

# Protein Crystals on the Nanometer Scale

- Ian Robinson
- Sébastien Boutet

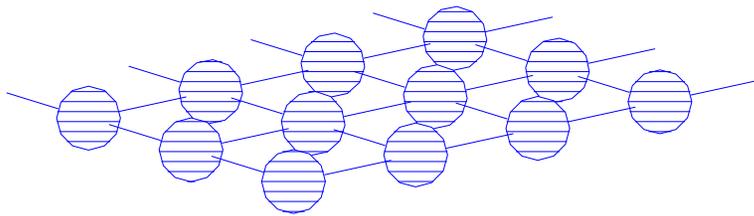
Department of Physics  
University of Illinois

ANL Life Sciences Division  
21 March 2002

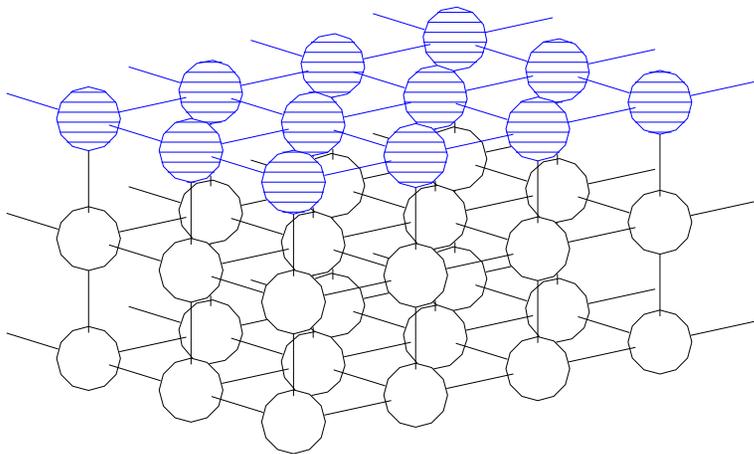
# Outline

- Crystal Truncation Rods
- Surface studies of Protein Crystals
- Coherent X-ray Diffraction
- **The Phase Problem can be solved!**
- Nanocrystal Shapes
- Lensless X-ray Microscope
- Applications to Protein Crystallization

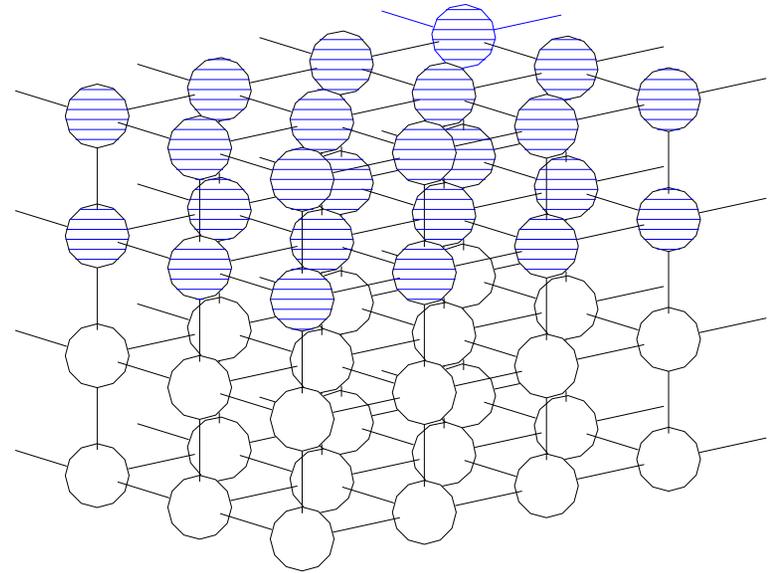
# Surfaces and Interfaces



Isolated monolayer

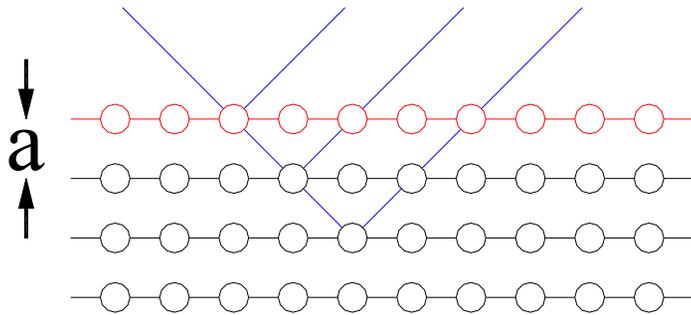


Surface of Crystal

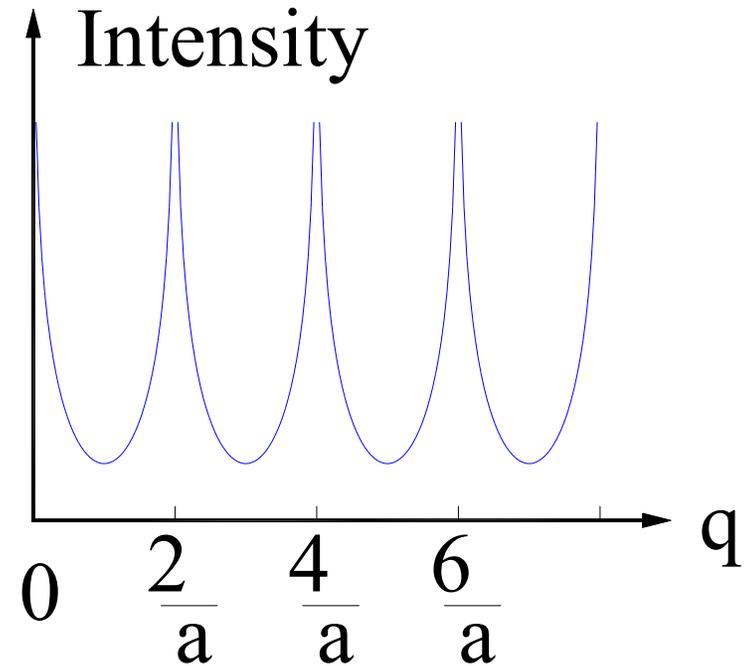


Crystal-Crystal Interface

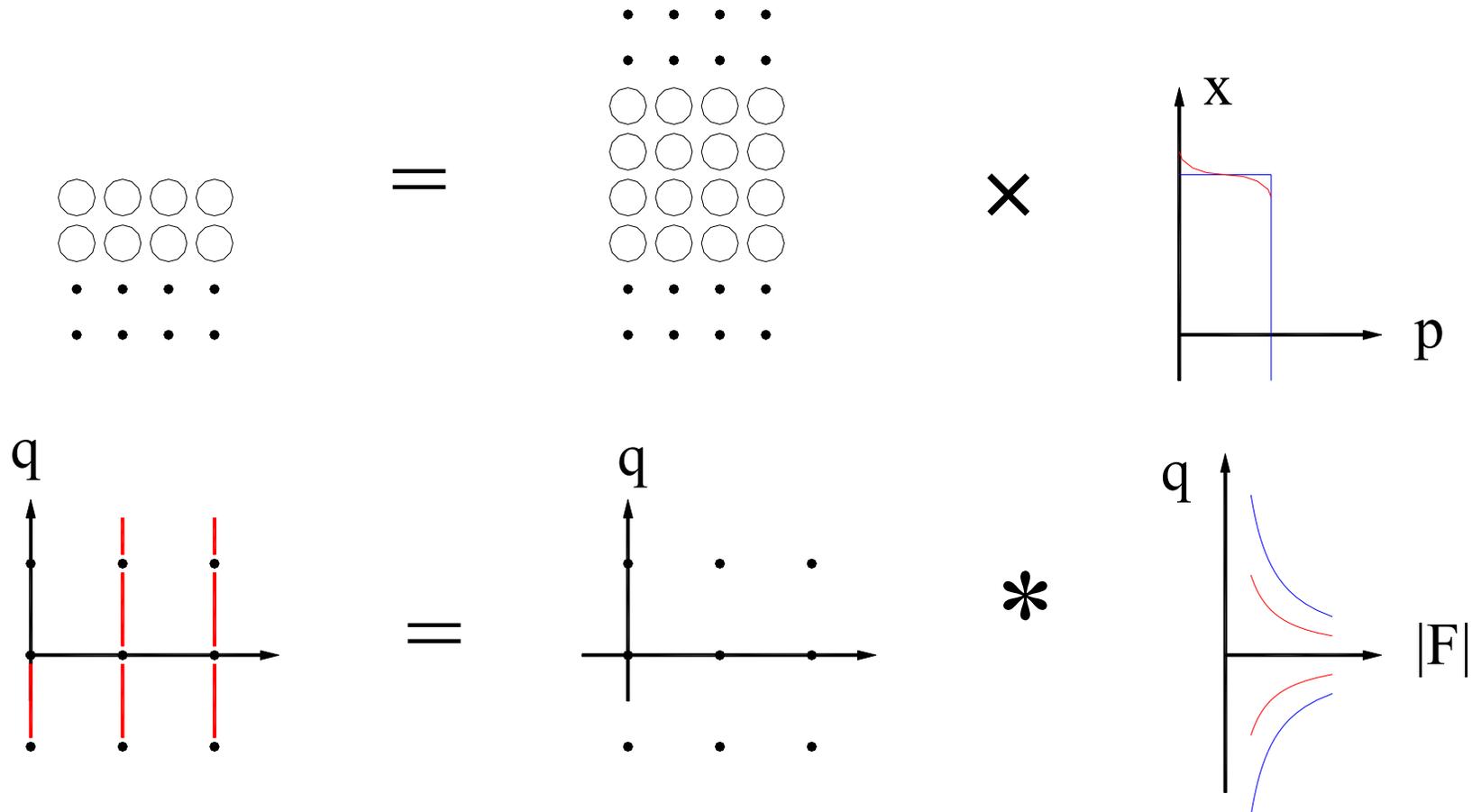
# Origin of Truncation Rods



$$\begin{aligned} F_{CTR} &= \sum_{n=0}^{\infty} A_n \\ &= \sum_{n=0}^{\infty} f_L e^{inqa} \\ &= \frac{f_L}{1 - e^{iqa}} \end{aligned}$$



# CTR as Convolution



# 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)

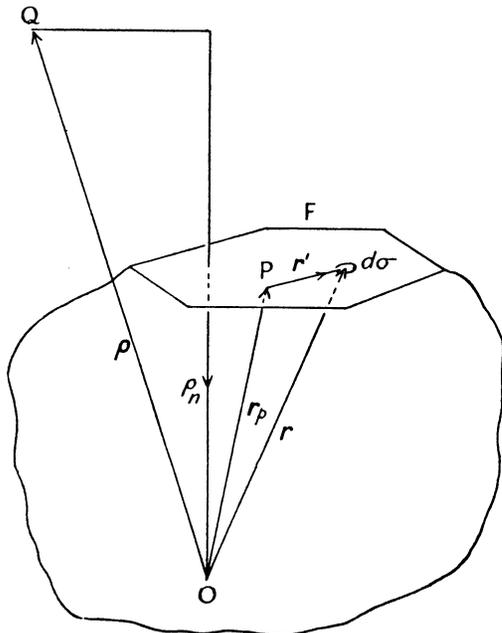
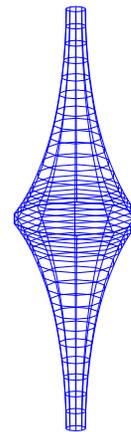
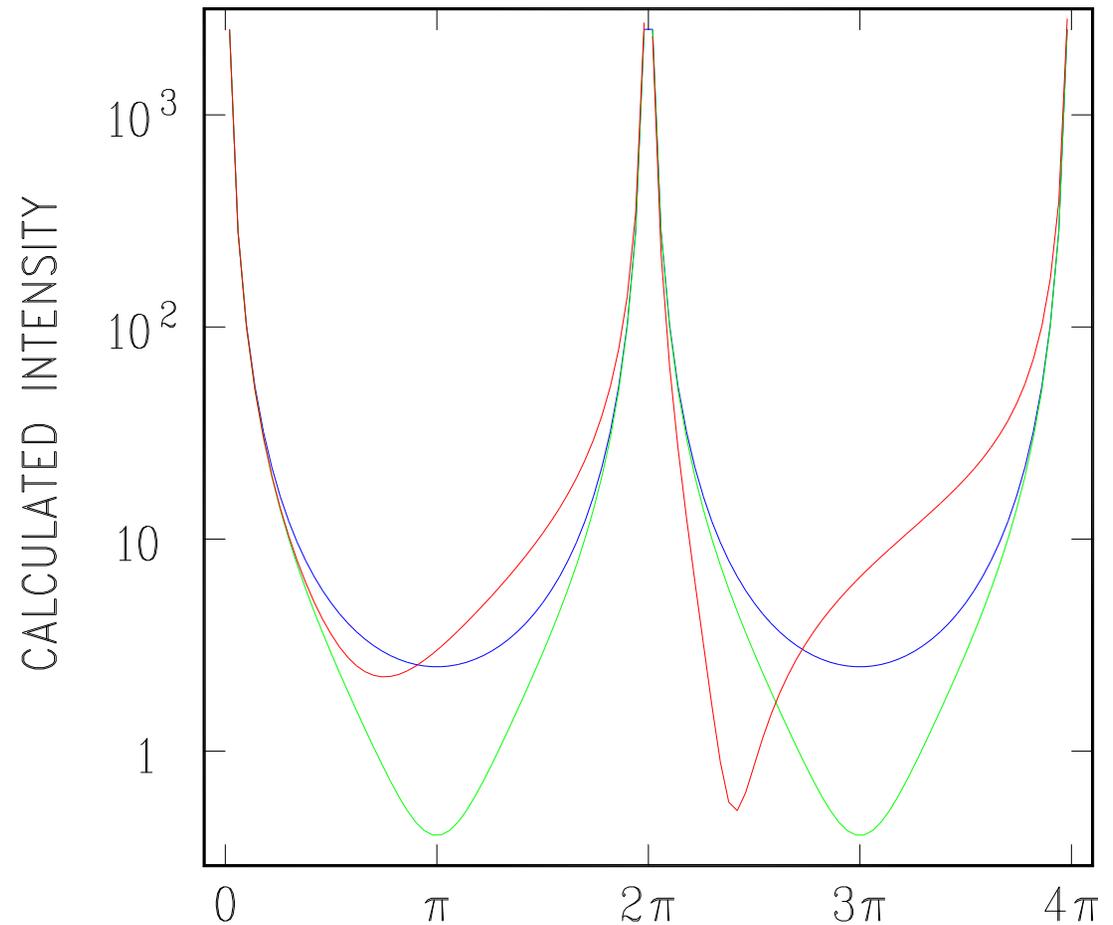
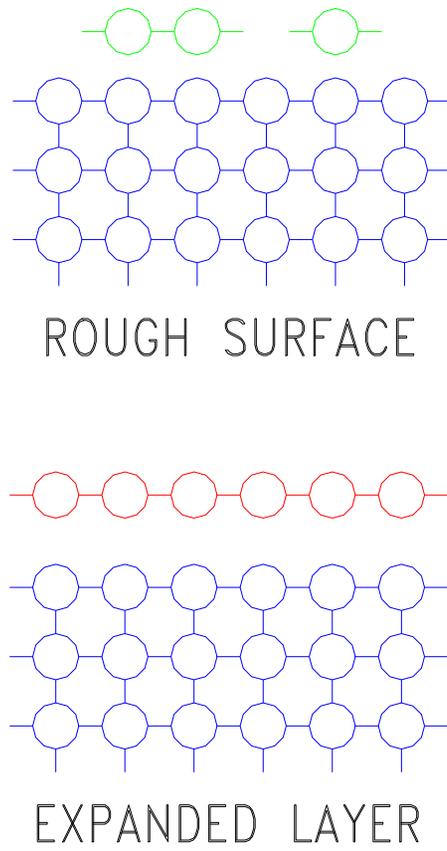


FIG. 200

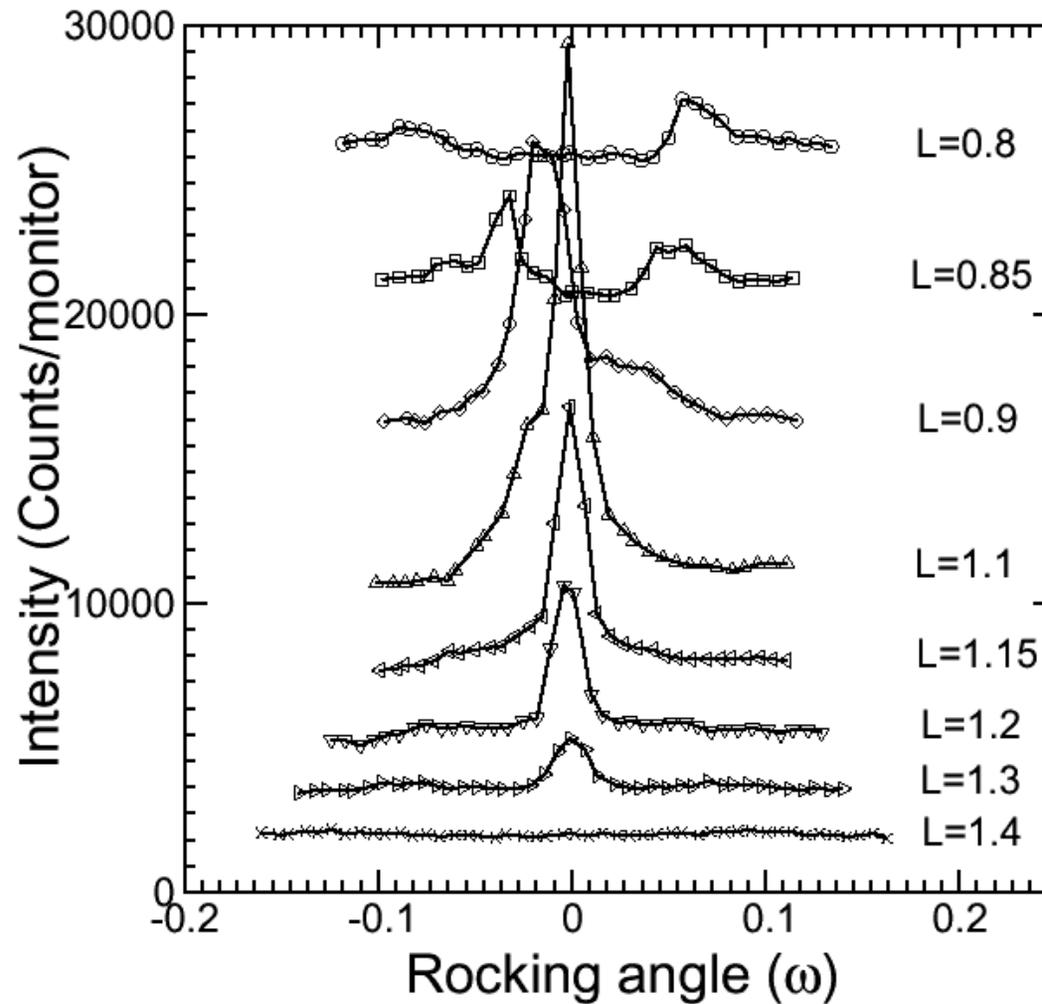


“Stacheln”

# CTR is Sensitive to Surface Structure

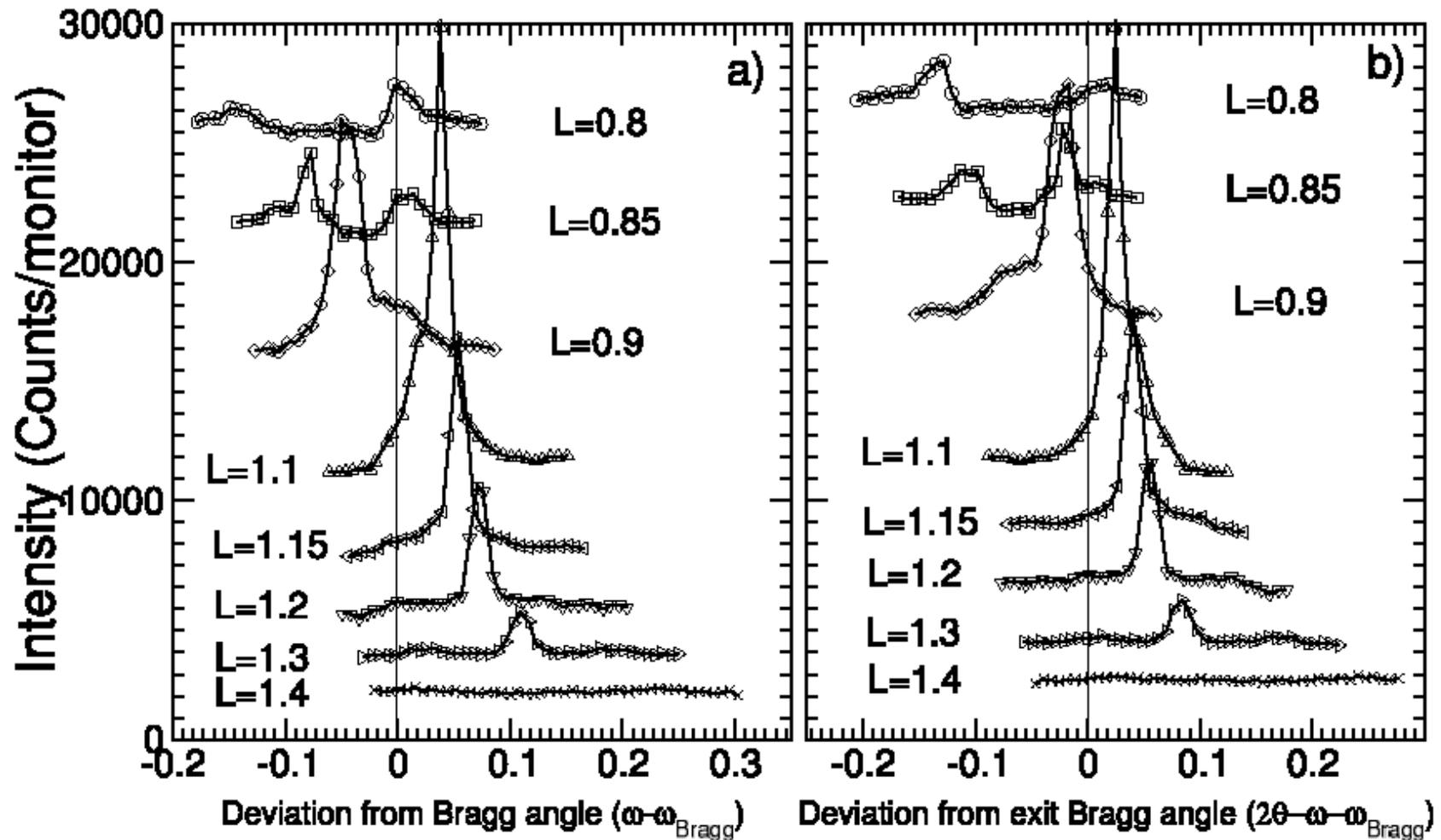


# CTR of Ferritin Crystals

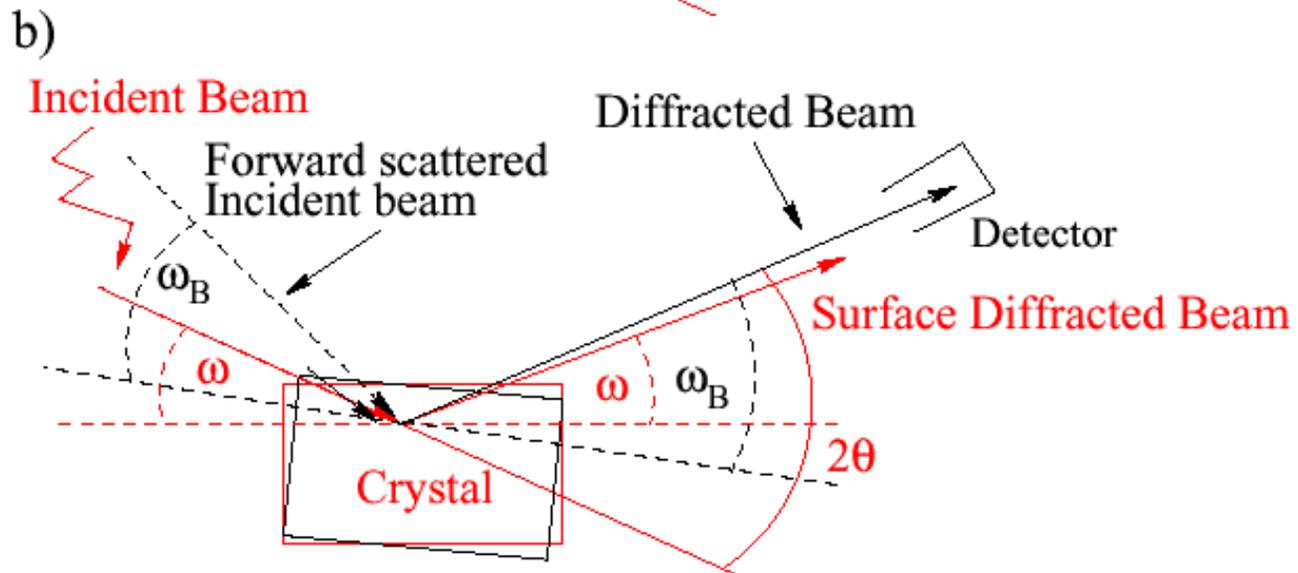
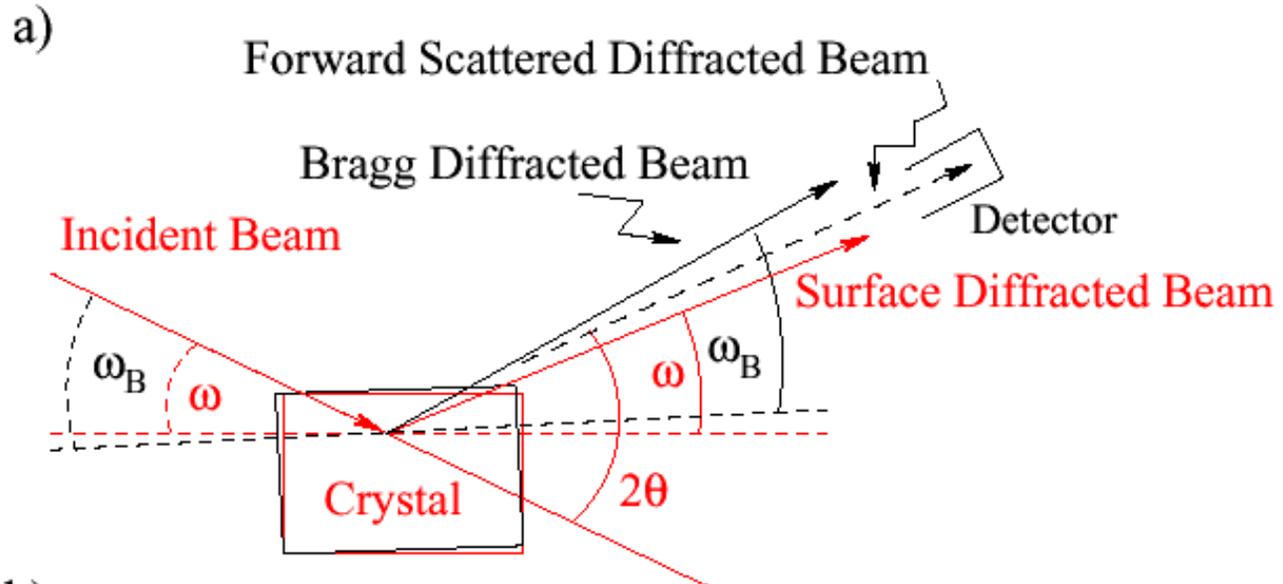


In collaboration  
with:  
A. Chernov  
Z. Hu  
B. R. Thomas

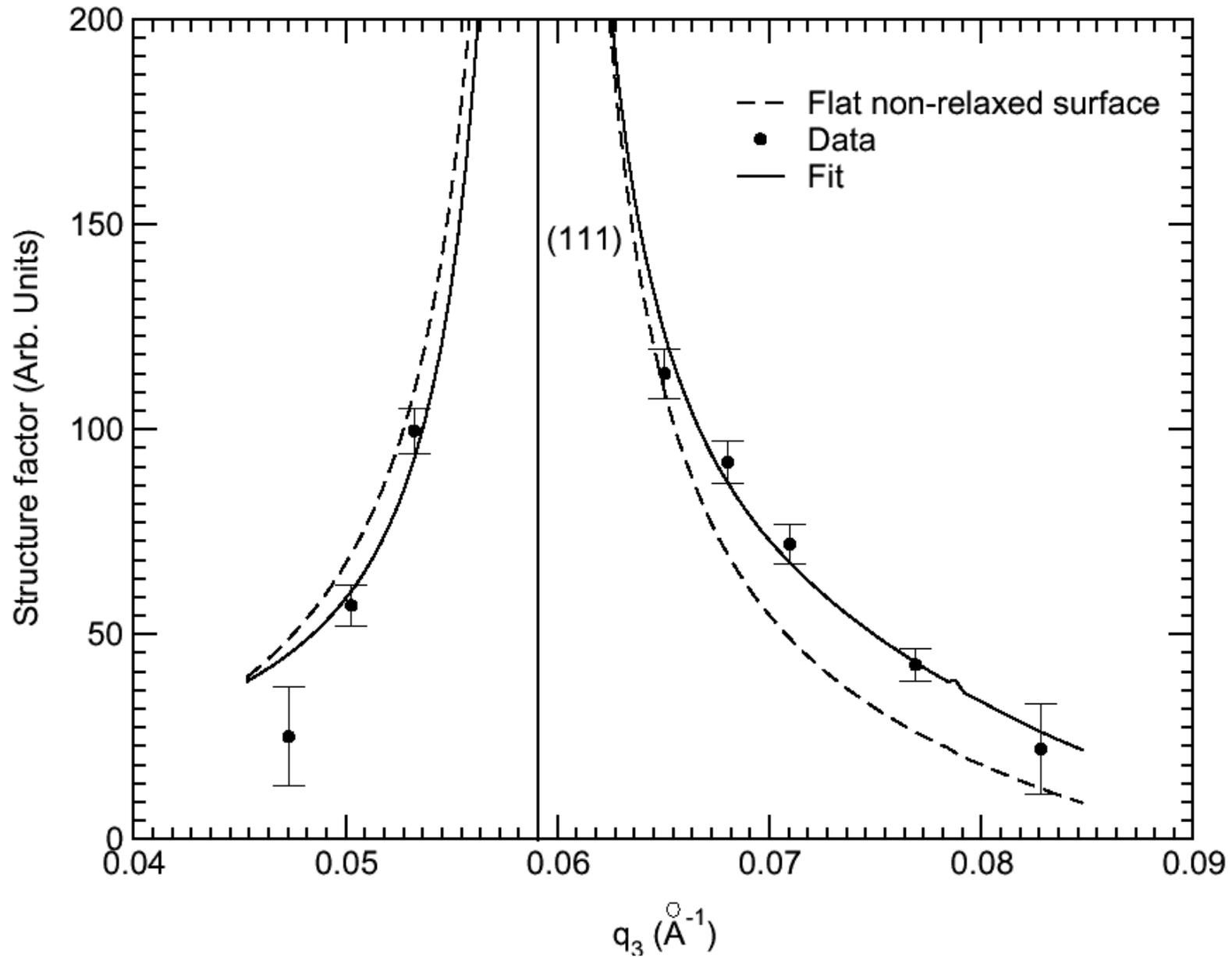
# Replotted with Angle Offset



# Origin of Extra Peaks

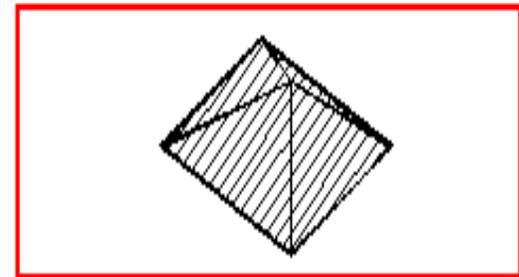
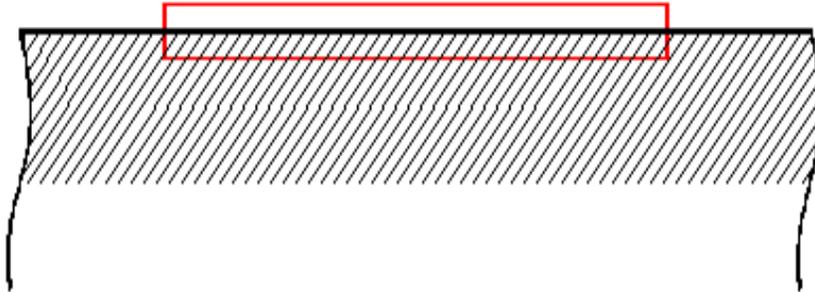


# (111) Surface is Contracted 3.9%

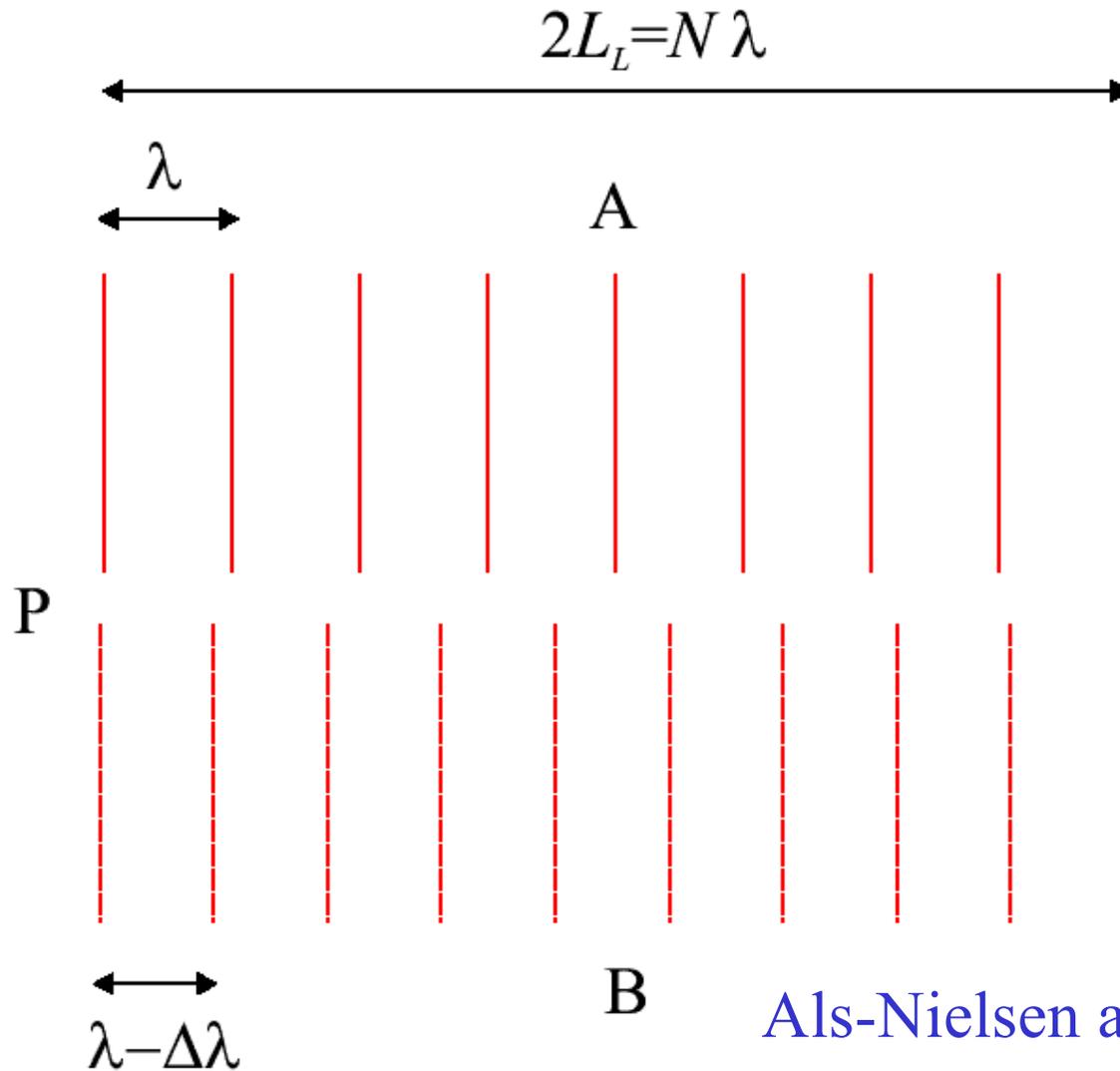


# Coherence at the APS

Coherence of	$\xi_{\text{VER}}$	$\xi_{\text{HORIZ}}$	$\xi_{\text{LONG}}$	Flux
Raw Undulator	35 $\mu\text{m}$	9 $\mu\text{m}$	0.004 $\mu\text{m}$	$2 \times 10^{12}$
Si(111) Monochromator	35 $\mu\text{m}$	9 $\mu\text{m}$	1 $\mu\text{m}$	$1 \times 10^{10}$
C(111) Monochromator	35 $\mu\text{m}$	9 $\mu\text{m}$	3 $\mu\text{m}$	$3 \times 10^9$



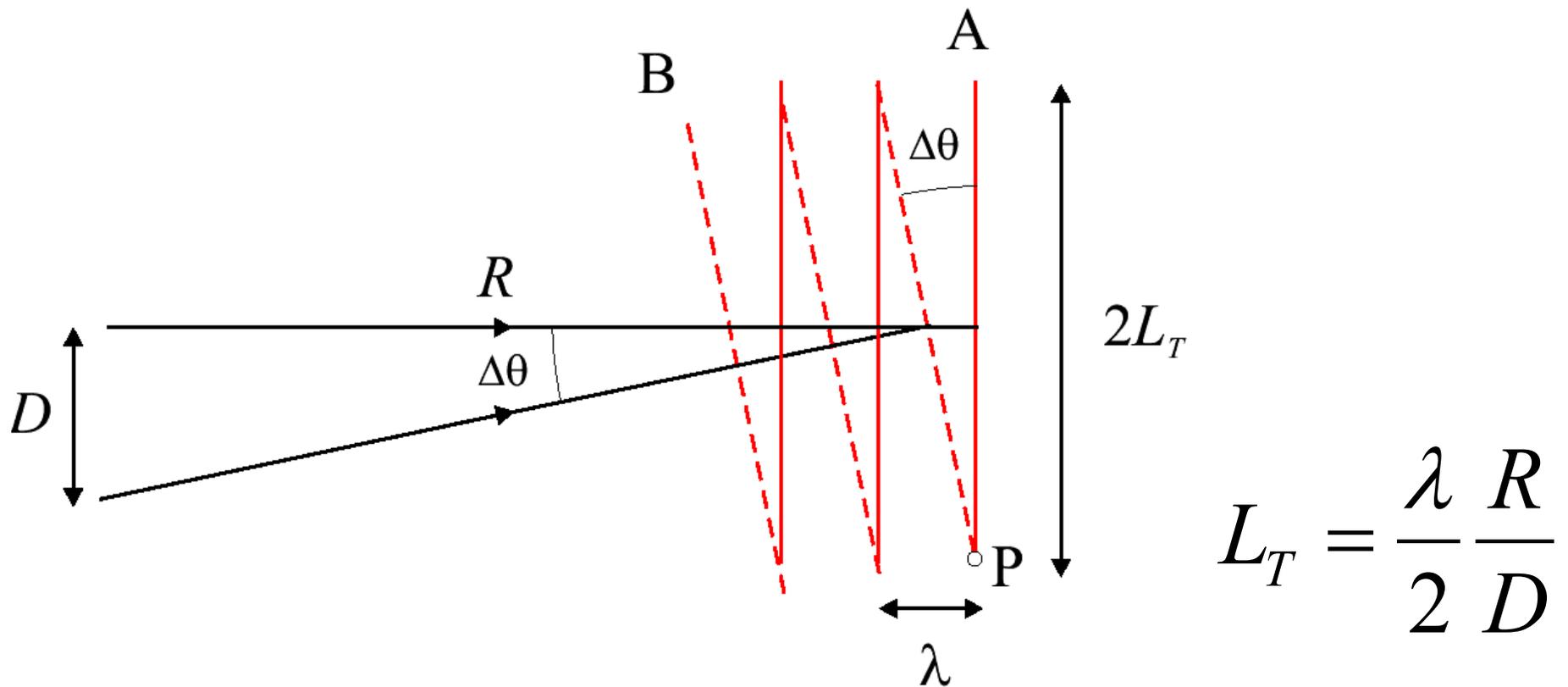
# Longitudinal Coherence



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

Als-Nielsen and McMorro (2001)

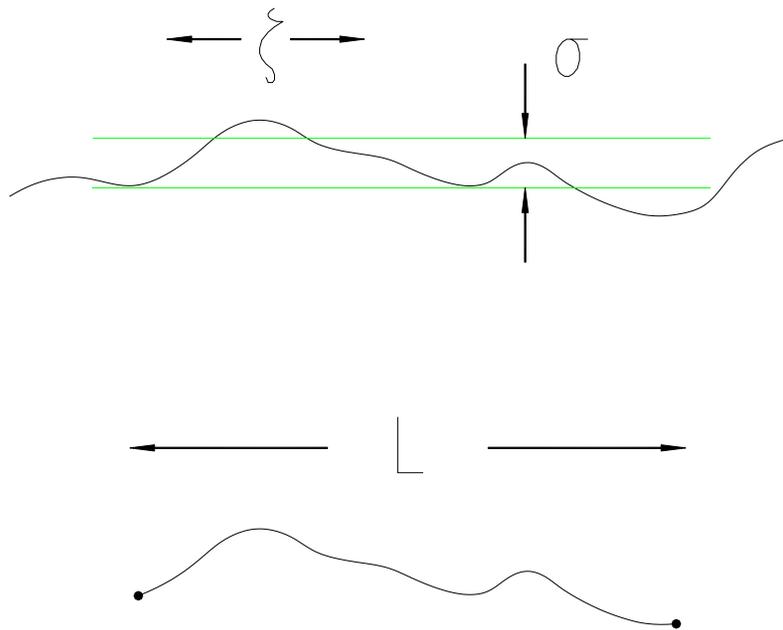
# Lateral (Transverse) Coherence



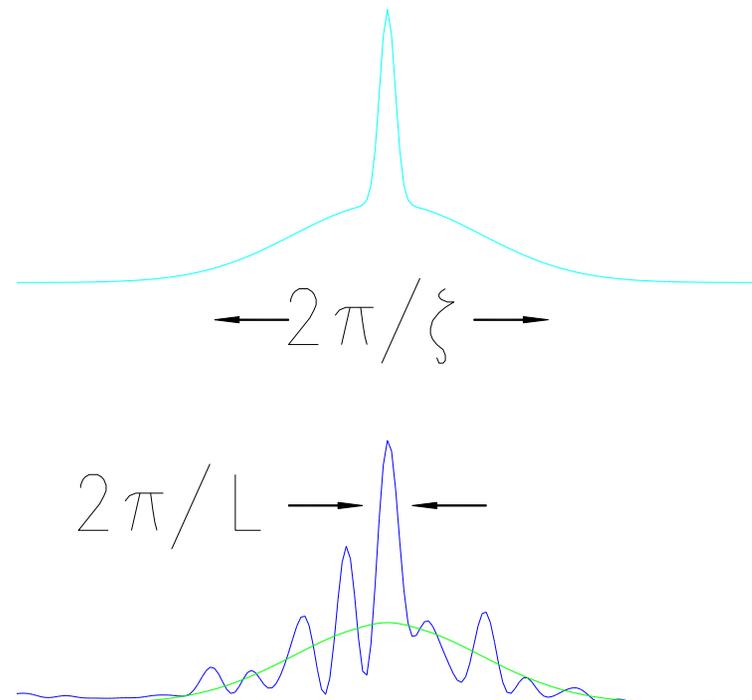
Als-Nielsen and McMorrow (2001)

# Diffuse Scattering acquires Structure using CXD

Real Space

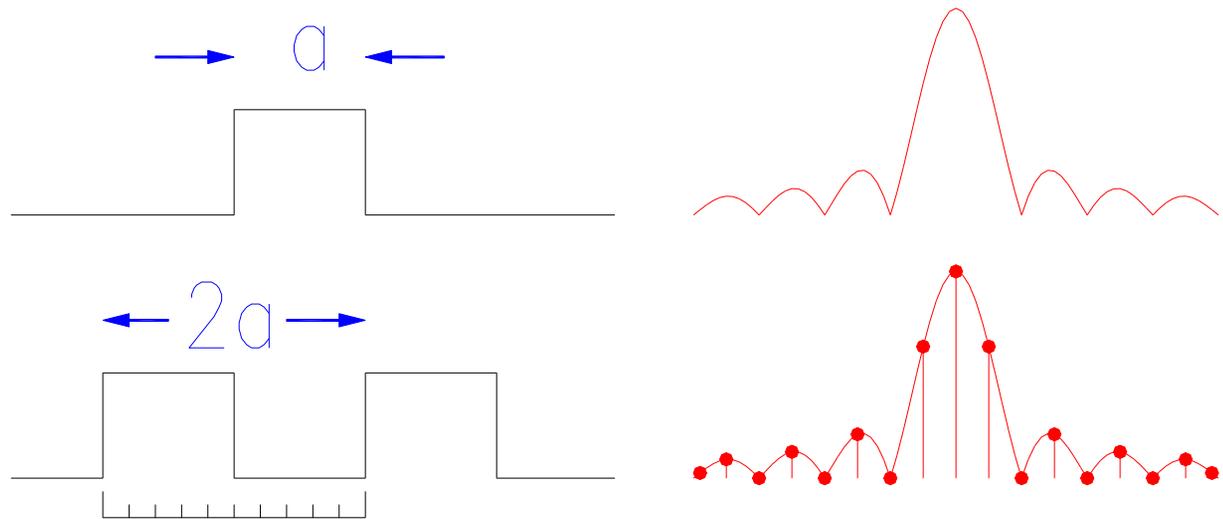


Reciprocal Space

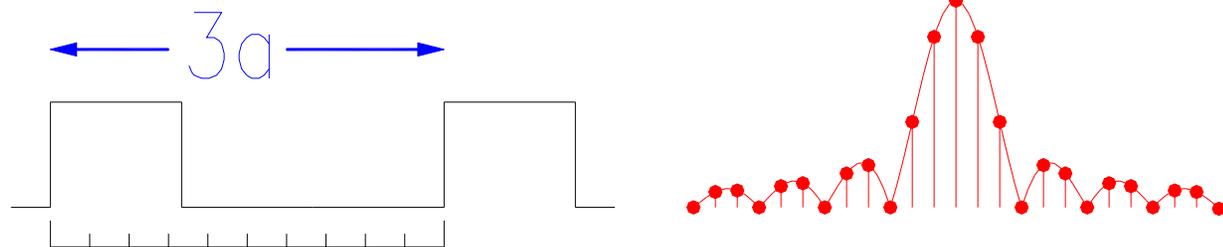


# Oversampling for Small Crystals

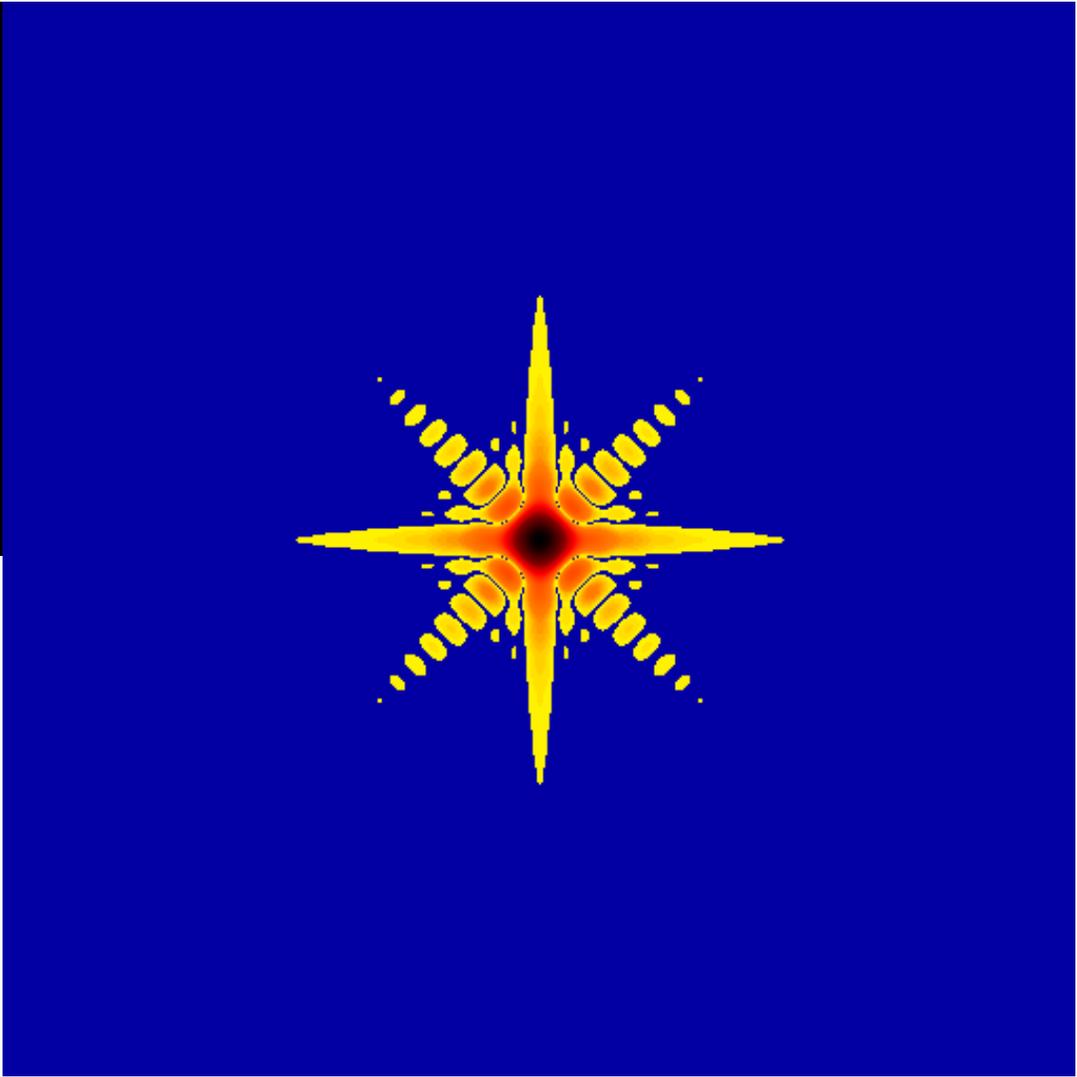
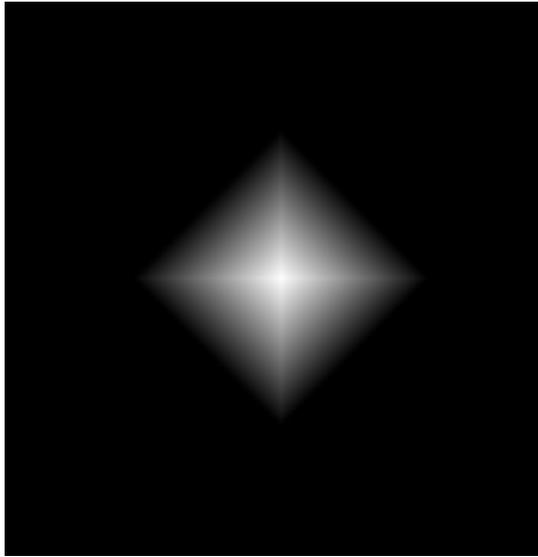
N points in  $2a$ :  
N/2 unknowns

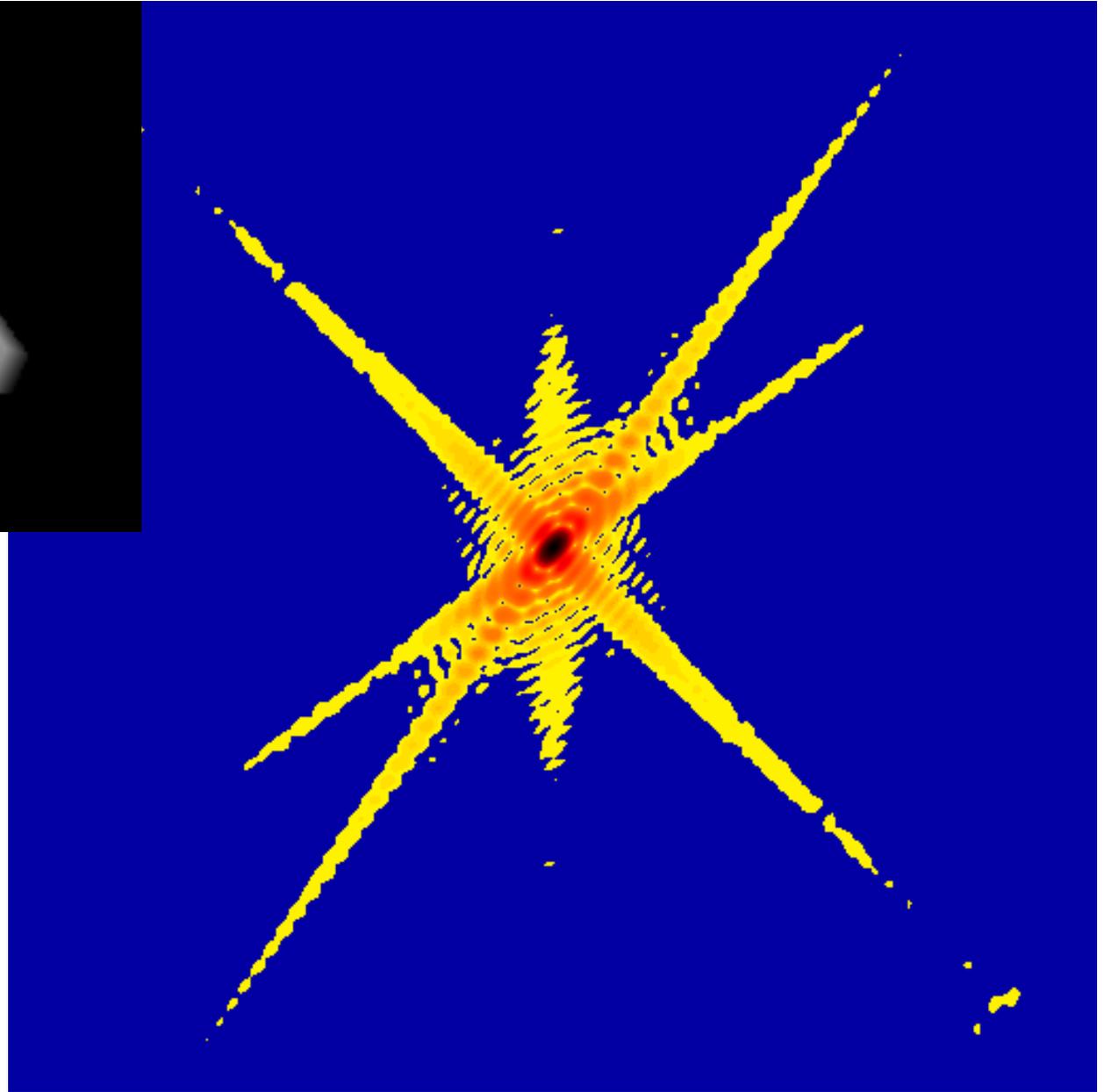
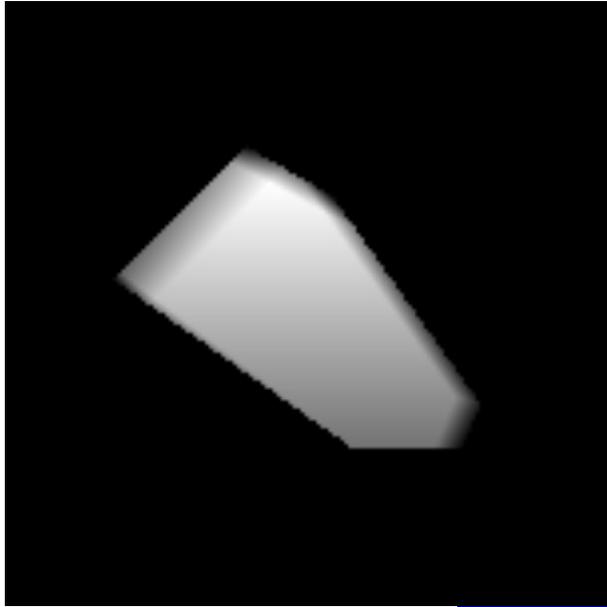


N points in  $3a$ :  
N/3 unknowns



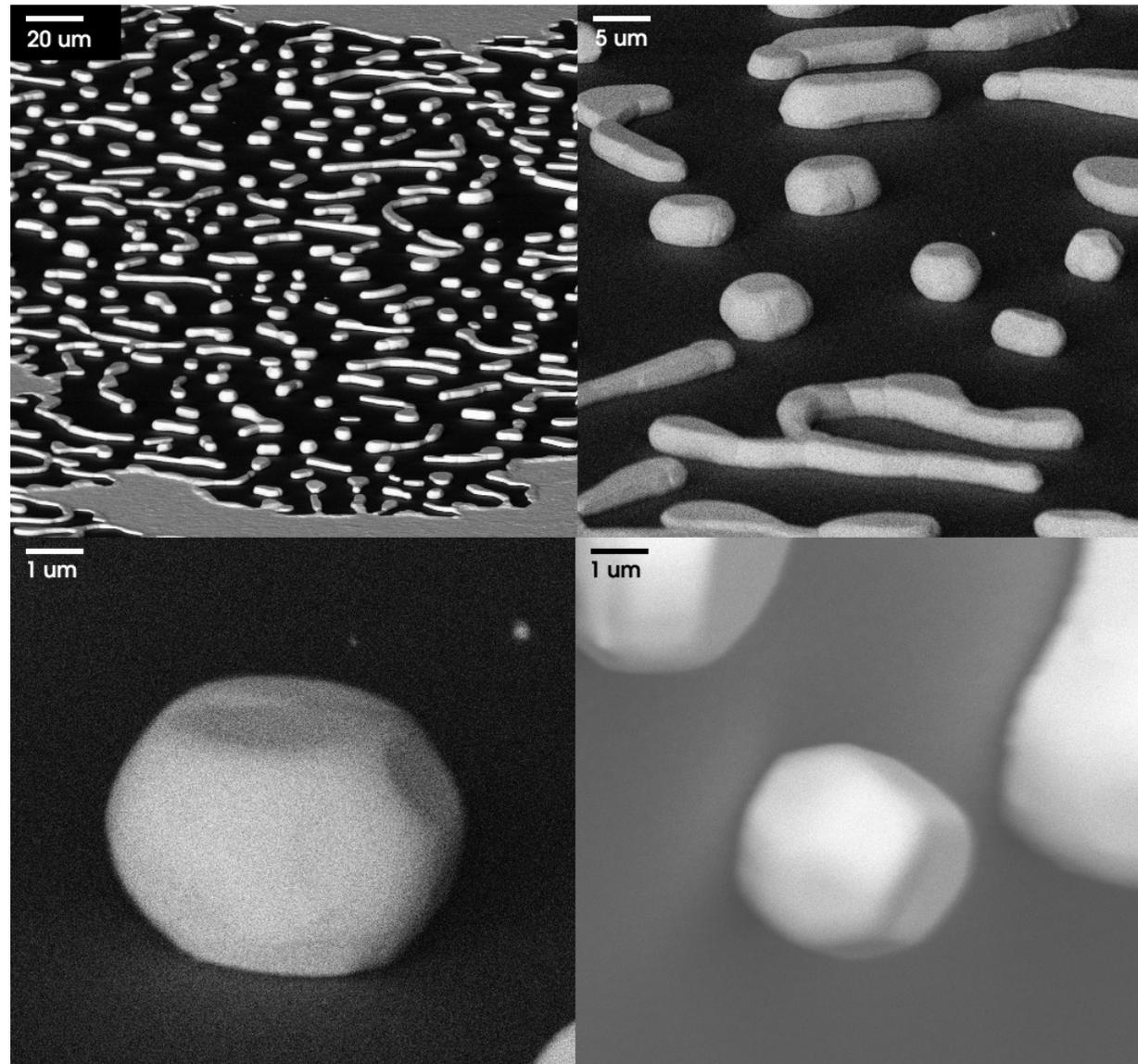
Always N/2 measurements



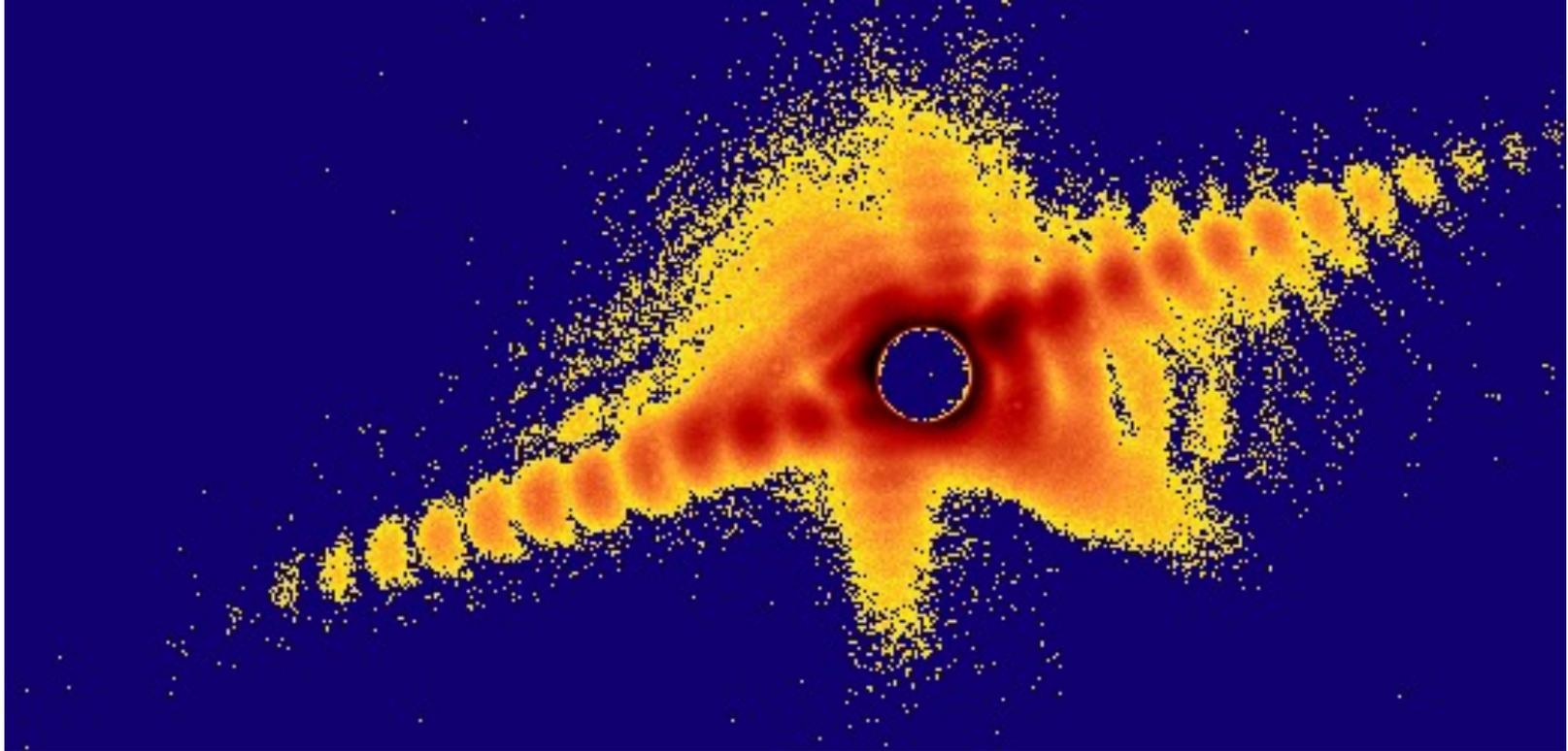


# SEMS

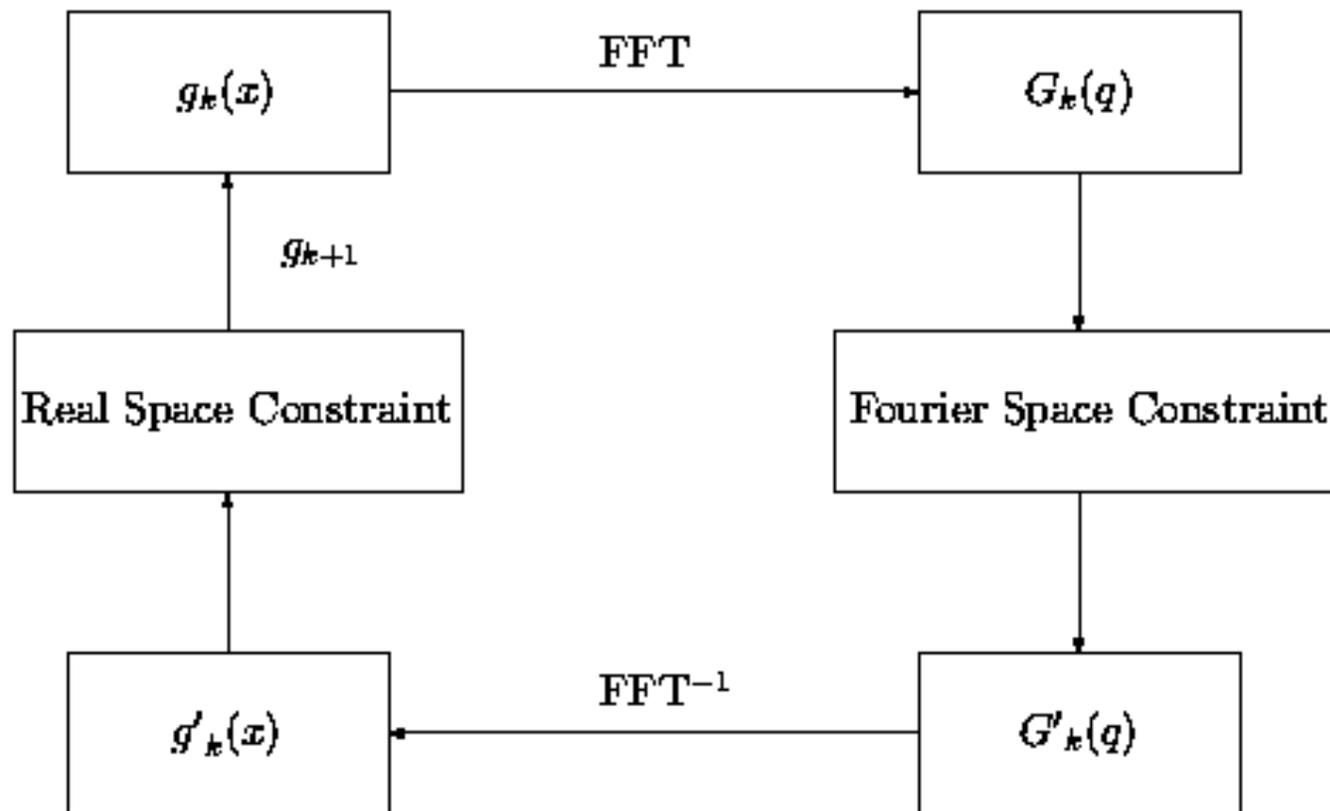
- Au blanket film
- Quartz substrate
- Annealed at 950°C for 70 hrs.



# Micron-sized gold crystal: (111) Bragg reflection

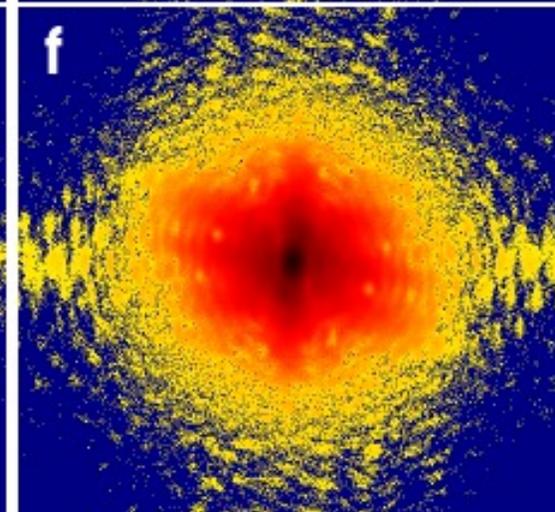
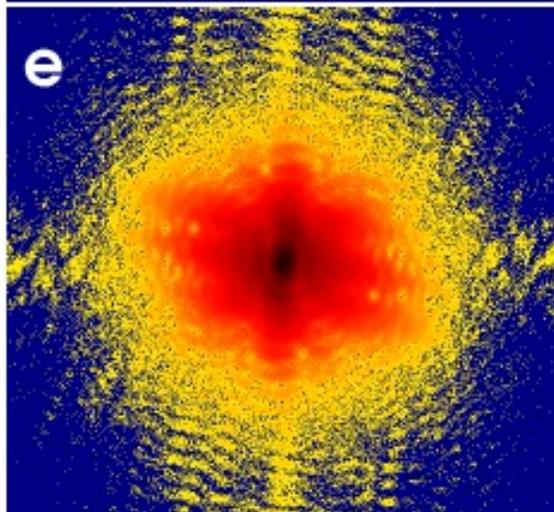
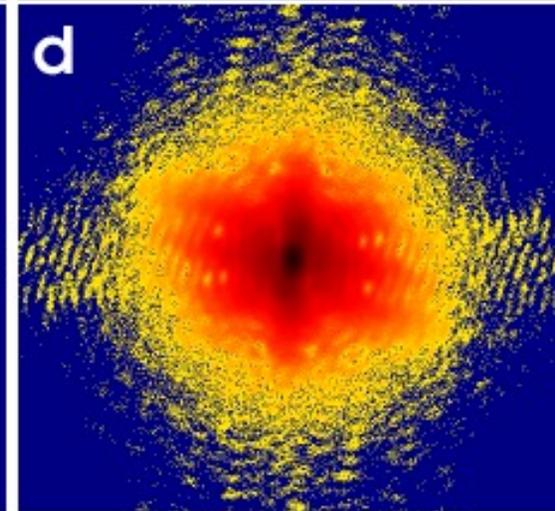
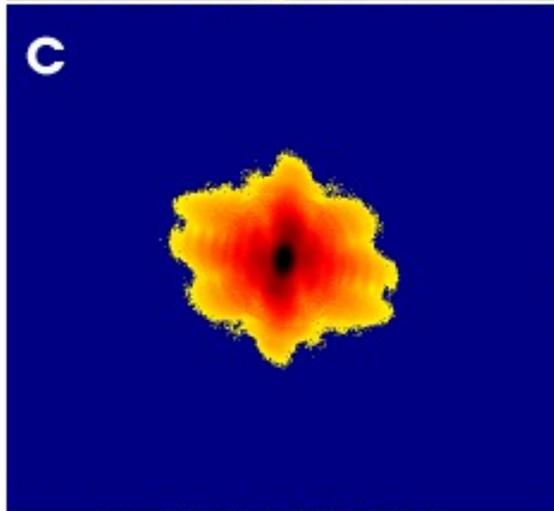
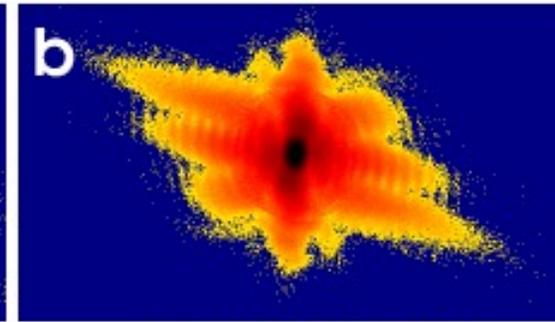
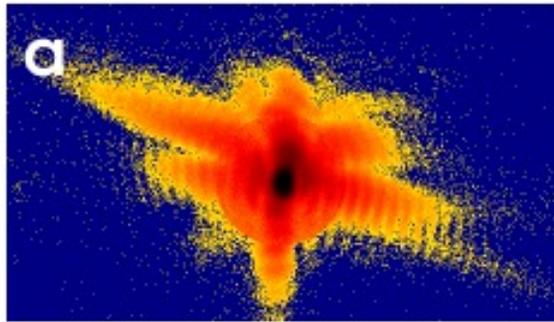


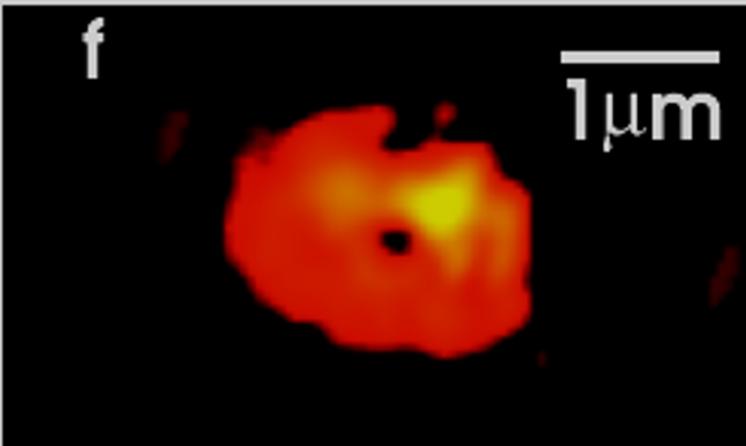
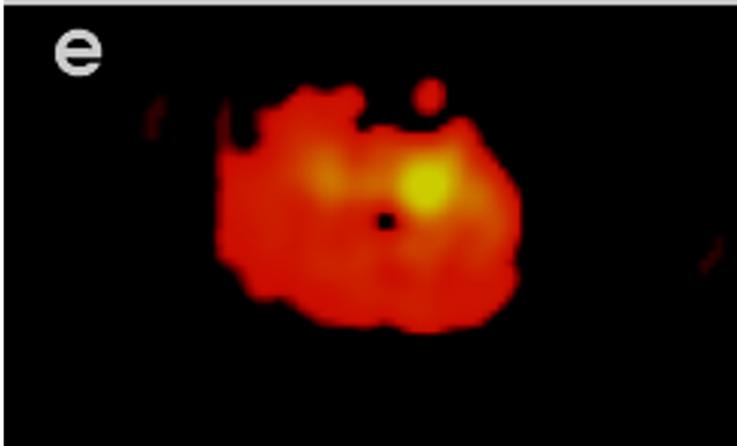
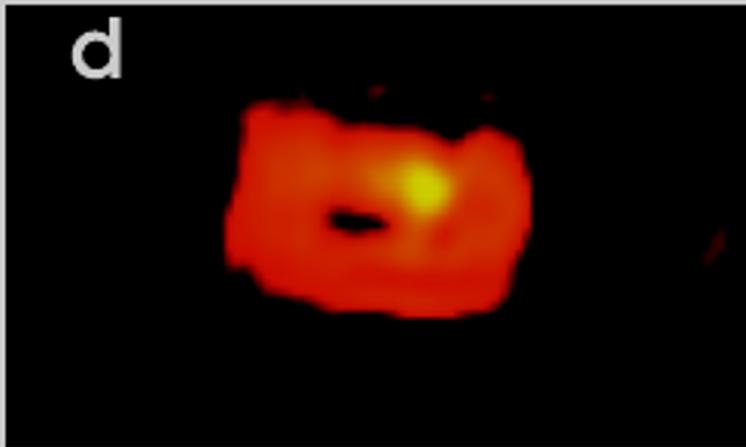
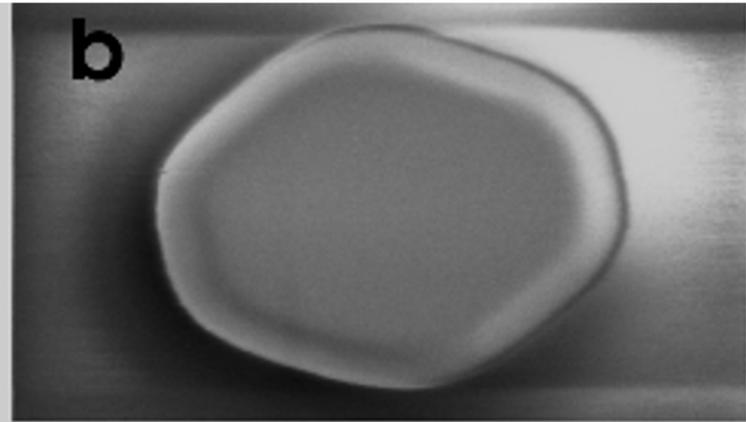
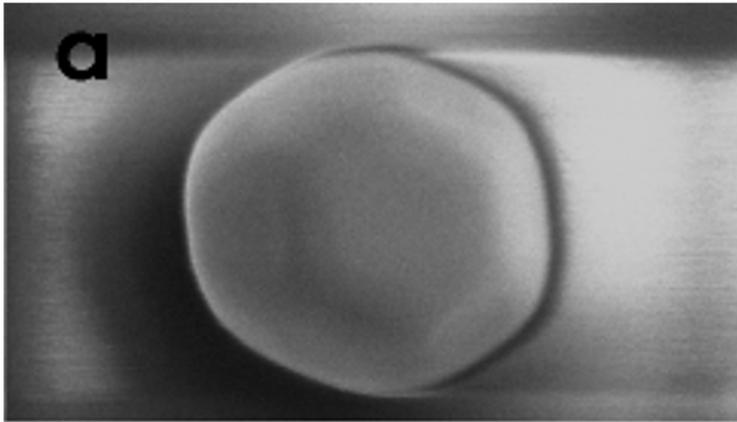
# Generic “Error Reduction” method



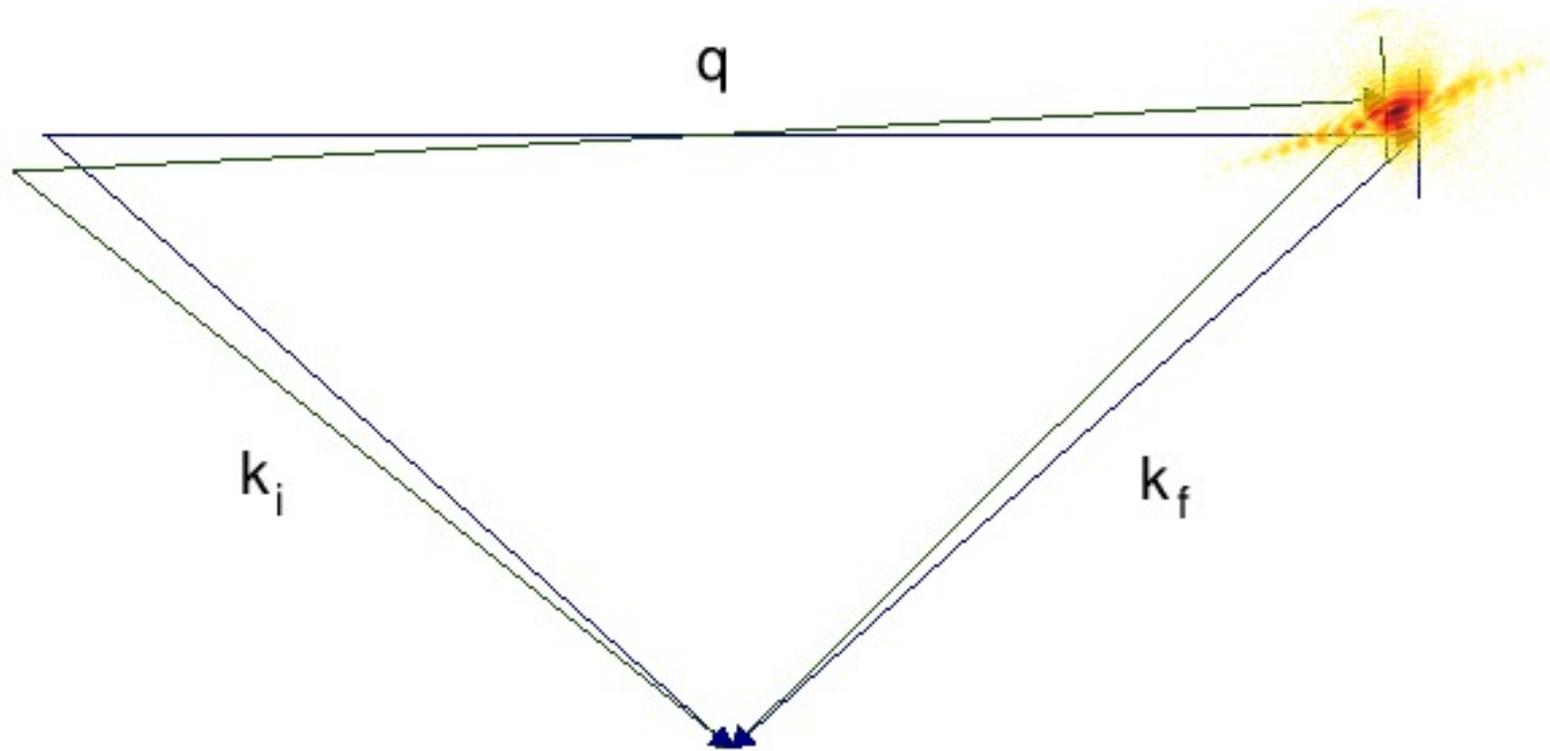
J. R. Fienup *Appl. Opt.* 21 2758 (1982)

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

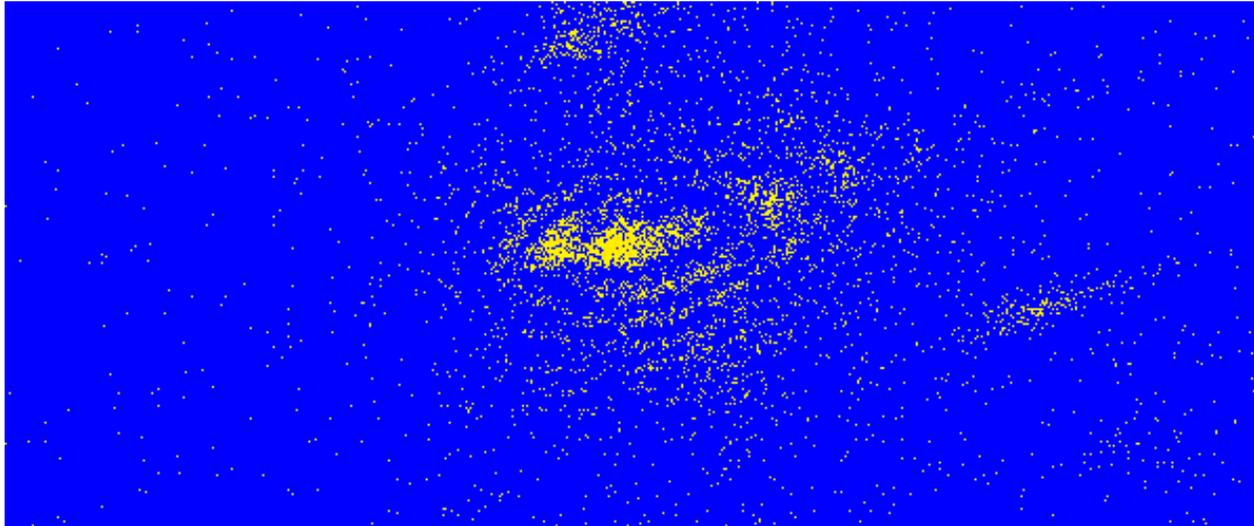




# 3D Diffraction Method

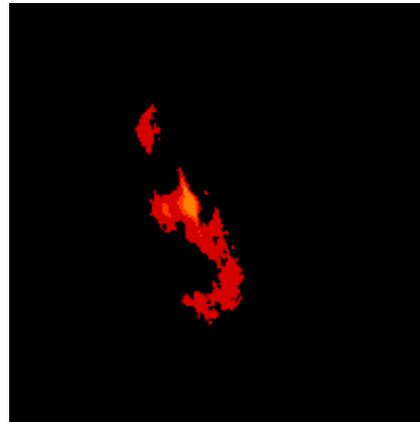


# 3D Diffraction Data 1 micron Au crystal

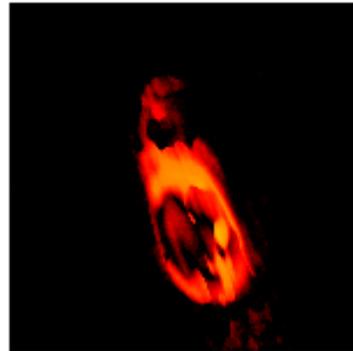
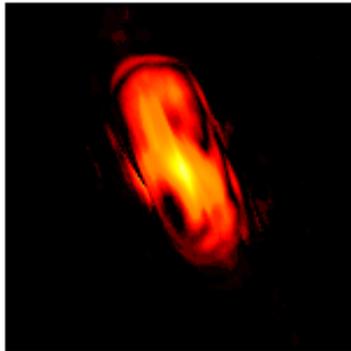
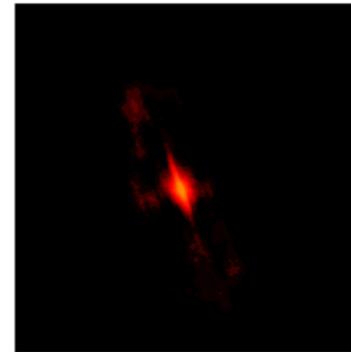
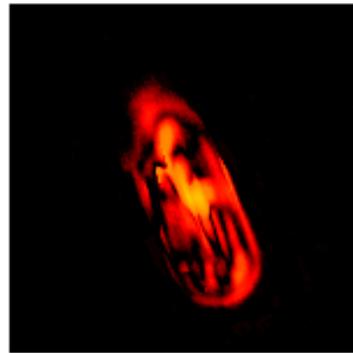
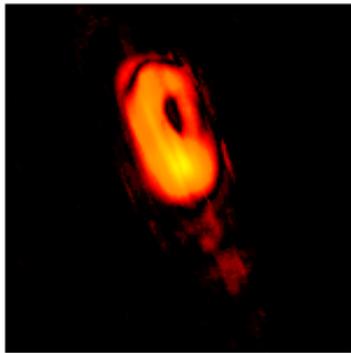
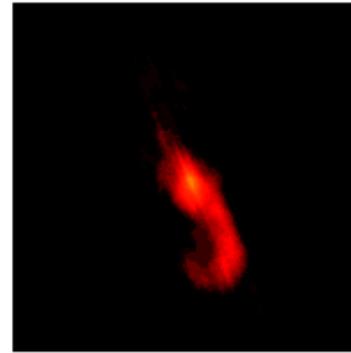
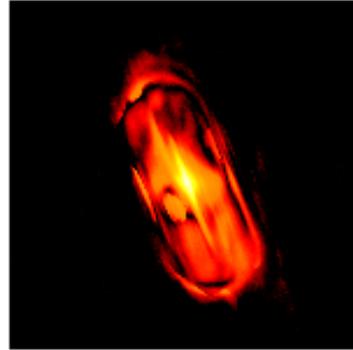
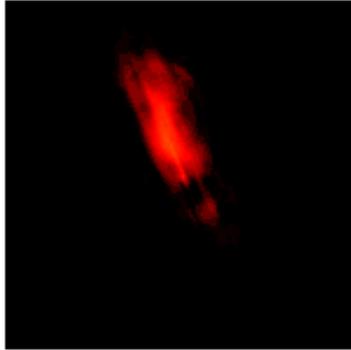
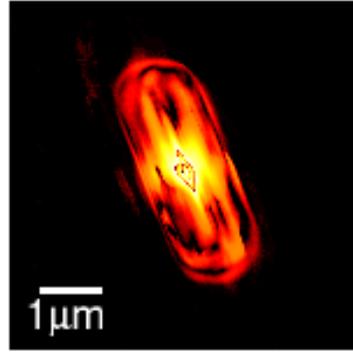
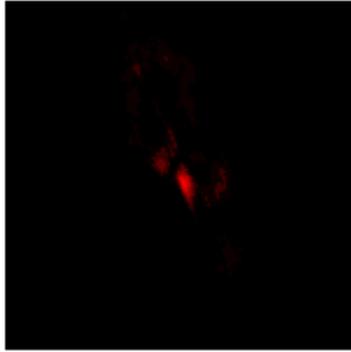


\* Center is Symmetric \*

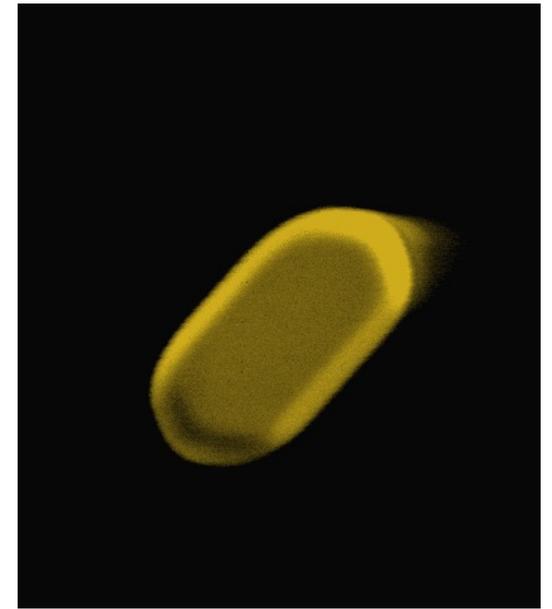
# 30 frames of 3D reconstruction 1 micron gold crystal

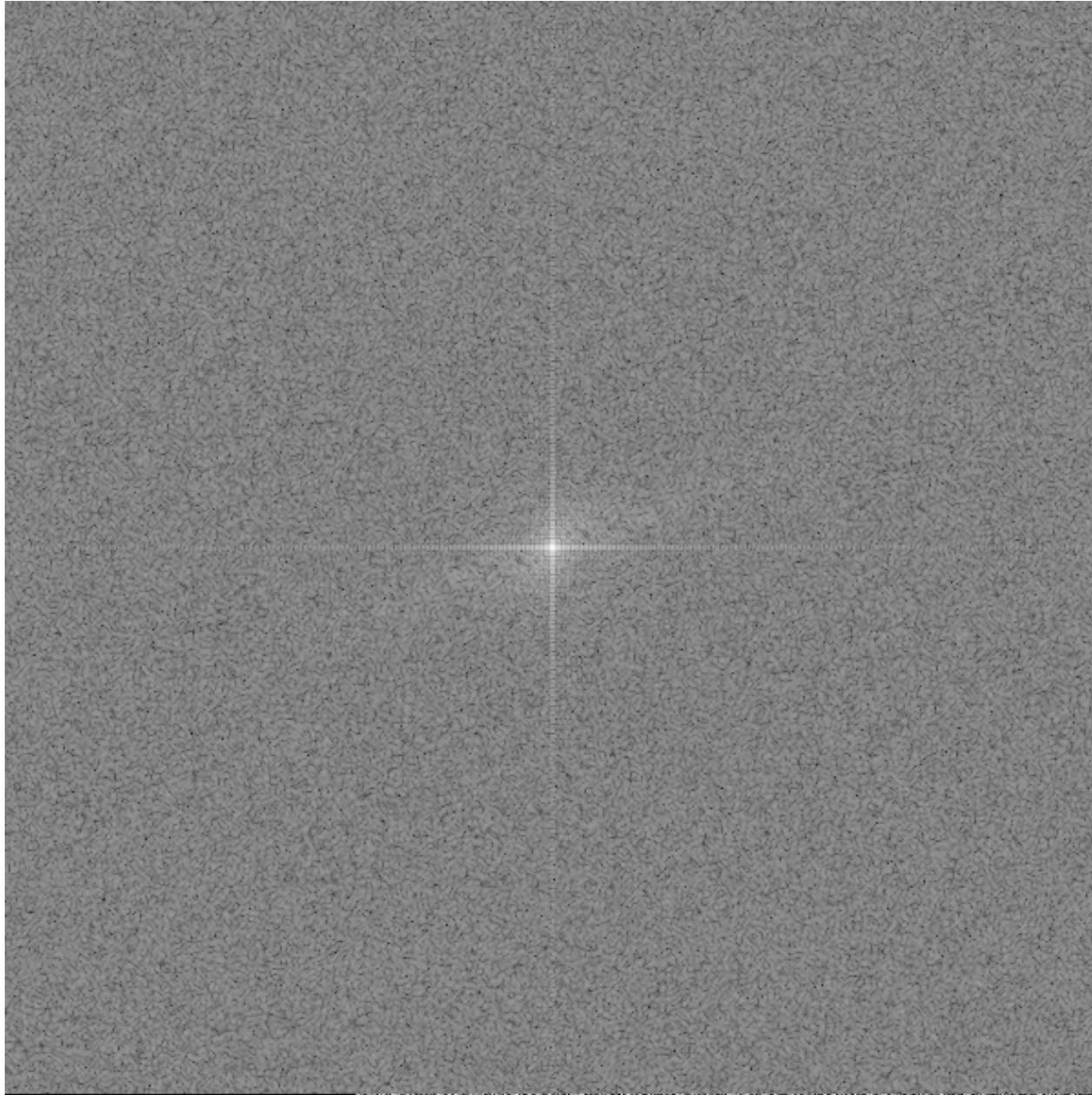


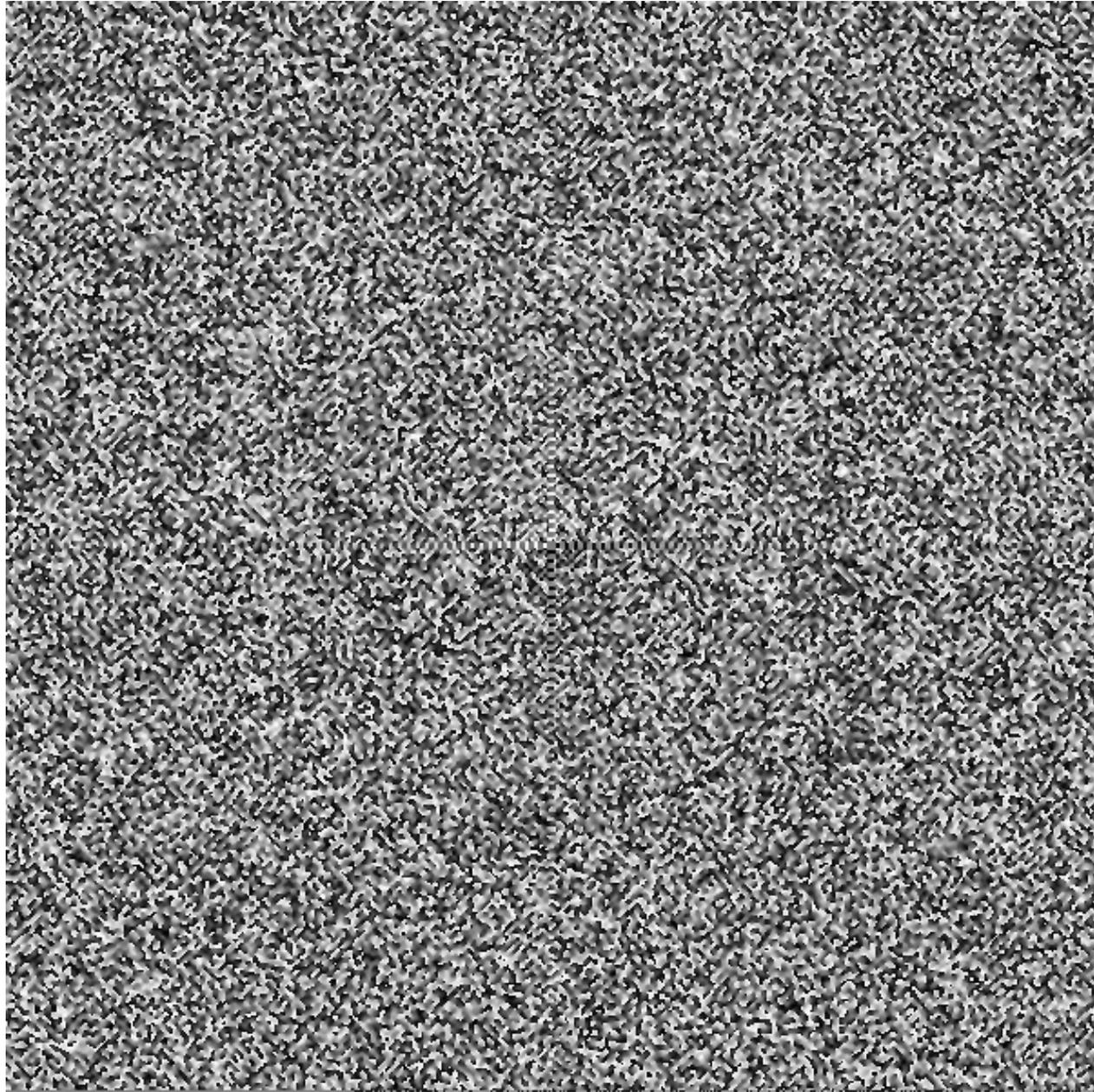
\* CENTER \*



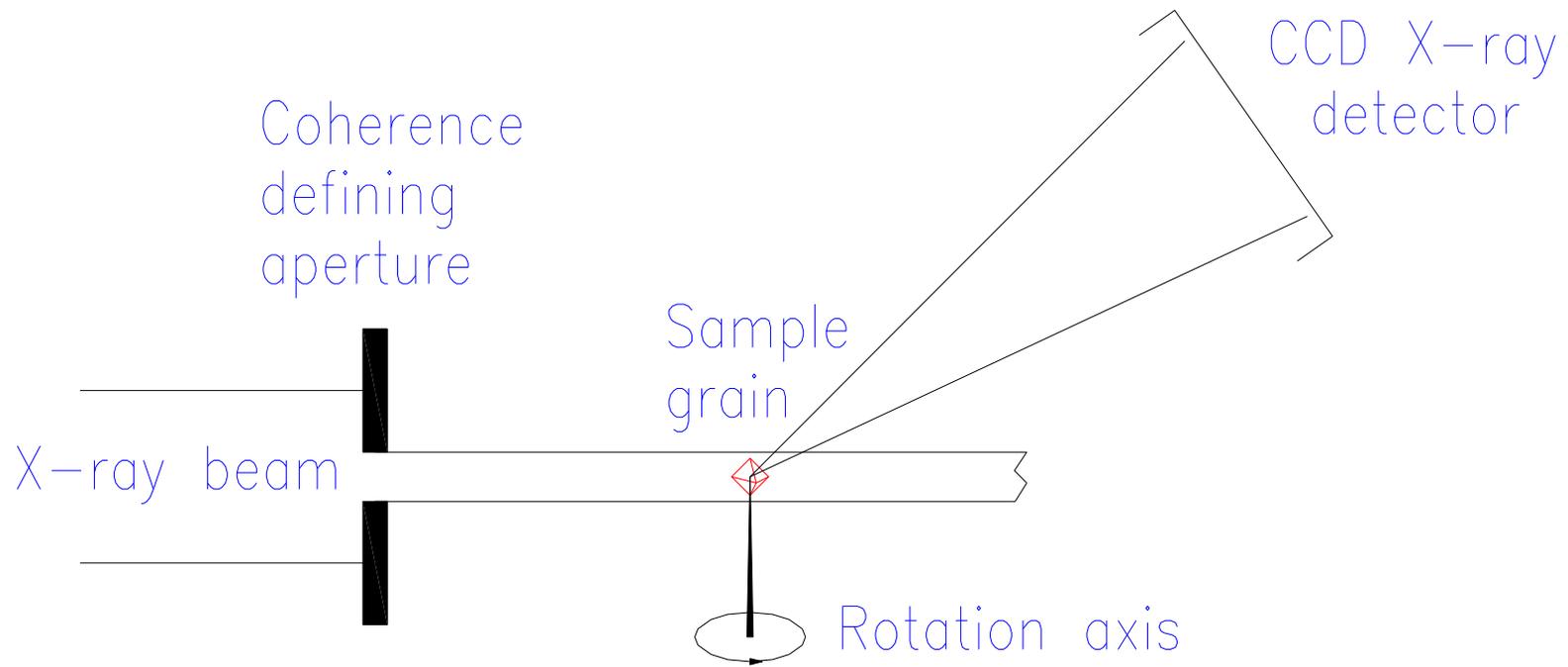
Shape is similar to plan view SEM:



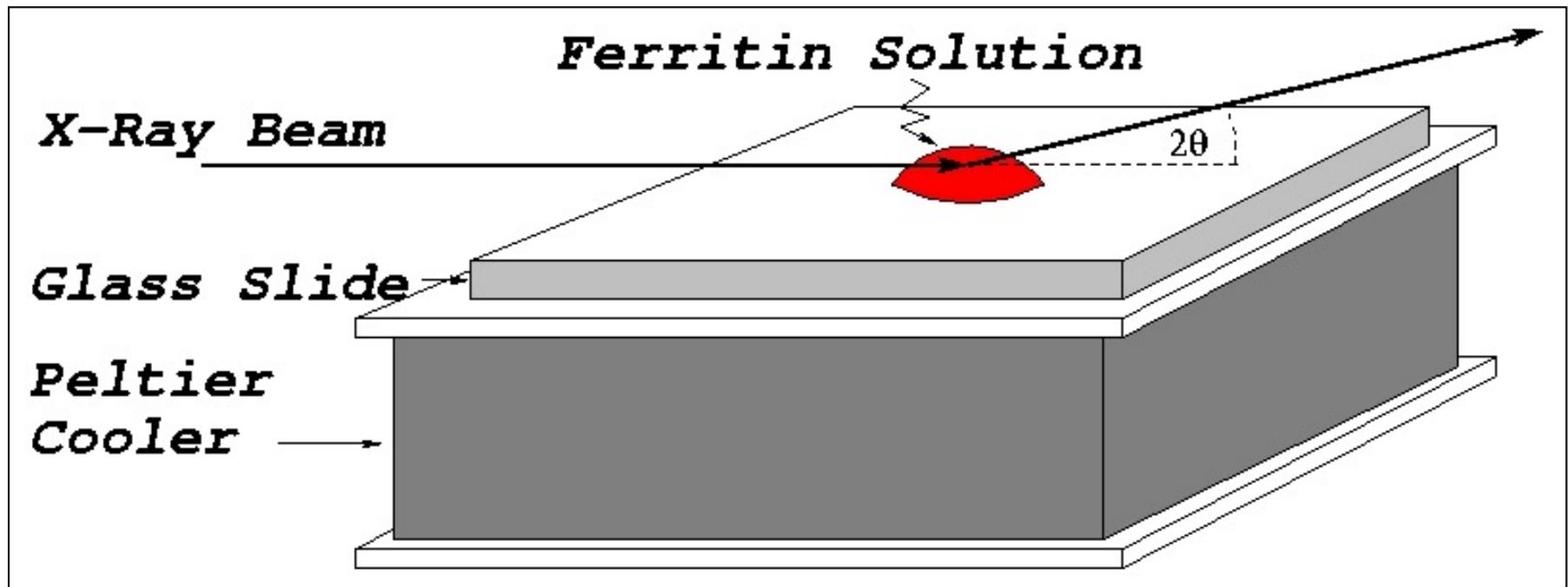




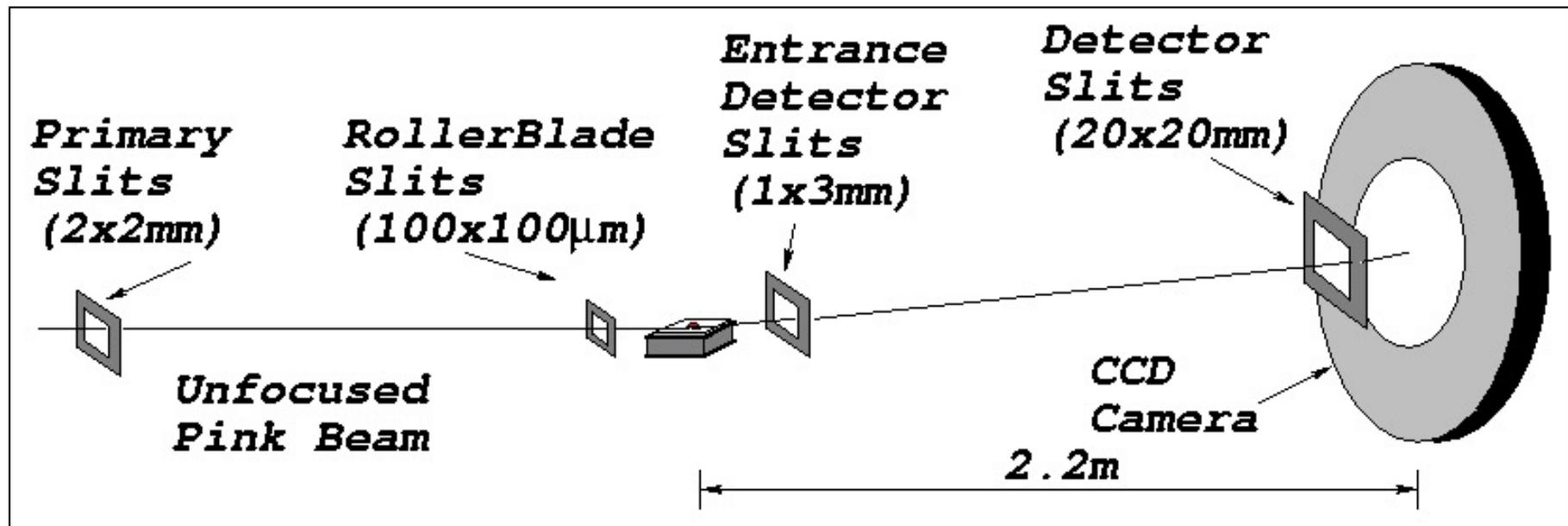
# Lensless X-ray Microscope



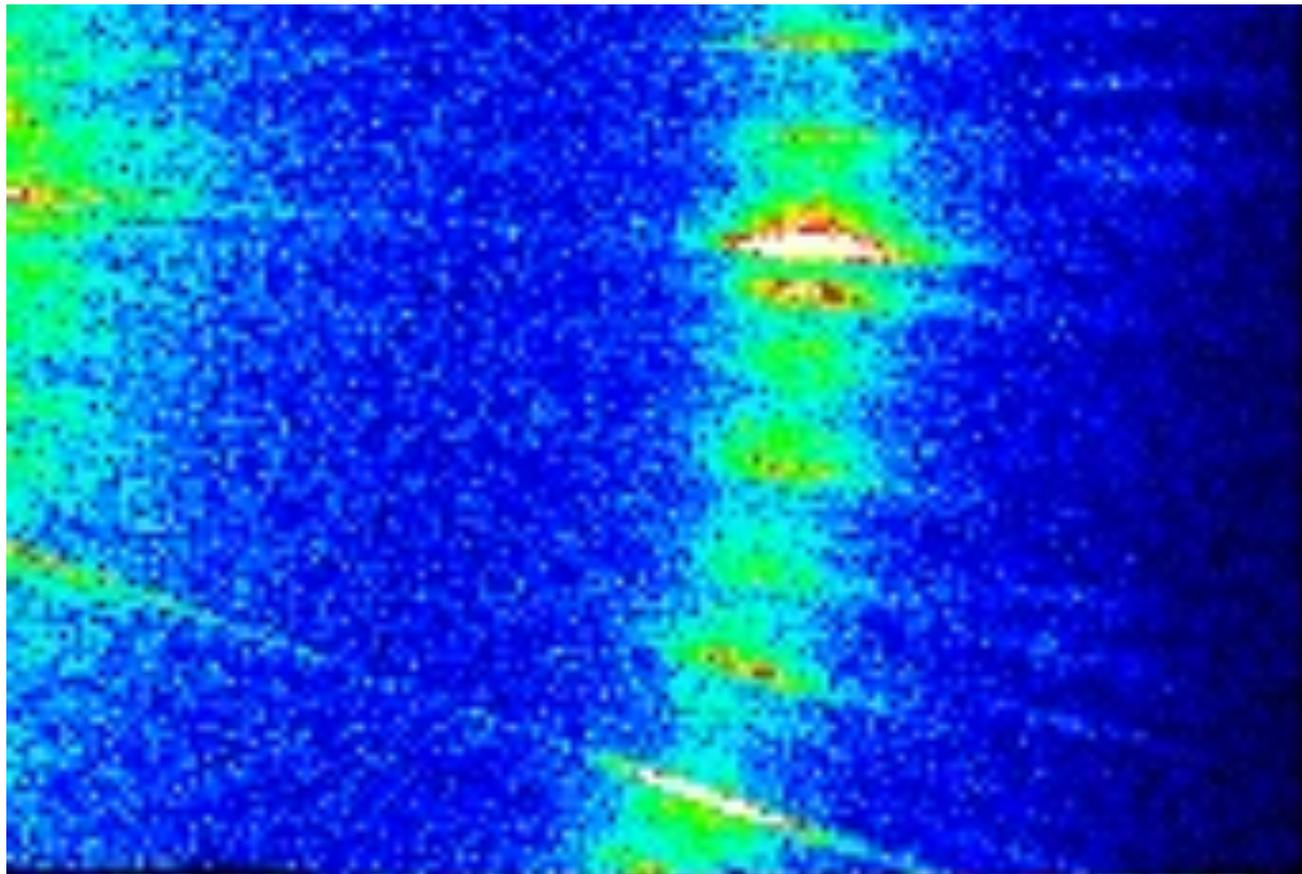
# In-situ Study of Crystallization



# Experiment at APS Sector 34

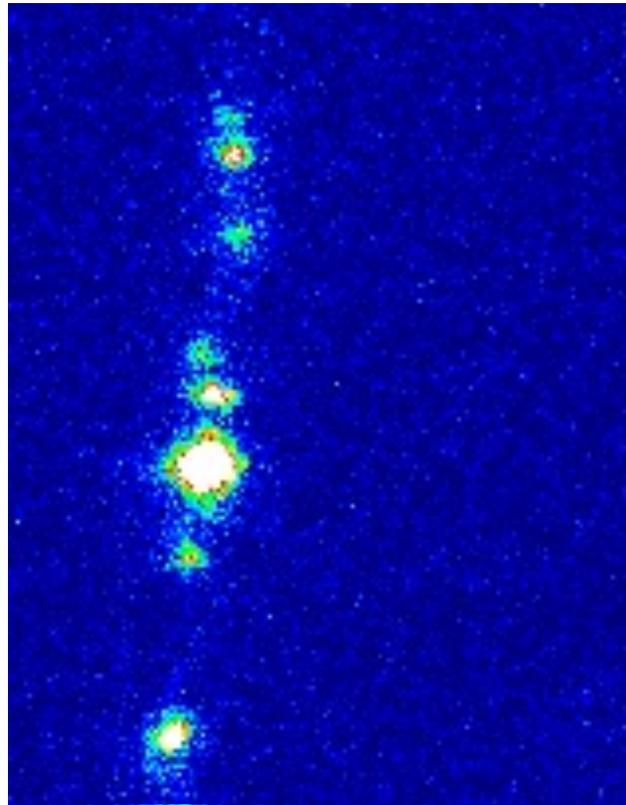


“Pink beam” sees CTRs

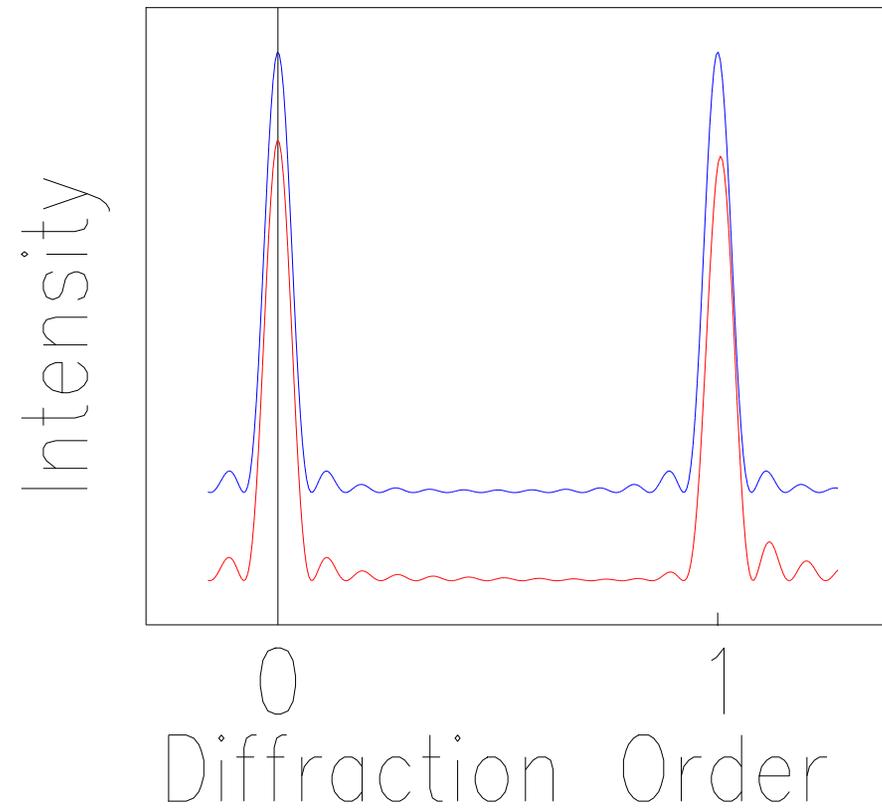
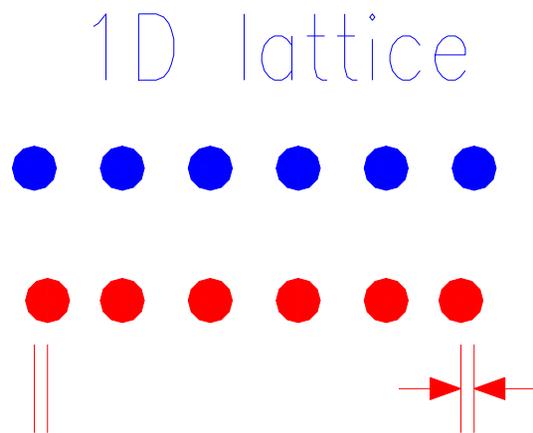


# Ferritin (111) Powder Ring

- 50 frames
- 30sec exposure
- 0.3sec playback
- 150x200 pixels  
of 22.5 $\mu\text{m}$



# Imaging of Lattice Strains



# Conclusions

- “Diffuse” scattering acquires fine structure
- Surface of Ferritin crystal is contracted
- Shapes of small particles
- 3D imaging of internal strains possible