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Scanning Electron Microscopy

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Scanning Electron Microscopy

Université Technologique de Troyes - 2024

Scanning Electron Microscopy – Franck CARCENAC
LAAS – CNRS 7, avenue du Colonel Roche 31031 TOULOUSE - FRANCE

- **Switch from microscopy to characterization**
 - ☞ Knowledges, concepts, tools, tips, tricks, ...
- **Scanning Electron Microscopy:**
 - A technique with performances and limits
 - ☞ **Learn how to use it to be able to quickly achieve the best possible result**
 - ☞ Choose parameters
 - ☞ Get the best results
 - ☞ Quickly adapt your set of parameters if needed
- **Characterization:**
 - ☞ **Gathering useful and valuable informations from your observation/analysis using SEM**

- **Histories**
- **SEM presentation**
 - Principle
 - Electron beam generation
 - Electron optics
- **Electron/Matter interactions**
 - Phenomena involved
 - Origin of contrasts
 - Detectors - examples
- **SEM in practice**
 - Observation parameters choices
 - Influence of choices
 - Procedures: alignments, astigmatism/focus adjustment

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- 1932 Reinhold Rüdénberg – Patented electron microscope
- 1937 Manfred Von Ardenne - SEM 1st prototype
- 1938 First commercial electron microscope: Siemens
- Today ... world competition:
 - Electron optics
 - Electrons sources
 - Detectors
 - Software
 - Additional capabilities:
 - Correlated microscopies (optical, fluorescent, AFM, ...)
 - Spectroscopies: electrons, photons
 - Materials properties: crystallography, strength, corrosion, ...

- Entirely numerical microscopes
- Less “frightening” ergonomics
- Alignment coils
- Numerous useful automatic functions
- **Remember: physics does not change with microscopes**



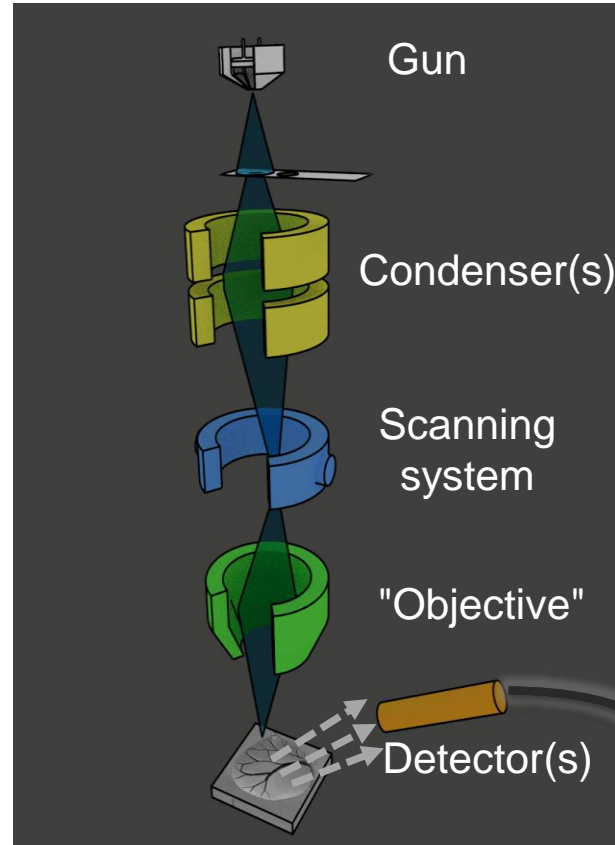
today ...



- **Schottky - Field Effect Gun (S-FEG)**
- **IN-LENS detectors (secondary & backscattered electrons)**
- **Imaging at very low energy (<1kV):**
 - Polarizing sample up to ~4kV
 - Imaging apparent voltage = electron beam accelerating voltage - sample polarization voltage
 - Keeps resolution and limits charging effects
- **Environmental / low vacuum microscopes:**
 - Direct observation on insulating samples without treatment:
 - Variable pressure in the chamber
 - Ionizing molecules (+) on the electrons' trajectory
 - Neutralizing charges (-) from the beam (electrons) at sample's surface
 - At the cost of resolution (electrons diffusion on their trajectory)
- **Monochromator (increase resolution at low energy # $\propto \Delta E/E$)**
- ...

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- Focused electron beam
- 1 point on sample = 1 point on display
- Display point brilliance:
 - f (interaction, detector)
- Screen/sample synchronous scanning
 - Point per point reconstructed image
- Magnification = f (distance between pts)
 - Resolution do not depend on magnification
 - Resolution is mainly given by beam diameter
- Fixed point: screen center



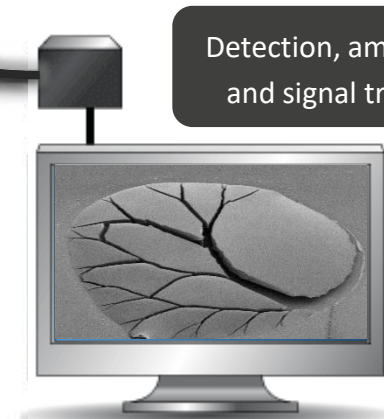
B. Reig - LAAS-CNRS

Source production for the optical system

Size's source reduction (beam/spot size)

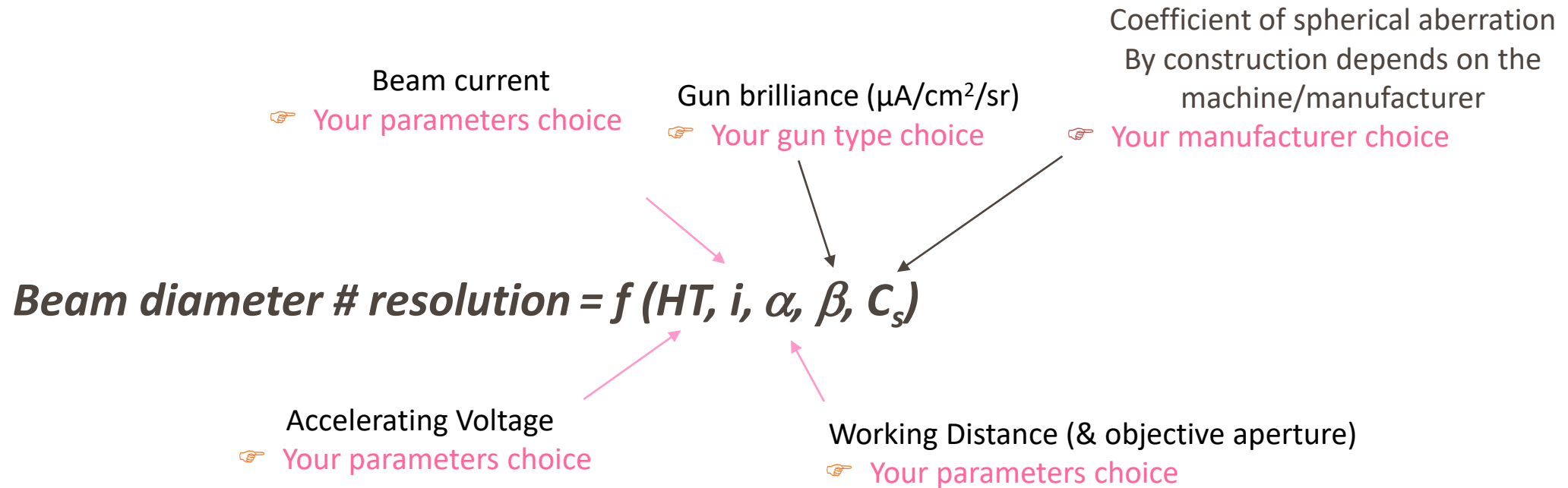
Projection of the source on the sample (focus)

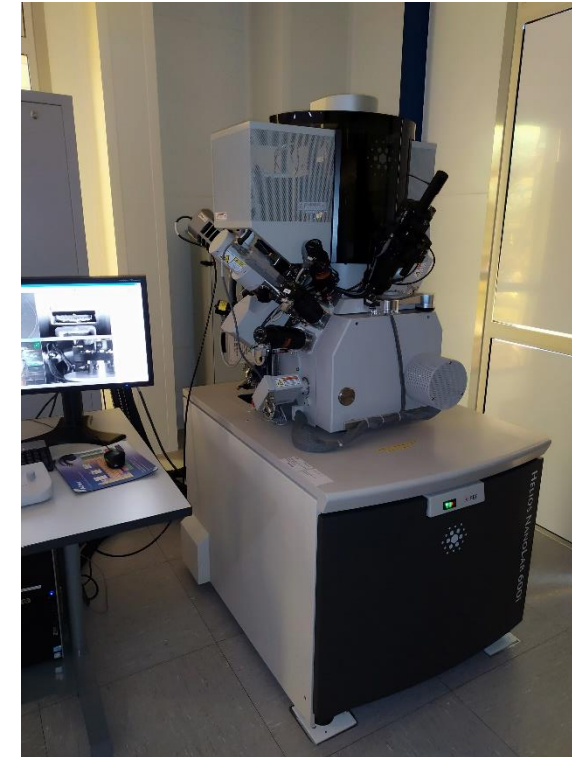
Detection, amplification and signal treatment



Display

Principle: resolution driving parameters





- 3 different SEM with 3 different gun types and 3 different resolutions ...
- How to choose one for your characterization ?
- What do you want to characterize ? Do you need high resolution ?

- Electrostatic lenses
- 3 electrodes (minimum):
 - Filament or tip (attached to a filament)
 - Extractor: extract electrons from the filament
 - Anode: fix the energy of electron beam

2 families of electron guns:

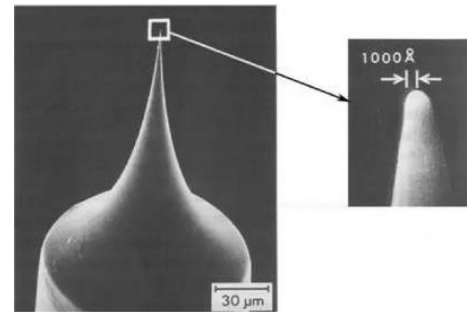
- Thermo-electronic:

- Electrons extracted from an heated filament (extraction: $\sim 500V$)
- 2 subfamilies:
 - W filament
 - LaB₆, CeB₆ crystal soldered on W wire

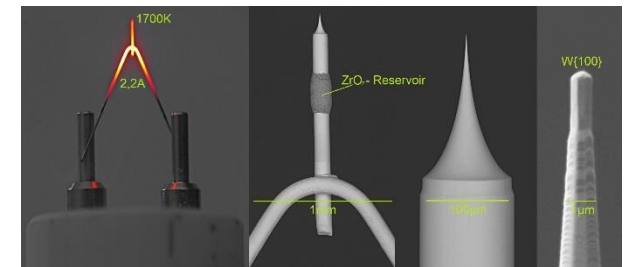


- Field effect guns (FEG):


- Electrons are extracted from a sharp tip by a high electric field (extraction: $\sim 5kV$)
- 3 subfamilies:
 - Cold FEG
 - Thermal FEG
 - Schottky FEG



Cold FEG tip

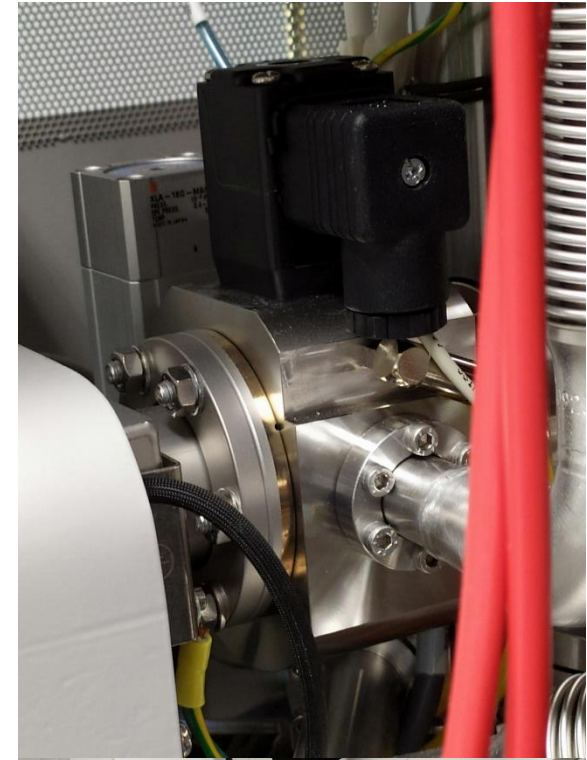


Wikipedia Schottky FEG tip - Erwin Meier

- Thermo-electronic guns:
 - W filament:
 - $<10^{-1}$ Pa ... easily achieved by a Turbo Molecular Pump (TMP) + Dry Pump (DP)
 - Filament lifetime: few hundred hours
 - LaB₆, CeB₆ crystal:
 - \leq few 10^{-5} Pa ... achieved by TMP + DP + ionic pump
 - Filament lifetime: few thousands hours
- Field effect guns (FEG):
 - Cold FEG:
 - $\leq 10^{-8}$ Pa - TMP + DP + baking out + ionic pumps
 - Tip lifetime ~ 15 years (emission unstable: depends on surface contamination)
 - Schottky FEG:
 - $\leq 10^{-7}$ Pa - TMP + DP + baking out + ionic pumps
 - Tip lifetime: few thousands hours
 - Tip heated at $\sim 1700^{\circ}\text{C}$  1 supplementary electrode to repel thermo-electrons

Look at vacuum pipes connections :

- 4 screws = vacuum $> 10^{-5}$ Pa = elastomer o-ring: cannot be baked out
- > 4 screws = vacuum $< 10^{-5}$ Pa = high vacuum = metal o-ring: compatible with baking out

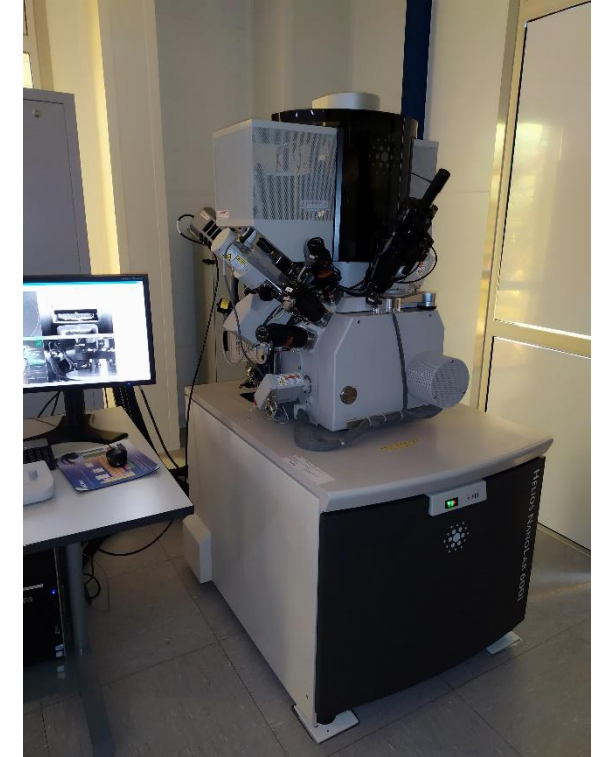




W filament SEM
Hitachi S-3700 N
Low vacuum



FEG SEM
Hitachi S-4800
C-FEG



FEG SEM (+FIB)
ThermoFisher HELIOS 600i
S-FEG



Ionic pump

Gun Type ?

LaB₆ filament SEM - Jeol 840 A

- Lenses: electromagnets or electrostatic lenses (e.g.: gun)
- Definitions:
 - Optical axis: neutral point for electrons # no deviation
 - Paraxial approximation: small angles, close to optical axis
- Circular symmetry defects induce aberrations:
 - Due to mechanical manufacturing
 - ☞ Make aberrations depending on lens parameters
- **Adjusting any parameter on any lens:**
(Electrons' energy, filament T° , extracting voltage, beam current, working distance)
 - Modifies optical axis position (XY image position), focus/astigmatism
 - Induces the need of re-alignments and re-adjustment of focus/astigmatism lenses

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- **Scattering of electrons from the beam**
 - ☞ Backscattered electrons (5-50%) - $E \geq 50\text{eV}$
 - ☞ *Transmitted electrons (if sample is sufficiently thin)*
- **Energy transfer to matter**
 - ☞ **Atoms relaxation = emission of particles**
 - **Secondary electrons (~50-80%):**
 - $E \leq 50\text{eV}$
 - ☞ Mean free path few nanometers
 - ☞ Close to the scattered electrons' trajectory
 - *Auger electrons*
 - **Photons : X, UV, visible, IR**
 - *Induced current*
 - ...
- **From one point to another, all events are independent**

- **Backscattered electrons** (*belong to primary beam*)
- **Monte-Carlo simulation :**
 - **Elastic diffusion on atoms**
(*Rutherford backscattering: Z^2/E^2*)
 - **Energy loss between 2 elastic diffusion**
(*Bethe slowing down law: Z/E*)

- 5-50% of all electrons exiting from the sample
- $50\text{eV} \leq E \leq E_{\text{beam}}$
- Sensitive to the local chemical composition
- High kinetic energy
 - High directivity
 - Low sensitivity to topography
 - 45° surface escape half-angle
 - Detector located above the sample: can be broken easily

Detectors: BackScattered Electrons Detector (BSED)

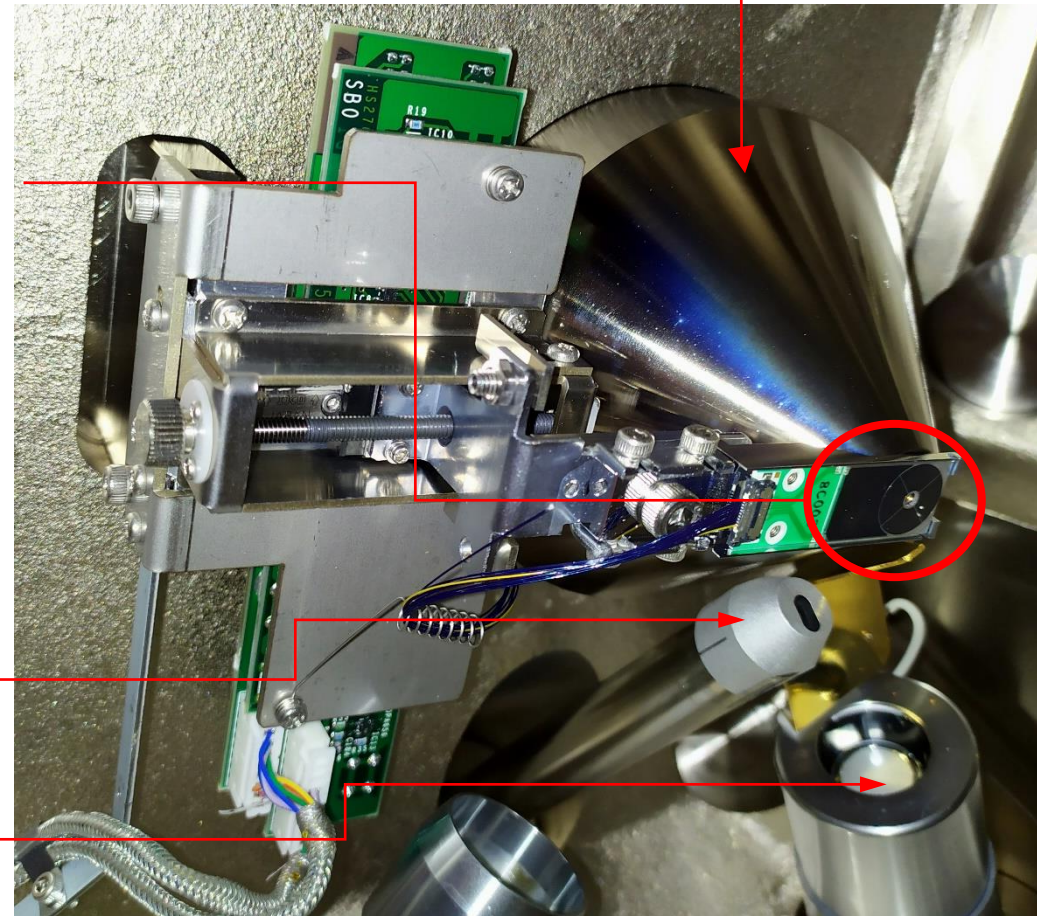
"Objective" lens

BSE Detector

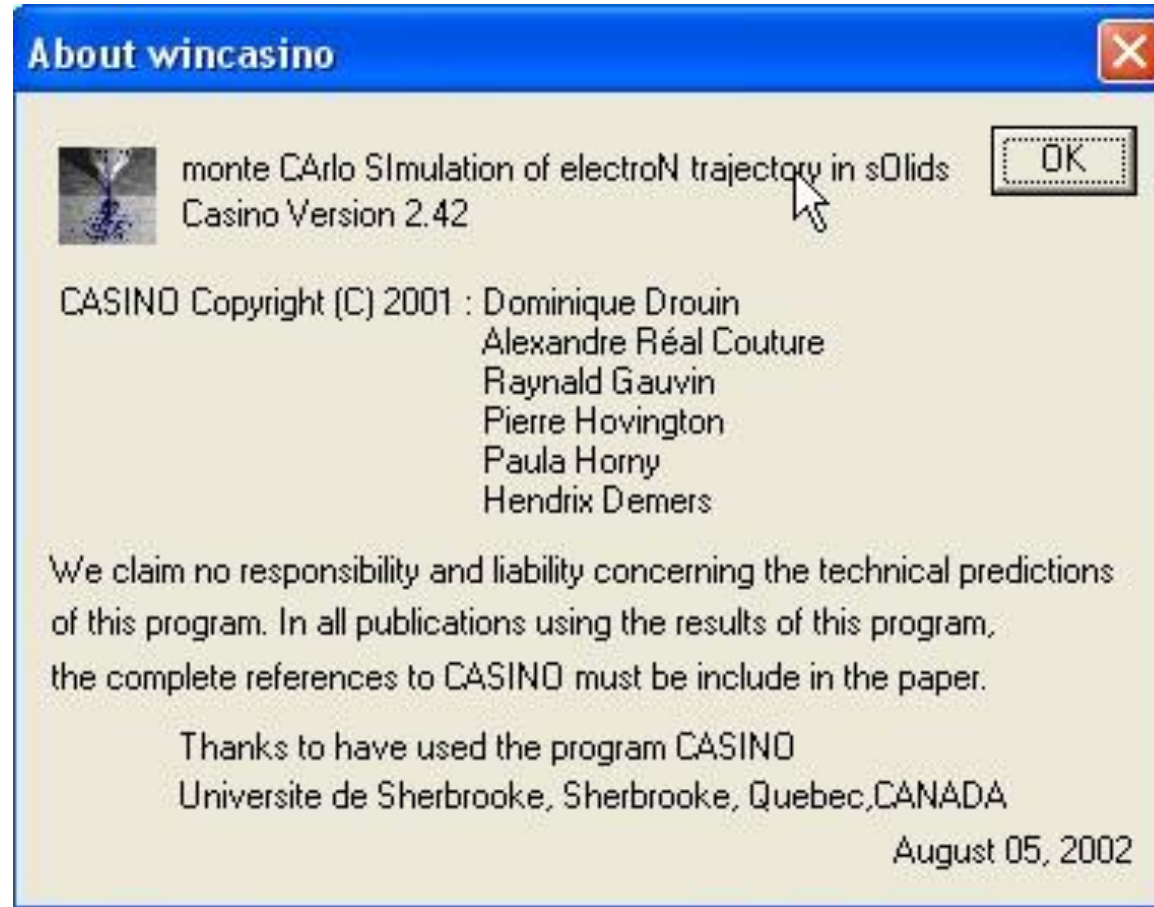
Principle: creation of charges in a polarized diode
Current proportional to the number of BSE produced

X-Rays Detector

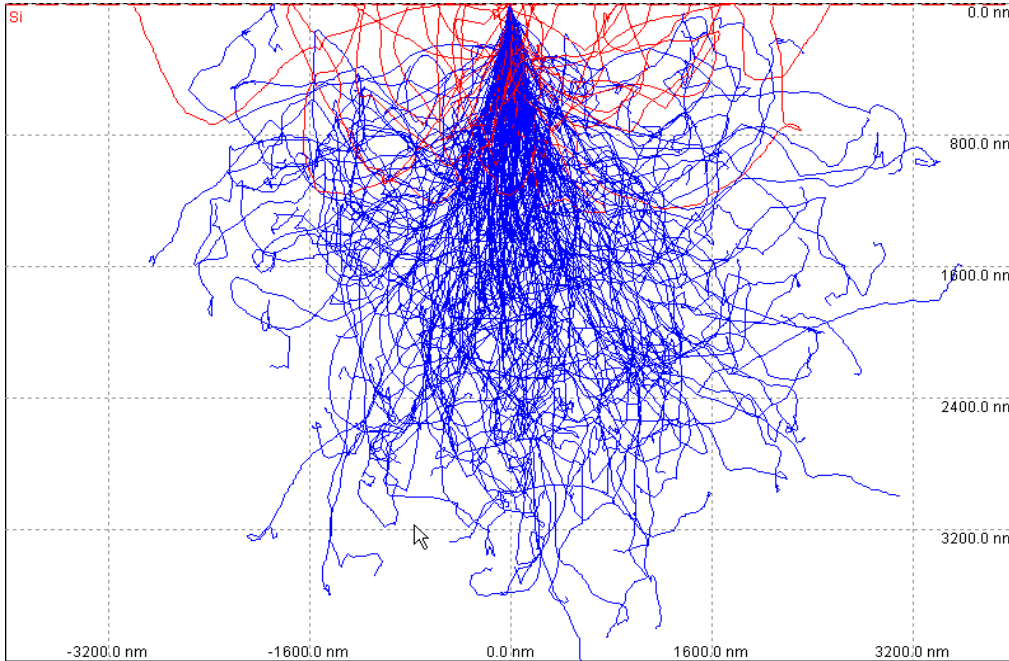
Secondary Electrons Detector



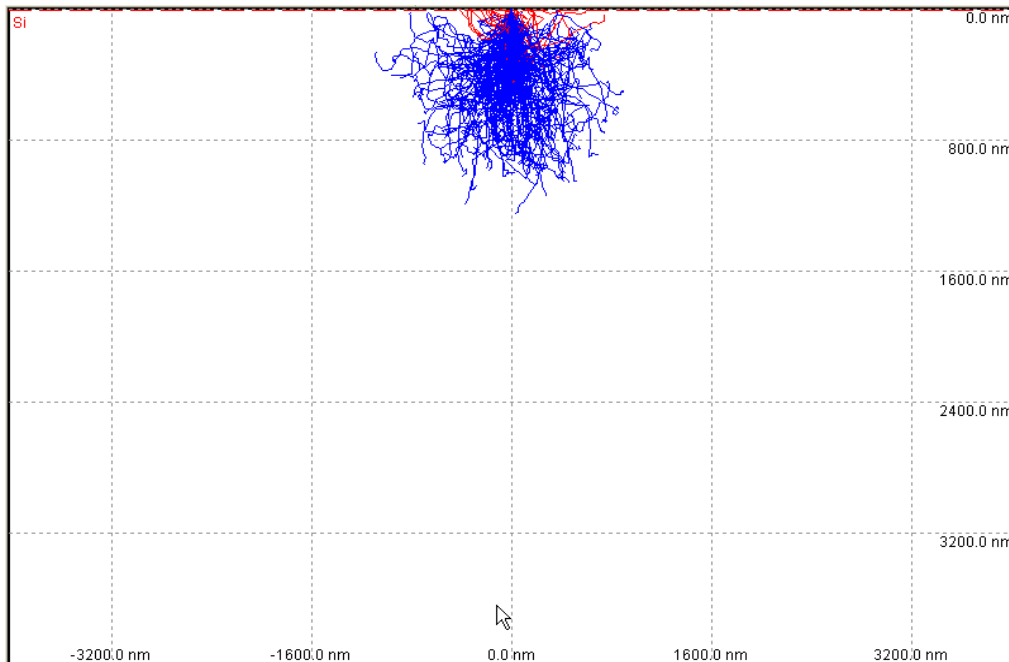
Chamber view from inside



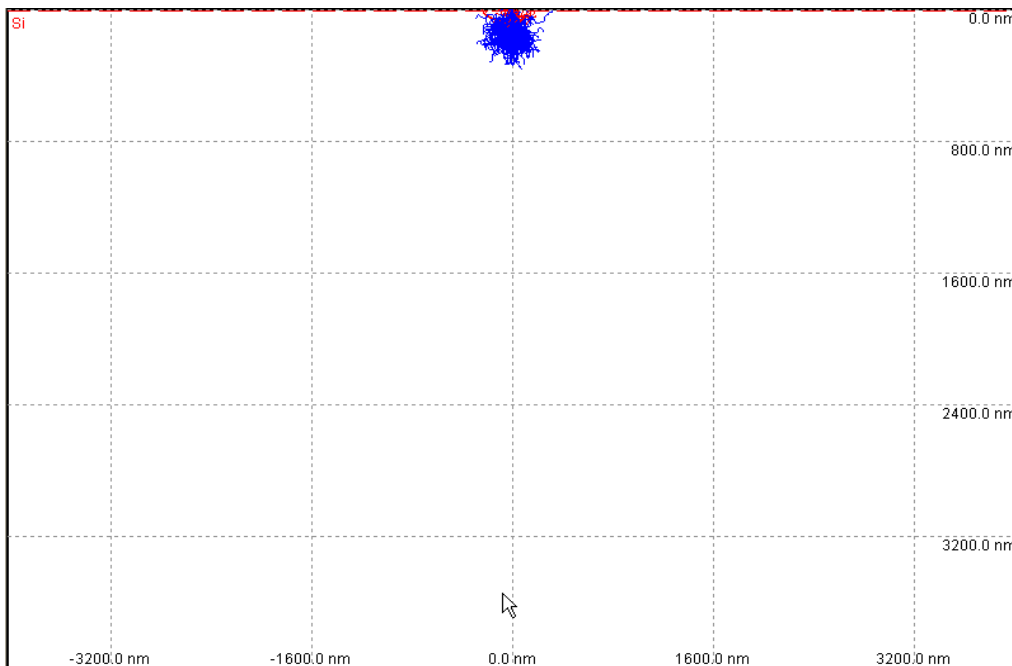
<http://www.gel.usherbrooke.ca/casino/>



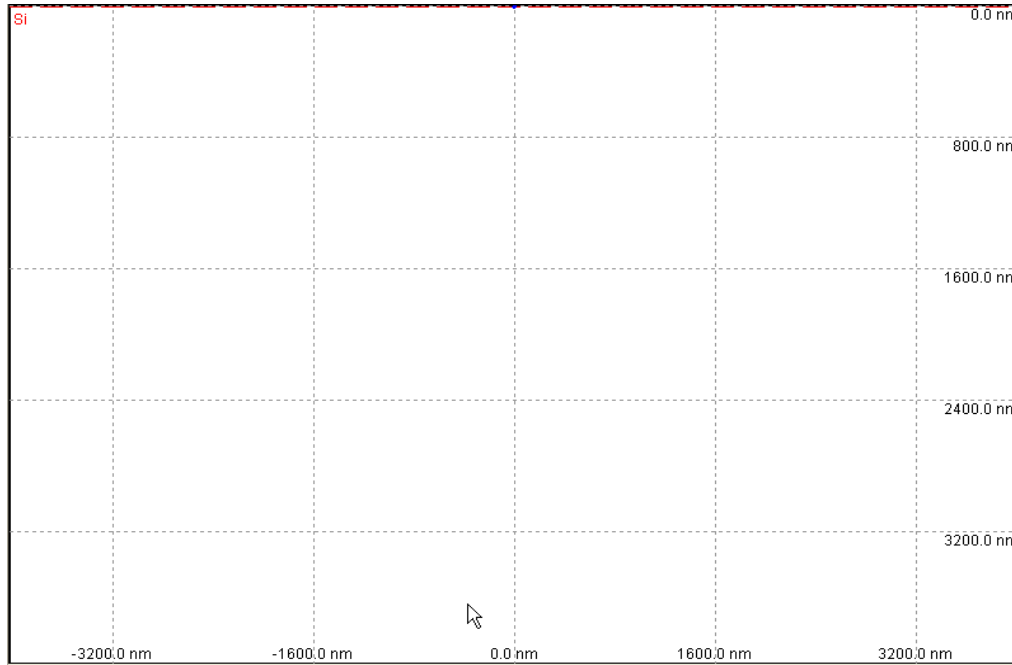
- Substrate: Si
- Beam Energy: 20keV
- Backscattered coefficient: 17.5%
- Interaction volume:
 - Max depth: 4 μ m
 - Max radius: 3 μ m



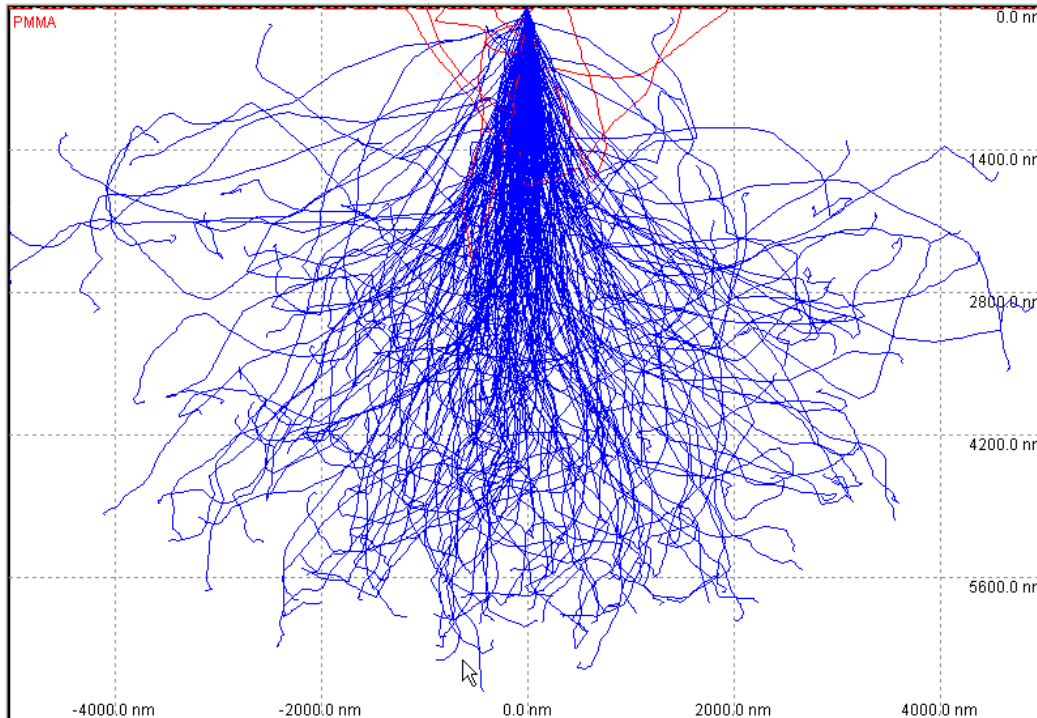
- Substrate: Si
- Beam Energy: 10keV
- Backscattered coefficient: 15.5%
- Interaction volume:
 - Max depth: 1.300μm
 - Max radius: 950nm



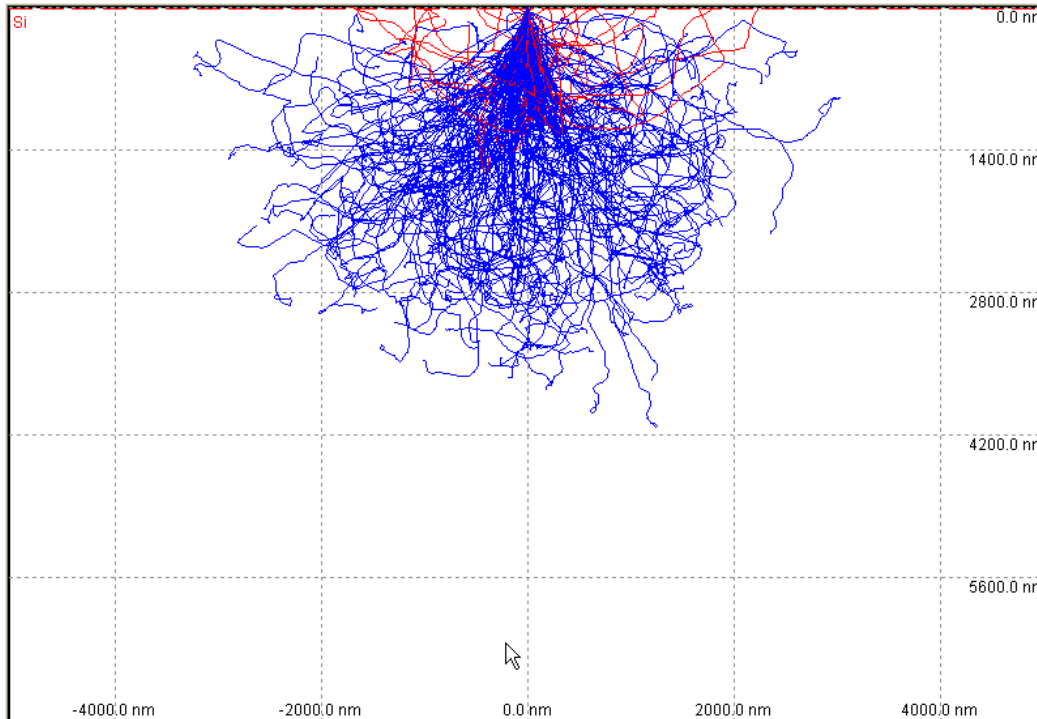
- Substrate: Si
- Beam Energy: 5keV
- Backscattered coefficient: 17%
- Interaction Volume:
 - Max depth: 370nm
 - Max radius: 280nm



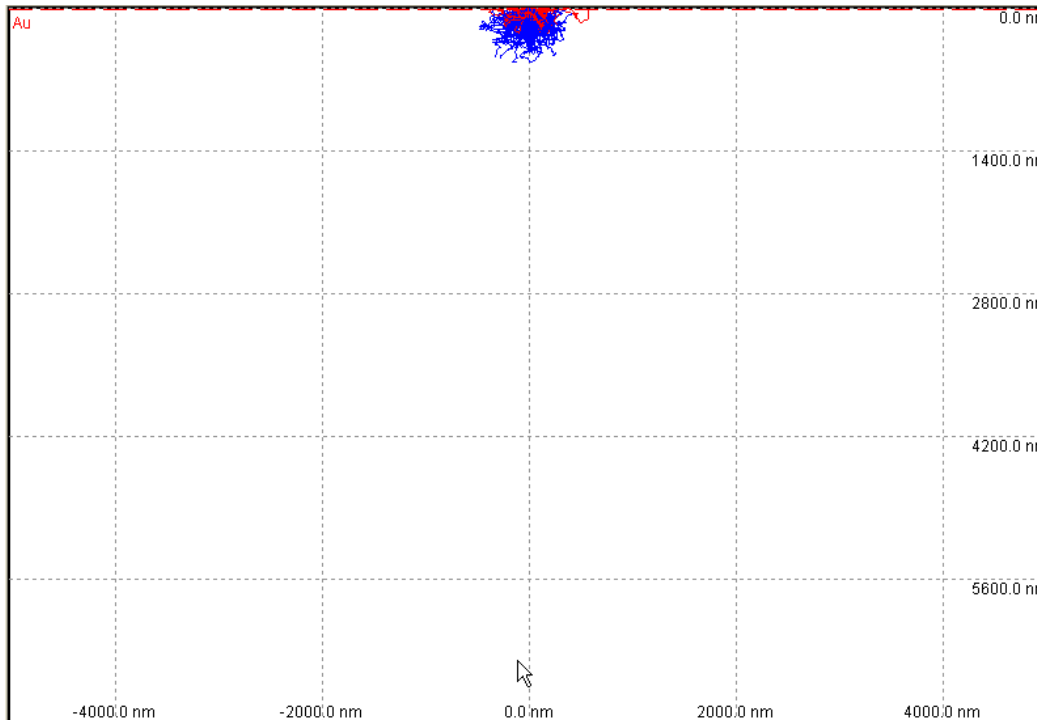
- Substrate: Si
- Beam Energy: 1keV
- Backscattered coefficient: 15.5%
- Interaction Volume:
 - Max depth: 30nm
 - Max radius: 30nm



- Substrate: PMMA ($Z_{av}=3.6$)
- Beam Energy : 20keV
- Backscattered coefficient: 4.5%
- Interaction volume:
 - Max depth: 7 μ m
 - Max radius: 5 μ m

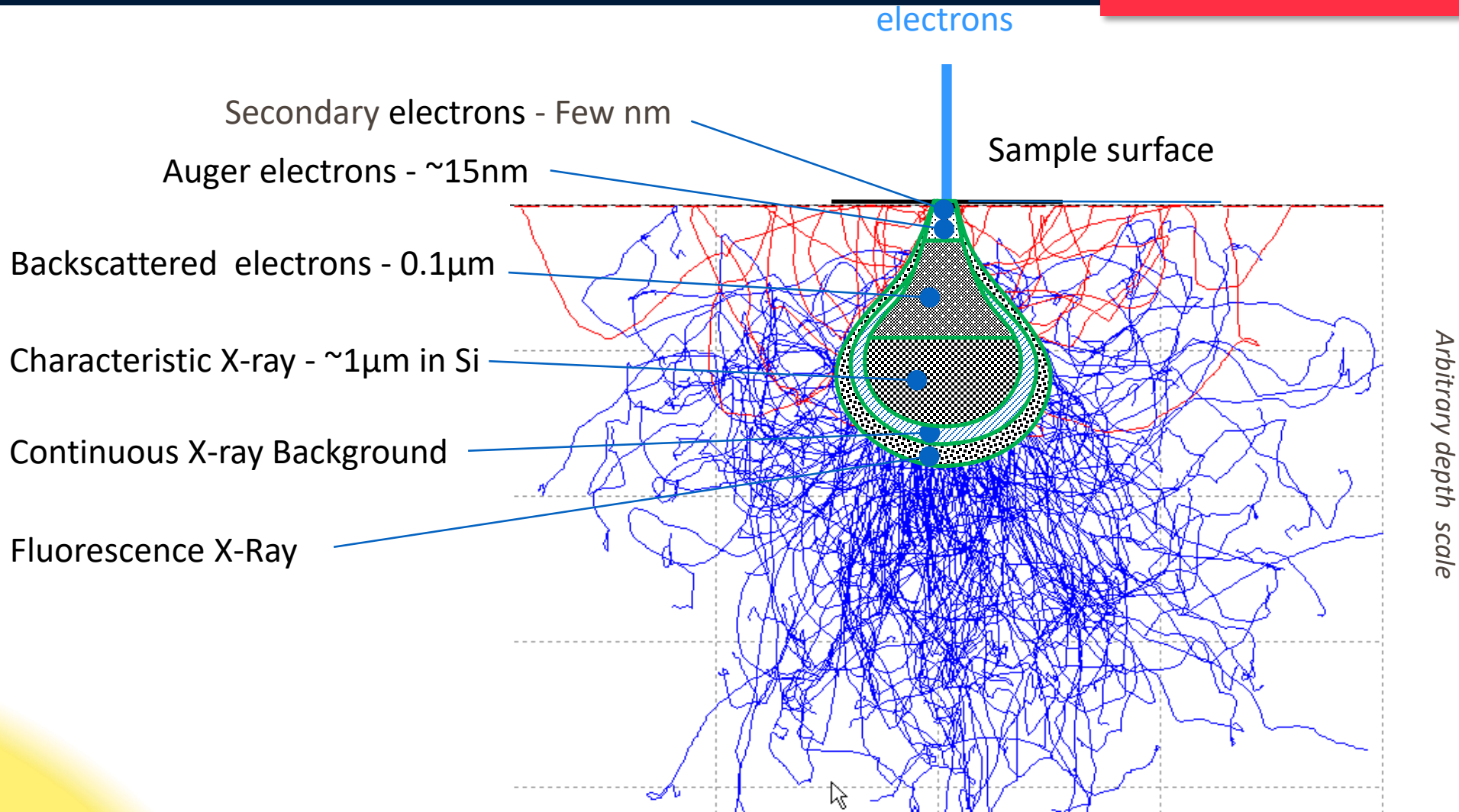


- Substrate: Si (Z=14)
- Beam Energy: 20keV
- Backscattered coefficient: 17.5%
- Interaction volume:
 - Max depth: 4μm
 - Max radius: 3μm



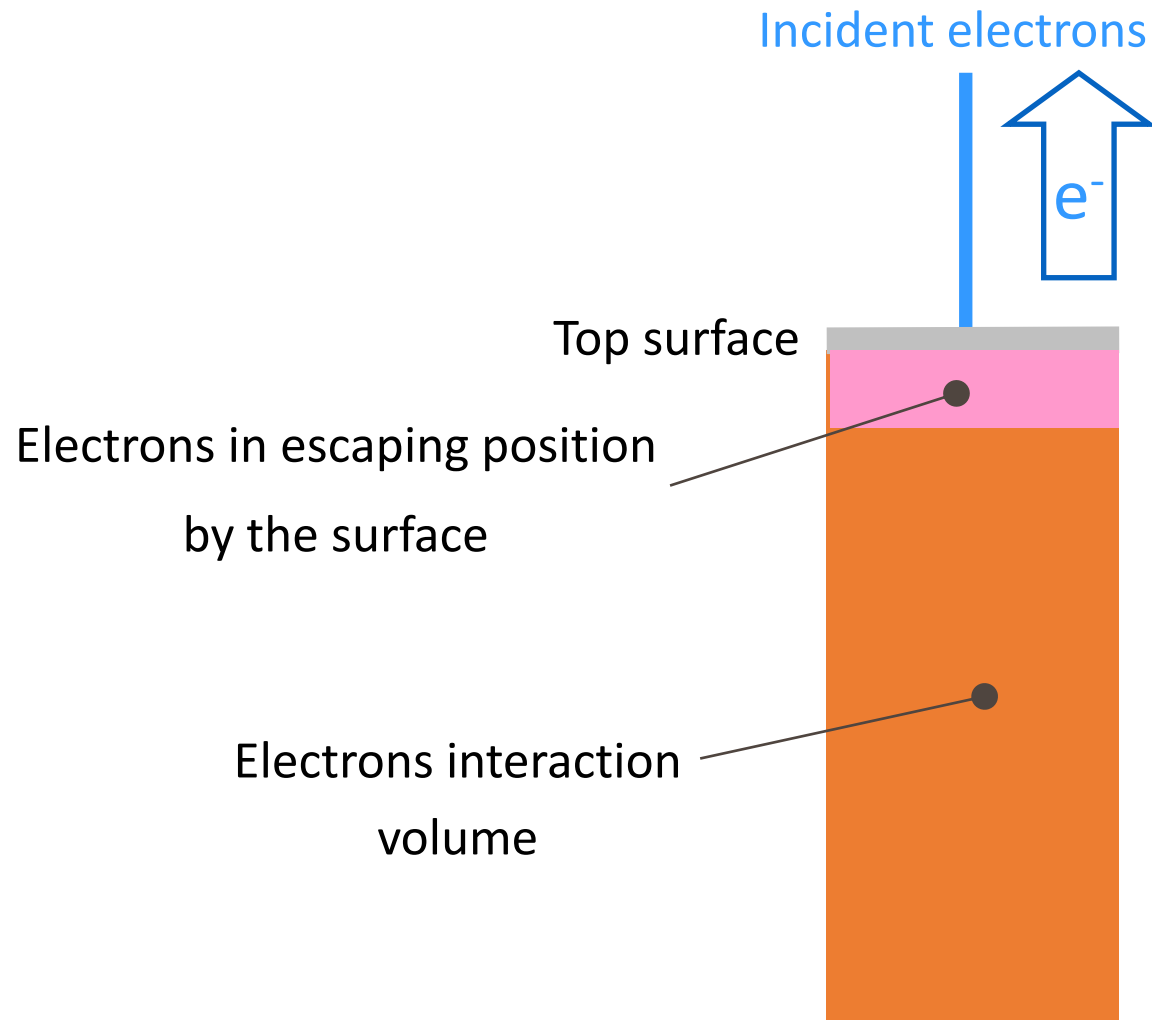
- Substrate: Au (Z=79)
- Beam Energy: 20keV
- Backscattered coefficient: 50%
- Interaction volume:
 - Max depth: 0.3 μ m
 - Max radius: 0.4 μ m

Phenomena involved: max depth of escape

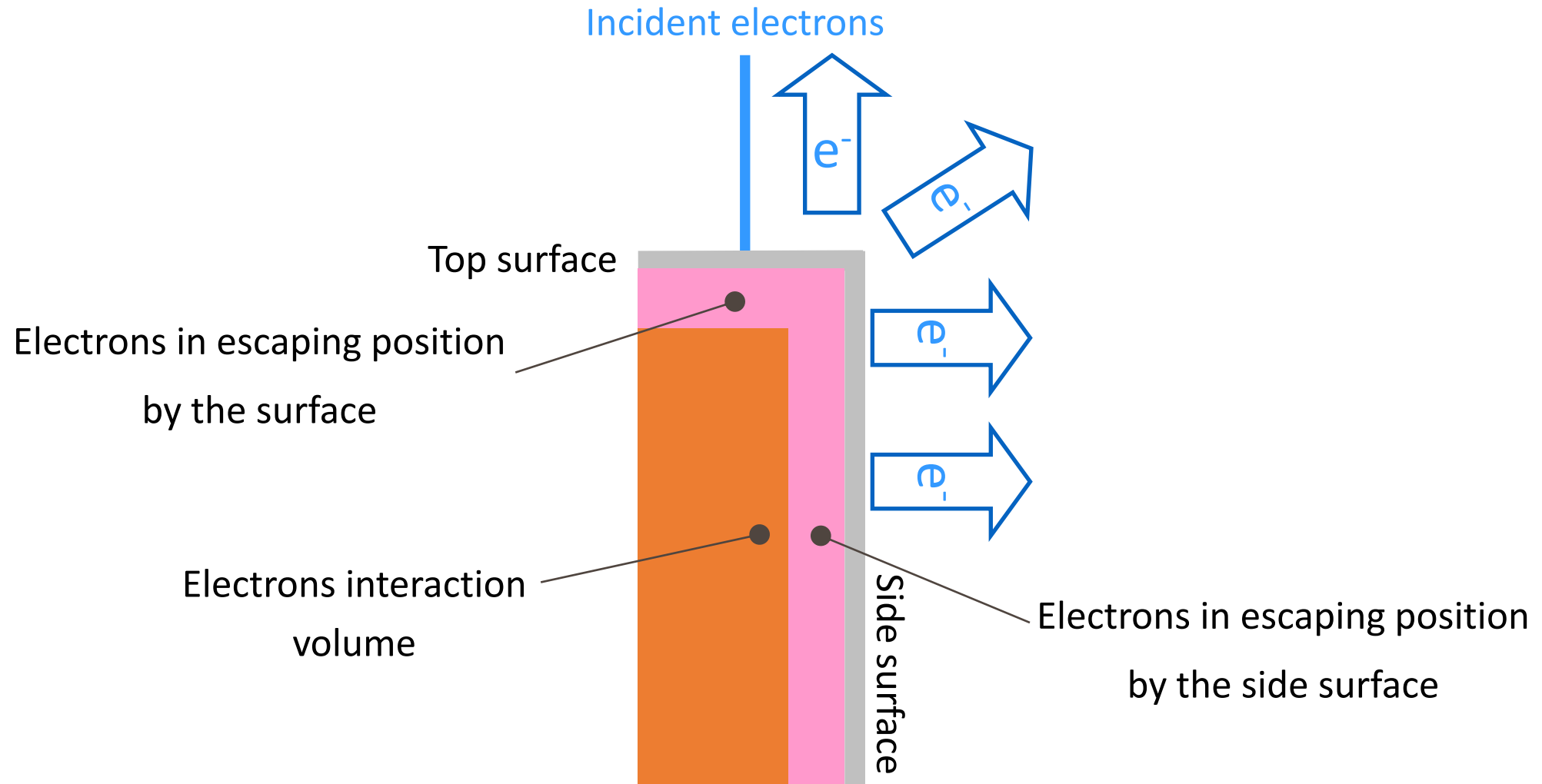


- Emitted by the sample
- Energy < 50eV
- Mean free path: few nm
 - Close to scattered electrons trajectories
 - Emitted from few nanometers near the surface
- Source of contrast:
 - Intensity depends mainly on topography
 - Depends also on:
 - Atomic number
 - Organic contamination of the surface

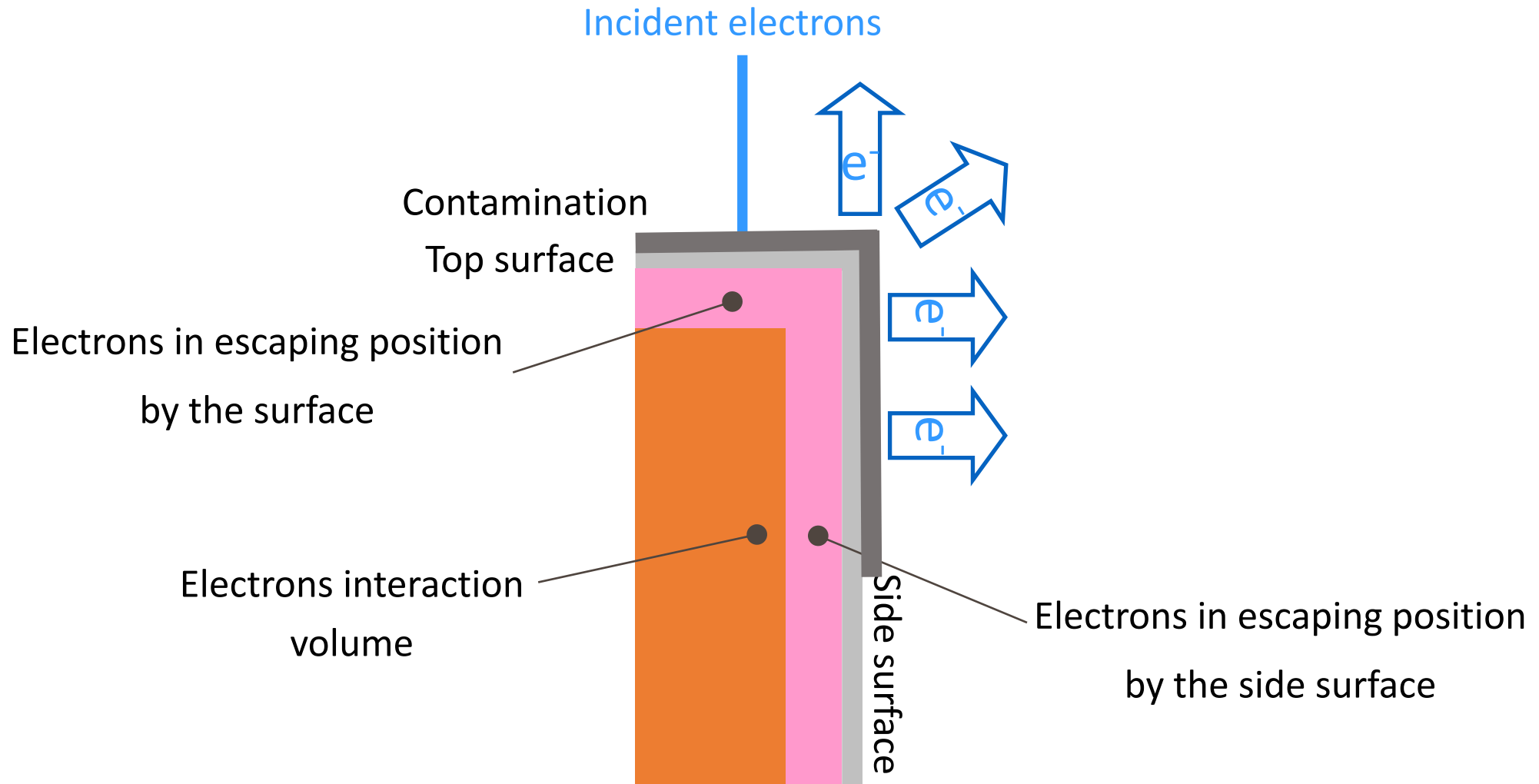
Origin of contrast: topography



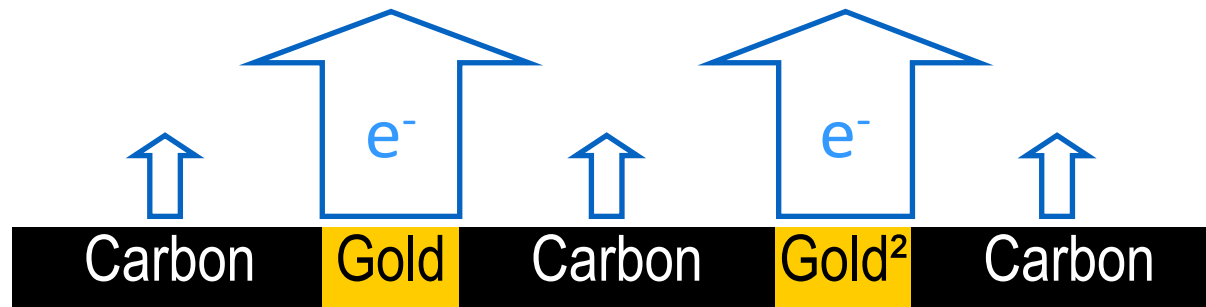
Origin of contrast: topography



Origin of contrast: topography

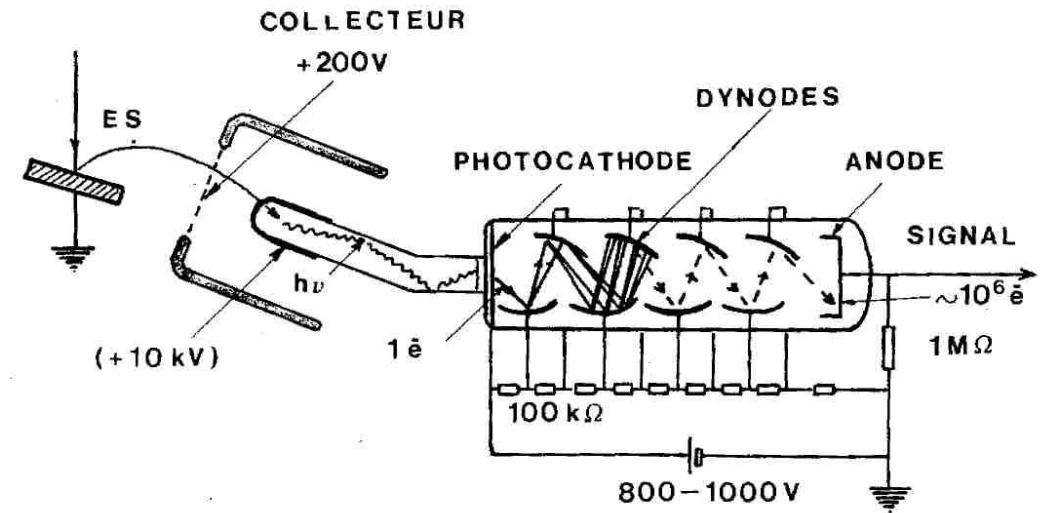


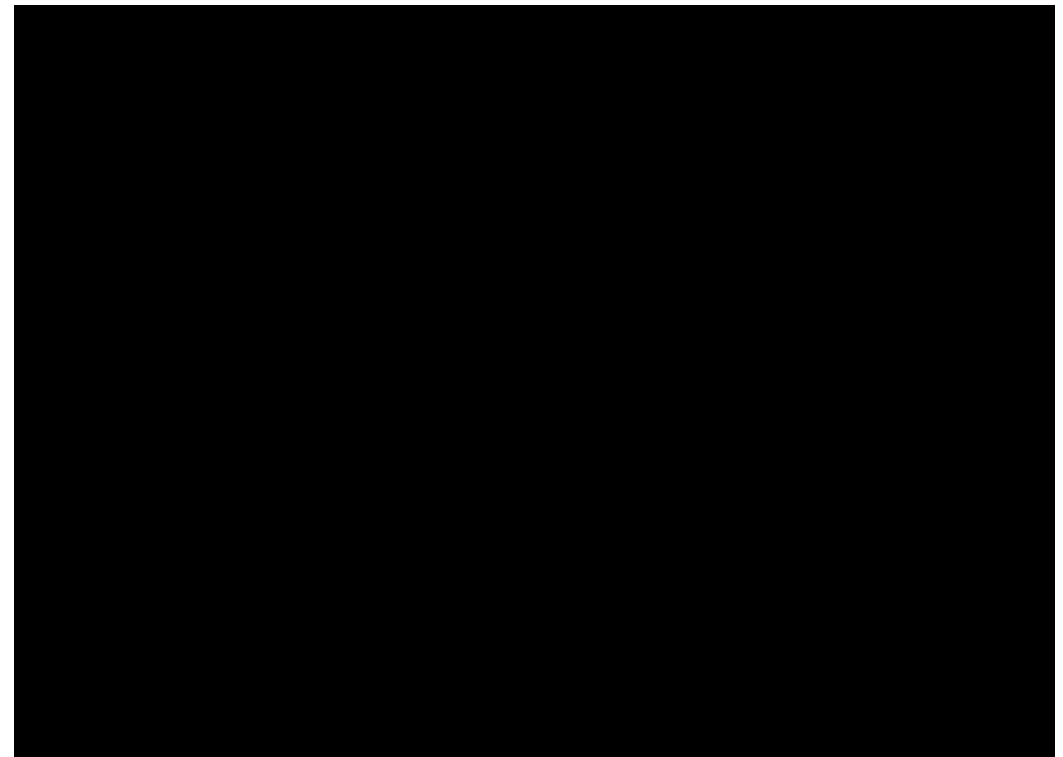
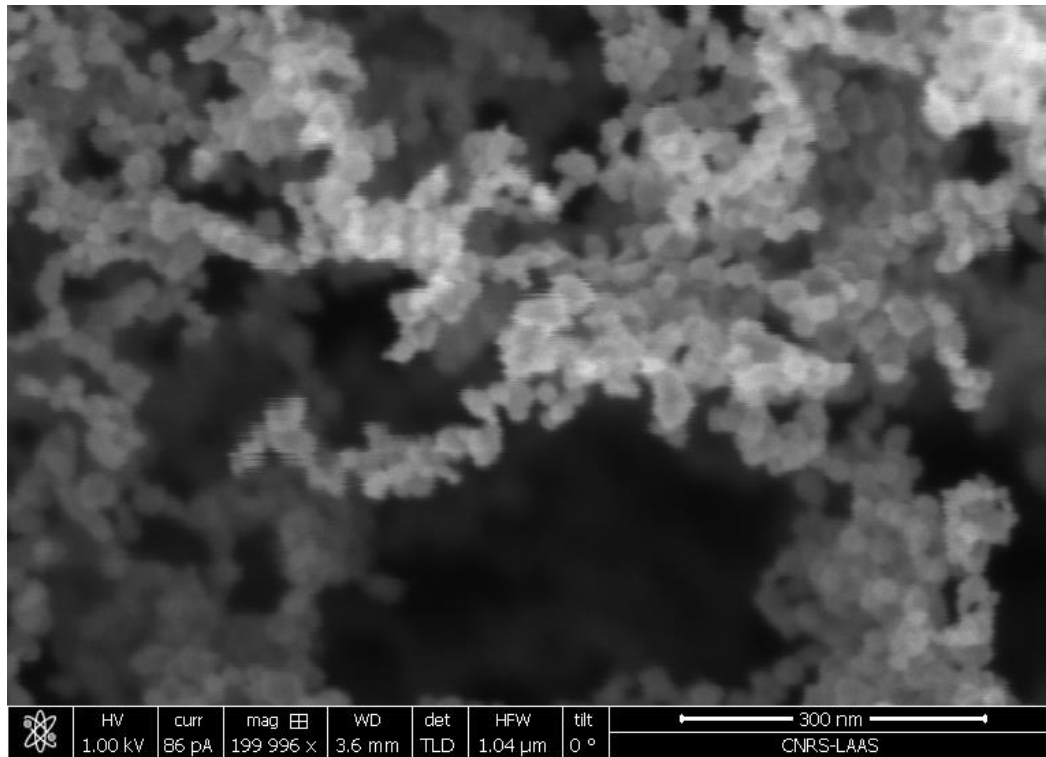
Sample without topography composed of materials with different Z:



- Interaction is different:
 - More backscattering from Au
 - More secondary electrons in escaping position
- Secondary electrons emission intensity is different
- Chemical contrast (without topography)

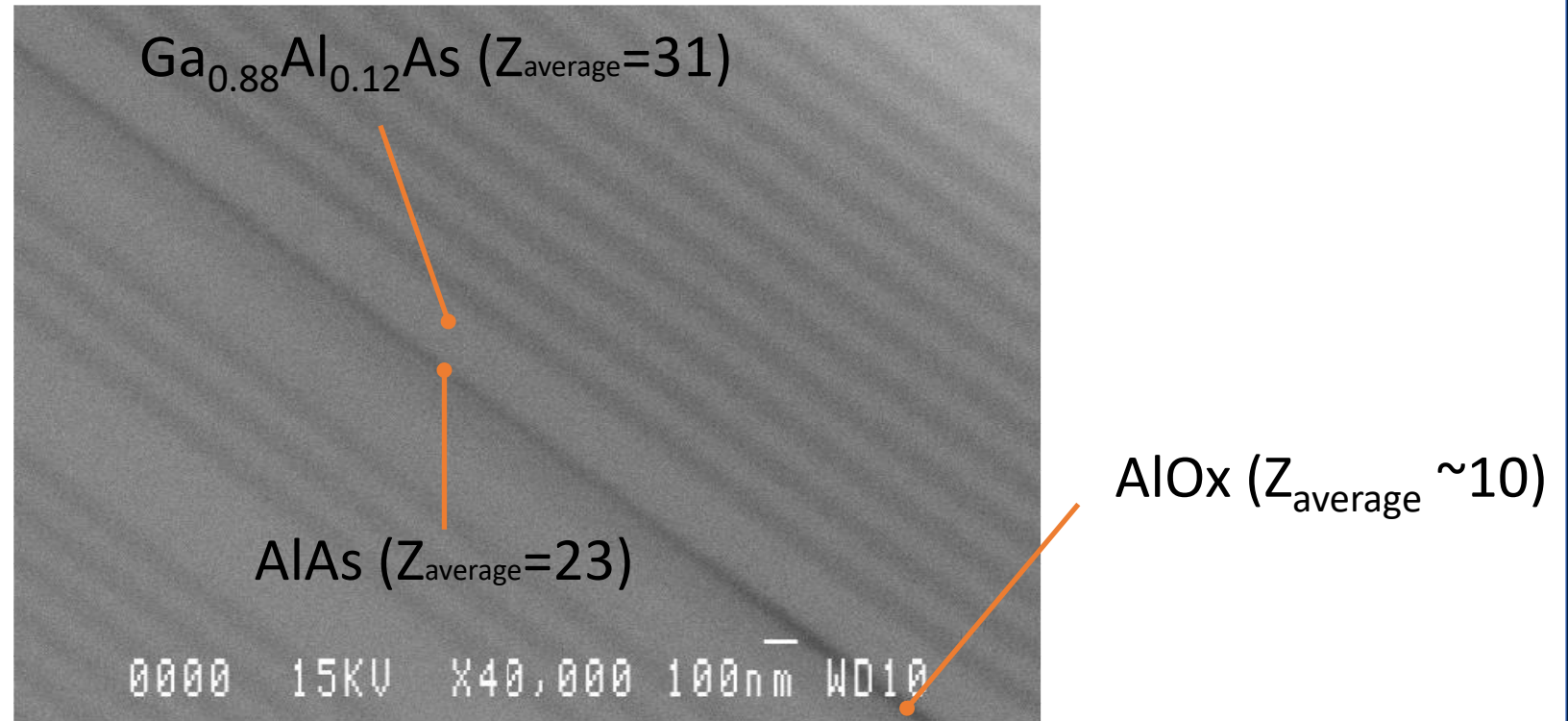
- > Everhart & Thornley Detector (ETD - 1960)
- > If objective lens is close to the sample:
 - ETD collector potential is screened
 - Electrons are trapped between sample and lens
 - ☞ No detection
 - ☞ Use the detector inside the column





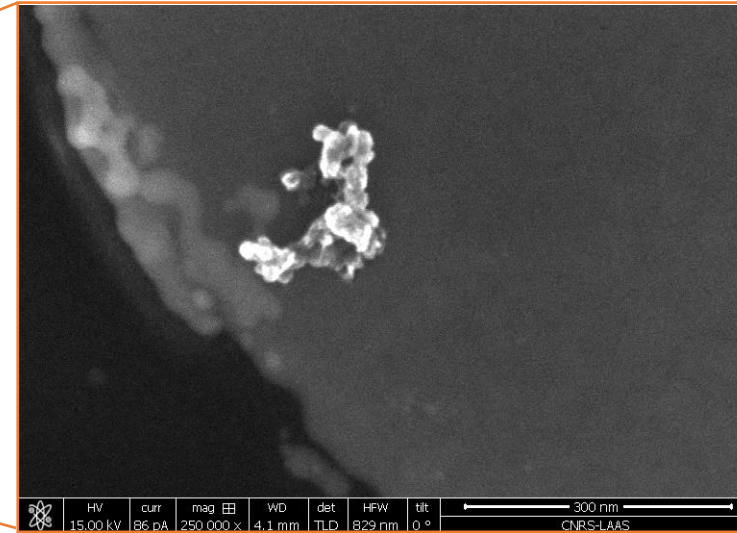
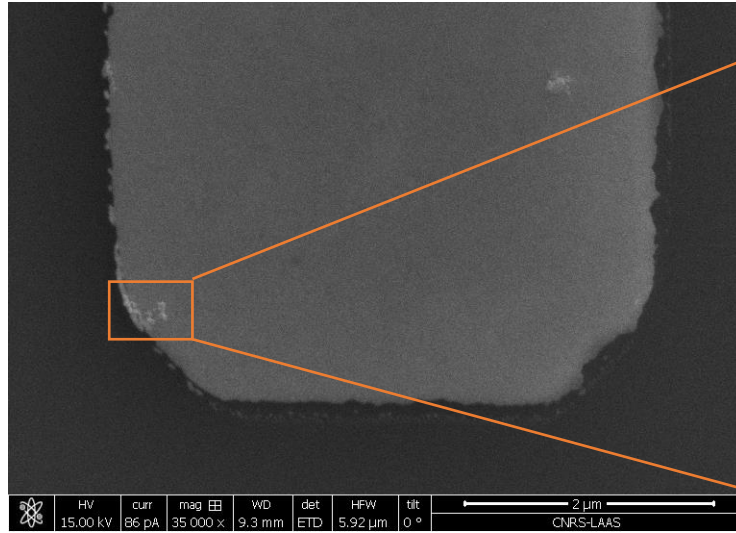
Topographical image example: candle stick fume nanoparticles (carbon)

Origin of contrast: chemical contrast with SED

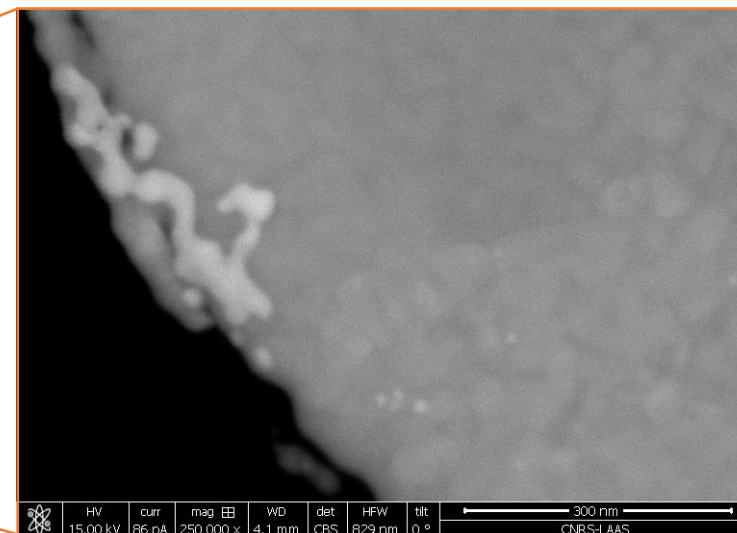
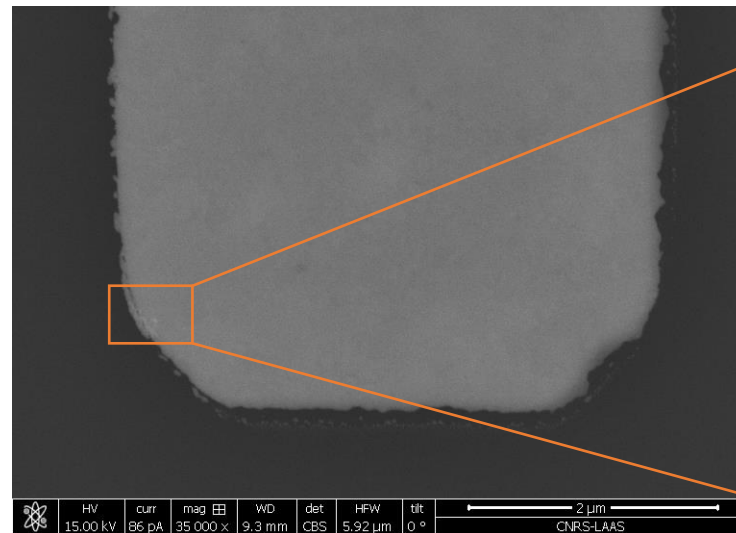


Flat freshly cleaved epitaxial sample without chemical etching

Origin of contrast: BSED vs SED (Au on Si)



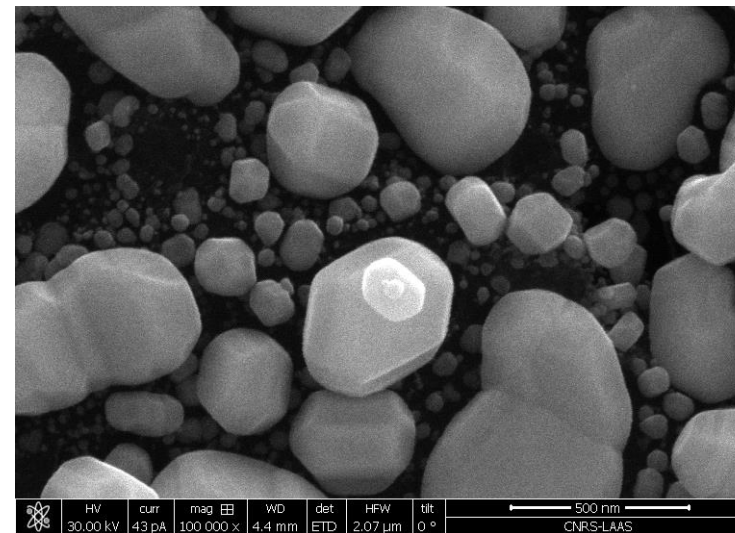
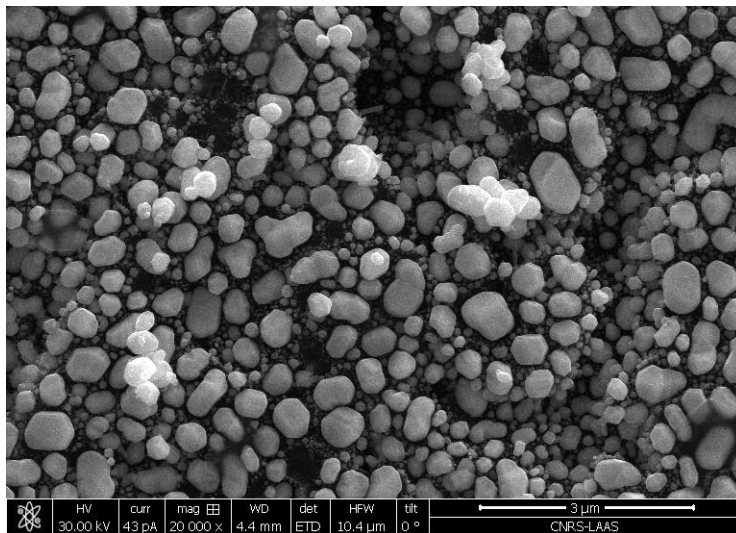
SED



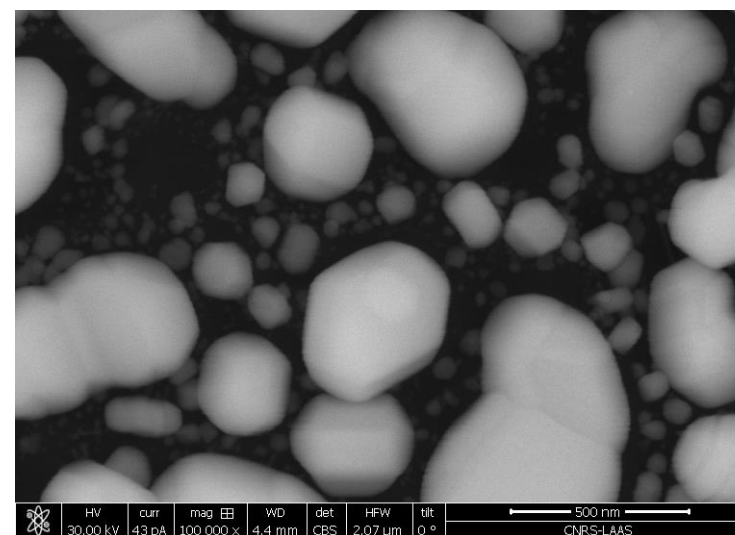
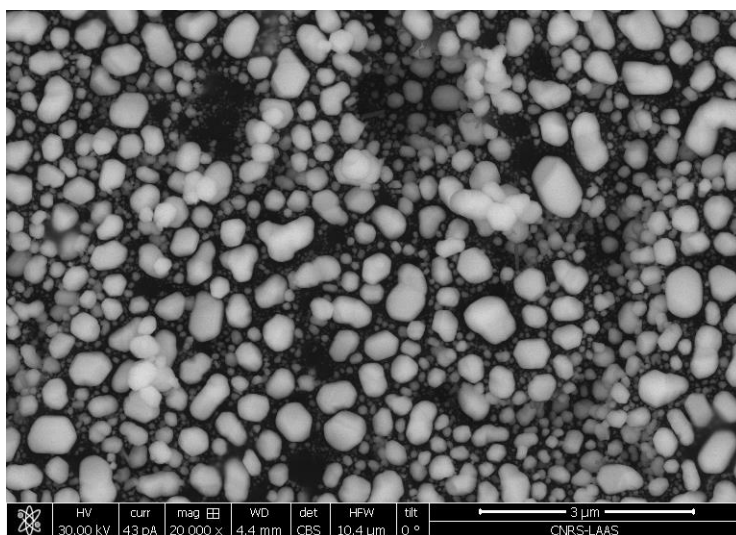
BSED

Origin of contrast: BSED vs SED

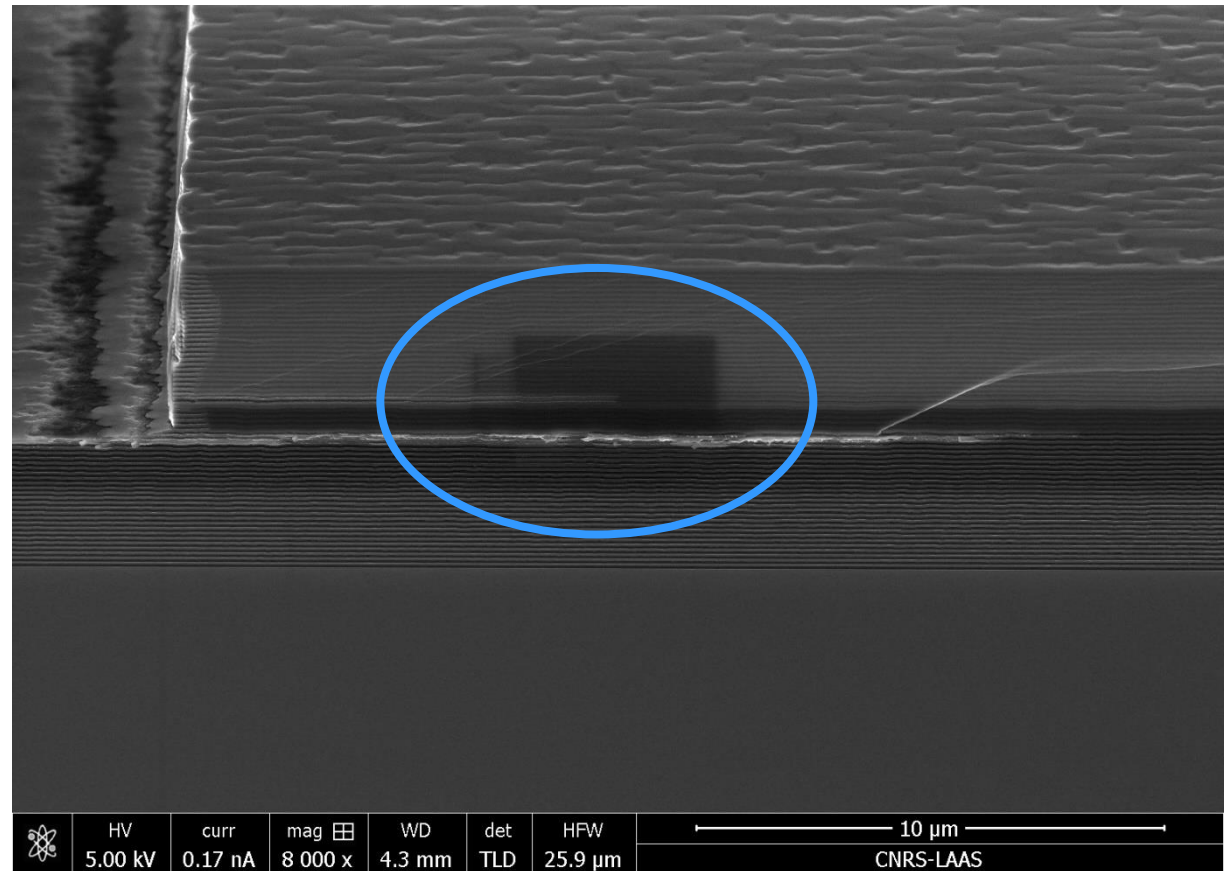
SED



BSED

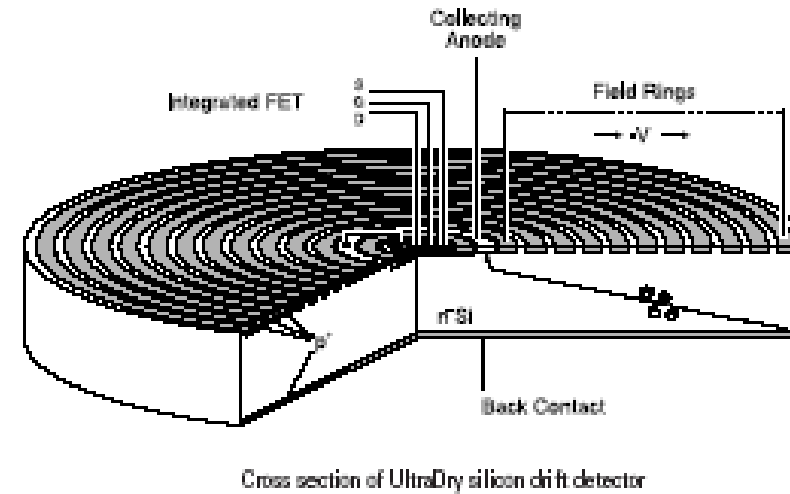


- Surface contaminant cross-linking
 - Coming from the substrate
 - Coming from the vacuum
- Adding a material based on C:
 - Low SE emission
 - Low contrast
 - ☞ Black rectangles



Tilted view of epitaxially grown layers after local etching

Detectors: X-Rays detector

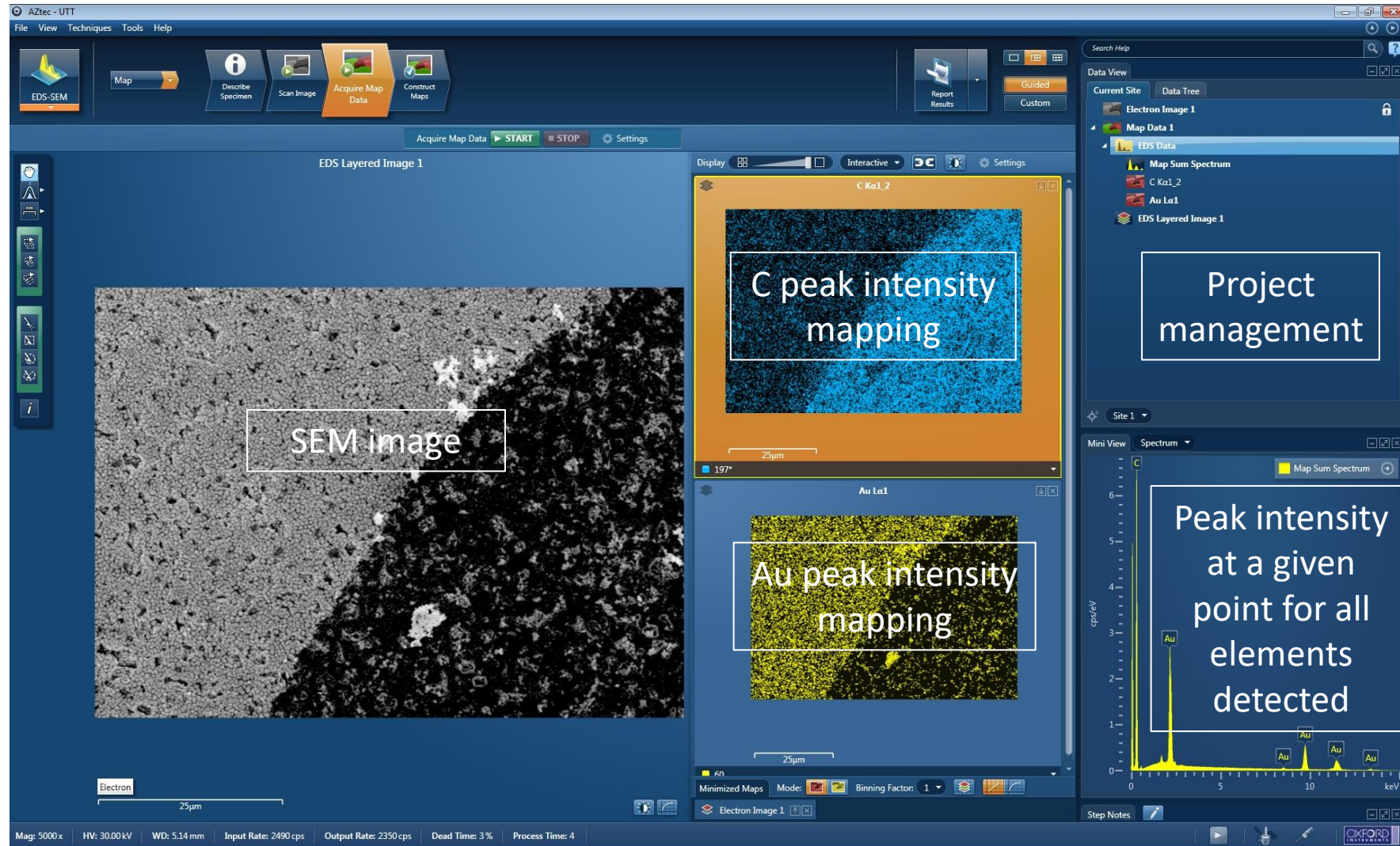


Energy Dispersive X-ray Spectroscopy (EDS or EDX or ...)

- High energy electron beam: $\geq 10\text{keV}$
 - Max. depth penetration > X-Rays max. depth of escape
 - Resolution depend on X-Rays max. depth of escape ($\sim 1\mu\text{m}$ for Si)
 - Not a surface analysis
- Elementary analysis:
 - Resolution: $\sim 125\text{eV}$ (50 - 130 eV for the Mn $K\alpha$ line (5.89 keV))
- Point analysis
- Mapping
 - Qualitative
 - Quantitative:
 - 1% precision
 - Calibration \rightarrow full-time job

- Low energy X-rays detection
- High sensitivity for light elements: Li, Be, B, C, O, N
- High Resolution SEM:
 - Low energy electron beam: $\leq 3\text{keV}$
 - Interaction volume < escape volume: $\sim 10\text{nm}$
 - Spatial resolution depend on interaction volume: $\sim 10\text{nm}$
- Surface analysis with high resolution

Detectors: standard X-Rays detector



Electron/Matter interactions

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1. Choices :

- SEM type
- High voltage
- Beam current
- Object orientation
- Working distance

2. Adjustments :

- Alignments of all elements on optical axis: lenses, apertures
- Focus
- Astigmatism
- Layout

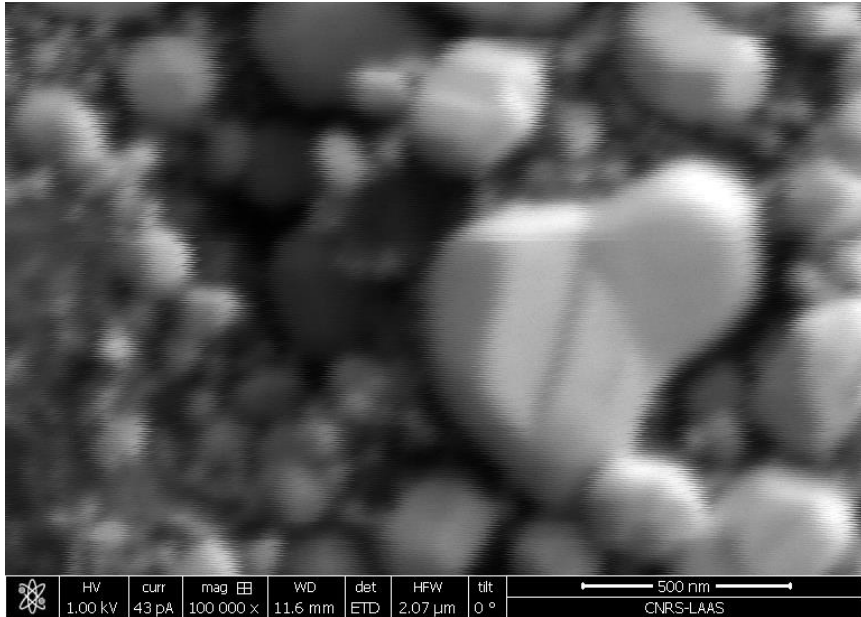
... It's simple !

Influence of choices: summarized effects

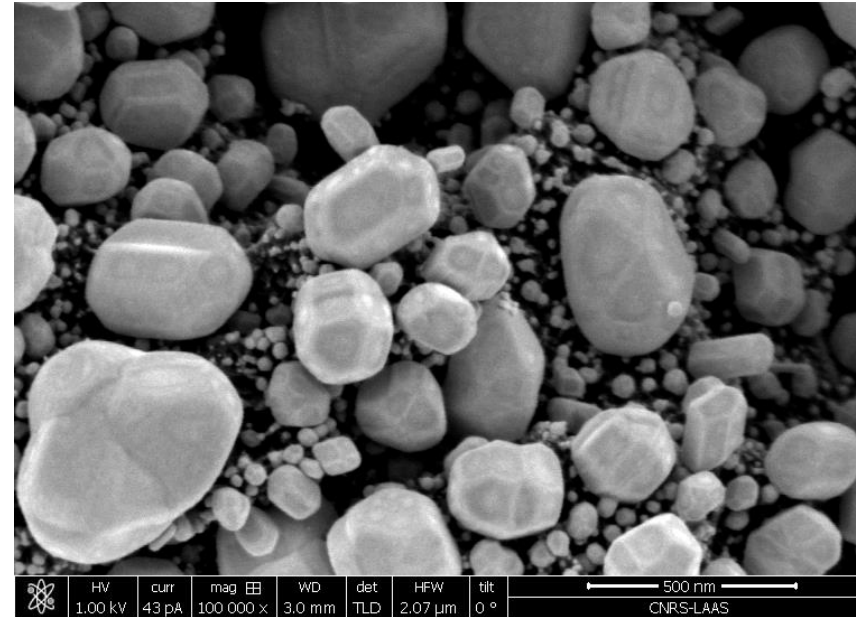
Working distance ↘	Voltage ↗	Current ↗	Bad alignment
Resolution ↗↗↗ Depth of focus ↘	Resolution ↗↗ Tip effect ↗ Charges ↗↗ Damages ↗↗ Surface details ↘	Resolution ↘↘ Charges ↗↗ Damages ↗↗ Noise ↘	Resolution ↘↘ Noise ↗

Once parameters are chosen, the main resolution improvement is achieved by decreasing WD

Influence of choices: Working Distance



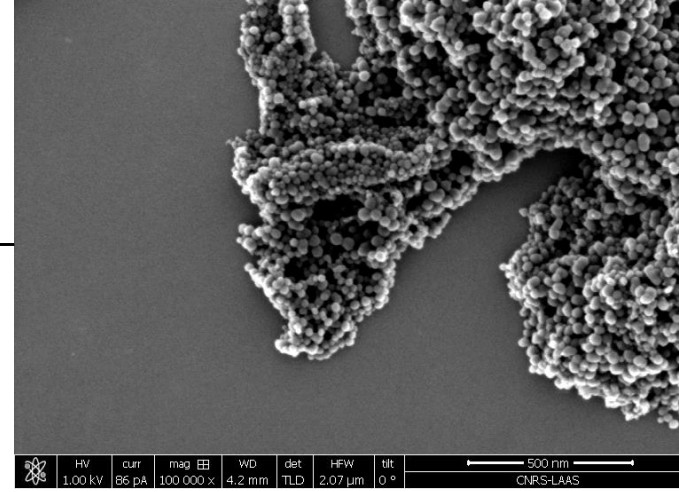
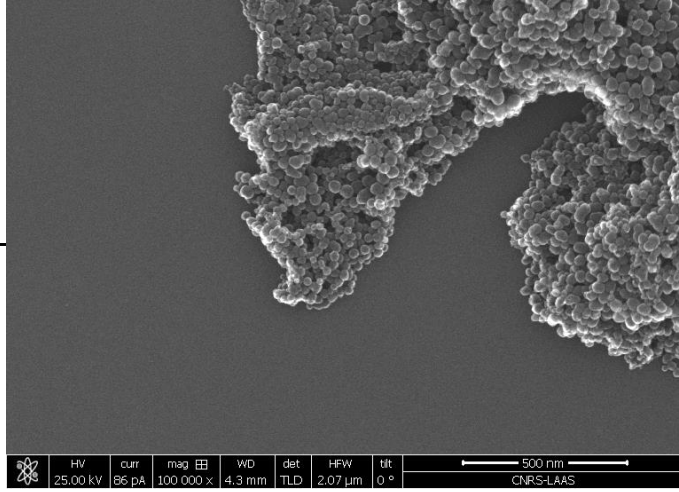
Long working distance



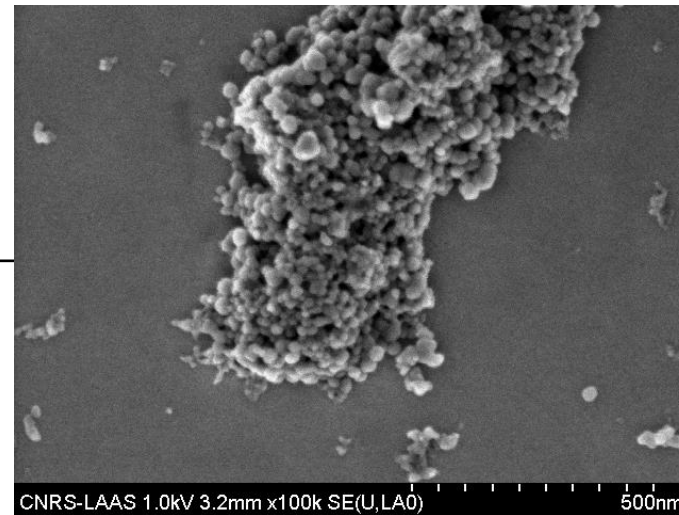
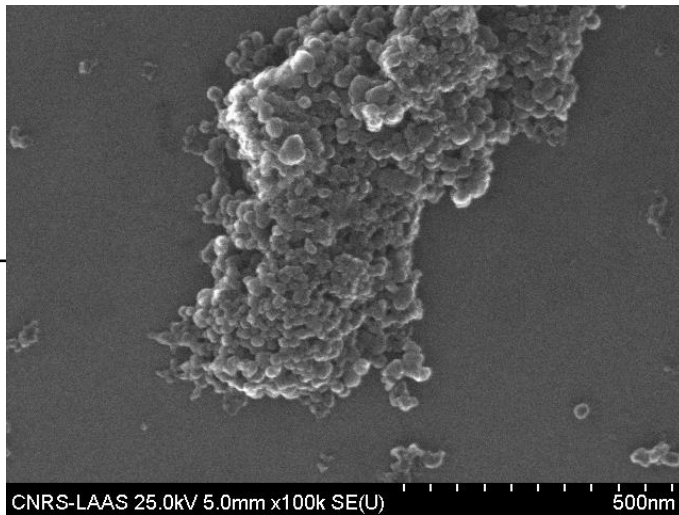
Short working distance
High resolution

Influence of choices: High Voltage

S-FEG



C-FEG



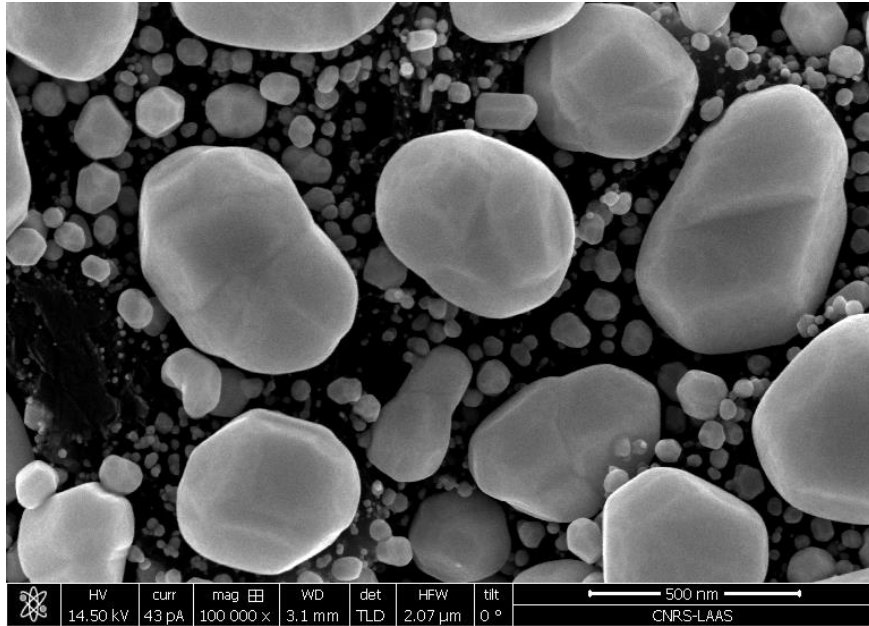
High accelerating voltage

Low accelerating voltage

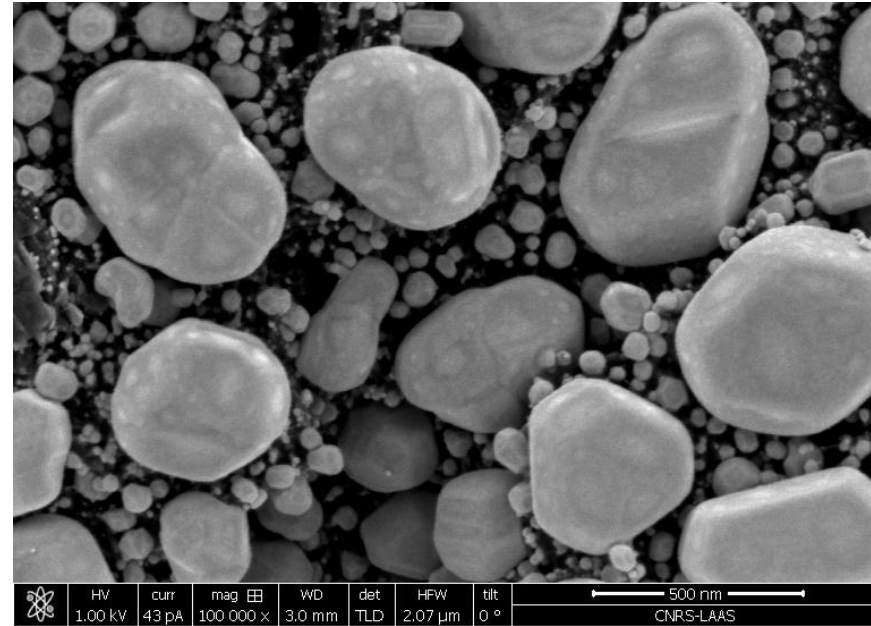
Candle stick fumes:

- Same sample
- ~ same parameters

Influence of choices: High Voltage



High accelerating voltage



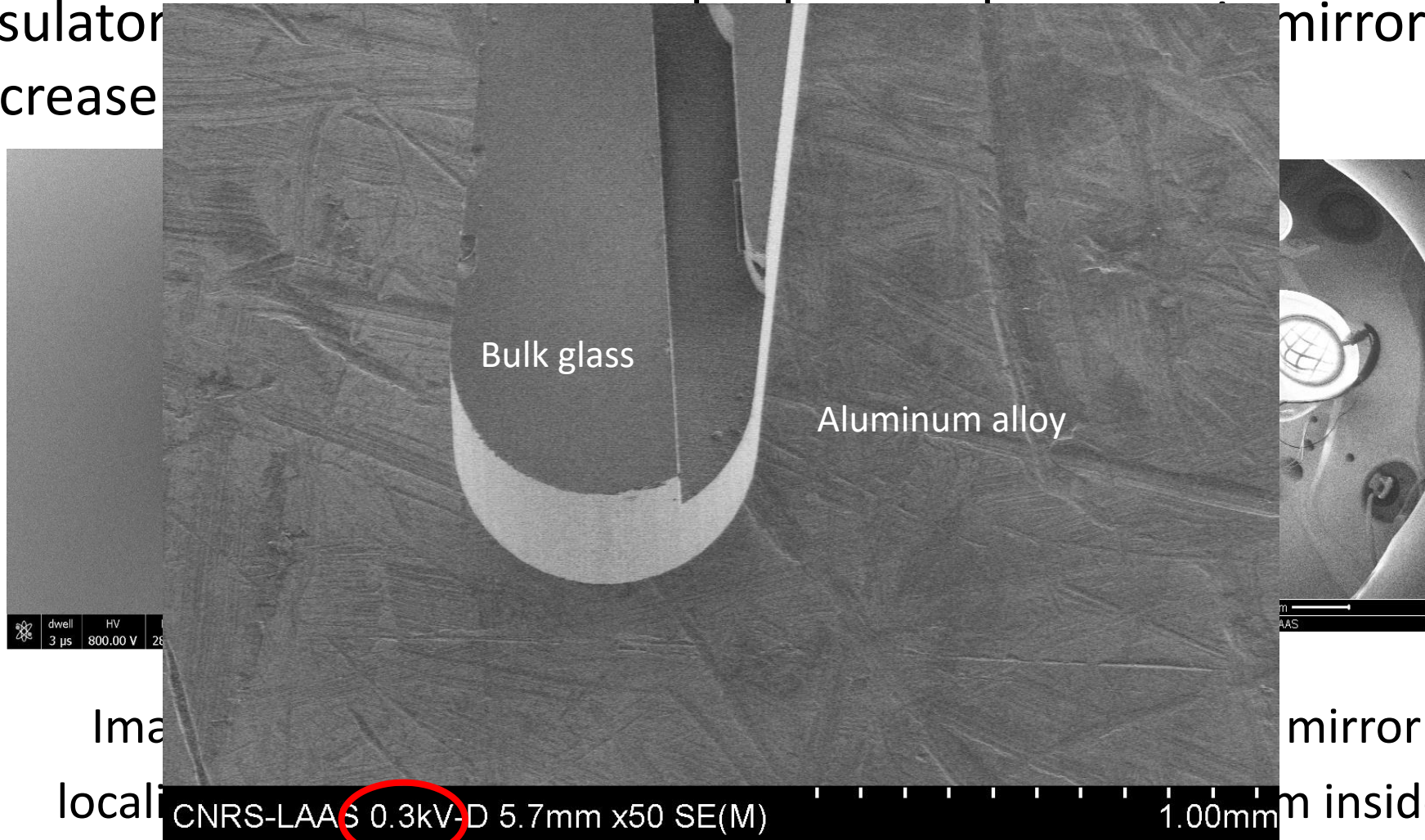
Low accelerating voltage

Influence of choices: charging effect

On insulator



decrease



mirror



Ima

local

mirror:

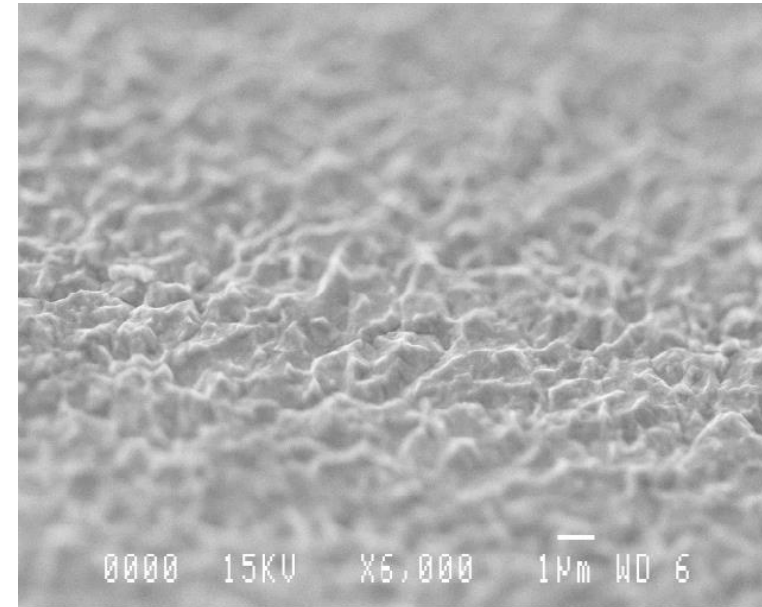
m inside

Influence of choices: depth of focus

- Height on which the beam size does not vary
- DoF = f(WD, obj. aperture size)
- Built-in synchronous focus variation across field of view

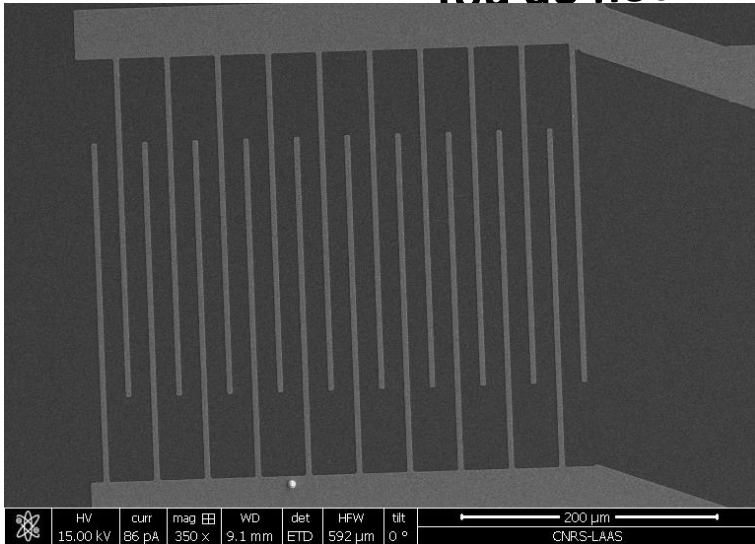
Example:

- Electrolytic Au / Si
- Tilt angle: 45°
- Working Distance: 6mm
- Depth of Focus: ~5 μ m

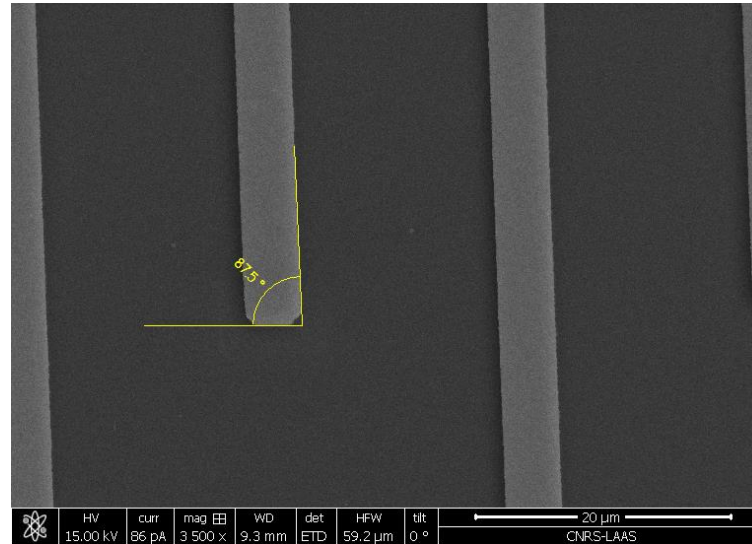


Influence of choices: layout

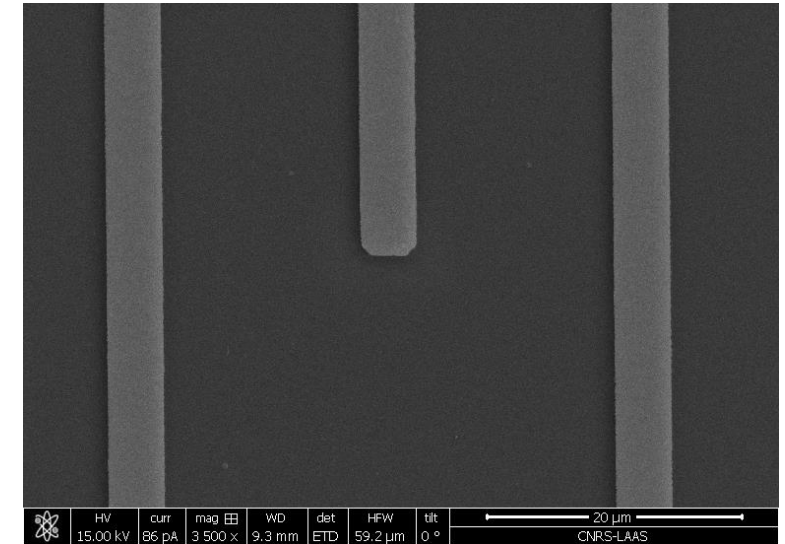
- Brain is organized in horizontal and vertical analysis
- When an image does not fit brain organization:
 - You induce constraints on the observer
 - You do not ease communication



Angle evaluation !?



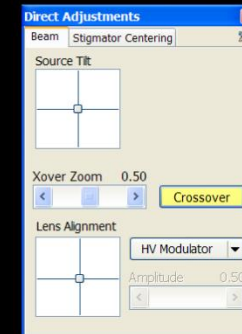
You are sensitive to $<2.5^\circ$



Use integrated function
to align your layout

- **A SEM is not calibrated at better than ~2.5 - 10%**
- **Depending on:**
 - HV, I, WD, Magnification
 - SEM quality
 - Regular calibration
- **Precise measurements need calibrated objects:**
 - Polystyrene beads deposited close to the area of interest
 - Known objects coming from a calibrated equipment: e.g. E-Beam writer

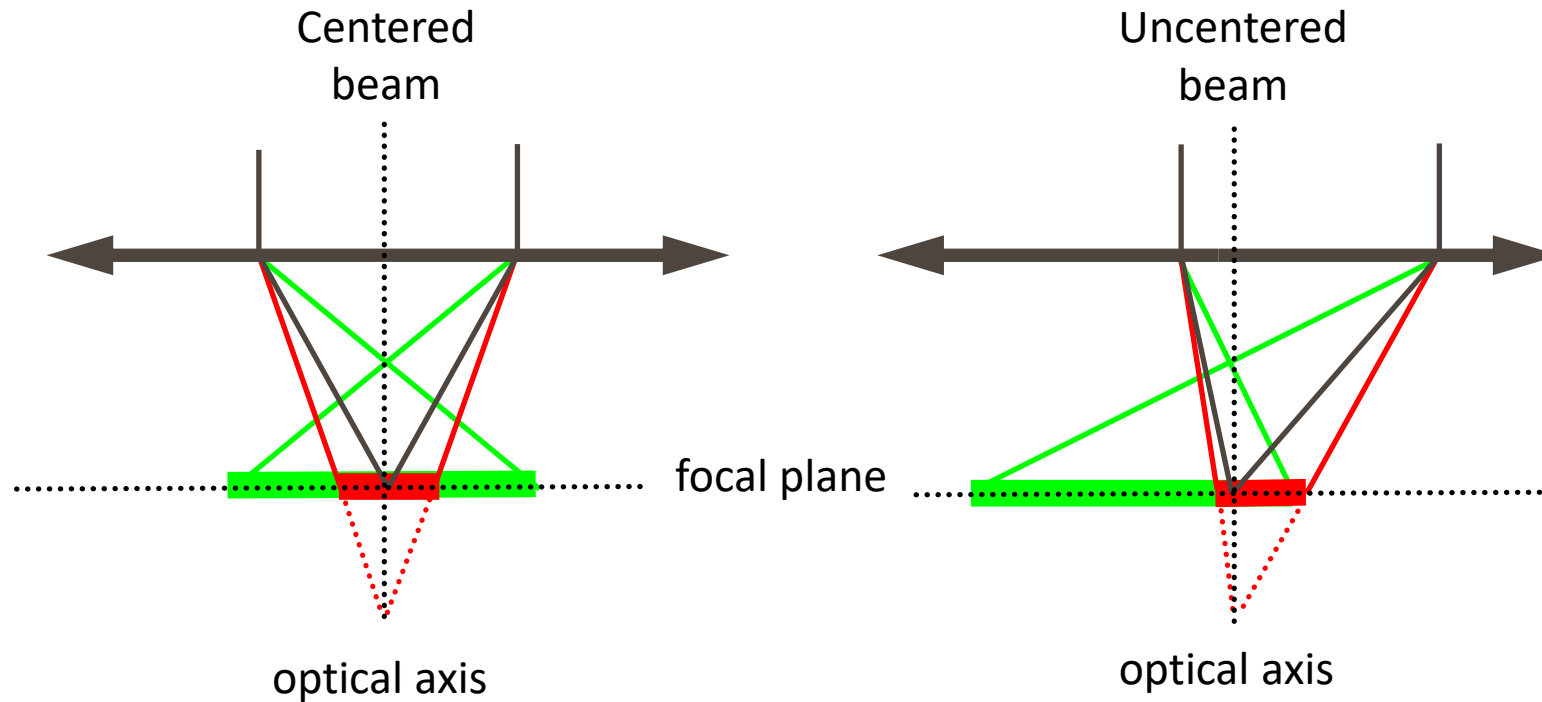
Defines optical axis

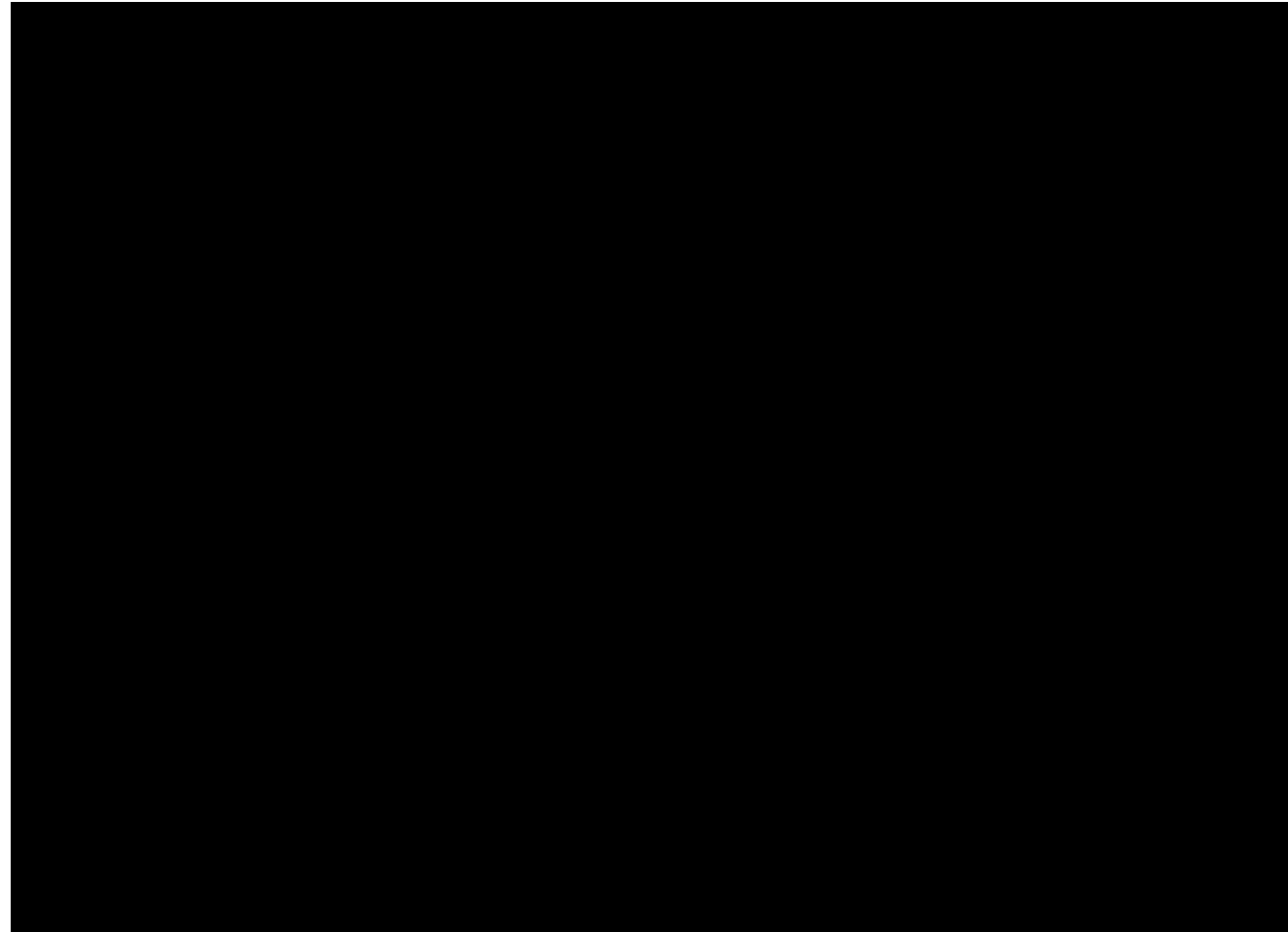


	HV	curr	mag	WD	det	HFV	tilt	500 μ m	Q1
	1.00 kV	86 pA	100 x	4.2 mm	ETD	2.07 mm	0 °		Act

- **Purpose is to use electrons :**
 - With the same energy
 - With the same trajectory
 - ☞ Focus them at the same location # high resolution
 - ☞ Misalignment induces bad resolution
- **All elements are mechanically pre-aligned**
- **Mini-coils inside the column to push the beam**

Procedures: alignment on optical axis



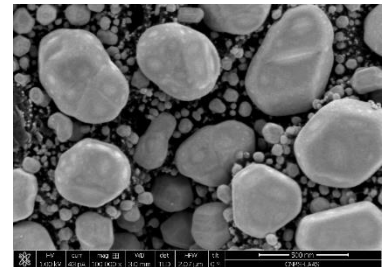
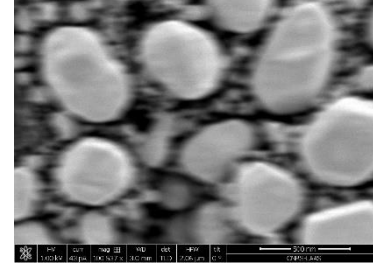
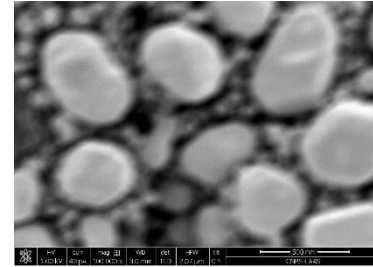
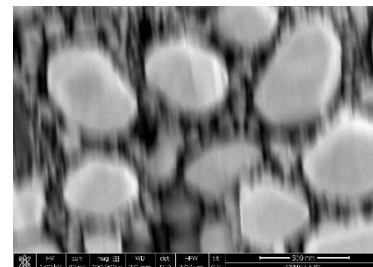
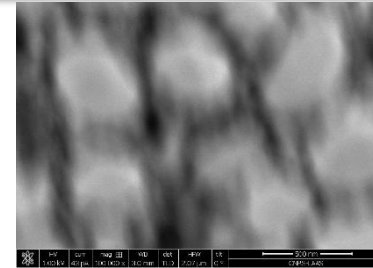
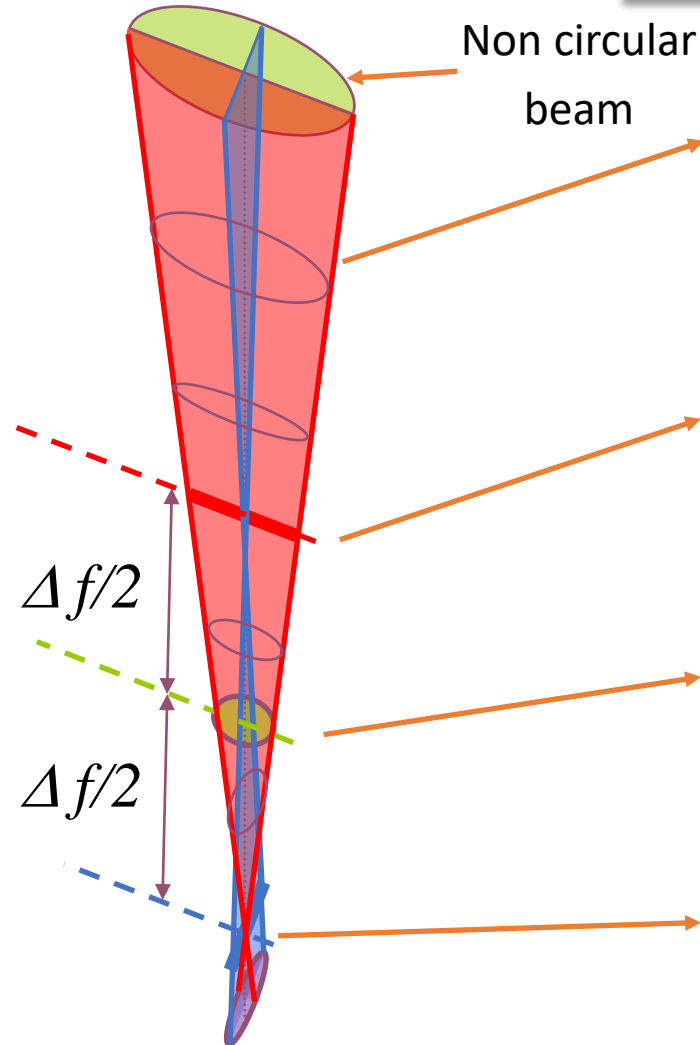


Procedures: astigmatism (adjustable)

sagittal focal
beam = line

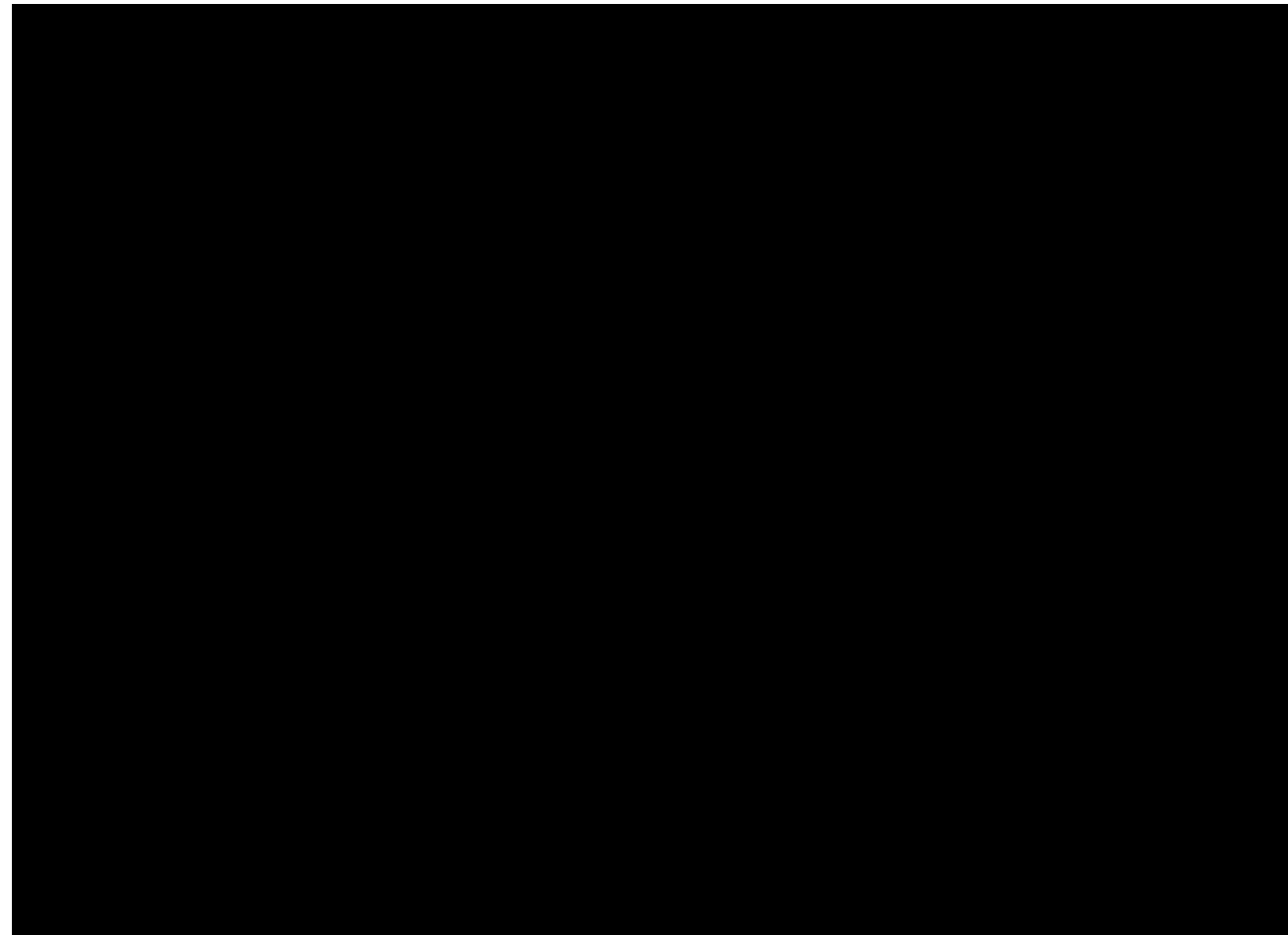
main focal
beam = circle

transverse focal
beam = line



SEM in practice

Procedures: astigmatism (adjustable)



Procedures: adjustments flow chart

Select electron beam energy and current:

- Starting energy: high energy for conductive samples (15keV), low energy for insulating samples (1keV)
- Starting current (beam size, spot size, ...):
 - Mean value for conductive sample to minimize the noise
 - Low value to minimize charging

Adjust image brilliance/contrast (use buttons or automatism)

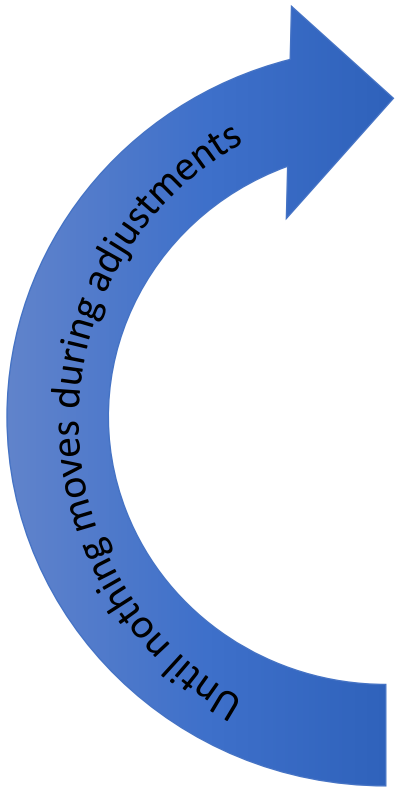
If there is too much noise or no image: verify GUN alignment

- Use the built-in "gun", "beam", "source", "cross-over", ... alignment procedure

Adjust focus roughly (use buttons / mouse)

Adjust sample position: X, Y, Z, tilt, focus distance (use buttons / mouse)

Procedures: adjustments flow chart



Start by adjusting focus ("objective" lens current)

If image moves during focus adjustment:

- Roughly adjust stigmatism to obtain an image of sufficient quality
- Use the built-in alignment procedure to push the beam onto the "objective" lens optical axis ("aperture align", ...)

Adjust stigmatism (stigmators lens X and Y currents)

If image moves during stigmatism adjustment:

- Use the built-in alignment procedure to push the beam on the optical axis of the X stigmator lens ("stigmator align", ...)
- Use the built-in alignment procedure to push the beam on the optical axis of the Y stigmator lens

Finalize focus/stigmatism and layout adjustments

Only with the right parameters (E, i) and the right position (X, Y, WD, Tilt, Rotation)

- > **Scanning Electron Microscopy characterization:**
 - Imaging: topographical and chemical contrast
 - Analysis: EDX, WDX, Auger, EBSD, ...
 - Simple to implement: training ~1 day for simple use
 - Surface characterization technique
 - "non destructive" (surface modification by pollution - black rectangles)
- > **2 main procedures:**
 - Lenses/aperture alignment: source (gun), "objective", stigmator X and Y
 - Focus/Astigmatism adjustment loop
 - Trick: "As long as you can: zoom in !. Resolution does not depend on magnification ... but your capability to adjust does !"
- > **Principal limiting factor: user !**
- > **50% job** ➡ **knowledge of the sample (location of structures to characterize)**
- > *Your efficiency is ecoresponsible*

- Academic websites:
 - [Société Française de Microscopies \(Sfμ\)](#)
 - [Réseau d'Imagerie en Microscopie Electronique \(RIME\)](#)
 - [Plateforme Toulouse Réseau Imagerie \(TRI Genotoul - Life Sciences\)](#)
- All manufacturers' websites:
 - [Hitachi](#)
 - [Jeol](#)
 - [Thermo Fisher Scientific](#)
 - [Tescan](#)
 - [Zeiss](#)
- Webinars from manufacturers (free registration required)
- Wikipedia