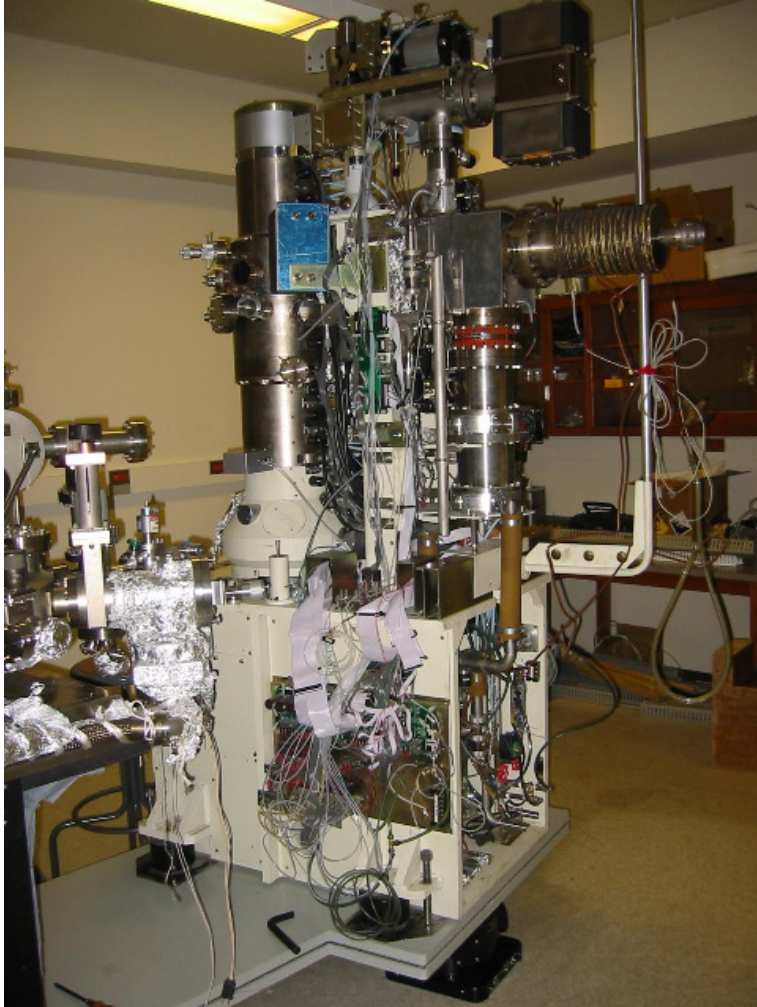
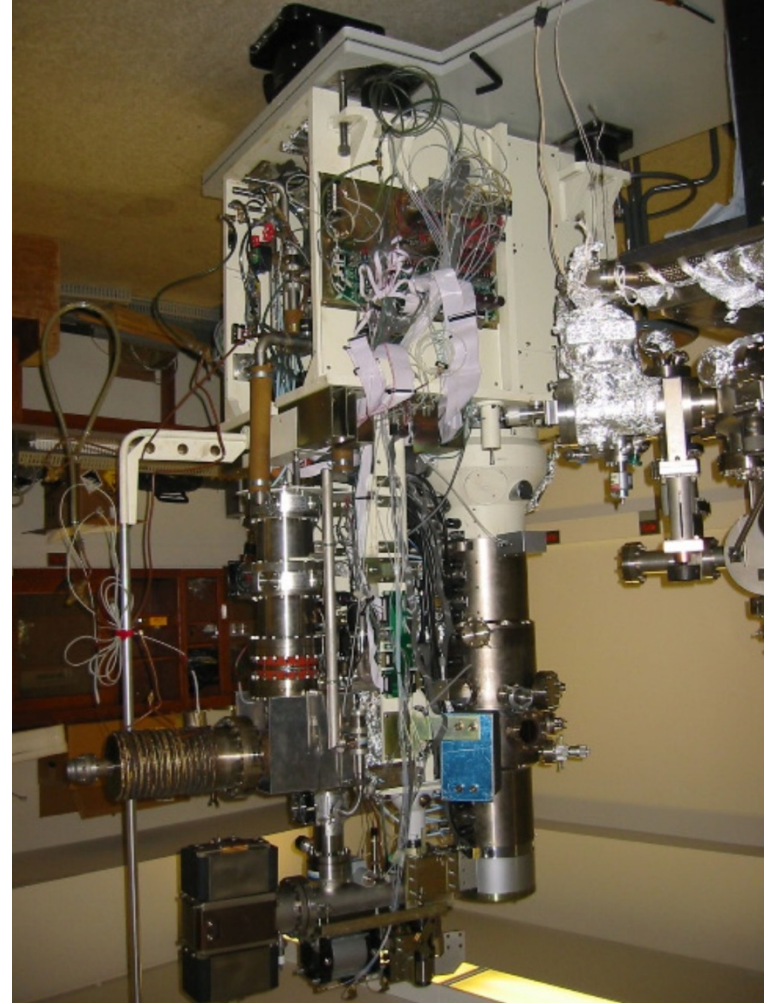
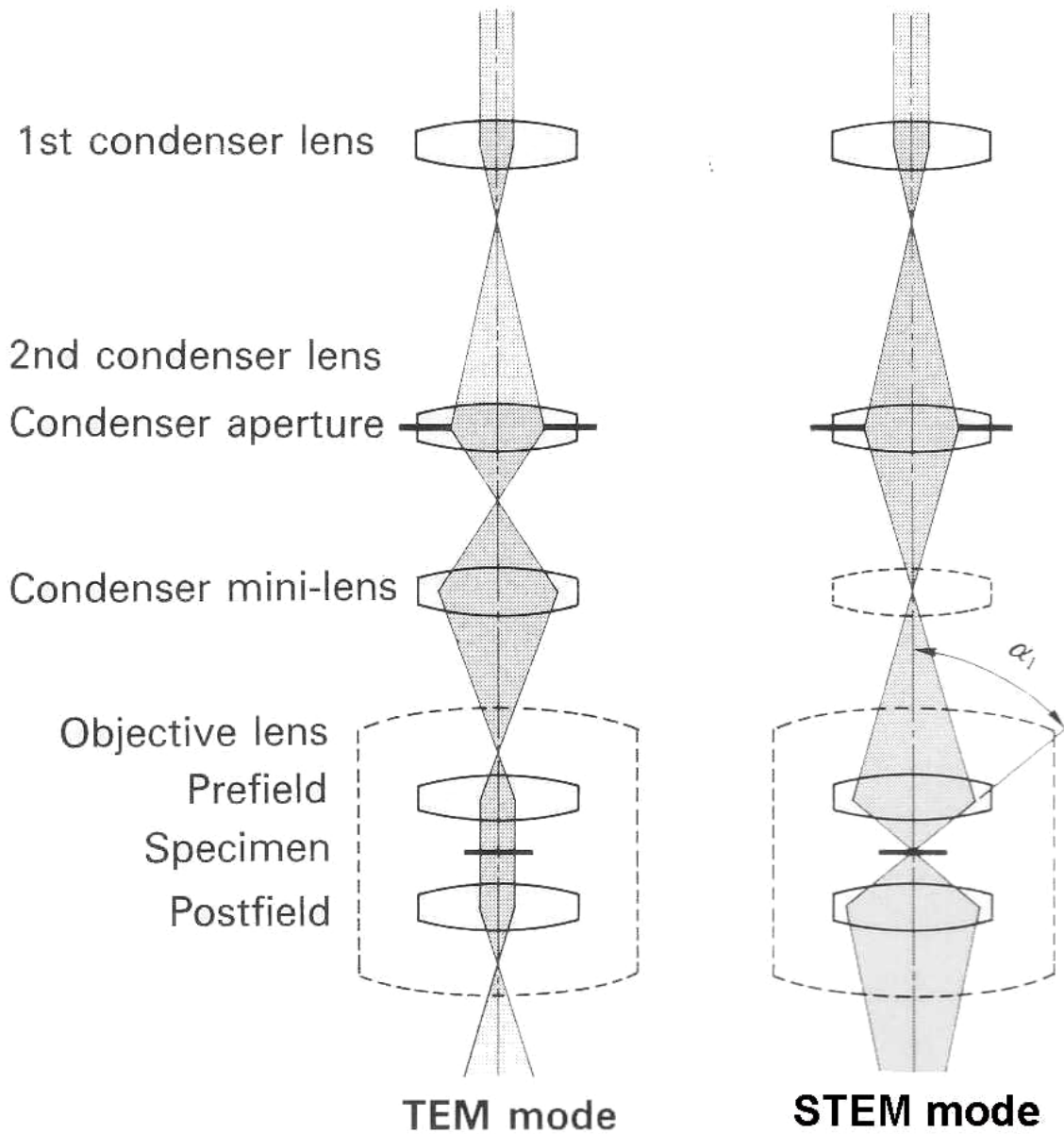


Conventional TEM



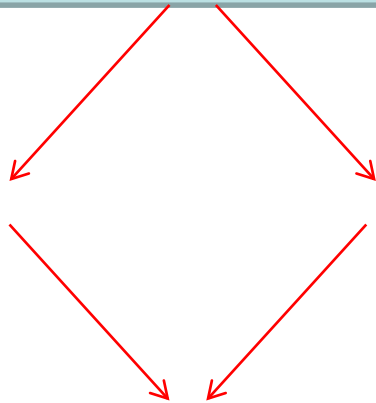
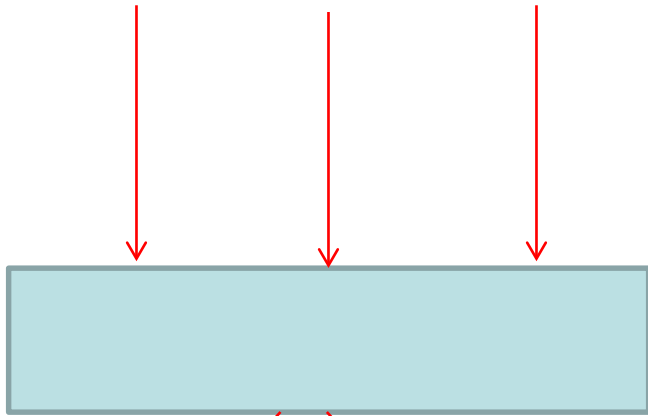
STEM





# Reciprocity

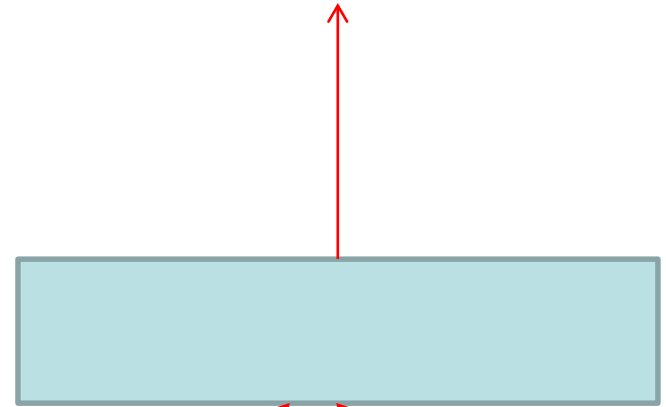
Source



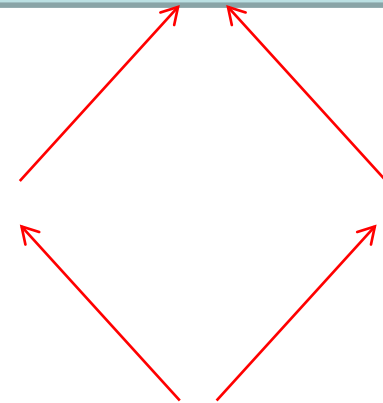
Detector

TEM Imaging

Detector



Reverse Time



Source

STEM Imaging

# STEM v TEM

- STEM = TEM, but with the optics reversed (if you ignore inelastic scattering)
- BF, DF, HREM can be done in a STEM almost the same as a TEM
- However:
  - The imaging is serial as against parallel, so in general has more noise and lower signals
  - Scan coils and electronics involved, which leads to noise
  - Some types of imaging can be done easier in a STEM than a TEM (they have all been done in both)
  - STEM needs a small source (demagnified further)
  - Some manufacturers have decided to reinvent the wheel

# Comparison

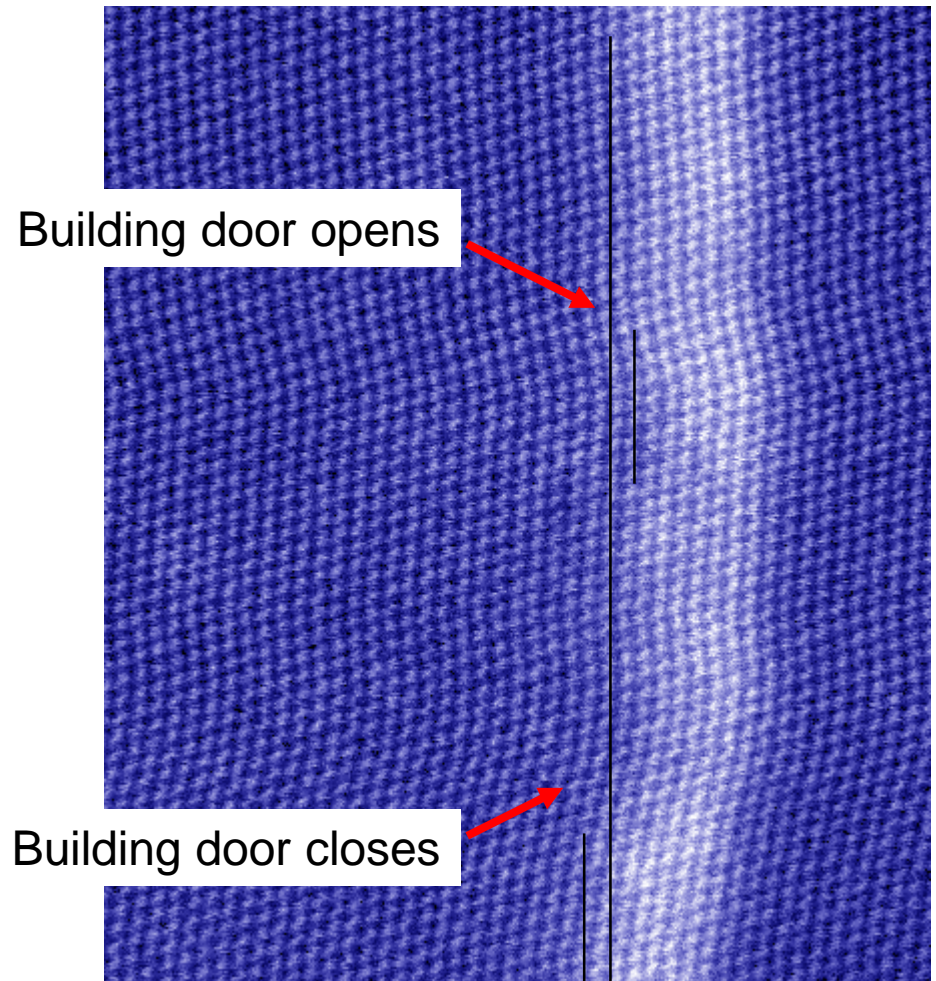
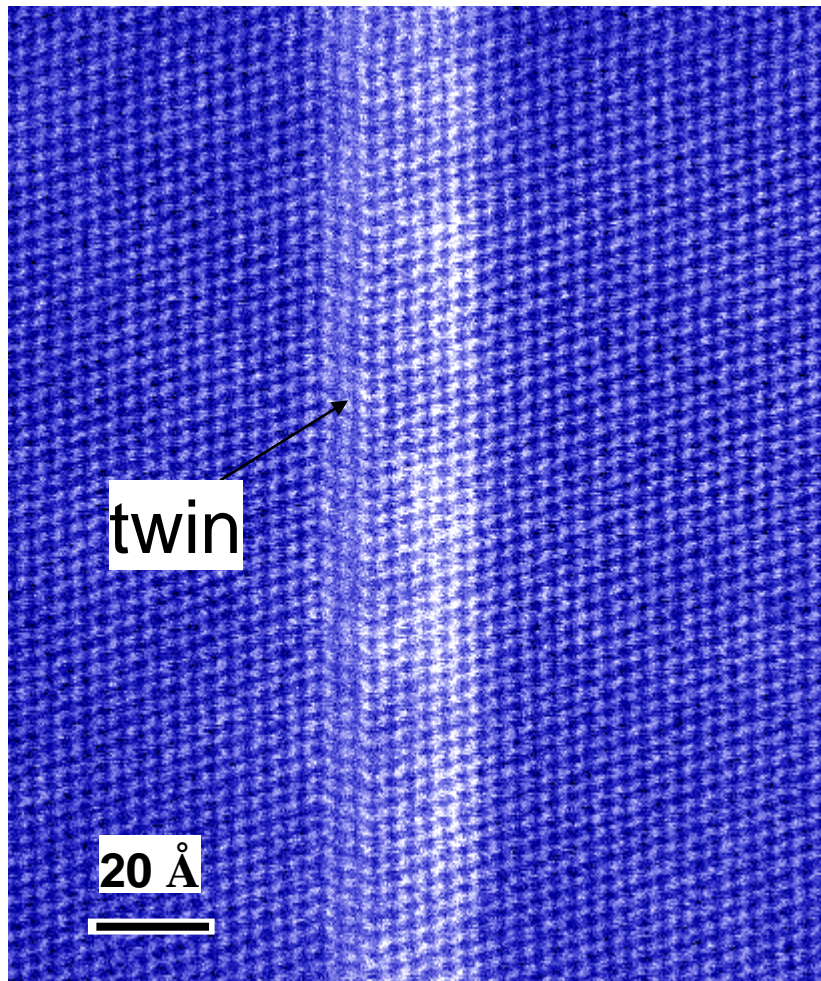
## TEM

- Condensor Aperture
- Objective aperture after sample
- No analogue
- Selected Area Aperture

## STEM

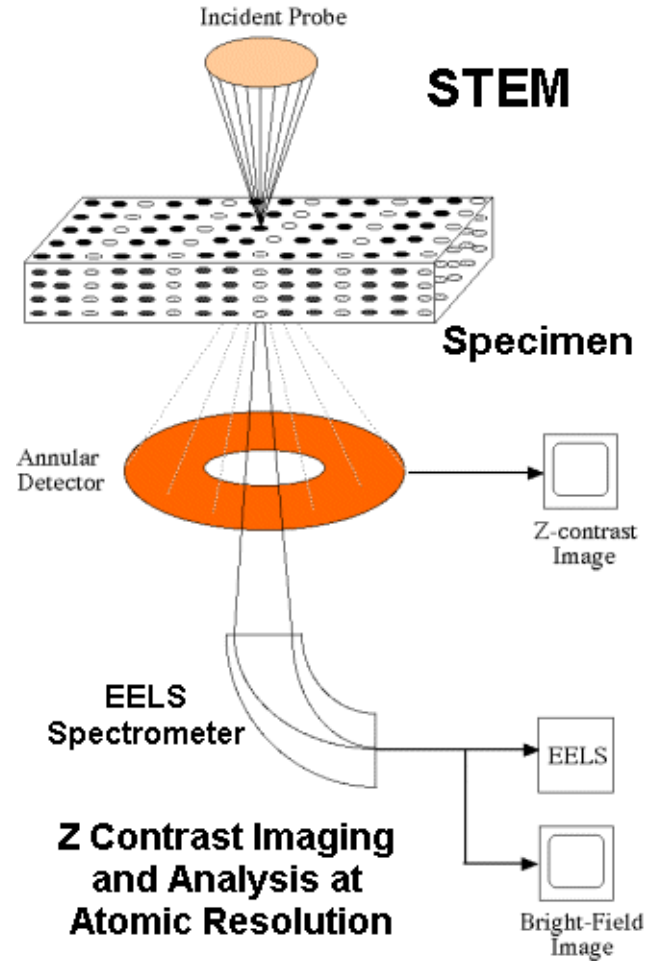
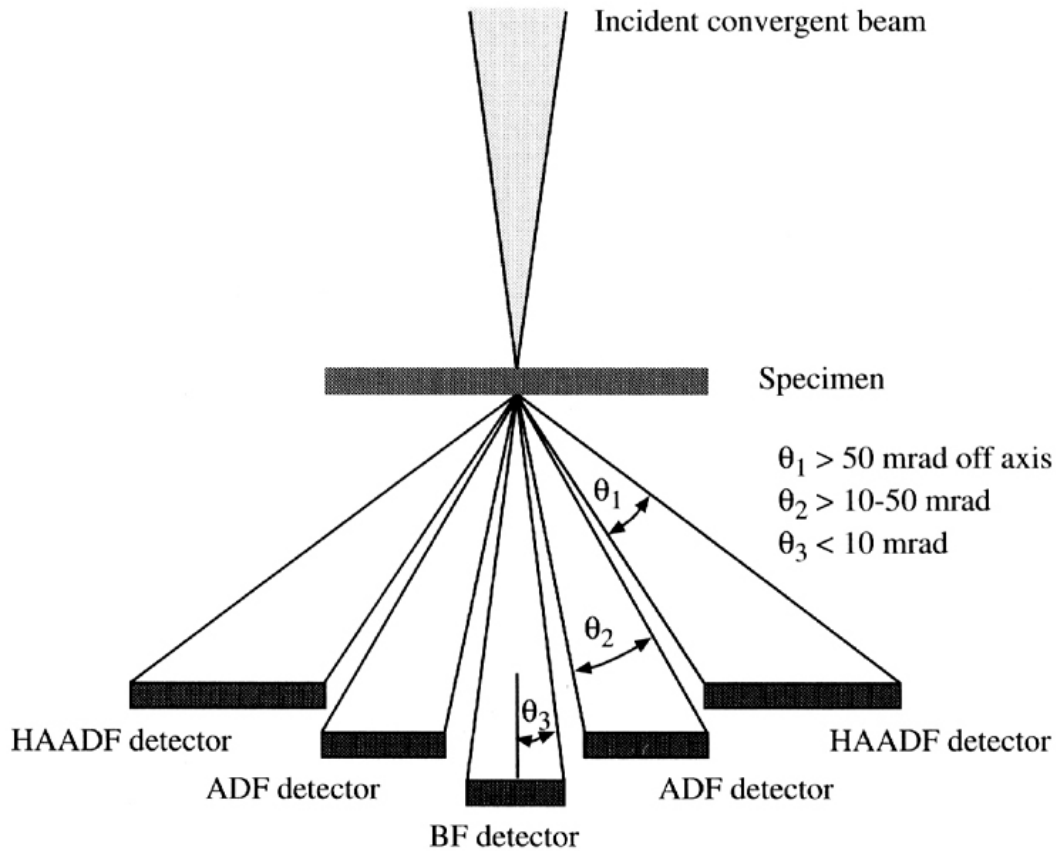
- Detector
- Objective aperture before sample
- Virtual objective aperture (condensor)
- No analogue

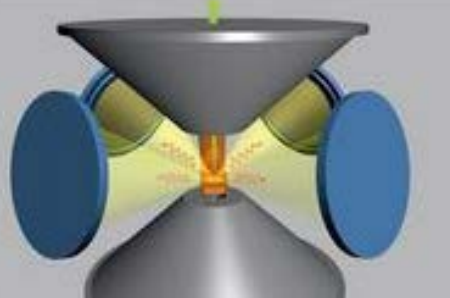
# Environmental Sensitivity



1 Pa change in air pressure  $\Rightarrow$  1 Å stage deflection

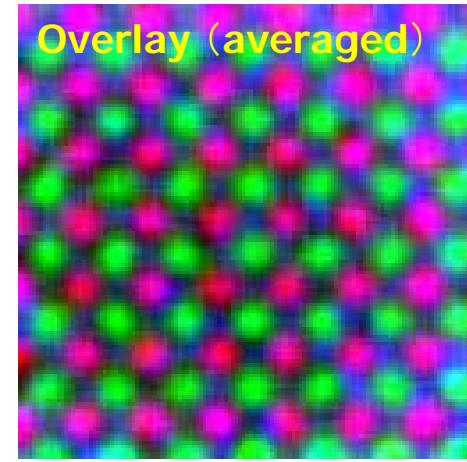
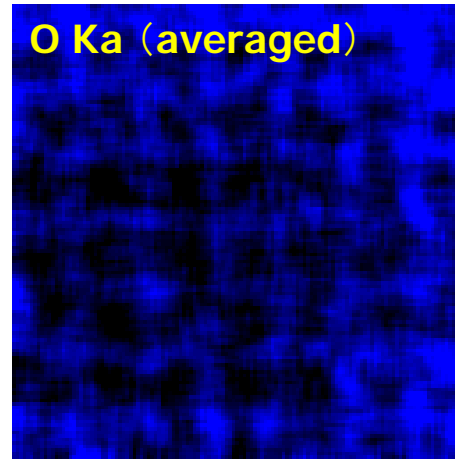
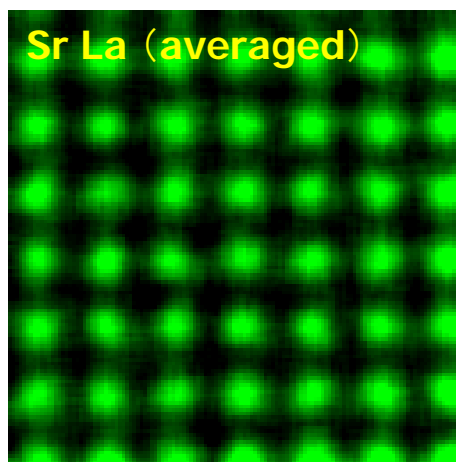
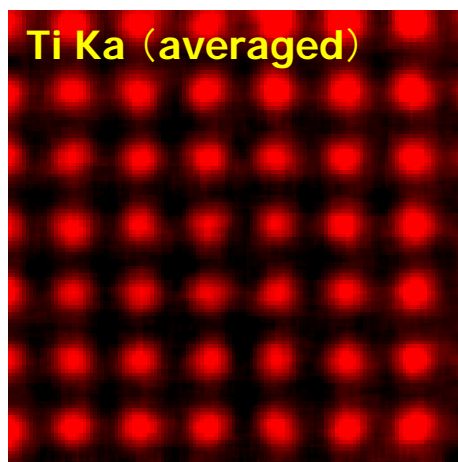
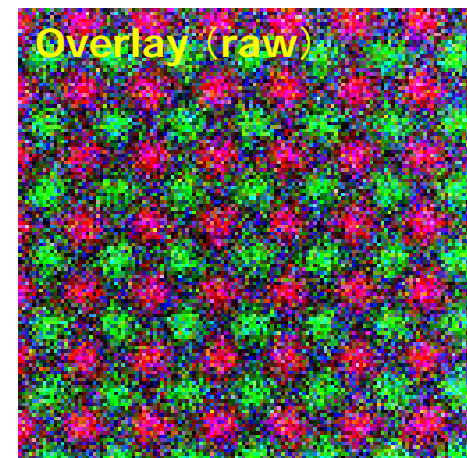
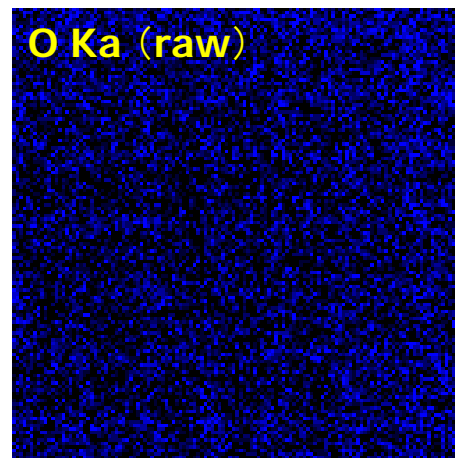
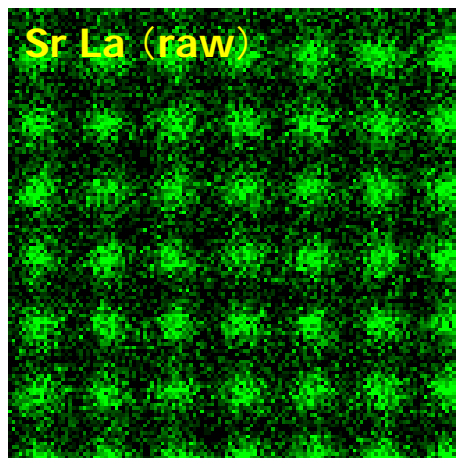
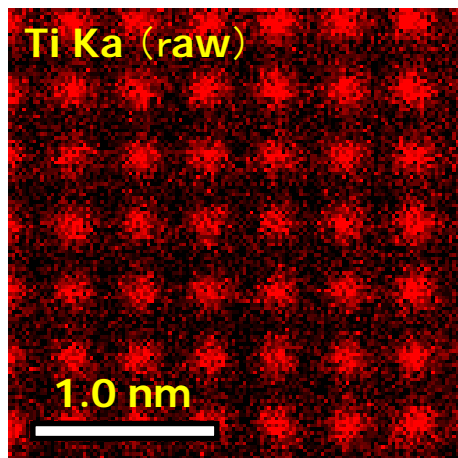
# More detectors are available!





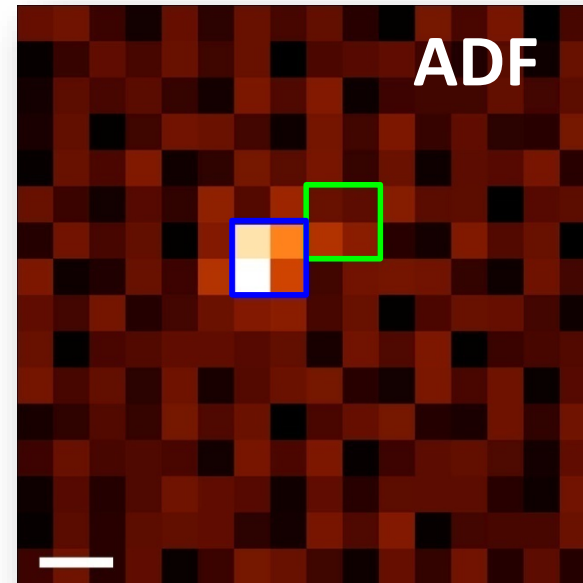
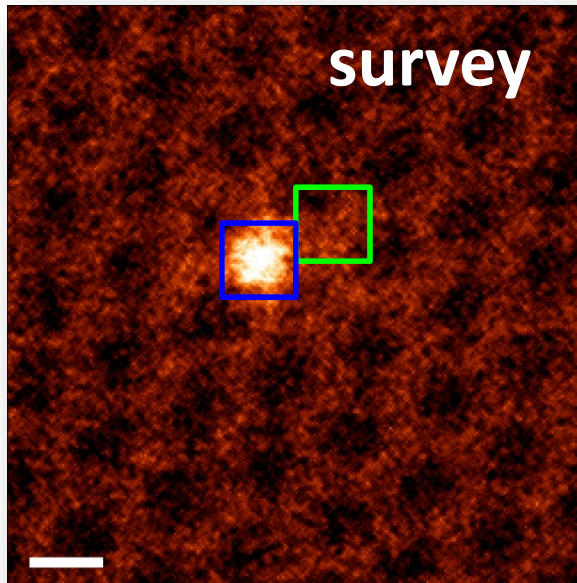
128x128, 175f, T4, 0.2ms  
RT: 9m33s, DT: 3.71%  
Count rate: 1119.57cps

# Atomic Resolution EDS

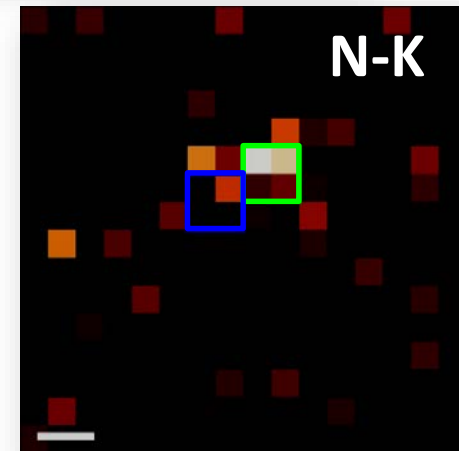
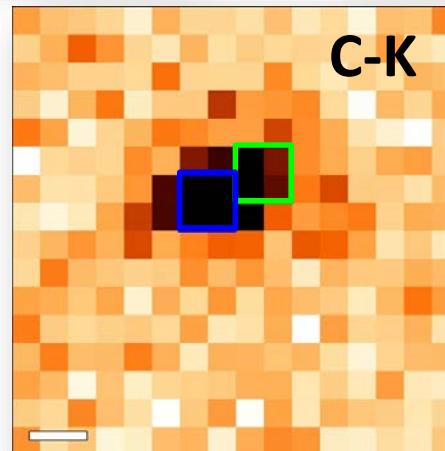
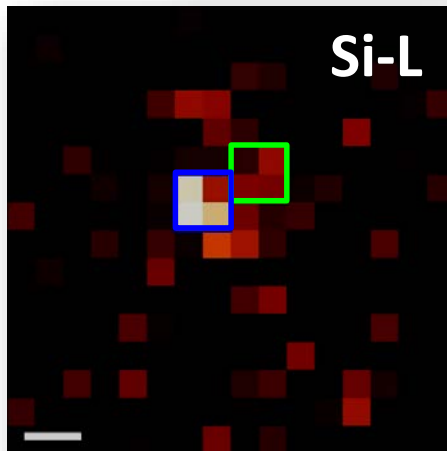




# Atom by atom spectroscopy



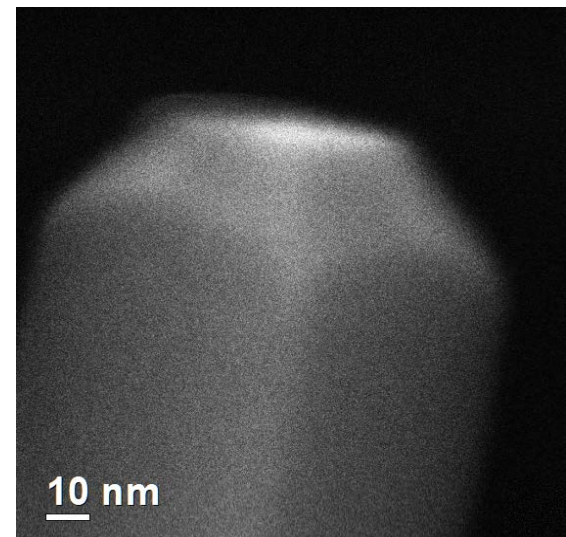
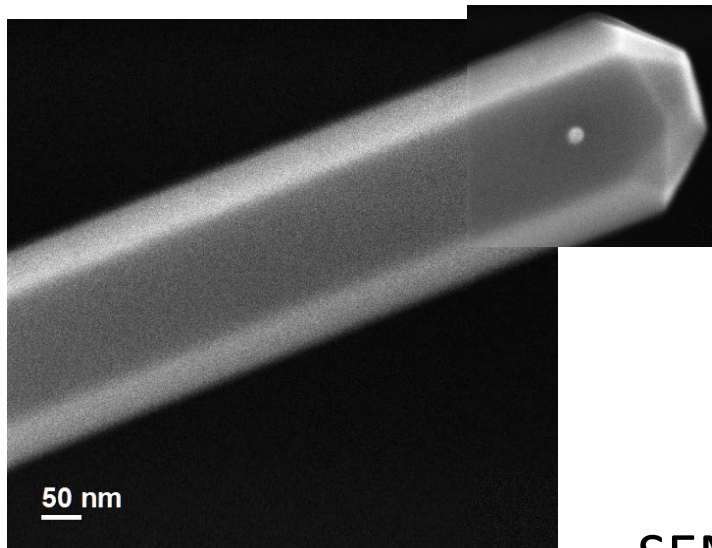
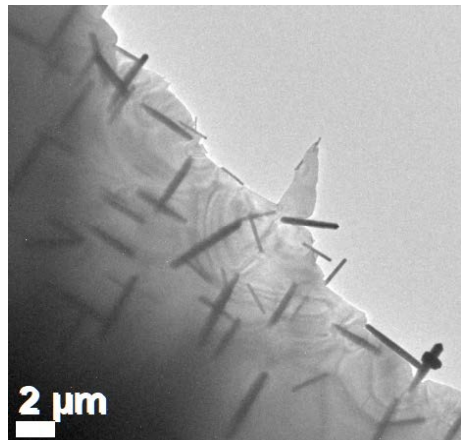
□ N  
□ Si



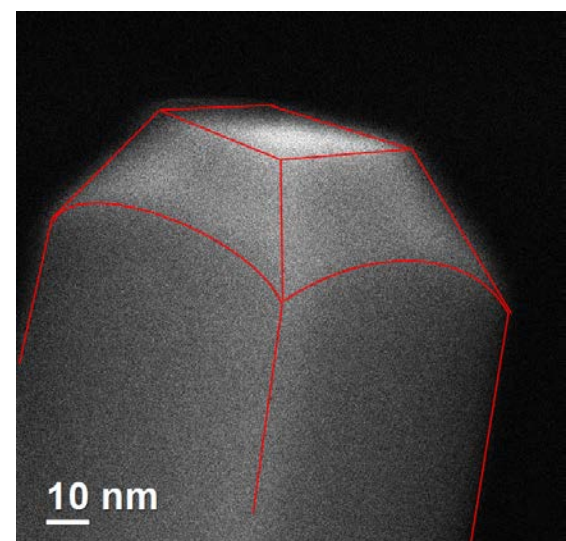
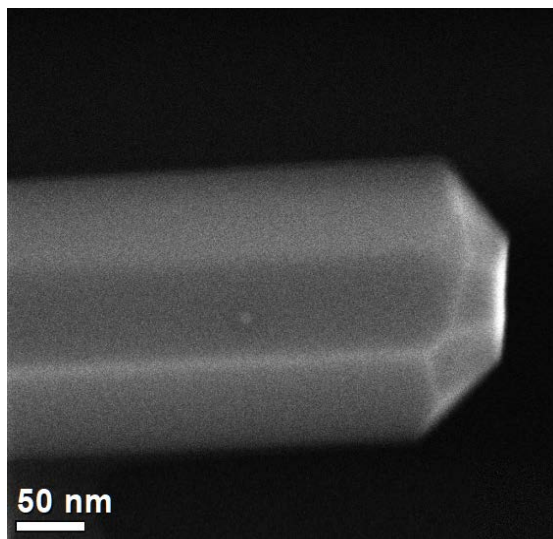
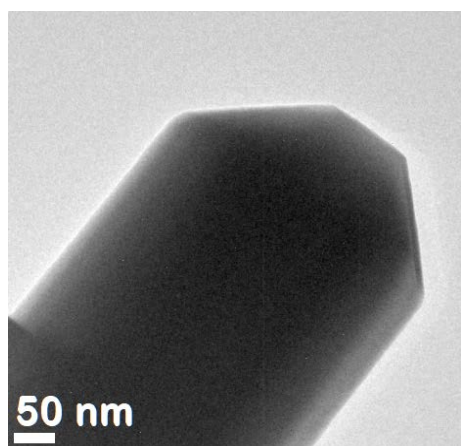
Zhou, W., Lee, J., Nanda, J., Pantelides, S. T., Pennycook, S. J., & Idrobo, J.-C. (2012). Atomically localized plasmon enhancement in monolayer graphene. *Nature nanotechnology*, 7(3), 161–165.

**Materials Science and  
Engineering and Scientific  
User Facilities Divisions**

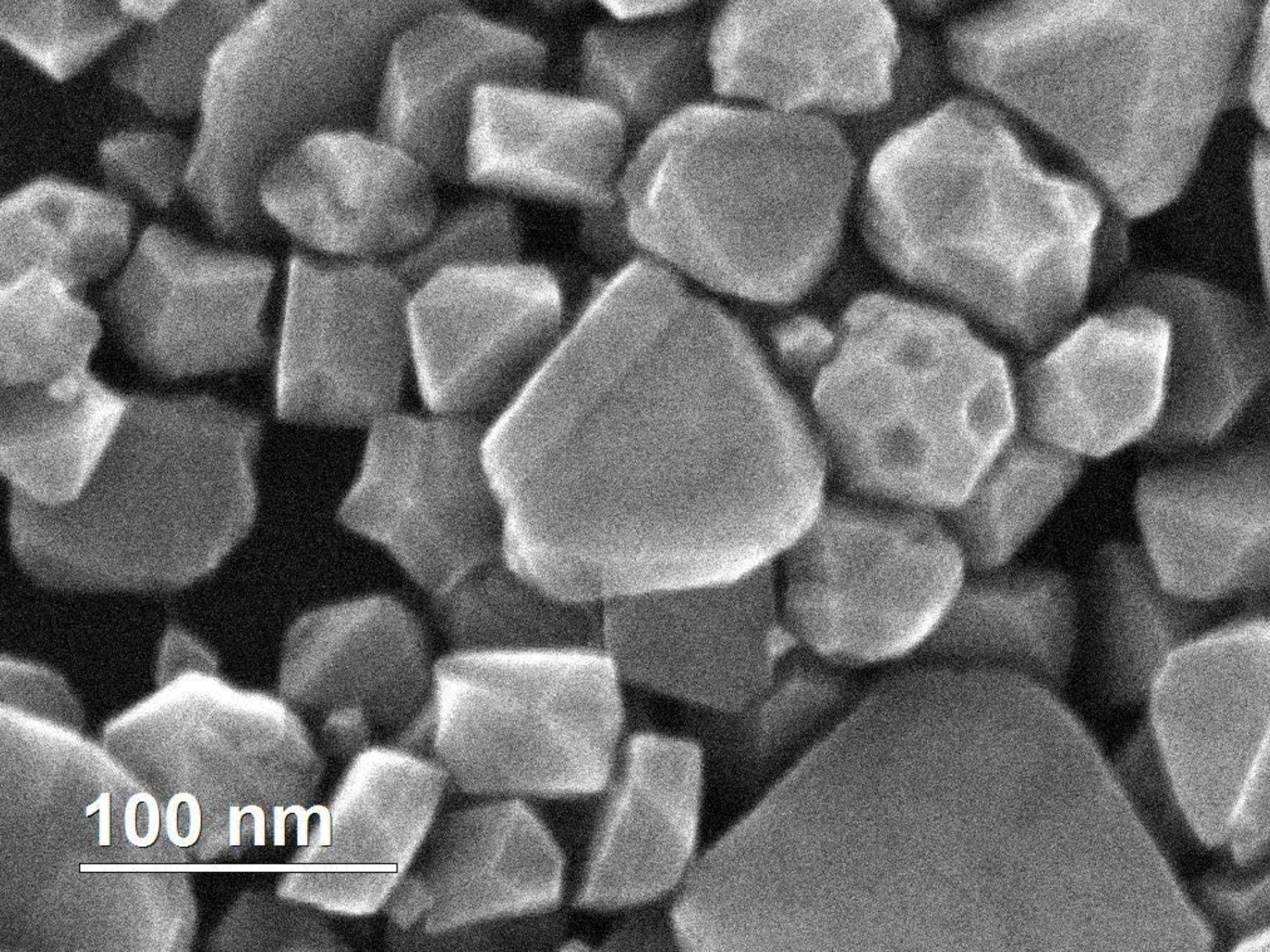
**Rods in  $\text{KTaO}_3$**



SEM



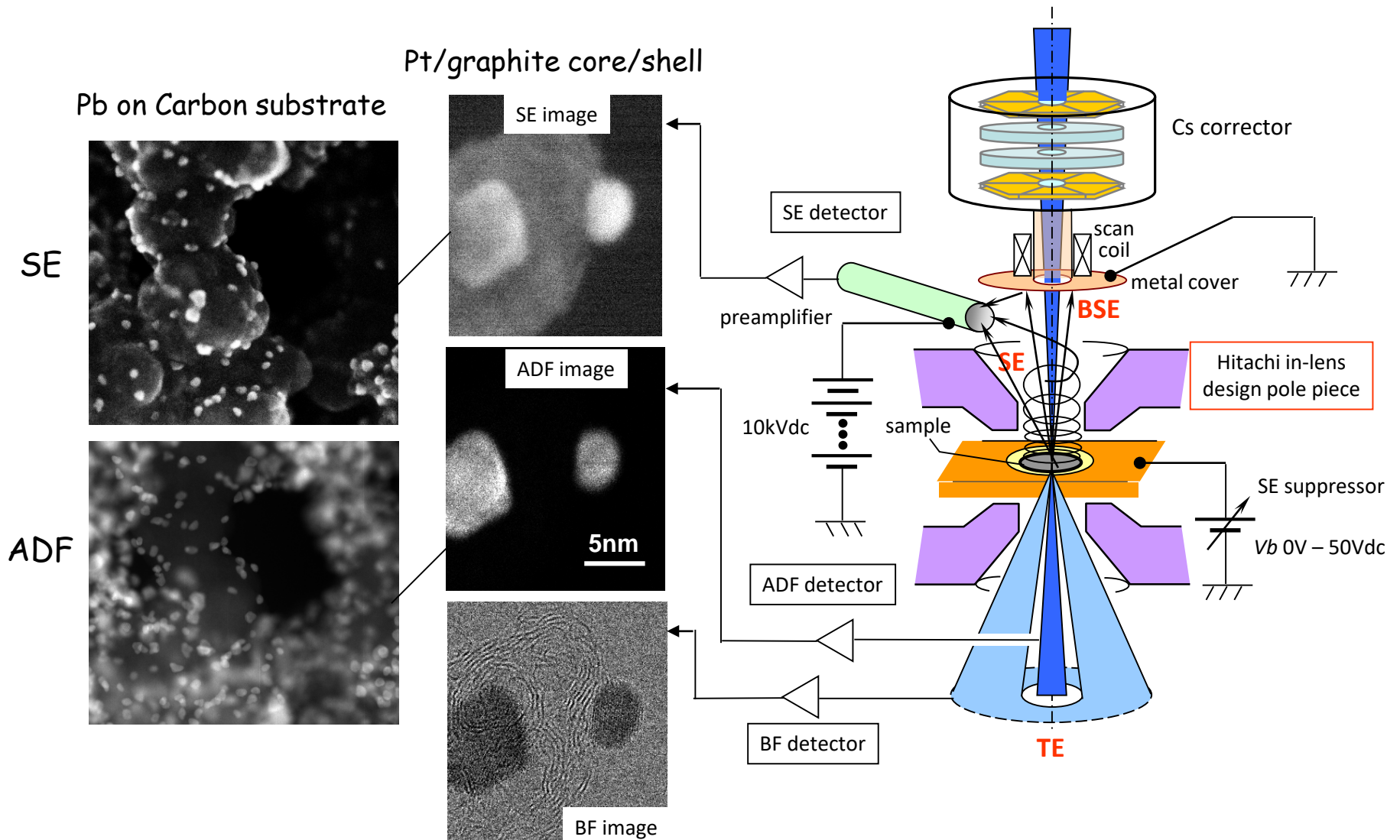
**Bright Field TEM**



100 nm



# Any STEM, just add a SE detector (Hitachi, JEOL...)





nature materials

LETTERS

PUBLISHED ONLINE: 20 SEPTEMBER 2009 | DOI: 10.1038/NMAT2532

# Imaging single atoms using secondary electrons with an aberration-corrected electron microscope

Y. Zhu<sup>1\*</sup>, H. Inada<sup>2</sup>, K. Nakamura<sup>2</sup> and J. Wall<sup>1</sup>

Aberration correction has embarked on a new frontier in electron microscopy by overcoming the limitations of conventional round lenses, providing sub-angstrom-sized probes<sup>1-4</sup>. However, improvement of spatial resolution using aberration correction so far has been limited to the use of transmitted electrons both in scanning and stationary mode, with an improvement of 20-40% (refs 3-8). In contrast, advances in the spatial resolution of scanning electron microscopes (SEMs), which are by far the most widely used instrument for surface imaging at the micrometre-nanometre scale<sup>9</sup>, have been

about the particles' locations, much of which is lacking in the transmission image. In the past decade or so, high-resolution SEM has proven an indispensable critical-dimension-metrology tool for the semiconductor industry. The semiconductor nanotechnology road map identifies the need for ultrahigh-resolution SEM in the quest for ever-decreasing device sizes<sup>11</sup>.

We attempted to achieve the highest possible SEM resolution and to determine whether it is limited by the basic physics of secondary production or by the instrumentation. We explored well-defined samples (single uranium atoms) in an instrument

news & views

SCANNING ELECTRON MICROSCOPY

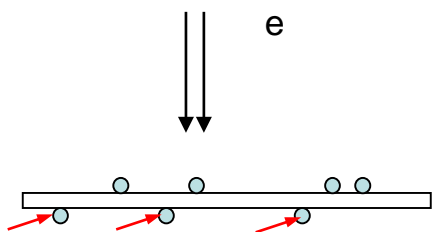
## Second best no more

Secondary electron imaging in electron microscopy can achieve resolutions that compete with transmission electron microscopy, and allows imaging of both surface and bulk atoms simultaneously.

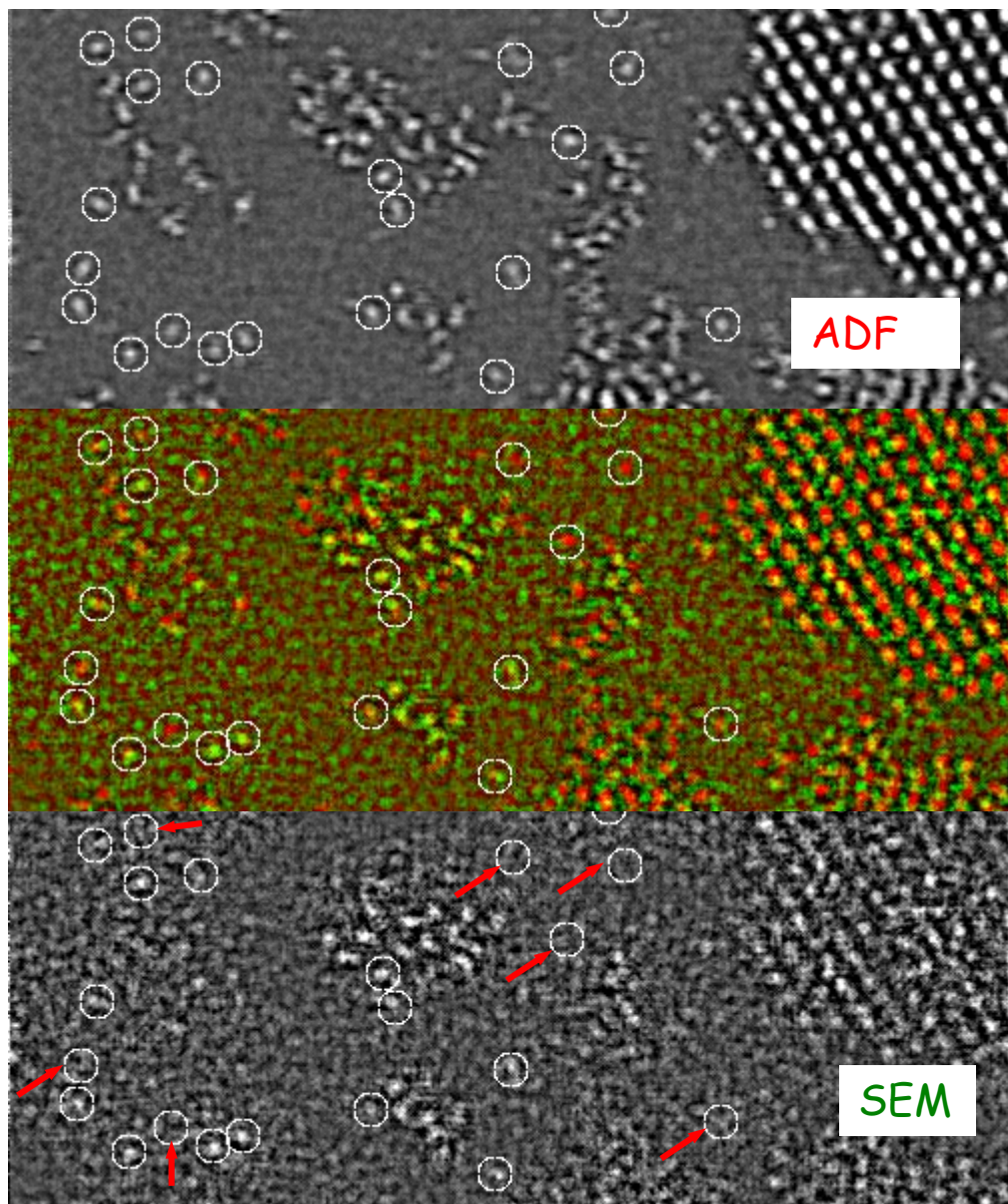
David C. Joy

Secondary electron imaging is the most popular mode of operation of the scanning electron microscope (SEM).

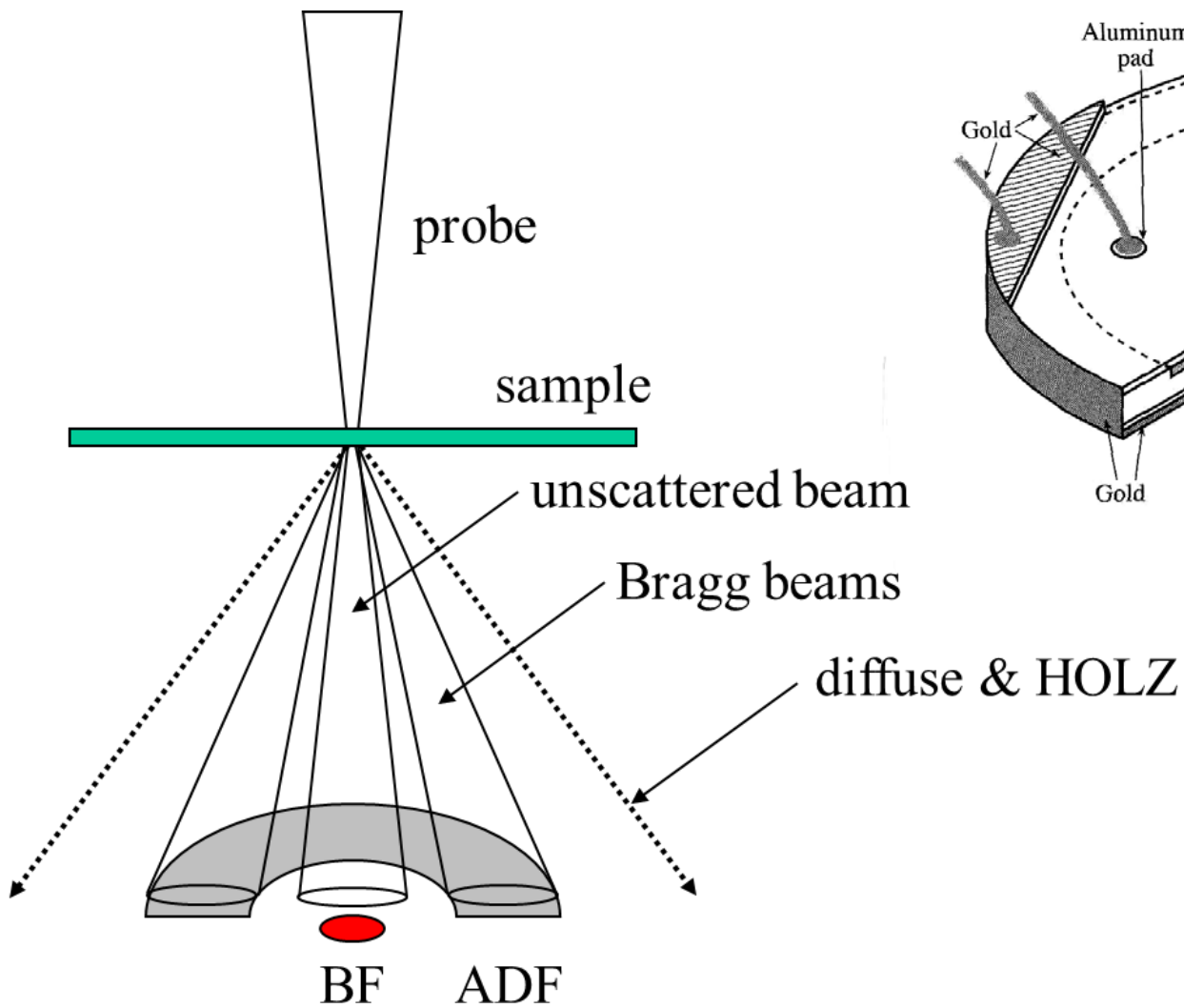
Figure 1 shows a secondary electron image of interconnect lines in a semiconductor device. Each is seen to be outlined by



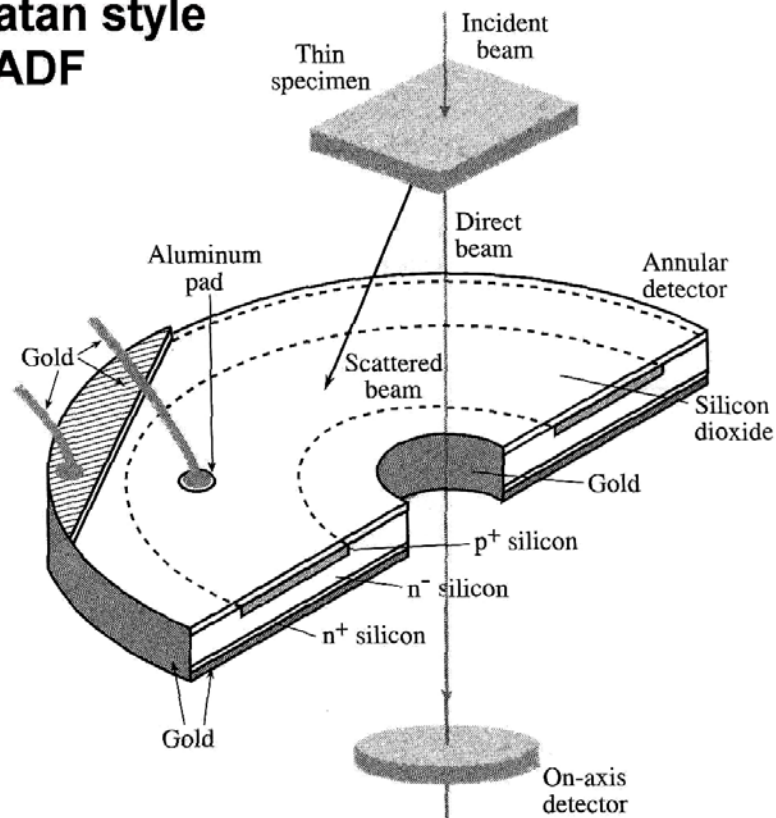
## Imaging surface U atoms



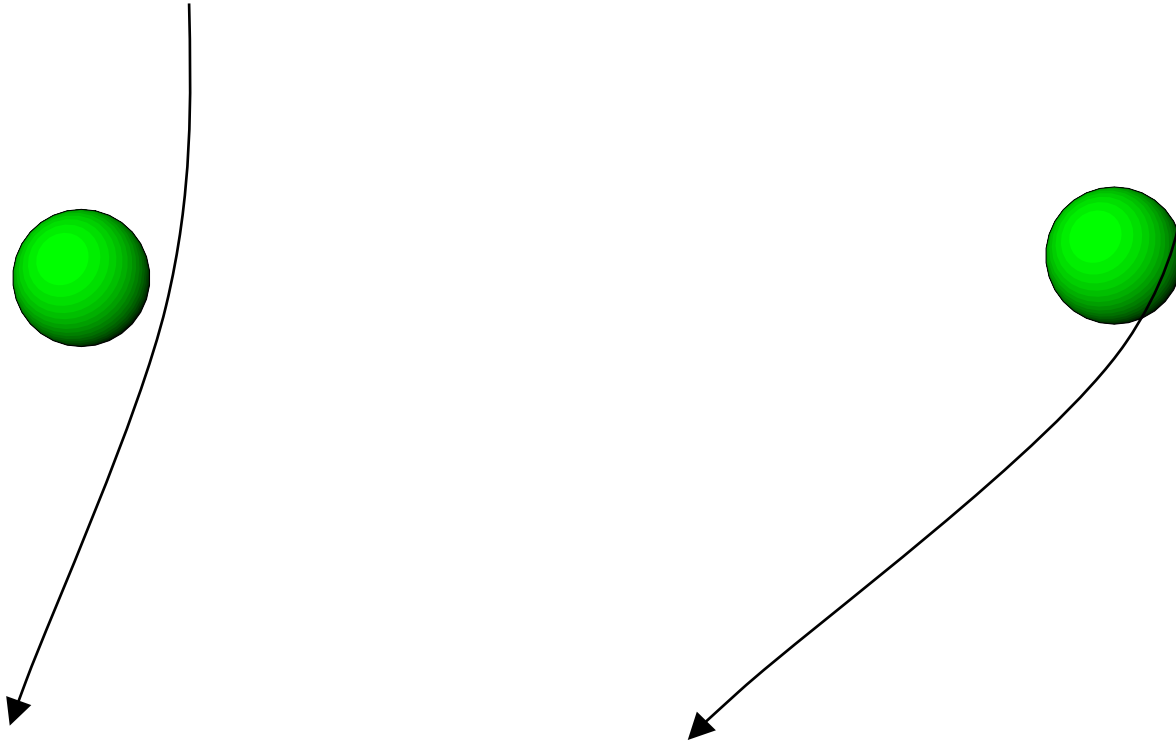
Nature Materials, 8, 808 - 812 (2009)



## Gatan style HADF



# HAADF

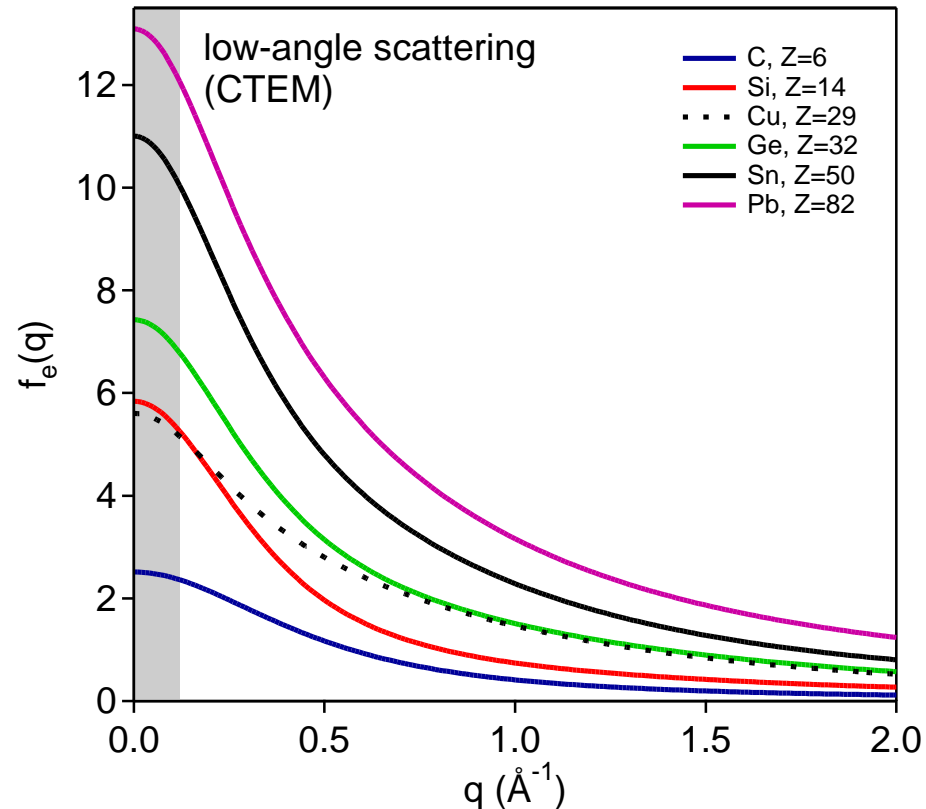


The closer the electron comes to the nucleus, the higher the probability of high-angle scattering (elastic or inelastic)

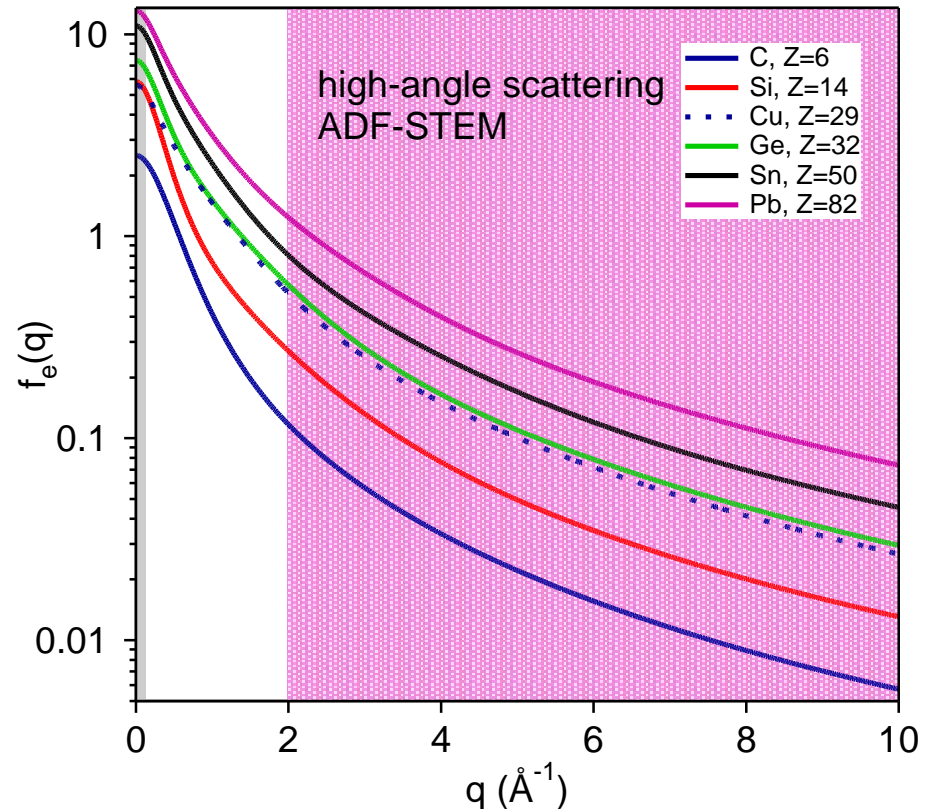
1s states dominate the high-angle scattering

# Partitioning Signal in Scattering Space

Scattering from one atom is proportional to scattering factor  $f_e(q)$ .



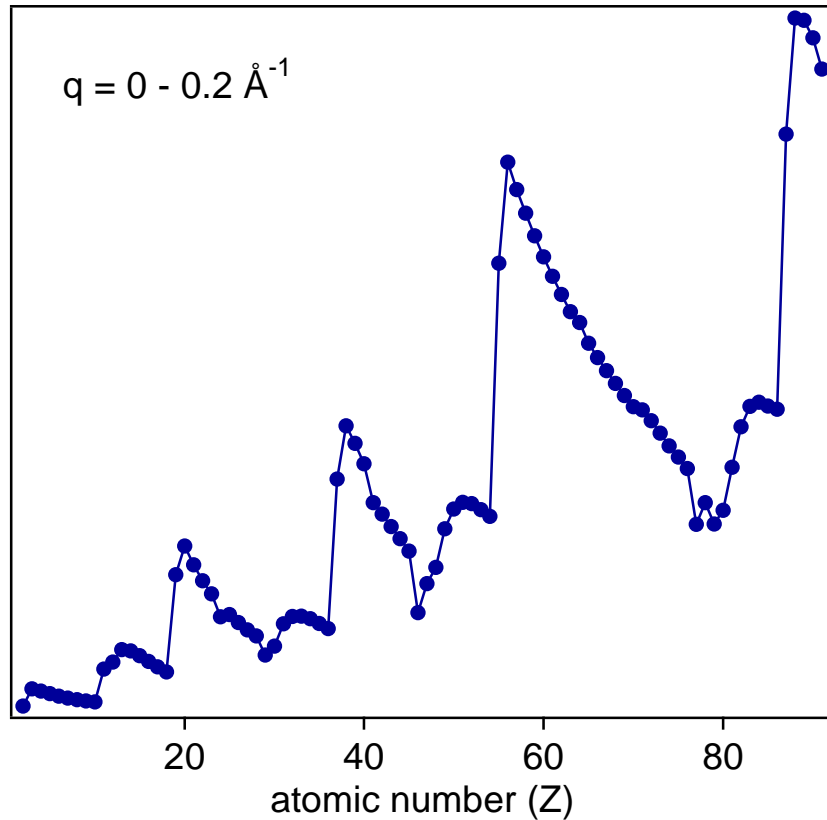
BF CTEM involves mostly low-angle scattering: Cu & Si cross.



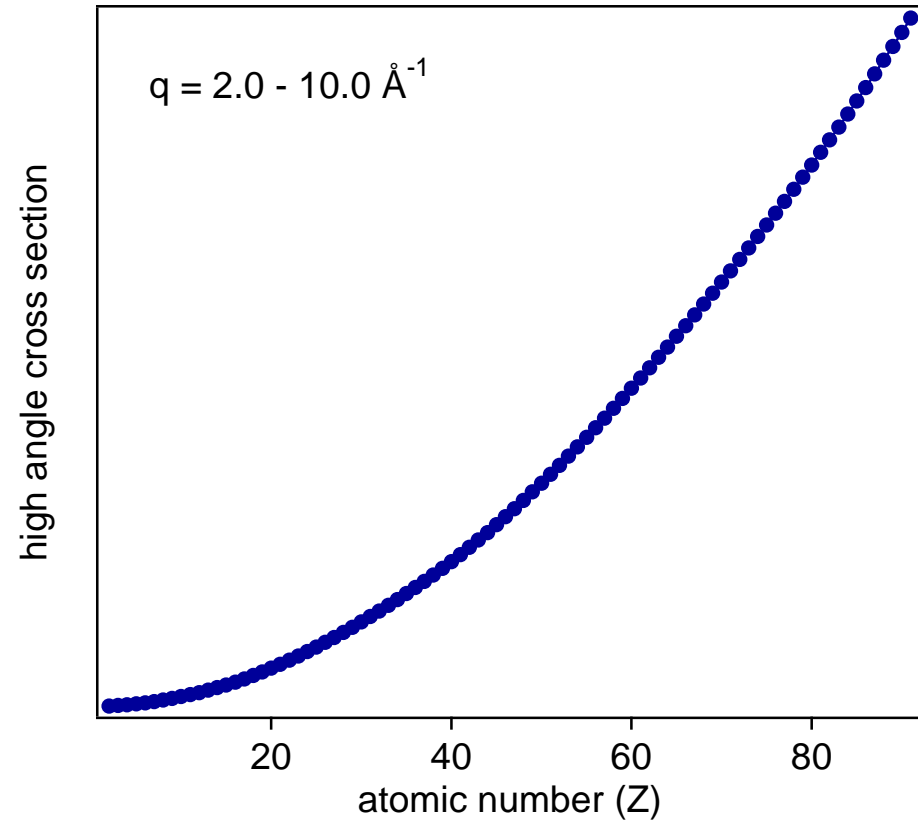
ADF-STEM involves high-angle scattering only.



# Partitioning Signal in Scattering Space



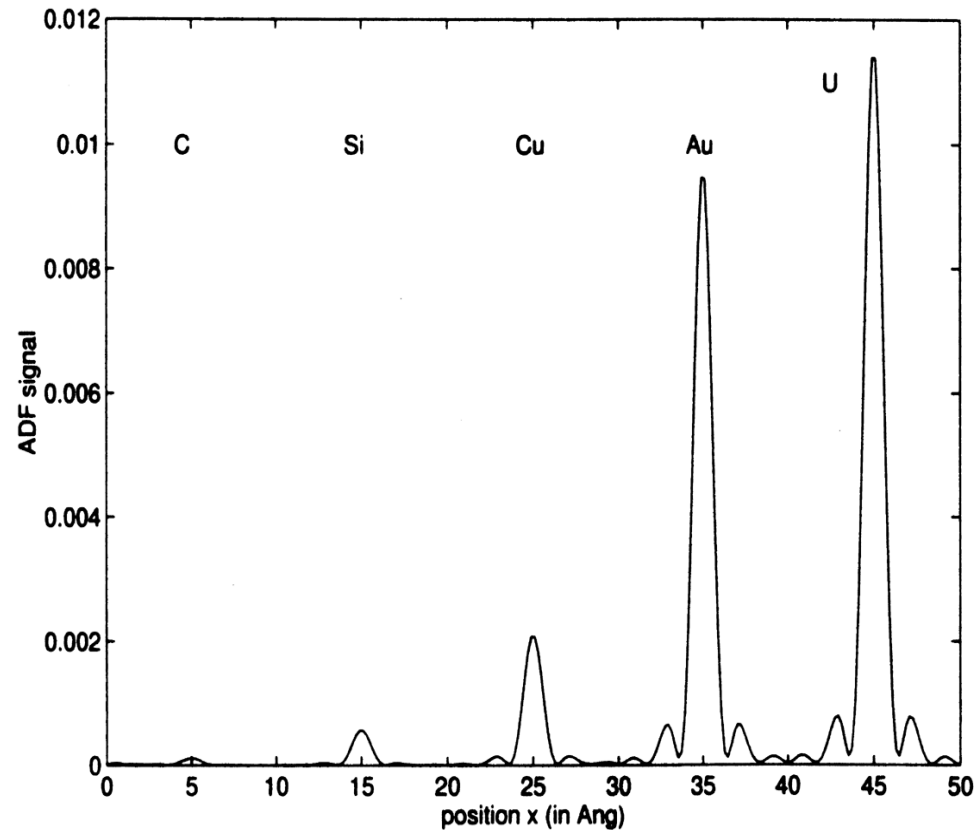
sensitive to electron shell filling, complicated (also diffraction & channelling, later)



monotonic, proportional to  $Z^2$   
Rutherford “billiard ball” scattering

# Z-contrast

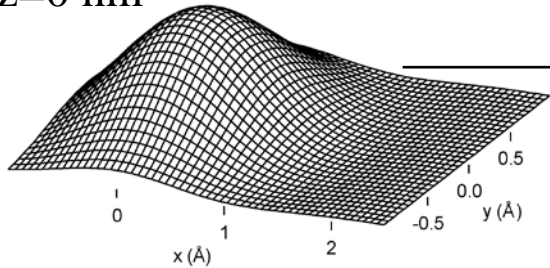
- Scattering scales as  $\sim Z^{1.7}$  for common scattering angles
- Images are readily interpretable
- Images contain some chemical information
- Many problems with this simple picture (good enough for Govt work)



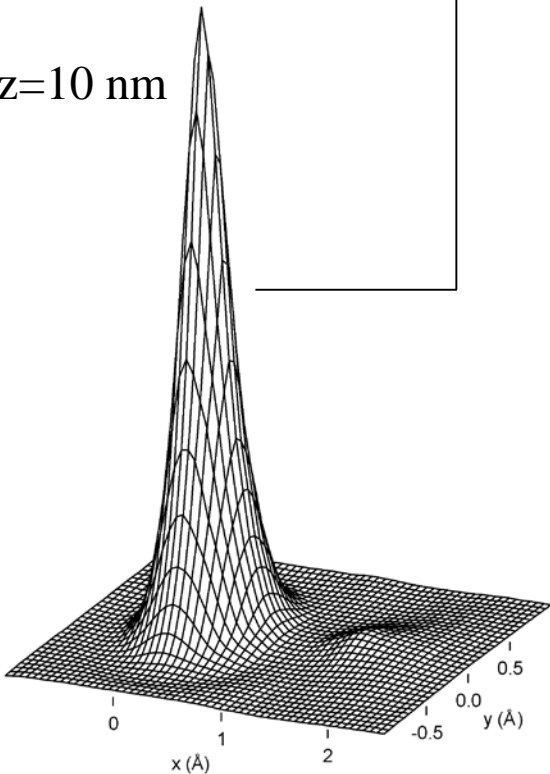
E. J. Kirkland, *Advanced Computing in Electron Microscopy* (Plenum, 1998).

# Probe Channeling

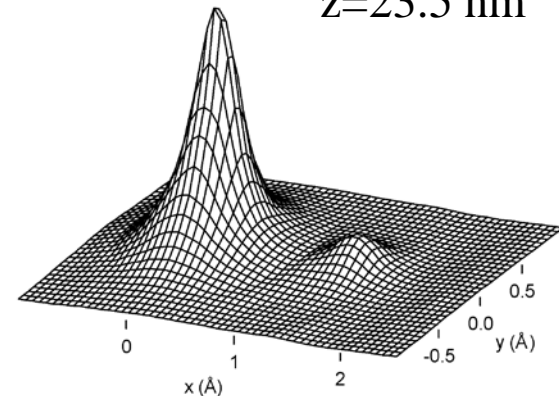
$z=0$  nm



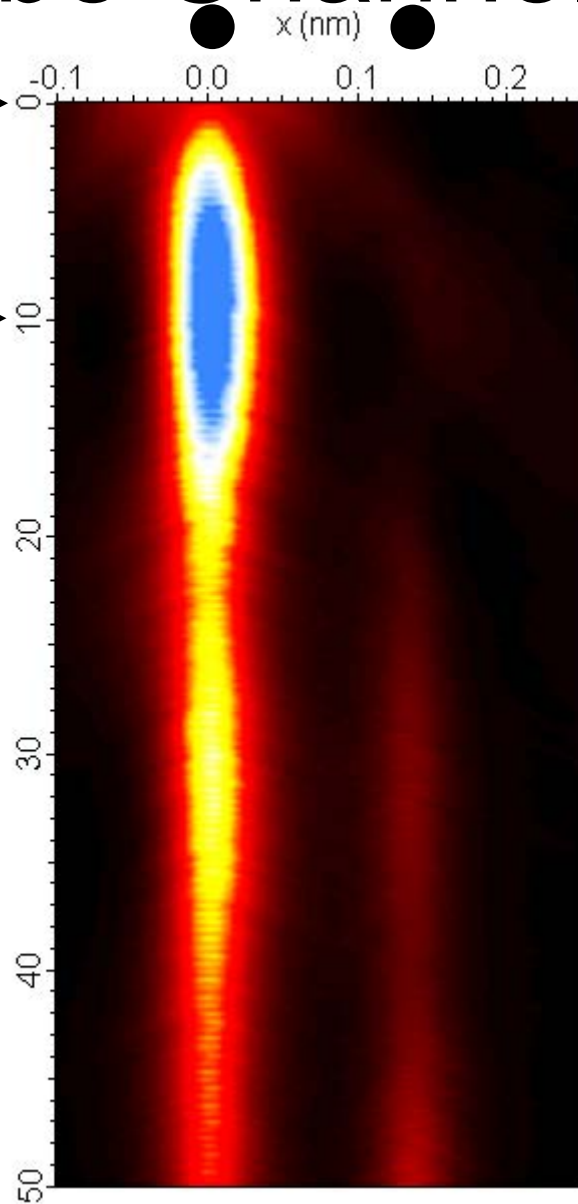
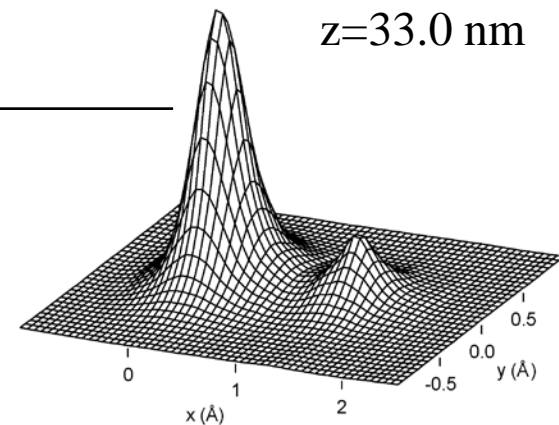
$z=10$  nm



$z=23.5$  nm



$z=33.0$  nm



# Caveat

- Consider via Fermi's Golden Rule

$$\text{Signal} = \rho(r) |\langle f|V|i\rangle|^2$$

- “Z-contrast” only corresponds to the  $|\langle f|V|i\rangle|^2$  contribution (HOLZ, phonons)
- But  $\rho(r)$  comes from dynamical diffraction
- True Z-contrast occurs iff  $\rho(r)$  has a simple form, which it does in special cases

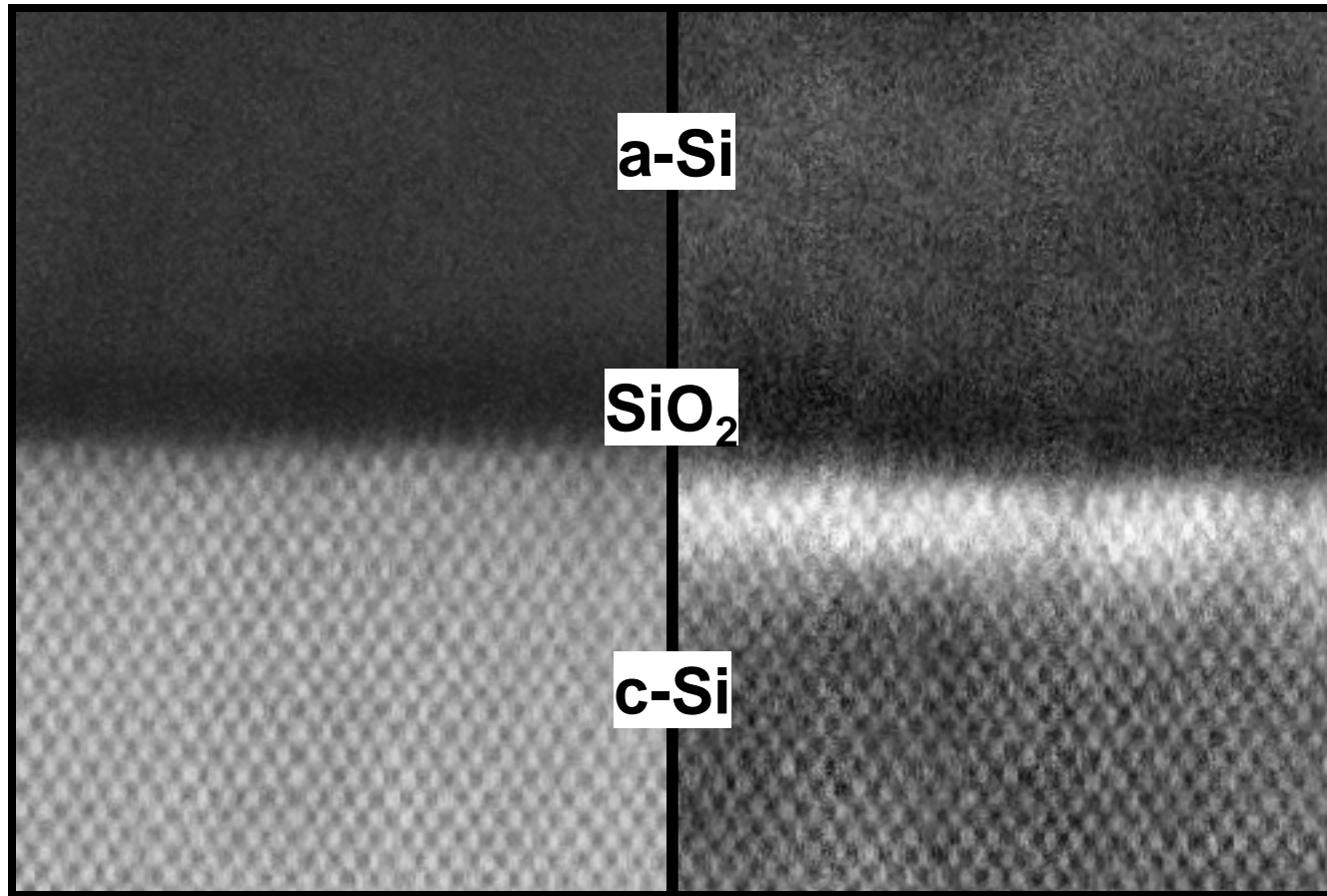
# Strain Contrast at Si/SiO<sub>2</sub> Interfaces

(JEOL 2010F, 200 kV, C<sub>s</sub>=1mm)

ADF Inner angle:

50 mrad

25 mrad



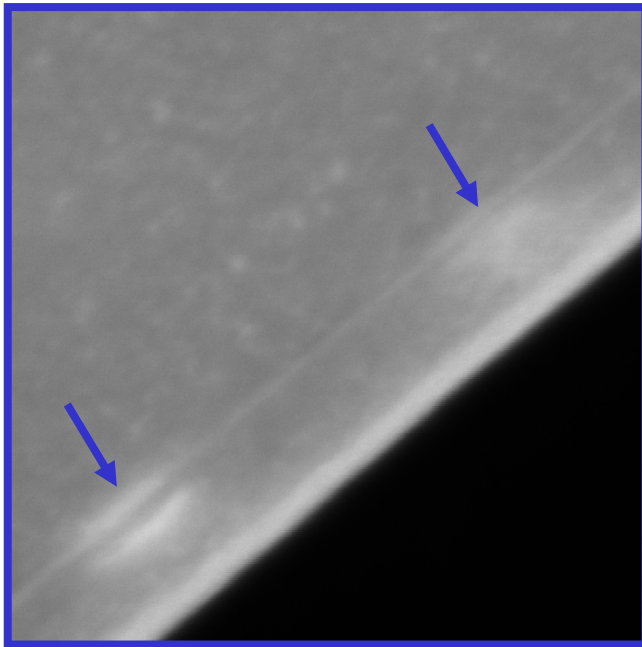
Strain Fields cause dechanneling (and scattering to small angles)

Z. Yu, D. A. Muller, and J. Silcox, *J. Appl. Phys.* **95**, 3362 (2004).

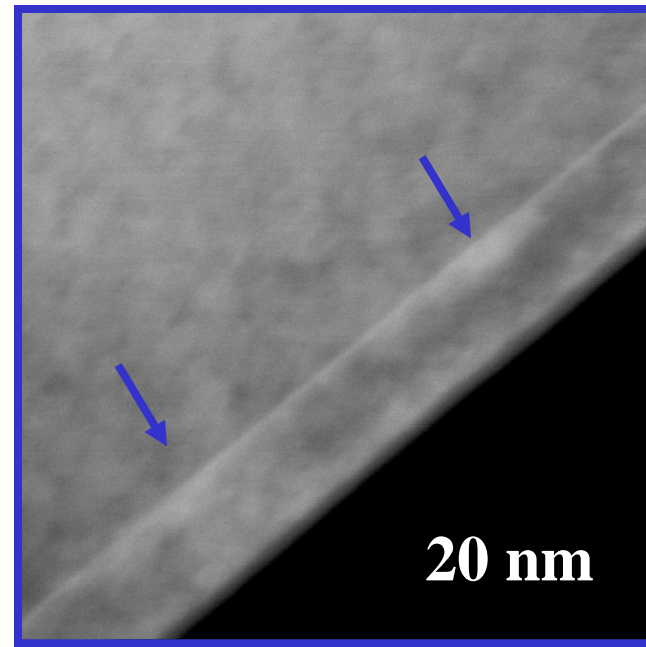
# Comparison between High-Angle and Low-Angle Annular Dark-Field Image

---

*Cross-sectional sample through InGaAs quantum dots and associated quantum well grown on GaAs and capped with GaAs. Dots are arrowed.*



*Low-angle annular dark field showing strain contrast*



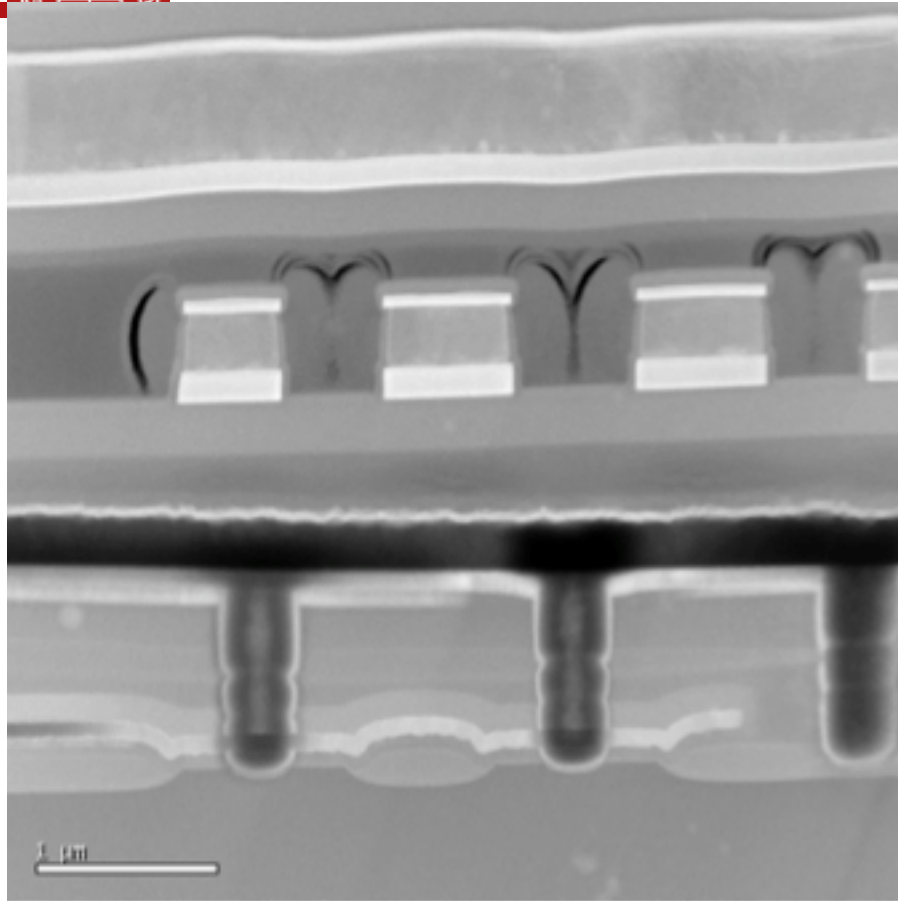
*High-angle annular dark field showing mostly Z contrast*

ASU Winter School 2013

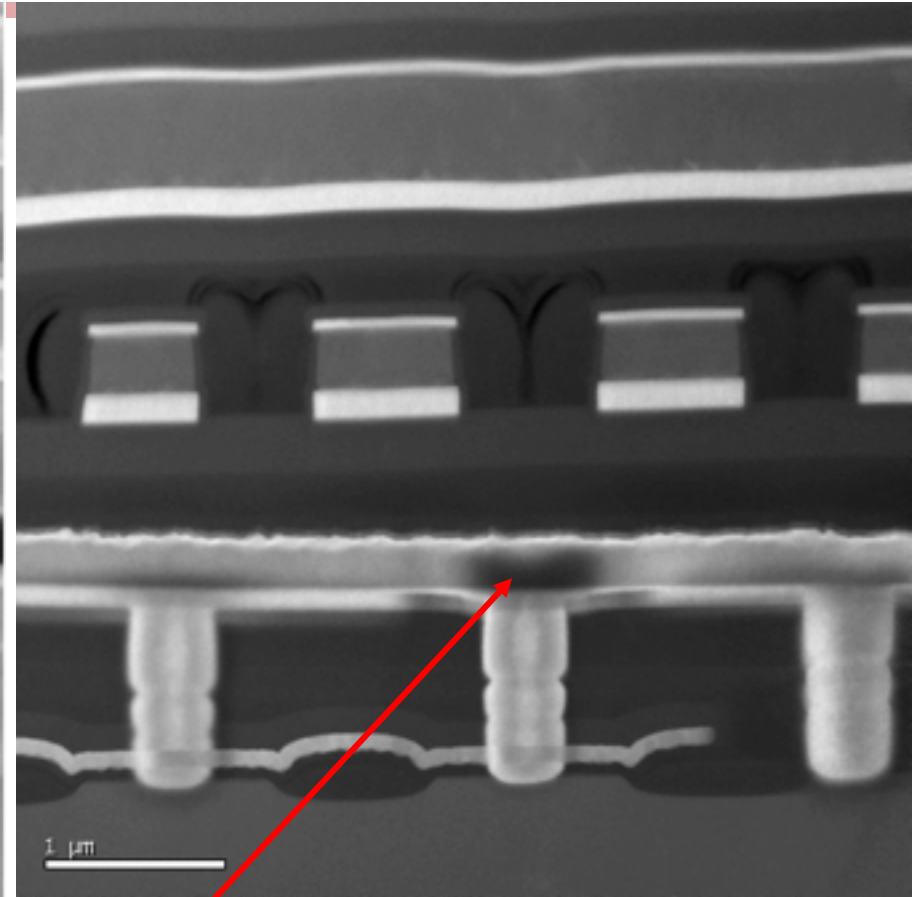
# Contrast Reversals in Thick Samples at 200kV



ADF-STEM ( $\phi_c > 45$  mr)



ADF-STEM ( $\phi_c > 75$  mr)



- No more diffraction contrast
- Signal in W plug not monotonic, could be mistaken for voids
- Effect reduced by increasing the collector angle

# Strain Contrast at Si/SiO<sub>2</sub> Interfaces

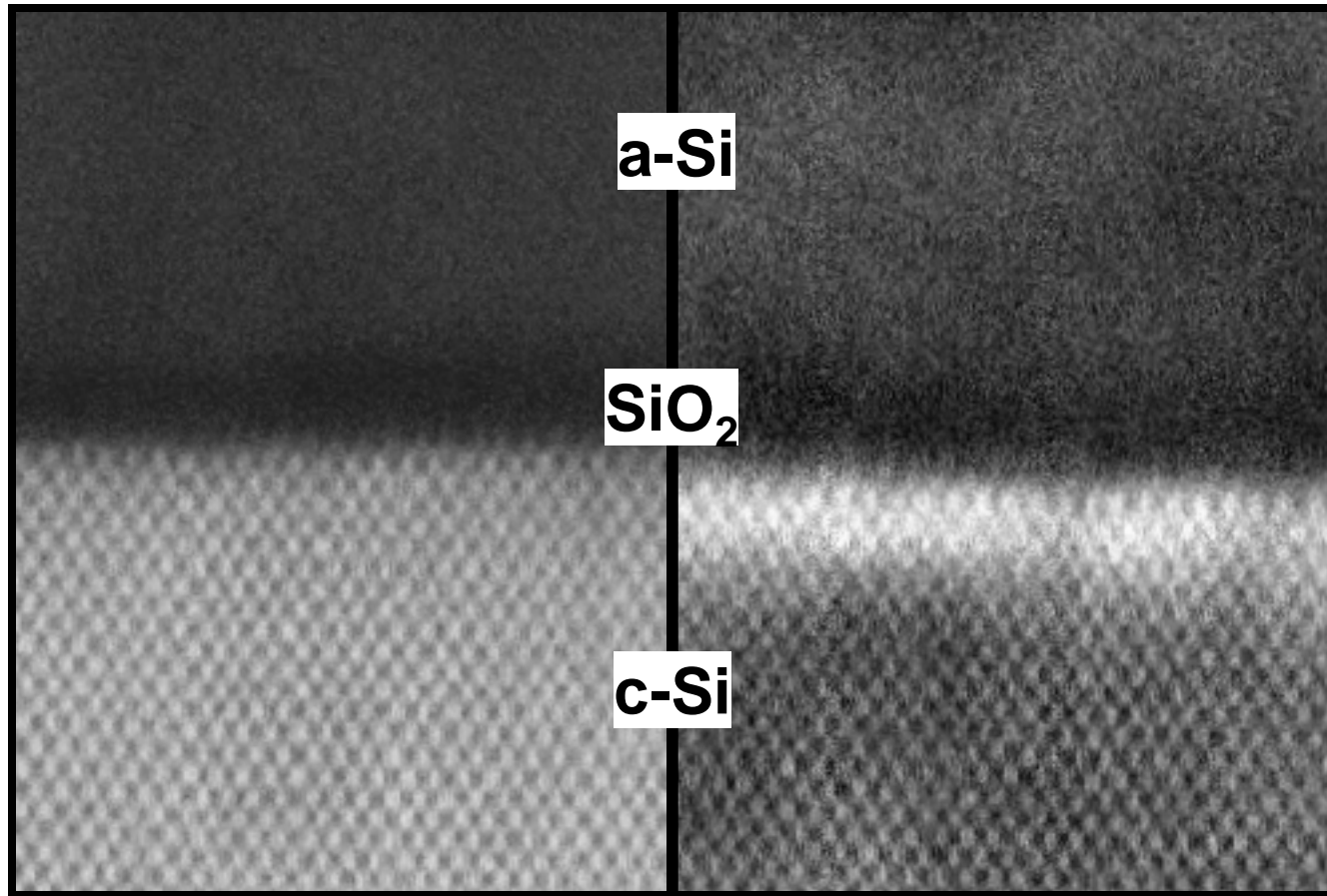
(JEOL 2010F, 200 kV, C<sub>s</sub>=1mm)

---

ADF Inner angle:

50 mrad

25 mrad



Strain Fields cause dechanneling (and scattering to small angles)

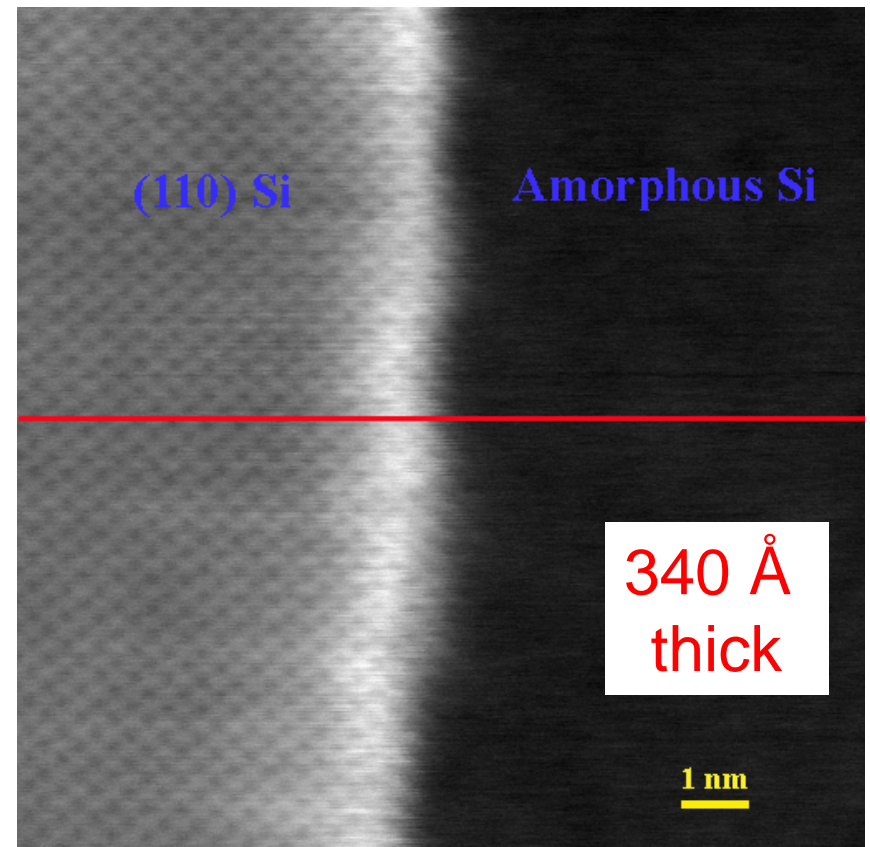
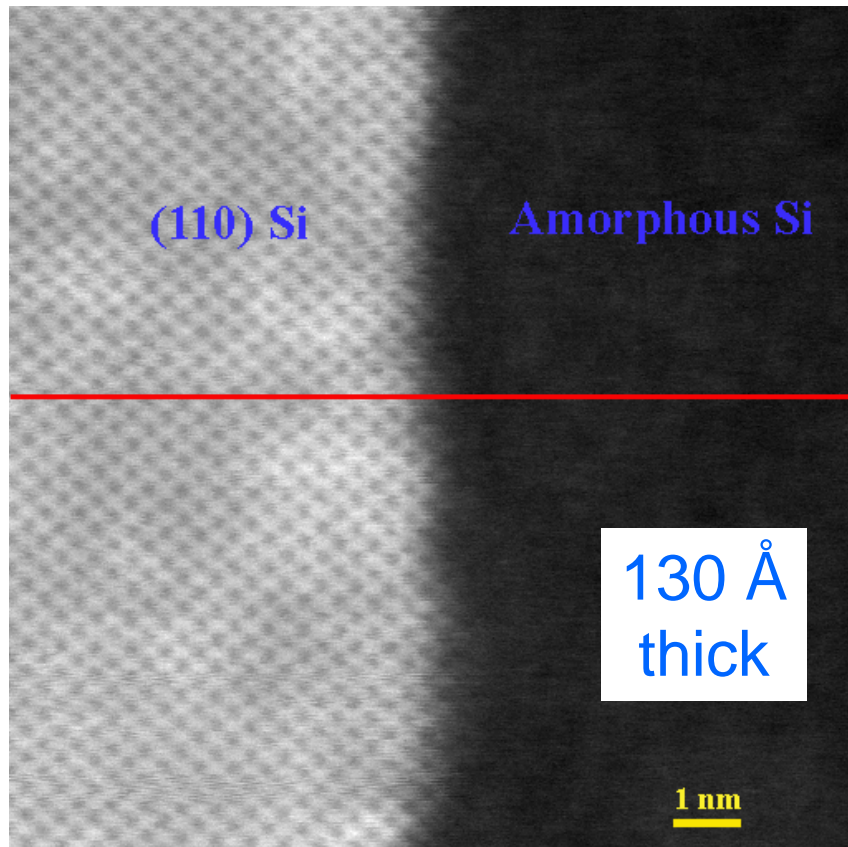
Z. Yu, D. A. Muller, and J. Silcox, *J. Appl. Phys.* **95**, 3362 (2004).



# Strain Contrast vs. Sample Thickness

Contrast at a c-Si/-aSi is strongly depends on sample thickness

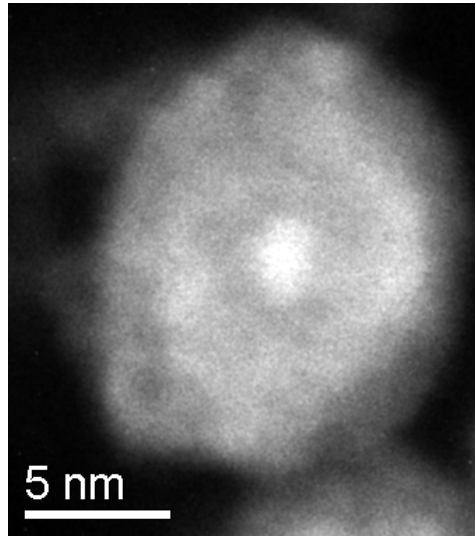
100 kV, 45 mrad ADF inner angle



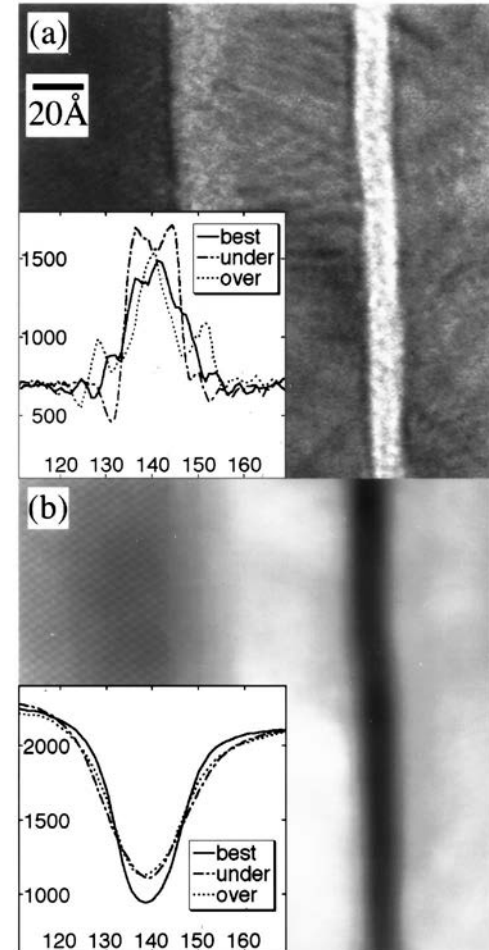
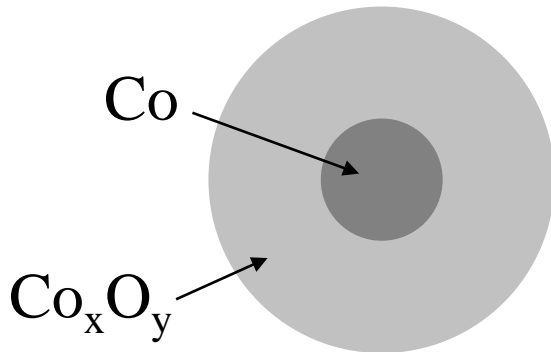
Strain Contrast effects at the interface:

for 130 Å thick sample, ~0%;    for 340 Å thick sample, 15%.

# Examples: Low Resolution



oxidized Co nanoparticle

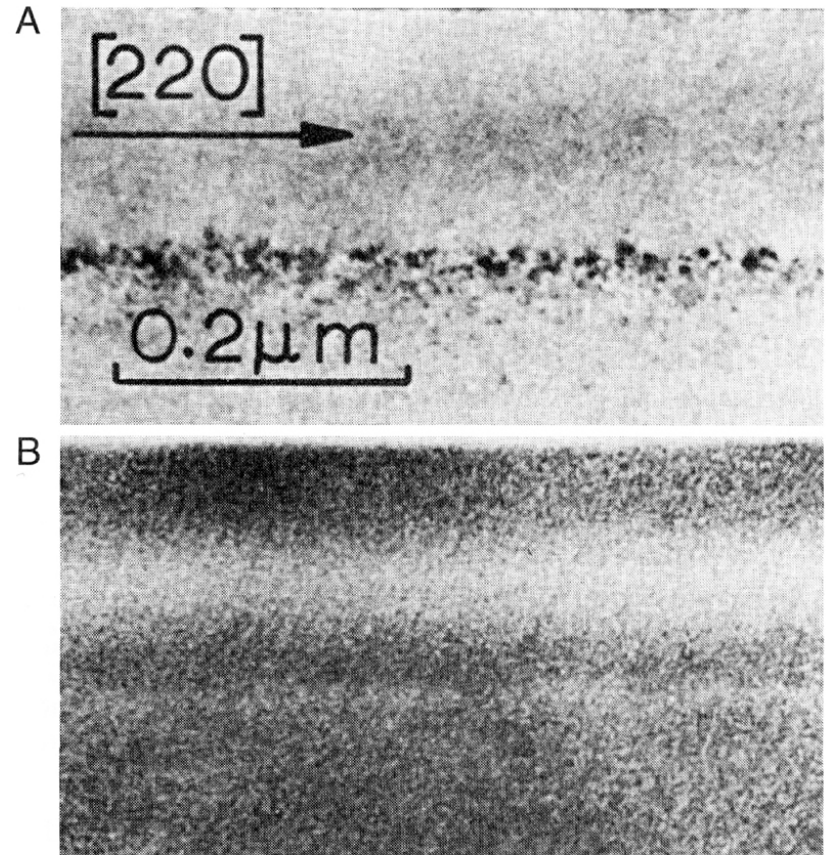


Co / AlO<sub>x</sub> / Co tunnel junction

M. J. Plisch et al. APL **79**, 391 (2001).

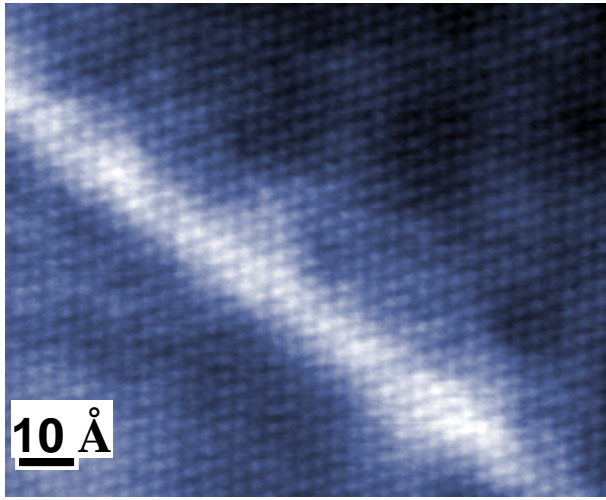
# Bi-Implanted Si

- A shows low-res BF
  - Can't really see implant
  - Can see damage
- B is Z-contrast image
  - Bi lights up like a Christmas Tree
  - The damage layer is not so visible.
    - No phase contrast



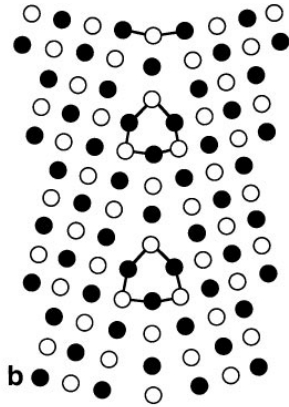
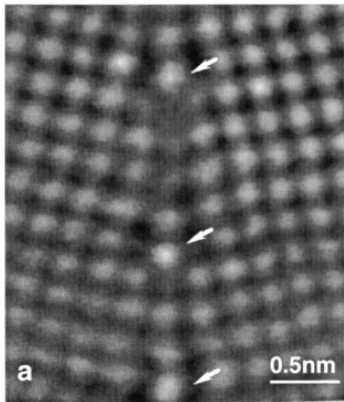
**Figure 22.14.** (A) Low-resolution TEM BF image showing a row of defects in Bi-implanted Si. In (B), obtained under Z-contrast conditions, the defects associated with the implant are invisible but the specimen is bright in the region implanted with Bi.

# Examples: High Resolution



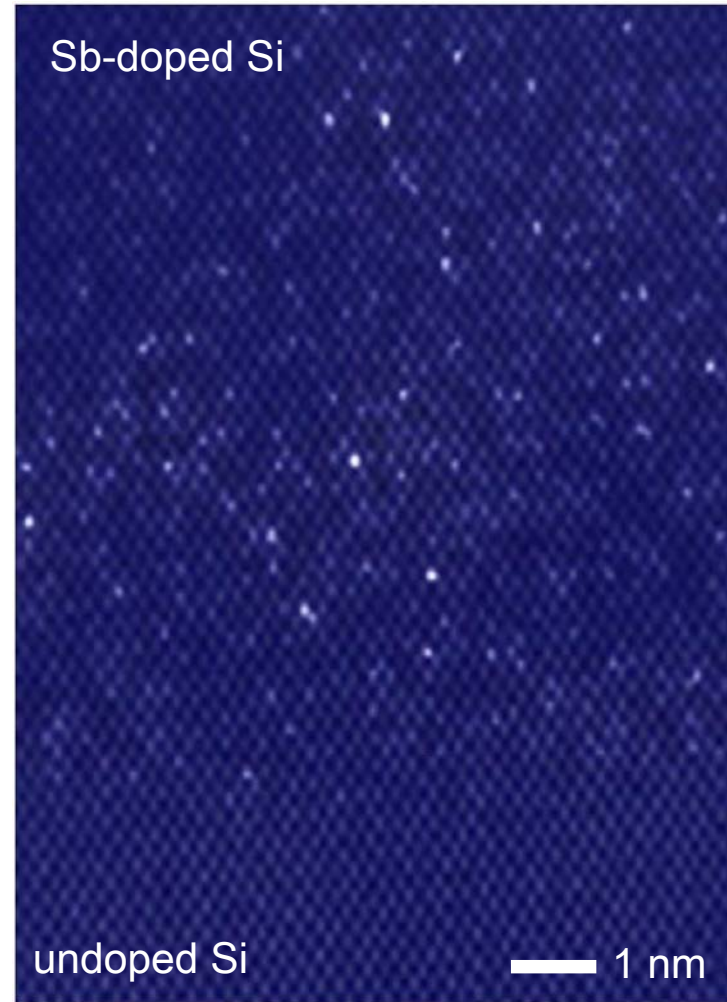
As  $\delta$ -doped layer in Si

D. A. Muller PRL **83**, 3234 (1999)



Grain boundary in MgO

Y. Yan PRL **81**, 3675 (1998)

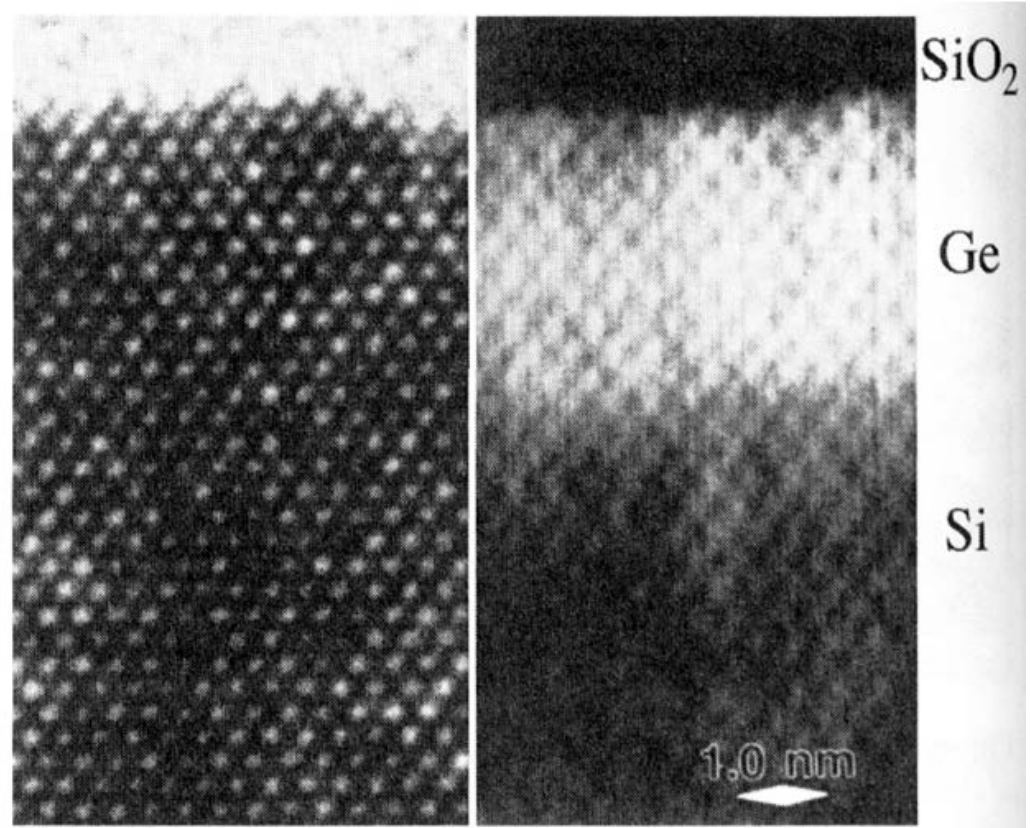


Individual Sb atoms and defect nanoclusters in Si

P. M. Voyles, Nature **416**, 826 (2002)

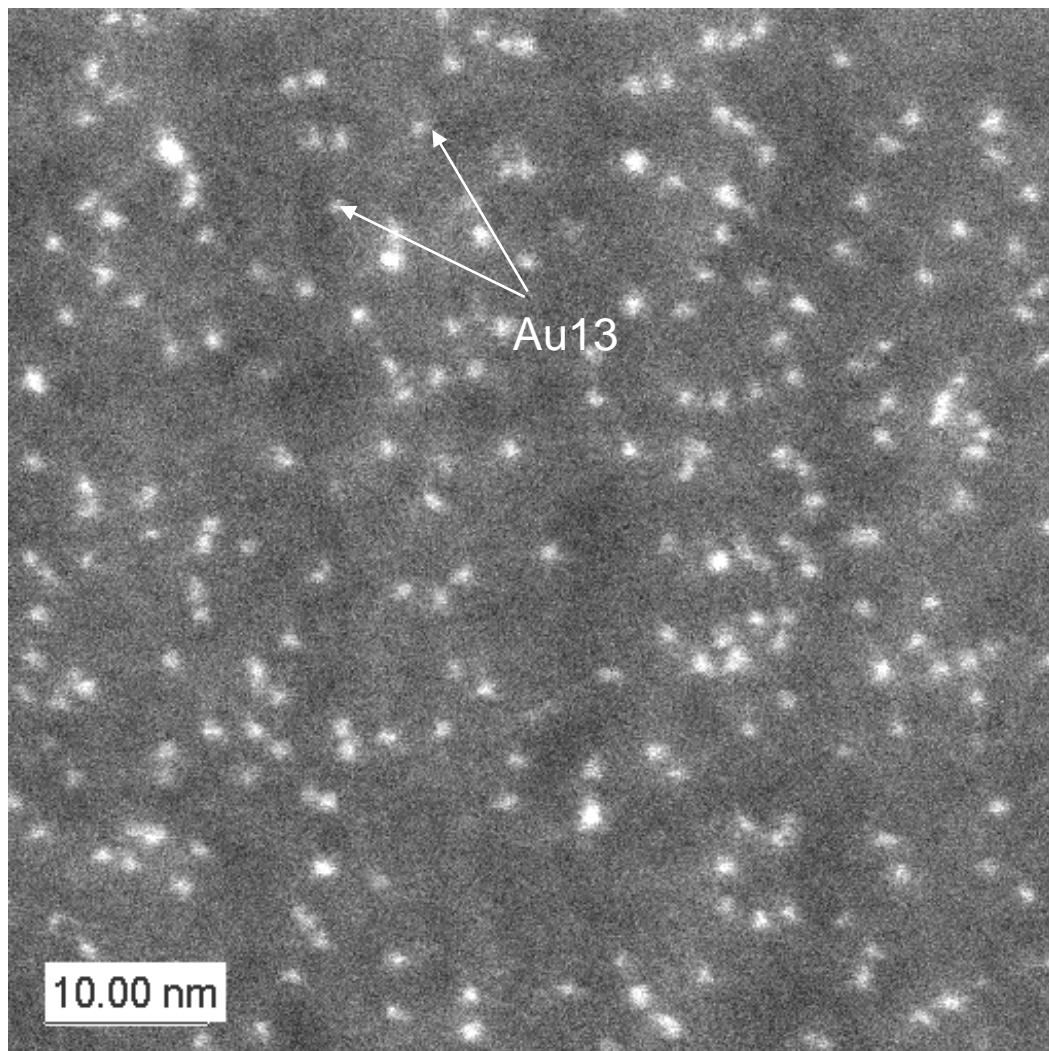
# SiO<sub>2</sub> on Ge on Si

- Amorphous region is visible in hi-res
- Oxide is dark, Ge is light, Si in between - can see lattice



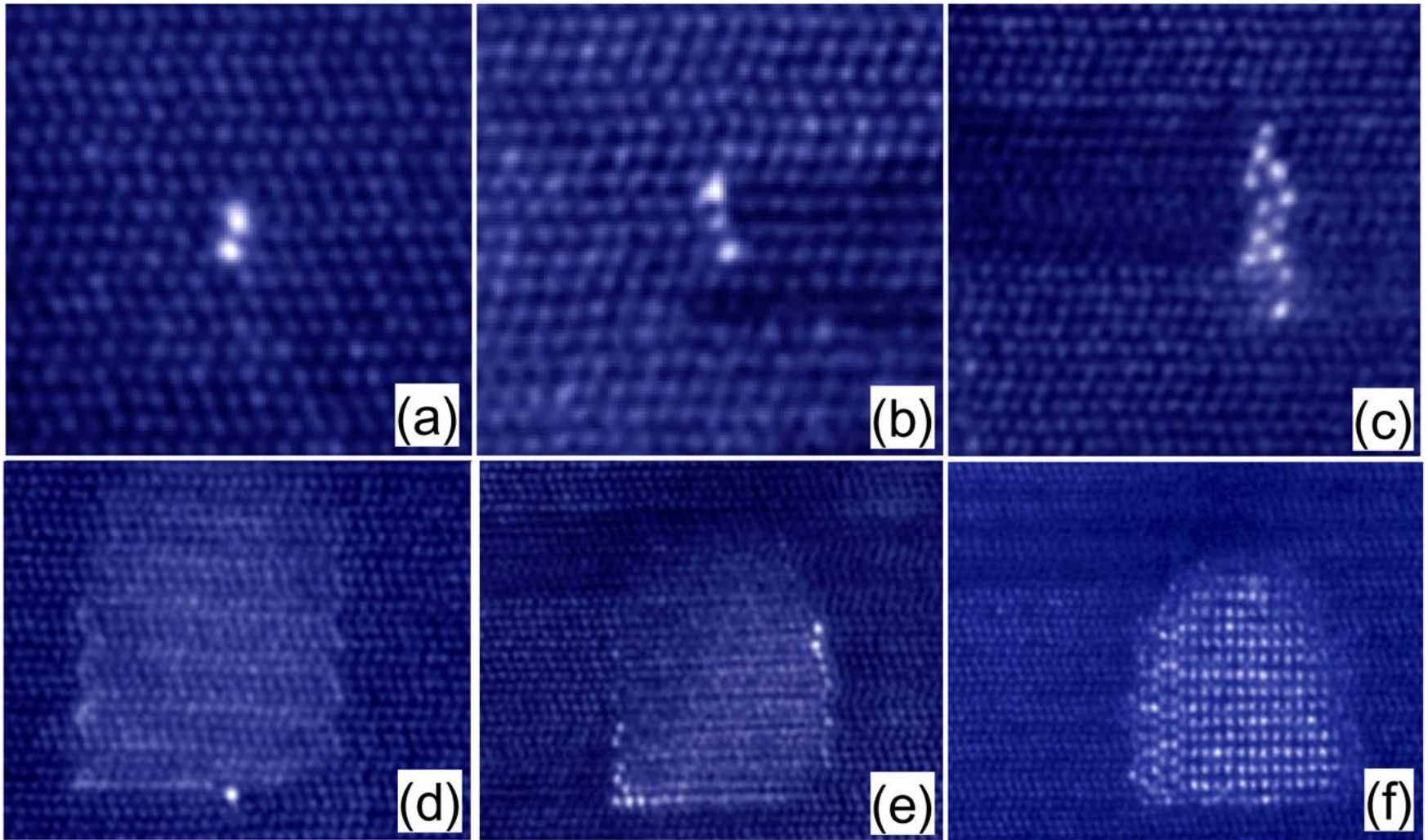
# HAADF-STEM

High-Angle Annular Dark-Field Scanning Transmission Electron Microscopy



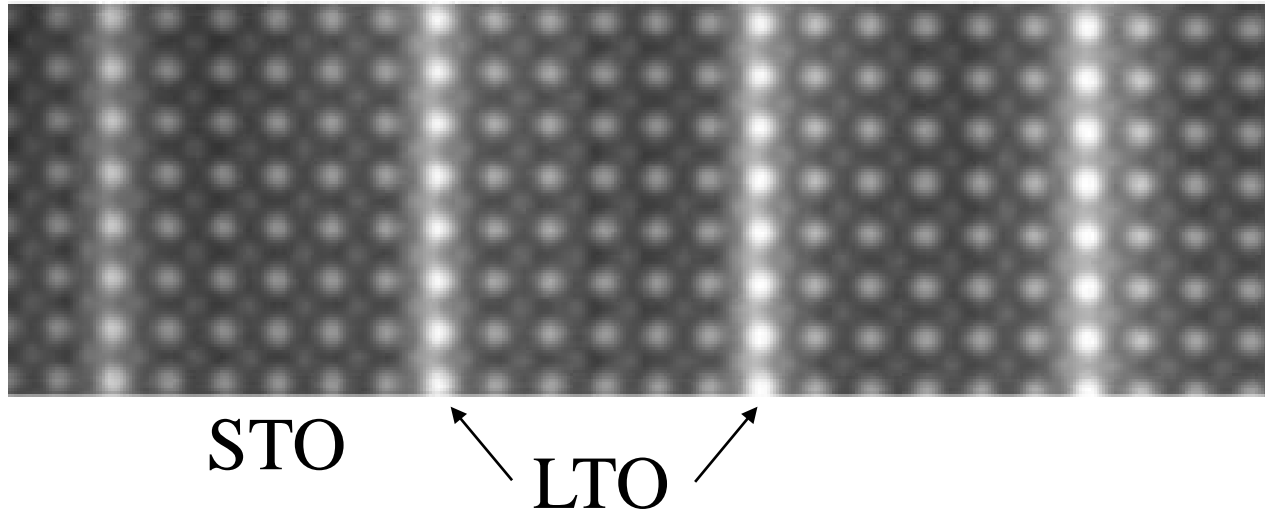
A representative STEM-HAADF image (HB 501, 100kv, inner angle: 96mgrad) of sample  $\text{Au}_{13}(\text{PPh}_3)_4(\text{SC12})_4$

# Examples



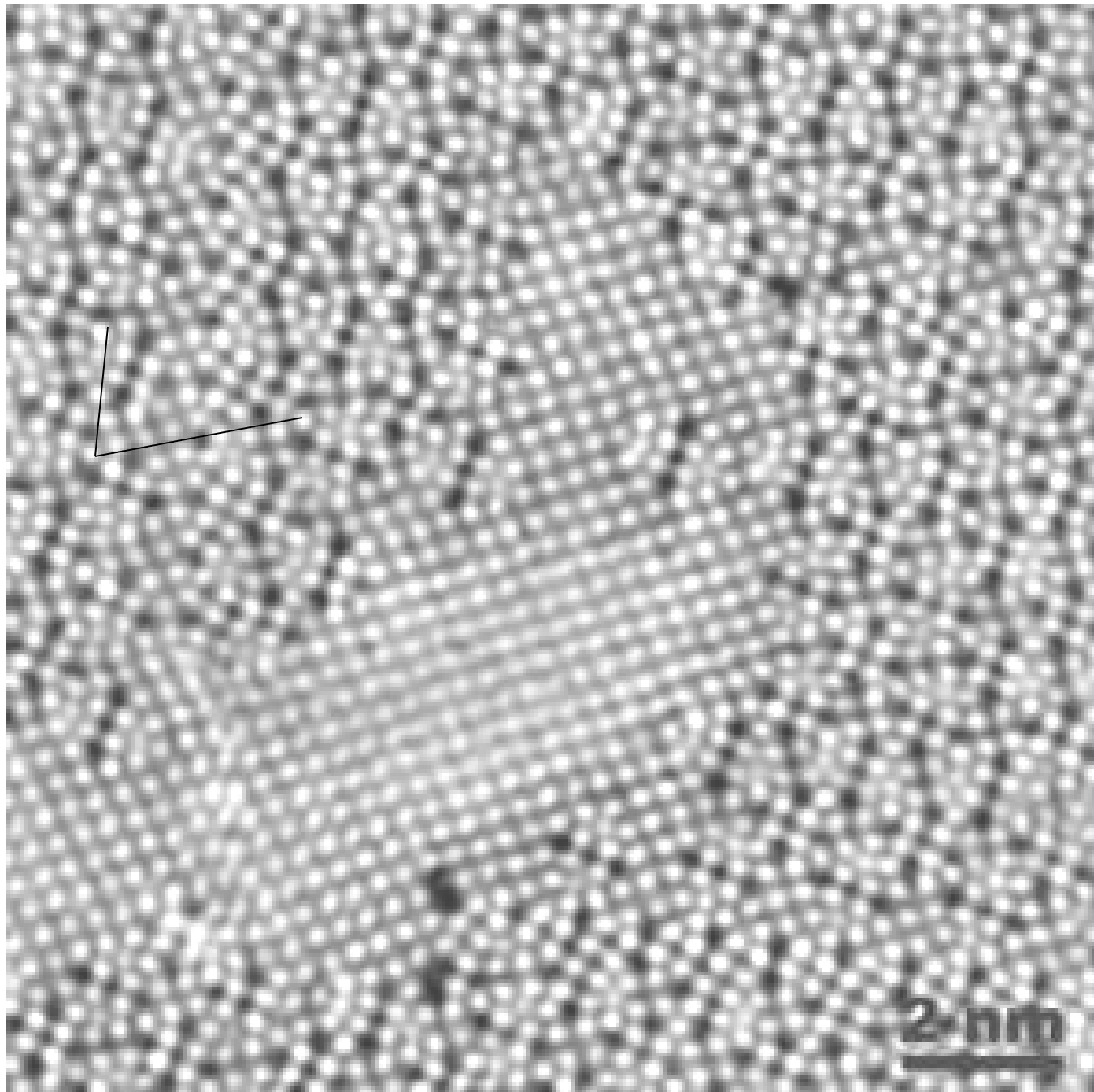
Growth of Er clusters in SiC on annealing.

# Examples



$\text{SrTiO}_3$  /  $\text{LaTiO}_3$  multilayers





$$\mathbf{I} = \beta \mathbf{Z}^n$$

$$Z_{\text{O}} = 8$$

$$Z_{\text{Fe}} = 26$$

$$Z_{\text{Pb}} = 82$$

$\text{Pb}_2\text{Fe}_2\text{O}_5$  - HAADF

$\text{Pb}_2\text{Fe}_2\text{O}_5$  - HRTEM

Abakumov et al.  
*Angewandte Chemie (2006)*

# Summary: STEM

- Similar signals to TEM, often easier to obtain some types
- Parallel collection of different signals, but serial detection
- Easier for EELS/EDS mapping and similar
- Annular dark-field is not as simple as often thought, serious misinterpretations exist in the current literature
  - Strain/diffraction contrast effects in ADF
  - Approaches Z-contrast in HAADF
  - Sometimes not Z-contrast