

Phasing of Three Dimensional Diffraction Patterns from Finite-Sized Objects

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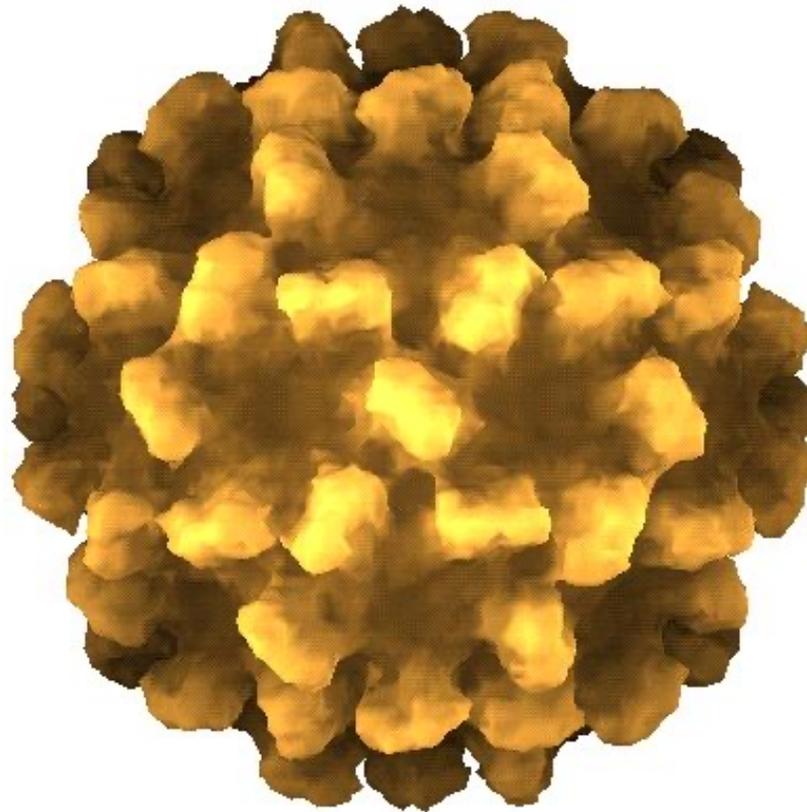
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Outline

- Coherence in Diffraction
- The **Phase** Problem
- Nanocrystal Shapes
- Crystallization of Proteins
- Applications at LCLS

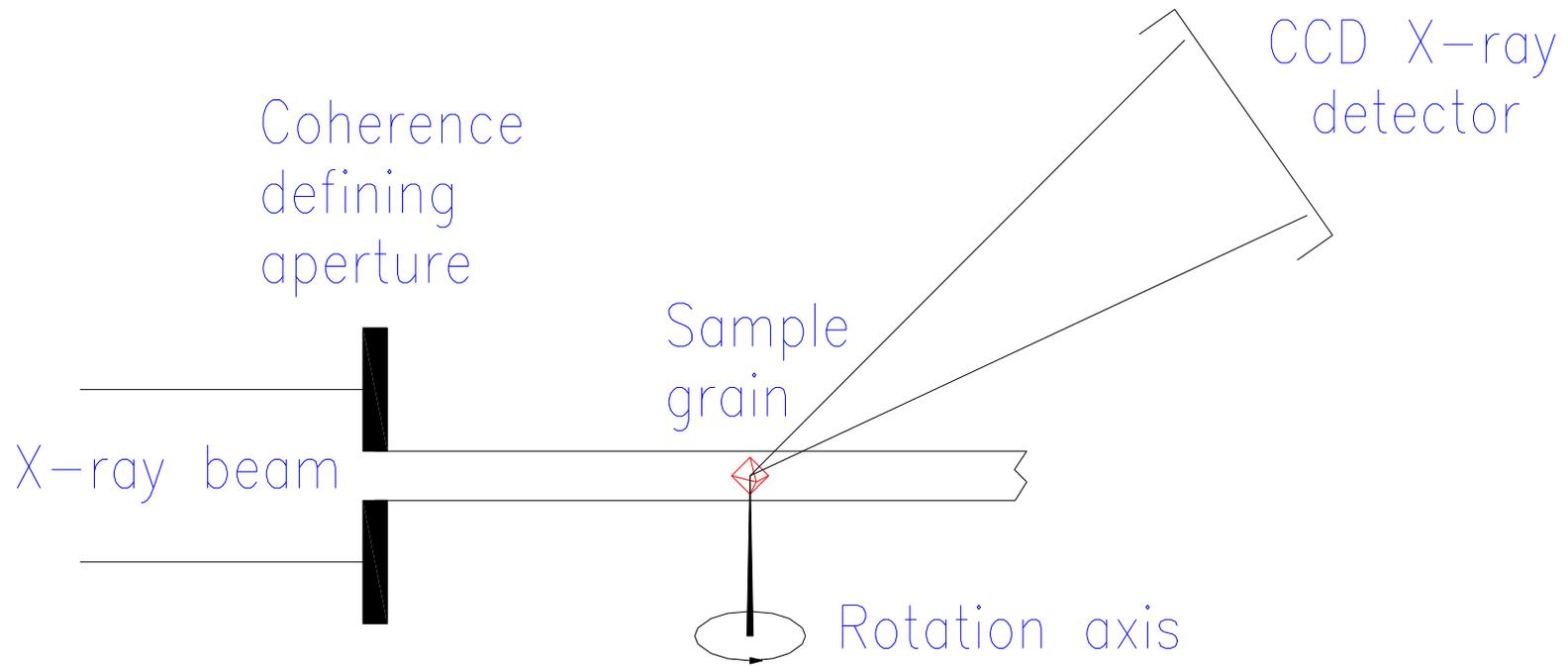
Tomato Bushy Stunt Virus 1980



Goals of Coherent Diffraction

- Thermodynamic fluctuations
 - No ensemble average in CXD
- Probe of structure on **nm** scale
 - 1D, 2D and 3D
 - non-periodic object gives **continuous** $F(\mathbf{q})$
- **Oversampling** (in reciprocal space) permits solution of the **phase** problem

Lensless X-ray Microscope based on diffraction from crystal lattice

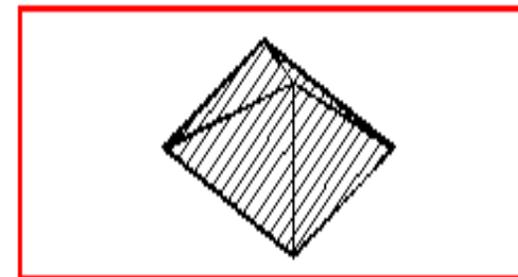
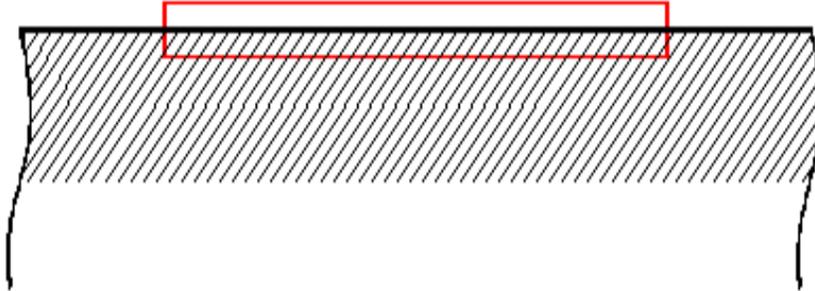


Coherence at the APS

Typical 3rd Generation (undulator) Synchrotron Source
LCLS coherence will be better

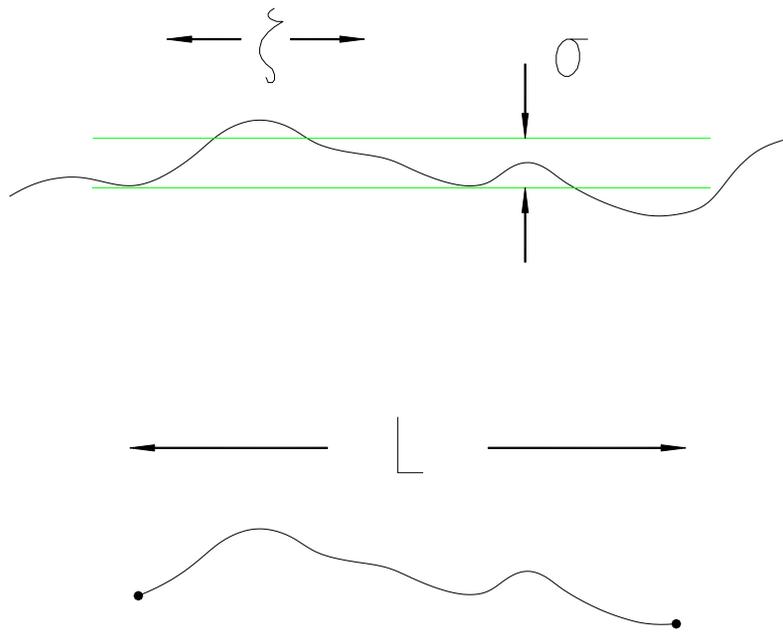
Coherence of	ξ_{VER}	ξ_{HORIZ}	ξ_{LONG}	Flux
Raw Undulator	35 μm	9 μm	0.004 μm	2×10^{12}
Si(111) Monochromator	35 μm	9 μm	1 μm	1×10^{10}
C(111) Monochromator	35 μm	9 μm	3 μm	3×10^9

Coherent region defined by slits

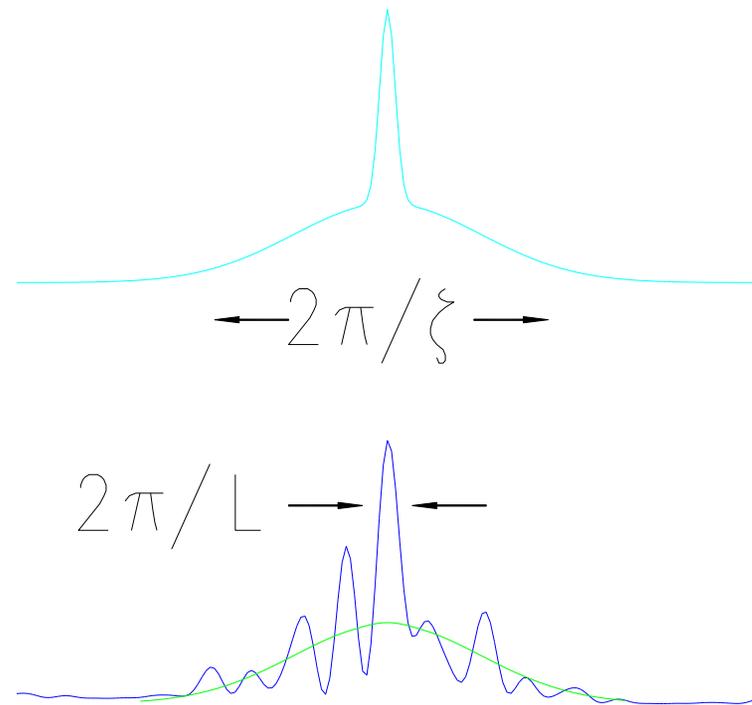


Diffuse Scattering acquires Structure using CXD

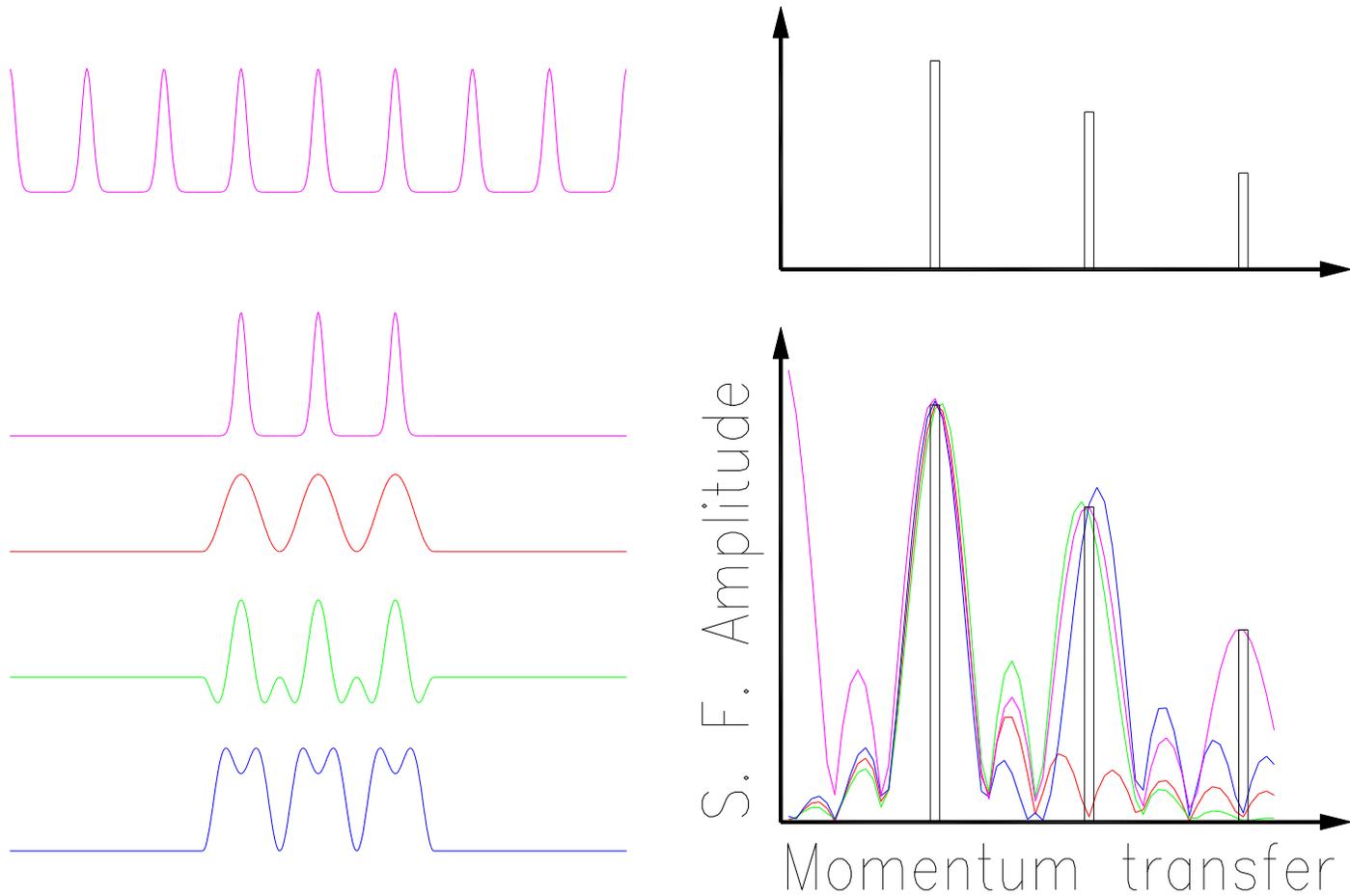
Real Space



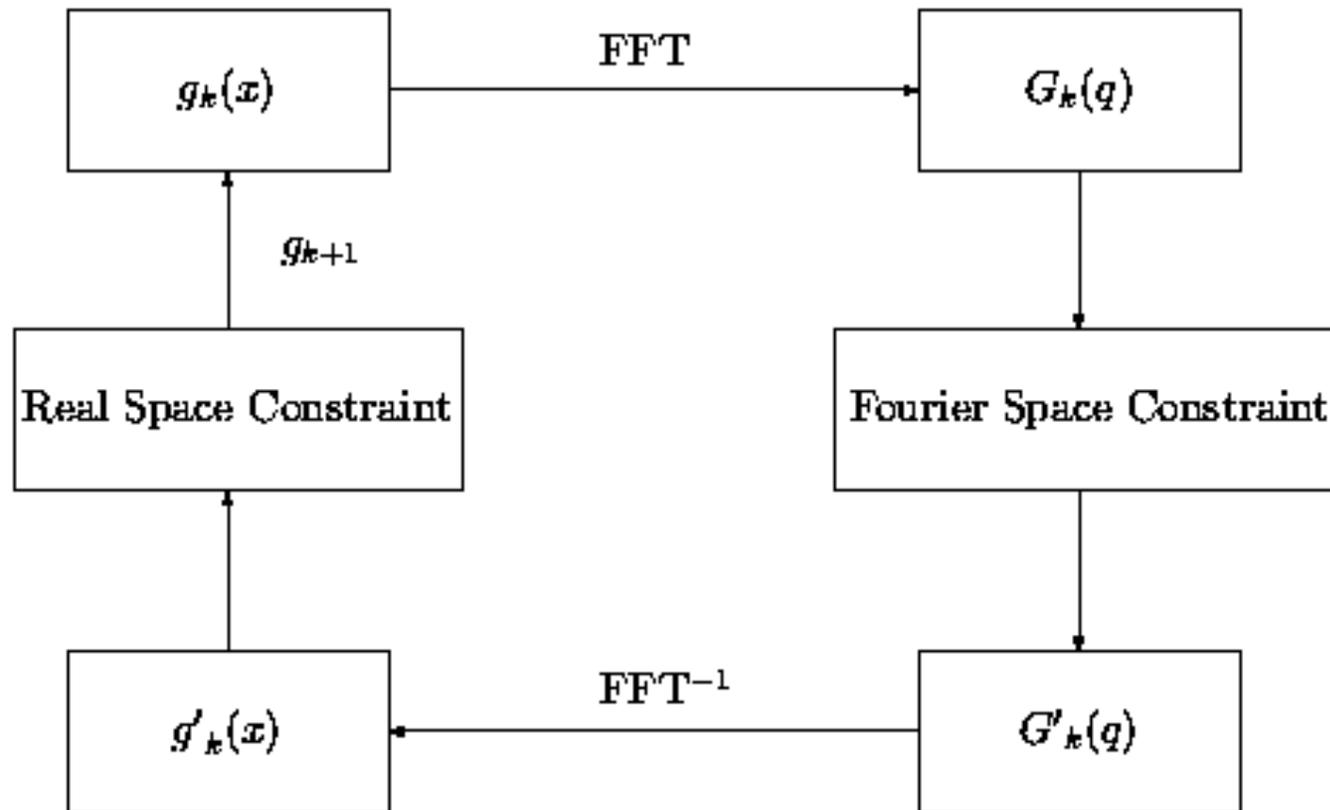
Reciprocal Space



Phase Problem: Finite-size Effect



Generic “Error Reduction” method



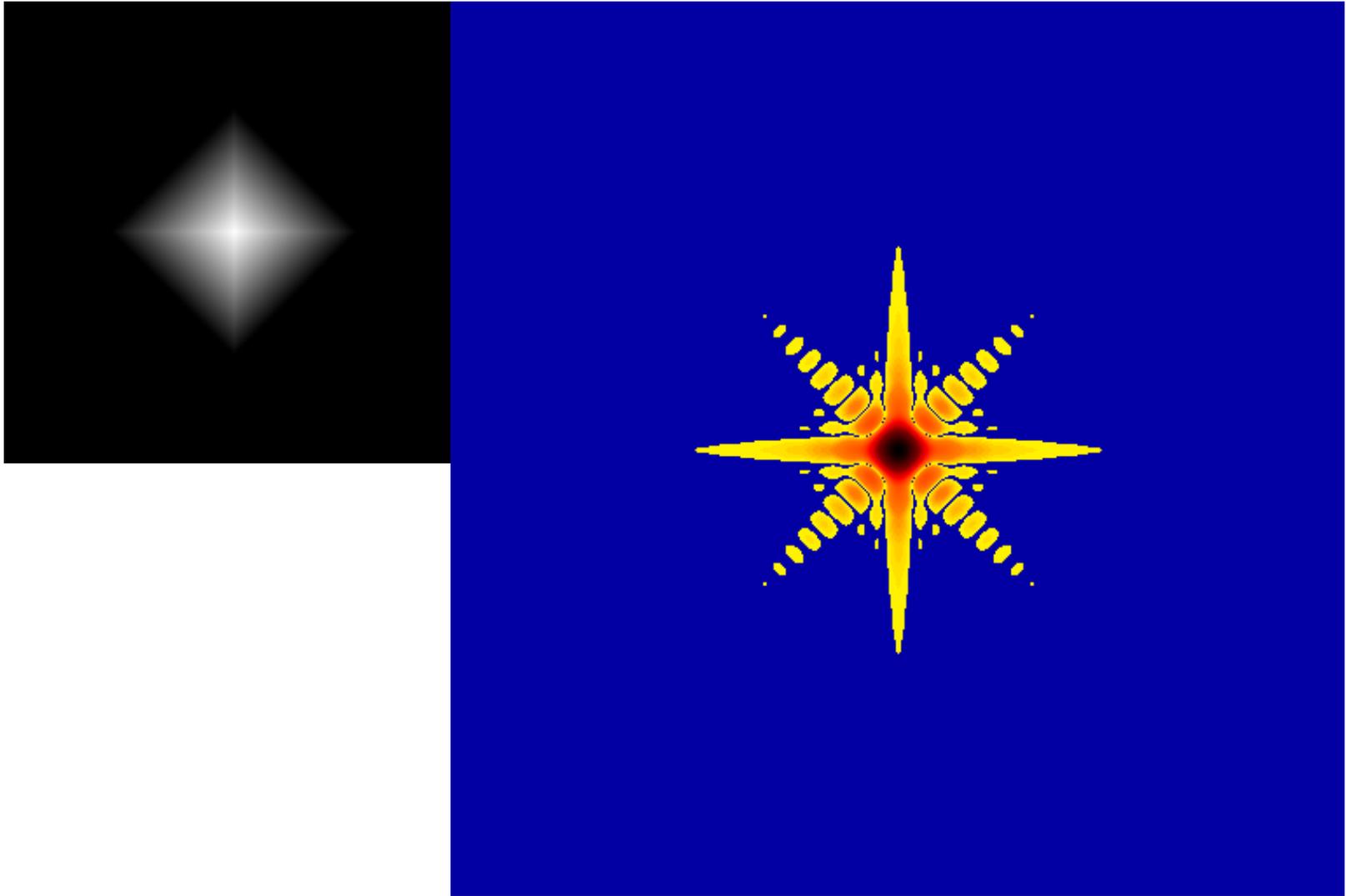
J. R. Fienup *Appl. Opt.* 21 2758 (1982)

R. W. Gerchberg and W. O. Saxton *Optik* 35 237 (1972)

“ER” Methods in Crystallography

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

- ‘Positivity’ constraint
- Finite **support**, molecular envelope
- Solvent flattening
- Molecular replacement
- Non-crystallographic symmetry
- Basis of ‘direct methods’ (Sayre, Brice)
- Non-uniqueness is ‘pathologically rare’ ($d > 1$)



Diffraction as a Surface Integral

**Die äußere Form der Kristalle
in ihrem Einfluß auf die Interferenzerscheinungen
an Raumgittern**

Von M. v. Laue

Annalen der Physik [5] 26 55 (1936)

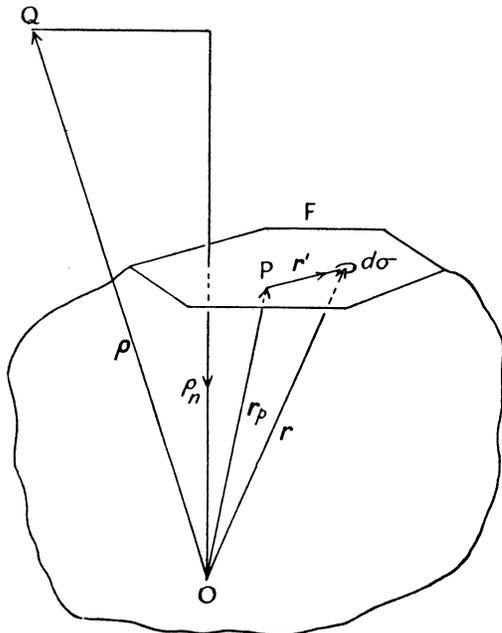
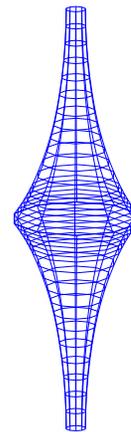
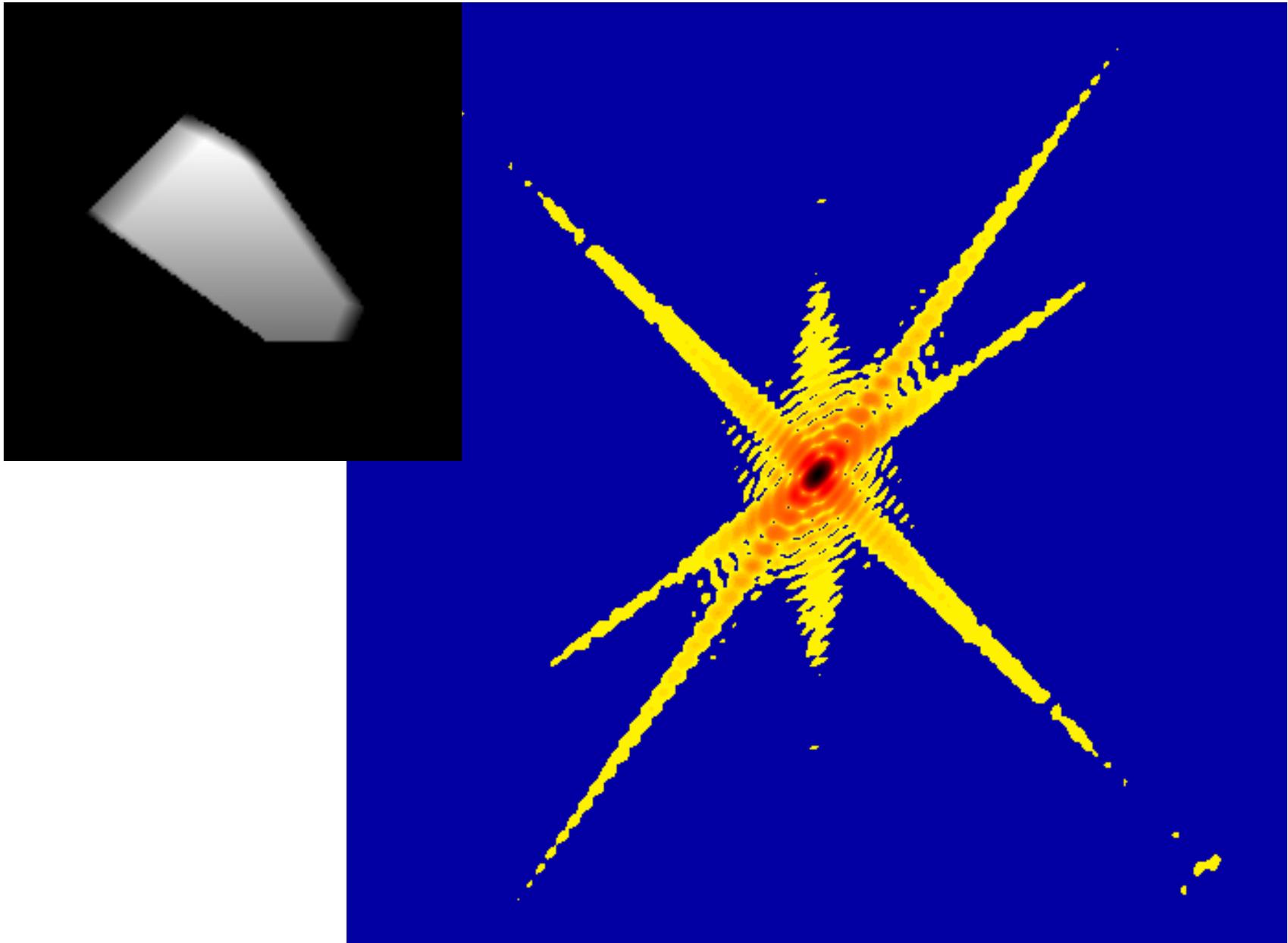


FIG. 200

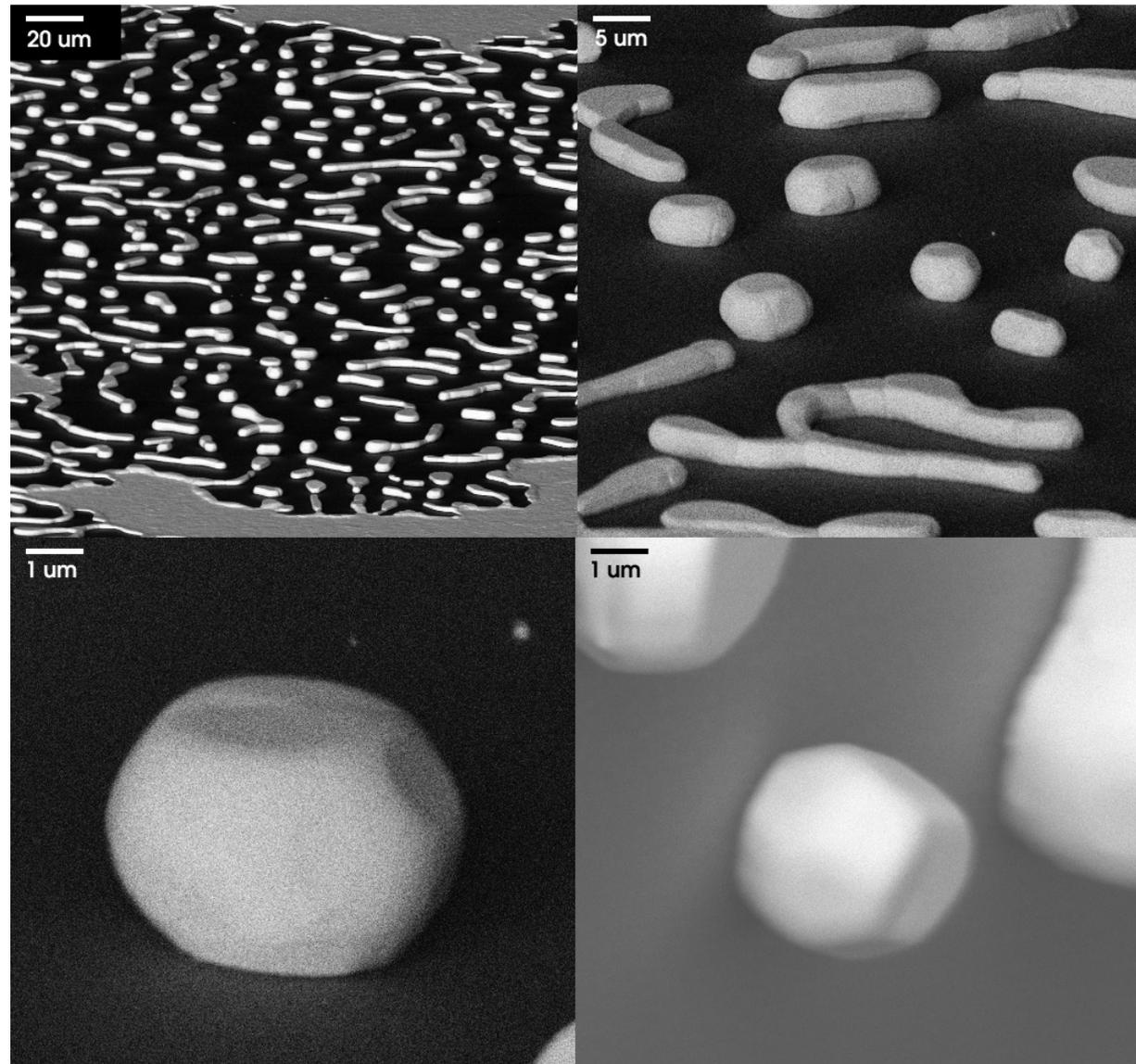


“Stacheln”

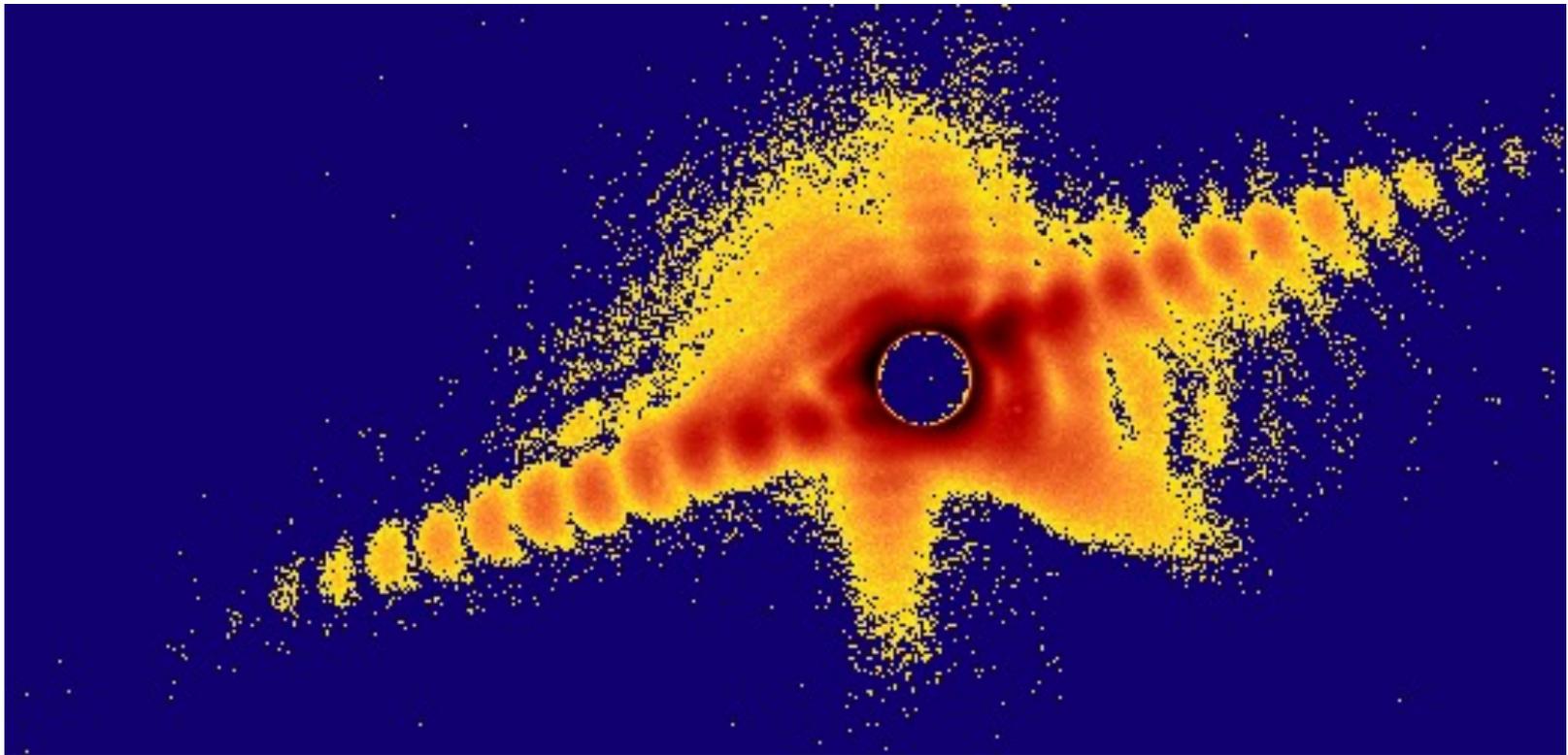


SEMS

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

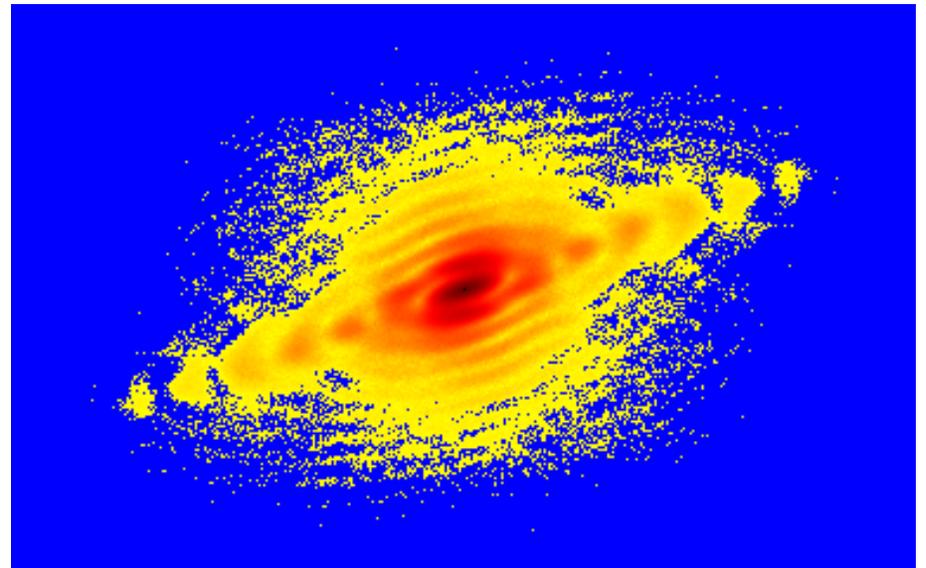
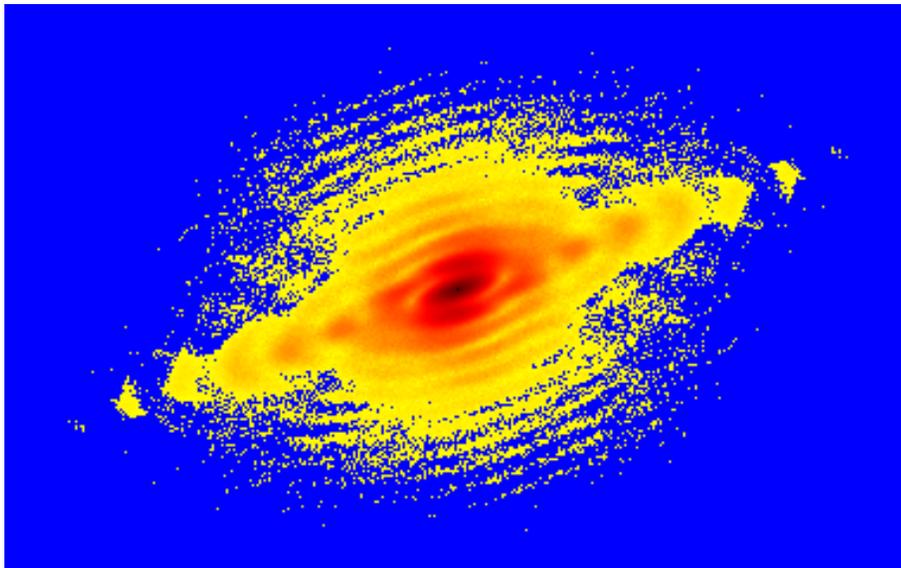
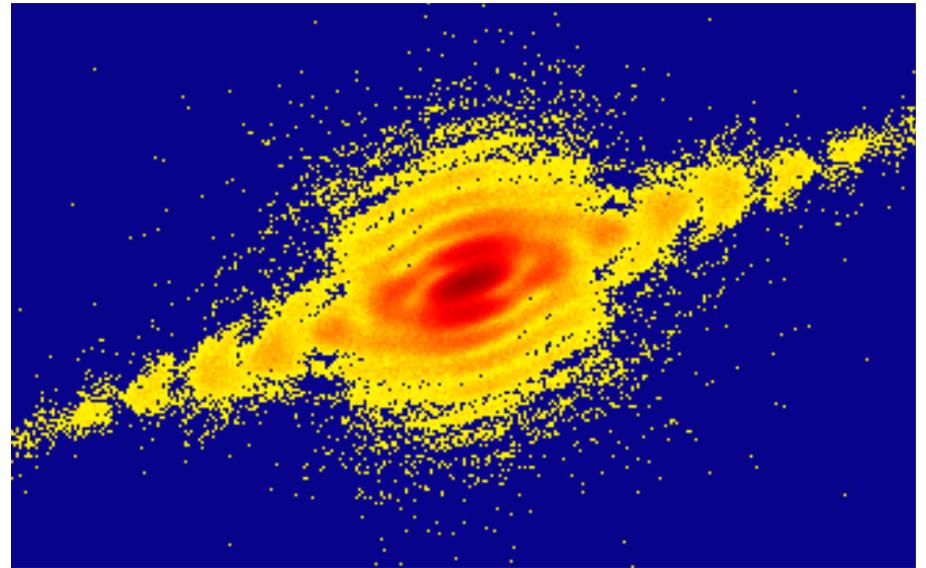


Micron-sized gold crystal: (111) Bragg reflection



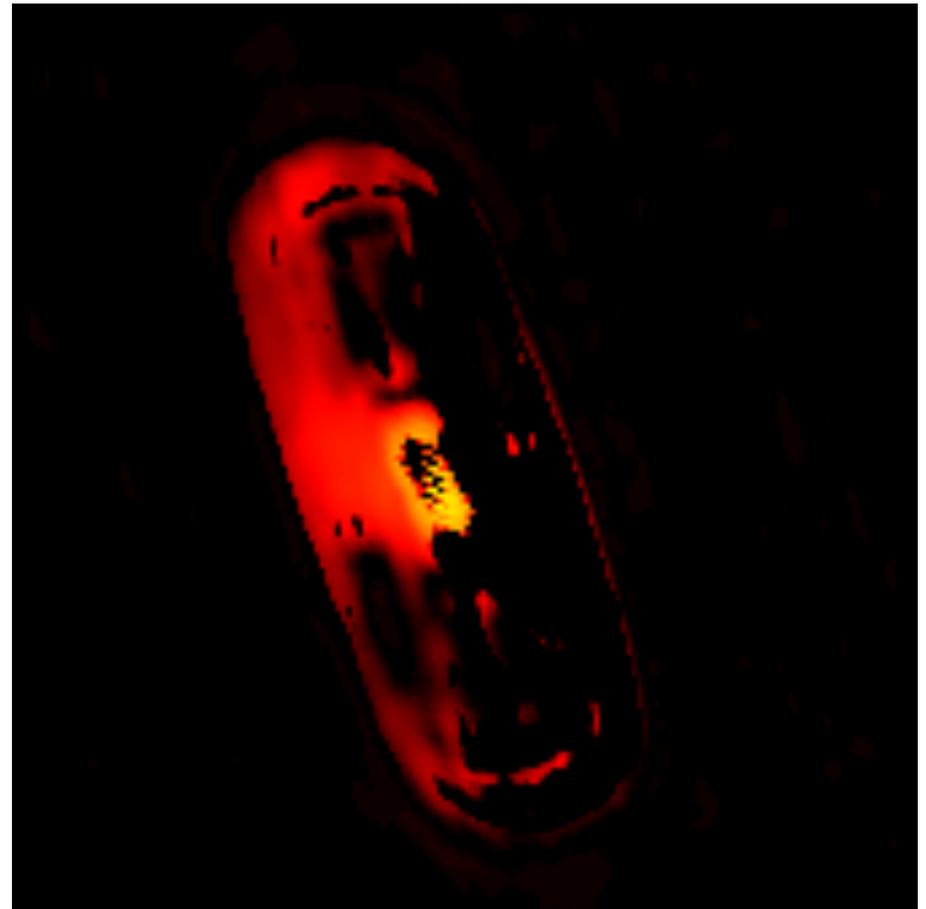
Symmetrized Data and two best fits

Chisq=0.0005

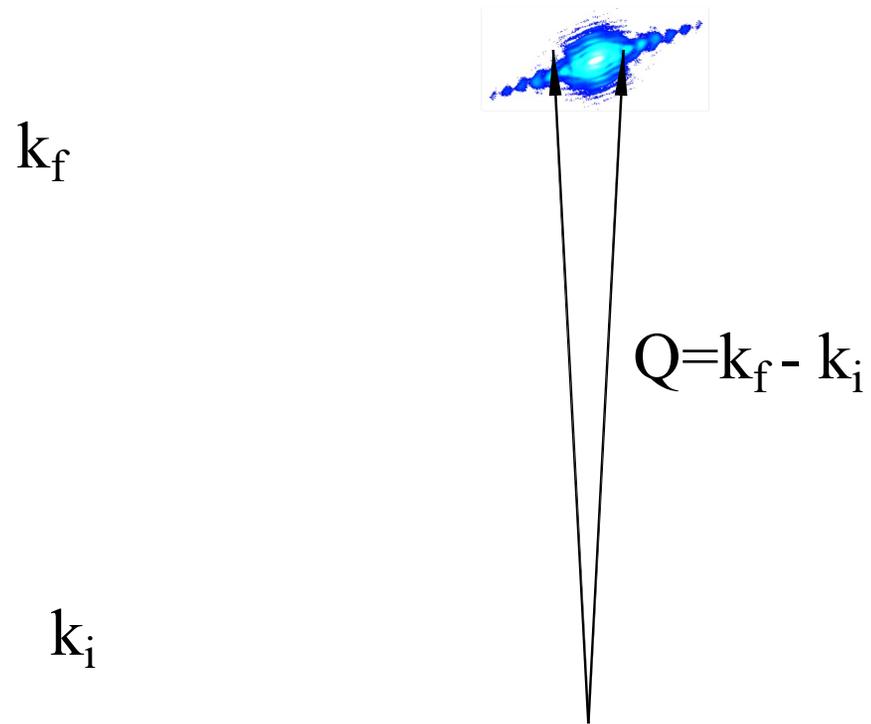


2D Reconstructions

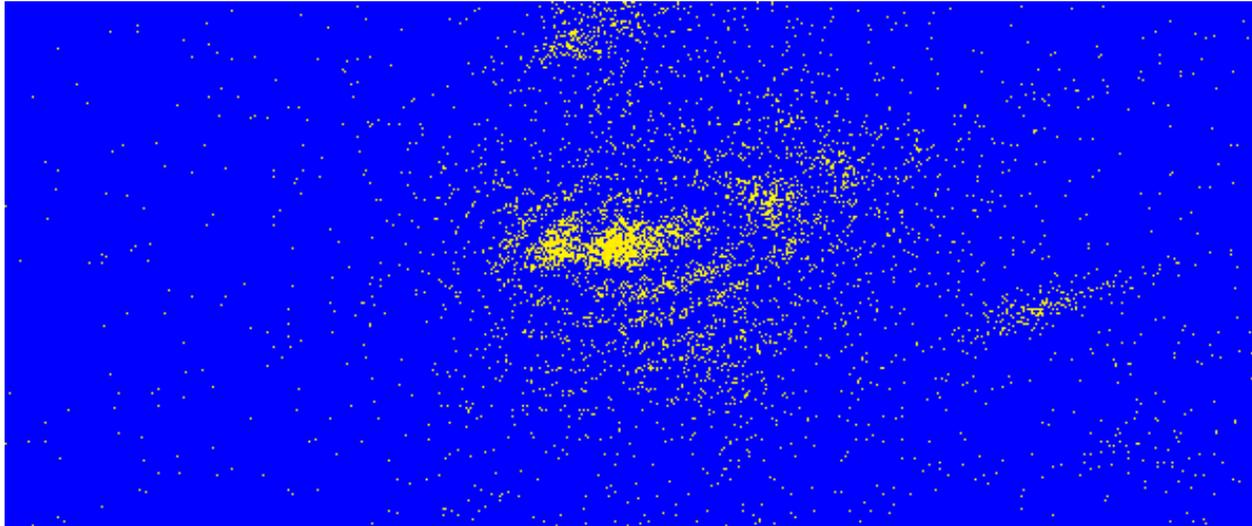
chisquare = 0.0005



3D Diffraction Method

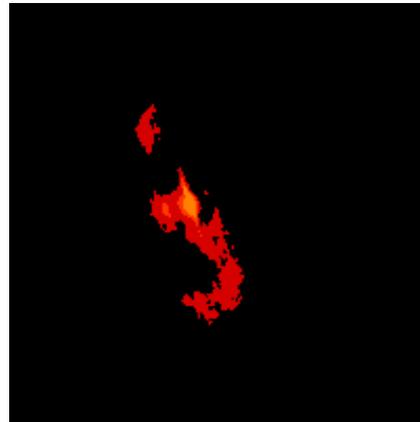


3D Diffraction Data 1 micron Au crystal

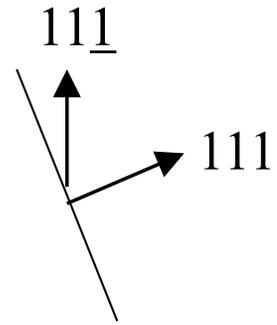
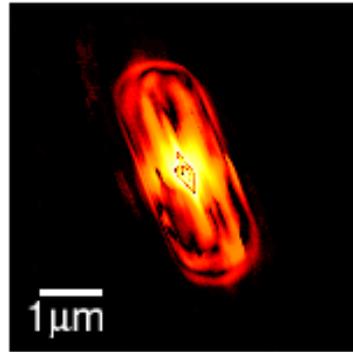
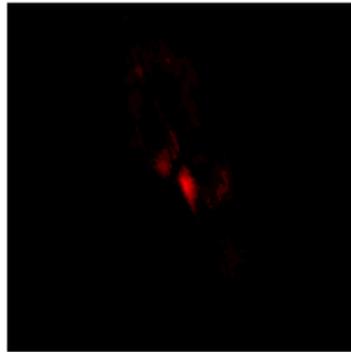


* Center is Symmetric *

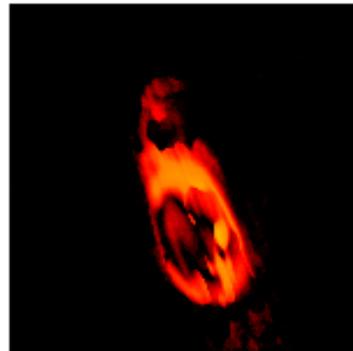
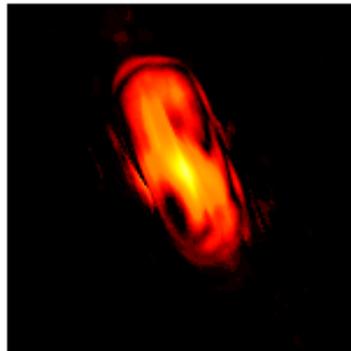
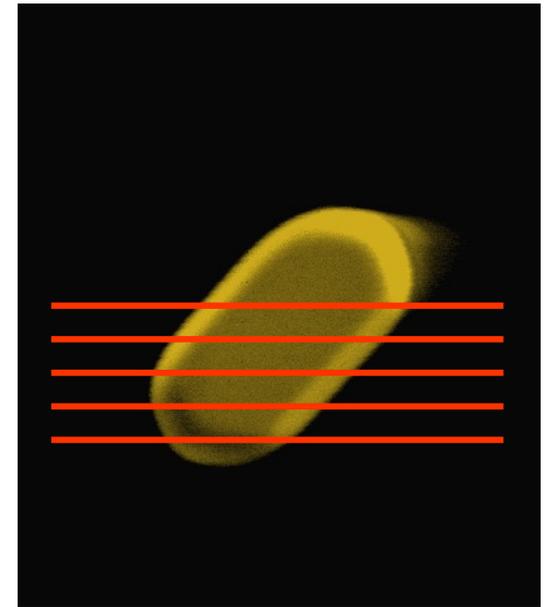
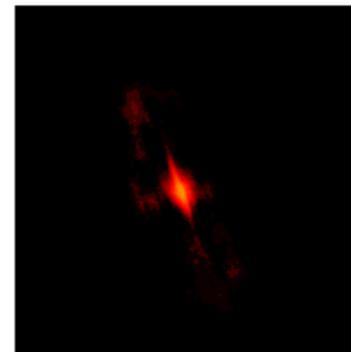
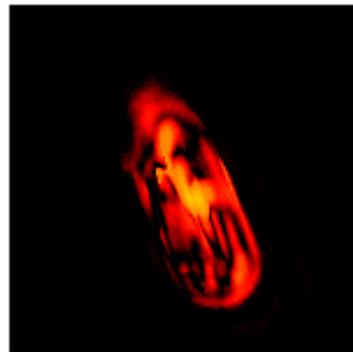
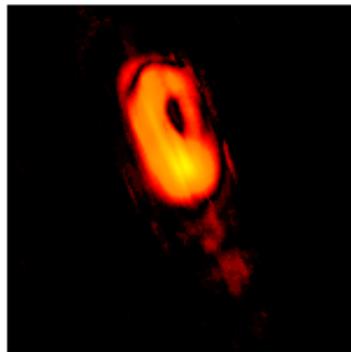
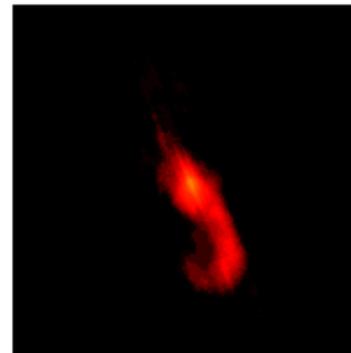
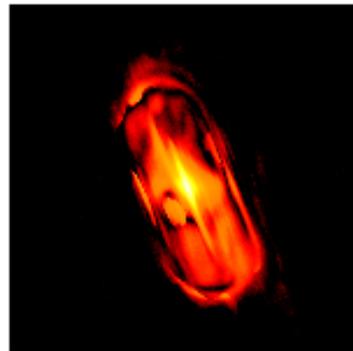
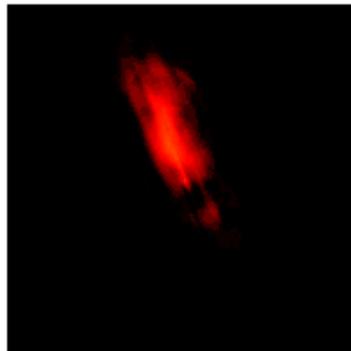
30 frames of 3D reconstruction 1 micron gold crystal



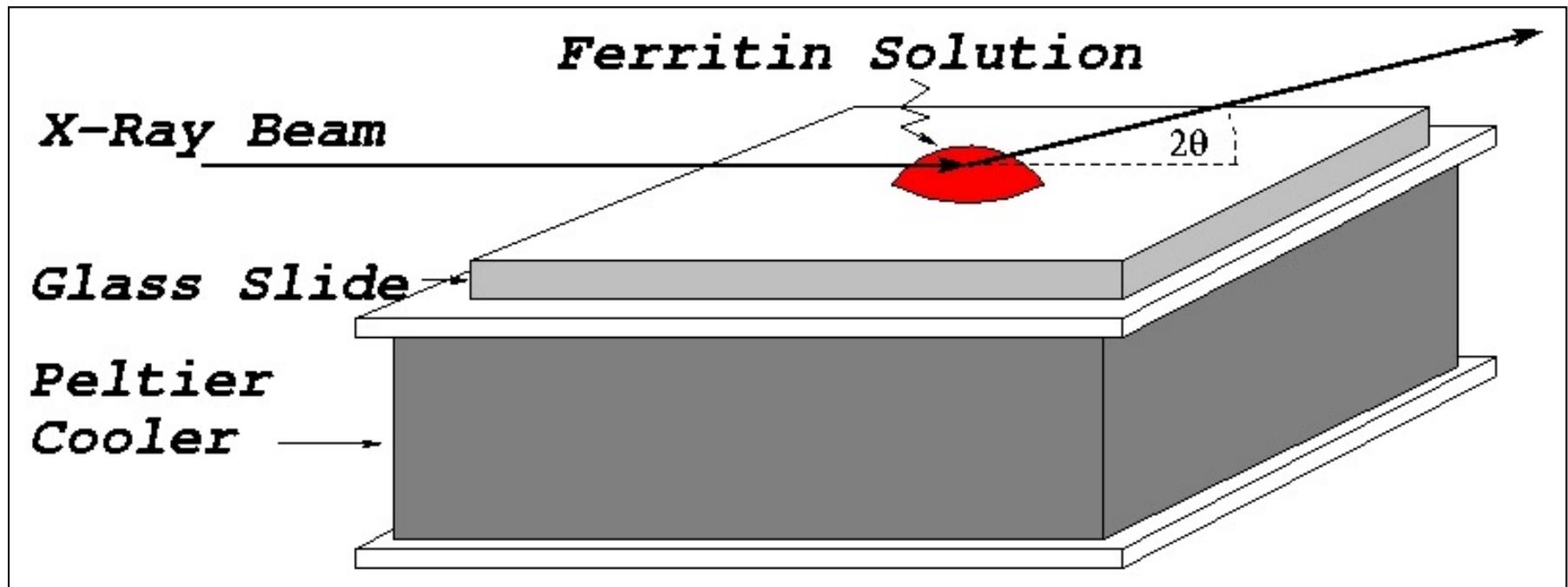
* CENTER *



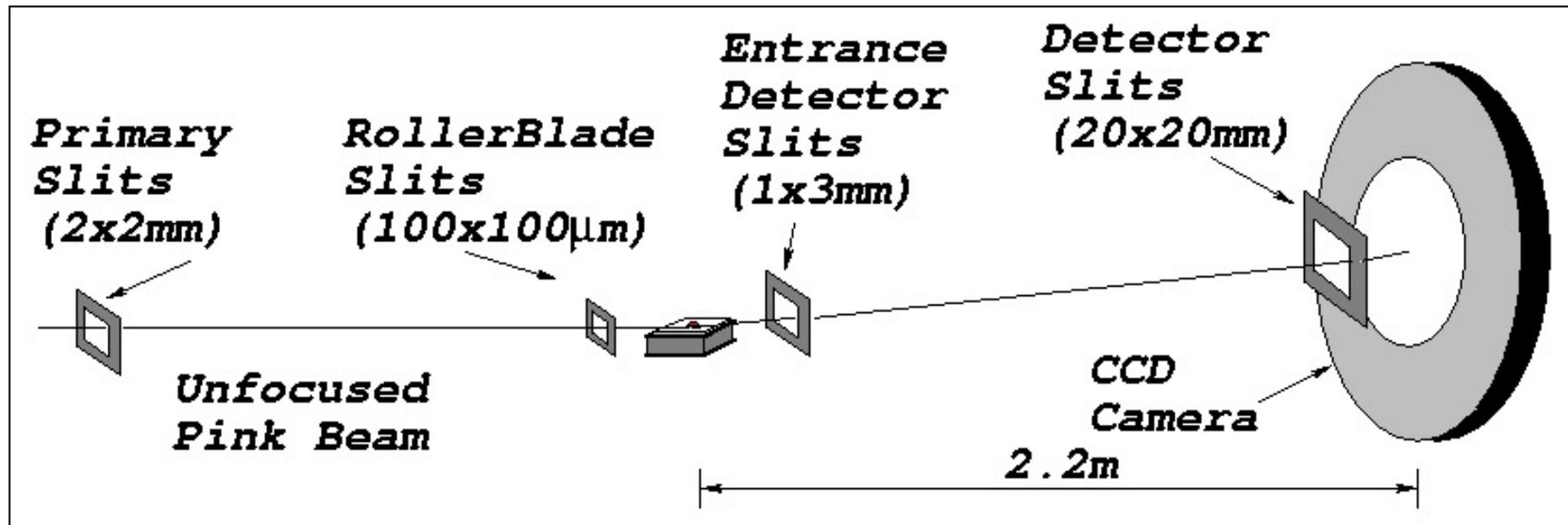
Slices through
plan view SEM:



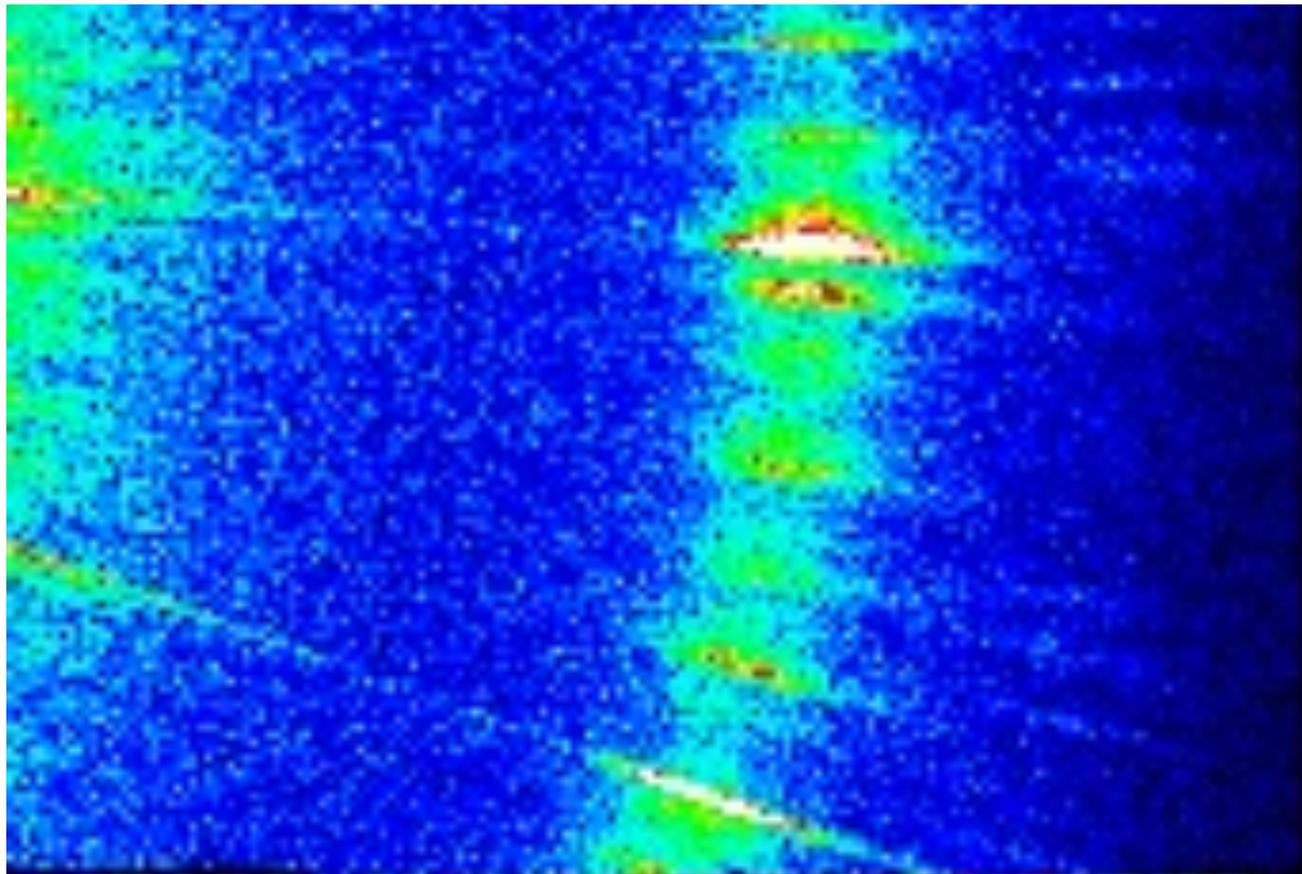
In-situ Study of Crystallization



Experiment at APS Sector 34

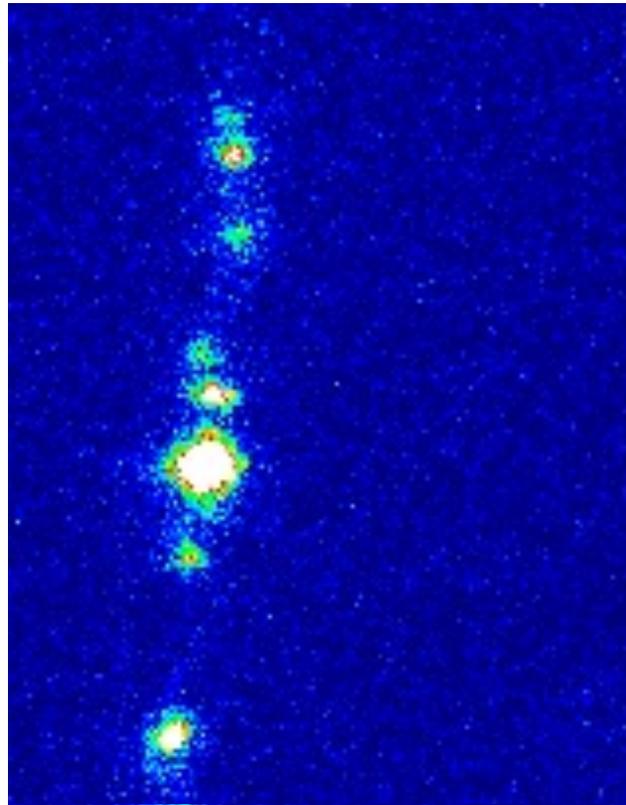


“Pink beam” sees CTRs



Ferritin (111) Powder Ring

- 50 frames
- 30sec exposure
- 0.3sec playback
- 150x200 pixels
of 22.5 μm



Conclusions and Outlook

- “Diffuse” scattering acquires fine structure
- Phase problem solved by oversampling
- Images of small particles in 3D
- Applications of CXD at LCLS
 - (physics) fluctuations at the atomic level
 - (biology) imaging of viruses and proteins