

Use of coherent X-ray beams in biophysics

Ian Robinson

Jyotsana Lal

Felisa Berenguer

Ross Harder

Ross Harder

Lee Makowski

Richard Bean

Cameron Kewish

Max-Planck-Institut fuer Kolloid- und
Grenzflaechenforschung,

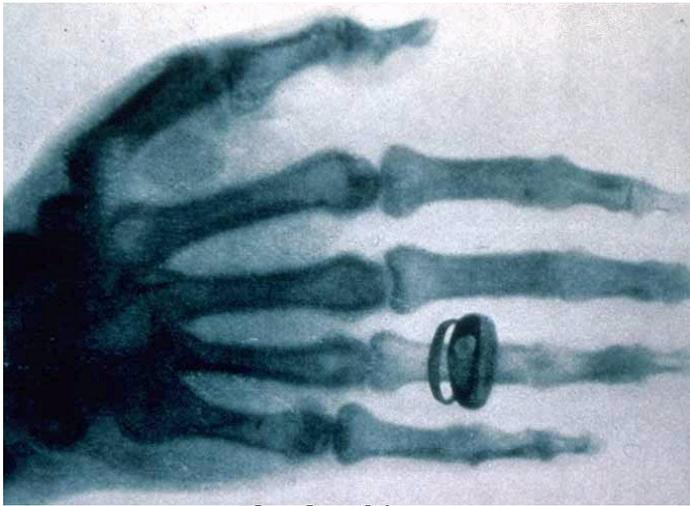
Joan Vila

Golm, October 2010

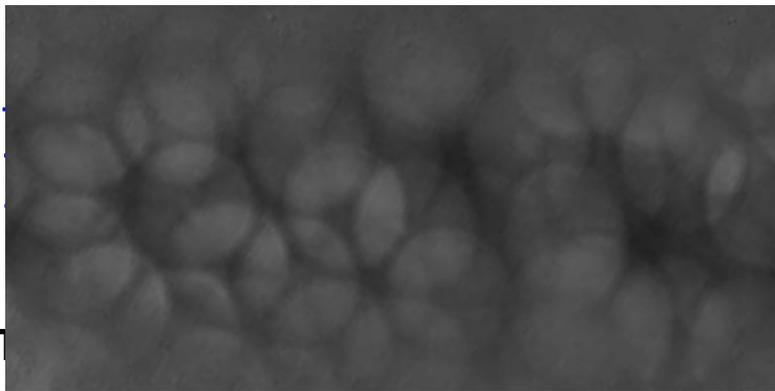
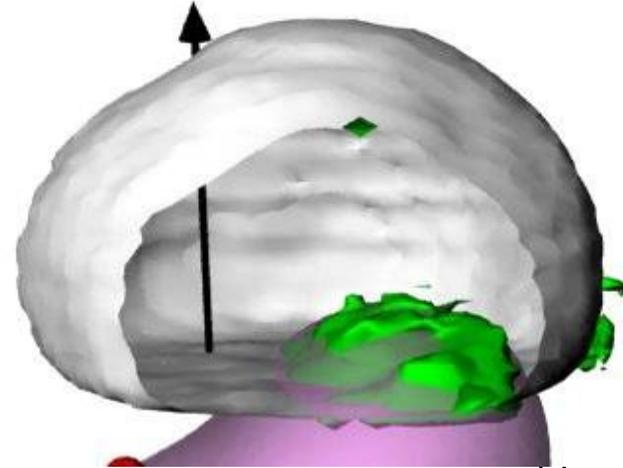
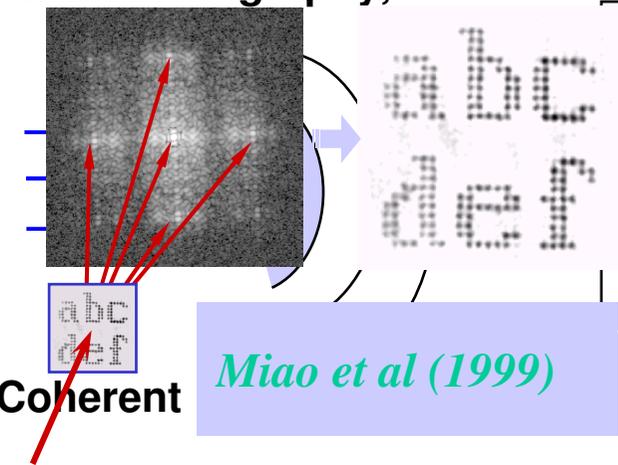
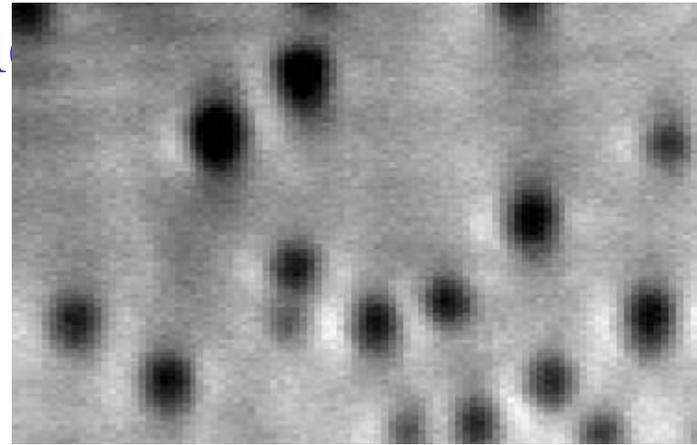
Fucaï Zhang

Outline

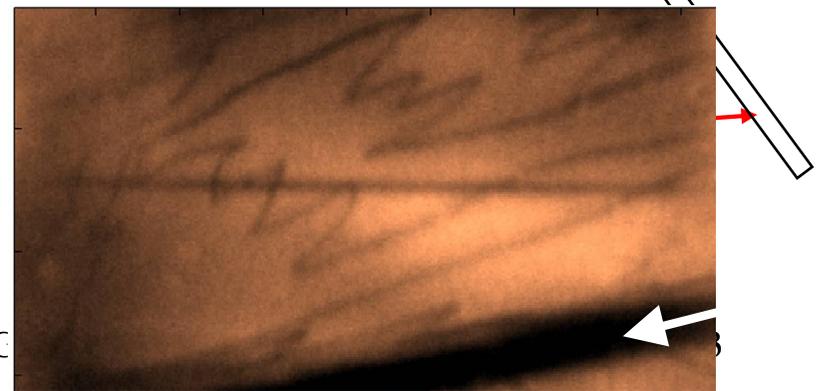
- Imaging with X-rays
- Coherence based imaging
- Energy bio-materials
- Collagen and biomineralisation
- Grating methods
- Future applications



eld Mi

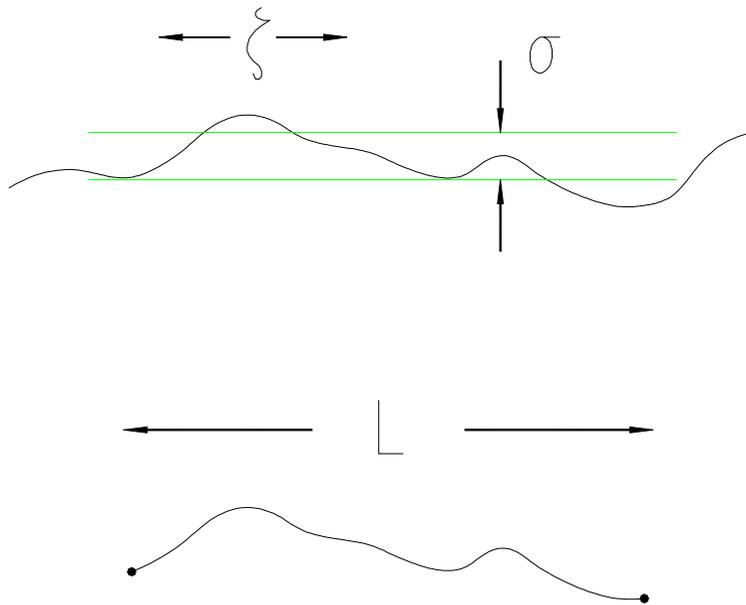


n, MPI C

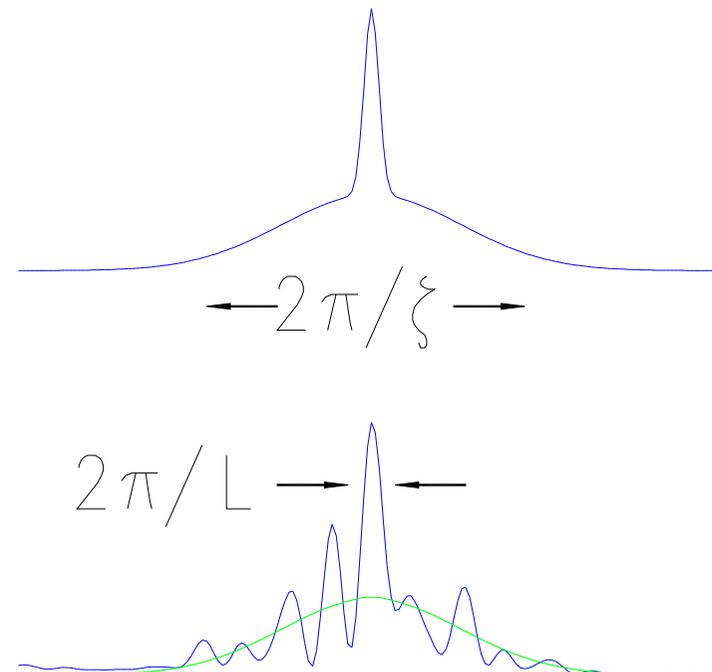


Diffuse Scattering acquires fine structure with a Coherent Beam

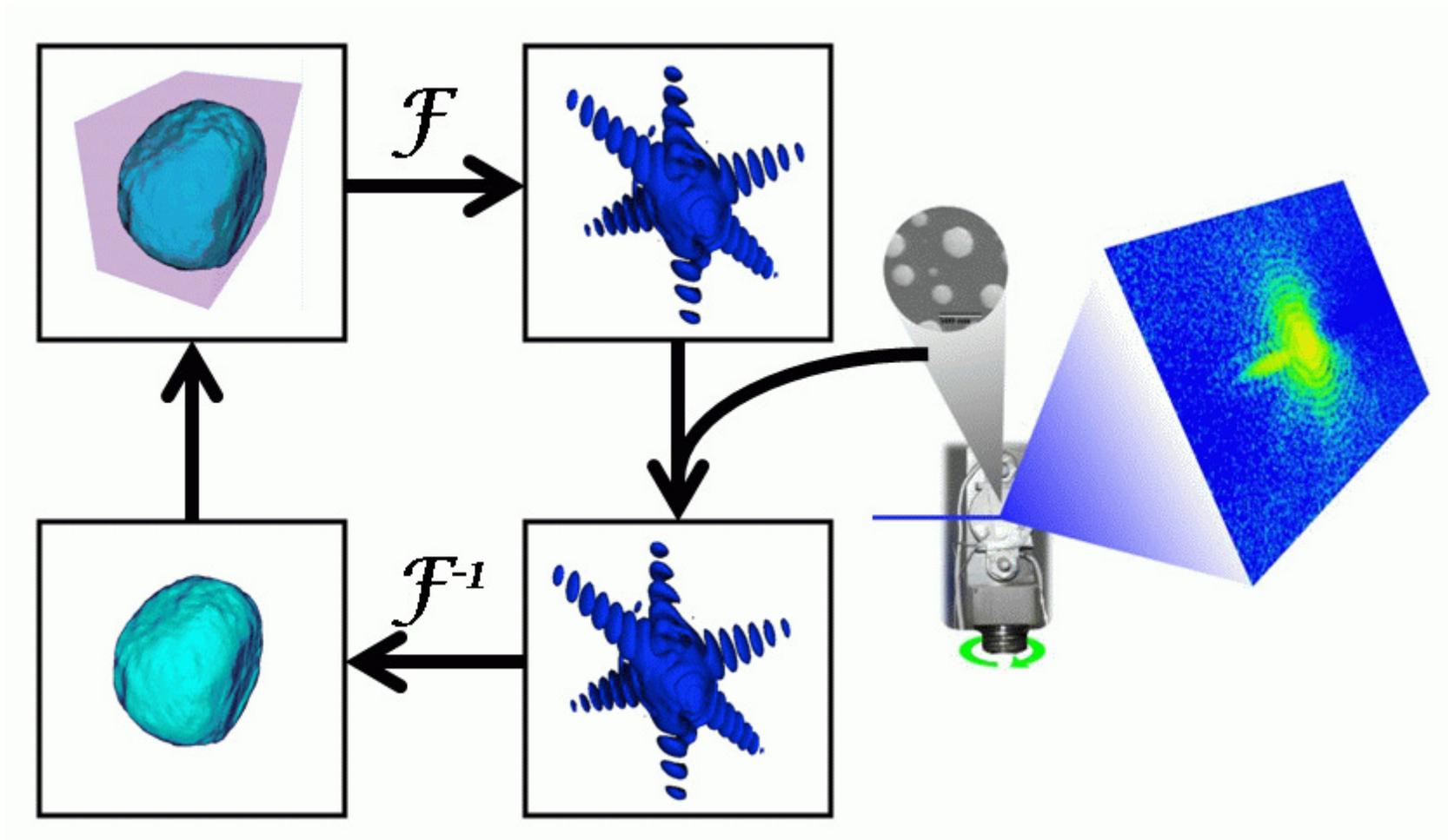
Real Space



Reciprocal Space



Generic “Error Reduction” method



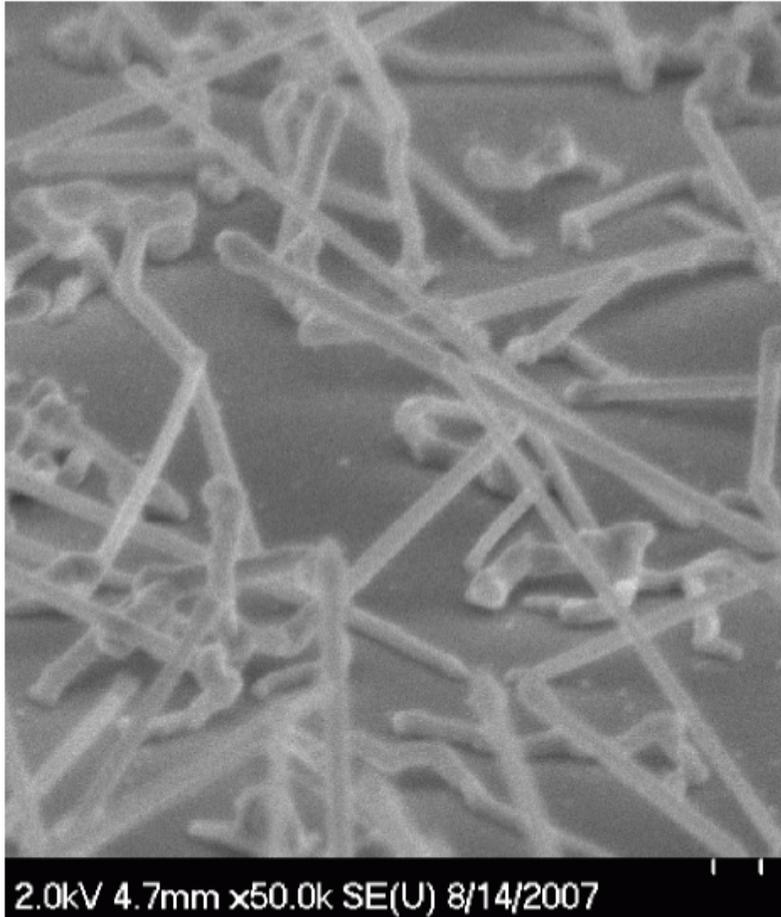
J. R. Fienup *Appl. Opt.* 21 2758 (1982)

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

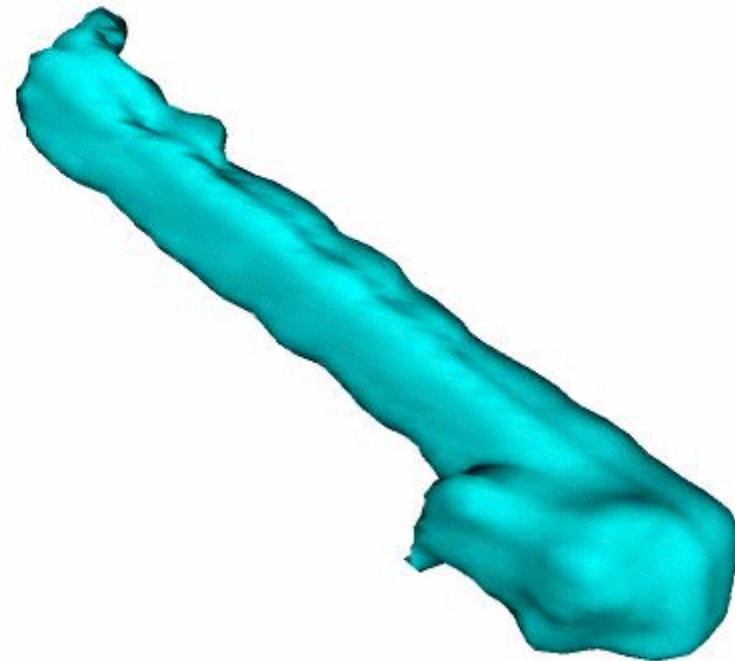
I. K. Robinson, MPI Golm 2010

Reconstruction of InP nanowire

CVD on Si, Suneel Kodambaka, UCLA

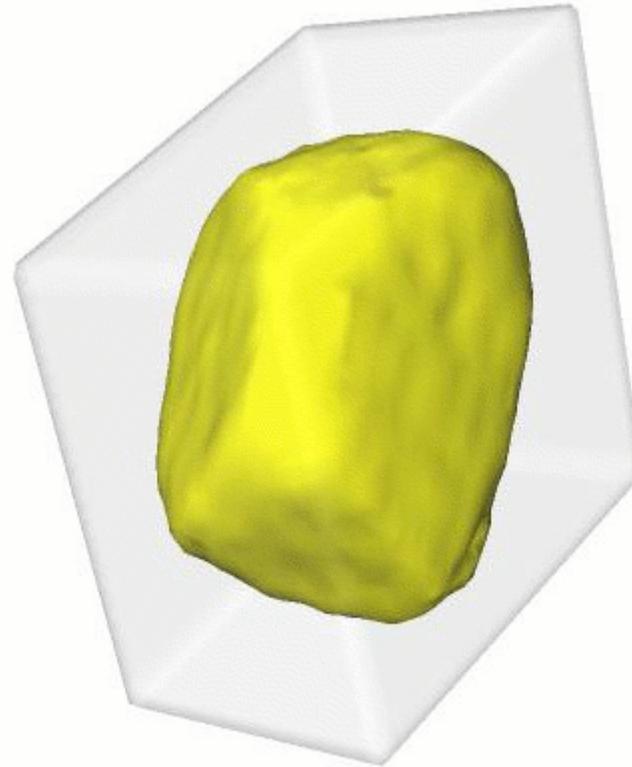
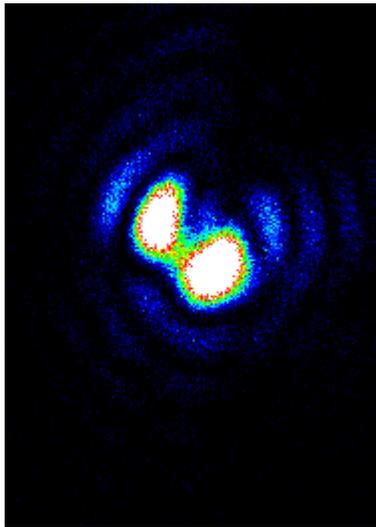


InP nanowires grown on Si (111)



Gold nanocrystal reconstruction

showing support used for 20 HIO followed by 10 ER



Cellulose Granules in Corn (Maize)

Jyotsana Lal, Ross Harder, Lee Makowski, ANL

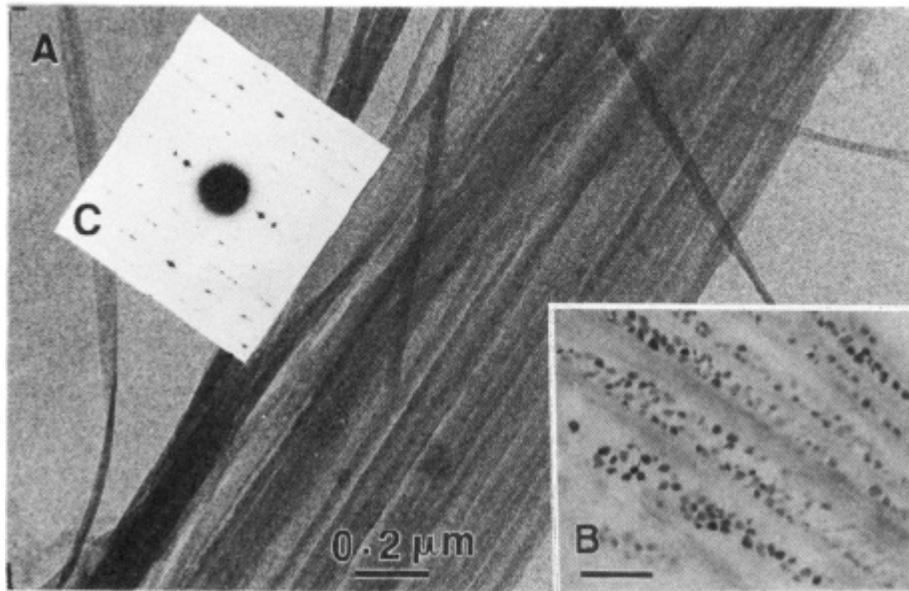
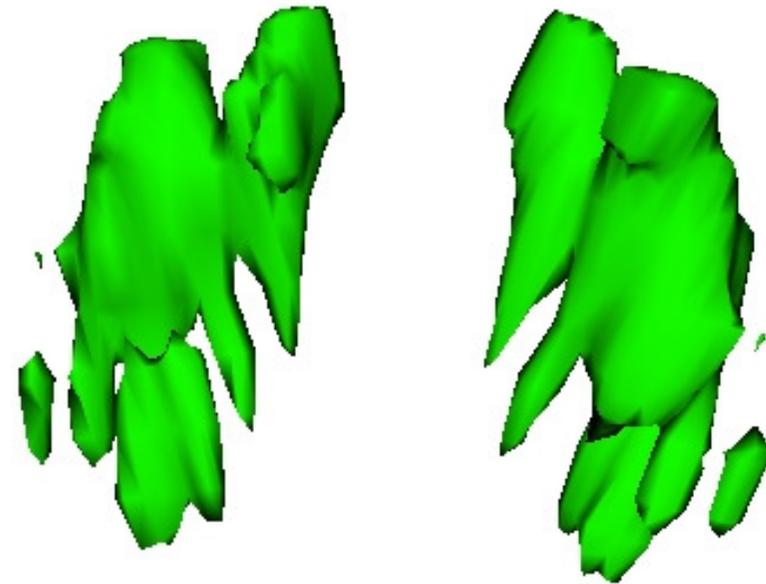
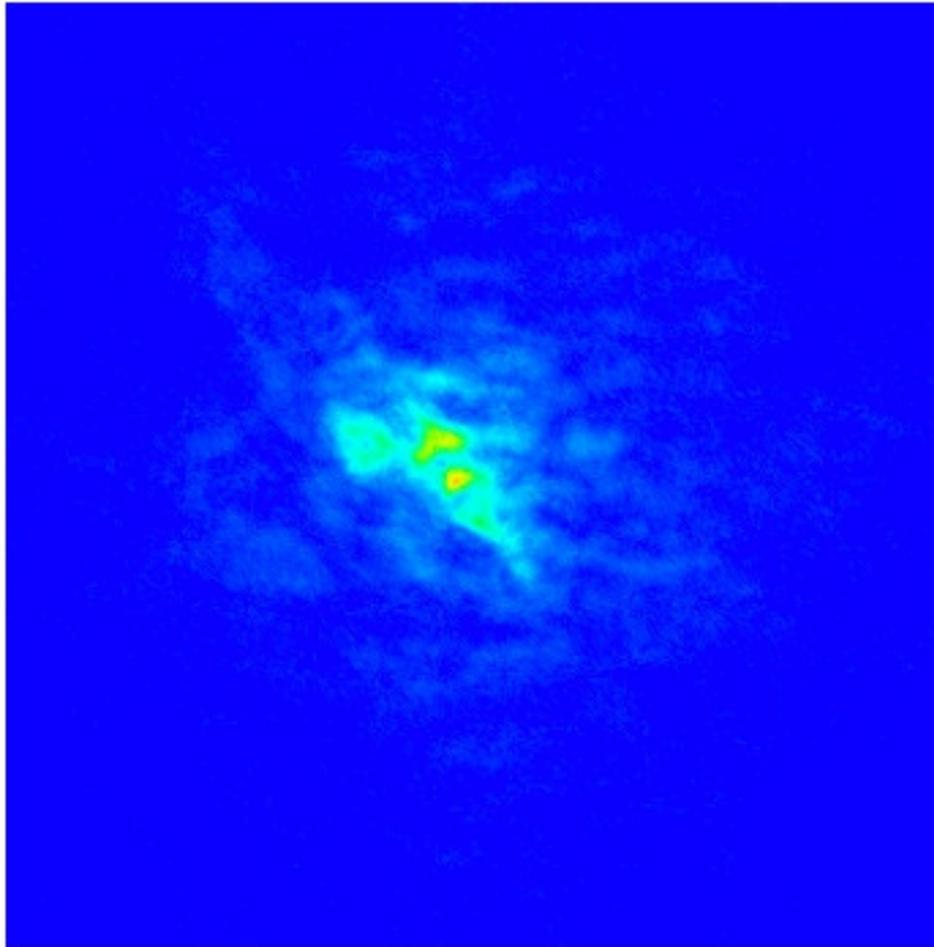
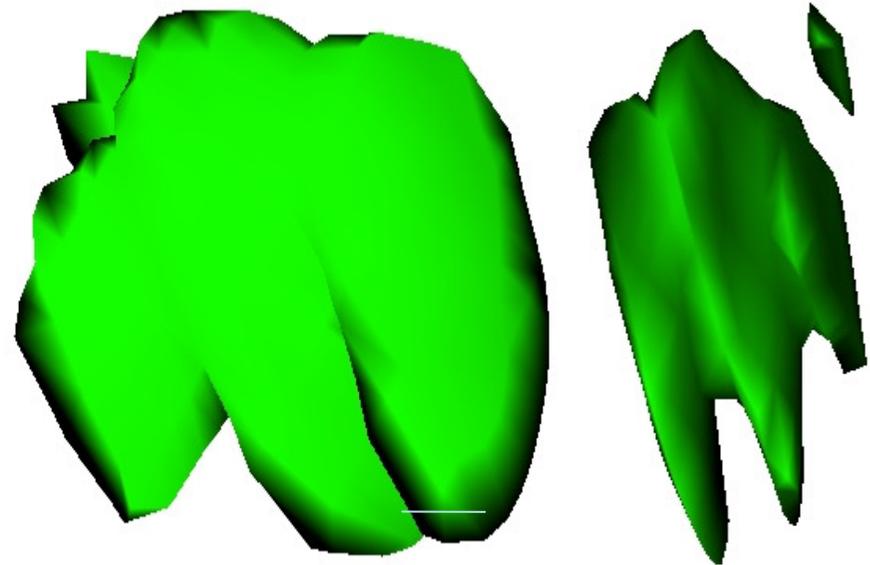
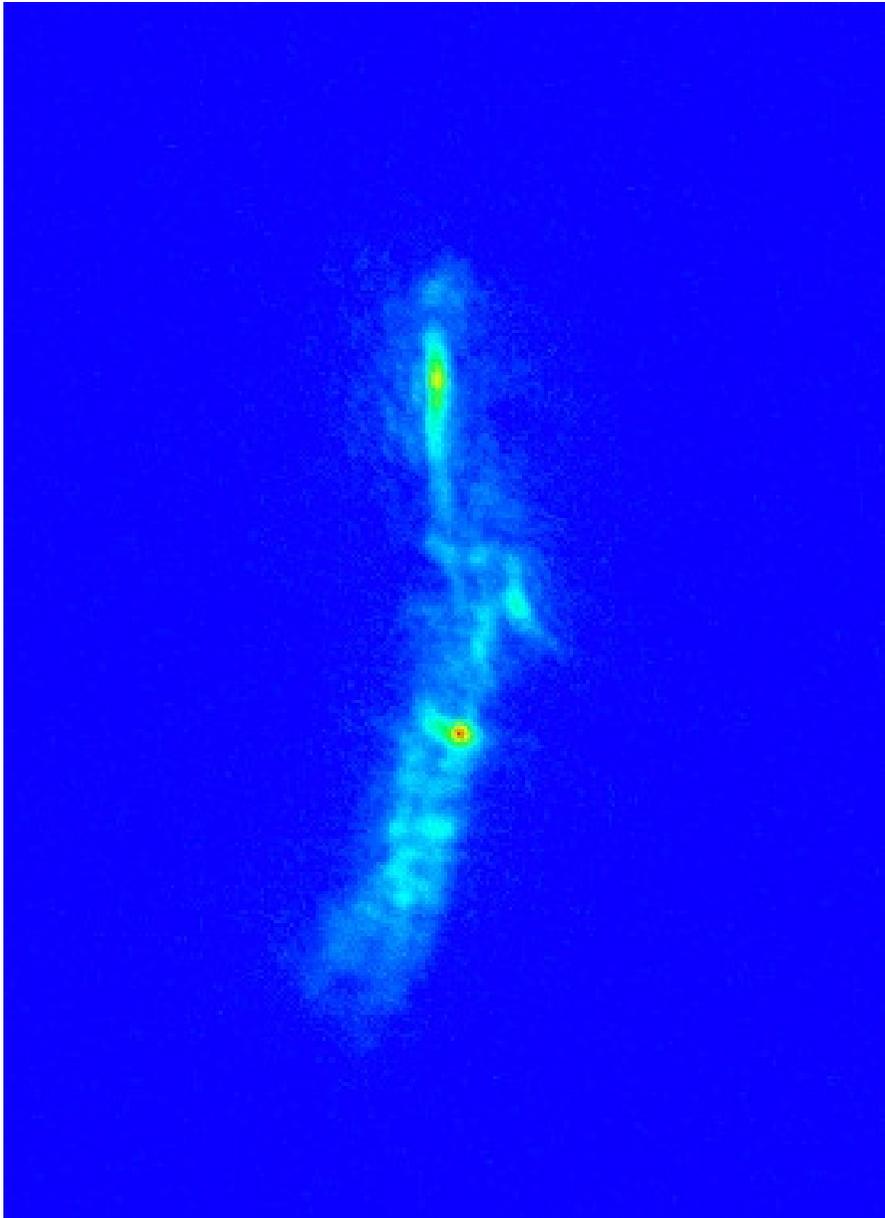


Figure 1. Ultrastructural features of *Microdictyon* cellulose microfibrils: (A) a thin layer of microfibrils unstained and unshadowed observed under low-dose imaging conditions; (B) bright-field electron micrograph of the cross section of a cell wall fragment of *Microdictyon* (the almost square section of the microfibrils is well denoted in this diffraction contrast image); (C) electron diffraction diagram of an area such as that located in the lower part of Figure 1A and recorded on an area having 200 nm in diameter.

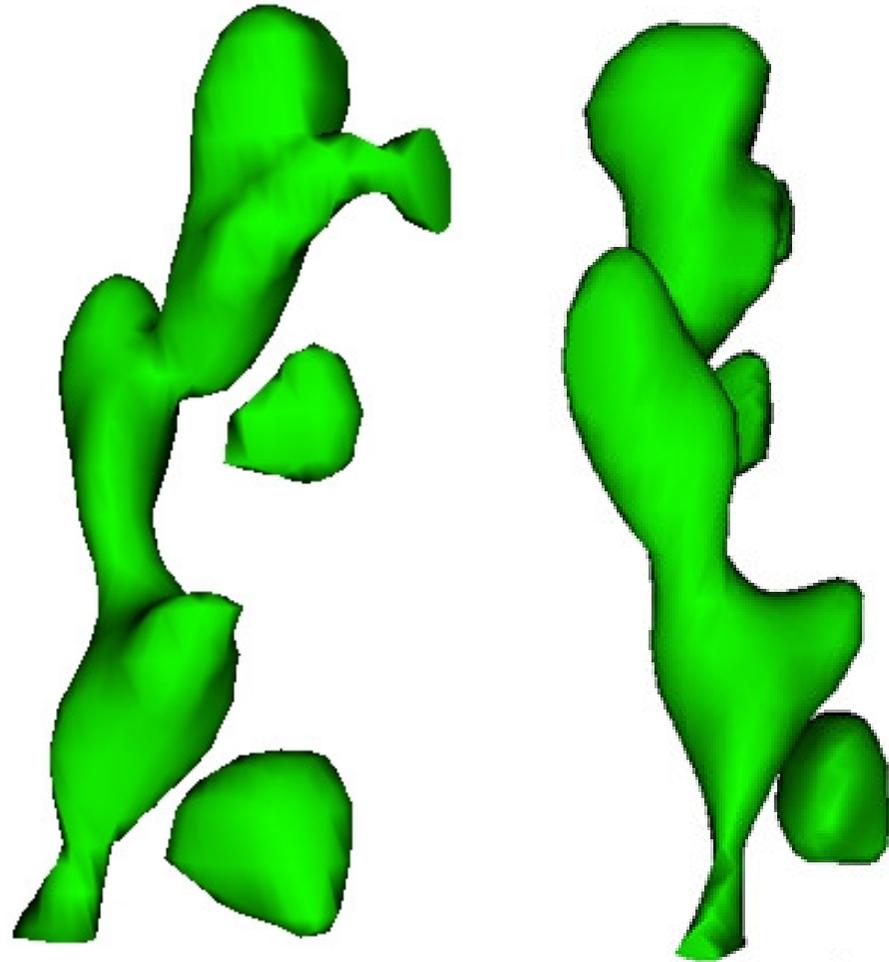
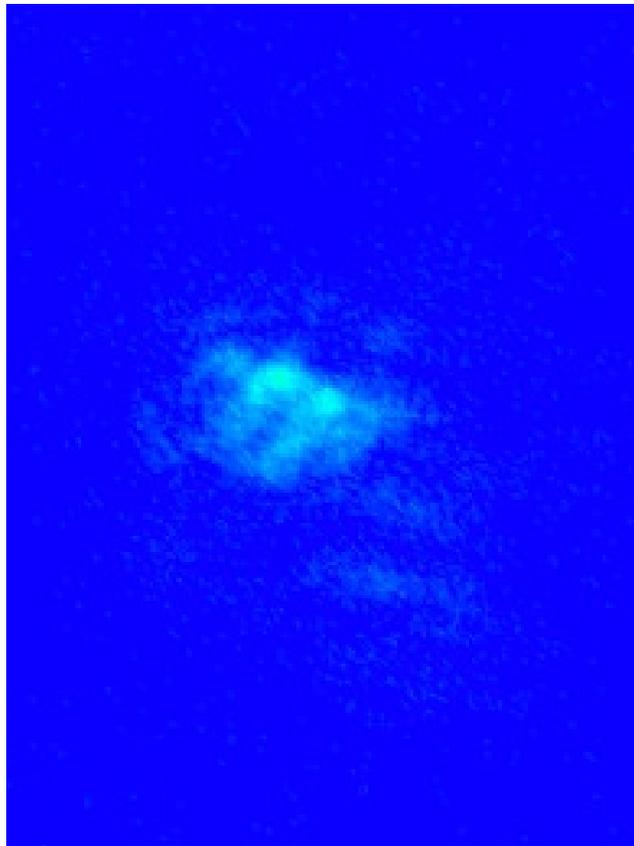
Crystals formed through coalescence of fibrils



Crystals formed through packing of flat plates



Helical arrangements of compact domains

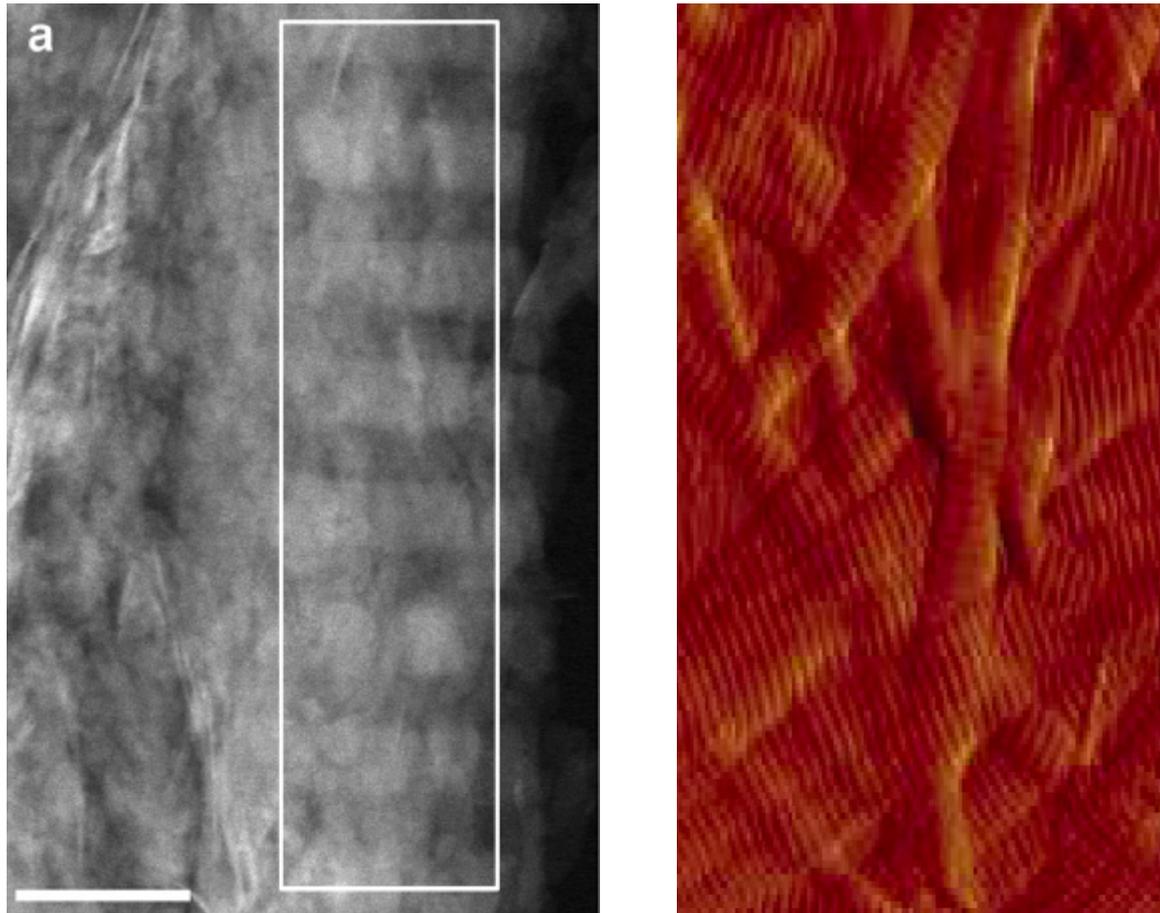


I. K. Robinson, *Journal of Statistical Mechanics*

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Mike Horton's inspiration

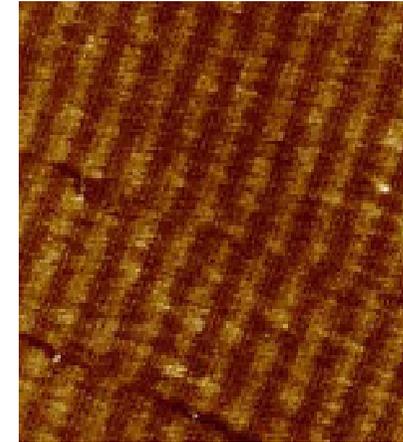
STEM biomineralisation, AFM



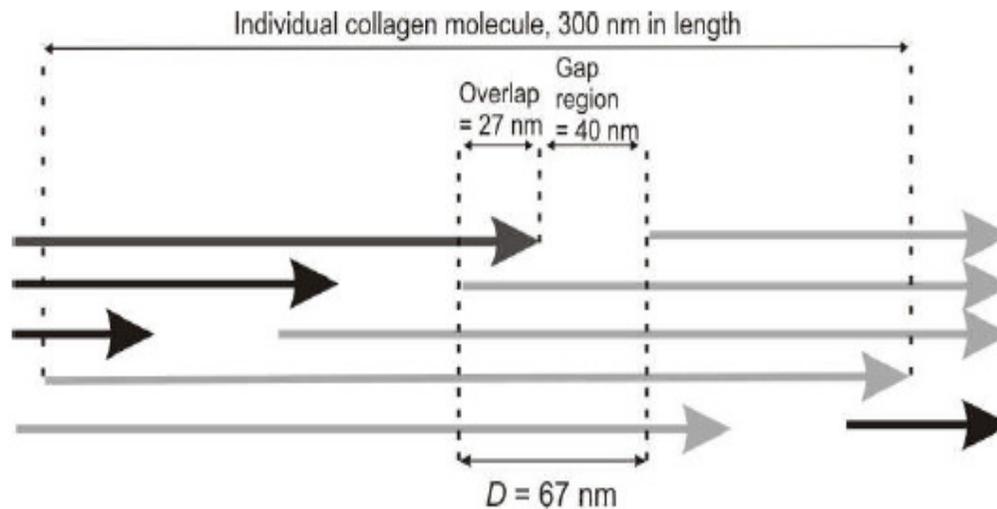
Collagen Primary Structure

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]

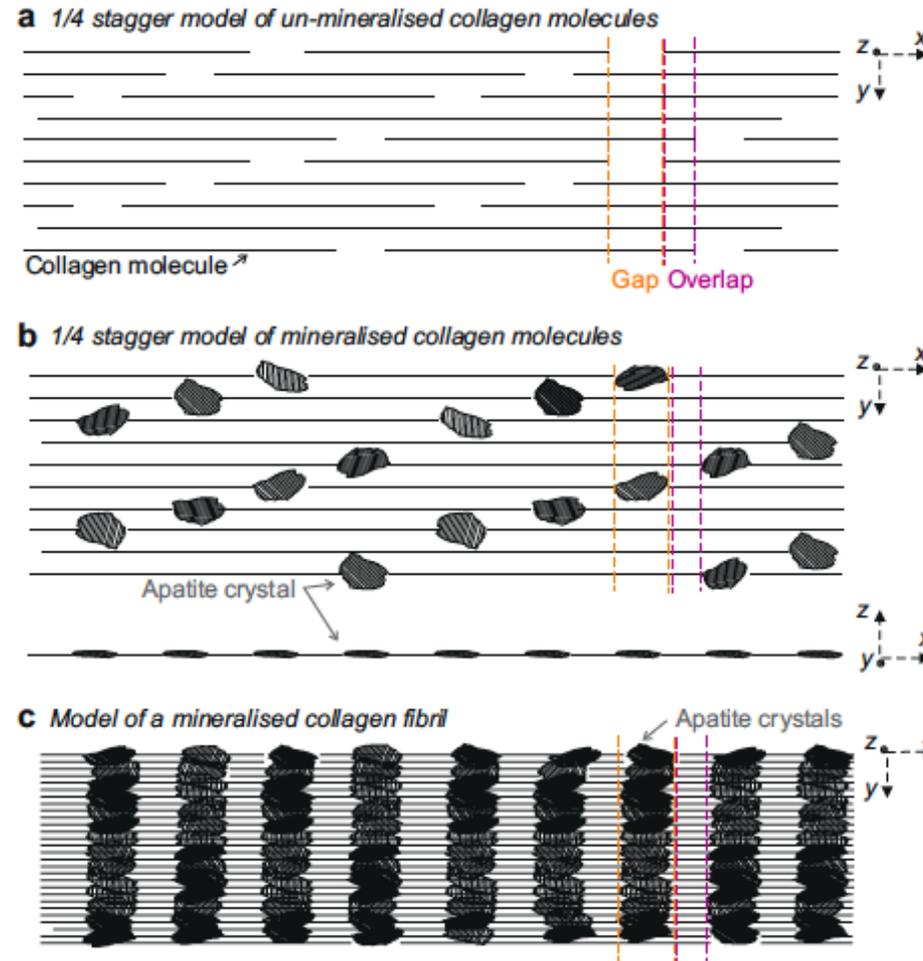


[Hodge and Petruska, 1976]

I. K. Robinson, MPI Golm 2010

Biom mineralisation Model

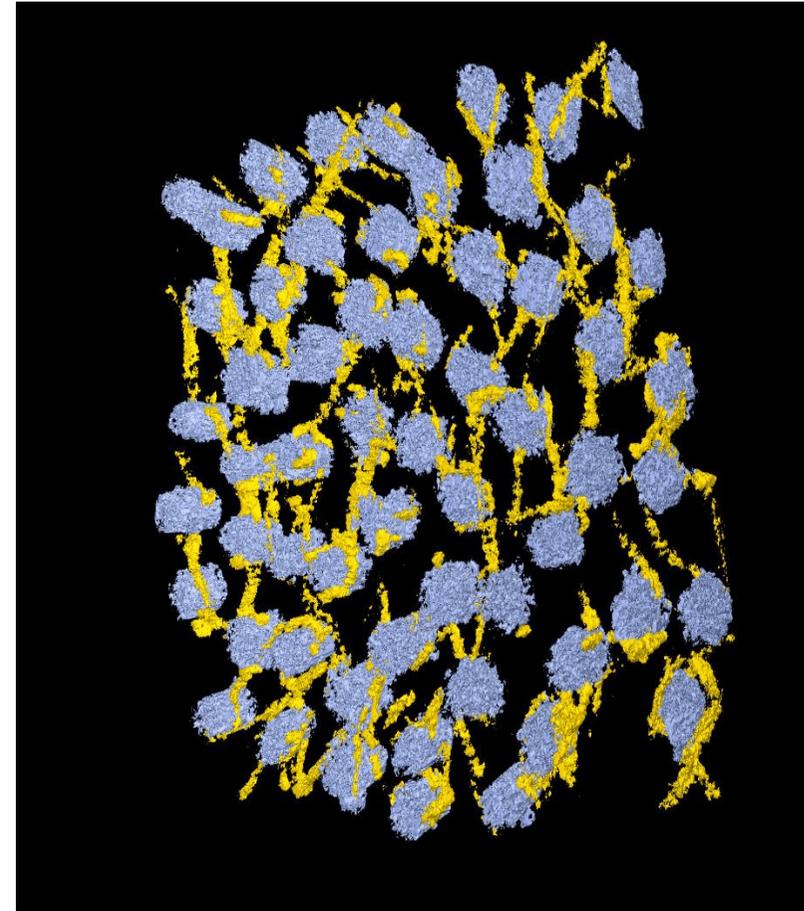
Jantou-Morris, V; Horton, MA; McComb, DW
Biomaterials 31 5275 (2010)



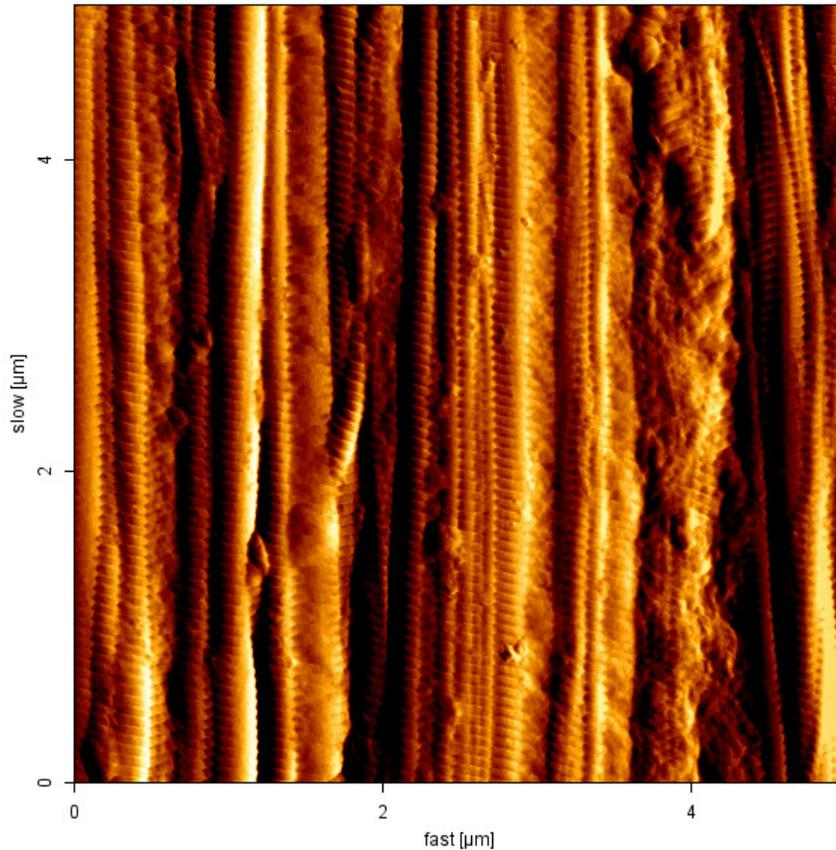
BONE ·3D tomographic reconstruction, X-ray
(J. Ritchie)



CORNEA ·3D tomographic reconstruction from EM
(K. Meek)



AFM imaging of rat-tail collagen



Dehydrated rat tail tendon tissue

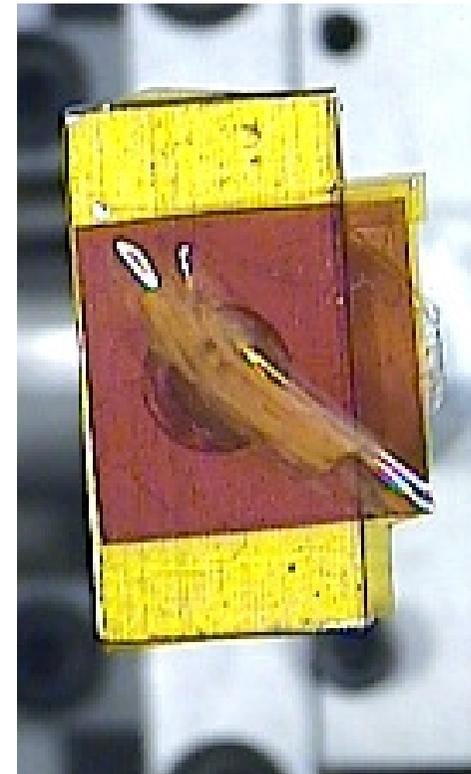
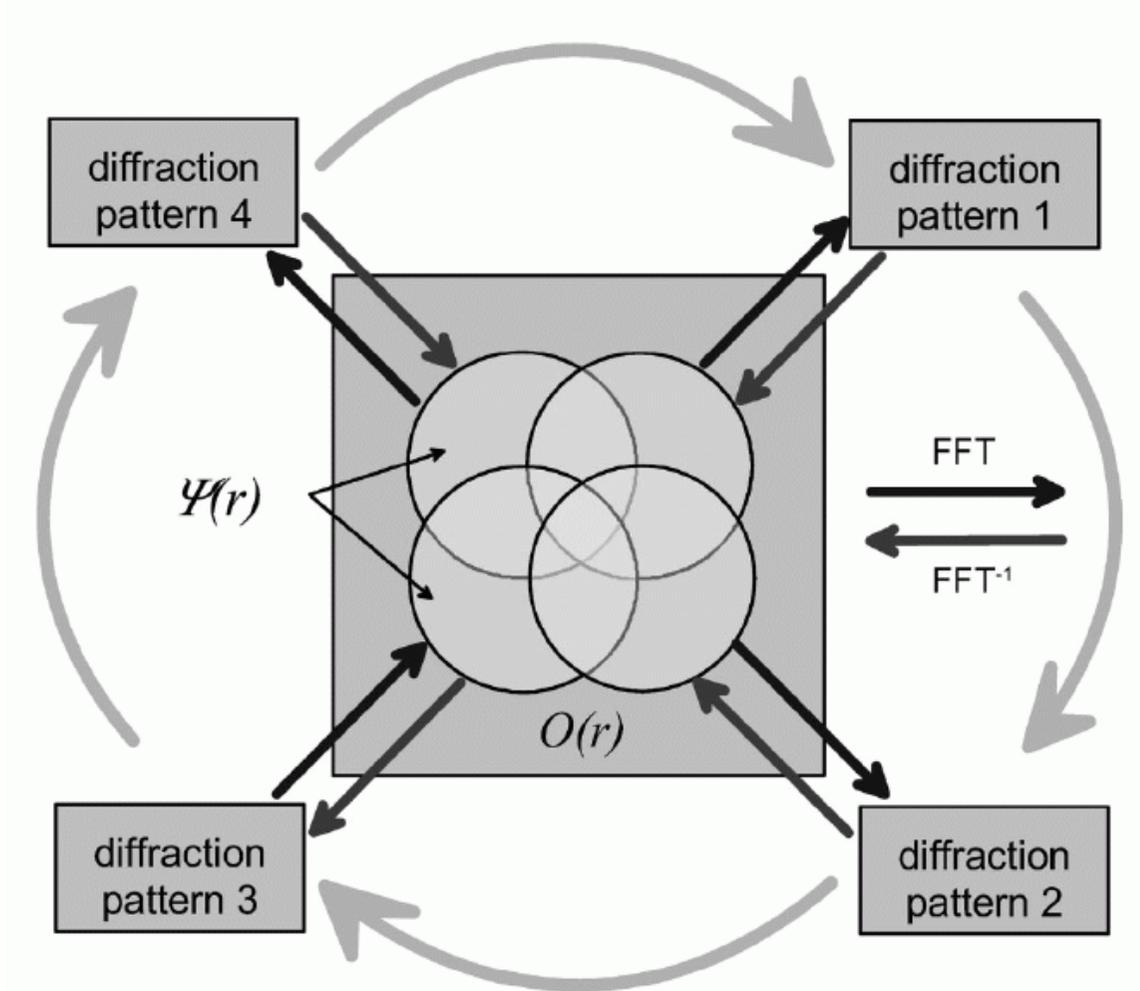


Strong D-banding with 67 nm periodicity:

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

I. K. Robinson, MPI Golm 2010

X-ray Ptychography

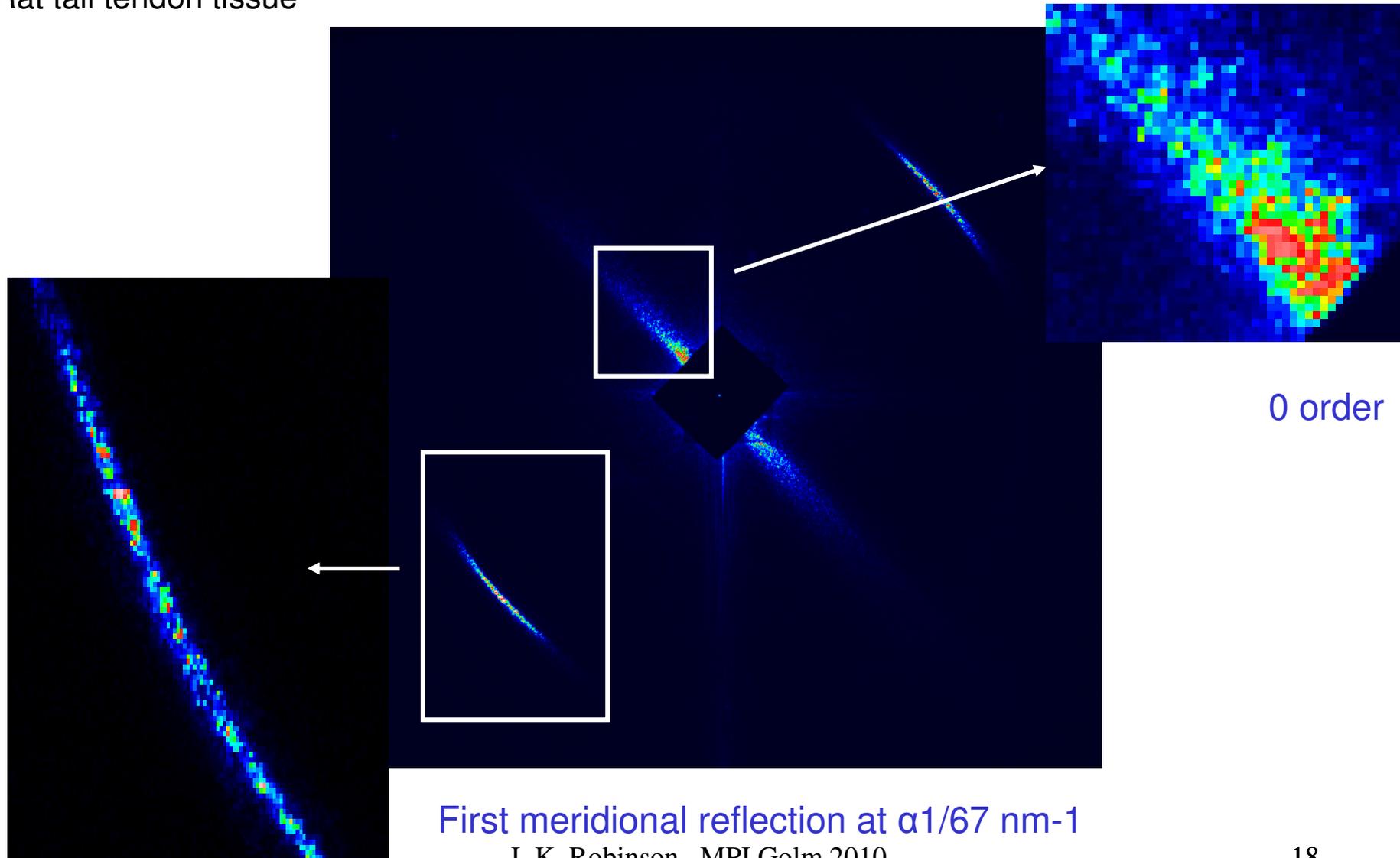


J. M. Rodenburg et al, Phys. Rev. Lett. 98 034801 (2007)

I. K. Robinson, MPI Golm 2010

First experimental results on collagen

Rat tail tendon tissue

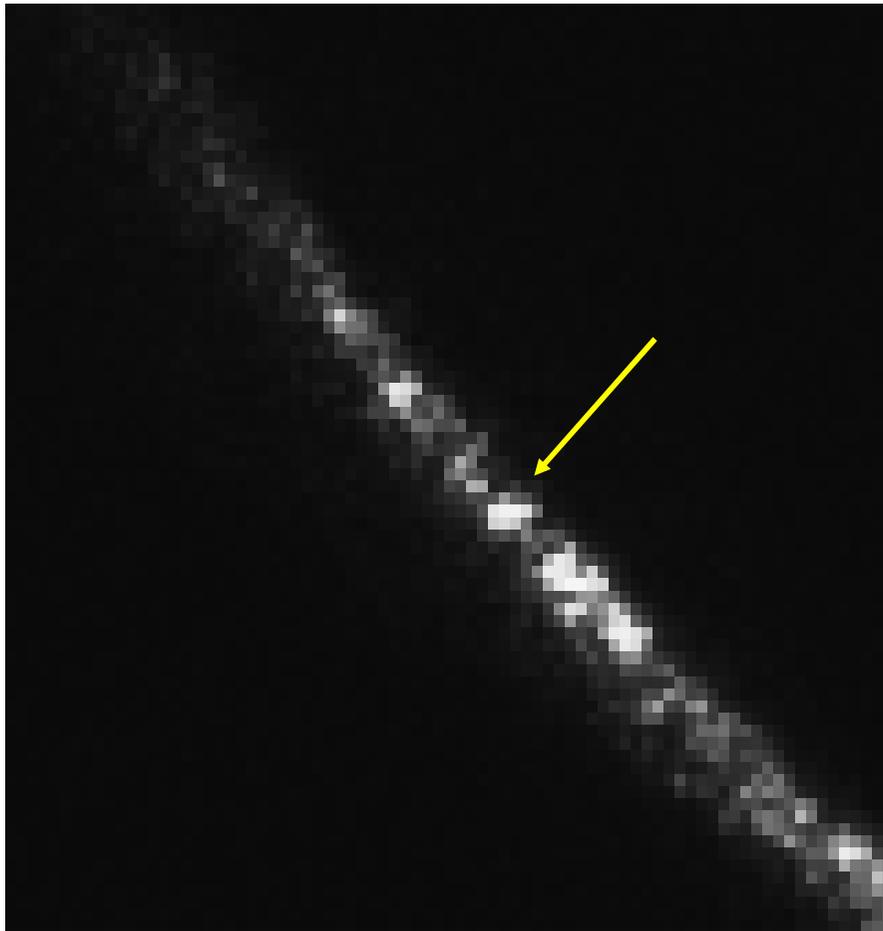


First meridional reflection at $\alpha 1/67 \text{ nm}^{-1}$

I. K. Robinson, MPI Golm 2010

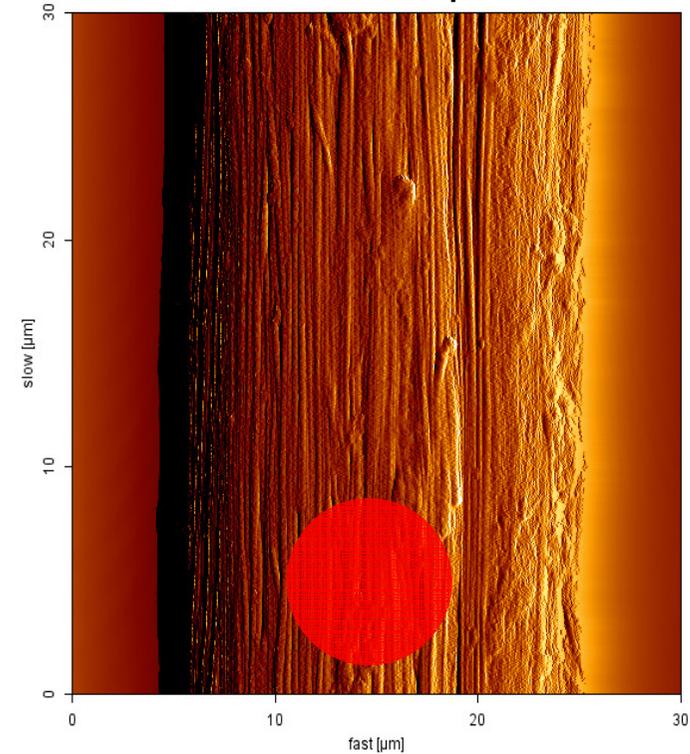
X-ray Ptychography

First meridional reflection



I. K. Robinson, MPI Golm 2010

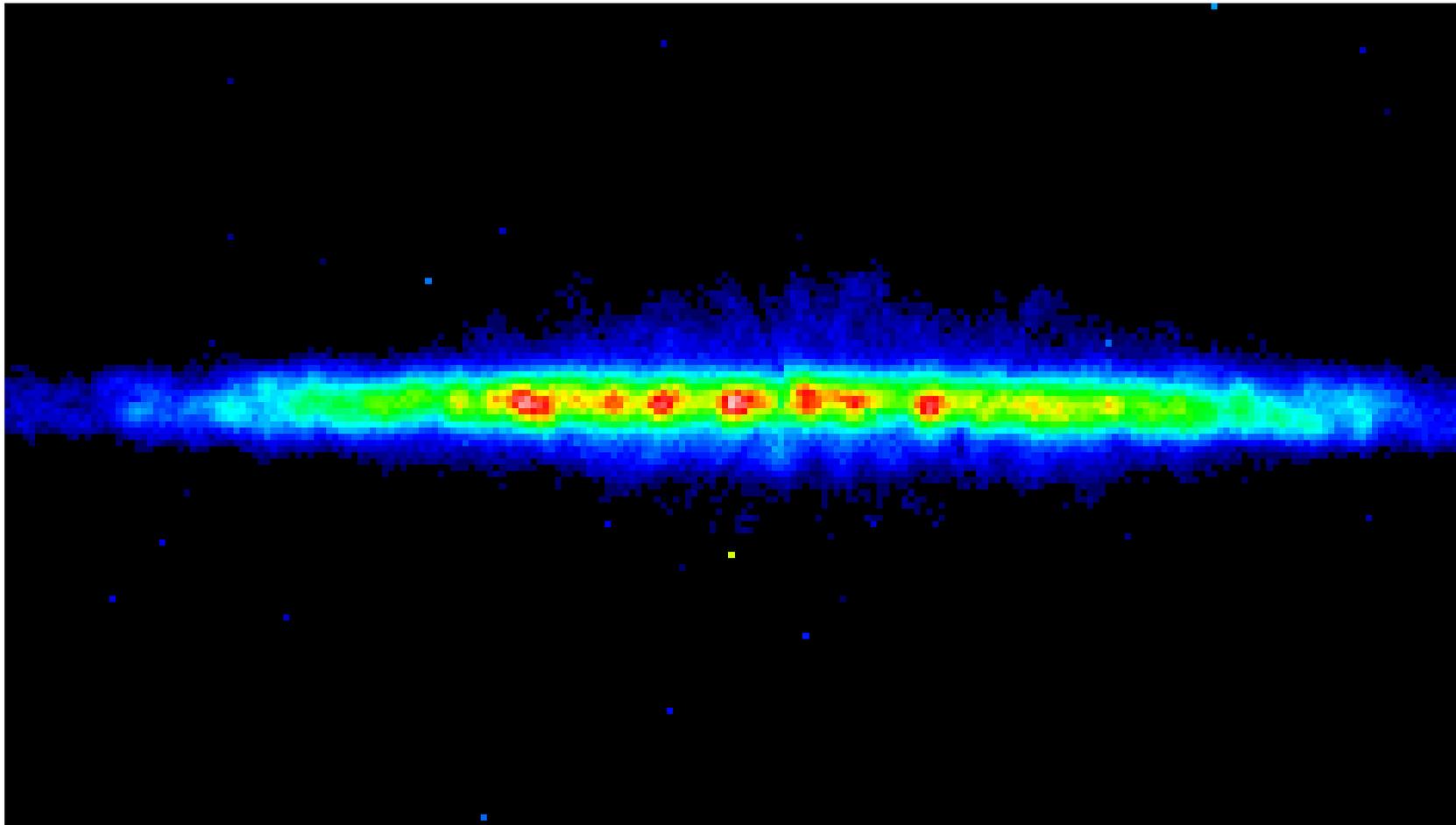
10 μm beam



Dark field imaging:
collagen distribution in different
tissues

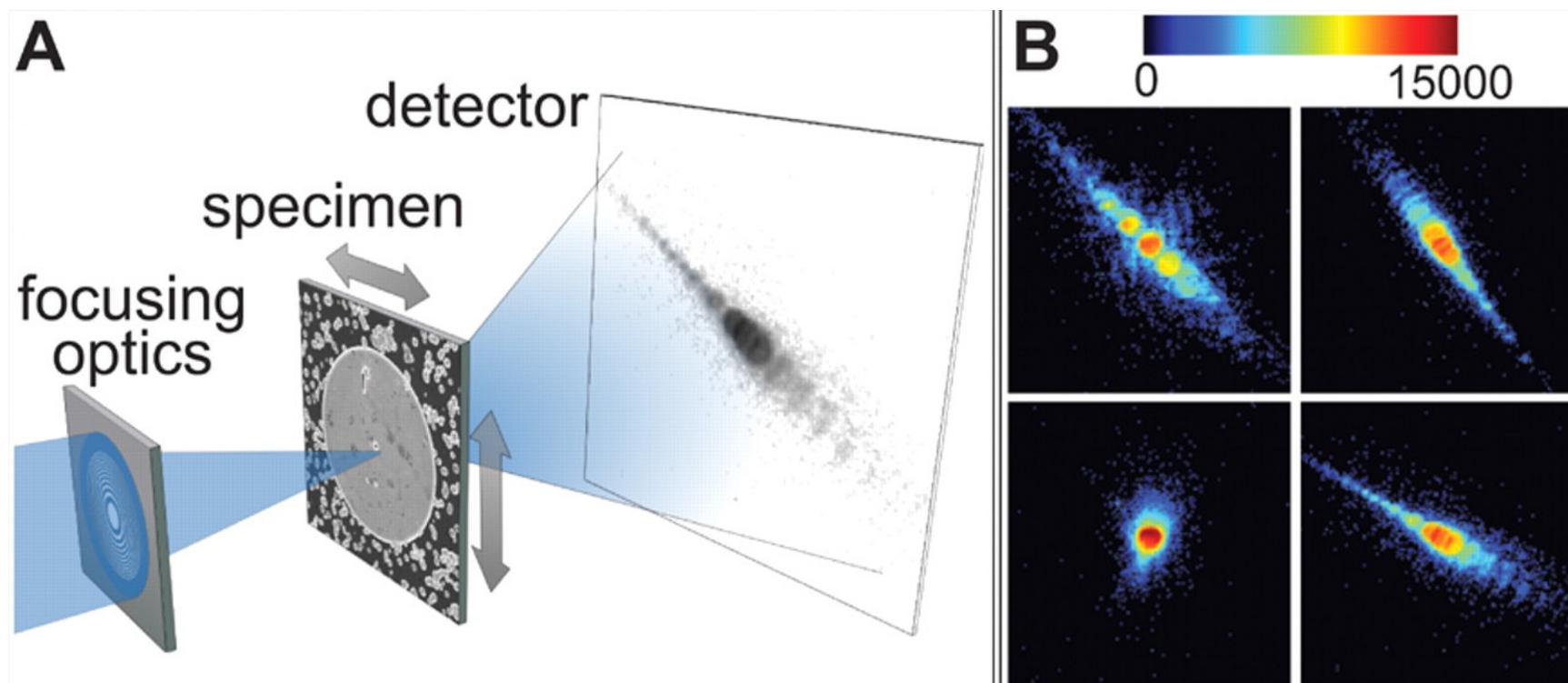
Improved collagen sample prep

Diamond I-22, Nov 2008



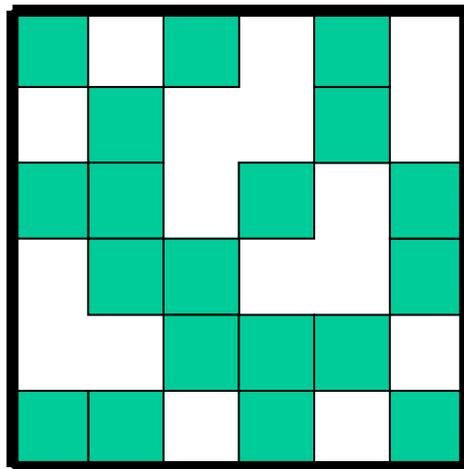
X-ray Ptychography

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

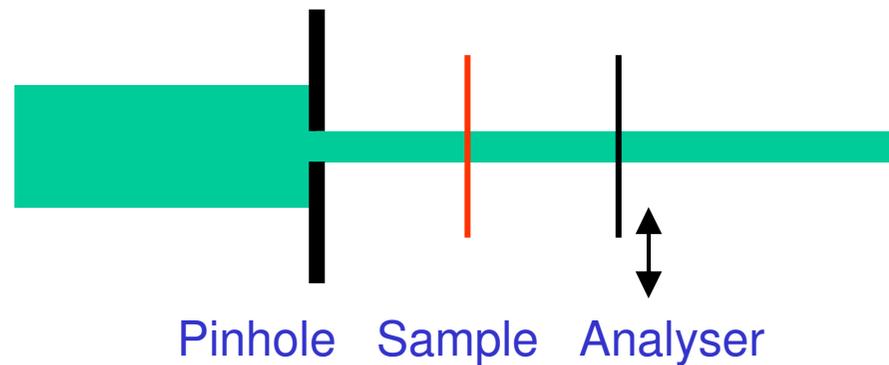


'Random' phase plate analyser

Joan Vila (PSI), Fucai Zhang (Sheffield)



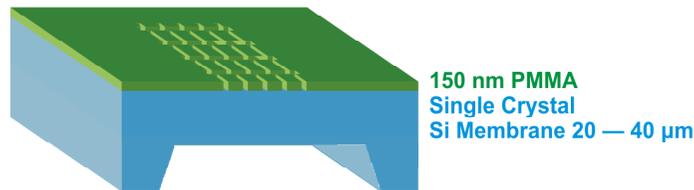
↔
1 μm



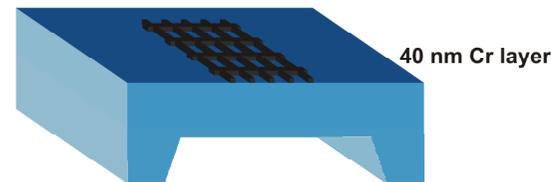
Phase plate fabrication

Joan Vila-Comamala, PSI

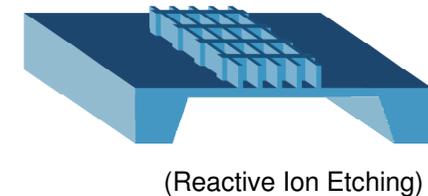
1) e-beam lithography



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

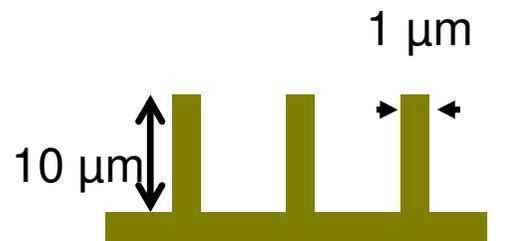


3) Pattern transfer to Si by RIE

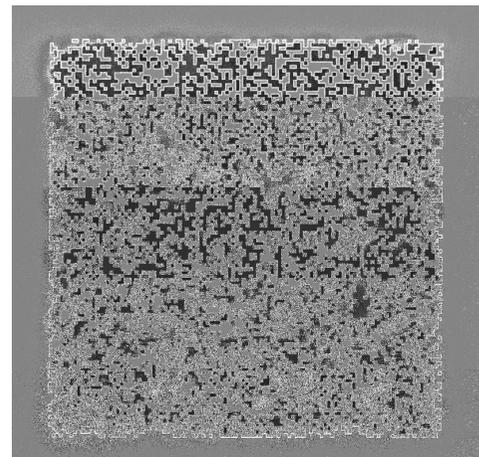


At 8.0 keV ($\lambda=0.155$ nm) $\rightarrow \delta = 7.67 \cdot 10^{-6}$ **$h=10.10$ μm**

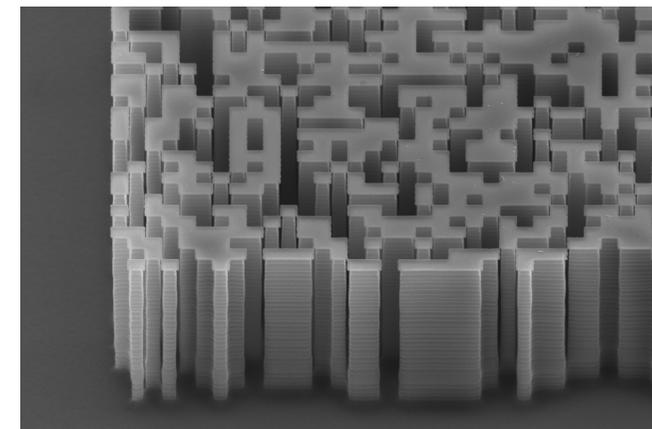
At 10.0 keV ($\lambda=0.124$ nm) $\rightarrow \delta = 4.88 \cdot 10^{-6}$ **$h=12.70$ μm**



Cross section



I. K. Robinson, MPI Golm 2010

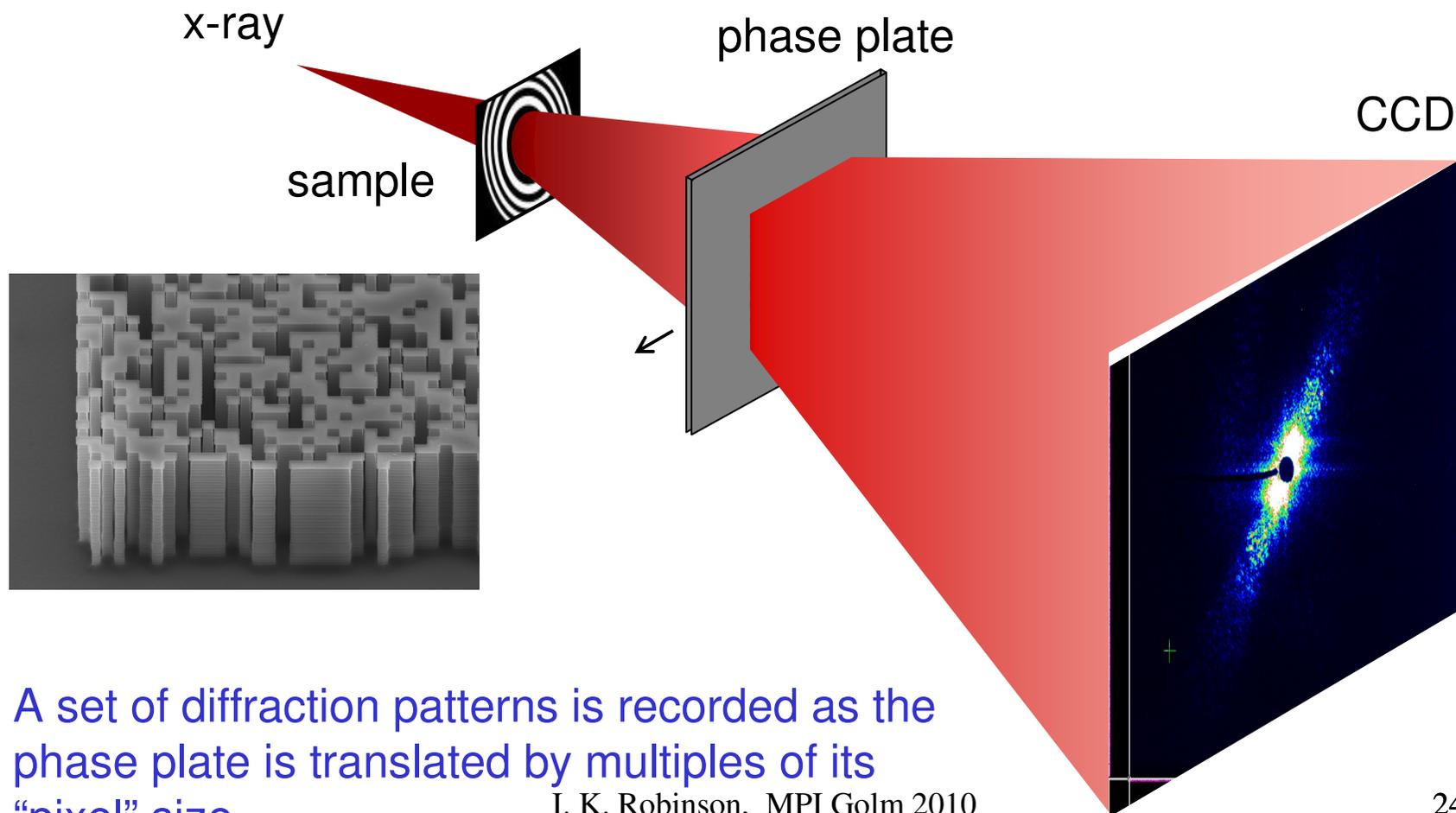


23

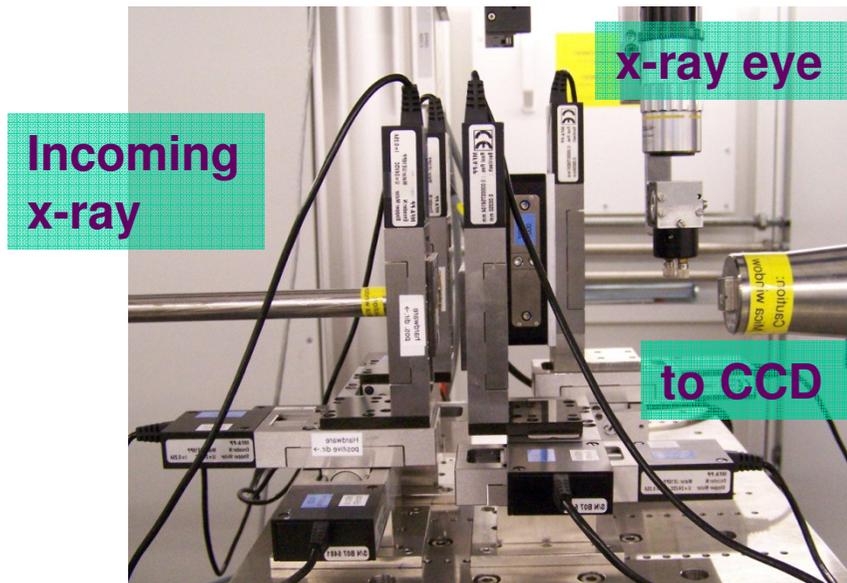
Imaging by wavefront modification

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

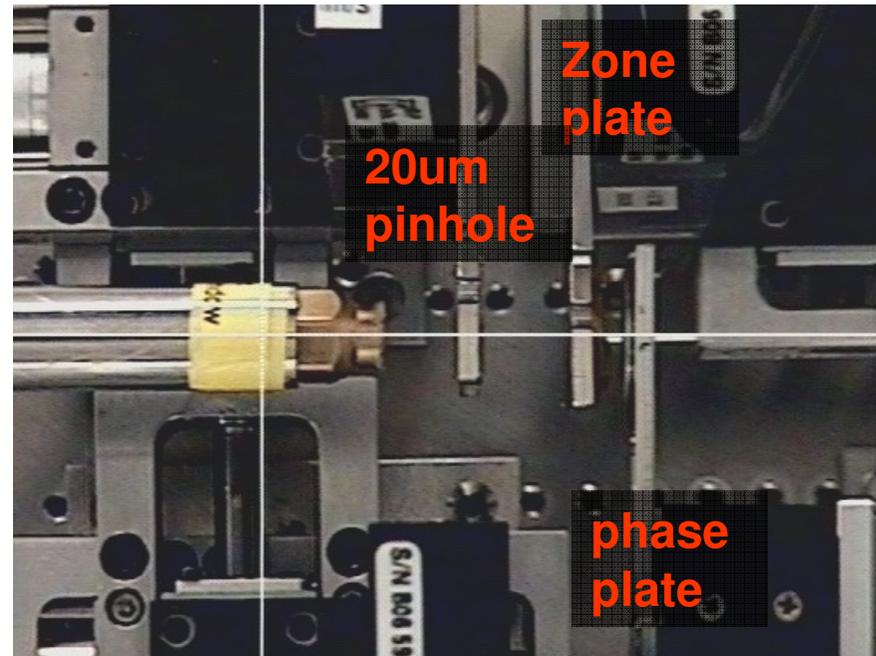
I. Johnson *et al.*, Phys Rev Lett 100 (2008)



Experimental setup cSAXS (SLS)



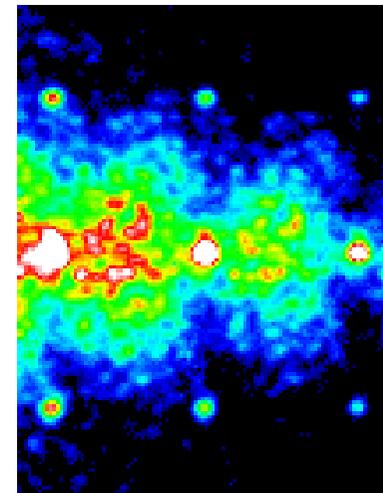
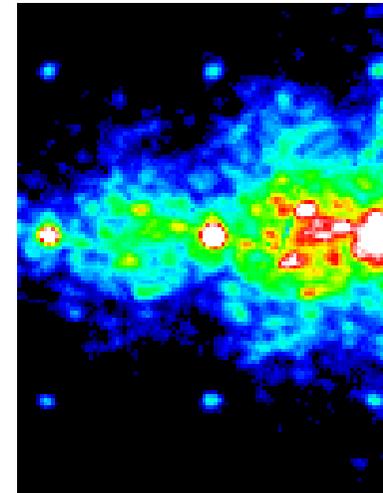
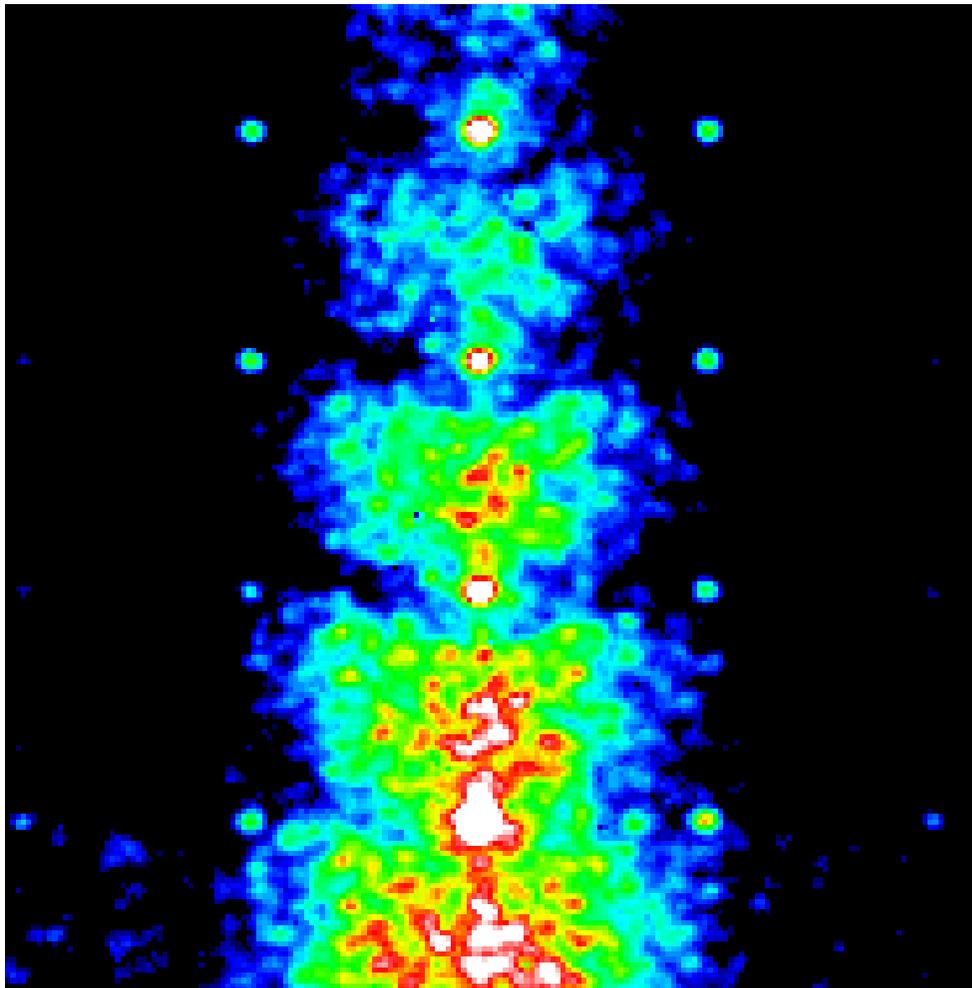
Side view



Top view (zoomed in)

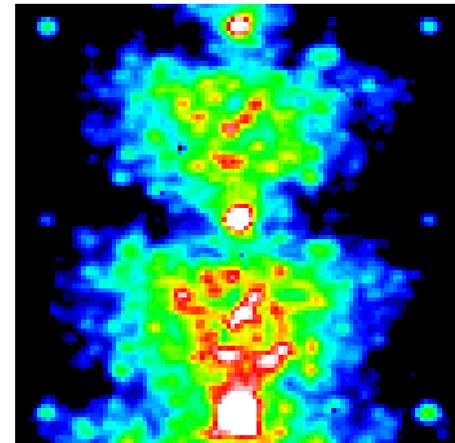
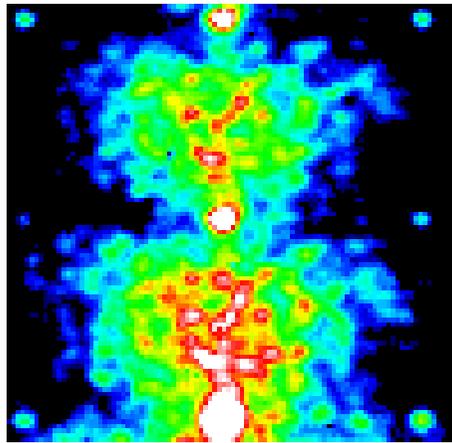
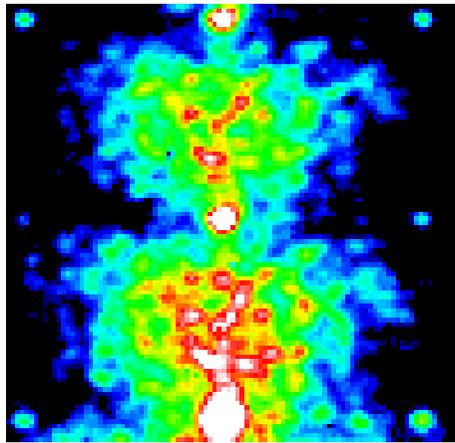
Phase plate scan near forward direction

1 μm step per frame

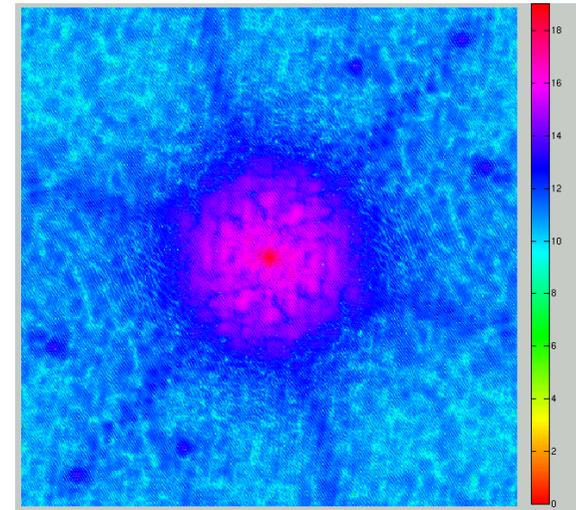
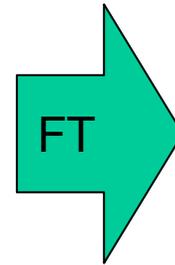
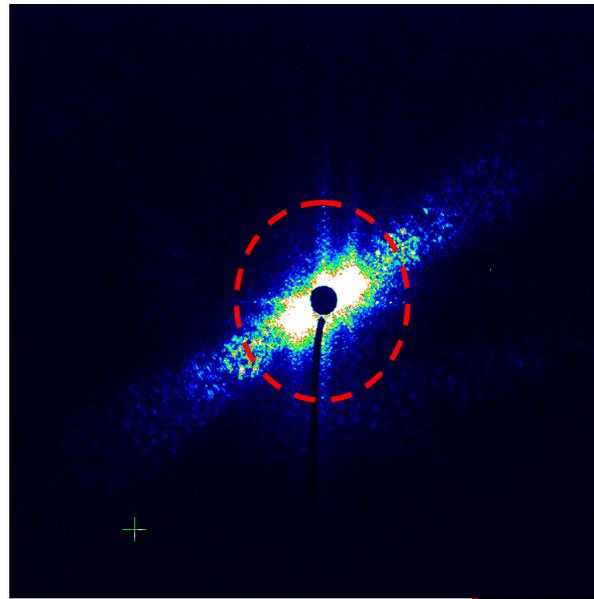


Collagen, buffer and empty cell

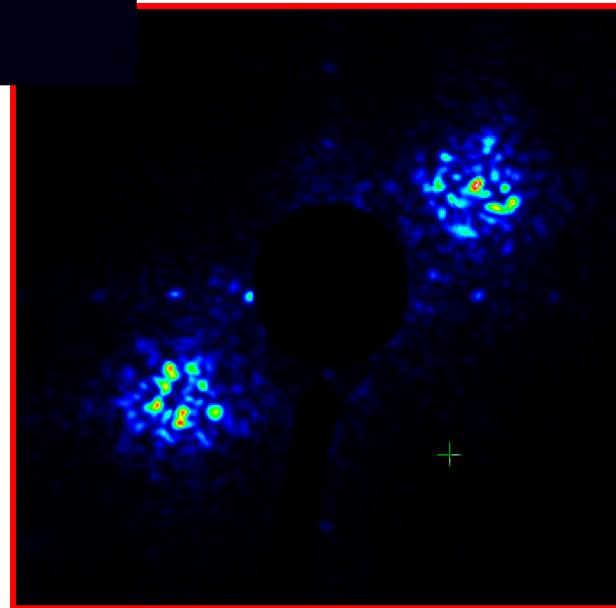
phplateFZP-236, -246 and -249



Results: Zone Plate as object

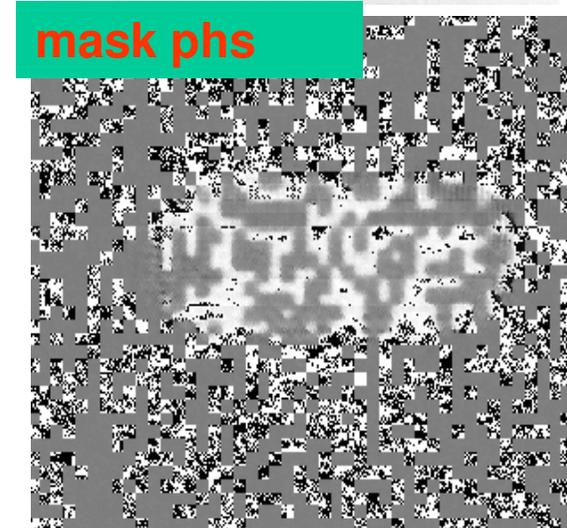
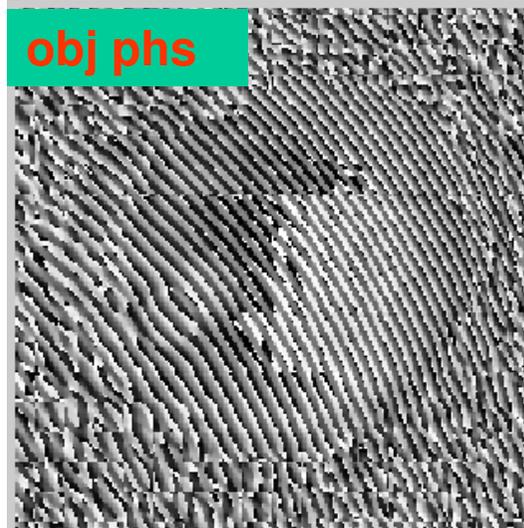
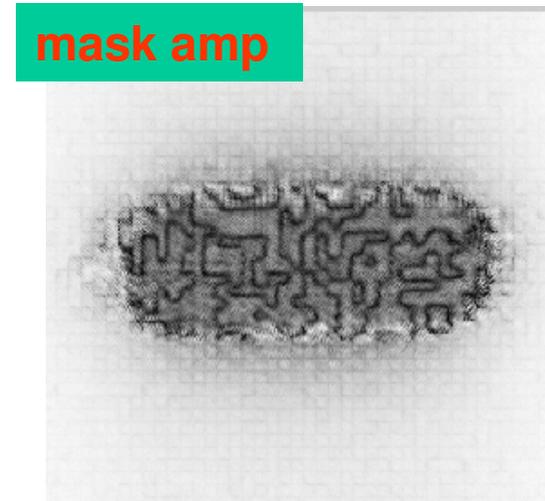
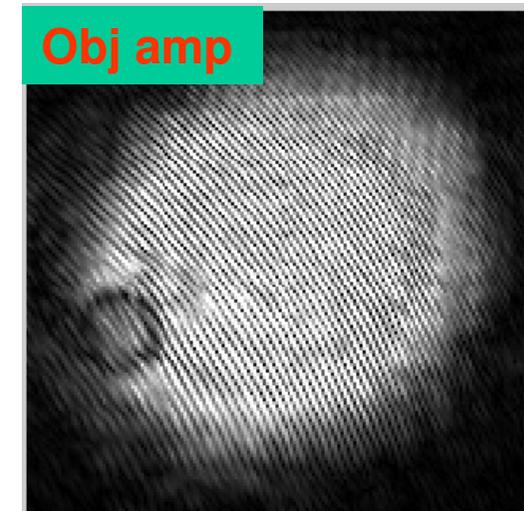


Auto correlation function



Reconstruction of Phase plate scan

1 μm step per frame (512x512)

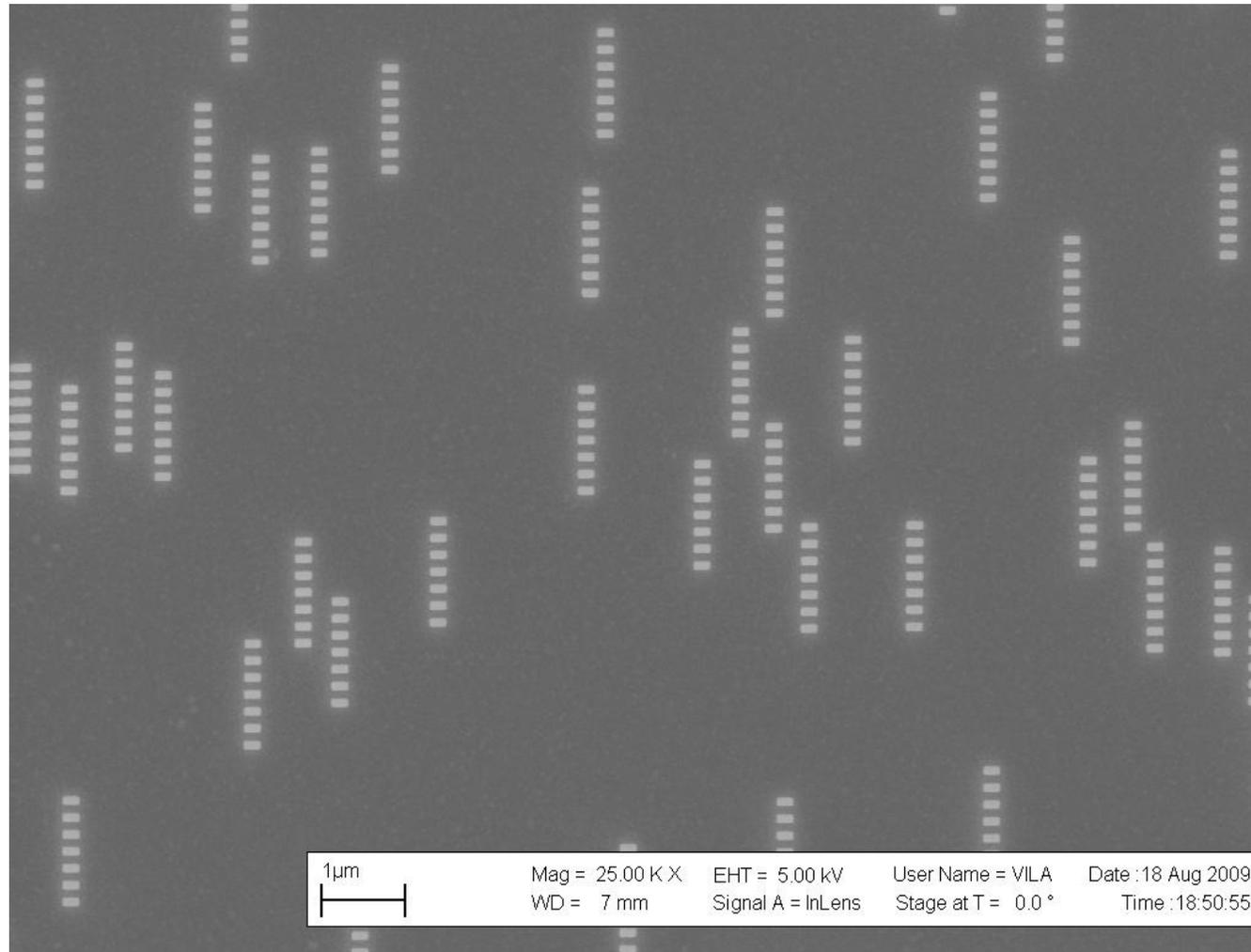


Reconstruction by F. Zhang (paper in preparation)

J. K. Robinson, MPI Golm 2010

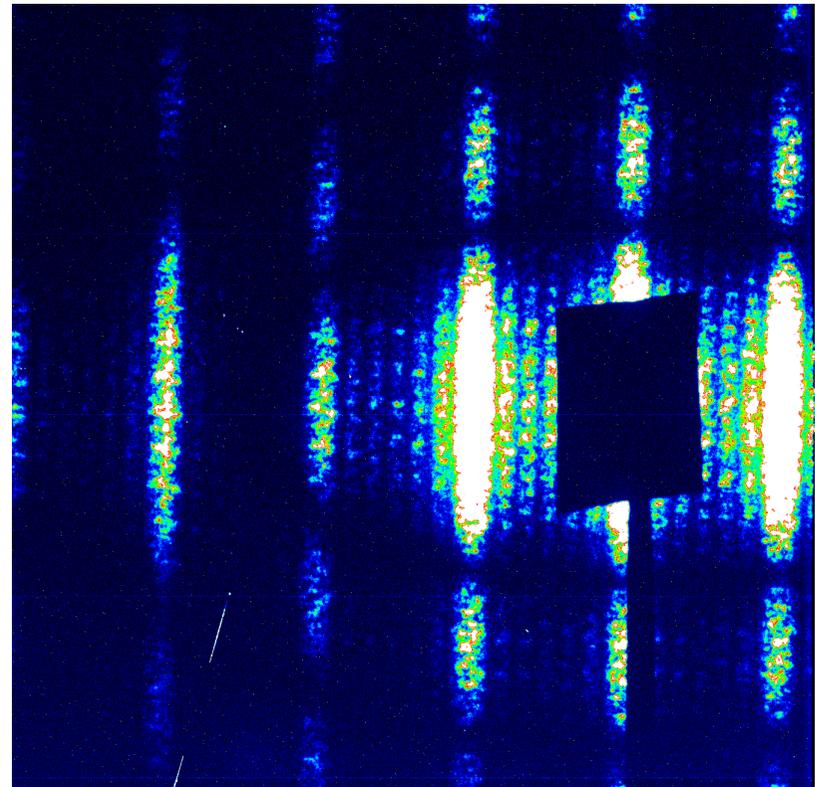
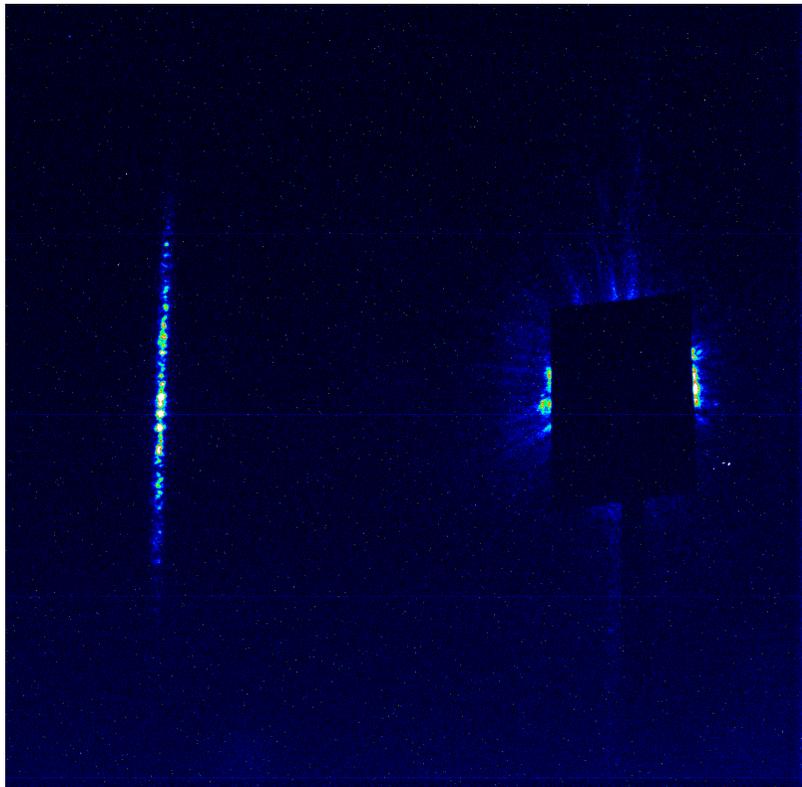
New phase plate design

R. Bean and J. Vila-Comamala, PSI

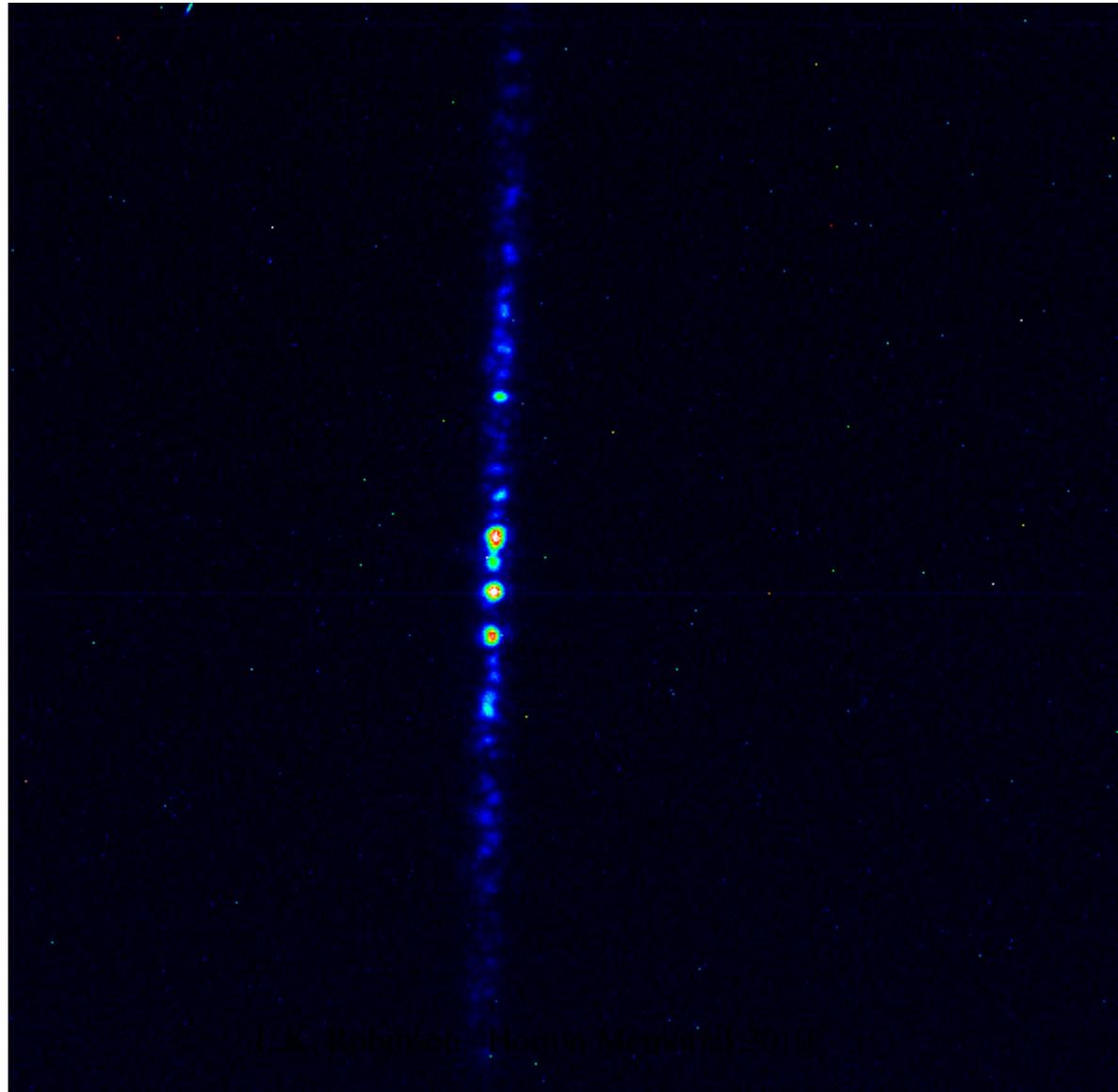


I. K. Robinson, MPI Golm 2010

Collagen in liquid cell +/- phase plate



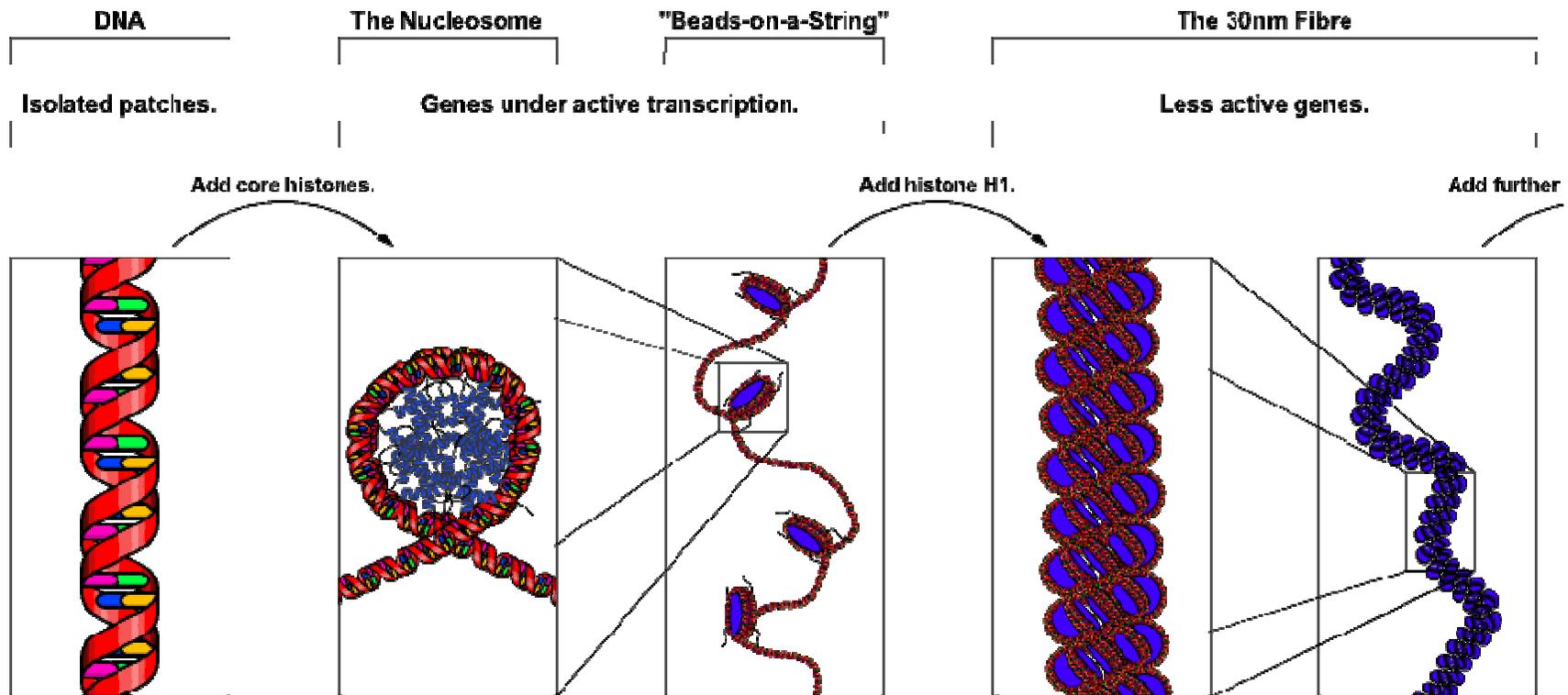
Sample+phase plate interference



J. K. Robinson, Honon Memorial 2010

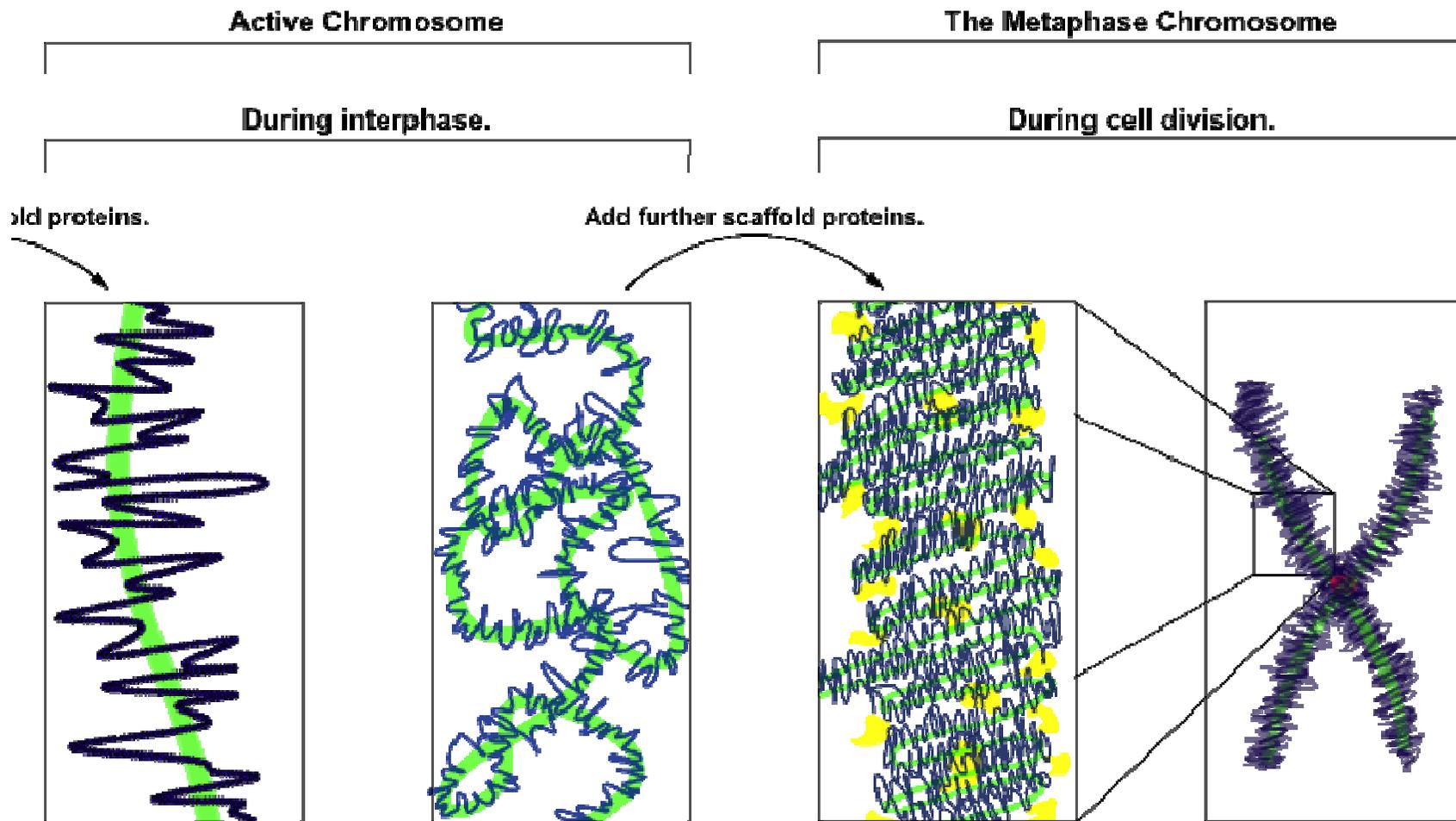
Chromosome structure I

a la wikipedia



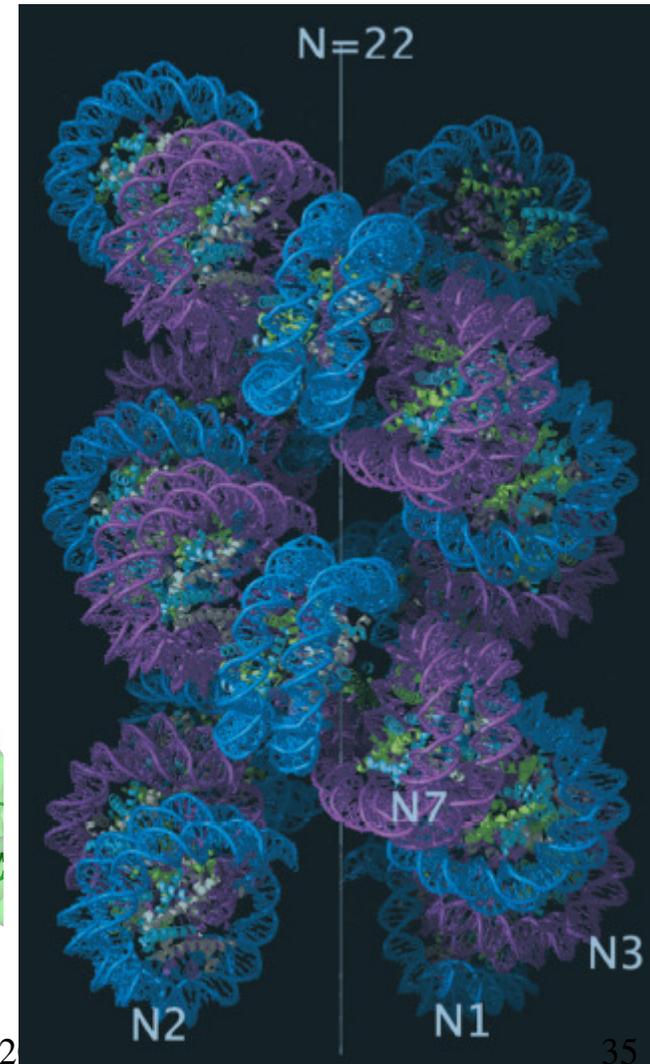
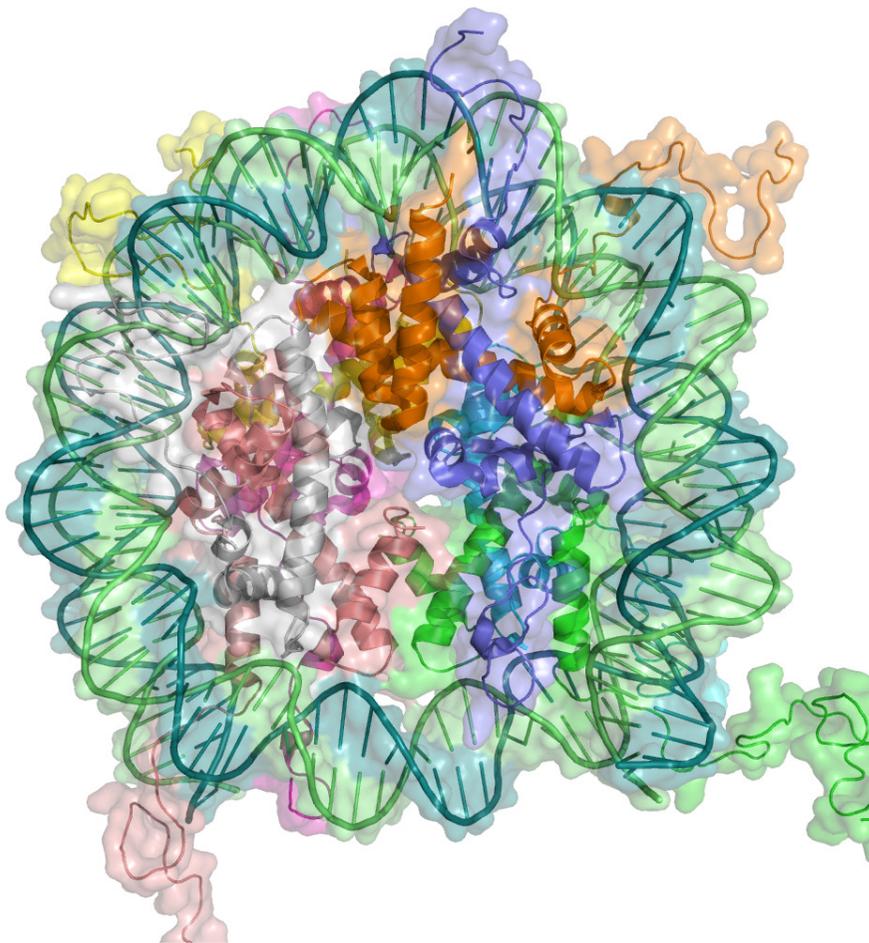
Chromosome structure II

a la wikipedia



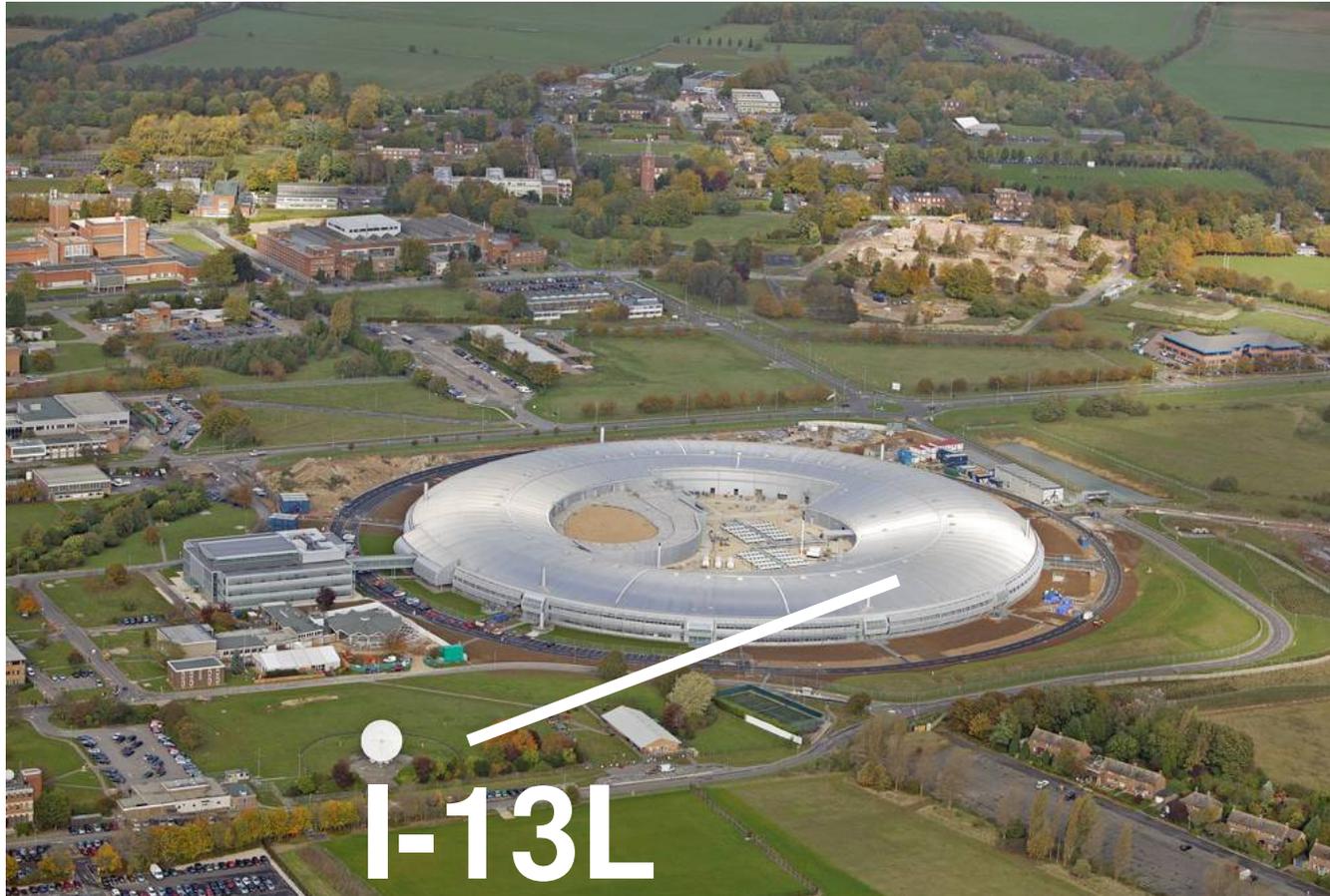
Chromosome structure III

D. Rhodes et al PNAS 103 6506 (2006)



I. K. Robinson, MPI Golm 2

Diamond Light Source (RAL)

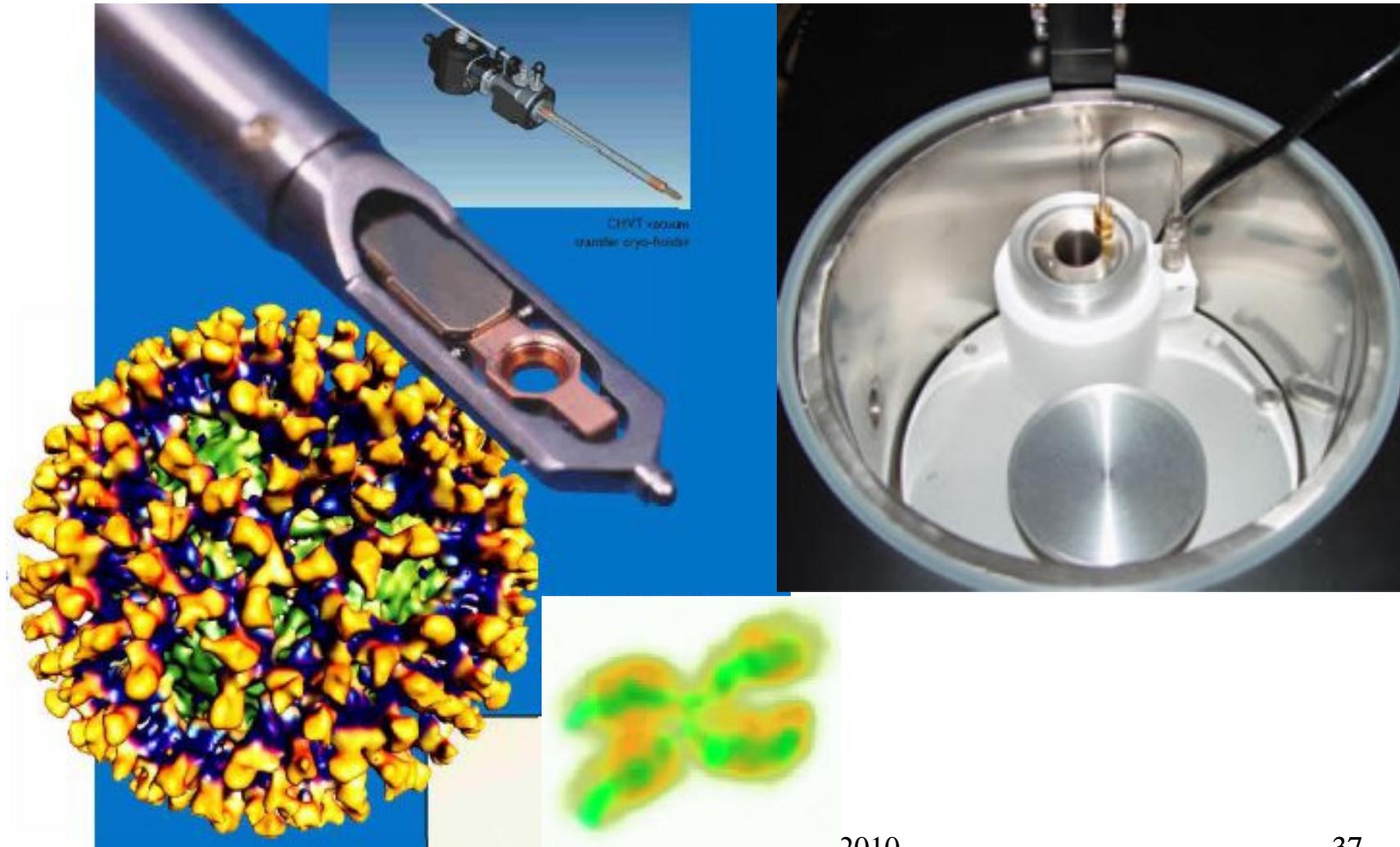


i-13L

I. K. Robinson, MPI Golm 2010

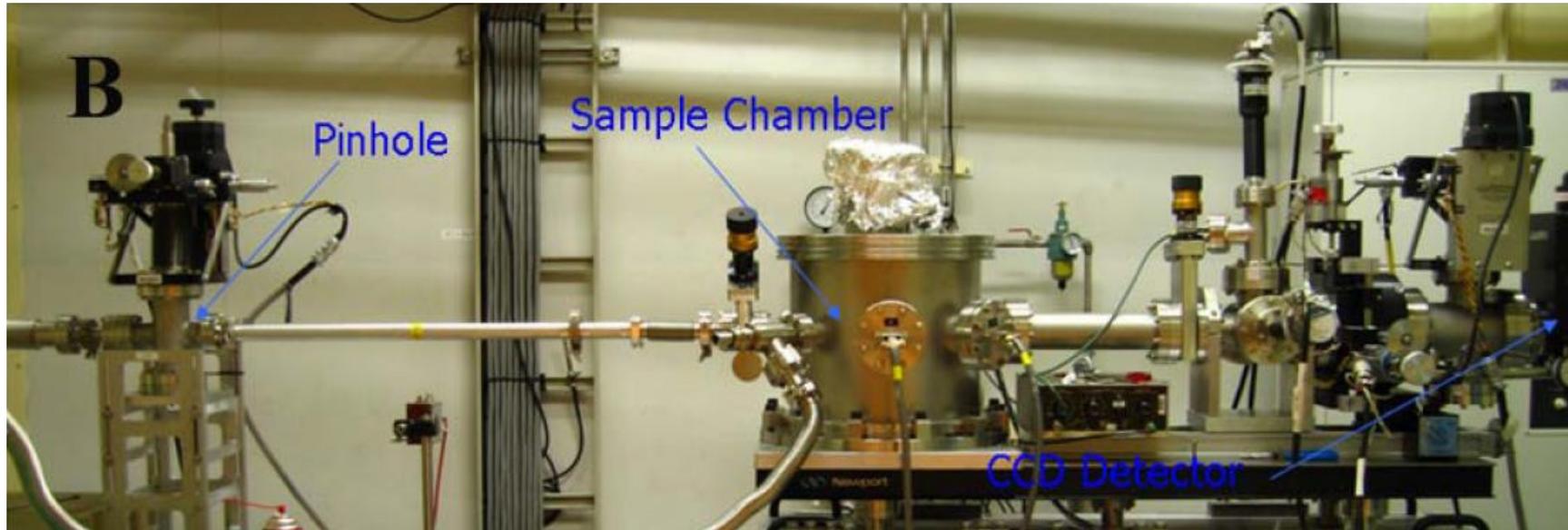
Cryotransfer system (Gatan)

Plunge cryo immersion (EMS)



Spring8 beamline BL29XUL

Nishino_BL29XUL.jpg



X ray energy 5 keV through a 20 μ m pinhole 1x10¹⁰ ph/sec.

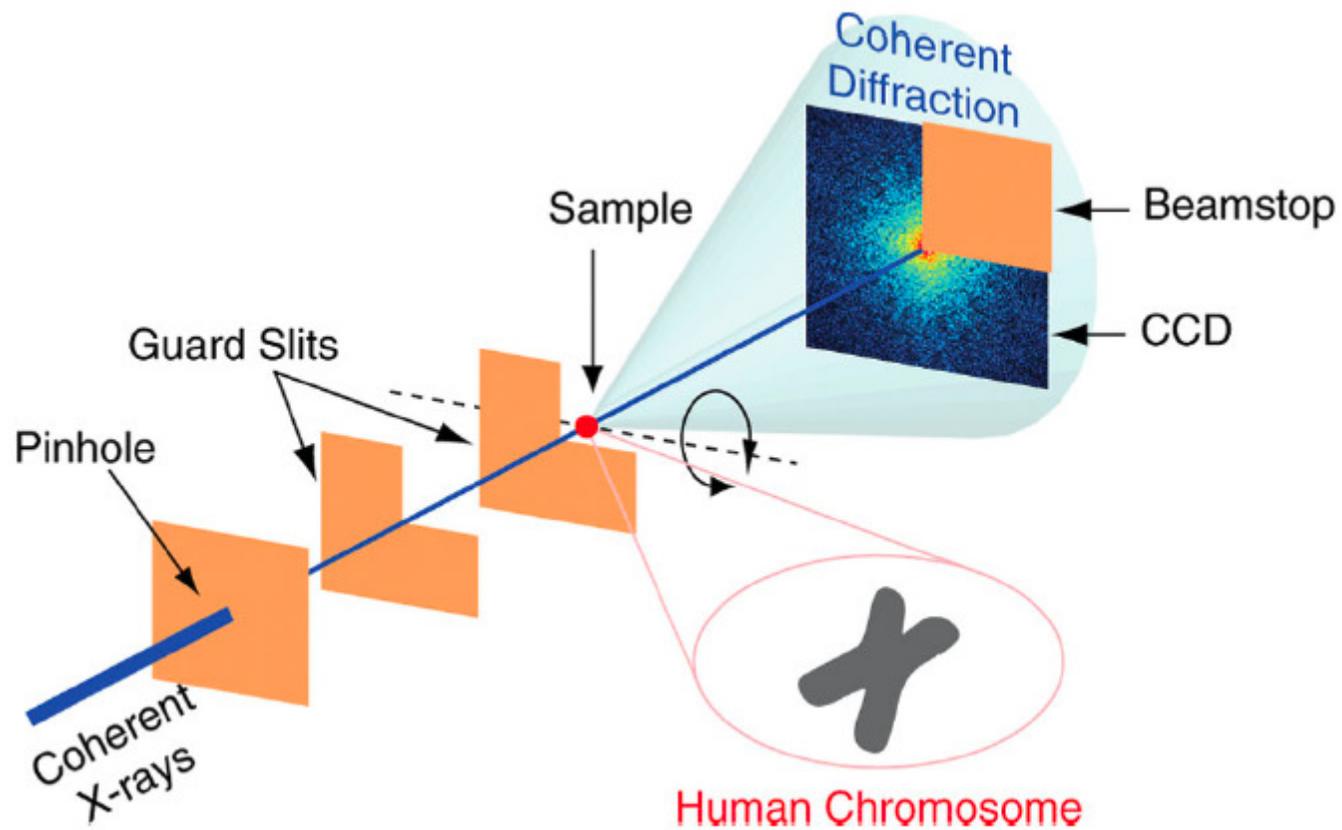
CCD pixel size 20 μ m, 1340x1300 pixels at 1.3m

Exposure time 2700 s

Radiation dose 4x10⁸ Gy (2x10¹⁰ Gy for full 3D)

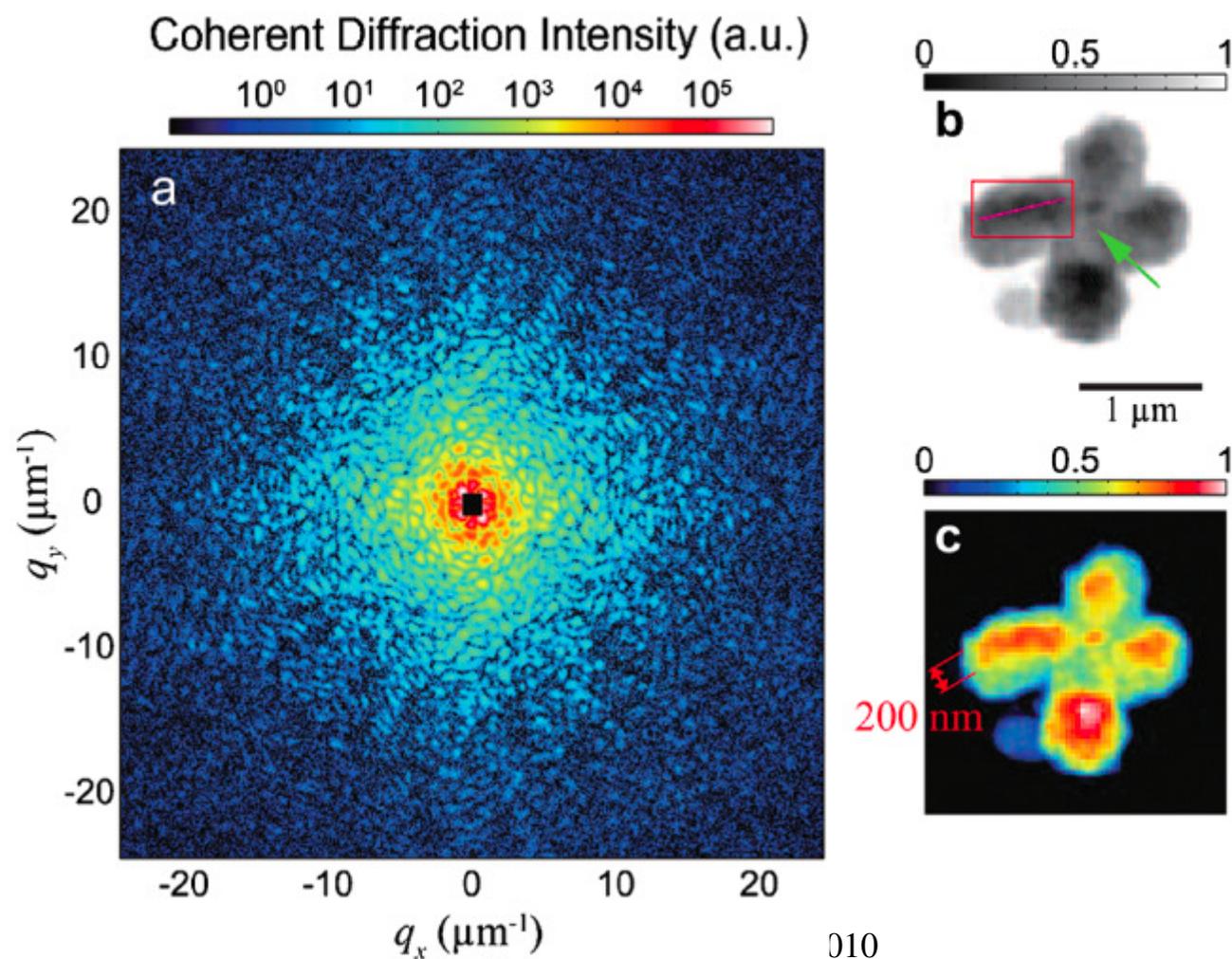
Schematic in-line CDI setup

Y. Nishino et al PRL 102, 018101 (2009)



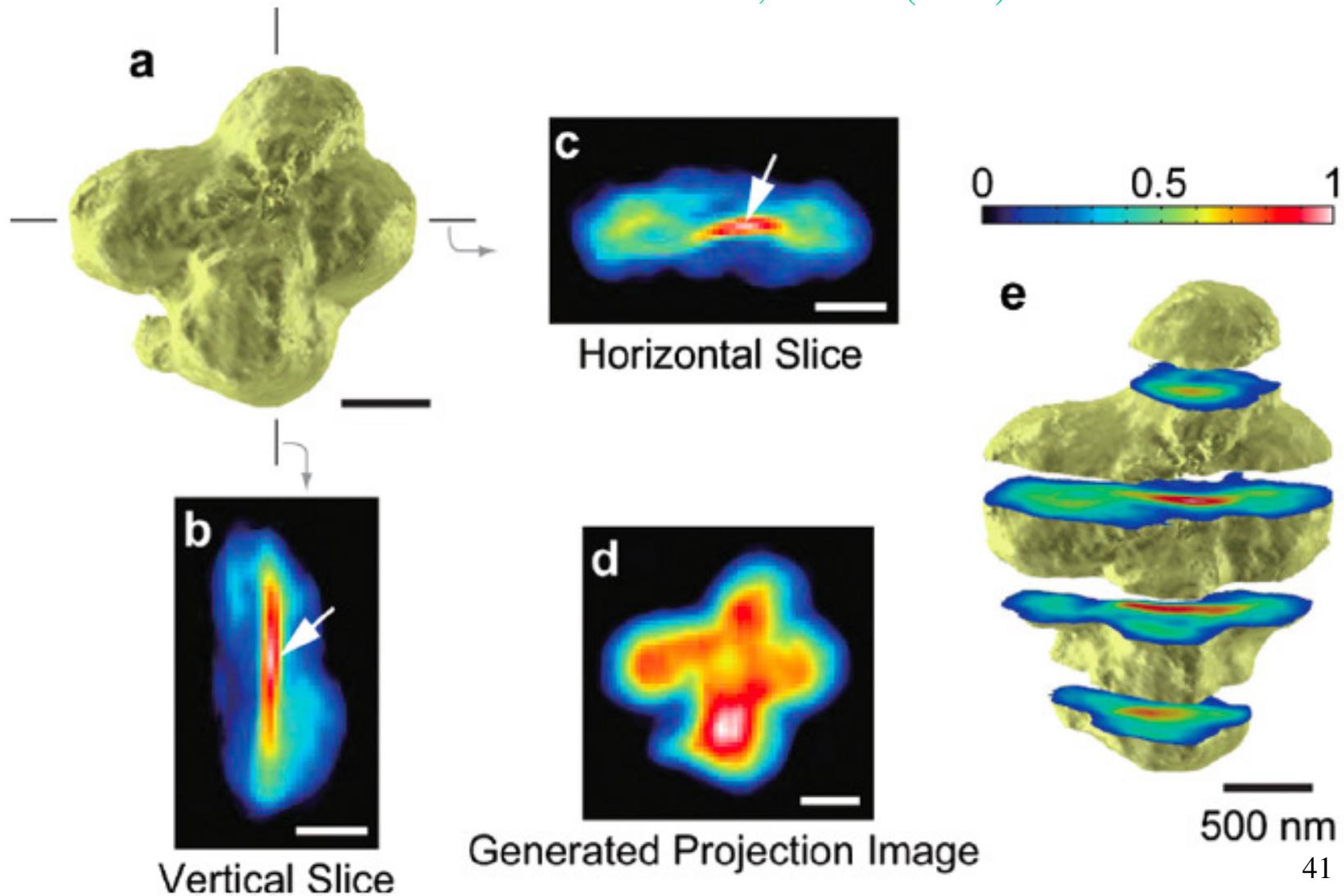
Chromosome diffraction pattern

Y. Nishino et al PRL 102, 018101 (2009)

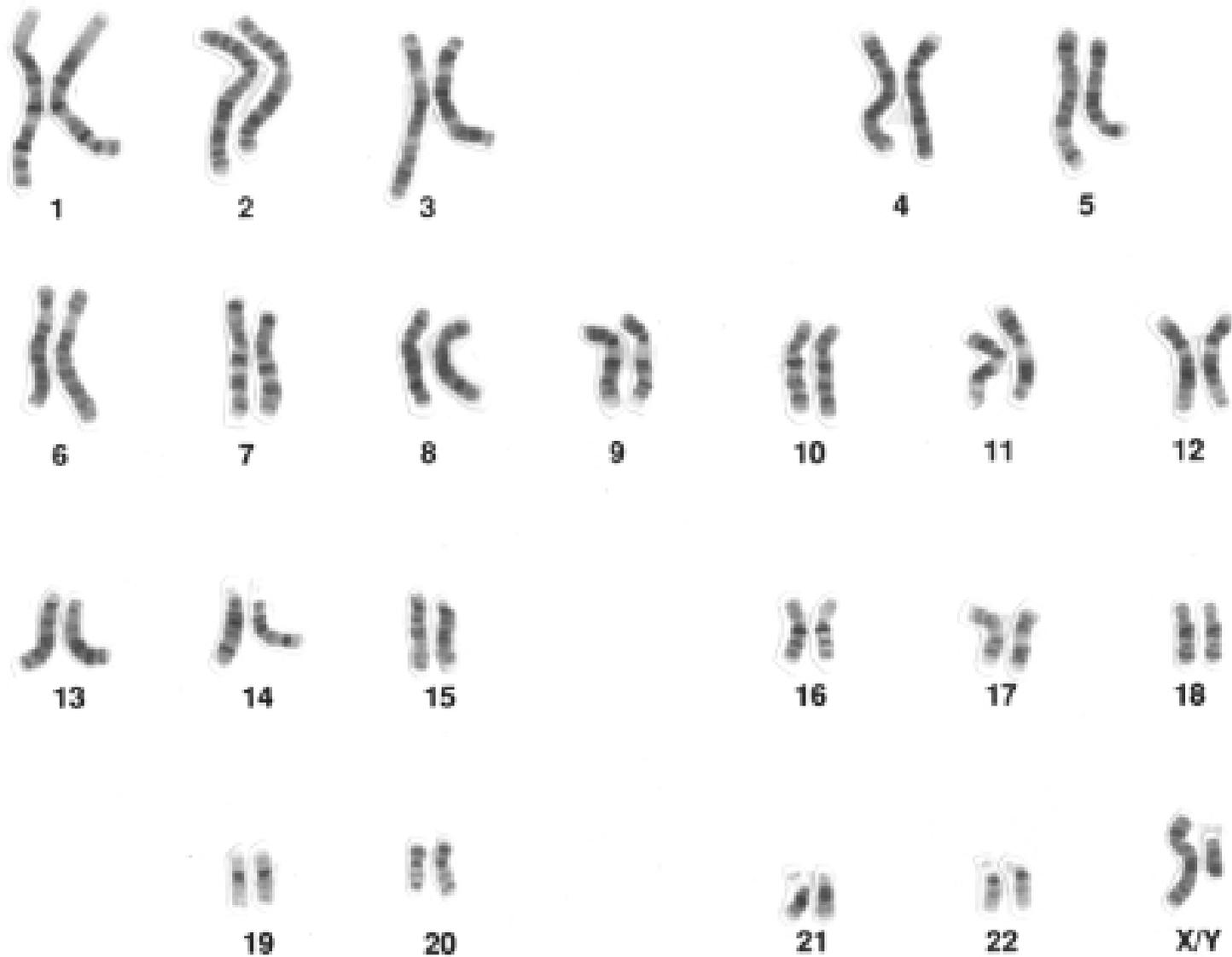


Chromosome 3D CDI result

Y. Nishino et al PRL 102, 018101 (2009)



The Human Karyotype



Conclusions

- X-ray 3D imaging on the nanoscale
- Phasing by computation instead of lens
- Biological tissue accessible by ptychography
- New phase-plate methods
- Future application to chromosome structure