JEOL JXA-8200 Superprobe

Electron gun
Condenser lens
Optical Microscope
Objective lens
_high_vacuum_

Liq N₂ for EDS

EDS
WDS

Sample exchange chamber

Perpendicular geometry
Back-scattered electron detector

Top view of specimen
Compositional and topographic imaging with BSE

Scanning backscattered electron images of a Zn-Sn composite collected through a solid-state diode detector in (A) A+B, or compositional mode; (B) A-B, or topographic mode.
Secondary electrons

- Specimen electrons mobilized by electron beam through inelastic scattering

- Secondary electrons have lower energy compared to backscattered electrons

(useful in studying surface topography)
Secondary electron detector

Side view of specimen
Secondary electron imaging

-ve Faraday cage bias
less SE
less topographic contrast

+ve Faraday cage bias
more SE
better topographic contrast
Cathodoluminescence

Light generated from sample through electron beam interaction

- Band gap energy, $E_{\text{gap}}$, is a property of the semiconductor
- Trace impurities change $E_{\text{gap}}$ by adding additional energy states in the band gap
Photomultiplier for secondary electron imaging is used as CL detector.

Optical arrangement is the same as for the optical microscope.
Cathodoluminescence spectrometer

Optical microscope
Camera (not used)

Optical microscope
Light (turned off)

JXA-8200

Optical spectrometer
Integrated CL and X-ray spectrometry

CL spectrum

3 band CL map

3 element X-ray map

CL wavelength band may be correlated with an element

Scatter plot
Wavelength Dispersive Spectrometer (WDS)
WDS Analyzing crystal

Flipping motor
Bragg's Law

\[ n\lambda = 2d \sin \theta \]

= path length ABC

\( n \) = order of reflection

(any integer)

\( \theta \) = angle of diffraction

\( d \) = lattice spacing
First and second order reflections

\[ 1\lambda = 2d \sin \theta_1 \]
\[ = \text{ABC} \]

\[ 2\lambda = 2d \sin \theta_2 \]
\[ = \text{DEF} \]

\[ \text{path DEF} = 2\times \text{path ABC} \]
Diffraction angle changes with wavelength being diffracted (for the same order of reflection, n)
WDS Analyzing crystals with different “d” spacings

<table>
<thead>
<tr>
<th>Name</th>
<th>2d (Å)</th>
<th>Type</th>
<th>Elements usually analyzed</th>
</tr>
</thead>
<tbody>
<tr>
<td>LDEC</td>
<td>98</td>
<td>Ni/C Multi-layer</td>
<td>B-O (Kα), optimized for C analysis</td>
</tr>
<tr>
<td>STE</td>
<td>100.4</td>
<td>Pb stearate</td>
<td>B-O (Kα), optimized for C analysis</td>
</tr>
<tr>
<td>LDE1</td>
<td>59.8</td>
<td>W/Si Multi-layer</td>
<td>C-F (Kα), optimized for O analysis</td>
</tr>
<tr>
<td>TAP</td>
<td>25.8</td>
<td>Thallium acid phthalate</td>
<td>Na-P (Kα); Cu-Zr (Lα); Sm-Au (Mα)</td>
</tr>
<tr>
<td>PET</td>
<td>8.742</td>
<td>Pentaerythritol</td>
<td>S-Mn (Kα); Nb-Pm (Lα); Hg-U (Mα)</td>
</tr>
<tr>
<td>LIF</td>
<td>4.028</td>
<td>Lithium fluoride</td>
<td>Ti-Rb (Kα); Ba-U (Lα)</td>
</tr>
</tbody>
</table>
WDS detector: Proportional counter

- **Count rate depends on bias and gas used**

- **Tungsten collection wire set at 1-3 kV bias**

- **Flow counter: 90% Ar + 10% CH$_4$ (P-10); poly-propylene window**

- **Sealed counter: Xe or Kr; Be window**
Bias in proportional counter

best count rate
Counting efficiency of gas

Heavy elements

Light elements
WDS signal processing

Single channel analyzer (SCA) and pulse height analysis (PHA)

Only pulses in this voltage interval are counted
WDS Focusing geometry

\[ L = n\lambda \cdot \frac{R}{d} \]
Curved diffracting crystals

Johansson type
- bending curvature: 2R
- polished and ground to R

Johan type
- only bent to 2R, not ground

FWHM of fully focusing Johansson-type crystal \( \sim 10 \) eV

Some defocusing in Johan-type, but resolution is not compromised
Defocusing in beam-rastered WDS X-ray maps

As the beam moves off the optic axis, the displacement in the specimen plane is equivalent to a change in the angle of incidence of the x-rays on the crystal by an angle $\Delta \theta$. 

major axis of WDS focusing ellipsoid

direction of defocusing
WDS: changing the angle of diffraction

\[ n\lambda_1 = 2d \sin \theta_1 \]
\[ L_1 = n\lambda_1 \cdot \frac{R}{d} \]

\[ n\lambda_2 = 2d \sin \theta_2 \]
\[ L_2 = n\lambda_2 \cdot \frac{R}{d} \]

or, \[ n_2\lambda_1 = 2d \sin \theta_2 \]
\[ L_2 = n_2\lambda_1 \cdot \frac{R}{d} \]
Theoretical and actual limits of spectrometer movement

$2R \leq L \leq 0$
EPMA: Quantitative analysis procedure

- Sample preparation
- Qualitative analysis with EDS
- Standard intensity measurement (calibration)
- Measurement of X-ray intensities in the specimen
- Data reduction through matrix corrections
Sample preparation

- Sample cut and mounted in epoxy
- Polished first with coarse SiC paper, then with alumina grit slurry (final size: ≤0.25 μm) ¹
- Washed with water in ultrasonic cleaner ²
- Dried with blow duster and air
- Carbon coated ³

¹: diamond paste or colloidal silica for some samples; dry polishing paper for water-soluble samples
²: ethanol may be used sparingly; cleaned with blow duster and cloth for samples that dissolve in water
³: for insulators; if standards are coated, however, all samples must be coated
Sample prep: carbon coating

Sample chamber (bell jar)
carbon coater

carbon rod #1
carbon rod #2
samples

150Å  200Å  250Å  300Å  350Å
12.119 Analytical Techniques for Studying Environmental and Geologic Samples
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