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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 ...

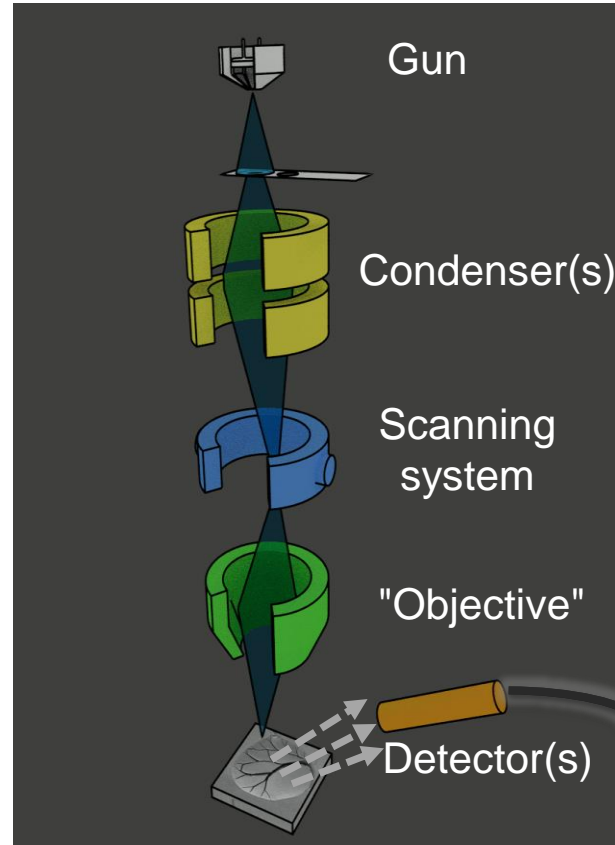


- **Schottky - Field Effect Gun (S-FEG)**
- **IN-LENS detectors (secondary & backscattered electrons)**
- **Imaging at very low energy (<1kV):**
  - Polarizing sample up to ~4kV
  - Imaging apparent voltage = electron beam accelerating voltage - sample polarization voltage
  - Keeps resolution and limits 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

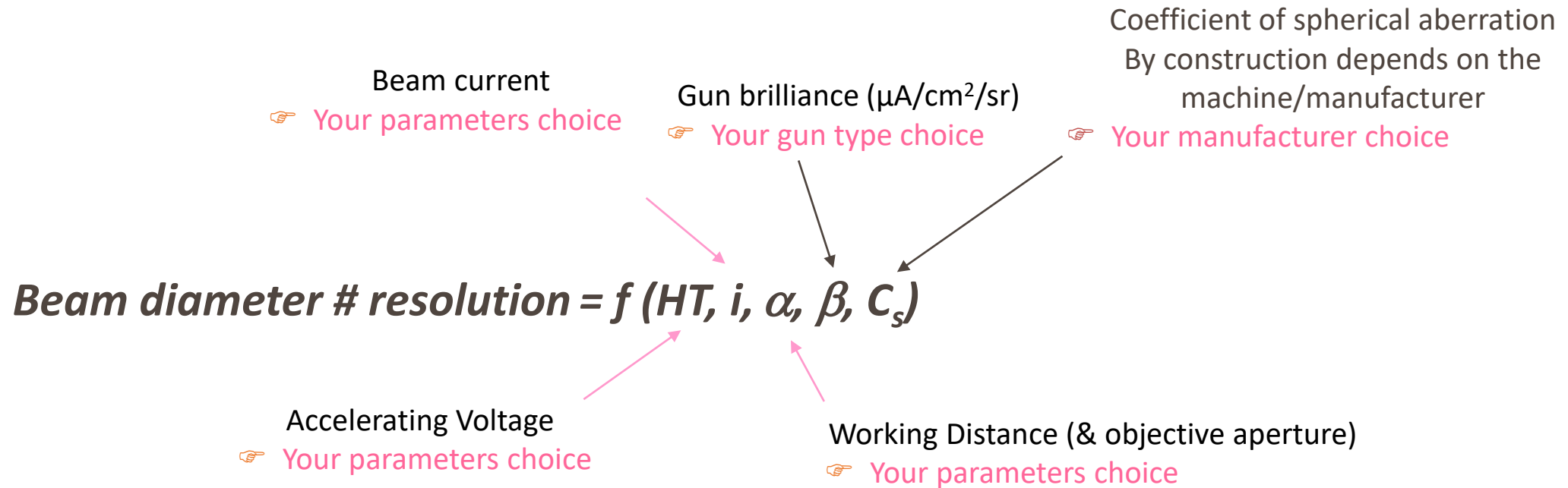
Size's source reduction (beam/spot size)

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:

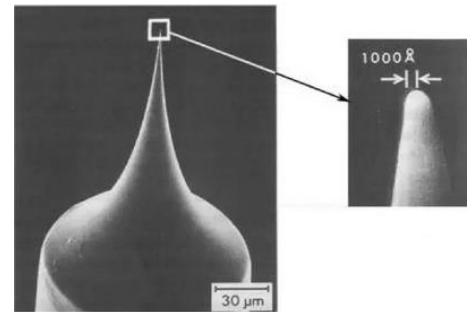
- Thermo-electronic:

- Electrons extracted from an heated filament (extraction:  $\sim 500V$ )
- 2 subfamilies:
  - W filament
  - LaB<sub>6</sub>, CeB<sub>6</sub> crystal soldered on W wire

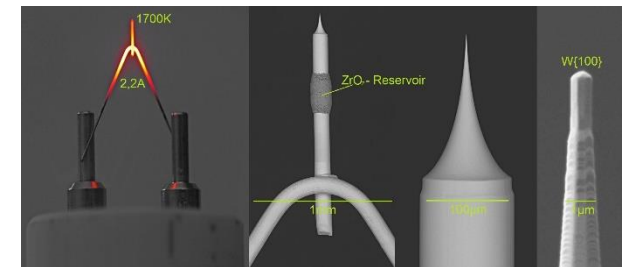


- Field effect guns (FEG):


- 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

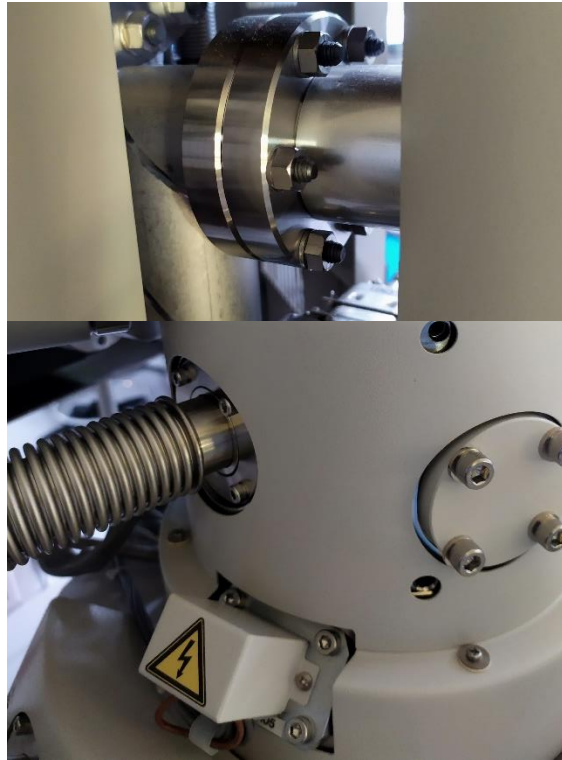


Wikipedia Schottky FEG tip - Erwin Meier

- Thermo-electronic guns:
  - W filament:
    - $<10^{-1}$  Pa ... easily achieved by a Turbo Molecular Pump (TMP) + Dry Pump (DP)
    - Filament lifetime: few hundred hours
  - LaB<sub>6</sub>, CeB<sub>6</sub> 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  $\sim 15$  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





# Electron beam generation: guns... choosing your SEM



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 ?

LaB<sub>6</sub> 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  $T^\circ$ , 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
  - $45^\circ$  surface escape half-angle
  - Detector located above the sample: can be broken easily

# Detectors: BackScattered Electrons Detector (BSED)

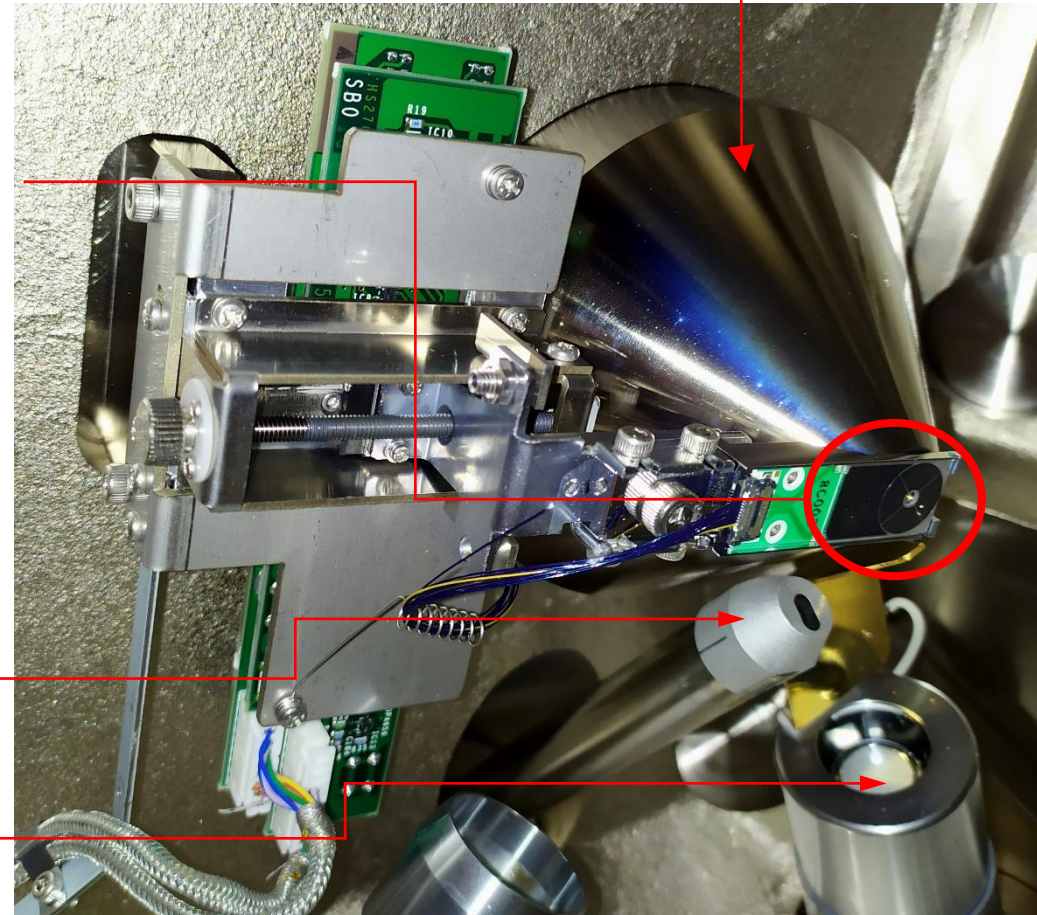
*"Objective" lens*

## BSE Detector

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

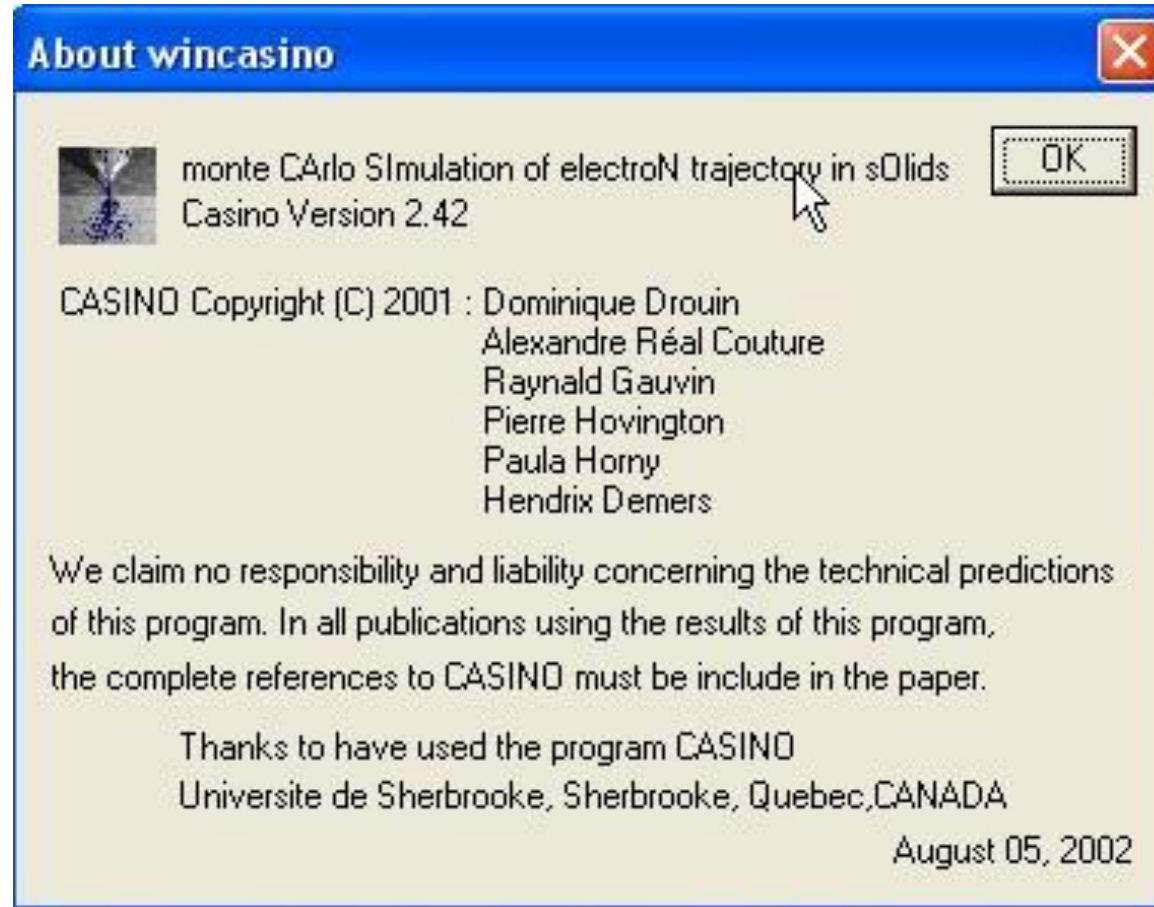
*X-Rays Detector*

*Secondary Electrons Detector*

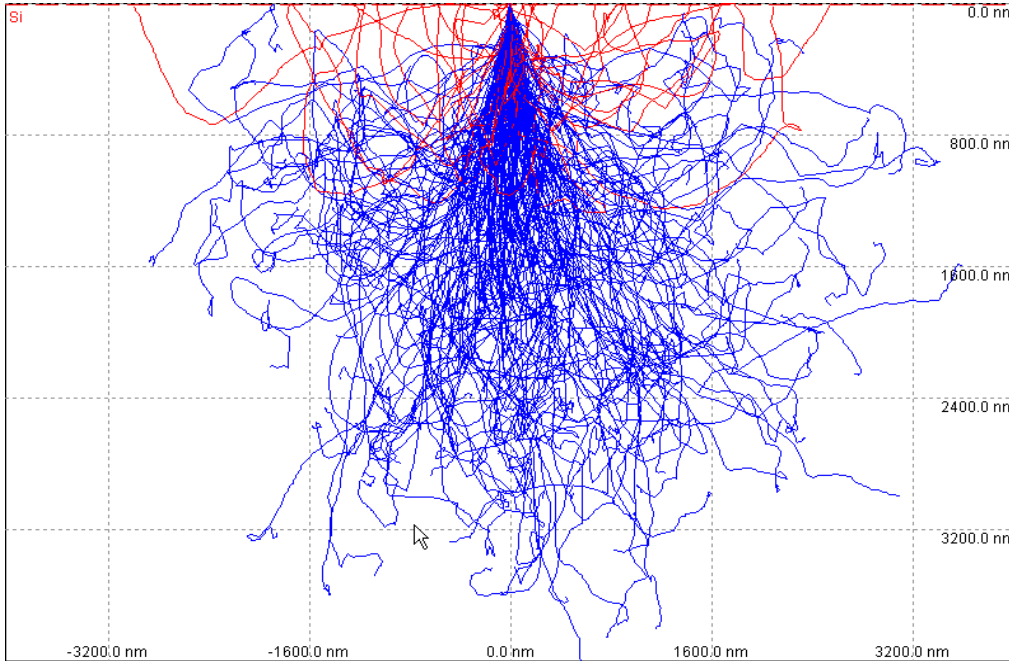


Chamber view from inside

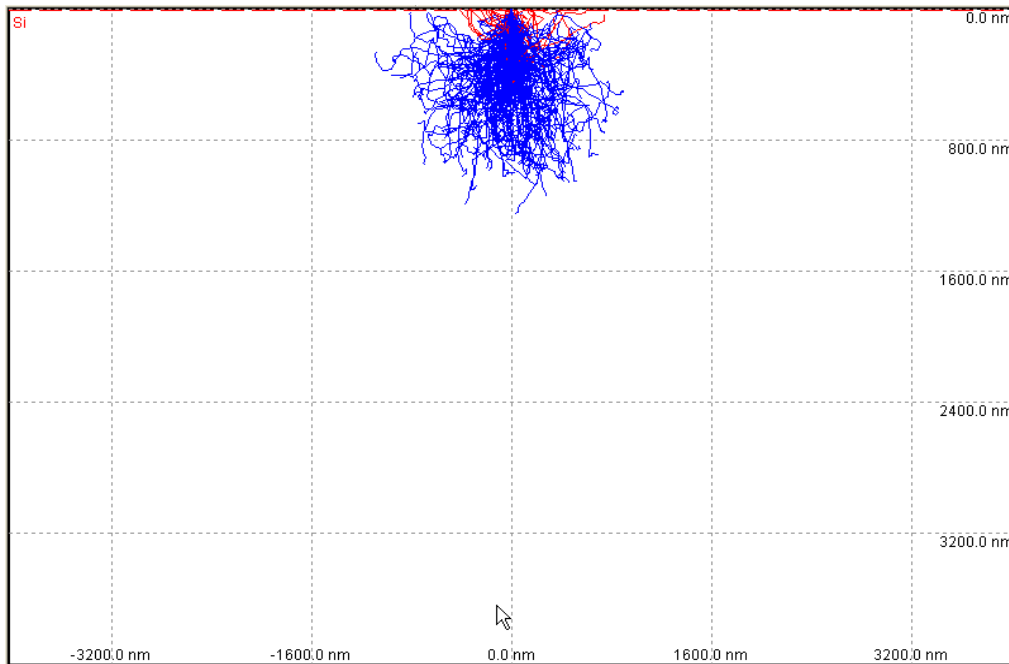




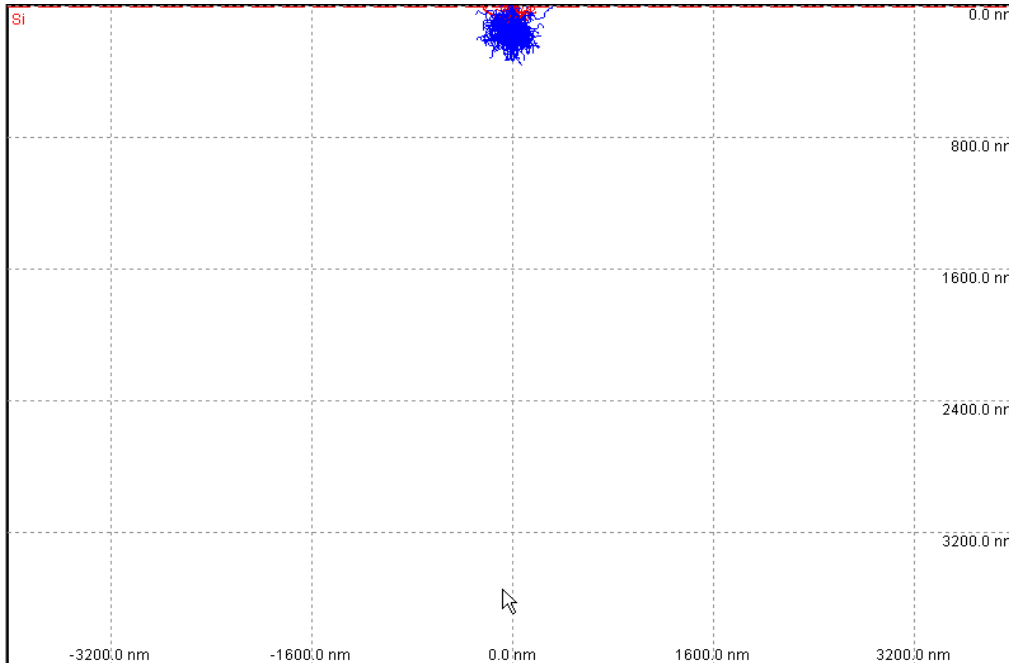
<http://www.gel.usherbrooke.ca/casino/>



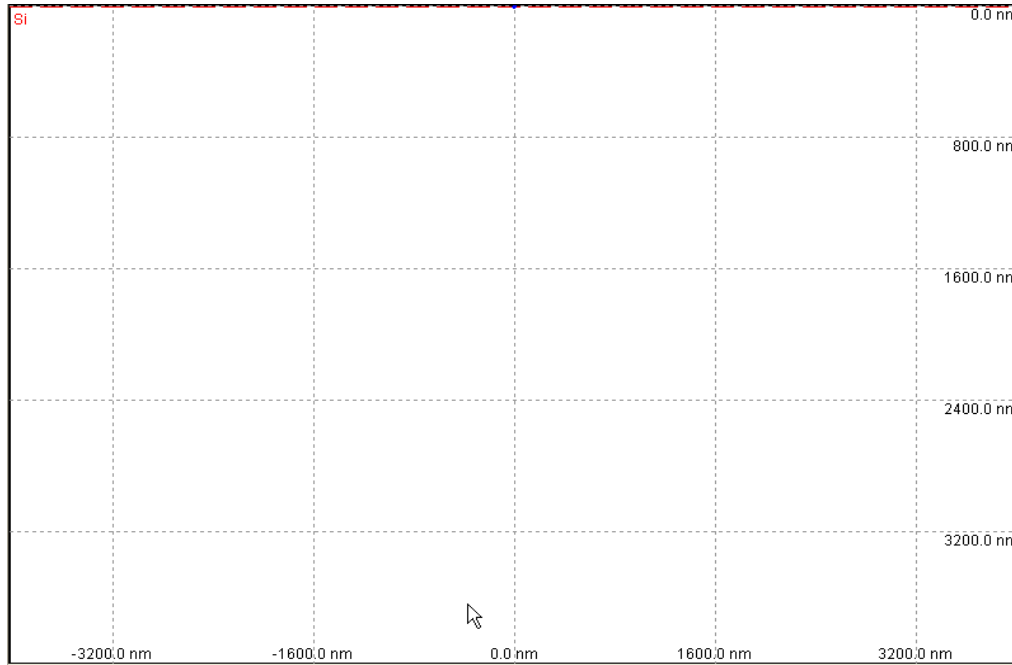
- Substrate: Si
- Beam Energy: 20keV
- Backscattered coefficient: 17.5%
- Interaction volume:
  - Max depth: 4 $\mu$ m
  - Max radius: 3 $\mu$ m



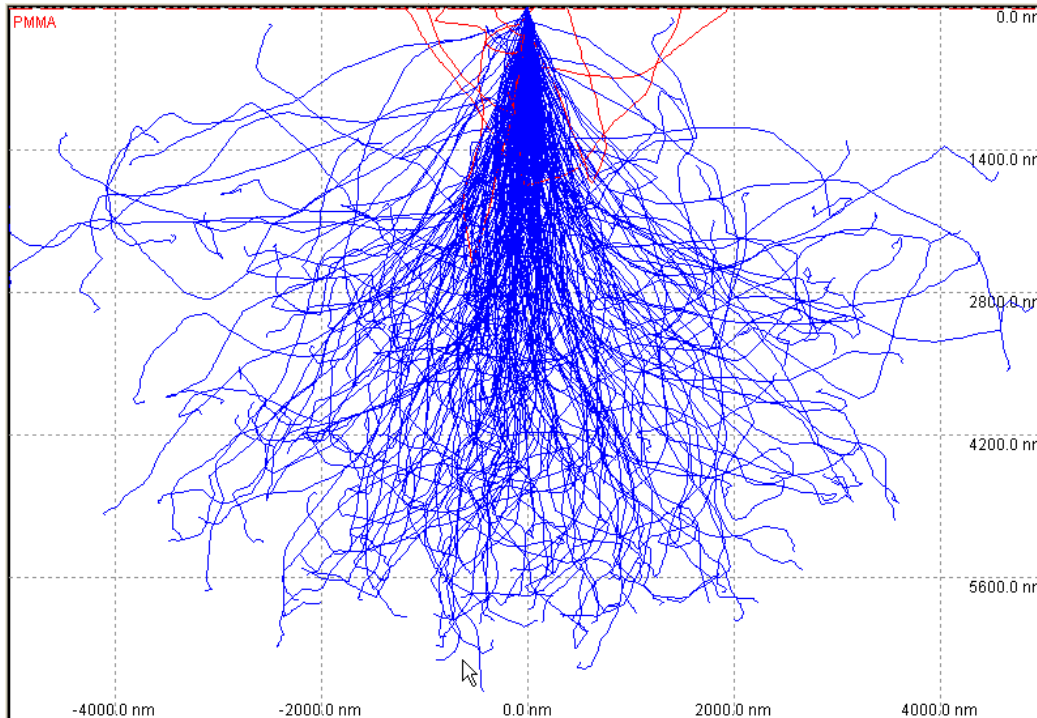
- Substrate: Si
- Beam Energy: 10keV
- Backscattered coefficient: 15.5%
- Interaction volume:
  - Max depth: 1.300μm
  - Max radius: 950nm



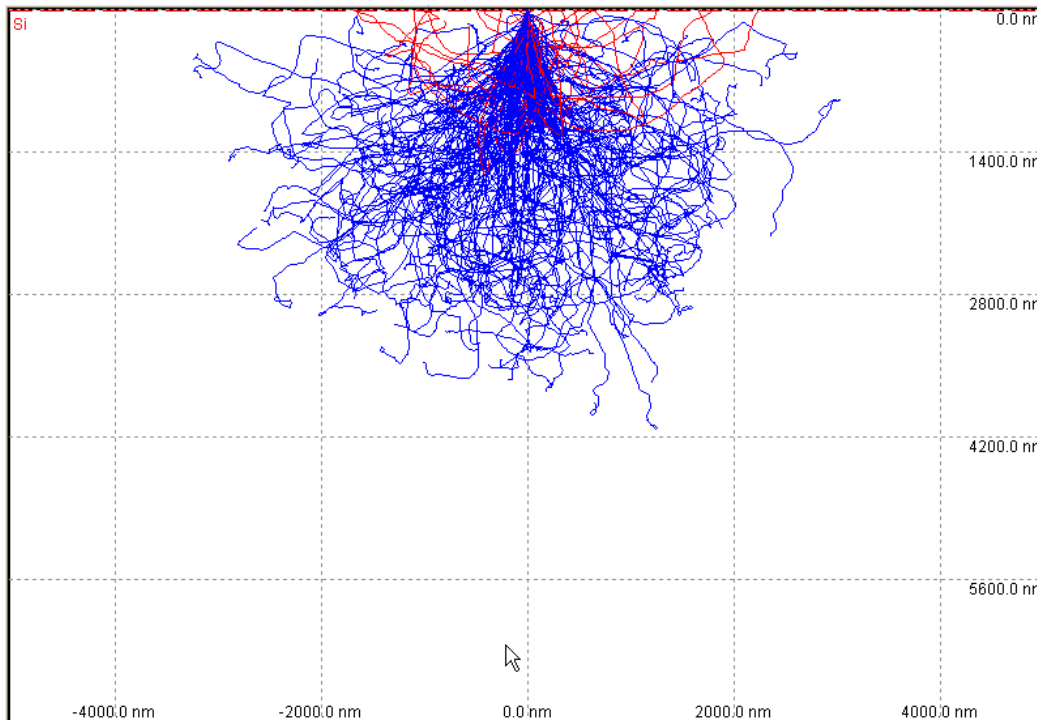
- Substrate: Si
- Beam Energy: 5keV
- Backscattered coefficient: 17%
- Interaction Volume:
  - Max depth: 370nm
  - Max radius: 280nm



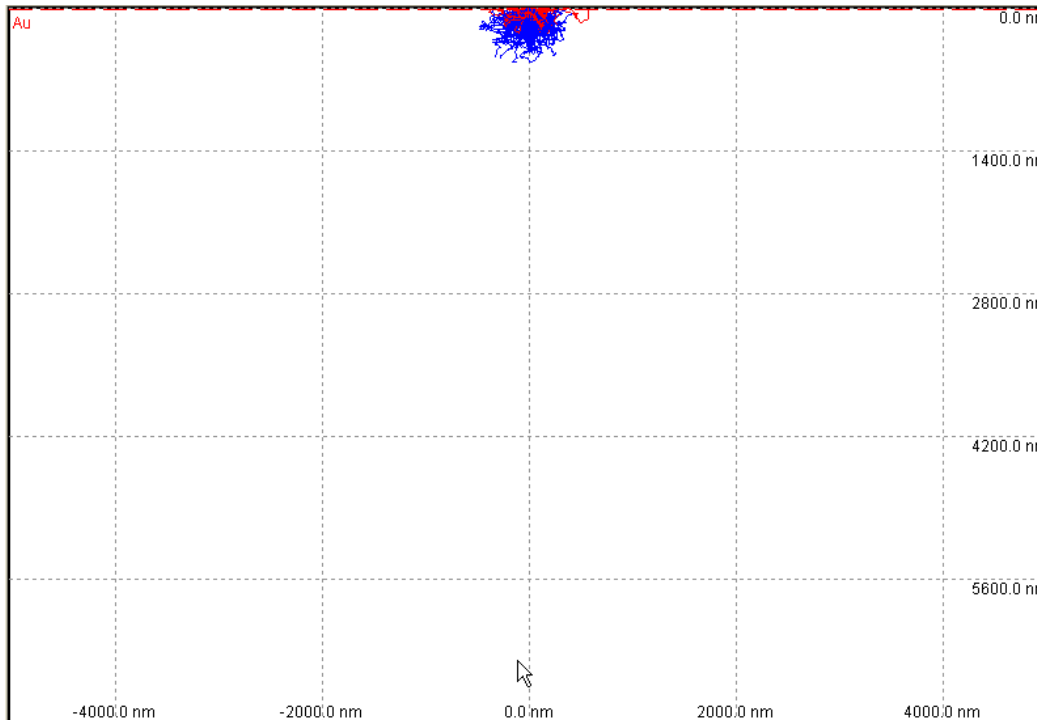
- Substrate: Si
- Beam Energy: 1keV
- Backscattered coefficient: 15.5%
- Interaction Volume:
  - Max depth: 30nm
  - Max radius: 30nm



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



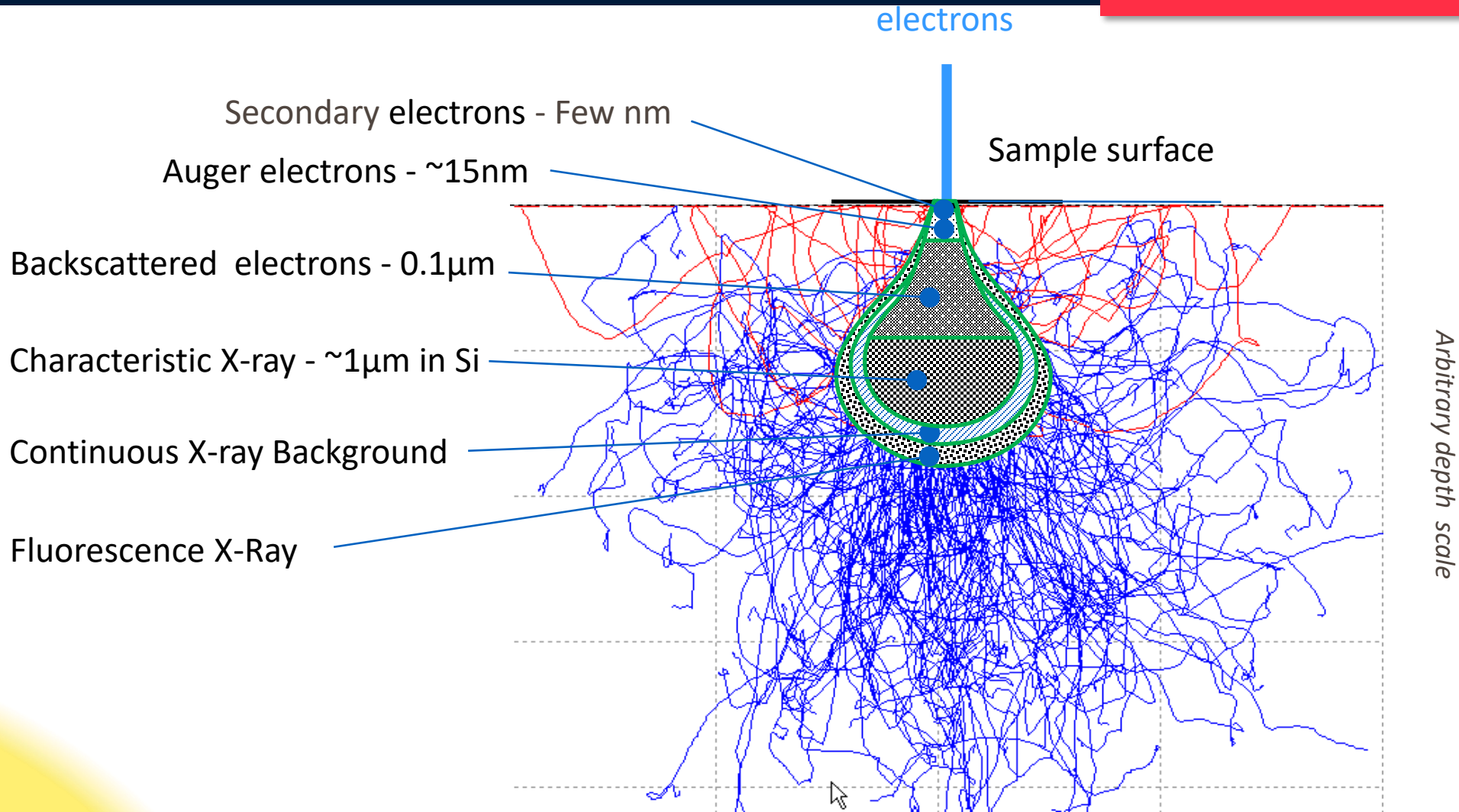
- Substrate: Si (Z=14)
- Beam Energy: 20keV
- Backscattered coefficient: 17.5%
- Interaction volume:
  - Max depth: 4 $\mu$ m
  - Max radius: 3 $\mu$ m



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

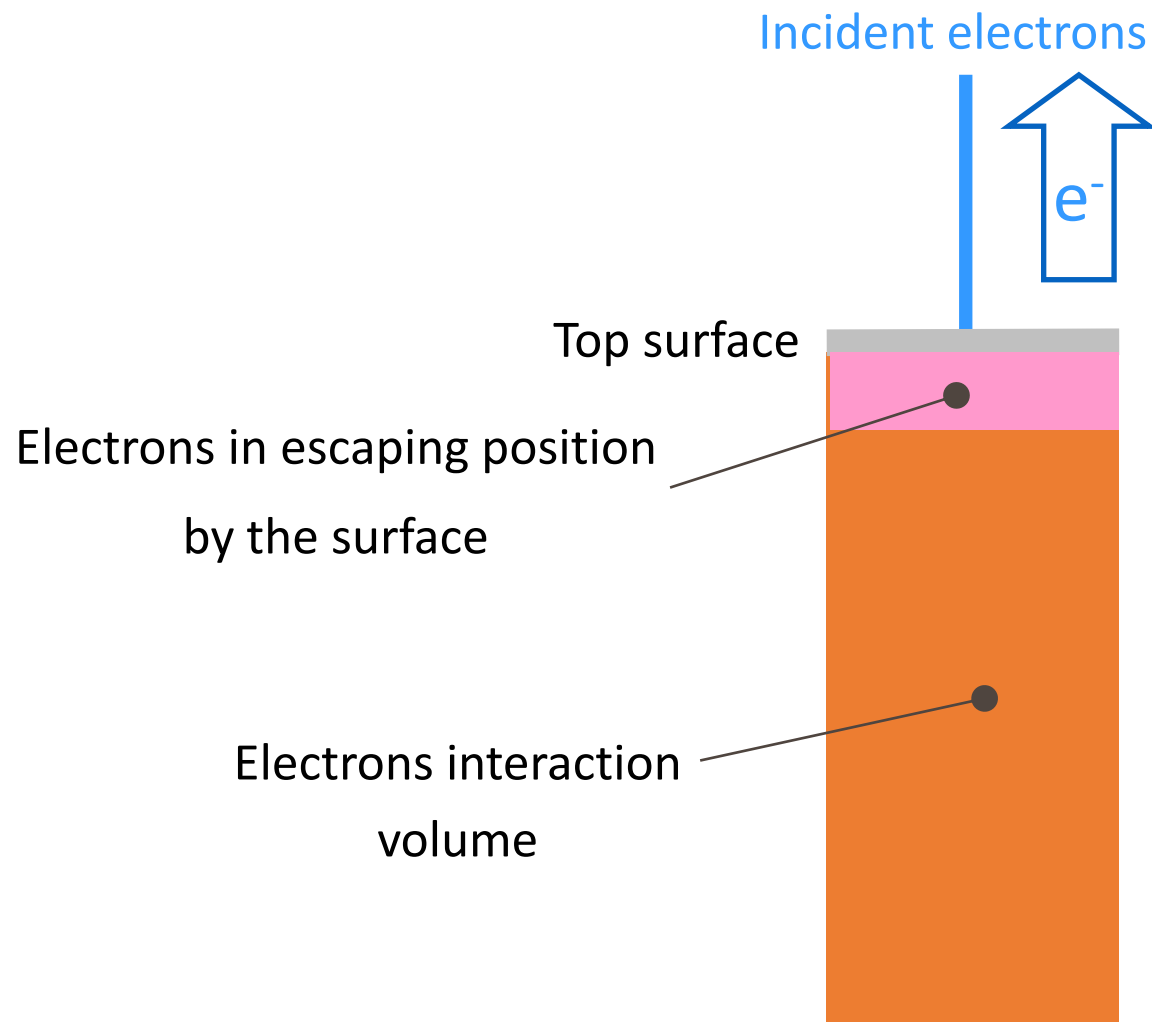


# Phenomena involved: max depth of escape

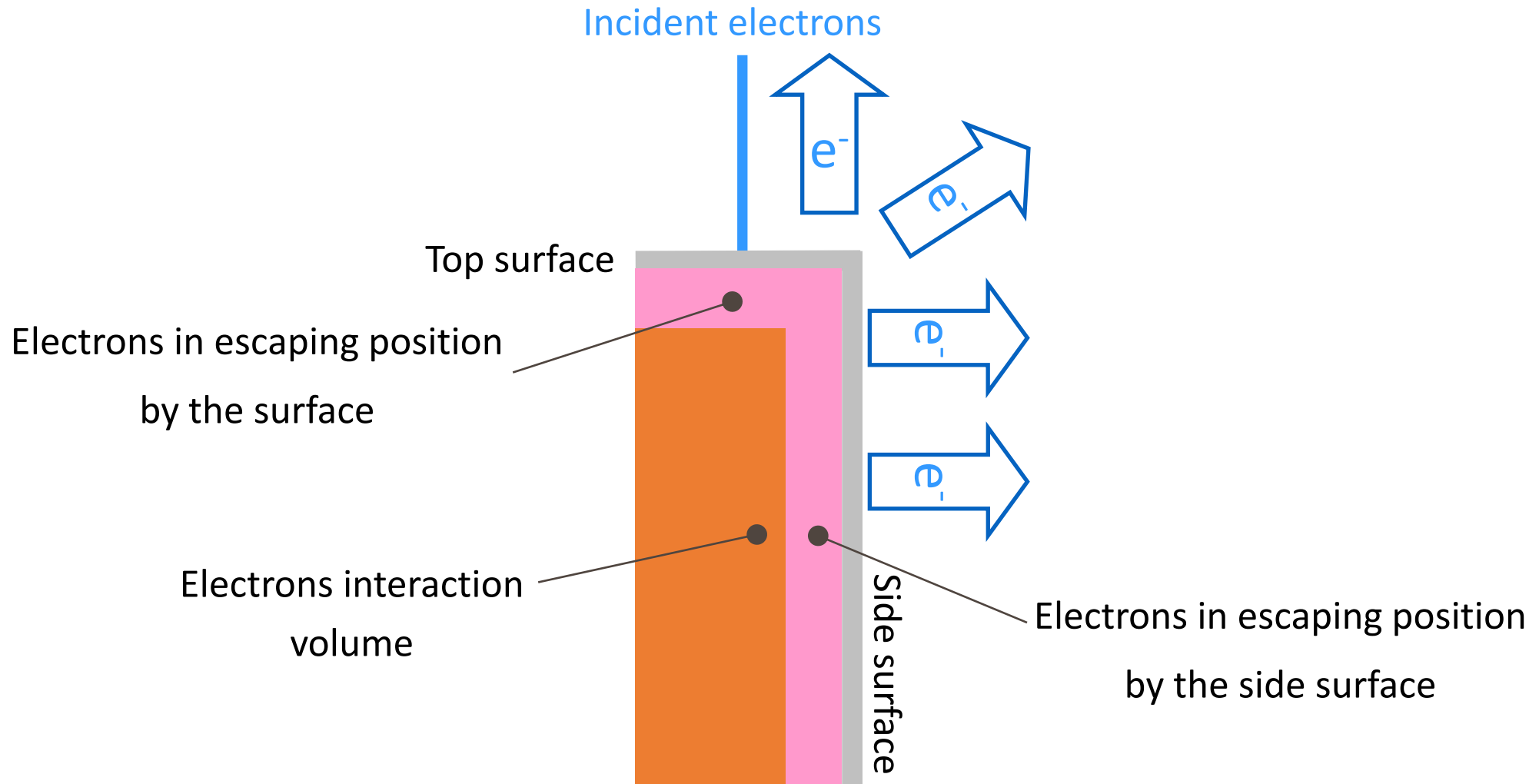


- Emitted by the sample
- Energy  $< 50\text{eV}$
- Mean free path: few nm
  - Close to scattered electrons trajectories
  - Emitted from few nanometers near the surface
- Source of contrast:
  - Intensity depends mainly on topography
  - Depends also on:
    - Atomic number
    - Organic contamination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

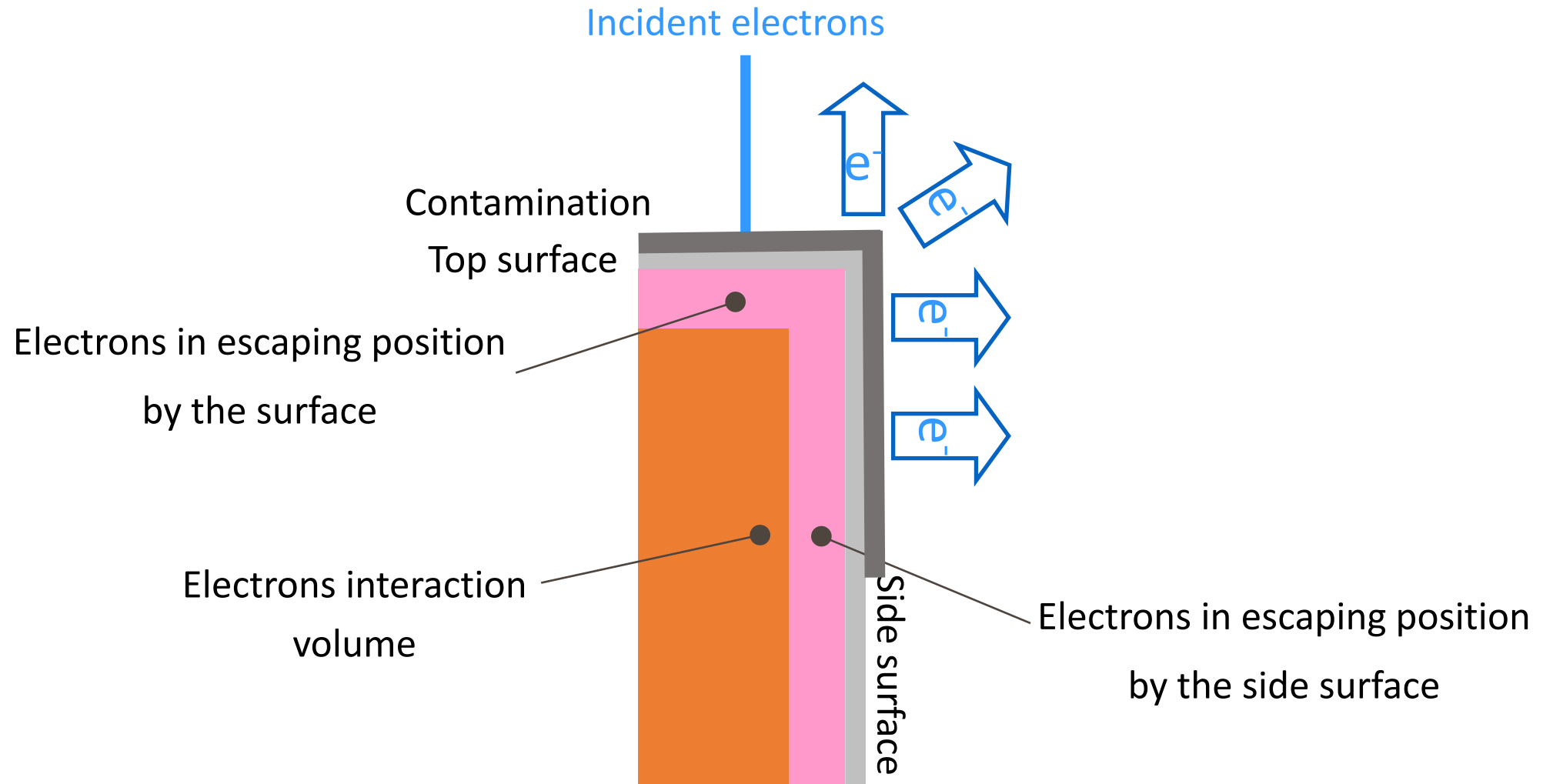
# Origin of contrast: topography



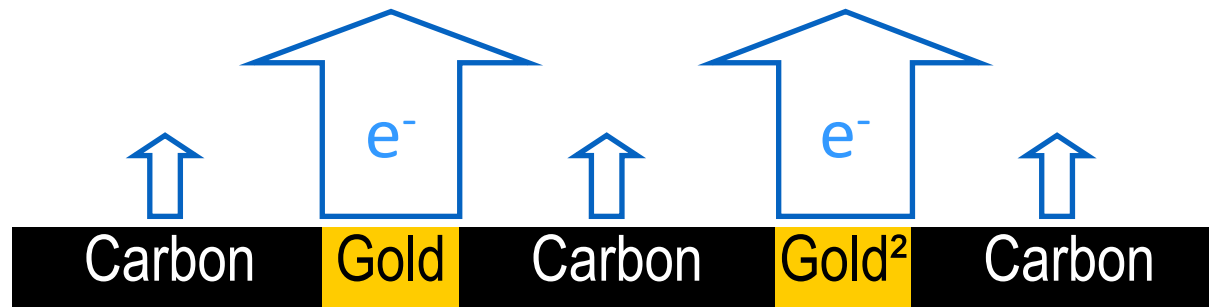
# Origin of contrast: topography



# Origin of contrast: topography

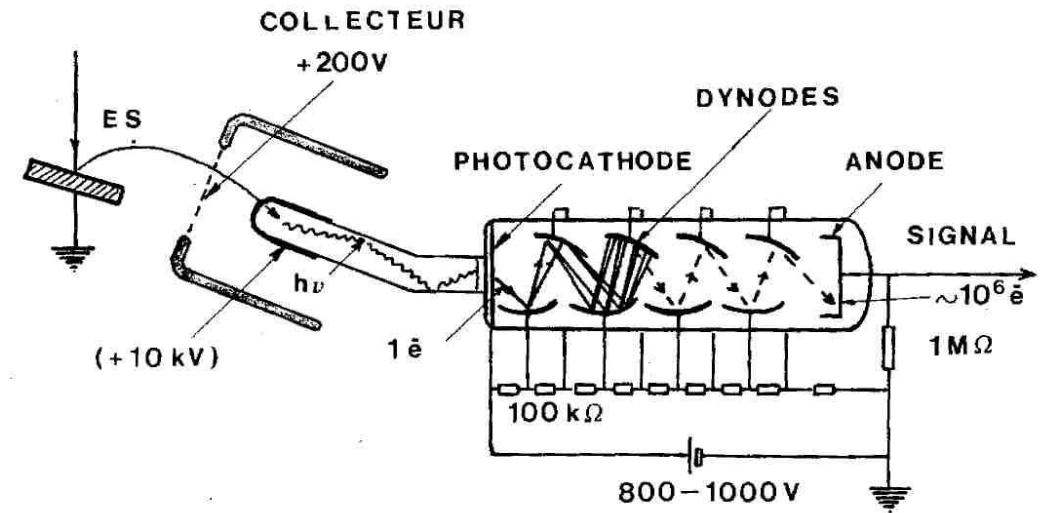


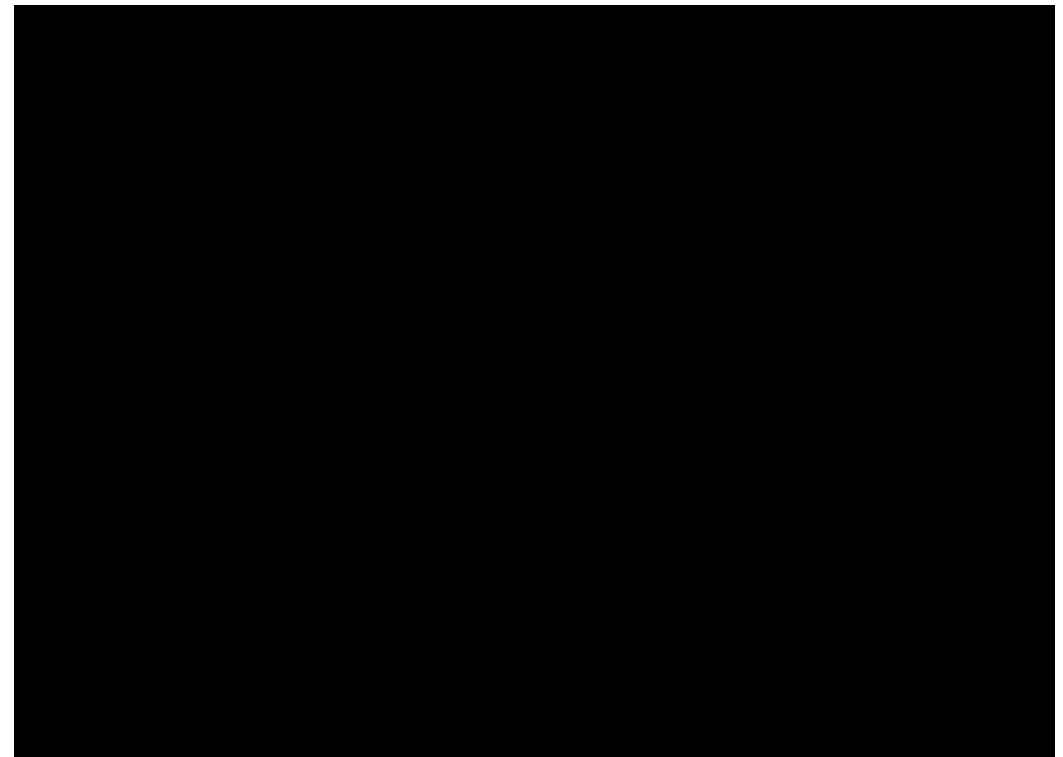
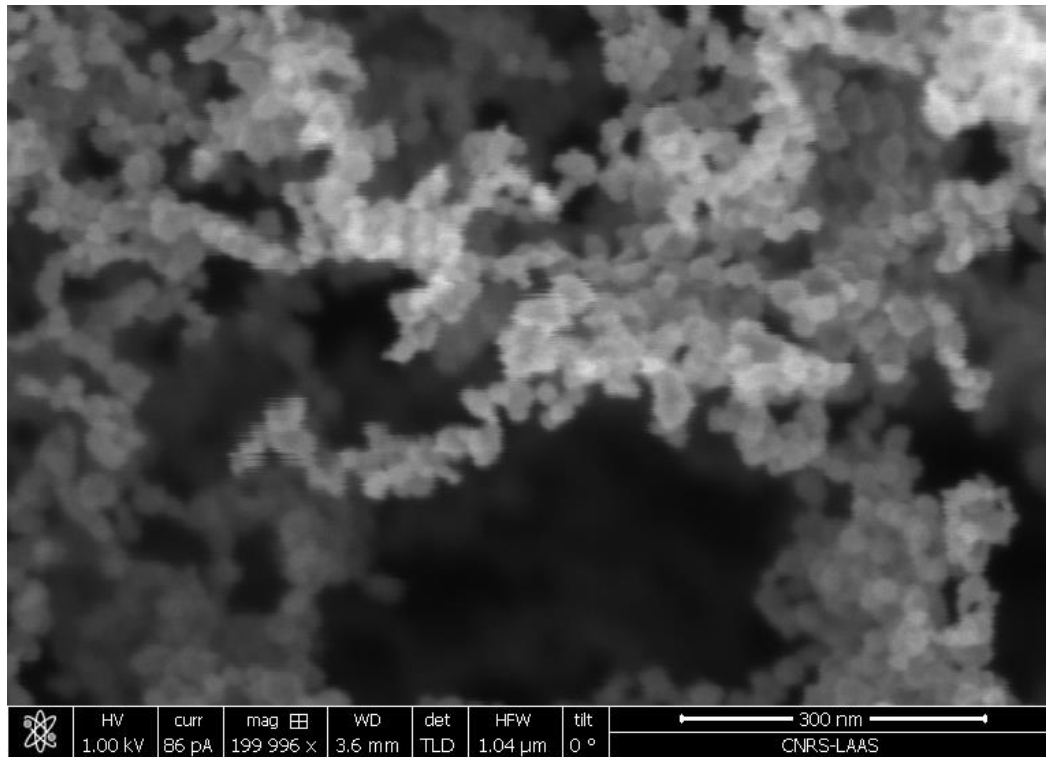
Sample without topography composed of materials with different Z:



- 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

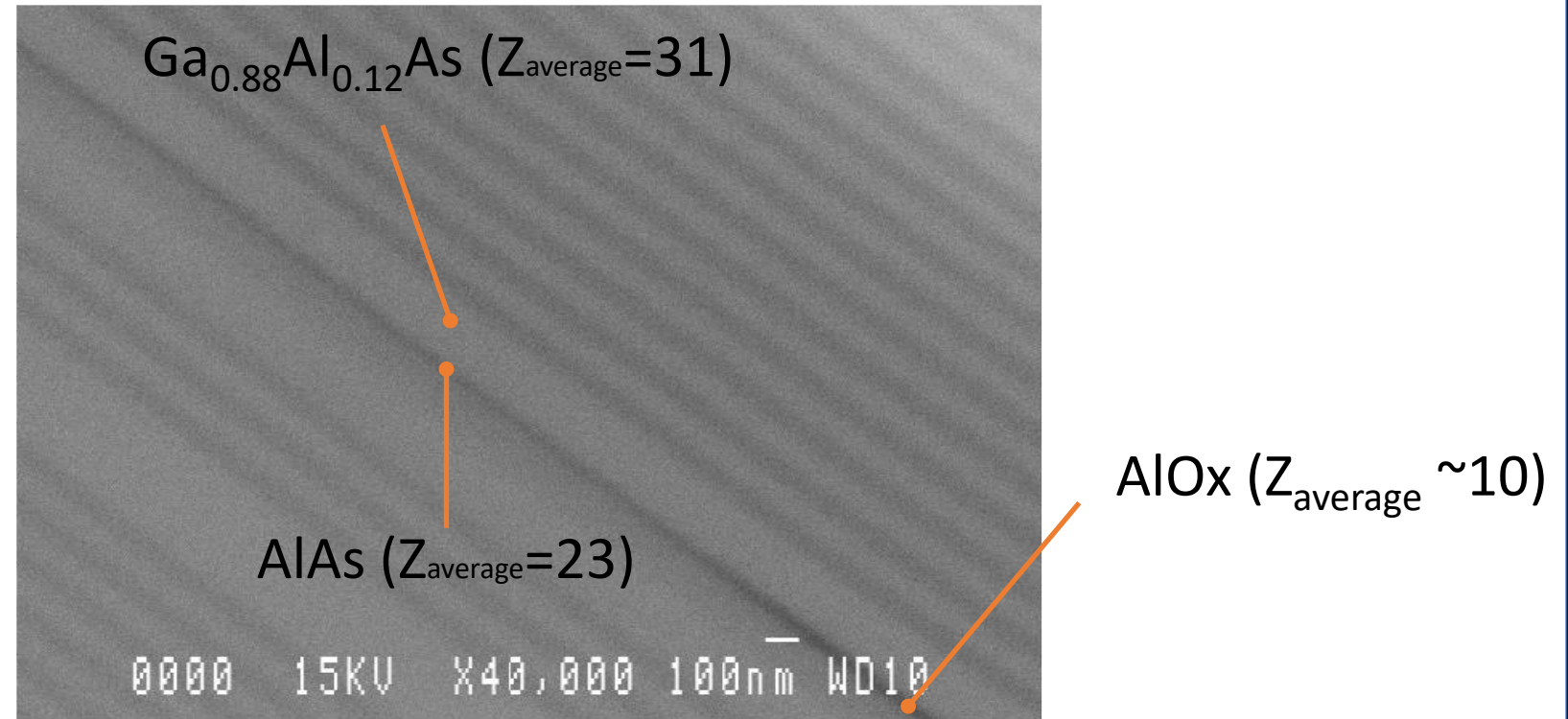




Topographical image example: candle stick fume nanoparticles (carbon)

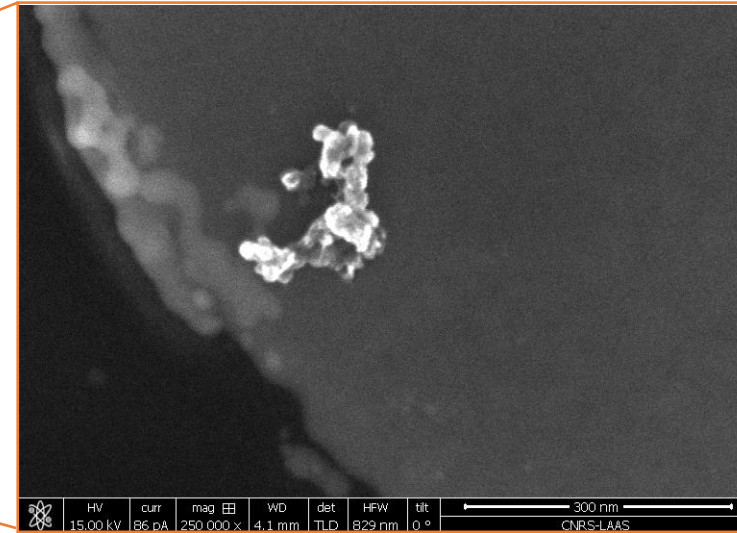
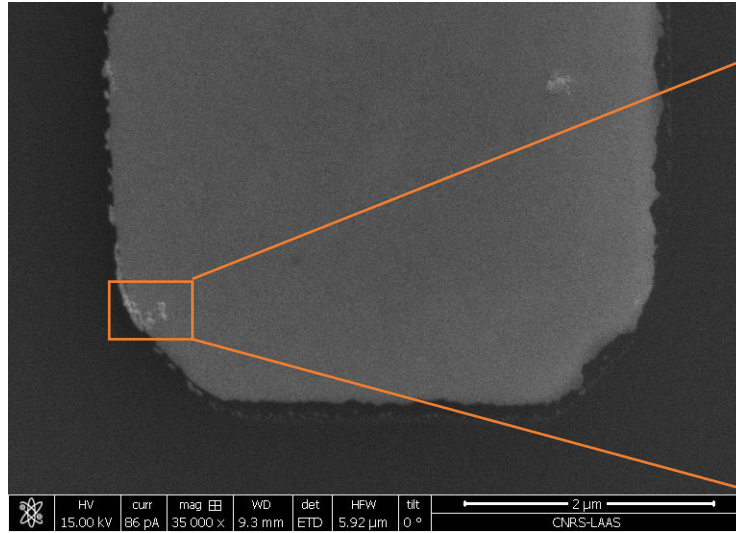


# Origin of contrast: chemical contrast with SED

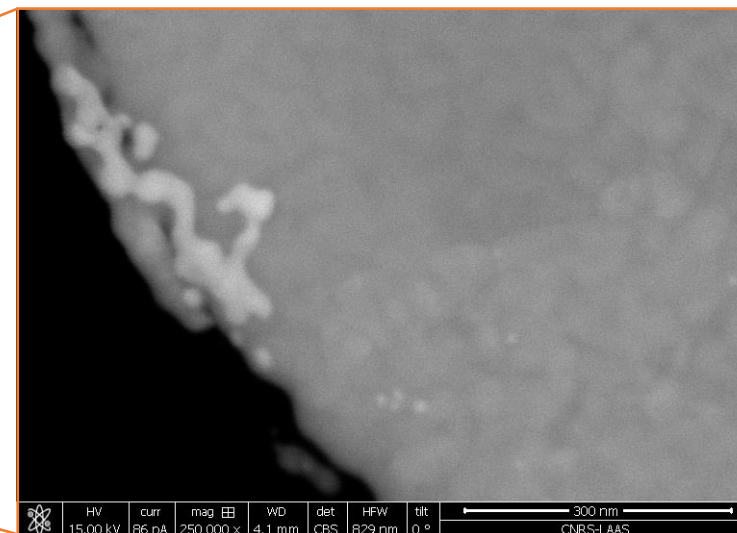
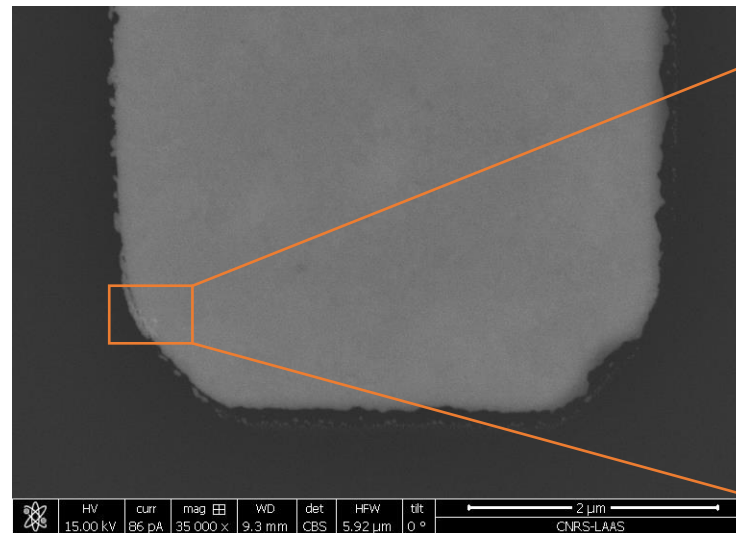


Flat freshly cleaved epitaxial sample without chemical etching

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



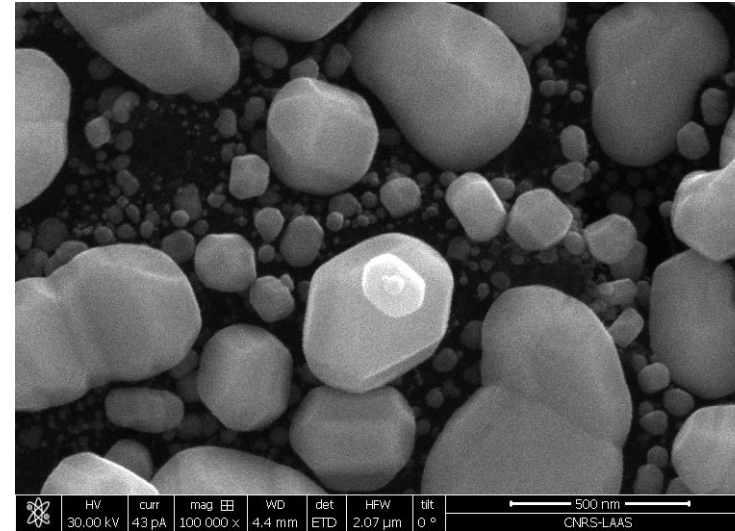
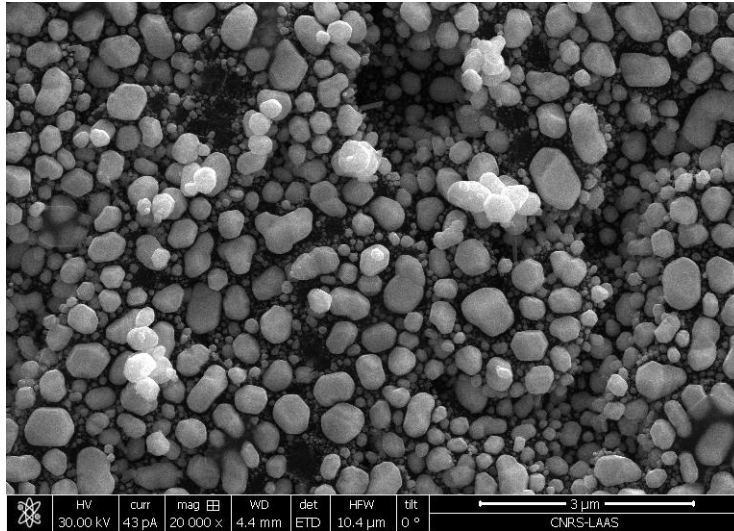
SED



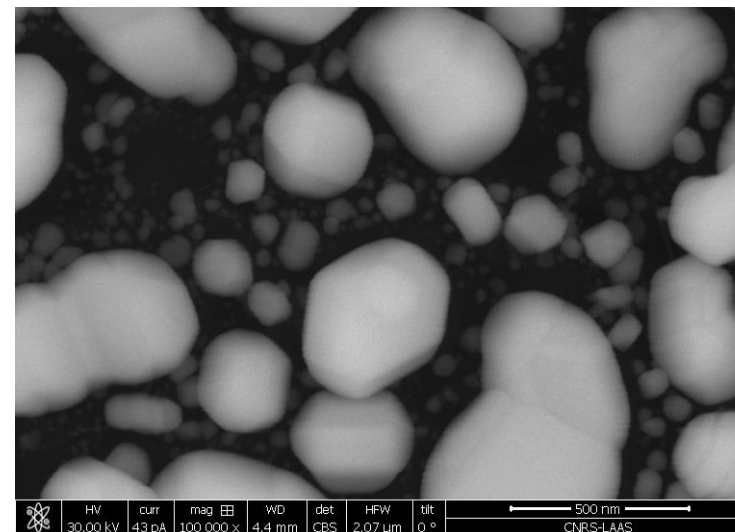
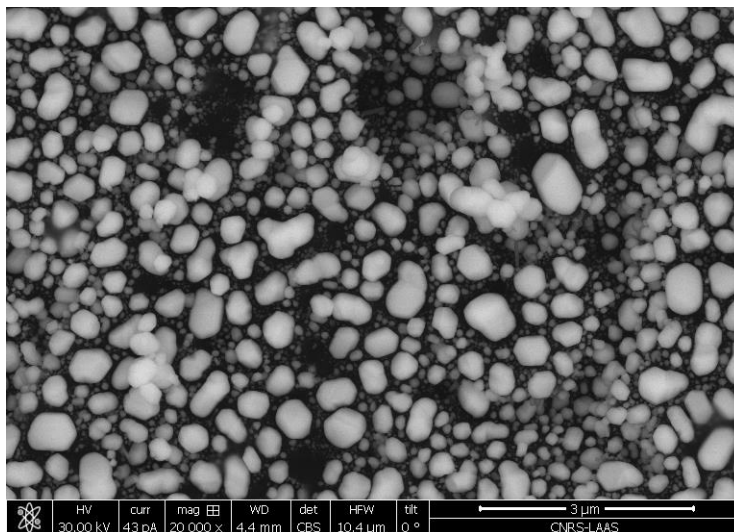
BSED

# Origin of contrast: BSED vs SED

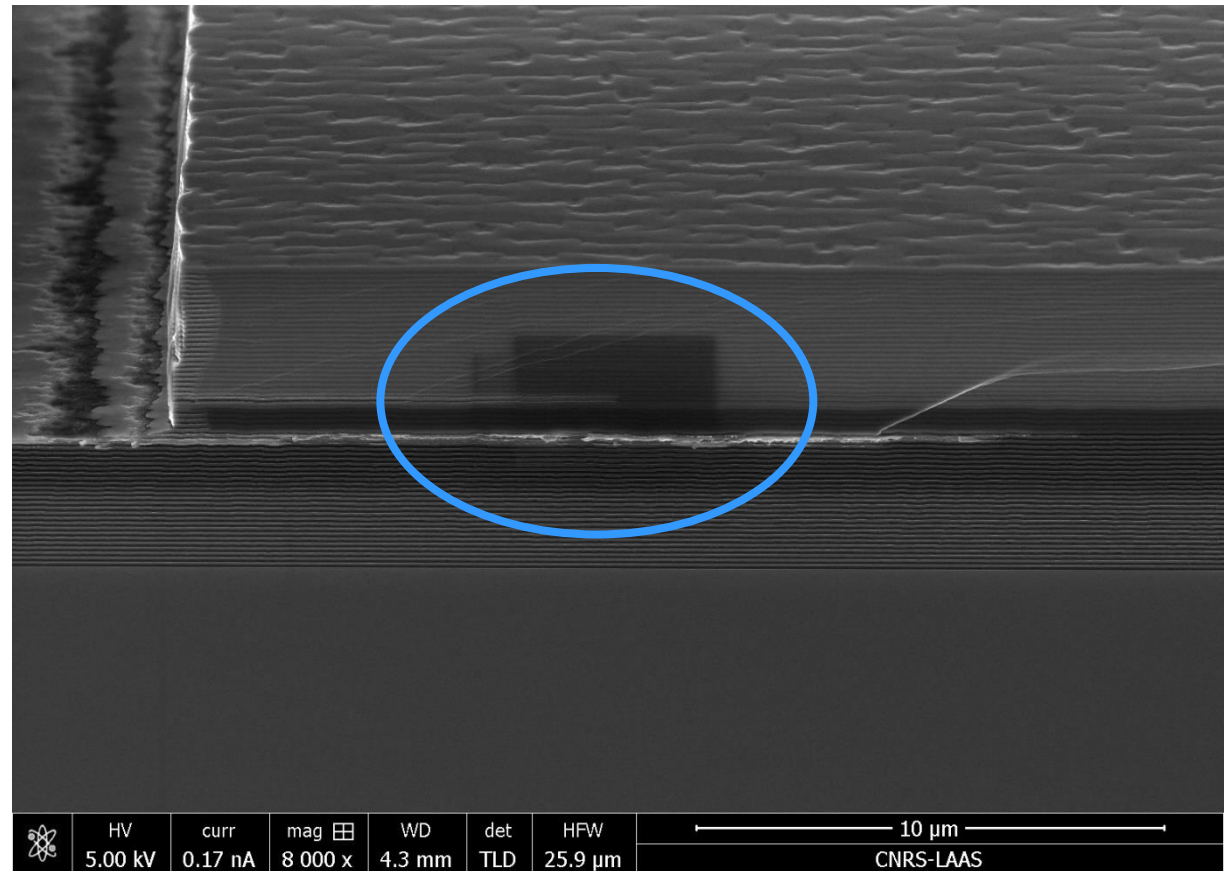
SED



BSED

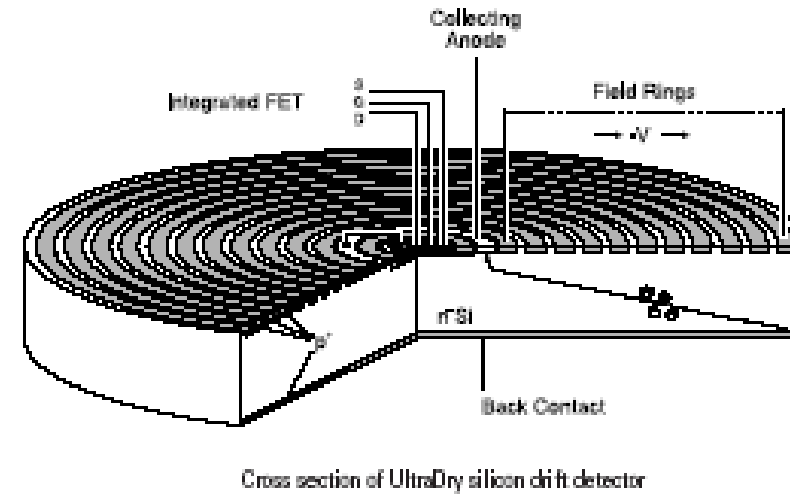


- Surface contaminant cross-linking
  - Coming from the substrate
  - Coming from the vacuum
- Adding a material based on C:
  - Low SE emission
  - Low contrast
  - ☞ Black rectangles



Tilted view of epitaxially grown layers after local etching

# Detectors: X-Rays detector

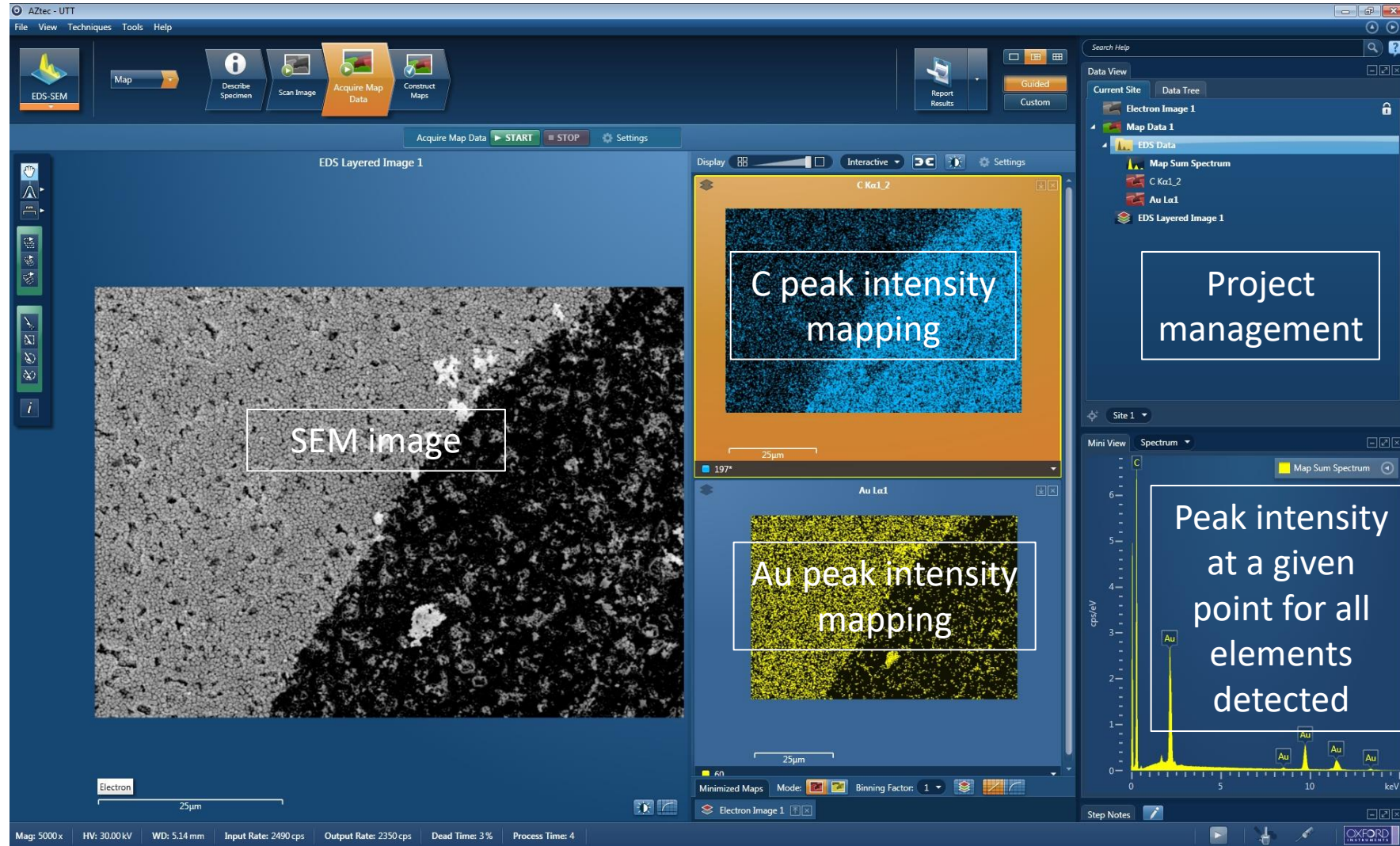


Energy Dispersive X-ray Spectroscopy (EDS or EDX or ...)

- 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

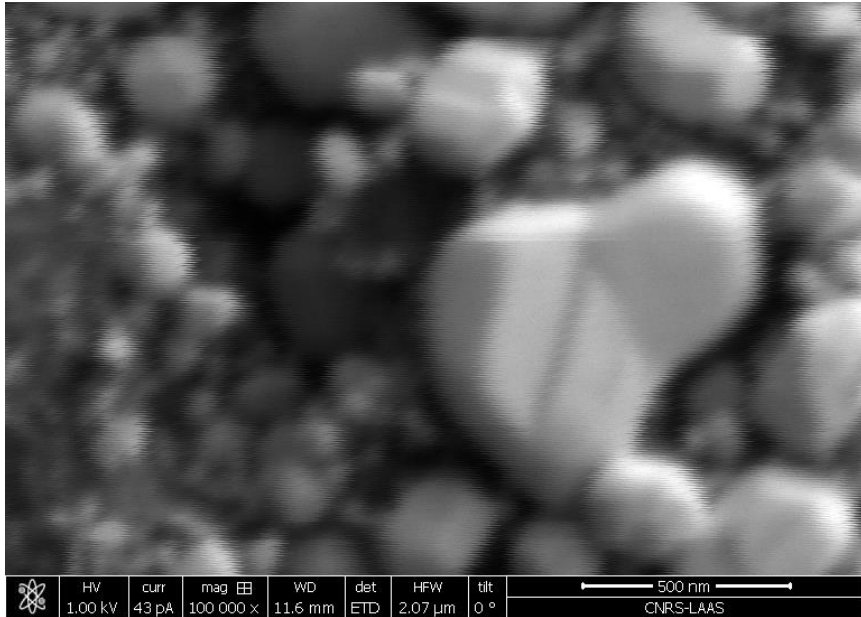
**... It's simple !**

# Influence of choices: summarized effects

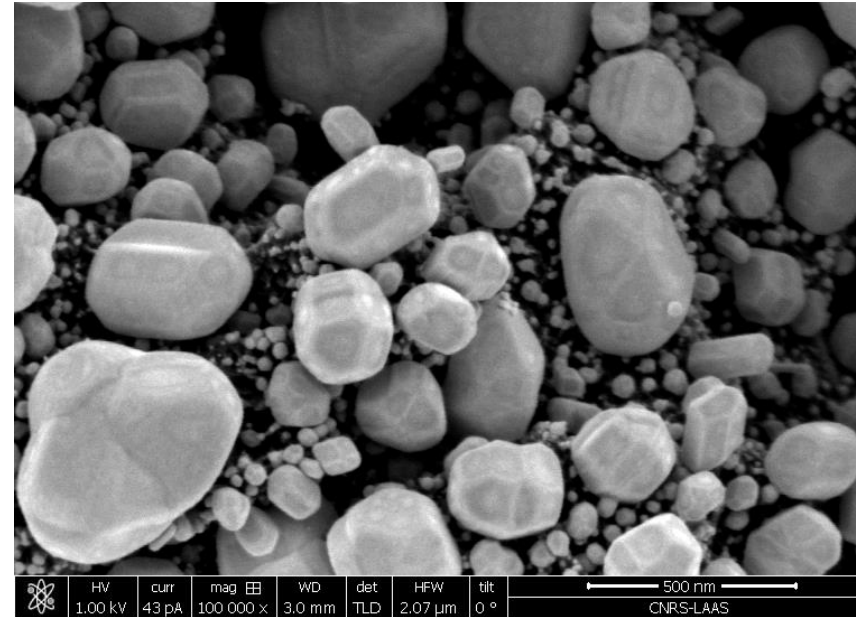
Working distance ↘	Voltage ↗	Current ↗	Bad alignment
<b>Resolution</b> ↗↗↗ Depth of focus ↘	<b>Resolution</b> ↗↗ Tip effect ↗ <b>Charges</b> ↗↗ <b>Damages</b> ↗↗ Surface details ↘	<b>Resolution</b> ↘↘ <b>Charges</b> ↗↗ <b>Damages</b> ↗↗ Noise ↘	<b>Resolution</b> ↘↘ Noise ↗

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

# Influence of choices: Working Distance



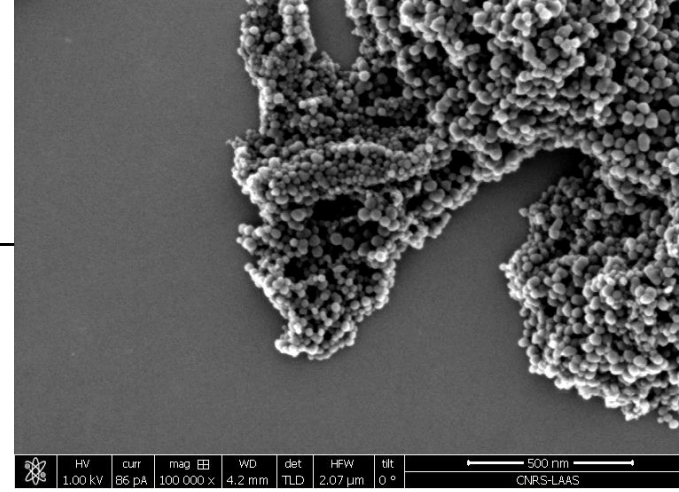
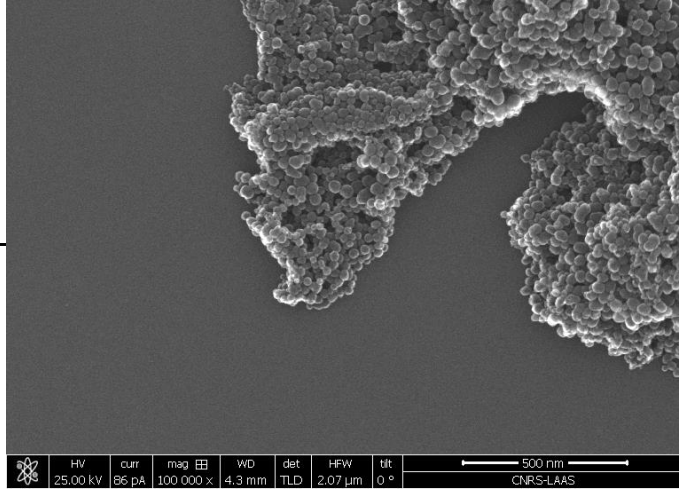
Long working distance



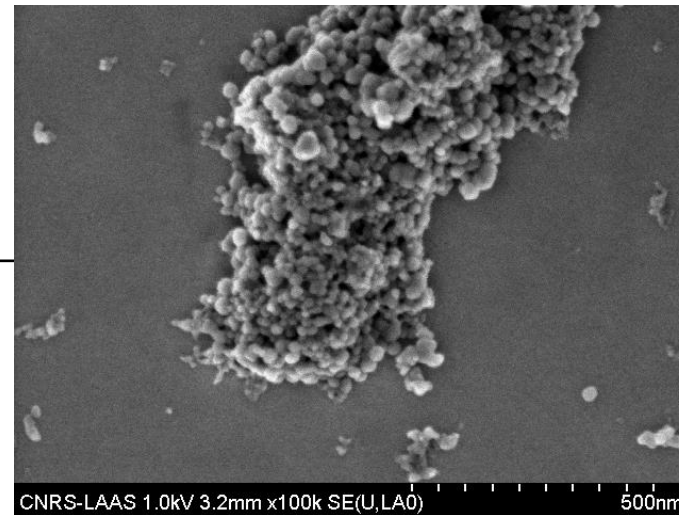
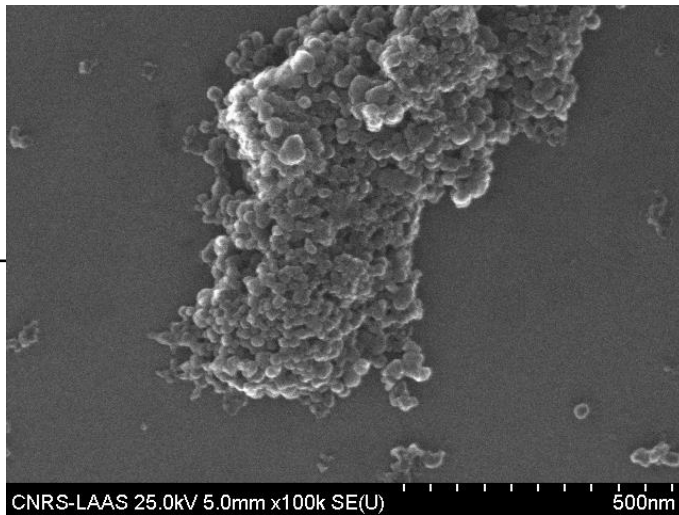
Short working distance  
High resolution

# Influence of choices: High Voltage

S-FEG



C-FEG

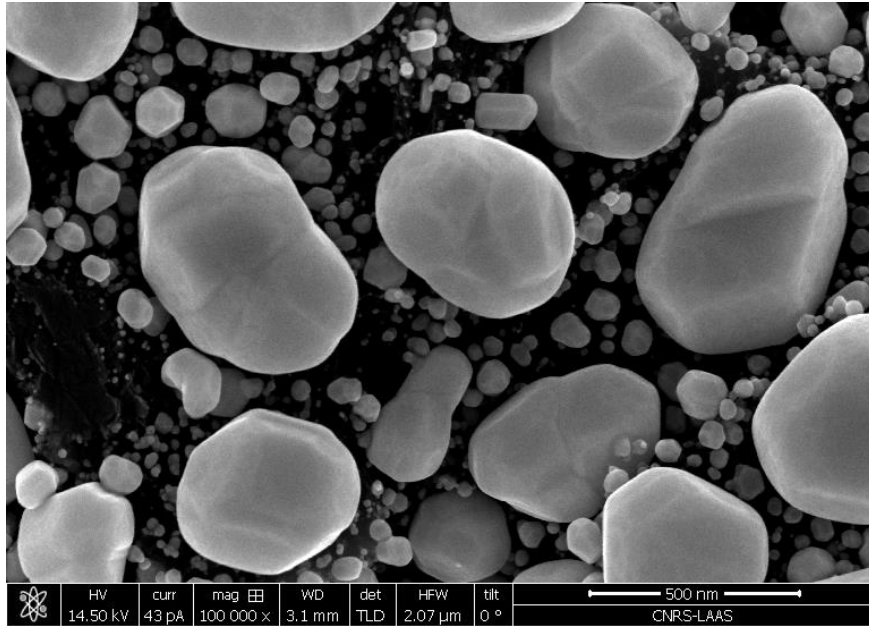


High accelerating voltage

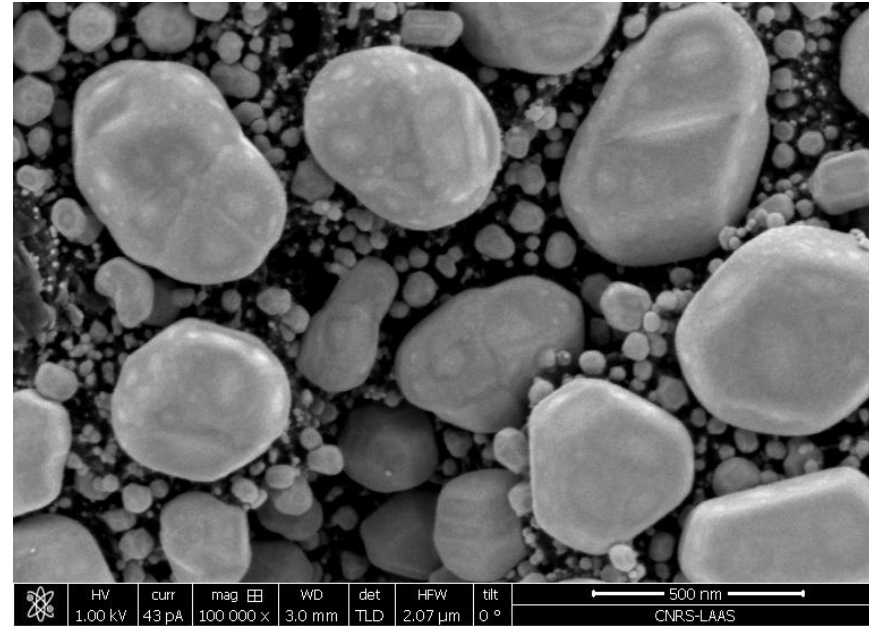
Low accelerating voltage

- Candle stick fumes:
- Same sample
  - ~ same parameters

# Influence of choices: High Voltage



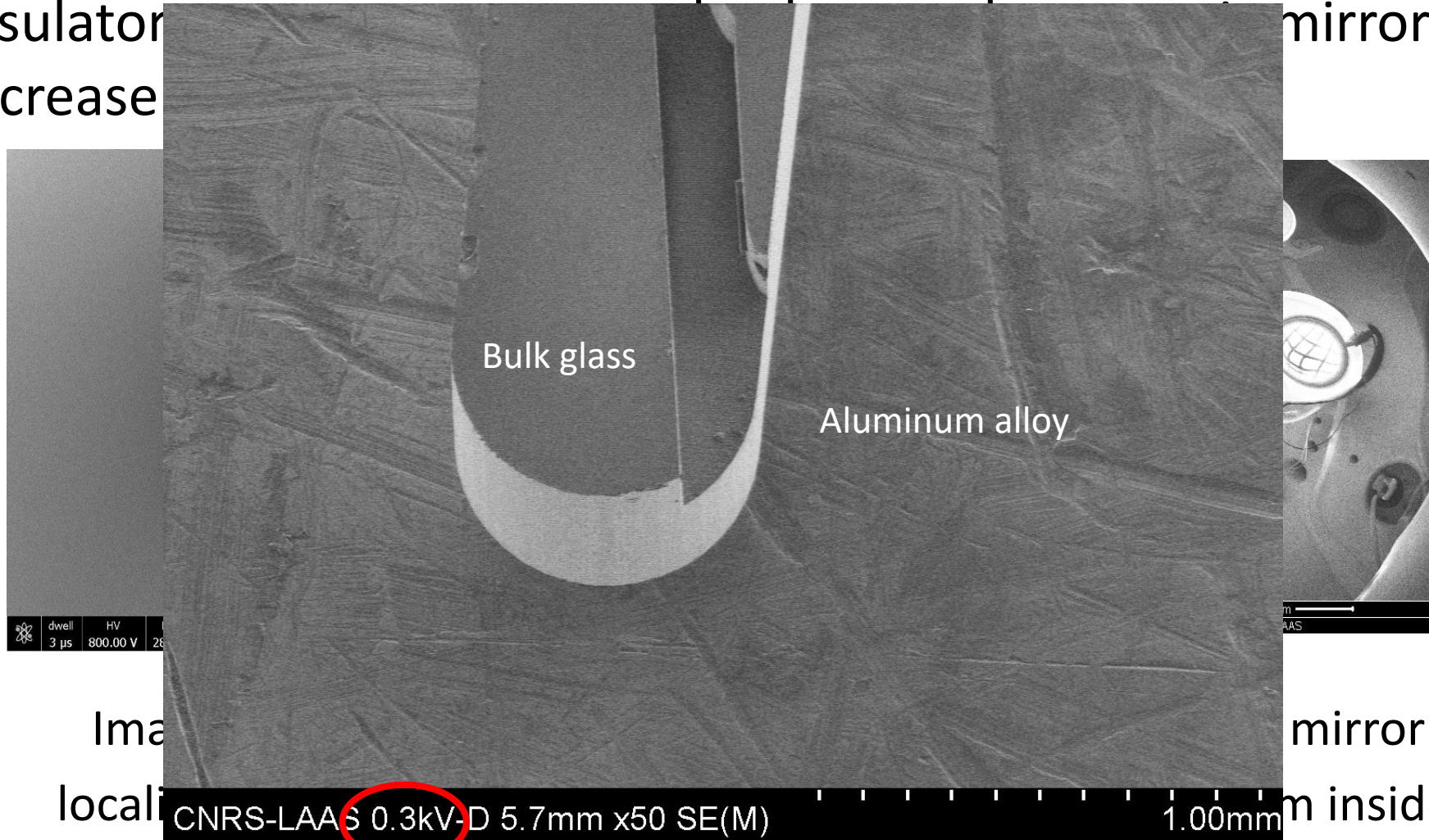
High accelerating voltage



Low accelerating voltage

# Influence of choices: charging effect

On insulator  
decrease



mirror

mirror:  
inside

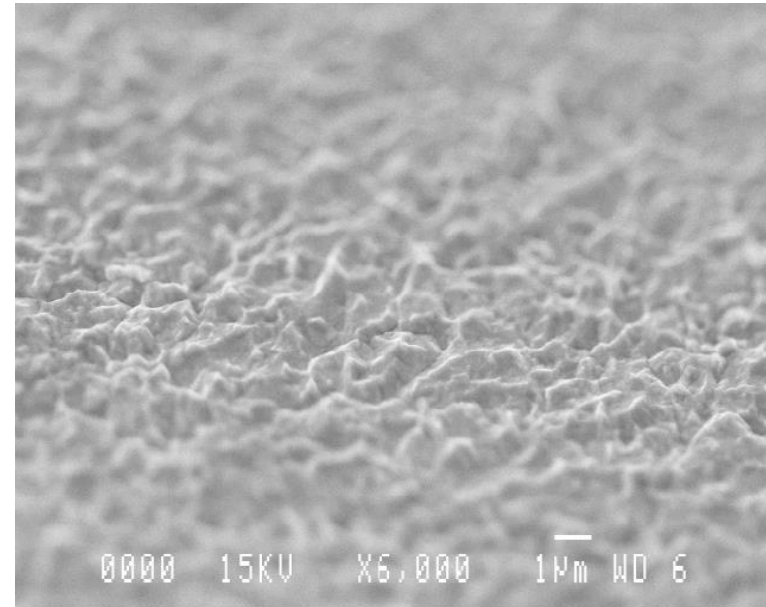
SEM in practice

# 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

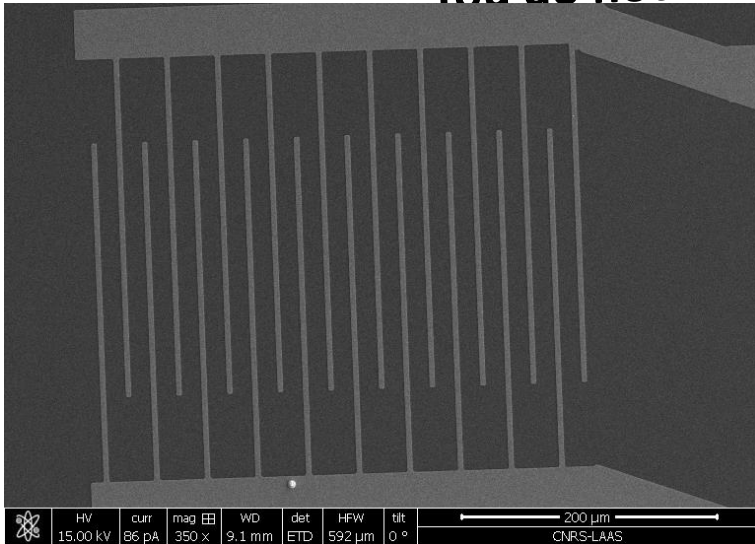
Example:

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

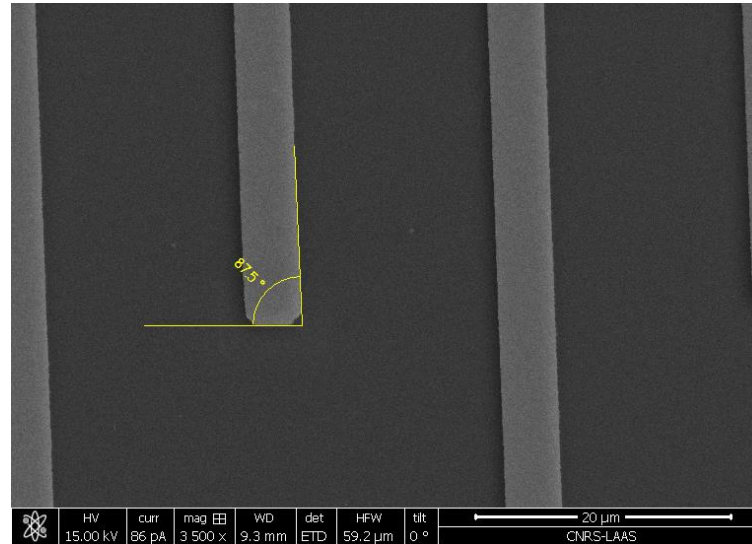




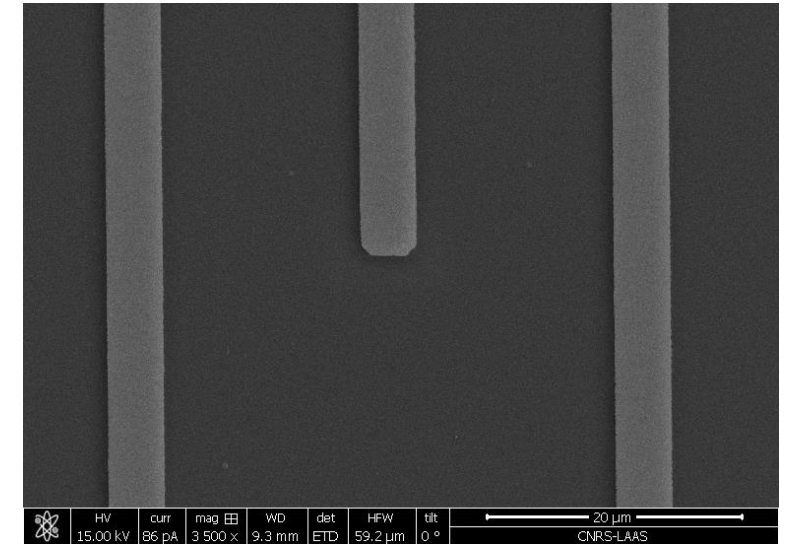
- Brain is organized in horizontal and vertical analysis
- When an image does not fit brain organization:
  - You induce constraints on the observer
  - You do not ease communication



Angle evaluation !?



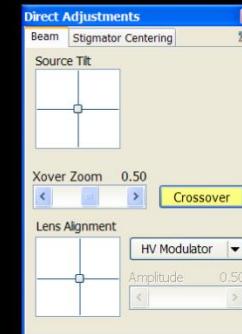
You are sensitive to  $<2.5^\circ$



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

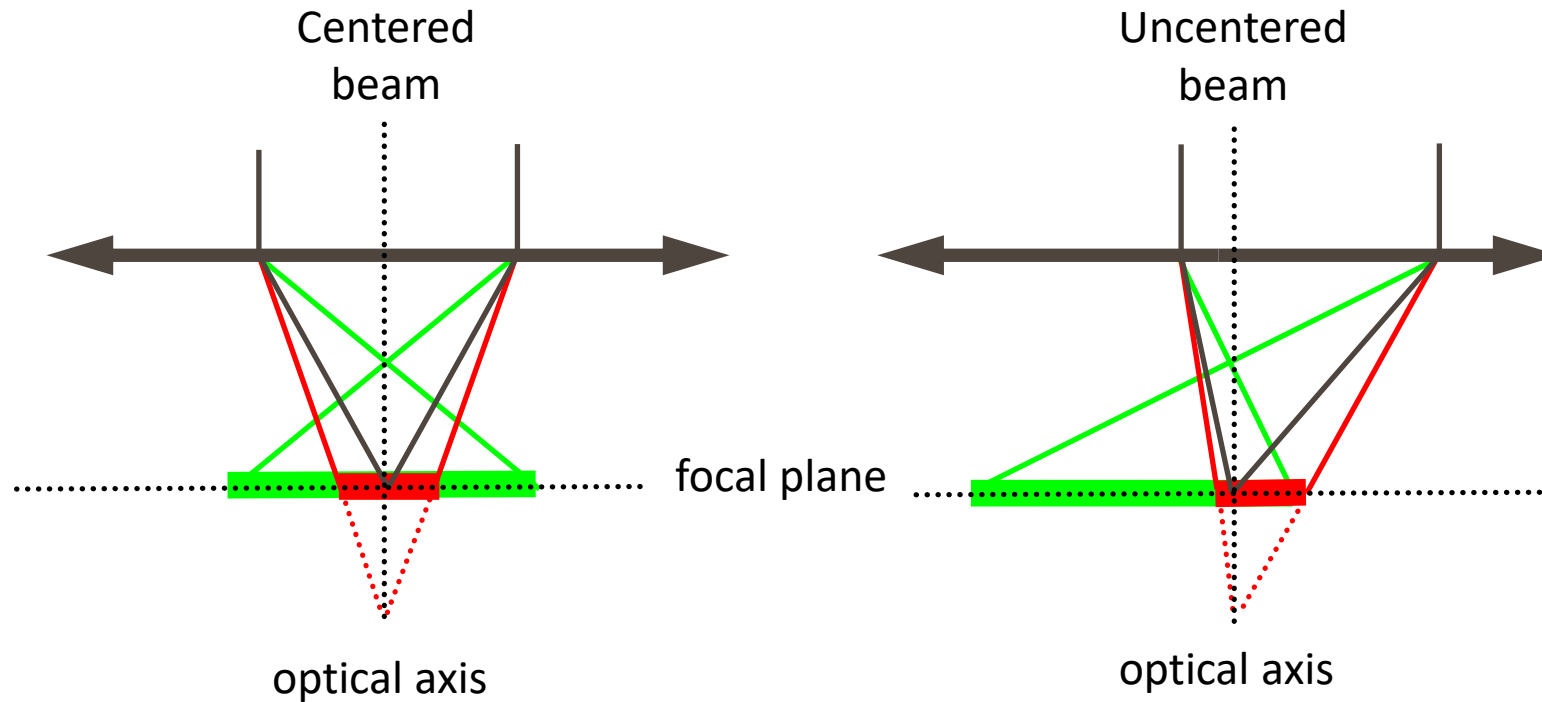
Defines optical axis

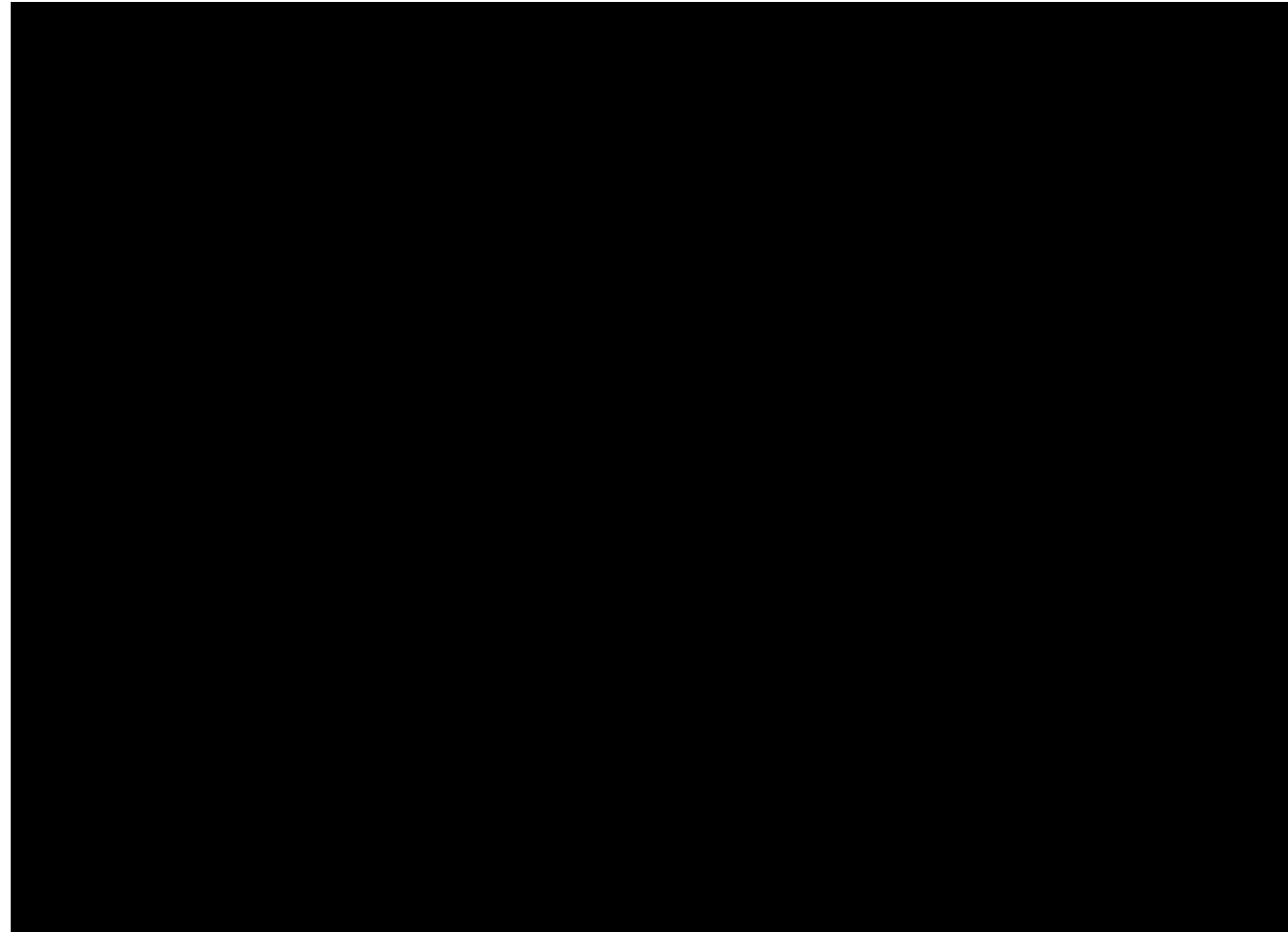


	HV	curr	mag	WD	det	HFV	tilt	500 $\mu$ m		Q1
	1.00 kV	86 pA	100 x	4.2 mm	ETD	2.07 mm	0 °			Act

- **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



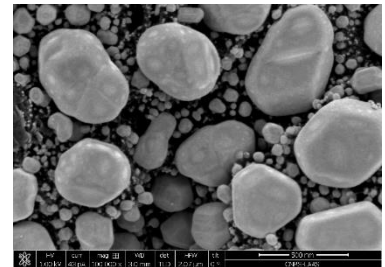
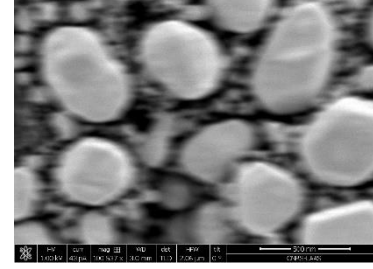
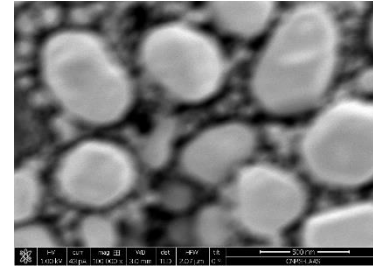
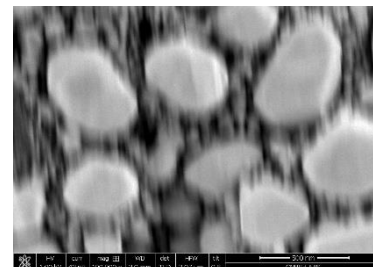
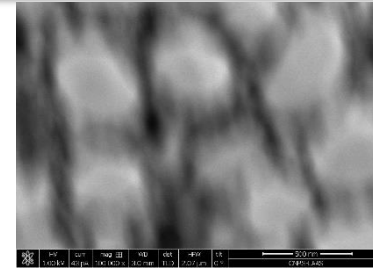
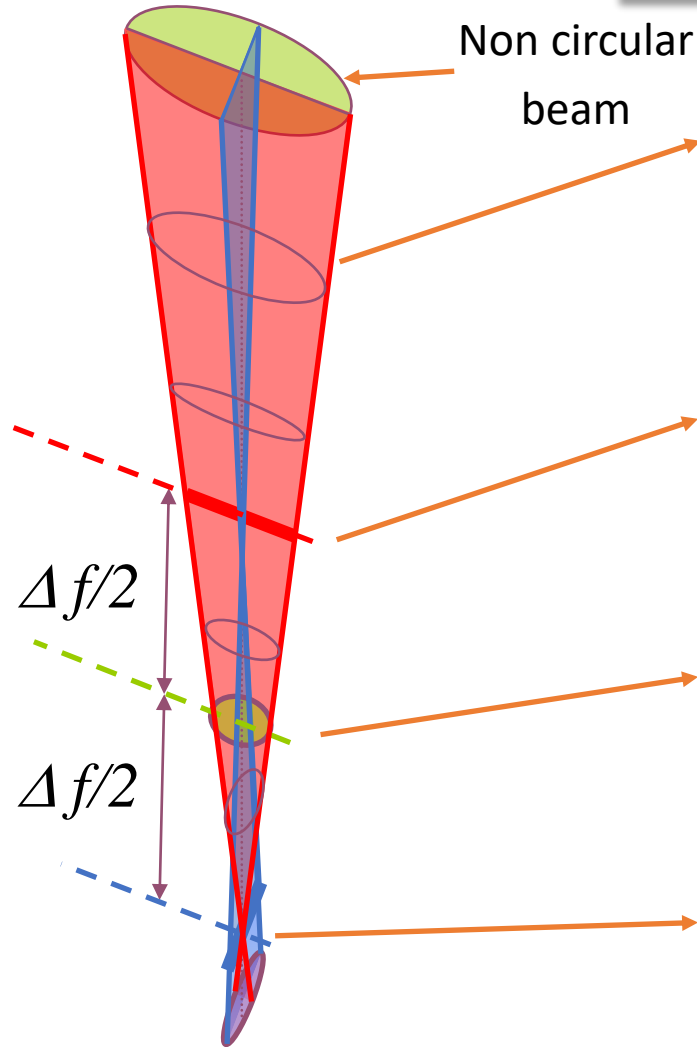


# Procedures: astigmatism (adjustable)

*sagittal focal*  
beam = line

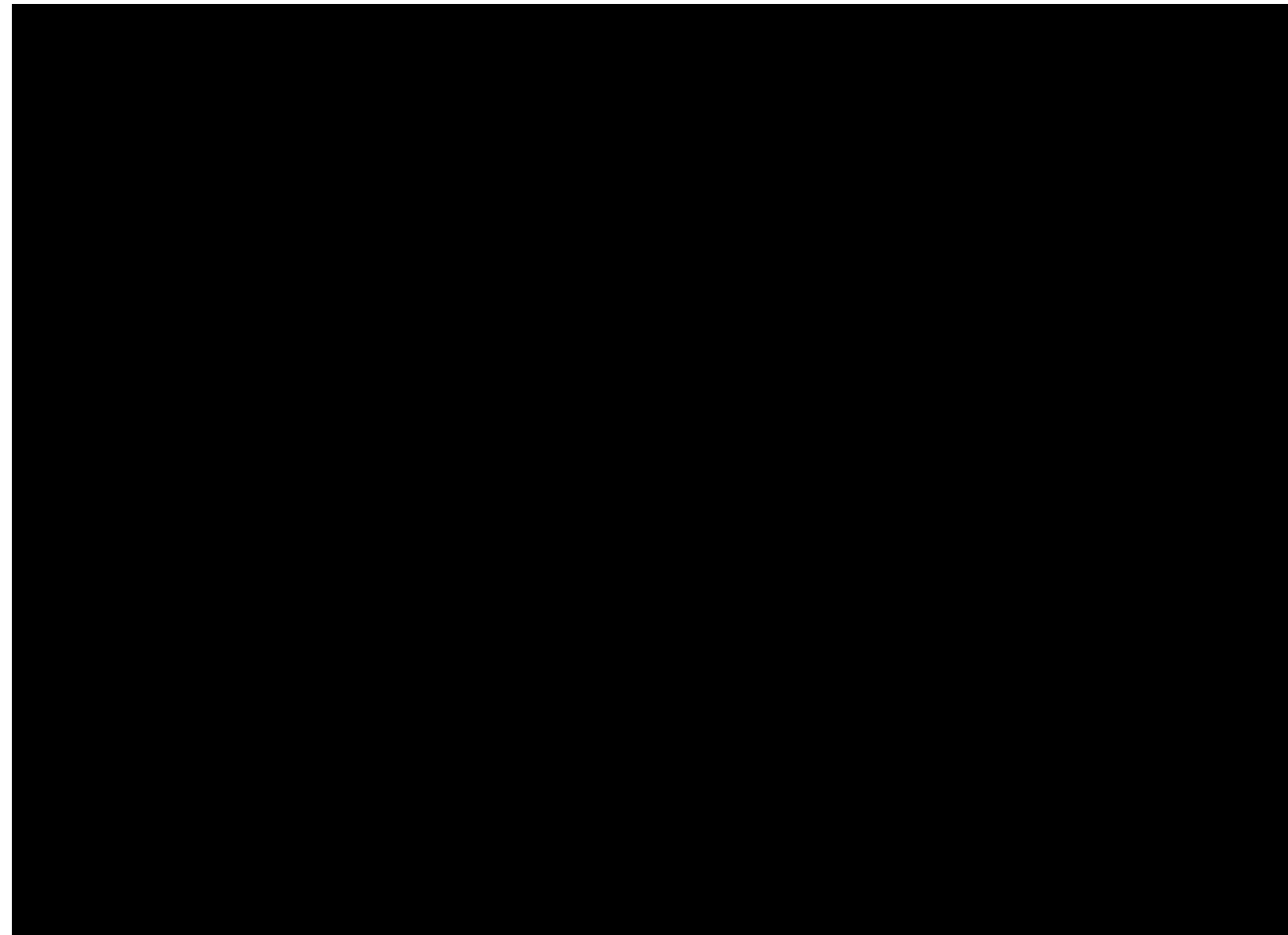
*main focal*  
beam = circle

*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)

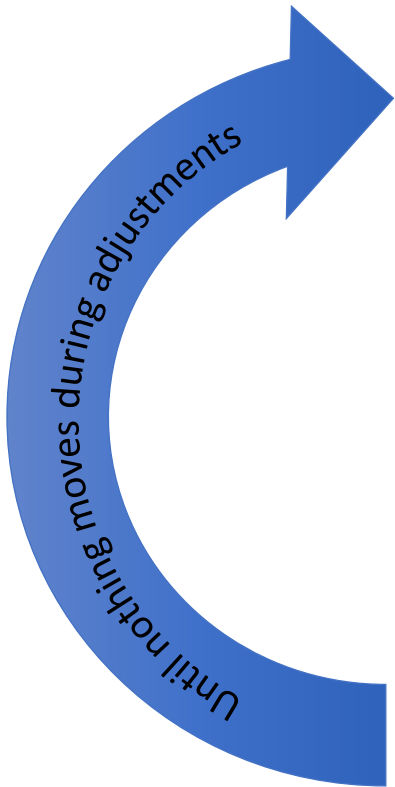
## If there is too much noise or no image: verify GUN alignment

- 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