Transmission Electron Microscopy

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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
Why Use Transmission Electron Microscopy?

**Optical Microscope**

- AN is 0.95 with air up to 1.5 with oil
- Resolution limit: ~200 nm

**Transmission Electron Microscope (TEM)**

- Resolution
  \[ d = \frac{\lambda}{2A_N} \]
  - AN is 0.95 with air up to 1.5 with oil
- Resolution limit: ~0.15 nm (uncorrected)
  \[ \lambda = \frac{h}{(2mE_k)^{1/2}} \sim (1.5 / U)^{1/2} \text{ [nm]} \]
  - 200 keV electrons: \( \lambda = 0.0027 \text{ nm} \)

**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

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

Gun and Illumination part

Gun: LaB$_6$, FEG 80 – 300 keV

TEM Sample

Objective lens part

View screen

Mode selection and Magnification part

Electron source

Anode

Aperture

Condenser

Specimen

Objective

EDS

ED

Images

Final image

Transmitted signal image
How does a TEM Obtain Image and Diffraction?

Incident Electrons

Sample

Objective Lens

Back Focal Plane

First Image Plane

Conjugate planes

Object

Image

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

Mag. = \frac{v}{u}

Structural info

Morphology

View Screen

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Electron Diffraction I

Bragg’s Law

\[ 2d \sin \theta = n\lambda \]

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

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)
Electron Diffraction II

Diffraction patterns from single grain or multiple grains

Single crystal

Polycrystal

Amorphous

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Selected Area Electron Diffraction (SAED)

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

Example of SAED
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.

**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
Nanoarea Electron Diffraction (NAED)

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 \( \rightarrow \) Need to switch between NAED and TEM modes

**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 \( \rightarrow \) A 10 \( \mu \)m aperture can form a \( \sim \) 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 \( \rightarrow \) Need to switch between NAED and TEM modes

A. B. Shah, S. Sivapalan, and C. Murphy
Nanoarea Electron Diffraction


5 µm condenser aperture → 30 nm

This technique was developed by CMM
Aperture-Beam Nanoarea Electron Diffraction

20 µm condenser aperture \rightarrow 20 \text{ nm} probe size

This technique was developed by CMM
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

Carbon Nanotube

5-fold symmetry

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

Helical graphene sheet

More advanced electron diffraction techniques
Convergent Beam Electron Diffraction (CBED)

- **Parallel beam**
- **Convergent-beam**

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

SAED | CBED | Bright-disk | Dark-disk | Whole-pattern

Large-angle bright-field CBED
Major Imaging Techniques

Major Imaging Contrast Mechanisms:
1. Mass-thickness contrast
2. Diffraction contrast
3. Phase contrast
4. Z-contrast (S-TEM)

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)
TEM Imaging Techniques

I. Diffraction Contrast Image:
Contrast related to crystal orientation

Phase Contrast Image

Application:
Morphology, defects, grain boundary, strain field, precipitates
II. Diffraction Contrast Image: Bright-field & Dark-field Imaging

Two-beam condition

Bright-field Image

Dark-field Image
TEM Imaging Techniques

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 $\Theta_B$.

Dislocations & Stacking Faults


Near dislocations, electrons are strongly diffracted outside the objective aperture.

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

Dislocations can be imaged as 1.5 nm narrow lines

Bright-field

Weak-beam

Taken by I. Petrov

Experimental weak-beam

0.1 µm

150 nm

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TEM Imaging Techniques

II. Diffraction Contrast Image

Two-beam condition for defects

Dislocations
Use $g \cdot b = 0$ to determine Burgers vector $b$

Stacking faults
Phase $= 2\pi g \cdot R$
Each stacking fault changes phase $\frac{2}{3}\pi$

Diffraction contrast images of typical defects

Dislocations
Dislocation loop
Stacking faults

Howie-Whelan equation

\[
\frac{d\phi_0}{dz} = \frac{\pi i}{\xi_g} \phi_g \exp\{2\pi i (sz + g \cdot R)\}
\]
Lattice Beam Imaging

Two-beam condition

Many-beam condition

M. Marshall

C.H. Lei
Lattice Imaging

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

From a LaB$_6$ Gun

S-Field-Emission Gun

Lattice image of film on substrates

YBa$_2$Cu$_3$O$_7$

BaZrO$_3$

YSZ
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

\[ \Delta f_{sch} = -1.2 (C_s \lambda)^2 \]

Resolution limit

\[ r_{sch} = 0.66 C_s^{\frac{1}{4}} \lambda^{\frac{3}{4}} \]

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

J.G. Wen

Contrast transfer function
Scanning Transmission Electron Microscopy

**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.

Primary e-beam 0.5-30 keV
backscattered electrons
secondary electrons <50 eV
Auger electrons
x-rays

STEM
Primary e-beam 60-300 keV
x-rays
Probe size 0.1 - 0.5 nm
Thickness <100 nm
“Incoherent” Scattering i.e. Rutherford

Dark-field
Bright-field
“Coherent” Scattering (i.e. Interference)

magnetic prism
Inelastically scattered electrons

1 µm
TEM vs ADF-STEM

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

Ge quantum dots on Si substrate

Ir nanoparticles

STEM imaging gives better contrast

STEM images show Z-contrast

$\theta$

$I \propto Z^2$

10nm

5 nm

Z-contrast imaging

Annular Dark-Field (ADF) detector

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Superlattice of LaMnO$_3$-SrMnO$_3$-SrTiO$_3$  

Dopant atoms in Si

A. B. Shah et al., 2008  
P. M. Voyles et al., 2002
Bright Field STEM vs ADF STEM

Si – Ge Superlattice

40kX 1Mx 15Mx

Ge_xSi_{1-x}  Si

0.5 μm 20 nm 1 nm

40kX 1Mx 15Mx

Ge_xSi_{1-x}  Si

0.5 μm 20 nm 1 nm
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

\[ C_s + (-C_s) = 0 \]
Spherical Aberration Corrector for TEM & STEM

Harald Rose
Univ. of Tech. Darmstadt

Max Haider
CEOS, Germany

Uli Dahmen
NCEM

Hexapole $C_s$ Corrector

Optical Axis

Hexapole 1

Round Lens 1

$C_s < 0$

Round Lens 2

Hexapole 2

e-Beam
The STEM can obtain 1 Å spatial resolution only if the instabilities of the room are controlled.
Spherical Aberration Correction

JEOL 2010F
Cs = 1 mm
Probe size 0.3 nm

JEOL 2200FS
with probe corrector
Probe size 0.1 nm

\[ r_{sch} = 0.66 \frac{1}{C_s^4} \frac{3}{\lambda^4} \]
Spherical Aberration Corrector for STEM

Ondrej Krivanek
Nion Company

Quadrupole-Octupole $C_3 - C_5$ corrector

First sub-Å image resolved in a STEM

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

Annular dark field STEM image of hexagonal GaN [211]

Fourier transform of the image; image Fourier components extend to below 50 pm.
New Applications Possible Only with Aberration Correction

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
Analytical (Scanning) Transmission Electron Microscopy

Incident High-kV Beam

Secondary signals
- Back scattered electrons (BSE)
- Secondary electrons (SE)
- Auger Electrons

Primary signals
- Direct Beam
- Inelastically scattered electrons
- Elastically scattered electrons

Specimen
- Electron-Hole pairs (for EBIC)
- "Absorbed" Electrons

X-ray Energy Dispersive Spectroscopy
- Characteristic X-rays
- Visible light (Cathodoluminescence)
- Bremsstrahlung X-rays

Electron Energy Loss Spectroscopy
The emission of Auger electrons is an alternative to X-ray emission as an ionized atom returns to its ground state.

Characteristic Signals: EELS, EDS, XPS Signals
Experimental XEDS, EELS, & XPS

Copper L shell

NiO: O K-shell and Ni L shell

Energy resolution, Spatial resolution, Elements resolving

EDS
XPS
EELS

EDS
Ni L3
EELS
L2

Energy (eV)

700 800 900 1000 1100

Energy (eV)

400 600 800 1000

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EELS: Electron Optical System

Post-Column Filter (GIF)

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

Compare: Hemispherical Electron Energy Analyzer (Surface Analysis)

\[ V_0 = \frac{(V_1R_1 + V_2R_2)}{2R_0} \]

Williams and Carter 2009

Casaxps.com
EELS: Electron Optical System

In-Column (Omega) Filter

- 4 Dispersive elements
- Integrated into column
Conduction/Valence bands

Low Loss

Zero Loss

Plasmon

Experimental EELS: NiO

O Edge

Ni Edge

X100, boosted vertically

Energy-loss [eV]

532 ev

855 ev

O_k

Ni_L_{2,3}

Core shell energy levels
Sub Angstrom in Cs-corrected STEM!

Spectral Imaging:

- 3D/4D data cube
- x-y image
- z spectrum (EDS/EELS)
STEM-EELS Spectrum Imaging: Line Scan

C. Chen, J. Mabon, 2200FS
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**

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*Gatan Review of EFTEM Fundamentals*
EF-TEM Imaging: Using Core Loss

Three-window method

Jump ratio

Three Window Mapping

Pre-edge image

Post-edge image

Post-edge 1

Post-edge 2

Post-edge

Fast and Easy!

C. Chen, on 2010F, MRL
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
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)**
  
  \[ \frac{N_A}{N_B} = \frac{I_A^K (\beta \Delta)}{I_B^K (\beta \Delta)} \cdot \frac{\sigma_B^K (\beta \Delta)}{\sigma_A^K (\beta \Delta)} \]

- **Absolute quantification (atoms / nm²)**
  
  \[ N = \frac{I_K (\beta \Delta)}{I_1 (\beta \Delta) \sigma_K (\beta \Delta)} \]

  \( I_K \): sum of counts in edge k,
  
  \( I_1 \): total intensity (including zero loss),
  
  \( \sigma_K \): partial cross-section for ionization.
  
  \( \Delta \): integration window, \( \beta \): collection angle

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*Williams and Carter 2009*
White lines in transition metals

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

Williams and Carter 2009
**Log-ratio method**

\[ t = \lambda_p \times \ln\left(\frac{I_o}{I_T}\right) \]

- \( t \) = thickness
- \( \lambda_p \) = Plasmon MFP,
- \( I_o \) = zero-loss peak
- \( I_T \) = total spectrum

**Relative thickness:**
- Reliable,
- Fast and online,
- High spatial resolution

**Thin specimen**

\[ E_p = \frac{h}{2\pi} \omega_p = \frac{h}{2\pi} \left( \frac{n e^2}{\varepsilon_0 m} \right)^{\frac{1}{2}} \]

**Thick specimen**

\[ 2E_p \]
Quantitative Composition Analysis: Combining EDS-EELS

Combine STEM-EDS & STEM-EELS

![Graph showing the relationship between Na/Te(Pb$_x$S$_{1-x}$) atomic ratio and relative thickness ($\lambda$). The graph displays a downward trend as $\lambda$ increases.]

C. Chen, 2013
## Comparison of EELS and EDS

<table>
<thead>
<tr>
<th>EELS (primary)</th>
<th>EDS (Secondary)</th>
</tr>
</thead>
</table>
| • Atomic composition  
  • Chemical bonding  
  • Electronic properties  
  • Surface properties  
  .... | • Atomic composition only |
| • Energy resolution: ~ 0.15-1 eV | • Energy resolution: ~ 130 eV (Mn K$_\alpha$) |
| • 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 |
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)

Several Relaxation Events (~EDS/AES)

\[ E_{\text{loss}} > E_c > E_{\text{EDS}} \]

Table 4.2: Difference Between \( E_c \) and \( E_K \)

<table>
<thead>
<tr>
<th>Element</th>
<th>Critical ionization energy ( E_c ) (keV)</th>
<th>X-ray energy ( E_K ) (keV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>0.282</td>
<td>0.277</td>
</tr>
<tr>
<td>Al</td>
<td>1.562</td>
<td>1.487</td>
</tr>
<tr>
<td>Ca</td>
<td>4.034</td>
<td>3.692</td>
</tr>
<tr>
<td>Cu</td>
<td>8.993</td>
<td>8.048</td>
</tr>
<tr>
<td>Ag</td>
<td>25.531</td>
<td>22.163</td>
</tr>
</tbody>
</table>

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

Williams and Carter 2009
**Spatial Resolution (R) and Sensitivity (MMF, minimum mass fraction)**

**Bulk**
- MMF: 0.01%
- 3x10^6 atoms

**Thin**
- MMF: 0.01%
- 300 atoms

**Ultra-thin**
- MMF: 0.01%
- 1 atom

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**Beam diameter & spreading**

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

- E₀ 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%**
**• Cs correction improves both**
**• Achieving atomic resolution**
**• Approaching atomic level detection**

*Williams and Carter 2009*
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)

4. **JEOL JEM 2010 F Analytical (S)TEM**
   - EDS, STEM, Z-contrast imaging

5. **JEOL JEM 2200 FS Cs 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**
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
ThermoFisher Scientific Themis Z STEM/TEM:

- X-FEG Thermal Field Emission Gun
  - High Brightness (>7x 10^7 A/m² 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 4th order aberrations (5th 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
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
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/
High throughput spectrum imaging: Microprocessor X-TEM EDS

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

J. Mabon, C. Chen - Themis Z, MRL
EDS Spectrum Imaging and Analysis:

AlGaAs – GaAs multilayer

DCFI HAADF images (~20 Frames)

EDS 4 minute acquisition time, 50 pA, 300kV

J. Mabon -Themis Z, MRL
Fast EDS spectrum imaging @ high resolution: LMO/SMO

300KV, 140 pA, ~5 minutes

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Atomic resolution EDS Spectrum Imaging and Mapping

Strontium Titanium Oxide (SrTiO₃)

12 minute acquisition time, 100pA, 300kV

J. Mabon - Themis Z, MRL
Atomic resolution EDS spectrum imaging - GaAs

HAADF

Raw

Filtered (Radial Wiener de-blur )

Overlay of filtered maps on HAADF

EDS 1.5 minute acquisition time, 50 pA, 300kV

J. Mabon - Themis Z, MRL
Atomic resolution EELS mapping and analysis (with monochromator):

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

BaTiO3/SrTiO3 interface

DCFI HAADF

Also possible:

- Lanthanum Manganese Oxide/Strontium Manganese Oxide Multilayer


EELS Spectra J. Mabon

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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.

Influence of a nanotwinned, nanocrystalline microstructure on aging of a Ni-25Mo-8Cr superalloy
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)
Study of lattice distortions in an fcc High Entropy Alloy, Al0.1CrFeCoNi

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

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 WSe₂

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)
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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Questions?