The Abdus Salam International Centre for Theoretical Physics

United Nations Educational, Scientific and Cultural Organization
IAEA

2332-27

School on Synchrotron and FEL Based Methods and their Multi-Disciplinary Applications

19 - 30 March 2012

Lens vs. lensless imaging using synchrotrons and applications

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Lens vs. lensless imaging using synchrotrons and applications

Janos Kirz
ALS

- The message:
  - Lens-based microscopy is now routine - both TXM and STXM.
  - XRF will not go lensless
  - PEEM, SPEM will not go lensless
  - Lensless needs coherence, more challanging at today's synchrotrons (ERL, USR, FEL)
  - Ptychographic imaging and tomography making rapid progress

Lens based vs lensless

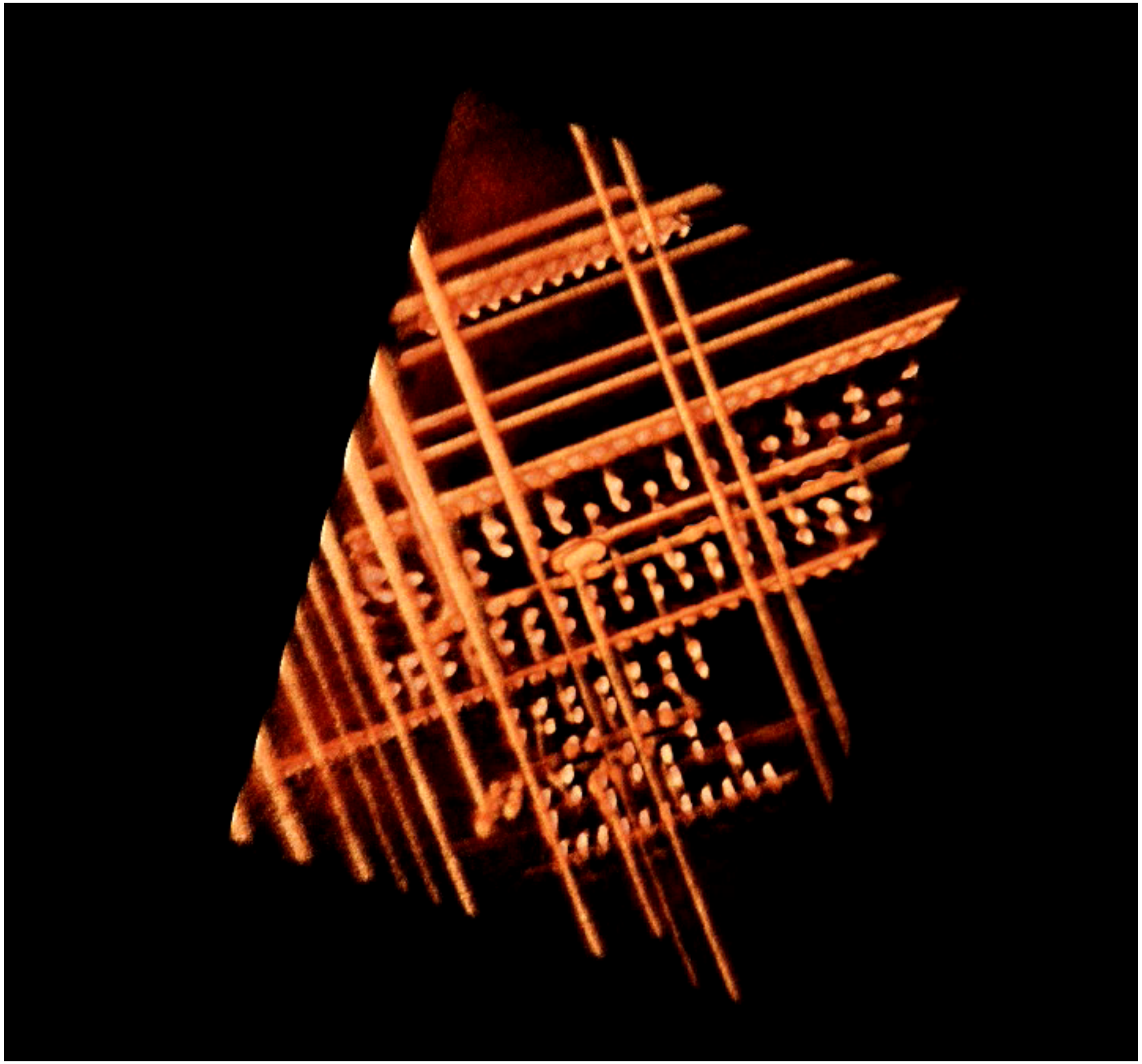
- Lens based
  - covered by previous lectures
- TXM tomography – routine at lightsources, commercially available for lab-installations too (XRADIA)

TXM tomography

- Movie

from XRADIA

Toshiba semiconductor
Packaging
~30 micron field,
~50 nm resolution

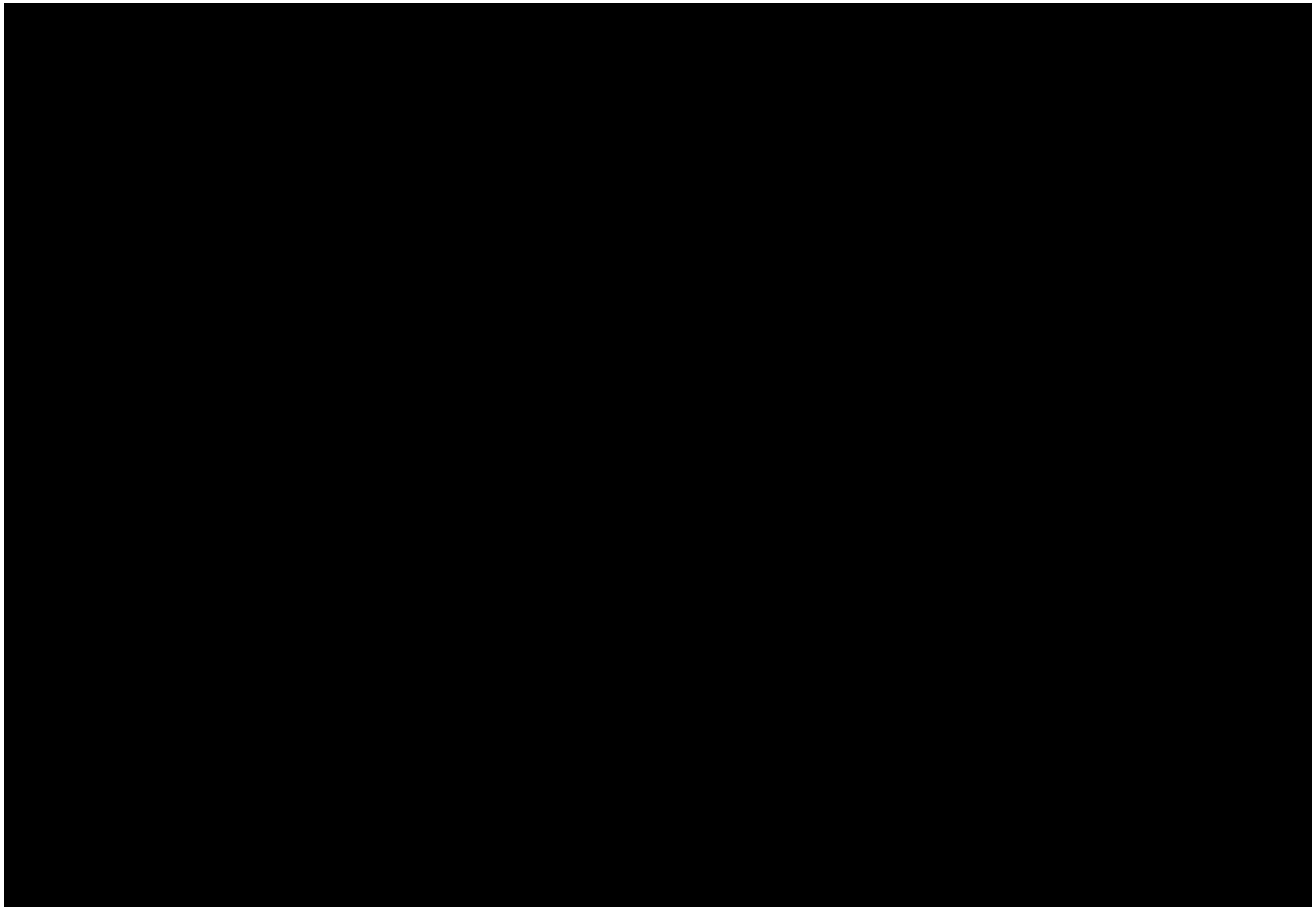


TXM tomography

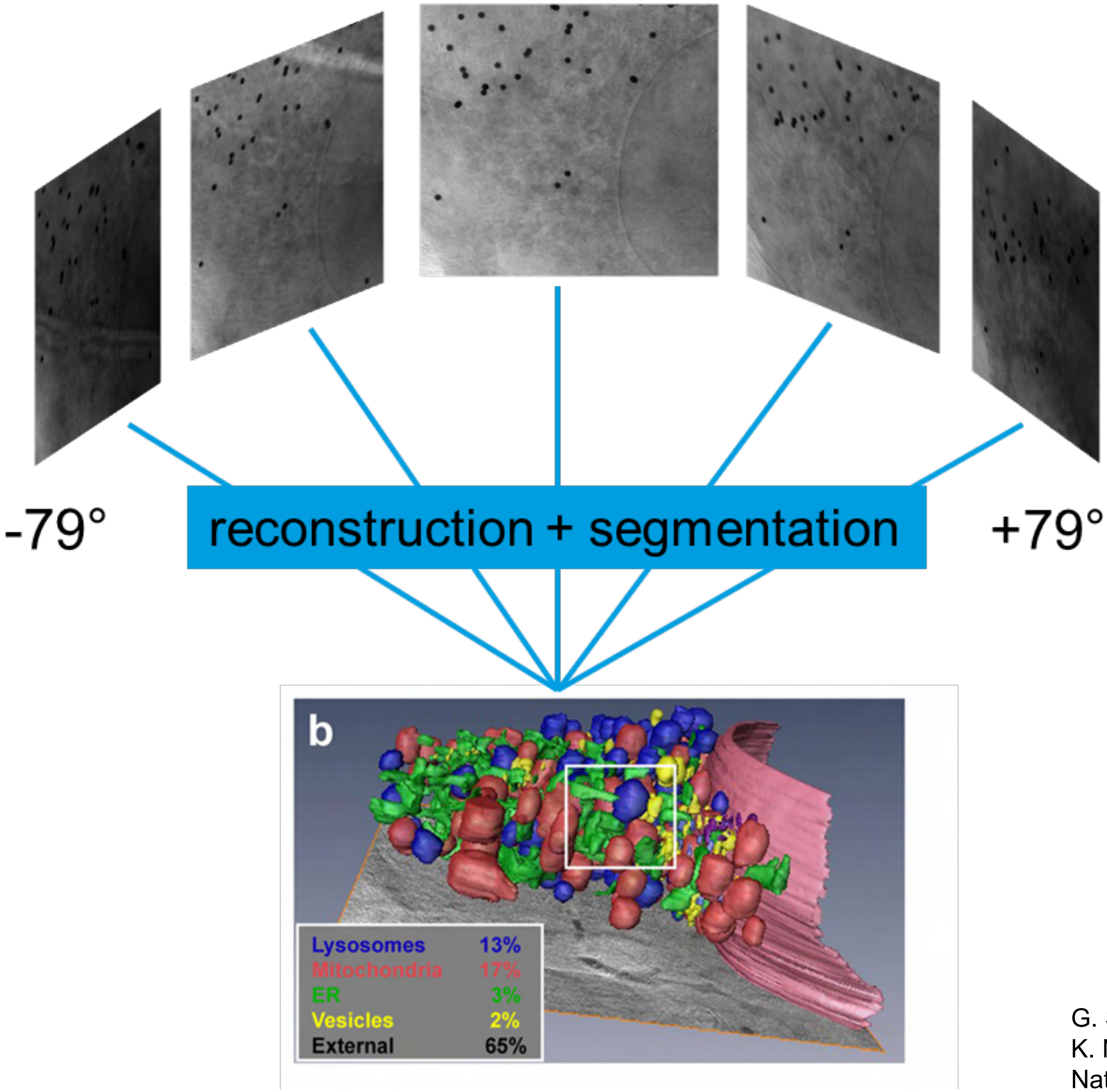
- Movie from Marco Stampanoni, SLS
- Zernike phase contrast tomography of MC3 preosteoblast

TOMCAT Nanoscope
- 10 keV
- Pixel Size 70 nm
- True 3D res: ~ 200 nm
- 20 micron FOV
- High penetration power !
- High depth of focus !
- No vacuum
- No cryo-cooling
- Low dose deposition

Phys. Rev B 81, 140105 (2010)



Nanoscale Tomography of Cells



automated tomography system:

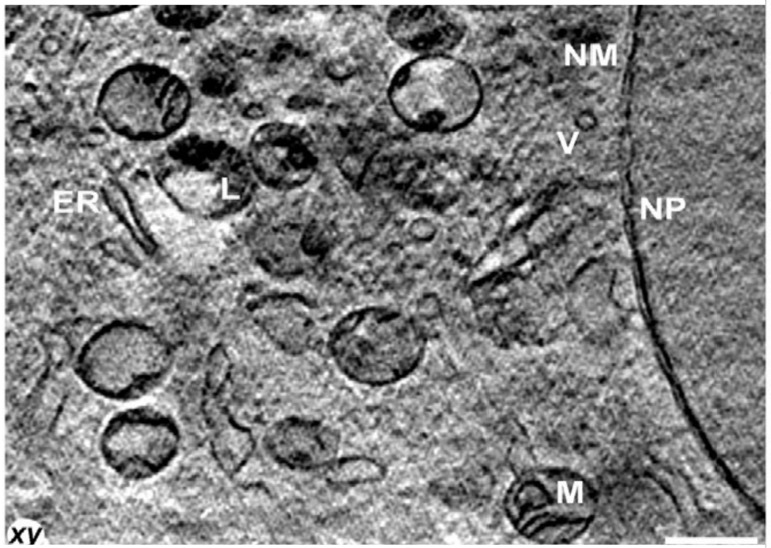
- about 400 projections in 20 – 30 min exposure time
- significantly increased 3-D resolution
- small angular steps permit automated image alignment using cross correlation or phase correlation

G. Schneider, P. Guttmann, S. Heim, S. Rehbein, F. Mueller, K. Nagashima, J.B. Heymann, W.G. Müller, J.G. McNally, Nature Methods 7 (2010), 985-987

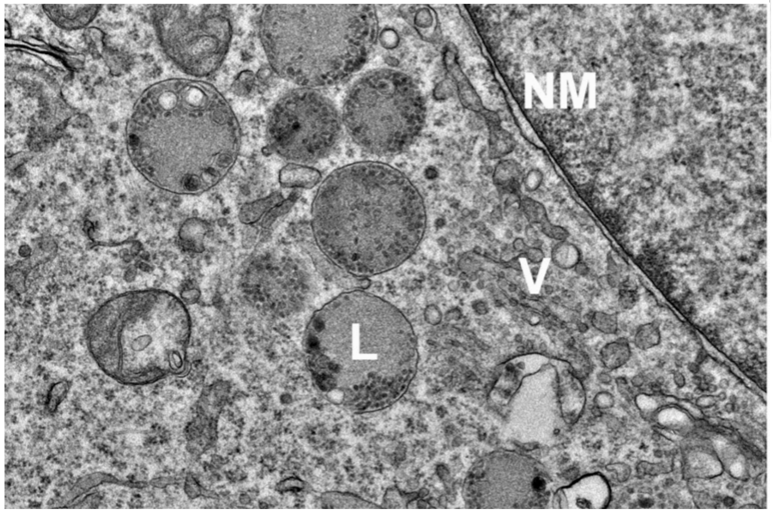
X-ray tomography: Slices show ultrastructure of adenocarcinoma mouse cell

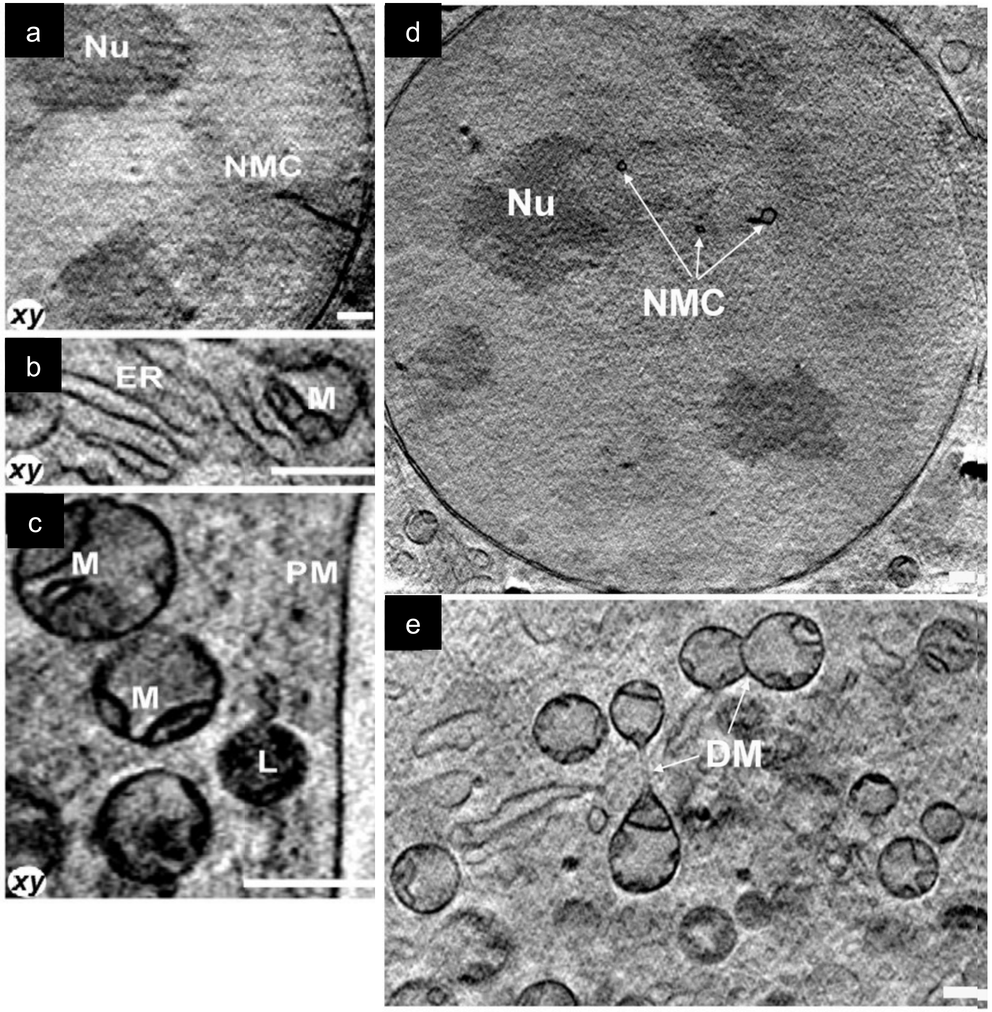
HZB Helmholtz Zentrum Berlin

X-ray tomography: Whole cell with native contrast



TEM thin section: Dried, plastic embedded and stained





Nu (Nucleoli)

NM (Nuclear membrane)

L (Lysosomes)

NP (Nuclear pores)

V (Vesicles)

PM (Plasma membrane)

NMC (Nuclear membrane channels)

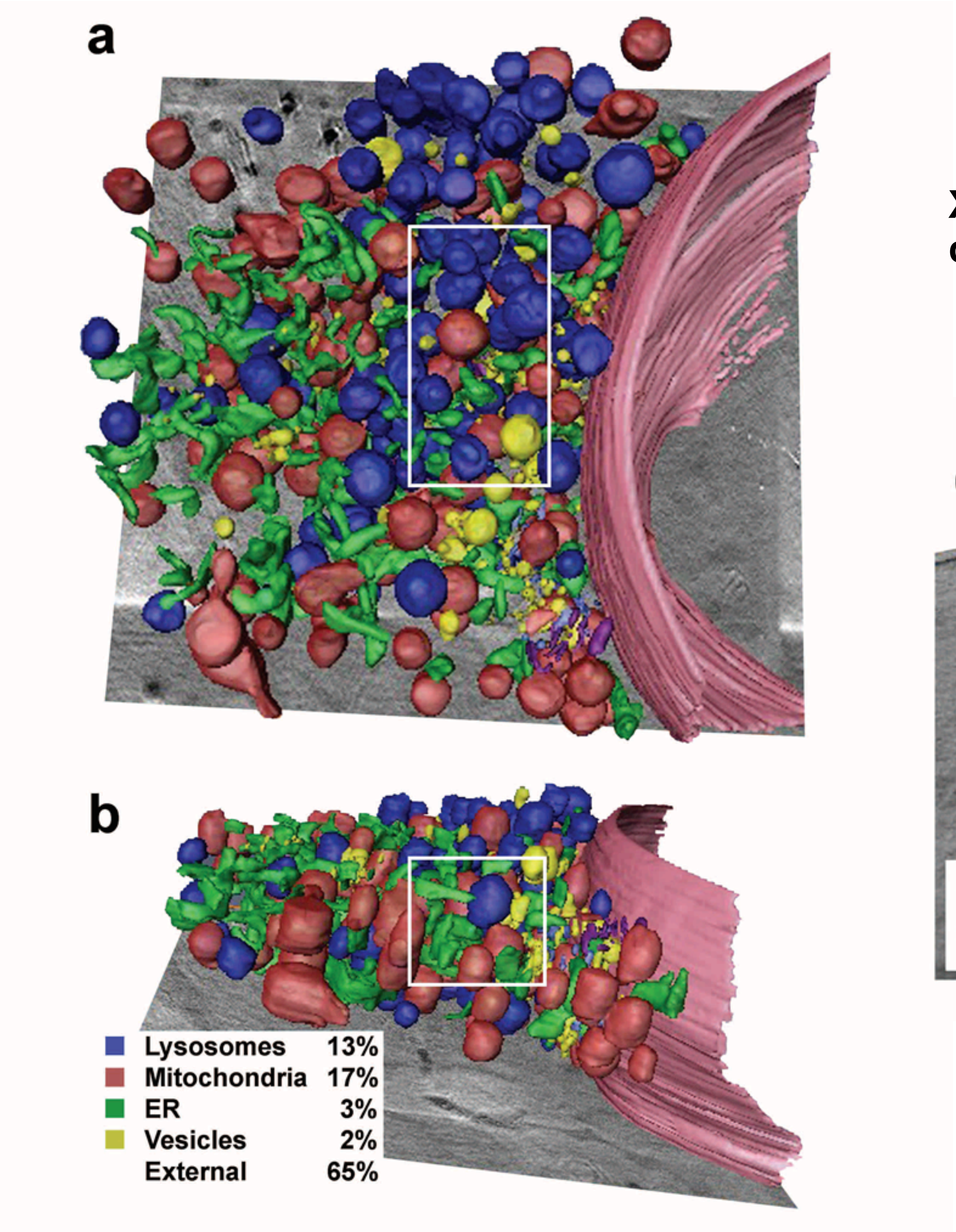
ER (Endoplasmatic reticulum)

M (Mitochondria)

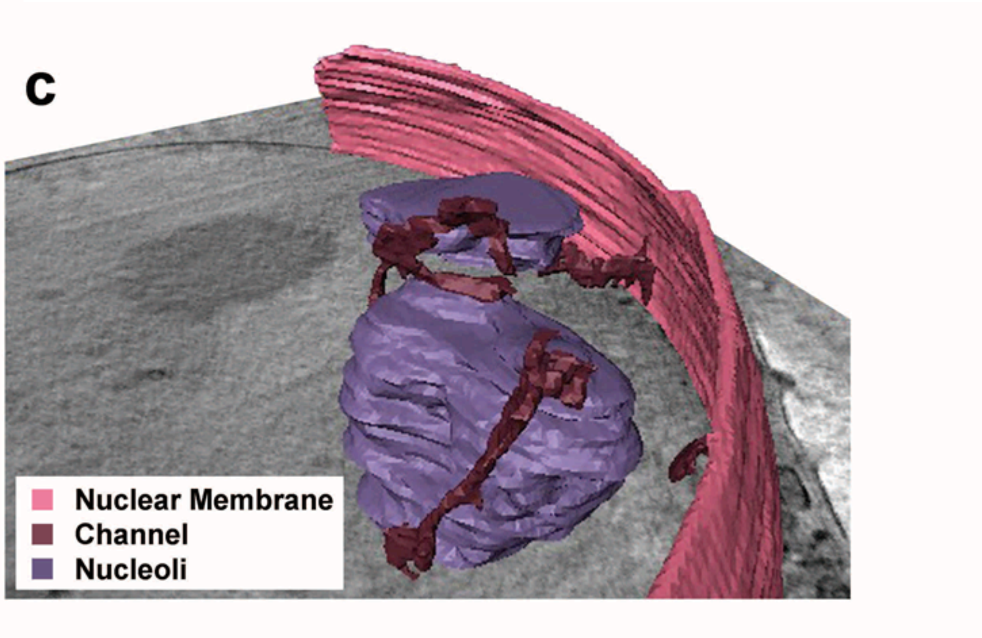
Thickness of the tomographic slices: 20 nm,
Scale bars: 0.78 µm

G. Schneider, P. Guttmann, S. Heim, S. Rehbein, F. Mueller, K. Nagashima, J.B. Heymann, W.G. Müller, J.G. McNally, Nature Methods 7 (2010), 985-987

Segmentation of the cellular volume of mouse cells



X-ray nanotomography visualizes the packing density of different organelles in the cytoplasm



G. Schneider et al., Nature Methods **7** (2010), 985-987

STXM

- Routine at most lightsources
- Elemental mapping by X-ray fluorescence
- Chemical mapping by XANES
- 2D or 3D

STXM XRF

- Trace element mapping
- X-ray absorption and fluorescence emission: can not be done with "lensless" arrangement
- Much more sensitive than electron-probe
- Rapidly growing in popularity: better detectors, brighter nanoprobes
- Minimize background: 90 degree geometry

STXM XRF

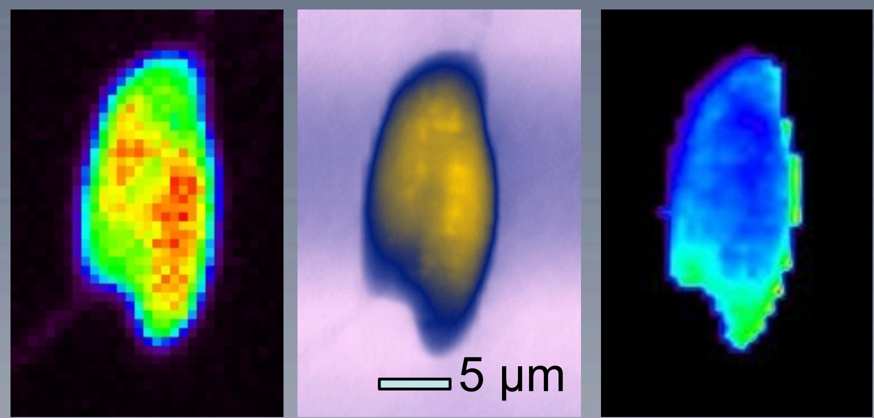
- Movie from APS

Phosphorous, Sulfur, Silicon

2 µm

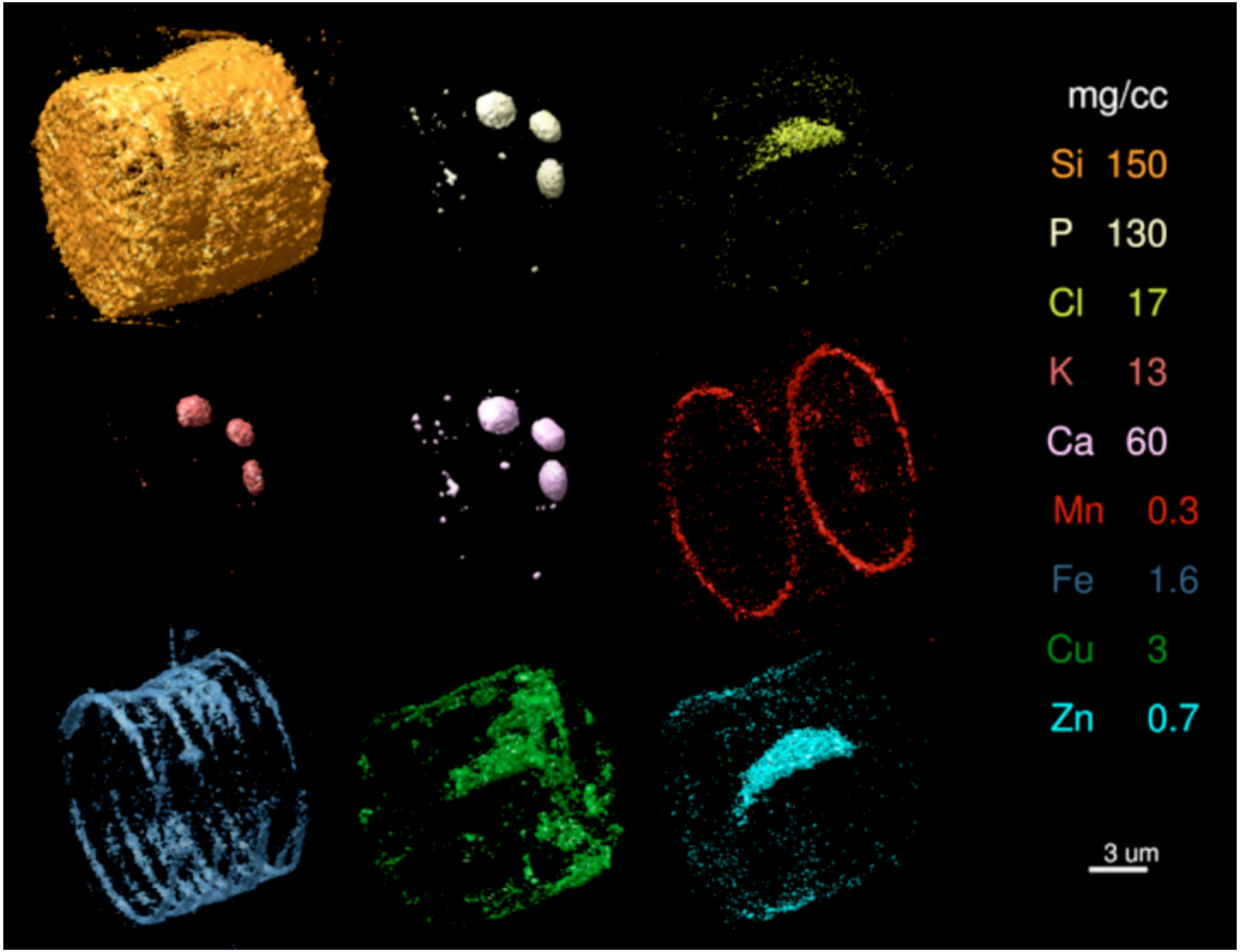
Phase contrast needed to align low-statistics fluorescence tilt series. de Jonge *et al.*, APS; Holzner *et al.*, Stony Brook

Phase contrast tomography of cyclotella (marine protist)



Sulfur content Protist mass Sulfur concentration

Quantitative 3D fluorescence of a diatom



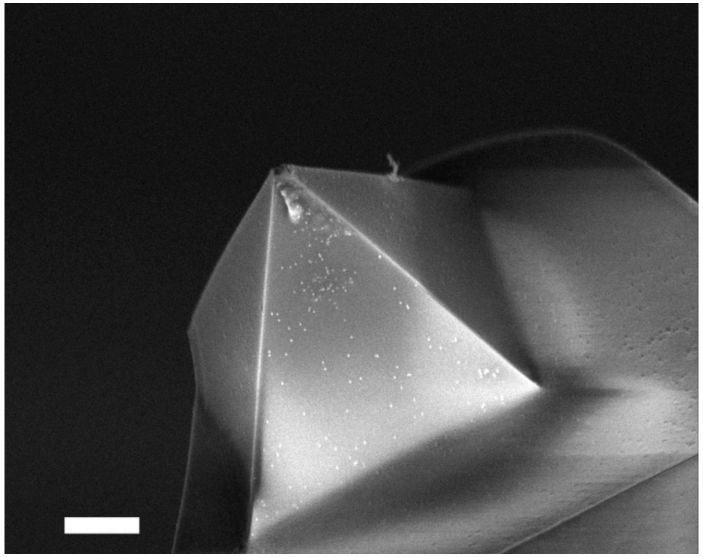
de Jonge et al, *Proc. Nat. Acad. Sci.* *107* 15676 (2010)

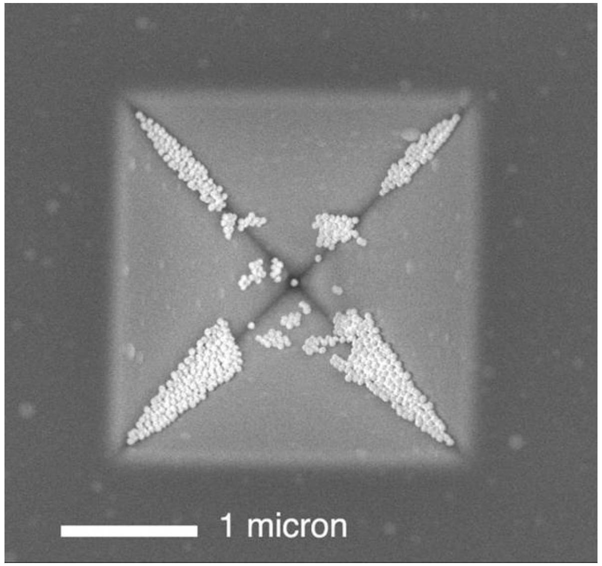
Where does lensless fit in?

- Quantitative mapping of optical constants
  - Absorption and phase shift
- Low contrast specimens
  - Hard X-ray tomography in bio: low absorption
    - (soft X-ray: low penetration, narrow depth of focus)
- Maximal info for given radiation dose
- Ideal for FELs: Diffract and destroy

3D diffraction microscopy of materials science specimens

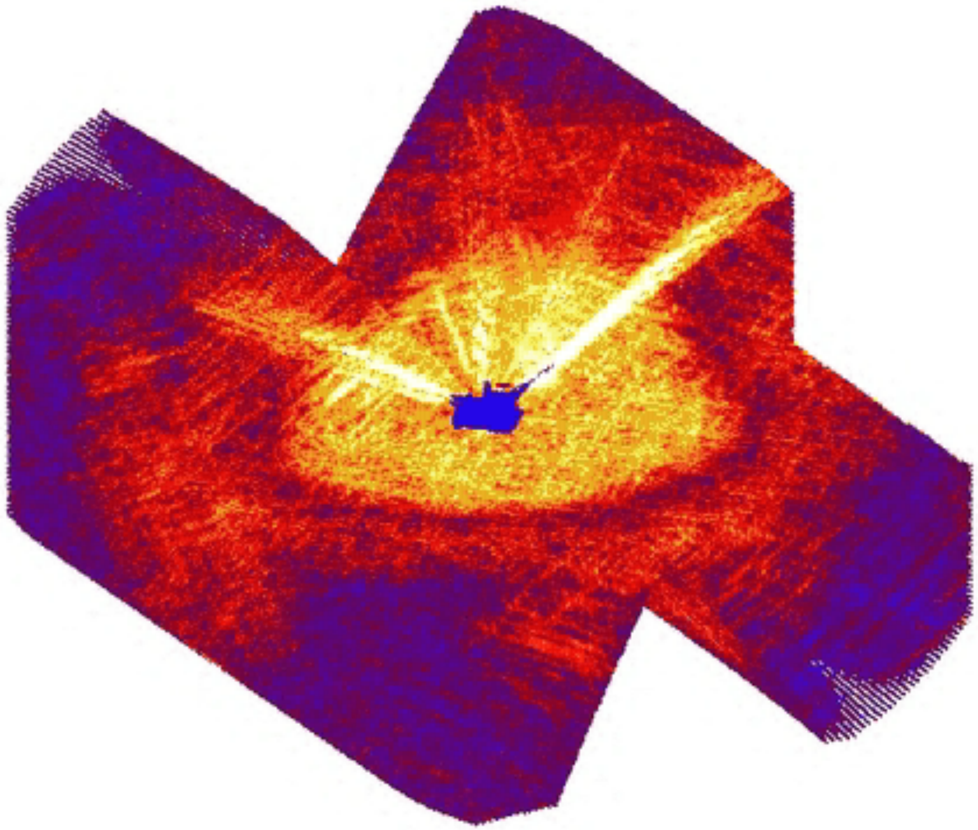
- Chapman, Barty, Marchesini, Noy, Hau-Riege, Cui, Howells, Rosen, He, Spence, Weierstall, Beetz, Jacobsen, Shapiro, *J. Opt. Soc. Am. A* **23**, 1179 (2006)
- 50 nm gold spheres placed on hollowed AFM tip "pyramid"
- Data taken using Stony Brook apparatus at ALS beamline 9.0.1





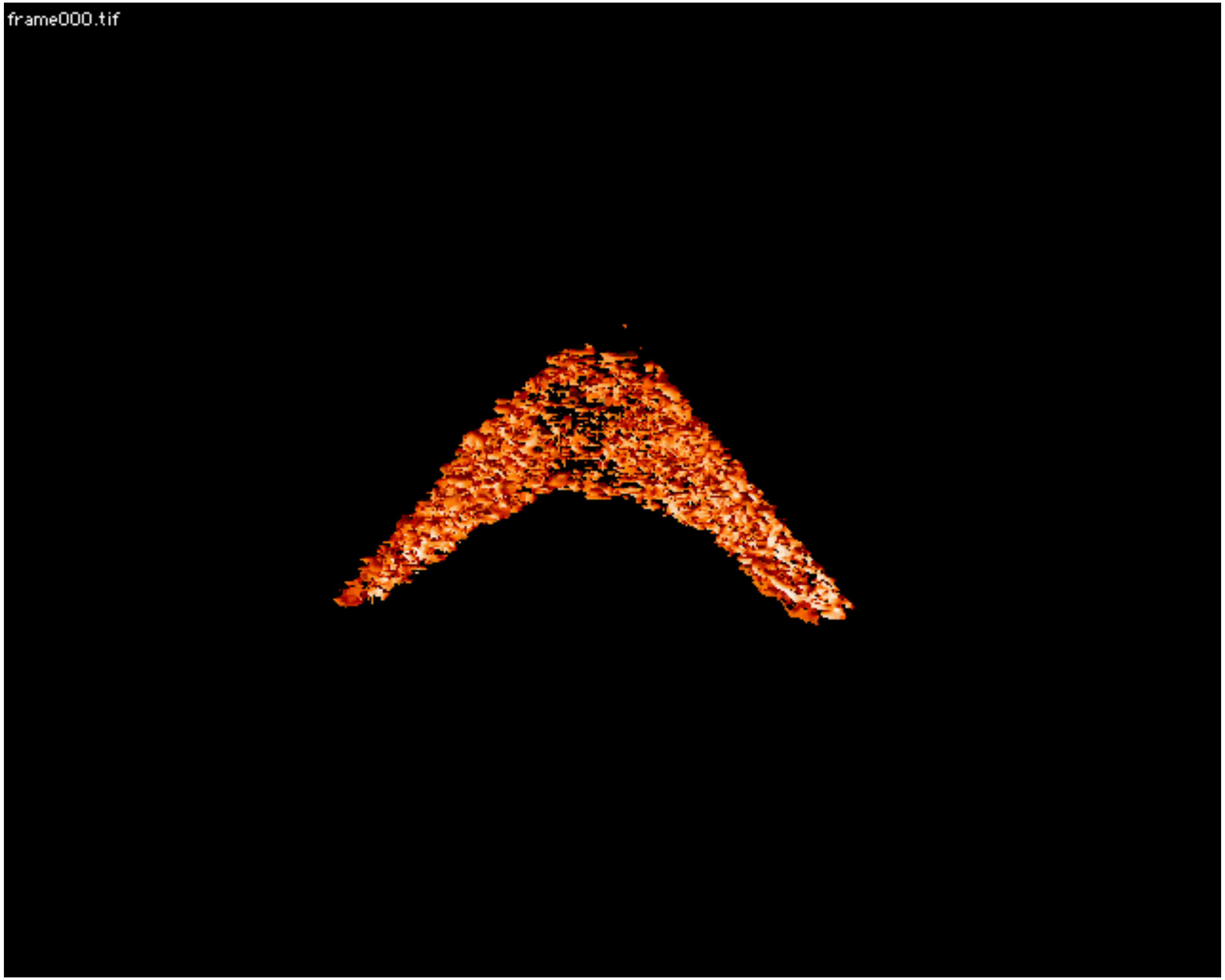
3D data cube

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



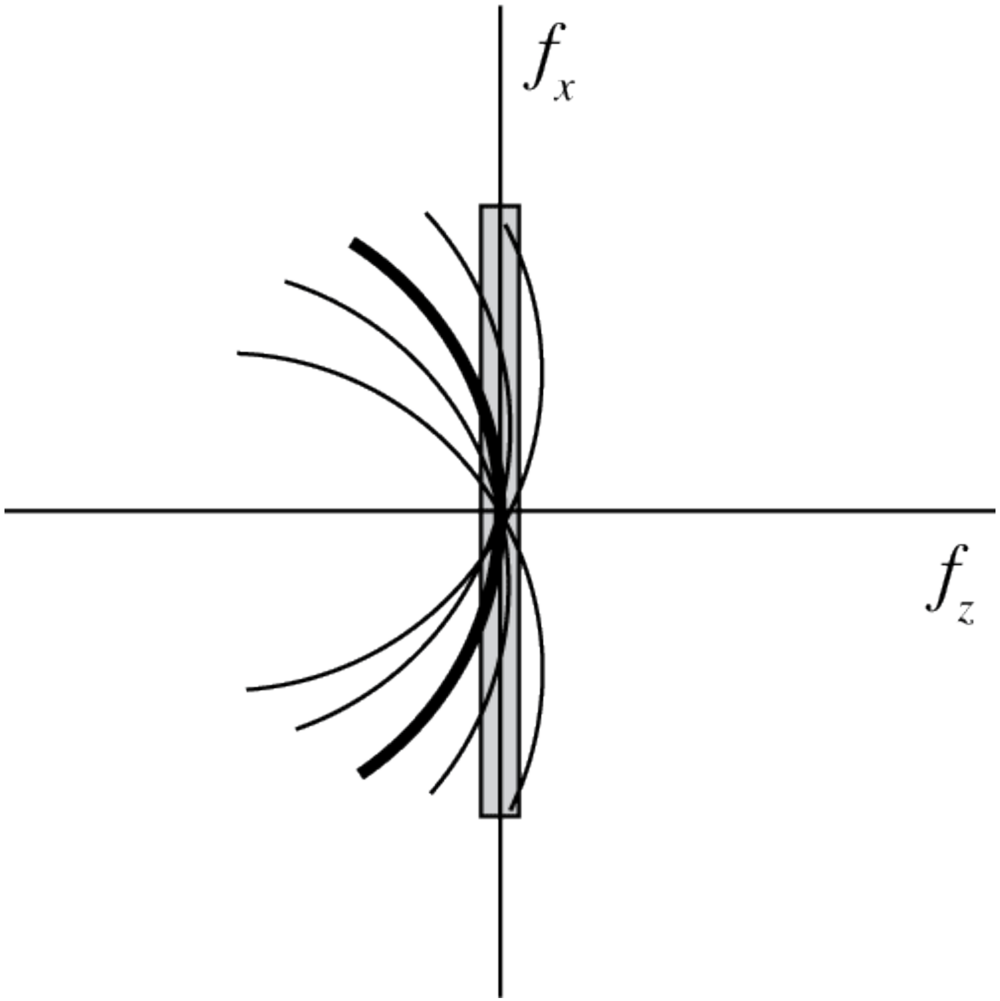
Reconstruction

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

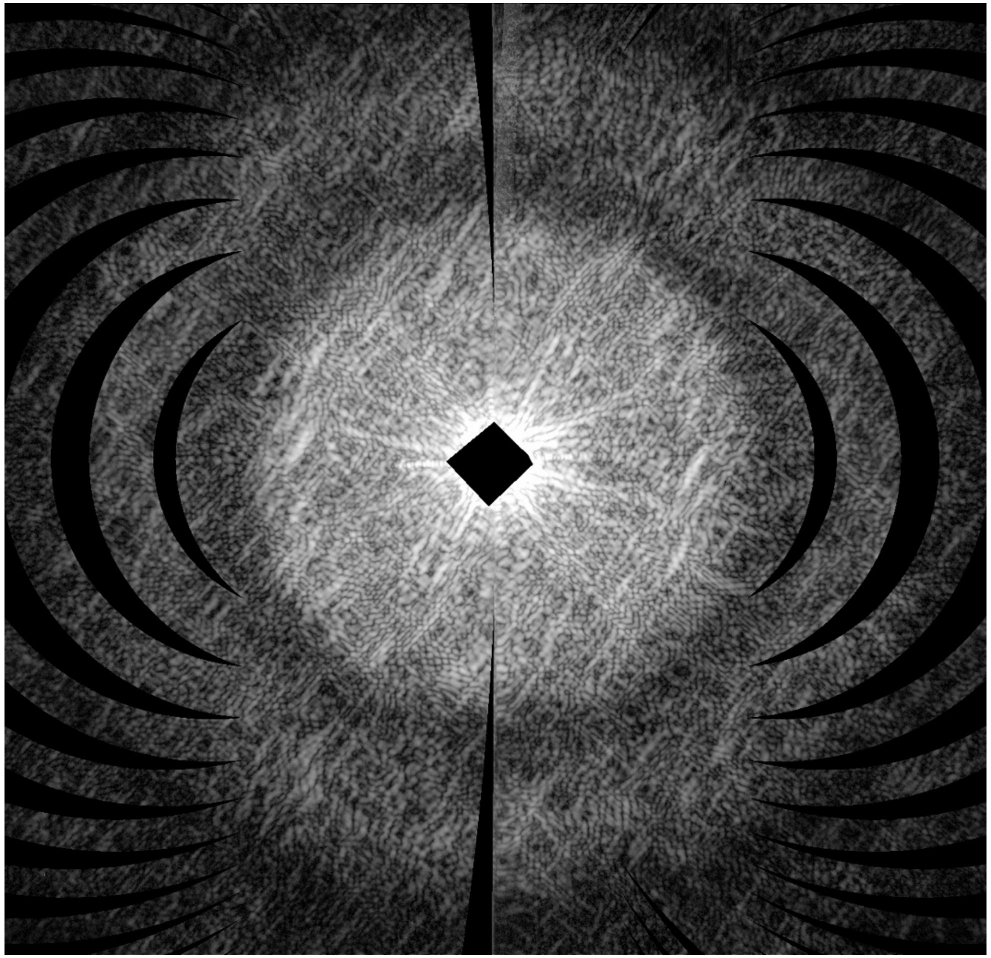


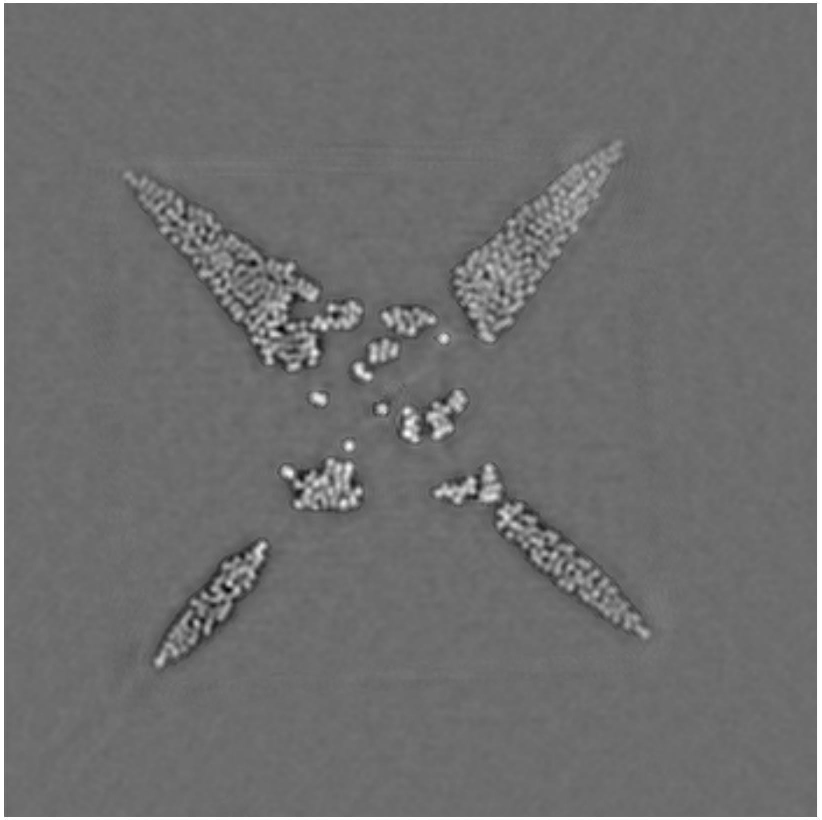
Pure projections from phased 3D data

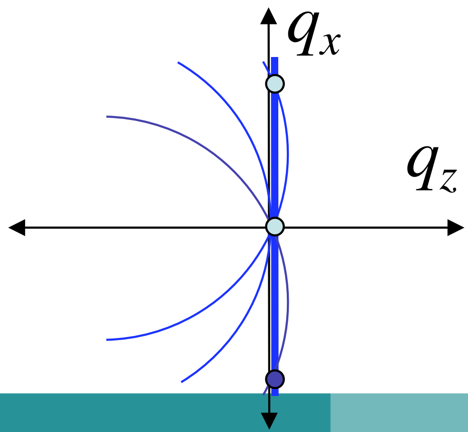
Chapman, Barty, Marchesini, Noy, Hau-Riege, Cui, Howells, Rosen, He, Spence, Weierstall, Beetz, Jacobsen, Shapiro, *J. Opt. Soc. Am. A* **23**, 1179 (2006)



Experimental realization

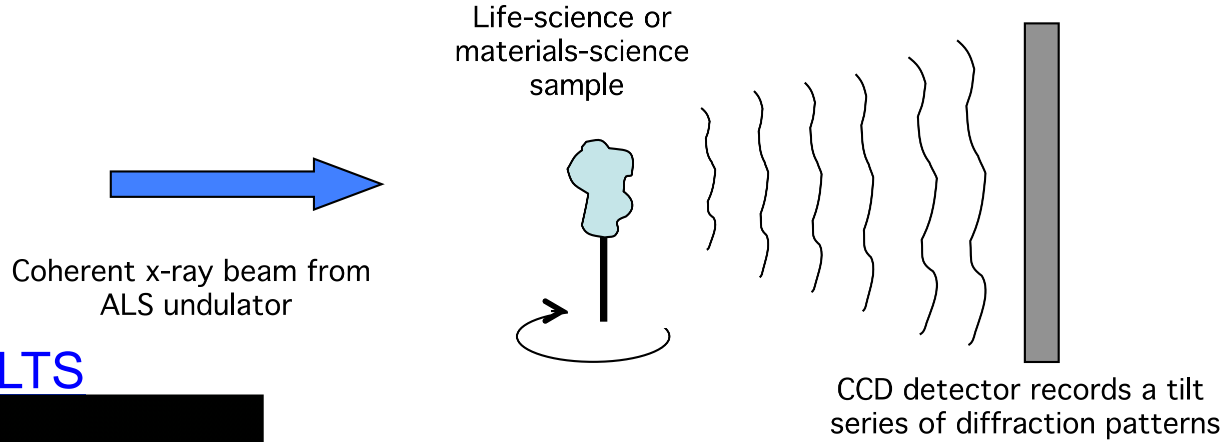




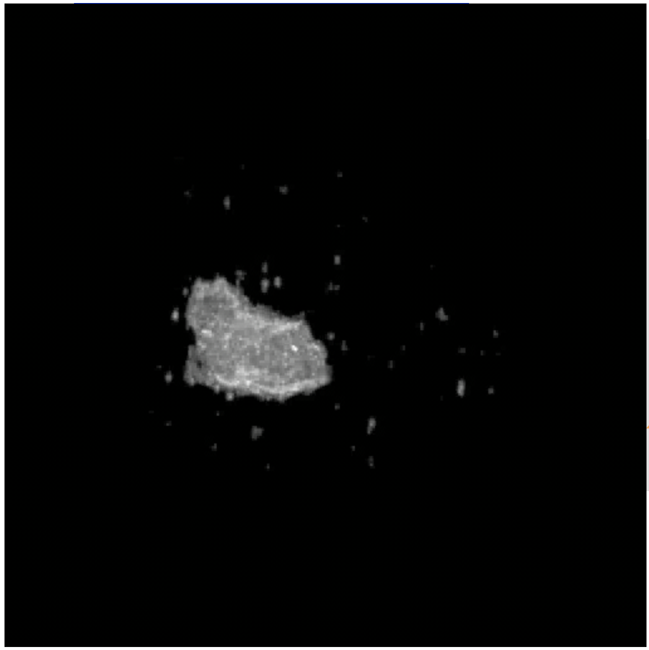


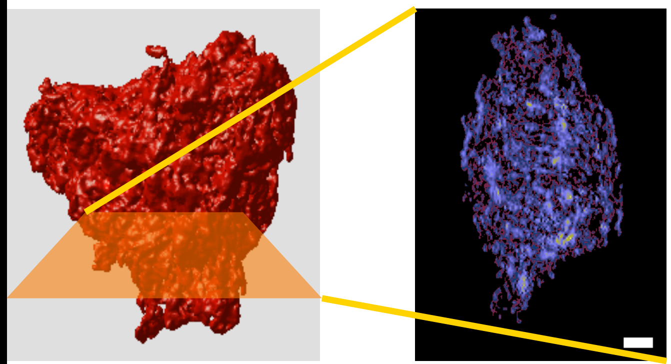
Chapman, Barty, Marchesini, Noy, Hau-Riege, Cui, Howells, Rosen, He, Spence, Weierstall, Beetz, Jacobsen, Shapiro, *J. Opt. Soc. Am. A* **23**, 1179 (2006)

A. Barty, H. Chapman, M. Howells, S. Marchesini et al., PRL 101, 055501 (2009)



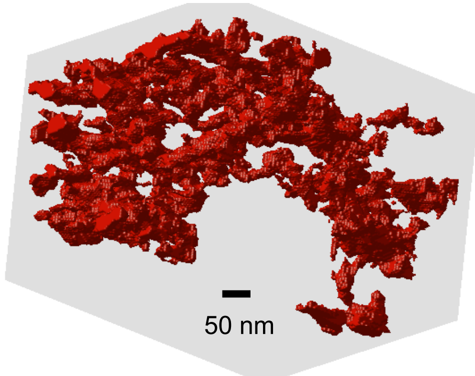
SOME RESULTS





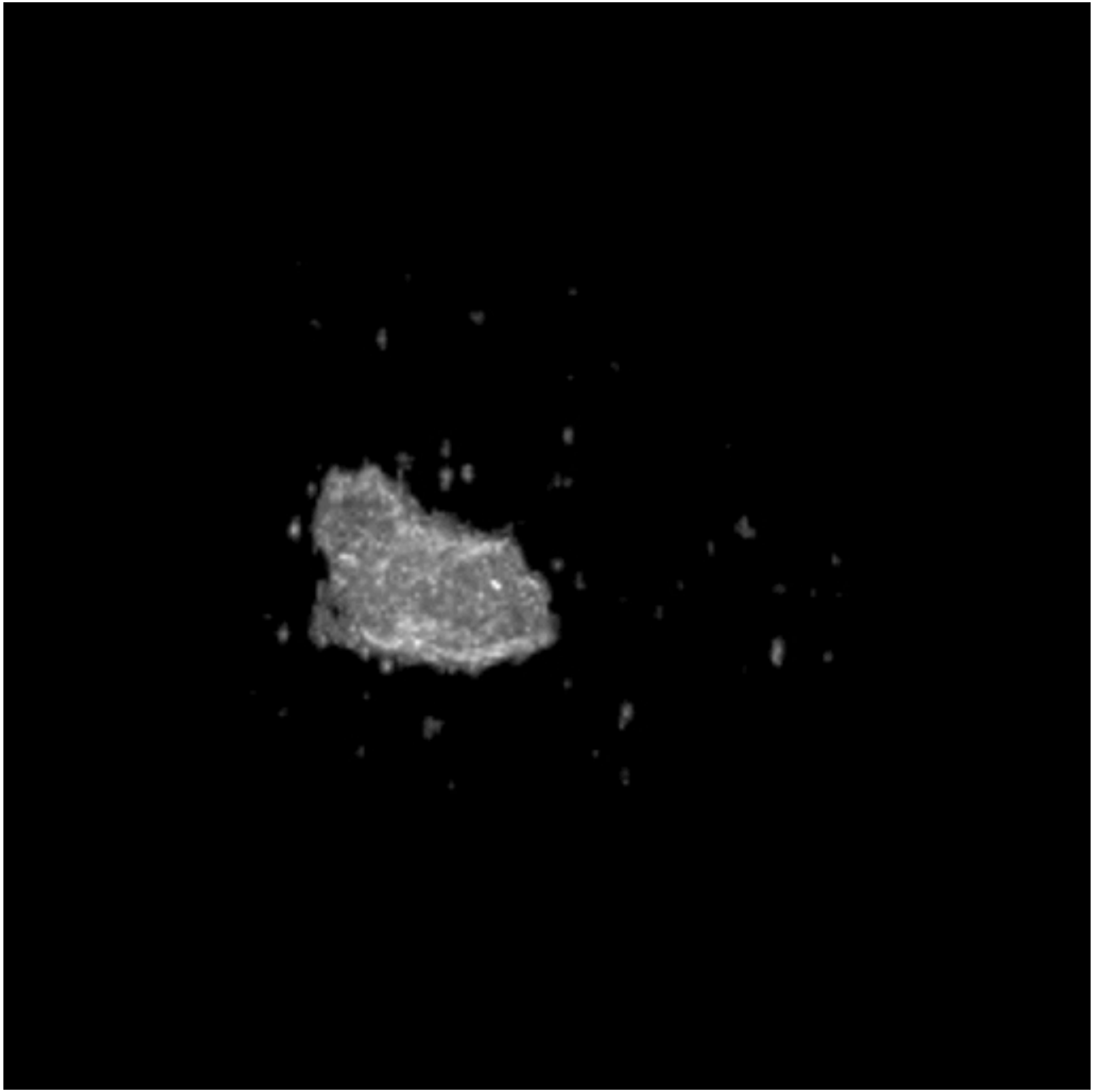
3D isosurface

Cross-section (bar = 200nm)

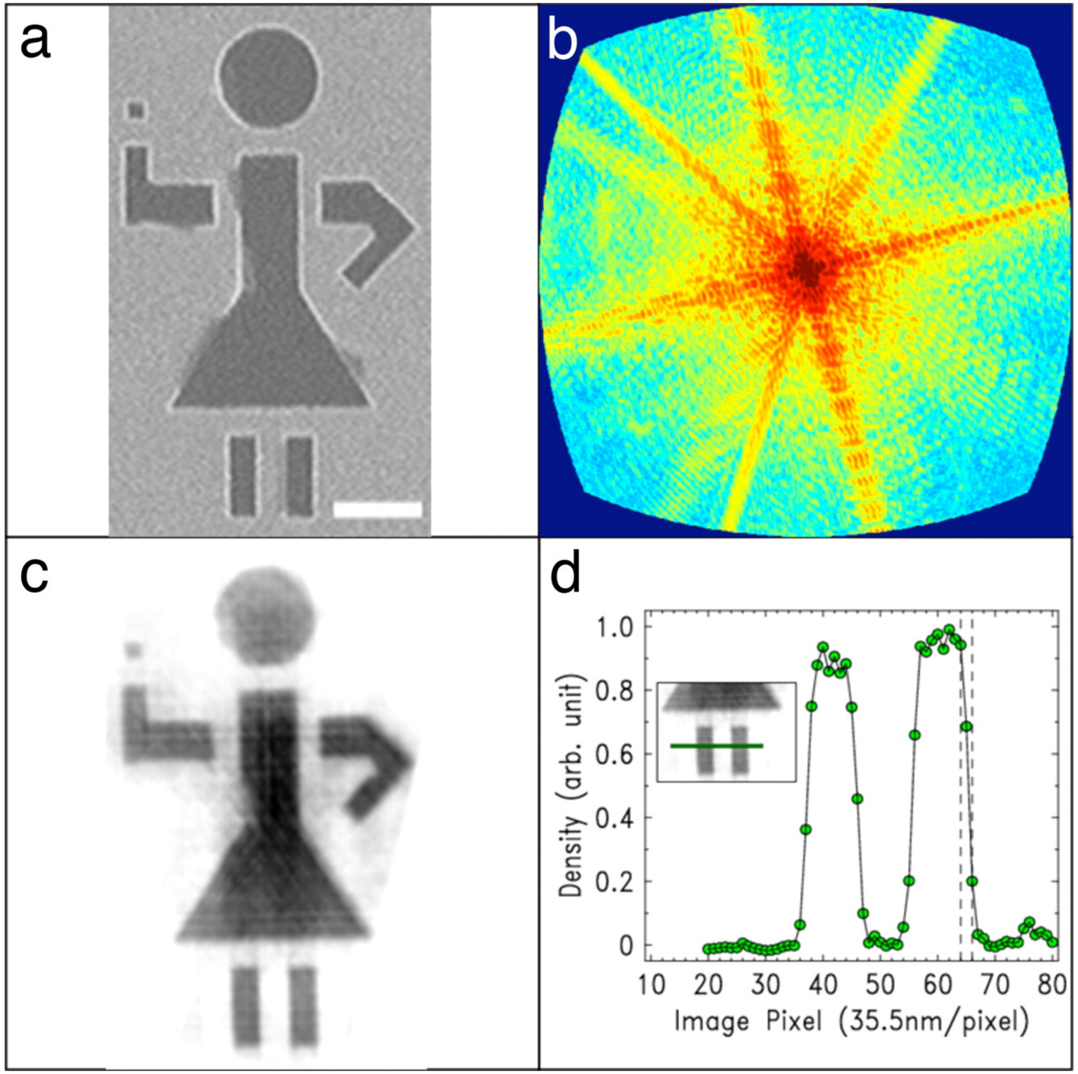


Res'n 20-30 nm

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

Way forward for lensless imaging with Synchrotron light

- Bragg – geometry diffraction microscopy
- Fresnel CDI
- Scanning diffraction microscopy (ptychography)
- References:
  - H. N. Chapman & K. A. Nugent, Coherent lensless X-ray imaging, Nature Photonics 4, 833 (2010)
  - B. Kaulich, P. Thibault, A. Gianoncelli & M. Kiskinova, J. Phys.: Condens. Matter 23, 083002 (2011)
  - R. Falcone et al., Contemporary Physics 52, 293 (2011)

Nanometer Scale Imaging Through Coherent X-ray Diffraction

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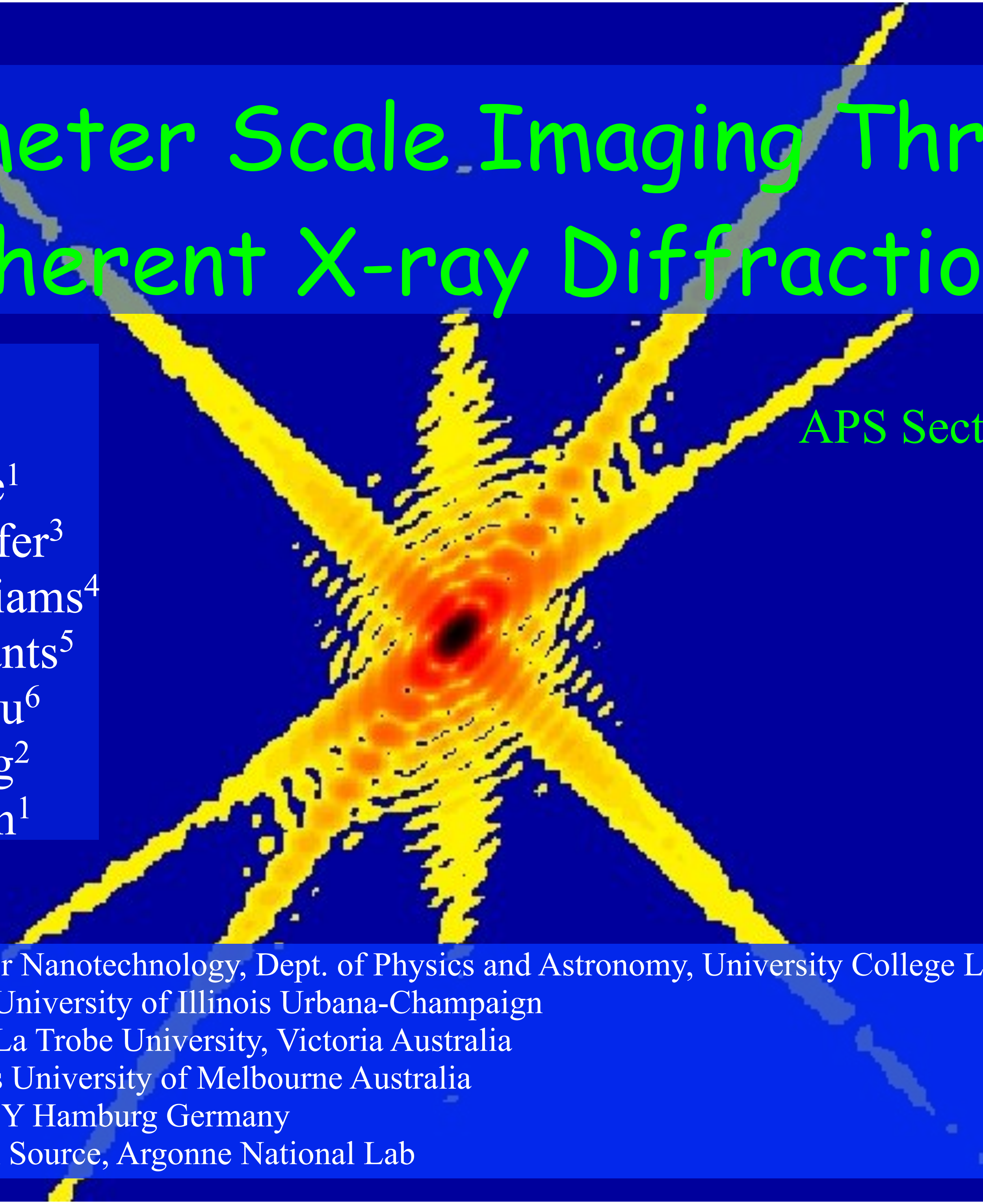
APS Sector 34ID-C



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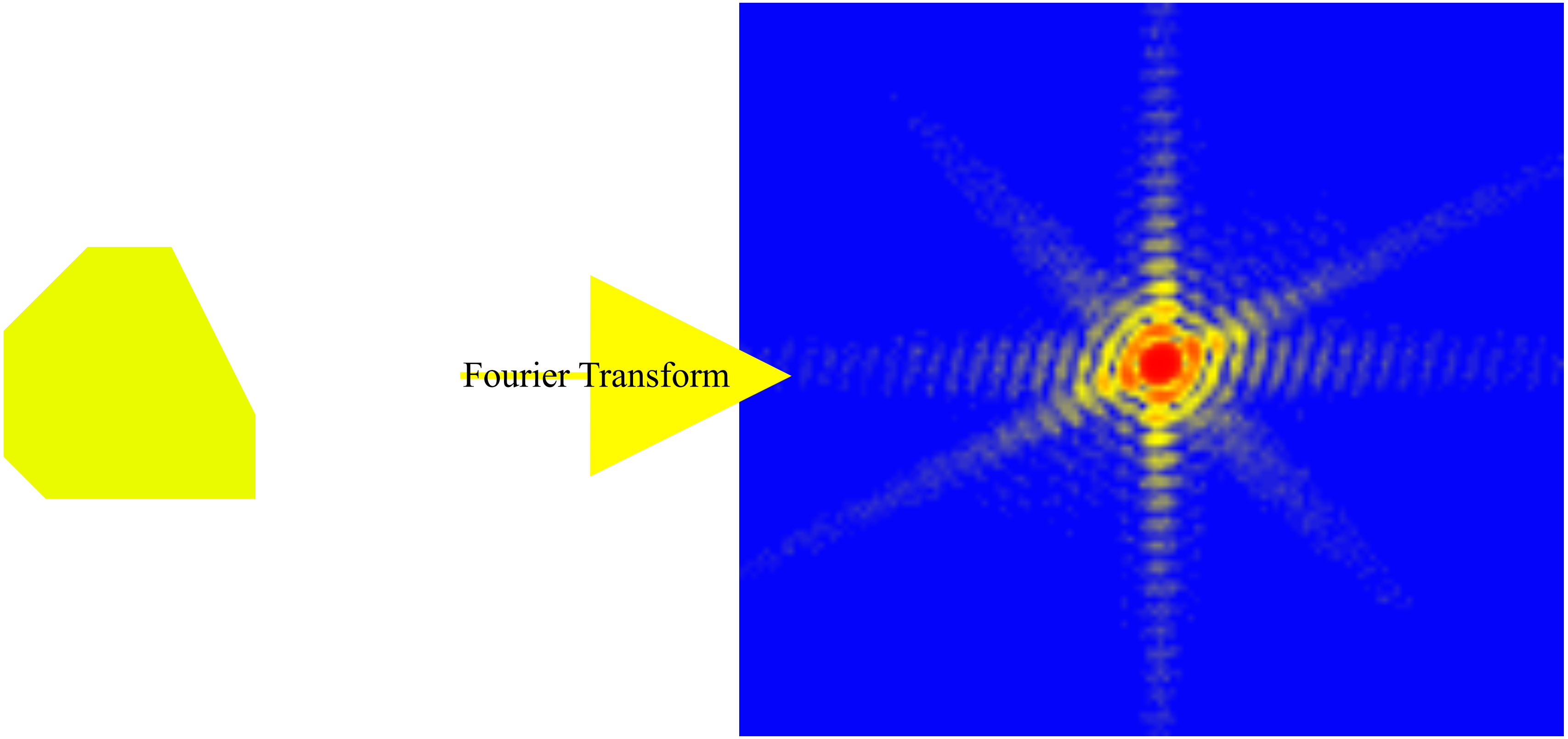
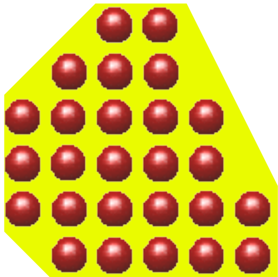
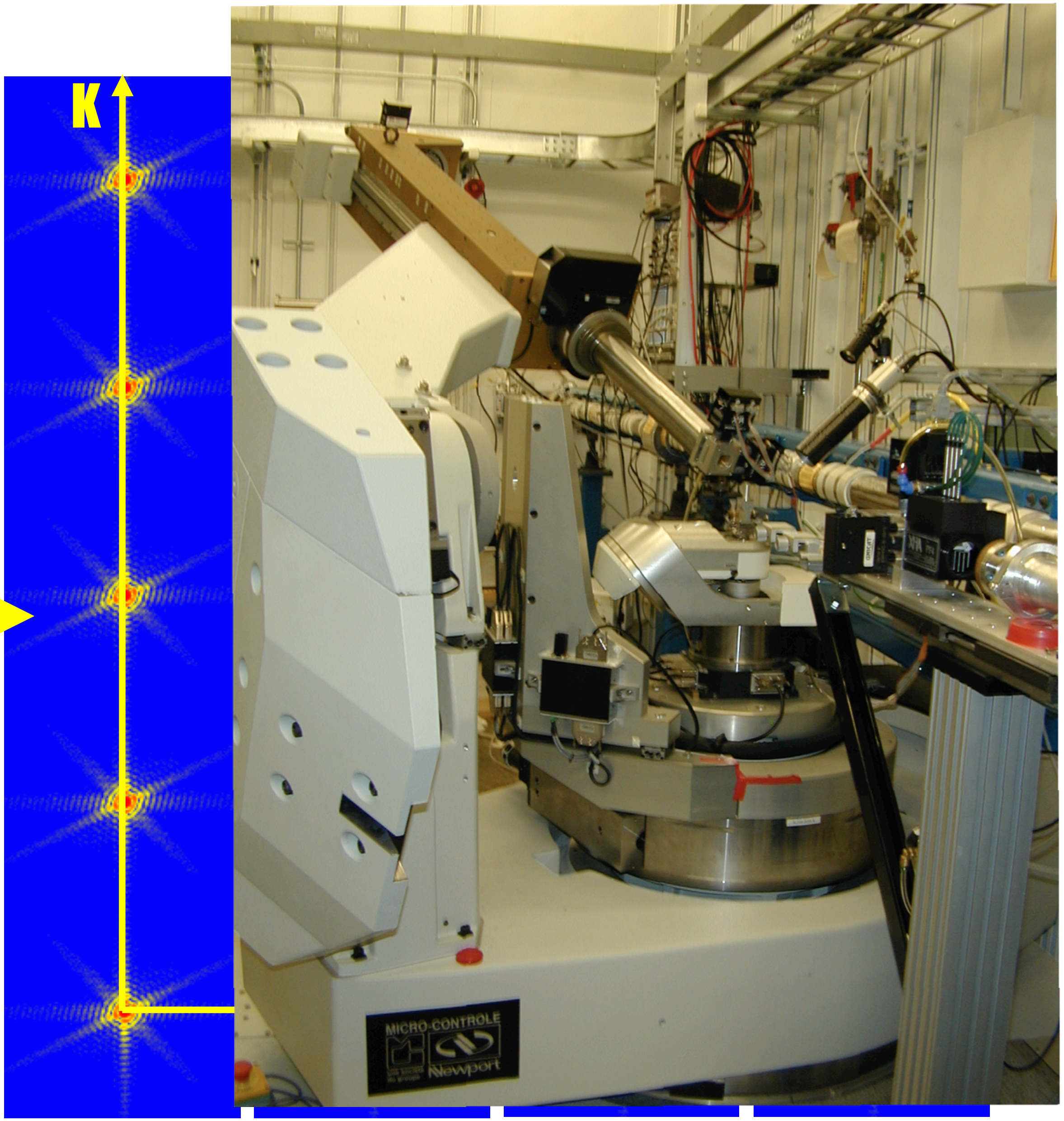
[6] Advanced Photon Source, Argonne National Lab

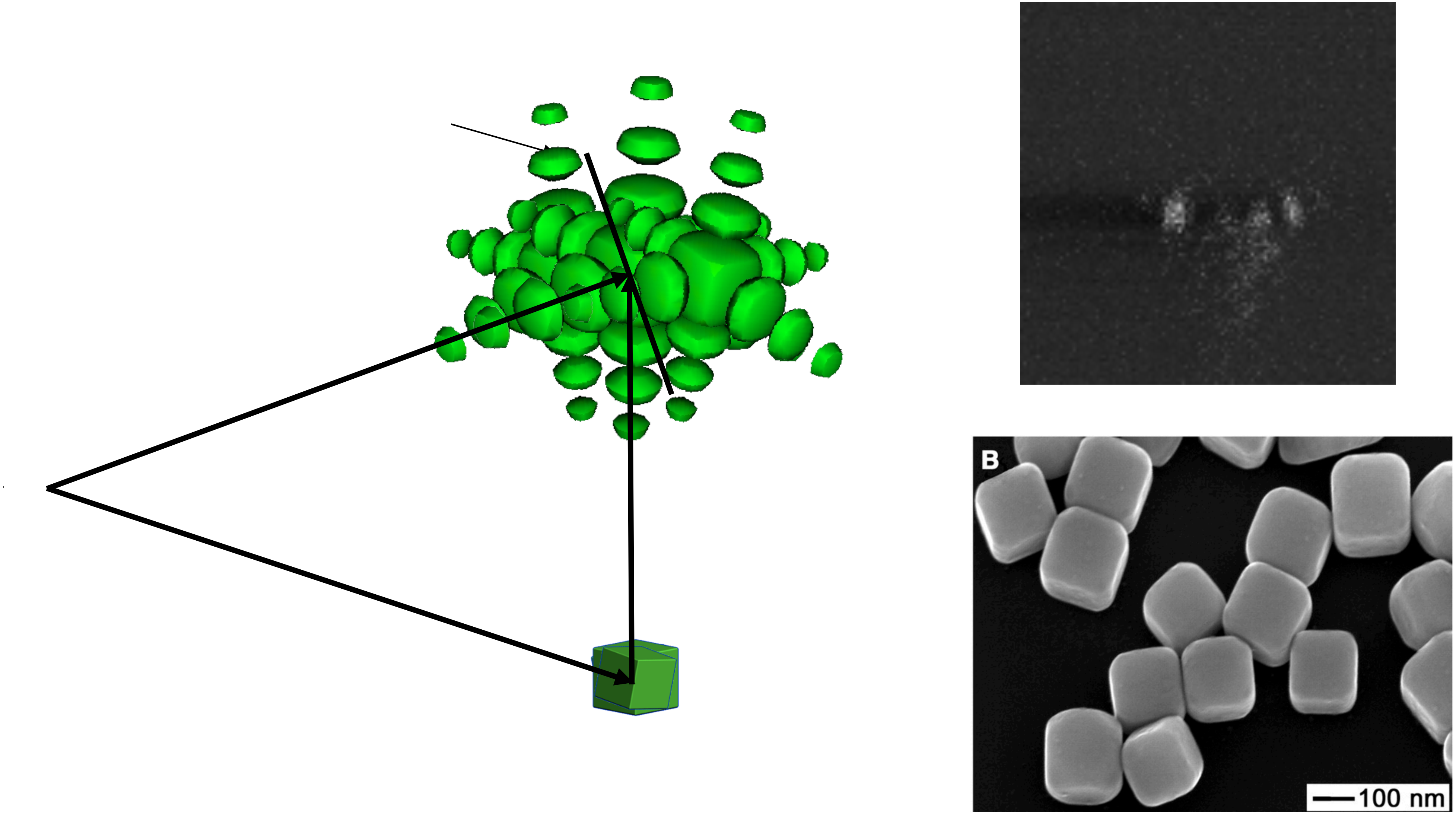

Fourier Transform

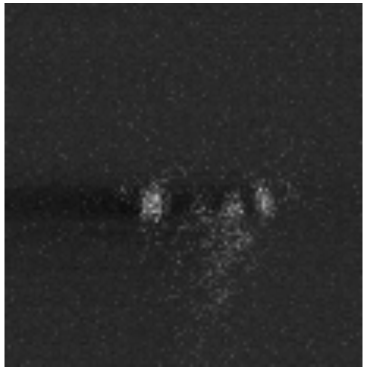


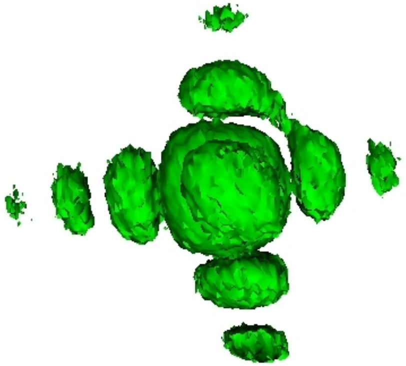
Fourier Transform

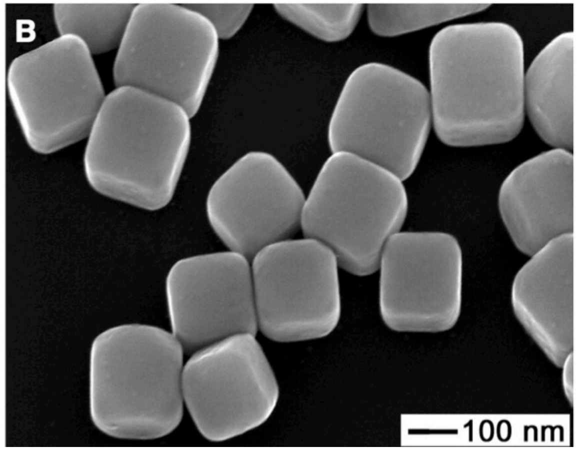


No beamstop needed!

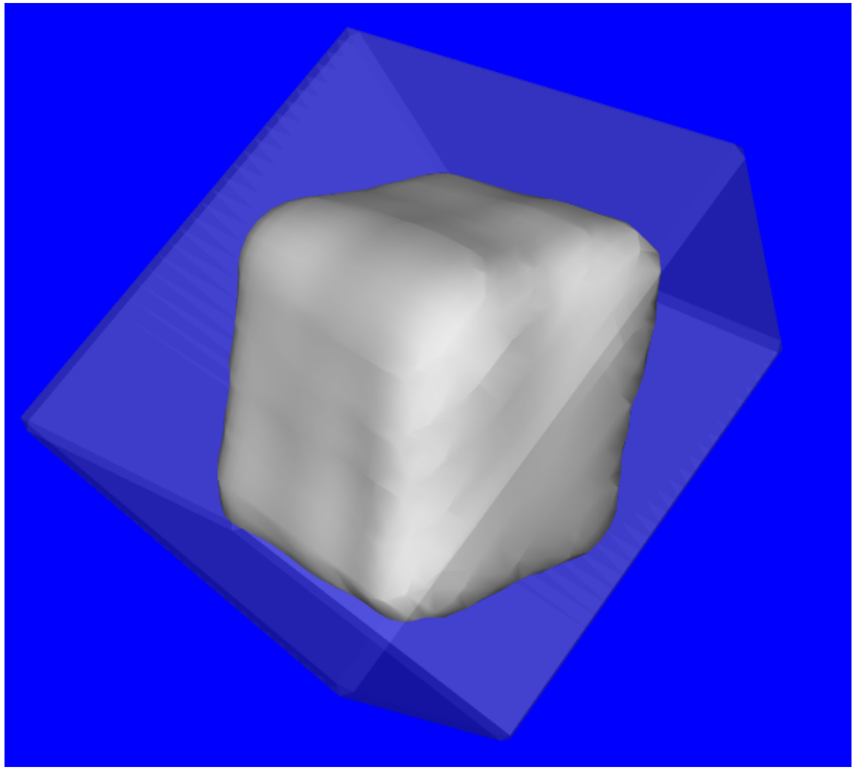

B
100 nm

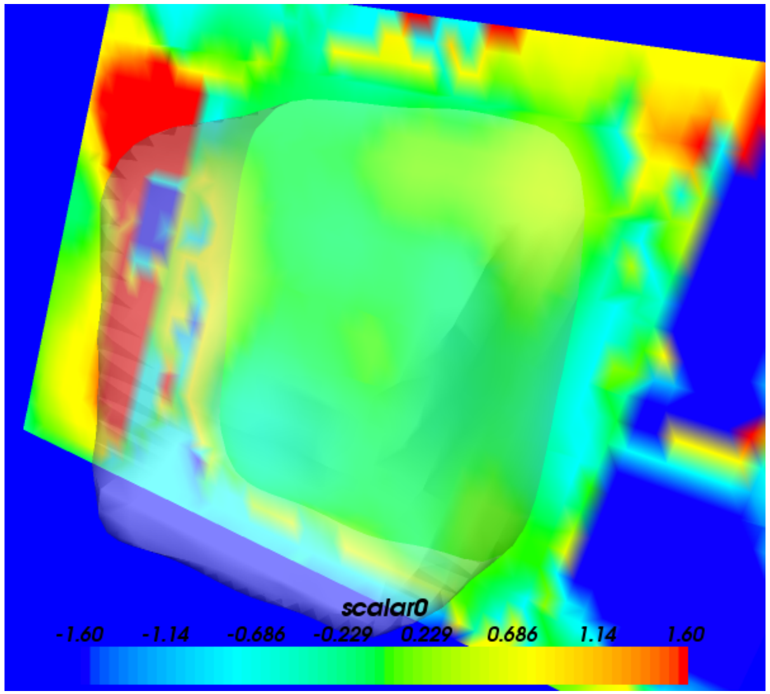






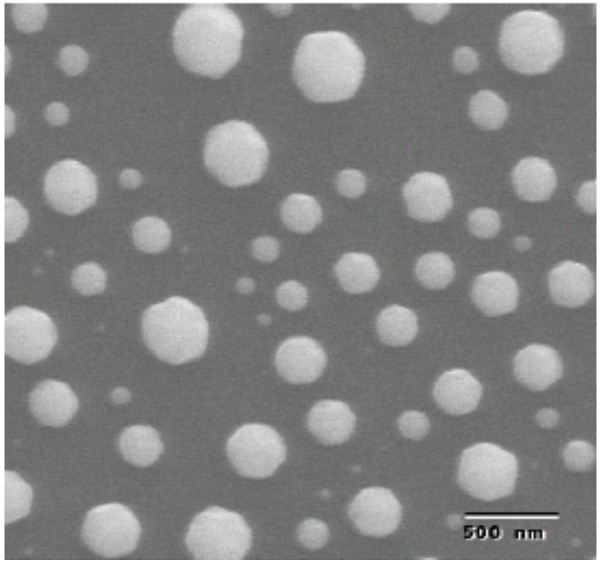
Au nanocrystals

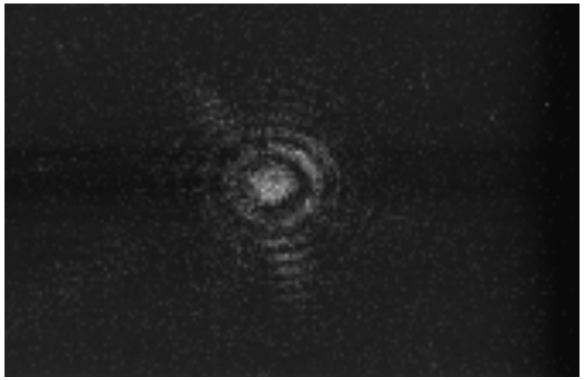


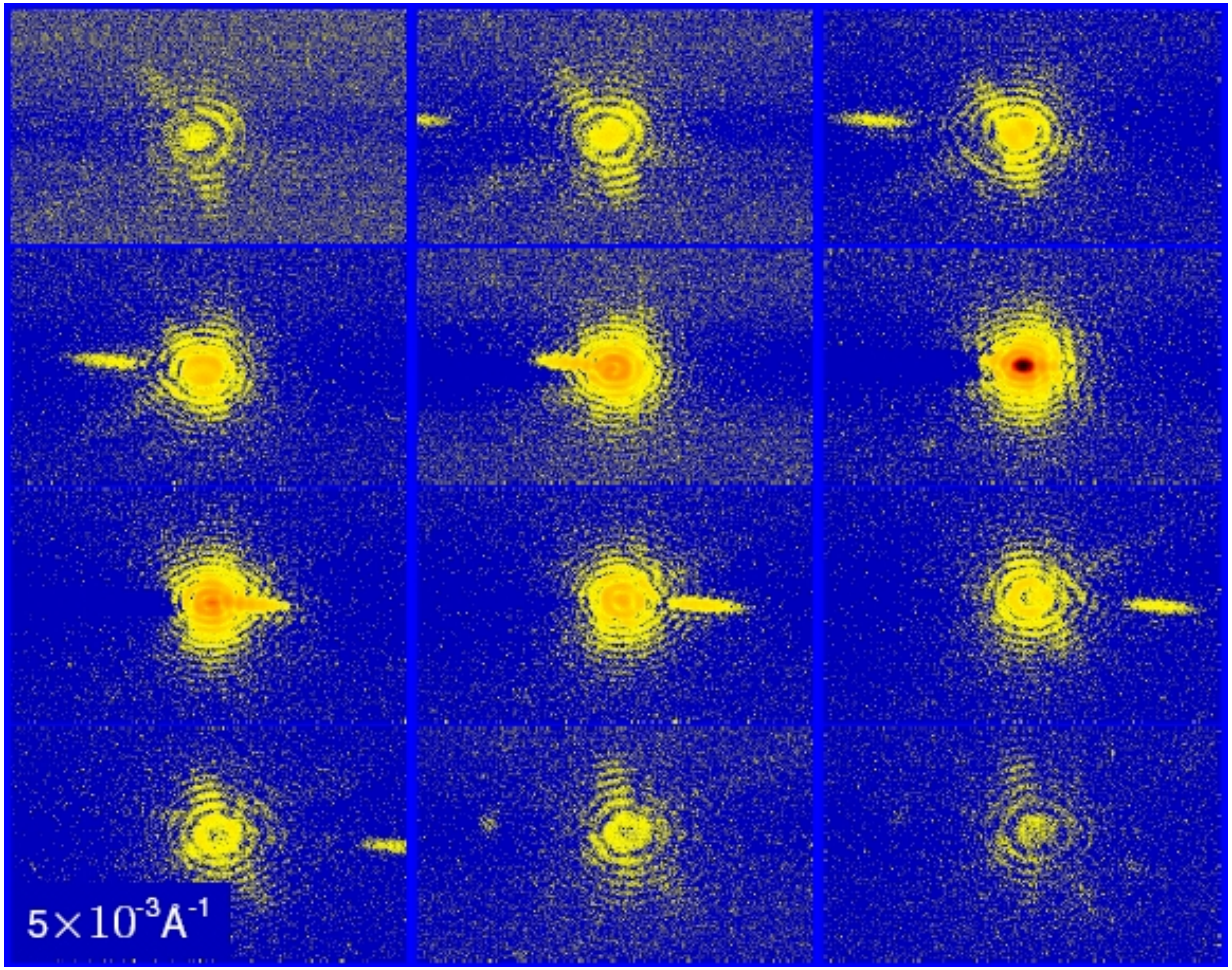


Hot Science! (T=573K)

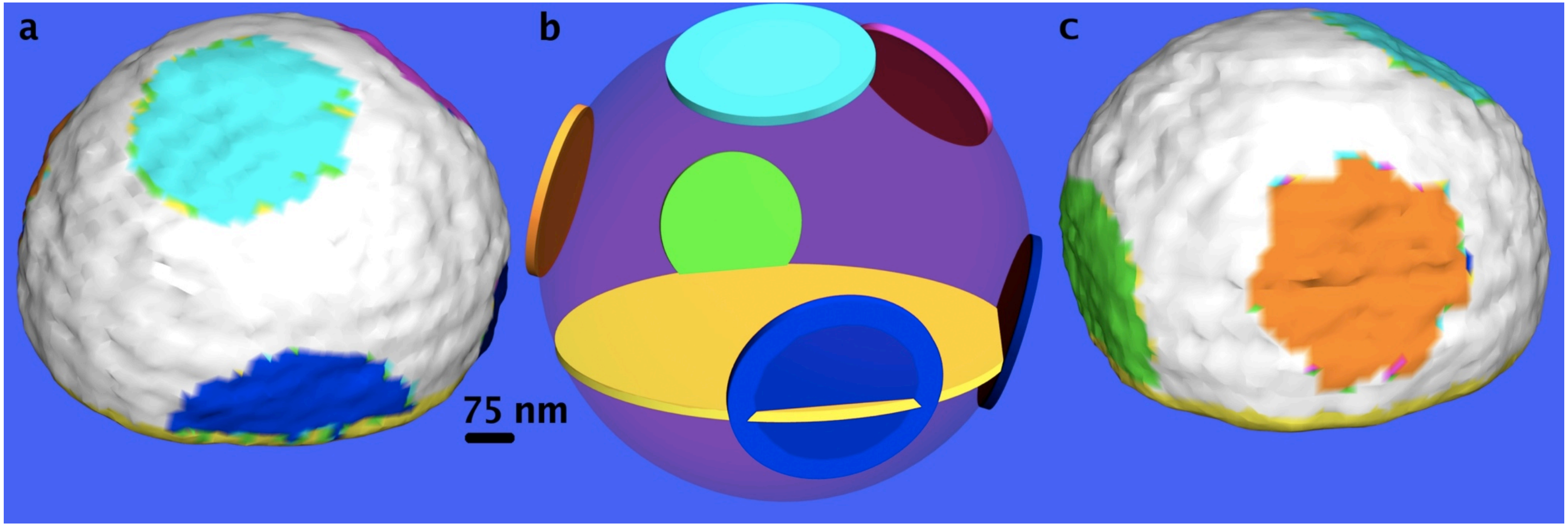
3D Diffraction From Lead



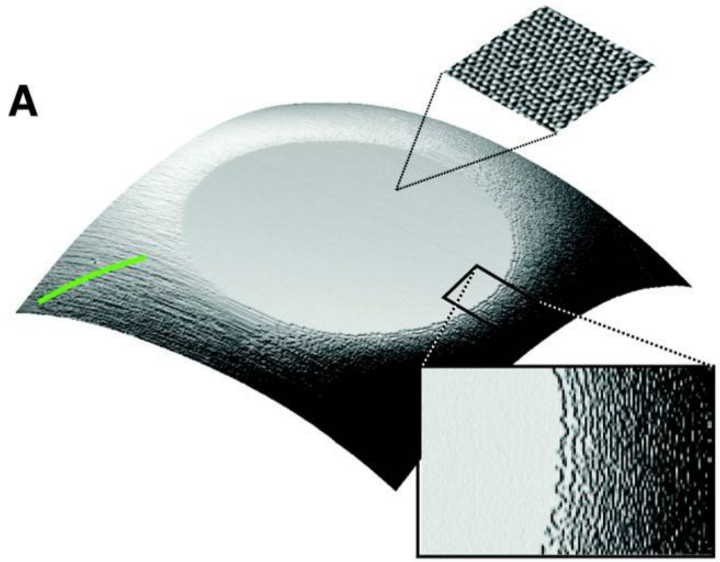




Lack of symmetry due to strain



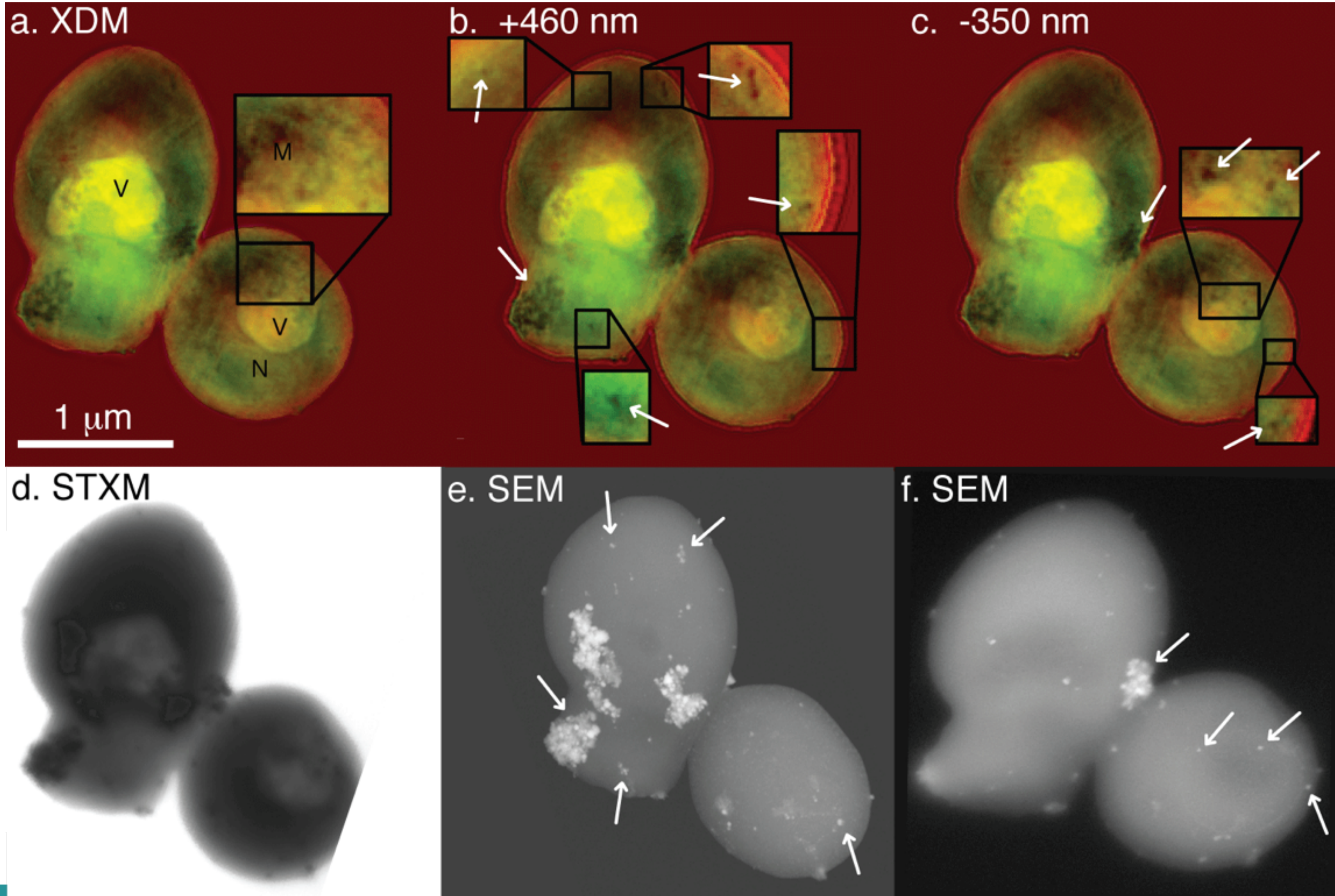
M. A. Pfeifer, G. J. Williams, I.A. Vartanyants, R. Harder and I. K. Robinson, Nature 442, 63 (2006)



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

Gold-labeled yeast

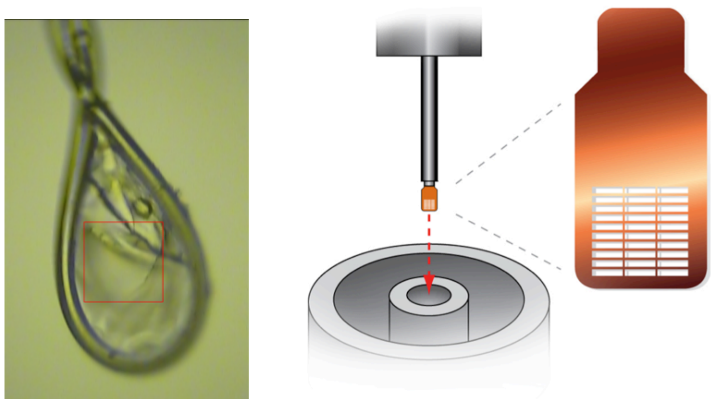
1.8 nm gold, silver-enhanced, freeze-dried: Johanna Nelson, Stony Brook.
Propagation of complex reconstructed wave to "focus" on different planes.



PNAS 107, 7235 (2010)

Sample preparation and data collection

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



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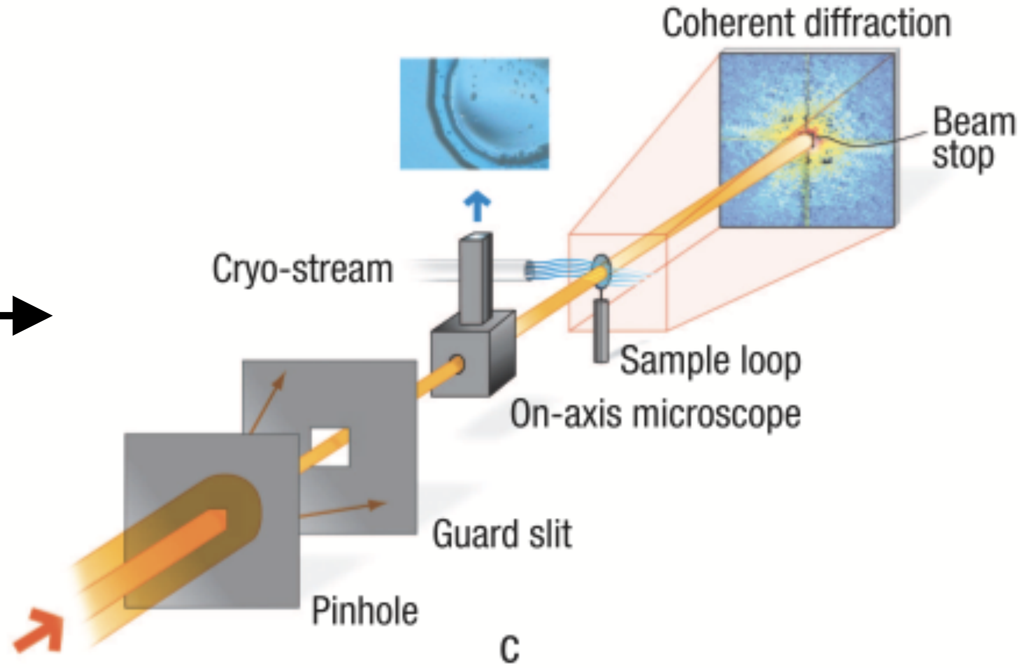
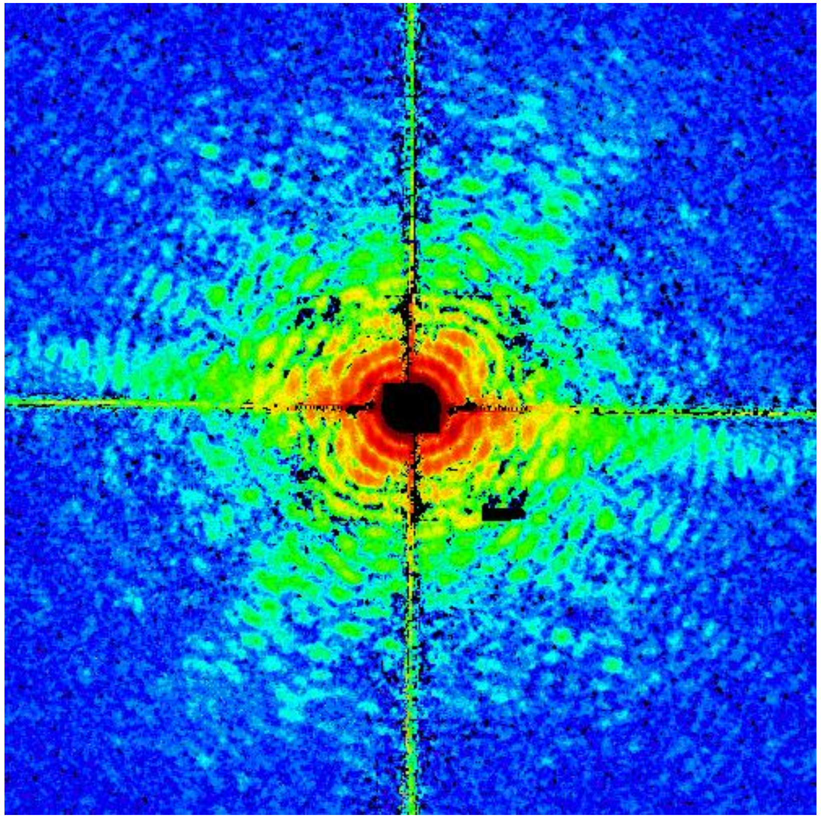


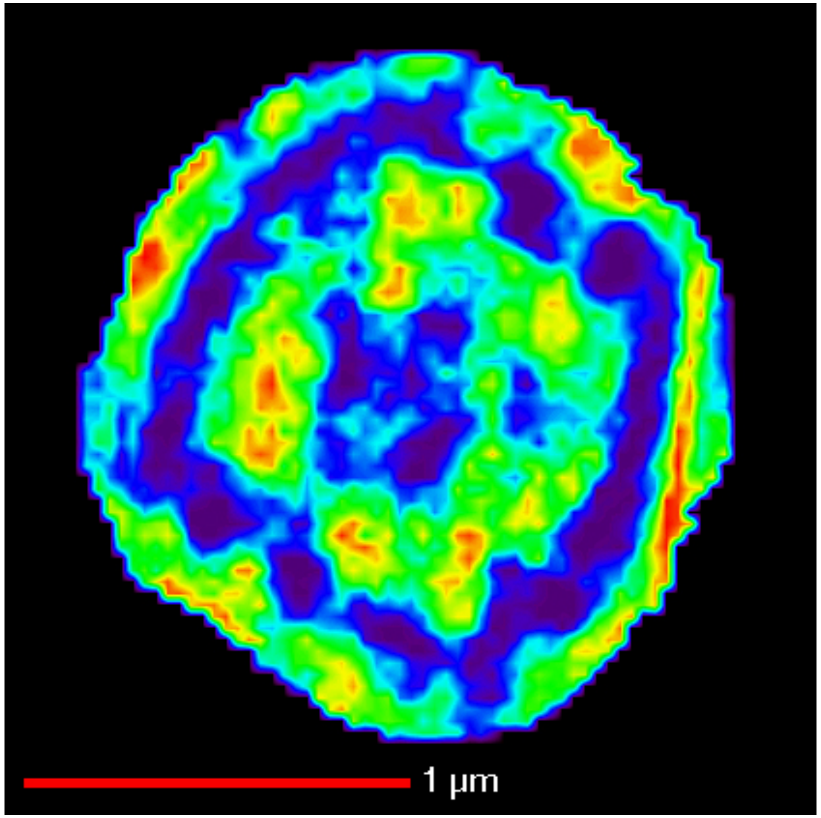
- Vitrification by plunge-freezing into liquid ethane

- Experimental setup for data collection using cryogenic gas stream

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

Diffraction pattern and reconstruction

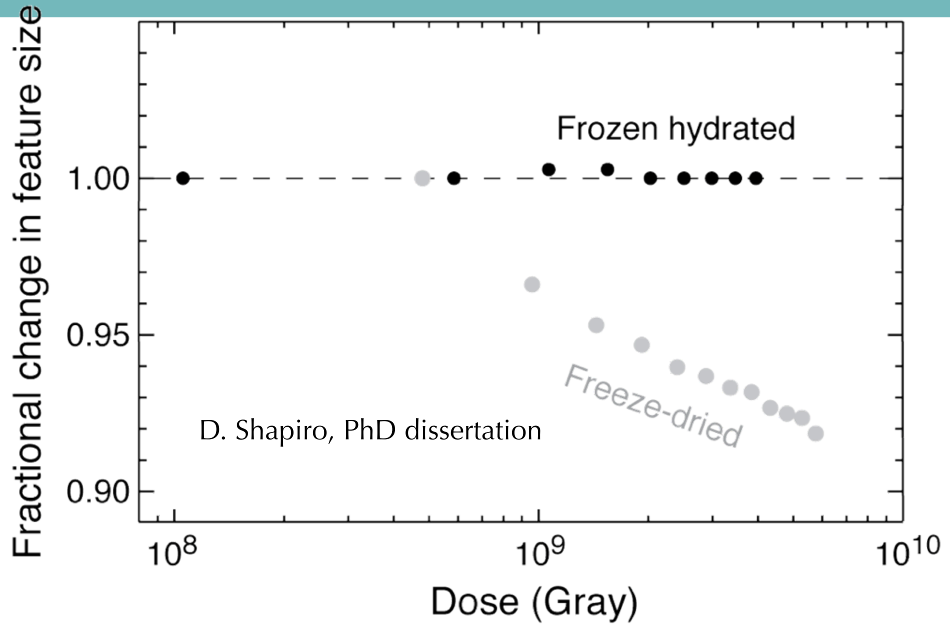


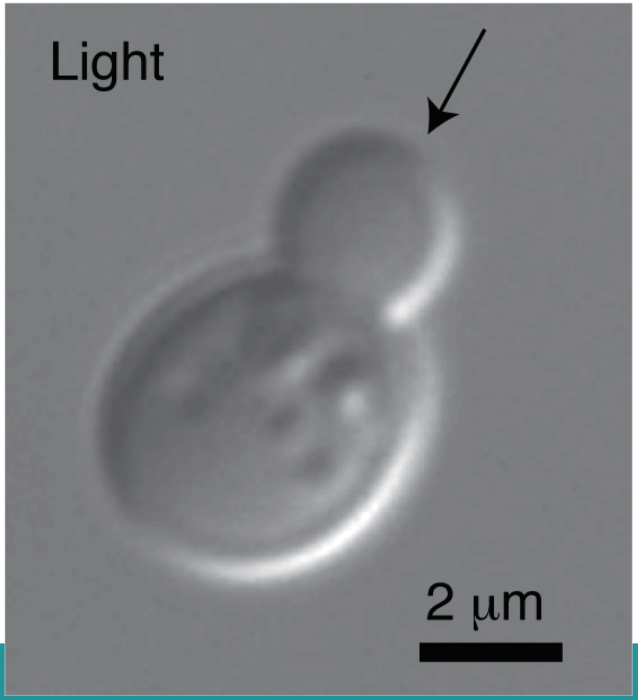


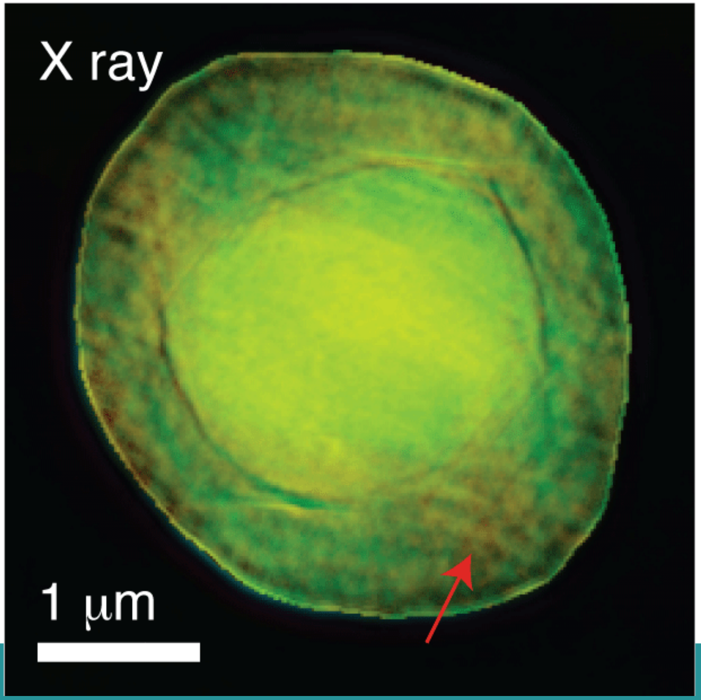
- Diffraction pattern from *D. radiodurans* using 8 keV x-rays
- Reconstructed image of *D. radiodurans*

Frozen hydrated yeast

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

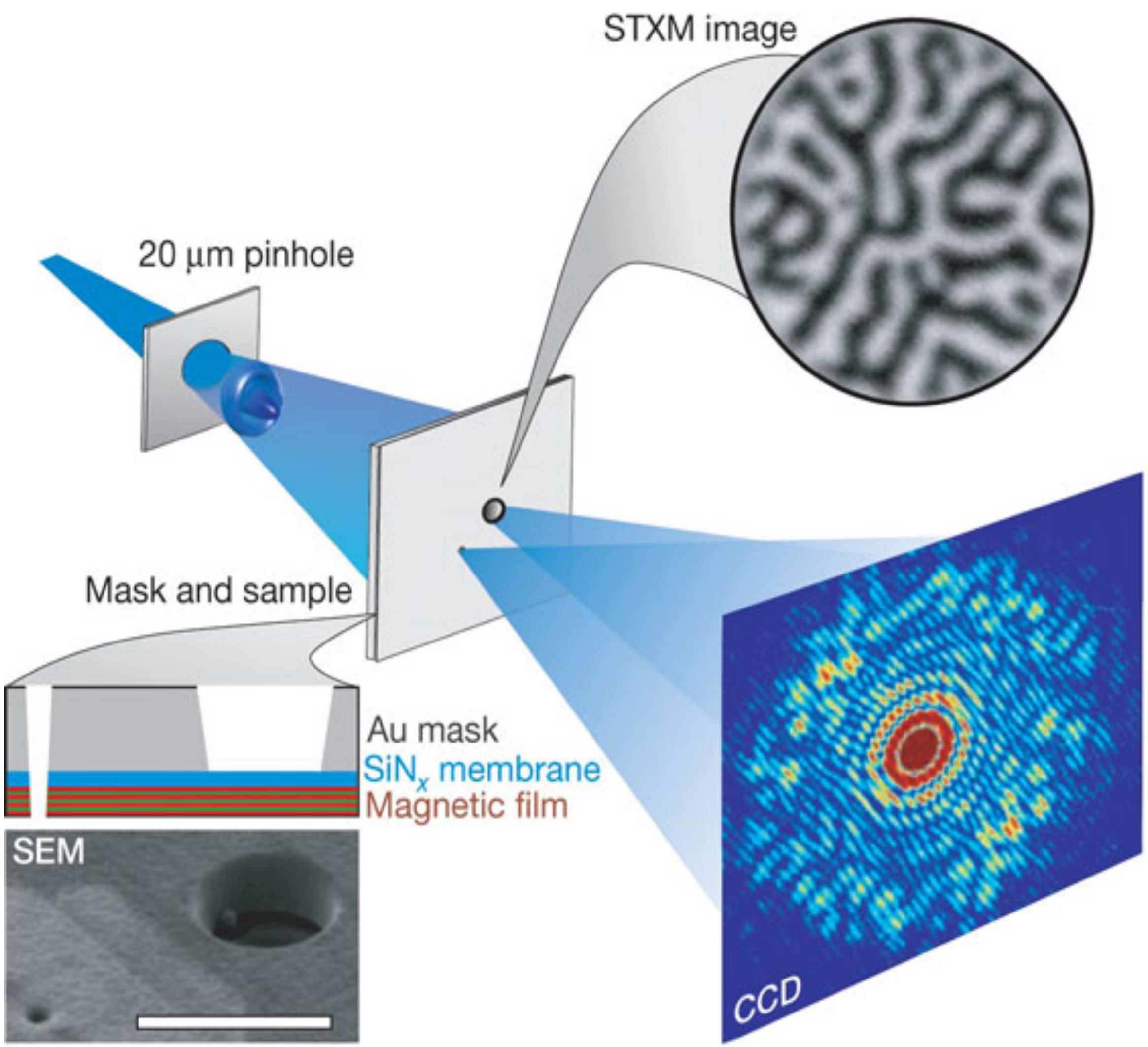






Fourier transform holography

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.

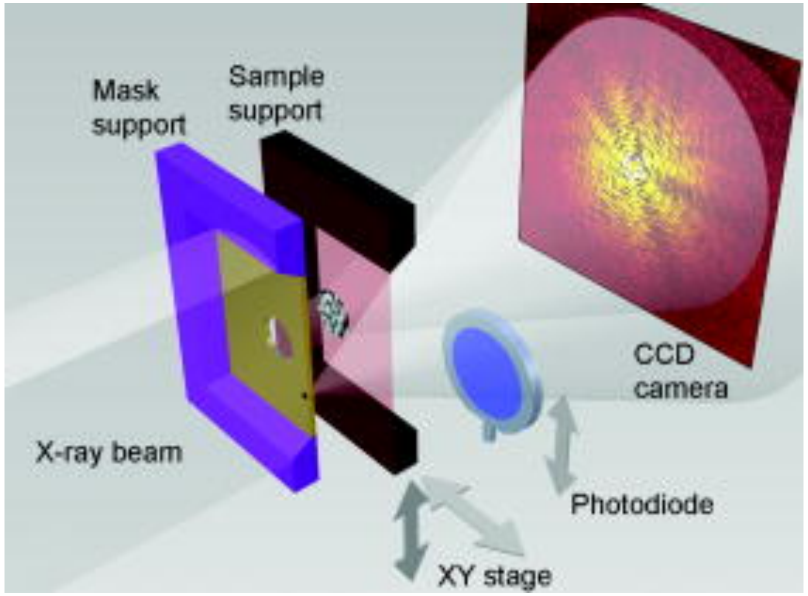


FIG. 1. Schematic of the x-ray holographic microscopy setup. It consists of a holography mask support, a movable sample support, and a CCD detector. The membrane with the optical elements (mask), i.e., the object and reference holes, is fixed in the center of the...

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

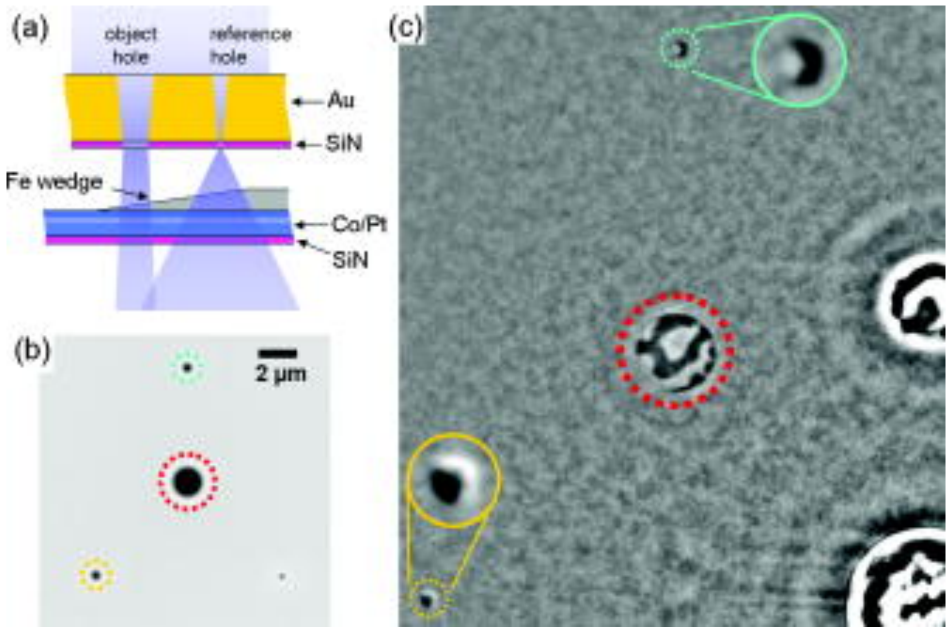


FIG. 2. Sample geometry and reconstruction of a single magnetic domain image. A cut through the scattering plane is shown in (a). The sample is illuminated through the optics membrane [SEM micrograph (b)]. Image (c) is a cutout of the real part of the FFT reconst...

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

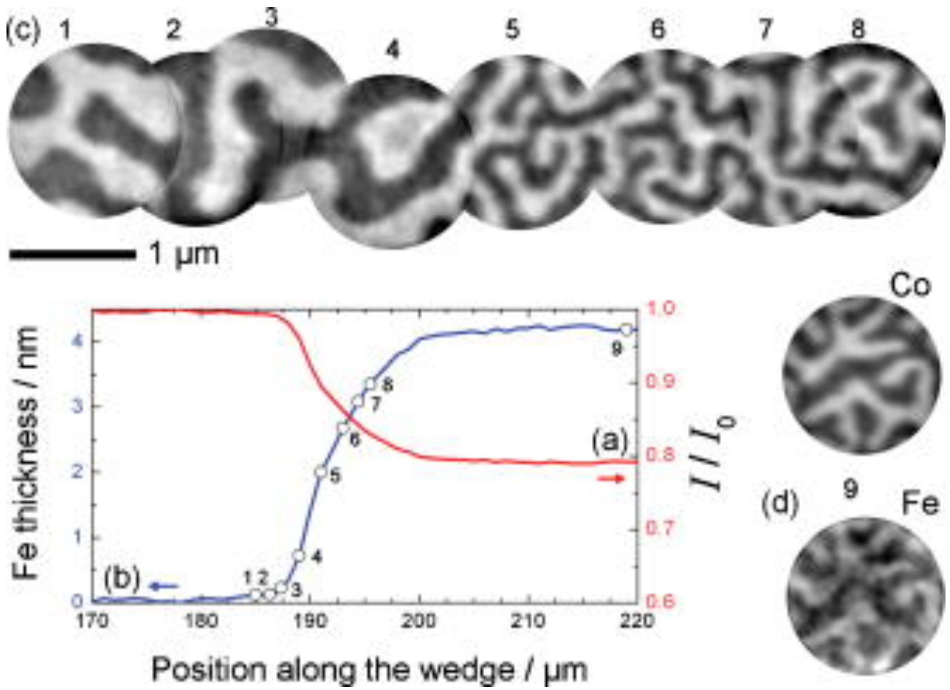
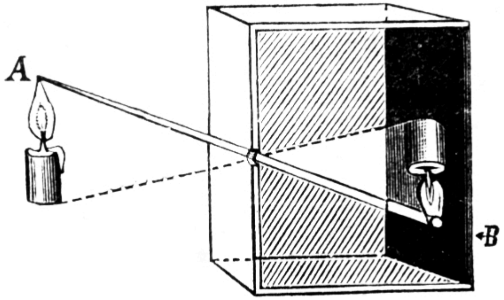


FIG. 3. Domain size evolution of a Co/Pt multilayer film covered by an iron wedge. Plot (a) gives the absorption profile (normalized photodiode current) at the Fe L3L3 absorption edge when scanning over the Fe wedge. The absorption is used to calculate the local ...

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

Coded aperture imaging

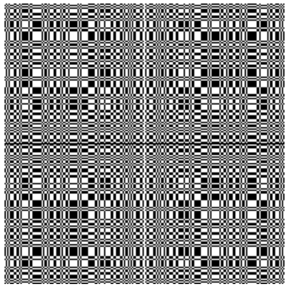
Pinhole camera



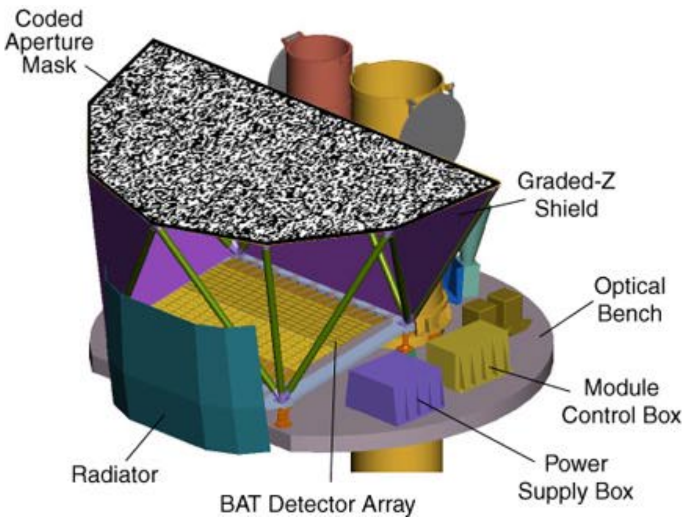
spectroscopy

Resolution or SNR

Solution: use a uniformly redundant array



γ-ray astronomy



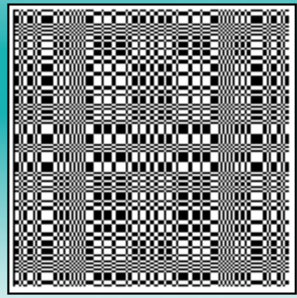
Medical imaging

Homeland security

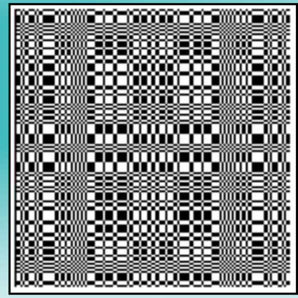
Coded aperture holography overcomes resolution vs brightness limitations

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)



*

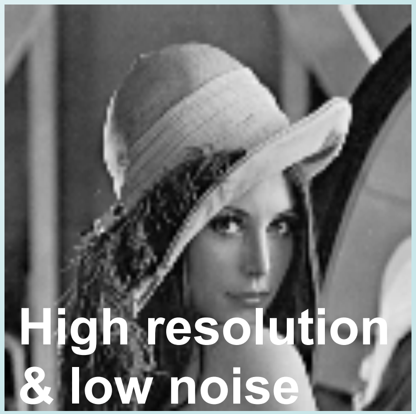


=

Same number of photons

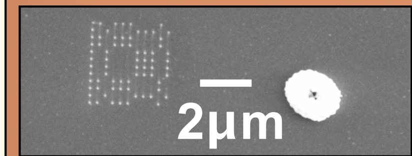
Fourier transform holography

with coded apertures: **High resolution, low noise**



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

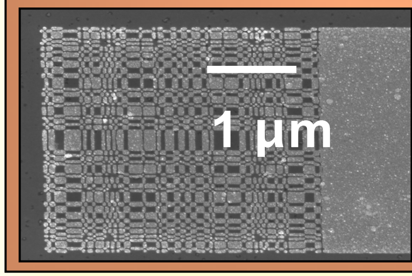
samples



Focused Ion Beam (50 nm res)

S. Boutet

SLAC

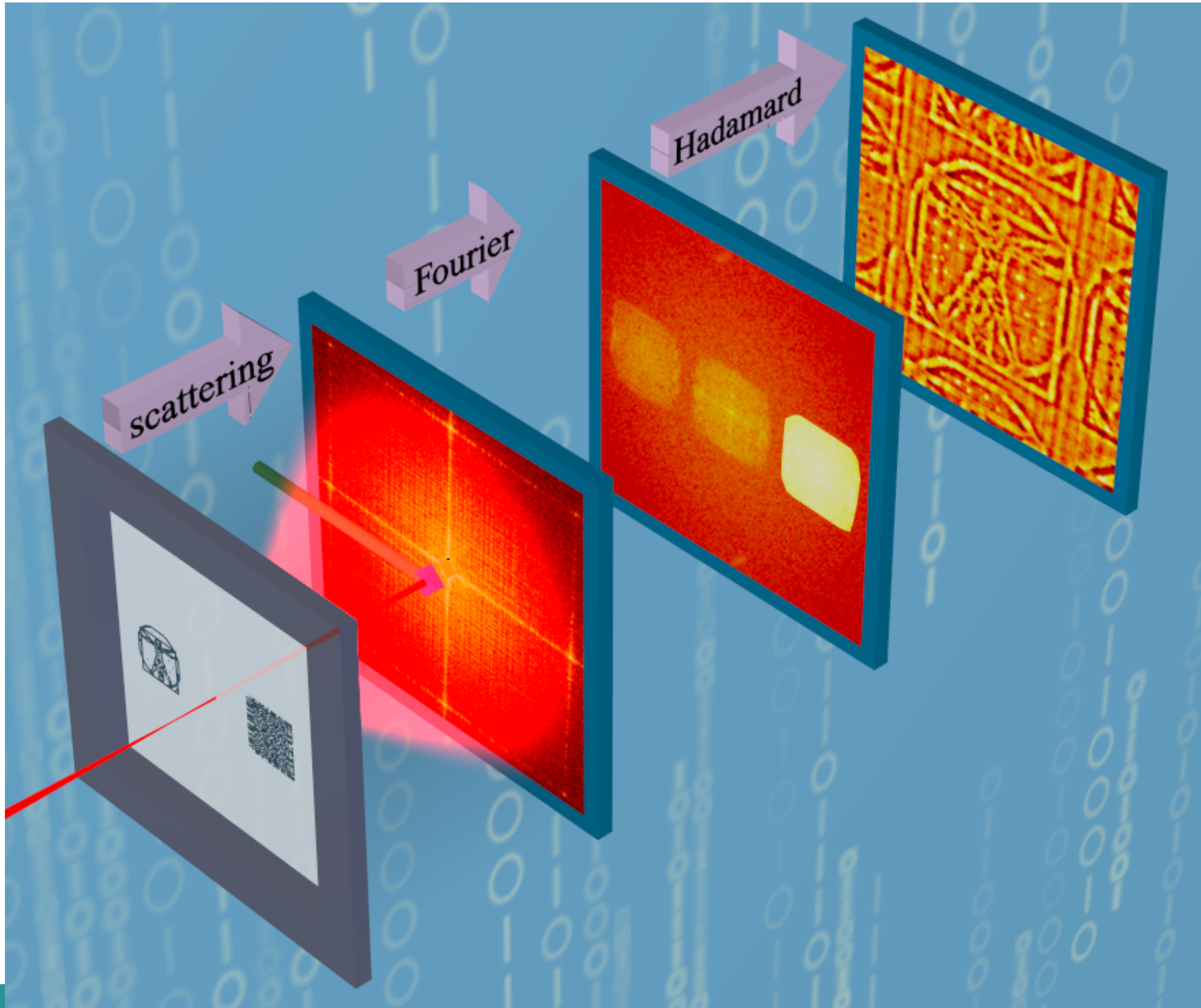


E- beam lithography (12 nm res)

Sakdinawat

CXRO

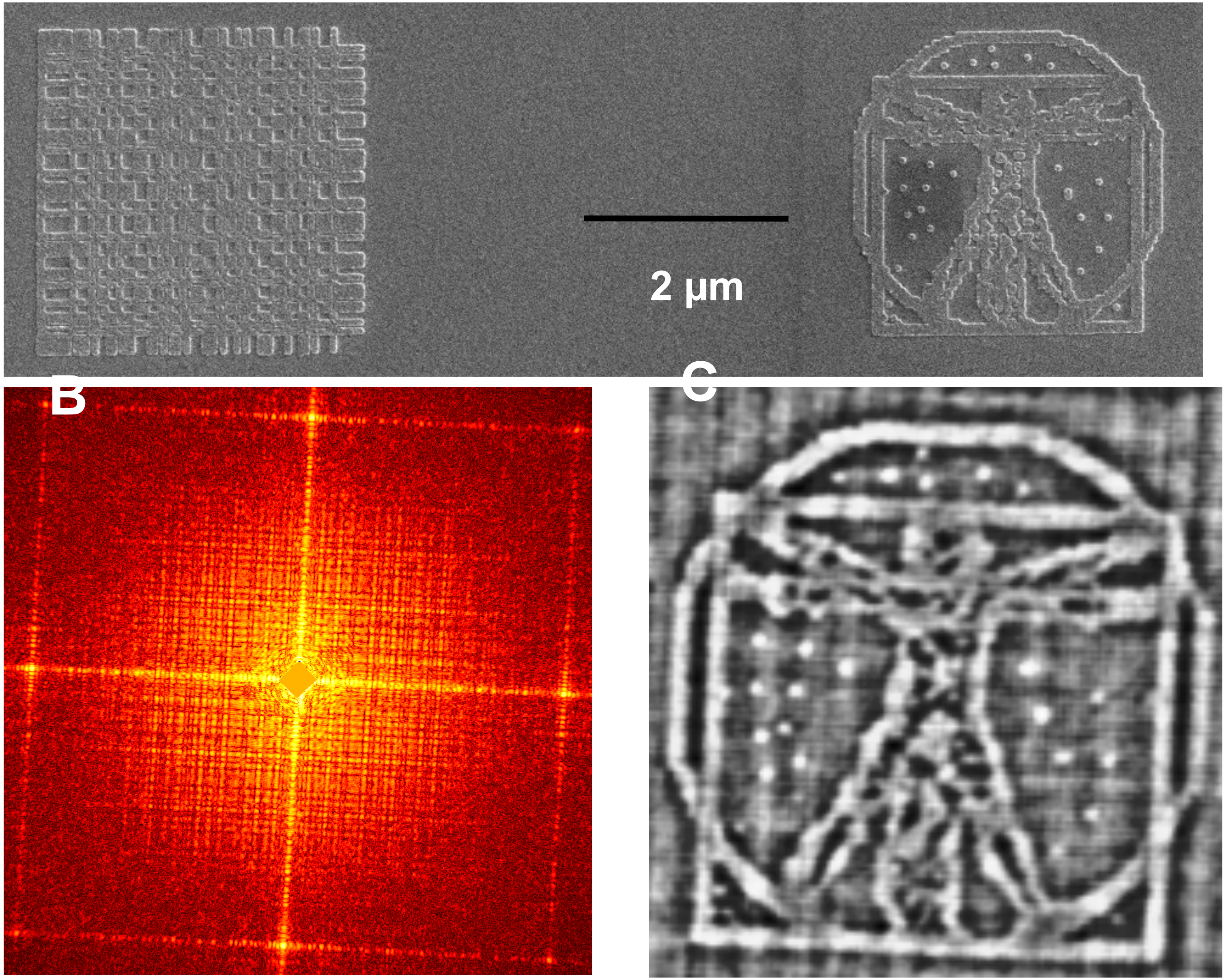
S. Marchesini, S. Boutet, A. Sakdinawat, et al. Nat. Phot. 2, 560 - 563 (2008).



S. Marchesini, S. Boutet, A. Sakdinawat, et al.
Nat. Phot. 2, 560 - 563 (2008).

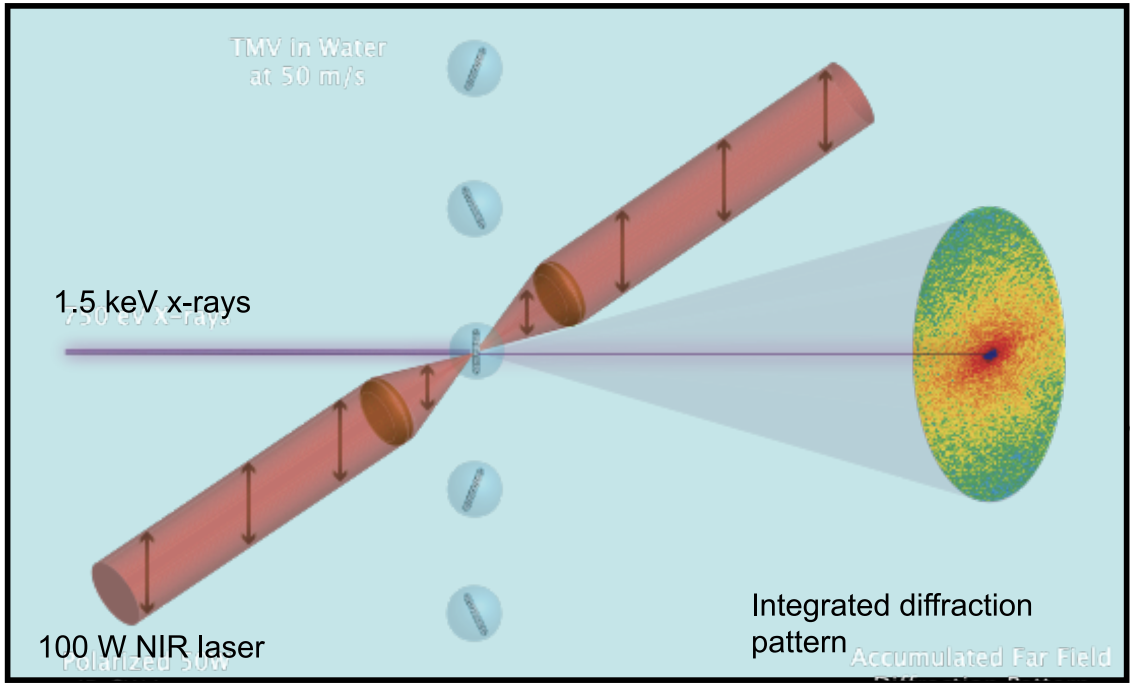
λ=2.2 nm Resolution=43 nm, SNR X ~70

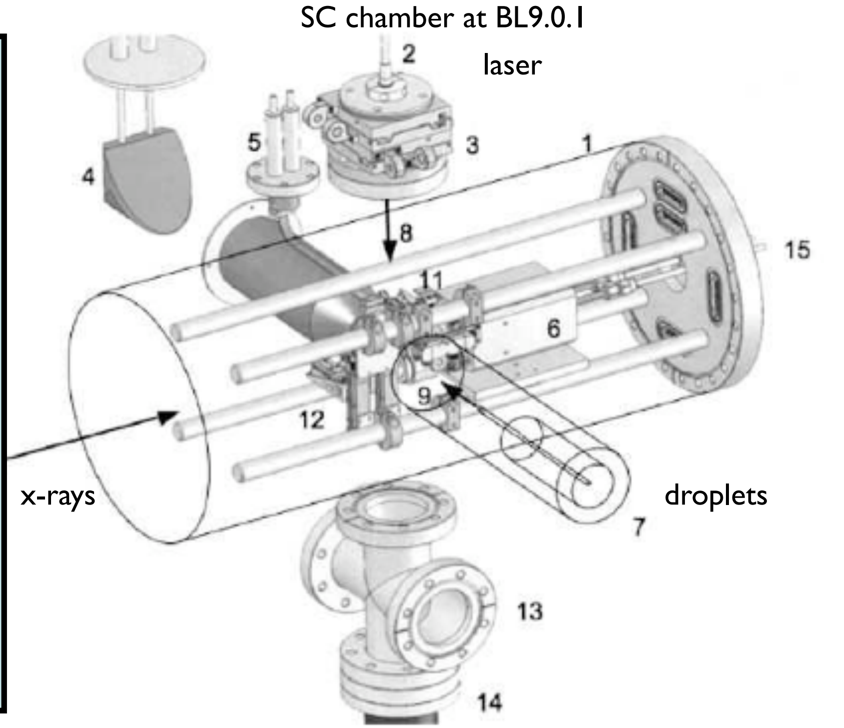
Sample from A. Sakdinawat



Serial Crystallography

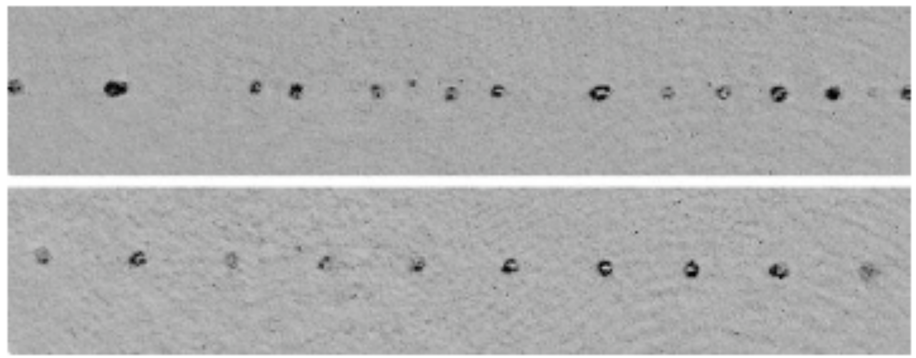
GOAL: image uncrystallized proteins using the same basicXDM method
PROBLEM: how to collect hi resolution diffraction from single proteins?





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)*



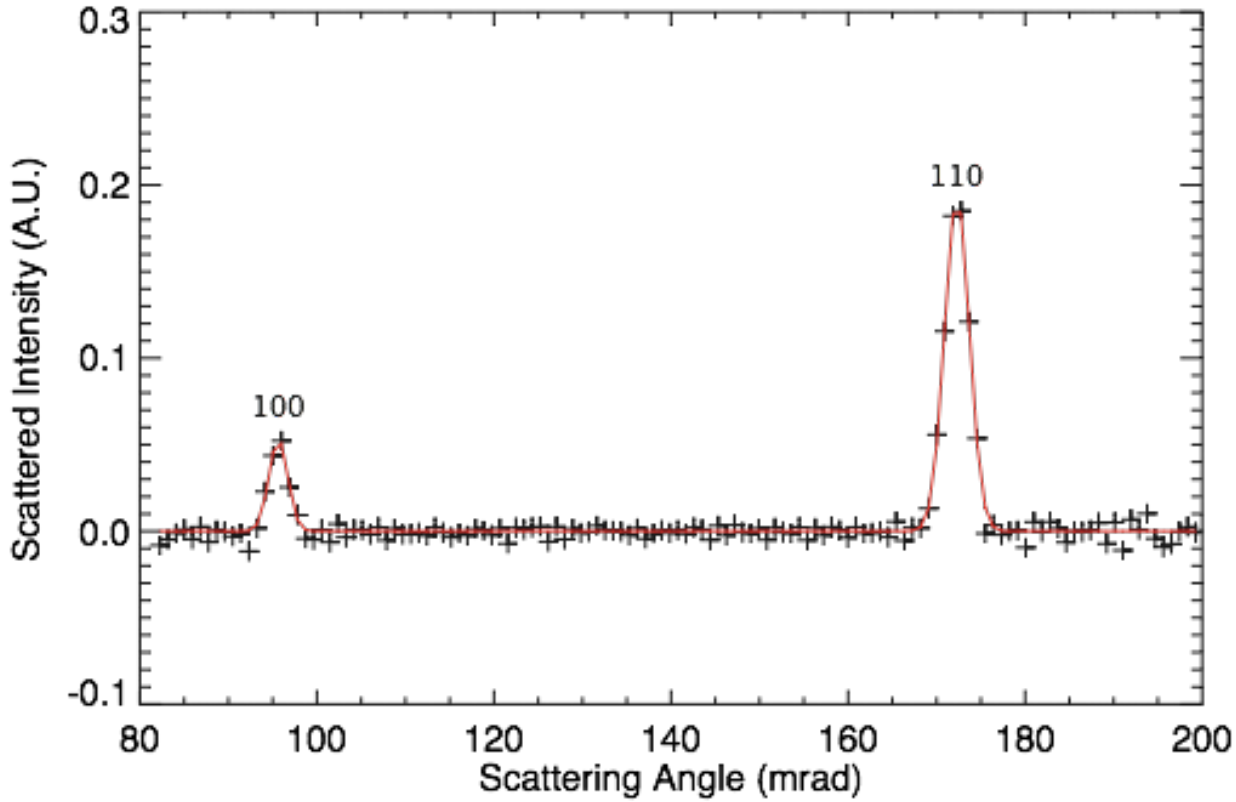
Photosystem I Protein

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
$\|F_{110}\|/\|F_{100}\|$	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



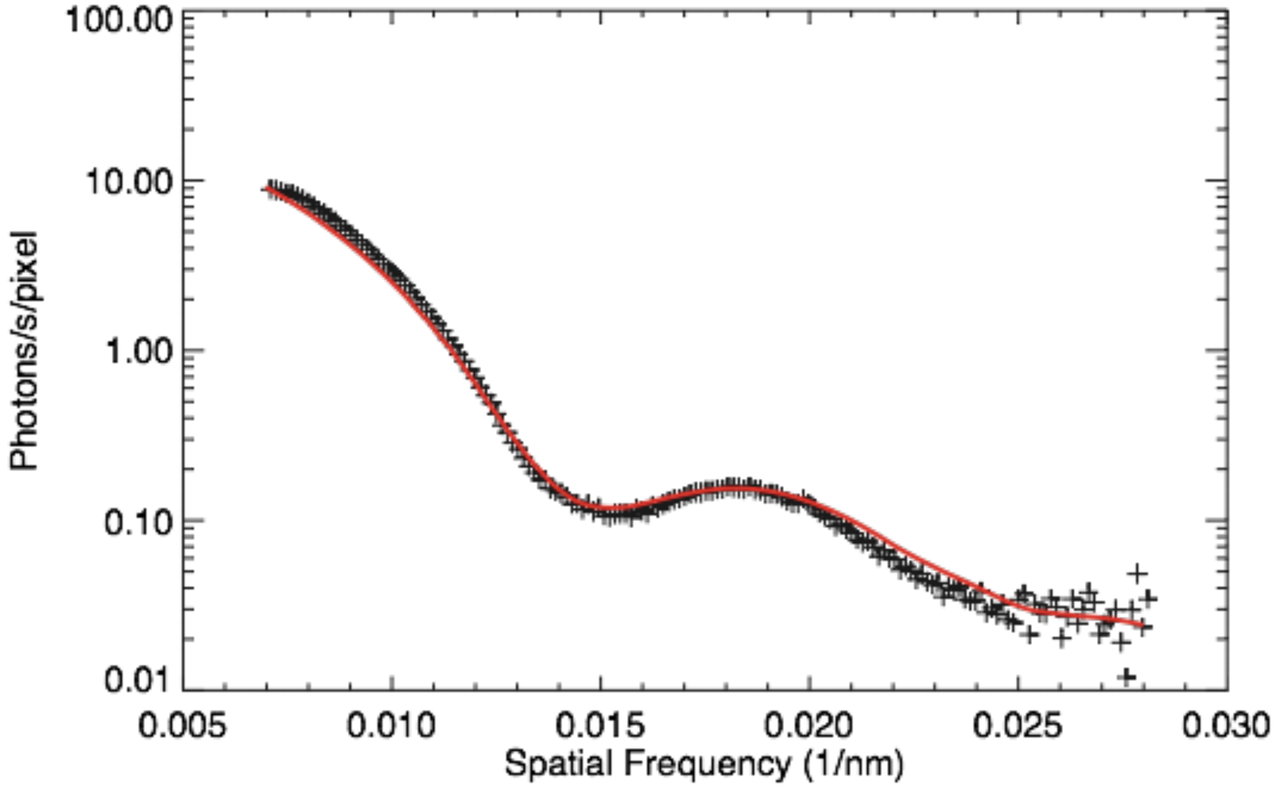
Protein powder diffraction without freezing or crushing
NO RADIATION DAMAGE!

D. A. Shapiro et al., J. Synchr. Rad. 15, 593 (2008)

Gold Nano-Spheres

Un-aligned particles give a SAXS pattern

- 50nm gold spheres, 5×10^{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

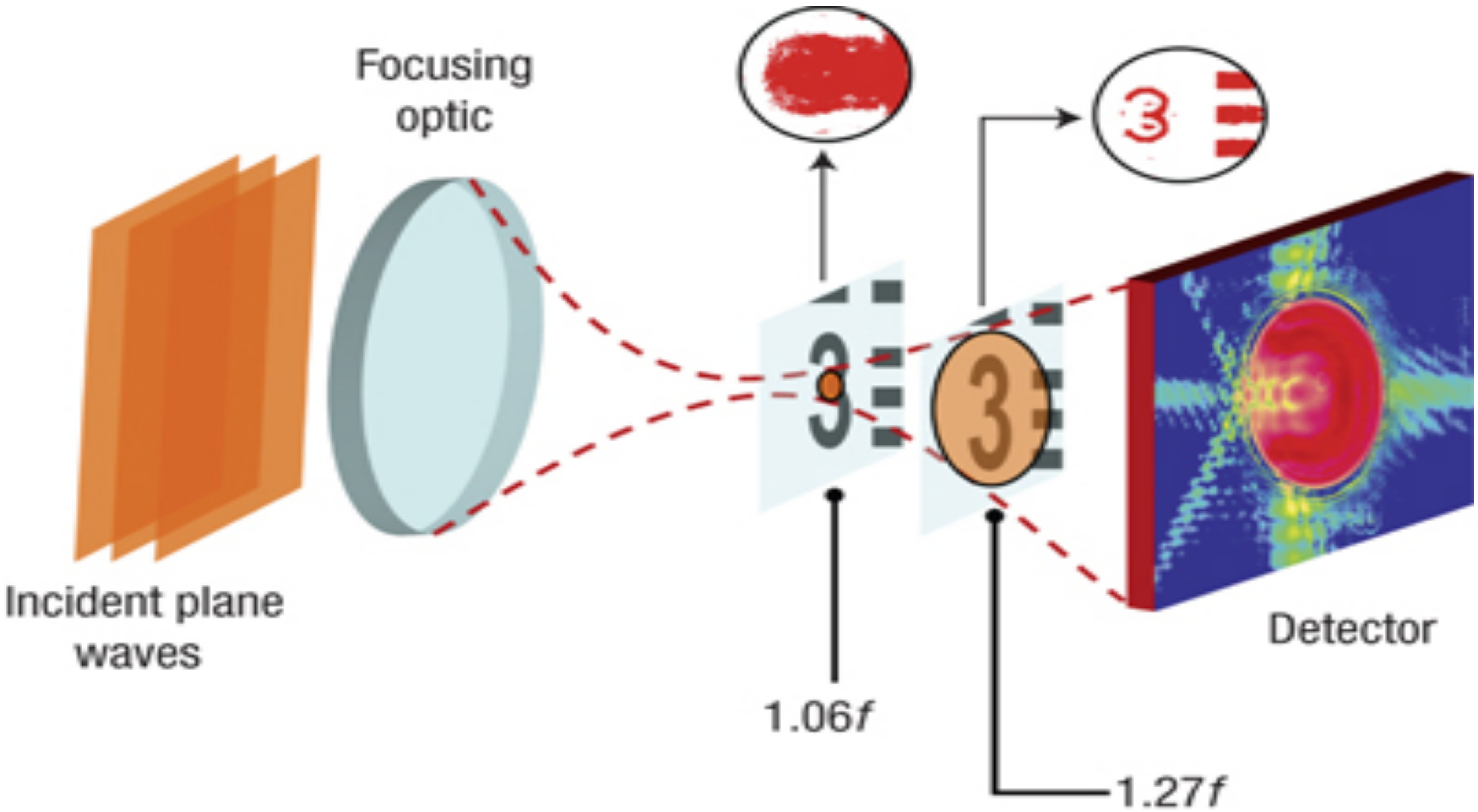


Size Distribution has: sigma=6nm

D. A. Shapiro et al., J. Synchr. Rad. 15, 593 (2008)

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

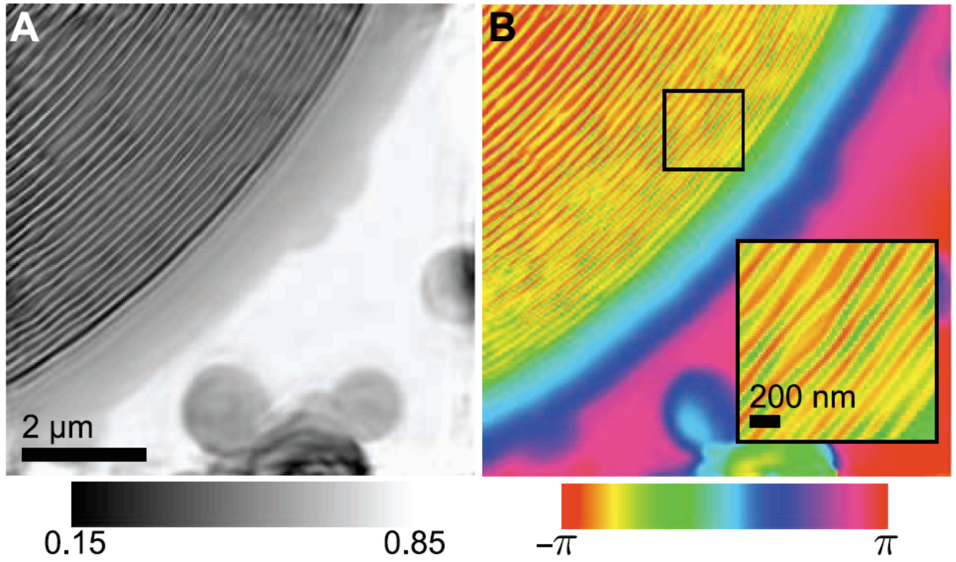
Ptychography with Pilatus

High-Resolution Scanning X-ray Diffraction Microscopy

Pierre Thibault,[1*] Martin Dierolf,[1] Andreas Menzel,[1] Oliver Bunk,[1] Christian David,[1] Franz Pfeiffer[1,2]

Coherent diffractive imaging (CDI) and scanning transmission x-ray microscopy (STXM) are two popular microscopy techniques that have evolved quite independently. CDI promises to reach resolutions below 10 nanometers, but the reconstruction procedures put stringent requirements on data quality and sample preparation. In contrast, STXM features straightforward data analysis, but its resolution is limited by the spot size on the specimen. We demonstrate a ptychographic imaging method that bridges the gap between CDI and STXM by measuring complete diffraction patterns at each point of a STXM scan. The high penetration power of x-rays in combination with the high spatial resolution will allow investigation of a wide range of complex mesoscopic life and material science specimens, such as embedded semiconductor devices or cellular networks.

SCIENCE VOL 321 18 JULY 2008 379



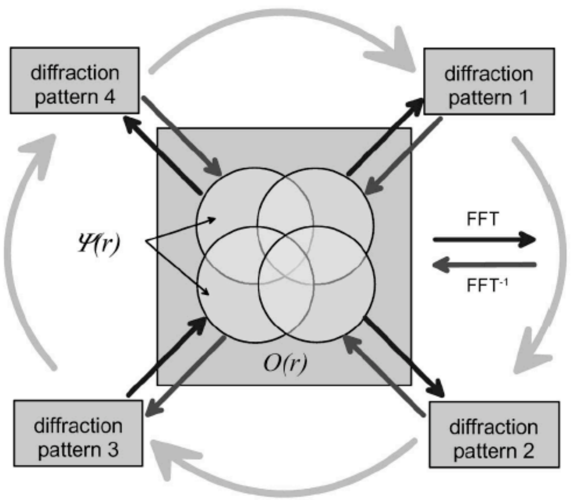
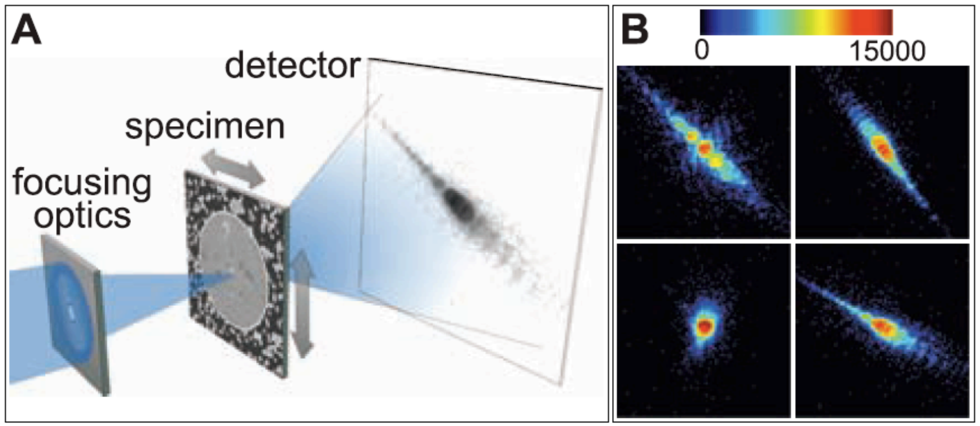
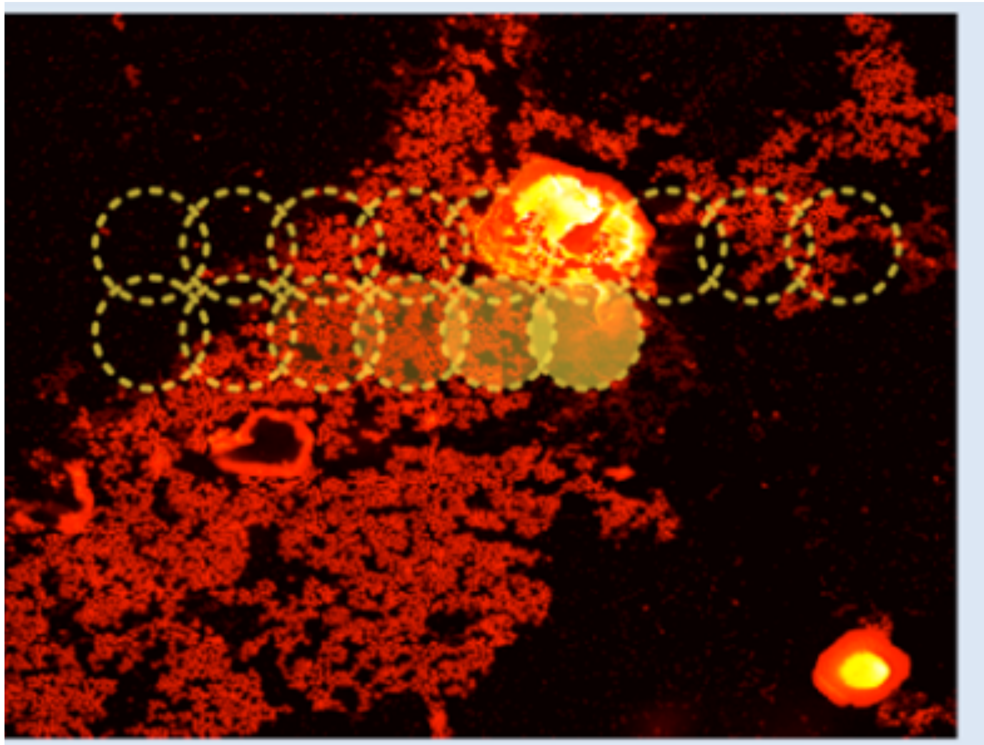
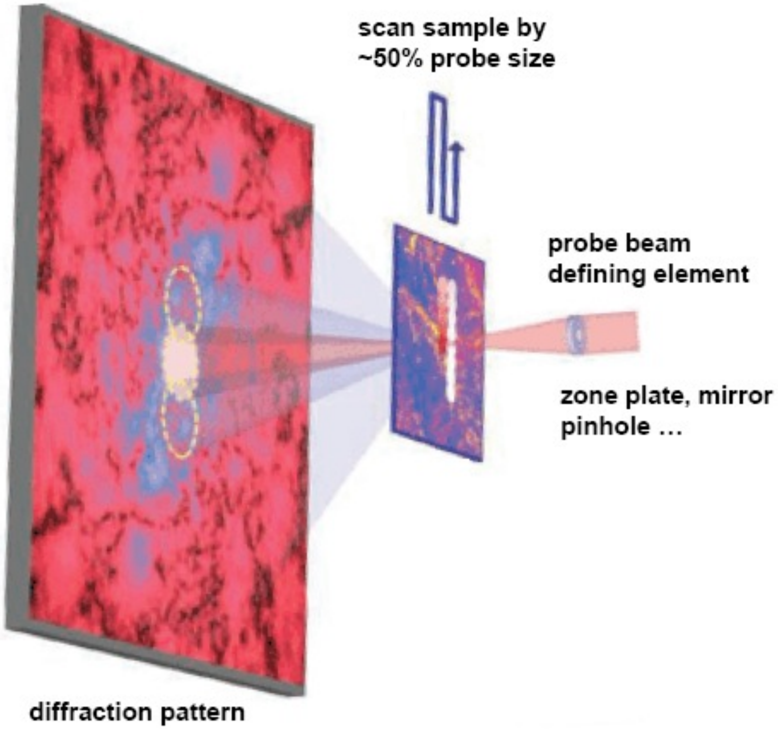


FIG. 2. Diagram of the phase-retrieval algorithm. The outer circular arrows indicate the position stepping within one iteration. The arrows within indicate (inverse) Fourier transforms and the desired input-output information.



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



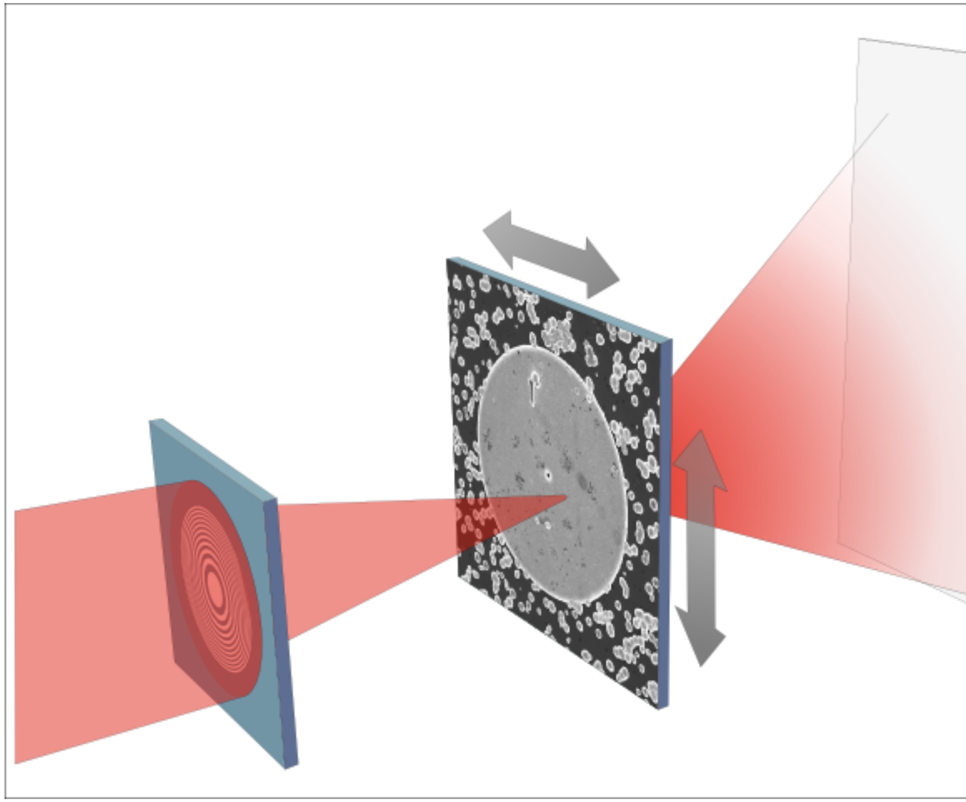


H. Chapman, Science 321, 352 (2008)

Scanning X-ray diffraction microscopy

Ptychography with a focused X-ray probe

Not suitable for flash & destroy

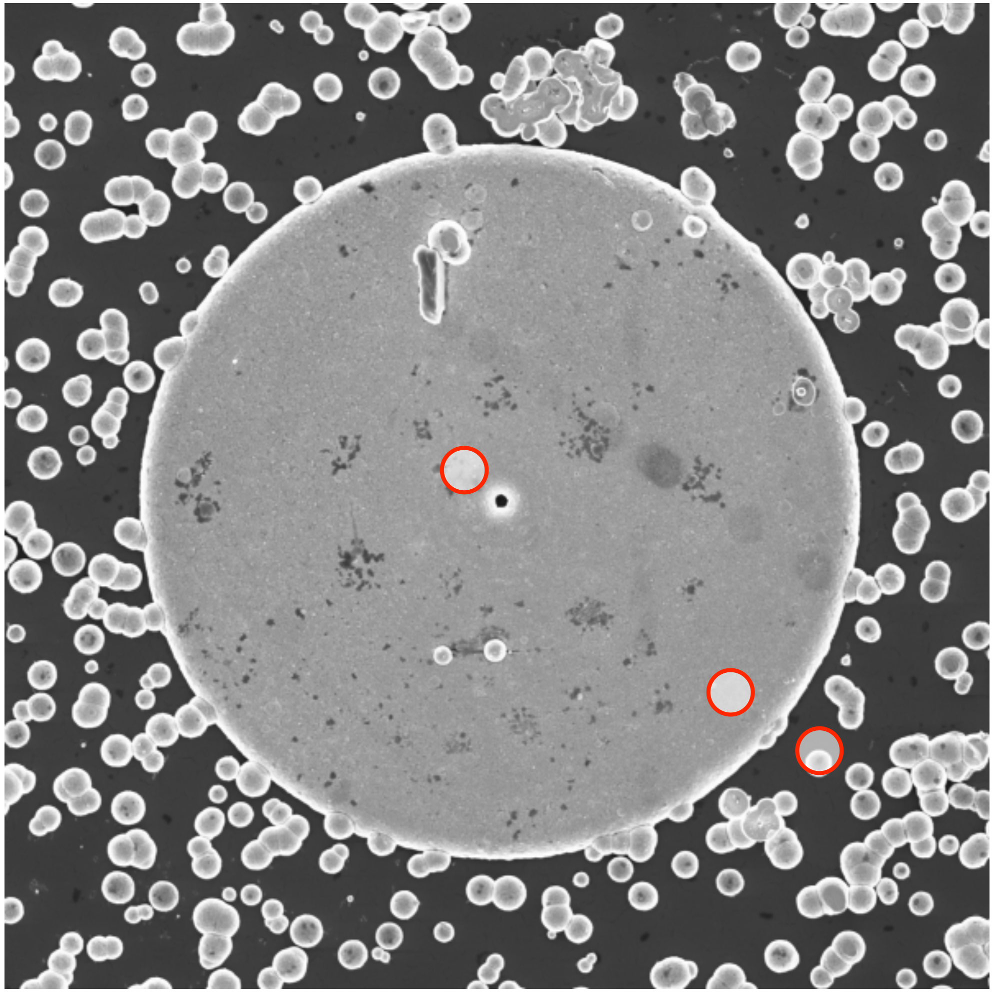


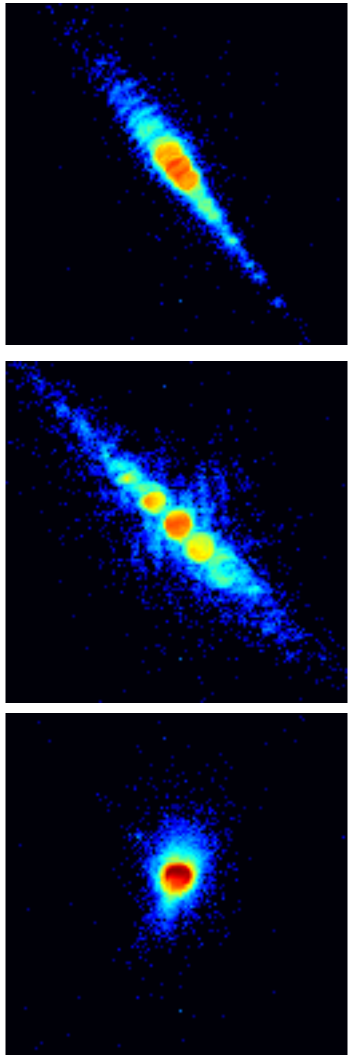
Pilatus 2M

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

Scanning X-ray diffraction microscopy

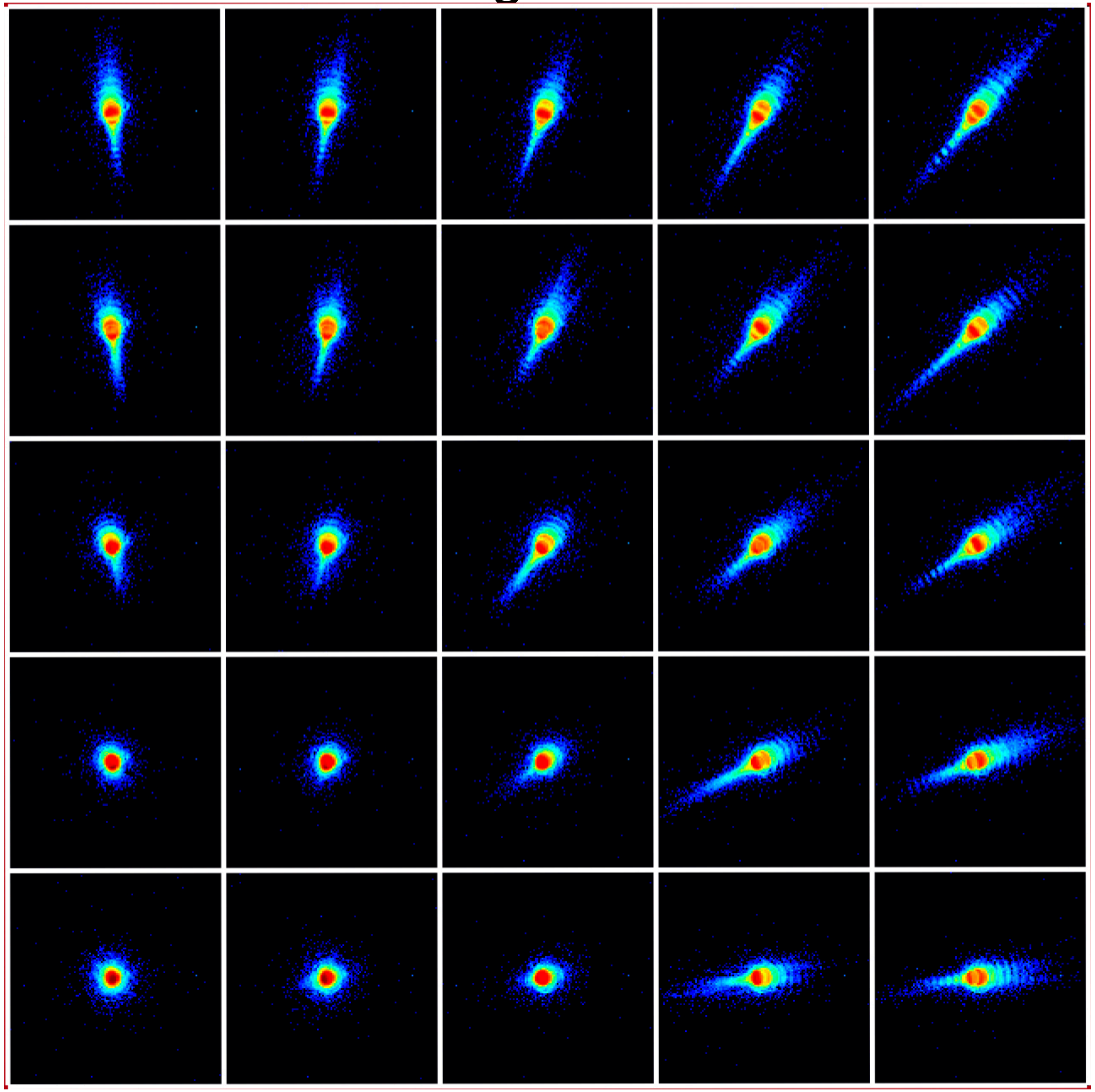
Test specimen : Fresnel zone plate





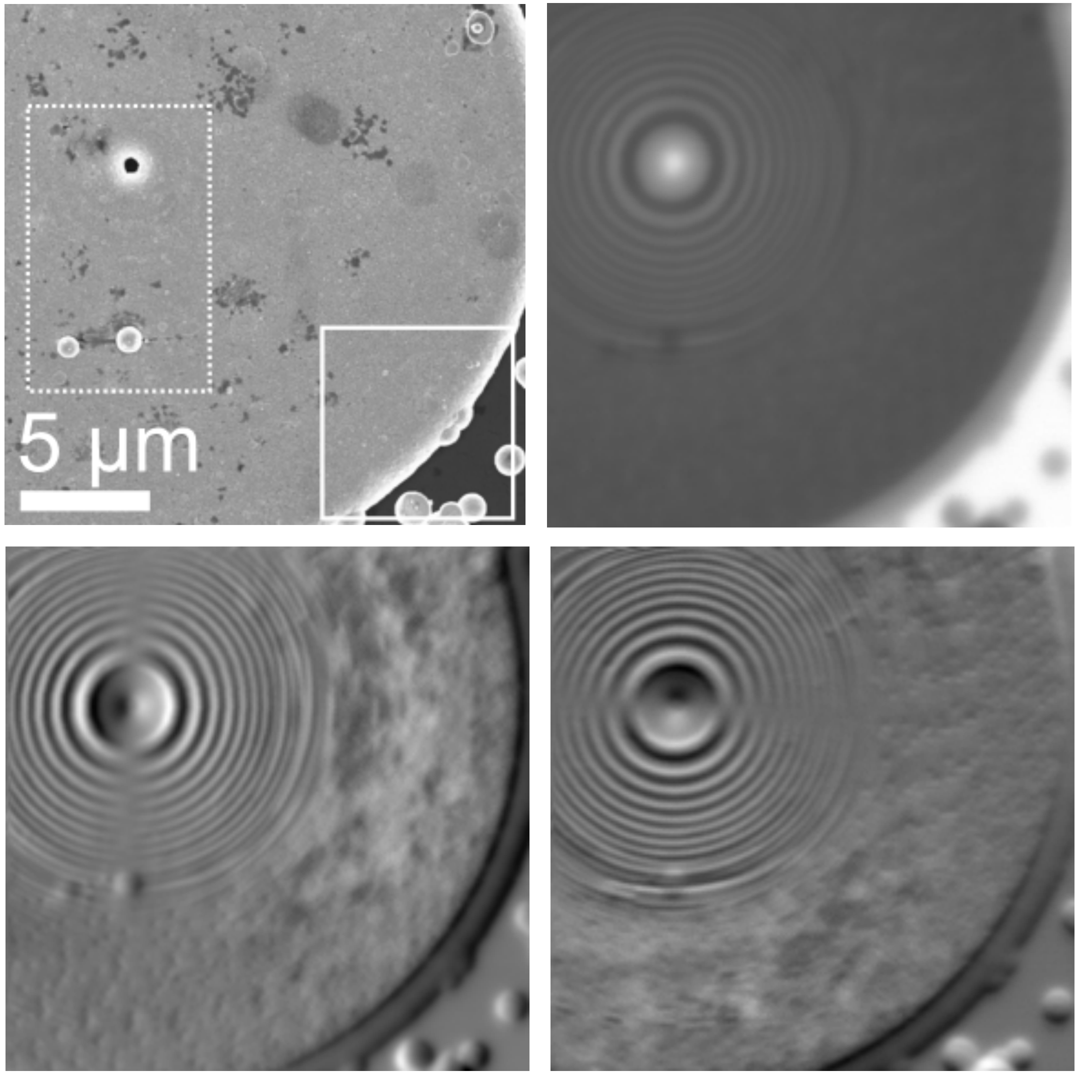
Thibault *et al.*, Science, **321**, 379-382 (2008).

25 out of typically 10000diffraction images used to make a single 2D reconstruction



Scanning X-ray diffraction microscopy

First analysis of the dataset "à la STXM"



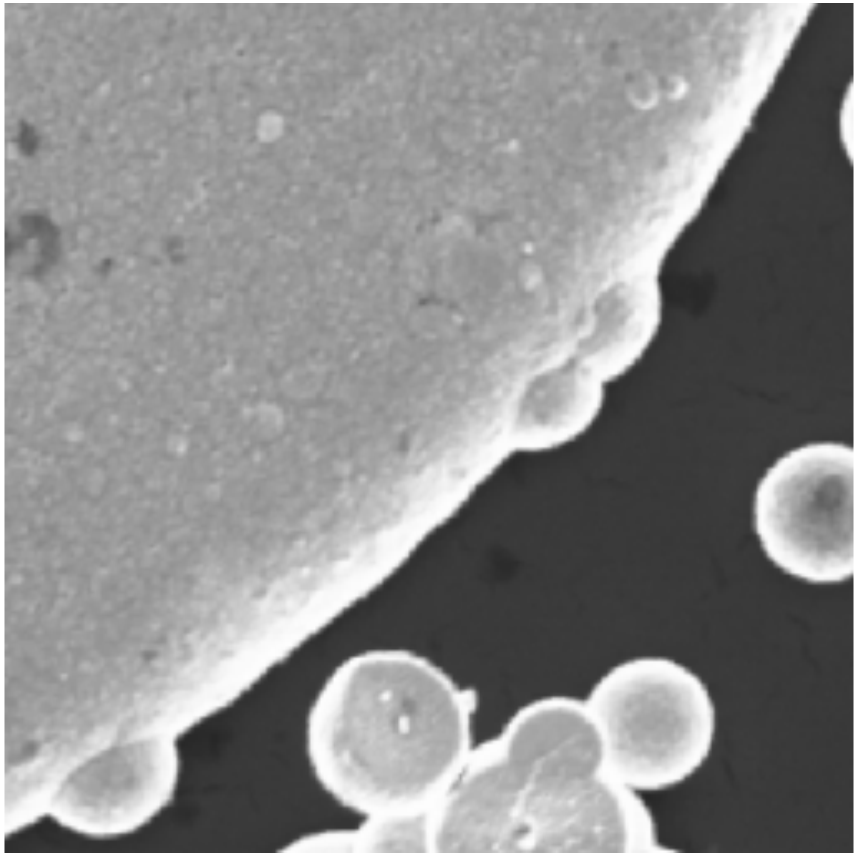
Absorption

Differential phase contrast

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

Scanning X-ray diffraction microscopy

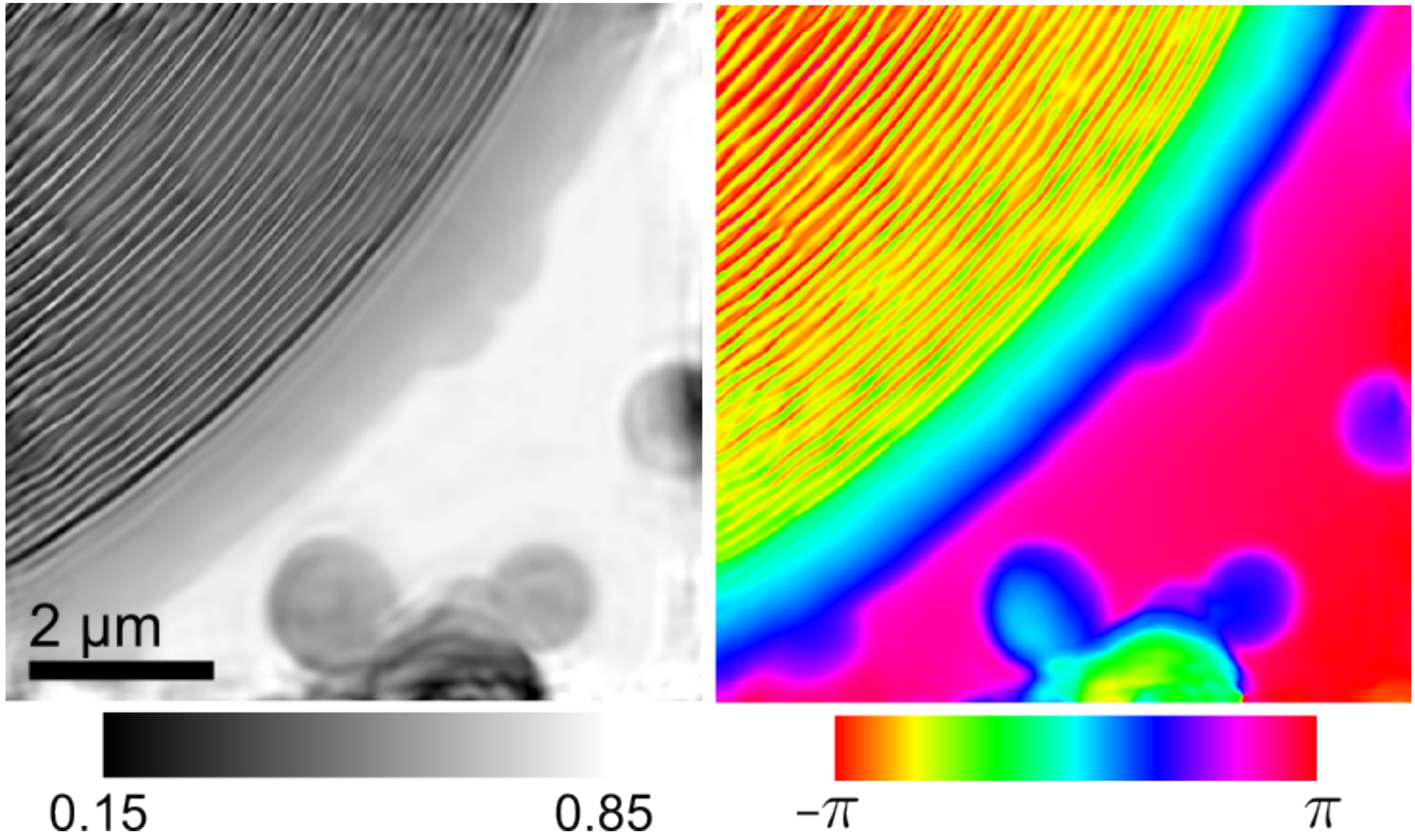
Reconstruction of a selected region



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

Scanning X-ray diffraction microscopy

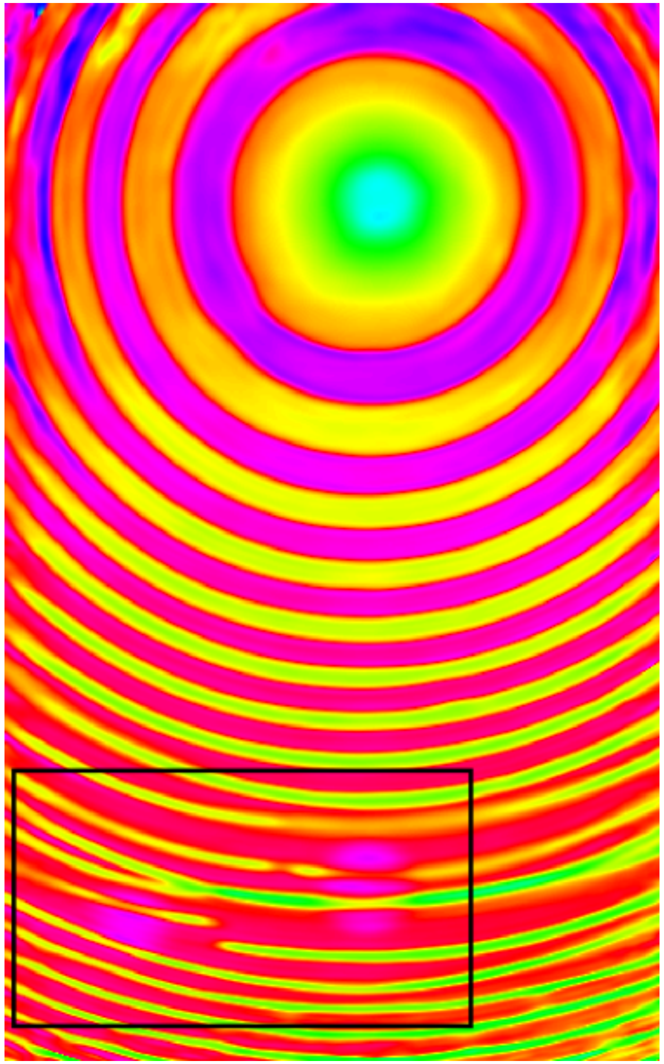
Reconstruction of a selected region

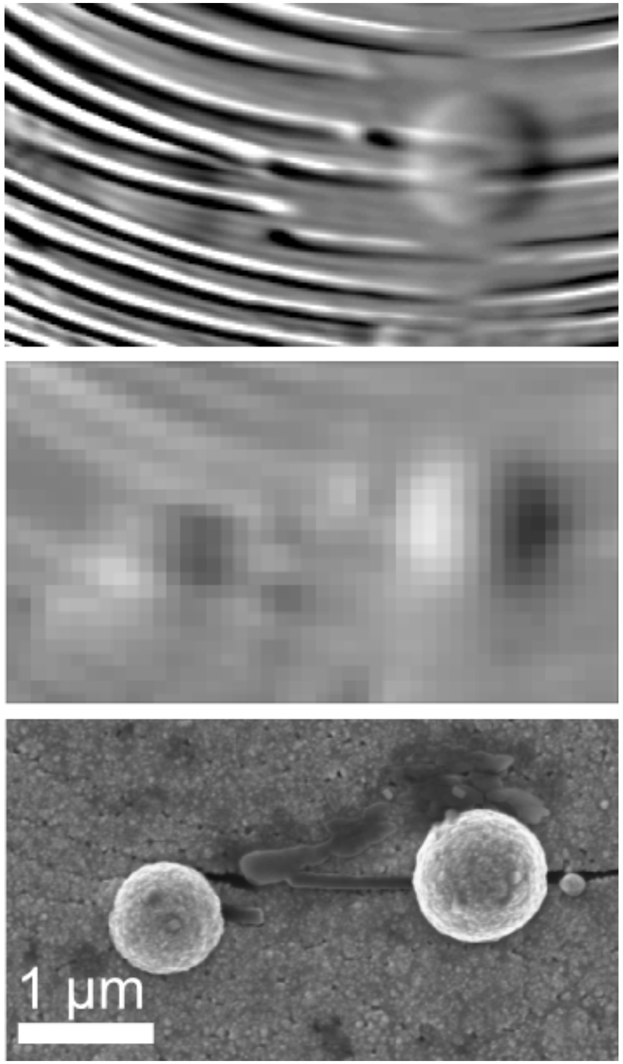


Thibault *et al.*, Science, **321**, 379-382 (2008).

Scanning X-ray diffraction microscopy

Resolution enhancement

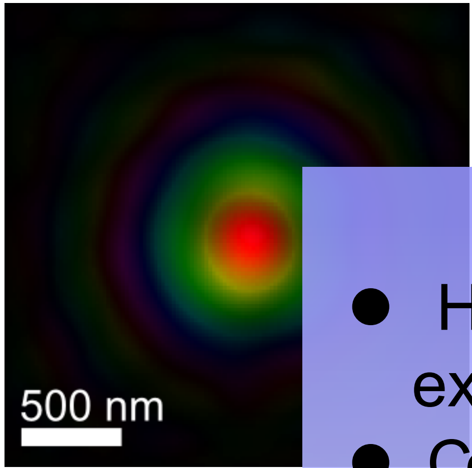




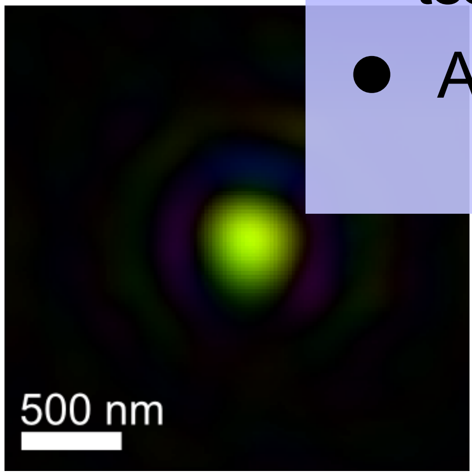
Thibault *et al.*, Science, **321**, 379-382 (2008).

Scanning X-ray diffraction microscopy

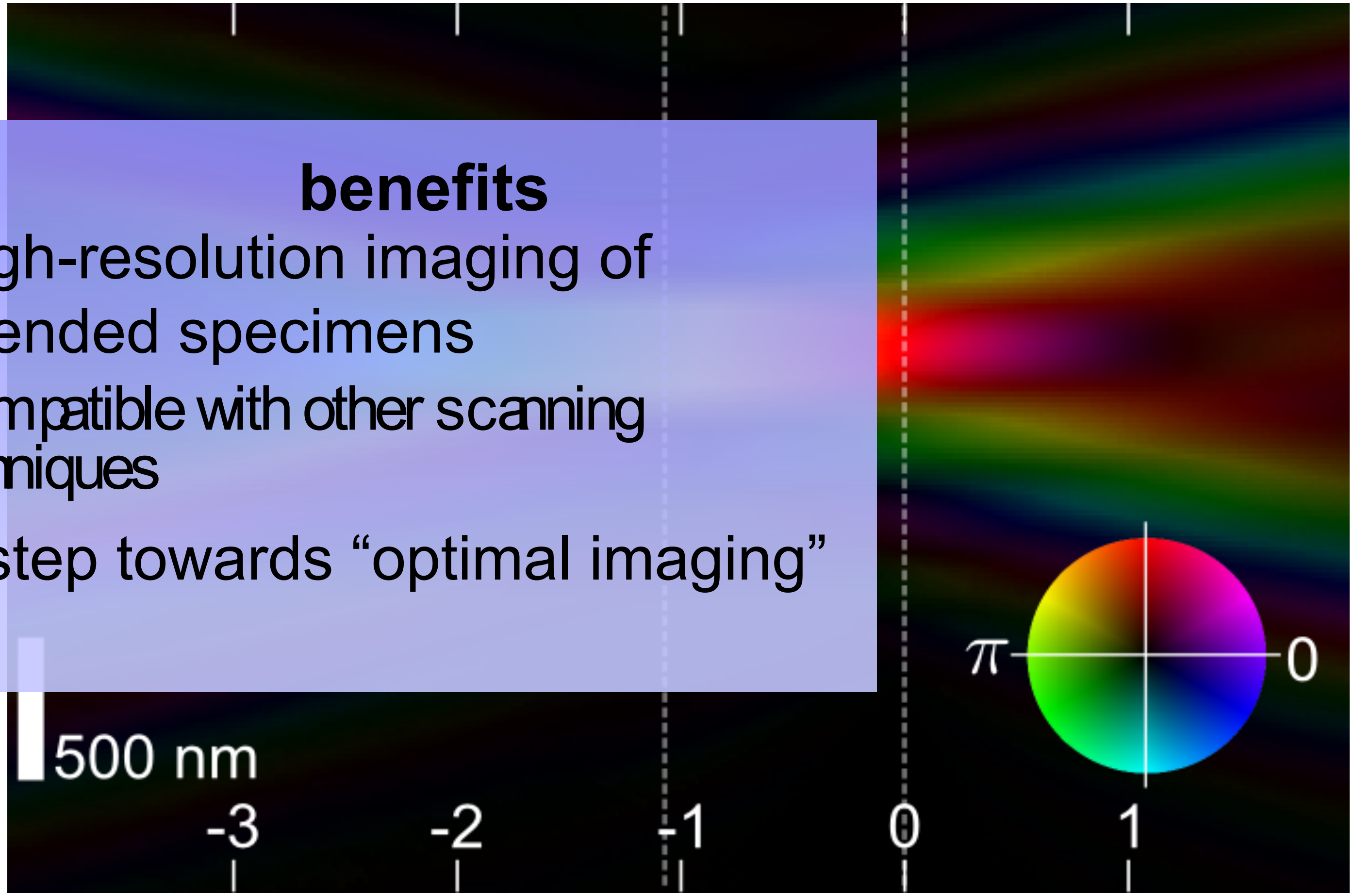
simultaneous retrieval of the probe



probe



propagated in the focal plane



propagation distance (mm)

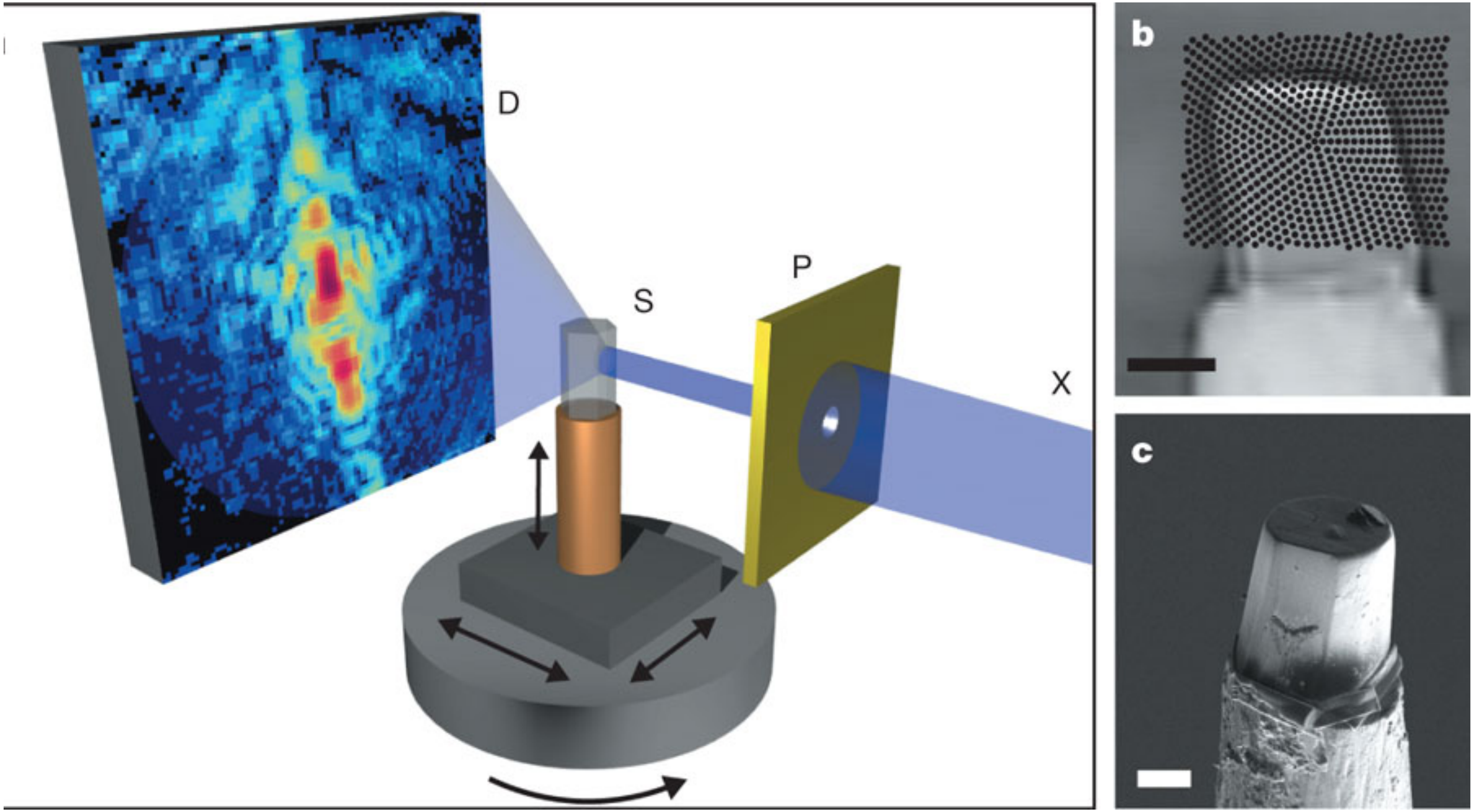
benefits

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

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

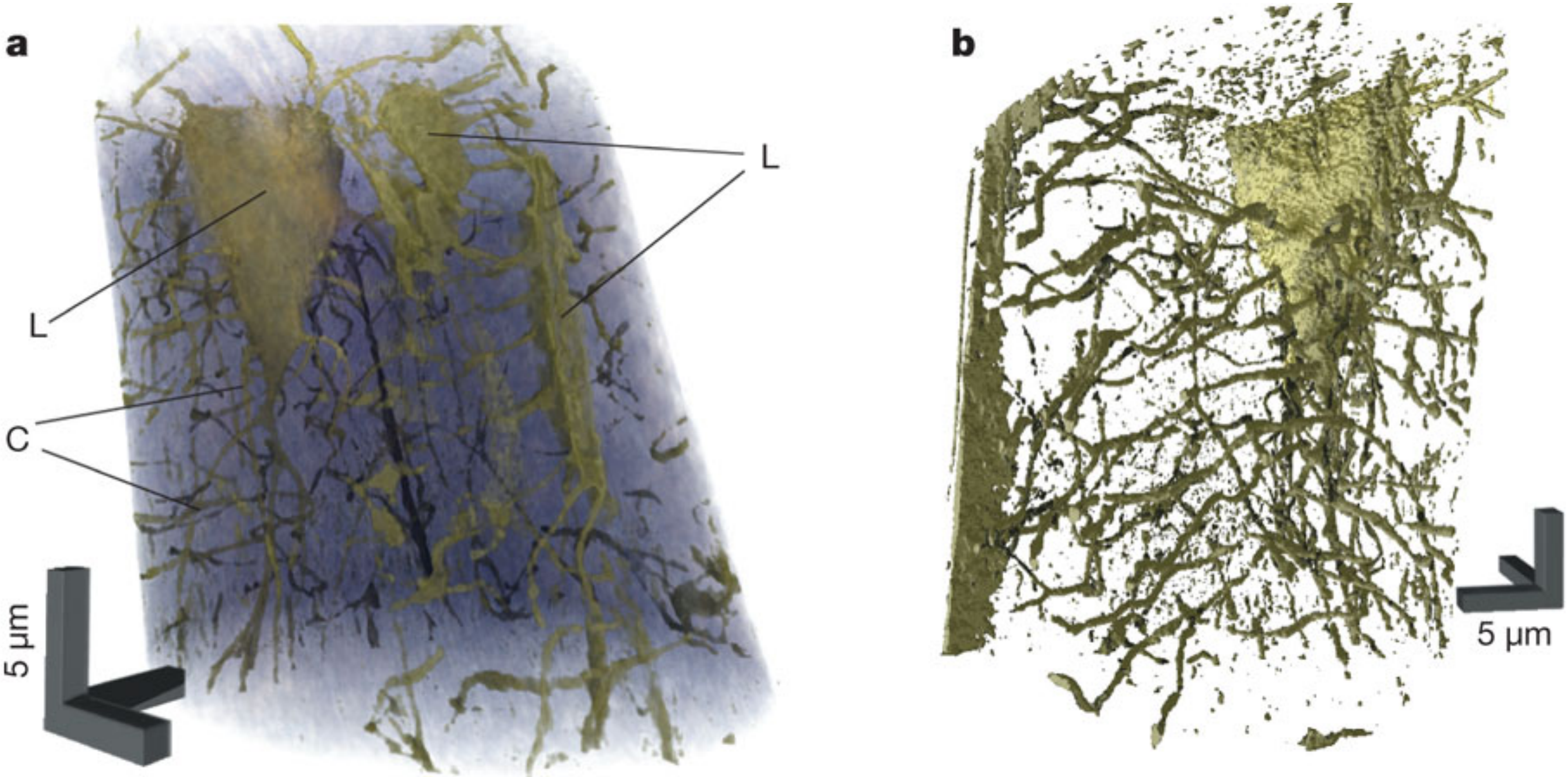
Martin Dierolf, Andreas Menzel, Pierre Thibault, Philipp Schneider,
Cameron M. Kewish,, Roger Wepf, Oliver Bunk & Franz Pfeiffer

Nature 467, 436–439 (23 September 2010)



704 scan points, 181 angles PILATUS 2M detector

3D rendering of the tomographic reconstruction.

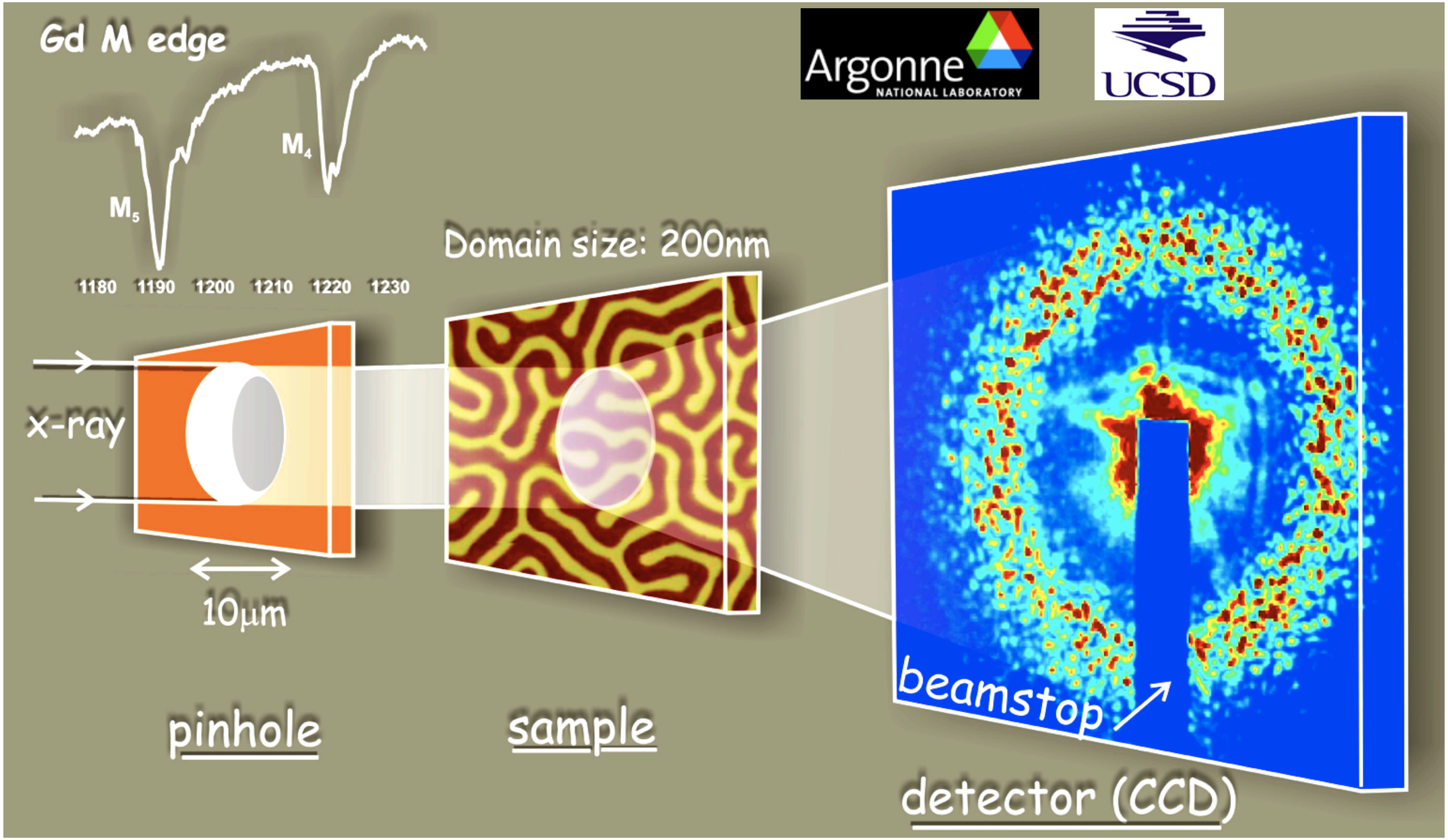


M Dierolf *et al. Nature* **467**, 436-439 (2010) doi:10.1038/nature09419

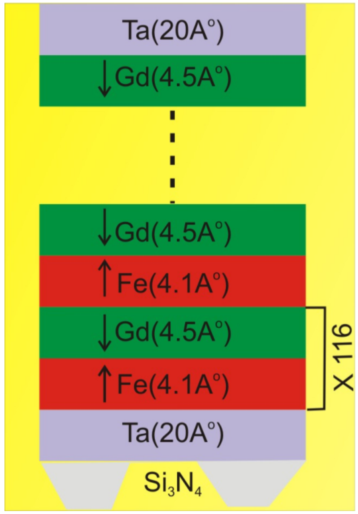
nature

Magnetic imaging by coherent x-ray diffraction

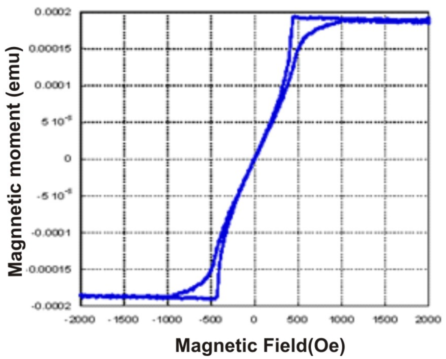
Ash Tripathi, SangSoo Kim, Ian McNulty (ANL)
Jyoti Mohanty, Eric Shipton, Eric Fullerton, Oleg Shpyrko (UCSD)



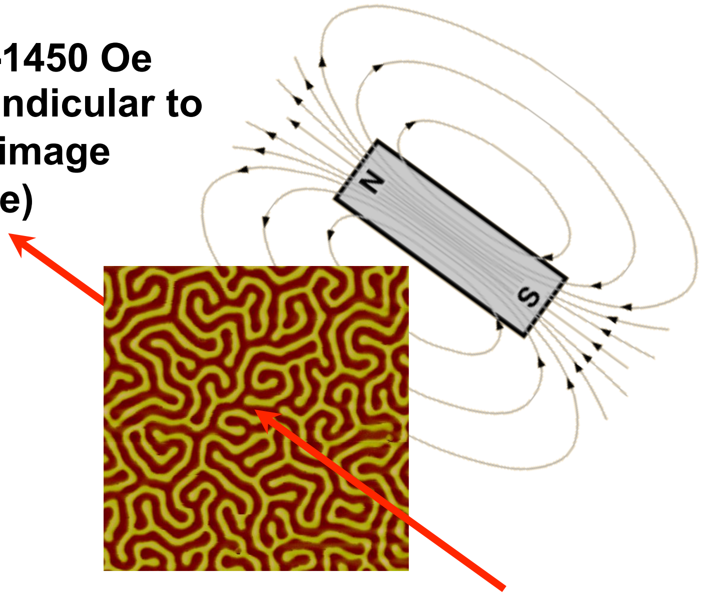
Resonant coherent diffraction enables element-specific tracking of domain formation and transport



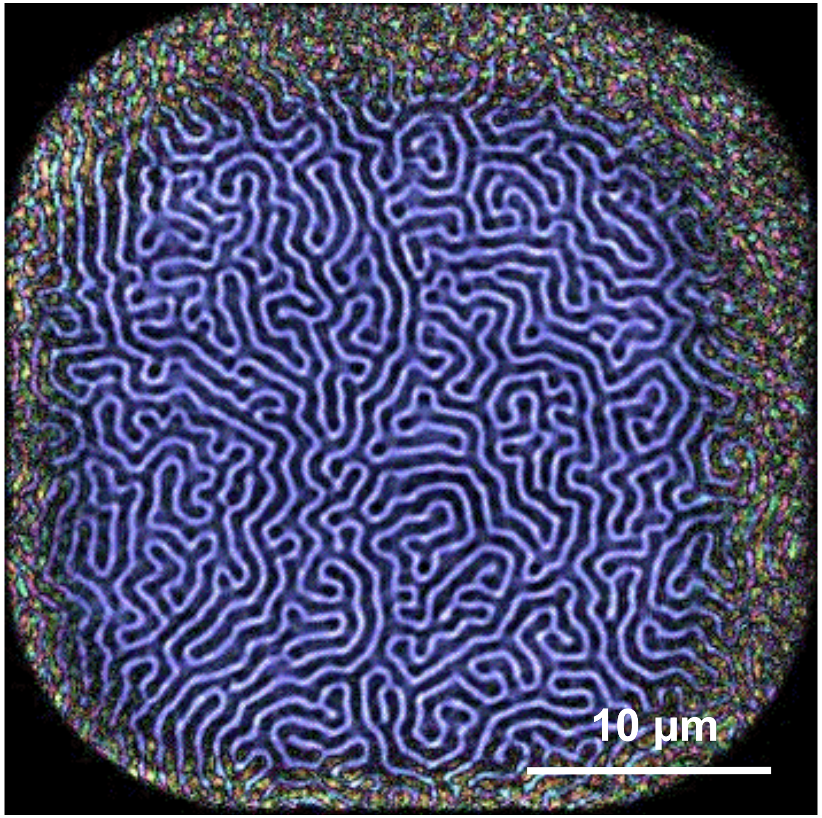
Ferromagnetic "worm" domains form in GdFe multilayer film due to perpendicular anisotropy



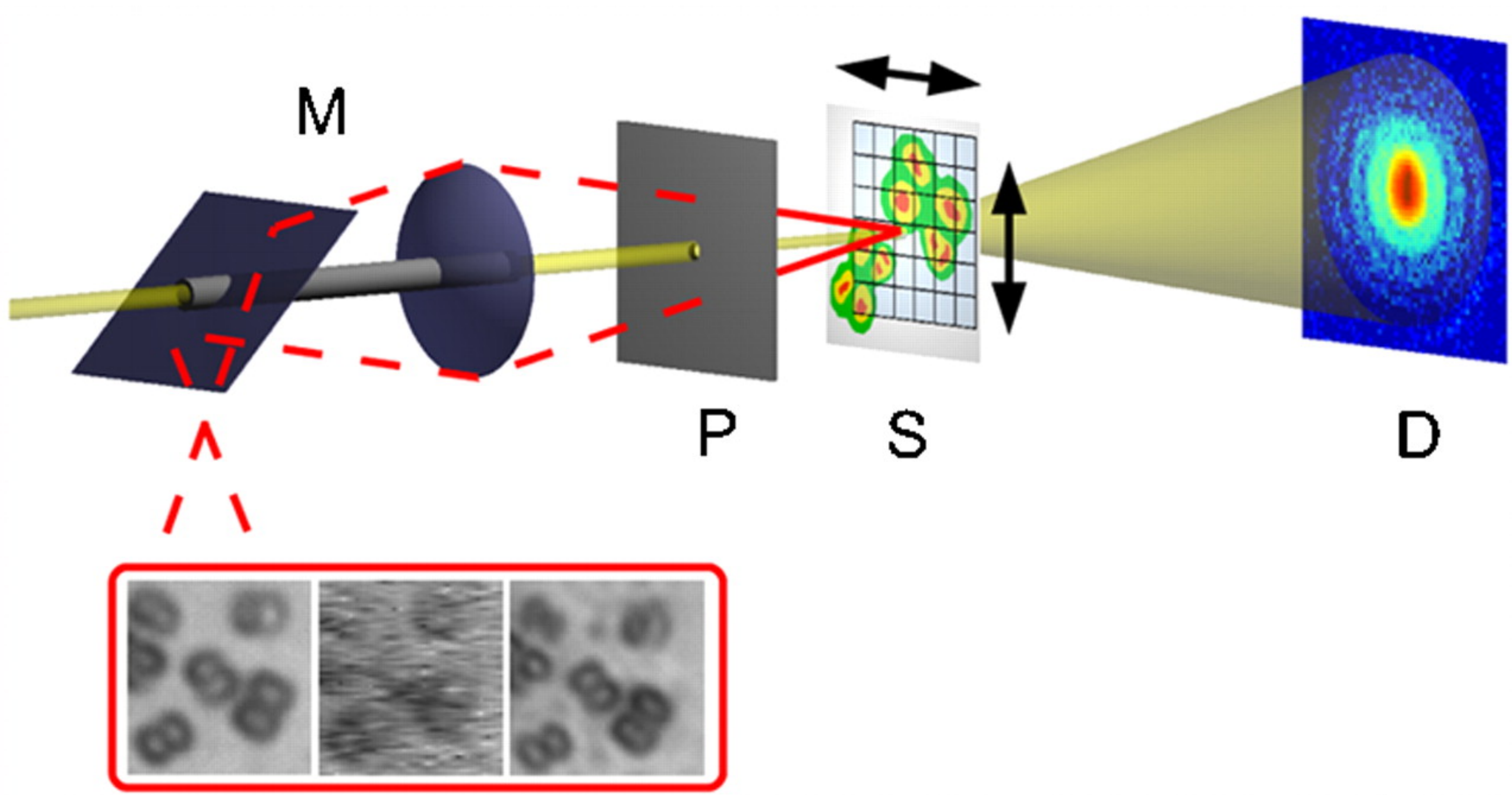
Apply 300-1450 Oe field perpendicular to film (MFM image shown here)



Ptychographic reconstruction of speckle pattern scan series

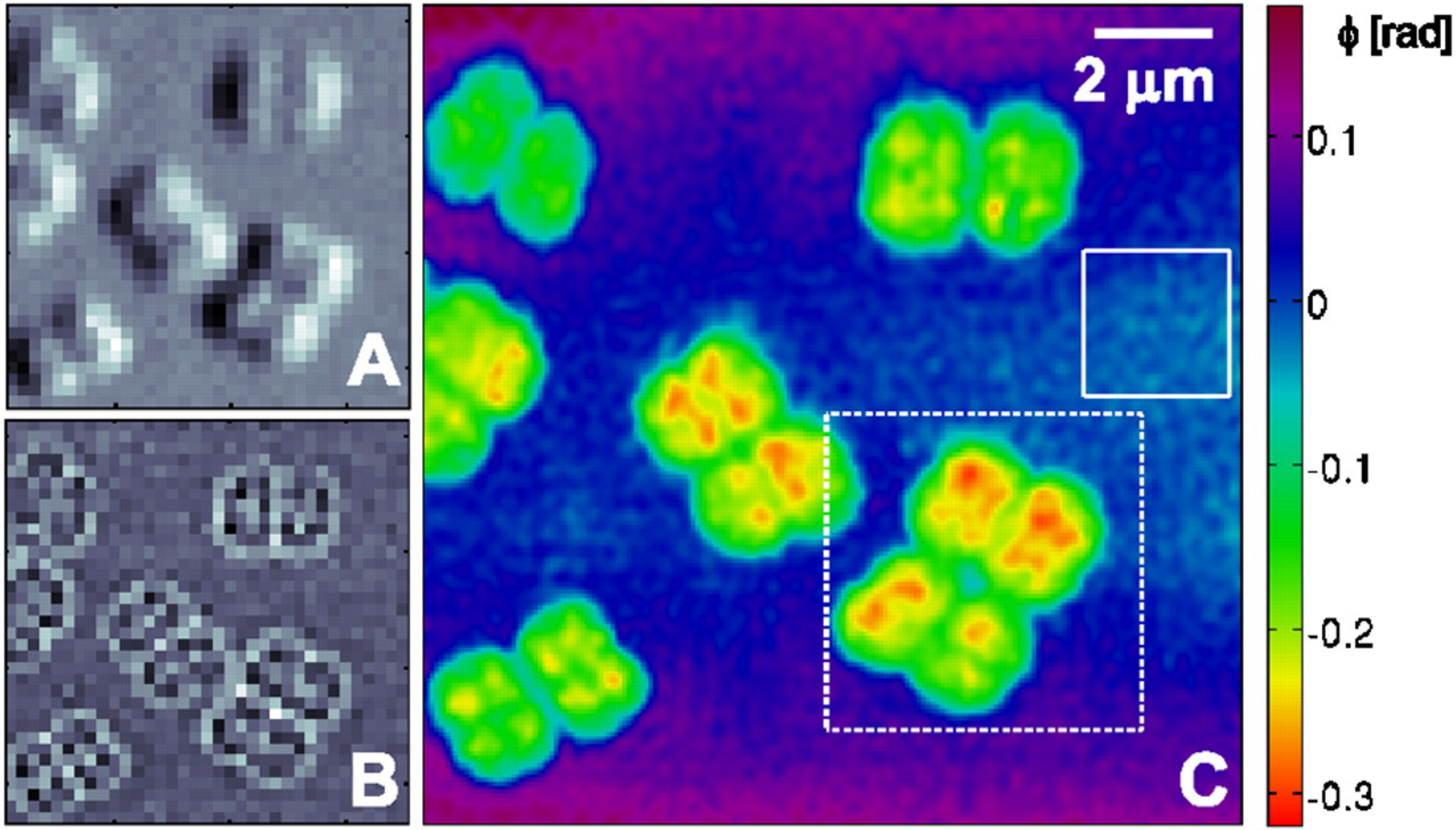


Experimental setup.



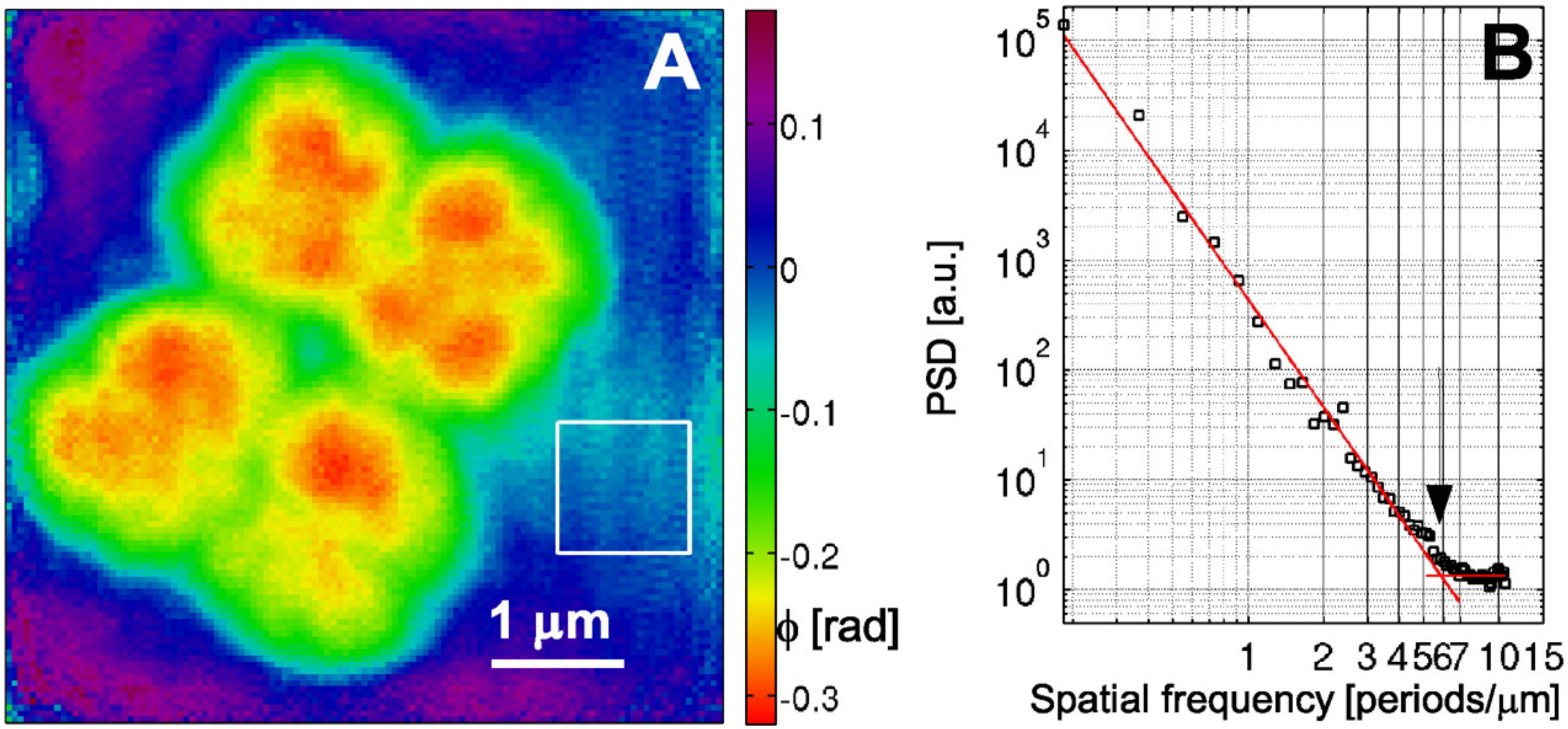
Giewekemeyer K et al. PNAS 2010;107:529-534

Experimental setup.



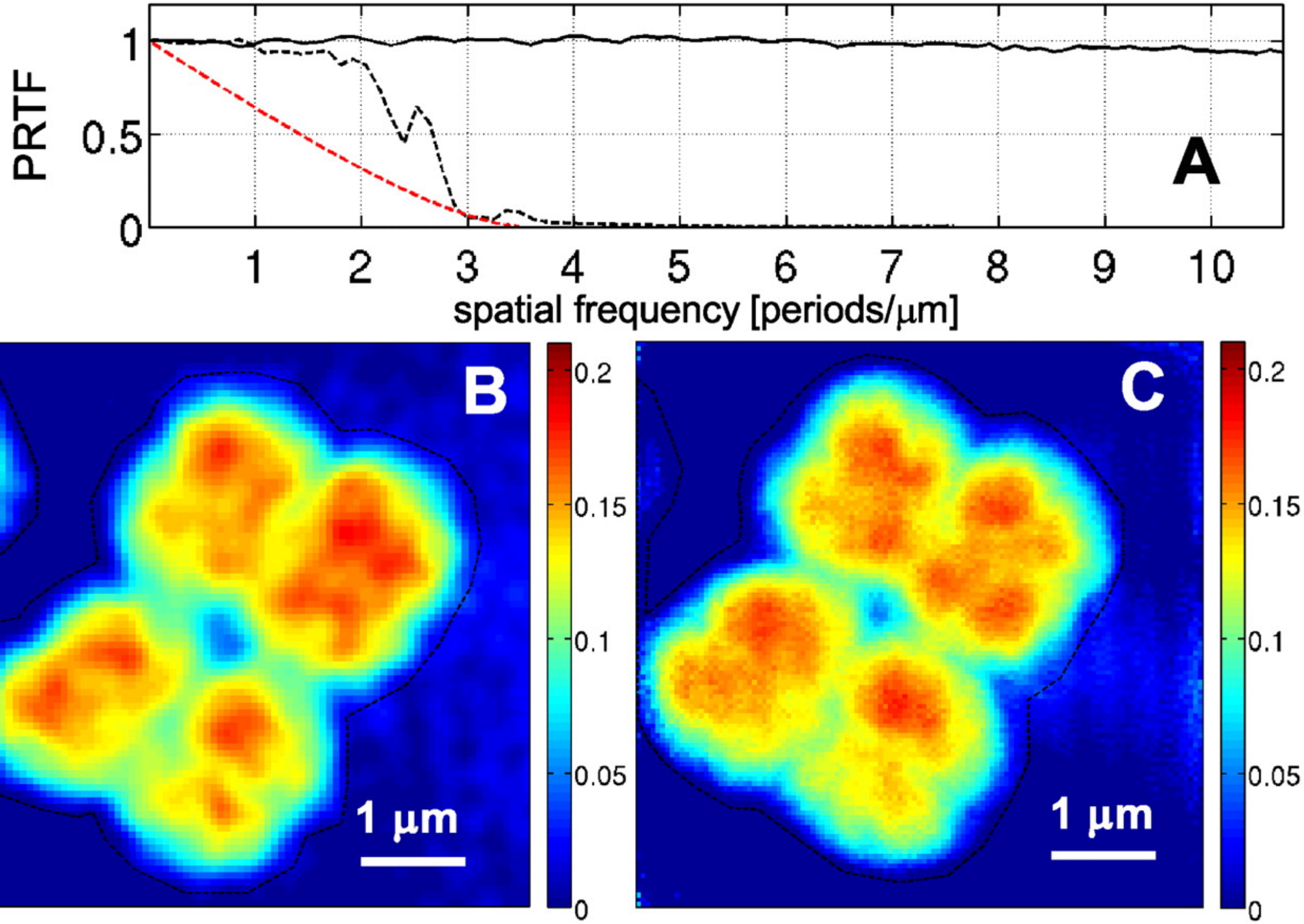
Giewekemeyer K et al. PNAS 2010;107:529-534

Experimental setup.



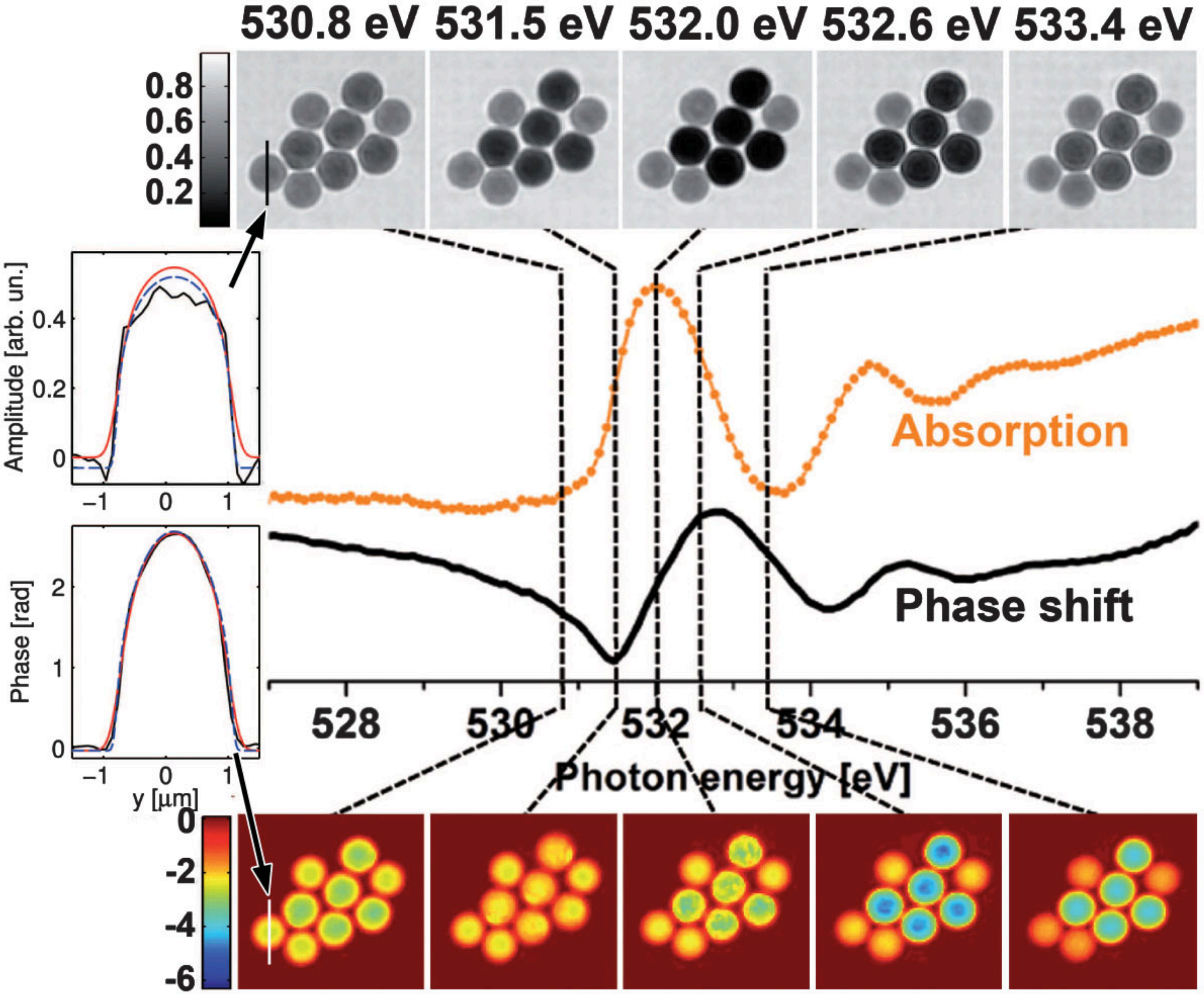
Giewekemeyer K et al. PNAS 2010;107:529-534

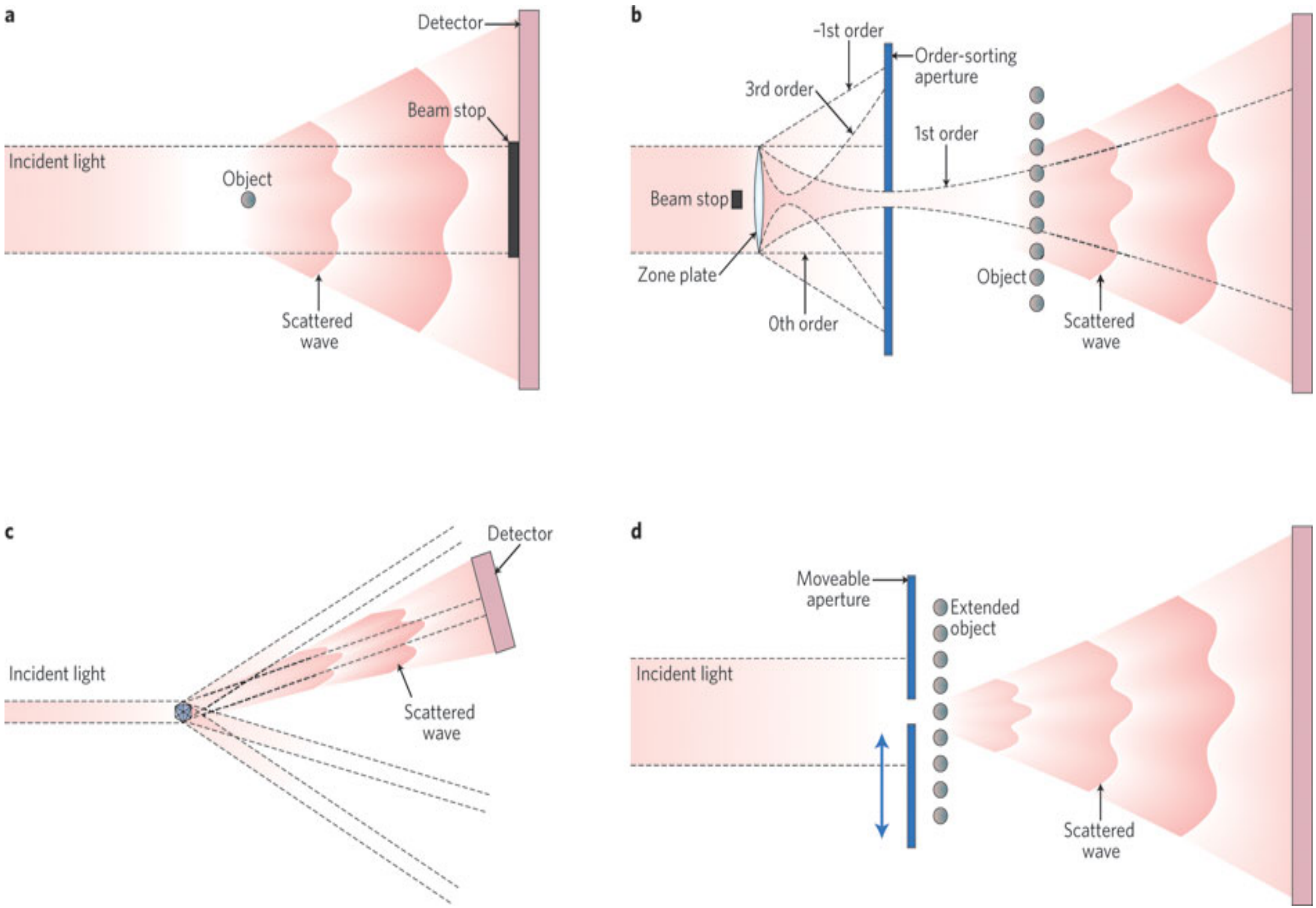
(A) PRTF, corresponding to the dataset with 1-s dwell time (black dashed line) and 60-s dwell time (solid line).



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

M. Beckers et al., Chemical contrast in Soft X-ray Ptychography
PRL 107, 208101 (2011)





Chapman & Nugent, Nature Photonics 4, 833 (2010)

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…

TXM tomography

SSRL