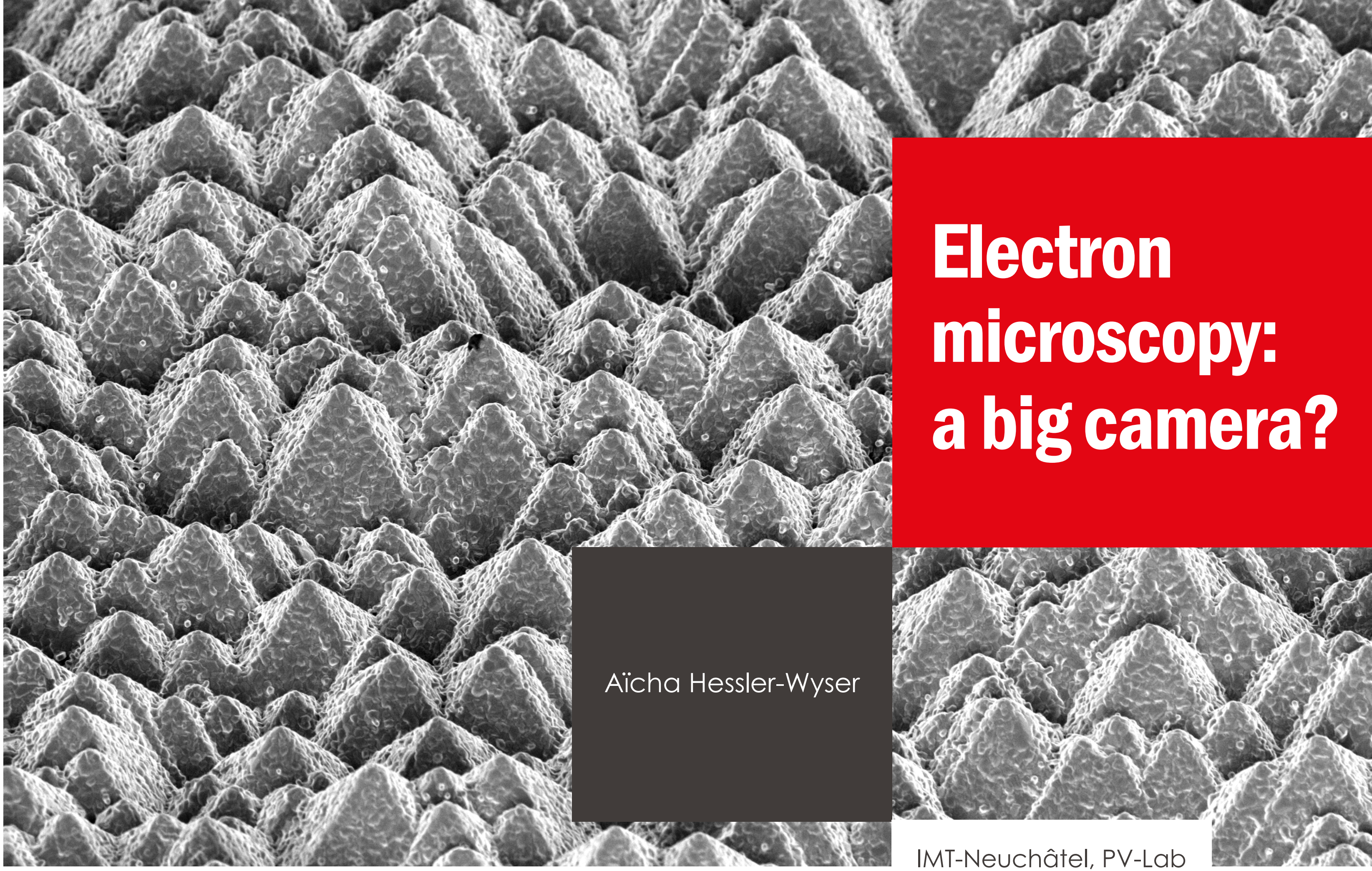
EPFL

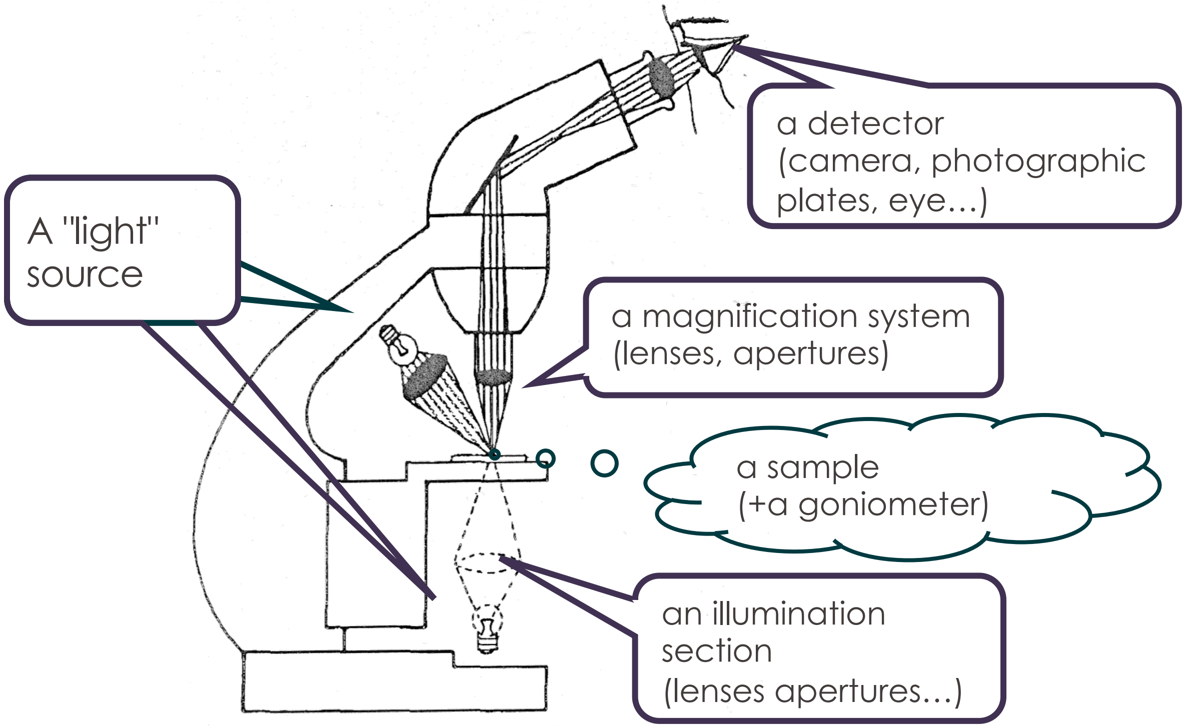


Electron microscopy: a big camera?

Aïcha Hessler-Wyser

IMT-Neuchâtel, PV-Lab

A microscope, it is...



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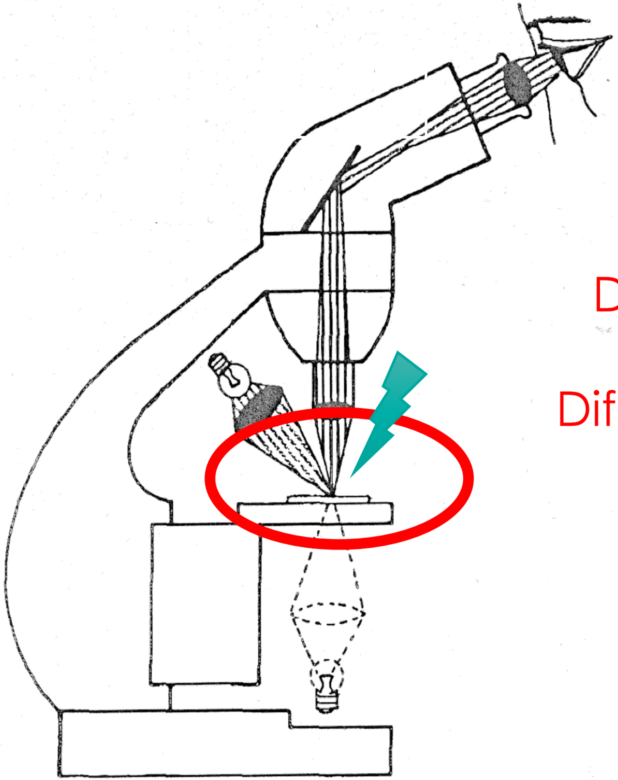


Different signals
Different detectors
=
Different informations!

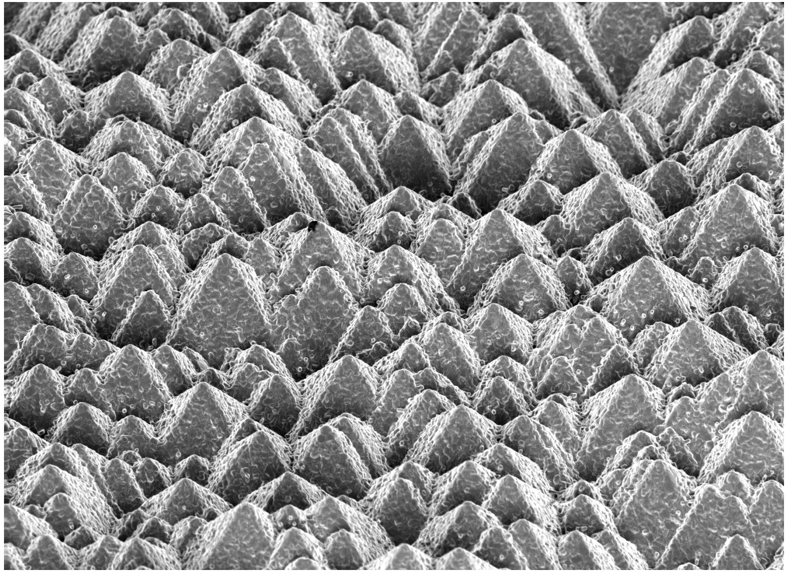
Content

- Brief introduction and history
- Why electrons?
- Scanning electron microscope (SEM)
- Transmission electron microscope (TEM)
- Other (analytical) techniques (EDS, EELS, EBSD, FIB)

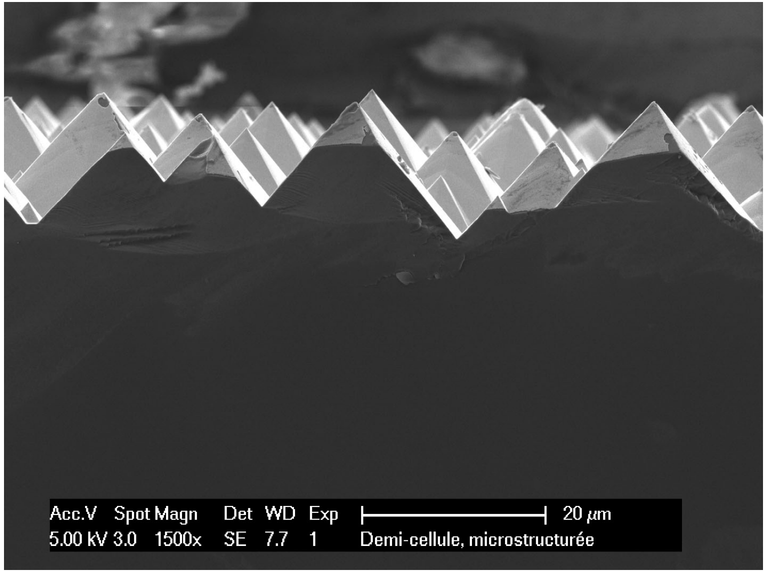
Learn about the potential and capability of electron microscopy, and chose the right method for your desired information

Intro: what can be done with electron microscopy?

- Topography: SE imaging



Perovskite/silicon tandem solar cells, with textured wafer, *Q. Jeangros*

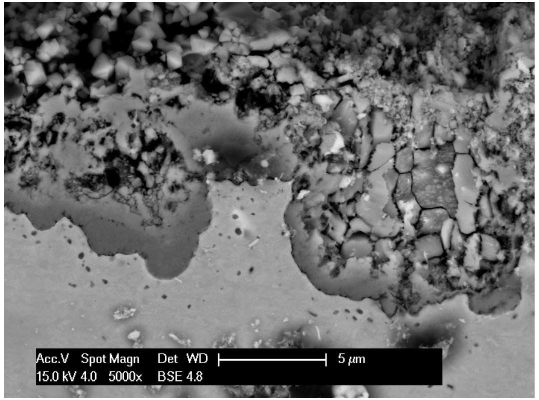


Si-based solar cells, with textured wafer, *G. Pasche*

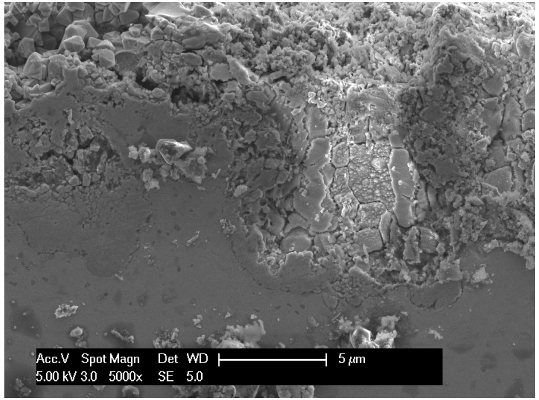
Aïcha Hessler-Wyser

Intro: what can be done with eletron microscopy?

- Imaging with Z contrast: BSE imaging



Backscattered electron detector



Secondary electron detector

Steel interconnects used in solid oxide fuel cells, after >1000h of operation

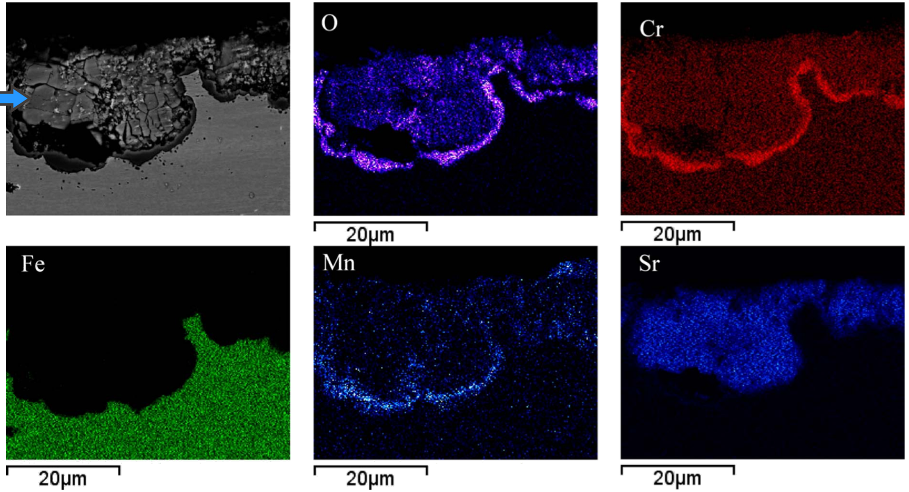
Aïcha Hessler-Wyser

Intro: what can be done with eletron microscopy?

Aïcha Hessler-Wyser

- Imaging by element maping: EDS

Attacked steel formation of $SrCrO_4$ precipitates

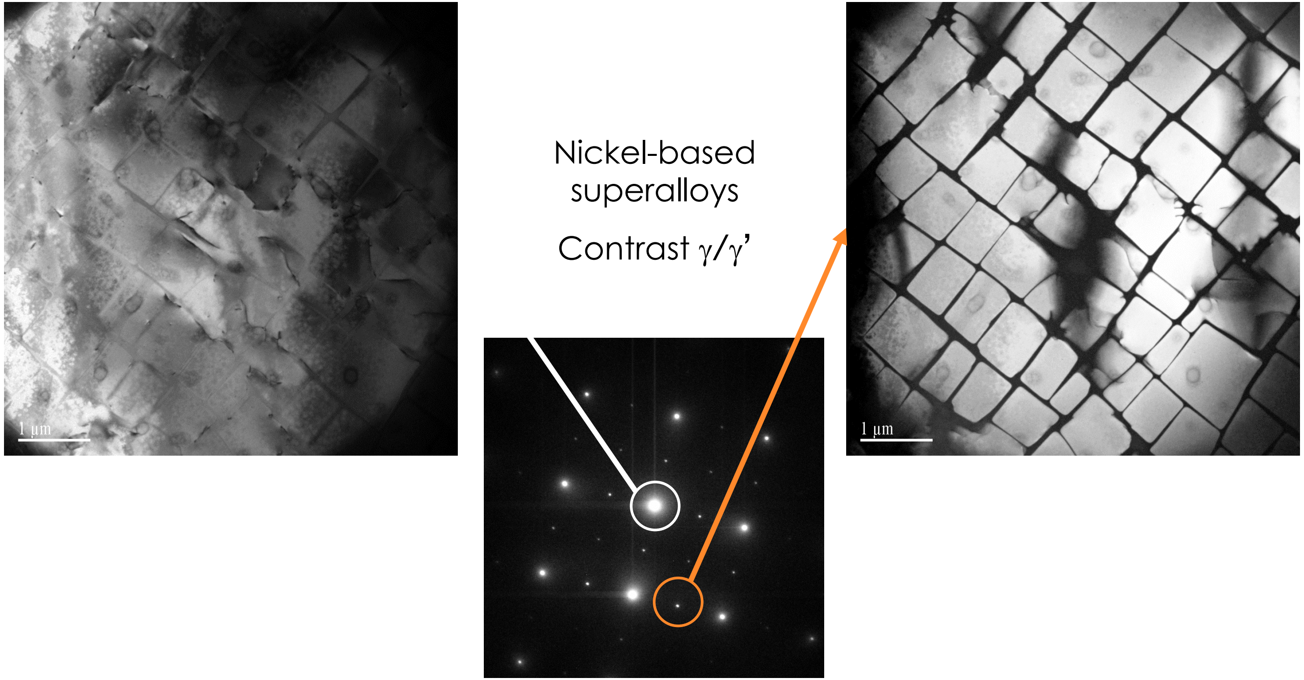


Cr_2O_3

Steel interconnects used in solid oxide fuel cells, after >1000h of operation

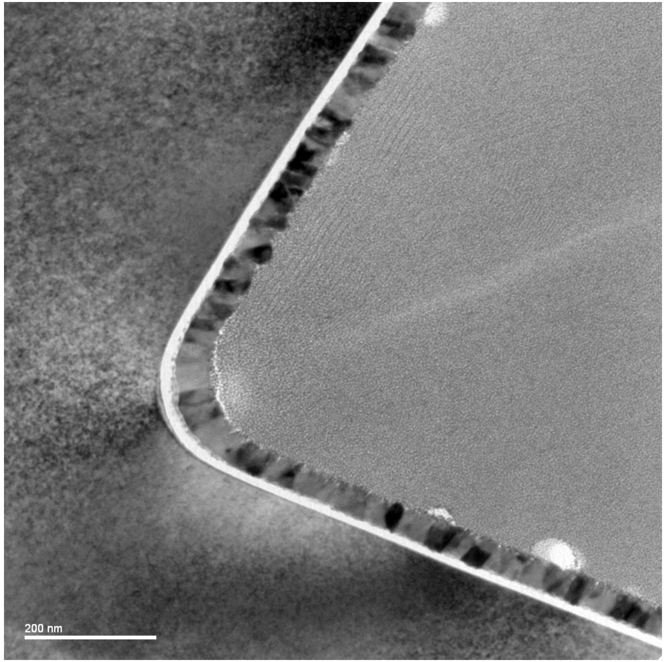
Intro: what can be done with eletron microscopy?

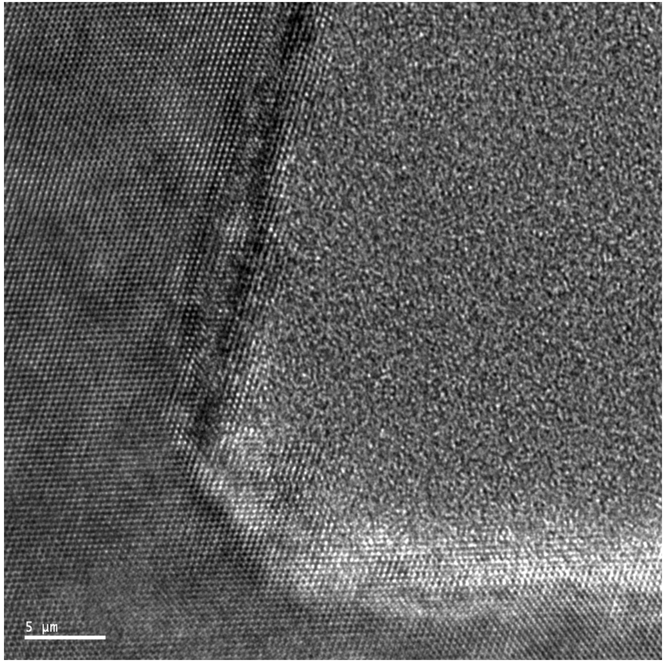
- Imaging the diffraction contrast



Intro: what can be done with eletron microscopy?

- Imaging the atomic column: BF vs HRTEM

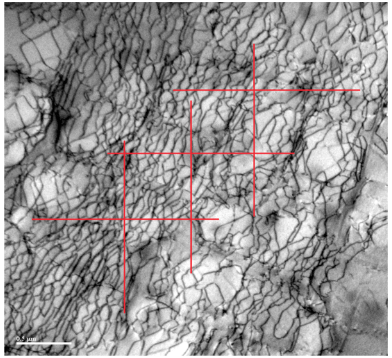




Si heterojunction solar cell: monocrystalline Si substrate, textured

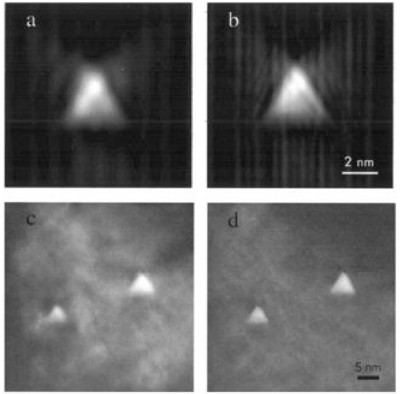
Intro: what can be done with eletron microscopy?

- Imaging crystal defects

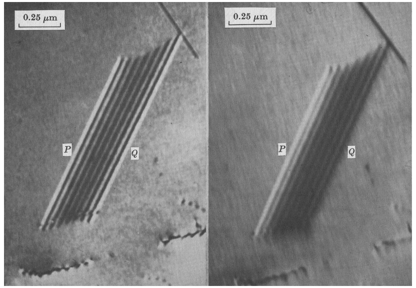


Dislocation network

Stacking faults tetrahedras



R. Schäublin

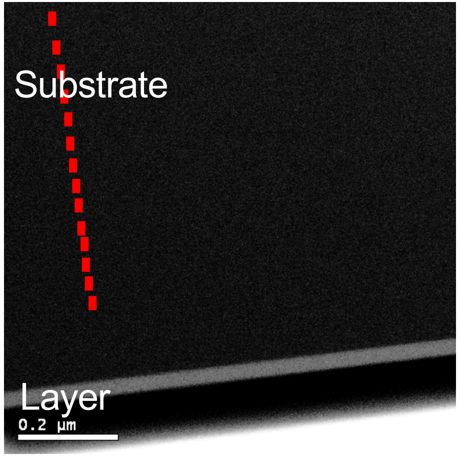


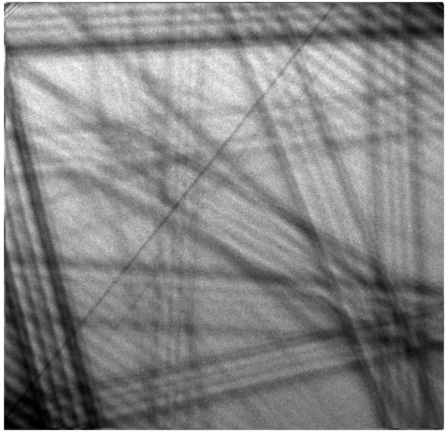
Stacking faults

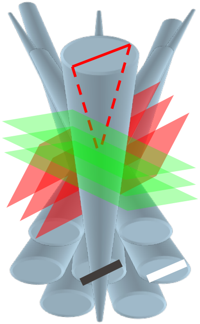
Carter et Williams

Intro: what can be done with eletron microscopy?

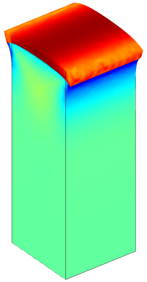
- Imaging stress strain: convergent beam diffraction







Lines profile evolution

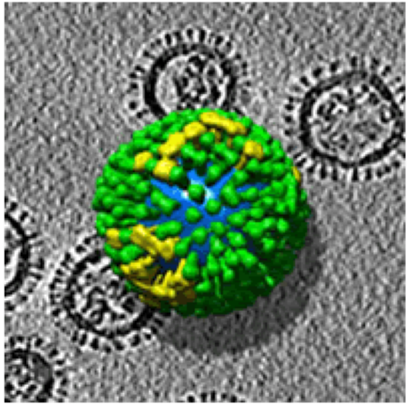


Compressive case

Layer $Si_{0.5}Ge_{0.5}$ on Si wafer, *F. Houdelier*

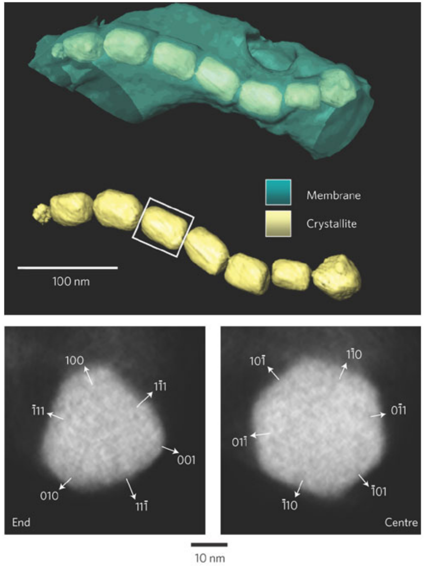
Intro: what can be done with eletron microscopy?

- Imaging volumes: tomography



3D structure of the influenza virus (size env. 120 nm)

Harris A, *et al*. Influenza virus pleiomorphy characterized by cryoelectron tomography. *PNAS* 2006;103(50):19123-19127.

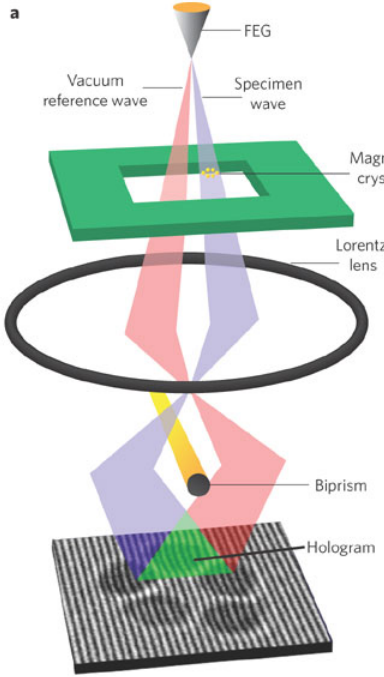


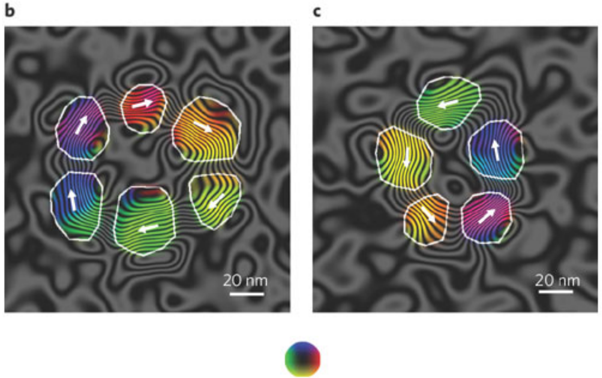
3D reconstruction of a magnétotactic bacteria: magnetite inside a bacteria membrane

Paul A. Midgley & Rafal E. Dunin-Borkowski
Nature Materials **8**, 271 - 280 (2009)

Intro: what can be done with eletron microscopy?

- Imaging magnetic field: electron holography





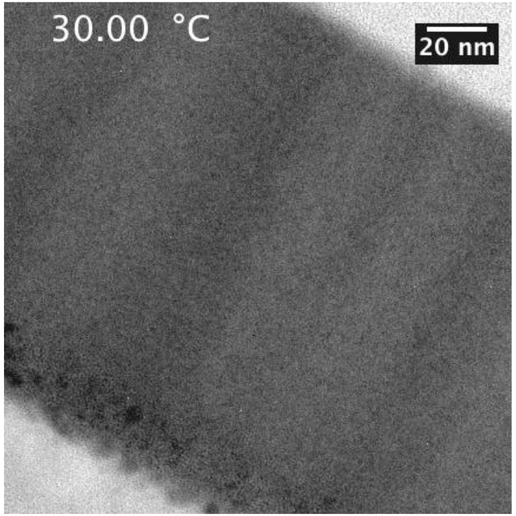
Hologramme off-axis, montrant la projection de la densité de flux magnétique à l'intérieur et autour de nanocristaux de Co.

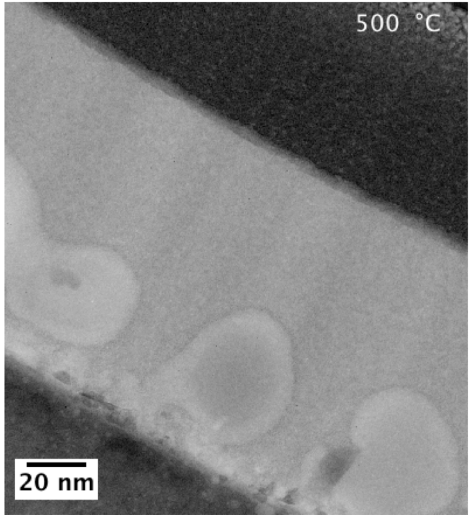
Paul A. Midgley & Rafal E. Dunin-Borkowski
Nature Materials **8**, 271 - 280 (2009)

Aïcha Hessler-Wyser

Intro: what can be done with eletron microscopy?

- Imaging dynamic events: *in situ* microscopy

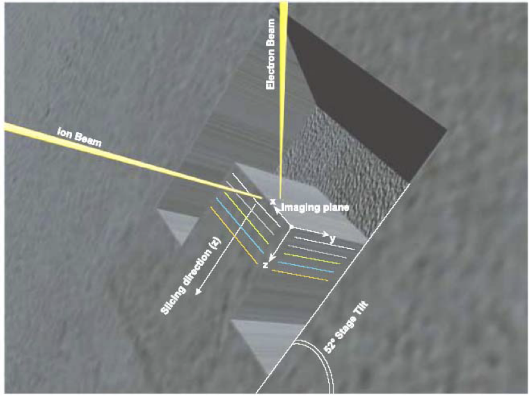


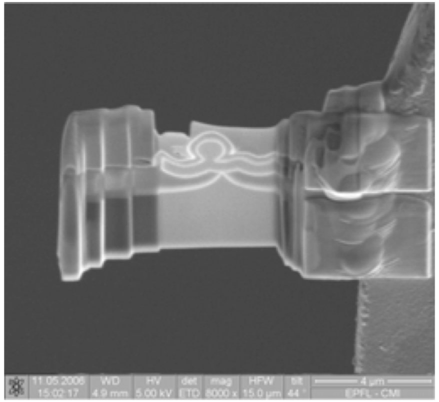


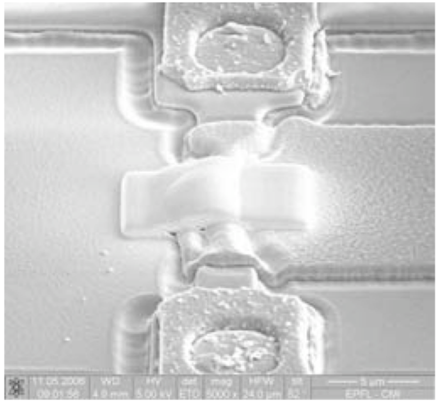
TCO on glass during heating and cooling, Q. Jeangros

Intro: what can be done with eletron microscopy?

- Nano-machining materials: FIB



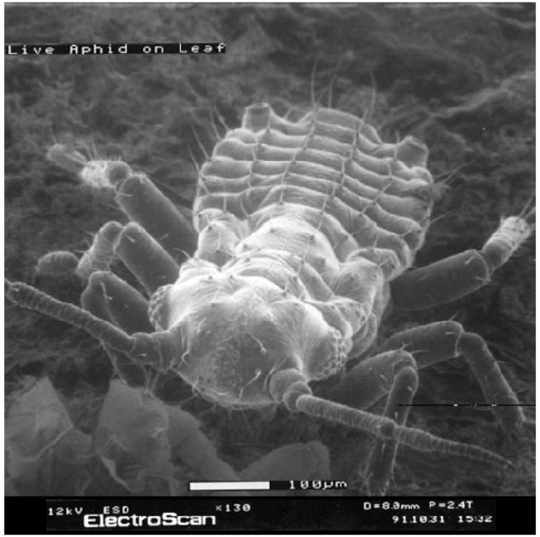




Intro: what can be done with eletron microscopy?

- Imaging "living" samples

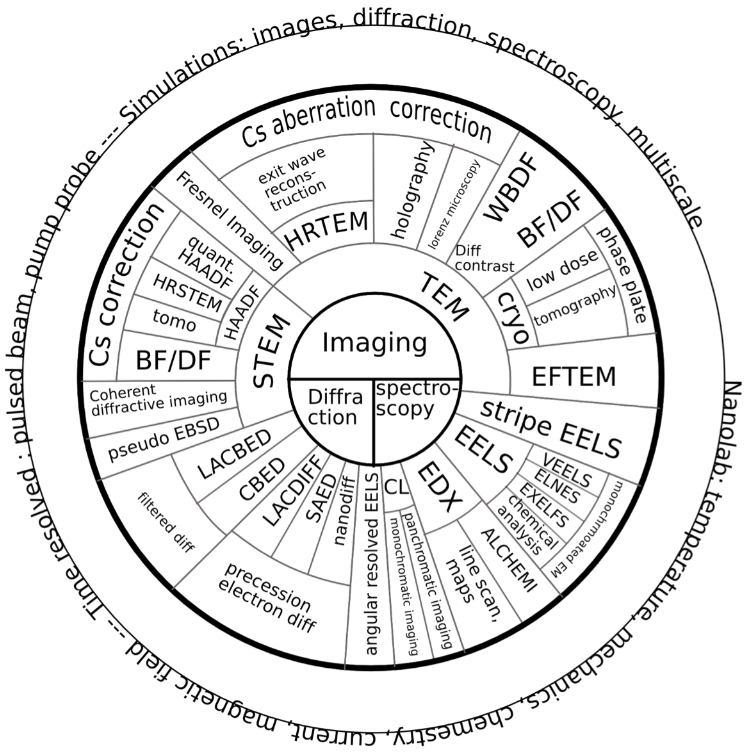
- Partial atmosphere



Intro: what can be done with eletron microscopy?

- And much more…

This is only for TEM!



EPFL

A bit of history...

Aïcha Hessler-Wyser

1665

2017

A bit of history: optics

- Optical microscopy

  Antiquity: first etch of convex lenses

  XII-XIIIth centuries: magnification power of convex lenses, magnifier, glasses

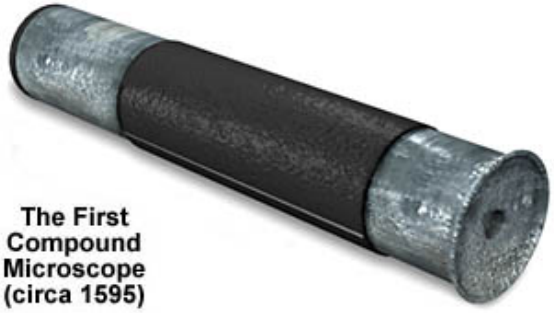
  1590 Janssen, first composed microscope

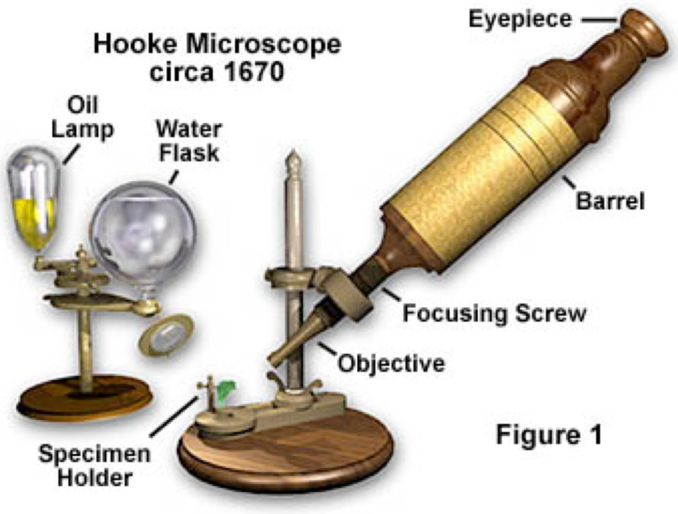
  1609 Galilei: occhiolino

  1665 Hooke: first cell image

  1801 Young: wave nature of light

  1872 **(~) Abbe: the resolution limit is linked to wave length of the used beam**





A bit of history: electrons

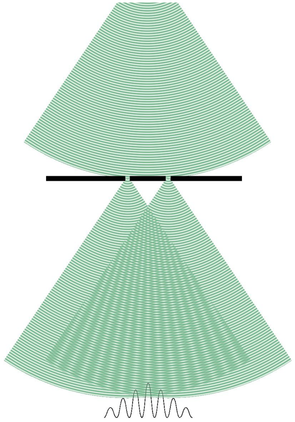
Aïcha Hessler-Wyser

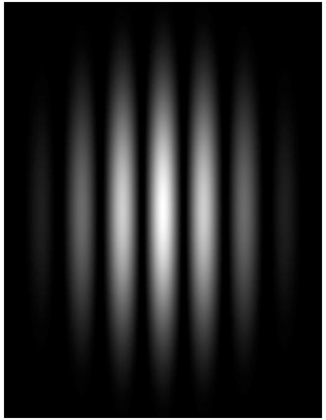
- Electron microscopy

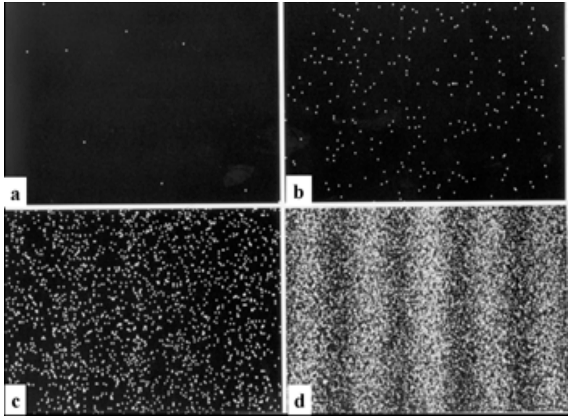
  ***1923* De Broglie: concept of wavelength associated to particles, confirmation by Young's experiment**

  1927 Busch: focalisation low for magnetic fields
  Davisson, Germer, Thomson: electron diffraction

  1931 Ruska, Knoll: first images by electron microscopy







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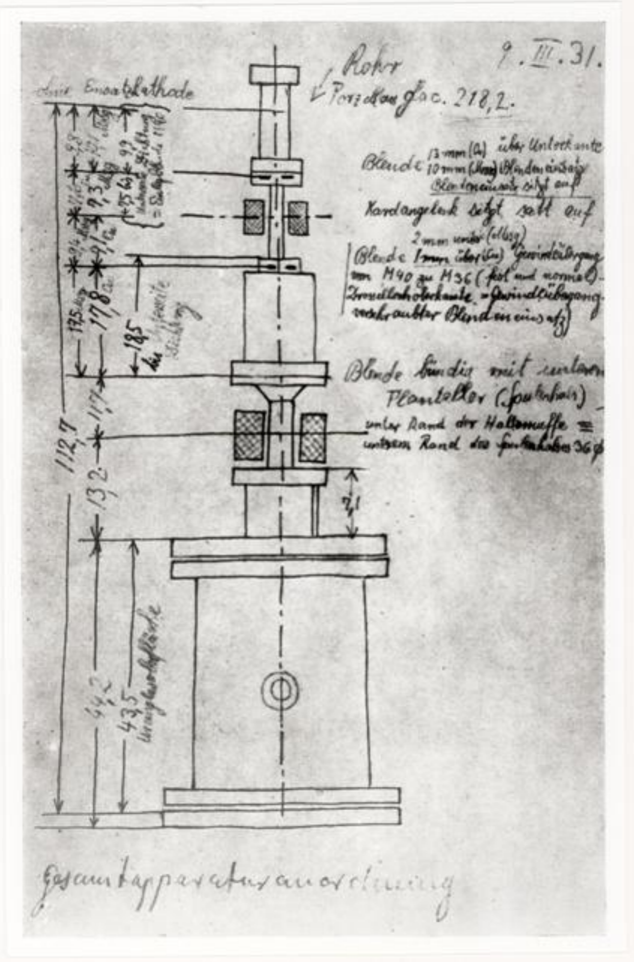

Aïcha Hessler-Wyser

A bit of history: electron microscopy

Aïcha Hessler-Wyser

1936 Scherzer: main electromagnetic lens aberrations cannot be avoided

1938 Von Ardenne: first microprobe scanning electron microscope

1939 Siemens: first industrial electron microscopes

1945 Hillier: invention of stigmators, open door for high resolution

1948 Gabor: holography invention

1951 Castaing: first X-ray micro-analyser

1960 XX: first MV microscope, competition for resolution

1965 Crewe: first scanning transmission electron microscope

1982 Binnig et Rohrer: scanning tunnelling microscope

1986 Ruska, Binnig et Rohrer: Prix Nobel Physics

1990 Rose: proposes the Cs corrector principle

1995 Haider: first realisation of the Cs corrector

2003 First commercial Cs corrected transmission electron microscope

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A bit of history: electron microscopy

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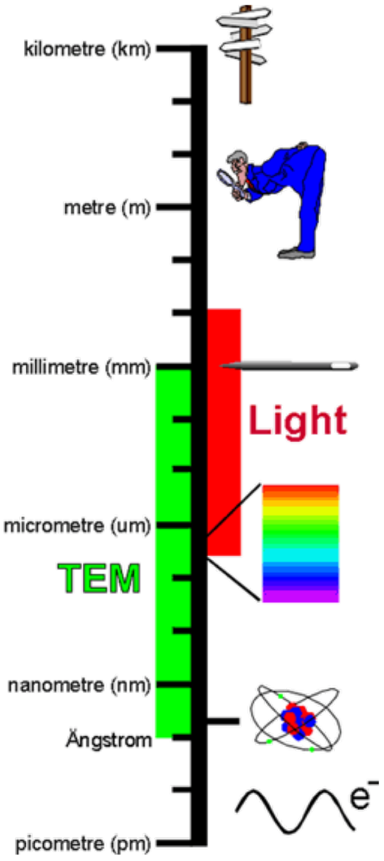
A bit of history: Why electrons?

- Smallest visible objects….
  - with eye : 0.1 mm = 10^{-4} m (size of one eye « stick »)
  - with light microscope : 10^{-7} m (magnification max ~ 2000 x)

Can we simply magnify the image of an object to observe every detail ?

- NO ! There are fundamental limitations linked to :
  - The type of probe (wave length, interaction, …)
  - The observation techniques
- With light microscopy : probe = visible light
  - One anlayses the modification of visible light chacteristics after its interaction with matter.



A bit of history: Why electrons?

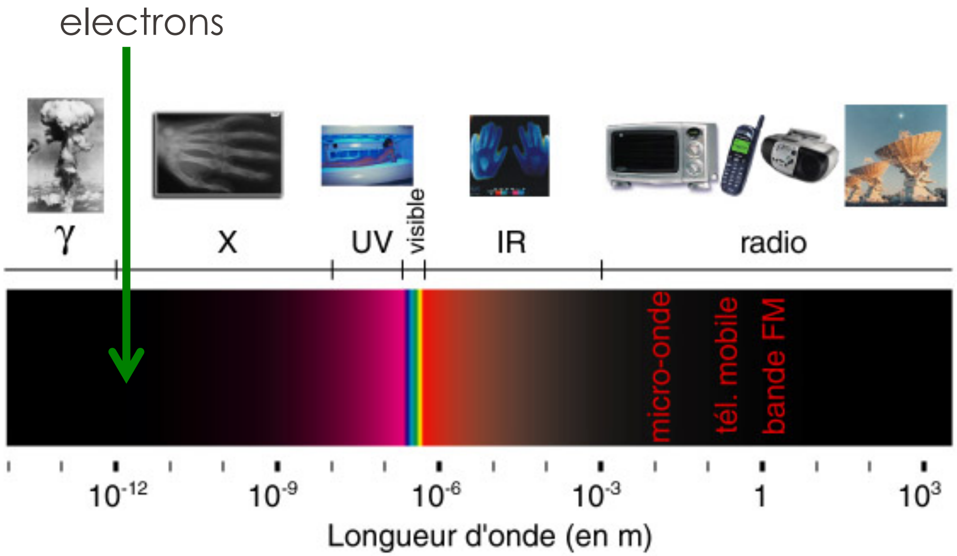
- Electromagnetic radiation: $E = hc/\lambda$ so if $\lambda < 5$ nm, $E > 1$ keV (RX)
  - Sources? Lenses?
- Electrons according wave length of de Broglie: $\lambda = h/p$ (with $p = m_o v = 2m_o eV$)

é non-relativistic:

$$\lambda = \frac{h}{(2m_0 eV)^{1/2}}$$

é relativistic (> 100 kV):

$$\lambda = \frac{h}{\left[2m_0 eV\left(1+\frac{eV}{2m_0c^2}\right)\right]^{1/2}}$$



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A bit of history: Why electrons?

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Electron charge (e)	-1.602×10^{-19} C
1 eV	1.602×10^{-19} J
Electron rest mass (m_o)	9.109×10^{-31} kg
Electron rest energy ($m_o c^2$)	511 keV
Kinetic energy (charge x tension)	1.602×10^{-19} Nm (pour tension 1 volt)
Plank's constant (h)	6.626×10^{-34} Nms
1 ampère	1 C/sec
Light speed in vacuum (c)	2.998×10^{8} m/sec

SEM: 10 kV

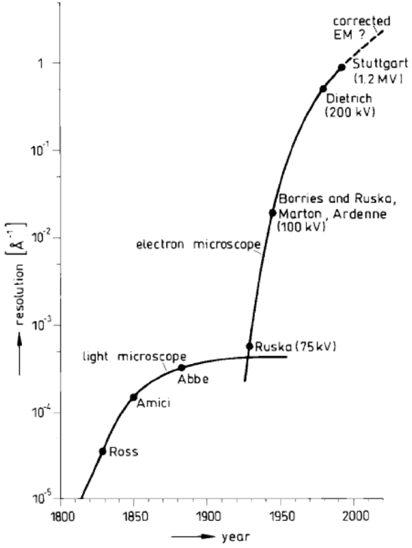
12.2 pm

Accelerating voltage [KV]	**Non-relativistic λ [nm]**	**Relativistic λ [nm]**	**Mass [x m_o]**	**Velocity [x 10^8 m/s]**
100	0.00396	0.00370	1.196	1.644
120	0.00352	0.00335	1.235	1.759
200	0.00273	0.00251	1.391	2.086
300	0.00223	0.00197	1.587	2.330
400	0.00193	0.00164	1.783	2.484
1000	0.00122	0.00087	2.957	2.823

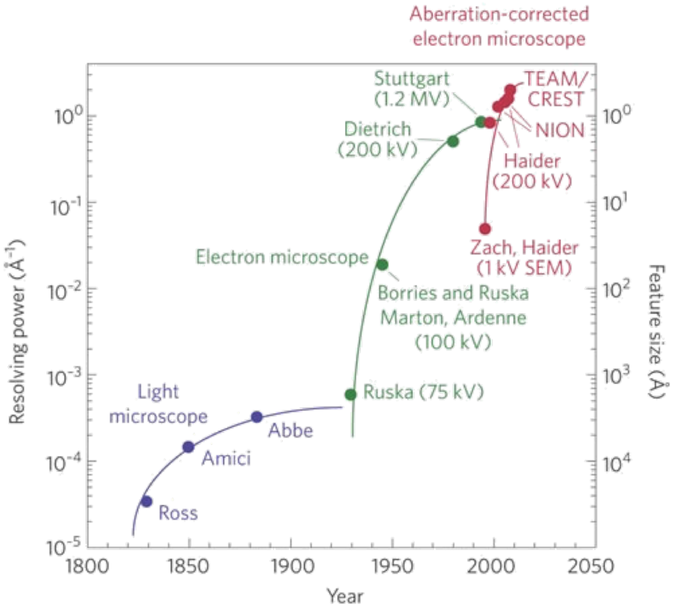
A bit of history: resolution

Aïcha Hessler-Wyser

Evolution of the spatial resolution

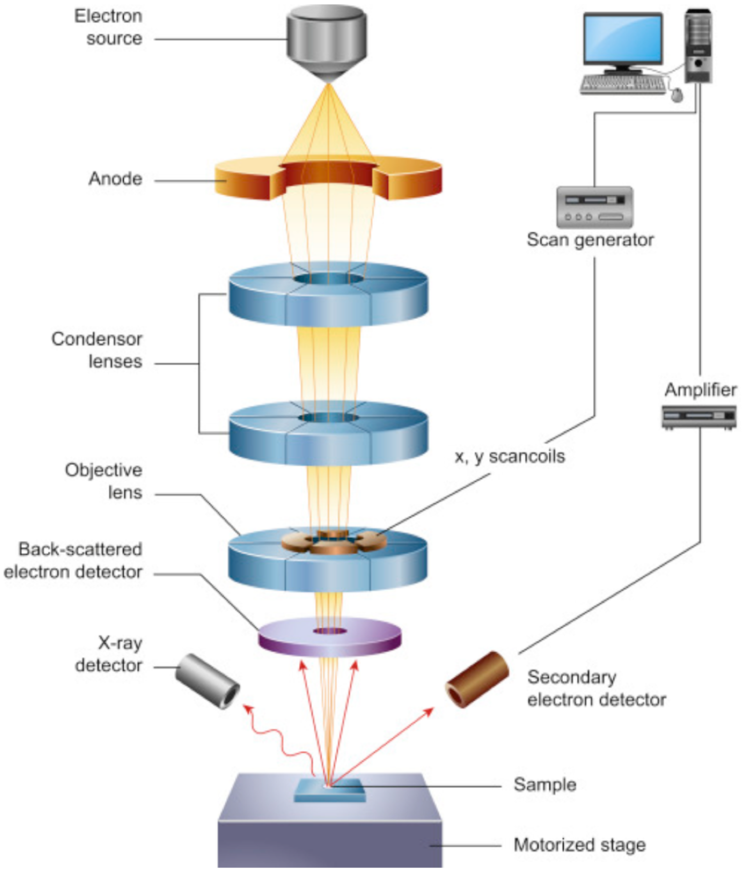


H. Rose, 1994



D. A. Muller, Nat. Mat. 8, 263-270 (2009)

Scanning Electron Microscopy: principle



- Electrons are generated in the gun, accelerated, then collimated in a thin probe
- Probe is deflected to scan the sample surface
- Incident electrons interact with the sample and generate different signals
- Signals are collected by different detectors and recorded for each probe position
- Image is reconstructed

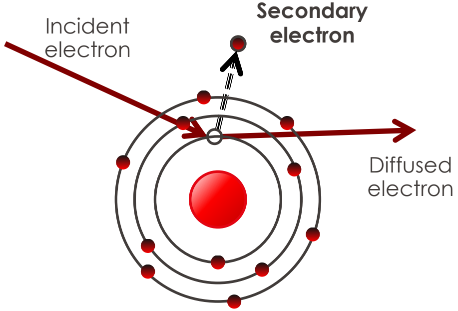
ttps://www.liberaldictionary.com/scanning-electron-microscope/

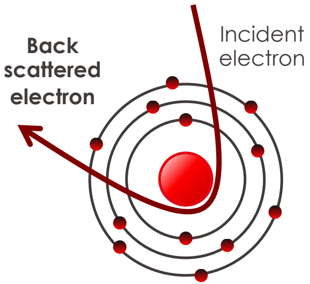
Scanning Electron Microscopy: principle

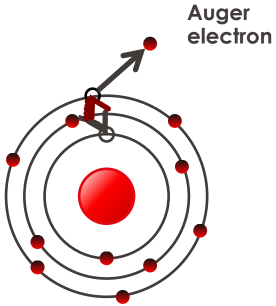
Electron incident onto a bulk material

Electron radiation

- **Secondary electrons** (SE): electrons ejected from material at low energy (5 to 50 eV).
- **Back scattered electrons** (BSE): incident electrons that are successively diffused and leave the sample: they have high energy, close to initial energy E_0.
- **Auger Electrons**: electrons ejected from the surface with an energy characteristic from the target atoms.







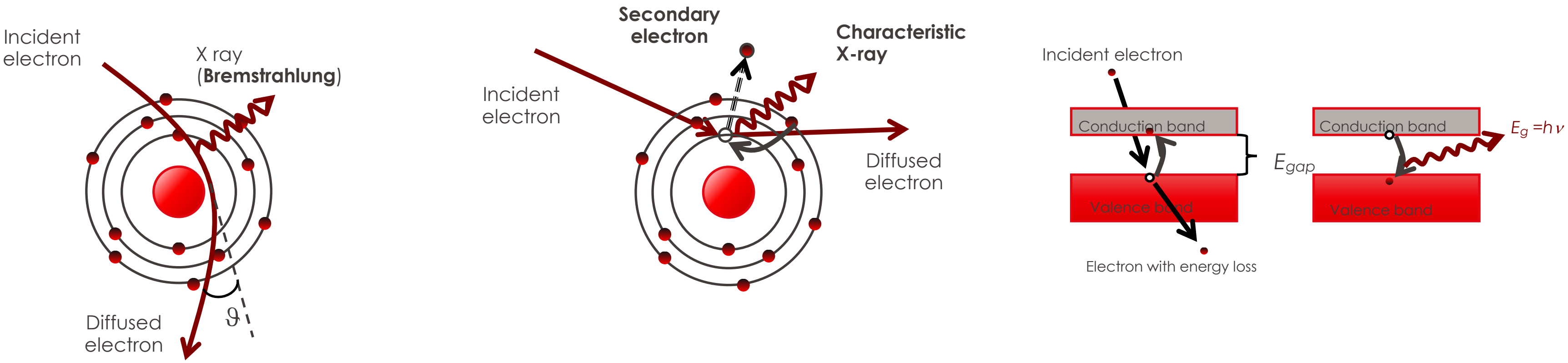
Scanning Electron Microscopy: principle

Aïcha Hessler-Wyser

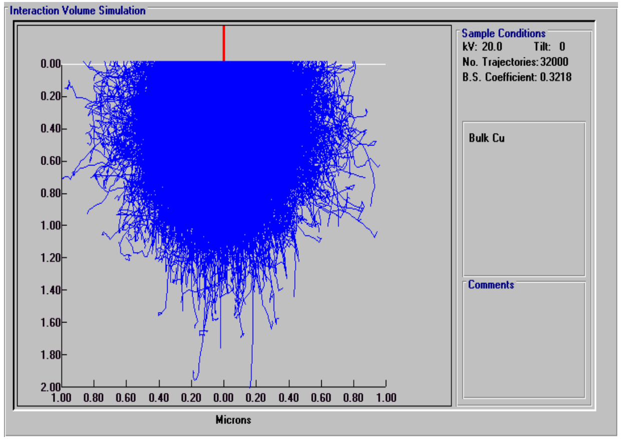
Electron incident onto a bulk material

Electromagnetic radiation

- X rays with continuous energy, resulting from desceleration of incident electrons ("**Bremstrahlung**" or "slowing down radiation").
- **X rays with energy characteristic** from the target atoms.
- **Cathodoluminescence**: Visible radiation mainly emitted by isolating or semi-conducting materials.



Scanning Electron Microscopy: principle

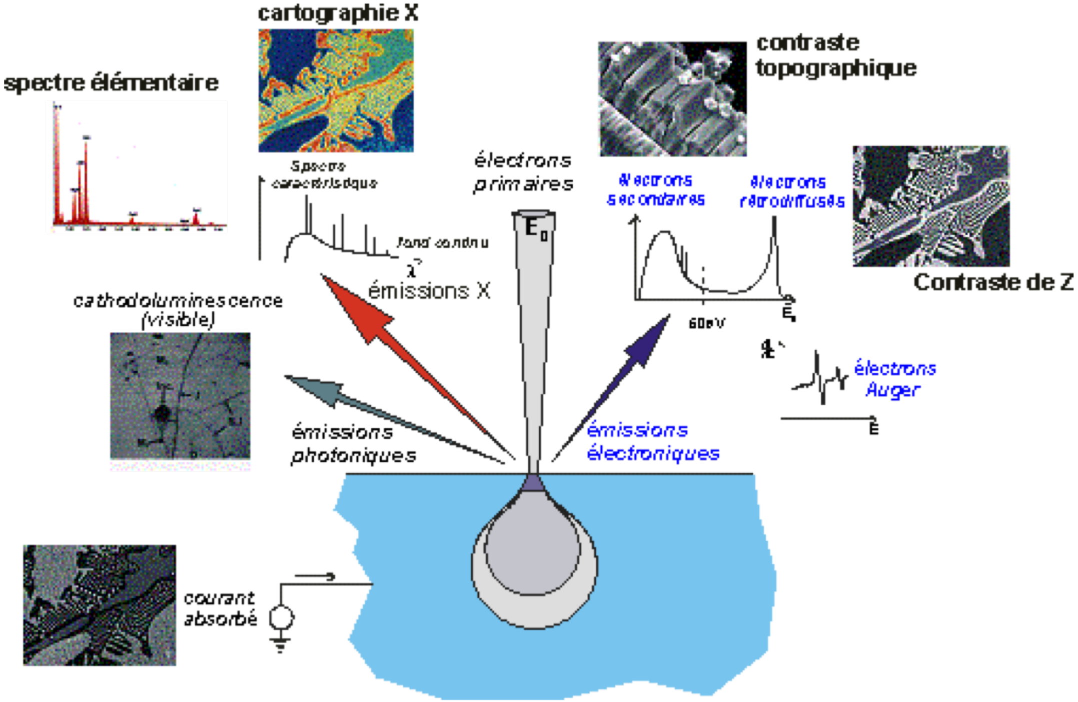


Monte-Carlo simulation in Cu with incident electrons of 20 keV

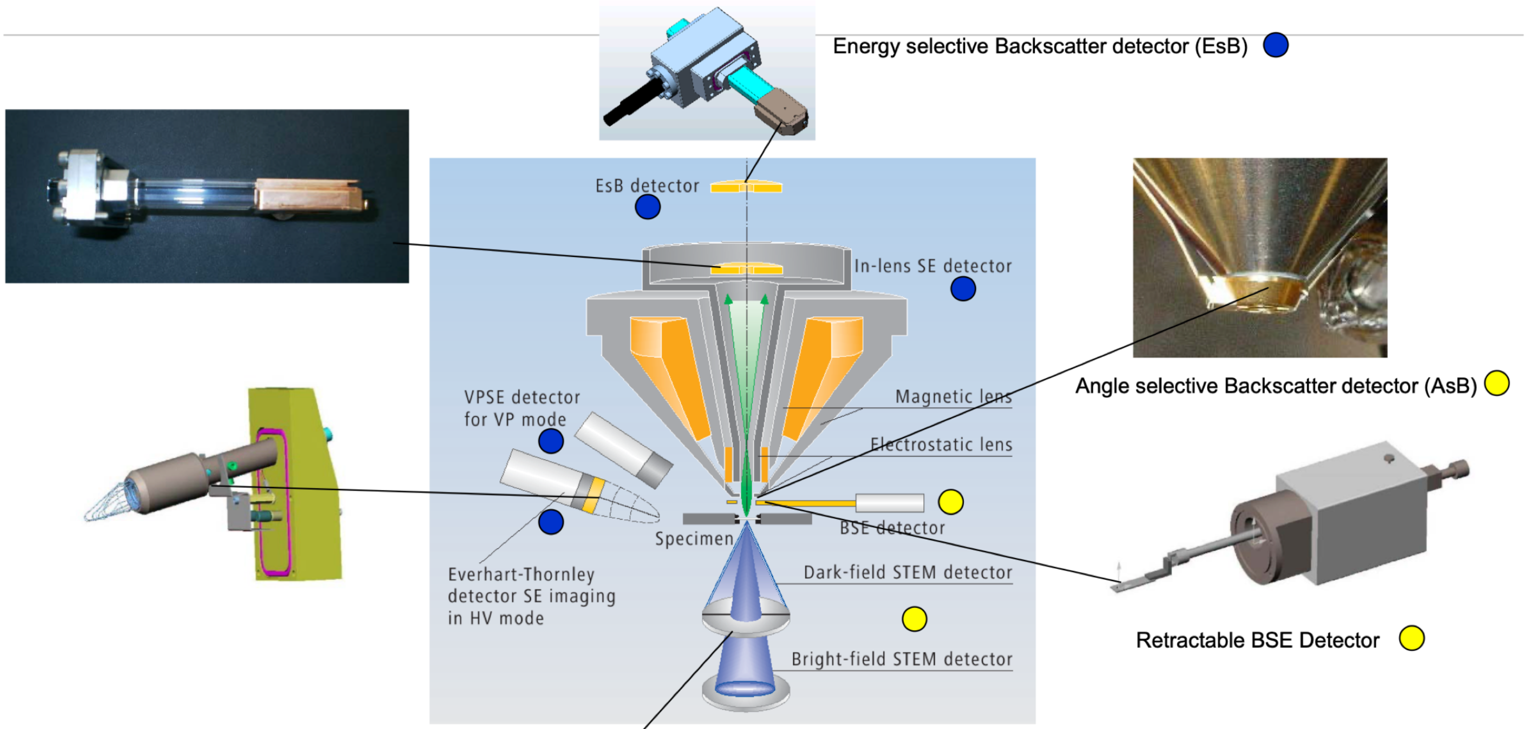
Interaction volume

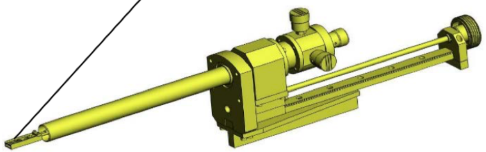
or

Interaction pear



SEM: detectors





ET detector: a 200-300 V bias is applied to attract low energy SE

- **Scintillator Detector** (blue)
- **Semiconductor Detector** (yellow)

SEM: resolution

- SEM: Limiting parameters on resolving power ρ with SE

1. *At high magnification*

   The **probe size** (generation of SE1)

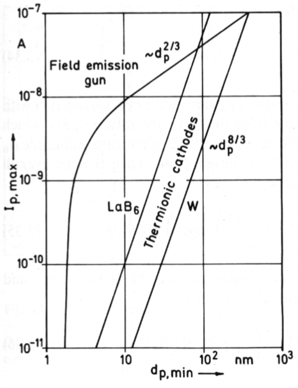
2. The **volume of interaction** (generation of SE2+SE3 from BSE): energy and atomic number influence

3. *At low magnification*

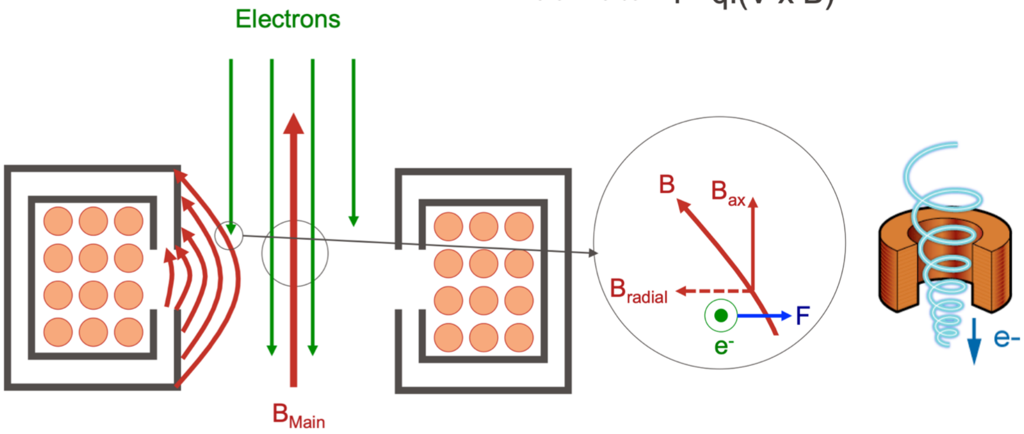
   The screen (or recording media) **pixel size** d_{screen}

SEM: resolution

- Reduce the probe size by
  - Reducing the probe current (gun, aperture, condenser lens, …)
  - Increasing the acceleration voltage
  - Reduce aberrations (Cs, astigmatism, chromatic)

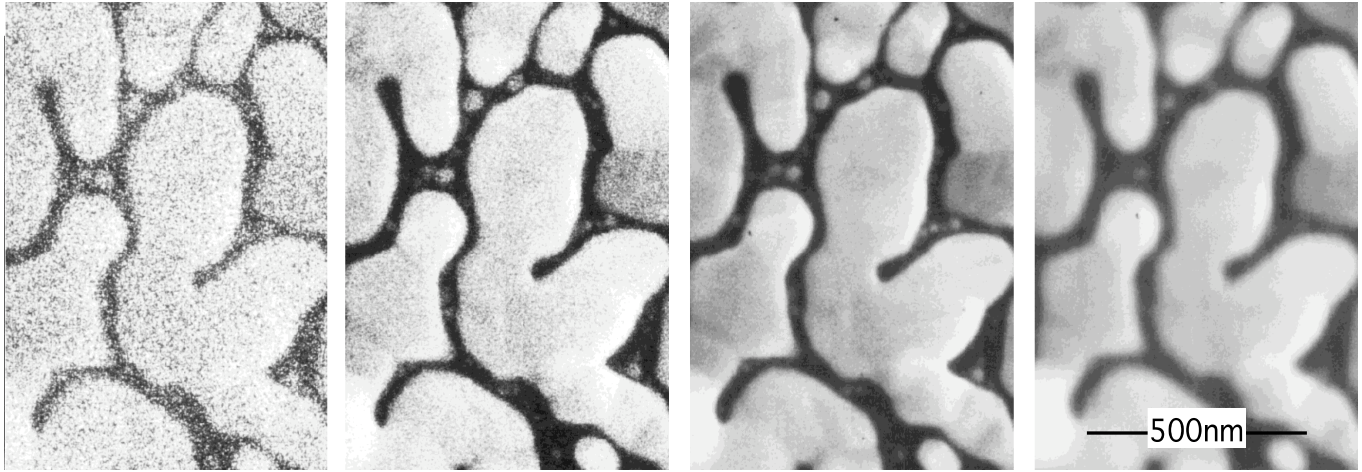


Lorenz force: $\vec{F}=q.(\vec{V} \times \vec{B})$



SEM: resolution

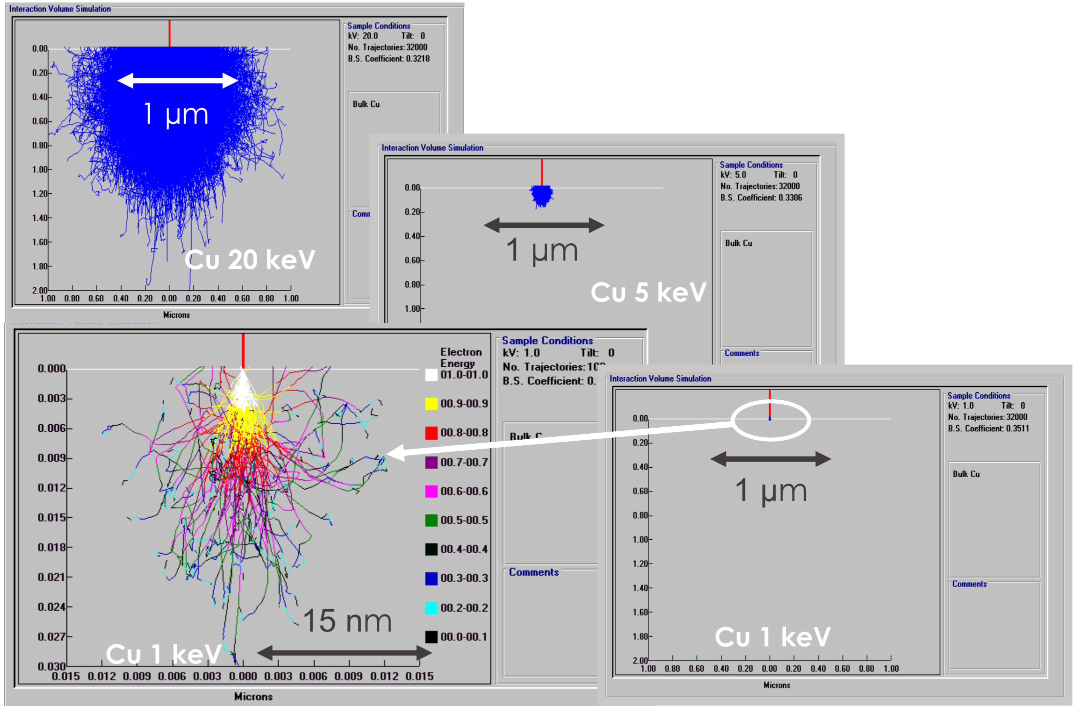
- Effect of current, probe diameter and image acquisition time

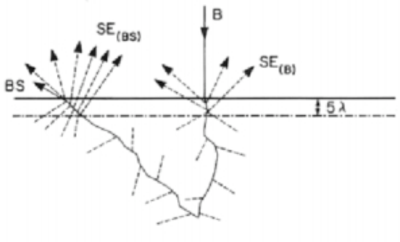


10 pA/10 s	10 pA/160 s	100 pA/160 s	1 nA/160 s
good resolution, but statistical noise	Good resolution, less statistical noise	smal loss of resolution, still less statistical noise	very few statistical noise, but high resolution loss!

SEM: resolution

- Reduce the interaction volume by… reducing voltage!



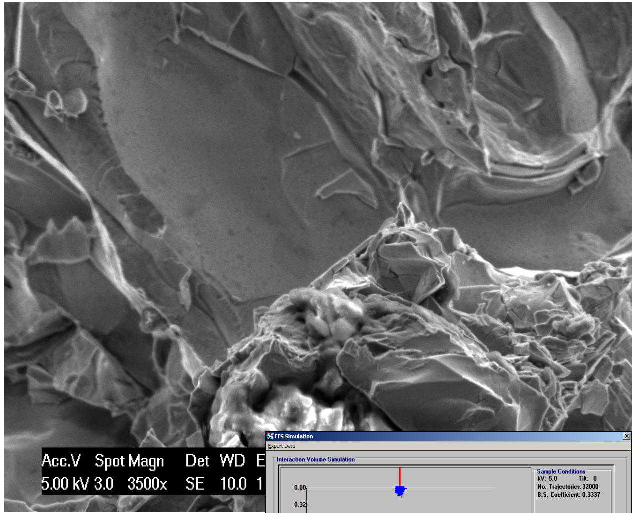


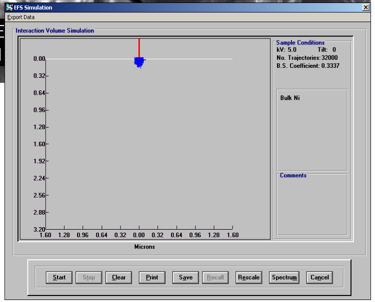
Delocalisation of the information!

Voltage compromise!

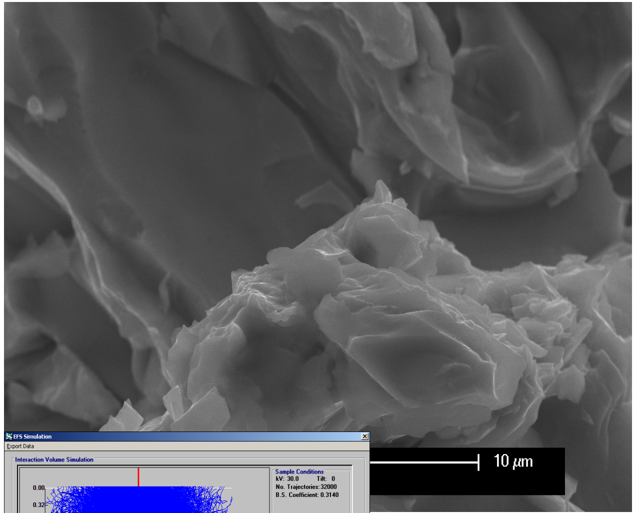
SEM: resolution

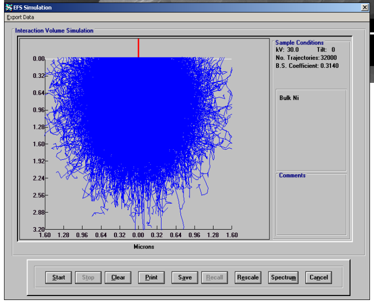
- Contrast change in SE with acceleration voltage





SE, 5 kV





SE, 30 kV

An example: a fracture in Ni-Cr alloy

SEM: resolution

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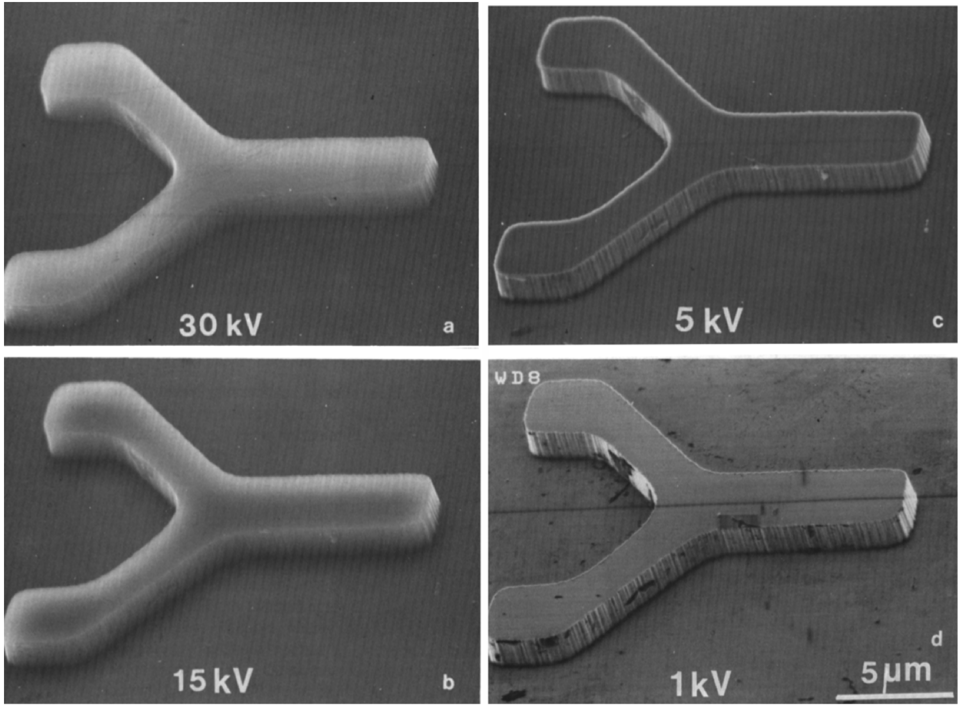
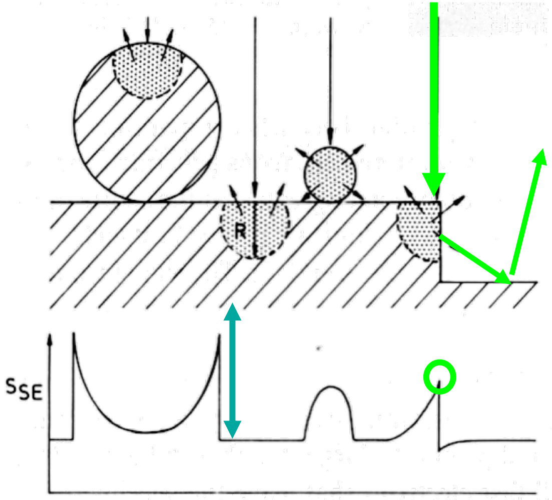
- Contrast change in SE with acceleration voltage



Edge effect!



Intensity profile on image

(from L.Reimer, Image formation in the low-voltage SEM)

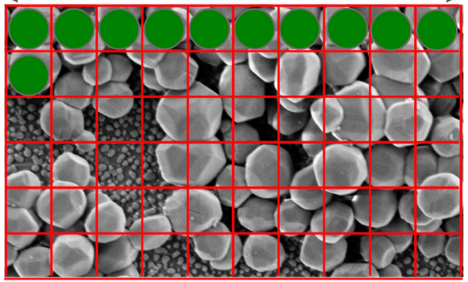
SEM: resolution

Aïcha Hessler-Wyser

- Control the pixel size

Sample

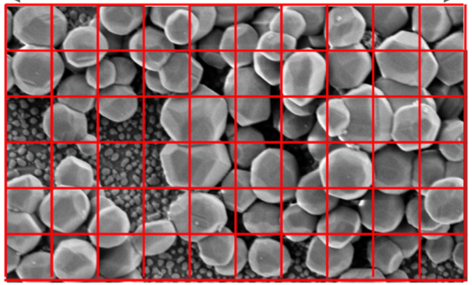
100 nm



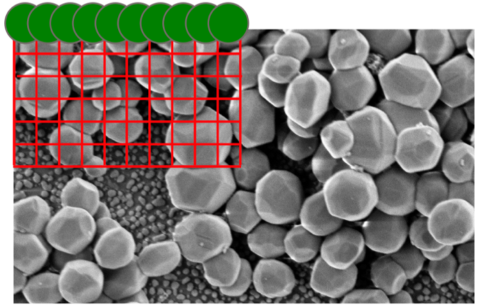
Scan step (i.e. pixel size) on the sample?

Monitor

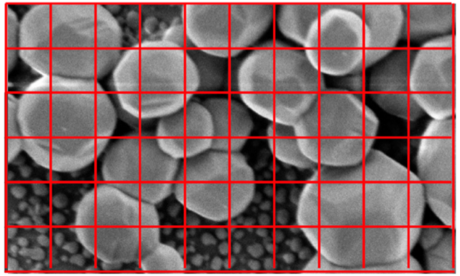
10 cm



Pixel size on the screen?



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



Pixel size on the screen?

SEM: topographical contrast

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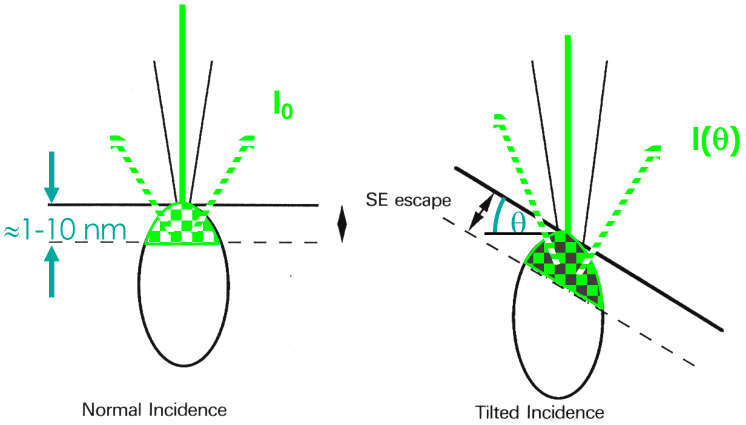
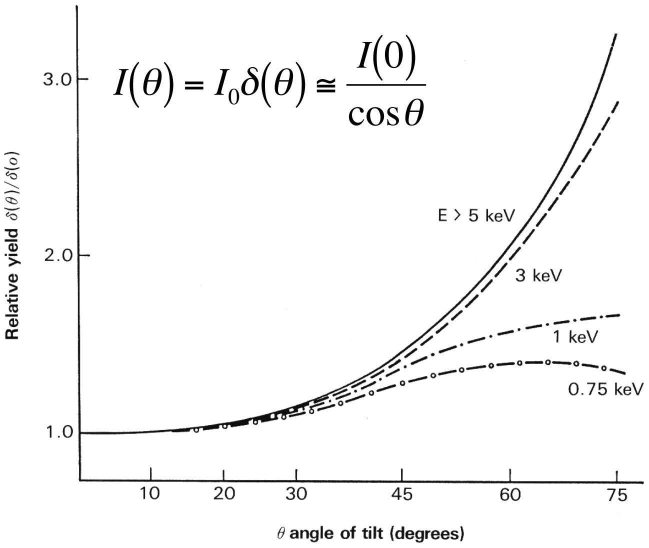
- In SE mode: more SE can escape from the surface when it is tilted…



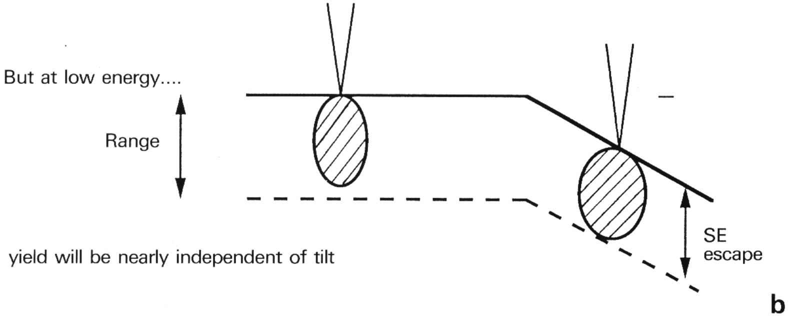


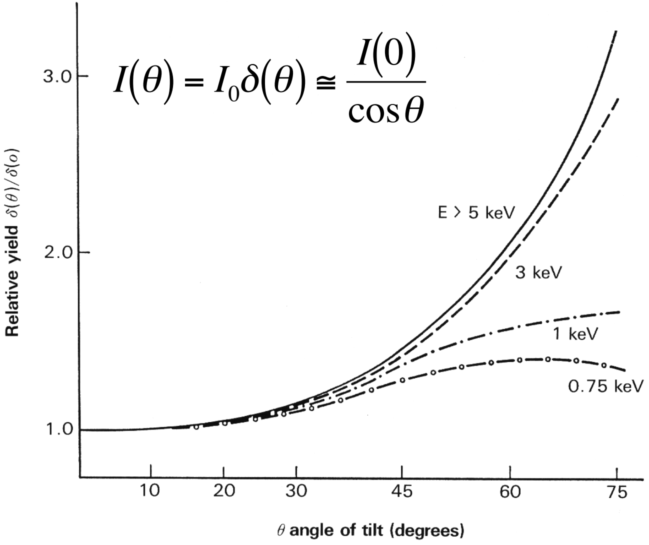
Relative yield of SE vs angle of incidence on the sample surface

(adapted from D.C. Joy Hitachi News 16 1989)

SEM: topographical contrast

- In SE mode: more SE can escape from the surface when it is tilted… but not at low energy!





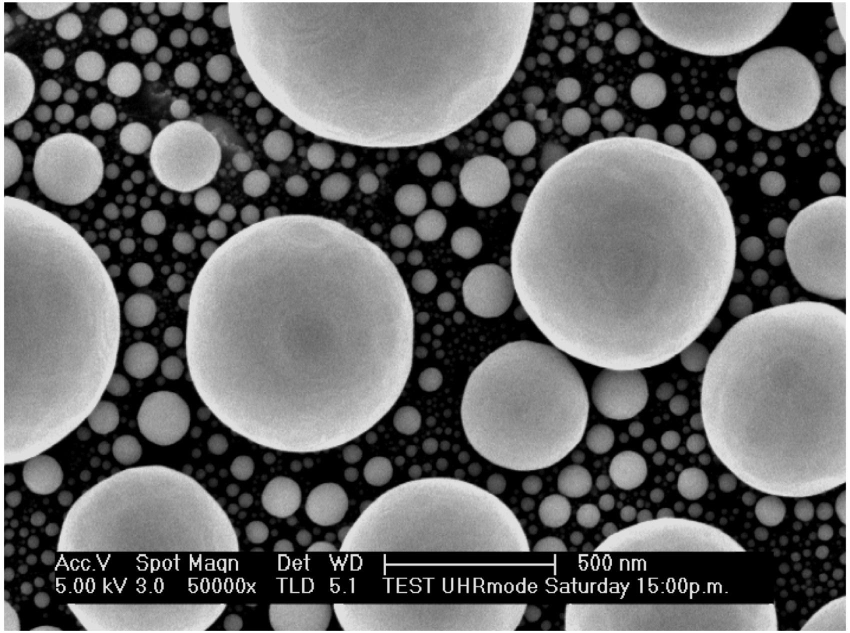
Relative yield of SE vs angle of incidence on the sample surface

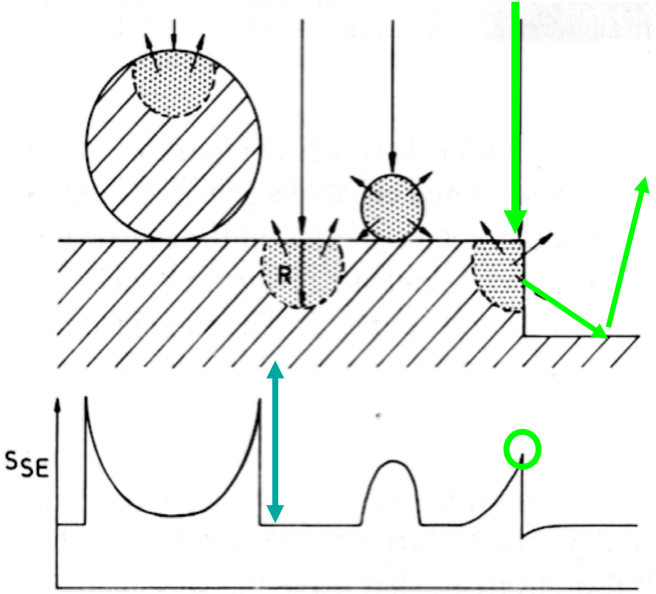
(adapted from D.C. Joy Hitachi News 16 1989)

SEM: topographical contrast

Aïcha Hessler-Wyser

- Size and edge effects

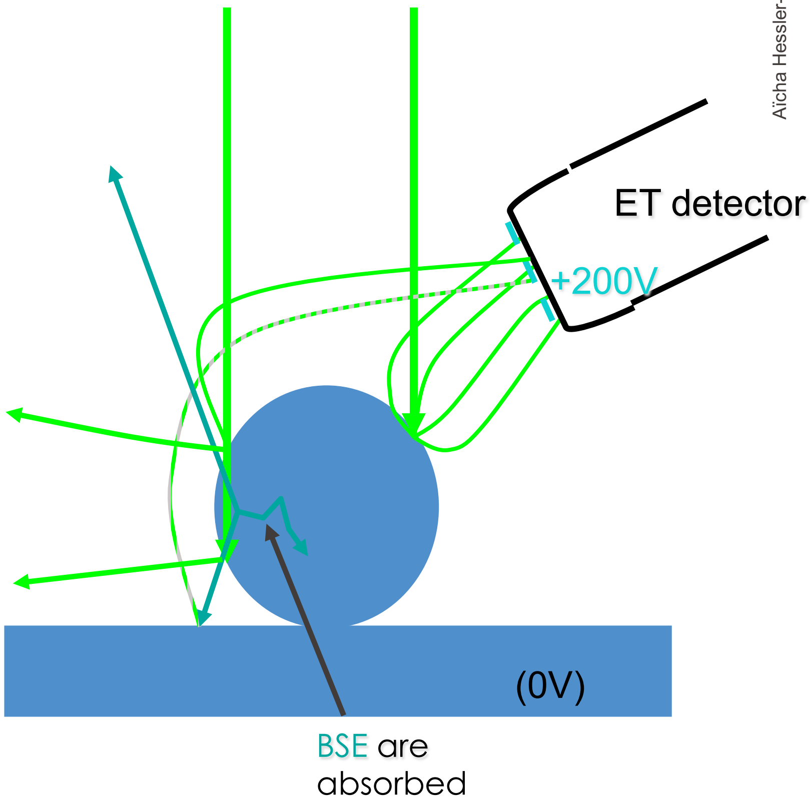




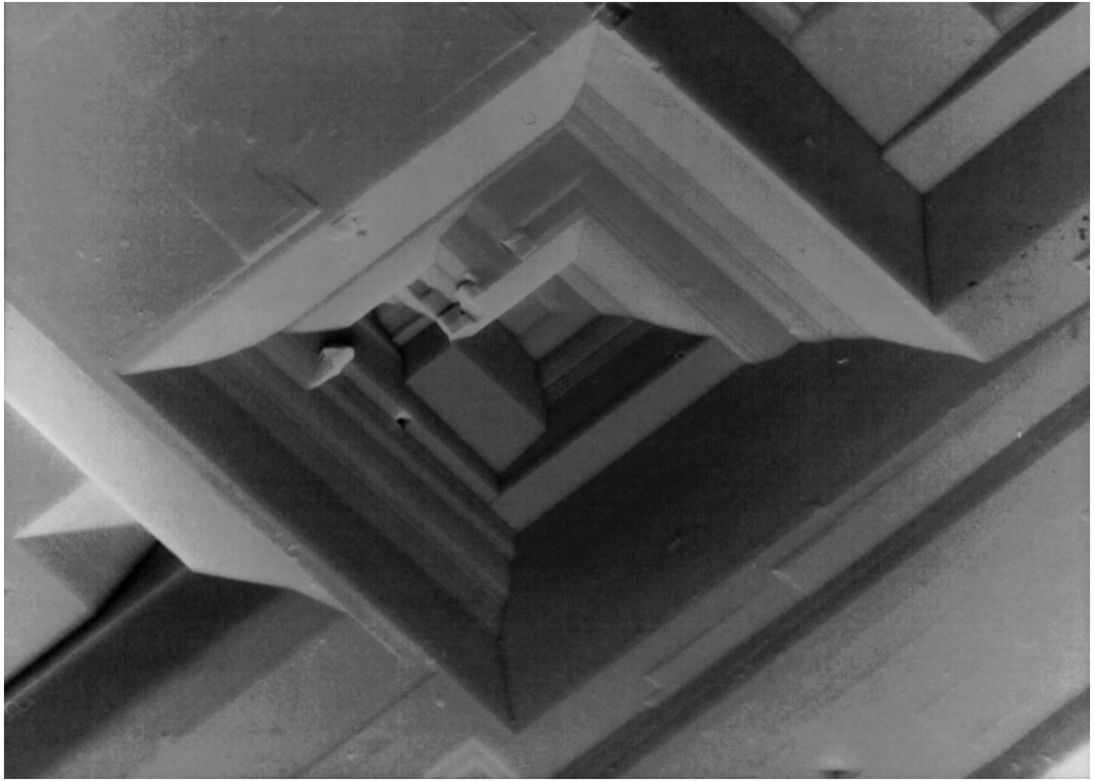
Intensity profile

SEM: topographical contrast

- Shadow effect
  - SE detector detects more SE from the facing surface than from the opposite one
  - You need to know where your detector is placed in your chamber
  - No shadow effect with in-lens detectors



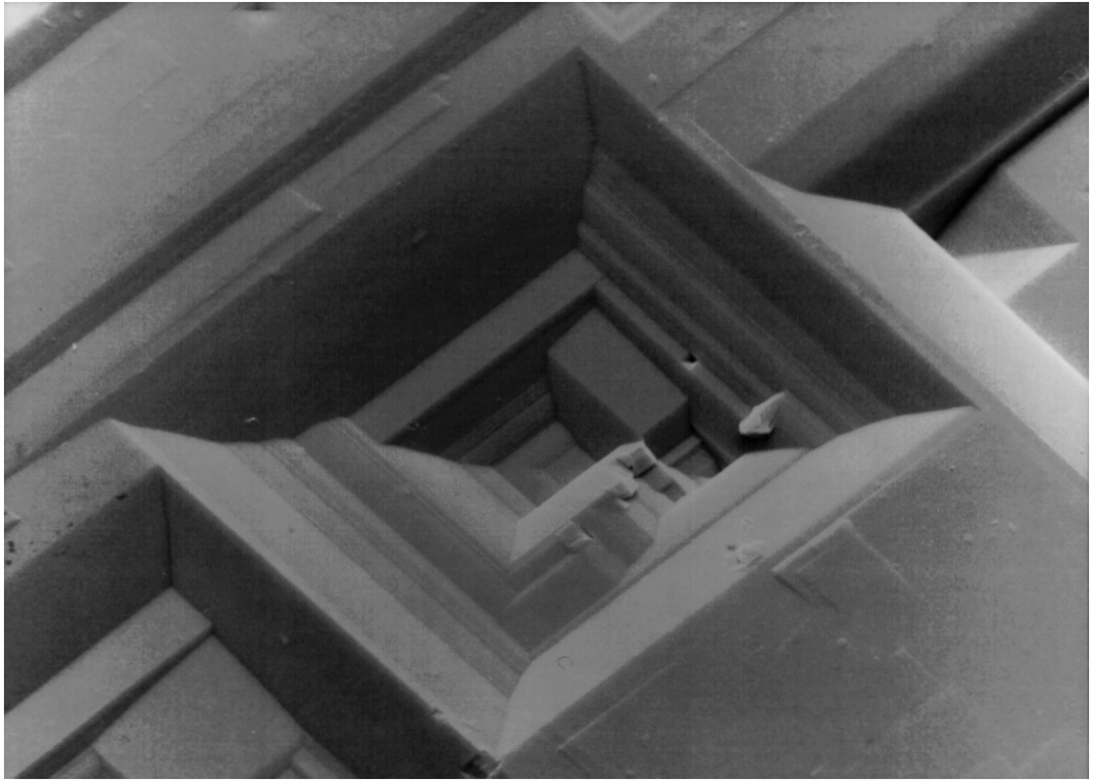
SEM: topographical contrast



What does it suggest?

Which objective information?

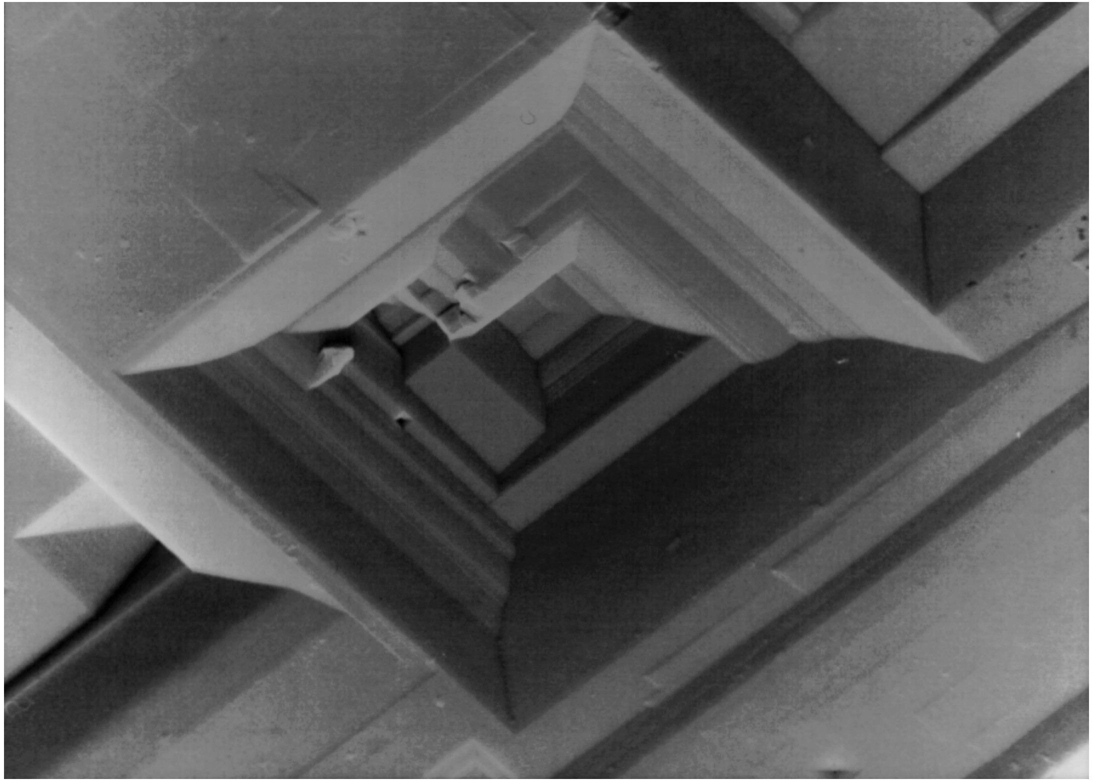
SEM: topographical contrast



What does it suggest?

Which objective information?

SEM: topographical contrast



What does it suggest?

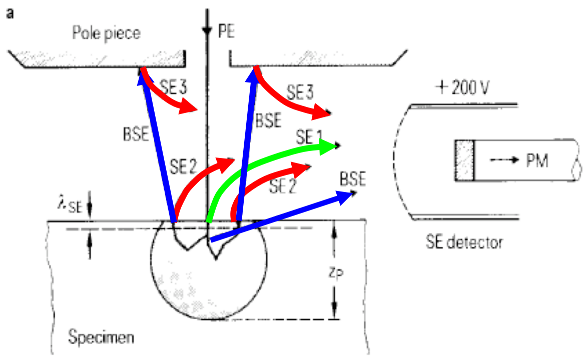
Which objective information?

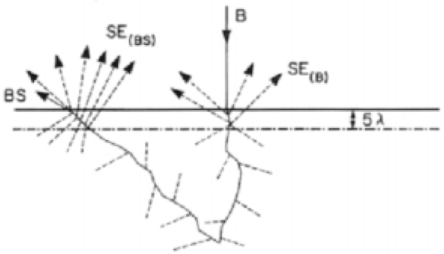
Need to know where the ET detector is placed!

SEM: topographical contrast

- "True" secondary electrons ***SE1*** and "converted BSE" secondaries ***SE2+SE3***

Various SE types from
- SE1: incident probe
- SE2: BSE leaving the sample
- SE3: BSE hitting the surroundings





Although this signal is gathered around the probe, its intensity is only attributed to the pixel corresponding to the actual probe position

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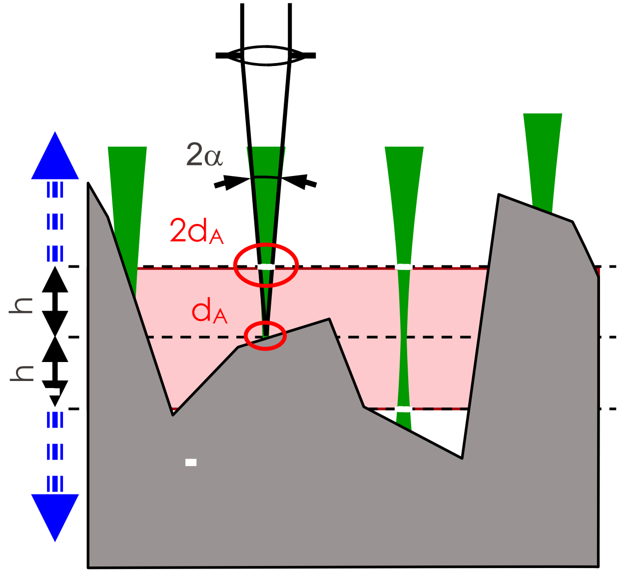
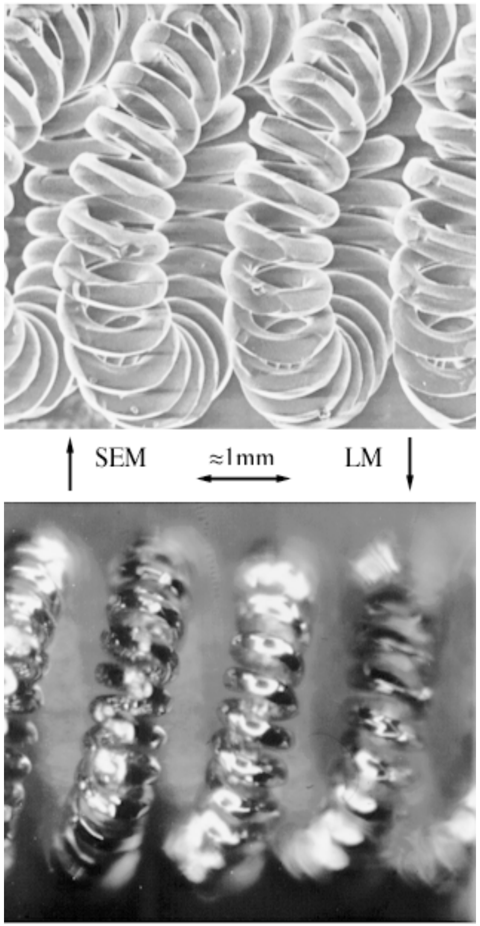
SEM: depth of field

- High depth of field at low magnification
  - An image is considered in focus if the probe size is smaller than $2d_A$, with d_A the minimum probe size





Light bulb filament

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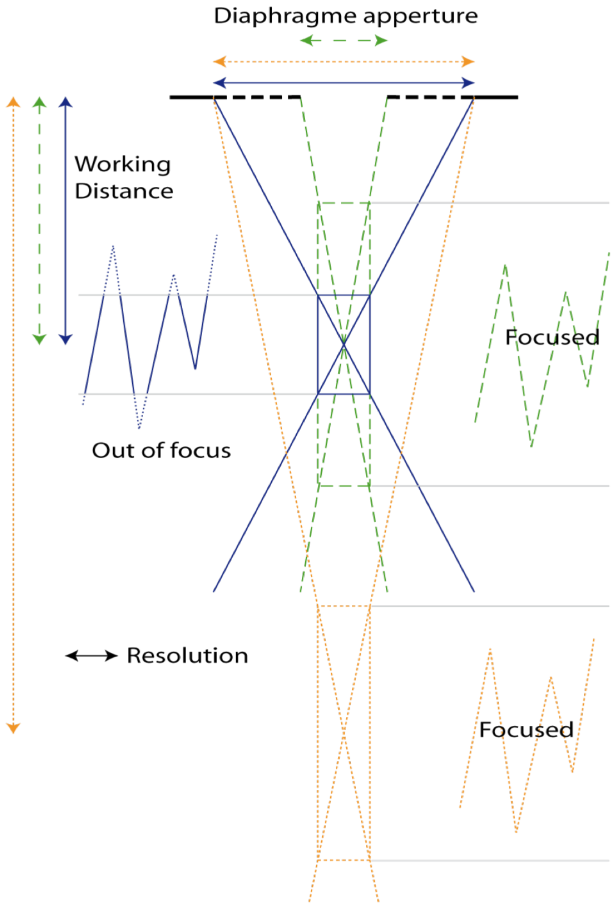
SEM: depth of field

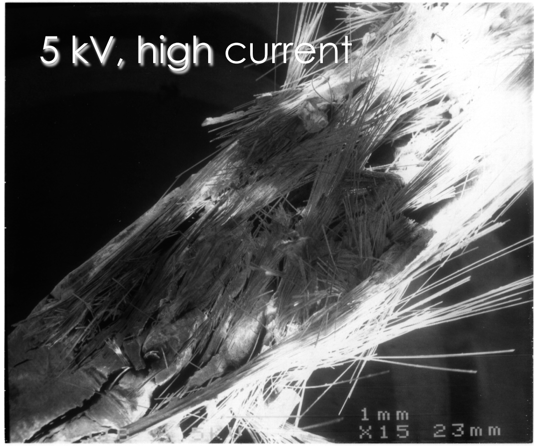
- Need to reduce the convergence angle of the beam
- Effect of working distance (WD) and aperture on depth of field
- Working distance is the distance between the exit of the objective lens and its focus point
- Aperture reduces the exit from the objective lens, thus reduces the convergence angle

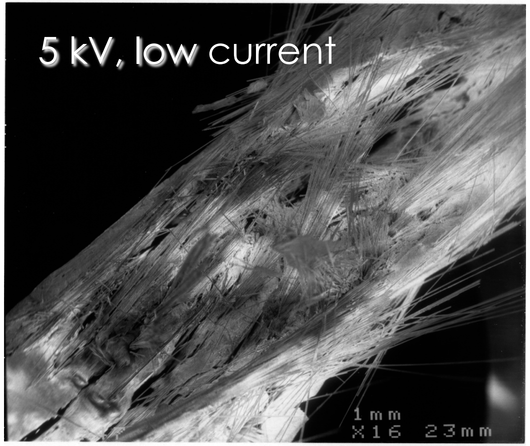


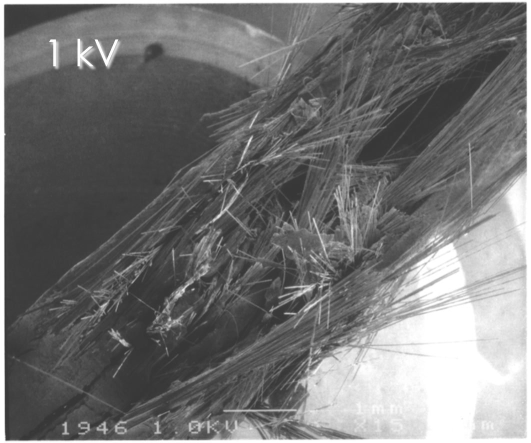
SEM: charging effect

- Fiberglass on epoxy
  - Electrons arrive onto the sample
  - BSE and SE escape from the sample
  - A charge balance is needed
  - In case of unbalance, contrast are altered

(Courtesy of B. Senior CIME/EPFL)

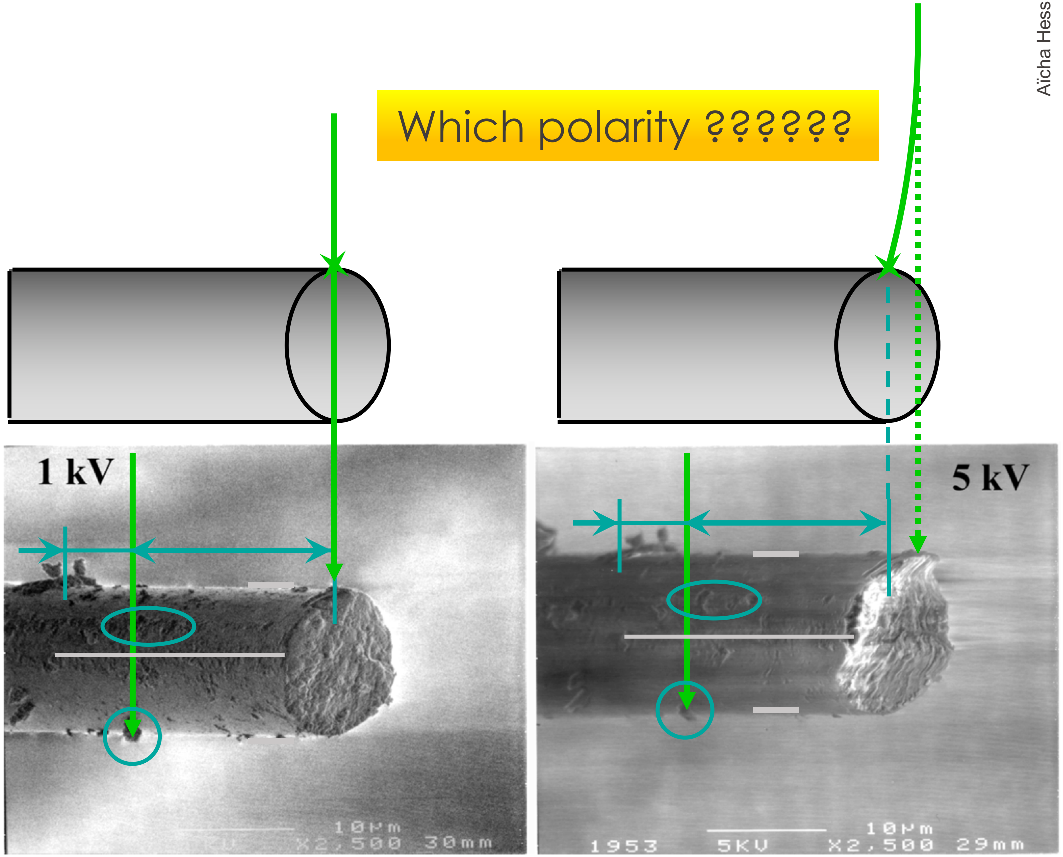






SEM: charging effect

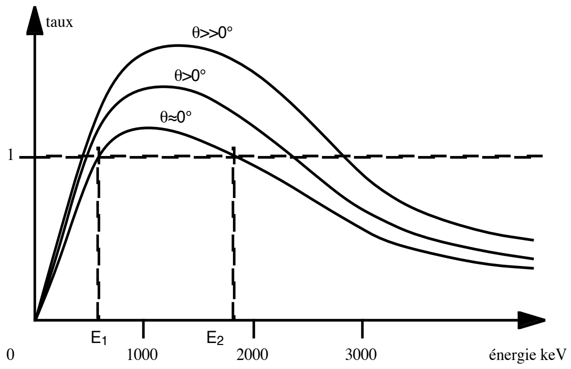
- SE contrast improves at low energy
- When charging occurs, the distances are modified
- Quantification is not possible anymore



Courtesy B. Senior/CIME

SEM: charging effect

- Total yield for electron emission (SE + BSE) on insulators
  - E_1 and E_2 are critical energies where 1 electron leaves the surface for each incident electron: neutrality
  - when $eV_{acc}= E_2$ charging-up disappears!
  - $eV_{acc}= E_1$ is unstable,
    $eV_{acc}= E_2$ is stable
  - Caution: E_1 and E_2 are specific to the material, but also change with the incidence angle θ!



Caution: this simple (simplist!) model is not quantitative for insulators because charge implantation and removal depends of the scanning speed and precise sample geometry

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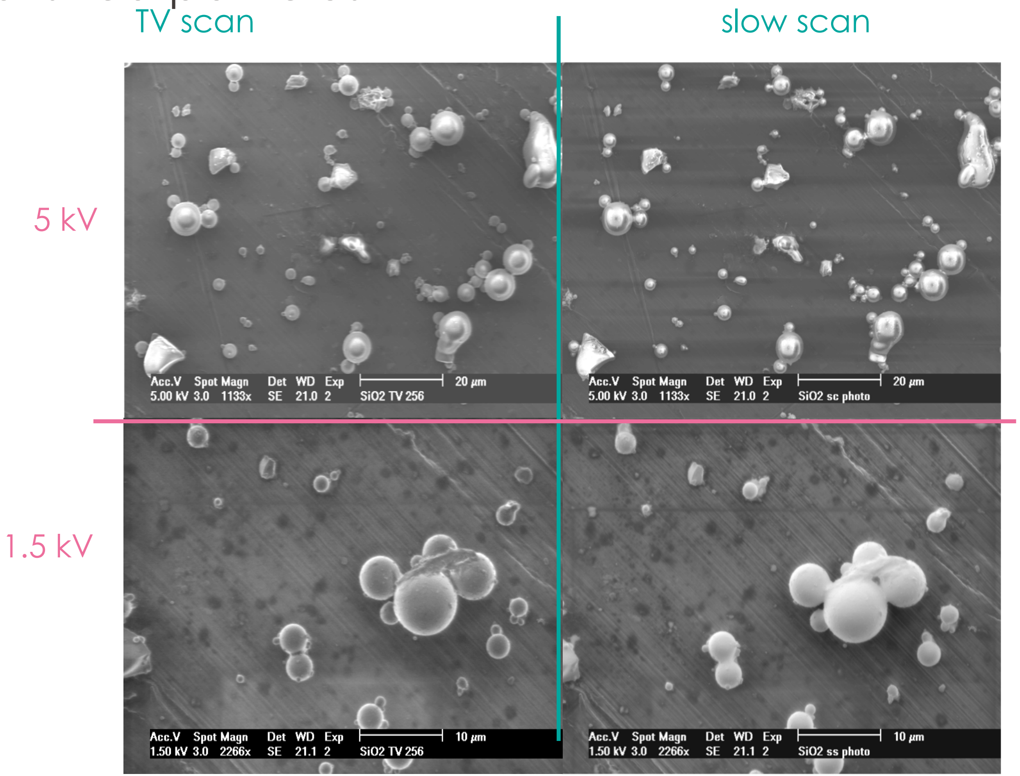
SEM: charging effect

- Charging-up on spherical silica particles

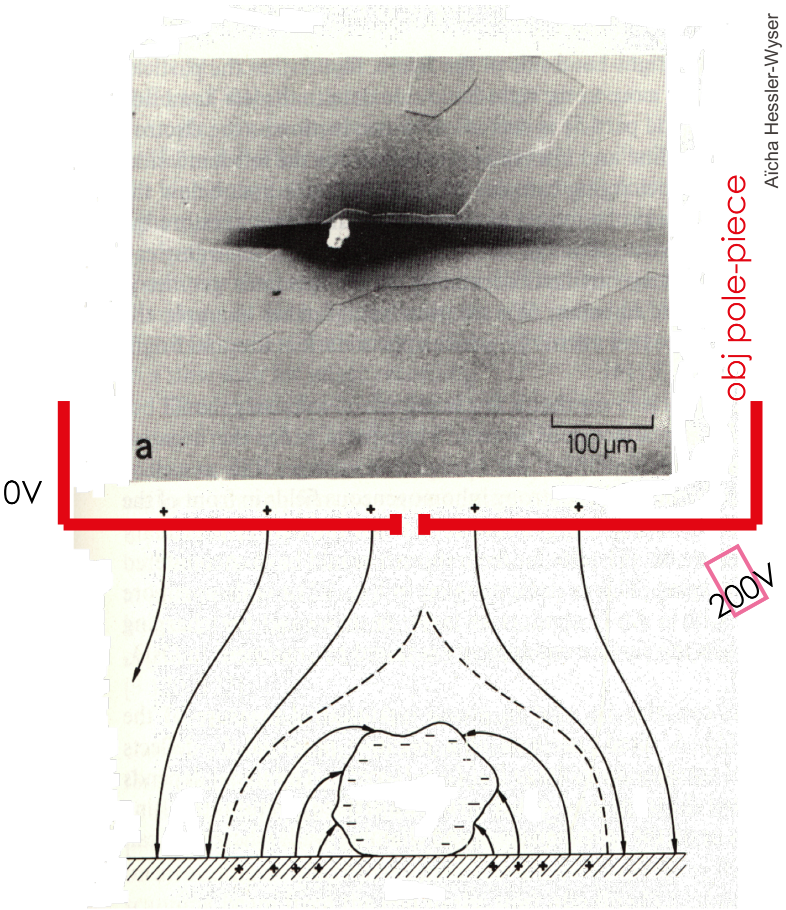
charges at the particle surface lead to anomalous contrast as a flying saucer

at 1.5 kV, close to the neutrality point, particles recover their sphere contrast



SEM: charging effect

- Charging-up of an insulating particle of dust
  - Negative charges left on the particle create an electric field that repells the SE toward the substrate around the dust
  - Those SE don't reach the ET detector => no contrast



(adapted from L. Reimer Scanning Electron Microscopy)

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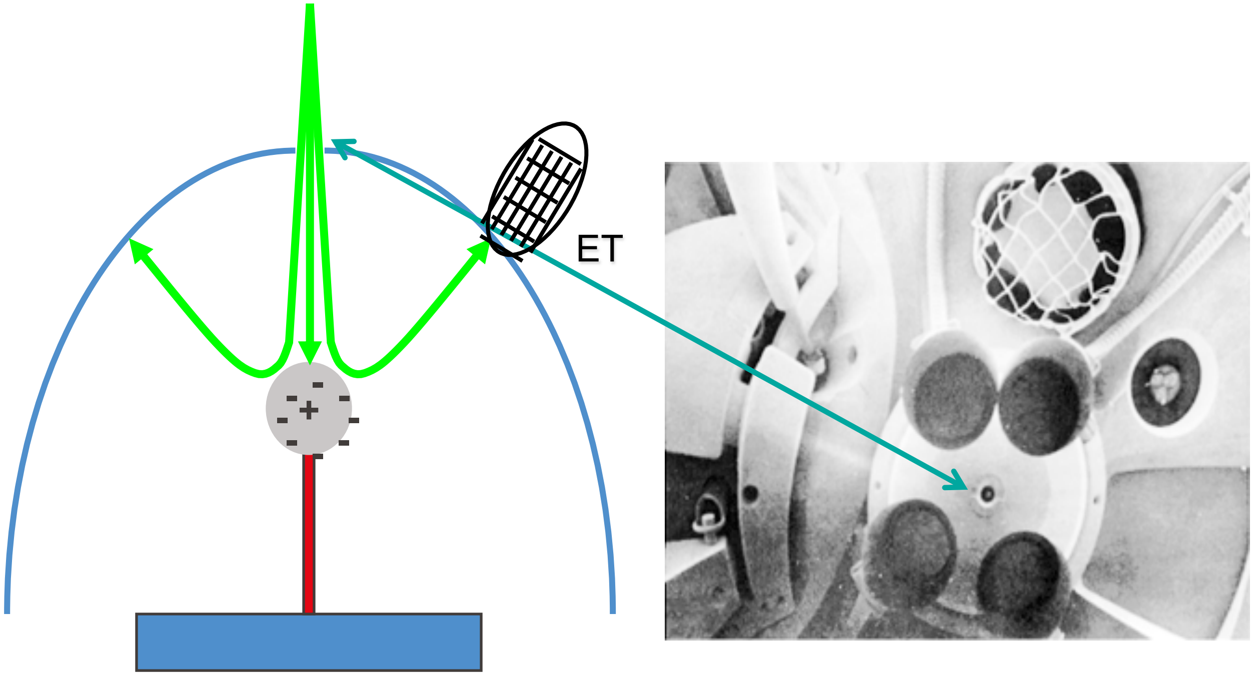
SEM: charging effect

- Extreme charging-up: electrons are reflected by the sample and hit the microscope chamber instead of the sample!!!



(adapted from Philips Bulletin)

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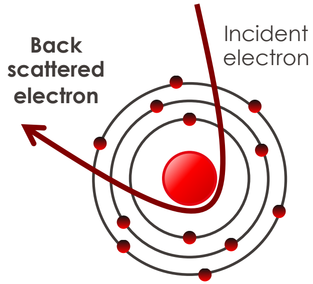
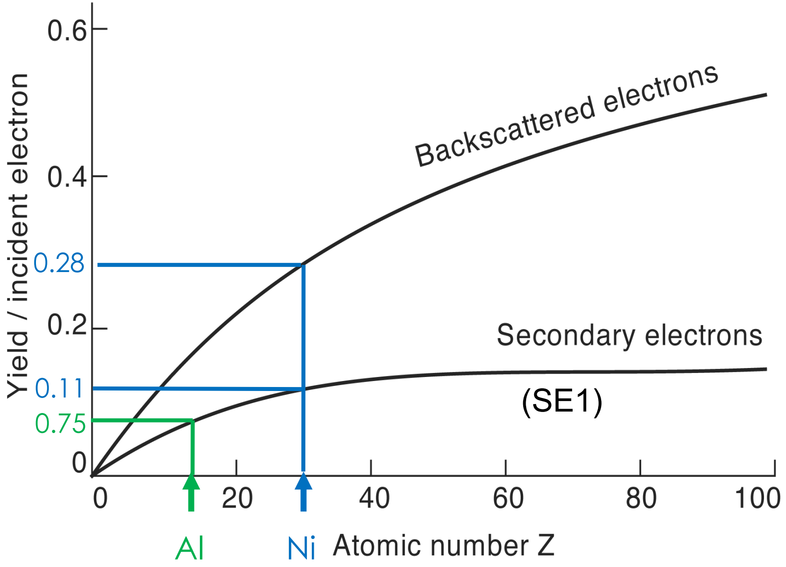
SEM: SE vs BSE

- Yield for SE and BSE emission per incident electron vs atomic number Z
- sample surface ***polished*** (no topography) and perpendicular to the incident beam direction (intermediate energy $E_0 \approx 15$ keV)
- BSE: "chemical" contrast for all elements
- SE: low or no "chemical" contrast but for light elements





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SEM: Z contrast

- Contrast changes with incident energy

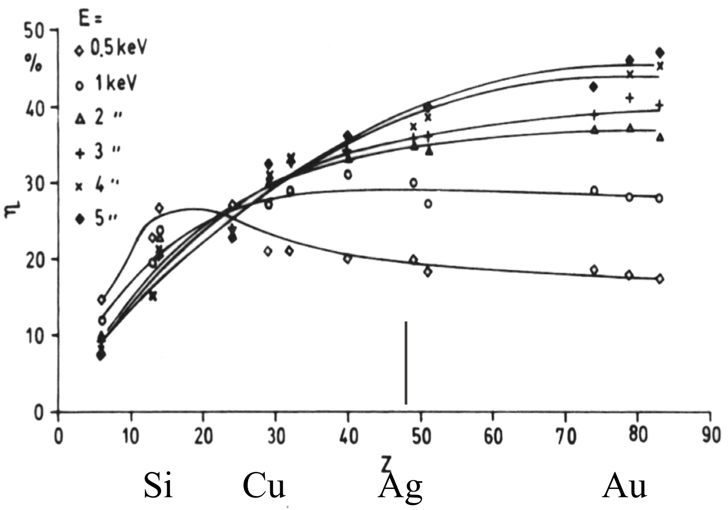
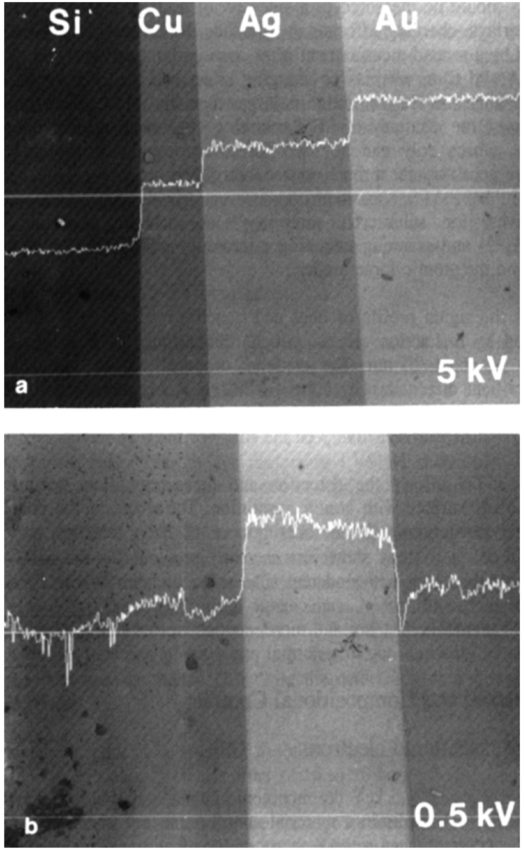
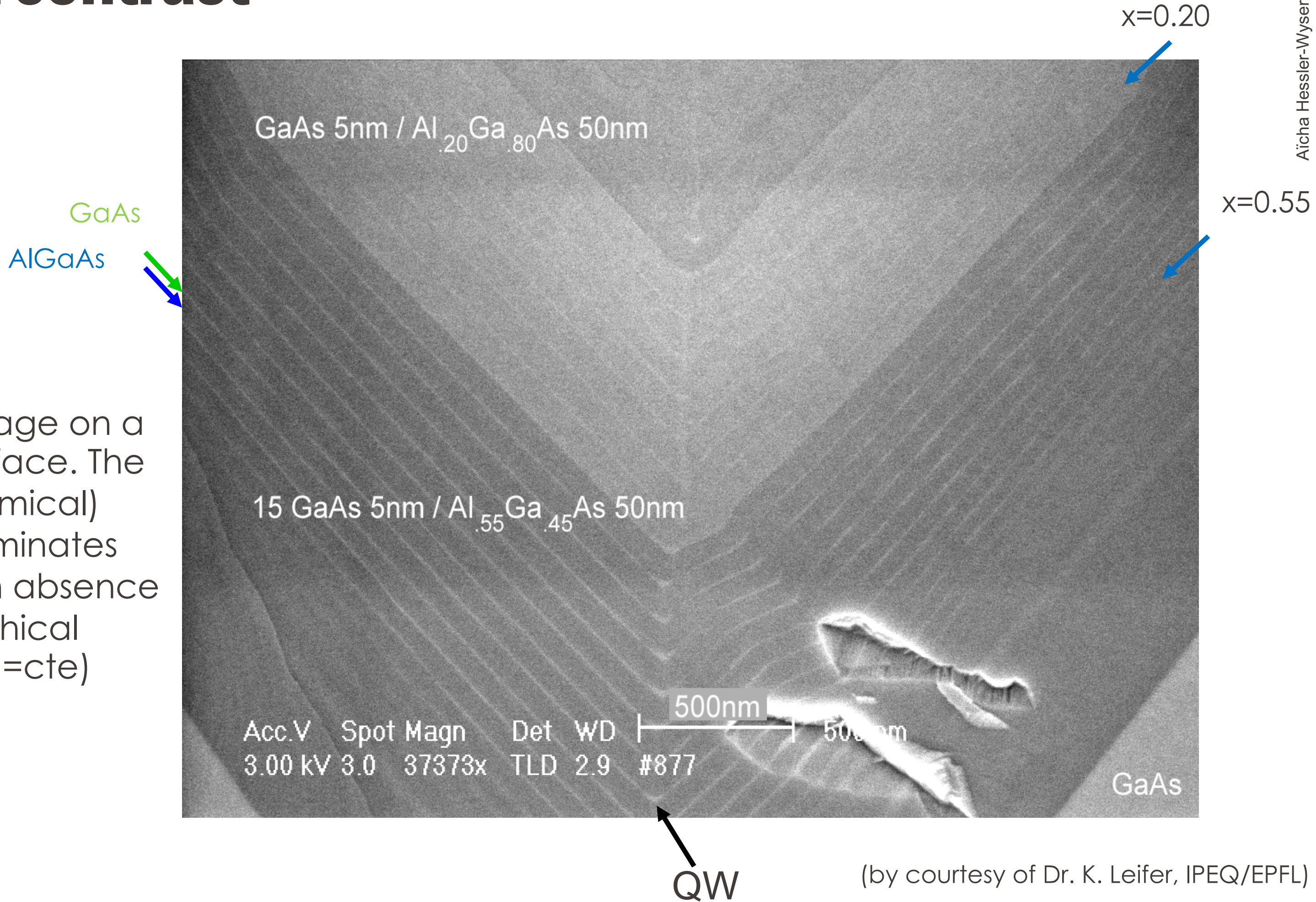


Fig. 4.1. Dependence of the backscattering coefficient η at normal incidence (ϕ=0) on atomic number Z for different electron energies.



from L.Reimer, Image formation in low-voltage SEM

SEM: Z contrast

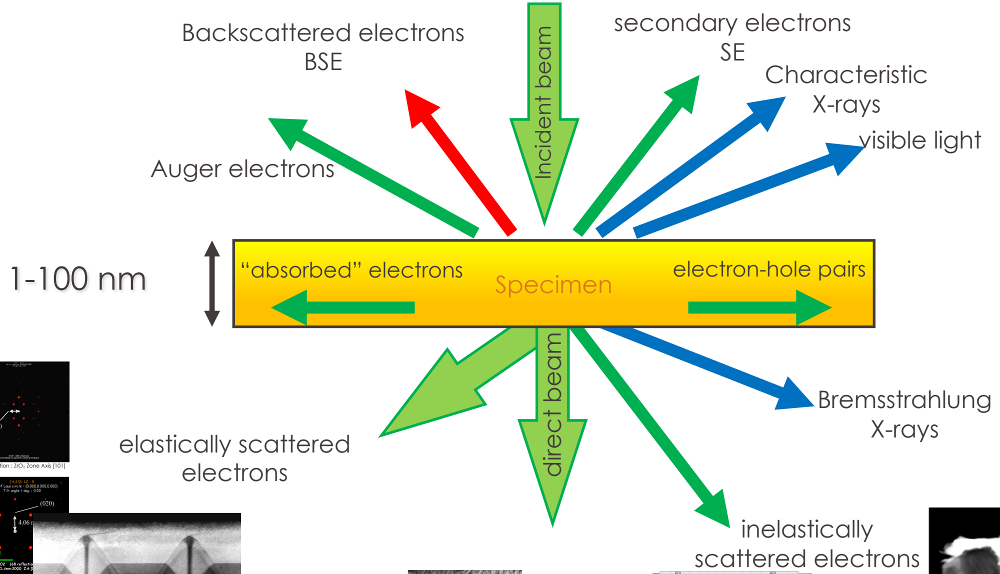


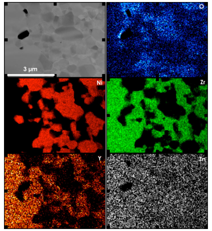
SE mode image on a cleaved surface. The SE_2 (BSE chemical) contrast dominates this image in absence of topographical contrast (SE_1=cte)

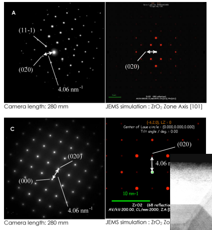
(by courtesy of Dr. K. Leifer, IPEQ/EPFL)

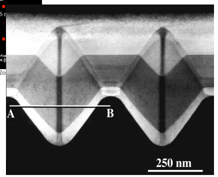
TEM: principle

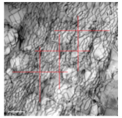
- Interaction of electrons with a thin sample

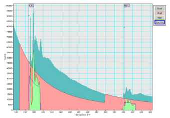


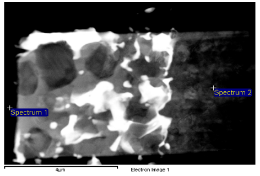






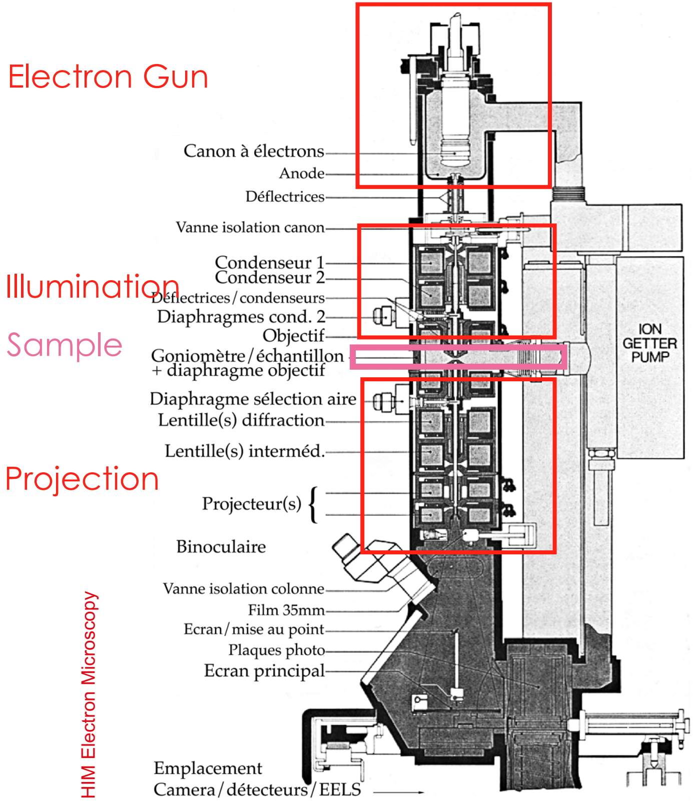






TEM: principle

Hessler-Wyser



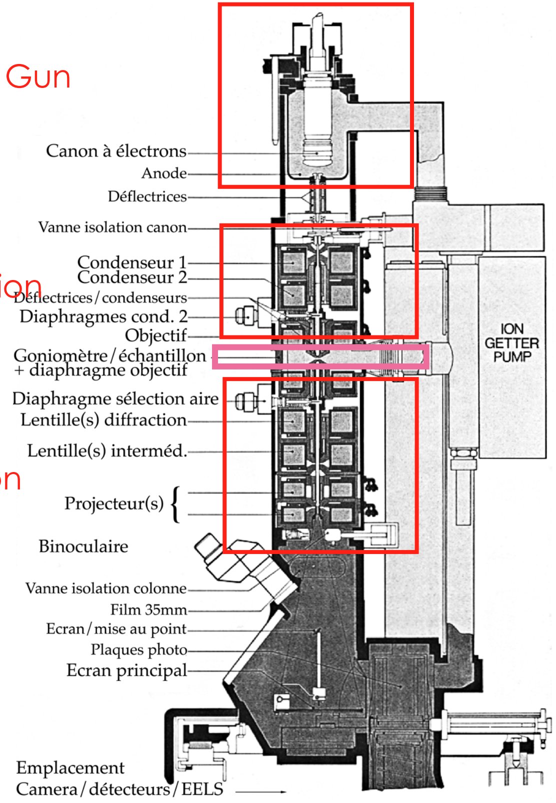
TEM: principle

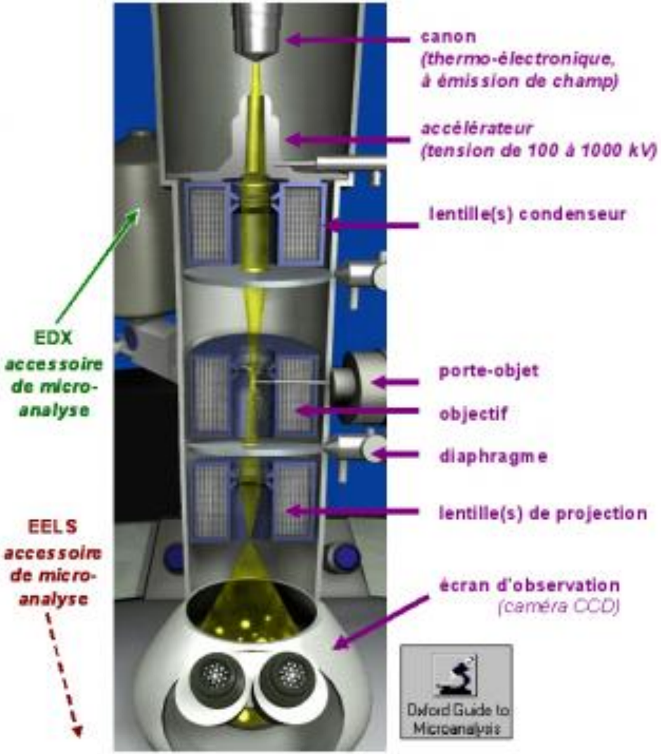
Electron Gun

Illumination

Sample

Projection



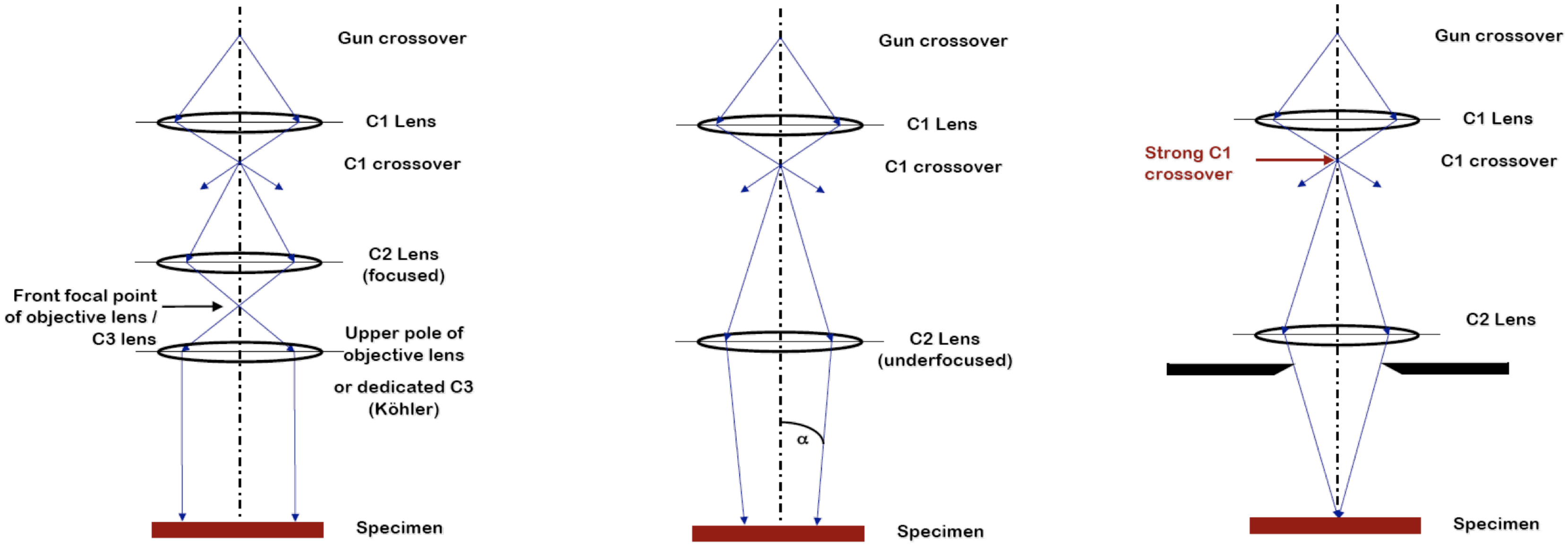


Aïcha Hessler-Wyser

TEM: principle

Aïcha Hessler-Wyser

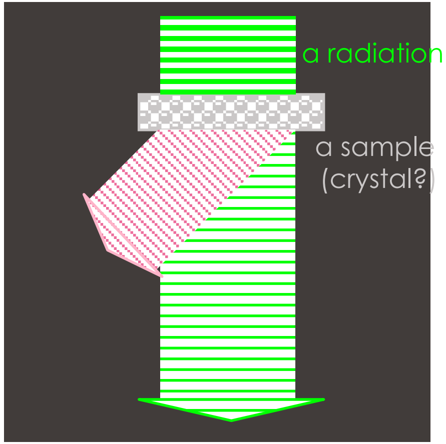
- Parallel or converging illumination

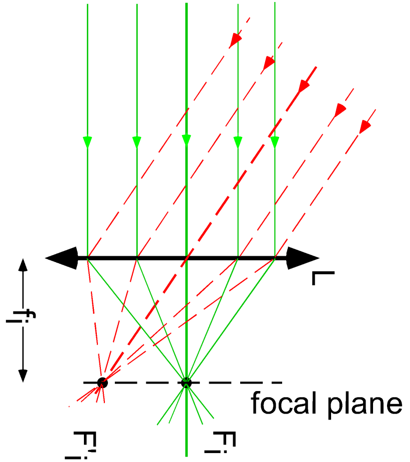


A third lens is needed to make sure to have a parallel illumination

TEM: principle

- When electrons interact with a crystal: Fraunhofer diffraction
  - Diffracted electrons are all parallel when collected by the objective lense and focused in a single point on the focal plane.
  - Similarly, the transmitted electrons are also focused on the same plane, but on a different focal point.





TEM: principle

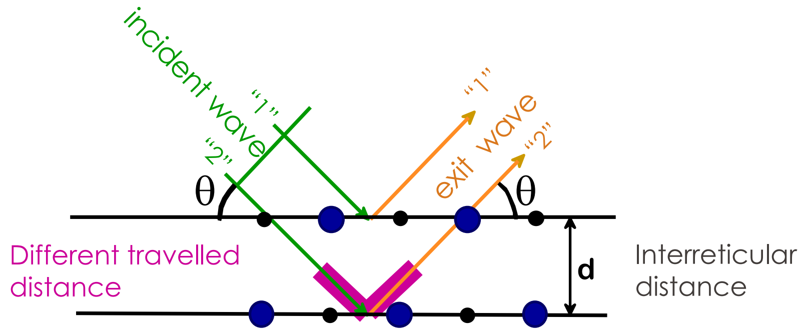
Aïcha Hessler-Wyser

- The Bragg's law

  - Considering an electron wave incident onto a crystal, Bragg’s law shows that waves reflected off adjacent scattering centres must have a path difference equal to an integral number of wavelengths if they have to remain in phase (constructive interference)

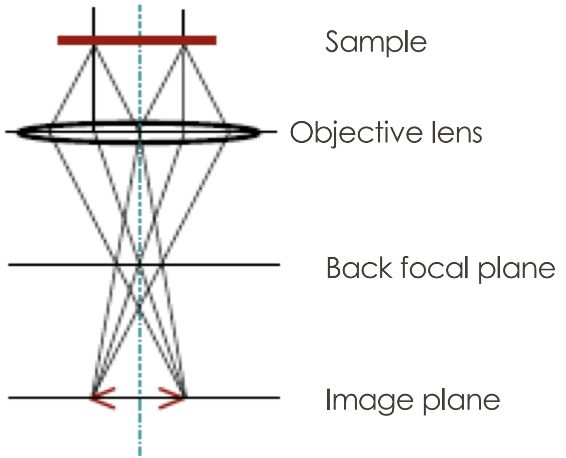
  - In a TEM, the total path difference is $2d\sin\theta$ if the reflecting hkl planes are spaced a distance d apart and the wave is incident and reflected at an angle θ_B.

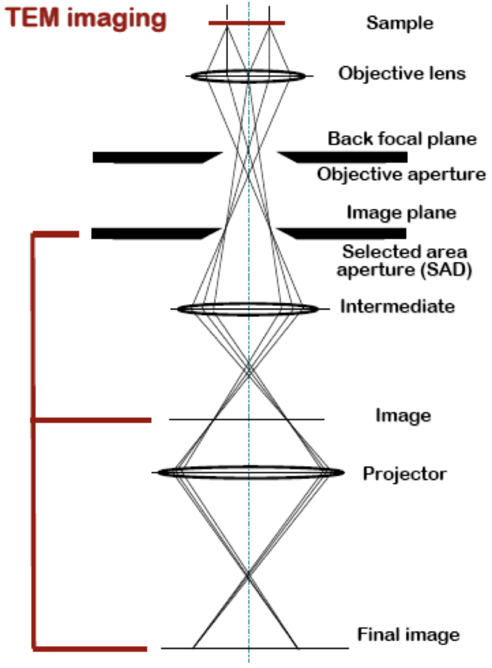
$$2\sin\theta\, d_{hkl} = n\,\lambda$$



TEM: principle

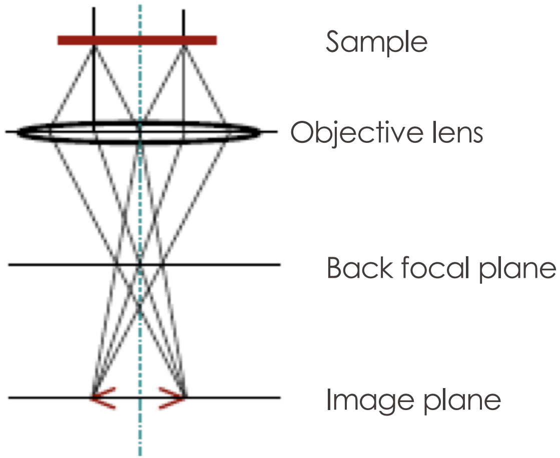
- Image mode

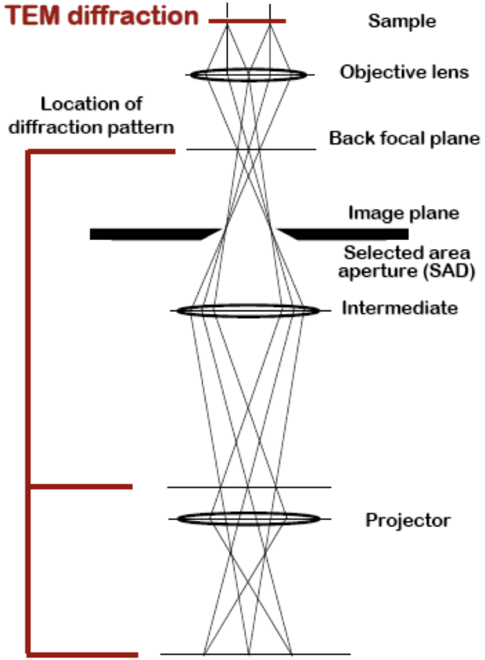




TEM: principle

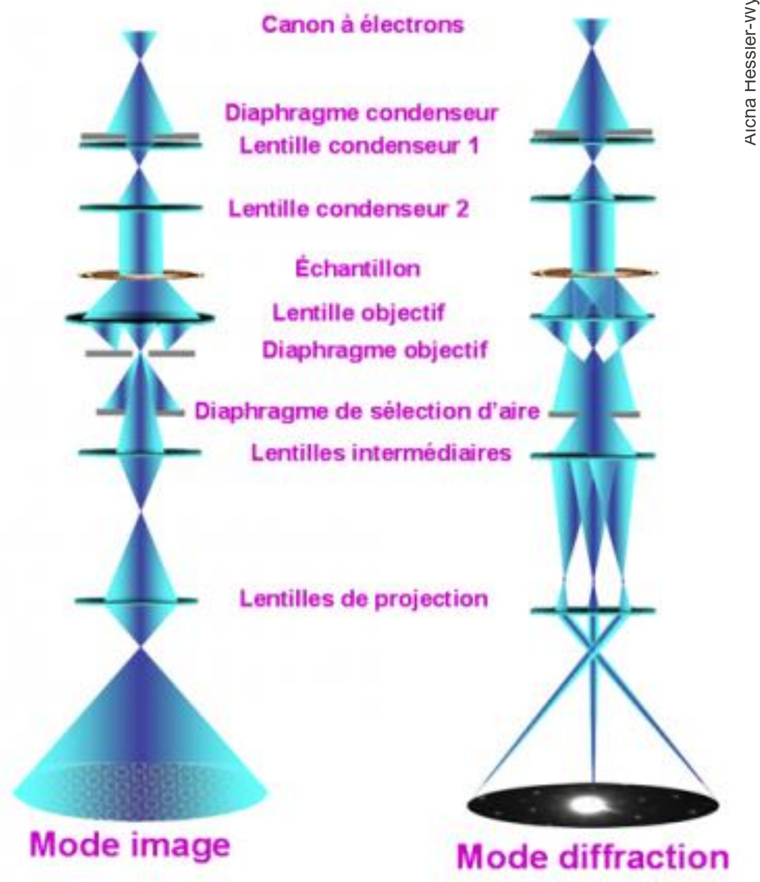
- Diffraction mode





TEM: principle

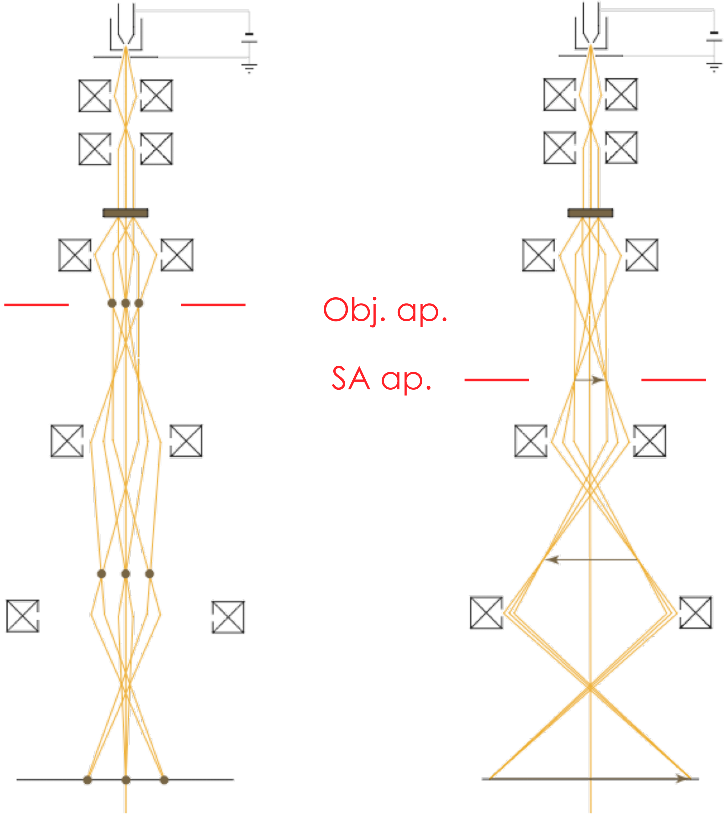
- Diffraction mode
  - Direct correlation between the back focal plane (first diffraction pattern formed in the microscope) of the objective lens and the screen
- Image mode
  - Direct correlation between the image plane (first image formed in the microscope) of the objective lens and the screen



Aïcha Hessler-Wyser

TEM: principle

- Different type of contrats
  - Thickness contrast
  - Z contrast
    } HAADF (D)STEM
  - Diffraction contrast => BF and DF
  - Phase contrast => HRTEM
- The objective aperture allows to select a transmitted spot to increase the contrast in image mode
- The selected area aperture allows to select a region from which the diffraction pattern is considered



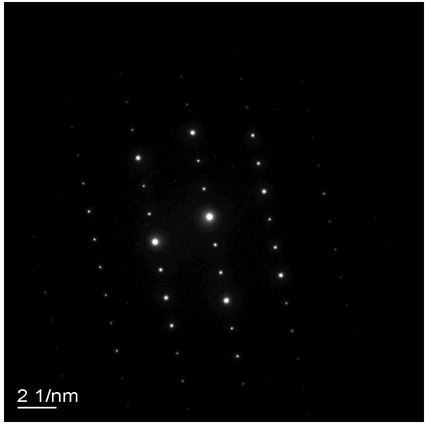
Aïcha Hessler-Wyser

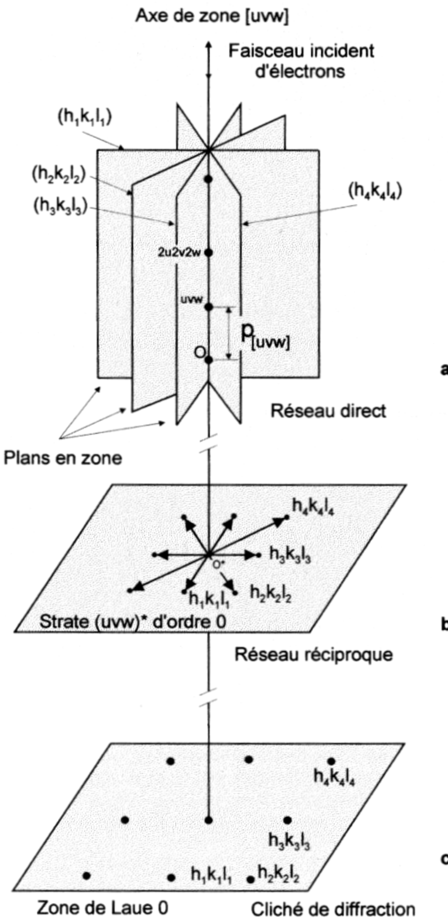
TEM: diffraction

- Diffraction: Zone axis
  - Several (h_i k_i l_i) planes intersect with a common direction [u v w] (zone axis) of the crystal.
  - If electron beam is along [u v w] direction, they all will be in Bragg condition. They satisfy the zone equation:

    $$hu+kv+lw=0$$

  - Each family of crystalline plane diffracts in a single direction. This corresponds to a single spot the the focal plane.





picture from Morniroli

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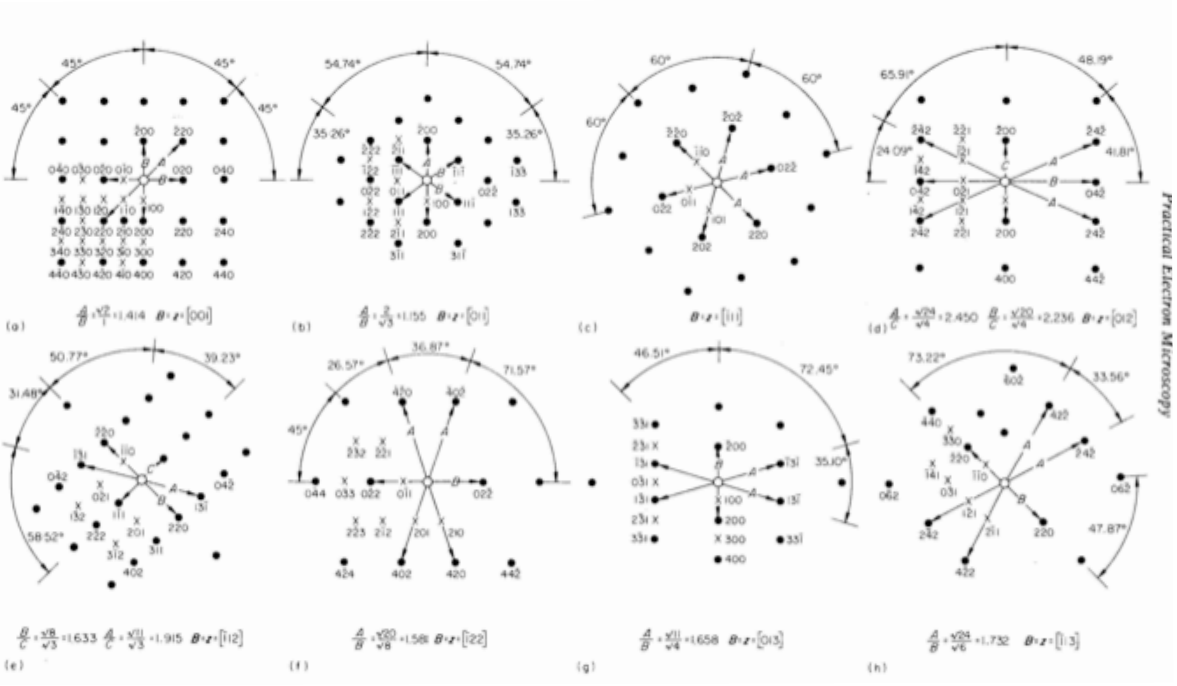
TEM: diffraction

Aïcha Hessler-Wyser

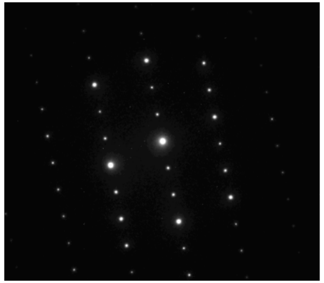
- Typical diffraction patterns for fcc crystal

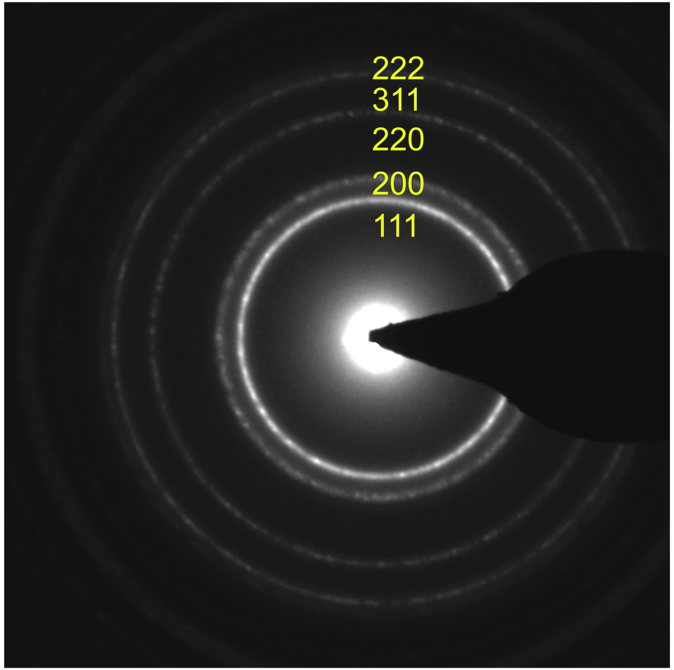


TEM: diffraction

- Powder diffraction

  - Polycrystaline TiCl
  - All reflexions (i.e. all atomic planes with structure facteur) are present
  - They are also called "ring pattern"
  - Angular relations between the atomic planes are lost.





TEM: diffraction contrast

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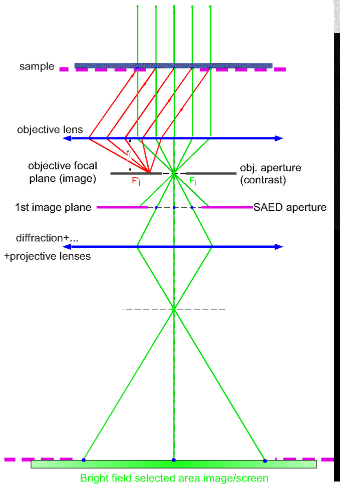
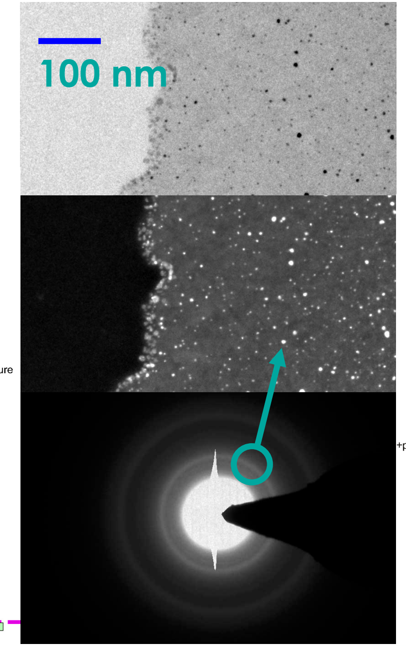
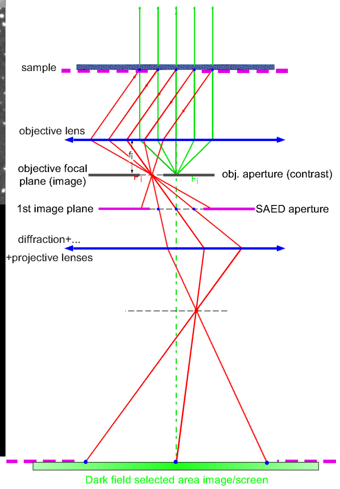
- Bright field (BF) or dark field (DF)
  - Transmitted or diffracted electrons are projected on the image plane

Bright field



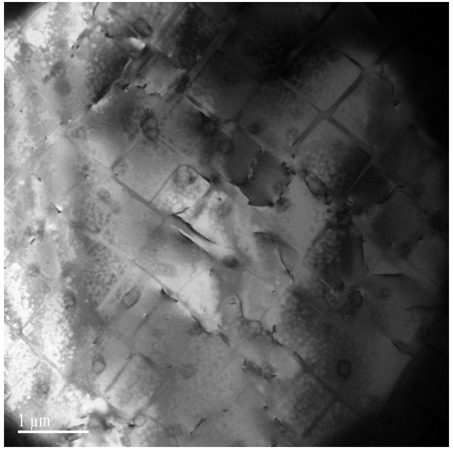


Dark field



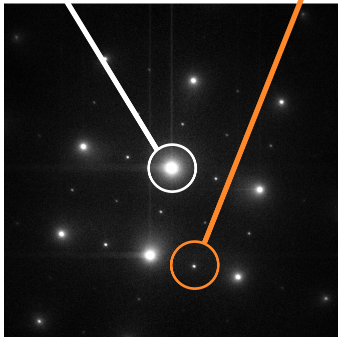
TEM: diffraction contrast

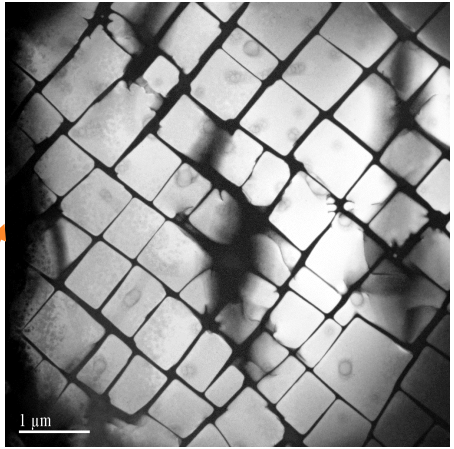
- Bright field (BF) vs dark field (DF)



Nickel based superalloys

Contrast γ/γ'





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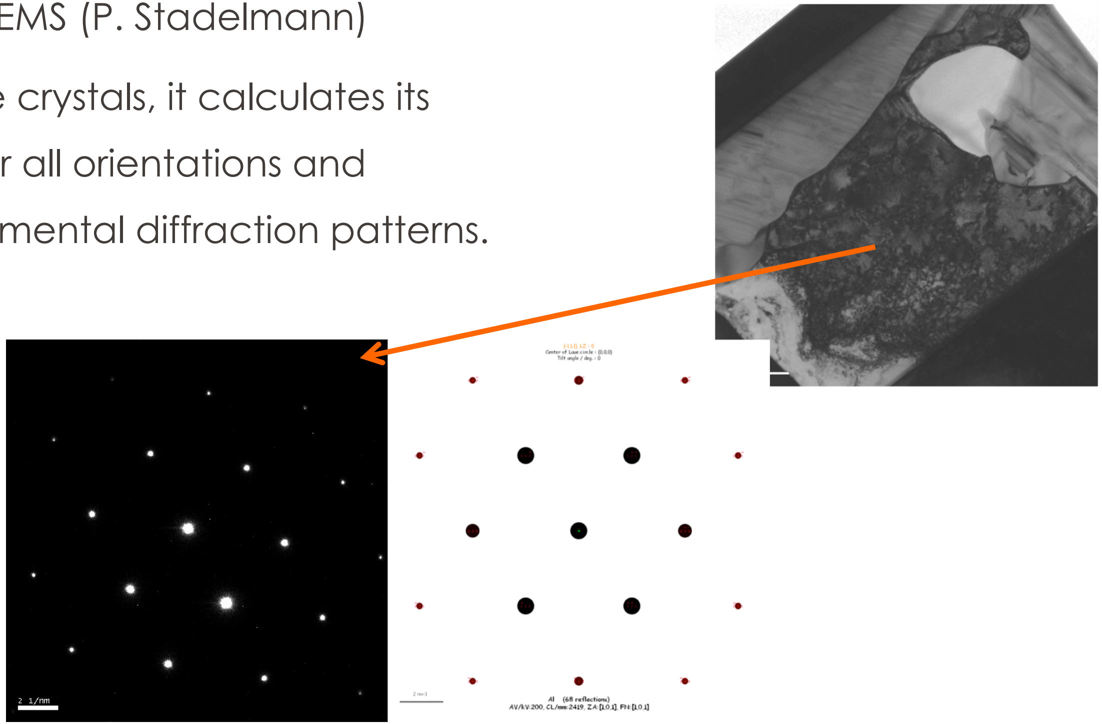
TEM: diffraction contrast

- Diffraction pattern indexing
  - Simulation: Software JEMS (P. Stadelmann)
  - If we propose possible crystals, it calculates its electron diffraction for all orientations and compares with experimental diffraction patterns.



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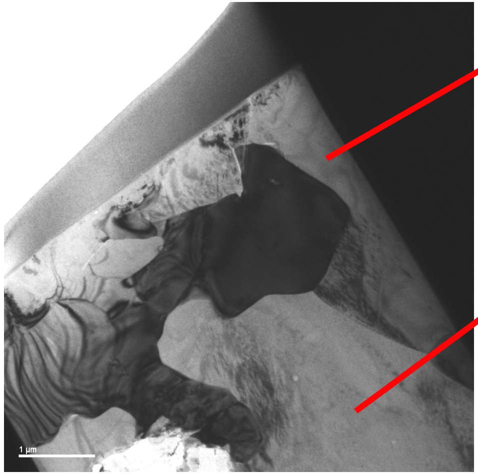
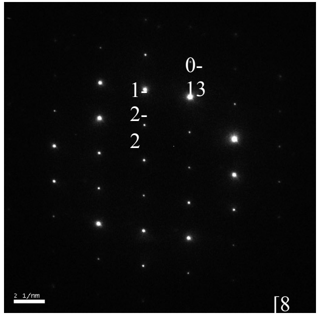
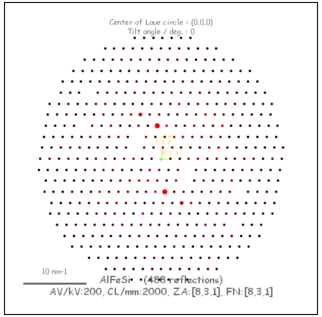
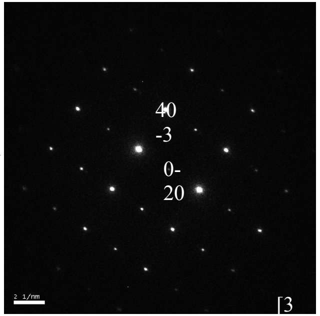
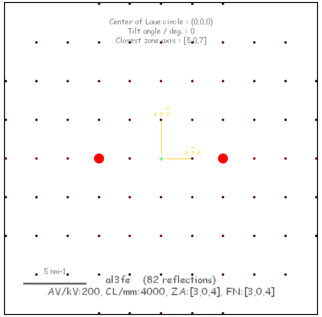






Hexagonal α-(AlFeSi)





Monoclinic Al_3Fe

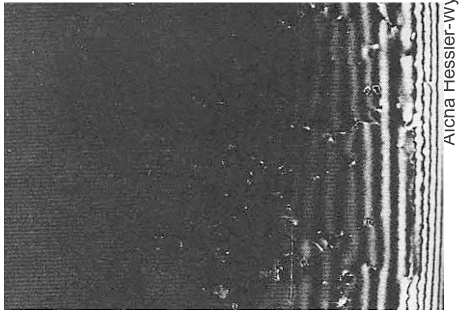
TEM: diffraction contrast

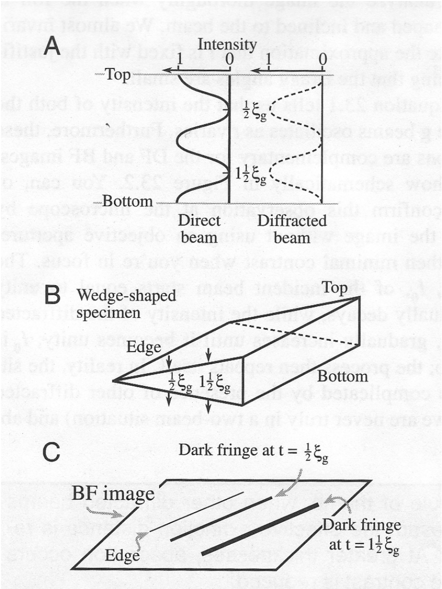
- Thickness fringes
  - If we admit at this stage that a transmitted beam and a diffracted beam can interact in the material, we can calculate the intensity of each one. It varies periodically with the thickness *t*, resulting in equal thickness fringes.
  - If sample is not ultrathin, multiple diffraction can occur. The beam interaction is described by the **Howie-Wheelan equations**:

$$\frac{\partial \phi_g}{\partial z} = \frac{\pi i}{\xi_0}\phi_g + \frac{\pi i}{\xi_g}\phi_0 \exp[-2\pi i s z]$$

$$\frac{\partial \phi_0}{\partial z} = \frac{\pi i}{\xi_0}\phi_0 + \frac{\pi i}{\xi_g}\phi_g \exp[2\pi i s z]$$

(ϕ_g: diffracted beam, ϕ_0, transmitted beam,
ξ_g et ξ_0 extinction distance, z position in the thickness)





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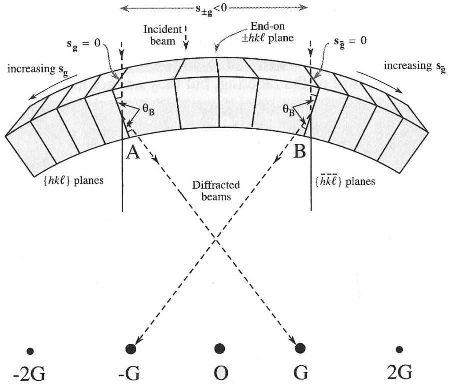
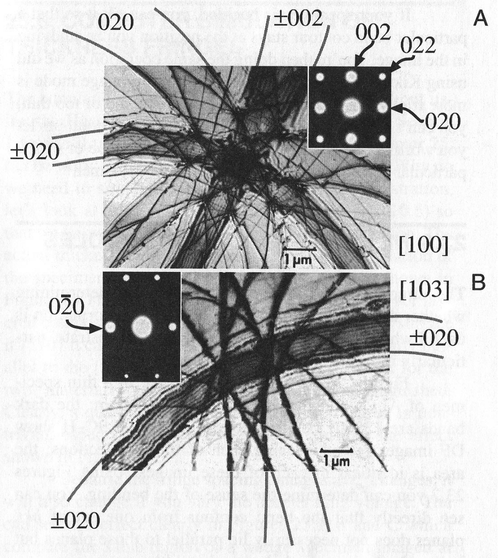
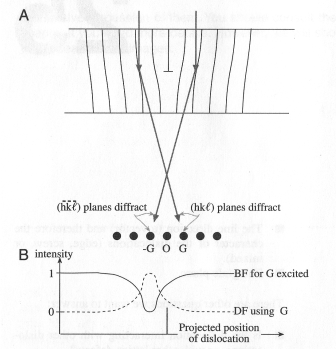
TEM: diffraction contrast

- Bend contours, dislocations
  - When a sample is deformed, the diffraction conditions are not the same in two different regions.
  - In bright field, the diffracting area appears in dark.
  - It is then possible to observe lines with a different contrast: they are called bend contour.
  - Each line can be associated with a family of diffracting planes.
  - Similarly, dislocations can be visible in BF or DF







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EPFL

TEM: diffraction contrast

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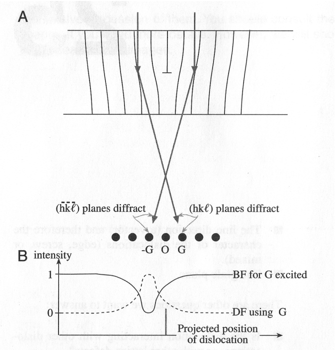
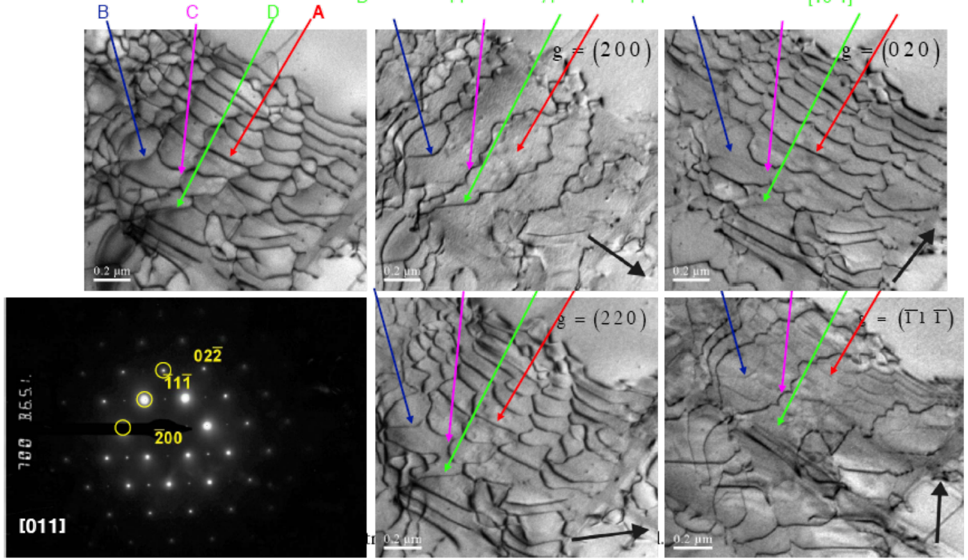
- Dislocation network in Ni-based superalloys



reflection	200	020	220	-11-1	b // to
A	--	++	++	-	[0-11]
B	++	--	+	-/+	[10-1]
C	++	--	++	++	[???]
D	++	-/+	++	--	[10-1]



Burger's vertor ***b*** identification

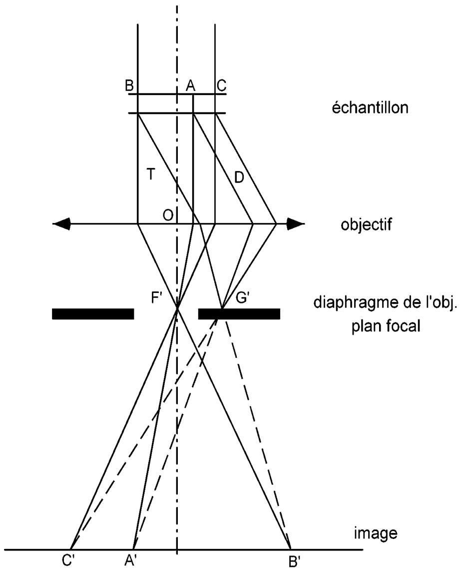
4 different orientations (***g***)

If ***g*** // ***b***, the dislocation is visible
If ***g*** ⊥ ***b***, the dislocation is not visible

TEM: phase contrast

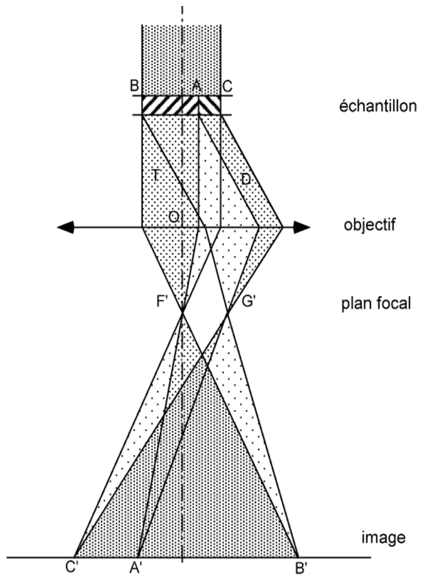
- A high-resolution image is an **interference** image of the transmitted and the diffracted beams!
- Diffracted electrons: coherent elastic scattering (the electrons have seen the crystal lattice)
- The quality of the image depends on the optical system that makes the beams interfere

BF



a) Réglage en champ clair

HRTEM



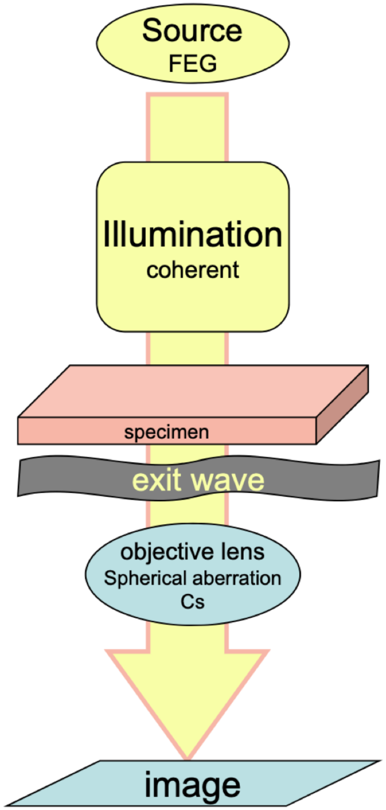
b) Microscopie Electronique à Haute Résolution

TEM: phase contrast

- Image formation
  - **Source:** coherent and monochromatic
  - **Illumination:**parallel
  - **Sample:** thin, nicely prepared (no amorphization), orientation (zone axis)
  - **objective lens:** aberrations, focus, stability !
  - **Projection** lens system (magnification)

- Lens system is a sort of a black box that can be modelled by the **transfer function**

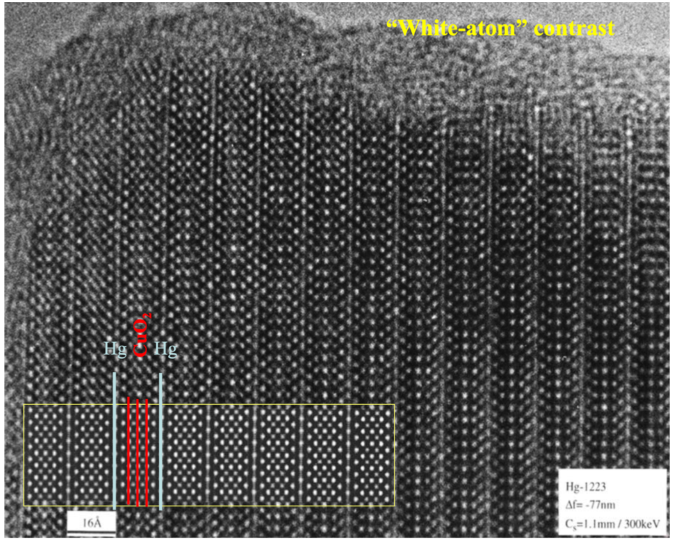
- Experimental image has to be compared to simulations to take into account aberrations and defocus of the objective lens.

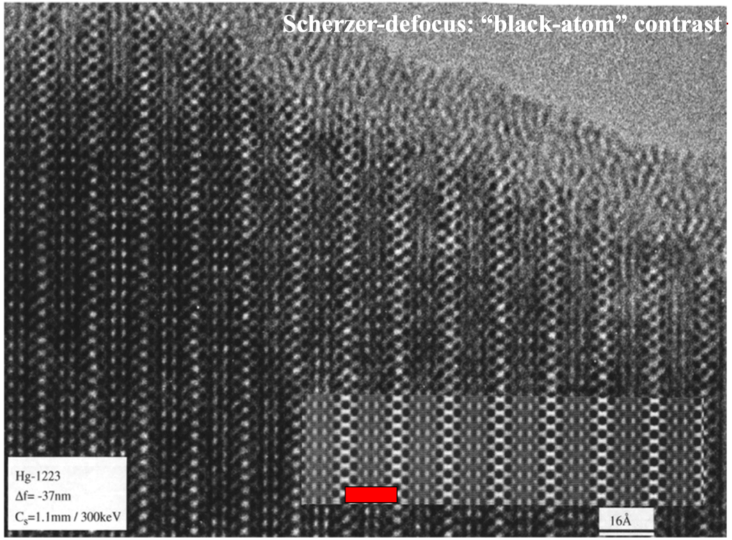


Courtesy of M. Cantoni

TEM: phase contrast

- Contrast changes with defocus and spherical aberration
- The atoms are not necessarily white dots on black background!
- Simulations are necessary!

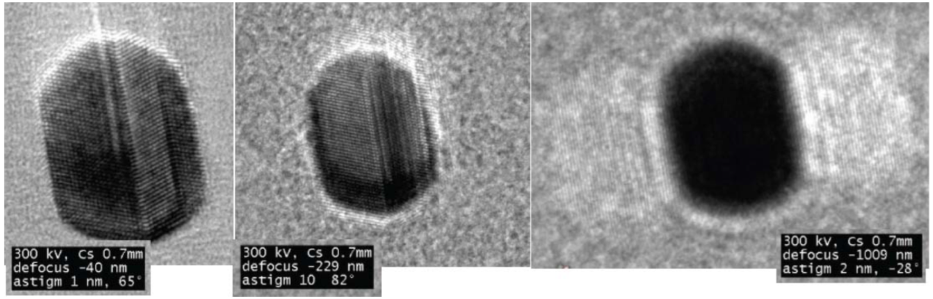




Courtesy of M. Cantoni

TEM: phase contrast

- Gold crystal
  - Defocus, Cs and astigmatism induce delocalisation of the information
  - Cs correction:
  - No more delocalisation!
  - Accurate interfaces



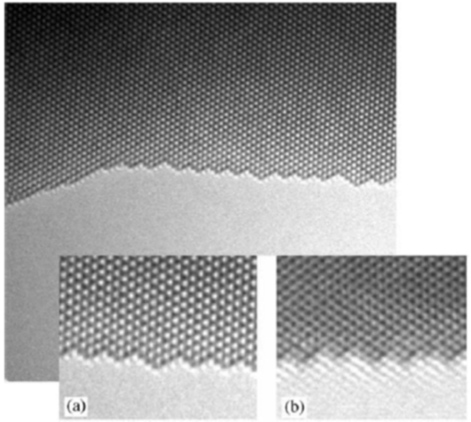
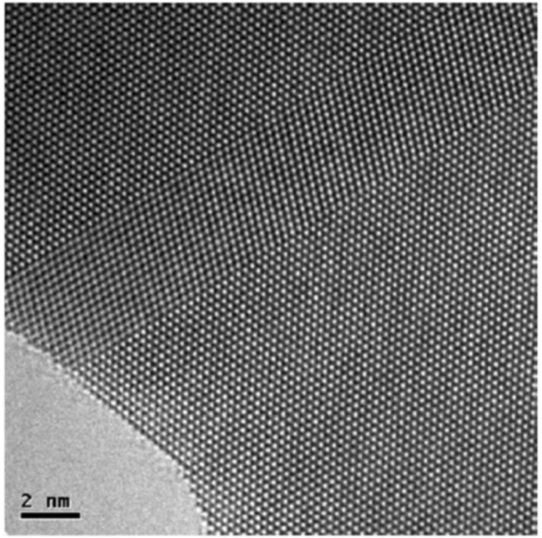


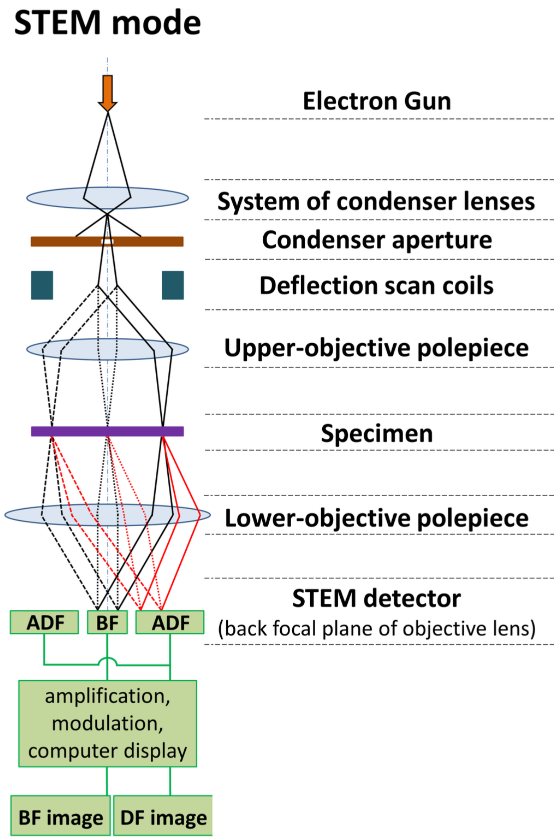
Fig. 5. Images of gold crystal recorded with monochromator on and Cs corrector on, with insets illustrating the difference between Cs corrector on and Cs corrector off: (a) The edge is clearly imaged, without fresnel fringes or delocalization effects. (b) The image without Cs correction, illustrating the difficulty of directly interpreting an image from a conventional field emission gun system.



Courtesy of M. Cantoni

STEM: principle

- Beam is convergent
- Probe scans the sample
- Central detector collects the transmitted electrons
- Annular detector collects the diffracted electrons: ADF (anular dark field)
- Detectors collect signal, point per point
- An image is reconstructed with a **diffraction contrast**
- With a third annular detecor (HAADF: high angle annular dark fiedl), one can collect the electrons that are diffused
- The reconstructed image shows a Z contrast (atomic number)



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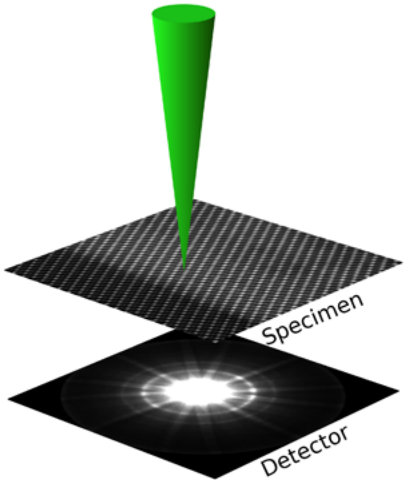

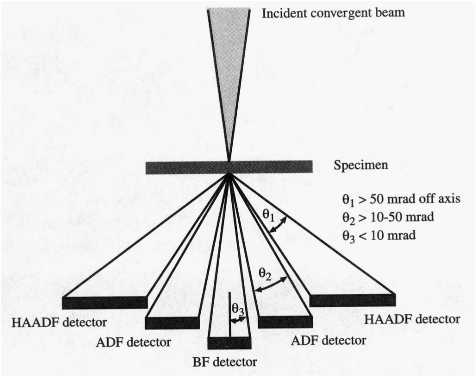

STEM: principle

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- HRTEM vs STEM HAADF

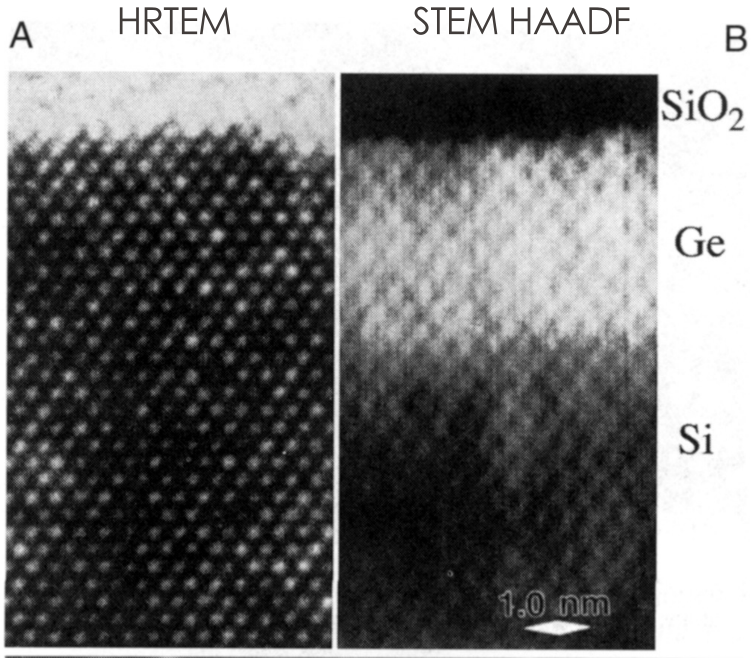
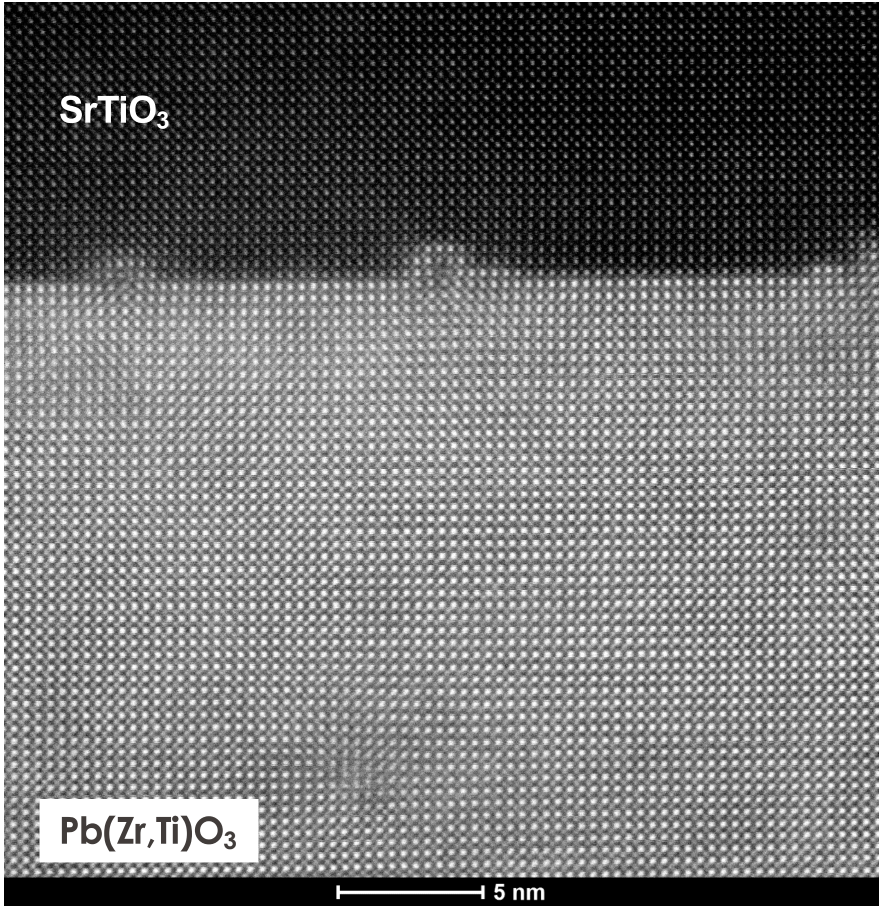


Figure 1–16. Images of a Ge film grown epitaxially on Si by an implantation and oxidation method. (**a**) Conventional TEM image from a JEOL 200CX, (**b**) Z-contrast image obtained with a VG Microscopes HB501UX clearly delineating the Ge layer, reproduced from Pennycook (1989a).

Pennycook et al.

STEM: principle

- Correction of spherical aberration
- The interface is resolved at the atomic scale!



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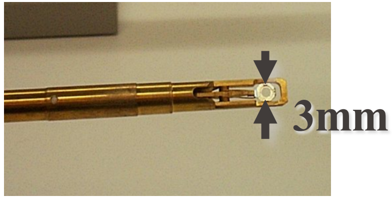
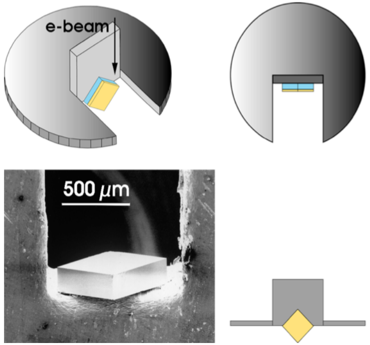
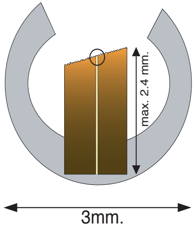
TEM: sample preparation

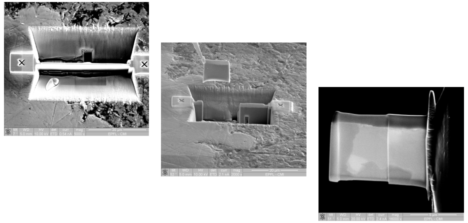
- Electron transparency
- < 100-150 nm thickness
- Fit in the sample holder
- 3 mm in diameter
- Keep the microstructure of the sample!!





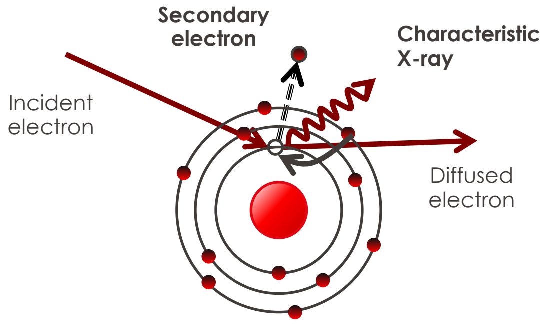
- Mechanical polishing
- Focussed ion beam
- Cleavage method
- Powder on a thin film
- …



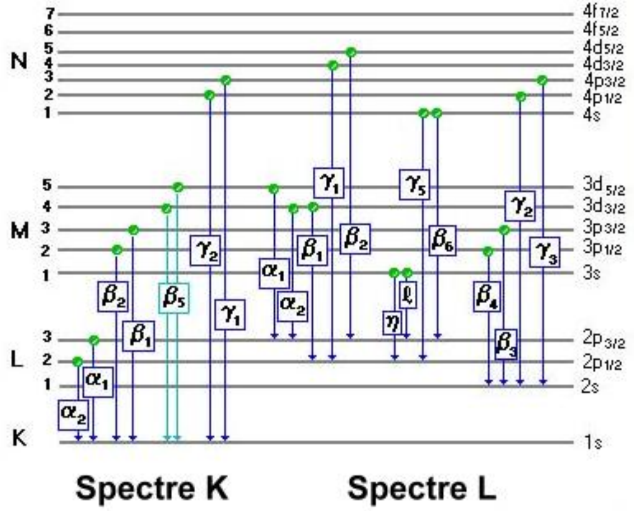


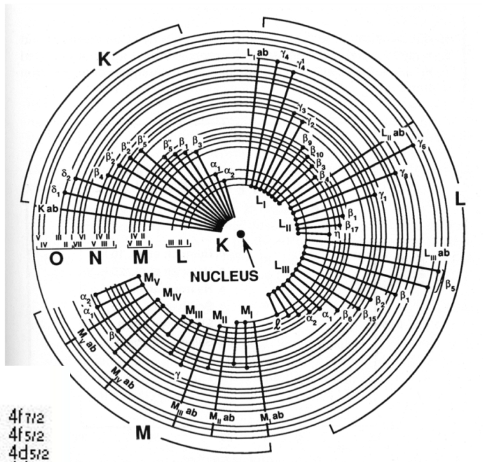
Other techniques: EDS

- Characteristic X-rays



Each element has its proper serie of emission raies





3.37. Comprehensive energy-level diagram showing all electron transitions which give rise to K, L, and M x rays (Woldseth, 1973).

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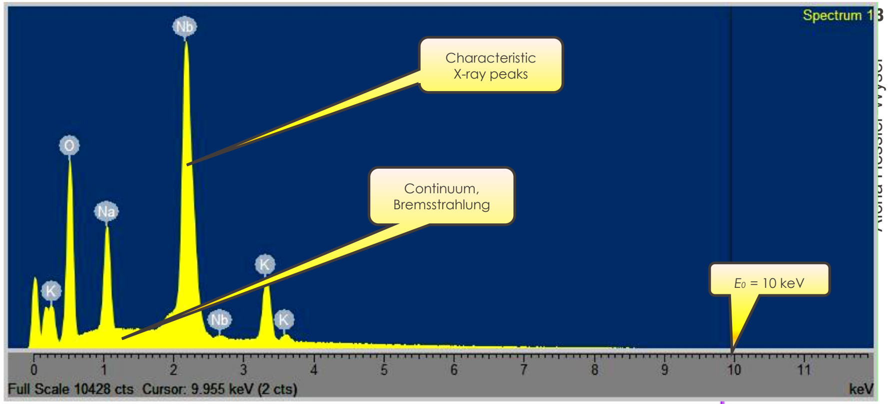
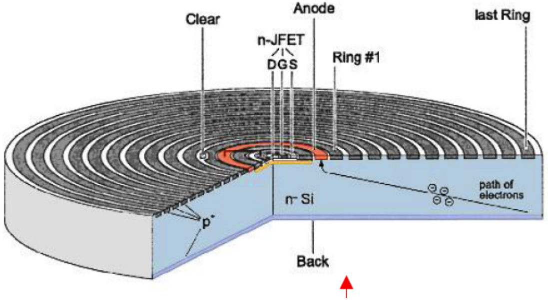
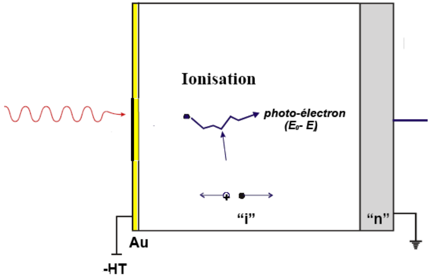
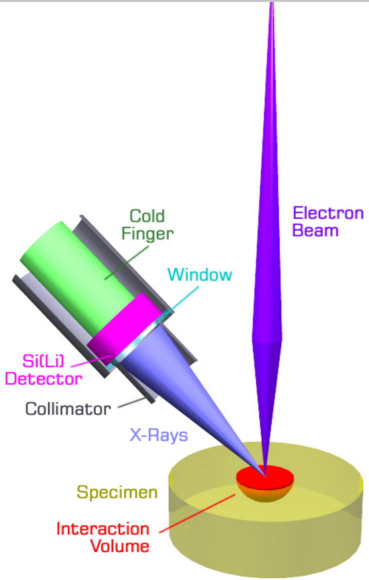
Other techniques: EDS

- Detection of X-rays
- Count as a function of energy
- Correction ZAF
  - Z: atomic number
  - A: absorption
  - F: fluorescence
- Determination of element concentration









Other techniques: EDS

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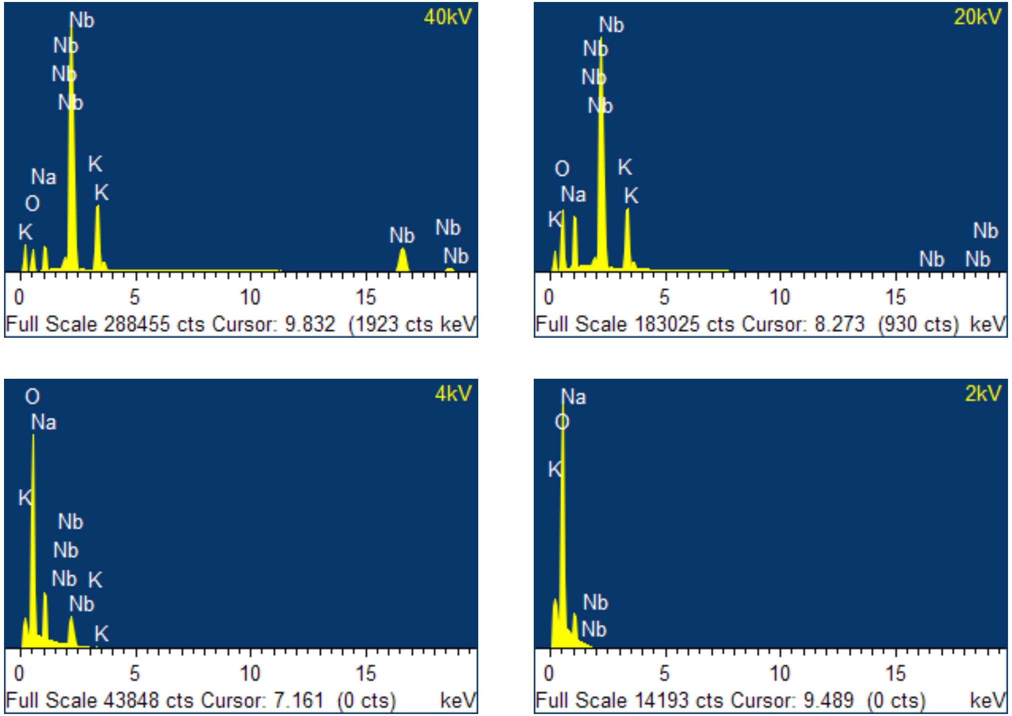
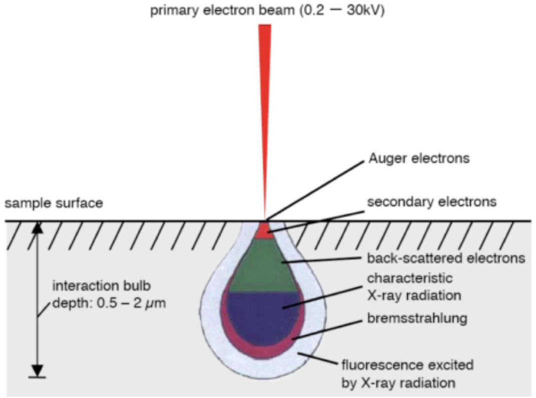
Same sample
Different conditions

- Quantification: intensity ≠ concentration!!

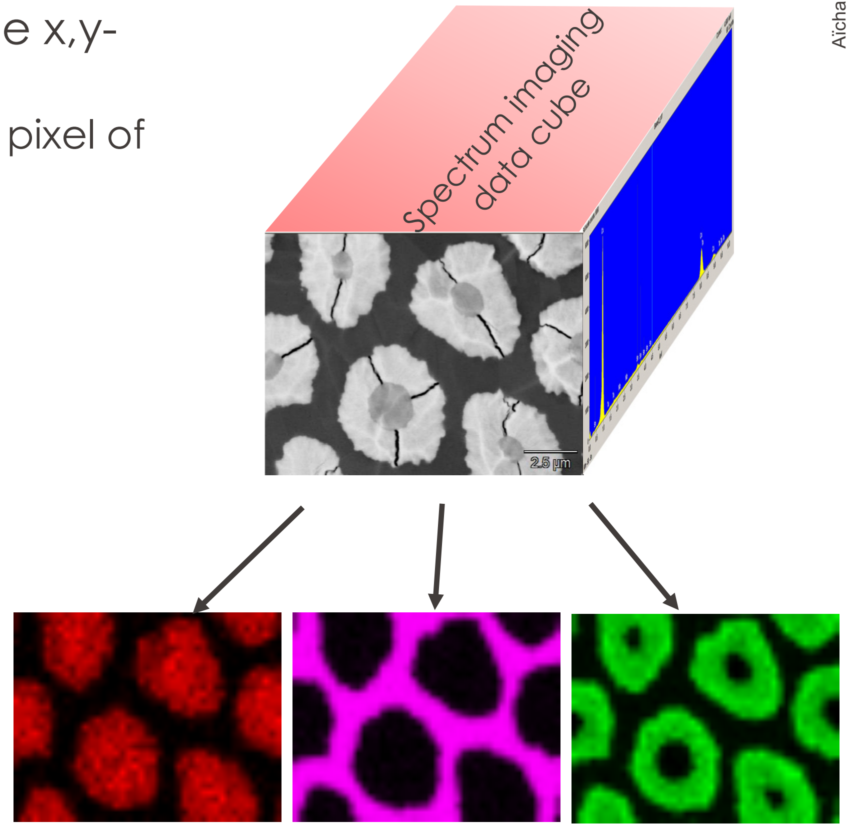




Other techniques: EDS

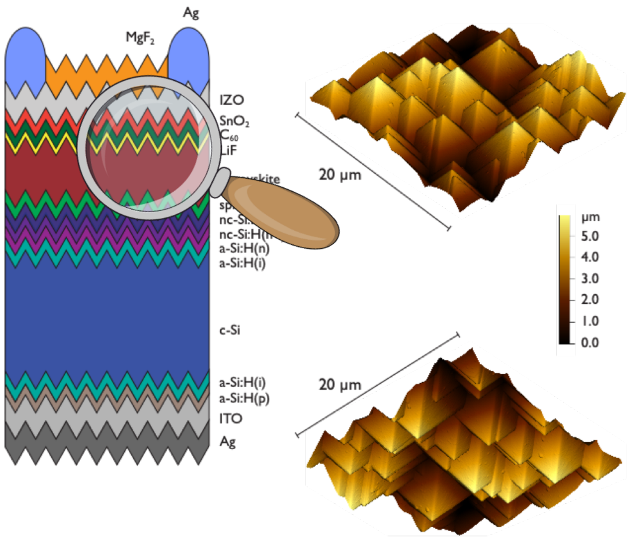
- Element mapping: datacube space x,y-energy
  - A full spectrum is acquired for each pixel of the image.
  - The data are recorded in 3D: x, y, E

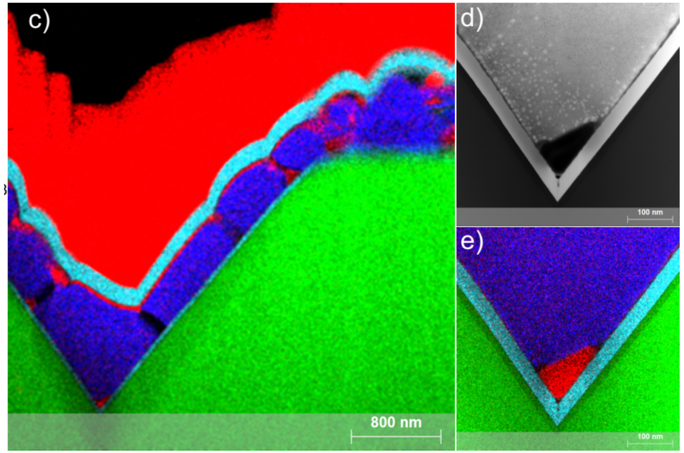
- Post-acquisition processing
  - Each spectrum can be analyzed and quantified
  - Each pixel is assigned a concentration of one element
  - This represents **mapping by element**



Other techniques: EDS

- Example of perovskite/Si tandem solar cells

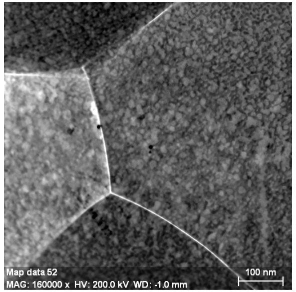


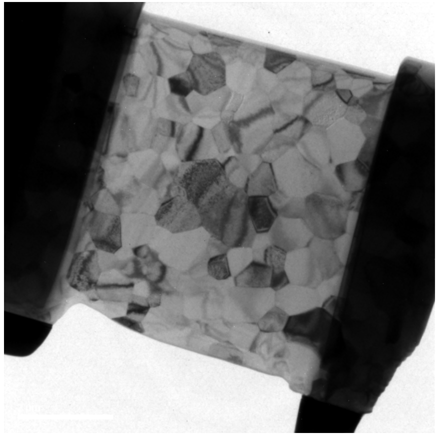


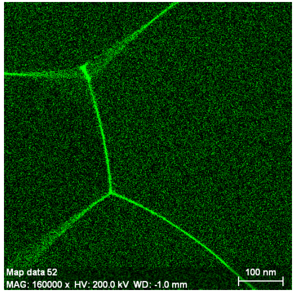
Other techniques: EDS

Aïcha Hessler-Wyser

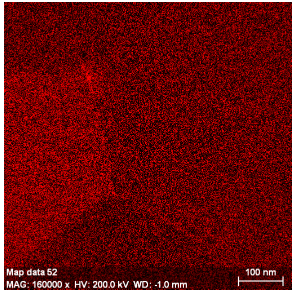
- Can also be done in a TEM
- Synthetic saphire
- La-doped Al_2O_3
- STEM + EDS shows La segregation at grain boundaries







La at grain boundaries

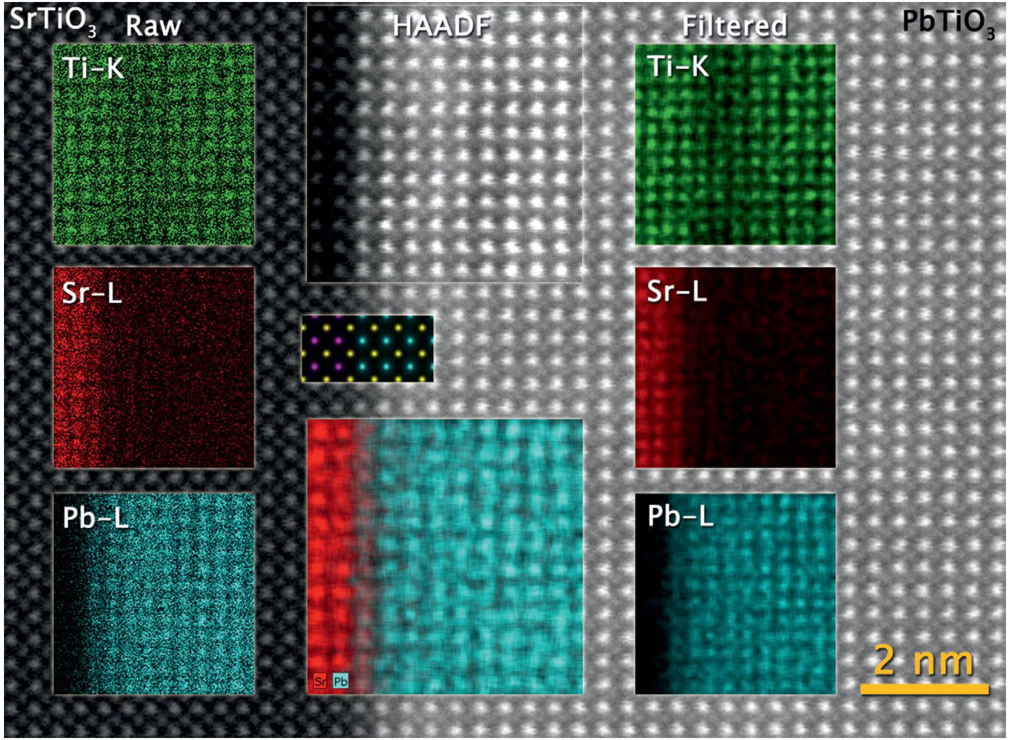


Cl (from synthesis)

P. Bowen, M. Stuer

Other techniques: EDS

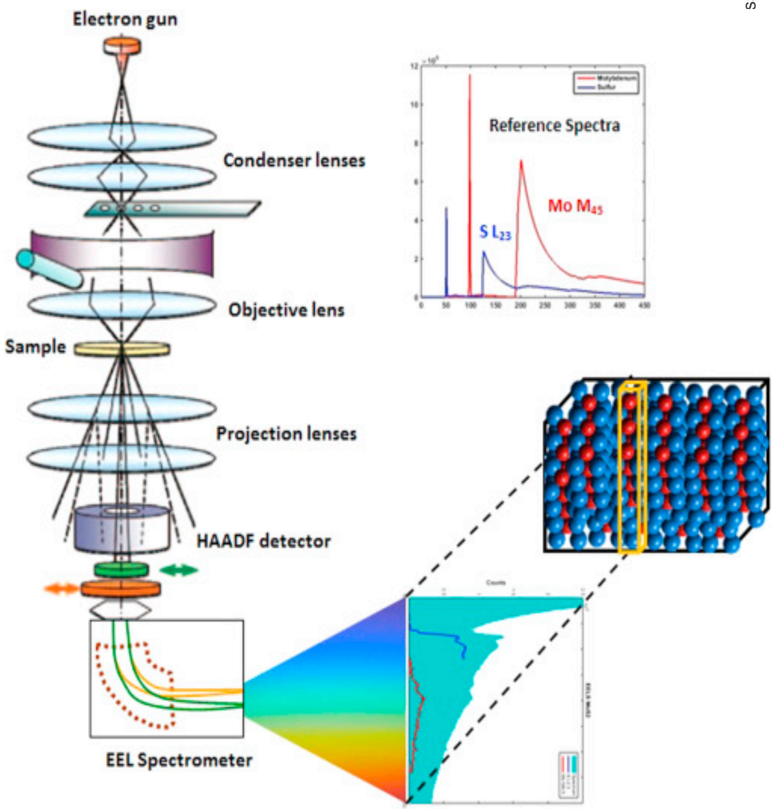
- $SrTiO_3/PbTiO_3$ interface
- Atomic resolution for imaging
- Atomic resolution for element mapping



Aïcha Hessler-Wyser

Other techniques: EELS, EFTEM

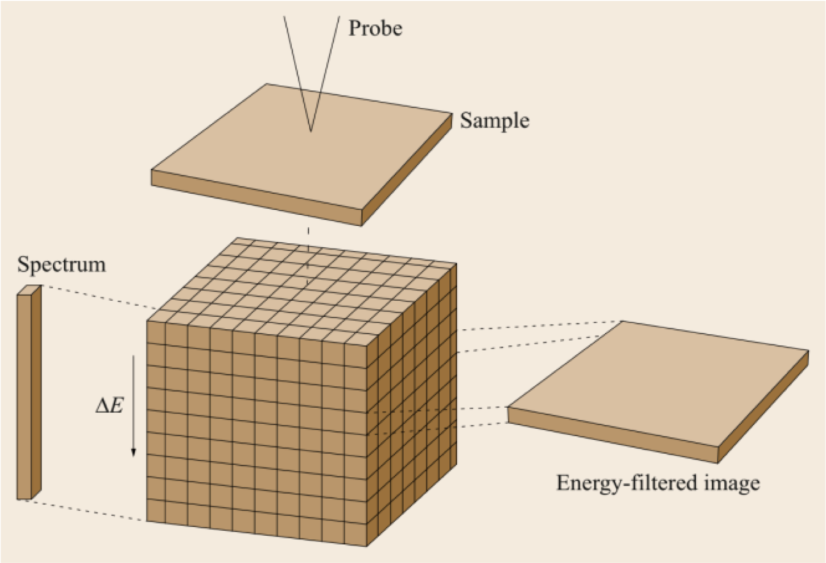
- Electron energy loss spectroscopy
  - Electrons lose energy when interacting with the thin sample
  - The loss is directly linked to the sample atoms
  - The sample composition can be determined
  - The electric structure can also be obtained
- Energy filtered TEM
  - A slit is placed after the filter
  - An image is formed only with the electrons of the selected energy loss



Other techniques: EELS, EFTEM

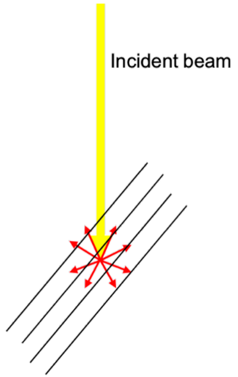
- Energy filtered TEM
  - A slit is placed after the filter
  - An image is formed only with the electrons of the selected energy loss

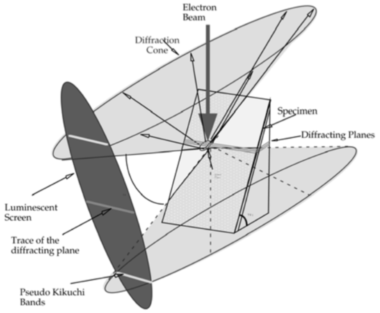
- Datacube can be constructed either in STEM + EELS or in EFTEM

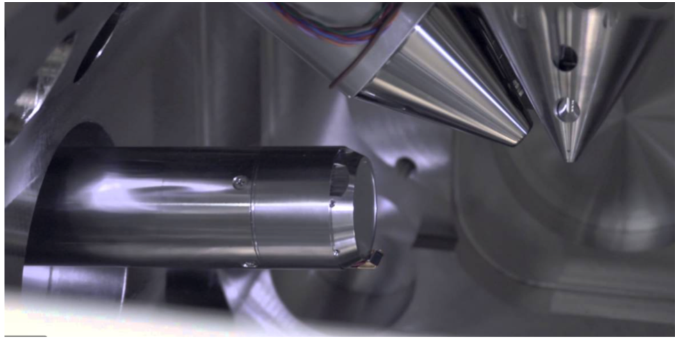


Other techniques: EBSD

- Electron back-scattered diffraction
  - Electrons are inelastically diffused
  - Crystal planes diffract the electrons according to Bragg's law
  - If electrons are close to the surface, they can escape in well defined directions
  - A fluorescent screen collect them

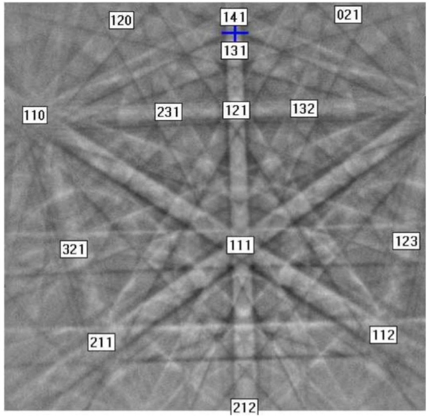


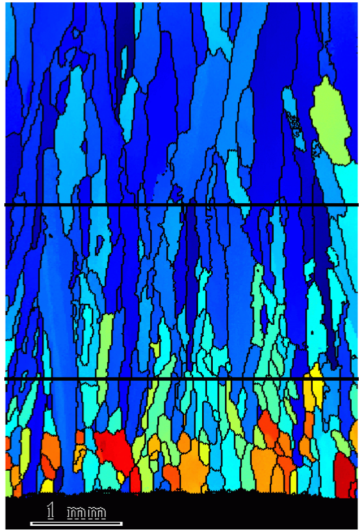




Other techniques: EBSD

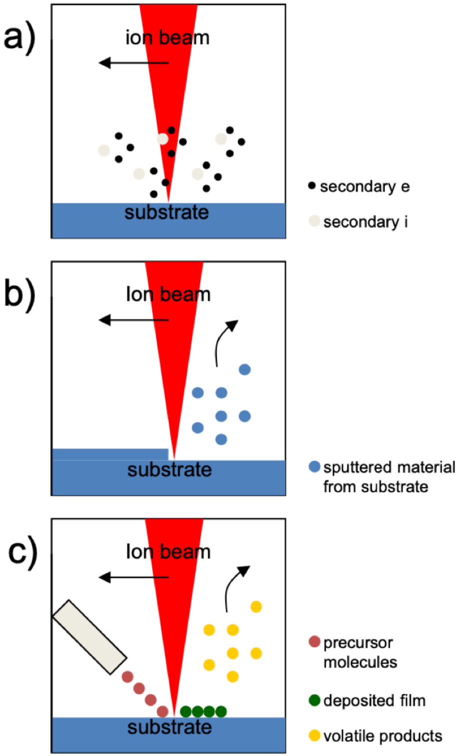
- Kikuchi pattern
  - Patterns are indexed by comparison with simulations
  - Each pixel is attributed a phase and orientation
  - Construction of orientation maps





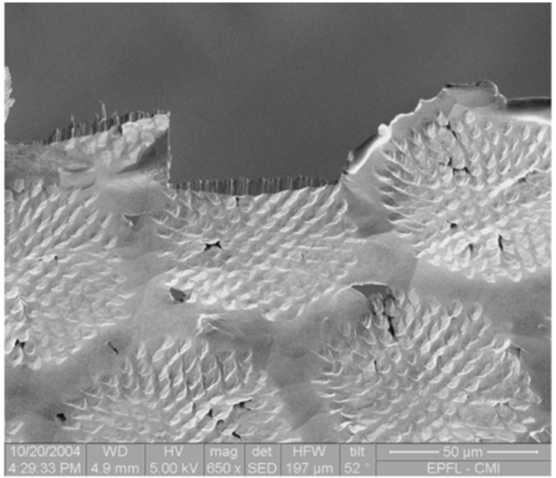
Other techniques: FIB

- Focussed ion beam
  - Emission of secondary ions and electrons: **FIB imaging**
  - Sputtering of substrate atoms: **FIB milling**
  - Chemical interactions (gas assisted): **FIB deposition**

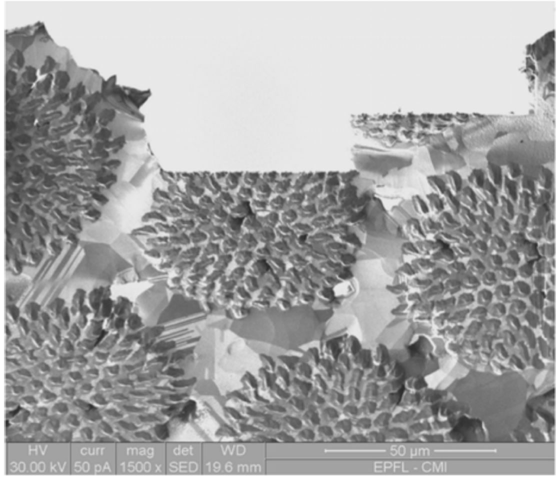


Other techniques: FIB

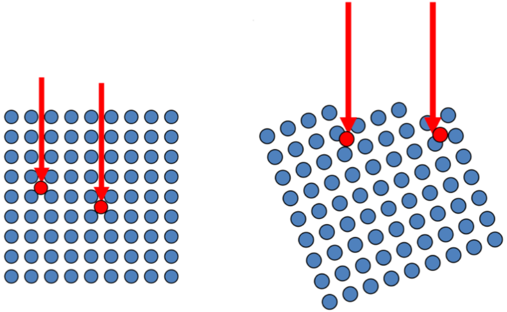
- FIB imaging (SE detector)



e-beam 5 kV



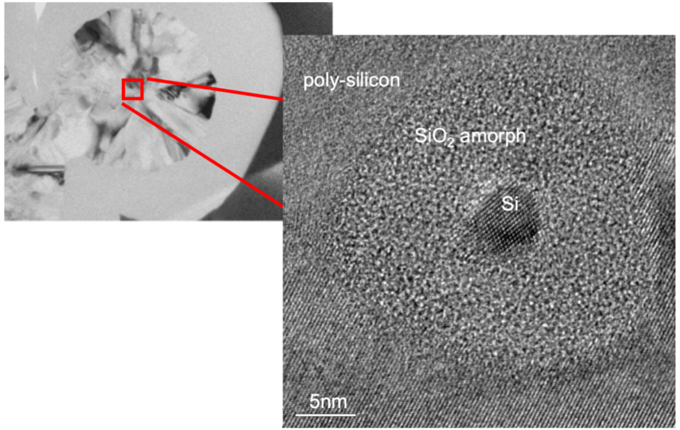
Ga⁺-beam 30 kV, 50 pA



Channeling contrast

Other techniques: FIB

- Deposition, milling, imaging: TEM sample preparation



Si nano-wire

M. Pavius, V. Pott, CMI

Other techniques: FIB

- Milling + imaging

- Nanotomography

- 3D reconstruction of 5x5x5 µm volumes