MSE 582 - Introduction to transmission electron microscopy
&
MSE 640 - Transmission electron microscopy & crystalline imperfections

Instructor: Eric Stach
Goals

• Goals:
  – MSE 582: Perform simple imaging and diffraction experiments with the microscope
  – MSE 640:
    • In depth understanding of fundamentals of scattering, diffraction and imaging
    • Practical application of these concepts towards materials characterization

• Taught in four units:
  – Operation of the TEM (MSE 582 content)
  – Diffraction
  – Imaging
  – Spectroscopy
Course mechanics

MSE 582: Pass/Fail
- Can you take a picture with the microscope?

MSE 640:
- Homework (20%)
- Laboratories (30%)
  - We’ll engage in a systematic study of the microstructure of an advanced aluminum alloy
- Exams (50%)
  - Midterm (after diffraction unit - 20%) and a Final (30%)
About me ...

Education
- B.S.E. - Duke University
- M.S.M.S.E. - University of Washington
- Ph.D. - University of Virginia

Professional Experience
- Graduate research - IBM Watson Research Labs
- Staff Scientist, Principal Investigator and Program Leader, National Center for Electron Microscopy & Materials Sciences Division, Lawrence Berkeley National Laboratory
- Associate Prof @ Purdue since January 2005

Research expertise
- Crystalline defects, crystal growth, electronic thin films, electron microscopy, mechanical behavior, nanostructured materials
What can you do with a TEM?
What can you do with a TEM?

Selected Area Diffraction (SAD)

- Crystallographic structure from particular areas of a sample.
- Used to distinguish and identify crystalline (and amorphous) phases in a material.

Selected area diffraction pattern
10\overline{1}0 Zone axis pattern of a hexagonal GaN / cubic GaN heterostructure

Pattern reveals extensive twinning in the cubic layer
What can you do with a TEM?

Convergent Beam Electron Diffraction (CBED)
- Point and Space Group determination
- Local strain
- Nanoscale diffraction

Convergent beam electron diffraction pattern
111 zone axis pattern of silicon
Note detailed structure in the central disk
What can you do with a TEM?

Large-angle Convergent Beam Electron Diffraction (LACBED)

- Misorientation across grain boundaries
- Dislocation Burgers vector
- Crystalline symmetry

From J.P Mornoroli
What can you do with a TEM?

Diffraction Contrast Imaging

Strain fields
- Dislocations
- Stacking faults
- Grain boundaries
- Precipitates
- Second phases

Typical bright field image
Dislocation configurations at the interface between a SiGe heteroepitaxial layer and a Si (100) substrate viewed in plan view (along [100])
What can you do with a TEM?

**Diffraction Contrast Imaging**

One beam selected for imaging
- Transmitted - "bright field"
- Diffracted - "dark field"
What can you do with a TEM?

**Diffraction Contrast Imaging**

**Strain fields**
- Dislocations
- Stacking faults
- Grain boundaries
- Precipitates
- Second phases

**Typical bright field image**

Dislocation configurations at the interface between a SiGe heteroepitaxial layer and a Si (100) substrate viewed in plan view (along [100])
What can you do with a TEM?
What can you do with a TEM?

‘Weak beam’ dark field image
What can you do with a TEM?

High-resolution imaging
Atomic column images at resolutions from 0.7Å and above
  - Interference of transmitted and diffracted electron waves

High resolution micrograph of a precipitate at a high angle grain boundary in aluminum

Courtesy of U. Dahmen
What can you do with a TEM?

High angle annular dark field (HAADF) imaging

- Accomplished in a dedicated Scanning TEM (STEM)
- Collects incoherent scatter, yields atomic resolution

Mixed dislocation in GaN

Dissociated mixed dislocation in GaN

Courtesy of I. Arslan
What can you do with a TEM?

Electron holography

Map the mean inner potential of a material

Courtesy of J. Cumings
What can you do with a TEM?

“Lorentz” microscopy for imaging of magnetic structures

Defocus = -15µm  In focus  Defocus = +15µm

Data courtesy G. Kuskiniski
What can you do with a TEM?

Energy Dispersive X-ray Spectroscopy (EDS)

- Detection of characteristic x-rays excited by incident electrons.
- Spatial resolution on the order of probe size (can be as low as 2-3 Å)

Simple EDS spectrum from Al$_2$O$_3$

From Williams & Carter
Electron energy loss spectroscopy

- Measures the amount of energy lost by the incident electrons.
- Similar spatial resolution, energy resolution of $\approx 1$ eV.
- Probes density of state (DOS) locally.

EELS spectrum from CaCO$_3$

From Williams & Carter
What can you do with a TEM?

Ti valence determination using EELS

Ti L\textsubscript{2,3} edge

- Ti\textsubscript{2}O\textsubscript{3} (green)
- Ti\textsuperscript{3+} (red)
- Ti\textsubscript{O} (red)
- Ti\textsuperscript{4+} (blue)

CaTiO\textsubscript{3} (black)

Energy Loss (eV)

Ti L\textsubscript{2,3} edge from trivalent Ti\textsubscript{2}O\textsubscript{3} differs markedly from tetravalent compounds TiO\textsubscript{2} and CaTiO\textsubscript{3}

TiO\textsubscript{2} (red) and Twist Boundary (blue)

Energy Loss (eV)

Ti L\textsubscript{2,3} edge from twist boundary closely matches edge structure of TiO\textsubscript{2} standard (Ti\textsuperscript{4+}).

Data courtesy Seth Taylor
What can you do with a TEM?

Energy Filtered Imaging

- Zero loss imaging removes inelastically scattered electrons from image
- Selective imaging of electrons that have lost a particular energy
- Most commonly used to create a map of local (≈ 1 nm) chemistry

Energy filtered image of a SiGe graded buffer structure using the Si K edge.
The brighter the pixel, the larger the concentration of Si.
What can you do with a TEM?

**Dynamical behavior**
- Possible to apply many types of stimuli to samples during simultaneous imaging.
- Probe interrelationships between structure / properties and processing
- Stimuli include:
  - Temperatures to 1300°C
  - Temperatures to LN₂
  - Chemical flux
  - Nanomanipulation
  - Nanoindentation
  - Electrical bias in combination with heating
  - Uniaxial strain

Interaction between threading and misfit dislocations in a SiGe heterostructure
A way to organize our thoughts

In MSE 582 we will learn how to “use” the microscope

We will discuss:

– Electrons & scattering, briefly
– Electron sources
– Lenses & aberrations
– How the instrument goes together
– Alignment
– The sample

From Reimer
In MSE 640, we will cover:

- Elastic & inelastic scattering
- Diffraction
  - Single scattering (kinematical)
  - Multiple scattering (dynamical)
- Image formation
  - Diffraction contrast
  - Phase contrast
  - Incoherent imaging
- Spectroscopy
  - Energy Dispersive X-ray Spectroscopy
  - Electron Energy Loss Spectroscopy
Basic properties of electrons

a reminder ...