Electron Microscopy and Analysis

A review of electron-optics and its applications in nanoscience
Microscopy in the middle ages

- The development of high quality glass and polishing techniques enabled the definition of lenses for telescopes and microscopy in the 1500s.
The first electron microscope: The TEM

- The resolution is determined by the wavelength of the electron along with lens aberrations.

Work done: 1931
Nobel prize: 1986
Resolution vs. wavelength

- Electron wavelength depends on energy: could be as small as several picometers
- Compares well to optical wavelengths in the 100s of nanometer range
Electrons in a vacuum: the trend to higher energies

- Electrons can be accelerated to higher velocities by using high voltages.
- The higher the energy, the shorter the wavelength.
- For many years, higher electron energy systems were used for improved resolution.
- Higher penetration depth (Bethe range) can also be obtained at higher electron energies.
Evolution of the electron microscope: resolution improved to 0.05nm
Modern TEMs

• Maximum magnification in excess of 1,000,000x
• Better than 0.1nm line-to-line resolution
• Highest resolution: 0.05nm
• Uses aberration correction optics to make up for lens and sample imperfections
The Scanning Electron/Ion Microscope

- The resolution is determined by the spot size of the electron or ion beam.
- An overall “system magnification” determines how the final spot size relates to the initial electron source size.
The dual-beam SEM/FIB

- Electron beam with 1nm resolution and ion beam with <4nm resolution can image simultaneously
- Metals can be deposited through injectors of organometallics
- Ions and electrons are scanned and milling of the sample can be observed in SEM mode
Sizes of things

- As we develop new technologies, our capabilities to observe smaller objects has evolved to the nanoscale
• Single-walled carbon nanotubes with 1.4nm can be imaged
The electron source

• The electron source uses an electrostatic lens, called the Wehnelt cylinder, to develop a high brightness beam of electrons

• The most common sources are: thermionic emission, LaB$_6$ and field emission sources
The electromagnetic lens

- A strong electromagnetic field is generated through coils
- This field is focused into a small cylinder by using a pole piece
- The electrons on the outside of the cylinder are deflected towards the center – resulting in a convex lens
Aberration-corrected images

- How to record TEM images:
  - Used to use film
  - Now we use CCD cameras
  - Also use STEM mode, by measuring transmission changes of a small beam swept over the sample surface
Imaging modes of TEM
Scanning Electron Microscope (SEM)

- An electron beam is focused at the sample surface and rastered.
- Electrons emitted from the surface are imaged.
- The ratio of the beam raster to the screen determines the magnification.
Imaging modes:

- Secondary electrons (< 5eV)
- Backscattered electrons (~50eV)
- Auger electrons (~500eV)
- Diffracted electrons
- Characteristic x-rays
- Current imaging (absorbed e-)
- Cathodoluminescence (light)
Electron interaction volumes

- The resolution of the SEM is ultimately determined by the source of the electrons (or other radiation) as the spot size is typically very small.
- Monte-Carlo simulations can provide an accurate model of where electrons come from.
Environmental SEM (ESEM)

- Many biological and insulating samples are difficult to image in the SEM
- Traditionally, these are metallized before imaging
- 20 years ago, a new imaging mode was developed using H2O ions as imaging species
- Special detectors needed to be developed for this imaging mode
Getting into the electron microscope

- Specimens are collected and glued to a sample stage.
- The samples are placed on the 5-axis xyz stage and inserted into the vacuum.
Focused ion beams: The liquid metal source (LMS)

- Metals can be heated in a crucible and wicked down to the end of a tungsten tip.
- The high field of at the end of the tip extracts ionized metal from that surface.
- These ions can be focused by electrostatic lenses and scanned over the sample.
- Ga is the most common metal used.
The FIB

- In a focused ion beam system, the ion beam is focused and rastered over the sample by using deflection.
- Ions have a higher mass than electrons – therefore higher forces are required to focus them.
Early FIB history

- Orloff and Swanson generated ion beams with liquid metal sources (main application: space propulsion)
- Ion beam systems were constructed using glass column supports
- Gallium, indium, and eutectic alloys were used for the first LMI sources
- Reactive gas was added for cleaner etch results in the 1980s
Focused ion beam examples

Figure 10. Folded cavity laser (FC-SEL) with FIB cut 45° total internal reflection mirror along with laser L-I curve (red) compared with VCSEL (blue curve).
More examples:
FIB Art
Field Ion Microscopy
Erwin W. Mueller (1951)
Inside of a FIM

- Gas atoms are ionized at the needle surface.
- These ions are accelerated towards the image intensifier.
- The system magnification is the ratio of the needle radius to the size of the imaging plate.

+20-50kV

Ne, Ar, He at 10^{-7} Torr

ZnO deposited onto the glass surface

Cold-trap

Image intensifier

Glass window
The Field Emission Microscope (FEM)

- Electrons are extracted from a needle
- These electrons are accelerated towards a plate
- The directionality of electron emission (relative work functions) can be measured
- It is very difficult to obtain high quality images of surface morphology or topography
Electron-beam induced radiation

An electron beam hitting a surface will generate:

• Secondary electrons
• Backscattered electrons
• Auger electrons
• Continuum x-rays
• Characteristic x-rays
• Cathodoluminescence
• Absorbed electrons (current)
Backscattered electrons vs. secondary electrons

• The difference between backscattered and secondary electrons is their energy. The vast majority of electrons coming from the sample surface are low-energy (secondary) electrons with 2-5eV energy. These electrons require a complex scintillator/ photomultiplier detector system called the Everhart-Thornley detector.

• Another maximum in energy is around the 50eV range, and electrons with such higher energies are called backscattered electrons. These come from deeper in the sample, and can be easily detected using a p-n diode (often called a Robinson Detector).
Related Analytical Techniques

- X-ray diffraction
- Electron diffraction
- Electron Energy loss spectroscopy (EELS)
- Characteristic x-rays
  - Energy Dispersive x-ray Analysis (EDX)
  - Wavelength-dispersive x-ray Analysis (WDX)
- Auger electrons
- Cathodoluminescence
- Secondary ion mass spectroscopy
- Current imaging and EBIC
X-ray or electron diffraction

• Bragg’s condition for diffraction: \( n\lambda = 2d\sin(\theta) \)

• The d-spacing is the distance between any two lattice planes with the same orientation
  – i.e. for a cubic crystal: \( d = a_0/\sqrt{h^2+k^2+l^2} \)
  – \( \Theta \) is the angle between the surface normal and the incident beam
  – Wavelength \( \lambda \) is determined by the characteristic x-ray emission or by the electron wavelength
  – Peaks in x-ray intensity will be measured when the Bragg condition is met – this enables the determination of the lattice constant \( a_0 \) and (hopefully) the identification of the material
  – Certain Bragg conditions are not permitted (extinction conditions)
In Electron energy loss spectroscopy, the electron energy is measured with a spectrometer. The energy loss is correlated with bonding energies (plasmon energies) of the material. High energy loss relates to excessive absorption in the material. Very thin samples and low surface contamination are required as otherwise the electrons may lose energies through other mechanisms.
Electron Channeling Patterns

• Certain directions in the lattice are open to electron channeling
• In these directions, large numbers of electrons are transmitted through the lattice
• Kikuchi lines result from such channeling directions
• These channeling directions can be determined through TEM or backscattered electrons
Energy-dispersive x-ray analysis (EDX)

- EDS, EDX or EDAX is a technique through which characteristic x-ray energies are measured.
- Characteristic x-rays come from inelastic electron collisions that generate x-rays with specific energies relating to the inter-shell energy loss.
- These characteristic x-rays are found on top of the continuum x-ray spectrum (Bremsstrahlen).
- X-ray energies can be measured by observing how many electrons are generated in a low-noise p-n diode (a Si-Li detector) by the individual x-rays.
- A histogram of number of x-rays vs. x-ray energy is developed and peaks can be observed.
- These peaks are typically characteristic of the x-ray energies of specific atoms and atomic concentrations can be calculated by looking at the relative intensities of the peaks.
- The detector needs to be LN2 cooled for low noise, which in turn requires a thin window – this limits the minimum energy of x-rays and hence the lowest Z materials that can be identified.
Wavelength-dispersive x-ray analysis
WDS

• Instead of measuring the x-ray energies by current pulses, WDS measures x-ray wavelengths by using gratings and crystals. The Bragg condition is again used to disperse x-rays in front of a x-ray detector.
• This method for measuring characteristic x-rays is more quantitative and does not have the low-Z limitations that EDX suffers from.
• Typically, crystals and gratings are tilted with respect to the x-rays generated from the sample and spectra are acquired.
• This method of analysis is often used in electron microprobes.
Auger Electron Spectroscopy (AES)

• Auger electrons have a very short mean-free path, and cannot escape from anywhere except for the very surface of the sample. This is why Auger electrons are often used for surface analysis.

• The problem is that the detection of the characteristic energies of these electrons requires careful spectroscopy and high vacuum cleanliness.

• Another problem is that the number of collected Auger electrons is often low (compared to characteristic x-rays) and requires higher beam currents.

• The AES is typically a high current electron beam that is scanned over a sample. Depth profiling can be done by using an ion beam for milling of the sample.
Cathodoluminescence

• The same method that is used in the CRT tubes (i.e. luminescence generated by an incident electron beam) can be used to characterize light-emitting semiconductors.
• In Cathodoluminescence, an electron beam (usually in an SEM) is used to excite electron-hole pairs in the semiconductor, and these recombine to provide light at specific wavelengths.
• This light is collected by using an elliptical mirror, where the sample is located at one of the foci and the collection optics at the other.
• The time-dependent dynamics of electron and hole diffusion and recombination can be studied by this technique. It is also possible to measure local changes in band structure by using the higher spatial resolution of electron-beam excitation.

An optical spectrometer is often used at the end of the waveguide outside of the vacuum system to characterize the luminescence spectrum.
Secondary ion mass spectroscopy

- When an ion beam hits the surface of a sample, secondary electrons as well as secondary ions are generated and leave the surface.
- These secondary ions hold very local information on the composition of the sample surface.
- SIMS is often used to determine surface composition within a scanning focused ion beam system.
- The key part of the SIMS system is the ion mass spectrometer, which could be either a time-of-flight or a quadrupole mass analyzer.
- SIMS has the additional ability to determine isotope concentrations.
Current and Voltage Imaging

- Instead of measuring the electrons emitted by the sample, we can also measure the electrons absorbed by the sample. This is often called “current imaging”. Although it does not provide us with very high resolution, we can determine many things about the electronic structure of diodes. In EBIC (electron beam induced current) imaging, a field is applied across a junction or other electronic device, and barrier height variations or traps can be observed.

- Voltage imaging uses an applied voltage to obtain contrast from active circuit elements in an electronic circuit. This mode of imaging allows an electron microscope to monitor the functions of a circuit.