5. modern SEM technology and trends

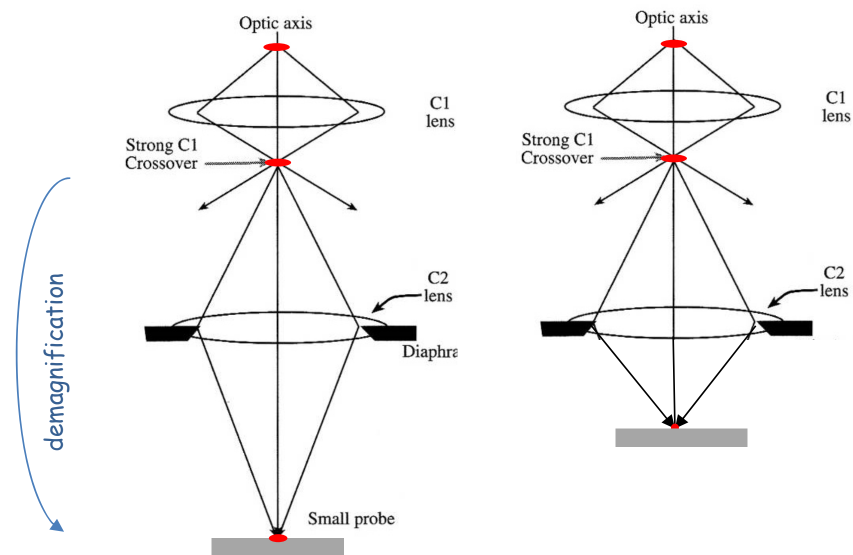
SEM

- Why low voltage SE imaging
- Condition of the surface, coatings, plasma cleaning
- Low voltage BSE imaging
- Polishing for BSE, EDX and EBSD, effect of ion beam etching/polishing

Spherical aberration as limiting factor for small probe sizes

Otto Scherzer:
Spherical aberration arises because the focus depends on the axial distance of the electron path. Physically this is a consequence of the fact, that lens fields have to satisfy the Laplace equation.
The aberration coefficient is substantially of the same order of magnitude as the **focal length**. If the specimen is immersed in the lens field, the minimum value of the aberration coefficient is **half of the focal length**. It diminishes when stronger lenses are used.

typical «2 condensor» lens system

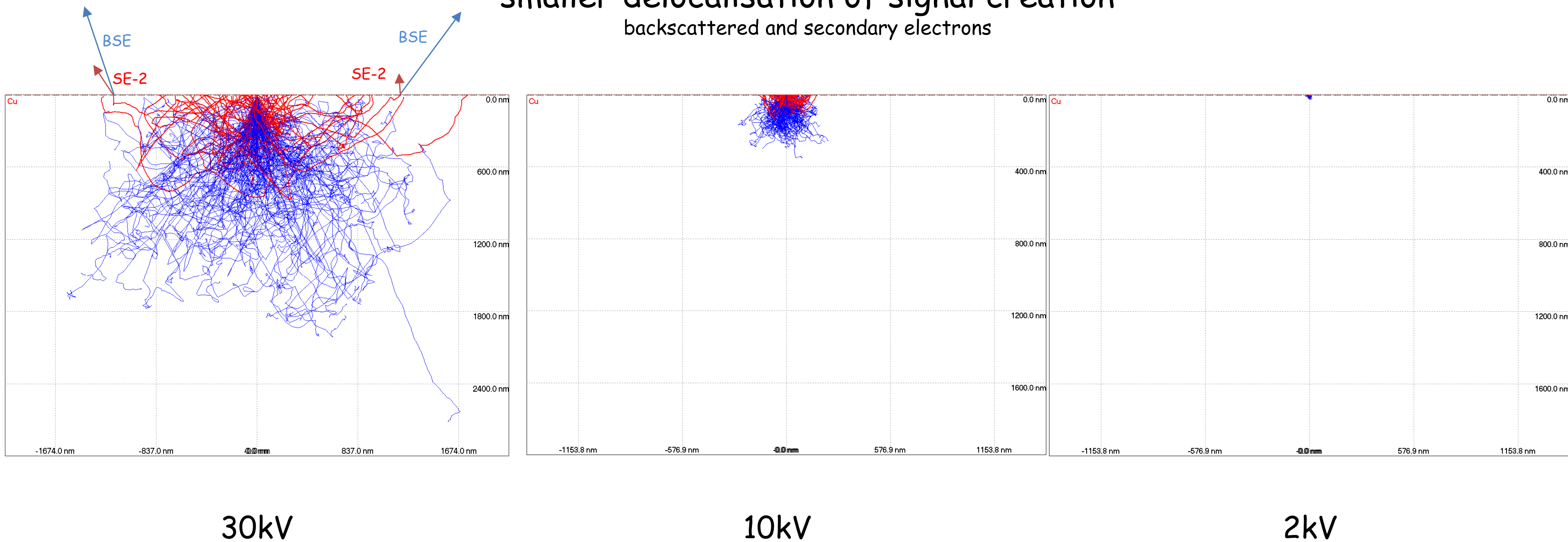


strong fields, small working distances, immersion lenses

interaction of electrons with sample (Cu)

smaller delocalisation of signal creation

backscattered and secondary electrons



30kV 10kV 2kV

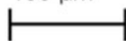
Low acceleration voltages !

ETD

BSD

Position of detectors

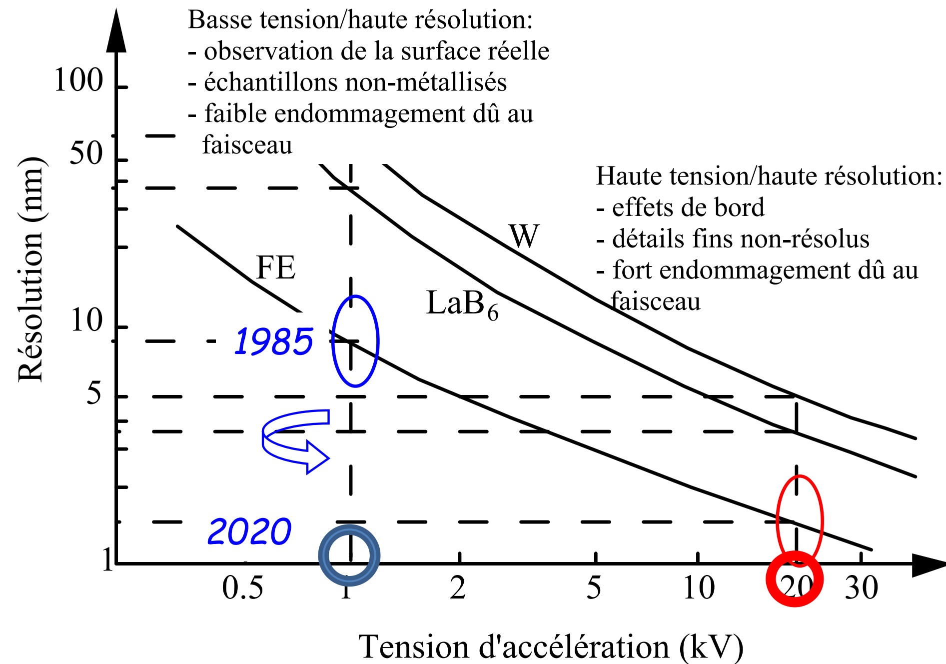
Mag = 206 X	EHT = 2.00 kV	Signal A = USB TV2	Chamber Status = at HV
100 µm	WD = 7.1 mm	Aperture Size = 30.00 µm	Chamber = 0.00e+00 Pa
	Image Pixel Size = 1.340 µm	Stage at T = 0.0 °	Date :16 Jan 2018



Latest generation of SEM

Electron optics:
Field emission guns, monochromators
beam boosters,beam deceleration,
Lens-design: In-lens, Semi-in-lens,
immersion lens
Short working distance

Detectors:
Everhard-Thornley (SE)
In-column (through the lens, in-lens,
“in-beam”) SE detectors
Low Voltage BSE detection,
energy filtering (separation of
materials and topography contrast)

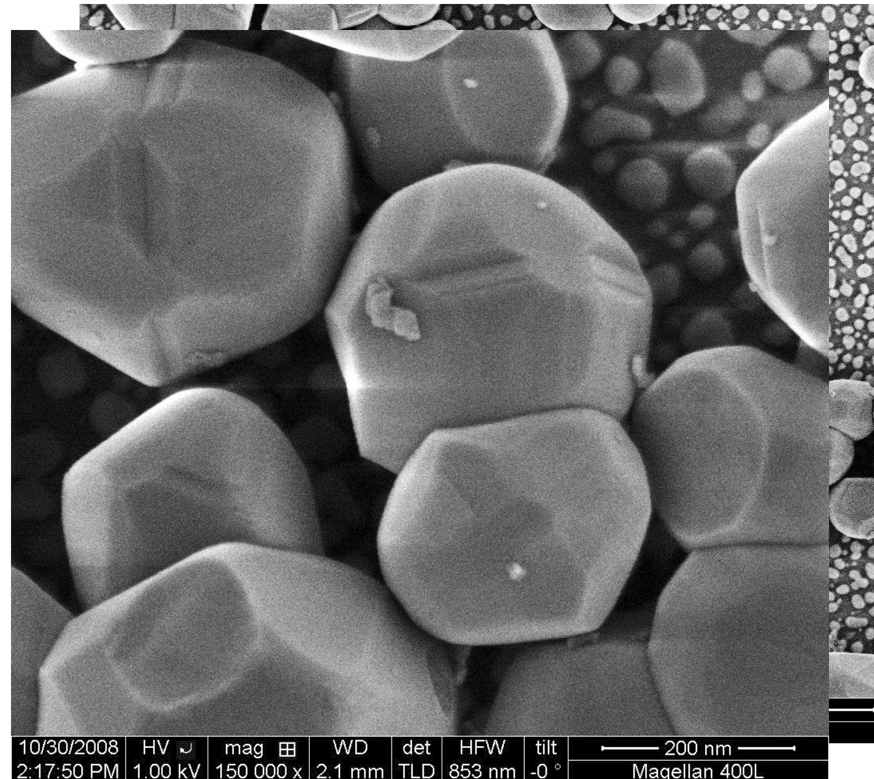


Analytical SEM:
SDD EDX detectors (high throughput, large collection angle)
High-speed EBSD detectors
Beam currents of several 100 nA

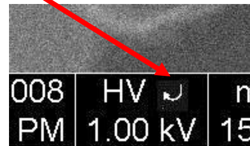
SEM low kV imaging

No specimen preparation needed: low voltage reduces charging effects
Low kV imaging of non-conducting, low density samples

Al2O3 Nano-crystals



Technology: stage bias
(beam deceleration between pole piece and sample)

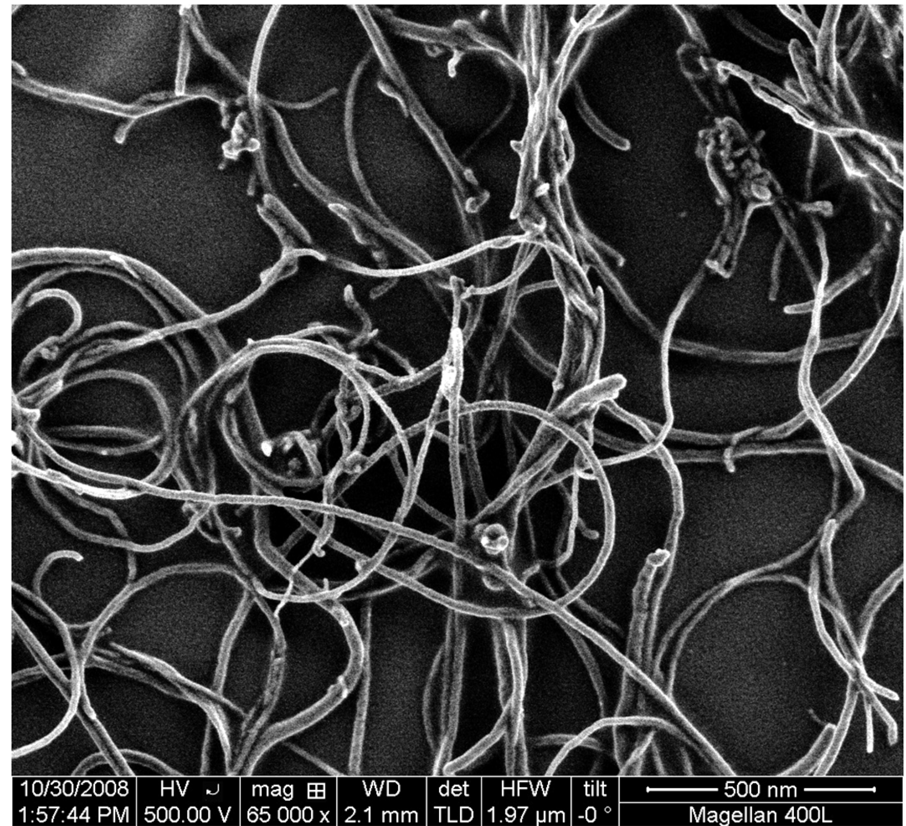


FEI Magellan
Operator: Ingo Gestmann
Samples: Marco Cantoni

SEM low kV imaging

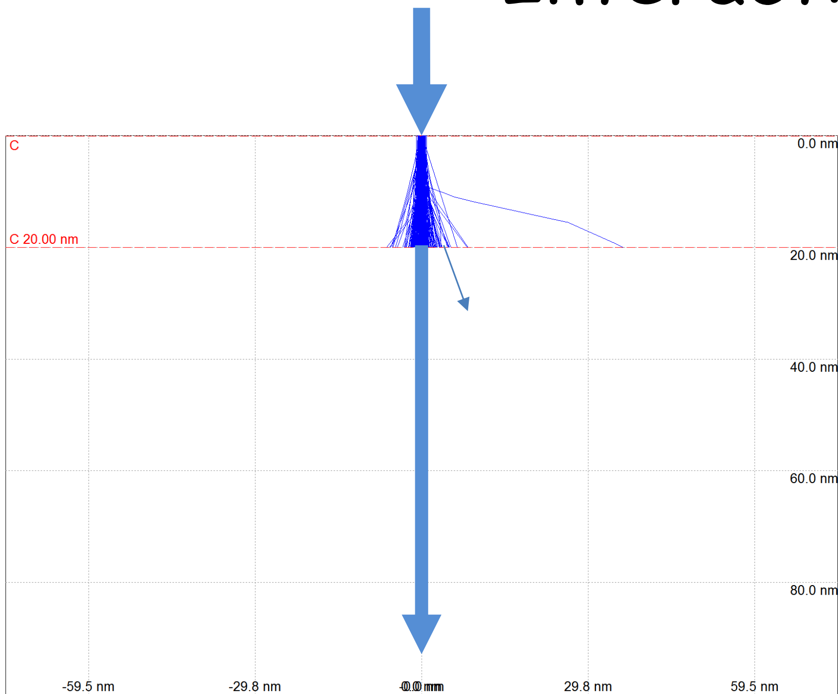
No specimen preparation needed:
Low kV imaging of low density samples

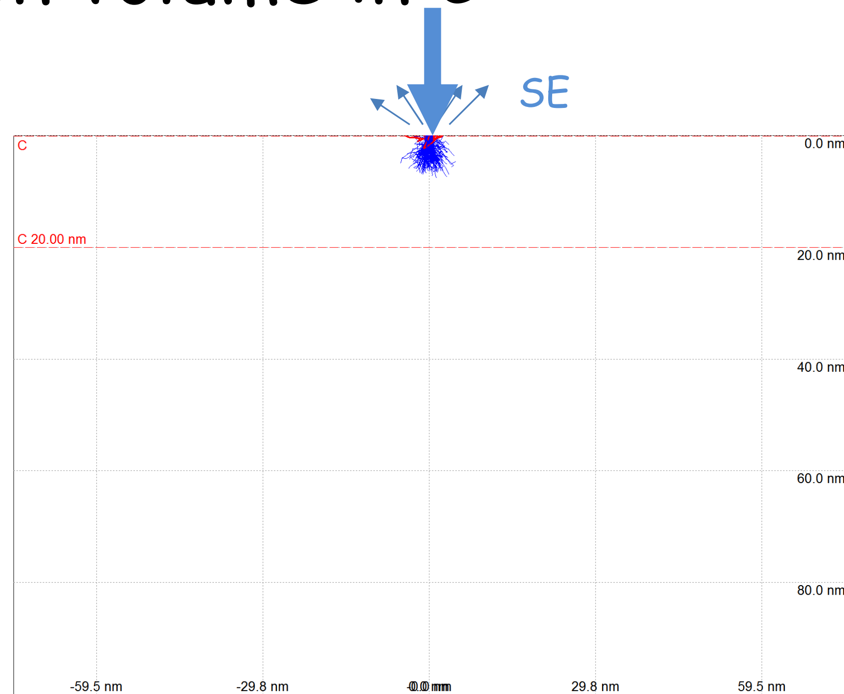
Carbon nano-tubes
(MWCT)



FEI Magellan
Operator: Ingo Gestmann
Samples: Marco Cantoni

Interaction volume in C





5kV

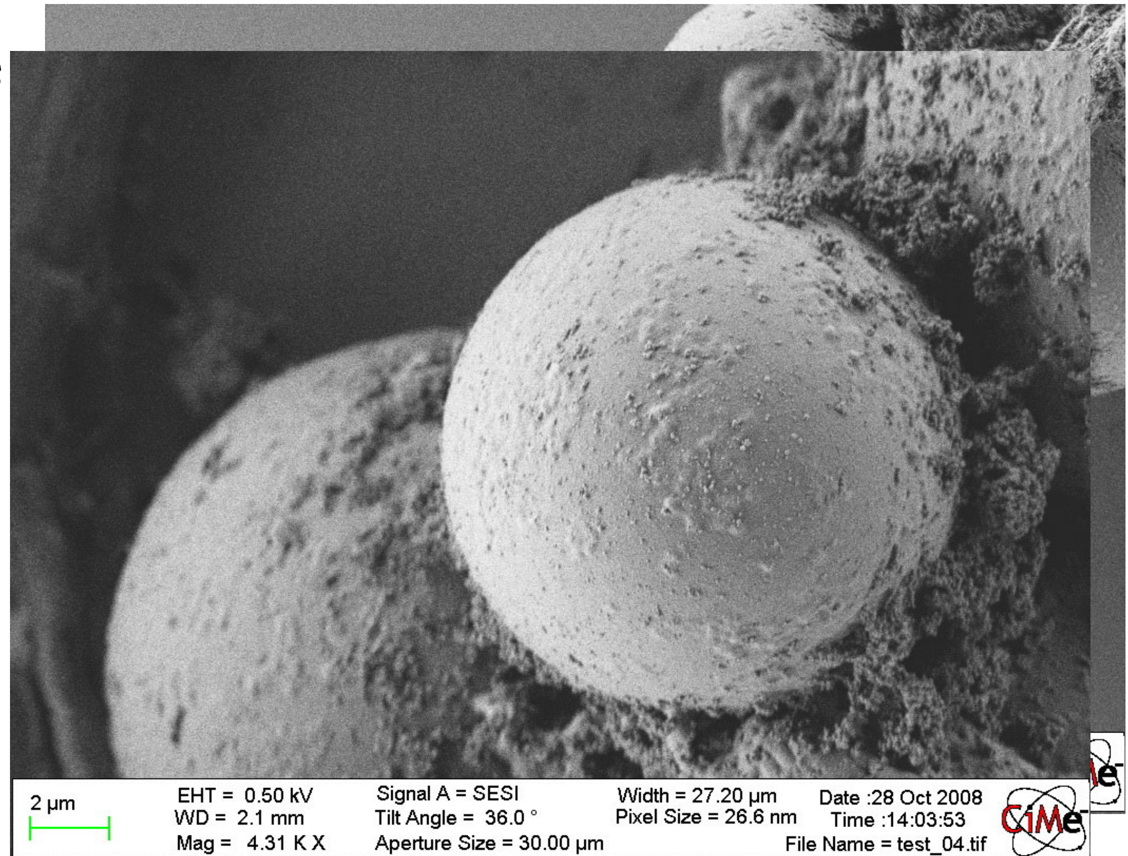
Electrons are transmitted through nano-tube
almost no interaction, very few SE !
almost invisible

0.5kV

Electrons interact heavily with nano-tube:
Lots of SE

SEM low kV imaging

No specimen preparation needed:
Low kV imaging of non-conducting samples

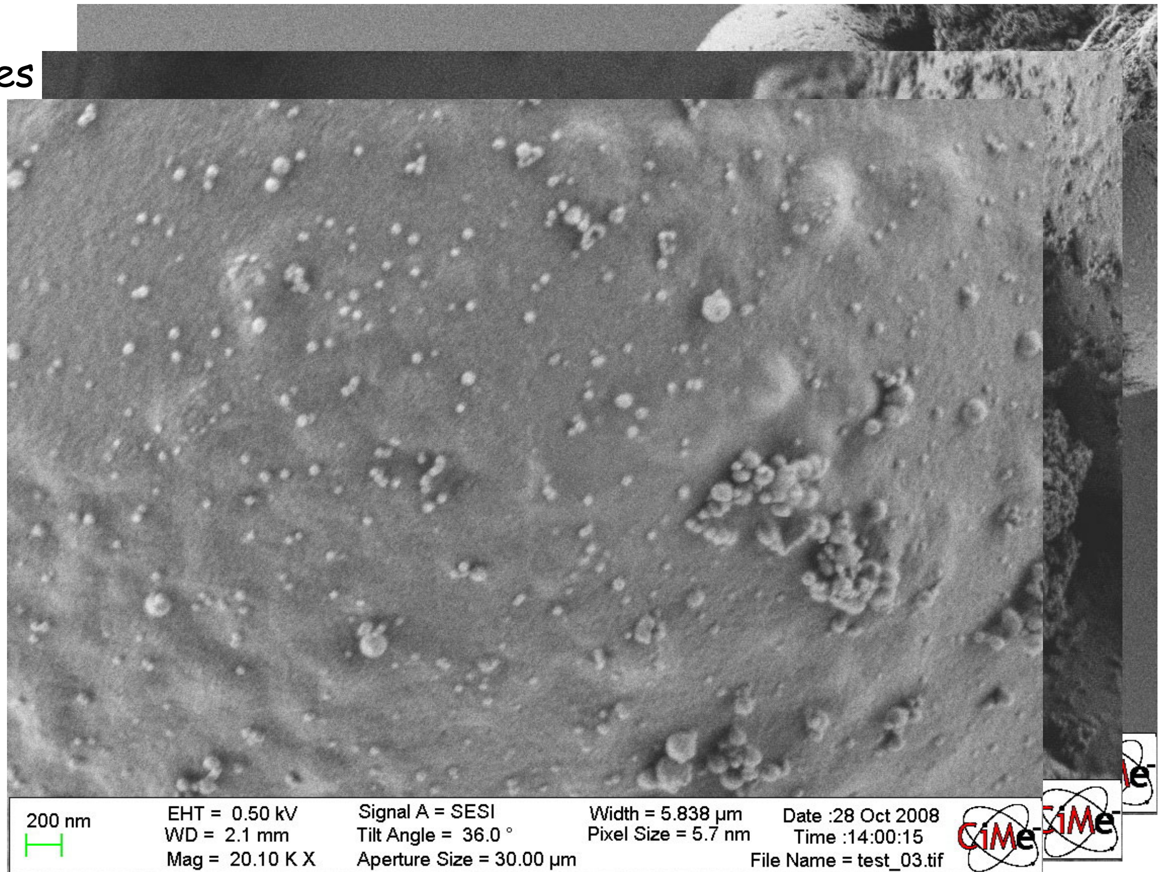


Liquid filled
organic membranes

Zeiss Nvision 40
Marco Cantoni

SEM low kV imaging

No specimen preparation needed:
Low kV imaging of non-conducting samples

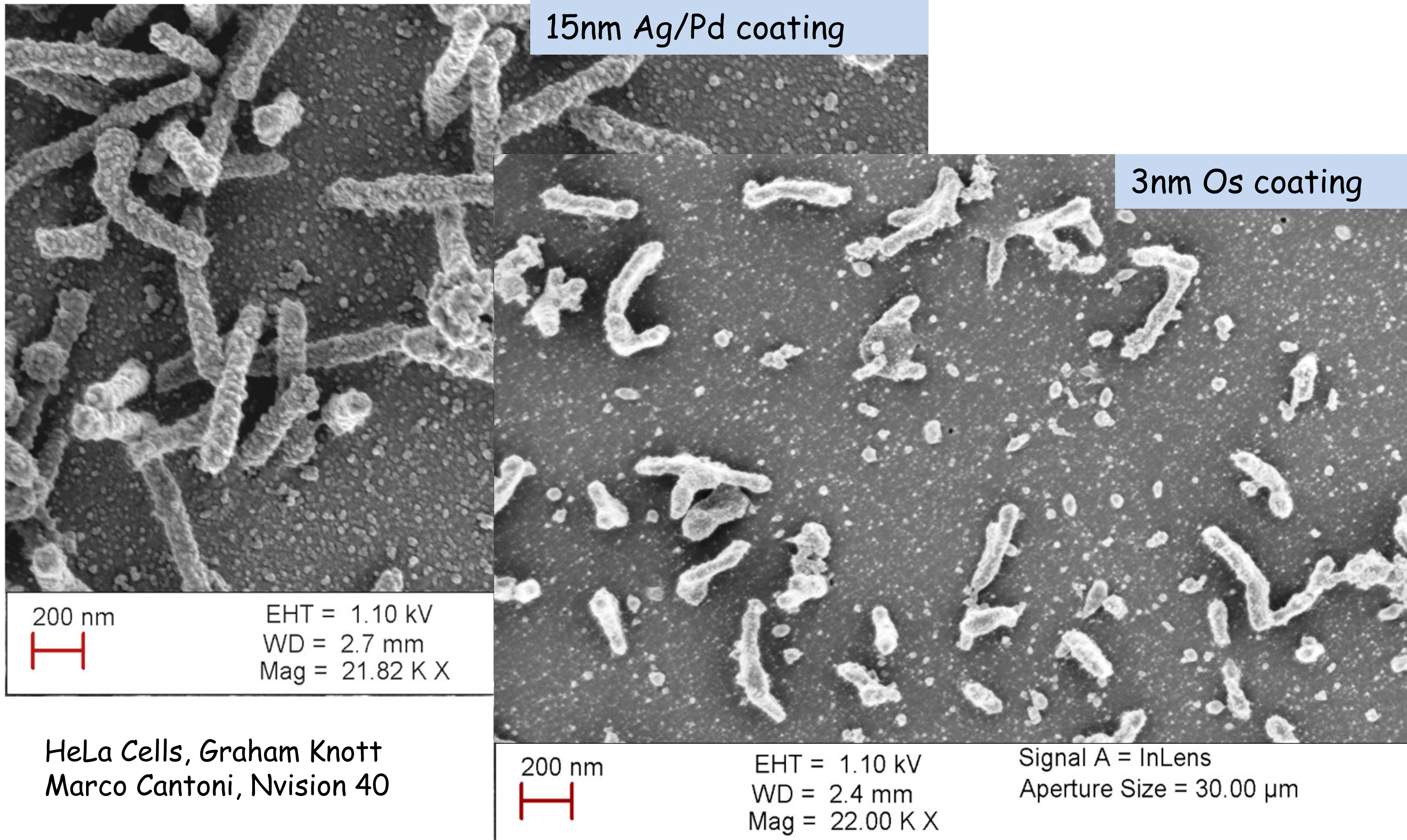


Liquid filled
organic membranes

Zeiss Nvision 40
Marco Cantoni

SEM low kV imaging

Purely organic specimen: non-conductive, low density: biological cells, thin (!) metal coating



HeLa Cells, Graham Knott
Marco Cantoni, Nvision 40

MERLIN™ – Analytical power for the sub-nanometer world

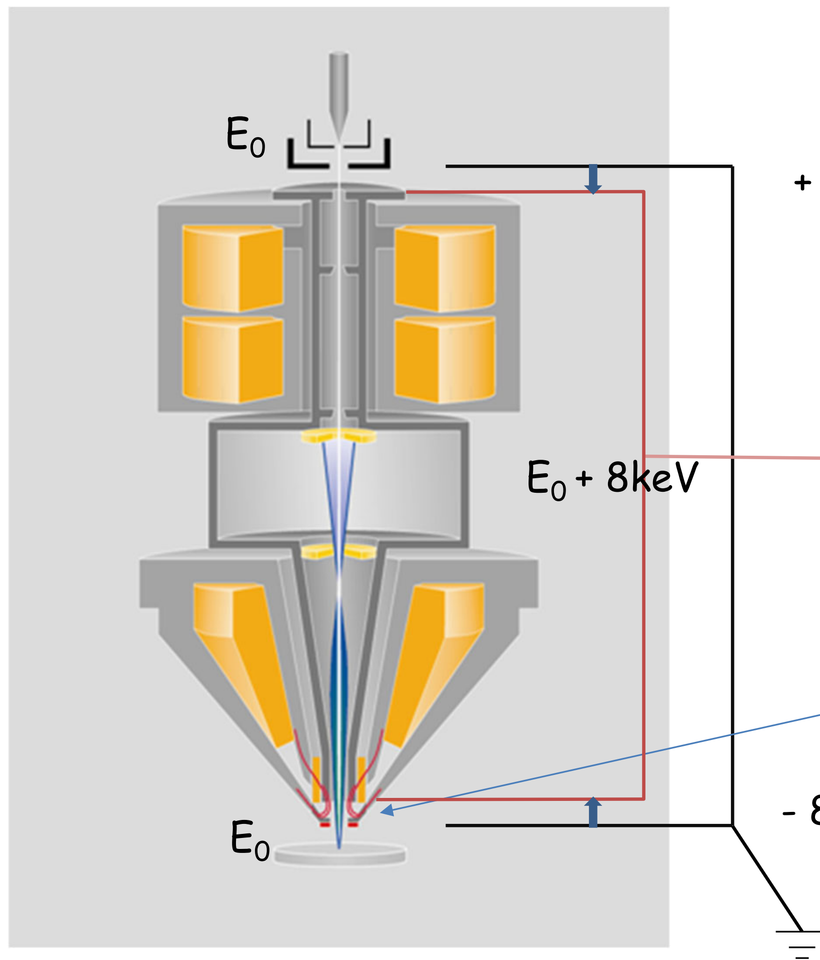
Gemini® II column

Low current configuration (Max. probe current 40 nA):

For high resolution investigations:

- 0.6 nm at 30 kV (STEM mode)
- 0.8 nm at 15 kV at optimal WD
- 1.4 nm at 1 kV at optimal WD
- 2.4 nm at 0.2 kV at optimal WD
- 3.0 nm at 20 kV at 10 nA @ WD = 8,5 mm

Beam “booster” technology
Electron optics operates with high energy beam inside the column



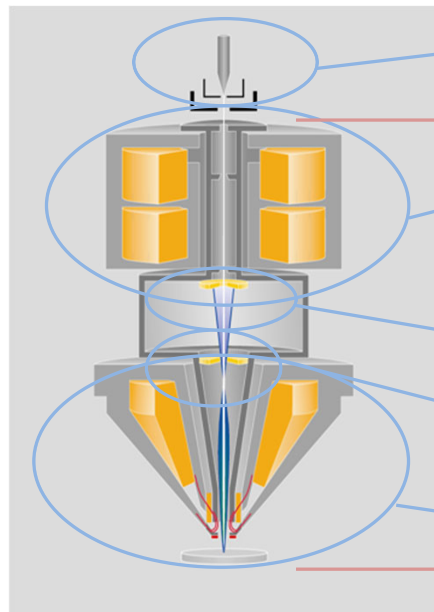
+ 8 keV

- ▪ Beam Booster 8keV
- ▪ Brightness of the electron probe maintained for low landing energies

- ▪ GEMINI® II electro-static final lens

- 8 keV

MERLIN™ – Analytical power for the sub-nanometer world –



- High stability field emitter cathode
- Maximum probe current 300 nA

- Double condenser lens
- Aperture independent probe current adjustment

- Beam Booster
- Brightness of the electron probe maintained for low landing energies

- Energy selective Backscatter detector (EsB)

- In-lens Secondary Electron detector

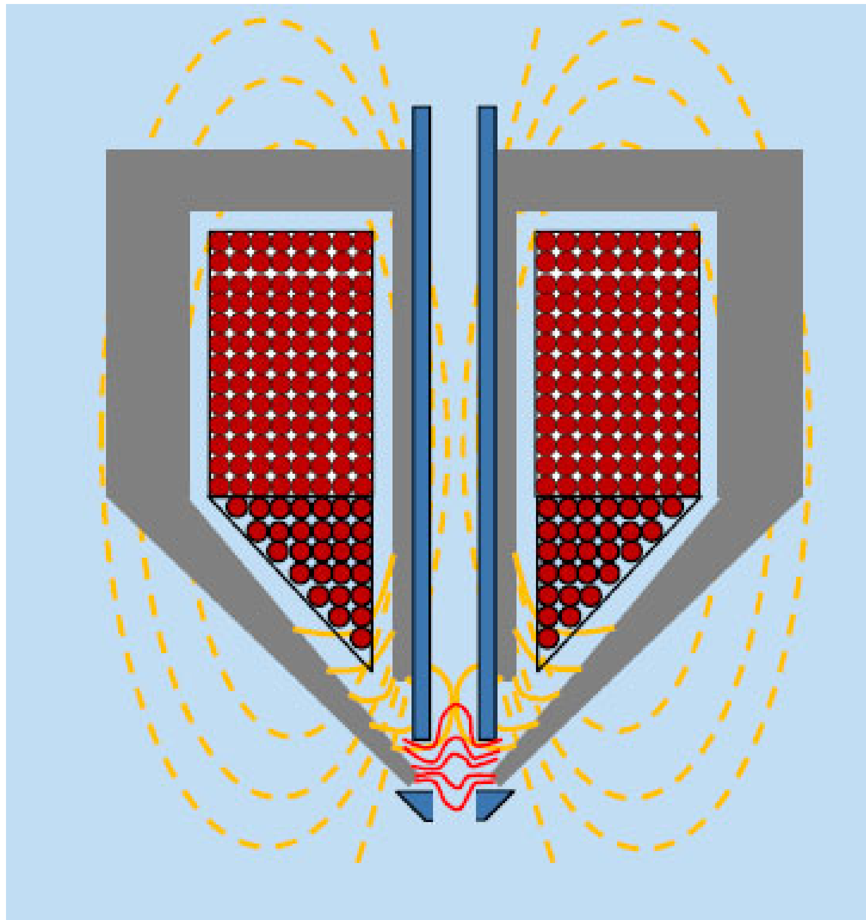
- GEMINI® II final lens

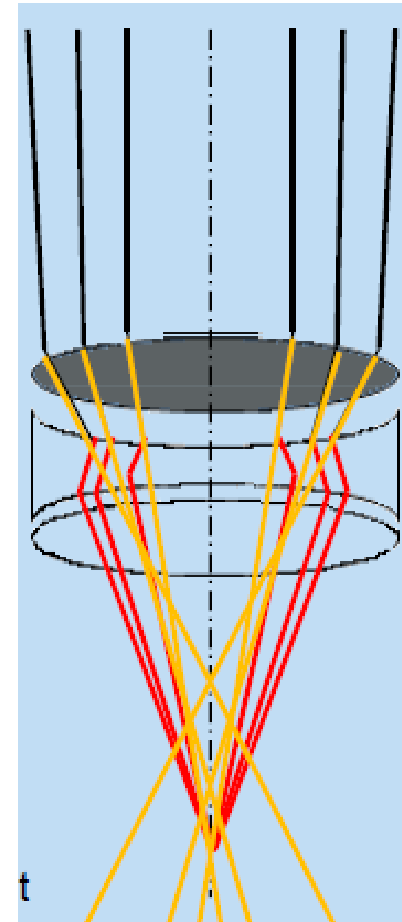
▶ **GEMINI® II design**
"Complete" detection system

- Proven GEMINI® final lens design
- New double condenser lens for highest probe current possibilities (300 nA)
- Beam booster technology maintains brightness of all electron probes including low landing energies
- True on-axis in-lens SE and BSE detectors

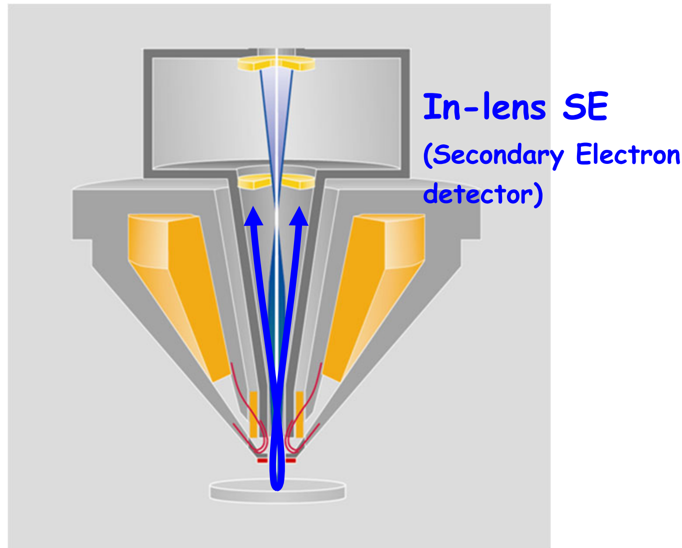
« gemini »-lens:

combination of magnetic (convergent) and electro-static (divergent = neg Cs) lens

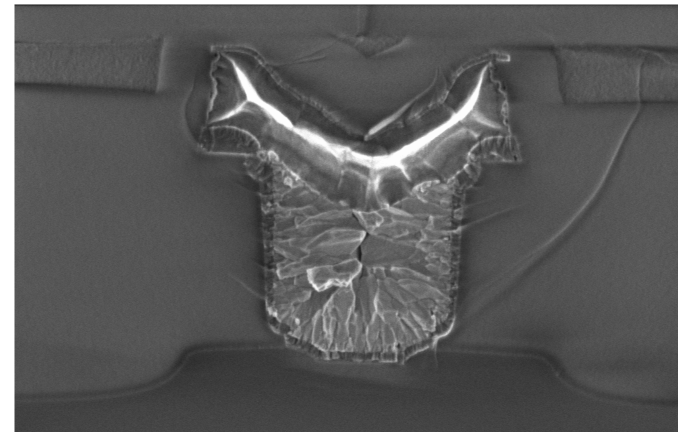




MERLIN™
– Analytical power for the sub-nanometer world –



Topographical information
with on-axis in-lens SE detector

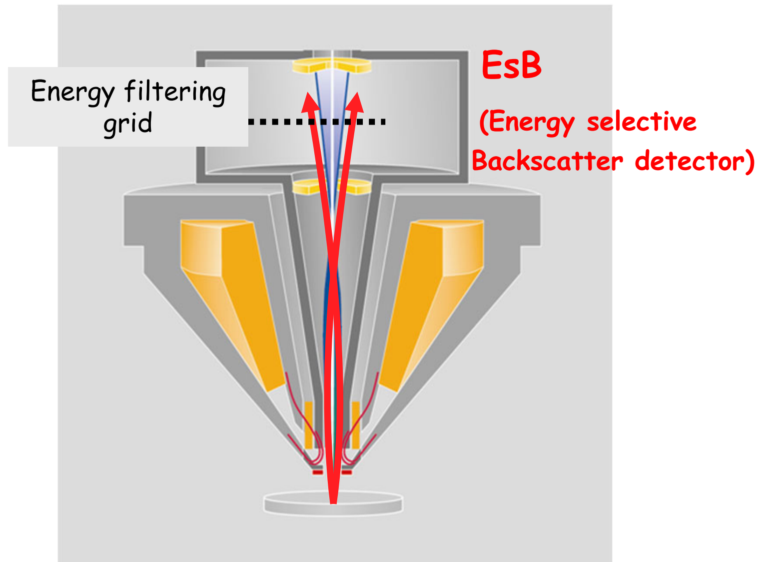


GEMINI® II design
► **Complete detection system**

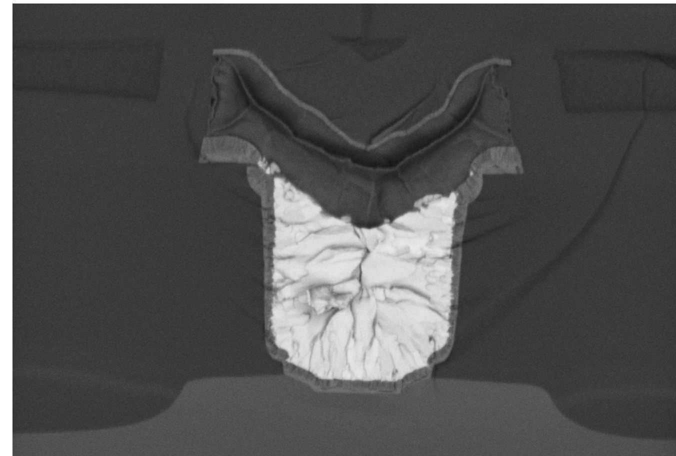
Complete detection system:

- Unique double in-lens detection
- Acquisition of pure secondary and backscatter electron signals
- Separation of compositional, topographical and crystalline surface information

MERLIN™
– Analytical power for the sub-nanometer world –



Compositional contrast
with on-axis in column EsB detector



GEMINI® II design
► Complete detection system

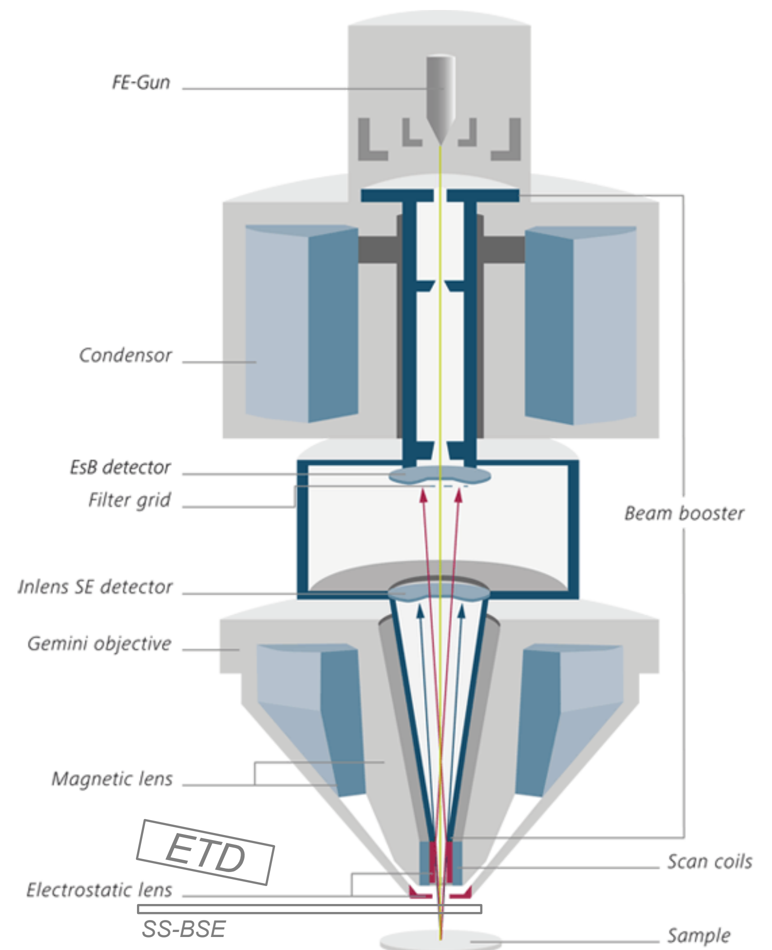
Complete detection system:

- Unique double in-lens detection
- Acquisition of pure secondary and backscatter electron signals
- Separation of compositional, topographical and crystalline surface information

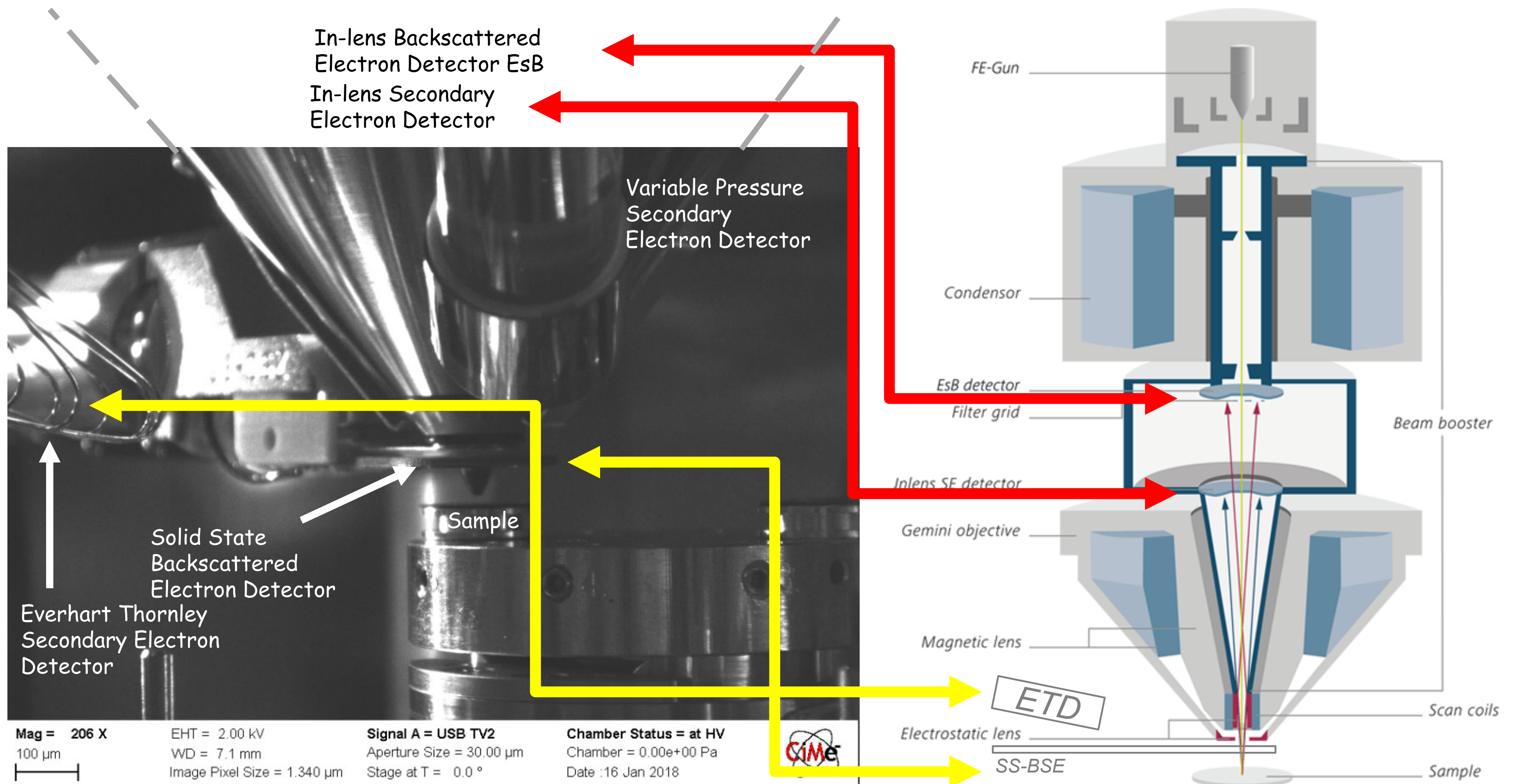
Secondary Electrons and Backscattered Electrons

In-lens Backscattered
Electron Detector EsB

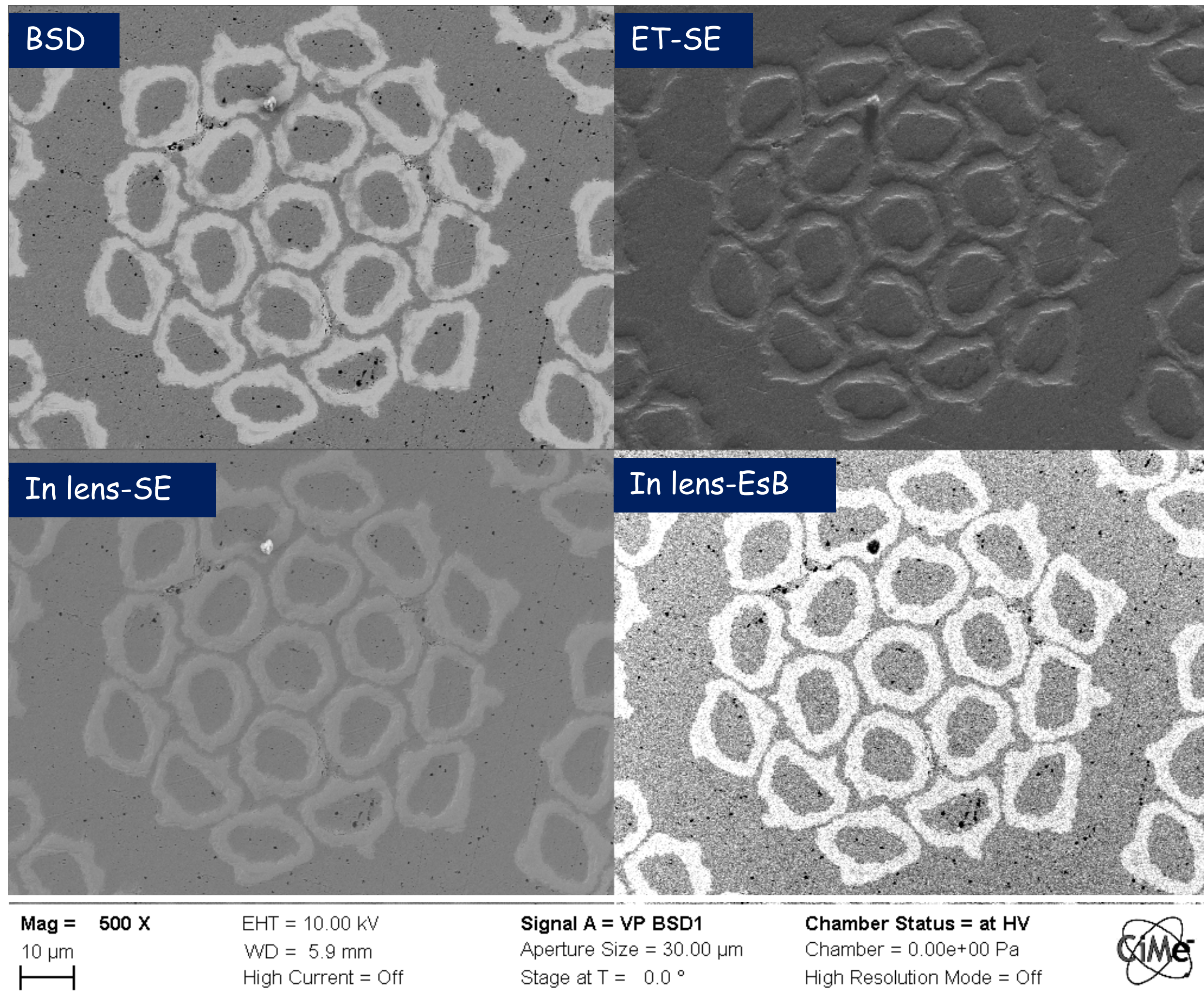
In-lens Secondary
Electron Detector



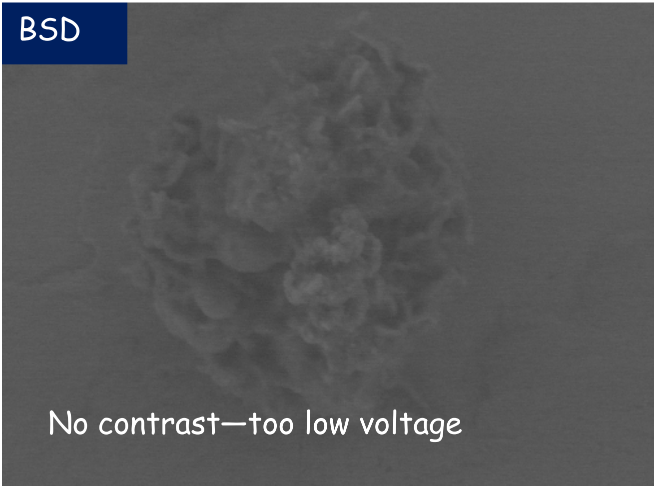
Secondary Electrons and Backscattered Electrons

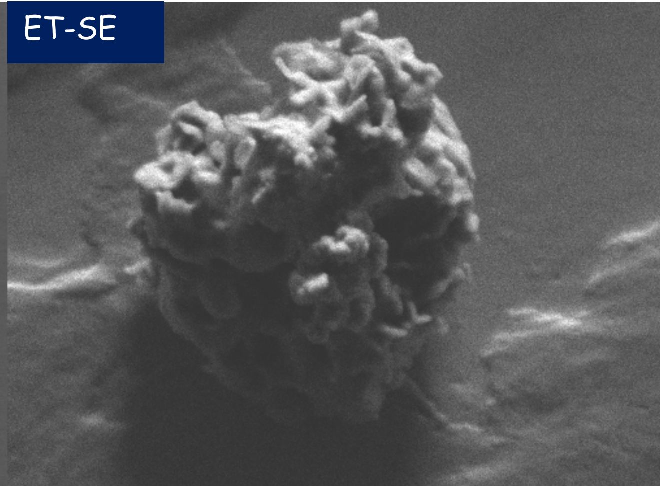


Secondary Electrons (SE) and Backscattered Electrons (BSE) images @10kV



Soild state
BSE detector:
Too weak
signal
No efficiency

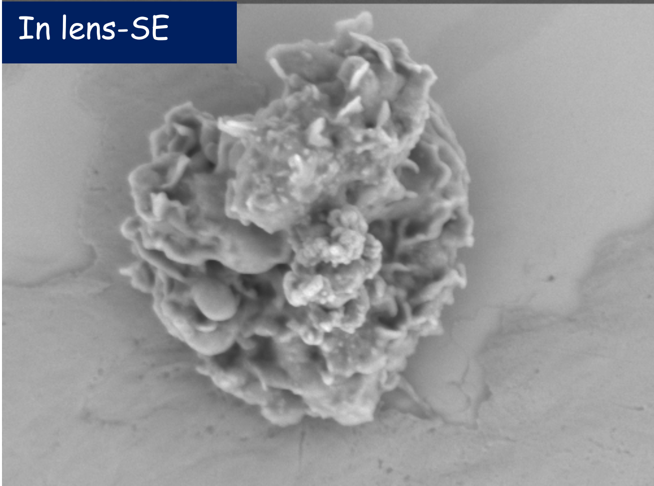


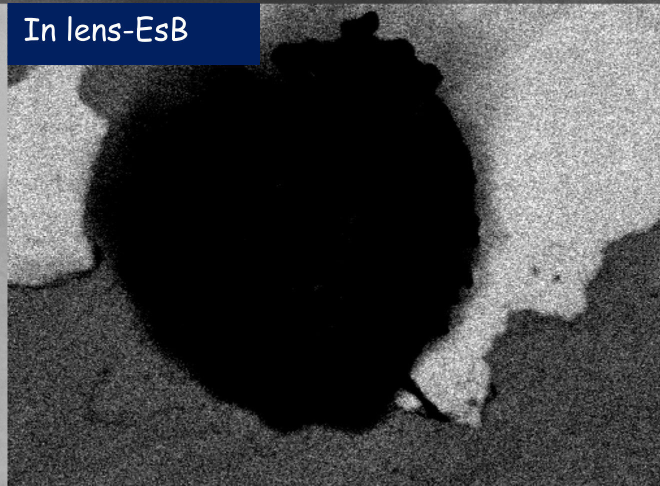


ETD:
“illumination” or
“shadow”effect

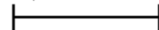
2kV

In-column
SE-detector:
topography,
“no illumination”
effect

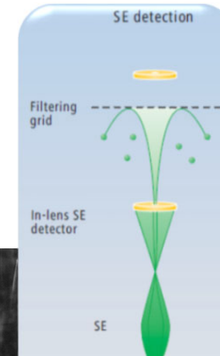


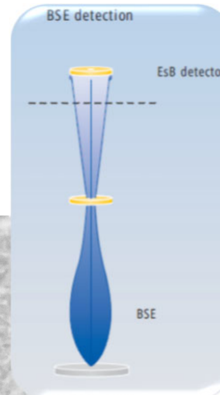


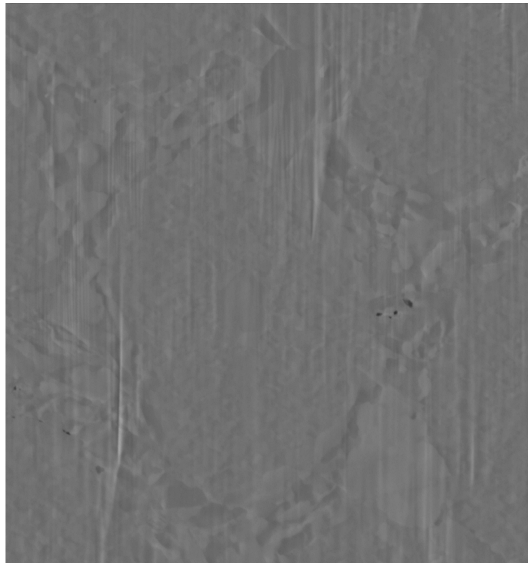
In-lens Energy
Selective
Backscatter
detector:
Almost pure
mass density
contrast

Mag = 12.50 K X
1 µm

EHT = 2.00 kV
WD = 6.0 mm
High Current = Off
Signal A = VP BSD1
Aperture Size = 30.00 µm
Stage at T = 0.0 °
Chamber Status = at HV
Chamber = 0.00e+00 Pa
High Resolution Mode = Off

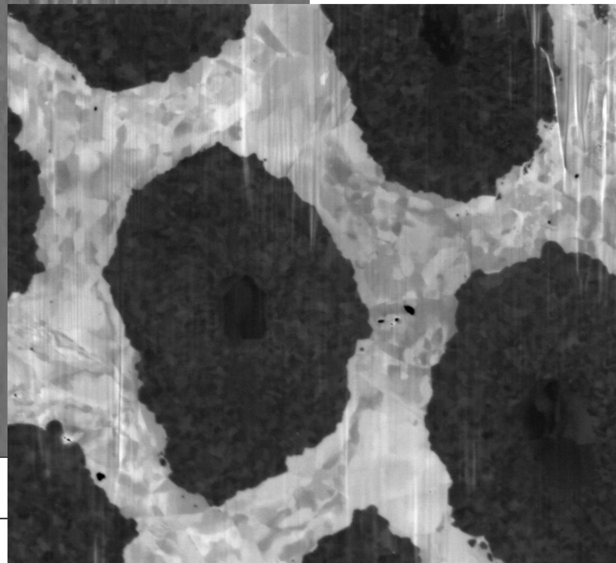
Surface contrast



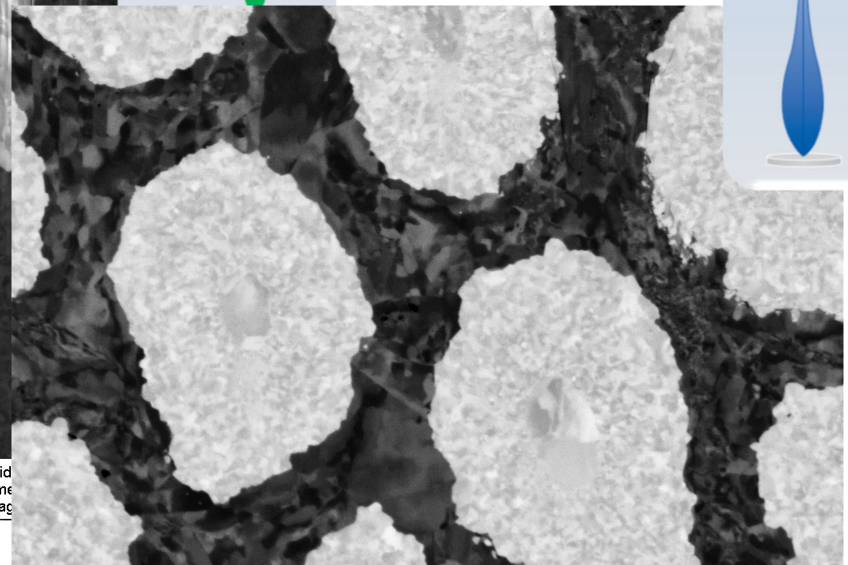




in-chamber SE
ET-detector
topography contrast



in-column
"InLens", SE-detector
"electronic" property contrast

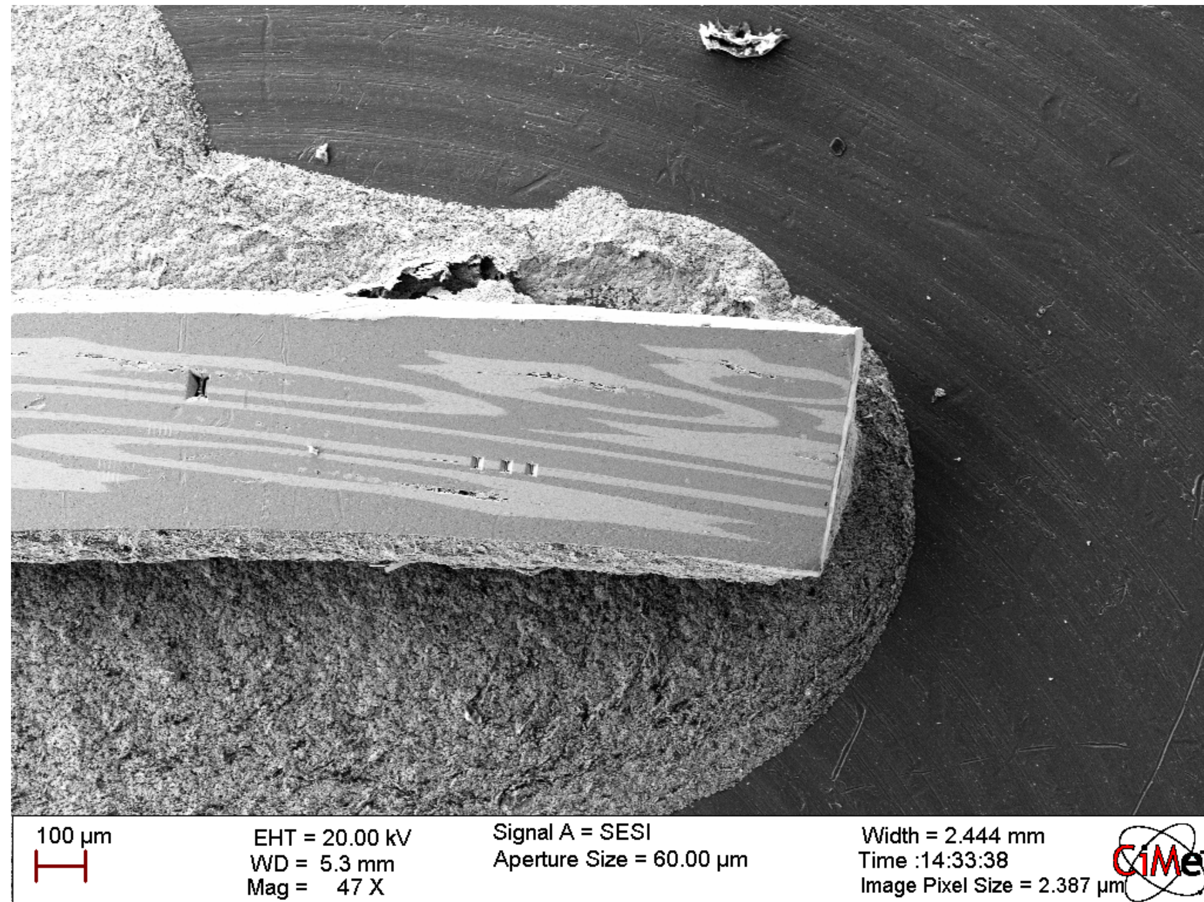


in-column EsB, "energy-selective" BSE-detector
mass-density, "composition"contrast

SEM low kV imaging

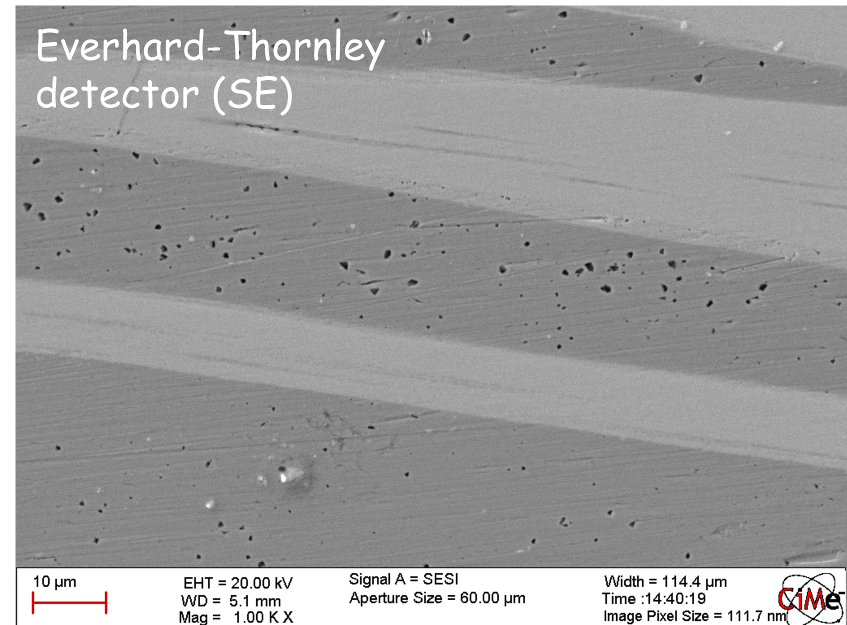
"Easy" sample:

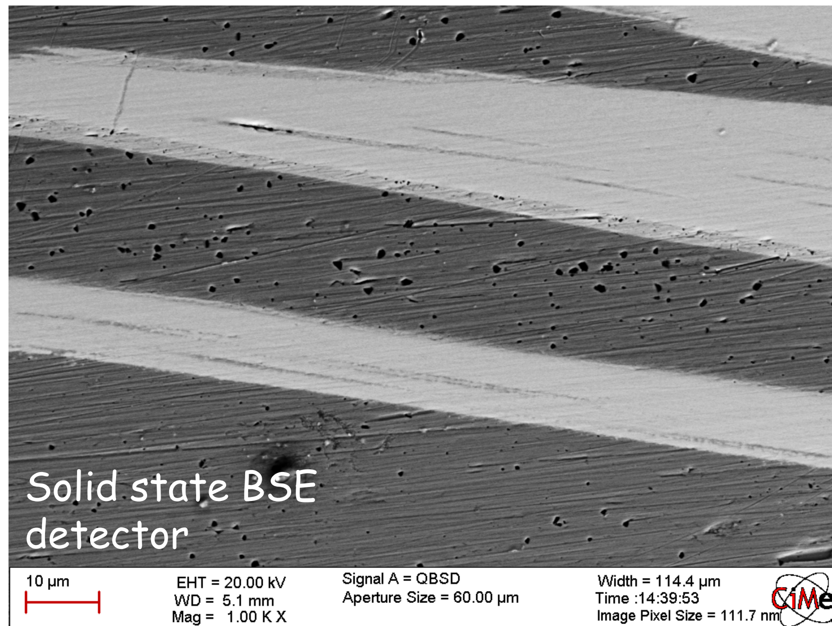
SC wire
Nb_3Sn in
Cu matrix



SE imaging

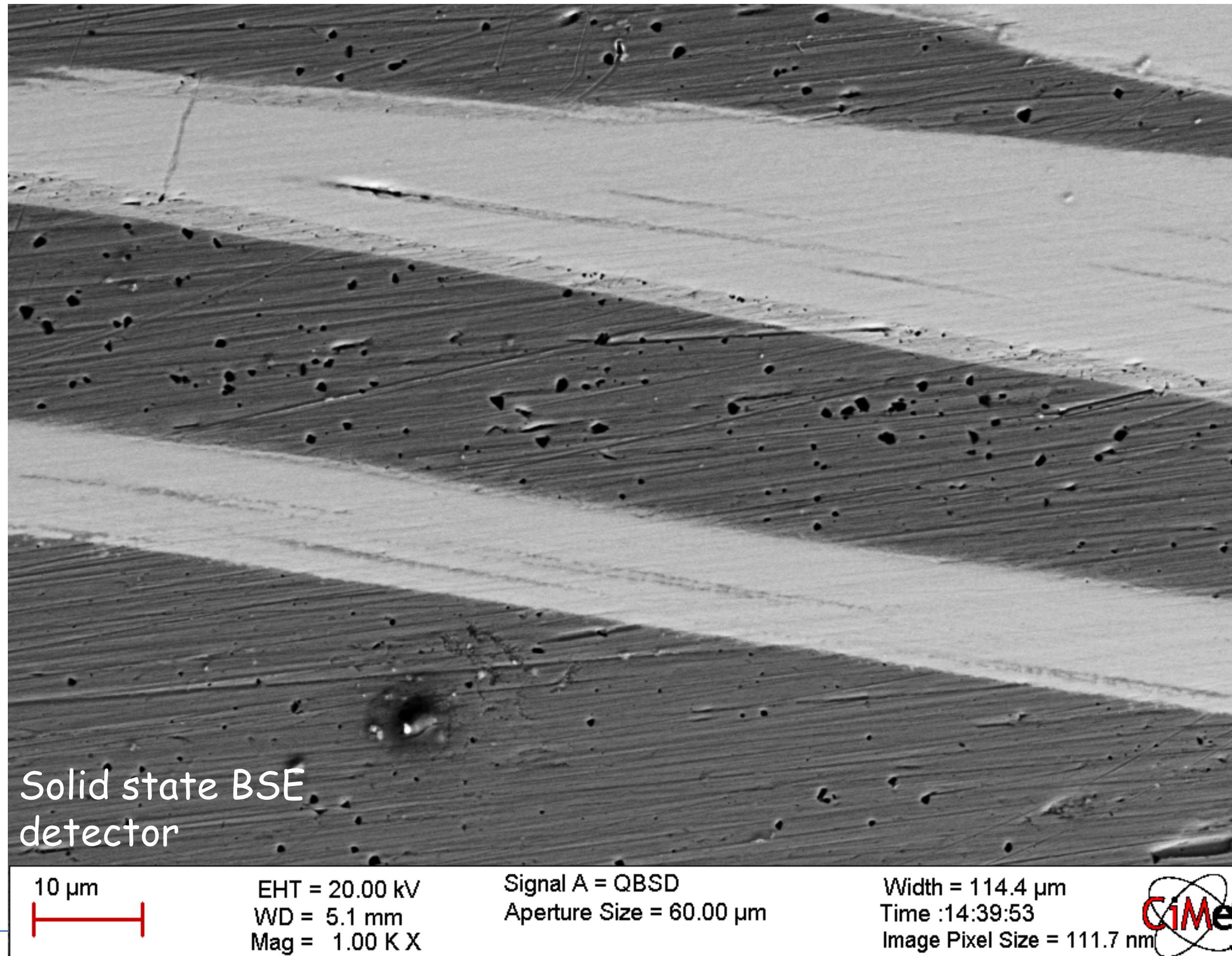
"Easy" samples:



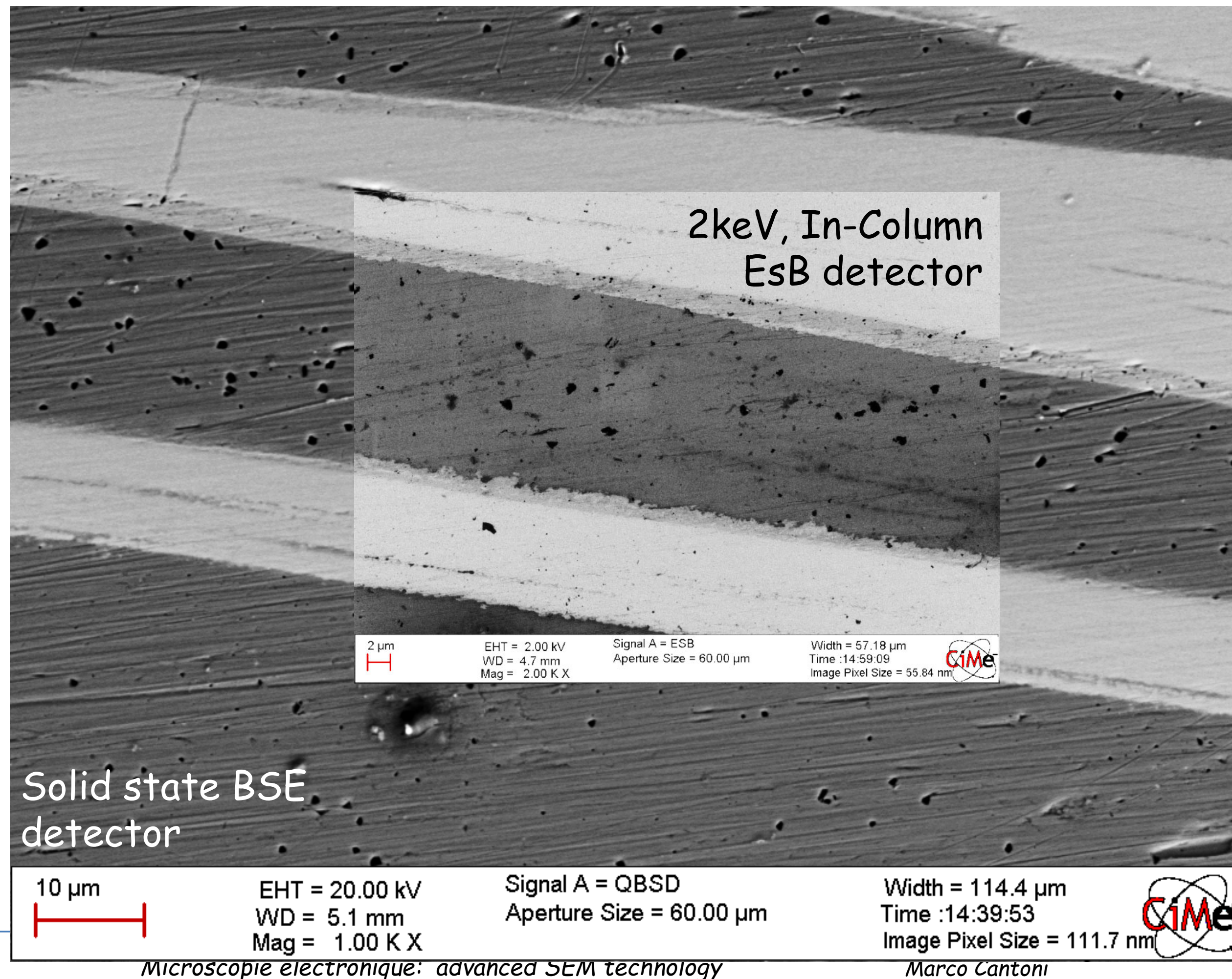


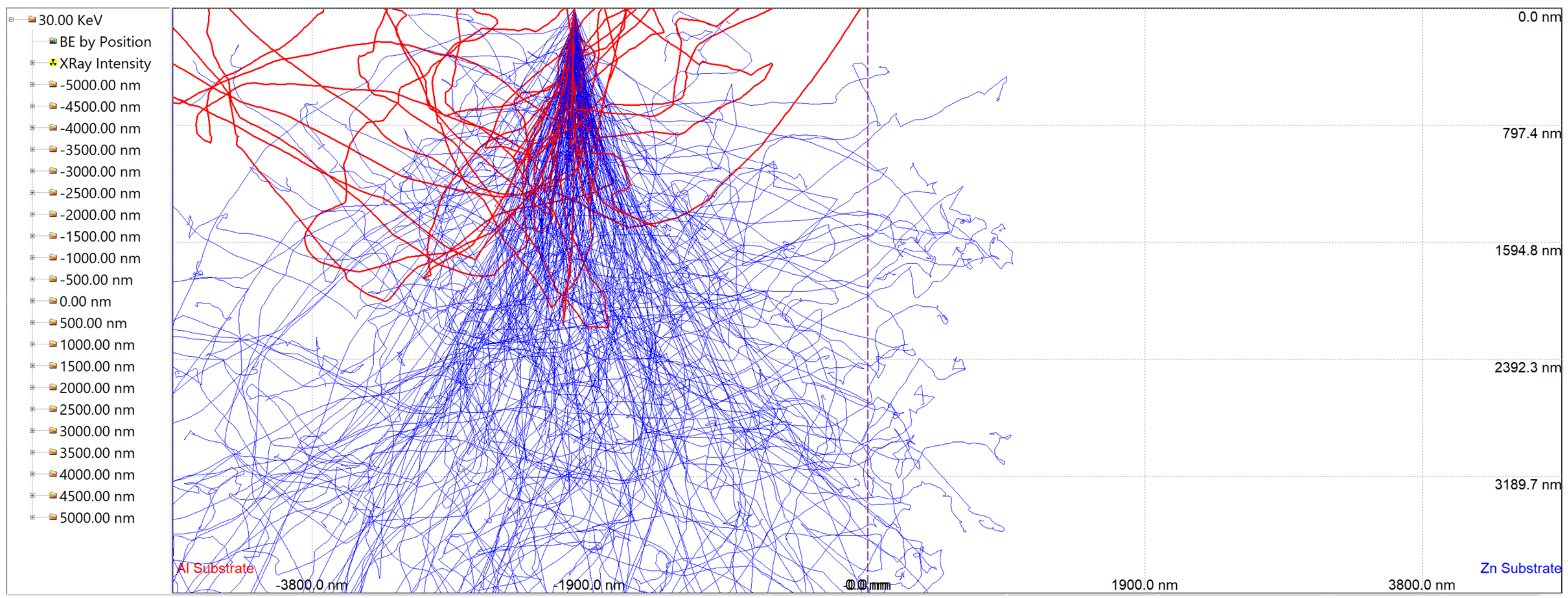
Polished sample: almost no topography !

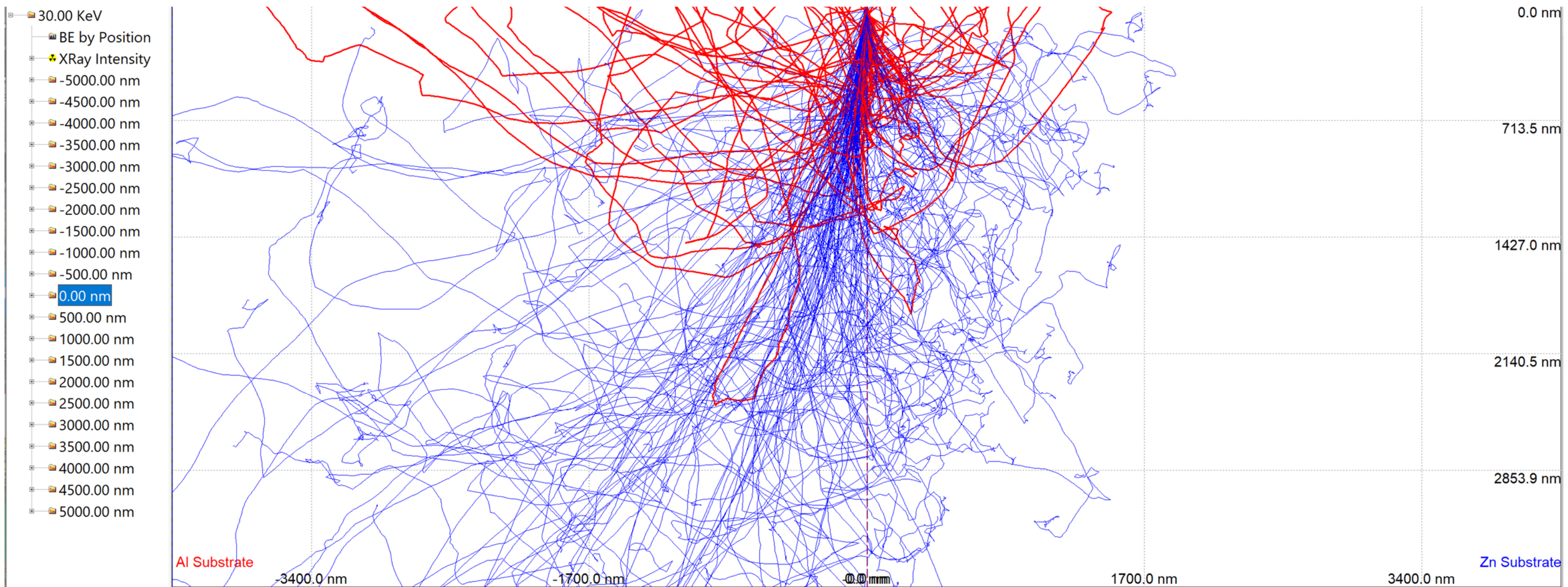
low kV BSE imaging

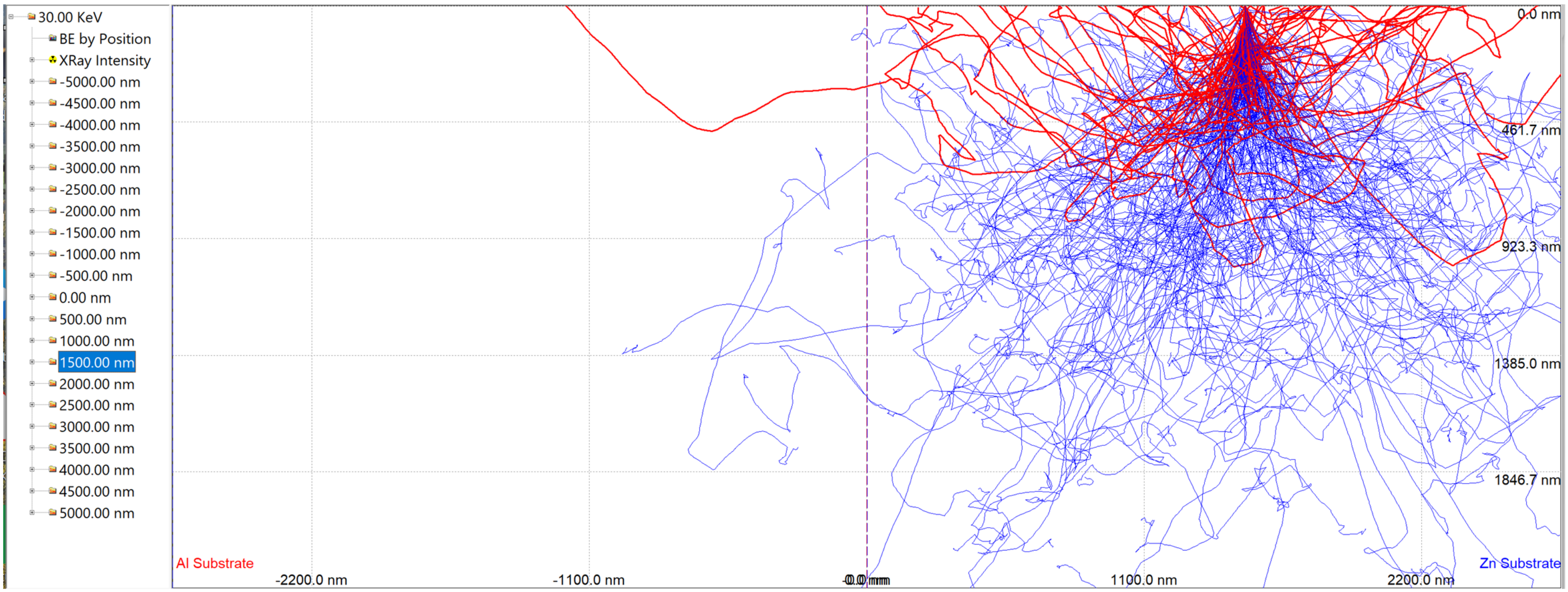


low kV BSE imaging

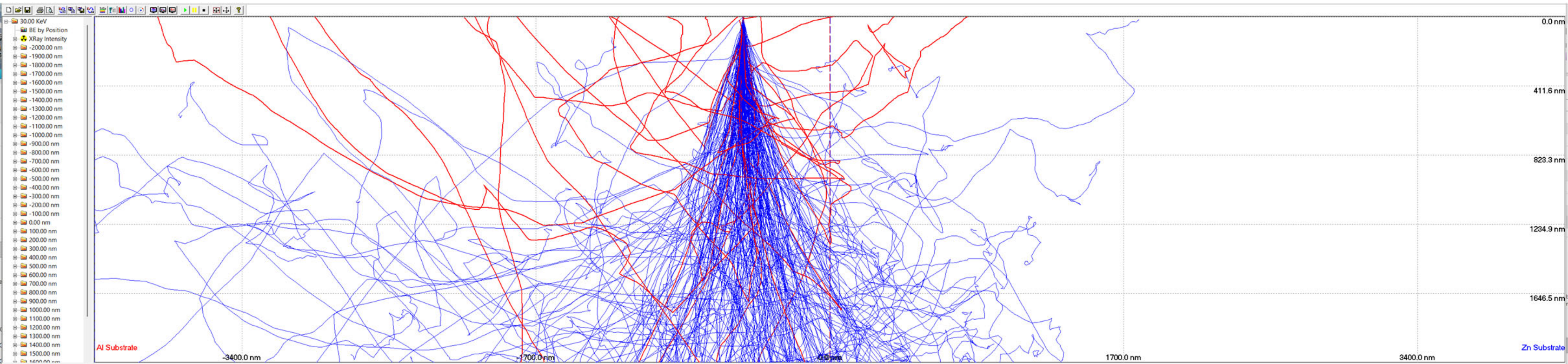



30.00 KeV
BE by Position
XRay Intensity
-5000.00 nm
-4500.00 nm
-4000.00 nm
-3500.00 nm
-3000.00 nm
-2500.00 nm
-2000.00 nm
-1500.00 nm
-1000.00 nm
-500.00 nm
0.00 nm
500.00 nm
1000.00 nm
1500.00 nm
2000.00 nm
2500.00 nm
3000.00 nm
3500.00 nm
4000.00 nm
4500.00 nm
5000.00 nm
0.0 nm
797.4 nm
1594.8 nm
2392.3 nm
3189.7 nm
Al Substrate
Zn Substrate
-3800.0 nm
-1900.0 nm
0.0 nm
1900.0 nm
3800.0 nm

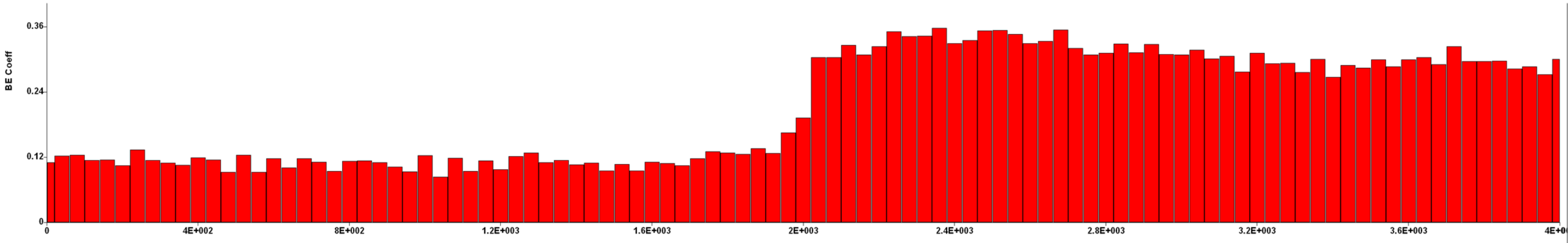

30.00 KeV
BE by Position
XRay Intensity
-5000.00 nm
-4500.00 nm
-4000.00 nm
-3500.00 nm
-3000.00 nm
-2500.00 nm
-2000.00 nm
-1500.00 nm
-1000.00 nm
-500.00 nm
0.00 nm
500.00 nm
1000.00 nm
1500.00 nm
2000.00 nm
2500.00 nm
3000.00 nm
3500.00 nm
4000.00 nm
4500.00 nm
5000.00 nm
0.0 nm
713.5 nm
1427.0 nm
2140.5 nm
2853.9 nm
Al Substrate
Zn Substrate
-3400.0 nm
-1700.0 nm
0.0 nm
1700.0 nm
3400.0 nm


30.00 KeV
BE by Position
XRay Intensity
-5000.00 nm
-4500.00 nm
-4000.00 nm
-3500.00 nm
-3000.00 nm
-2500.00 nm
-2000.00 nm
-1500.00 nm
-1000.00 nm
-500.00 nm
0.00 nm
500.00 nm
1000.00 nm
1500.00 nm
2000.00 nm
2500.00 nm
3000.00 nm
3500.00 nm
4000.00 nm
4500.00 nm
5000.00 nm
0.0 nm
461.7 nm
923.3 nm
1385.0 nm
1846.7 nm
Al Substrate
Zn Substrate
-2200.0 nm
-1100.0 nm
0.0 nm
1100.0 nm
2200.0 nm

Simulation of interaction volumes across interface Al (left) and Zn (right)
30 kV

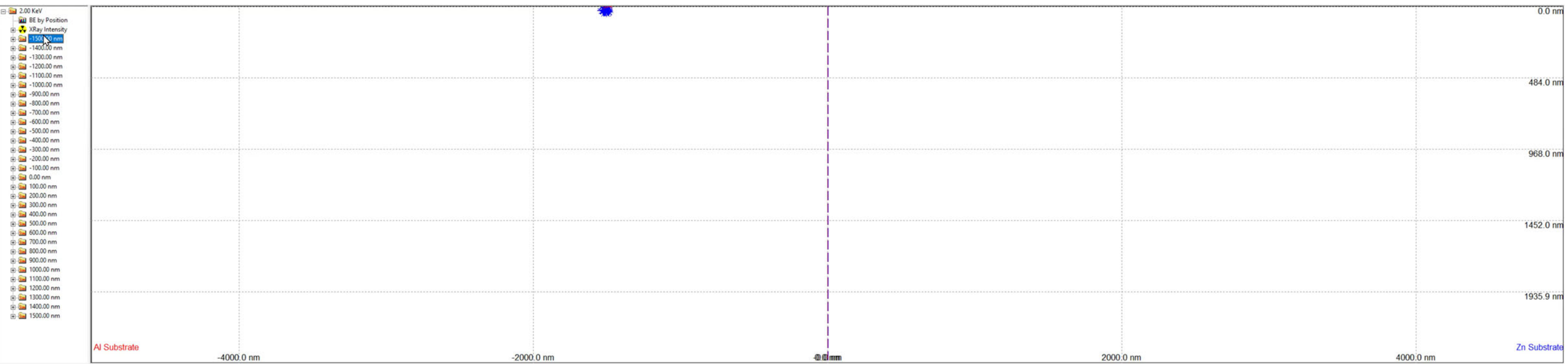


Al Zn

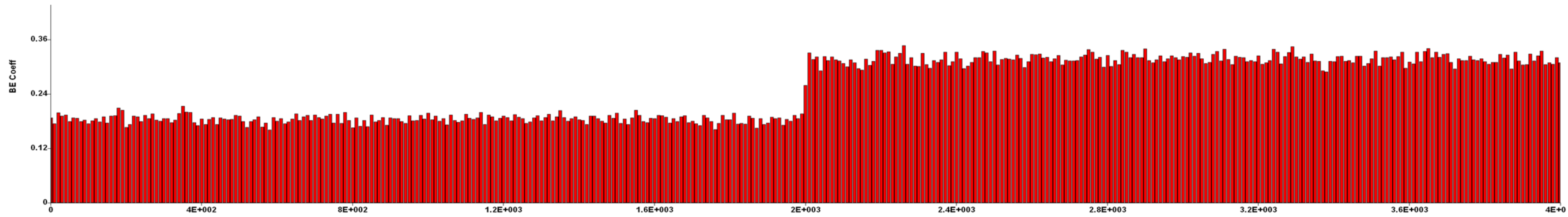


BSE signal

Simulation of interaction volumes across interface Al (left) and Zn (right)
2 kV



Al Zn

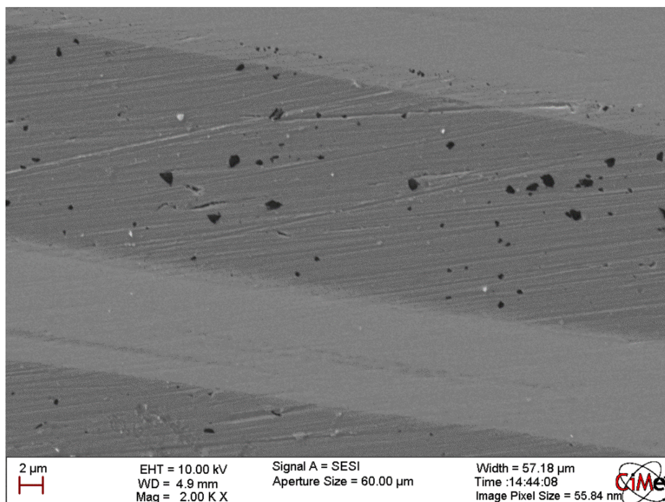


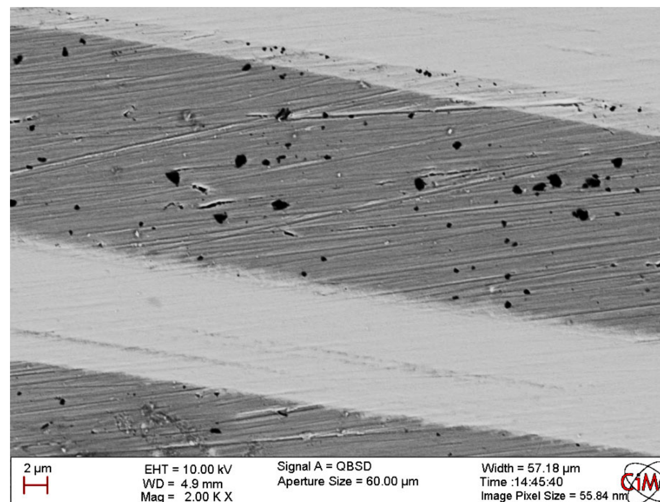
BSE signal

SEM low kV imaging

"Easy" samples:

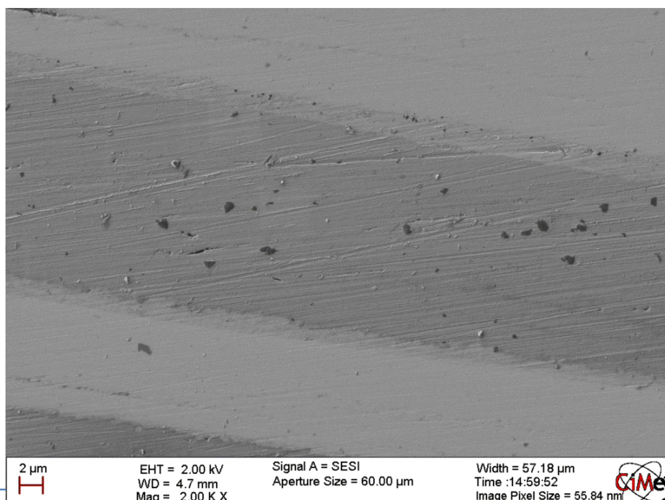
10kV
SE
(ETD)

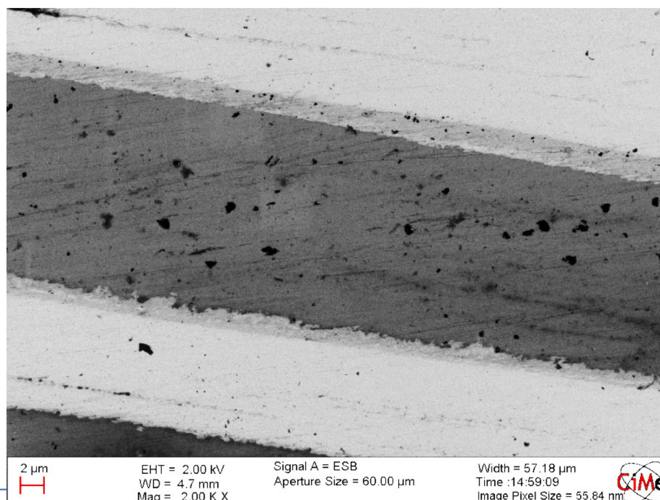




10kV
BSE

2kV
SE
(ETD)

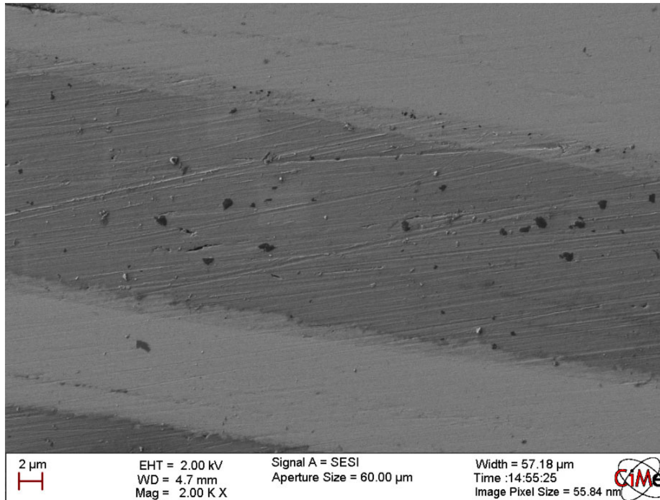


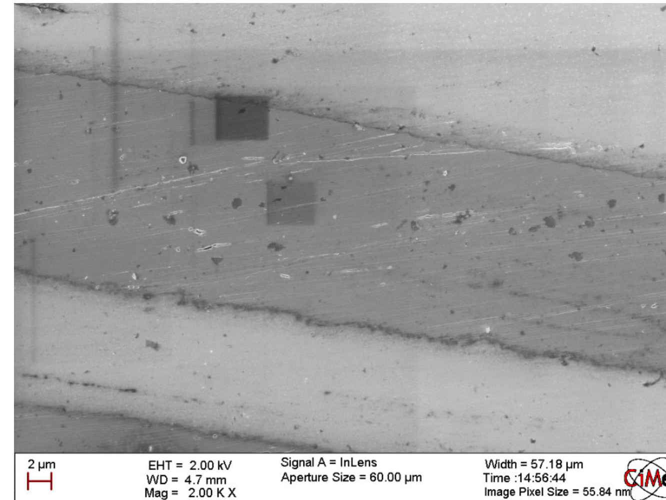


2kV
BSE
(EsB)

low kV imaging: surface contamination !

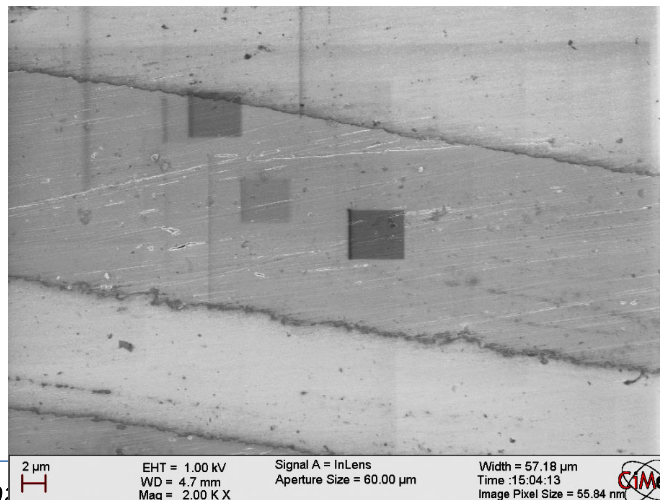
2kV
SE
(ETD)

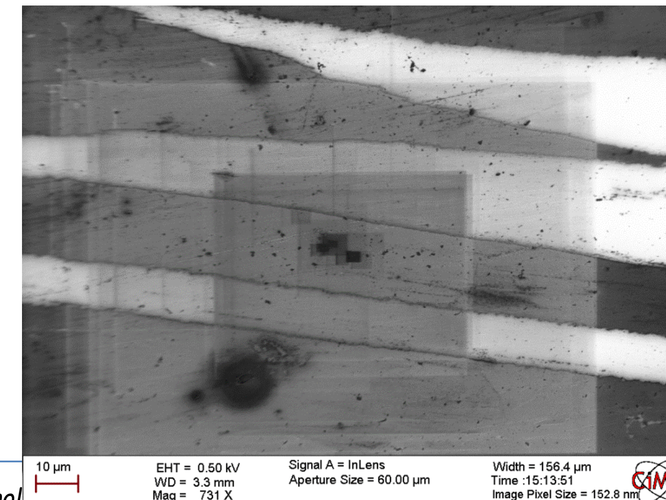




2kV
SE
(in-lens)

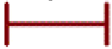
1kV
SE
(in-lens)





0.5kV
SE
(in-lens)

10 µm



EHT = 0.50 kV
WD = 3.3 mm
Mag = 731 X

Signal A = InLens
Aperture Size = 60.00 µm

Width = 156.4 µm
Time :15:13:51
Image Pixel Size = 152.8 nm

SEM low kV imaging

Contamination by hydro-carbons

- contamination “spoils” imaging at low kV
- How to avoid (at CIME):
  - plasma cleaning of the sample before inserting (oxygen plasma destroys hydro-carbons)
  - Plasma clean the chamber at each insertion (multi-user environment)

first HR-SEM at CIME

- Starting point: XL-30 SFEG SIRION (since 2001)
- First semi-in-lens HR-SEM:
  in-lens (through the lens) detection of SE and BSE

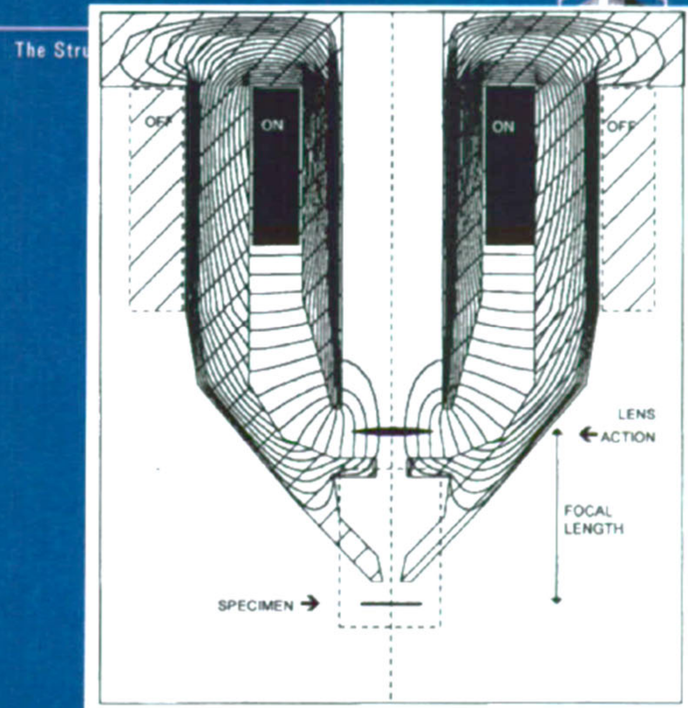
Resolution in UHR mode: 1.5 nm at 10 kV (or higher); 2.5 nm at 1 kV

Conventional Mode

magnetic field inside pole-piece

Mode I

- Conventional SE
- TLD
- (Solid State BSE)
- STEM

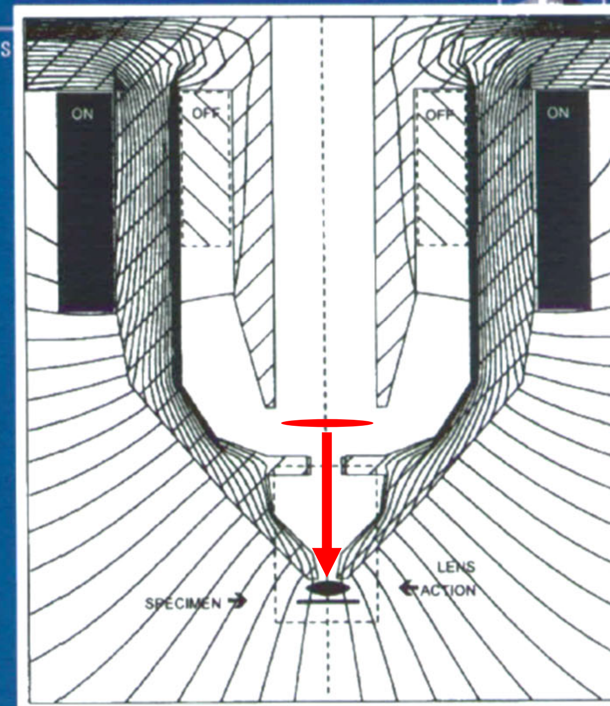


Immersion lens

specimen inside "lens"

Mode II

- TLD (SE & BSE)
- Solid State BSE
- STEM



FEI

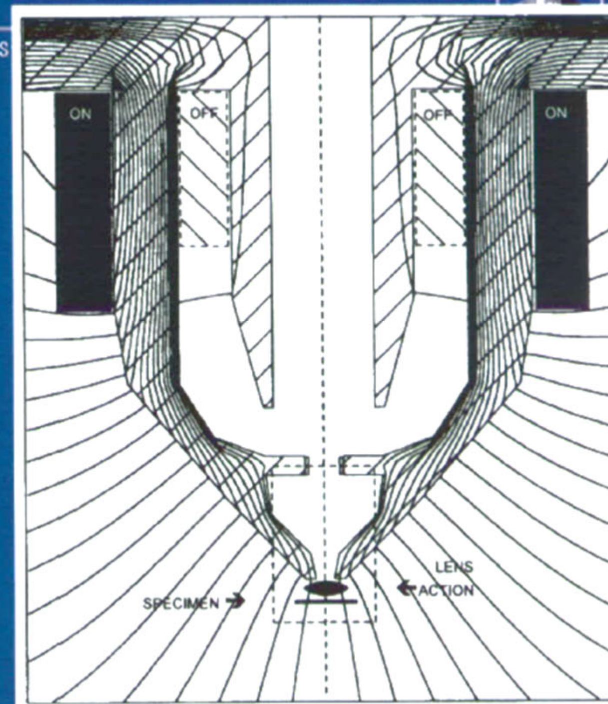
CIMe

Immersion lens

specimen inside "lens"

Mode II

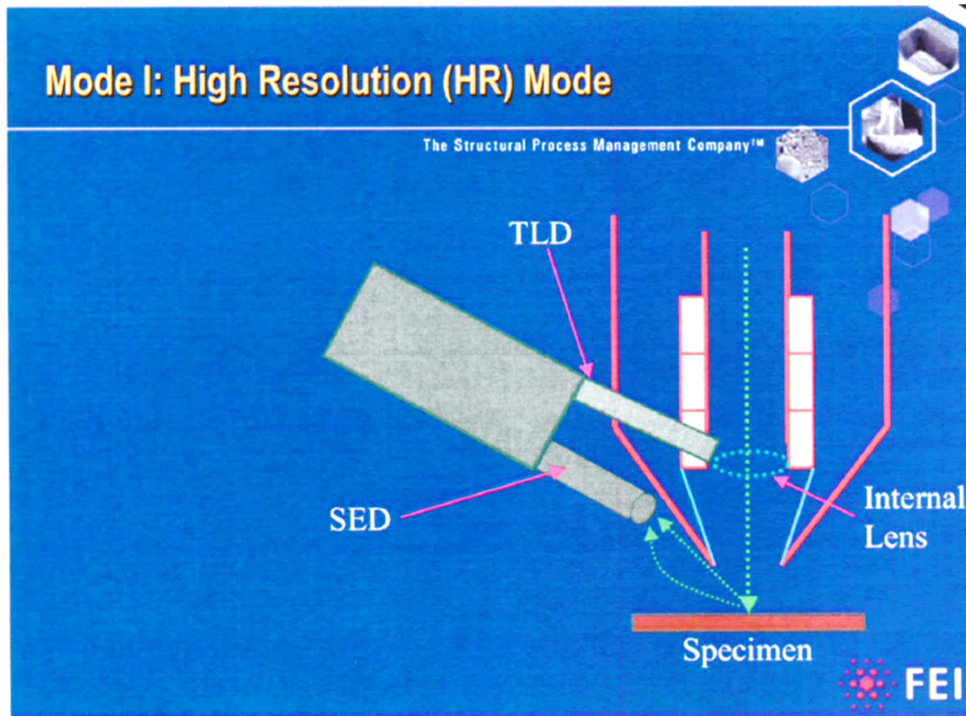
- TLD (SE & BSE)
- Solid State BSE
- STEM



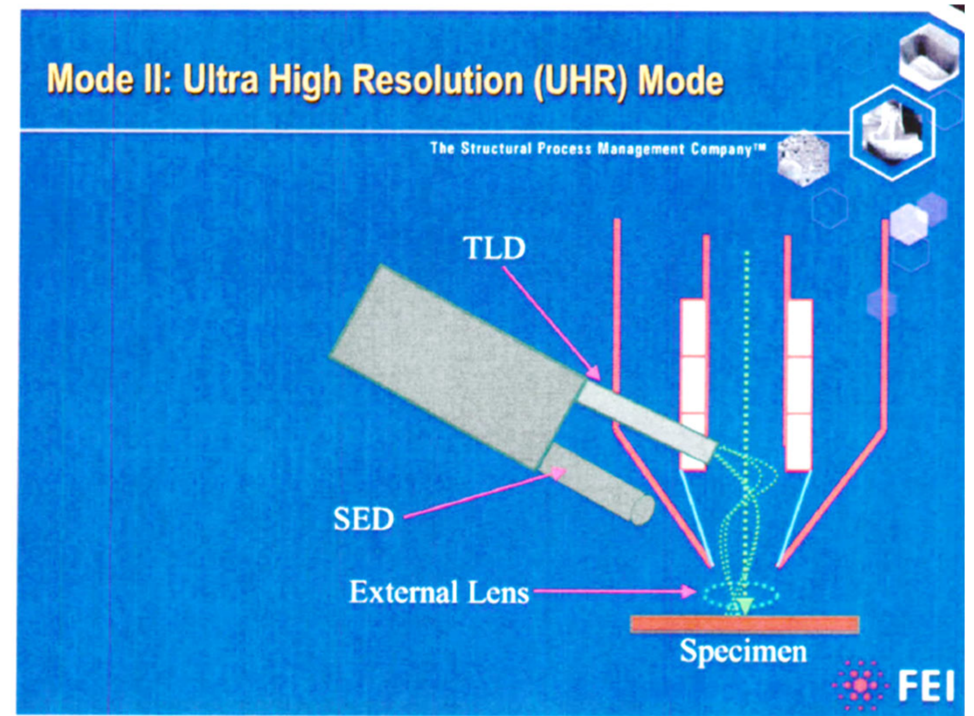
FEI

CiMe

detection of SE



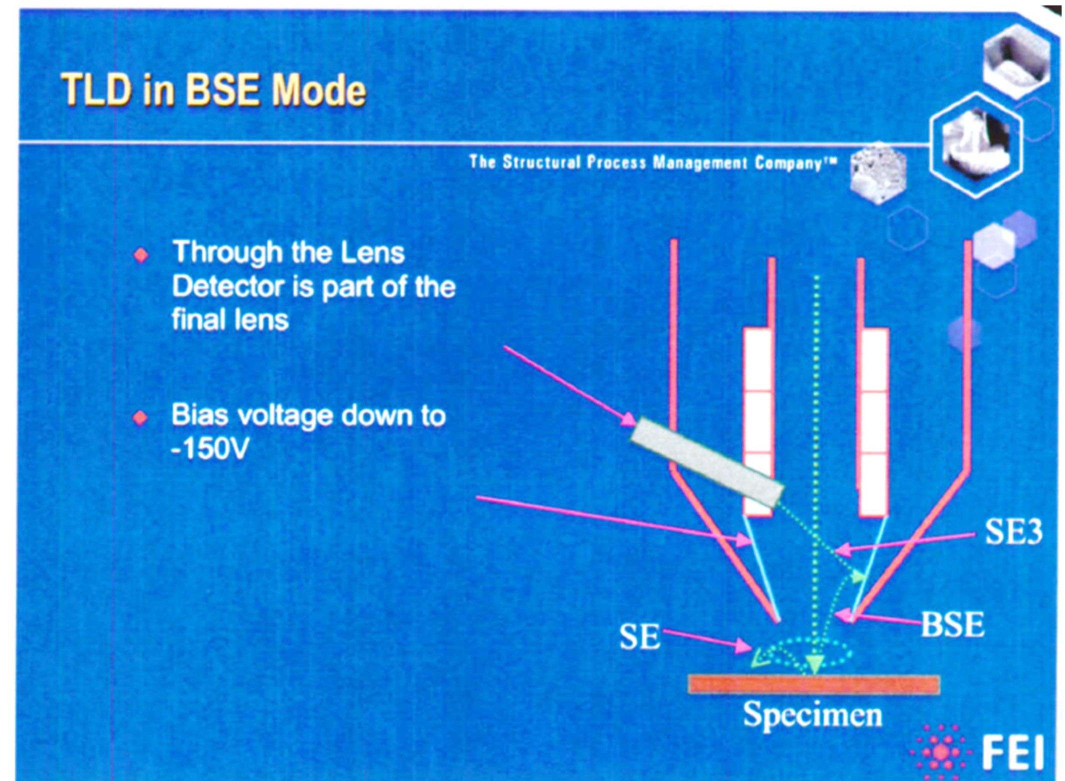
Non-immersion mode



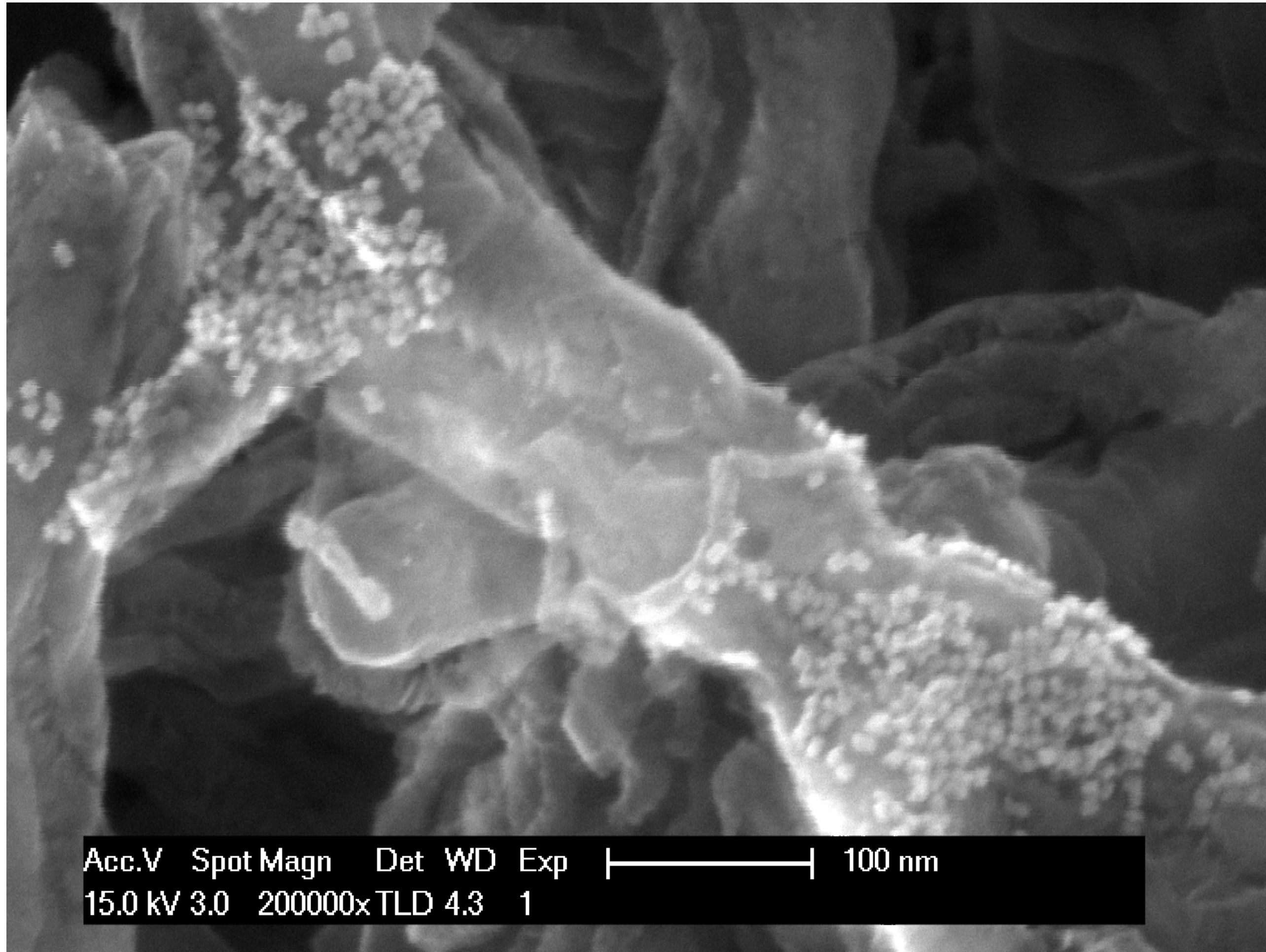
Immersion mode

detection of BSE

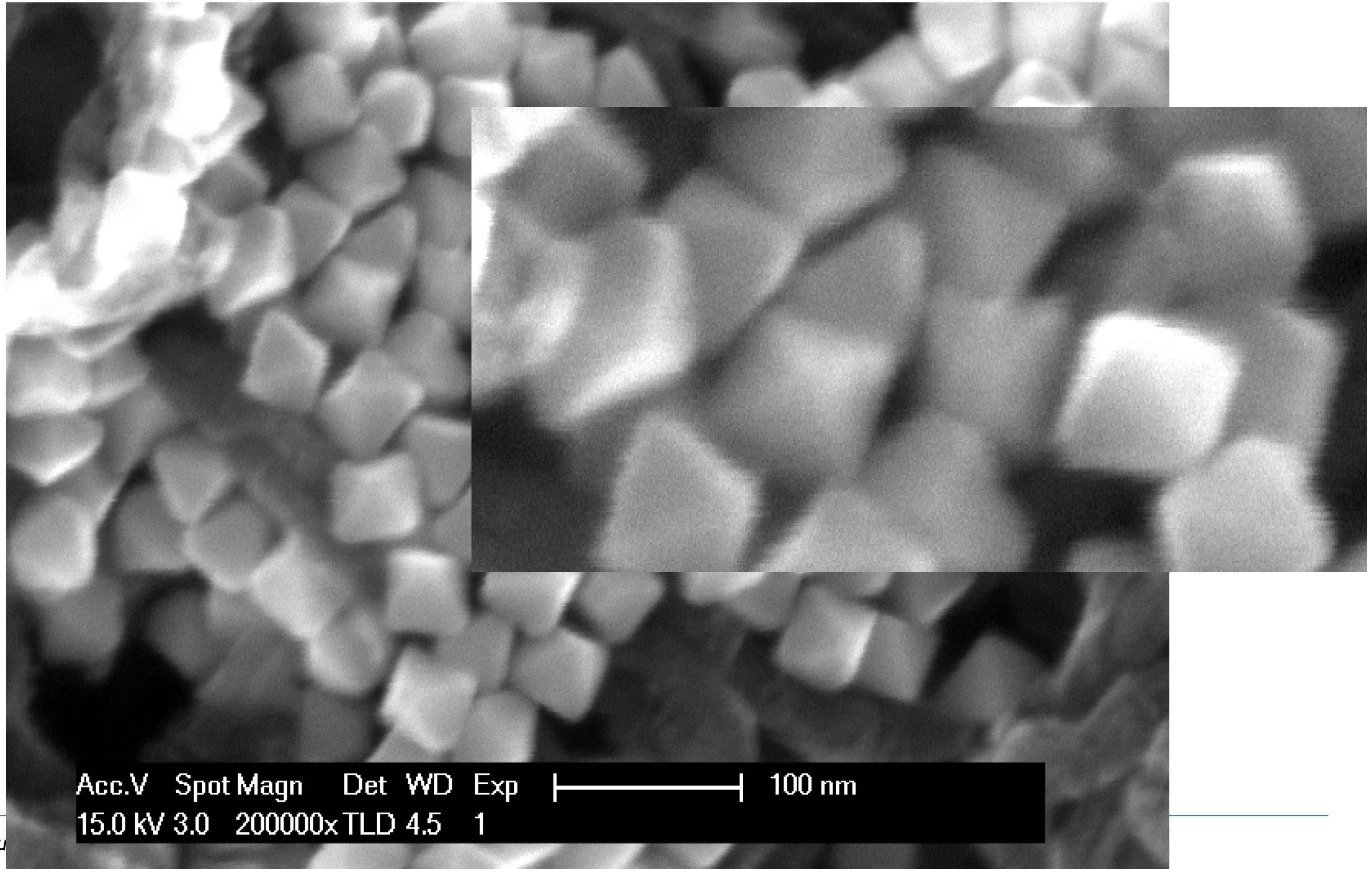
- Bias: repulsion of SE-1 and SE-2 from the sample
- BSE enter the column and hit the inside of the column ->> SE-3
- Choice: either SE or BSE mode !!!



Pd catalyst particles on carbon fibres



"flagging", electronic noise at high magnification



CiMe

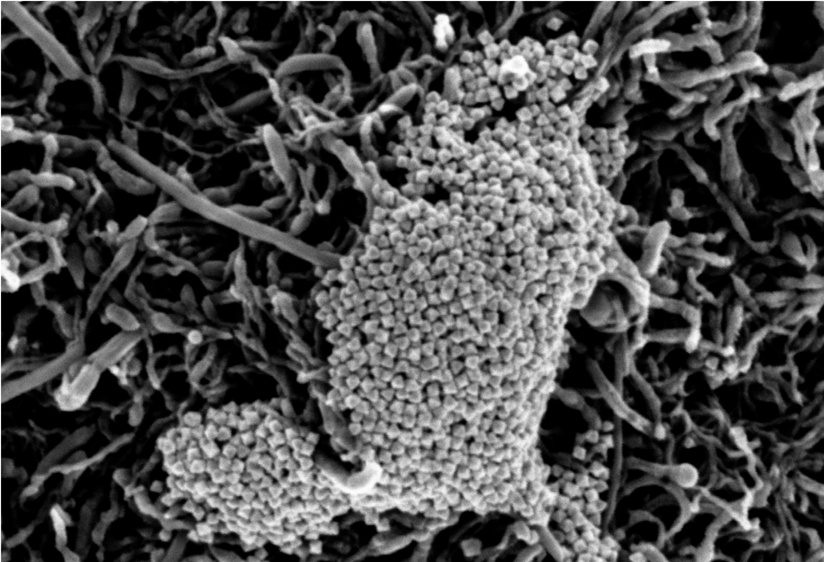
Best "SEM" at EPFL: FIB NVision@CIME, beam booster technology

20 nm

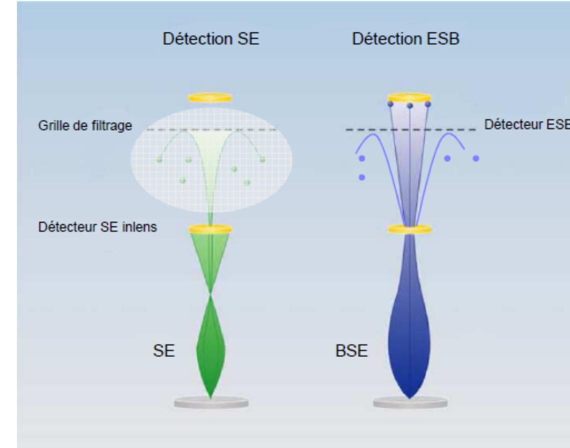
EHT = 1.50 kV
WD = 2.0 mm
Mag = 226.38 K X

Signal A = InLens
Aperture Size = 30.00 µm

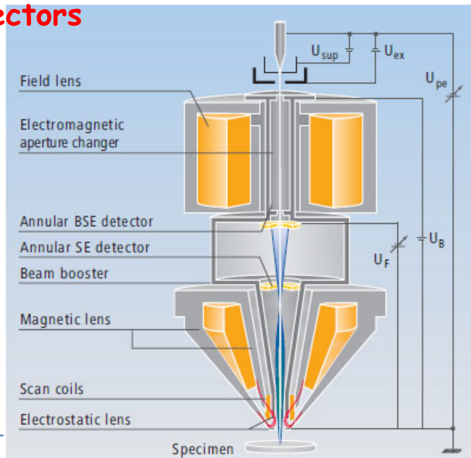
Width = 505.0 nm
Time :10:21:20
Image Pixel Size = 493.2 pm

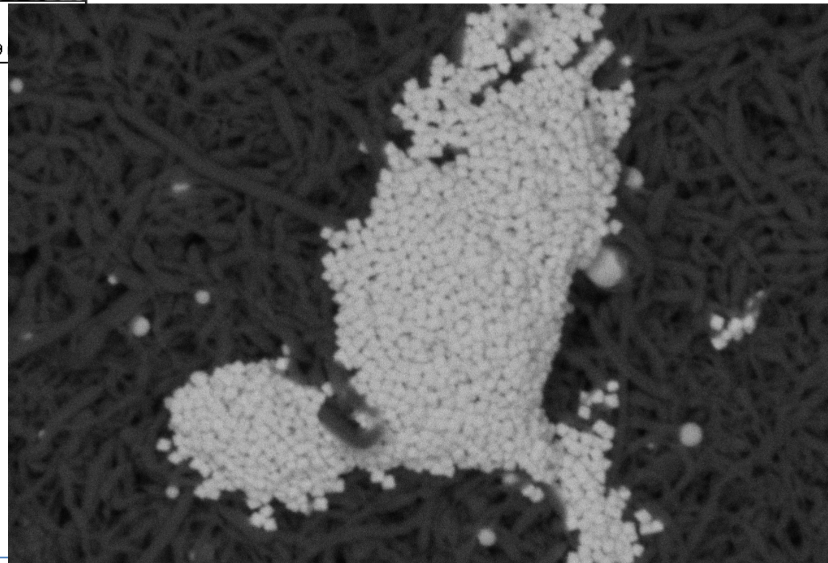


100 nm | EHT = 2.00 kV | WD = 4.8 mm | Mag = 41.83 K X | Signal A = InLens | Aperture Size = 30.00 µm | Width = 2.733 µm | Time :9:58:54 | Image Pixel Size = 2.669



Two different contrasts with one scan: parallel detectors





100 nm | EHT = 2.00 kV | WD = 4.8 mm | Mag = 41.83 K X | Signal A = ESB | Aperture Size = 30.00 µm | Width = 2.733 µm | Time :10:02:11 | Image Pixel Size = 2.669 nm