The probe

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EPMA - electron probe microanalysis

Probe signals

- Cathodoluminescence (CL)
- Characteristic X-ray
- Back-scattered electron (BSE)
- Secondary electron (SE)
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'New' signal - cathodoluminescence
visible and near-visible radiation

Application: identify impurities in semiconductors
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The instrument
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Measuring x-ray wavelengths

In Braggs' law, keep d constant (d is known)
use single crystal (remember monochromator)

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

Measure \( \theta \) to get \( \lambda \) - identify & quantify element

Use curved single crystal for focusing
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Measuring x-ray wavelengths

Focusing or Rowland circle radius kept constant. Vary $\theta$ by translating crystal away from specimen & rotating. Counter moved to stay near focus point on circle.

$$n\lambda_1 = 2d \sin \theta_1$$

$$L_1 = n\lambda_1 . R/d$$
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**Qualitative**
- Identify and characterize phases (shape, size, surface relief, etc.)
- Elements present in each phase

**Quantitative**
- Complete chemical analysis on a sub-micro scale
- Elemental concentration mapping
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Interaction volume details
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Interaction volume

Depth increases with accelerating voltage
decreases with at. no.
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Interaction volume

Diameter increases with probe current
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**Matrix corrections for quantitative x-ray analysis**

For a each element:

\[
\frac{C}{C^\circ} \sim \frac{I}{I^\circ} = k
\]

or:

\[
\frac{C}{C^\circ} = k \cdot ZAF
\]

- \(C\) = concn in specimen
- \(C^\circ\) = concn in std
- \(I\) = intensity from specimen
- \(I^\circ\) = intensity from std
- \(ZAF\) = matrix corrections
- \(Z\) - at. no.
- \(A\) - absorption
- \(F\) - fluorescence
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Atomic no. correction

Function of electron backscattering factor & electron stopping power - depend upon the average at. nos. of unknown and standard

Varies with composition and accelerating voltage
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Absorption correction

\[ I = I_0 \exp^{-\left(\mu \right) \left(\rho x\right)} = I_0 \exp^{-\left(\mu \right) \left(\rho z \csc \psi\right)} \]
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Absorption correction

\[ A_i = \frac{f(\chi_i)}{f(\chi_i)^*} \]

Absorption function,

\[ f(\chi_i) = \frac{I_i(\text{emitted})}{I_i(\text{generated})} \]

* sample

A varies with \( \mu \), takeoff angle, accelerating voltage

\( A_{\text{NiK}\alpha} \) in Fe-Ni alloy
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Fluorescence correction

electrons $\rightarrow$

primary fluorescent x-rays $\rightarrow$

secondary fluorescent x-rays
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Fluorescence correction

electrons $\rightarrow$

primary fluorescent x-rays $\rightarrow$

secondary fluorescent x-rays

Varies with composition and accelerating voltage

$F_{FeK\alpha}$ in Fe-Ni alloy
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Can't calculate ZAFs unless concns known. Use k values \((I/I^\circ = k)\) to estimate compositions of each element.

Then calculate ZAFs, and refine by iteration.
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Micro scale elemental analysis

Microprobe compositional line analyses for Zr–Ag–Cu and Ti–Ag–Cu on AlN reacted for 60 min at 1000 °C.
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Micro scale elemental analysis
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Micro scale elemental analysis
High strength concrete, Na elemental quantitative compositional map, mosaic combined stage and beam scan, 15 keV