The Abdus Salam
International Centre for Theoretical Physics

United Nations
Educational, Scientific and
Cultural Organization

IAEA
International Atomic Energy Agency

2139-31

School on Synchrotron and Free-Electron-Laser Sources and their Multidisciplinary Applications

26 April - 7 May, 2010

Diffraction Microscopy using synchrotrons

J. Kirz
State University of New York
USA

Diffraction Microscopy using synchrotrons

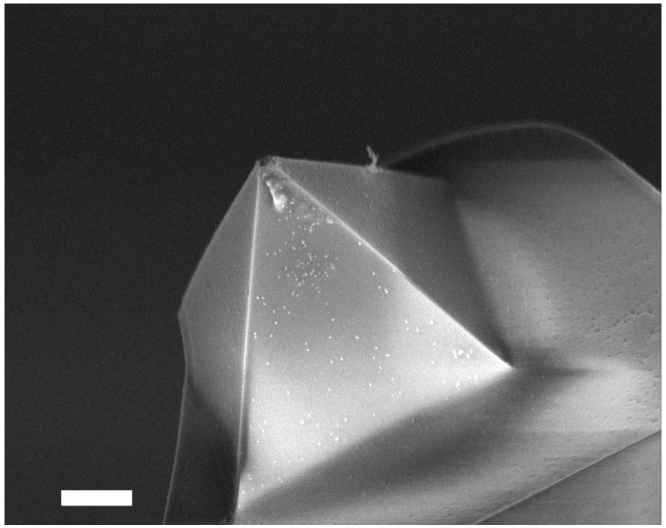
Janos Kirz
ALS

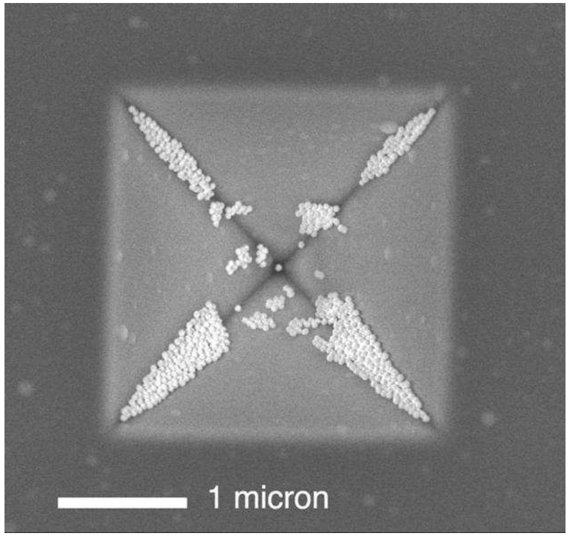
Outline lecture 2

- “Conventional” CDI
- FTH: Eisebitt, Schlotter, Marchesini
- New ideas: Spence (serial crystallography)
- New ideas: Nugent et al.
- New ideas: Rodenburg, Pfeiffer

3D diffraction microscopy of materials science specimens

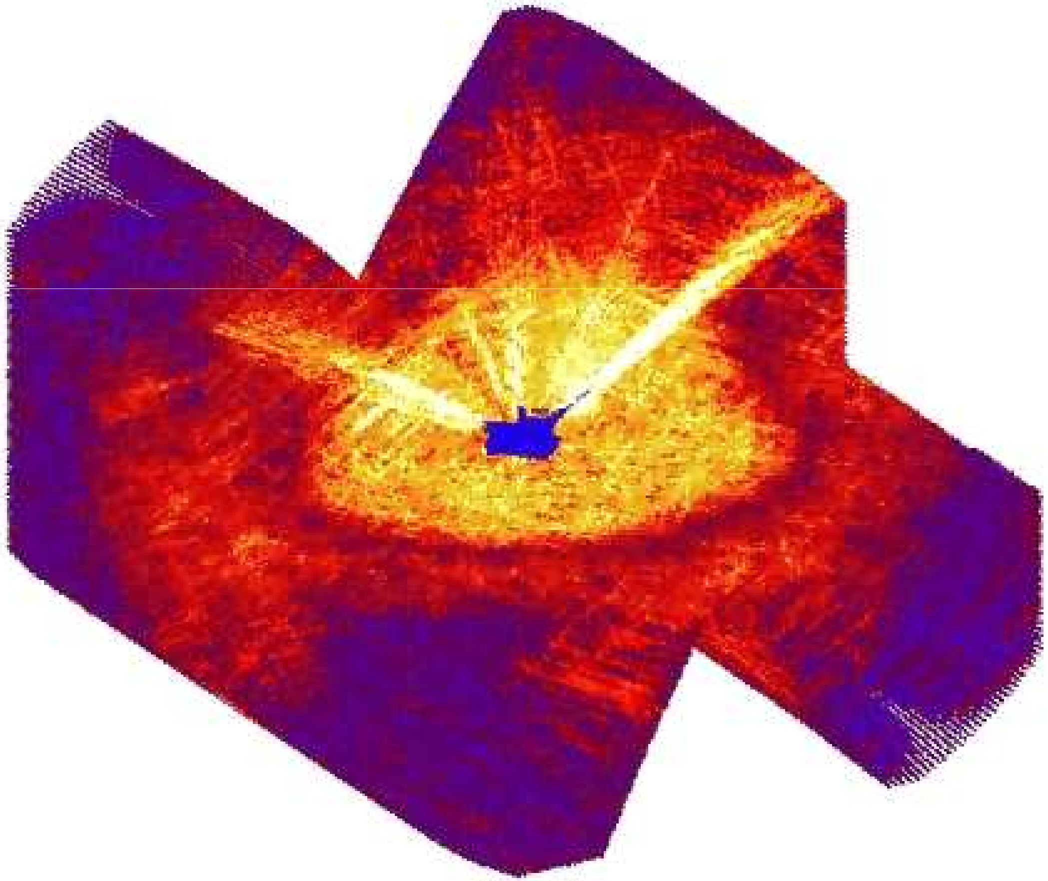
- Chapman, Barty, Marchesini, Noy, Hau-Riege, Cui, Howells, Rosen, He, Spence, Weierstall, Beetz, Jacobsen, Shapiro, *J. Opt. Soc. Am. A* **23**, 1179 (2006)
- 50 nm gold spheres placed on hollowed AFM tip “pyramid”
- Data taken using Stony Brook apparatus at ALS beamline 9.0.1





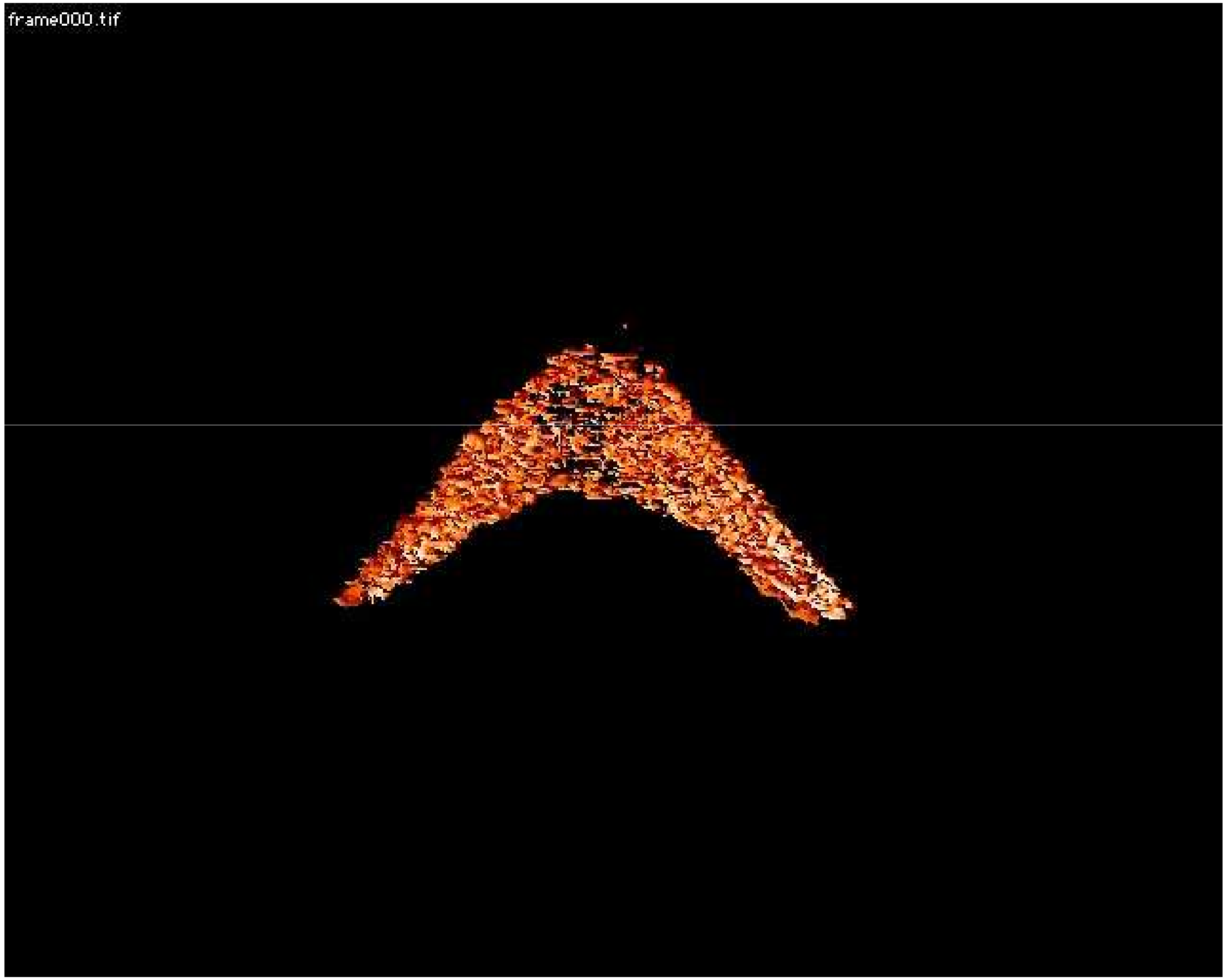
3D data cube

•Chapman, Barty, Marchesini, Noy, Hau-Riege, Cui, Howells, Rosen, He, Spence, Weierstall, Beetz, Jacobsen, Shapiro, *J. Opt. Soc. Am. A* **23**, 1179 (2006)



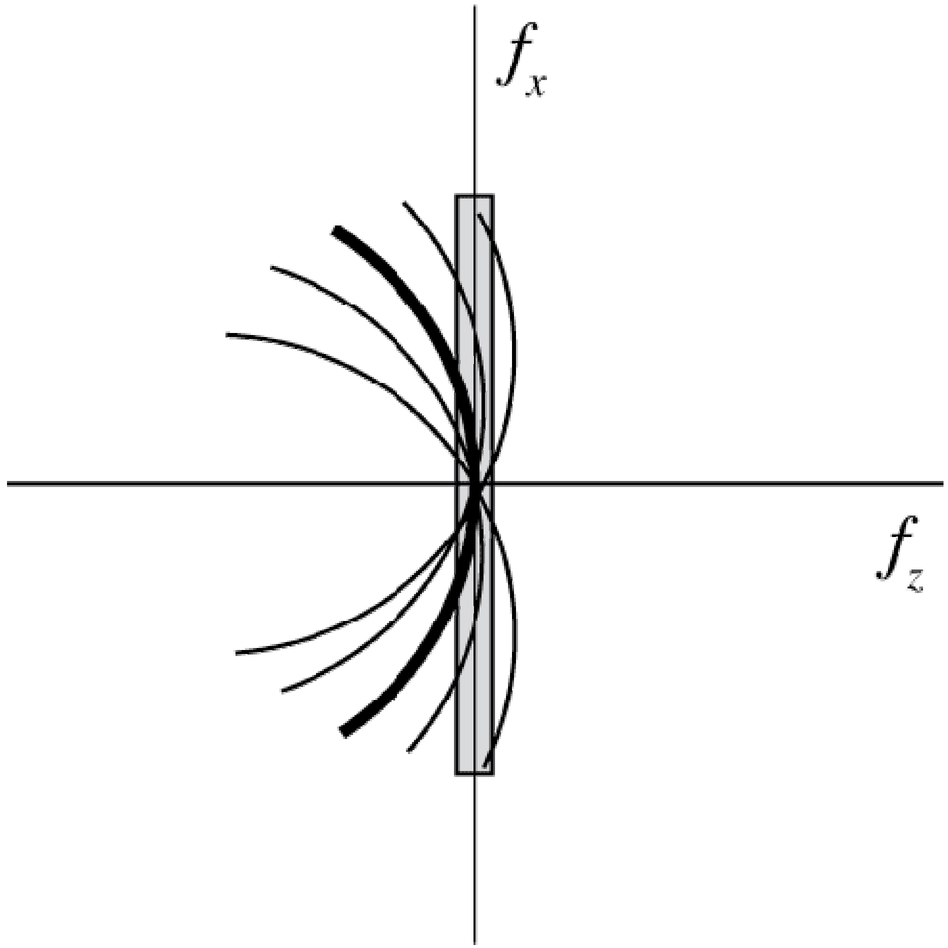
Reconstruction

•Chapman, Barty, Marchesini, Noy, Hau-Riege, Cui, Howells, Rosen, He, Spence, Weierstall, Beetz, Jacobsen, Shapiro, *J. Opt. Soc. Am. A* **23**, 1179 (2006)

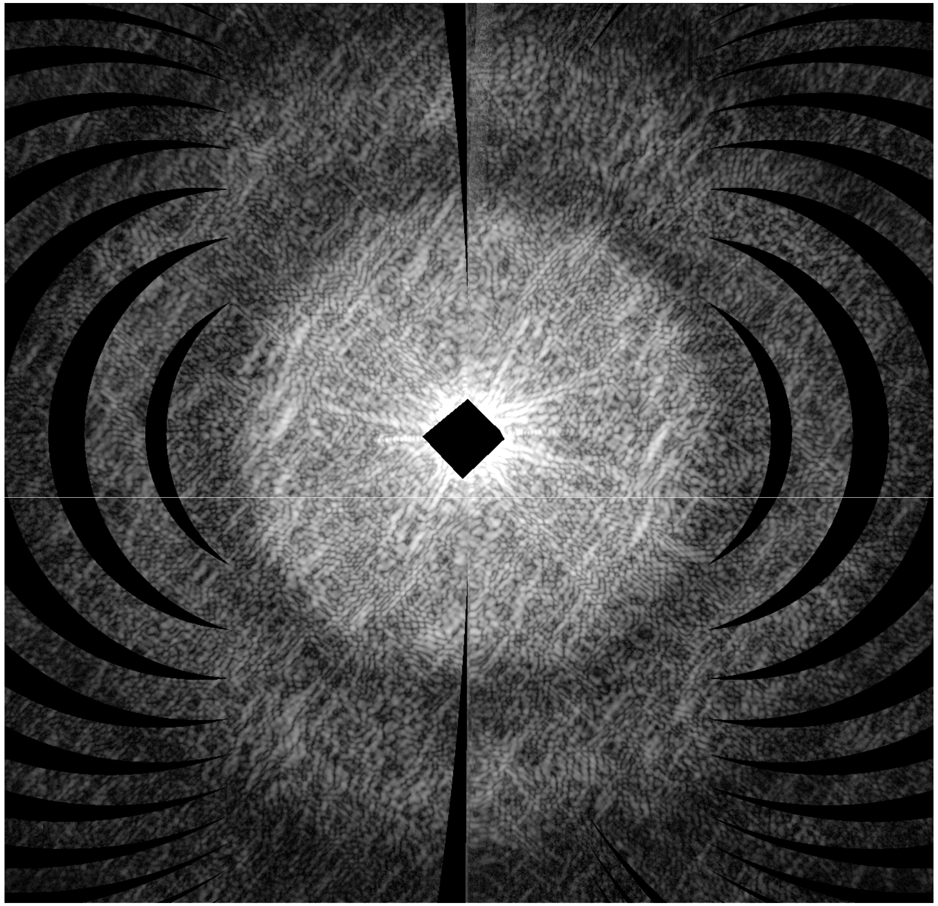


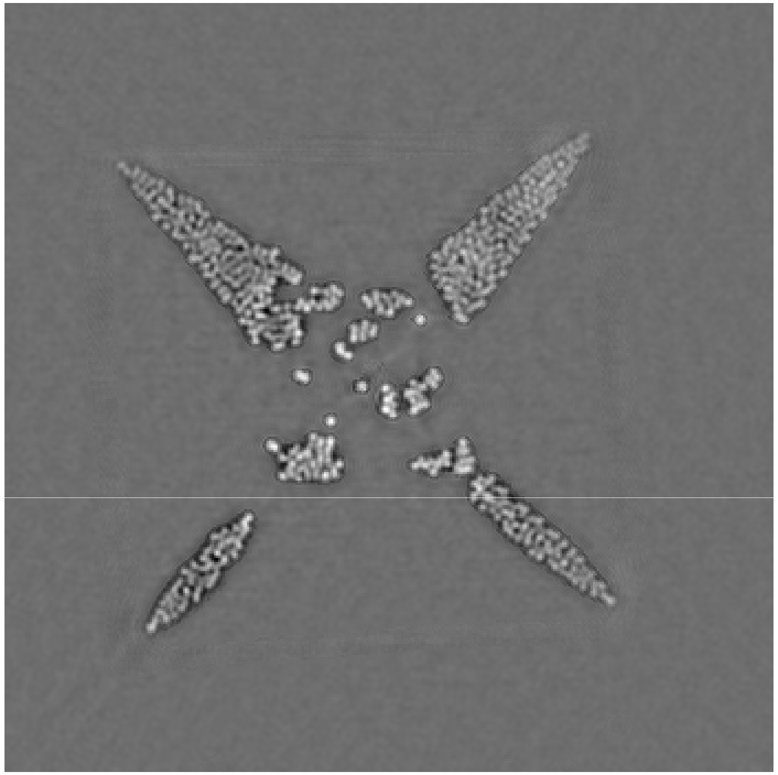
Pure projections from phased 3D data

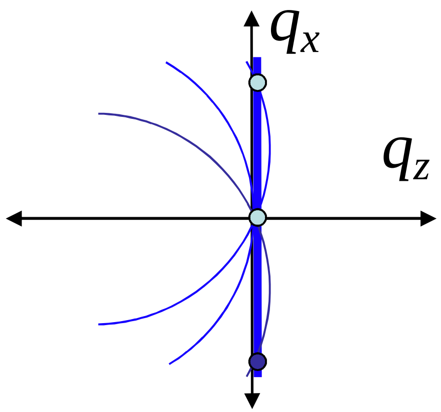
Chapman, Barty, Marchesini, Noy, Hau-Riege, Cui, Howells, Rosen, He, Spence,
Weierstall, Beetz, Jacobsen, Shapiro, *J. Opt. Soc. Am. A* **23**, 1179 (2006)



Experimental realization

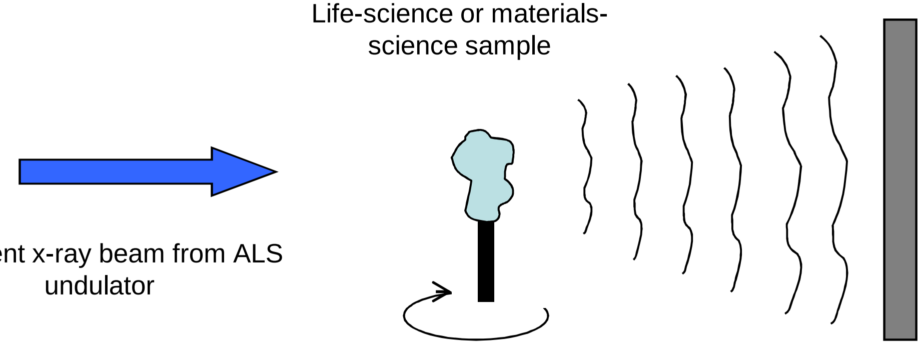




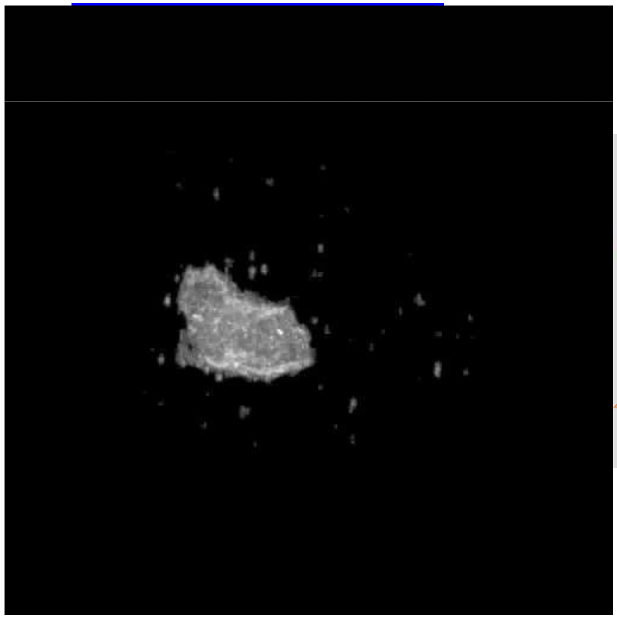


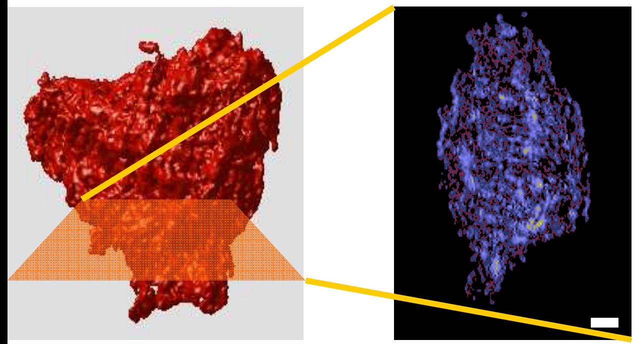
Chapman, Barty, Marchesini, Noy, Hau-Riege, Cui, Howells, Rosen, He, Spence, Weierstall, Beetz, Jacobsen, Shapiro, *J. Opt. Soc. Am. A* **23**, 1179 (2006)

H. Chapman, A. Barty, M. Howells, S. Marchesini et al., LLNL, LBL



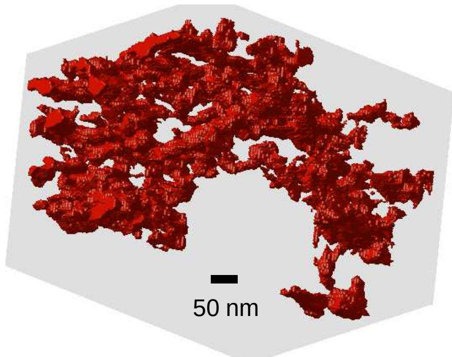
SOME RESULTS





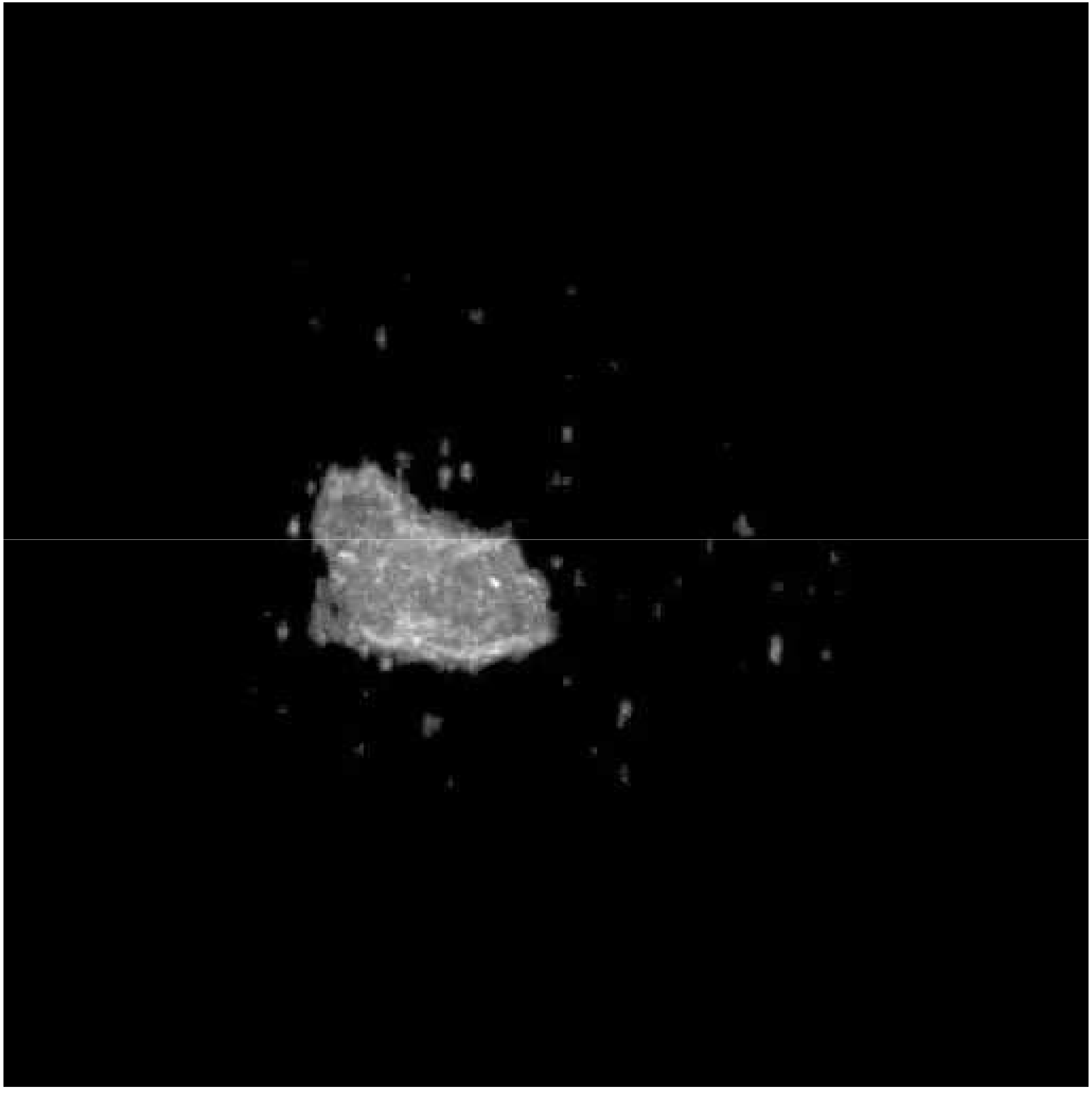
3D isosurface

Cross-section (bar = 200nm)

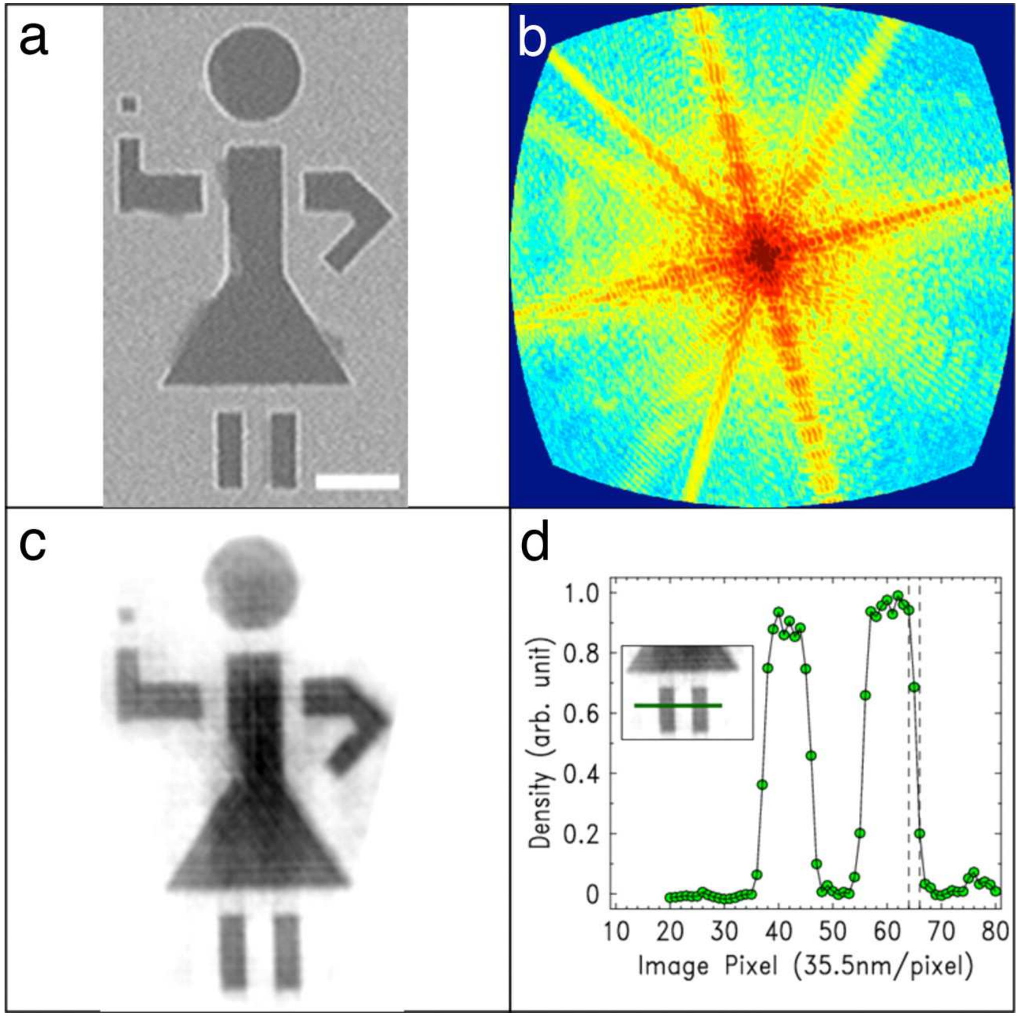


Res'n 20-30 nm

2-micron-wide particle of tantalum oxide foam of density about 0.1 gm/cm^3 which is about 1.2% of bulk density. The dataset of 280 views for the latter image was collected over two 8-hour shifts at 3.7 minutes per angle



.Lensless imaging with coherent soft x-ray laser beams at 47 nm

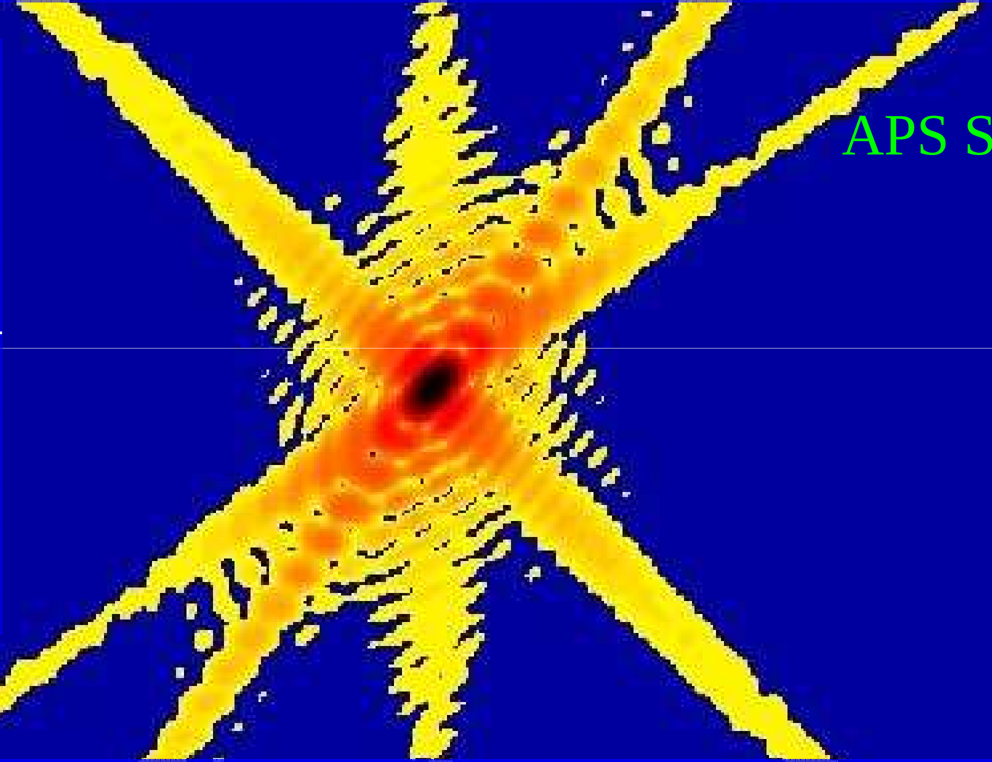


Sandberg, Richard L. et al. (2008) Proc. Natl. Acad. Sci. USA 105, 24-27

PNAS

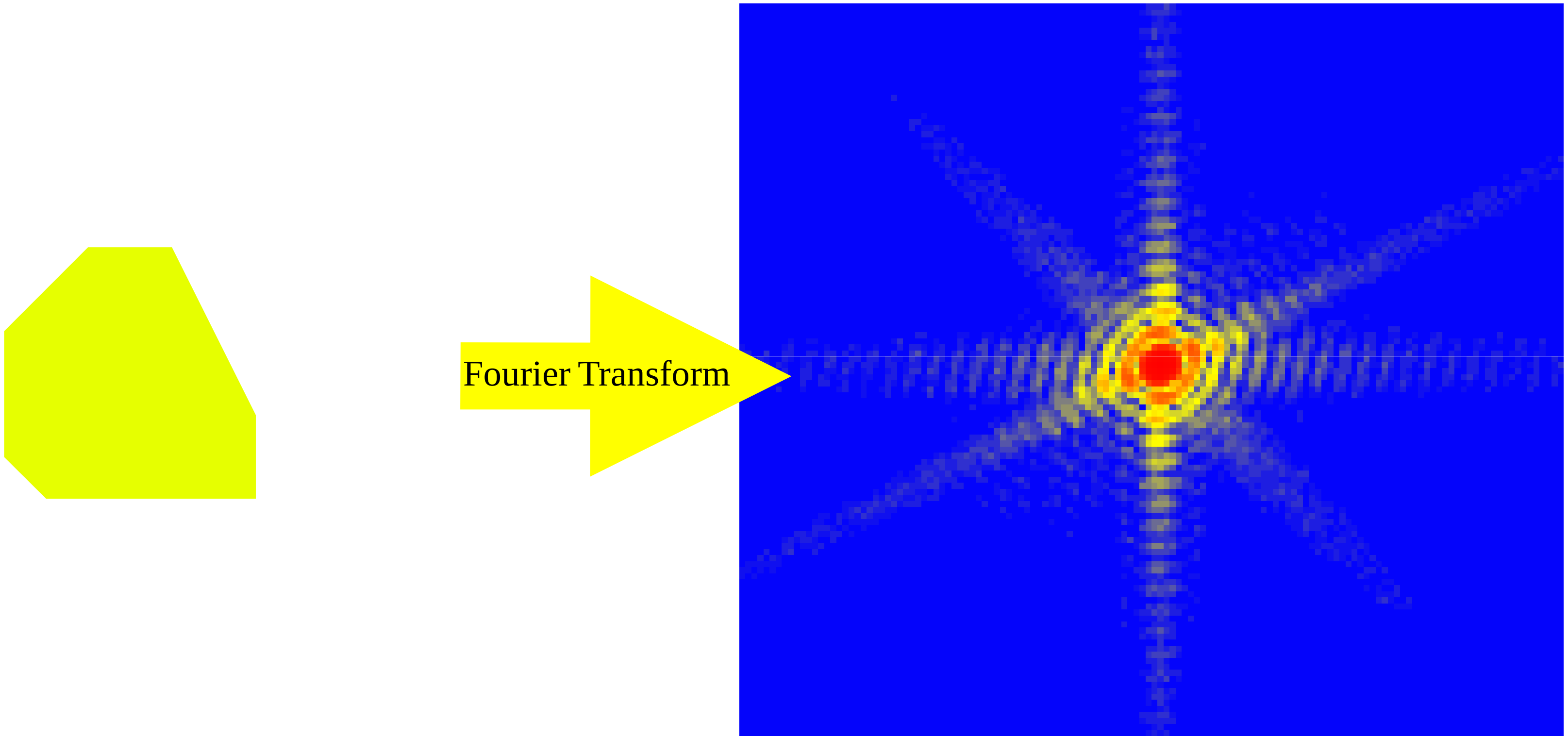
Nanometer Scale Imaging Through Coherent X-ray Diffraction

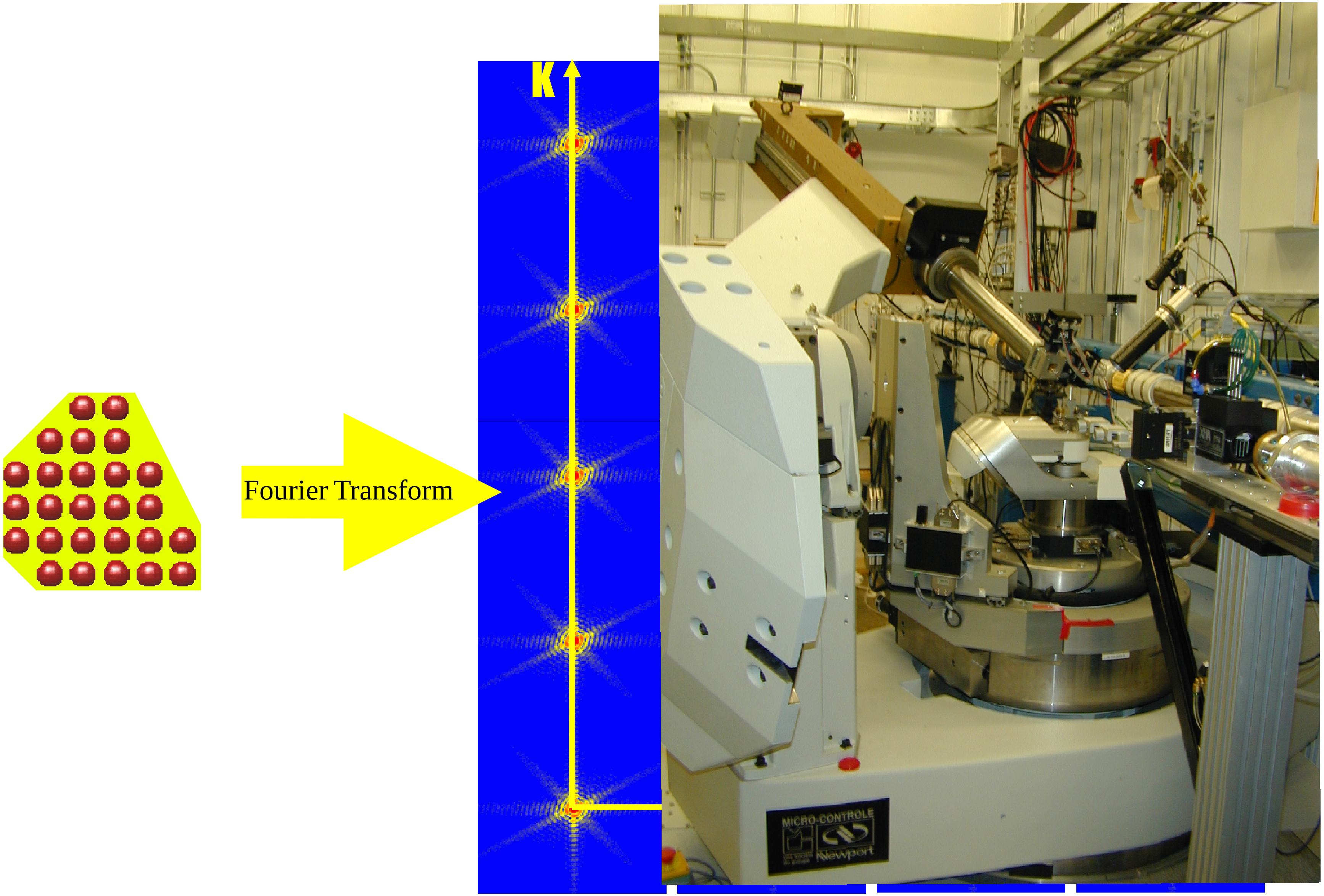
Ross Harder[1]
Meng Liang[2]
Steven Leake[1]
Mark A. Pfiefer[3]
Garth J. Williams[4]
Ivan Vartaniants[5]
Christoph Rau[6]
Zhauyu Wang[2]
I.K. Robinson[1]

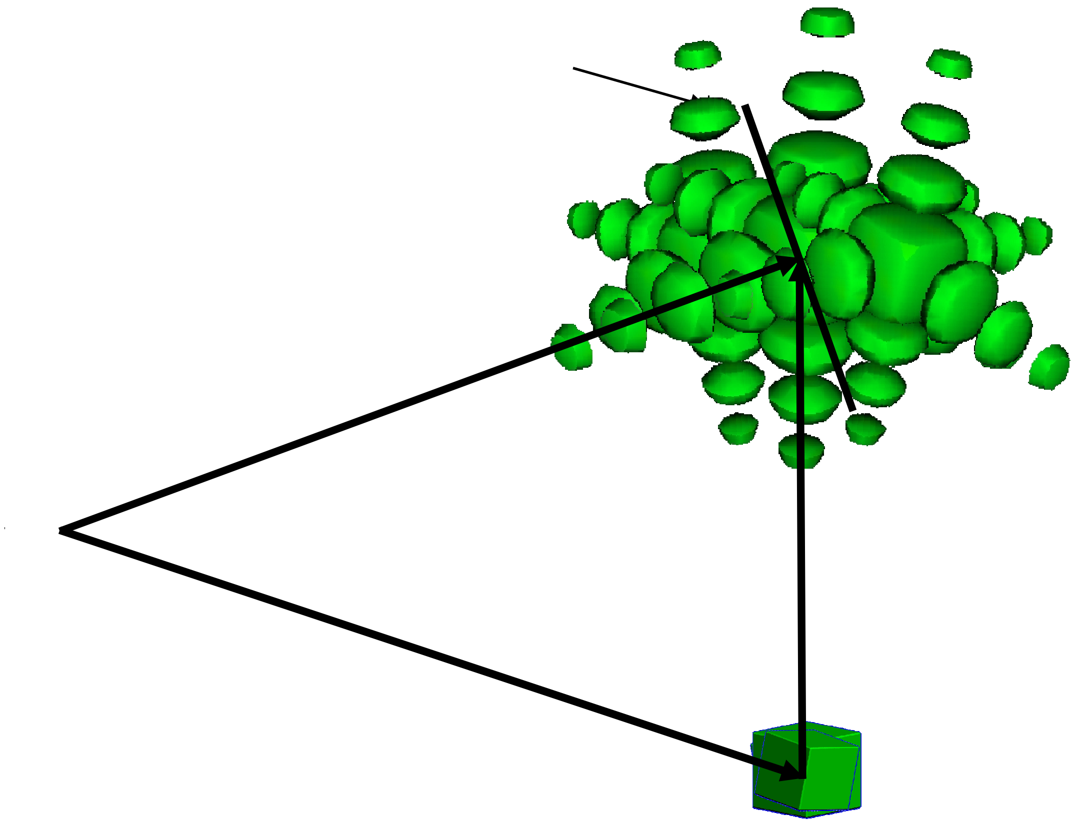


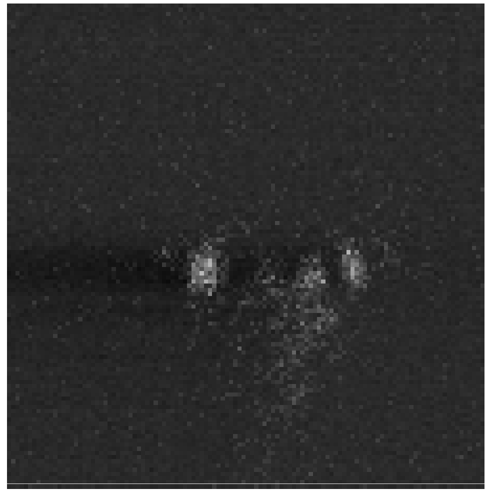
APS Sector 34ID-C

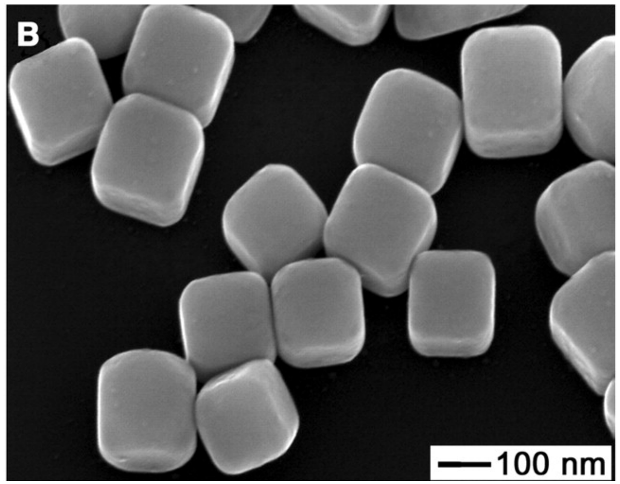
[1] London Centre for Nanotechnology, Dept. of Physics and Astronomy, University College London
[2] Dept. of Physics University of Illinois Urbana-Champaign
[3] Dept. of Physics La Trobe University, Victoria Australia
[4] School of Physics University of Melbourne Australia
[5] HASYLAB, DESY Hamburg Germany
[6] Advanced Photon Source, Argonne National Lab

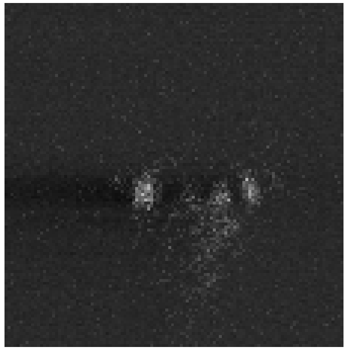

Fourier Transform

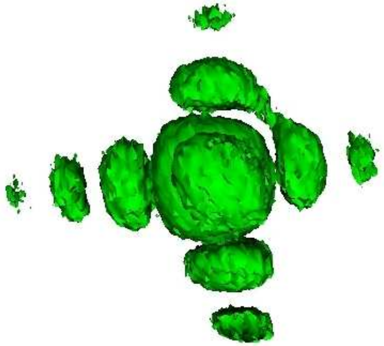

K
Fourier Transform
MICRO-CONTROLE
Newport

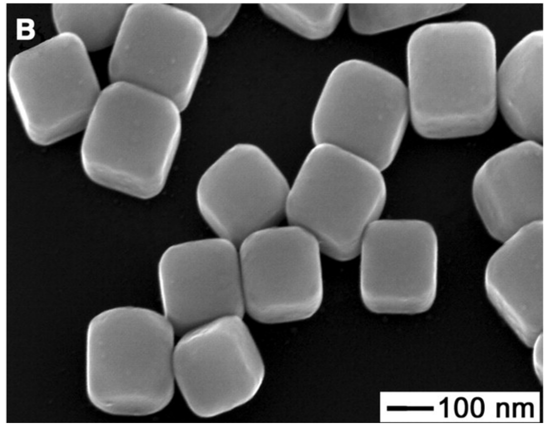


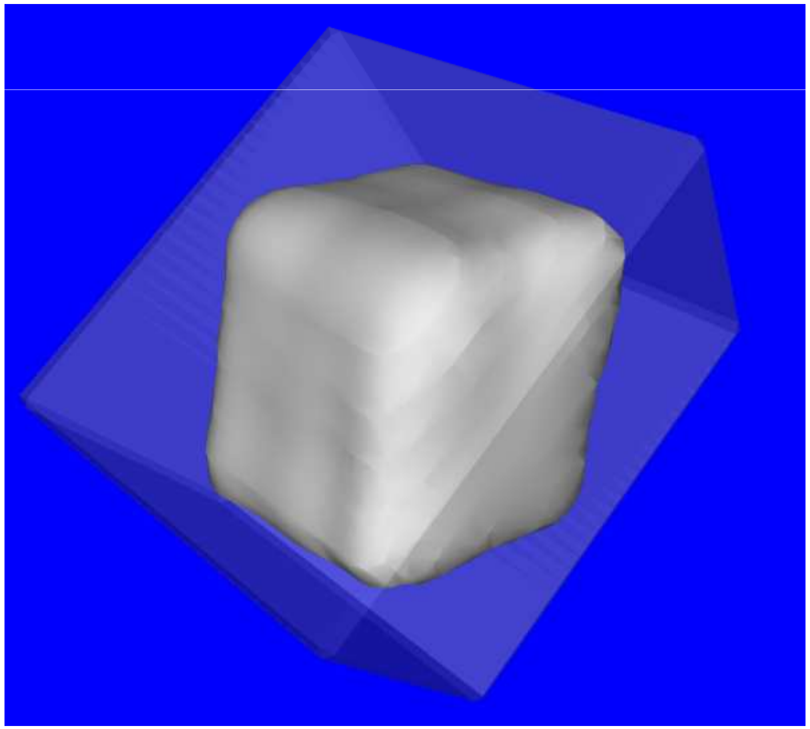


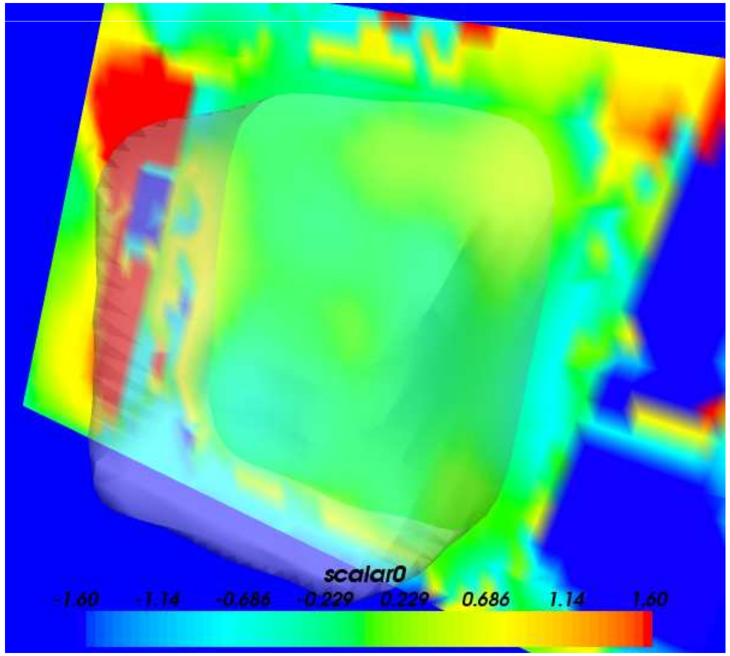

B
100 nm





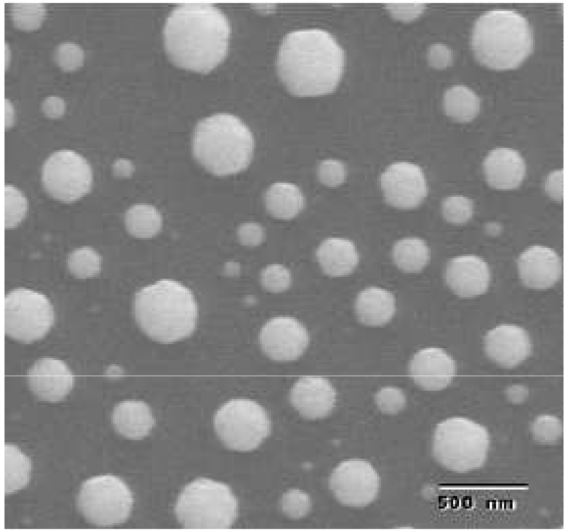

B
100 nm

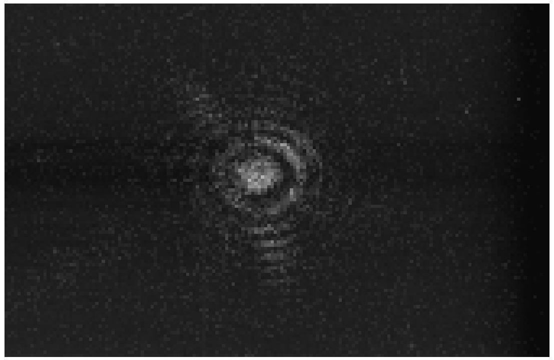


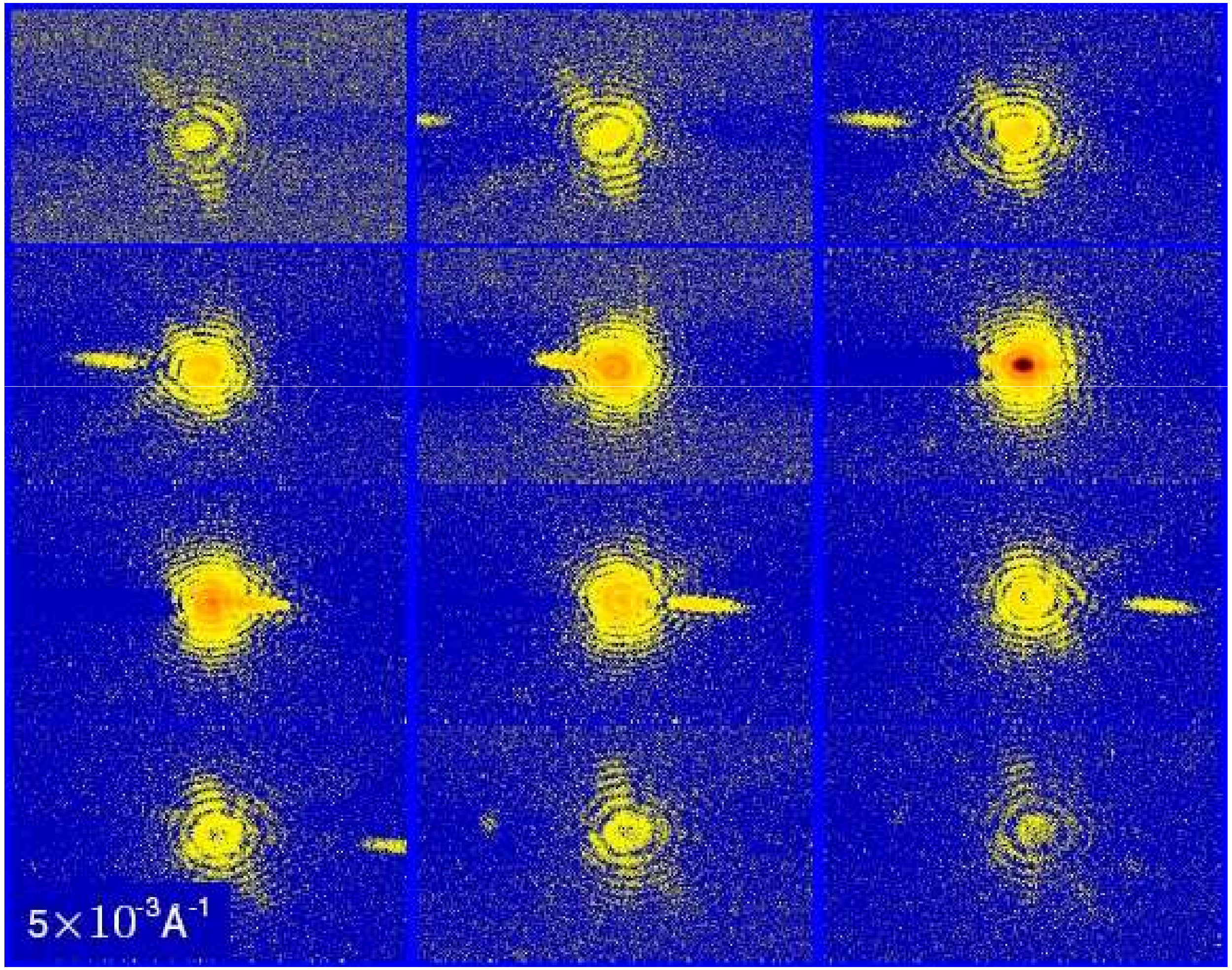

scalar0
-1.60 -1.14 -0.686 -0.229 0.229 0.686 1.14 1.60

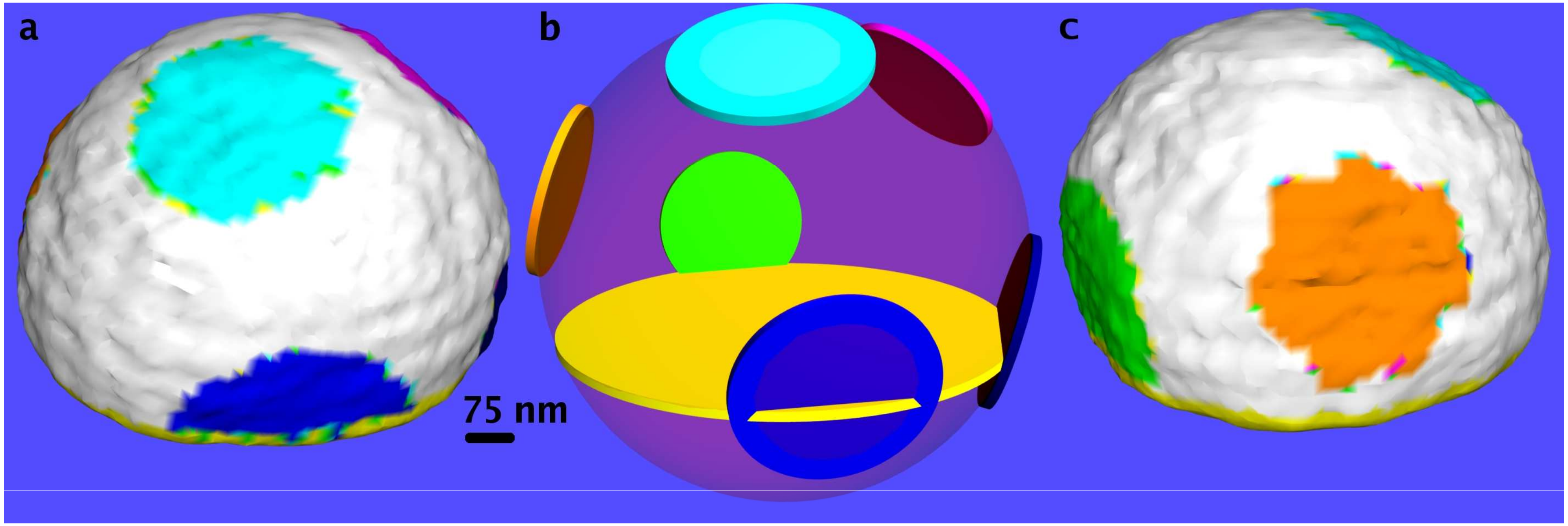
Hot Science! (T=573K)

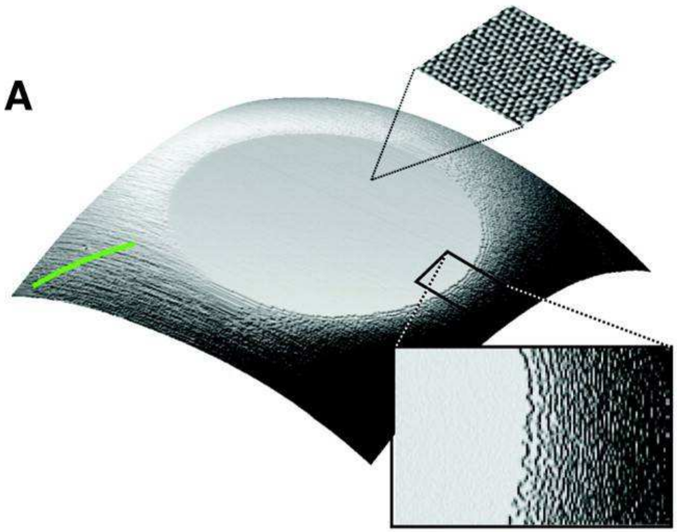
3D Diffraction From Lead







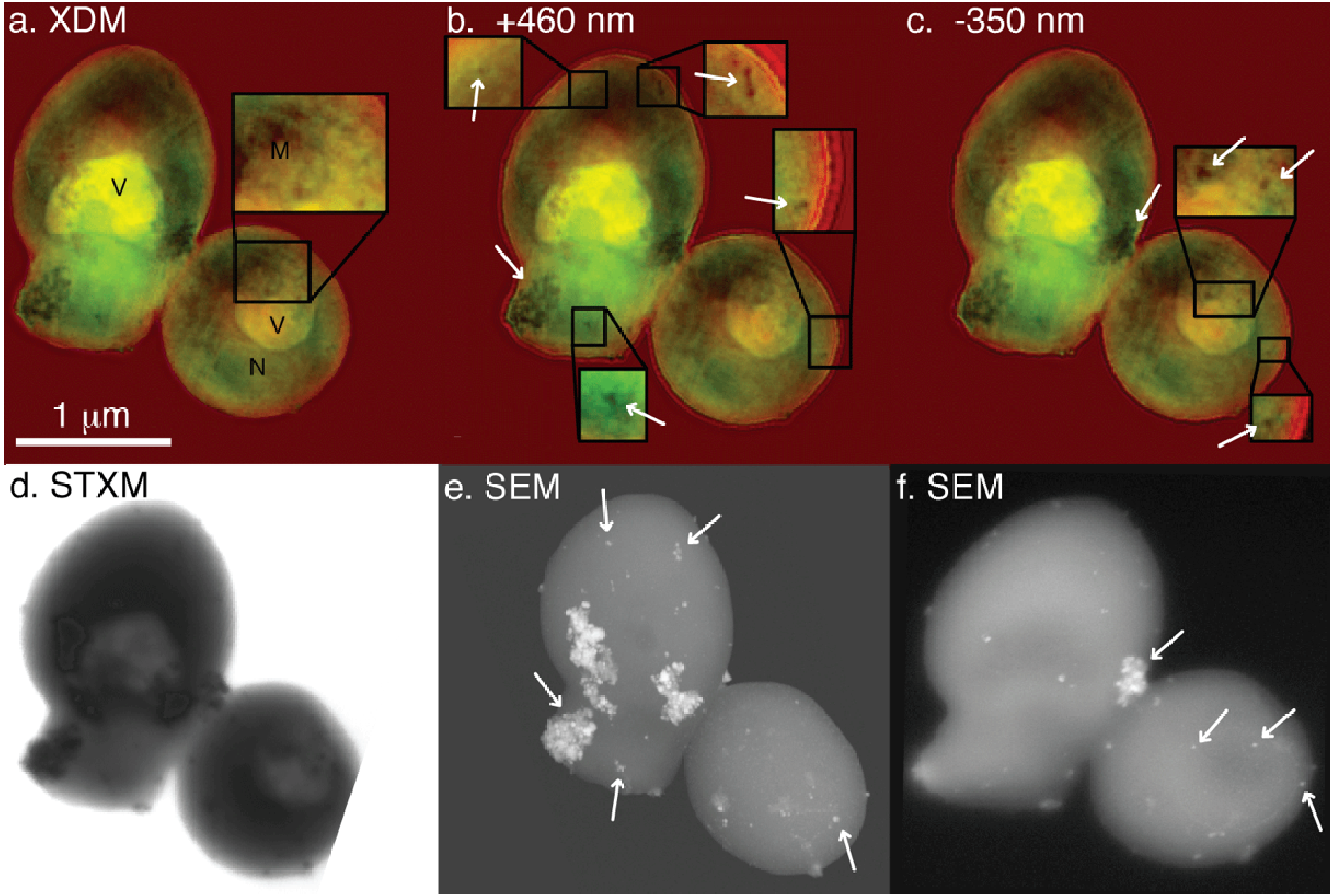




Thurmer K, Williams E, Reutt-Robey J
Science **297** 2033 (2002)

Gold-labeled yeast

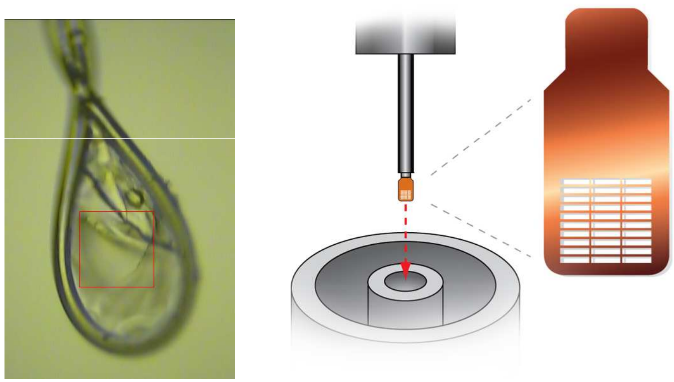
1.8 nm gold, silver-enhanced, freeze-dried: Johanna Nelson, Stony Brook.
Propagation of complex reconstructed wave to "focus" on different planes.



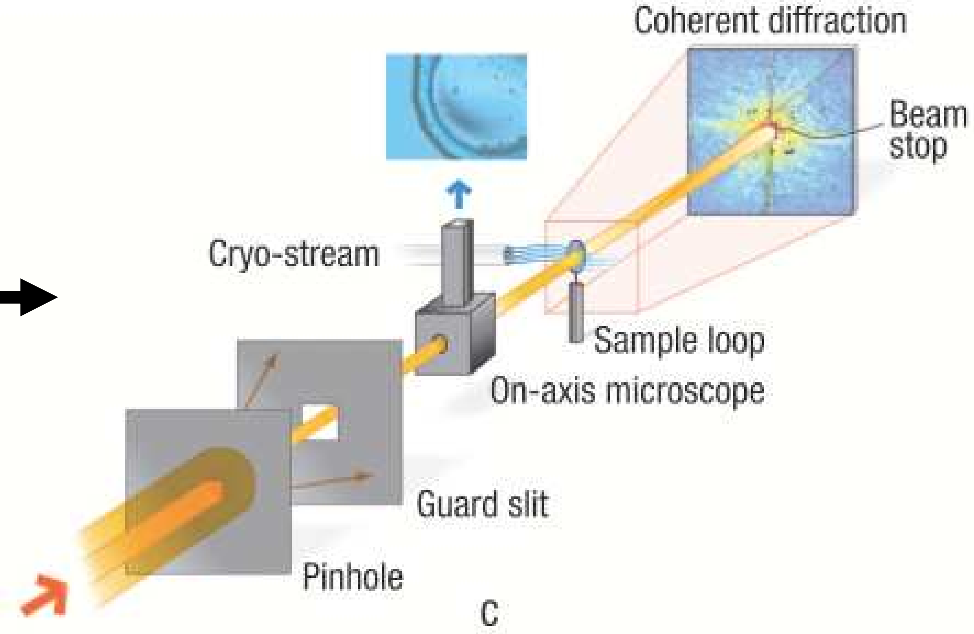
PNAS 107. 7235 (2010)

Sample preparation and data collection

E. Lima et al. PRL 103, 198102 (2009) ESRF



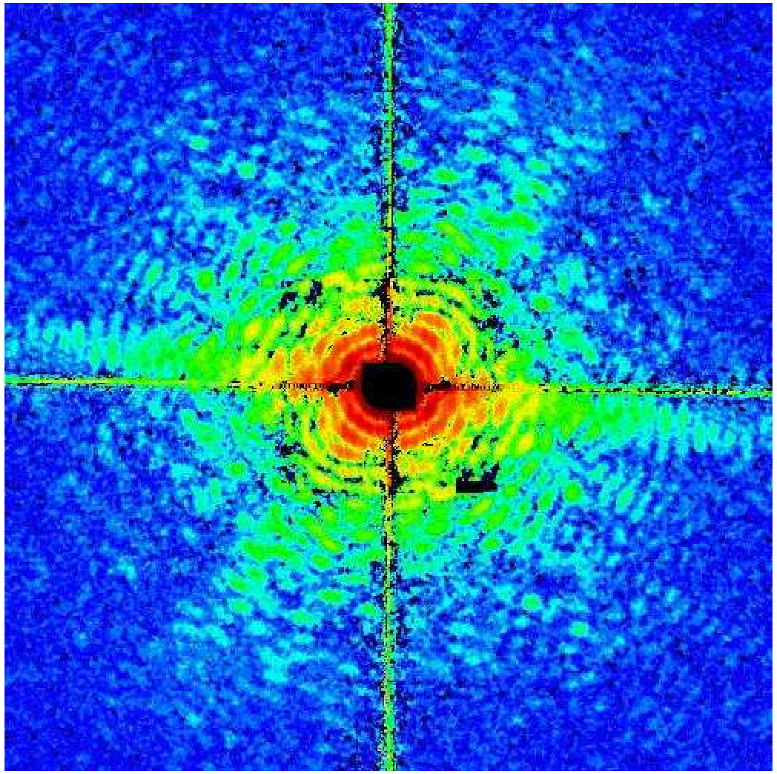
- Vitrification by plunge-freezing into liquid ethane

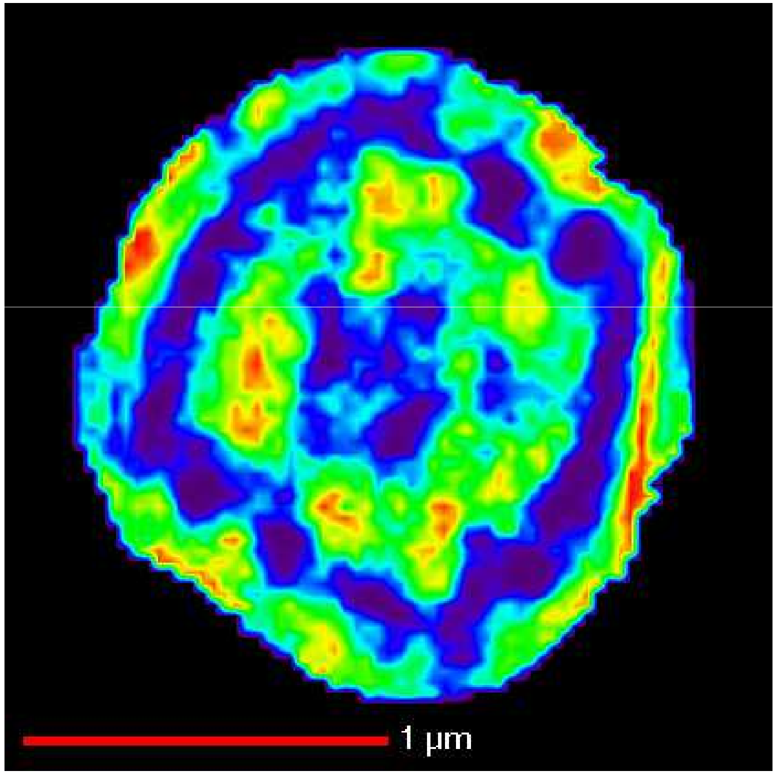


- Experimental setup for data collection using cryogenic gas stream

E. Lima et al. PRL 103, 198102 (2009) ESRF

Diffraction pattern and reconstruction

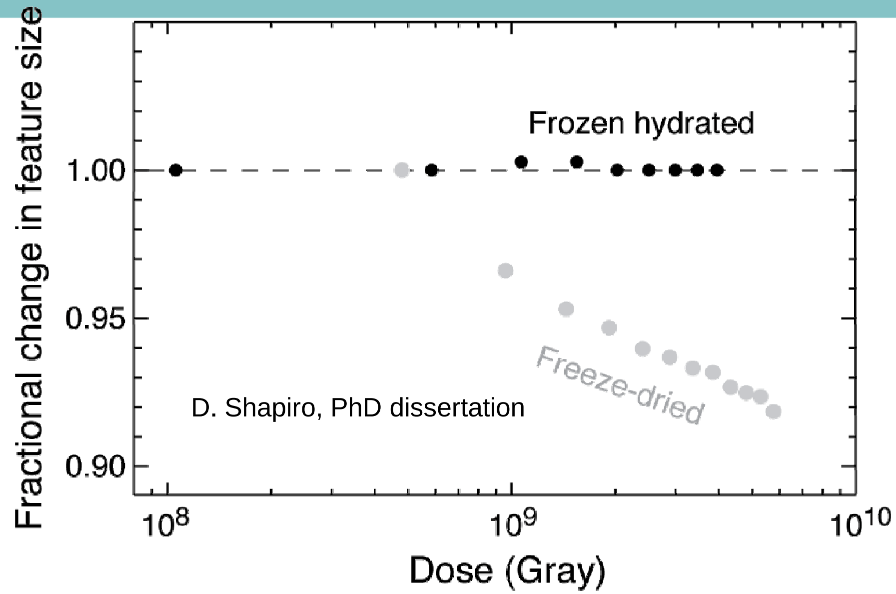


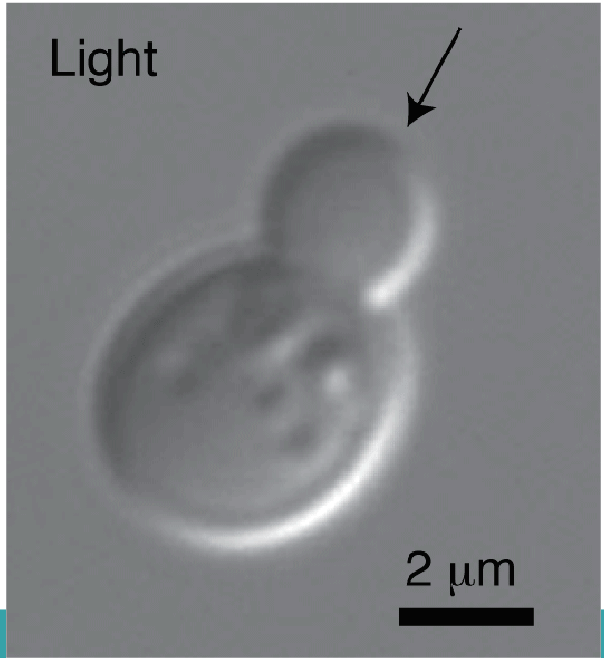


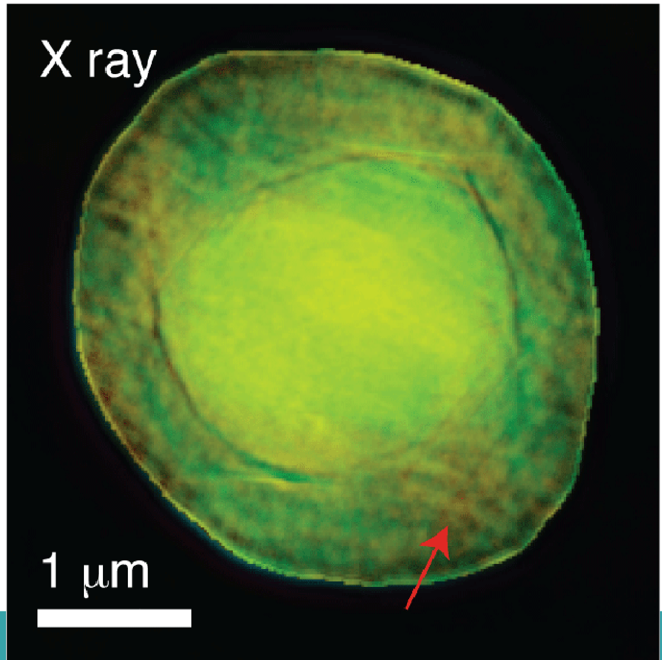
- Diffraction pattern from *D. radiodurans* using 8 keV x-rays
- Reconstructed image of *D. radiodurans*

Frozen hydrated yeast

- X. Huang *et al.*, Stony Brook/ALS PRL 103, 198101 (2009)

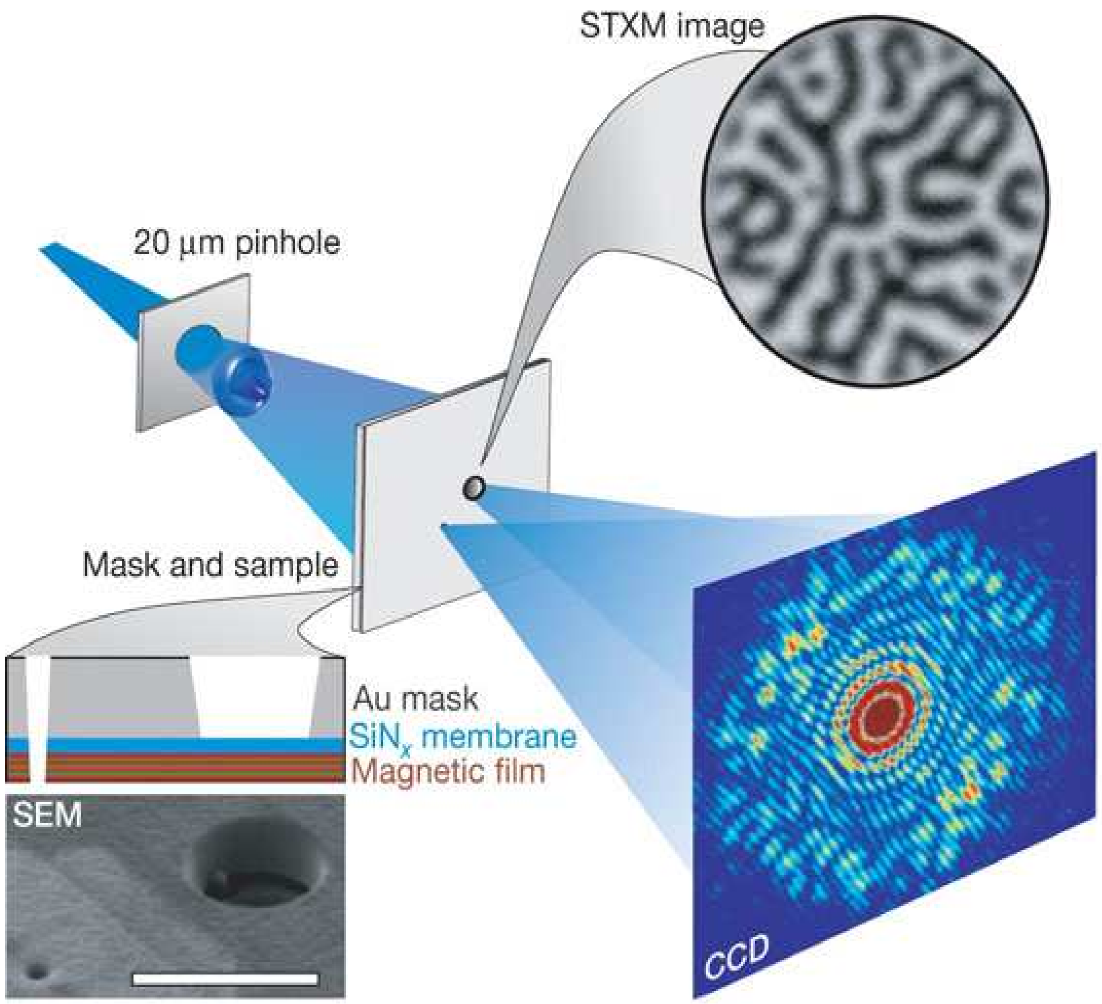






Fourier transform holography

S. Eisebitt, J. Lüning, W. F. Schlotter, M. Lörgen, O. Hellwig, W. Eberhardt and J. Stöhr Nature 432, 885-888(2004)



Fourier transform holography

- Size of pinhole sets resolution
- How to get enough photons through?
- Multiple holes (Schlotter et al.)
- Uniformly redundant arrays
  - (Marchesini et al. http://arxiv.org/abs/0801.4969)

D. Stickler et al.

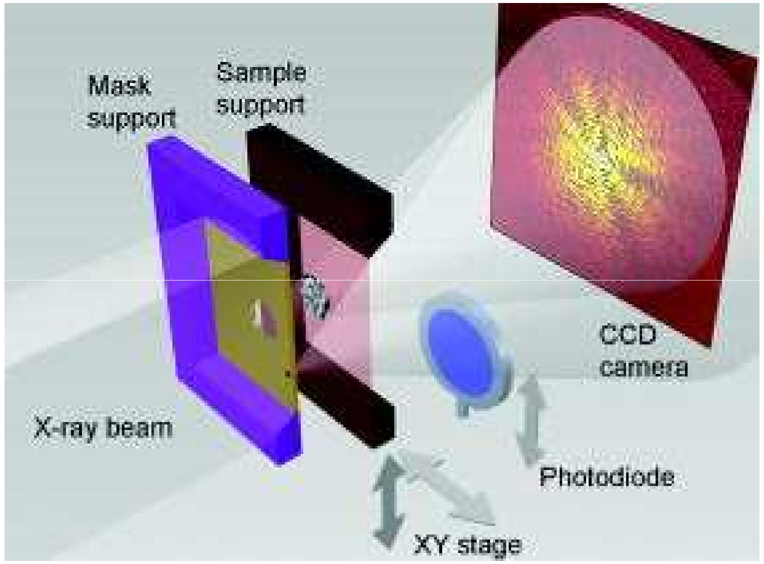


FIG. 1. Schematic of the x-ray holographic microscopy setup. It consists of a holography mask support, a movable sample support, and a CCD detector. The membrane with the optical elements (mask), i.e., the object and reference holes, is fixed in the center of the...

Appl. Phys. Lett. 96, 042501 (2010)

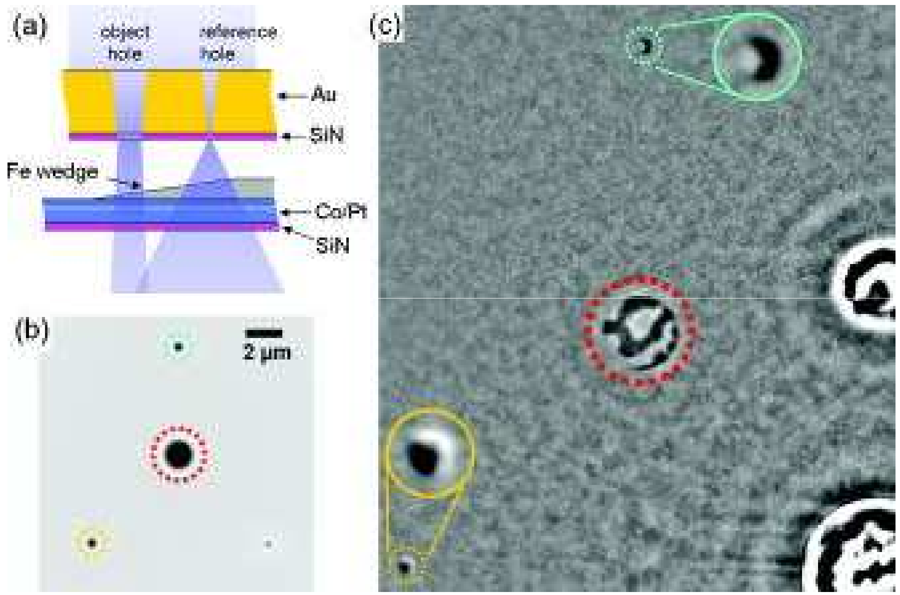


FIG. 2. Sample geometry and reconstruction of a single magnetic domain image. A cut through the scattering plane is shown in (a). The sample is illuminated through the optics membrane [SEM micrograph (b)]. Image (c) is a cutout of the real part of the FFT reconst...

Appl. Phys. Lett. 96, 042501 (2010)

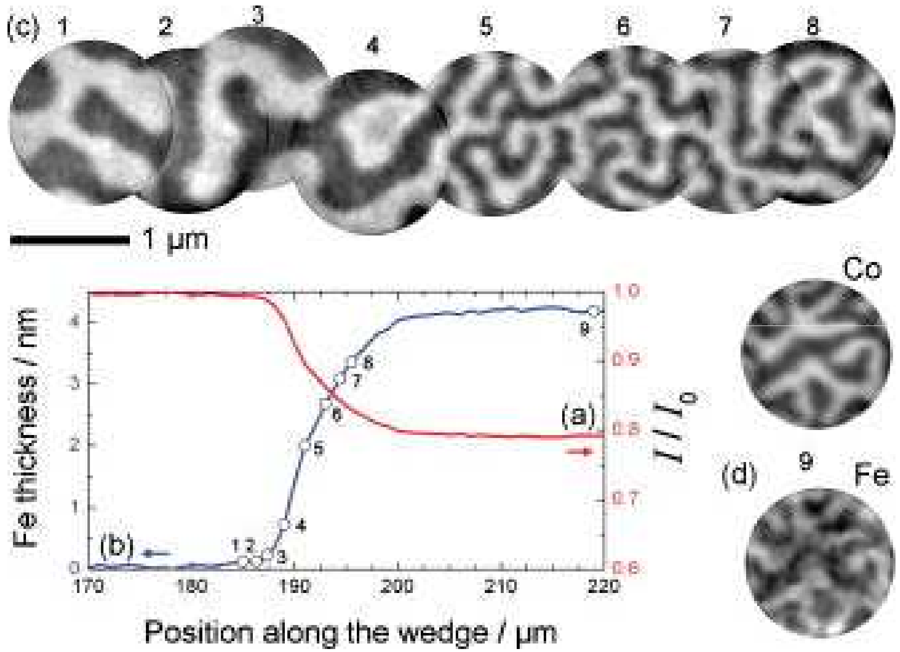
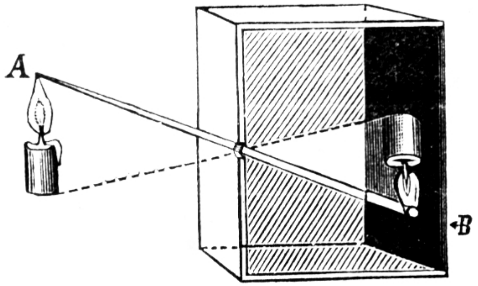


FIG. 3. Domain size evolution of a Co/Pt multilayer film covered by an iron wedge. Plot (a) gives the absorption profile (normalized photodiode current) at the Fe L3L3 absorption edge when scanning over the Fe wedge. The absorption is used to calculate the local ...

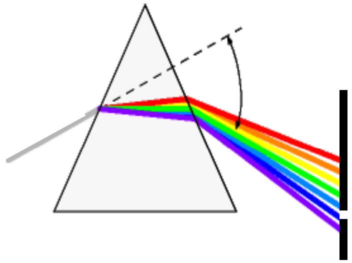
Appl. Phys. Lett. 96, 042501 (2010)

Coded aperture imaging

Pinhole camera

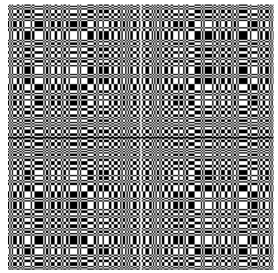


spectroscopy

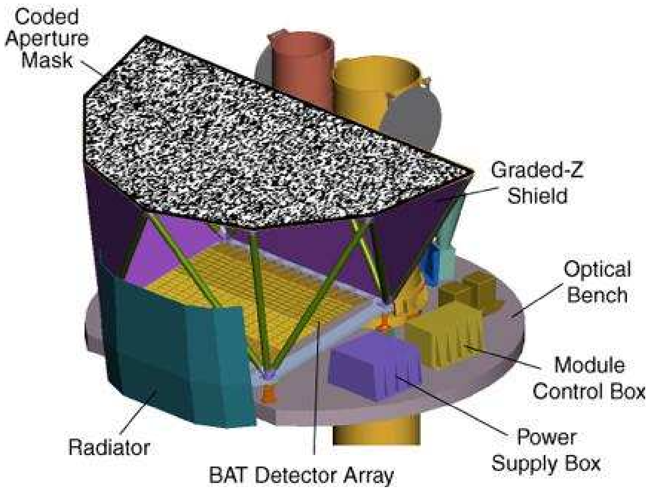


Resolution or SNR

Solution: use a uniformly redundant array

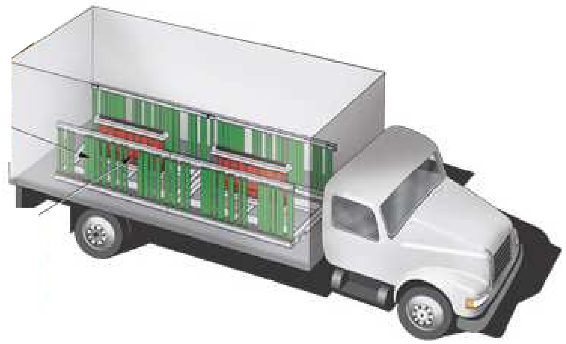


γ-ray astronomy



Medical imaging

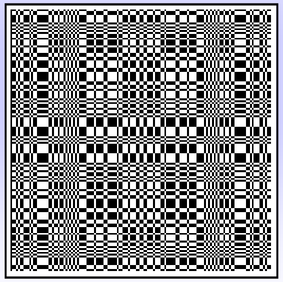
Homeland security



Coded aperture holography overcomes resolution vs brightness limitations

One point creates a hologram, many points create overlapping holograms: like a pinhole camera with many pinholes.

The “magic trick”:
An extended object with point-like autocorrelation (uniformly redundant array)



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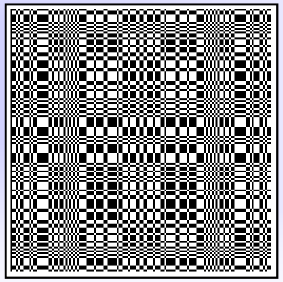

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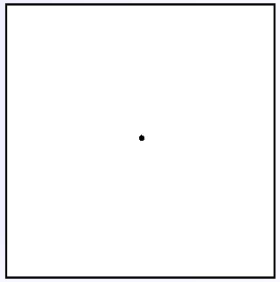
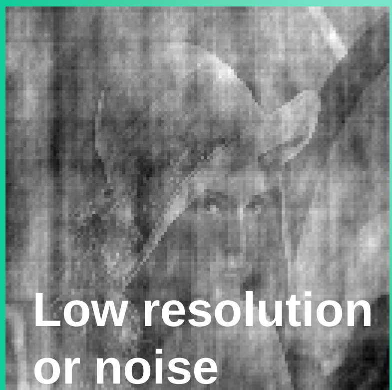
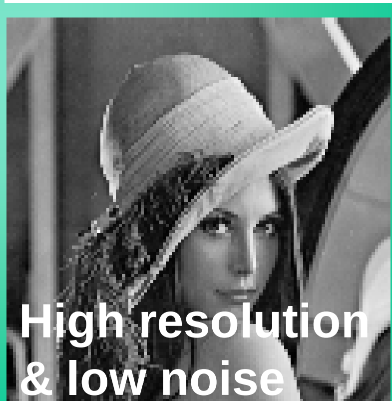


Same number of photons

Fourier transform holography

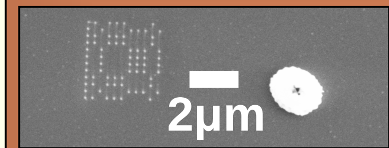


with coded apertures:
High resolution, low noise



Brightness and resolution improved by orders of magnitude by placing a coded aperture near a specimen

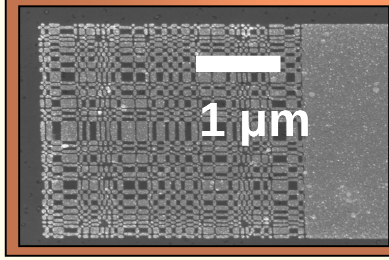
samples



Focused Ion Beam (50 nm res)

S. Boutet

SLAC

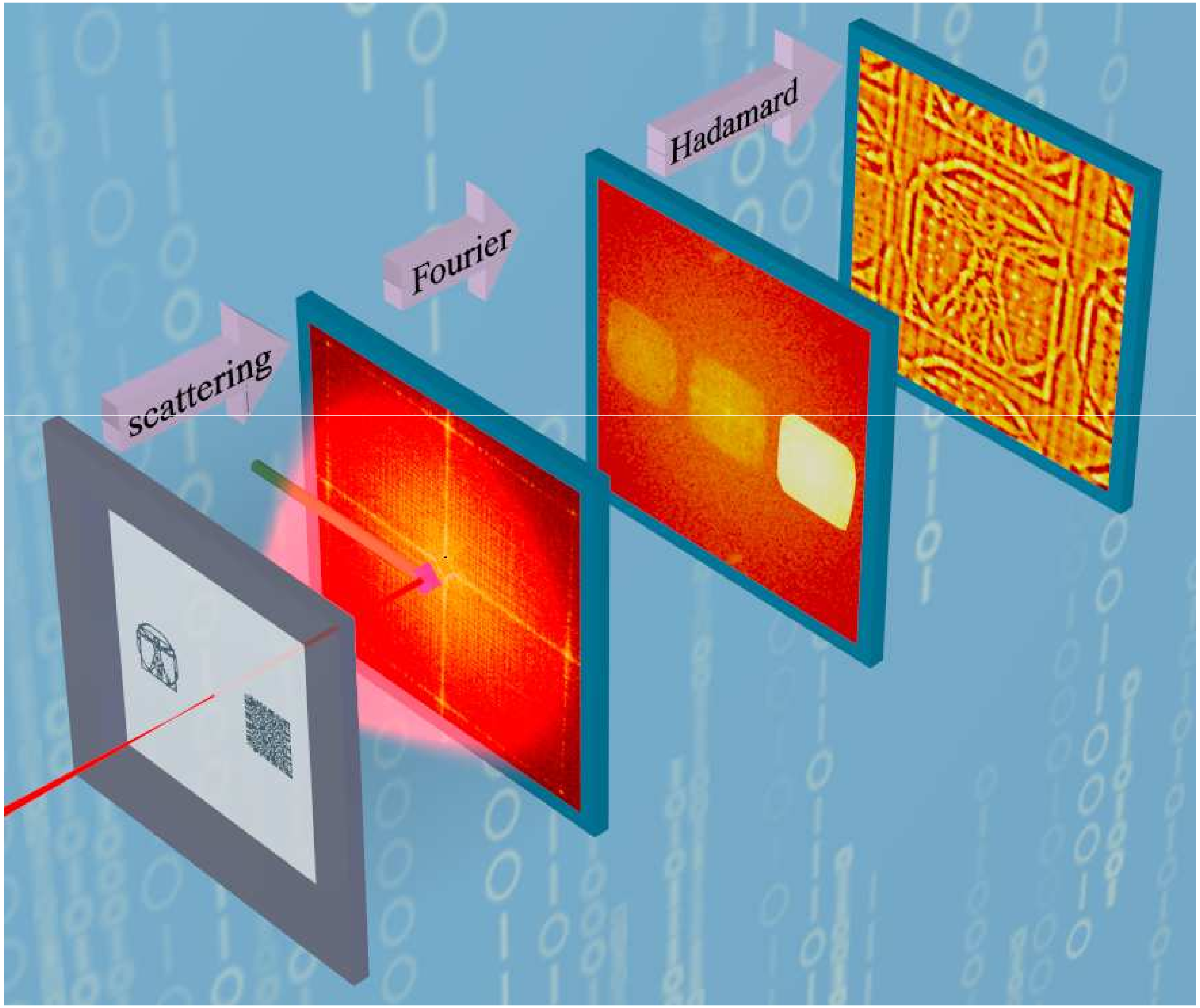


E- beam lithography (12 nm res)

Sakdinawat

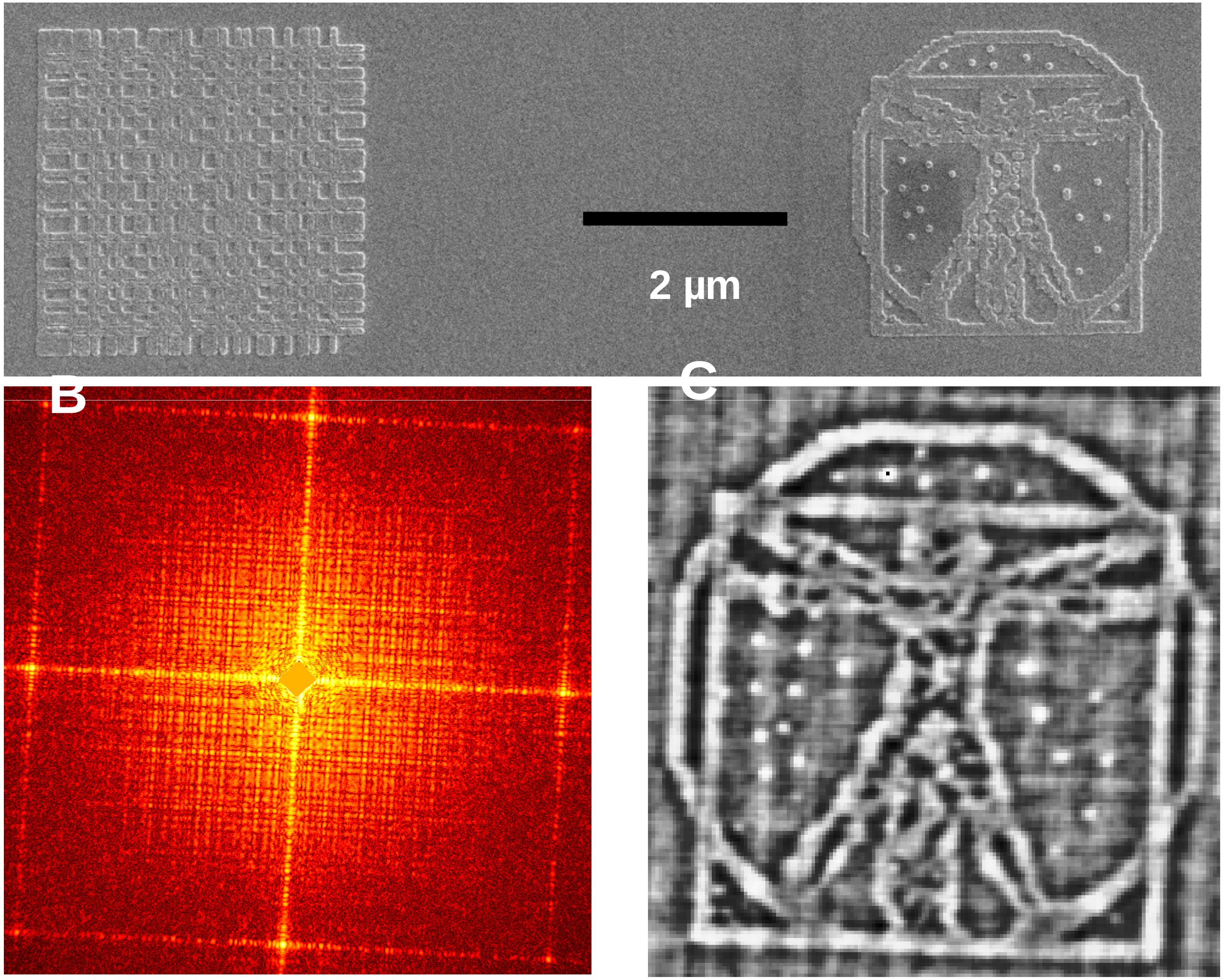
CXRO

Marchesini, 2007-09-10211, Fig. 1



Marchesini, 2007-09-10211, Fig. 2

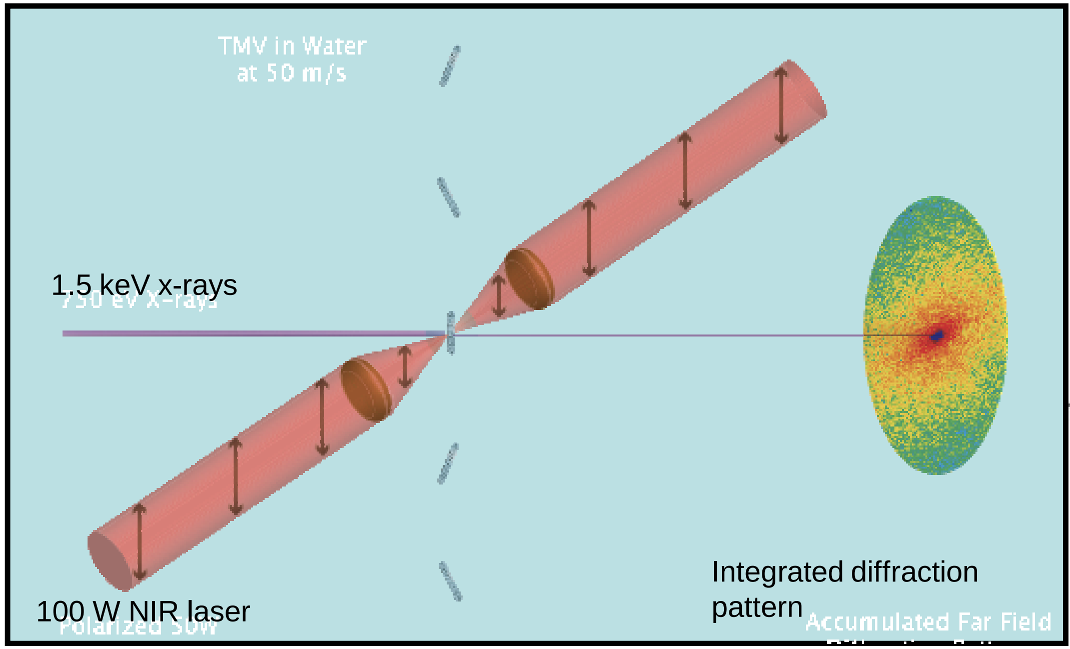
λ=2.2 nm Resolution=43 nm, SNR X ~70 Sample from A. Sakdinawat



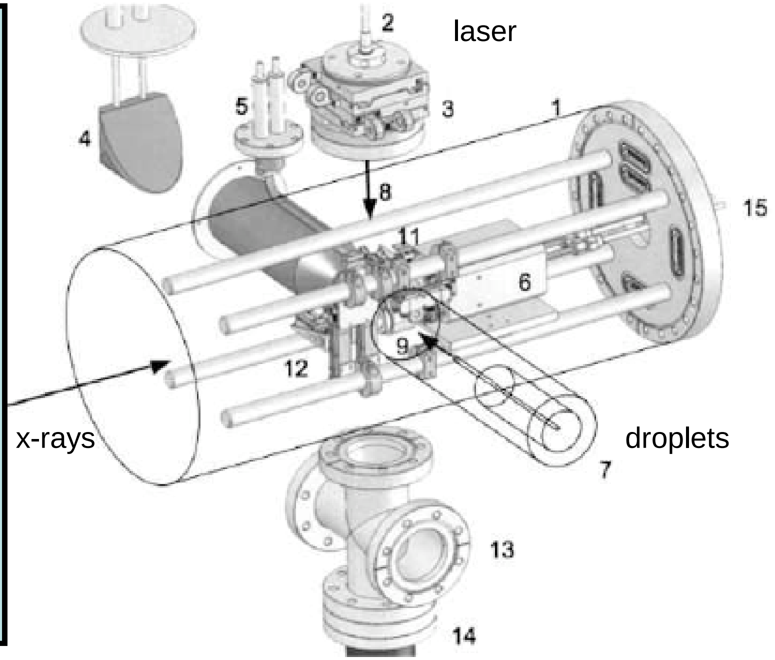
Serial Crystallography

GOAL: image uncrystallized proteins using the same basicXDM method

PROBLEM: how to collect hi resolution diffraction from single proteins?

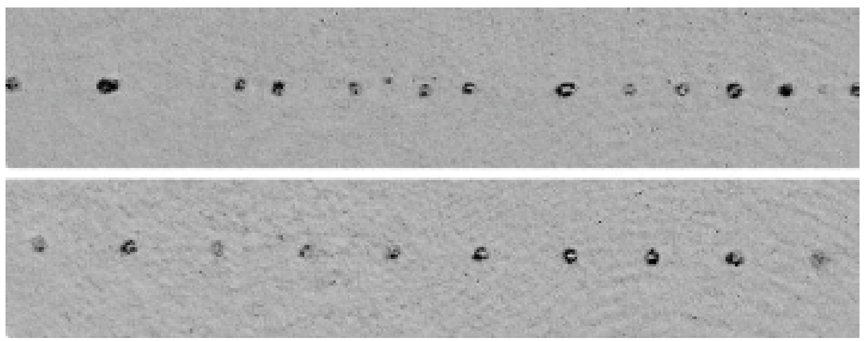


SC chamber at BL9.0.1



ASU droplet source - in use summer 07

J.C.H. Spence and R.B. Doak, Phys. Rev. Lett. ***92****, 198102 (2004)*
J.C.H. Spence et al., Acta Cryst. A ***61****, 237 (2005)*



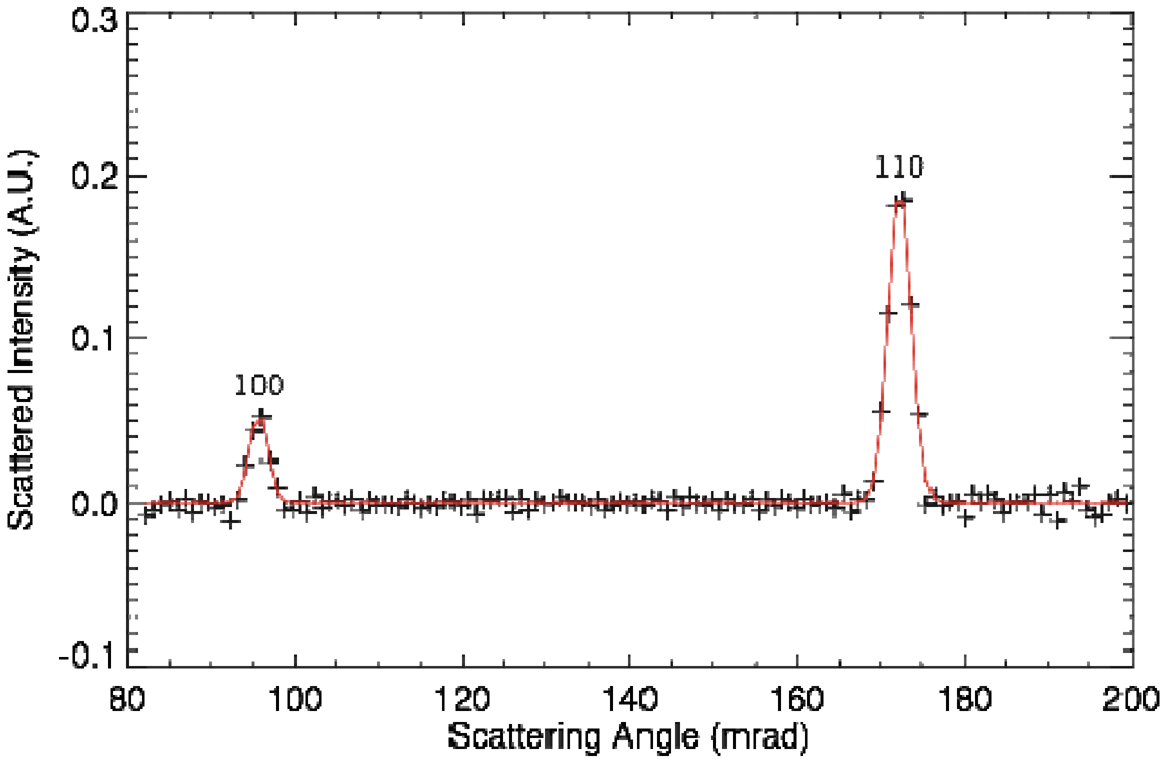
Photosystem 1 Protein

PS1 nano-crystals filtered with 500nm filter
Without alignment gives powder diffraction pattern

Low resolution because λ=2.34nm

	Measured	Expected
100 peak	96 mrad	94 mrad
110 peak	172 mrad	169 mrad
$\lvert F_{110}\rvert / \lvert F_{100}\rvert$	3.6	unknown
$\Delta\Theta_{100}/\Theta_{100}$	0.011	
$\Delta\Theta_{110}/\Theta_{110}$	0.008	
$\lambda/\Delta\lambda$	<126	<132

Powder Peaks with <0.025° width

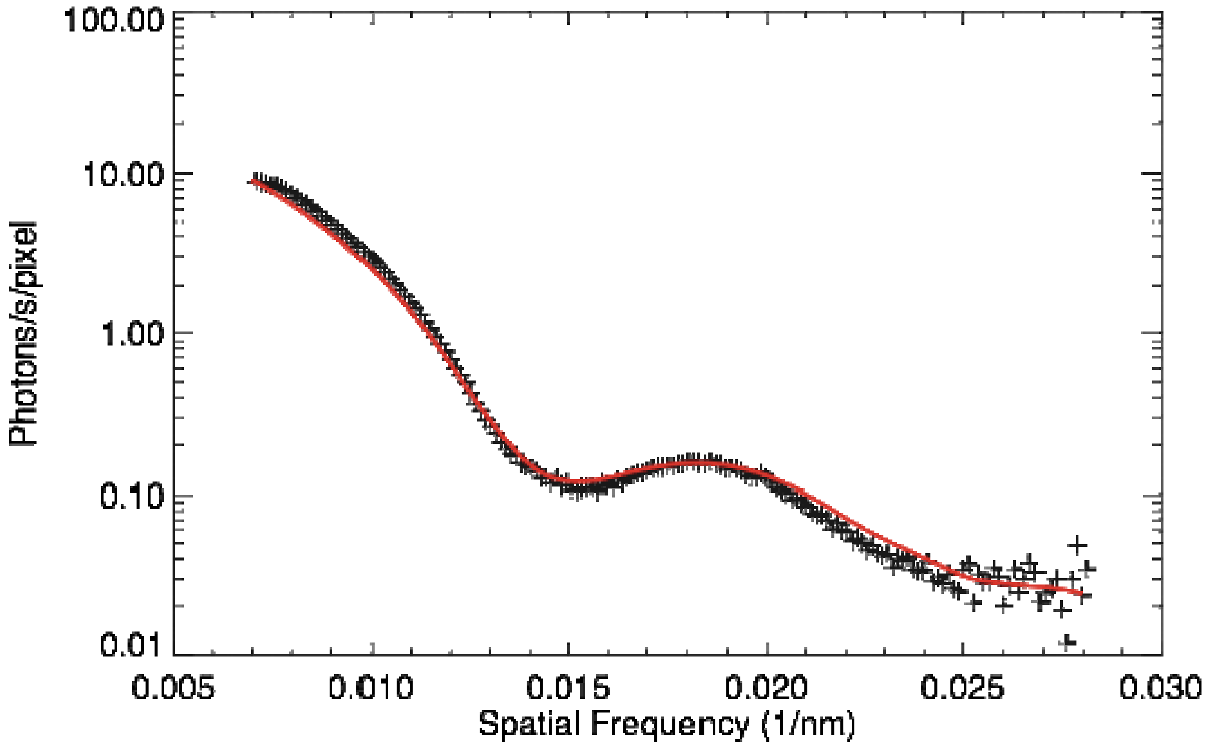


Protein powder diffraction without freezing or crushing
NO RADIATION DAMAGE!

Gold Nano-Spheres

Un-aligned particles give a SAXS pattern

- 50nm gold spheres, $5x10^{10}$ part./mL
- ~200 balls per 10 micron droplet
- Flow rate = 10μL/min
- 50,000 drops/second at 50m/s
- Total CCD integration = 2 minutes
- Equivalent exposure = 2 spheres for 2 minutes

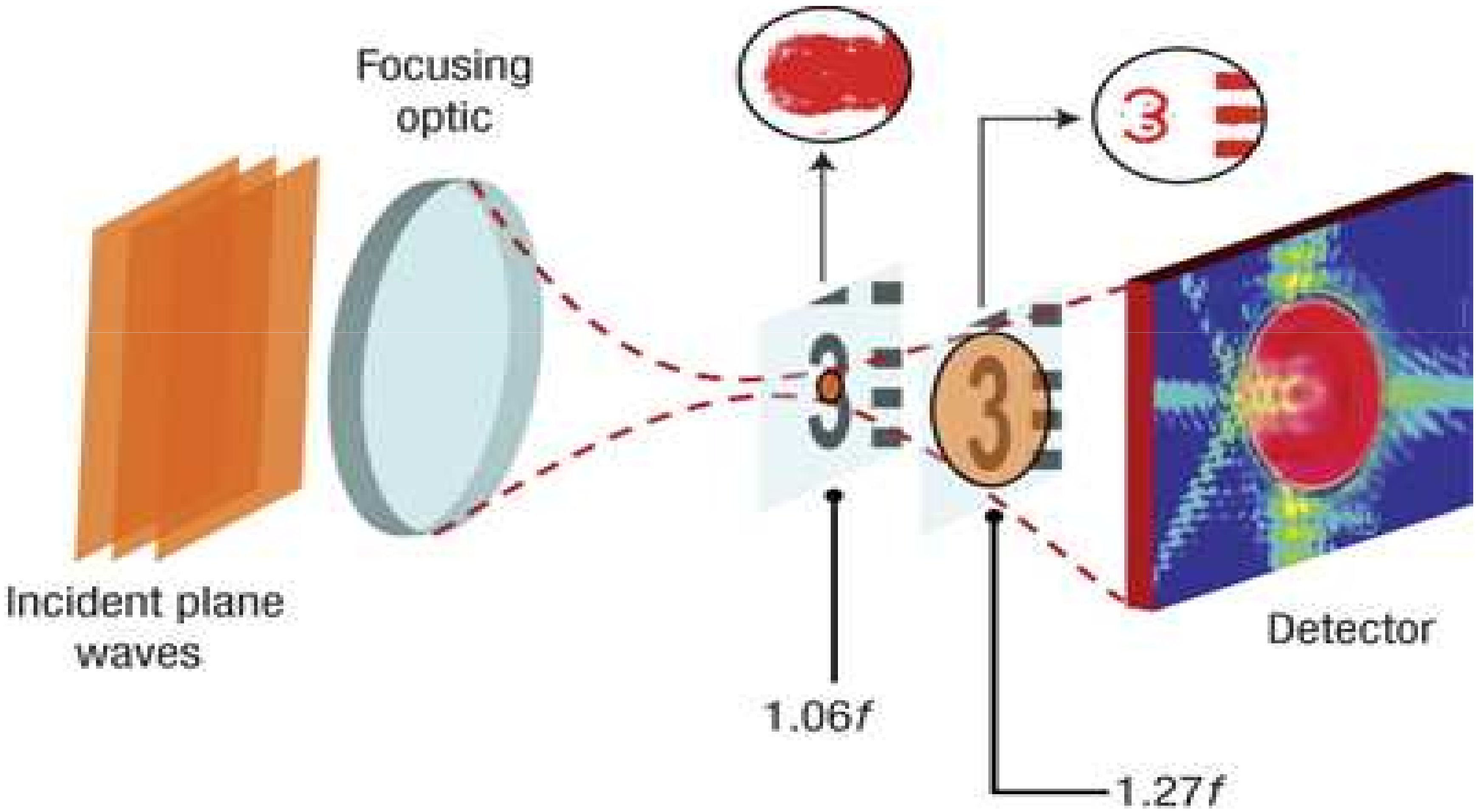


Size Distribution has:
sigma=6nm

D. Shapiro

Other ways to recover the phase

- Transport of Intensity (Nugent)
  - Measure the intensity and its gradient
- Spherically structured illumination (Williams, Nugent et al.)
  - Record Fresnel diffraction intensity
  - Converges fast!
- Keyhole Coherent Diffraction Imaging (Abbey, Nugent et al.)
  - Illuminated area defines the support
- Ptychography (Rodenburg, Pfeiffer et al.)
  - Precisely defined overlapping areas
  - Converges fast!

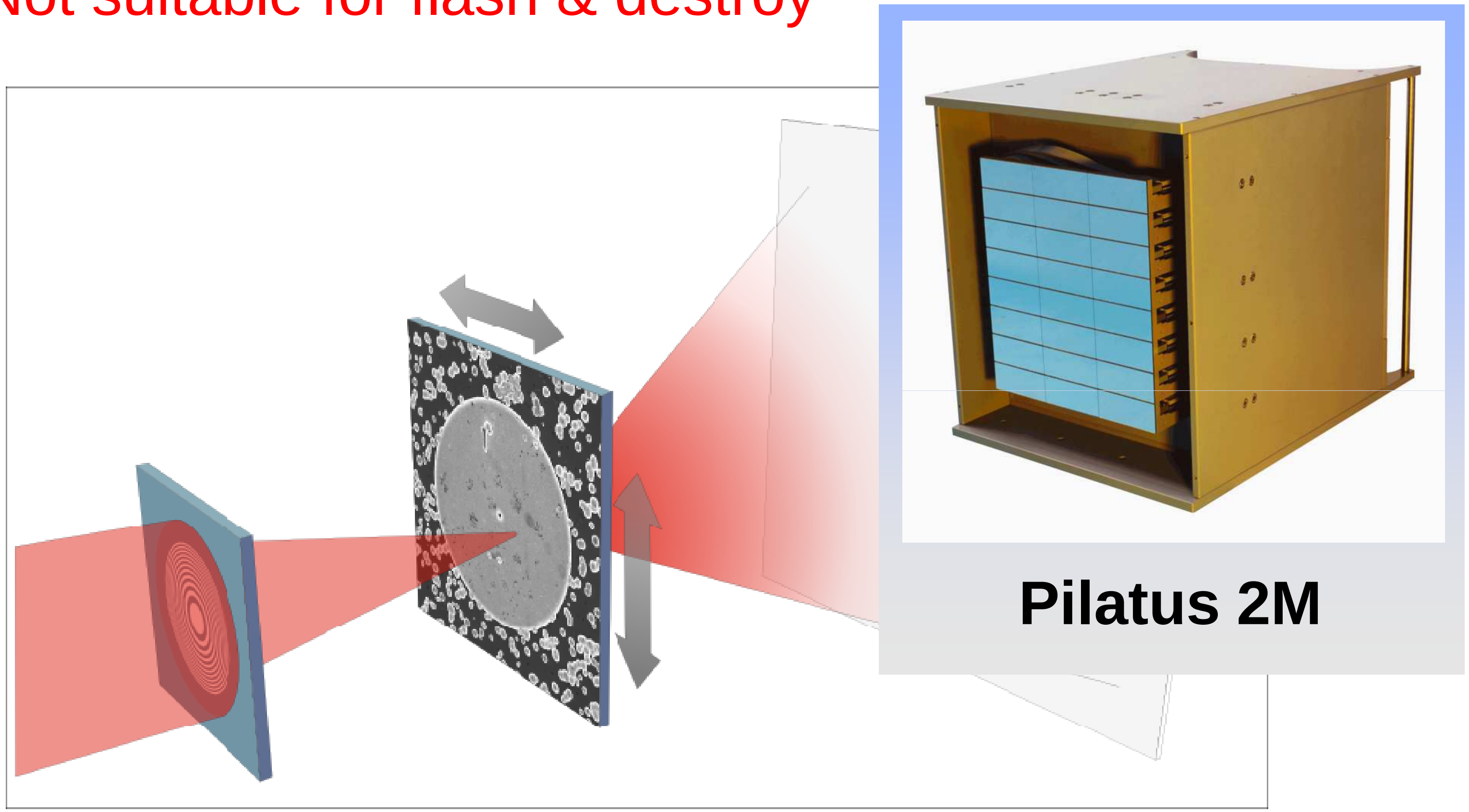


B. Abbey, K. A. Nugent, et al., Keyhole Coherent Imaging Nature Physics 2008

Scanning X-ray diffraction microscopy

Ptychography with a focused X-ray probe

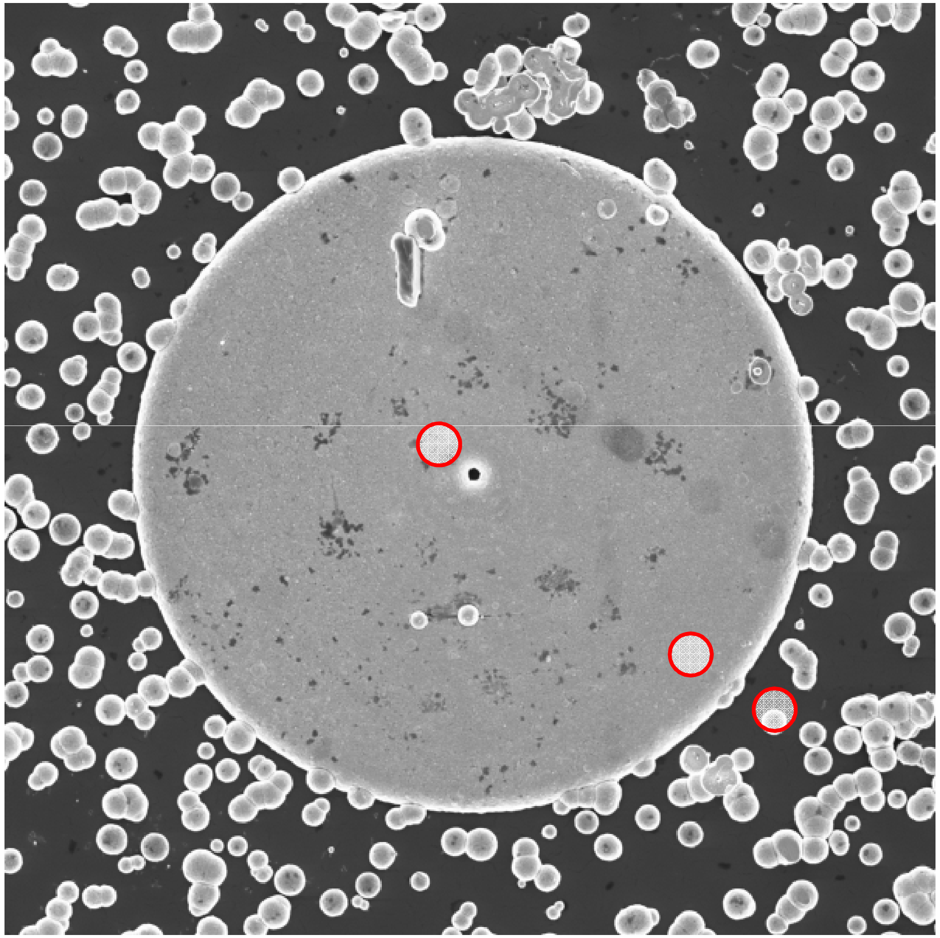
Not suitable for flash & destroy

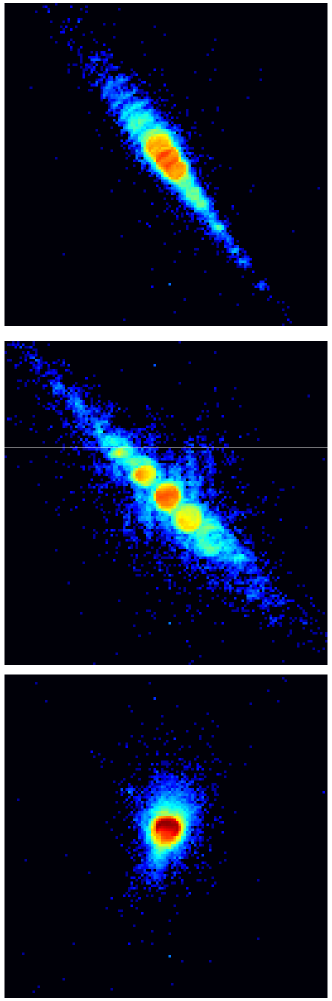


P. Thibault, M. Dierolf, A. Menzel, O. Bunk, C. David, F. Pfeiffer, Science, **321**, 379-382 (2008).

Scanning X-ray diffraction microscopy

Test specimen : Fresnel zone plate





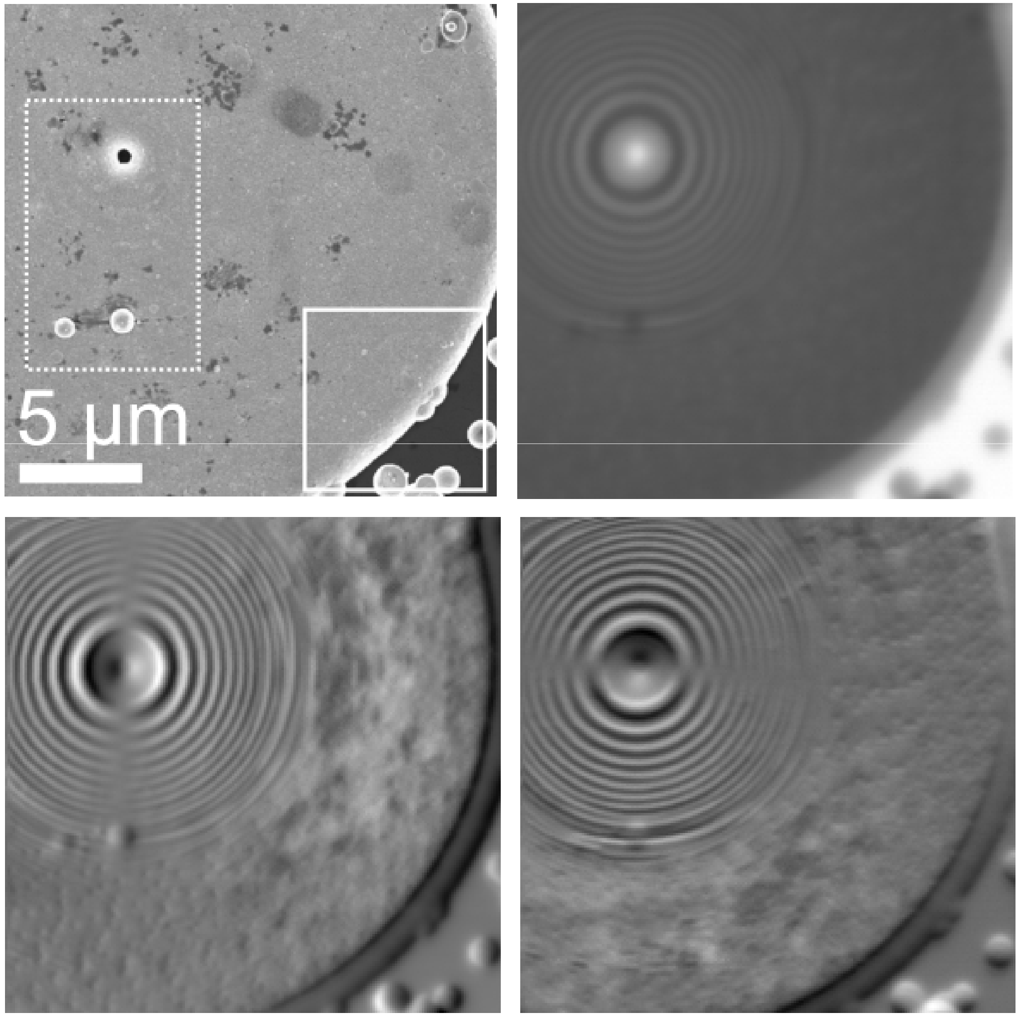
Thibault *et al.*, Science, **321**, 379-382 (2008).

Scanning X-ray diffraction microscopy

First analysis of the dataset “à la STXM”

Absorption

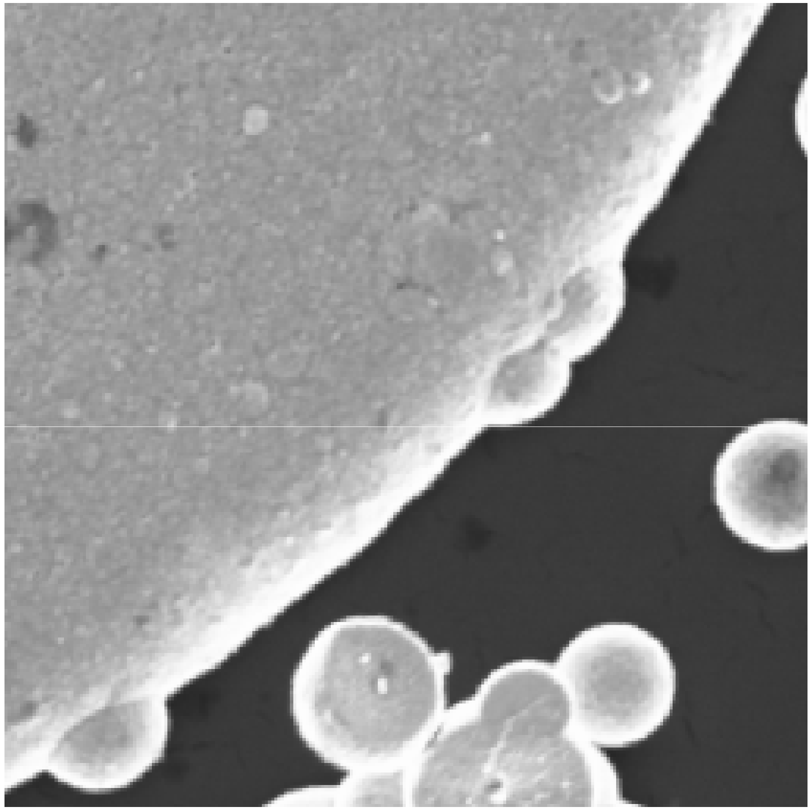
Differential phase contrast



Thibault *et al.*, Science, **321**, 379-382 (2008).

Scanning X-ray diffraction microscopy

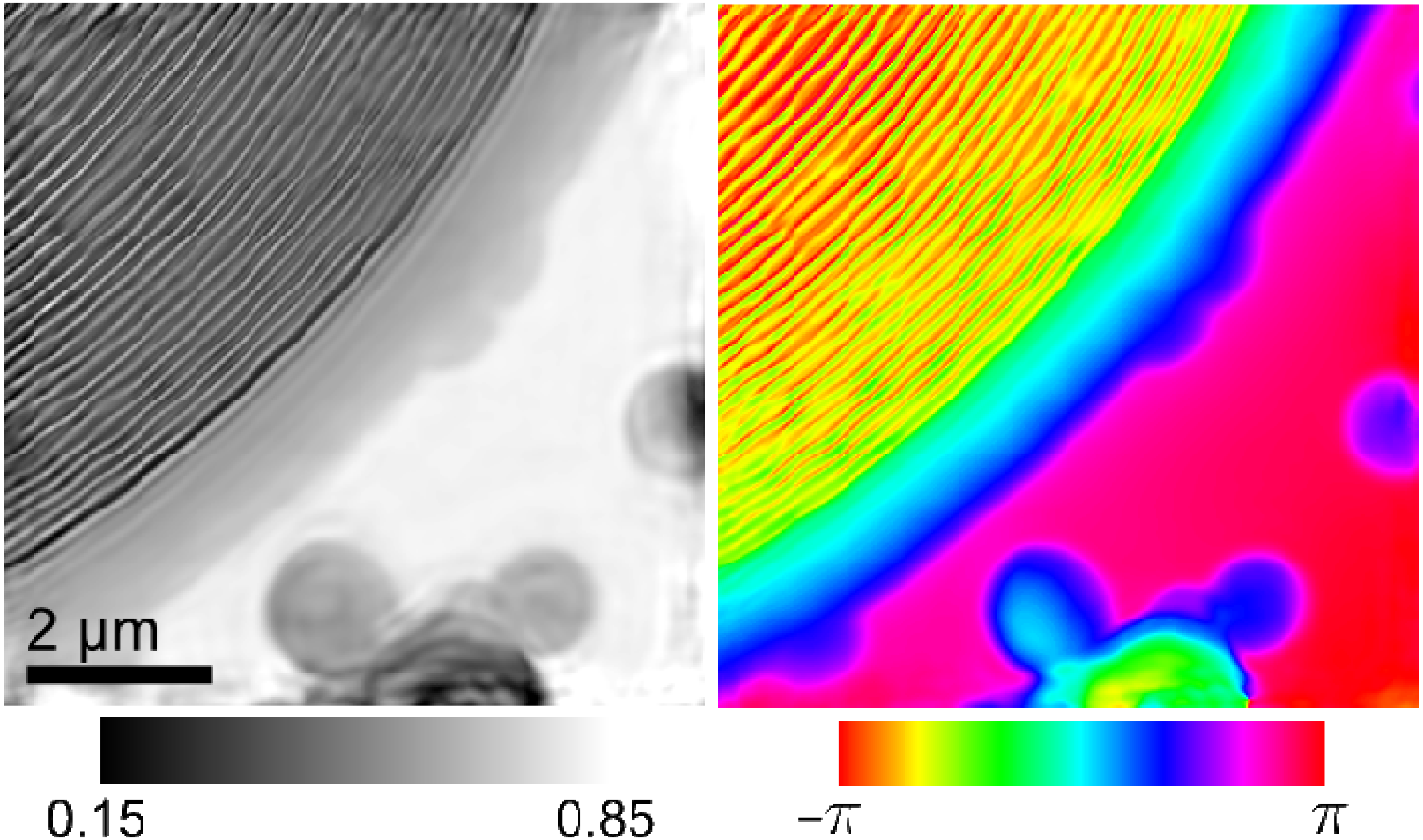
Reconstruction of a selected region



Thibault *et al.*, Science, **321**, 379-382 (2008).

Scanning X-ray diffraction microscopy

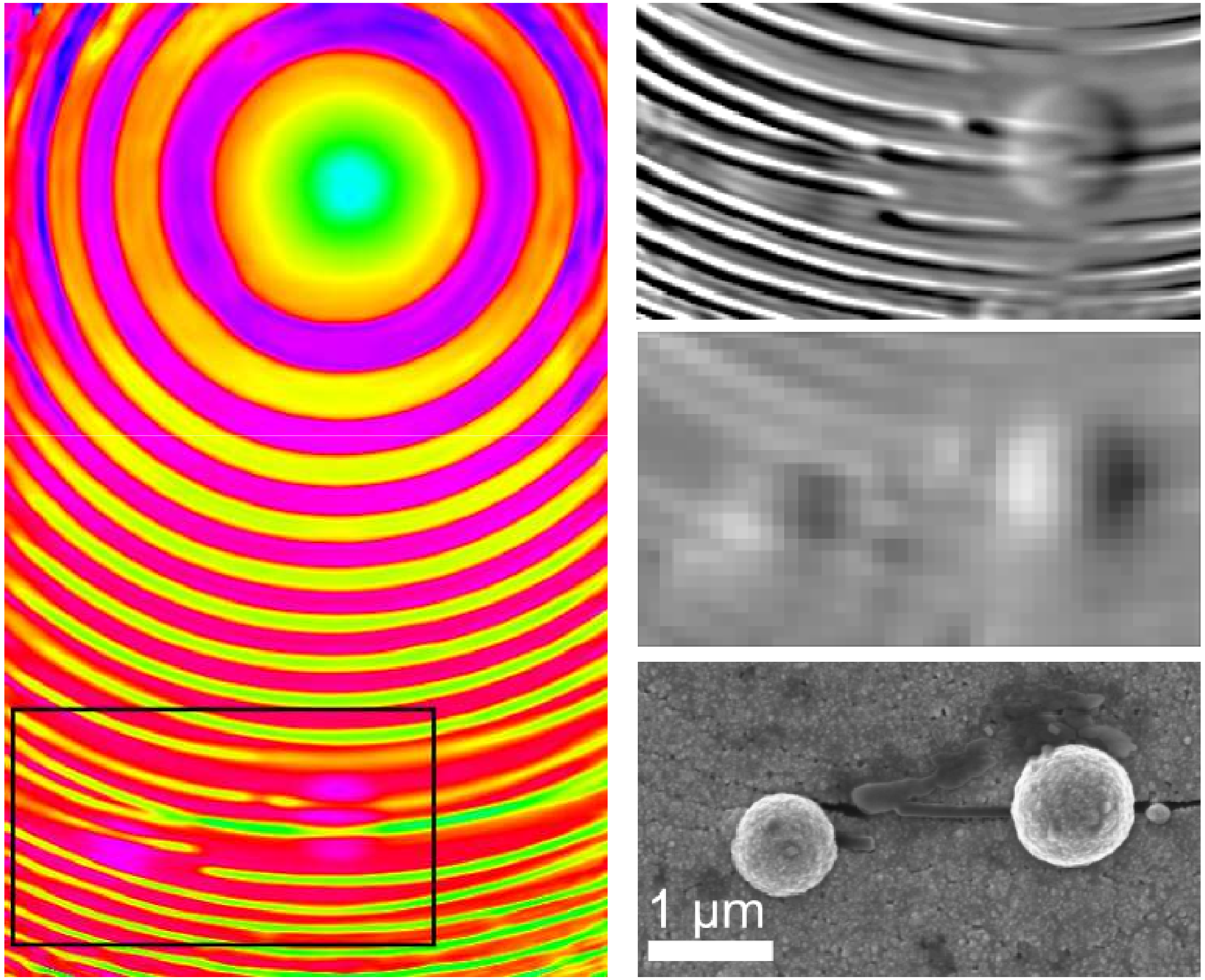
Reconstruction of a selected region



Thibault *et al.*, Science, **321**, 379-382 (2008).

Scanning X-ray diffraction microscopy

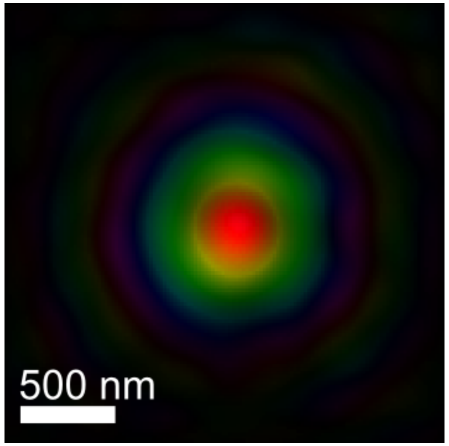
Resolution enhancement



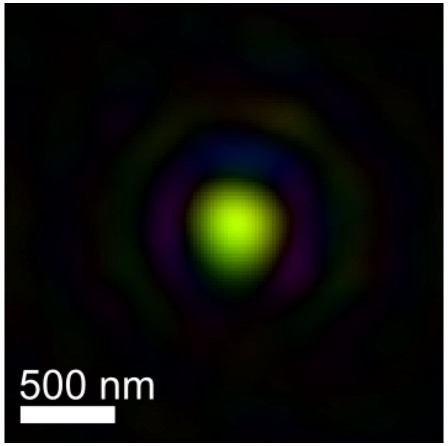
Thibault *et al.*, Science, **321**, 379-382 (2008).

Scanning X-ray diffraction microscopy

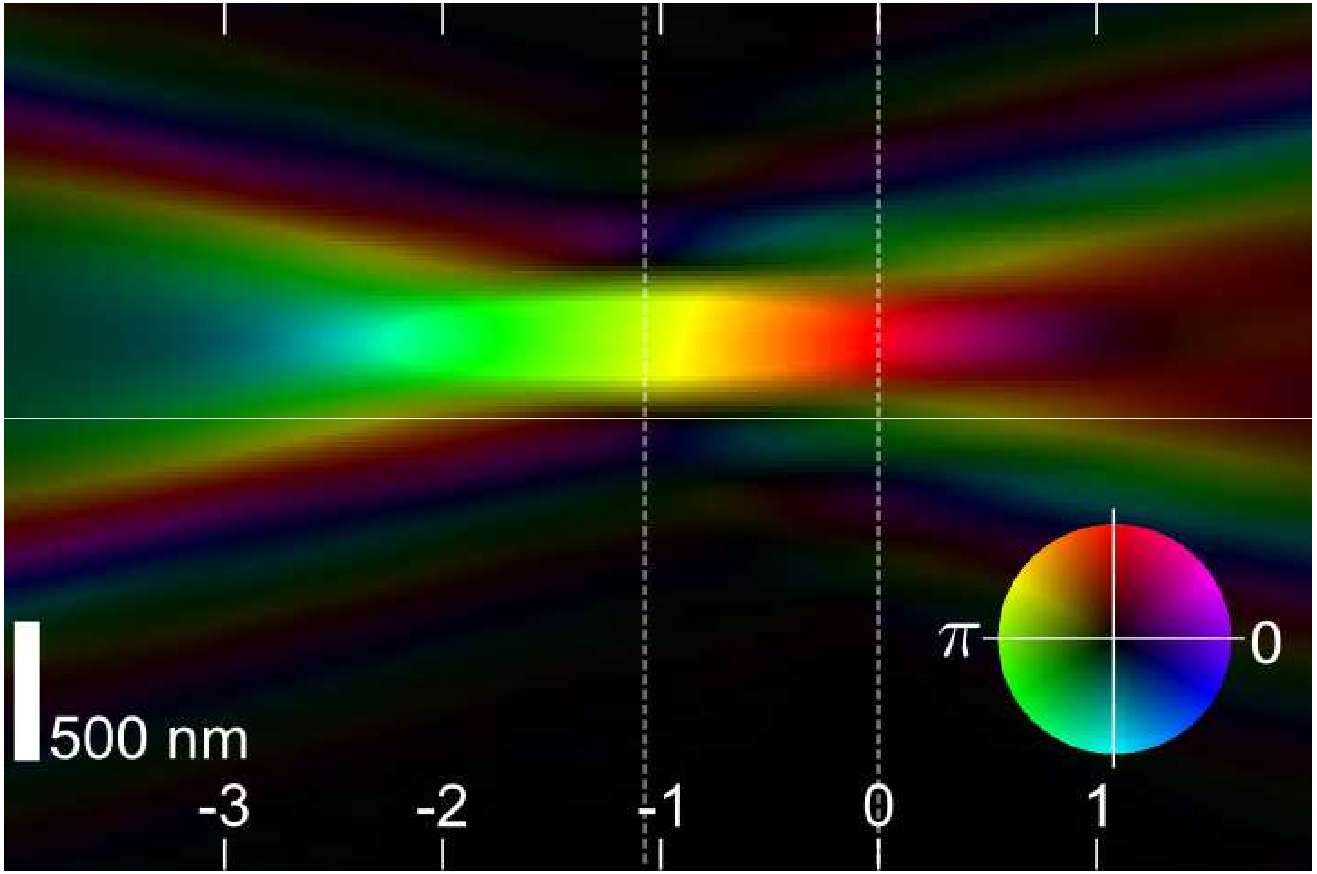
simultaneous retrieval of the probe



probe



propagated in the focal plane



propagation distance (mm)

Thibault *et al.*, Science, **321**, 379-382 (2008).

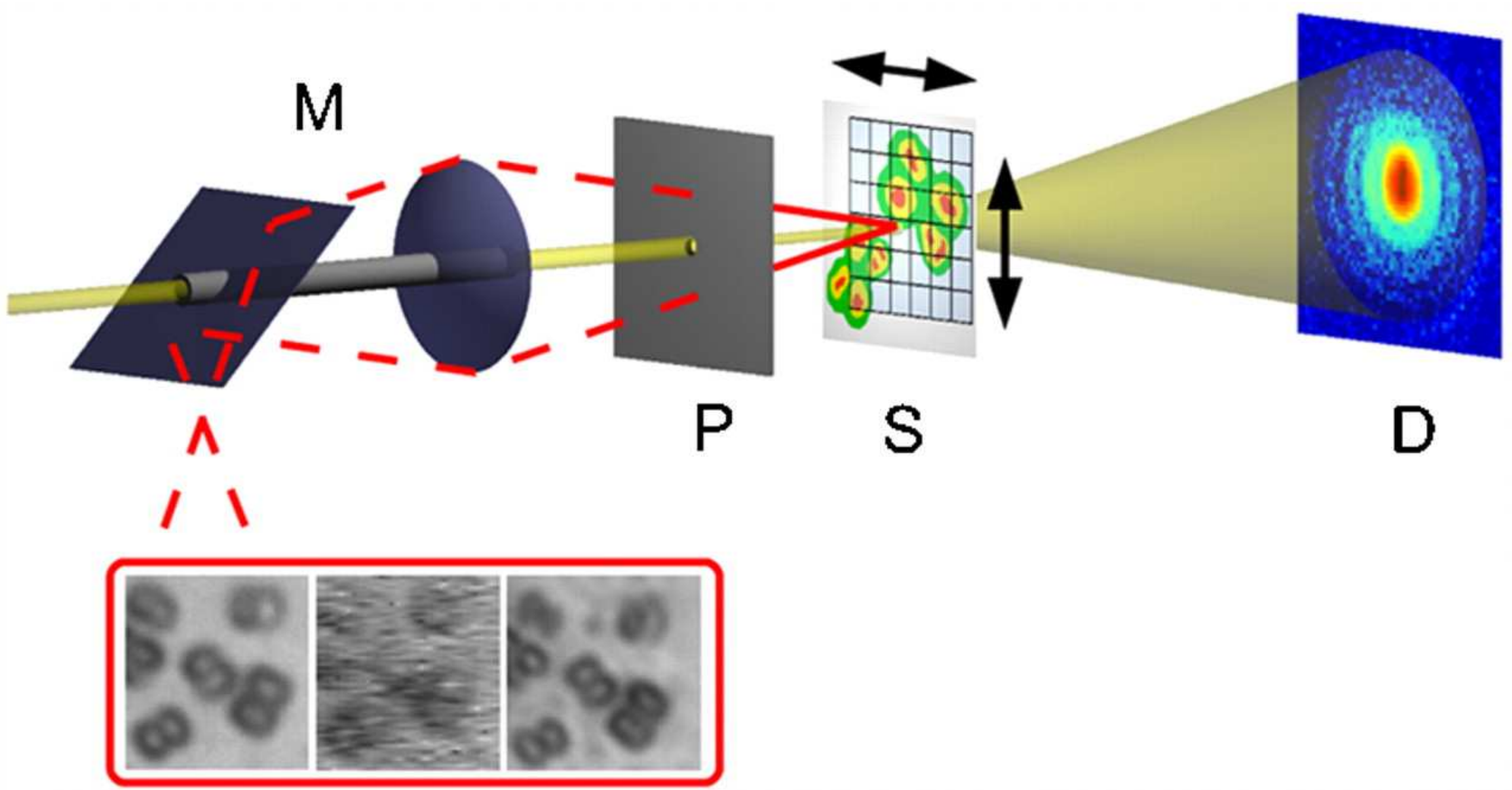
Scanning X-ray diffraction microscopy

benefits

- High-resolution imaging of extended specimens
- Compatible with other scanning techniques
- A step towards “optimal imaging”

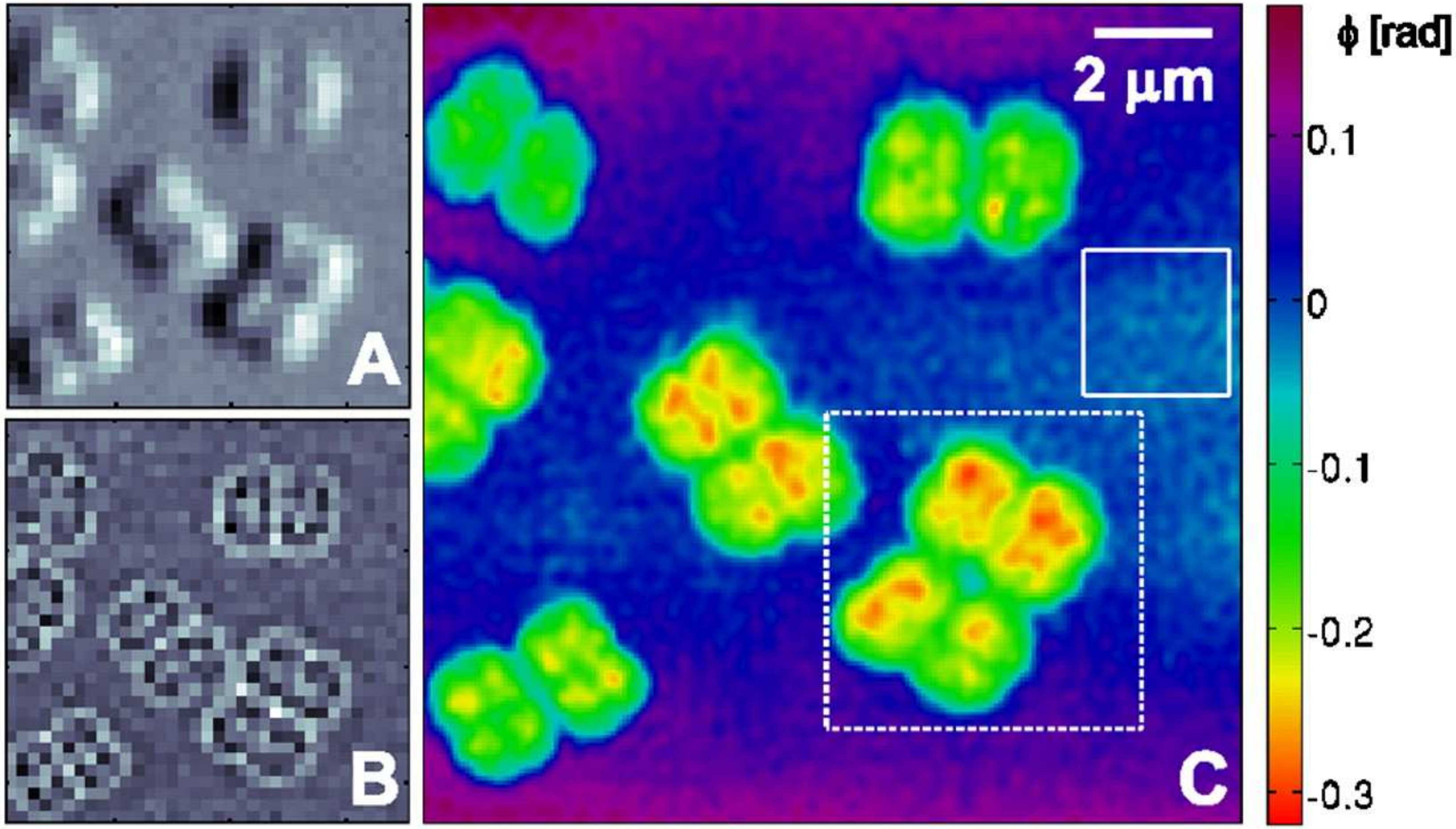
Thibault *et al.*, Science, **321**, 379-382 (2008).

Experimental setup.



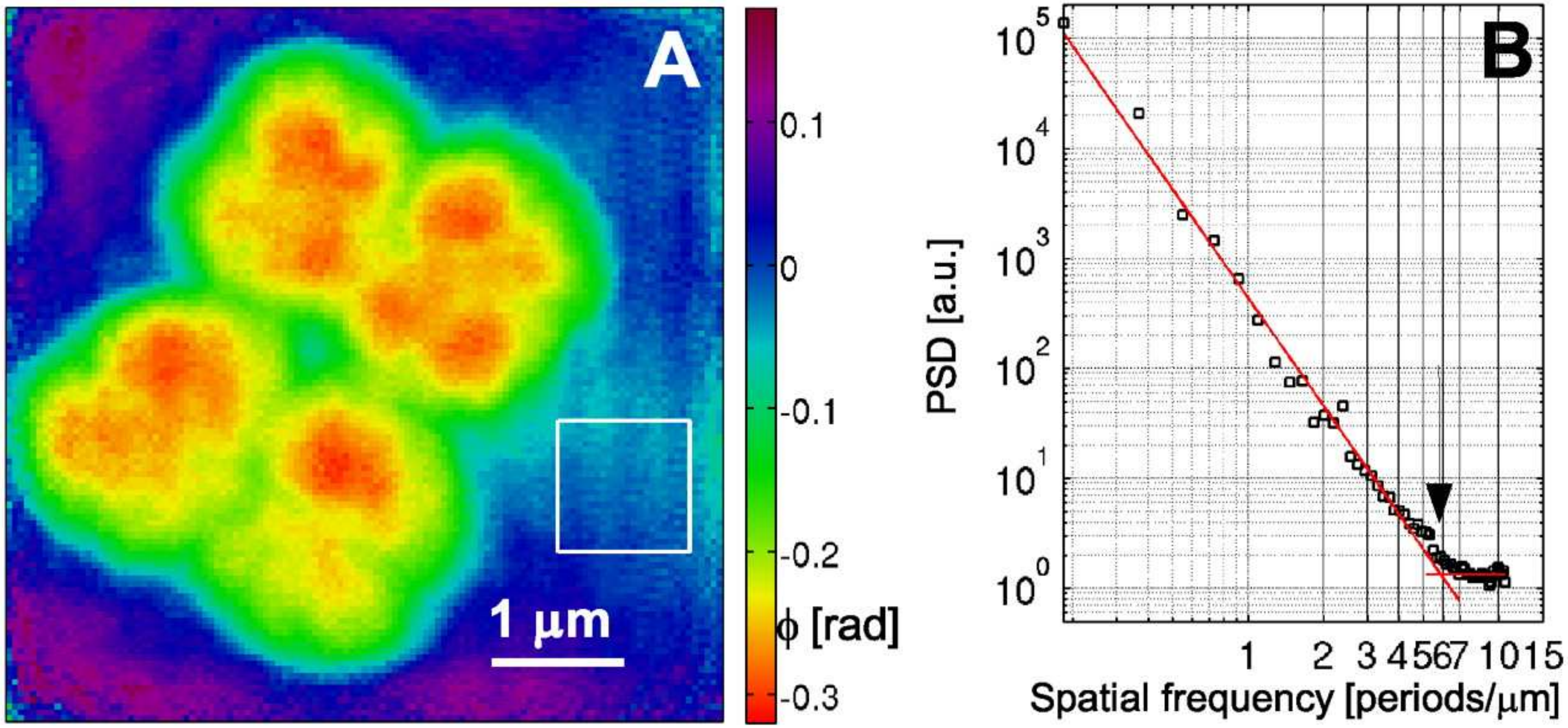
Giewekemeyer K et al. PNAS 2010;107:529-534

Experimental setup.



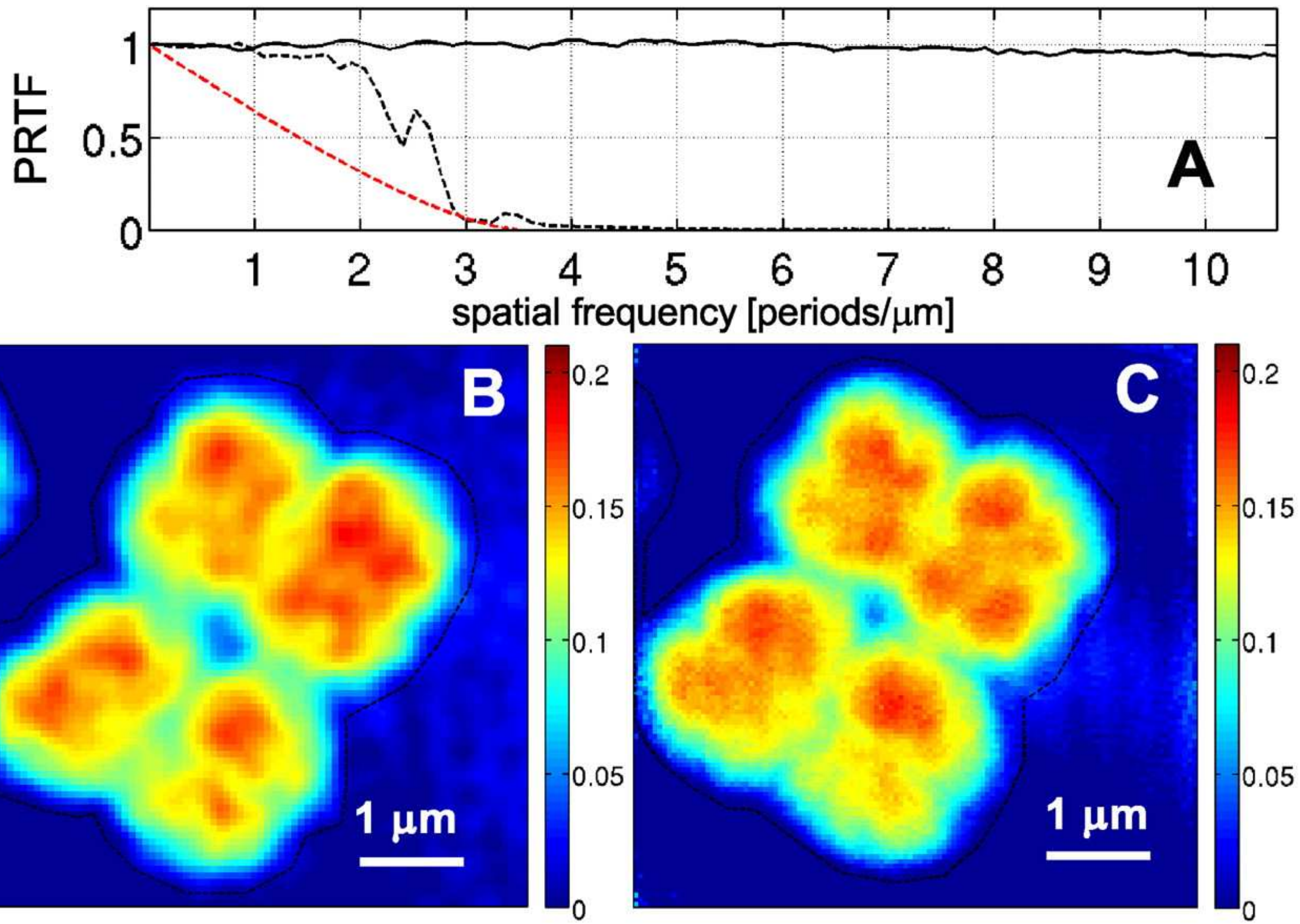
Giewekemeyer K et al. PNAS 2010;107:529-534

Experimental setup.



Giewekemeyer K et al. PNAS 2010;107:529-534

(A) PRTF, corresponding to the dataset with 1-s dwell time (black dashed line) and 60-s dwell time (solid line).



Giewekemeyer K et al. PNAS 2010;107:529-534

99% work by others!

- Thanks for Maya and the other organizers
- Thanks to my collaborators
- Trying to be publicist for a lot of smart people…