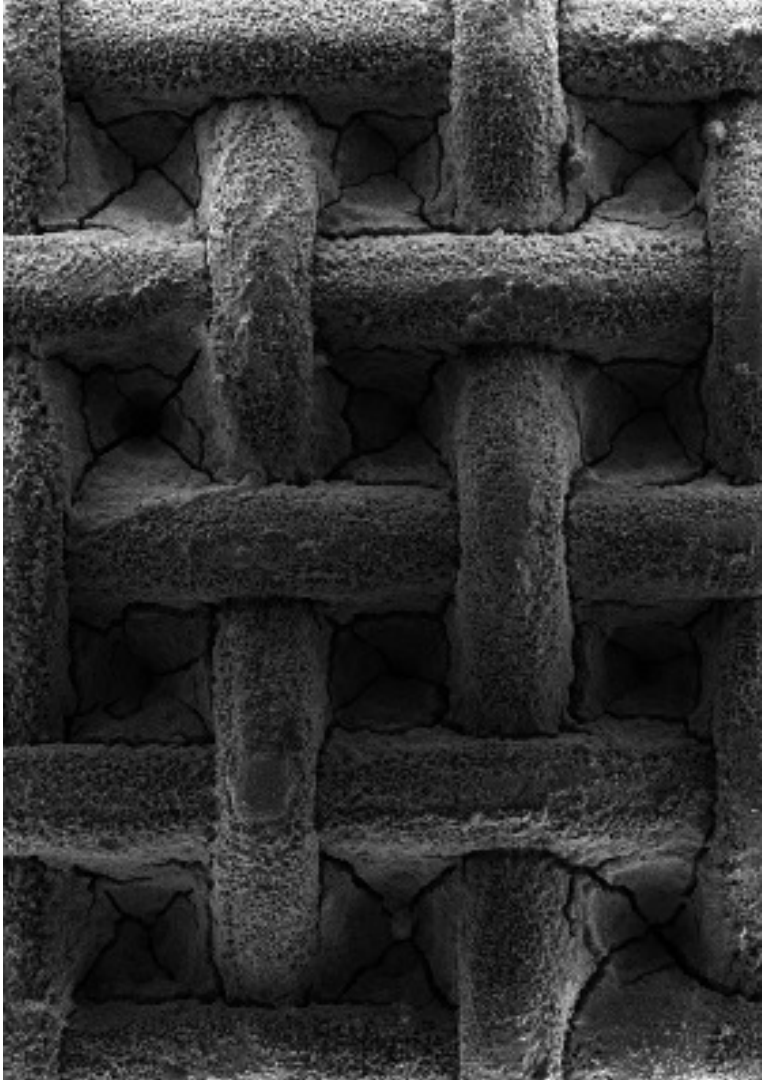


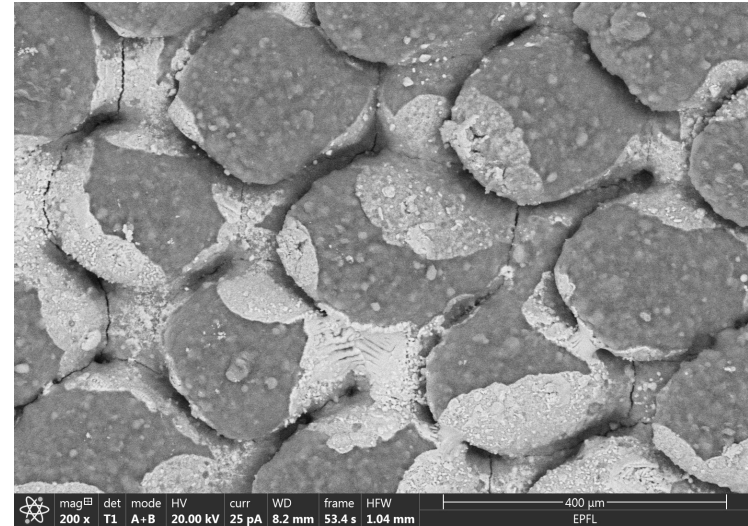
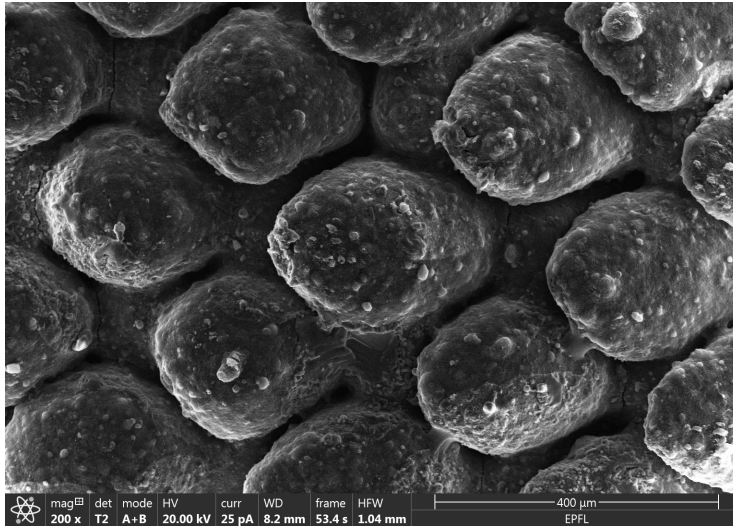


Scanning Electron Microscopy (SEM)

CH-633

How the SEM works?

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

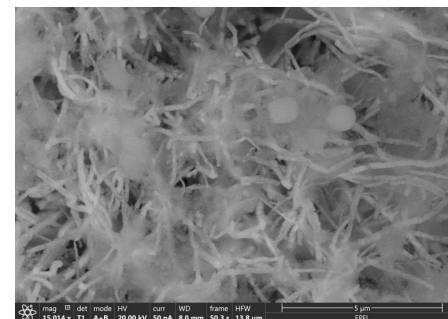
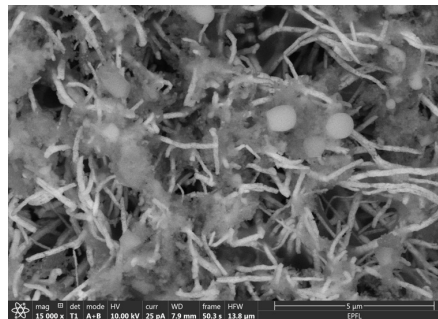


Why these two SEM images from identical locations are different?

Learning Outcomes

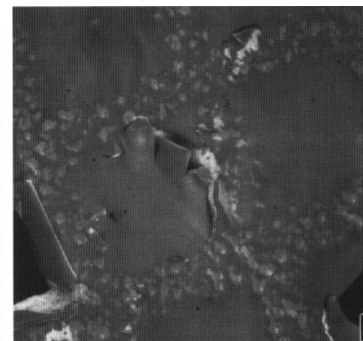
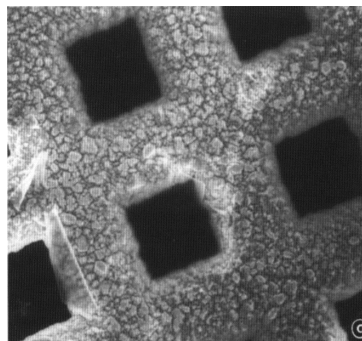
	mag 	det	mode	HV	curr	WD	frame	HFV	400 μm	
	200 x	T2	A+B	20.00 kV	25 pA	8.2 mm	53.4 s	1.04 mm	EPFL	

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?

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

Scanning Electron Microscope (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.

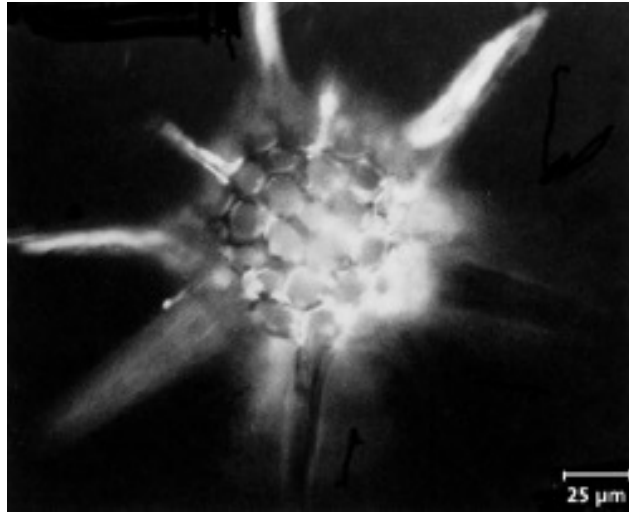


Coloured image of fruit fly mutant

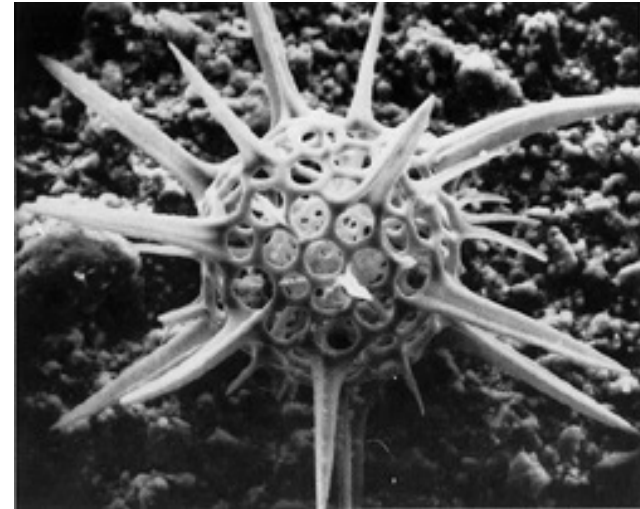
<https://www.sciencephoto.com>

Why fast electrons?

Light microscope



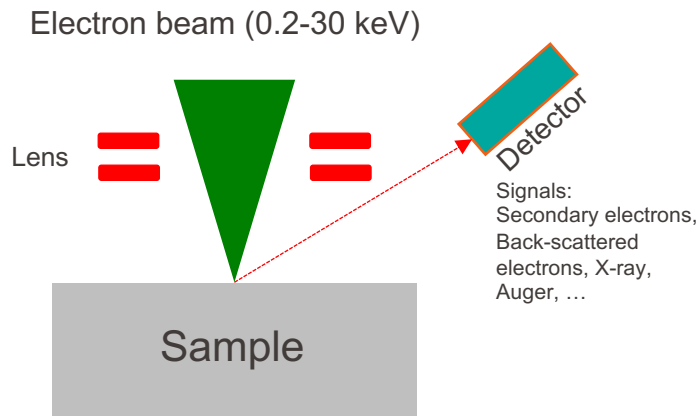
SEM



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



The whole system is kept under high vacuum

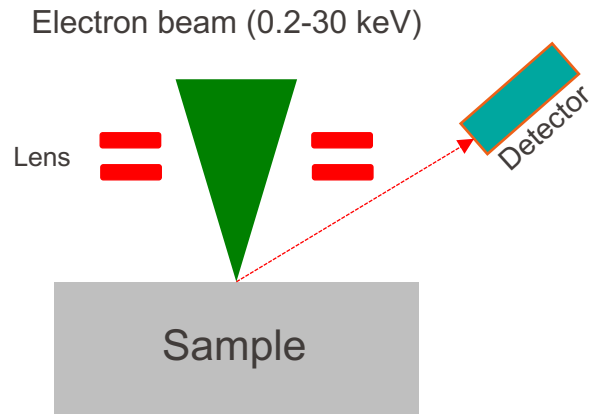
Electrons gun generates electron and accelerates them to high speed (energies).

Electron will go through the 1st lens system to form 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



Why we need a vacuum system?

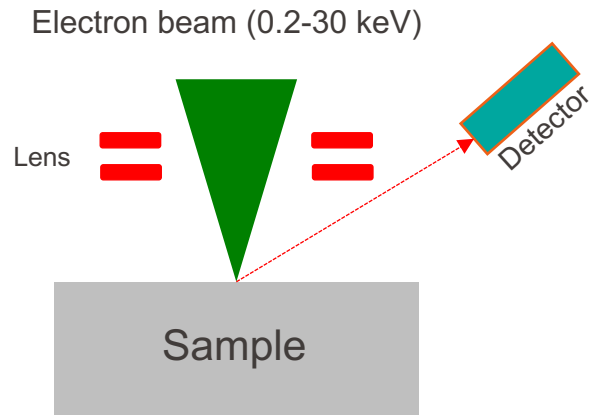
1. Electron propagation is only possible through vacuum (e^- interacts heavily with the matter)
2. A good vacuum system is crucial to reduce contamination and surface modification

Vacuum system

Electron gun
Electron optics
Detectors

Vacuum system has to be **VERY** clean:

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



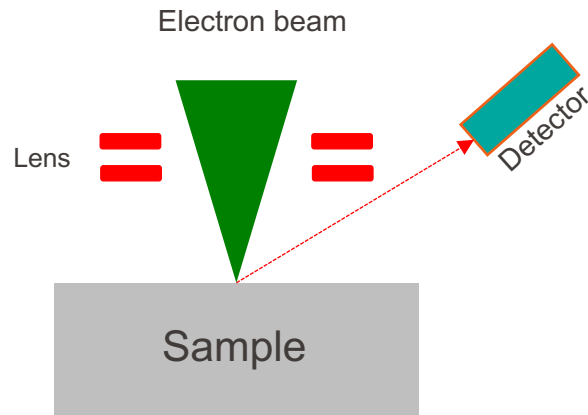
Vacuum system
Electron gun
Electron optics
Detectors

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



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**)

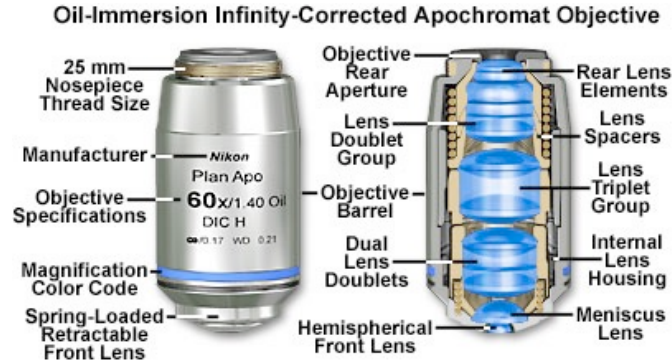
Scan coils raster scan the beam over the sample

Electrons pass through a series of lenses and apertures

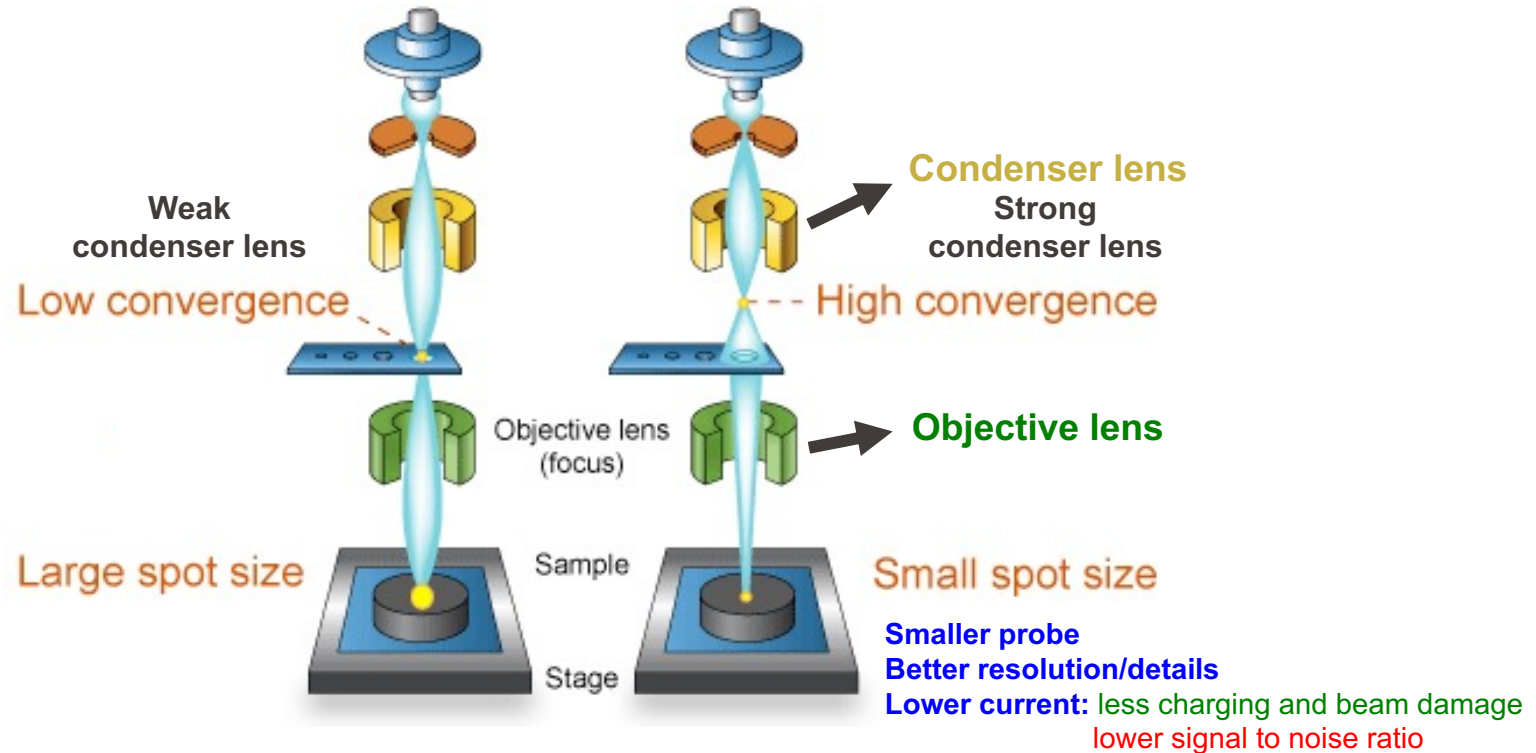
The smaller the probe size, the better the resolution, but the lower the probe current (number of electrons that hit the sample)

Vacuum system
Electron gun
Electron optics
Detectors

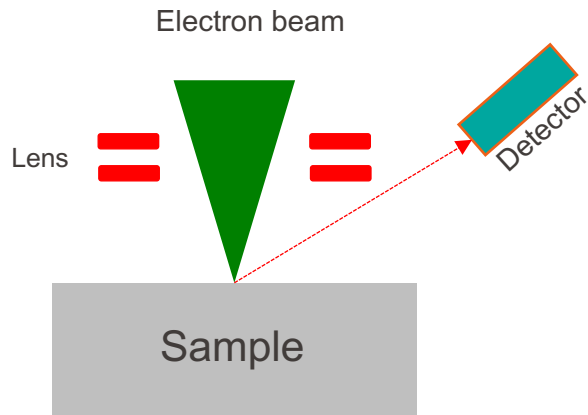
- Lenses for light
 - Glass or polymer lenses
 - Deflection of light through changing refraction index
- Lenses for electrons
 - Variable focus
 - Electrostatic
 - Electromagnetic: Lorentz force



Components of the SEM | Electron optics



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



Purpose:

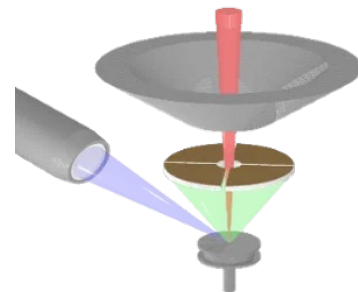
To “see” the electrons, means create signal that is proportional to the number of electrons emanating from the sample at each scan position

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



Emad Oveisi

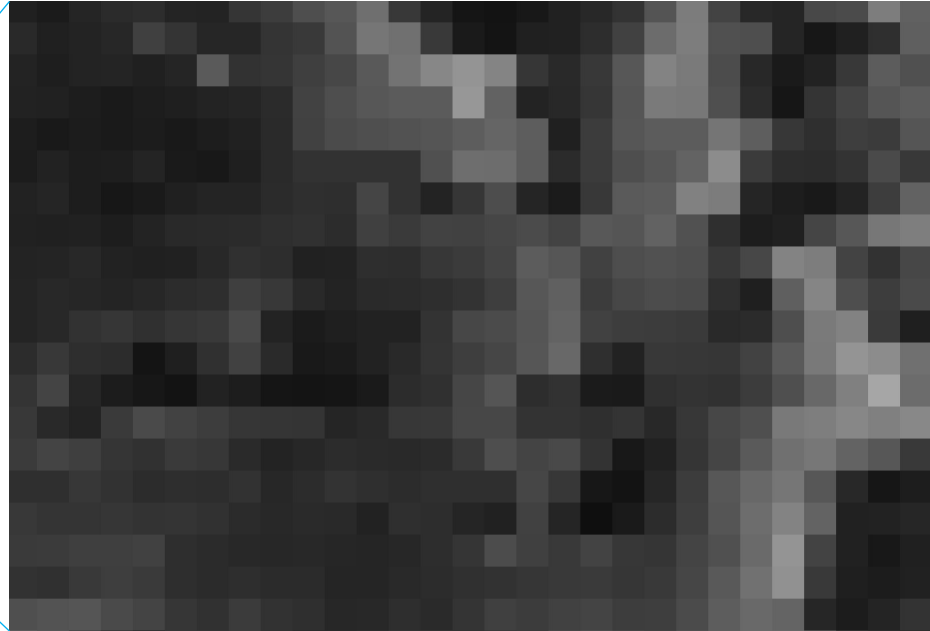
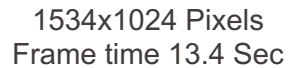
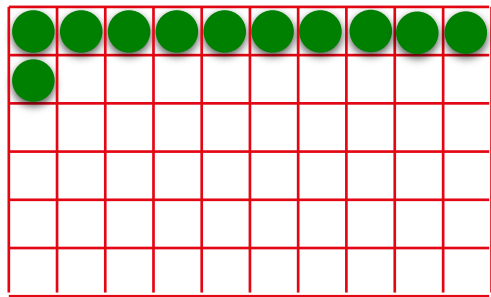
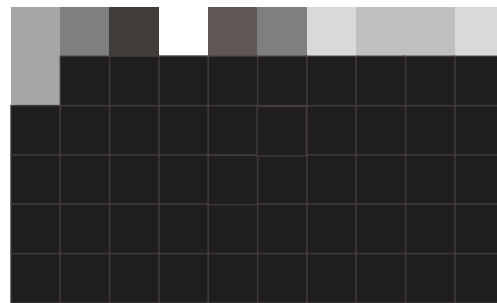


Image is made of pixels of different grey level

How an SEM image is generated?



Beam locations on the specimen

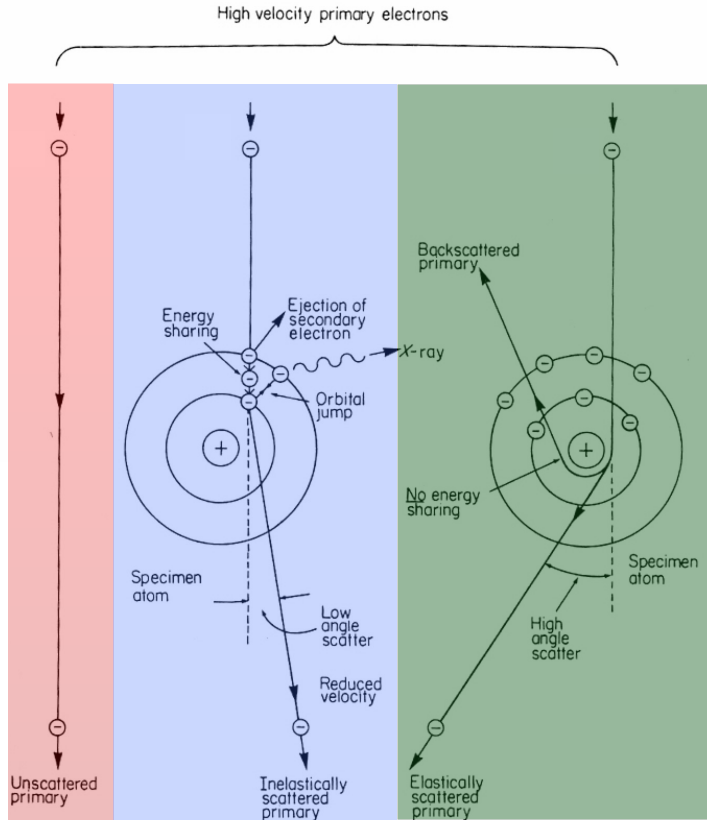


Area scanned on the screen

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



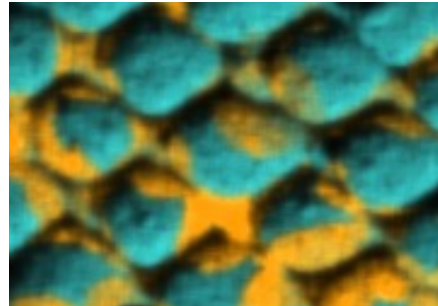
Inelastic events: The result is a **transfer of beam energy** to the specimen atom (**energy loss**) and a potential expulsion of an electron from that atom as a **secondary electron (SE)**.

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.



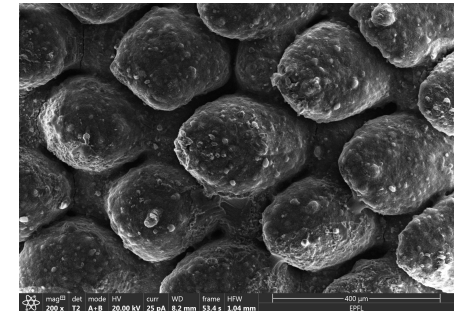
That's why TEMs are shielded!

Characteristic X-rays elemental map

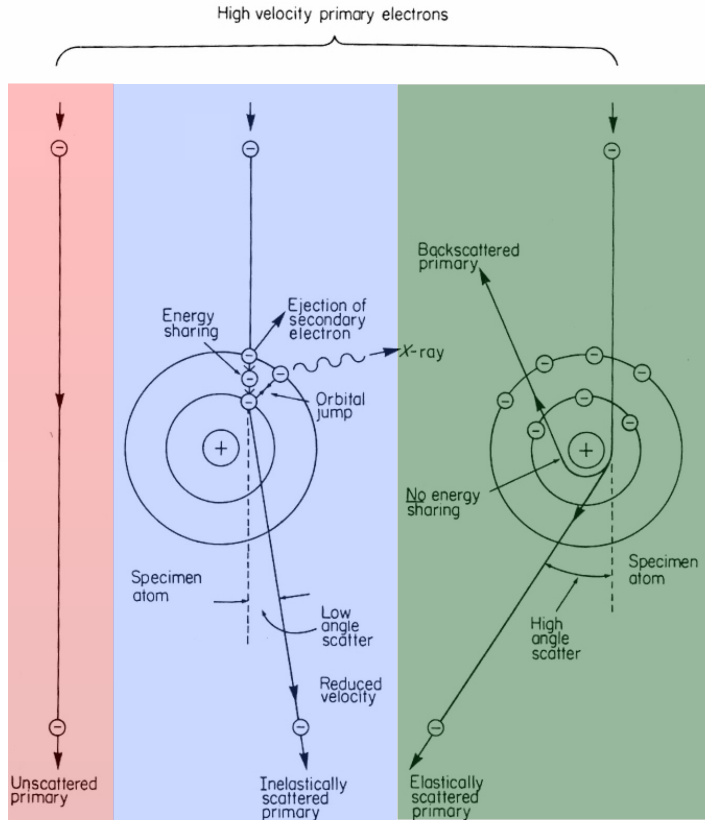


Chemical composition

Secondary electron SEM image



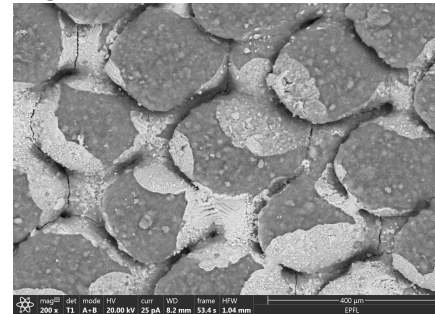
Topography



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).

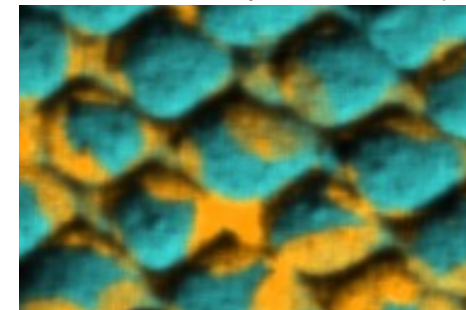
Coulombic interaction within the electron cloud, Low-angle scattering
Coulombic attraction by the nucleus, Higher-angle scattering

Larger nucleus $\rightarrow$ More backscattering



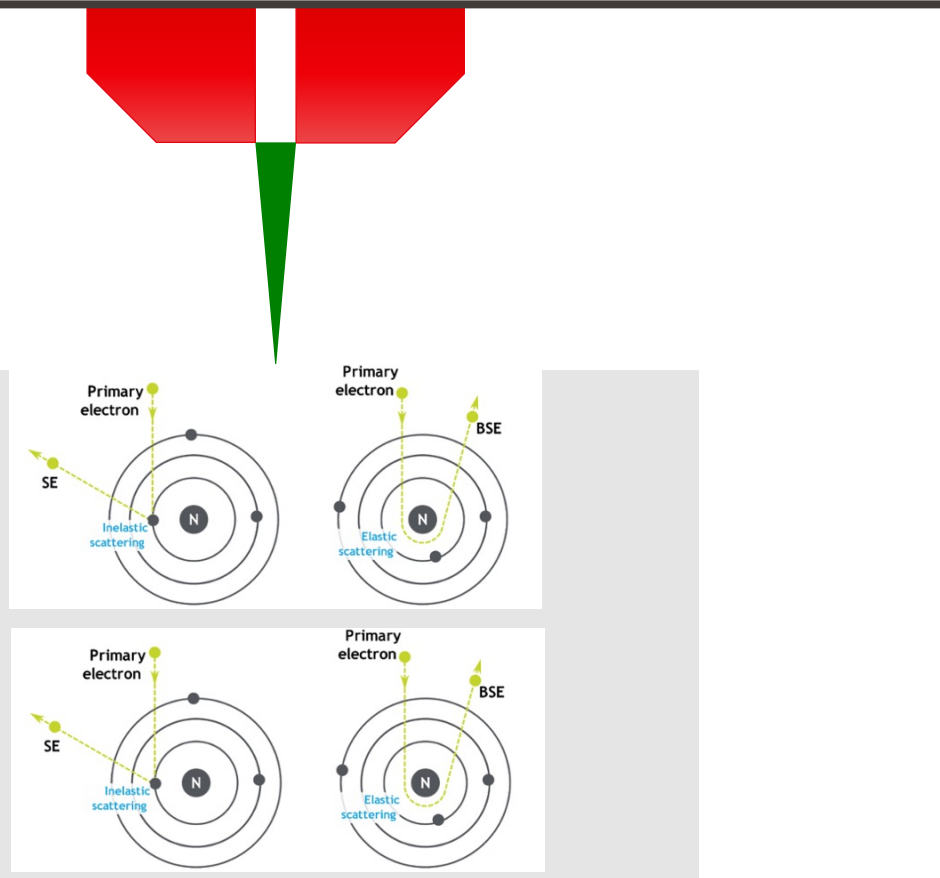
Mass contrast
Backscattered SEM image

Characteristic X-rays elemental map

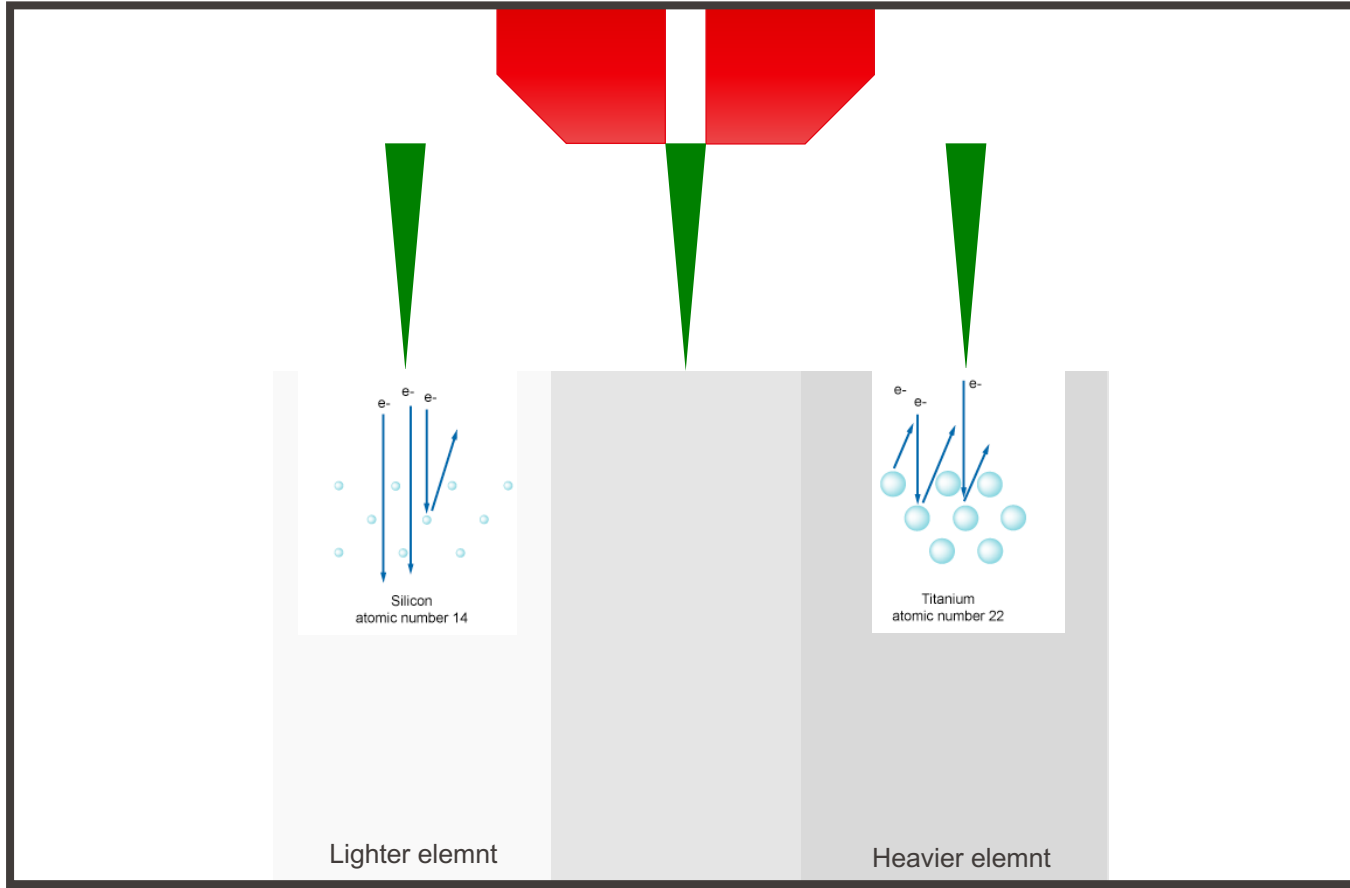


Chemical composition

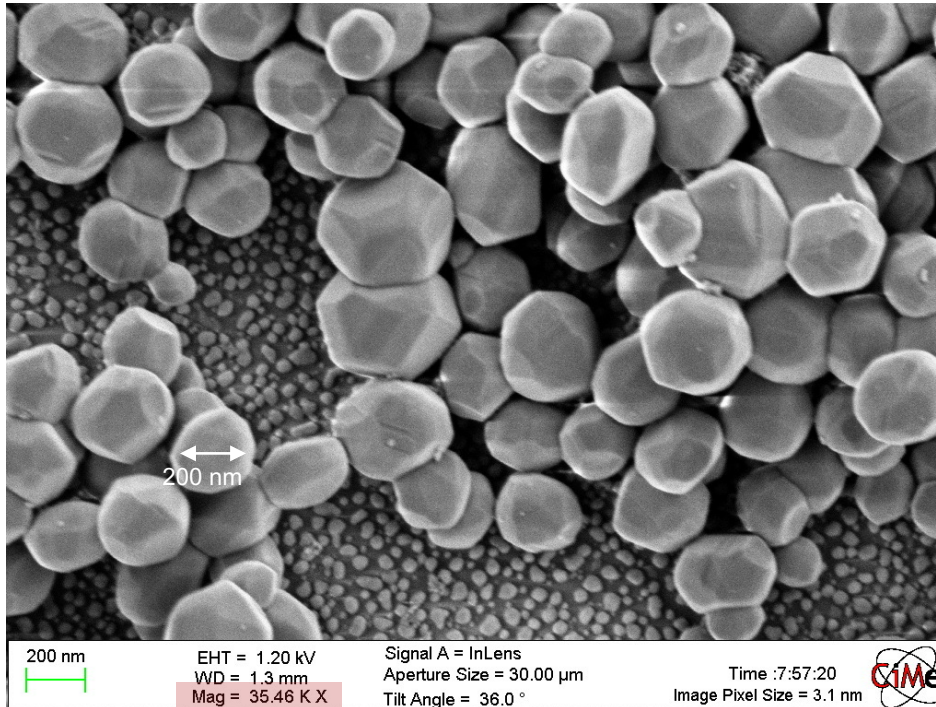
How an SEM image is generated?



How an SEM image is generated?



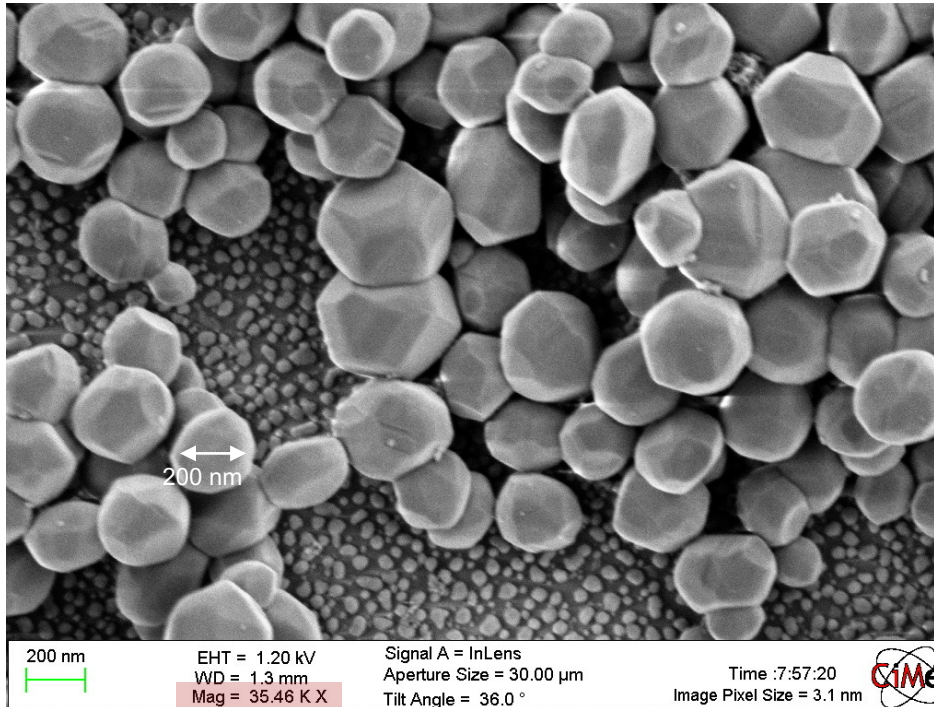
Measure the size of the particle and calculate the magnification



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

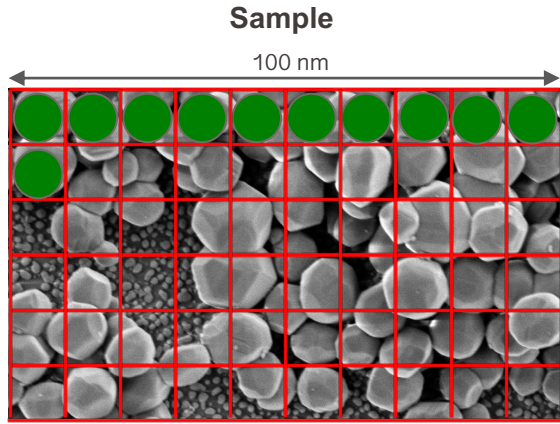
Measure the size of the particle and calculate the magnification



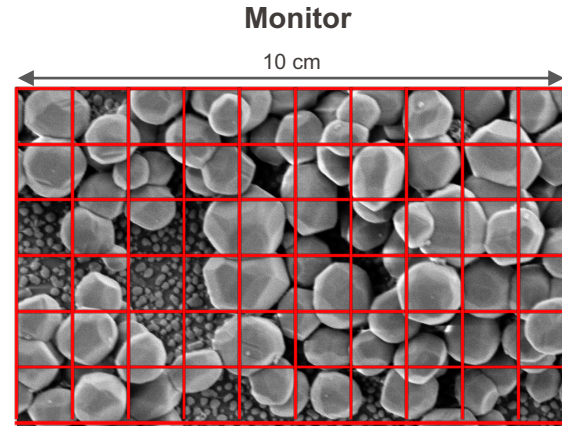
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- a) 35.46 kX
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- c) 100 kX
- d) 200 nm

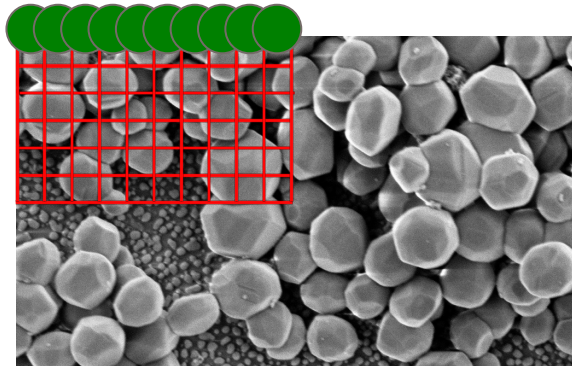
How an SEM image is generated?



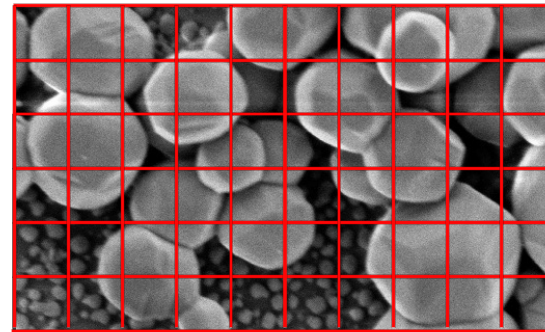
Scan step (i.e. pixel size) on the sample?



Pixel size on the screen?

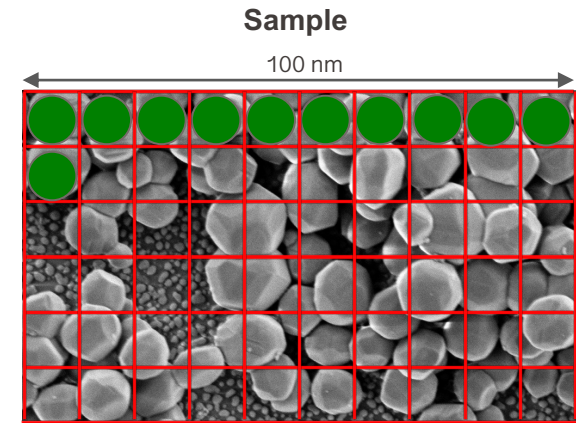


Scan step (i.e. pixel size) on the sample?

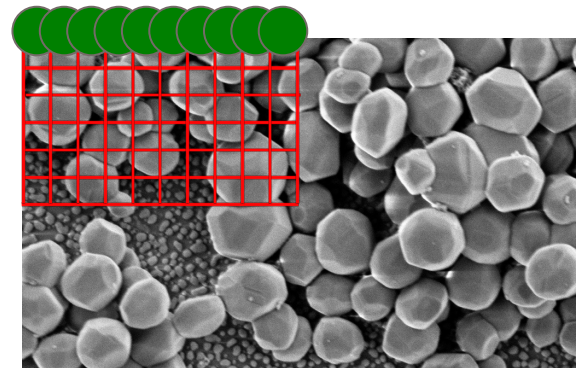


Pixel size on the screen?

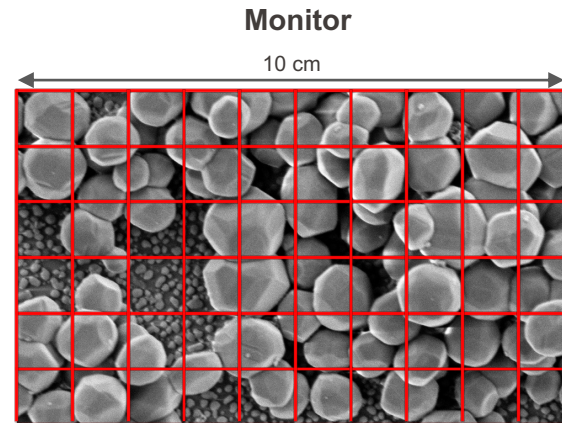
How an SEM image is generated?



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

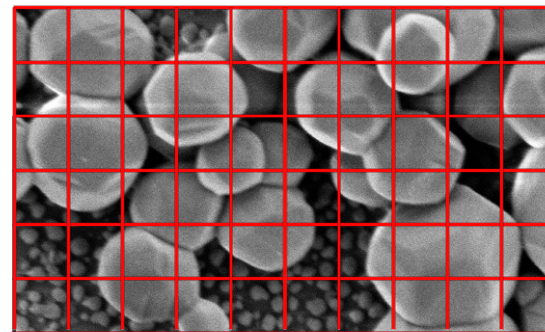


Scan step (i.e. pixel size) on the sample = 5 nm



Pixel size on the screen? = 1 cm

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



Pixel size on the screen? = 1 cm

$$\text{Magnification} = 10^{-2} / 5 \cdot 10^{-9} \\ = 2 \text{ MX}$$

What happens to resolution?

Parameters affecting Resolution (and Visibility)

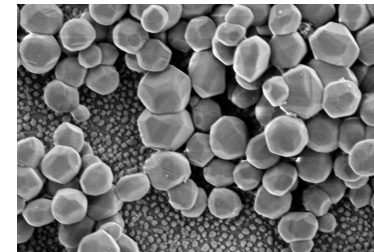
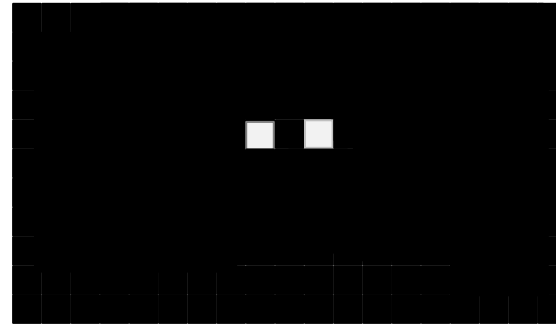
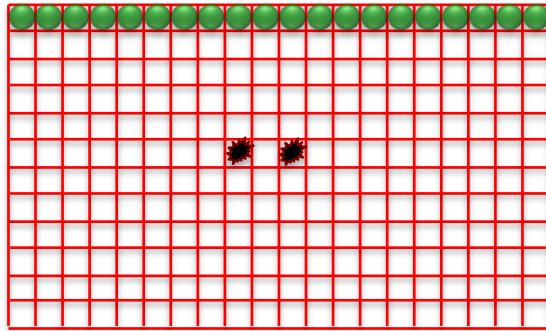
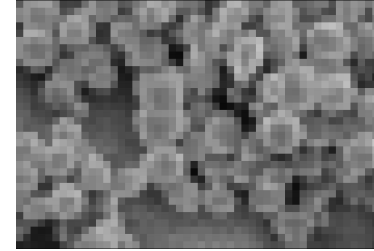
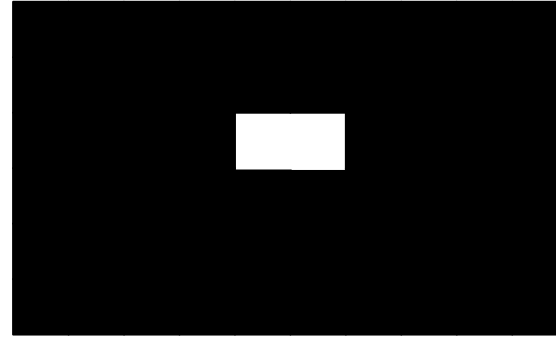
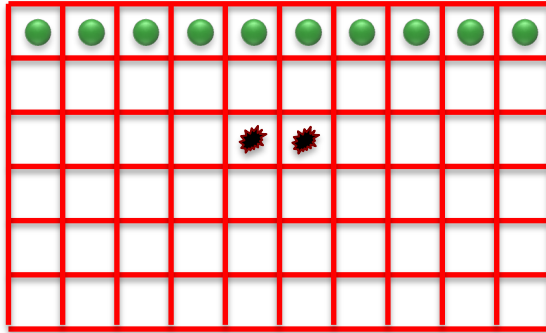
- **Fundamental**

- 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

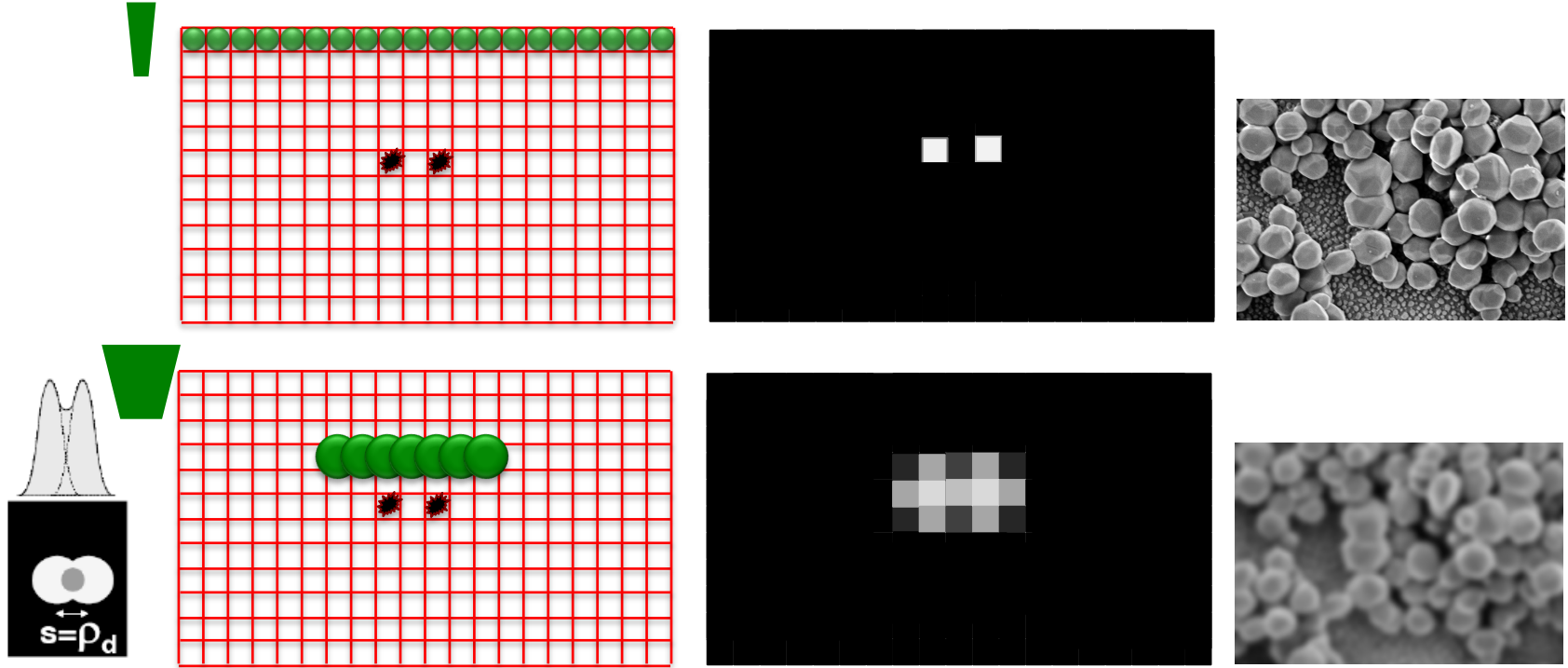
- **Operational and sample**

- 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)

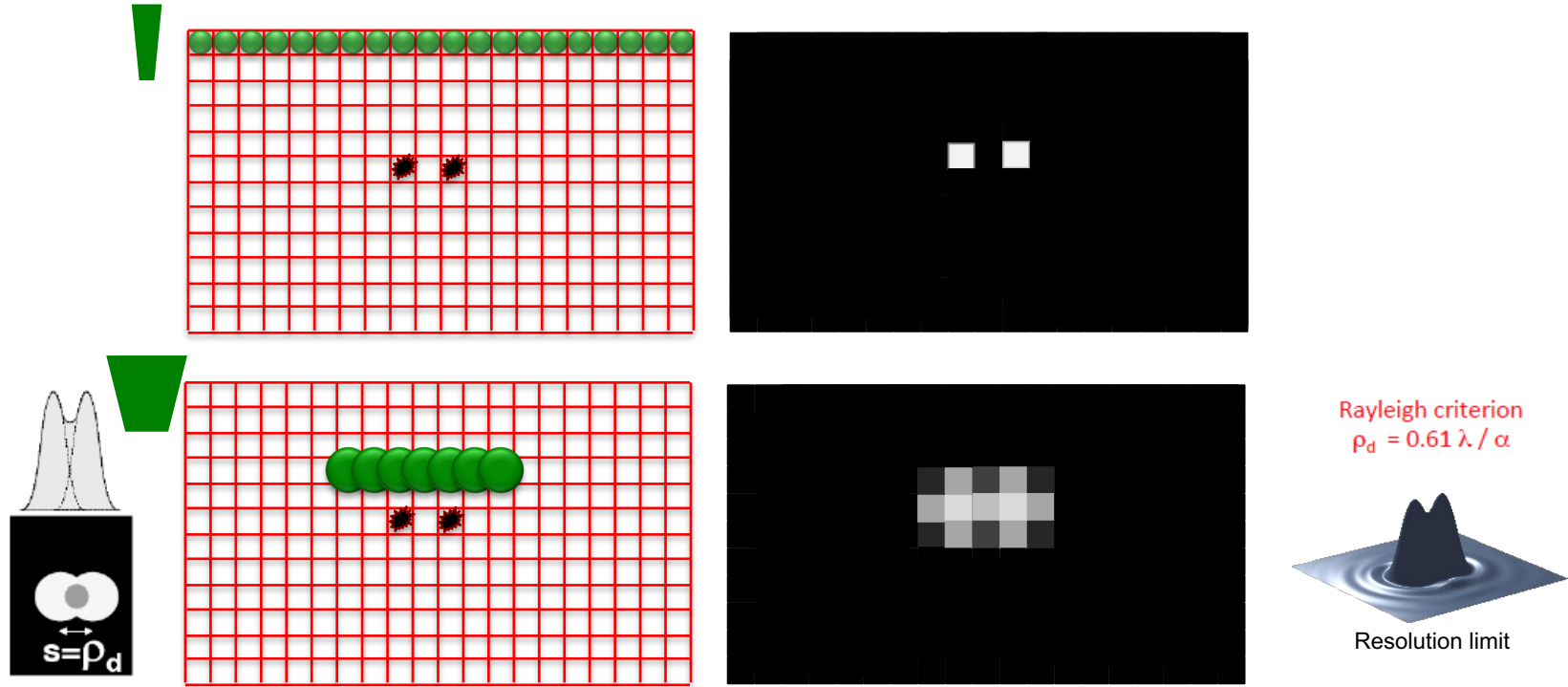


Nyquist sampling (f) = $d/2$, where “d” is the smallest object, or highest frequency

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).



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



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)

Parameters affecting Resolution (and Visibility)

- **Fundamental**

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

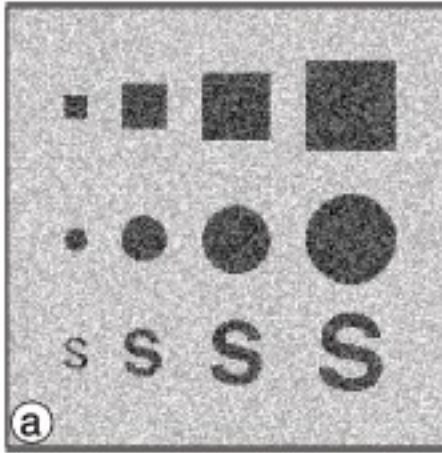
Probe size (or spot size) means the diameter of the final beam at the surface of the specimen

- **Operational and sample**

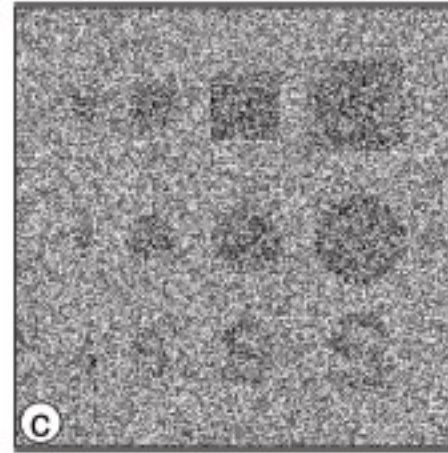
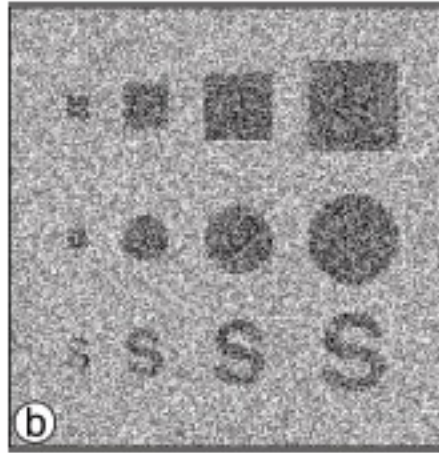
- Pixel size = scan step size
- Probe size (also defines probe current and affects visibility)
- Visibility:
 - Scan speed (i.e. dwell time) and “signal to noise ratio”
 - Contrast
- Type and depth of emitted electrons signal
- System/Specimen stability → Challenges (charging, contamination, beam damage)

Visibility and “signal to noise” ratio

Features on a noisy background:



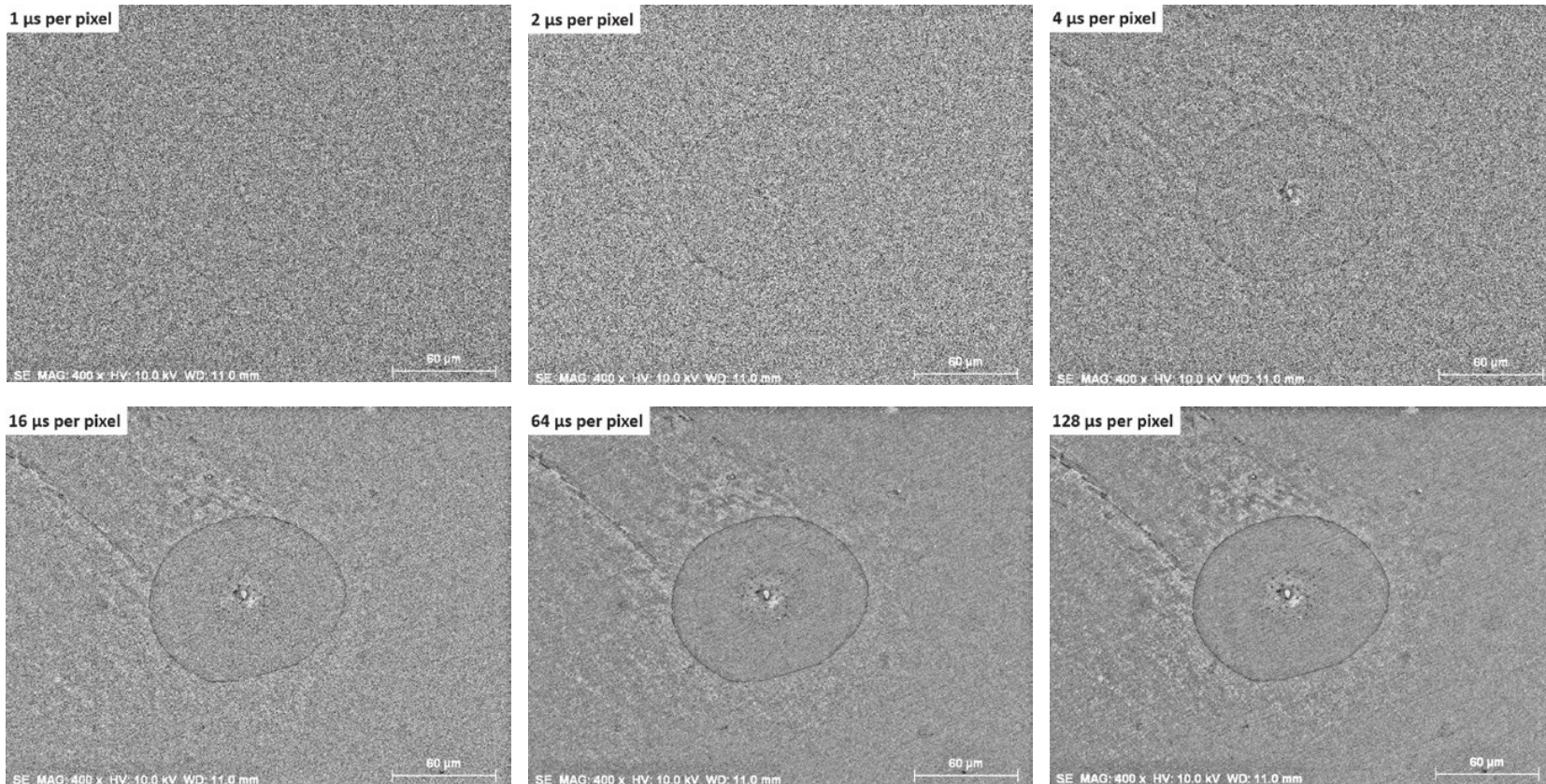
High signal to noise ratio



Low signal to noise ratio

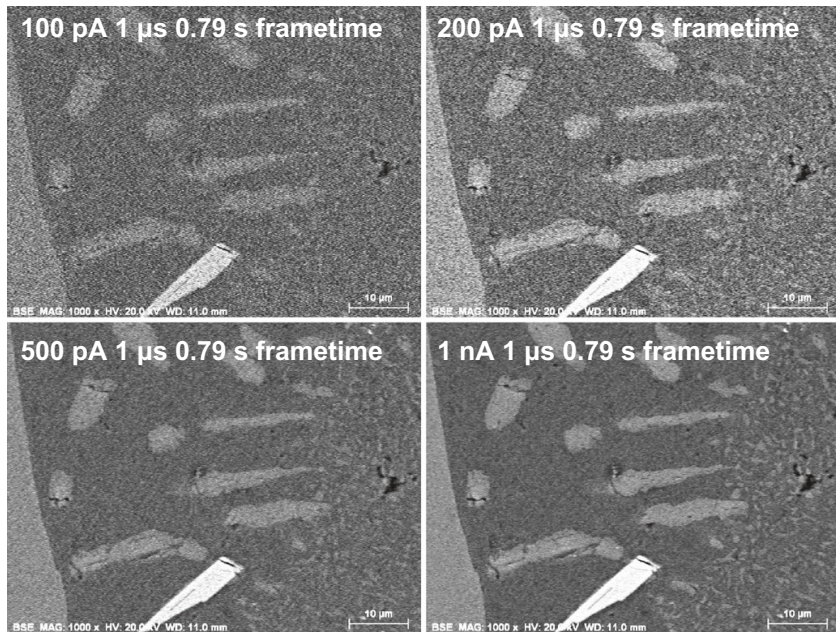
How to improve signal to noise ratio?

Visibility and “signal to noise” ratio



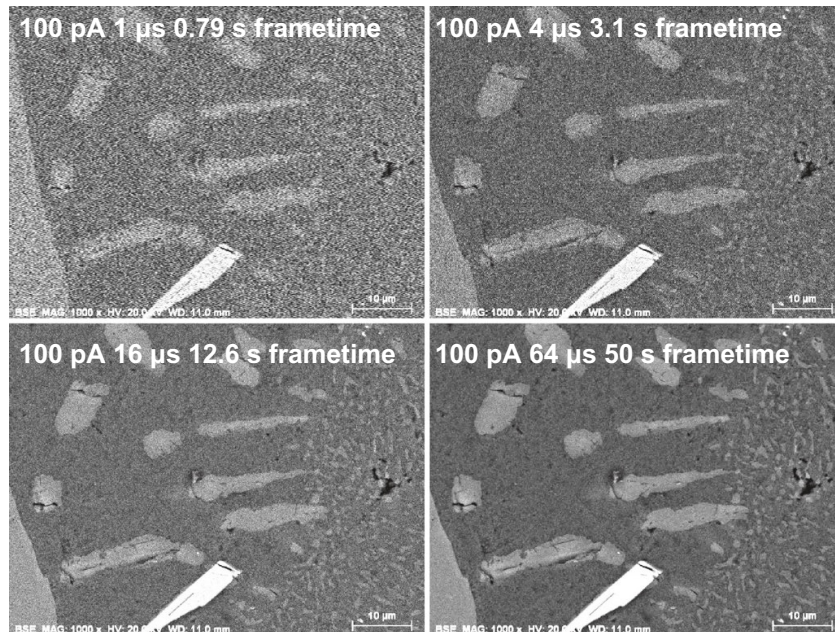
Threshold imaging visibility; image sequence with increasing pixel **dwell time** at constant beam current.
Sample: Inkjet deposited droplet on carbon planchet; ($E_0 = 10$ keV; Everhart–Thornley)

Effect of beam current



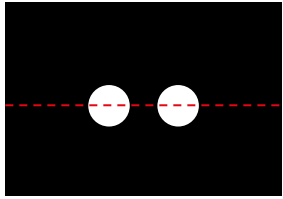
Possible charging or sample damage

Effect of pixel dwell time

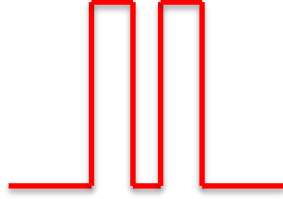


Sample drift may become the limiting factor

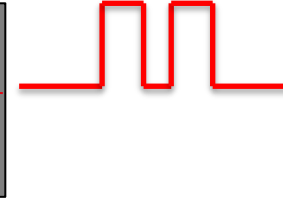
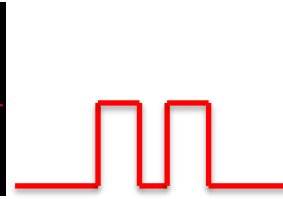
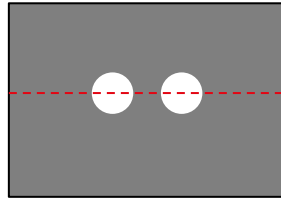
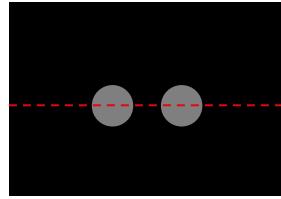
100% Intensity change



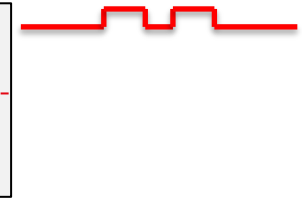
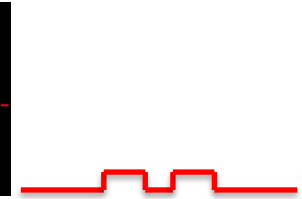
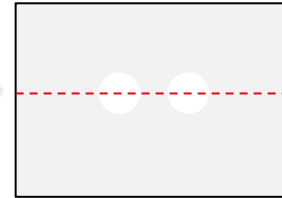
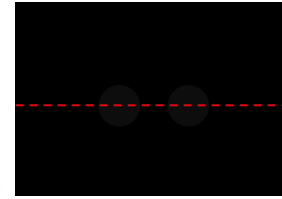
Particles with
positive contrast



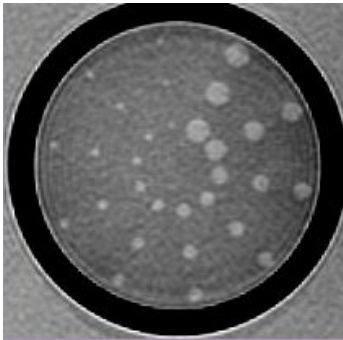
50% Intensity change



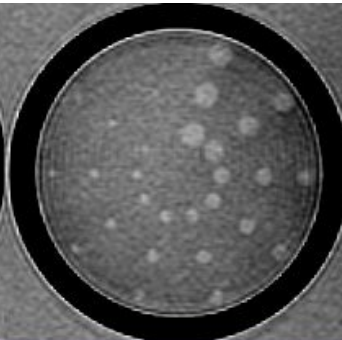
5% Intensity change



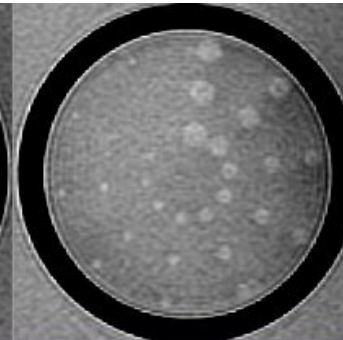
5.1% contrast



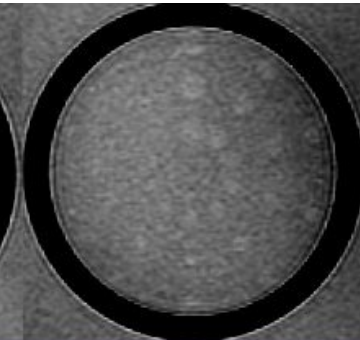
3.7% contrast

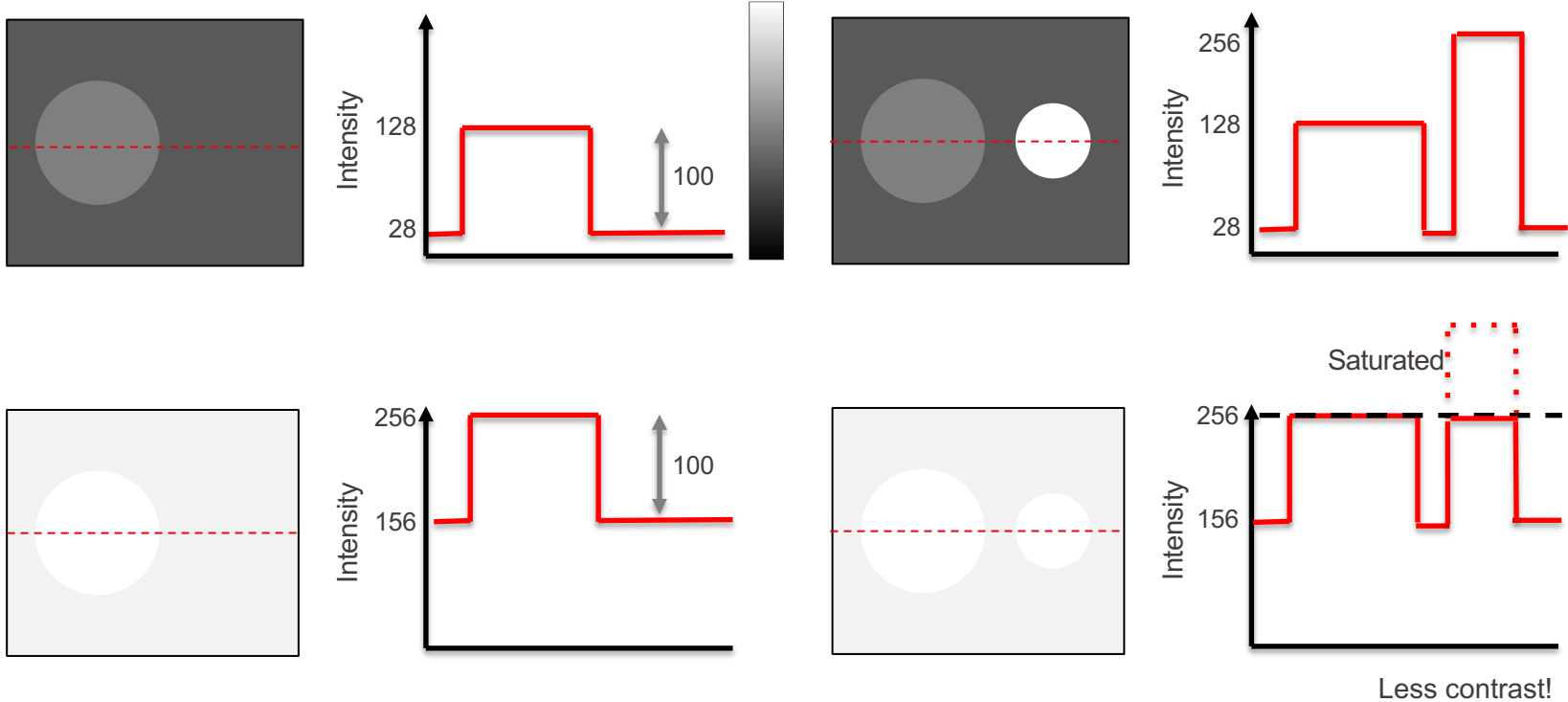


2.2% contrast



1% 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

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)

- 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

Incident electrons interaction with the sample produces:

Electron signals:

Secondary electrons SE

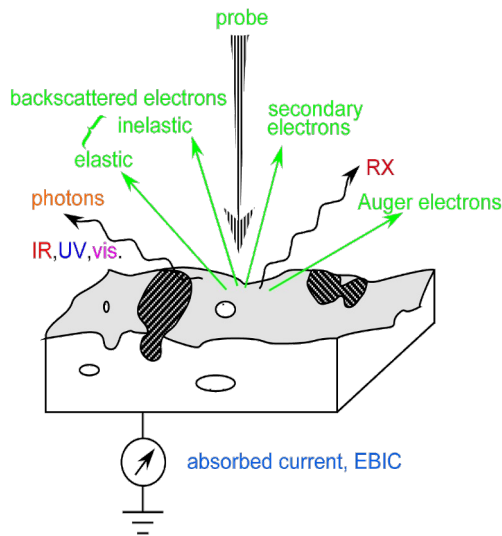
Electrons ejected from the conduction or valence bands of the target atom: low energy $\approx 5\text{-}50\text{ eV}$
 Free electrons: not associated with a specific atom and contain no specific elemental information
 Can only escape if they are near the specimen surface → **Topography**

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



Incident electrons interaction with the sample produces:

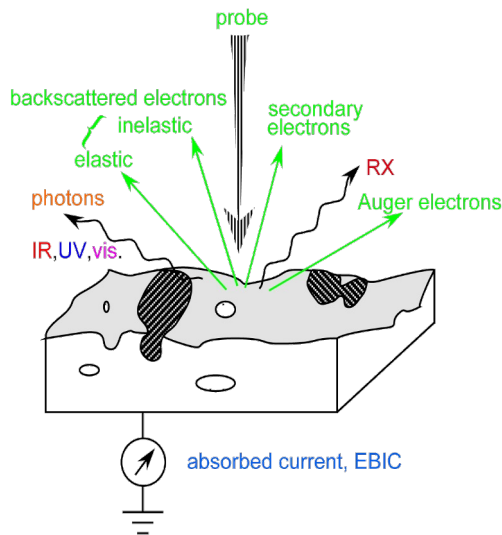
Other signals:

Electromagnetic radiations:

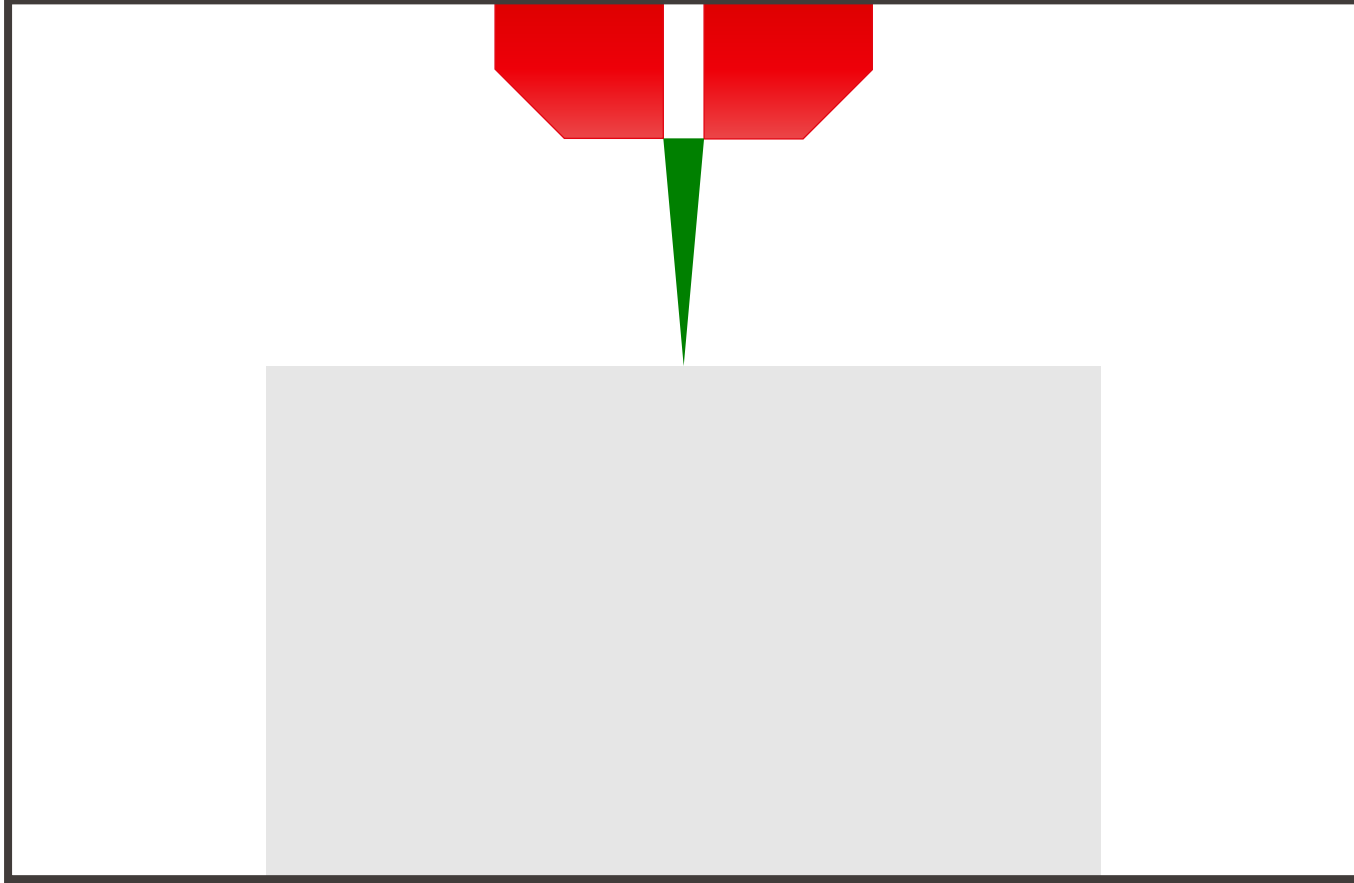
- X-rays with continuous energy, resulting from deceleration of incident electrons ("Bremsstrahlung" or "braking radiation")
- Characteristic X-rays with a distinct energy associated to the target atoms.
- Cathodoluminescence: Visible radiation mainly emitted by insulating or semi-conducting materials.

Others:

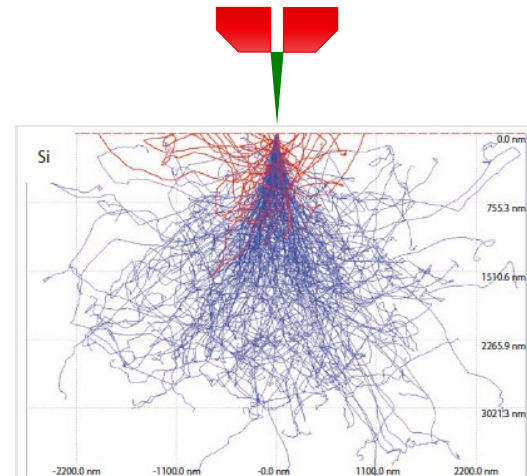
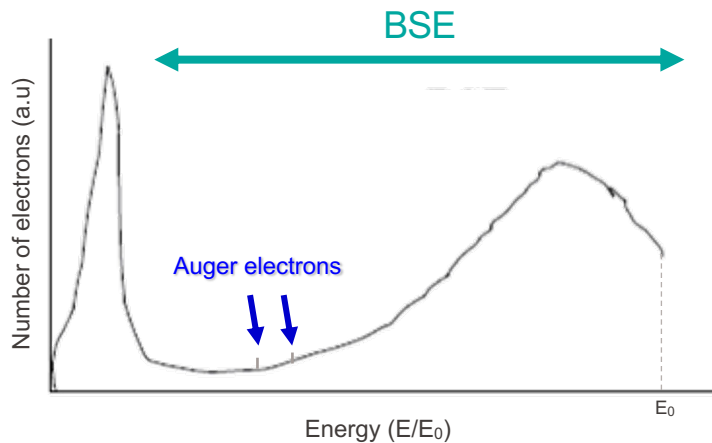
- Absorbed current, electron-holes pairs creation, EBIC
- Plasmon
- Sample heating (phonons)
- Radiation damages: chemical bonding break, atomic displacement (*knock-on*) damage



SEM signals | Where they come from



Energy spectrum of electrons leaving the sample

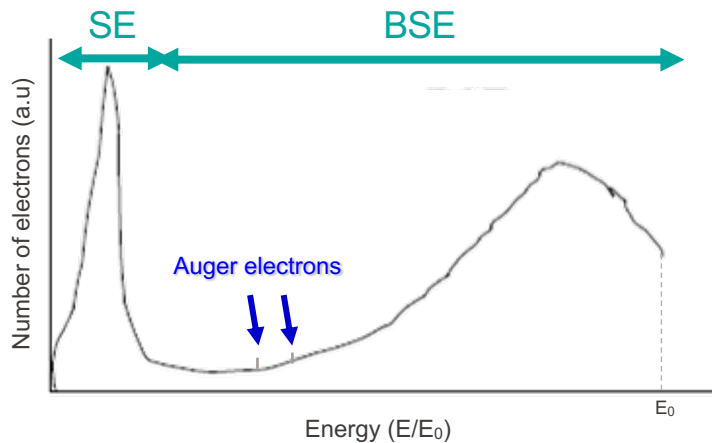


$$\text{BSE: } 50 \text{ eV} < E_{\text{BSE}} < E_0$$

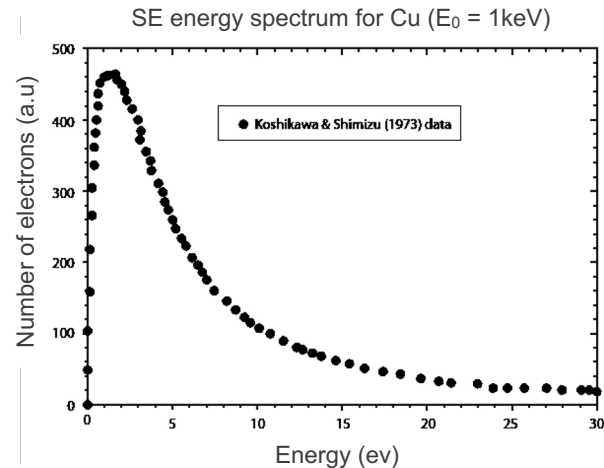
What is the source of the prominent peak in the low-energy region of the spectrum?

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.

Energy spectrum of electrons leaving the sample



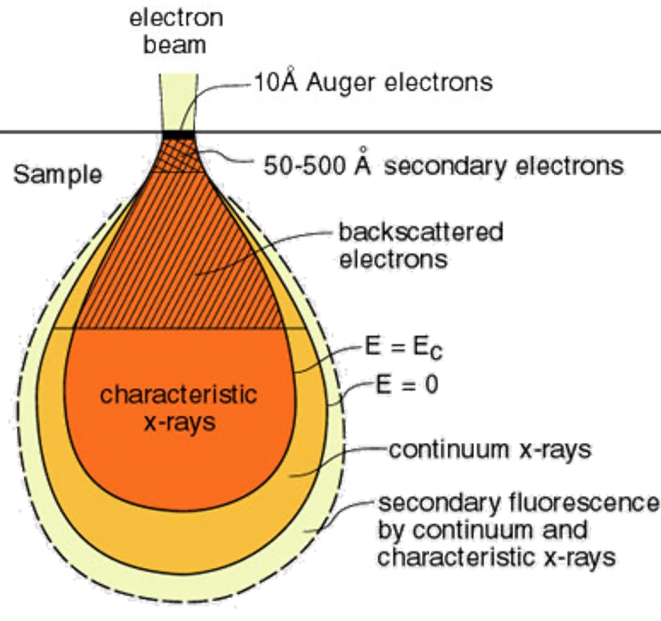
$$\text{BSE: } 50 \text{ eV} < E_{\text{BSE}} < E_0$$



$$\text{SE: } E_{\text{se}} < 50 \text{ eV}$$

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



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 fluorescence (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)

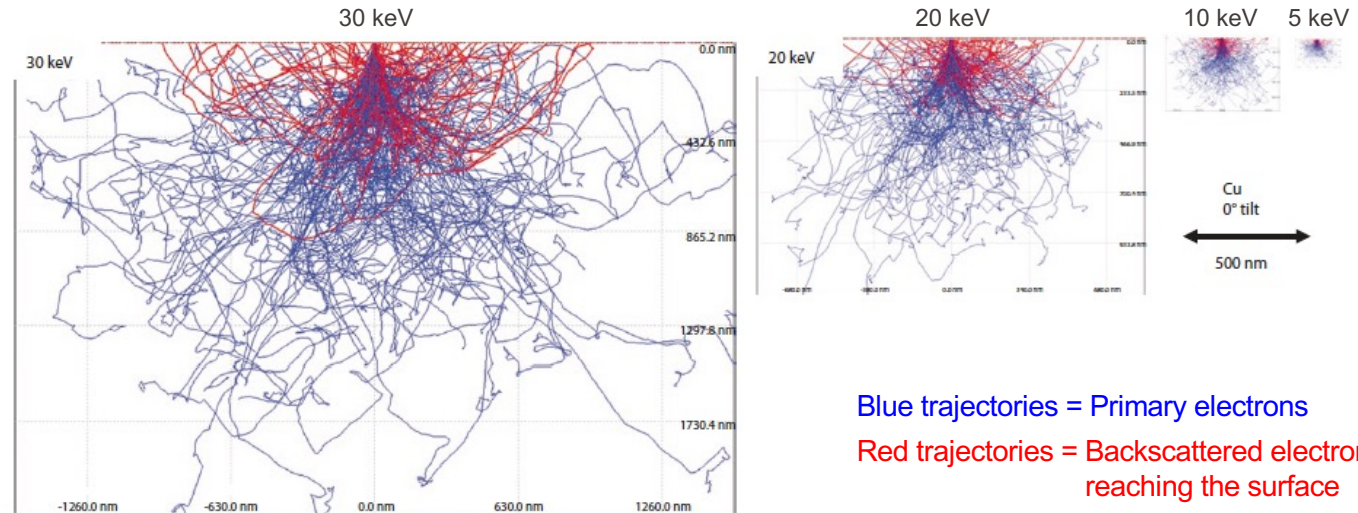
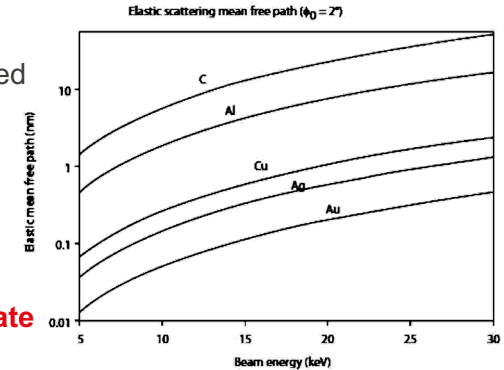
Monte-Carlo simulations (Details in MSE636)

The higher the beam energy,

- The longer the scattering MFP, so the less the rate of energy loss with distance travelled
Electrons enter 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.

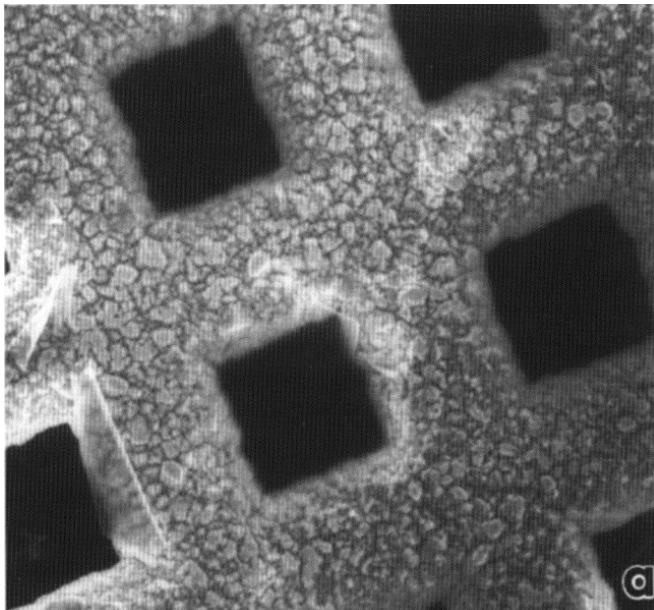


Blue trajectories = Primary electrons

Red trajectories = Backscattered electrons reaching the surface

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



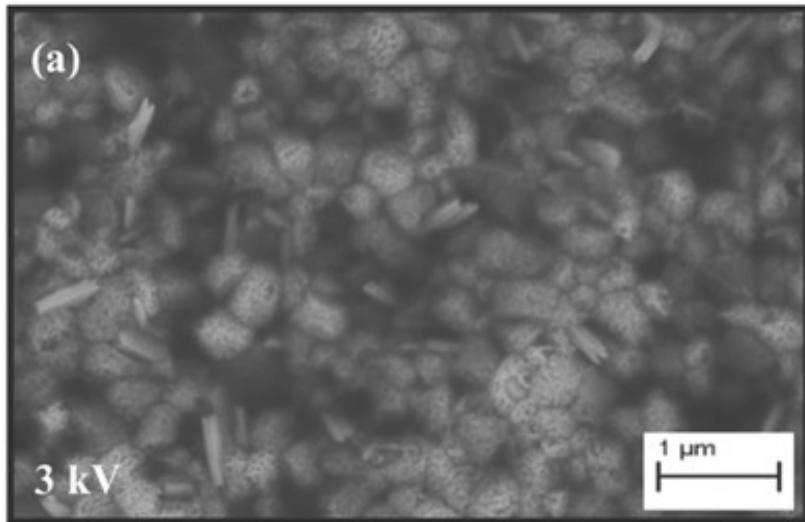
2 kV:

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

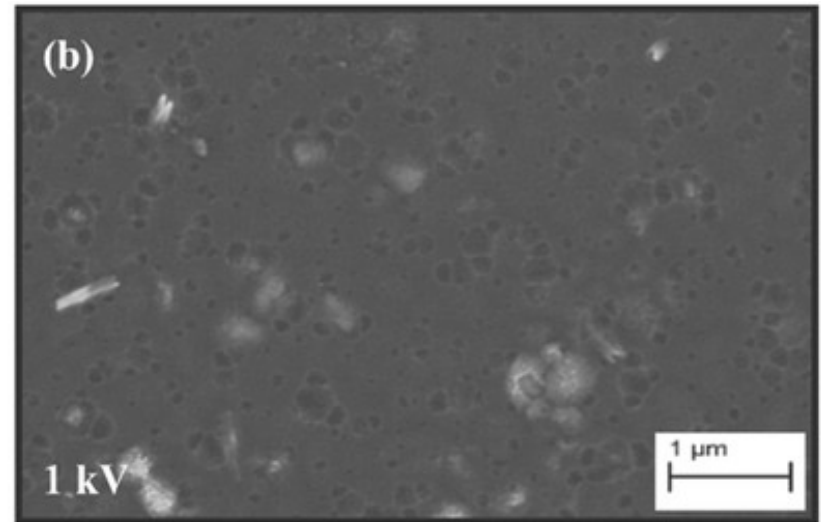
Effect of the accelerating voltage (beam energy) on penetration depth and signal

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.



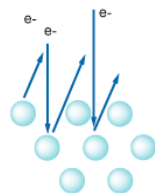
1 kV:

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

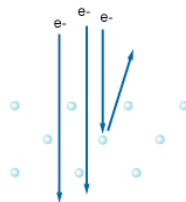
The higher the atomic number “Z”,

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

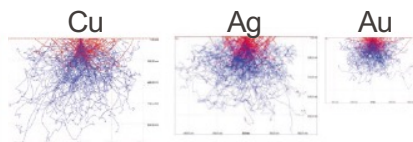
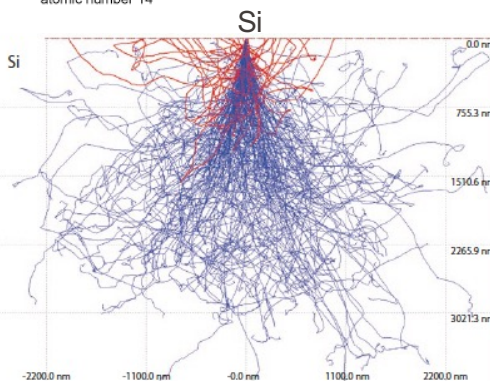
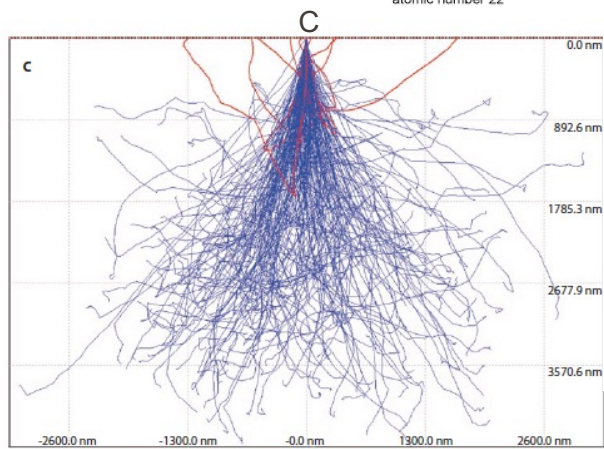
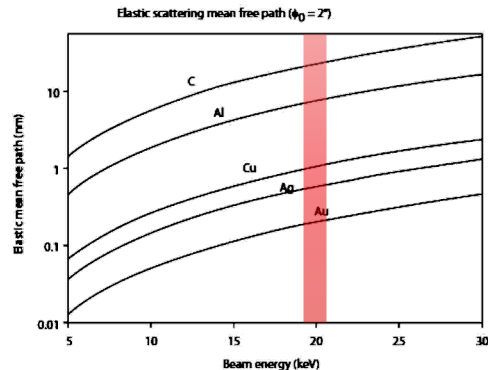
So, the shorter the penetration depth (i.e. smaller interaction volume).



Titanium
atomic number 22



Silicon
atomic number 14



$E_0 = 20 \text{ keV}$
 0° tilt

1 μm

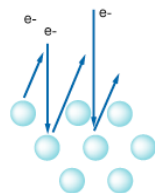
Blue trajectories = Primary electrons

Red trajectories = Backscattered electrons reaching the surface

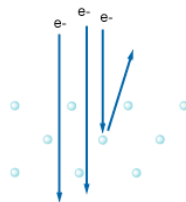
The higher the atomic number “Z”,

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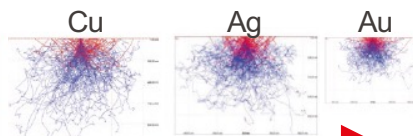
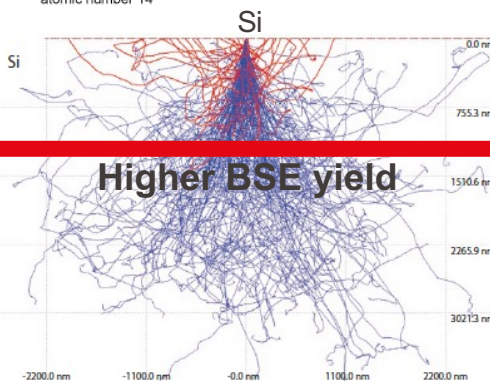
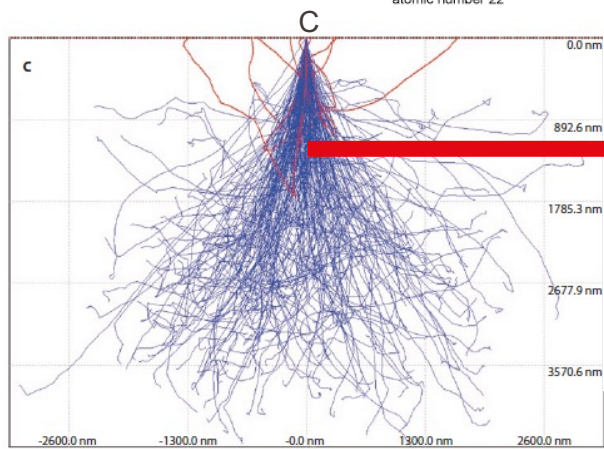
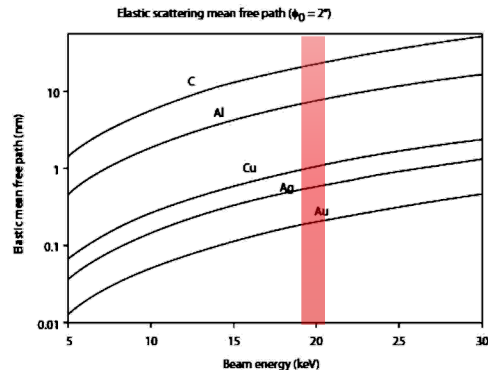
So, the shorter the penetration depth (i.e. smaller interaction volume).



Titanium
atomic number 22



Silicon
atomic number 14



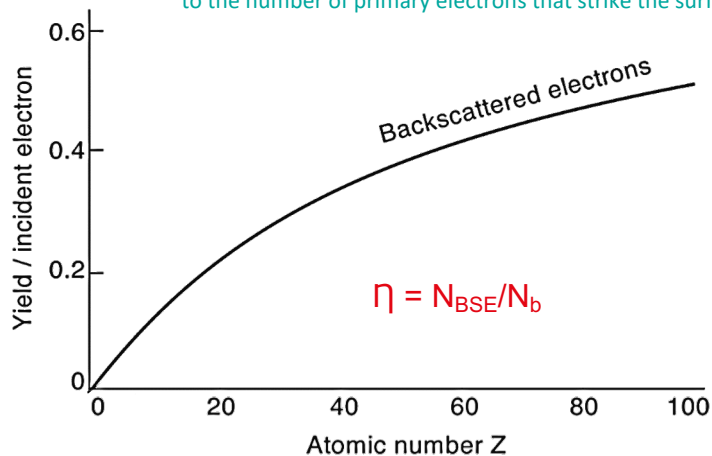
Higher BSE yield

$E_0 = 20 \text{ keV}$
 0° tilt
1 μm

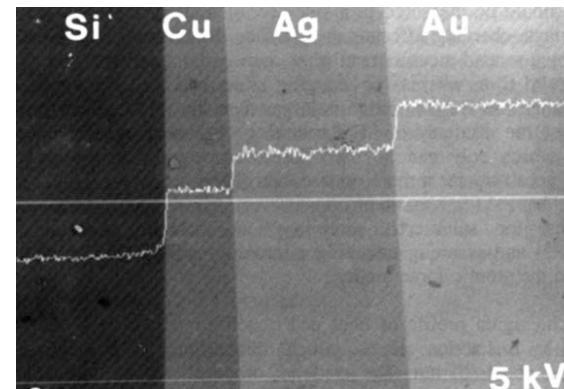
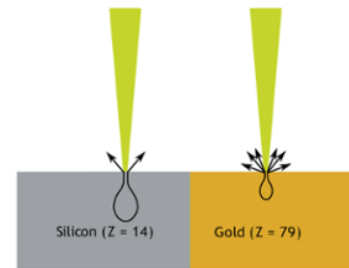
Blue trajectories = Primary electrons

Red trajectories = Backscattered electrons reaching the surface

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



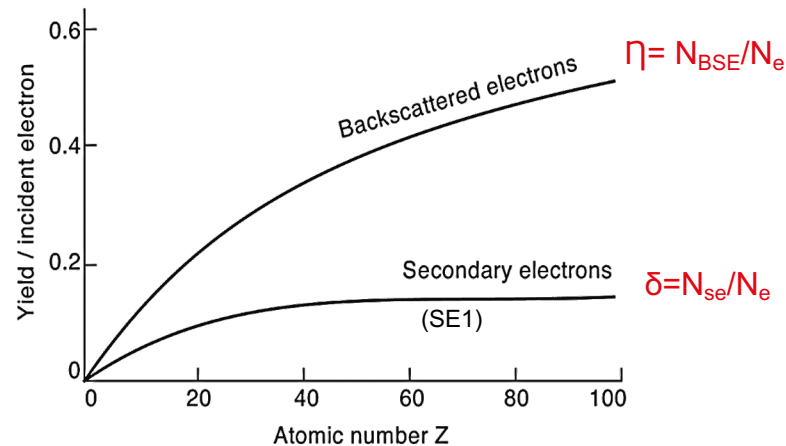
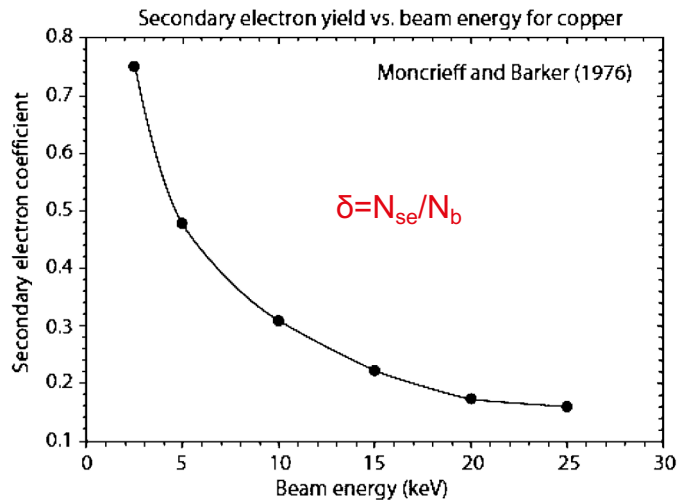
BSE yield η increases with “Z”



- 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).



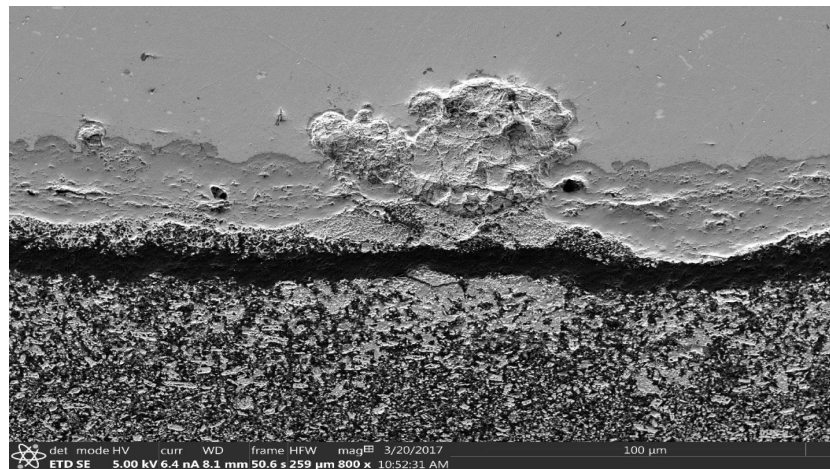
There is a general rise in δ as the beam energy is decreased, primarily due to the reduction in the interaction volume.

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

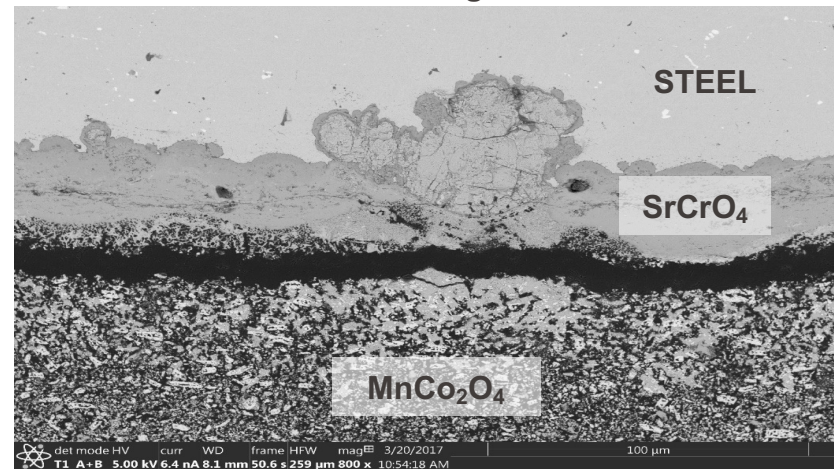
BSE yield increases monotonically with the “Z” of material, and SE yield depends mainly on the beam energy

Example1 : Ferritic stainless substrate coated with a porous MnCo_2O_4

SE image



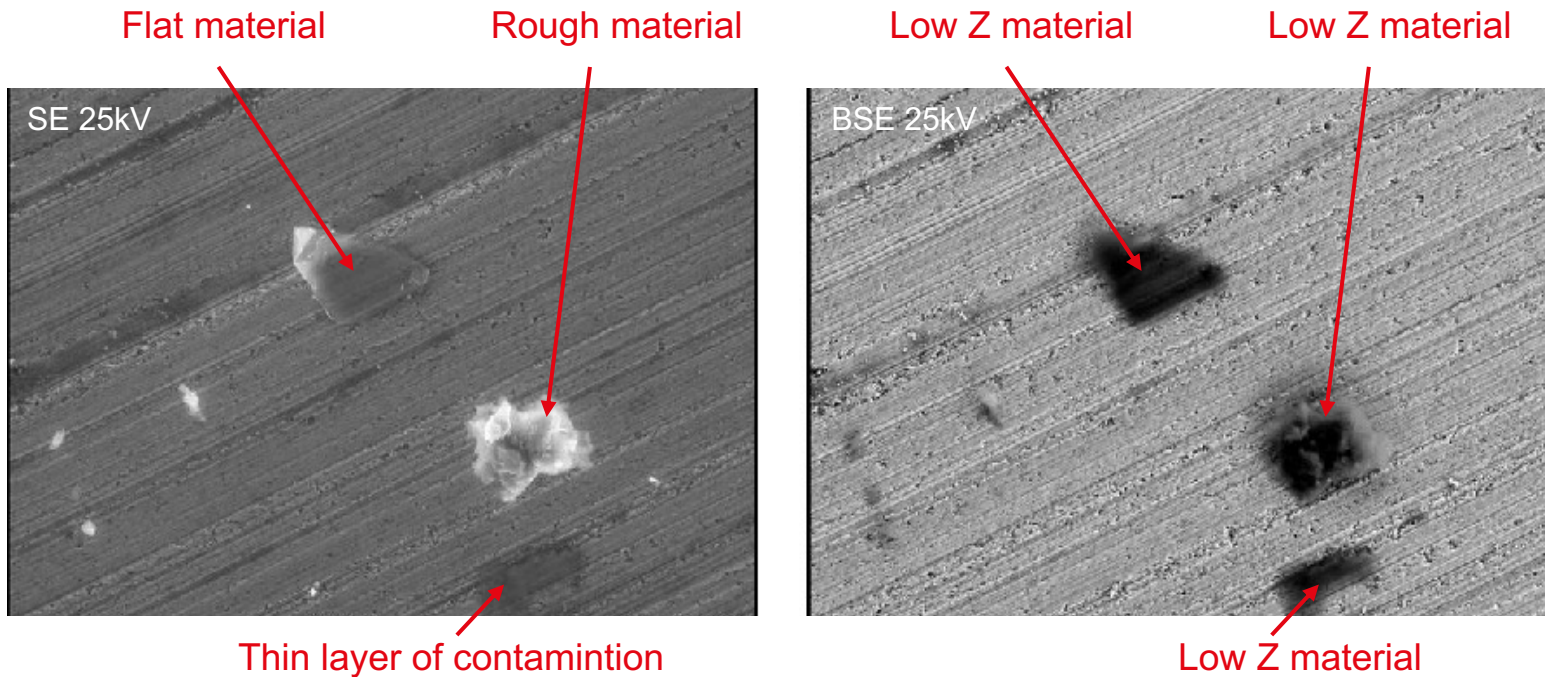
BSE image



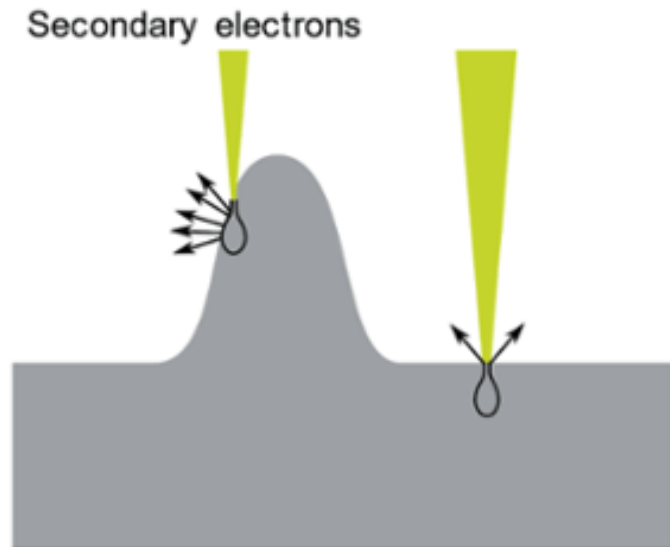
SE have low energies (5-50eV), and thus are **emitted** only from surface and possess information about topographical features.

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

Example 2: Dust on WC (different Z materials)



Why the rough particle appears brighter in the SE image?



- If a sample is tilted, 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 tilted and have higher SE yield.

Rough surfaces generates higher SE image signal.

Titling the specimen can change SE image contrast.

Parameters affecting Resolution (and Visibility)

- **Fundamental**

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

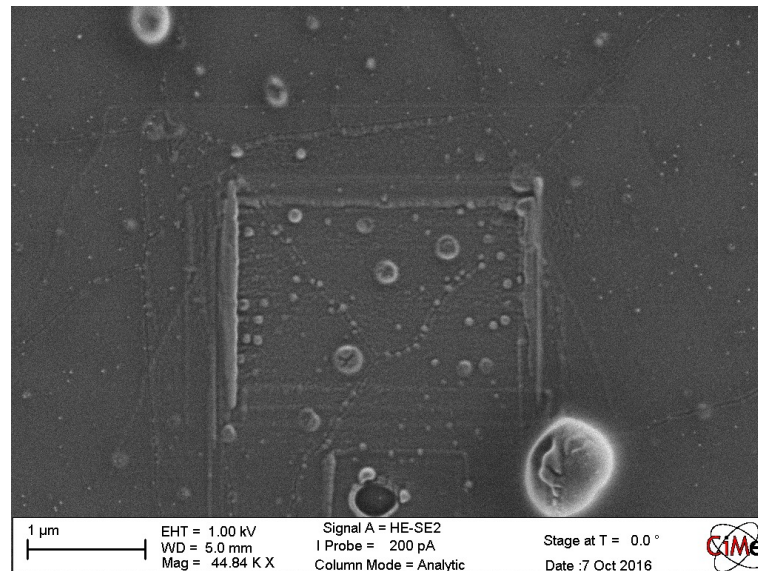
Probe size (or spot size) means the diameter of the final beam at the surface of the specimen

- **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)

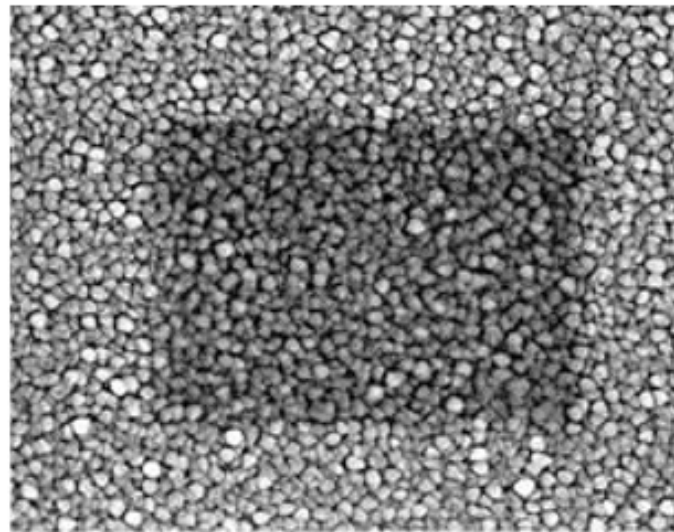
Beam induced changes to the sample:

- 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



Hydrocarbon build-up on surface

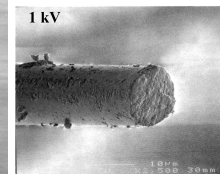
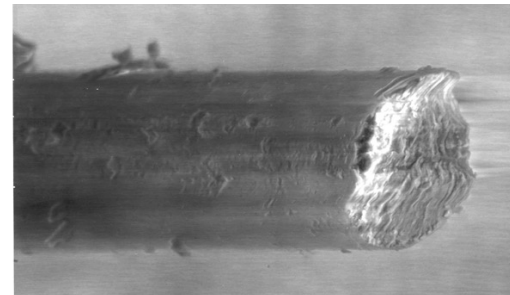
- Masks surface features and information about the sample
- Sources:
 - Sample surface
 - SEM chamber
 - Beam induced degradation and migration of sample compounds
- To avoid / minimize:
 - Use gloves when handling samples
 - Plasma cleaning sample prior to observation



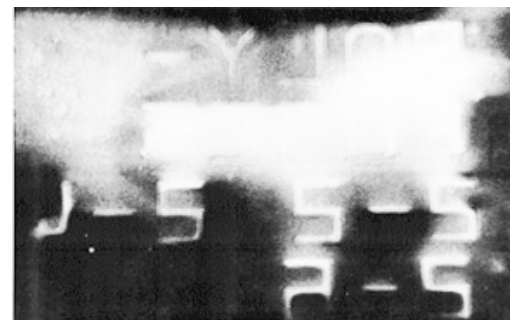
Accumulation of negative charge

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

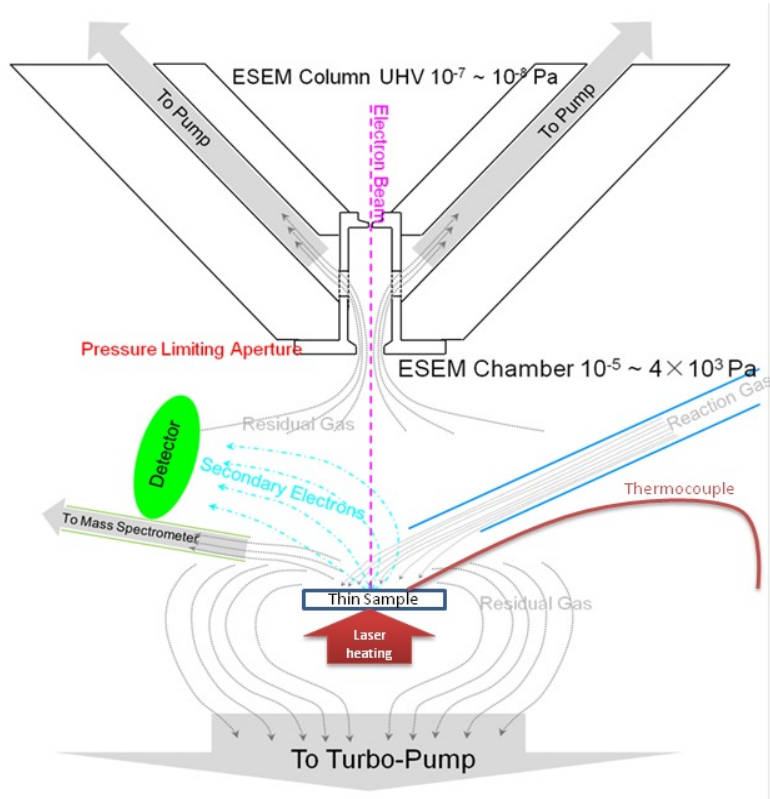
- Charging deflects the low-energy secondary electrons (mainly, but can affect BSE) causing image distortions and contrast changes
- Depends on:
 - Material properties (surface resistivity)
 - Beam energy and current
 - Scan rate
- Ways to mitigate charging
 - Coat the sample with a conductive layer
 - Work at low kV
 - Use low currents (noisy images)
 - Use low-vacuum mode
 - Charge compensation devices
 - Use the “magic” charge neutrality voltage



Fiberglass



SiO₂ substrate

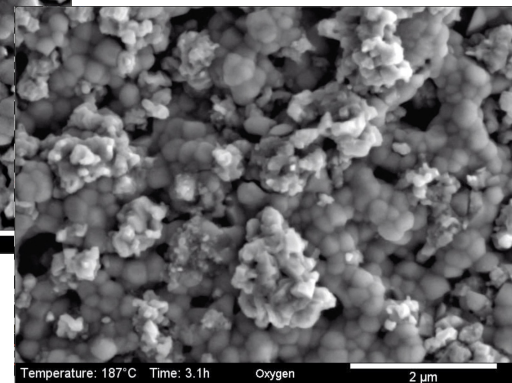
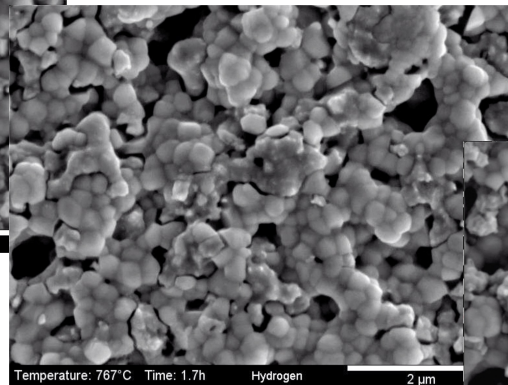
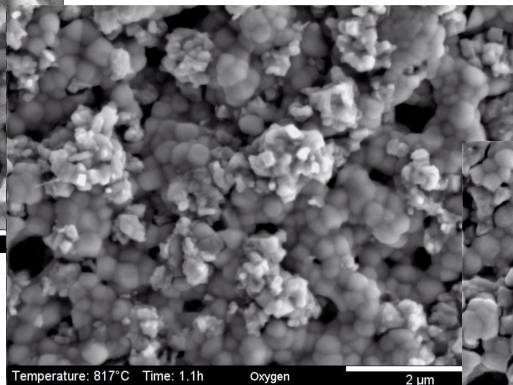
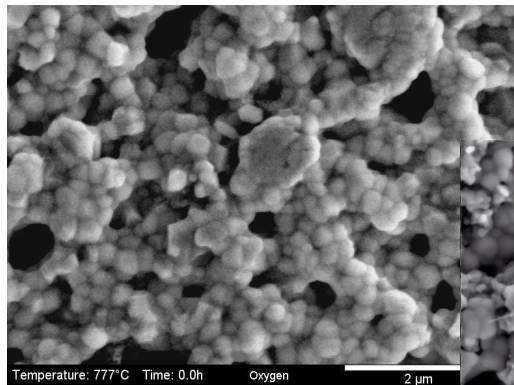


Modified Quanta200:

- 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



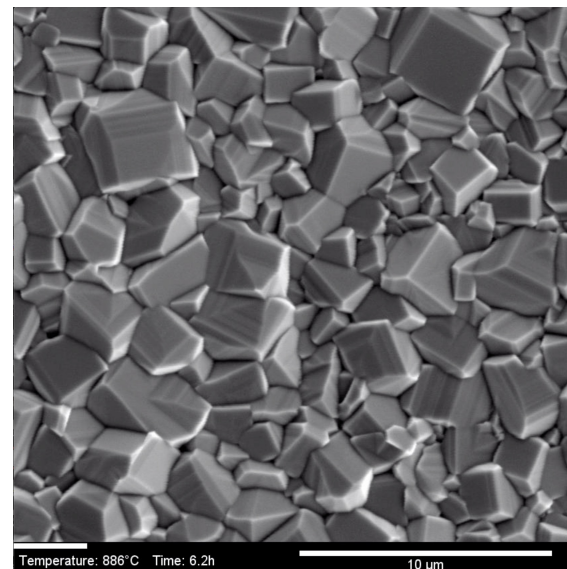
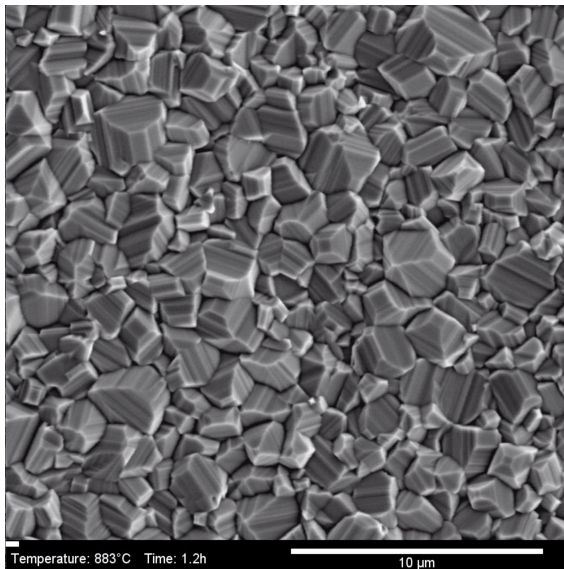
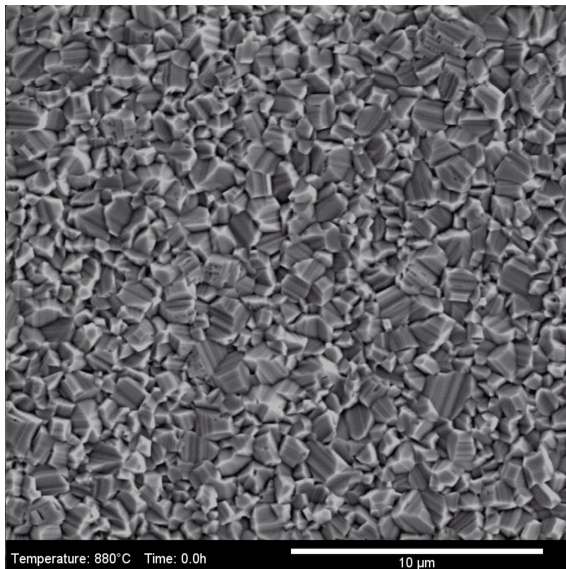
Example: SOFC anode under Redox cycling



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)

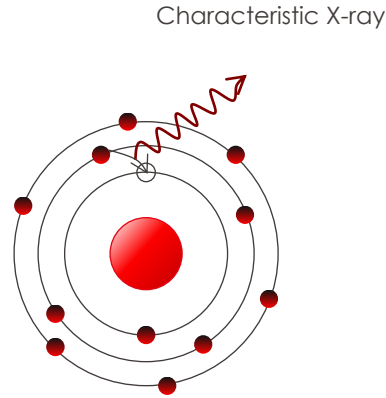
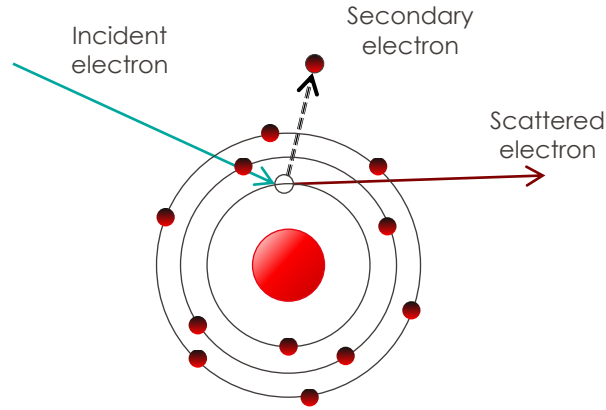
Example: SOFC coated interconnect oxidation



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

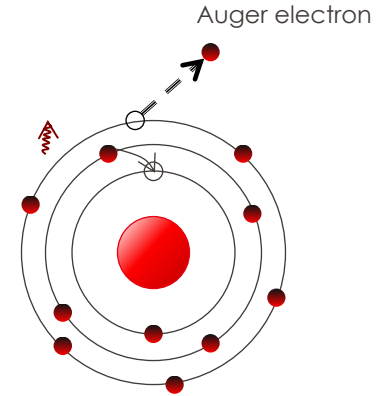
Sample: SOFC interconnect made of steel and coated with Co and Cerium.

EPFL SEM techniques | Energy dispersive x-ray spectroscopy



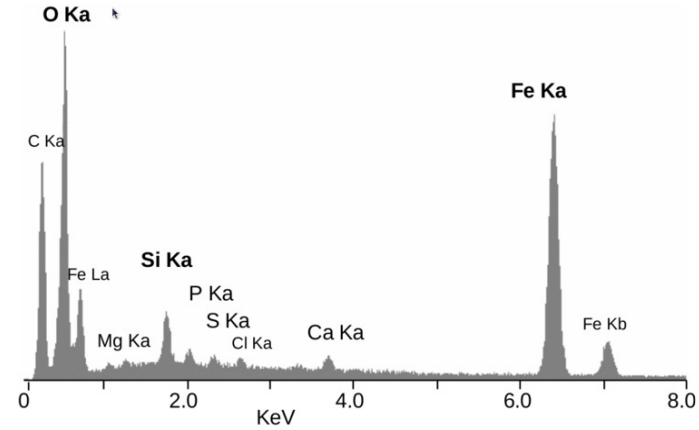
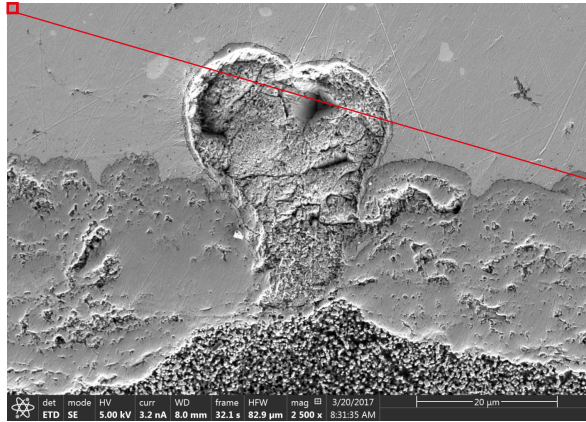
X-ray generation

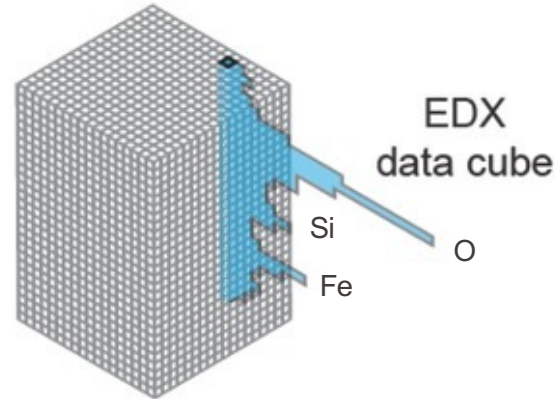
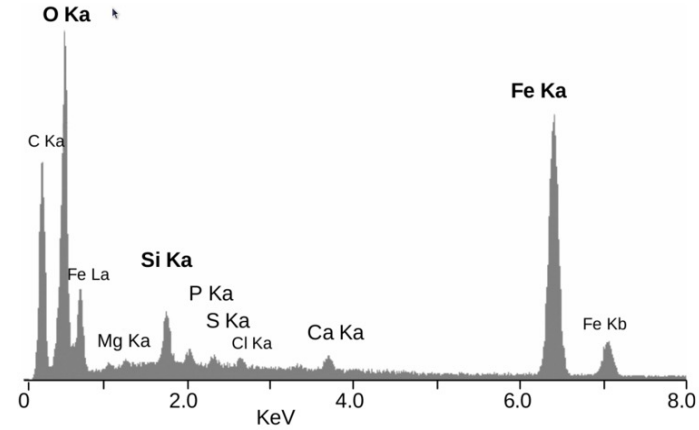
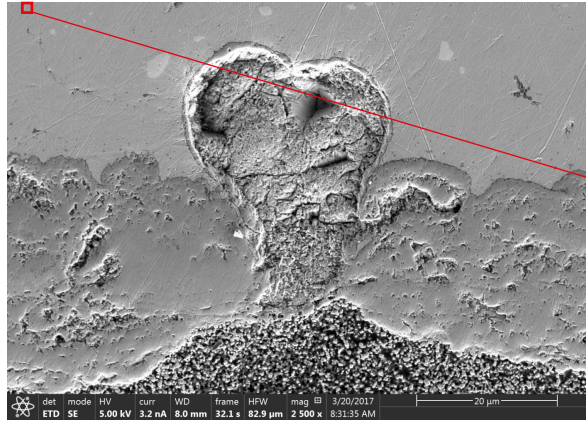
X-ray energy characteristic interorbital electron transitions and thus of the element



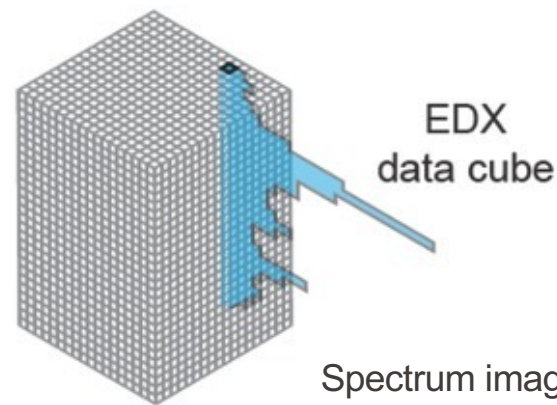
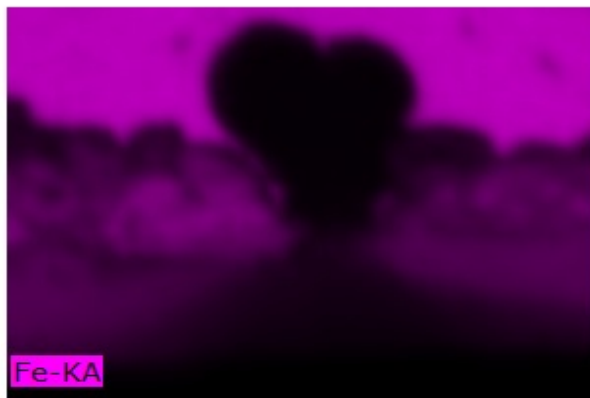
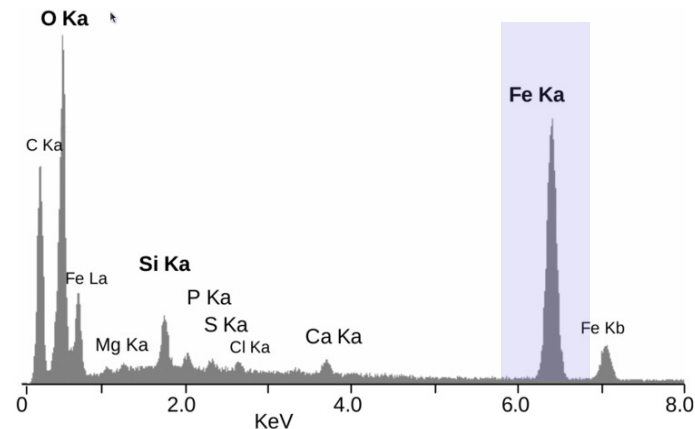
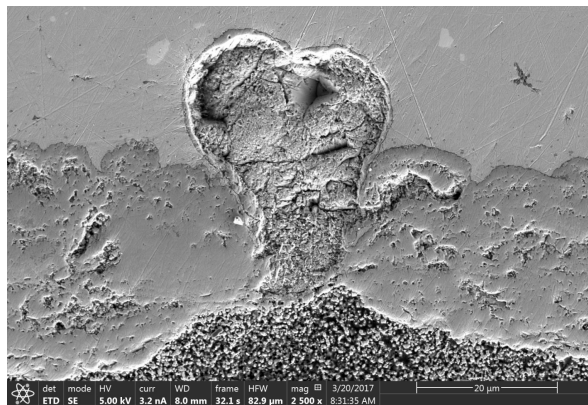
Auger emission

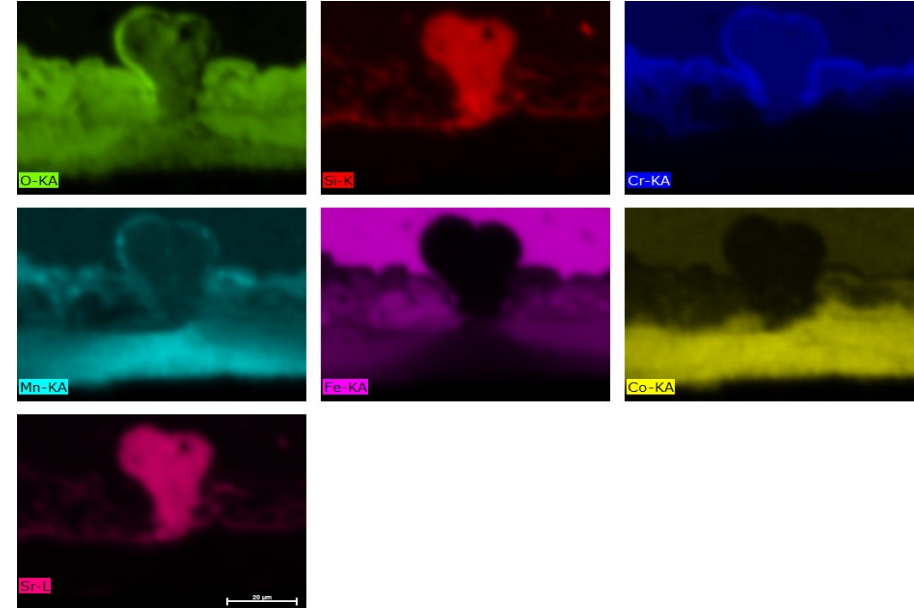
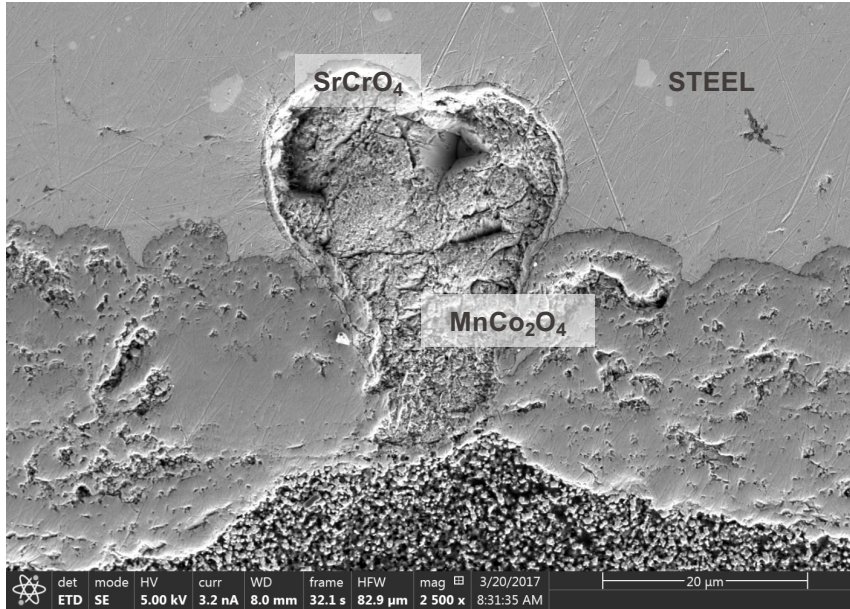
The relaxing process interacts with an electron with a characteristic energy





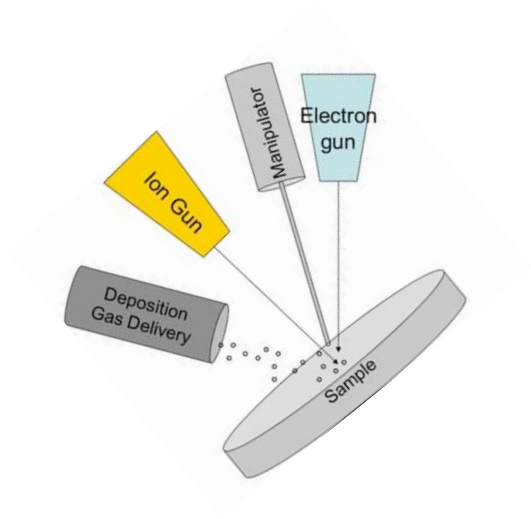
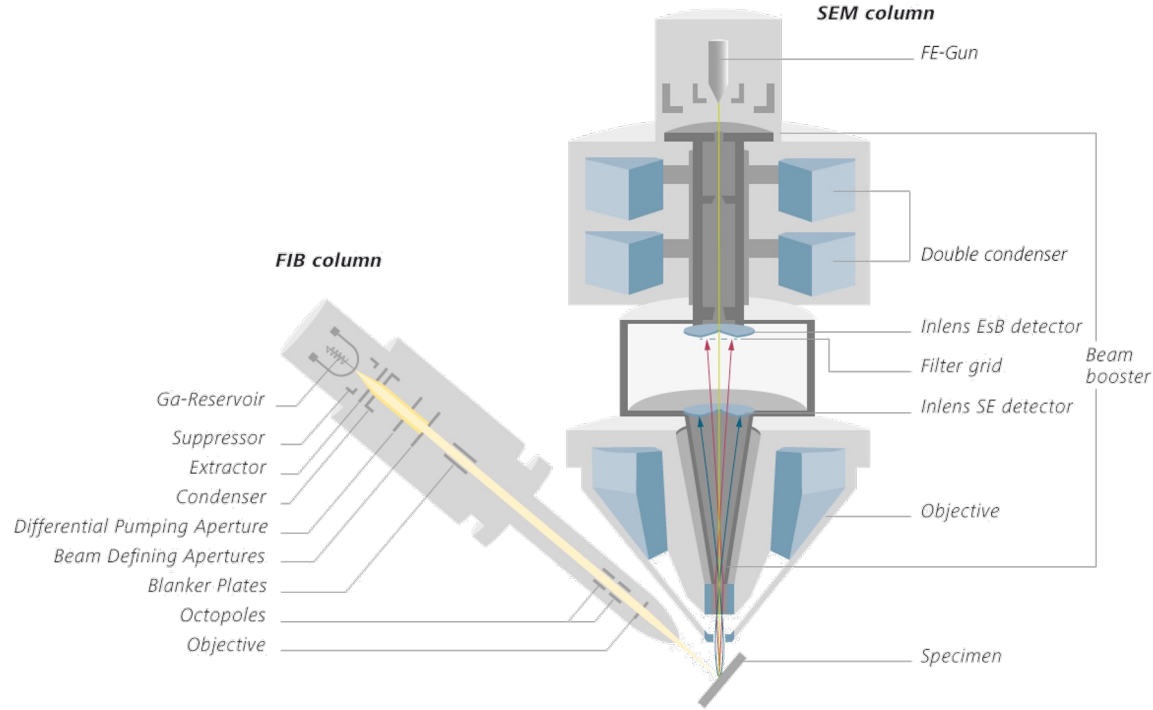
SEM techniques | Energy dispersive x-ray spectroscopy



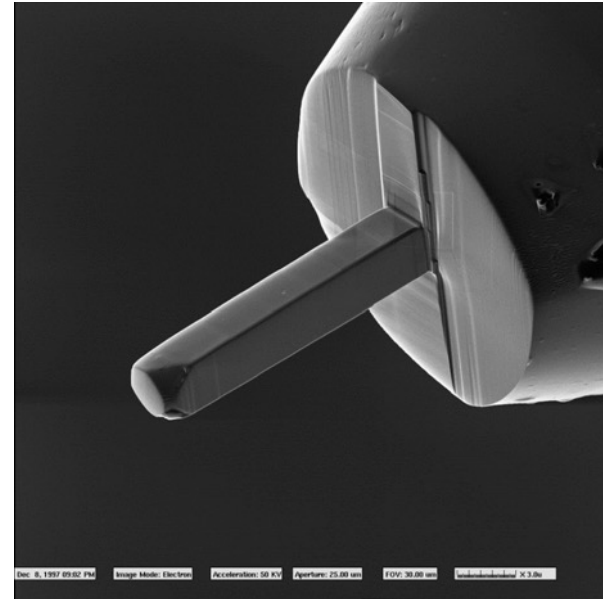
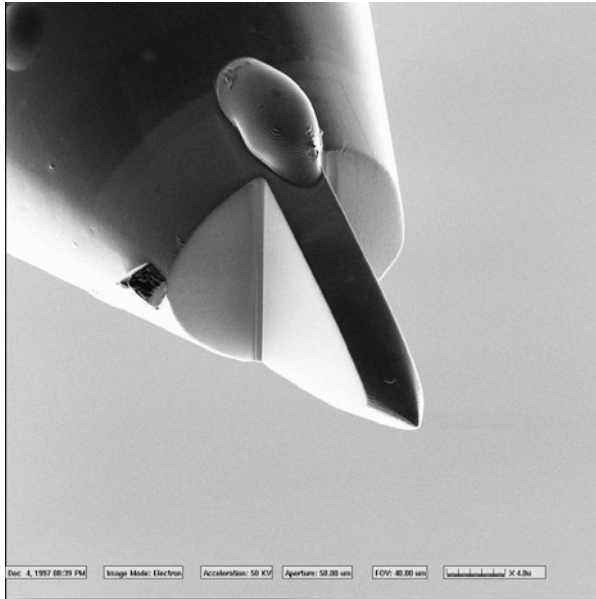


Strontium poisoning of stainless steel

- 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

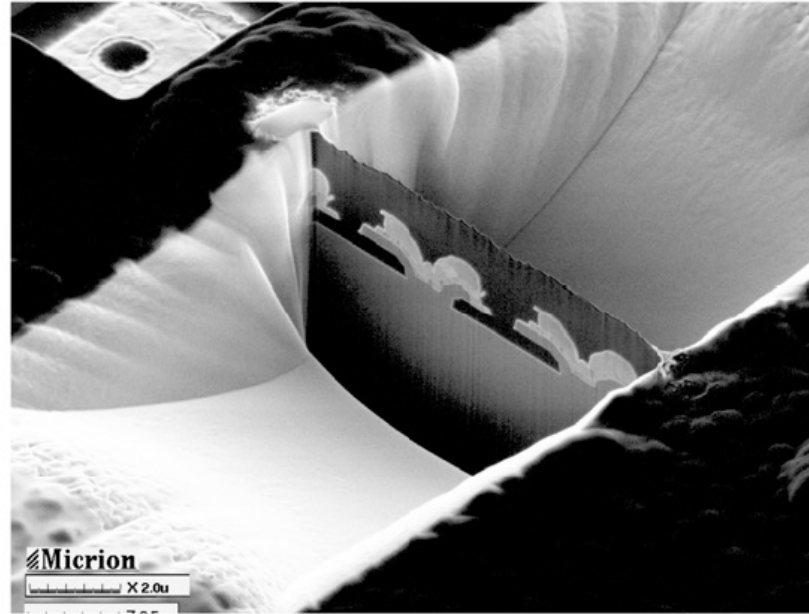


Nano fabrication



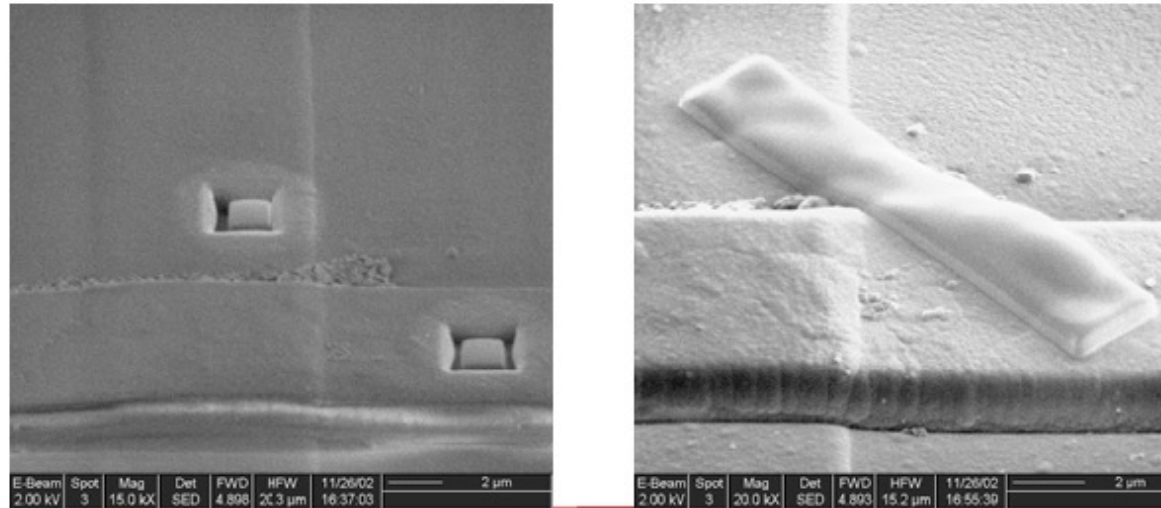
AFM tip

Cross-sectioning SEM imaging



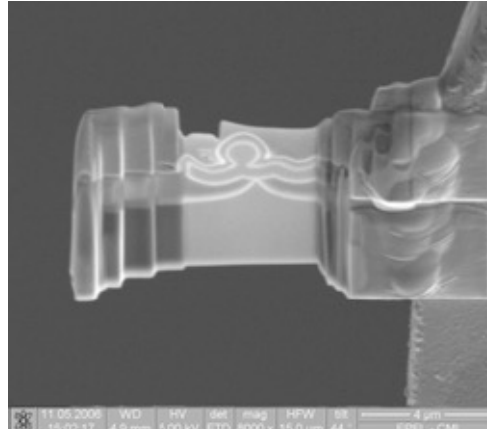
Failure analysis

Deposition

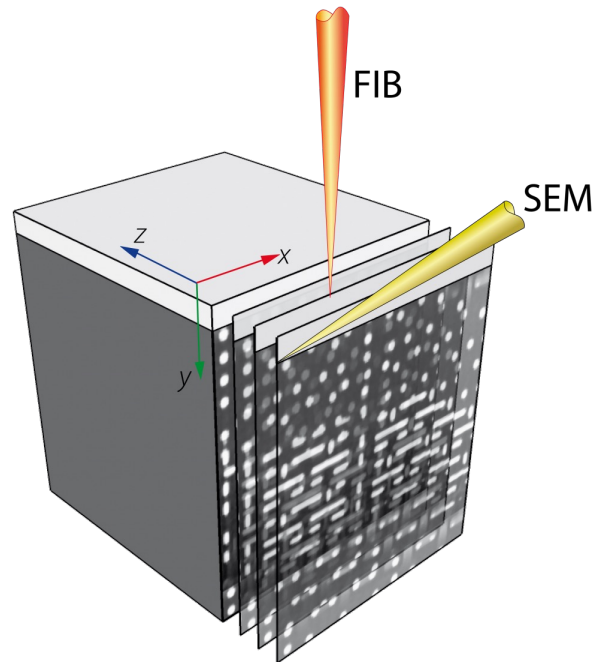


Insertion of electrical connection

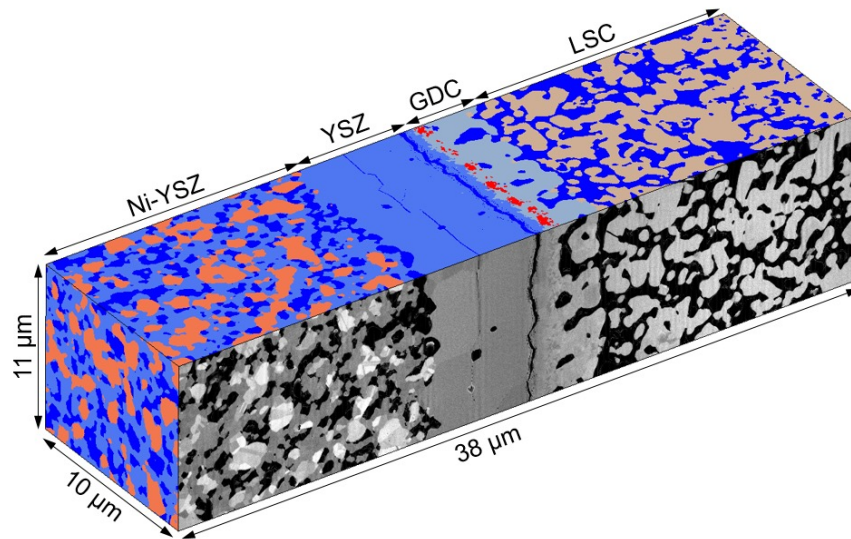
11.05.2006 WD 4.0 HV 15.0kV det. PTV mag. 400x HPW 16.0 um ttt. 4.0 min. 1.00 sec



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



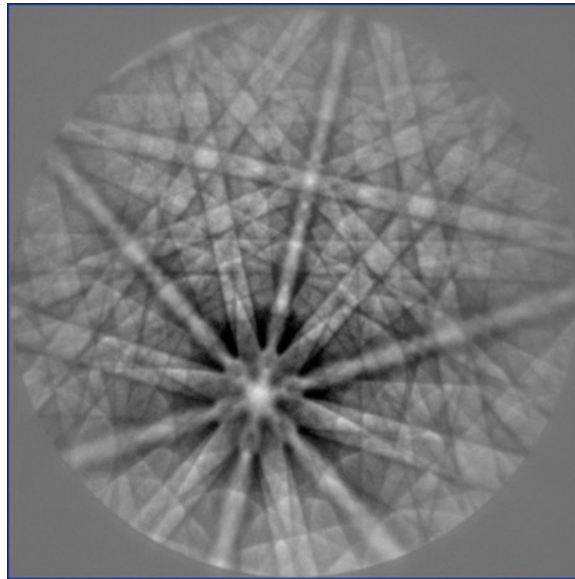
3D microscopy: SEM/FIB + EDX



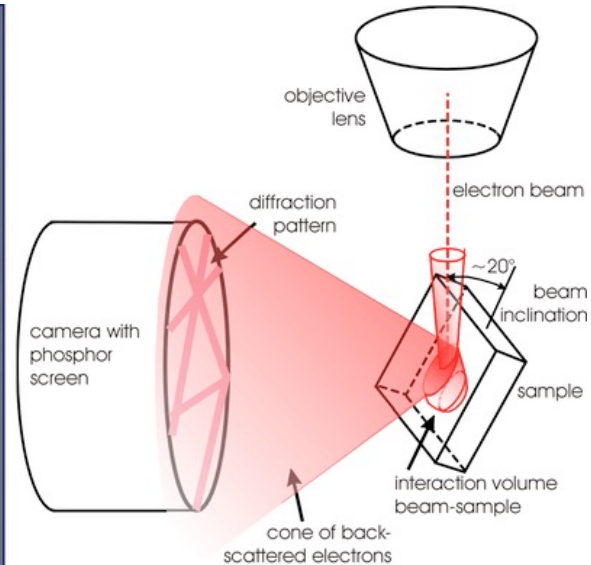
Sample: Multilayer of heterogeneous materials, e.g. Ni-YSZ/YSZ/GDC/LSCF-GDC

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

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

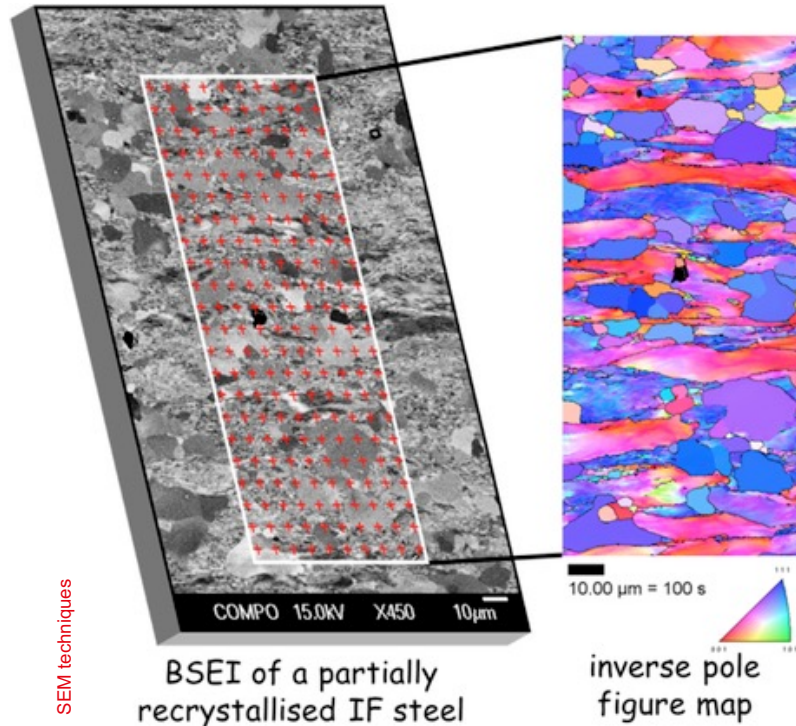


EBSD pattern of Nb (15 kV)



Geometric set-up of an EBSD measurement

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.



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

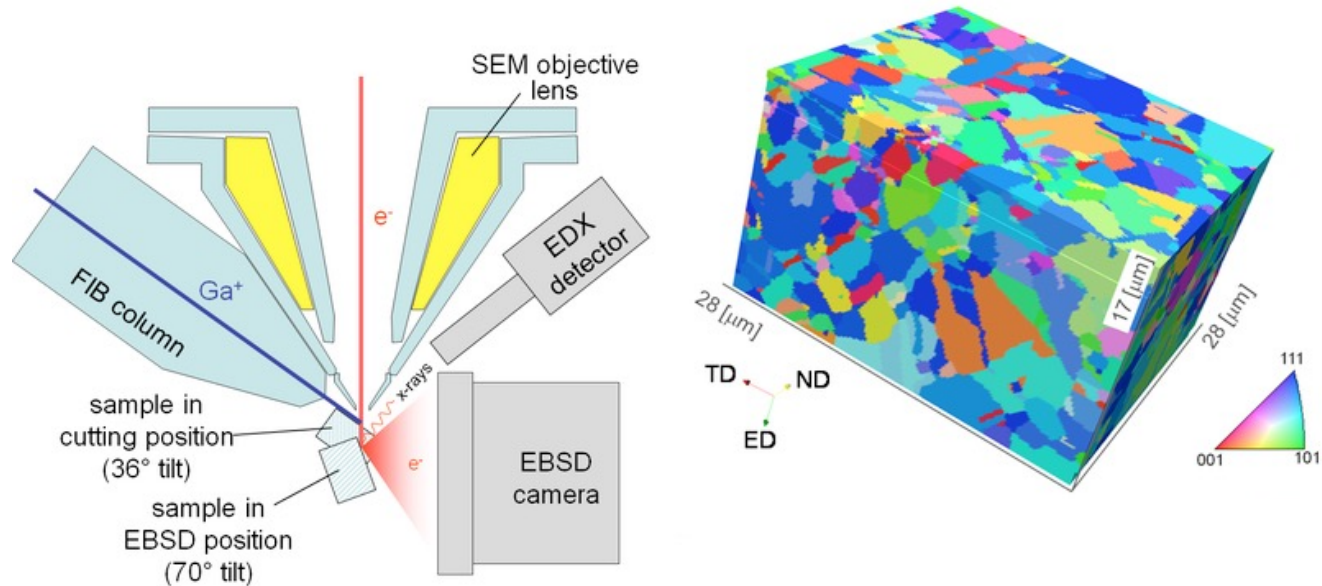
Measurement rate currently up to hundreds of patterns per second.

Investigated areas: few μm^2 to few cm^2 .

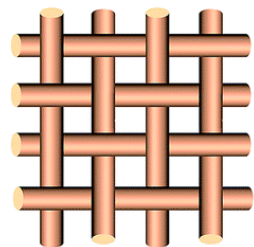
Angular resolution: 0.5° down to 0.01° with special techniques ($\rightarrow$ XR EBSD).

The technique can also be extended into a 3D technique ($\rightarrow$ grain boundaries).

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



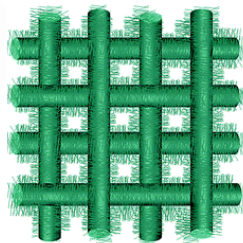
Colours correspond to the inverse pole figure position of the sample normal direction.



Cu gauze

Wet chemical oxidation

2.5 M KOH
0.125 M $(\text{NH}_4)_2\text{S}_2\text{O}_8$
10 min

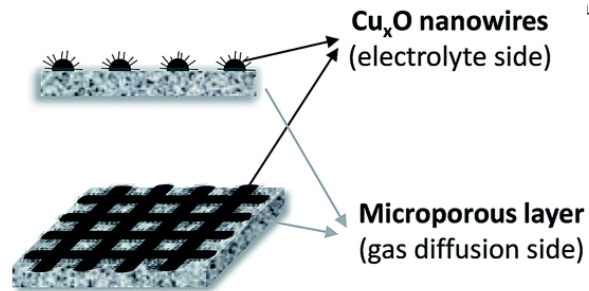


Spray coating

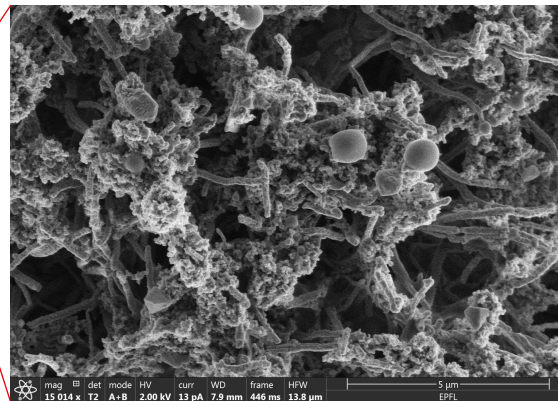
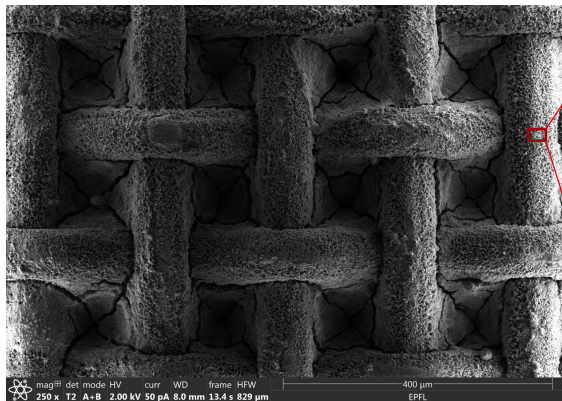
PTFE + carbon black

Heating

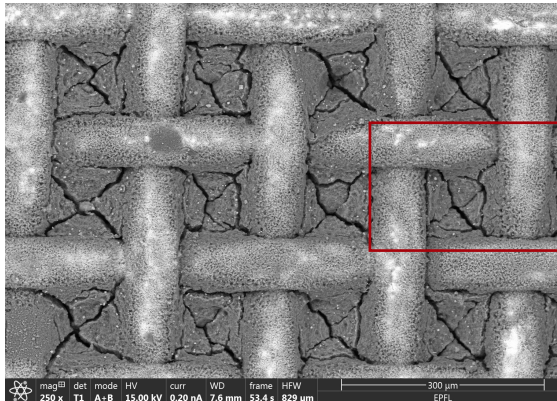
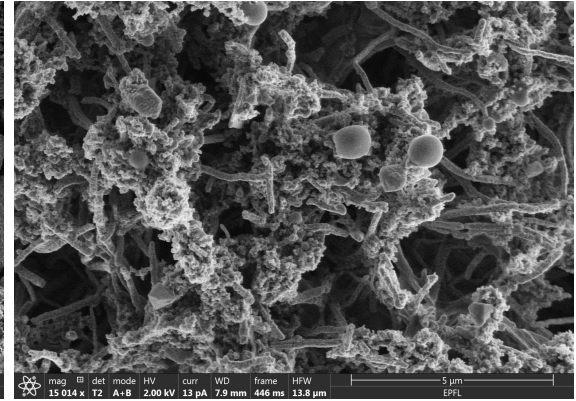
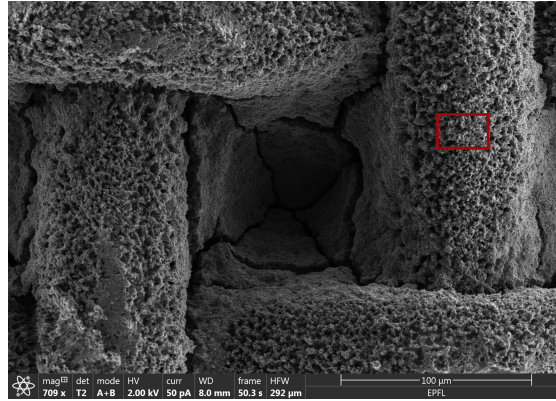
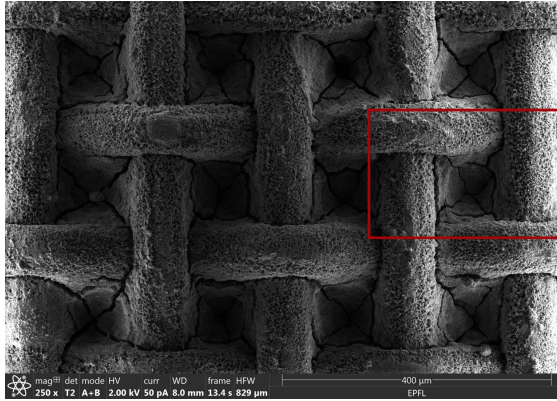
350 °C in air for 1h

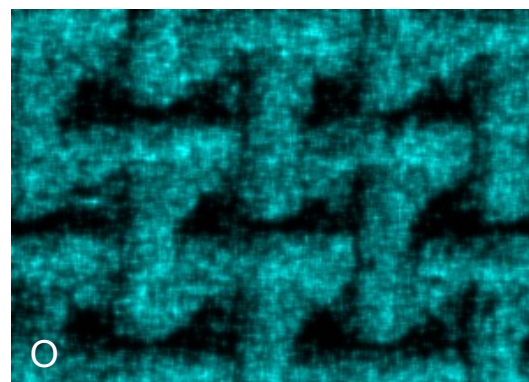
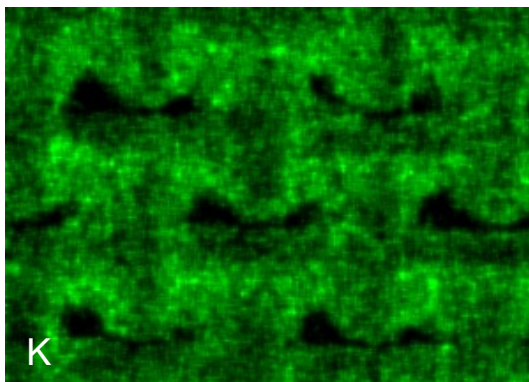
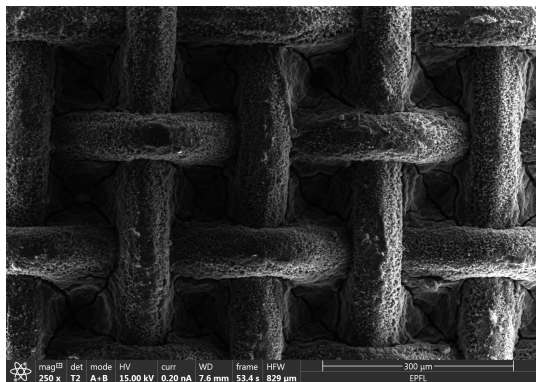
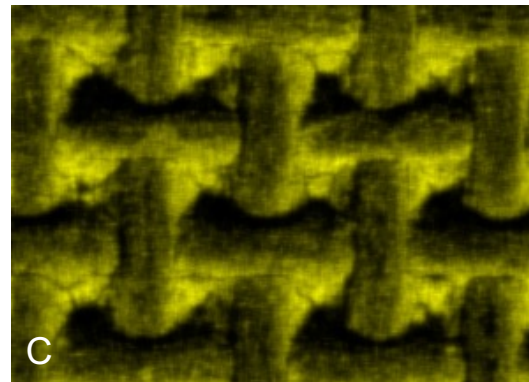
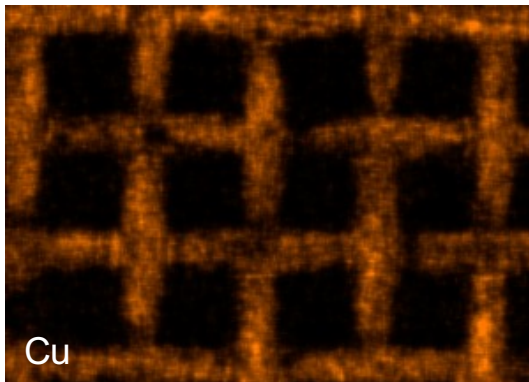
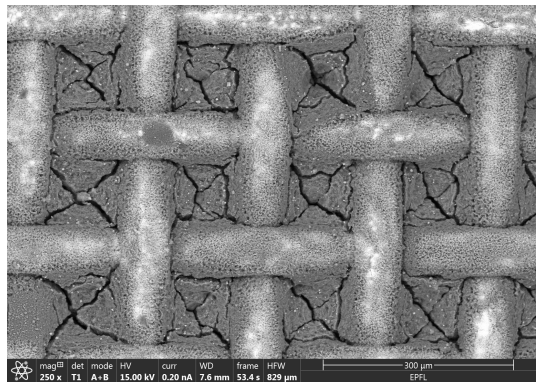


Gas diffusion electrode

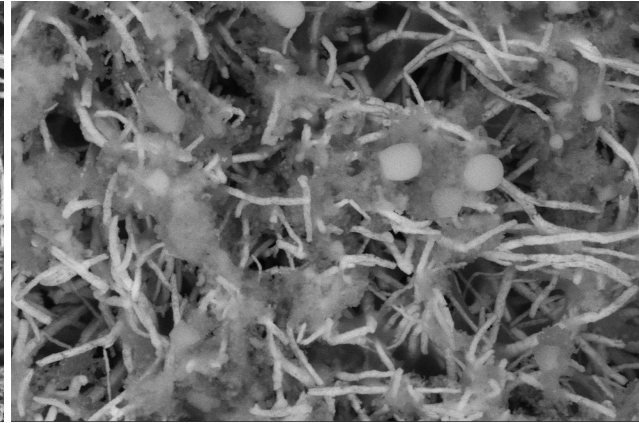
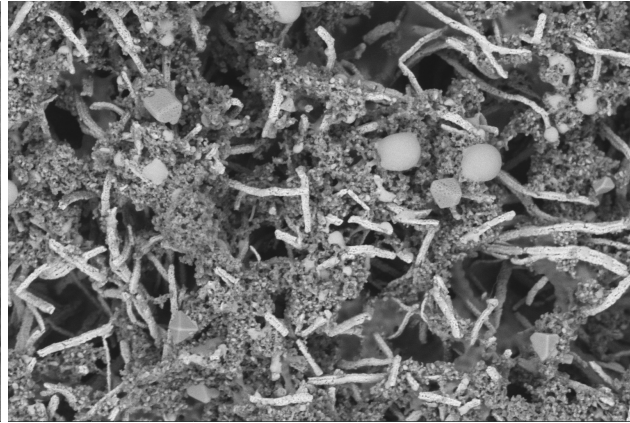
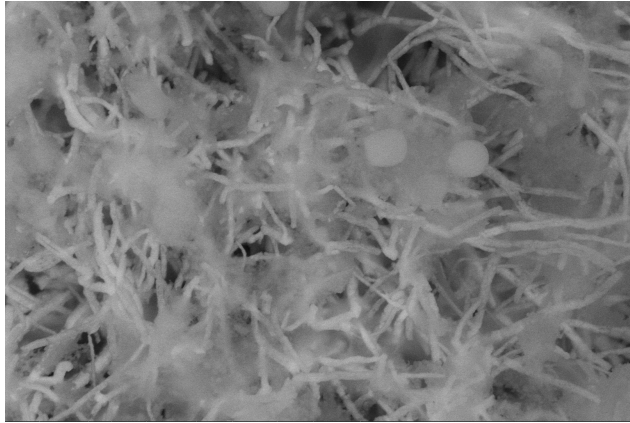


Compare and interpret the contrast you see in the BSE/SE images

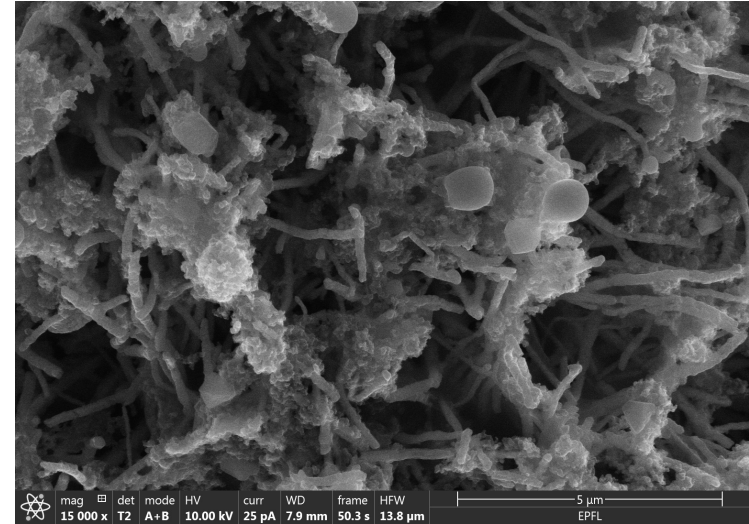
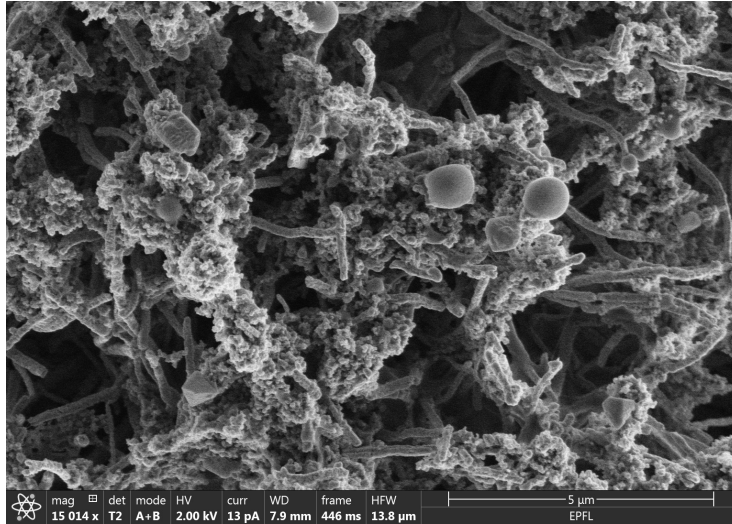




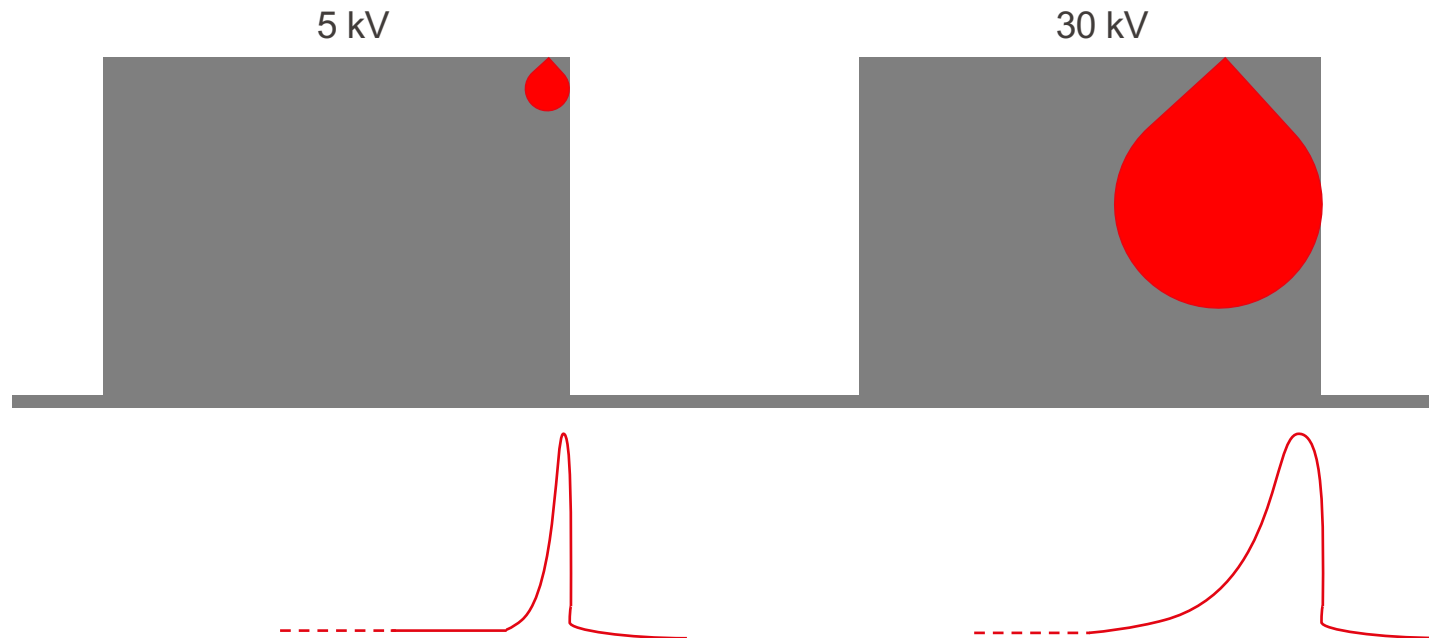
Discuss the changes visible in these BSE images?



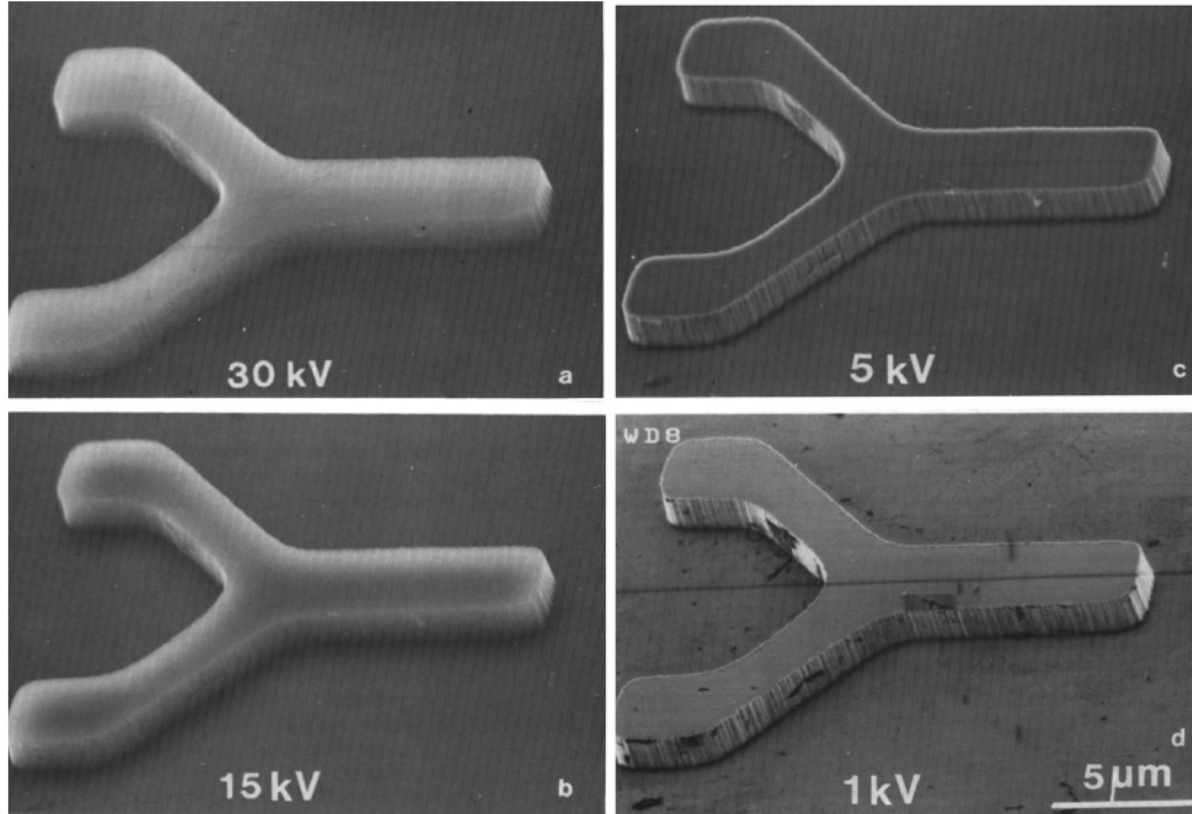
Discuss the changes visible in these SE images acquired at high and low beam energies?

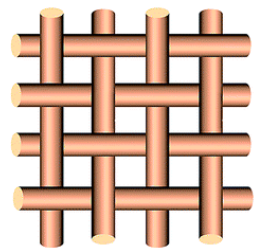


Note the “Edge effect”



SEM signals | Edge effect & beam energy

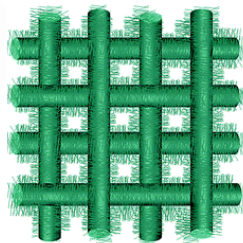




Cu gauze

Wet chemical oxidation

2.5 M KOH
0.125 M $(\text{NH}_4)_2\text{S}_2\text{O}_8$
10 min

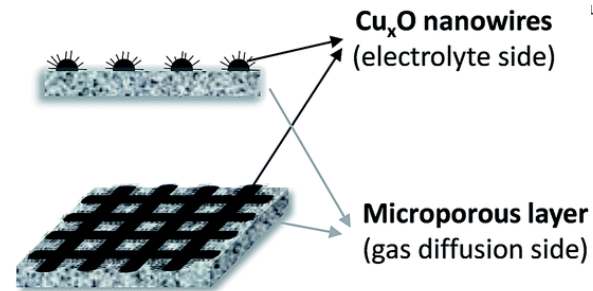


Spray coating

PTFE + carbon black

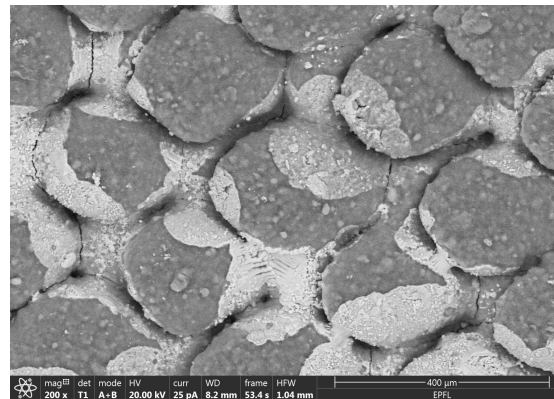
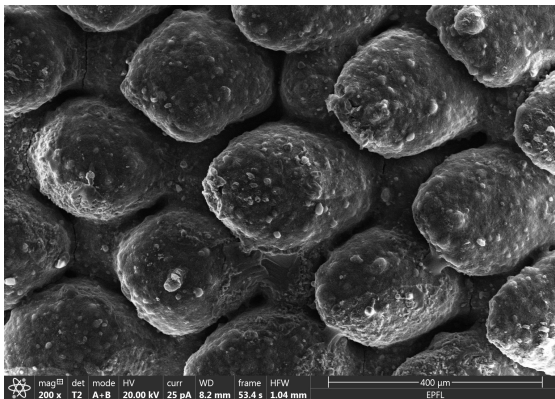
Heating

350 °C in air for 1h

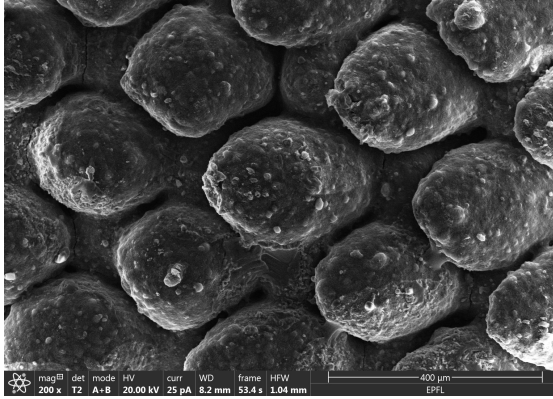


Gas diffusion electrode

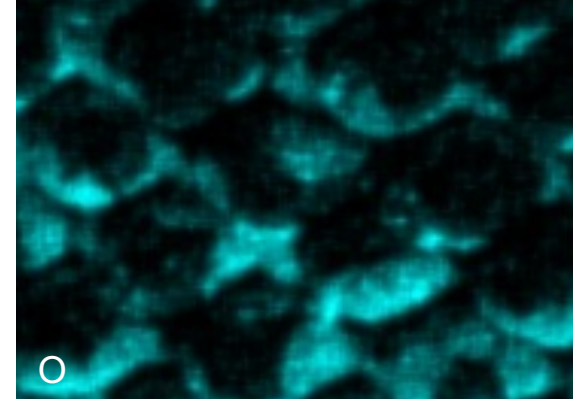
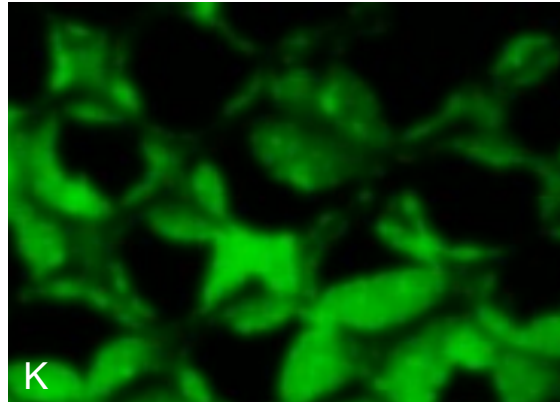
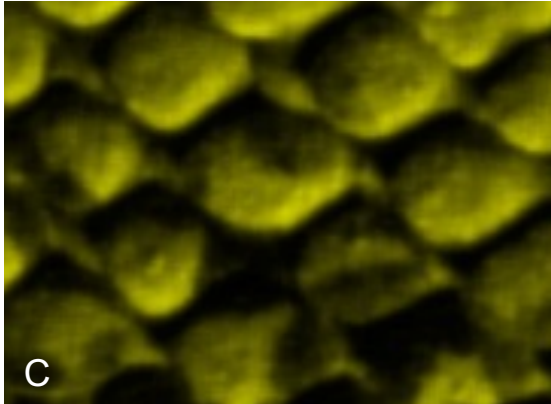
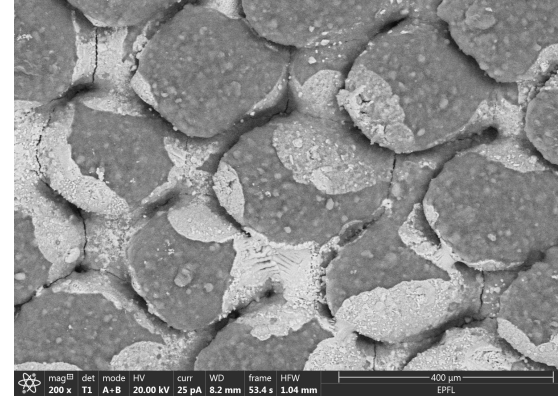
Discuss the contrast difference between the images?



SE

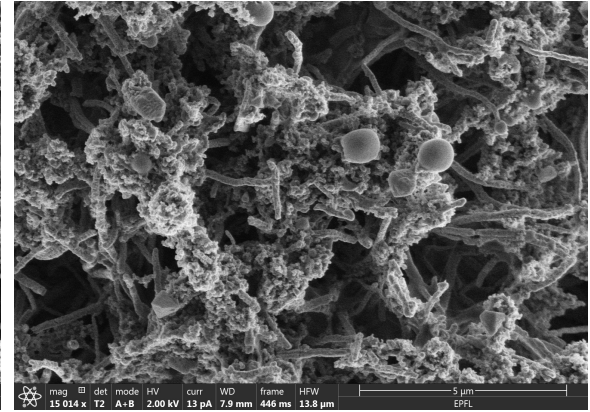
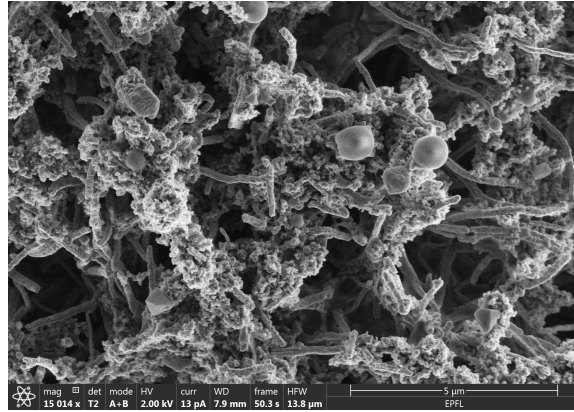
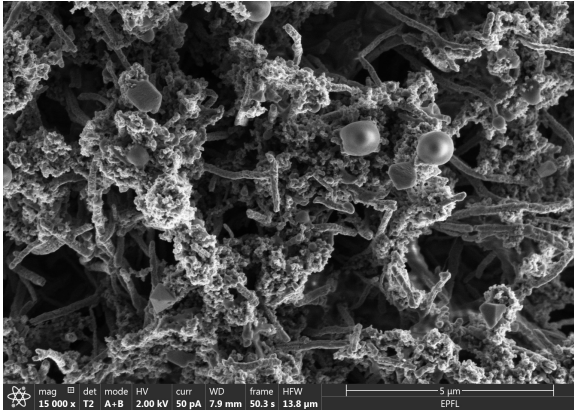


BSE



Discuss the artefacts visible in some of the images.

How it has been resolved?



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

