

Imaging of Domain Structures by Coherent X-ray Diffraction

Ian Robinson London Centre for Nanotechnology

Felisa Berenguer Diamond Light Source

Richard Bean Swiss Light Source

Loren Beitra

Fucaï Zhang (Sheffield)

Joan Vila (PSI)

Frontiers in Optics

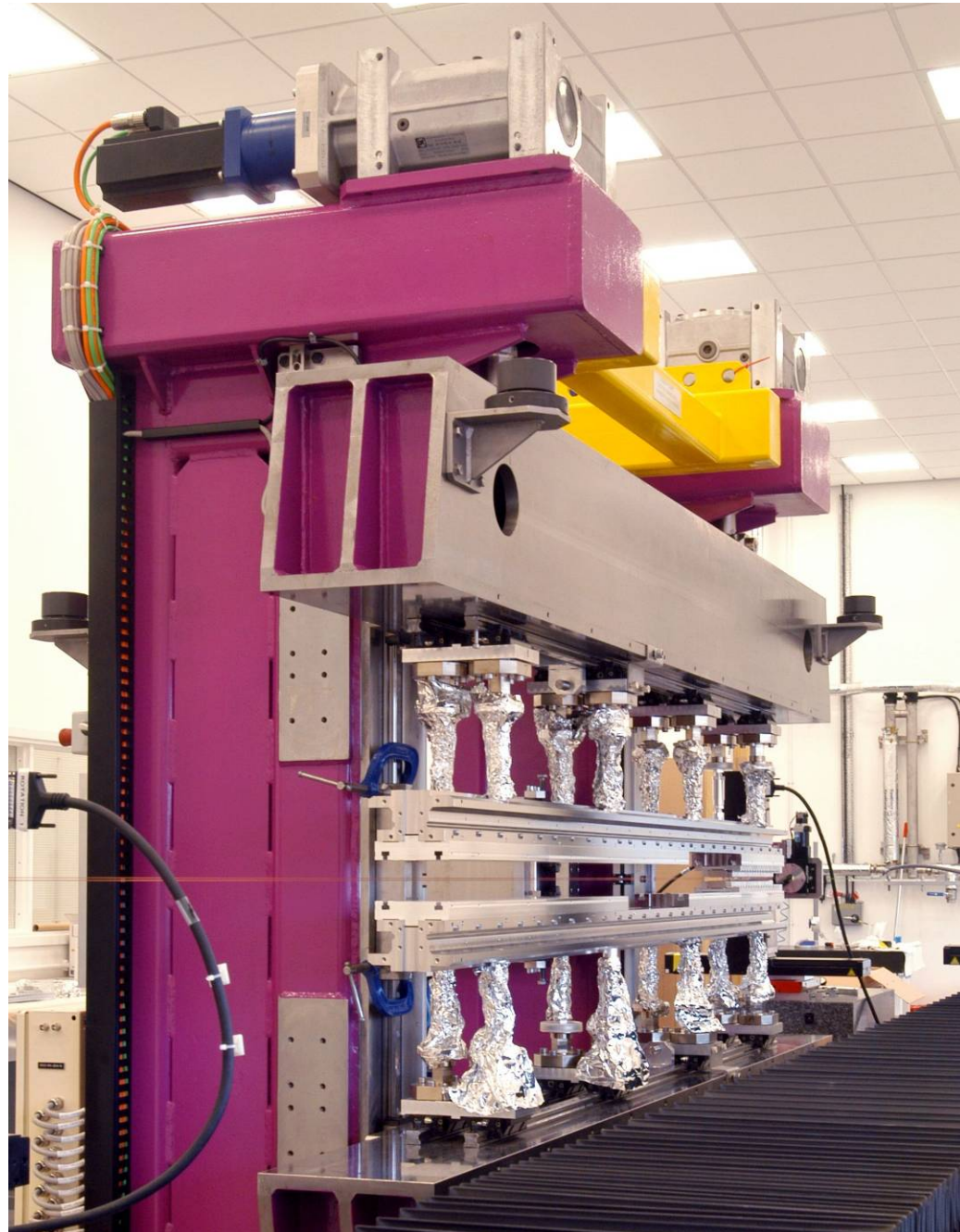
San Jose, CA

October 2009

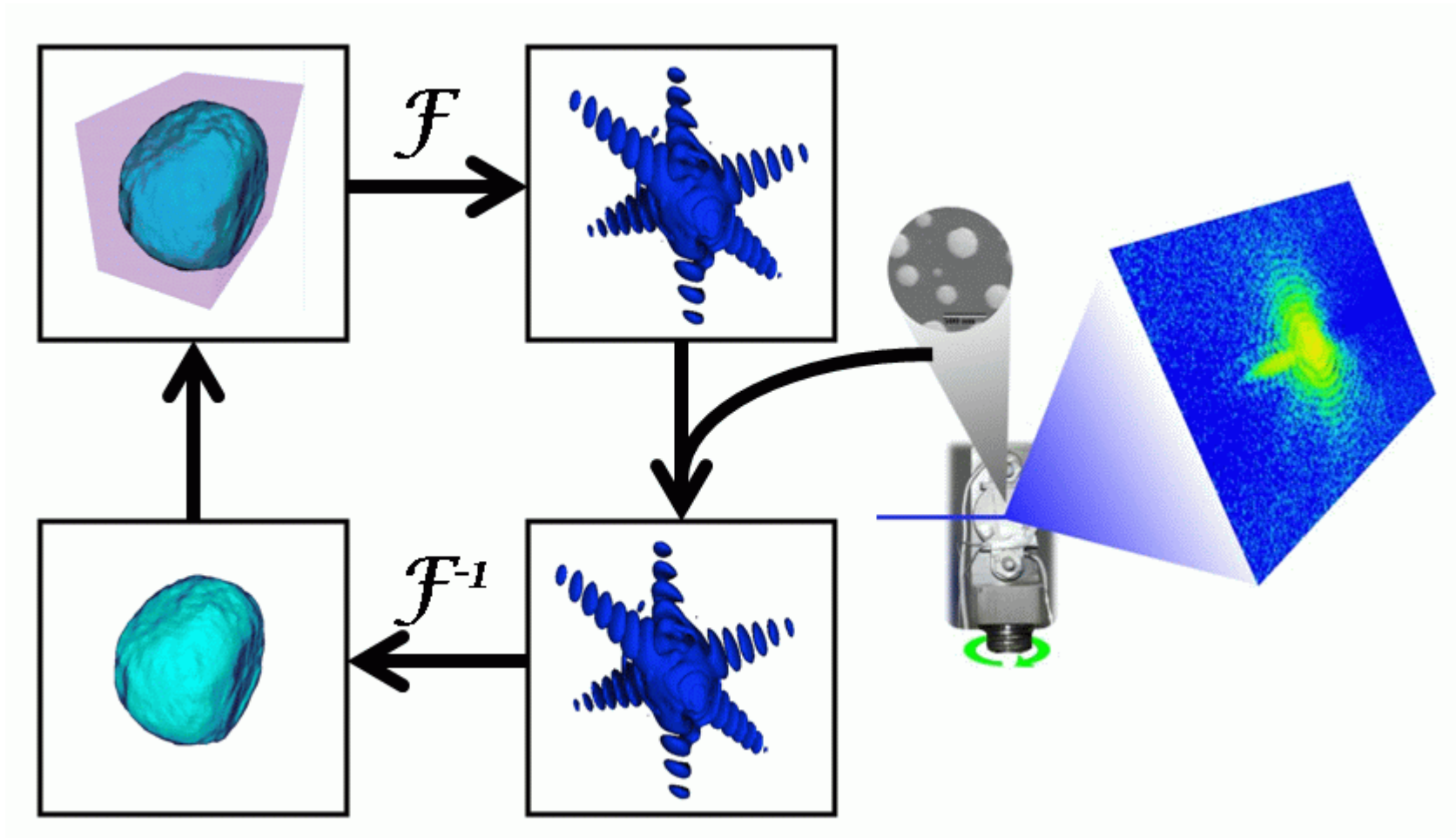
Outline

- Coherent X-ray Diffraction
- Domain speckle patterns
- Ptychography
- Phase-plate approach
- Phase domains in collagen
- Preparations for LCLS

Diamond
in-vacuum
X-ray
Undulator



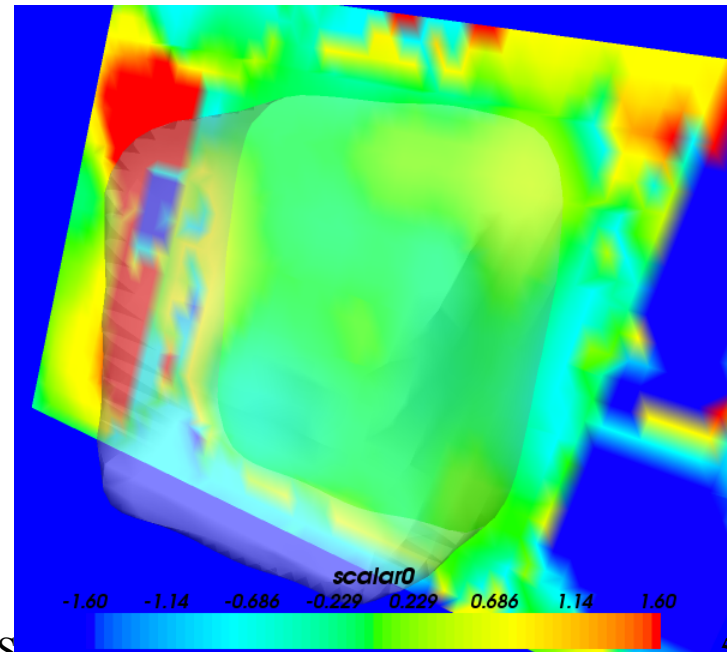
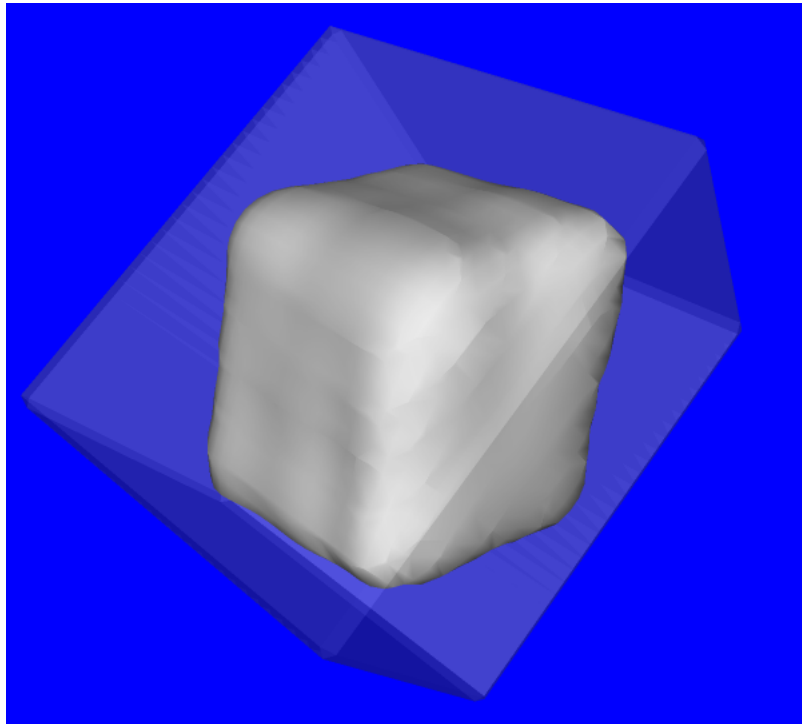
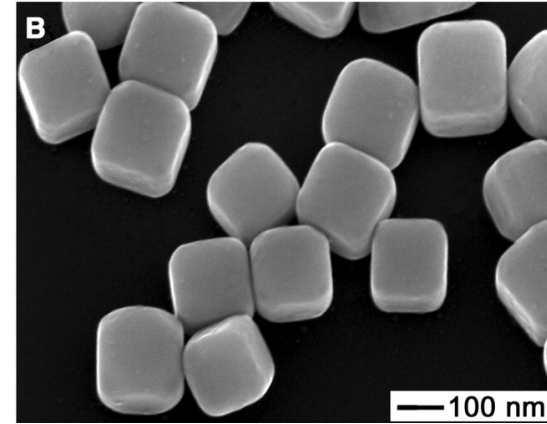
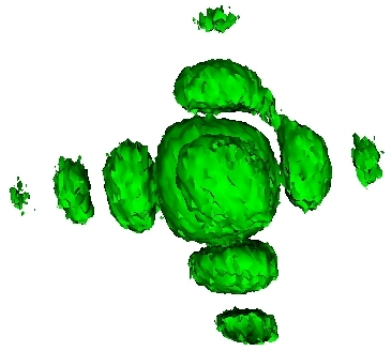
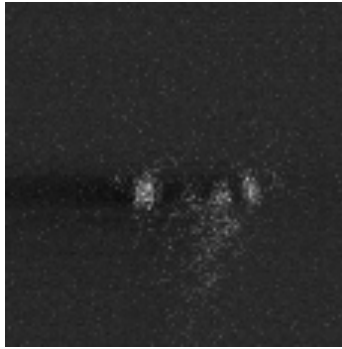
Generic “Error Reduction” method



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

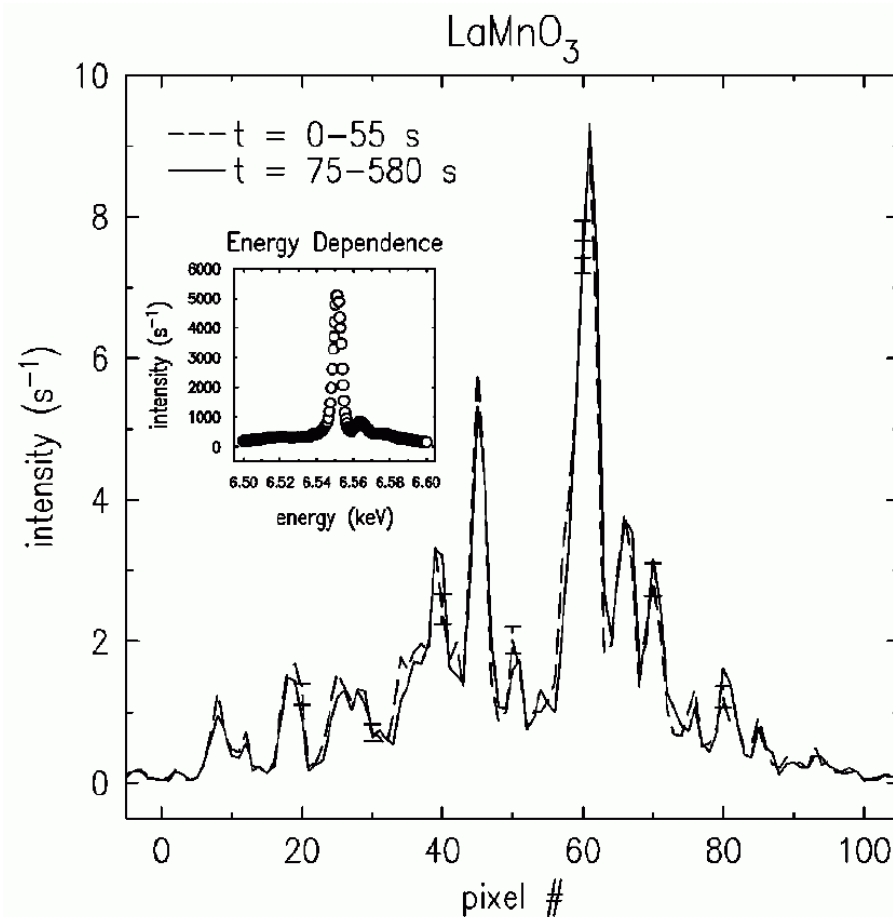
R. W. Gerchberg and W. O. Saxton *Optik* 35 237 (1972)

I. K. Robinson, OSA 2009

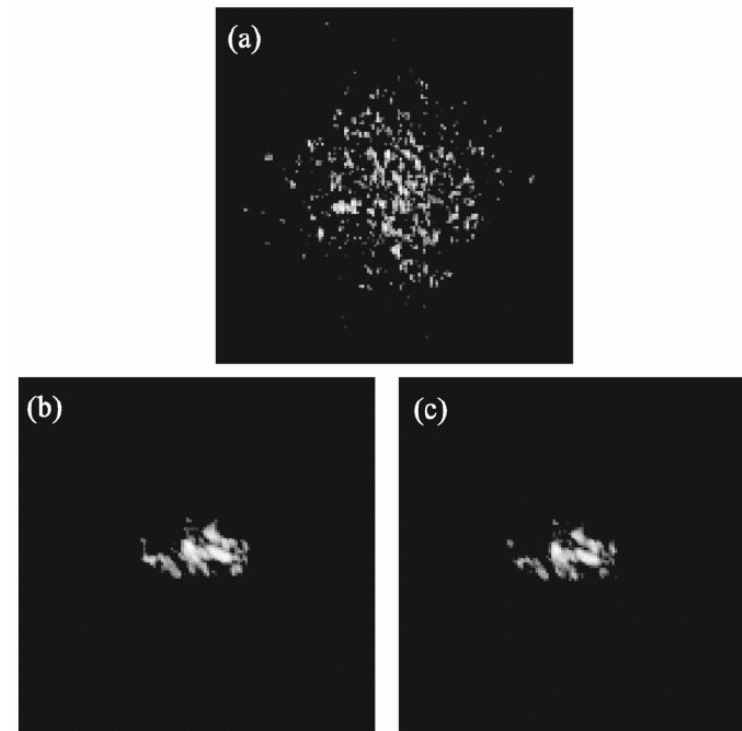


LaMnO₃ and Pr_{0.6}Ca_{0.4}MnO₃ speckle

C. S. Nelson, J. P. Hill, Doon Gibbs, F. Yakhou, F. Livet, Y. Tomioka, T. Kimura and Y. Tokura, PRB 66 134412 (2002)



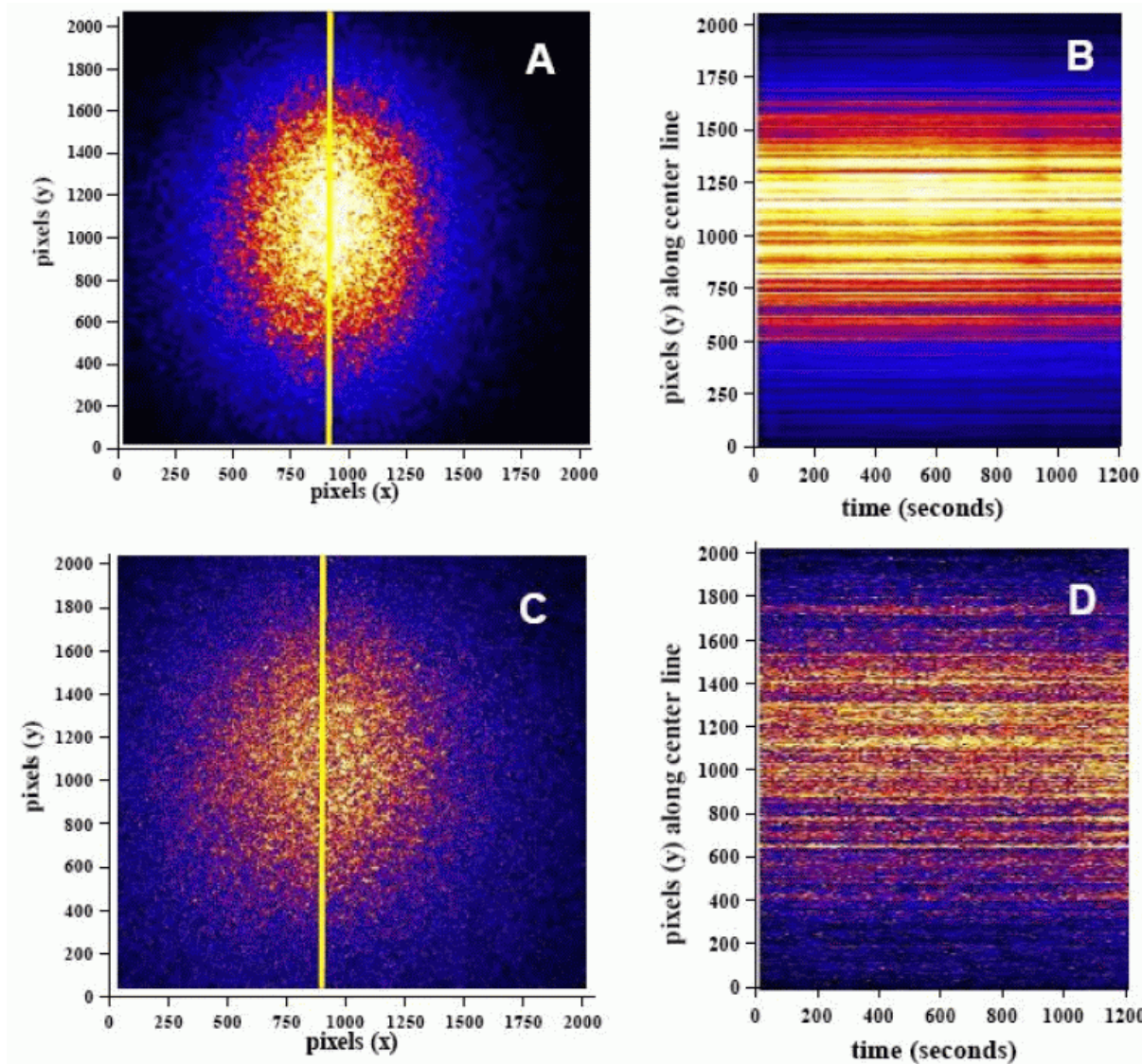
Pr_{0.6}Ca_{0.4}MnO₃, T = 150 K



. Images of orbital (a) and charge (b), (c) order

$\text{Pr}_{0.5}\text{Ca}_{0.5}\text{MnO}_3$ at MnL_{III} edge (650 eV)

J. J. Turner et al, New Journal of Physics 10 053023 (2008)

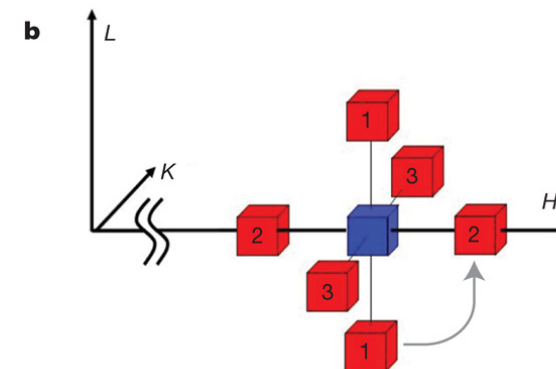
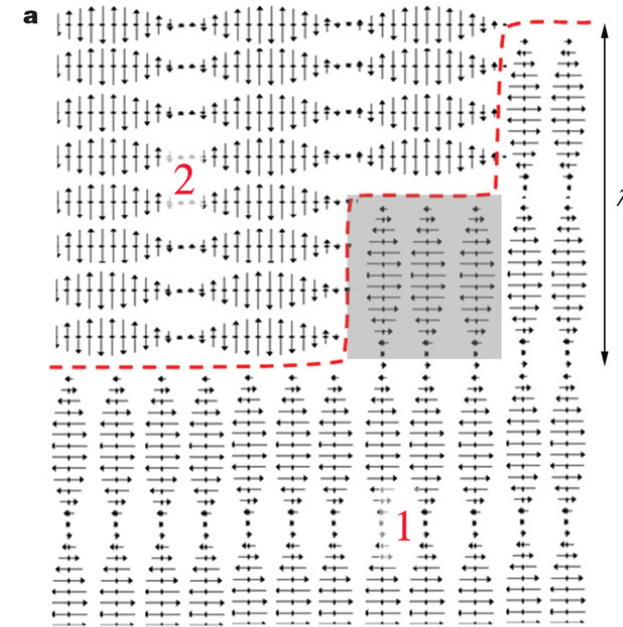
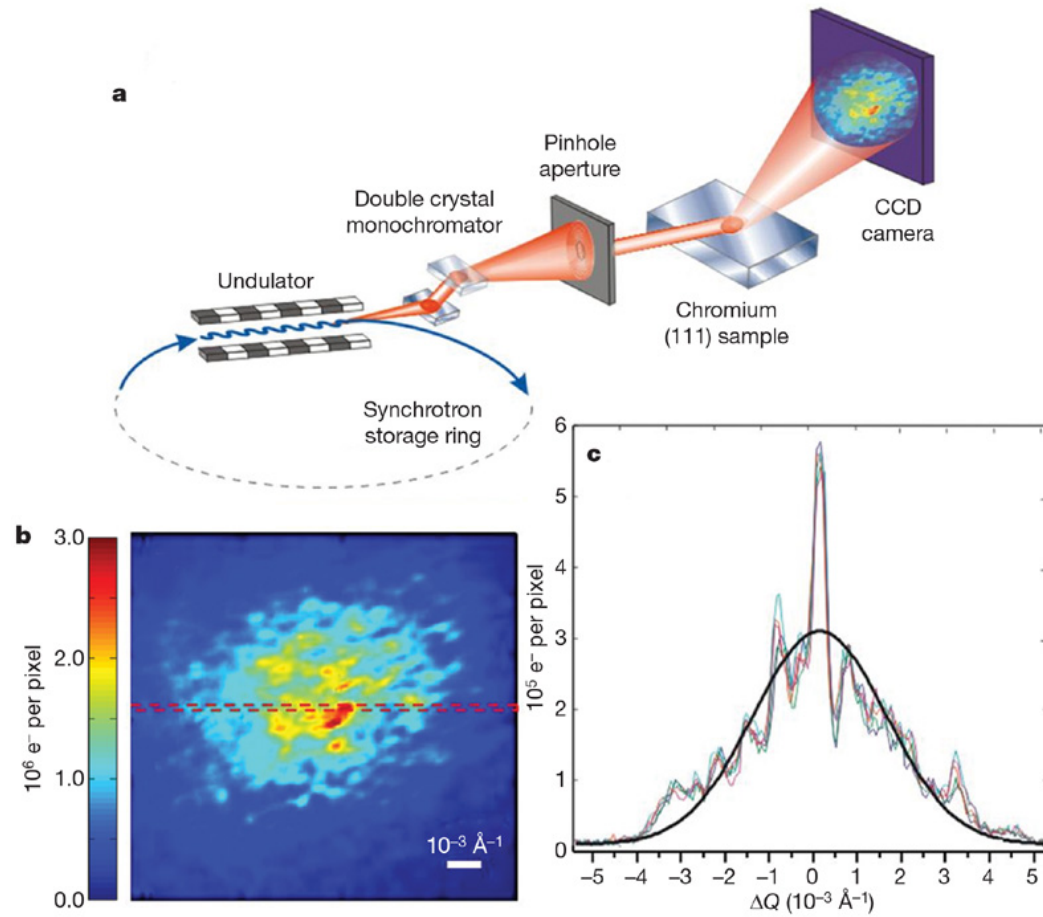


$(0, \frac{1}{2}, 0)$
Orbital ordered
T=205K

$(0, \frac{1}{2}, 0)$
Near phase
transition
T=232K

Antiferromagnetic Domains in Cr

O. Shpyrko et al, Nature 447 68 (2007)



I. K. Robinson, OSA 2009

FeAl antiphase domains (001)

Lorenz Stadler, PhD dissertation, TU Wien (2005)

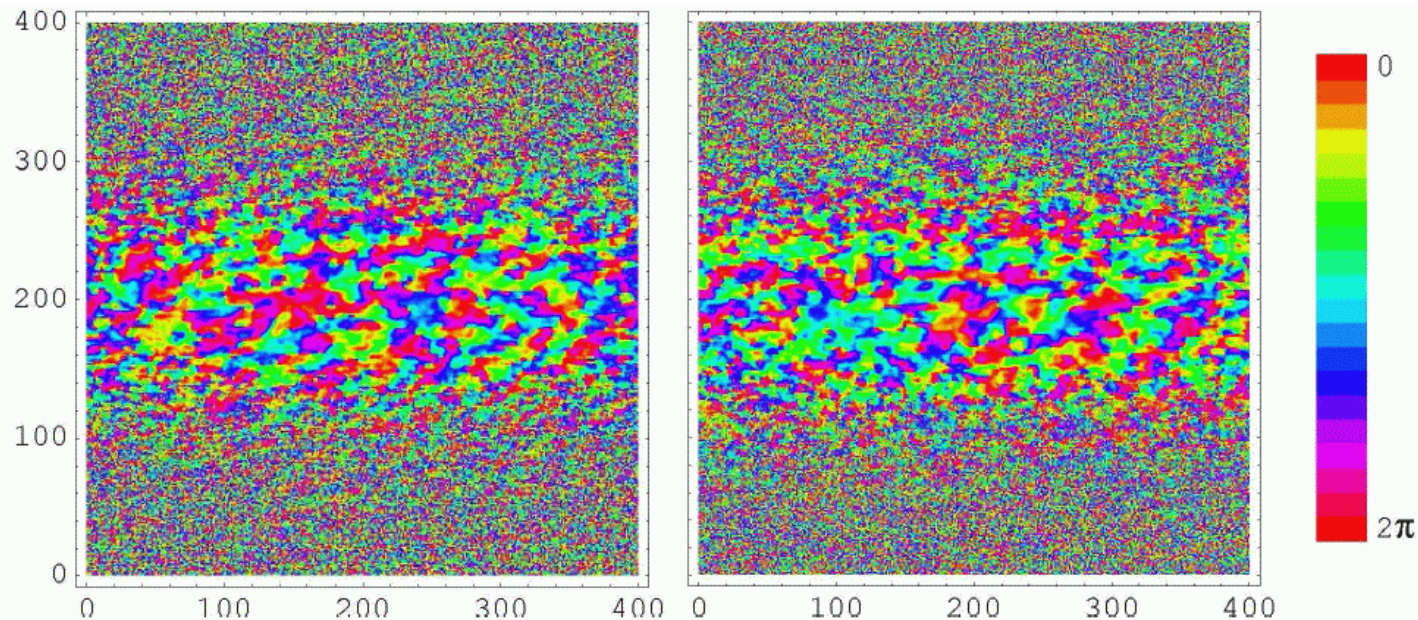
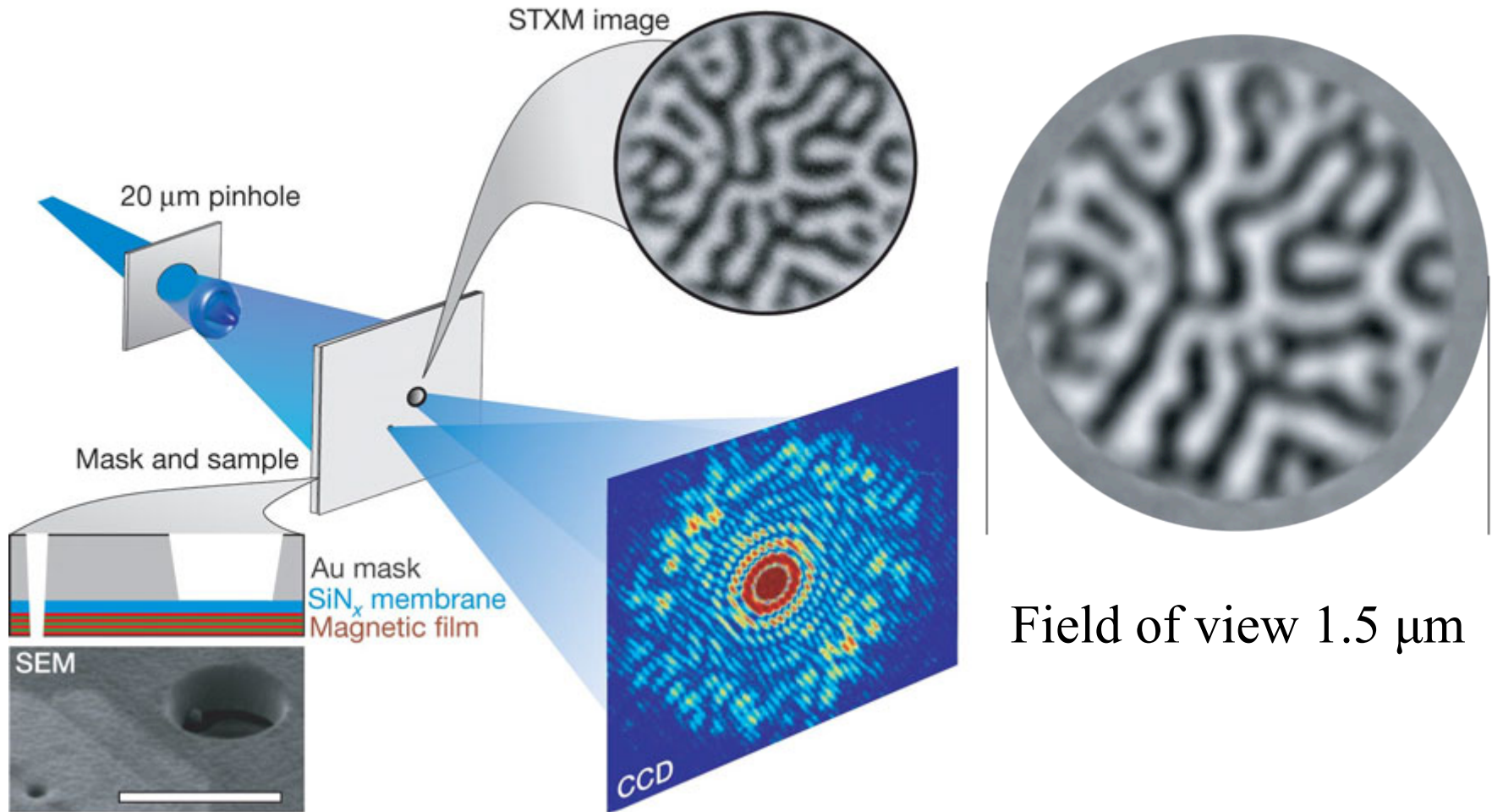


Figure 7.11: Typical reconstructed phases from runs with different combinations of algorithms and supports derived from the 2D Gaussian fit of the illumination function. Numbers in brackets denote how many iterations of the particular algorithm were done each cycle. Graphs on the left are from reconstruc-

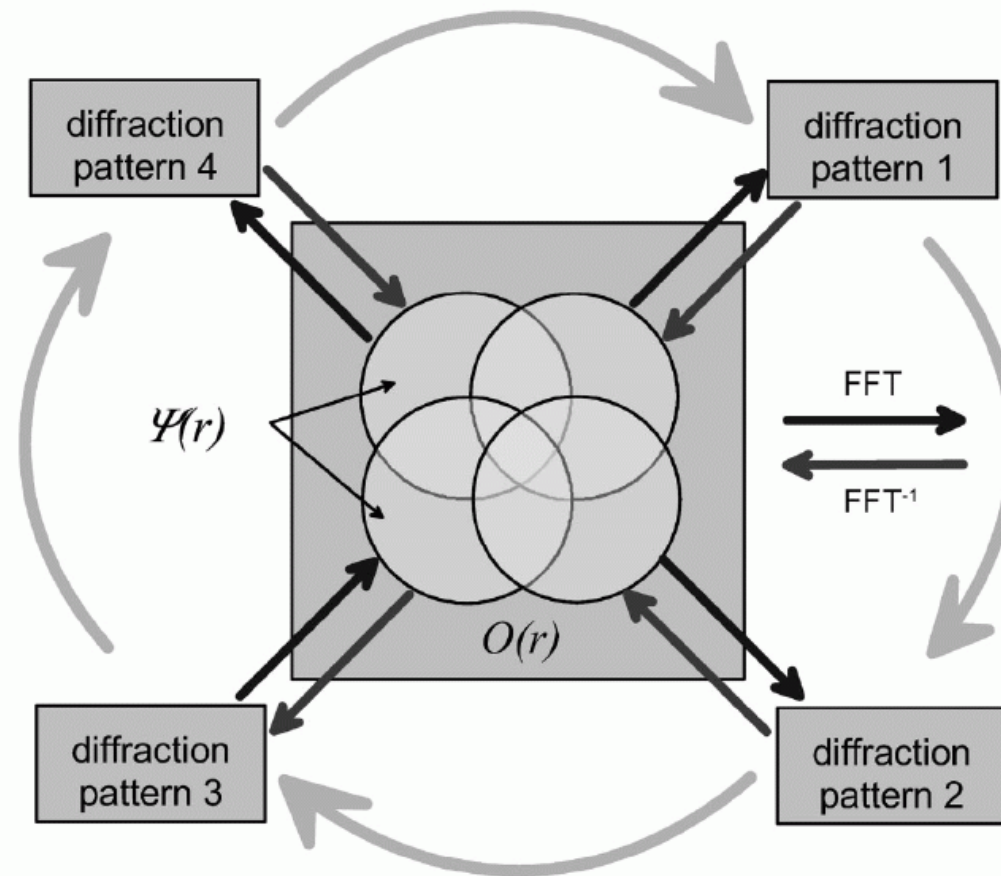
X-ray Holography of Pt/CoML Domains

S. Eisebitt et al. Nature 432 885 (2004)



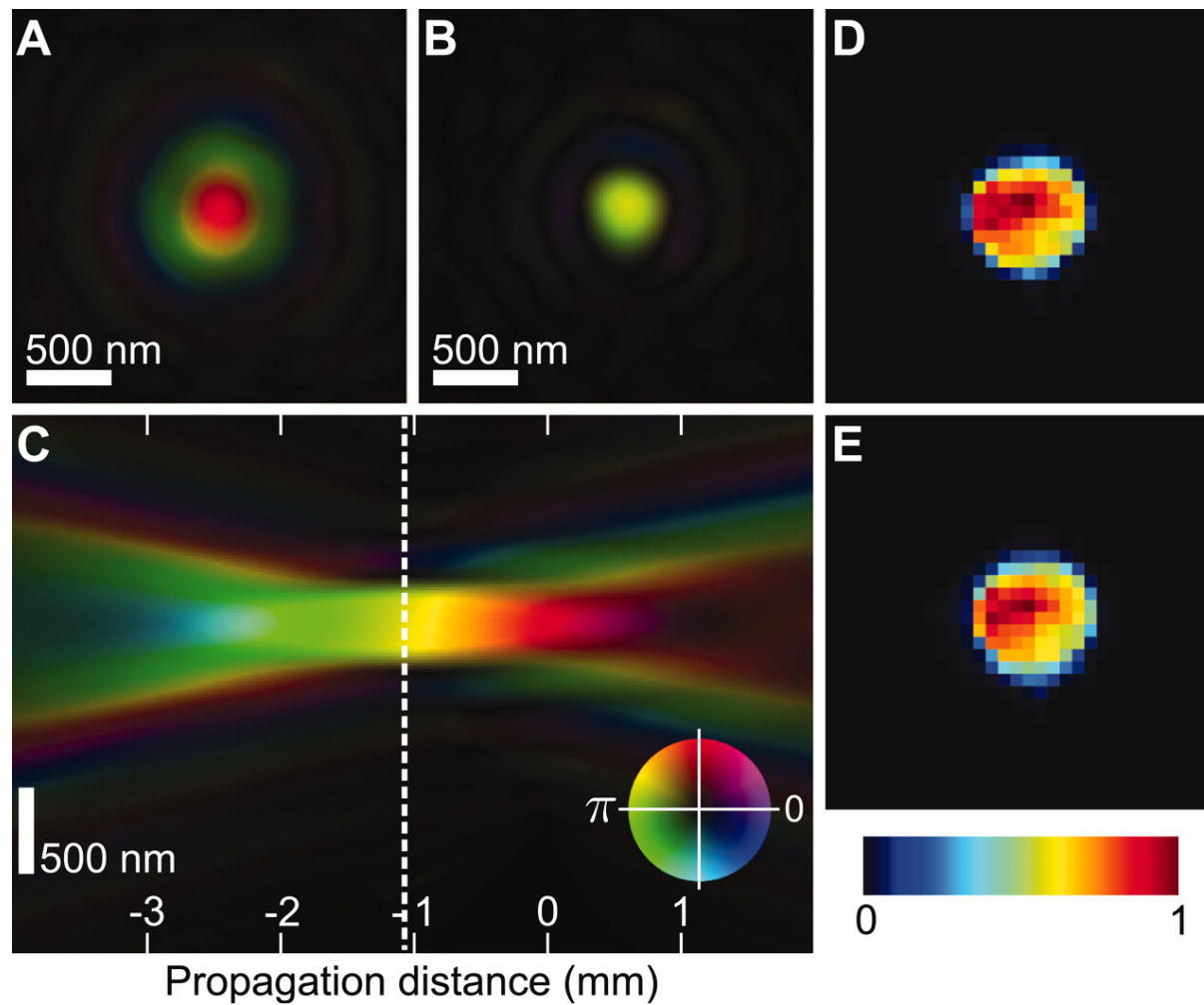
X-ray Ptychography

J. Rodenburg et al, PRL 98, 034801 (2007)



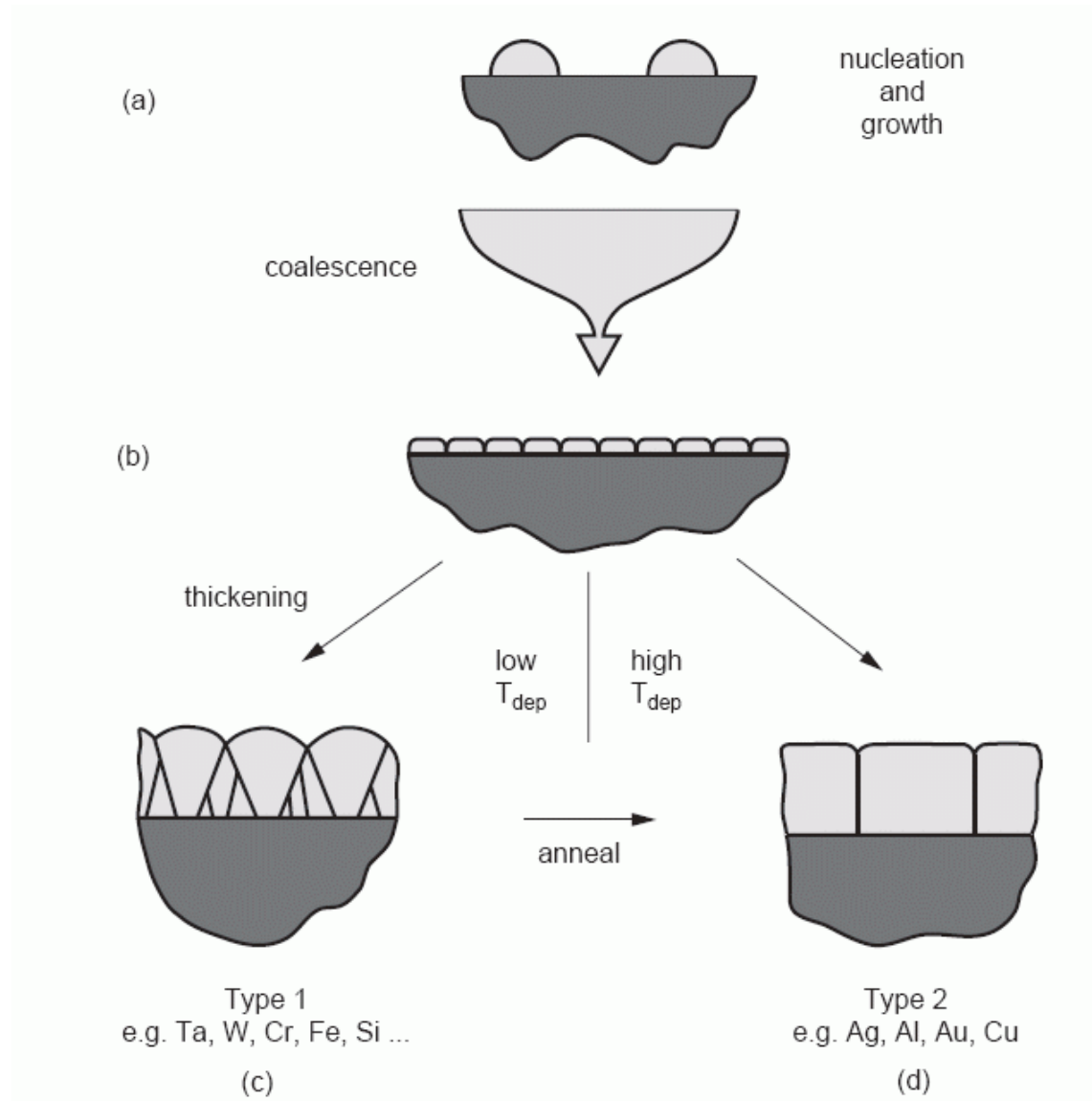
Reconstruction of Probe

P. Thibault et al, Science 321 379 (2008)

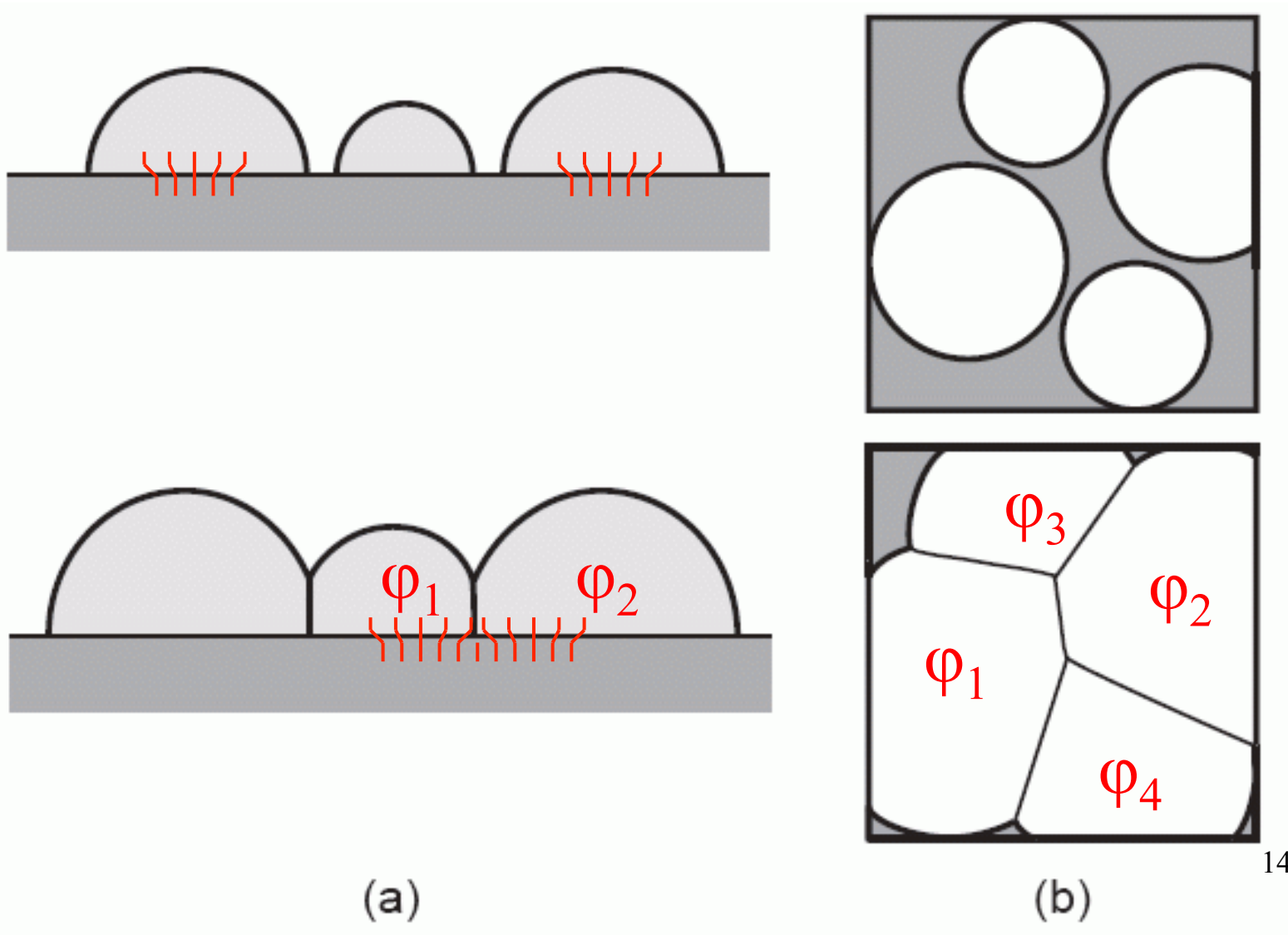


Thin film growth after deposition

C. V. Thompson, Annu. Rev. Mater. Sci. 2000. 30:159–90



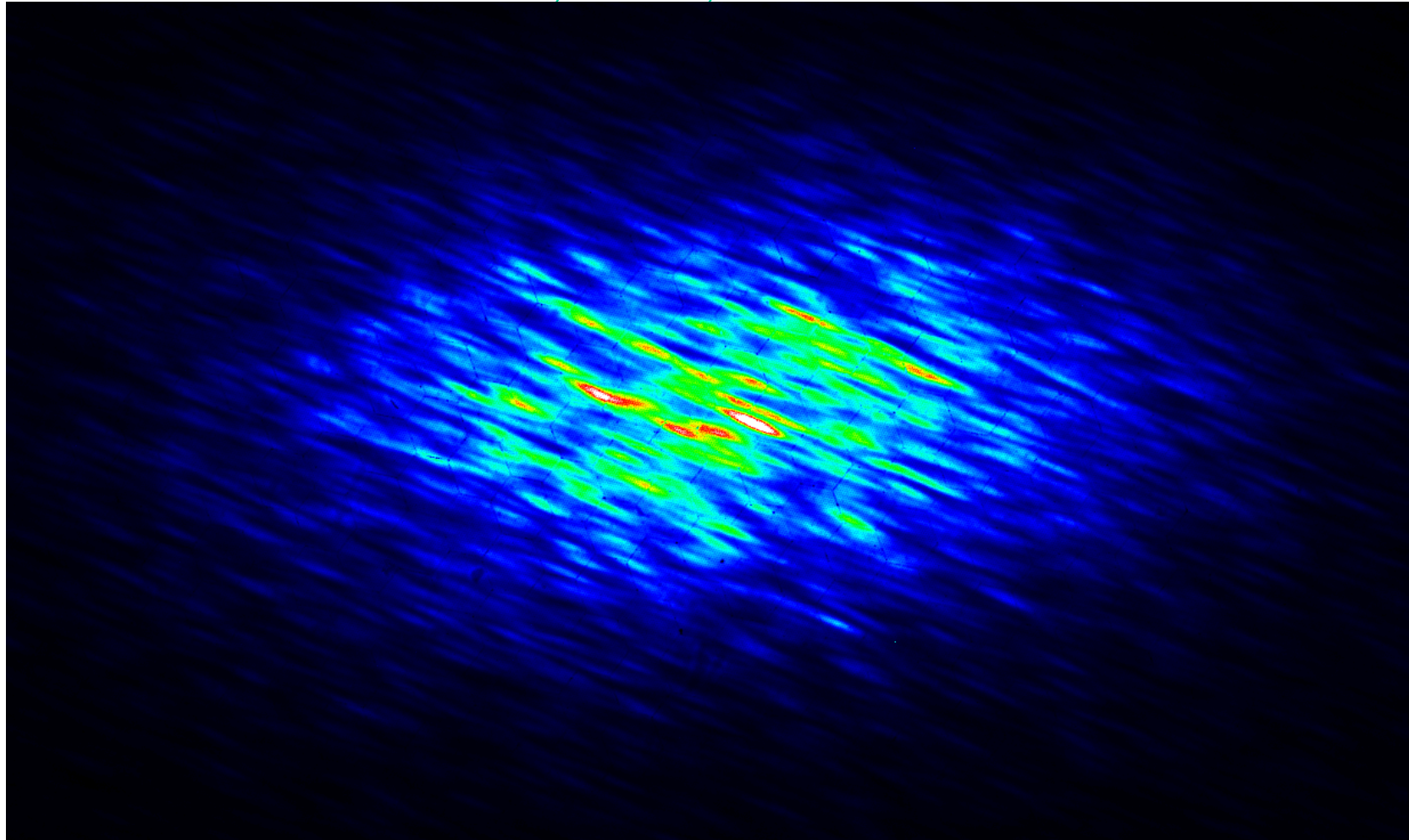
Epitaxial growth effects



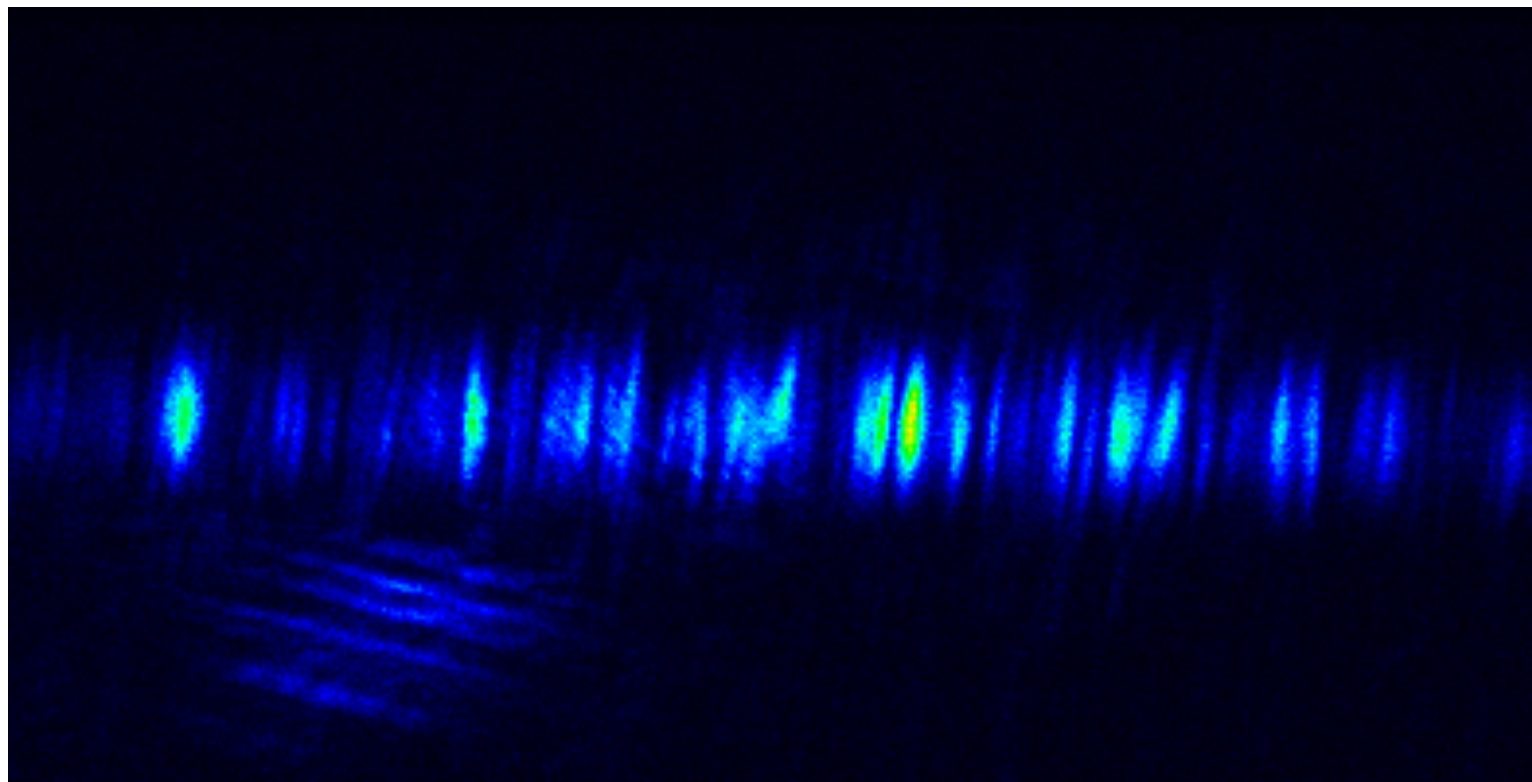
Niobium (110) Thin Film Grains

1 μm steps across 3 μm beam of KB mirror focus

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

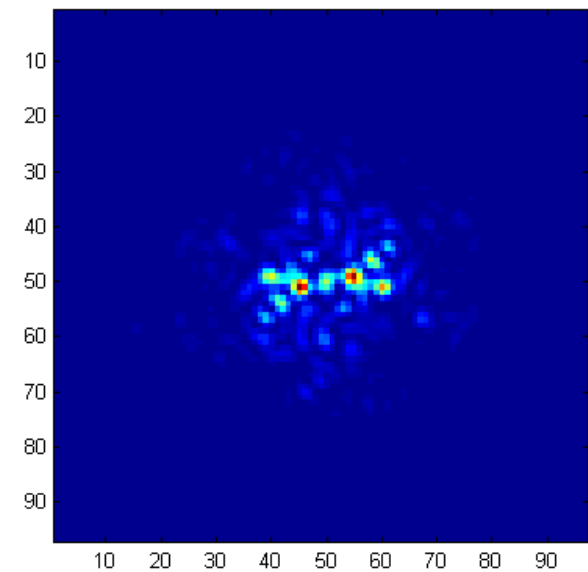
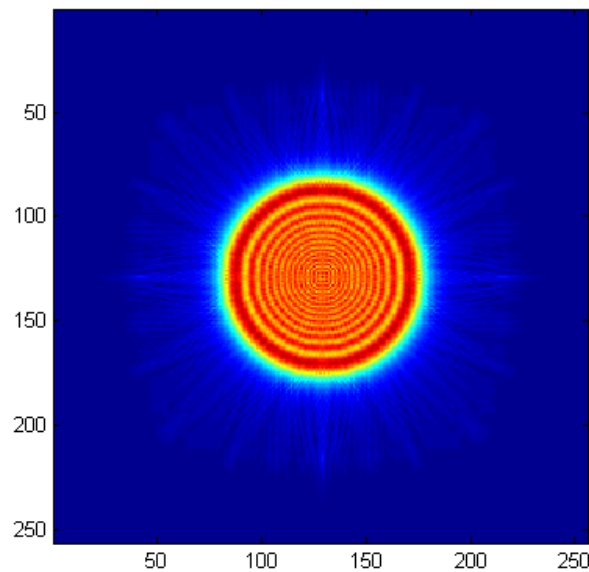
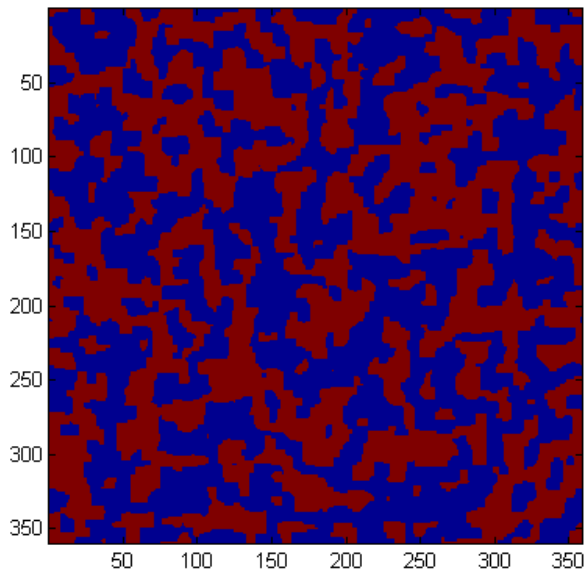


“Barcode” diffraction of GaAs nanowire



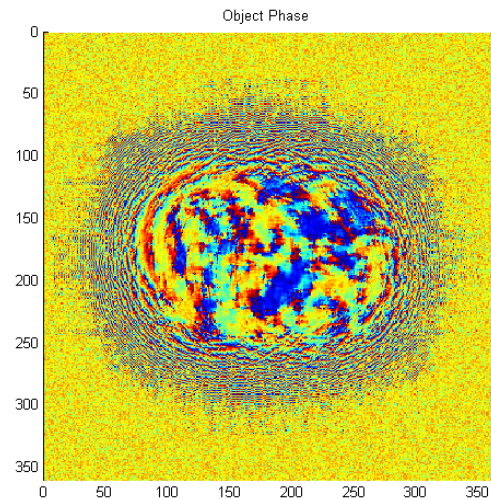
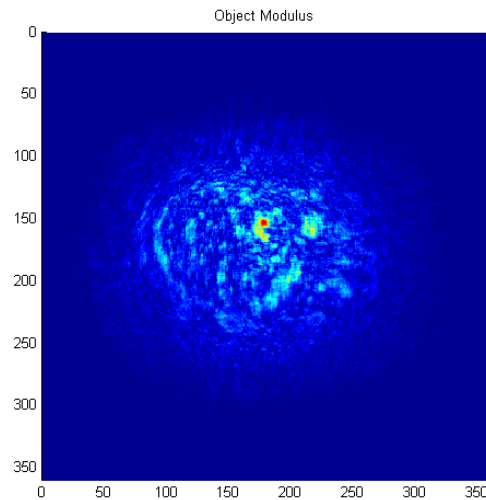
Ptychography phasing tests

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

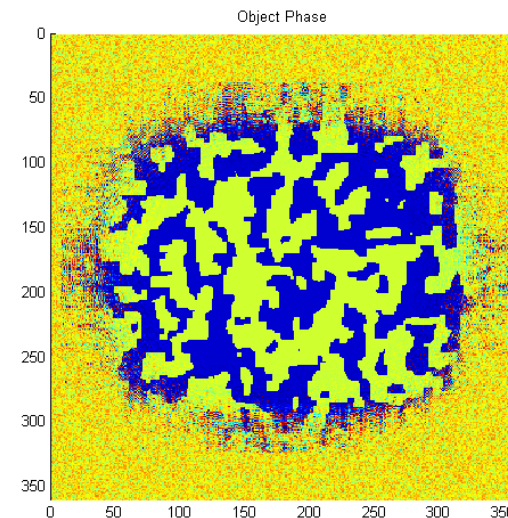
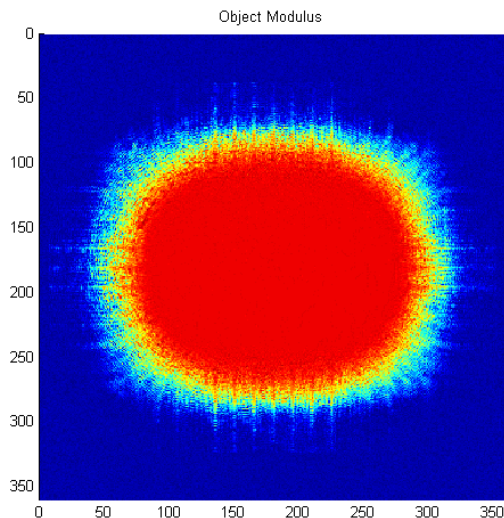


Ptychography phasing tests

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.

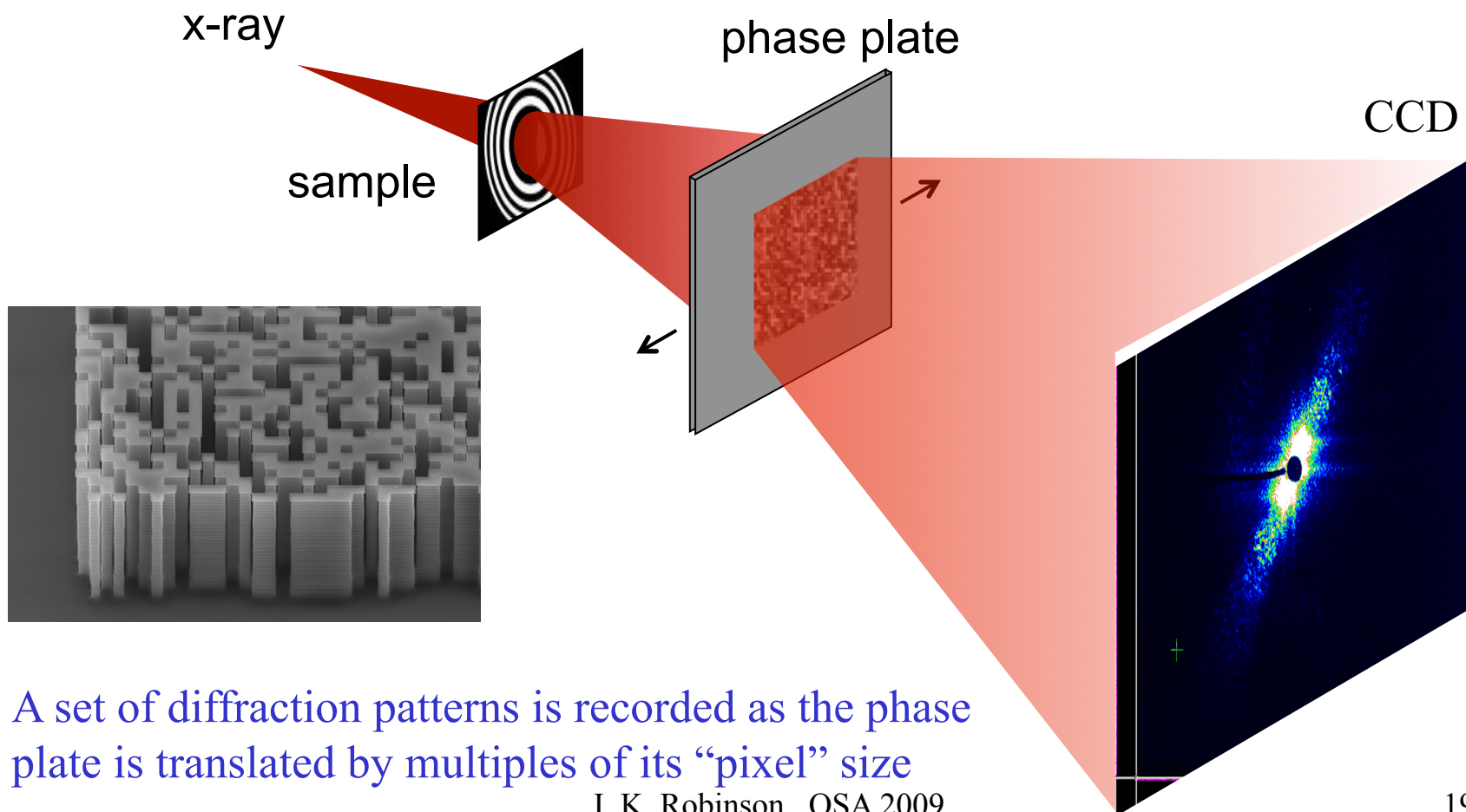


Amplitude and phase after 20 iterations.

Imaging by Wavefront Modification

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

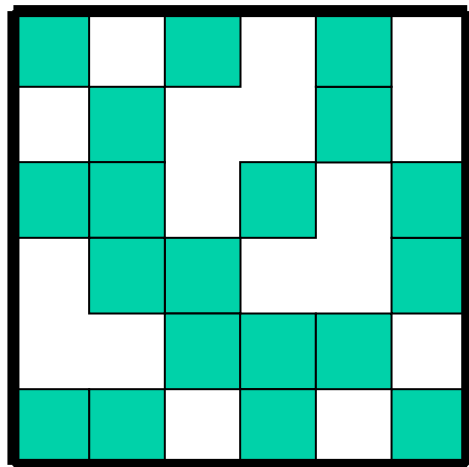
I. Johnson et al., Phys Rev Lett 100 (2008)



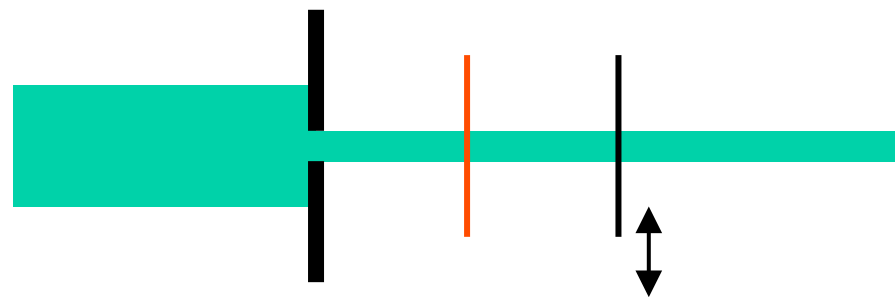
I. K. Robinson, OSA 2009

'Random' phase plate analyser

Joan Vila (PSI), Fucai Zhang (Sheffield)



$1\ \mu\text{m}$

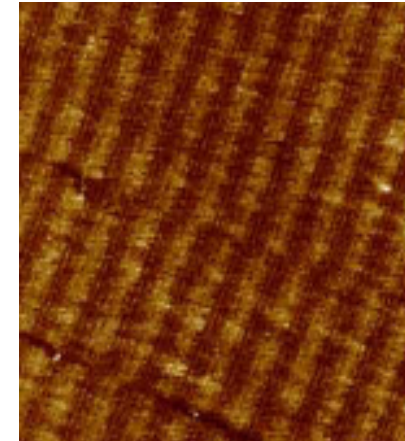


Pinhole Sample Analyser

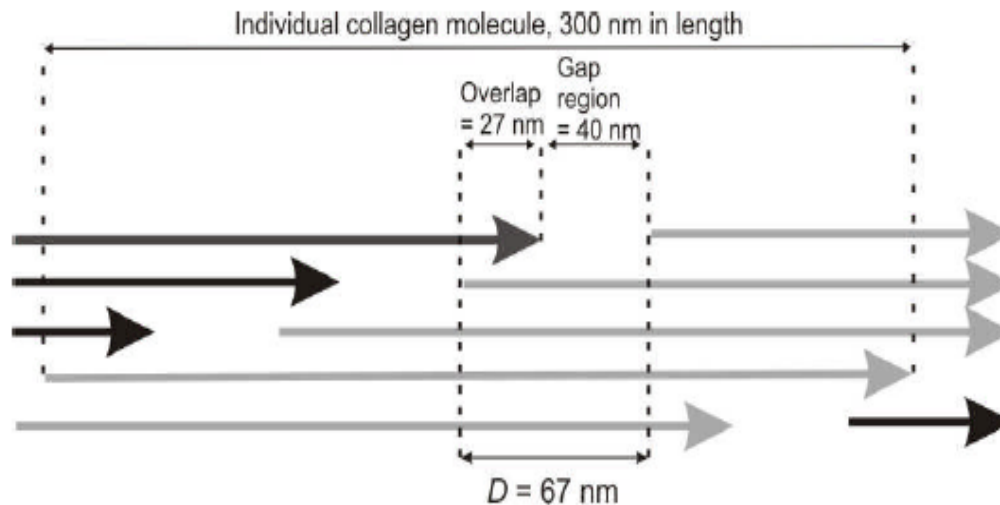
CXD applied to collagen

Felisa Berenguer, LCN

- Collagen is the most common protein in animal tissue (bones, teeth, tendon, cartilage, connective tissue)
- Potential applications in medicine (artificial bone, skin diseases)
- Collagen packing to built-up fibres is not completely understood
Different proposed models by Orgel 2007, Wess 2006, Bozec 2007 ...



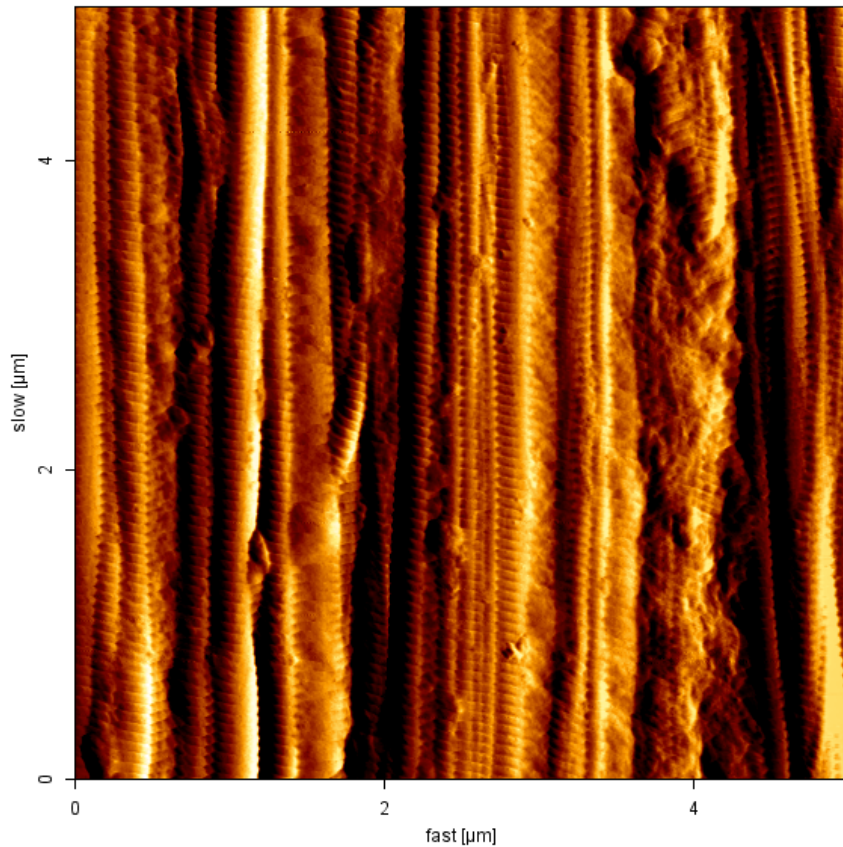
SEM [Cisneros, 2006]



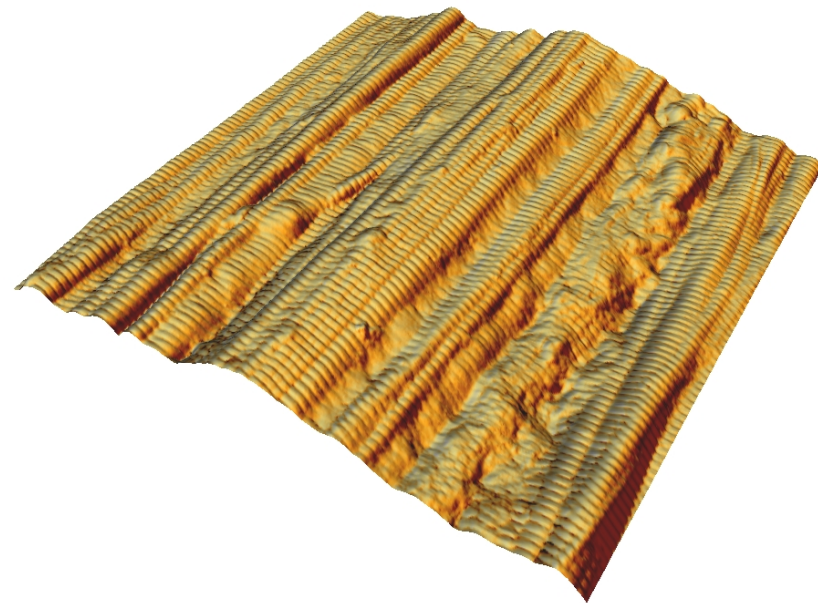
[Hodge and Petruska, 1976]

I. K. Robinson, OSA 2009

AFM imaging of rat-tail collagen



Dehydrated rat tail tendon tissue



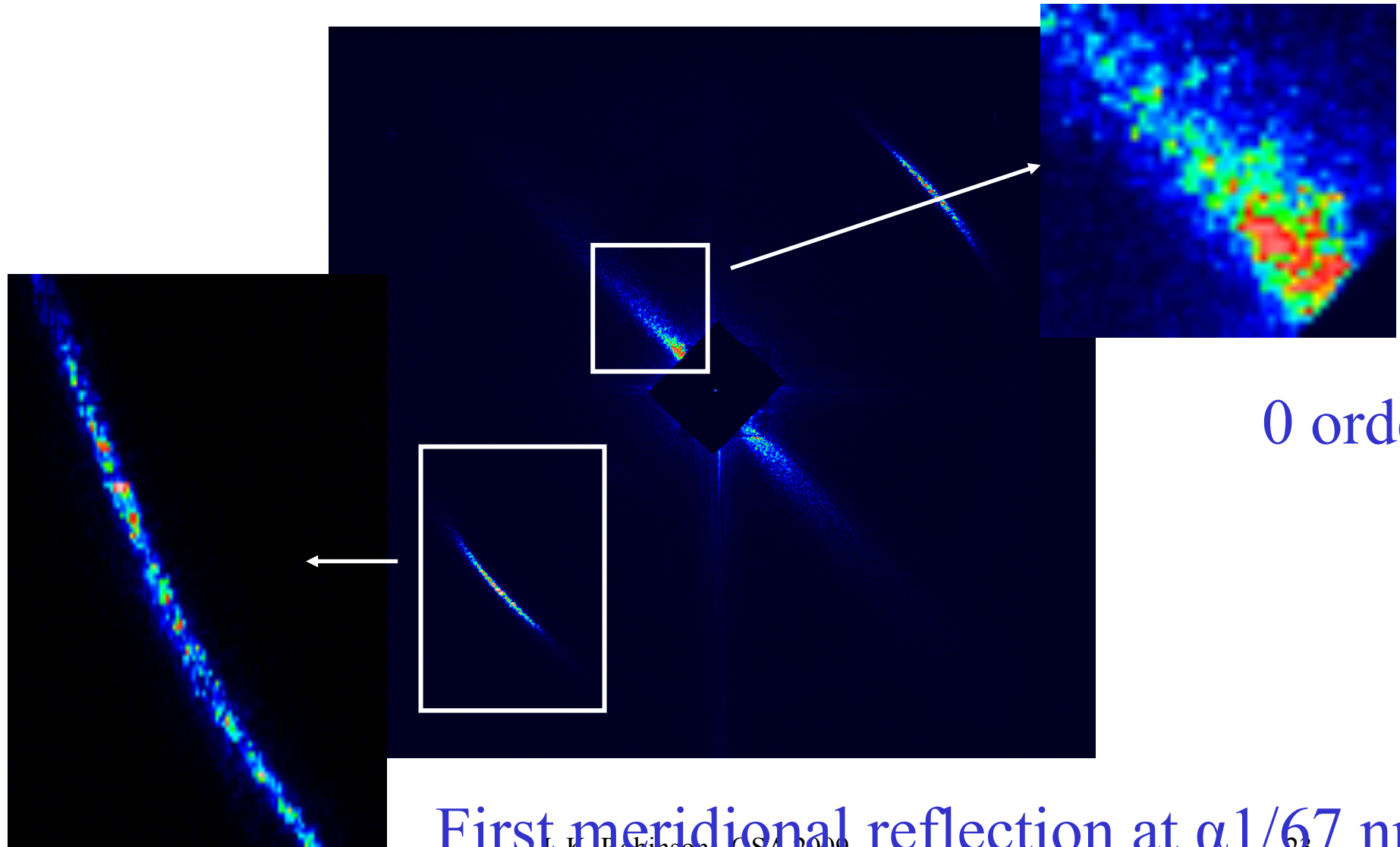
Strong D-banding with 67 nm periodicity:

→ Diffraction pattern with strong meridional maxima at multiples of $1/67 \text{ nm}^{-1}$

I. K. Robinson, OSA 2009

First experimental results on collagen

Rat tail tendon tissue

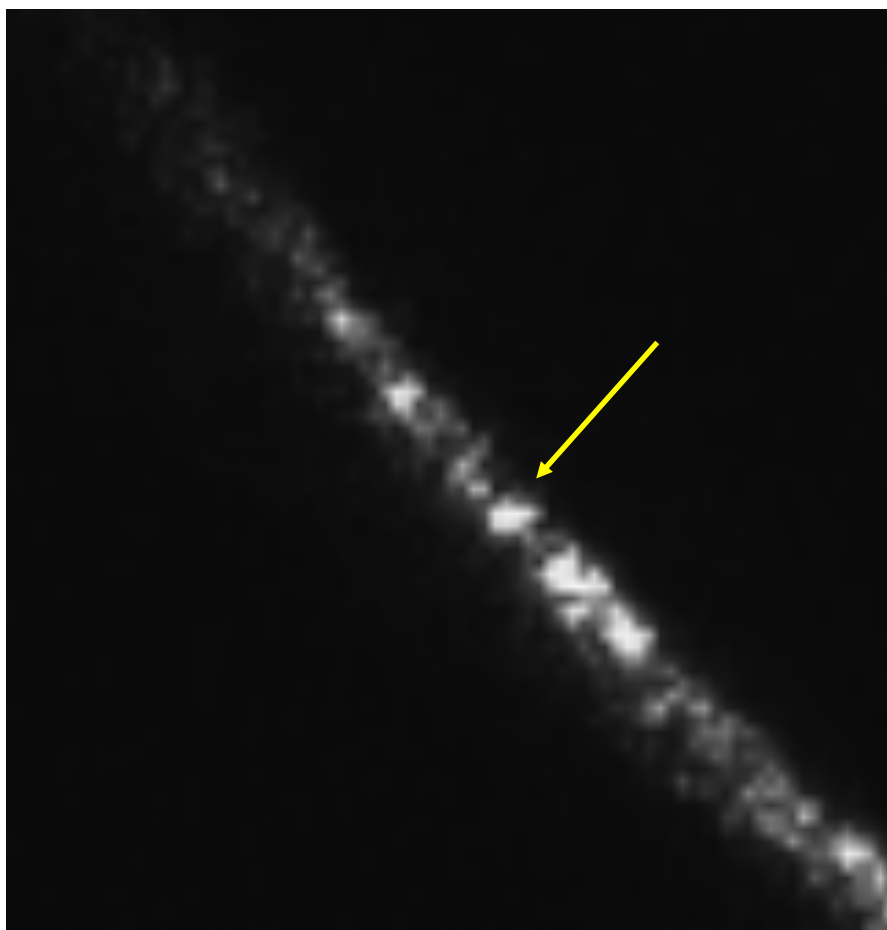


0 order

First meridional reflection at $\alpha 1/67 \text{ nm}^{-1}$

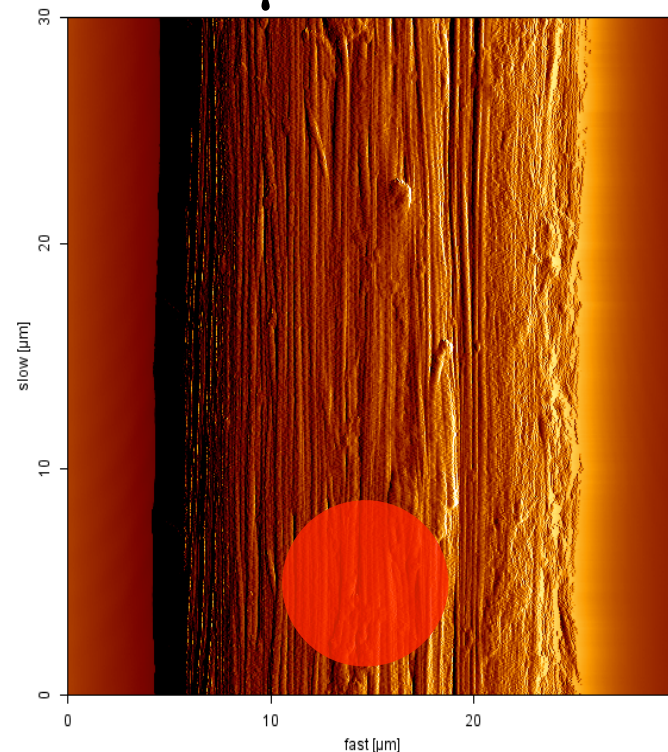
X-ray Ptychography

First meridional reflection



I. K. Robinson, OSA 2009

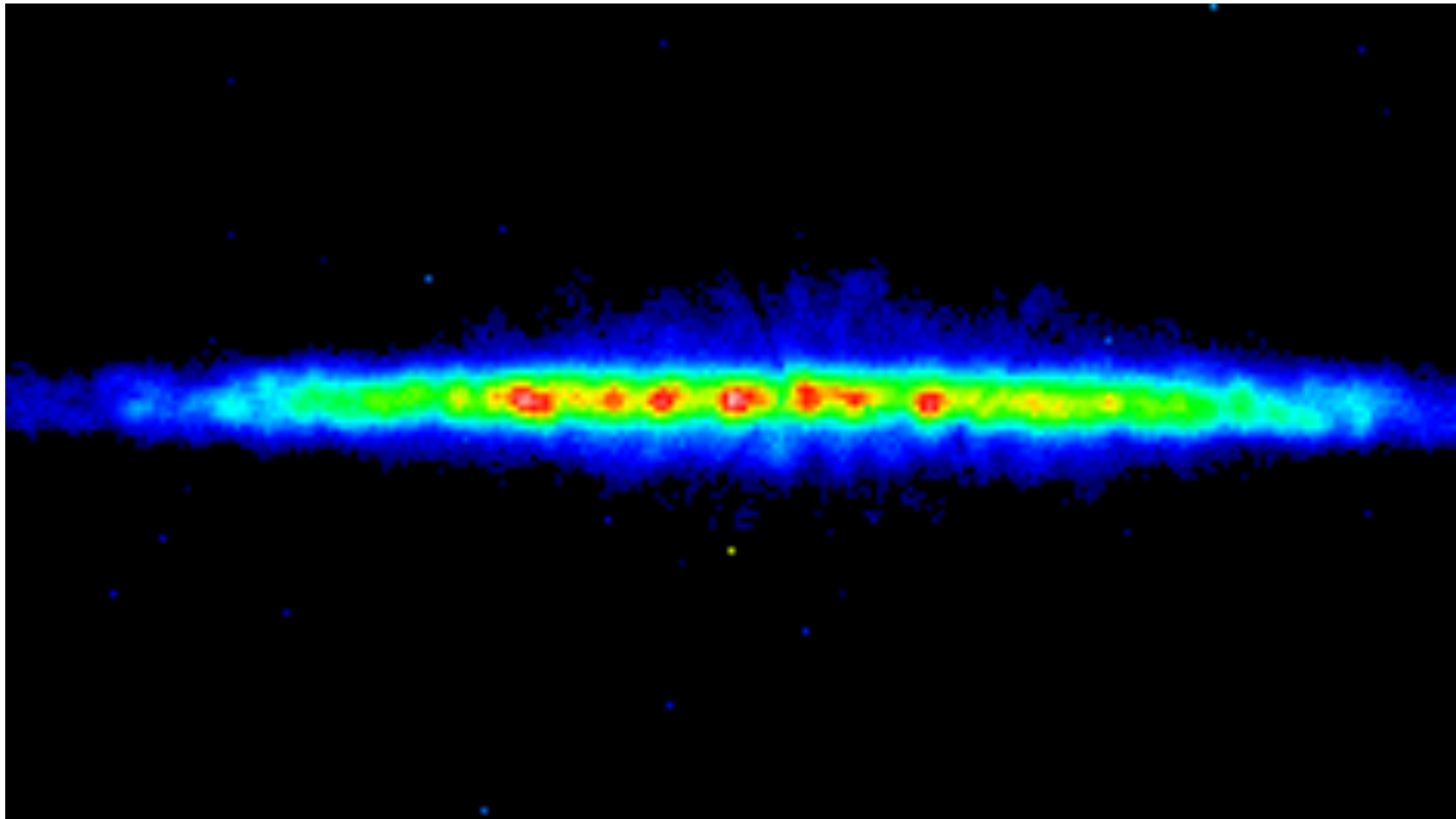
10 μm beam



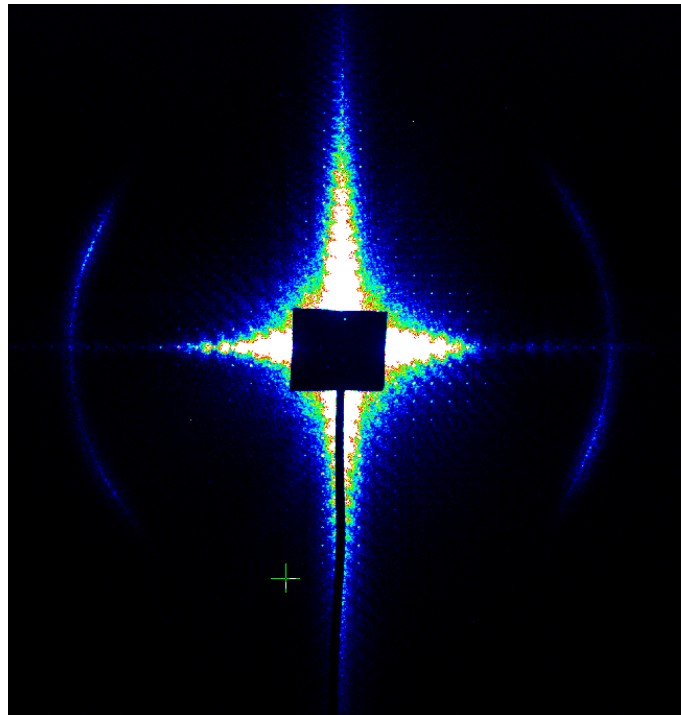
Dark field imaging:
collagen distribution in different
tissues

Improved collagen sample prep

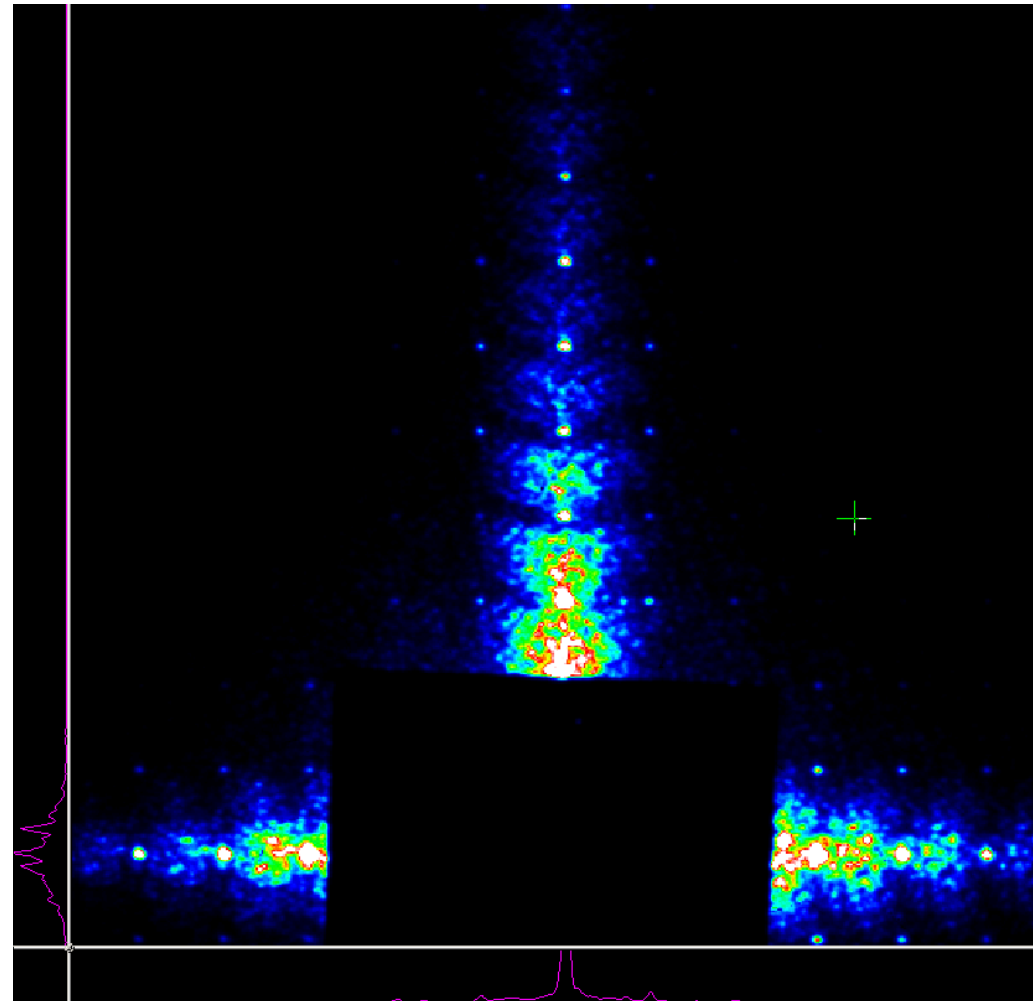
Diamond I-22, Nov 2008



Collagen rat tail tendon + phaseplate

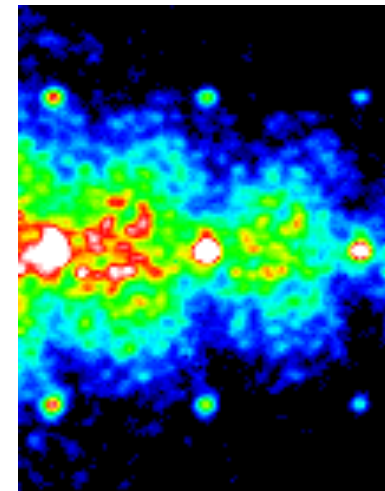
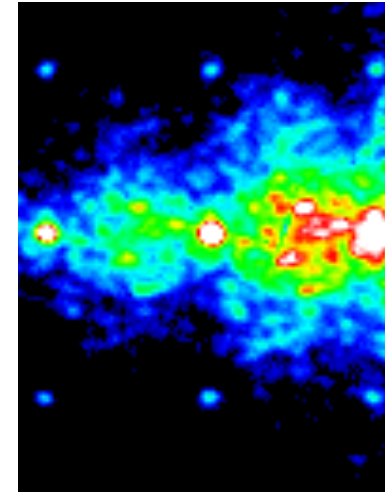
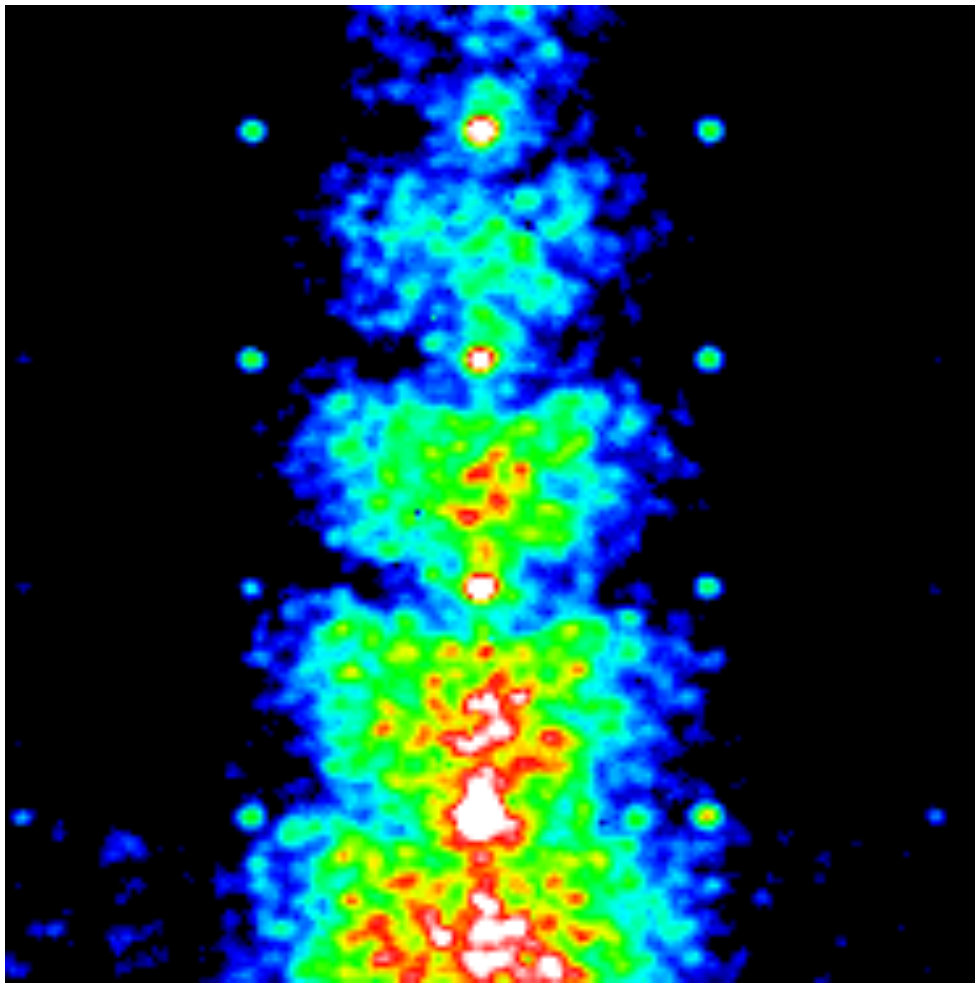


- * 0th order “repeated” in the meridian and equator
- * Strong phase modification due to phase plate!
- * No successful reconstruction



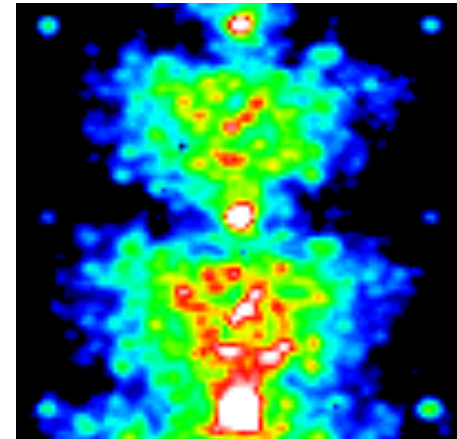
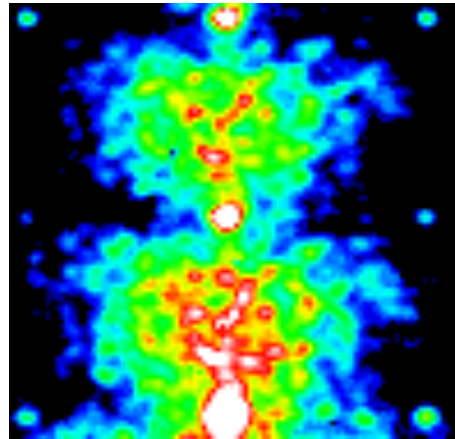
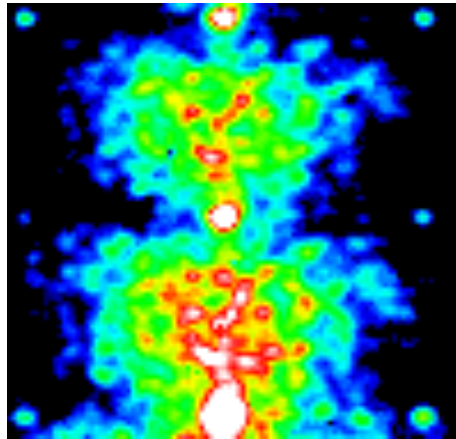
Phase plate scan near forward direction

1 μ m step per frame



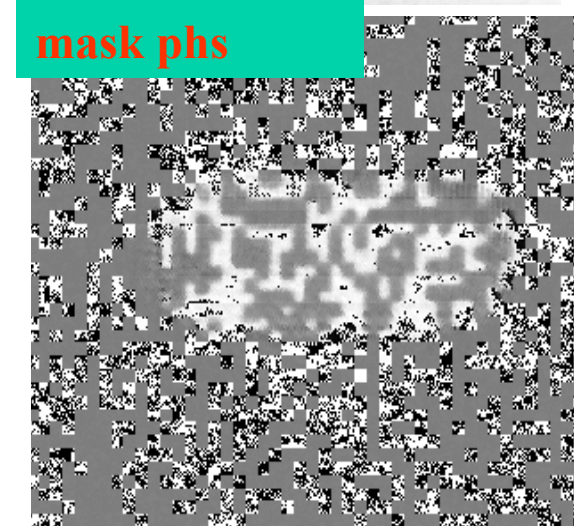
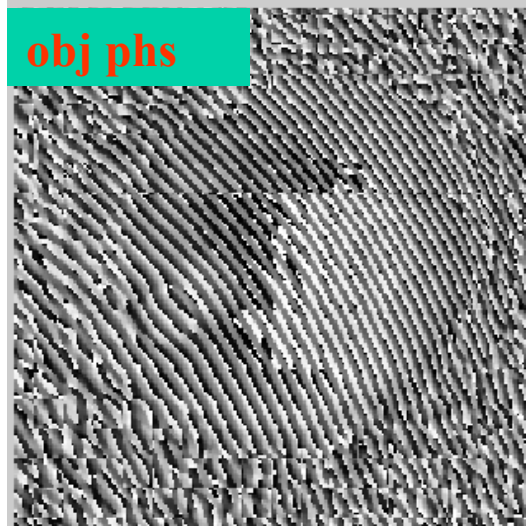
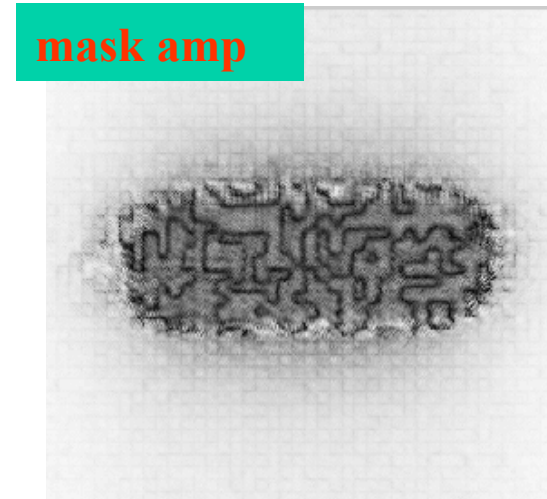
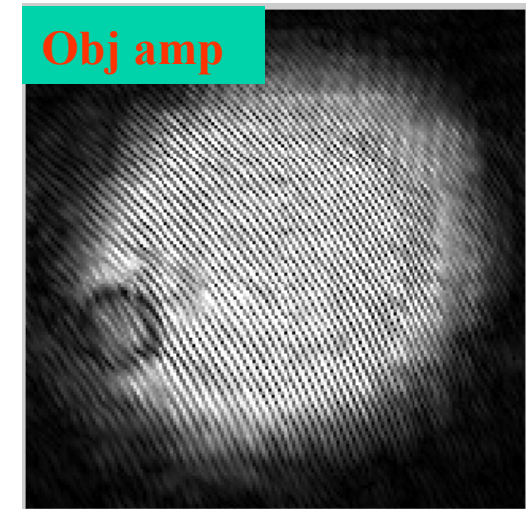
Collagen, buffer and empty cell

phplateFZP-236, -246 and -249



Reconstruction of Phase plate scan

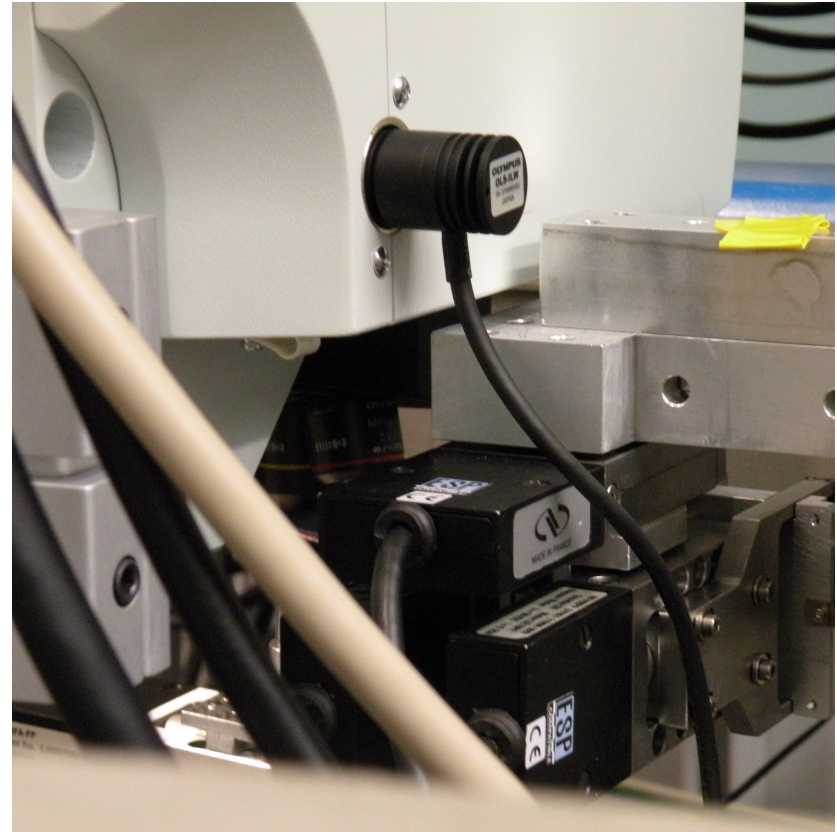
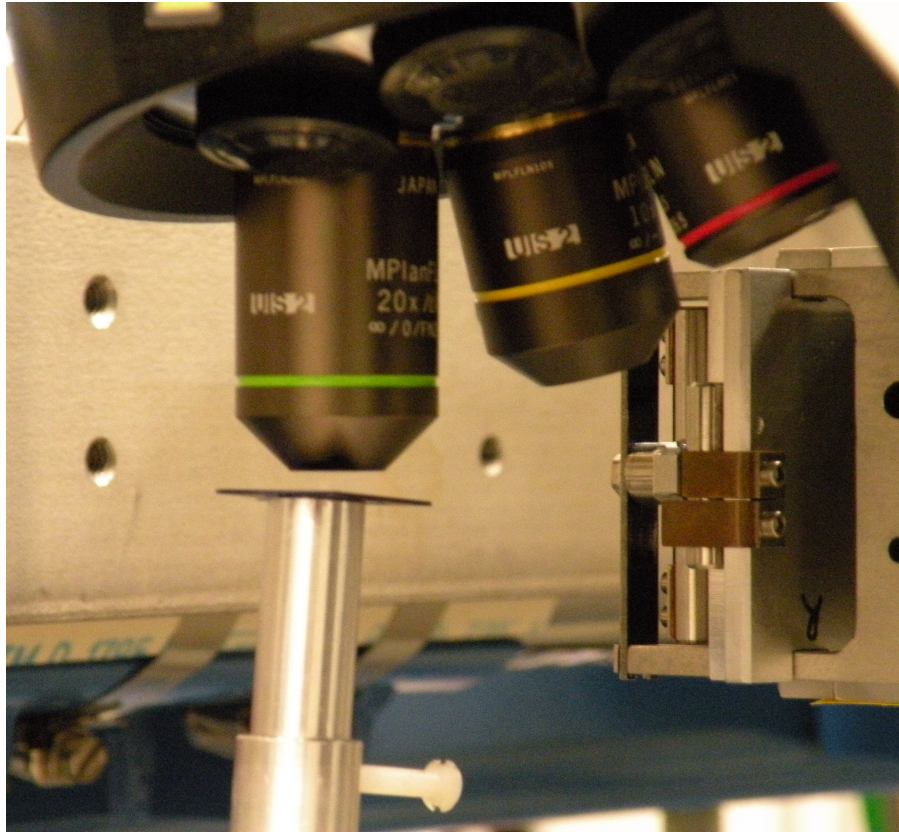
1 μm step per frame (512x512)



Reconstruction by F. Zhang (paper in preparation)

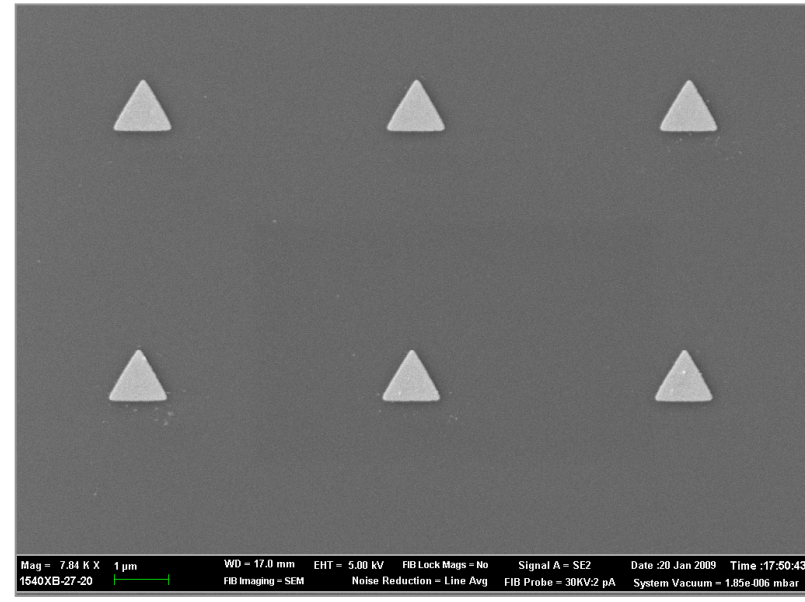
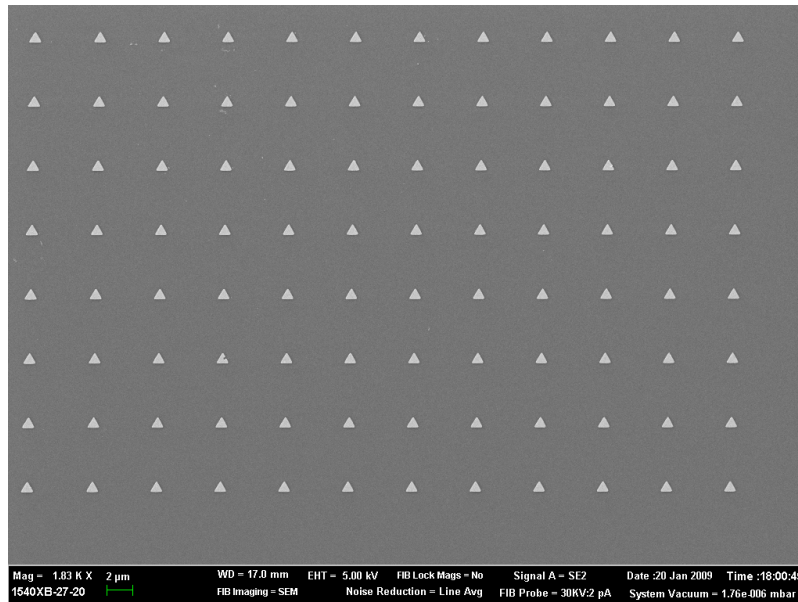
I. K. Robinson, OSA 2009

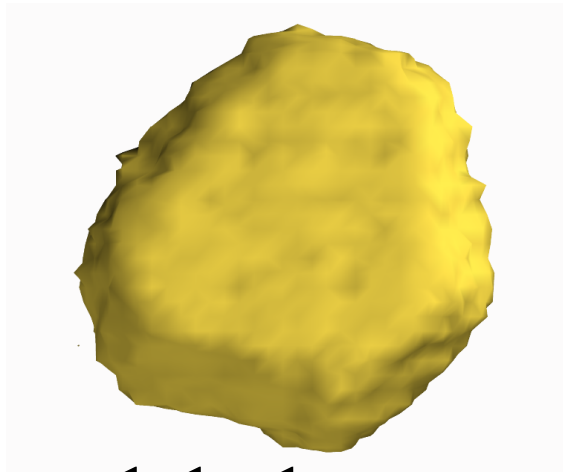
Confocal Alignment Microscope



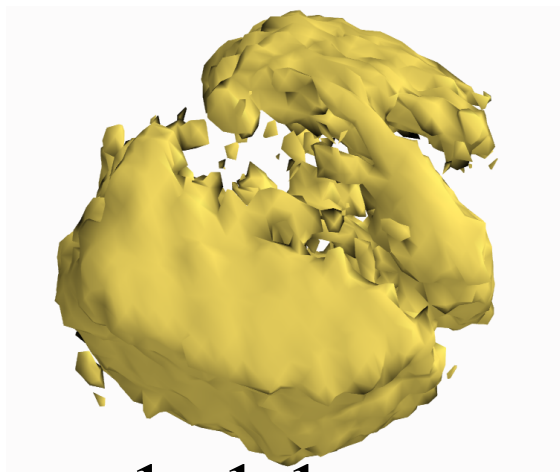
Patterned Au nanocrystal samples

N. Shimamoto, Waseda University, Japan

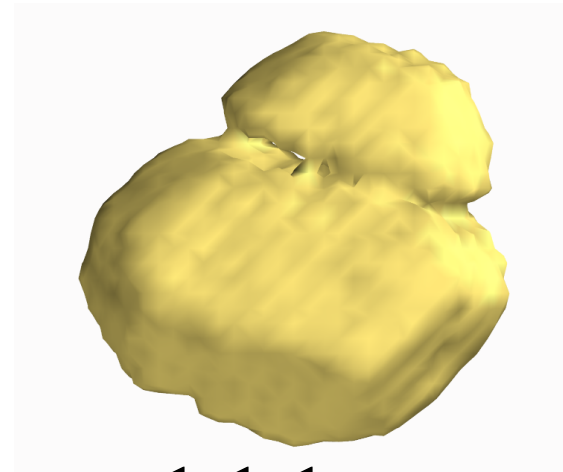




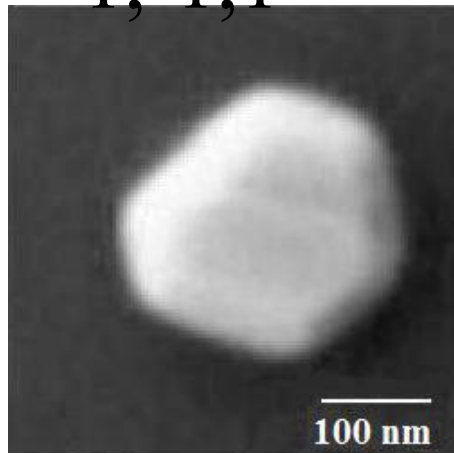
1,1,-1



1,-1,1

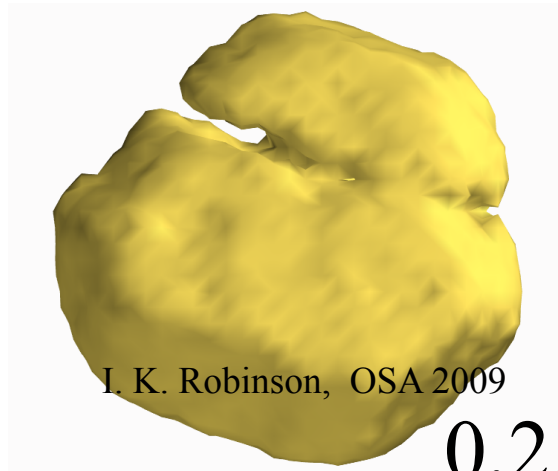
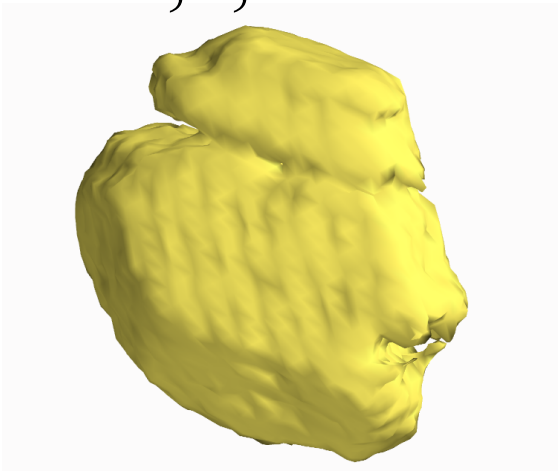


-1,1,1



100 nm

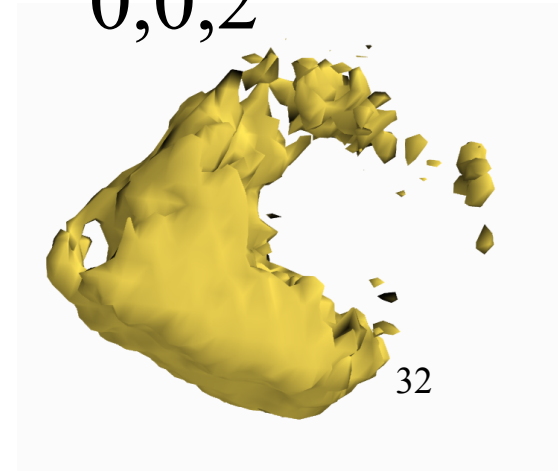
2,0,0



I. K. Robinson, OSA 2009

0,2,0

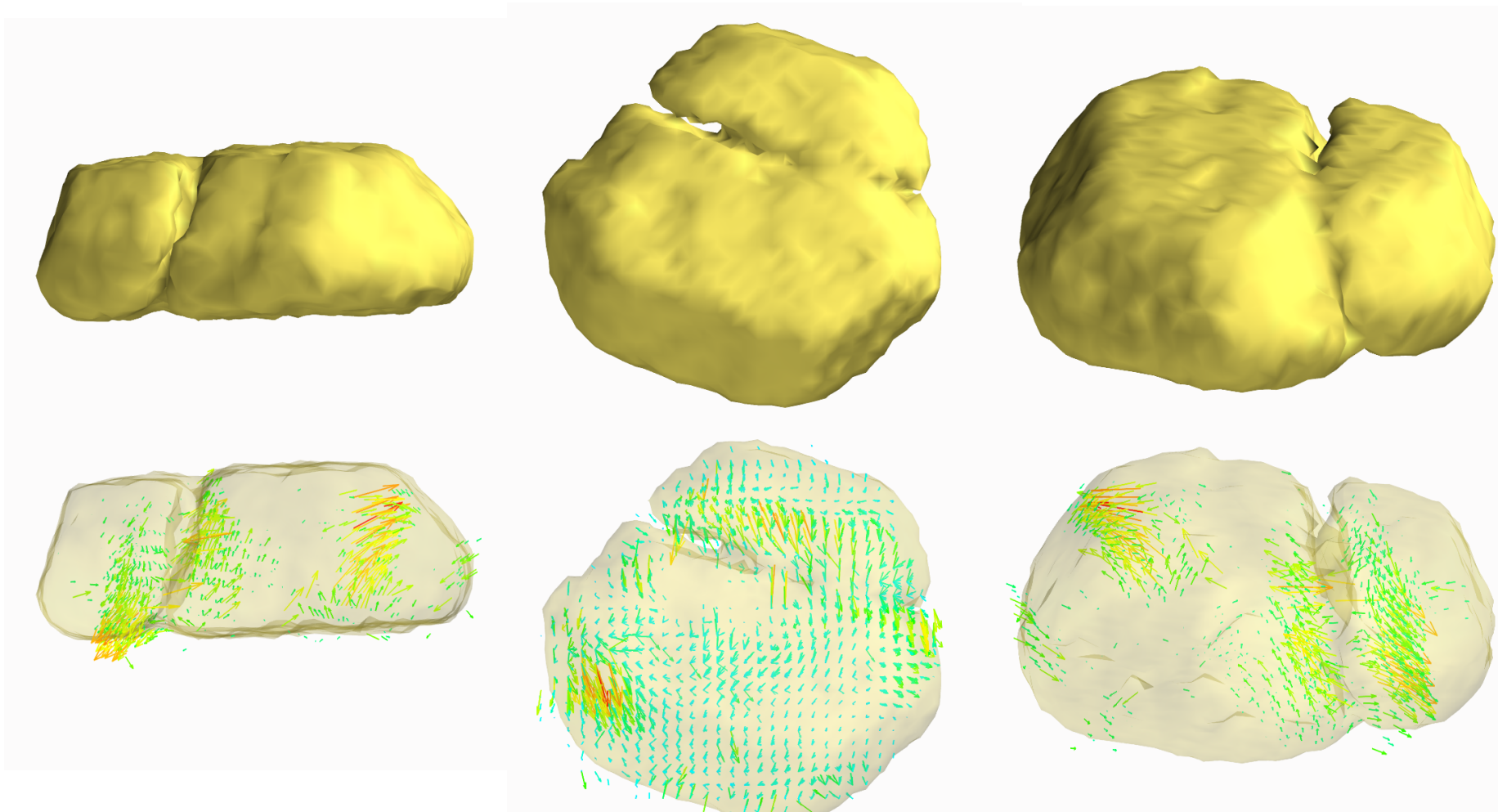
0,0,2



32

Vector displacement field

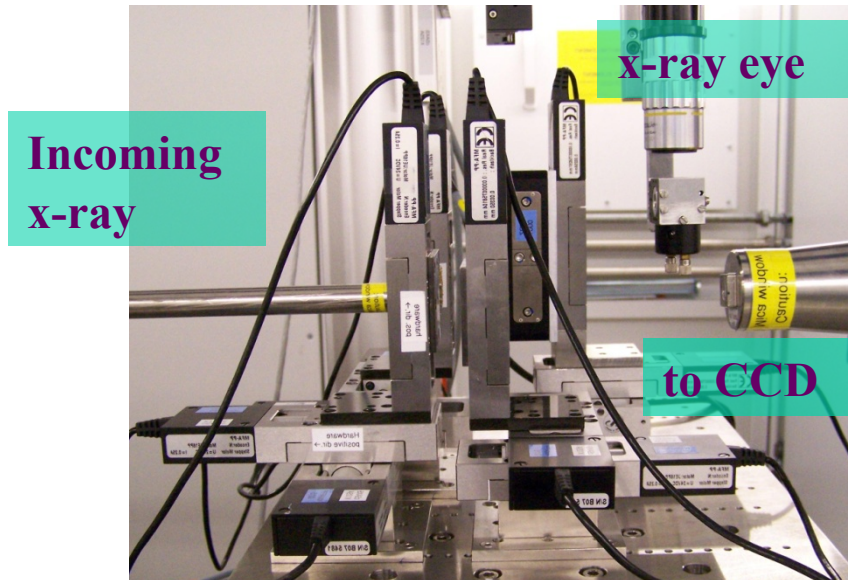
Merged reconstructions from (11-1) (020) and (-111)



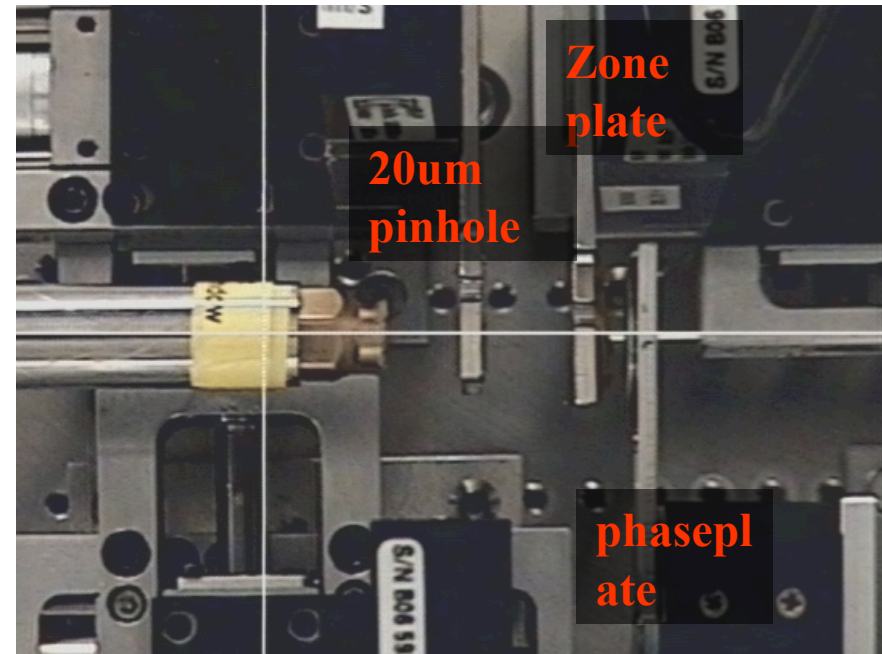
Conclusions

- Small crystal imaging
- Ptychography reveals probe structure also
- Phase plate demonstrated
- New custom phase plates
- Preparations under way for LCLS

Experimental setup cSAXS (SLS)



Side view



Top view (zoomed in)