Transmission Electron Microscopy

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Outline

1. Introduction to TEM
2. Basic Concepts
3. Basic TEM techniques
   Diffraction contrast imaging
   High-resolution TEM imaging
   Diffraction
4. Scanning-TEM techniques
5. Spectroscopy
   X-ray Energy Dispersive Spectroscopy
   Electron Energy-Loss Spectroscopy
6. Advanced TEM techniques
   Spectrum imaging
   Energy-filtered TEM
7. Other TEM techniques
   Low-dose; in-situ; etc.
8. Summary
### Why Use Transmission Electron Microscope?

**Resolution limit:** 200 nm

\[ d = \frac{\lambda}{2A_s} \]

- **Air:** \( A_s = 0.95 \) nm
- **Oil:** \( A_s = 1.5 \) nm

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

**Resolution record by TEM**
- 0.63 Å

**TEM Sample**
- Thin foil, thin edge, or nanoparticles

### Basic Structure of a TEM

- **Gun:** LaB₆, FEG 100, 200, 300 kV
- **Illumination part**
- **TEM Sample**
- **Objective lens part**
- **View screen**
- **Mode selection and Magnification part**
- **Gun and Illumination part**
- **Electron source**
- **High voltage**
- **Anode**
- **Aperture**
- **Condenser**
- **Double lens**
- **Detection of secondary and reflected radiation**
- **Specimen**
- **Objective**
- **EDS**
- **ED**
- **Aperture**
- **Electron lens**
- **Images**
- **Final image**
- **Transmitted signal image**
How does a TEM get Image and Diffraction?

Incident Electrons
Sample
Objective Lens
Back Focal Plane
First Image Plane
Object Image
View Screen
u    v
1 1
u' v'
1
f
Conjugated planes

Basic Concepts
High energy electron – sample interaction

1. Transmitted electron (beam)
2. Diffracted electrons (beams)
3. Coherent beams
4. Incoherent beams
5. Elastically scattered electrons
6. Inelastically scattered electrons
Electron Diffraction I

Bragg’s Law
\[ 2d \sin \theta = n\lambda \]

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

Zero-order Laue Zone (ZOLZ)
First-order Laue Zone (FOLZ)
High-order Laue Zone (HOLZ)

Electron Diffraction II

Diffraction patterns from single grain and multiple grains.

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

Polycrystal
Amorphous
Major Imaging Techniques

1) Imaging techniques in TEM mode
   a) Bright-Field TEM (Diff. contrast)
   b) Dark-Field TEM (Diff. contrast)
   Weak-beam imaging
   hollow-cone dark-field imaging
   a) Lattice image (Phase)
   b) High-resolution Electron Microscopy (Phase)
   Simulation and interpretation
2) Imaging techniques in scanning transmission electron microscopic (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)

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

TEM Imaging Techniques

I. Diffraction Contrast Image:
   Contrast related to crystal orientation
   Many-beam condition
   Two-beam condition

Application:
Morphology, defects, grain boundary, strain field, precipitates
TEM Imaging Techniques

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

- Two-beam condition
- Bright-field Image
- Dark-field Image

III. Thickness fringes and bending contour

- Howie-Whelan equation
- Extinction distance
- Excitation error
- Thickness fringes
- Bending contour
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 \( = 2 \pi \cdot g \cdot R \)

Each staking fault changes phase \( \frac{2\pi}{3} \)

Howie-Whelan equation

\[
\frac{d\phi_g}{dz} = \frac{\pi i}{\xi g} \exp \left\{ 2\pi i (sz + g \cdot R) \right\}
\]

Diffraction contrast images of typical defects

Dislocations
Dislocation loop
Stacking faults

Weak-beam Dark-field imaging

High-resolution dark-field imaging

Exact Bragg condition

Weak-beam means
Large excitation error

Planes do not satisfy Bragg diffraction

Possible planes satisfy Bragg diffraction

Experimental weak-beam

Bright-field
Weak-beam

Dislocations can be imaged as 1.5 nm narrow lines

Taken by I. Petrov
Lattice imaging

Two-beam condition

Many-beam condition

Delocalization effect from a field-emission gun (FEG)

From a LaB\textsubscript{6} Gun

Field-Emission Gun

Lattice image of film on substrates
**High-resolution Electron Microscopy (HREM)**

Weak-phase-object approximation (WPOA)

\[ f(x,y) = \exp(i \sigma V_t(x,y)) \sim 1 + i \sigma V_t(x,y) \]

\[ V_t(x,y) : \text{projected potential} \]

Indirect imaging

Scherzer defocus

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

Resolution limit

\[ r_{sch} = 0.66 \left( \frac{C_s}{\lambda} \right)^{\frac{3}{4}} \]

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

**Selected-area electron diffraction (SAD)**

Major Diffraction Techniques

1) Selected-area Diffraction
2) Nanobeam Diffraction
3) Convergent-beam electron diffraction

Example of SAD and dark-field imaging

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Electron Nanodiffraction

5 μm condenser aperture → 30 nm


This technique was developed by CMM

Convergent-beam electron diffraction (CBED)

Parallel beam Convergent-beam

Sample Sample

Back Focal Plane

Large-angle bright-field CBED

SAD CBED Bright-disk Dark-disk Whole-pattern

1. Point and space group
2. Lattice parameter (3-D) strain field
3. Thickness
4. Defects
5. Chemical bonding
Convergent-beam electron diffraction
Quantitative Analysis of Local Strain Relaxation


Use High-order Laue zone (HOLZ) lines to measure strain field.

SEM vs STEM

Scanning electron microscopy (SEM)
- 1st primary e-beam: 0.5-30 keV
- Secondary electrons: <50 eV
- Backscattered electrons
- Auger electrons

Scanning transmission electron microscopy (STEM)
- Primary e-beam: 100-300 keV
- Characteristic & Bremsstrahlung x-rays

Probes:
- SEM: Thickness <100 nm
- STEM: Probe size 0.18 nm

Detectors:
- Bright-field
- Dark-field Detector

"Coherent" Scattering (i.e., interference) vs "Incoherent" Scattering (i.e., Rutherford)
**TEM vs STEM**

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

**HRTEM vs STEM**

1. **Contrast**
   - High-resolution TEM (HRTEM) image is a phase contrast image (indirect image). The contrast depends on defocus.
   - STEM image is a direct atomic column image (average Z-contrast in the column).

2. **Delocalization Effect**
   - High-resolution TEM image from FEG has delocalization effect.
   - STEM image has no such an effect.

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*From Pennycook’s group*
X-ray Energy-Dispersive Spectroscopy (EDS)

1) TEM mode → spot, area
2) STEM mode → spot, line-scan and 2-D mapping

Spatial resolution ~ 1 nm

Line scan

Ti$_{0.85}$Nb$_{0.15}$ metal ion etch
Creates a mixed amorphised surface layer ~ 6 nm

Electron Energy-loss Spectroscopy (EELS)

EELS spectrum:
1. Zero-loss Peak (ZLP)
2. Low-loss spectrum (<50eV)
   Interacted with weakly bound outer-shell electrons
   Plasmon peaks
   Inter- & Intra-Band transition

Application:
   Thickness measurement
   Elemental mapping
Edge Peaks in EELS

3. High-loss spectrum
   Interacted with tightly bound inner-shell electrons
   Edge peaks
   Application:
   Elements identification
   Chemistry

Edge peak shape

Edge peak position

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Spectrum imaging

TEM mode
- Energy filtered TEM
- 1. ZLP imaging
- 2. Plasmon imaging
- 3. Edge imaging

STEM mode
- 1. Plasmon imaging
- 2. Edge imaging

Energy-filtered TEM
- Fill in data cube by taking one image at each energy

STEM mode
- Fill in data cube by taking one spectrum at each location
Energy-Filtered TEM (EFTEM)

EFTEM - Zero-Loss Peak imaging

Only elastic electrons contribute to image – remove the “inelastic fog”
1. Improve contrast (especially good for medium thick samples)
2. Z<12, the inelastic cross-section is larger than elastic cross-section

EFTEM – Plasmon Peak imaging

Spectrum image (20 images)  Al mapping image  W mapping image

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EFTEM – Edge Peak imaging

Three-window method

Jump ratio

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STEM + EELS Spectroscopy

convergence angle ~ 15 mrad

scan coils

incident probe size ~ 0.2 nm

specimen

HAADF detector

magnetic prism

LaMnO₃

SrTiO₃

LaMnO₃

SrTiO₃

Z-contrast image

EELS spectrum

Ti

O

Mn

La

Z-contrast image
**STEM + EELS Spectroscopy**

Z-contrast image shows where columns of atoms are and EELS spectrum identify chemical components.

Electronic structure changes are observed in the fine structure of O K-edge.

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**New TEM: Cs-corrected Analytical STEM/TEM**

With this setup we can achieve a probe-size of <0.1nm.
Small probe size for high-resolution scanning transmission electron microscopic images

Si [110] Zone Axis

2 x 2 LaMnO$_3$-SrTiO$_3$ superlattice

Dec. 2006
Thick specimen

June 2007
Same specimen

Dec. 2007
Thin Specimen

JEOL 2010F, Cs = 1 mm (2nd smallest Probe)

JEOL 2200FS with probe forming Cs Corrector
Polarized neutron scattering shows interfacial ferromagnetic moment measured at 10 Kelvin is enhanced in LaMnO$_3$ at the sharp LaMnO$_3$-SrMnO$_3$ interface and reduced at the rough SrMnO$_3$-LaMnO$_3$ interface. 

Epitaxial Oxide Films Grown on SrTiO$_3$

STEM Image of Superlattice

Polarization of CdSe

CdSe nanoparticles

View along [110] zone axis

Polarity of CdSe
Hetero-structure nanoparticle

Quantitative STEM Imaging
Monometallic Pt

The intensity at each atomic column is proportional to numbers of atoms

No defects in Pt nanoparticles

Monometallic Pd

Pd nanoparticles contain many defects such as twin boundaries
Study Nanoparticles by TEM

Size distribution: STEM will give better contrast

> 5 nm: Both TEM & STEM better contrast

2 nm < Size < 5 nm: Ultra thin carbon grid, STEM better contrast

< 2 nm: Ultra thin carbon grid, Only STEM

Composition study: EDS counts are low

2200 FS EDS system
detector area 2 times bigger
beam 4 times brighter

Minimum-Dose for beam sensitive samples

Minimum-dose in TEM mode
Search in low magnification
Focus at another area
Photo with minimum dose

Minimum-dose in STEM mode
HAADF-STEM image
Long exposure time
BF-STEM image
Short exposure time

Z-contrast tilt-series
MDS + tomography
will be available on
2100 Cryo-TEM soon
+/- 80 degree tilt

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In-situ capabilities

1. Heating (hot stage 1000°C)
2. Cooling (liquid N₂)
3. Tensile-stage
4. MEMS tensile stage
5. Universal MEMS holder
6. Wet-cell
7. Nanomanipulator
8. Environmental holder
9. Applied voltage to sample
10. Cryo transfer holder

In-situ holders

- Universal MEMS holder
- MEMS straining stage
- Nanomanipulation
- Liquid cell

List of TEMs and functions

1. CM12 (120 KeV) (S)TEM
   - TEM, BF, DF, CBED (good), EDS, large tilt angle, etc
2. 2010 LaB6 TEM
   - TEM, low dose, NBD, good for HREM, video function
3. 2100 LaB6 (Cryo-TEM)
   - TEM, Low dose, special cryoshielding; high-tilt angle (+/-80) (using special retainer).
4. 2010F (S)TEM
   - TEM, BF, DF, NBD, CBED, EDS, STEM, EELS, EFTEM, Spectrum imaging, etc.
5. HB501 STEM
   - STEM, BF, DF, EDS, EELS (cold FEG), ultra-high vacuum
6. JEOL 2200FS (S)TEM
   - Cs-corrected probe, TEM, BF, DF, NBD, CBED, EDS, STEM, EELS, EFTEM, Spectrum imaging, etc.