Three-Dimensional and Spectroscopic Characterization of Devices at the Atomic Scale Using Aberration-Corrected Electron Microscopy

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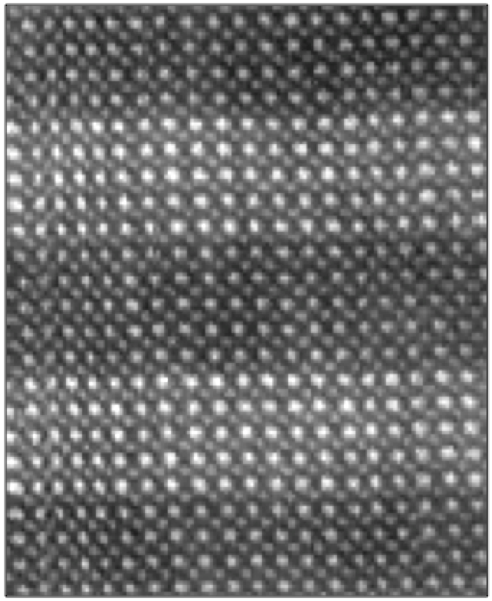
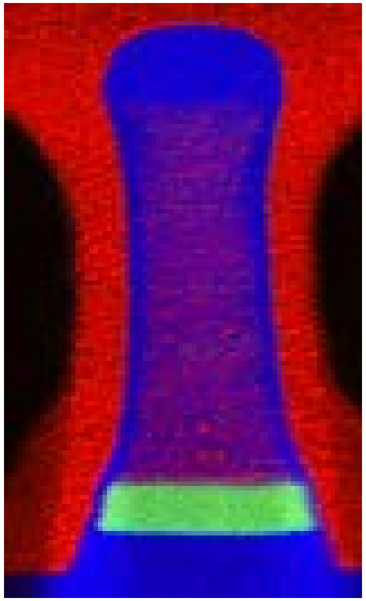
Atomic Imaging

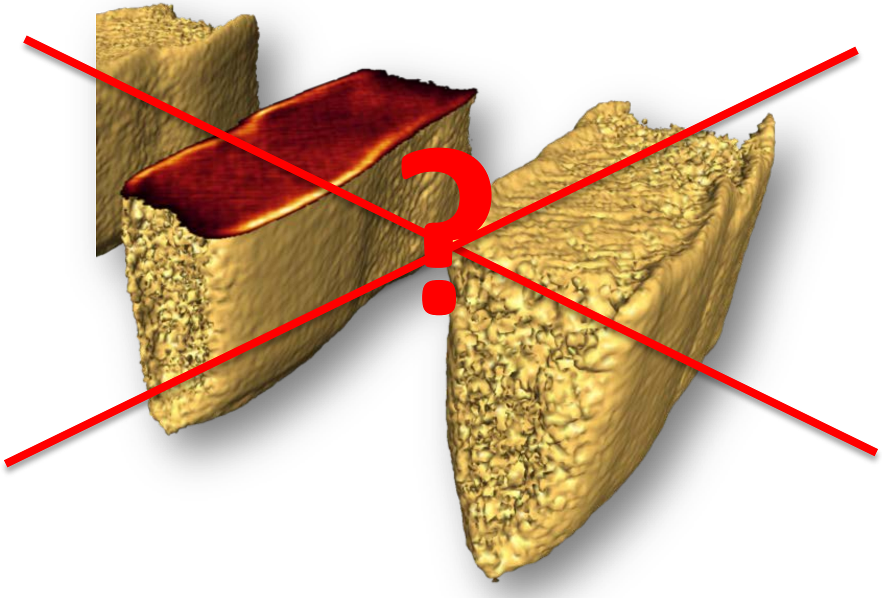


Chemical Mapping



C , SiO_2

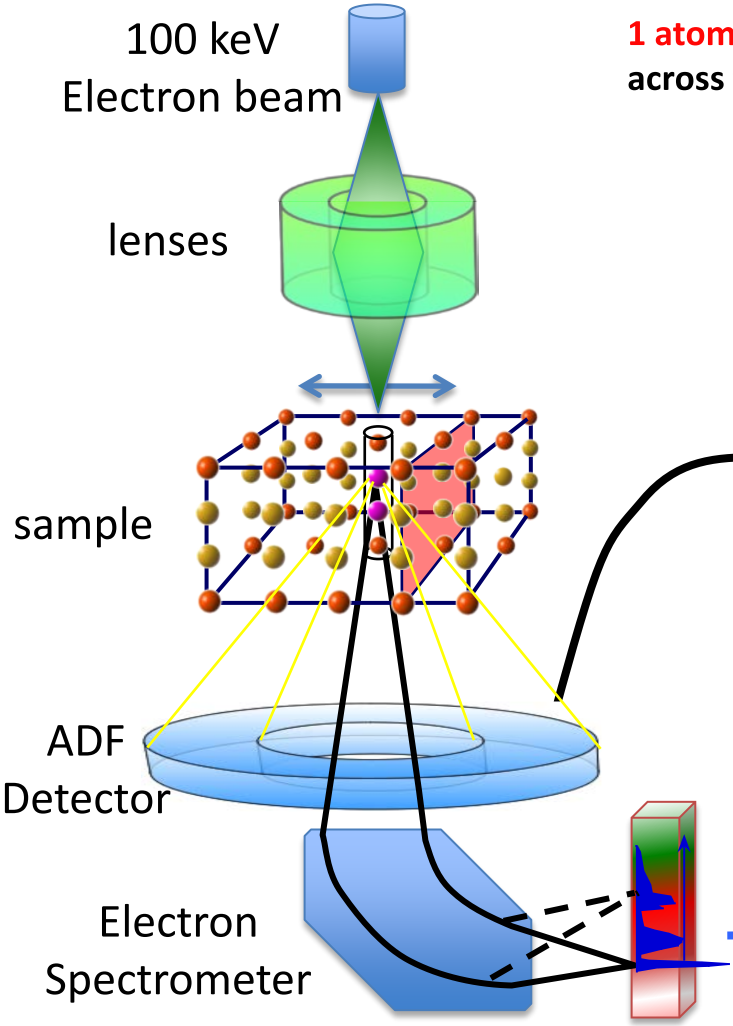
3D Imaging

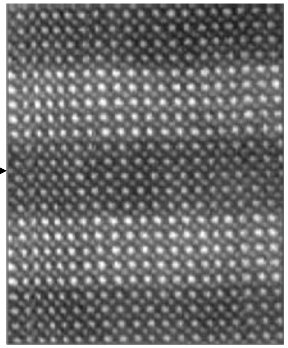


New challenges in 3D imaging with the new generation of microscopes

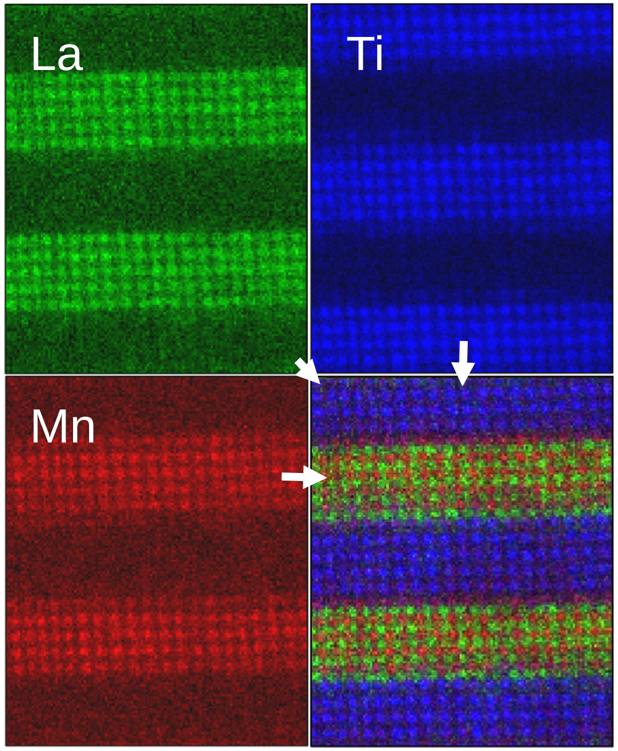
Scanning Transmission Electron Microscopy

1 atom wide (1 Å) beam is scanned across the sample to form a 2-D image





Elastic Scattering
~ "Z contrast"

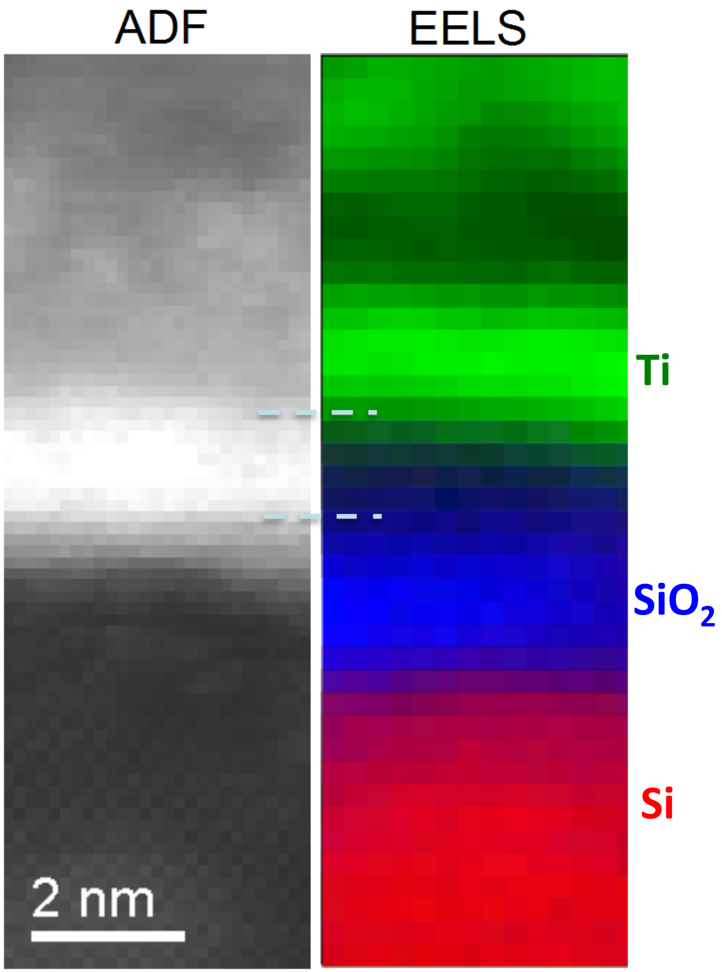


EELS: Chemical Imaging

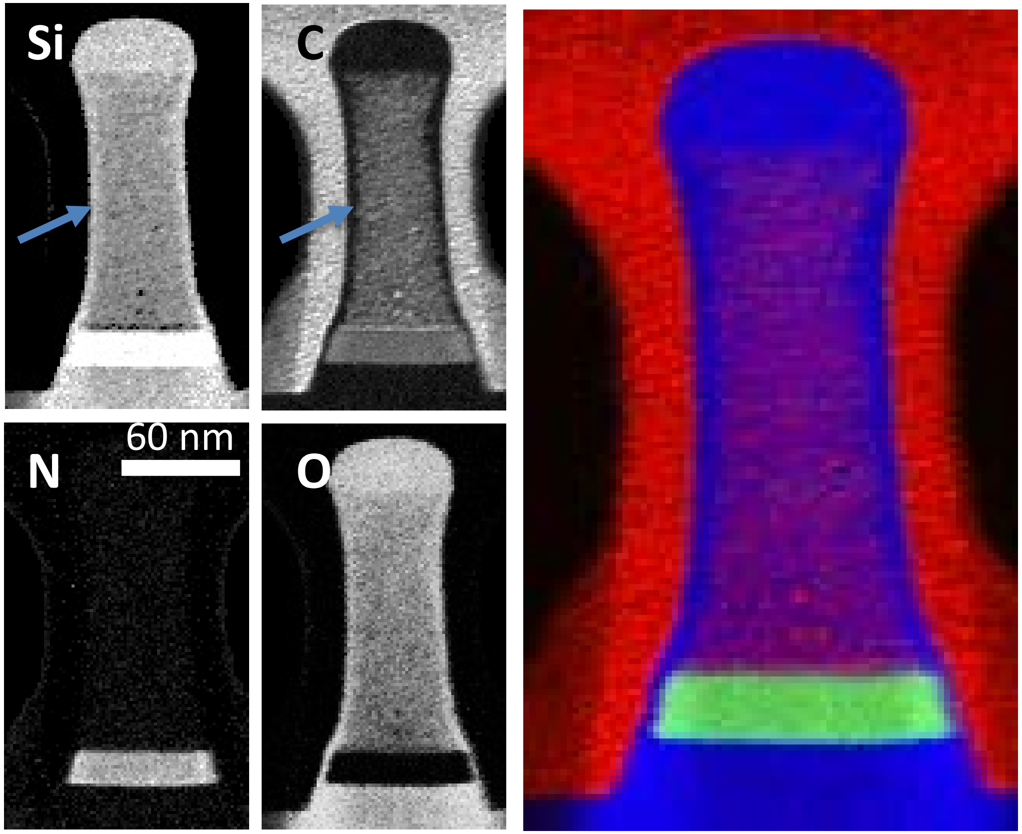
Muller, Kourkoutis, Murfitt, Song, Hwang, Silcox, Dellby, Krivanek, *Science* **319**, 1073 (2008).

Composition Maps from EELS

High-k Gate Stack



Etch Damage in Porous Low-K Dielectric



"a-C-like" C-bonding

"SiC-like" C-bonding

SiO$_2$-like Si bonding

2D➔3D: Electron Tomography

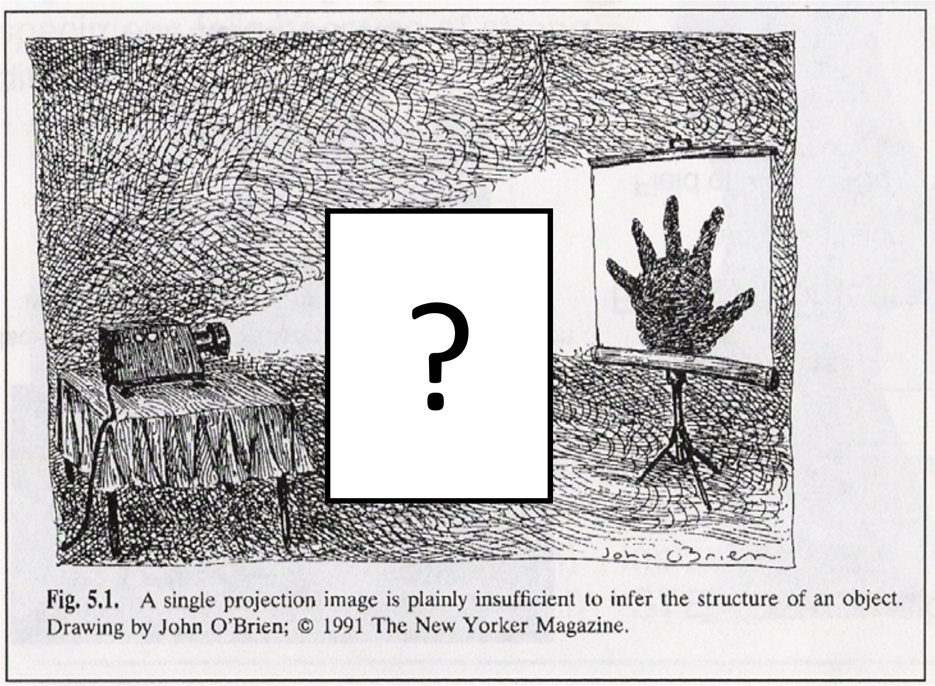
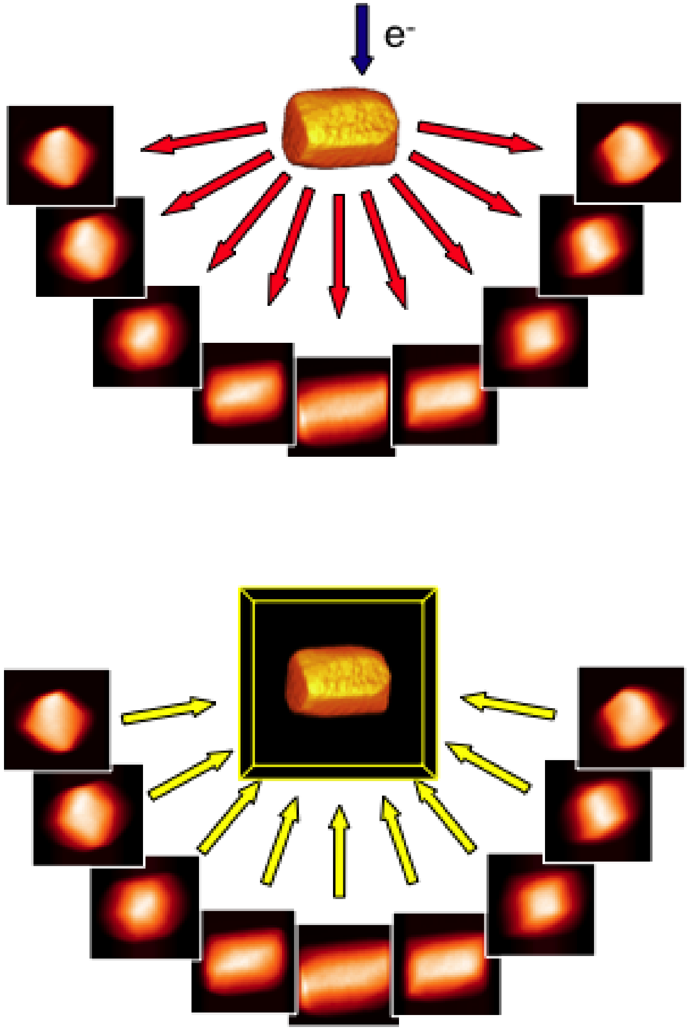


Fig. 5.1. A single projection image is plainly insufficient to infer the structure of an object. Drawing by John O'Brien; © 1991 The New Yorker Magazine.

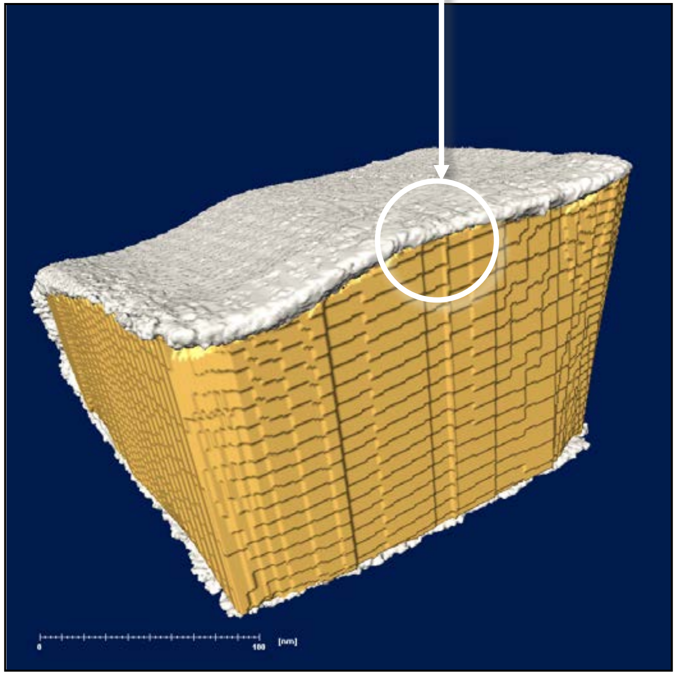
De Rosier, Nature 217, **130** (1968); Hoppe, Naturwissenschaften **55**, 333 (1968)

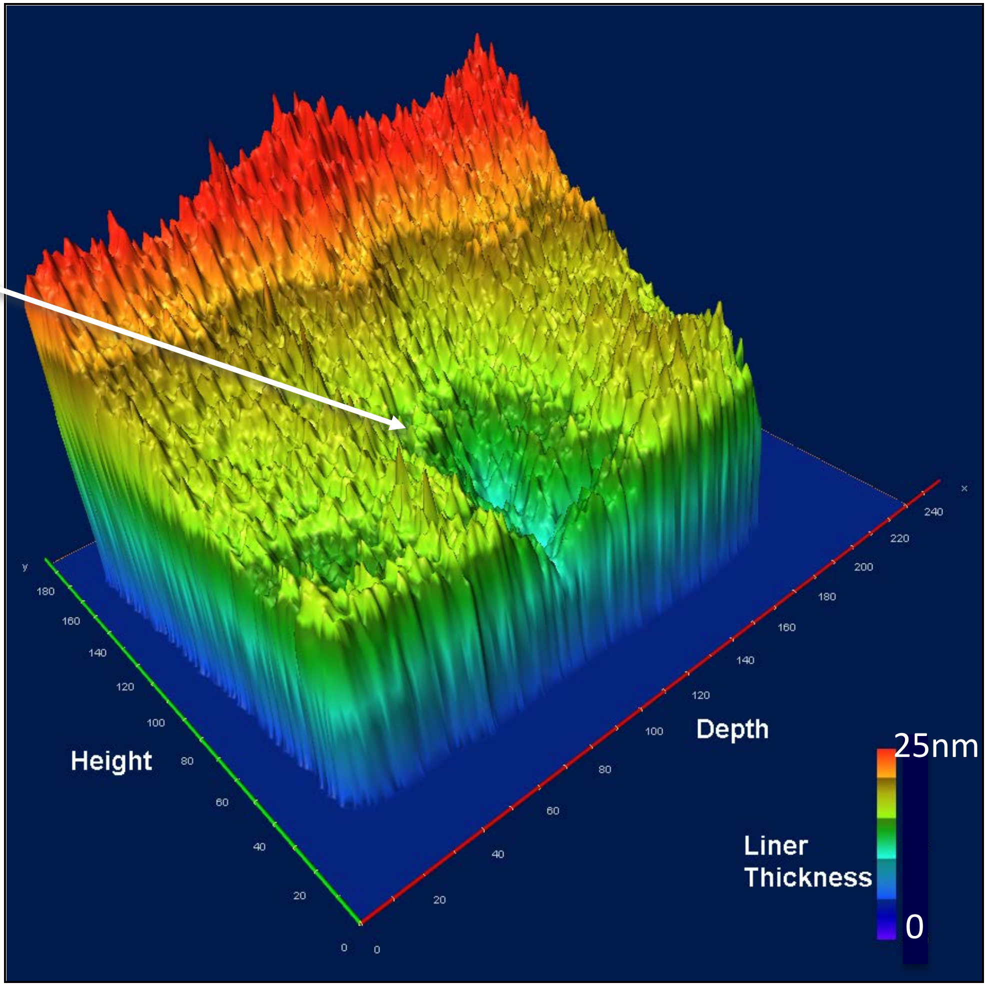


Midgley, Ultramicroscopy **96**, 413 (2003)

Liner Thickness; Position Specific

Liner thins near top of copper line



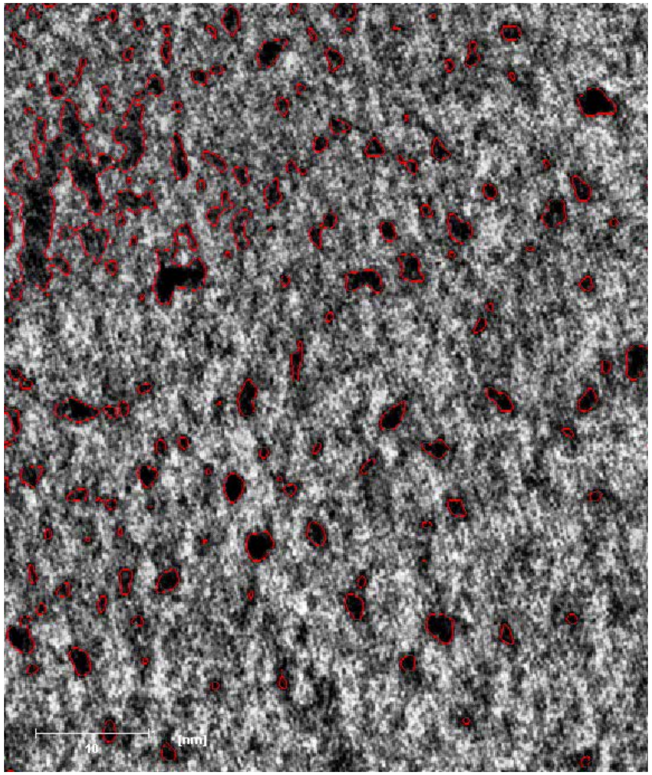


Microscopy and Microanalysis **15**, 244-250 (2009)

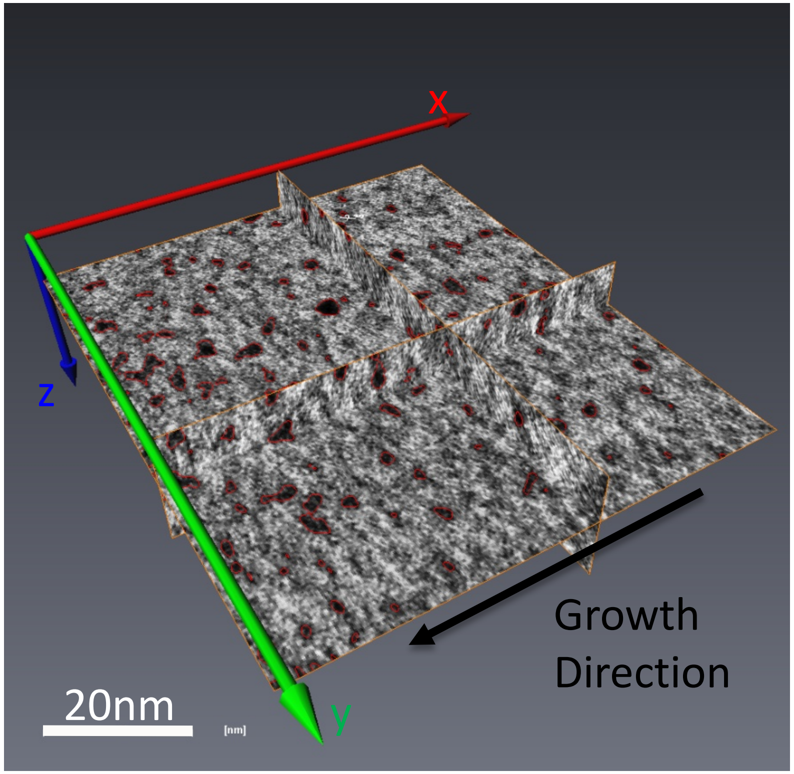
3D Imaging of Porous Low-K Dielectric

Testing Resolution Limits

Single slice



3D visualization

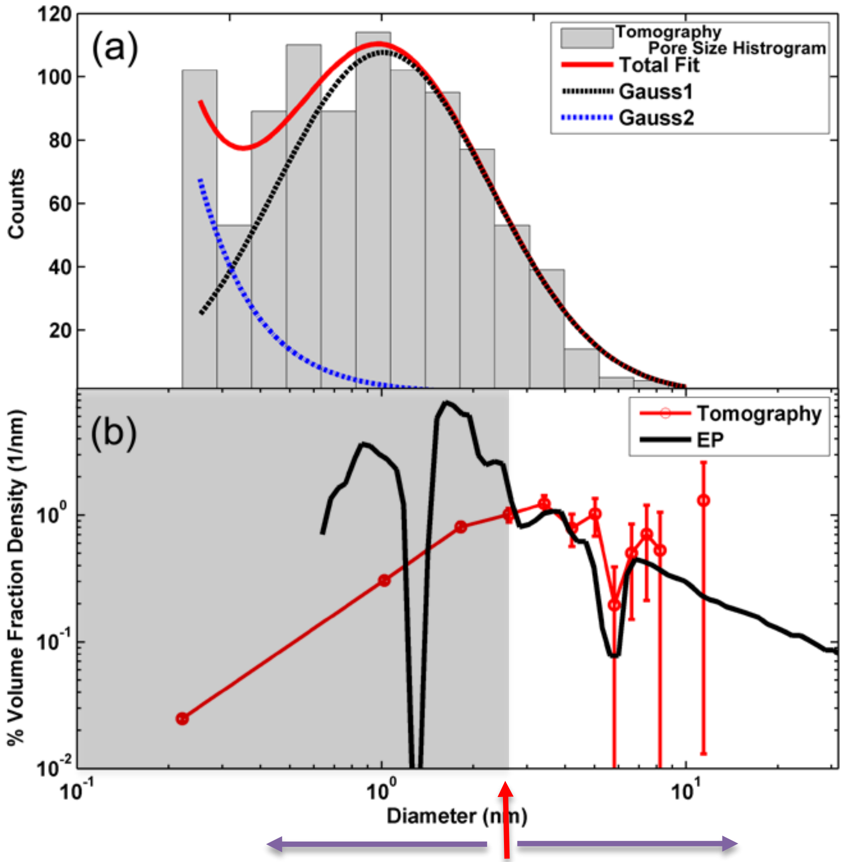


- Pores look predominantly elliptical.
- Despite the presence of some large pore networks, on average there is little multi-pore connectivity
- Pore size, connectivity and porosity can be determined from auto segmented pores.

H. L. Xin et al, *Appl. Phys. Lett.* ***96***, 223108 (2010).

Pore Size Distributions

Comparison with Ellipsometric Porosimetry (Engstrom 1292.047)



Disagreement

Good agreement

- Most Likely Pore Size:

$$1.0nm$$

- 95% of pore diameters:

$$D_{95\%} < 5.0nm$$

- Porosity:

Ellipsometric Porosimetry: 18.4%
Tomography 6.9%

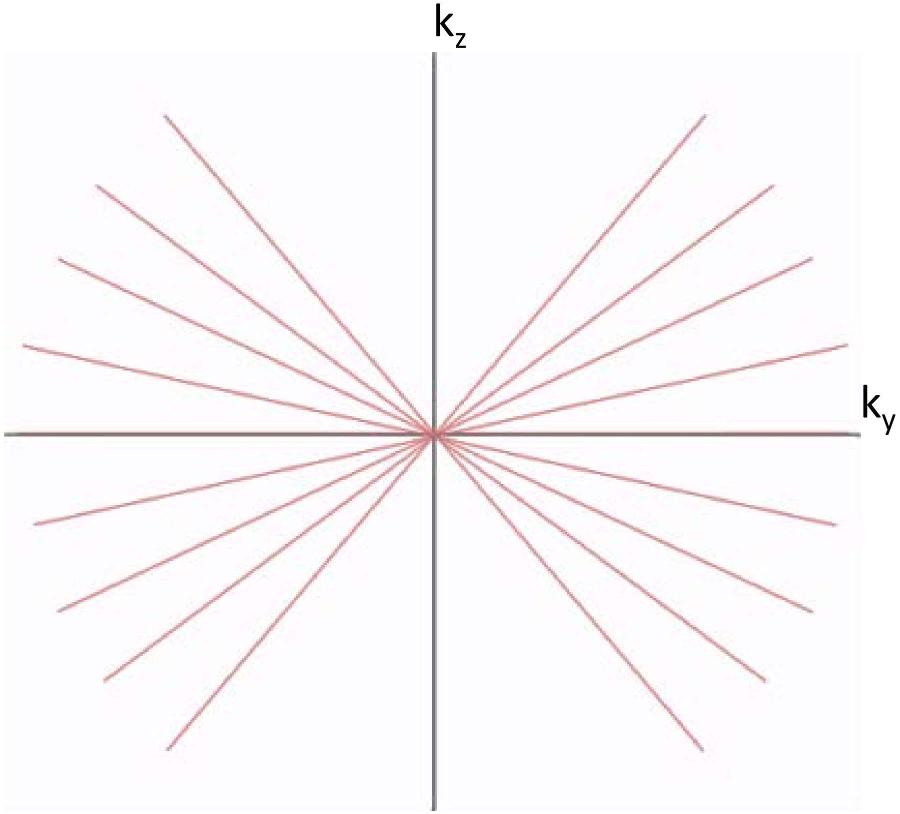
- Both techniques show quantitative agreement in the volume fraction for pore diameters larger than 2.5 nm, but demonstrate large discrepancies for smaller pores.
- Tomography fails to detect pores < 2 nm

(need different segmentation thresholds for different pore sizes)

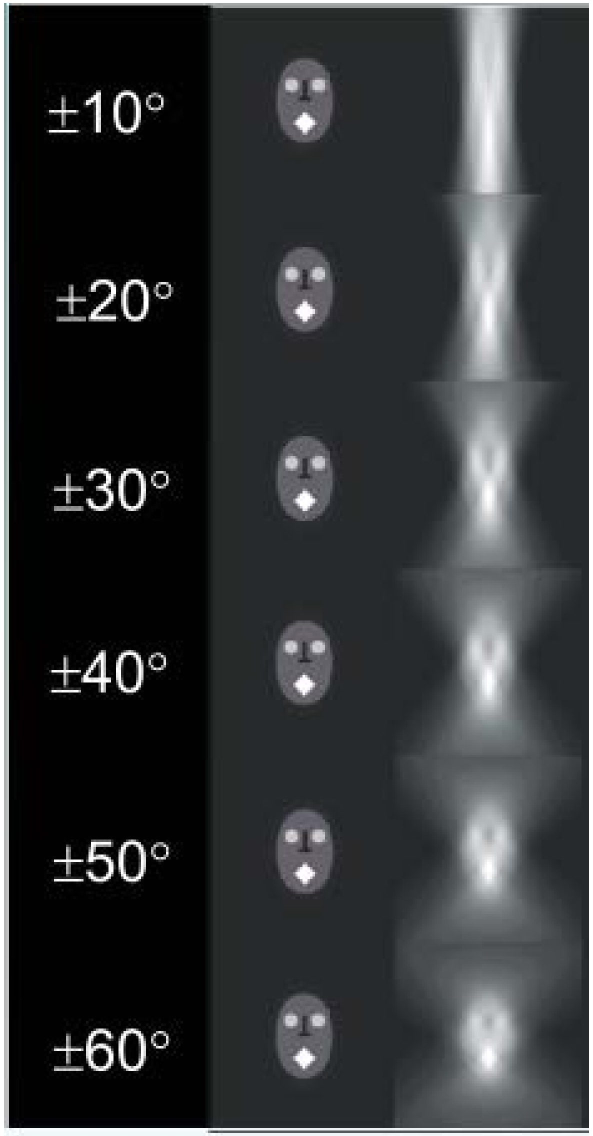
Appl. Phys. Lett. ***96***, 223108 (2010).

Missing Information In Tilt Series

Tomographic Tilt Series In Fourier Space



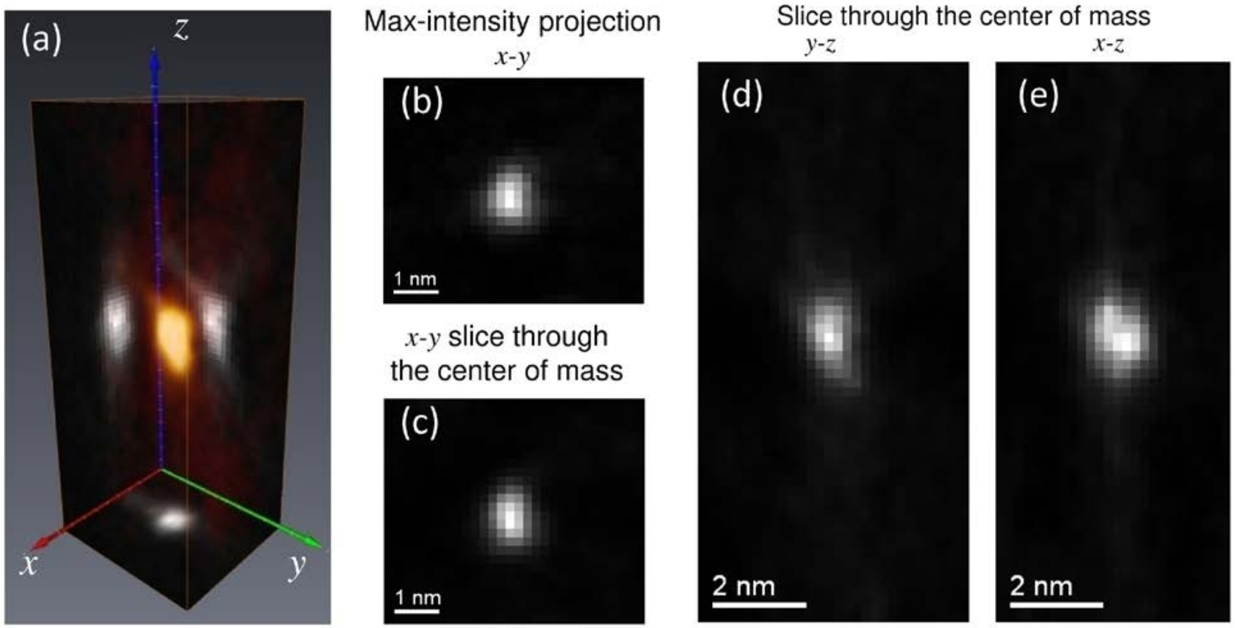
Effect of Tilt Range



Finite Objects Elongate!

Midgley & Weyland, *Ultramicroscopy* **96** (2003), 413.

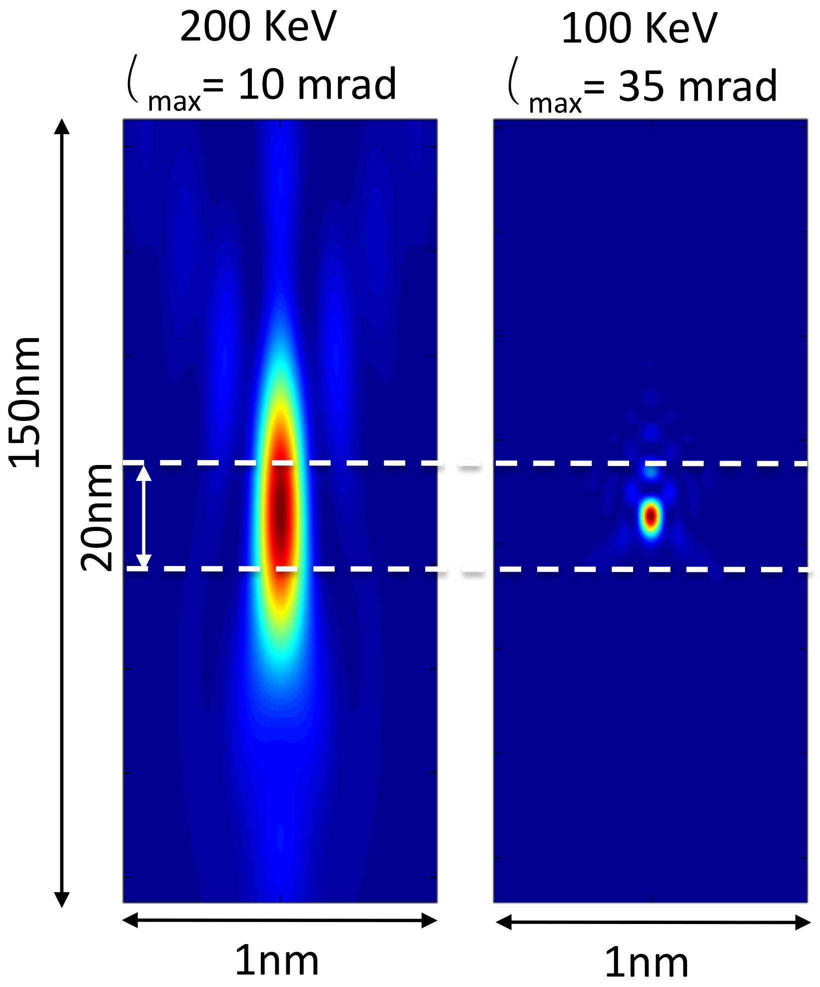
Tomography Resolution



Estimated Resolution Volume $0.5\pm0.1 \times 0.5\pm0.1 \times 0.7\pm0.2\ \mathrm{nm}^3$

Can resolution in 3D improve with aberration correction?

Depth of Field of Aberration Corrected and Uncorrected STEMs



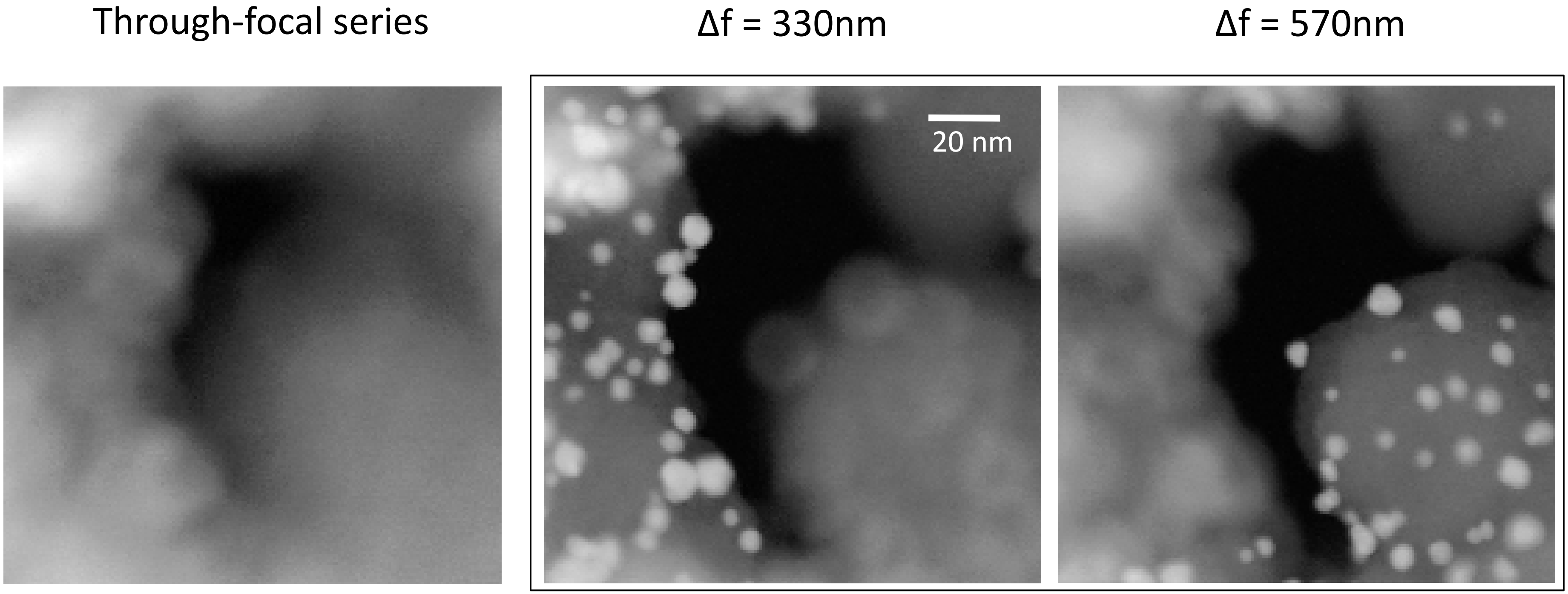
Model	Voltage (KeV)	Aperture (mrad)	Depth of Field (nm)
Uncorrected VG501	100	10	63
Uncorrected Tecnai F20	200	10	43
Corrected Titan	300	23	6.5
Corrected Nion Super STEM	100	35	5.1

Intaraprasonk, V et al, *Ultramicrosopy* **108 (2008)**

Nellist P D, et al, *Microsc. Microanal.* ***14: 82–88. (2008)***

Xin, HL, Muller, DA, *Journal of electron microscopy*: ***1-9 (2009)***

Cannot simultaneously image all particles in-focus



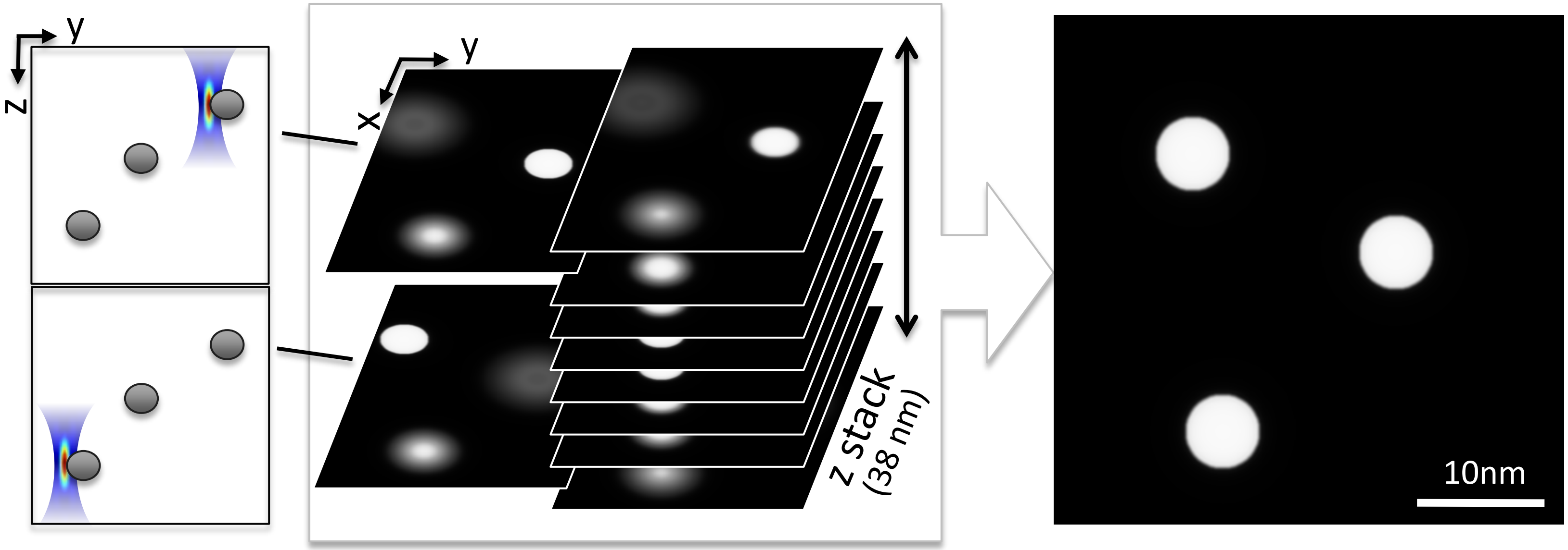
PtCo nano-particles on carbon black support

Traditional tomography fails for extended objects!

R. Hovden et al, Microscopy and Microanalysis **17** (2011)

NION UltraStem, 100KeV, 33 mrad

Extended Depth of Field

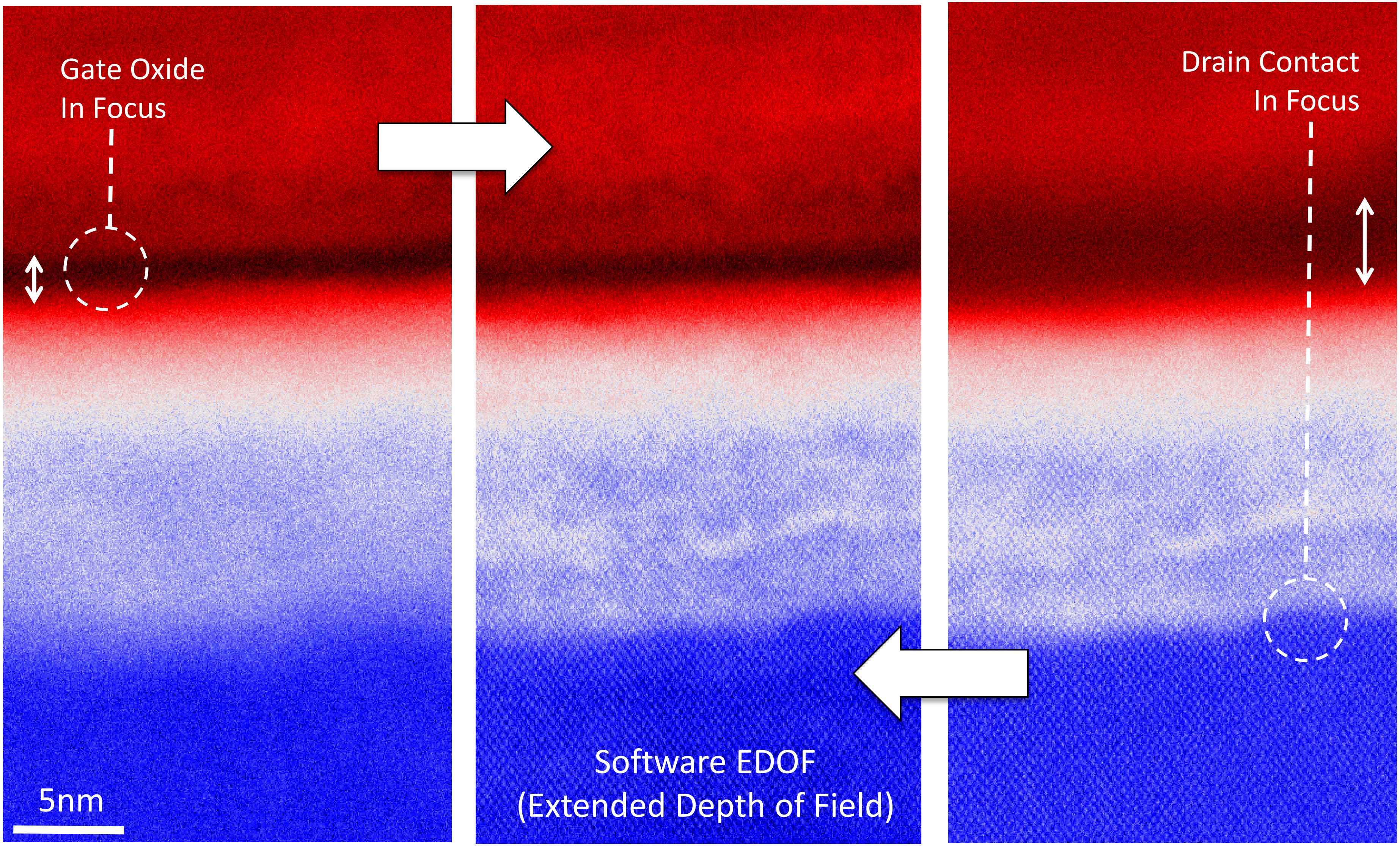


EDOF uses wavelet coefficients to identify the in-focus information in a through focal image stack—then merging to create an image with all regions in-focus

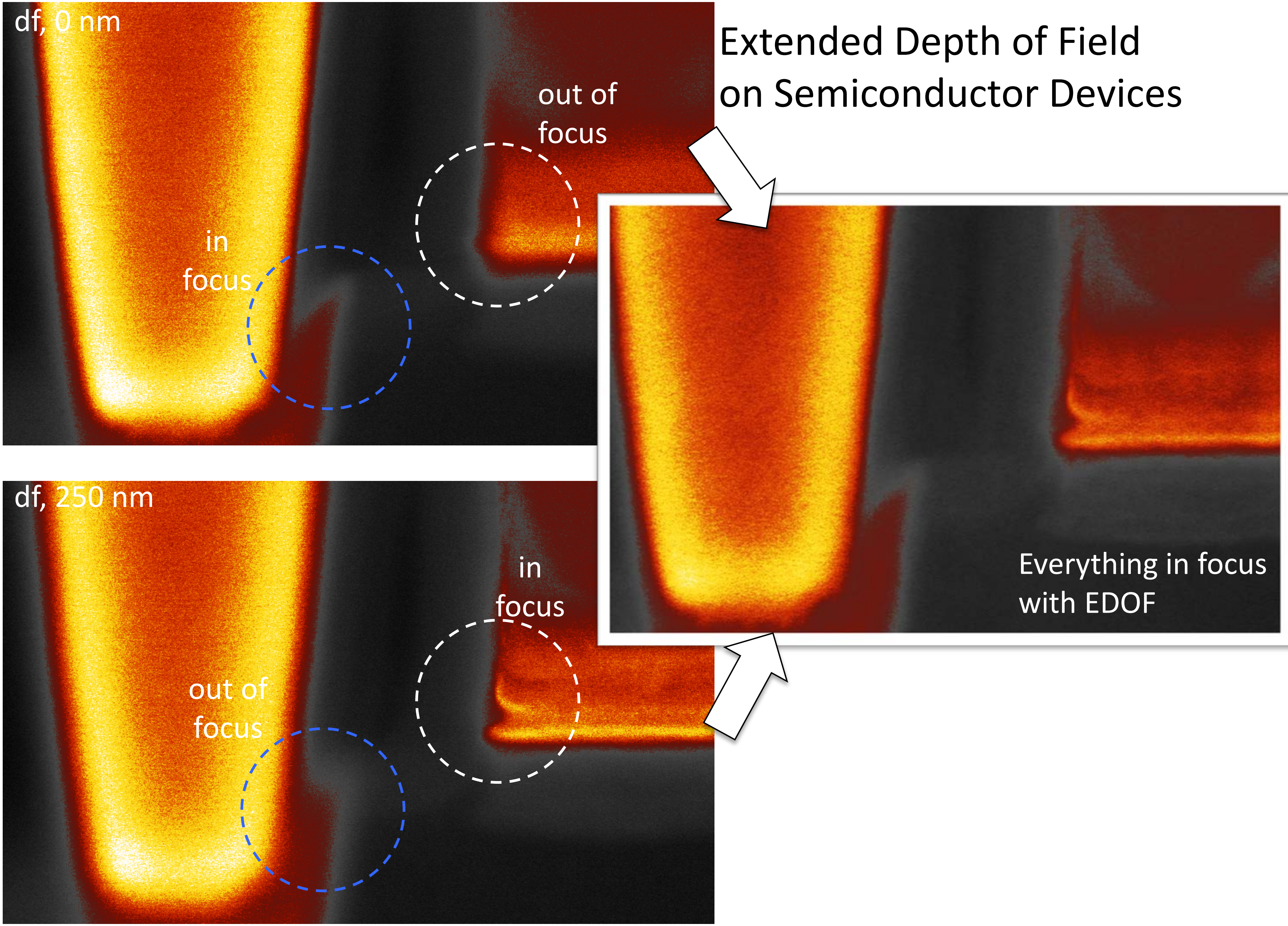
R. Hovden, et. al. *Microscopy and Microanalysis*, **17**: 75-80 (2011)

NION UltraStem, 100KeV, 33 mrad

Aberration Correction:
Small depth of field is bad for tomography



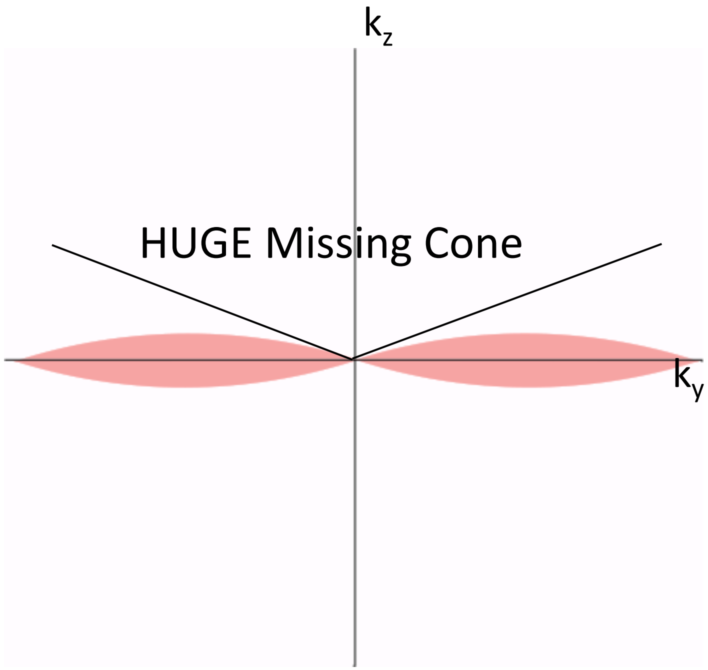
Extended Depth of Field on Semiconductor Devices



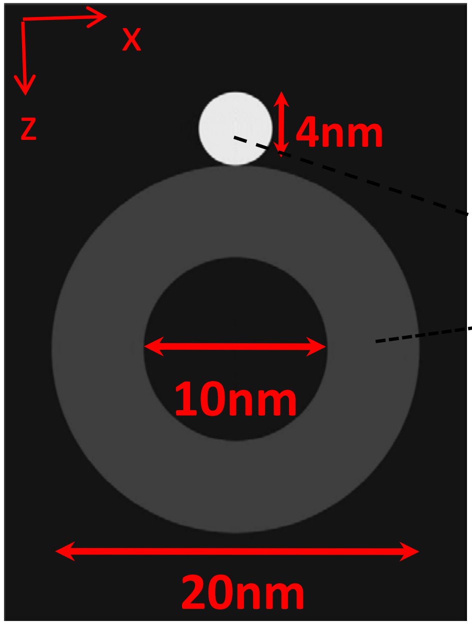
Everything in focus with EDOF

Through Focal STEM

Information in Fourier Space

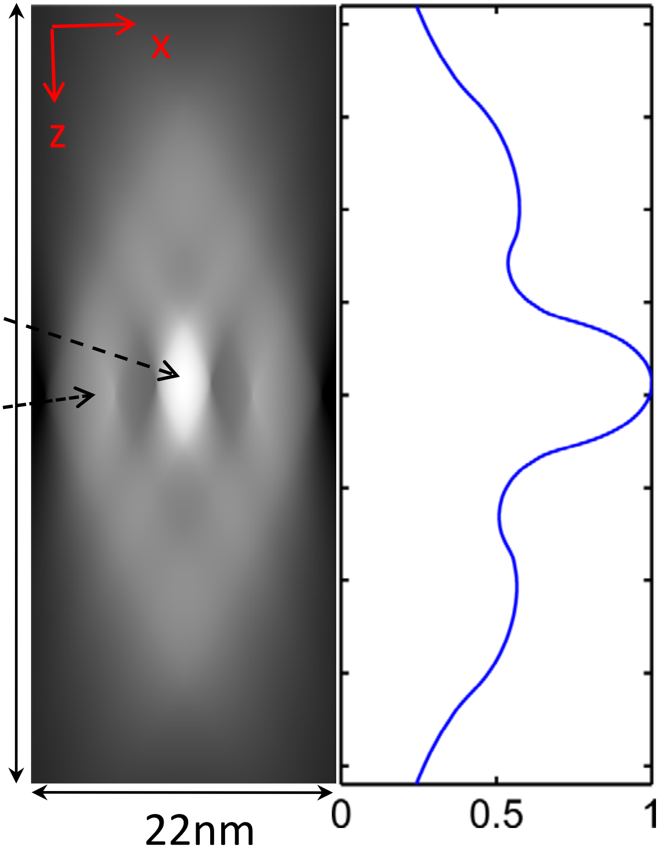


Original Objects



Through Focal Recon

Z axis compressed by 15 fold

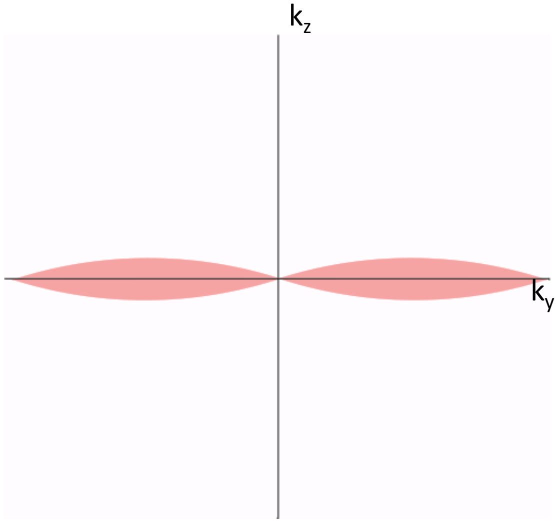


Xin, Intarapasonk, Muller, APL **92** (2008)

STEM depth sectioning fails for 3D imaging

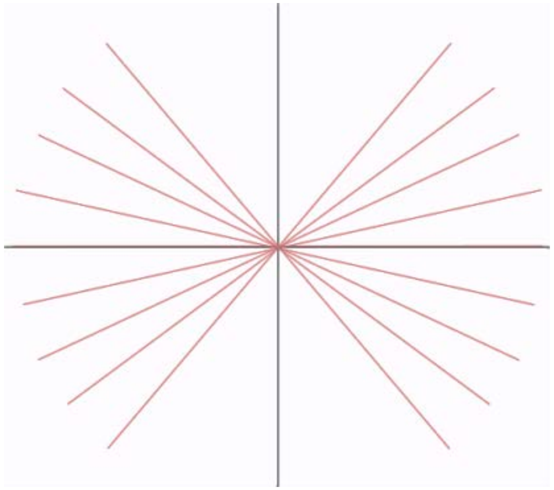
Comparison of Reconstruction Methods and how they fill in Fourier Space

Focal Series



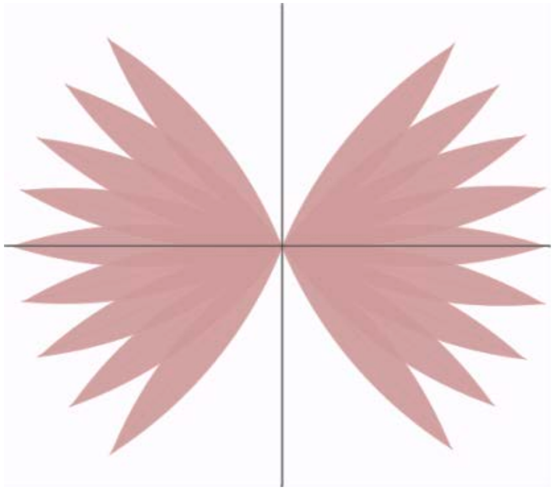
>Missing most information needed for 3D imaging

Tilt Series



>Assumes perfect projections
>Fails for large objects and high-resolution
>Crowther Criterion LImited

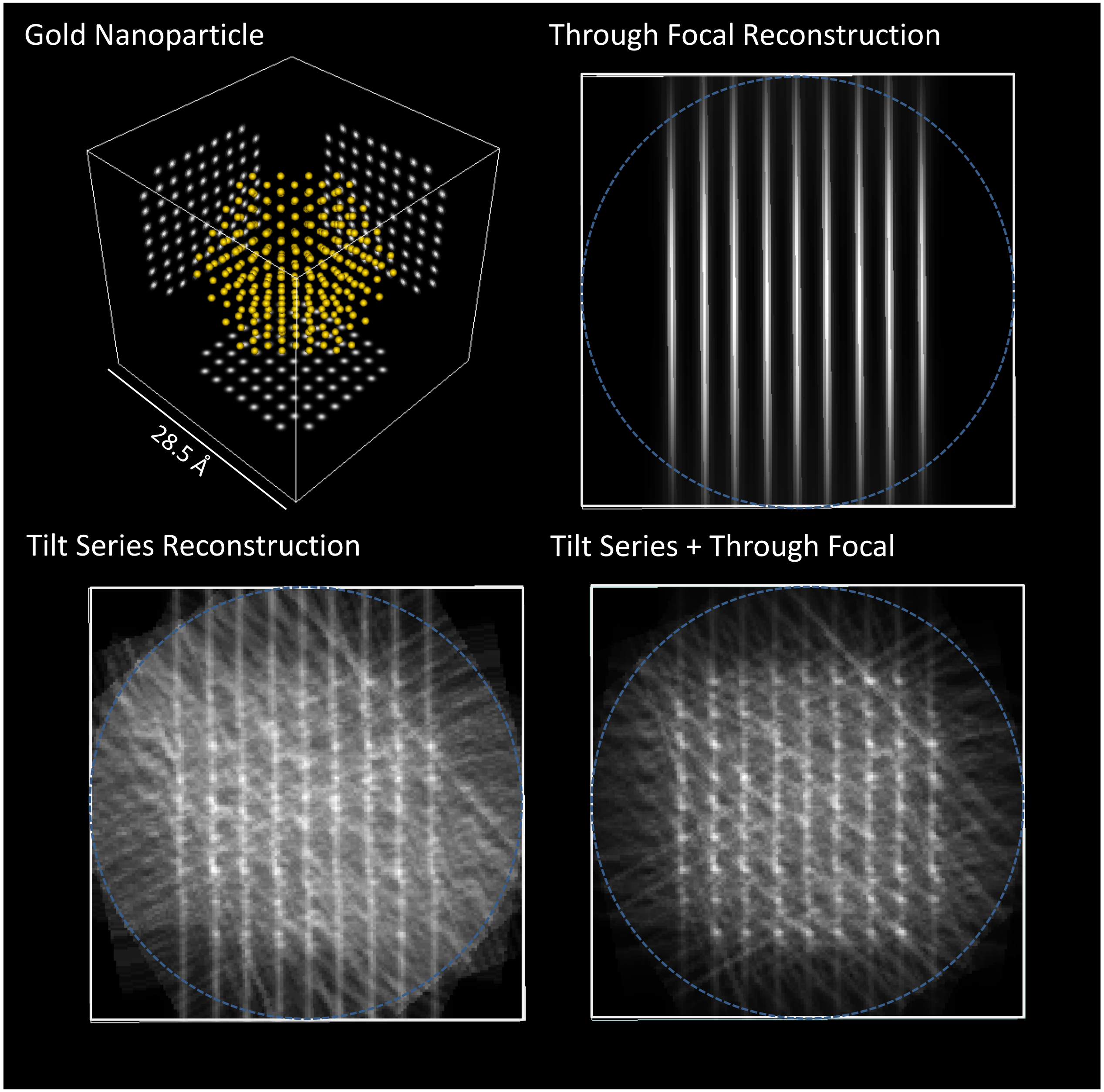
Focal & Tilt Series



>High-resolution over large objects
>Requires fewer tilts
>Provides continuum of information
>Breaks the Crowther Criterion

Depth-Sectioning can fill Fourier Space between tilts, reducing the number of tilts needed

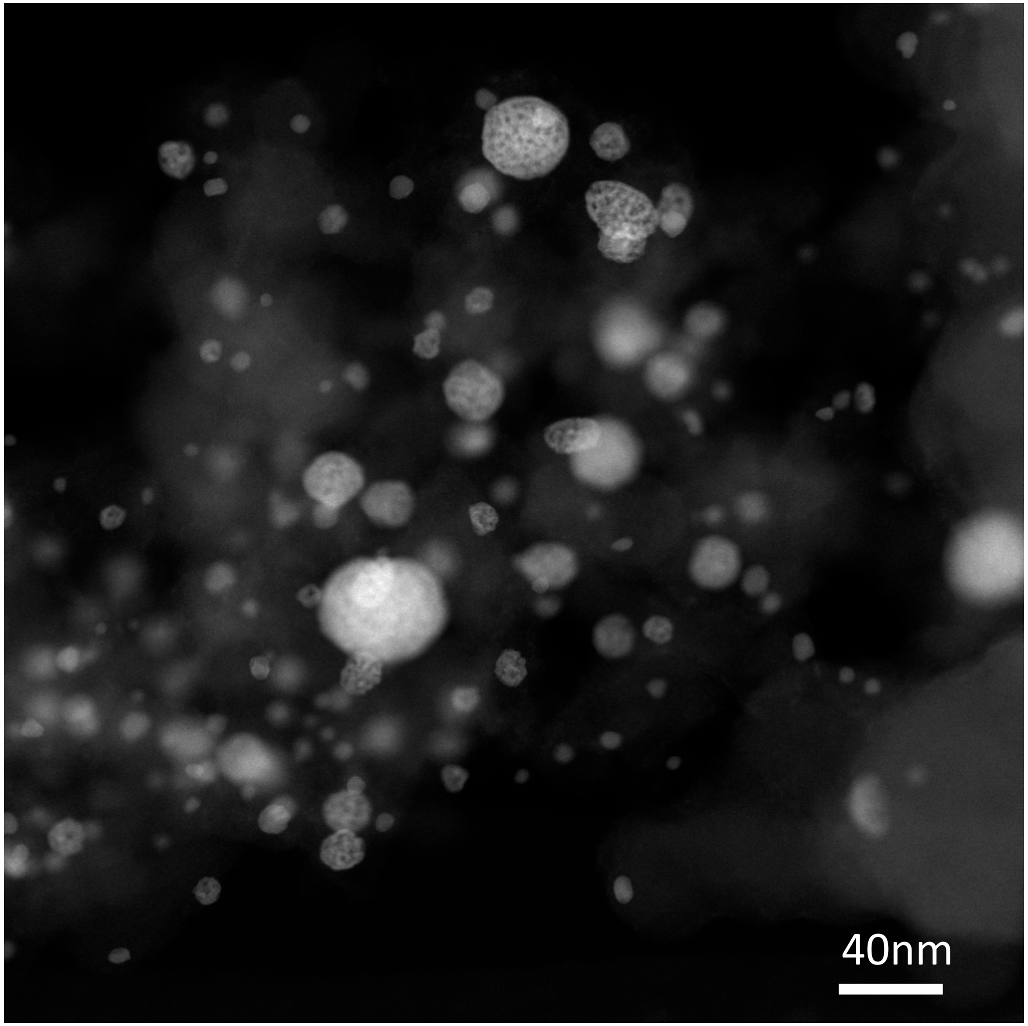
Comparison of 3D Reconstructions

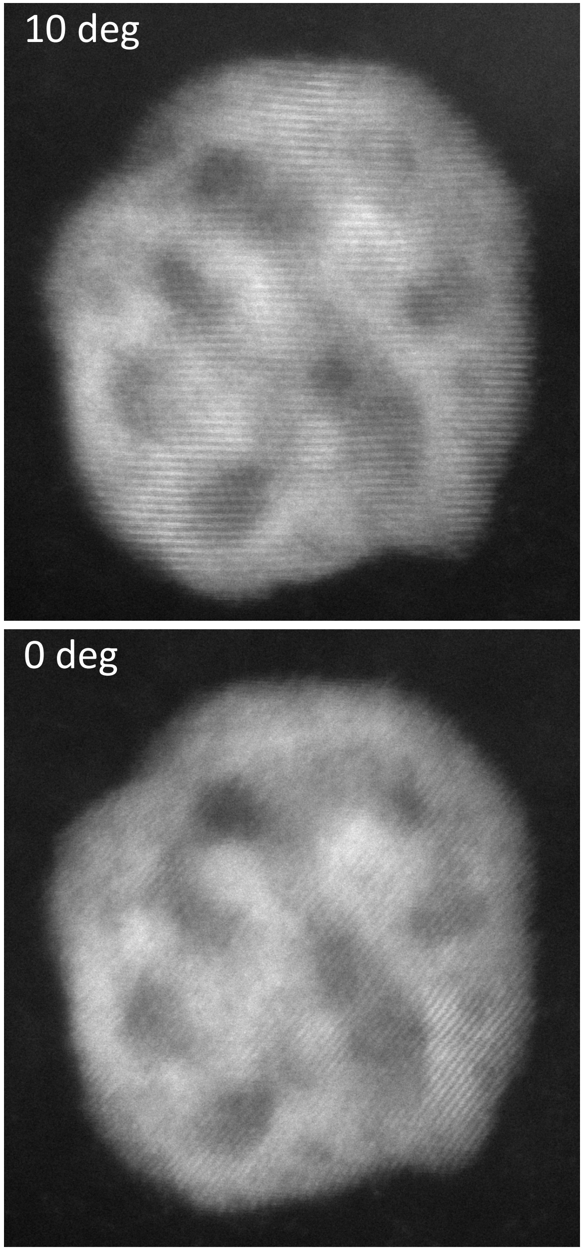


NION UltraStem, 100KeV, 25 mrad; ±70°, 20° tilts

Porous Pt-Cu Nanoparticles

PtCu nanoparticles treated in Nitric Acid acquire porous structure.



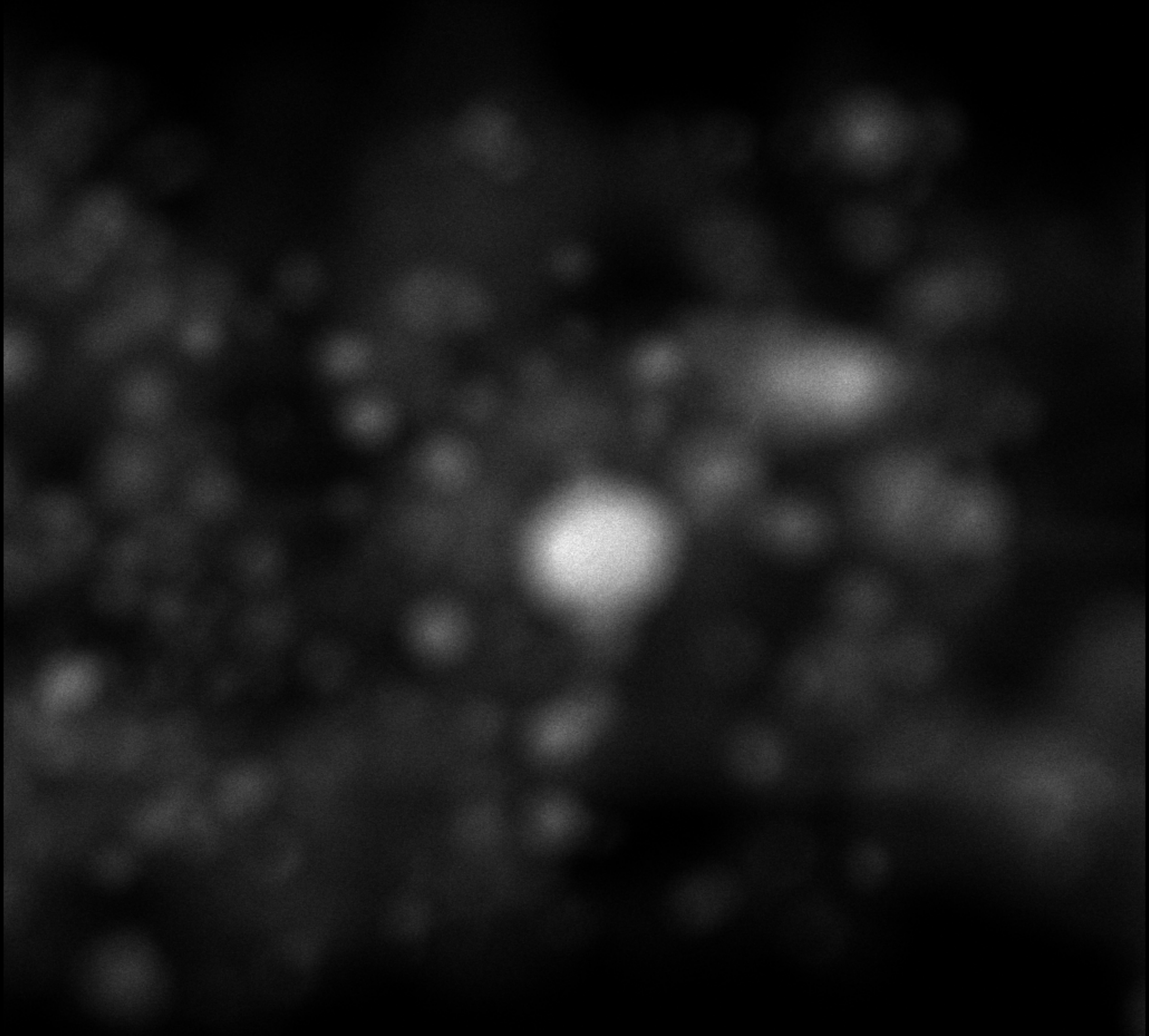


Traditional Tilt Series

fov
391nm

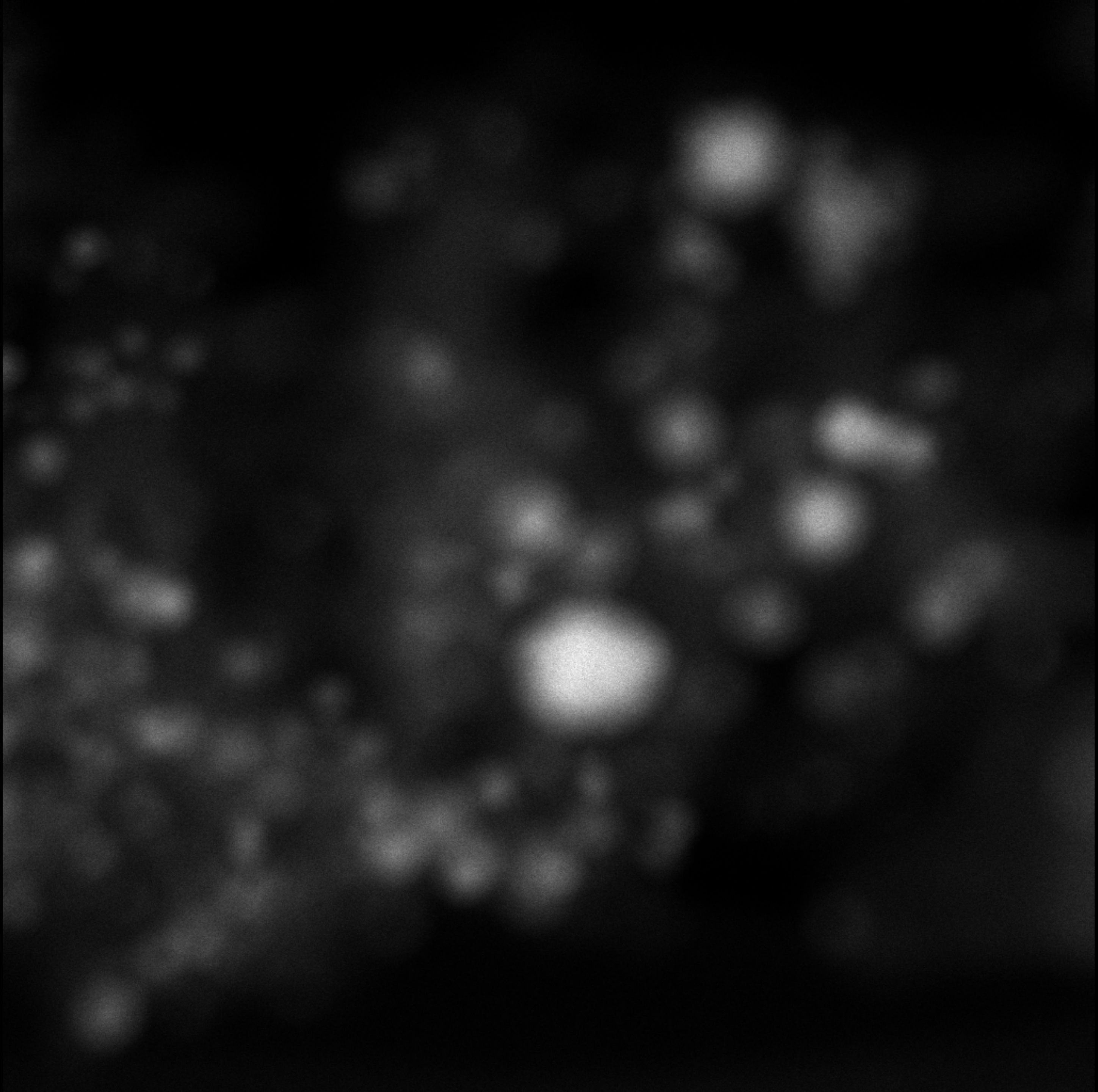
Through-Focal Tilt Series

+68°



fov
391nm

Through-Focal Tilt Series

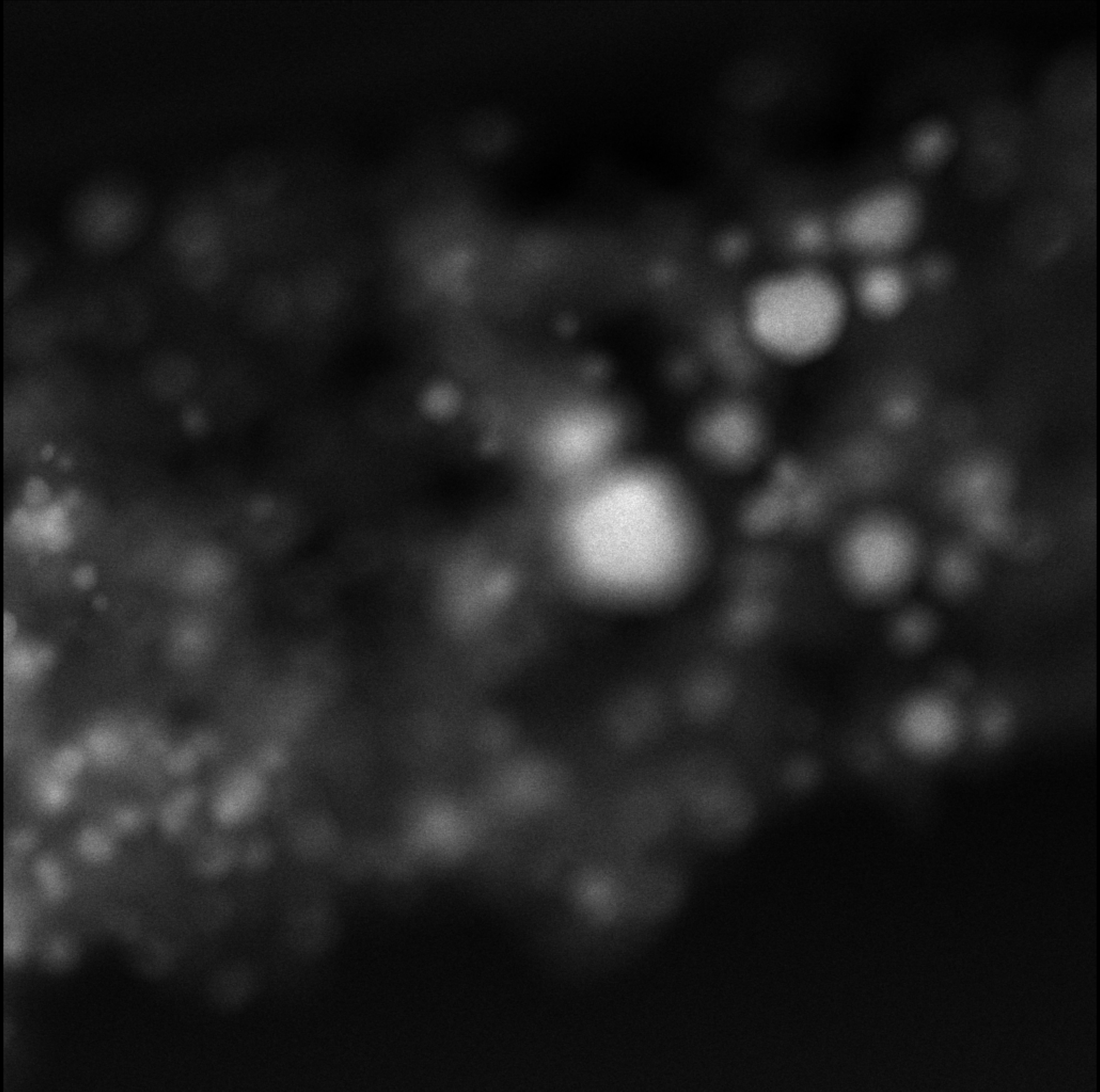


-1°

fov
391nm

Through-Focal Tilt Series

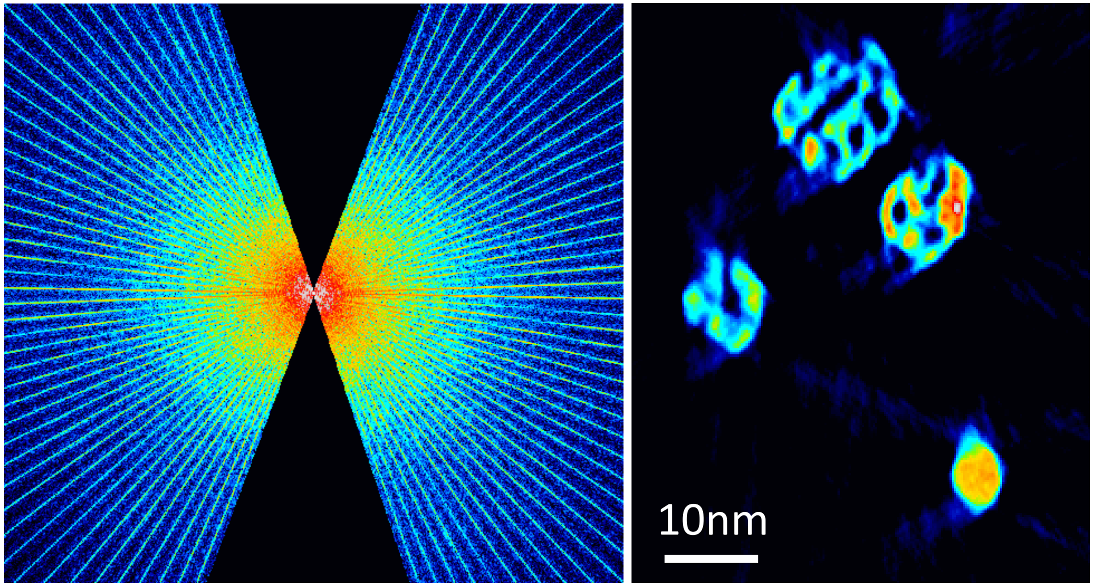
-70°



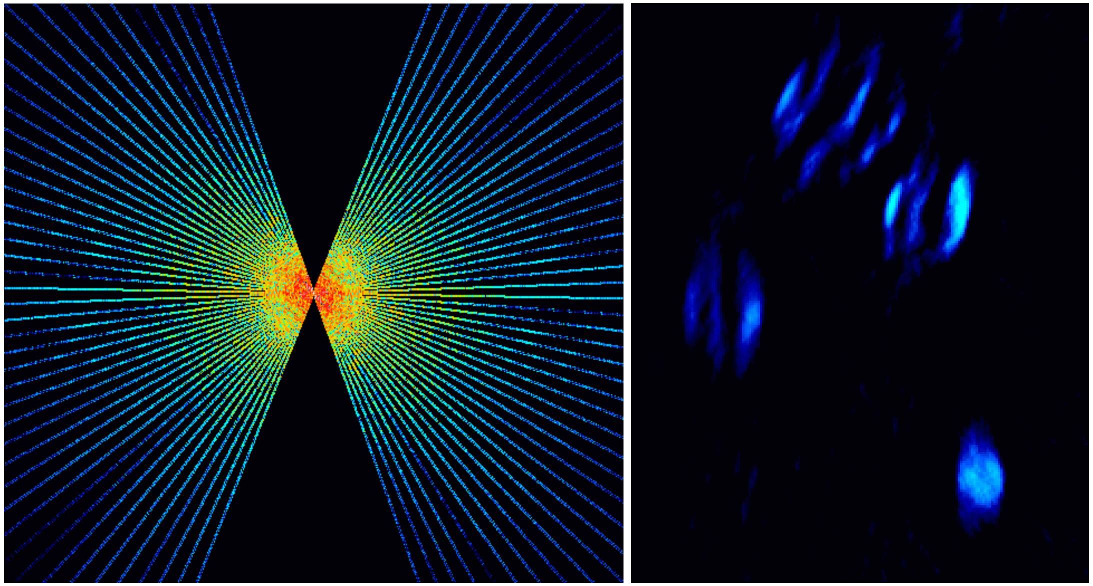
fov
391nm

Tomography of Porous PtCu Nanocatalysts

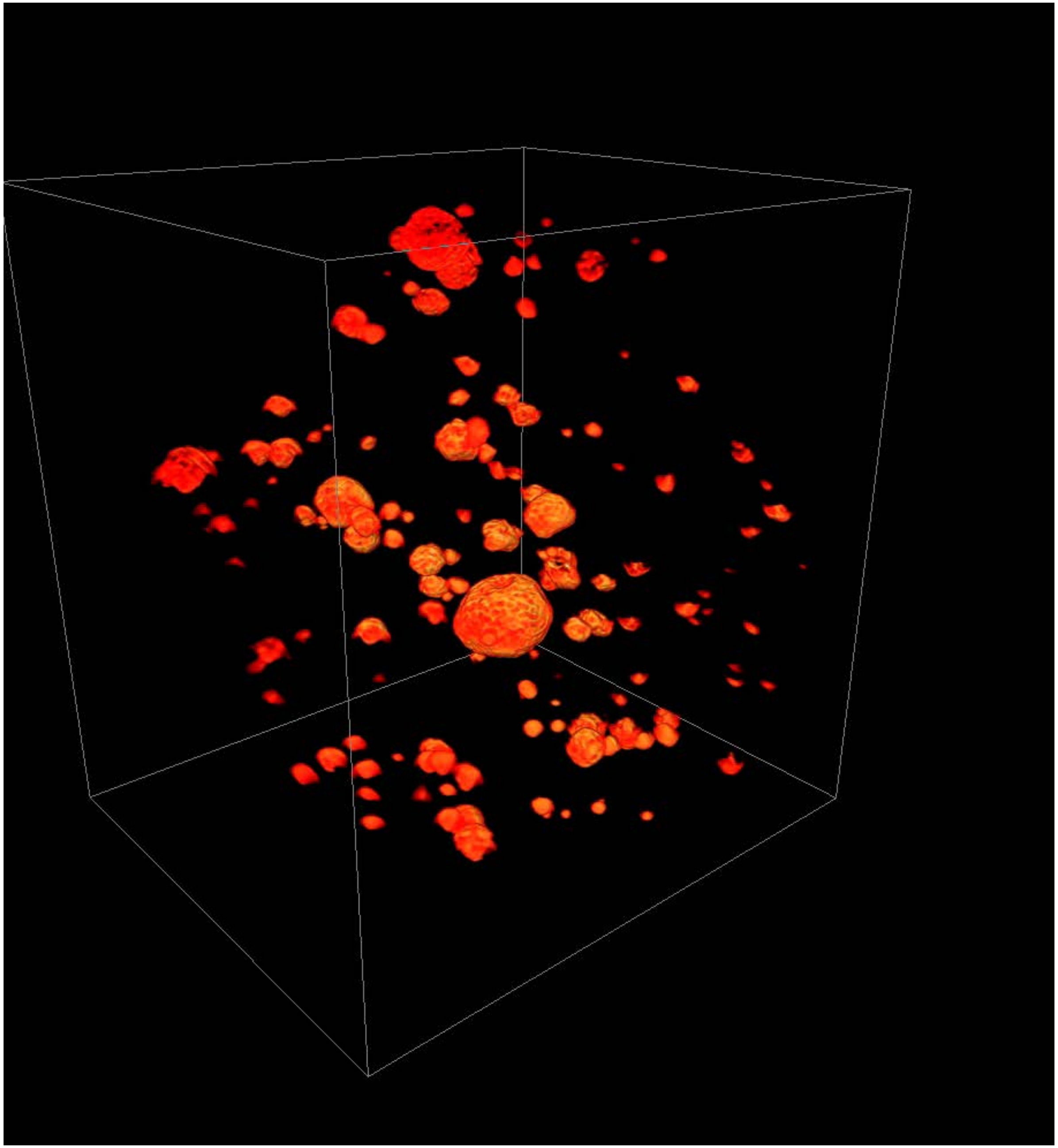
Through-Focal Tomography



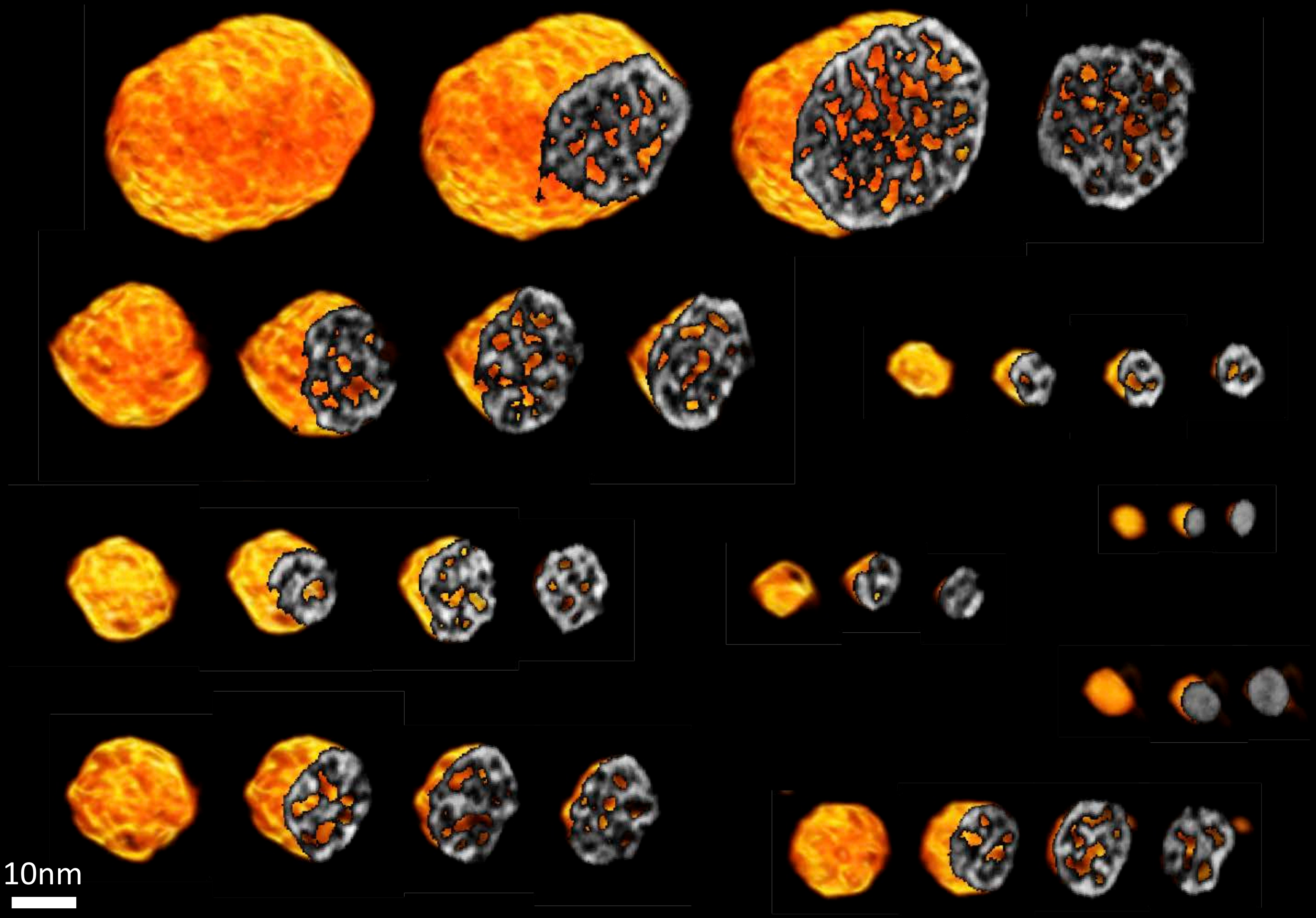
Traditional Tomography



Tomography of Porous PtCu Nanocatalysts

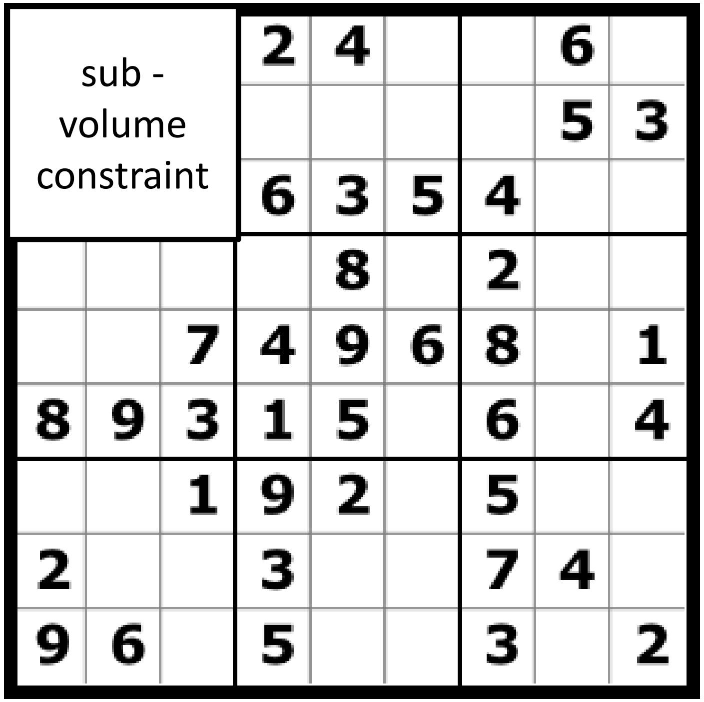


Pore Structure of Acid Treated PtCu Nanocatalysts



The SUDOKU Analogy

SUDOKU

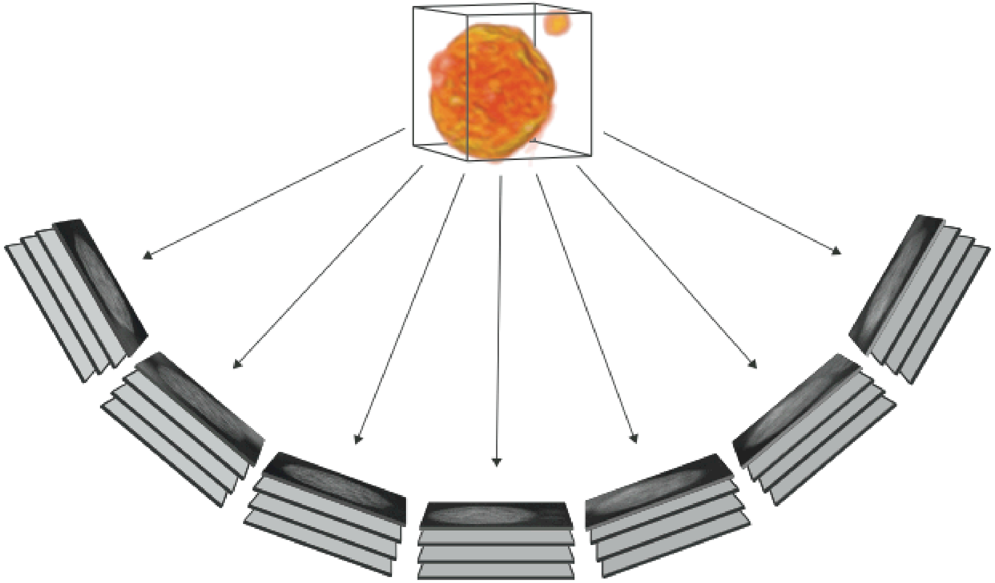


sub - volume constraint			2	4			6	
							5	3
			6	3	5	4		
				8		2		
		7	4	9	6	8		1
8	9	3	1	5		6		4
		1	9	2		5		
2			3			7	4	
9	6		5			3		2

Projection Contraints

Projection Contraints

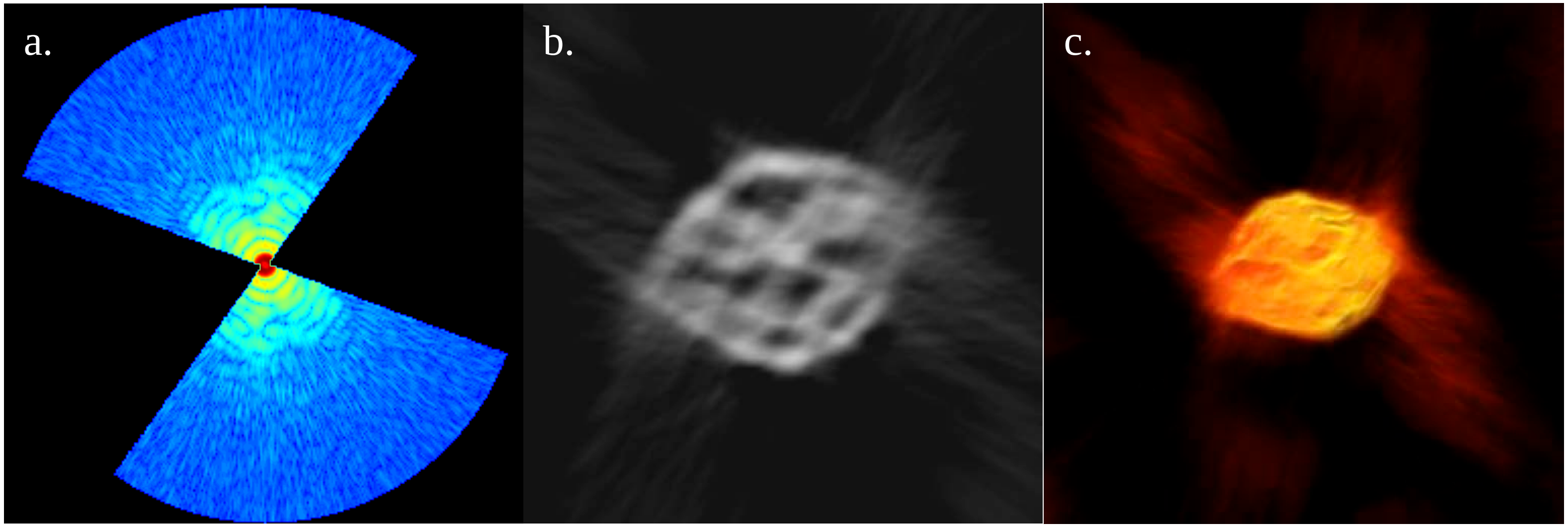
Through-Focal Tomography



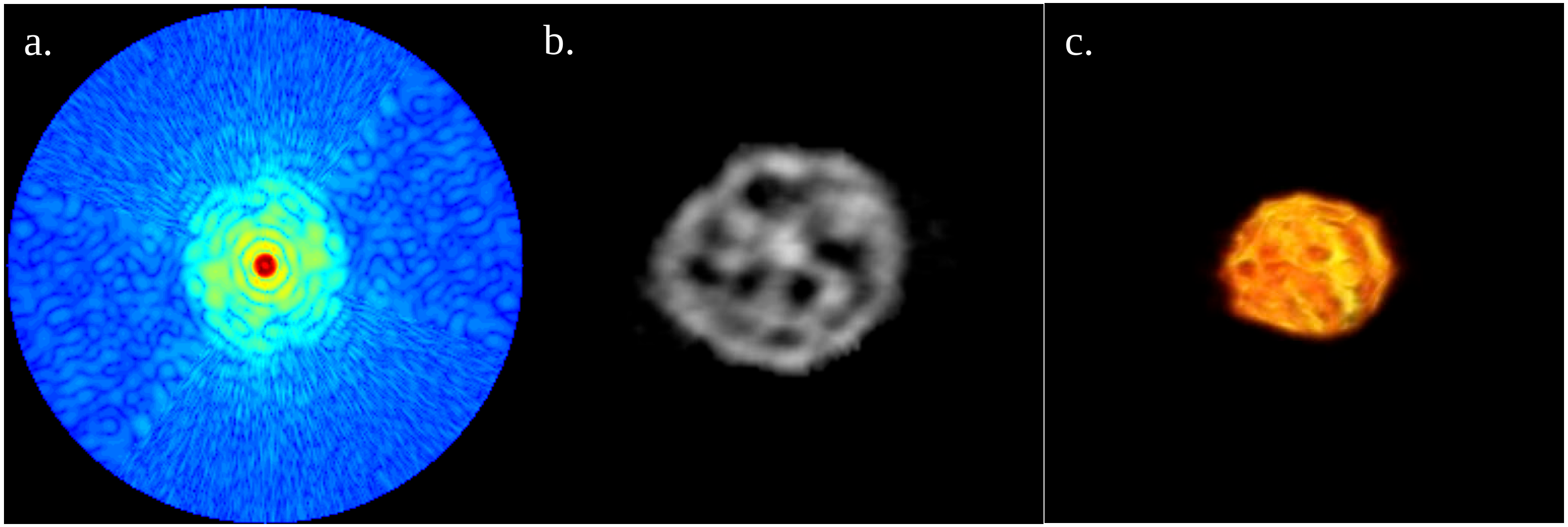
Through-Focal Methods Provides Sub-Volume Constraints in Addition To Projection

Iterative Constraints and Difference Map Reconstructions

Direct Fourier Recon of Large Missing Wedge Object



Iterative Constraint + Diff Man Recon of Large Missing Wedge Object



Summary & Acknowledgements

Collaborators

Peter Ercius, Yi Jiang, Deli Wang,
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Veit Elser, David A. Muller

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