

High resolution Scanning Electron Microscopy



Damien VALENTE

Séminaire CERTeM - 29/09/2016





- 1. Overview**
- 2. HR imaging**
- 3. Analytical capabilities**
- 4. CERTeM 2020 AO4 investment**

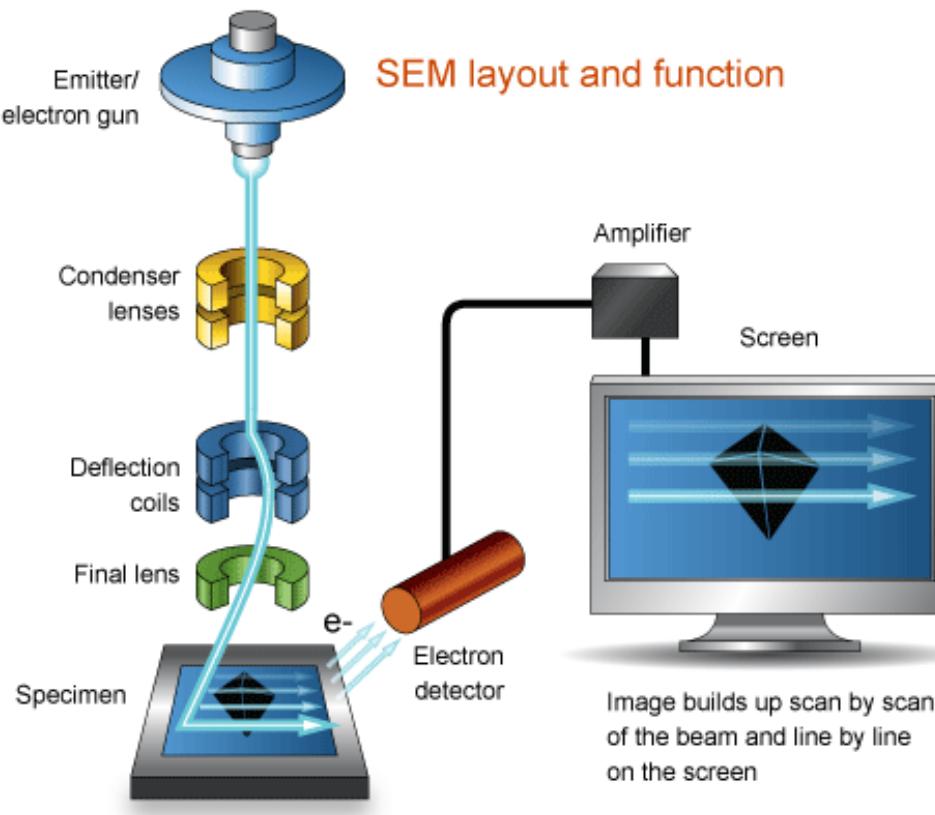


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What is needed for High Resolution SEM?

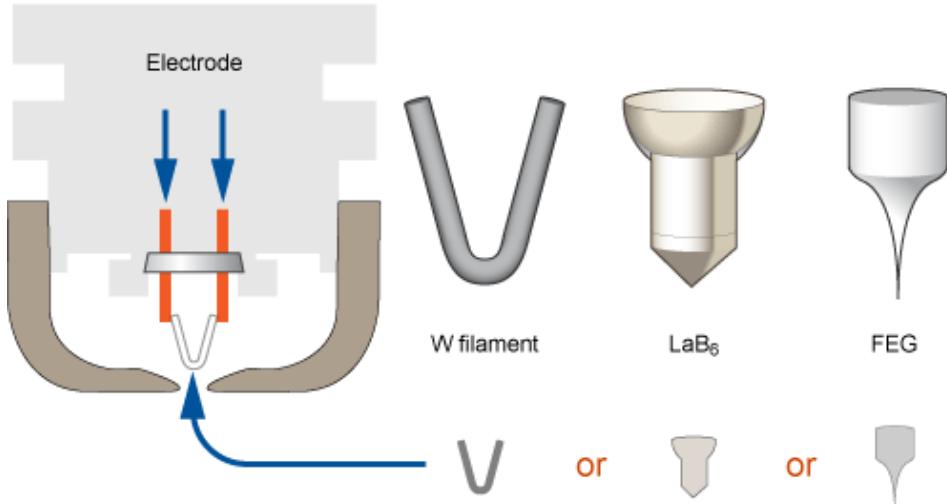
- A small probe size
- High beam current
- A mechanically stable microscope and a quiet lab environment
- A skilled operator





Electron gun

3 commonly used types of electrons sources:



Which FEG source for HR ?

➤ Cold FE

- Highest resolution (energy spread)
- Low running cost
- Max probe current : ≤ 20 nA

➤ Schottky FE

- better beam current stability
- less vacuum requirements
- no need for periodic emitter flashing
- Higher probe currents : ≥ 200 nA

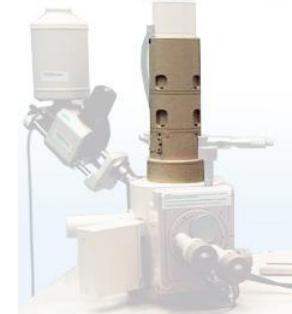
Emission	Thermionic		Field Emission
	W	LaB6	FE
Size (nm)	1×10^5	2×10^4	0.2
Brightness (A/cm ² .steradian)	$10^4 - 10^5$	$10^5 - 10^6$	$10^7 - 10^9$
Energy Spread (eV)	1 - 5	0.5 – 3.0	0.2 – 0.3
Operating Lifetime (hrs)	>20	>100	>300
Vacuum (torr)	$10^{-4} - 10^{-5}$	$10^{-6} - 10^{-7}$	$10^{-9} - 10^{-10}$



Electromagnetic lens system

➤ Condenser lens

controls the intensity of the electron beam reaching the specimen



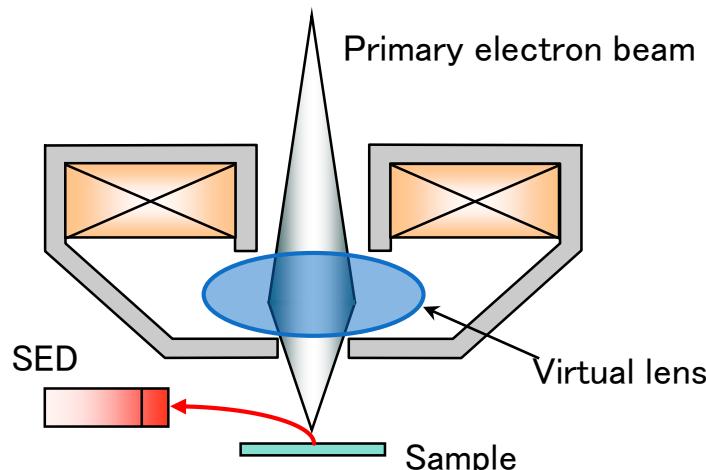
➤ Deflection coils

Deflect the beam in the x and y axes

➤ Final/objective lens

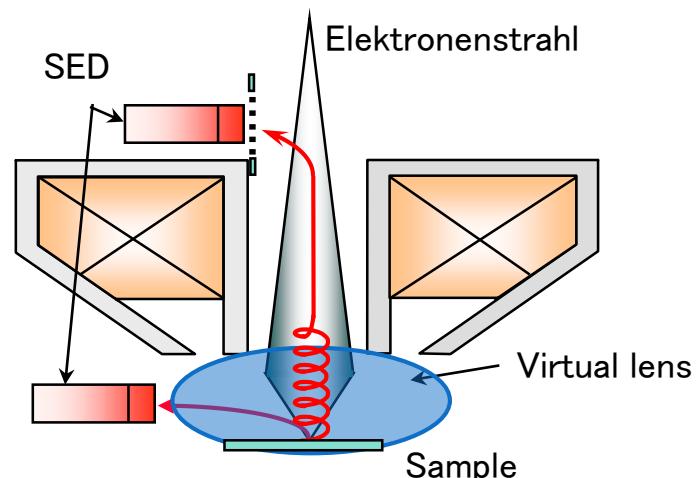
Brings the electron beam into focus (de-magnifies) on the specimen

Conventional Lens



Semi in-lens

Magnetic Immersion Lens, "Snorkel lens"





Electron-matter interactions

When high energy electrons impinge on the specimen, a number of signals are generated :

➤ Secondary electrons

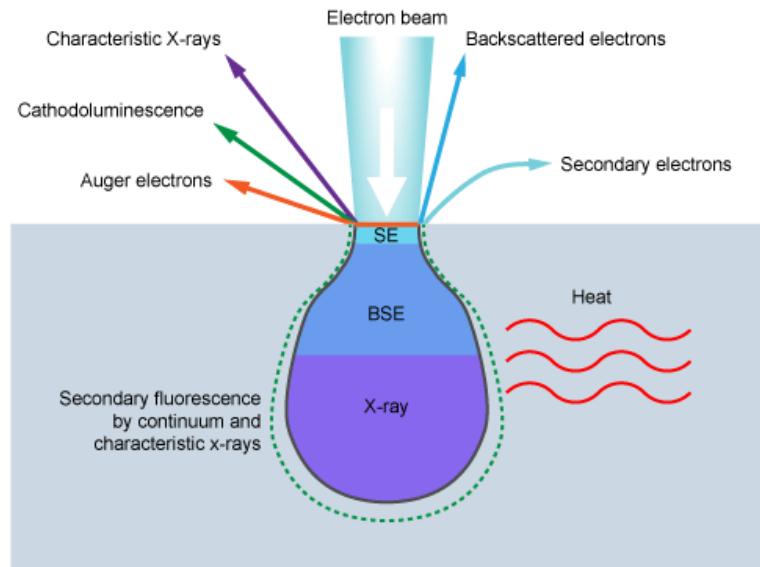
- low energy electrons (~2 to 50 eV)
- Topographical info
- High spatial resolution

➤ Backscattered electrons

- High energy (> 50eV)
- Atomic number and compositional info
- Less charge up effect and edge contrast

➤ X-rays

Elemental composition information

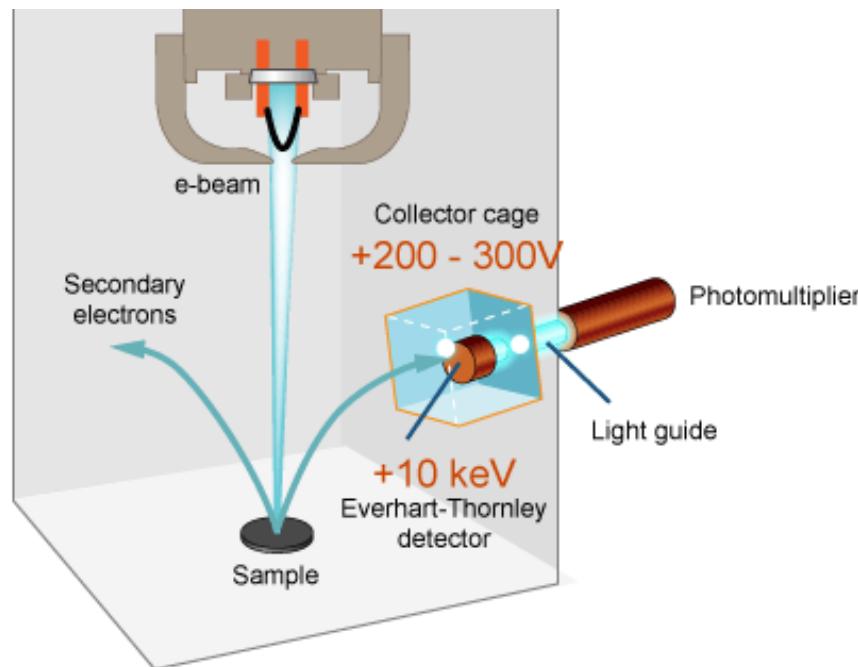




Detectors

Secondary Electron Detector (SED) – Everhart-Thornley Detector

- A scintillator emits photons when hit by high-energy electrons.
- The emitted photons are collected by a light guide and transported to a photomultiplier for detection.
- A metal grid known as a Faraday cage surrounds the scintillator, and is usually held at a positive potential to attract the secondary electrons.

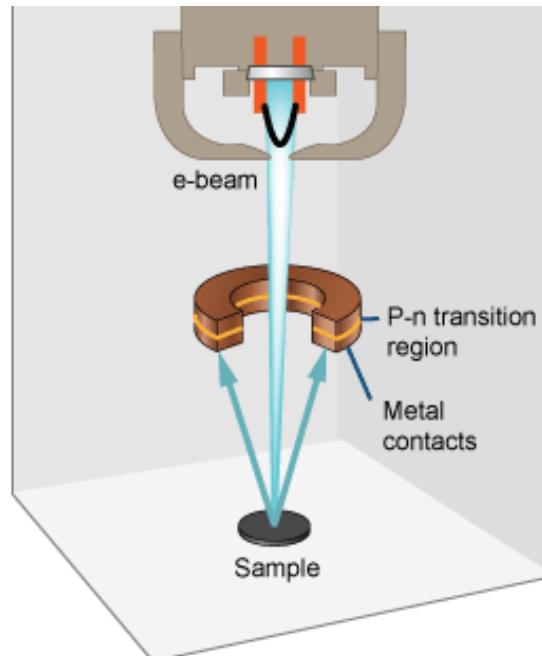




Detectors

BackScattered Electron Detector (BSED) – solid state diode detector

- The BSED is mounted below the obj. lens pole piece and centered around the optic axis.
- Both compositional or topographical backscattered electron images can be recorded depending on the window of electron energies selected for image formation.

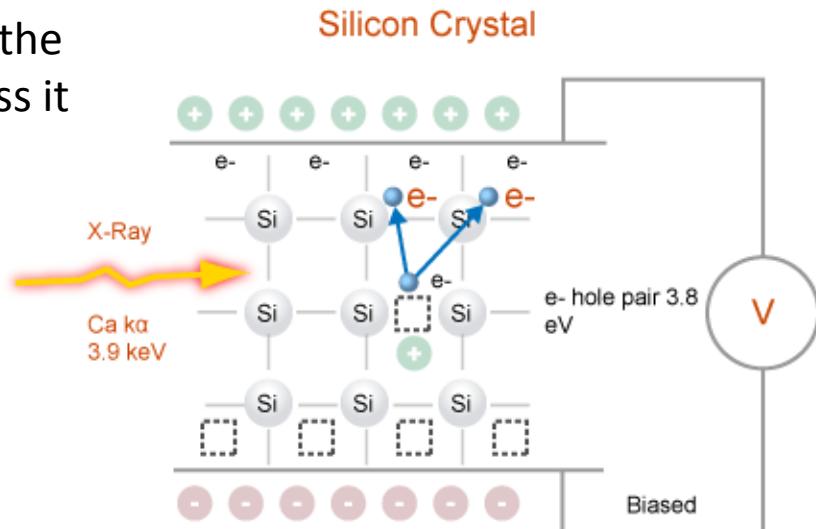




Detectors

X-ray detection by EDS* – silicon drift detector

- A collimator ensure that only X-rays generated from where the primary electron beam interacts with the sample will be collected.
- An electron trap ensure no electrons enter the detector, just X-rays
- A semiconductor crystal detector
- Electronics to detect the charge recorded by the detector, convert it to a voltage pulse and pass it to the pulse processor



$\sim 1000 \text{ e-} / \square$

Total charge = 10^{-16} Coulombs

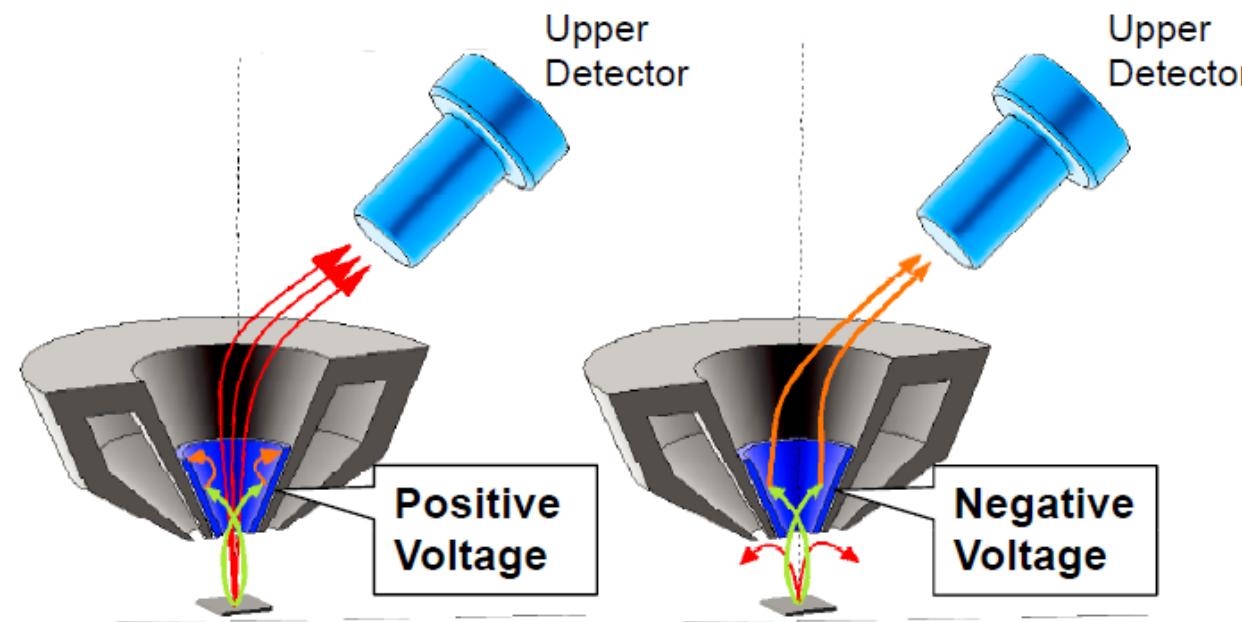
- Si chips will have inherent formation of e-hole pair
- Time is required to take a measurement



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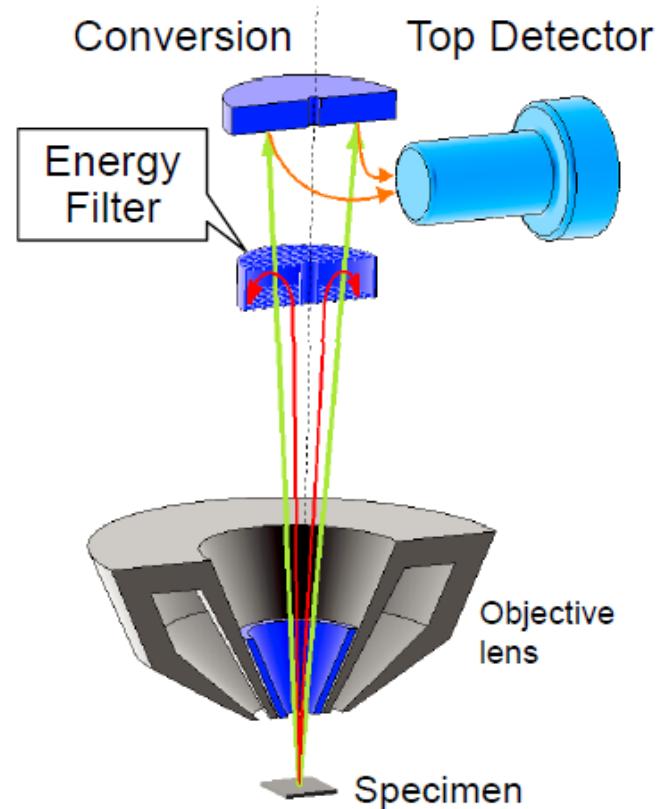
Hitachi - in column twin detector system



SE mode

Low Angle BSE mode

Filtering technology in column
With Hitachi patented ExB Filter



High Angle BSE mode

With New Energy Filter
(High Energy Pass)



What you see depends on the detector !

The diagram illustrates the internal components of an HR-SEM. It shows a vertical stack of components: an 'Electrode' at the top, followed by a 'Top' detector, a dashed horizontal line, a yellow 'EXB' (Electrostatic Beam Deflector) region, an 'Upper' detector, another dashed horizontal line, a brown 'Lower' detector, and finally a 'Sample' at the bottom. Arrows indicate the paths of particles: a red arrow labeled 'SE' points upwards from the sample, and a blue arrow labeled 'BSE' also points upwards. To the right, a grayscale topographical image shows a cluster of spherical particles. A blue box labeled 'LOWER(SE)' is positioned above the image. Below the image, the text '3.0kV x50.0k SE(L)' is displayed, along with a scale bar indicating '1.00um'. At the bottom, a blue oval encloses the text 'Topographical image with shadow'. Technical details listed include: Sample: Photocatalyst; Vacc: 3.0V; Mag.: x 50k; courtesy of Nagaoka University of Technology, Faculty of Engineering, Dr. Kazunori Sato; Model: SU8020.

Electrode

Top

EXB

Upper

Lower

Sample

SE

BSE

LOWER(SE)

3.0kV x50.0k SE(L)

1.00um

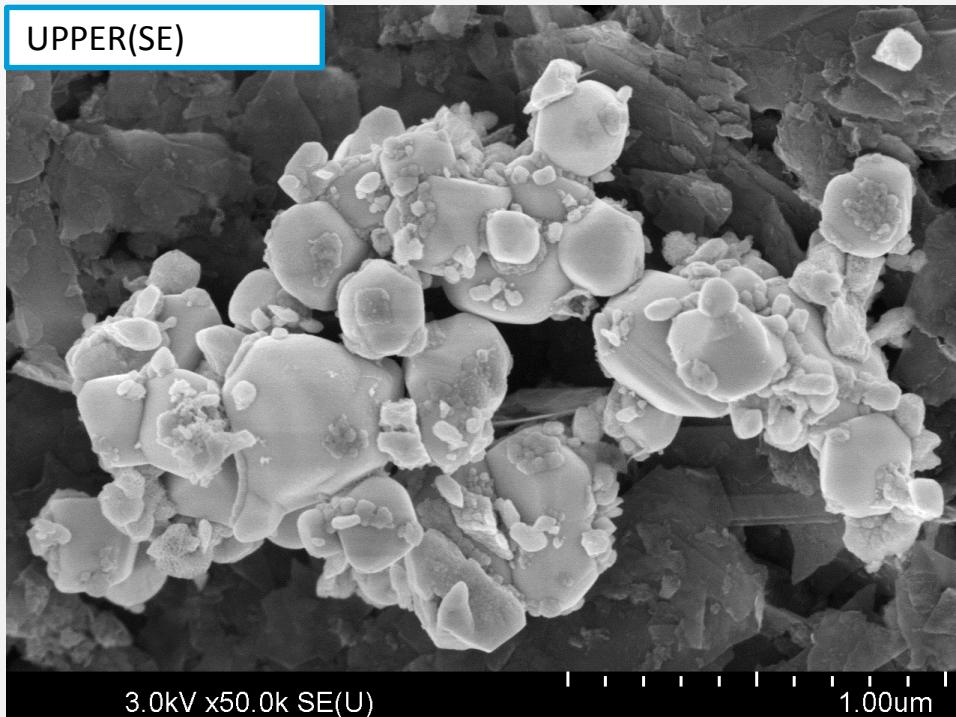
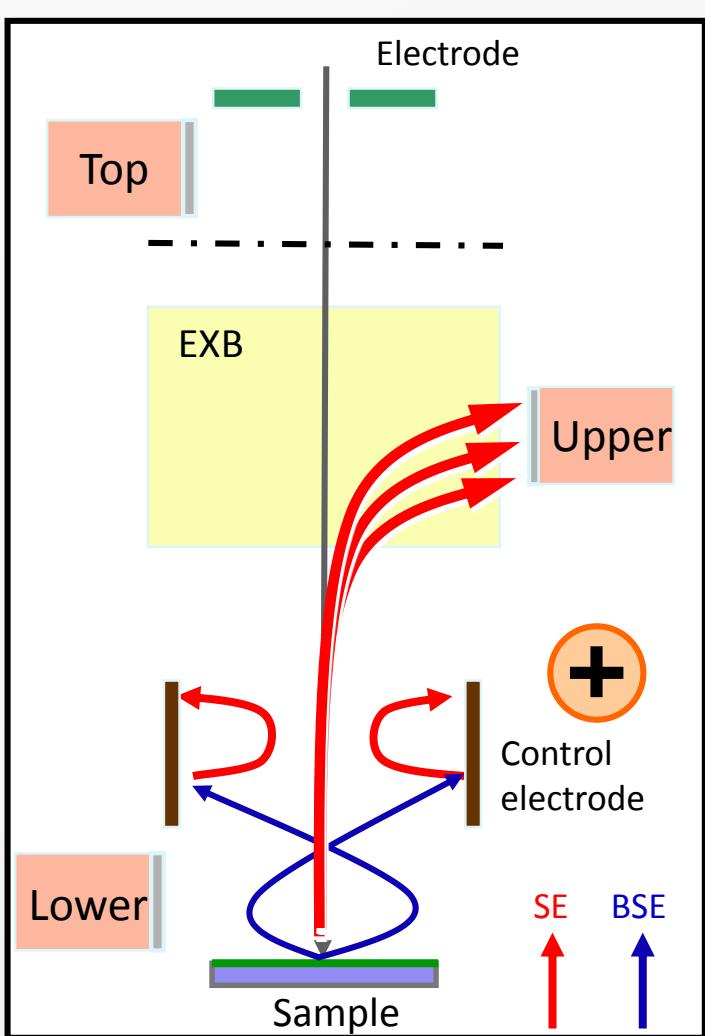
Sample : Photocatalyst
Vacc : 3.0V
Mag. : x 50k

courtesy of
Nagaoka University of Technology,
Faculty of Engineering,
Dr. Kazunori Sato
Model : SU8020

Topographical image with shadow

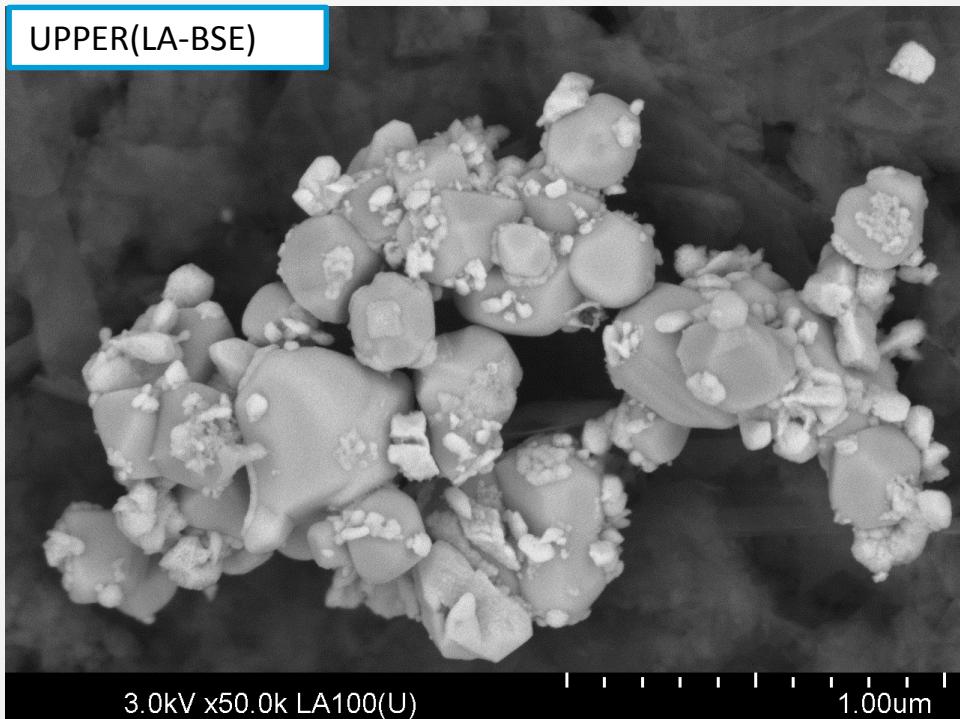
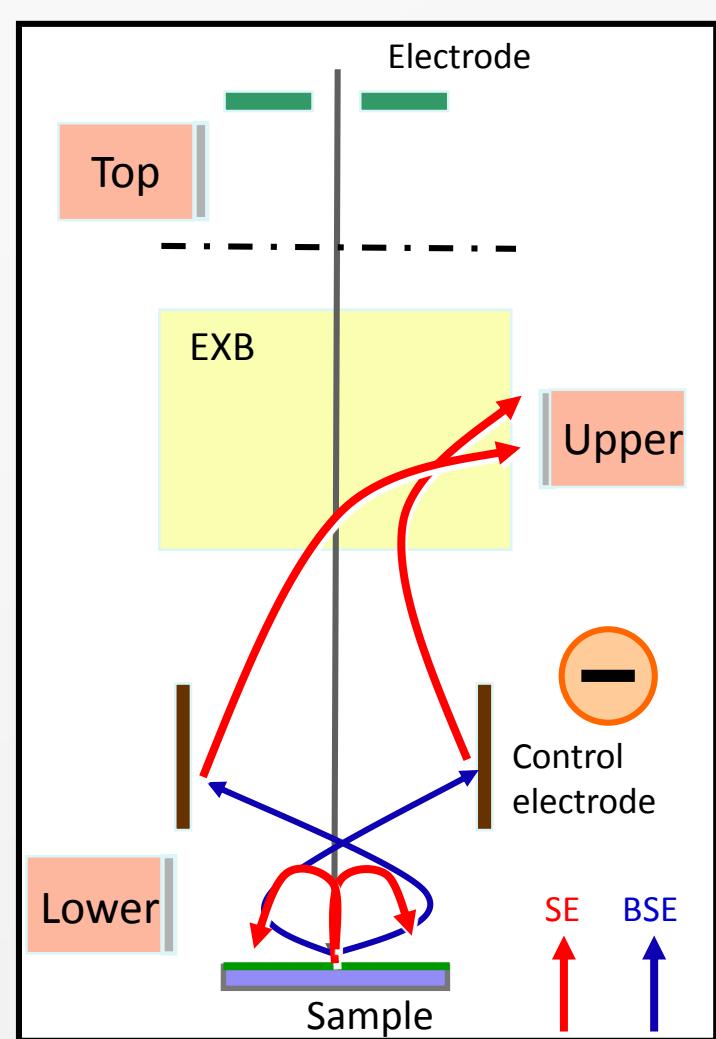


What you see depends on the detector !





What you see depends on the detector !



Sample : Photocatalyst
Vacc : 3.0V
Mag. : x 50k

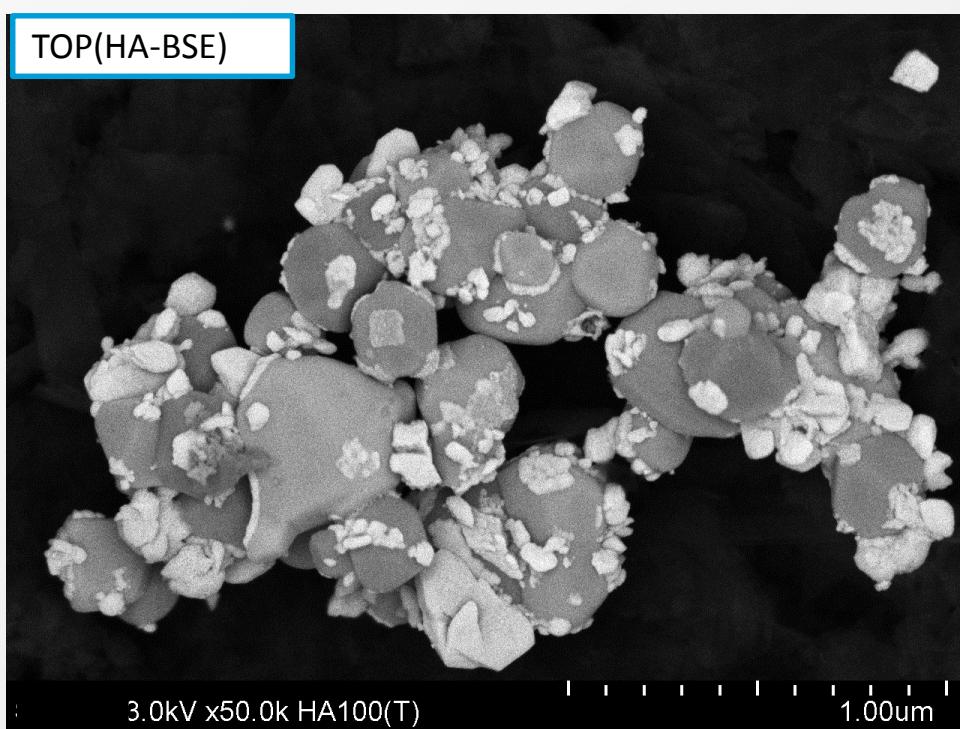
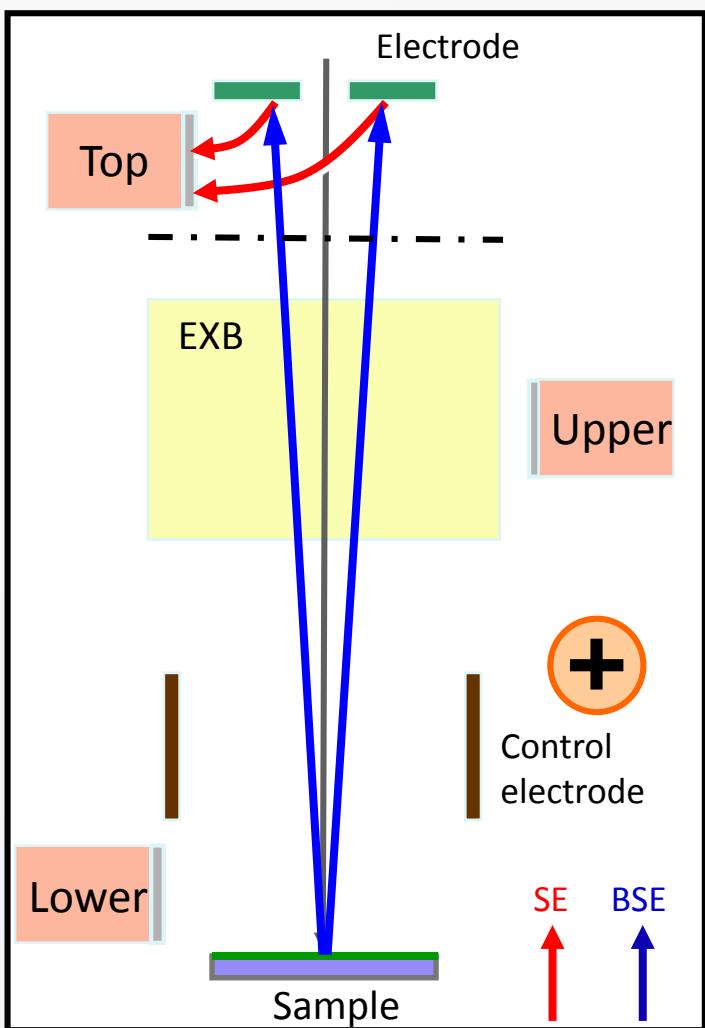
courtesy of
Model

Nagaoka University of Technology,
:Faculty of Engineering,
Dr. Kazunori Sato
:SU8020

Topographical + Compositional information



What you see depends on the detector !



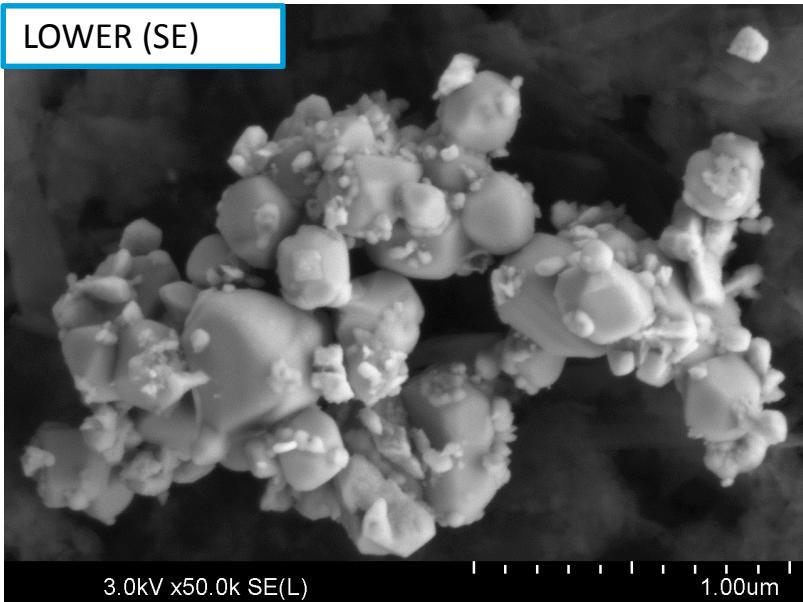
Compositional + Crystal information
(Less topographical information)

HR - SEM

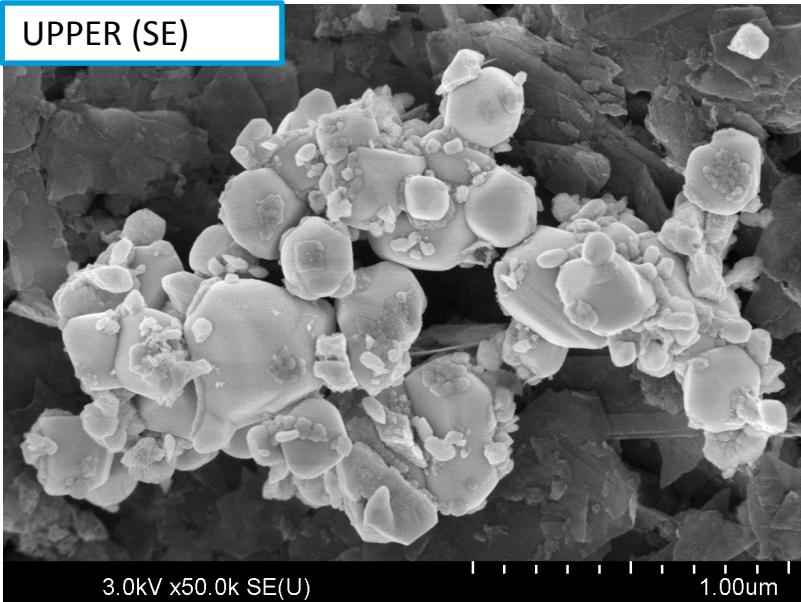
HR imaging



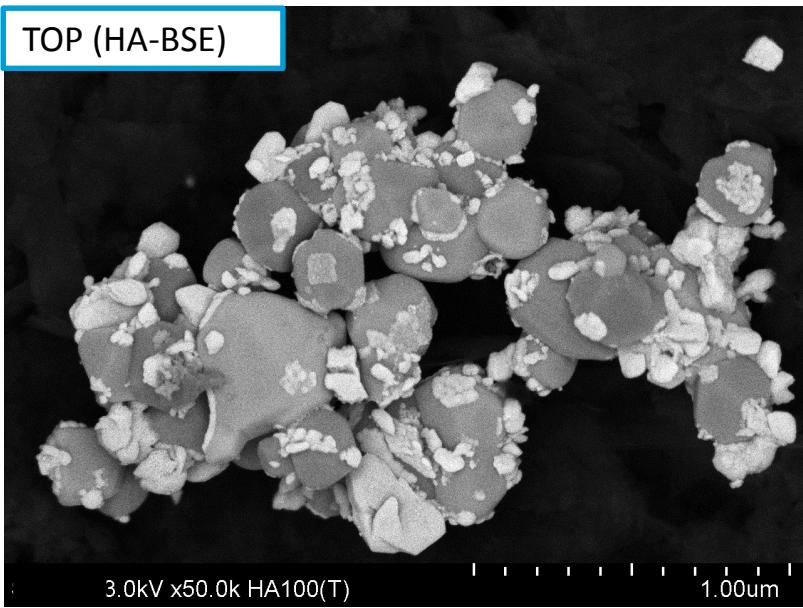
LOWER (SE)



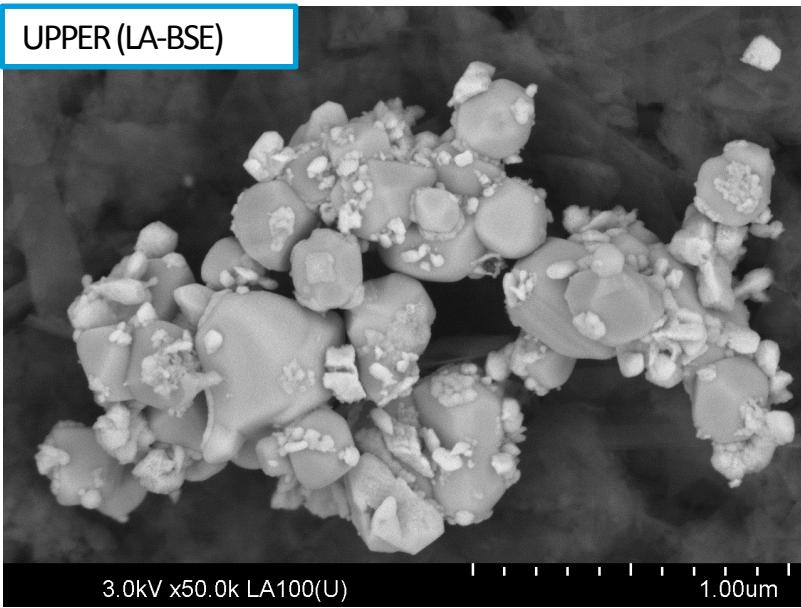
UPPER (SE)



TOP (HA-BSE)



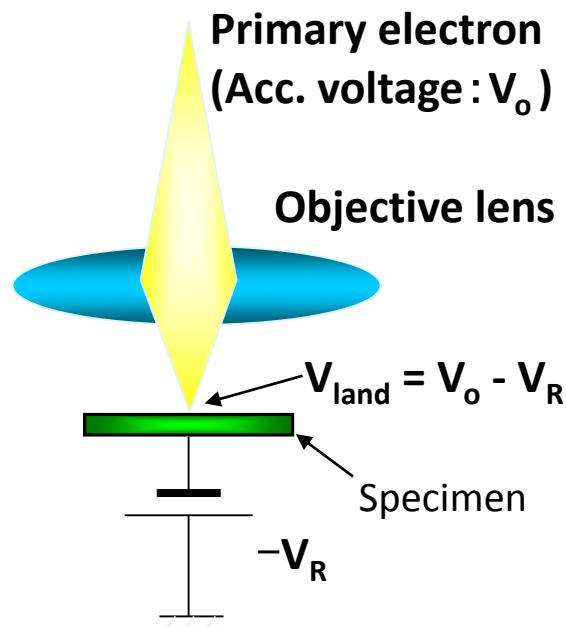
UPPER (LA-BSE)





Beam Deceleration Technology

A negative voltage is applied to the specimen to decelerate primary electrons before arriving at the surface.



$-V_R$: Deceleration voltage

Normal mode
(no deceleration)

$$V_R = 0 \text{ V}$$

$$V_{acc} = V_{land} = 0.5 \text{ kV}$$

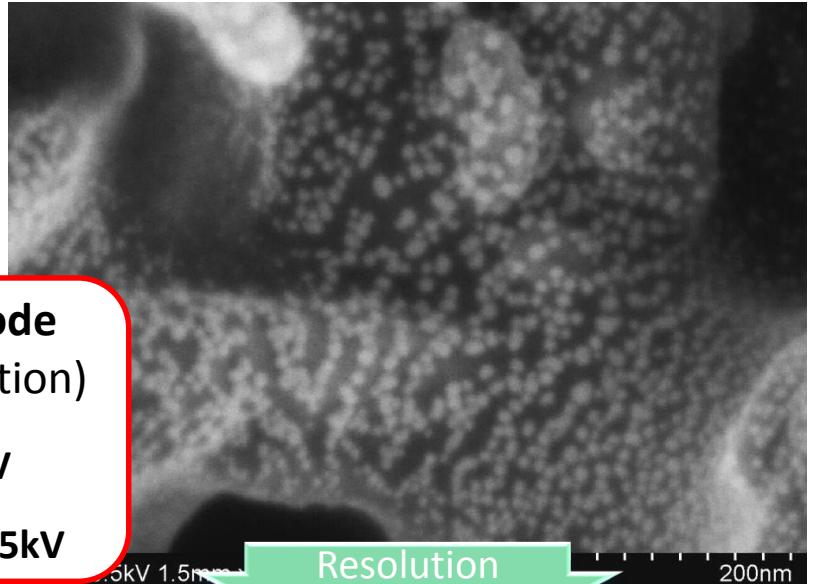
Landing voltage

Deceleration method

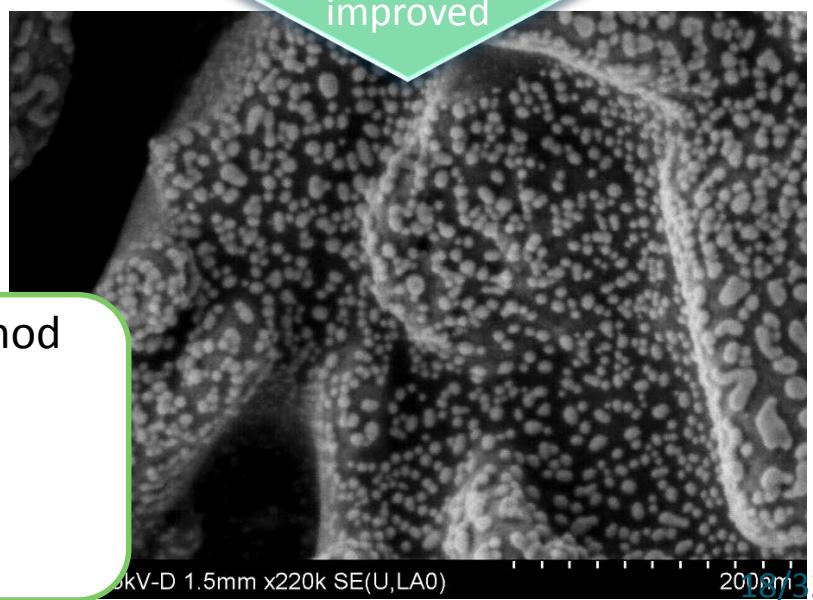
$$V_{acc} = 4 \text{ kV}$$

$$V_R \leq 3.5 \text{ kV}$$

$$V_{land} = 0.5 \text{ kV}$$



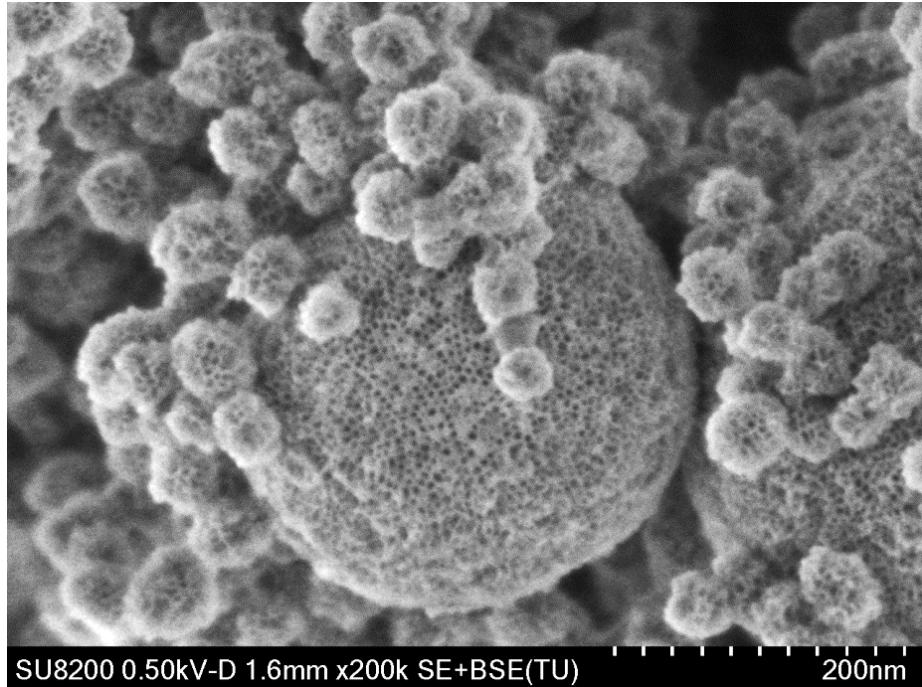
Resolution
improved



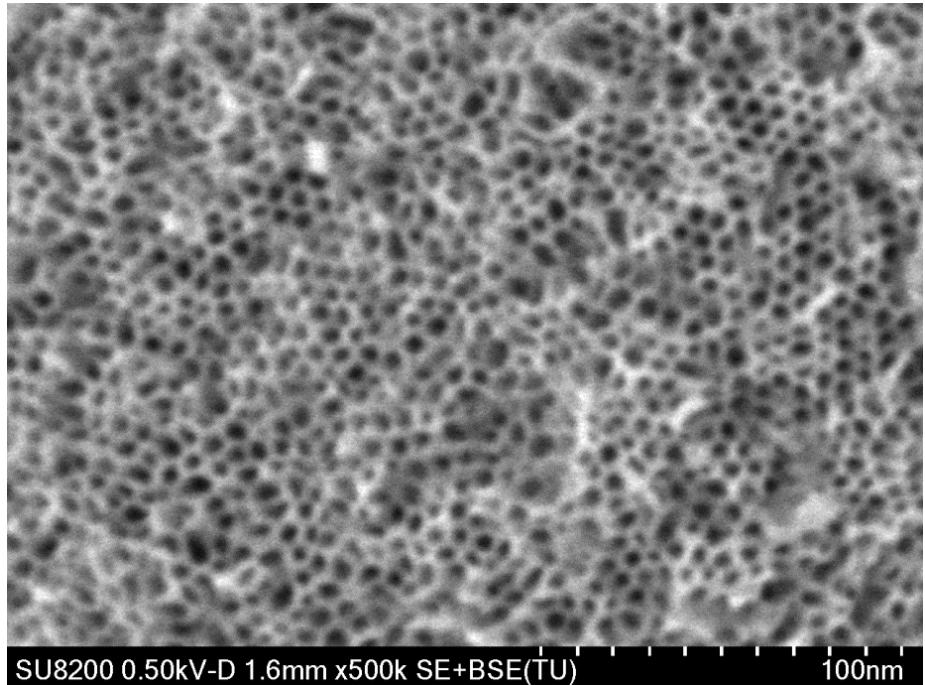


Beam Deceleration Technology

Low acceleration voltage observation for fine surface structure details

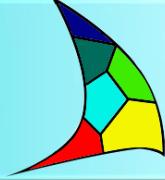


High resolution imaging of :
Mesoporous silica
Landing voltage : 500 V
Deceleration voltage : 3500 V
Mag. : x200k~x500k



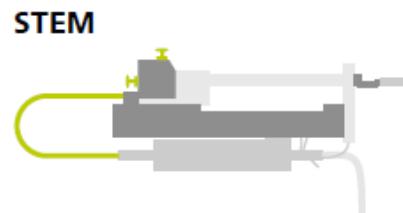


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The most versatile instrument for a materials scientist ?

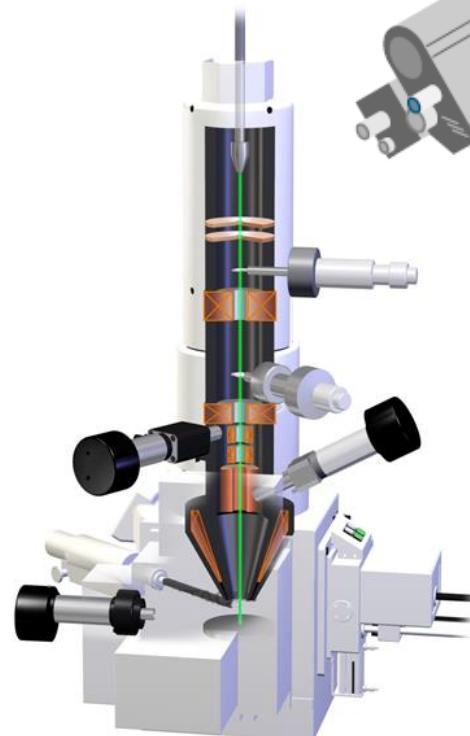
- Topography and morphology
- Chemistry
- Crystallography
- Orientation of grains
- In-situ experiments
- Reactions with atmosphere
- Effects of temperature



STEM



WDX



EDX

Plasma Cleaner



Stage options

(e.g. AFM, tensile, cryo, heating)

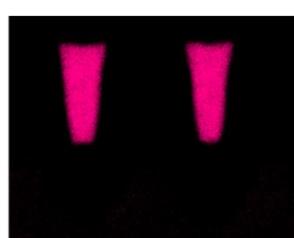
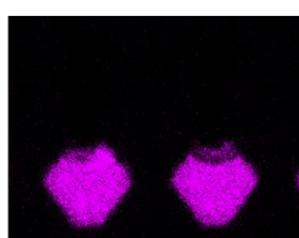
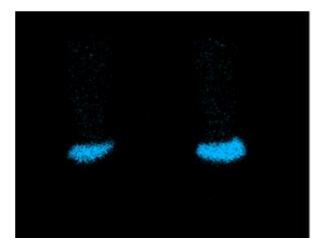
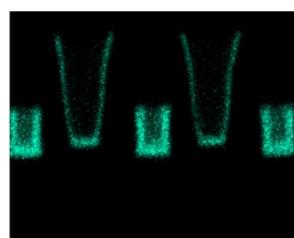
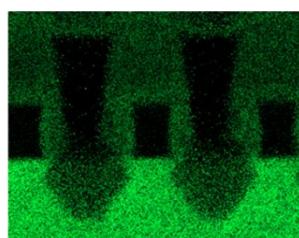
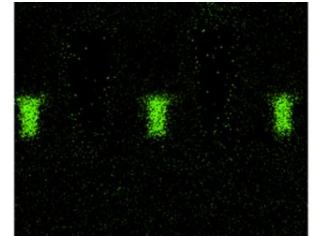
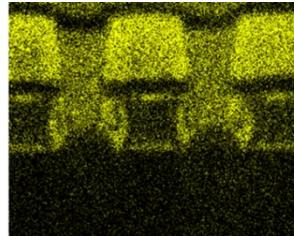
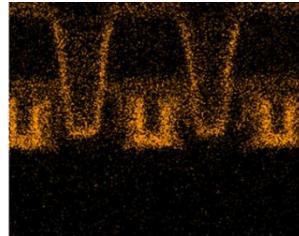
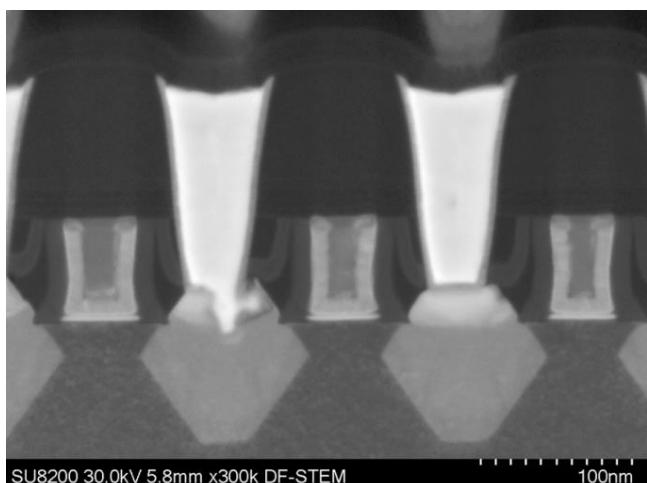
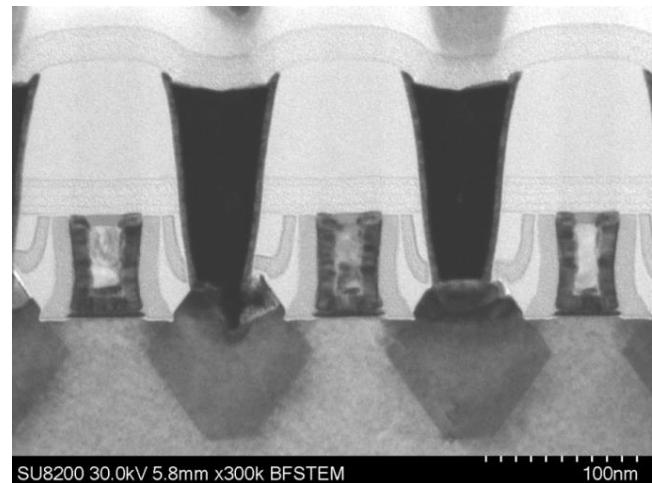


EBSD





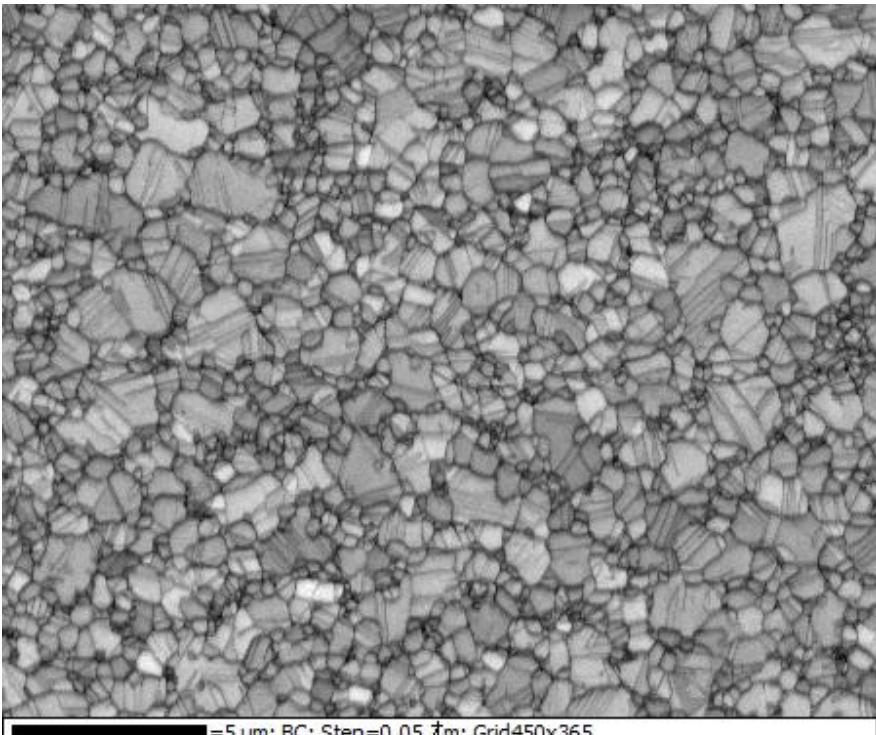
Combined multi-imaging and analysis



Specimen : PMOS transistor
Vacc. 30 kV
Mag. : x300K



EBSD* mapping



=5 μm ; BC; Step=0.05 $\text{\AA}\text{m}$; Grid450x365

Sample : Cu Line (100 μm)

Vacc : 25 kV

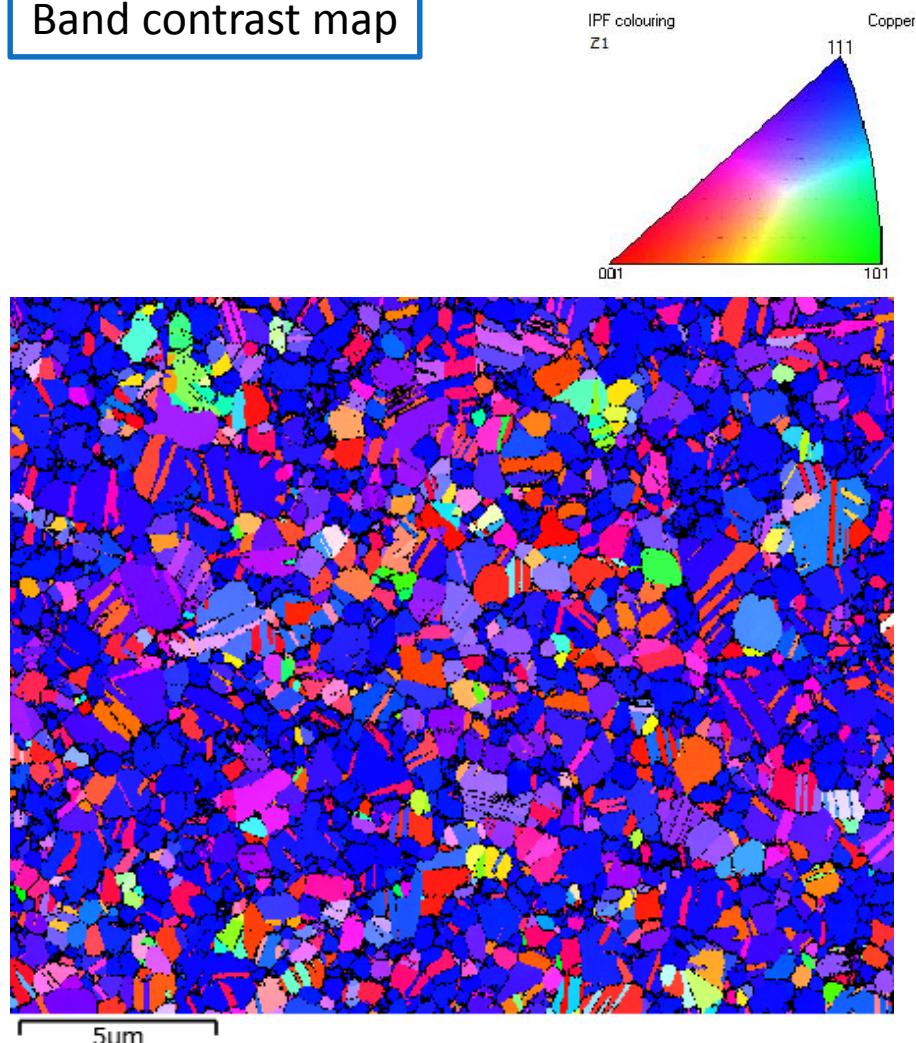
Specimen current: 3 nA

Mapping area: 22.5 μm x 18.3 μm (50nm step)

Time : 40 min

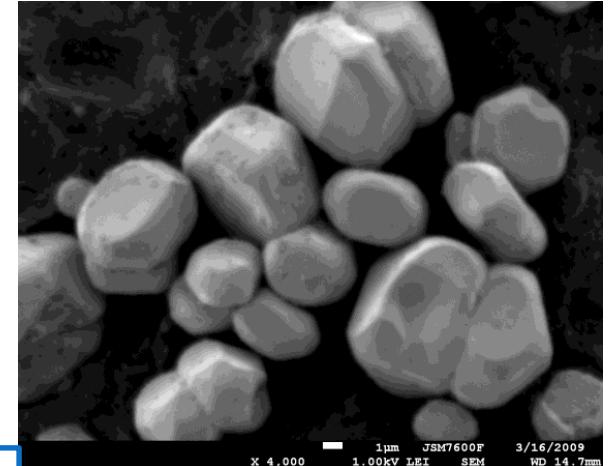
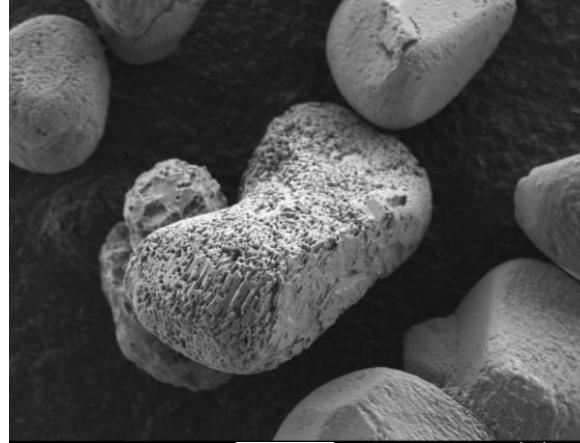
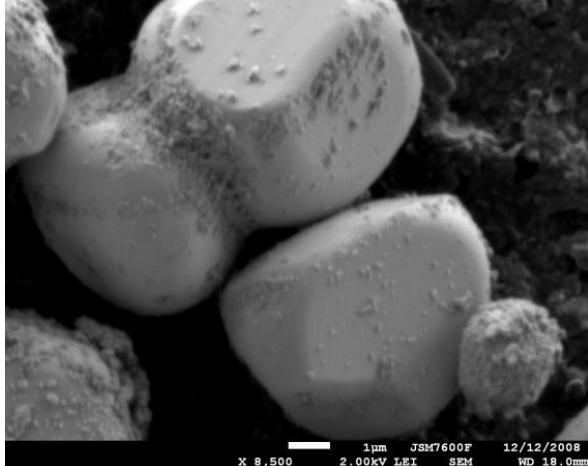
Inverse pole figure map
Z direction

Band contrast map

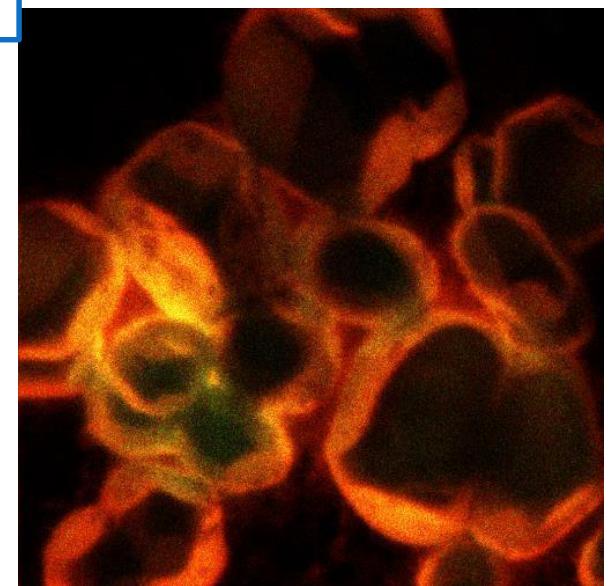
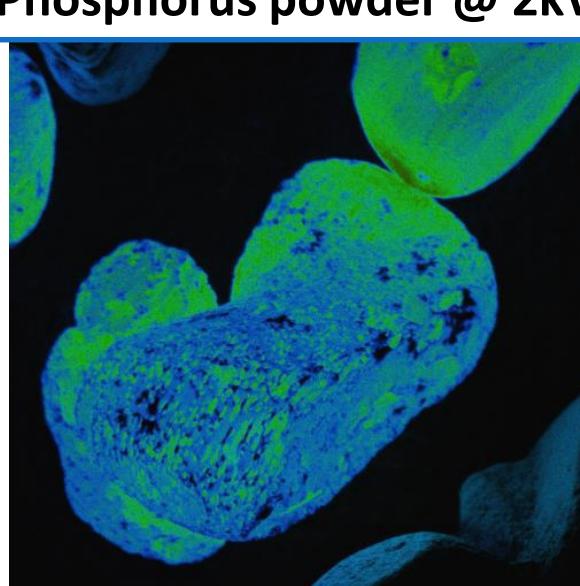
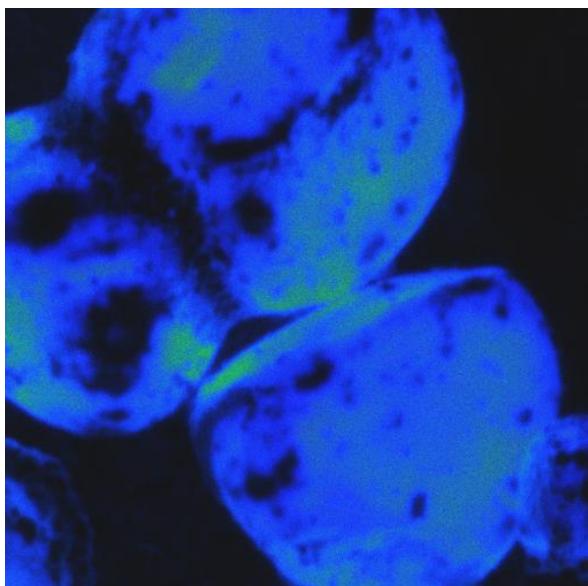




Cathode-luminescence (CL)



Phosphorus powder @ 2kV





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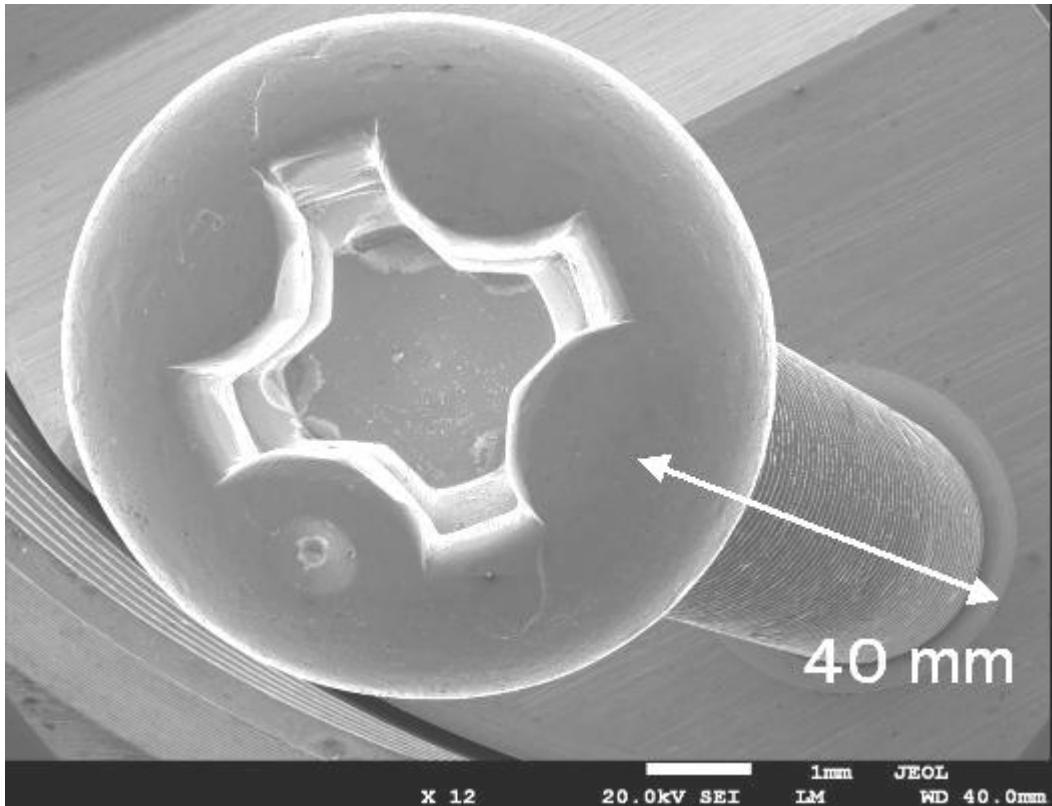
FABRICANT	TESCAN	JEOL	HITACHI	FEI	ZEISS
Modèle	MIRA3	JSM7800F	SU8220	TENEO	SIGMA 500
Source d'électron	Chaudé	Chaudé	Froide	Chaudé	Chaudé
Résolution	3.5 nm @ 1 kV	1.2 nm @ 1kV	1.1 nm @ 1kV	1.4 nm @ 1 keV	1.6 nm @ 1 kV
Budget MEB (k€)	338,4	398	479	devis en attente	448,3
Budget EDS (k€)	50	50	69,4	devis en attente	73
Plasma cleaner (k€)	inclus	27	non inclus	devis en attente	non inclus
contrat maintenance tout inclus (k€)	12,7	12	12,1	devis en attente	15,6
Budget total (k€)	401,1	487	560,5	?	536,9
BASE SAV régionale	PARIS	CHATELERRAULT	PARIS	PARIS	PARIS



FABRICANT	TESCAN	JEOL	HITACHI	FEI	ZEISS
Avantage	PRIX	RESOLUTION	RESOLUTION (cold FEG)		RESOLUTION
	Faible grossissement x2	Profondeur de champ	Ultra low landing voltage (10 volts)		Colonne Gemini réputée
		Charge Suppression Scan			InLens SE/BSE axial
		Courant de sonde élevée			
		Rapport qualité/prix			
		n°1 national MEB/TEM, proximité du SAV	n°1 MEB semi-cond (SAV connu)		N°2 MEB national
Inconvénient	RESOLUTION	PRIX	PRIX		PRIX
	Position sur le marché				
	SAV ?				



Large depth of focus mode



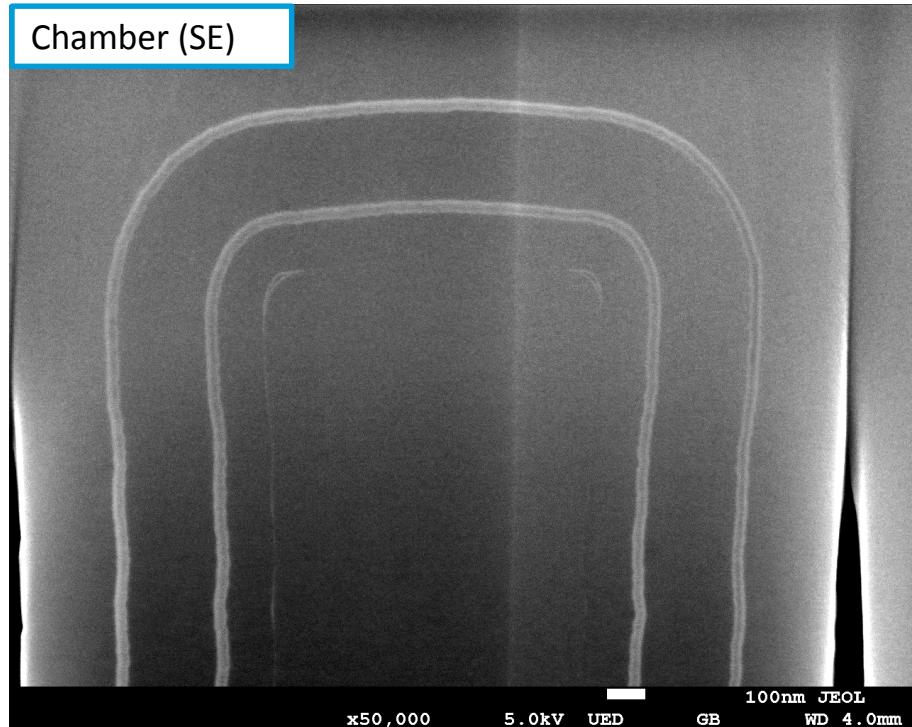
- The LDF mode provides very large depth of focus
- Useful when you observe a large rough specimen



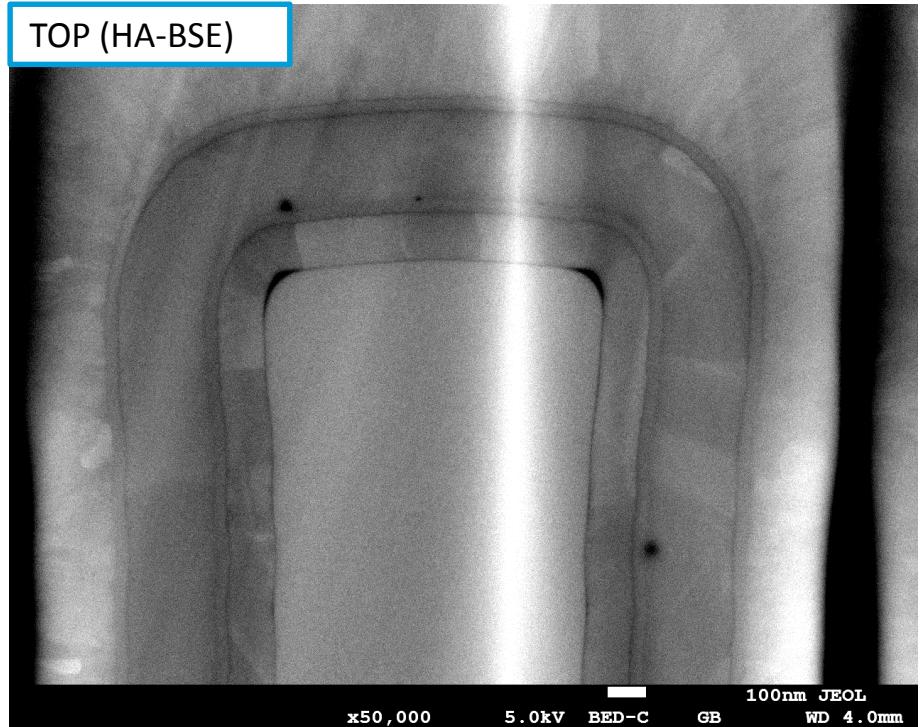
Construction analysis – CP cross section

DEMO JEOL / Hitachi

Chamber (SE)



TOP (HA-BSE)

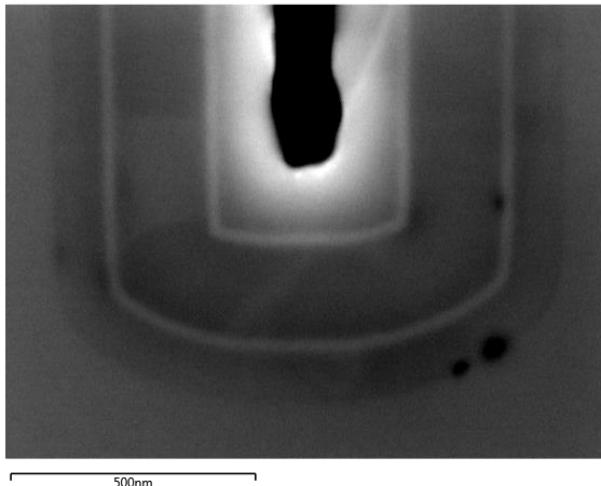




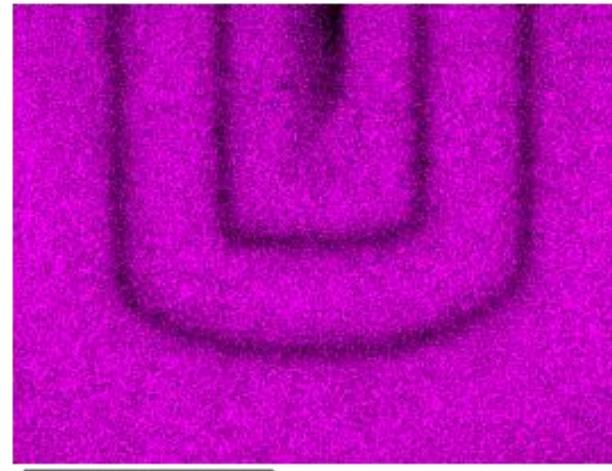
Construction analysis – CP cross section

JEOL / Hitachi DEMO

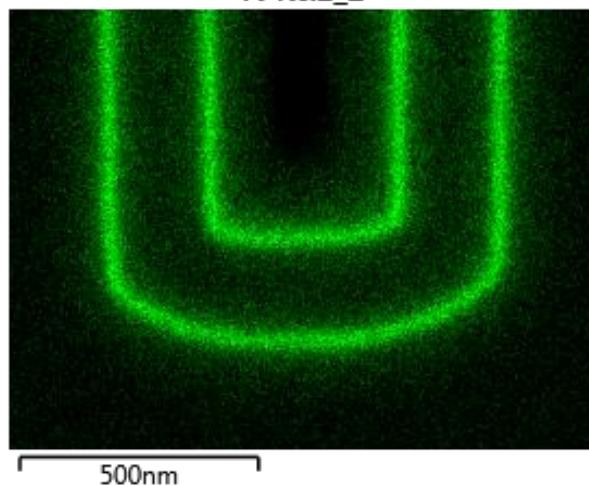
Electron Image 7



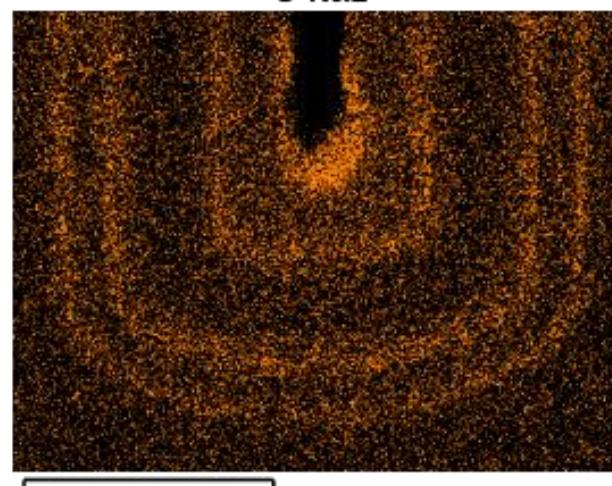
Si K α 1



N K α 1_2



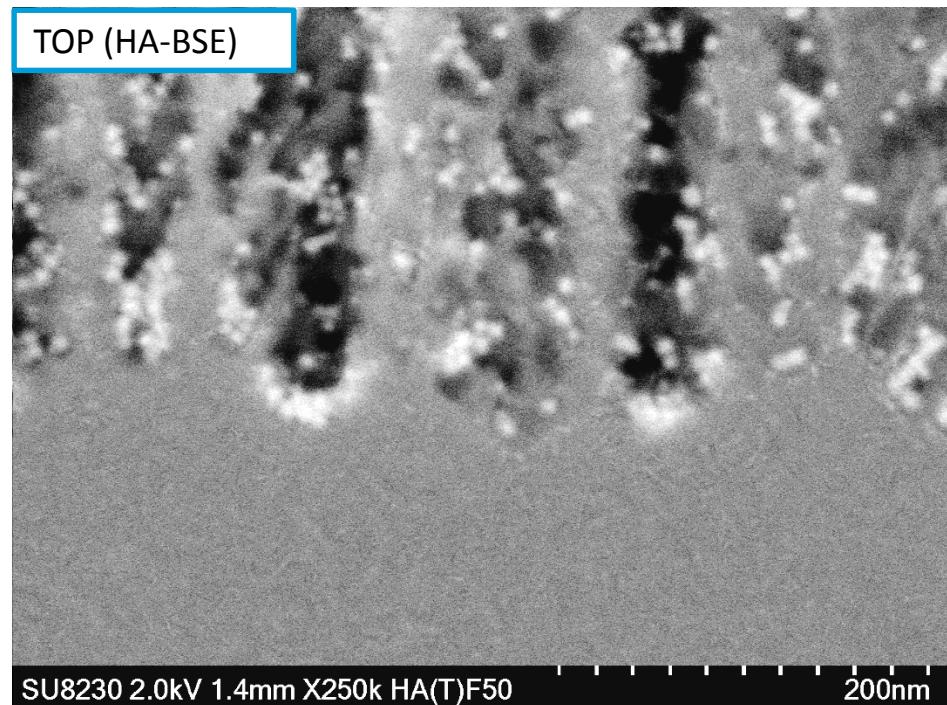
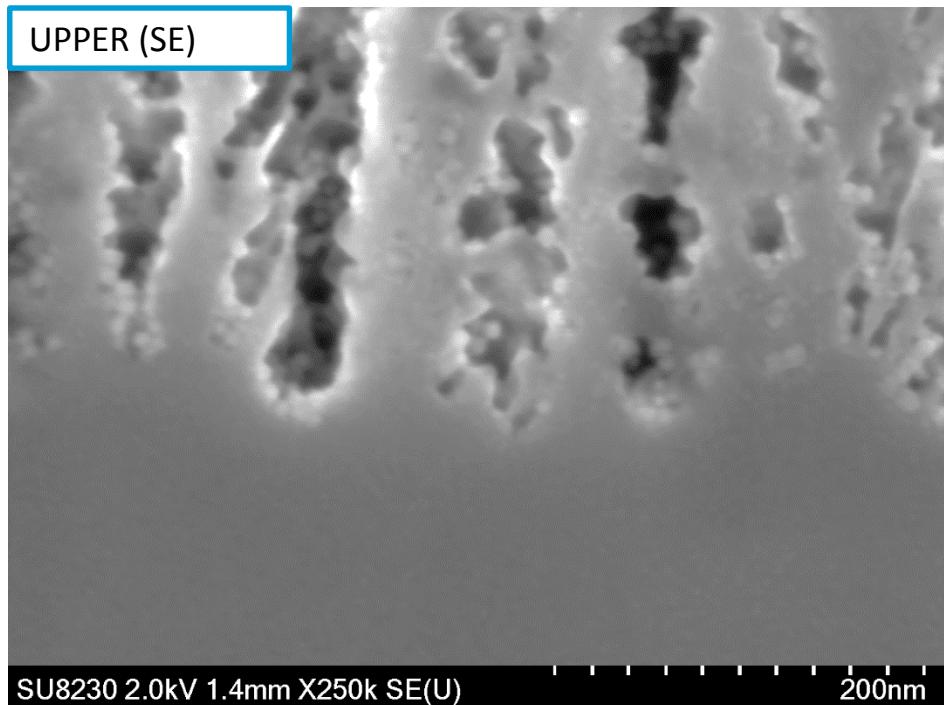
O K α 1





HR imaging – CP cross section

DEMO Hitachi



Porous Silicon with FeCo particles

Sample prepared by B.Bardet, PhD student – TOURS2015



Merci de votre attention !