Imaging at the Nanoscale - Electron and X-Ray Beams

David Muller

Judy Cha, Peter Ercius, Lena Fitting, Jerome Hyun, Aycan Yurtsever,

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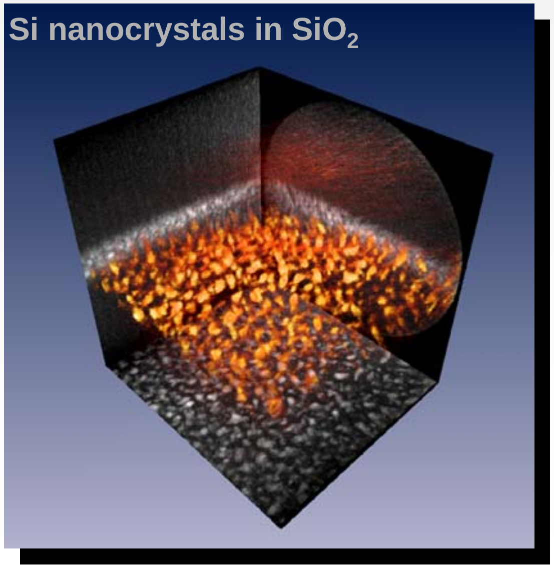
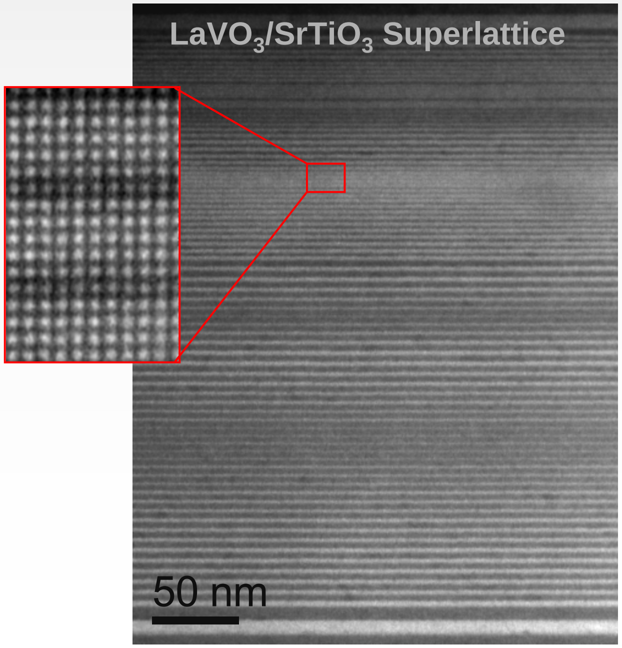
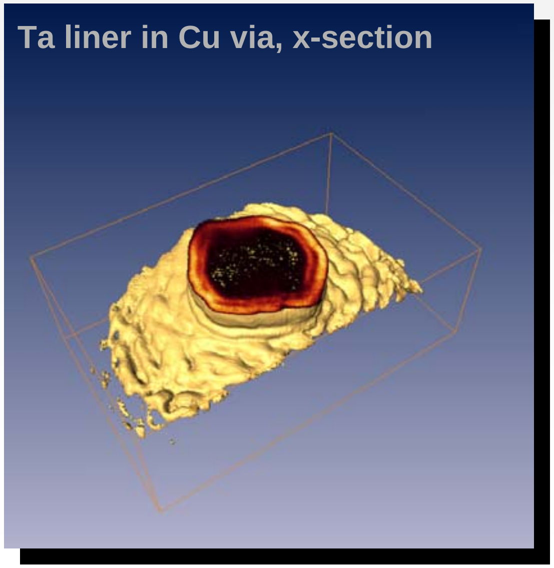
Nanoparticles



New Materials

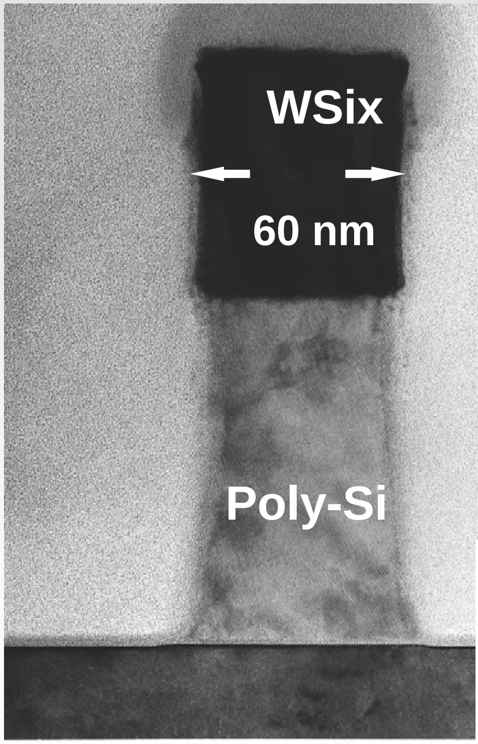


Integrated Circuits

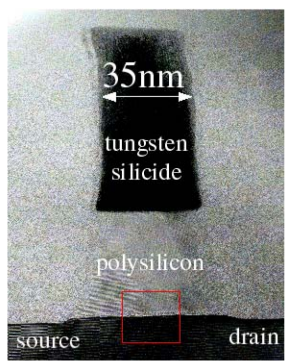


Similar challenges (and tools) for semiconductor and life sciences

Commercial CMOS

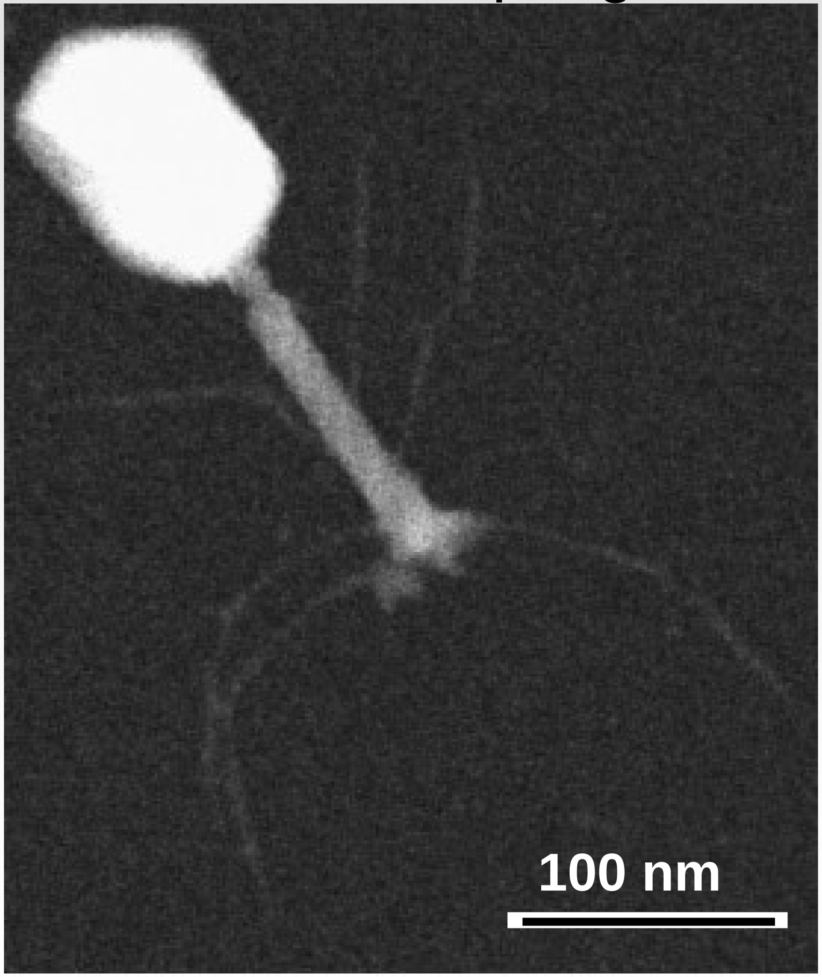


2003



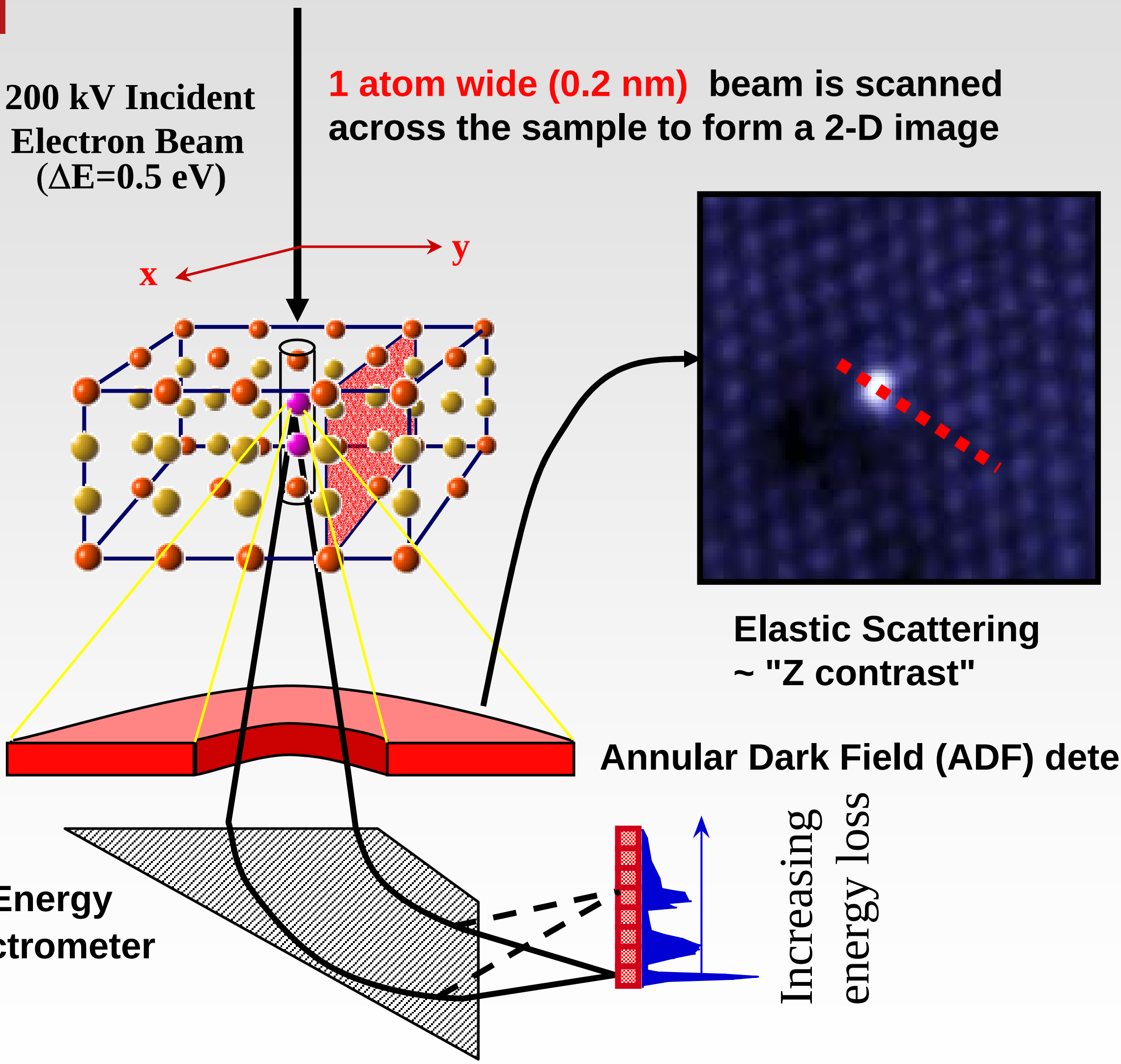
2007

T2 Bacteriophage



STEM (J. Wall, BNL)

Scanning Transmission Electron Microscopy



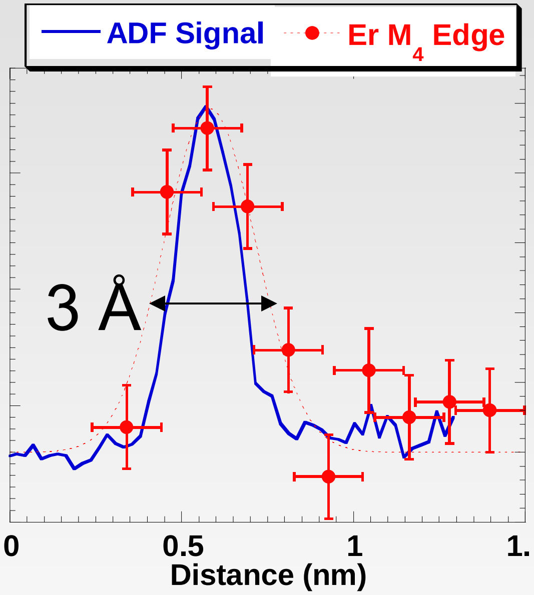
200 kV Incident Electron Beam (ΔE=0.5 eV)

1 atom wide (0.2 nm) **beam is scanned across the sample to form a 2-D image**

Elastic Scattering ~ "Z contrast"

Annular Dark Field (ADF) detector

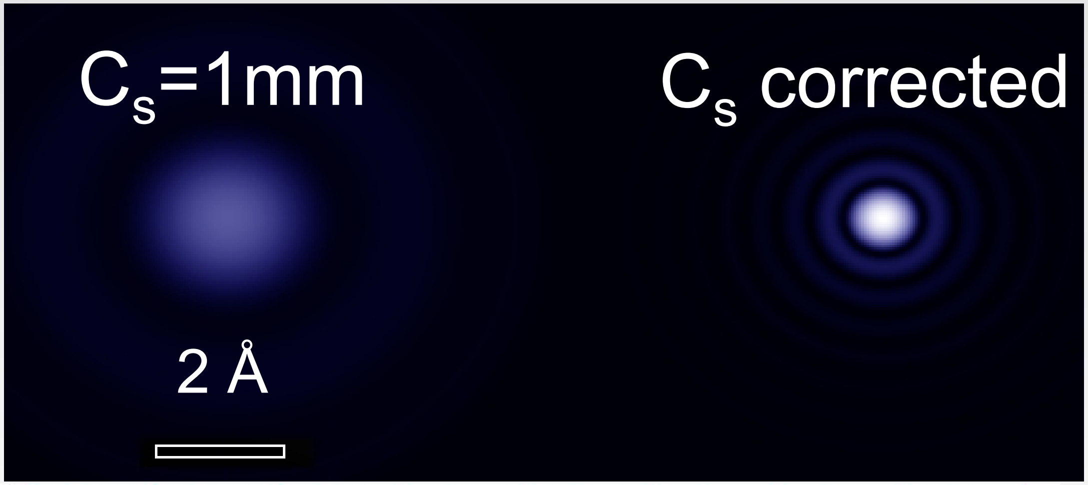
Electron Energy Loss Spectrometer

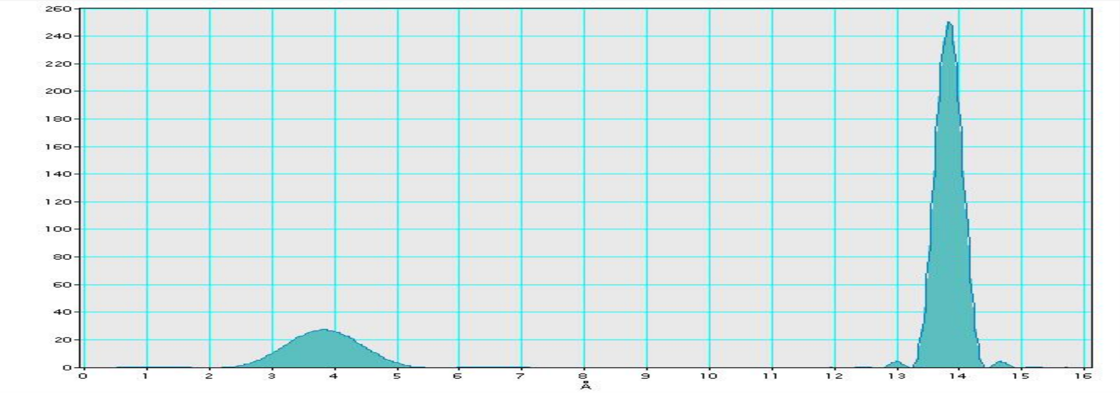


Single atom Sensitivity: P. Voyles, D. Muller, J. Grazul, P. Citrin, H. Gossmann, *Nature* **416** 826 (2002)
U. Kaiser, D. Muller, J. Grazul, M. Kawasaki, *Nature Materials*, **1** 102 (2002)

Why is the Probe so Large?

- Probe diameter is ~ 2 Å
- Electron Wavelength at 200 kV is 0.0251 Å
- Non-ideal lenses ⇨ large aberrations ⇨ tiny numerical apertures

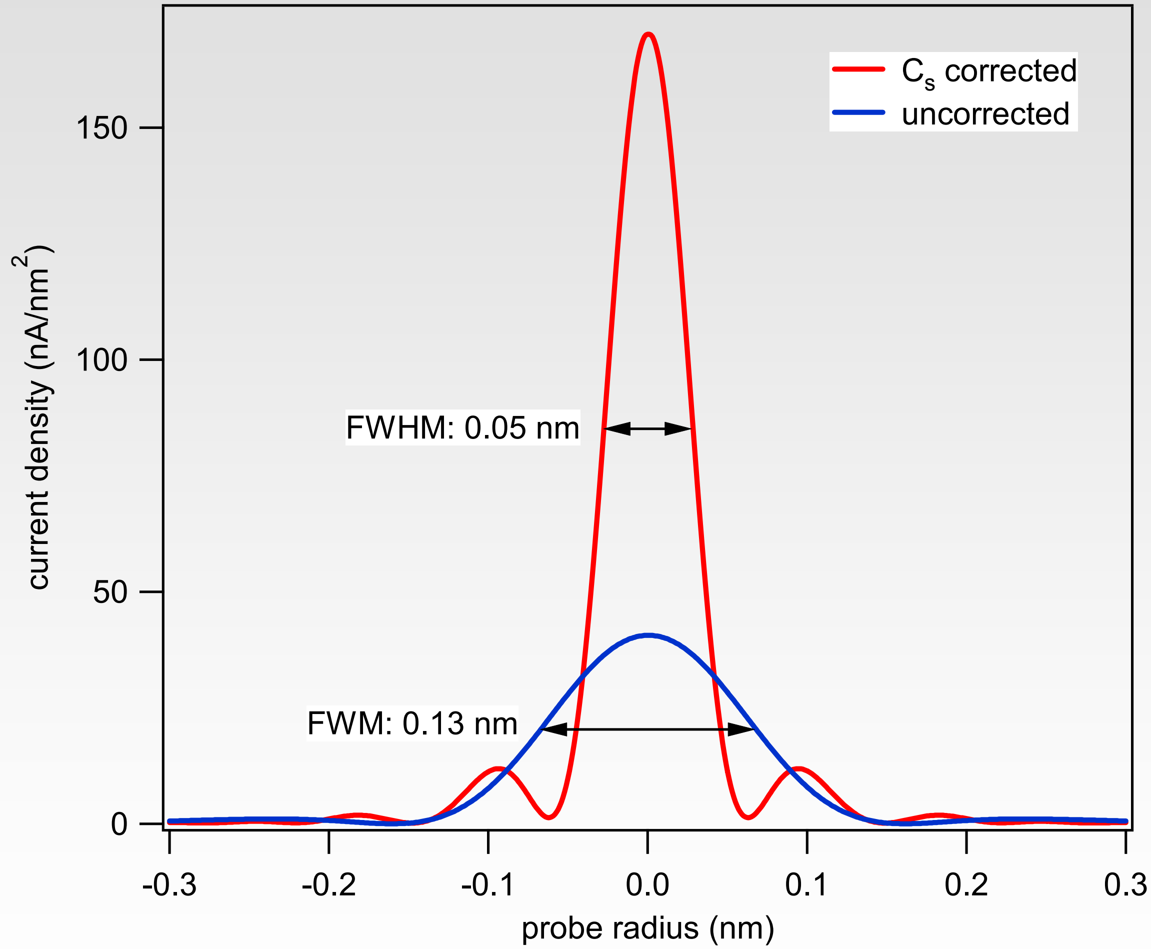




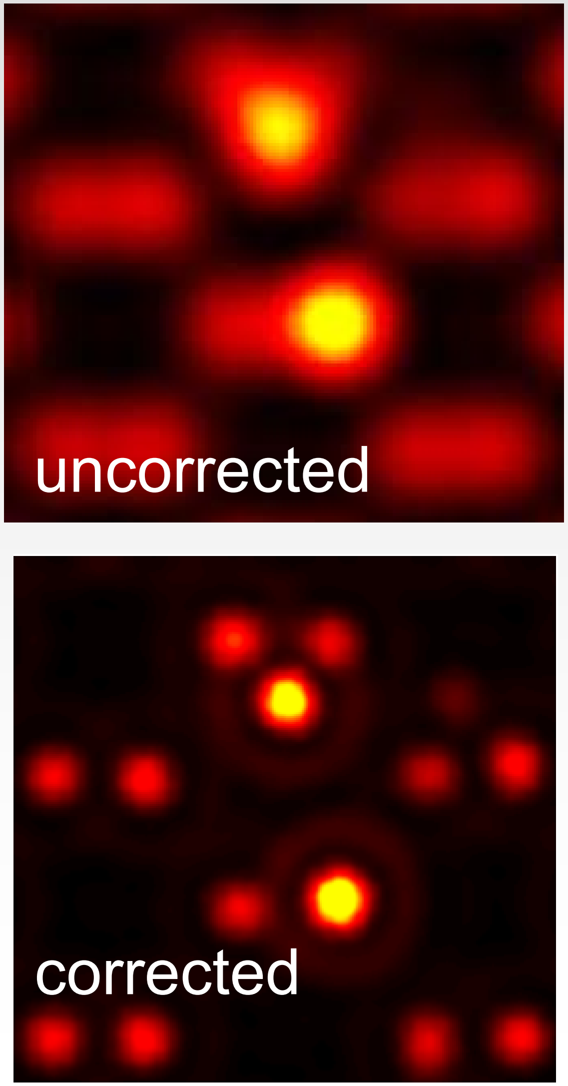
Corrector Benefits: Increased current, resolution, contrast

[see P. E. Batson *et al.*, *Nature* **418**, 617 (2002)]

C_s-Corrected STEM

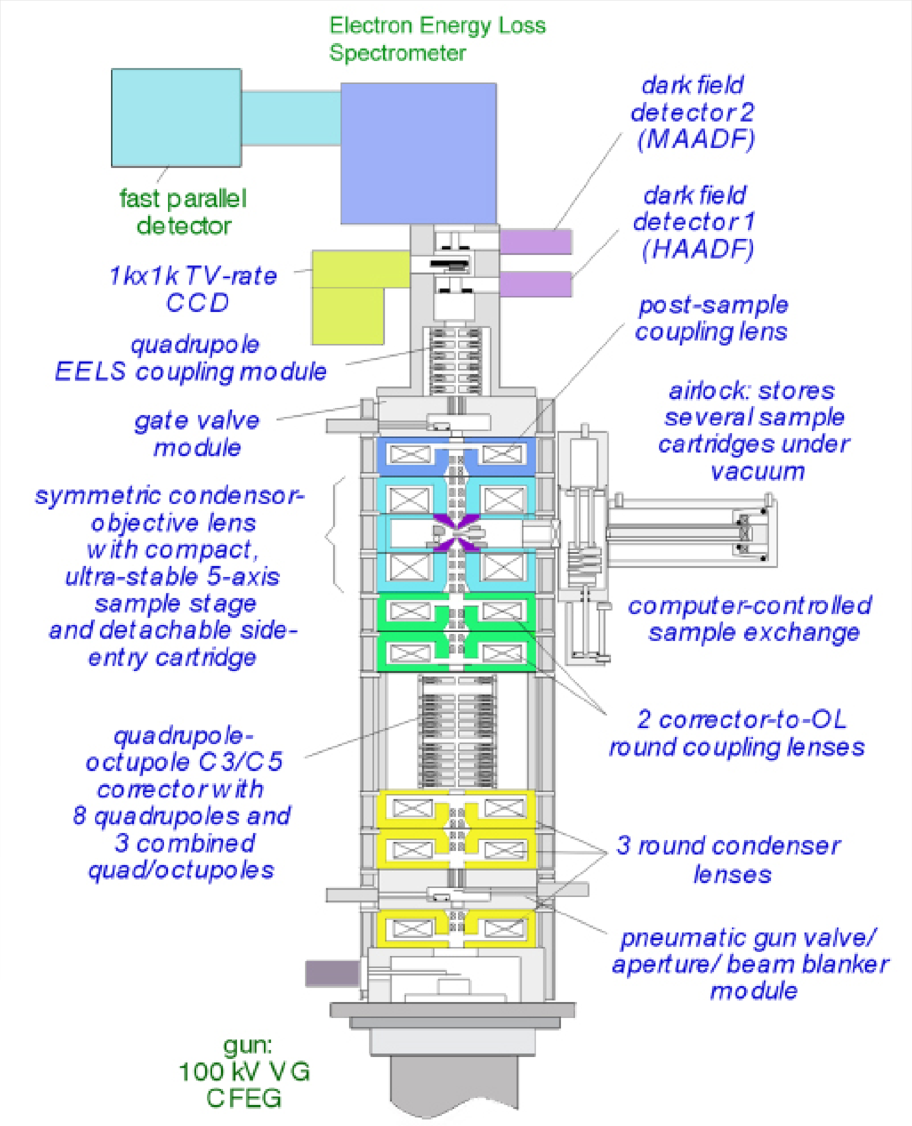


DP2 paired-Sb defect



Corrector Benefits: Increased resolution, contrast or current.

Aberration-Corrected STEM



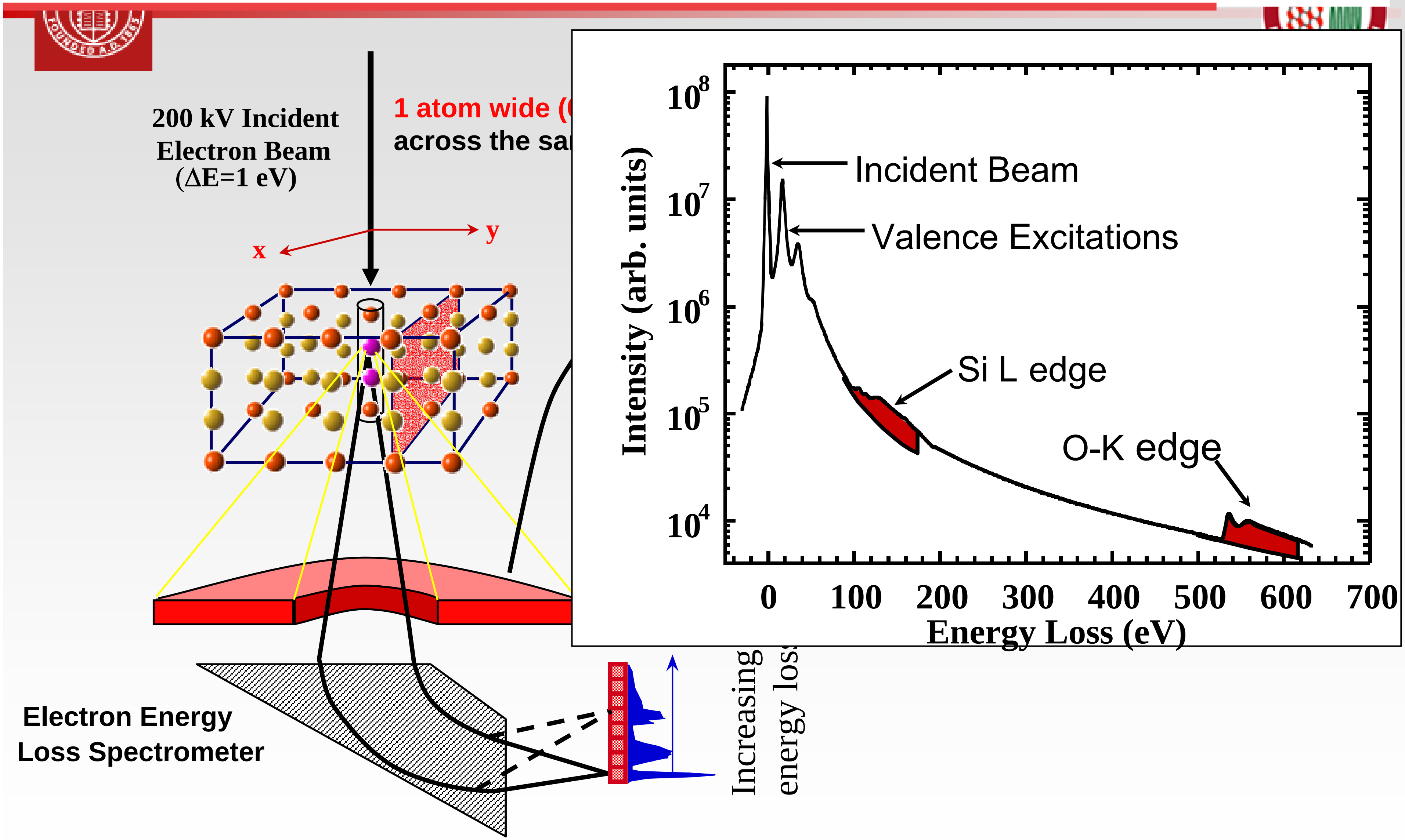
NION SuperSTEM with PEELS

- 0.4 eV energy resolution
- **0.05 nm** spatial resolution
- 1 nm depth of focus -> 3D!
- EELS spectral maps in real time

This will be the world’s first
5th-order corrected STEM
(x 4 improvement over previous)

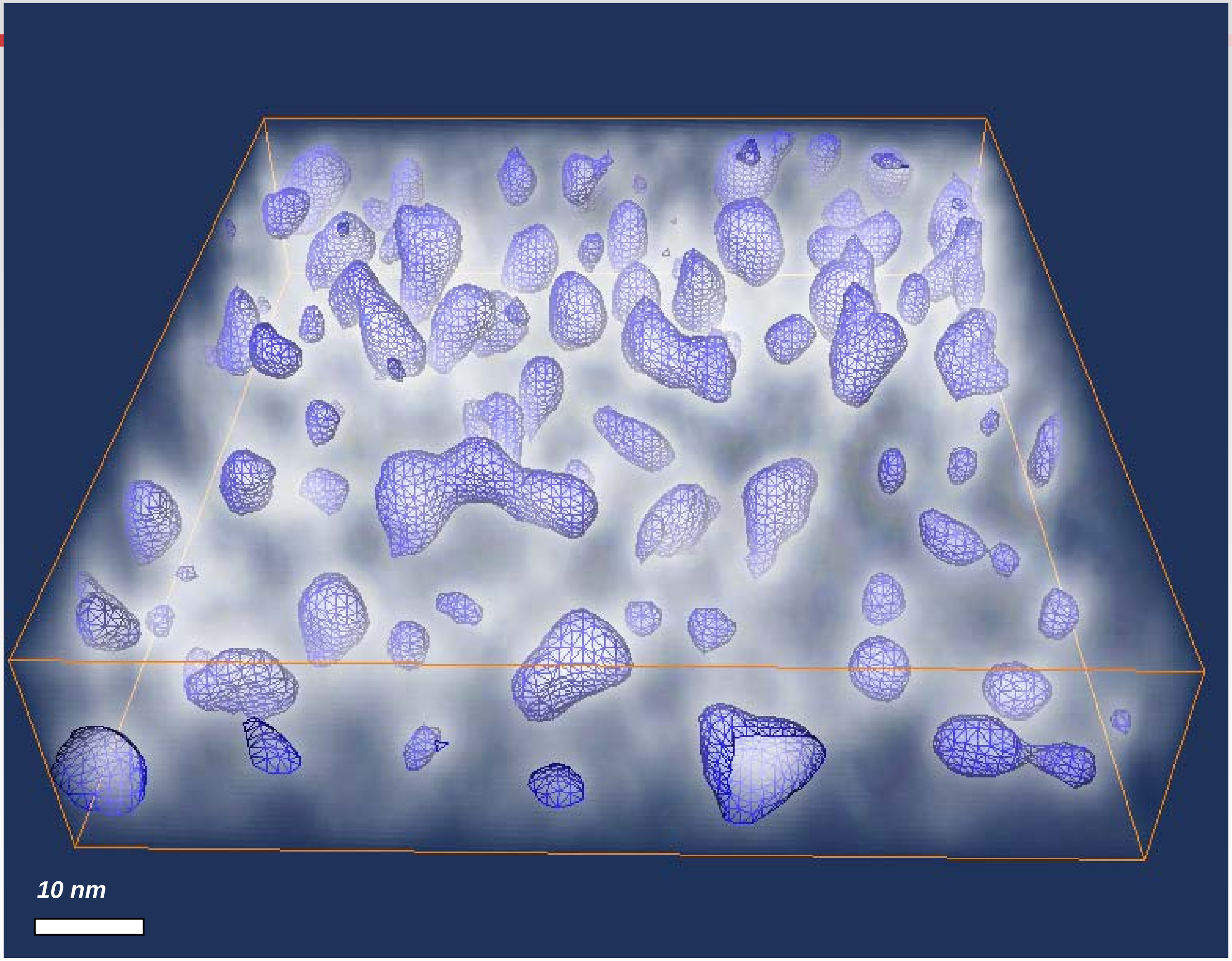
Due ~~early~~ *late* ‘06

Scanning Transmission Electron Microscopy



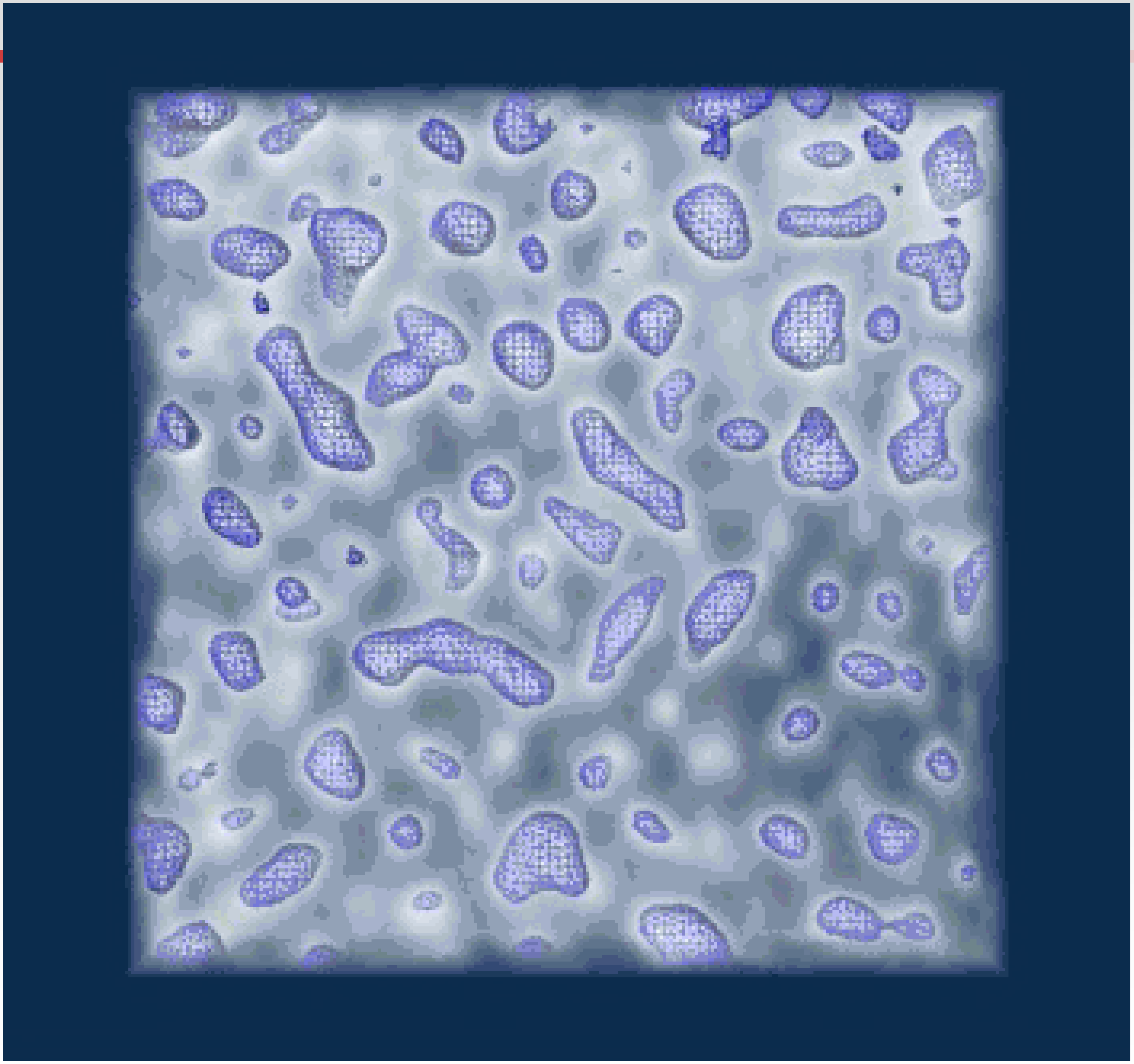
Single atom Sensitivity: P. Voyles, D. Muller, J. Grazul, P. Citrin, H. Gossmann, *Nature* **416** 826 (2002)
U. Kaiser, D. Muller, J. Grazul, M. Kawasaki, *Nature Materials*, **1** 102 (2002)

3D-Characterization of Si Nanoparticles embedded in Silicon Oxide



Tomographic reconstruction of the Silicon plasmon signal at 17eV

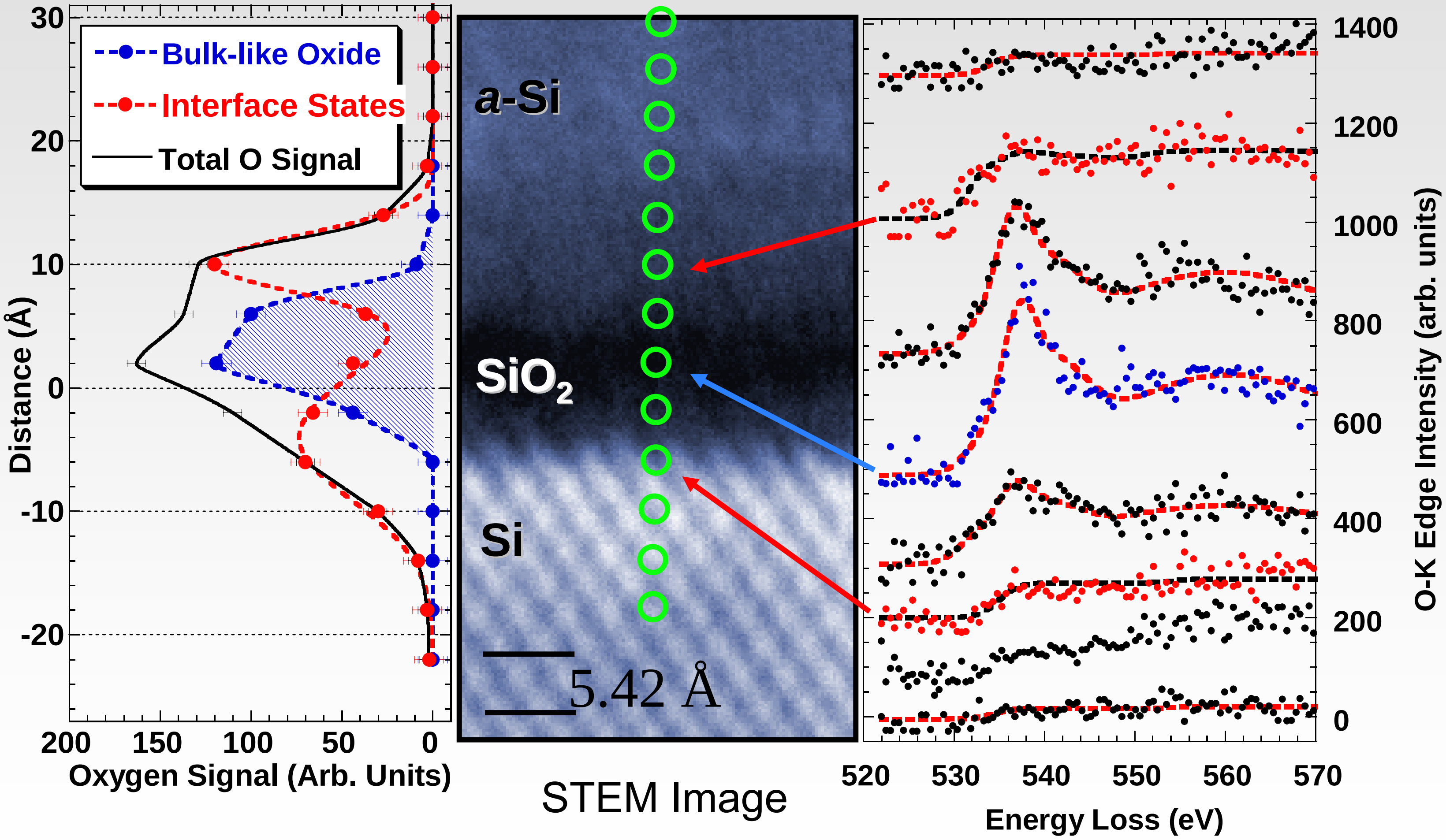
Silicon Nano-particles Embedded in Silicon Oxide



Tomographic reconstruction of the Silicon plasmon signal at 17eV

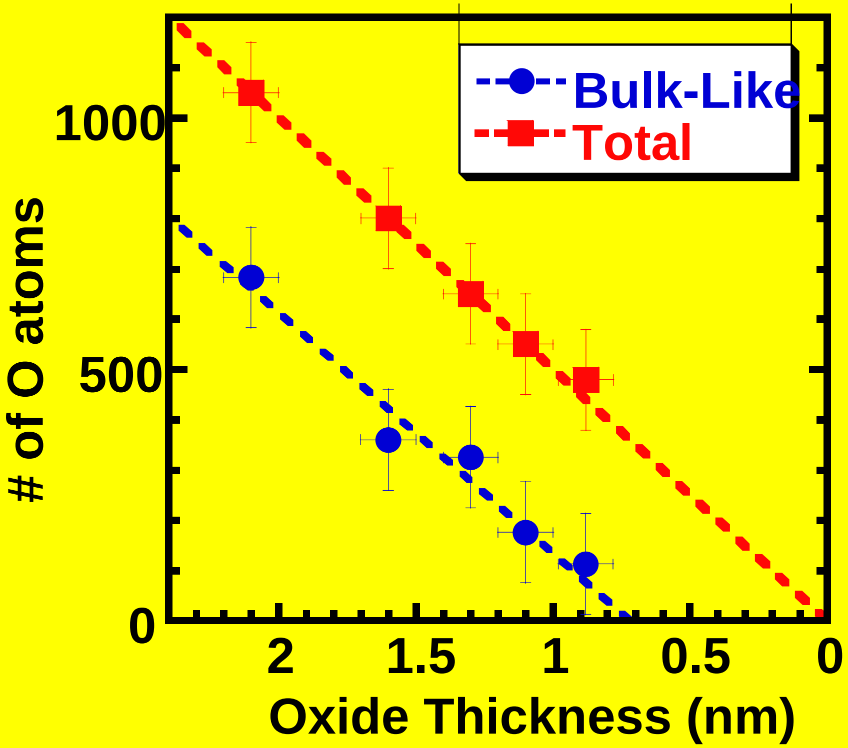
Atomic Scale Oxygen Bonding in SiO_2

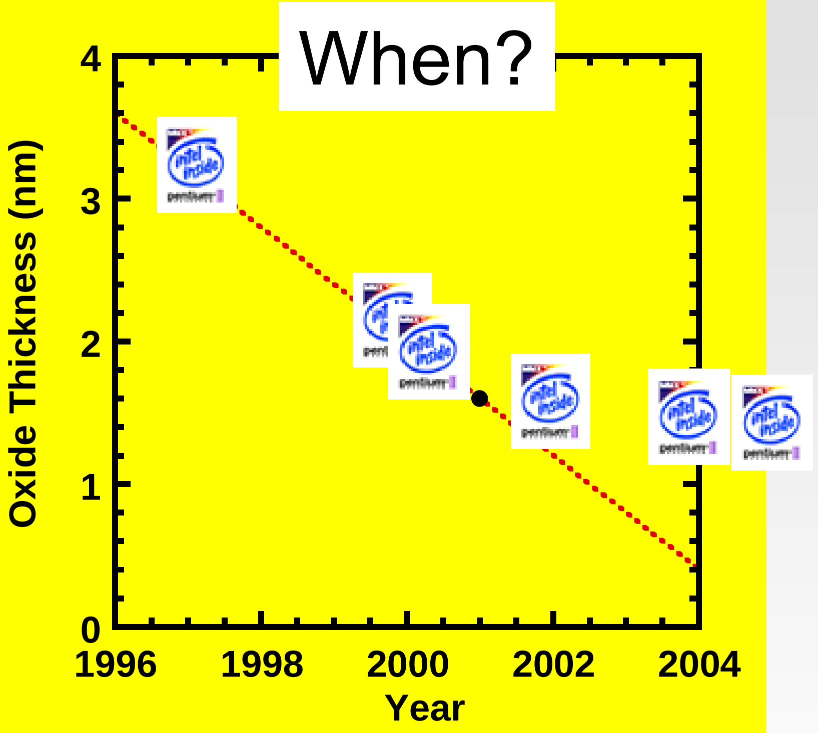
1.6 nm wide oxygen profile with 0.8 - 1 nm Bulk SiO_2



D. A. Muller et al., Nature **399**, 758 (1999).

Implications for Scaling SiO_2



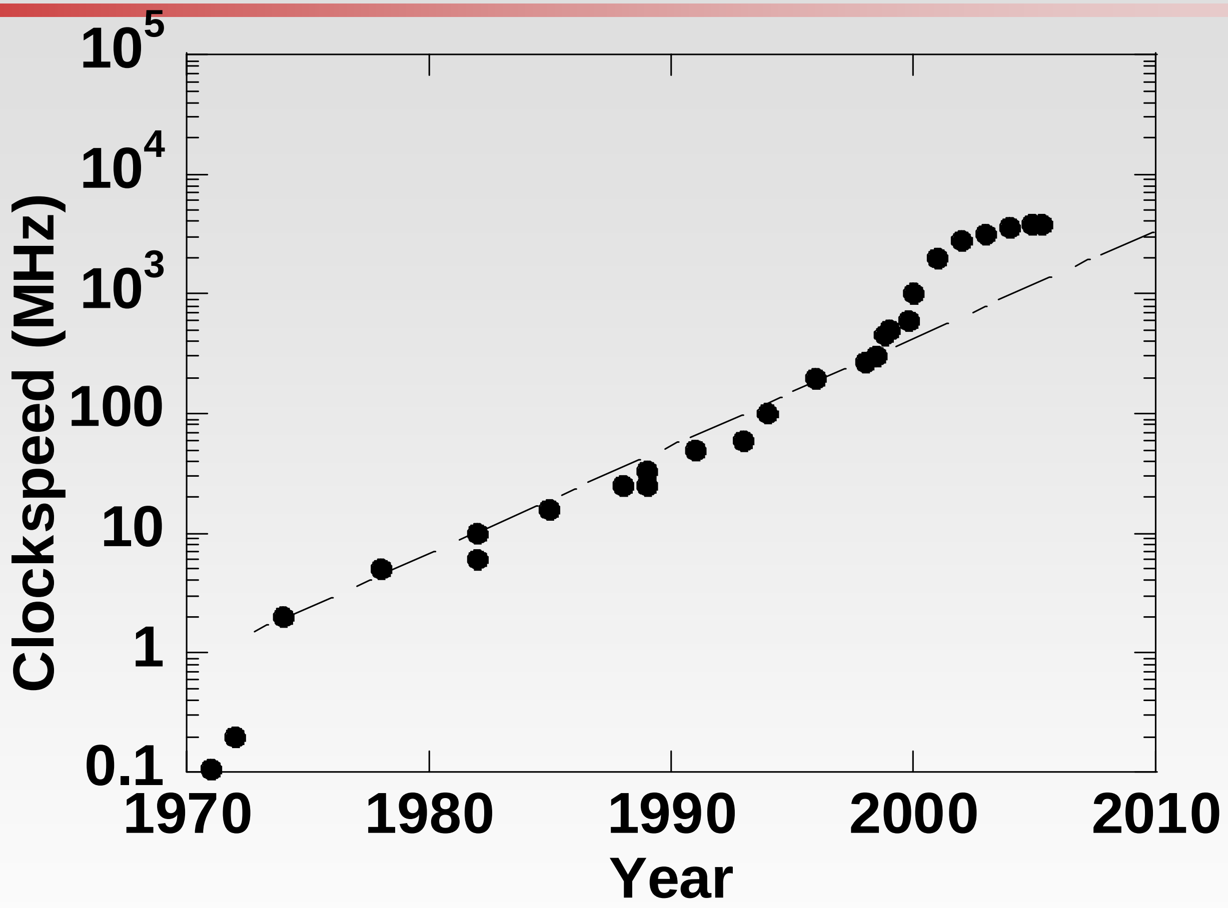


The Interface width is fixed

⇒ There will be no more Bulk-like bonding when the Oxide is less than 0.7 nm.

Theory in
J. B. Neaton, D. A. Muller, and N. W. Ashcroft, Phys. Rev. Lett. **85**, 1298 (2000).

Does Clockspeed Matter?



From 1970-2005, bits/second increased by x 3,000,000

Clockspeed increased x 40,0000

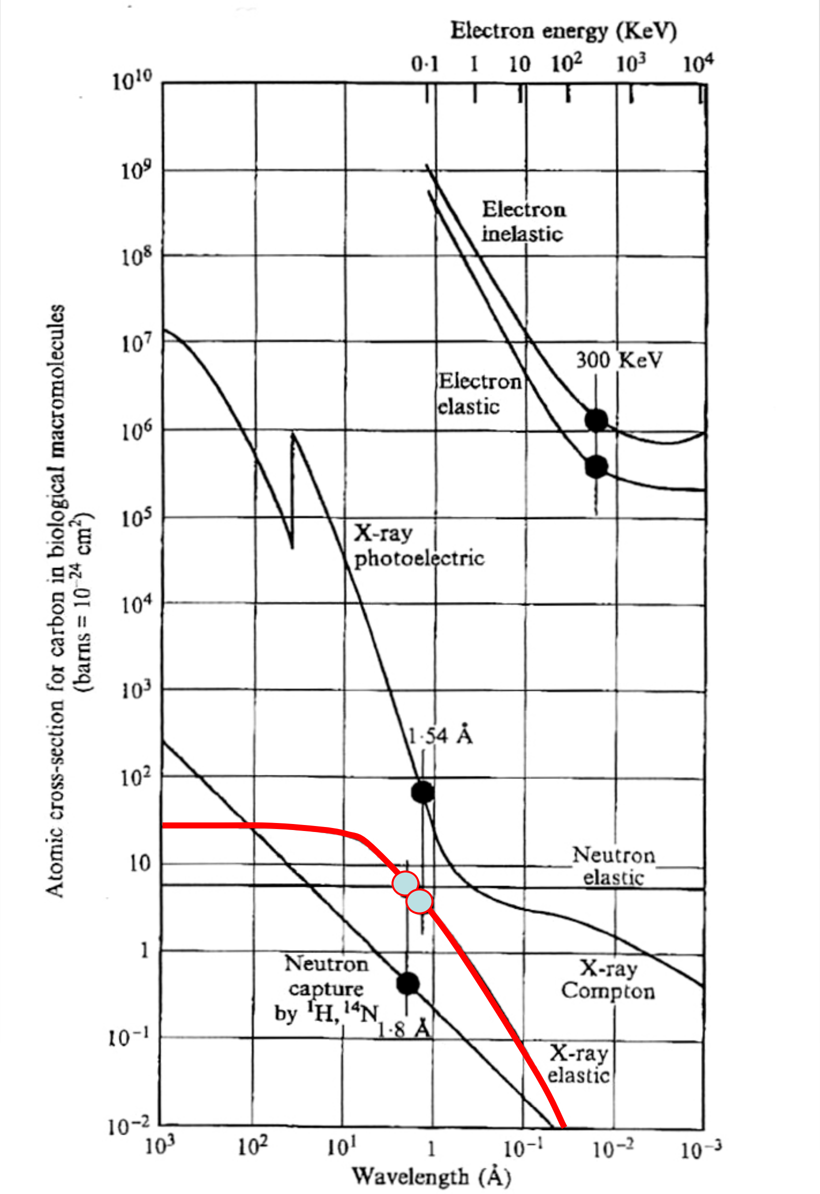
Bus Width increased x 8

"Smarter Design" - only x 5

D. A. Muller, Nature Materials, **4** p 645 (2005)

How Bad is Radiation Damage?

R. Henderson, Quarterly Reviews of Biophysics 28 (1995) 171-193.

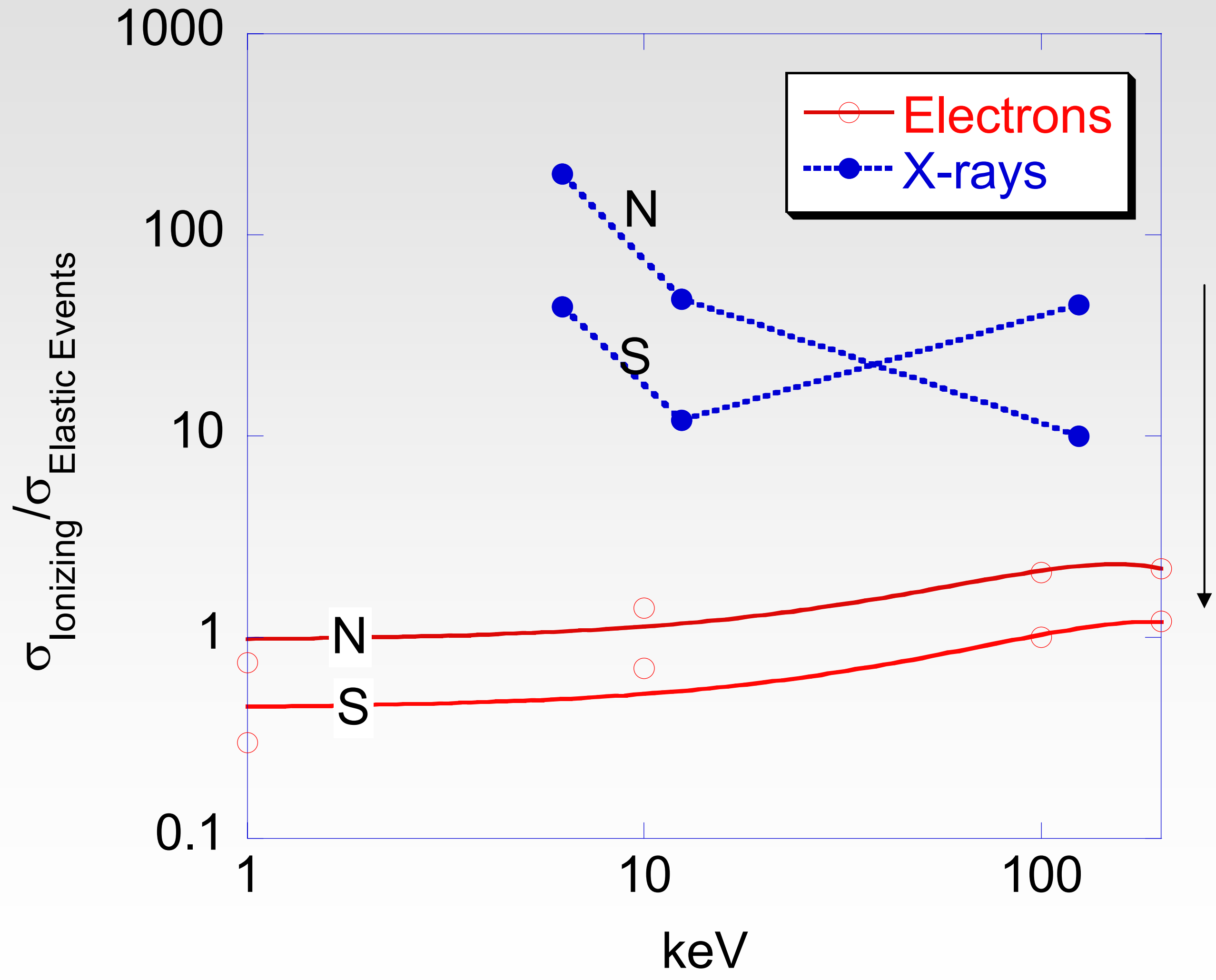


It's not the cross-section, but

How many damaging events per useful imaging event?

Least Damage:
Elastic imaging - Electrons wins
Inelastic imaging - Soft X-rays win

Radiation Damage as a Fundamental limit



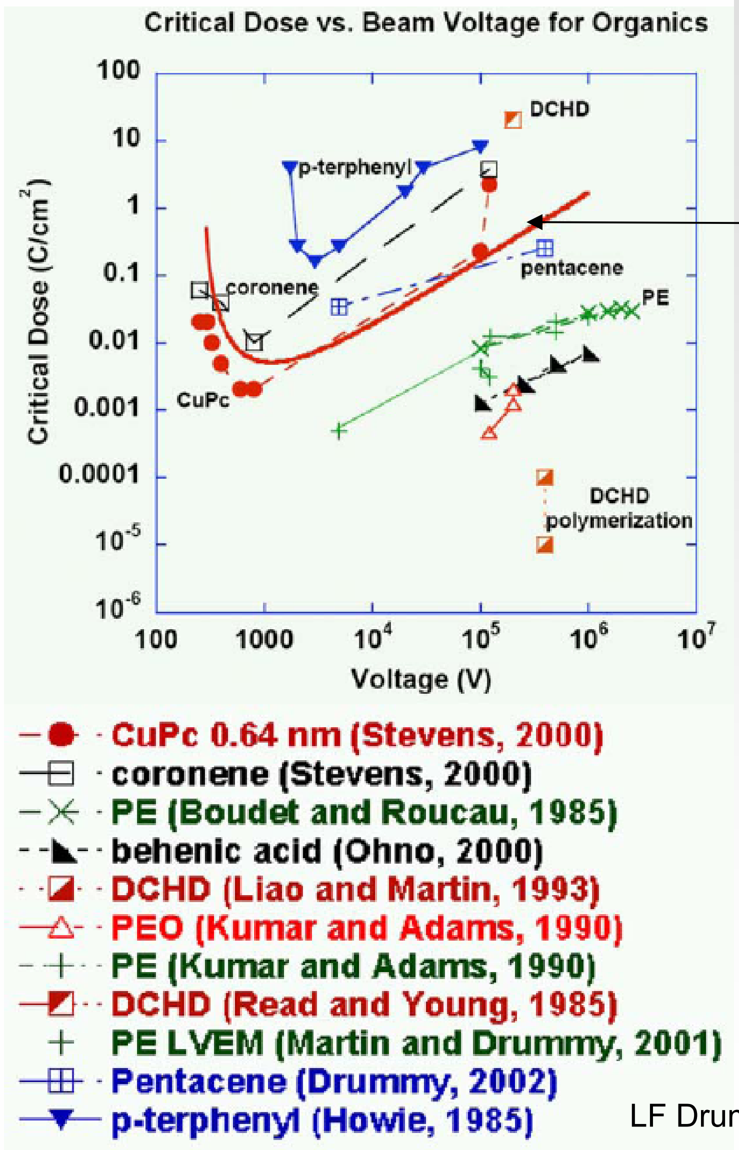
More Elastic information per damaging event

Data from Breedlove and Trammell, Science 170 (1970) 1310-1313

For electrons σ_i / σ_e ~ln (E)

What Causes the Damage?

(Temperature rise is < 2K- smaller beam is less)



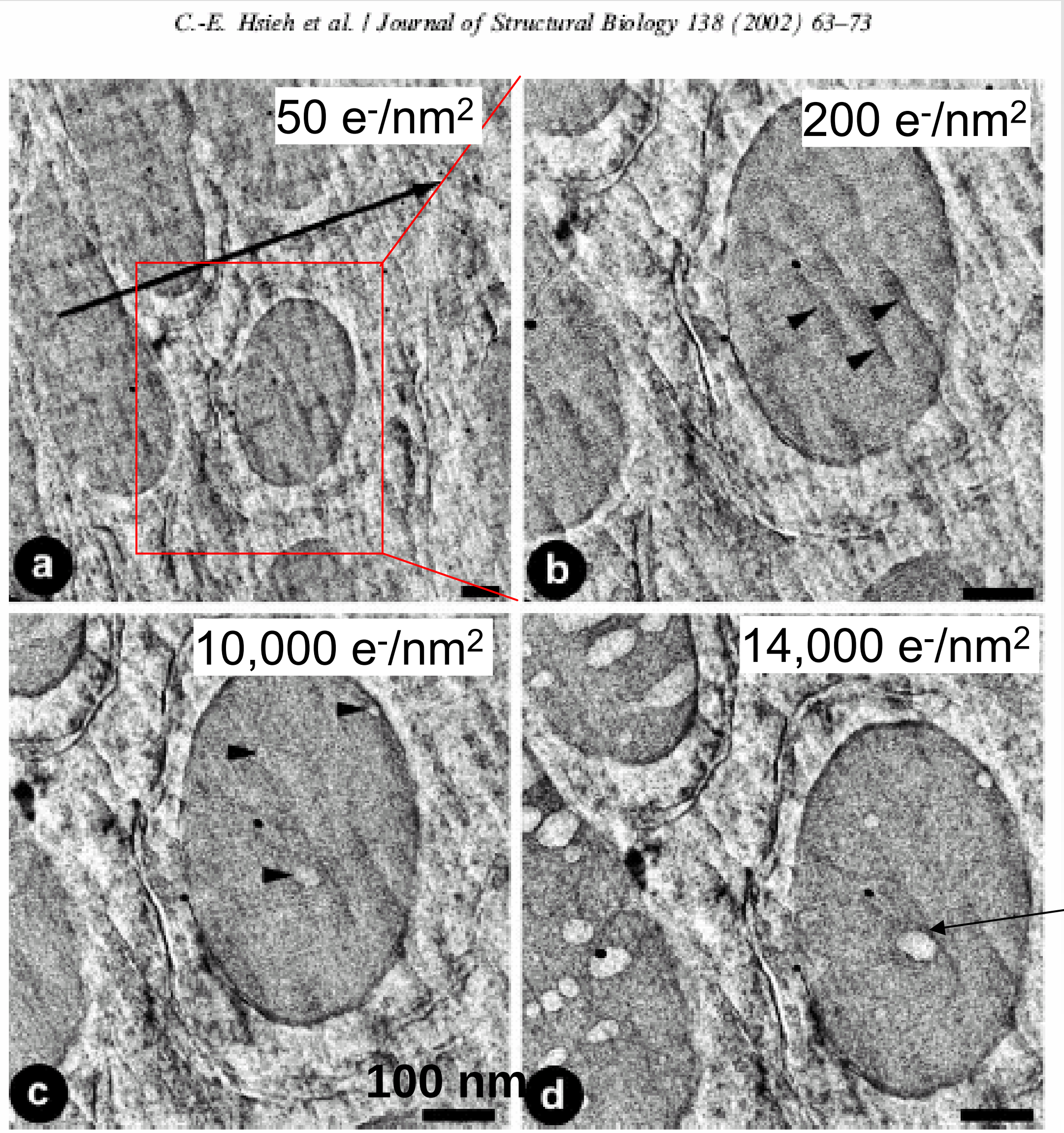
Calculated
C-K shell ionization
Cross-section

Suggests
Auger Transitions
could be suspect,
Rather than the
20 eV valence losses

LF Drummy et al. Ultramicroscopy 99 247-256

Electron Beam (400 keV) Radiation Damage in Vitreous Ice

(Damage Threshold ~ 500 e^-/nm^2)

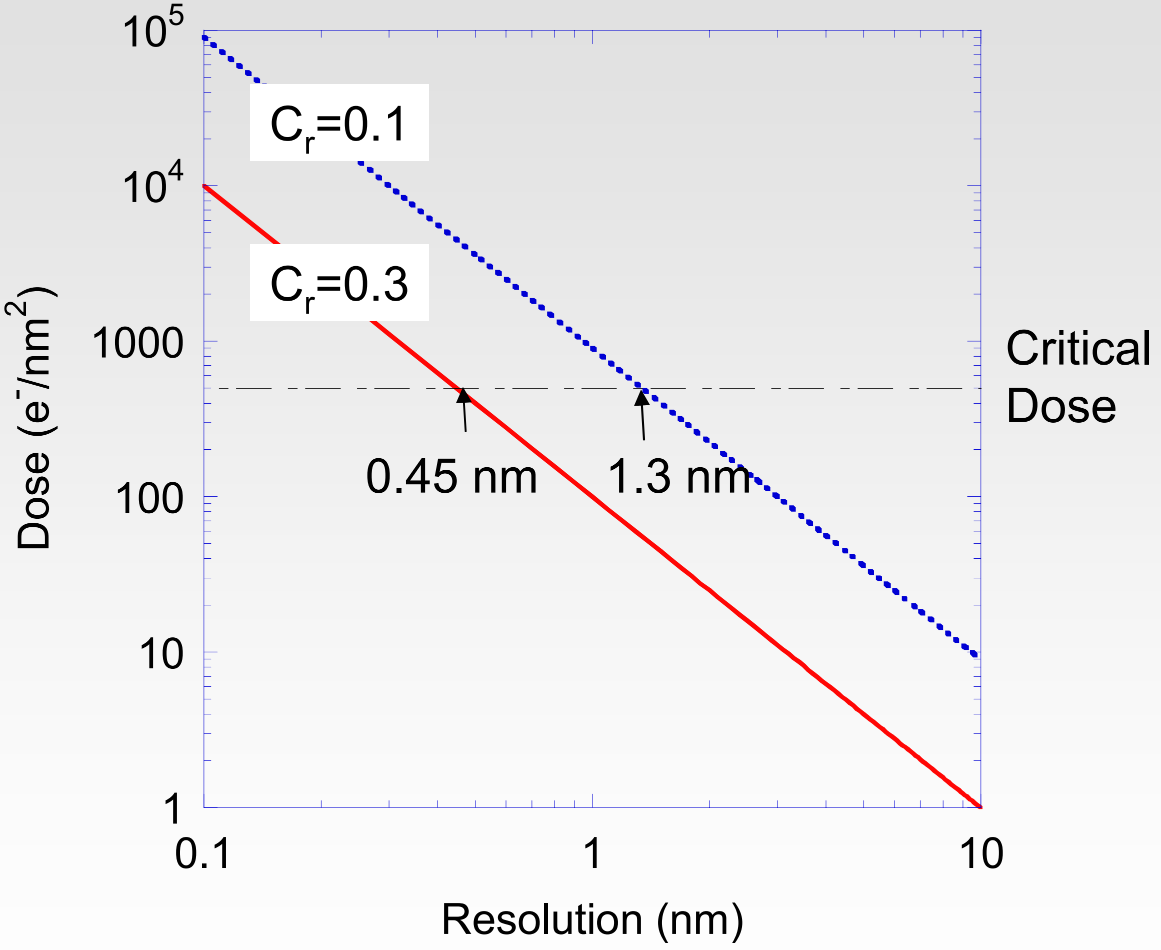

C.-E. Hsieh et al. / Journal of Structural Biology 138 (2002) 63–73

Hydrogen
Bubbles
form in densest
sections

Dose Required for 2D-Imaging

$$n_0 > \frac{k^2}{f C_r^{\,2} r^2}$$

k : S/N = 3
f : fraction contributing to Background = 1
C_r: contrast = 0.3
r: resolution



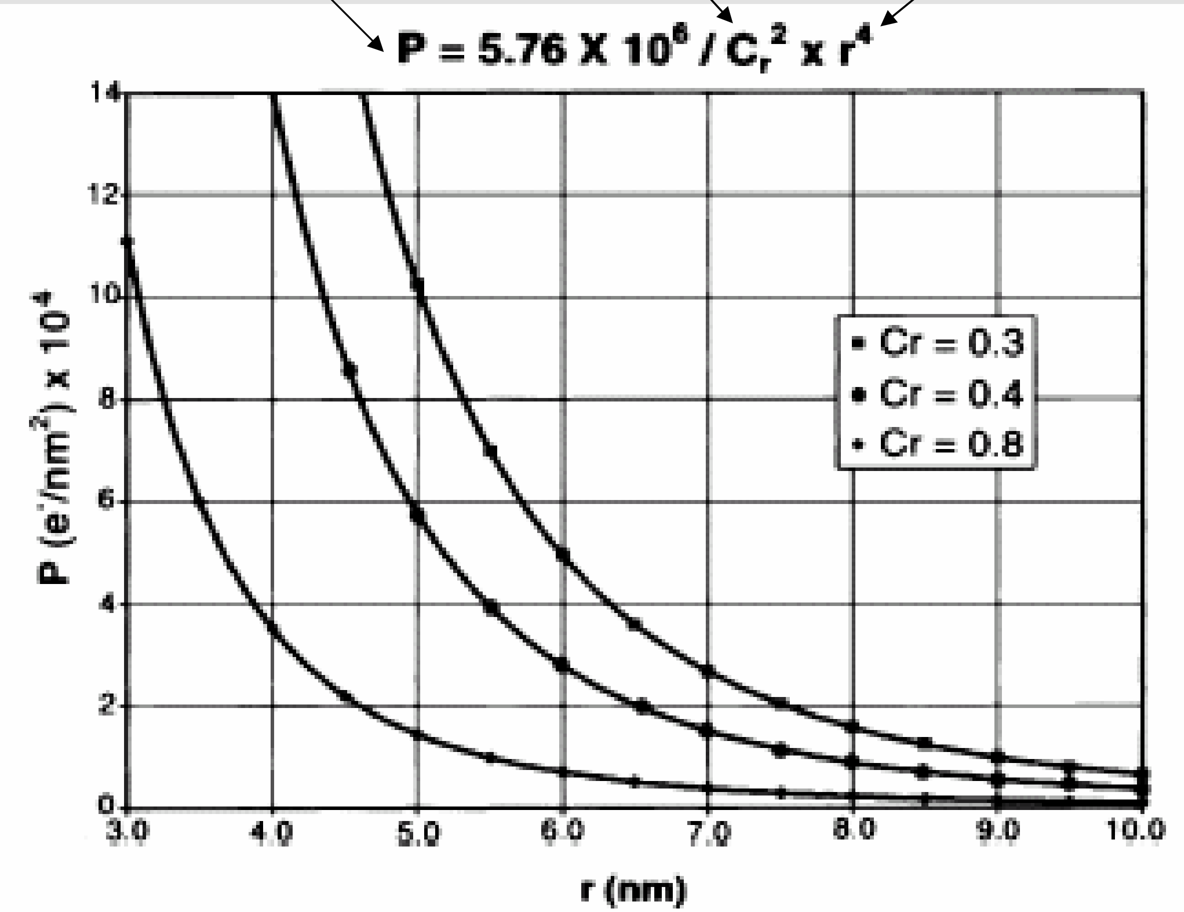
It's almost impossible to do atomic-resolution phase contrast imaging with biological samples (except by averaging over many similar molecules)!

Dose Required for 3-D Reconstructions is worse!

Dose

Contrast

Resolution

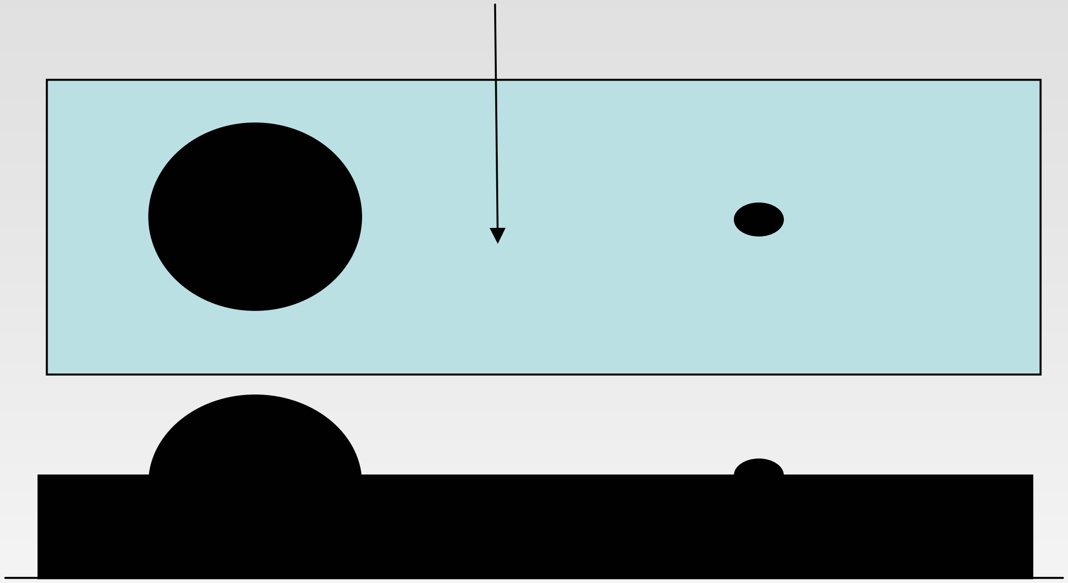


Dose α 1/(Resolution)^4

P α 1/(Contrast)^2

B. F. McEwen et al, Journal of Structural Biology **138** 47–57 (2002)
Saxberg & Saxton, W.O., Ultramicroscopy **6**, 85–90 (1981)

High Resolution= Thin Sections



Small features have low contrast (and for a fixed dose we trade 2D resolution for contrast)

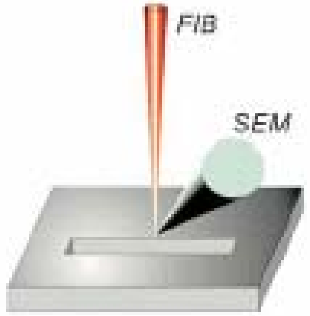
Resolution α Sample Thickness

⇨ Need to make thin samples (true for x-rays as well as electrons)

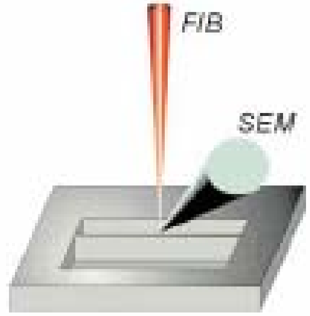
(unless we have a fluorescence detection method)

Focused Ion Beam Milling

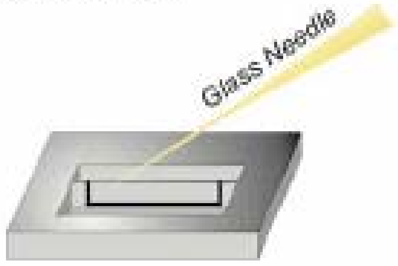
Cut out a shape with a 5-30 keV Ga+ ion beam



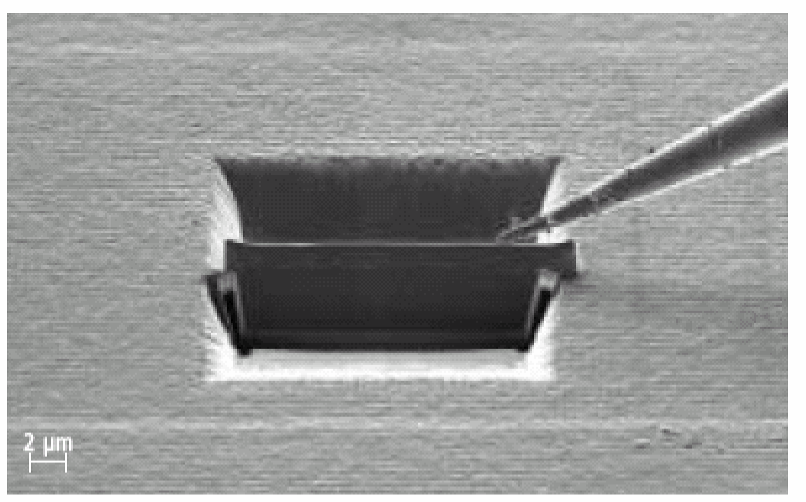
FIB milling and polishing of front side of TEM lamella



Sample is rotated 180 °and the back is milled and polished



Pick up of TEM lamella with Lift-out Tool



Sample can be
As thin as 100 nm
(but damage layer
Is 10-30 nm/side)

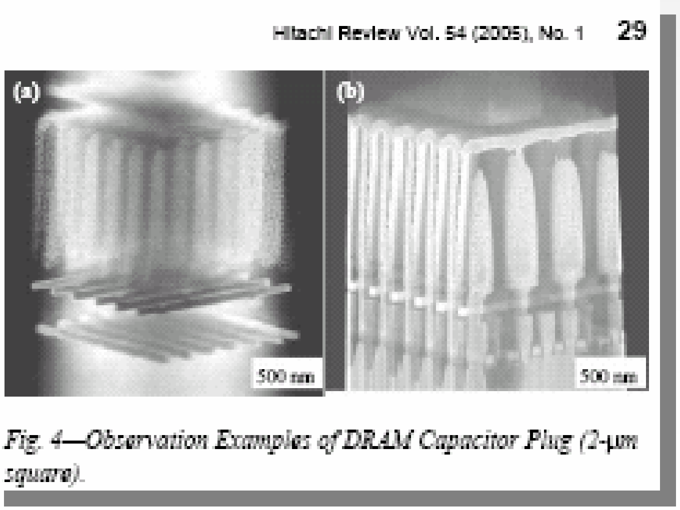
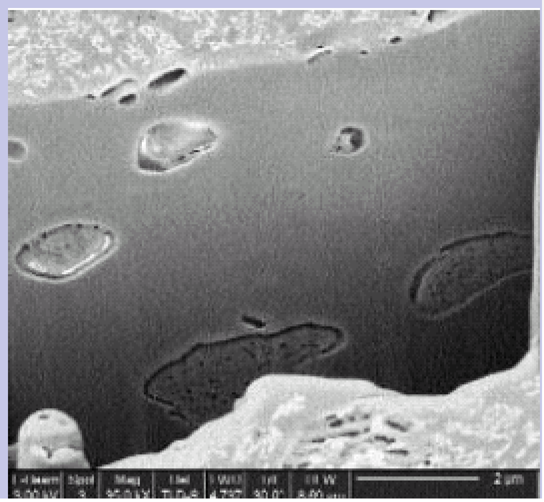
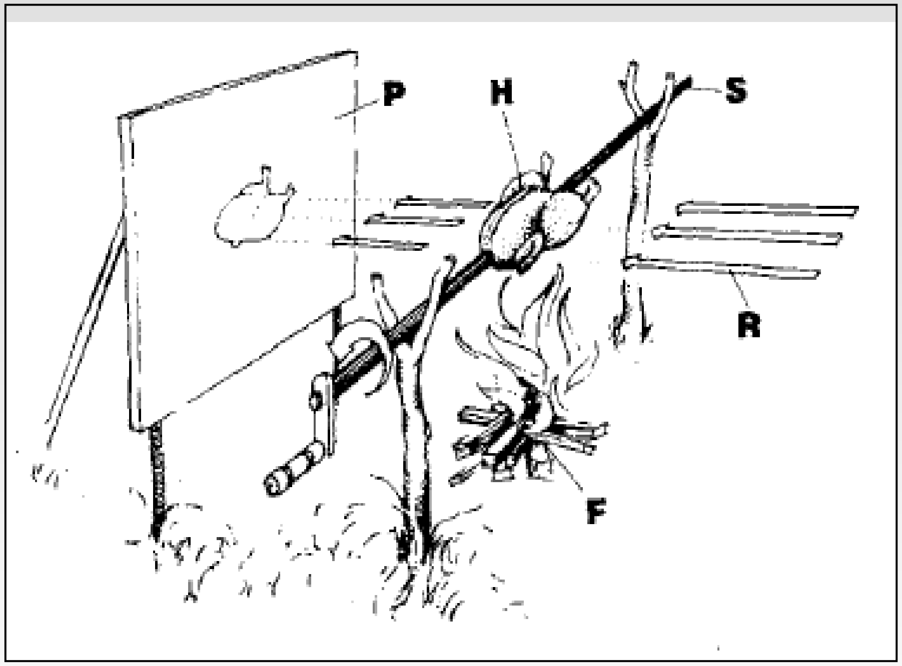

Hitachi Review Vol. 54 (2005), No. 1 29

Fig. 4—Observation Examples of DRAM Capacitor Plug (2-µm square).



Water Droplets in Liquid Margarine

Tomography at the Nanoscale

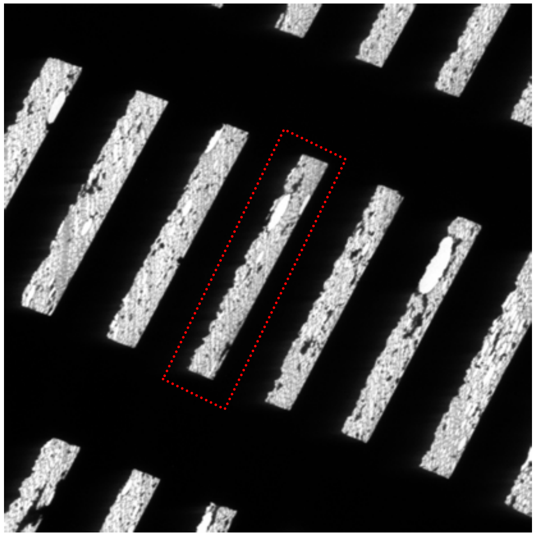


Walter Hoppe, *Angew. Chem. Int. Ed. Engl.* ***22*** *(1983) 456-485*

3D resolution function
along X, *dx* ~ 0.2 nm
along Y, dy ~ 1 nm
along Z, *dz* ~ 1 nm
(due to limited tilt range and finite number of projection images)

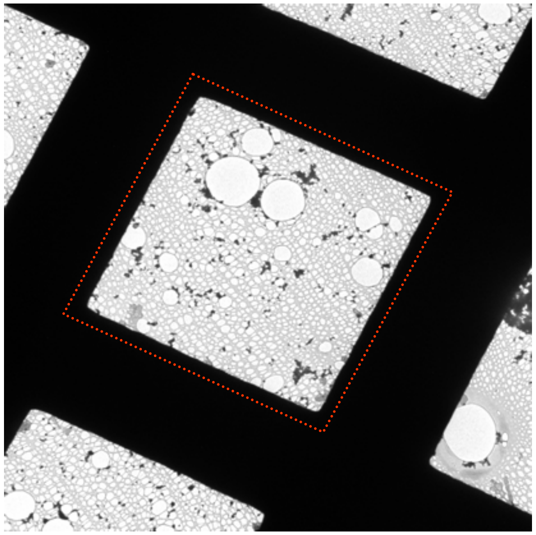
Sample thickness: 20-600 nm

High tilt tomography holder (Fischione 2020)



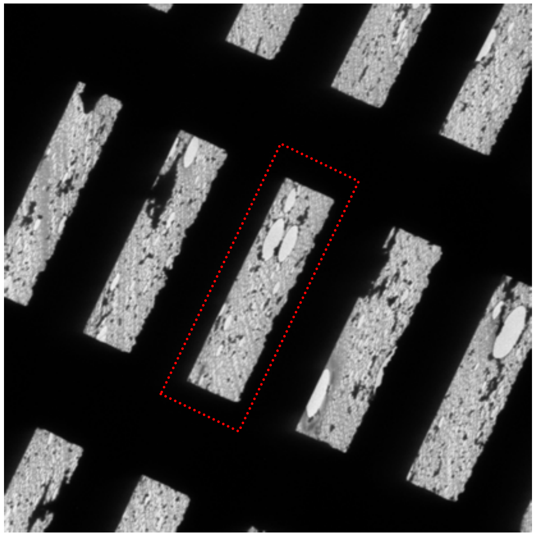
-80º

Limit of goniometer α tilt



No tilt (0º)

Low magnification (57x)
CCD image

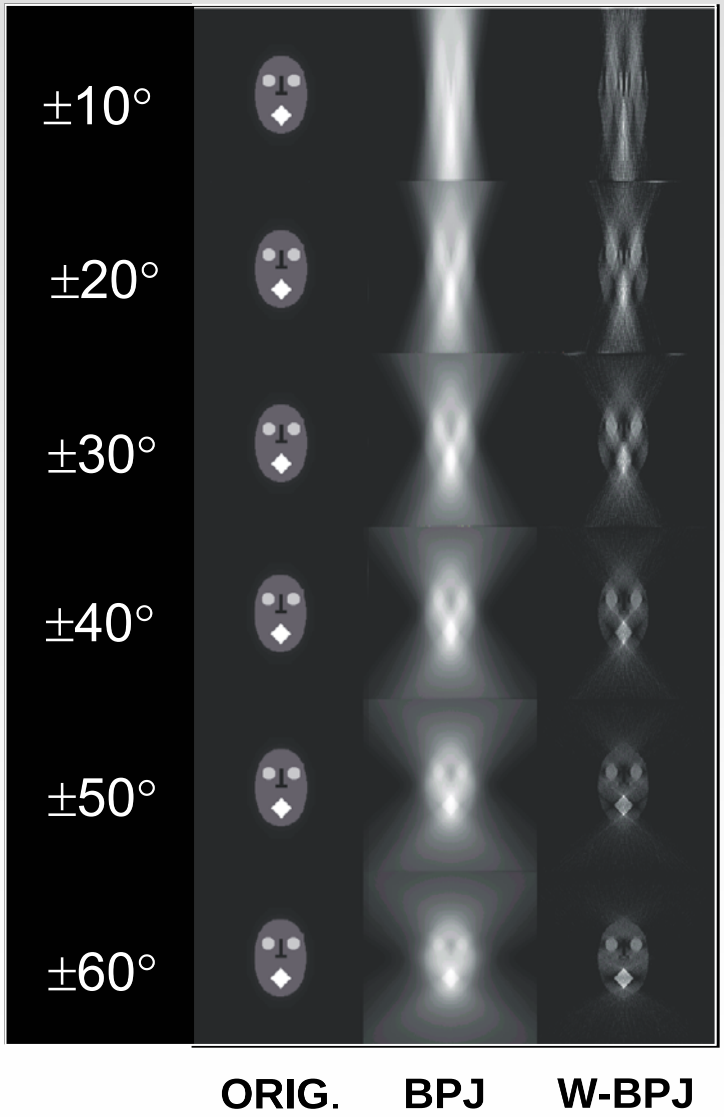


+80º

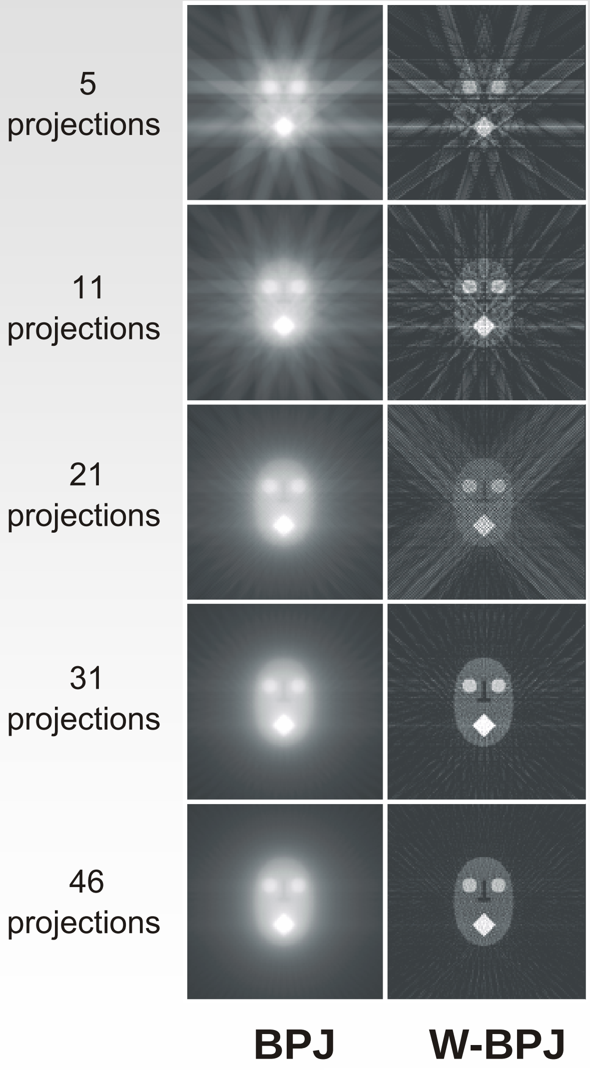
Limit of goniometer α tilt

Finite Sampling

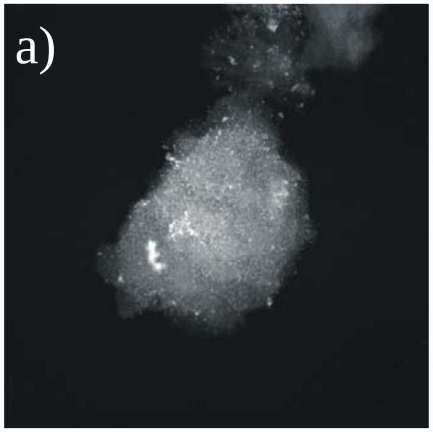
Effect of Tilt



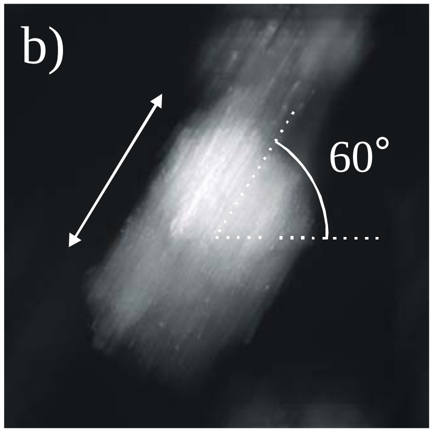
Effect of Sampling



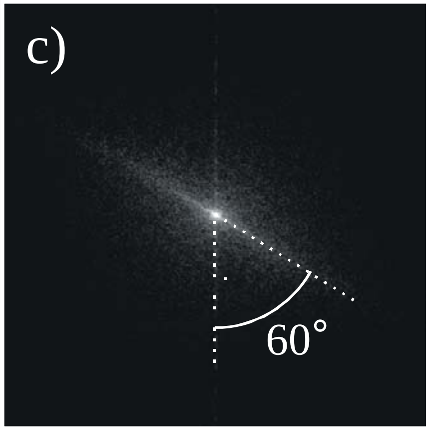
Determining the tilt axis



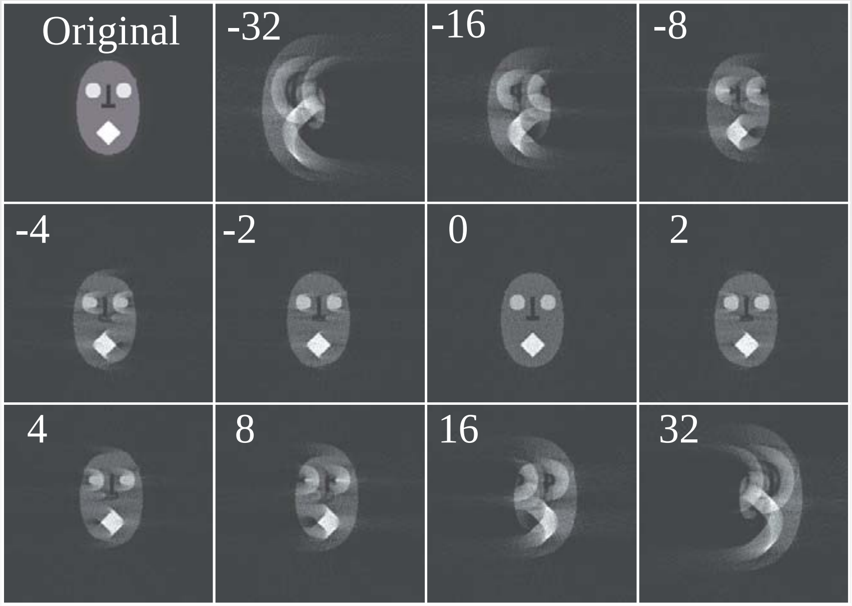
Single Image



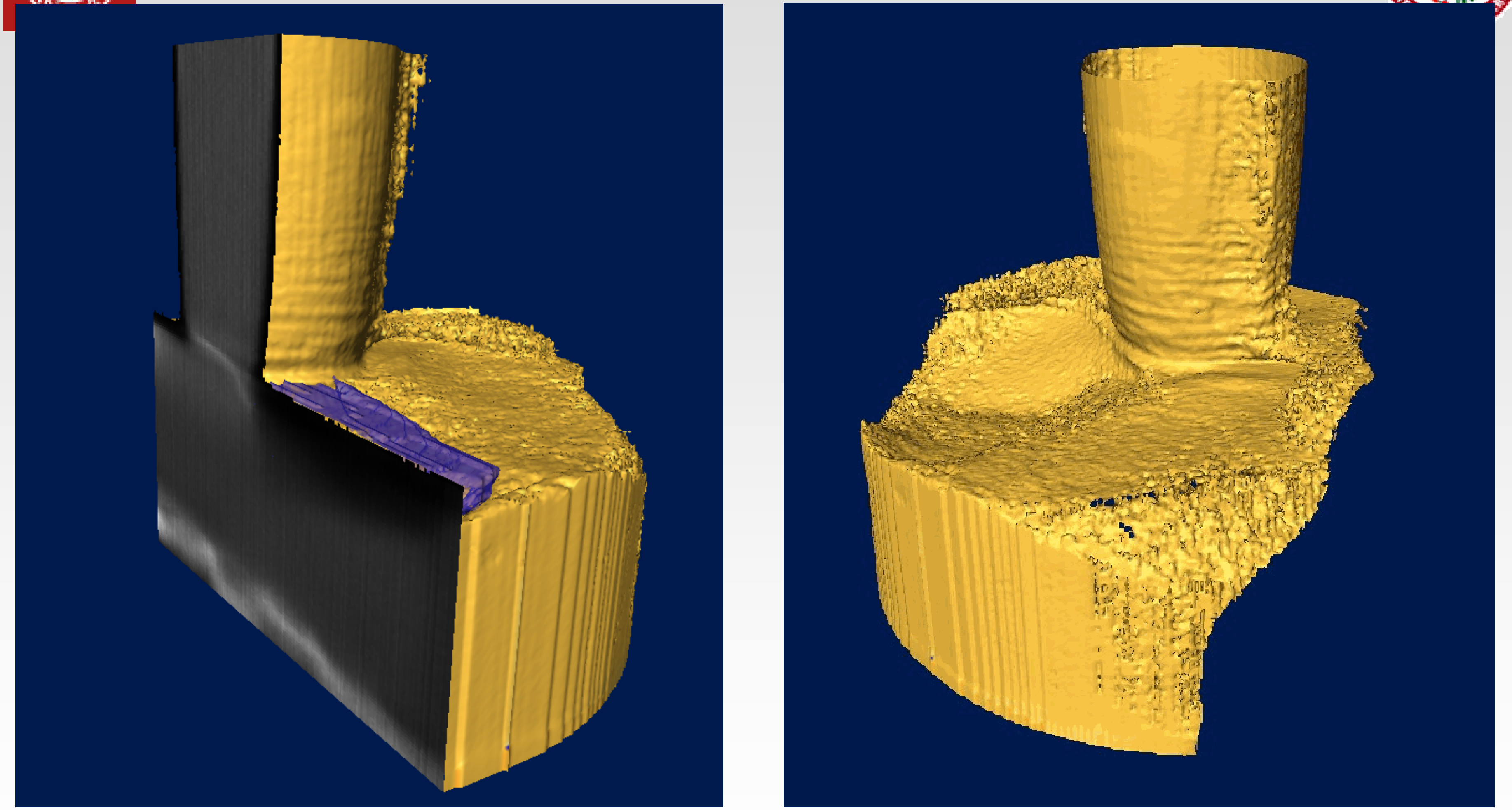
Projection through aligned series



Power spectrum of (b)



Stress Void Reconstruction

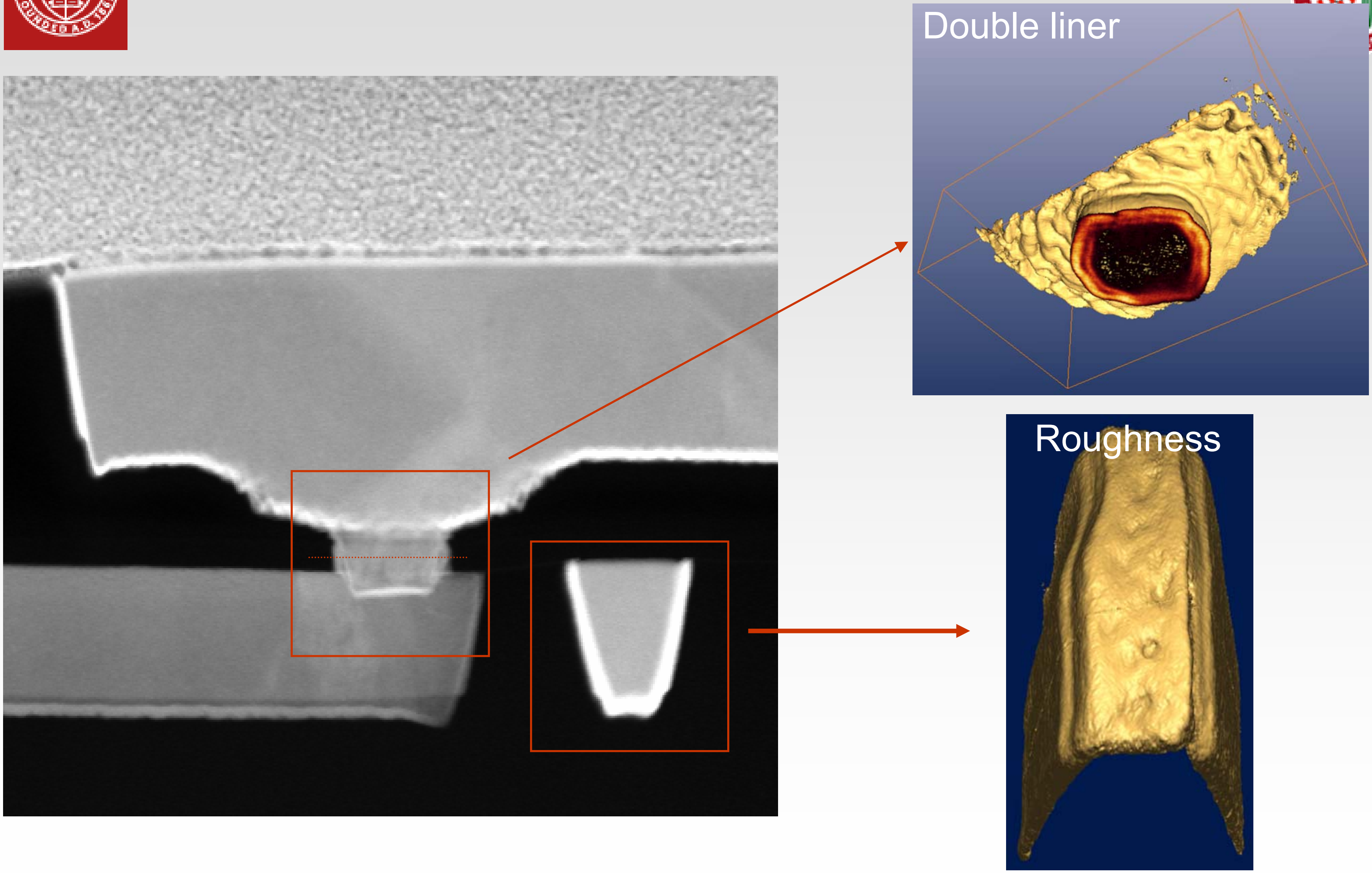


Via is 250 nm thick, inside a 500 nm thick Cu section

P. Ercius, M. Weyland, D. A. Muller, L. M. Gignac, *Appl. Phys. Lett.* **88** 243116 (2006).

3D Imaging Inside Interconnects

(100 nm wires inside an IBM chip)



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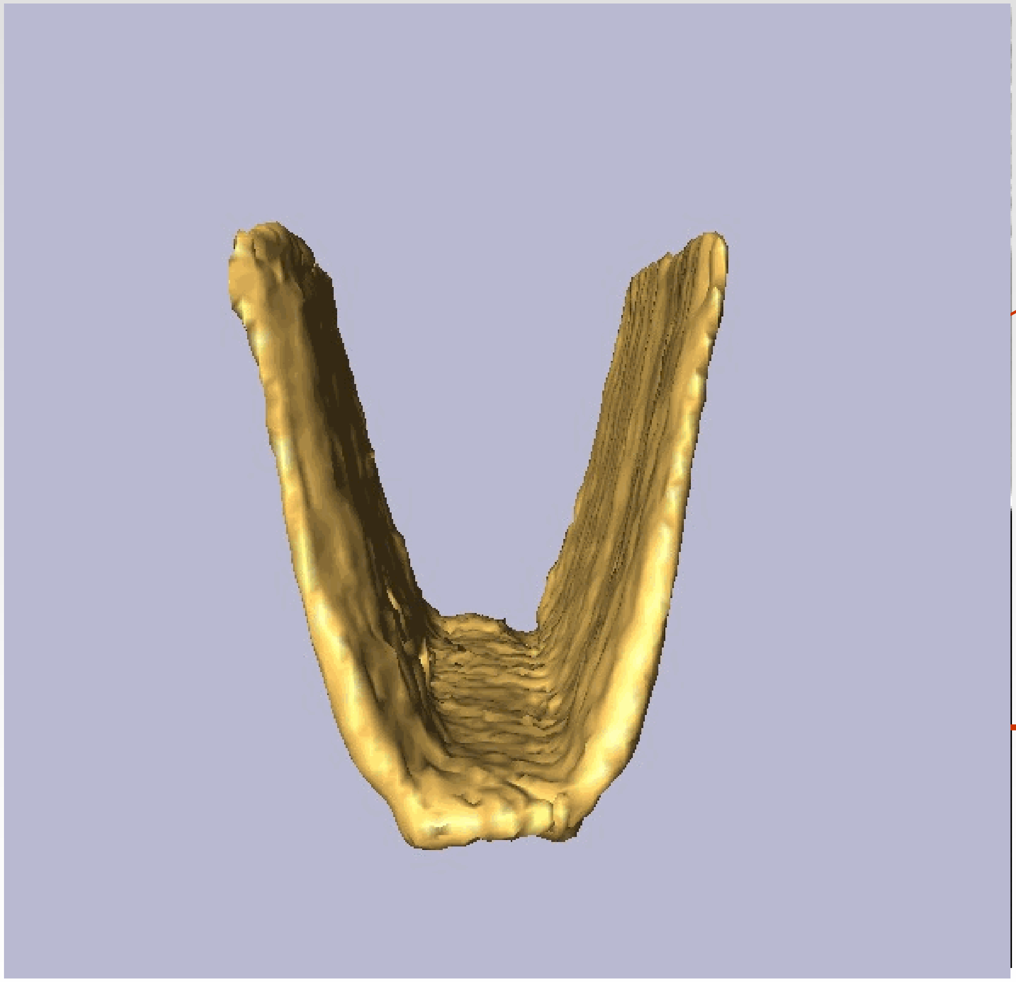
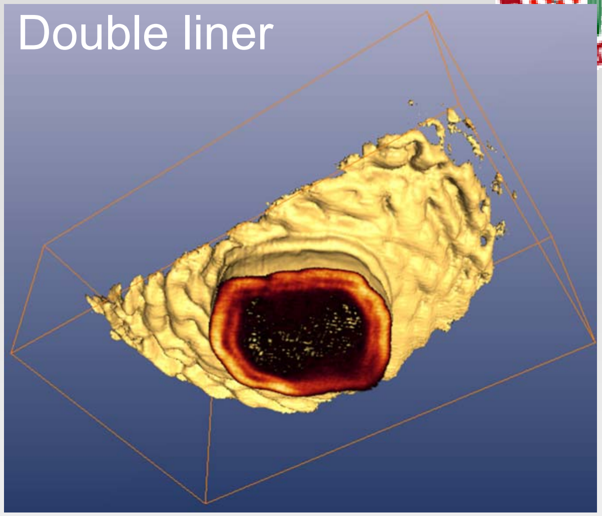
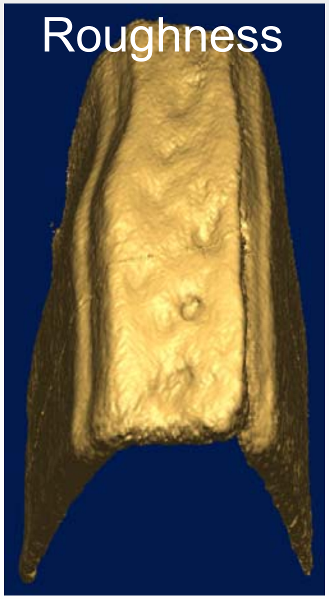


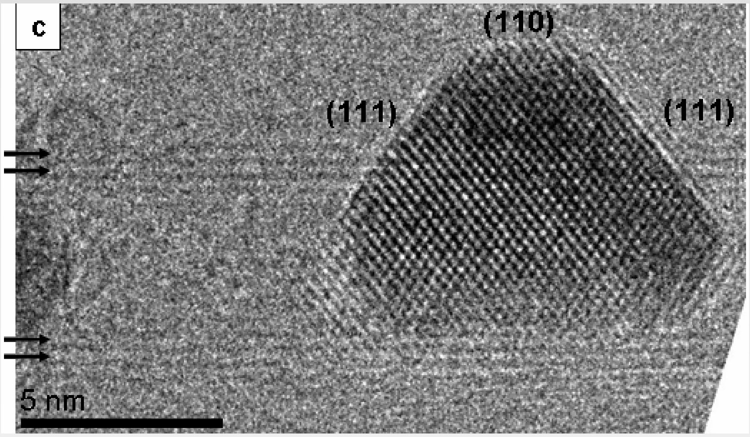


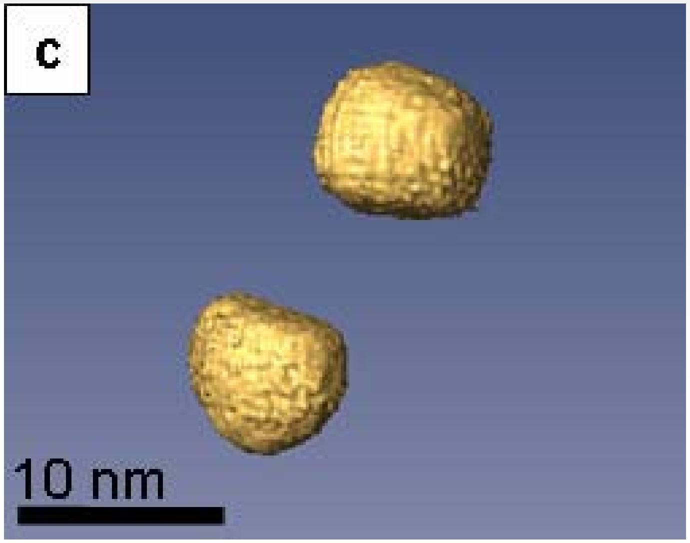
Double liner

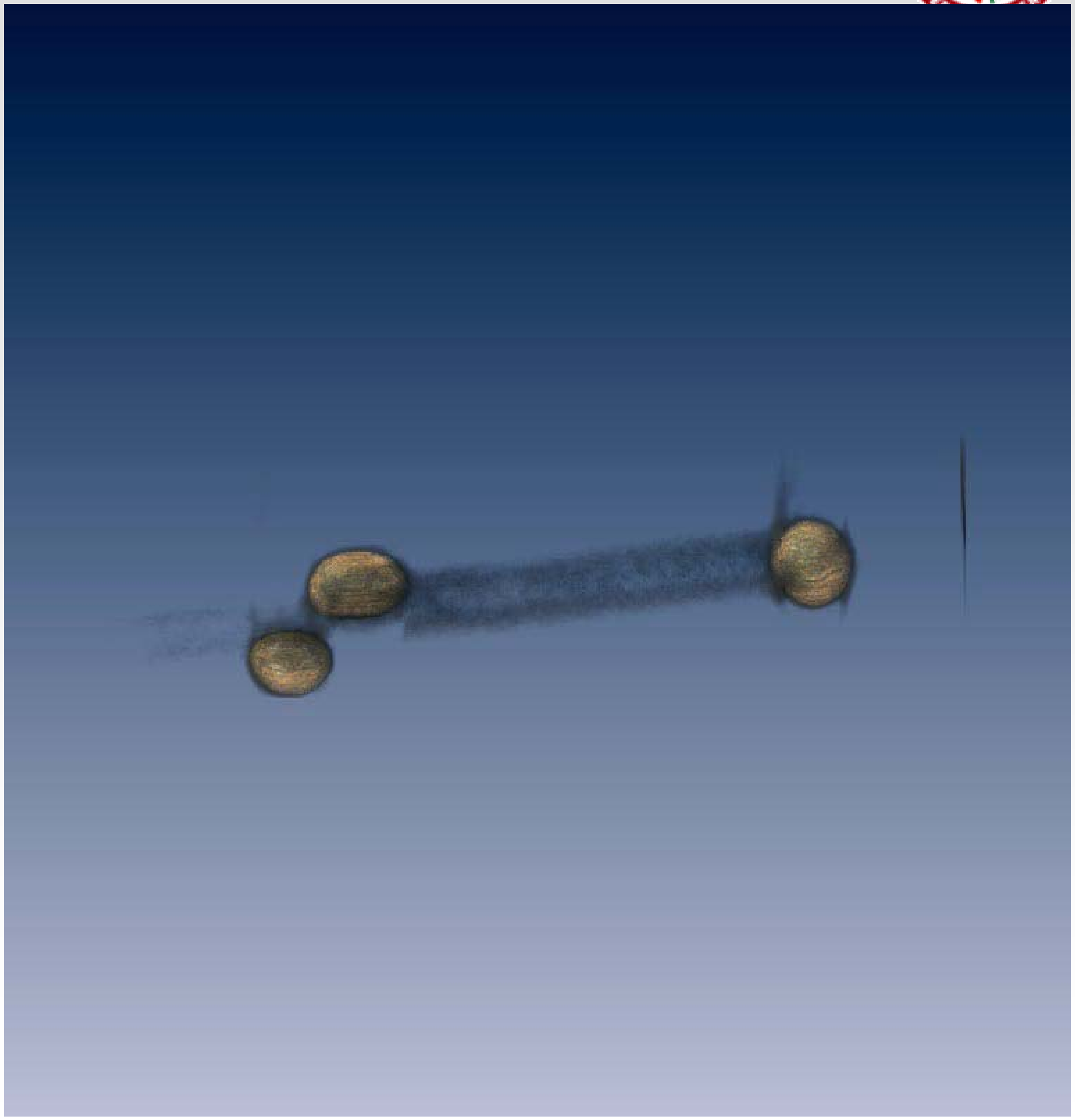


Roughness

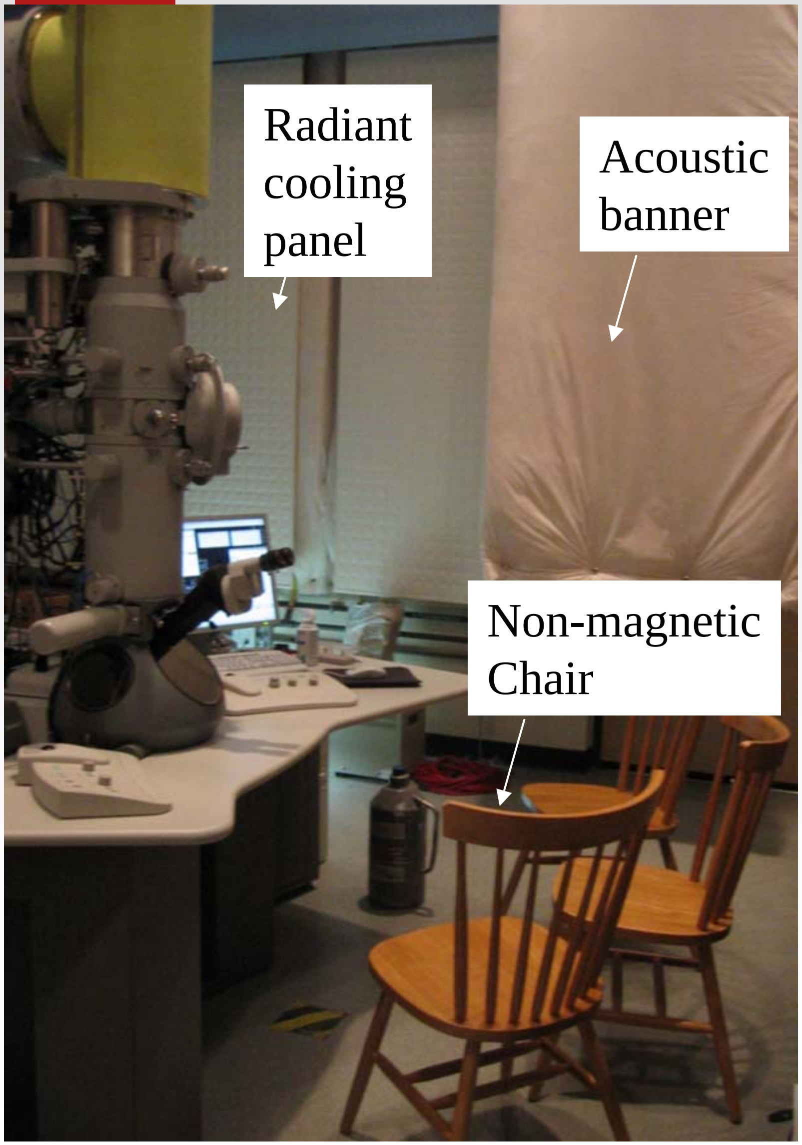
How Metal Contacts Form on a Carbon Nanotube





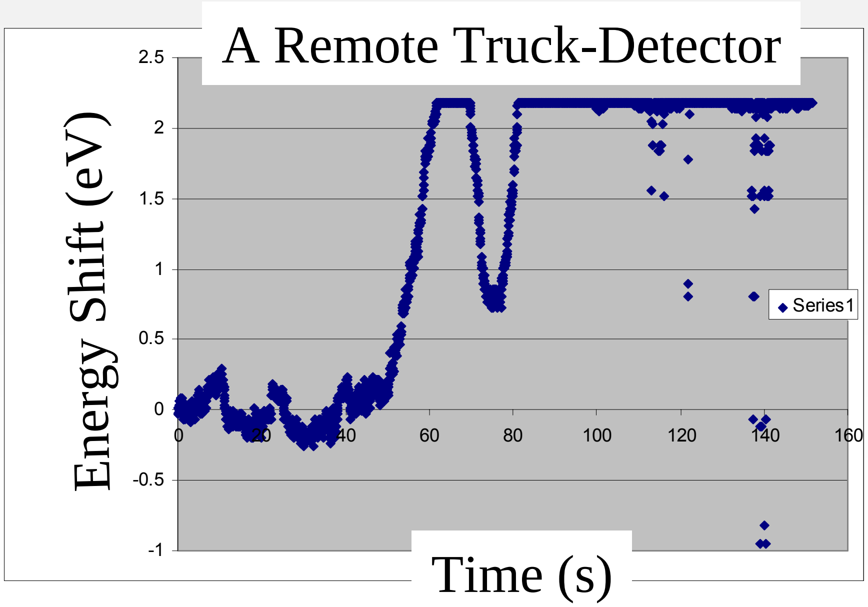


Environmental Sensitivity



We don’t just see atoms:

- •Can detect moving chairs, elevators, trucks and air pressure changes.
- •“Drift to the right, rain tonight”


A Remote Truck-Detector

Cooling and Airflow

"DuctSox" porous mesh for uniform, low airflow

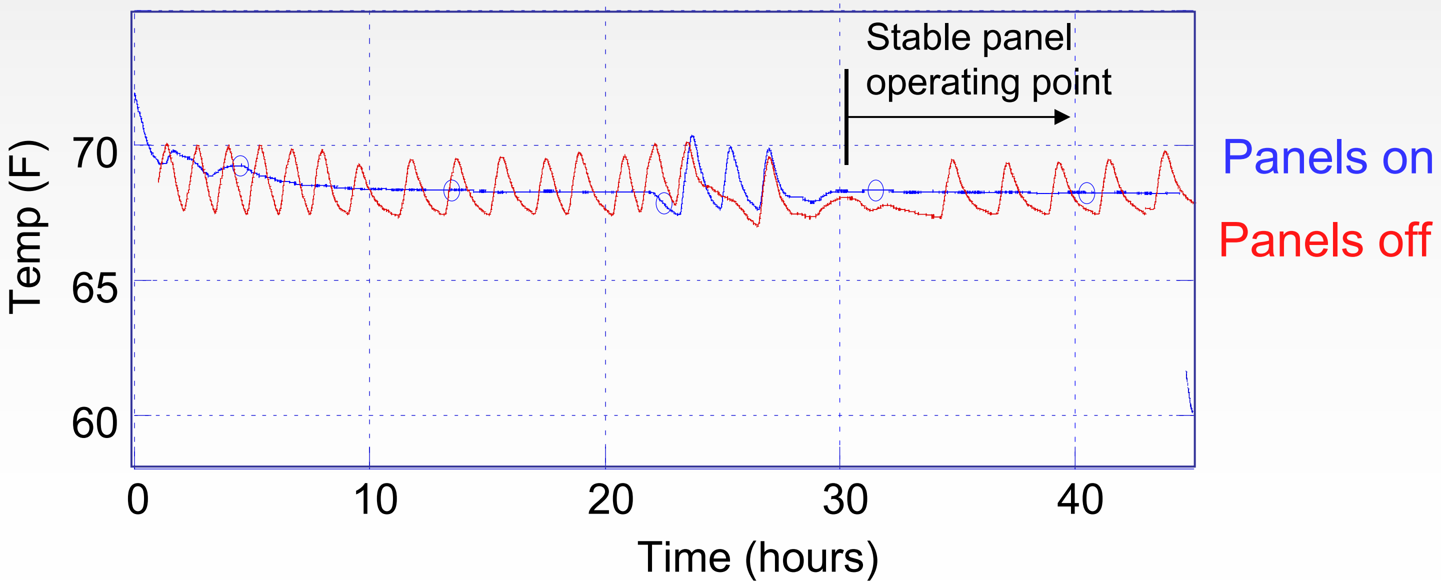
Radiant cooling panel

Acoustic damping material

Radiant Cooling Panels: Heat Transfer without Airflow

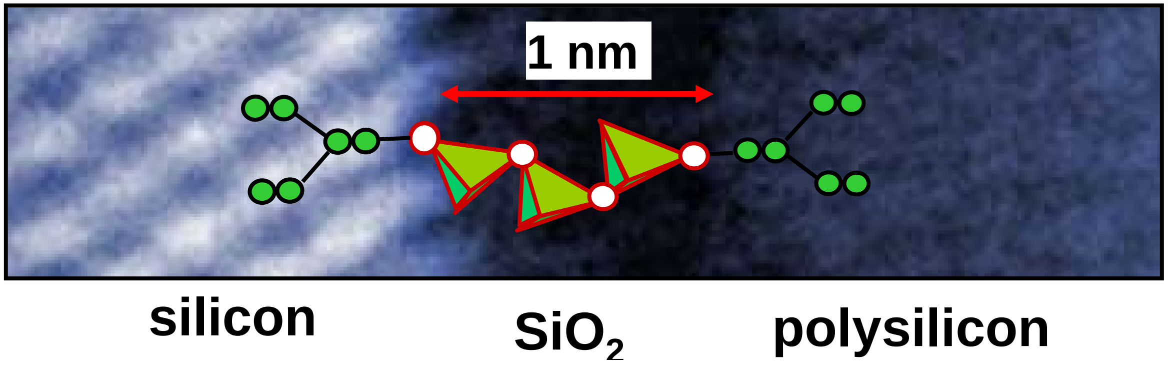
(allows us to cut back airflow – which is now used to control humidity)

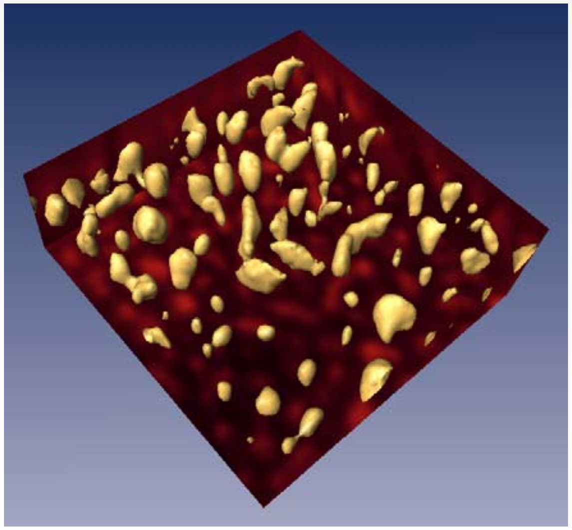
- Radiant cooling panel temperature regulated by closed-loop chiller
- Brings room into thermal equilibrium with panels by radiative transfer
- By tuning the panel temperature, we can keep the building heat from pulsing
- Effect is to add a huge thermal mass to the room (a giant wine cellar)



Outlook

- *Electron Microscopy: 0.5-0.7 Å resolution (1-2 Å standard today)*
  0.1-0.5 eV energy resolution
  Sample thickness < 100-1000 nm
  Small working distance (~3-10 mm)
  Nitride-window e-cells for imaging liquids
- *X-Ray Microscopy:*
  - Radiation damage will be worse for elastic imaging ($1/r^4$ in 3D)
  - Best resolution will require TEM-like sample preparation
  - 10 nm res & 1- 10 µm thick samples for whole-cell mapping





Acknowledgements

Peter Ercius

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Grain Boundaries in Ni_3Al (Cornell)

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Imaging Individual Dopant Atoms (Bell Labs)

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SiO_2 and High-k Gate Oxides (Bell Labs)

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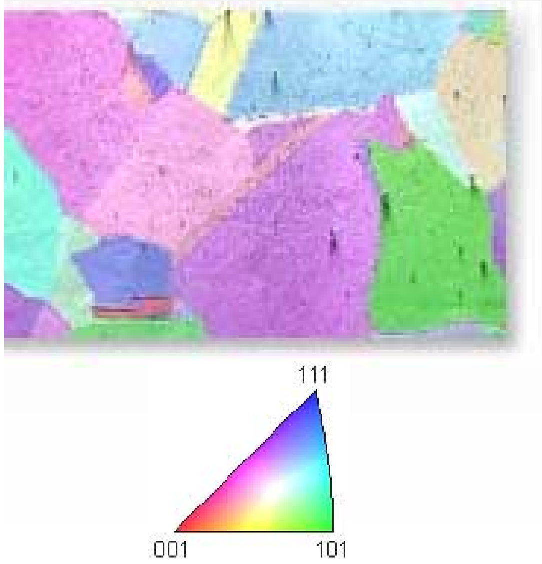
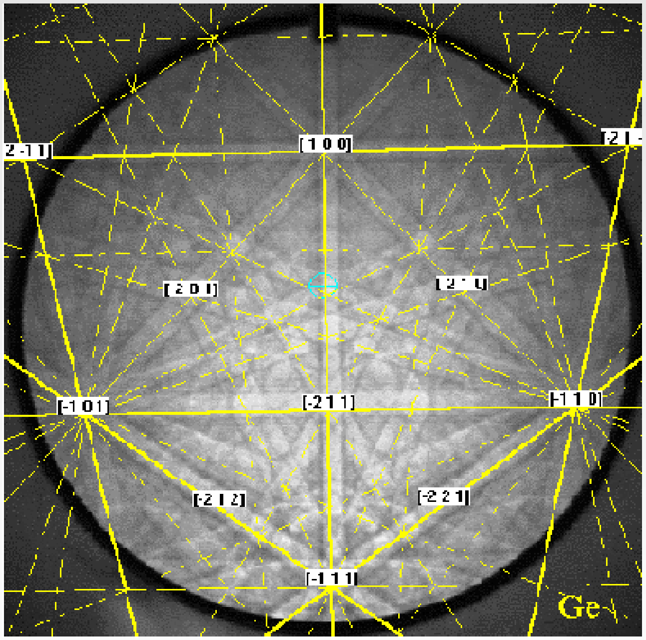
Electron Backscatter Imaging

EBSD, EMPA,
John Hunt
hunt@ccmr.cornell.edu



Use electron channeling patterns to
Produce maps of grain orientations



Needs clean surfaces, grains > 200 nm

(With a FEG-SEM, as small as 20-50 nm)