

CHEM-E5140 Materials Characterization Laboratory

Scanning electron microscope (SEM) Lecture
12.10.2020

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Outline

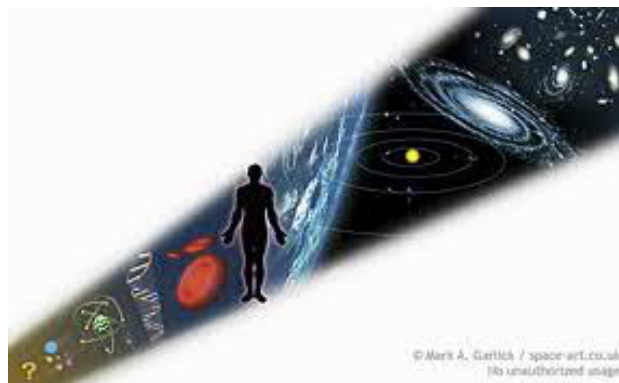
1. Introduction
2. How do I get the data?
 - Group discussion
3. What can I analyze with SEM?
4. What kind of samples can be studied?
5. Case examples

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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.

Why microscopes?



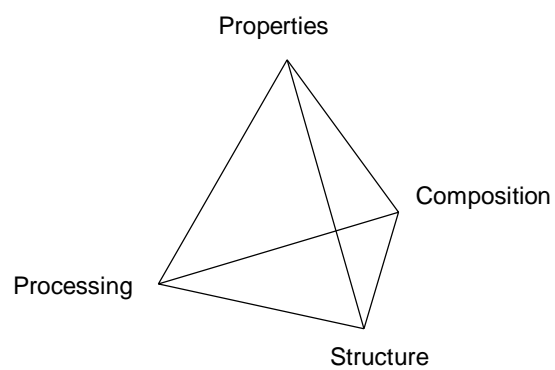
There is no scale symmetry in nature



1000x

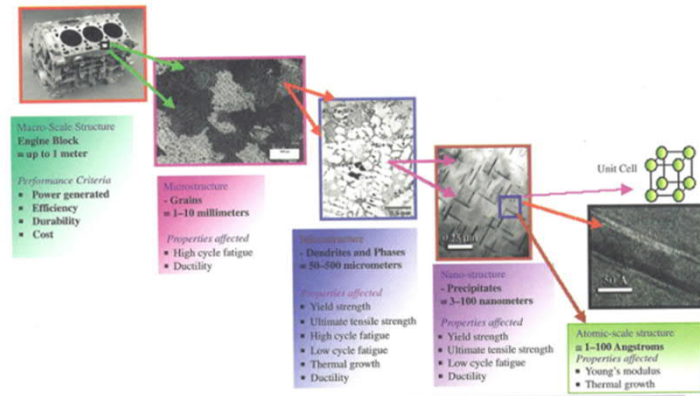
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Materials science tetrahedron



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Structural hierarchy in materials



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Materials characterization techniques

	Magnification	Resolution	Depth of field	Sample	Other
Basic optical microscope	10-1000x	1-0,2 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 ...

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2. How do I get the data?

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

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Microscopes



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Pretask: How to prepare

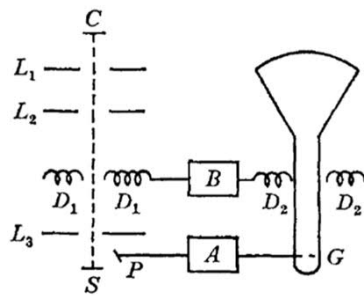


terrapsina005.weebly.com

- 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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Image formation in scanning electron microscope



C.W. Oatley



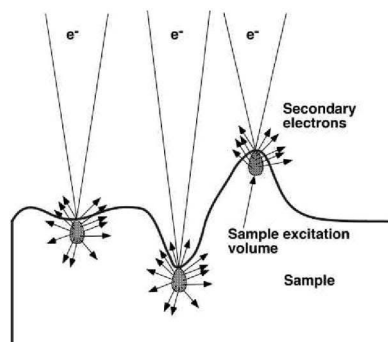
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SEM operation principle

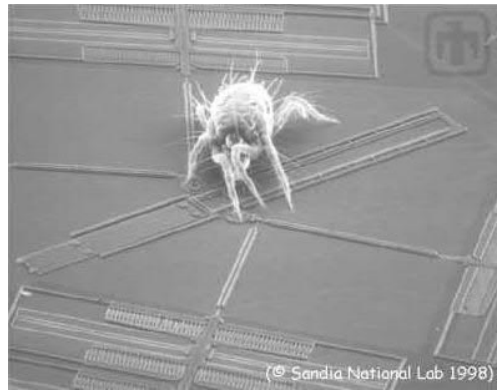


- Based on focused electron beam that interacts with the specimen
- Electron source and sample are in vacuum
- Information is collected point by point

Effect of topography on secondary electron emission



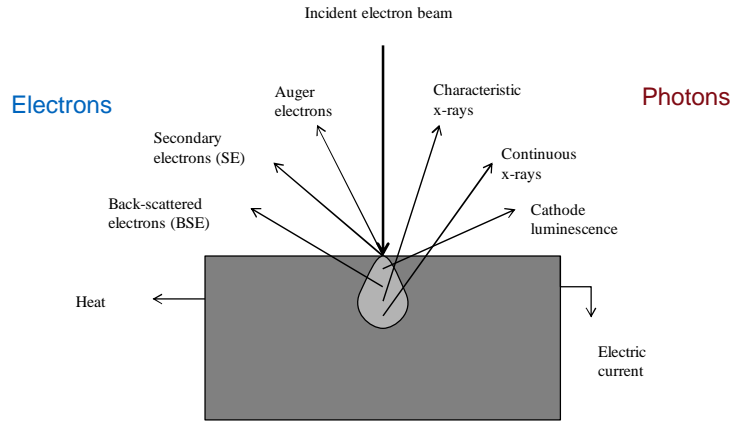
Contrast in SE-electron image



Physical background of the method

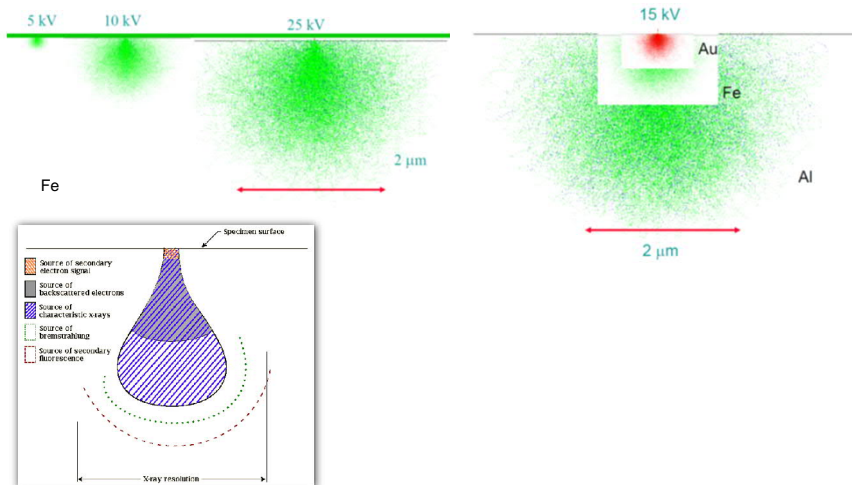
- Electrons interact with atoms in the sample, producing various signals that can be detected and that contain information about **surface topography**, **local composition** and **crystallography** of the sample as well as some other information in specific cases.

Electron beam specimen interactions



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Interaction volume

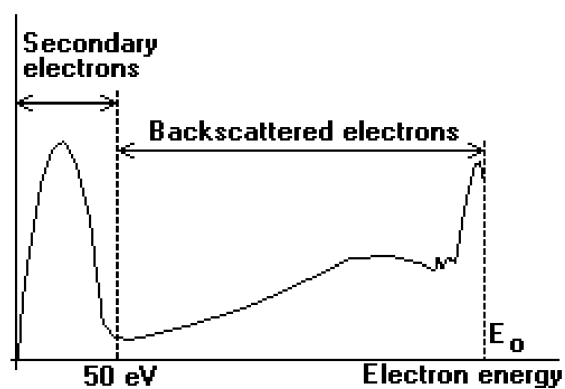


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Basic signals for SEM imaging

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

Energy distribution of emitted electrons



Equipment technology

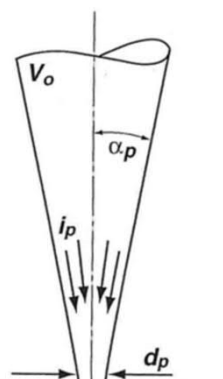
- Vacuum system
- **Electron optical system**
- **Detection system**
- Specimen handling system
- Computer control and display system



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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

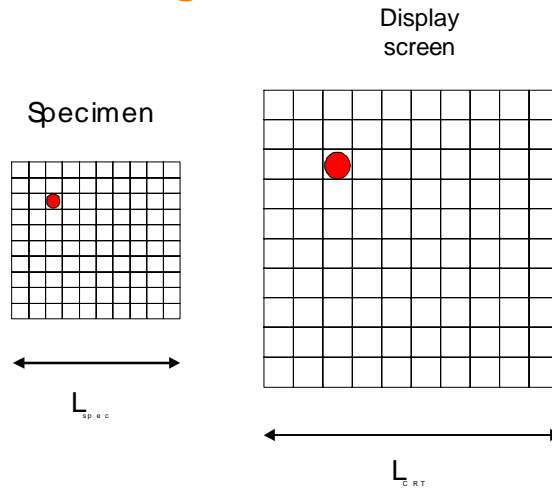
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Microscope performance

- Magnification
- Resolution
- Depth of field

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Rastering



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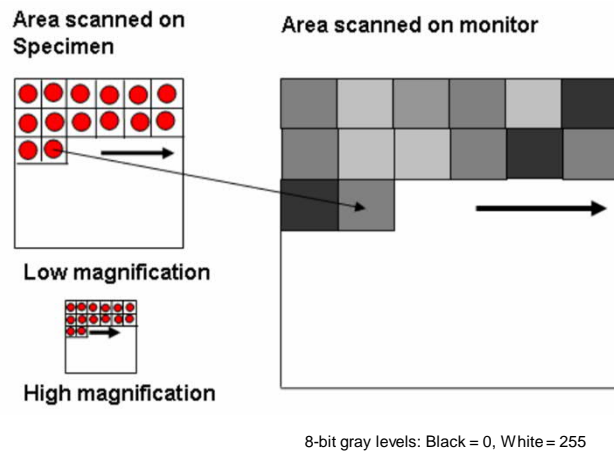
Definition of magnification

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

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

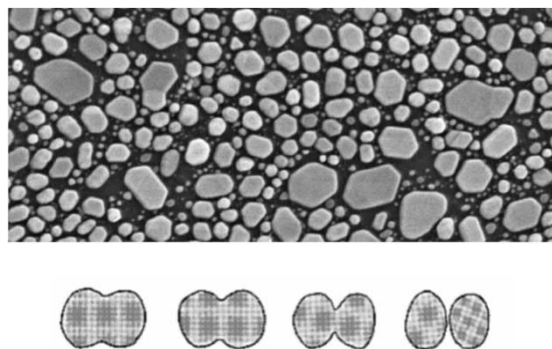
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Image formation



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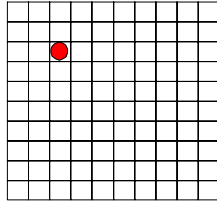
Resolution



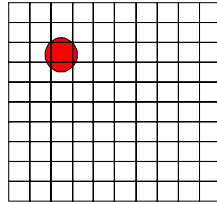
Resolution R is in practice minimum resolvable distance in the object

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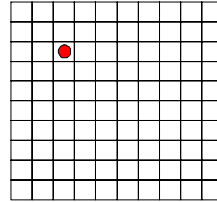
Optimum resolution



optimal



empty
resolution



poor SN
ration

Empty magnification



Empty magnification



2x

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Empty magnification



4x

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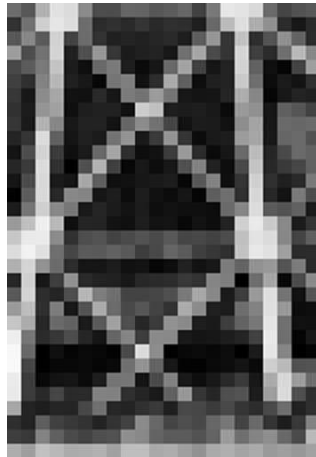
Empty magnification



8x

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Empty magnification

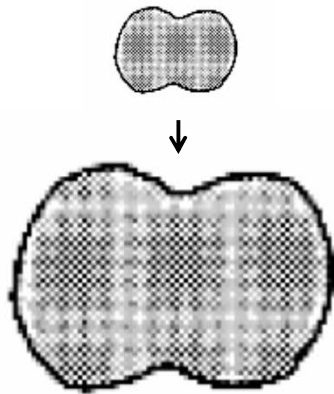


16x

Useful
magnification
is related to
resolution

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Empty magnification



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

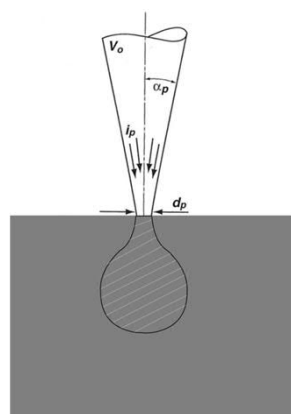
if

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

Making image larger
does not increase
Information

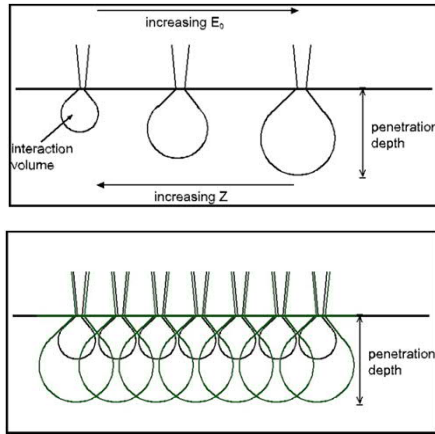
$$(R_{\text{eye}} \approx 0,2 \text{ mm})$$

Factors effecting resolution



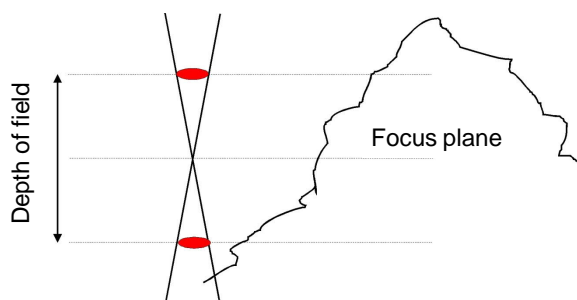
- "Spot size" d_p
- Interaction volume
- Contrast

Resolution and interaction volume



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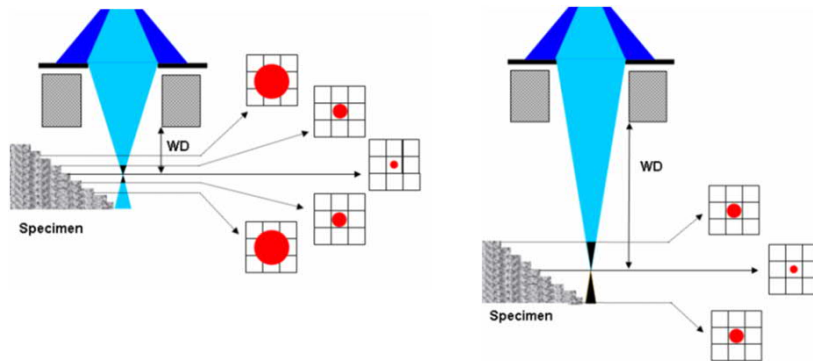
Depth of field



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

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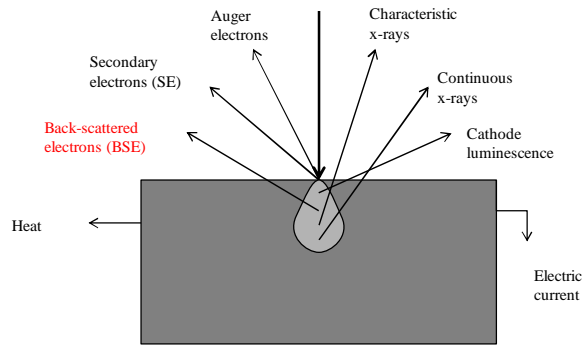
Depth of field



Analysis of local chemical composition tentatively

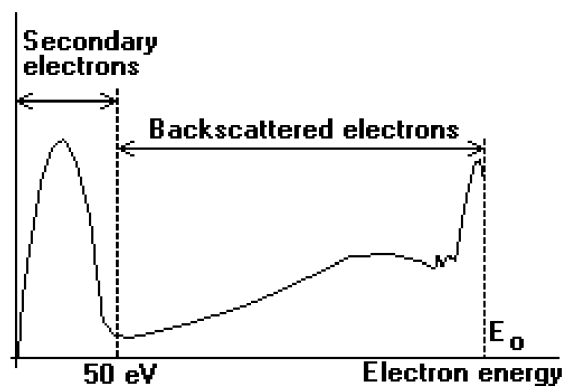
- Back-scattered electrons
- Back-scattered electron yield is a function on atomic number

Electron beam specimen interactions



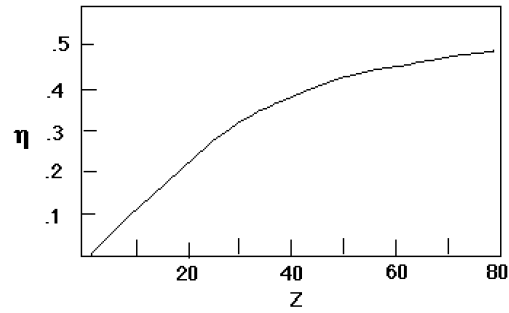
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Energy distribution of emitted electrons



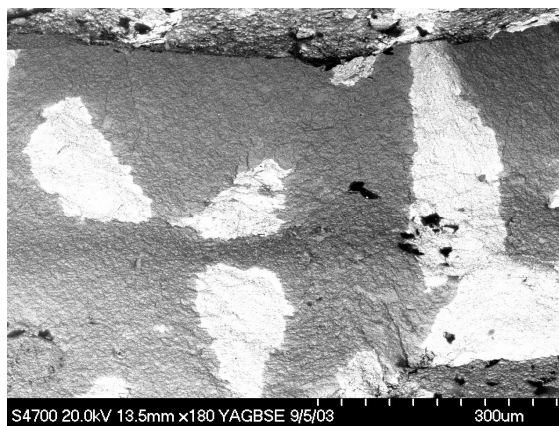
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Backscattered electron yield as a function of atomic number



η = backscattering coefficient

BSE image from a metal matrix composite



Overview of instrument capabilities

- High magnification
 - Large depth of field
 - Chemical information in micrometer scale (BSE, EDS, WDS)
 - Crystallographic information (EBSD)
 - Special techniques (EBIC, CL, voltage contrast)
 - In-situ experiments (temperature, strain, etc.)
- More than just a microscope
- More than just composition and structure

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 preparation as a main rule

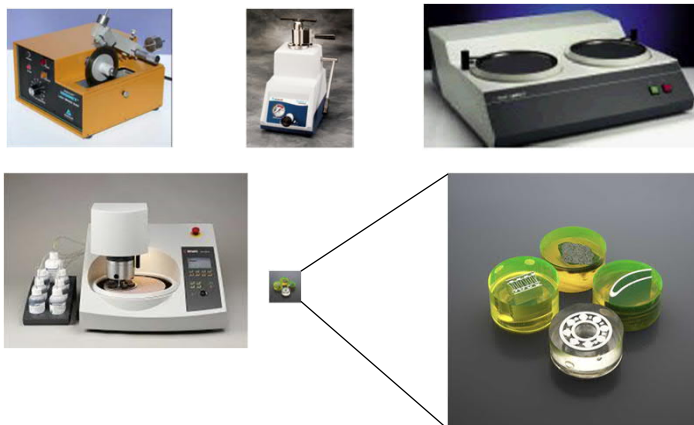
Sample preparation

- 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 very careful preparation
- Special preparation techniques for biological samples

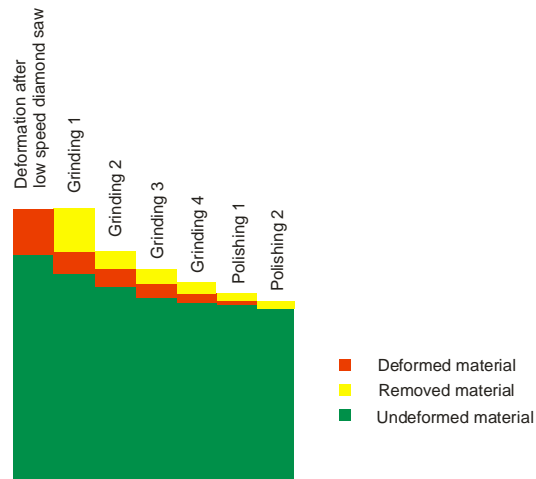
Materialographic sample preparation

- 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 precision 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

Coating methods



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

5. Case examples

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

Thank you for your attention