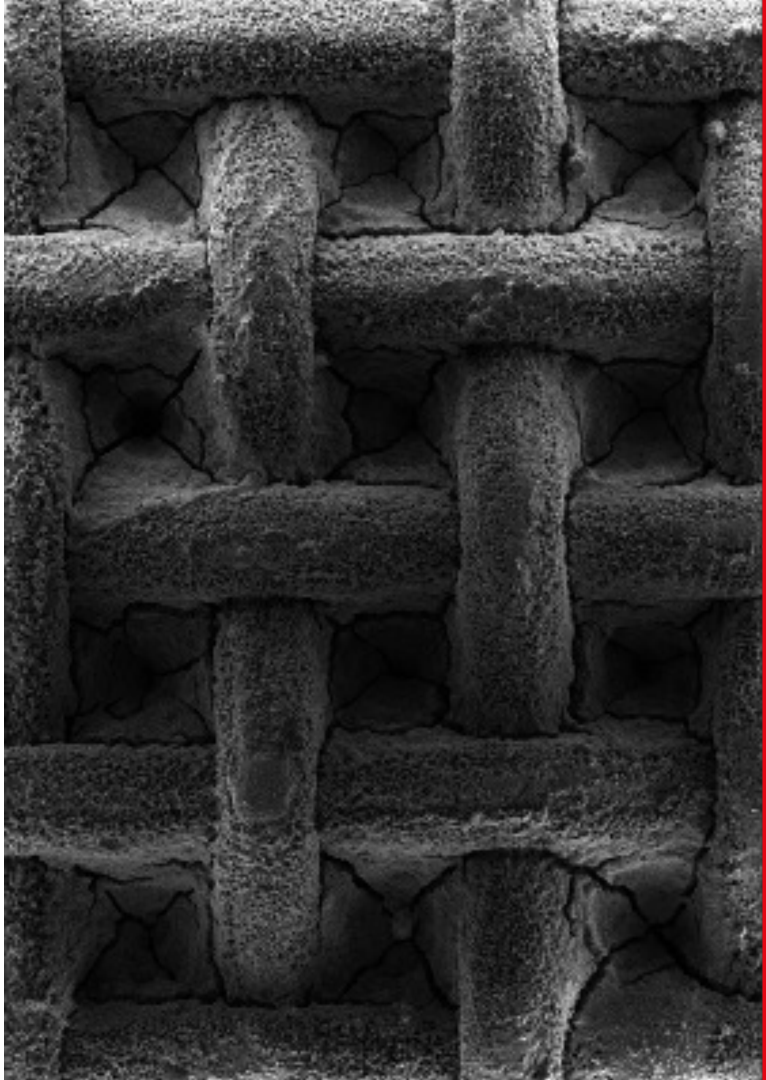


Scanning Electron Microscopy (SEM)

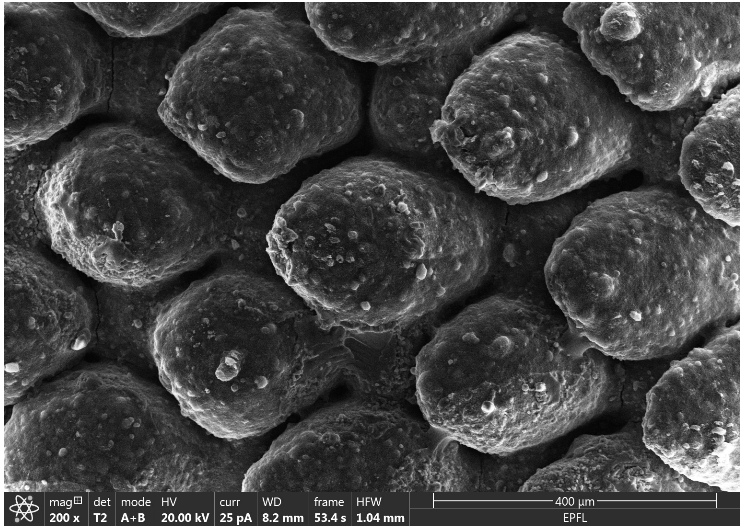
CH-633

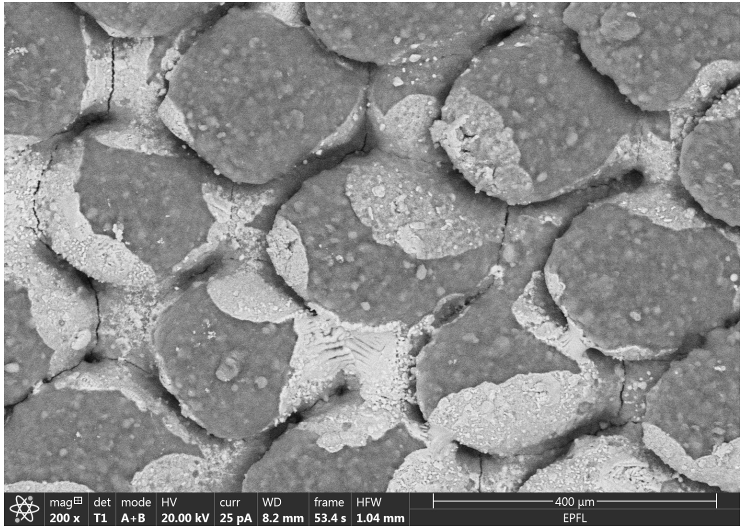
Emad Oveisi

Learning Outcomes

How the SEM works?

What type of information we can obtain from looking at the SEM images?

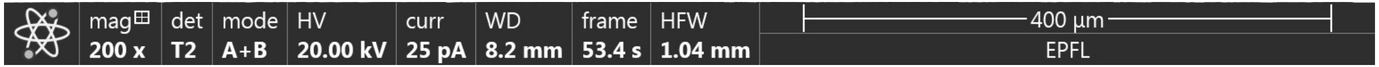




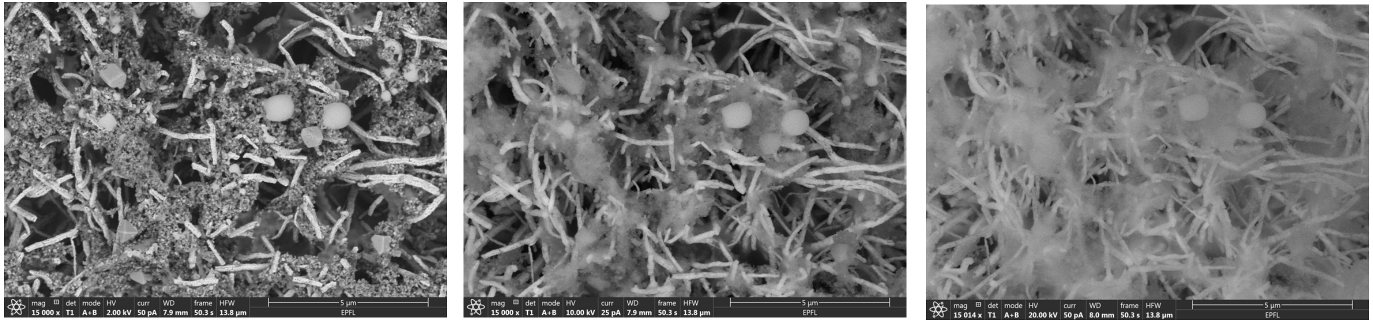
Why these two SEM images from identical locations are different?

Sample: Cu grid treated with KOH

Learning Outcomes

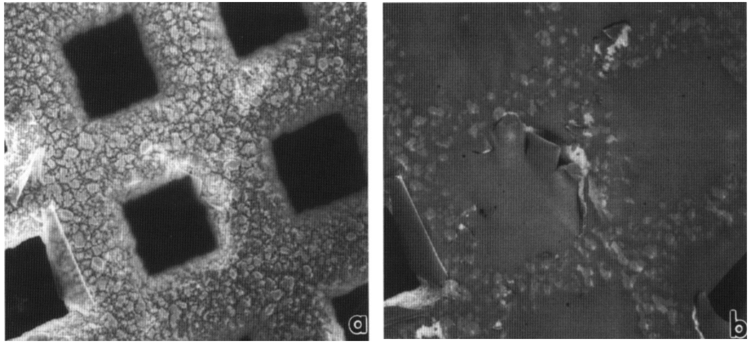


What can this information tell us?



Cu grid treated with KOH

How can we use it to interpret the images?



Carbon supported TEM grid

And how do we tune the microscope parameters to obtain the specific information we are seeking?

Contents

- SEM setup
- Imagining with SEM
  - Operation, Signals
  - Contrast mechanism
  - Interpretation of images
  - Challenges and artefacts
- Related techniques
- Image Quescussion (requires interaction and your input)

What is an SEM

Scanning **E**lectron **M**icroscope (SEM) is an instrument for observing and analyzing the surface structure of a bulk sample using a finely focused probe that scans the sample in raster.

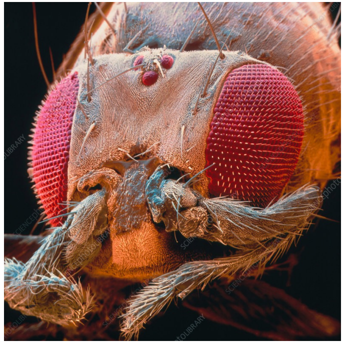
1st electron microscope (TEM) in 1933 by Ernst Ruska (1986 Nobel Prize in Physics)
The first true SEM was described and developed in 1942 by Zworykin
1st commercial SEM in 1965 by the Cambridge Scientific Instruments Mark I "Stereoscan"

Teneo, TFS

Primary applications:

- Surface topography and morphology; in life and materials sciences
- Composition analysis (e.g. EDX or WDX)
- Crystallography (e.g. EBSD)
- Optical and electronic properties (e.g. Cathodoluminescence), and more …

SEM can achieve 1-5 nm resolution, depending on the instrument and the imaging condition being used.



Coloured image of fruit fly mutant

https://www.sciencephoto.com

Bogner et al., A history of scanning electron microscopy developments, Micron 2007.

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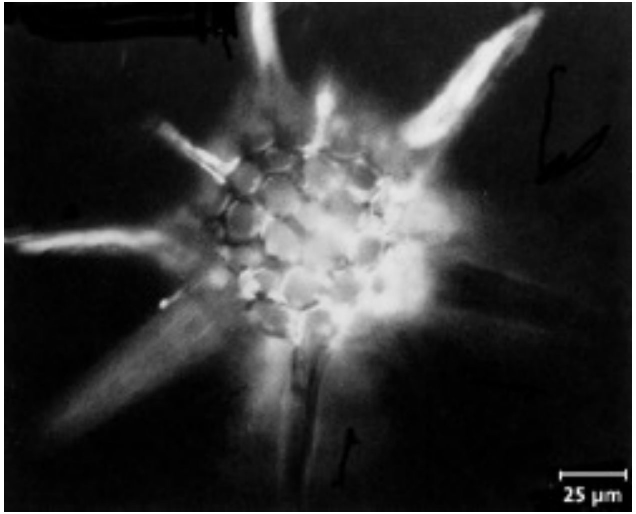
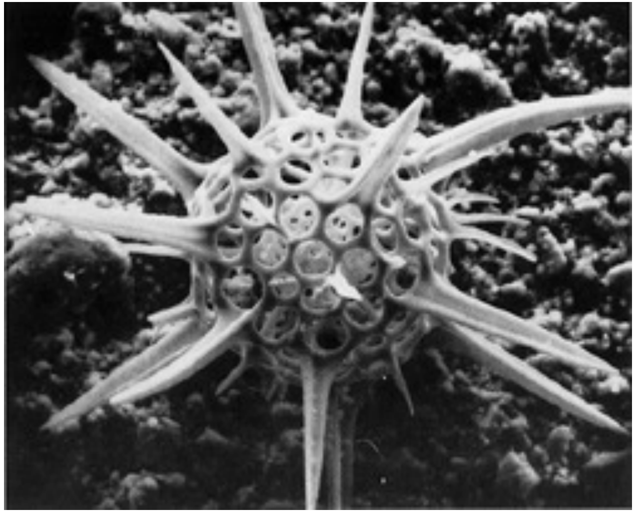
Why fast electrons?

Light microscope



SEM



Radiolarian

Compared to light microscopy, SEM offers:

- Better resolution; shorter wavelength of electrons
- Better depth of field (i.e. how much of depth is sharp = in focus); lower convergence angle of the electron beam, in the order of mrad

From J.I. Goldstein et al., eds., Scanning Electron Microscopy and X-Ray Microanalysis, (Plenum Press, NY 1980).

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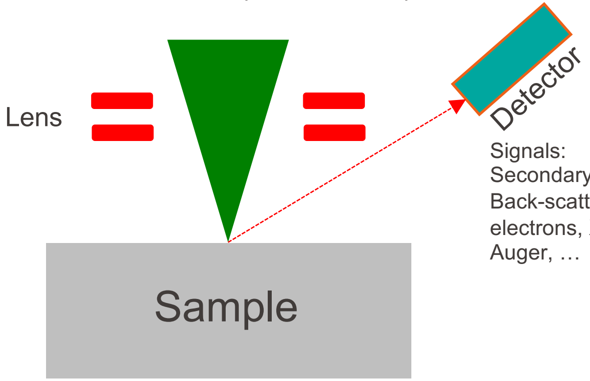
How SEM works?

Electron beam (0.2-30 keV)



Signals:
Secondary electrons, Back-scattered electrons, X-ray, Auger, …

The whole system is kept under high vacuum

Electrons gun generates electron and accelerates them to high speed (energies).

Electron will go though the 1st lens system to form a the smallest possible probe of desired current
(i.e. number of electrons / time / surface area)

2nd lens system focuses and scans the probe on sample

Electrons interact with the sample and different signals are generated

Various detectors surrounding sample collect radiated signals for each scan point to form an image
→ Image is formed point by point

Components of the SEM | Vacuum system

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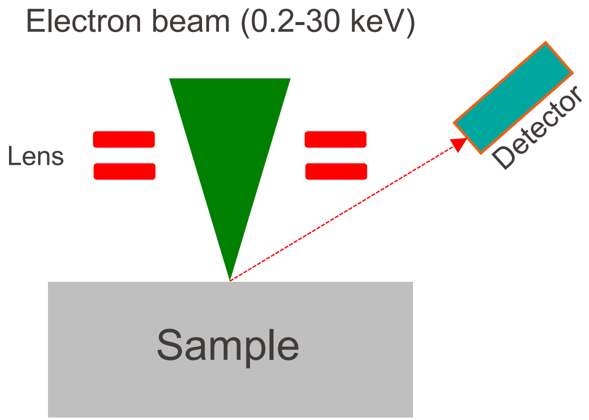


Vacuum system
Electron gun
Electron optics
Detectors

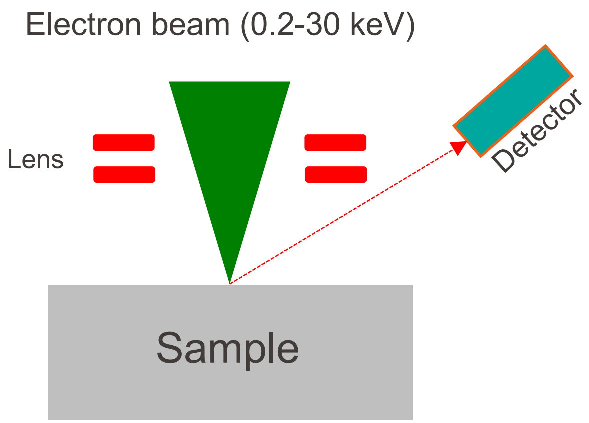
Why we need a vacuum system?

1. Electron propagation is only possible through vacuum (e^- interacts heavily with the matter)
2. A good vacuum system is crucial to reduce contamination and surface modification

Vacuum system has to be VERY clean:

- Clean samples, not dusty or oily or other solvents
- Use gloves to handle the sample

Components of the SEM | Electron gun



Vacuum system
Electron gun
Electron optics
Detectors

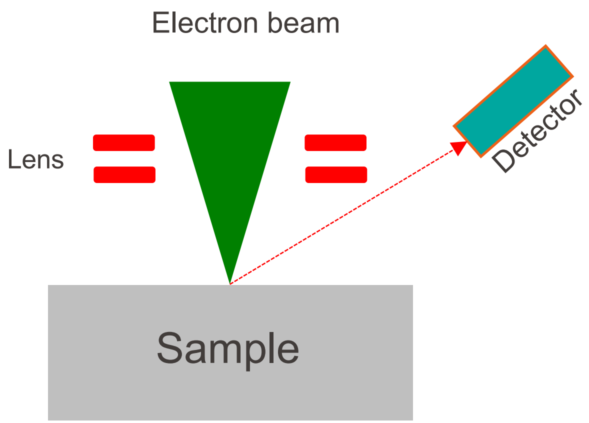
Purpose: To create a narrow intense beam of electrons

Electrons can be released by heat or an electric field

3 types of electron guns:

- Thermionic gun (Thermal)
  Heat only
- Cold field emission gun (cold FEG)
  Electric field: Potential (voltage) difference
- Heat assisted field emission gun: Schottky emitter
  Heat + Electric field

Components of the SEM | Electron optics



Purpose:

In the SEM we use lenses **(condenser system)** to:
1- condense the electron beam into a fine probe (defines probe size and current);
2- focus the probe on the sample **(objective lens | e.g. final lens)**

Scan coils raster scan the beam over the sample

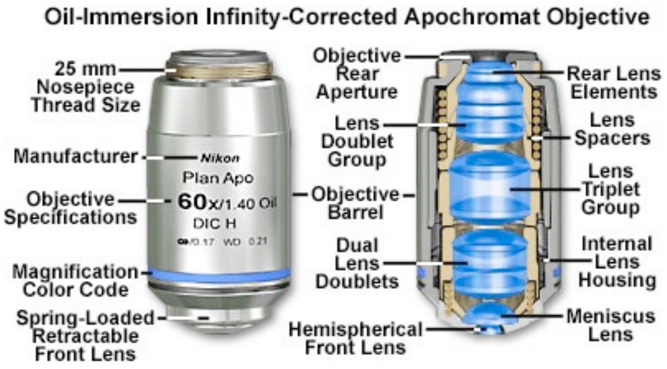
Electrons pass through a series of lenses and apertures

Vacuum system
Electron gun
Electron optics
Detectors

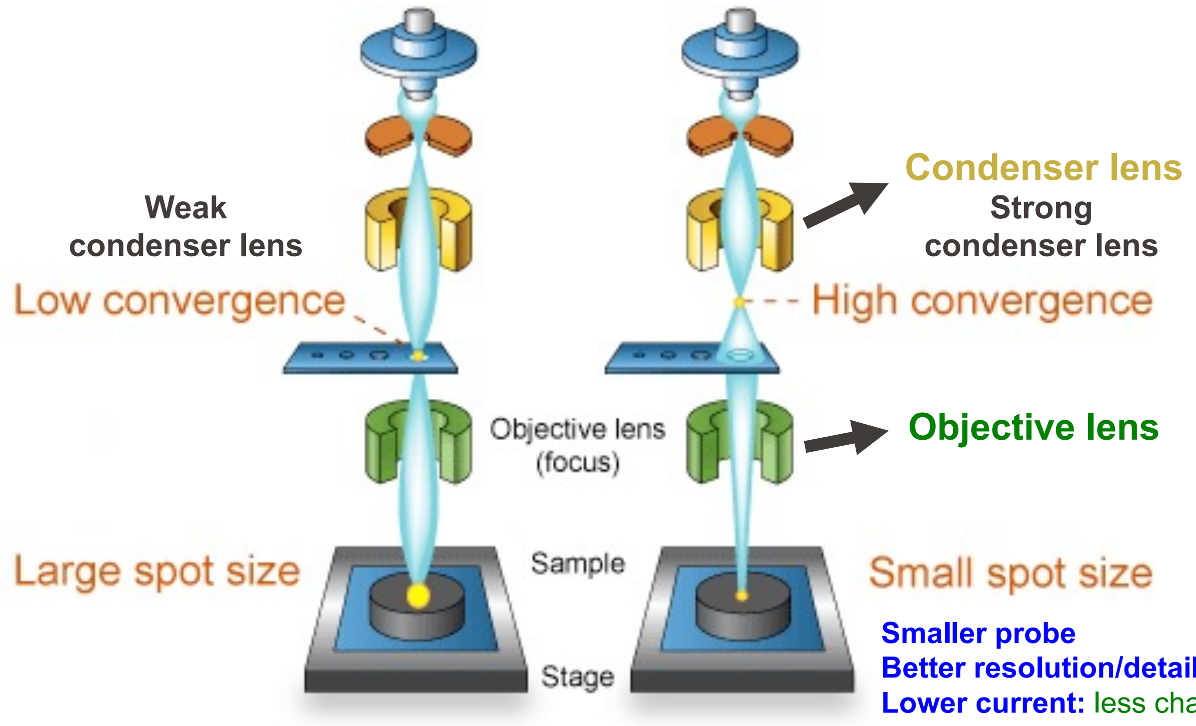
The smaller the probe size, the better the resolution, but the lower the probe current (number of electrons that hit the sample)

Components of the SEM | Electron optics

- Lenses for light
  - Glass or polymer lenses
  - Deflection of light through changing refraction index
- Lenses for electrons
  - Variable focus
  - Electrostatic
  - Electromagnetic: Lorentz force



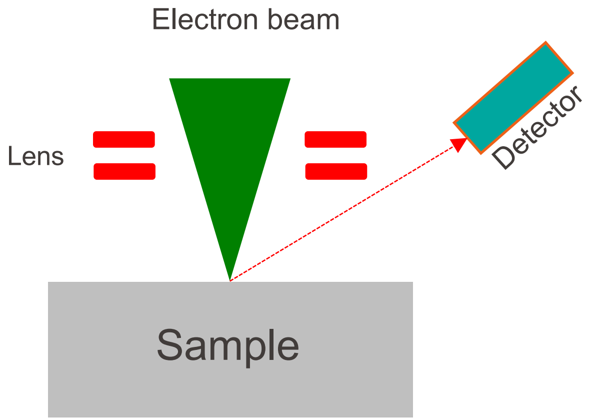
Components of the SEM | Electron optics



Smaller probe
Better resolution/details
Lower current: less charging and beam damage
lower signal to noise ratio

In SEM, the lens system is used to de-magnify the image of the beam source and to focus the beam on the specimen.

Components of the SEM | Detectors



Vacuum system
Electron gun
Electron optics
Detectors

Purpose:

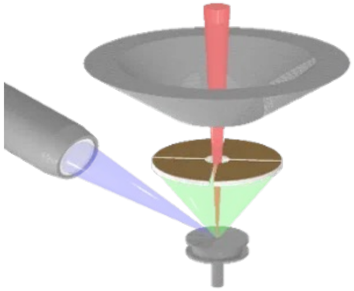
To “see” the electrons, means create signal that is proportional to the number of electrons emanating from the sample at each scan position

Scan coils, detector and display (monitor) are synchronized

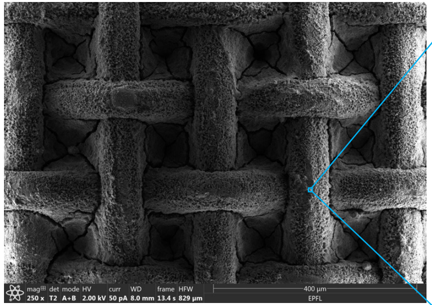
There exist detectors for electrons, x-ray, etc.

Two typical electron detectors for the SEM:

- Scintillator/Photomultiplier
  Everhart-Thornley detector
- Semiconductor detector
  Silicon diode with a *p*-*n* junction



How an SEM image is generated?



1534x1024 Pixels
Frame time 13.4 Sec

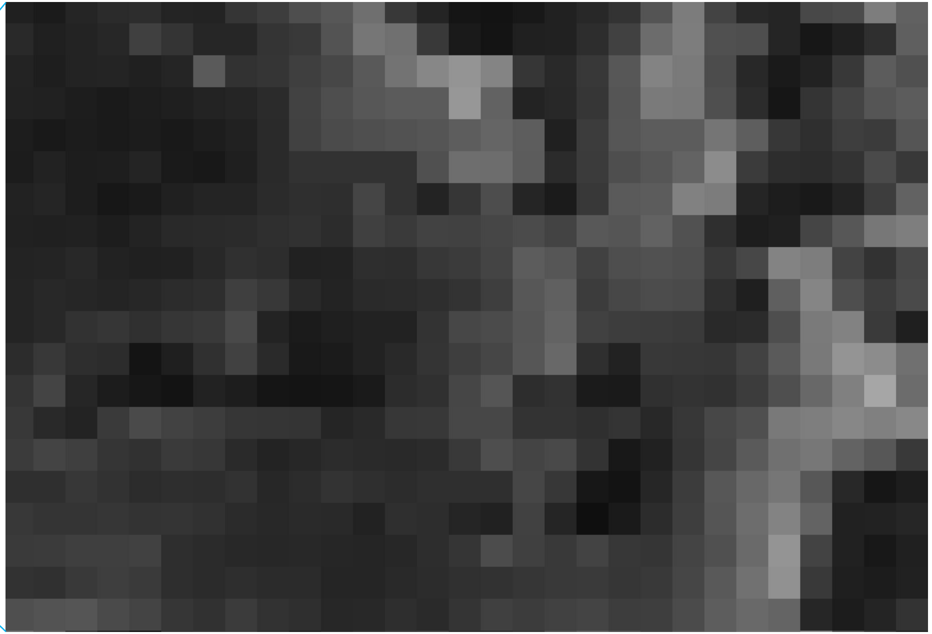
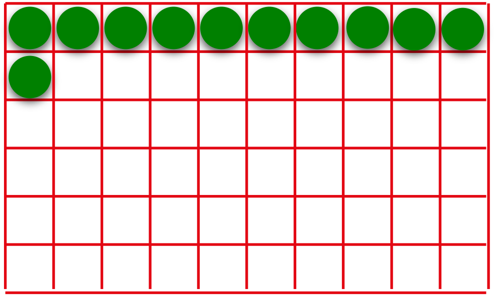


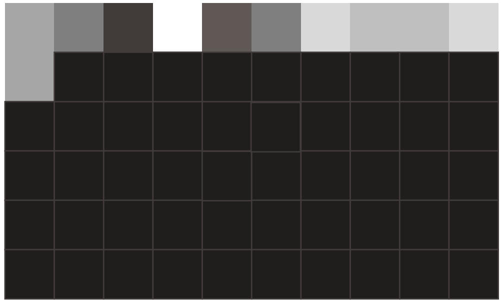
Image is made of pixels of different grey level

EPFL

How an SEM image is generated?



Beam locations on the specimen



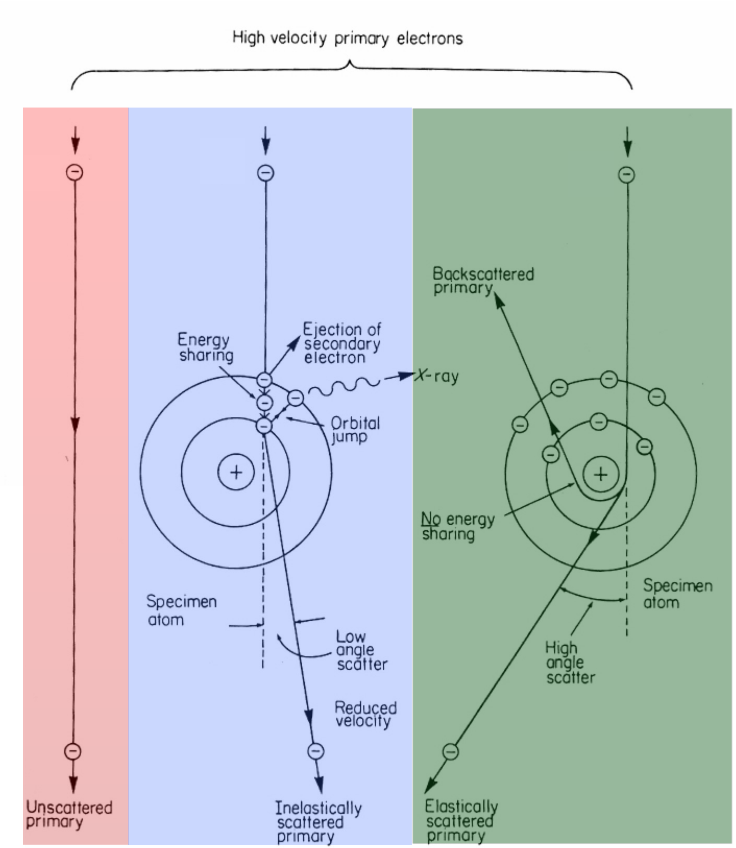
Area scanned on the screen

Information transfer f(x,y,S)

- Image formed step by step by the sequential scanning of the sample with the electron probe (using pair of deflector or scan coils, controlled by the scan generator)
- Monitor and scanning coils are synchronized
- Intensity of each pixel is proportional to signal received (collected SE/BSE electrons)
- When changing the magnification, we just change the raster size (no change in optics)

Magnification = Image size (e.g. display) / Raster size on the specimen

e- matter interactions

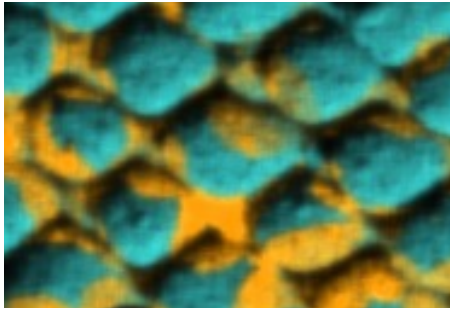


Inelastic events: The result is a **transfer of beam energy** to the specimen atom (**energy loss**) and a potential expulsion of an electron from that atom as a **secondary electron (SE)**.

If the vacancy due to the creation of a secondary electron is filled from a higher level orbital, an X-Ray or Auger characteristic of that energy transition is produced.

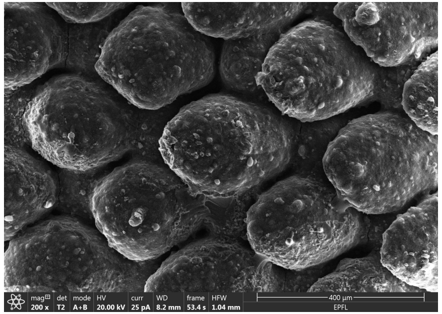
That's why TEMs are shielded!

Characteristic X-rays elemental map



Chemical composition

Secondary electron SEM image



Topography

Cu grid treated with KOH

SEM techniques

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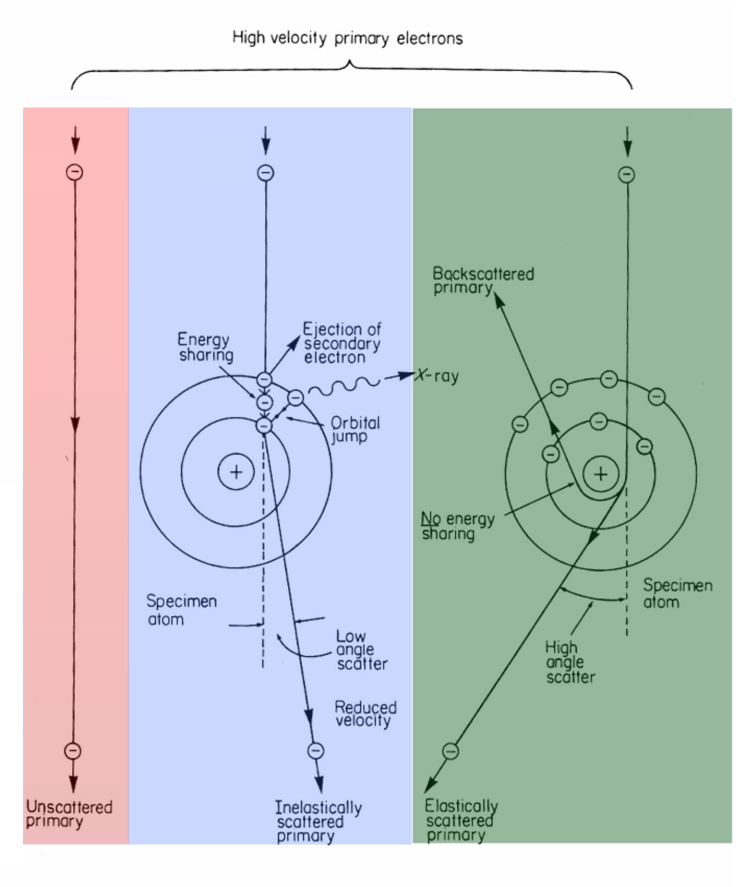
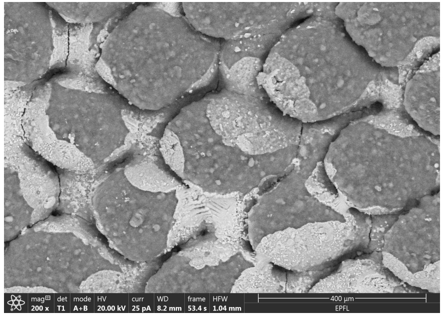
e- matter interactions



Elastic events occur when a beam electron interacts with the electric field of the nucleus or electron cloud of a specimen atom (Coulomb forces), resulting in a change in the direction of the beam electron **without a significant change in the energy** of the beam electron (< 1 eV).

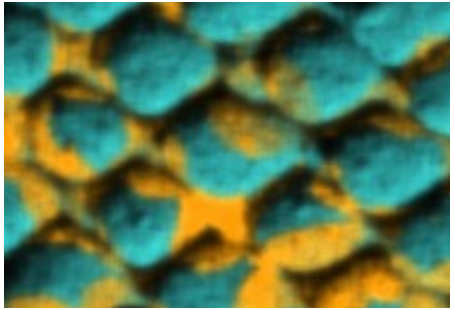
Coulombic interaction within the electron cloud, Low-angle scattering
Coulombic attraction by the nucleus, Higher-angle scattering

Larger nucleus → More backscattering



Mass contrast
Backscattered SEM image

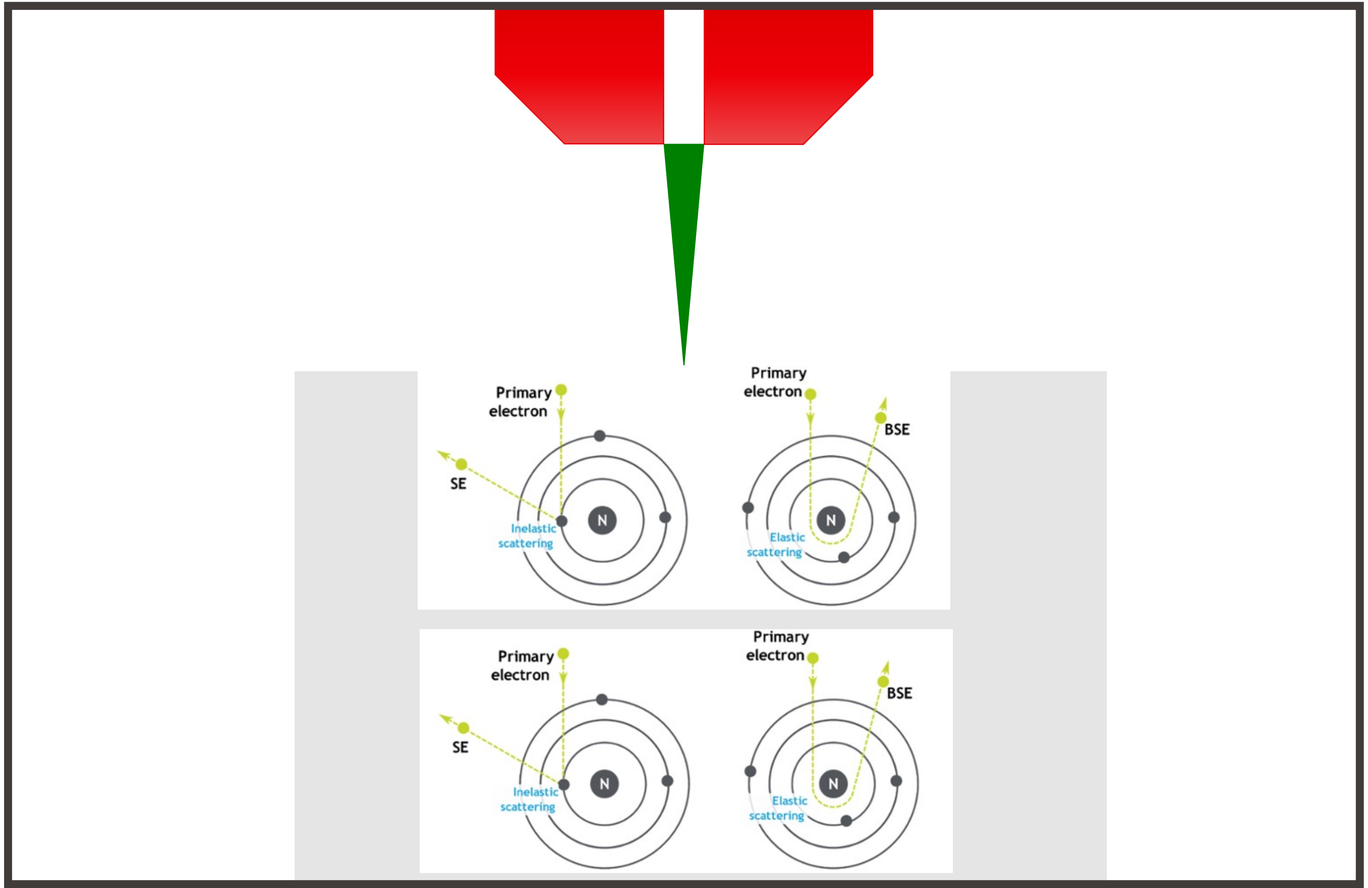
Characteristic X-rays elemental map



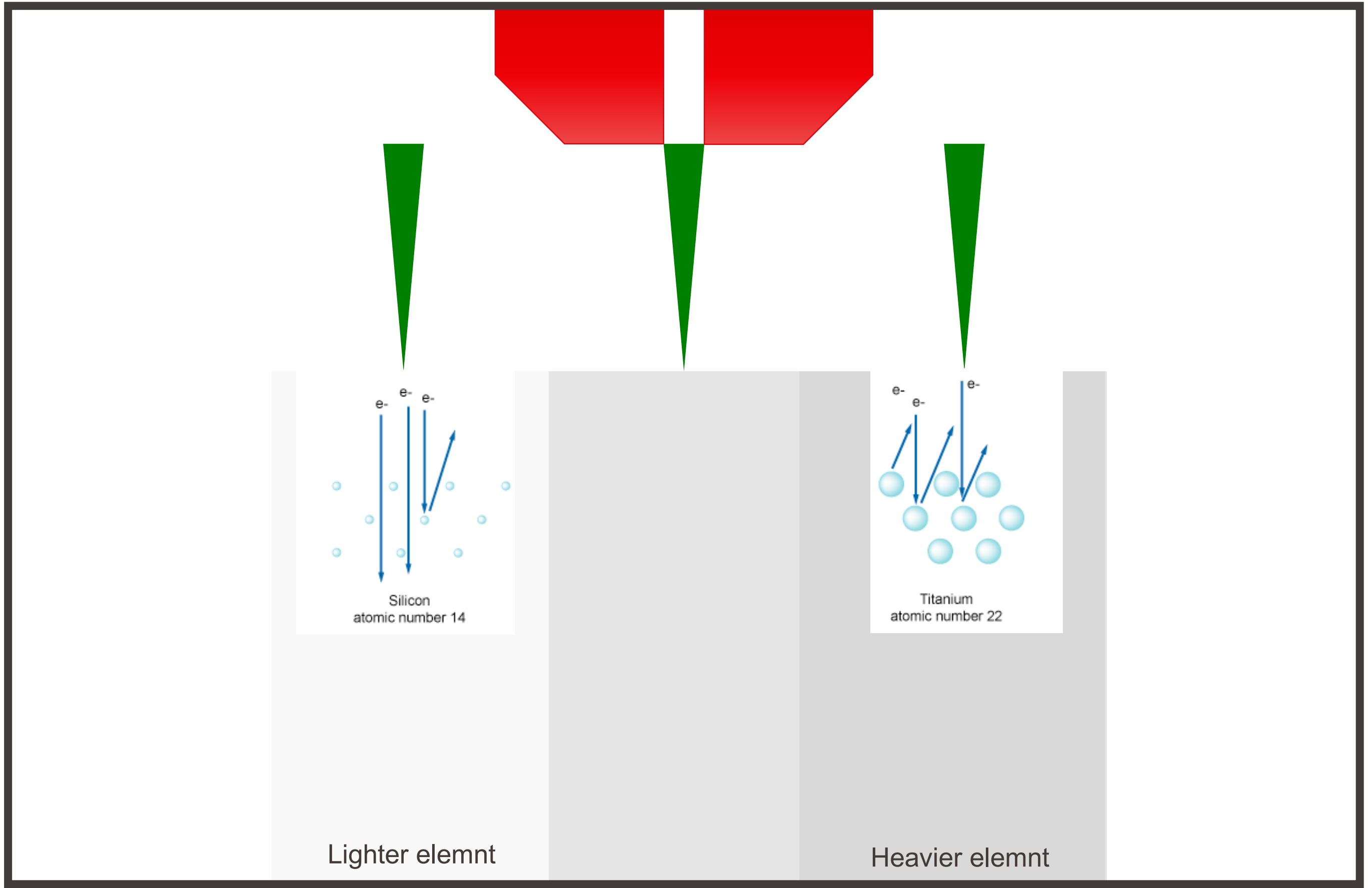
Chemical composition

Cu grid treated with KOH

How an SEM image is generated?

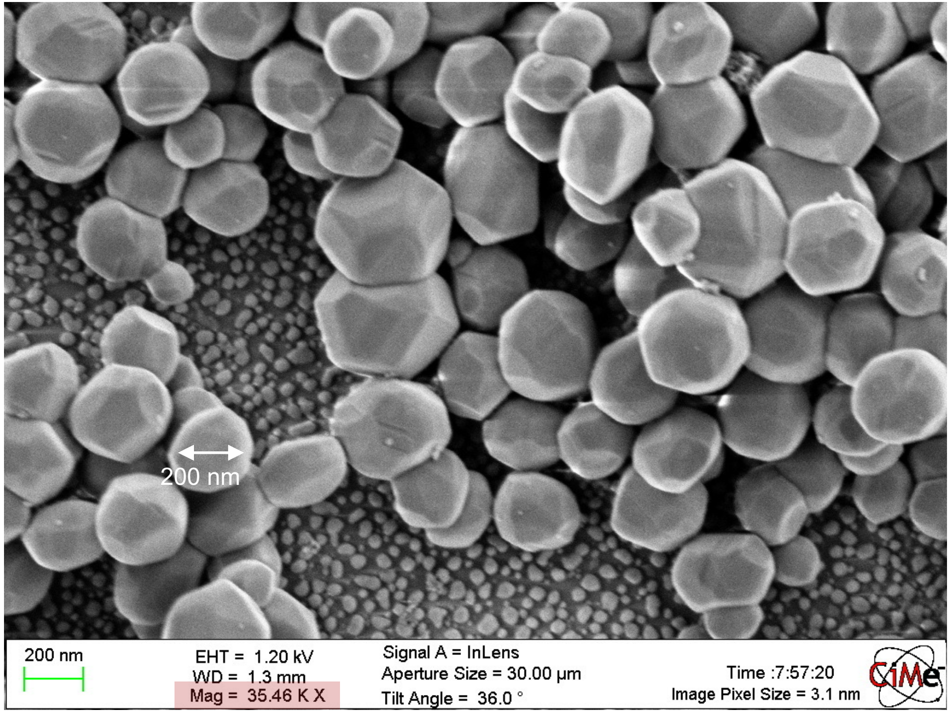


How an SEM image is generated?



How an SEM image is generated?

Measure the size of the particle and calculate the magnification

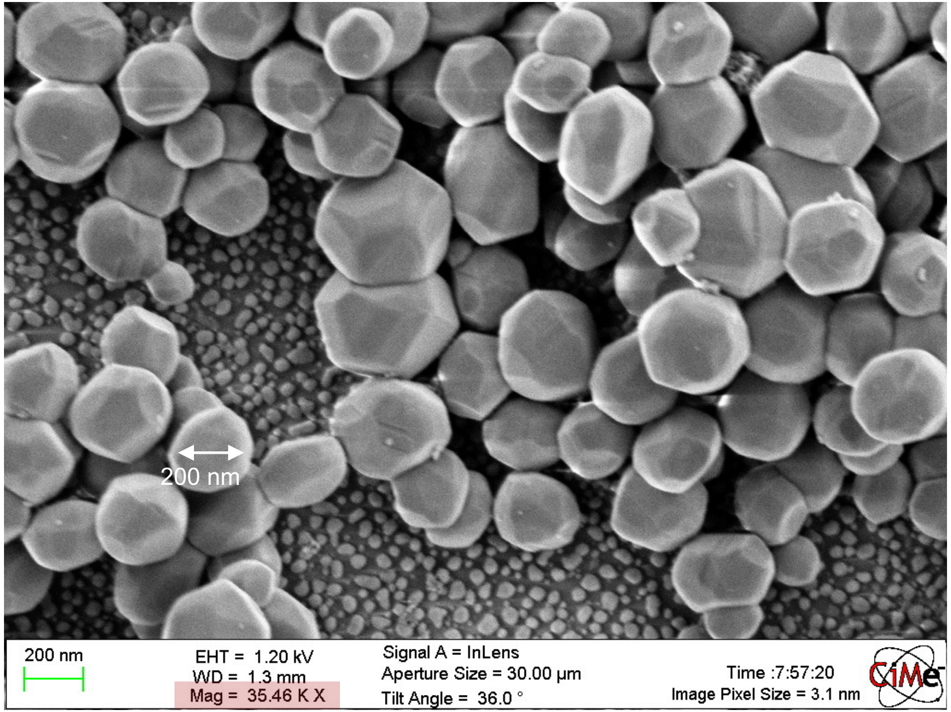


Assuming the size of the indicated particle on your screen is 2 cm, what is the magnification of the image?

a) 35.46 kX
b) 500 kX
c) 100 kX
d) 200 nm

How an SEM image is generated?

Measure the size of the particle and calculate the magnification



Assuming the size of the indicated particle on your screen is 2 cm, what is the magnification of the image?

a) 35.46 kX
b) 500 kX
c) 100 kX
d) 200 nm

Trust the scale bar and not the indicted magnification

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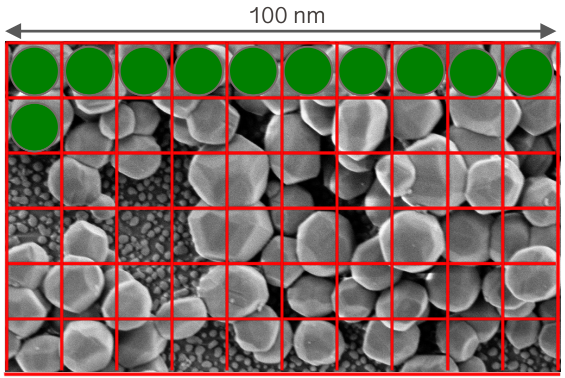
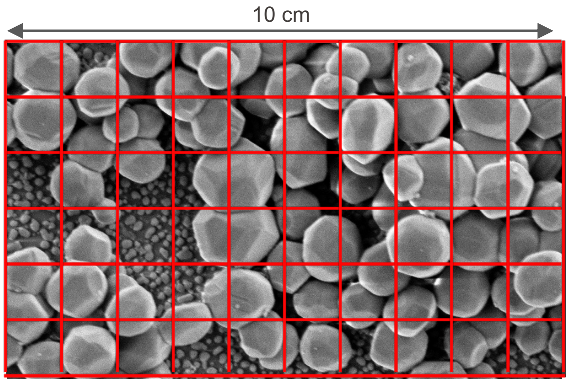
How an SEM image is generated?

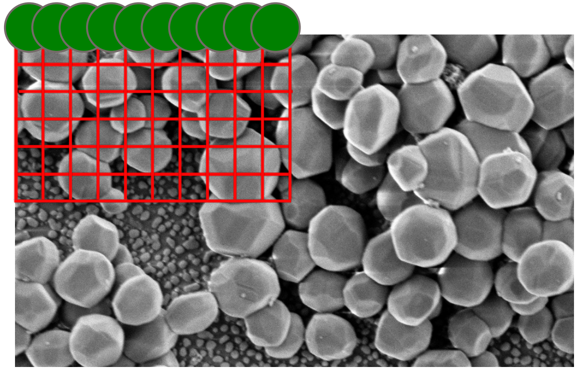
Sample | **Monitor**



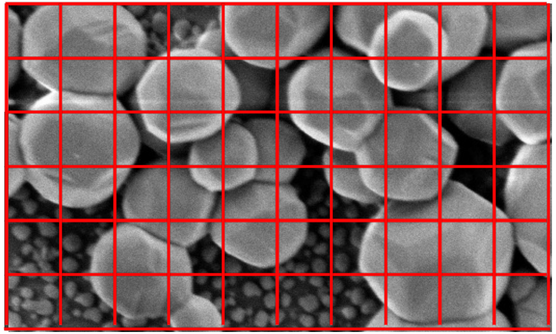
Scan step (i.e. pixel size) on the sample?



Pixel size on the screen?



Scan step (i.e. pixel size) on the sample?

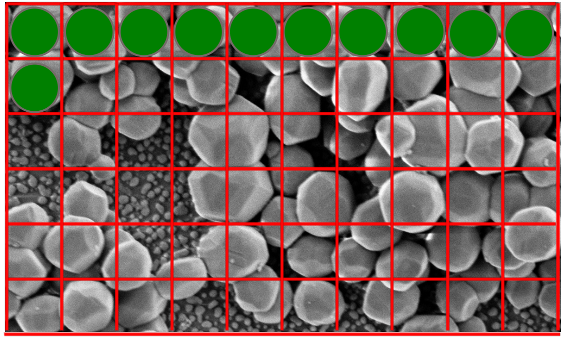


Pixel size on the screen?

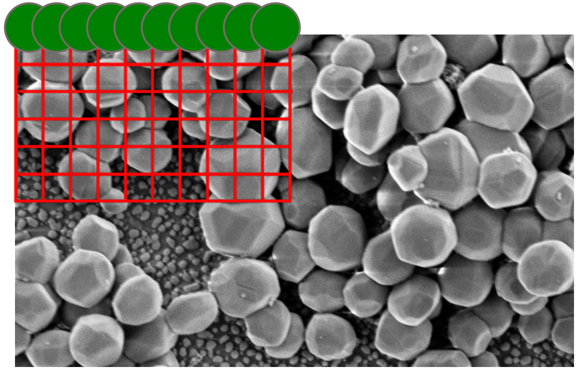
How an SEM image is generated?

Sample

100 nm



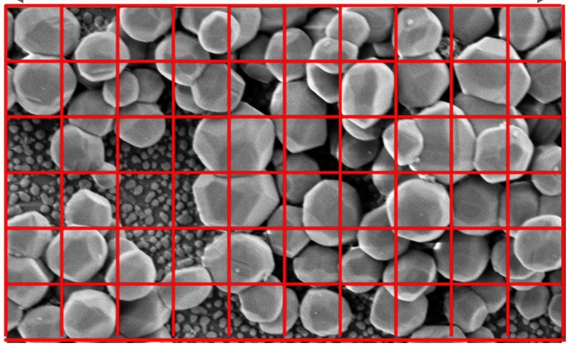
Scan step (i.e. pixel size) on the sample? = 10 nm



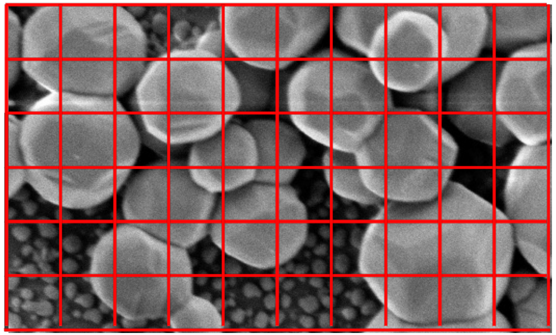
Scan step (i.e. pixel size) on the sample = 5 nm

Monitor

10 cm



Pixel size on the screen? = 1 cm



Pixel size on the screen? = 1 cm

Magnification = 10^{-2} / 10^{-8} = 1 MX

Magnification = 10^{-2} / 5.10^{-9} = 2 MX

What happens to resolution?

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Imaging parameters

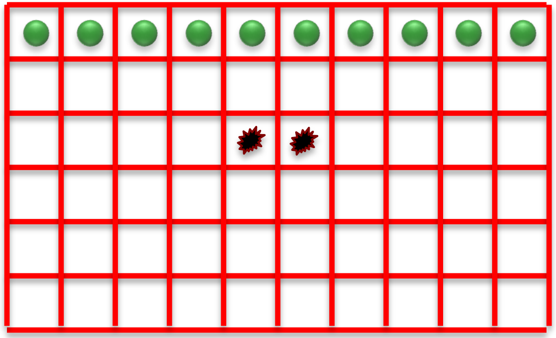
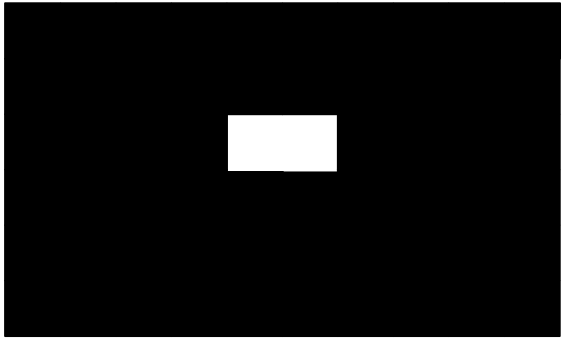
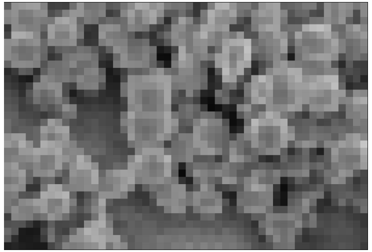
Parameters affecting Resolution (and Visibility)

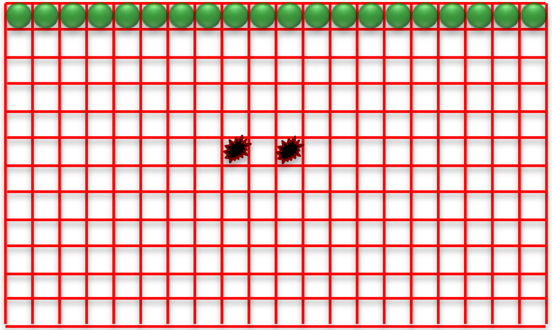
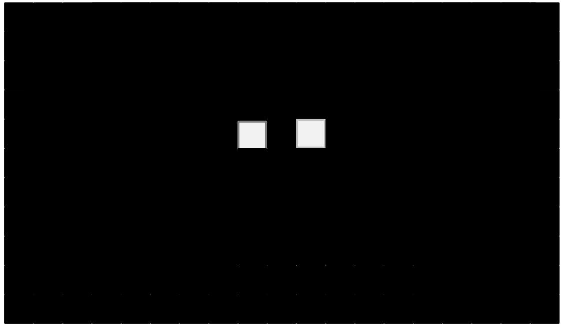
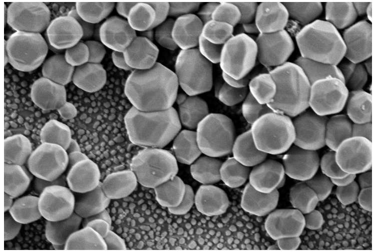
- **Fundamental**
  - Electron wavelength (beam energy) and diffraction limit: → Rayleigh criterion
  - Aberrations: enlarges the probe size

    Probe size (or spot size) means the diameter of the final beam at the surface of the specimen

- **Operational and sample**
  - Pixel size = scan step size
  - Probe size (also defines probe current and affects visibility)
  - Visibility:
    - Scan speed (i.e. dwell time) and "signal to noise ratio"
    - Contrast
  - Type and depth of emitted electrons signal
  - System/Specimen stability → Challenges (charging, contamination, beam damage)

Resolution | Pixel size

Nyquist sampling (f) = d/2, where “d” is the smallest object, or highest frequency

Thus, the imaging sample rate (or pixel) size should be roughly half the size of the smallest object you wish to observe; e.g. if you need 100 nm resolution, then scan every 50 nm (at least).

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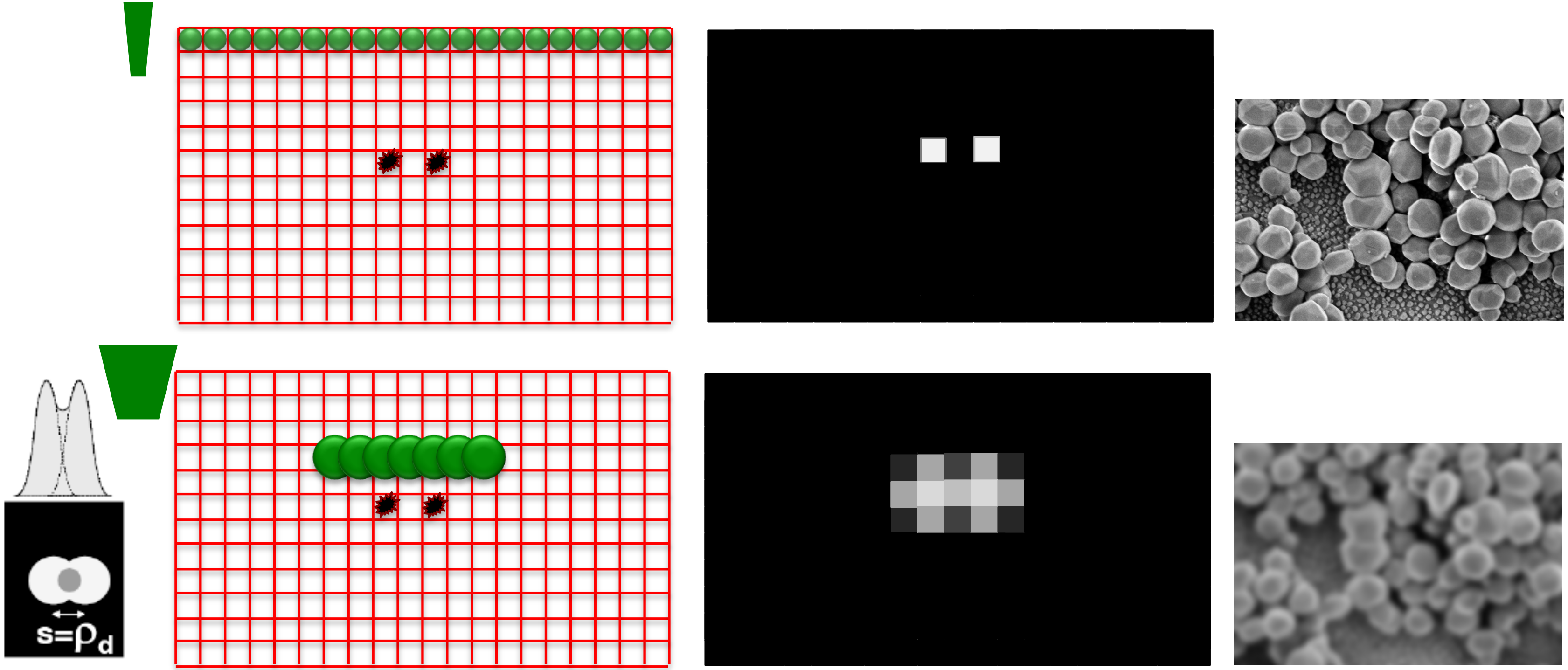
Resolution | Probe size



If a smaller probe has the potential to provide a higher resolution image, why is the smallest possible probe not always utilized?

Resolution | Probe size

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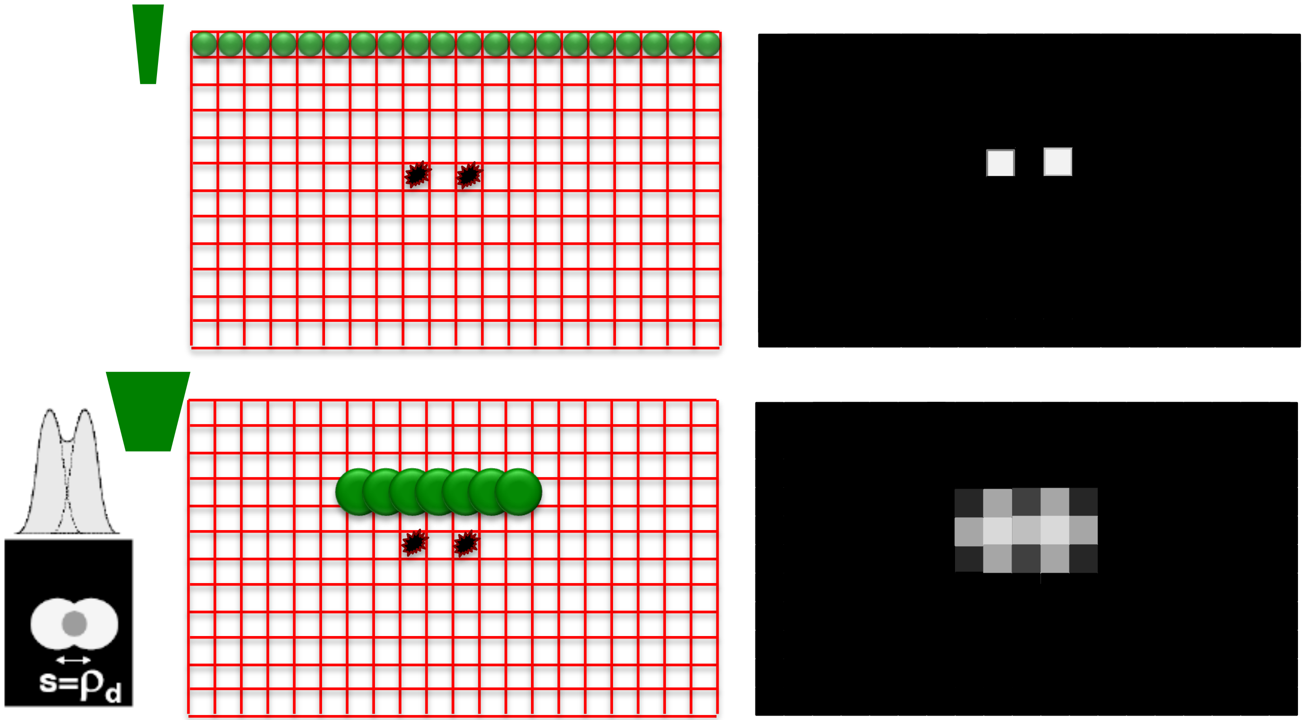
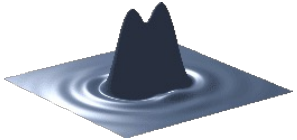


Rayleigh criterion

$\rho_d = 0.61\ \lambda / \alpha$



Resolution limit

A small probe diameter comes with a decrease in probe current:
means less electrons that impinges upon the specimen and generates the various imaging signals
→ **Less signal to noise ratio: indirectly affects resolution (visibility issue)**

Imaging parameters

Parameters affecting Resolution (and Visibility)

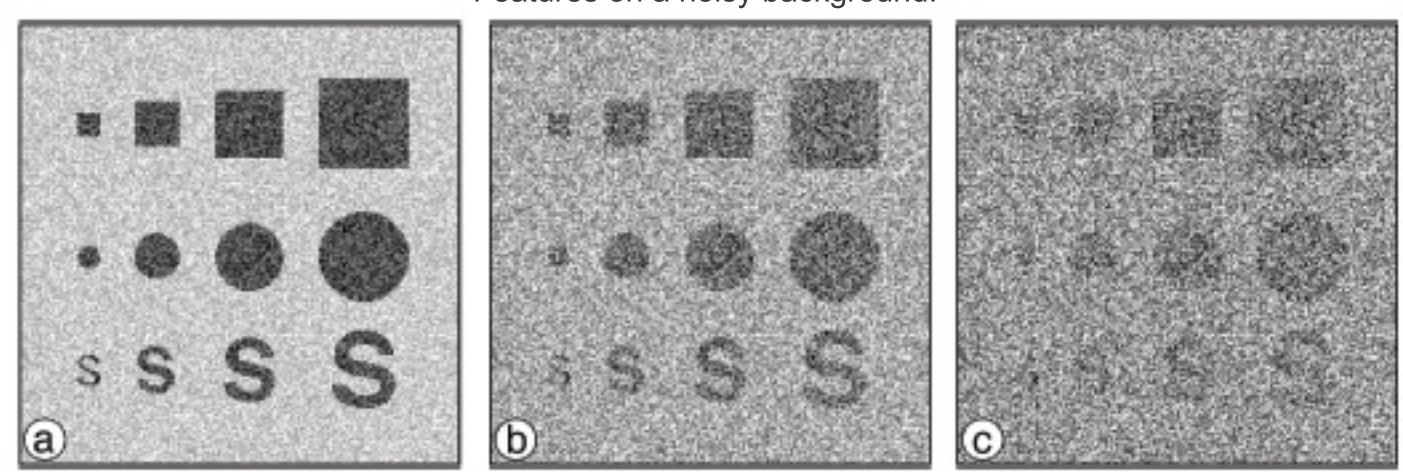
- **Fundamental**
  - Electron wavelength (beam energy) and diffraction limit: → Rayleigh criterion
  - Aberrations: enlarges the probe size

    Probe size (or spot size) means the diameter of the final beam at the surface of the specimen

- **Operational and sample**
  - Pixel size = scan step size
  - Probe size (also defines probe current and affects visibility)
  - Visibility:
    - Scan speed (i.e. dwell time) and “signal to noise ratio”
    - Contrast
  - Type and depth of emitted electrons signal
  - System/Specimen stability → Challenges (charging, contamination, beam damage)

Visibility and "signal to noise" ratio

Features on a noisy background:

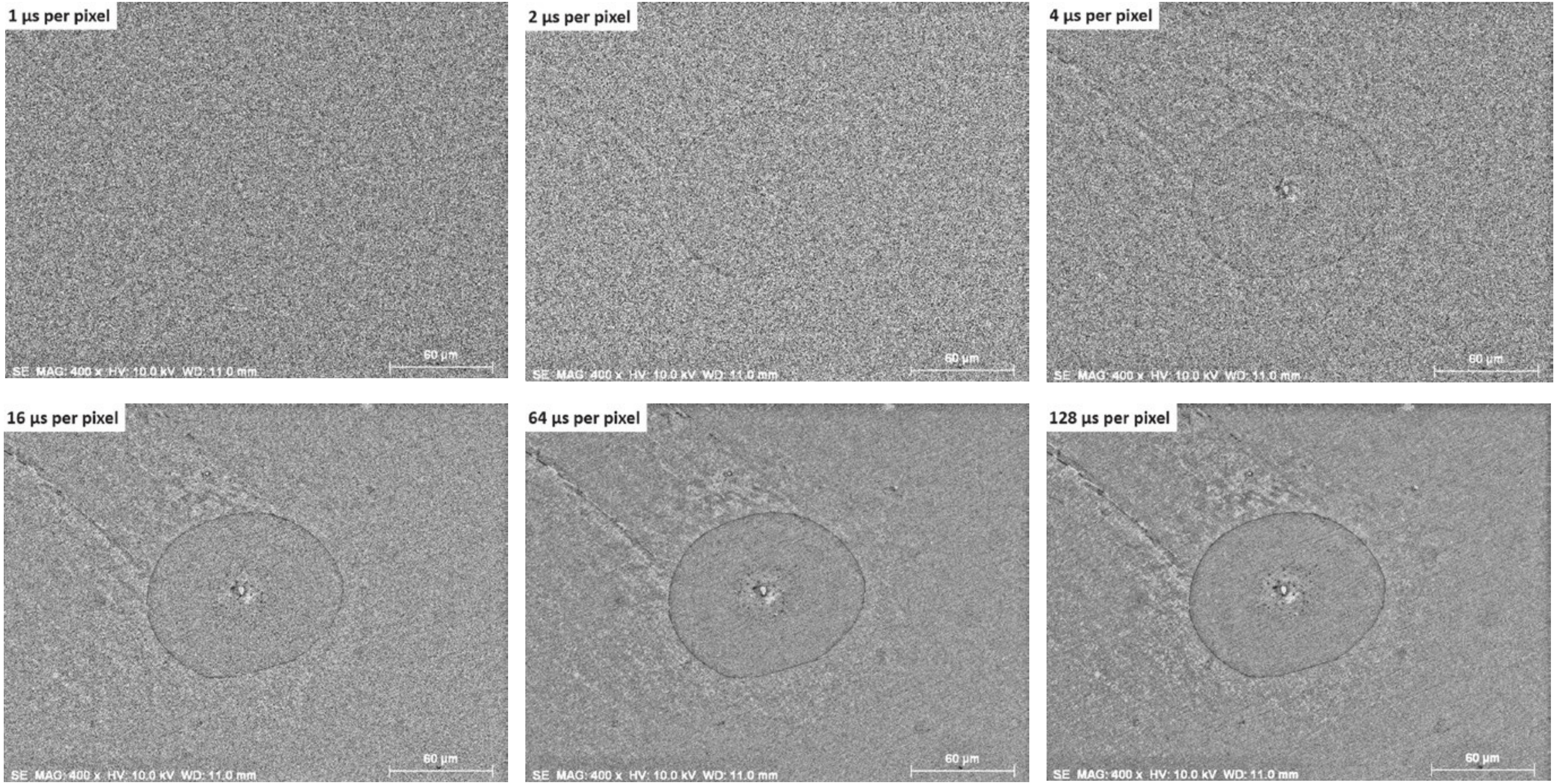


High signal to noise ratio

Low signal to noise ratio

How to improve signal to noise ratio?

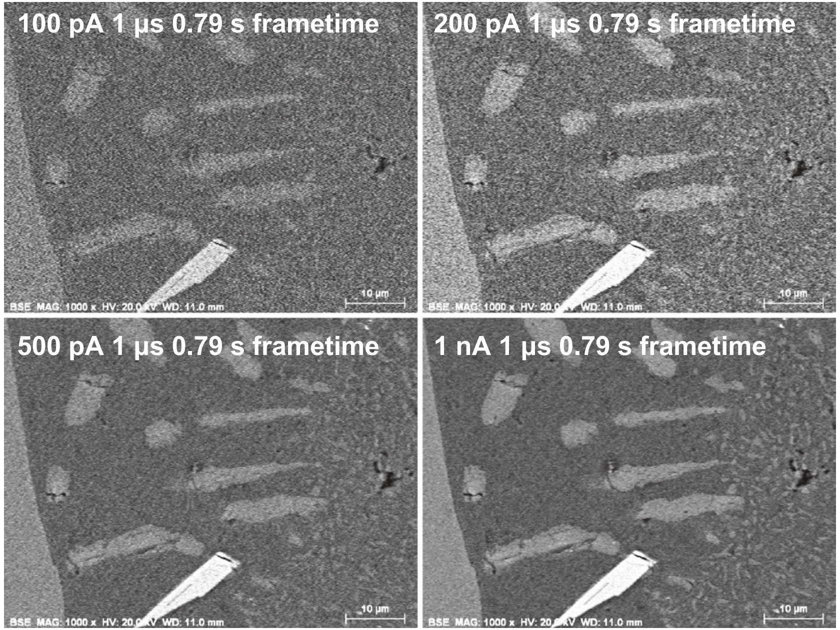
Visibility and “signal to noise” ratio



Threshold imaging visibility; image sequence with increasing pixel **dwell time** at constant beam current.
Sample: Inkjet deposited droplet on carbon planchet; (E_0 = 10 keV; Everhart–Thornley)

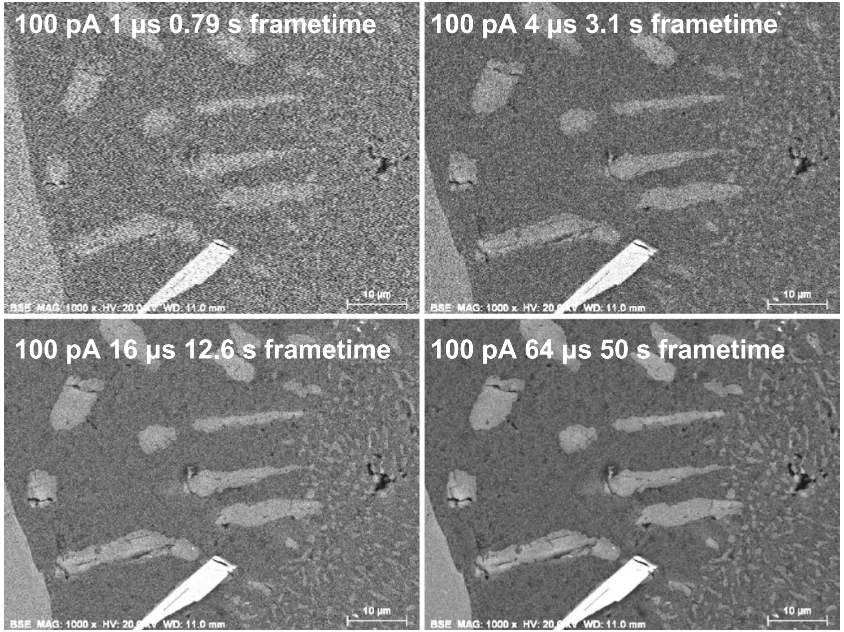
Visibility and "signal to noise" ratio

Effect of beam current



Possible charging or sample damage

Effect of pixel dwell time



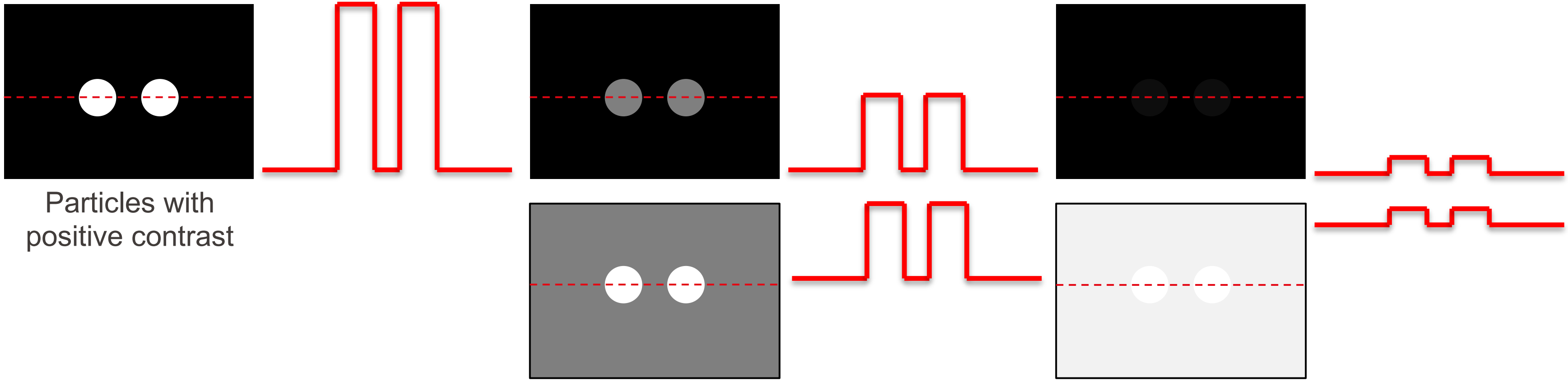
Sample drift may become the limiting factor

Visibility and contrast

100% Intensity change

50% Intensity change

5% Intensity change



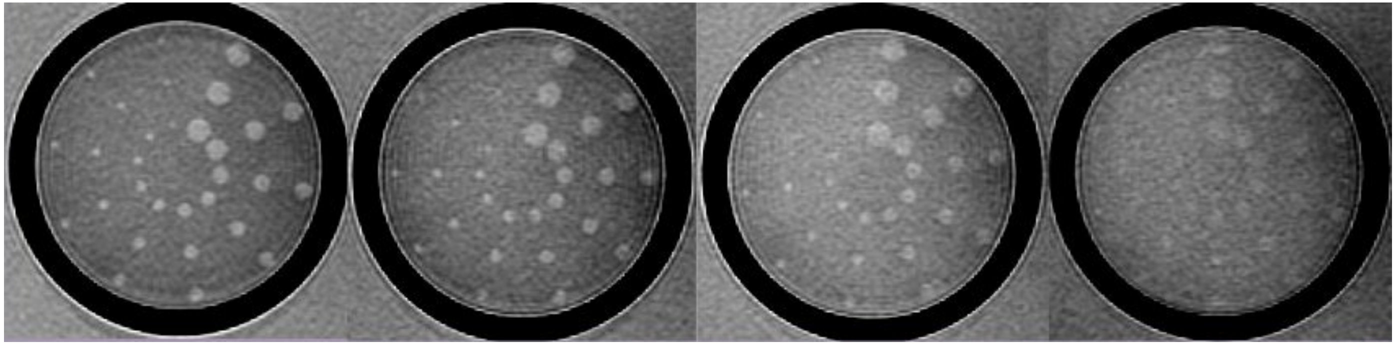
Particles with positive contrast

5.1% contrast

3.7% contrast

2.2% contrast

1% contrast



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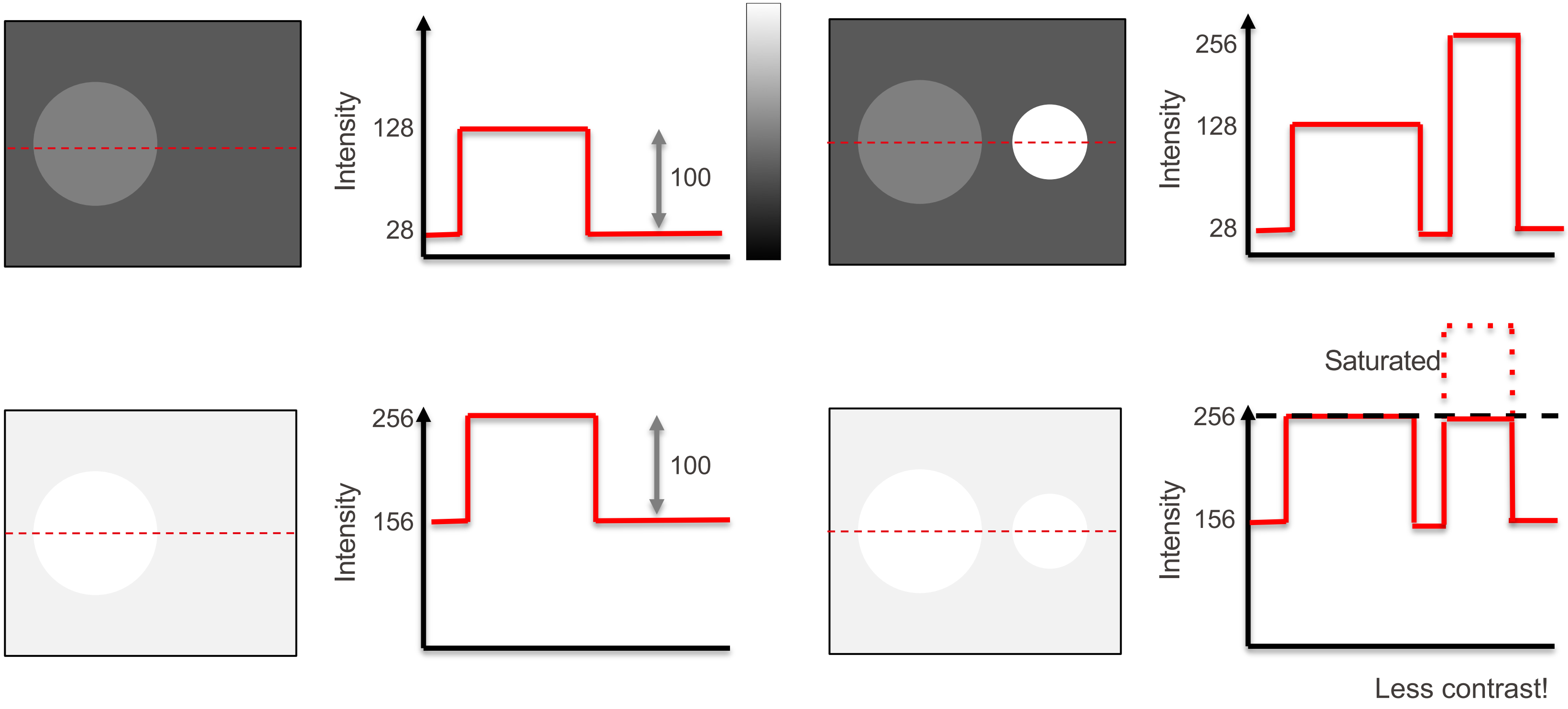
Visibility and contrast



Do not throw away the information

If the detector's brightness and contrast are not properly adjusted, then it is not possible later to correct with image processing; i.e. over/under saturation problem

Imaging parameters

Parameters affecting Resolution (and Visibility)

- **Fundamental**
  - Electron wavelength (beam energy) and diffraction limit: → Rayleigh criterion
  - Aberrations: enlarges the probe size

    Probe size (or spot size) means the diameter of the final beam at the surface of the specimen

- **Operational and sample**
  - Pixel size = scan step size
  - Probe size (also defines probe current and affects visibility)
  - Visibility:
    - Scan speed (i.e. dwell time) and "signal to noise ratio"
    - Contrast
  - Type and depth of emitted electrons signal
  - System/Specimen stability → Challenges (charging, contamination, beam damage)

SEM signals

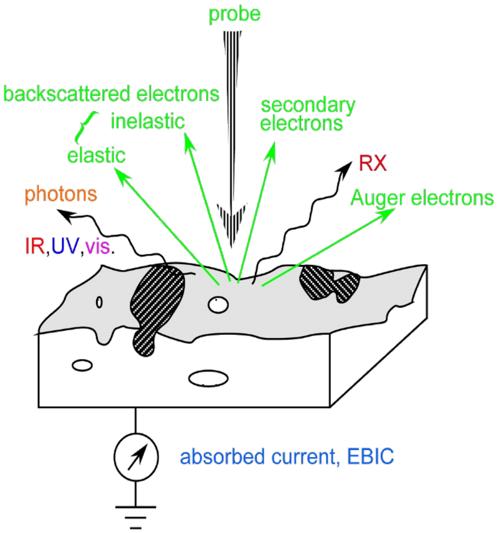
- Type of signal
  - Secondary and backscattered electrons
  - X-ray, Cathodoluminescence, etc.

- Where the signal is generated
  - Depth
  - Spatial resolution

- How the signal is detected
  - Detector position/type → Signal selection
  - Energy/type selection → Affects spatial resolutions

SEM signals | Electron signals

Incident electrons interaction with the sample produces:



Electron signals:

Secondary electrons SE

Electrons ejected from the conduction or valence bands of the target atom: low energy ≈ 5-50 eV
Free electrons: not associated with a specific atom and contain no specific elemental information
Can only escape if they are near the specimen surface → **Topography**

Backscattered electrons BSE

Incident electrons that scatter (elastically or inelastically) and leave the sample
Energy range from 50 eV to an energy close to initial energy eV_0
Most backscattered electrons retain at least 50% of the incident beam energy.
Yield depends on the atomic number → **Z-contrast**

Auger electrons

If the electrons are ejected from an inner shell by the energy released when an ionized atom returns to the ground state, then these SEs are called Auger electrons.
Ejected electrons with an energy characteristic of target elements → **surface spectroscopy**
Not detected in conventional SEM

SEM signals | Other signals

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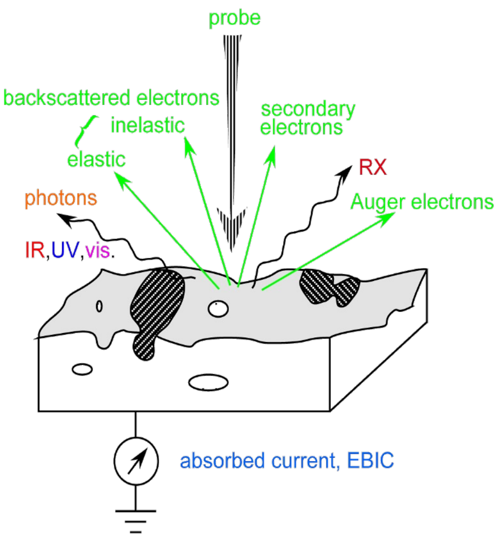
Incident electrons interaction with the sample produces:



Other signals:

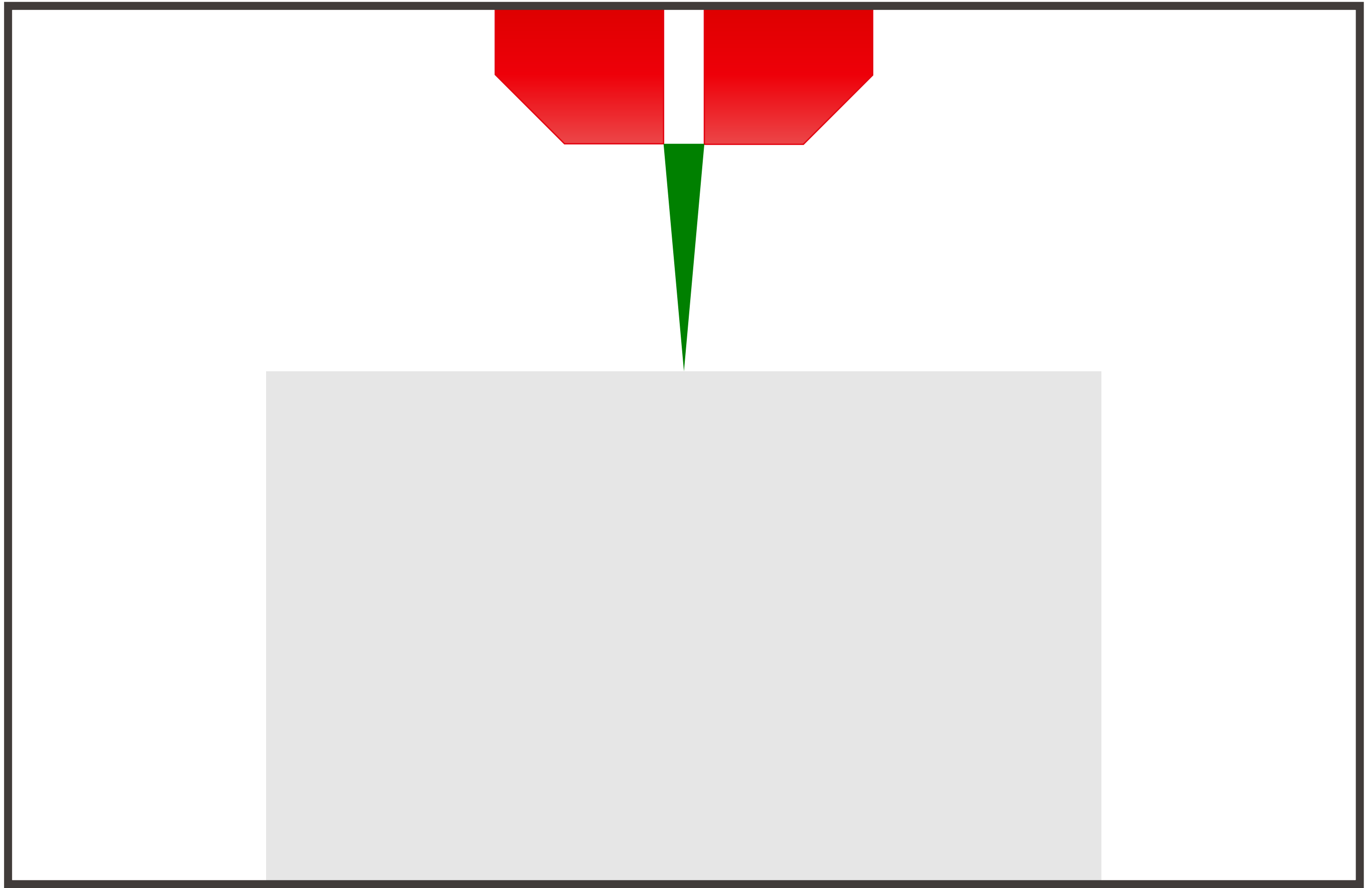
Electromagnetic radiations:

- X-rays with continuous energy, resulting from deceleration of incident electrons ("Bremsstrahlung" or “breaking radiation")
- Characteristic X-rays with a distinct energy associated to the target atoms.
- Cathodoluminescence: Visible radiation mainly emitted by insulating or semi-conducting materials.

Others:

- Absorbed current, electron-holes pairs creation, EBIC
- Plasmon
- Sample heating (phonons)
- Radiation damages: chemical bounding break, atomic displacement (*knock-on*) damage

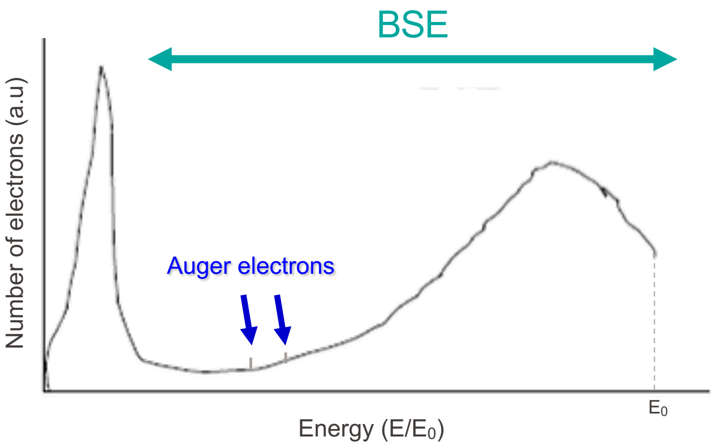
SEM signals | Where they come from

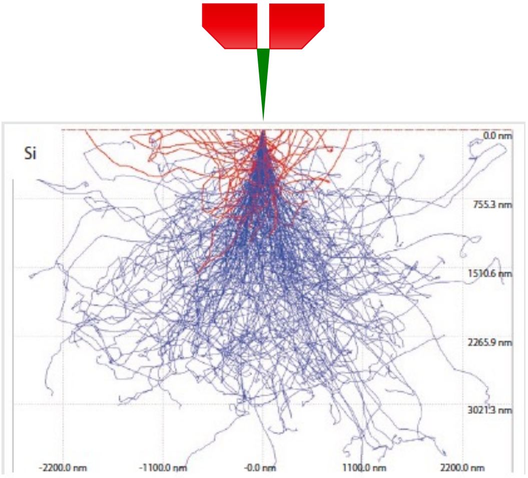


EPFL

SEM signals | And their energy

Energy spectrum of electrons leaving the sample





BSE: 50 eV < E_{BSE} < E_0

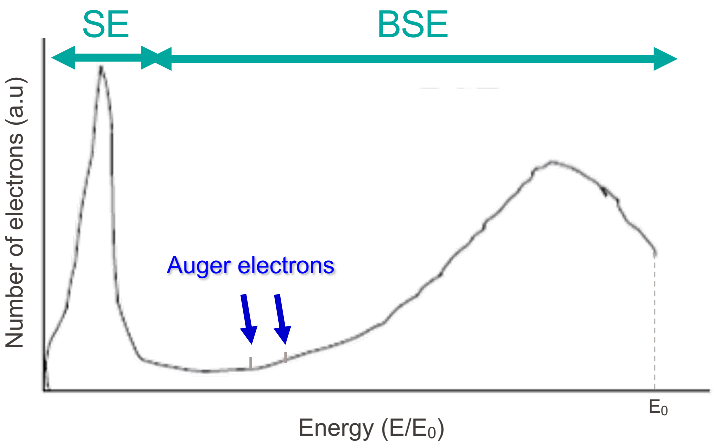
What is the source of the prominent peak in the low-energy region of the spectrum?

BSE energy range: BSEs follow trajectories which involve very different distances of travel in the specimen before escaping. The energy range for BSEs is thus wide (from 50 eV to that of the incident beams energy). The majority of BSEs, however, retain at least 50% of the incident beam energy (E_0).

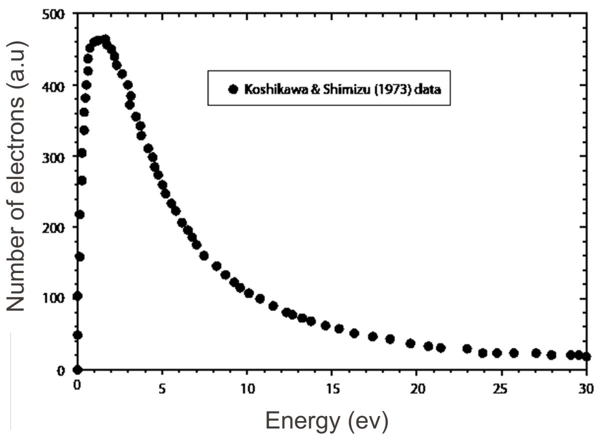
Generally speaking, higher atomic number elements produce a greater number of higher energy BSEs and their energy peak at the higher end is better defined.

SEM signals | And their energy

Energy spectrum of electrons leaving the sample



BSE: 50 eV < E_{BSE} < E_0

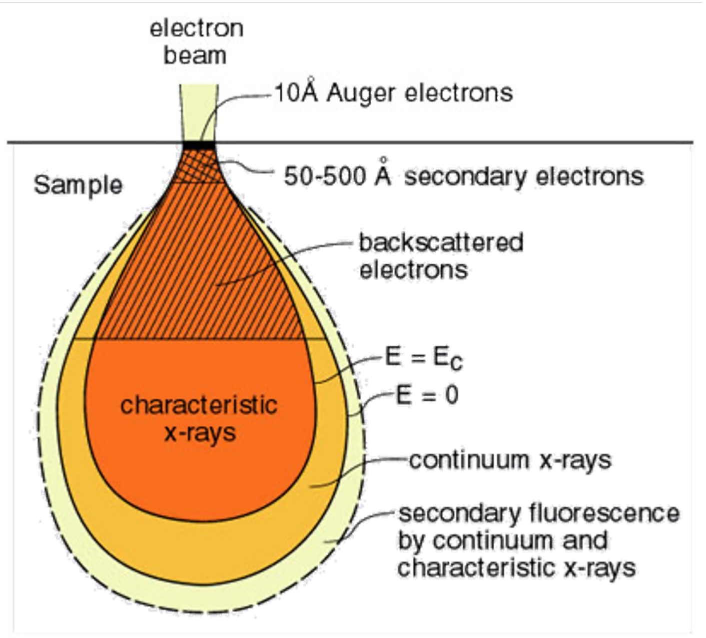


SE: E_{se} < 50 ev

The most important characteristic of SE is their extremely low kinetic energy, as the transfer of kinetic energy from the primary electron to the SE is relatively small. After ejection, the SE must propagate through the specimen while undergoing inelastic scattering, which further decreases their kinetic energy.

SE <u>are generated along the complete trajectory of the beam electron within the specimen</u>, but only a very small fraction of SE can reach the surface and escape → SE is mainly a Surface signal

SEM signals | Where they come from



Surface signals:

- Secondary electrons (topography)
- Auger electrons (electronic states, chemistry)

Sub-surface signals:

- Backscattered electron (Z contrast, crystallographic information)
- Characteristic X-ray (compositional information)
- Secondary florescence (Cathodoluminescence, band-gap)

Spatial resolution depends on the size of the interaction volume

Interaction volume differs with material, beam energy (depth and widening), and spot size (widening)

Monte-Carlo simulations (Details in MSE636)

SEM signals | Where they come from

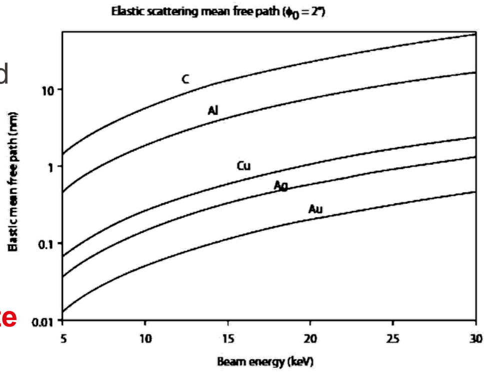
The higher the beam energy,

- The longer the scattering MFP, so the less the rate of energy loss with distance travelled
  Electrons entre the specimen with more energy and lose it at lower rate.

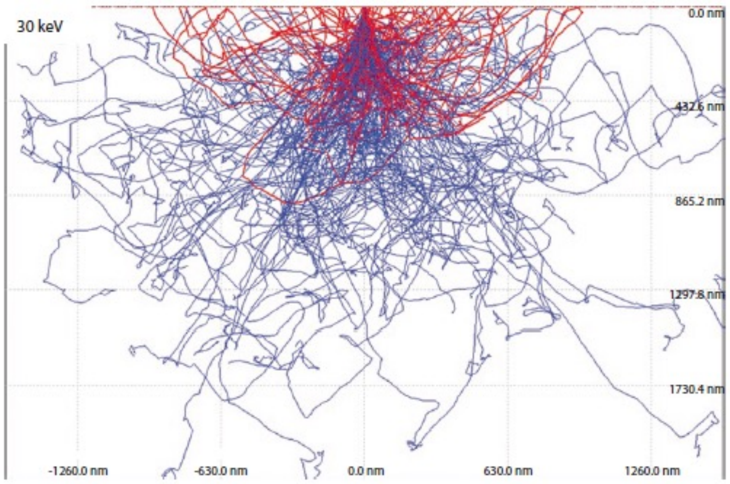
- The trajectories near the surface become straighter,

So, the more the penetration depth (i.e. larger interaction volume)

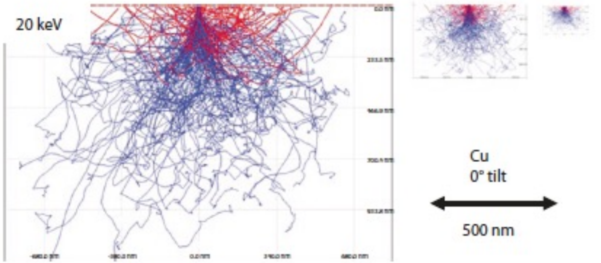
Cumulative effects of multiple elastic scatterings cause some electrons to propagate back towards the surface, thus widening the interaction volume.



30 keV



20 keV　10 keV　5 keV



Blue trajectories = Primary electrons

Red trajectories = Backscattered electrons reaching the surface

MFP= Mean free pass

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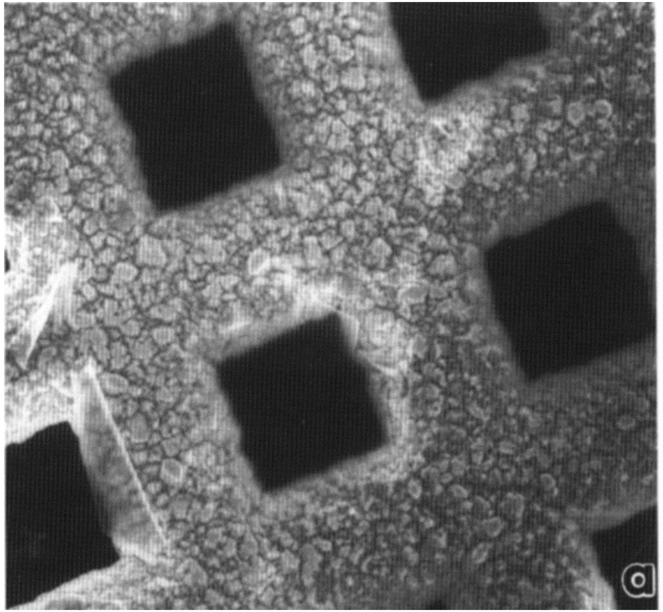
SEM signals | Where they come from

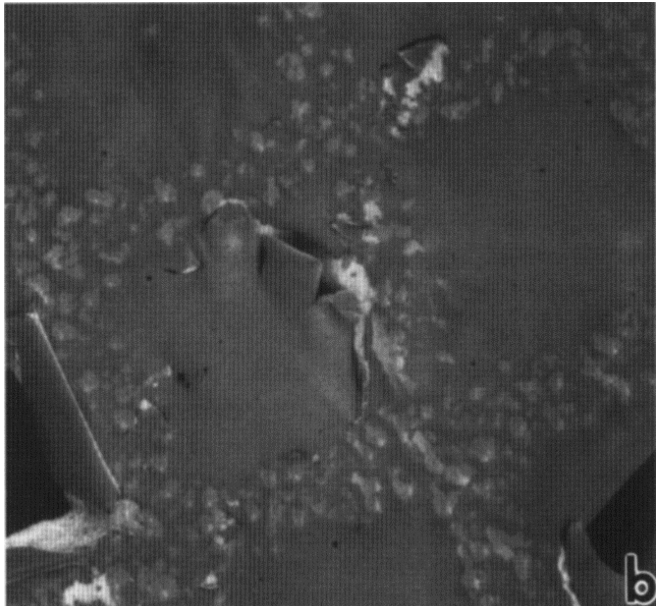
Effect of the accelerating voltage (beam energy) on penetration depth and signal

Example 1: Carbon foil on top of copper grid



20 kV:

Strong penetration:
It reveals the copper grid under the C film via the electron backscattering, but the structure of the film itself is hidden



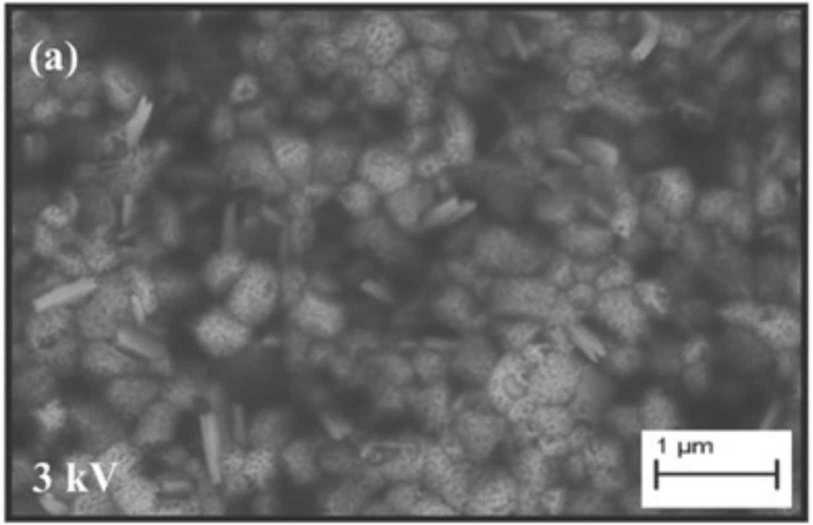
2 kV:

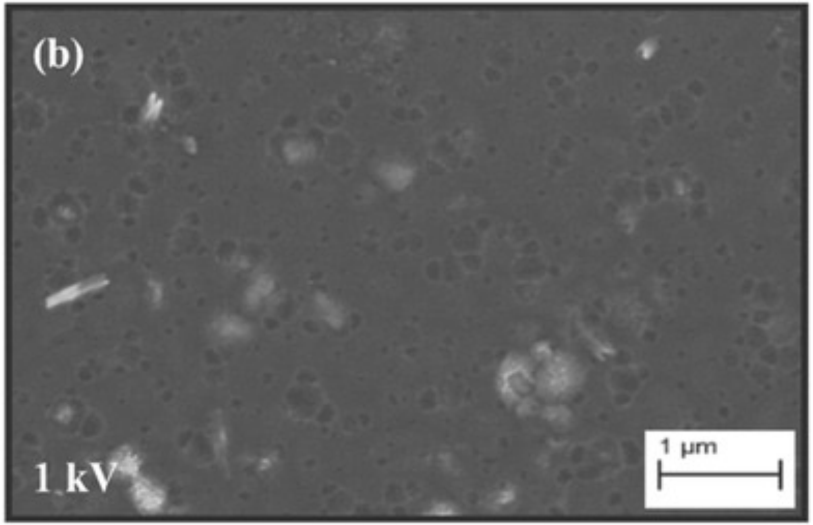
Low penetration,
Only a few electrons reach the copper grid and most of the signal is produced in the C film. The C film and its defects become visible

SEM signals | Where they come from

Effect of the accelerating voltage (beam energy) on penetration depth and signal

Example 2: Top surface images of a TiO2/perovskite/FA-CN device





3 kV:

It reveals the perovskite grains, but the structure of the covering HTM layer (FA-CN) is not visible.

1 kV:

Only a few electrons reach the peroskite layer and most of the signal is produced in the HTM layer. The HTM layer is visible

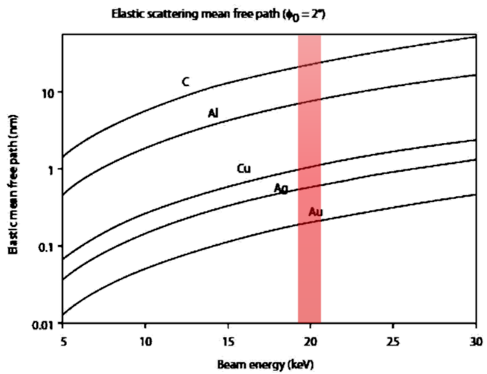
Sample courtesy of S. Paek , EPFL. *S. Paek et al. Advanced Materials (2017).*

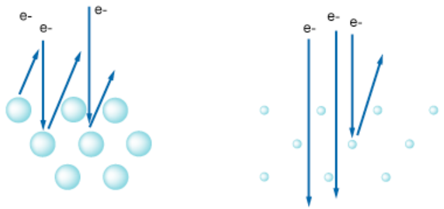
SEM signals | Where they come from

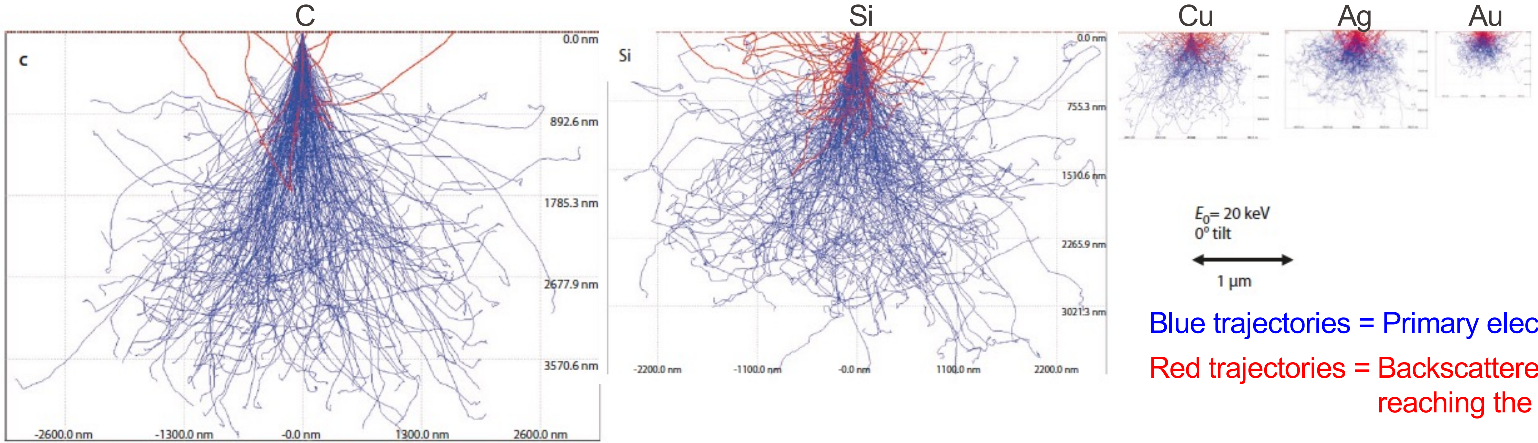
The higher the atomic number "Z",

- the more the probability for scattering (elastic and inelastic)
  (i.e. shorter scattering mean free path)

So, the shorter the penetration depth (i.e. smaller interaction volume).







Blue trajectories = Primary electrons

Red trajectories = Backscattered electrons reaching the surface

SEM signals | Where they come from

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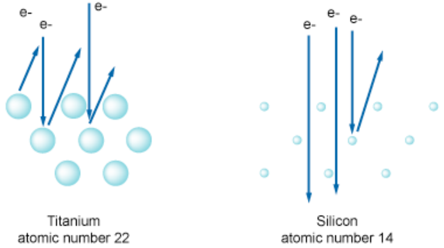

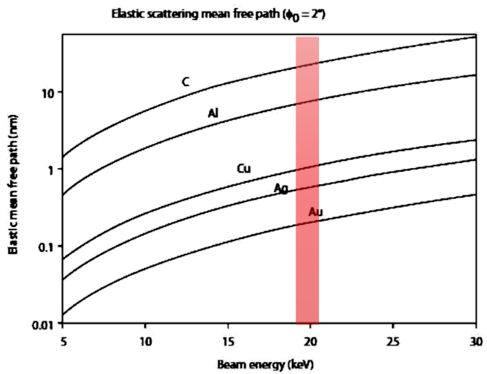

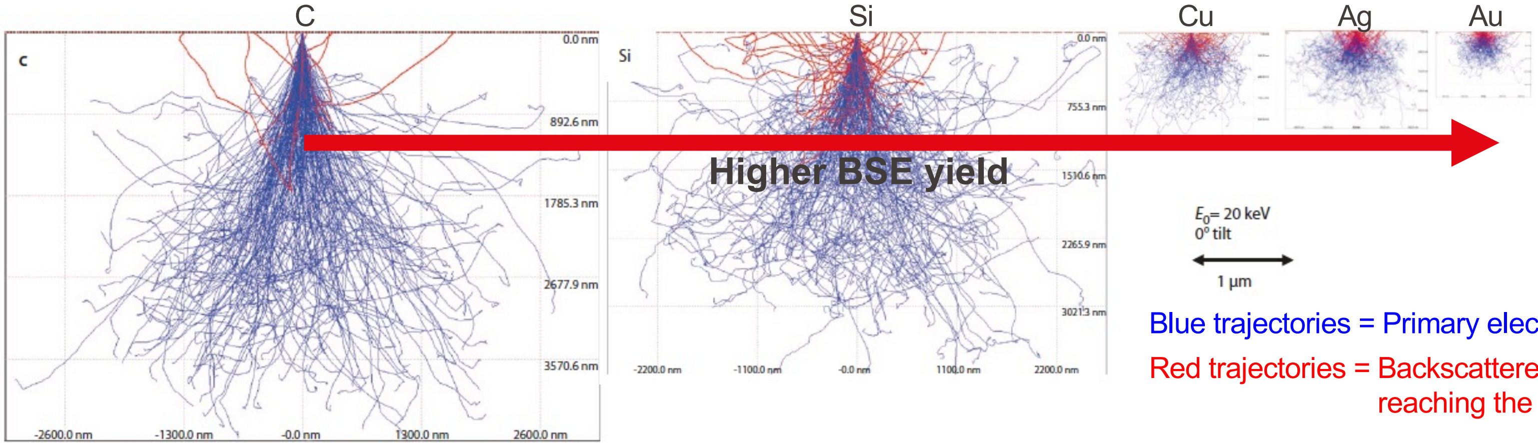

Blue trajectories = Primary electrons

Red trajectories = Backscattered electrons reaching the surface

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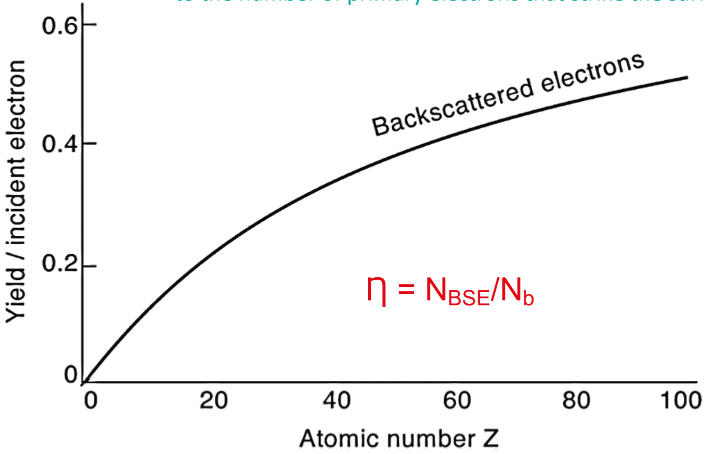
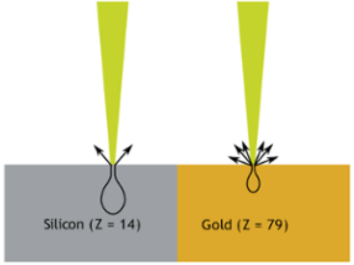
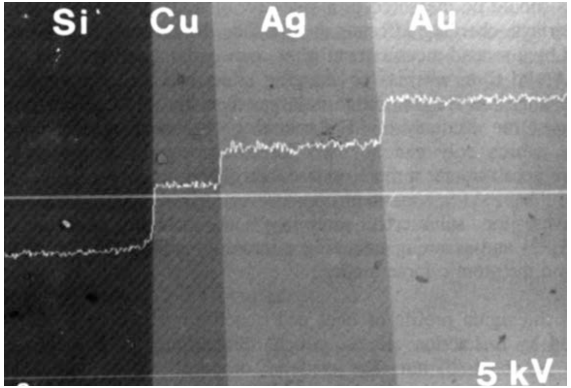
SEM signals | Backscattered electrons

BSE yield: The ratio of the number of BSE emitted from a surface to the number of primary electrons that strike the surface



$\eta = N_{BSE}/N_b$

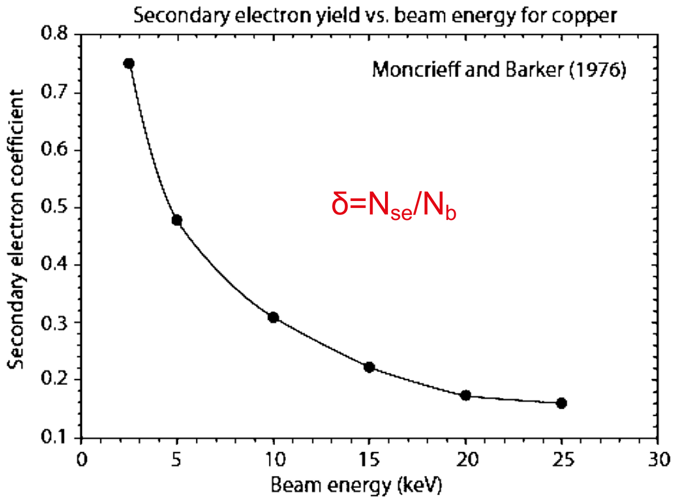


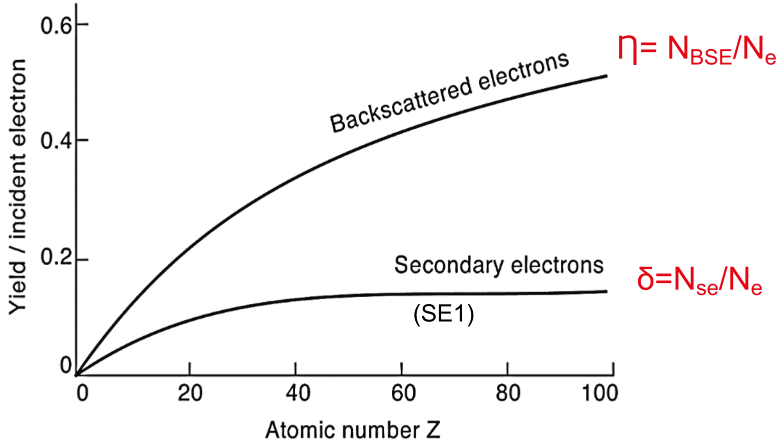


BSE yield η increases with "Z"

- η shows a monotonic increase with atomic number: Areas of the specimen composed of higher atomic number elements emit more backscatter signal and thus appear brighter in the image. **This relationship forms the basis of atomic number (Z) contrast imaging with BSE.**
- Z contrast is relatively stronger at lower atomic numbers (see the slope of the line).

SEM signals | Secondary electrons





There is a general rise in δ as the beam energy is decreased, primarily due to the reduction in the interaction volume.

δ is generally regarded as being independent of Z. If there is any (still a matter of debate) then it is very small.

BSE yield increases monotonically with the "Z" of material, and SE yield depends mainly on the beam energy

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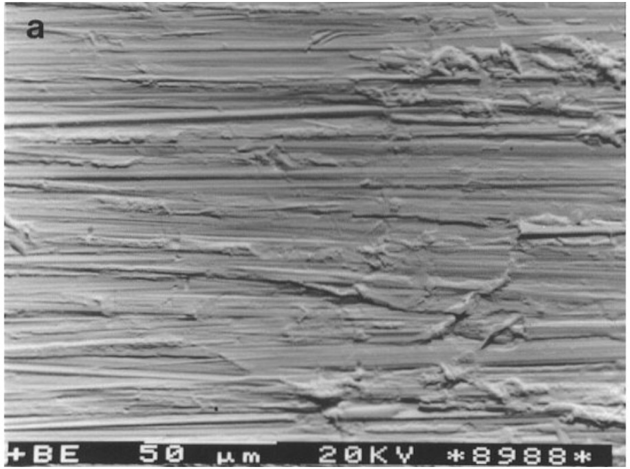
SEM signals | BSE vs SE imaging

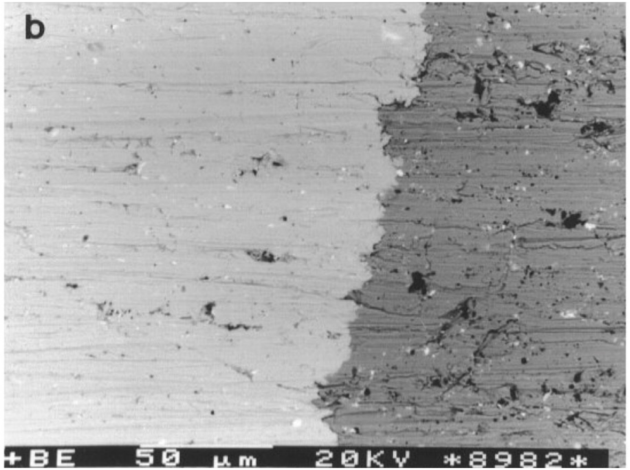
Example1 : Scanning electron micrographs of Mo-Cu weld interface

SE image



SE have low energies (5-50eV), and thus are **emitted** only from surface and possess information about topographical features.

BSE image

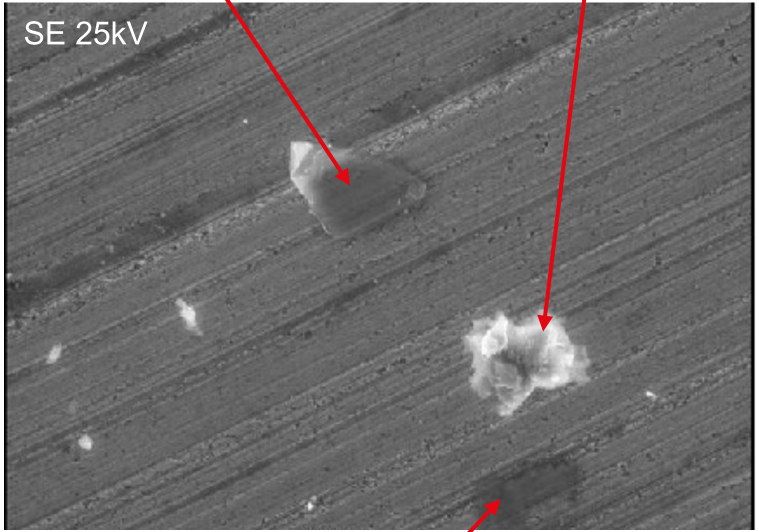


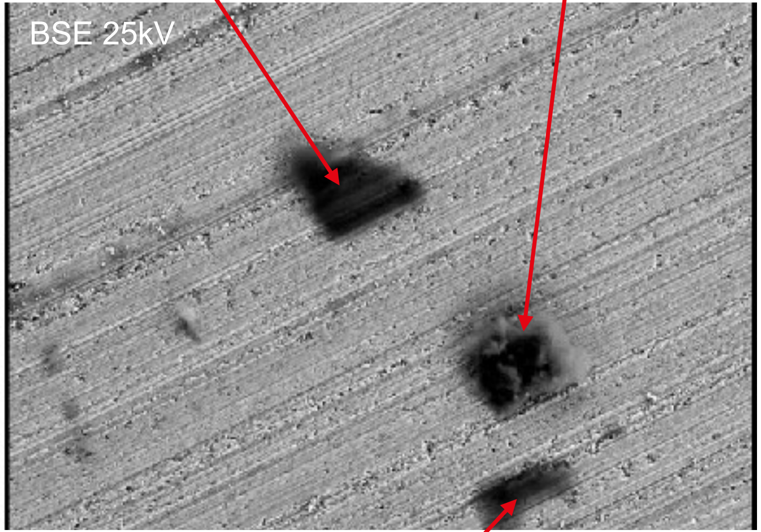
BSE emission depends on “Z”, thus intensity in the BSE images scales with atomic number and depends on local composition

Scanning Microscopy Vol. 12, No. 1, 1998 (Pages 161-169)

SEM signals | BSE vs SE imaging

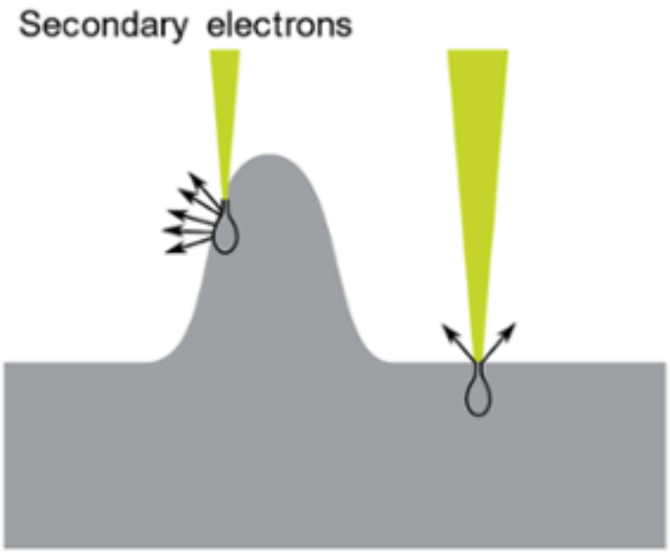
Example 2: Dust on WC (different Z materials)





Why the rough particle appears brighter in the SE image?

SEM signals | Effect of sample roughness on SE yield



- If a sample is titled, the interaction volume is tilted and is closer to the surface. Thus, more SE can escape from below the surface, giving higher signals received by the detector.
- The same principle is true for rough surfaces: Sloped surfaces and edges have an interaction volume that is effectively titled and have higher SE yield.

Rough surfaces generates higher SE image signal.

Titling the specimen can change SE image contrast.

Imaging parameters

Parameters affecting Resolution (and Visibility)

- **Fundamental**
  - Electron wavelength (beam energy) and diffraction limit: → Rayleigh criterion
  - Aberrations: enlarges the probe size

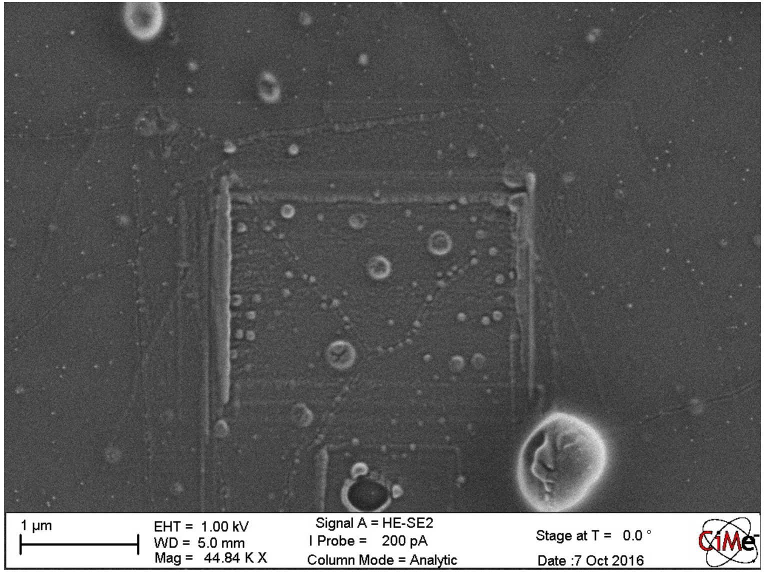
    Probe size (or spot size) means the diameter of the final beam at the surface of the specimen

- **Operational and sample**
  - Pixel size = scan step size
  - Probe size (also defines probe current and affects visibility)
  - Visibility:
    - Scan speed (i.e. dwell time) and “signal to noise ratio”
    - Contrast
  - Type and depth of emitted electrons signal
  - System/Specimen stability → Challenges (charging, contamination, beam damage)

Challenges | Beam damage

Beam induced changes to the sample:

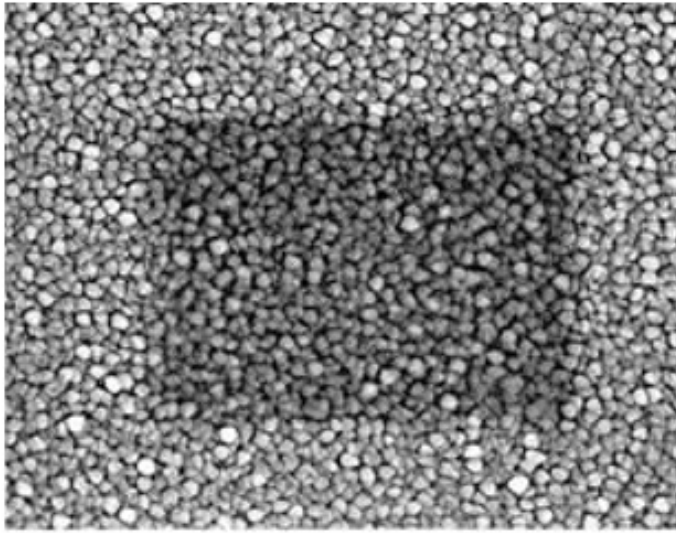
- Atom displacement ("knock on")
  - Radiation damage,
    More severe for high beam energies
- Chemical bound breaking
  - Radiolysis
    Increasing beam energy can lessen radiolysis
- Lattice atom vibrations (phonons)
  - Sample heating



Challenges | Contamination

Hydrocarbon build-up on surface

- Masks surface features and information about the sample



- Sources:
  - Sample surface
  - SEM chamber
  - Beam induced degradation and migration of sample compounds
- To avoid / minimize:
  - Use gloves when handling samples
  - Plasma cleaning sample prior to observation

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Challenges | Charging

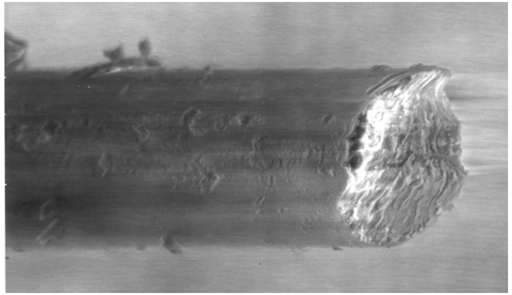
Accumulation of negative charge

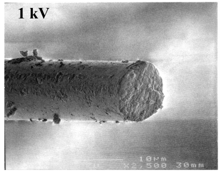
Occurs in insulating samples (also in samples that are not well grounded)

- Charging deflects the low-energy secondary electrons (mainly, but can affect BSE) causing image distortions and contrast changes

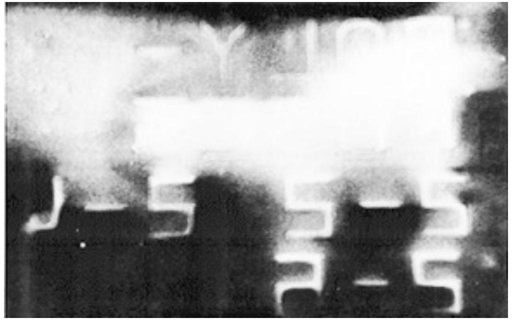
- Depends on:
  - Material properties (surface resistivity)
  - Beam energy and current
  - Scan rate

- Ways to mitigate charging
  - Coat the sample with a conductive layer
  - Work at low kV
  - Use low currents (noisy images)
  - Use low-vacuum mode
  - Charge compensation devices
  - Use the "magic" charge neutrality voltage





Fiberglass



SiO_2 substrate

SEM techniques | Environmental SEM

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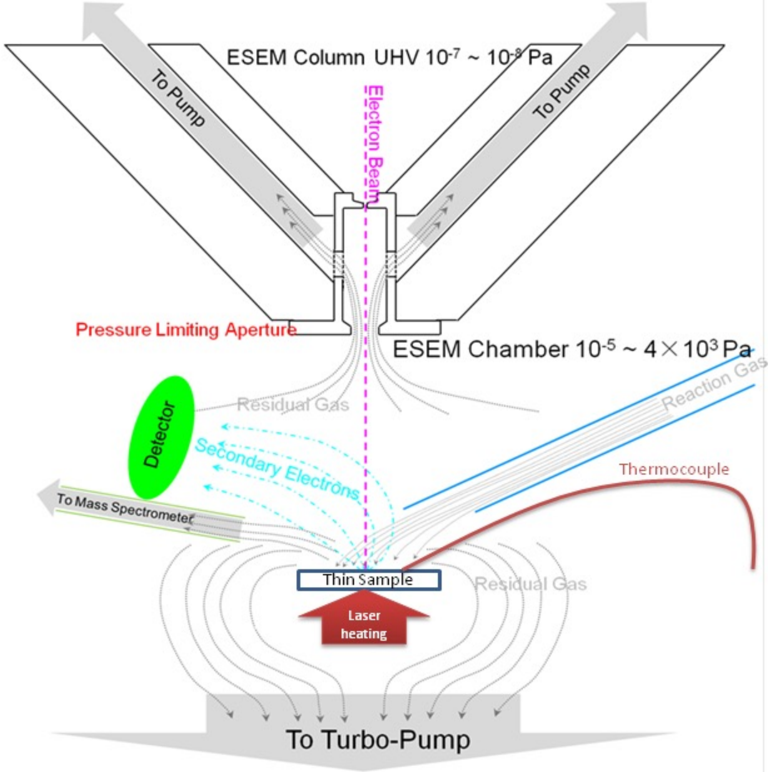
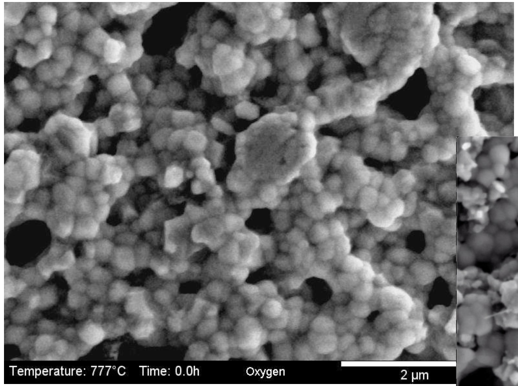
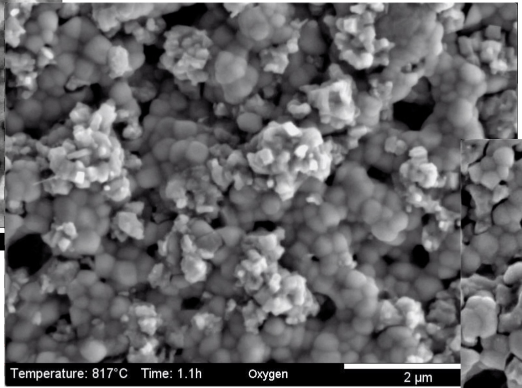


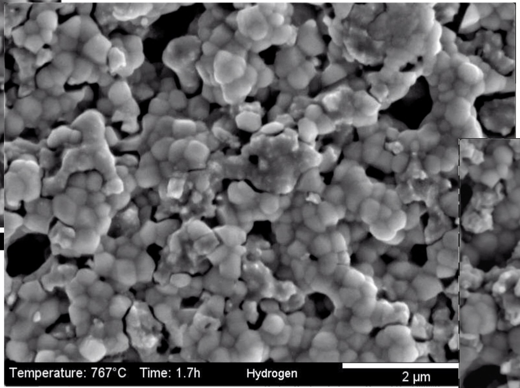
Modified Quanta200:
- Available gases (mass flow controller) H_2, O_2, N_2, CO, NH_3, C_2H_4
- Pressure up to 800Pa (8mbar)
- Temperature up to 1000°C
- QMS Mass Spectrometer

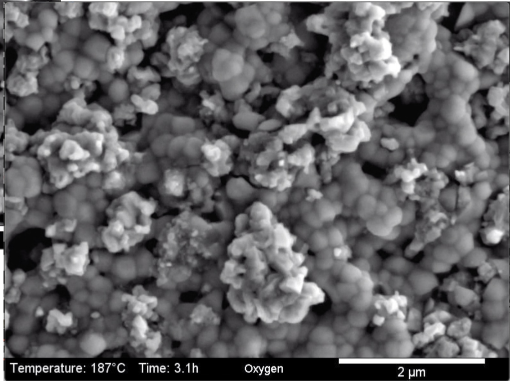
SEM techniques | Environmental SEM

Example: SOFC anode under Redox cycling









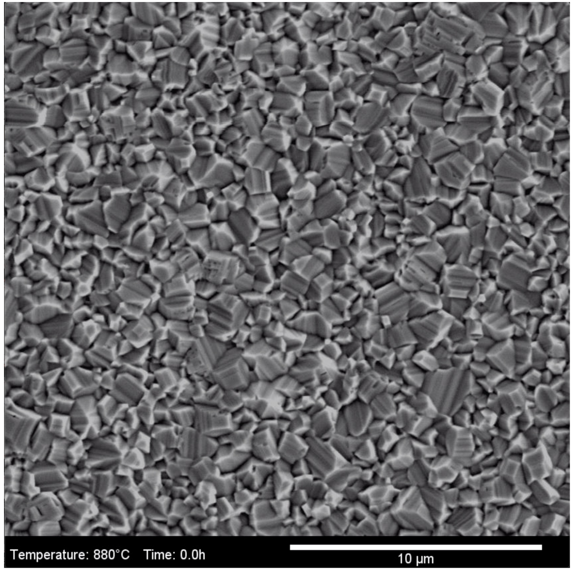
Redox Cycling between O_2 and H_2 at 800°C

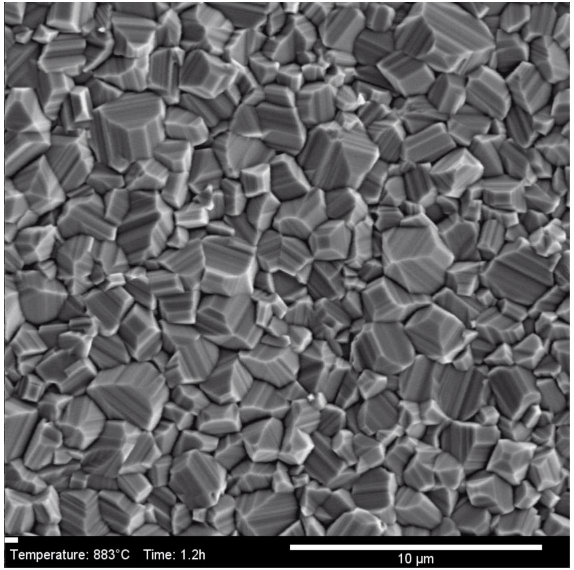
Sample: SOFC anode made of YSZ (Yttrium Stabilized Zirconium) and Ni (which is reduced and oxidized at each cycle)

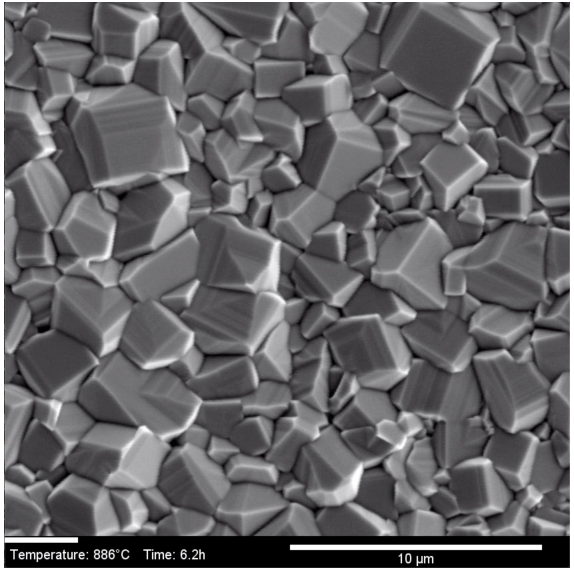
Slide courtesy of S. Poitel, LSME-EPFL

SEM techniques | Environmental SEM

Example: SOFC coated interconnect oxidation





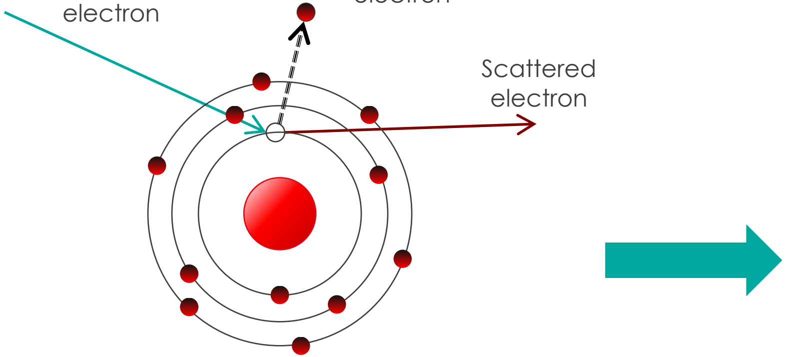


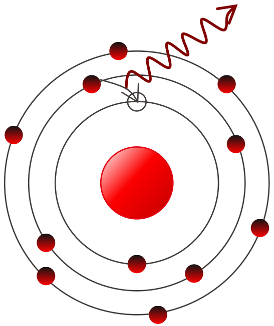
48 hours oxidation under 33 Pa of oxygen at 880°C

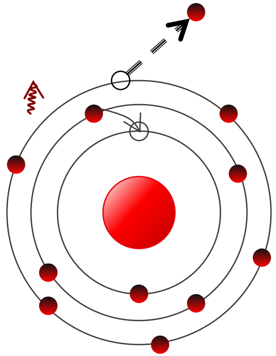
Sample: SOFC interconnect made of steel and coated with Co and Cerium.

Slide courtesy of S. Poitel, LSME-EPFL

SEM techniques | Energy dispersive x-ray spectroscopy







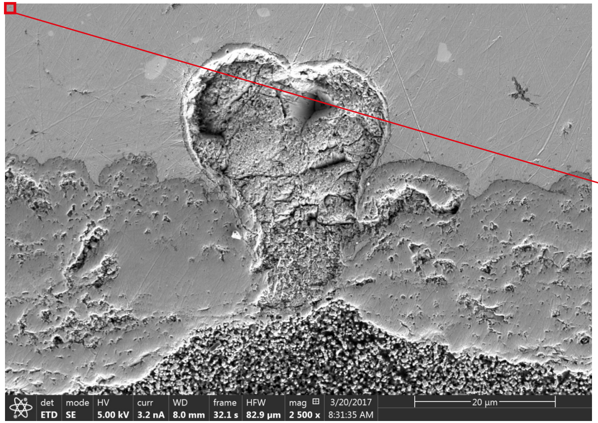
X-ray generation

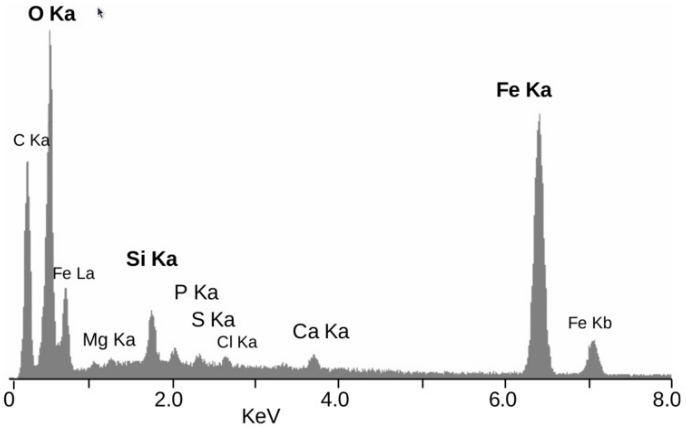
X-ray energy characteristic interorbital electron transitions and thus of the element

Auger emission

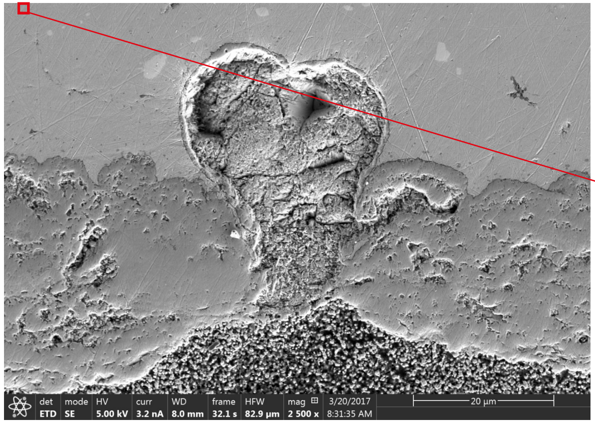
The relaxing process interacts with an electron with a characteristic energy

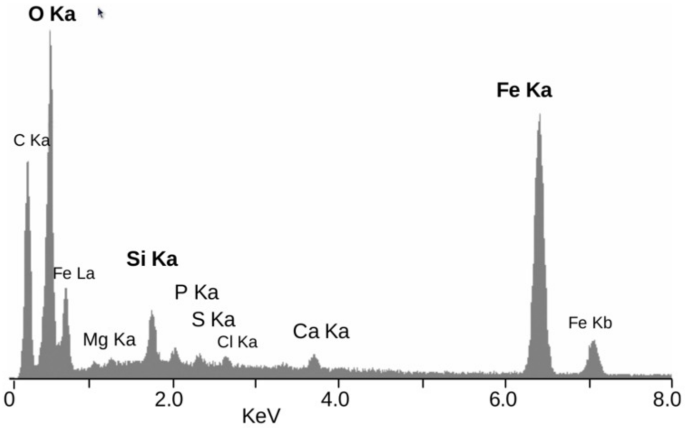
SEM techniques | Energy dispersive x-ray spectroscopy

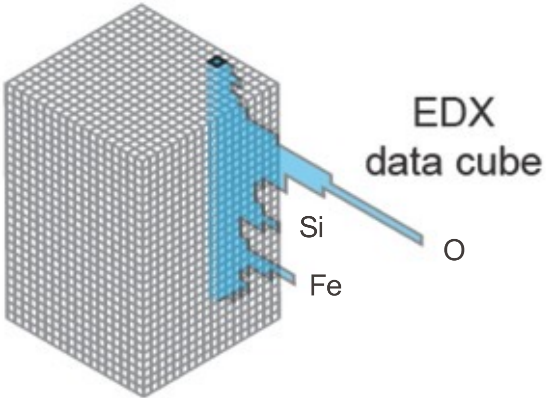




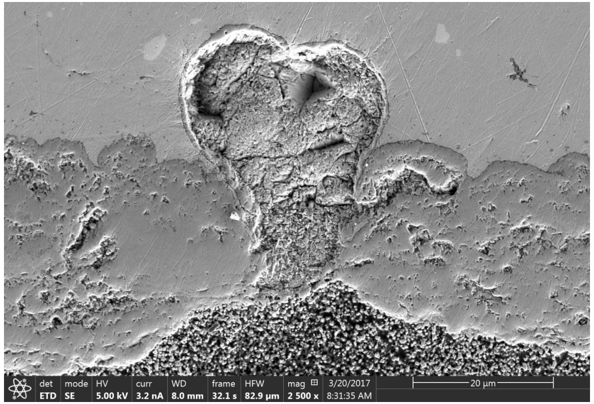
SEM techniques | Energy dispersive x-ray spectroscopy

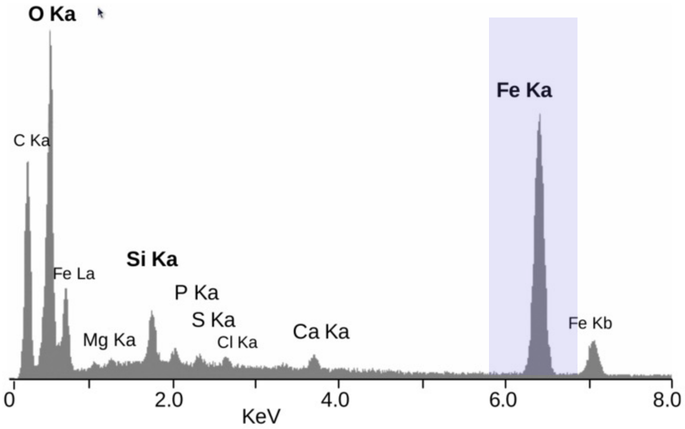


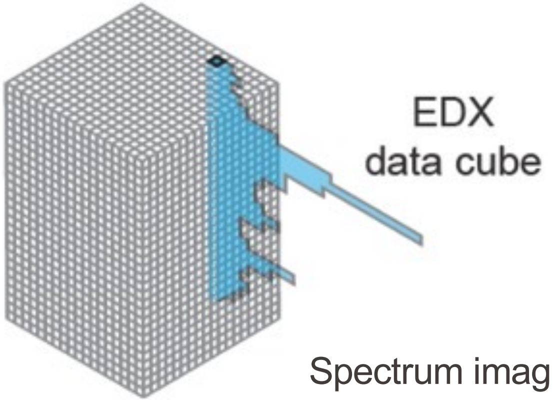




SEM techniques | Energy dispersive x-ray spectroscopy

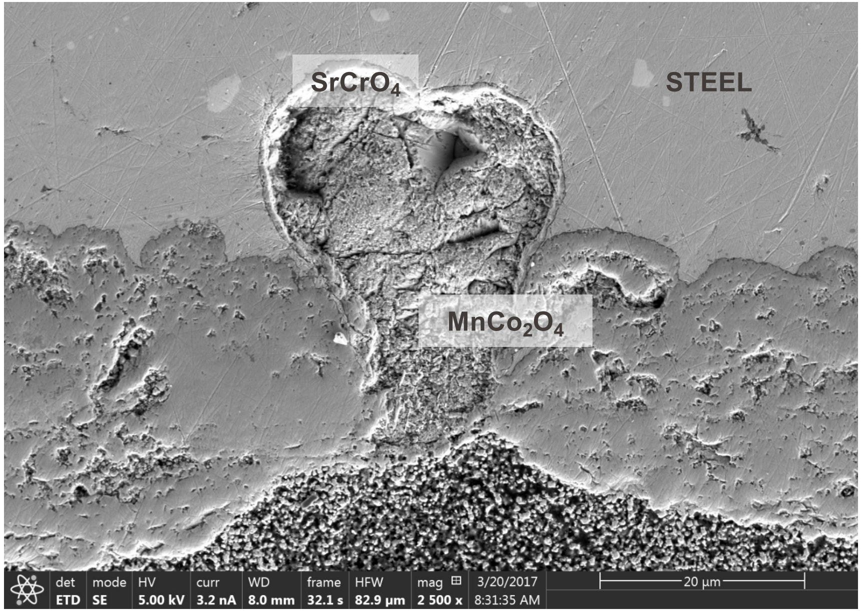


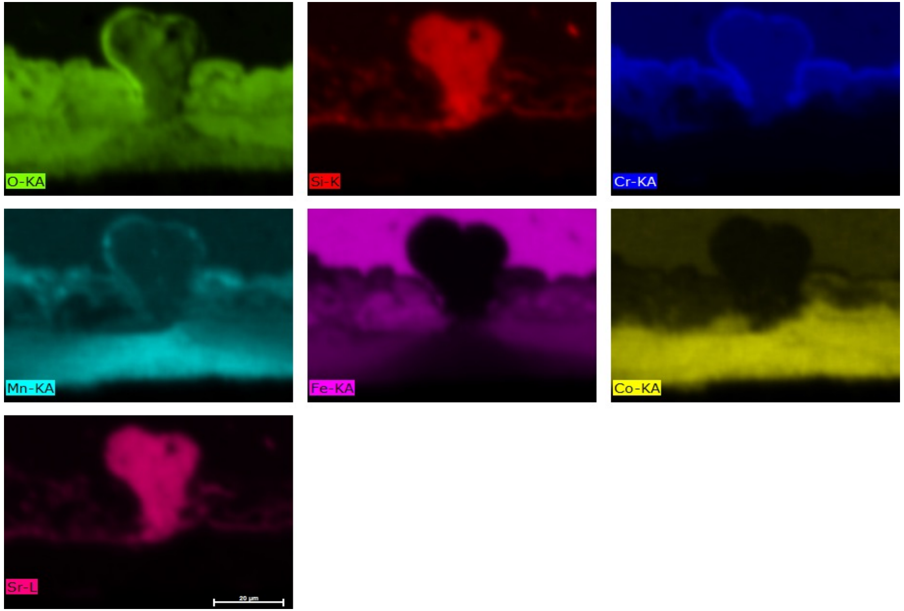




Spectrum imaging

SEM techniques | Energy dispersive x-ray spectroscopy





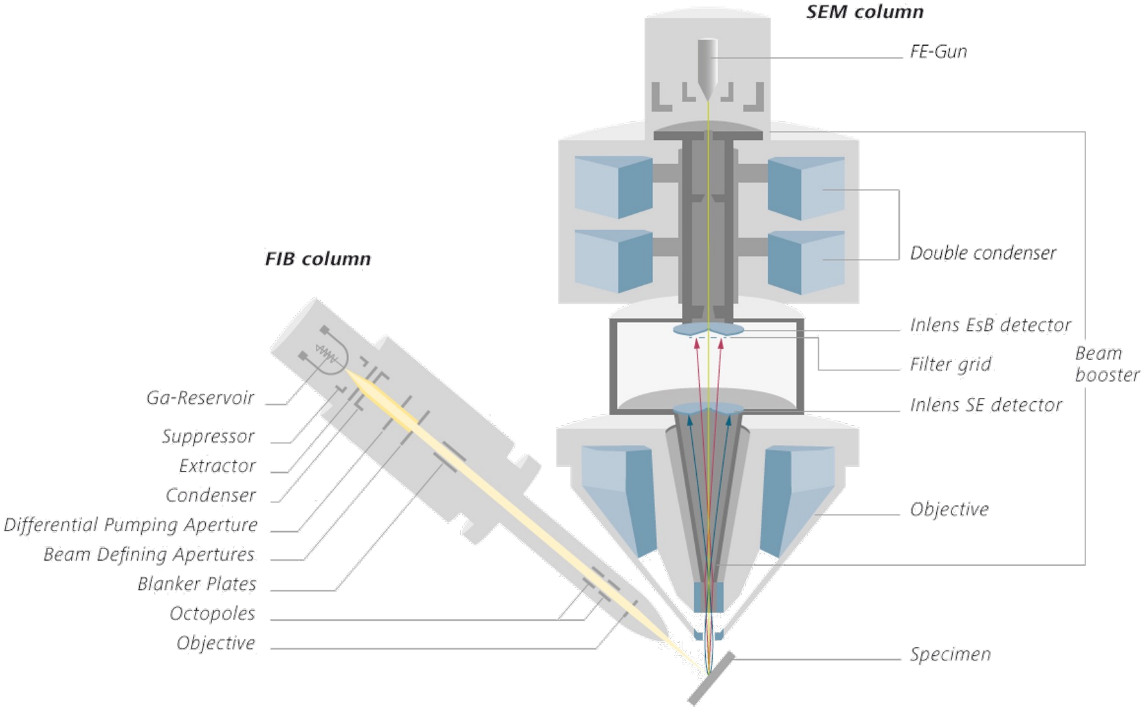
Strontium poisoning of stainless steel

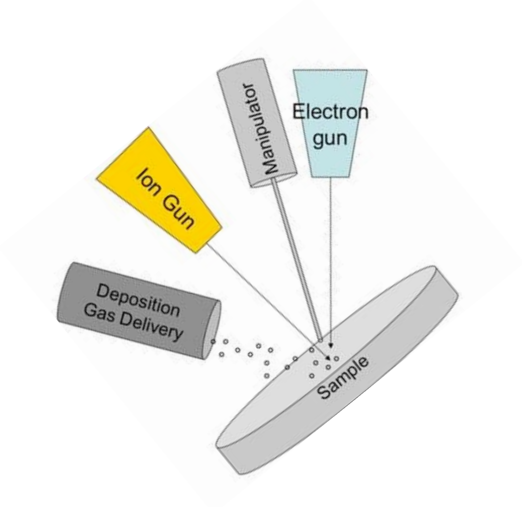
SEM techniques | Energy Dispersive x-ray Spectroscopy

Emad Oveisi

- From beryllium up to periodic table
- Sensitivity 0.1 wt.%
- Quantitate analysis (many requirements; e.g. flat sample)
- Point, line scan, and mapping
- To excite an EDX peak, the beam energy needs to be around twice the energy of the peak
- We should consider the interaction volume
- Interaction volume increases with beam energy
- About 130 ev energy resolution for Si(Li) detector

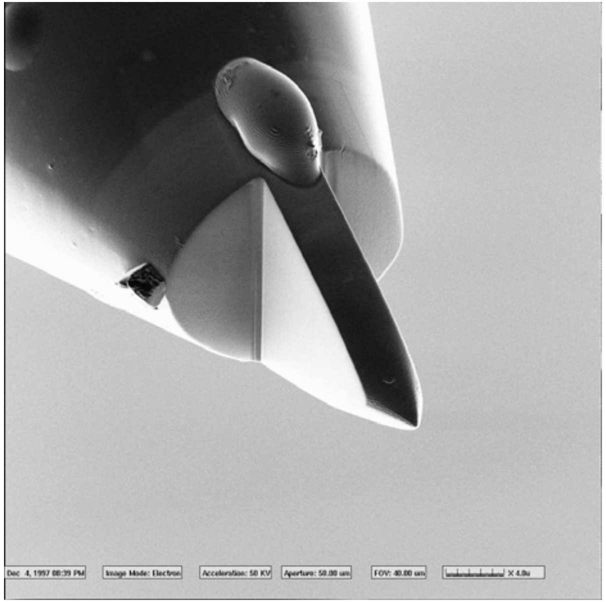
SEM techniques | Focused ion Beam

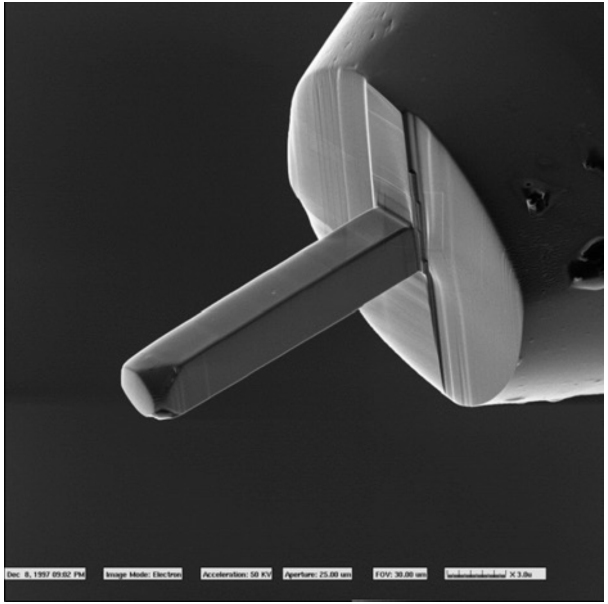




SEM techniques | Focused ion Beam

Nano fabrication

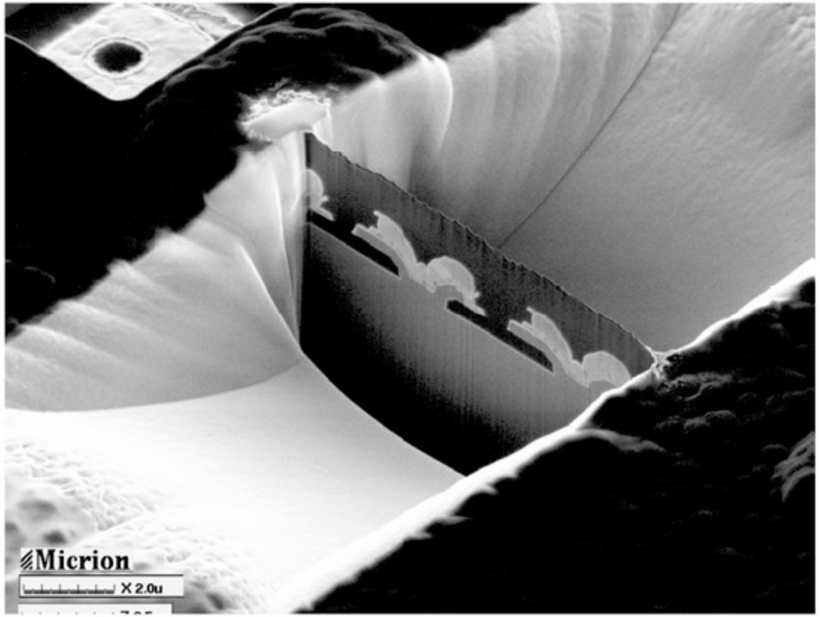




AFM tip

SEM techniques | Focused ion Beam

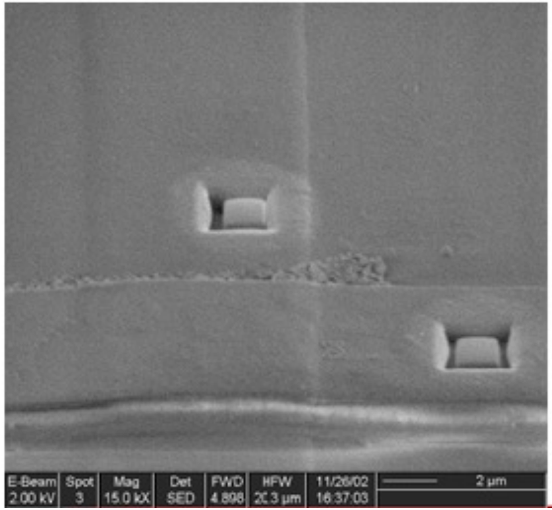
Cross-sectioning SEM imaging

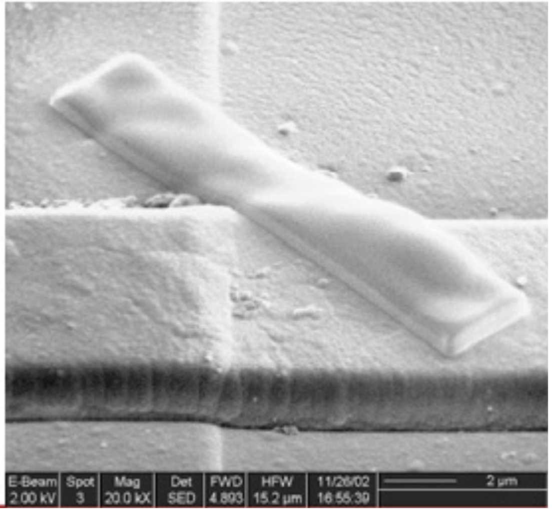


Failure analysis

SEM techniques | Focused ion Beam

Deposition





Insertion of electrical connection

SEM techniques | Focused ion Beam

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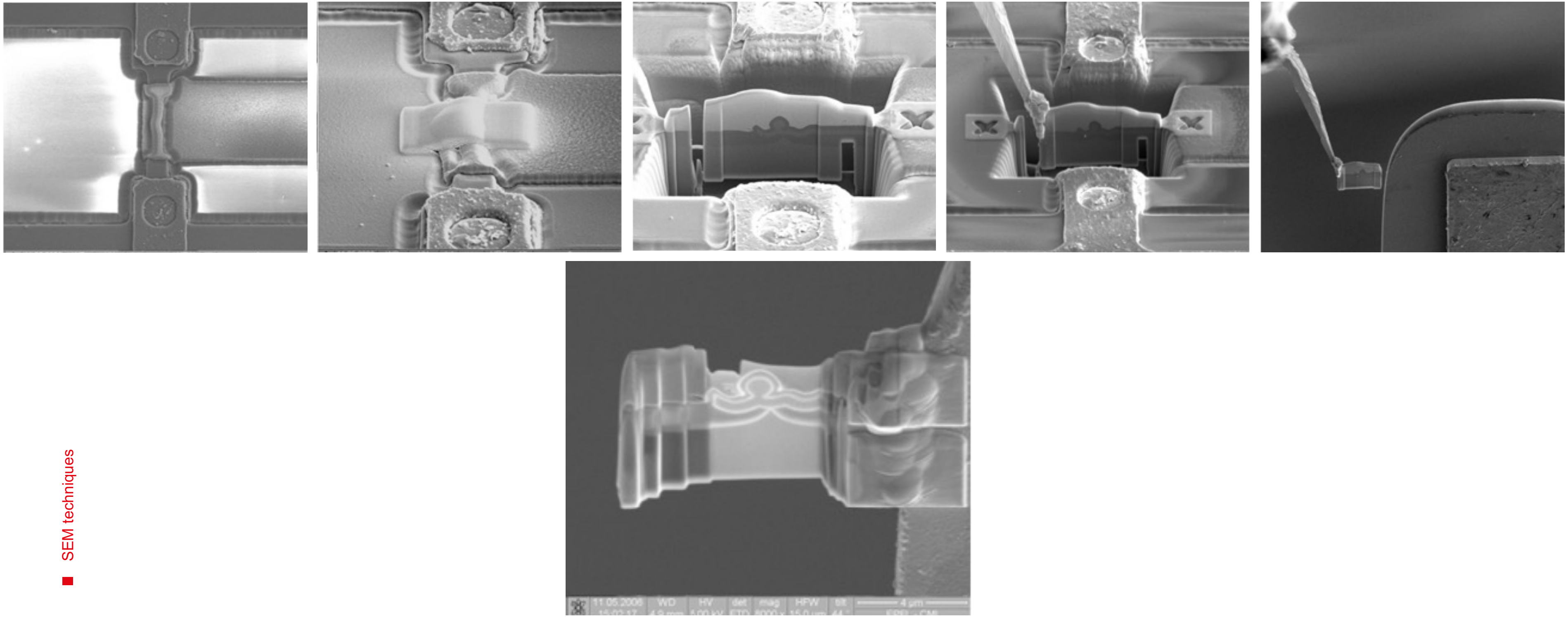
Site specific TEM lamella extraction



SEM techniques | Focused ion Beam

3D microscopy
(can be combined with other techniques, e.g. EDX, EBSD, …)

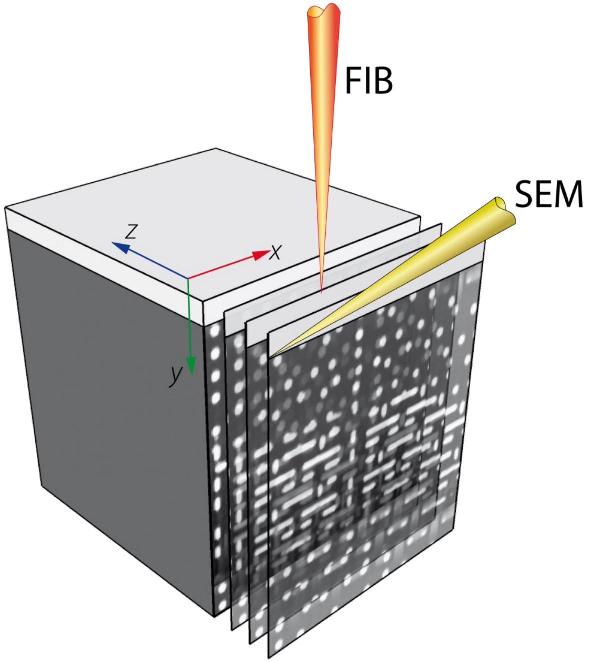
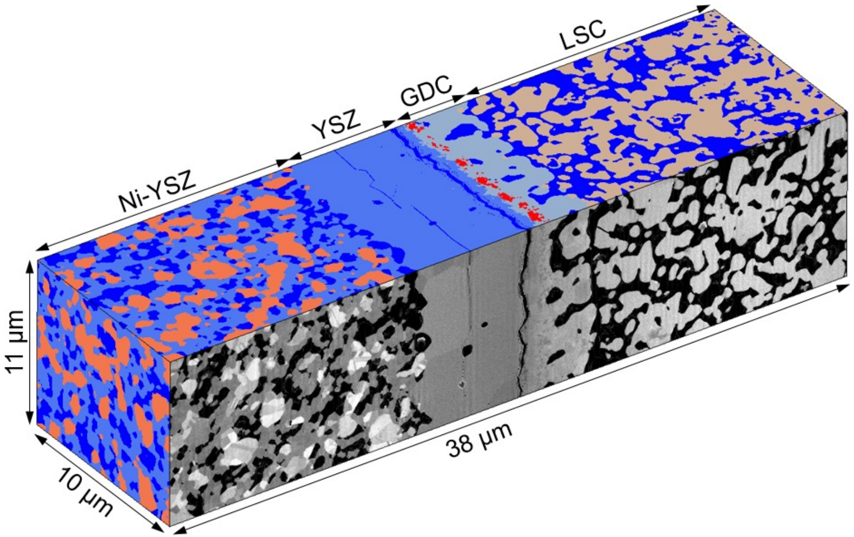


Photo from http://www.tescan.com/

SEM techniques | Focused ion Beam

3D microscopy: SEM/FIB + EDX



Sample: Multilayer of heterogeneous materials, e.g. Ni-YSZ/YSZ/GDC/LSCF-GDC

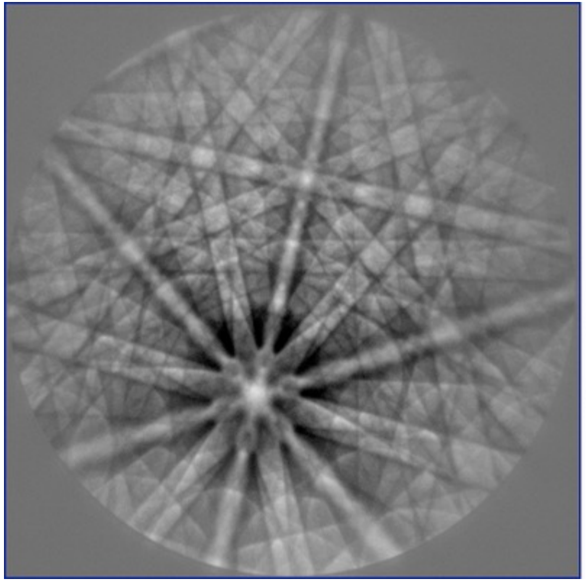
Studying degradation of fuel electrode/electrolyte interface, micro-cracking/delamination.

Courtesy of A. Nakaju, EPFL

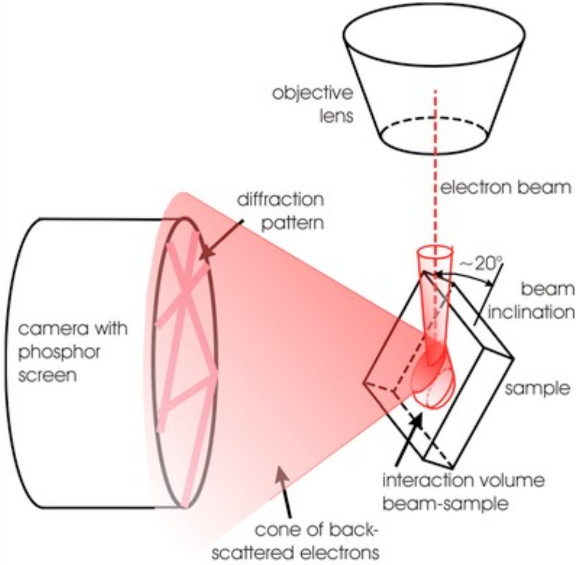
SEM techniques | Electron backscatter diffraction - EBSD

Emad Oveisi

Electron backscatter diffraction is an electron diffraction technique in SEM used to obtain information about crystallographic phase, crystal orientation and defect densities.



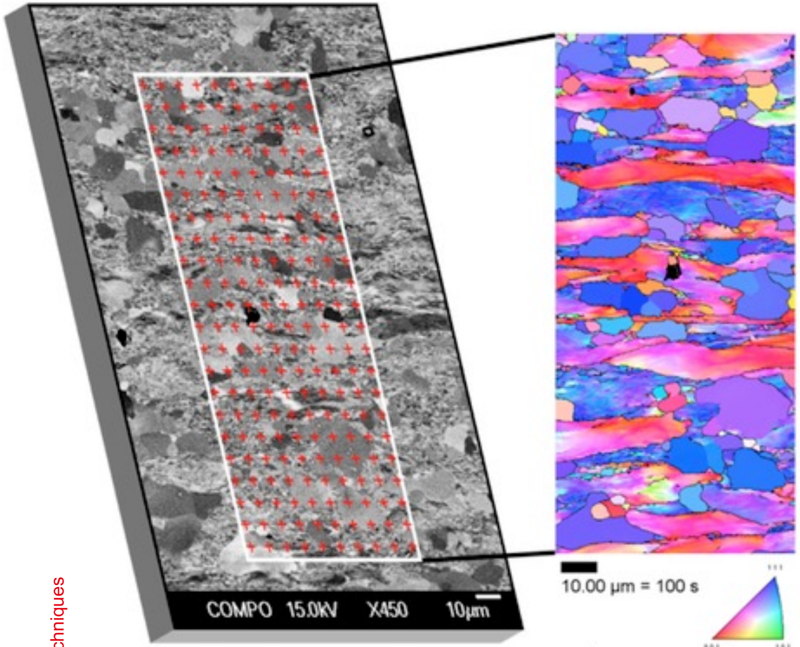
EBSD pattern of Nb (15 kV)



Geometric set-up of an EBSD measurement

SEM techniques | EBSD-based orientation mapping

Measures crystal orientations on a regular grid by electron diffraction. The so-determined data are used to produce orientation (or phase maps) of the scanned area on the sample.



BSEI of a partially recrystallised IF steel

inverse pole figure map

Lateral resolution: 20 to 100 nm (depending on the sample material).

Measurement rate currently up to hundreds of patterns per second.

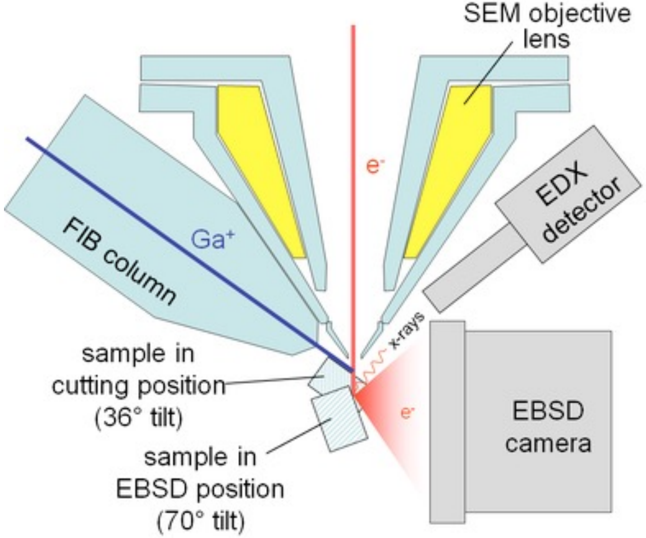
Investigated areas: few µm² to few cm².

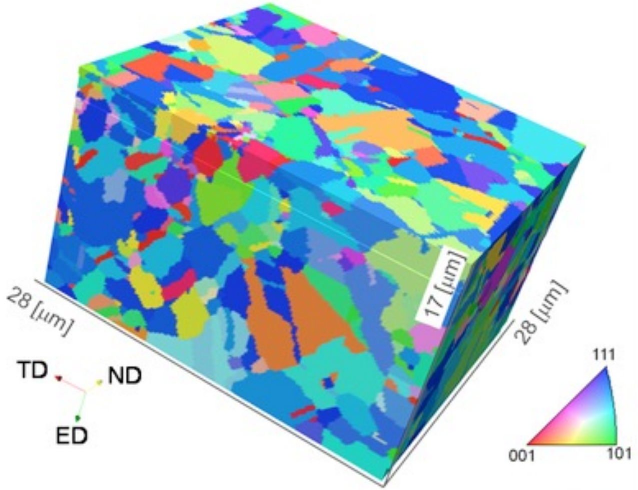
Angular resolution: 0.5° down to 0.01° with special techniques (→XR EBSD).

The technique can also be extended into a 3D technique (→ grain boundaries).

SEM techniques | 3D EBSD-based orientation mapping

3D orientation mapping of a severely deformed and recrystallized Cu-Zr alloy.





Colours correspond to the inverse pole figure position of the sample normal direction.

Resources

- Scanning Electron Microscopy and X-Ray Microanalysis, Springer, by Joseph Goldstein et al.

  Hardcopy at EPFL & CIME libraries

- Image formation in low-voltage scanning electron microscopy, Springer, by L. Reimer

  Available online

- Physics of image formation and microanalysis, Springer, by L. Riemer

- Optique: Fondements et applications, Dunod, by J.S. Perez