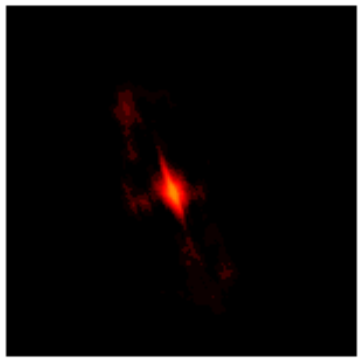
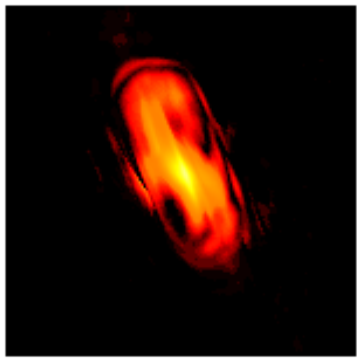
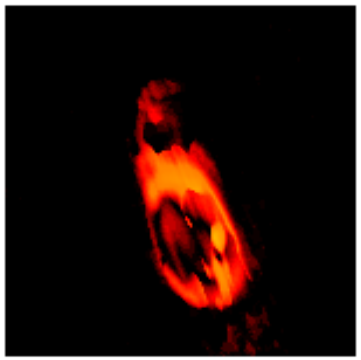
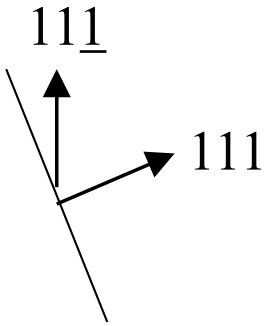


3D Diffraction Data
1 micron Au crystal

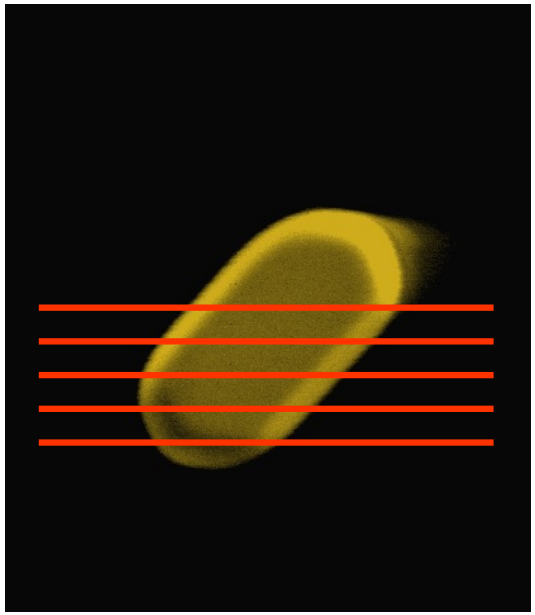


* Center is Symmetric *

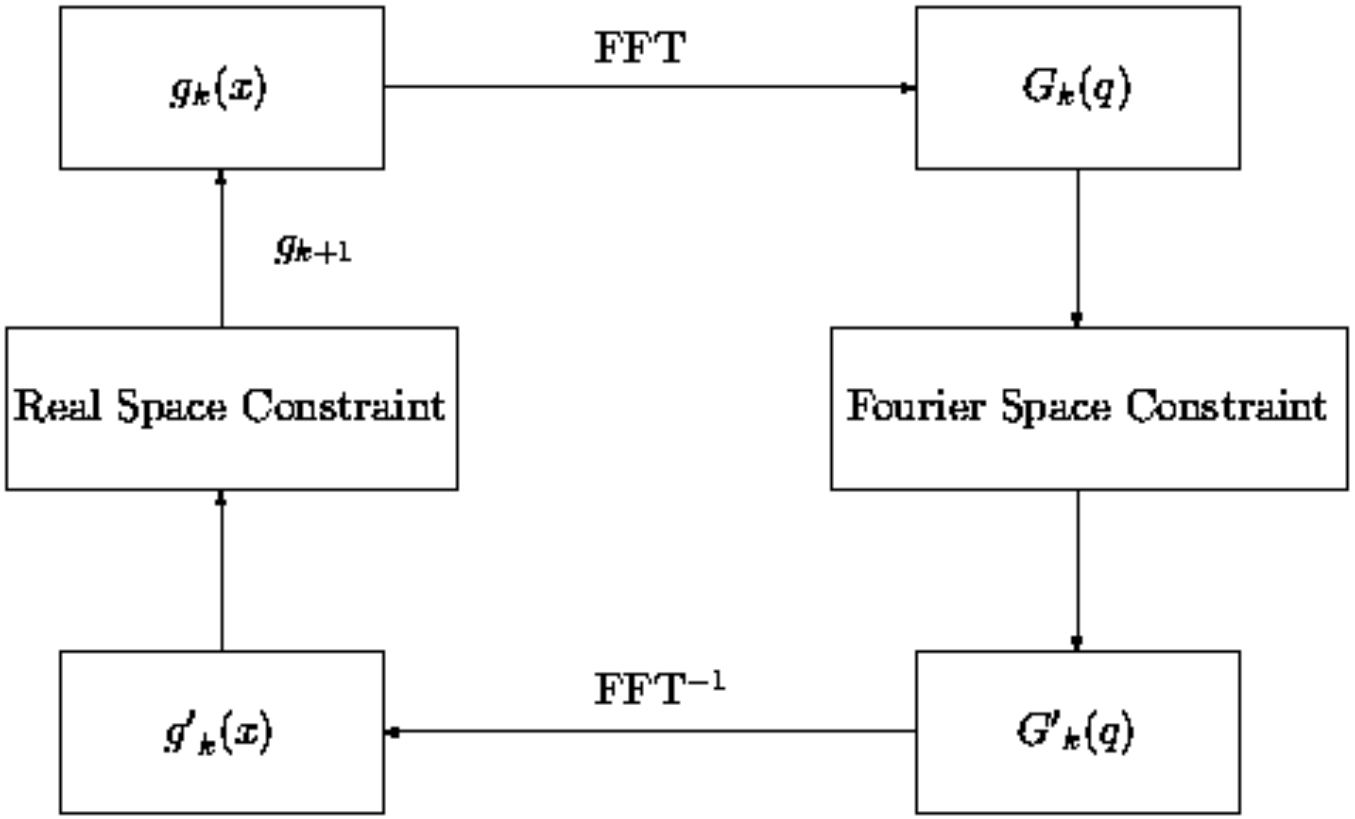



Slices through plan view SEM:



Generic "Error Reduction" method



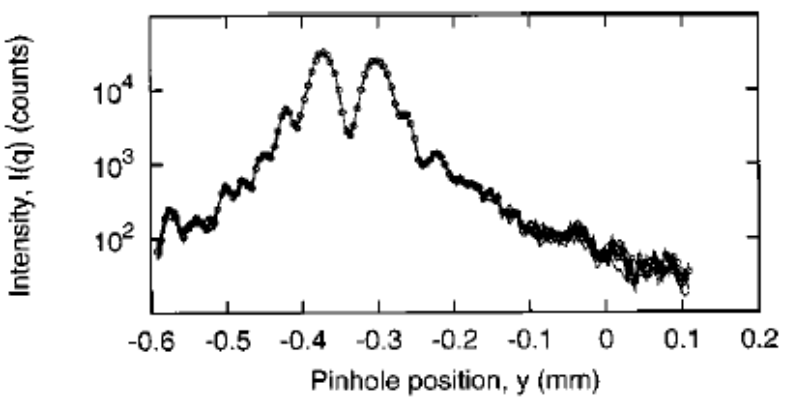
J. R. Fienup Appl. Opt. 21 2758 (1982)
R. W. Gerchberg and W. O. Saxton Optik 35 237 (1972)

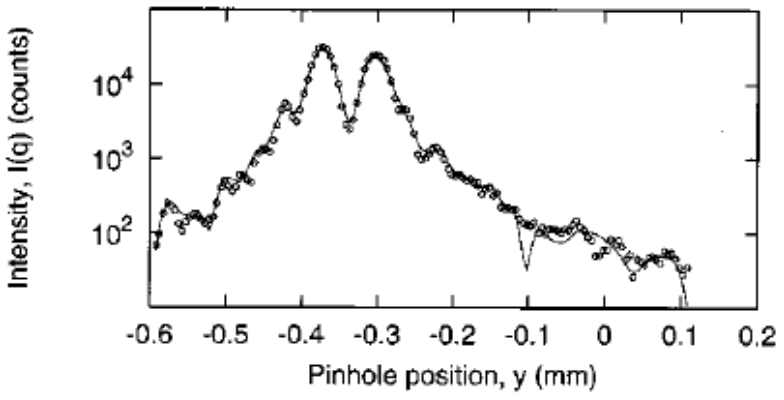
Real-space Constraints in Crystallograhy

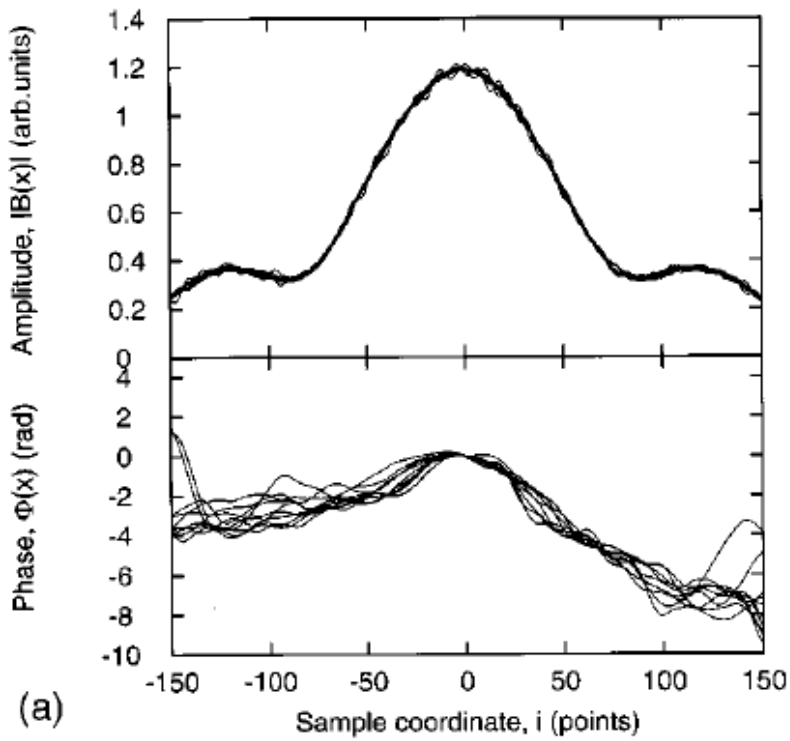
R. P. Millane, J. Opt. Soc Am. A **13** 725 (1996)

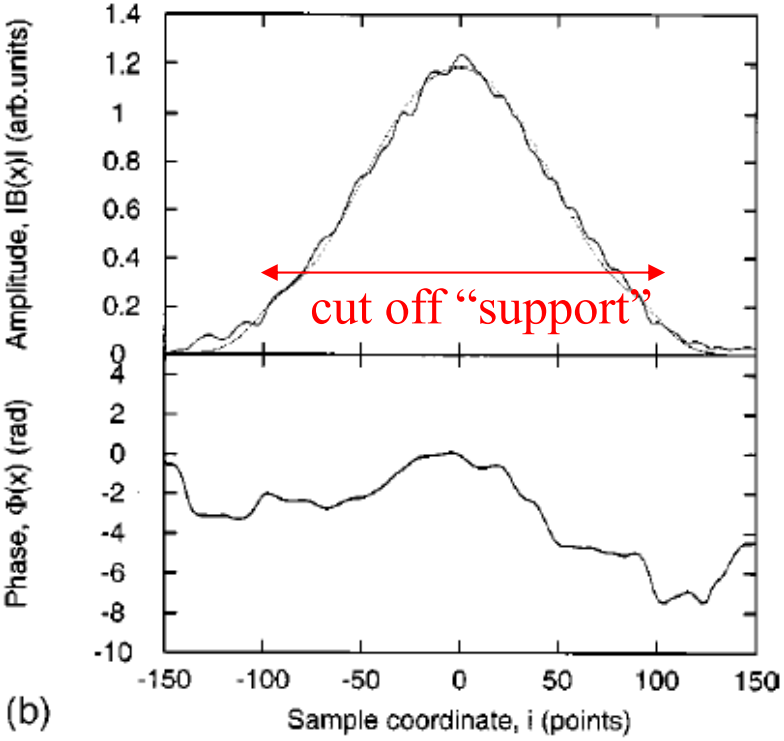
- 'Positivity' constraint (Sayre)
- Finite support, molecular envelope
- Solvent flattening
- Molecular replacement
- Non-crystallographic symmetry
- Non-uniqueness is 'pathologically rare' (d>1)

Phasing using G-S Algorithm

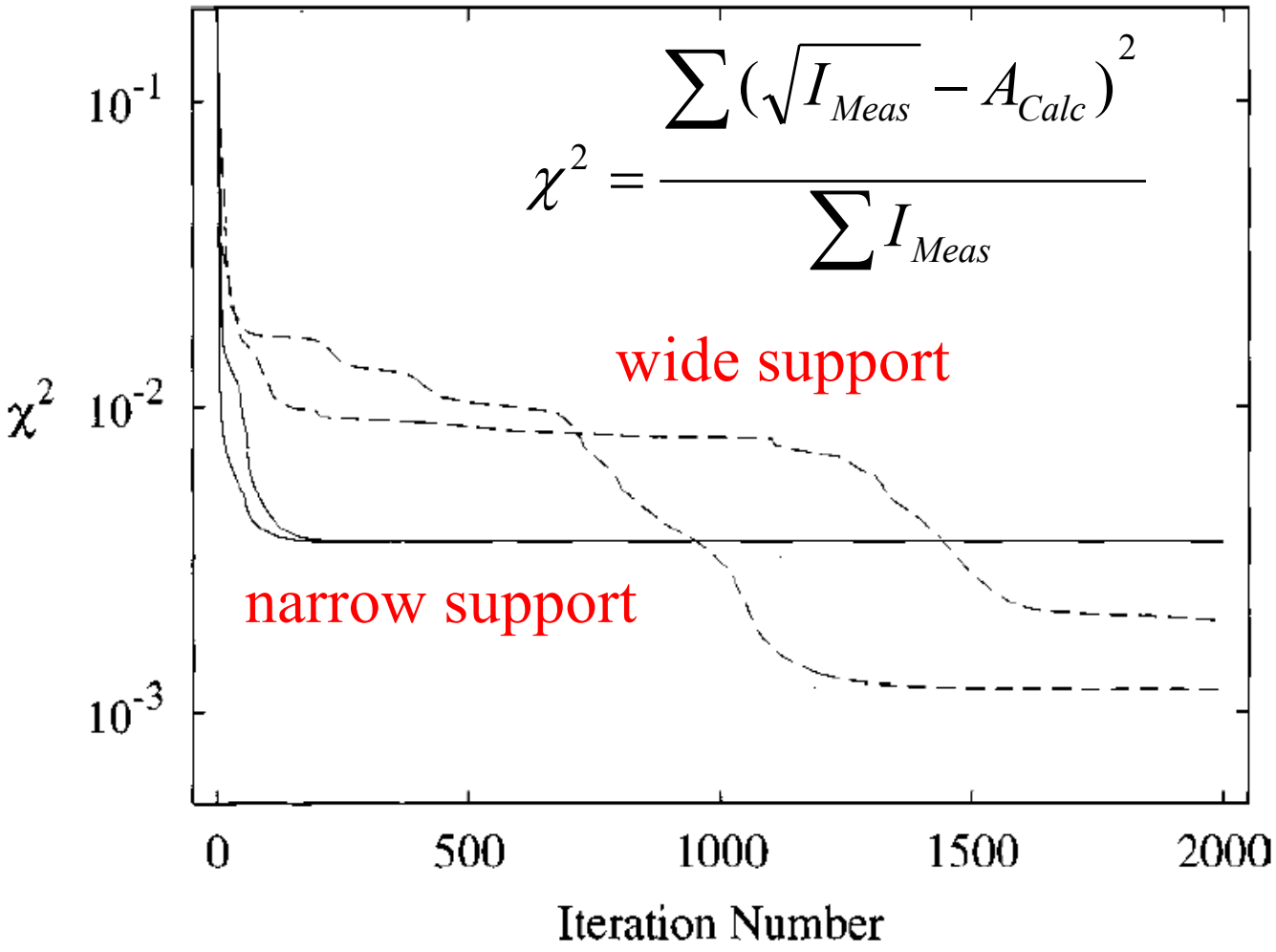






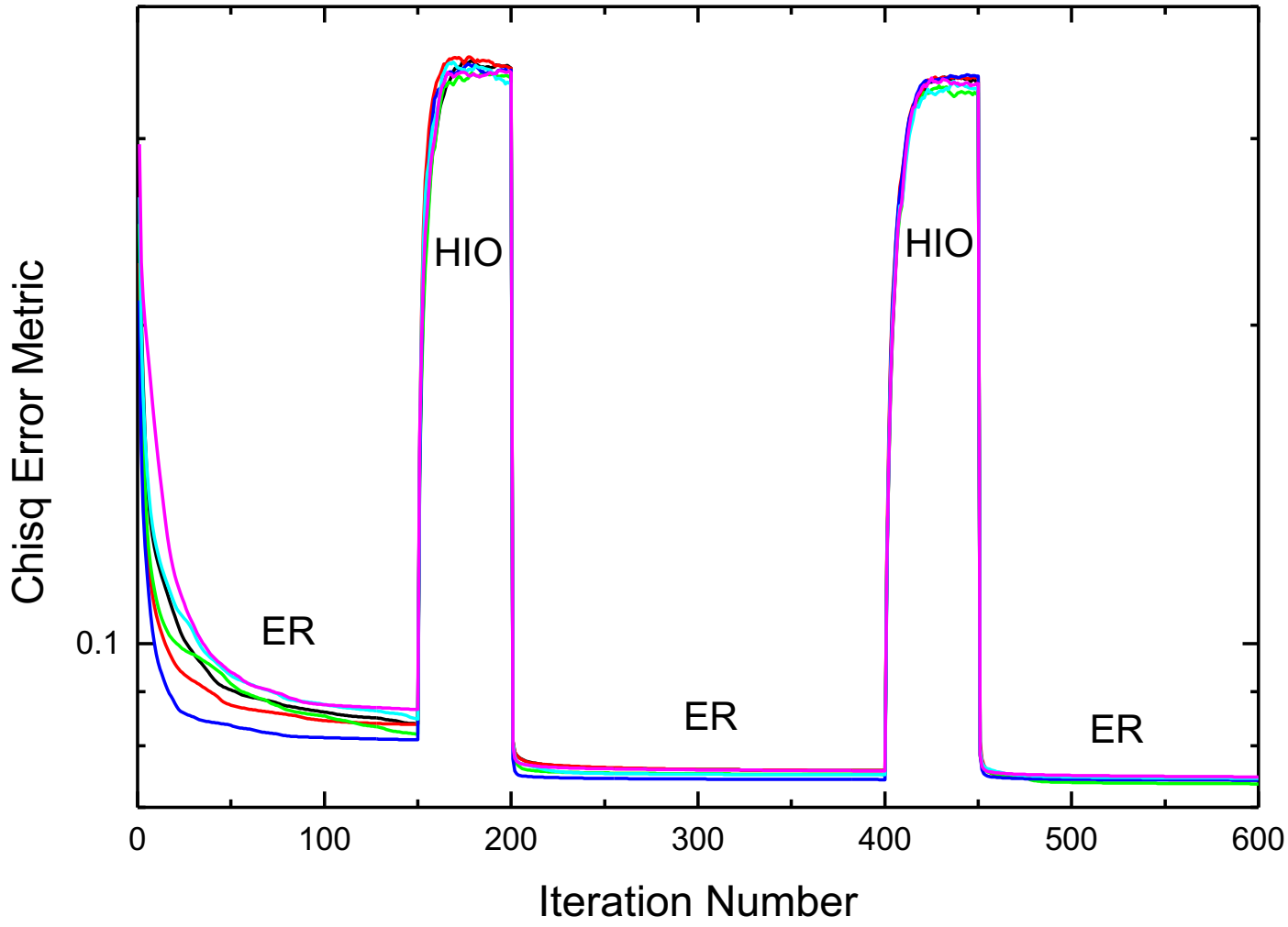


Convergence Trajectory



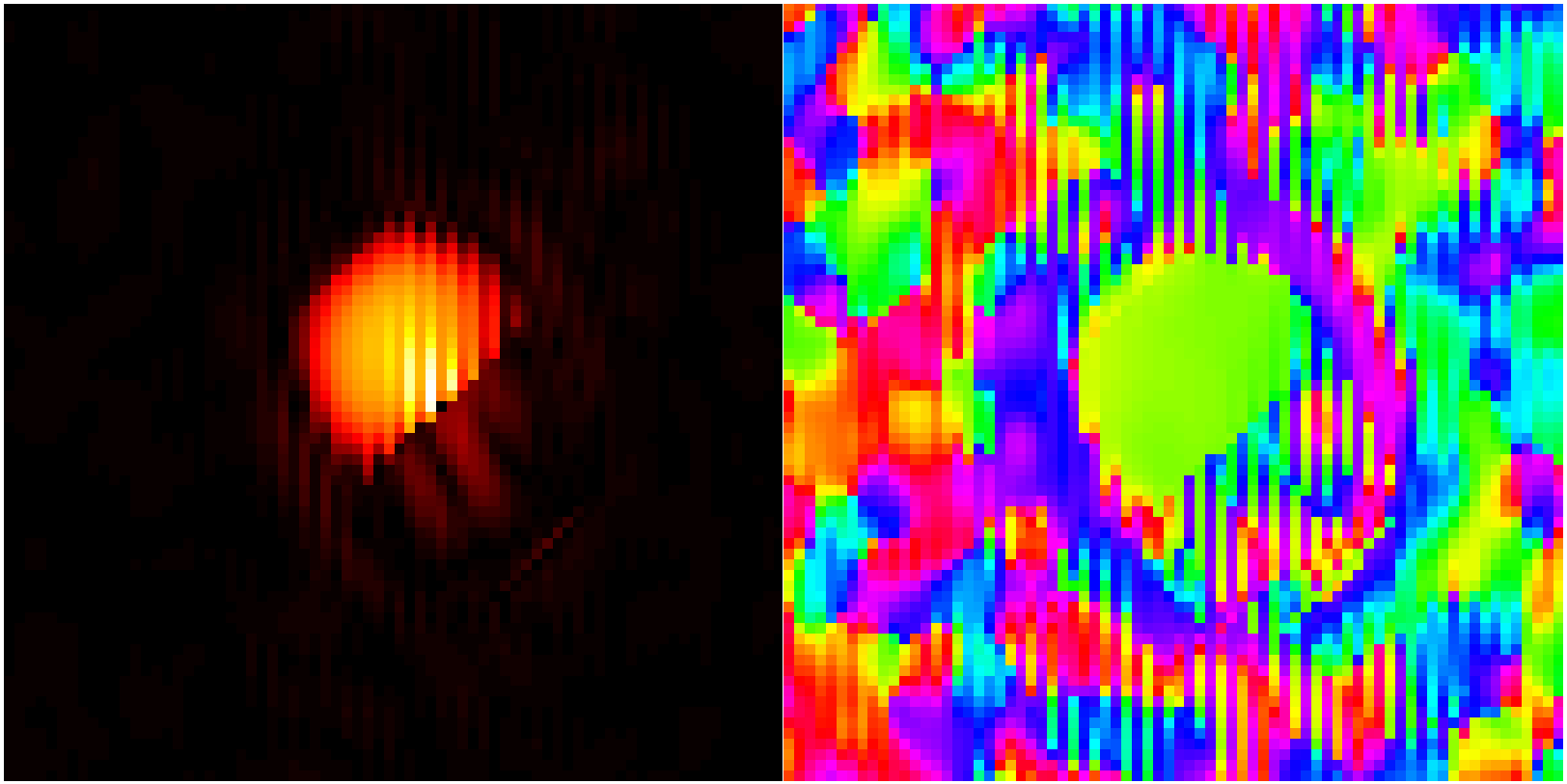
Alternation of ER and HIO

Helps to avoid stagnation



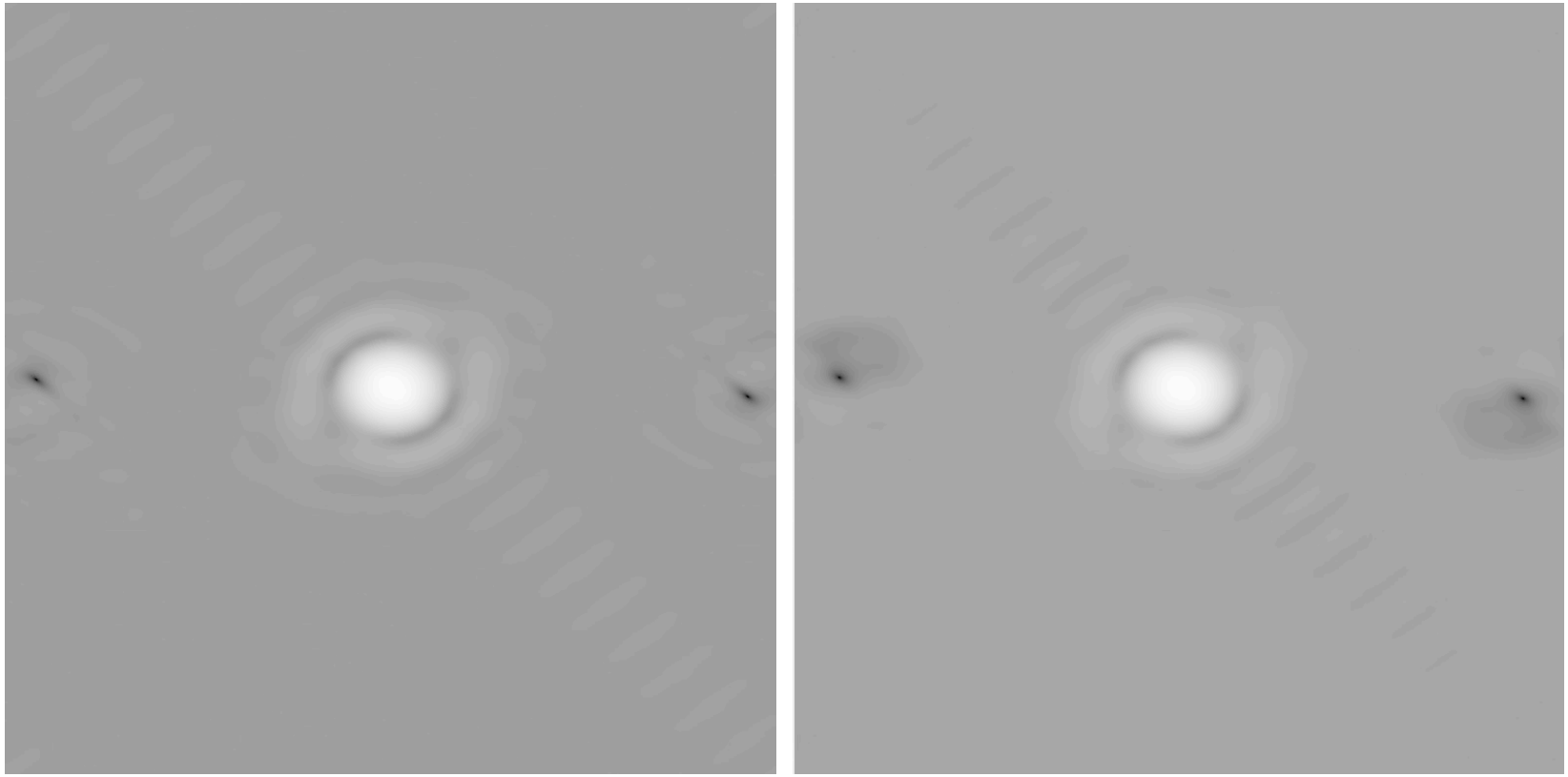
Incomplete Reconstruction can be Striped

0.5 micron Pb crystal on SiO_2 substrate

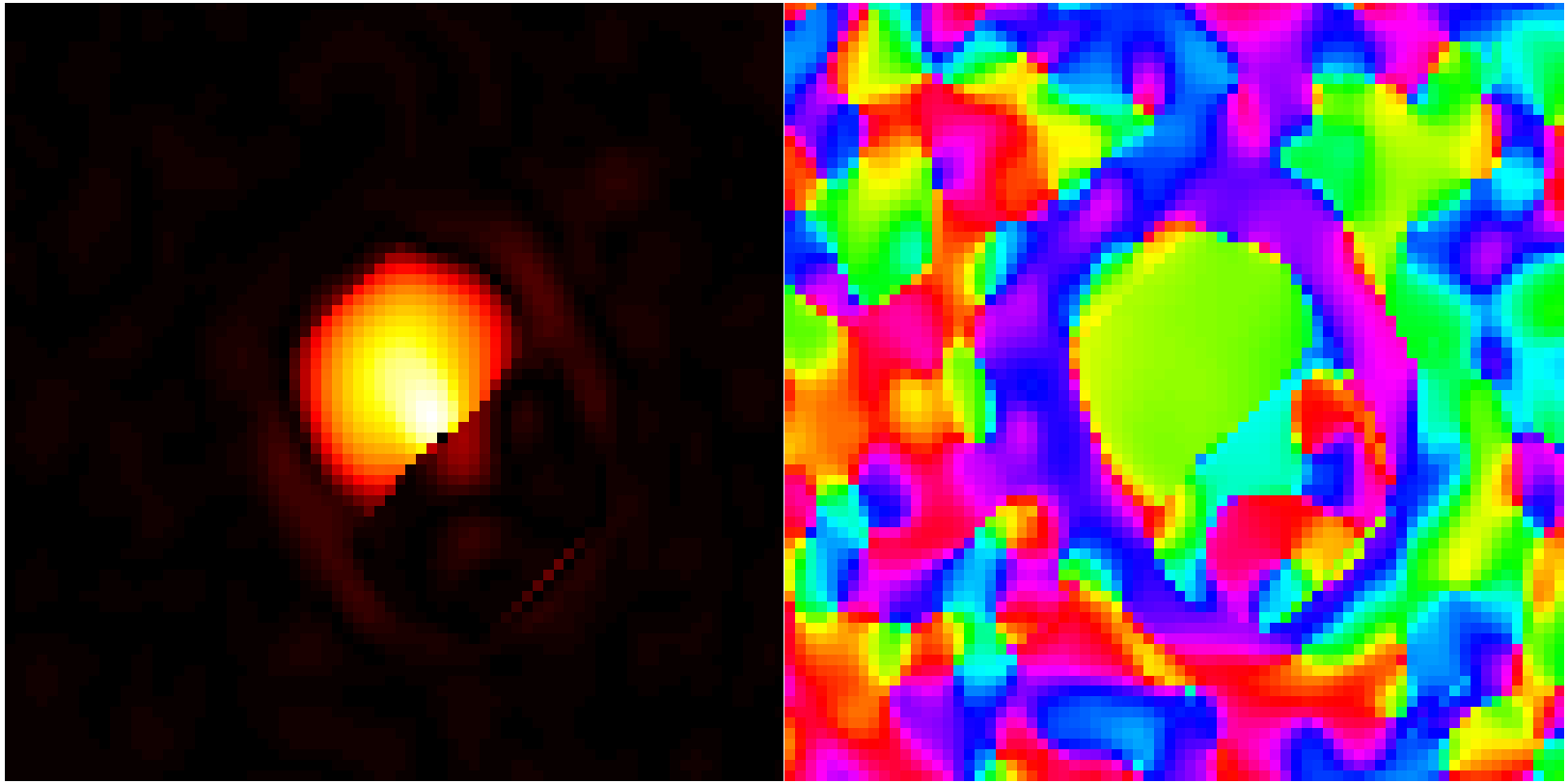


Stripes caused by “Vortices”

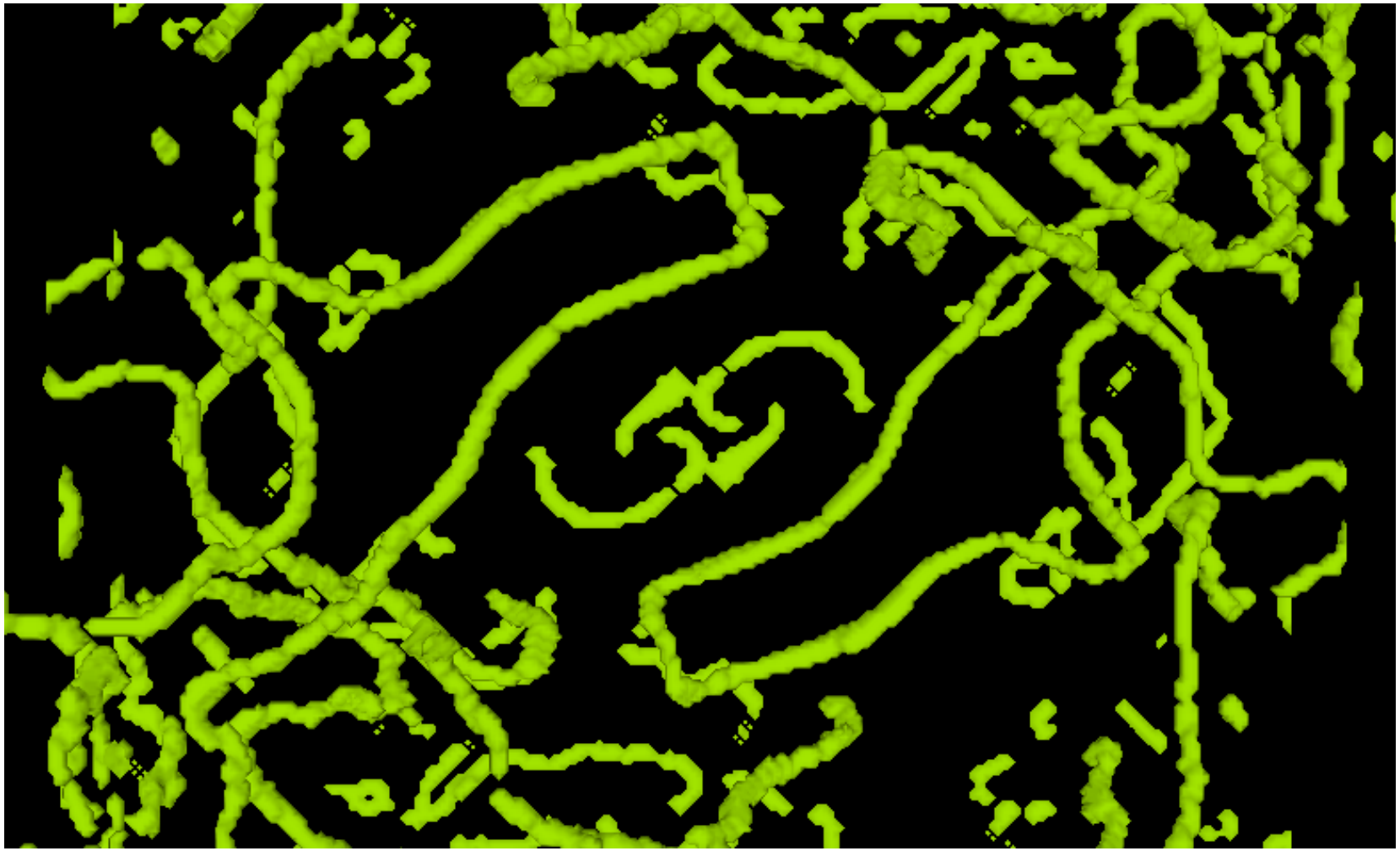
Vortex pairs separated by inverse of stripe spacing



Result of “Patching” in 2D

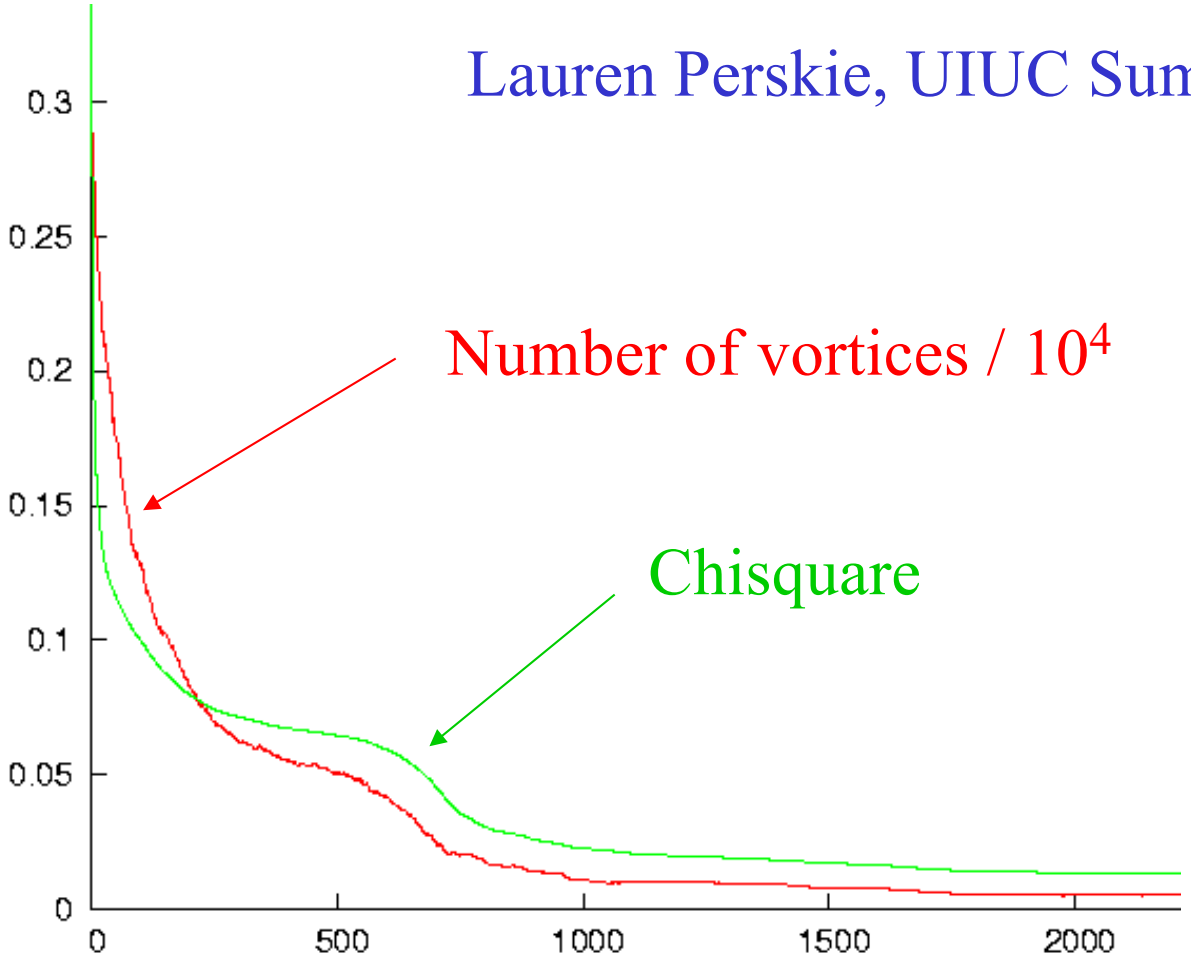


3D Vortices Form Pairs of Loops

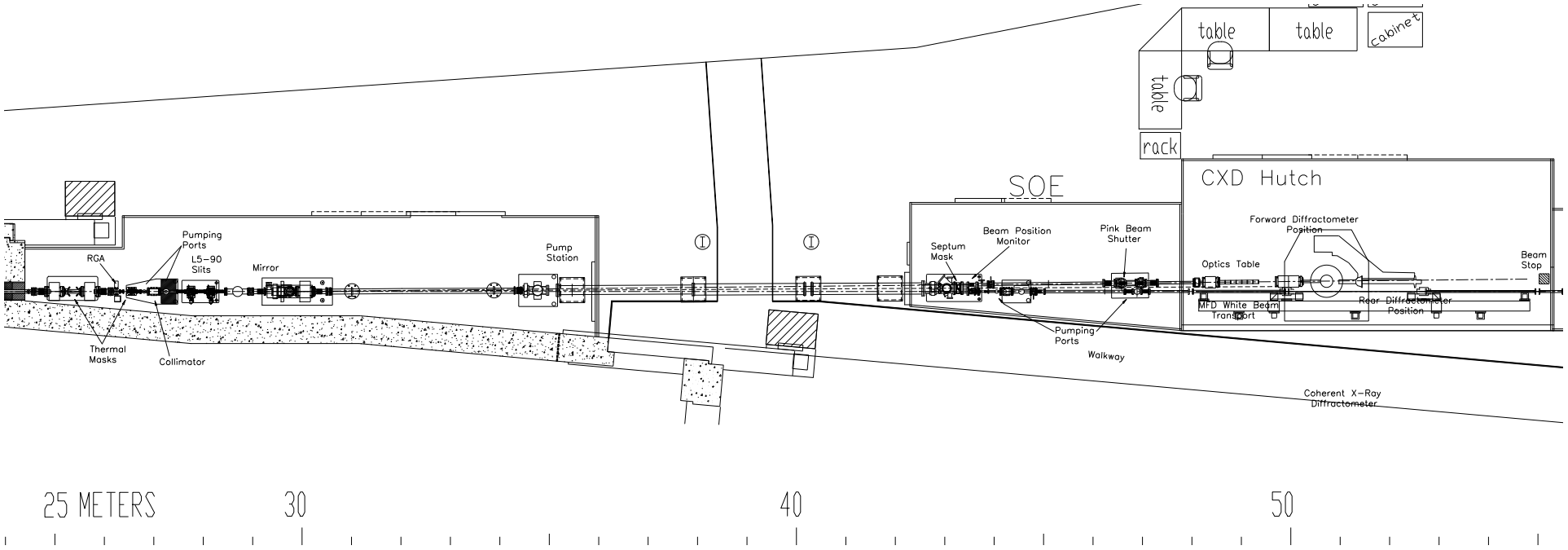


Vortices are a Cause of Stagnation during Error Reduction

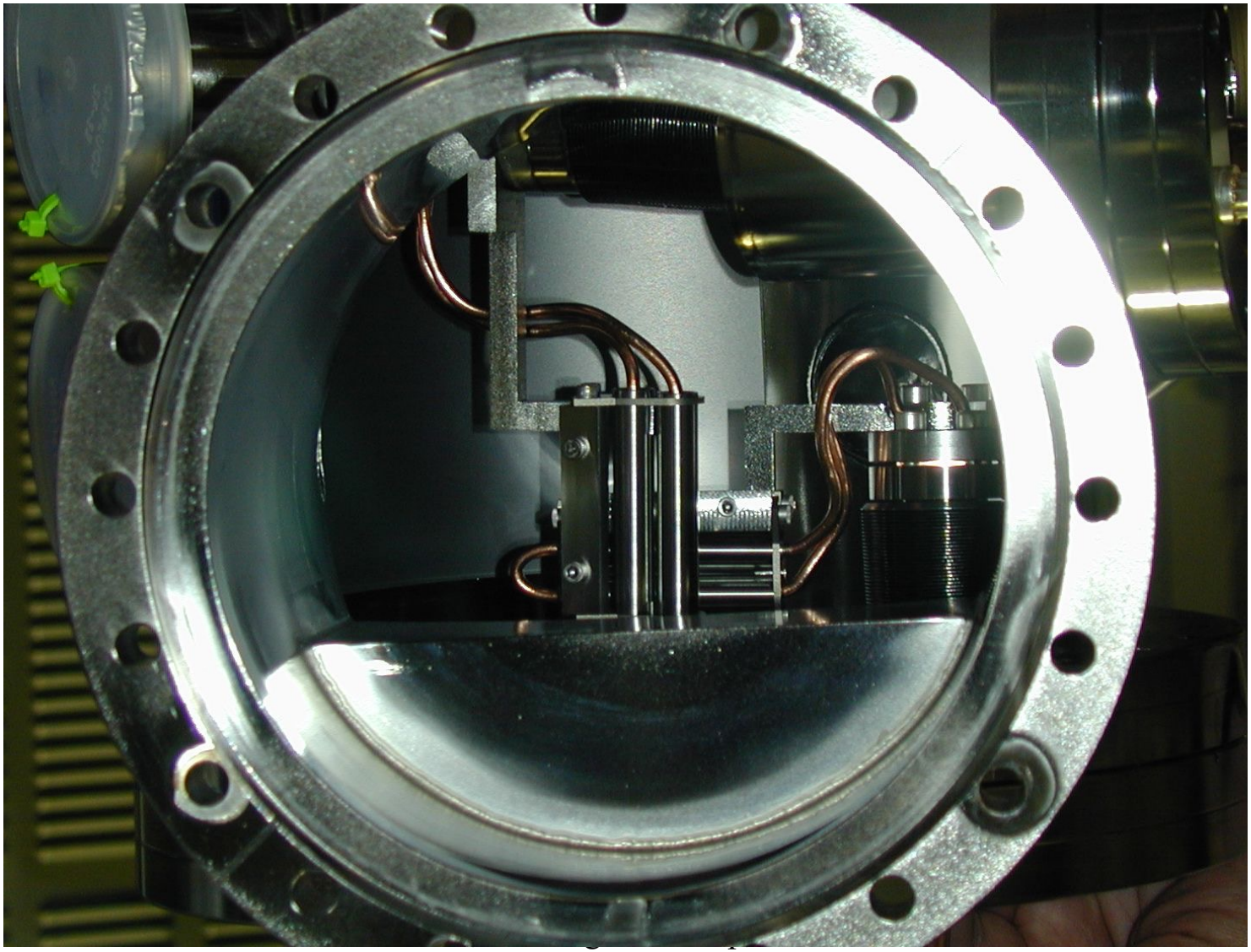
Lauren Perskie, UIUC Summer student



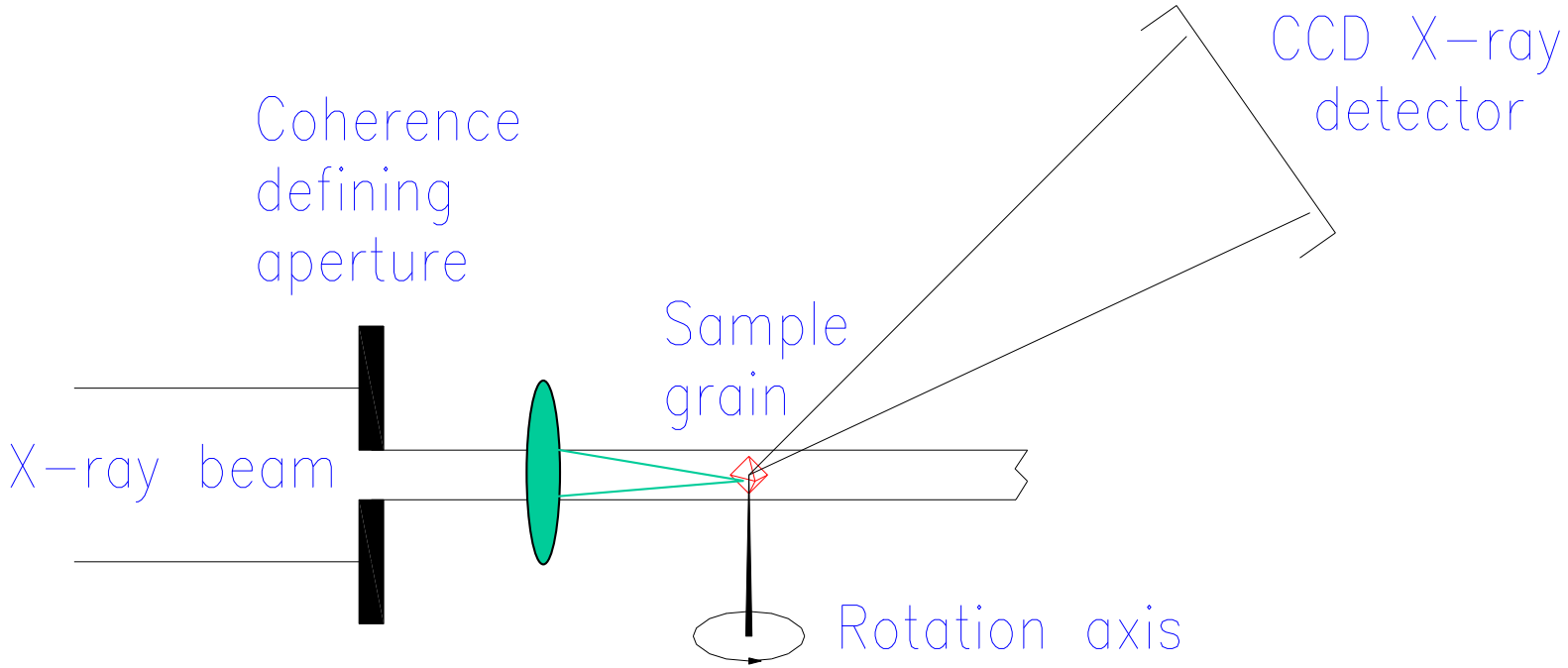
CXD Beamline at APS Sector 34



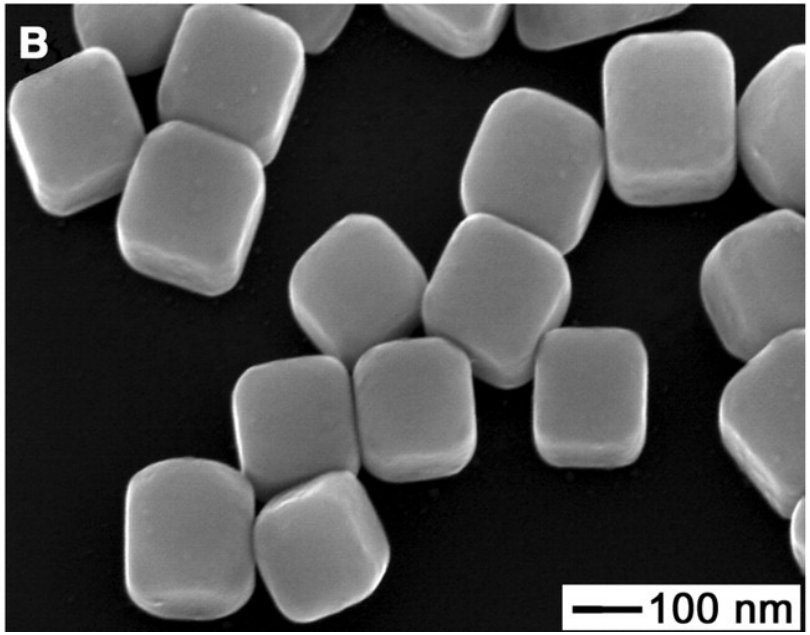
Roller-Blade Slits in UHV

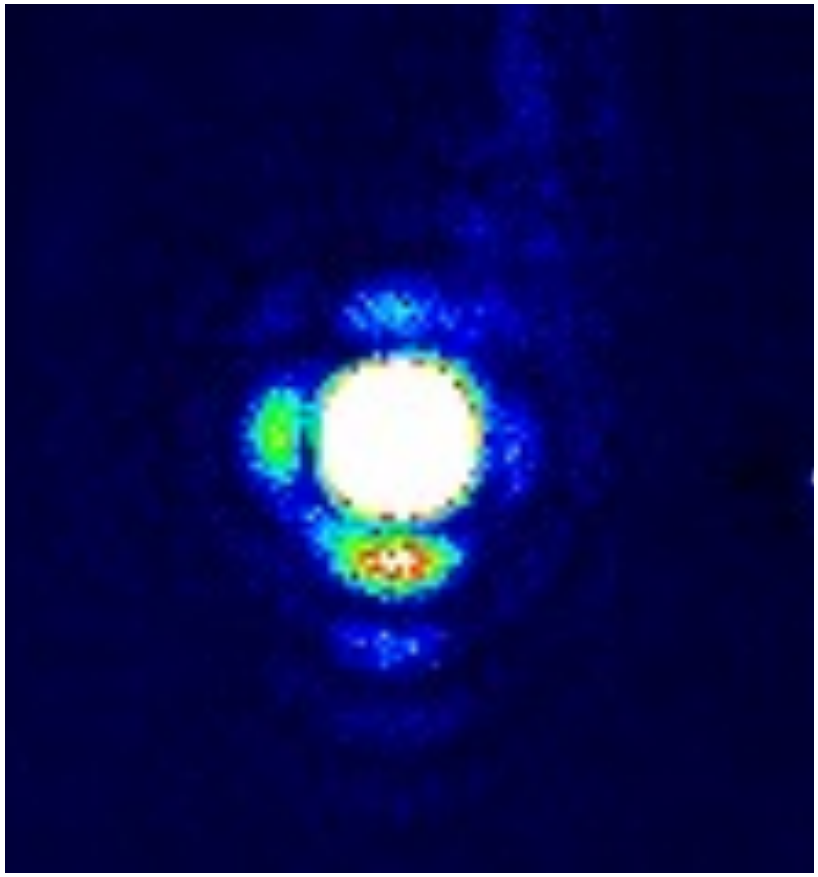


Lensless X-ray Microscope



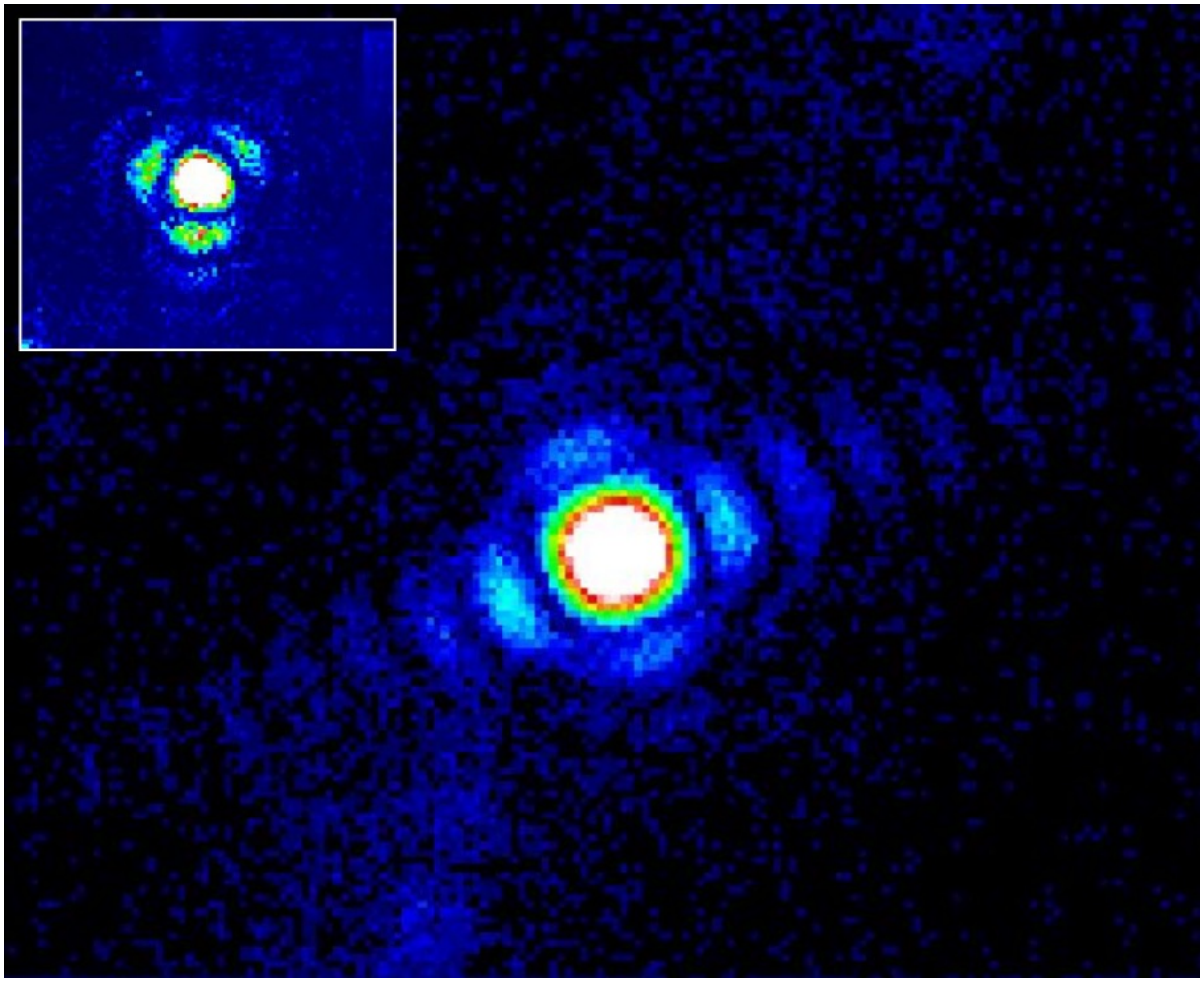
CXD from Silver Nanocubes





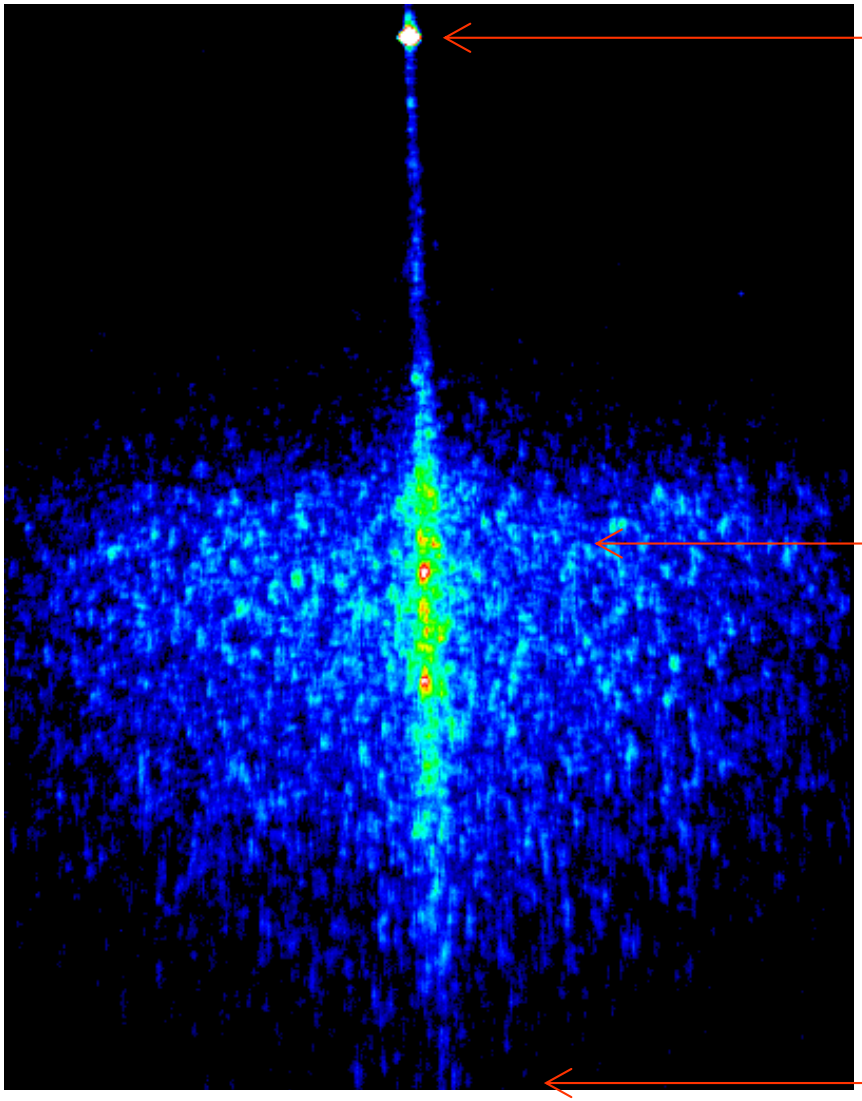
Yugang Sun and Younan Xia,
Science 298 2177 (2003)

170nm Silver Nanocubes



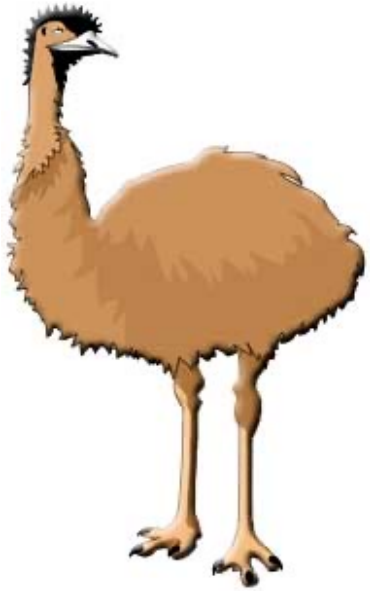
Structure in "Yoneda" Peak

Grazing-exit diffraction from a 1000A Au polycrystalline film



Specular ($\alpha_f \sim \alpha_i$)

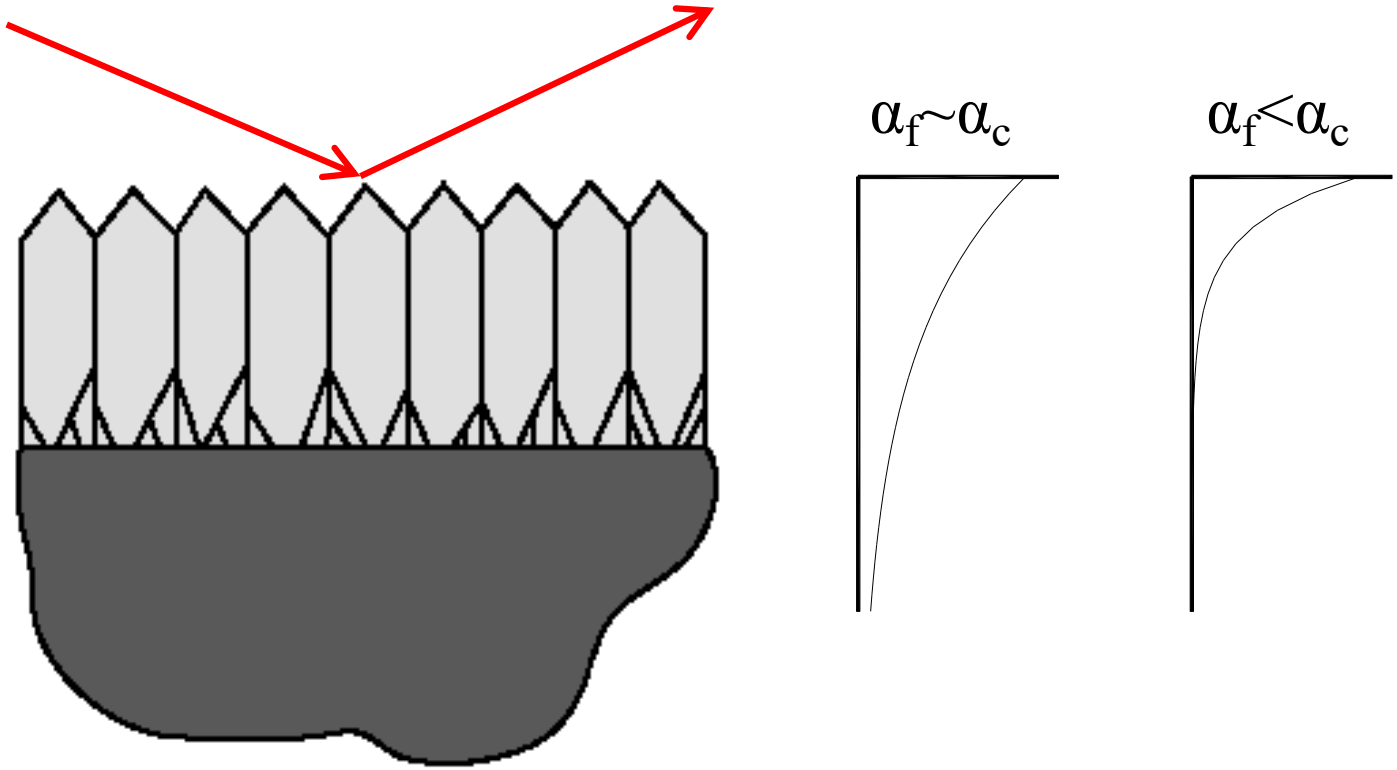
$\alpha_f \sim \alpha_c$



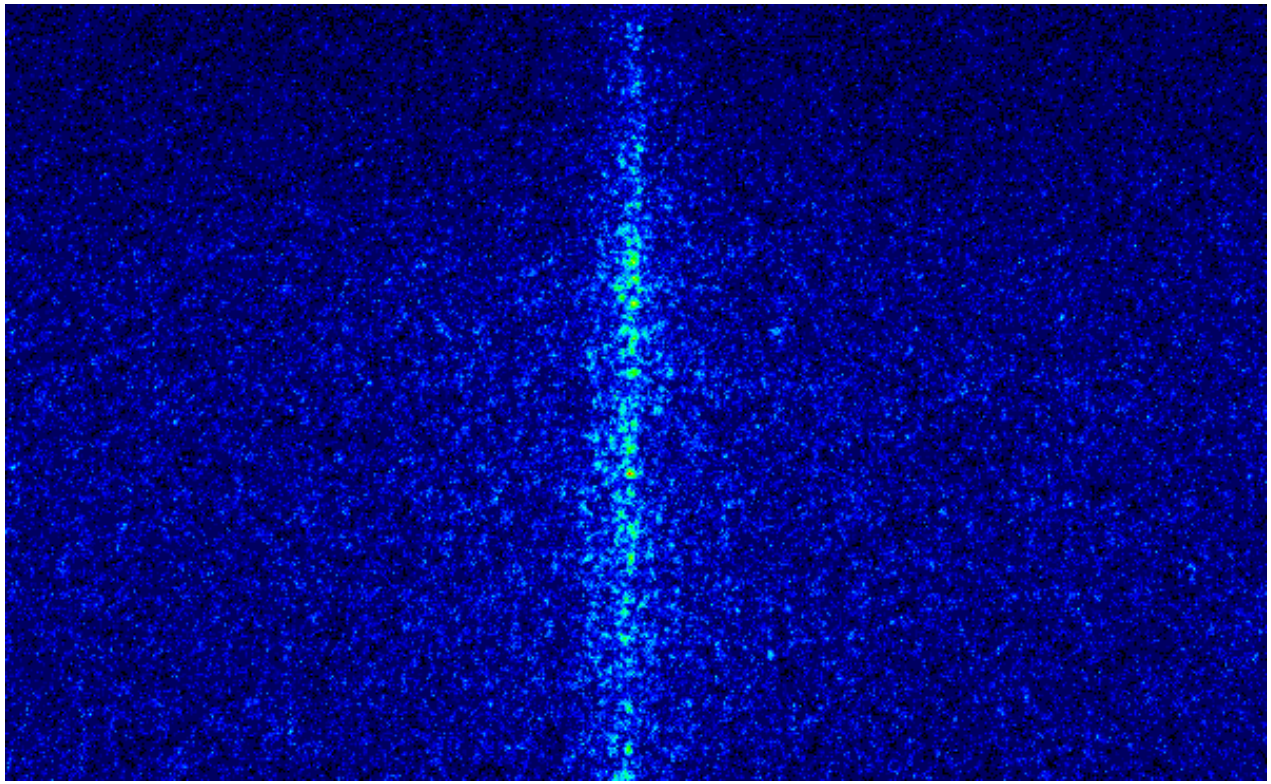
$\alpha_f \sim 0$

Competitive Grain Growth

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



Angle series, 0.01° steps

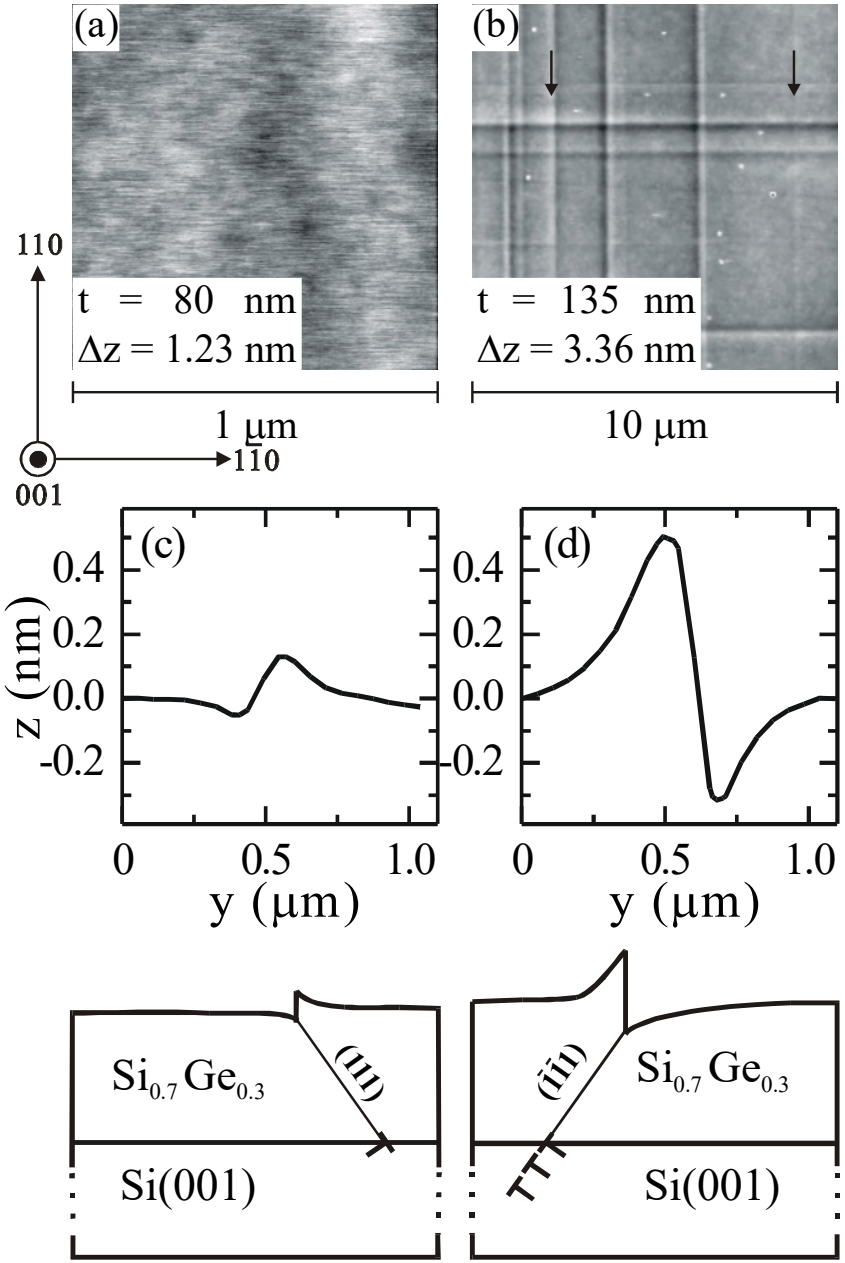


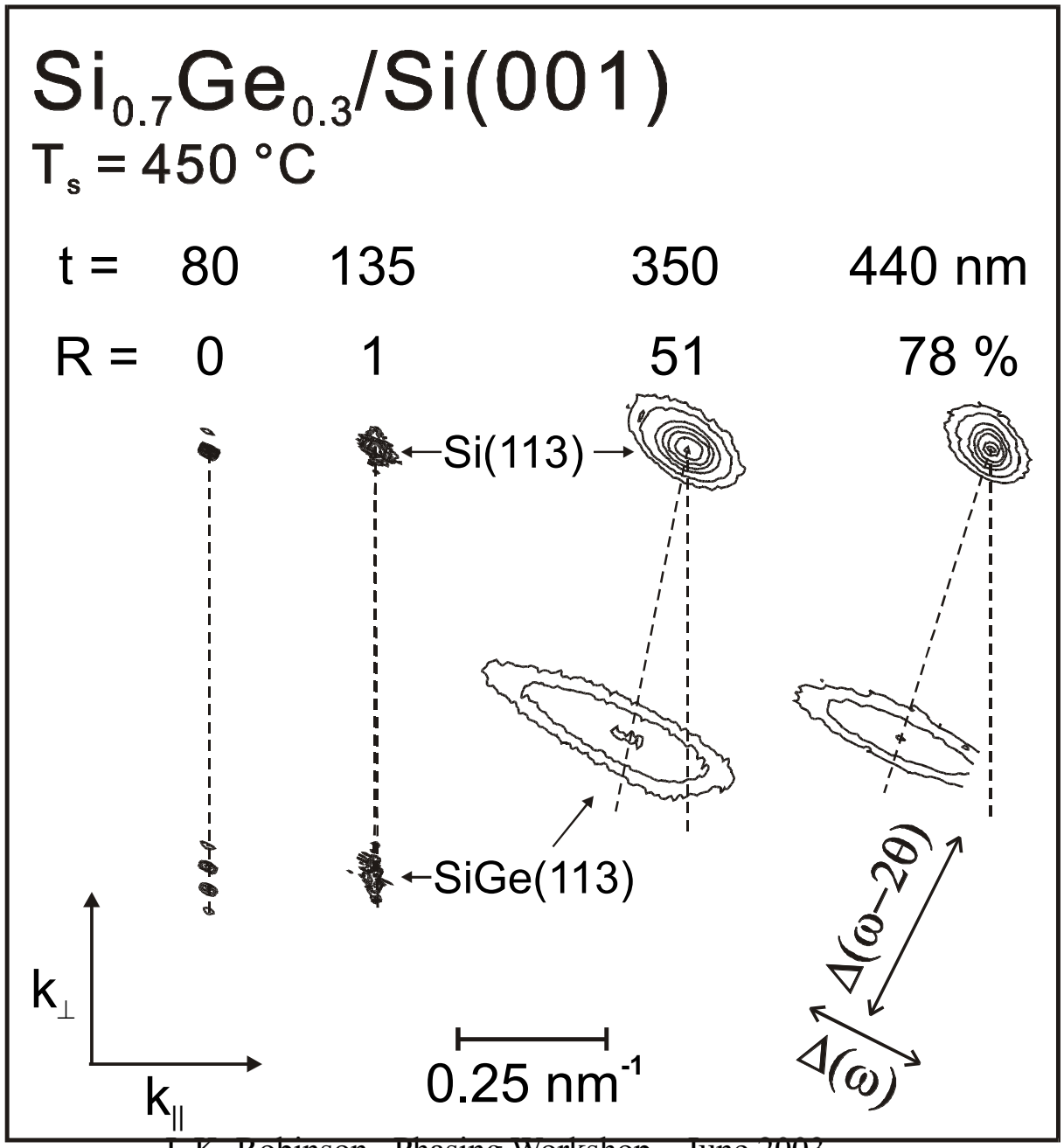
Low dislocation density GeSi films

Thickness close to critical thickness

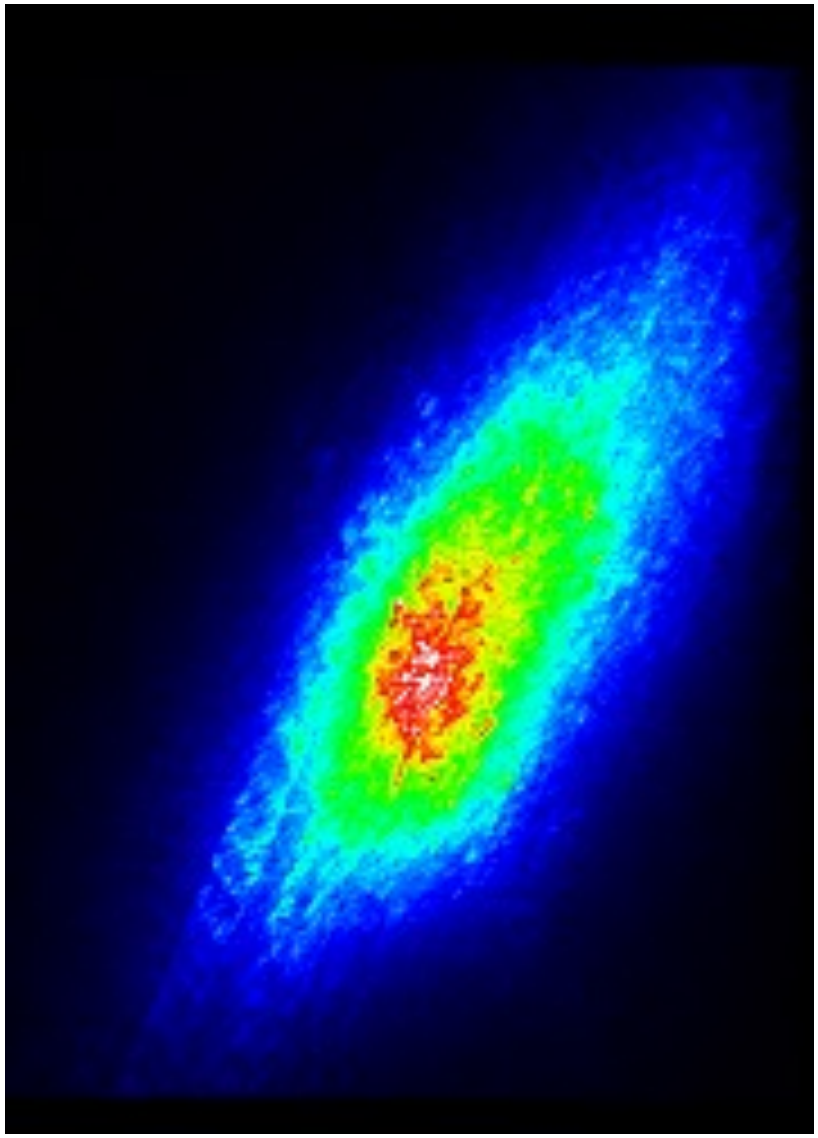
Dislocations aggregate at interface and glide to surface along {111}

T. Spila, UIUC Thesis




$Si_{0.7}Ge_{0.3}/Si(001)$
T_s = 450 °C
t = 80 135 350 440 nm
R = 0 1 51 78 %
Si(113)
SiGe(113)
$k_\perp$
$k_{||}$
0.25 nm^{-1}
$\Delta(\omega-2\theta)$
$\Delta(\omega)$

Ge_xSi_{1-x} Film Diffraction



- 202 Bragg Peak
- 2800A film
- 2° incidence angle
- 8.5 keV
- 20μm ×40μm beam onto KB mirror
- 1μm ×1μm focus
- 0.5μm sample steps
- APS 34-ID-C

Conclusions and Outlook

- Inversion of CXD by ER-HIO methods
- Internal structure of Au Nanocrystals
- Preservation of coherence upon focussing
- Smallest size now down to 170nm
- New CXD-Yoneda geometry
- Dislocation strain structure may be possible