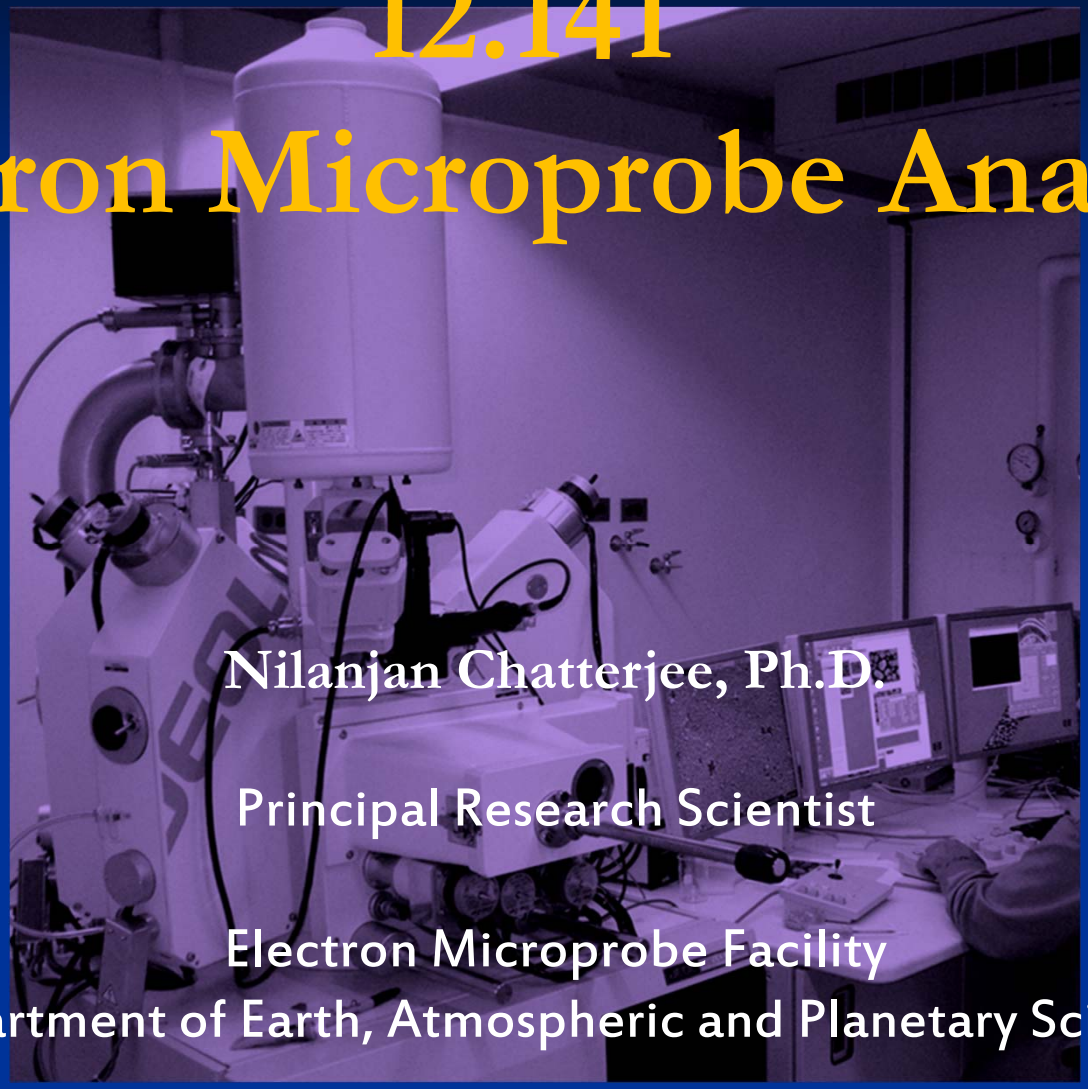
12.141
Electron Microprobe Analysis



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Electron Microprobe
or
Electron Probe Microanalyzer (EPMA)

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

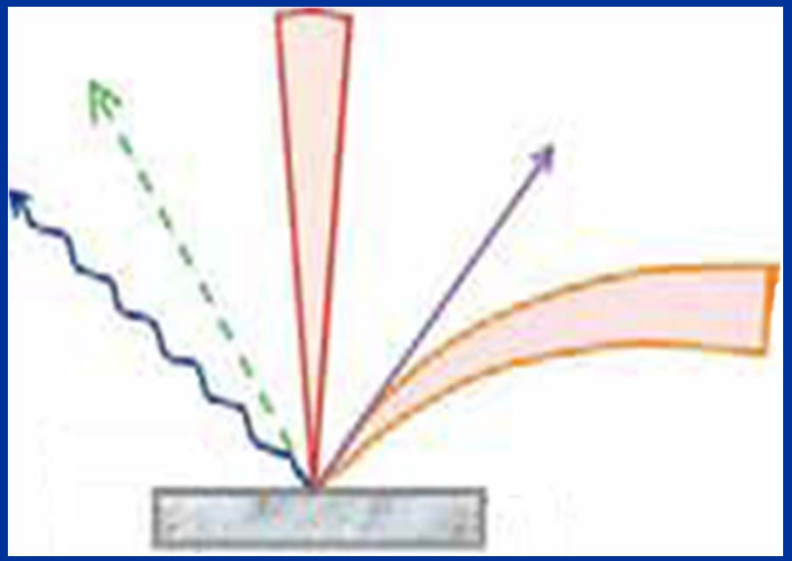
- Surface topography and compositional imaging
- Complete chemical analysis of microscopic volumes (Electron beam induced X-ray emission spectrometry)

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

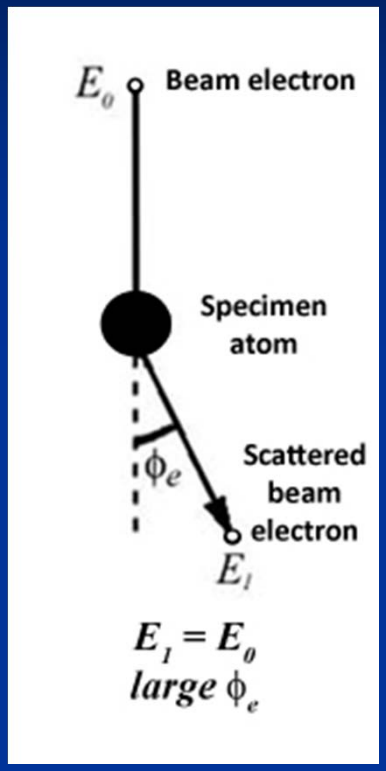
- Full quantitative micro-chemical analysis

  Concentration of all elements present at the spot

- Elemental concentration mapping

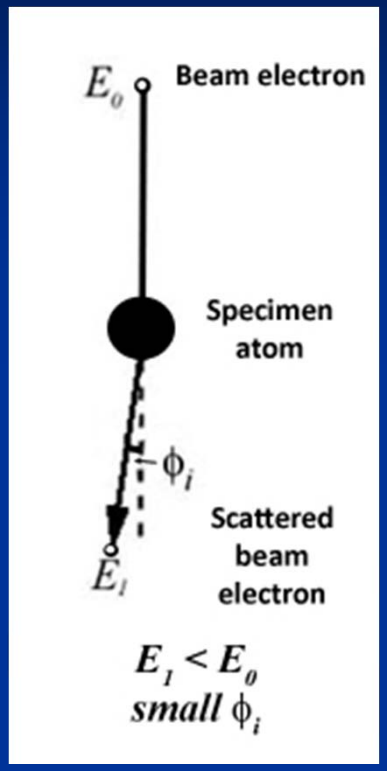
  Concentration of all elements present at each pixel of the image

Electron-specimen interactions



Elastic Scattering

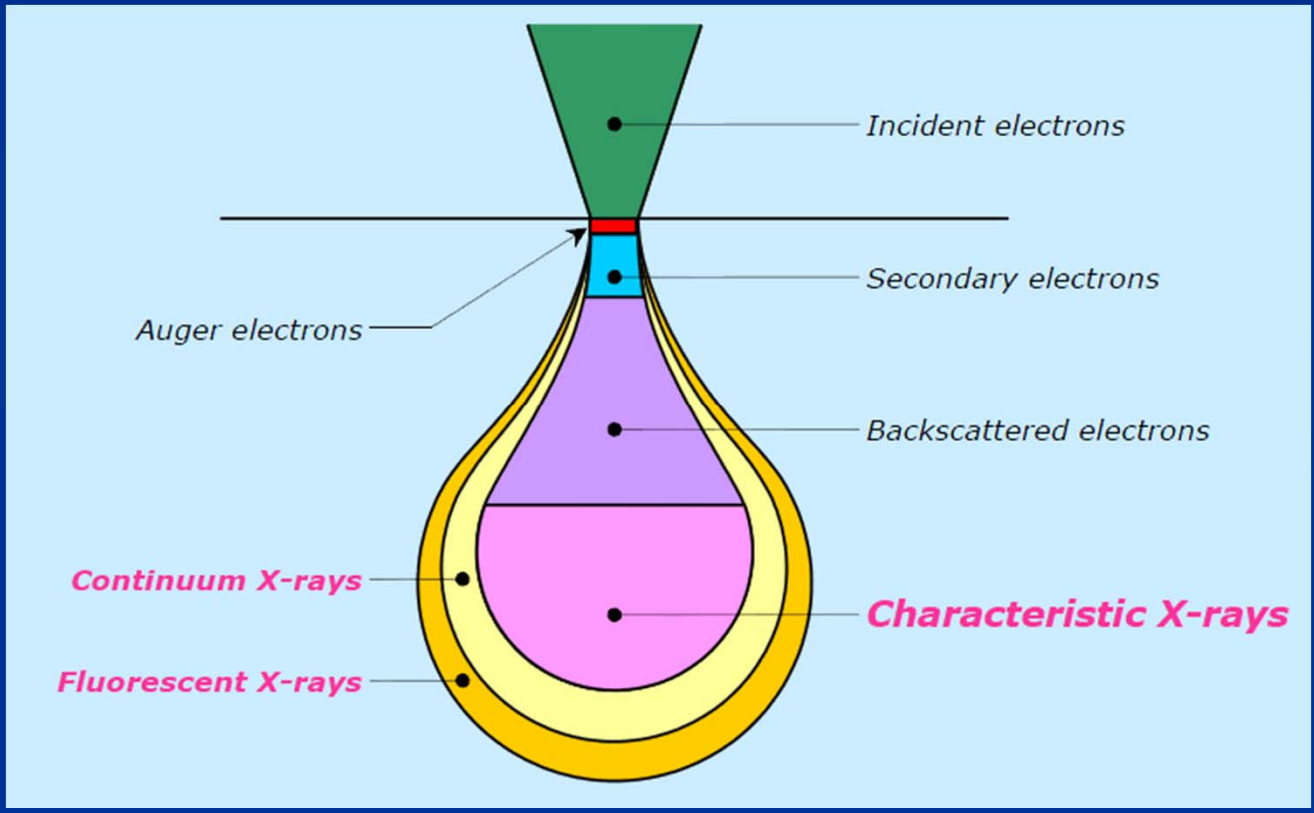
- Back-scattered electron



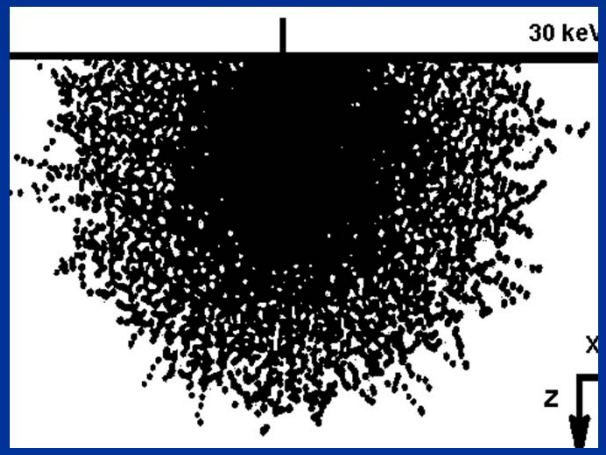
Inelastic Scattering

- Characteristic X-rays
- Secondary electron
- Cathodoluminescence

Electron interaction volume



Electron interaction volume



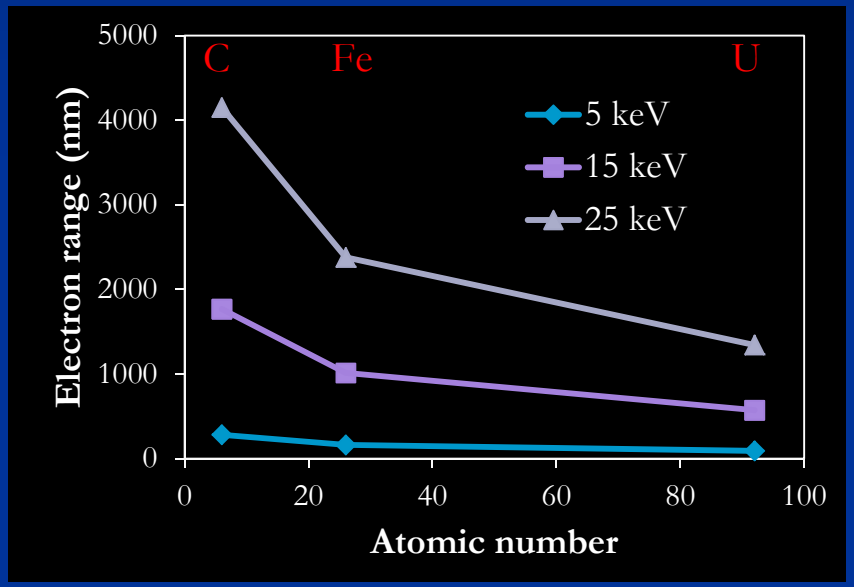
$$R = 0.0276 \, E^{1.67} \frac{A}{\rho \, Z^{0.889}}$$

(A = atomic weight, ρ = density)

(Kanaya-Okayama range)

- Increases with electron beam energy, E
- Decreases with sample atomic number, Z

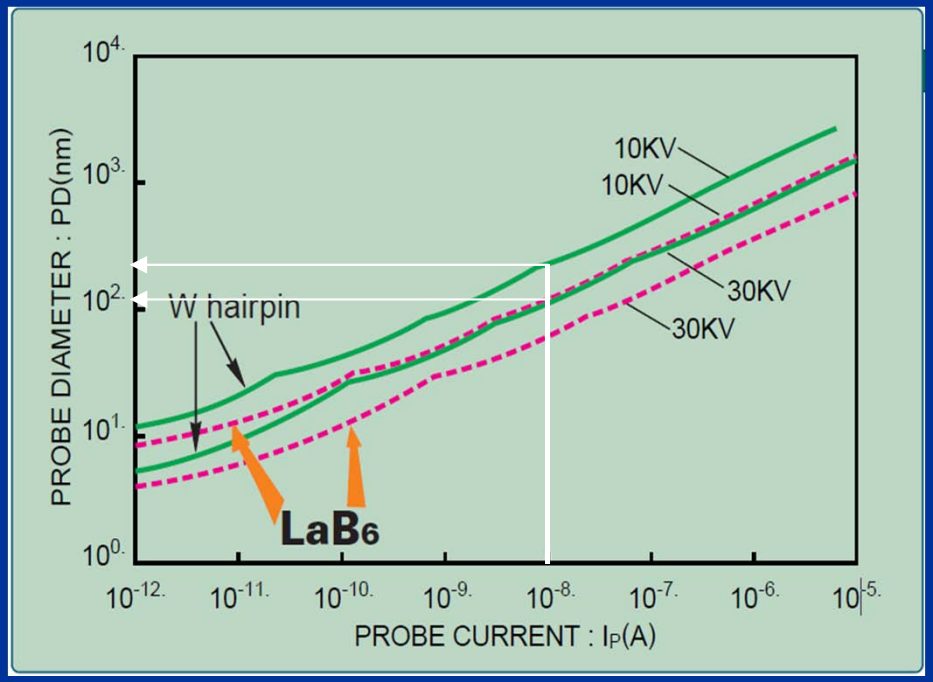
Electron interaction volume

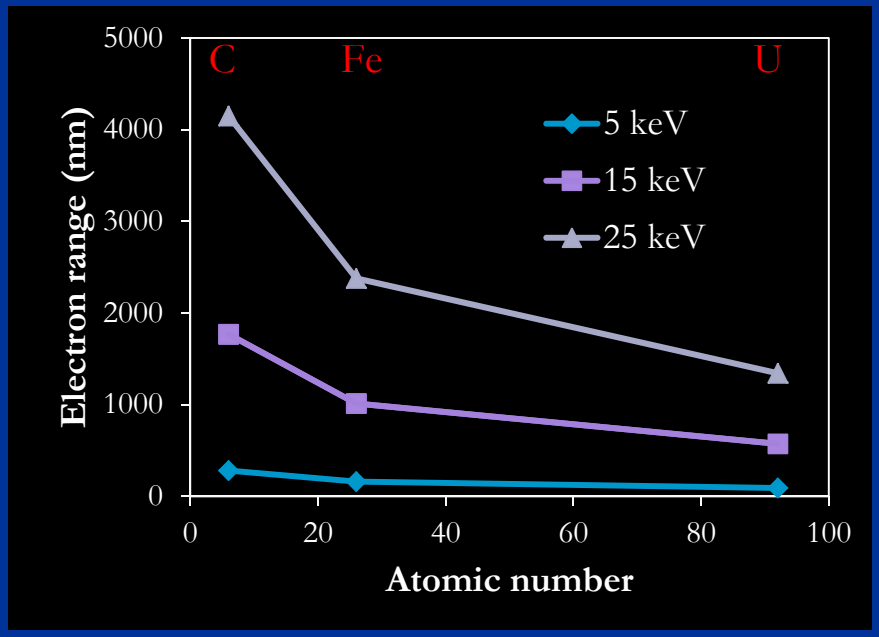


*Typical ranges (*15 kV, *perpendicular beam):*

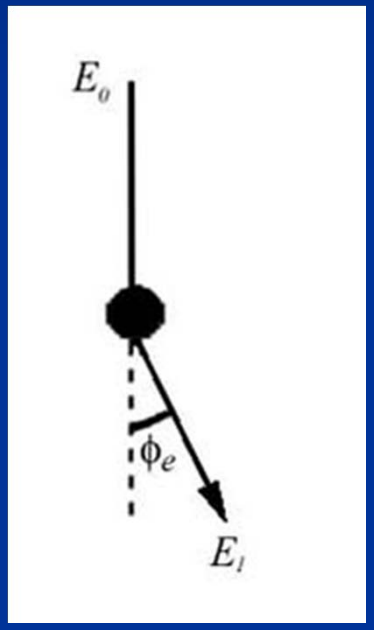
C (Z = 6)	**1.8 µm**
Fe (Z = 26)	**1.1 µm**
U (Z = 92)	**0.8 µm**

Electron probe diameter and Electron interaction volume





Elastic scattering cross-section



$E_1 = E_0$, large ϕ_e

For a scattering angle $>\phi_e$, cross-section (events.cm^2/e$^-$.atom)

$$Q_e = 1.62\text{x}10^{-20} \, (Z^2/E^2)\cot^2(\phi_e/2)$$

Z: atomic number

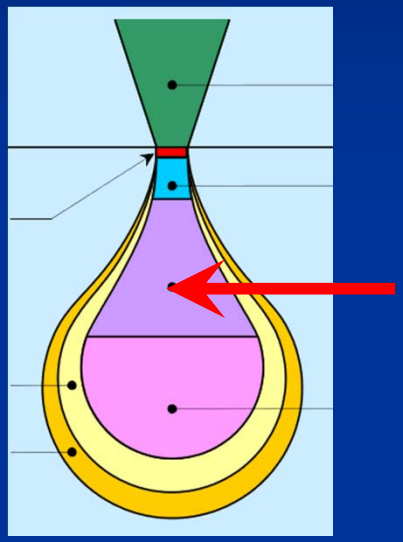
E: beam energy

ϕ_e: elastic scattering angle

- Increases with sample atomic number, Z
- Decreases with electron beam energy, E

Back-scattered electron (BSE)

(Elastic scattering)

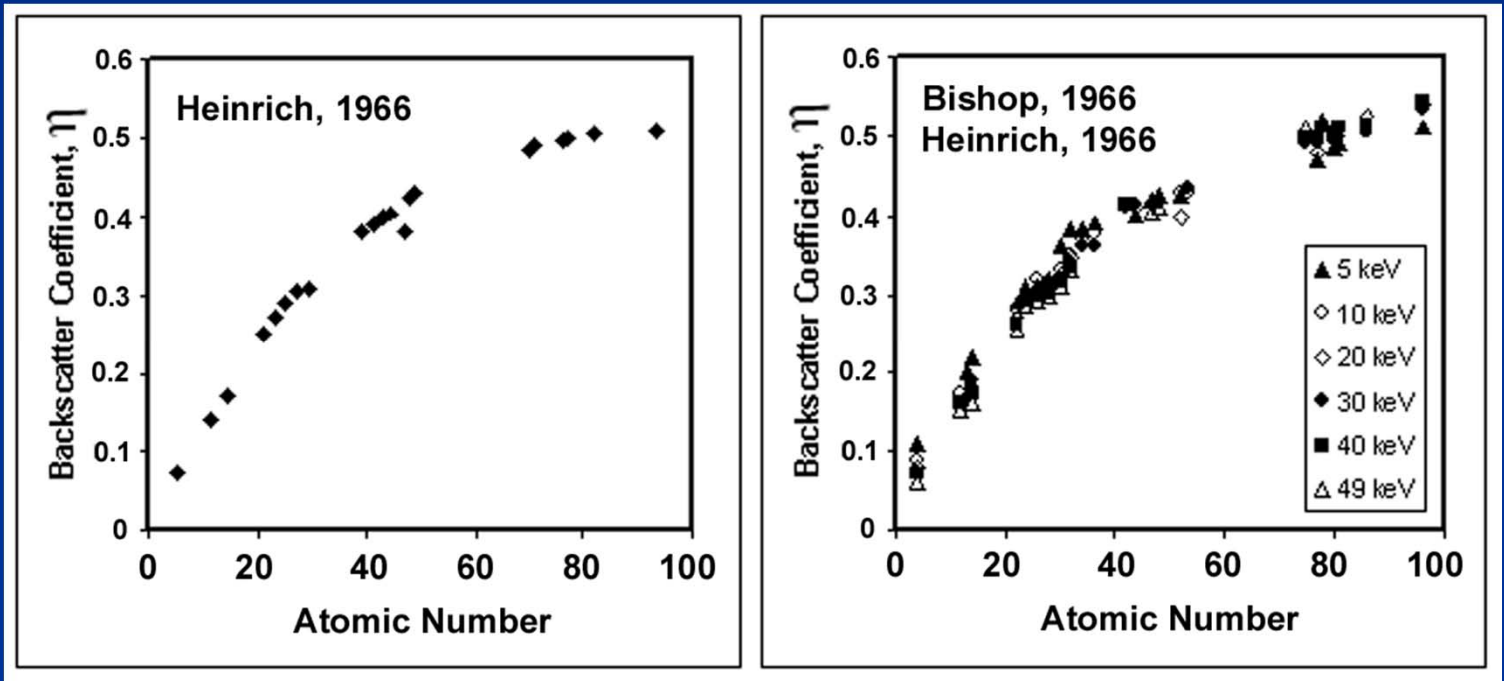


- Beam electrons scattered backward from specimen surface
- High energy electrons with energy about the same as that of the electron beam

- **BSE image resolution improves with shrinking of the electron interaction volume through:**

1. Decrease in beam energy
2. Increase in specimen atomic number

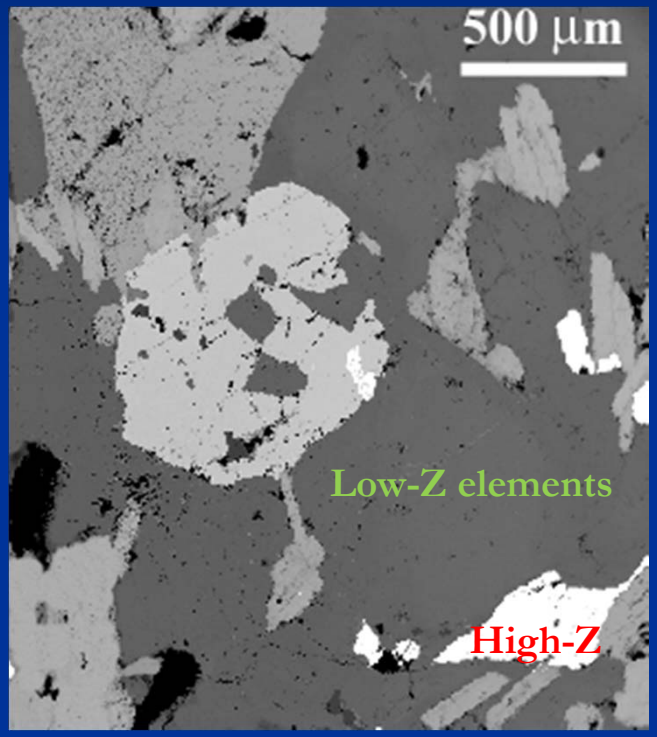
Electron backscatter coefficient



BSE image contrast is better among low Atomic Number elements

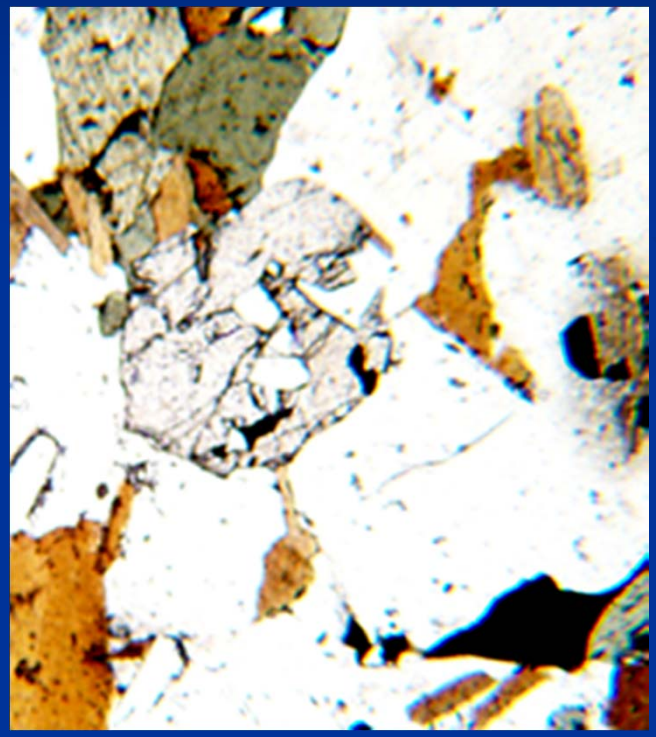
Backscattered electron image

Back-scattered electron



Polished surface

Plane polarized transmitted light

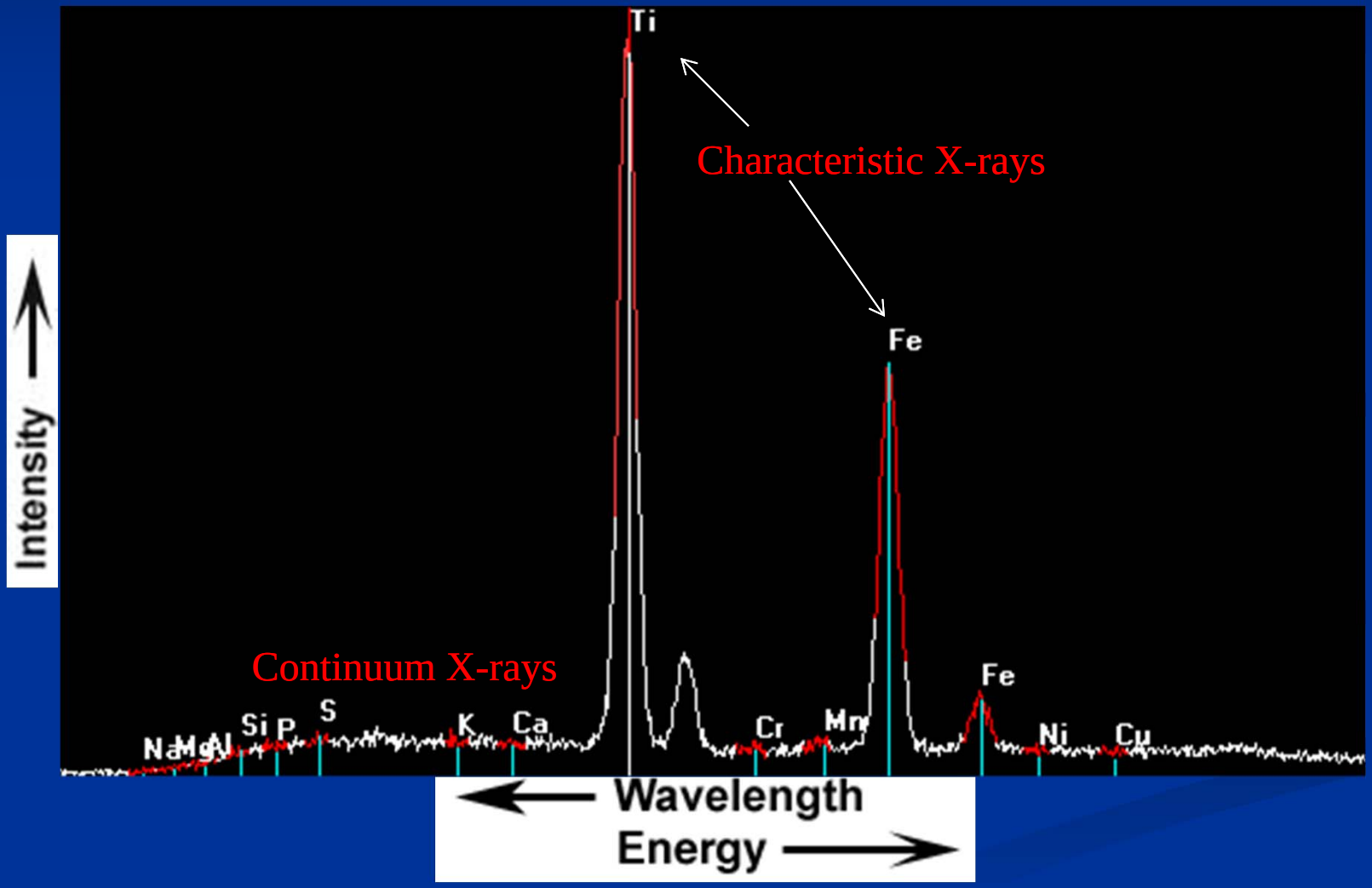


Thin section

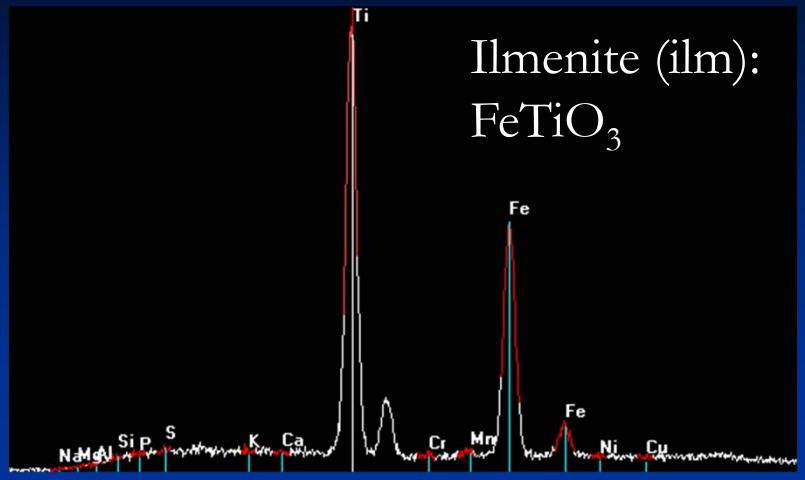
Function of composition

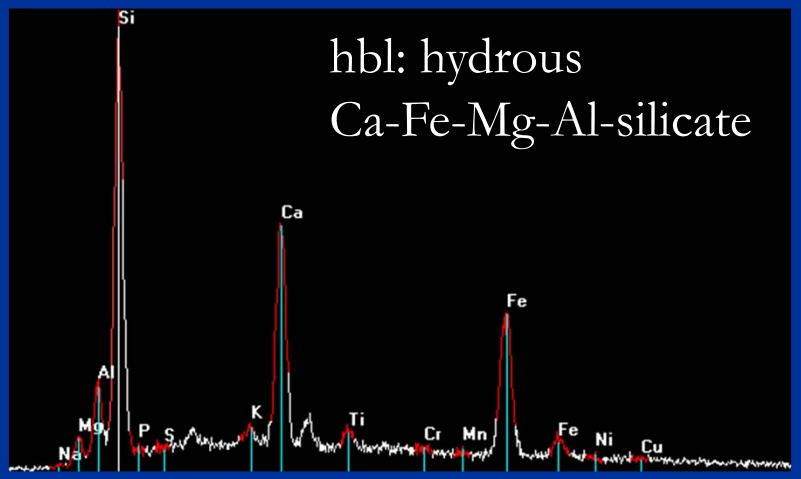
Function of optical properties

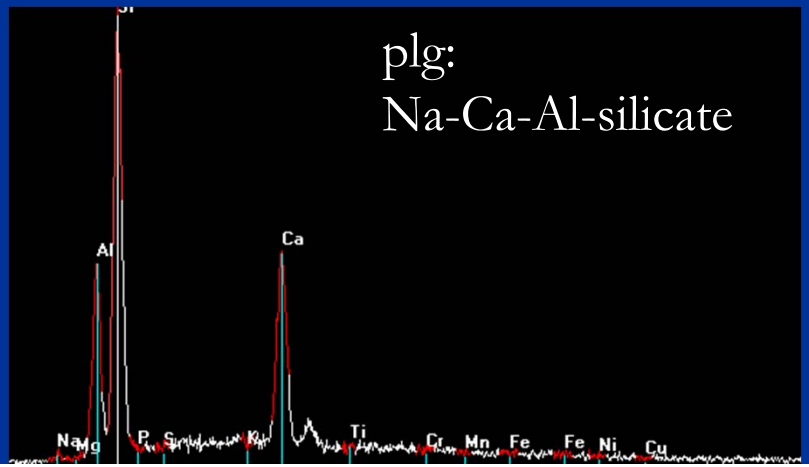
The X-ray spectrum

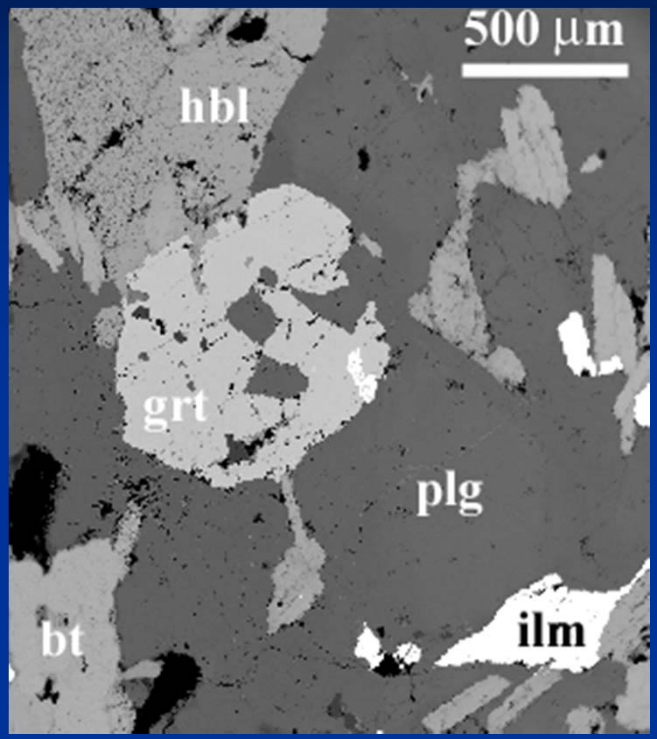


Phase identification: EDS X-ray spectra



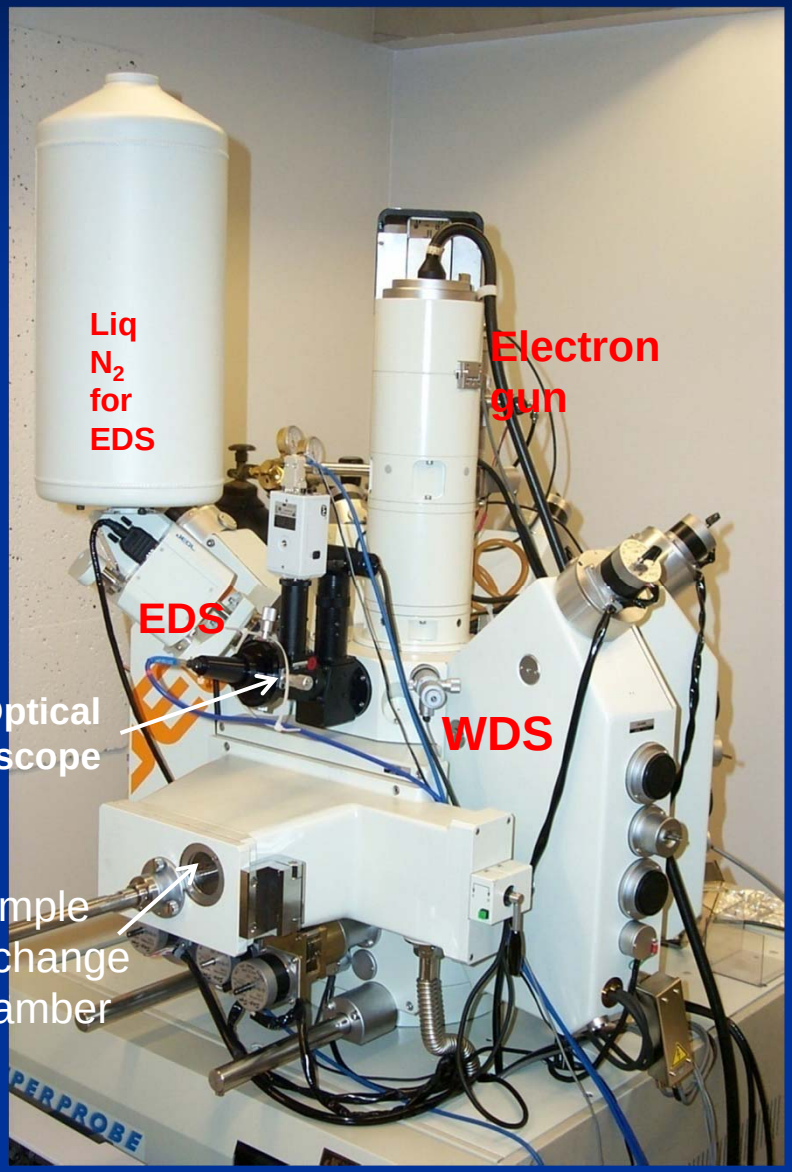


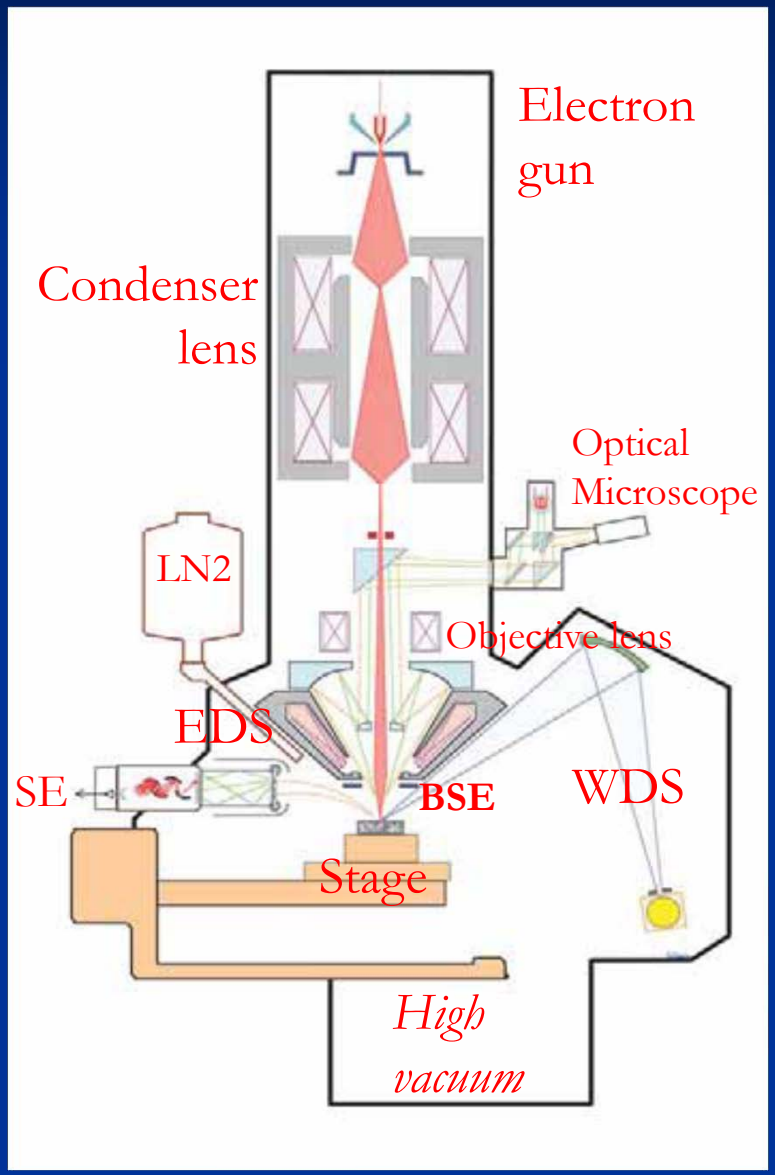




Z	1	8	11	12	13	14	19	20	22	26
ilm:		O							Ti	Fe
grt:		O		Mg	Al	Si		Ca		Fe
bt:	H	O		Mg	Al	Si	K			Fe
hbl:	H	O		Mg	Al	Si		Ca		Fe
plg:		O	Na		Al	Si		Ca		

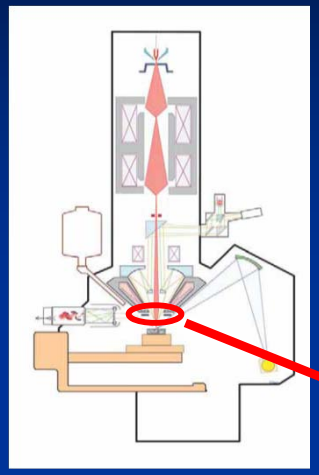
JEOL JXA-8200 Superprobe



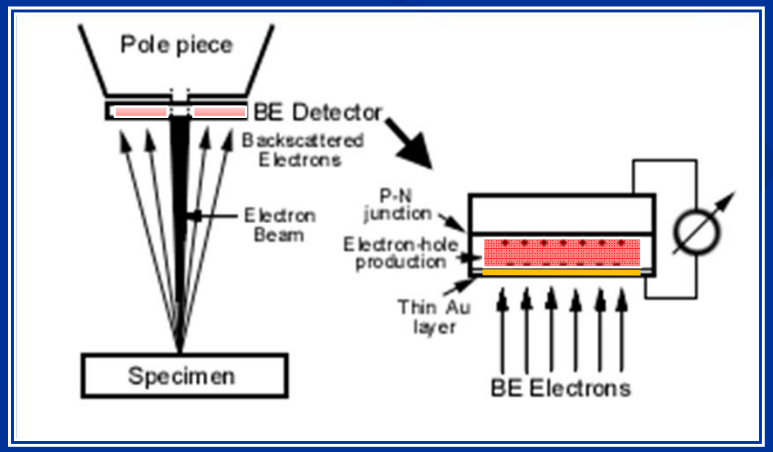


Sample surface is perpendicular to the electron beam

Back-scattered electron detector

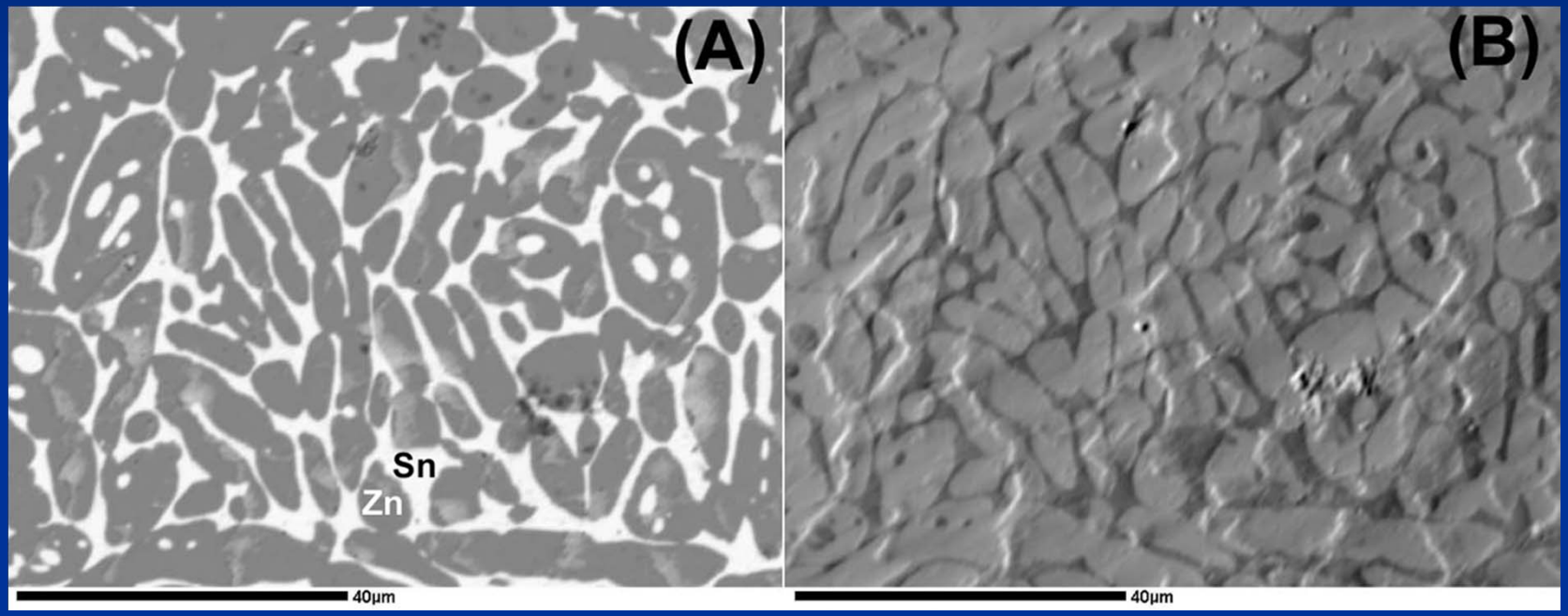


Located vertically above the specimen
A split ring shape



solid-state diode

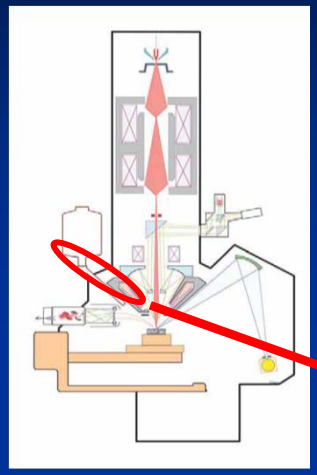
Compositional and topographic imaging with BSE detector

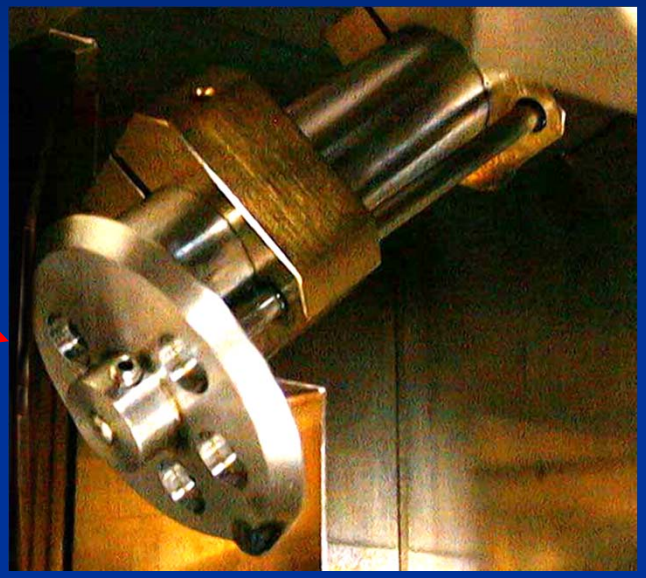


A+B
Compositional
mode

A-B
Topographic
mode

Energy Dispersive X-ray Spectrometer (EDS)

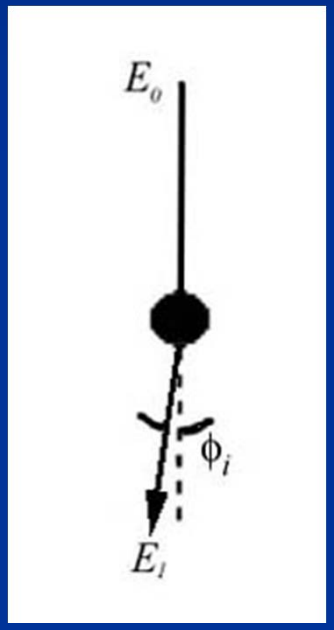




- *EDS detector: a **'p-n'** layer of intrinsic **Si(Li)** semiconductor; Be window; aperture wheel*
- *Multichannel analyzer (MCA) processes the X-ray signal*

Secondary electron (SE)

(Inelastic scattering)

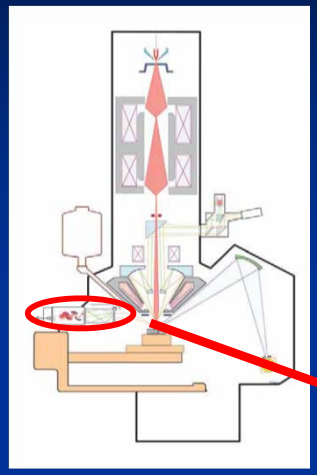


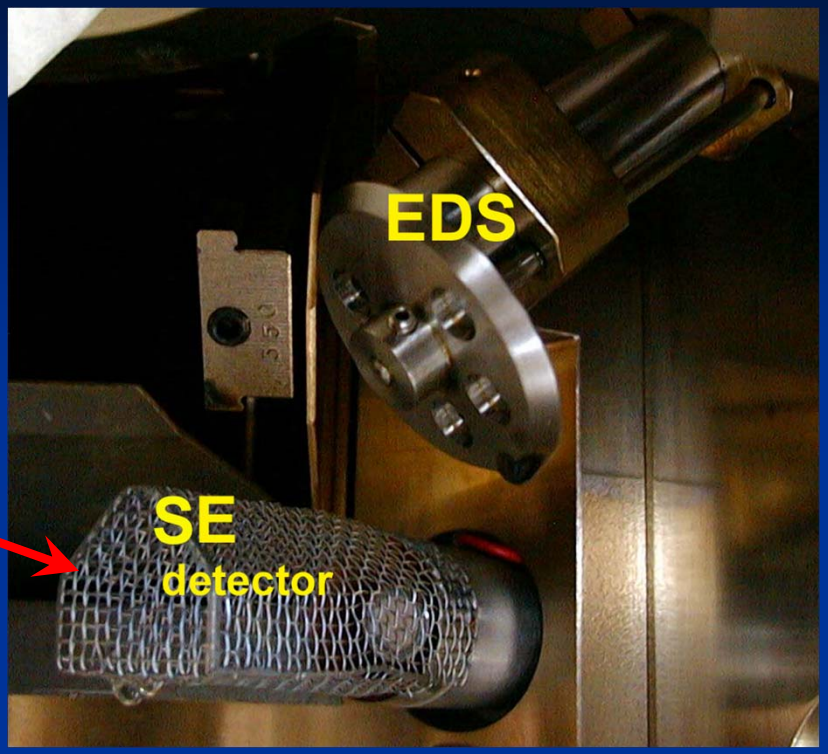
$E_1 < E_0$, small ϕ_i

- Electrons from specimen surface are mobilized by beam electrons
- Emitted at low energies (typical: <10 eV)

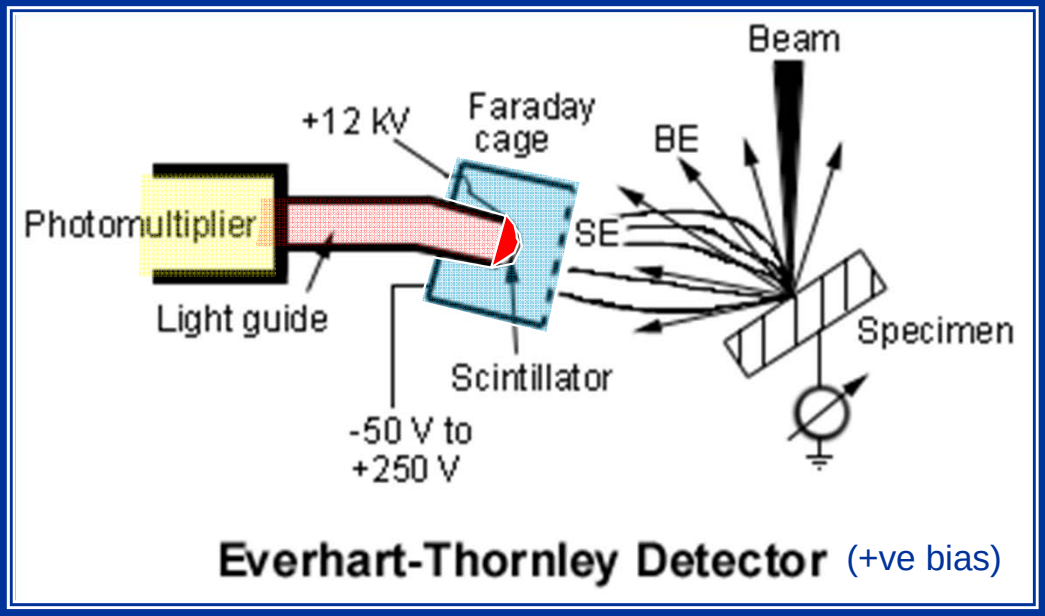
  (recall BSE are high energy beam electrons that underwent elastic scattering, $E_1 = E_0$, E_0 typically being 10-20 keV)

Secondary electron detector

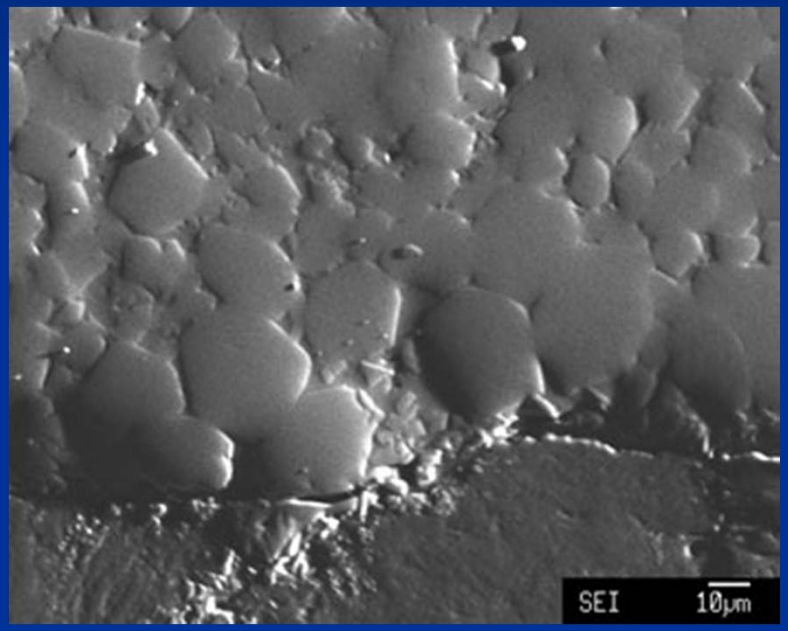




Located on the side wall of the sample chamber



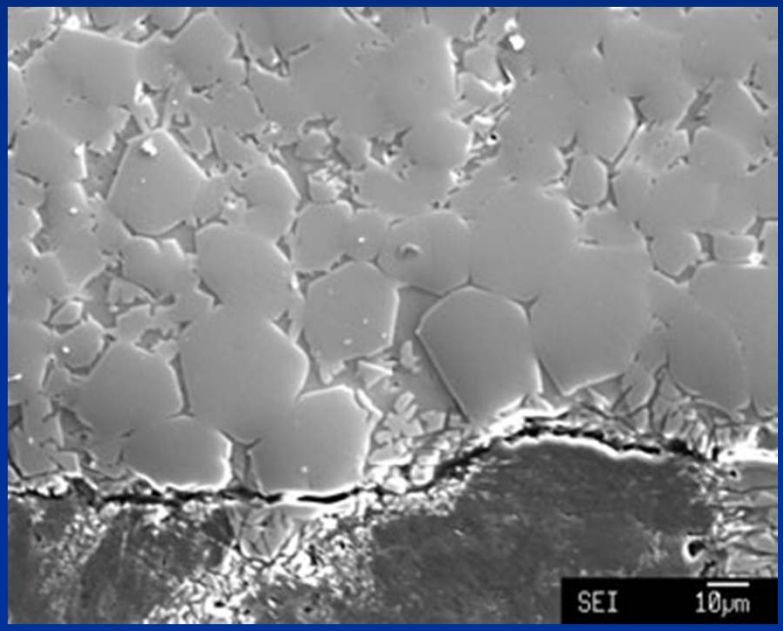
Imaging with the E-T detector



-ve Faraday cage bias

only BSE

Surfaces in direct line of sight are illuminated



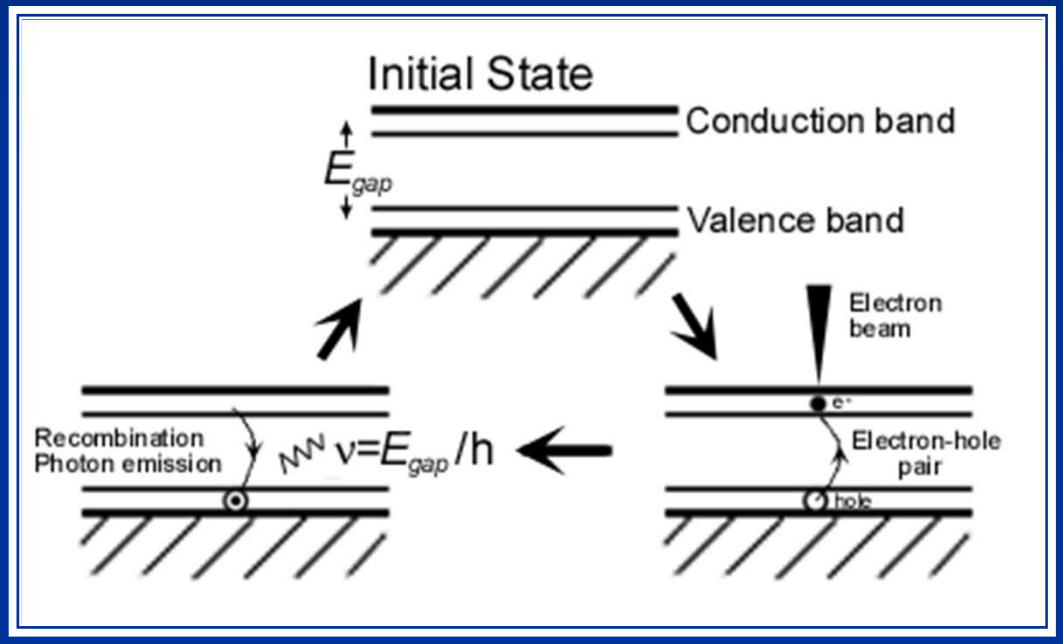
+ve Faraday cage bias

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*

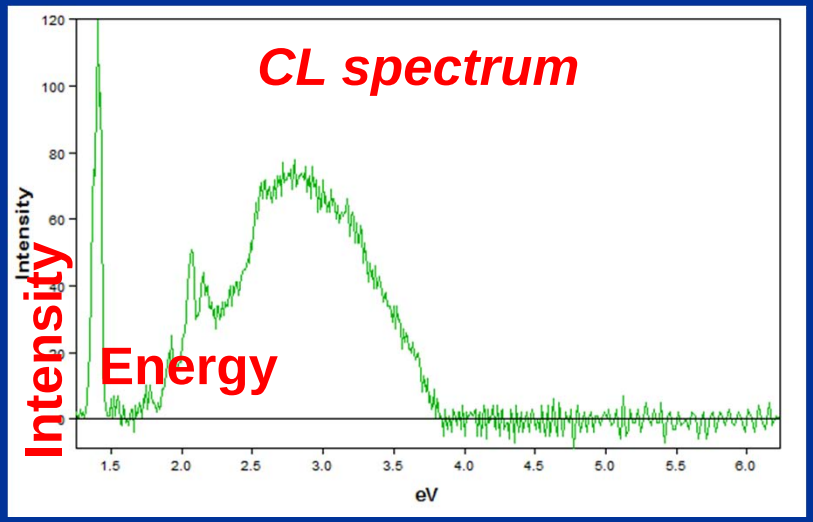
Cathodoluminescence spectrometer

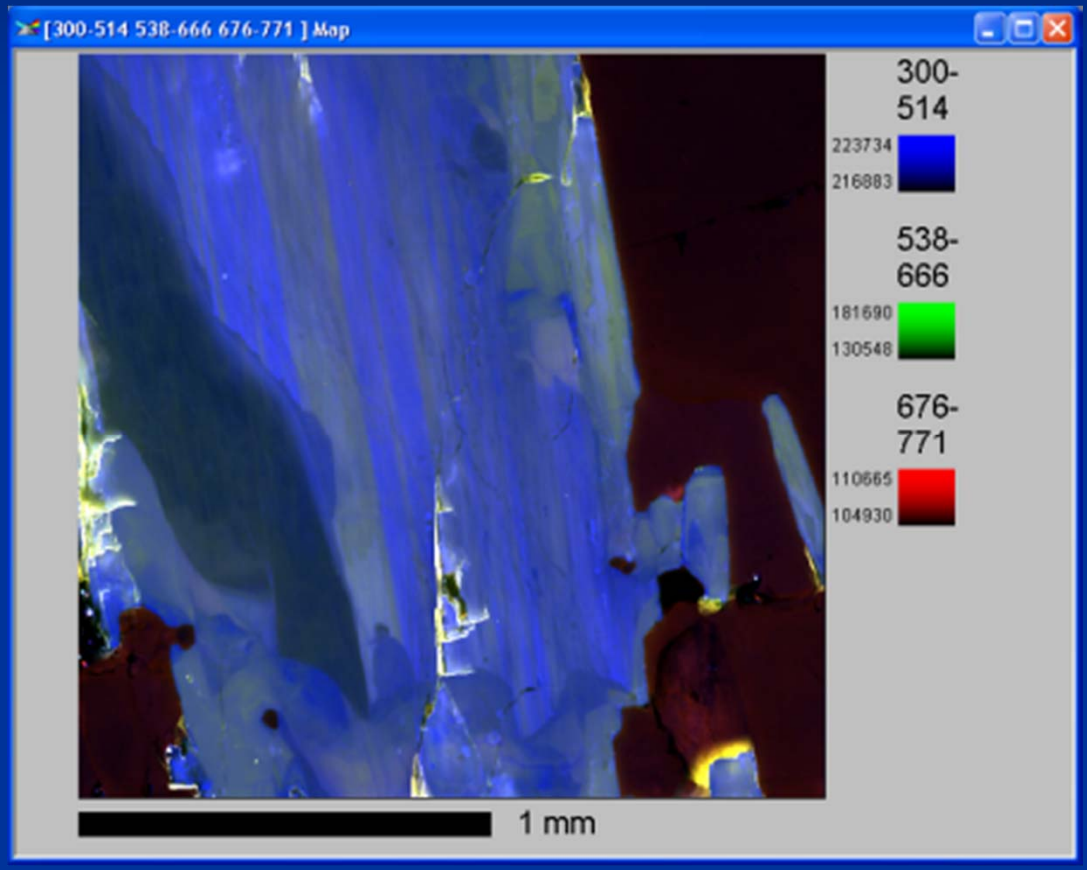
Optical microscope camera (not used)

Optical microscope light (turned off)

Optical spectrometer

Cathodoluminescence spectrometry





12.141 Electron Microprobe Analysis

January (IAP) 2012