CHEM-E5140
Materials Characterization Laboratory

Scanning electron microscope (SEM) Lecture
31.3.2025

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Outline

1. Introduction
2. How do I get the data?
   – Group discussion
3. What can I analyze with SEM?
4. What kind of samples can be studied?
5. Case examples

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1. Introduction

- Scanning electron microscope (SEM) is an instrument that has resolution and depth of focus superior to basic optical microscopes.
- Furthermore, electron probe microanalysis (EPMA), electron backscatterin diffraction (EBSD) and some other studies can be carried out with SEM that have additional instrumentation.

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Why microscopes?

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There is no scale symmetry in nature

1000x

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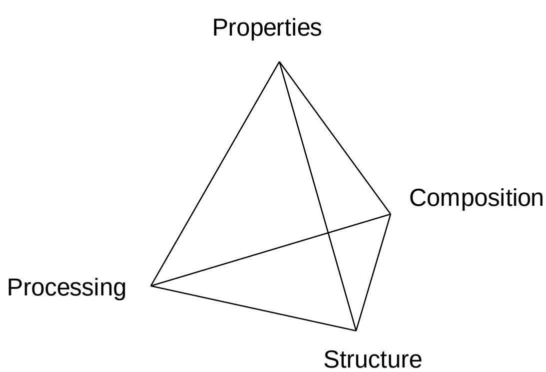
Materials science tetrahedron



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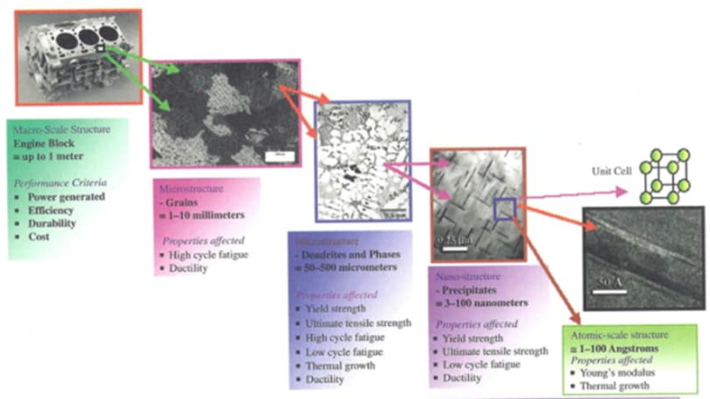
Structural hierarchy in materials



Materials characterization techniques

	Magnification	Resolution	Depth of field	Sample	Other
Basic optical microscope	10-1000x	1-<u>0,2</u> μm	2-0,2 μm	Flat (polished, etched)	Inexpensive, Reflectivity, FTIR, Raman
Scanning electron microscope	10-200000x	1-100nm	1 mm – 0,1 mm	Usually electrically conductive	**Vacuum,** EDS, WDS, EBSD, CL, EBIC
Transmission electron microscope	>600000x	0,15-0,3 nm	n. 20 nm	Very thin	**Vacuum,** Diffraction, EDS, EELS

Other methods: **XPS (ESCA)**, AES, ...
XRD, XRR,
XRF, **Raman**, AAS, SIMS,PIXE, ...
AFM, STM, μXCT ...

Microscopes

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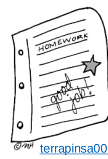
2. How do I get the data?

- How scanning electron microscope works?
- Physical background of the method
- Equipment technology

Pretask:

How to prepare



terrapinsa005.weebly.com

- Prepare 4-6 slides
  - What information the method provides and how does it work?
  - What kind of samples can be analysed?
  - Is the method destructive for the sample?
  - Your picture of the operating mechanism of the device (drawn with hand or by yourself with computer)

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Group discussion

- How scanning electron microscope works?

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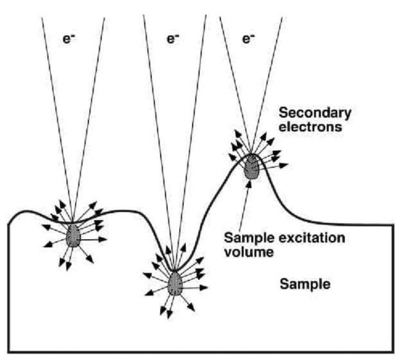
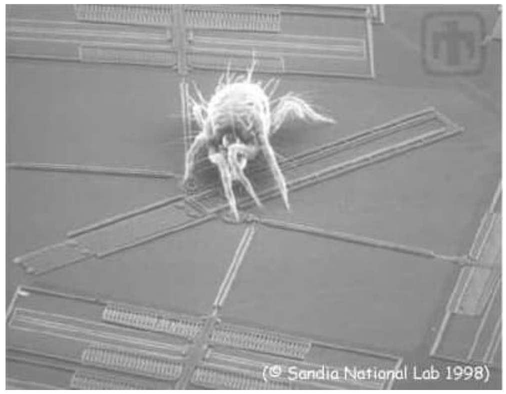
SEM operation principle

- Based on focused electron beam that interacts with the specimen
- Electron source and sample are in vacuum
- Information is collected point by point

Effect of topography on secondary electron emission



Contrast in SE-electron image



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Physical background of the method

- Electrons interact with atoms in the sample, producing various signals that can be detected and that contain information about ***surface topography***, ***local composition*** and ***crystallography*** of the sample as well as some other information in specific cases.

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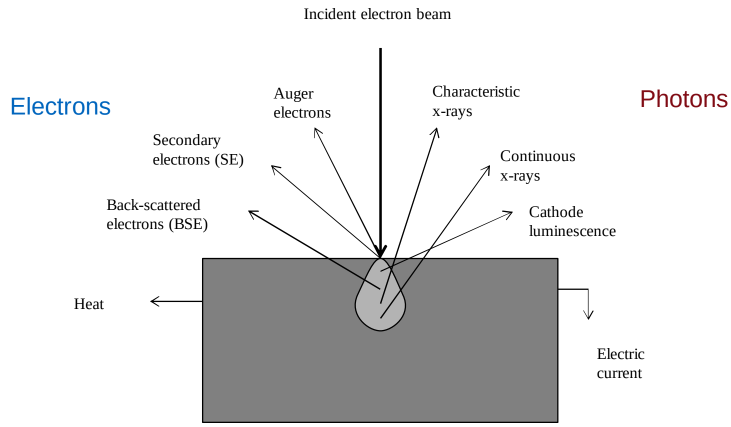
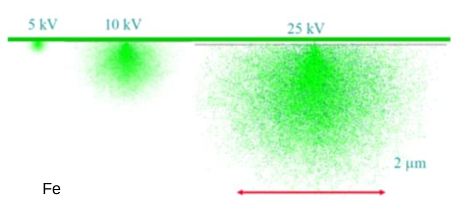
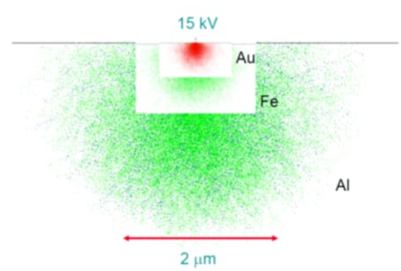
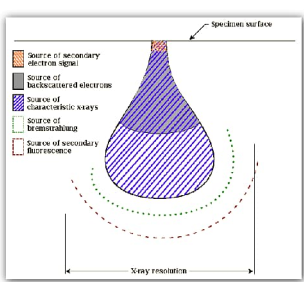
Electron beam specimen interactions



Interaction volume







Basic signals for SEM imaging

- Secondary electrons (SE)
- Backscattered electrons (BSE)

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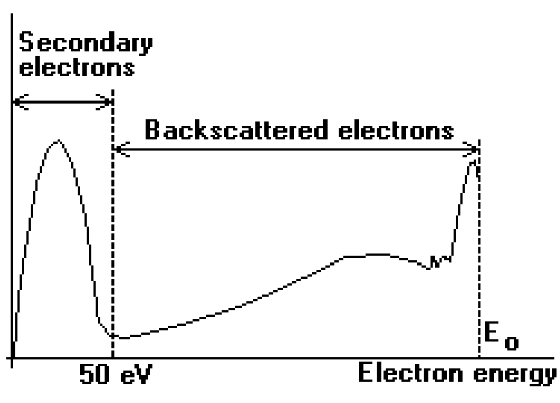
Energy distribution of emitted electrons



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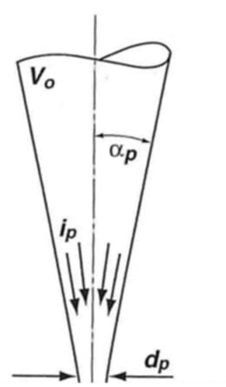
Equipment technology

- Vacuum system
- **Electron optical system**
- **Detection system**
- Specimen handling system
- Computer control and display system

Basic Operation Modes

- Resolution mode
- Depth mode
- High current mode
- Low acceleration voltage mode



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3. What can I analyze with SEM?

- Imaging of structural features and surface topography
  - high resolution
  - large depth of field
- Analysis of local chemical composition tentatively

- With additional hardware
  - Analysis of local composition qualitatively and quantitatively
  - Analysis of local crystallography
  - some other information in specific cases

Microscope performance

- Magnification
- Resolution
- Depth of field
- Contrast

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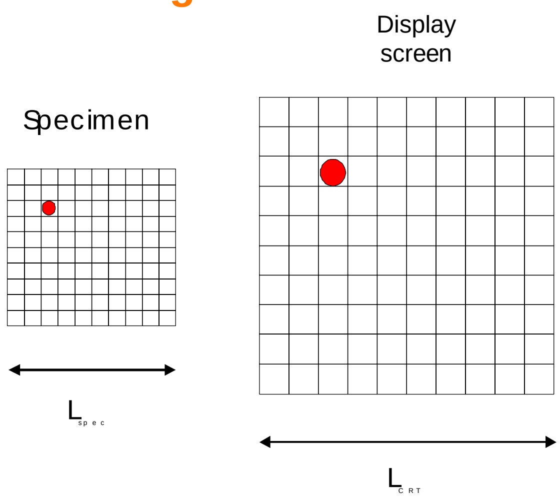
Rastering



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Definition of magnification

- Screen area is fixed
- Magnification is a function of the sample area that is scanned

$$M = L_{screen}/L_{sample}$$

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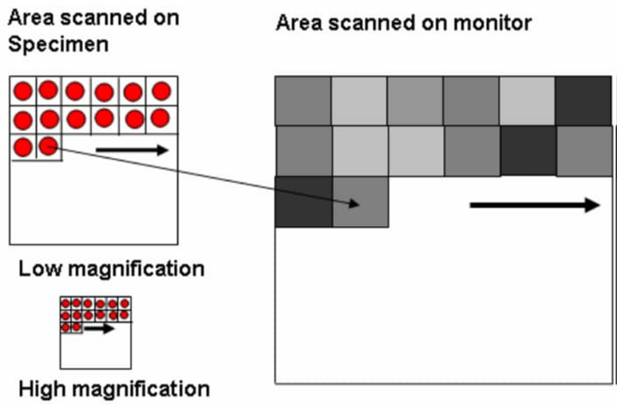
Image formation



8-bit gray levels: Black = 0, White = 255

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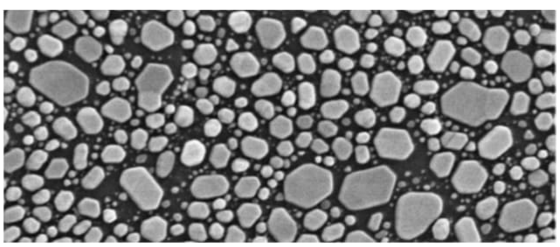
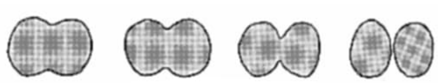
Resolution





Resolution R is in practice minimum resolvable distance in the object

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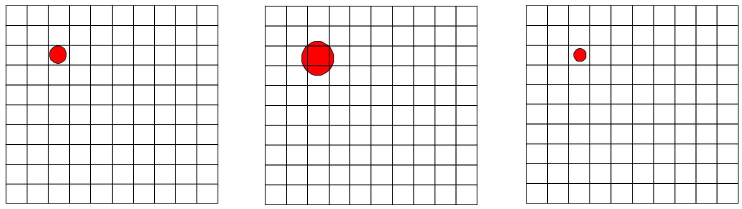
Optimum resolution



optimal

empty
resolution

poor S/N
ration

Empty magnification

Empty magnification

2x

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Empty magnification

4x

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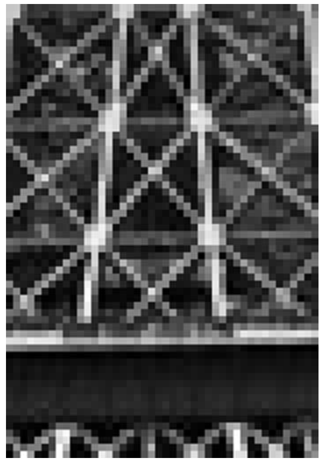
Empty magnification



8x

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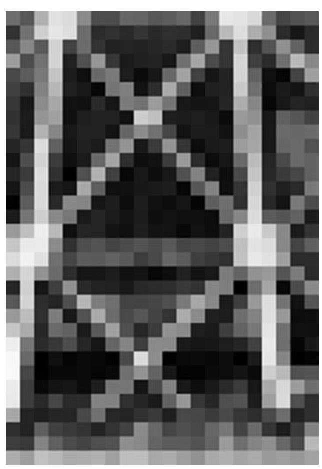
Empty magnification



16x

Useful magnification is related to resolution

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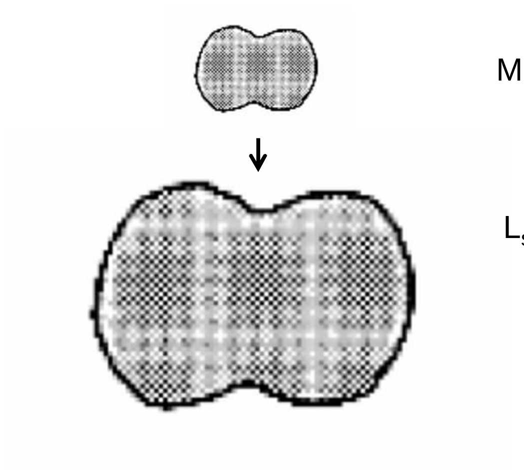
Empty magnification



$M = R_{eye}/R_{microscope}$

if

$L_{screen}/L_{sample} > R_{eye}/R_{microscope}$

Making image larger
does not increase
Information

($R_{eye} \approx 0{,}2$ mm)

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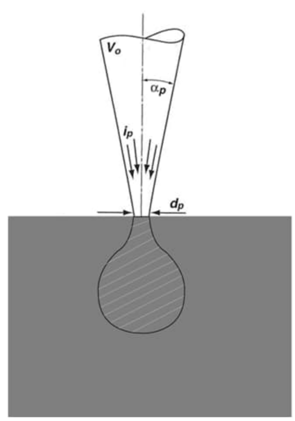
Factors effecting resolution



- "Spot size" d_p
- Interaction volume
- Contrast

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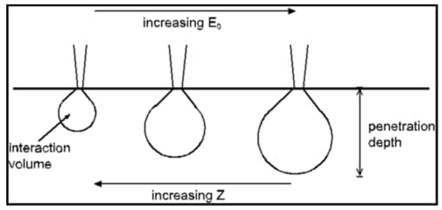
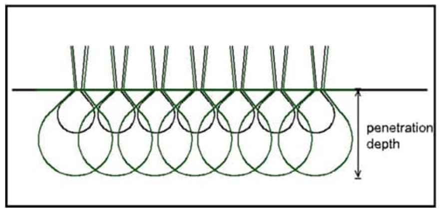
Resolution and interaction volume





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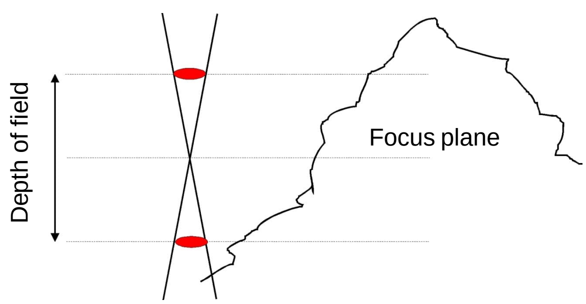
Depth of field



Depth of field is defined as the distance along the optical axis within witch a point in the object is is not spread by the microscope into a spot larger than the resolution limit

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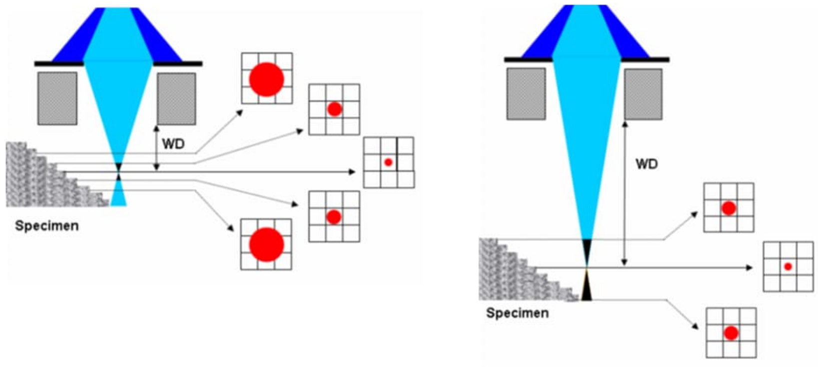
Depth of field



Analysis of local chemical composition tentatively

- Back-scattered electrons
- Back-scattered electron yield is a function on atomic number

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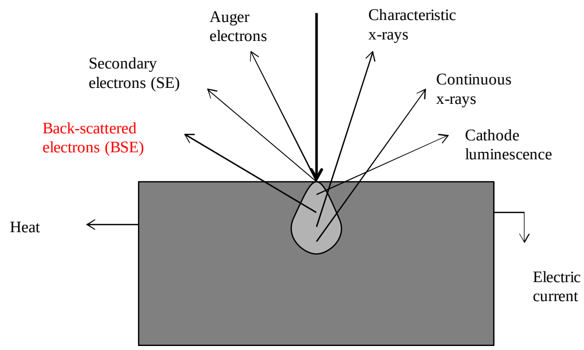
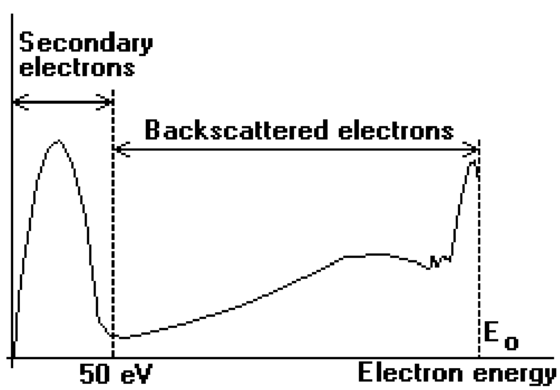
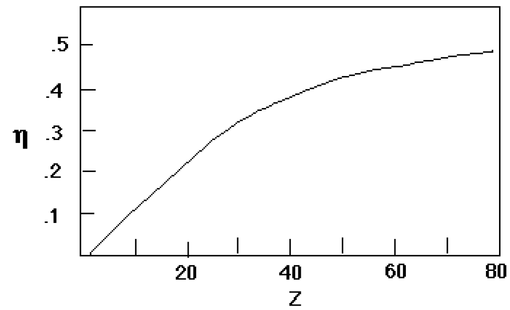
Electron beam specimen interactions



Energy distribution of emitted electrons



Backscattered electron yield as a function of atomic number



η = backscattering coefficient

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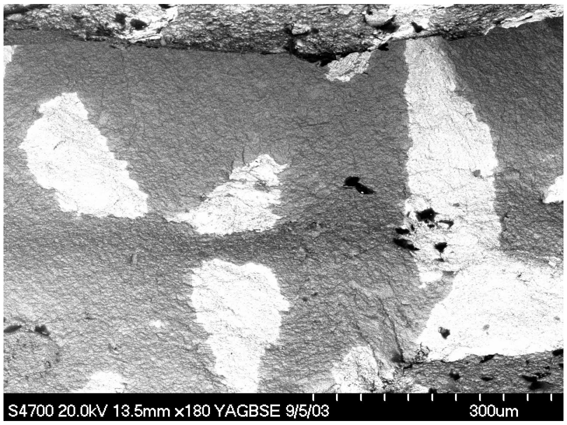
BSE image from a metal matrix composite



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Overview of instrument capabilities

- High magnification
- Large depth of field

- Chemical information in micrometer scale (BSE, EDS, WDS)
- Crystallographic information (EBSD)

More than just a microscope

- Special techniques (EBIC, CL, voltage contrast)
- In-situ experiments (temperature, strain, etc.)

More than just composition and structure

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4. What kind of samples can be studied?

- Basic requirements for suitable samples
- Sample preparation

Basic requirements for suitable samples

- Solid samples
- Small enough
- Dry
- No other volatile components in vacuum either
- Stable under electron beam
- No loose particles, especially magnetic
- Preferably non-magnetic

- Should conduct electricity after sample praparation as a main rule

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Sample preparation

- Many topographic samples can be examined with virtually no sample preparation
- Sample cleaning and de-greasing
- Coatings for electric conductivity
- Flat polished samples are needed for analytical work
  - Similar preparation as in optical microscopy (usually without etching)
  - EBSD samples need wery careful preparation
- Special preparation techniques for biological samples

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Materialographic sample preparation

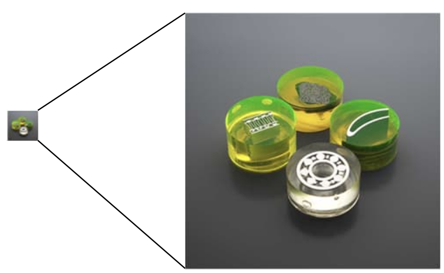
- Sectioning
- (Mounting if needed)
- Grinding
- Polishing
- (Coating if needed)
- (Sometimes etching)

- Several cleaning steps in between

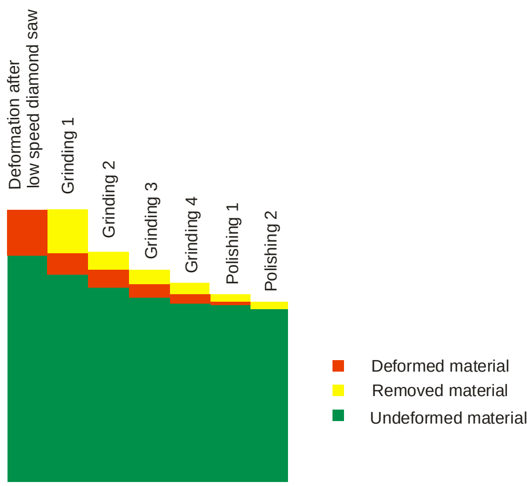
Materialographic sample preparation equipment







Deformation is removed step-by-step



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Ion beam milling

- Ion beam milling can be used as a final polishing step in SEM sample preparation
- In ion beam milling inert gas, typically argon, is ionized and then accelerated toward the sample.
- By means of momentum transfer, the impinging ions sputter material at a controlled rate.
- Focused ion beam (FIB) equipment uses heavier ions (Ga). They can be used for targeted precission sample cutting

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Specimen coating

- Purpose of coating:
  - To make the specimen surface conductive (prevention of charging
  - To increase SE electron yield
  - To prevent beam damage
  - To attach loose particles
- Minimum coating thickness to obtain required information should be applied
- It is necessary to prepare a thin film
- Typically:
  - 0,5-0,8 nm for high resolution
  - 2-4 nm for medium resolution
  - 3-5 nm for routine work

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Coating methods

- Sputtering
  - Au, Au/Pd
  - Thin
  - For imaging
- Evaporation
  - Carbon
  - For EDS/WDS

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5. Case examples

Typical application examples of SEM/EPMA in materials science and engineering

- Structural examination of microstructural features of metallographically prepared samples at **high magnifications**
- Identification and quantification of **local elemental composition** of features down to micrometer sizes from flat polished samples
- Evaluation of **crystallographic orientation** of microstructural features from flat polished sample
- Structural and compositional examination of cross-sectional samples of coatings and interfaces including gradients
- Examination of topography and qualitative local composition of unprepared surfaces such as fracture surfaces, wear tracks and deeply etched surfaces with **high depth of field**
- Morphological analysis of particles, fibres and porous structures
- Examination of semiconductor and micromechanical devices for failure analysis, functional control and design verification
- In-situ experiments (temperature, voltage, strain, etc.)

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Quantitative image analysis

- Image aquisition
- Image processing
- Feature extraction
- Representation of microstructural geometry
  - Features: volume, surface area, size, shape, orientation etc.
  - How much?
  - Distribution
  - Clustering correlations

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Thank you for your attention

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