EE213. Microscopic Nanocharacterization of Materials
Spring 2016

Lecture 3

Tentative Outline

Week 1: Introduction: What is Micro/Nano Characterization?

Week 2: Electron Beam Induced Excitation Methods
A. Reflection Scanning Electron Microscopy
B. Auger Electron Microscopy/Spectroscopy
C. Electron Beam Induced X-Ray Analysis
D. Electron Energy Loss Spectroscopy
E. Transmission Electron Microscopy
   a. Scanning Transmission Electron Microscopy (STEM)
   b. Conventional Transmission Electron Microscopy (TEM)
   c. Energy Filtered Electron Microscopy
From Where do the secondaries Come?

only SE1 were produced by incident electrons at point of impact near surface.

- SE2 depends on sample
- SE3 indep. of sample
Secondary Electron Yield (1)

I_0

\[ r = \sqrt{x^2 + z^2} \]

1. Secondary e^- produced at depth z will have probability of escape of \( P(z)/\rho(x) = e^{-r/\rho_{\text{ex}}}. \) Beer's law.

2. Assume isotropic emission of secondary in the xz plane. Then \# produced at depth z at point 0 = \# which escape:

\[
\frac{dI(z)}{dz} = \int_0^\infty \frac{1}{4\pi r^2} e^{-r/\rho_{\text{ex}}} \cdot 2\pi r dr
\]

3. Probability of incident electron of energy \( E_0 \) produce inelastic collision at depth z is:

\[ P_{\text{in}}(z) = \frac{d^2}{\Lambda_{\text{in}}(z)} \]

4. If current at depth z is \( I(z) \), then rate of production at z is \( I(z)P_{\text{in}}(z) \).

If we assume that most x-rays that escape are within \( \Lambda_{\text{in}} \) of surface \( (\Lambda_{\text{ex}} < \Lambda_{\text{in}}) \) then \( I(z) \approx I_0 \)

\[ \text{rate of production of inelastic events at } z \text{ is:} \]

\[ I_0 \frac{d^2}{\Lambda_{\text{in}}(z)} \]
5. If we assume that if then a enough energy
lost in an incl. collision, we produce a
secondary e⁻, then 

\[ \frac{N_{\text{sec}}}{N_{\text{inc}}} \approx \frac{E_{\text{loss}}}{E_{\text{sec}}} \text{ the secondary e⁻ energy} \]

6. More just use average energy lost per incl. and

\[ dI_{\text{sec}}(z) = I_0 \frac{dE_{\text{loss}}}{dE_{\text{sec}}} dz \]

7. For sample thickness \( t \),

\[ I_{\text{sec}} = \int_0^t I_0 \frac{dE_{\text{loss}}}{E_{\text{inc}}} \frac{1}{4\pi E_{\text{sec}}} \exp \frac{-\frac{E_{\text{inc}}}{2E_{\text{sec}}}}{2\pi t} dx \]

8. If \( N_{\text{sec}} \ll N_{\text{inc}} \), so \( N_{\text{inc}}(E_2) = N_{\text{inc}}(E_1) \), and \( E_{\text{loss}} \ll E_{\text{sec}} \)

\[ d_{\text{sec}} = \frac{I_{\text{sec}}}{I_0} = \frac{1}{2} \frac{E_{\text{loss}}}{E_{\text{sec}}} \int_0^t \frac{dx}{x} \exp \frac{-E_{\text{loss}}}{2E_{\text{sec}}} \]

\[ \text{approximate integral of 2nd kind} \]

9. \[ S_{\text{sec}} \approx \frac{1}{2} \frac{E_{\text{loss}}}{E_{\text{sec}}} \frac{N_{\text{sec}}}{N_{\text{inc}}} \left[ 1 - E_2(t/V_{\text{inc}}) \right] \]
Secondary Electron Yield 3

\[ \delta_{\text{SEC}} \approx \frac{1}{2} \frac{E_{\text{in}}^{\text{loss}}}{E_{\text{sec}}} \frac{A_{\text{sec}}}{A_{\min}(E_{\text{in}})} \left[ 1 - E_{2}(t/A_{\text{sec}}) \right] \]

where \( E_{2}(t/A_{\text{sec}}) = \int_{t}^{\infty} \frac{dy}{y} e^{-t/y} \) \text{sec tables.}

\( E_{2}(t/A_{\text{sec}}) \to 0 \) as \( t/A_{\text{sec}} \to \infty \), ie bulk sod

\( \to 1 \) as \( t/A_{\text{sec}} \to 0 \)

\[ \therefore \text{for bulk yielding} \quad \delta_{\text{SEC}}(\infty) \approx \frac{1}{2} \frac{E_{\text{in}}^{\text{loss}}}{E_{\text{sec}}} \frac{A_{\text{sec}}}{A_{\min}(E_{\text{in}})} \]

should be valid for \( E_{\text{in}} > E_{\text{m}} \), energy where secondary yield is max.

**NOTE:** This predicts inset shapes of yield curve vs energy as well as agreement with experiment.
Electron Beam Induced Secondary Electron Emission

\[ \frac{\delta}{\delta_{\text{max}}} \]

- Range of experimental data
- KK
- MI, KK
- Theory

\[ E_0/E_{\text{max}} \]
Backscattered Produced Secondary 1

\[ \delta_{\text{SE}} = \frac{1}{2} \frac{E_{\text{in}}}{E_{\text{sec}}} \frac{\Lambda_{\text{sec}}}{\Lambda_{\text{in}}(E_{\text{in}})} \]

\( \delta_{\text{BSE}} \) sec. yield by primary electrons of energy \( E_{\text{in}} \)

# secondary produced by BSE

\[ \delta_{\text{BSE}} = \eta \frac{I_{\text{BSE}}}{I_{\text{B}}} \]

where \( \eta = \frac{I_{\text{B}}}{I_{\text{D}}} \) the BSE yield

\[ I_{\text{BSE}} = 2 \left[ \frac{1}{2} \frac{E_{\text{in}}}{E_{\text{sec}}} \frac{\Lambda_{\text{sec}}}{\Lambda_{\text{in}}(E_{\text{in}})} \right], \quad E_{\text{B}} = \text{avg. energy of BSE} \]

\( \eta = \frac{I_{\text{B}}}{I_{\text{D}}} \)

2 is due to fact that BSE are NOT ISOTROPICALLY emitted but rather follow a cosine distribution.

\[ \delta_{\text{total}} = \delta_{\text{SE}} + \delta_{\text{BSE}} \]

\[ \delta_{\text{SE1}} \quad \delta_{\text{SE2}} \]
Angular Distribution of Backscattered Electrons

From Reimer, 1985
Backscattered Produced Secondary 2

\[ S = S_{\text{SEC}} + S_{\text{BSEC}} \]

\[ = S_{\text{SEC}} \left( 1 + \frac{S_{\text{BSEC}}}{S_{\text{SEC}}} \right) \]

\[ = S_{\text{SEC}} \left[ 1 + \eta \left( \frac{\frac{E_{\text{in}}}{E_{\text{sec}}} \Lambda_{\text{sec}}}{\frac{1}{2} \Lambda_{\text{sec}} \Lambda_{\text{in}}(E_{\text{in}})} \right) \right] \]

\[ S = S_{\text{SEC}} \left[ 1 + \eta \left( \frac{2 \Lambda_{\text{in}}(E_{\text{in}})}{\Lambda_{\text{in}}(E_{\text{in}})} \right) \right] \]

define \( \beta = \frac{2 \Lambda_{\text{in}}(E_{\text{in}})}{\Lambda_{\text{in}}(E_{\text{in}})} \), sometimes called \( \gamma \) in literature.

Then total secondary yield is:

\[ S = S_{\text{SEC}} \left[ 1 + \eta \beta \right] \]

\( \beta \) normally around 1.5 - 2.5 \( \eta \)

Thus a significant fraction of secondaries can be produced by BSE.
Effect of Electron Backscattering on Secondary Electron Yield

**FIG. 4.** Backscattering coefficient $\eta$ obtained from Monte Carlo calculations and the experiment. Dotted line: experiment; solid line: Monte Carlo calculations.

**FIG. 5.** The ratio of the secondary yield due to the primary electron to the total secondary yield, calculated for various metals at normal incidence.

Thus, the emitted secondaries have different spatial distributions:

\[ \delta S = \delta S_{SE1} + \delta S_{SE2} \]

\[ \overrightarrow{SE1} \quad \overrightarrow{SE2} \]

\[ Io \]

\[ x_{g} = x_{g} \tan(\pi - \theta) \]

\[ \frac{1}{4} \text{ of elastic scattering} \]

To get \( x_{g} \), we just find \( \text{avg} (\pi - \theta) \).
Backscattered Produced Secondaries

How can we eliminate the effect of these BSE produced secondary electrons on the "image"?

\[ \delta = \delta_{\text{SE1}} + \delta_{\text{SE2}} + \delta_{\text{others}} \]

- \( \delta_{\text{SE1}} \) due to primary electron
- \( \delta_{\text{SE2}} \) due to BSE
- \( \delta_{\text{others}} \) can be removed by exp. design

\[ \delta = \delta_{\text{SE}} (1 + \beta n) \]

use another detector

an annular detector which detects the higher energy BSE. Signal it gets is:

\[ \frac{I_{B}}{I_{0}} = \eta_{B} \rightarrow \text{eff. yield of detector} \]

\[ I_{\text{DIFF}} = \frac{I_{\text{SEC}}}{I_{0}} - K \frac{I_{\text{ANN}}}{I_{0}} \]

\[ = \delta_{\text{SE}} (1 + \beta n) \]  

\[ \frac{I_{\text{DIFF}}}{I_{0}} = \delta_{\text{SE}} + \eta \left[ \delta_{\text{SE}} \beta f_{\text{SE}} - K f_{\text{ANN}} \right] \]

choose \( K \) so that \( \omega \rightarrow 0 \)
Backscattered produced secondary electrons, SE2

Primary produced secondary electrons, SE1

$K_{\delta p} I_0$

$K_{\delta p} r \eta I_0$

INTENSITY

radius
Contribution of Backscattered Electrons to Secondary Electron Image
In an SEM (fibroblast cell with Ag stained nucleus)

Secondary electron image (S)  Backscattered electron image (B)

From Crewe and Lin.
Ultramicroscopy.1.(3-4).231-238(1976)
Effect of Electron Backscattering on Secondary Electron Yield

Spatial Distribution of Secondaries.

- Delocalization of inelastic scattering,
  \[ \Delta \rho \propto \frac{\Delta E}{E} \]

\[ \Delta \rho \propto P_0 \sqrt{\Theta^2 + \Theta_E^2} = \tan \Theta, \quad \Theta_E = \frac{\Delta E}{P_0 \Delta \rho} \]

**NOTE:** For every energy loss there is a characteristic scattering angle \( \Theta_E \)
- Larger \( \Delta E \) \( \Rightarrow \) larger \( \Theta_E \)
  - But for small \( \Delta E \), \( \Theta_E \) \( \approx \) 10-20°

- Average scattering angle, \( \overline{\Theta} \)

\[ \overline{\Theta} = \frac{\int_{0}^{\Theta_{\text{max}}} \Theta \cdot \frac{df}{d\Omega} \cdot d\Omega}{\int_{0}^{\Theta_{\text{max}}} \frac{df}{d\Omega} \cdot d\Omega} \]

\[ \frac{df}{d\Omega} \propto \frac{1}{\Theta^2 + \Theta_E^2} \]

The inelastic scattering angular distribution.

\[ \overline{\Theta} = \frac{\int_{0}^{\Theta_{\text{max}}} \Theta \cdot \left[ \frac{1}{\Theta^2 + \Theta_E^2} \right] d\Omega}{\int_{0}^{\Theta_{\text{max}}} \frac{1}{\Theta^2 + \Theta_E^2} d\Omega} = 2 \alpha \sin \Theta \partial \theta \partial \alpha \]

\[ \overline{\Theta} = \frac{\int_{0}^{\Theta_{\text{max}}} \frac{\Theta^2 d\Theta}{\Theta^2 + \Theta_E^2}}{\int_{0}^{\Theta_{\text{max}}} \frac{\Theta d\Theta}{\Theta^2 + \Theta_E^2}} \quad \text{assumining} \quad \sin \Theta = \Theta \]

\[ \overline{\Theta} = \frac{\int_{0}^{\Theta_{\text{max}}} \Theta^2 d\Theta}{\int_{0}^{\Theta_{\text{max}}} \Theta d\Theta} \]
Spatial Distribution of Secondaries, z

Since $\theta_E \ll 1$ then $\theta_{max} \approx \sqrt{2\theta_E}$

and $\bar{\theta} \approx \frac{\sqrt{2\theta_E}}{\ln(2/\theta_E)} \gg \theta_E$

:: the average momentum transfer

$\Delta p = P_i \bar{\theta}$

and with $\Delta p \Delta x \approx h$ we get

$\Delta x \approx \frac{h}{\Delta p} = \frac{h}{P_i \bar{\theta}} \approx \frac{h}{P_i} \frac{\ln(2/\theta_E)}{\sqrt{2\theta_E}}$

$\lambda = h/P_i \leq \sqrt{\frac{150}{E_i}}$ non-relativistically

:: $\Delta x \approx \sqrt{\frac{150}{E_i}} \left( \frac{\ln(4E_i/E)}{\sqrt{E/E_i}} \right)$, where $\theta_E \approx \frac{E}{Z \lambda E_i}$

:: $\Delta x = \sqrt{\frac{150}{E}} \frac{\ln(4E_i/E)}{\ln(2/\theta_E)}$ in $\text{Å}, \text{eV}$

NOTE: $\Delta x$ relatively indep of $E_i$

and $\Delta x \downarrow$ as $E \uparrow$
Spatial Distribution of Semidemes

To find the spatial resolution attainable using semidemes we compare the spatial distribution of them with that of the incident electron beam.

\( R_{BS}(r) = \int_0^{E_0} dE \int 2\pi r'dr' \frac{dQ_0(e', r')}{dE} I_0(r') \)

radial dist. of incident beam.

radial distrib. of emerging semideme.

produced by energy loss, E

\( \text{then if we assume "Gaussian" distributions for both the beam and emerging semidemes (not quite true, but simplifies things)} \)

\[ \bar{r}_E \approx \sqrt{r_B^2 + (\Delta X)^2} \]

\[ \Delta X \approx \sqrt{\frac{150}{E}} \ln \left( \frac{4E_0}{E} \right) \]

avg. E loss

\( \therefore \text{we only notice effect of } \bar{r}_B \sim \Delta X // \)

Eg: \( \text{if } E_0 = E_i = 30 \text{ keV}, E = 30.1 \text{ keV } (\gamma = 0) \)

\( \Delta X \approx 1.8 \)
Demonstration of the Non-Localization of Inelastic Electron Scattering
(a manifestation of the Heisenberg Uncertainty Principle)

Pt on Thin Carbon Substrate

High Energy Loss, large momentum transfer secondaries

Backscattered produced secondary electrons, SE2

Primary produced secondary electrons, SE1

$ICOLLECTED$

$K \delta_p I_0$

$K \delta_p r \eta I_0$

radius
From Where do the secondaries Come?

only SE1 were produced by incident electrons at point of impact near surface.

- SE2 depends on sample
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Electron Beam Induced Secondary Electron Emission

\[ \frac{\delta}{\delta_{\text{max}}} \]

\( E_0/E_{\text{max}} \)

range of experimental data

KK

MI, KK

Theory
\[
\delta_{\text{SEC}} = \frac{1}{2} \frac{E_{\text{in}}}{E_{\text{SEC}}} \frac{\Lambda_{\text{SEC}}}{\Lambda_{\text{IN}}(E_0)} \left[ 1 - E_2(t/\Lambda_{\text{SEC}}) \right]
\]

References: Lecture 3,4:

*Electron Scattering:*

M. Inokuti, Rev. Mod. Phys. 43, 297 (1971)

P. Crozier, Phil. Mag. 61(3), 311-336 (1990)

*Secondary Emission:*


*Electron Backscattering:*


Sternglass, Phys. Rev. 95, 345 (1954)
\[
\frac{d\eta}{d(E/E_0)}
\]
Secondary electron emission

$\delta_M$

$\delta$

$I$

$E_M$

$\approx 300-800\text{eV}$

in this region sample can charge up positively

$\geq \text{KeV}$

in this region sample can charge up negatively

depends upon work function
Electron Backscattering

probability of interaction, \( P = n \sigma dx \)

- current scattered into a solid angle \( d \Omega \) by elastic scattering:
  \[
  \frac{dI_{\text{el}}}{d \Omega} = I_0 n t \frac{d \sigma}{d \Omega} \quad \text{(from thickness \( t \))}
  \]

- if we assume a large scattering event takes electron out of sample

- the backscattering yield \( \eta \) then:
  \[
  \eta = \frac{I_{\text{B}}}{I_0} = \frac{\int_{\Omega} 2 \pi \sin \theta d \theta \frac{d E_{\text{B}}}{d \Omega}}{\int_{\Omega} 2 \pi \sin \theta d \theta}
  \]

assuming large \( \lambda \) events are only Rutherford scattering
then
\[
\frac{d I_{\text{B}}}{d \Omega} = \frac{d I_{\text{el}}}{d \Omega} = I_0 n t \frac{d \sigma}{d \Omega} \quad \text{the Rutherford cross section.}
\]

but
\[
\frac{d \sigma}{d \Omega} = \text{const} \frac{Z^2}{E_0^2} \frac{1}{\sin^4(\theta/2)}
\]

- \( \eta = \int_{\Omega} 2 \pi \sin \theta d \theta \frac{I_0 n t (\text{meas.}) Z^2}{E_0^2} \frac{1}{\sin^4(\theta/2)} \)

\[
\eta = \text{Knt} Z^2 \quad \text{where } K = \frac{\pi^4}{16 \pi^2 a_0^2}
\]

NOTE: \( K \) is material independent
and depends only on the electron wavelength

\[
\lambda = \left[ \frac{h^2}{2 m E_0 (1 + E_0 / (2 m c^2))} \right]^{1/2} \text{relativistic wavelength}
\]
Electron Backscattering.

\[ \eta = K \text{r}t Z^2 \]

so this says the BSE yield is linear with thickness.

But, if \( t \) too large, multiple scattering, so electron cannot scattered back into material.

And if \( t \rightarrow \) Range of electrons in material, \( \eta \rightarrow \) constant.

One other problem.

If \( t \) in the linear region of \( \eta \), \( k \) is too small by 2-3x experimental values. (reasonable since we assumed only 1 death event)

But all other properties of \( \eta \) are predicted by this simple expression.

Let's see if we can now get the expression for a solid target. To do this we just need to find the "effective" depth from which the backscattered electrons come, \( t_{eff} \).

To do this, we need to find the range (depth) of the electrons in the material.
Electron Backscattering

The "depth" range or "maximum interaction depth" is the depth in the material beyond which few electrons travel. It is not the "total range" or total path of the electrons before stopping.

We calculate the "Bethe Range," which is path due to inelastic events, and such calculate \( \lambda_{\text{m}} \) for every nucleus until electron loses almost all its energy.

\[
\lambda_{\text{m}}(E_0) = \frac{E_0}{35.9 m_N \tilde{Z} \ln(4E_0/E_{\text{in}})}
\]

\( E_0 \) is the "stopping energy," it is an energy beyond which there is little additional effect on range because \( \lambda_{\text{m}} \) is so small.
Electron Backscattering

the "Bethe Range" or inelastic range is:

\[ R_\text{B}(E_0) = \int_{E_S}^{E_0} \frac{A_{\text{in}}(E) dE}{E_{\text{in}}} \]

The expression is not accurate at low energies, since
\[ E_S < E_{\text{in}}/4 \]
then \[ \frac{A_{\text{in}}(E)}{E_{\text{in}}} \] blows up.

To avoid this we generally take \[ E_S > E_{\text{in}} \]

Using \[ E_{\text{in}} = 12.3 \sqrt{E} \text{ meV} \] we can evaluate \( R_\text{B}(E_0) \) analytically.

For \( E_0 \gg E_S \) we get

\[ R_\text{B}(E_0) = \frac{11.32 \times 10^{-4} E_0^2}{n \cdot \ln\left(\frac{325 E_0}{\sqrt{Z}}\right)} \text{ in } \text{Å}, \text{ with } E_0 \text{ in } \text{meV}, \]

\[ n \text{ in } \text{cm}^{-3} \]

One can show using Monte Carlo calculations that the "depth Range" \( R \) is related to \( R_\text{B} \) as:

\[ R \approx R_\text{B} Z^{-\frac{1}{3}} \]

For large atoms, \( Z \) means more inelastic scattering.
Electron Backscattering

Now we estimate the effective BSE depth.

The avg. energy the backscattered electrons have after escaping the sample is

\[ E_b = E_0 - 2\Delta E \]

which assumes an energy loss of \( \Delta E \) on the way in and on the way out is about the same.

Thus, the effective energy the electron has before being backscattered is

\[ E_{\text{eff}} = E_0 - \Delta E = \frac{1}{2}(E_0 + E_b) \]

thus, using this we can find out how far into the ivpd the electron has gone before being backscattered.

\( E_b \) is gotten from experimental measurements or MC simulations.

\[ \frac{R_0(E_{\text{eff}})}{R_0(E_0)} = 1 - \left( \frac{E_{\text{eff}}}{E_0} \right)^2 \frac{\ln(-325E_0/\Delta E)}{\ln(-325E_{\text{eff}}/\Delta E)} \]

This changes by \( \pm 10\% \) from \( Z = 14 \rightarrow 74 \)

and with \( E_0 = 1 - 50 \text{keV} \).

We get

\[ \frac{R_0(E_{\text{eff}})}{R_0(E_0)} \approx 0.45 \]

and that range.
Electron Backscattering

\[ \tau_{\text{eff}} = R_{\text{eff}} = R_a Z^{-1/3} \]

or

\[ \tau_{\text{eff}} = 0.45 Z^{-1/3} R_a \]

\[ \eta = KN \tau_{\text{eff}} Z^2 \]

since the \( K \) for Rutherford scattering is about \( Z^{-3} \) less than experiment, we just multiply the \( K \) by 2.5 and get

\[ \eta = \frac{0.21 Z^{2/3}}{\ln(0.325 E_0/\sqrt{Z})} \text{ eV mev} \]

This gives reasonable agreement with exp. data.

It is a bit high at high \( Z \)

and is off a bit at low \( E_0 \)

but agrees within 20% with experiment

and Monte Carlo simulations.
Electron Backscattering Yield

\[ \eta_\infty \]

- NIEDRIG (1984)
- ARCHARD (1961)
- EVERHART (1960)
- simplified MSI

ATOMIC NUMBER, Z
Electron Backscattering Yield Measurements

(A) \[ \eta(T) \]

- \( \eta_{\infty} \)
- \( E_0 \)
- \( E_1 > E_0 \)
- self-supporting film

- single scattering
- multiple scattering

(B) \[ \eta(T) \]

- \( \eta_{L}(\infty) \)
- \( \eta_{S}(\infty) \)

- \( Z_{\text{OVERLAYER}} > Z_{\text{SUBSTRATE}} \)

THICKNESS, T

(C) \[ \eta(T) \]

- \( \eta_{S}(\infty) \)
- \( \eta_{L}(\infty) \)

- \( Z_{\text{OVERLAYER}} < Z_{\text{SUBSTRATE}} \)

THICKNESS, T
Experimental Measurements of Electron Backscattering in Thin Films

Electron Backscattering Yield Measurements

Figure 4. Comparison of the computed backscattering probability $R_{ST}$ as a function of the incident energy $E_0$ (a) and the atomic number $Z$ (b) with results of measurements from Neubert and Rogaschewski [13] (a) and from Bishop [15] for 5 ($\circ$), 10 ($\triangle$) and 30 keV ($\square$) (b).