Lecture 21
Electron Characterization
Schroder: Chapter 11.2

Announcements

Homework 5/6:
• Due Today at 10:00am.
• I will return it the following Wednesday (6th June).

Homework 6/6:
• Will be online on later today.
• Due Wednesday June 6th at 10:00am.
• I will return it at the final exam (14th June).
• I will make the solutions available on June 6th.

Office Hours
• Next week office hours will be Monday June 4th 13:00-14:00.
Lecture 21

• Scanning Electron Microscopy.
• Transmission Electron Microscopy.
• Electron Energy-Loss Spectroscopy (EELS)
• Auger Electron Spectroscopy (AES).
• Electron Diffraction Spectroscopy.

Electron Beam Techniques

• Electron beam techniques use electrons rather than photons.
• But the strategy remains the same; we just apply some radiation to our sample, and measure its properties.
• Unlike photons, electrons do not have to give up all of their energy to interact with matter.
Electron Beam Techniques

- As in the previous lecture, consider what can happen when an electron is incident on our sample:
  - reflection
  - absorption
  - emission

- SEM
- RHEED
- LEED
- AES
- CL
- EPMA
- EBIC
- TEM
- STEM
- EELS
- voltage contrast
- surface potential

Scanning Electron Microscopy
**Electron Emission**

- The electron yield, $N(E)$, from a surface subjected to a beam of electrons at an energy of a few keV might look something like the following.

![Graph showing Electron Emission](image)

- $N(E)$ vs. $E$

  - Secondary electrons
  - Auger electrons
  - Plasmons (bulk & surface)
  - Backscattered electrons

**Scanning Electron Microscopy**

- A scanning electron microscope (SEM) uses electrons to image a sample.
de Broglie Wavelength

• If an electron is subjected to an accelerating voltage, the wavelength is given by the de Broglie Equation:

\[ \lambda = \frac{h}{\sqrt{2m_0eV}} \]

• Where:
  • \( \lambda \) = Electron wavelength.
  • \( h \) = Planck Constant.
  • \( e \) = Charge on electron.
  • \( V \) = Accelerating voltage.
  • \( m_0 \) = Rest mass of electron.

• For a typical acceleration voltage of 10 – 30 keV:

\[ \lambda = 7 \text{ pm} - 12 \text{ pm} \]

• Thus, an SEM has much better spatial resolution than an optical microscope.

• There are reasons why we cannot reach a resolution of \(~\text{pm}\). But the instrument is not limited by wavelength.
Relativistic Correction

• At high voltages (> 100 kV) we are accelerating electrons to appreciable percentages of the speed of light.

• So instead we must use the relativistic equation for wavelength:

\[ \lambda = \frac{h}{\sqrt{2m_0eV}} \cdot \frac{1}{\sqrt{1 + \frac{eV}{2m_0c^2}}} \]

• Where:
  • \( c \) = Speed of light in vacuum.

Scanning Electron Microscope

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Electron Gun / Source

- Low energy: thermionic source (heated filament).
  - Tungsten.
  - Lanthanum hexaboride (LaB$_6$).
- High energy: field-emission source.
  - Electrons are pulled off of metal by a very large electric field.
  - Require a high vacuum and are difficult to fabricate.

Raster Scanning

- Secondary electrons or backscattered electrons are collected while raster-scanning a focused electron beam over a sample to provide an SEM image.
- More electrons detected → brighter image.
- Surface topography is the main source of contrast in an SEM.
Excitation Volume

- Using a thermionic source the beam diameter is around 0.4 nm, and using a field-emission source the beam diameter is around 0.1 nm.
- The resolution of SEM instruments is not this good.
- As electrons enter the sample they will be scattered.
Voltage Contrast SEM

• Voltage contrast is often employed in SEM semiconductor assessment.
• The idea here is that more electrons are collected from a portion of the device biased positively rather than negatively.
• Voltage-contrast is useful for IC failure analysis.
• You check if voltage is being dropped in the correct regions.

e-Beam Lithography

• When imaging you can damage the material being imaged when using high energies.
• This can be exploited to pattern materials with high resolution.
• 10nm resolution is routinely achieved.
• A computer is used to design patterns.
• PMMA is often used as a resist.
Advantages / Disadvantages

• Advantages of SEM:
  • Include high spatial resolution.
  • High voltage sensitivity (mV).
  • High temporal sensitivity (ns).
  • No capacitance loading.

• Disadvantages of SEM:
  • Cost.

Transmission Electron Microscopy
TEM

- Rather than imaging with backscattered / secondary electrons, transmitted electrons can be used to image a sample.
- Transmission electron microscope (TEMs) transmit electrons through samples.
- Samples must be very thin.
- TEMs have resolutions of ~0.1 nm.

TEM Operation

- Matter will attenuate the beam as it passes through the sample.

https://www.youtube.com/watch?v=I9JYuTpK8Fs
**STEM**

- A scanning-TEM (STEM) uses a focused electron beam to achieve better contrast.
- The STEM raster-scans an electron beam has a diameter of ~0.5 nm.

https://www.youtube.com/watch?v=fQjYuTpiKFs

**Focused Ion Beam**

- Very thin samples (~20-200 nm) must be prepared for TEM / STEM analysis.
- This is often accomplished using a focused ion beam (FIB).
- A FIB is similar to an SEM, except that it uses a Ga+ ion beam, rather than electrons.
- A rastered ion beam generates secondary electrons and ions, both of which may be imaged.
- FIBs can be used for ion milling, selective metal deposition, and nanosurgery, as well as for TEM / STEM sample preparation.
Example Images

- c-Silicon
- Graphene

Cross-Sectional TEM

Electron Energy-Loss Spectroscopy (EELS)
EELS

- Electron energy-loss spectroscopy (EELS) takes advantage of inelastic scattering of electrons.
- Consider an energetic electron interacting with core electrons in an atom:

\[ E_1 + e^- \rightarrow E_2 < E_1 \]

- The energetic electron will have to give up some energy in this interaction.
- We can use the magnitude of this change in energy to characterize the atom.

To cause this transition the energy of the electron must be \( \geq \) some certain amount of energy.
- By measuring the change in energy of the electron we can identify the element.
  - E.g. to remove an L-shell electron from silicon it requires \( \geq 99 \text{ eV} \).
  - We can use this information to quantitatively analyze the elemental composition of the sample.
- In reality we measure a spectrum of electron energy loses. We count the number of electrons losing each amount of energy.
EELS

• To cause this transition the energy of the electron must be $\geq$ some certain amount of energy.

\[
\begin{array}{c|c}
 n^* & SN^{**} \\
 1 & K \\
 2 & L \\
 3 & M \\
 4 & N \\
 5 & O \\
\end{array}
\]

* $n =$ principal quantum number  
** $SN =$ spectroscopic notation

\[
E_{\text{VACUUM}} \rightarrow E_C \rightarrow E_V \rightarrow E_{L_2,3} \rightarrow E_{L_1} \rightarrow E_K 
\]

sharp threshold @ $E_K - E_F$

EELS

• The details of the spectra are in reality quite complex, and beyond the scope of this course.
Auger Electron Spectroscopy (AES)

- We saw previously from our ‘teardrop’ figure that Auger electrons are produced at the surface.

- Auger Electron Spectroscopy (AES) is a surface-analysis technique.
Auger Electron Spectroscopy

- Because we are close to the “neck of the bottle” our analysis area is pretty close to the probe dimensions.
- It is based on the Auger process, which is a specific set of events in the core of the atom. The result of which is an electron being emitted with a characteristic energy.
- It involves 3-electrons (c.f. Auger recombination).

Auger Process

- The basic Auger mechanism involves electron-induced ionization of a core electron, with subsequent Auger recombination.

<table>
<thead>
<tr>
<th>n*</th>
<th>SN**</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>K</td>
</tr>
<tr>
<td>2</td>
<td>L</td>
</tr>
<tr>
<td>3</td>
<td>M</td>
</tr>
<tr>
<td>4</td>
<td>N</td>
</tr>
<tr>
<td>5</td>
<td>O</td>
</tr>
</tbody>
</table>

*K = principal quantum number
**SN = spectroscopic notation

\[ KL_1L_{2,3} = KLL \]
AES Spectra

- Auger peaks are rather weak in an electron yield plot, $N(E)$, since they ride on a background of inelastically scattered electrons.

- AES spectra are normally presented differentiated.

AES Spectrometer

- Below is what an Auger Electron Spectrometer looks like.

https://www.youtube.com/watch?v=SGFwsg8bhe0
Binding Energies

Below are some example binding energies for deep level electrons in transition metals:

<table>
<thead>
<tr>
<th>Element</th>
<th>$2p_{3/2}$ (eV)</th>
<th>3p (eV)</th>
<th>$\Delta$ (eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sc</td>
<td>399</td>
<td>29</td>
<td>370</td>
</tr>
<tr>
<td>Ti</td>
<td>454</td>
<td>33</td>
<td>421</td>
</tr>
<tr>
<td>V</td>
<td>512</td>
<td>37</td>
<td>475</td>
</tr>
<tr>
<td>Cr</td>
<td>574</td>
<td>43</td>
<td>531</td>
</tr>
<tr>
<td>Mn</td>
<td>639</td>
<td>48</td>
<td>591</td>
</tr>
<tr>
<td>Fe</td>
<td>707</td>
<td>53</td>
<td>654</td>
</tr>
<tr>
<td>Co</td>
<td>778</td>
<td>60</td>
<td>718</td>
</tr>
<tr>
<td>Ni</td>
<td>853</td>
<td>67</td>
<td>786</td>
</tr>
<tr>
<td>Cu</td>
<td>933</td>
<td>75</td>
<td>858</td>
</tr>
<tr>
<td>Zn</td>
<td>1022</td>
<td>89</td>
<td>933</td>
</tr>
</tbody>
</table>

Elemental Shifts

- The binding energy of each electron level increases with atomic number.
- The characteristic energy of each Auger process increases also with atomic number.
- This fact makes AES possible: each energy is unique to each element.

https://www.cnyn.unam.mx/~wencel/XPS/MANAES2.pdf
Atomic Identification

- A scan from 0-2000 eV is usually performed for basic atomic identification.
- The peak-to-peak height of first derivative $\rightarrow$ electron energy.


Atomic Identification

- Peak-to-peak heights and tabulated relative Auger sensitivity factors are used for quantitative compositional analysis, with an accuracy of $\sim$5-10%.
AES Depth Profiling

• We can add an ion gun to our system.

[Image: Diagram of AES depth profiling with an ion gun and sample, showing the process of profiling]

https://www.youtube.com/watch?v=5GFwsg8bhe0

• This allows to mill into the sample and study elemental composition as a function of depth.

AES Depth Profiling

• AES depth profiling is accomplished using a rastered ion beam to mill through a sample layer while sequentially monitoring various atomic spectra.

[Graph showing AES signal over sputter time with two layers of SiO₂ and Si]
LEED

- Low-energy electron diffraction (LEED) provides information about the 2D surface atomic structure.
- Low energy (~10-1000 eV) electrons elastically scatter off of a surface, resulting in a diffraction pattern that is monitored on a fluorescent screen.
- LEED, and most surface science measurements, must be accomplished in ultra-high vacuum (UHV), i.e., at a pressure of less than $10^{-6}$ torr.
- One reason for this is that the impingement rate of background gases onto a surface in a vacuum of $10^{-6}$ torr is approximately 1 monolayer per second.
RHEED

- Reflection high-energy electron diffraction (RHEED) is used to measure thickness during molecular beam epitaxy.
- Electrons are fired at surface at very small angle.
- Electrons are scattered by lattice.
- Electrons will interfere constructively at certain angles, when a complete monolayer is present.

Next Time...

- Ion Beam Spectroscopy.
- Scanning Probe Microscopy.

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Sample

Ion beam

Mass filter

Primary ions

Sputtered ions

Detector/mass filter

X-y scanned sharp tip (Pt, W...)

Conductive sample

D ~0.5-1 nm

Intensity vs. mass units

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