

Strains in Nanocrystals can be Imaged by Coherent X-ray Diffraction

- Ian Robinson
- Ivan Vartanyants Department of Physics
University of Illinois
- Mark Pfeifer
- John Pitney
- Garth Williams
- Sébastien Boutet Reading University, 13 May 2002

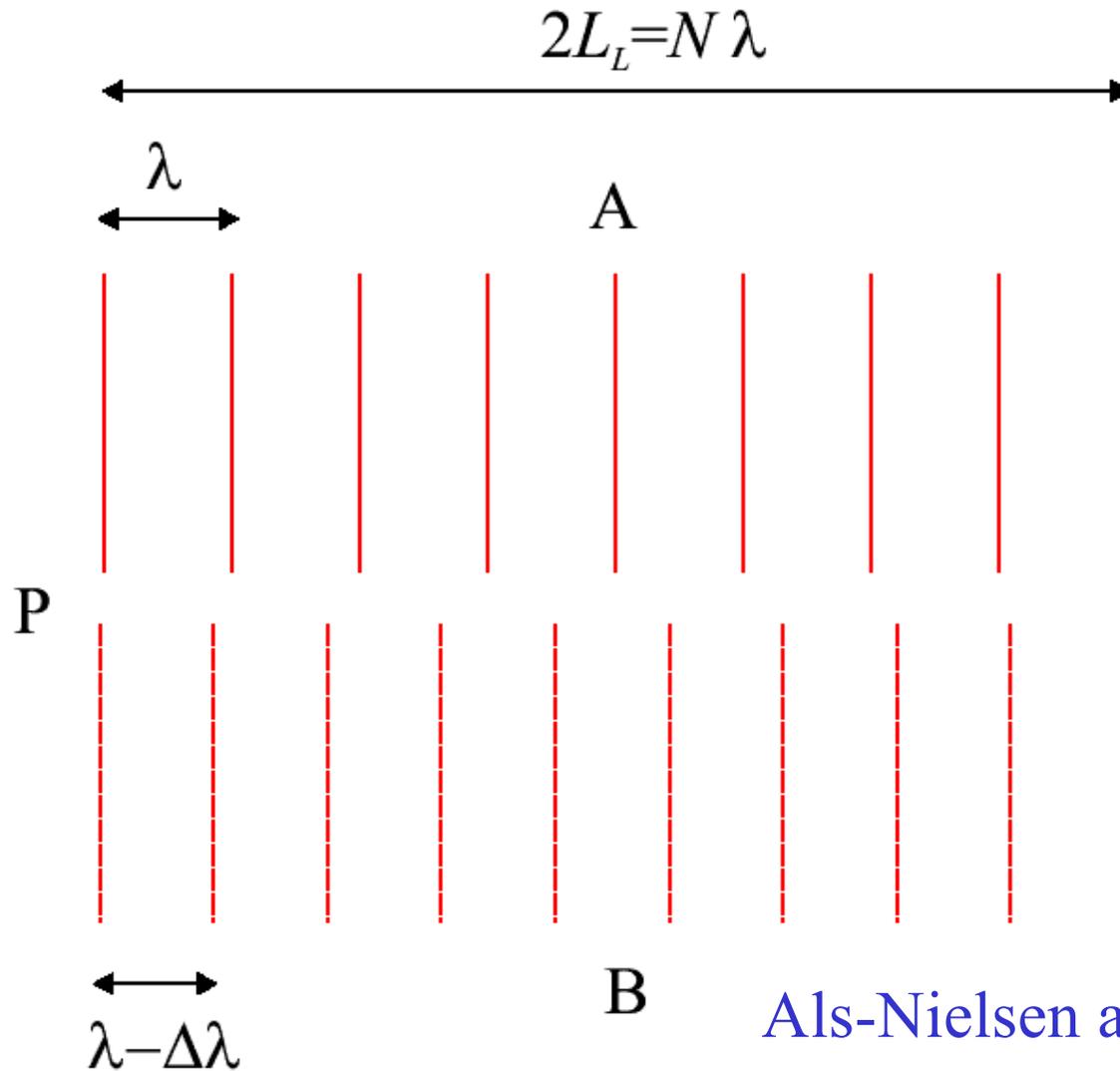
Outline

- Goals of Coherent Diffraction
- The **Phase** Problem
- Nanocrystal Shapes
- CXD beamline at Argonne
- Potential Applications

Goals of Coherent Diffraction

- Thermodynamic fluctuations
 - May be too hard because of beam-induced effects, sample damage
- 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

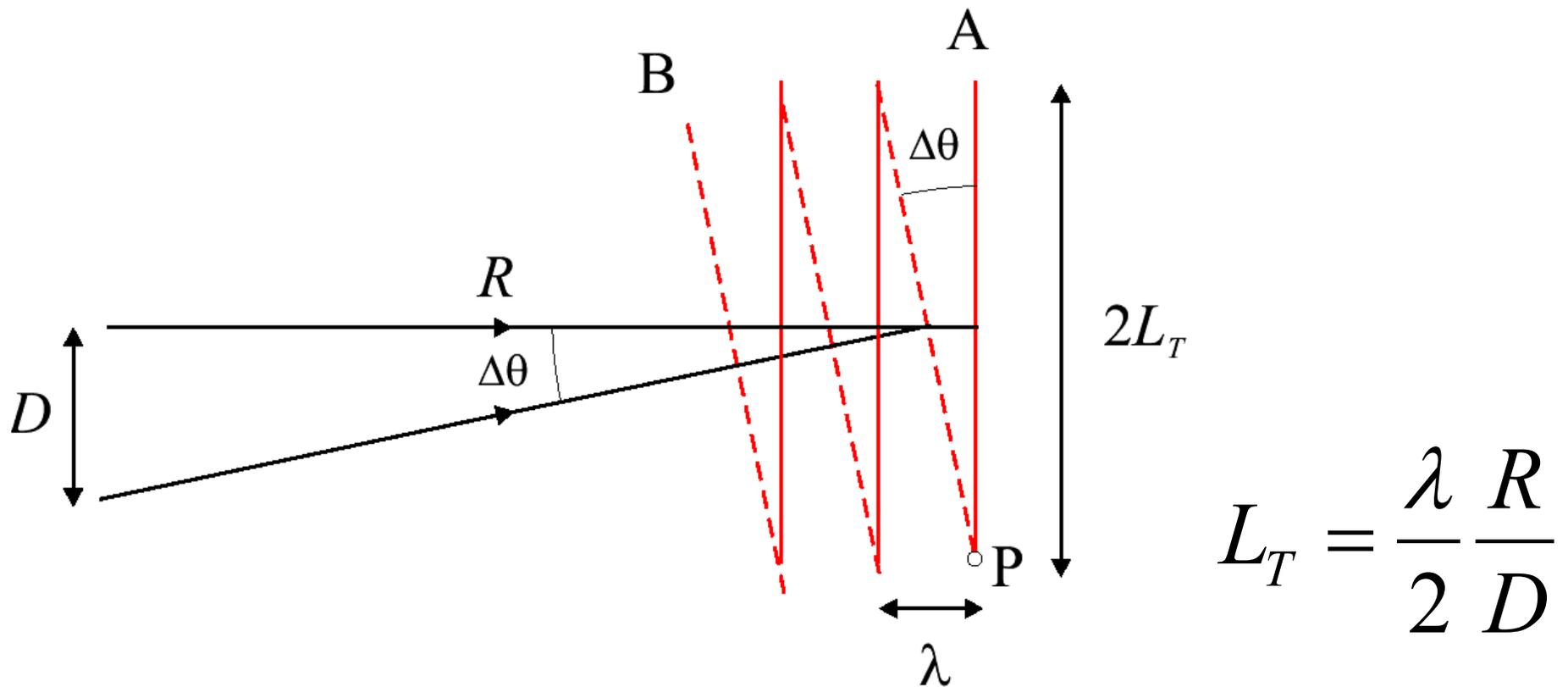
Longitudinal Coherence



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

Als-Nielsen and McMorow (2001)

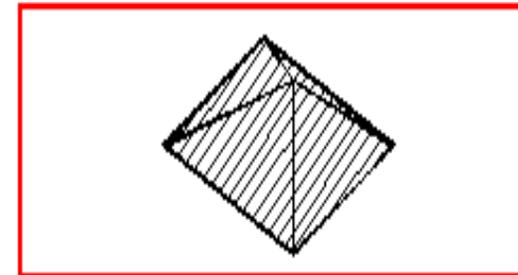
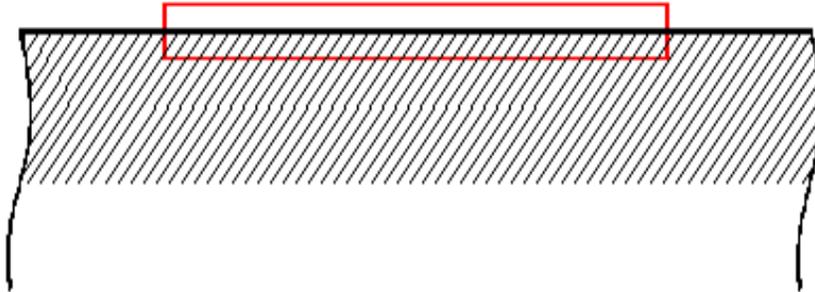
Lateral (Transverse) Coherence



Als-Nielsen and McMorrow (2001)

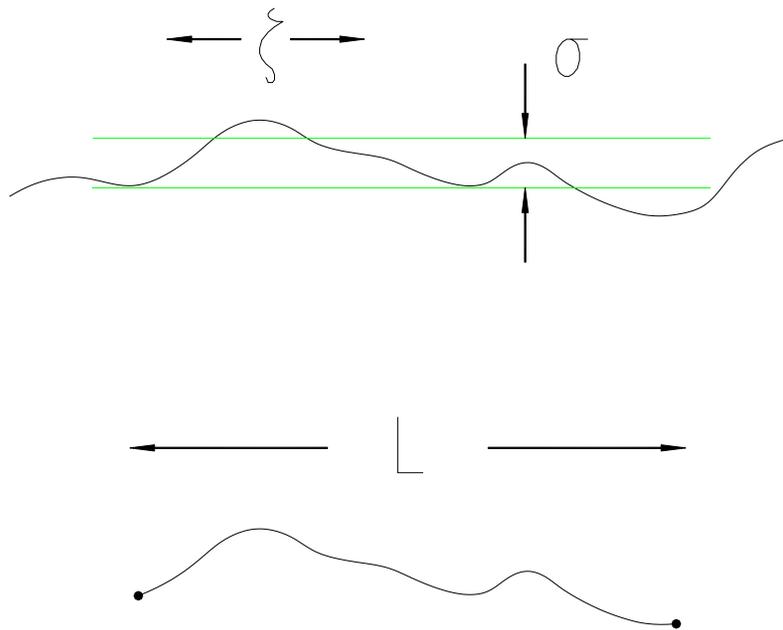
Coherence at the APS

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

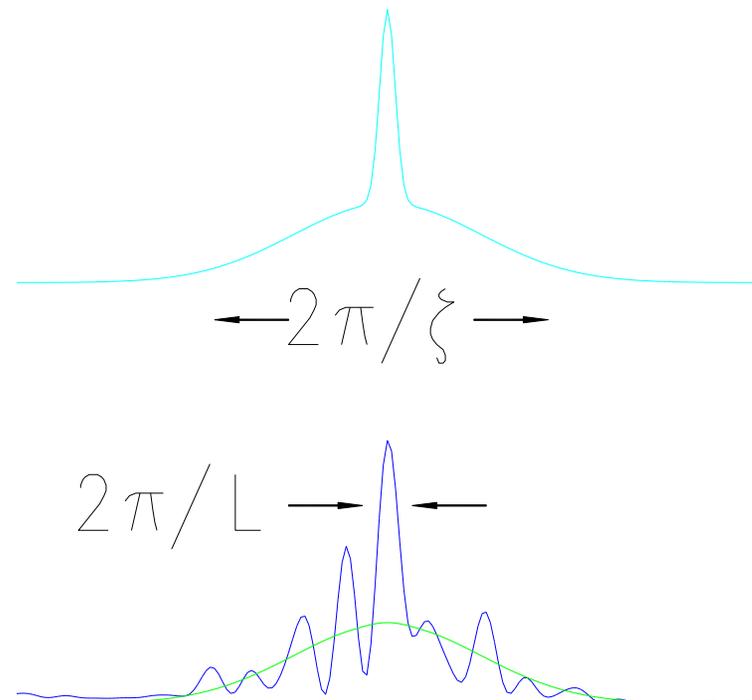


Diffuse Scattering acquires Structure using CXD

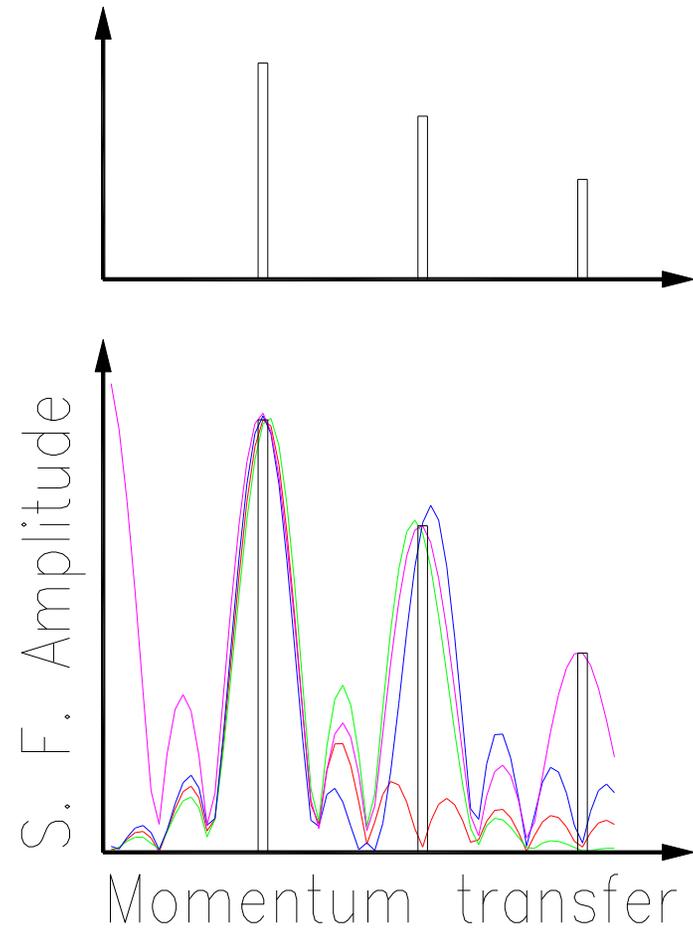
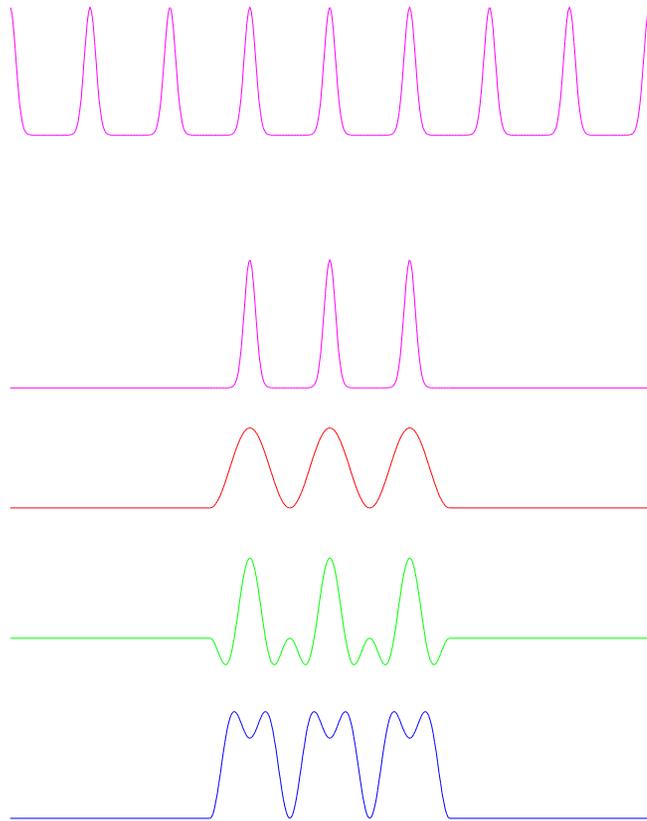
Real Space



Reciprocal Space

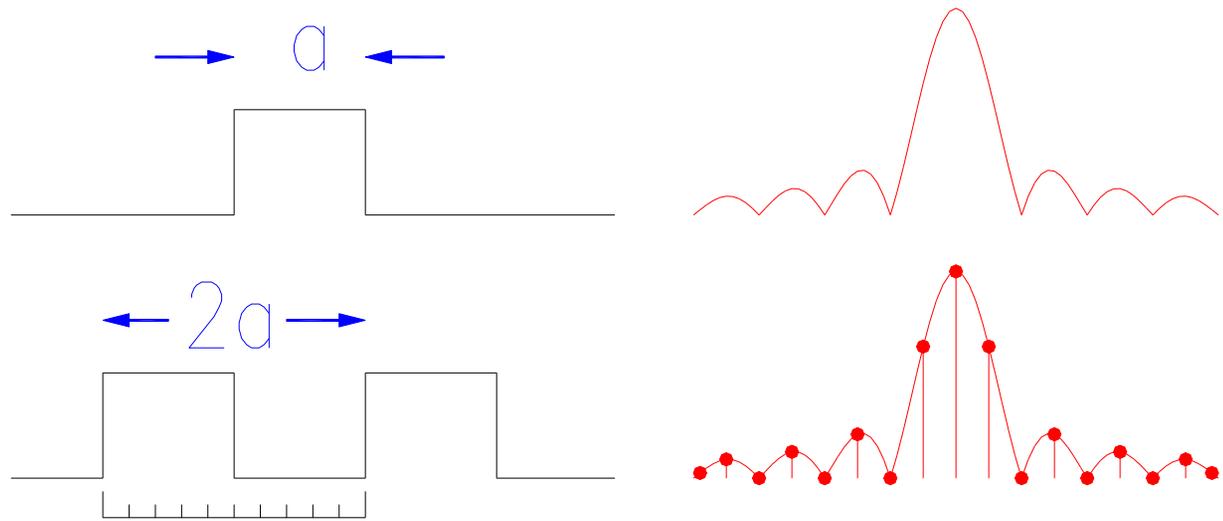


Phase Problem: Finite-size Effect

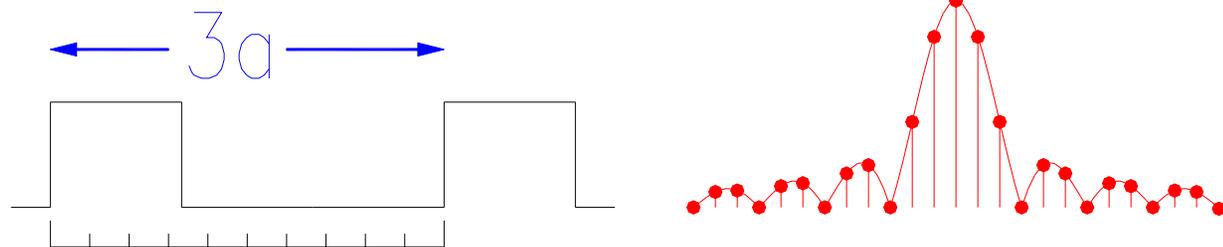


Oversampling for Small Crystals

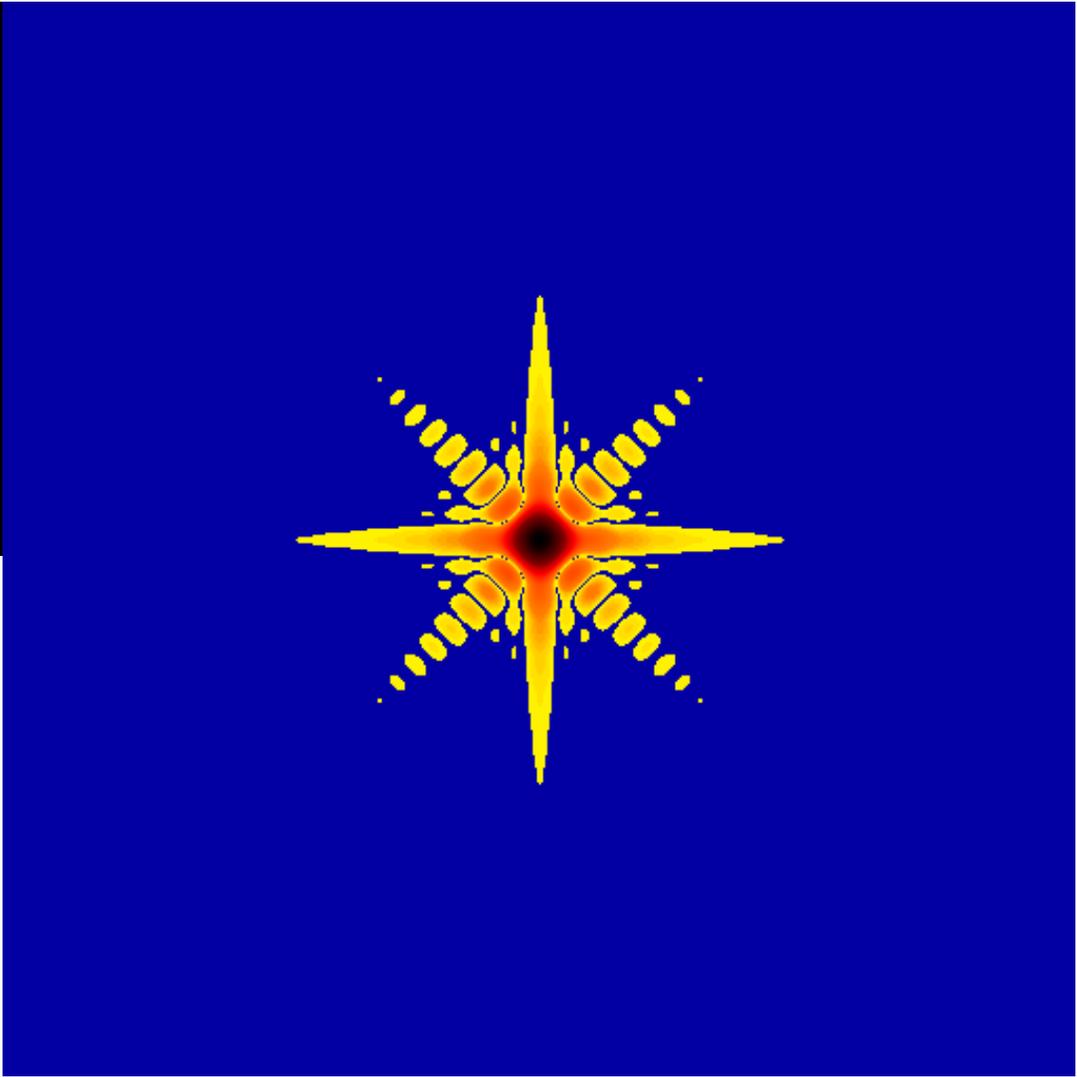
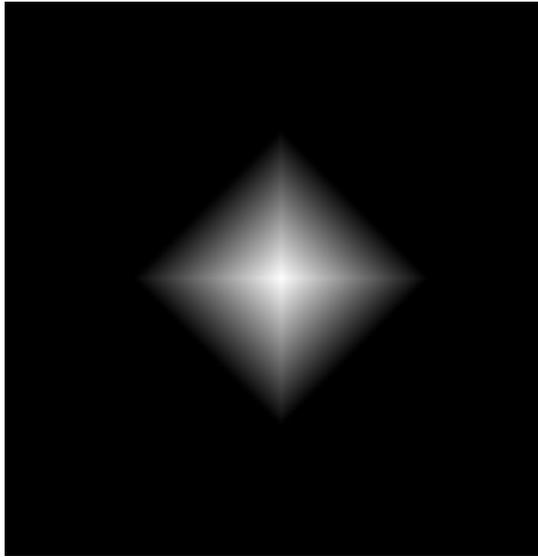
N points in $2a$:
N/2 unknowns



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



Always N/2 measurements



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)

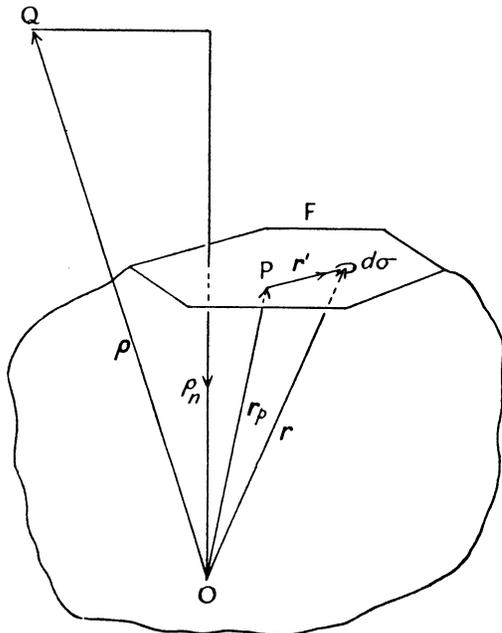
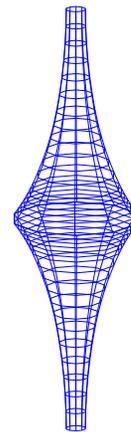
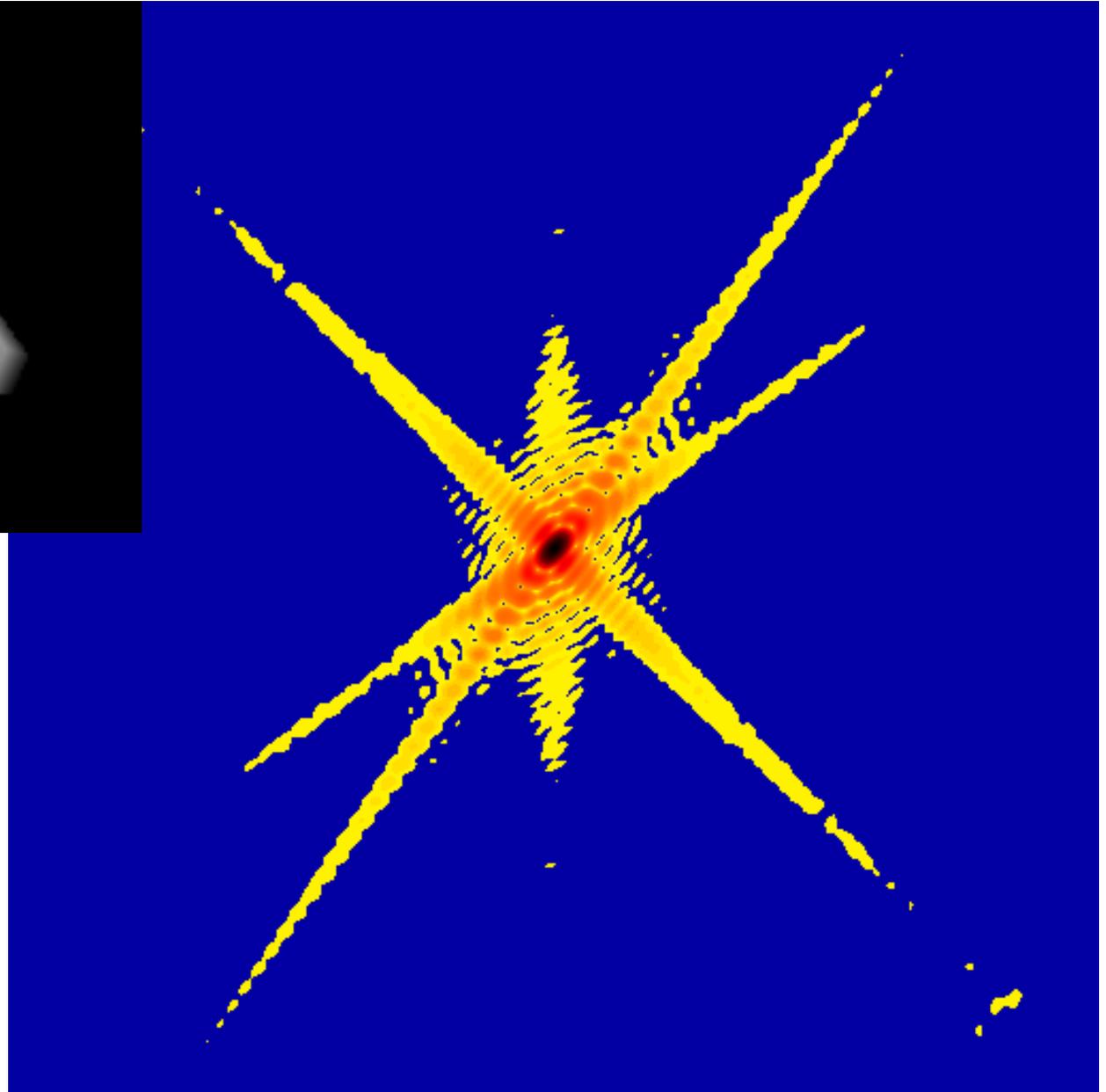
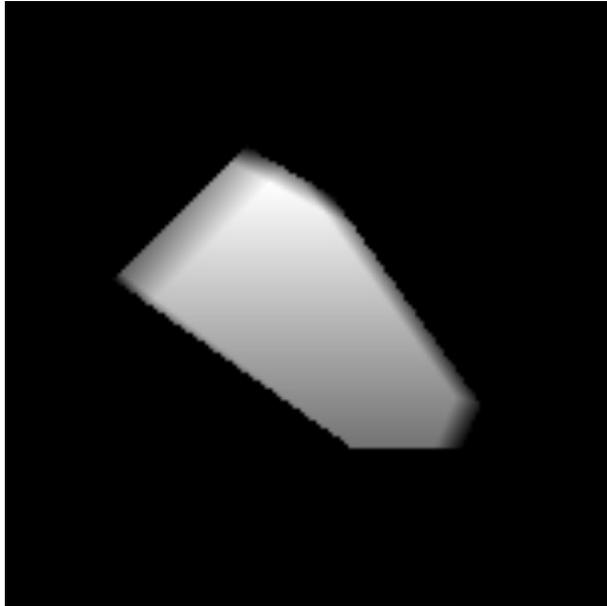


FIG. 200

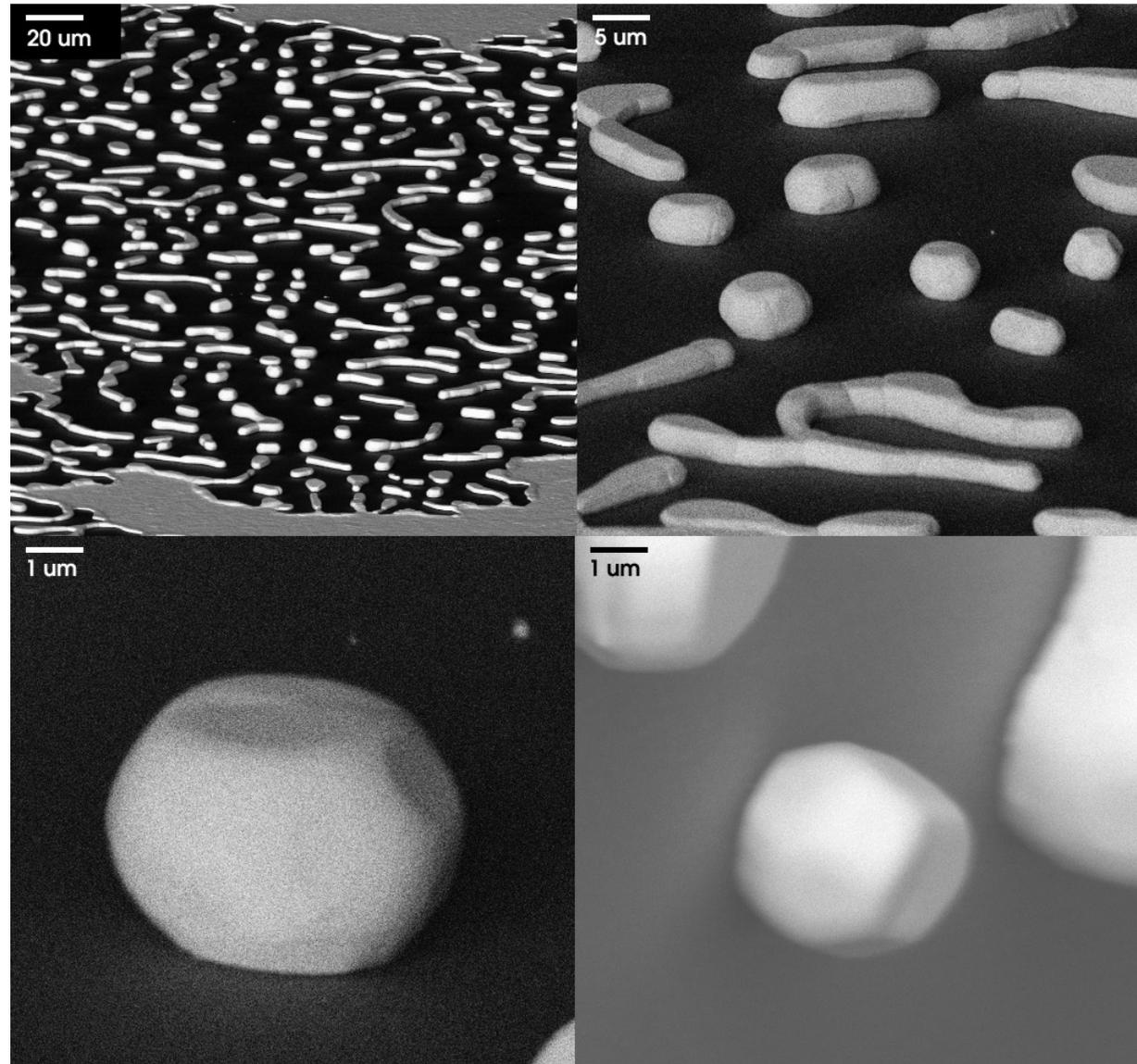


“Stacheln”

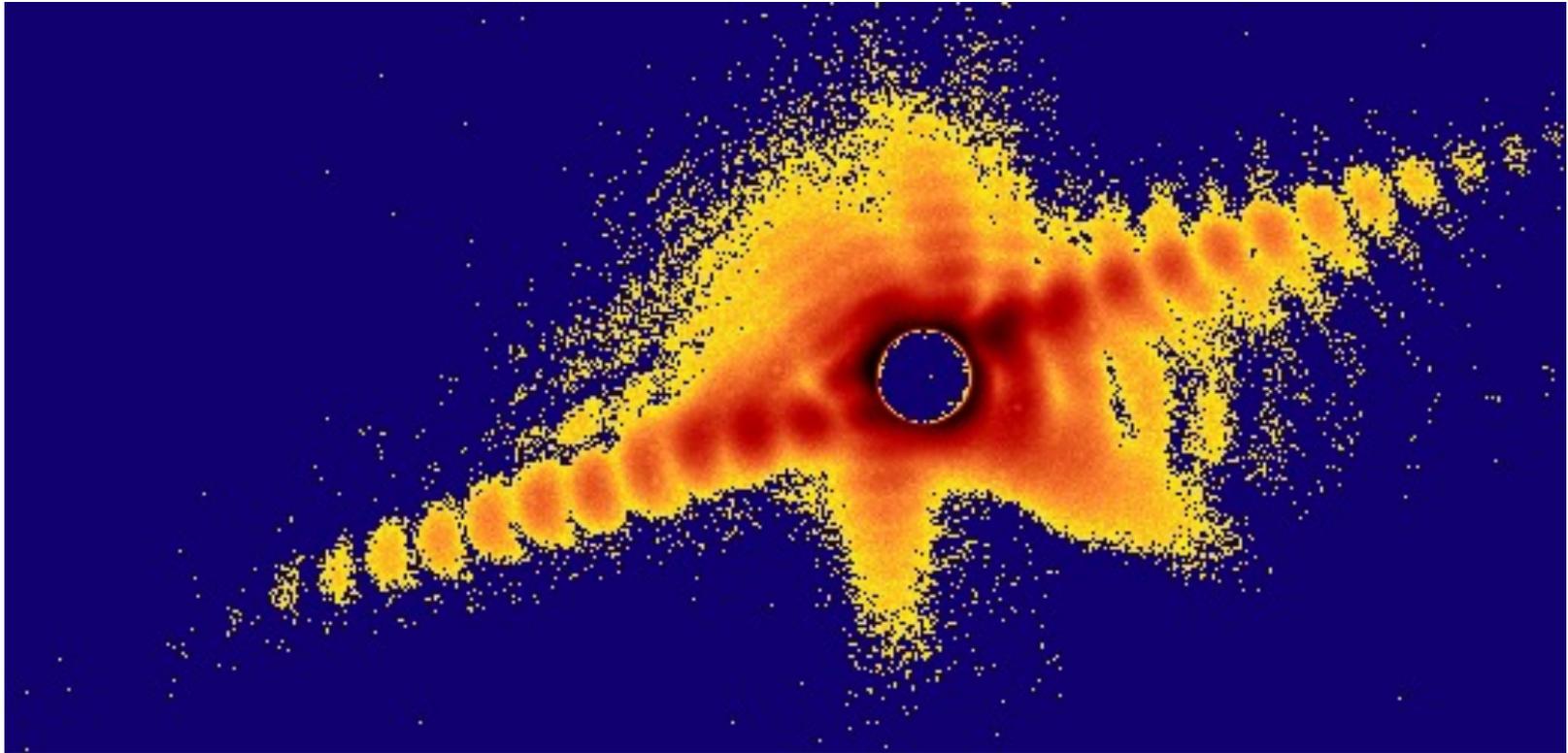


SEMS

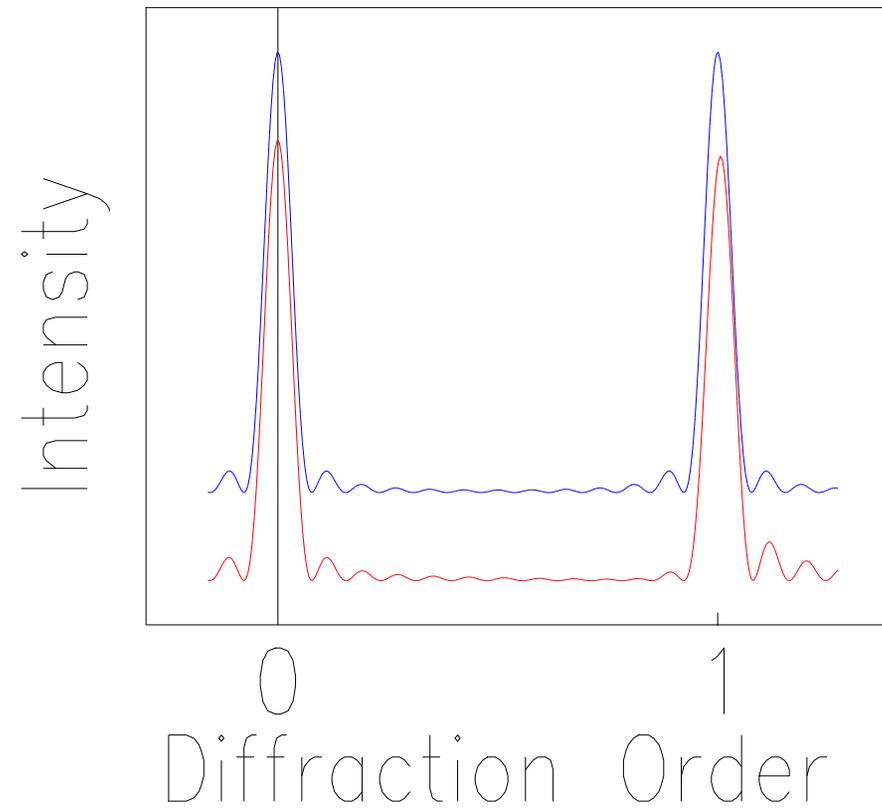
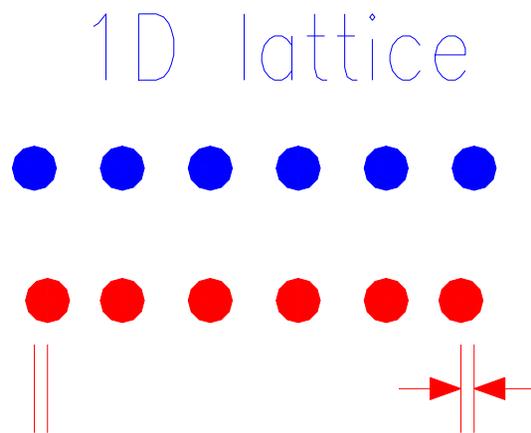
- Au blanket film
- Quartz substrate
- Annealed at 950°C for 70 hrs.



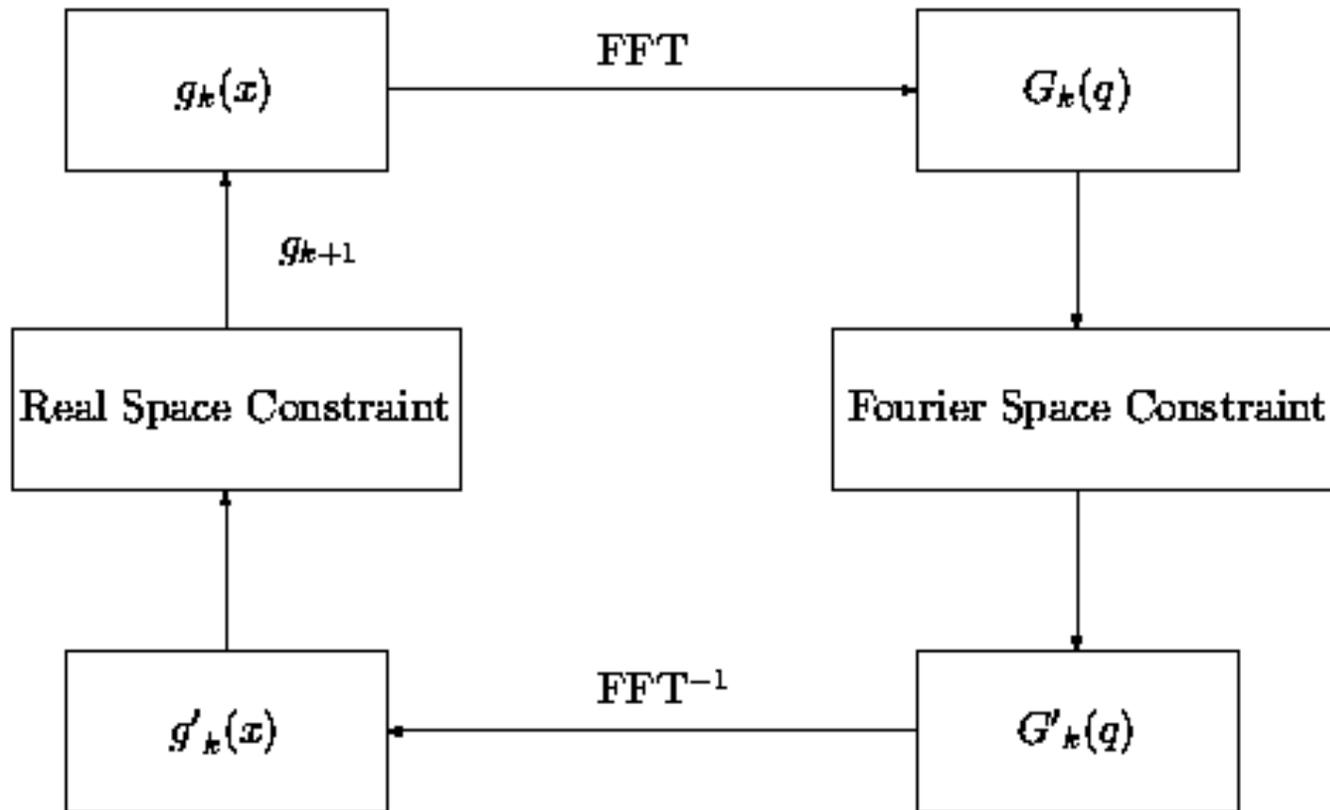
Micron-sized gold crystal: (111) Bragg reflection



Imaging of Lattice Strains

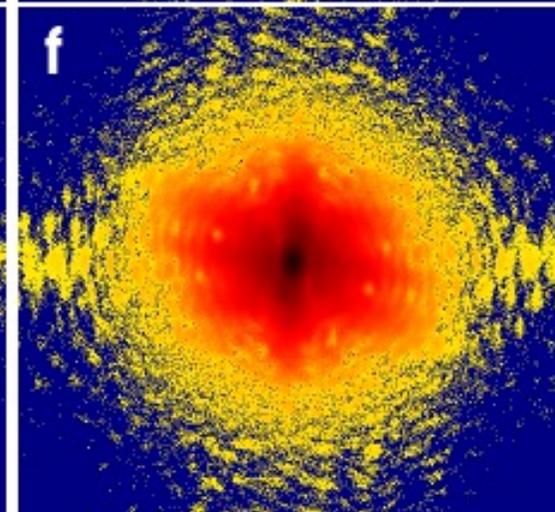
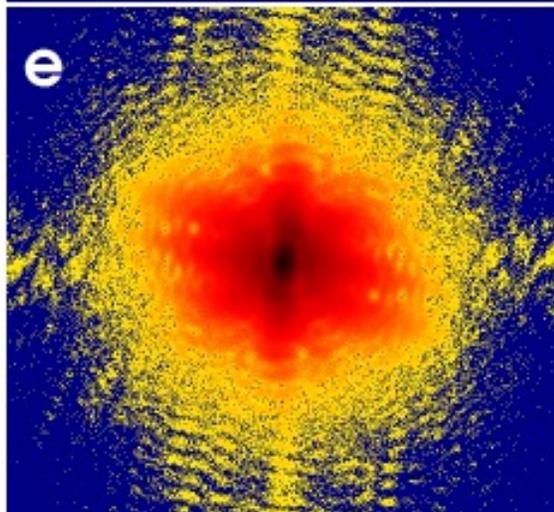
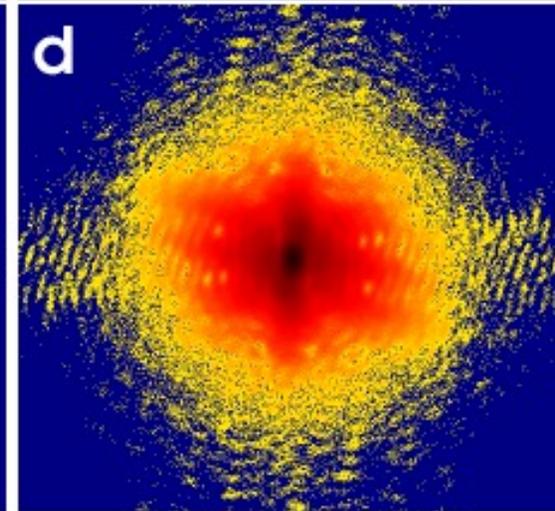
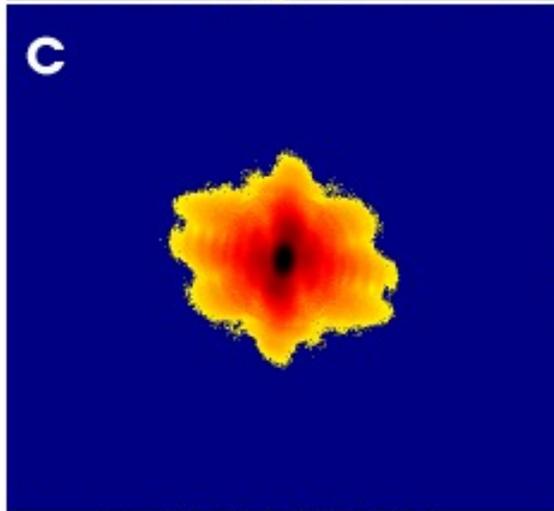
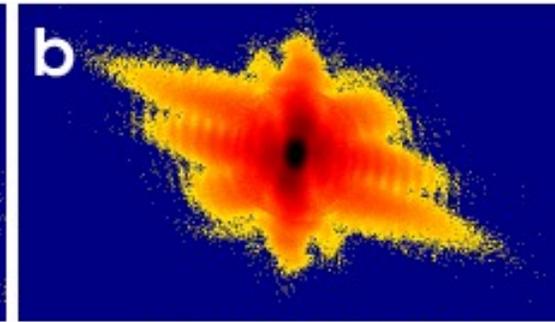
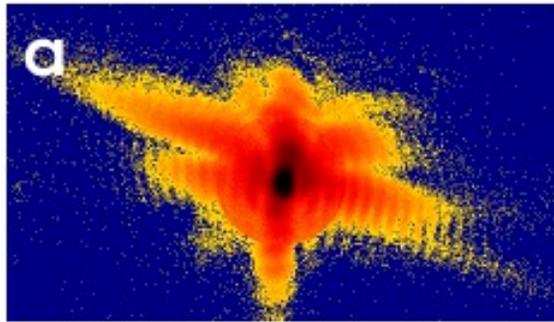


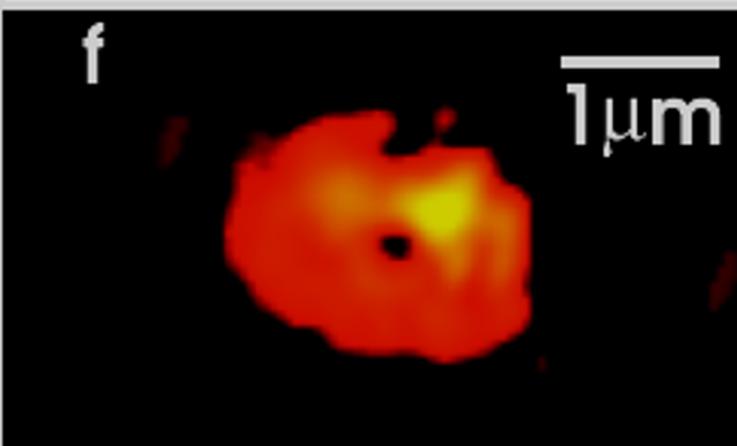
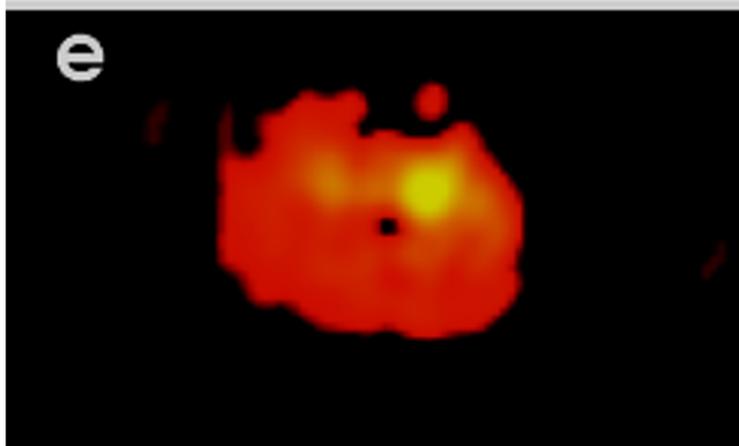
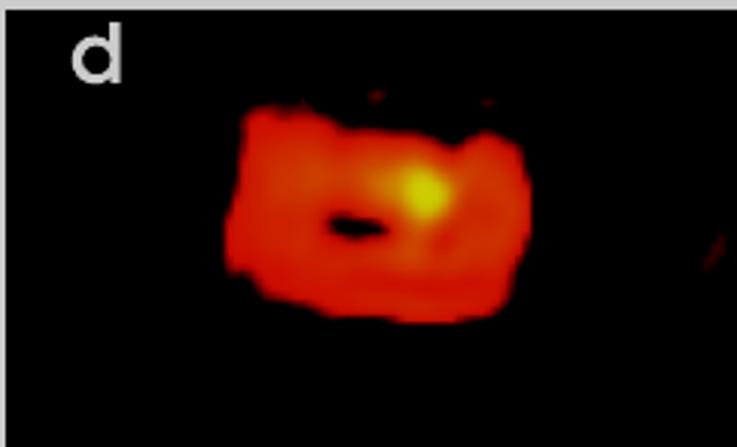
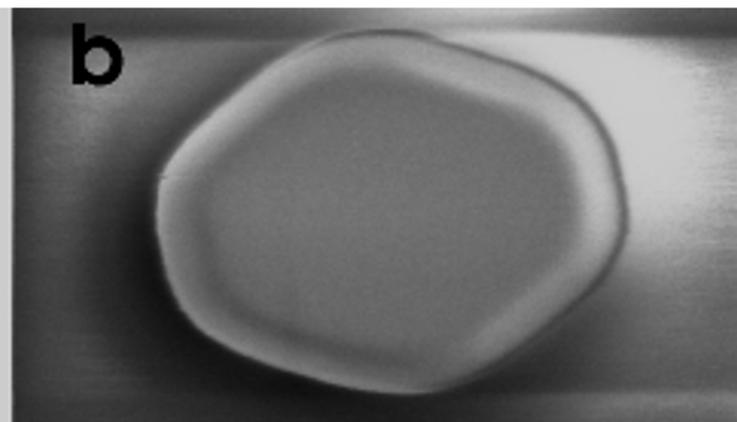
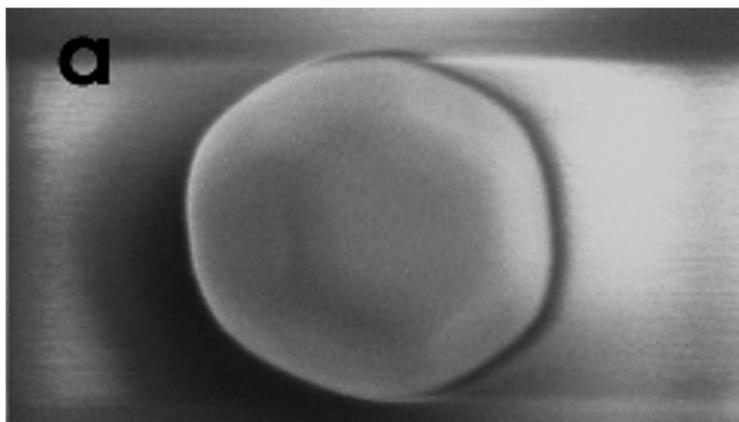
Generic “Error Reduction” method



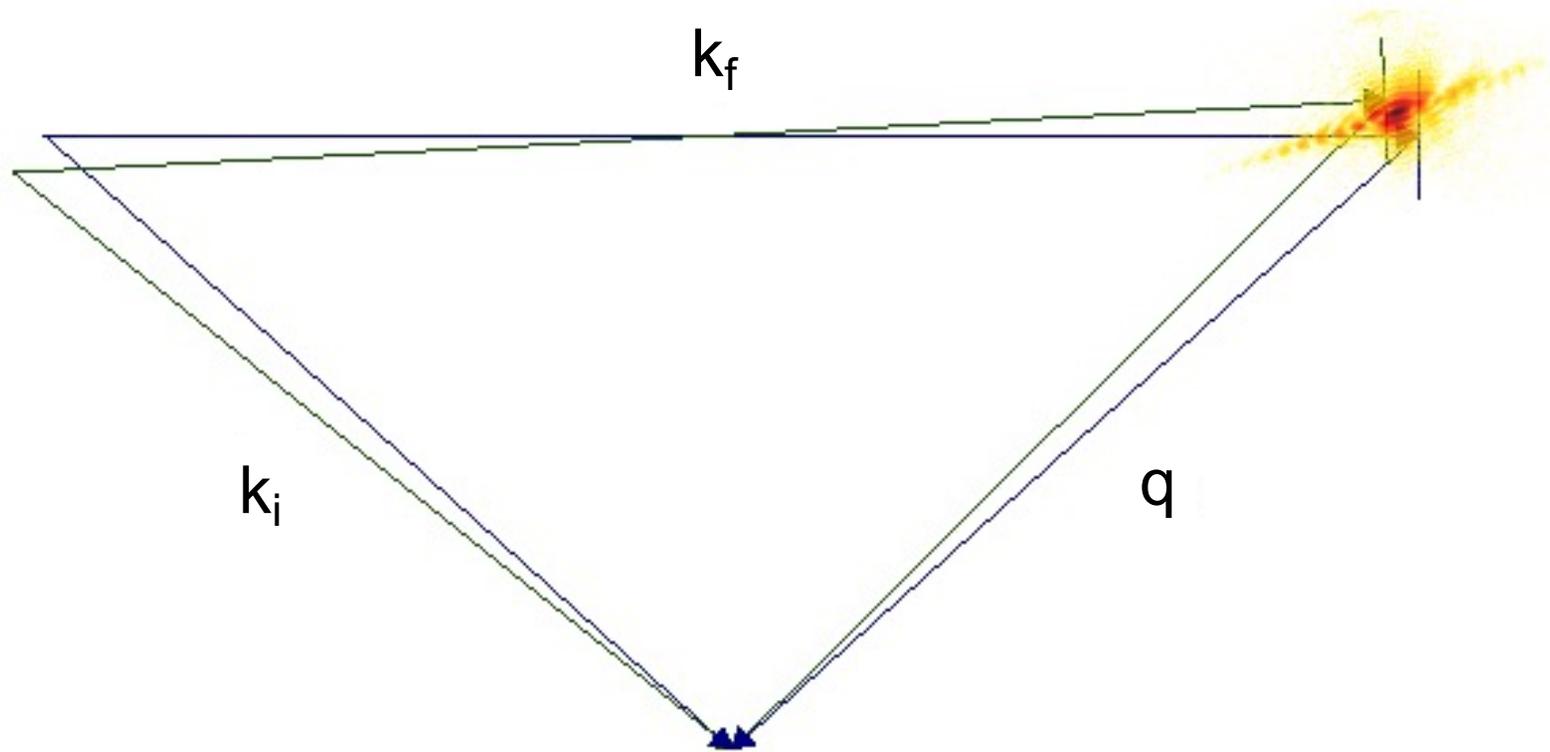
J. R. Fienup *Appl. Opt.* 21 2758 (1982)

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

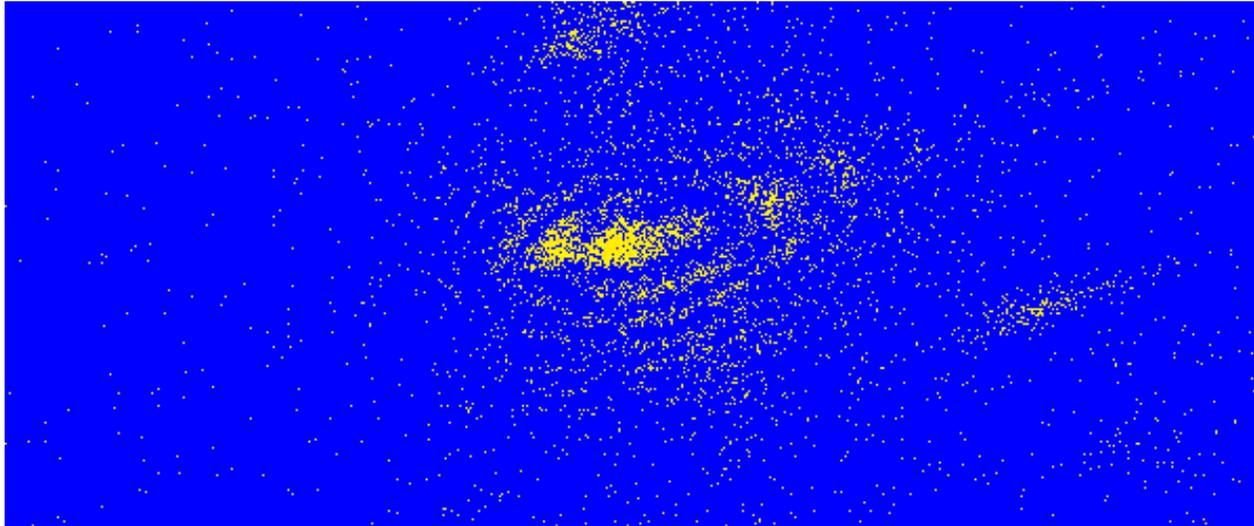




3D Diffraction Method

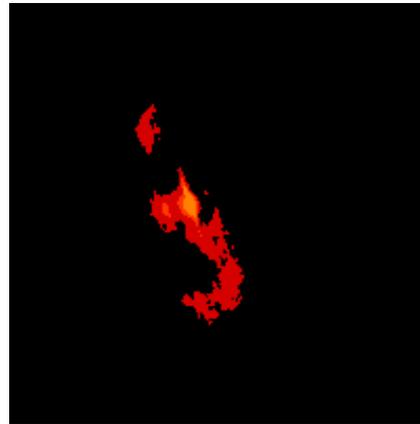


3D Diffraction Data 1 micron Au crystal

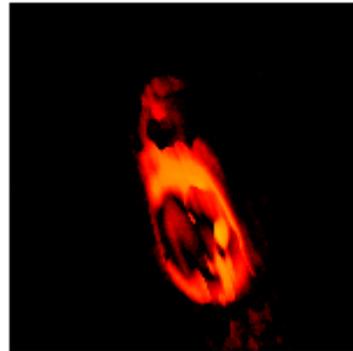
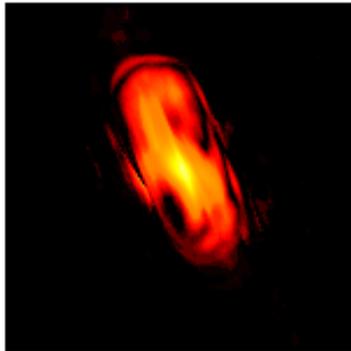
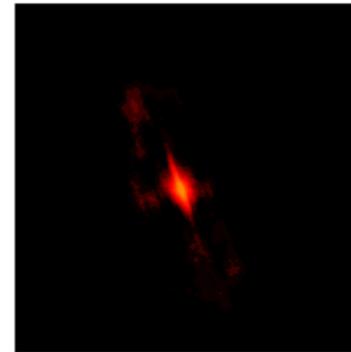
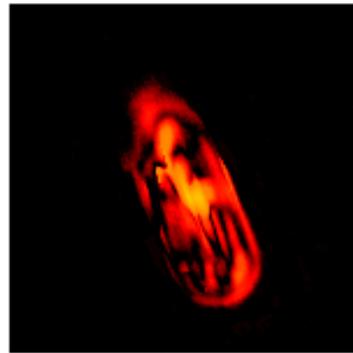
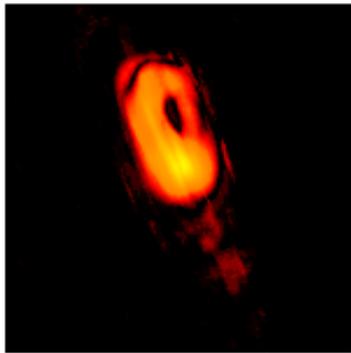
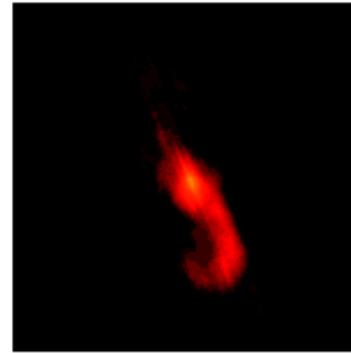
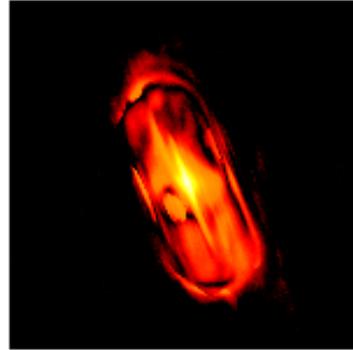
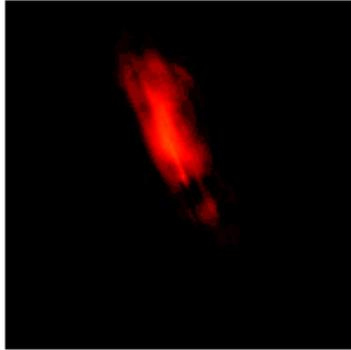
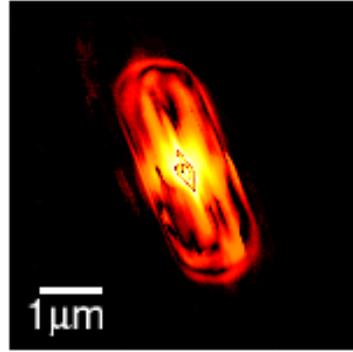
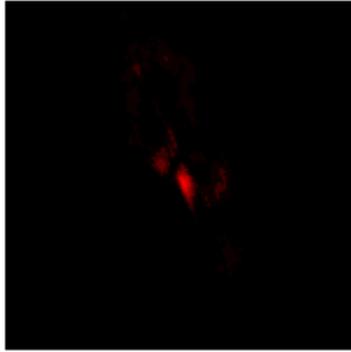


* Center is Symmetric *

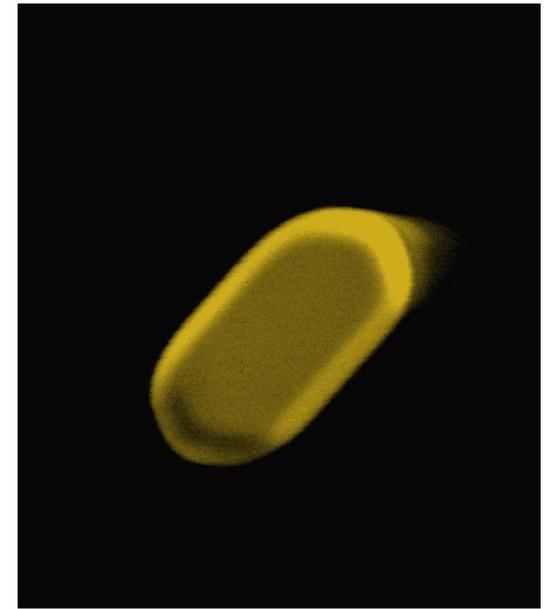
30 frames of 3D reconstruction 1 micron gold crystal

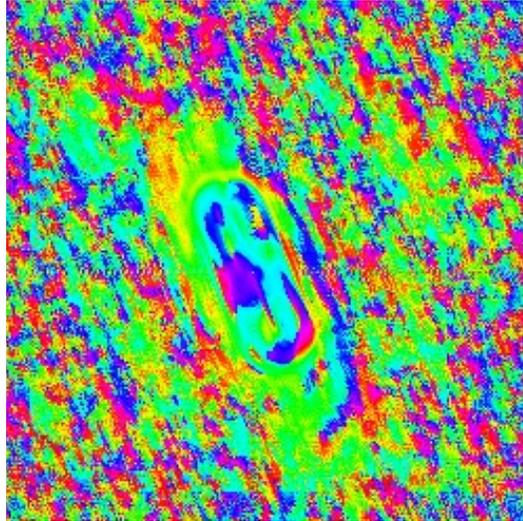
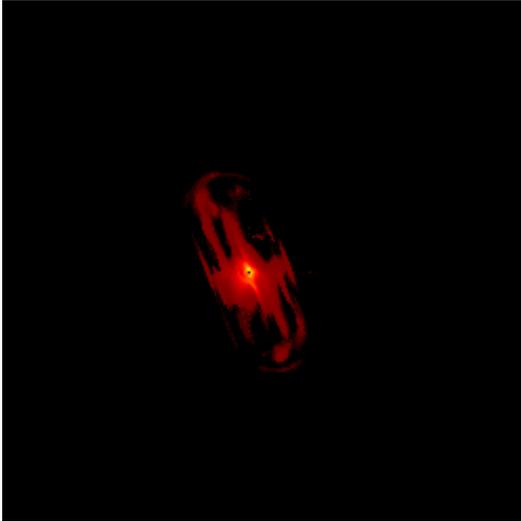
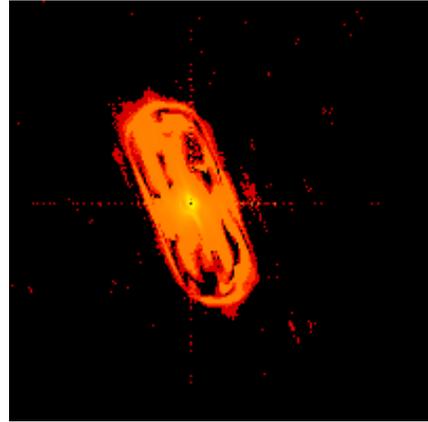
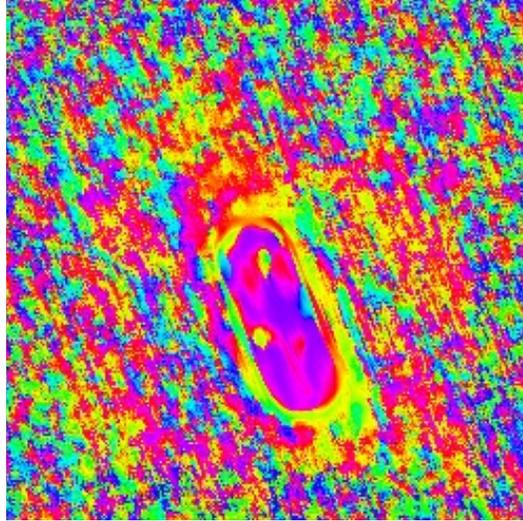
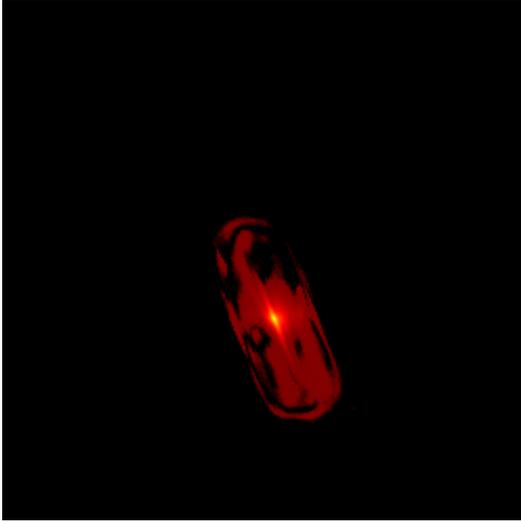


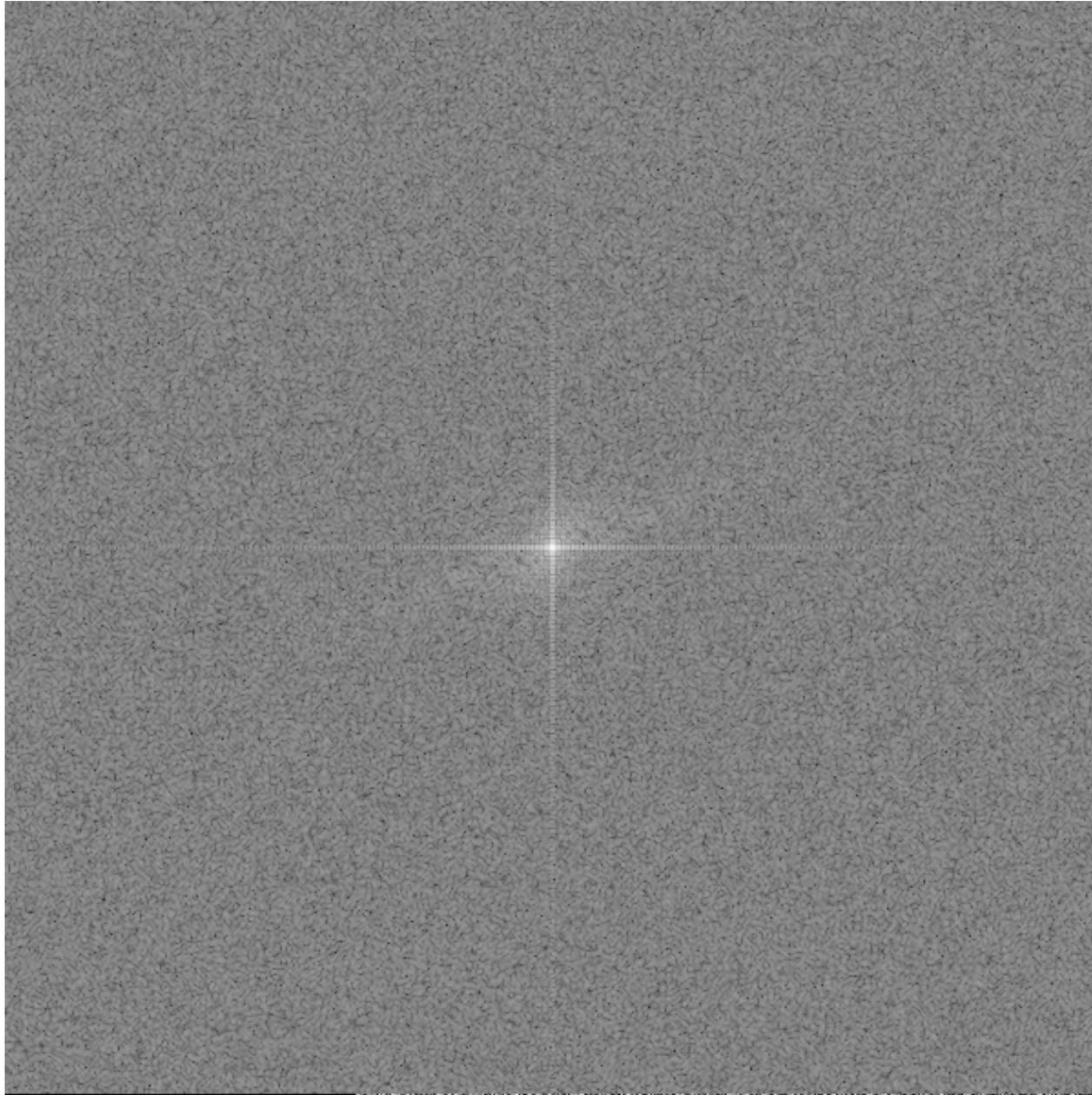
* CENTER *

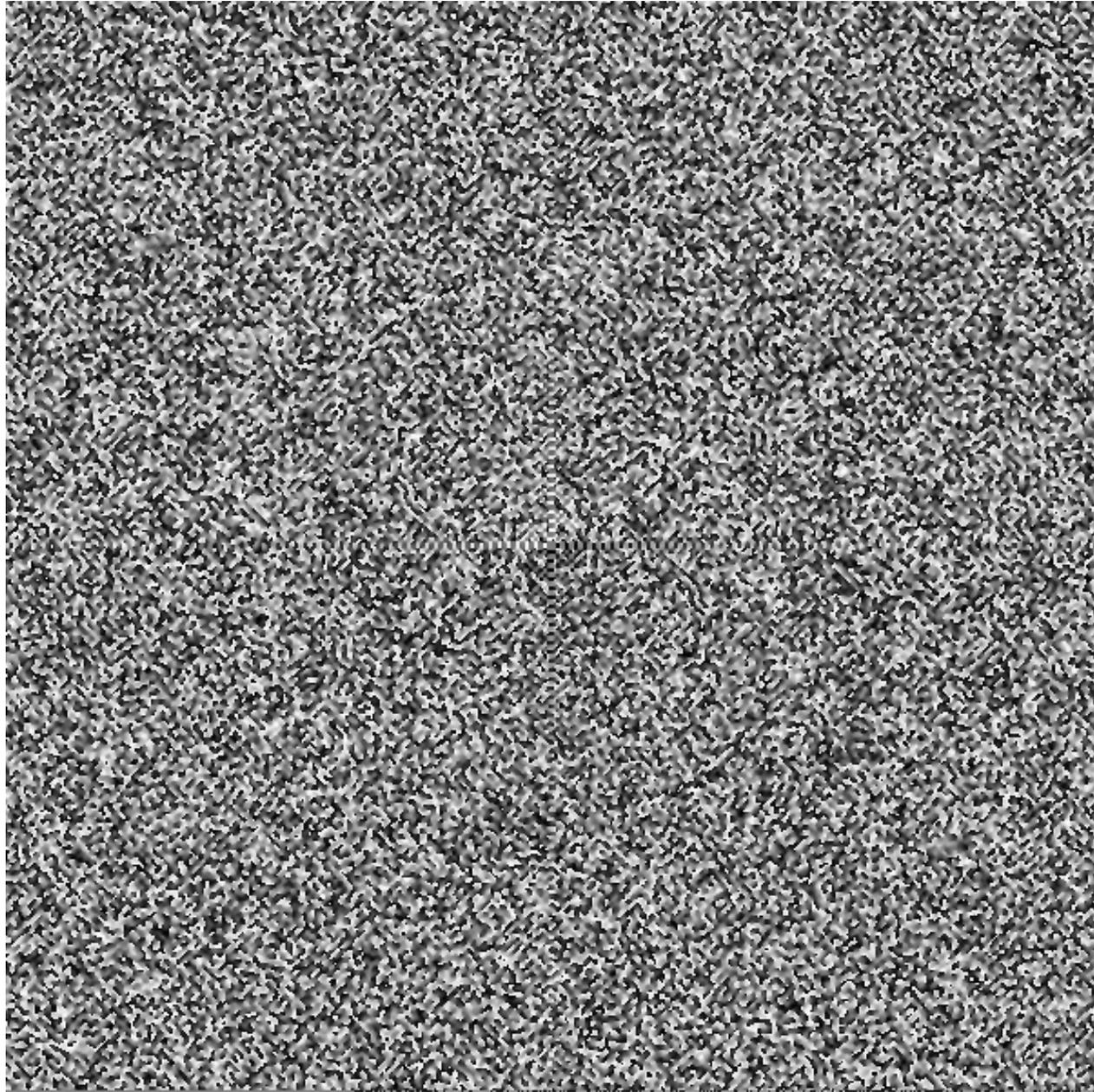


Shape is similar to plan view SEM:

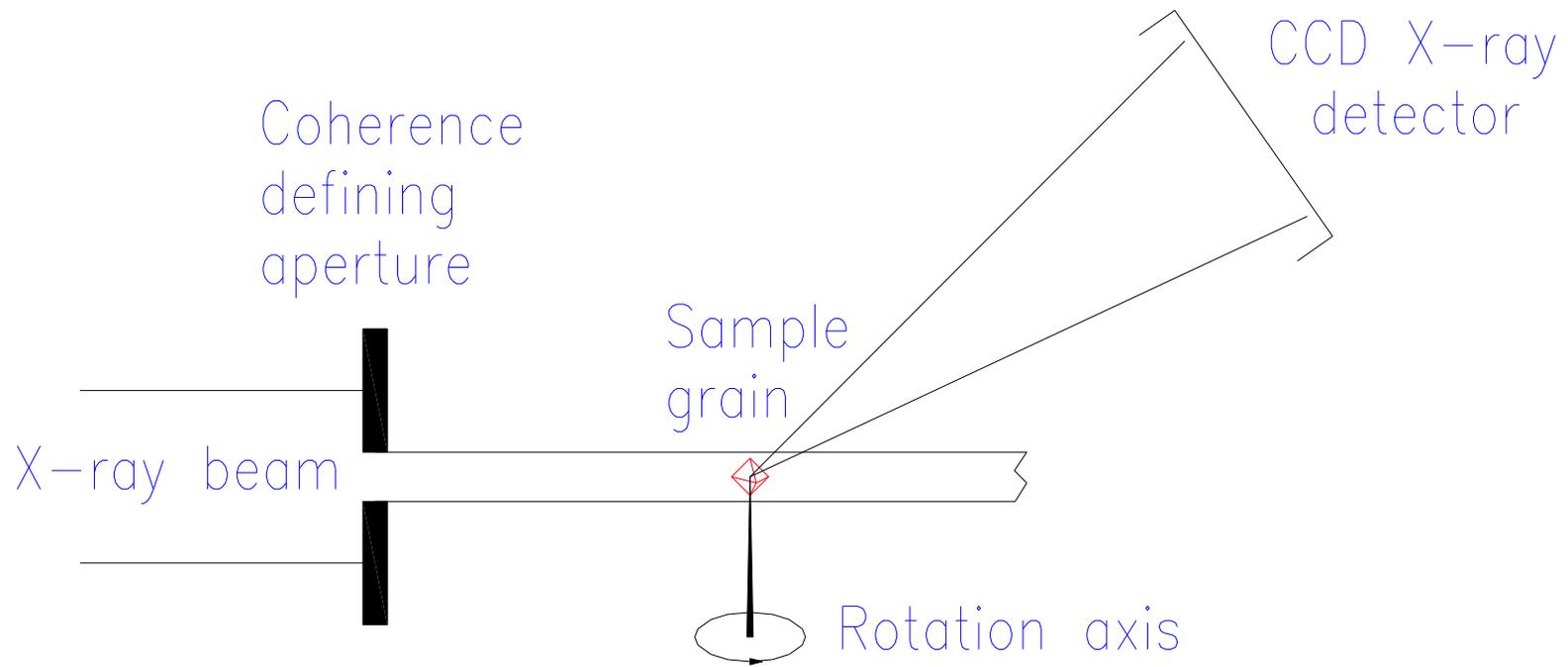








Lensless X-ray Microscope



UI research gets more crystal clear

■ X-ray beam gives close look at solids, liquids

By **GREG KLINE**
News-Gazette Staff Writer

Examine a sheet of steel, and you perceive a smooth surface. But down inside, near the atomic level, are multi-faceted crystalline structures, not unlike the much bigger crystals you can see with your eyes and turn into desk ornaments and chandeliers.

The nature of those microscopic structures is what makes metal strong.

Proteins, which have significant roles in biological processes, also can form crystals. Silicon, the base component of computer chips and other microelectronics, is full of them as well.

A greater understanding of how these tiny crystalline structures form and perform would be useful to everyone from structural biologists to steel makers.

Some University of Illinois scientists have developed a technique that should help. Their solution: Give the micro-

scopic crystals an X-ray.

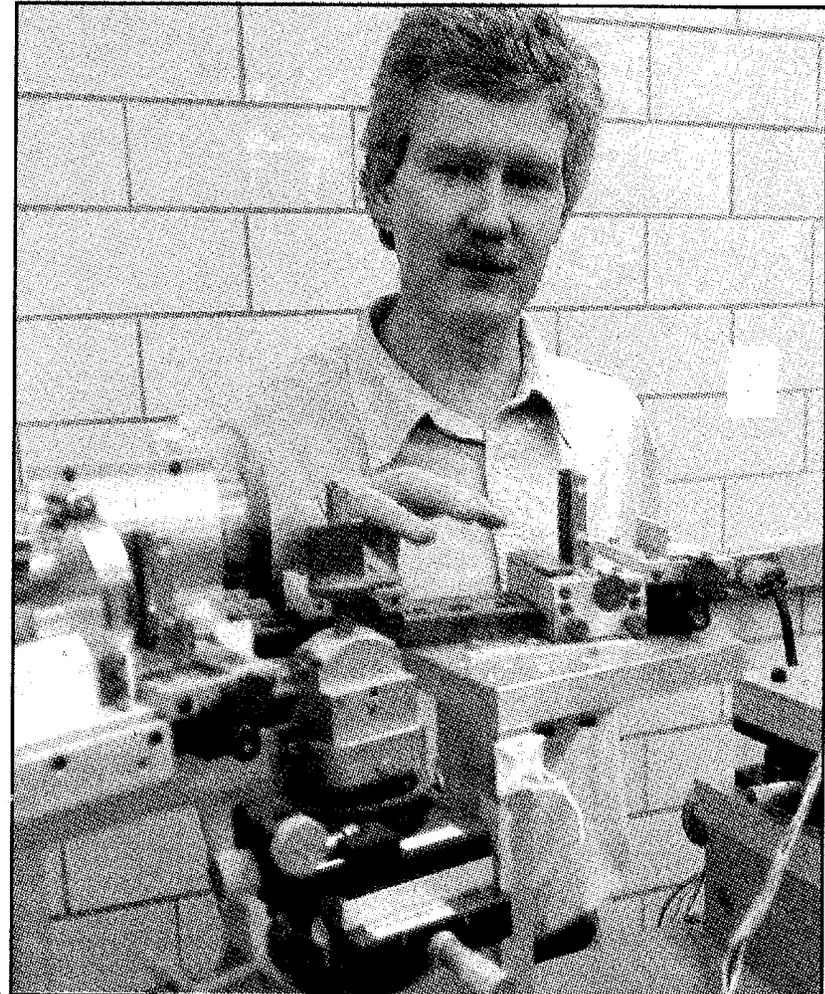
An X-ray microscope developed by UI researchers can make detailed, three-dimensional images of crystals formed inside both solids and liquids.

"You can find the positions of all the atoms inside," UI physics Professor Ian Robinson said recently. "We're getting additional measurements (than other methods). We're interpreting it more finely."

Robinson developed the microscope with graduate students Garth Williams, Mark Pfeifer and Sebastien Boutet and Ivan Vartanyants, a visiting scientist at the UI, using a high-powered X-ray beam generated at Argonne National Laboratory near Chicago. The National Science Foundation, U.S. Energy Department and state of Illinois funded the work.

The microscope isn't the kind where you peer through an eyepiece at something on a slide of glass below. It's lensless and it creates images on a computer screen using software routines developed by the UI researchers.

The images are formed by interpreting signals from the



News-Gazette photo by Robert K. O'Daniell

University of Illinois physics Professor Ian Robinson is using the X-ray microscope to study what happens when metals are cut and when they're ground together in low-temperature milling.

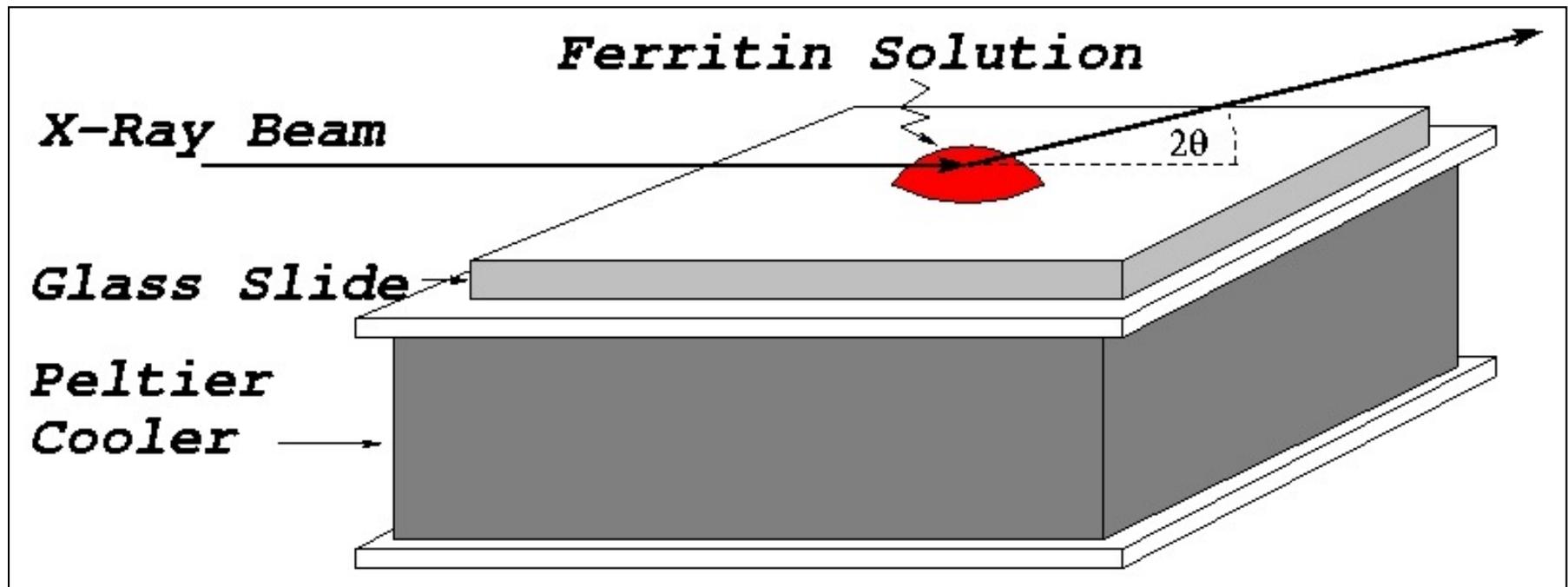
X-rays and collected with an electronic charge-coupled device similar to those used in digital cameras.

"You shine X-rays in a sam-

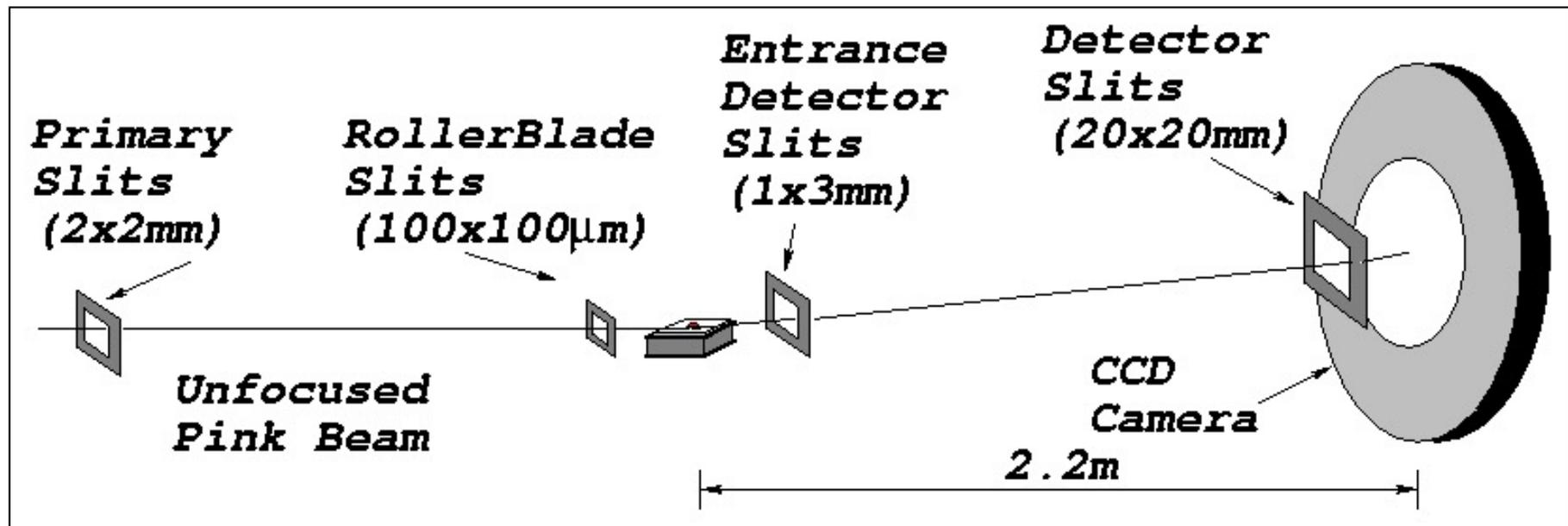
ple and you collect everything that is scattered, the light that is scattered by the sample,"

Please see RESEARCH, B-2

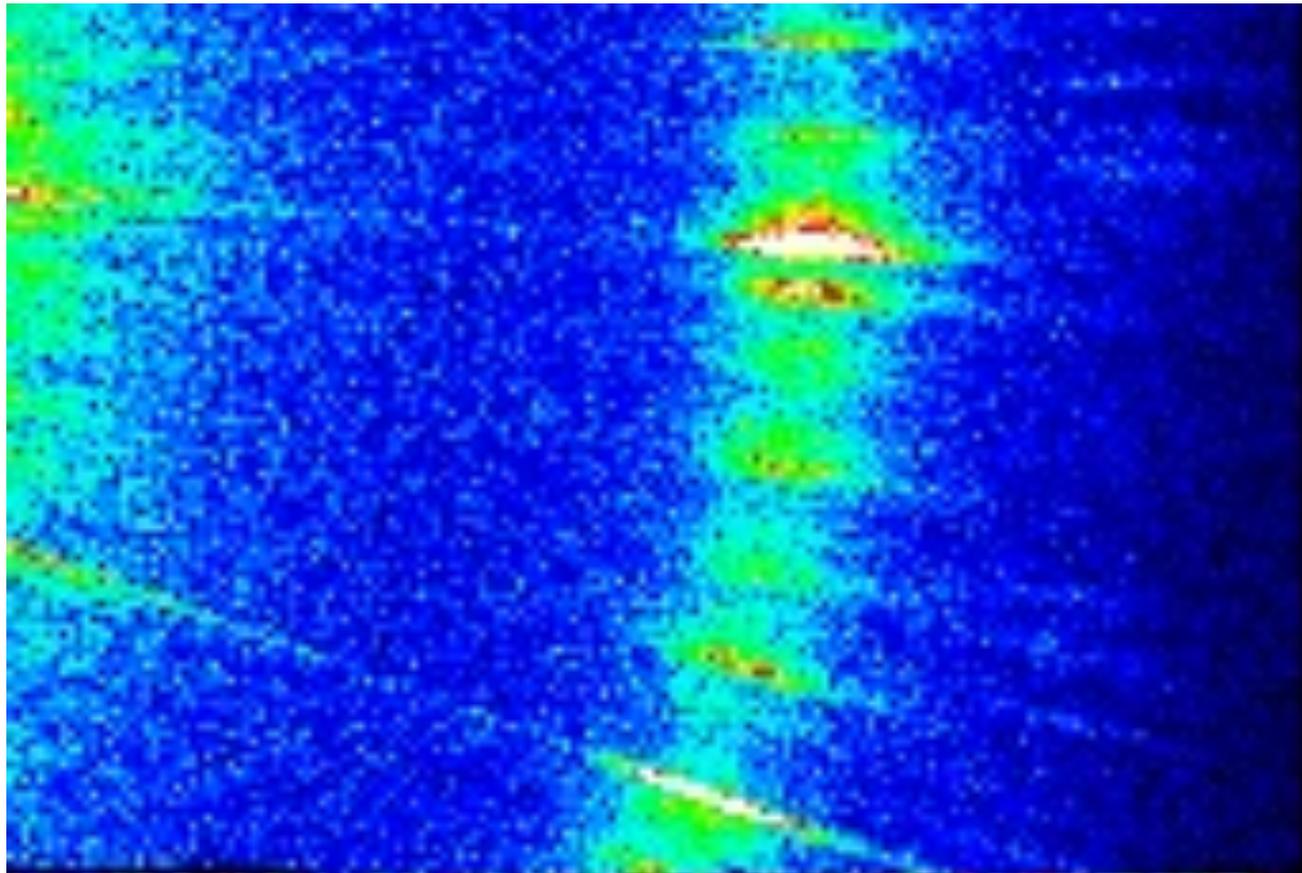
In-situ Study of Crystallization



Experiment at APS Sector 34

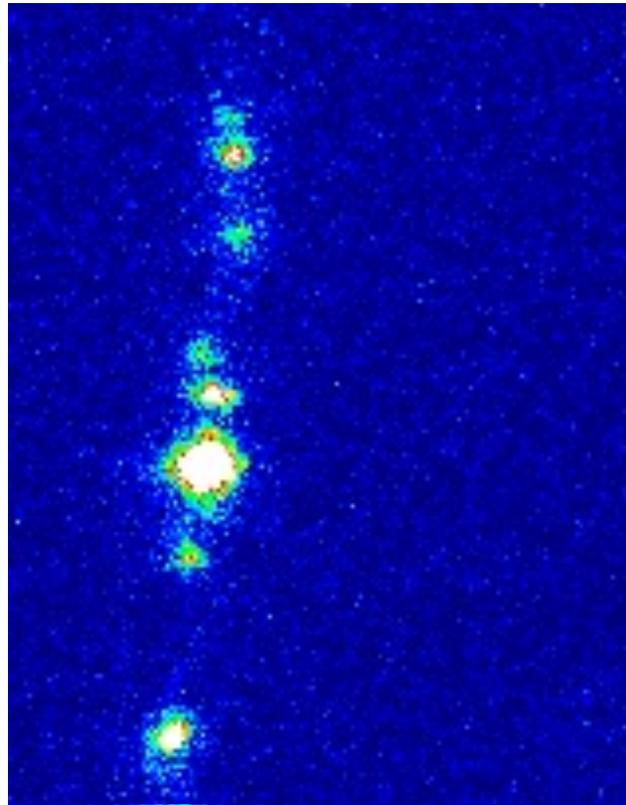


“Pink beam” sees CTRs

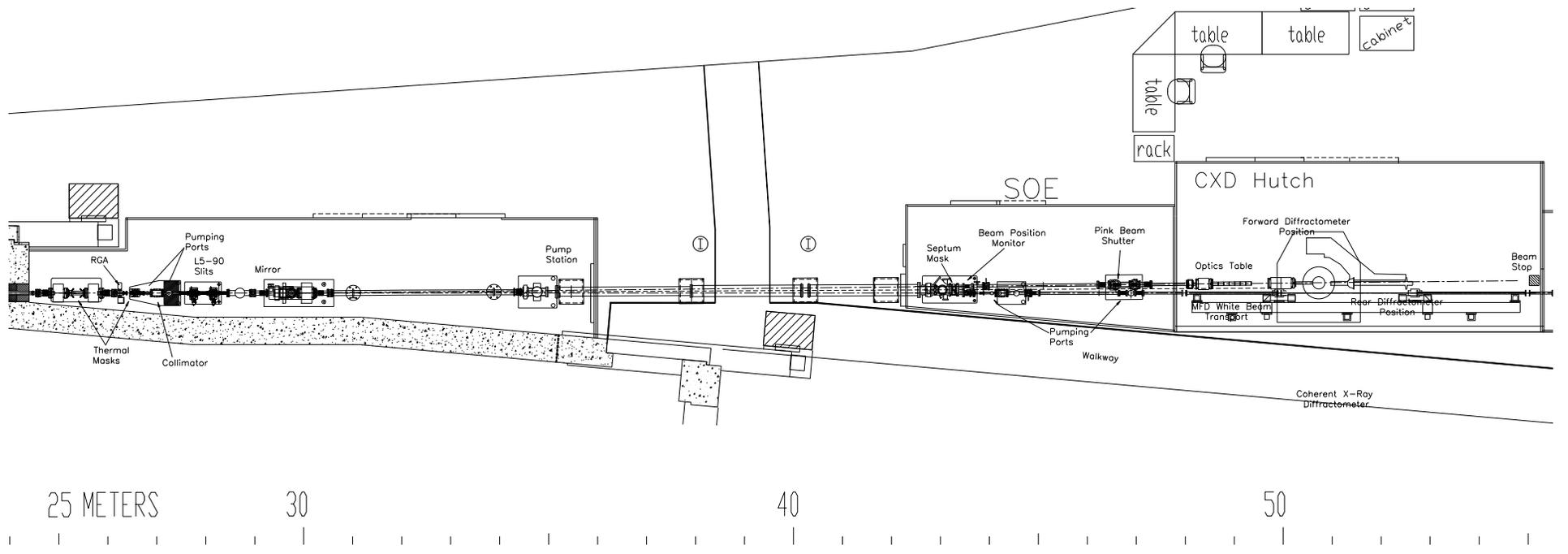


Ferritin (111) Powder Ring

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

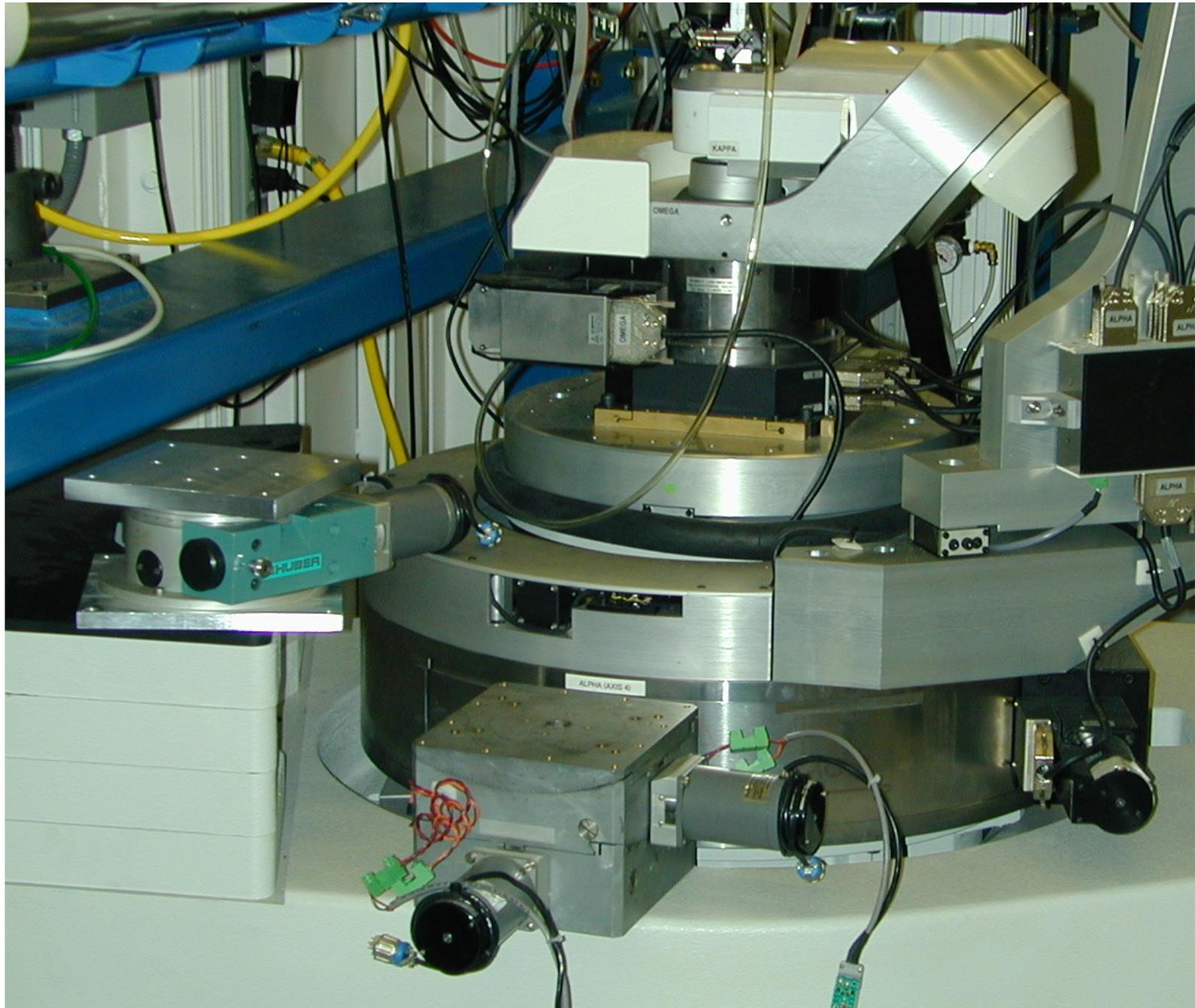


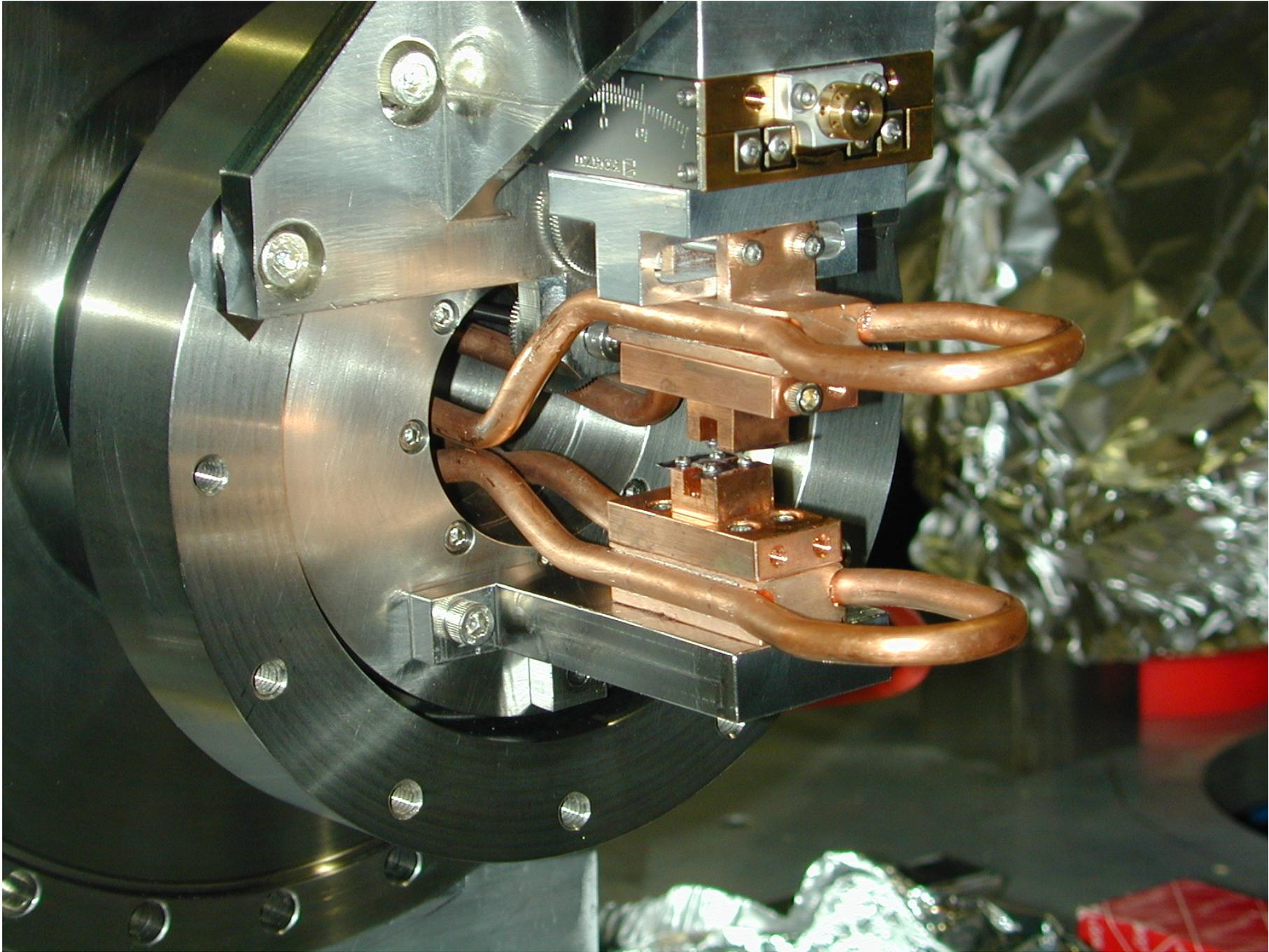
CXD Beamline at APS Sector 34

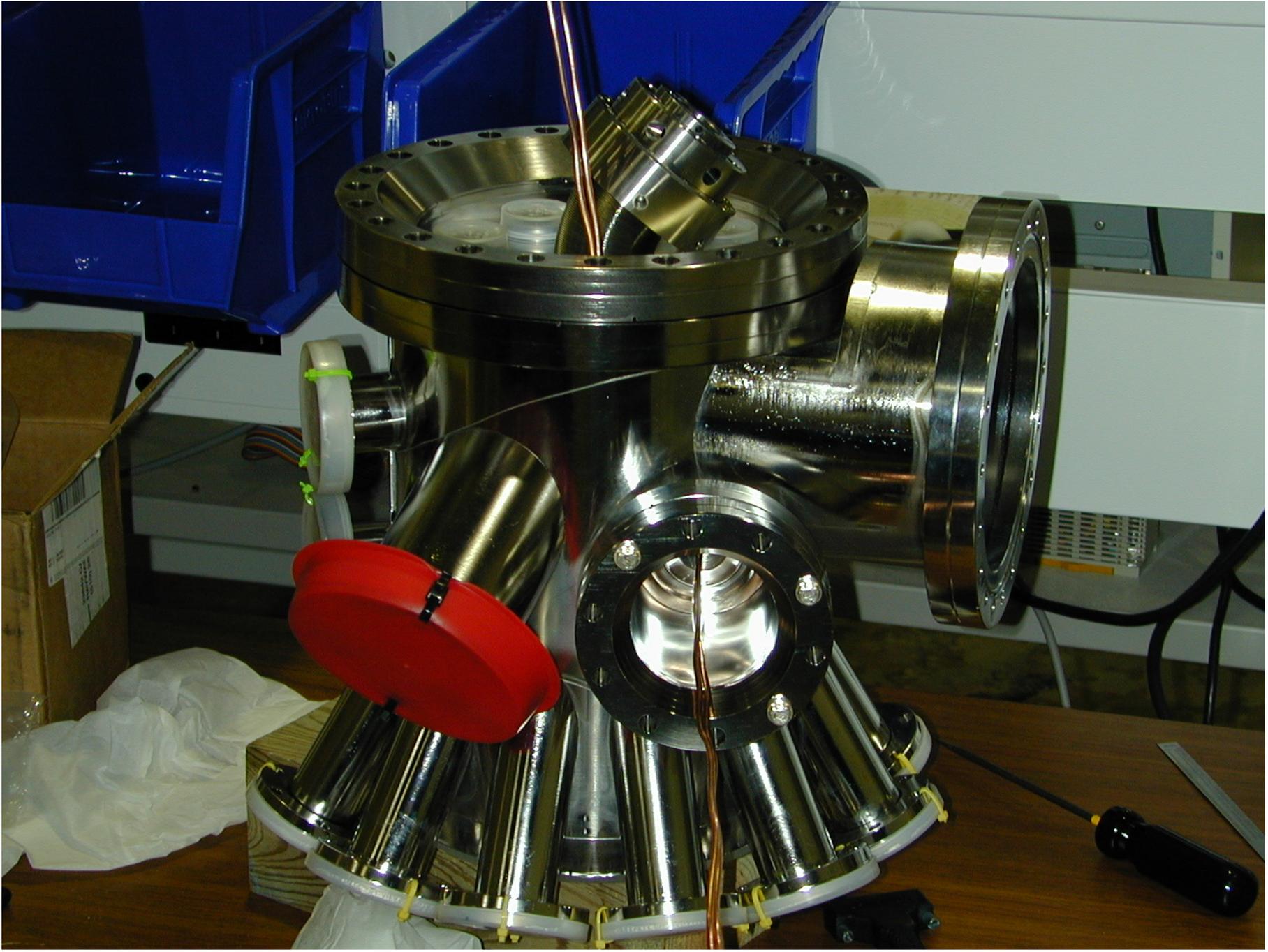


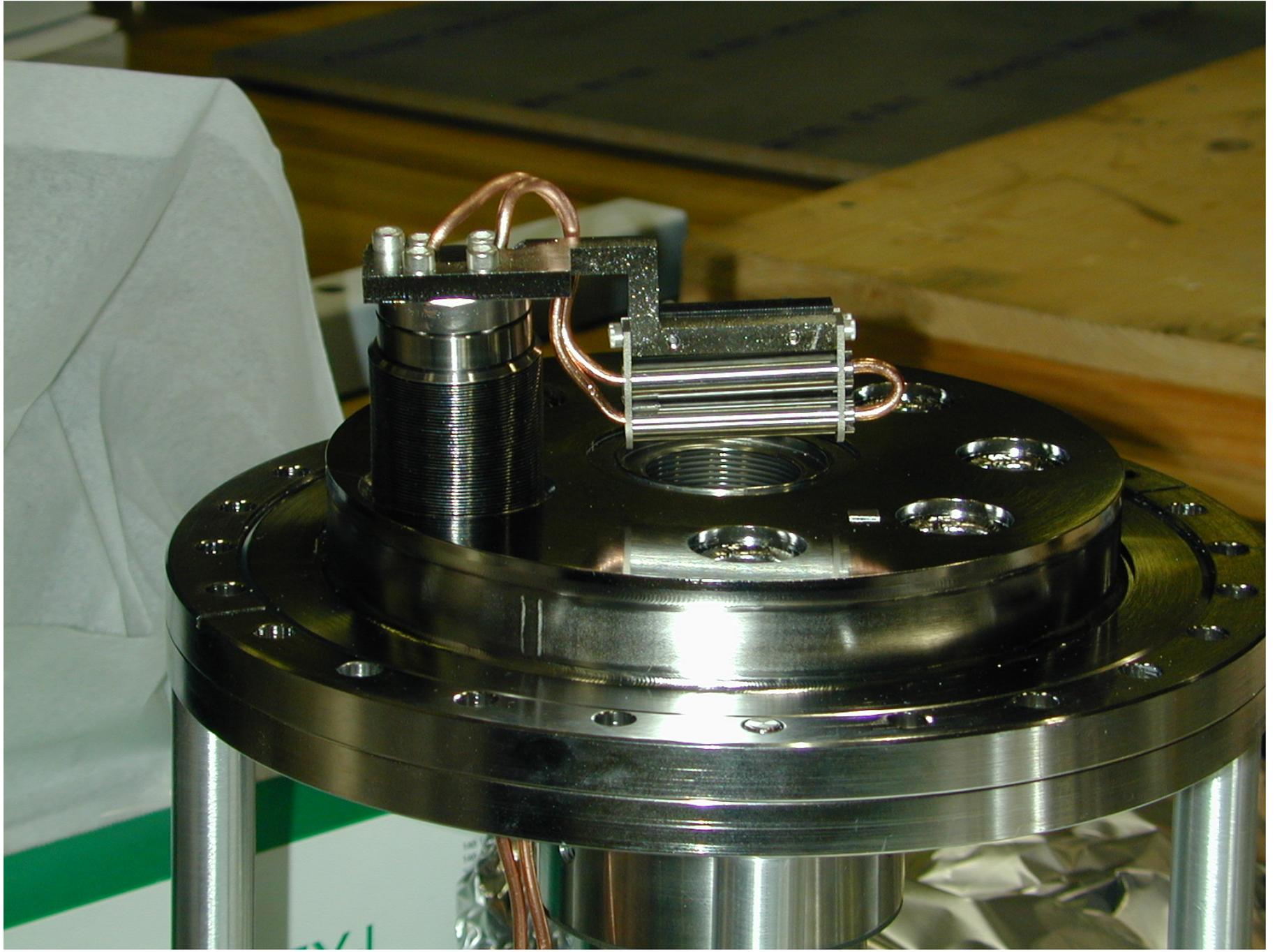












Conclusions

- “Diffuse” scattering acquires fine structure
- Surface/interface morphology
- Shapes of small particles
- CXD facility ready for experiments
- Potential applications
 - Atomic-scale fluctuations
 - Imaging of strain fields