

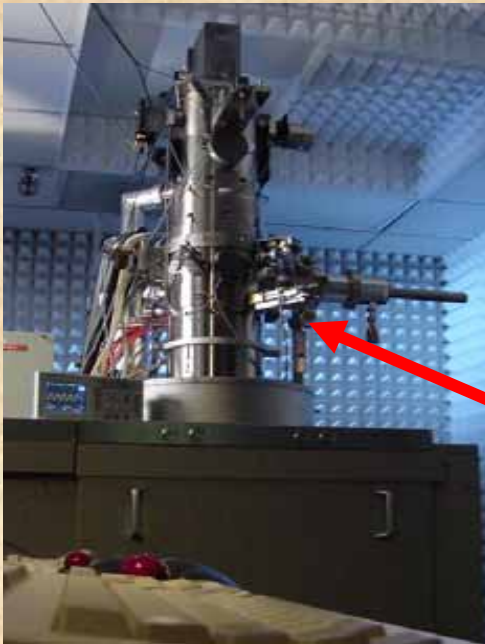
# 3D characterization of semiconductor interfaces using aberration-corrected STEM

**Klaus van Benthem**, Miyoung Kim, Andrew R. Lupini,  
Sergey N. Rashkeev, and Stephen J. Pennycook

Oak Ridge National Laboratory

*benthem@ornl.gov*

*http://stem.ornl.gov*



# Motivation

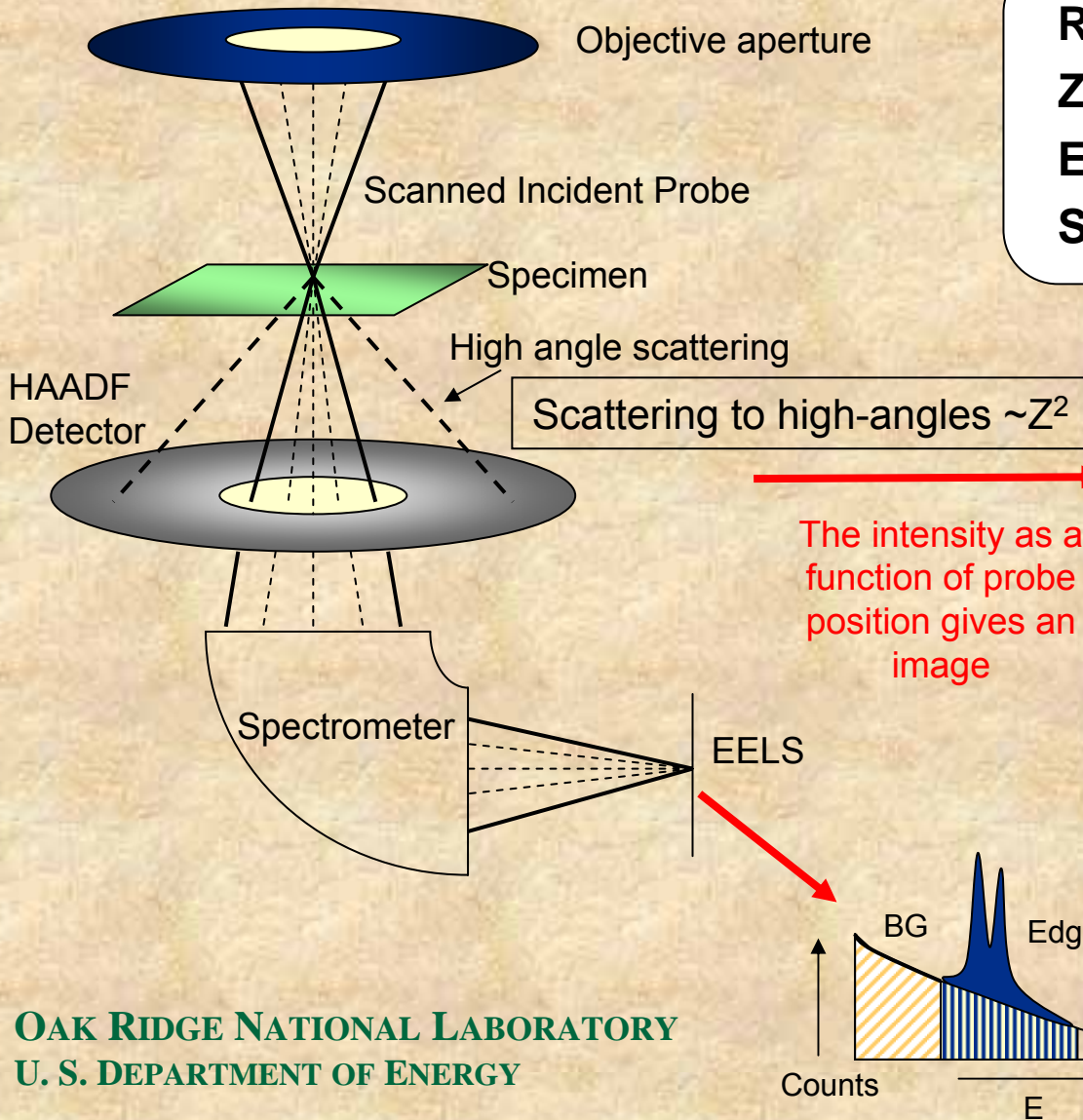


Richard F. Feynman  
1918-1988

- ***“What good would it be to see individual atoms distinctly?”***
- ***“Another direction of improvement is to make physical machines three dimensional [...].”***

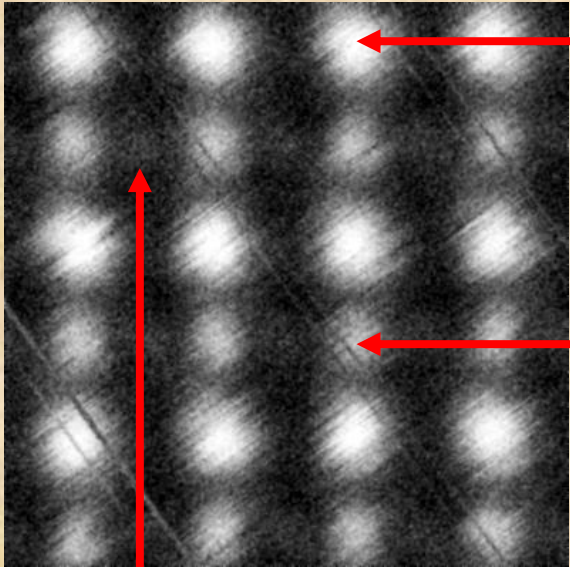


# STEM Z-contrast imaging



**Resolution = probe size**  
**Z-contrast  $\sim Z^2$**   
**Easy image interpretation**  
**Simultaneous EELS**

SrTiO<sub>3</sub> [110] HAADF image



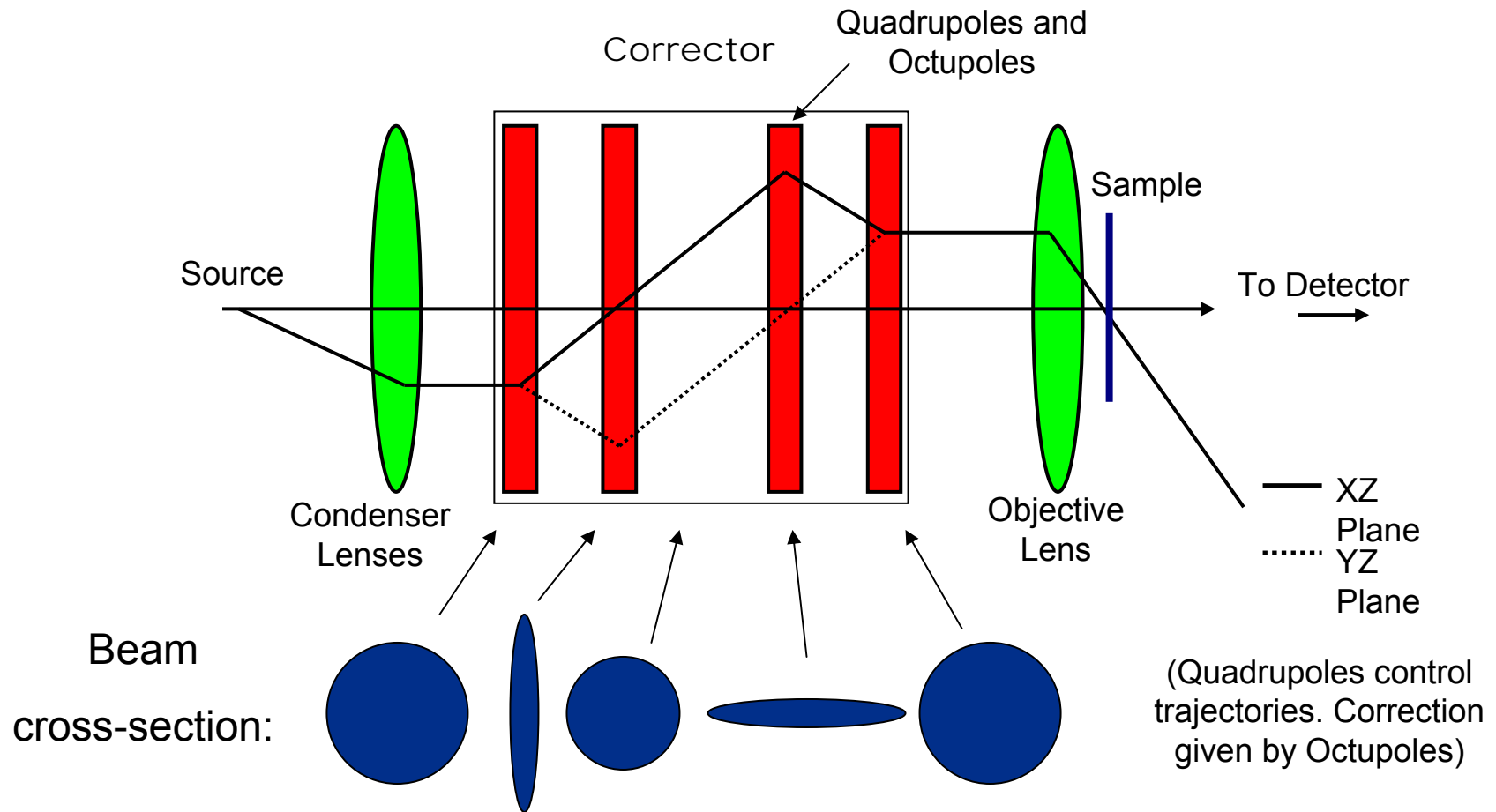
Sr  
(38)

Ti  
(22)

O  
(Z=8)

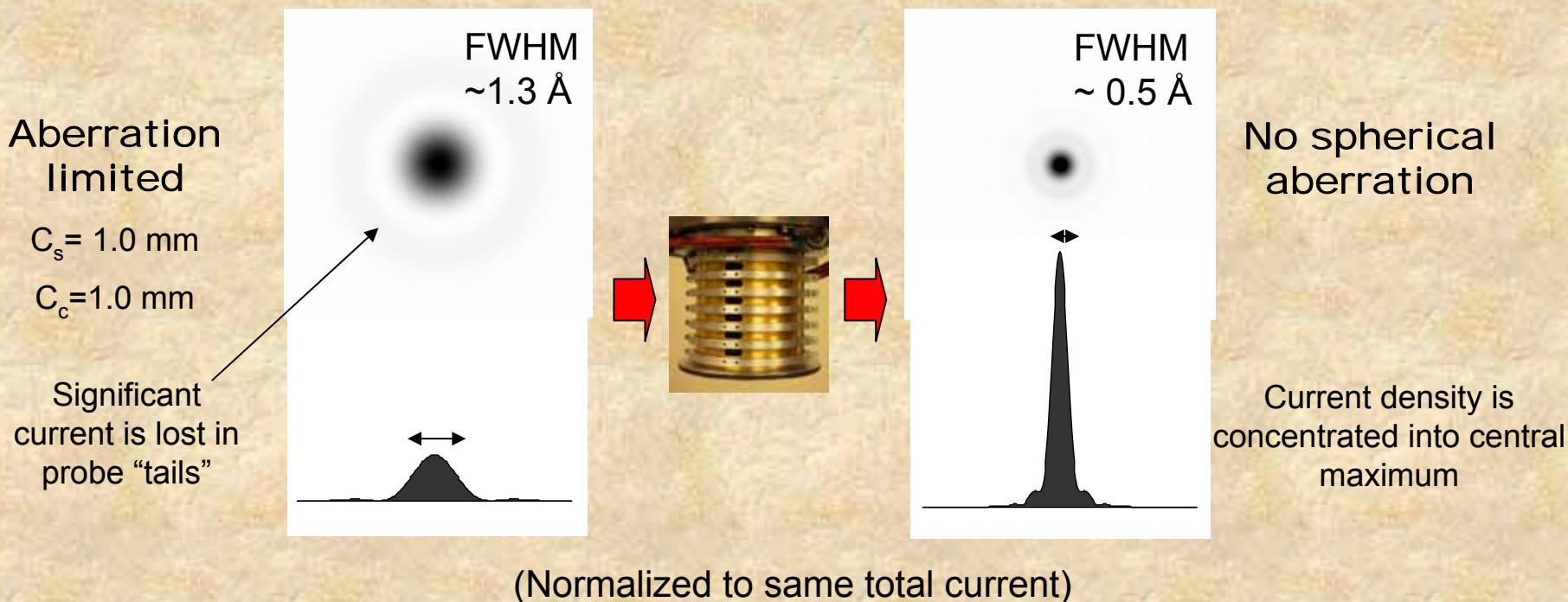
# Aberration Correction in STEM

## Nion aberration corrector



# Aberration corrected probe

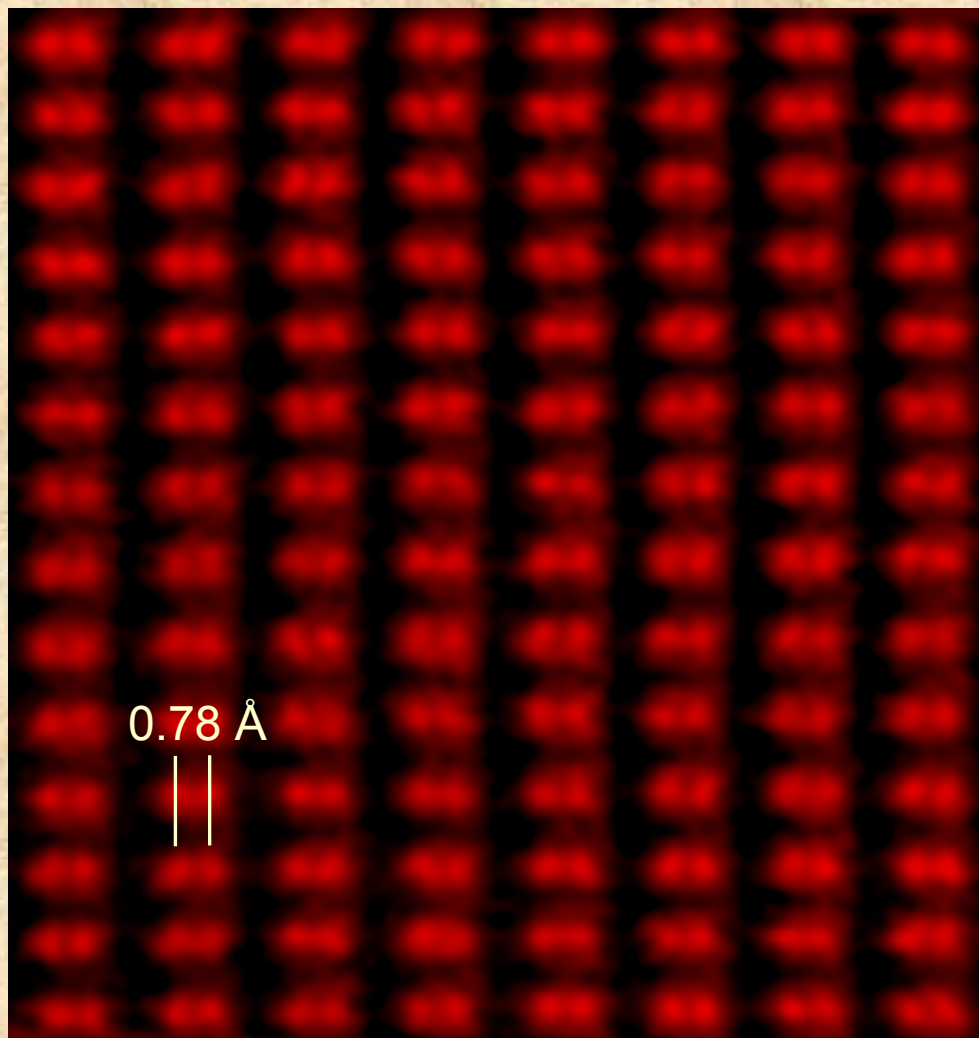
VG Microscope's HB603U, 300 kV



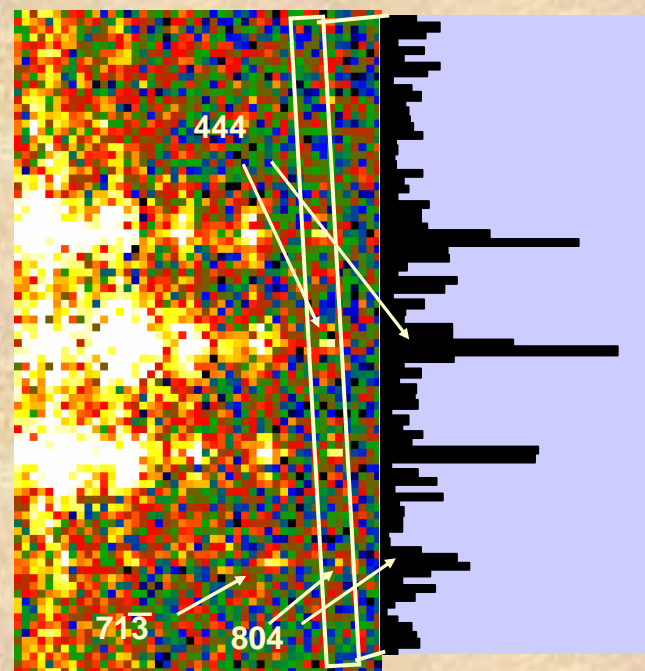
Aberration correction  $\Rightarrow$  "smaller" and "brighter" probe  
Critical for single atom sensitivity



# Pico-scale Z-contrast Imaging:



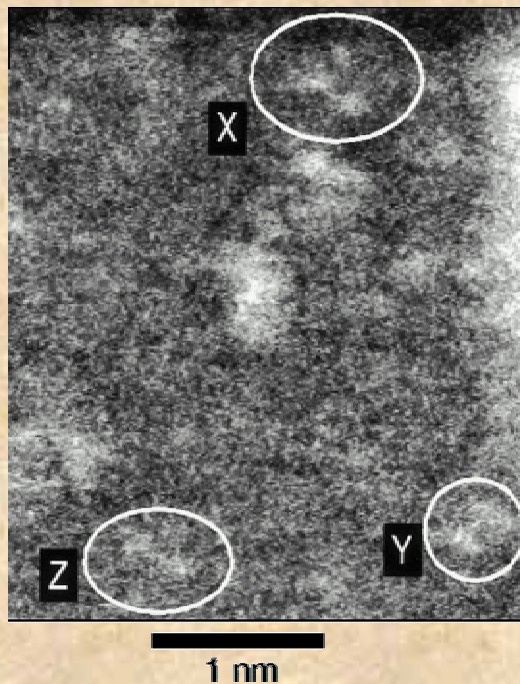
Information transfer to  $0.607 \text{ \AA}$   
(61 pm)



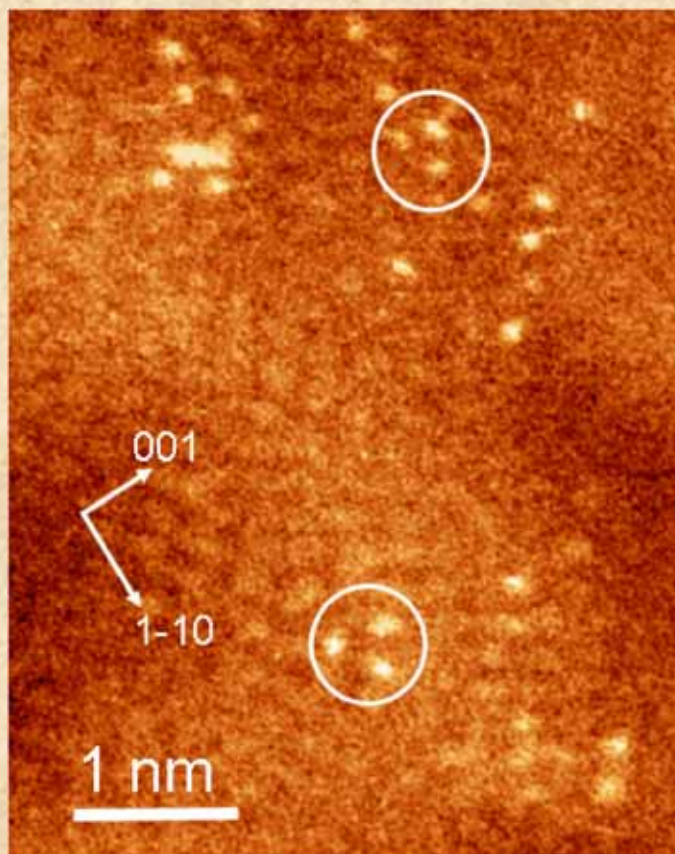
P.D. Nellist et al.,  
Science **305**, 1741(2004)

Si  $\langle 112 \rangle$  Direct Image  
Resolution at  $0.78 \text{ \AA}$

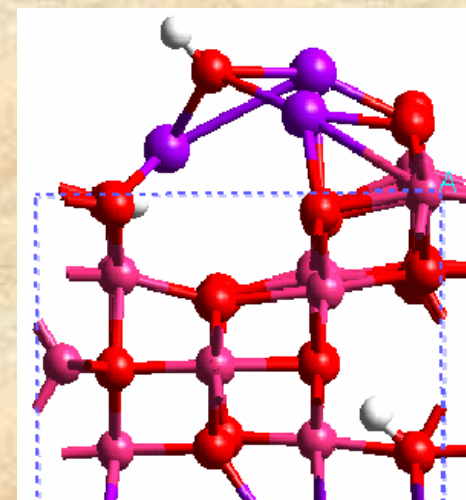
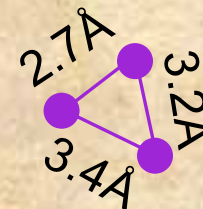
# The form of Pt on the $\gamma$ -Al<sub>2</sub>O<sub>3</sub> (110C) Surface



P.D. Nellist 1996



A.Y. Borisevich 2004



With OH-cap Pt spacings match calculations

Pt atoms change from electron-rich to electron-poor

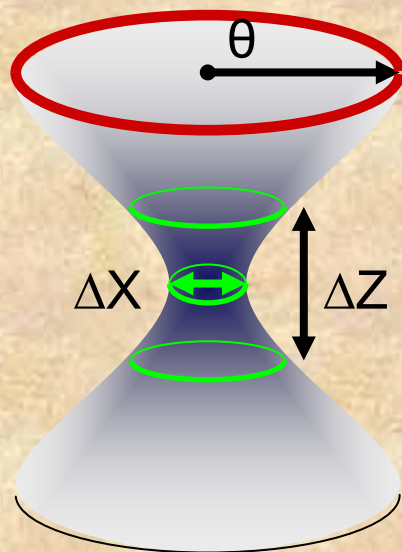
- good Lewis acid sites - Sohlberg et al, ChemPhysChem 5, N 12, 1893-1897 (2004).



# Unexpected Benefit of Aberration Correction

$$\Delta X \approx \lambda / \theta$$

$$\Delta Z \approx \lambda / \theta^2$$

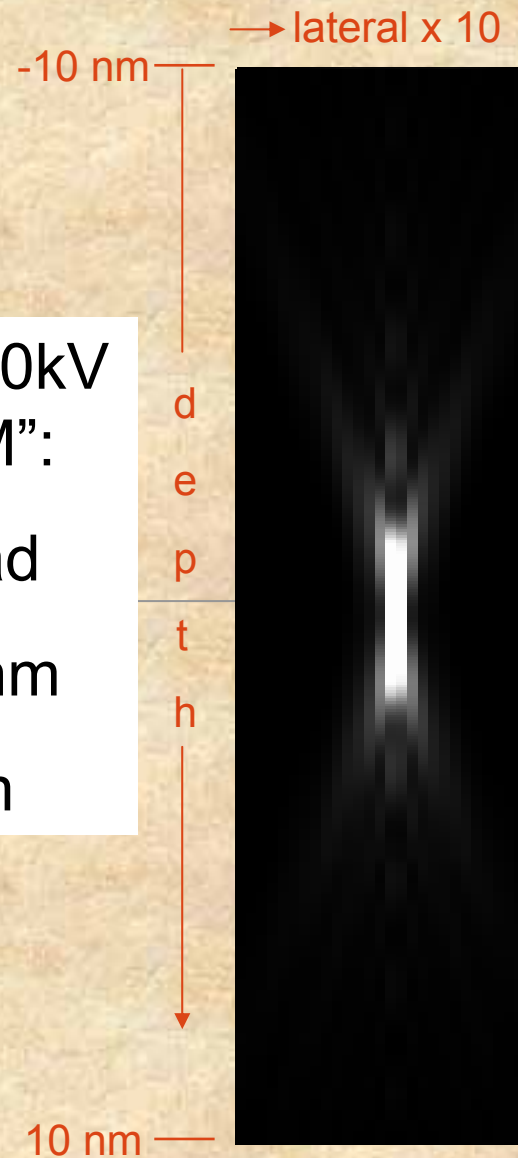


Corrected 200kV  
“UltraSTEM”:

$$\theta = 50 \text{ mrad}$$

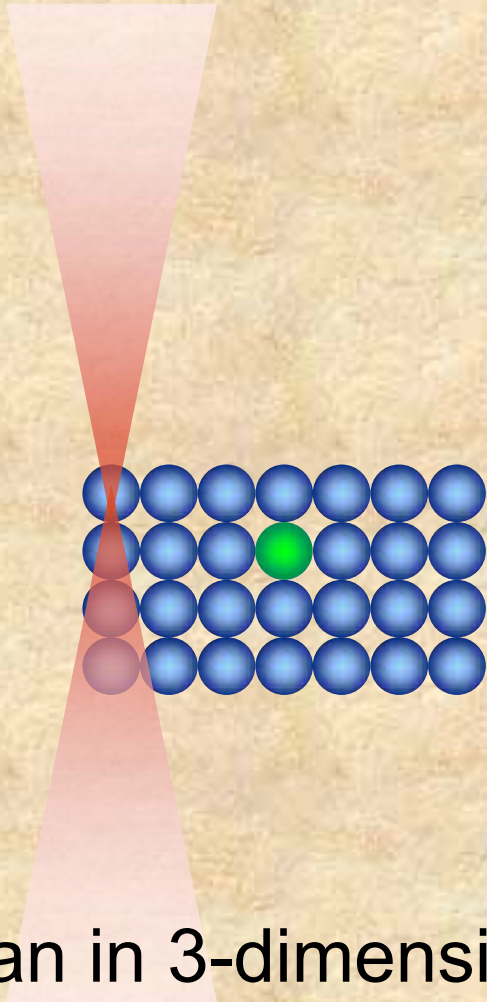
$$\Delta X \approx 0.05 \text{ nm}$$

$$\Delta Z \approx 1 \text{ nm}$$

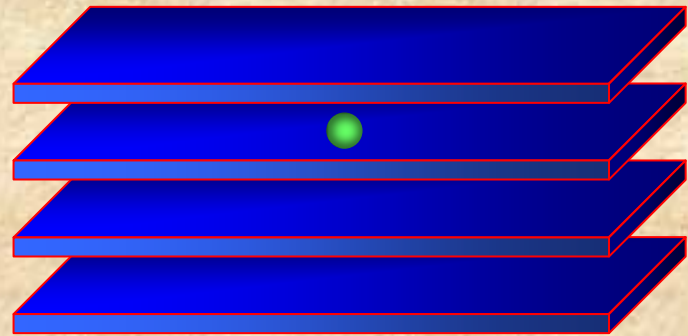




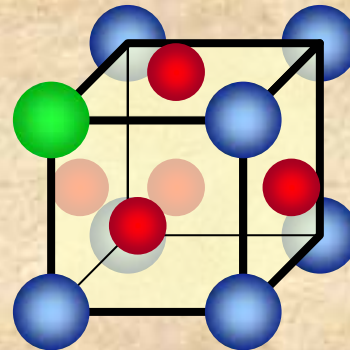
# 3D concept



Scan in 3-dimensions



Build 3D dataset by slices



Build and  
Analyze  
3D Model

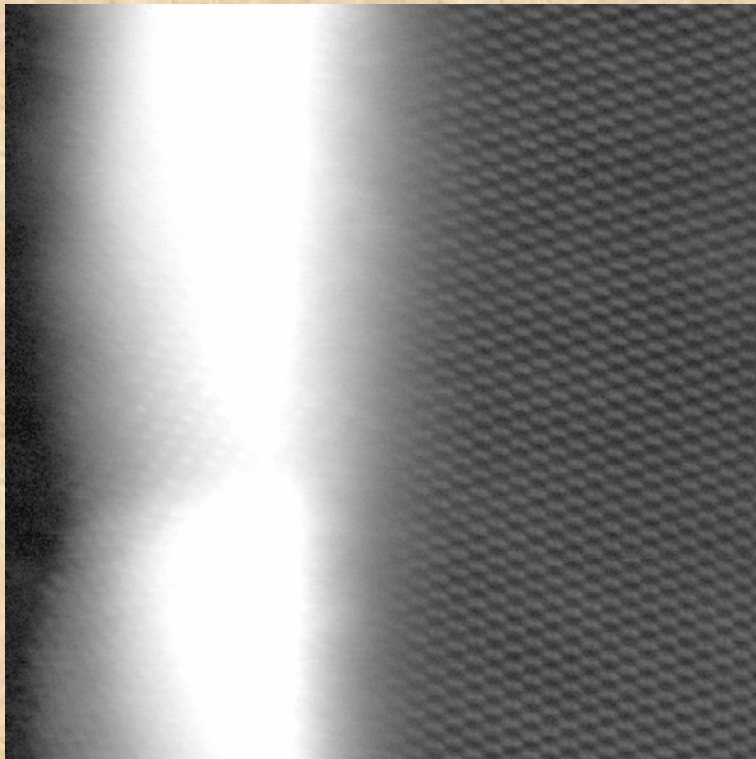
# Si/HfO<sub>2</sub>/poly-Si

poly-Si

HfO<sub>2</sub>

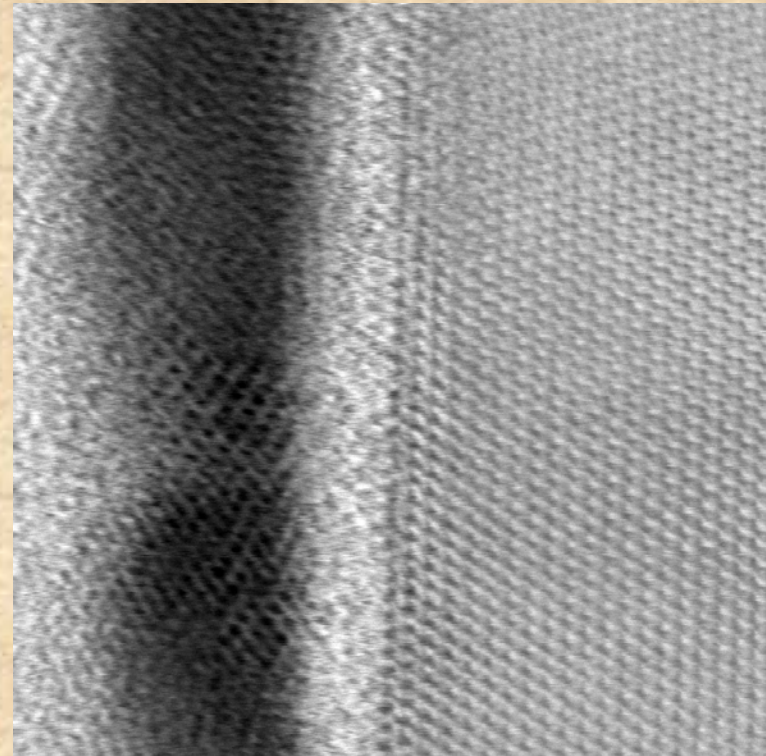
Si <110>

SiO<sub>2</sub>



1.4 nm

HAADF



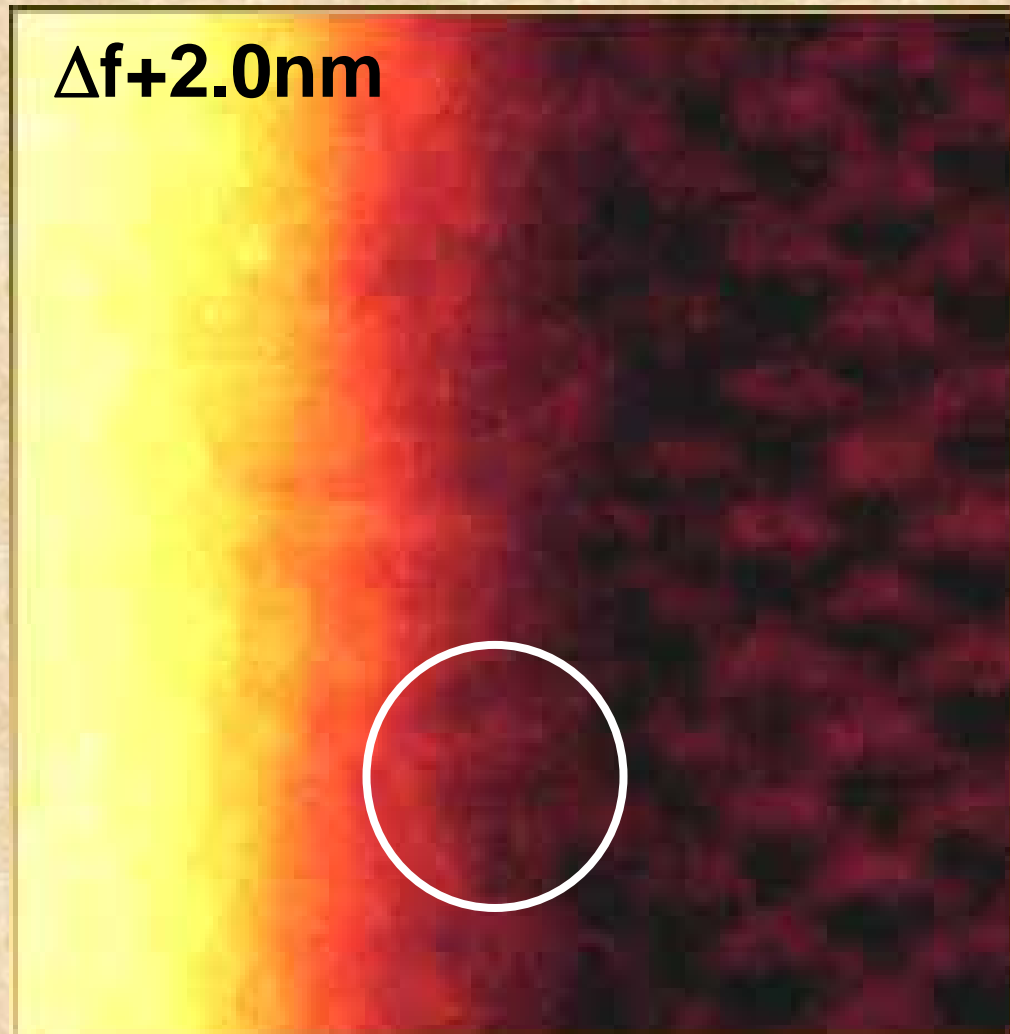
BF

1.4 nm

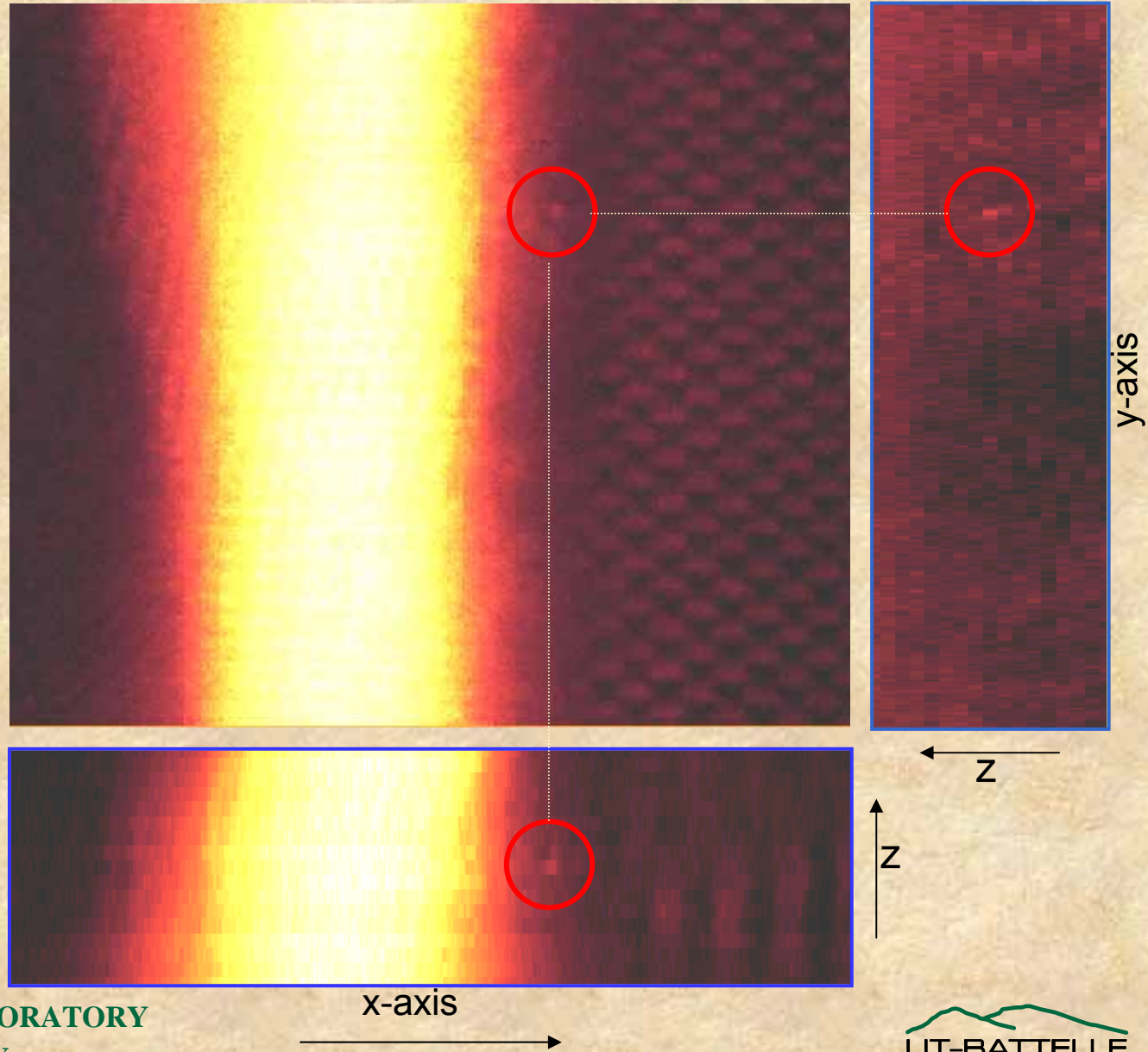
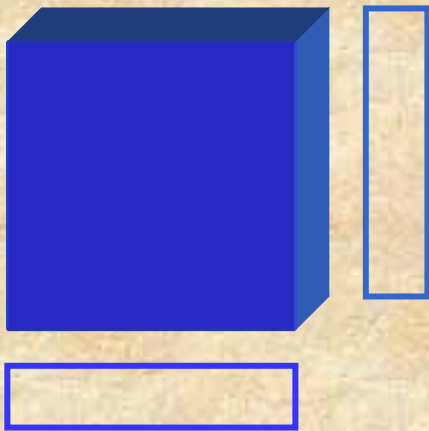
- Atomic layer deposition
- Substrate temperature 320°C
- Annealing at 950°C for 30sec. In N<sub>2</sub>



# 3D Analysis of Semiconductors

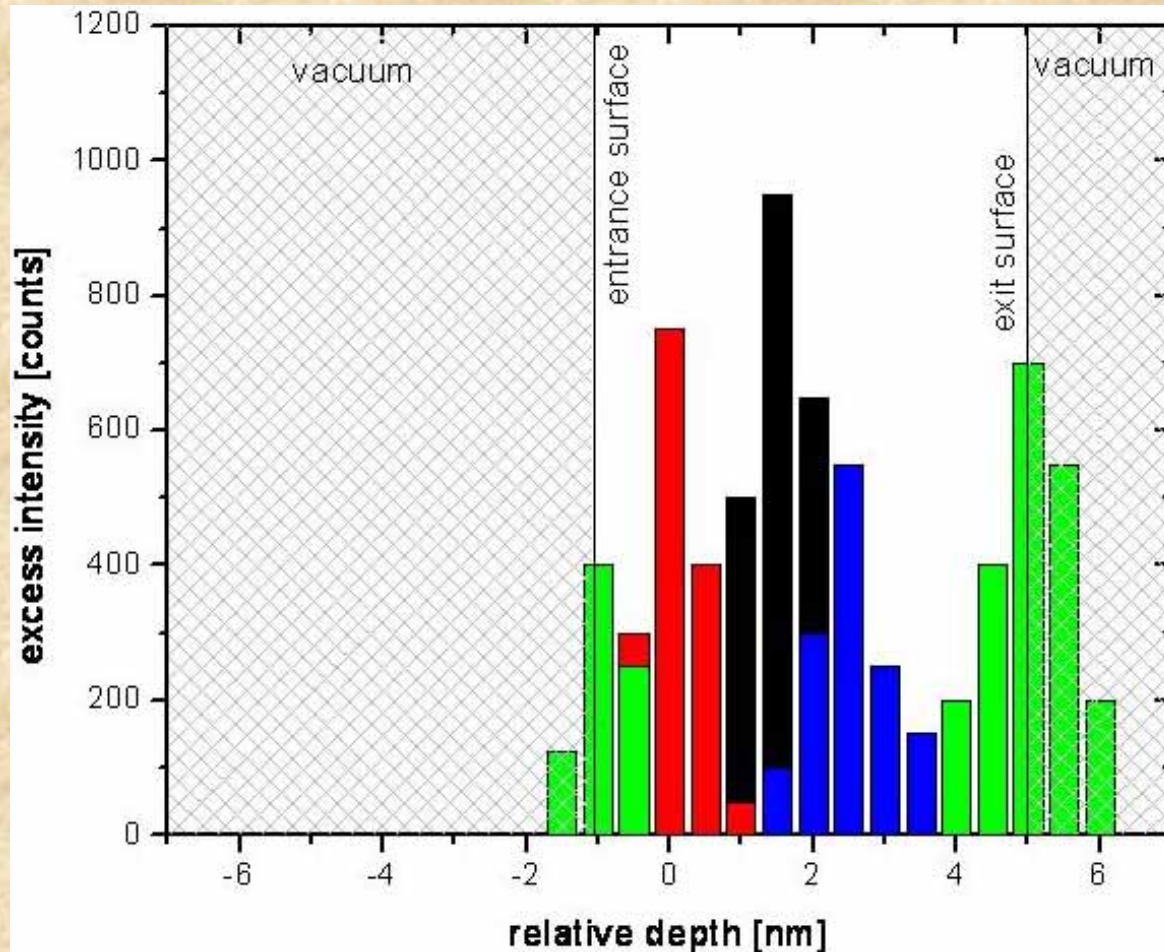


# Slice View

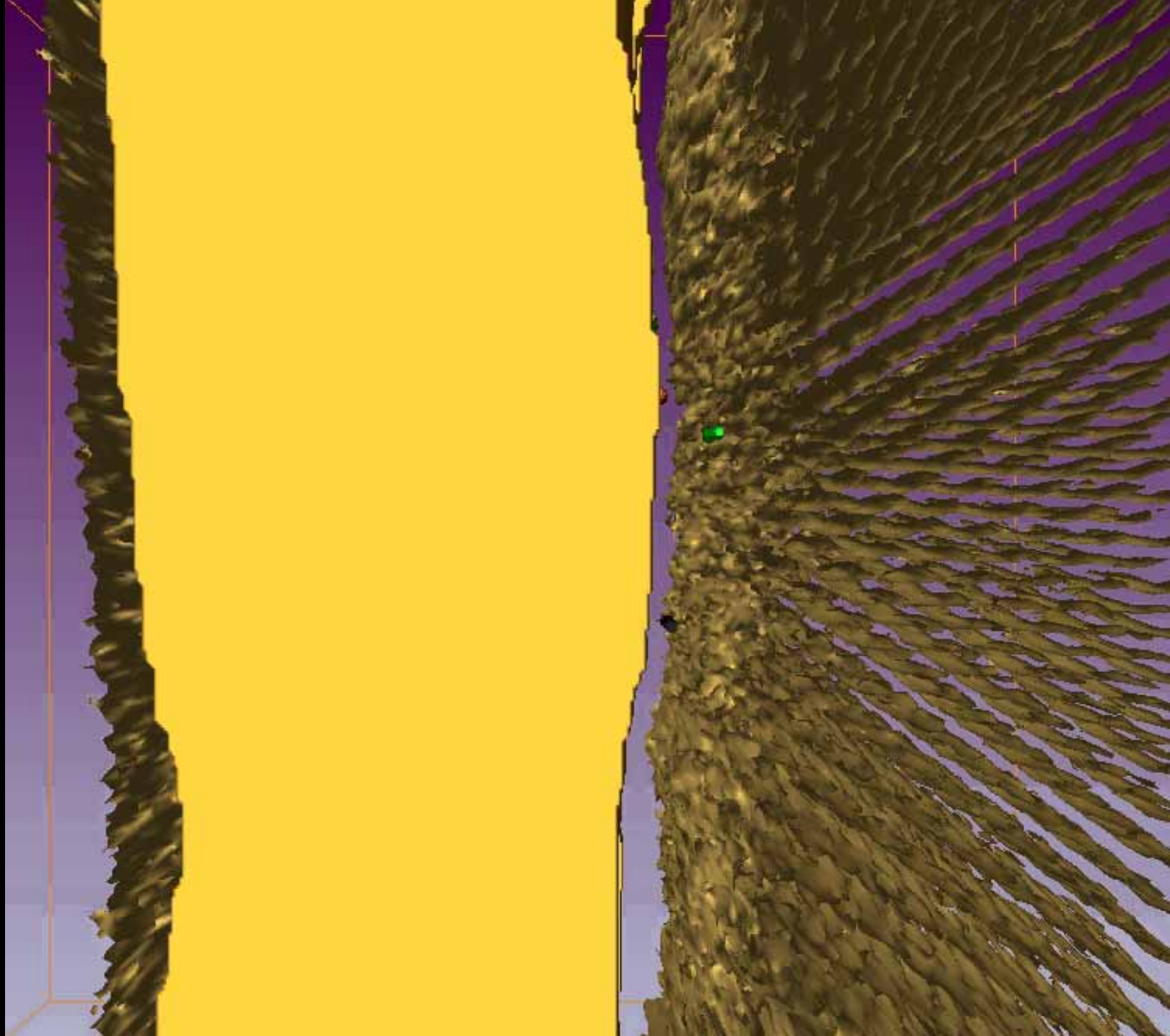




# Vertical position of Hf atoms

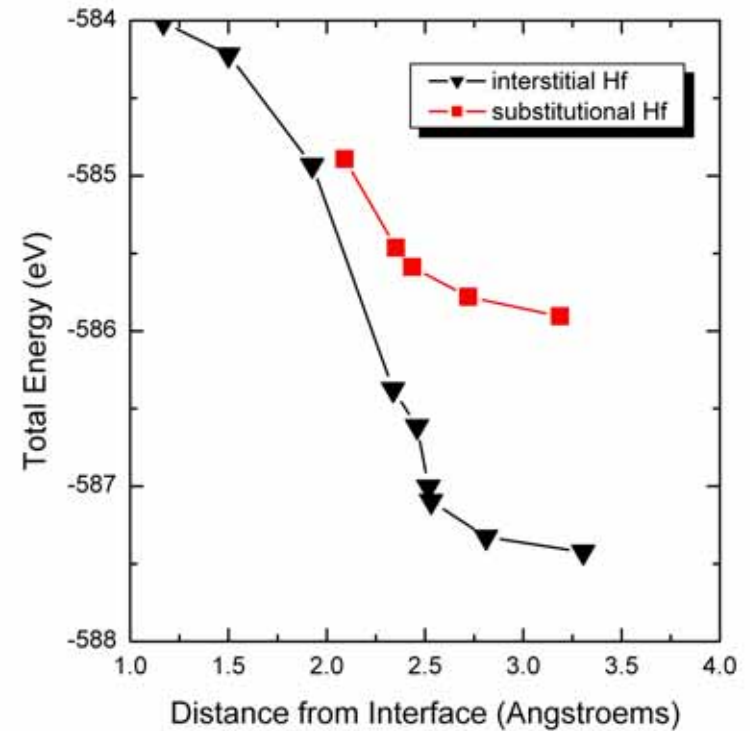
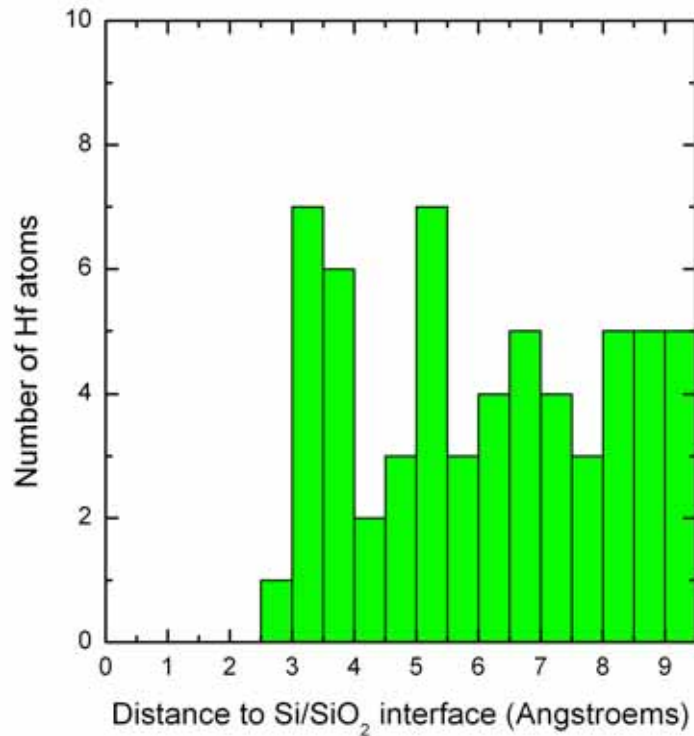


- **Atoms located inside the device**
- **Sample thickness  $6 \pm 1$  nm**



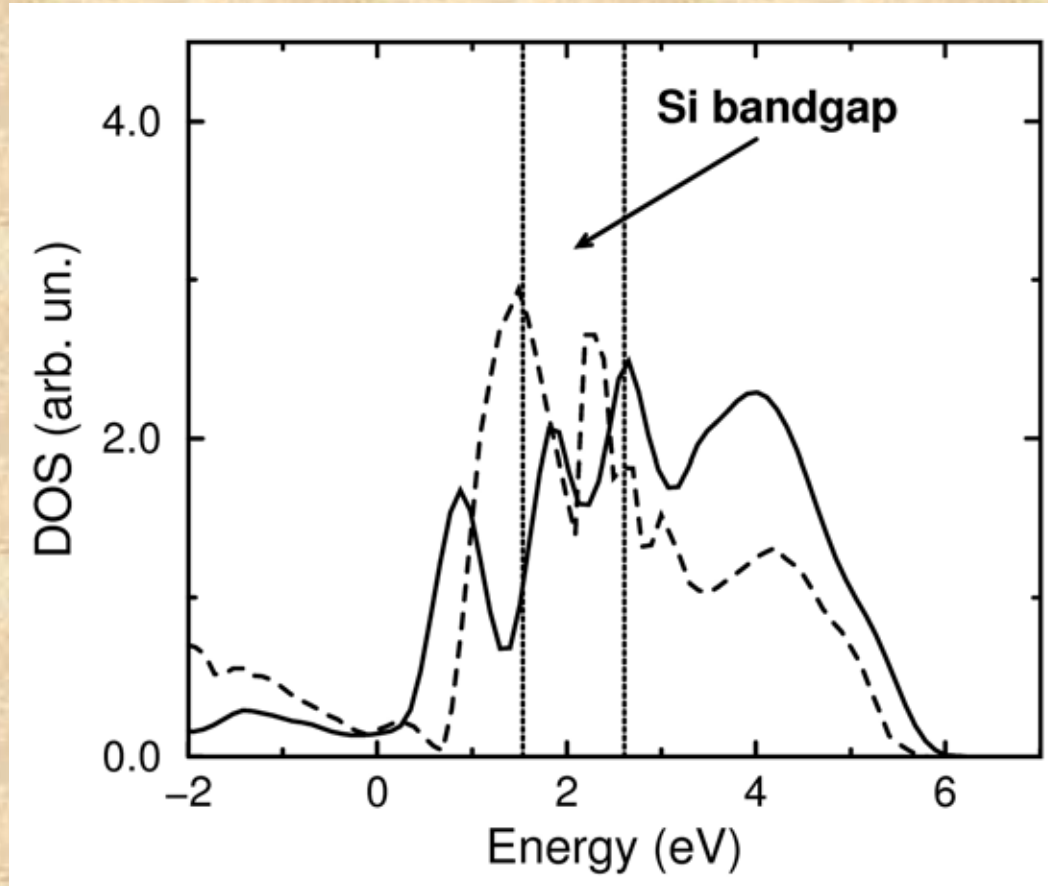
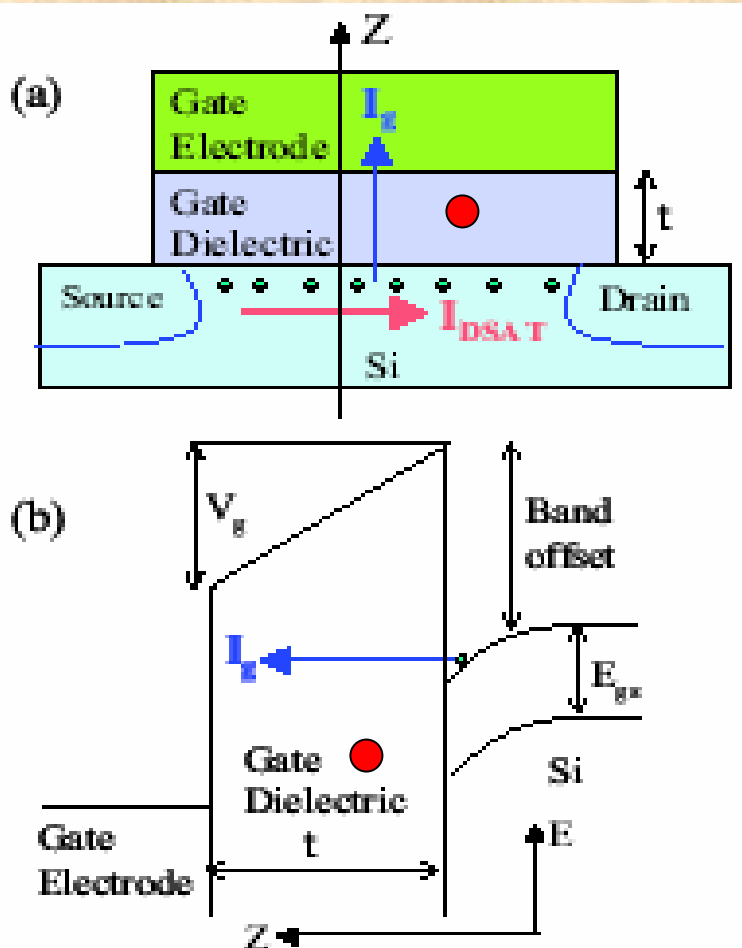


# Hf atom distribution in SiO<sub>2</sub>



S.J. Rashkeev (2004)

# Leakage current related to individual Hf atoms in SiO<sub>2</sub> films



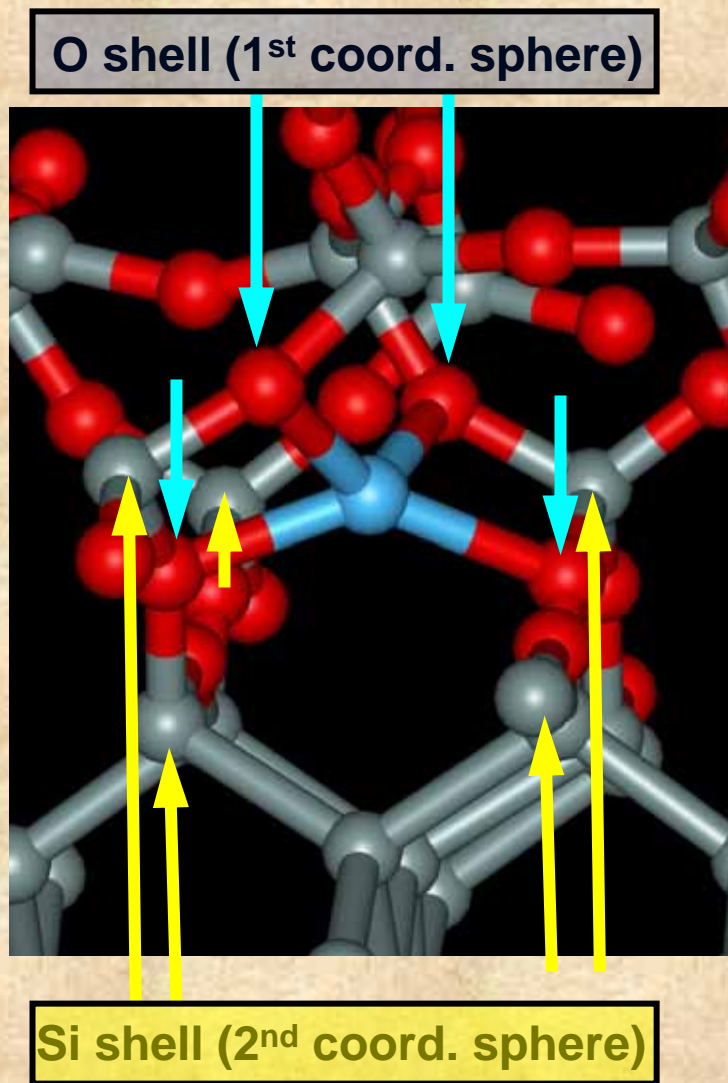
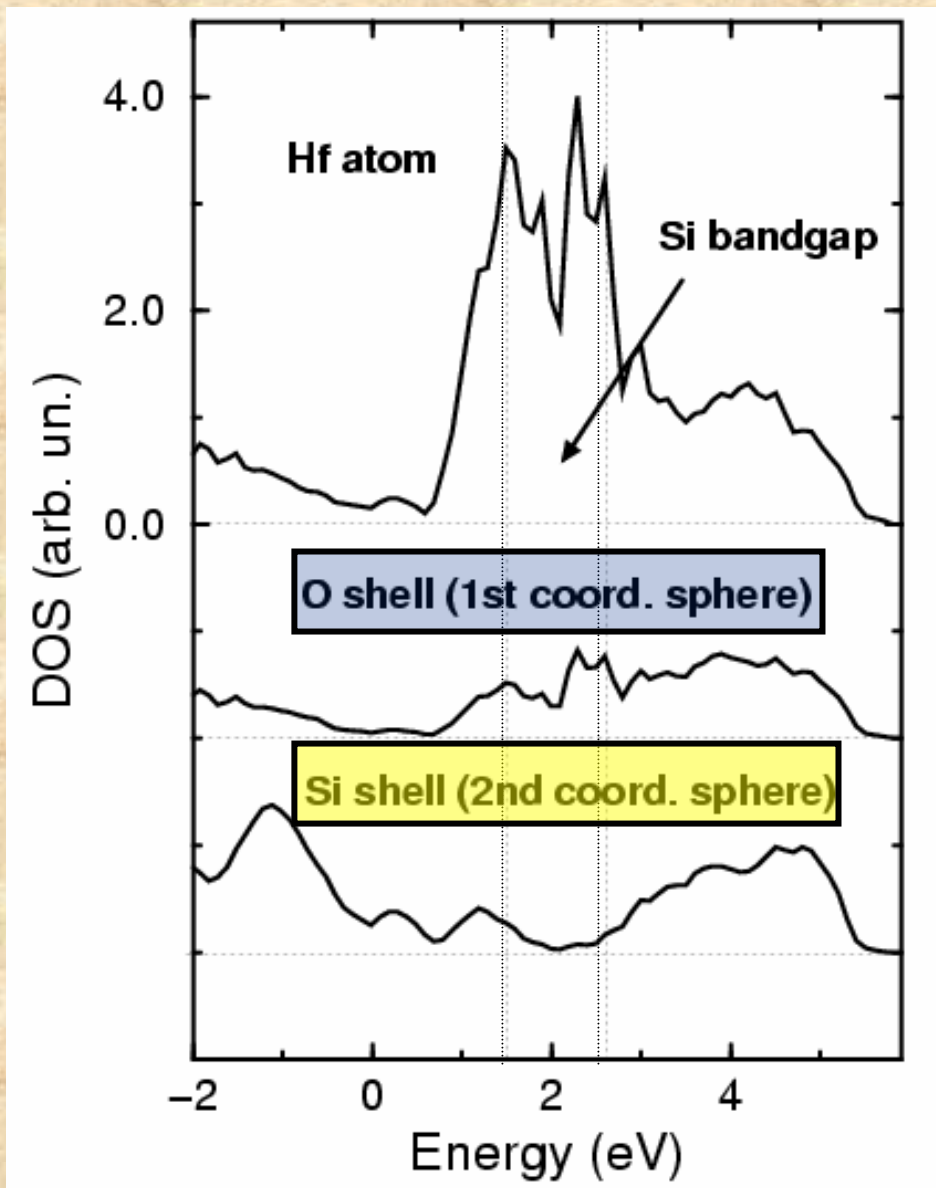
**Hf can act as a leakage current center**

S.N. Rashkeev (2004)

**From:** G. Bersuker, P. Zeitzoff, G. Brown, and H. R. Huff, *Materials Today*, January 2004, p.26

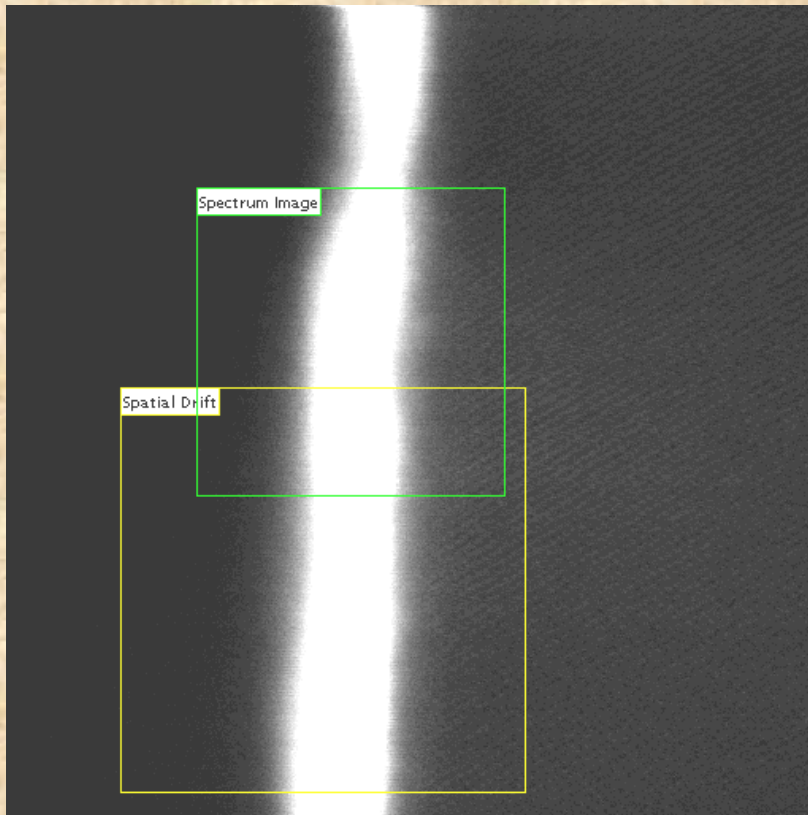


# Other atoms involved in leakage current

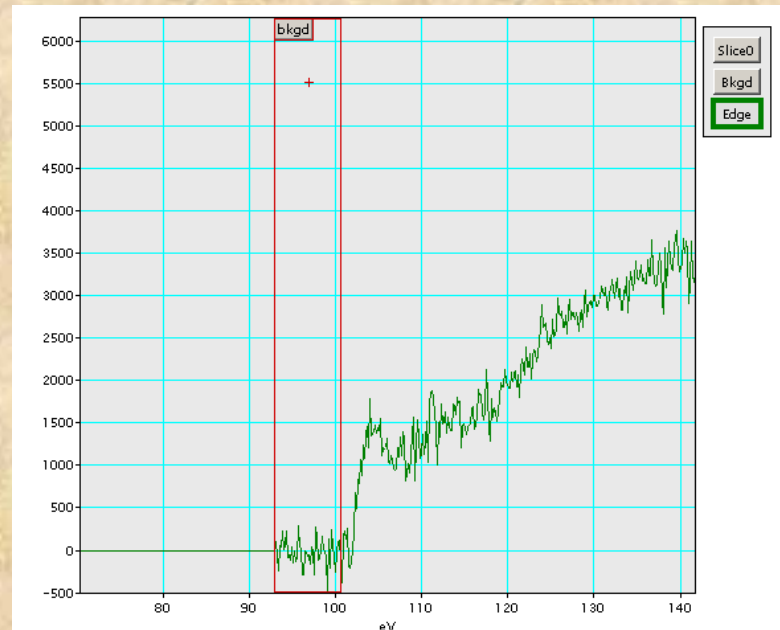
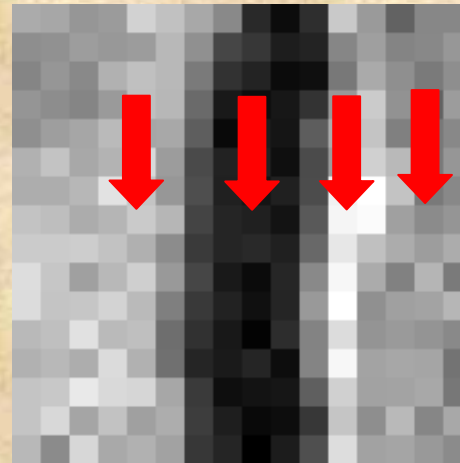


S.N. Rashkeev (2004)

# EELS Spectrum Imaging: Si L<sub>2,3</sub>-edge

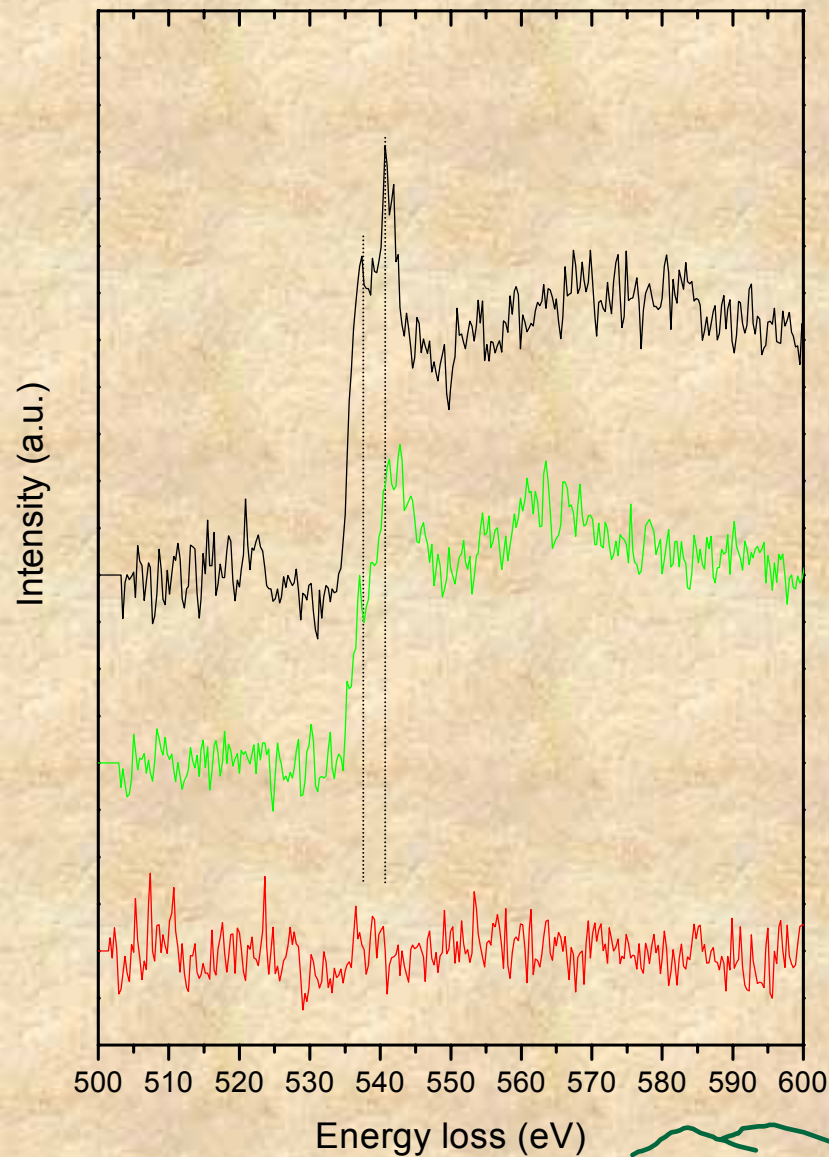
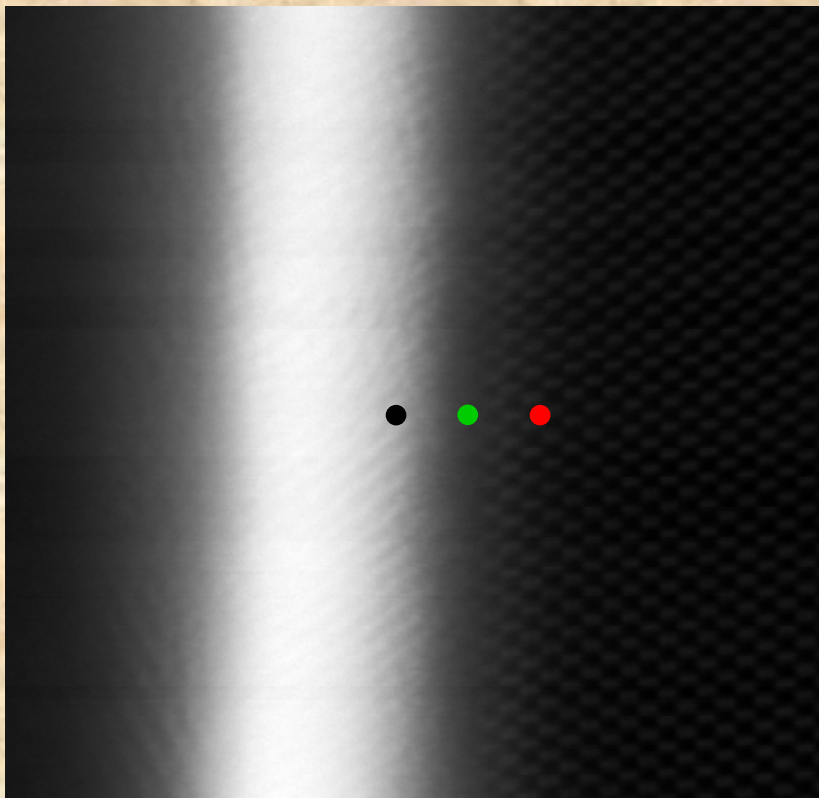


p-Si      HfO<sub>2</sub>/SiO<sub>2</sub>      Si<110>

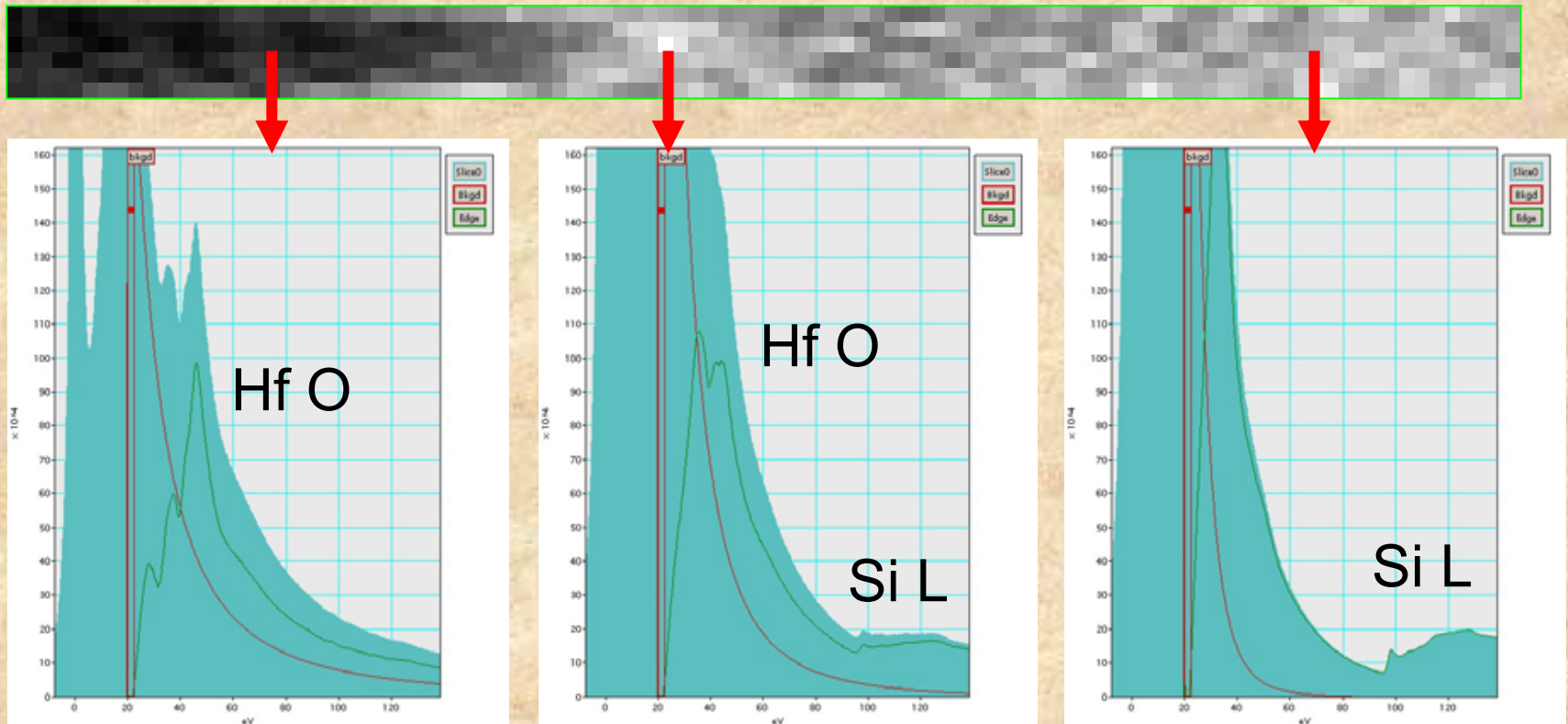




# Spot Analysis: O K edge



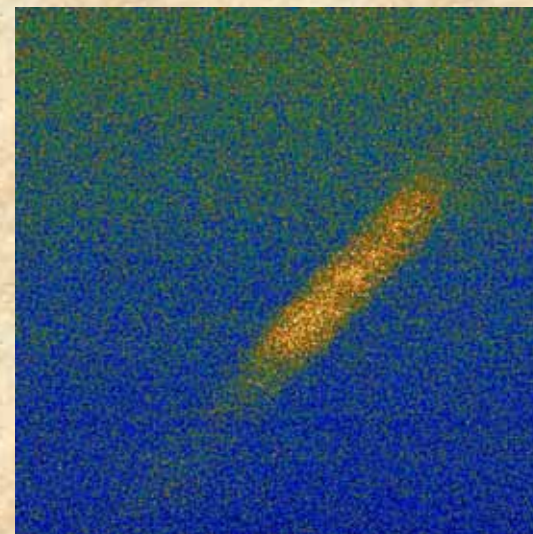
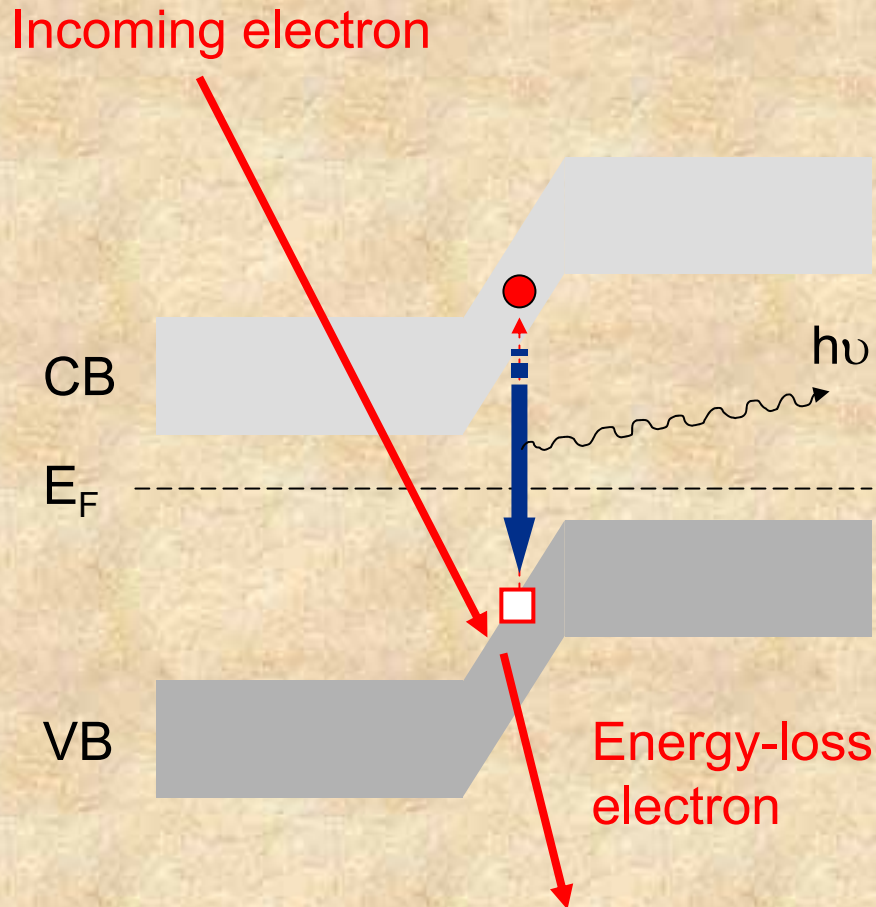
# Valence EELS = VEELS



- Local electronic structure (DOS)
- Local dielectric properties ( $\epsilon = \epsilon_1 + i\epsilon_2$ )
- Leakage paths (in 3D?)



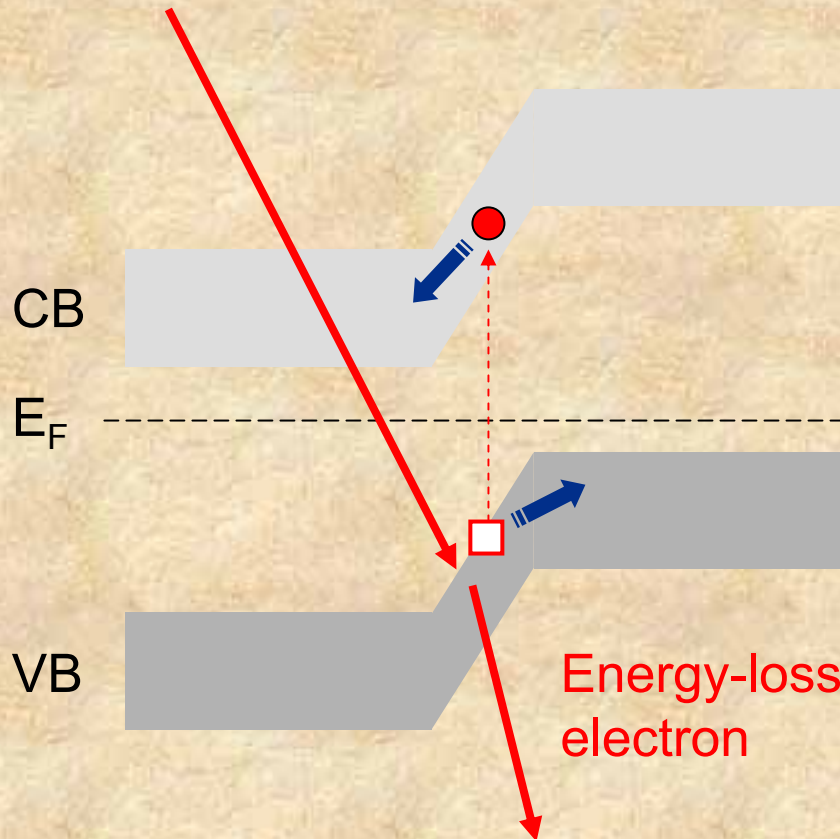
# Cathodoluminescence



ZnO(Mg) nanorods

# Electron Beam Induced Current

Incoming electron



**EBIC**

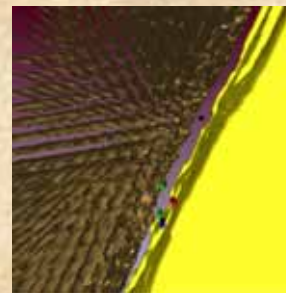
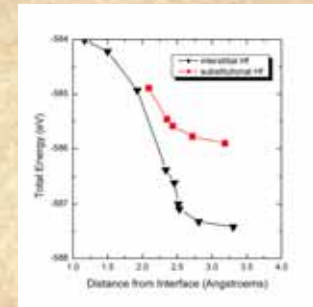
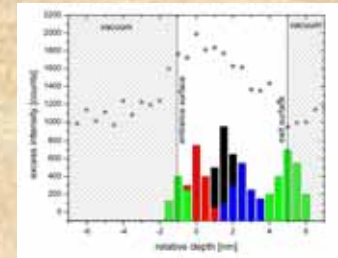
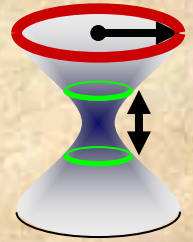
Detectable w/ and w/o  
applied potential

- Imaging contrast
- Charge Collection  
Microscopy (CCM)
- 3D-CCM



# Conclusions

- Volume resolution better than  $0.1 \times 0.1 \times 6 \text{ nm}^3$
- Single atom sensitivity in 3D
- Direct proof that dopant atoms are located inside the device
- Hf atoms stay away from the Si/SiO<sub>2</sub> interface
- Hf atoms occupy “interstitial” sites in SiO<sub>2</sub>
- Single Atom EELS (in 3D)
- Comparison of DOS and EELS/ELNES data
- VEELS
  - Local dielectric properties
  - Optical properties
- CL & EBIC



# Acknowledgements

- **A.Y. Borisevich, M.F. Chisholm, A.R. Lupini, Y. Peng, M. Varela**
- **S.N. Rashkeev, S.T. Pantelides (ORNL & Vanderbilt U)**
- **M. Kim et al. (Samsung, Korea)**
- **G. Duscher (NCSU)**
- **M.P. Oxley, S.D. Findlay, L.J. Allen (U Melbourne)**
- **J.T. Luck, W.H. Sides (technical support)**
- **Alexander-von-Humboldt Foundation (AvH)**
- **Oak Ridge Associated Universities (ORAU)**
- **Department of Energy (DOE)**