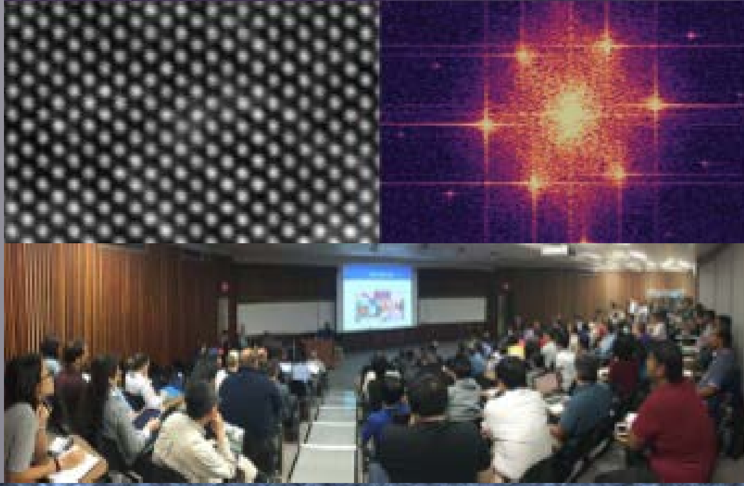
June 4 - 5, 2019

AMC

Advanced Materials Characterization Workshop



Transmission Electron Microscopy

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Materials Research Laboratory
University of Illinois

amc.mrl.Illinois.edu

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Outline

1. **Introduction to TEM**
2. **Basic Concepts**
3. **Basic TEM techniques**
   - Diffraction
   - Bright Field & Dark Field TEM imaging
   - High Resolution TEM Imaging
4. **Scanning Transmission Electron Microscopy (STEM)**
5. **Aberration-corrected STEM / TEM & Applications**
6. **Microspectroscopy & spectromicroscopy**
   - X-ray Energy Dispersive Spectroscopy
   - Electron Energy-Loss Spectroscopy
   - EFTEM and Spectrum Imaging
7. **Introduction to a New S/TEM: Themis Z**
8. **Summary**

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Why Use Transmission Electron Microscopy?

Optical Microscope

Resolution

$$d = \frac{\lambda}{2A_N}$$

A_N is 0.95 with air
up to 1.5 with oil

Resolution limit: ~200 nm

Transmission Electron Microscope (TEM)

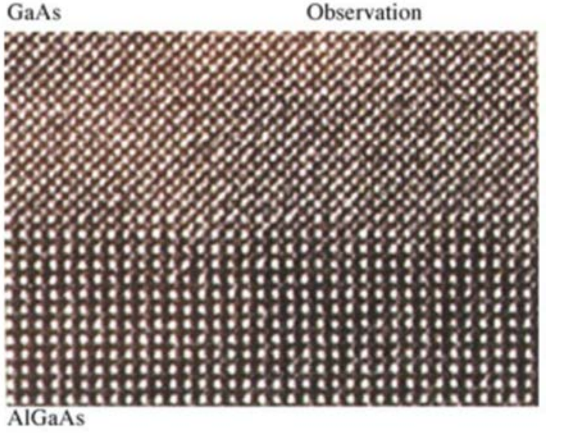
$\lambda = h / (2mE_k)^{1/2} \sim (1.5 / U)^{1/2}$ [nm]

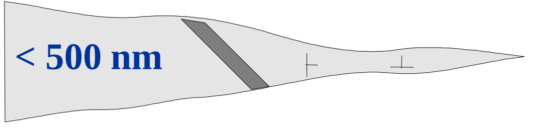
200 keV electrons: λ= 0.0027 nm

$R = 0.66(C_s\lambda^3)^{1/4}$

Resolution limit: ~0.15 nm (uncorrected)

High Resolution TEM





- **Sample thickness requirement:**
- **Thinner than 500 nm**
- **High quality image: <20 nm**

TEM is ideal for investigating thin foil, thin edge, and nanoparticles

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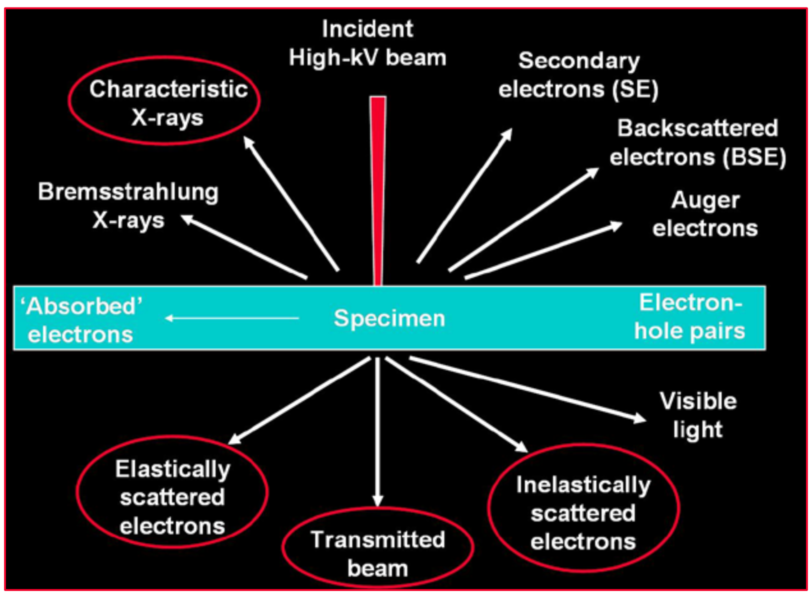
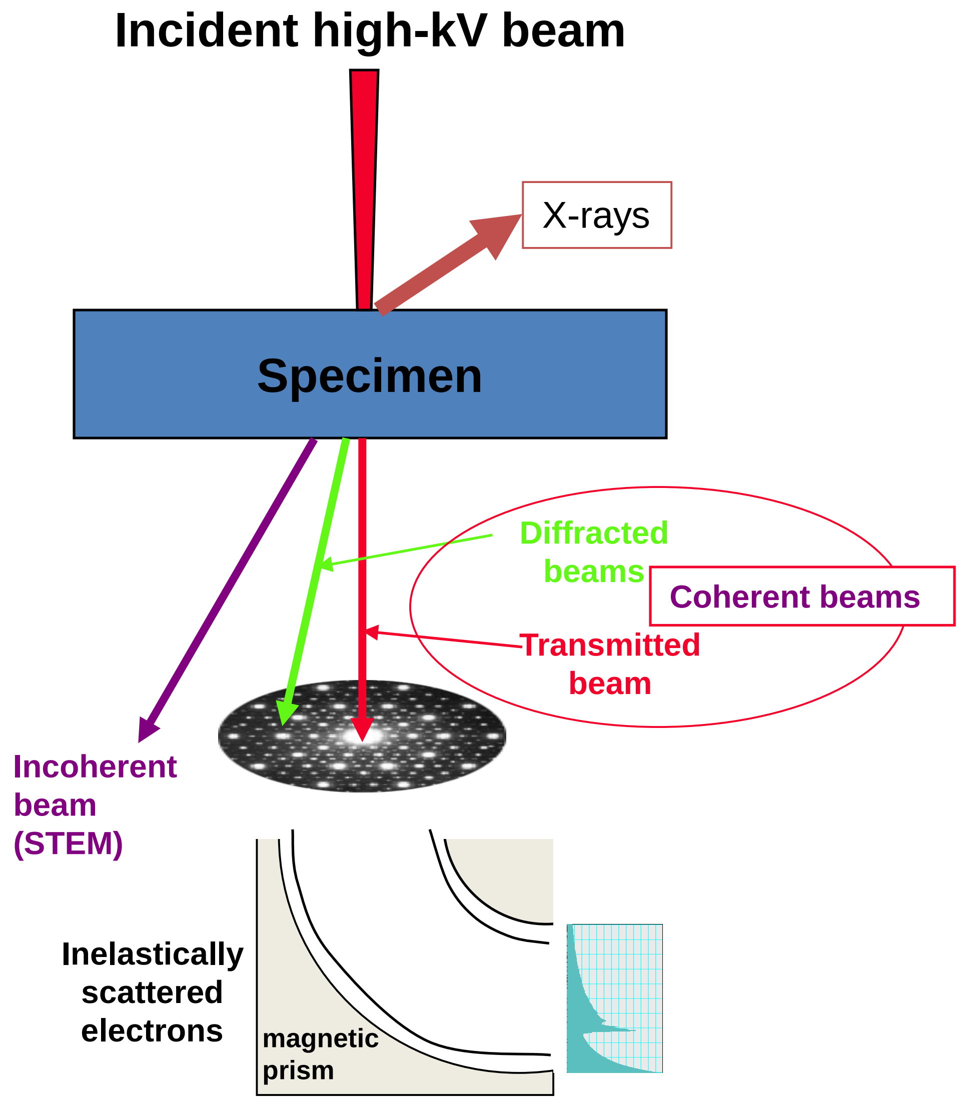
Basic Concepts – Electron-Sample Interaction

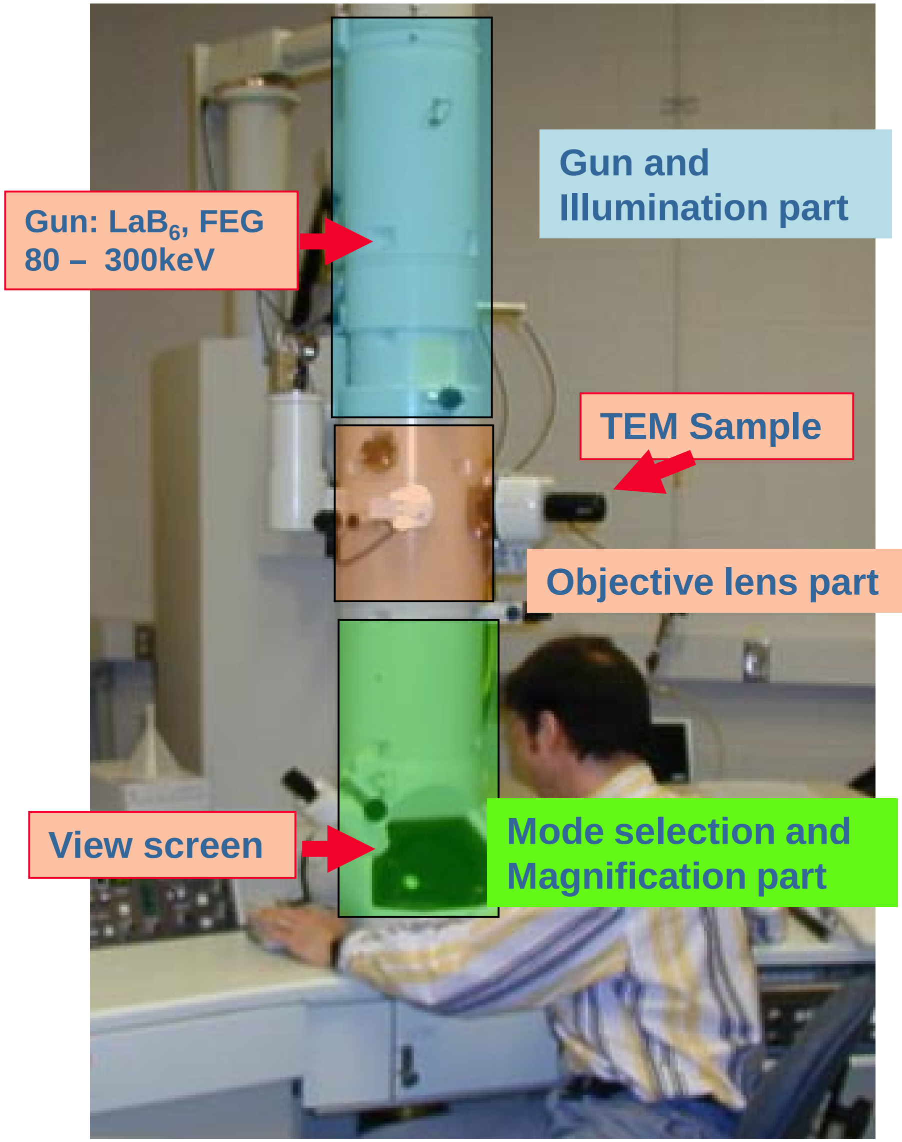
1. **Transmitted electrons (beam)**
2. **Diffracted electrons (beams)**
   (Elastically scattered)
3. **Coherent beams**
4. **Incoherent beams**
5. **Inelastically scattered electrons**
6. **Characteristic X-rays**

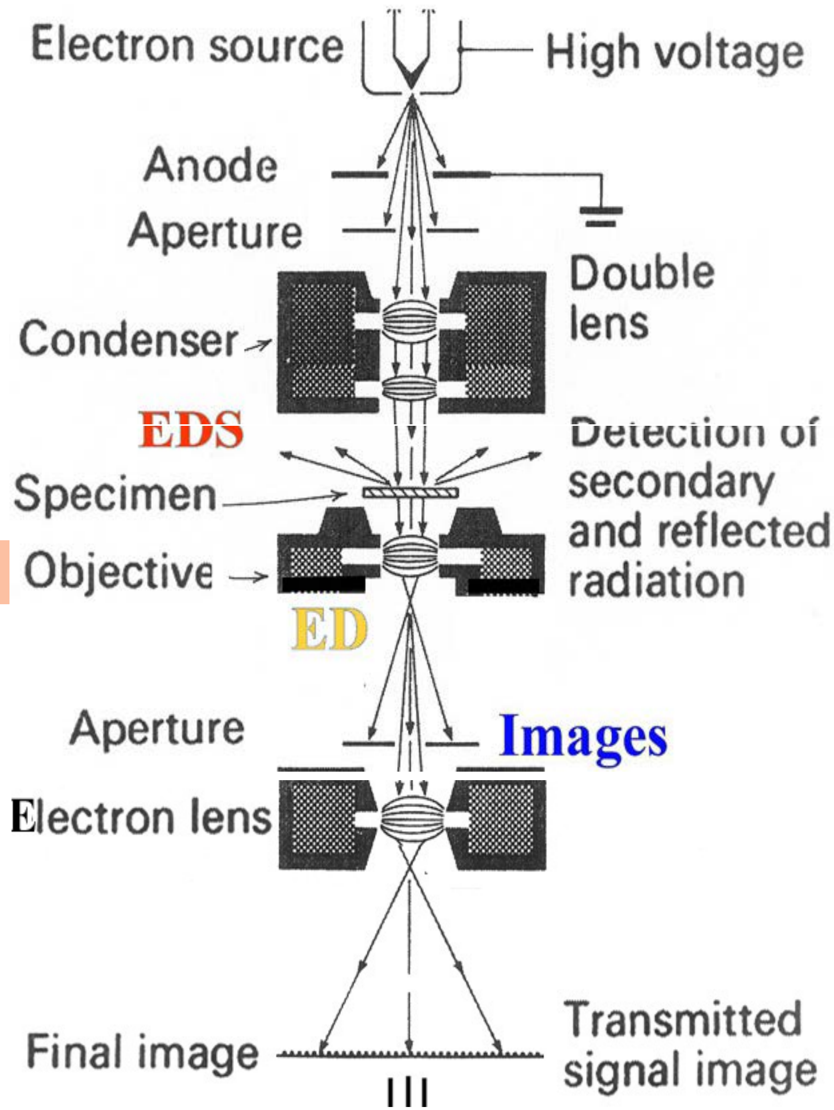




TEM can acquire images, diffraction patterns, spectroscopic and chemically sensitive images *at* ***resolution of 50 – 1000 of picometers.***

Structure of a TEM





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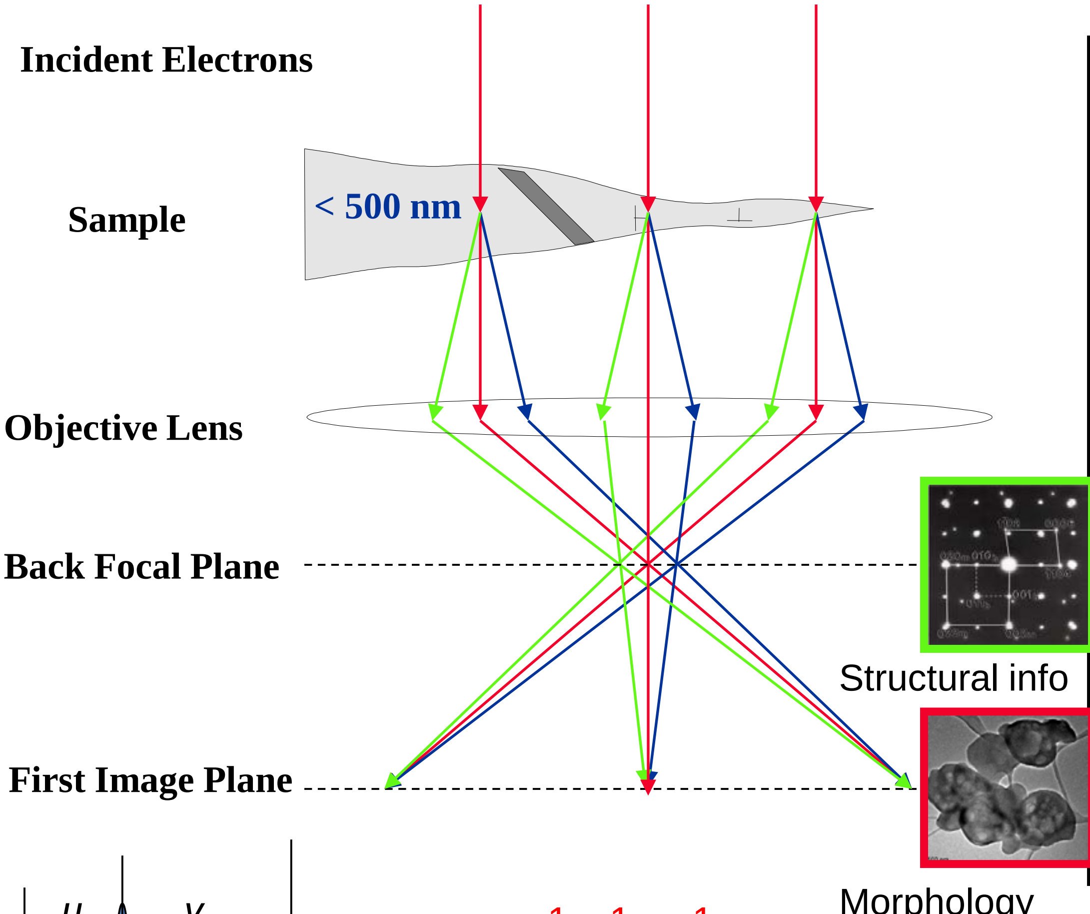
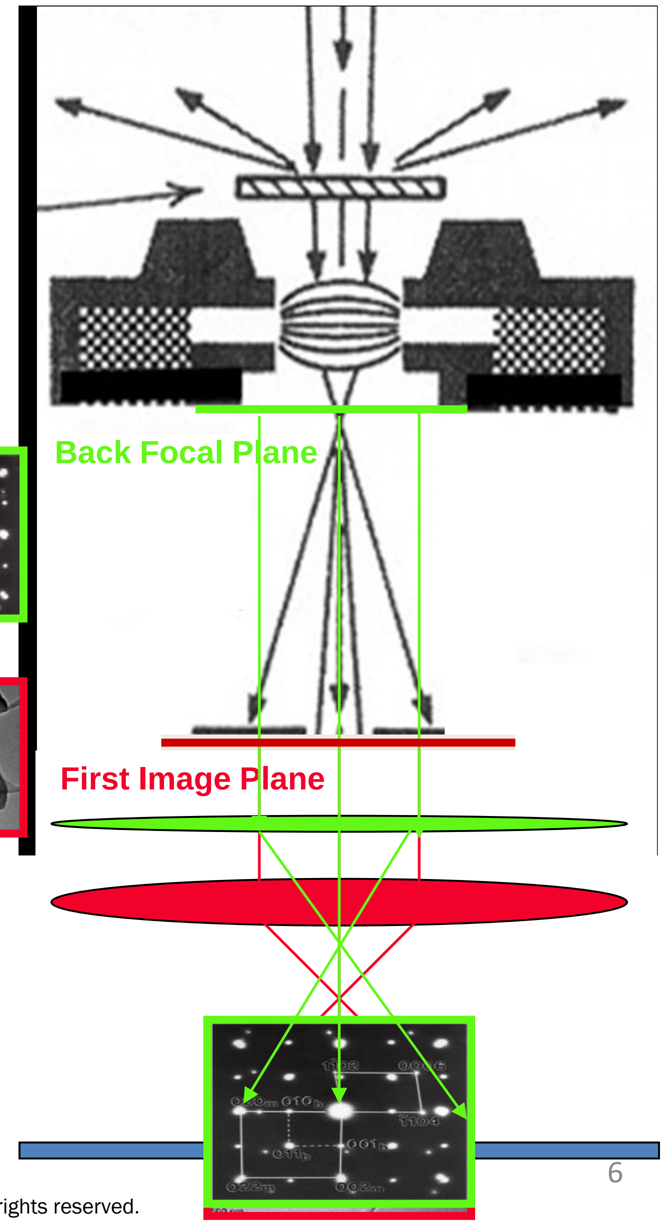
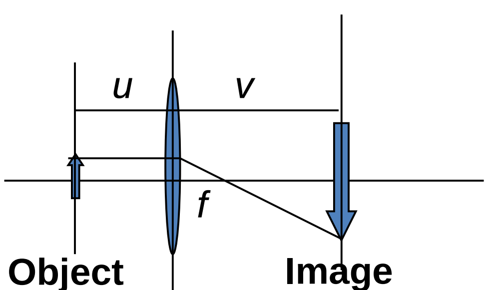
How does a TEM Obtain Image and Diffraction?





View Screen



Conjugate planes

$$\frac{1}{u}+\frac{1}{v}=\frac{1}{f}$$

Mag. = v/u

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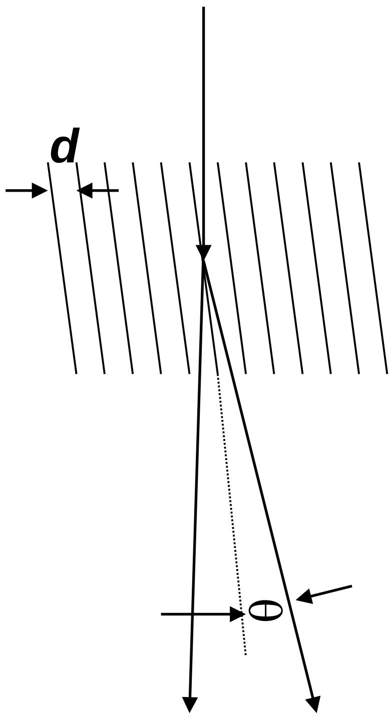


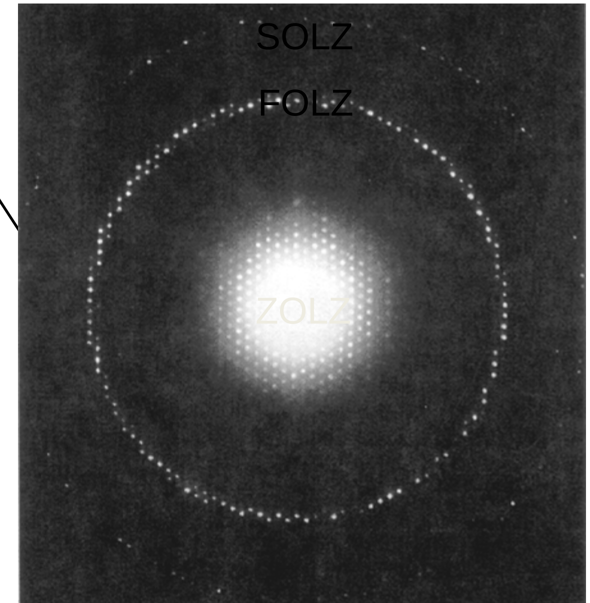
Bragg's Law

$2\, d \,\sin\theta = n\lambda$

Wavelength

X-rays: about 0.1nm

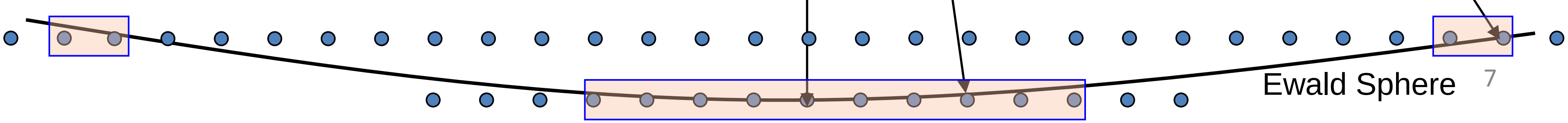
Electrons: 0.0027nm, @ 200kV



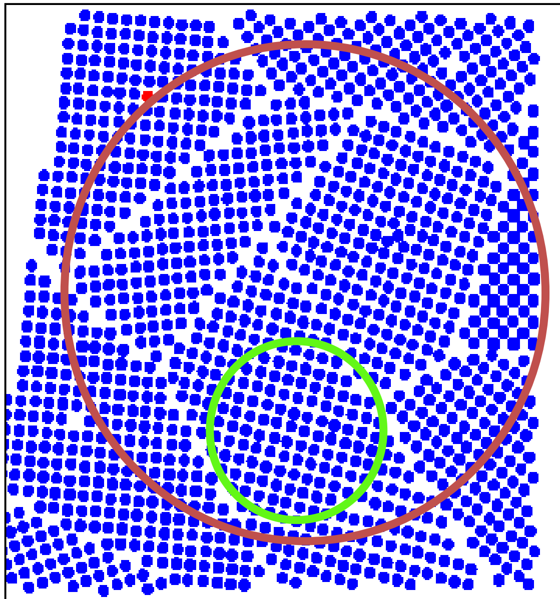
Zero-order Laue Zone (ZOLZ)
First-order Laue Zone (FOLZ)
….

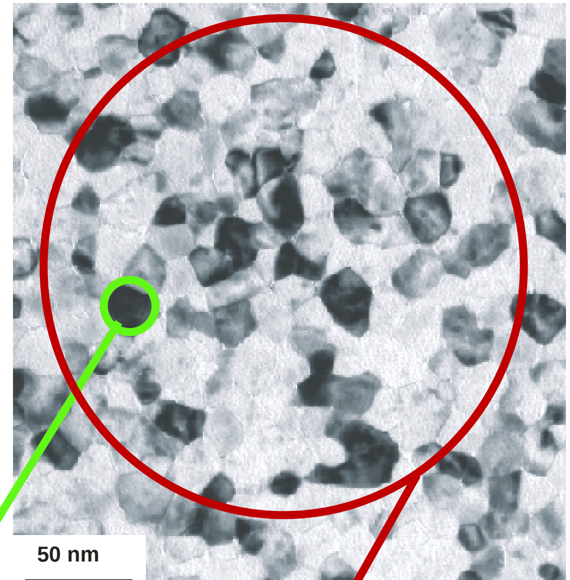
High-order Laue Zone (HOLZ)

λ is small, Ewald sphere ($1/\lambda$) is almost flat

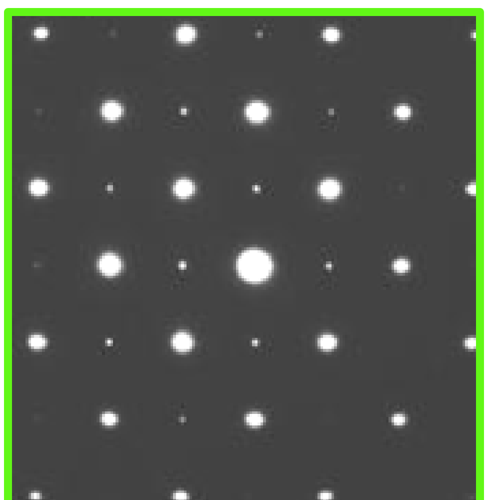


Electron Diffraction II

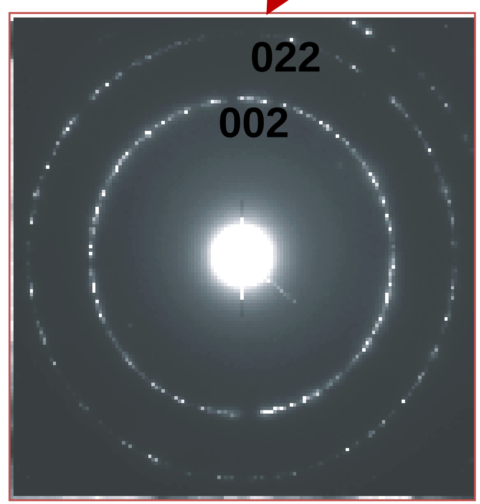




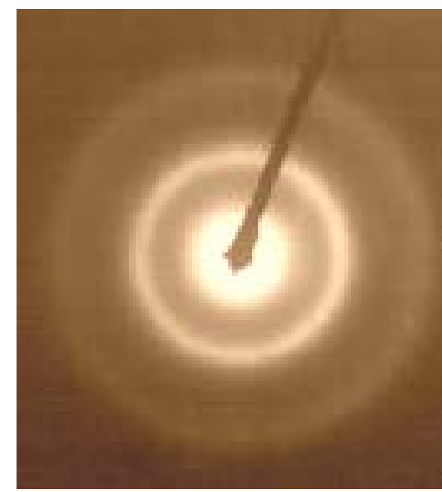
Diffraction patterns from single grain or multiple grains



Single crystal



Polycrystal



Amorphous

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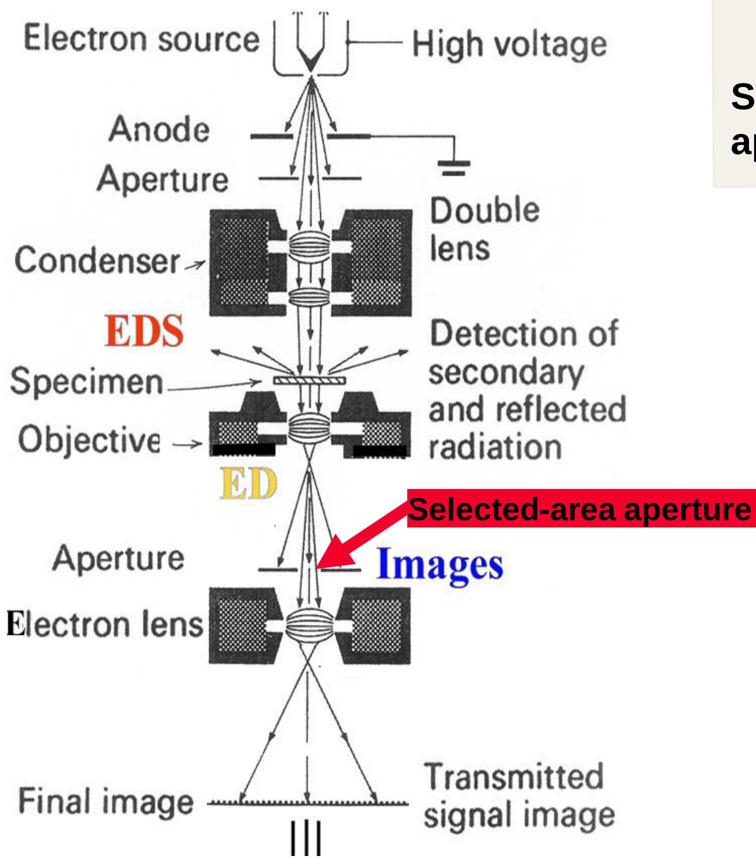
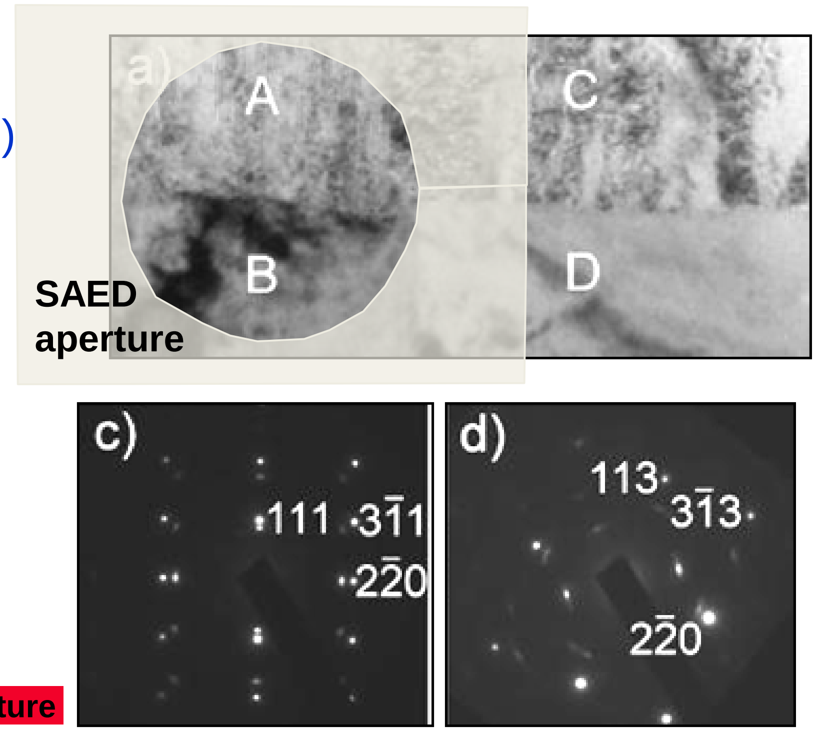
Major Diffraction Techniques

1) Selected-Area Electron Diffraction (SAED)
2) NanoArea Electron Diffraction (NBED)
3) Convergent Beam Electron Diffraction (CBED)



Example of SAED



J.G. Wen, A. Ehiasrian, I. Petrov

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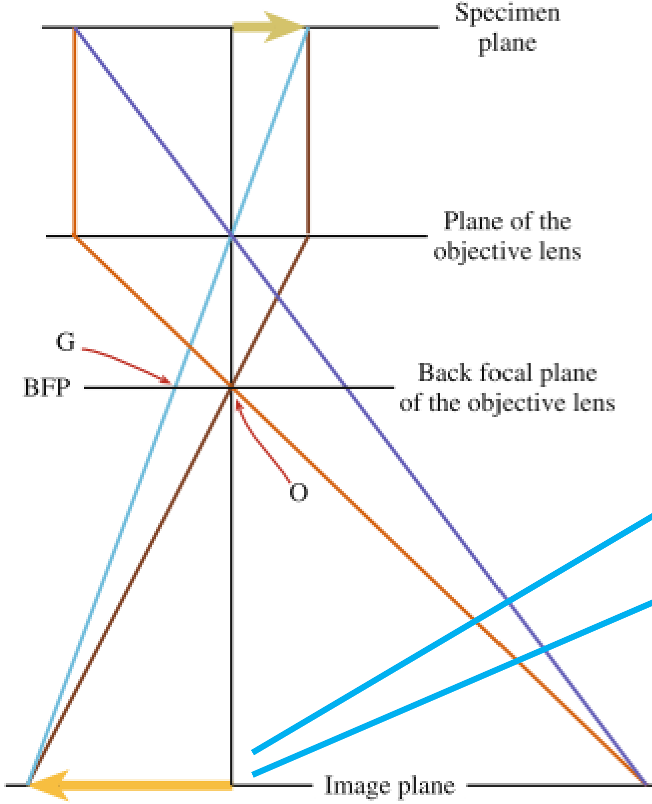
Selected Area Electron Diffraction (SAED)

1. Illuminate a large area of the specimen with a parallel beam.
2. Insert an aperture in the first image plane to select an area of the image.
3. Focus the first imaging lens on the OL Back Focal Plane



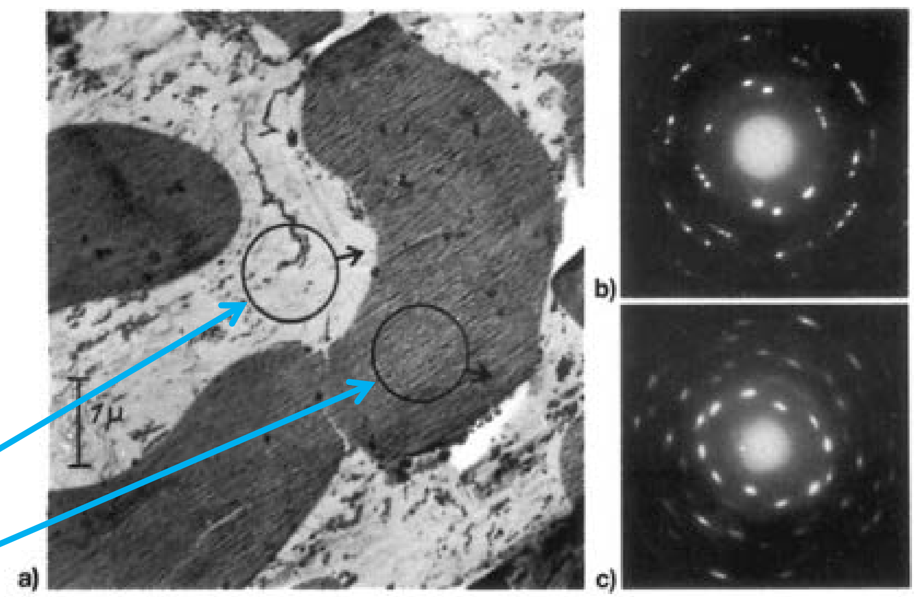


Fig. 8.1. Example of a selected-area electron diffraction (SAED) from a thin section of an Al-Cu eutectic cut with a microtome. (**b**) (Al matrix) and (**c**) ($AlCu_2$) contain the SAED pattern of the circles indicated in (**a**).

L. Reimer and H. Kohl, *Transmission Electron Microscopy, Physics of Image Formation*, 2008.

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Selected Area Electron Diffraction (SAED)

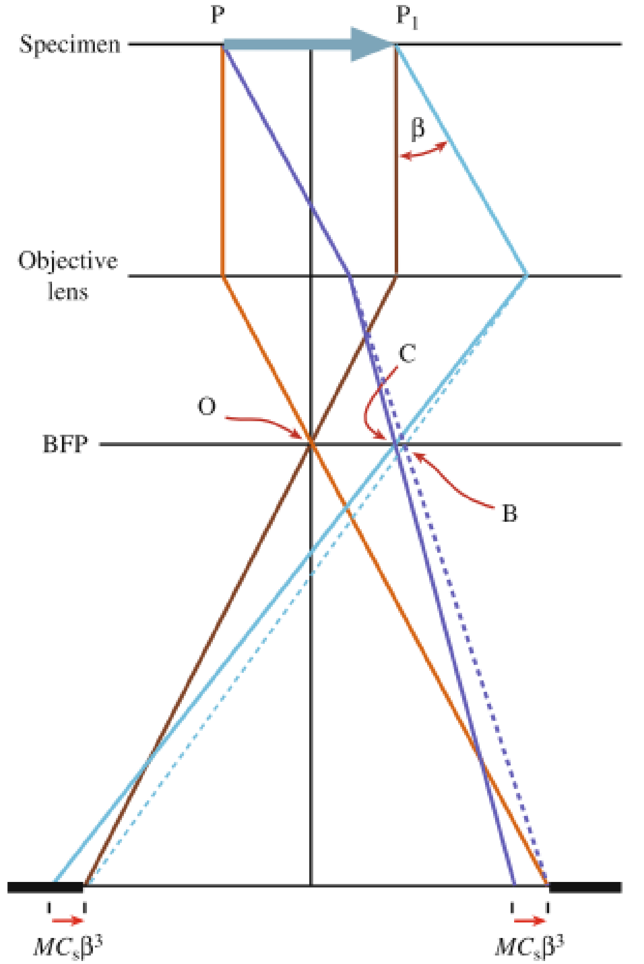
Advantages

1. Can observe a large area of specimen with a bright beam
2. Since the image plane is magnified, can easily select an area with an aperture → *A 50 μm aperture can select a 2 μm area*
3. Useful to study thick films, bulk samples, and in-situ phase transformations

Disadvantages

1. Cannot select an area less than ~1 μm due to spherical aberrations and precision in aperture position
2. Difficult to record diffraction from individual nanoparticles or thin films

Spherical Aberrations



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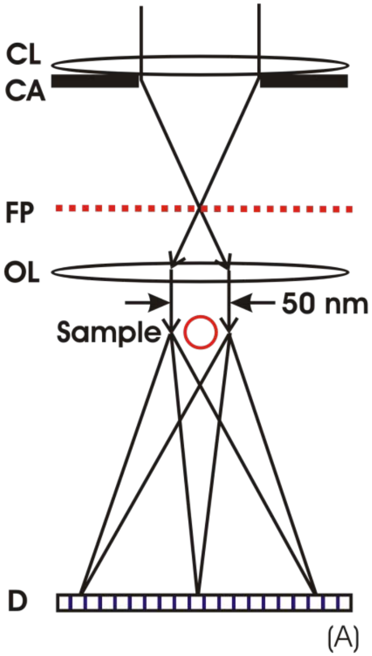
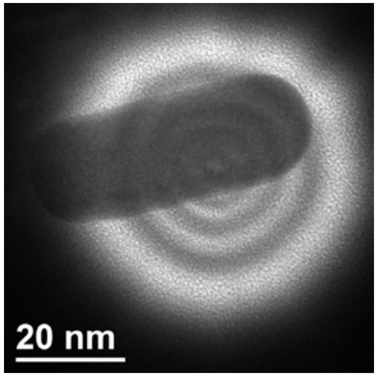
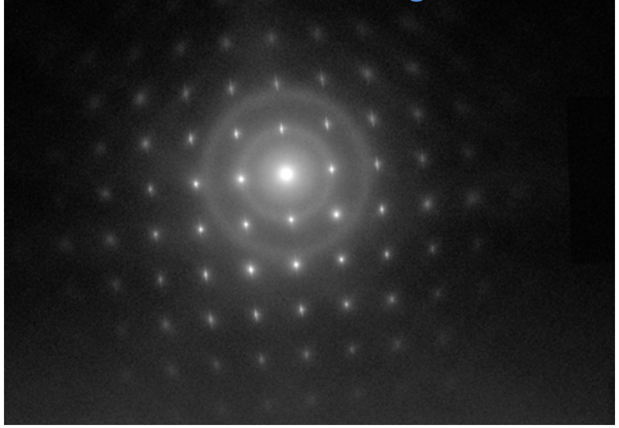
Nanoarea Electron Diffraction (NAED)





55 nm nano-probe

Diffraction from a single Au rod



A. B. Shah, S. Sivapalan, and C. Murphy

Technique

1. Focus the beam on the front focal plane of the objective lens and use a small condenser aperture to limit beam size
2. A parallel beam without selection errors → *A 10 μm aperture can form a ~ 50 nm probe size*
3. Useful to investigate individual nanocrystals and superlattices

Disadvantages

1. Very weak beam – *difficult to see and tilt the sample*
2. More complex alignment than conventional TEM
3. High resolution images are difficult to obtain → Need to switch between NAED and TEM modes)

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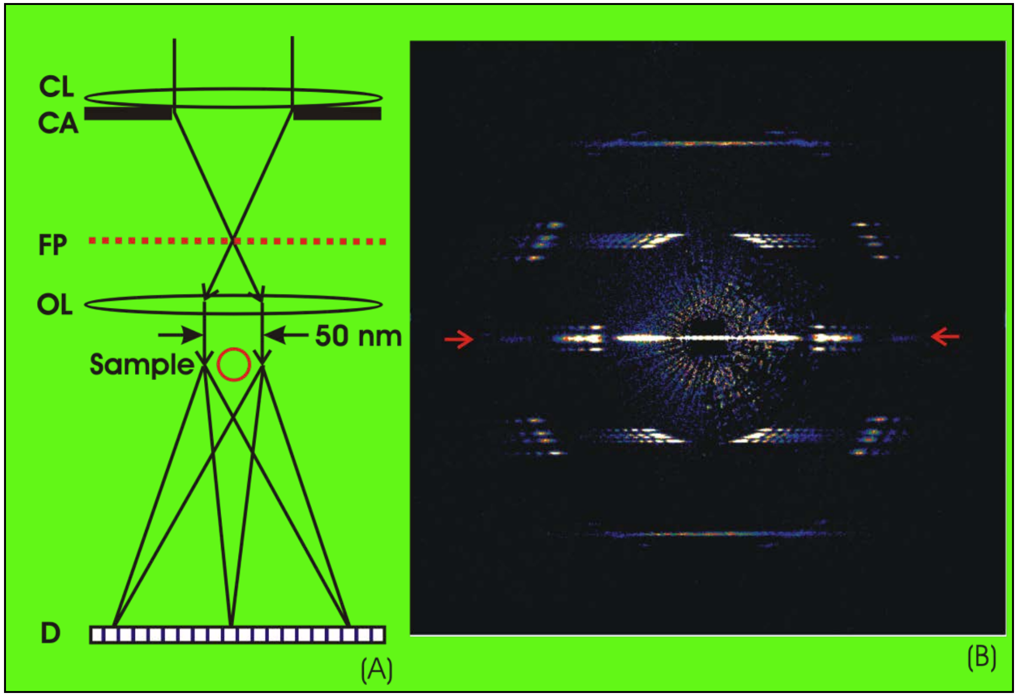
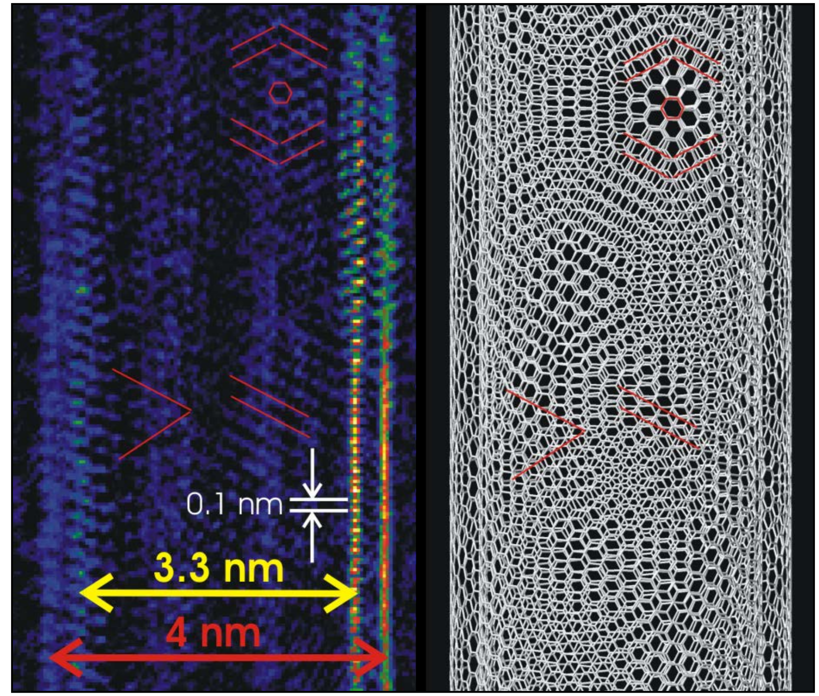
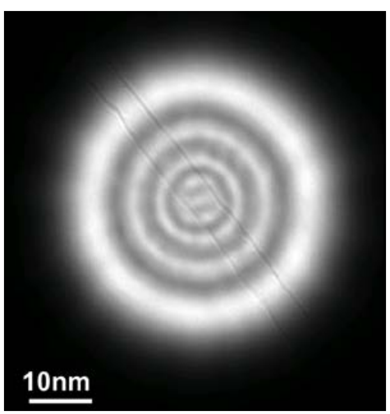
Nanoarea Electron Diffraction







5 μm condenser aperture → 30 nm

M. Gao, J.M. Zuo, R.D. Twesten, I. Petrov, L.A. Nagahara & R. Zhang, *Appl. Phys. Lett.* 82, 2703 (2003)

J.M. Zuo, I. Vartanyants, M. Gao, R. Zhang and L.A. Nagahara, *Science, 300,* 1419 *(2003)*

This technique was developed by CMM

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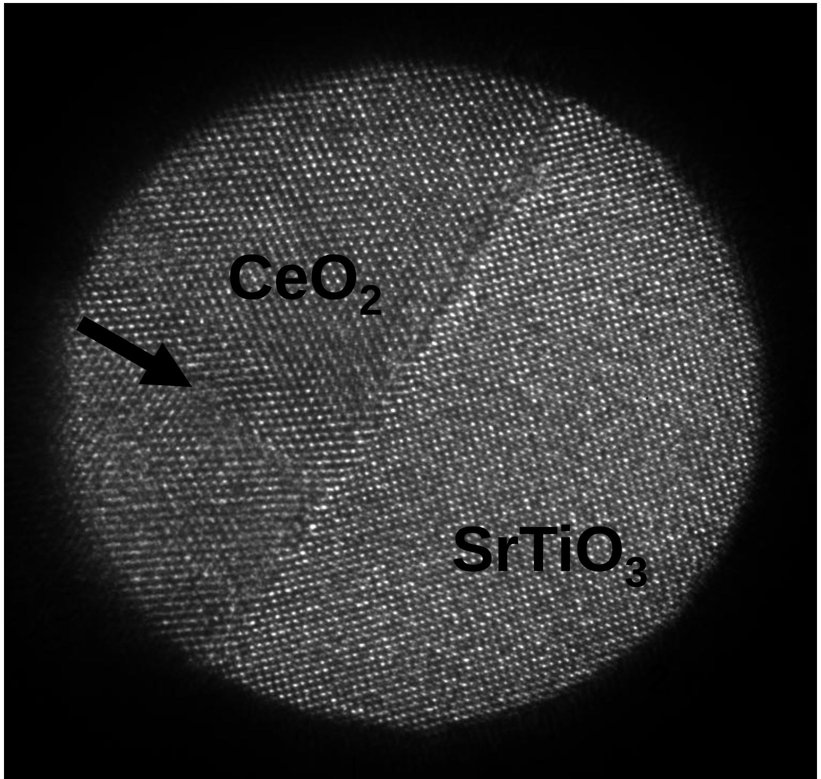
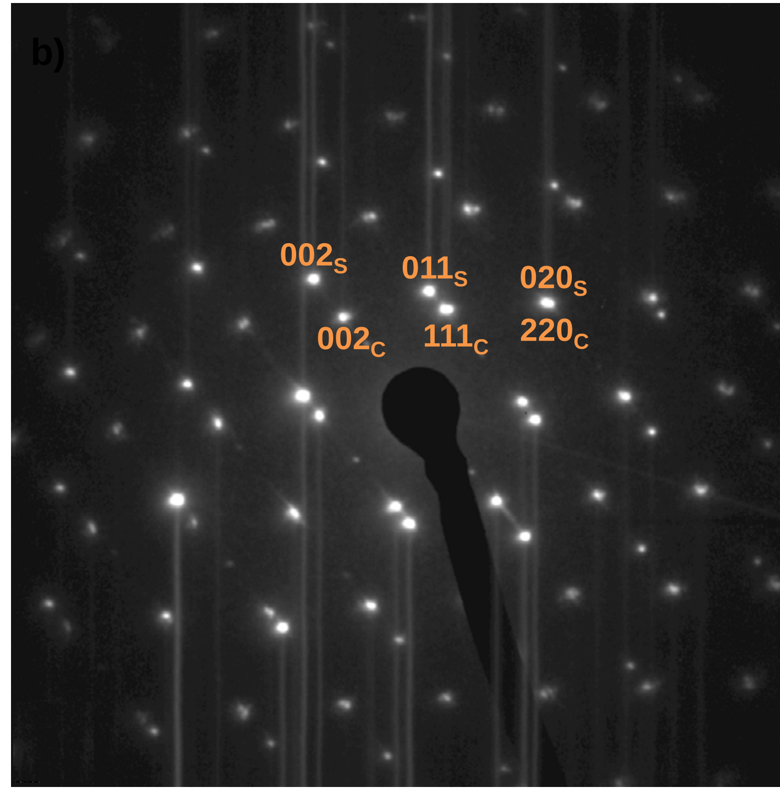
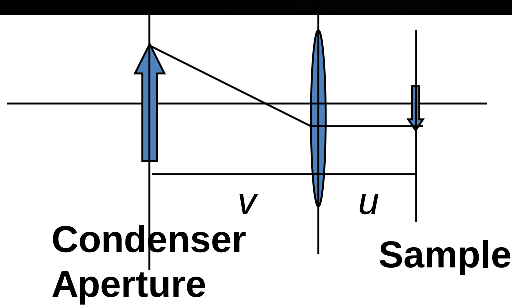
Aperture-Beam Nanoarea Electron Diffraction







Conjugate planes

20 μm condenser aperture → 20 nm probe size

This technique was developed by CMM

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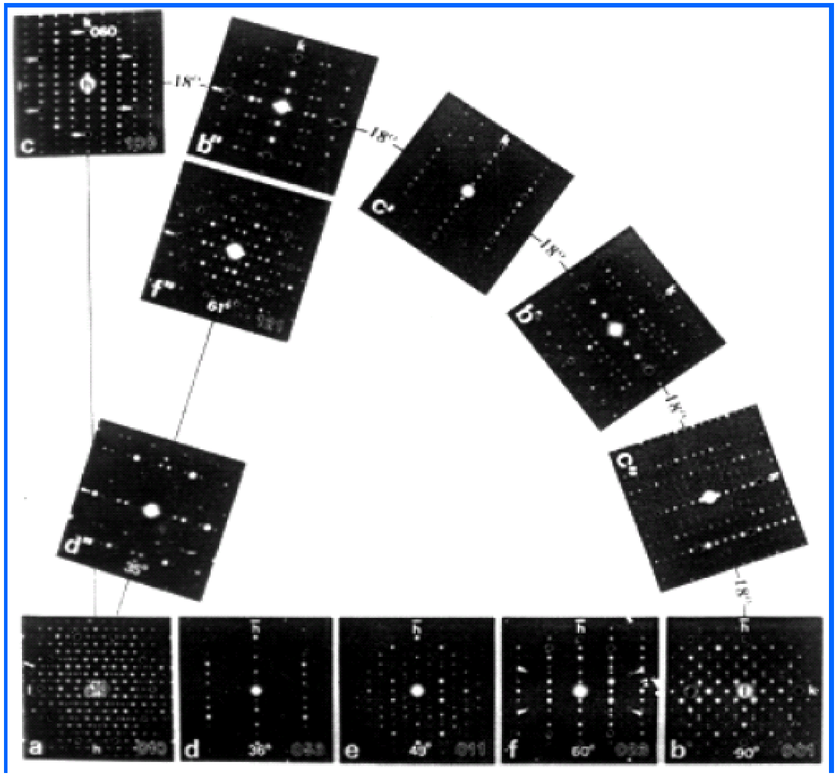
Applications of Electron Diffraction

Tilting sample to obtain 3-D structure of a crystal

Lattice parameter, space group, orientation relationship

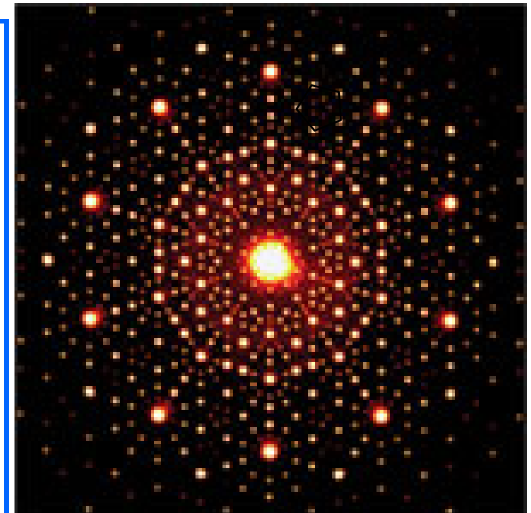


To identify new phases, TEM has advantages:
1) Small amount of materials
2) No need to be single phases
3) Determining composition by EDS or EELS

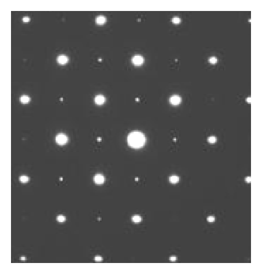
Disadvantage: needs experience

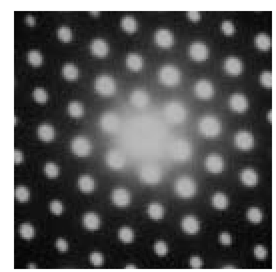
New materials discovered by TEM

Quasi-crystal



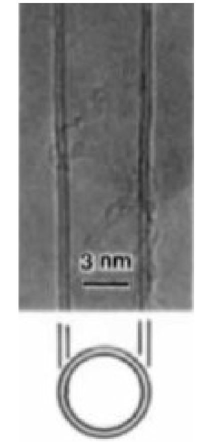
5-fold symmetry

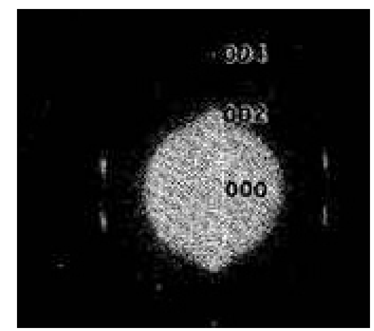




1, 2, 3, 4, 6-fold symmetry
No 5-fold for a crystal

Carbon Nanotube

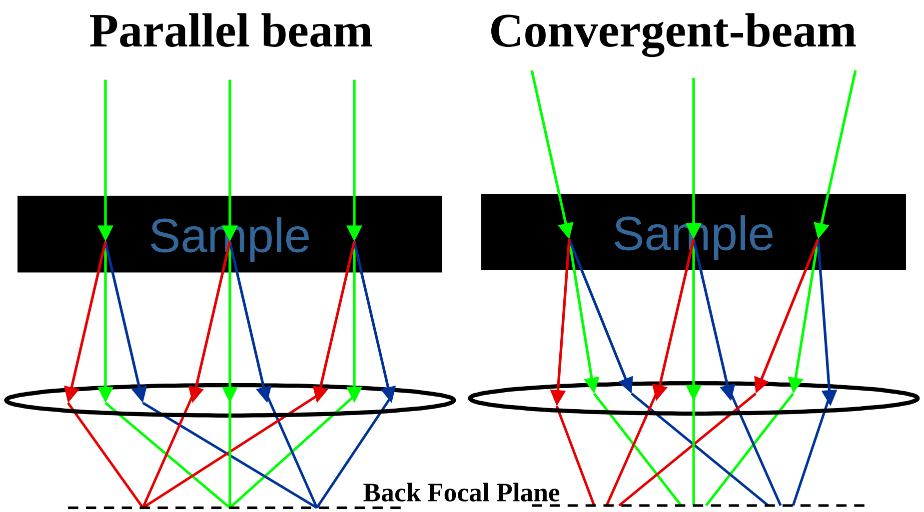


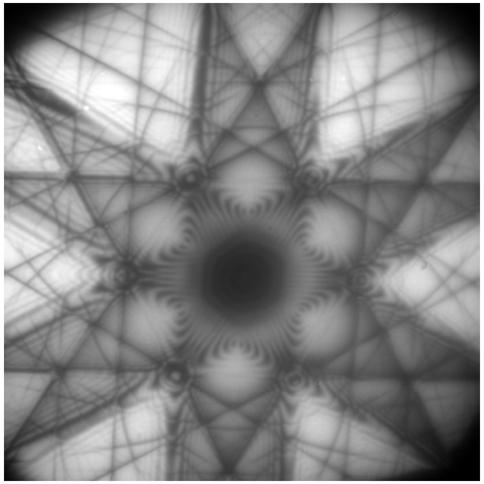


Helical graphene sheet

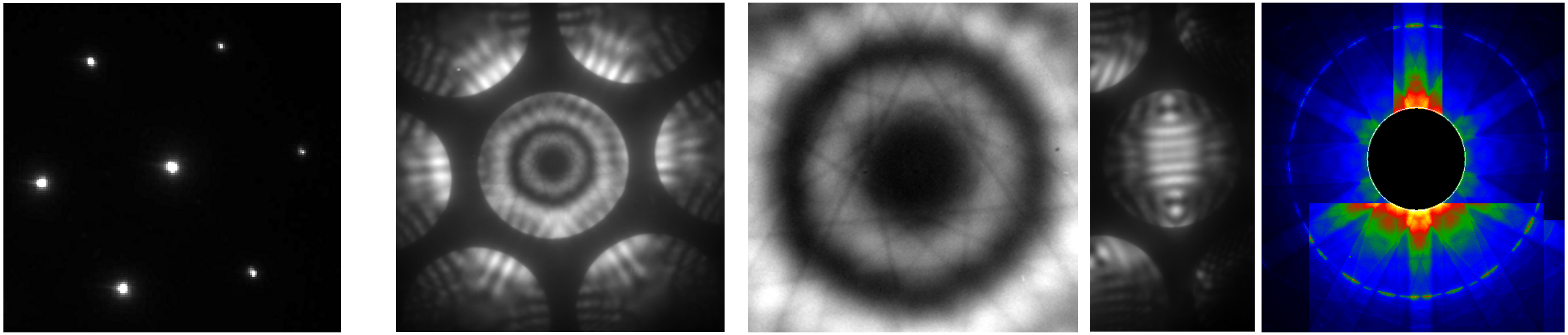
More advanced electron diffraction techniques

Convergent Beam Electron Diffraction (CBED)





Large-angle bright-field CBED



SAED CBED Bright-disk Dark-disk Whole-pattern

1. **Point and space group**
2. **Lattice parameter (3-D) strain field**
3. **Thickness**
4. **Defects**

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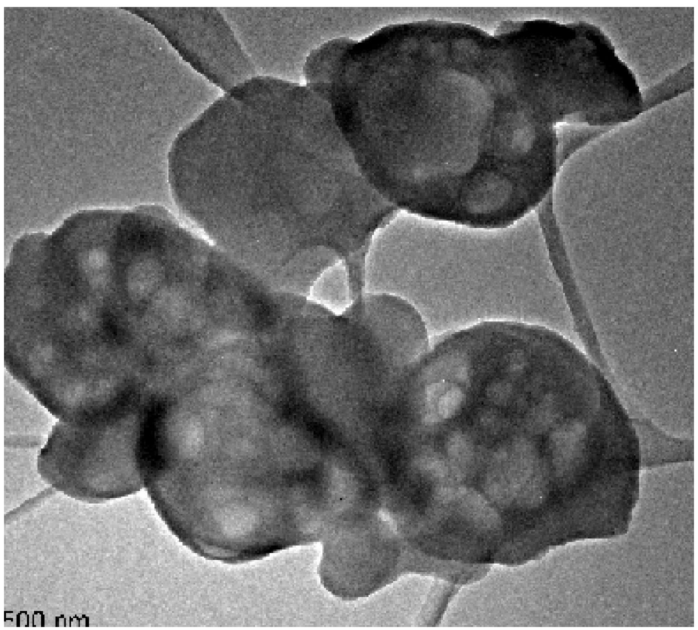
Major Imaging Techniques

Major Imaging Contrast Mechanisms:

1. Mass-thickness contrast
2. Diffraction contrast
3. Phase contrast
4. Z-contrast (S-TEM)



Mass-thickness contrast

1) Imaging techniques in **TEM** mode
   a) Bright-Field TEM (Diff. contrast)
   b) Dark-Field TEM (Diff. contrast)
   c) Weak-beam imaging
      hollow-cone dark-field imaging
   d) Lattice image (Phase)
   e) High-Resolution Electron Microscopy (Phase)
      Simulation and interpretation
2) Imaging techniques in **Scanning** Transmission Electron Microscope (**STEM**) mode
   1) Z-contrast imaging (Dark-Field)
   2) Bright-Field STEM imaging
   3) High-resolution Z-contrast imaging (Bright- & Dark-Field)
3) Spectrum imaging
   1) Energy-Filtered TEM (**TEM** mode)
   2) EELS mapping (**STEM** mode)
   3) EDS mapping (**STEM** mode)

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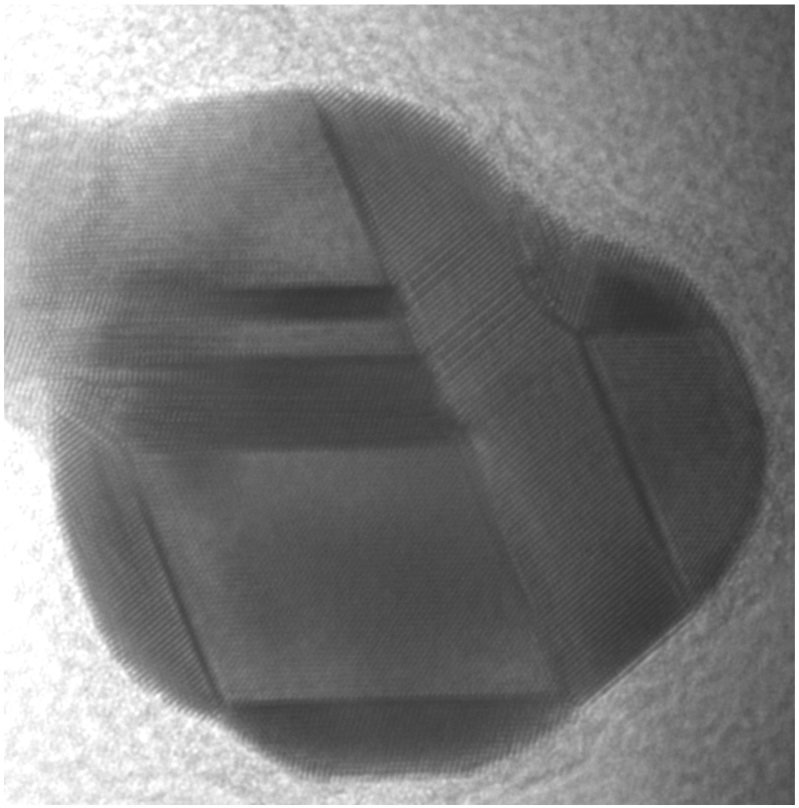
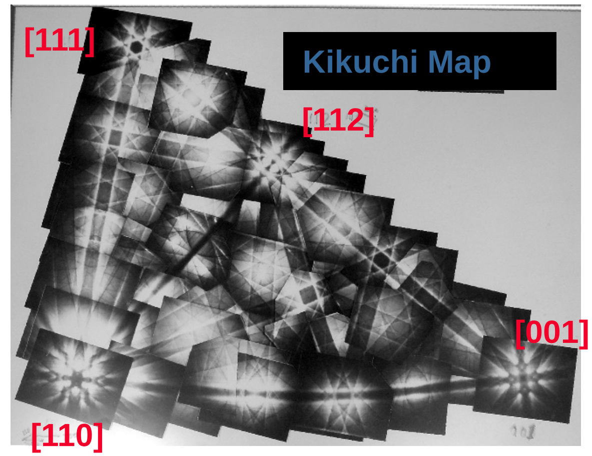
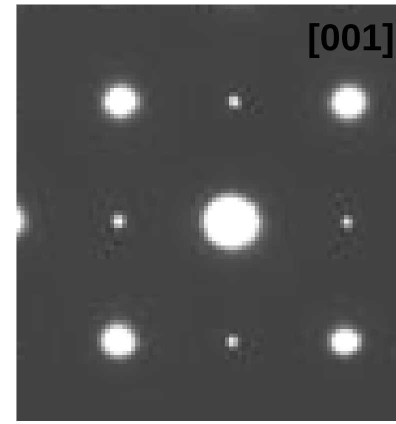
Phase Contrast Image



I. Diffraction Contrast Image: Contrast related to crystal orientation

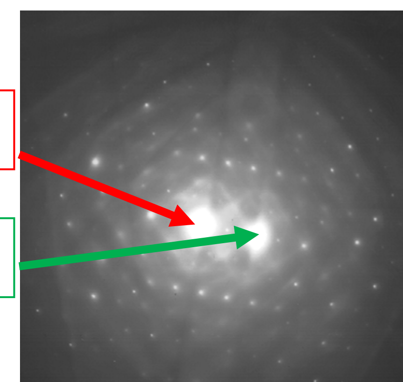




Two-beam condition

Transmitted beam

Diffracted beam



Application:
Morphology, defects, grain boundary, strain field, precipitates

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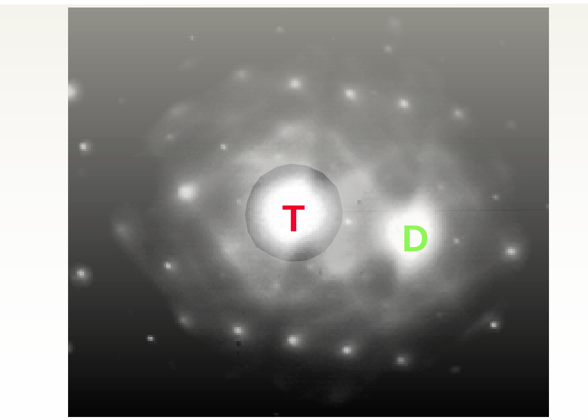
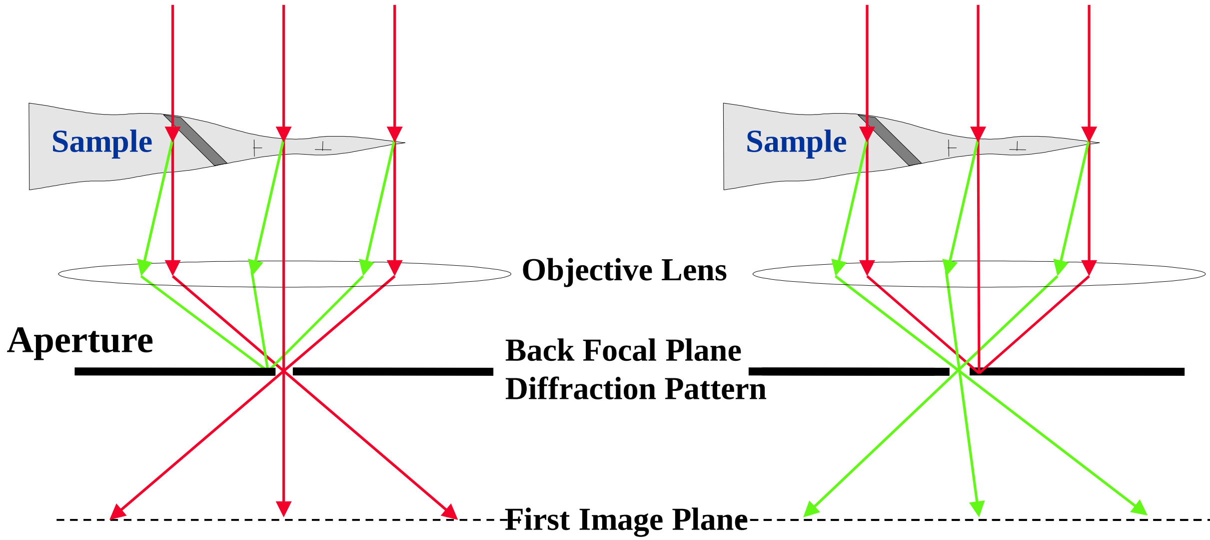
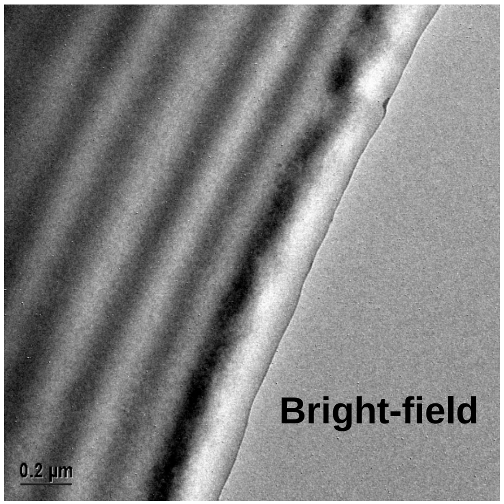
II. Diffraction Contrast Image: Bright-field & Dark-field Imaging



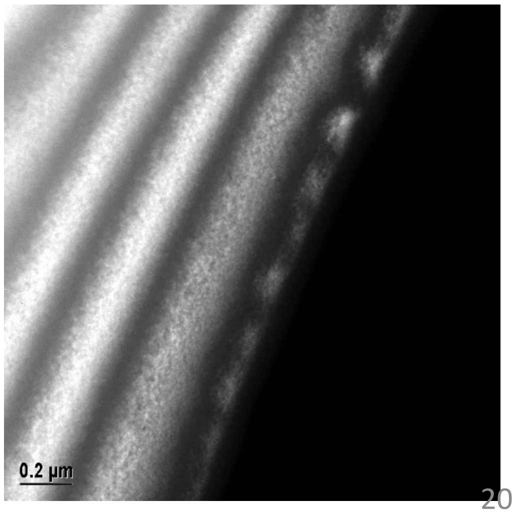
Two-beam condition



Bright-field Image



Dark-field Image



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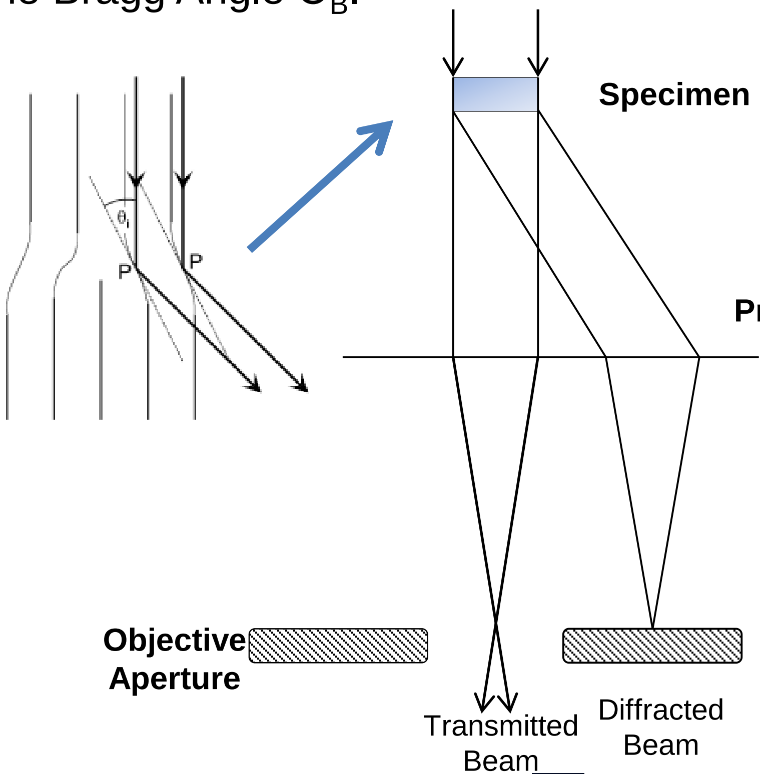
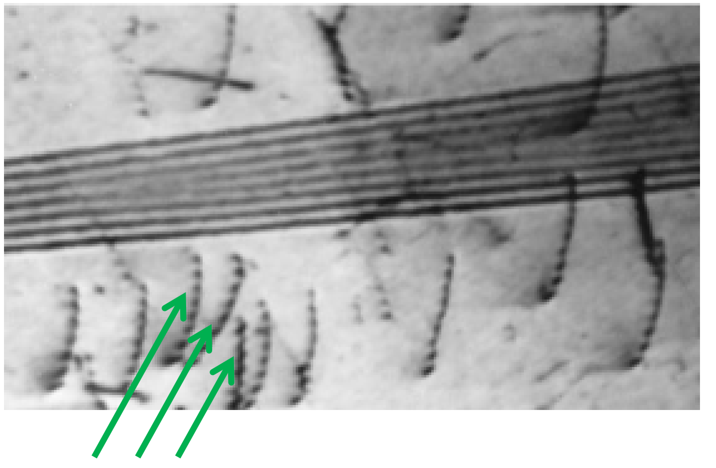
II. Diffraction Contrast Imaging

At edge dislocation, strain from extra half plane of atoms causes atomic planes to bend. The angle between the incident beam and a few atomic planes becomes equal to the Bragg Angle Θ_B.



Dislocations & Stacking Faults

Bright Field Image



Near dislocations, electrons are strongly diffracted outside the objective aperture

R. F. Egerton, *Physical Principles of Electron Microscopy*, 2007.

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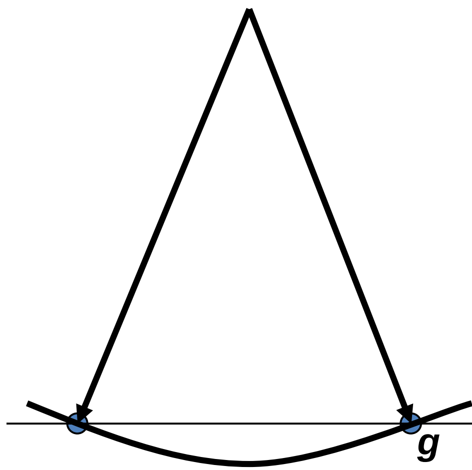
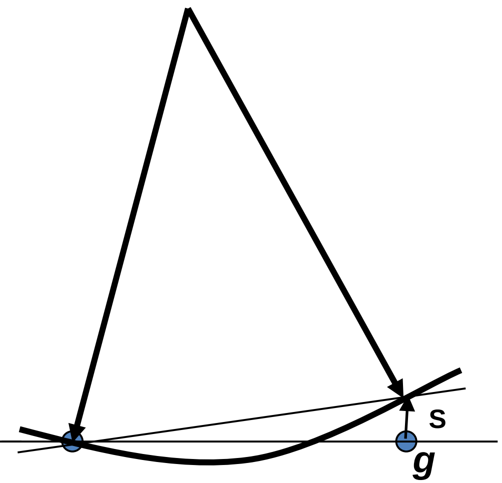
Weak-beam Dark Field Imaging

High-resolution dark-field imaging

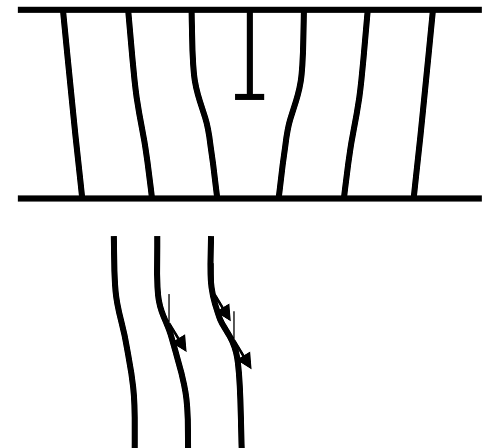
"Near Bragg Condition"



Exact Bragg condition



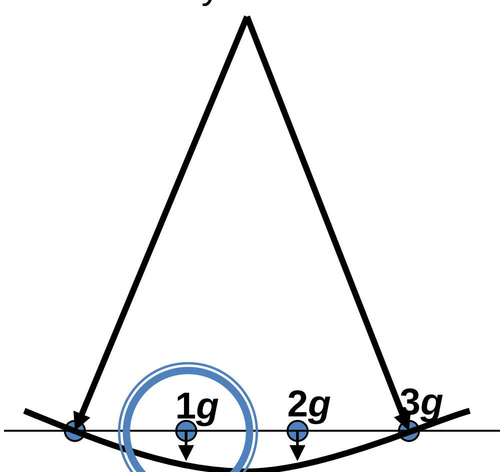
Weak-beam means
Large excitation error



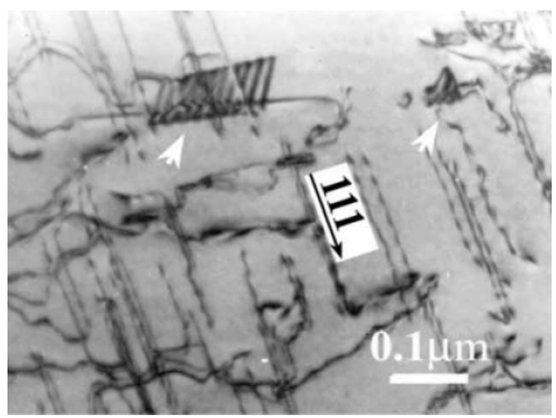
Planes do not satisfy Bragg diffraction

Possible planes satisfy Bragg diffraction

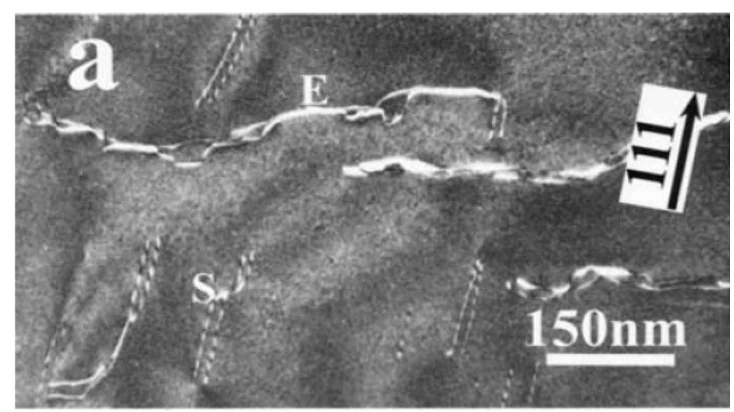
Taken by I. Petrov



Experimental weak-beam



Bright-field



Weak-beam

Dislocations can be imaged as 1.5 nm narrow lines

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II. Diffraction Contrast Image

Two-beam condition for defects

Howie-Whelan equation

$$\frac{d\phi_0}{dz} = \frac{\pi i}{\xi_g}\phi_g \exp\{2\pi i(sz+\boldsymbol{g}.\mathbf{R})\}$$

Dislocations

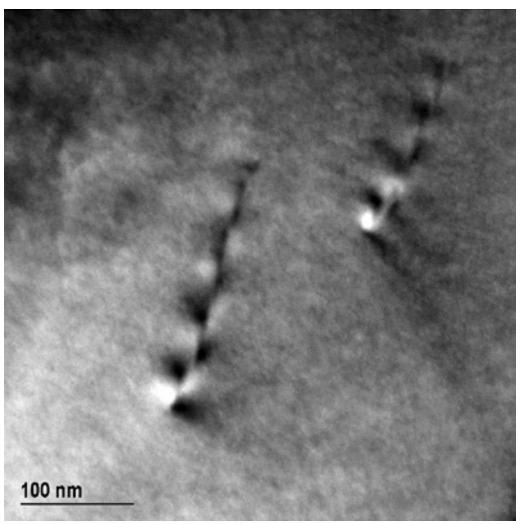
Use **g.b** = 0 to determine Burgers vector **b**

Stacking faults

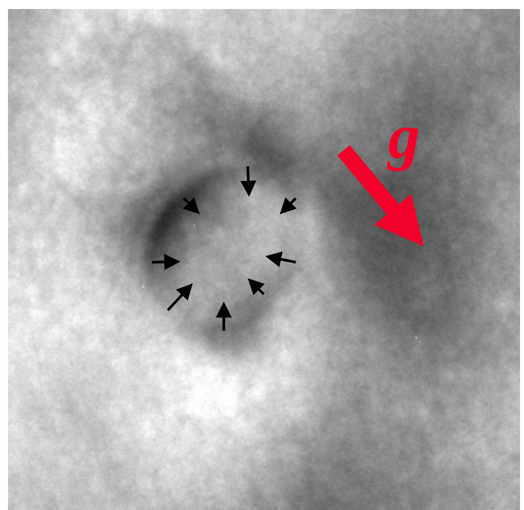
Phase = 2 π *g* • R

Each stacking fault changes phase $\frac{2}{3}\pi$

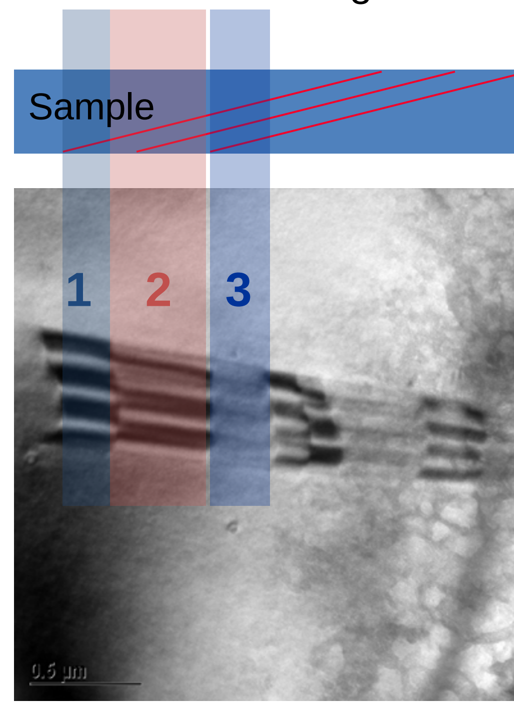
Diffraction contrast images of typical defects



Dislocations



Dislocation loop



Stacking faults

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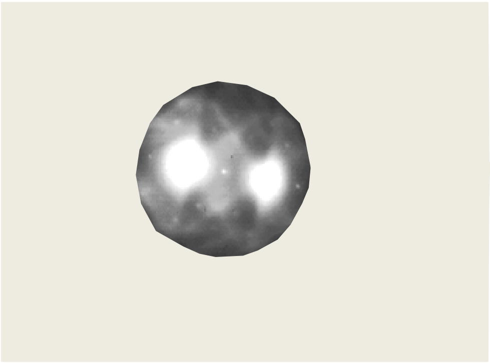
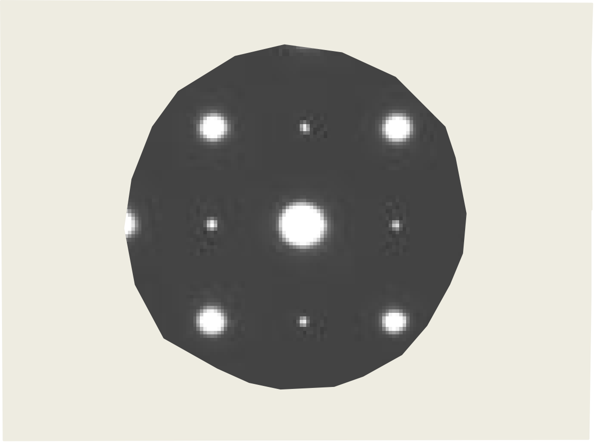
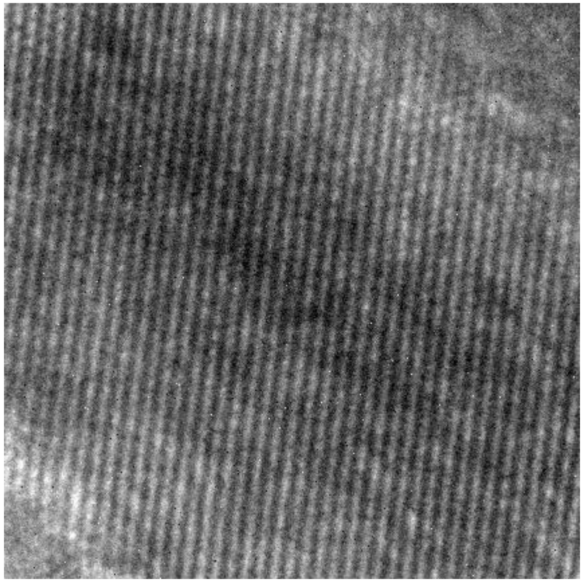
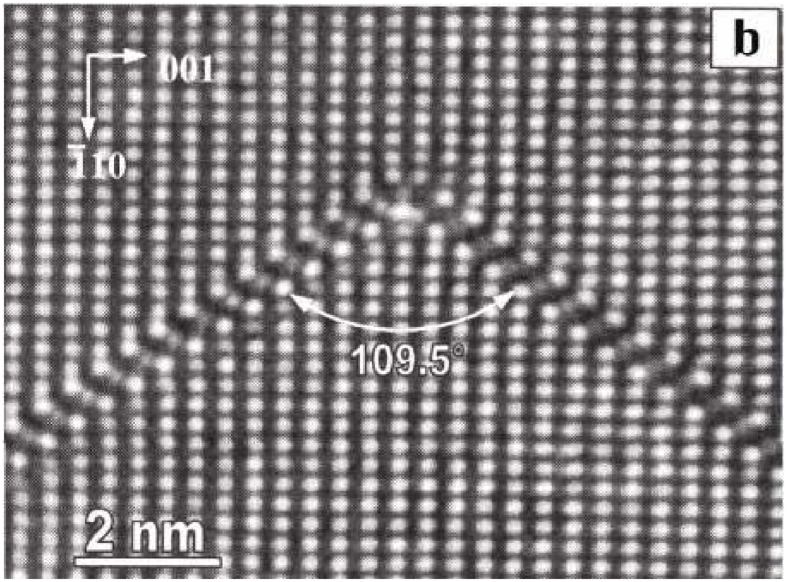
Two-beam condition | **Many-beam condition**









M. Marshall | **C.H. Lei**

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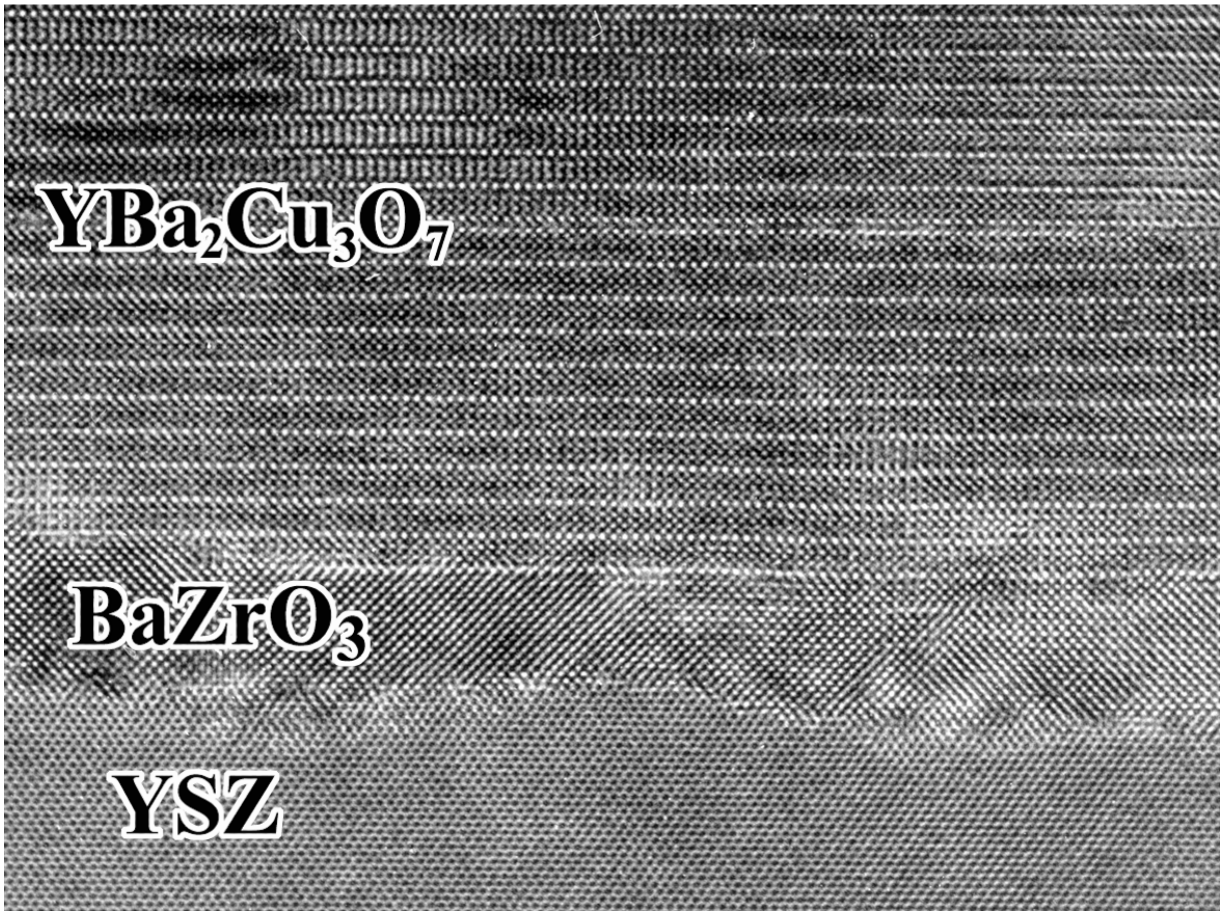
Lattice Imaging

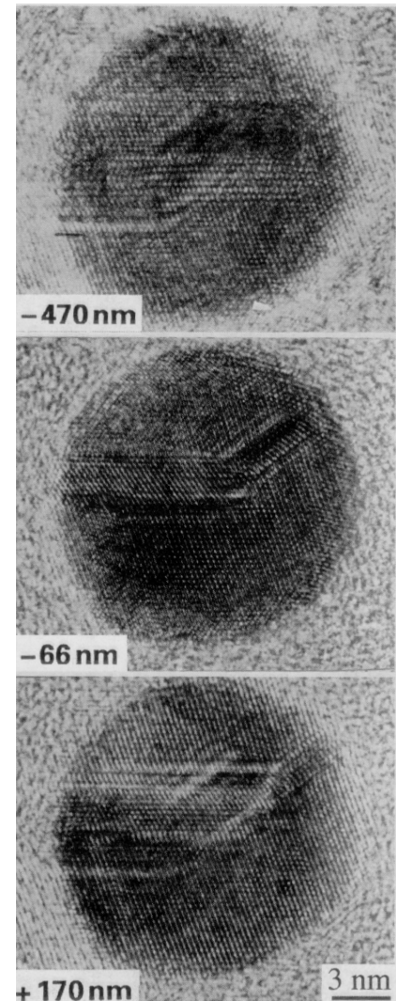
Delocalization effect from a Schottky-emission gun (S-FEG)

From a LaB_6 Gun



Lattice image of film on substrates

S-Field-Emission Gun



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High Resolution Transmission Electron Microscopy (HRTEM)

$f(x,y) = \exp(i\sigma V_t(x,y))$

$\sim 1 + i\,\sigma\, V_t(x,y)$

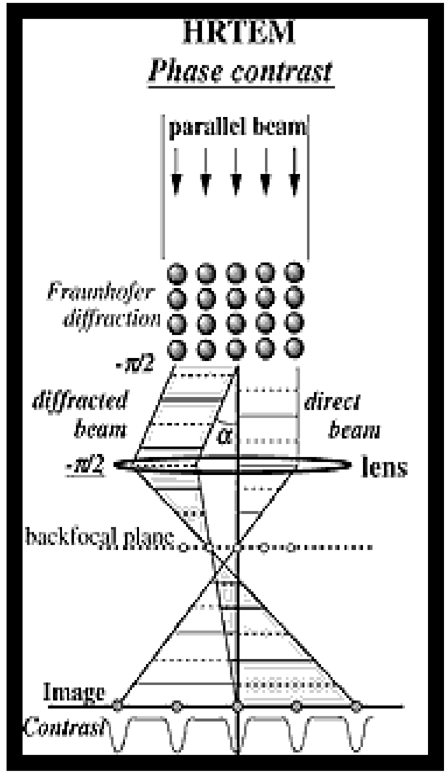
$V_t(x,y)$: projected potential

Scherzer defocus

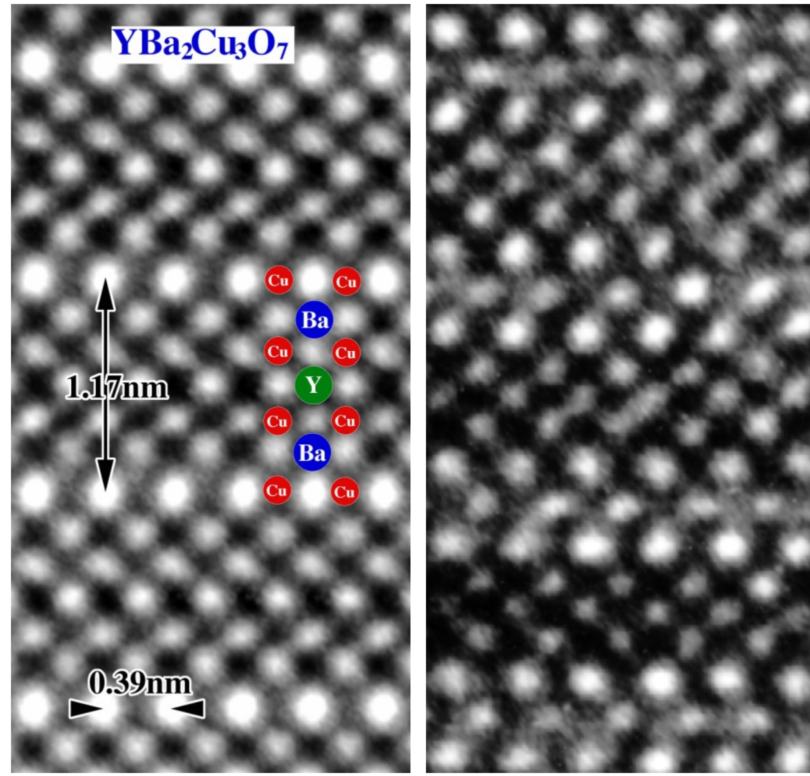
$$\Delta f_{sch} = -\,1.2\,(C_s\lambda)^{\frac{1}{2}}$$

Resolution limit

$$r_{sch} = 0.66\, C_s^{\frac{1}{4}}\, \lambda^{\frac{3}{4}}$$



1 Δf_{sch} 2 Δf_{sch}

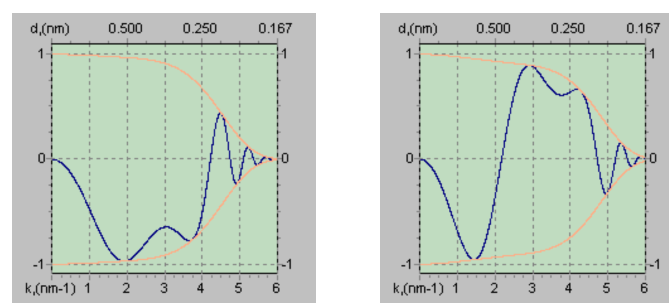


1 Scherzer Defocus:
Positive phase contrast “black atoms”
2 Scherzer Defocus: ("2nd Passband" defocus).
Contrast Transfer Function is positive
Negative phase contrast ("white atoms")

Simulation of images Software: Web-EMAPS (UIUC)
MacTempas

Contrast transfer function

J.G. Wen



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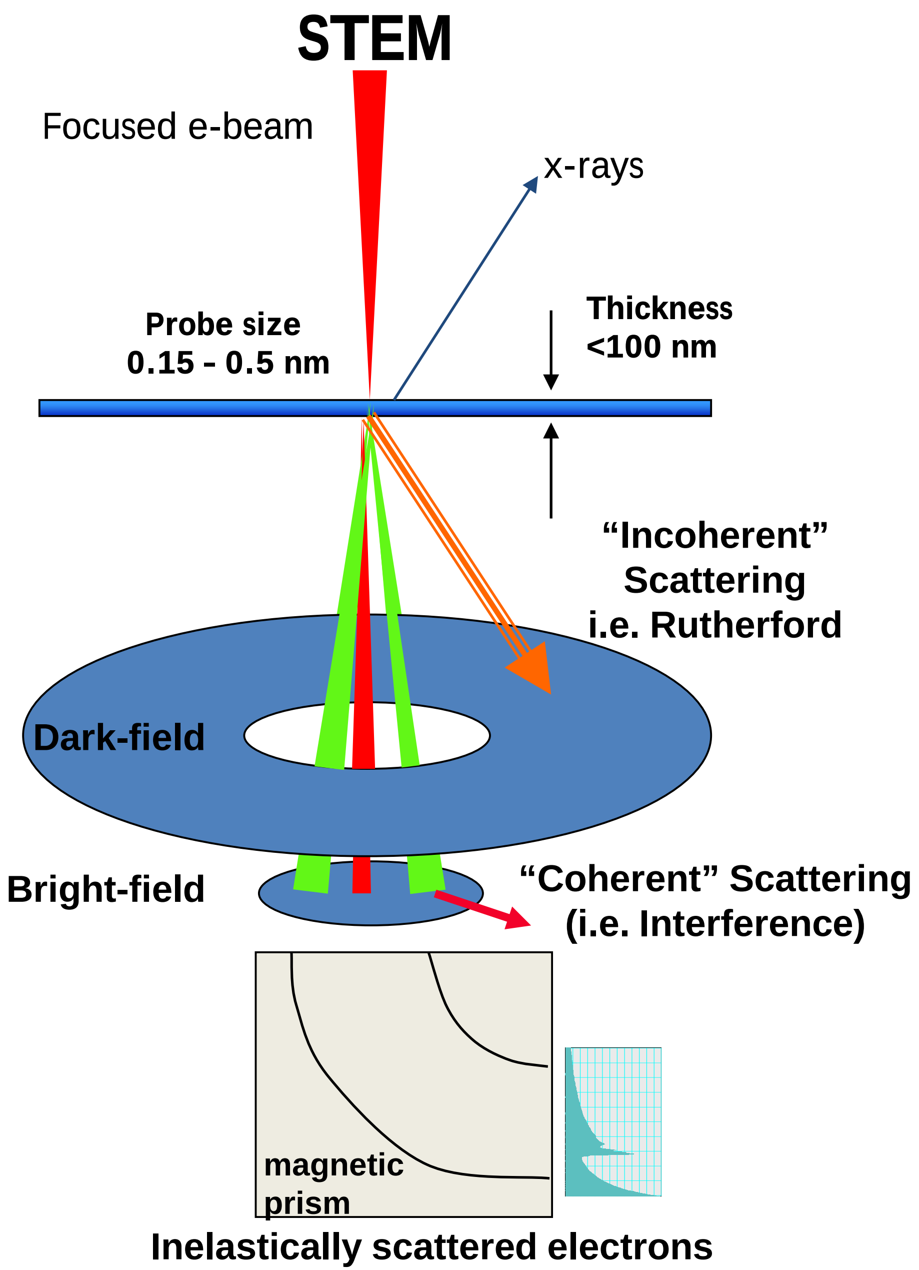
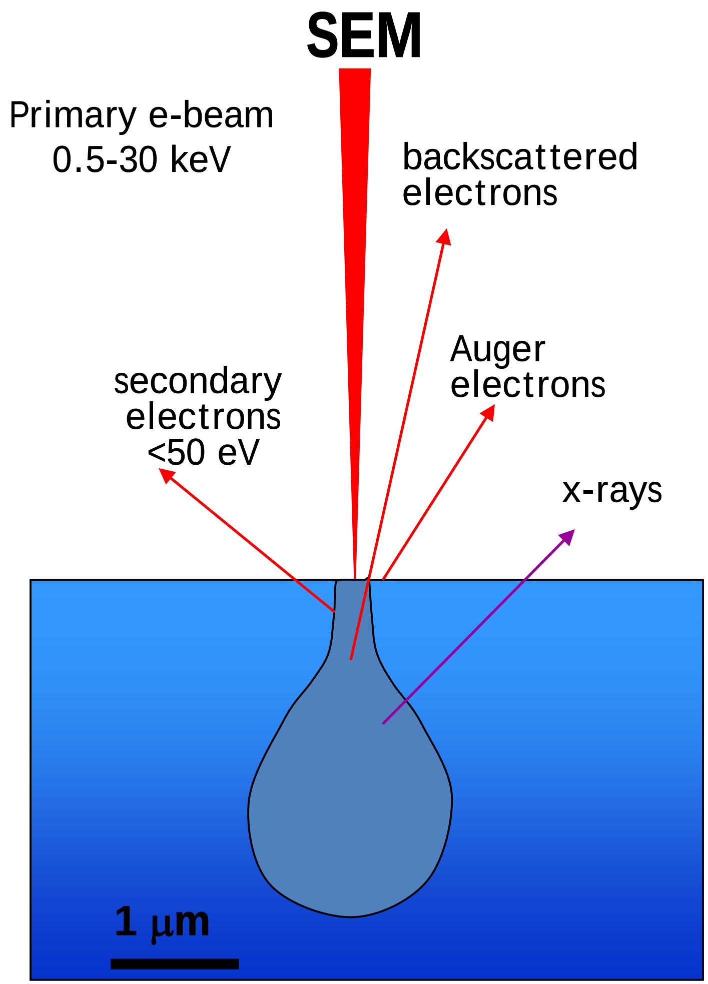
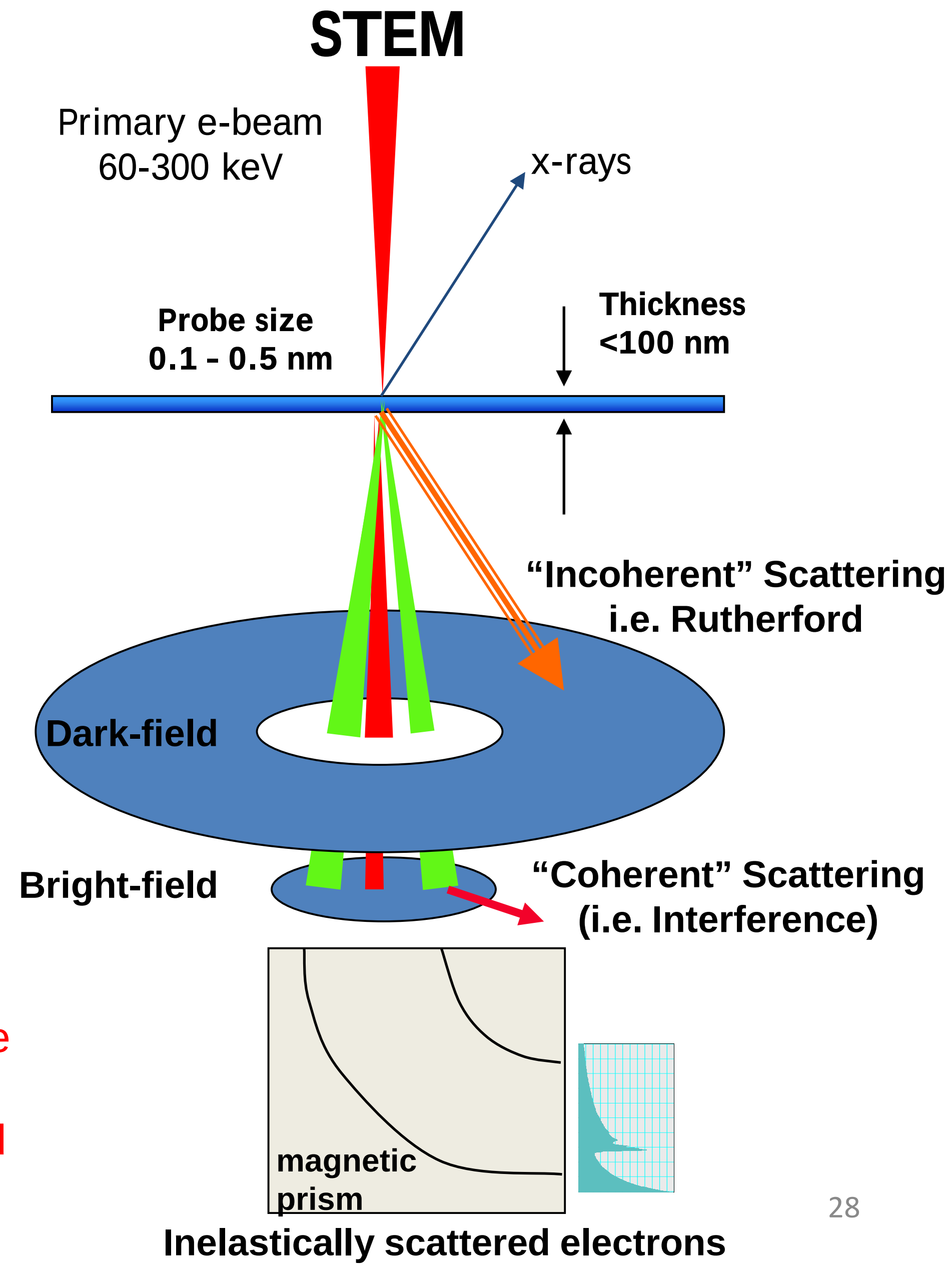


Technique

1. Raster a converged probe across and collect the integrated signal on an **Annular** detector (**Dark Field**) or a circular detector (**Bright Field**).
2. An incoherent image is chemically sensitive (Z-contrast) under certain collection angles
3. Annular Dark Field (ADF) STEM is directly interpretable and does not have contrast reversals or delocalization effects like HRTEM
4. STEM resolution is determined by the probe size, which is typically 0.15 to 0.5 nm for a modern S-FEG STEM.
5. Since STEM images are collected serially, the resolution is typically limited by vibrations and stray fields

SEM vs STEM





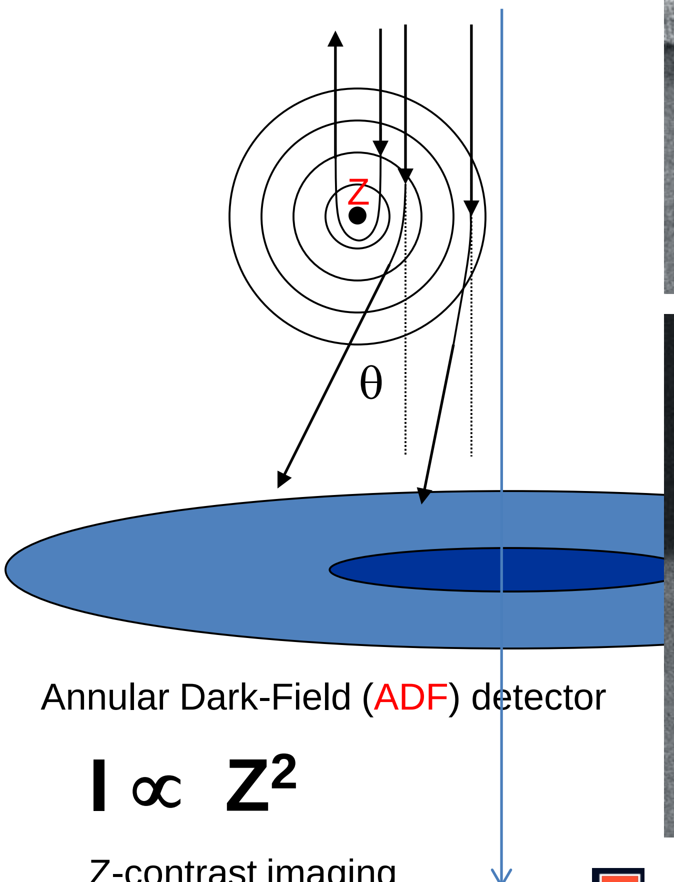
STEM technique is similar to SEM, except the specimen is much thinner and we collect the transmitted electrons rather than the reflected electrons.

TEM vs ADF-STEM

Ge quantum dots on Si substrate

Ir nanoparticles

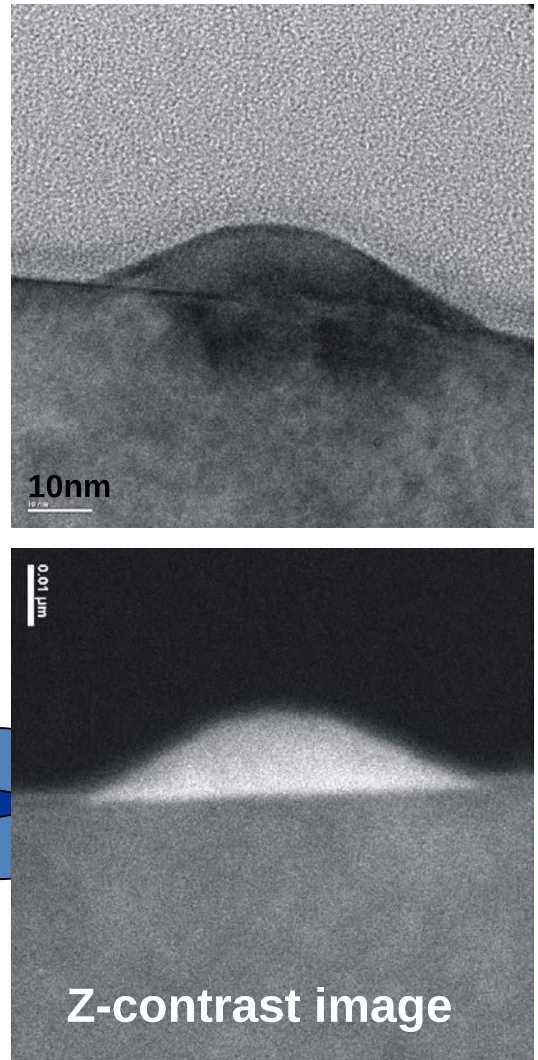
1. **STEM imaging gives better contrast**
2. **STEM images show Z-contrast**



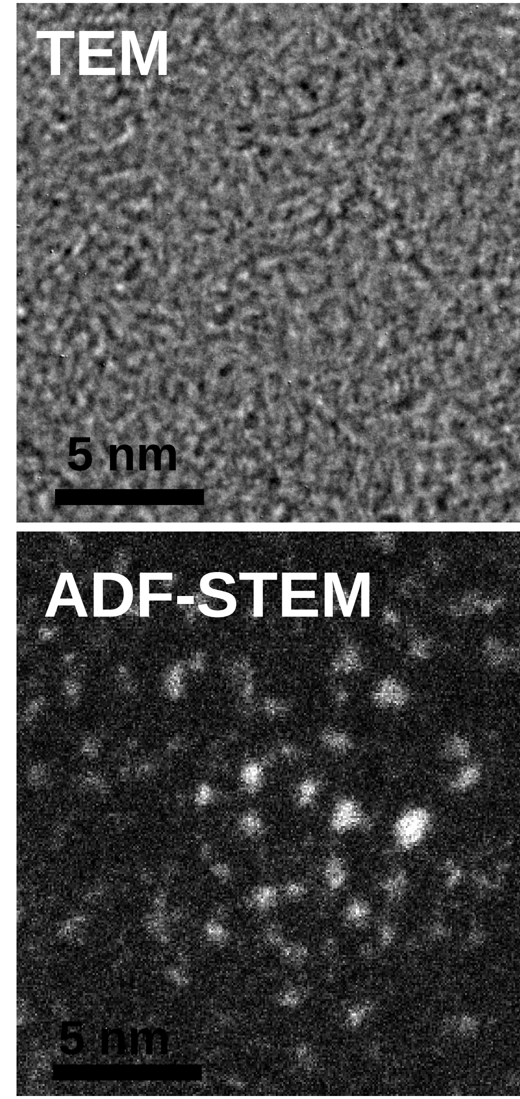
Annular Dark-Field (ADF) detector

$$\mathbf{I \propto Z^2}$$

Z-contrast imaging



J.G. Wen



L. Long

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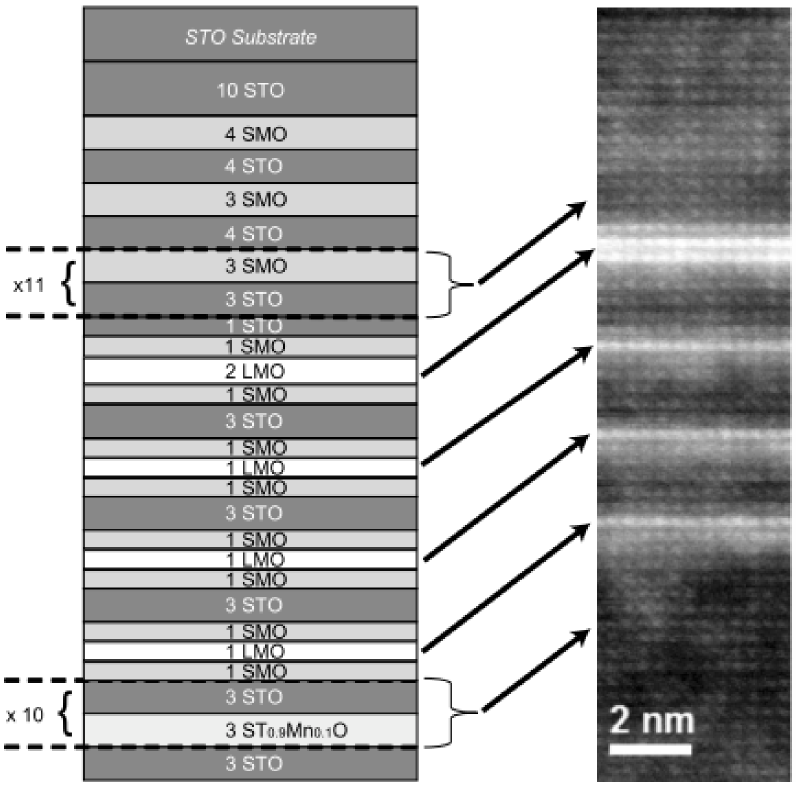
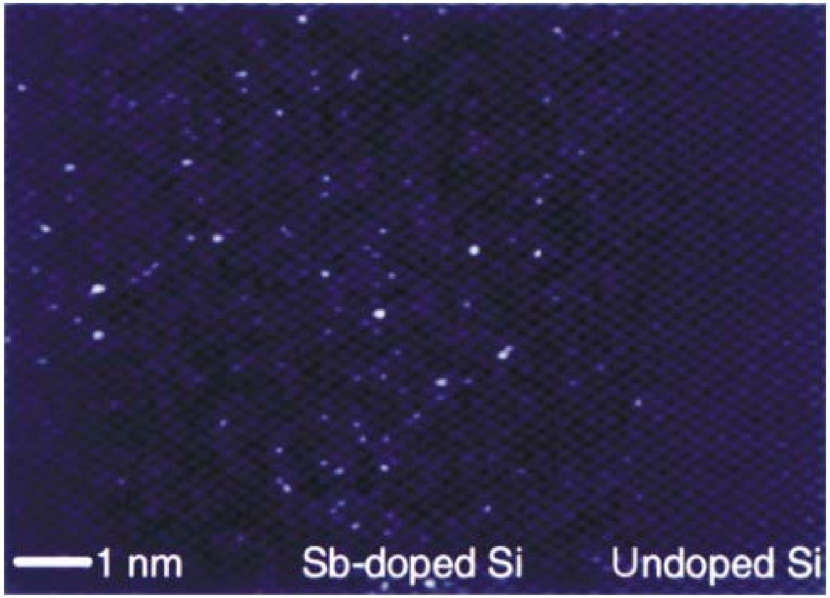
ADF STEM Applications

Superlattice of $LaMnO_3$-$SrMnO_3$-$SrTiO_3$



A. B. Shah et al., 2008

Dopant atoms in Si



P. M. Voyles et al., 2002

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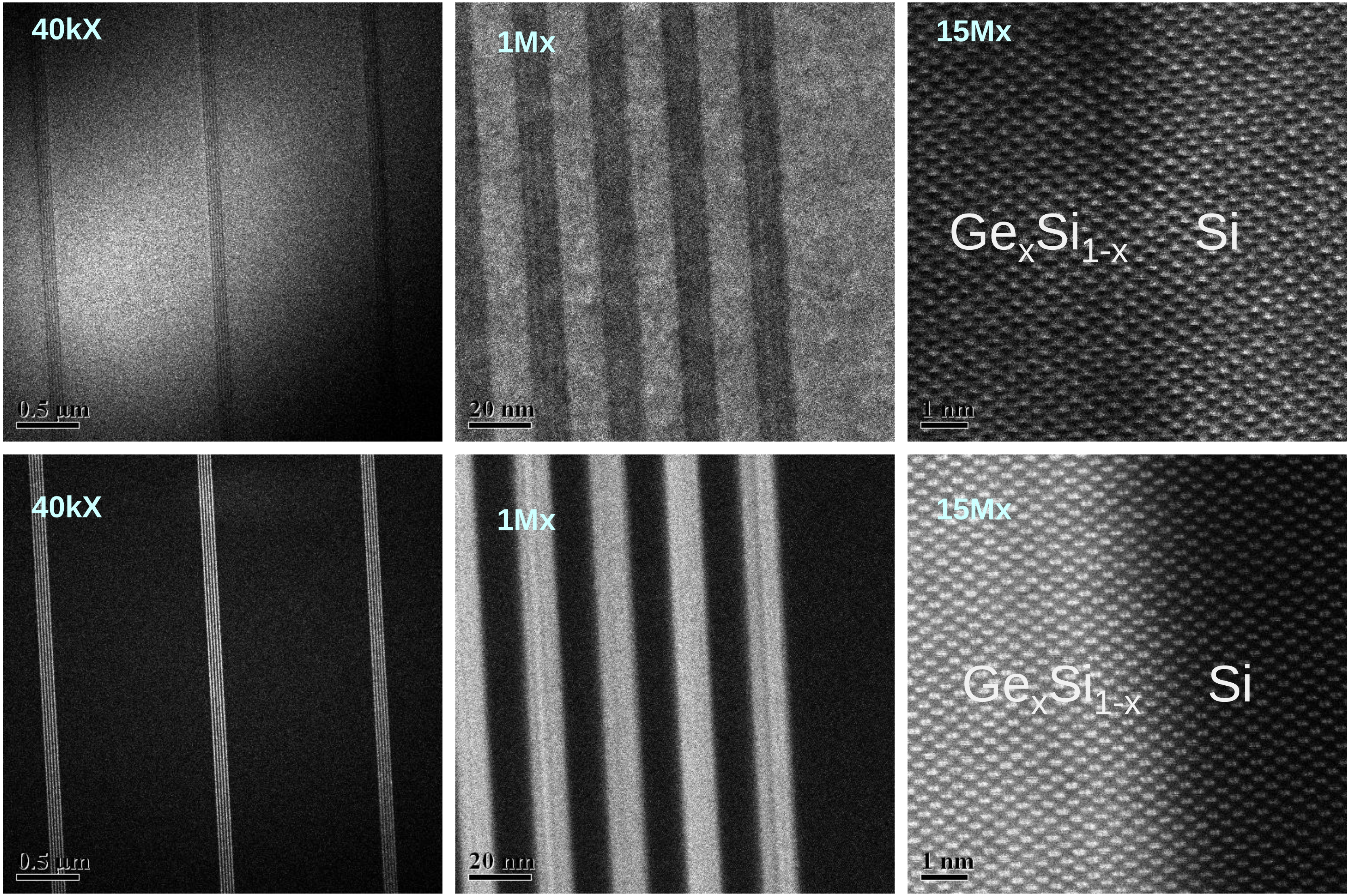
Bright Field STEM vs ADF STEM

Si – Ge Superlattice



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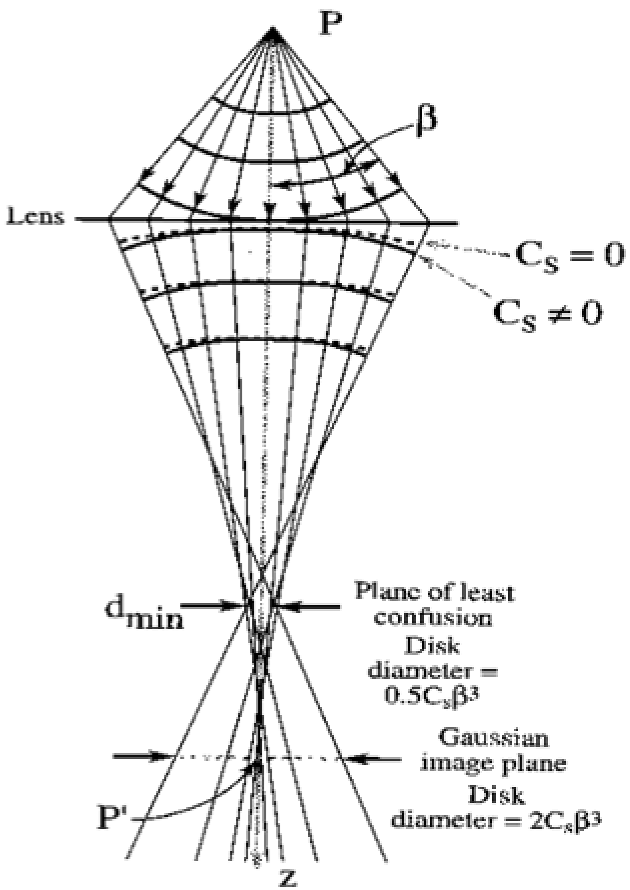
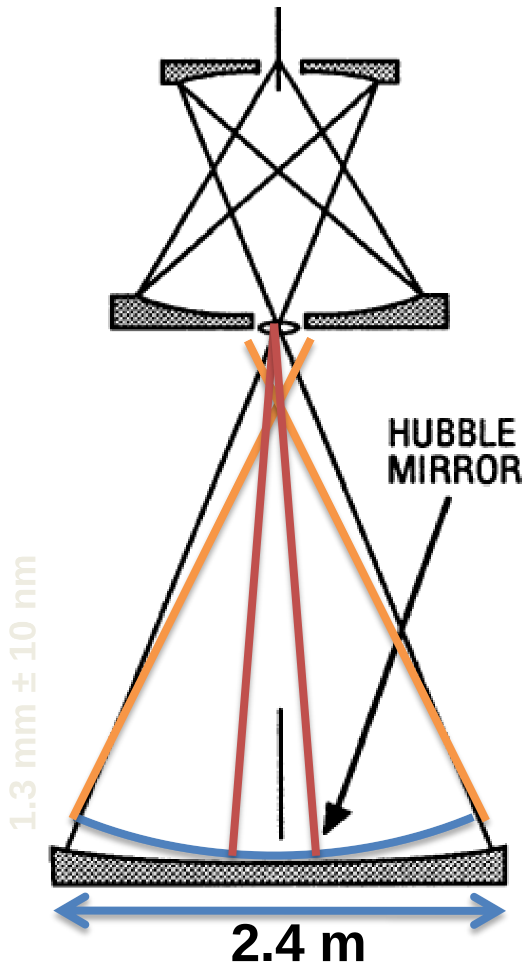
Spherical Aberration

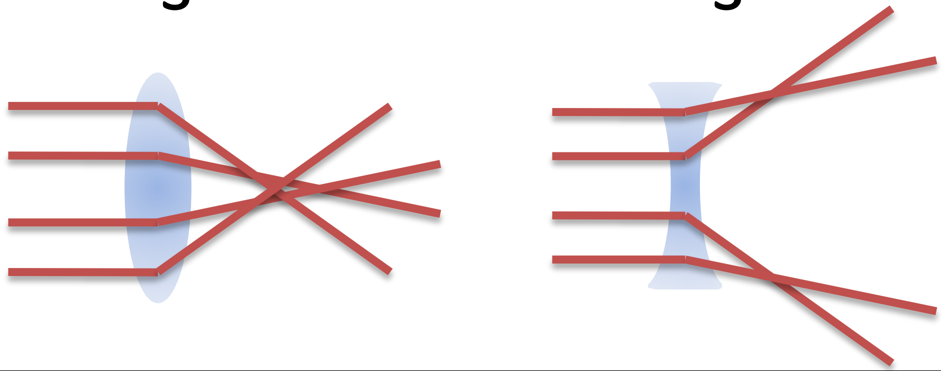


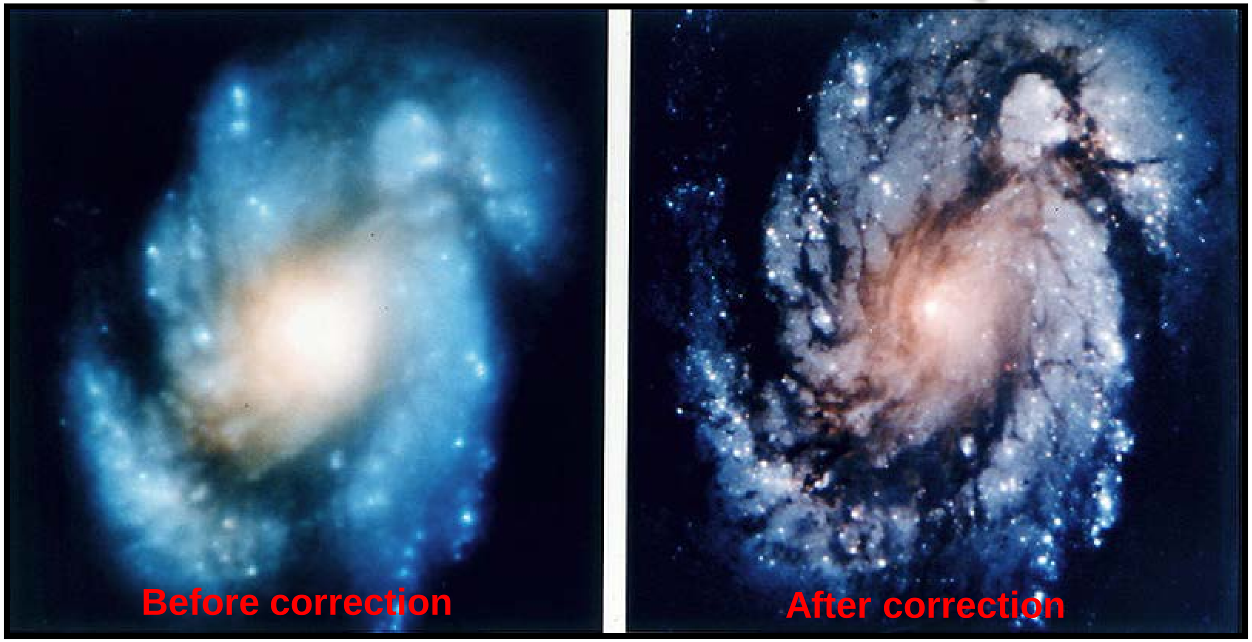


Since 1936, Scherzer proved that spherical and chromatic aberrations would ultimately limit the resolution of the electron microscope. The method to correct aberrations was well known, but experimental aberration correctors were not successful until ~1998 due to complexities in alignment and lack of computing power.

Spherical Aberration Correction

$$C_s \quad + \quad (-C_s) = 0$$





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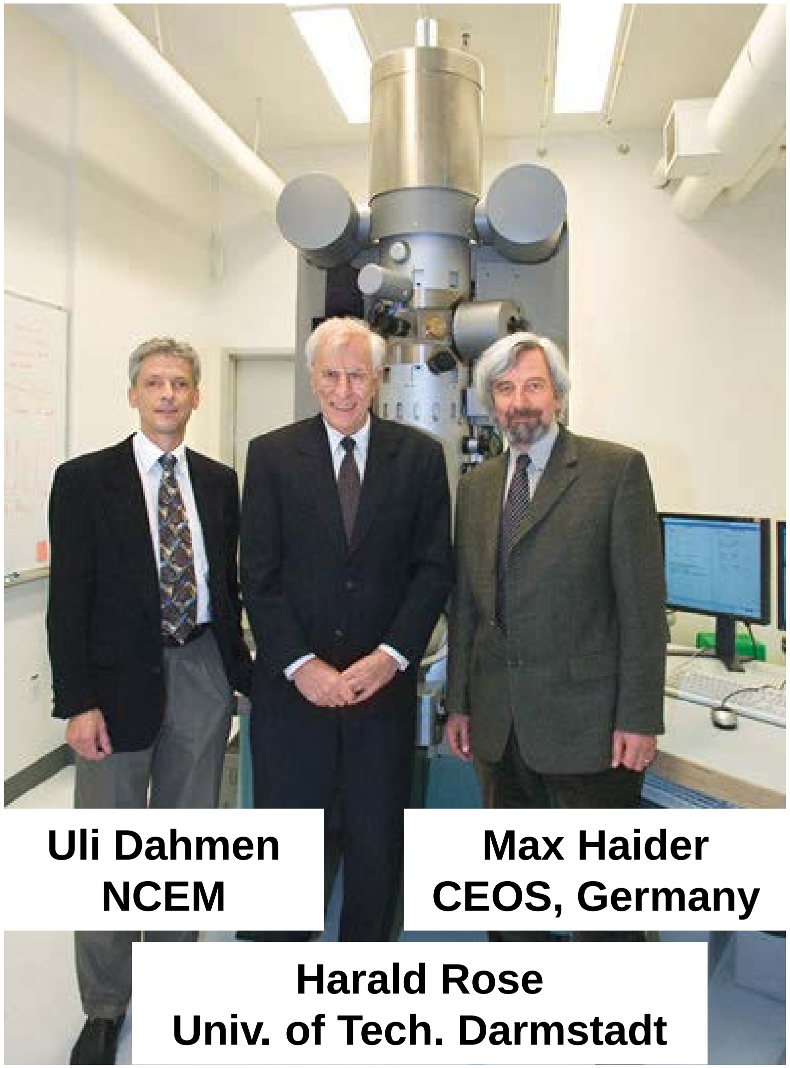
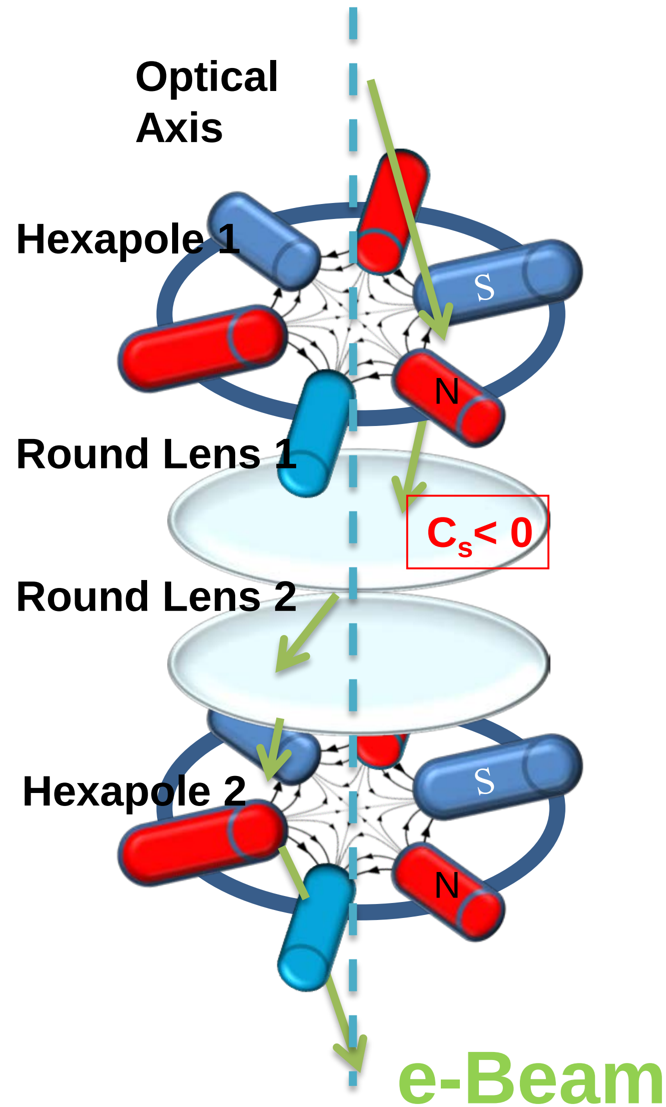
Spherical Aberration Corrector for TEM & STEM



Hexapole C_s Corrector



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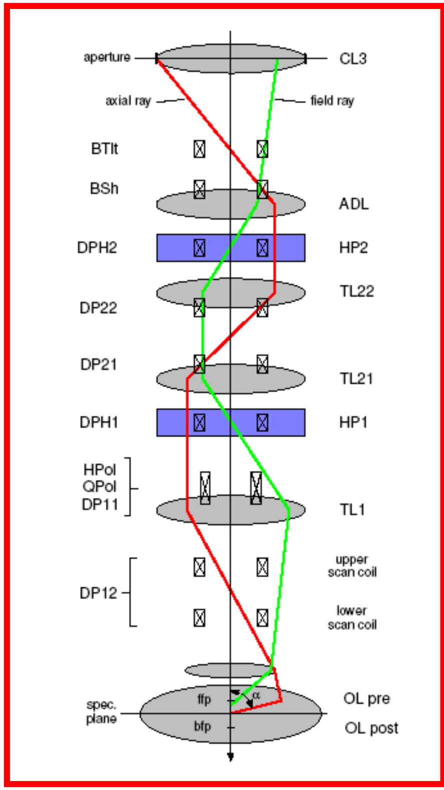
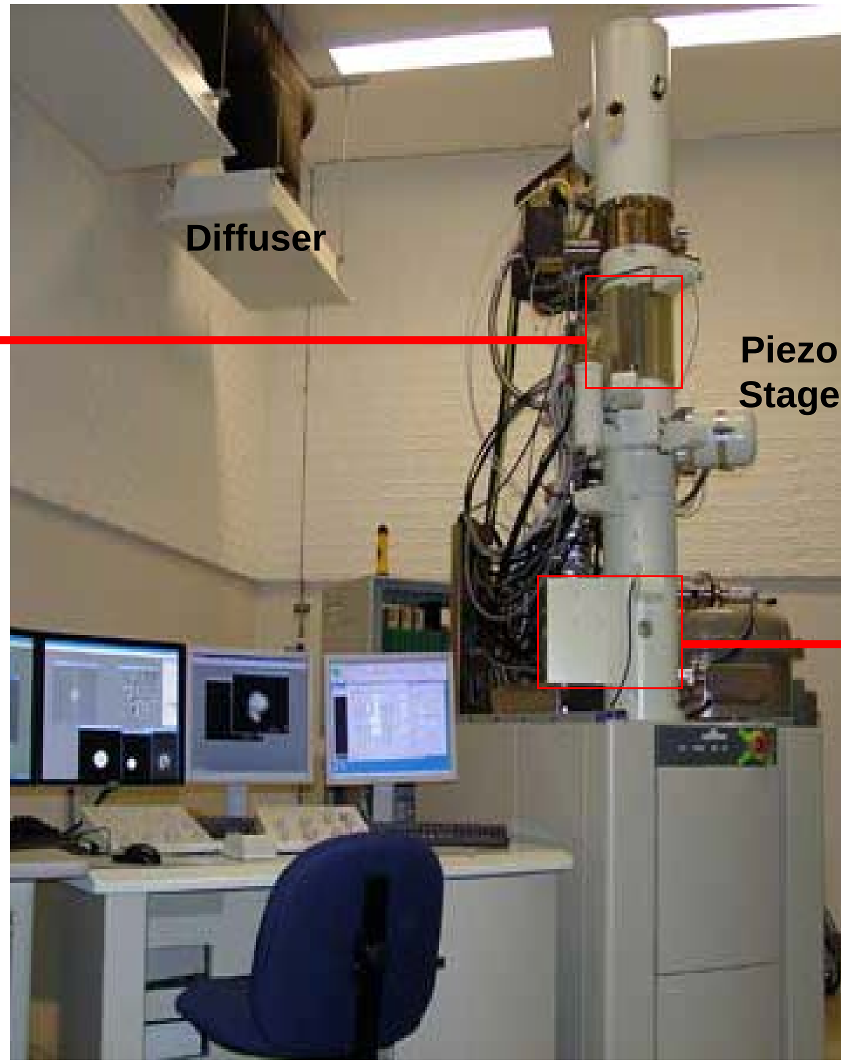
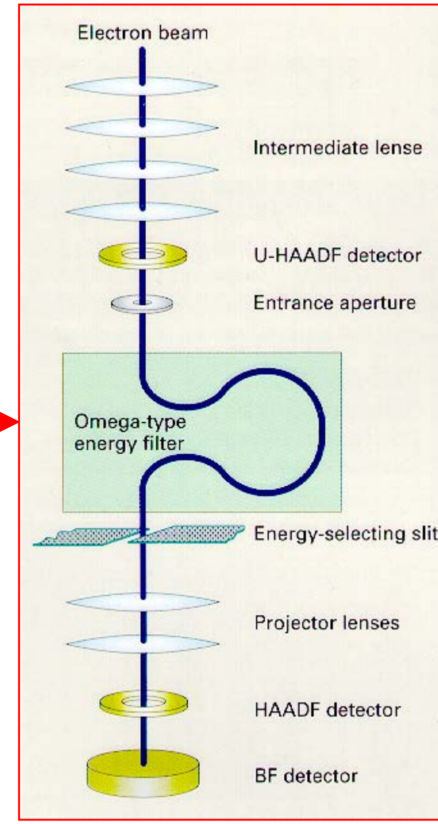
JEOL JEM2200FS with Probe Corrector @ UIUC





The STEM can obtain 1 Å spatial resolution



Only if the instabilities of the room are controlled

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Spherical Aberration Correction

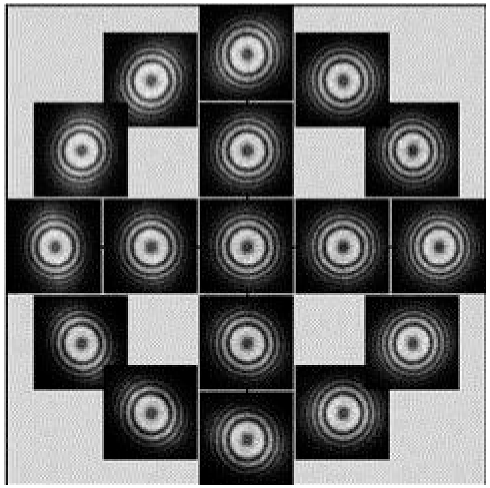
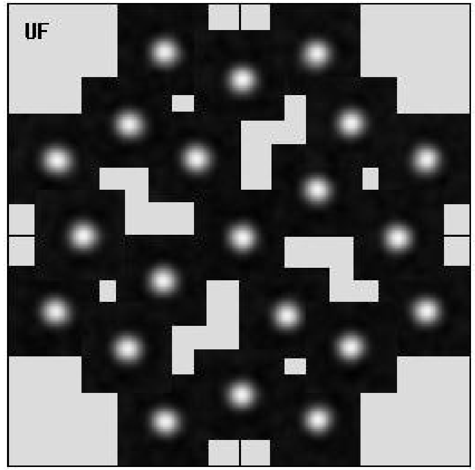
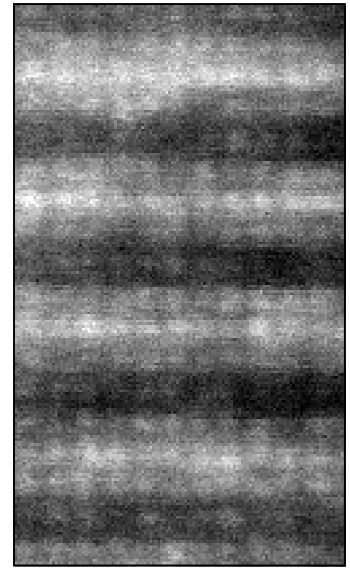


Image corrector



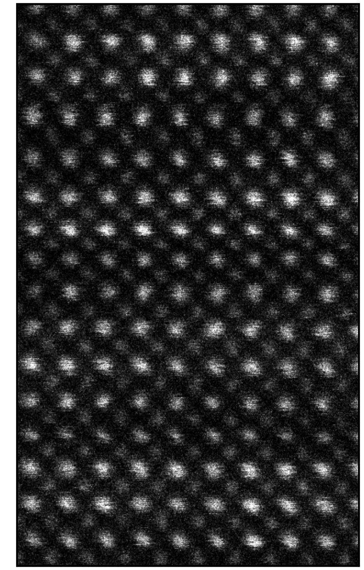
Probe corrector

```
#20 C1:   15.32nm A1: 10.3nm /   -157deg
#21 C1:   10.08nm A1: 8.85nm / -112.7deg
#22 C1:   20.04nm A1: 6.14nm / +135.8deg
dTime: 3840s Date: Tue Sep 18 16:48:00
1st order measured! (not used:)
Sall: 6.785nm Sused: 6.785nm (1.661%)
C1:   14.91nm              (95%: 8.08nm)
A1:   2.851nm /   +90.3deg (95%: 10.9nm)
A2:   36.73nm /   -40.5deg (95%:  124nm)
B2:   40.49nm /   +33.1deg (95%:  107nm)
C3:   1.721um              (95%: 8.12um)
A3:   1.137um / +103.8deg (95%: 1.81um)
S3:   1.574um / +115.4deg (95%:  764nm)
A4:   6.573um / -174.2deg (95%: 58.5um)
D4:   31.83um /   +69.4deg (95%:   39um)
B4:   29.75um /   +40.6deg (95%: 71.4um)
C5:   2.258mm              (95%: 9.74mm)
A5:   1.141mm / +101.9deg (95%:  2.1mm)
Btn 'Accept Aberr' pressed.
File /home/cscorr/sm_data/UIUC2200FS/0
```



JEOL 2010F
C_s = 1 mm

Probe size
0.3 nm



JEOL 2200FS
with probe corrector

Probe size
0.1 nm

$$r_{sch} = 0.66\, C_s^{\frac{1}{4}}\, \lambda^{\frac{3}{4}}$$

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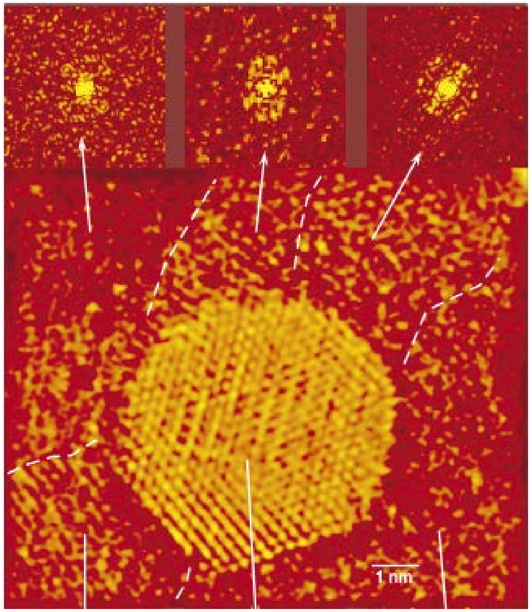
Spherical Aberration Corrector for STEM

Ondrej Krivanek
Nion Company

Quadrupole-Octupole C_3-C_5 corrector

First sub-Å image resolved in a STEM



P. E. Batson et al., *Nature*, 2002.

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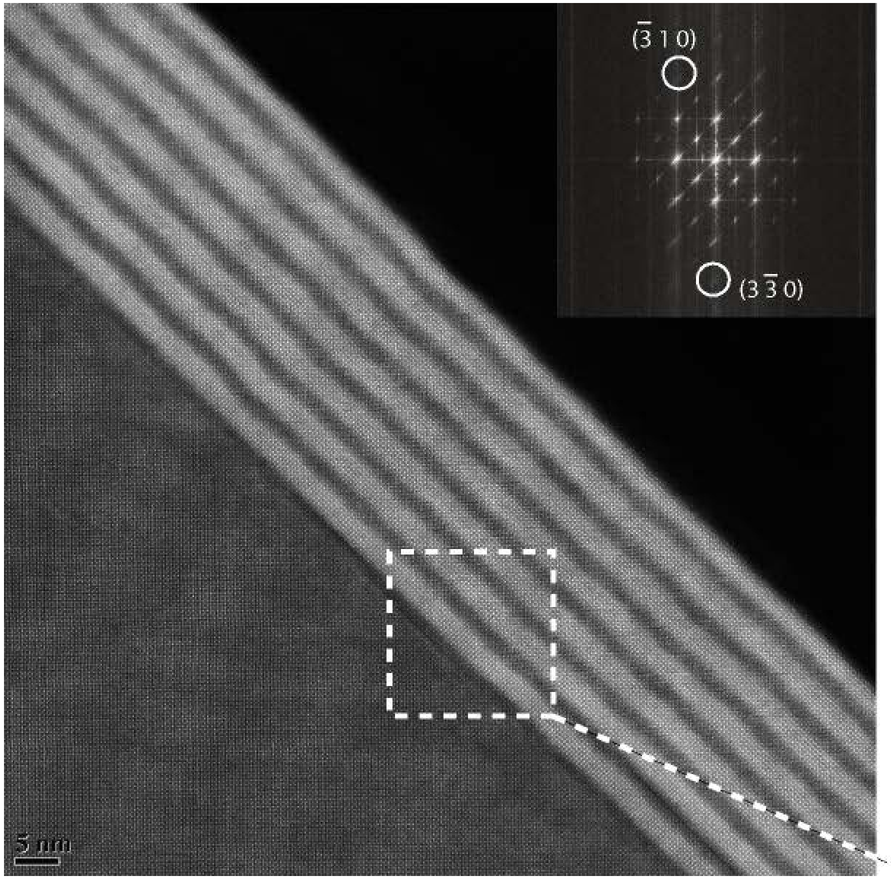
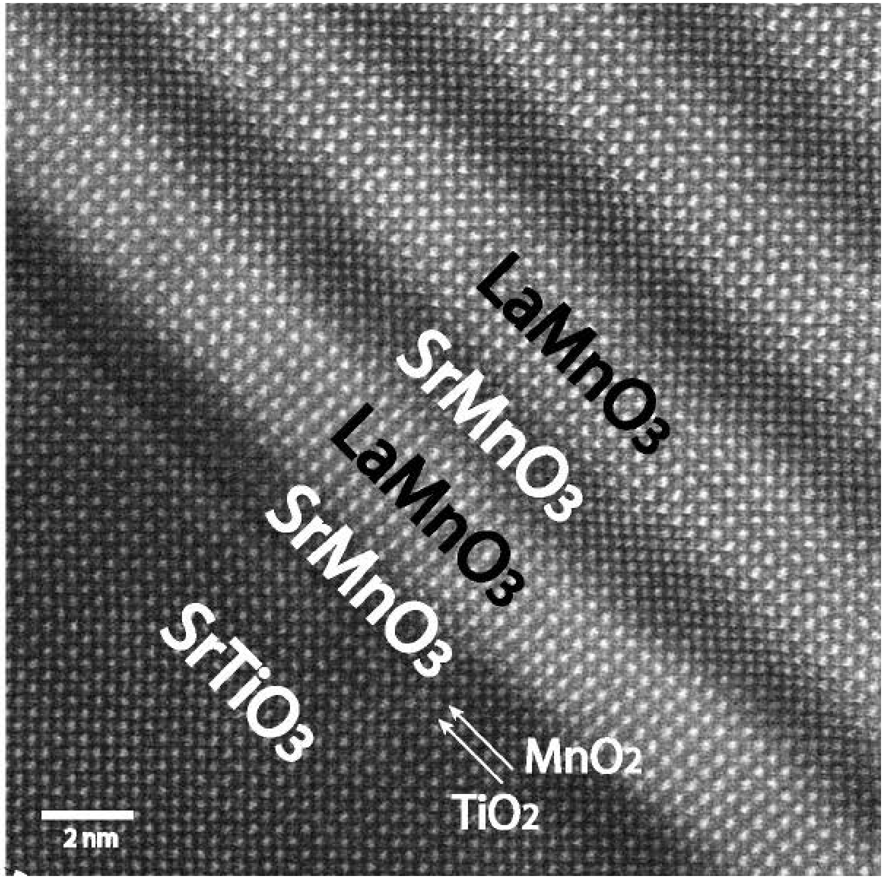
Improved Resolution and Contrast with C_s Corrector





Aberration correction combined with a high stability environment and high quality specimens allow for atomic resolution imaging over a large area. 4k x 4k image shown.

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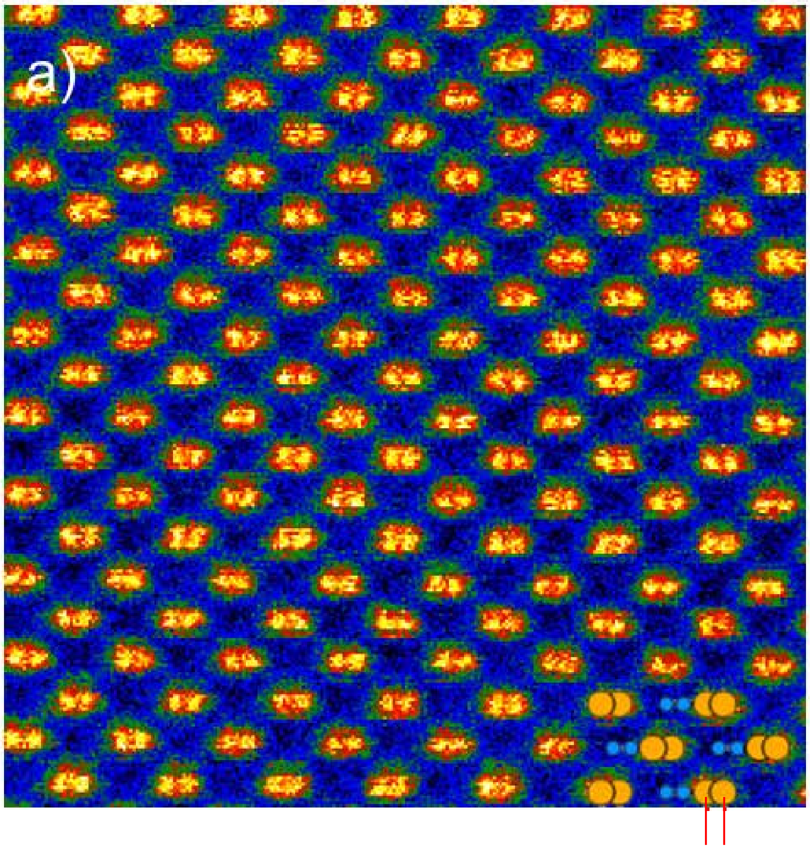
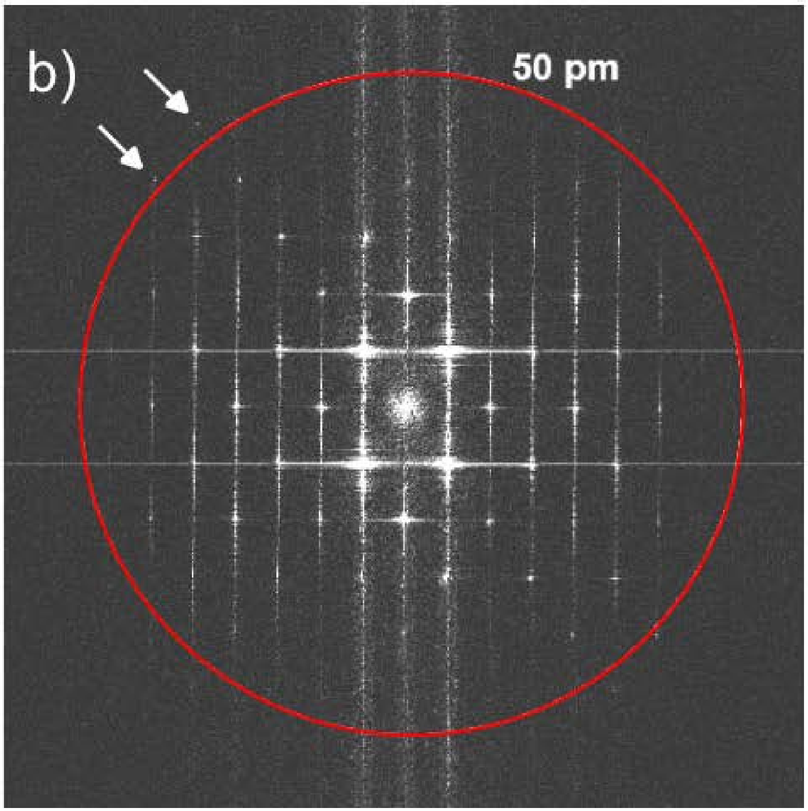
Sub-Å test using GaN film along [211] zone axis

TEAM Microscope at LBNL





Annular dark field STEM image of hexagonal GaN [211]

Fourier transform of the image; image Fourier components extend to below 50 pm.

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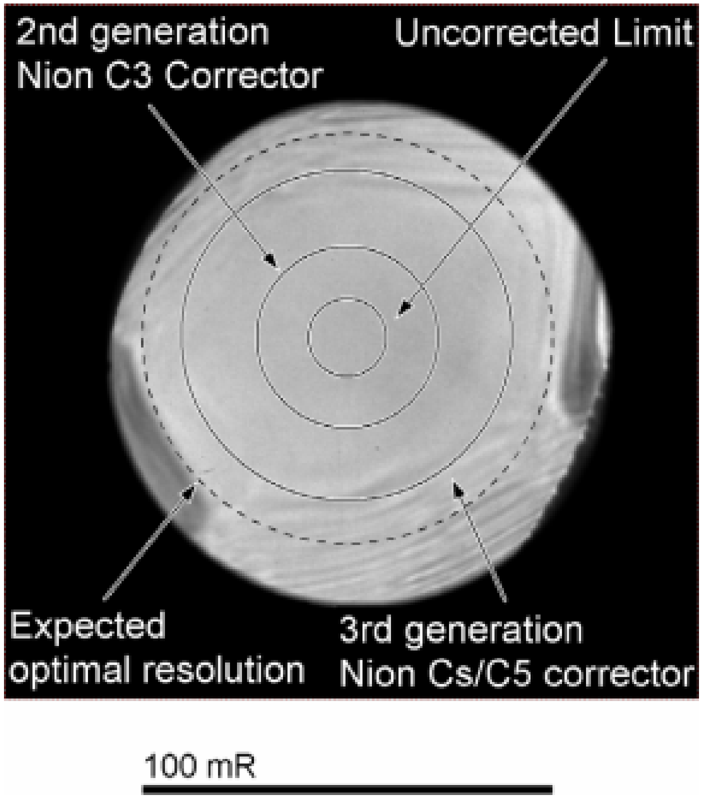
New Applications Possible Only with Aberration Correction

Ronchigram



1. **Better Contrast for STEM Imaging with smaller probe**
2. **Reduced delocalization for HRTEM imaging**
3. **Sub-Å resolution imaging at low voltage (60 – 100 kV) for TEM and STEM**
4. **10-20 X more probe current in the STEM for EELS and EDS spectroscopy**

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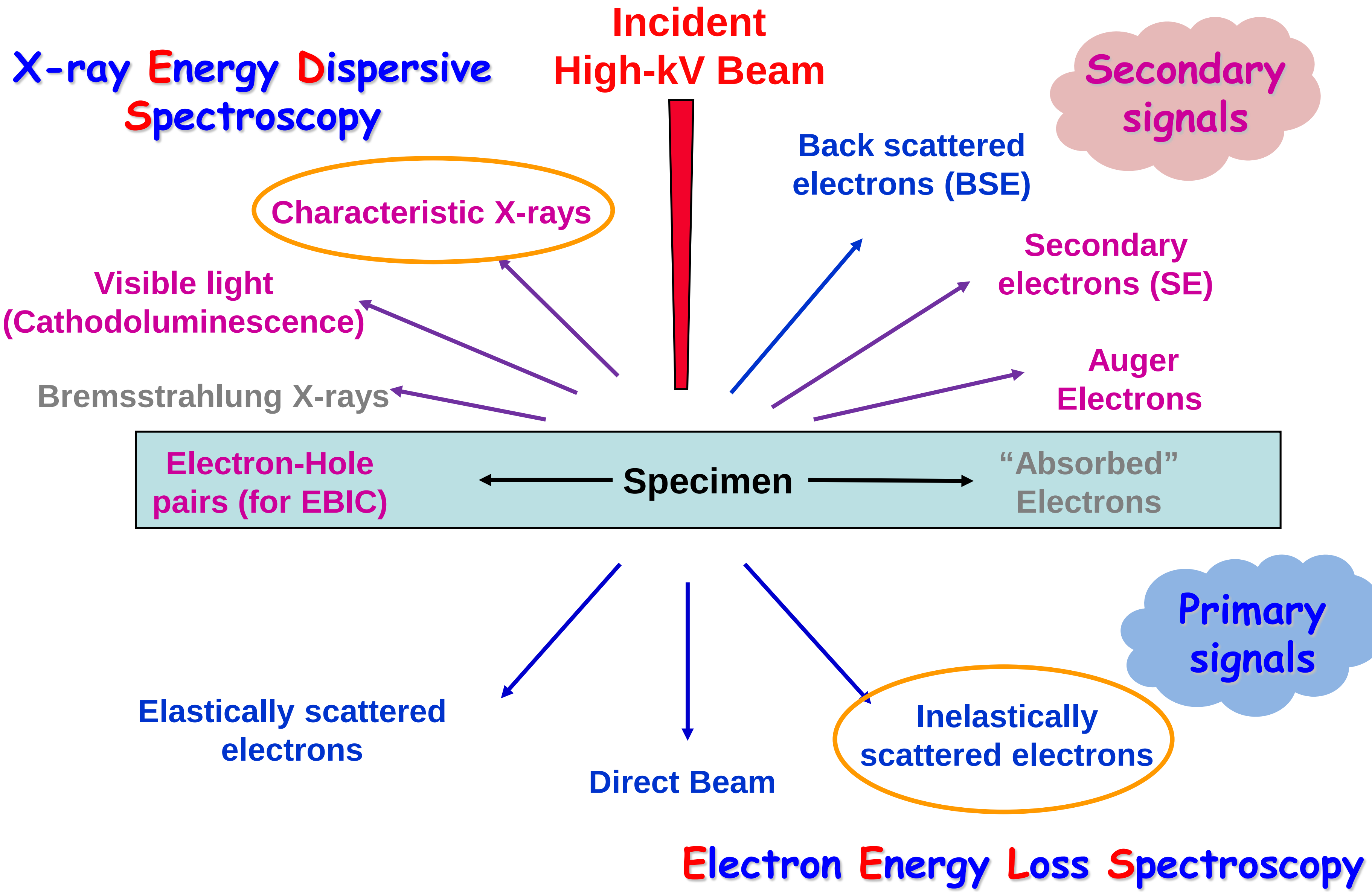
Analytical (Scanning) Transmission Electron Microscopy



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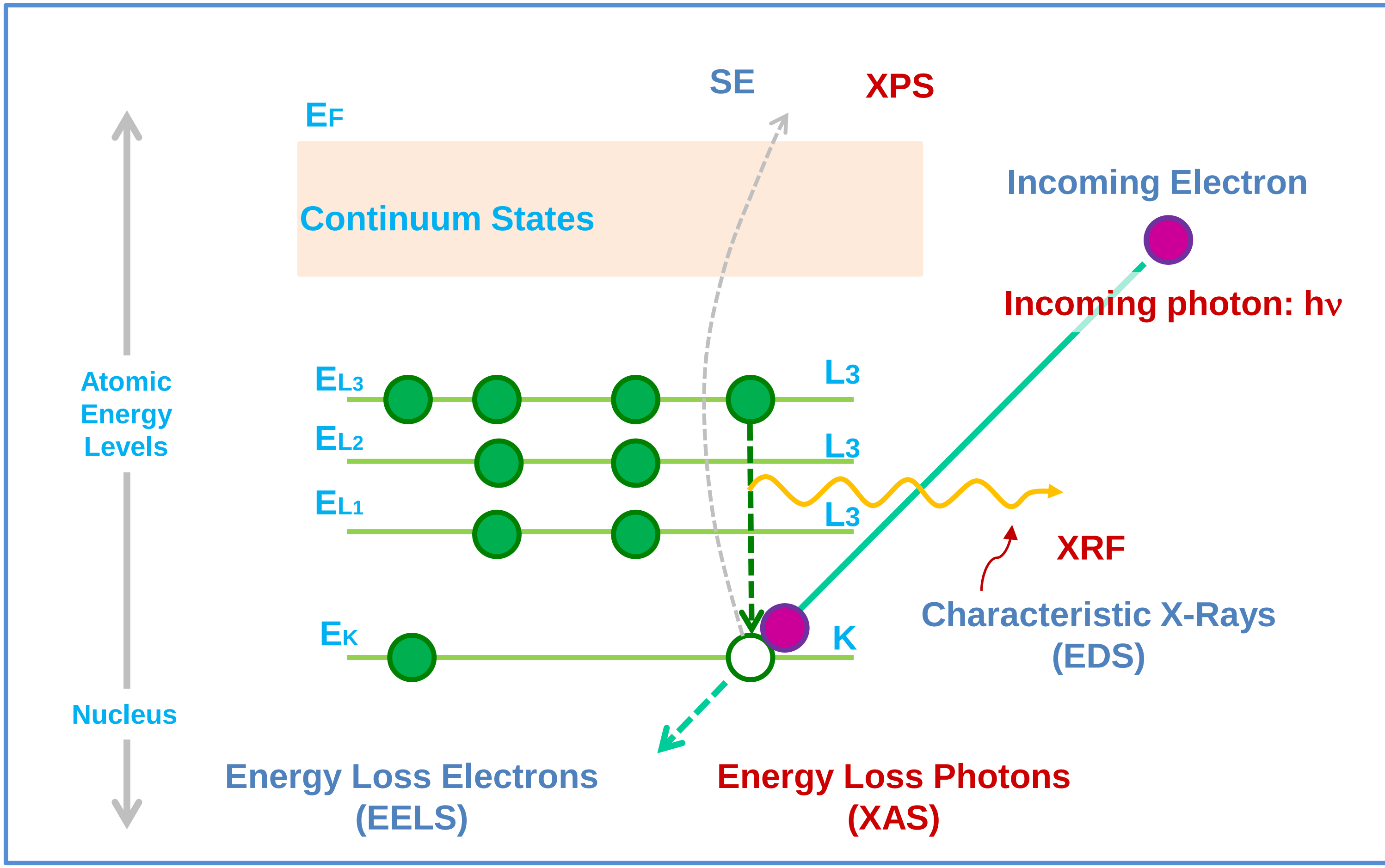
Characteristic Signals: EELS, EDS, XPS Signals



The emission of Auger electrons is an alternative to X-ray emission as an ionized atom returns to its ground state

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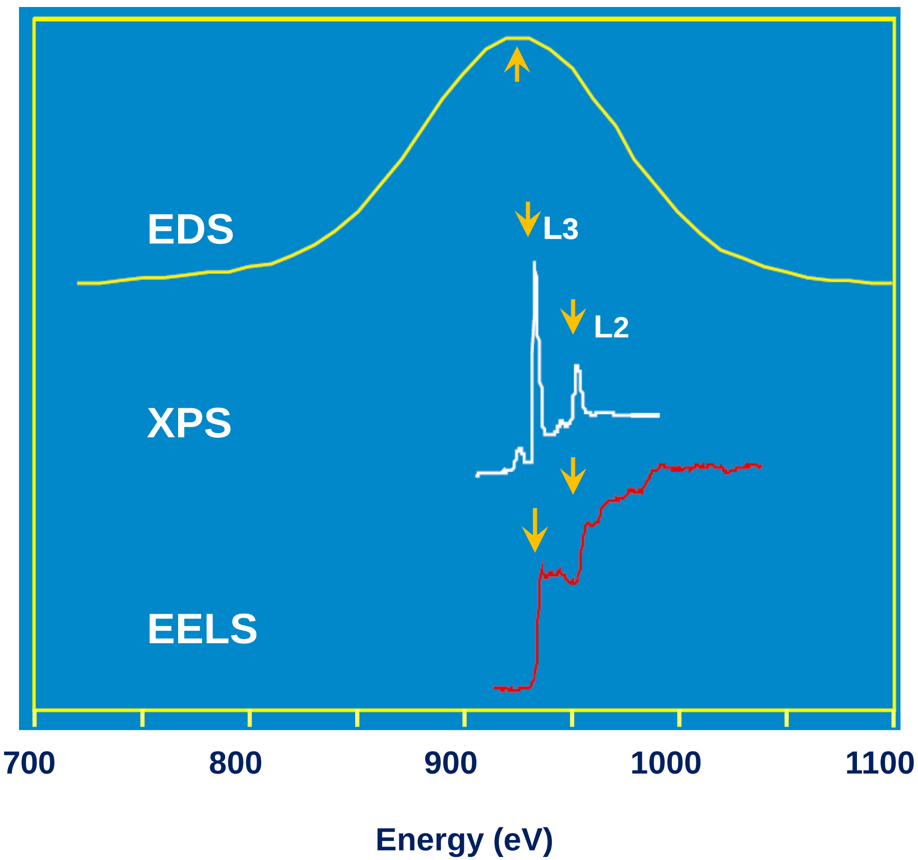
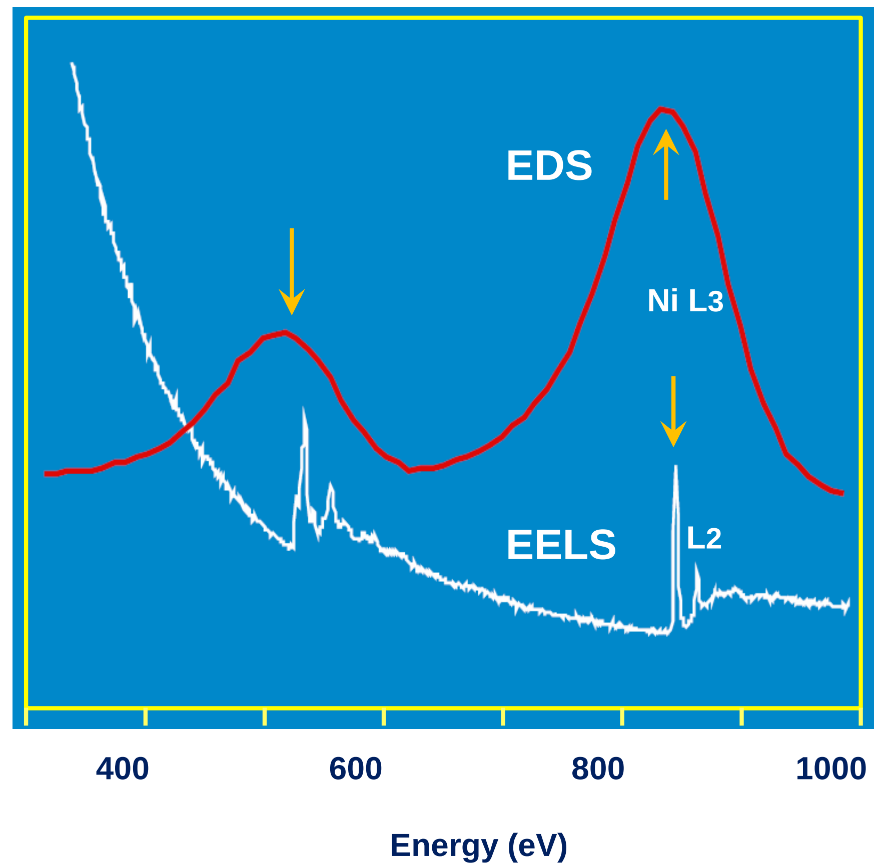
Copper L shell



NiO: O K-shell and Ni L shell



Energy resolution, Spatial resolution, Elements resolving

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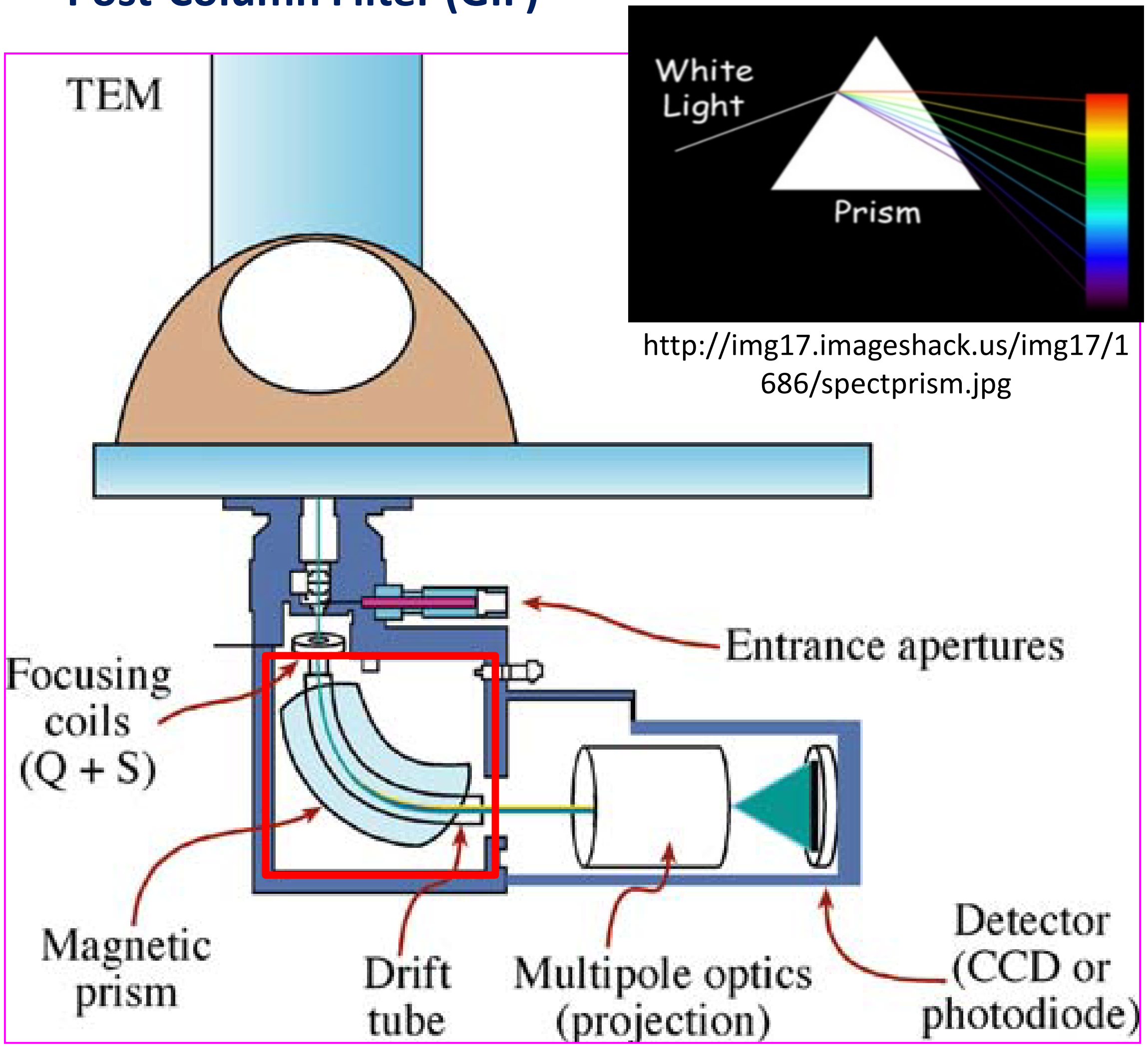
EELS: Electron Optical System

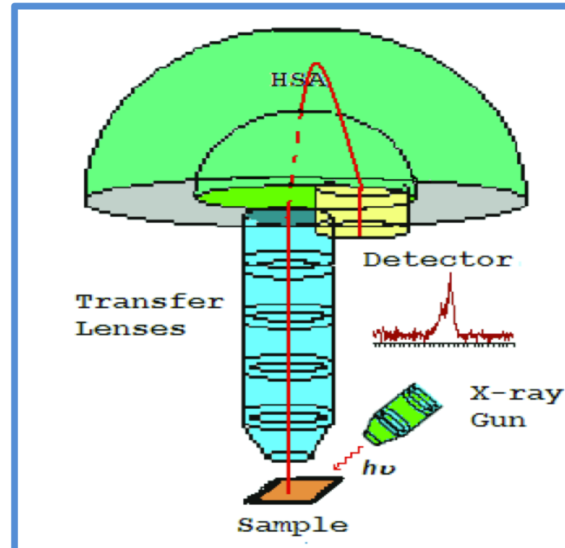
Post-Column Filter (GIF)

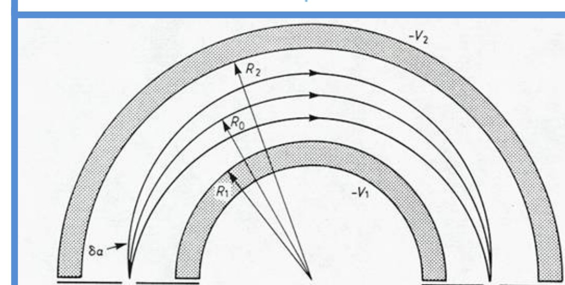


http://img17.imageshack.us/img17/1686/spectprism.jpg

Williams and Carter 2009

Compare: Hemispherical Electron Energy Analyzer (Surface Analysis)





$$V_0 = (V_1R_1 + V_2R_2)/2R_0$$

Casaxps.com

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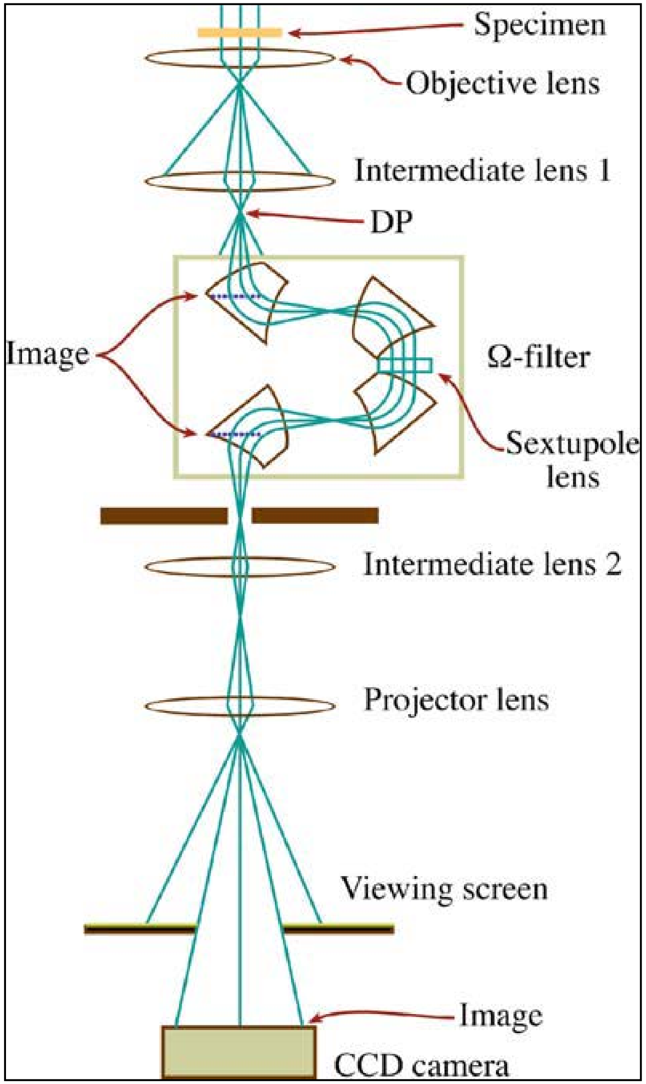
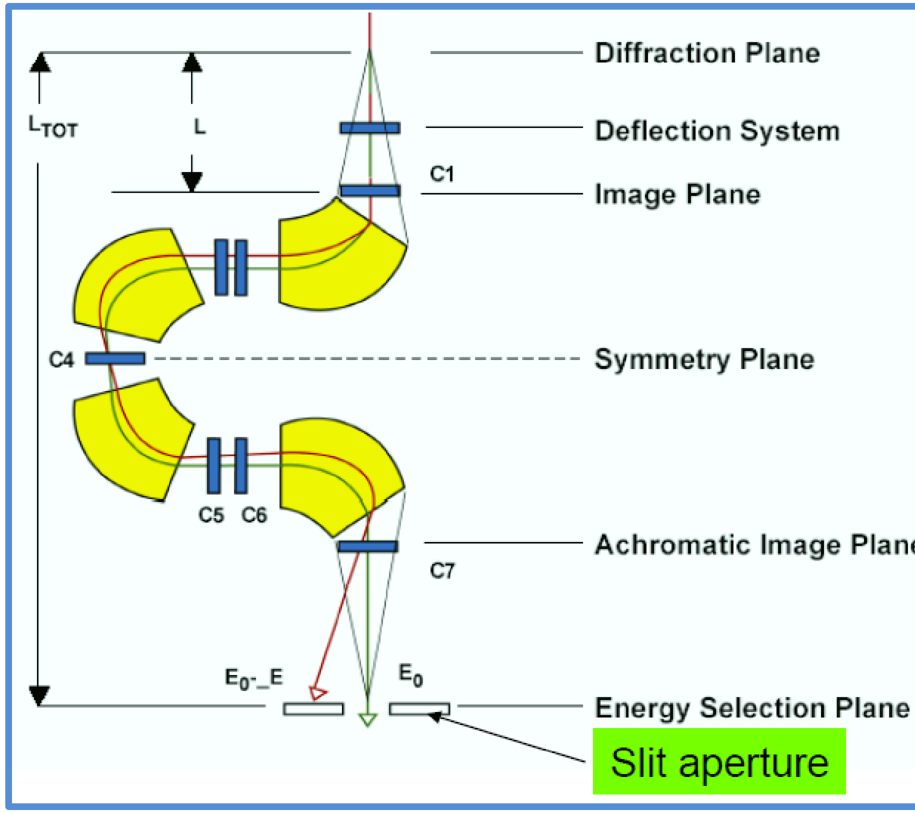
EELS: Electron Optical System



In-Column (Omega) Filter



- 4 Dispersive elements
- Integrated into column

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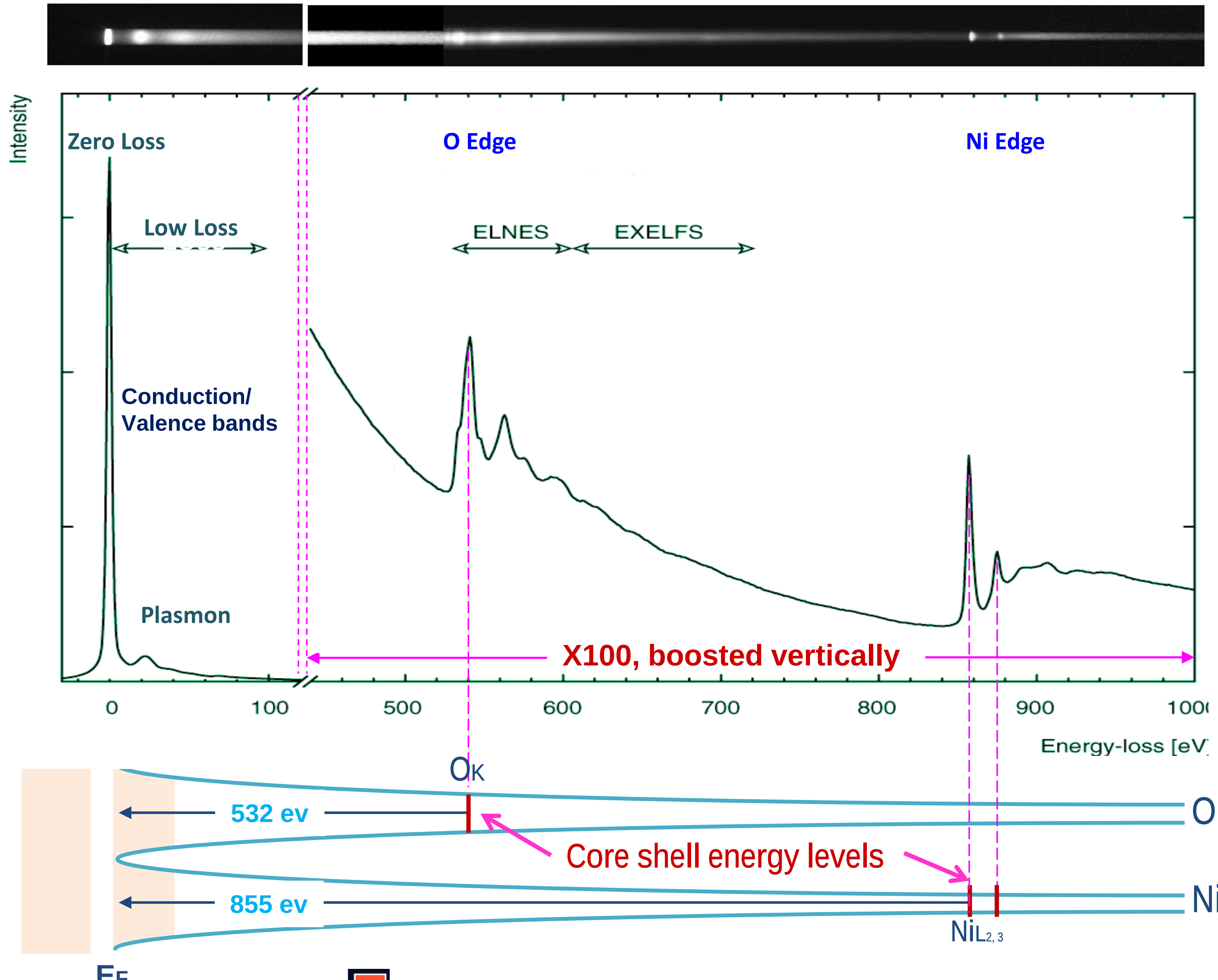
Experimental EELS: NiO



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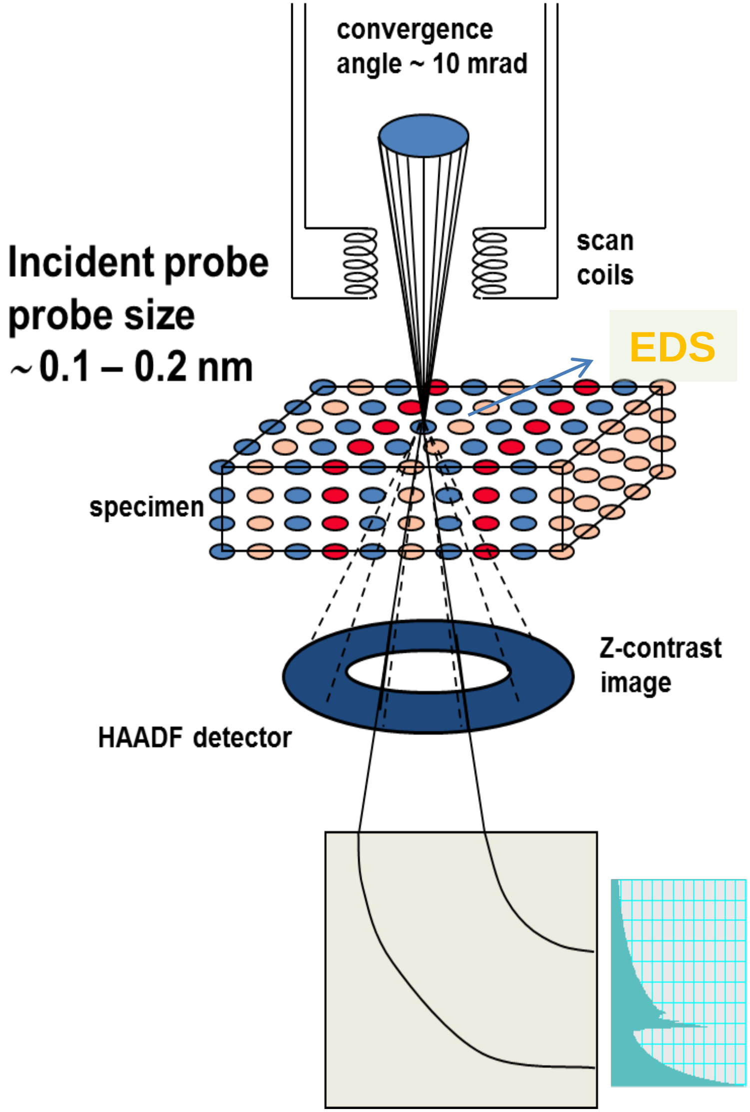
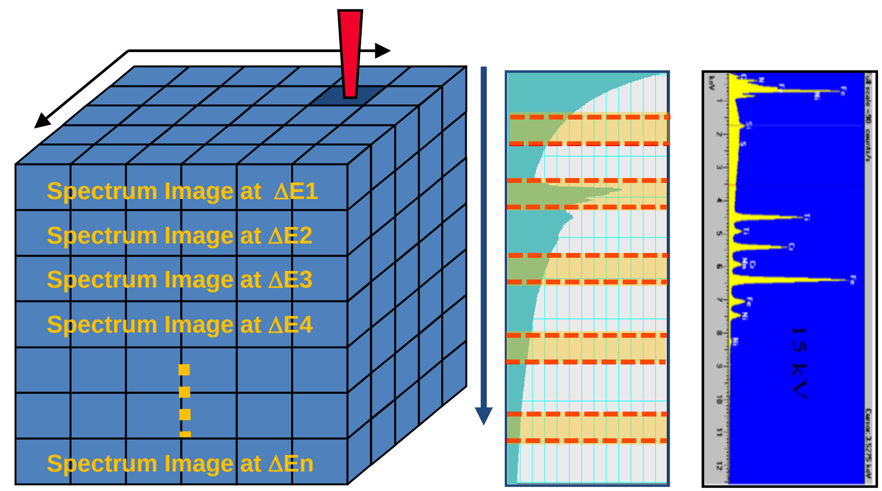
STEM-EELS/EDS Spectrum Imaging: Spatially Resolved Analysis



Spectral Imaging:

- **3D/4D data cube**
- **x-y image**
- **z spectrum (EDS/EELS)**



Sub Angstrom in Cs-corrected STEM !

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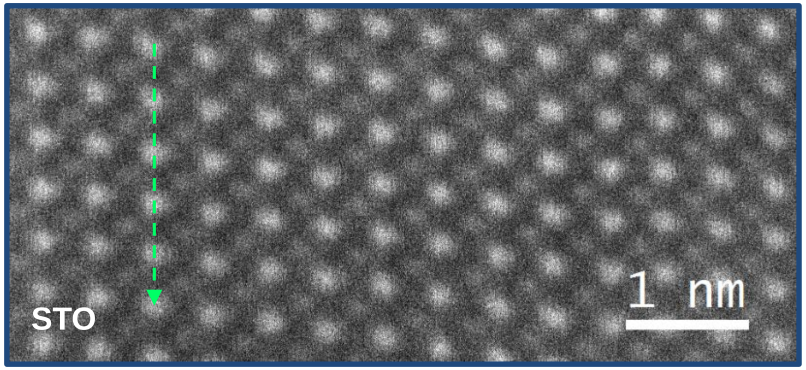
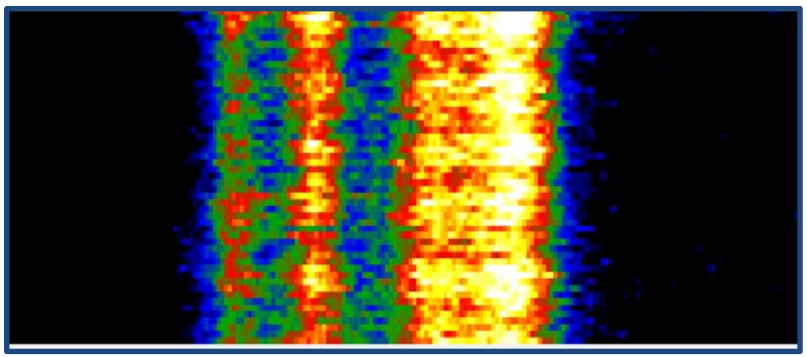
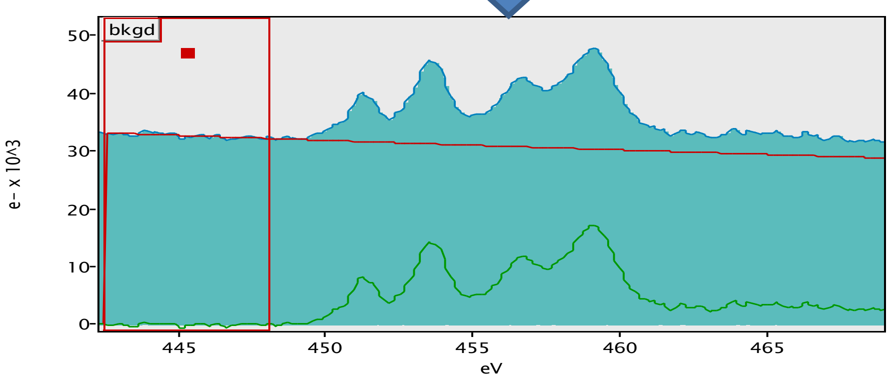
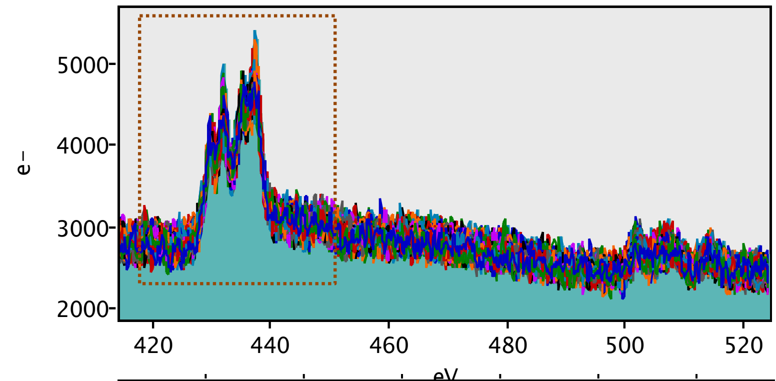
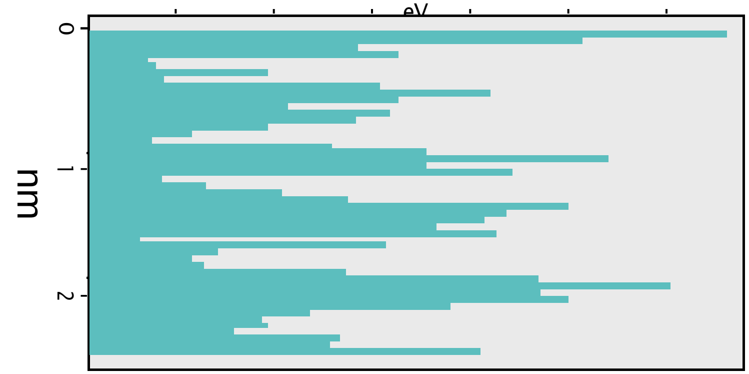
STEM-EELS Spectrum Imaging: Line Scan

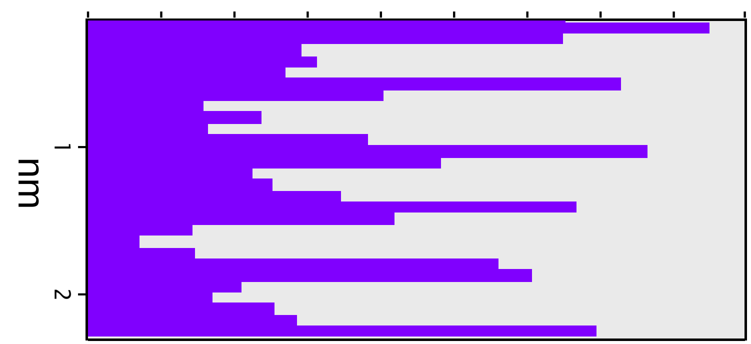












C. Chen, J. Mabon, 2200FS

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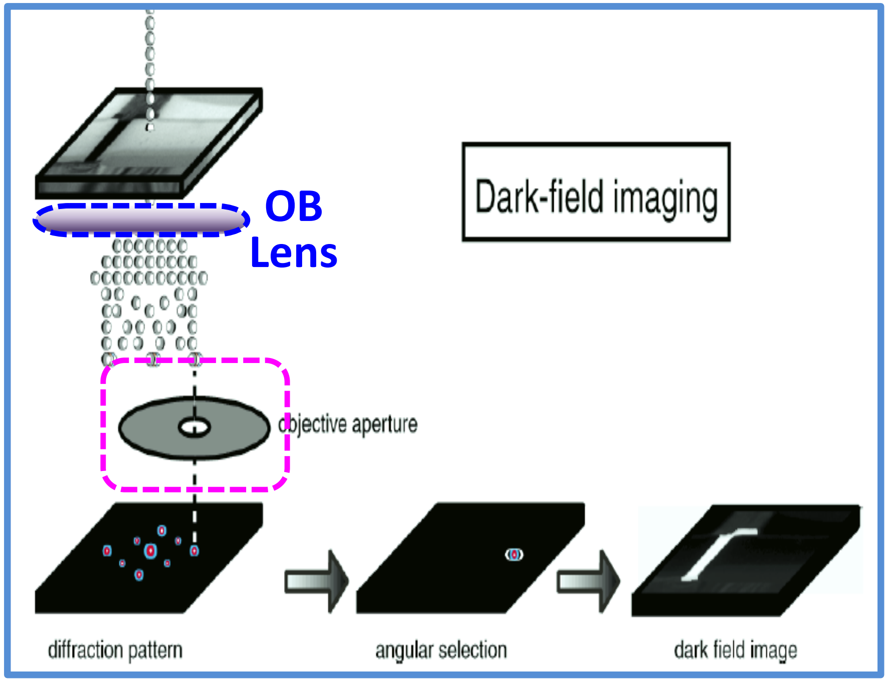
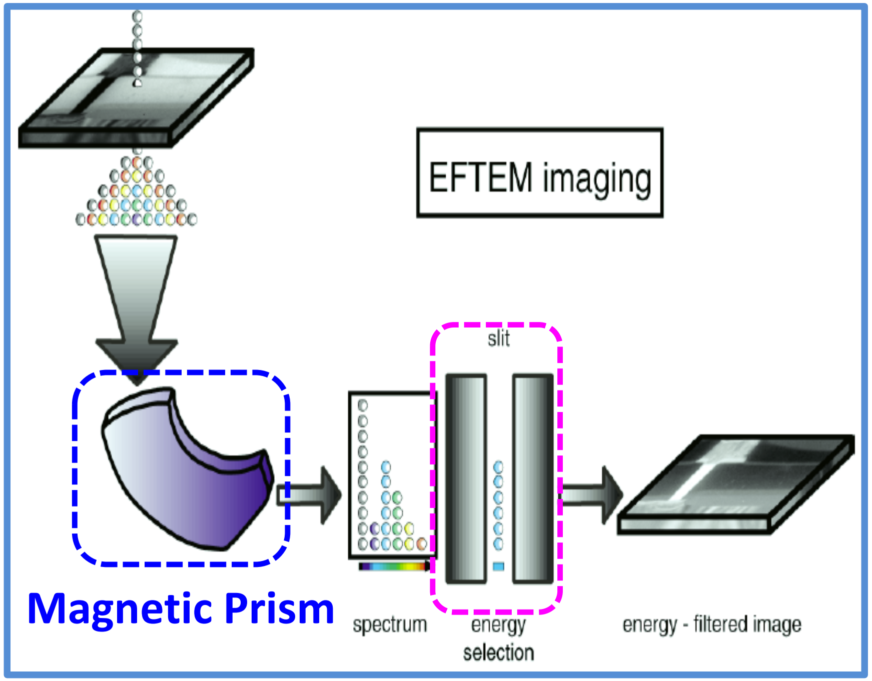
EF-TEM (Energy-Filtered TEM): Parallel Beam

- Use the slit to select electrons of a specific energy
- Allow only those electrons to fall on the screen or CCD
- Analogy to dark-field imaging

Dark–Field TEM



EF-TEM Imaging



Gatan Review of EFTEM Fundamentals

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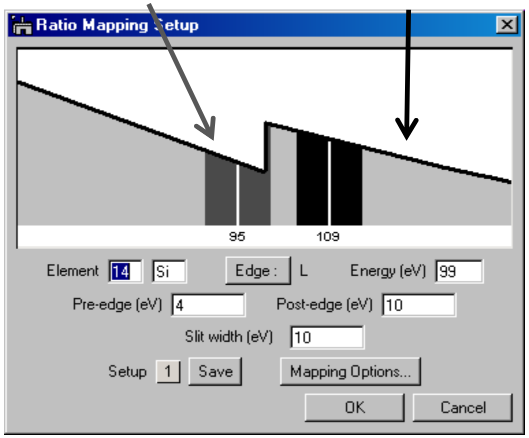
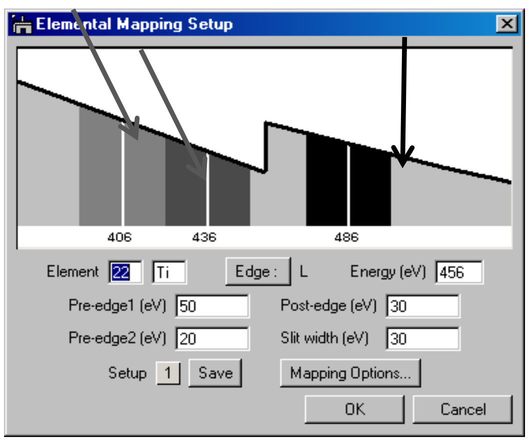
Pre-edge image

Post-edge image

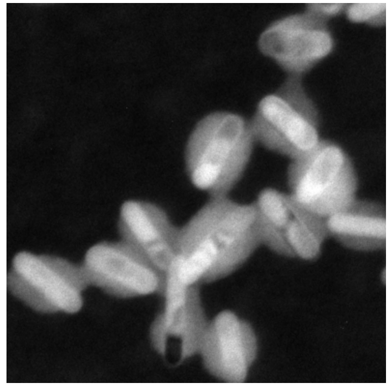


Jump Ratio Imaging

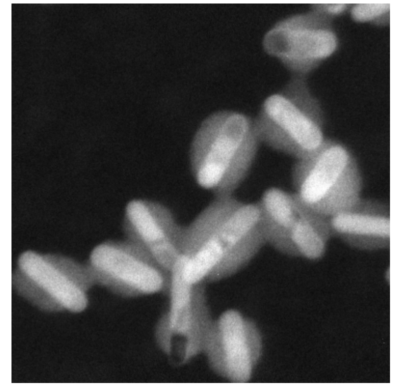


Three Window Mapping

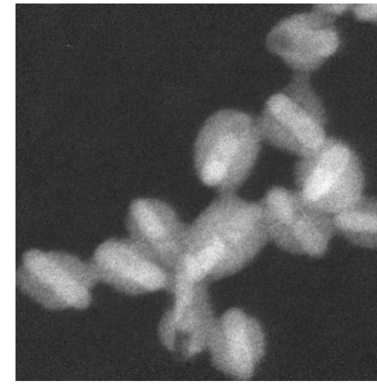
Post-edge 1

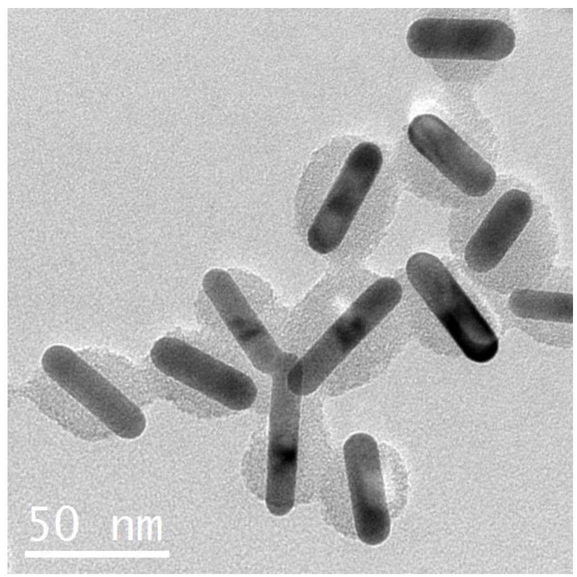


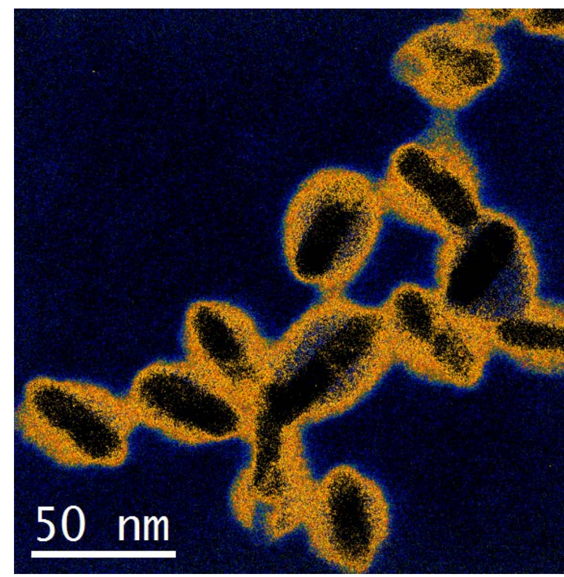
Post-edge 2



Post-edge







Fast and Easy!

C. Chen, on 2010F, MRL

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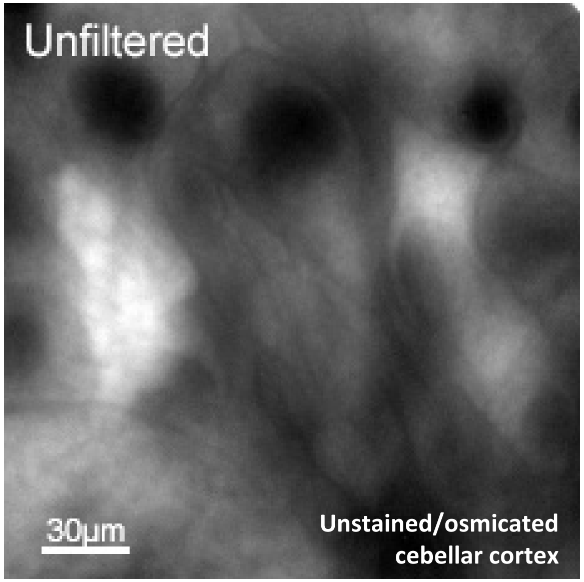
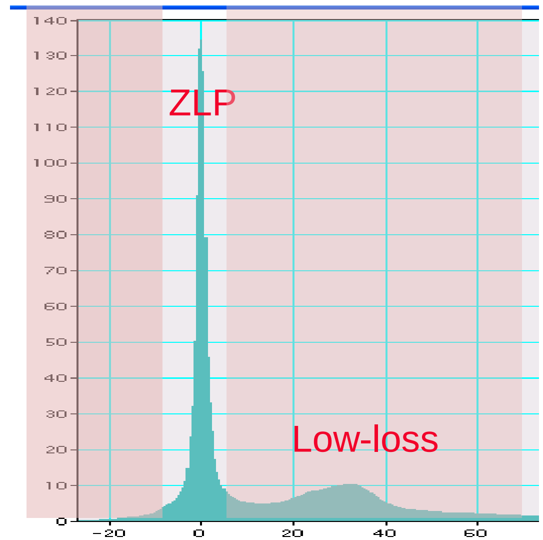
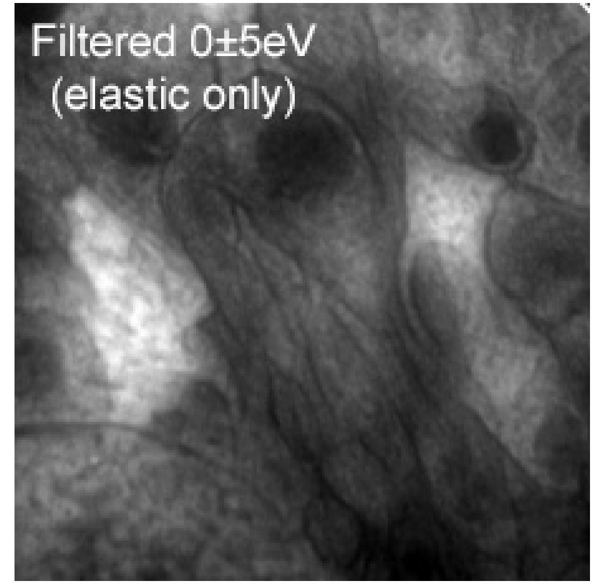
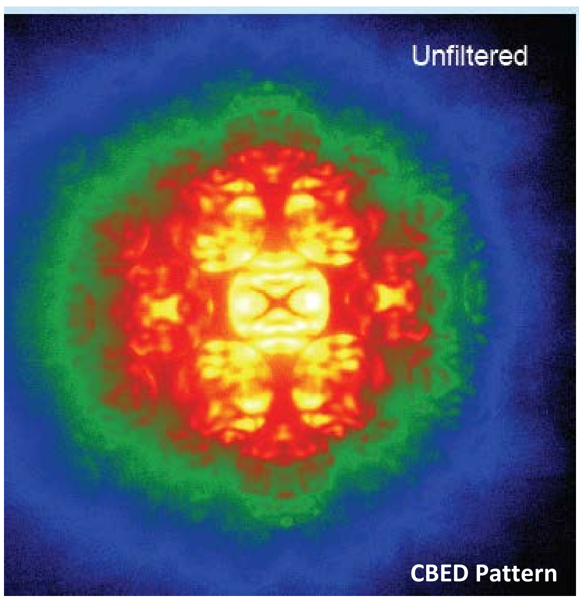
EF-TEM Imaging: Using Zero-loss



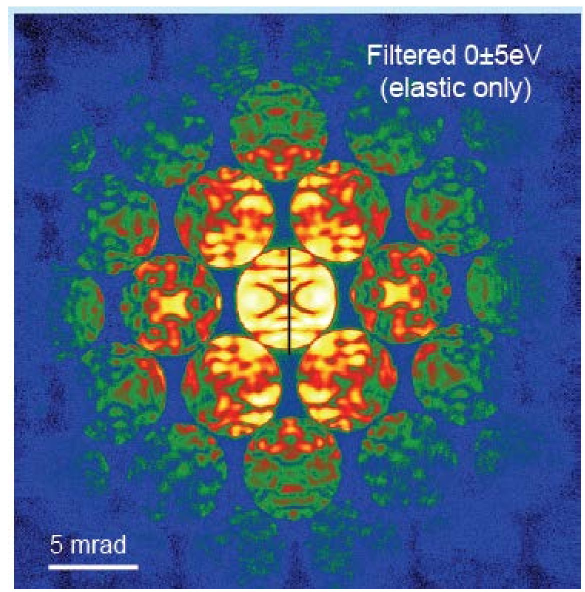






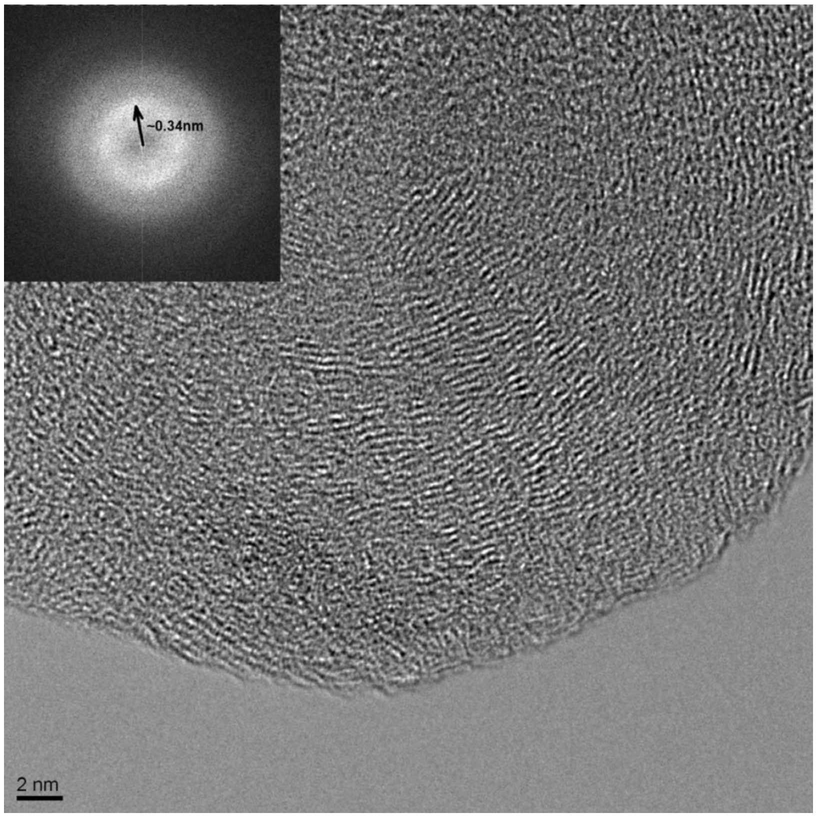
- Elastic electrons only
  "Inelastic fog" removed
- Contrast enhanced (Good for medium thick samples)

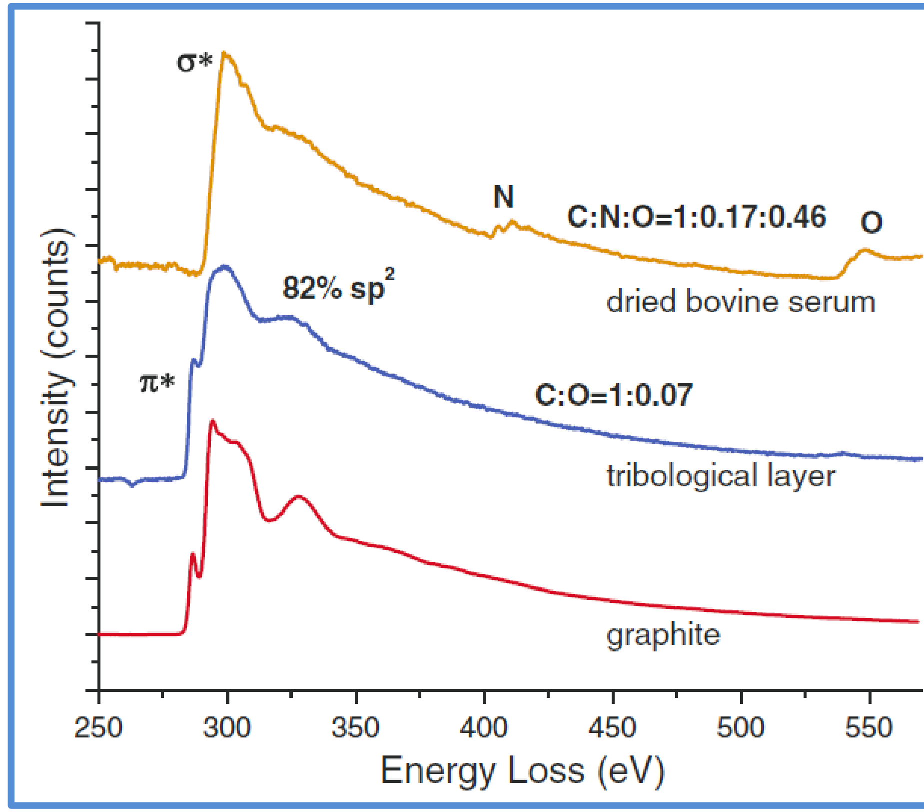
Fast and easy!



Gatan Review of EFTEM Fundamentals

Graphitic Tribological Layers in M-on-M Hip Replacements





Science, 334, 1687, 2011

HREM image and EELS spectrum showing the graphitic nature of a thin surface layer

Quantification

- **EDS**

$$\frac{C_A}{C_B} = k_{AB}\frac{I_A}{I_B}$$

Cliff-Lorimer equation for thin film EDS

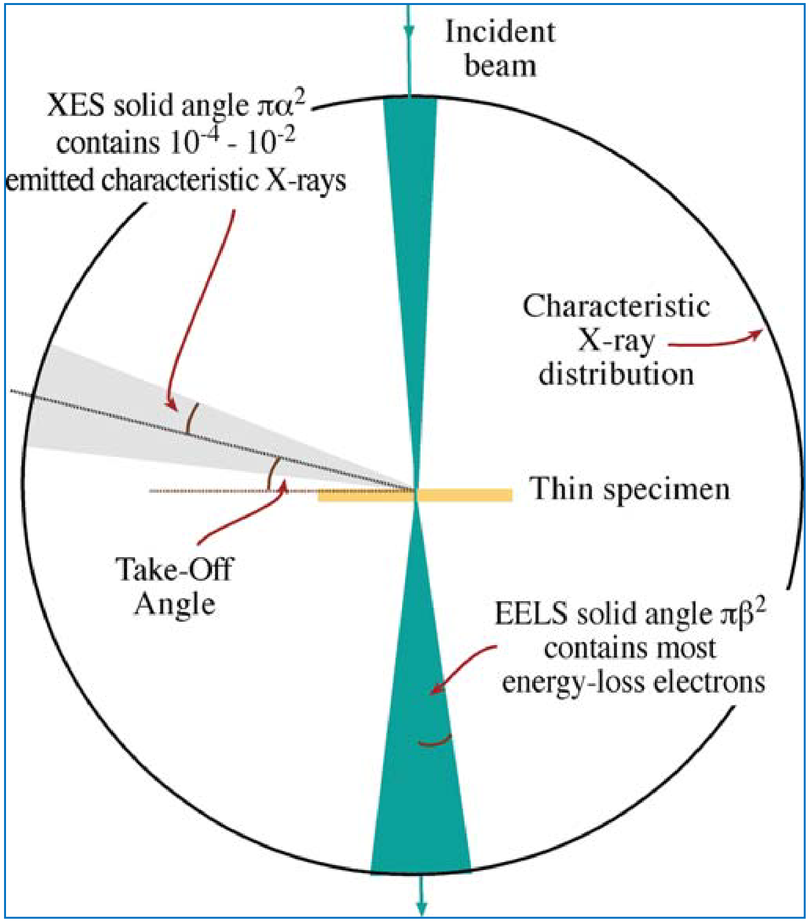
- **EELS (Ratio)**

$$\frac{N_A}{N_B} = \frac{I_K^A(\beta\Delta)\,\boxed{\sigma_K^B(\beta\Delta)}}{I_K B(\beta\Delta)\,\boxed{\sigma_K^A(\beta\Delta)}}$$

- **Absolute quantification (atoms / nm²)**

$$N = \frac{I_K(\beta\Delta)}{I_l(\beta\Delta)\sigma_K(\beta\Delta)}$$

I_K: sum of counts in edge k,
I_L: total intensity (including zero loss),
σ_K: partial cross-section for ionization.
Δ integration window, β collection angle



Williams and Carter 2009

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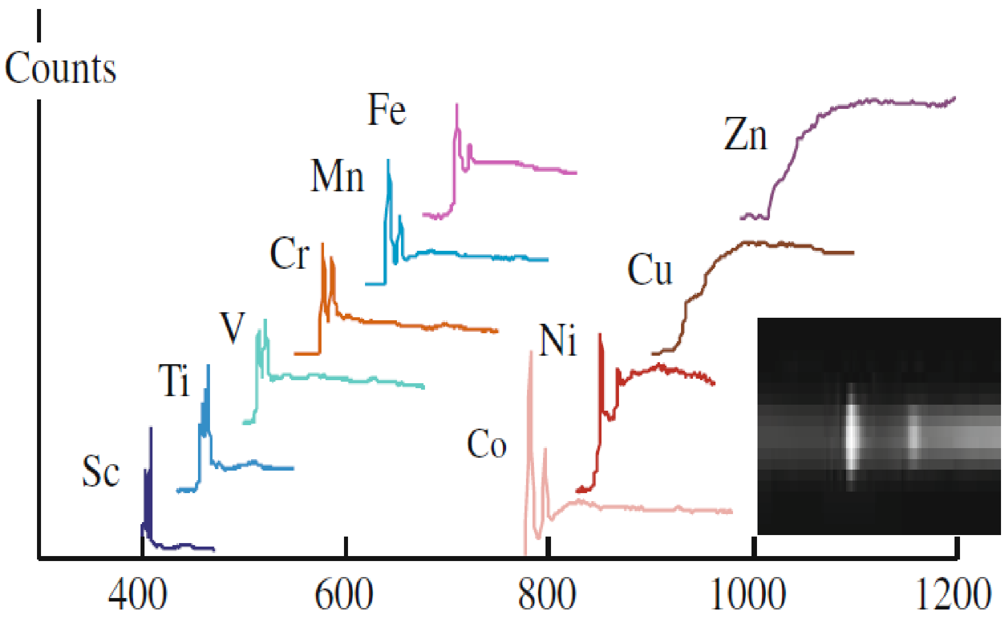
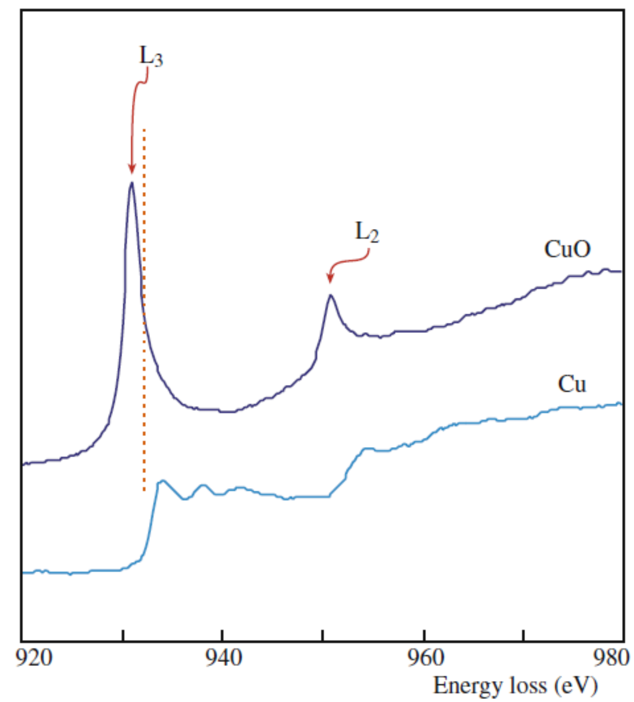
EELS Near Edge Fine Structure (ELNES): White Lines



Williams and Carter 2009



White lines in transition metals

Changes in the Cu $L_{2,3}$ edge with oxidation: ELNES Finger-Printing

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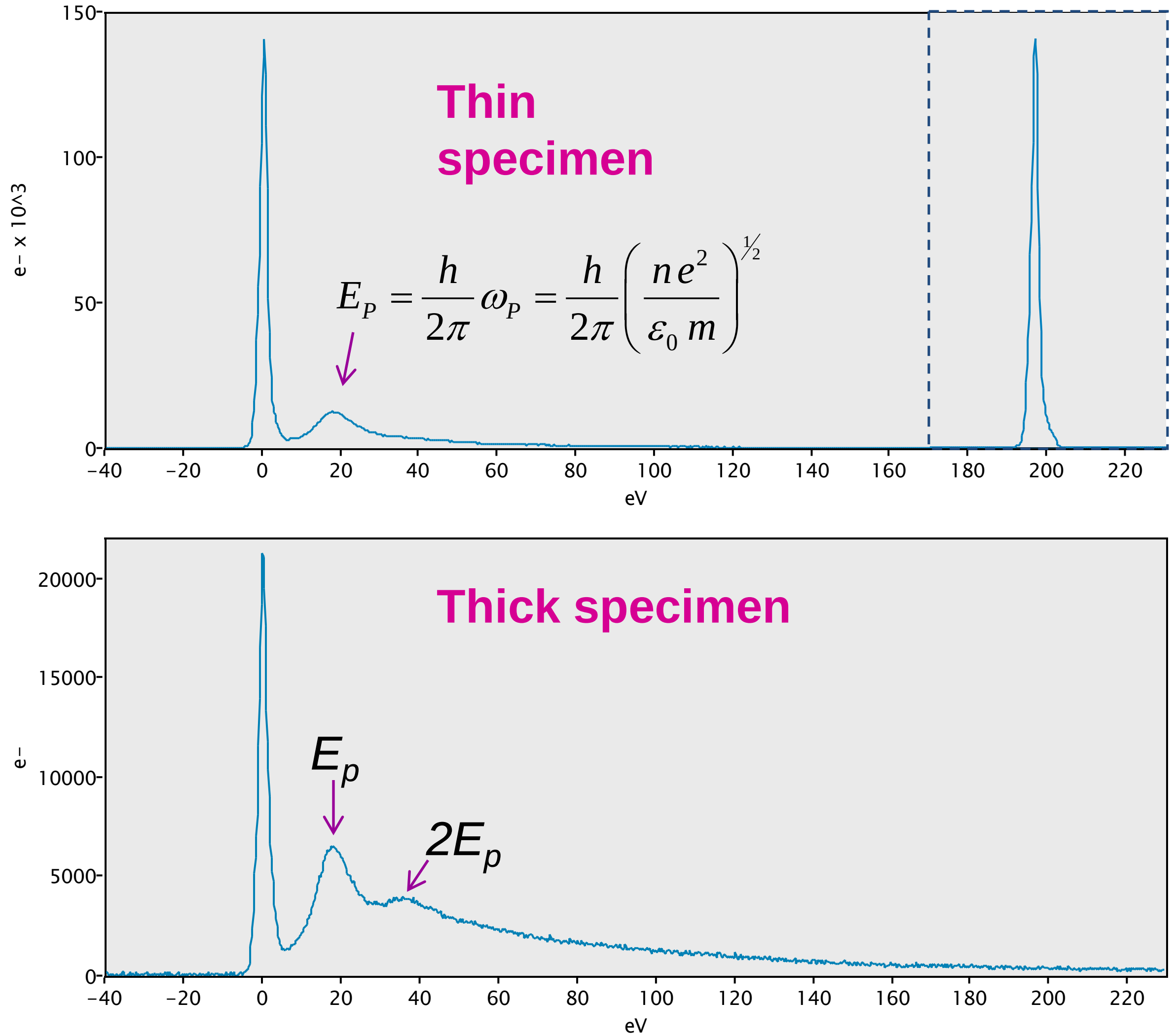
STEM-EELS Measurement of Local Thickness



Log-ratio method

$$t = \lambda_p \times \ln(I_o / I_T)$$

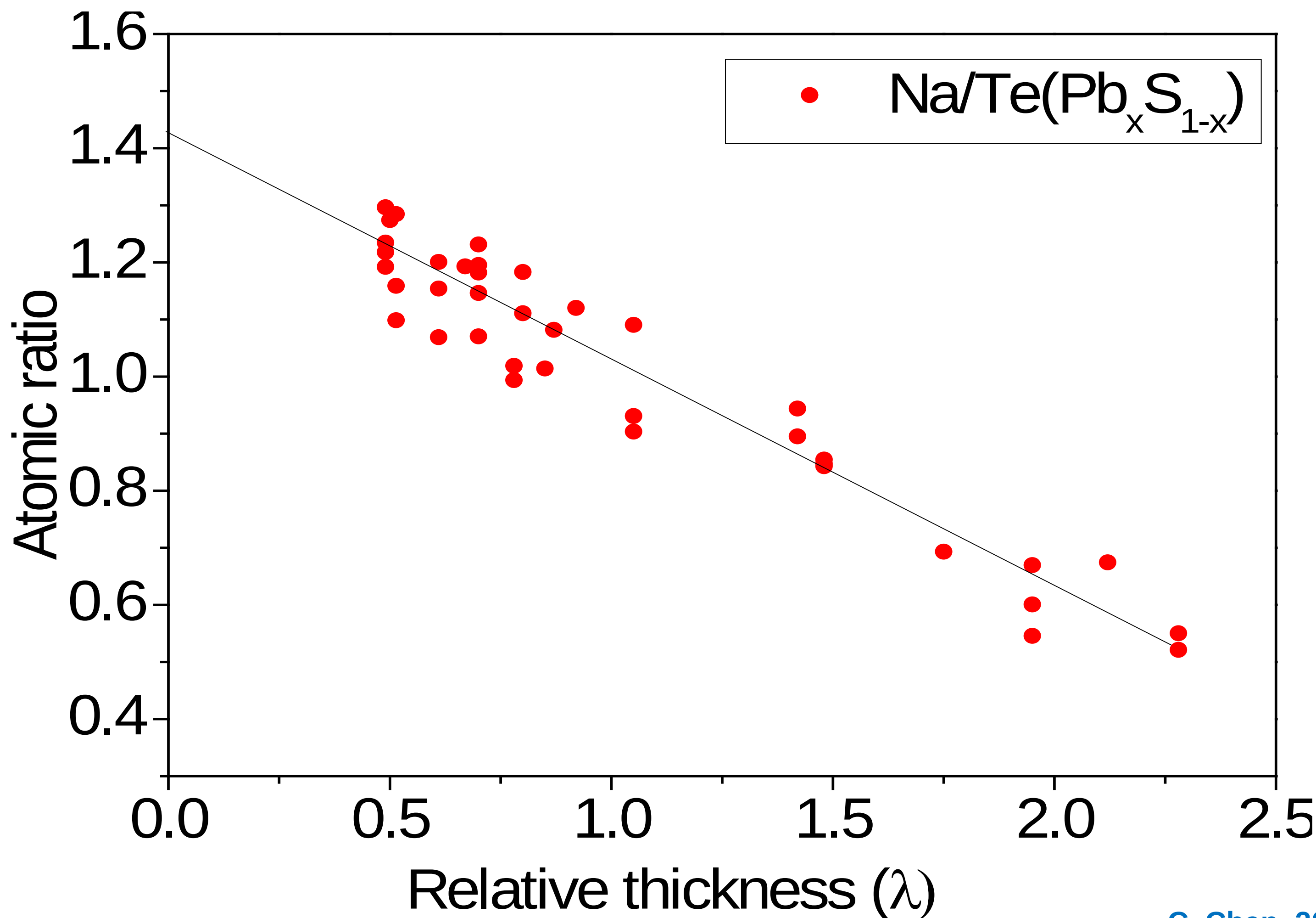
t = thickness
λ_P= Plasmon MFP,
I_o= zero-loss peak
I_T= total spectrum

Relative thickness:

Reliable,
Fast and online,
High spatial resolution

Quantitative Composition Analysis: Combining EDS-EELS

Combine STEM-EDS & STEM-EELS



C. Chen, 2013

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Comparison of EELS and EDS

EELS (primary)	EDS (Secondary)
• Atomic composition • Chemical bonding • Electronic properties • Surface properties	• Atomic composition only
• Energy resolution: ~ 0.15-1 eV	• Energy resolution: ~ 130 eV (Mn K_α)
• Spatial resolution: 0.1 - 1 nm	• Spatial resolution: 0.1 nm – 10 nm
• Relatively complicated to use and to interpret	• Easy to use • Easy to interpret
• High collection efficiency • (close to 100 %)	• Low collection efficiency • (1-3%)
• Sensitive to lighter elements • Signal weak in high loss region	• Sensitive to heavier elements • Low yield for light elements

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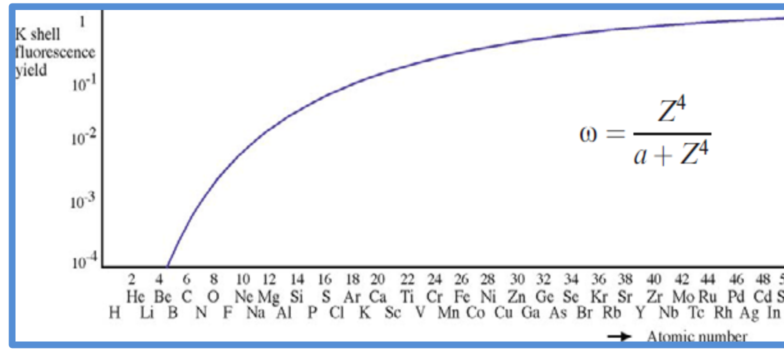
Comparison of EELS and EDS

- EDS: low x-ray yield for lower Z
- EDS: Low energy x-ray below detection limit
- EDS: higher energy favorable (>1KV ideally)
- EELS: Lower loss favorable (<1KV ideally)



Primary vs Secondary:

- EELS: Events causing ionization
- EDS: Events after Ionization

One Ionization Event (~EELS)

$\Downarrow$

Several Relaxation Events (~EDS/AES)

$E_{loss} > E_c > E_{EDS}$

TABLE 4.2 Difference Between E_c and E_K

Element	Critical ionization energy E_c (keV)	X-ray energy E_K (keV)
C	0.282	0.277
Al	1.562	1.487
Ca	4.034	3.692
Cu	8.993	8.048
Ag	25.531	22.163

Note that the energies may be affected by bonding states but shifts will only be a few eV.

Williams and Carter 2009

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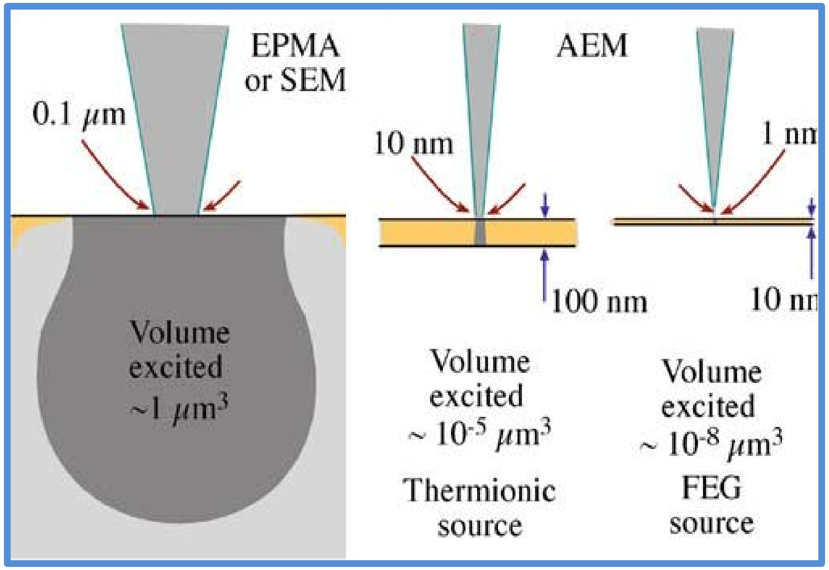
Spatial Resolution (R) and Sensitivity (MMF, minimum mass fraction)

Bulk **Thin** **Ultra-thin**



MMF 0.01%
$3x10^6$ atoms

MMF 0.01%
300 atoms

MMF 0.01%
1 atom

Williams and Carter 2009

Beam diameter & spreading

$$b = 8 \times 10^{-12} \frac{Z}{E_0} (N_v)^{1/2} t^{3/2}$$

E_0 energy, t thickness, Z atomic number,
N number of atoms per volume

- **Thin foil: better R,**
- **Thin foil: worse MMF**
- **Typical MMF: 0.1–1%**
- **C_s correction improves both**
- **Achieving atomic resolution**
- **Approaching atomic level detection**

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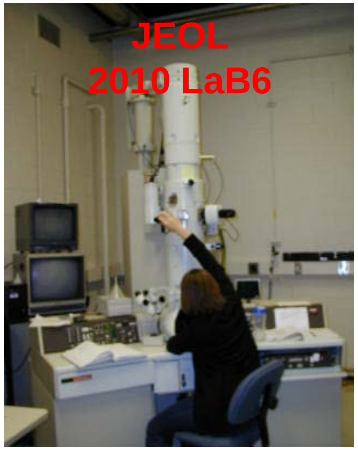
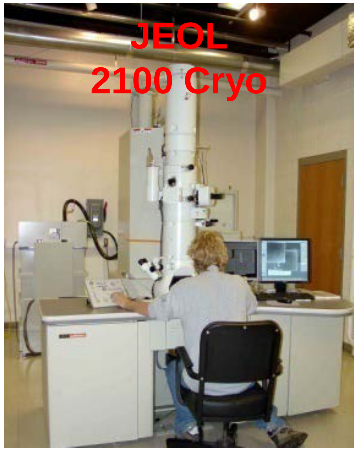
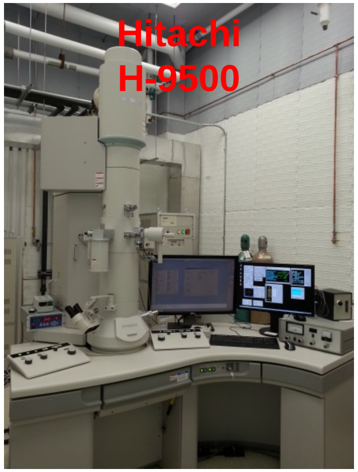
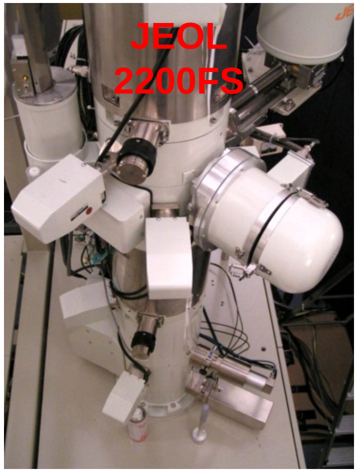
List of TEMs and Functions at UIUC

1. JEOL JEM 2010 LaB6 TEM
   - TEM, low dose, NBD, HRTEM, in-situ experiments
2. JEOL JEM 2100 LaB6 (Cryo-TEM)
   - TEM, Low dose, special cryo-shielding
3. Hitachi H-9500 TEM (LaB6, 300 kV)
   - Environmental TEM (in-situ heating and gas reaction)
   - Gatan K2 camera (400 fps, or 1600 fps at reduced size)
   - Dynamic TEM (developing)
3. JEOL JEM 2010 F Analytical (S)TEM
   - EDS, STEM, Z-contrast imaging
4. JEOL JEM 2200 FS C_s corrected (S)TEM
   - Ultra high resolution Z-contrast STEM, BF STEM, NBD, CBED, EDS, EELS, EFTEM
6. Hitachi H-600 TEM (W)
   - Biological samples, staff assisted only
6. ThermoFisher Scientific Themis Z STEM/TEM









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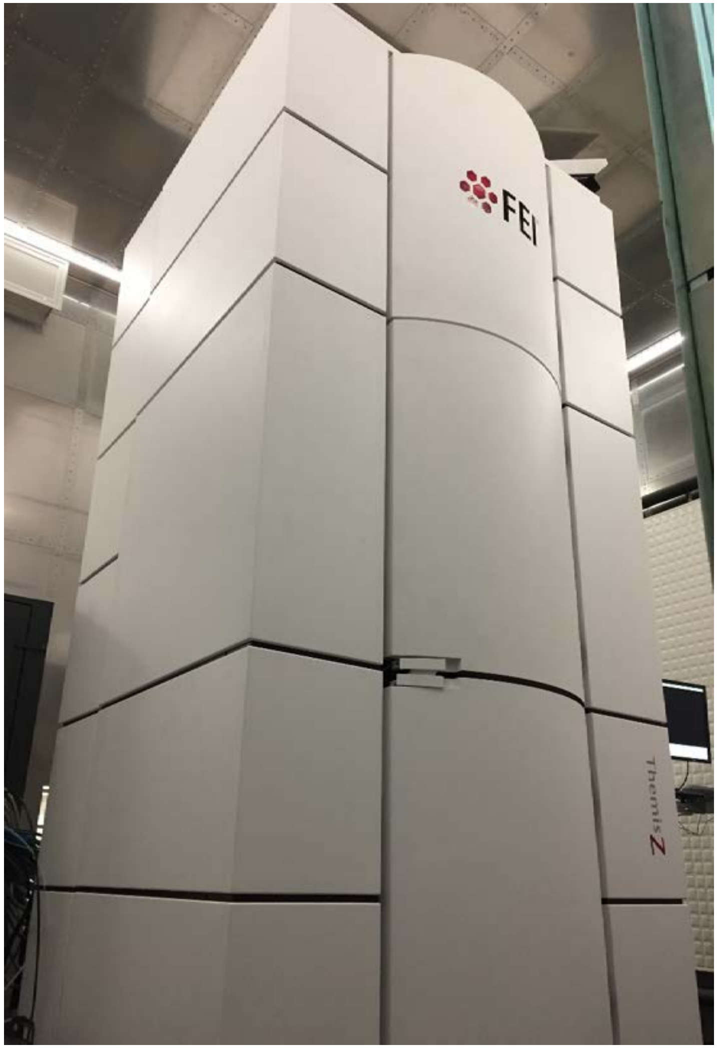
ThermoFisher Scientific Themis Z STEM/TEM:

60 – 300 kV Ultra-High Resolution Analytical STEM and TEM

- **X-FEG** high brightness electron source with energy **monochromator**
- Advanced **spherical aberration probe corrector** for STEM
    - <60 pm STEM resolution attainable @300kV
    - <110 pm STEM attainable @ 80 kV
    - <120 pm STEM attainable @ 60kV
- TEM mode attainable information transfer of: 300kV-60 pm (<0.2nm point-point), 200kV-80 pm, 60kV-100pm (Young's fringe information limit method).
- Chemical mapping down to atomic resolution in 2D and 3D
    - EELS - Ultra-fast Dual EELS detector for detection of light elements, mapping bonding states
    - EDS - 4-detector EDX for fast and atomic or nanoscale chemical analysis
- Monochromator for high energy resolution EELS
    - Mapping of plasmonic modes, fine structure for local chemical and electronic states
    - Measurement of local bandgap in semiconductors
    - Reduced chromatic aberration for imaging (in particular low kV's)
- Lower kV operation (60 and 80 kV) for 2D electronic materials and low dimensional molecular structures
- STEM/TEM Tomography acquisition
    - 3D imaging (down to ~1 nm resolution)
    - 3D chemical mapping (EDS)
    - Diffraction tomography
- OptiSTEM+: Automated fine tuning low order aberrations (C1\A1, A2\B2)
- OptiMono: Automated Monochromator tuning
- iDPC: Integrated differential phase contrast (a new ABF) for imaging light elements simultaneous with heavy element (more linear with Z) at low dose



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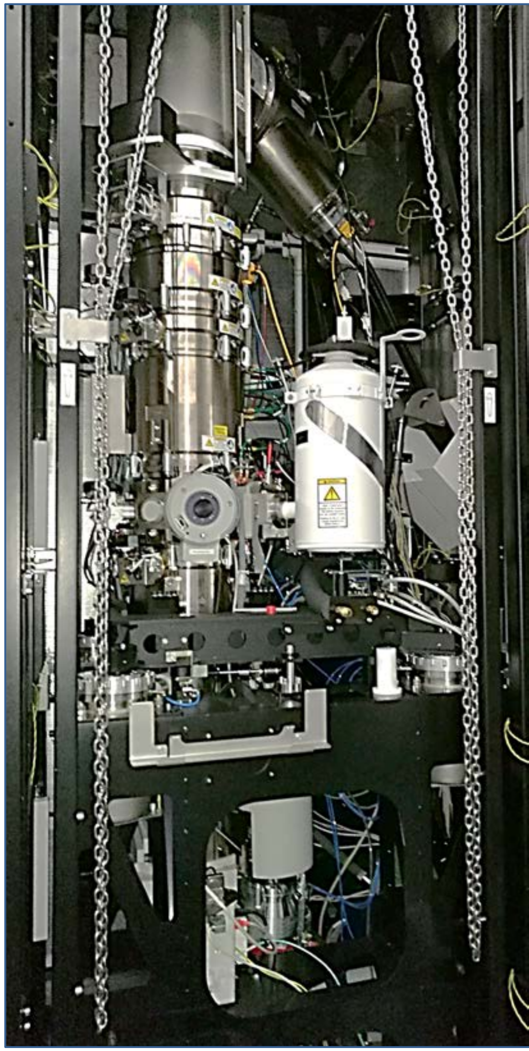
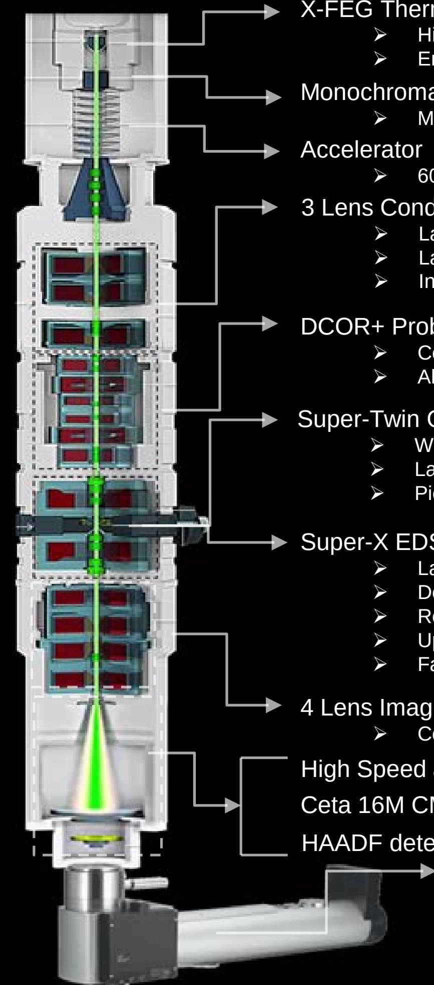
ThermoFisher Scientific Themis Z STEM/TEM:





X-FEG Thermal Field Emission Gun
- High Brightness (>7x 10^7 A/m^2 sr V)
- Energy Spread ~1eV without monochromator → unfiltered mode)

Monochromator
- Minimum energy spread better than 0.15eV @300kV

Accelerator
- 60-300 kV (60, 80, and 300kV alignments currently available)

3 Lens Condenser System
- Large parallel Illumination range
- Large convergence angle range
- Indication of convergence angle and size of illumination area

DCOR+ Probe Cs corrector
- Correction up to 4^{th} order aberrations (5^{th} order optimized)
- Allows use of larger apertures and higher currents in sub-angstrom probes.

Super-Twin Objective Lens
- Wide pole piece gap (5.4 mm)
- Large sample tilt range (+/-35+ deg, +/-70+ deg w/tomography holder)
- Piezo stage with high stability and 20 pm step size (x,y,z)

Super-X EDS X-ray detector
- Large solid angle of collection (0.7 srad, array of 4 windowless SDD's)
- Detection of all elements down to Boron
- Resolution @Mn Ka of <136 eV, 10kcps
- Up to 200 kcps count rate
- Fast mapping (dwell times down to 10μs/pixel)

4 Lens Imaging/Projector System
- Constant power (high stability)/ Rotation free

High Speed and dynamic range Flu Camera replaces conventional fluorescent screen

Ceta 16M CMOS 4k x 4k Camera

HAADF detector and the FEI-triple BF/DF detector for STEM incl. DPC and iDPC

Gatan GIF Quantum ERS with Ultrafast Dual EELS
- High speed (1000 spectra/second)
- Near simultaneous acquisition of 2 energy ranges
- Integrated Gatan BF/DF detector
- UltraScan 2Kx2K CCD camera
- Energy Filtered TEM

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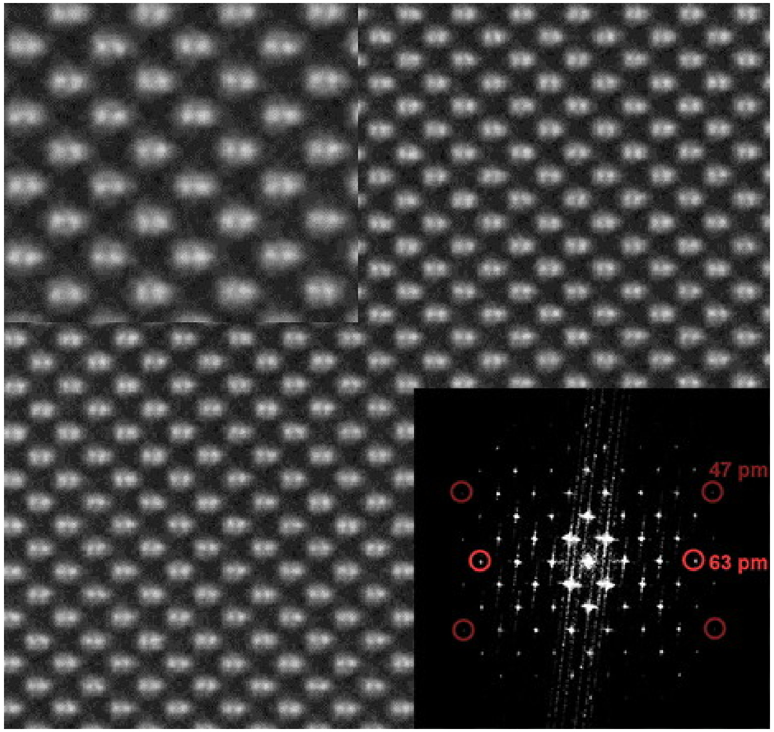
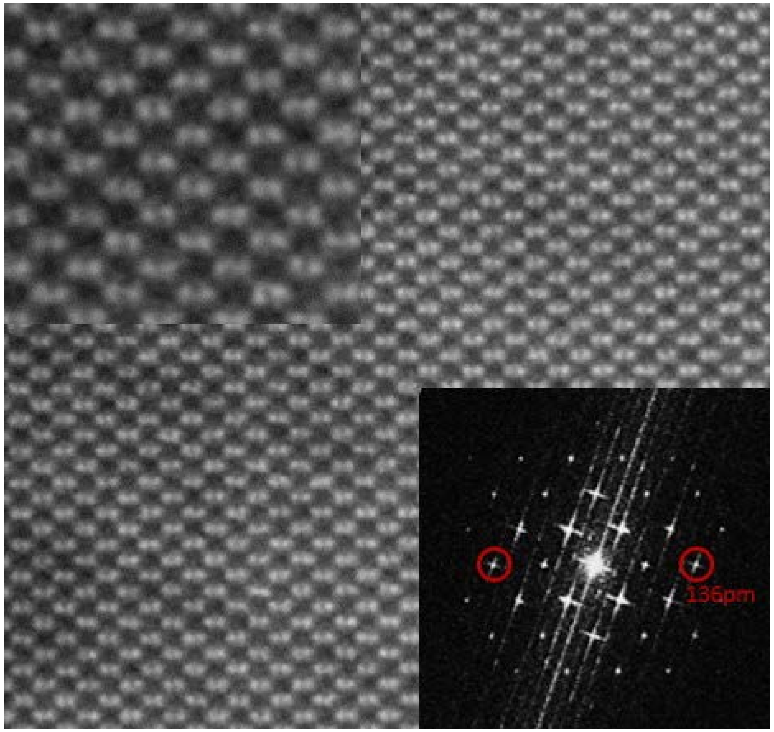
Imaging Performance: Themis Z STEM:

GaN [211] imaged at 300 kV showing <63 pm resolution

Si [110] imaged at 60 kV showing <136 pm resolution





Images - Thermo Fisher Scientific
https://www.fei.com/products/tem/themis-z-for-materials-science/

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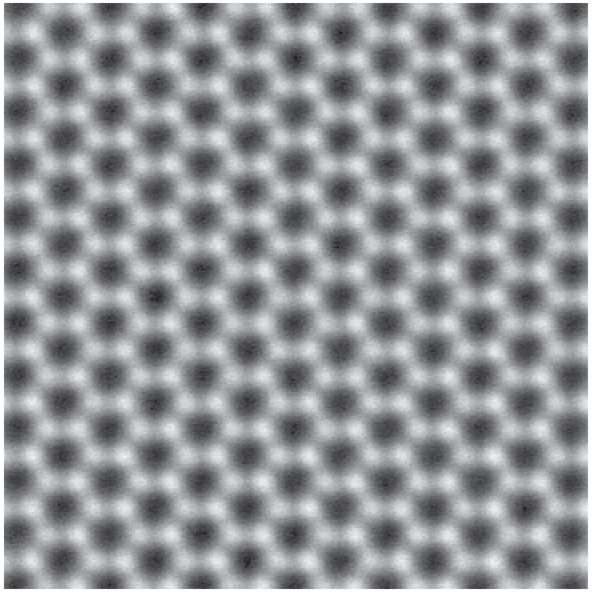
Imaging : Themis Z STEM Low kV & iDPC:

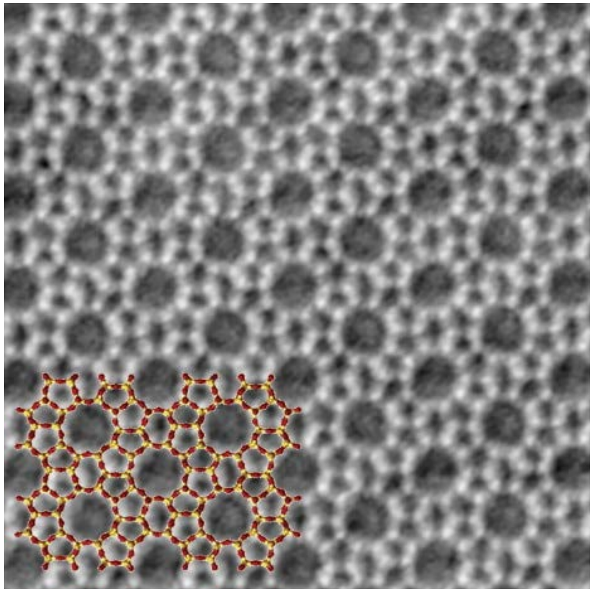
- Low kV operation (60/80 kV) for knock-on damage sensitive materials
- Low dose sensitivity for dose sensitive materials
- Low atomic number imaging sensitivity with iDPC

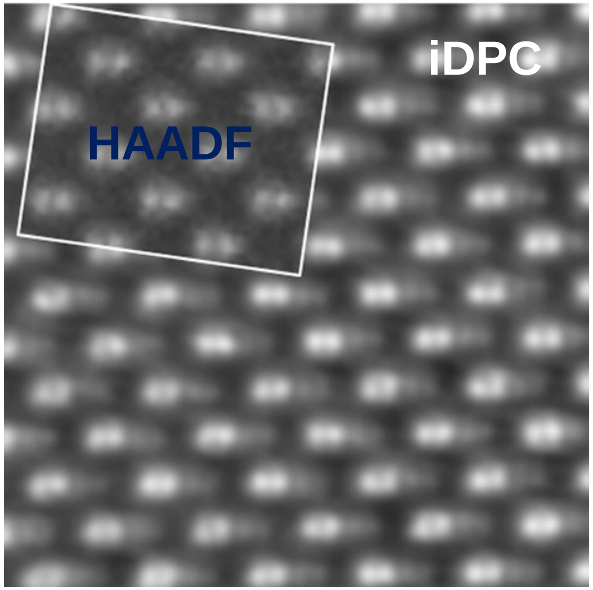
HAADF STEM image of a graphene lattice imaged at 60kV.

Zeolite imaged at 300kV and <1pA with iDPC. Oxygen atoms are visible with extreme low doses.

GaN [211] imaged at 300 kV with iDPC Ga and N dumbbells are clearly visible.







Images - Thermo Fisher Scientific
https://www.fei.com/products/tem/themis-z-for-materials-science/

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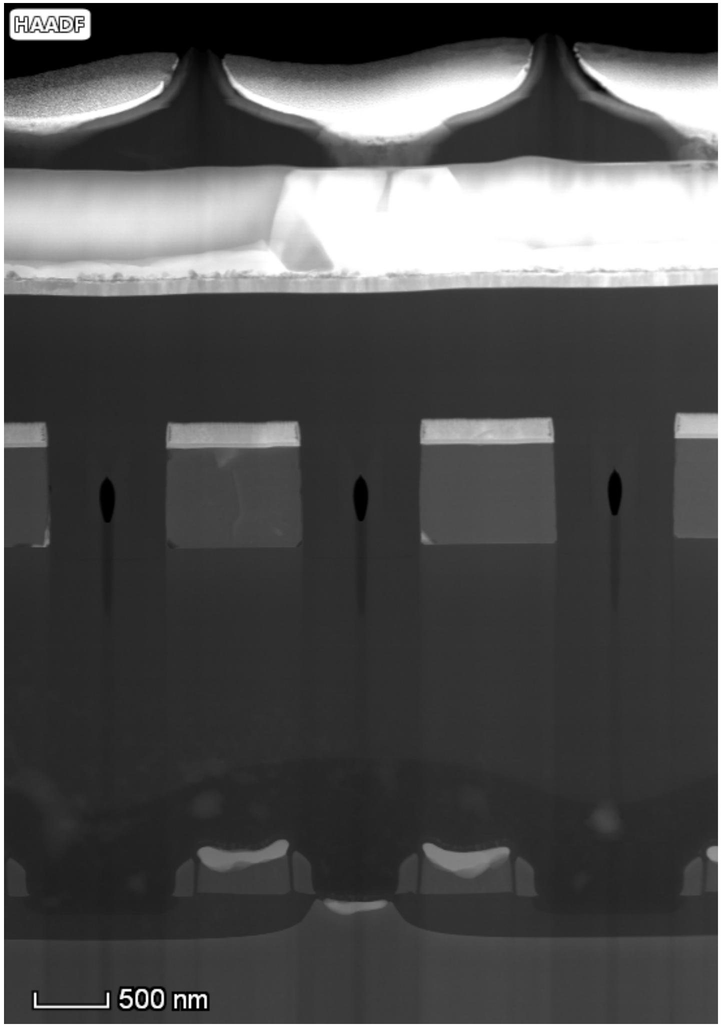
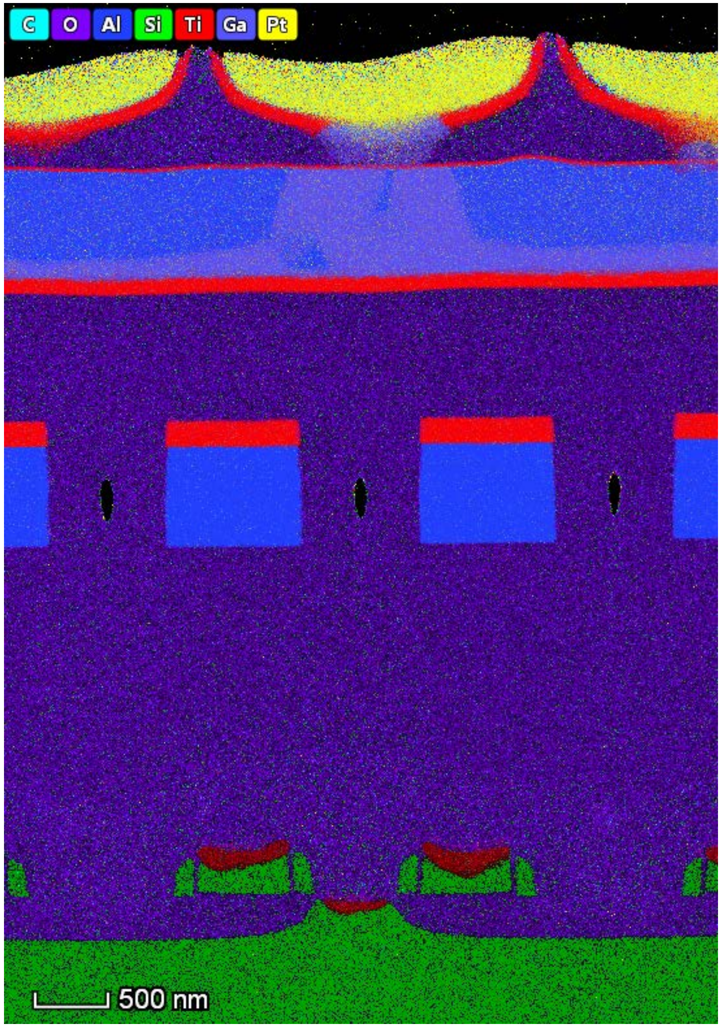
High throughput spectrum imaging: Microprocessor X-TEM EDS





300KV, 1.2nA, 40K cps, 12 minutes

J. Mabon, C. Chen - Themis Z, MRL

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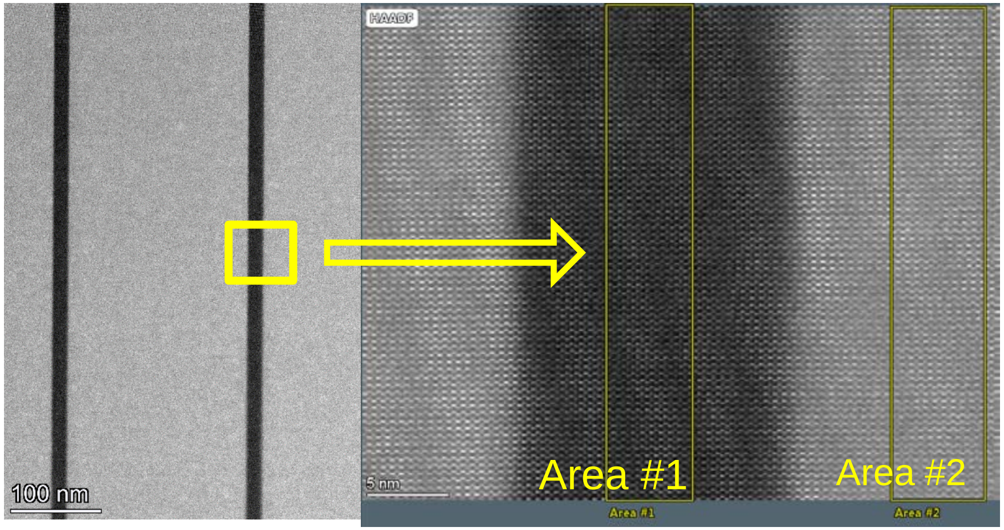
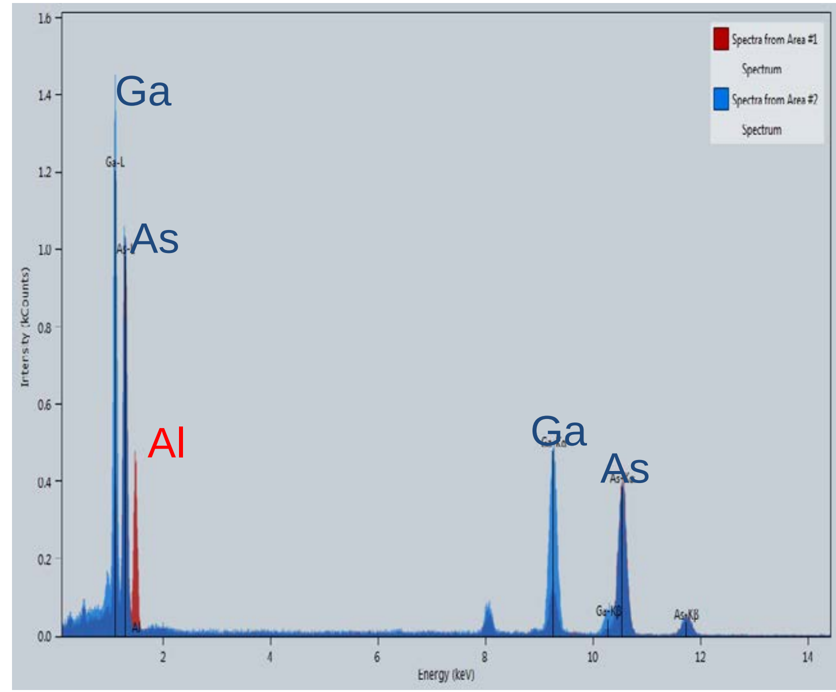
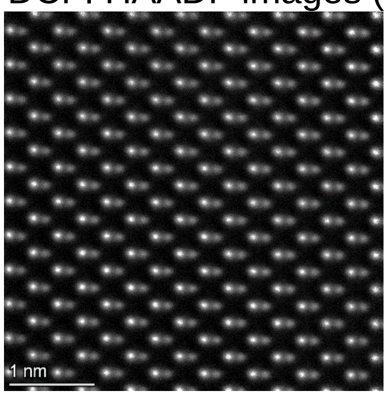
EDS Spectrum Imaging and Analysis:

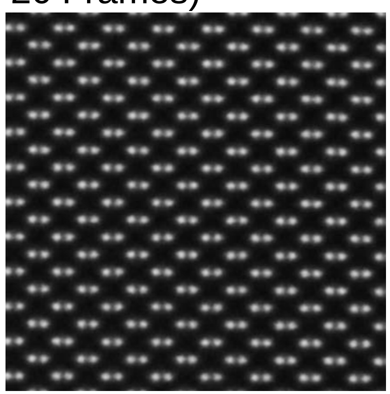
AlGaAs – GaAs multilayer





DCFI HAADF images (~20 Frames)





Area #1 Area #2

2019-05-28 09:13:25 Analysis of spectrum: Spectra from Area #1

Z	Element	Family	Atomic Fraction (%)	Atomic Error (%)	Mass Fraction (%)	Mass Error (%)	Fit error (%)
13	Al	K	33.04	5.05	15.26	1.47	2.38
31	Ga	K	12.83	2.57	15.32	2.47	0.39
33	As	K	54.13	10.83	69.42	11.18	0.16

2019-05-28 09:13:25 Analysis of spectrum: Spectra from Area #2

Z	Element	Family	Atomic Fraction (%)	Atomic Error (%)	Mass Fraction (%)	Mass Error (%)	Fit error (%)
13	Al	K	0.00	0.05	0.00	0.02	0.00
31	Ga	K	51.29	10.66	49.49	7.97	0.14
33	As	K	48.71	10.12	50.51	8.12	0.24

EDS 4 minute acquisition time, 50 pA, 300kV

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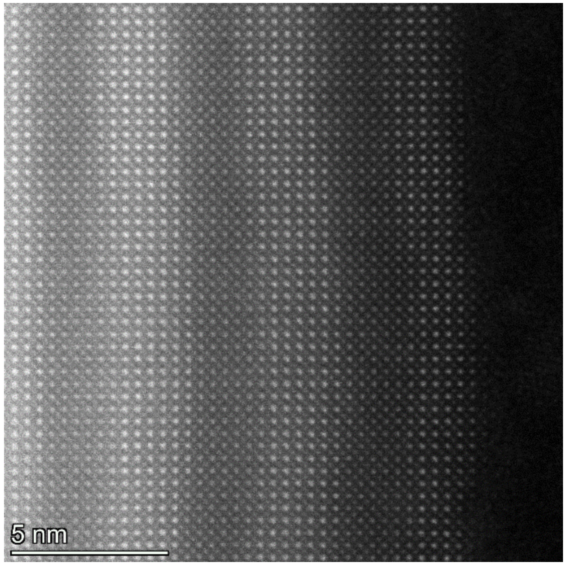
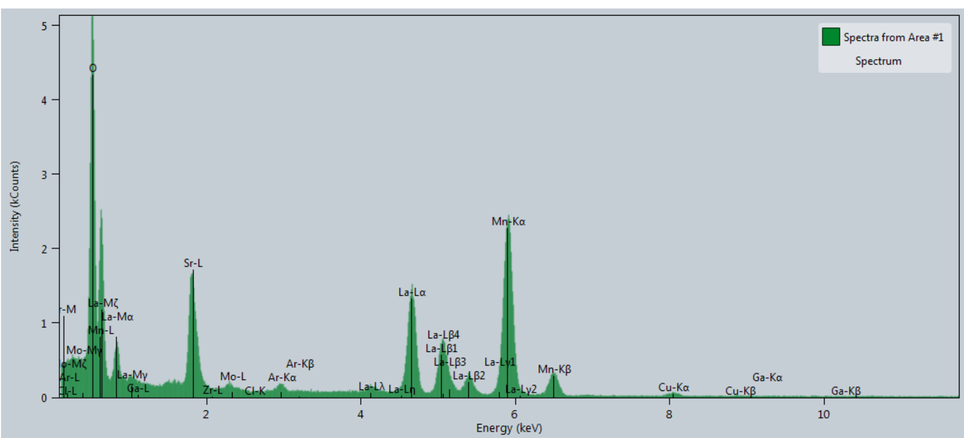
Fast EDS spectrum imaging @ high resolution: LMO/SMO

DCFI HAADF





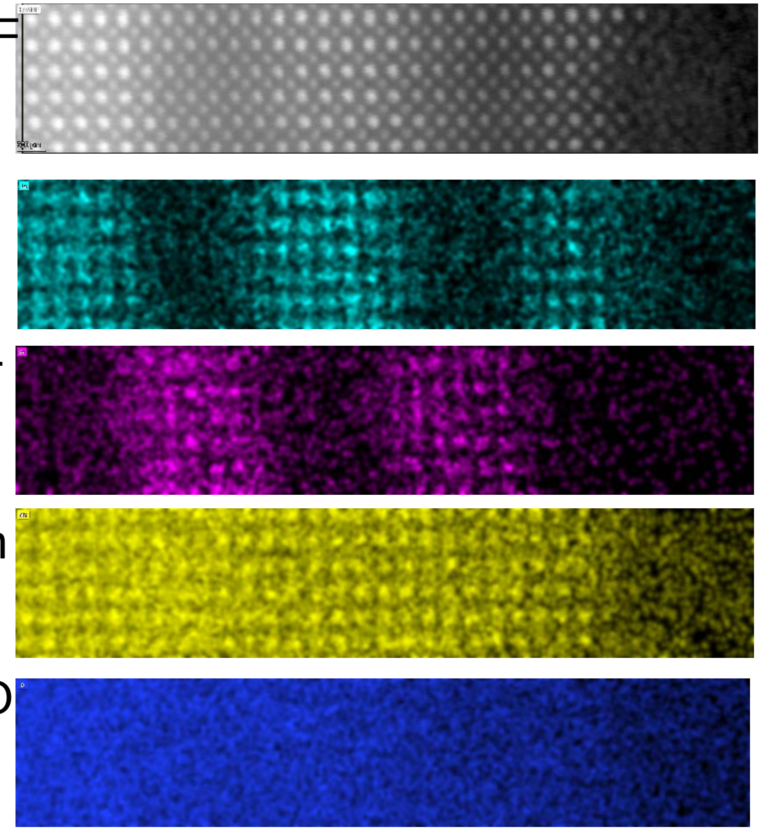
HAADF

La

Sr

Mn

O



300KV, 140 pA, ~5 minutes

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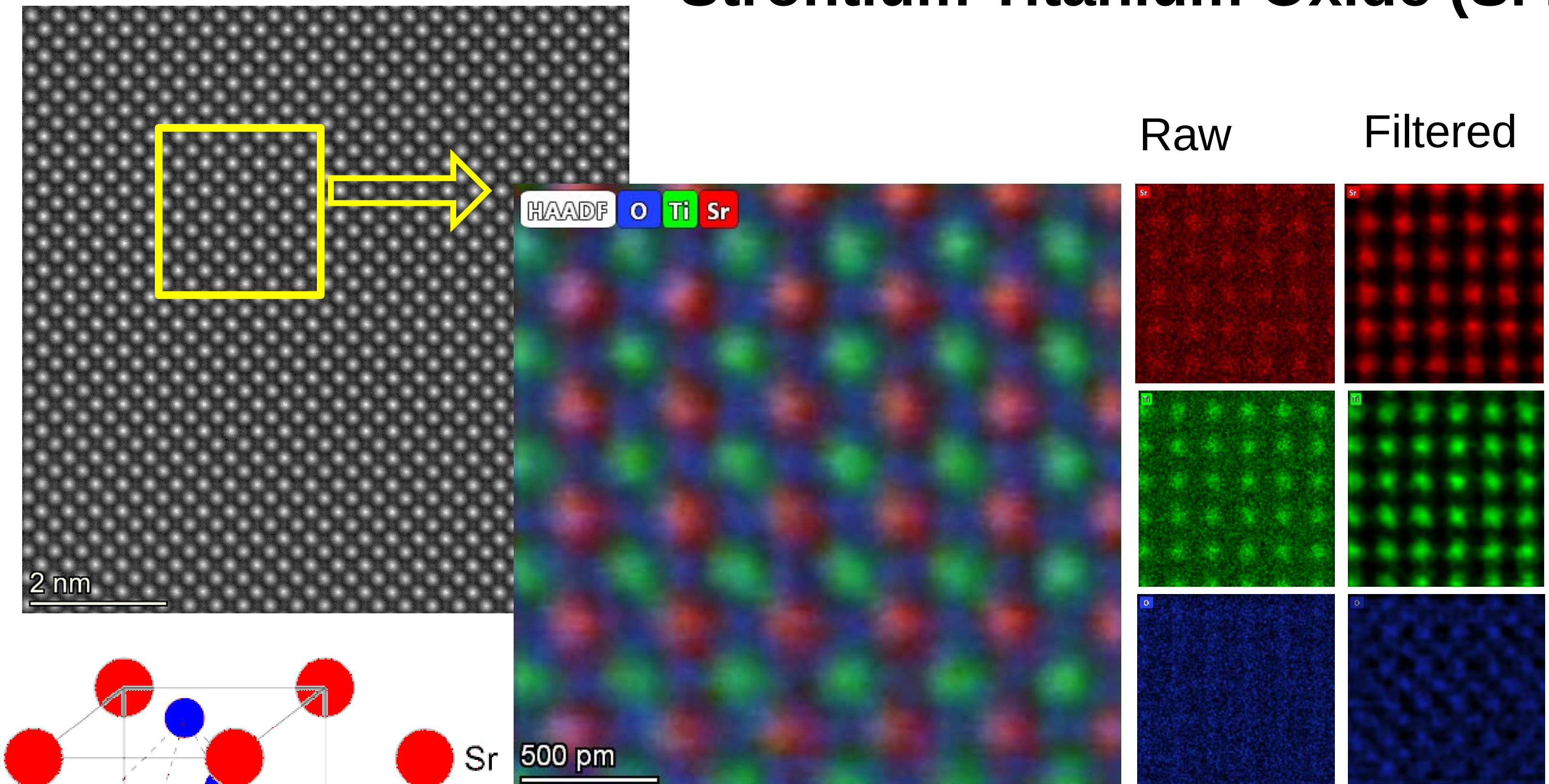
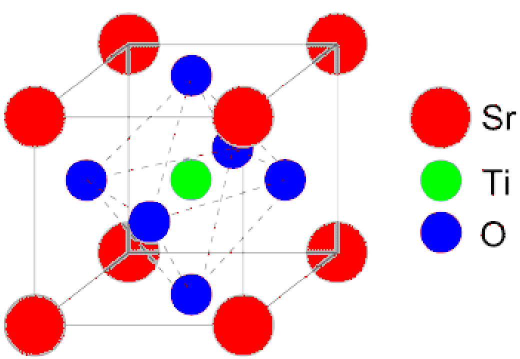
Atomic resolution EDS Spectrum Imaging and Mapping

DCFI HAADF

Strontium Titanium Oxide ($SrTiO_3$)

Raw Filtered





12 minute acquisition time, 100pA, 300kV

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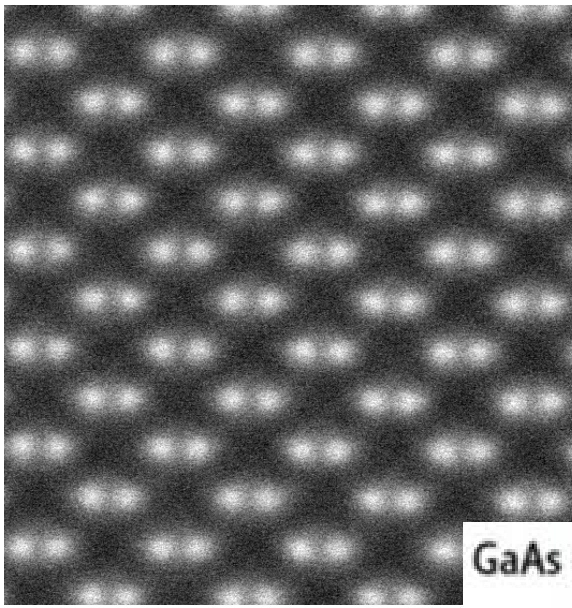
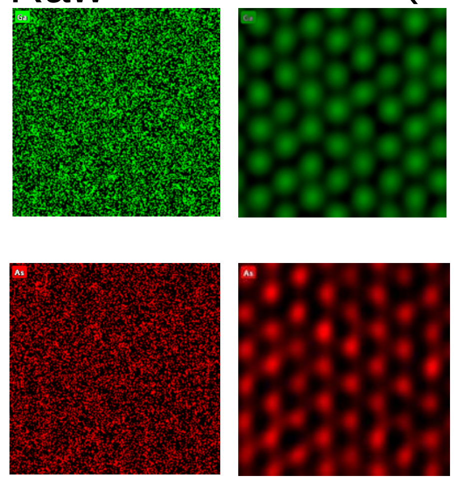
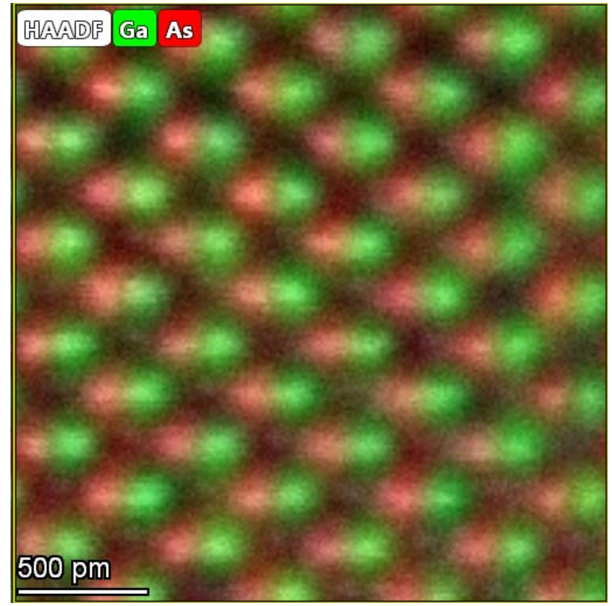
Atomic resolution EDS spectrum imaging - GaAs

HAADF

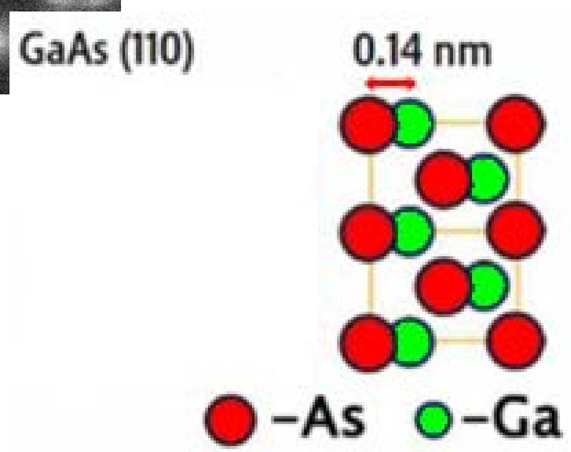


Raw | Filtered (Radial Wiener de-blur)





Overlay of filtered maps on HAADF



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EDS 1.5 minute acquisition time, 50 pA, 300kV

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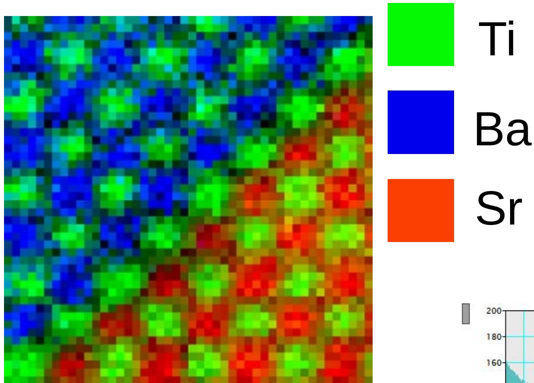
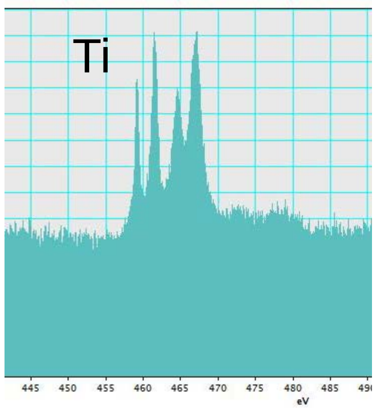
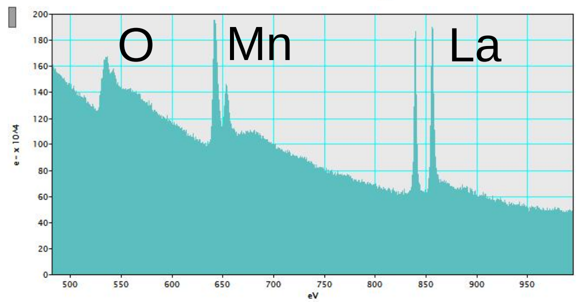
Atomic resolution EELS mapping and analysis (with monochromator):

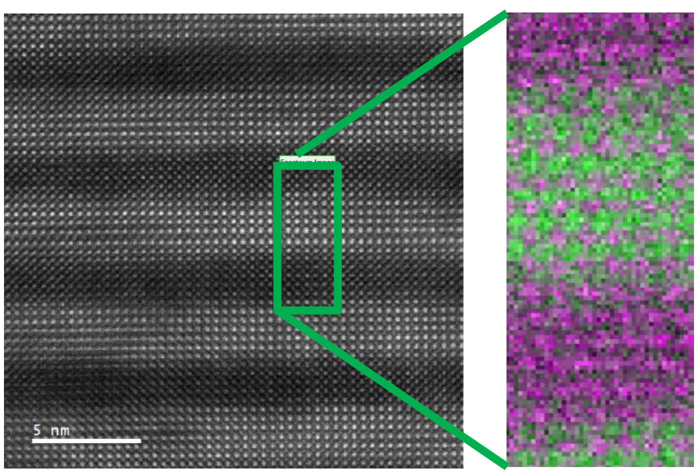
BaTiO3/SrTiO3
interface







DCFI HAADF



Lanthanum Manganese Oxide/
Strontium Manganese Oxide Multilayer

Images - ThermoFisher Scientific - U of I Instrument Demonstration and
https://www.fei.com/products/tem/themis-z-for-materials-science/

EELS Spectra J. Mabon

Also possible:

- Mapping of light elements
- Mapping of bonding, oxidation states
- Measurement of local electronic and optical states
- Mapping of plasmon excitations

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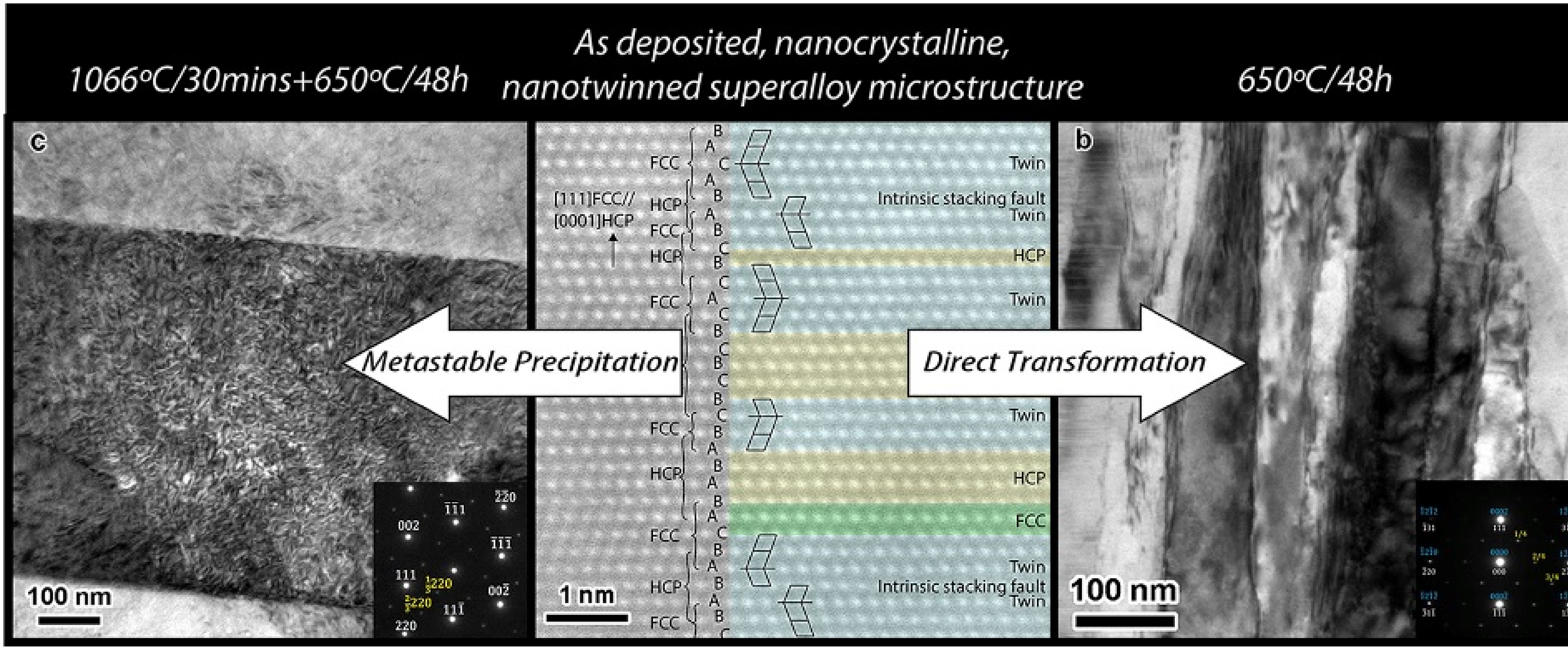
Example: Study of a defects in nanocrystalline alloy

Imaging of planar defects in the nanotwinned, nanocrystalline microstructure of a Ni-25Mo-8Cr superalloy



The HRSTEM shows the atomic ordering in the side view of a direct current magnetron sputtered film. FCC- and HCP-like ordering behavior are noted on the left; on the right FCC and HCP regions are indicated in green and yellow, and regions that contain nanotwinned FCC are indicated in blue.

M.G. Emigh, R.D. McAuliffe, C. Chen, J.C. Mabon, T. Weihs, K.J. Hemker, J.A. Krogstad
Influence of a nanotwinned, nanocrystalline microstructure on aging of a Ni-25Mo-8Cr superalloy
Acta Mater., 156 (2018), pp. 411-419

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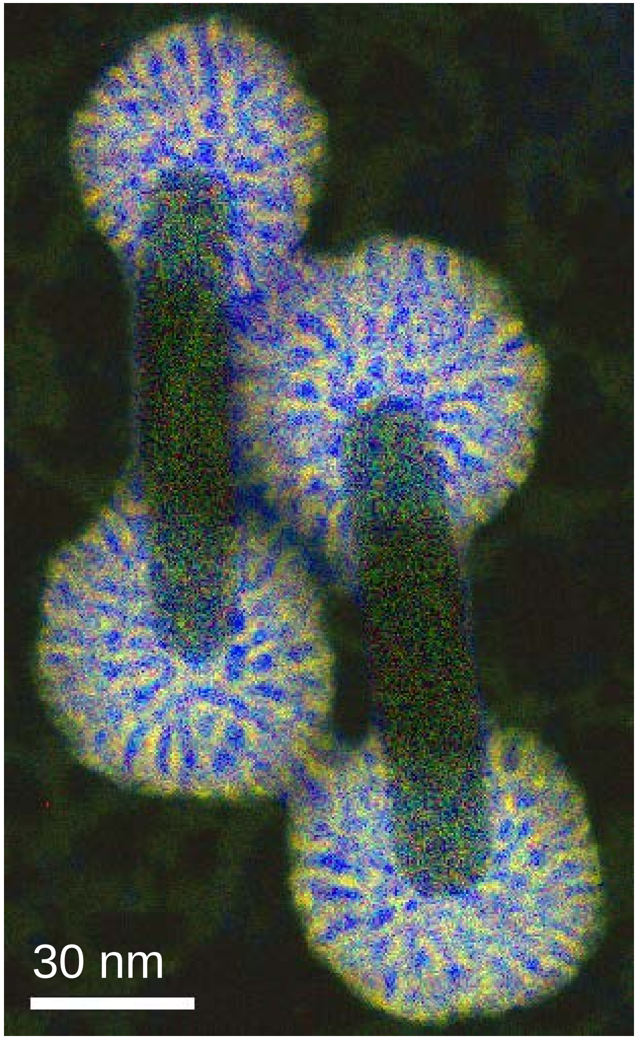
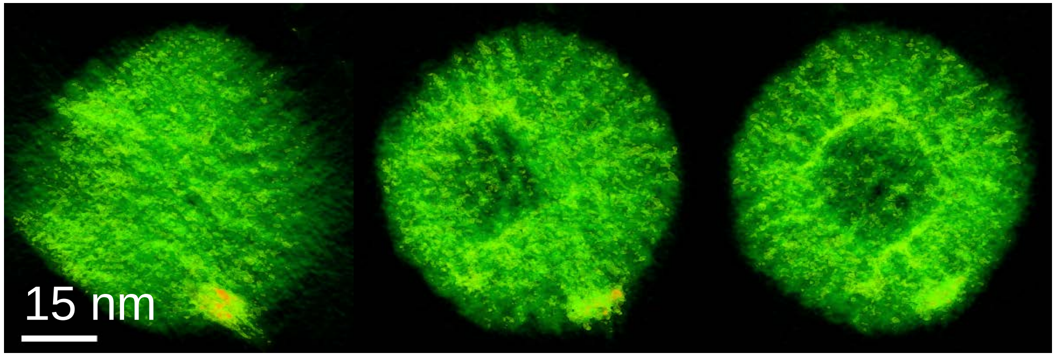
Example: mesoporous silica coating on gold nanorods



Tomographic reconstruction allows visualization of pore size and orientation for applications such as drug delivery.



Chemical mapping at the nanoscale: electron energy loss spectral image (EELS-SI) of a mesoporous silica coating on gold nanorods. Carbon is confined in the pores in mesoporous silica.

-Carbon -Silica

Images acquired by Blanka Janicek (Professor P. Huang Group)

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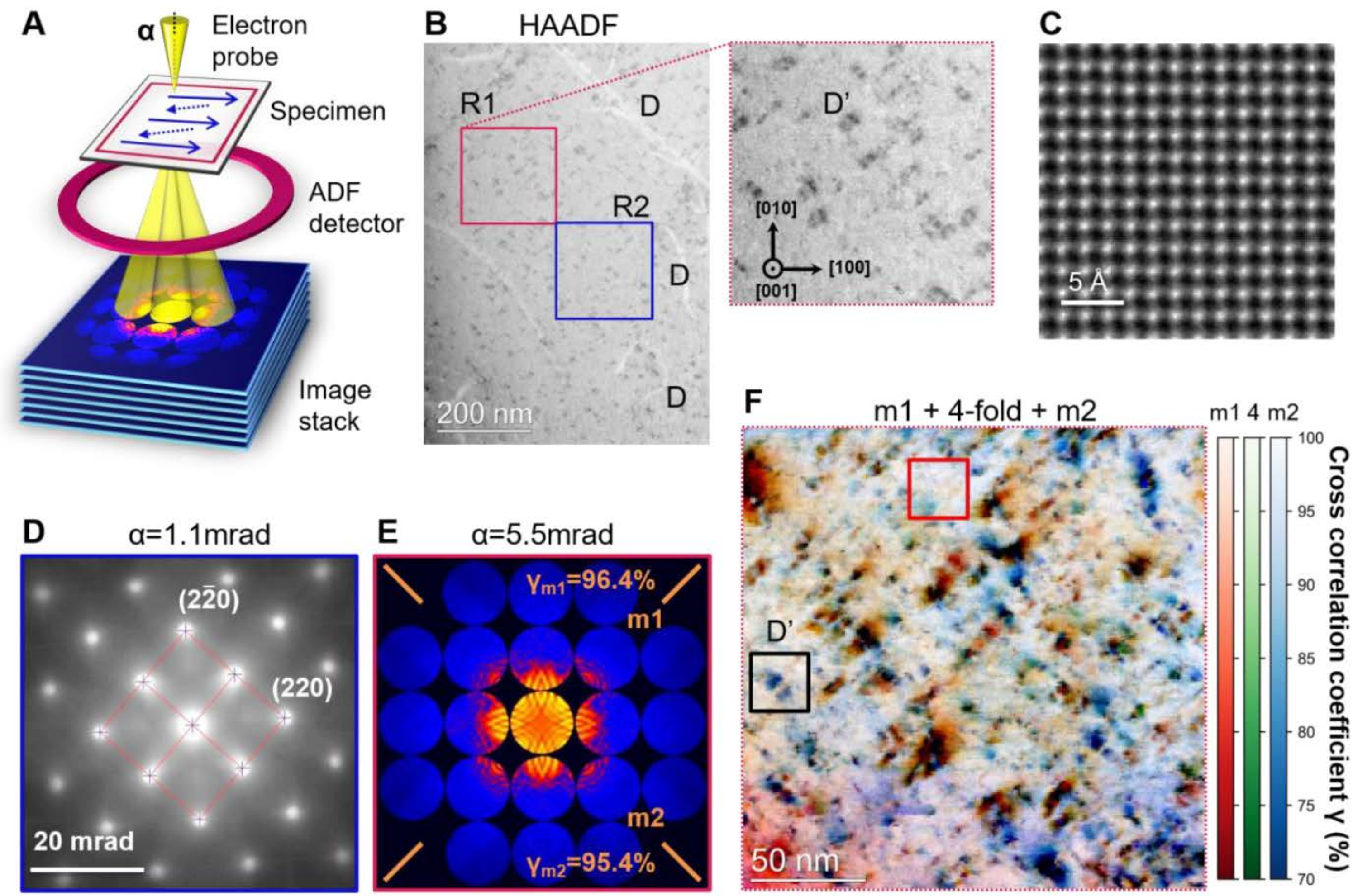
Example: Scanning Electron Diffraction- Nano-Diffraction (SEND) and CBED (SCBED)

Study of lattice distortions in an fcc High Entropy Alloy, Al0.1CrFeCoNi



Yu-Tsun Shao, Renliang Yuan, Yang Hu, Qun Yang, Jian-Min Zuo. (2019). The Paracrystalline Nature of Lattice Distortion in a High Entropy Alloy

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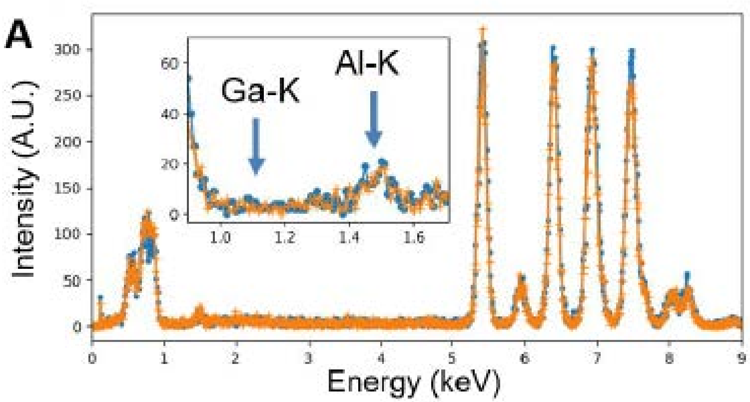
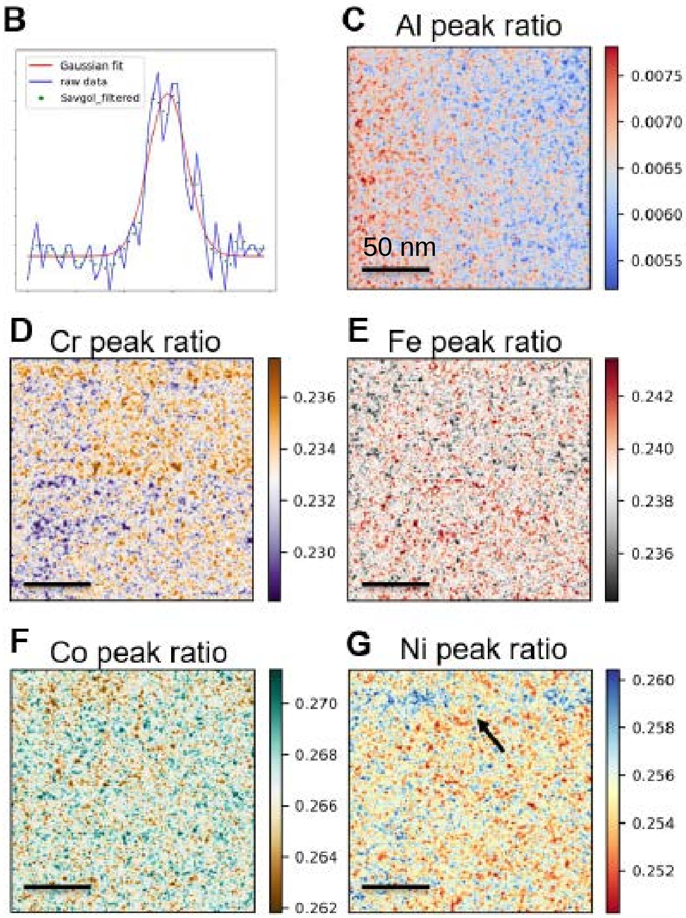
Example: Scanning Electron Diffraction- Nano-Diffraction (SEND) and CBED (SCBED)

EDS elemental X-ray analysis of Al0.1CrFeCoNi HEA

EDS spectrum image 180x180 pixels with step sizes of 1nm and dwell of 1s acquired about the same region as for SCBED. Total acquisition time of ~11hrs for detection of Al rich inclusions and fluctuation of composition at length scales of a few to tens of nm





Yu-Tsun Shao, Renliang Yuan, Yang Hu, Qun Yang, Jian-Min Zuo. (2019). The Paracrystalline Nature of Lattice Distortion in a High Entropy Alloy

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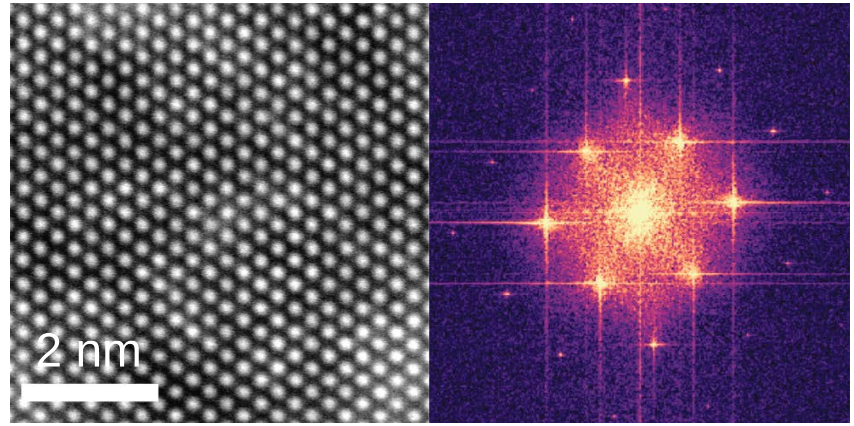
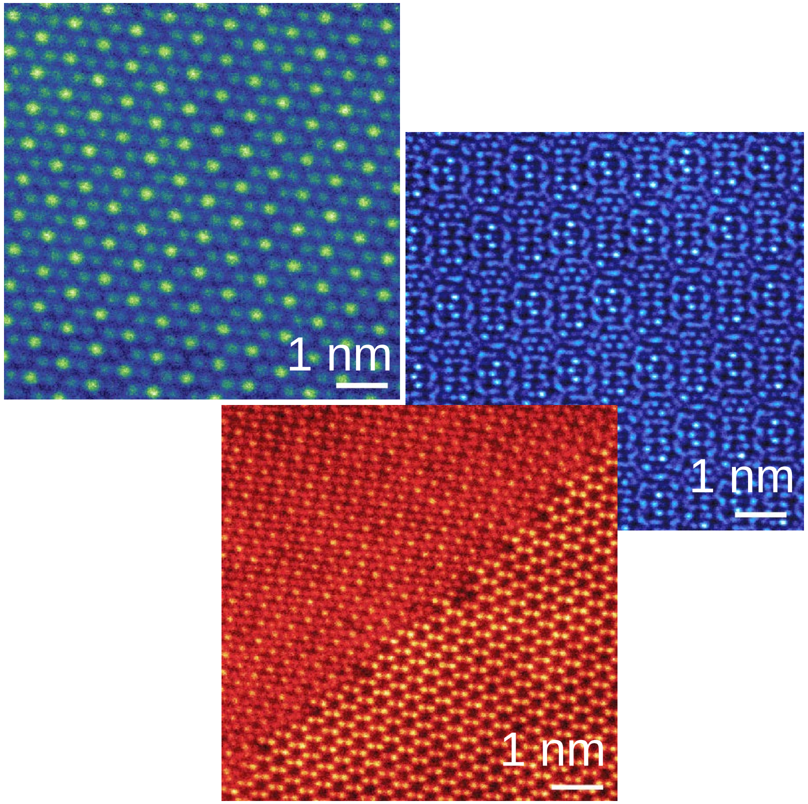
Examples: Tungsten Diselenide and Boron Arsenide



STEM image and FFT of cubic boron arsenide.
This material represents the experimental realization of new class of high thermal conductivity materials.
S. Li et al. High thermal conductivity in cubic boron arsenide crystals, Science 8982 (2018).

Image acquired by Yinchuan Lv (Professor P. Huang Group)



False-colored atomic-resolution scanning transmission electron microscope images of WSe_2
A) Two atomic layers of WSe2. The two layers are rotationally aligned with one another but shifted, forming the 3R structure

B) Moiré pattern in twisted bilayer WSe2 where the two layers are rotated by an angle of 17 degrees

C) A grain boundary of in bilayer WSe2 results in a change in stacking orders across the diagonal

Images acquired by Chia-hao Lee (Professor P. Huang Group)

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ThermoFisher Scientific Themis Z STEM/TEM:

For more information start with the following resources:

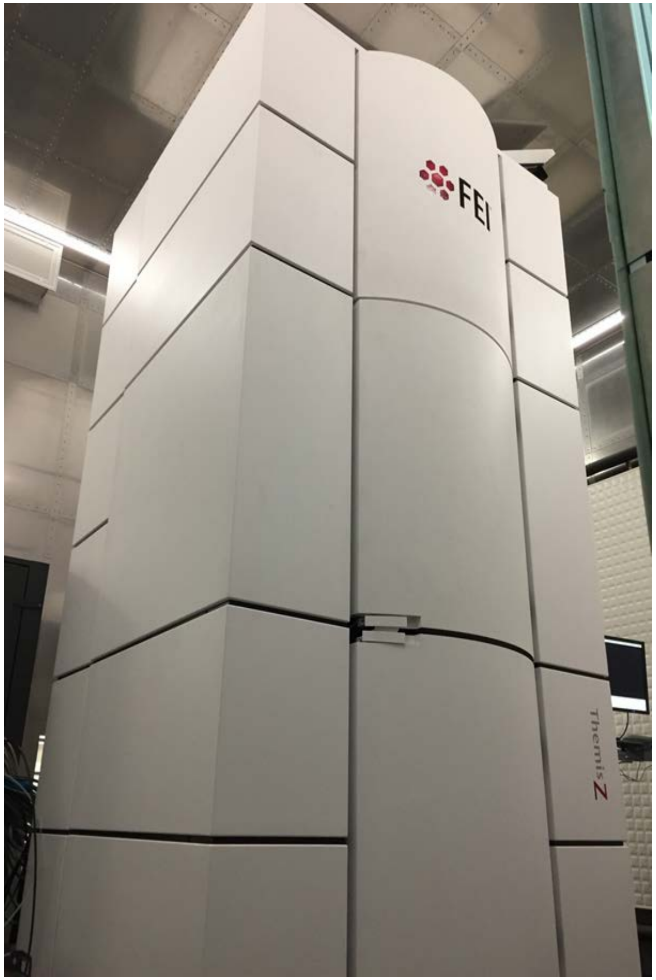
http://mrl.illinois.edu/facilities/equipment/fei-themis-z-advanced-probe-aberration-corrected-analytical-temstem

https://www.fei.com/products/tem/themis-z-for-materials-science/

Or

See MRL Facility Staff Members:

Dr. Jim Mabon or Dr. C.Q. Chen



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The End!

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Questions?