12.141
Electron Microprobe Analysis

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Electron Microprobe
or
Electron Probe Microanalyzer (EPMA)

Surface characterization of solids at the **micrometer-scale**:

- Surface topography and compositional imaging
- Complete chemical analysis of microscopic volumes
  *(Electron beam induced X-ray emission spectrometry)*
Signals produced in the Electron Microprobe

Electron beam

Cathodoluminescence (CL)  Back-scattered electron (BSE)

Characteristic X-ray  Secondary electron (SE)

Specimen
Qualitative analysis

- Visual characterization and identification of phases in image (shape, size, surface relief, etc.)

- Identification of elements in each phase (no concentration measurement)
Semi-quantitative analysis

- Spatial distribution of elements in an image
- Quick and approximate spot concentration measurement without calibration
Quantitative analysis

- Full quantitative micro-chemical analysis
  Concentration of all elements present at the spot

- Elemental concentration mapping
  Concentration of all elements present at each pixel of the image
Electron-specimen interactions

Elastic Scattering

- Back-scattered electron

Inelastic Scattering

- Characteristic X-rays
- Secondary electron
- Cathodoluminescence
Electron interaction volume
Electron interaction volume

\[ R = 0.0276 \, E^{1.67} \frac{A}{\rho \, Z^{0.889}} \]

(\( A \) = atomic weight, \( \rho \) = density)

(Kanaya-Okayama range)

- Increases with electron beam energy, \( E \)
- Decreases with sample atomic number, \( Z \)
Electron interaction volume

*Typical ranges (15 kV, perpendicular beam):*

- C (Z = 6) \(1.8 \, \mu m\)
- Fe (Z = 26) \(1.1 \, \mu m\)
- U (Z = 92) \(0.8 \, \mu m\)
Electron probe diameter and Electron interaction volume

- **Probe Diameter (PD):**
  - Probes of different diameters (e.g., 10KV, 30KV) are shown.
  - Labels for materials like W (tungsten) and LaB6 are indicated.

- **Electron Range:**
  - Graphs show electron range vs. atomic number for different 5 keV, 15 keV, and 25 keV.
  - Elements like C (carbon), Fe (iron), U (uranium) are shown.

- **Probe Current:**
  - X-axis represents probe current (I, in A).
  - Y-axis represents probe diameter (PD, in nm).
  - Different current levels are depicted with distinct curves.
Elastic scattering cross-section

For a scattering angle \( \phi_e \), cross-section

\( (\text{events.cm}^2/\text{e}^-.\text{atom}) \)

\[ Q_e = 1.62 \times 10^{-20} \left( \frac{Z^2}{E^2} \right) \cot^2\left( \frac{\phi_e}{2} \right) \]

- \( Z \): atomic number
- \( E \): beam energy
- \( \phi_e \): elastic scattering angle

- Increases with sample atomic number, \( Z \)
- Decreases with electron beam energy, \( E \)
Back-scattered electron (BSE) (Elastic scattering)

- Beam electrons scattered backward from specimen surface
- High energy electrons with energy about the same as that of the electron beam

- BSE image resolution improves with shrinking of the electron interaction volume through:
  1. Decrease in beam energy
  2. Increase in specimen atomic number
Electron backscatter coefficient

BSE image contrast is better among low Atomic Number elements
Backscattered electron image

**Back-scattered electron**

Polished surface

Function of composition

**Plane polarized transmitted light**

Thin section

Function of optical properties

High-Z elements

Low-Z elements

500 µm
The X-ray spectrum

Characteristic X-rays

Continuum X-rays

Intensity

Wavelength

Energy
Phase identification: EDS X-ray spectra

Ilmenite (ilm): FeTiO₃

hbl: hydrous Ca-Fe-Mg-Al-silicate

plg: Na-Ca-Al-silicate

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JEOL JXA-8200 Superprobe

Sample surface is perpendicular to the electron beam
Back-scattered electron detector

Located vertically above the specimen
A split ring shape

solid-state diode
Compositional and topographic imaging with BSE detector

A+B
Compositional mode

A-B
Topographic mode
Energy Dispersive X-ray Spectrometer (EDS)

- **EDS detector:** a ‘p-n’ layer of intrinsic Si(Li) semiconductor; Be window; aperture wheel

- **Multichannel analyzer (MCA)** processes the X-ray signal
Secondary electron (SE)  
(Inelastic scattering)

- Electrons from specimen surface are mobilized by beam electrons

- Emitted at low energies (typical: <10 eV)  
(recall BSE are high energy beam electrons that underwent elastic scattering, $E_1=E_0$, $E_0$ typically being 10-20 keV)
Secondary electron detector

Located on the side wall of the sample chamber
Imaging with the E-T detector

-ve Faraday cage bias
only BSE
Surfaces in direct line of sight are illuminated

+ve Faraday cage bias
BSE + SE
All surfaces are illuminated
Cathodoluminescence (CL)

Light generated from semiconductor samples through electron beam interaction

- Pure material has an empty conduction band; does not conduct
- Trace impurities add additional energy levels in the band gap that can accept electrons in the excited state
- CL photon is emitted as electron drops back to the valence band
Cathodoluminescence spectrometer

Optical microscope camera (not used)

Optical microscope light (turned off)

Optical spectrometer
Cathodoluminescence spectrometry

CL spectrum

Intensity vs. Energy