SEM Basics

Spectral Solutions

Mats Eriksson

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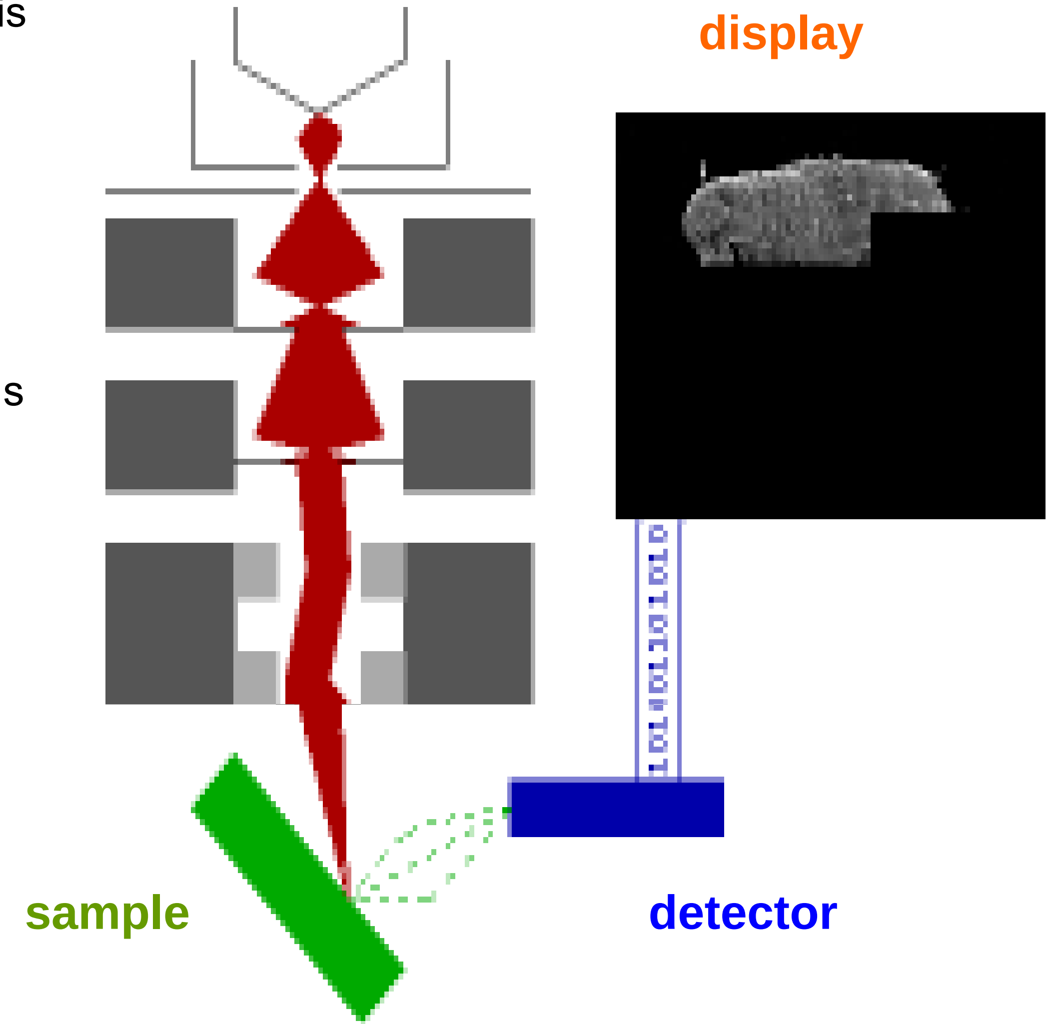
Agenda

- Why SEM ?
- How does it work ?
- Sampling
- Electron sources and optics
- Electron and sample interaction
- Contrast mechanisms
- Interaction volume
- Imaging problems
- Astigmatism
- Variable pressure
- X-ray microanalysis

How the SEM works

- A finely focused beam of electrons is moved across the specimen one point at a time
- These stimulate electron emission which travels to a detector where they are collected and amplified
- The image is assembled - like a mosaic - from the million or so pixels sequentially examined by the beam and presented on a computer screen



Macroscale to meso-scale…

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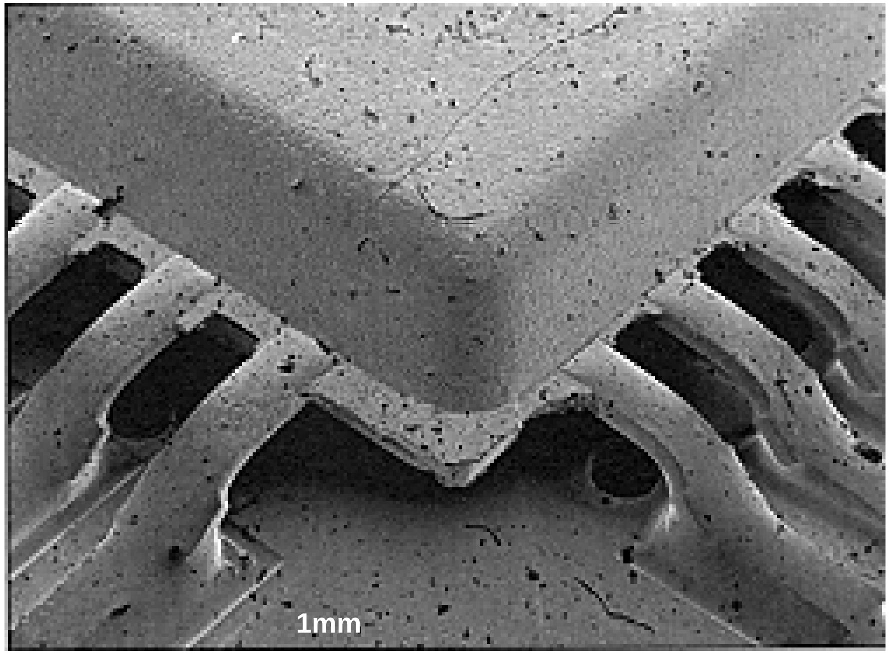
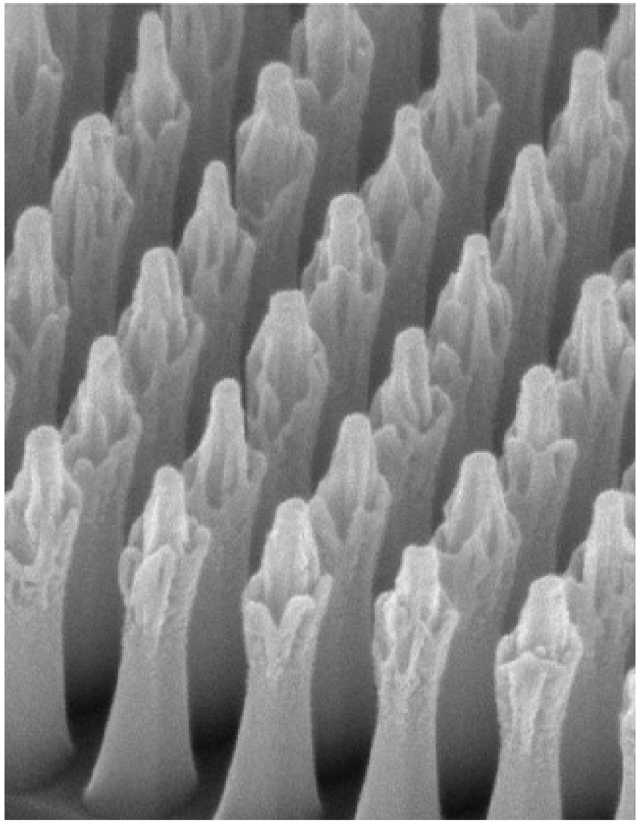


Packaged microchip ready to ship – Field of View 5mm S3400

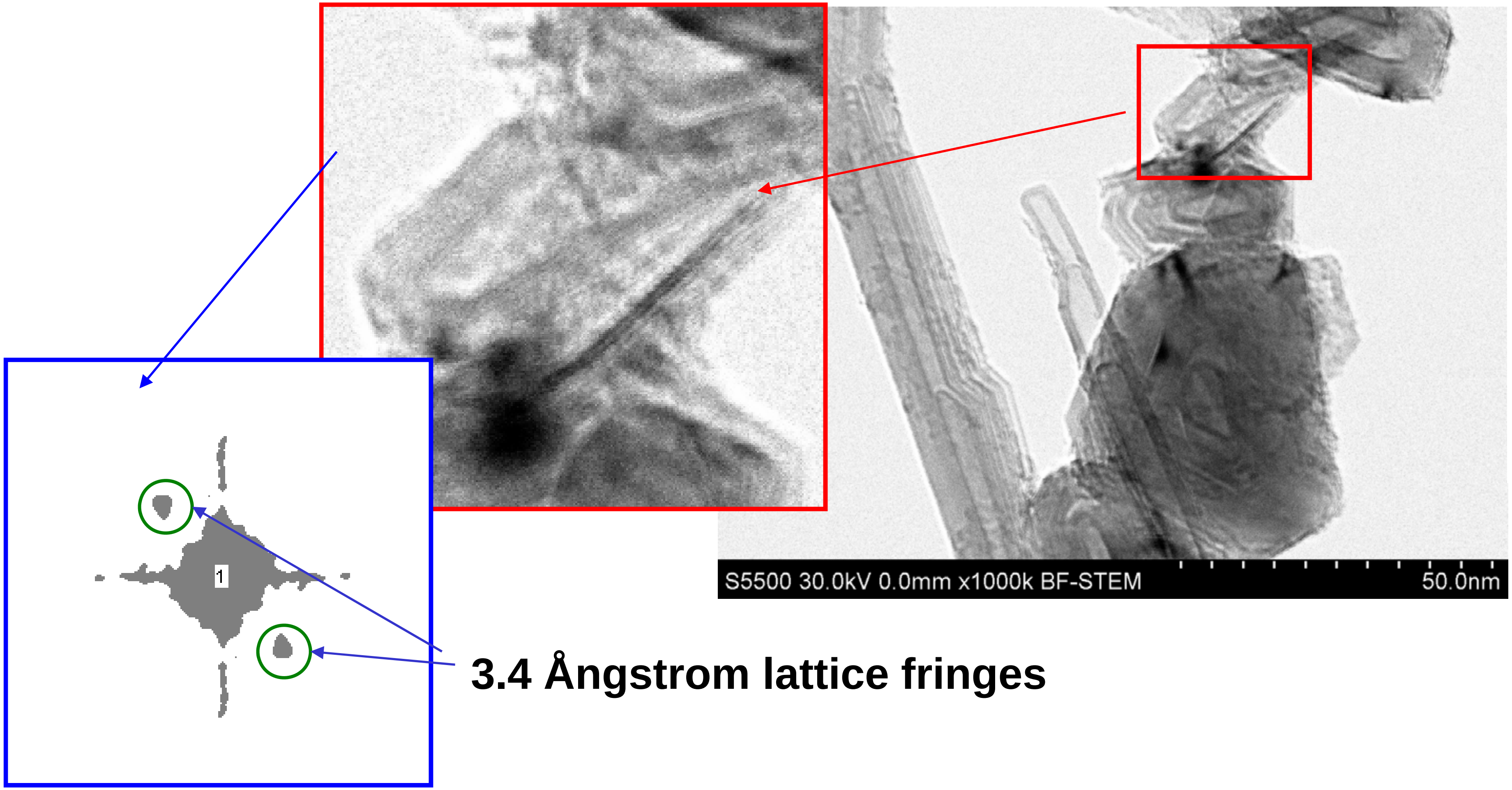
Imaging in context - low and high magnification for problem solving and flexibility of imaging mode

Ordered array of Carbon NanoRods on Si Field of view 1 mm

S4700



....To true nanoscale



SMART FFT analysis

What is the SEM?

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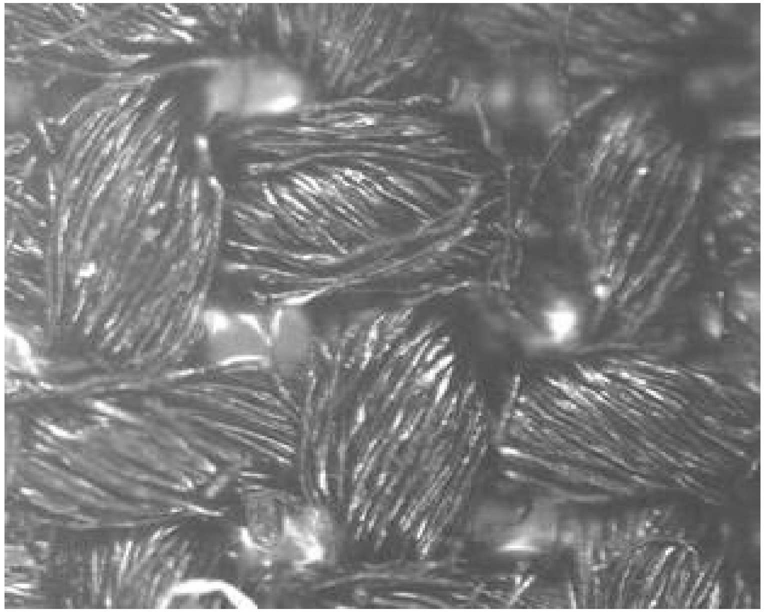

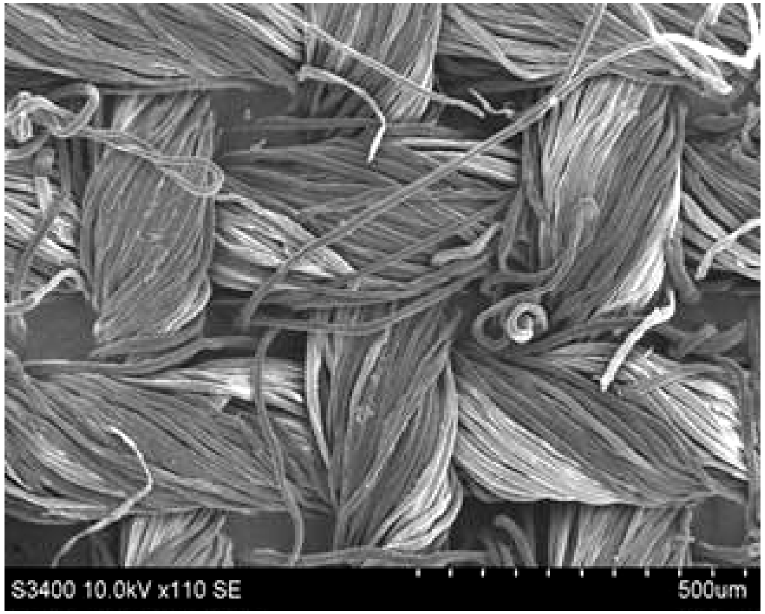

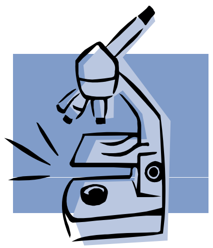

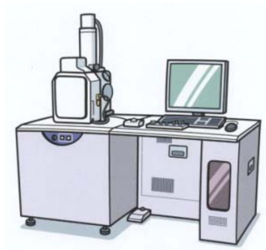

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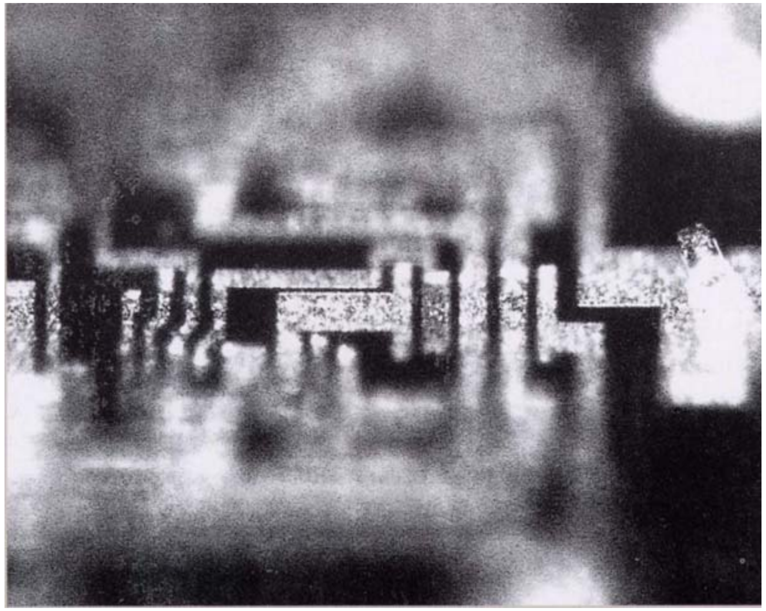
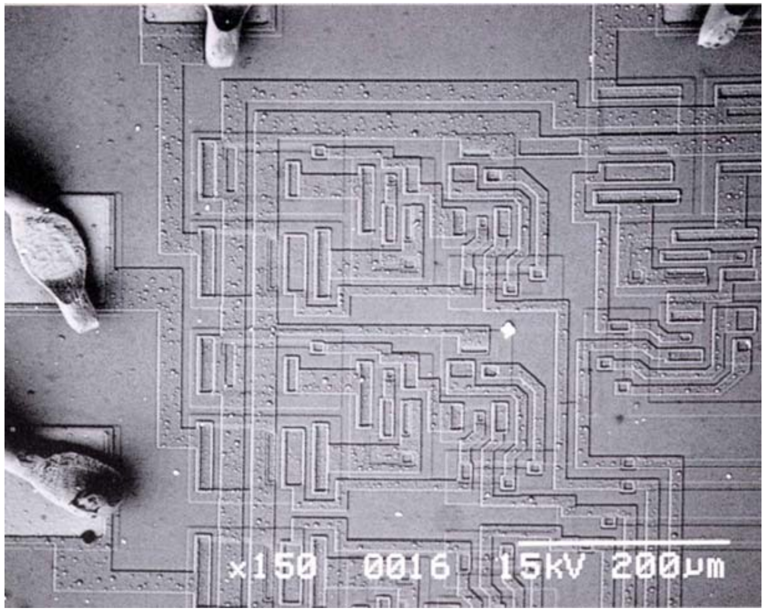
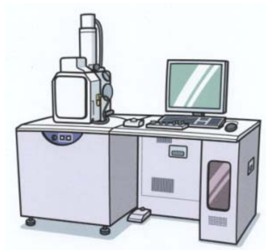
What is the SEM?

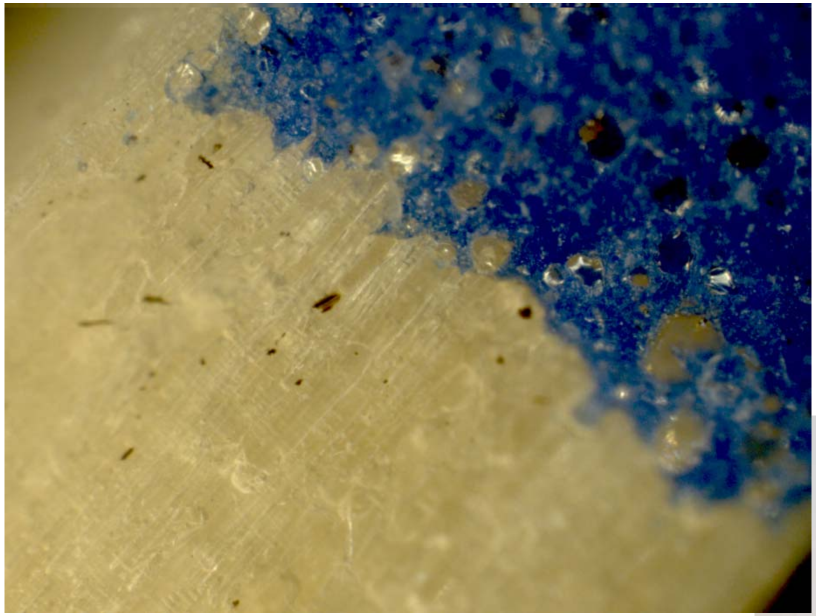


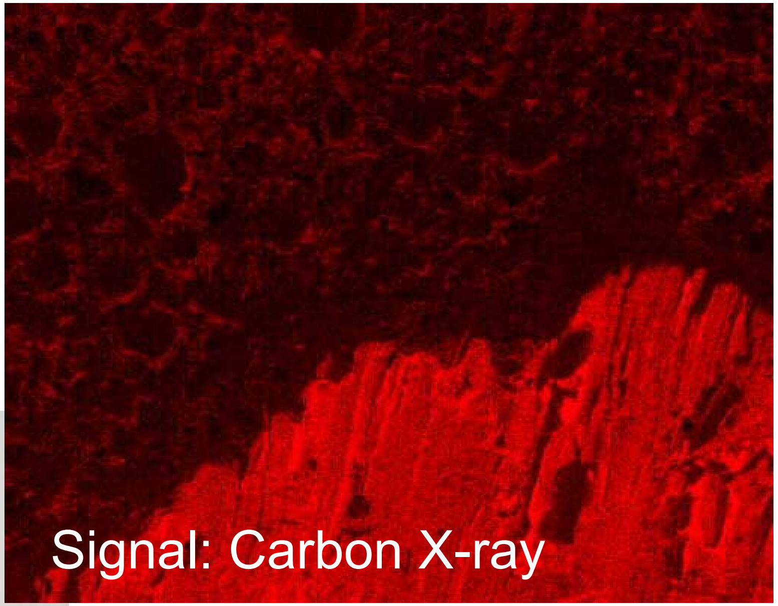


Better depth of focus

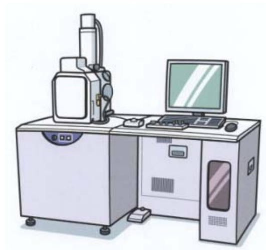


What is the SEM?





Better depth of focus



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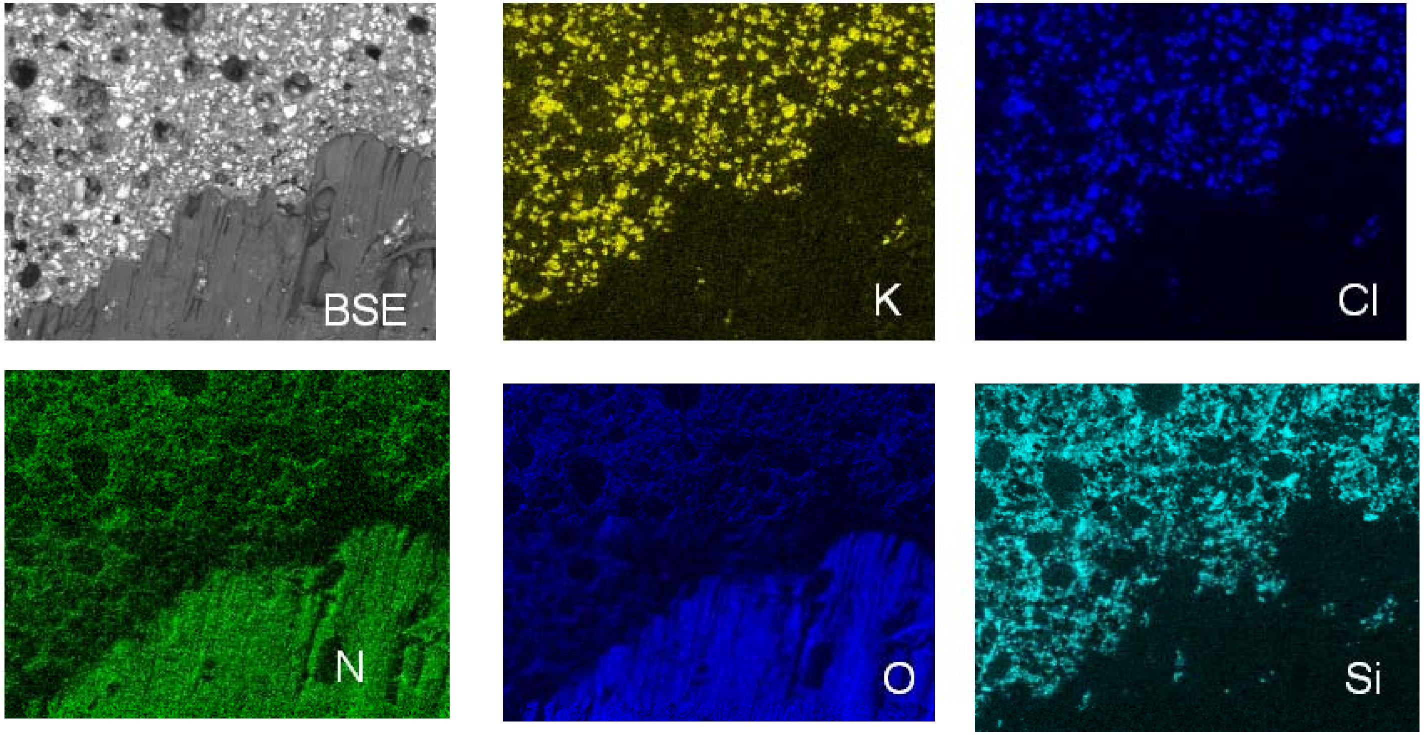
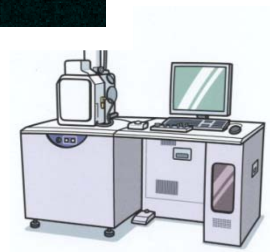


Possibility for chemical analysis



What is the SEM?

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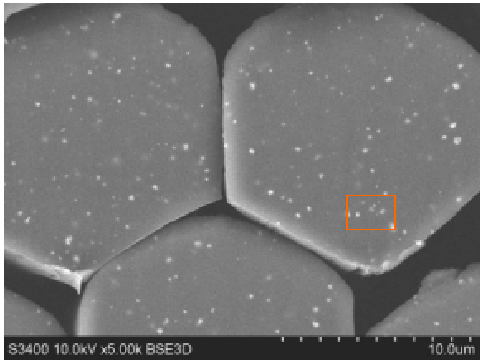
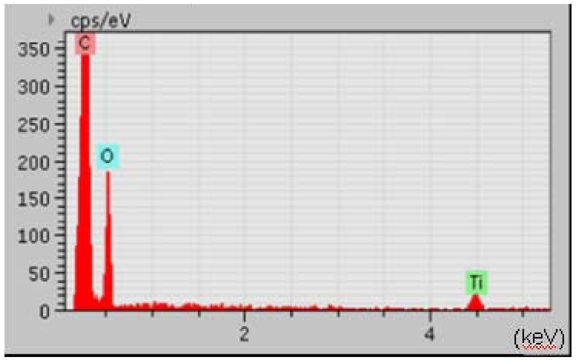
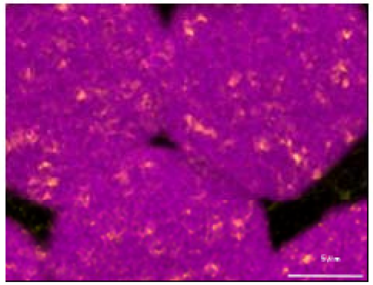


Backscattered electron image (×5,000 magnification)

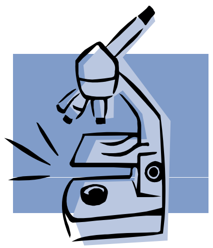
The cross-sectional structure of the fiber was observed with a backscattered electron detector. This permits conformation of how the white particles are dispersed in the fiber.



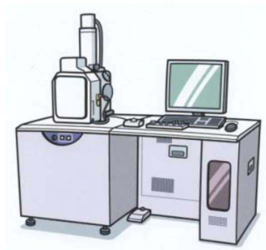
X-ray spectrum of area enclosed in rectangle above



C Ti X-ray mapping image of C and Ti



Possibility for chemical analysis



What is the SEM?

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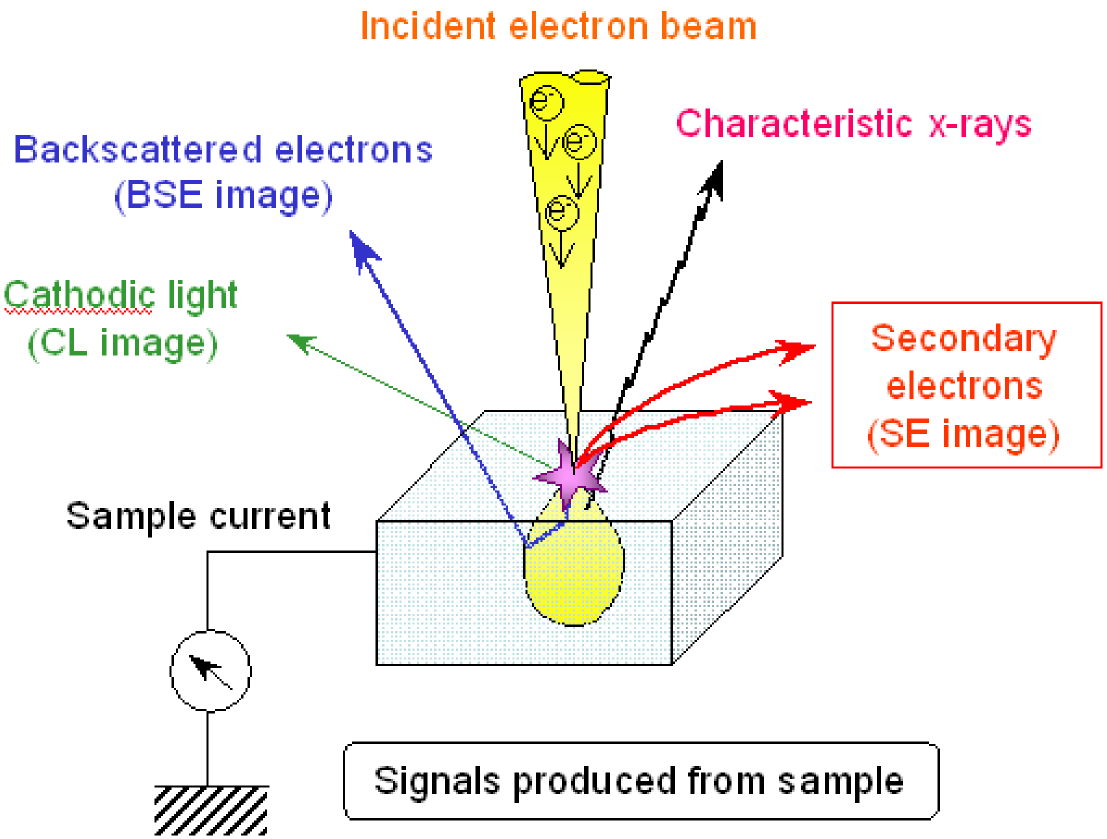

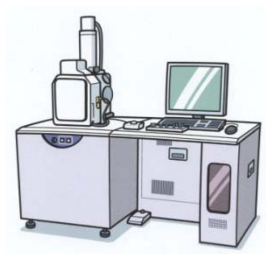

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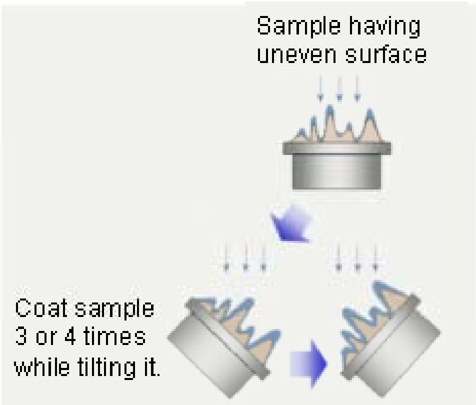
Sampling

		 Conductive both-side tape	 Conductive paste				
Name	Specimen stub	Double sided Carbon Tape	Conducting Graphite Paint	Tweezers	Wafer Tweezers	Blower	Diamond Scribing Pen

Sampling

Magnetron sputtering device



Coating of sample having uneven surface

(1) Purposes of coating

- To make the sample surface conductive (prevention of charge-up)
- To increase the production rate of secondary electrons (increase image information)
- To prevent damage to sample

Sampling

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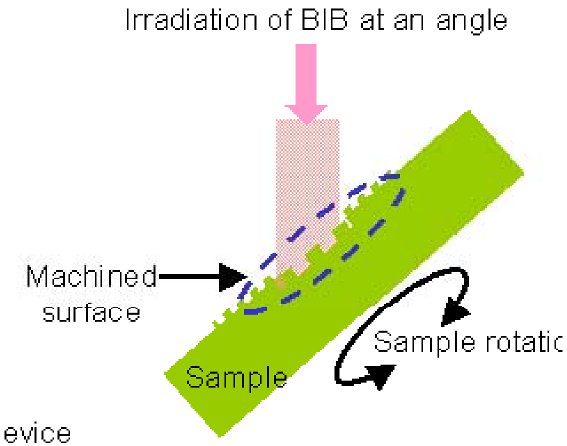
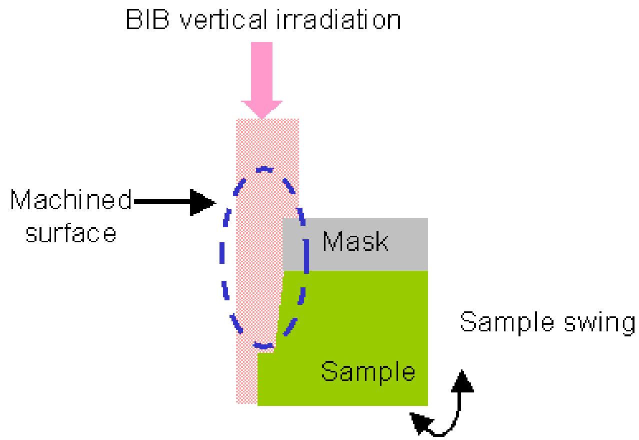
Outer view of IM-3000 ion milling device

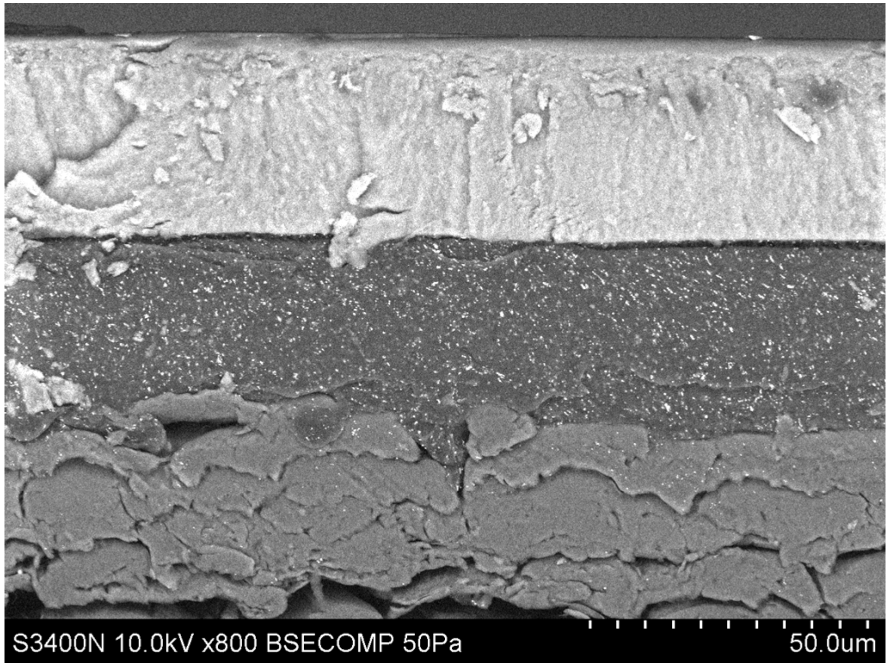




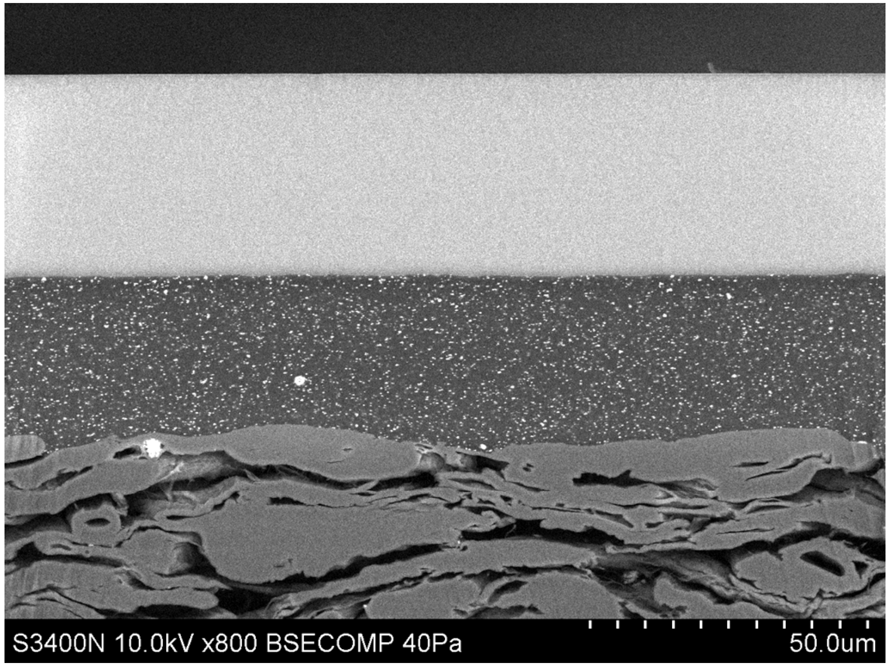
(1) Purposes of BIB (Broad Ion Beam) milling:

- To make the sample surface flat for EDX and EBSD analysis
- To eliminate oxid films or contamination, to enhance crystal orientation contrast
- To prepare “stressfree” a proper cross section of complex compound materials

Sampling



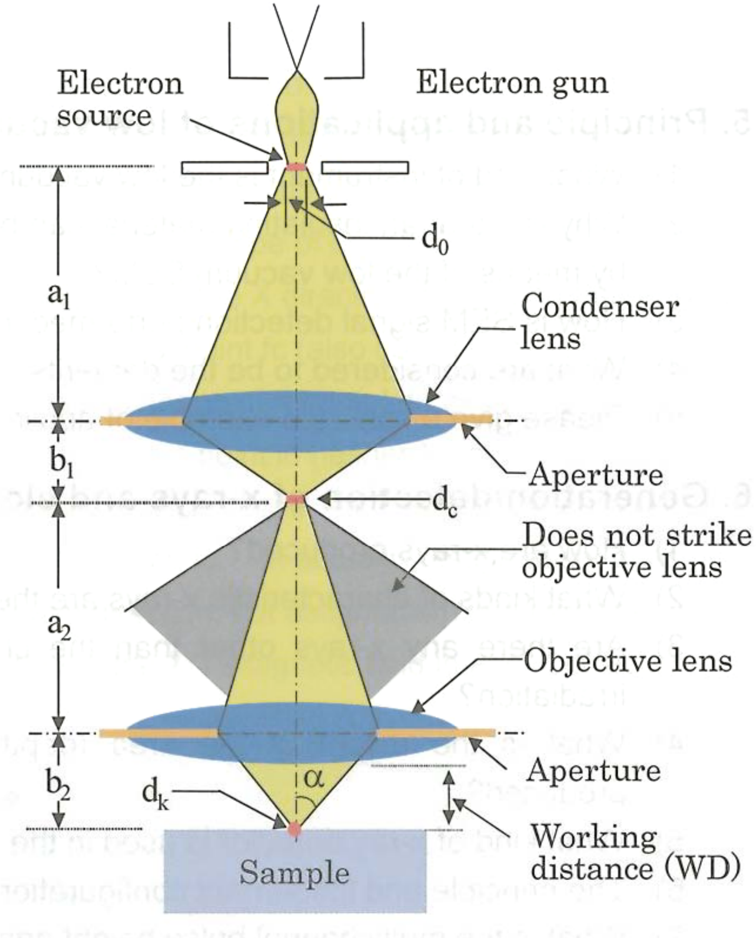
(a) BSE image after razor cutting



(b) After Milling (BSE Image)

Coated paper for colour printer

Electron beam formation



$M_1 = b_1 / a_1$

$M_2 = b_2 / a_2$

$d_k = d_0 * M_1 * M_2$

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Electron beam formation

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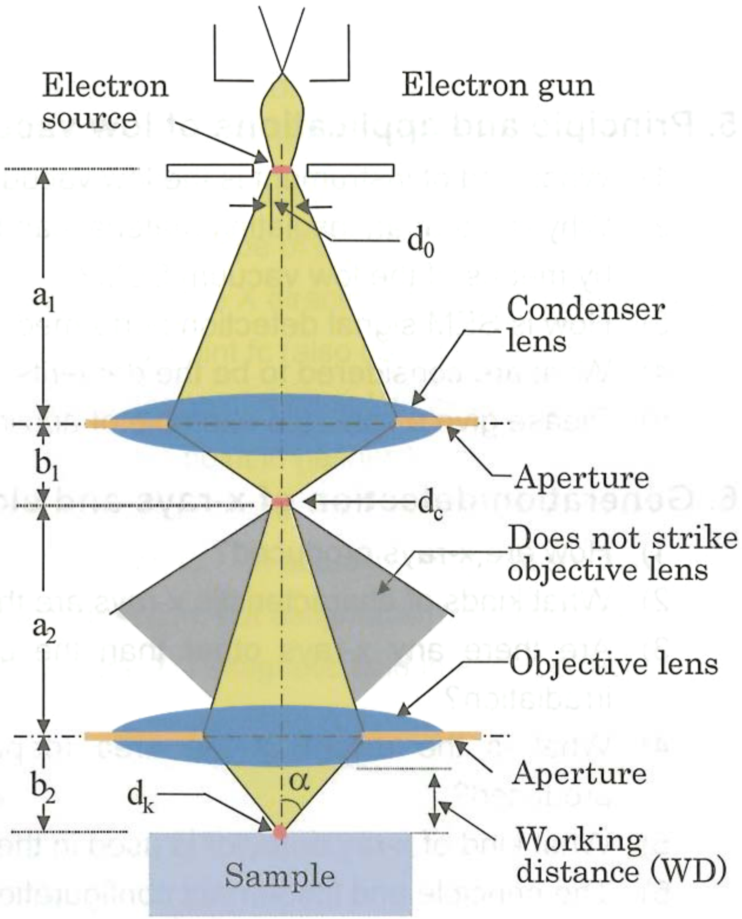
$M_1 = b_1 / a_1$

$M_2 = b_2 / a_2$



$$d_k = d_0 * M_1 * M_2$$

How can we obtain an even finer electron beam ?

Electron Optics

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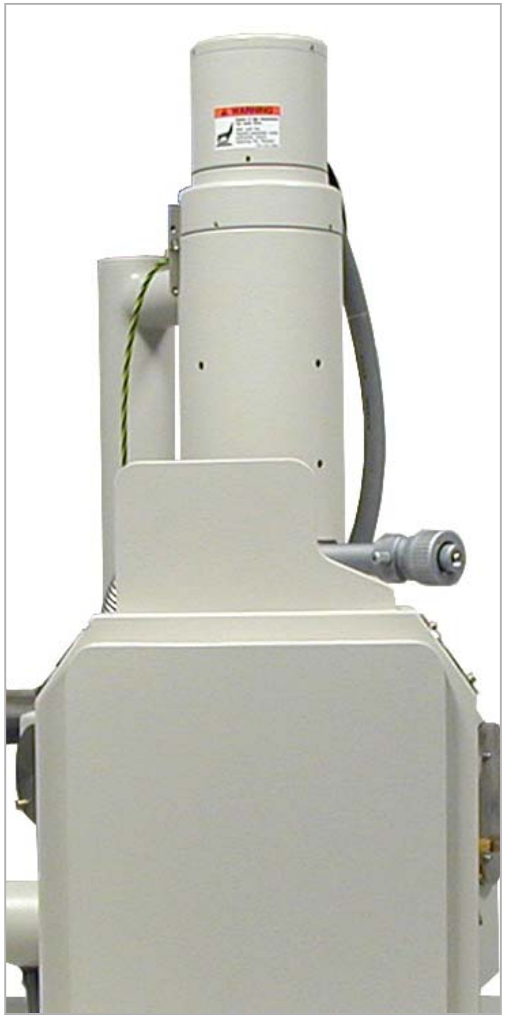

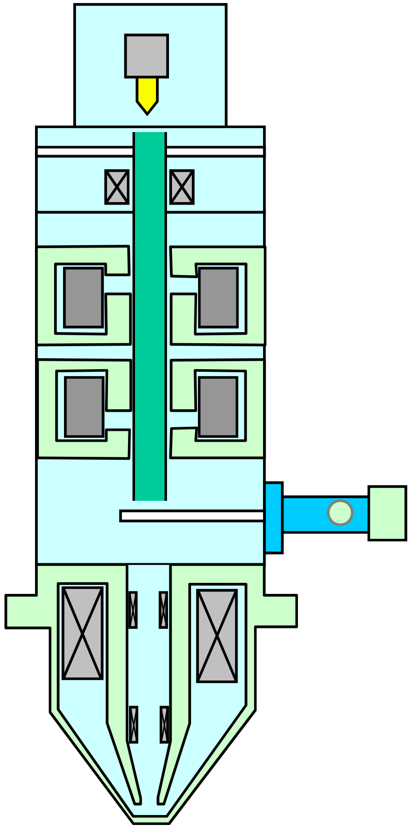

Tungsten (W) thermionic filament.
- pre-centered, self adjusting (ABS).

Auto Beam Setting (ABS).
- automatical optimization of filament saturation and beam axis.

Apertures integrated in removable Liner Tube (HITACHI patent)
- all fixed apertures can be accessed by the user for easy exchange

Quad Bias
- strong emission current also at low accelerating voltages

Objective aperture alignment
- electronical optimization of beam (AAA).

Objective lens.
- optimized for high resolution even at low beam energies.
- large field-of-view, also unlimited in VP.

Source Comparison

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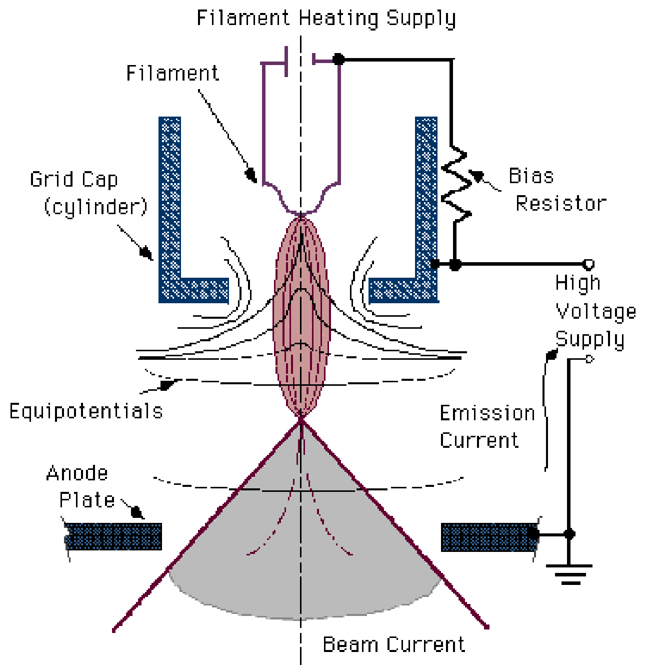
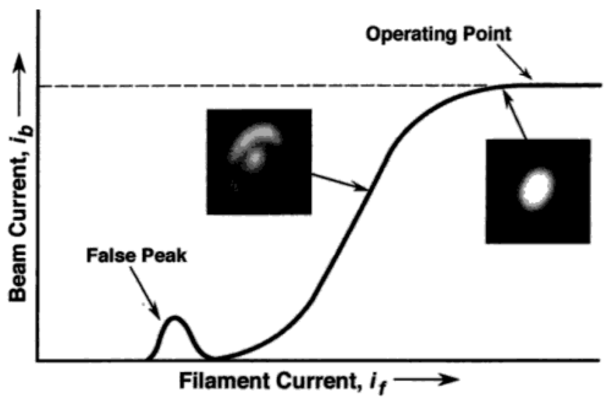
	W	LaB6	Schottky FE	Cold FE
Beam diameter	1 – 2 μm	1 – 2 μm	10 – 25 nm	3 - 5 nm
Temperature	2300° C	1500° C	1500° C	Room temp
Brightness	1	10	500	1000
Energy spread, ΔE	2.0 eV	1.5 eV	0.5 eV	0.2 eV
Stability,%/h	0.1 %	0.2 %	0.2 %	5 %
Probecurrent	50 nA -1 μA	50 nA -1 μA	>100 nA	20 nA
Life time	1 month	6 months	18 months	5 years
Gun vacuum	10^{-5} Torr	10^{-7} Torr	10^{-9} Torr	10^{-11} Torr

Electron Source: Tungsten Filament

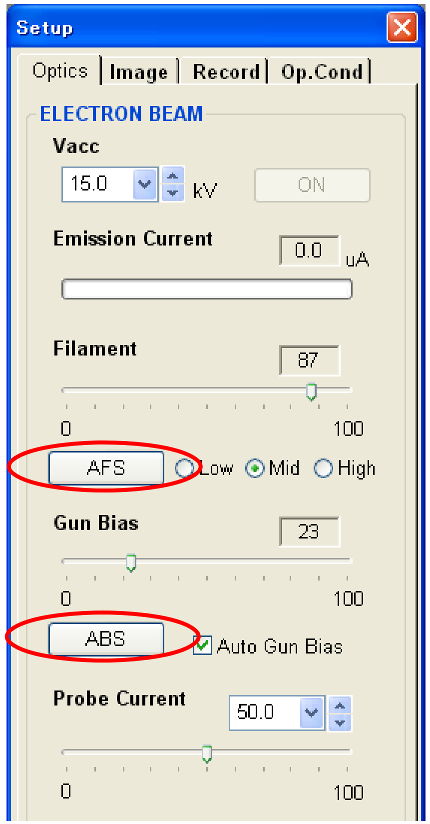




Tungsten filaments,
operating life about 100 hours.

Cathods are delivered pre-centered,
no fine-mechanical adjustment work required

Re-adjustement of optics after filament exchange (filament saturation, beam axis) is automated - a simple mouse-click is enough. (ABS-function)



Quad-Bias: More Probe Current - also at low Beam Energies

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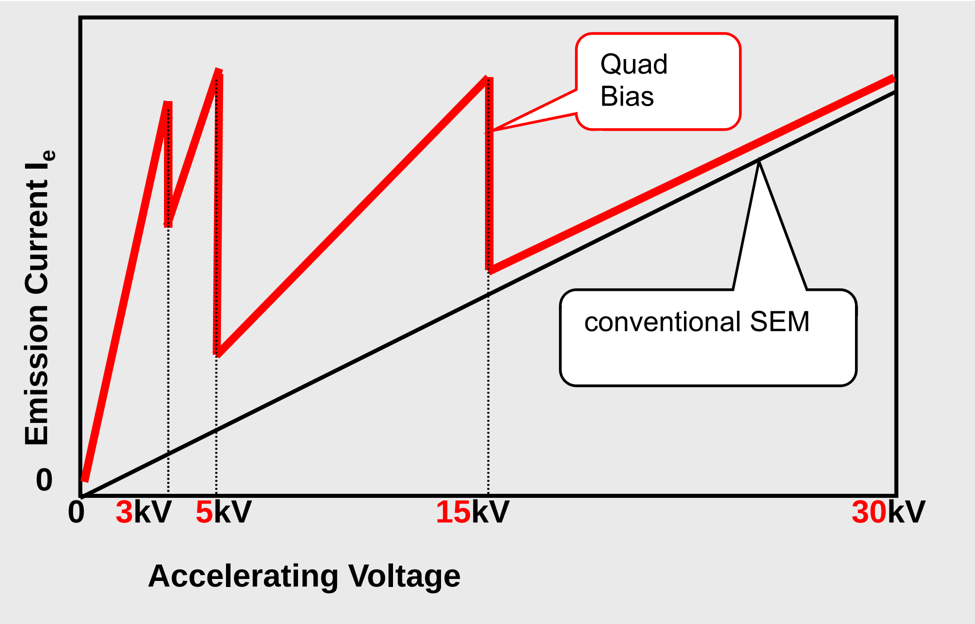
Quad Bias circuitry allows high probe currents even at low accelerating voltages.

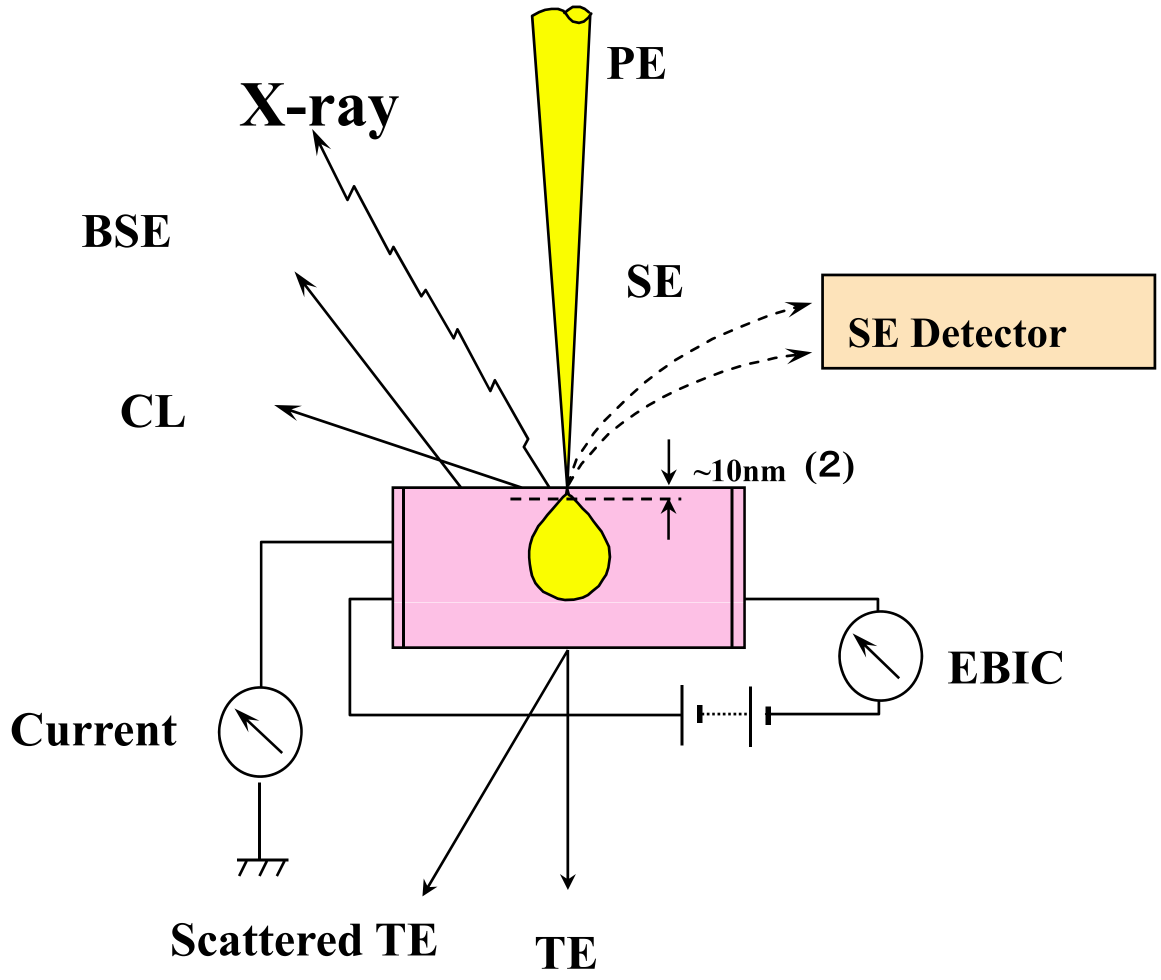


Advantages:

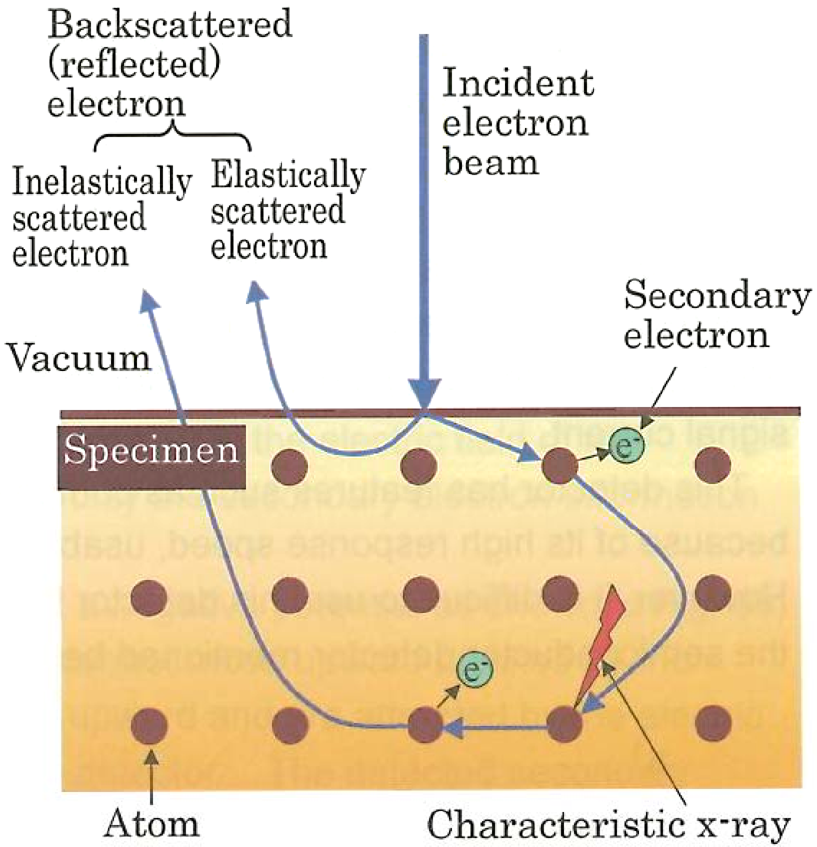
Superior imaging quality (signal-to-noise ratio, image contrast) for routine work at low beam energies

EDX analysis of thin layers at low beam energies is enabled.

Signals in the SEM



Emitted electrons



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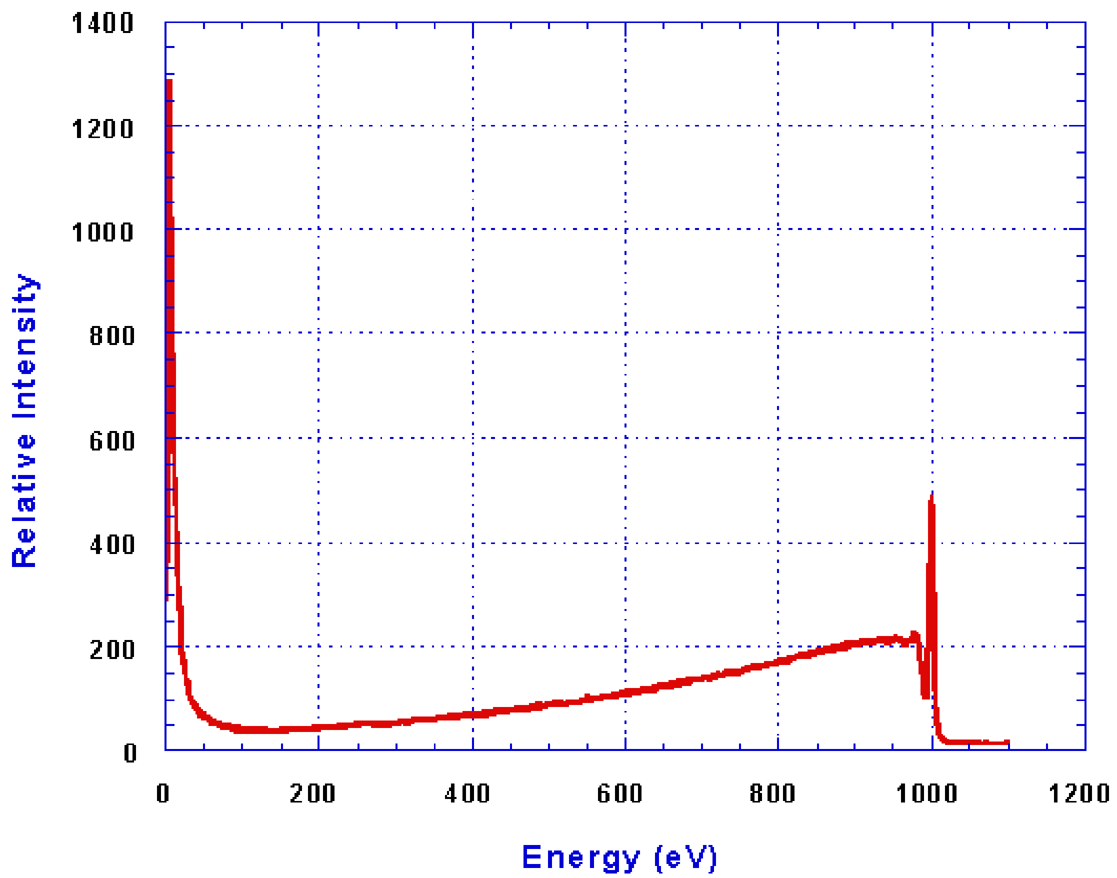
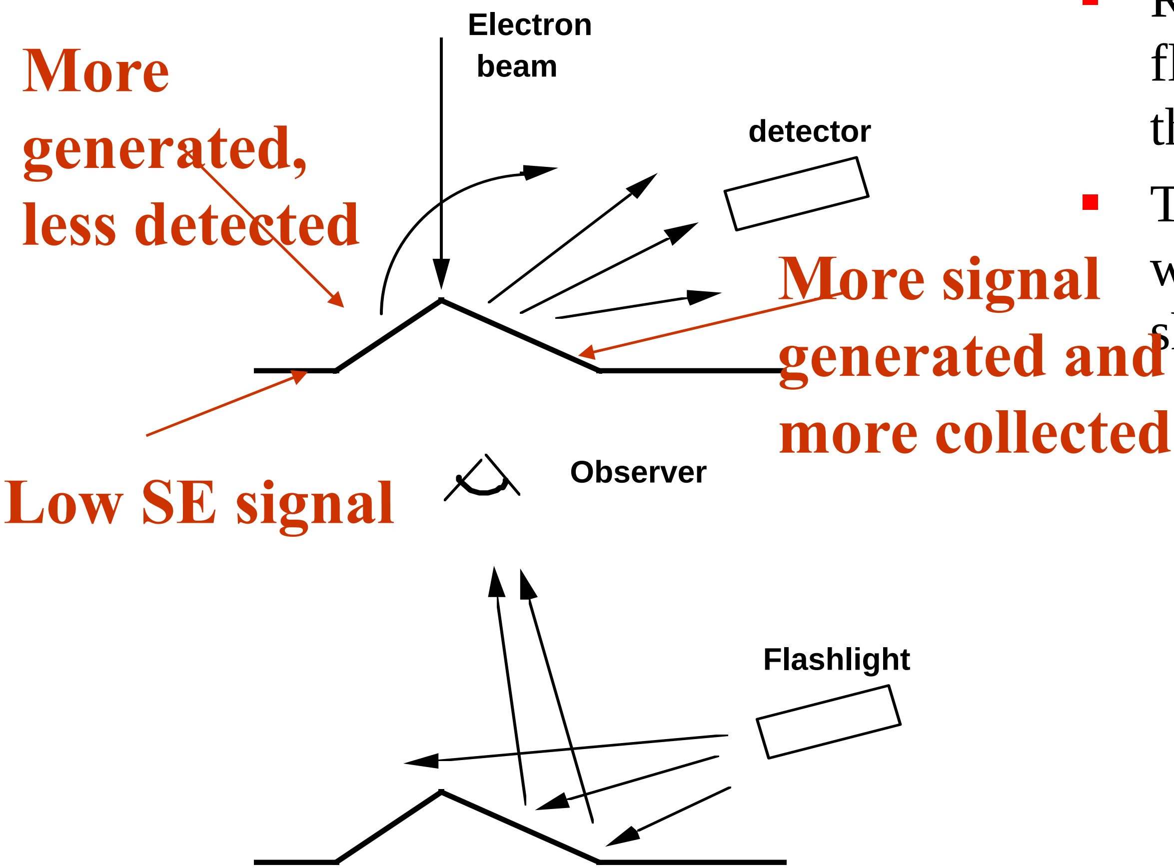
Emitted electrons

- When a sample is hit by an electron beam a variety of types of electron emission are available
  - Secondary electrons (energies 0 - 50eV)
  - Backscattered electrons (50eV to beam energy)
  - Elastically scattered electrons (i.e. those at the beam energy)



Energy spectrum from an InP wafer at 1.0keV incident beam energy

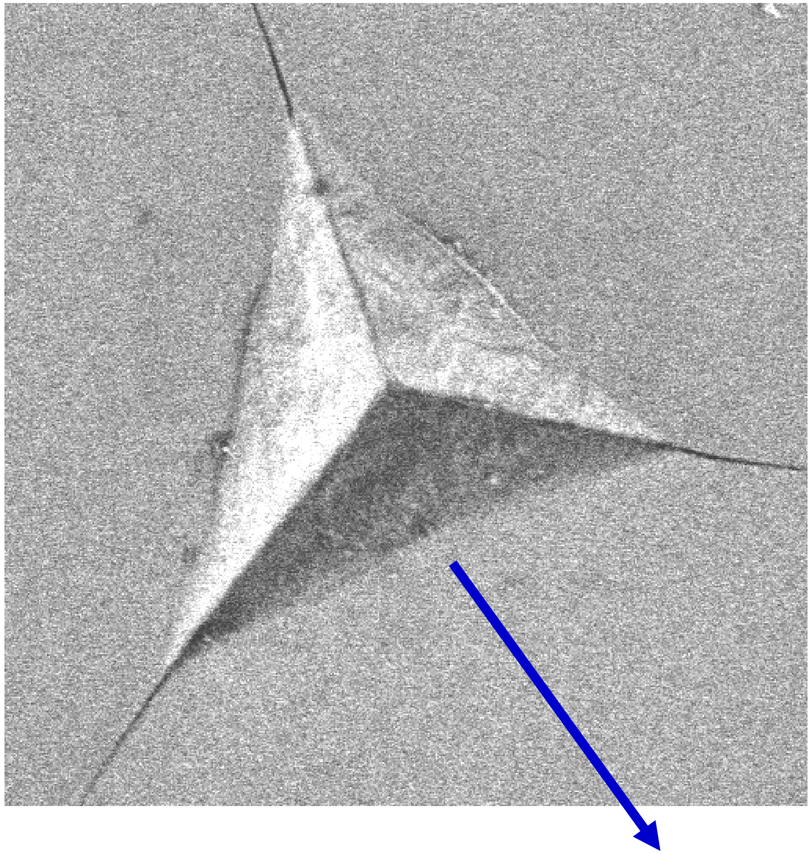
SE Contrast mechanisms



- Replace the detector by a flashlight, and imagine looking at the sample from the gun
- The SE image 'looks' like a real world image, light and dark, shadow and highlight
  - A detector on the sample horizon will give strong shadows
  - A detector above the sample gives no shadow information

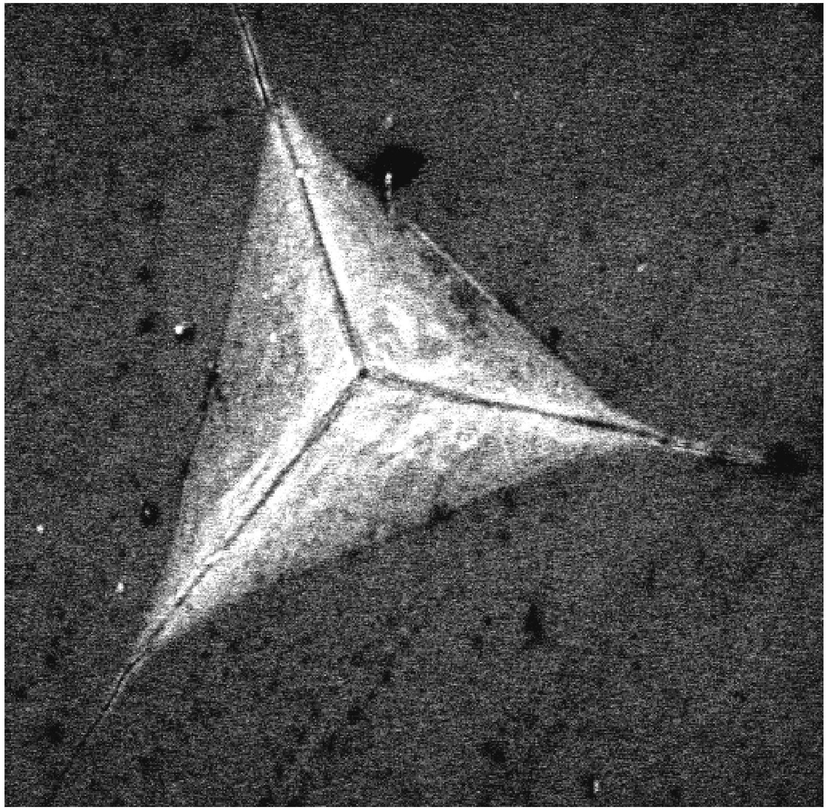
SE Contrast mechanisms

- The detector position therefore affects the image appearance
- The lower (ET) detector views the sample from one side and so the face looking away from the detector is shadowed
- Is this a pit or is it a pyramid?



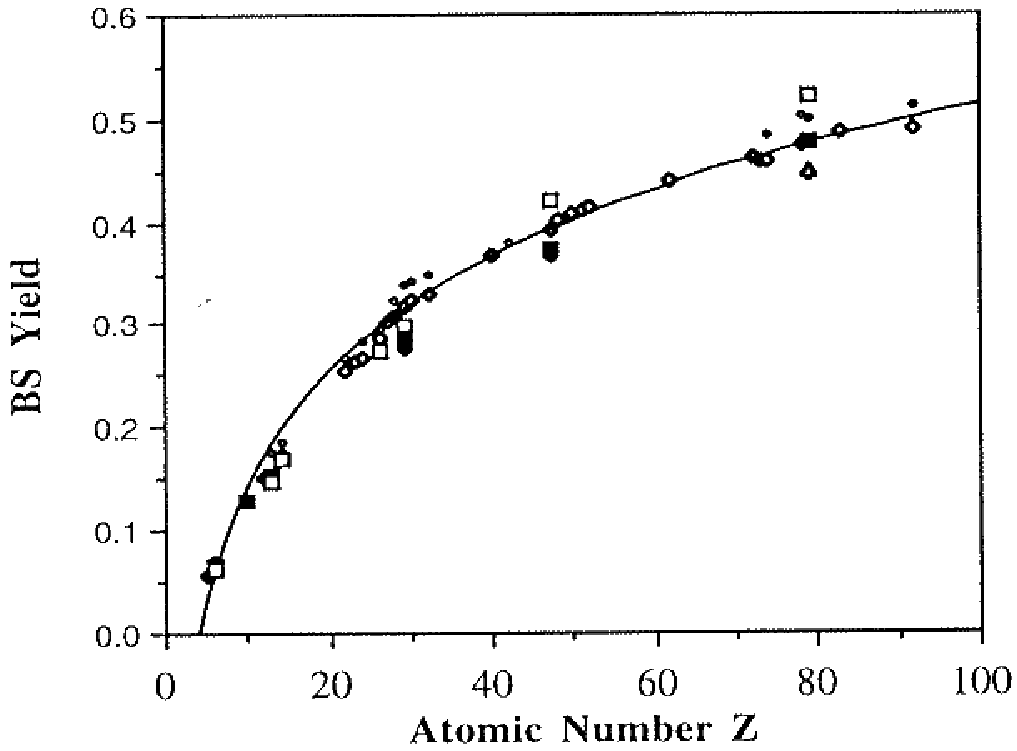
SE Contrast mechanisms

- The upper (in-lens) detector views the sample from above
- The SE collection is now symmetrical and so all faces of the indent are equally visible.
- They are brighter than the flat surface because of topographic contrast.



Backscattered electrons

- The Backscattered Electron yield is
  $\eta = N(BS) / N(\text{incident})$
- η increases with Z and incident angle
- η does not depend much on energy



Experimental BS yields

Interaction volume

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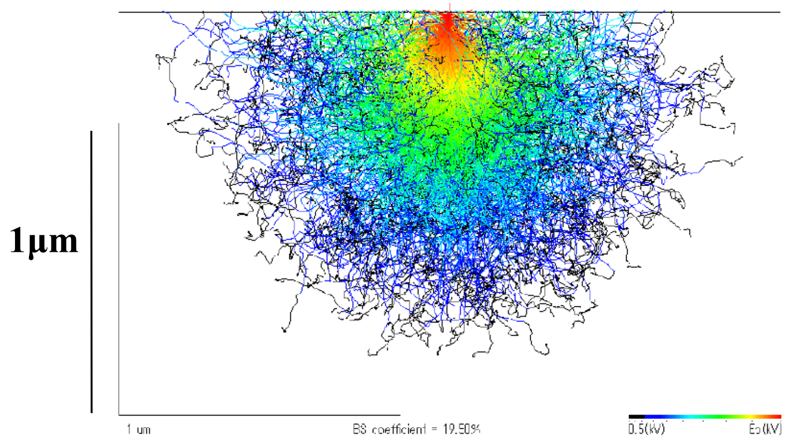
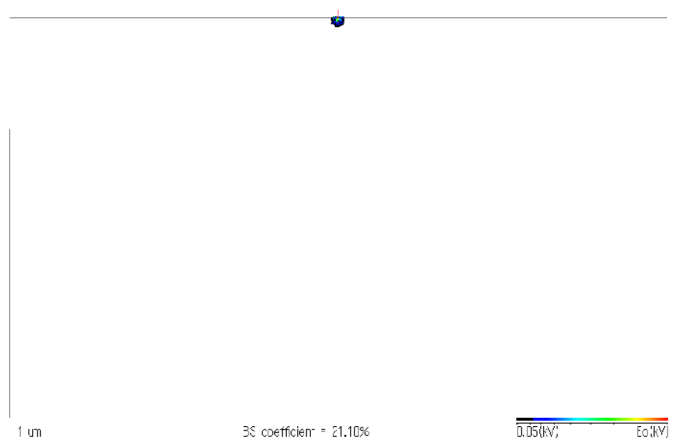
- Images are formed because of beam interaction with the sample
- This happens in a volume, not in a point
- The size of this volume varies with beam energy...

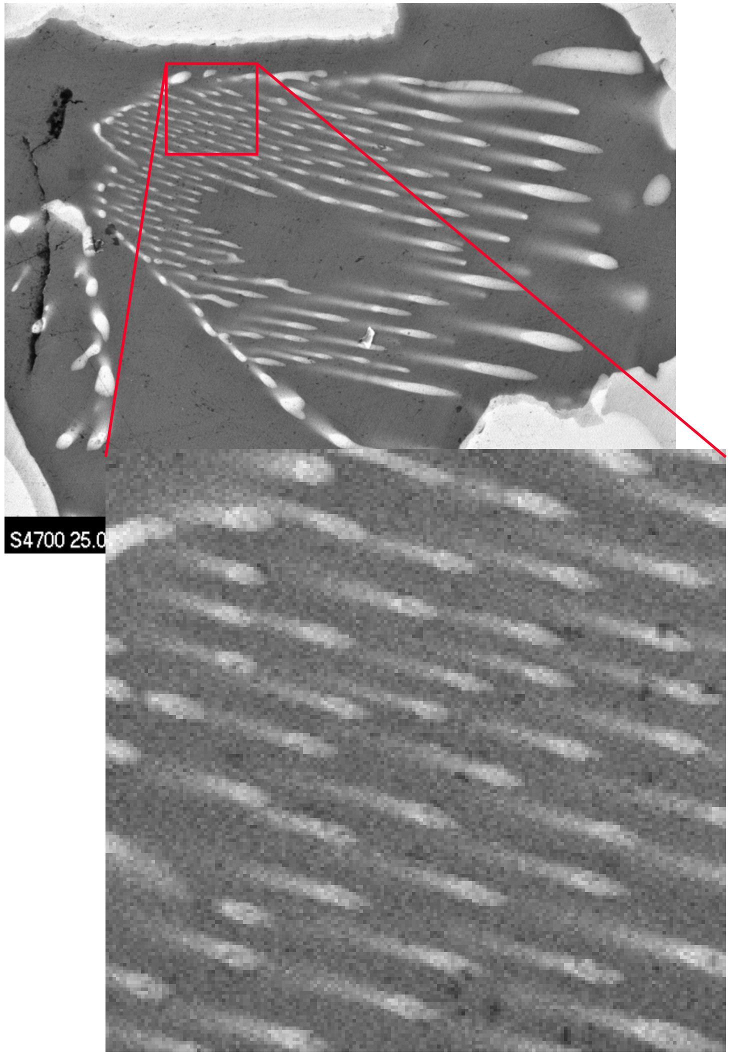


Vacc : 10kV

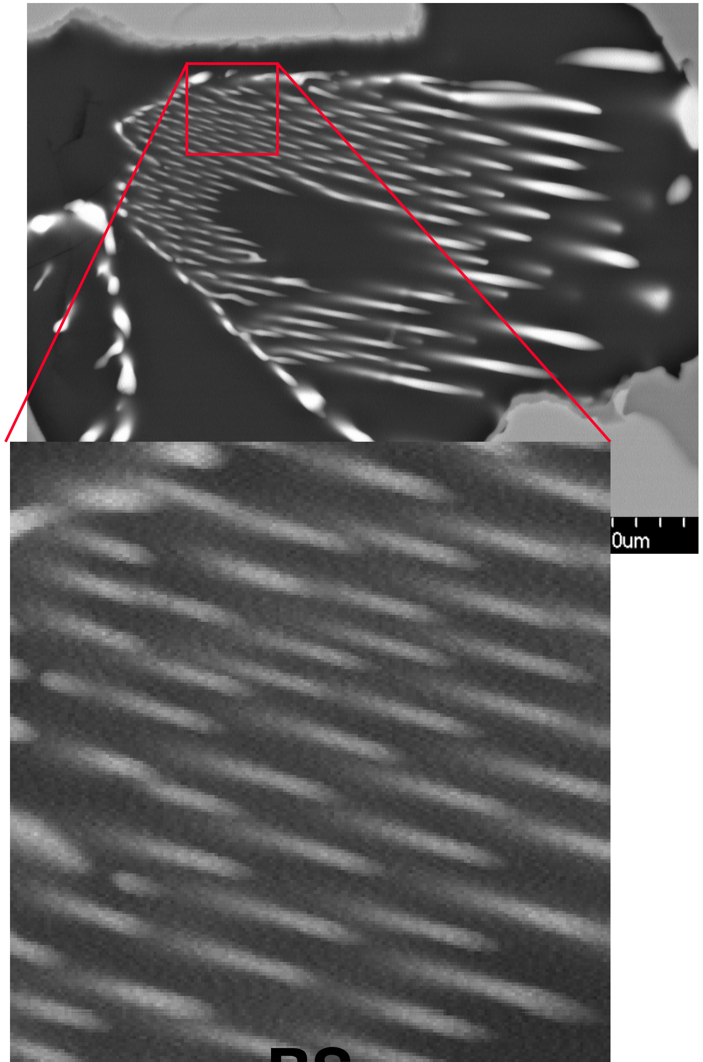


Vacc : 1kV

SE & BSE at 25 kV

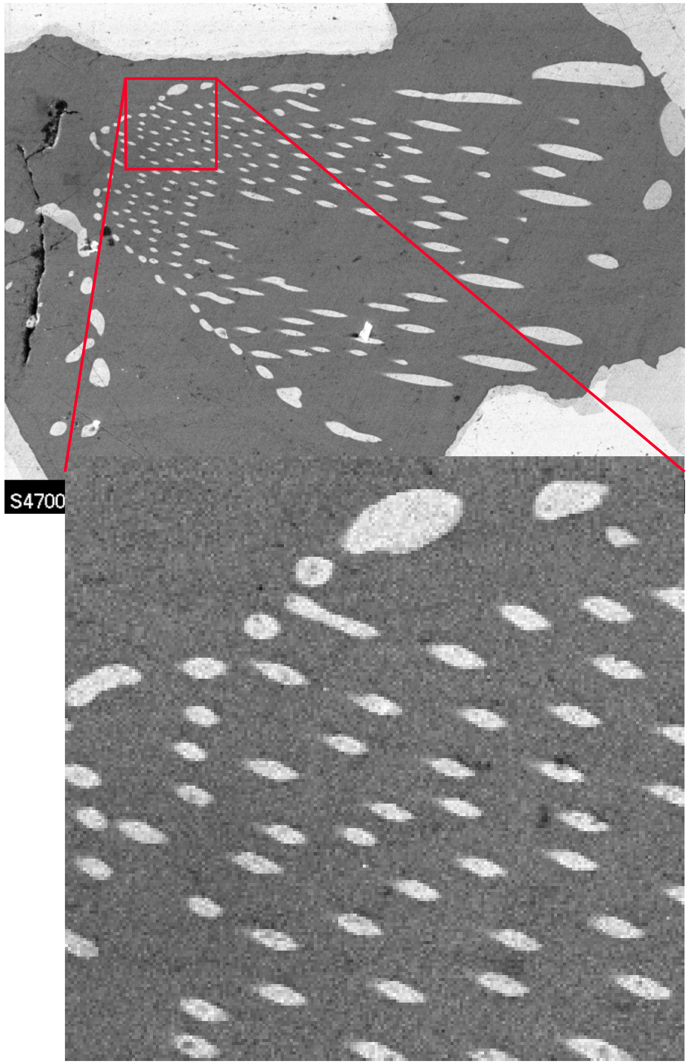


SE

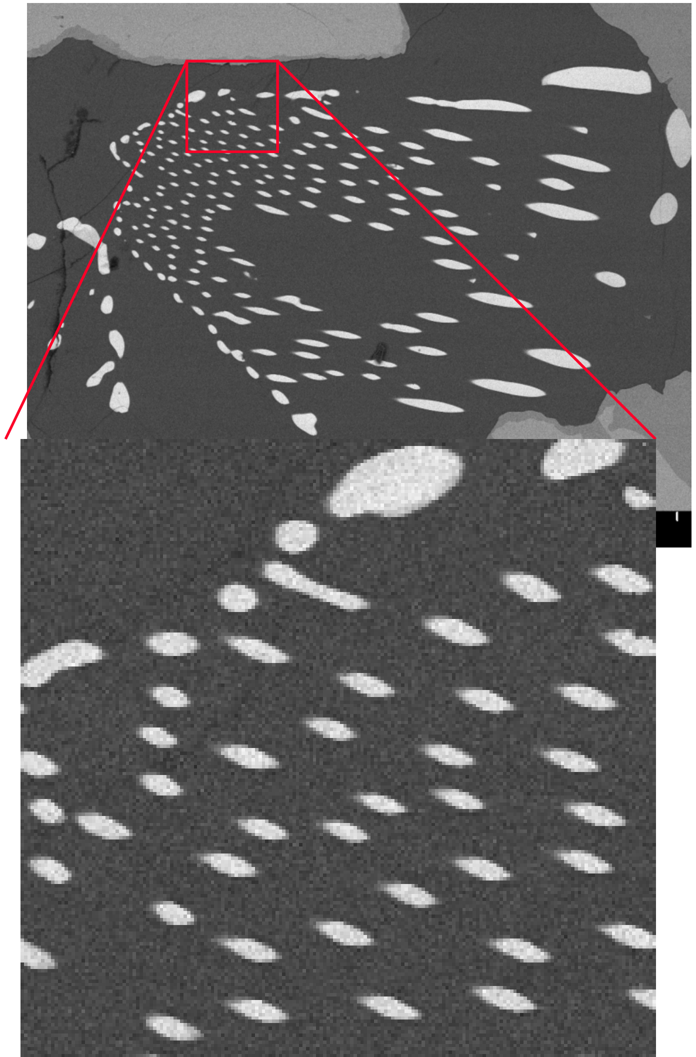


BS
E

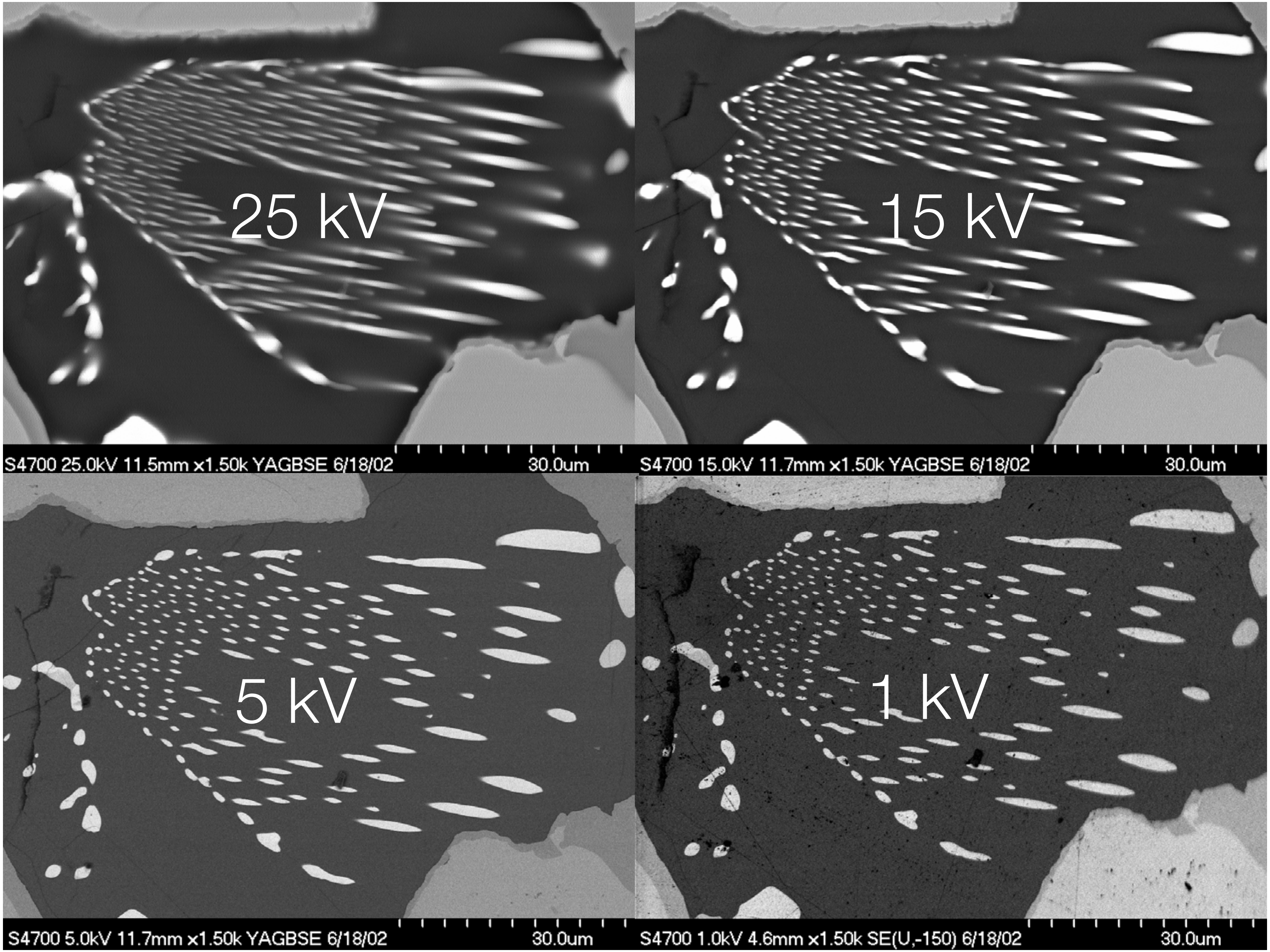
SE & BSE at 5 kV



SE



**BS
E**


25 kV
S4700 25.0kV 11.5mm x1.50k YAGBSE 6/18/02
30.0um
15 kV
S4700 15.0kV 11.7mm x1.50k YAGBSE 6/18/02
30.0um
5 kV
S4700 5.0kV 11.7mm x1.50k YAGBSE 6/18/02
30.0um
1 kV
S4700 1.0kV 4.6mm x1.50k SE(U,-150) 6/18/02
30.0um

Why to use Low Beam Energies?

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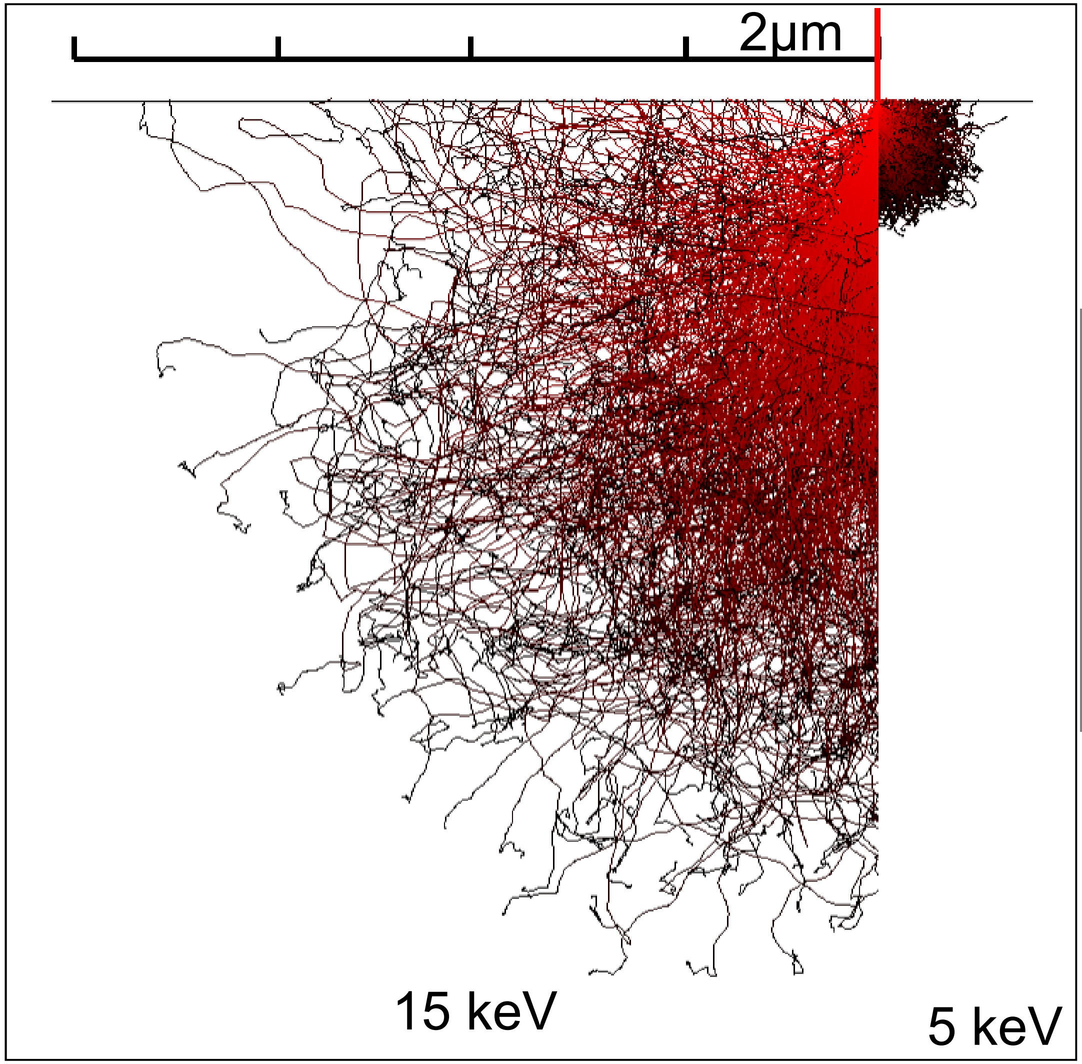
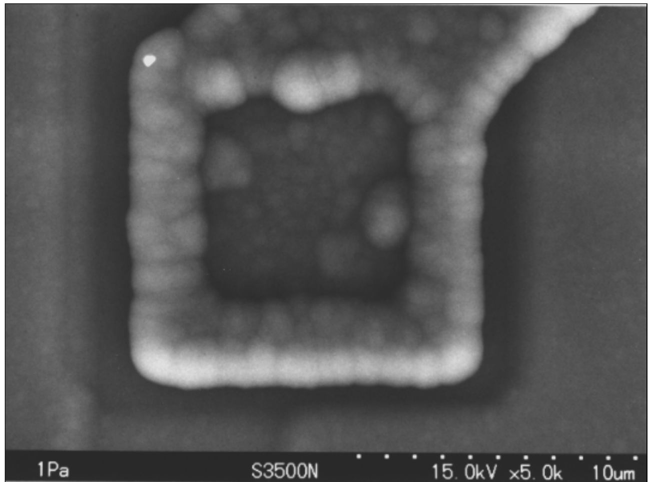
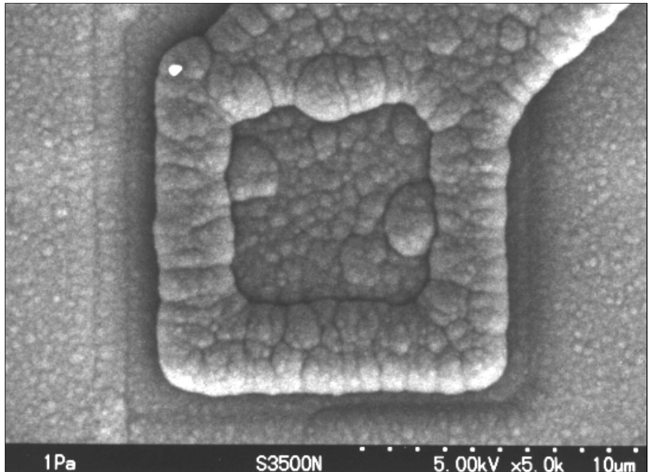


Sample: Aluminium



High kV: Loss of top surface details



Low kV: High detail resolution

BSE Detectors

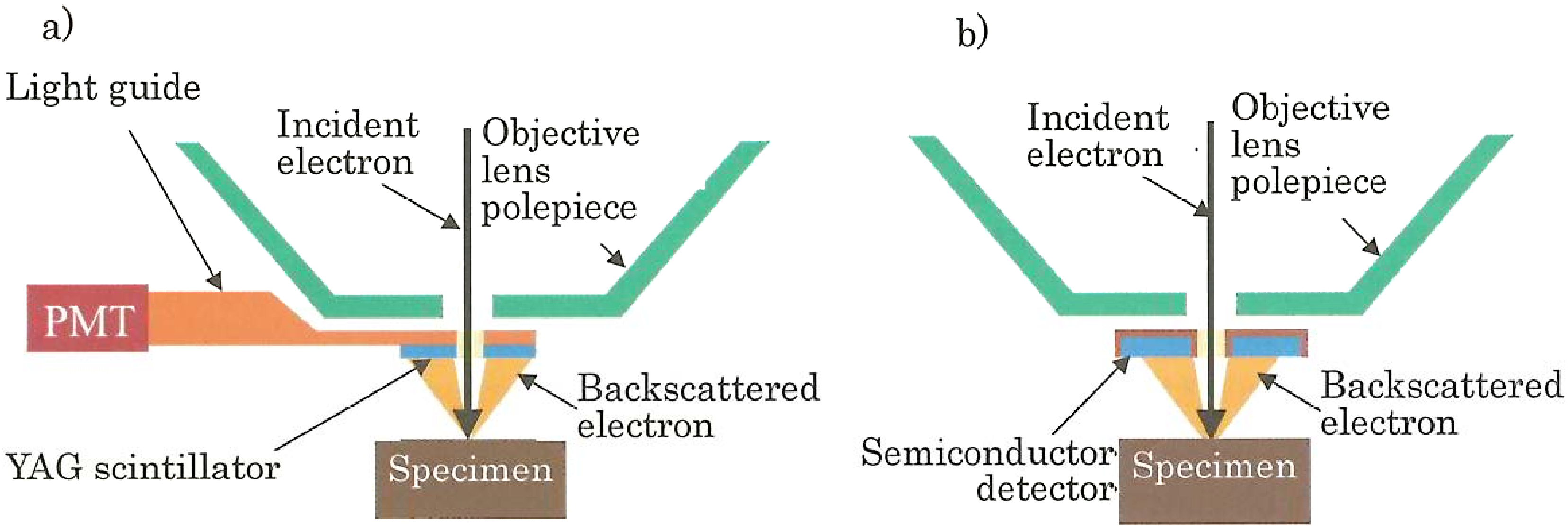


Fig. 6 Composition of YAG Detector a) and Semiconductor Detector b)

Available image signals

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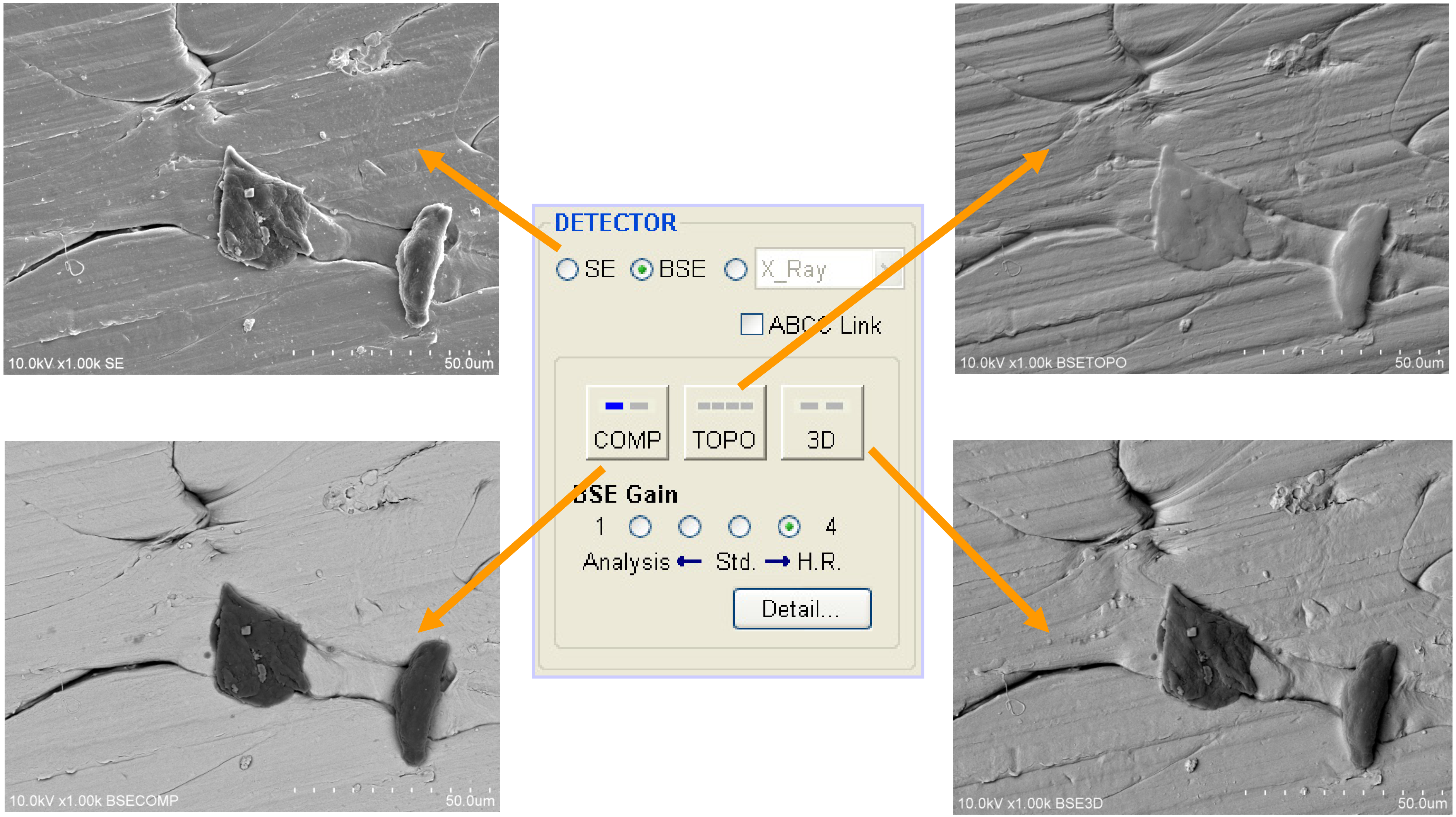
SE **BSE TOPO**



BSE COMPO **BSE 3D**

Material Contrast and More:
High Sensitivity 4+1 BSE Detector

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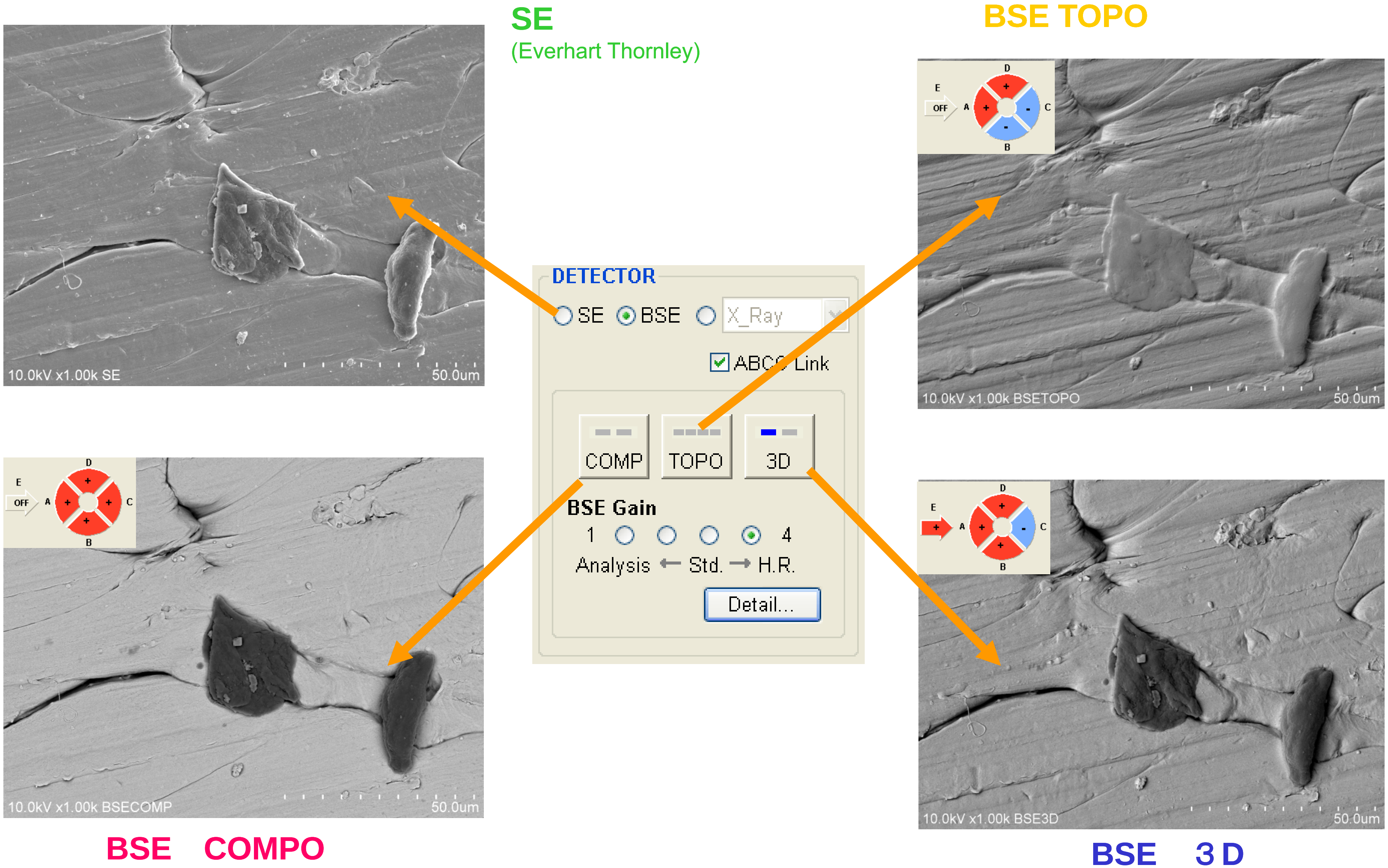


Imaging problems

Phenomenon

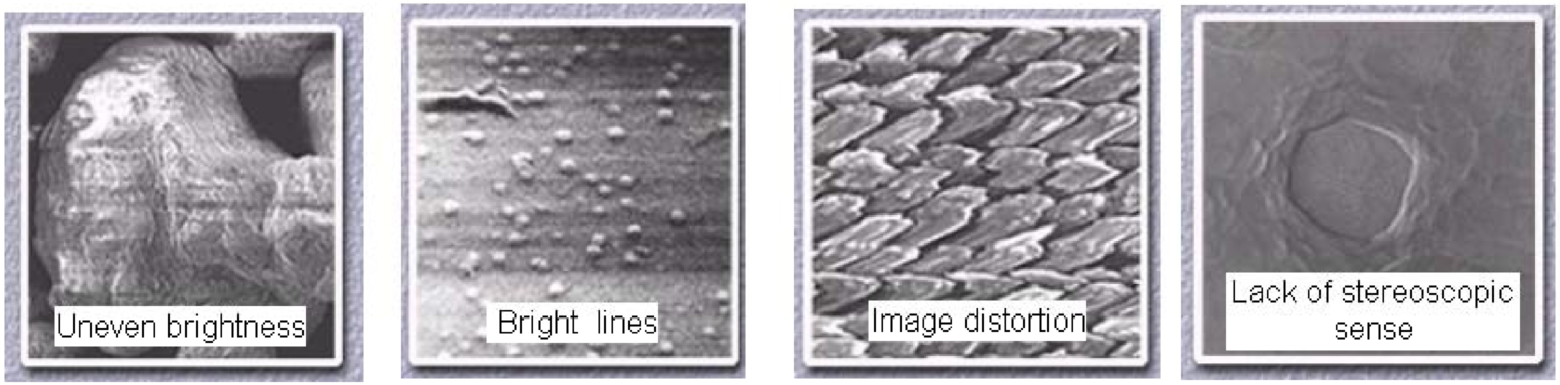
- Image moves
  A C D
- Sample structure cannot be discerned
  A B C
- Image is distorted
  A C D E
- Image fluctuates
  A E
- Brightness is unstable
  A B E
- Cannot be focused
  A B C E

Probable cause

- A Charge-up
- B Contamination
- C Beam damage
- D Effect of external disturbance
- E Other

*Causes and countermeasures are described on the following pages.

Charge up

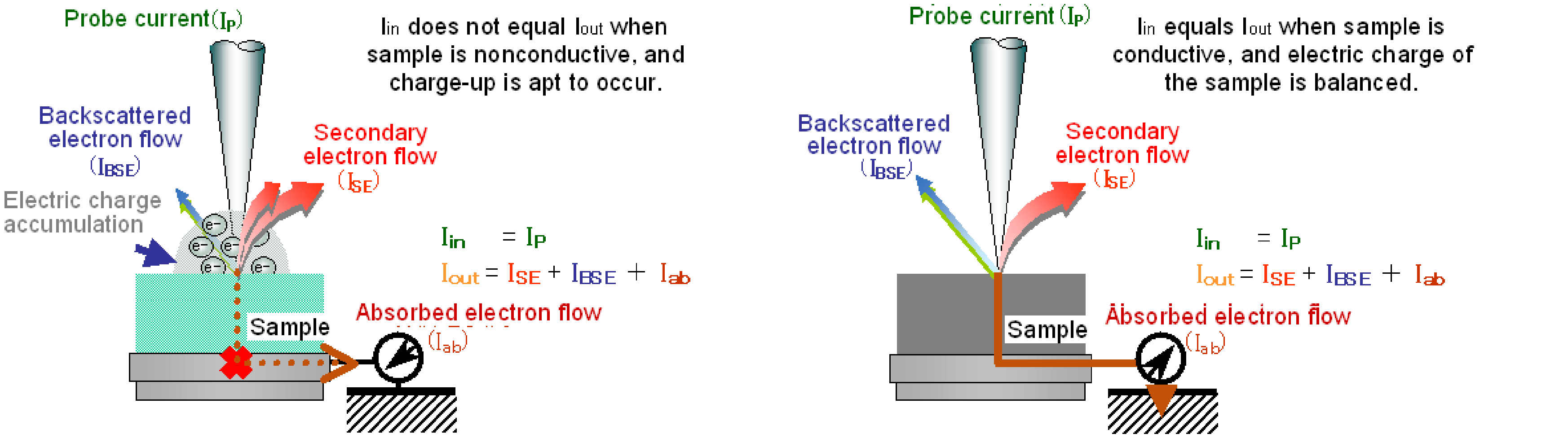


Examples of Charge-up Phenomenon

Charge-up occurs during observation of non-conductive samples, and may be conspicuous especially when scan speed or magnification is changed.

Charge up

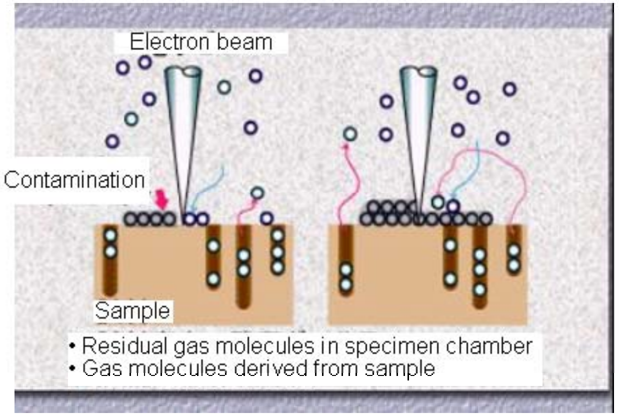
Spectral Solutions

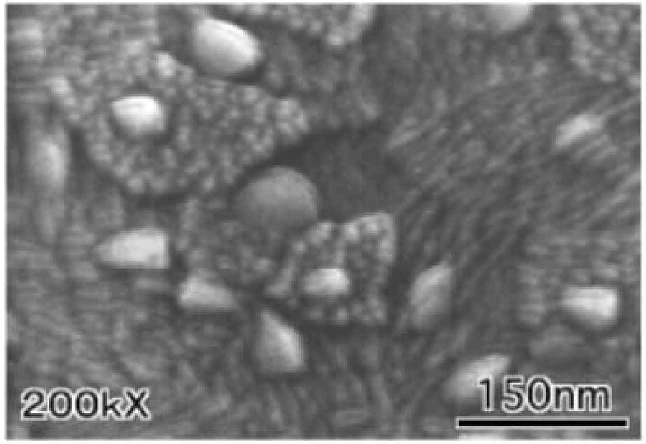


Countermeasures for the charge-up phenomenon.

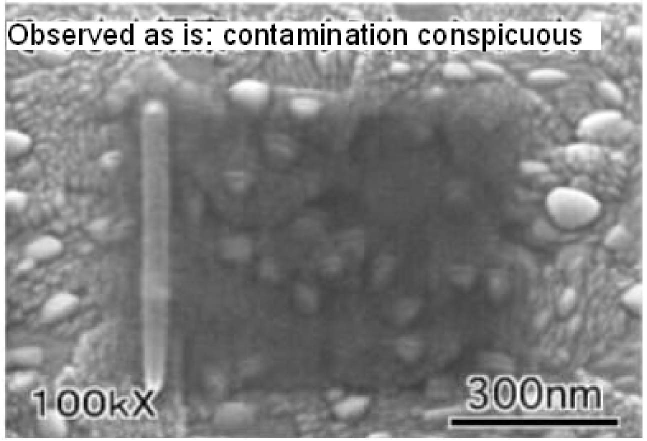
1. Reduce the accelerating voltage.
2. Reduce the sample irradiating current.
3. Apply a metal coating.
4. Integrate the image (form an image by superimposing images obtained at rapid scan)
5. Observe images in low vacuum mode
6. Utilize a low-acceleration BSE signal (eliminate SE signal by means of signal varying mechanism)

Contamination





Magnification reduced



Example of Sample Contamination

Hitachi High-Technologies Europe GmbH

Contamination

Countermeasures against contamination

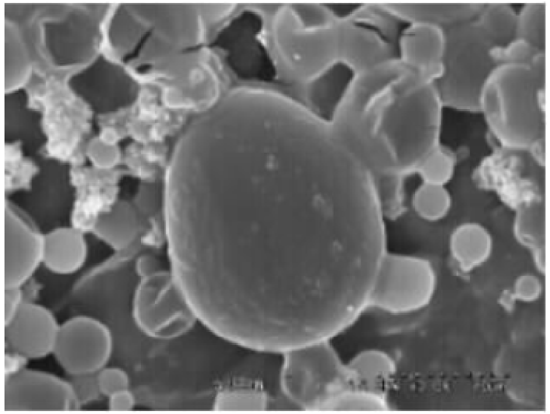
The following steps are required in order to reduce the contamination:

- Reduction of residual gas molecules in specimen chamber (improvement of vacuum level)
- Reduction of gas molecules derived from sample

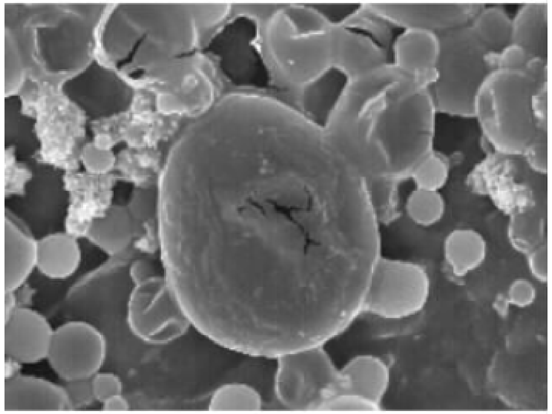
Concrete measures to achieve the above reductions are as follows.

1. Use a minimum amount of conductive paste or tape when mounting the sample in the instrument.
2. Thoroughly dry the conductive paste with a dryer or the like prior to inserting sample into the instrument for observation.
3. Heat and degas the sample in a vacuum device.
4. Carry out focusing as quickly as possible and avoid observing the same location for a long time especially at high magnification.
5. Observe samples while cooling the sample surroundings with a cold trap.

Beam damage



No beam damage



Damage due to beam irradiation

Countermeasures against contamination

1. Reduce the sample irradiating current
2. Lower the accelerating voltage
3. Apply (metal) coating to the sample (to imrove heat conductivity)
4. Observe the sample while cooling it

Outside disturbance

Countermeasures against vibration

1. Keep the instrument well away from vibration sources such as air-conditioner or pumps.
2. Do not let high-voltage cables from the column come in contact with the wall or other installation items Lower the accelerating voltage
3. Don’t let the draft from an air-conditioner outlet contact the column directly.

Countermeasures against magnetic field

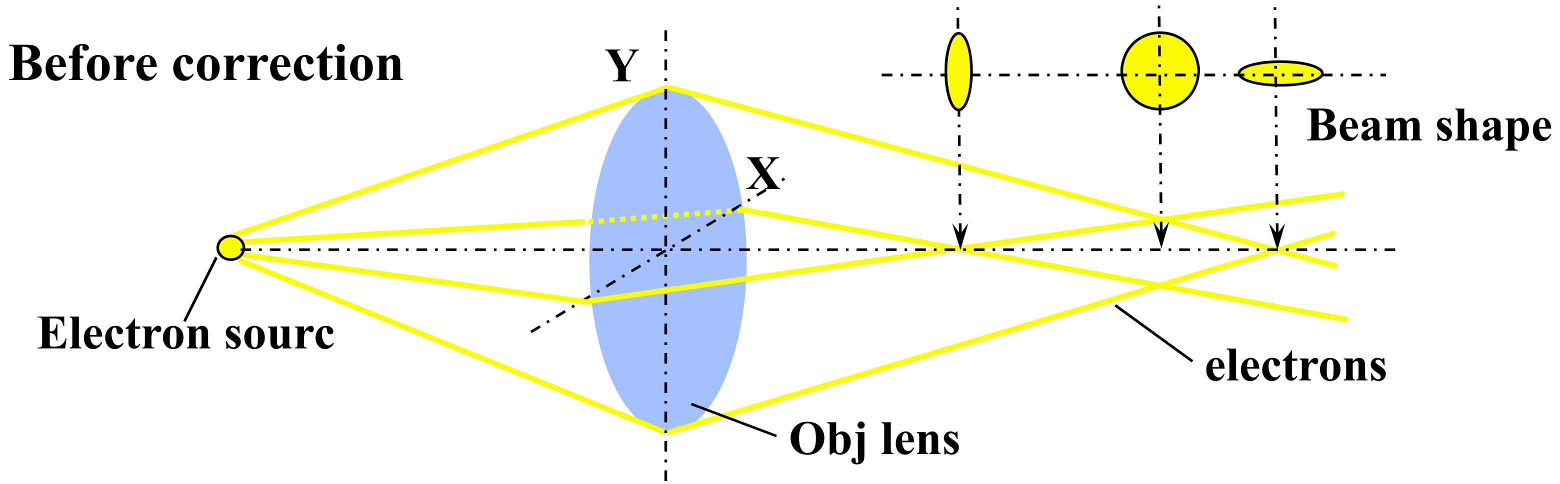
1. Keep the instrument well away from magnetic field sources such as transformer or large capacity power cables educe the sample irradiating current
2. Lower Shorten the working distance (see 1-7 or 4-5 in Chapter 6) and apply strong excitation to the condenser lens to counter the effect of a magnetic field.
3. Use a magnetic field cancelleing system

Other problems

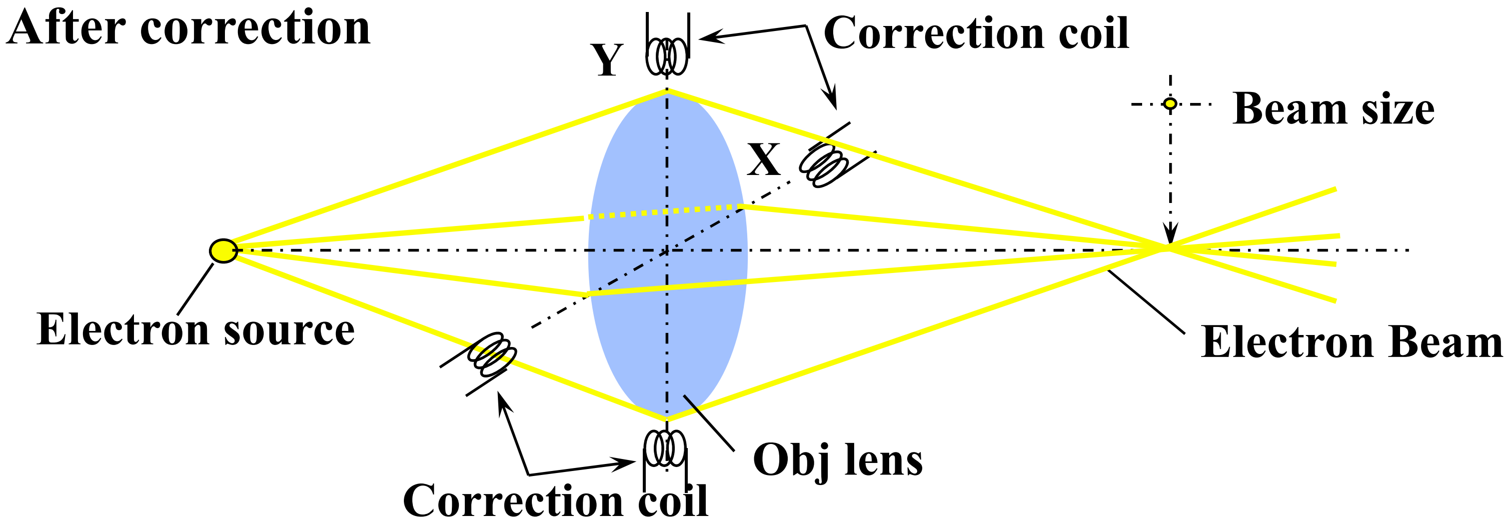
Spectral Solutions

Symptom	Possible causes
Sample moves	• Sample is not fixed in place adequately when sampling. • Screw of specimen holder is not tightened adequately. • Sample is inserted incompletely onto specimen stage. • Compressor operated while the stage is locked.
Image fluctuates	• Irradiating current is low (change the excitation of condenser lens). • Lower image is being observed at short WD in the case of semi in-lens SEM. • WD is long in low vacuum SEM observation mode.
Focus cannot be obtained	• Inadequate optical axis alignment • Objective aperture contaminated • Recheck the instrument parameters.

Astigmatism



Astigmatism



Astigmatism

Spectral Solutions

Before correction

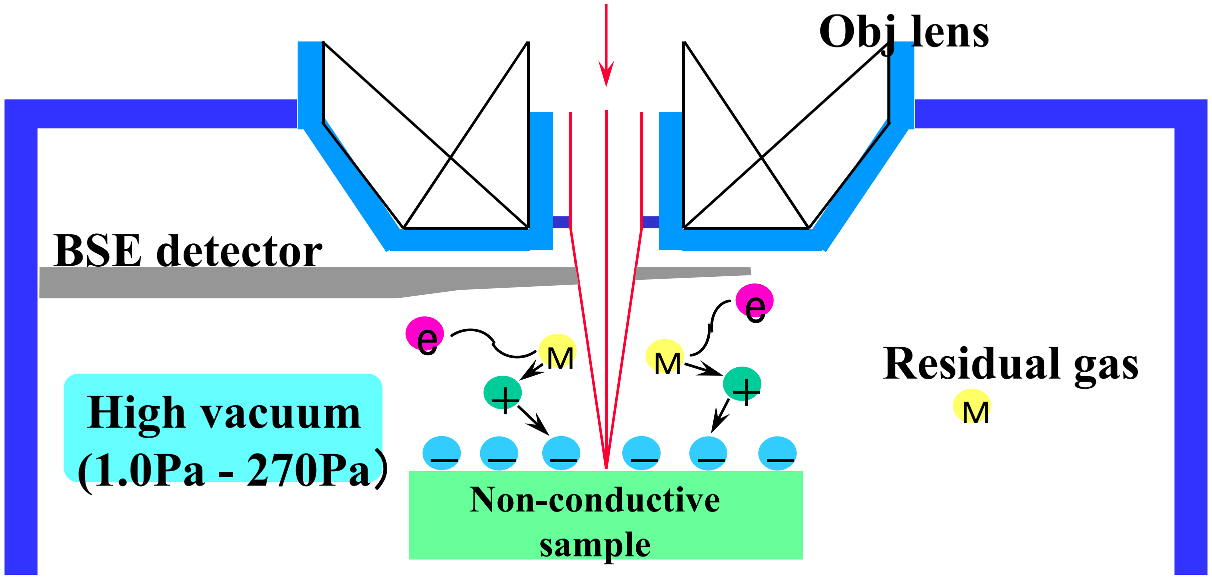
Specimen:Trachea of rat

Astigmatism

After correction

Specimen:Trachea of rat

Variable Pressure

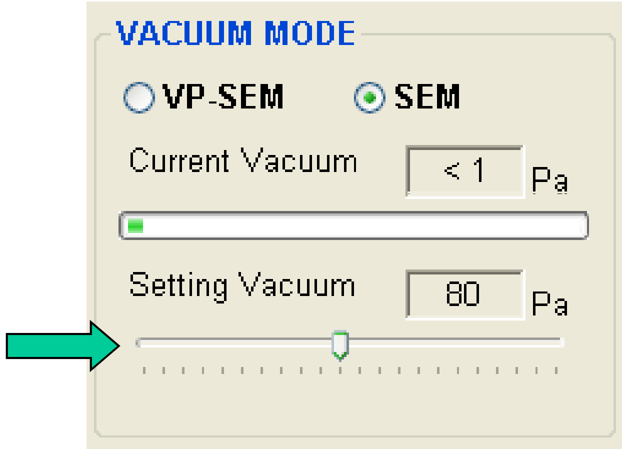


Pressure	Mean Free Path
10^{-3}Pa	40mm
13Pa	3mm
270Pa	0.1mm

SEM with Variable Chamber Pressure

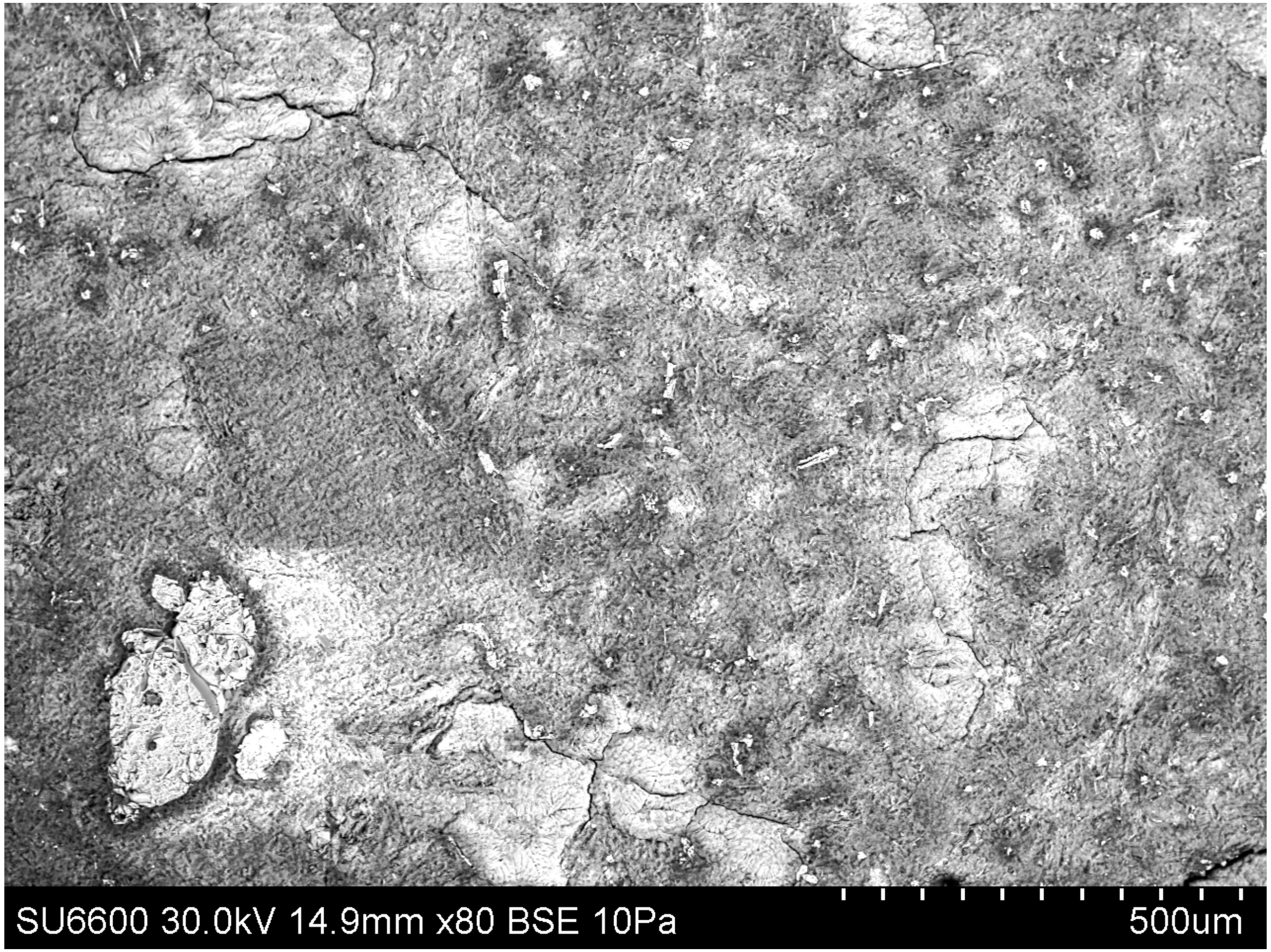
- Conductive samples: Observation in high-vacuum

- Non-conductive samples: Observation in low-vacuum
  - No sample coating required.
  - If charging occurs, simply the chamber pressure is gradually increased until the charging disappears.



- Investigation of humid or oily samples
  - An optional cool stage chills at ca. 60Pa chamber pressure the sample to –20°C, so that the evaporation of water is mostly suppressed
    (balance point between solid and gasous phase)

Variable Pressure

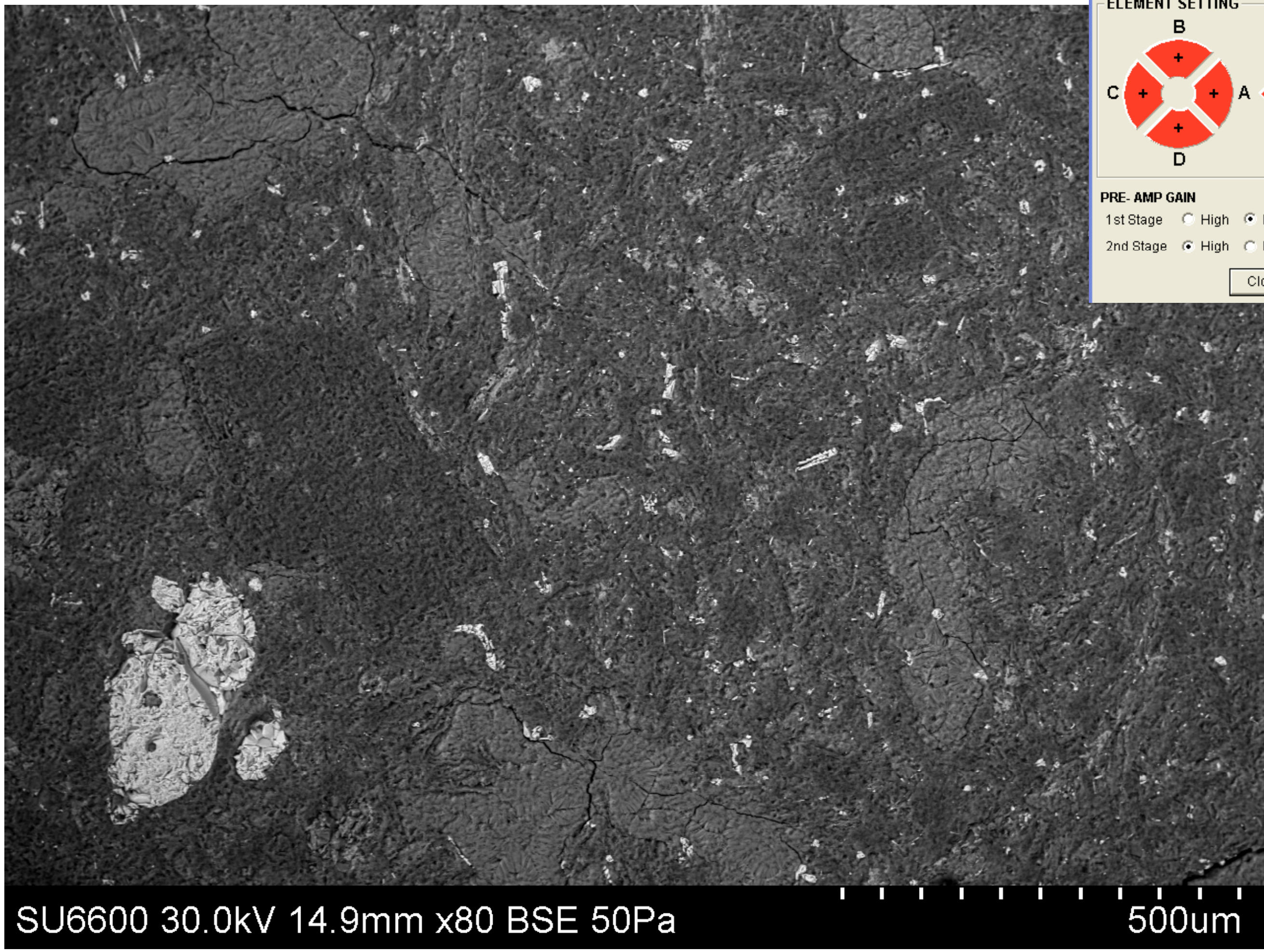


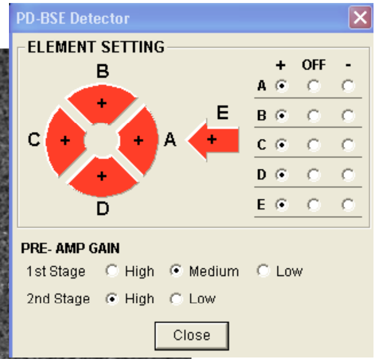
Basalt - uncoated

Hitachi High-Technologies Europe GmbH

Variable Pressure

Spectral Solutions



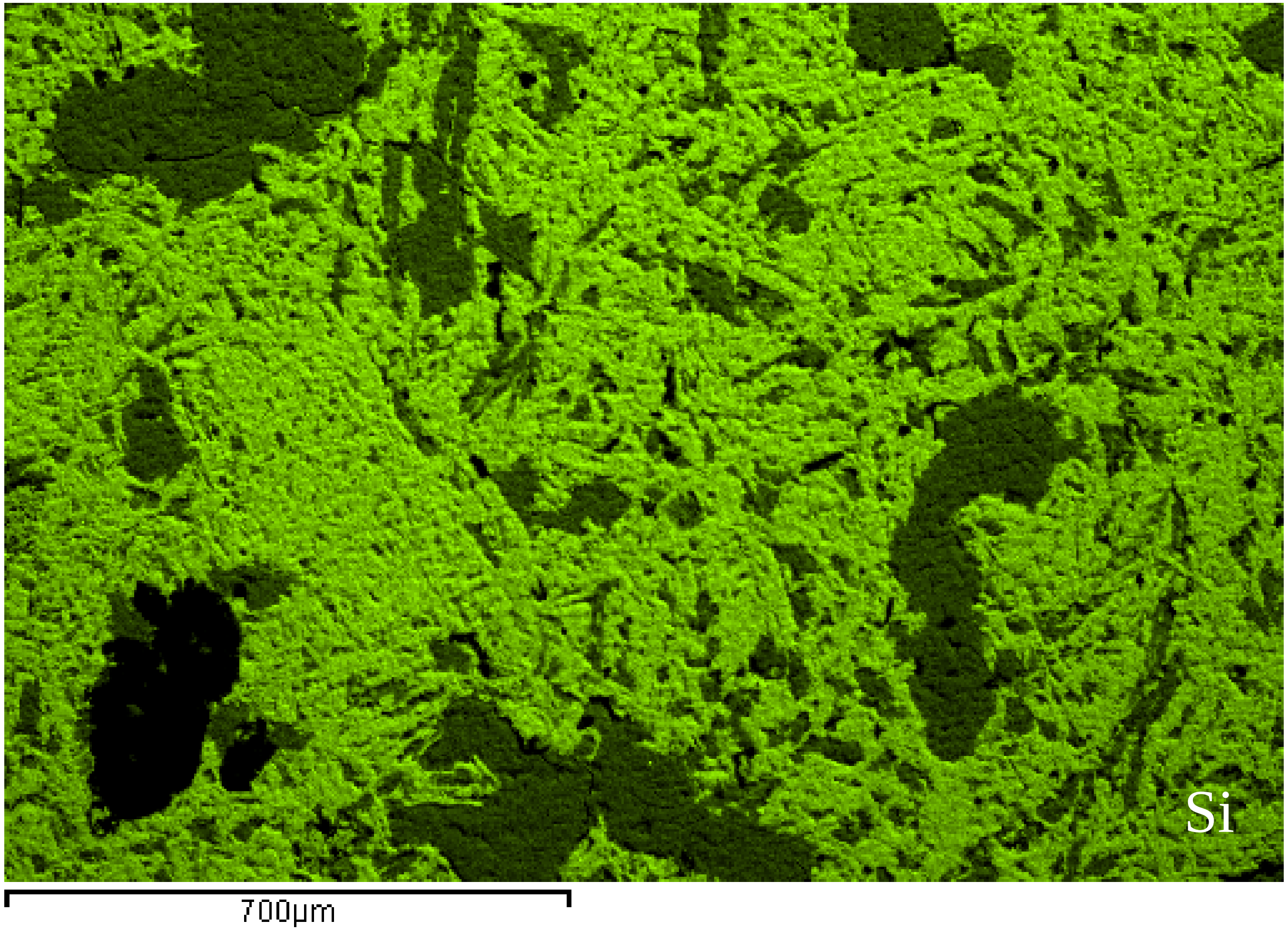


Basalt - uncoated

Hitachi High-Technologies Europe GmbH

X-ray mapping

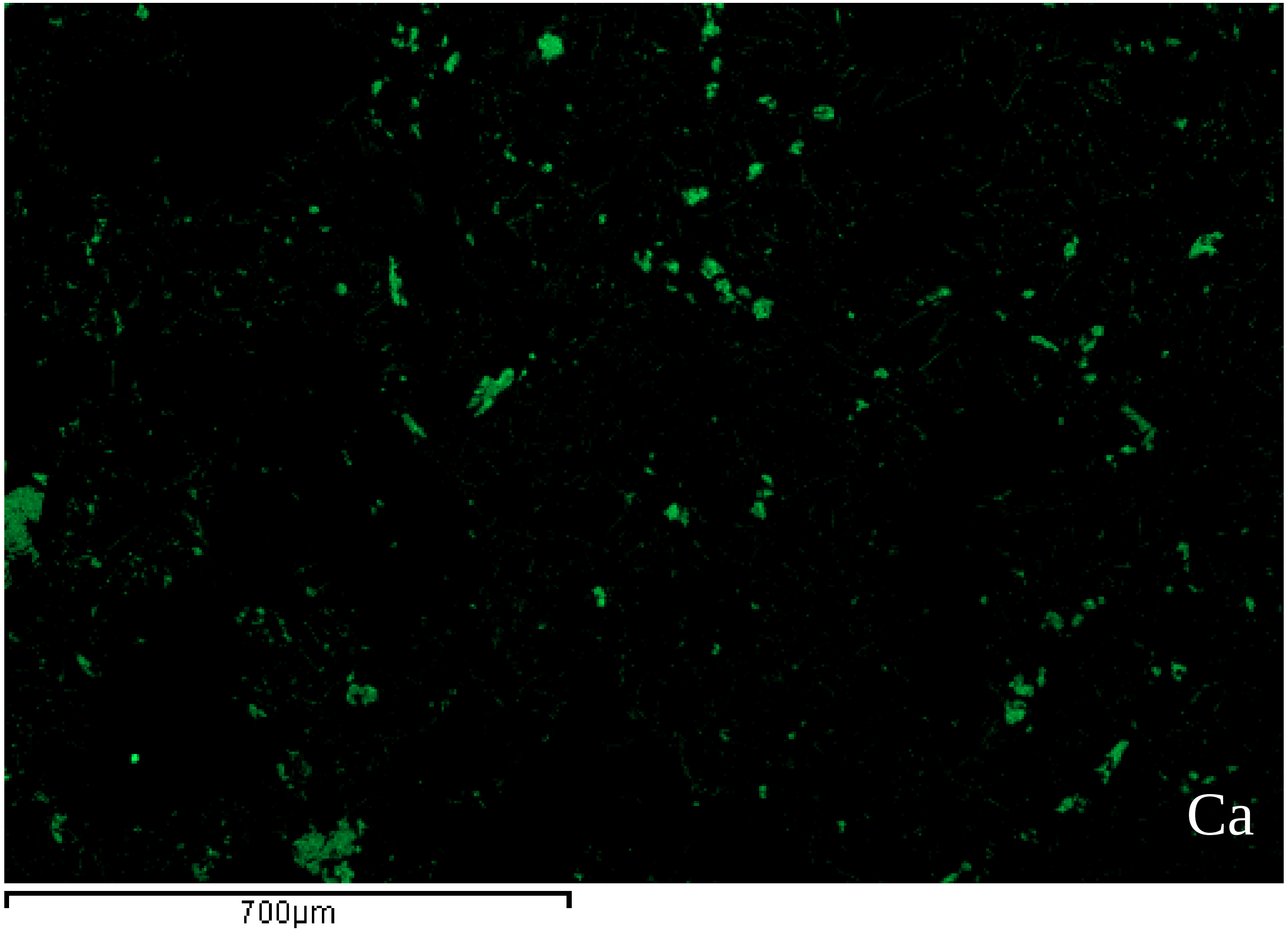
Spectral Solutions



77850cps, 38min., VP=50Pa, 60nA

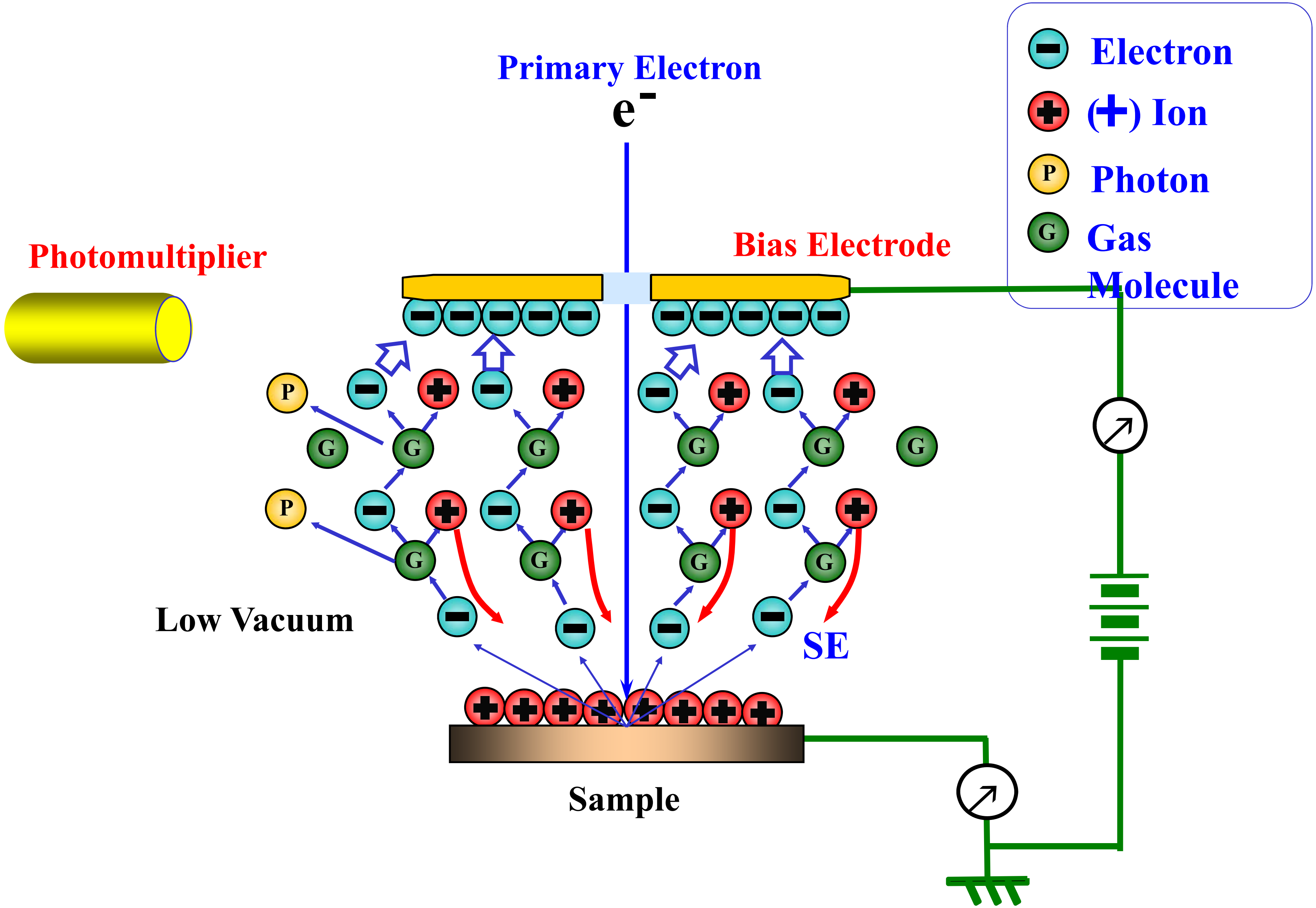
X-ray mapping

Spectral Solutions



77850cps, 38min., VP=50Pa, 60nA

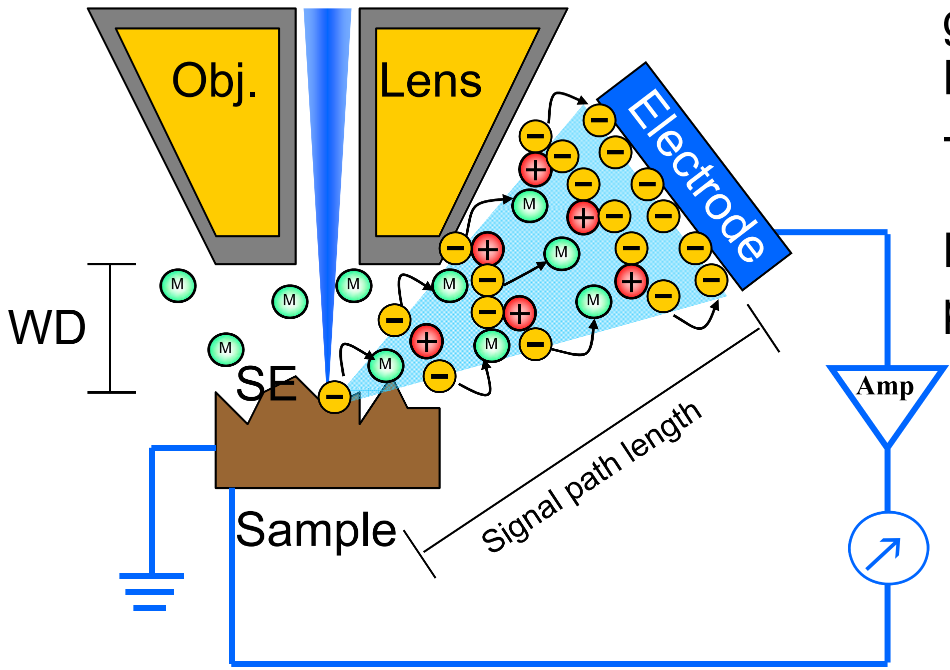
Variable Pressure SEM



SE Imaging at Low Chamber Vacuum

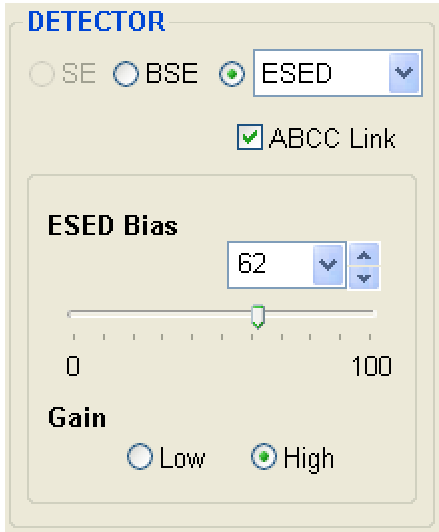
Spectral Solutions

<u>Application:</u> Operation under low chamber vacuum.
High surface detail resolution especially for light elements



Secondary electrons (SE) ionize gas molecules above the sample surface.
Electric collection field accelerates SE
-> further ionization, avalanche effect.

Recordable current per image pixel is proportional to the number of created SE.



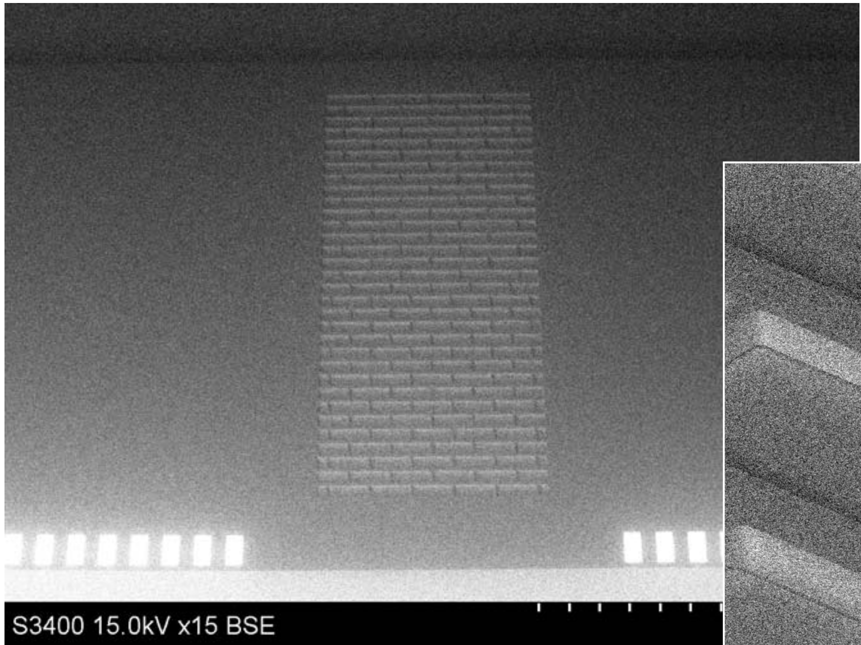
Working distance WD < signal path length:
- high beam quality, less scattering at gas molecules
- large signal path length for efficient signal amplification

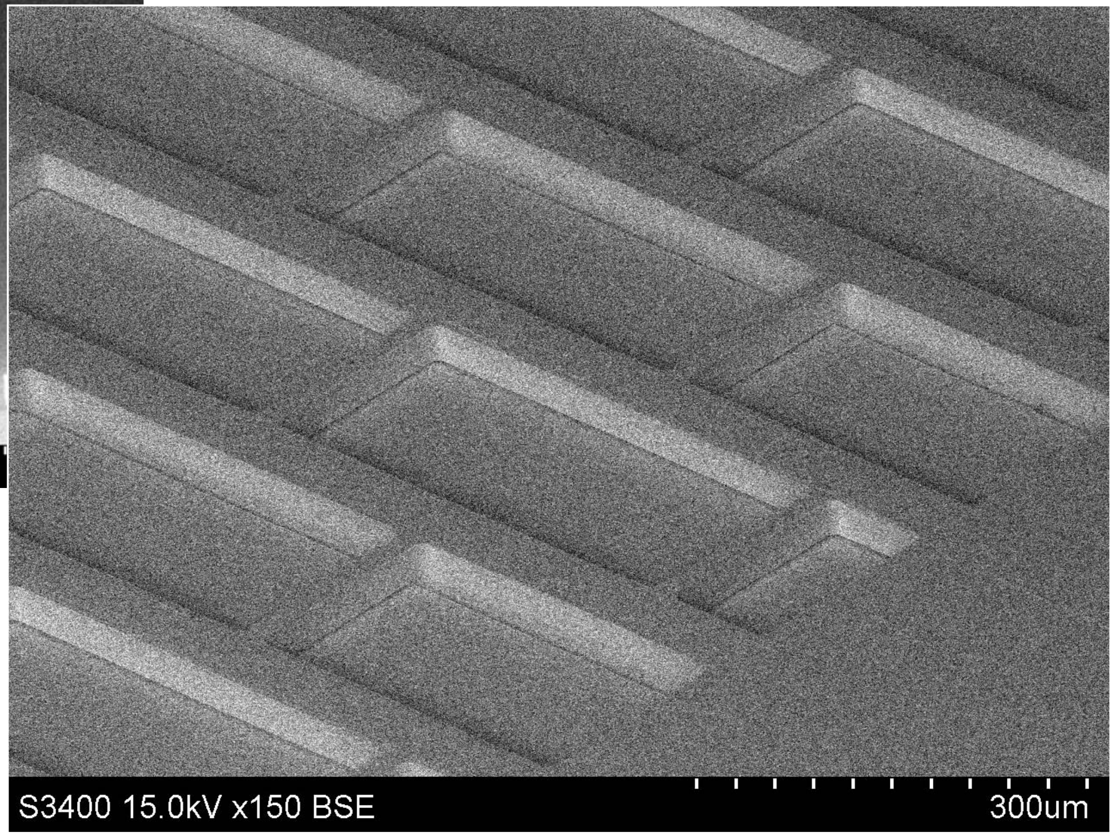
SE Imaging at Low Chamber Vacuum

Spectral Solutions

Sample : Quartz (low atomic numbers)

BSE image

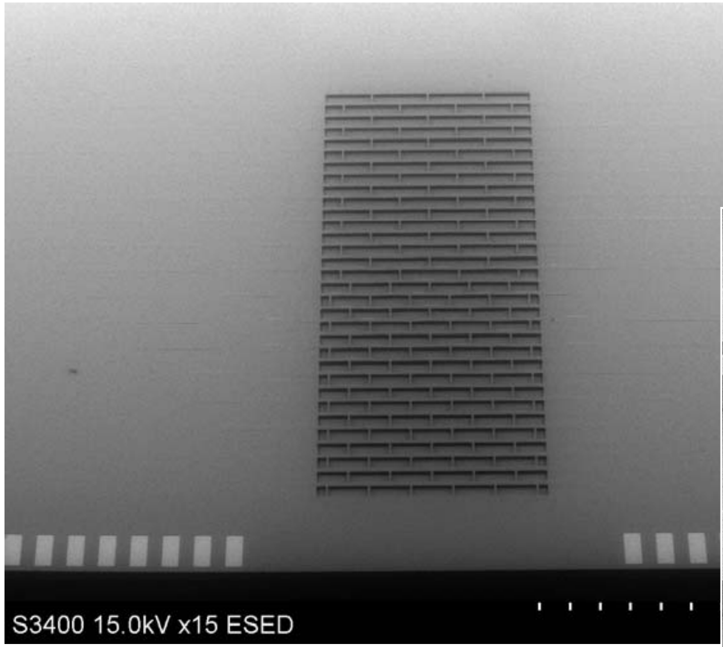




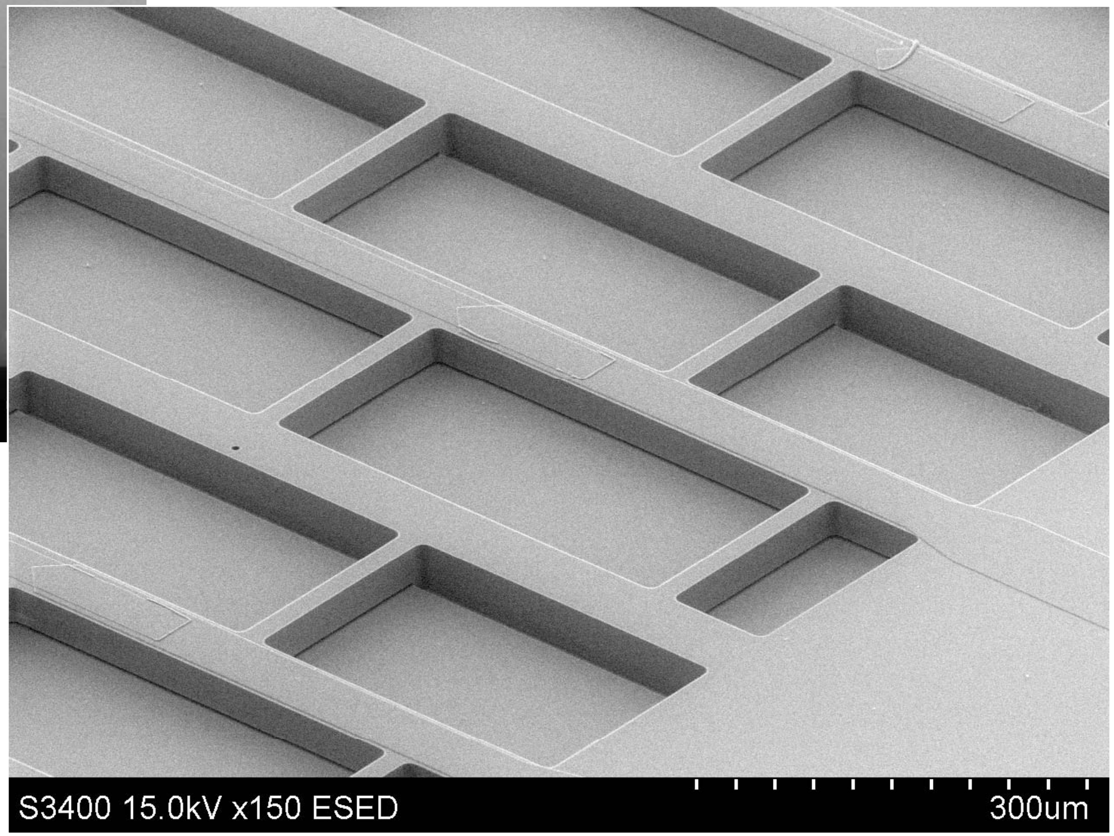
Chamber pressure: 60Pa
Vacc: 15kV

SE Imaging at Low Chamber Vacuum

Sample : Quartz (low atomic numbers)



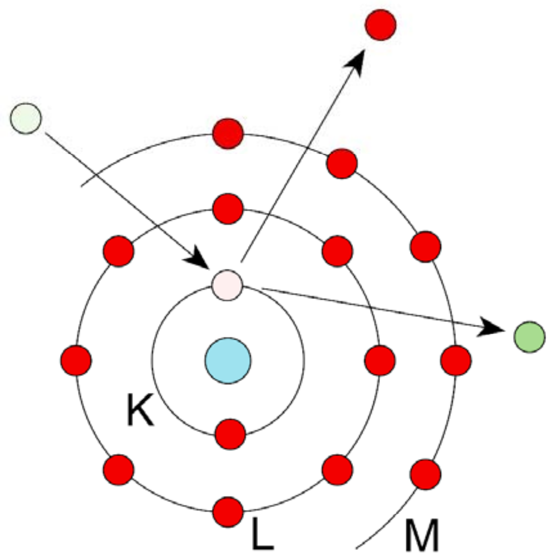
ESED image:
Many surface details
Good S/N ratio

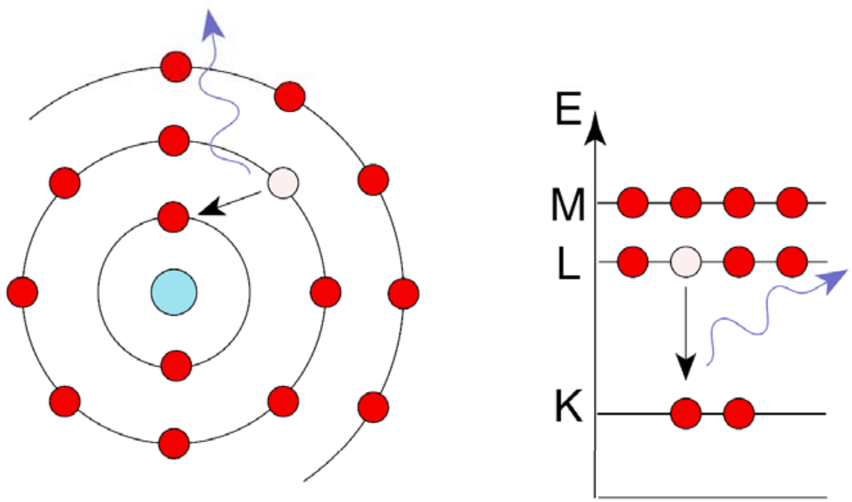


Chamber pressure: 60Pa
Vacc: 15kV

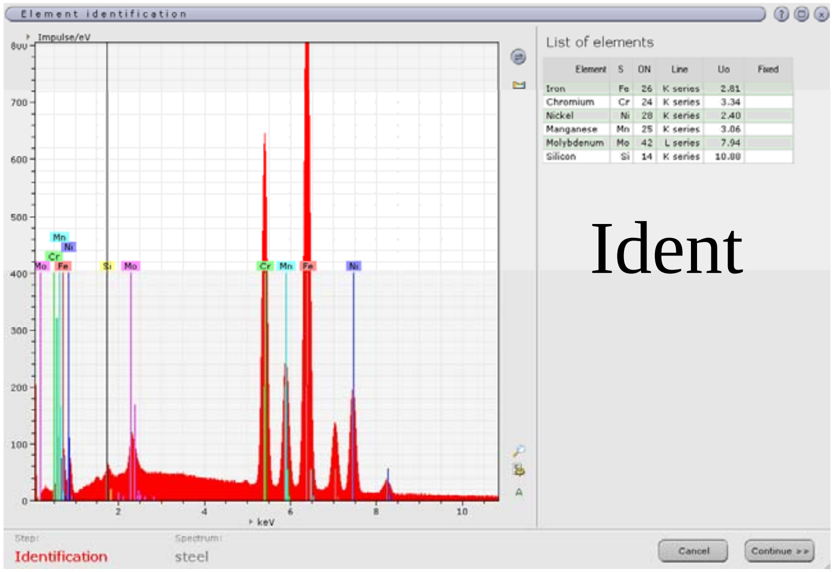
EDS theory on atomic level

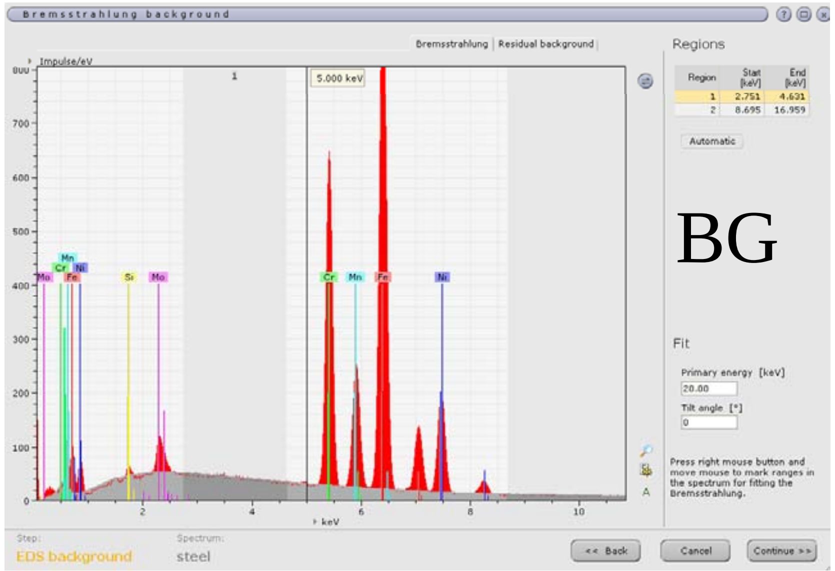
- Characteristic line energies: origin in atomic structure, different for every element in the periodic table
- Line intensities: depend on ionisation probability, photon generation probability, sample composition = **element concentrations**

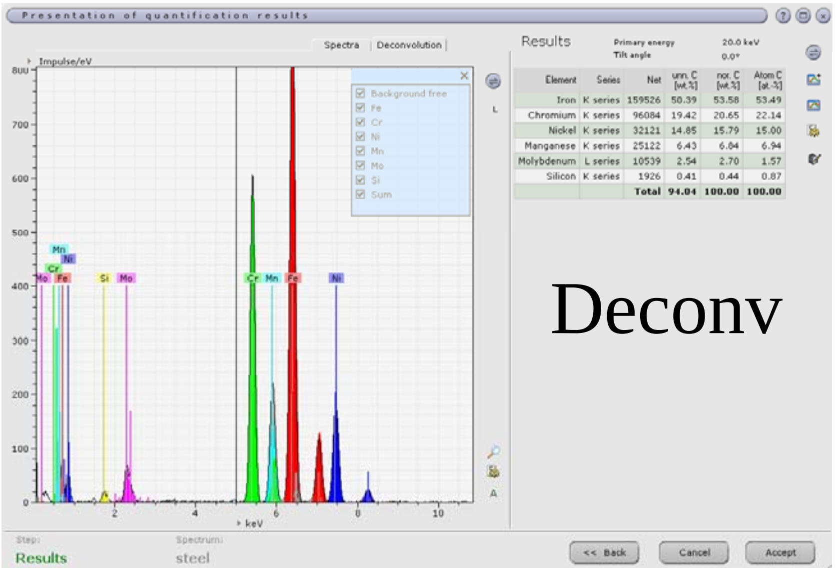


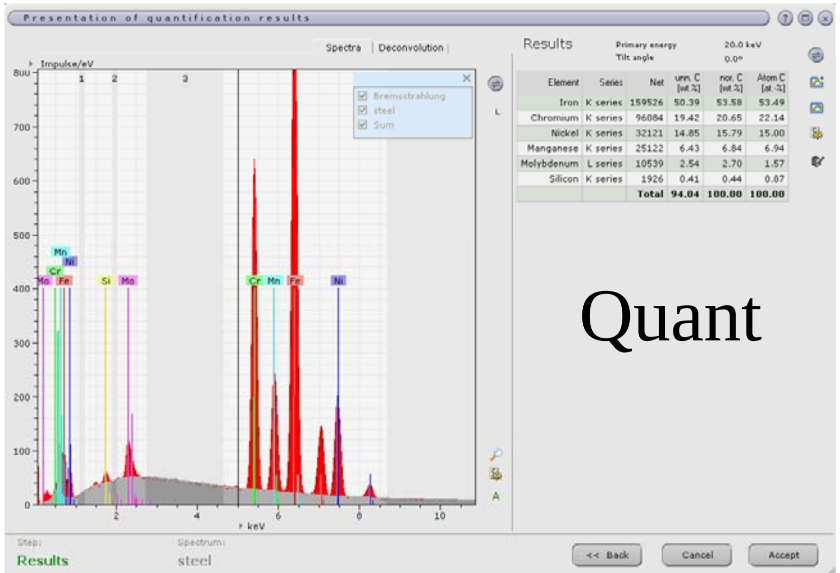


Quantification steps

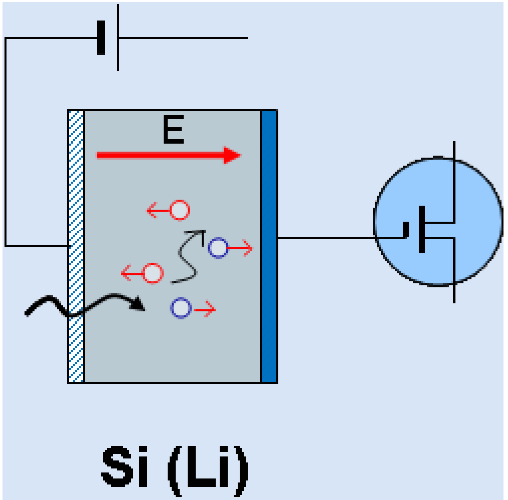


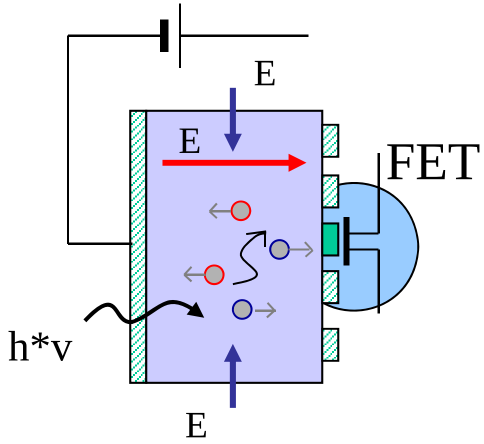






Si- based detector (schematic)





SDD

100…120 fF !

A Si(Li) is built on a bulk silicon semiconductor crystal which has an inert volume, drifted with Li

Charge carriers produced by incoming X-rays are swept to the anode which is the entire surface of the crystal

To reduce noise the detector crystals and the separate first stage amplifier (FET) need to be cooled at -180° C

The SDD is a semiconductor device with a greatly reduced anode spot and drift rings

The drift field guides the charge clouds to the designated anode spot

An integrated monolithically FET amplifies the signal

operating temperature (around -20°C) can easily be achieved by thermoelectric cooling (Peltier)

From LN2 cooled to SDD

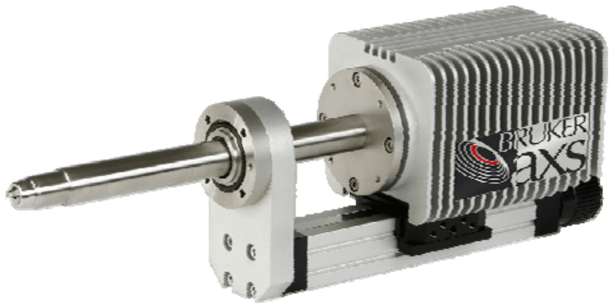
- Why are Silicon Drift Detectors replacing LN_2 cooled Si(Li) systems ?
  - No liquid nitrogen
  - High countrates
  - Good resolution at high countrates
  - Small, less risk for vibration or acoustic disturbance
- Lately also :
  - Resolution down to 123 eV
  - Detection down to Be
  - Countrates over 1.000.000 possible

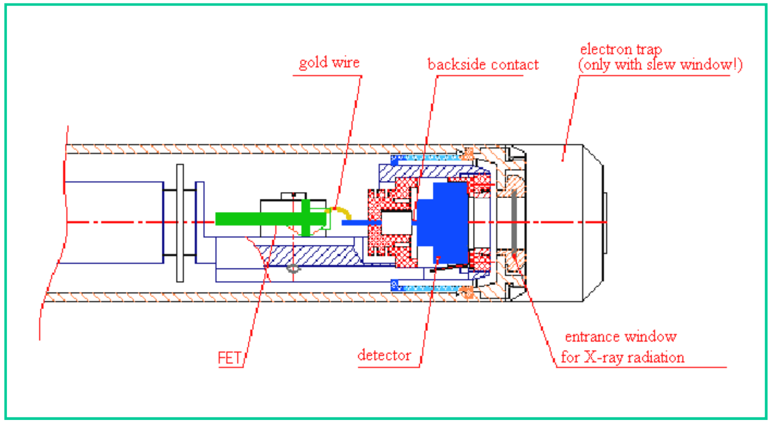
From LN2 cooled to SDD

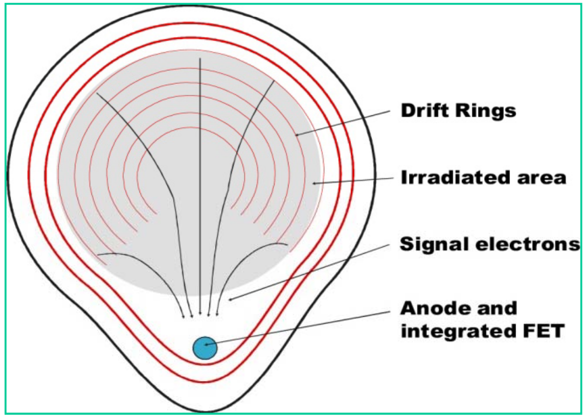
Spectral Solutions

Low capacitance

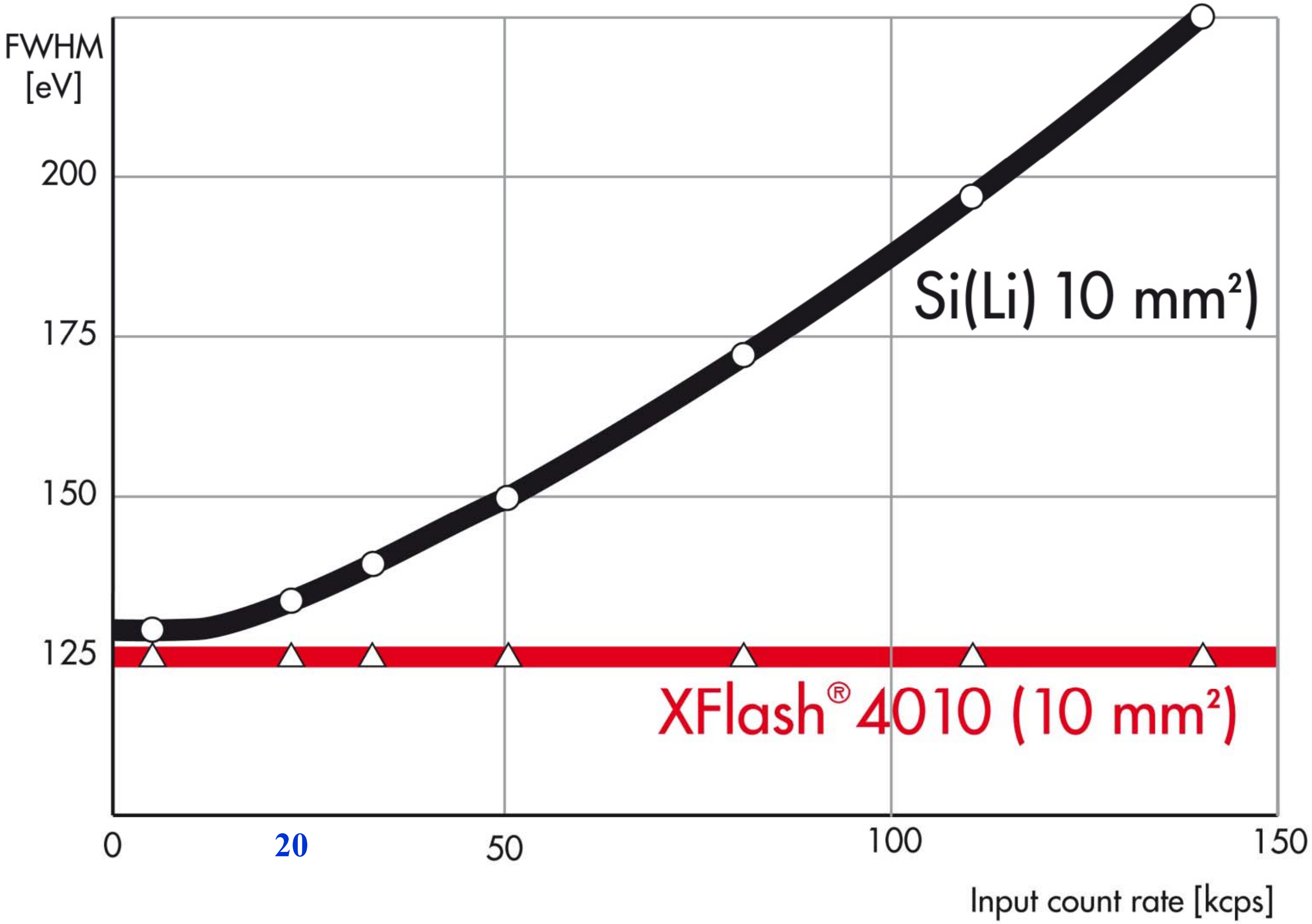
High speed



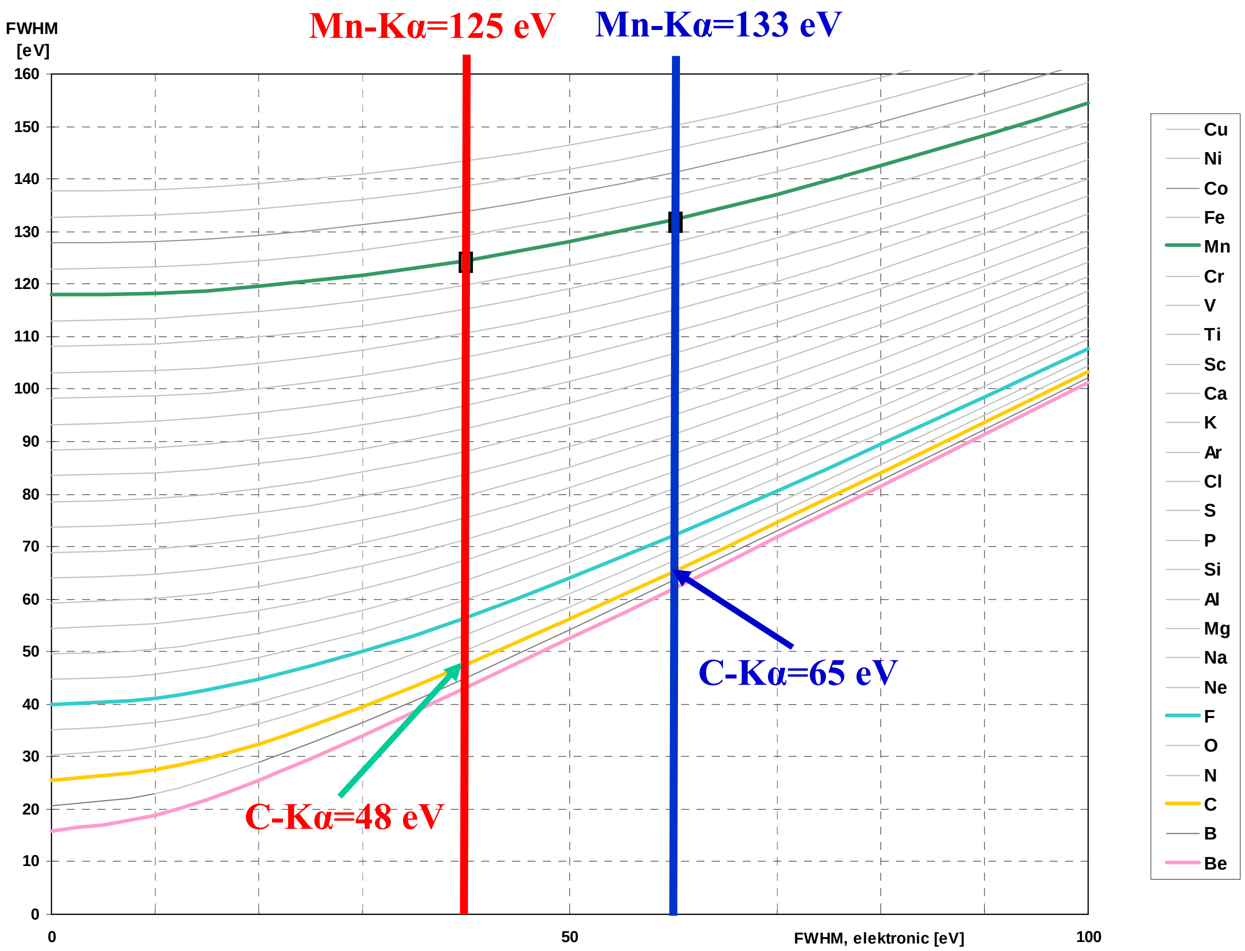




Resolution vs countrate



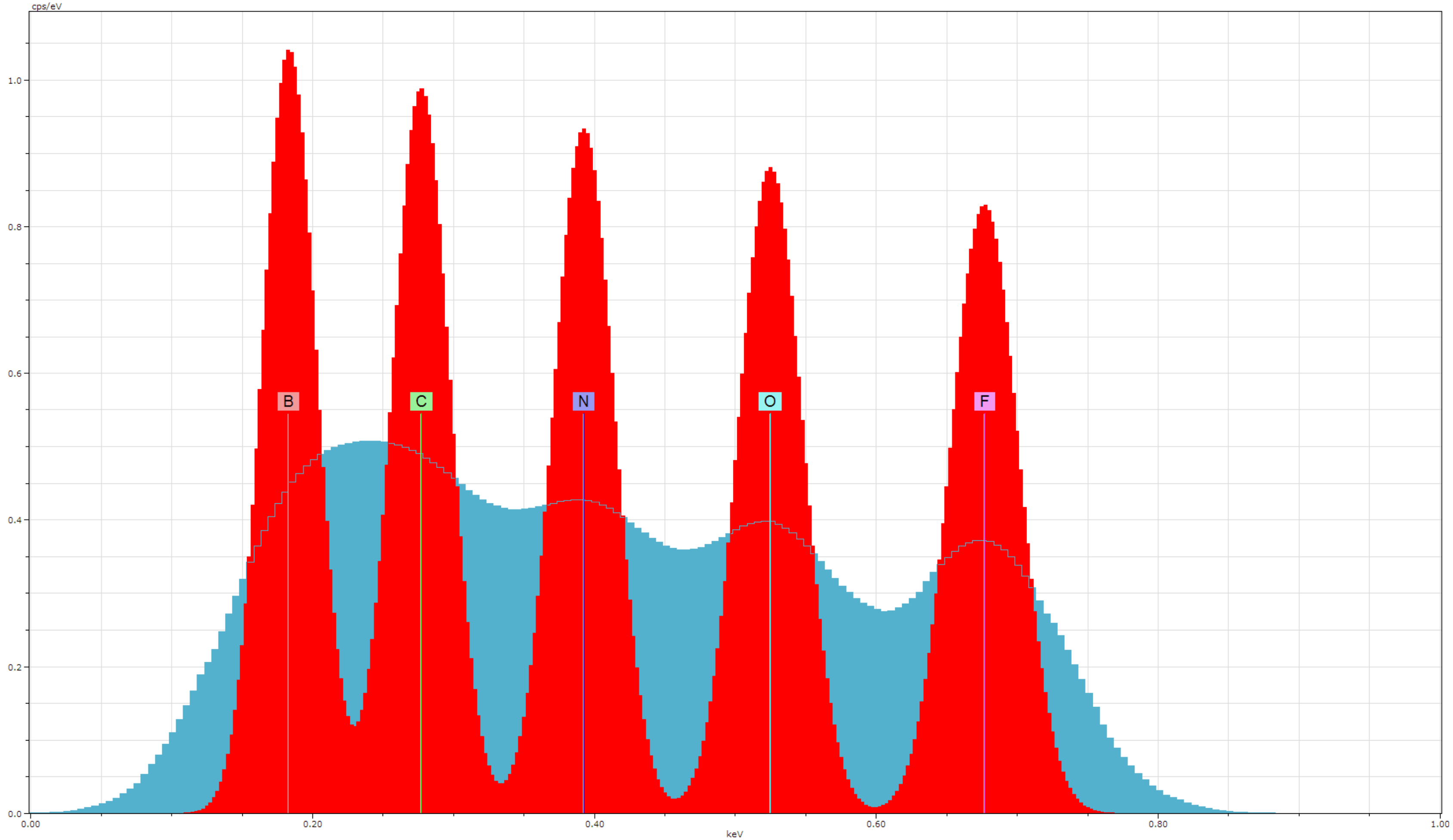
Resolution at low energies



- 8eV at Mn-Kα

- 17eV at C-Kα

Spectra comparison 123 eV / 150 eV @ 60.000 cps output



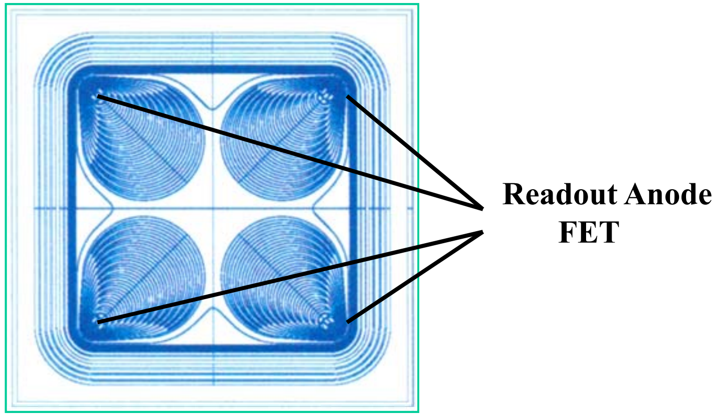
Dual Detector QUANTAX

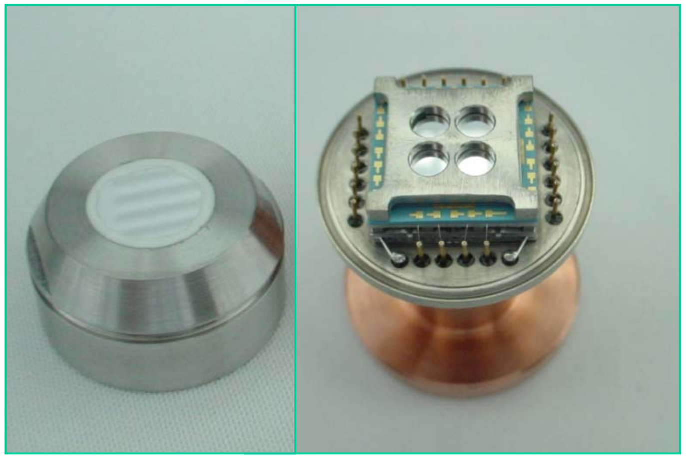
Spectral Solutions

Hitachi SU-1500
With dual XFlash QUANTAX

What about 4 x 10 mm^2 chips ?

- 4th SDD generation, Tear Drop Layout
- 40 mm² active area (4x10 mm²)
- Energy resolution: ≤ 123 - 133 eV
- Detection from Boron (5) and up
- Max. input pulse rate: 3 000 000 cps
- Up to 50 times faster than 30 mm2 Si(Li)
- Vibration-free, maintenance-free





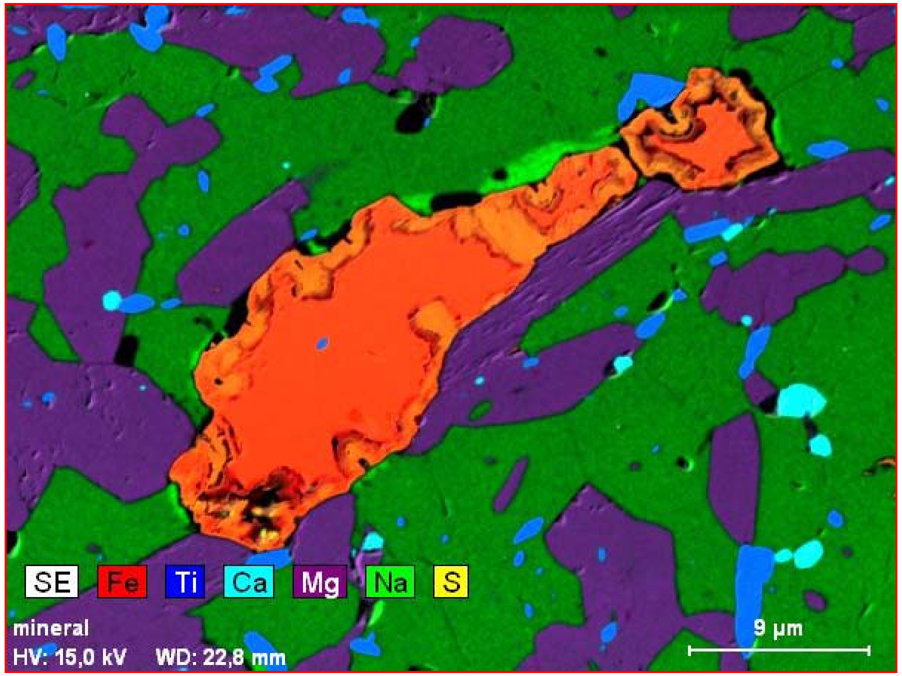
Practical consequence of speed

- XFlash® QUAD 5040
  Spectrometry mode
  - 138 eV resolution
  - 720,000 cps input
  - 40% dead time

  Acquisition time: 7 minutes

- Si(Li) detector (30 mm^2)
  - 138 eV resolution
  - 20,000 cps input
  - 60% dead time
  - Acquisition time: 6.25 hours

- Si(Li) takes 50 times longer

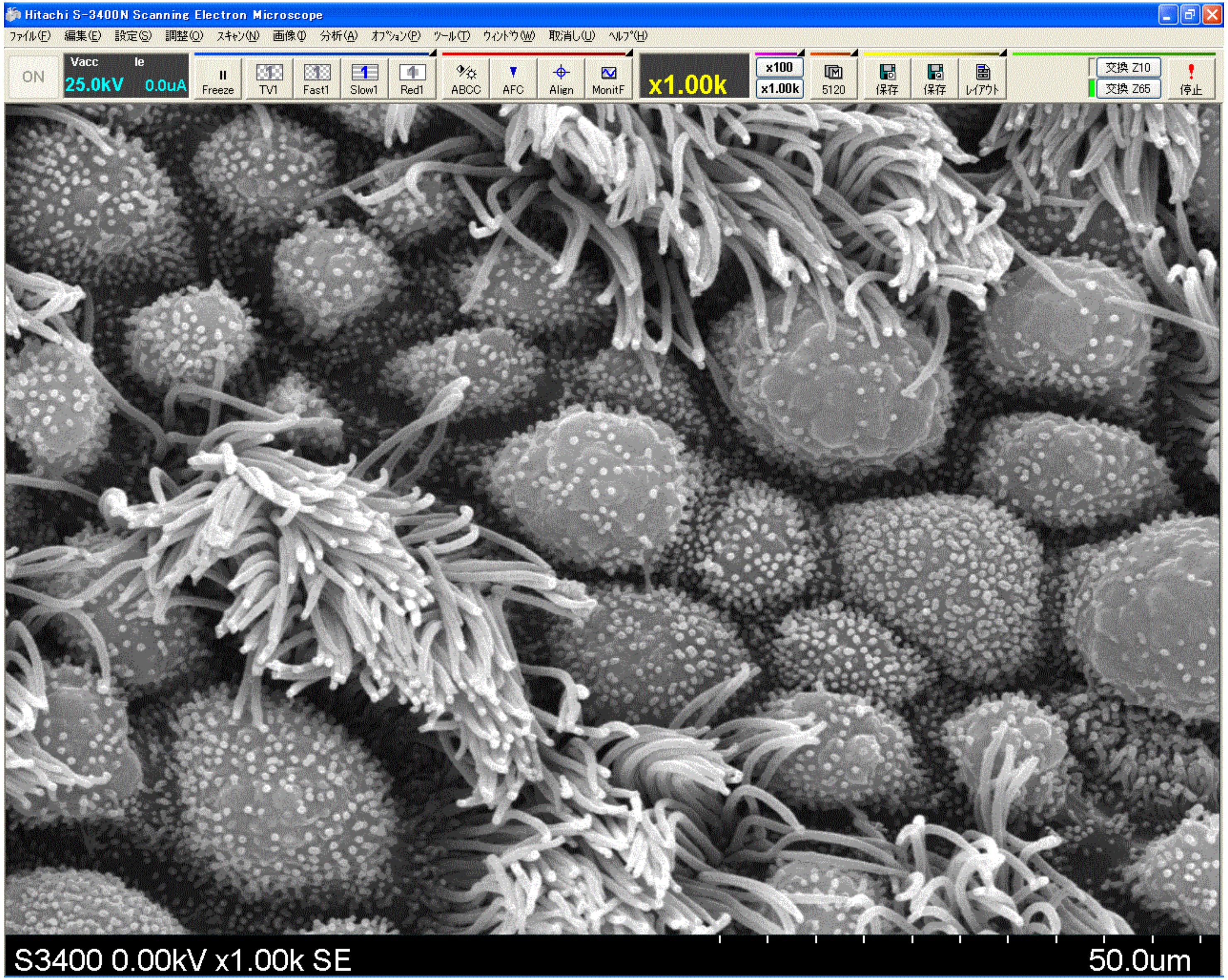


- Mineralogical sample
  - 15 keV
  - 600 x 450 pixels

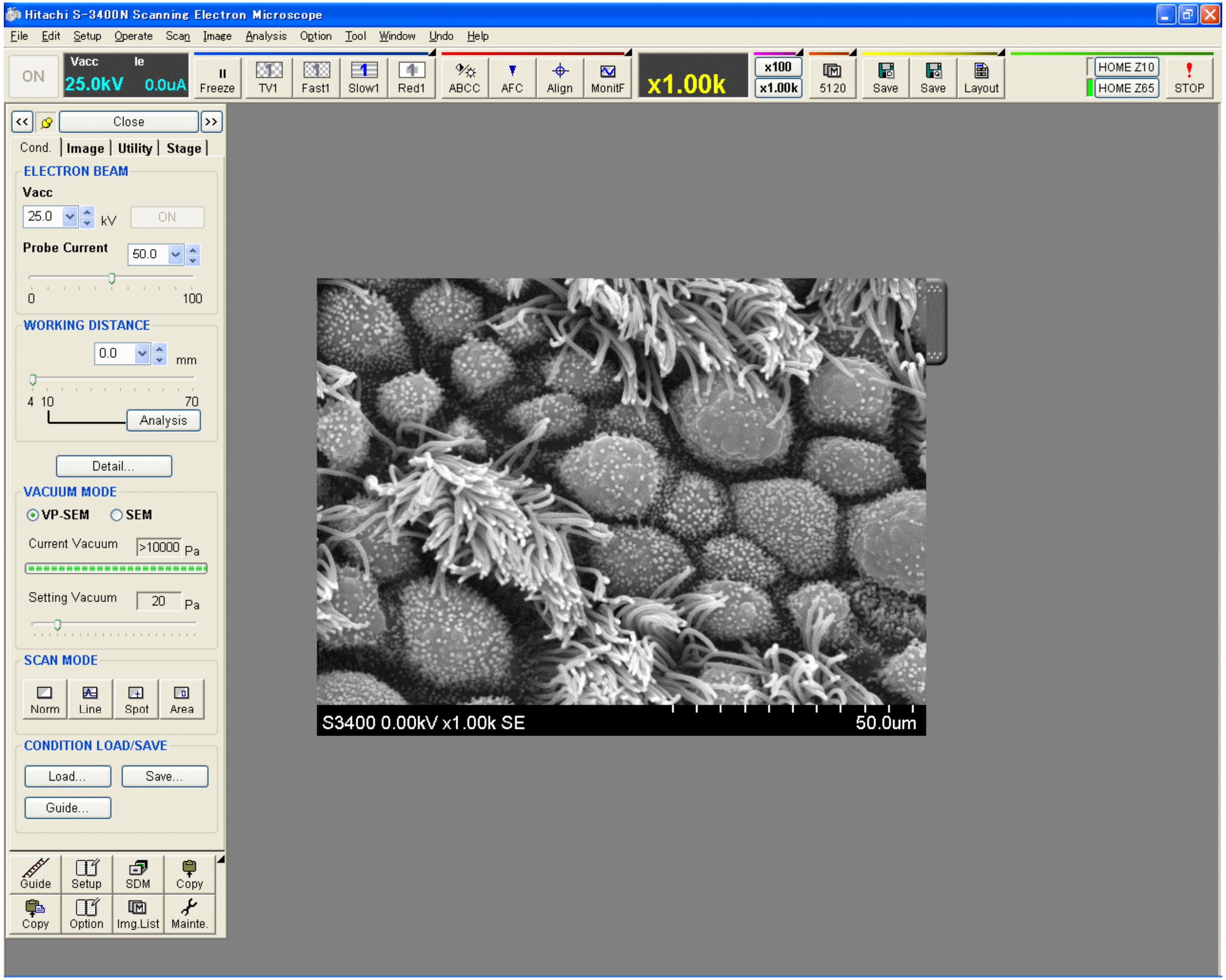
Thank you !

Hitachi SU-70

Display Mode – Full Screen



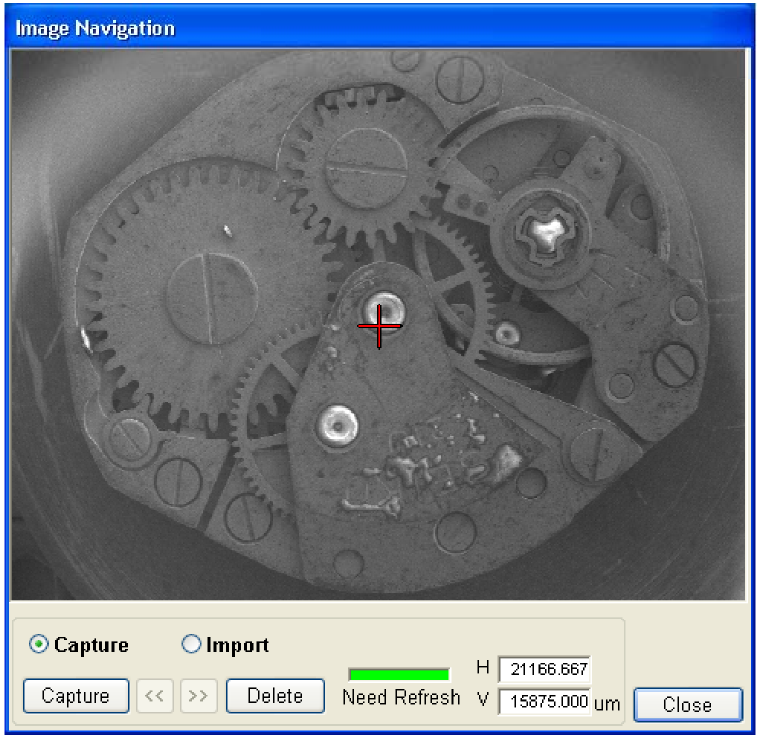
Display Mode - Standard



Sample Navigation

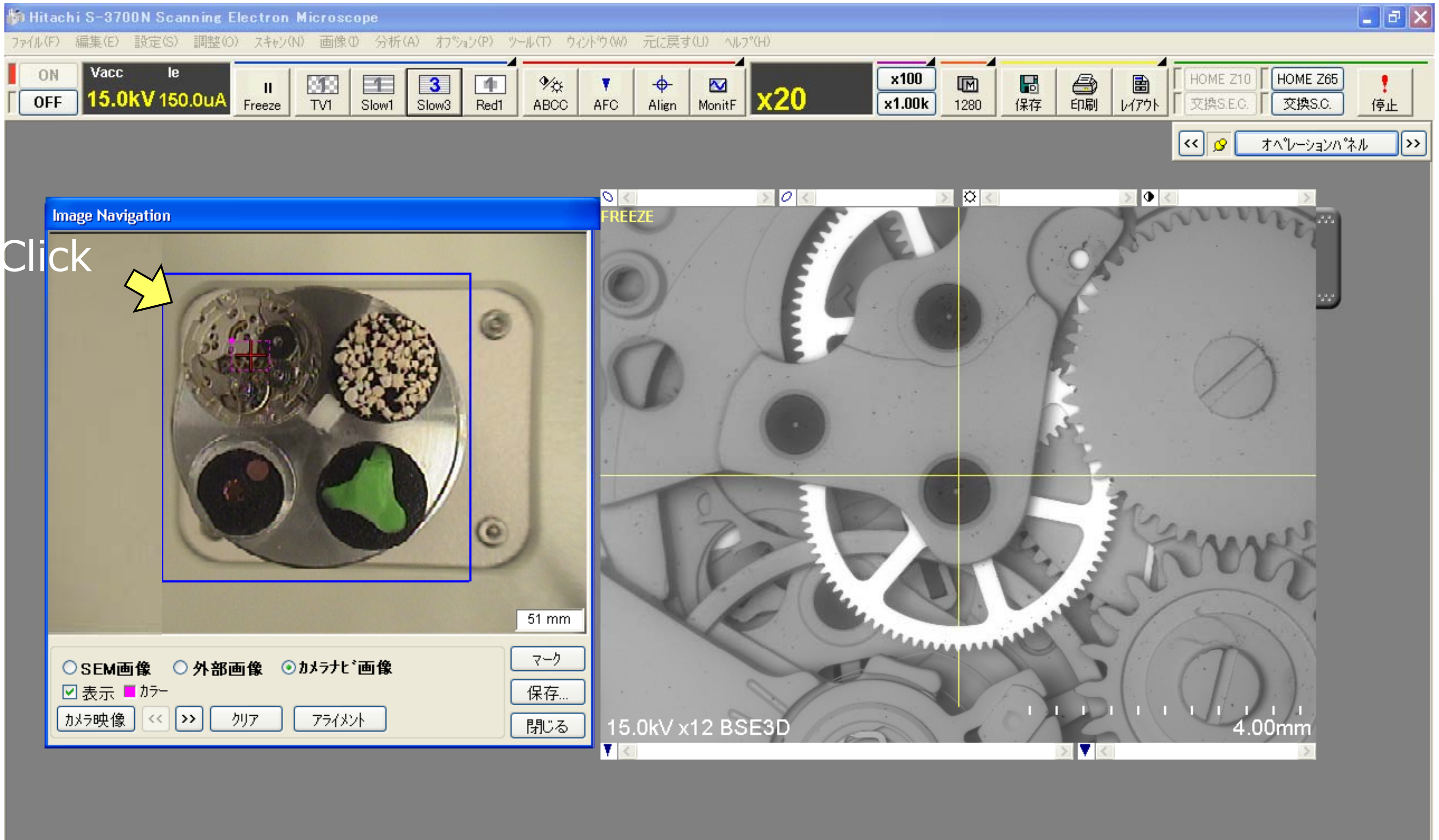
Navigation possible on

- Low-mag SEM images
- Imported Grafics (jpg, bmp, tif)
- Optical CCD-Camera-Images



Point of interest can be centered by mouse click or dragging.

Spectral Solutions



Mouse-Click at desired target location moves the point to the screen center.

Navigation on Sample

As each image is stored with the stage coordinate of its recording site, a return to a previous observation site is possible at any time.

Image Management

Spectral Solutions

SEM Data Manager allows post-processing (measurement, labeling, filtering) and administration of recorded images.