

Possible Phasing Methods for Highly Strained Crystals

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Felisa Berenguer Diamond Light Source

Richard Bean

Fucai Zhang

Marcus Newton

Coherence Workshop

SRI-09 satellite

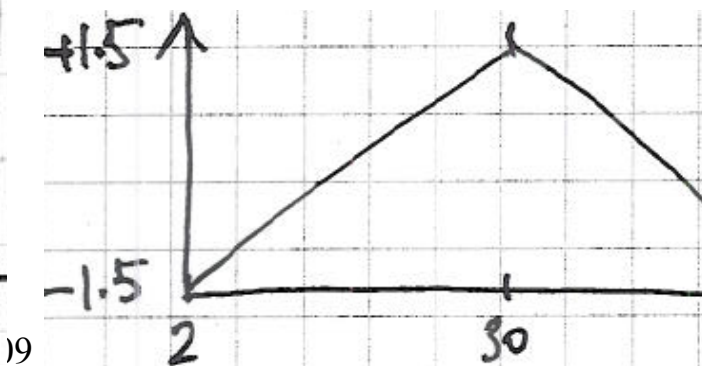
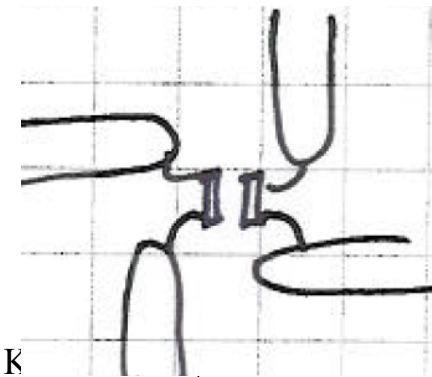
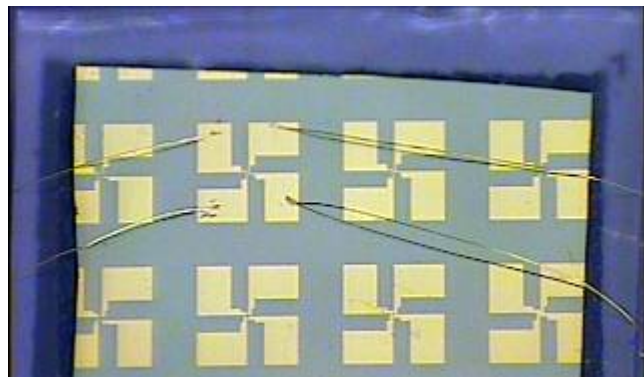
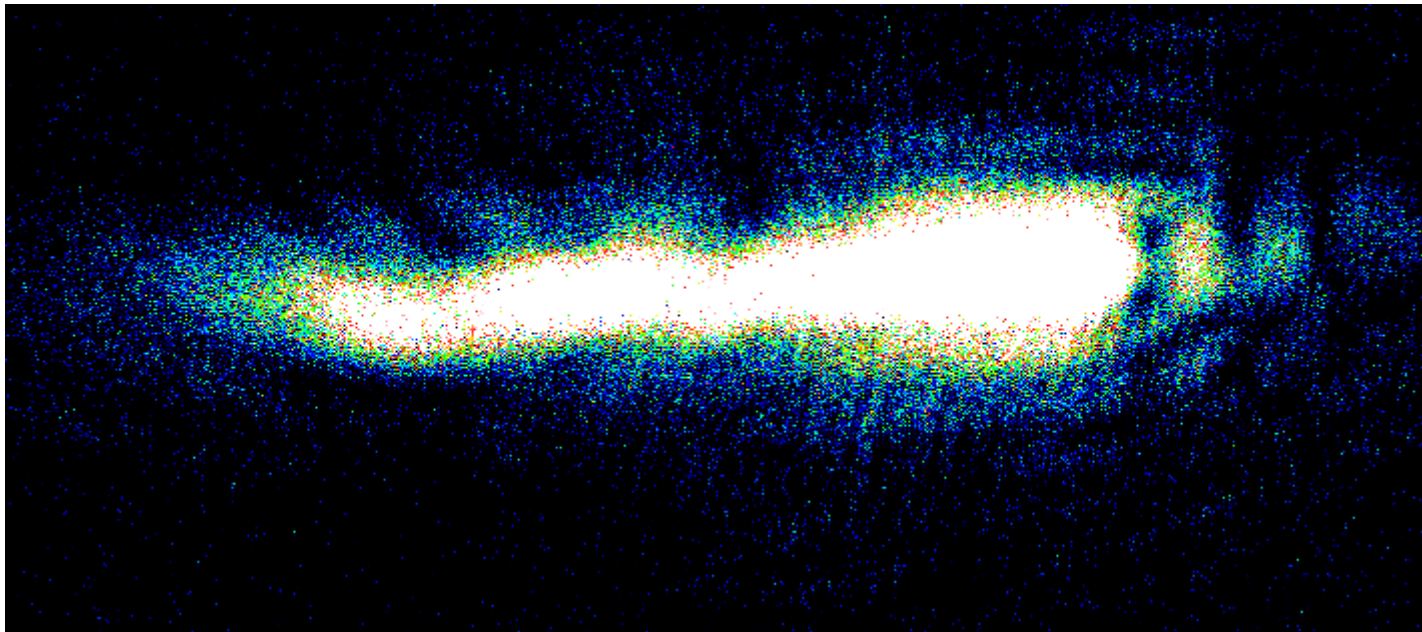
October 2009

Outline

- Coherent X-ray Diffraction
- Wired ZnO experiments
- Simulation of small crystals and strain fields
- Phase-plate analyser
- Phase plate fabrication
- Preparations for XFEL

Apply Electric Field along ZnO NC

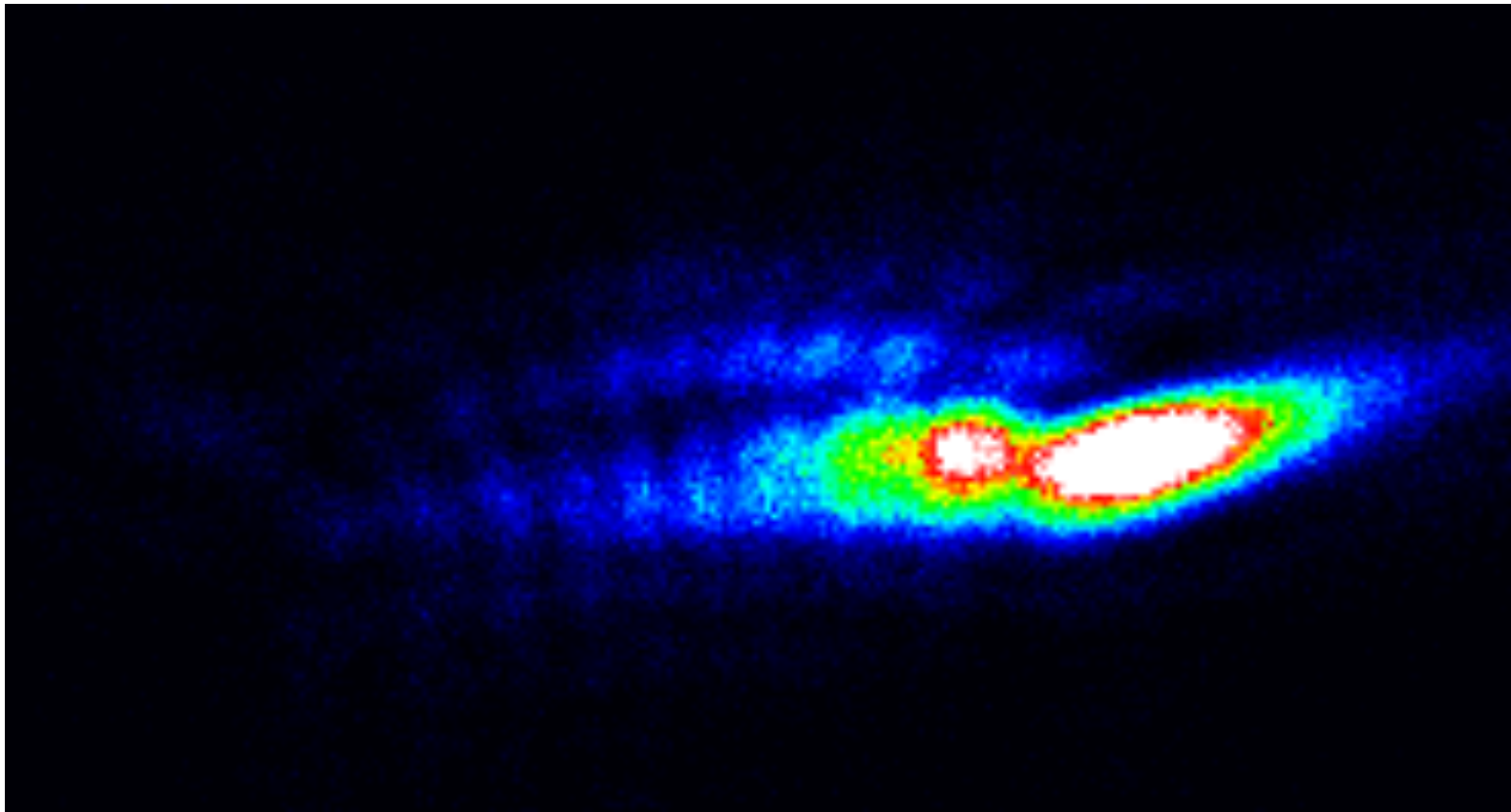
Marcus Newton. 34-ID-C zno808-18 Aug 2008



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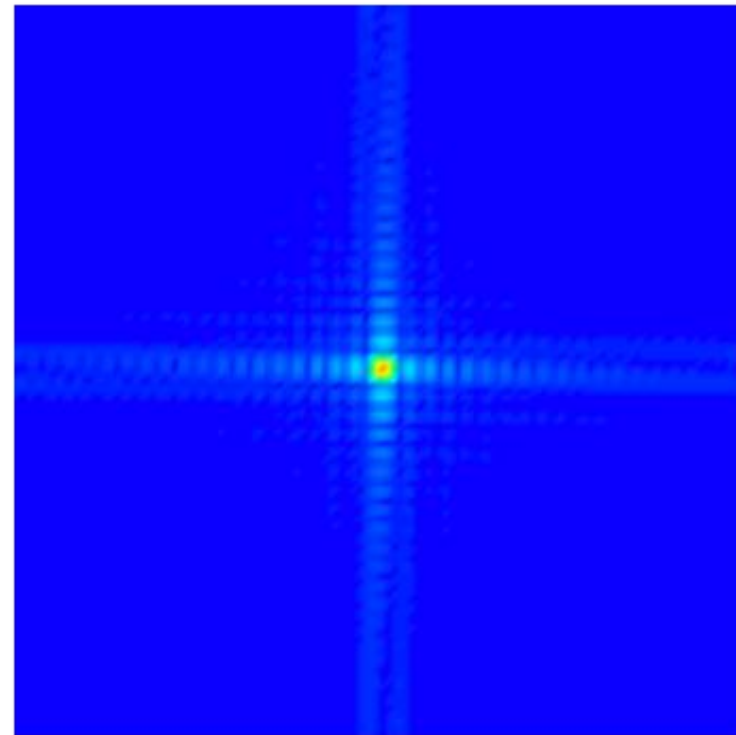
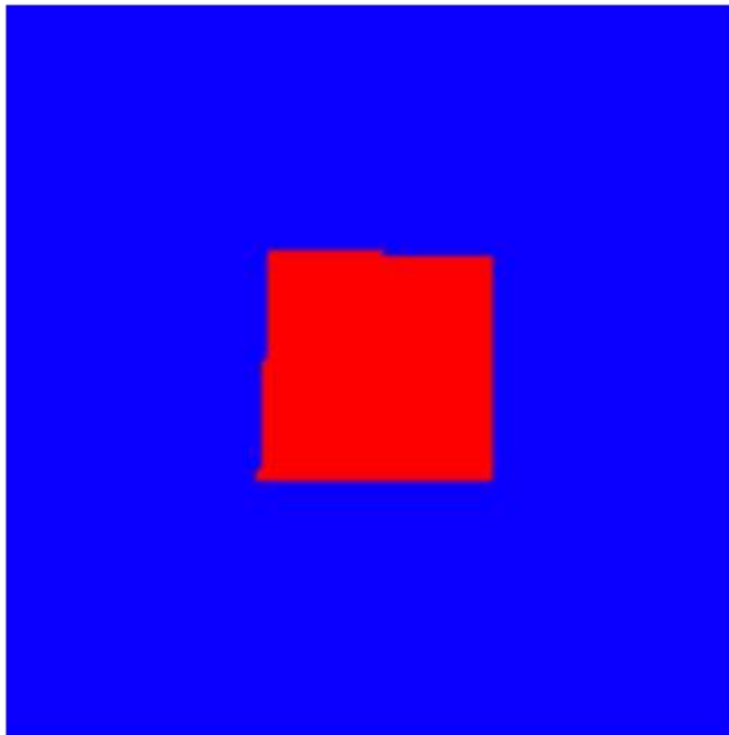
ZnO409-44 wired ZnO crystal



FFT simulations of phase objects

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

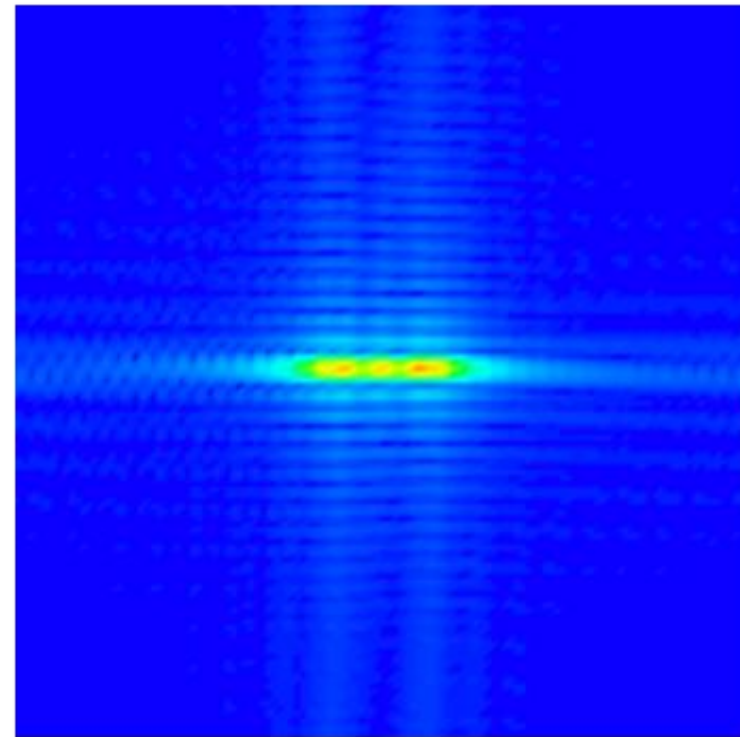
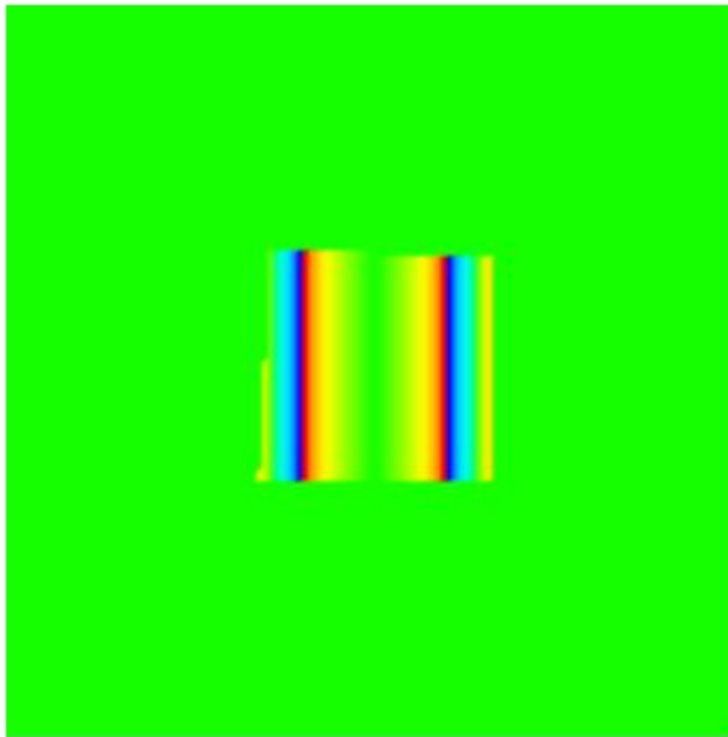
n=9 curY=0.0 curZ=0.0



FFT simulations of phase objects

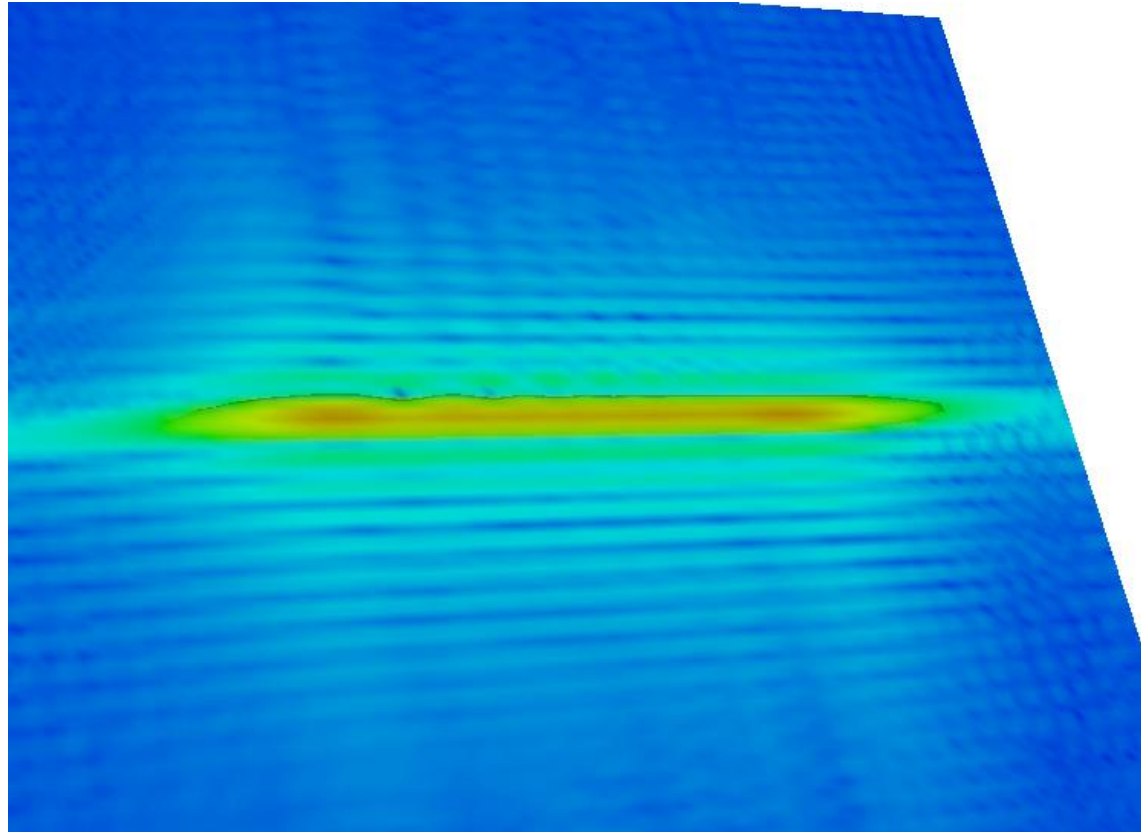
128x128x128 array 20x20x20 cube rotated (0.03 rad)³

n=9 curY=0.0 curZ=0.02



FFT simulations of phase objects

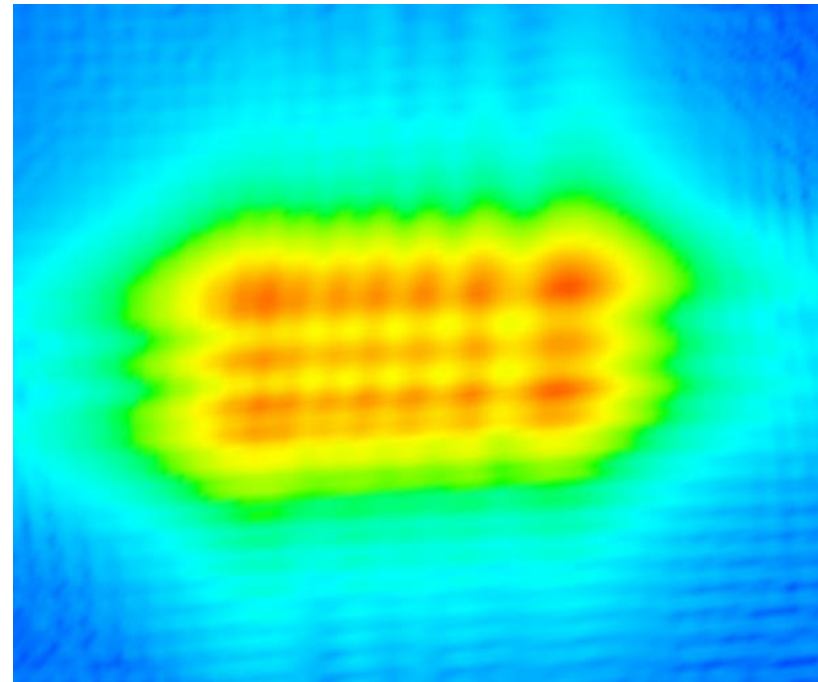
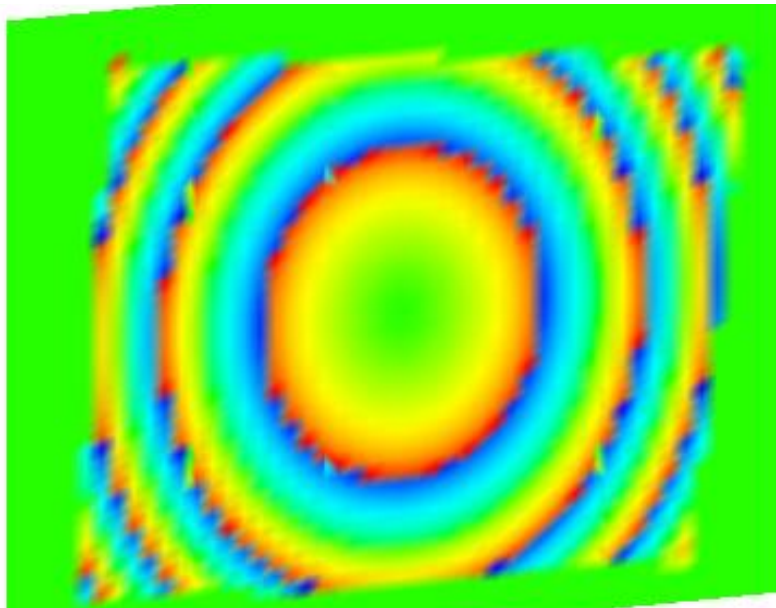
128x128x128 array 20x20x20 cube rotated (0.05 rad)³



FFT simulations of phase objects

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

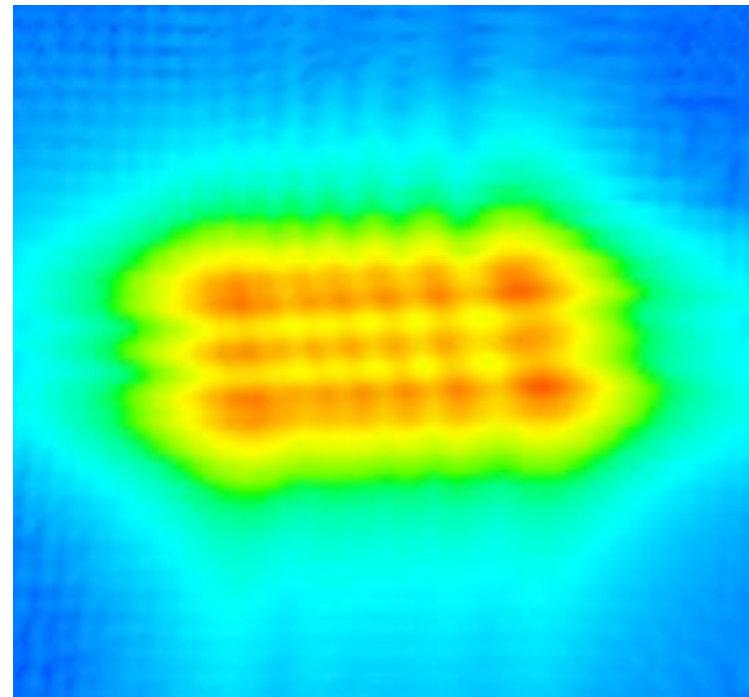
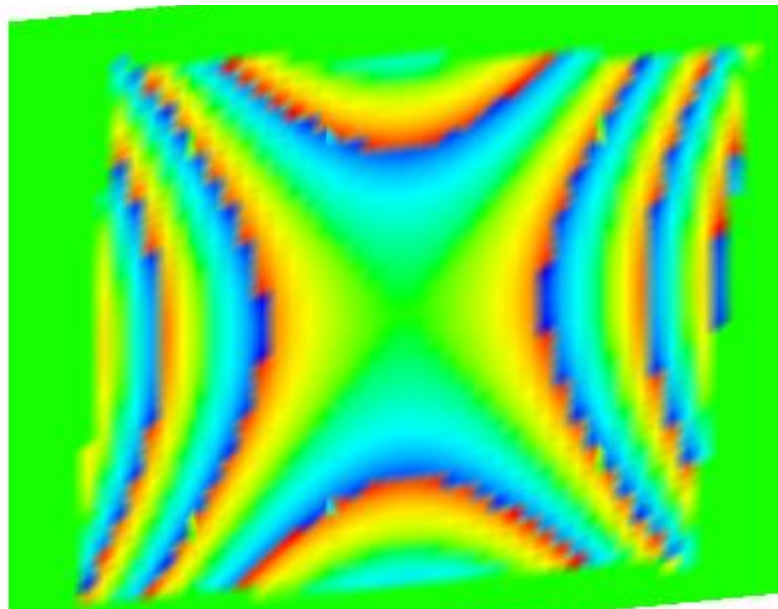
$n=5$ $\text{curX}=0.03$ $\text{curY}=0.02$



FFT simulations of phase objects

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

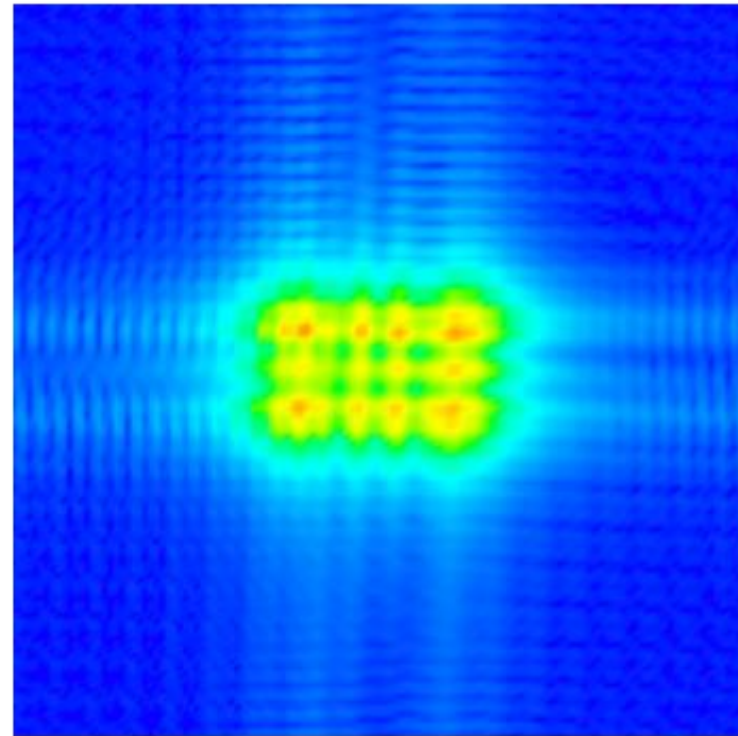
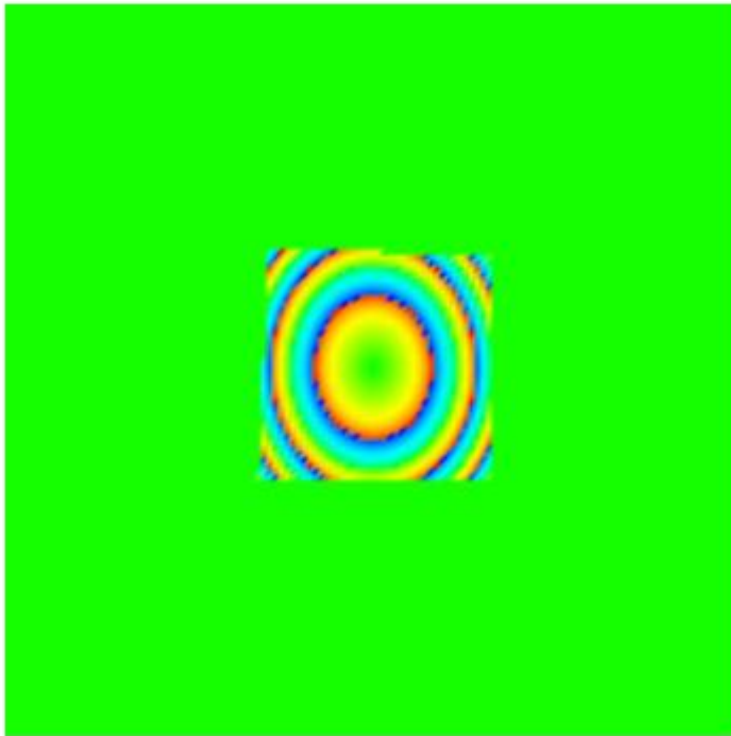
$n=6$ $\text{curX}=0.03$ $\text{curY}=-0.02$



FFT simulations of phase objects

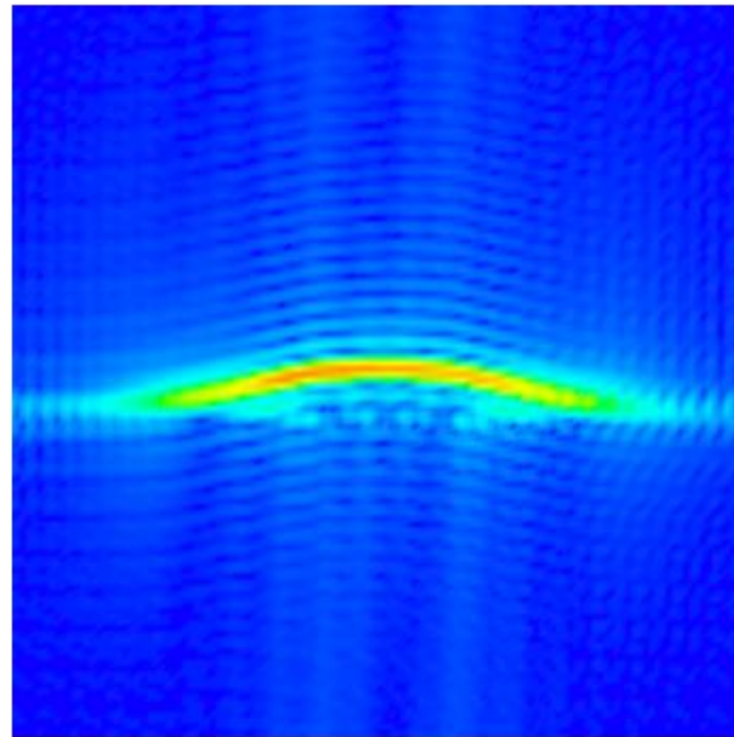
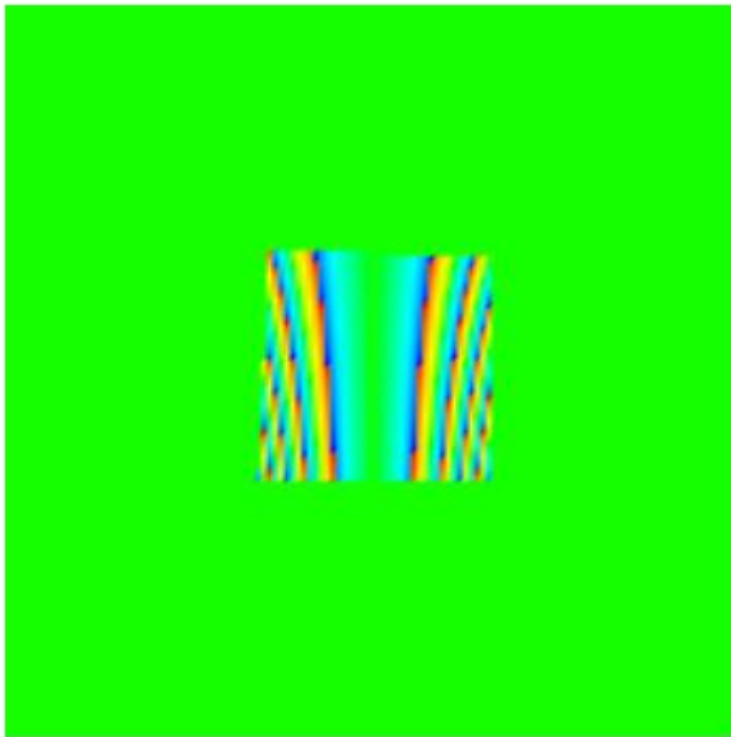
128x128x128 array 20x20x20 cube rotated (0.03 rad)³

n=8 curY=0.02 curZ=0.03



Poly_quad_ph2 curved phases

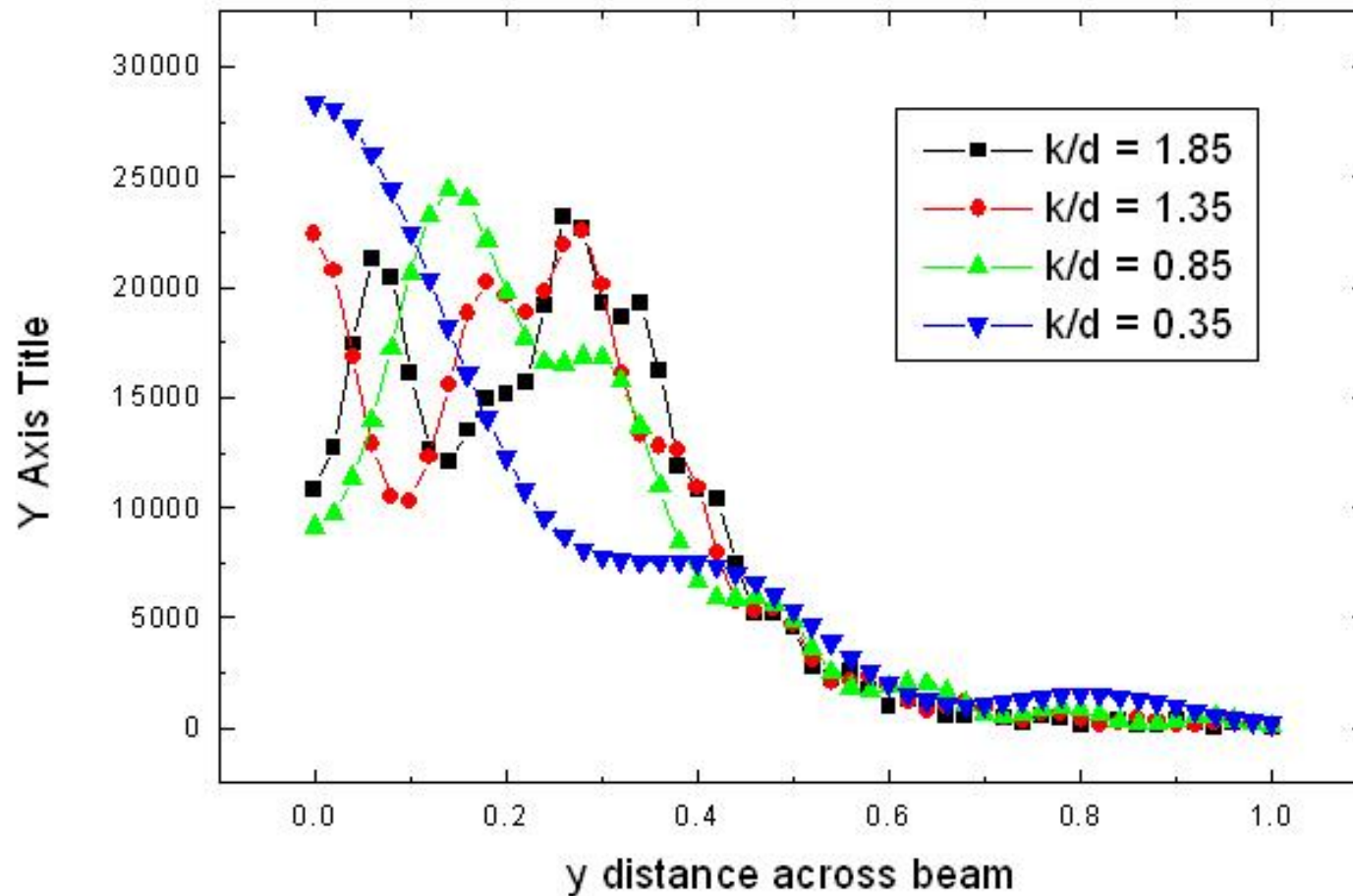
curY=50 curZ=0.001, $(y-\text{curY})*\text{curZ}*z*z$



Evaluate Fresnel Integral Directly

Fres05.wk1 Fresnel diffraction

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



Proposed Phasing Method (see Clare's poster)

- 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>

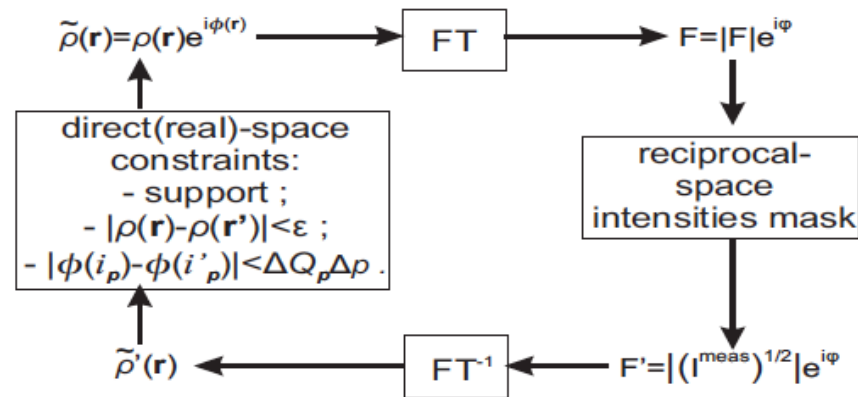
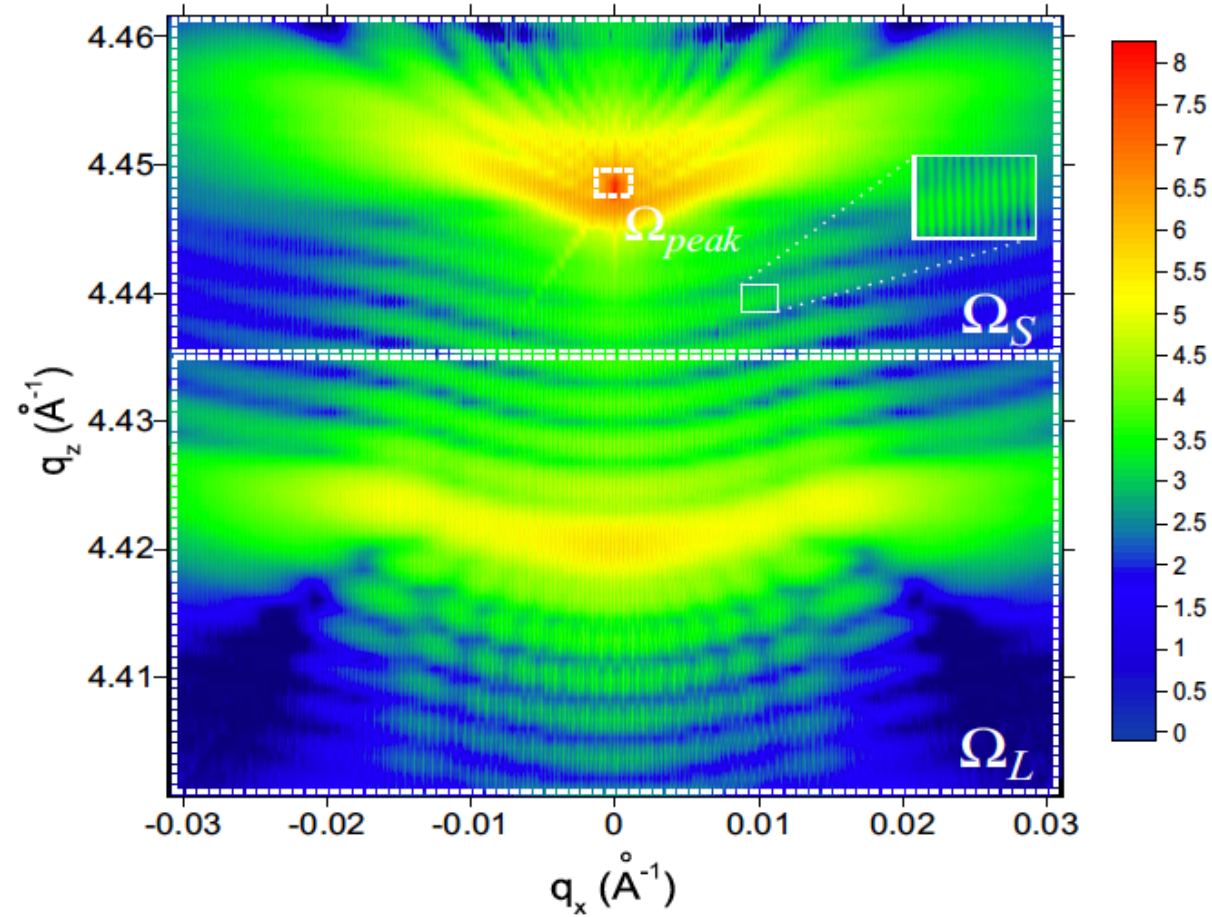


FIG. 1: Schematic diagram of the iterative loop of our phase retrieval algorithm. \mathbf{r} and \mathbf{r}' in "direct(real)-space constraints" box correspond to the neighbouring points within one constituent crystal and ϵ defines the threshold of electron density uniformity. i_p and i'_p are the neighbouring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

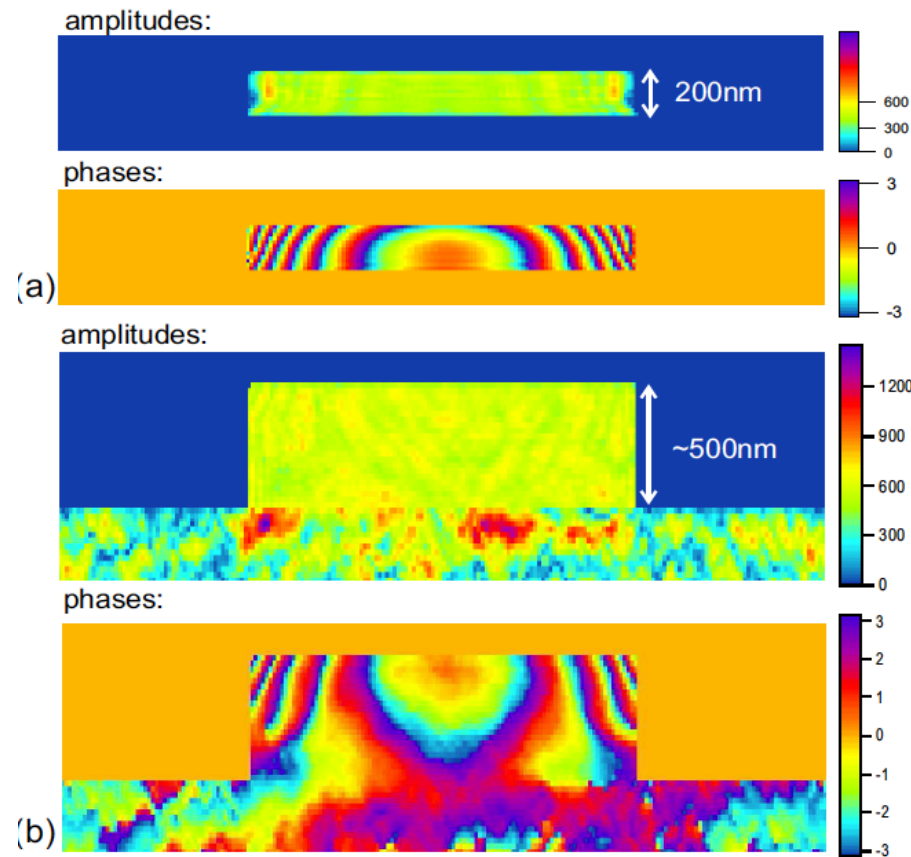
Andrey Minkevich (KFA Karlsruhe)

GaMnAs wires on GaAs



Andrey Minkevich (KFA Karlsruhe)

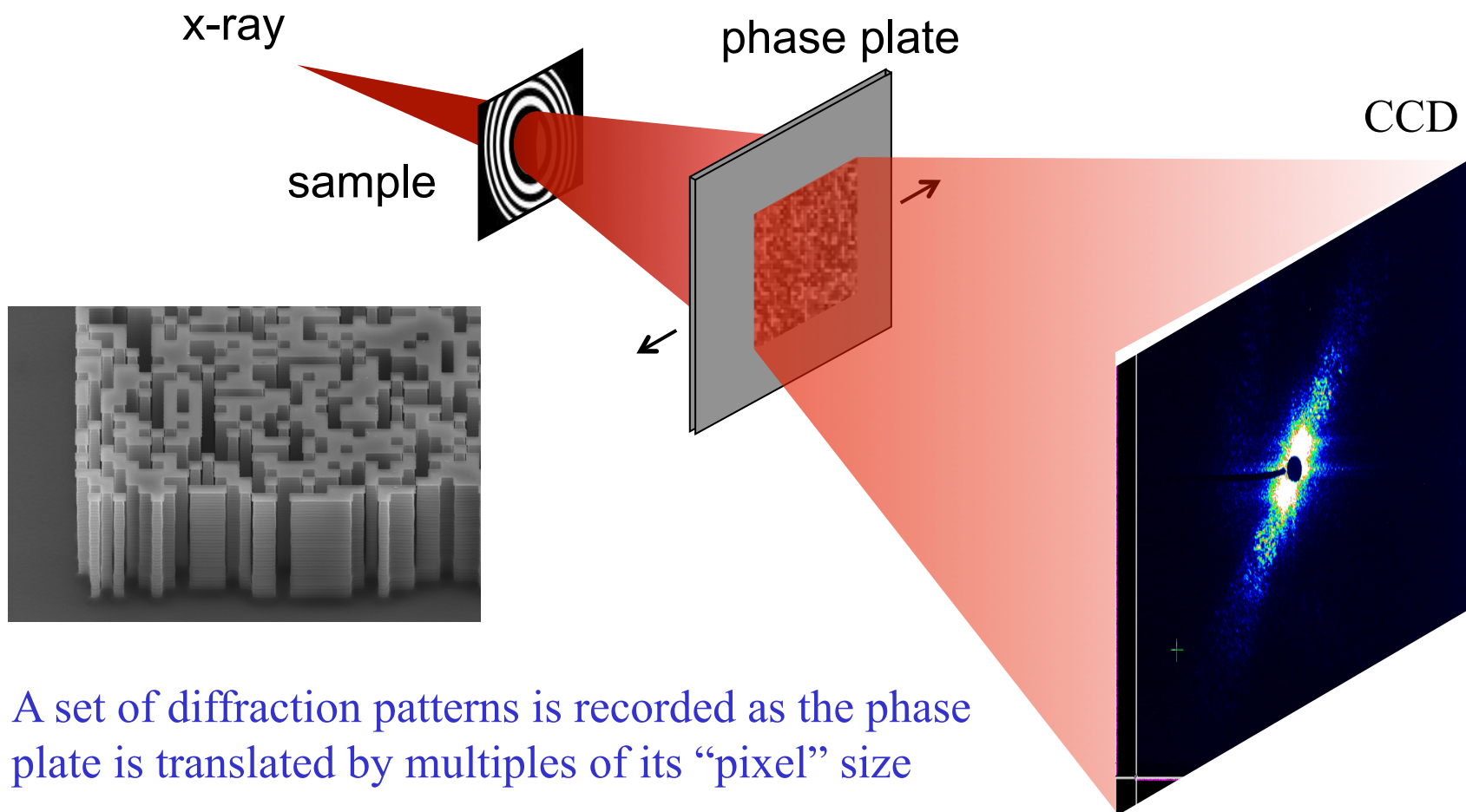
preprint <http://arxiv.org/abs/0909.4711>



Imaging by Wavefront Modification

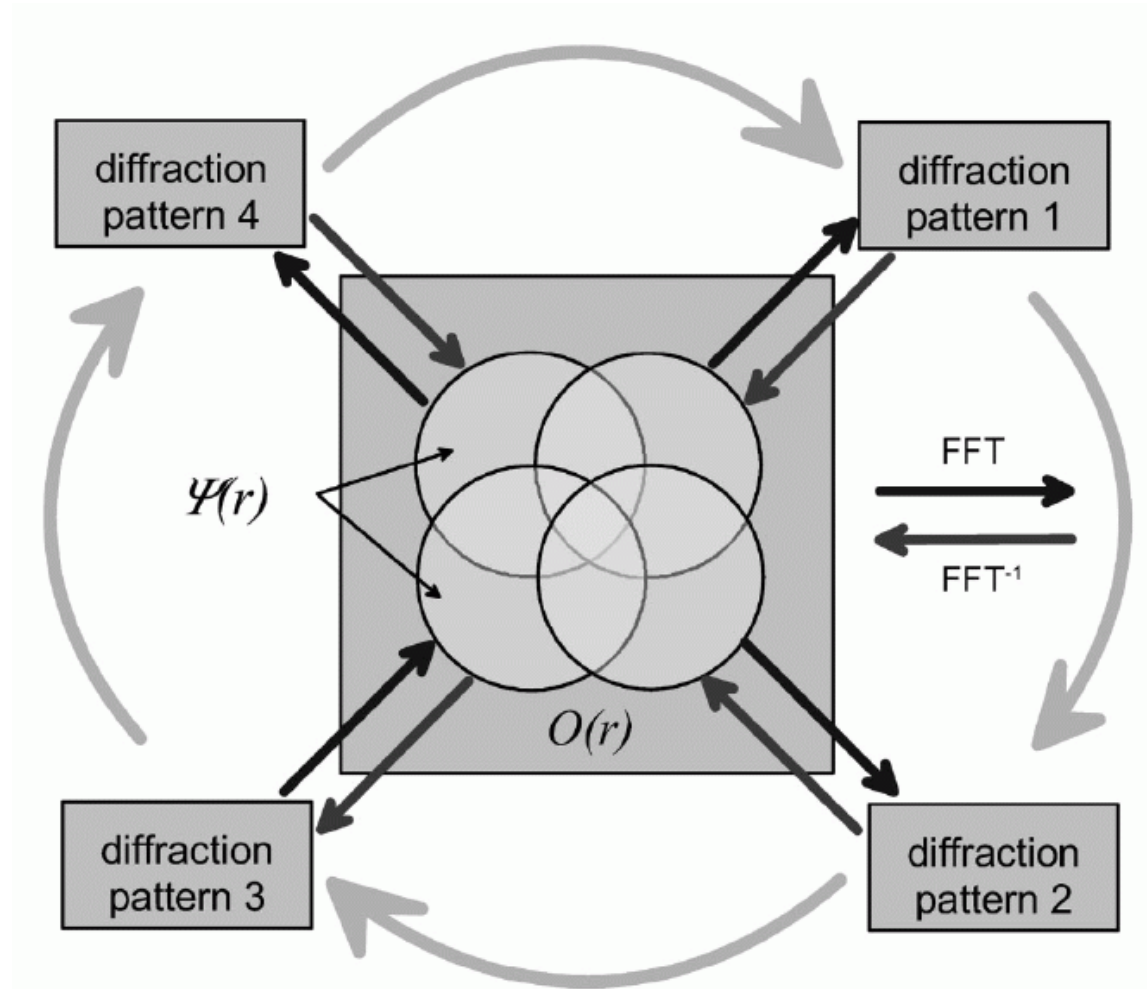
F. Zhang et al., Phys Rev A 75 (2007)

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



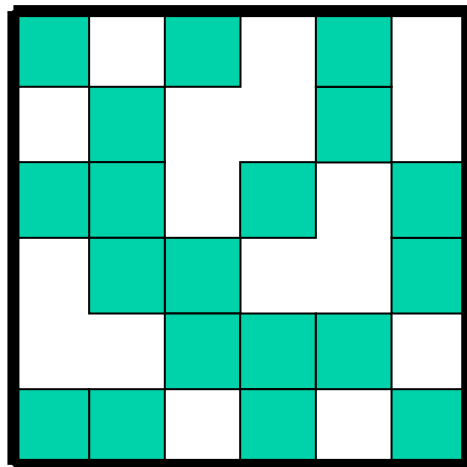
X-ray Ptychography

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

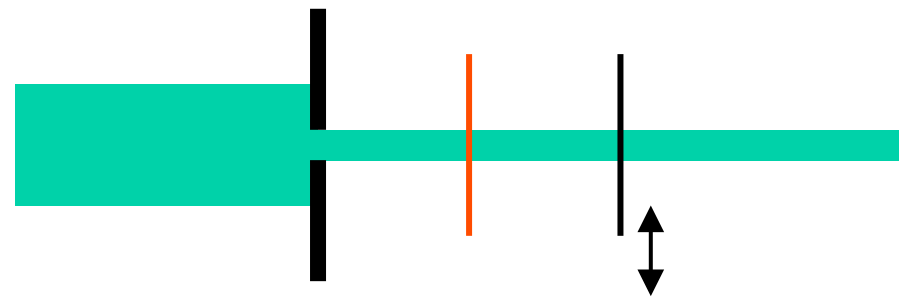


'Random' phase plate analyser

Joan Vila (PSI), Fucai Zhang (Sheffield)



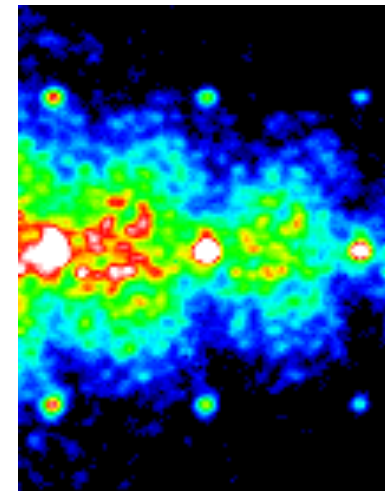
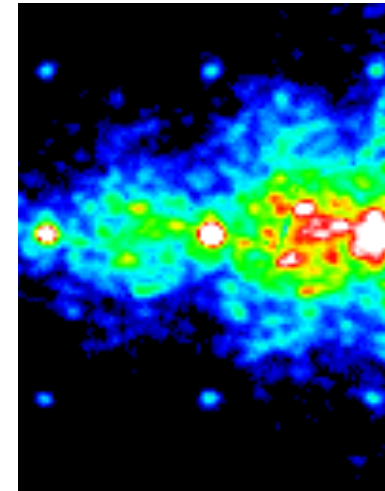
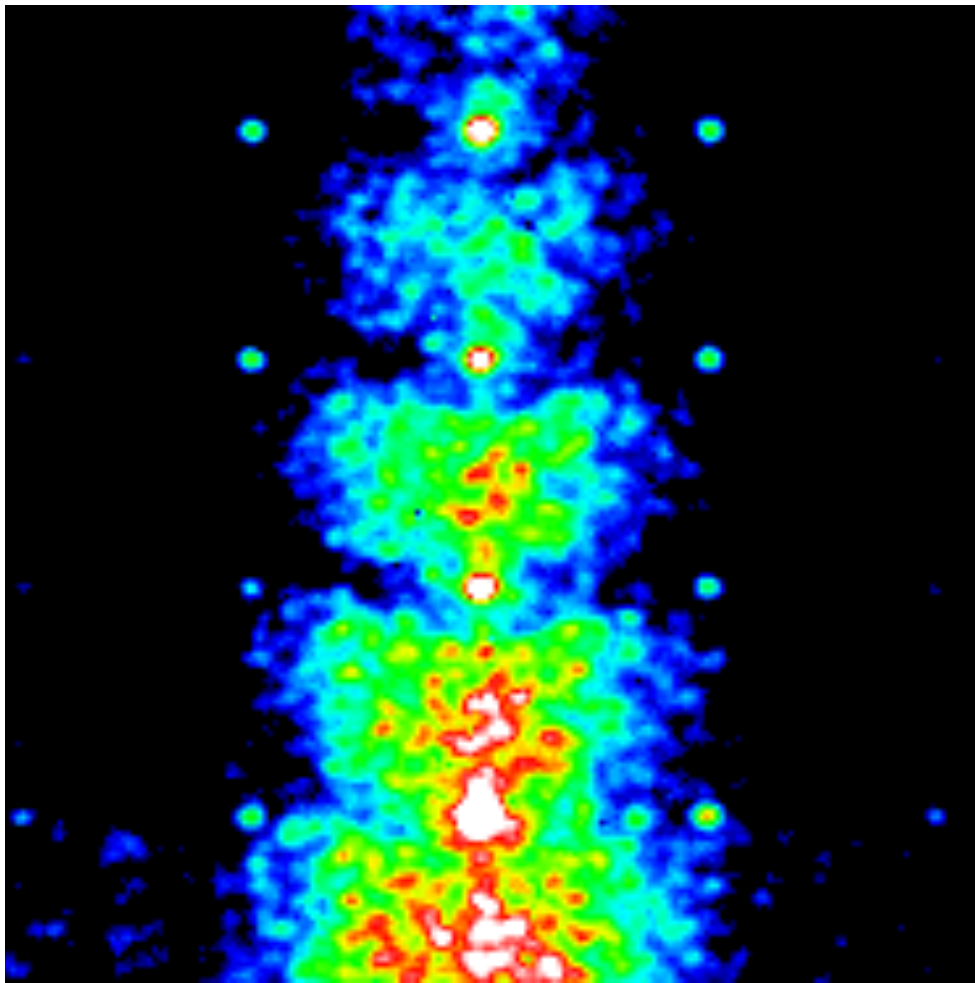
1 μm



Pinhole Sample Analyser

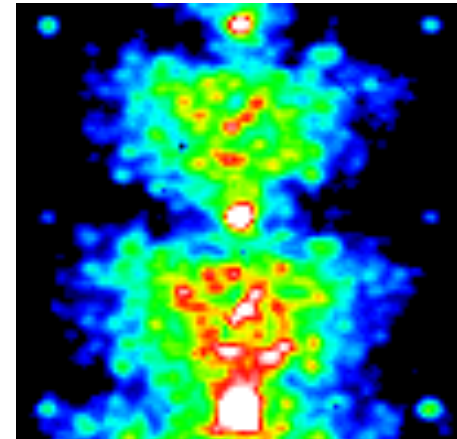
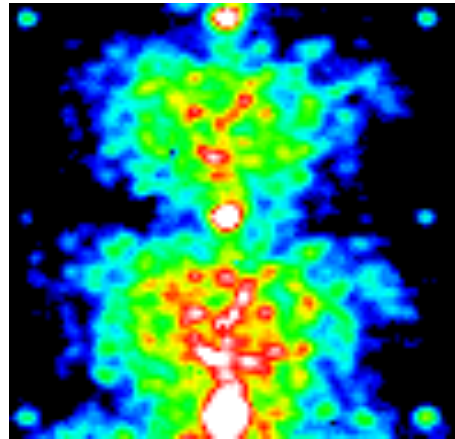
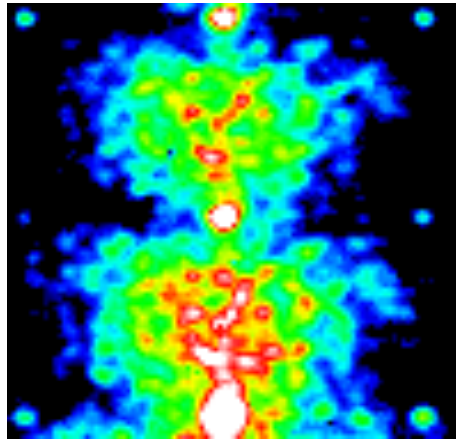
Phase plate scan near forward direction

1 μm step per frame



Collagen, buffer and empty cell

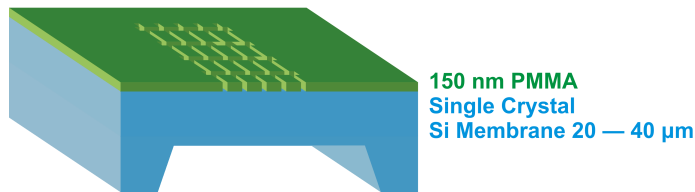
phplateFZP-236, -246 and -249



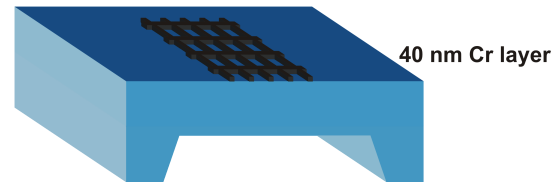
Phase plate fabrication

Joan Vila-Comamala, PSI

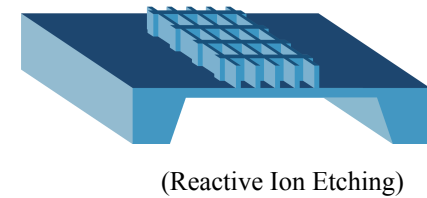
1) e-beam lithography



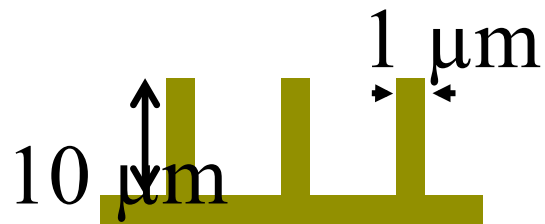
2) Pattern transfer to a Cr layer by evaporation and lift-off



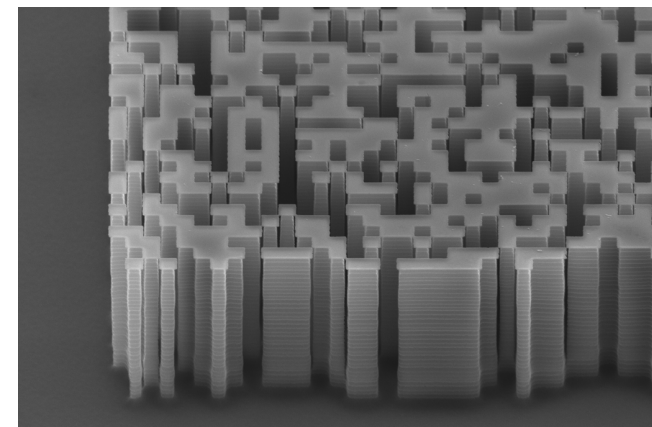
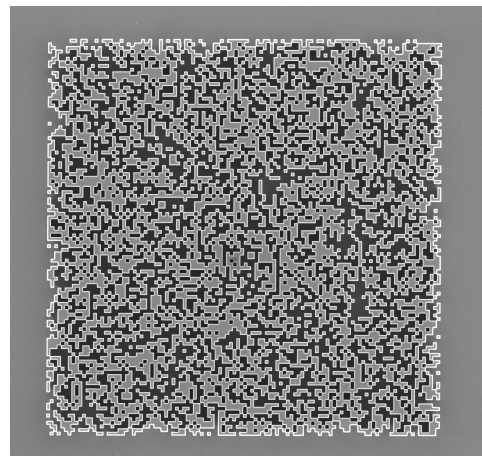
3) Pattern transfer to Si by RIE



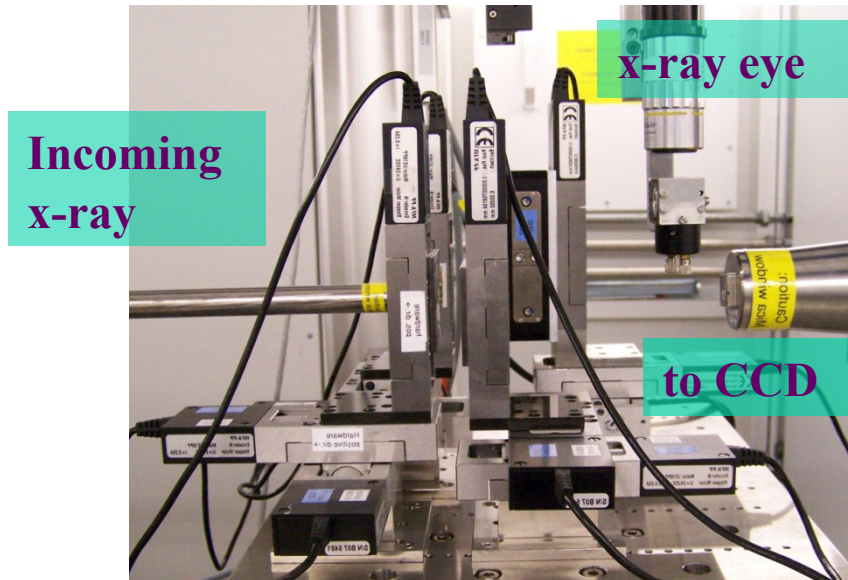
At 8.0 keV ($\lambda=0.155$ nm) $\rightarrow \delta = 7.67 \cdot 10^{-6}$ **$h=10.10$ μm**
At 10.0 keV ($\lambda=0.124$ nm) $\rightarrow \delta = 4.88 \cdot 10^{-6}$ **$h=12.70$ μm**



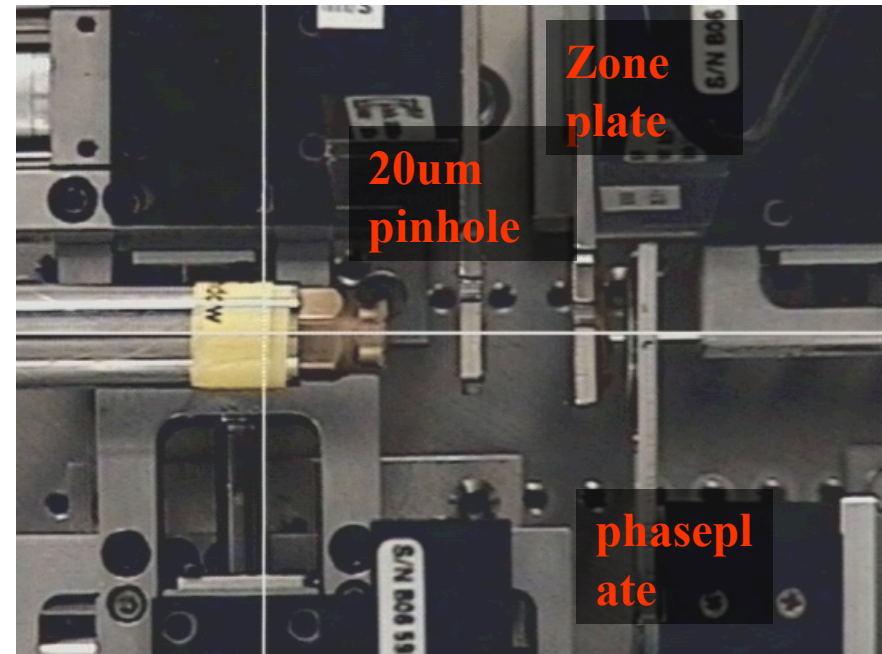
Cross section



Experimental setup cSAXS (SLS)

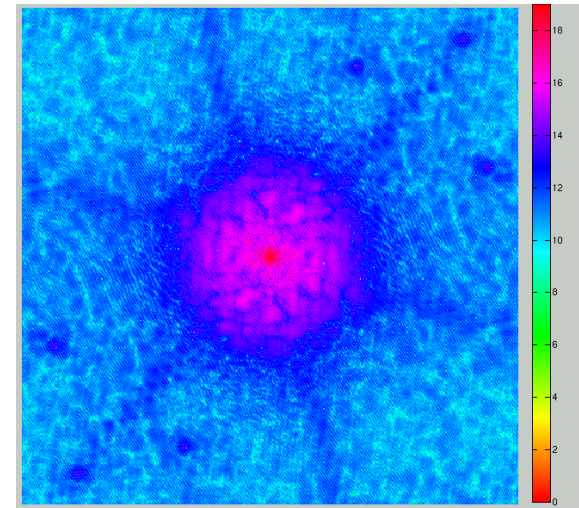
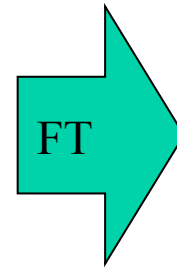
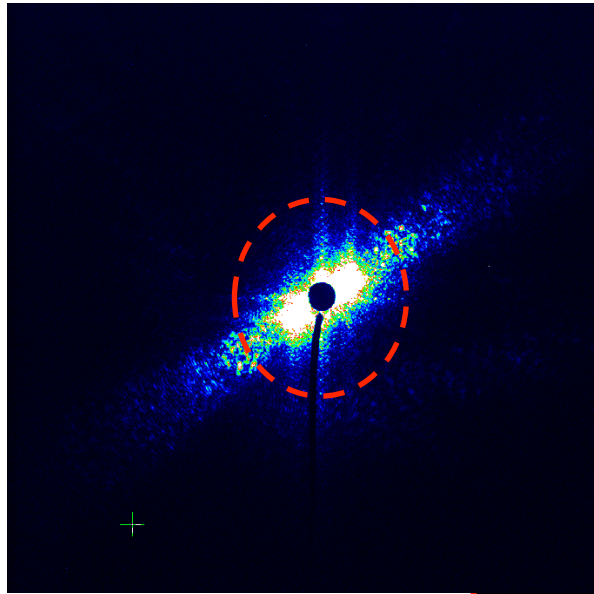


Side view

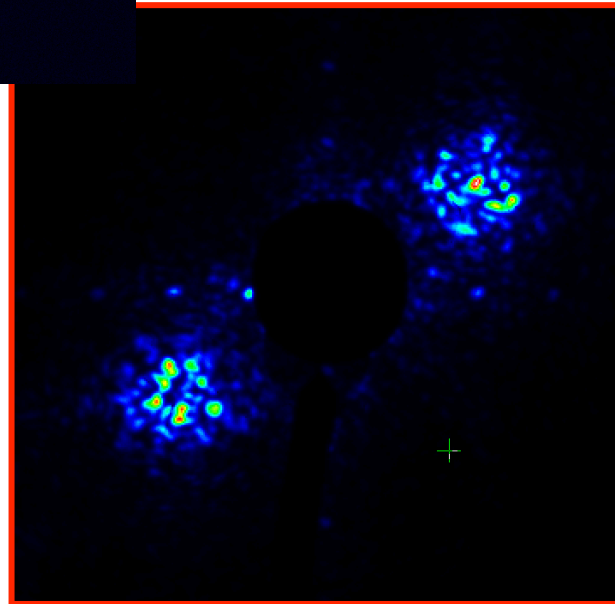


Top view (zoomed in)

Results: Zone Plate as object

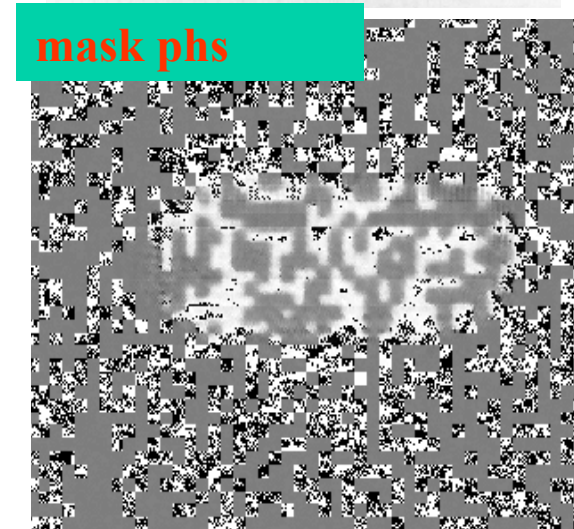
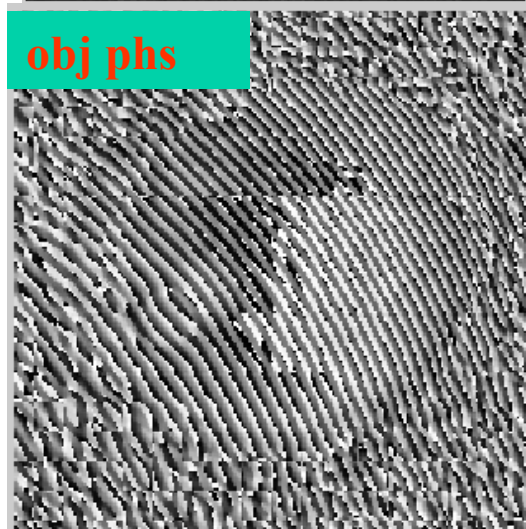
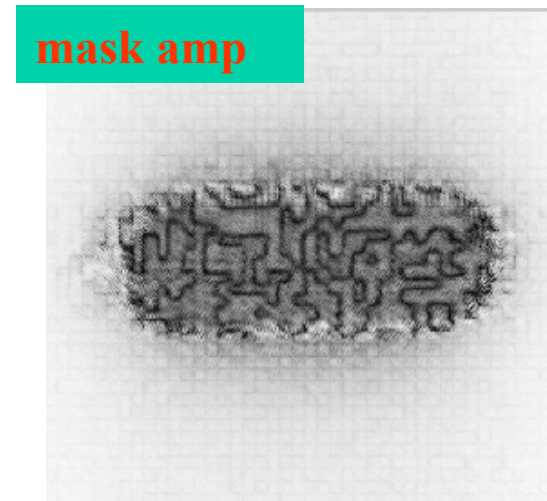
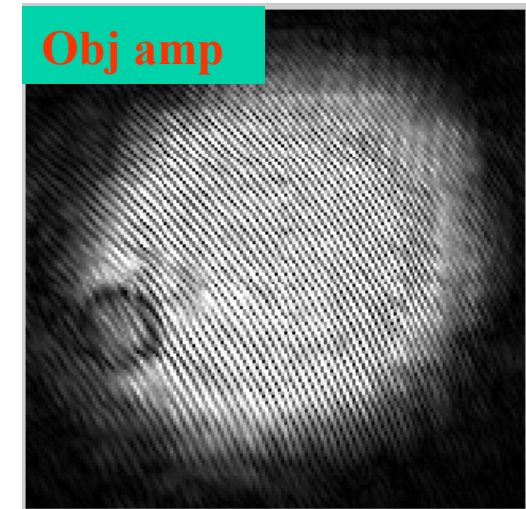


Auto correlation function



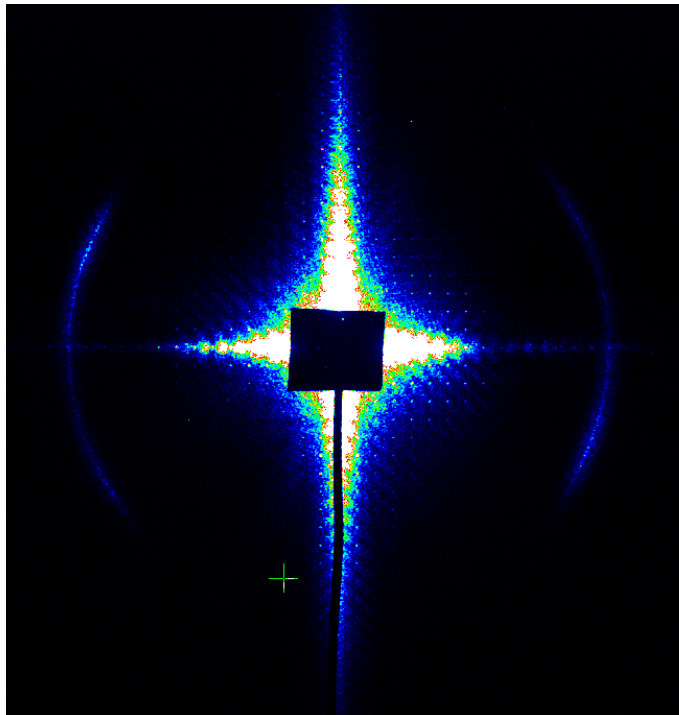
Reconstruction of Phase plate scan

1 μm step per frame (512x512)

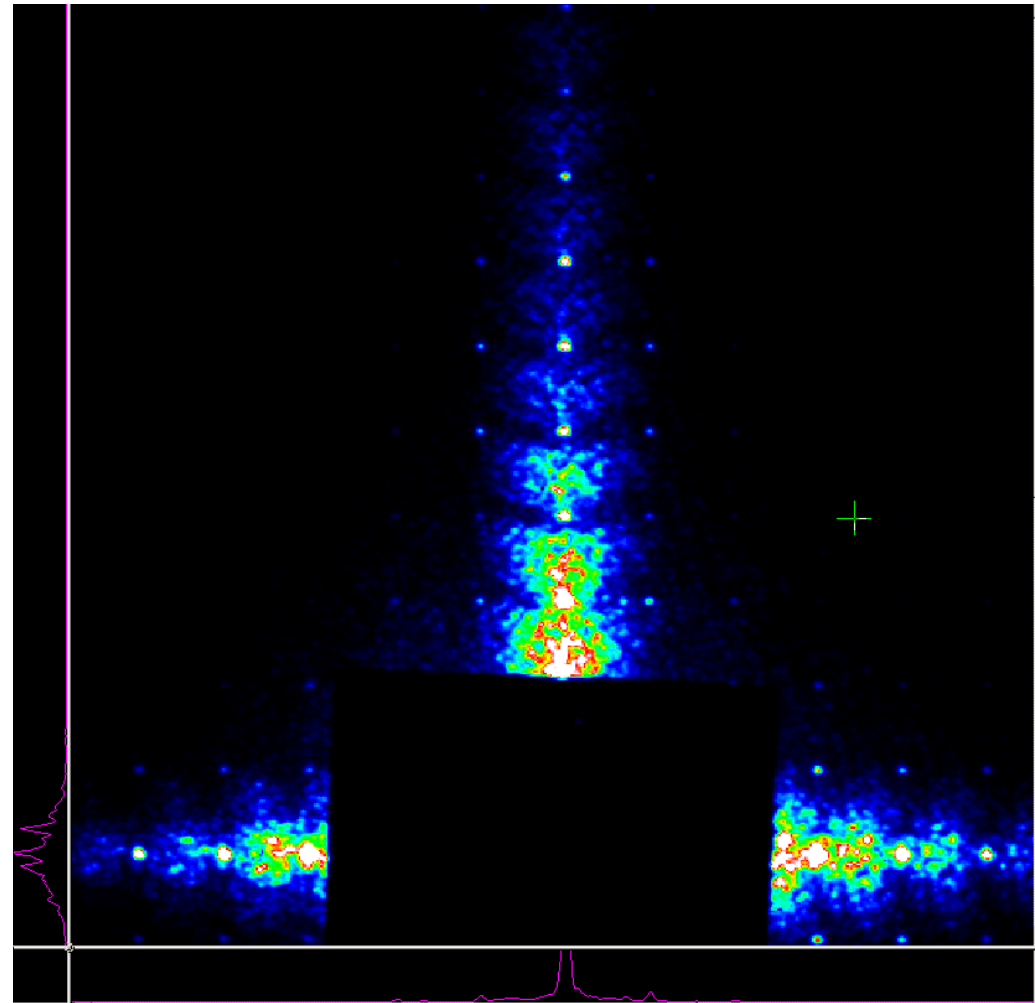


Reconstruction by F. Zhang (paper in preparation)

Collagen rat tail tendon as sample

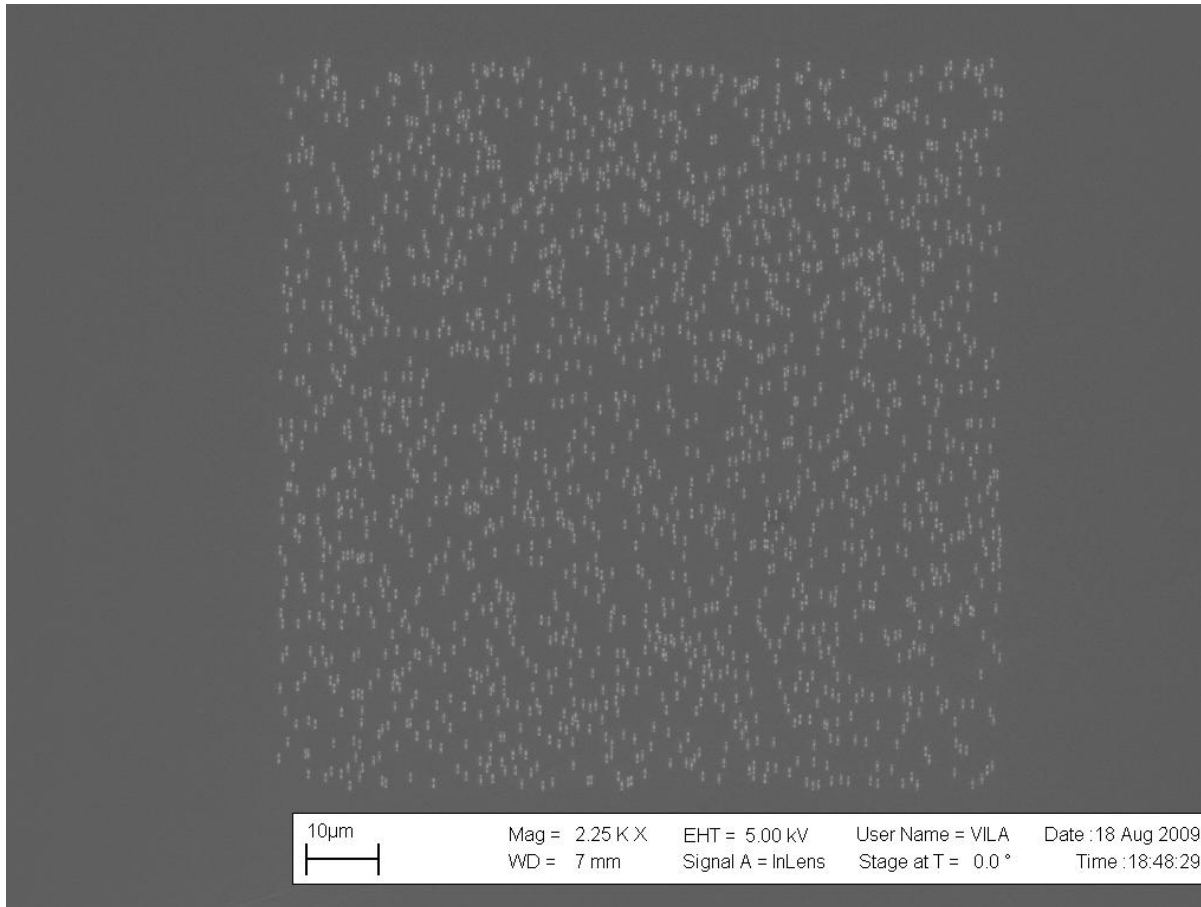


- * 0th order “repeated” in the meridian and equator
- * Strong phase modification due to phase plate!
- * No successful reconstruction



New phase plate design

R. Bean and J. Vila-Comamala, PSI



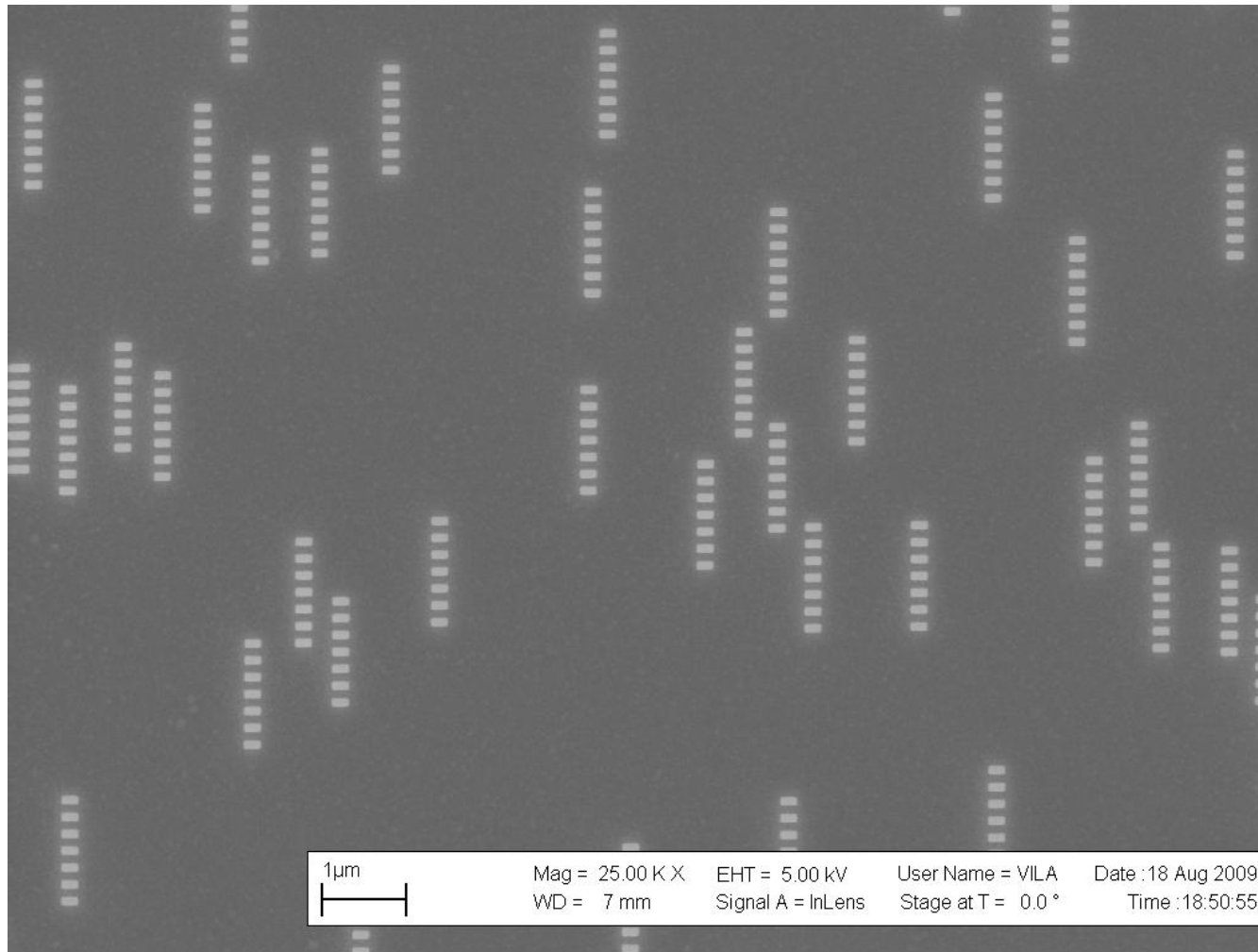
Pixel size = 10 nm
Total size = 100 x 100
microns²

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

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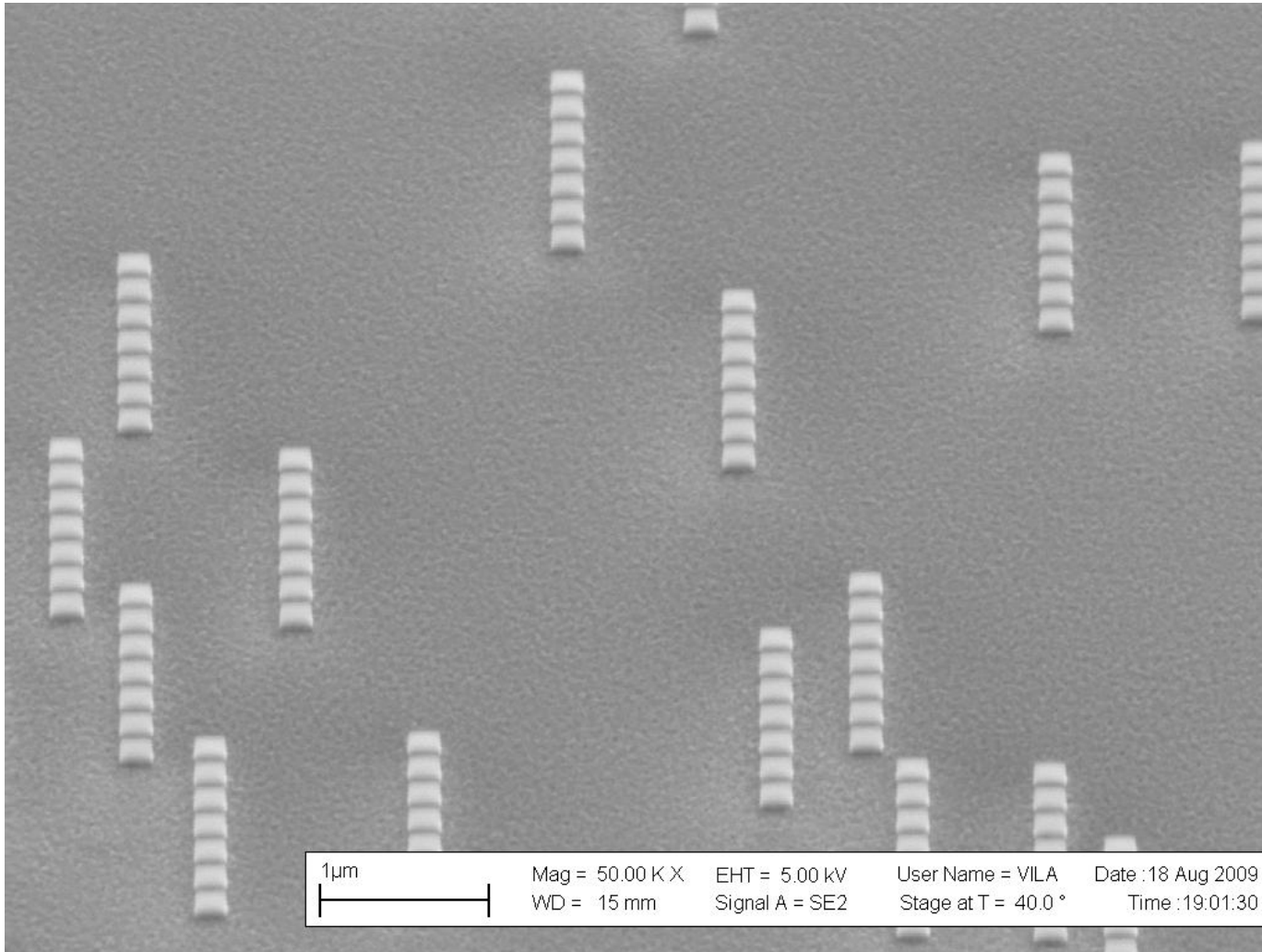
New phase plate design

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
- Ptychography reveals probe structure also
- Phase plate demonstrated
- New custom phase plates
- Preparations under way for XFEL