

Coherence and Crystallography

Ian Robinson London Centre for Nanotechnology

Felisa Berenguer Diamond Light Source

Richard Bean

Steven Leake

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Fucaï Zhang

April 2010

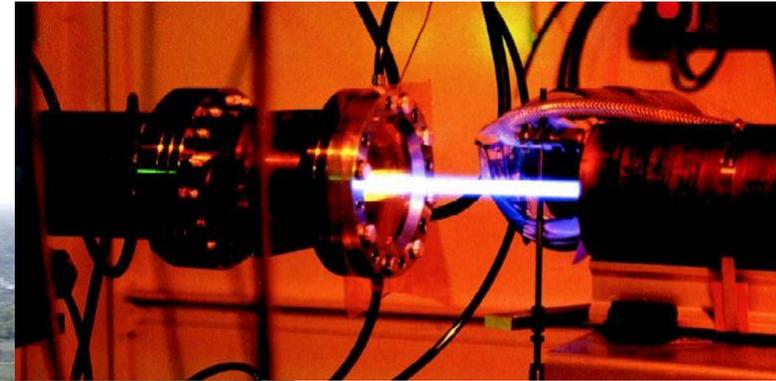
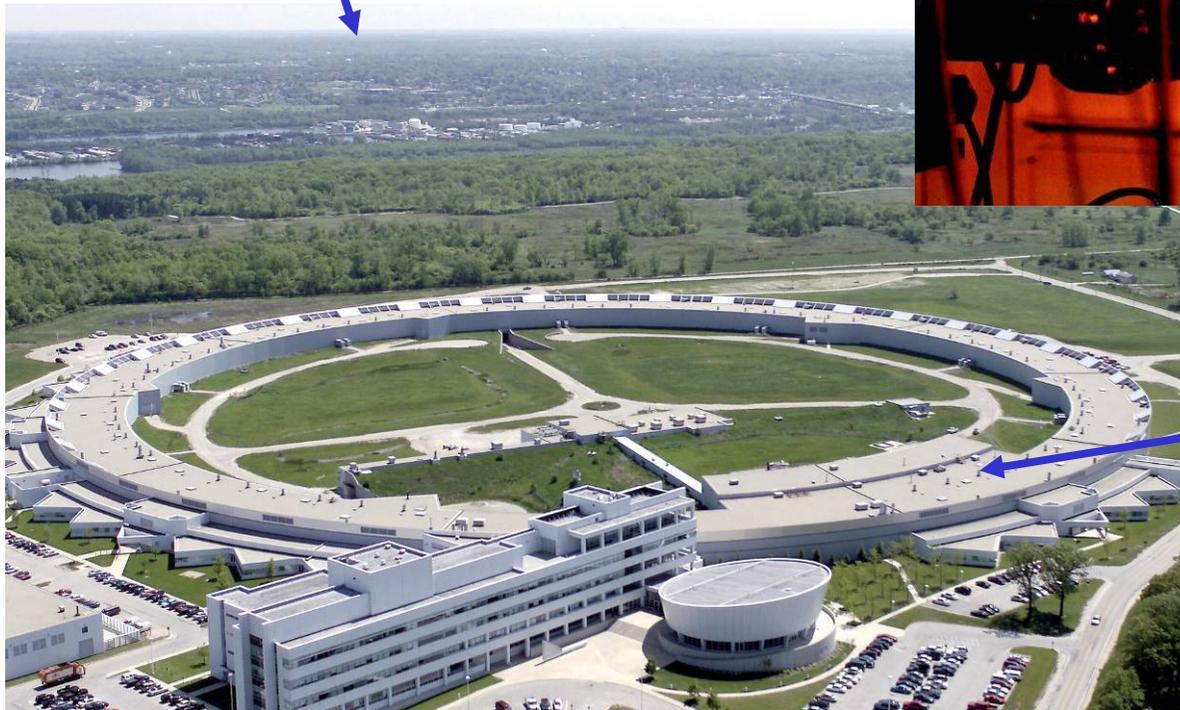
Marcus Newton

Outline

- Coherent X-ray Diffraction
- Synchrotron Source of X-rays
- Coherence
- Solving the phase problem
- Simulation of small crystals with strain
- Wired ZnO experiments

Synchrotron Radiation

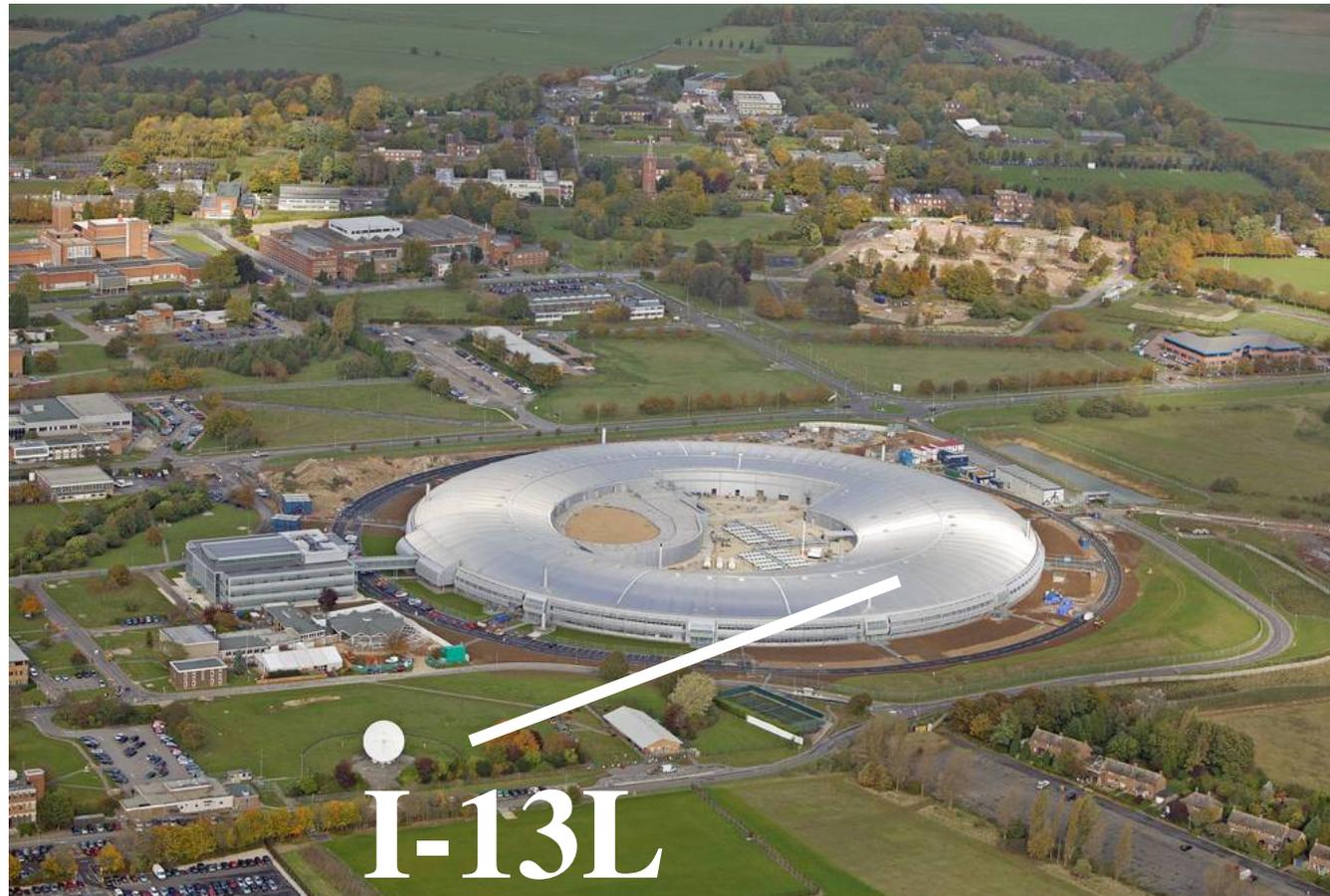
Urbana



34-ID-C



Diamond Light Source (RAL)

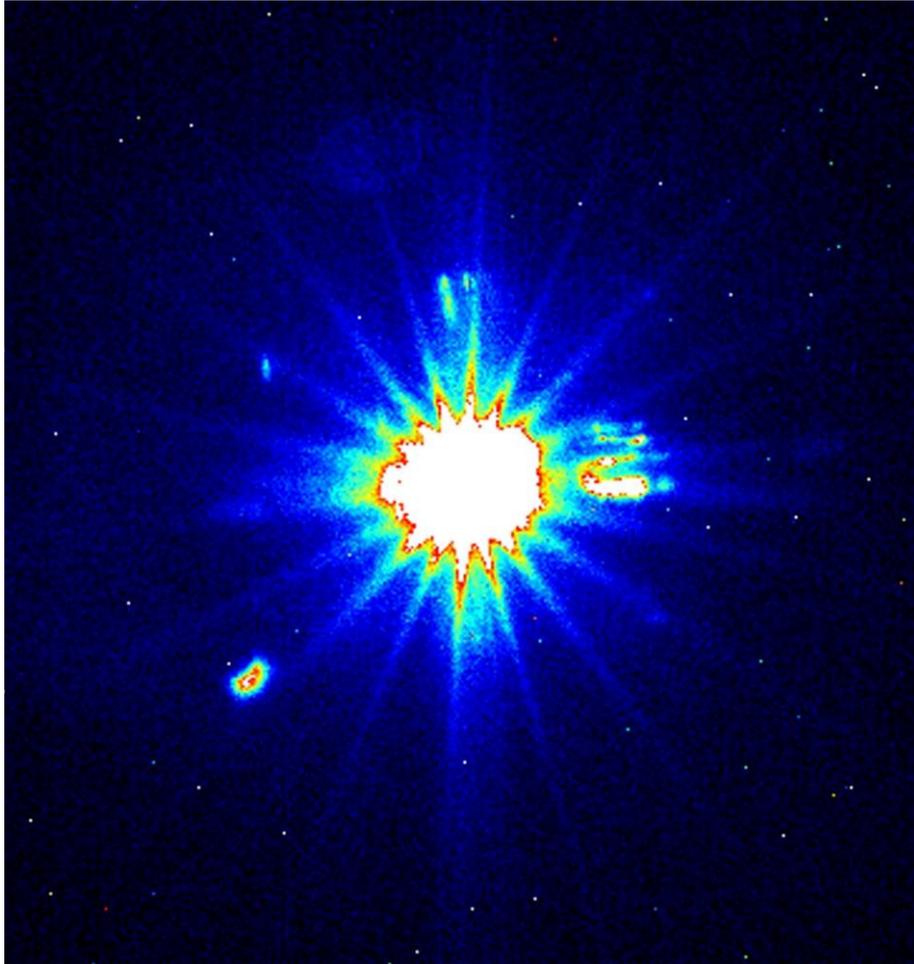


Last Girder – March 2006

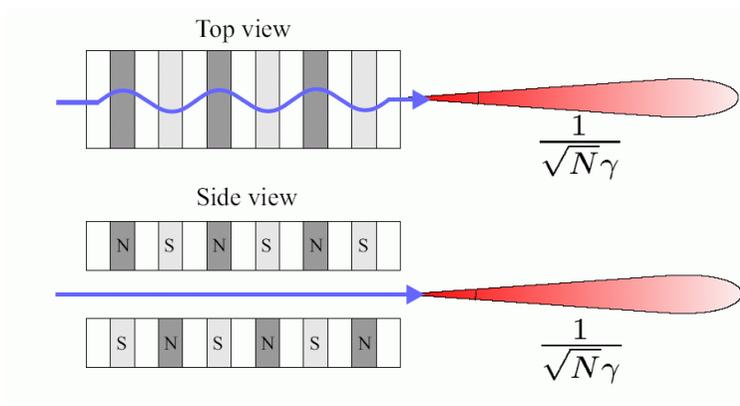


“First Light” - May 2006

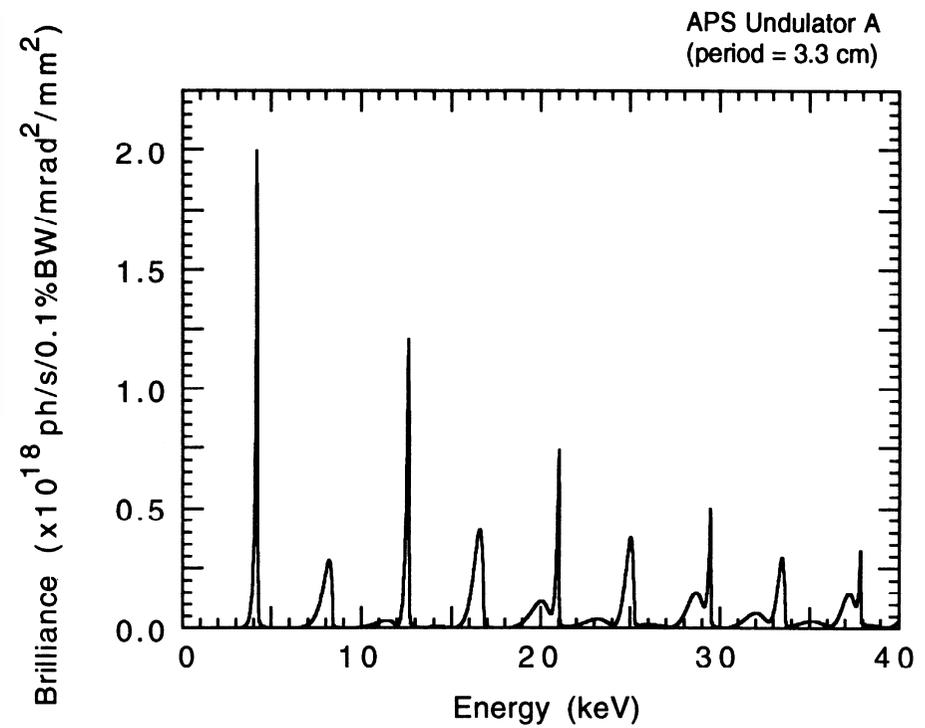
ramped to 3GeV – Sept 2006



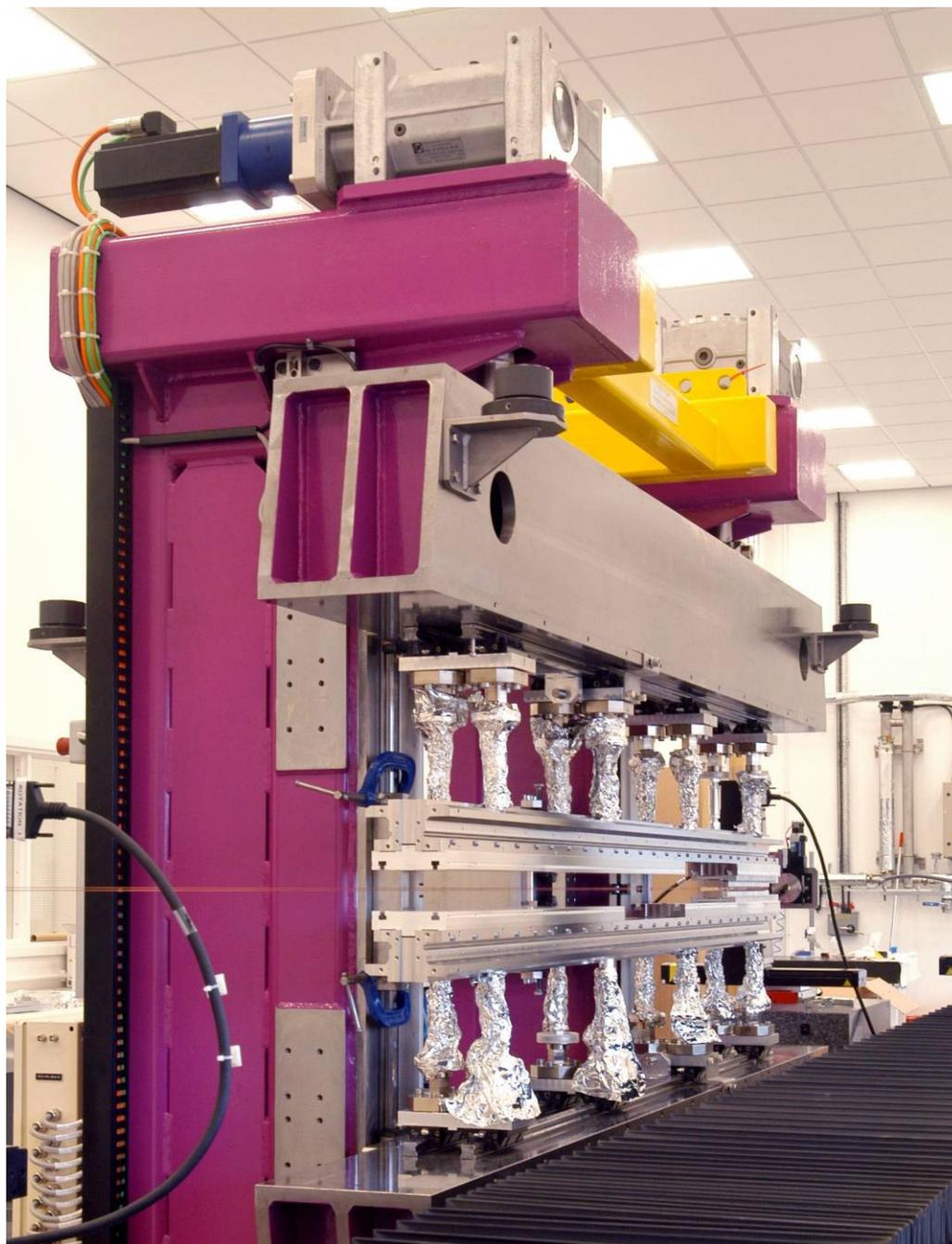
X-ray Undulator Principle



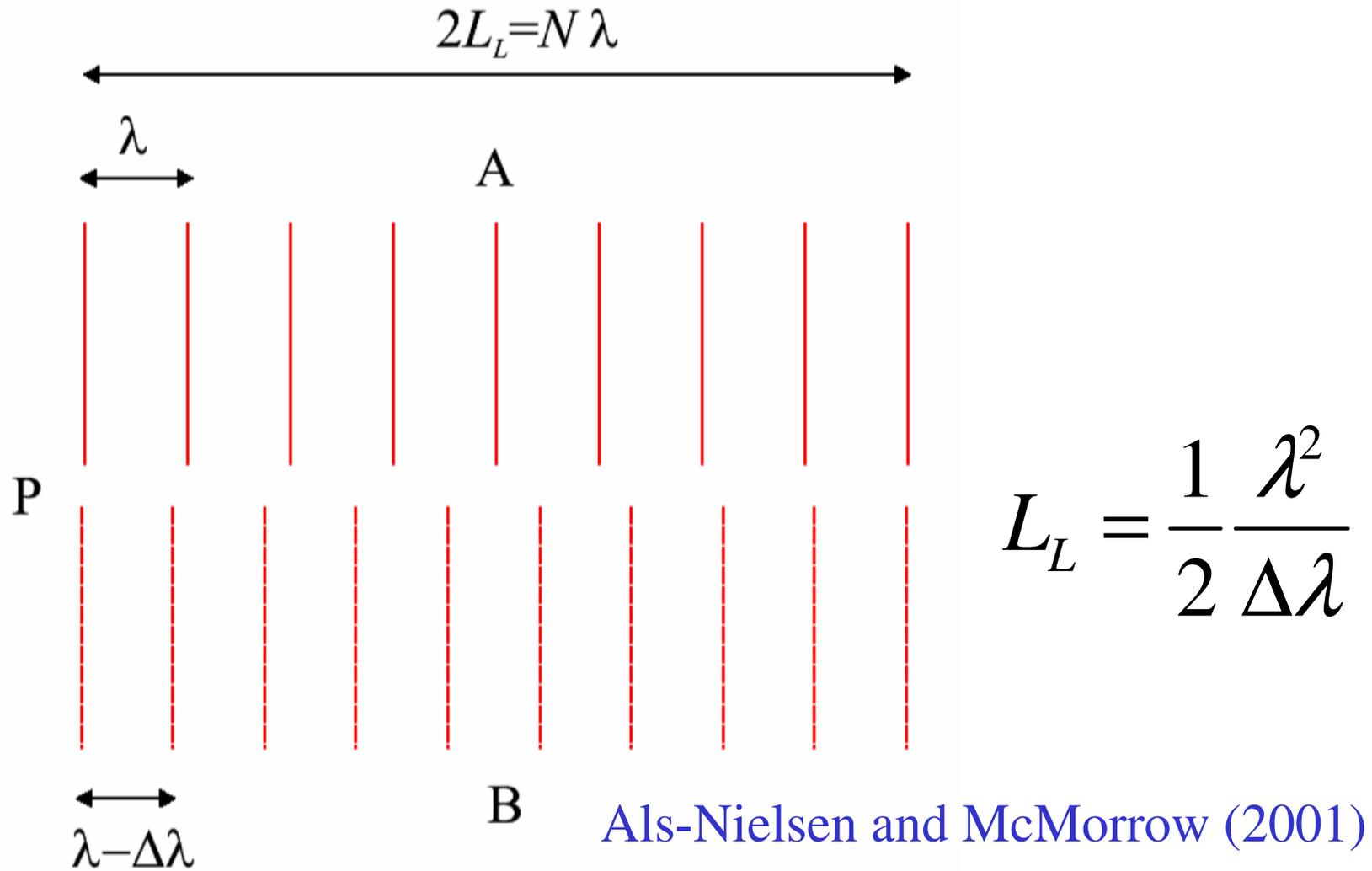
$$\lambda_X = \frac{\lambda_U}{2\gamma^2} \left\{ 1 + \frac{K^2}{2} + (\gamma\theta)^2 \right\}$$



Diamond
in-vacuum
X-ray
Undulator

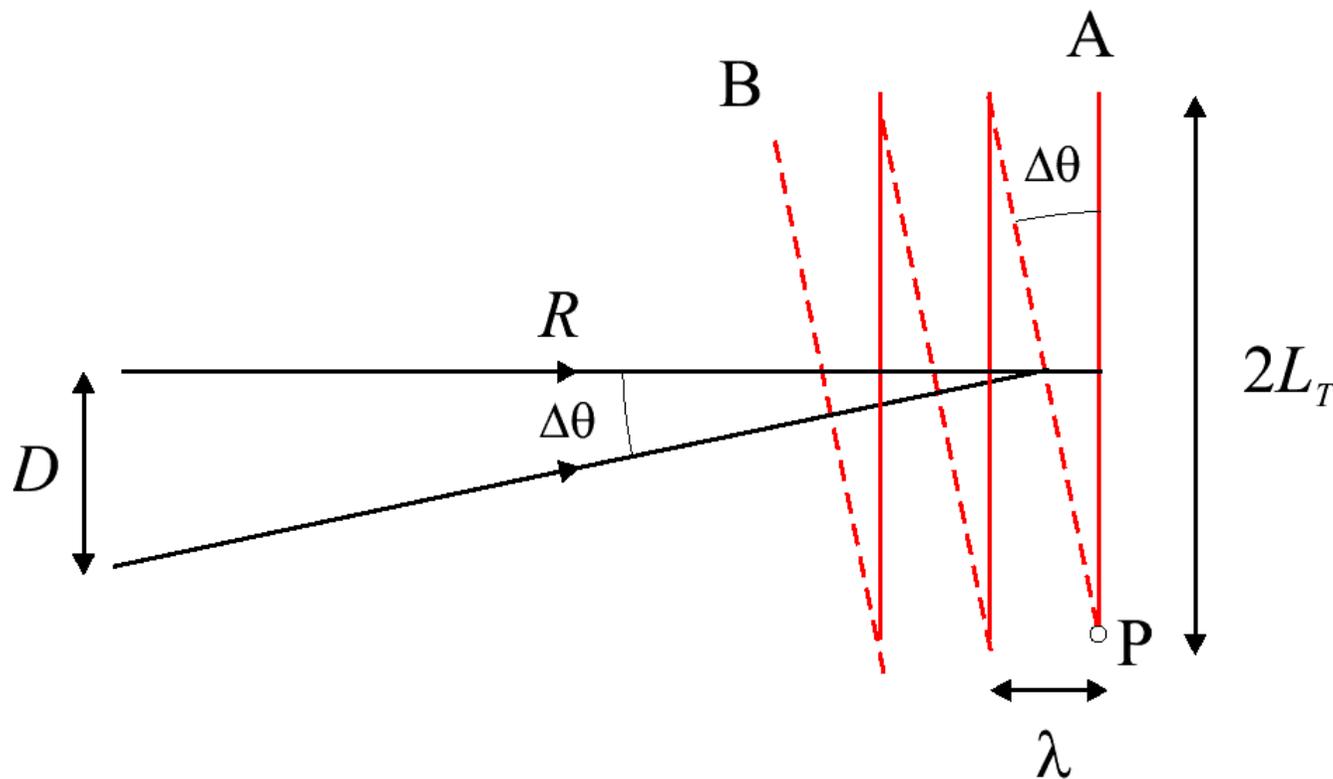


Longitudinal Coherence



Als-Nielsen and McMorro (2001)

Lateral (Transverse) Coherence

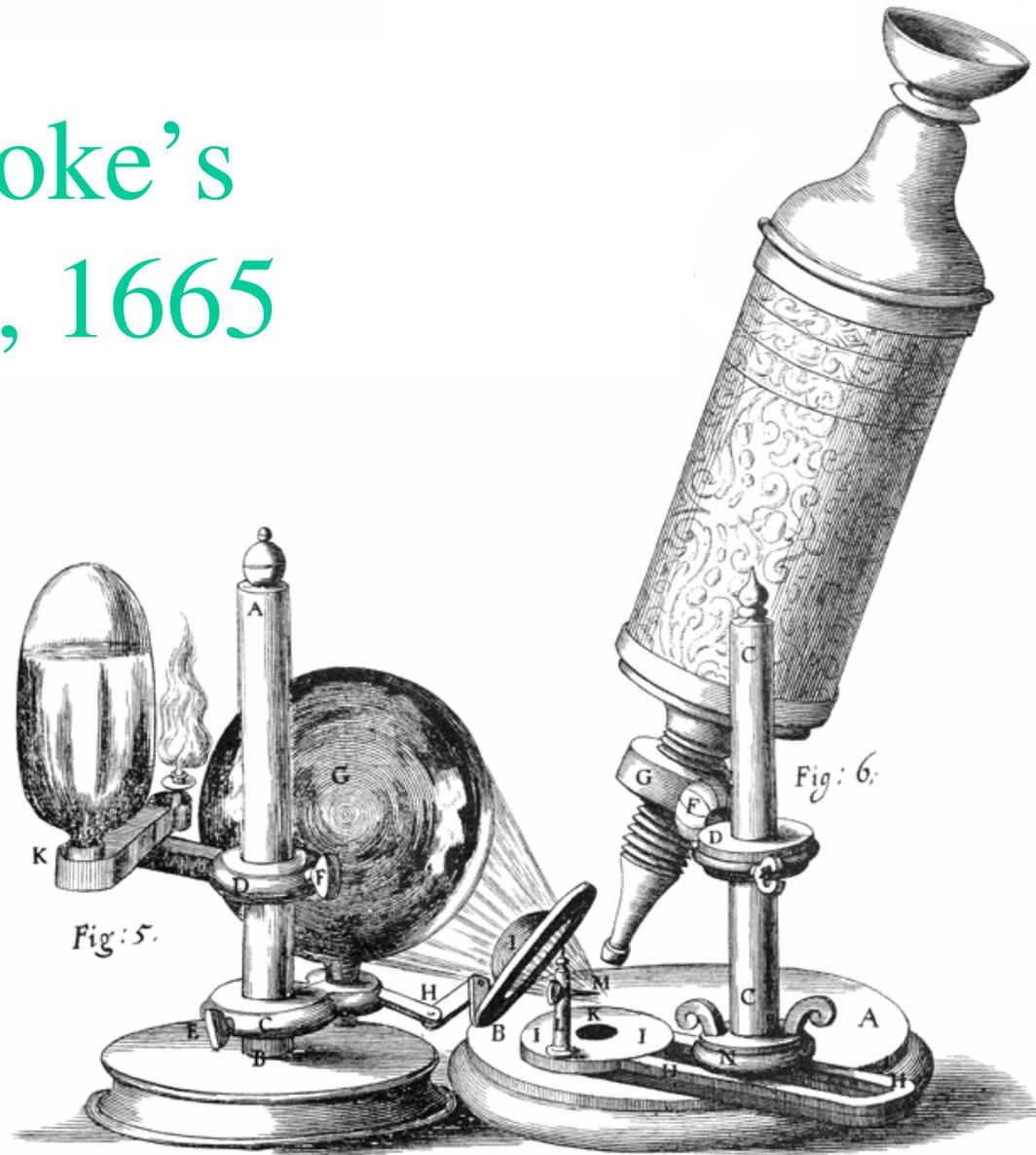


$$L_T = \frac{\lambda R}{2 D}$$

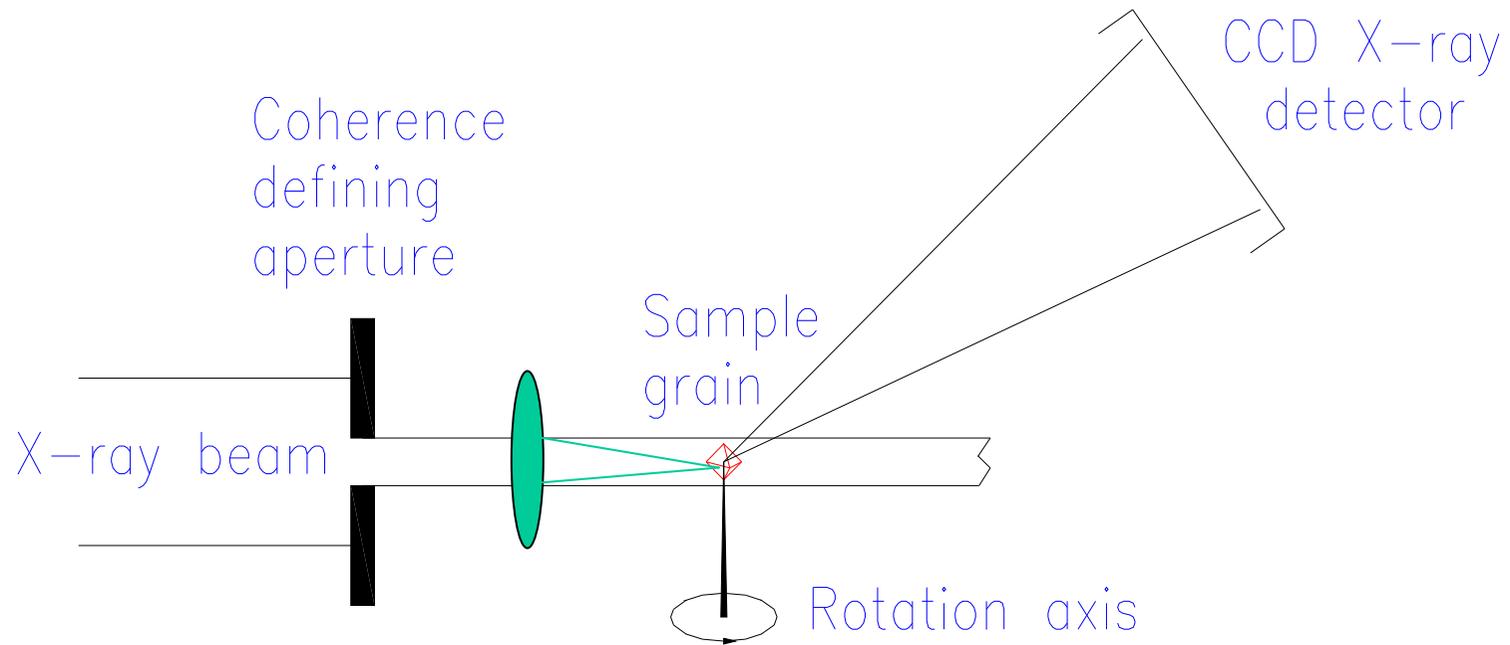
Als-Nielsen and McMorrow (2001)

Robert Hooke's microscope, 1665

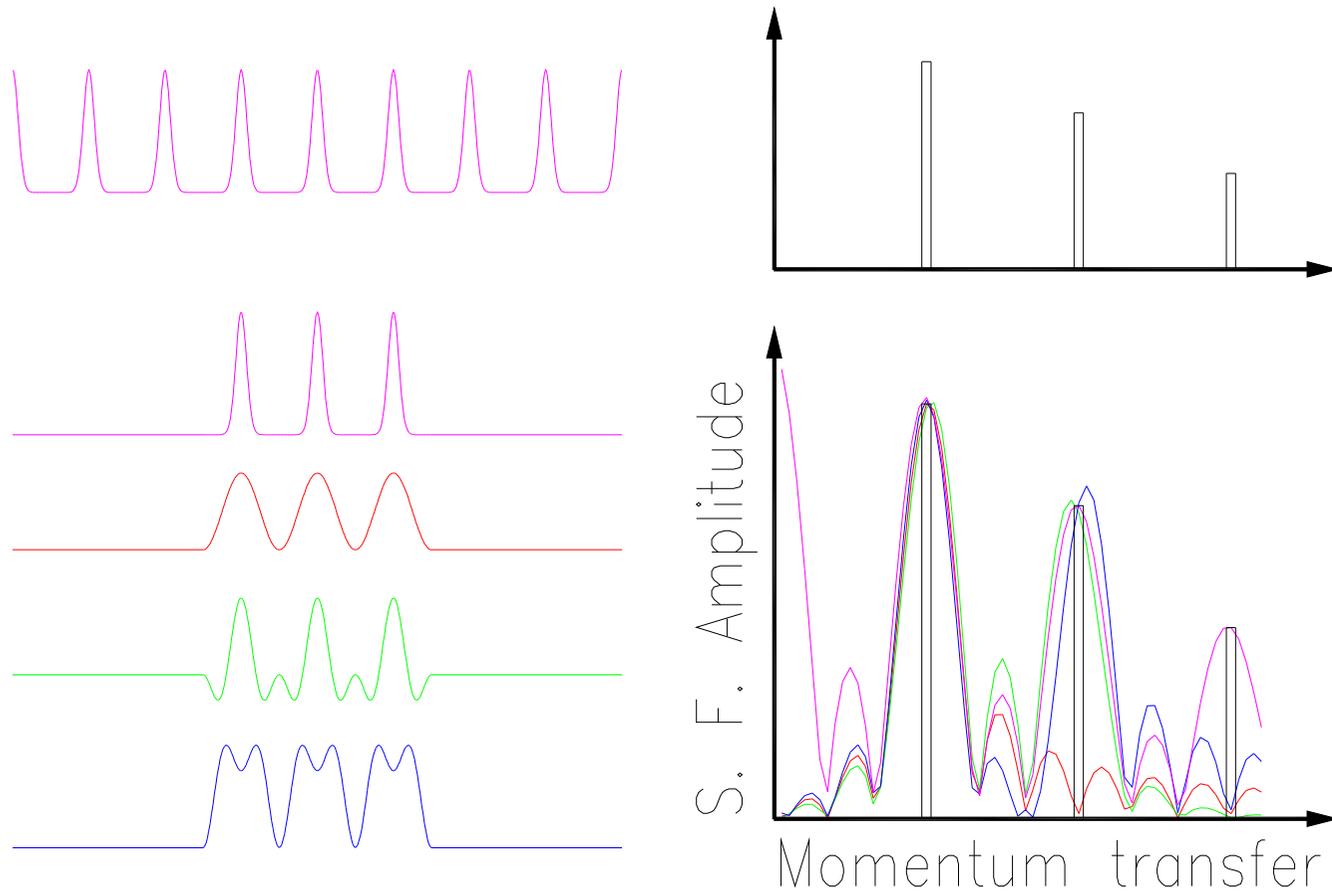
National Museum of
Health and Medicine,
Washington DC



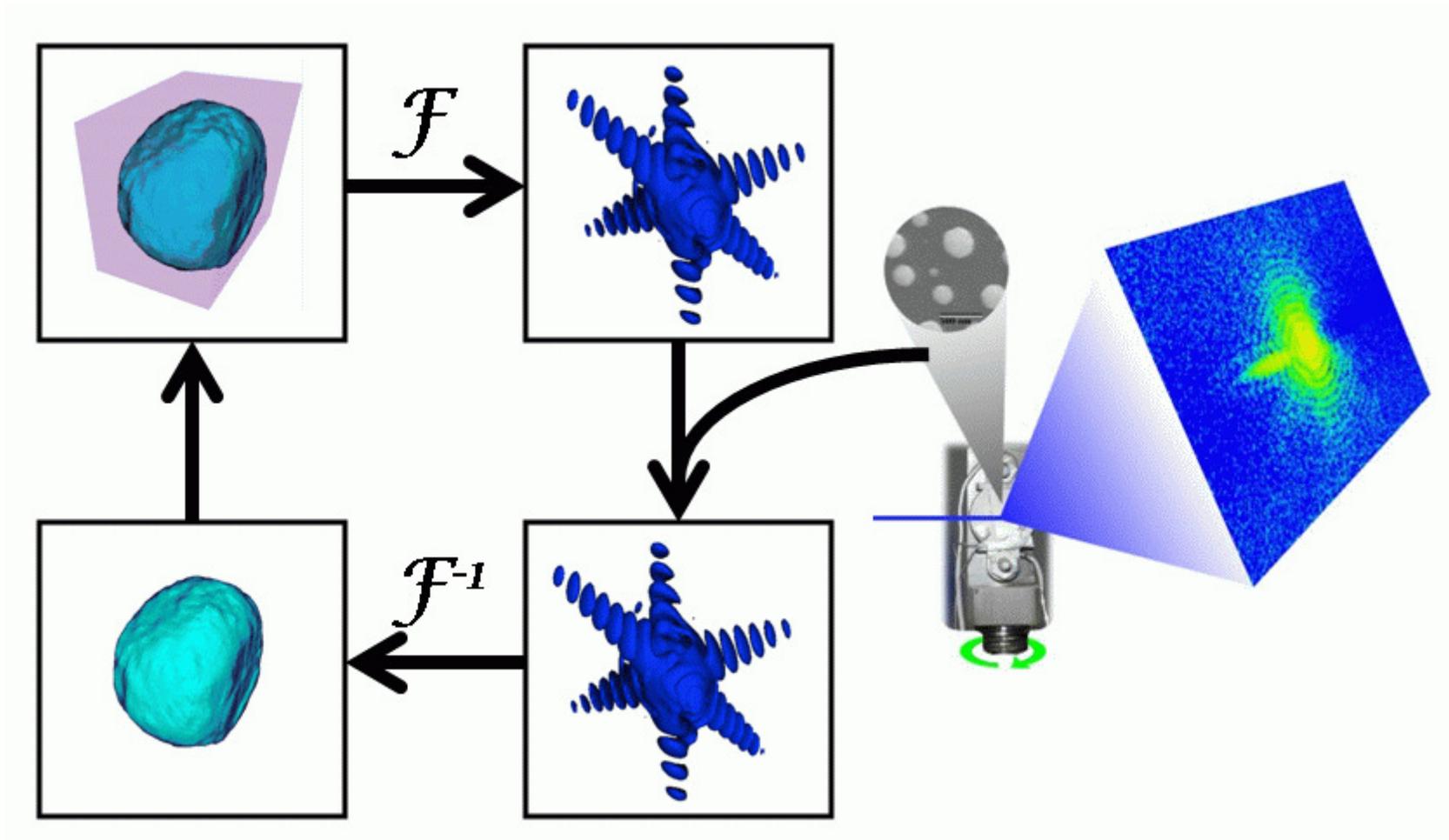
Lensless X-ray Microscope, 2003



Oversampling solves Phase Problem

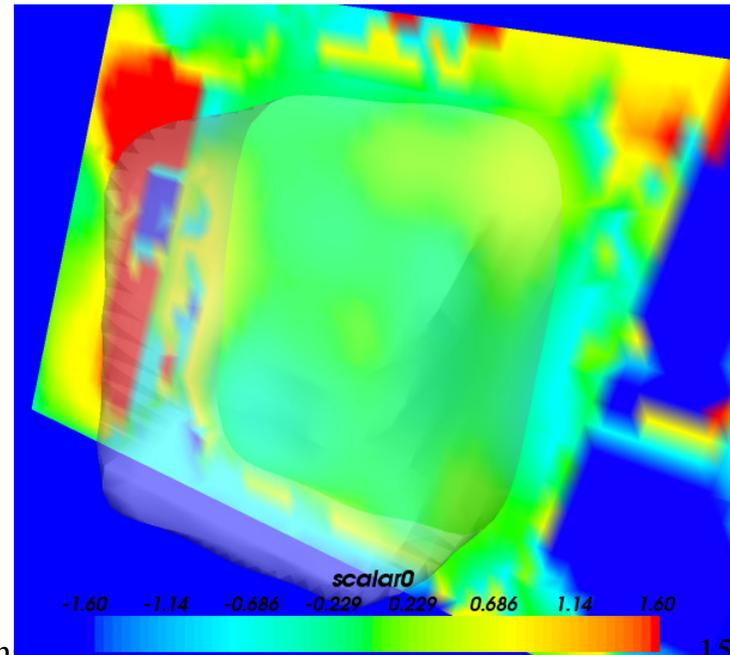
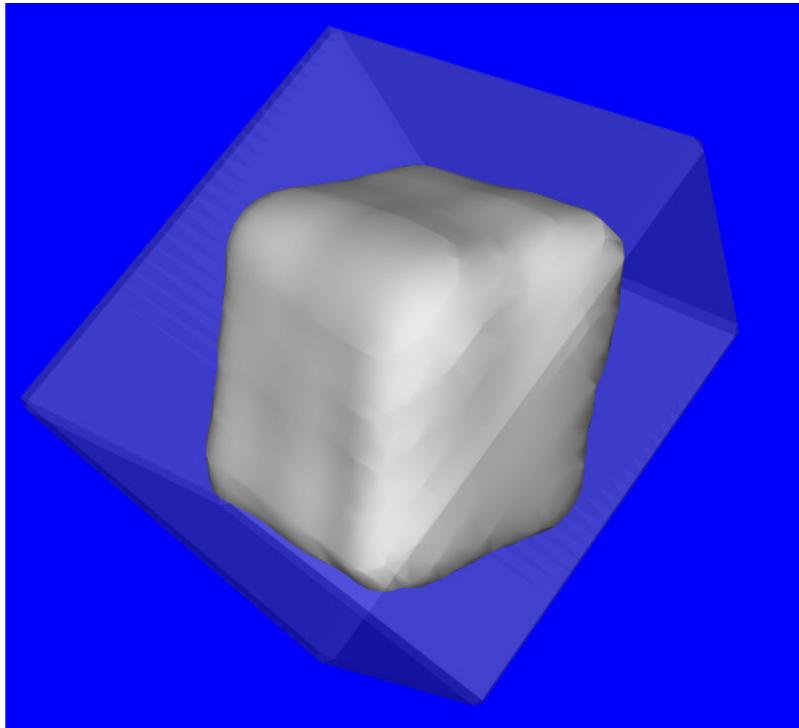
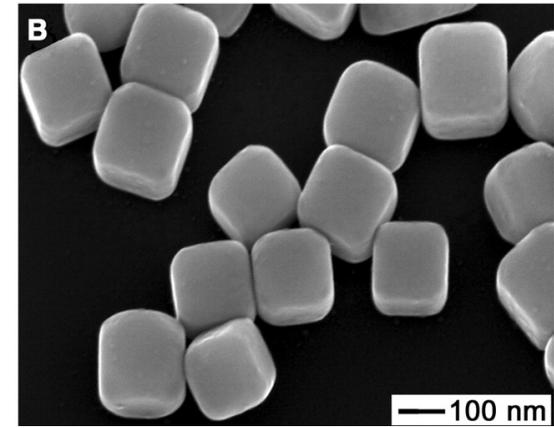
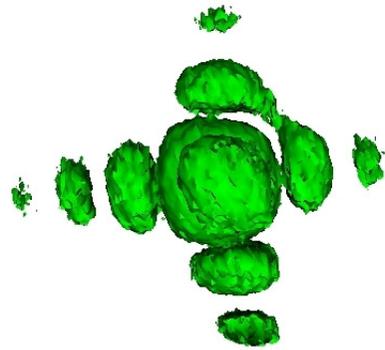
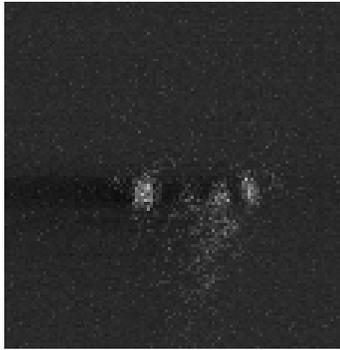


Generic “Error Reduction” method

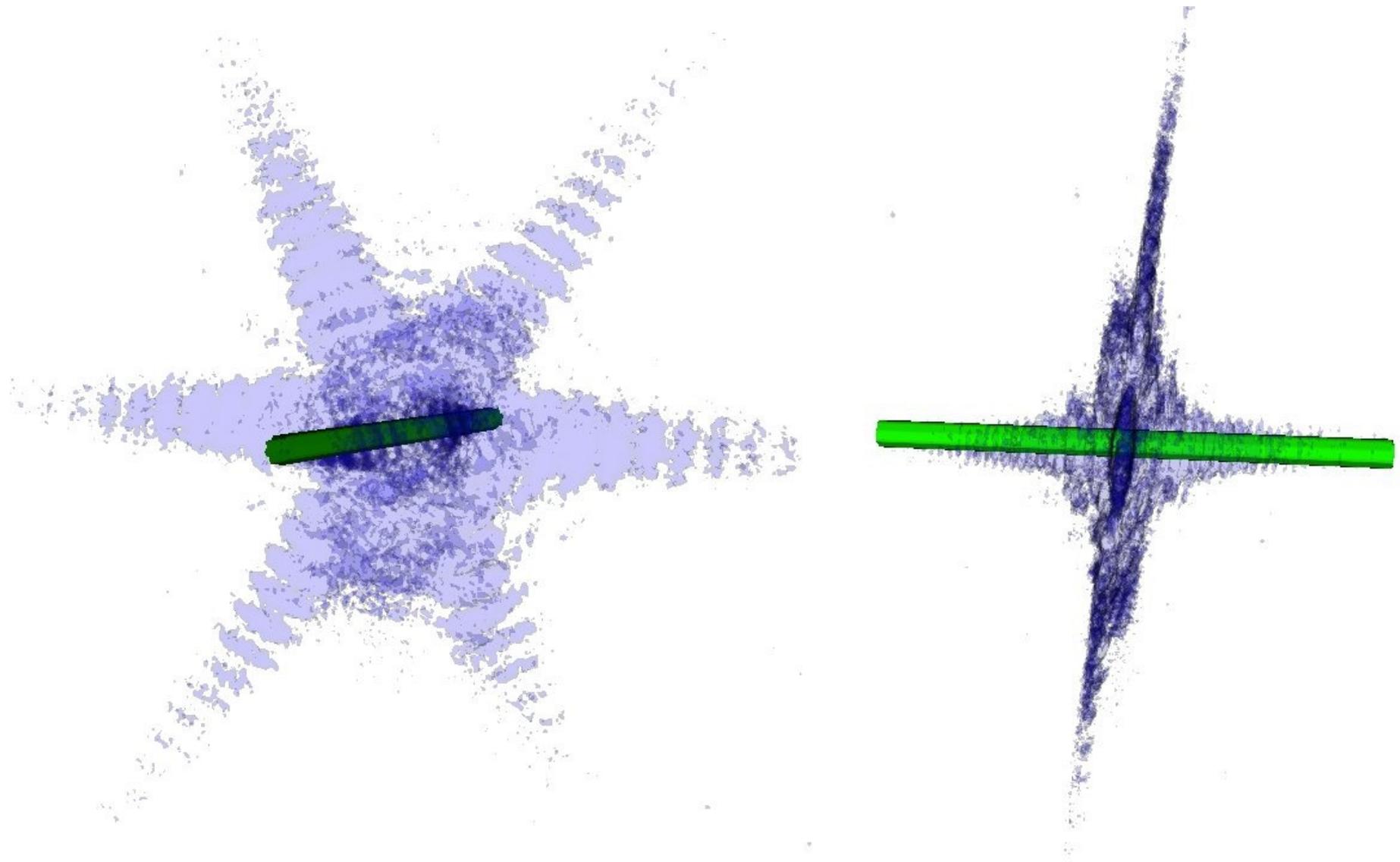


J. R. Fienup *Appl. Opt.* 21 2758 (1982)

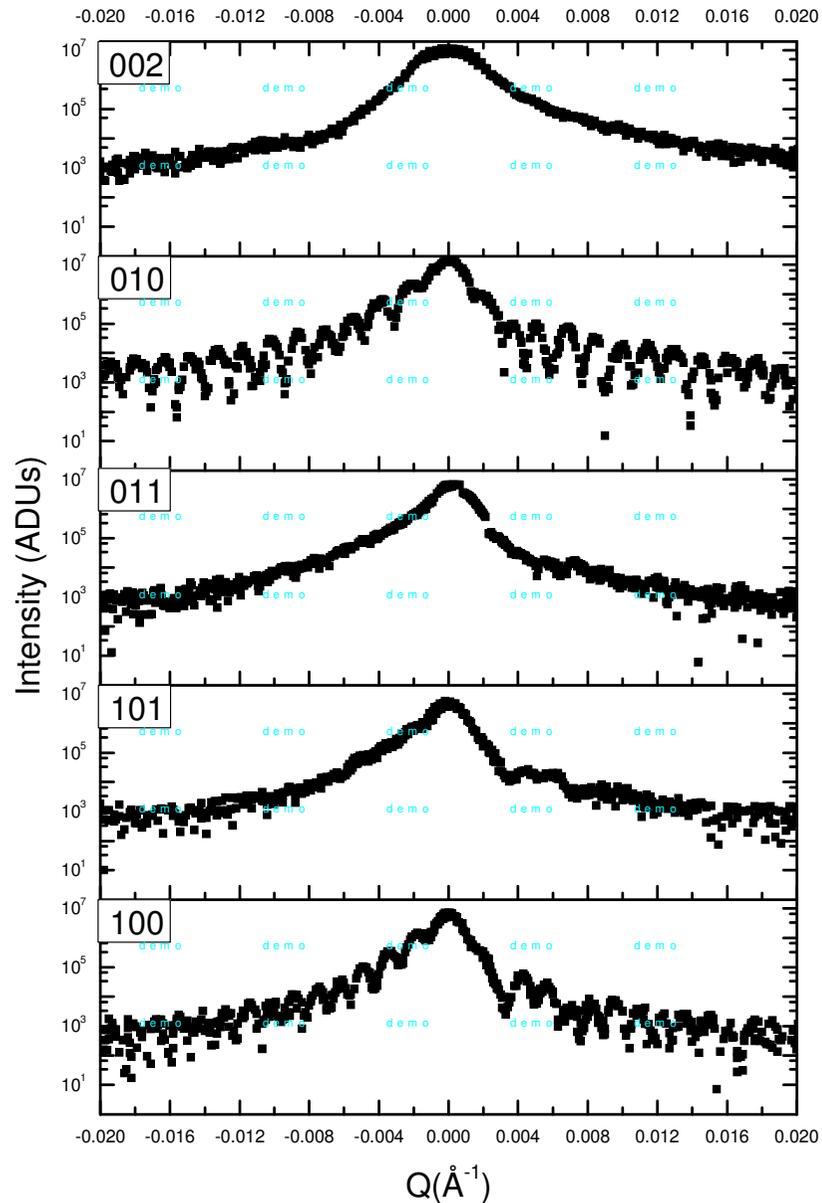
R. W. Gerchberg and W. O. Saxton *Optik* 35 237 (1972)



How to extract the contrast data



Five Bragg peaks



002 no fringe visibility

010 & 100 good fringe visibility

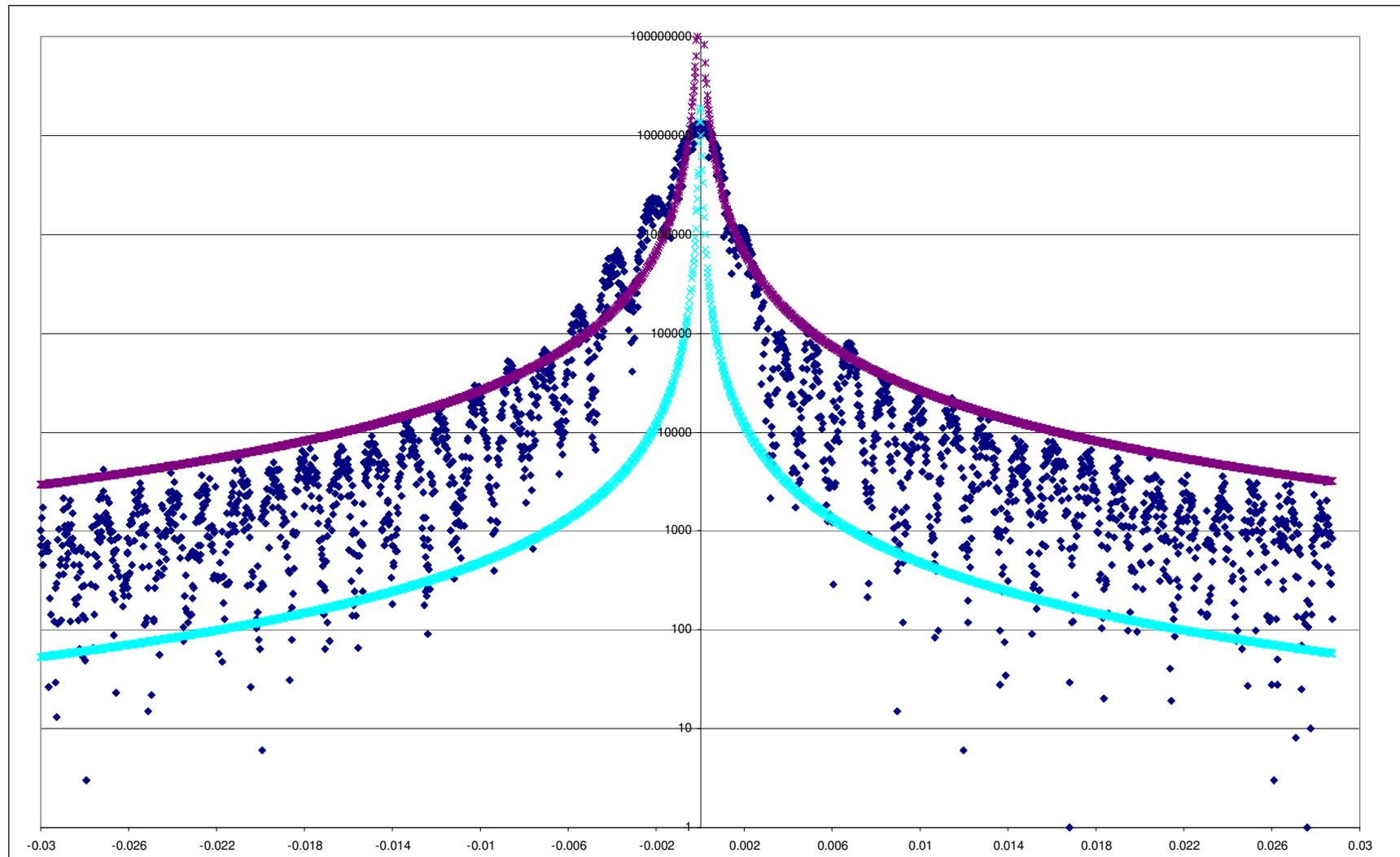
101 & 011 diminished fringe visibility
but fringes still evident

010:011 & 101:100
show complementary fringes
but not between each other

Error in coordinate transform ruled out

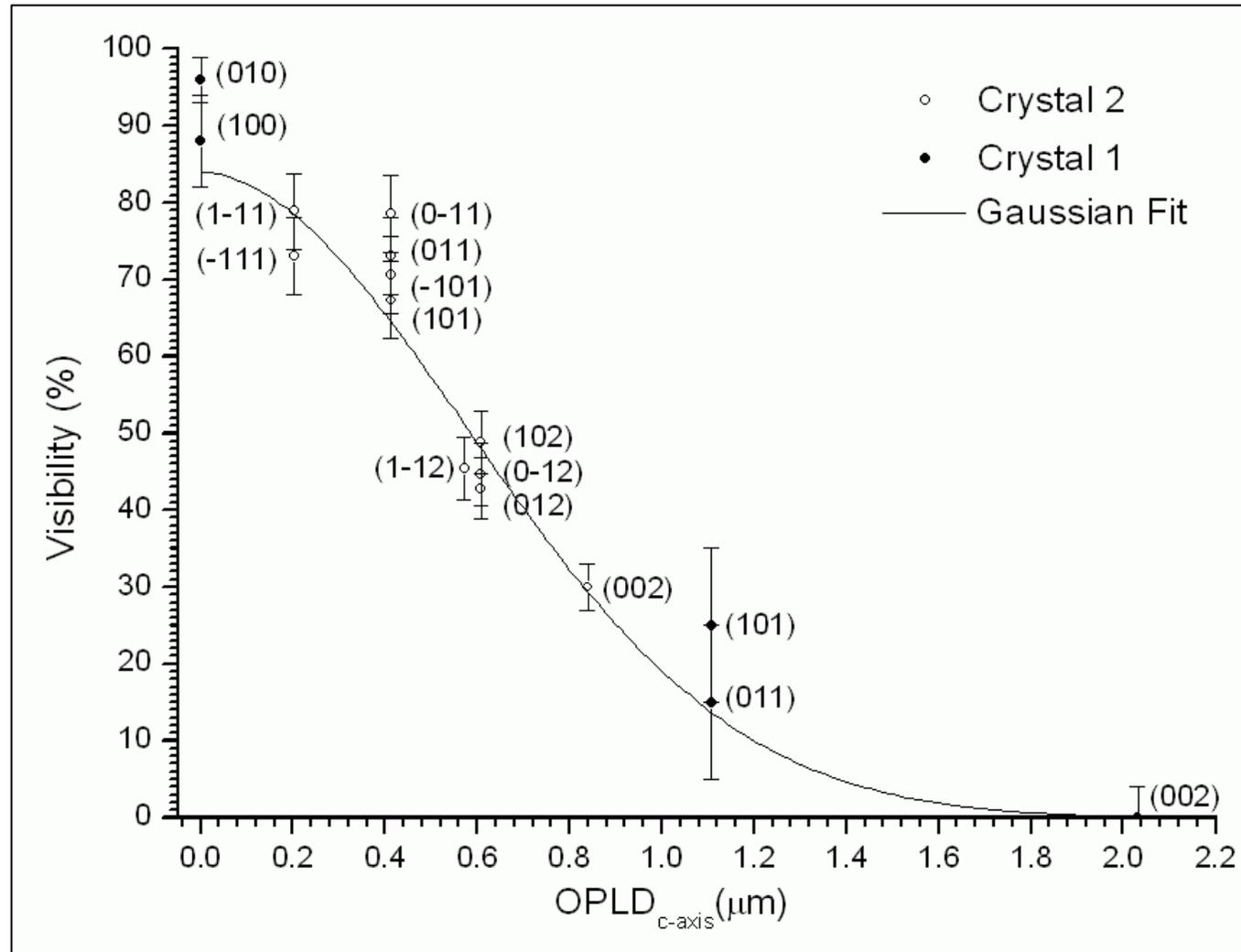
010 and 100 reflections fringe spacing
difference $\sim 20\%$

Fringe Visibility $96 \pm 2\%$ @010



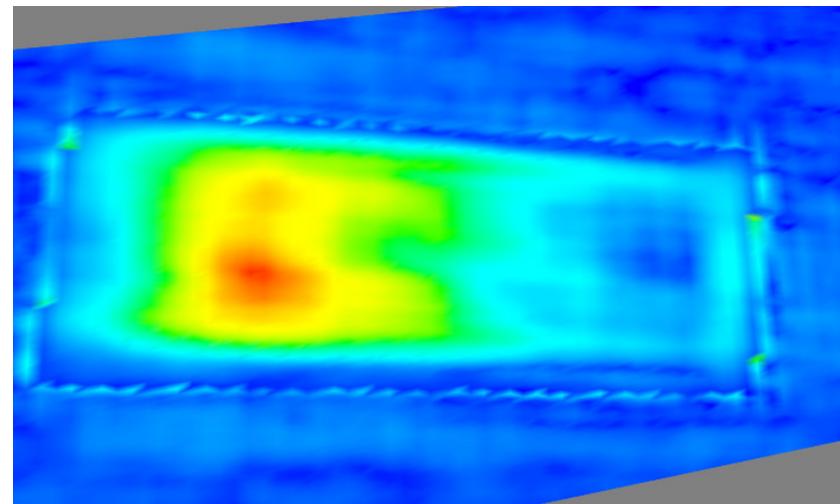
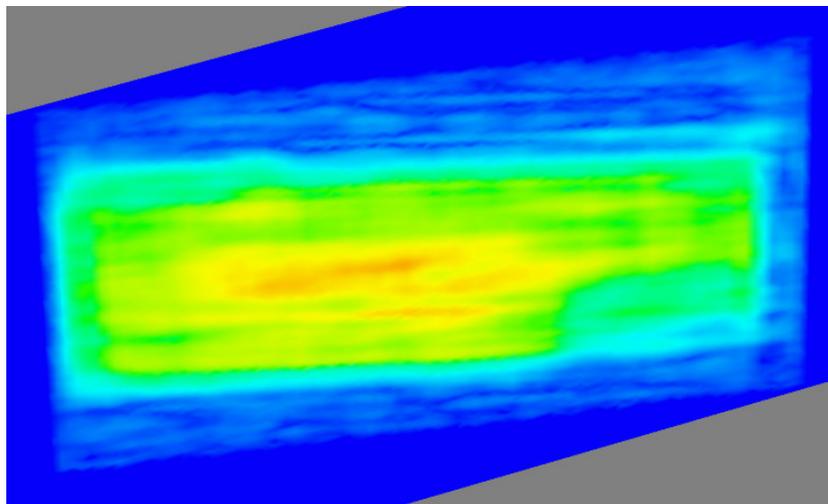
Measured Longitudinal Coherence

S. Leake, M. Newton, R. Harder and I. Robinson,
Optics Express 17 15853 (2009)

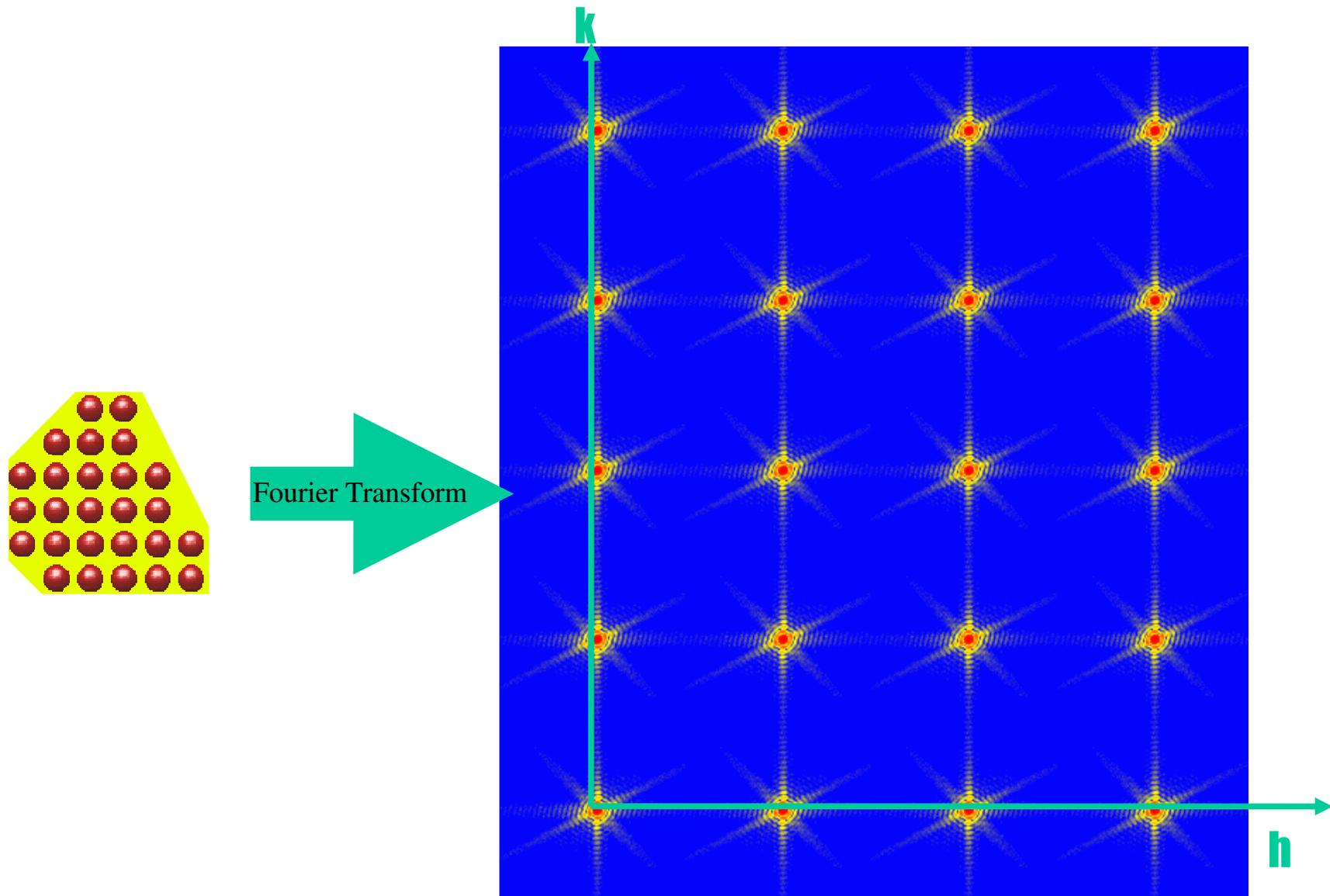


Density Section of Reconstruction using 100 and 101 reflections

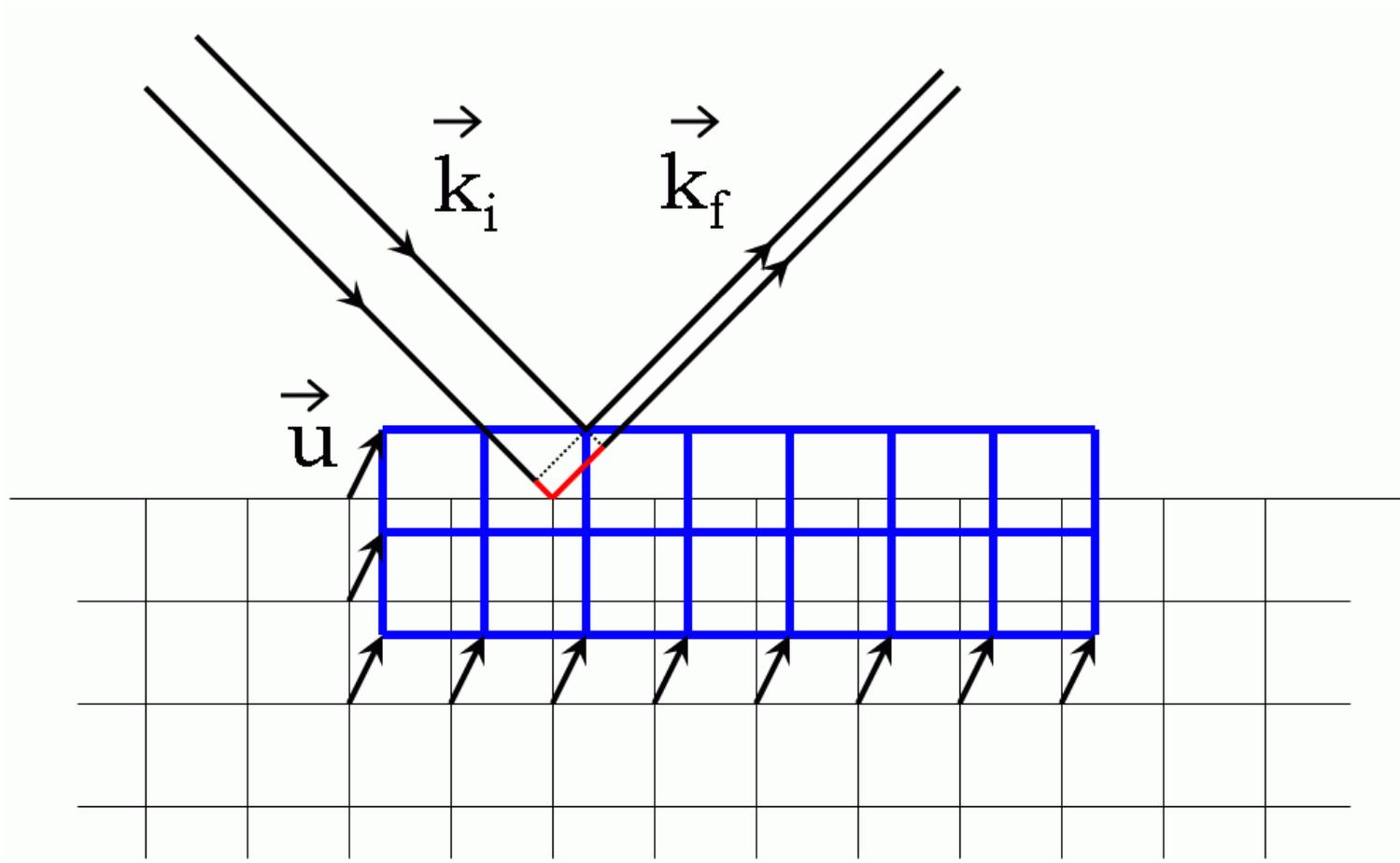
“hot spot” more developed for limited coherence
S. Leake, M. Newton, R. Harder and I. Robinson,
Optics Express 17 15853 (2009)



Coherent Diffraction from Crystals



Sensitivity to strain

$$\Delta\varphi = \mathbf{k}_f \cdot \mathbf{u} - \mathbf{k}_i \cdot \mathbf{u} = \mathbf{Q} \cdot \mathbf{u}$$


Curvature cures Stagnation

Image and its twin have same Fourier transform

$$\rho(x) \qquad \rho^*(-x)$$

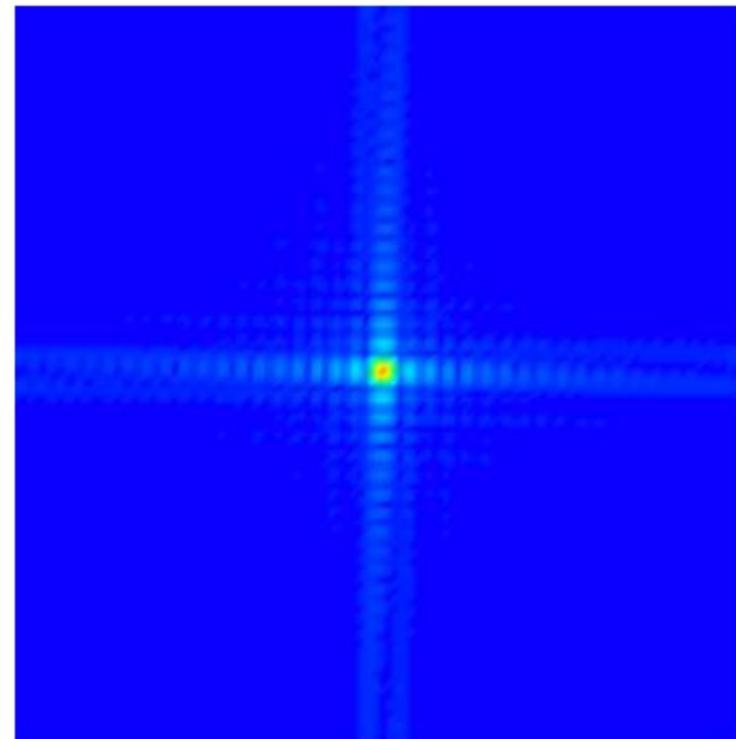
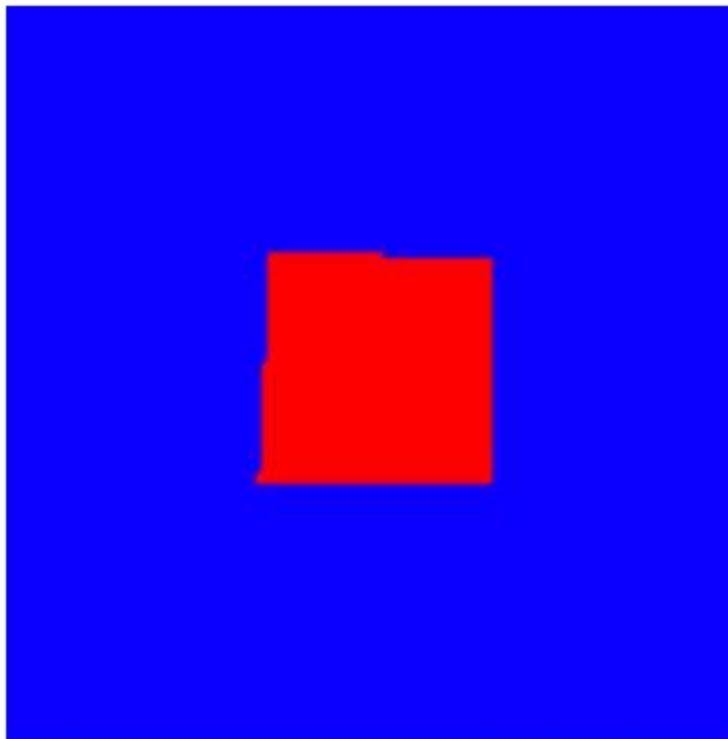
$$\rho(x) \exp\left(\frac{i\pi x^2}{\lambda D}\right) \qquad \rho^*(-x) \exp\left(\frac{-i\pi x^2}{\lambda D}\right)$$

Known modulation (phase curvature) allows twins to be discriminated using support

FFT simulations of phase objects

128x128x128 array 20x20x20 cube rotated (0.03 rad)³

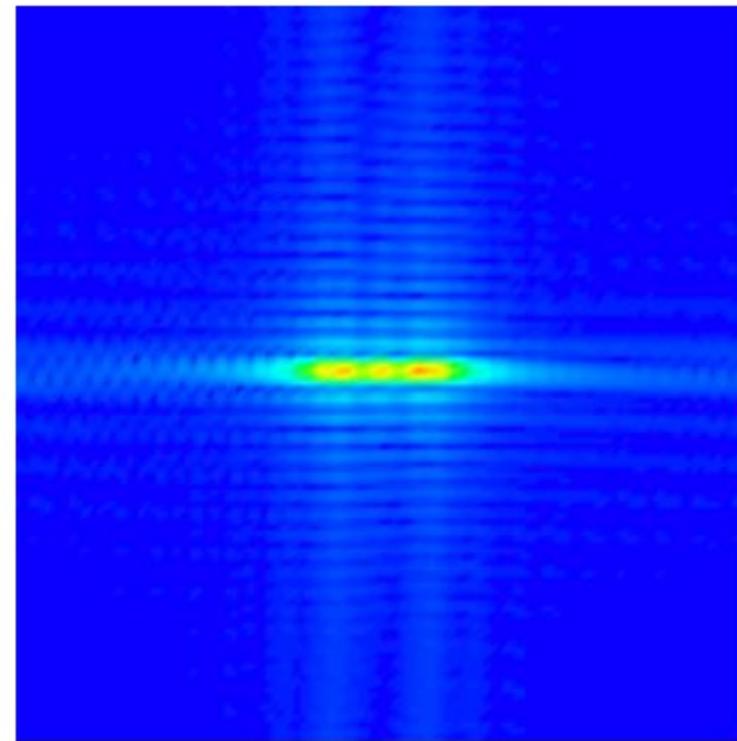
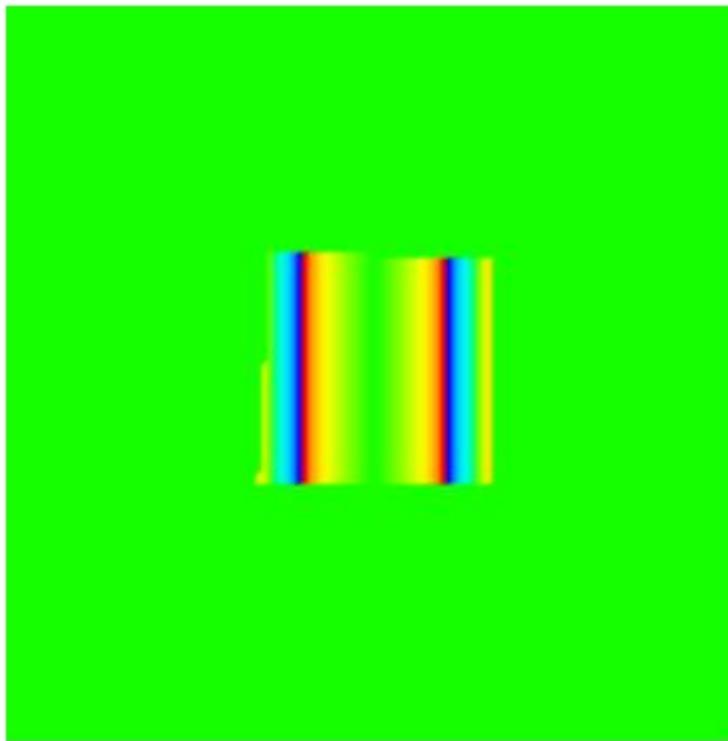
n=9 curY=0.0 curZ=0.0



FFT simulations of phase objects

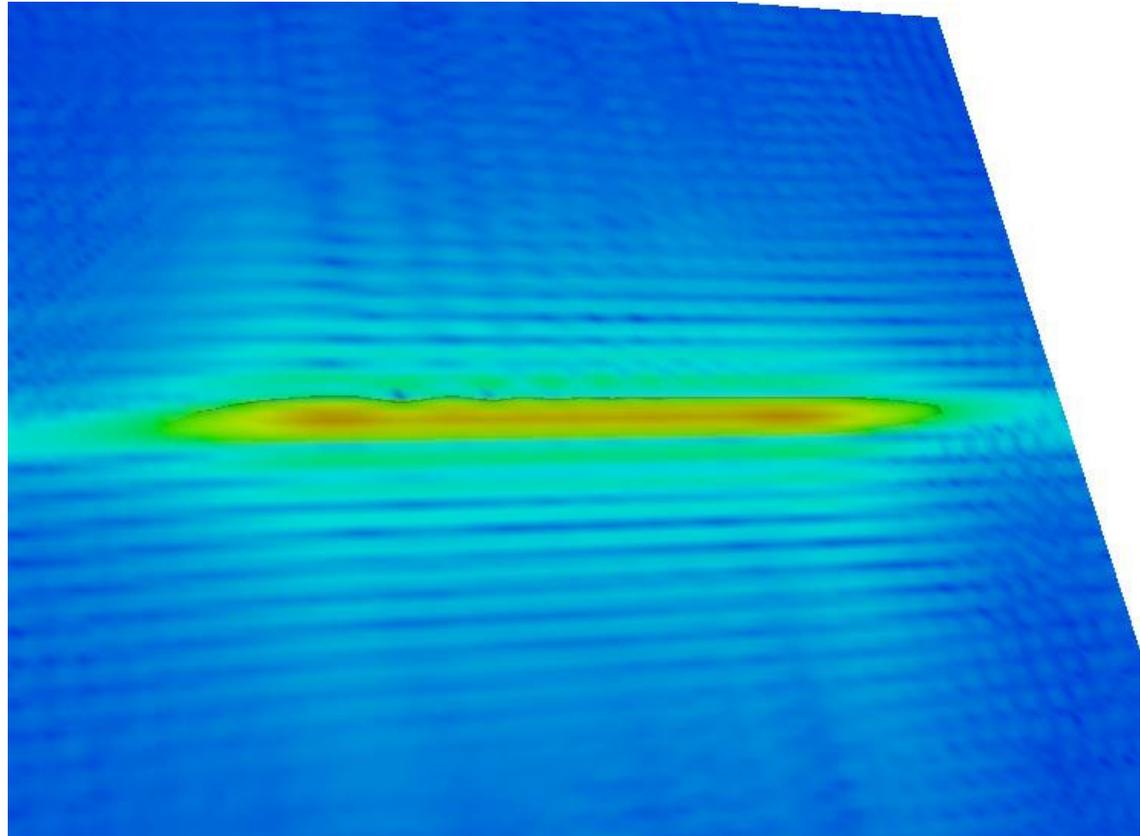
128x128x128 array 20x20x20 cube rotated $(0.03 \text{ rad})^3$

$n=9$ $\text{curY}=0.0$ $\text{curZ}=0.02$



FFT simulations of phase objects

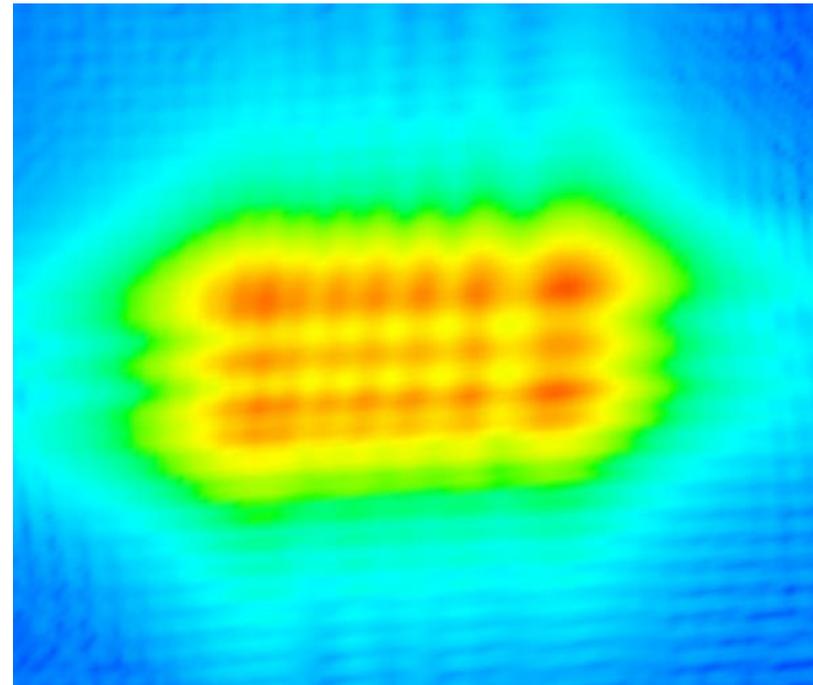
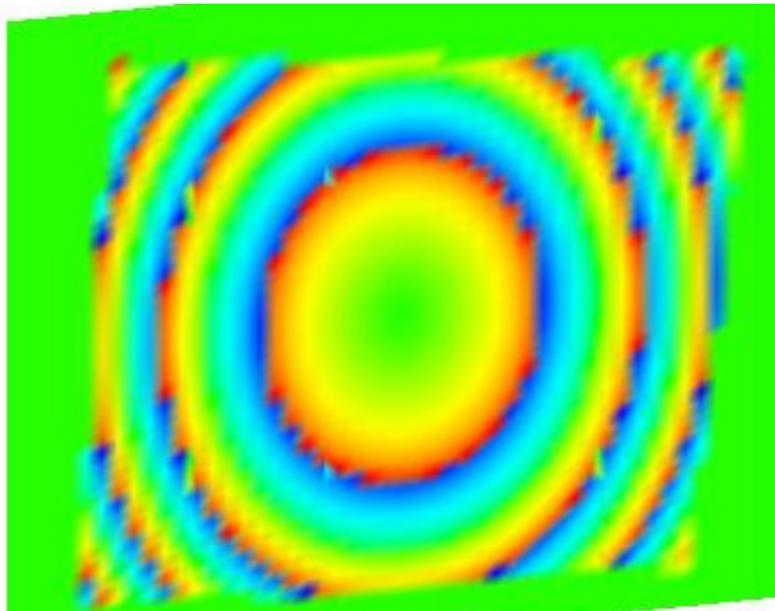
128x128x128 array 20x20x20 cube rotated $(0.05 \text{ rad})^3$



FFT simulations of phase objects

128x128x128 array 20x20x20 cube rotated $(0.05 \text{ rad})^3$

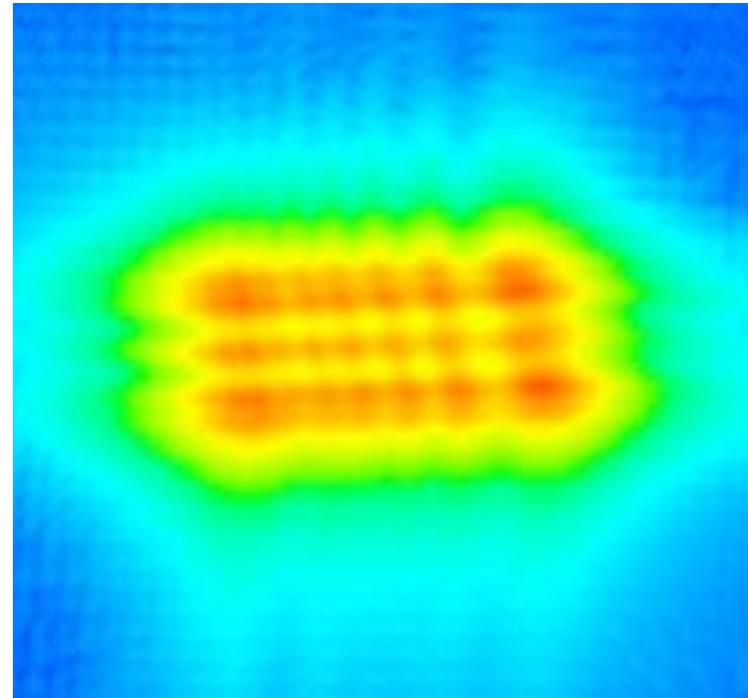
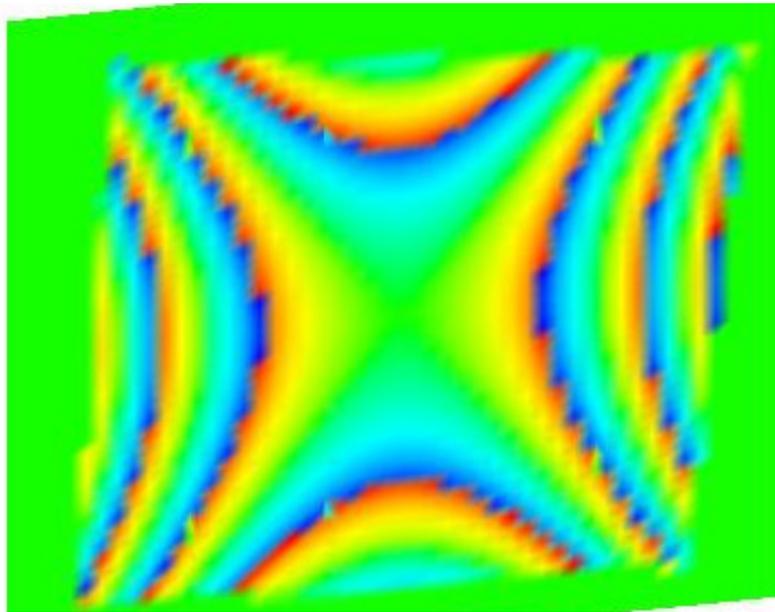
$n=5$ $\text{curX}=0.03$ $\text{curY}=0.02$



FFT simulations of phase objects

128x128x128 array 20x20x20 cube rotated $(0.05 \text{ rad})^3$

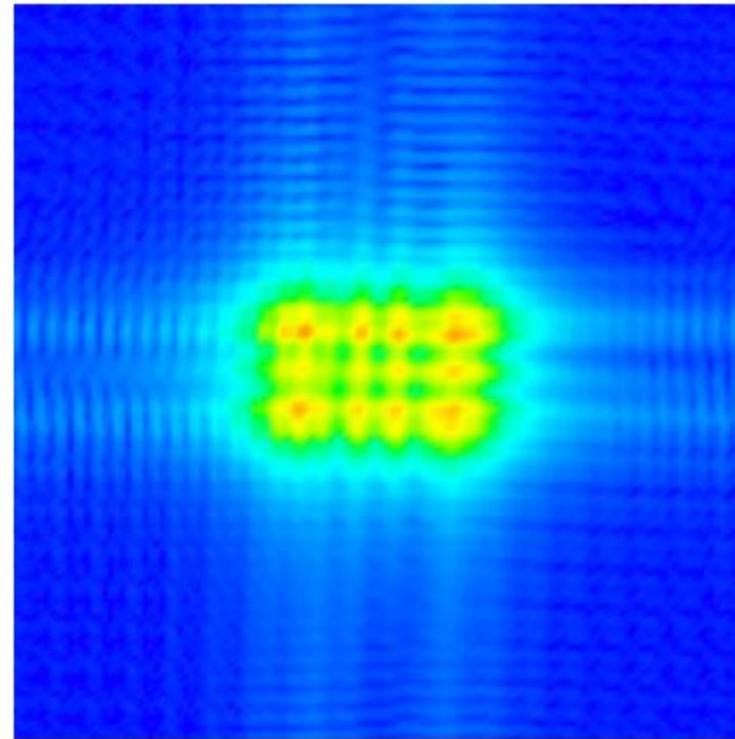
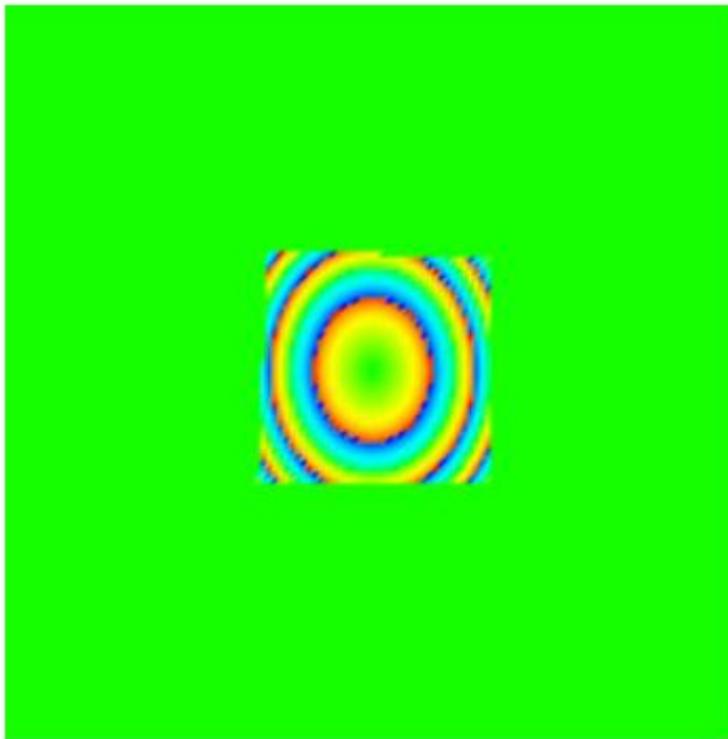
$n=6$ $\text{curX}=0.03$ $\text{curY}=-0.02$



FFT simulations of phase objects

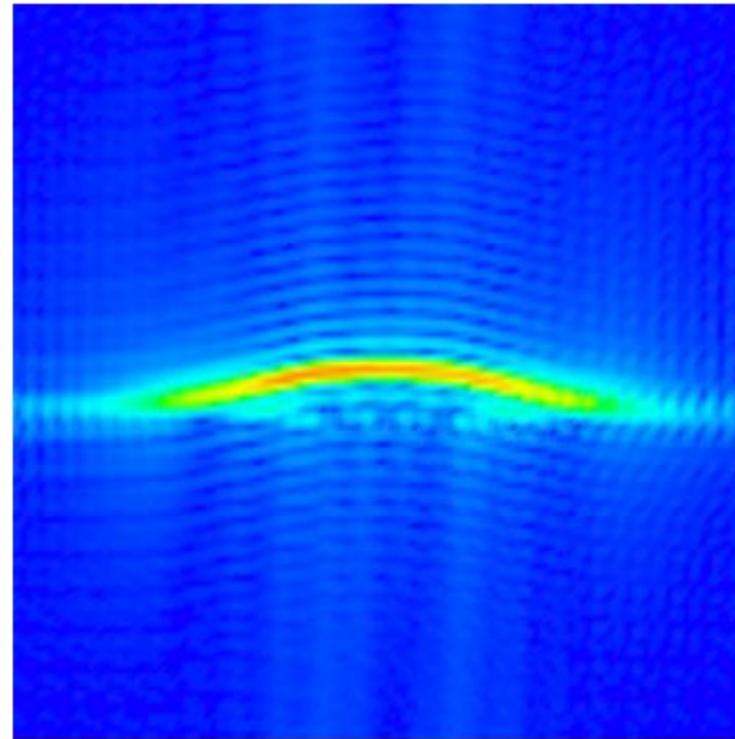
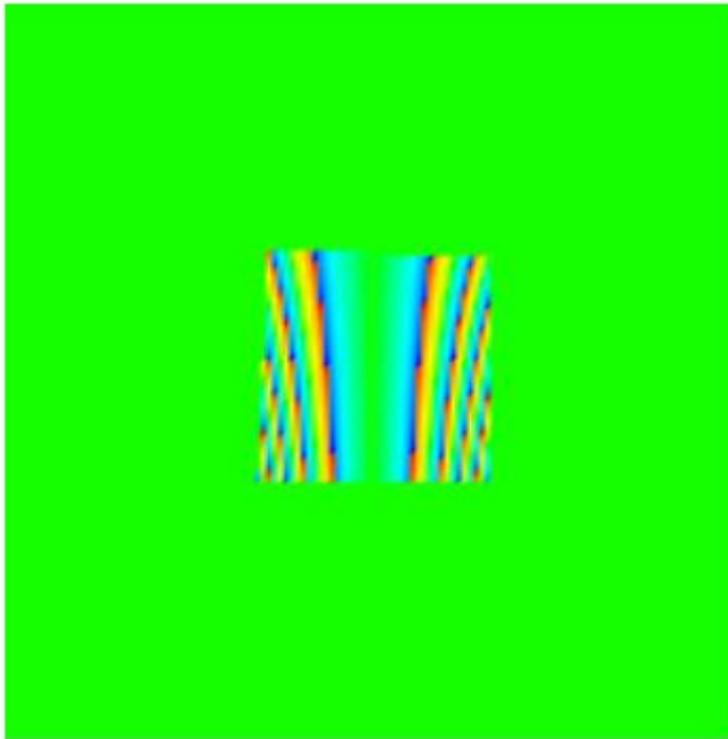
128x128x128 array 20x20x20 cube rotated $(0.03 \text{ rad})^3$

$n=8$ $\text{curY}=0.02$ $\text{curZ}=0.03$



Poly_quad_ph2 curved phases

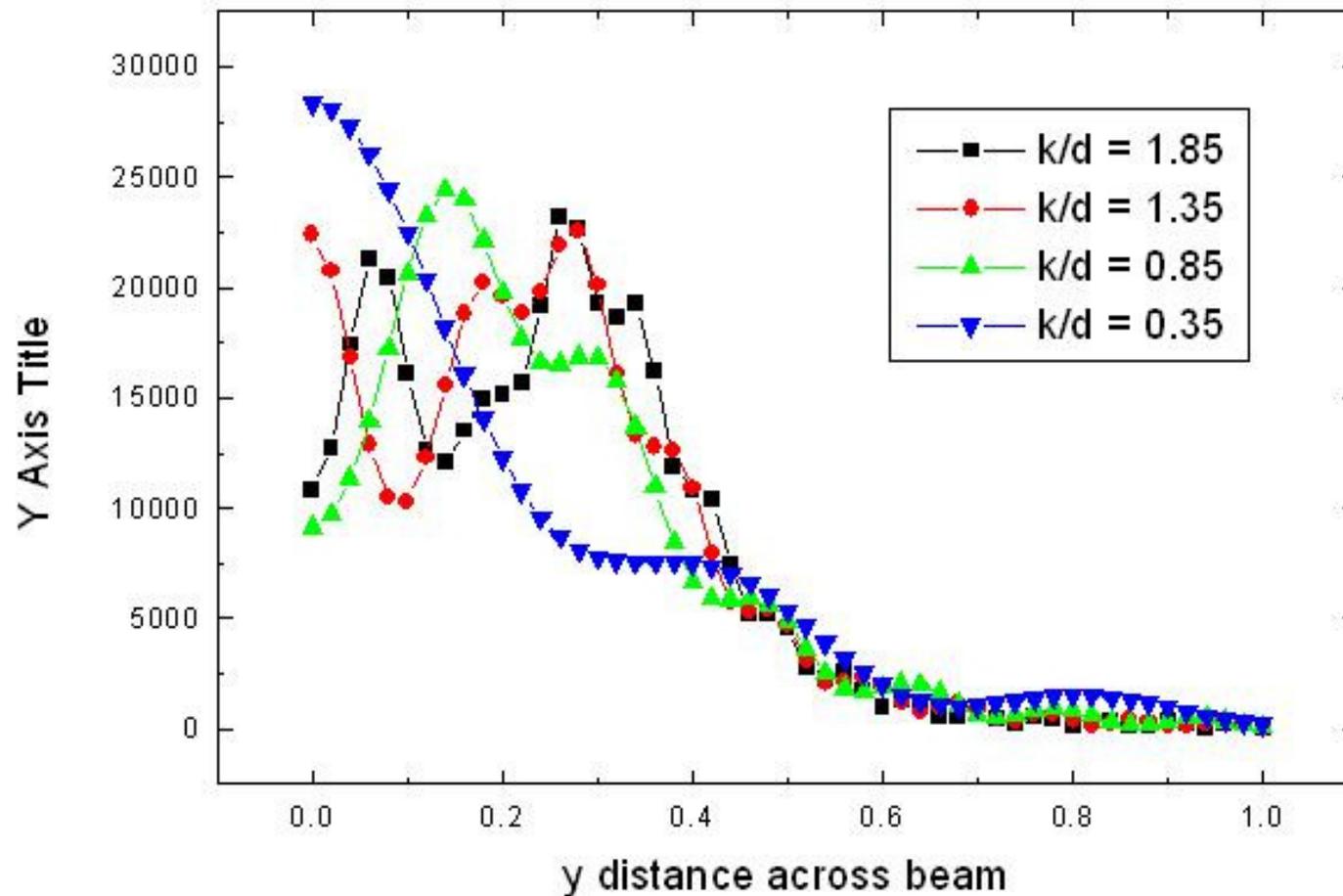
curY=50 curZ=0.001, (y-curY)*curZ*z*z



Evaluate Fresnel Integral Directly

Fres05.wk1 Fresnel diffraction

1-peak, 2-peak, 3-peak and 4-peak cases



Proposed Phasing Method

- Estimate phase curvature in XYZ
 - Fringe counting, simple modelling
- Estimate support from fringe spacing
- On each cycle of support-based ER/HIO:
 - Offset estimated phase structure
 - Apply Fourier modulus constraint
 - Remove estimated phase structure again

Andrey Minkevich (KFA Karlsruhe)

preprint <http://arxiv.org/abs/0909.4711>

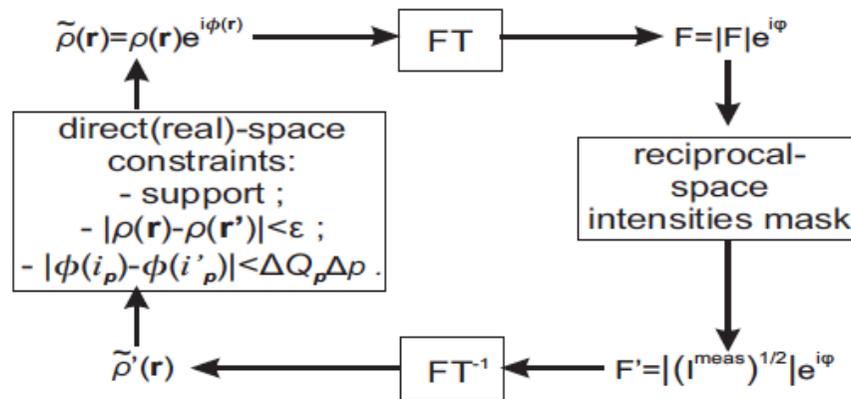
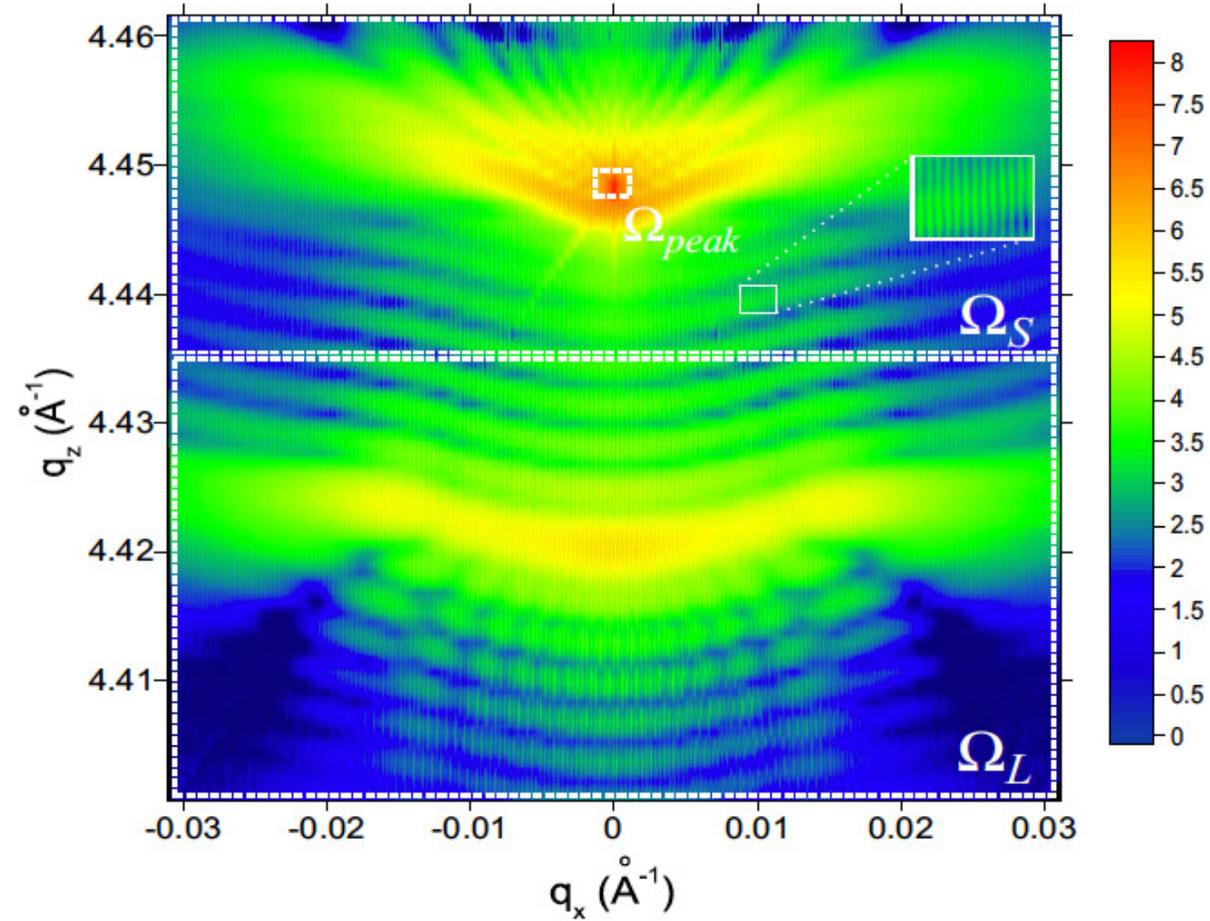


FIG. 1: Schematic diagram of the iterative loop of our phase retrieval algorithm. \mathbf{r} and \mathbf{r}' in "direct(real)-space constraints" box correspond to the neighbouring points within one constituent crystal and ϵ defines the threshold of electron density uniformity. i_p and i'_p are the neighbouring points along p -direction (normally lateral or vertical) and Δp corresponds to the step along this direction (achieved resolution).

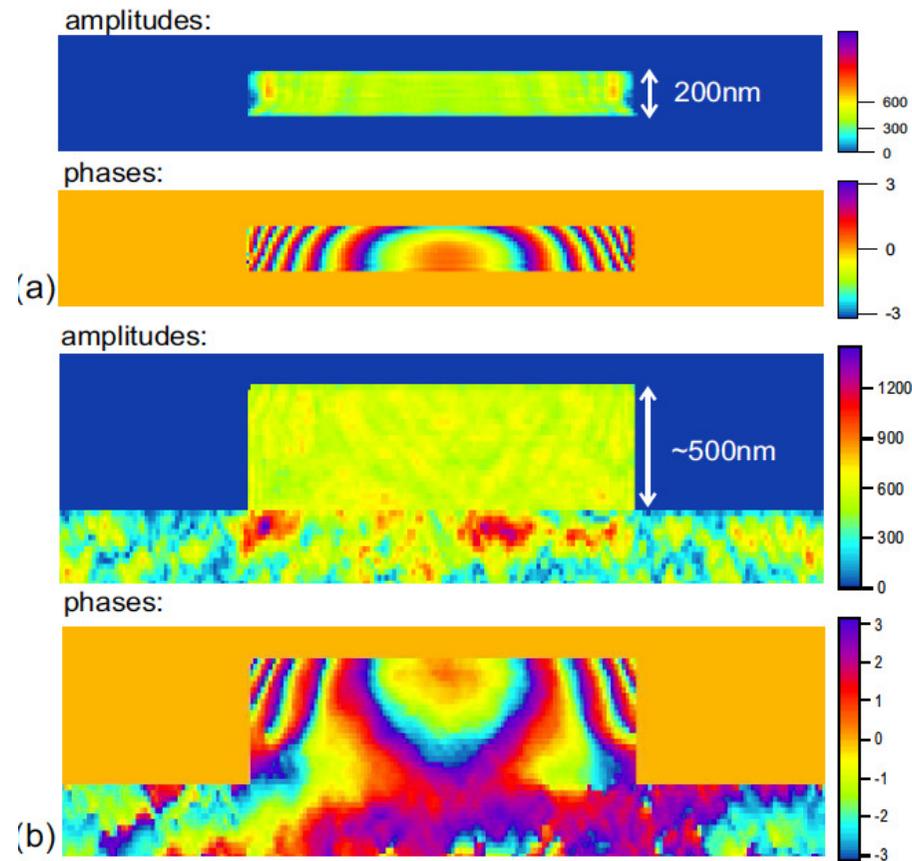
Andrey Minkevich (KFA Karlsruhe)

GaMnAs wires on GaAs



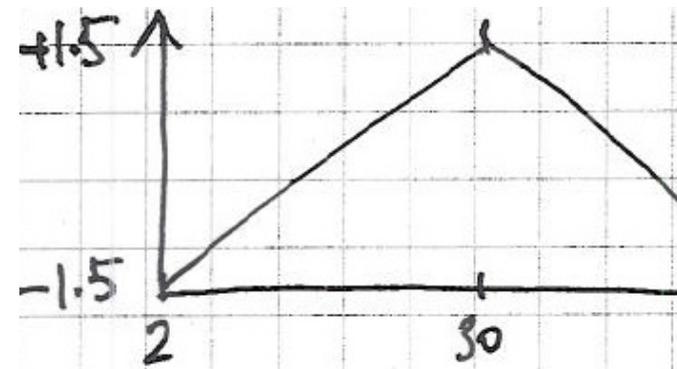
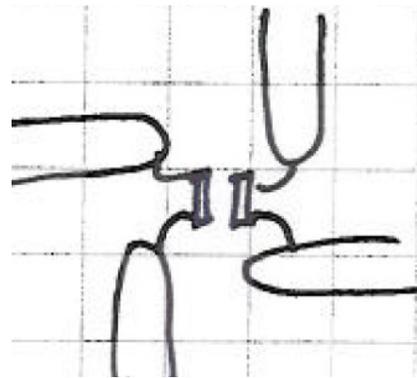
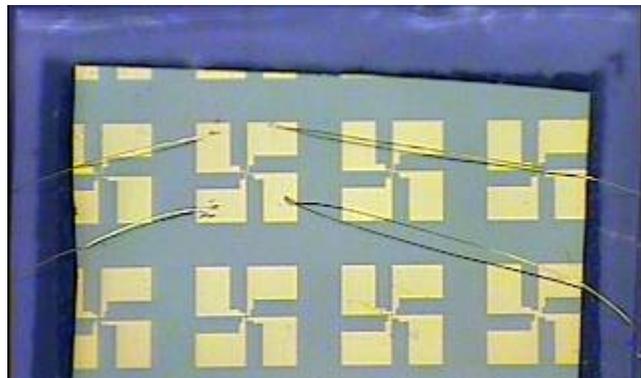
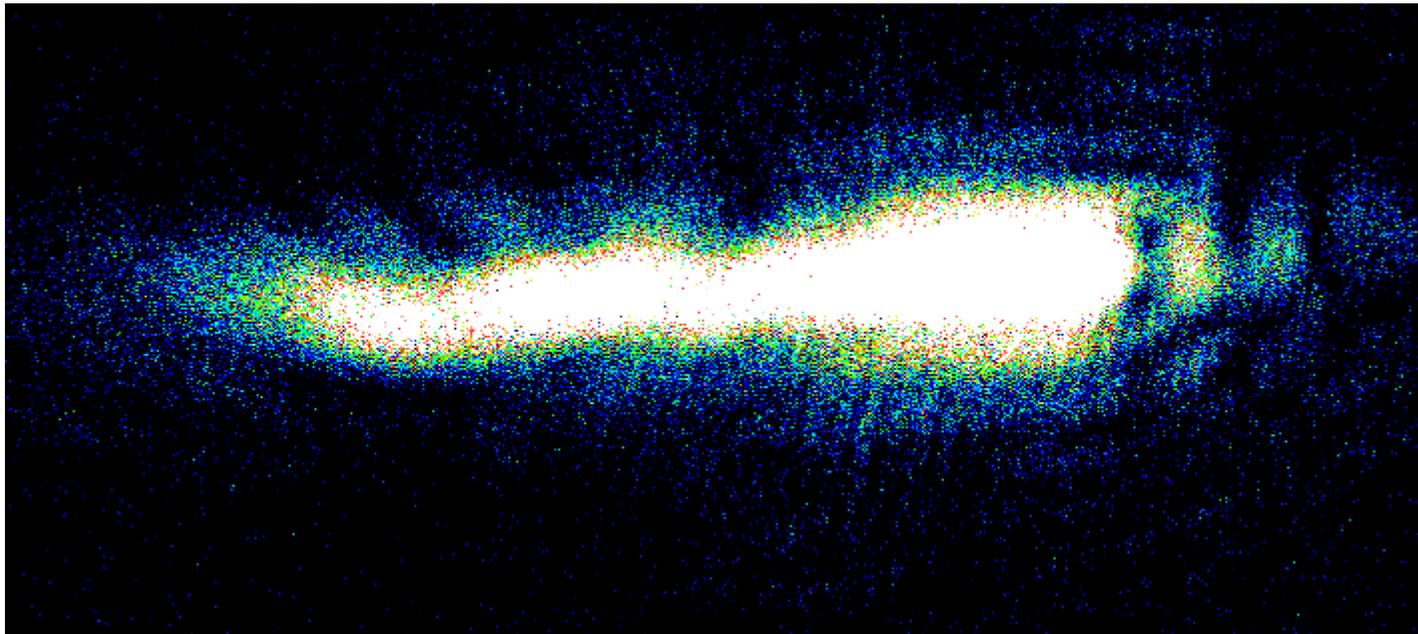
Andrey Minkevich (KFA Karlsruhe)

preprint <http://arxiv.org/abs/0909.4711>

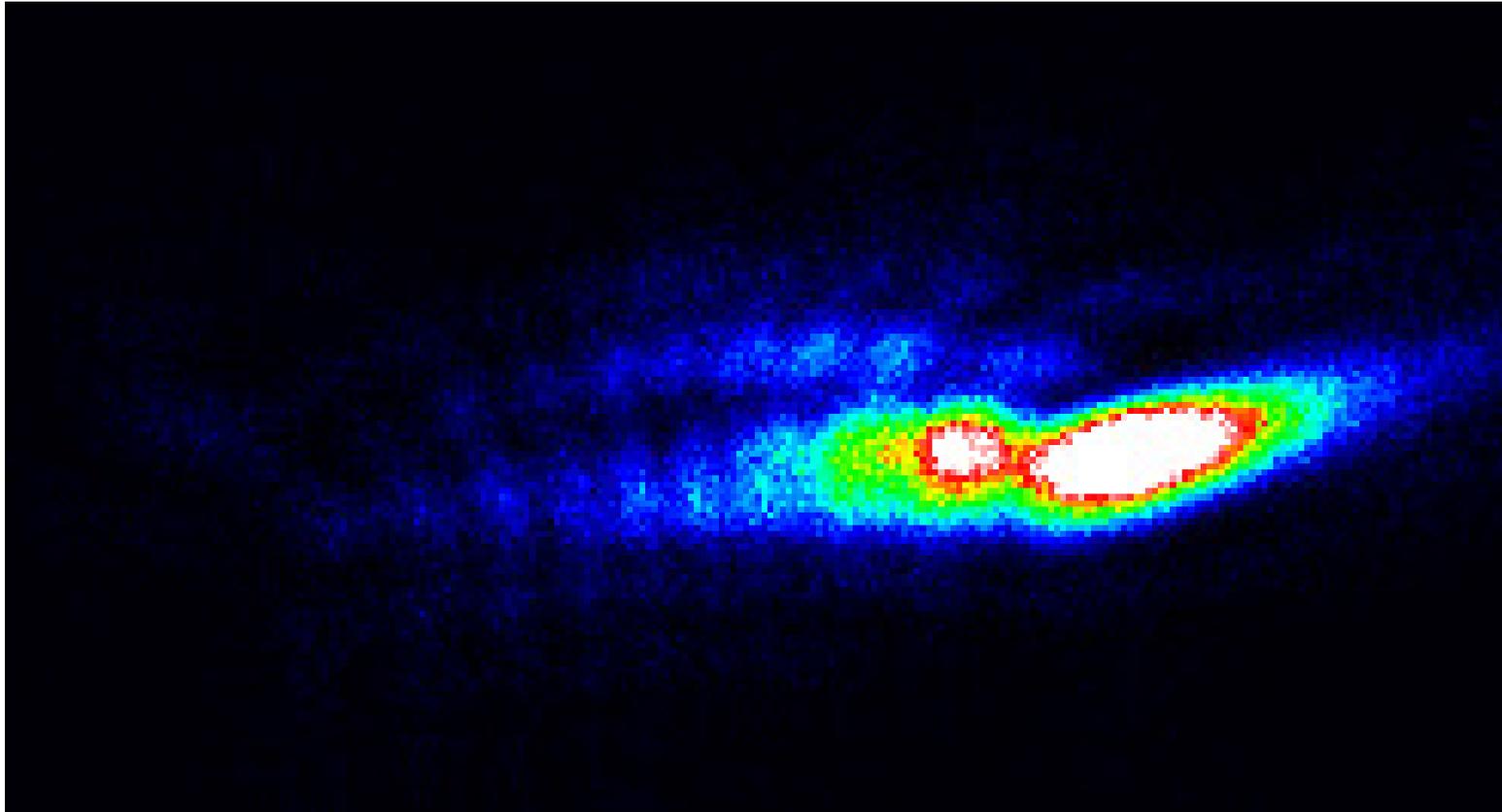


Apply Electric Field along ZnO NC

Marcus Newton. 34-ID-C zno808-18 Aug 2008

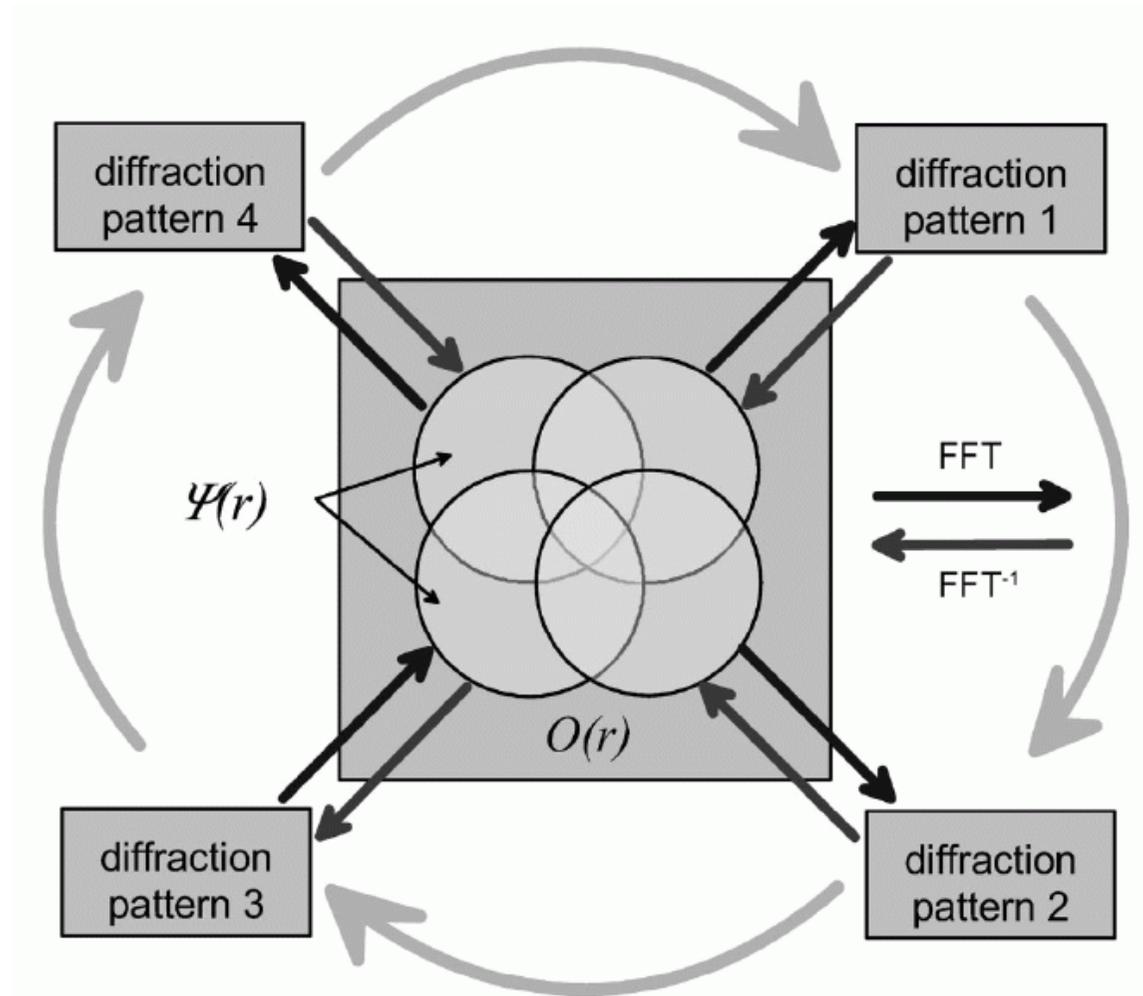


ZnO409-44 wired ZnO crystal



X-ray Ptychography

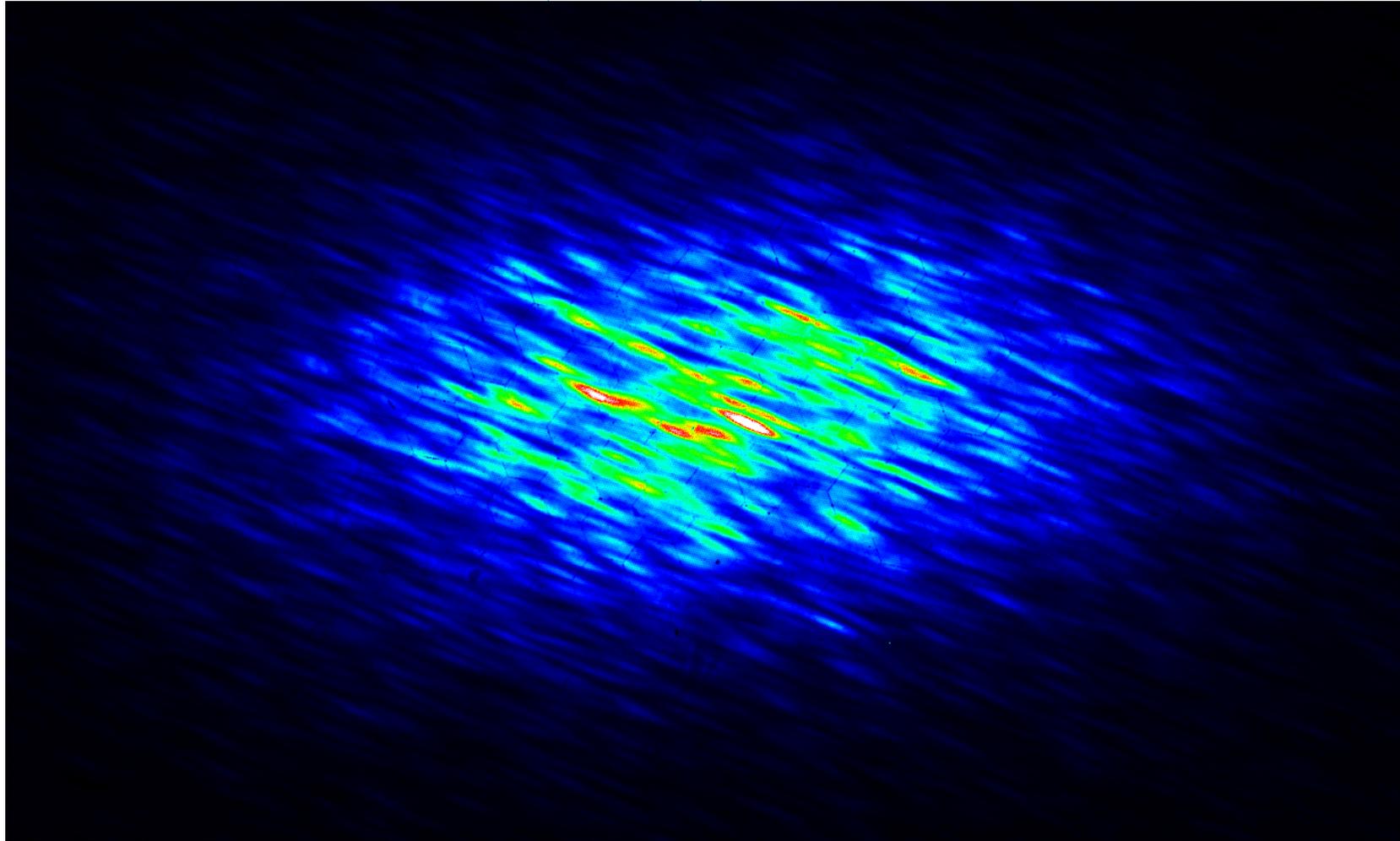
J. M. Rodenburg et al, Phys. Rev. Lett. 98 034801 (2007)



Niobium (110) Thin Film Grains

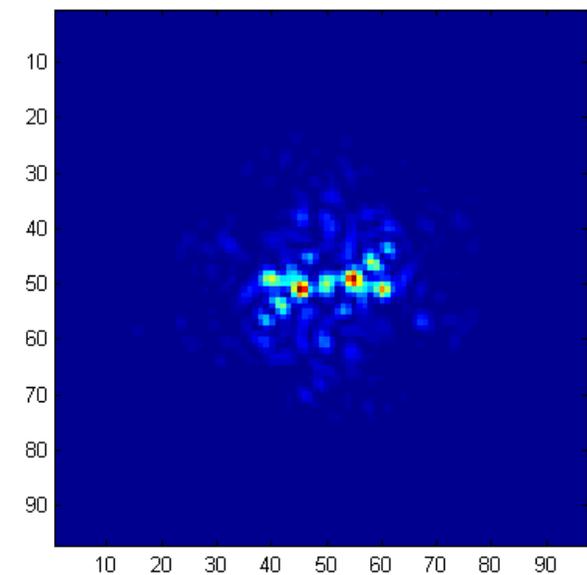
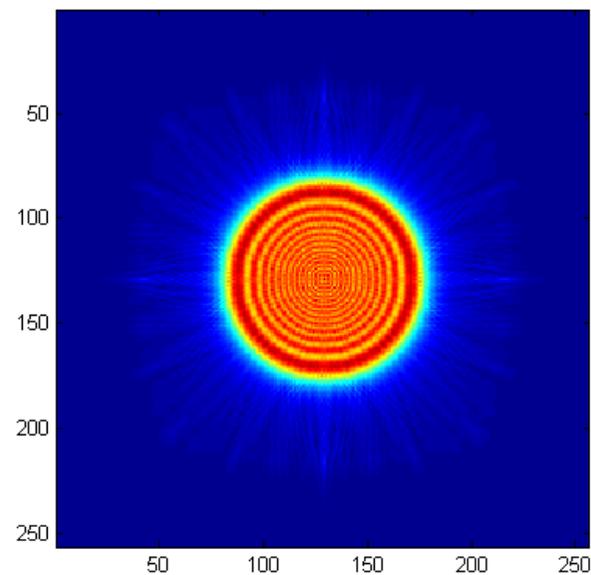
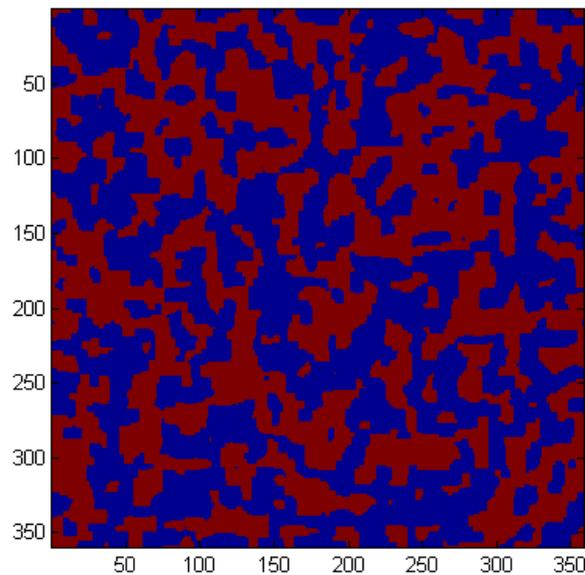
1 μm steps across 3 μm beam of KB mirror focus

Richard Bean, I-16, Nb110-35 Jan 2009



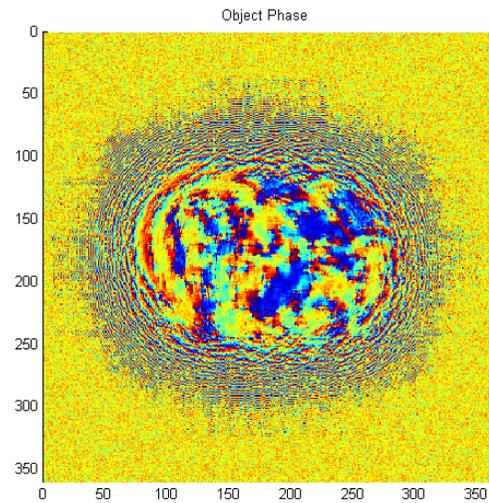
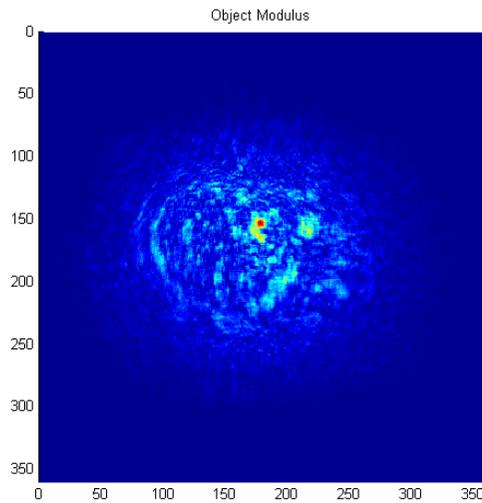
Ptychography phasing tests

Diffraction patterns computed from i) simulated domain array ii) propagated pinhole function with an overlap of $\sim 80\%$ between adjacent positions.

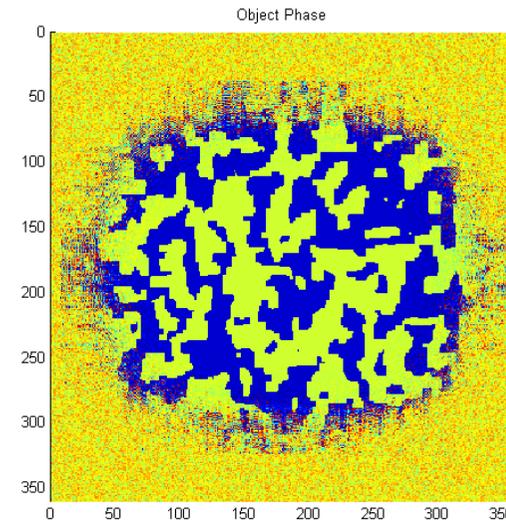
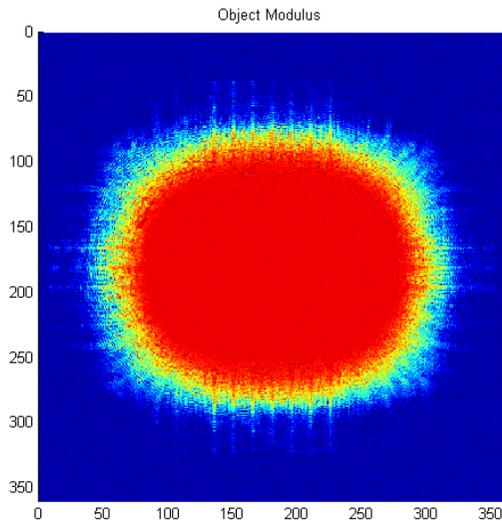


Ptychography phasing tests

Sheffield Ptychography algorithm starting with an array of random numbers in both amplitude and phase. 7x3 pinhole array. Richard Bean, Jan 2009

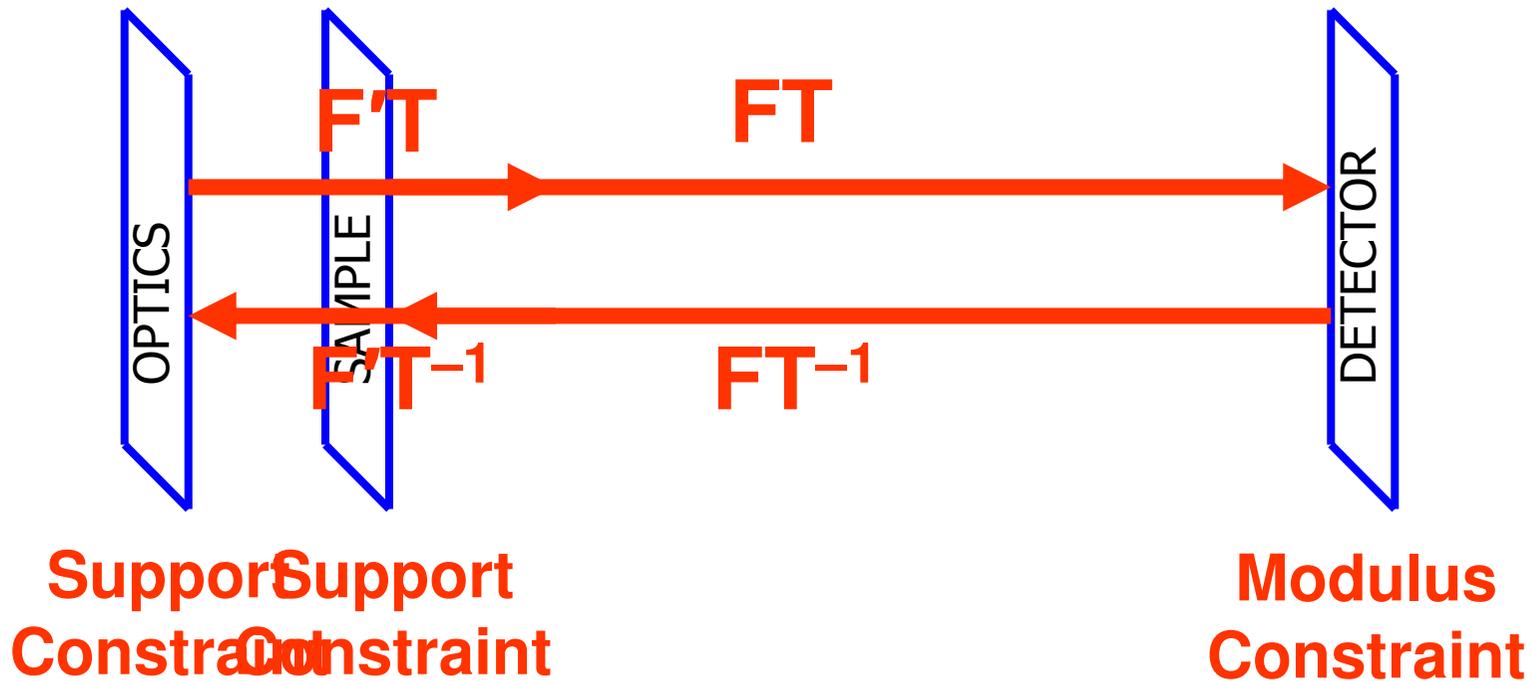


Amplitude and phase after 1 iteration.



Amplitude and phase after 20 iterations.

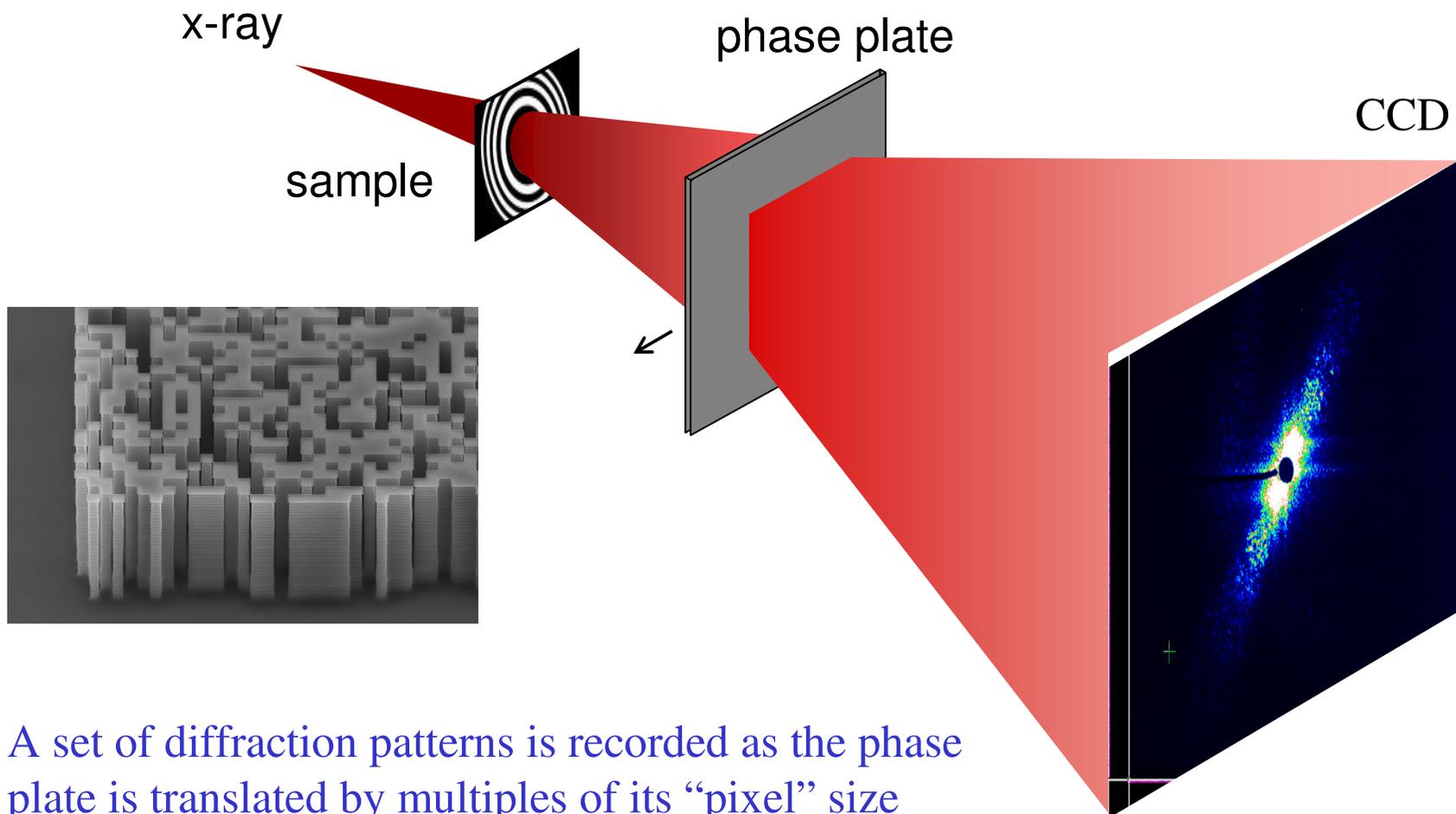
Coherent Diffractive Imaging General 3-plane Method



Imaging by Wavefront Modification

F. Zhang et al., Phys Rev A 75 (2007)

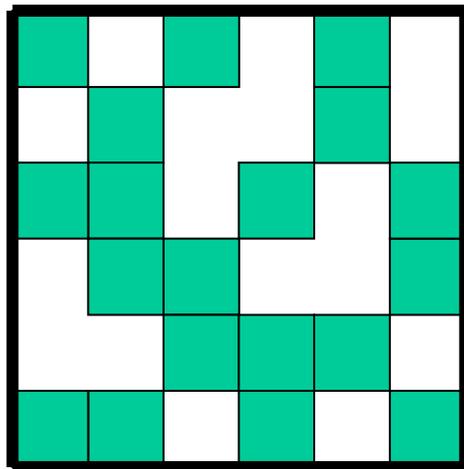
I. Johnson et al., Phys Rev Lett 100 (2008)



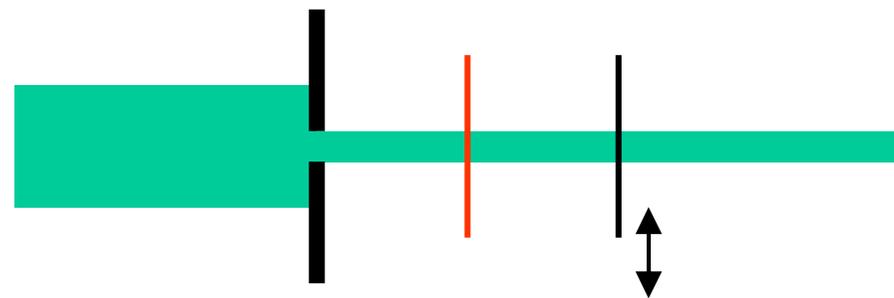
A set of diffraction patterns is recorded as the phase plate is translated by multiples of its “pixel” size

'Random' phase plate analyser

Joan Vila (PSI), Fucai Zhang (Sheffield)



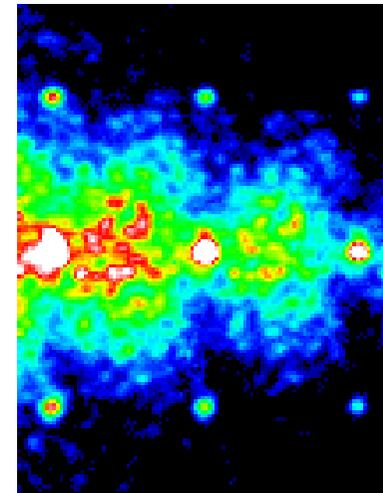
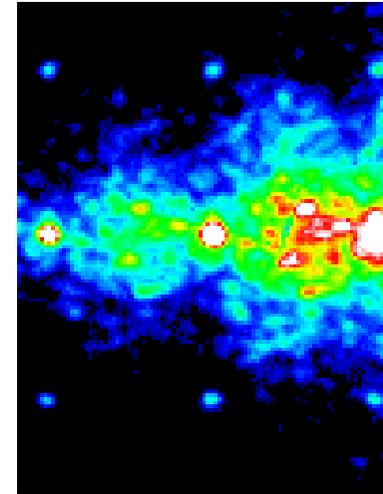
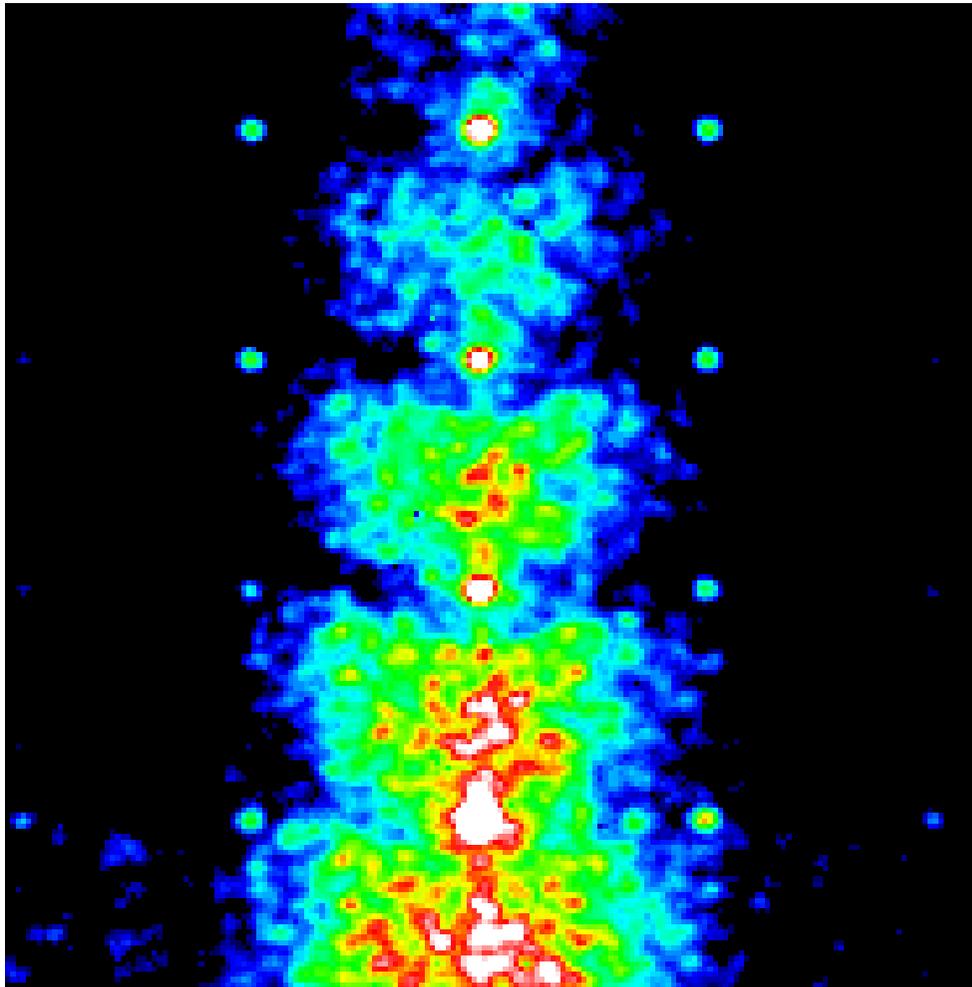
↔
 $1\ \mu\text{m}$



Pinhole Sample Analyser

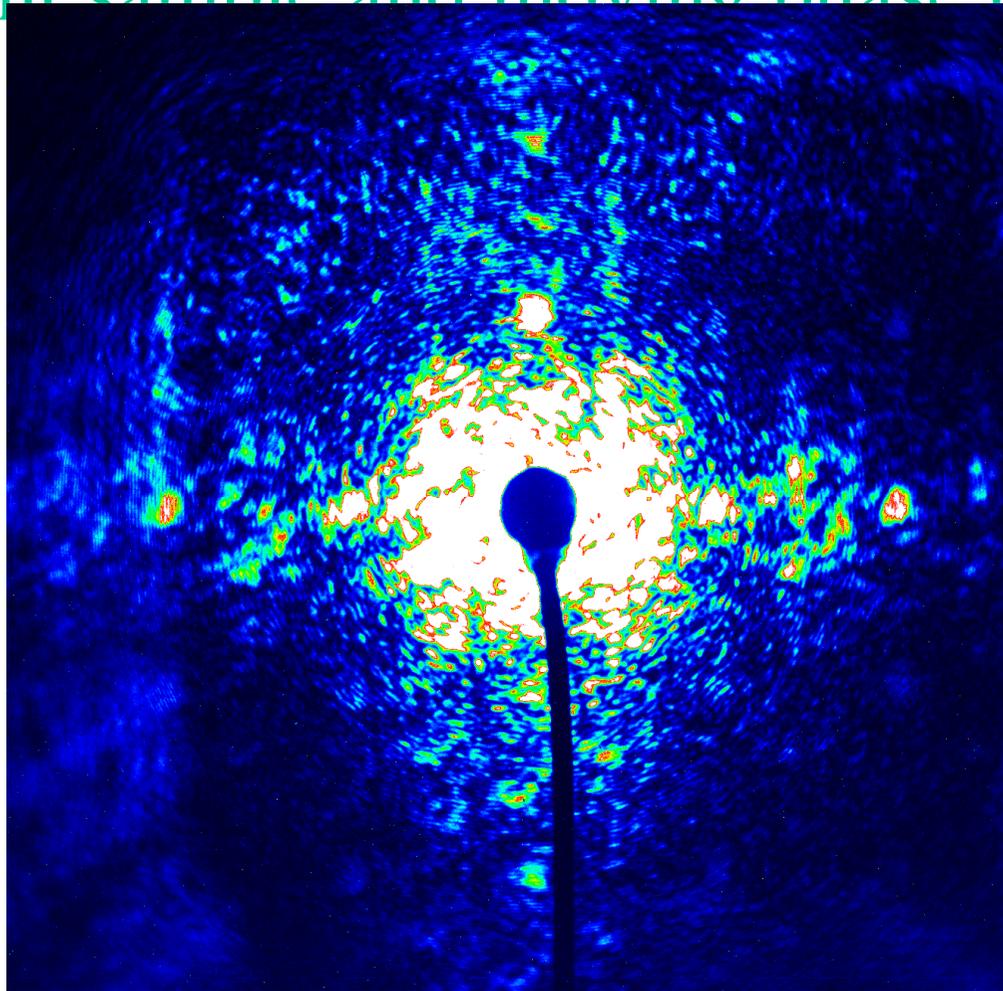
Phase plate scan near forward direction

1 μm step per frame



coll_1209-0109

Paint sample and moving phase plate



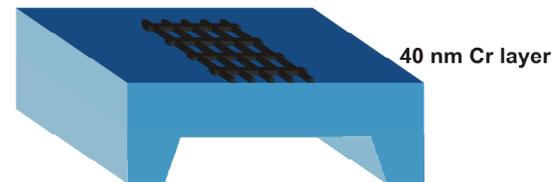
Phase plate fabrication

Joan Vila-Comamala, PSI

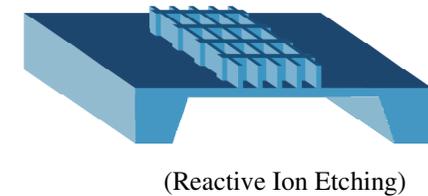
1) e-beam lithography



2) Pattern transfer to a Cr layer by evaporation and lift-off

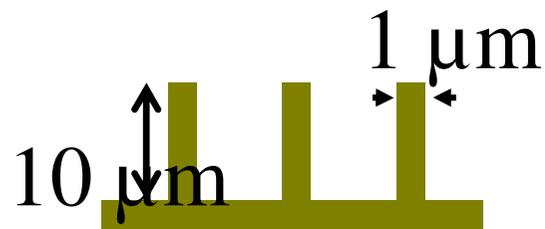


3) Pattern transfer to Si by RIE

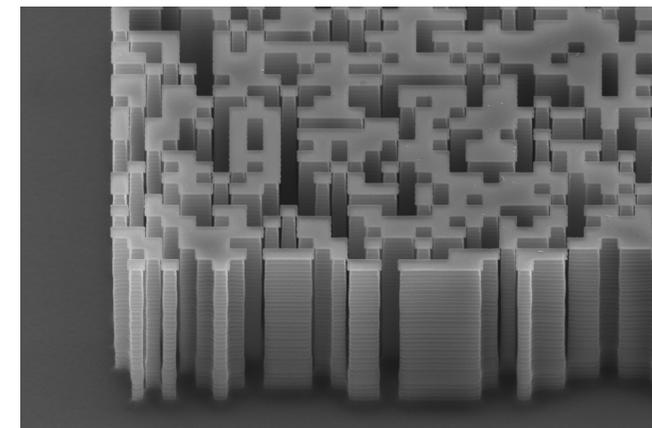
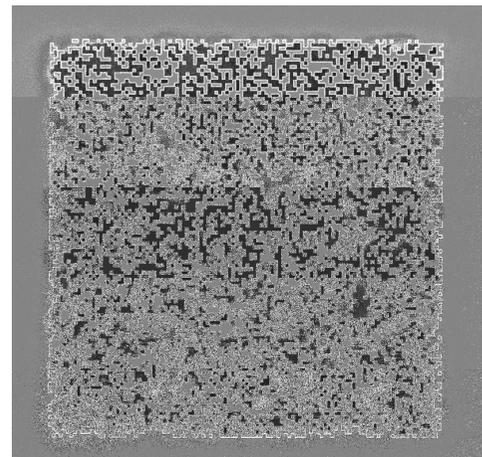


At 8.0 keV ($\lambda=0.155$ nm) $\rightarrow \delta = 7.67 \cdot 10^{-6}$ **$h=10.10$ μm**

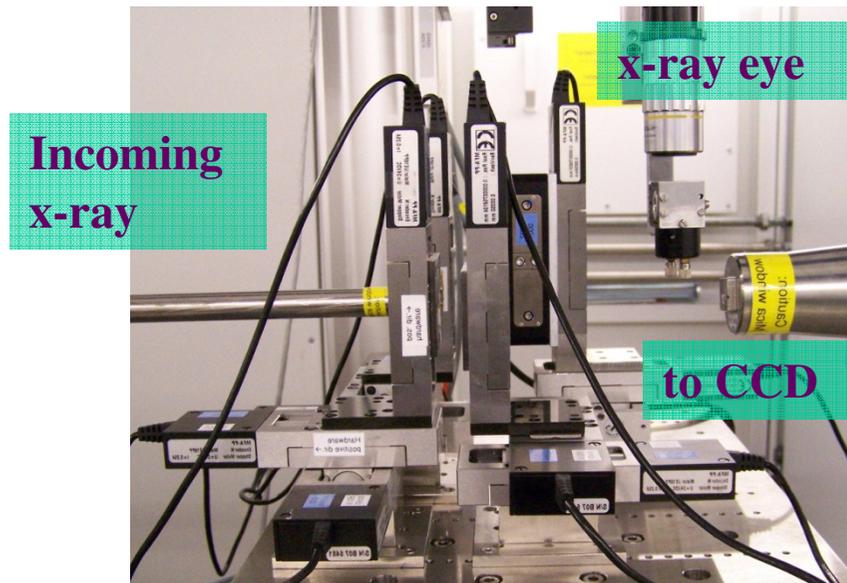
At 10.0 keV ($\lambda=0.124$ nm) $\rightarrow \delta = 4.88 \cdot 10^{-6}$ **$h=12.70$ μm**



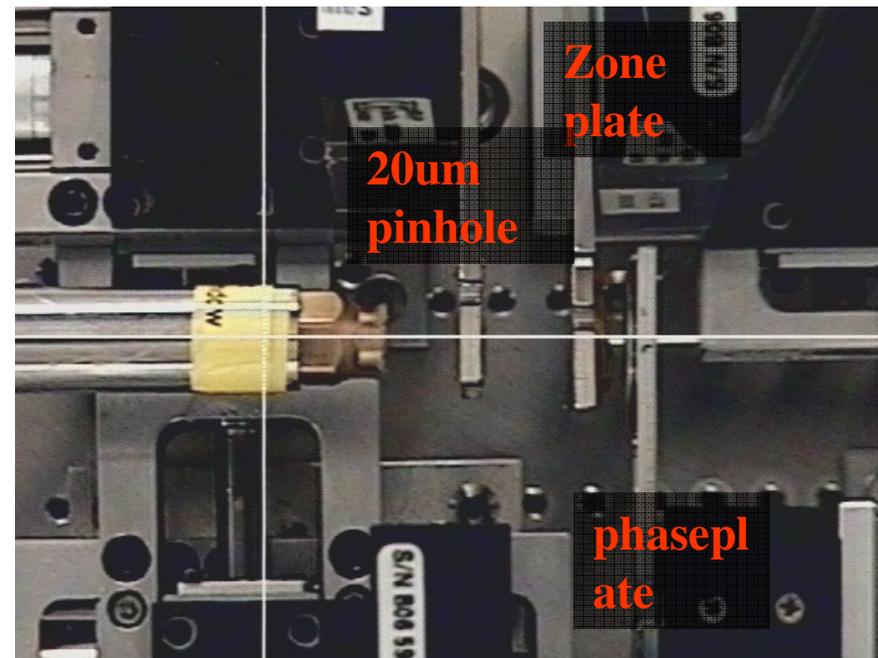
Cross section



Experimental setup cSAXS (SLS)

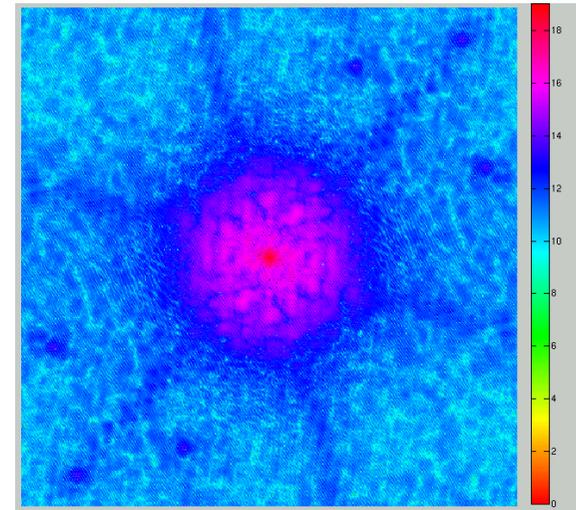
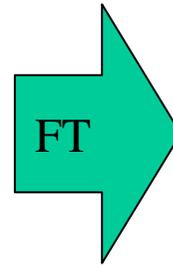
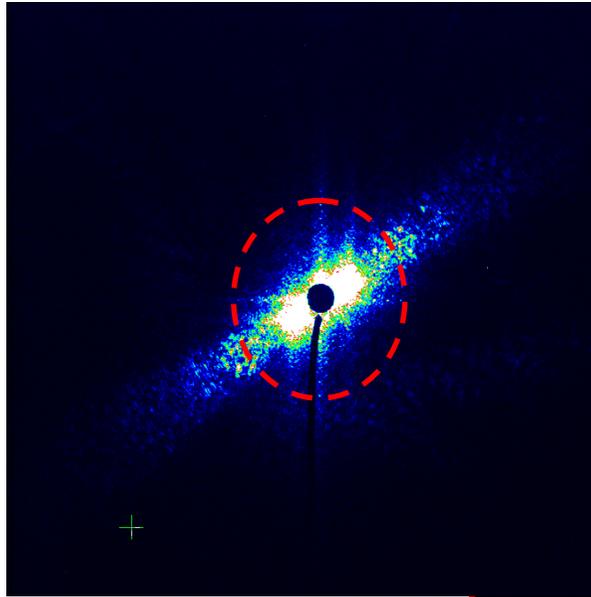


Side view

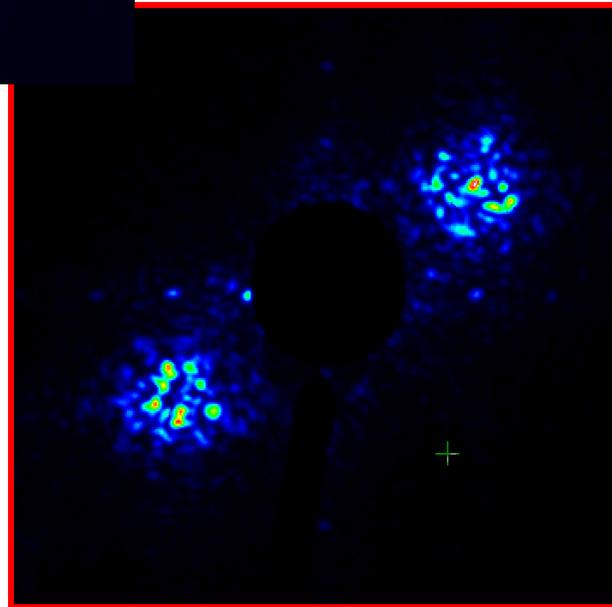


Top view (zoomed in)

Results: Zone Plate as object

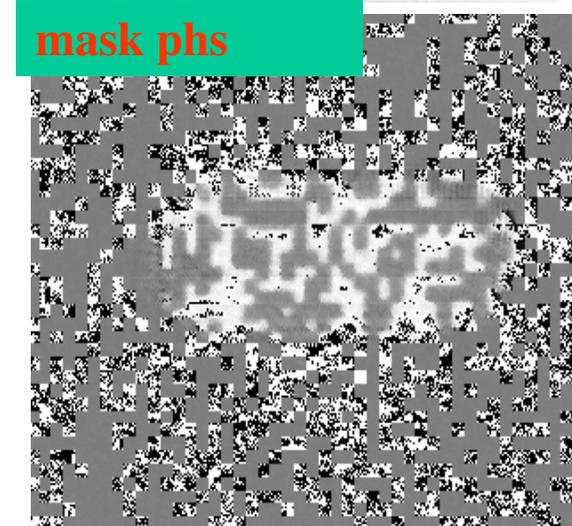
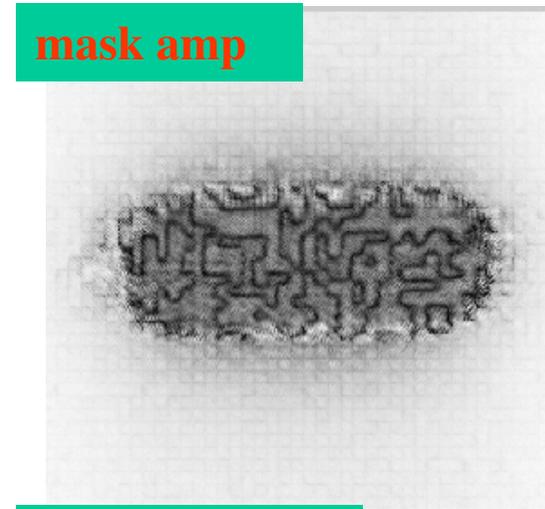
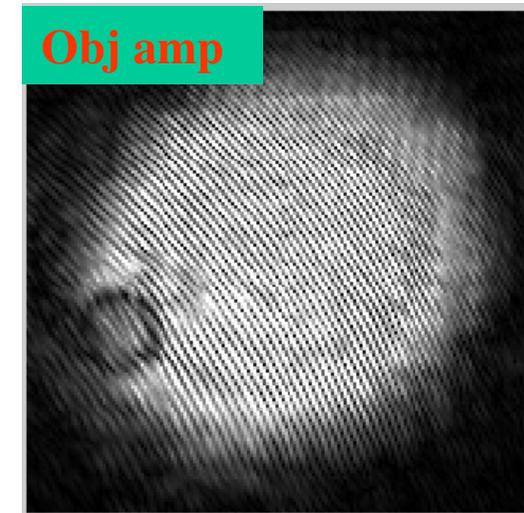


Auto correlation function



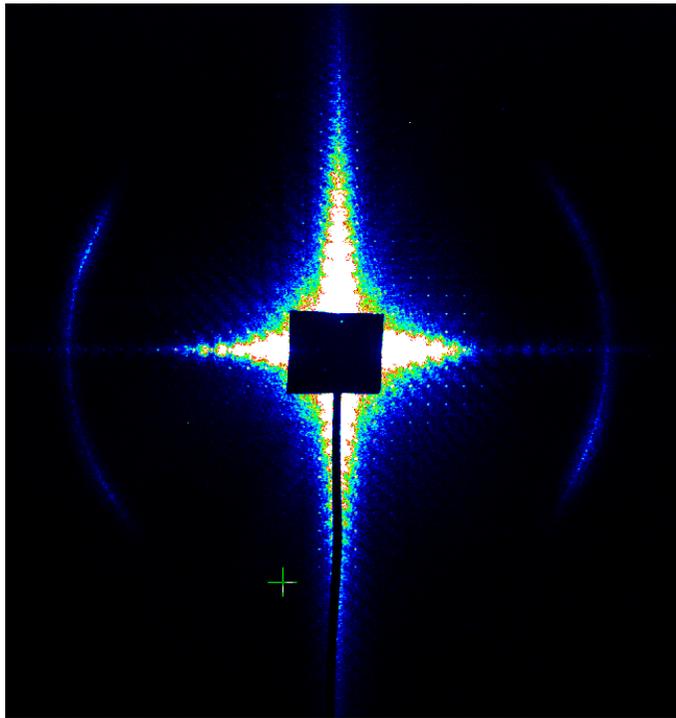
Reconstruction of Phase plate scan

1 μm step per frame (512x512)

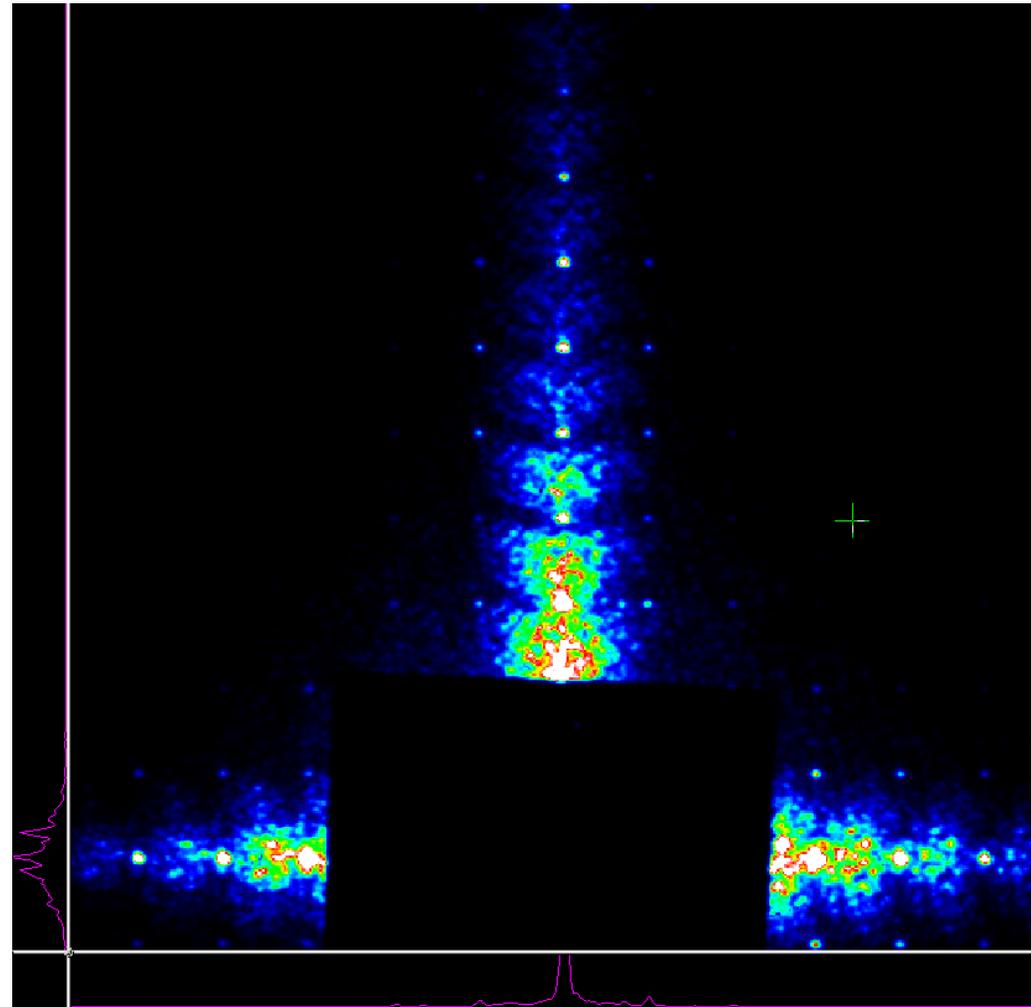


Reconstruction by F. Zhang (paper in preparation)

Collagen rat tail tendon as sample

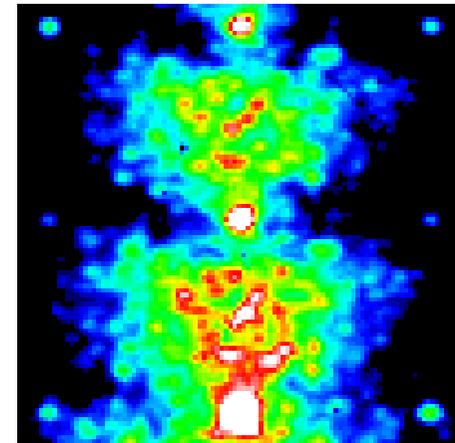
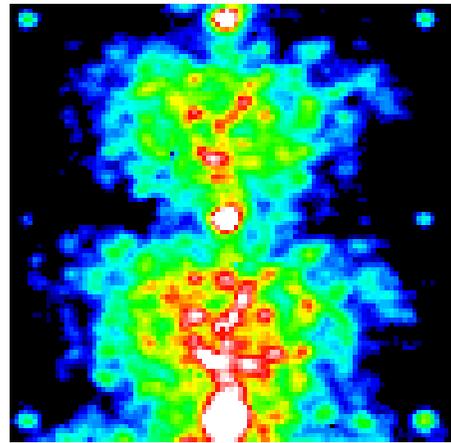
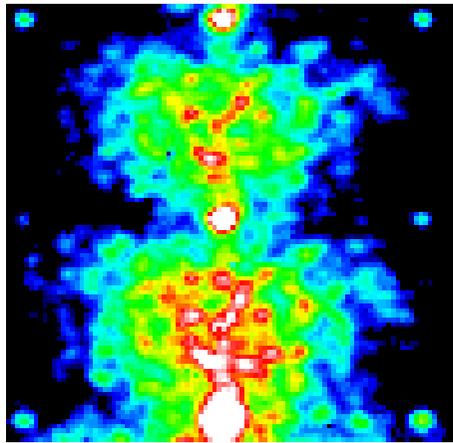


- * 0th order “repeated” in the meridian and equator
- * Strong phase modification due to phase plate!
- * No successful reconstruction



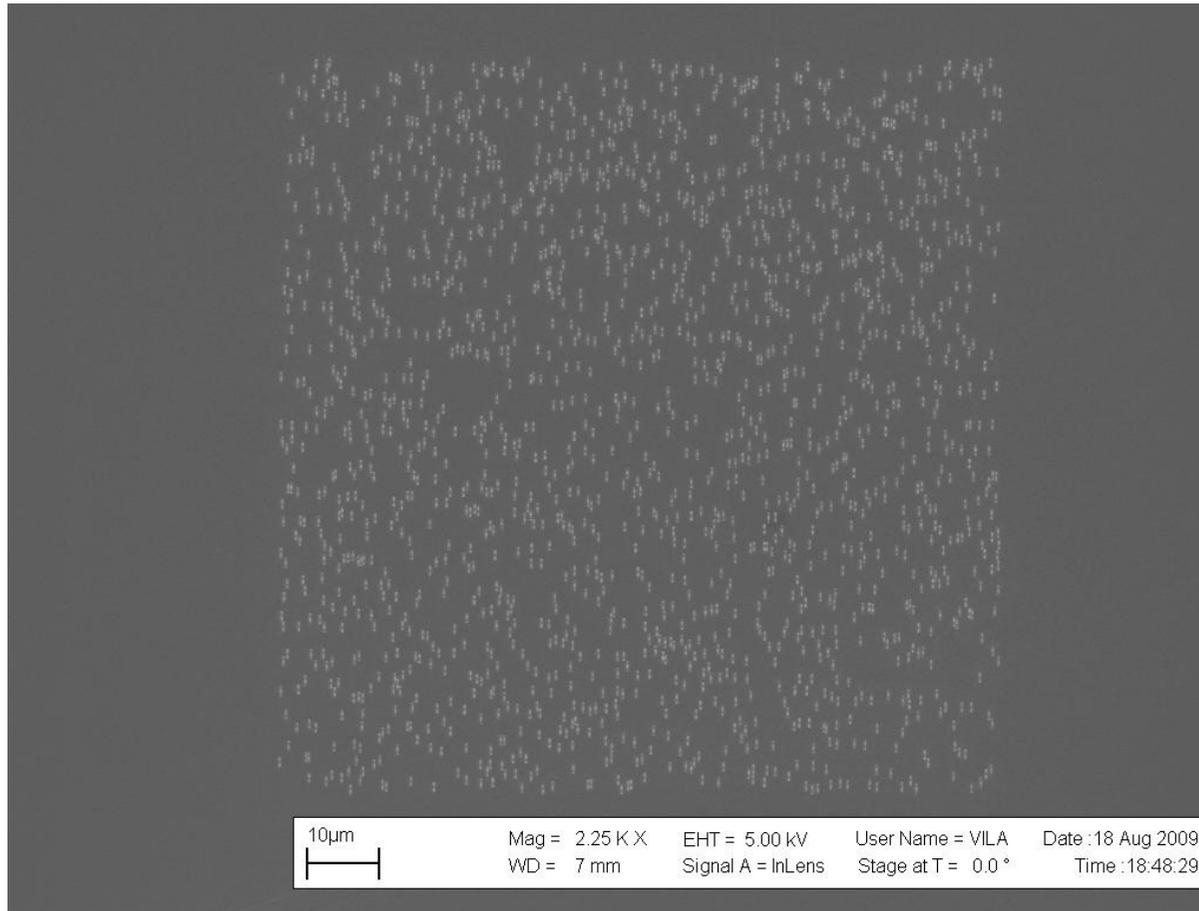
Collagen, buffer and empty cell

phplateFZP-236, -246 and -249



New phase plate design

R. Bean and J. Vila-Comamala, PSI



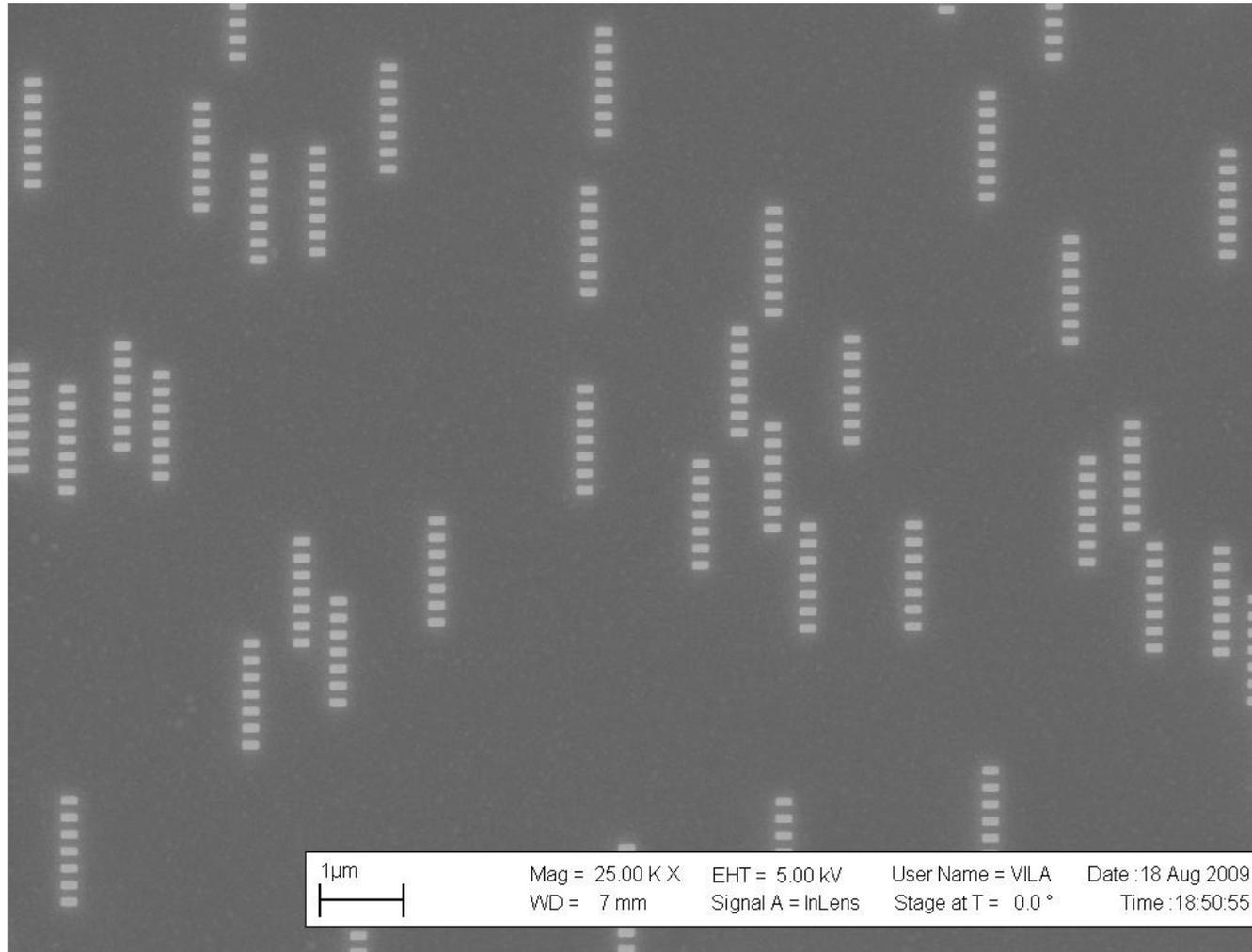
Pixel size = 10 nm
Total size = 100 x 100
microns²

Motif = 7 rectangles (200
nm x 100 nm), period of
200 nm (close to 3rd
collagen diff order)

•

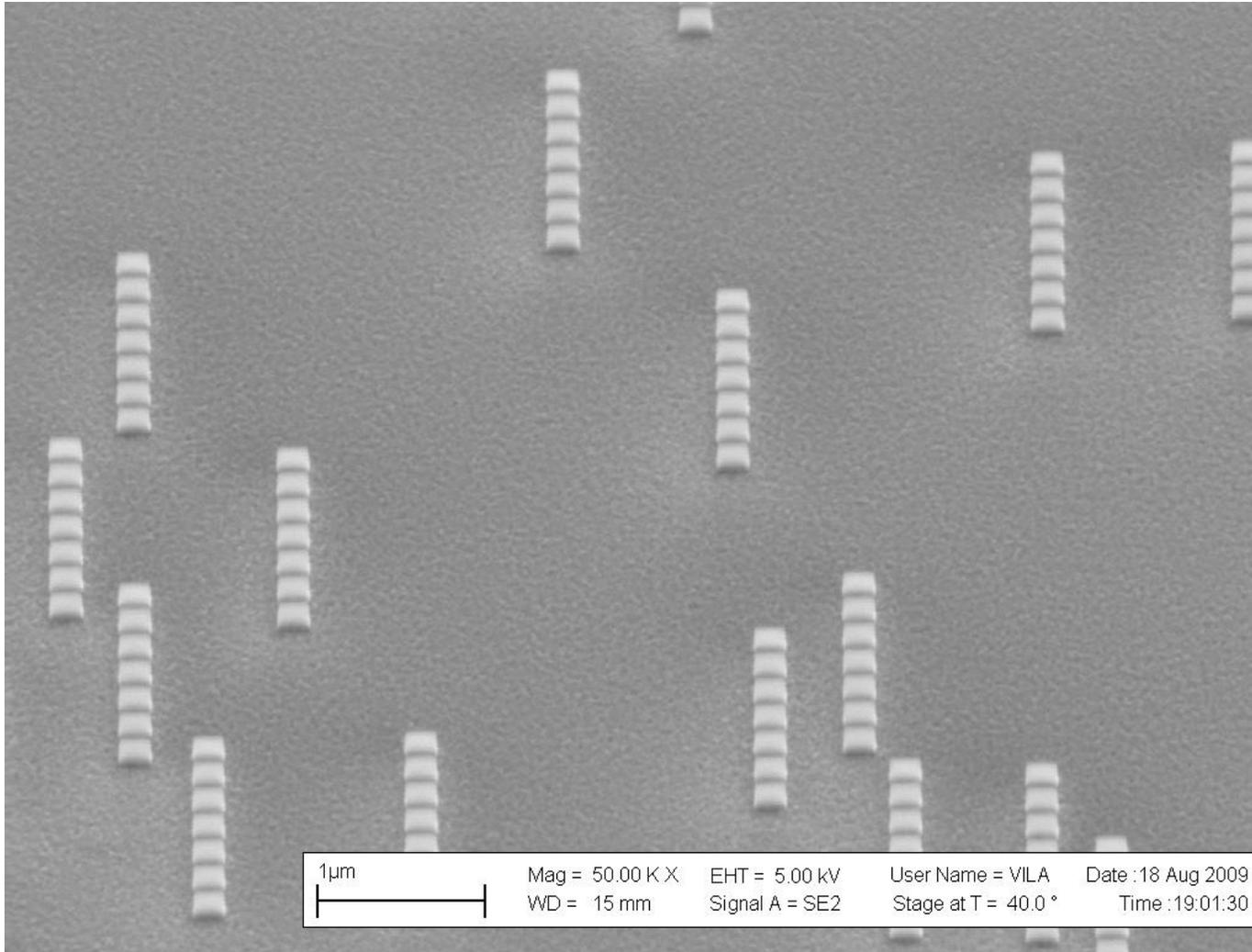
New phase plate design

R. Bean and J. Vila-Comamala, PSI



New phase plate design

R. Bean and J. Vila-Comamala, PSI



Conclusions

- Small crystal imaging
- Strains visible as real-space phase
- Curved beam resembles strain
- Phase plate demonstrated
- New custom phase plates
- Possible experimental phase measurement