FE SEM TRAINING:

Purdue University

"Hitachi" Bob Passeri

Hitachi High Technologies

Hitachi S-4800 II

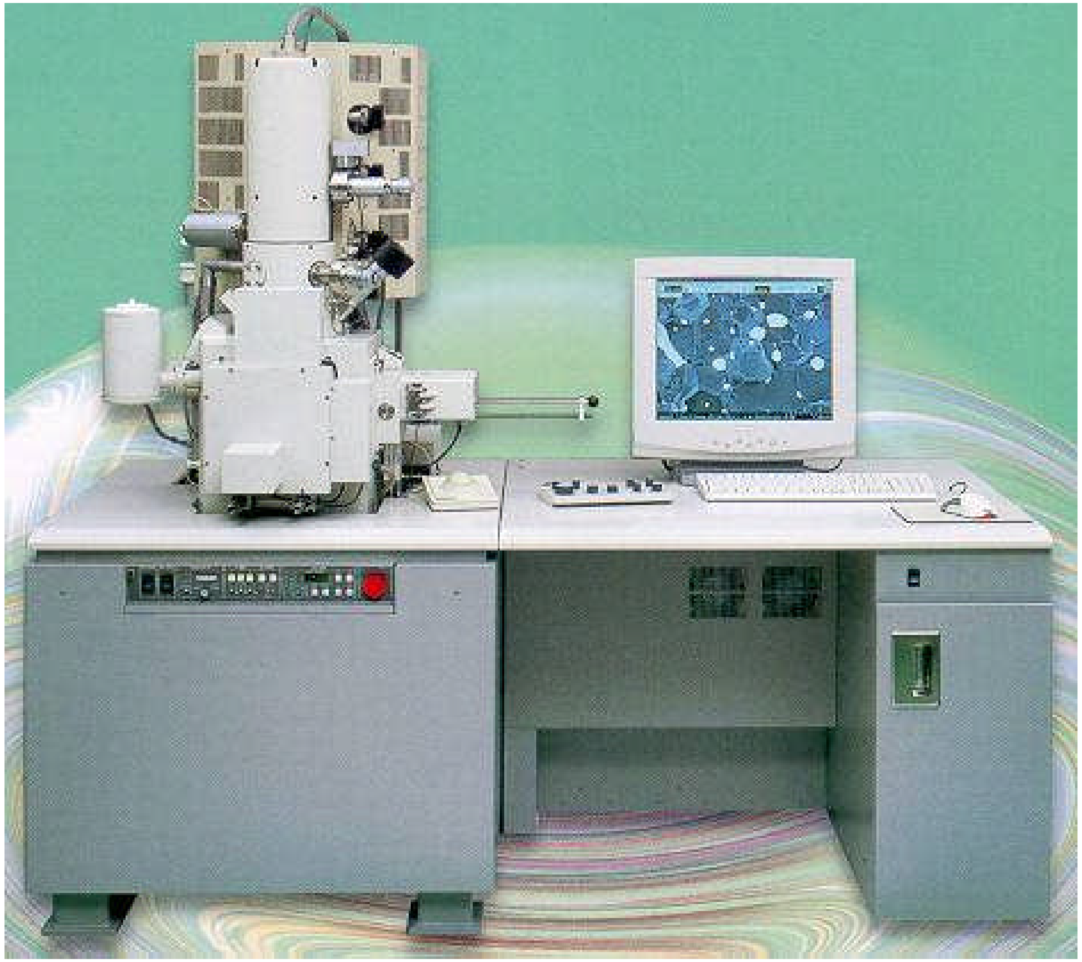
Field Emission Scanning Electron Microscopy

Course contents:

- Basic principle of SEM
- Cold Field Emission theory and tip flashing
- Electron beam formation
- Effect of accelerating voltage change
- Effect of spot size change
- Working distance
- Beam : Sample interaction
- Signal collection – detectors (SE upper and lower, BSE, EXB)
- Advanced imaging techniques – including low kV, charge suppression and signal mixing
- Deceleration mode
- Set up and daily operation

- S-4800 Practical sessions throughout the 2 days to apply the above

Hitachi S-4800 Field Emission SEM

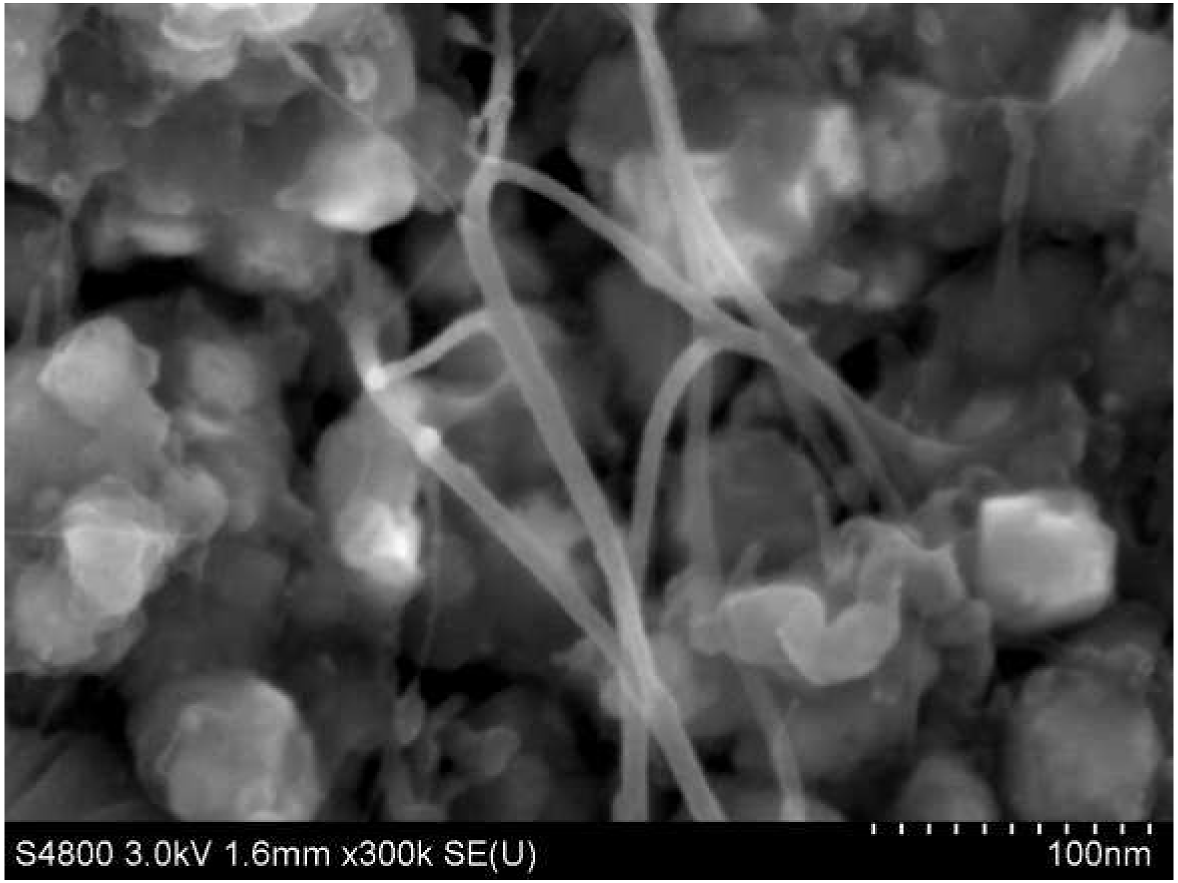


Hitachi S-4800 Type II specifications:

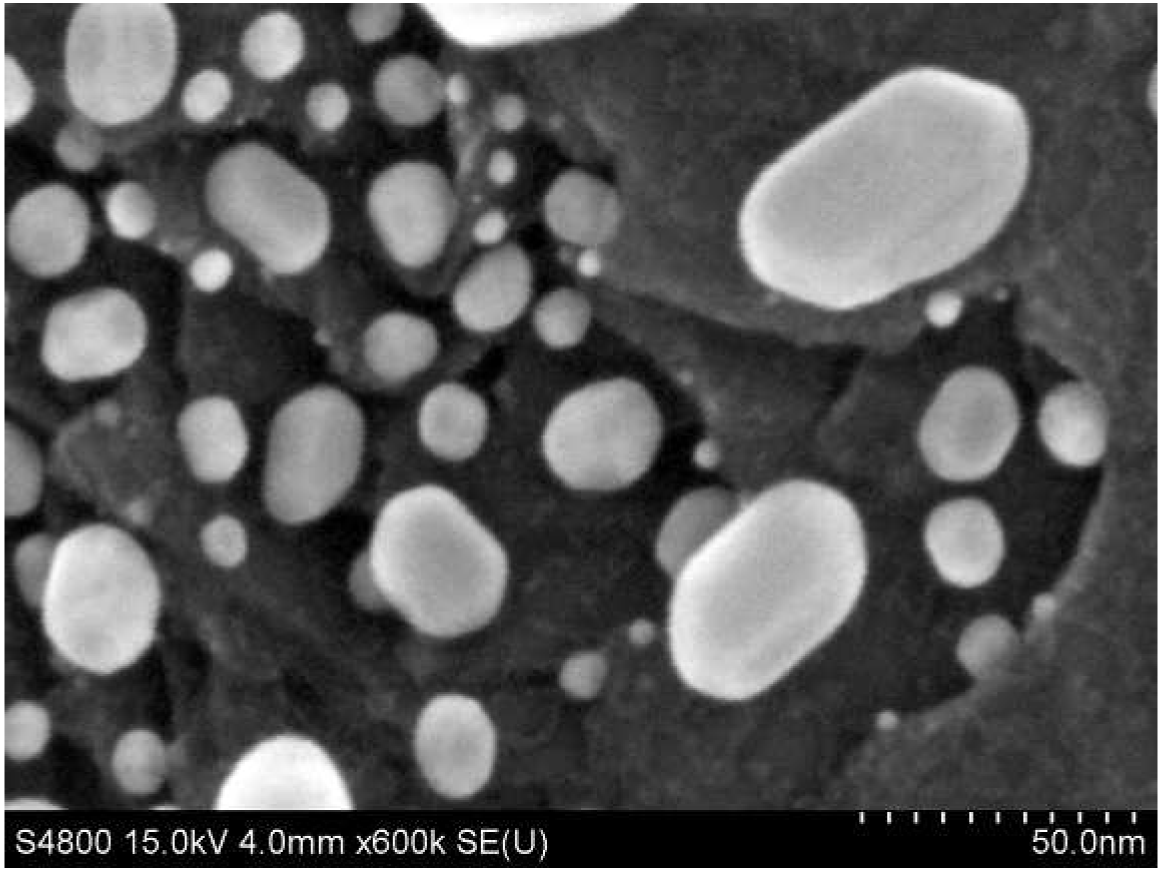
Cold cathode field emission electron source
1.0nm at 15kV (WD 4mm)
1.4nm 1kV (WD 1.5mm, deceleration mode)
2.0nm at 1kV (WD 1.5mm, Normal mode)
Magnification: Low mag mode 20x – 2000x/10,000x
High mag mode 100 – 800,000x
0.5kV – 30kV (V0)
0.1 – 2kV (Deceleration)
0 – 6.5kV (V1) Extraction voltage range
Probe current 1pA to 2nA
Stage – x = 110mm, y = 110mm, z = 1.5 - 40mm, tilt = -5 - +70 degrees
Max specimen size (for exchange chamber) 150mm diameter
3 stage electromagnetic lens system
4 position objective lens aperture strip 100, 50, 50 and 30µm (heated), click stop
1x dry scroll vacuum pump, 1x TMP (magnetic bearing type), 3x ion pumps
Compressed air line for pneumatic valves
1 – 1.5l/min circulating water to cool objective lens

Examples of S-4800 performance:

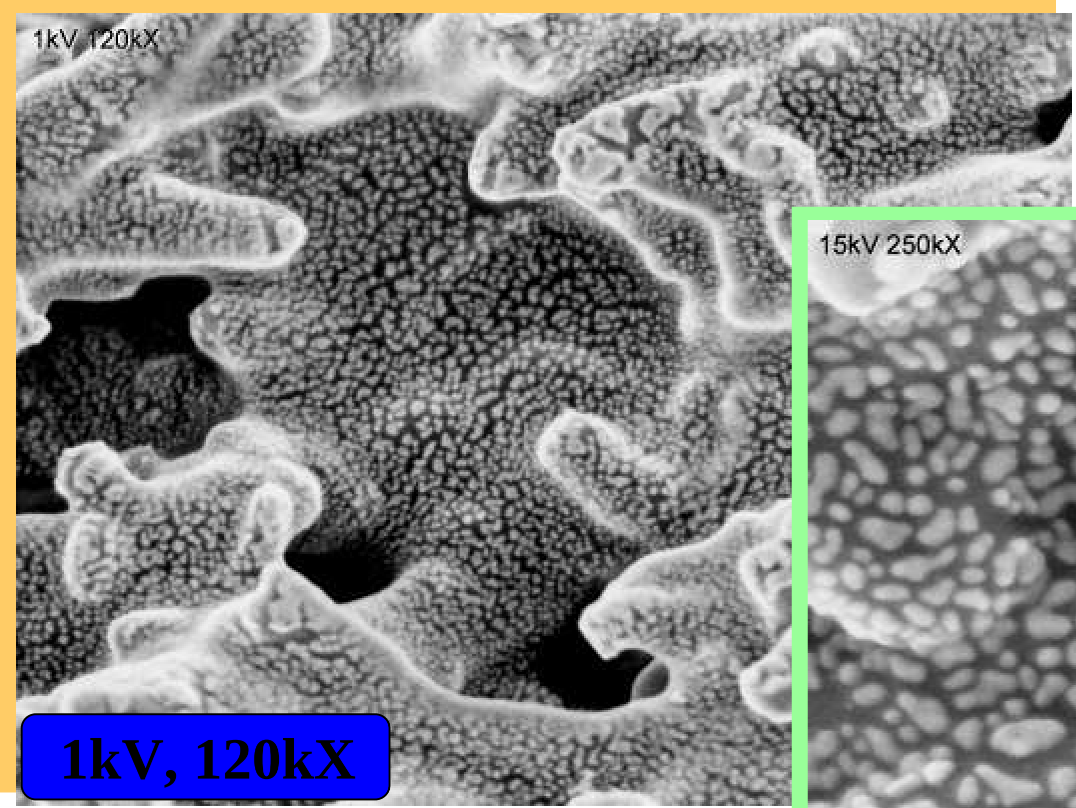
High resolution SE image of carbon nanotubes – low kV reduces penetration

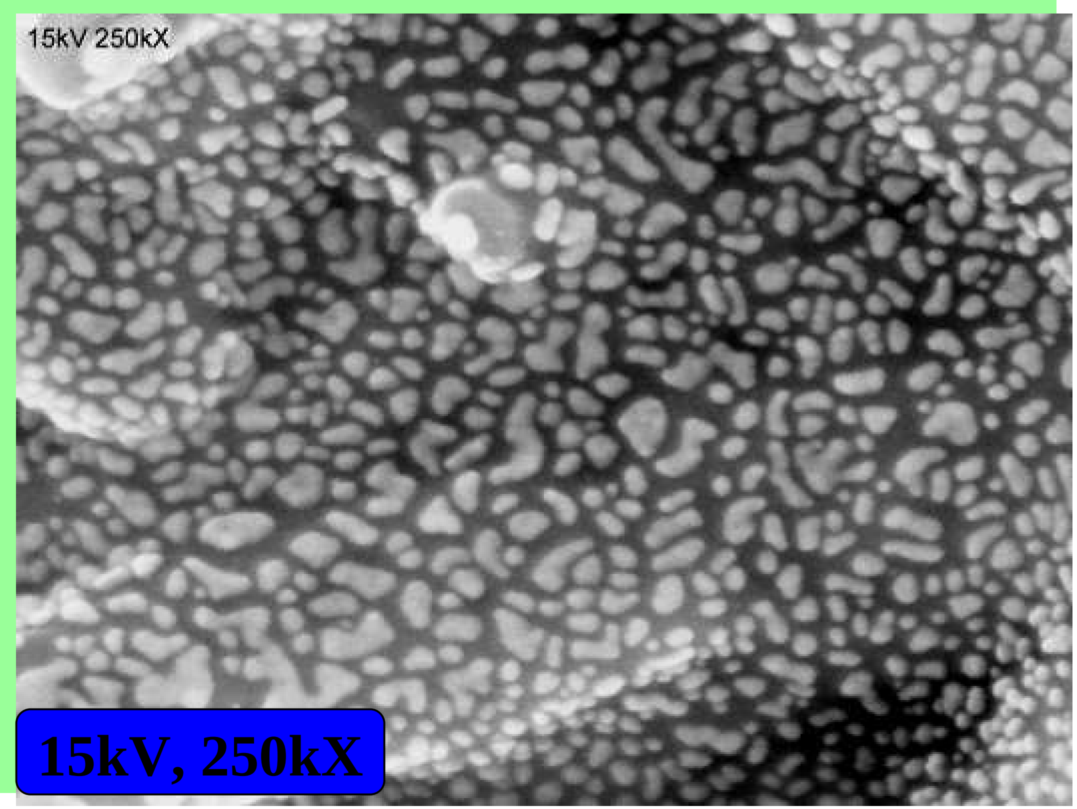


High resolution SE image at 15kV

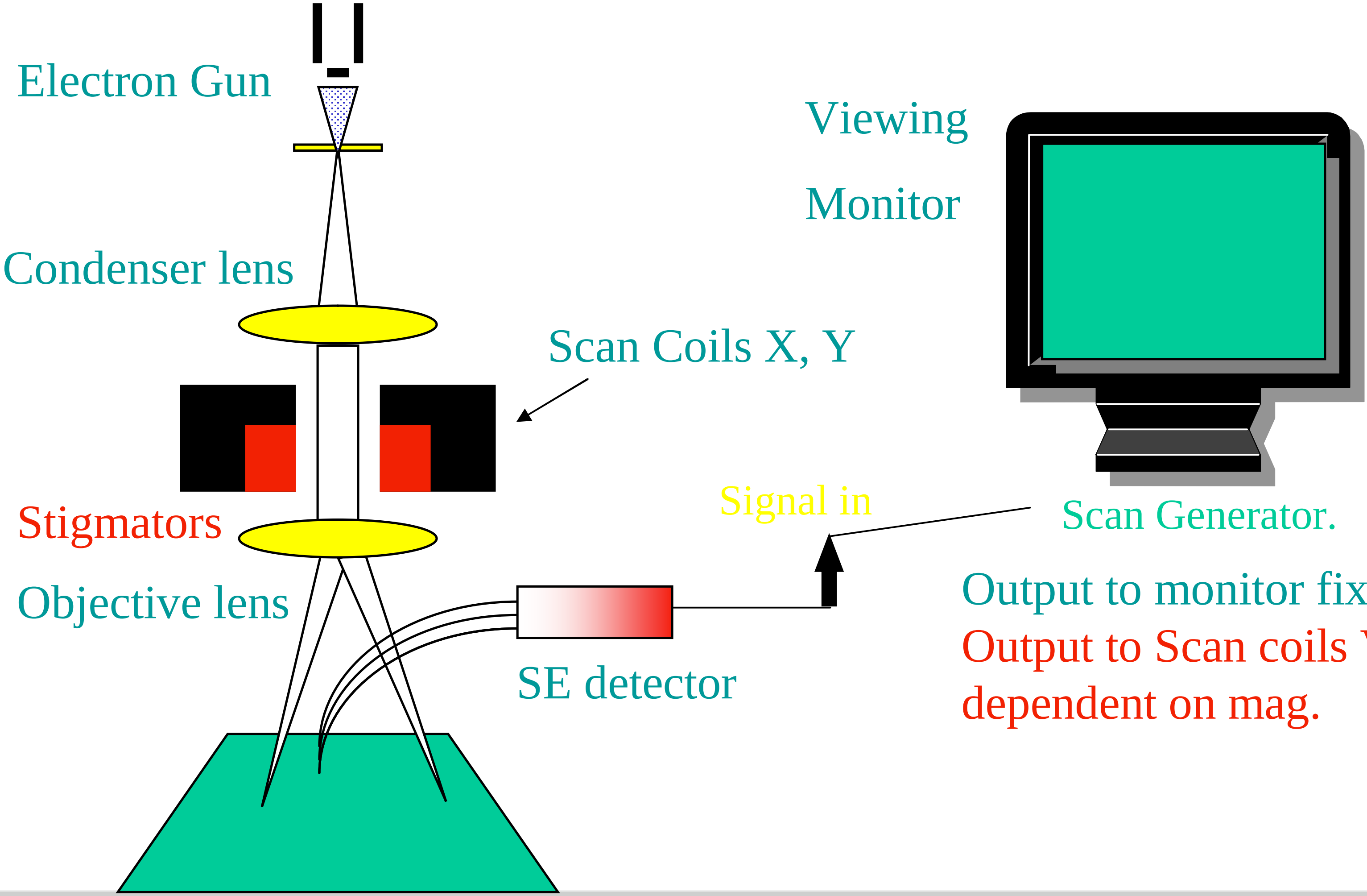


S-4800 Resolution





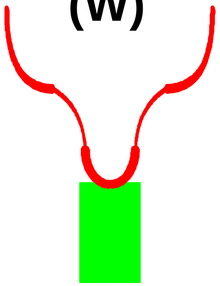
Basic Theory of SEM



Output to monitor fixed.
Output to Scan coils Varies dependent on mag.

Beam Formation: Electron Sources

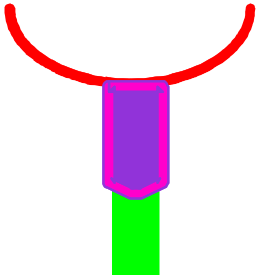
Tungsten (W)



10 - 20kÅ
Beam Diameter
2300ºC Operation
1x Brightness
300hrs. Life

10^{-5} Torr Vacuum

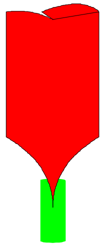
Lanthanum Hexaboride (LaB_6)



10 - 20kÅ
Beam Diameter
1500ºC Operation
10x Brightness
500 - 1000 hrs.

10^{-7} Torr Vacuum

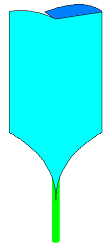
Thermally-Assisted FE (Schottky)



100 - 250Å
Beam Diameter
1500ºC Operation
500x Brightness
>4000 hrs. Life

10^{-9} Torr Vacuum

Cold-Cathode FE



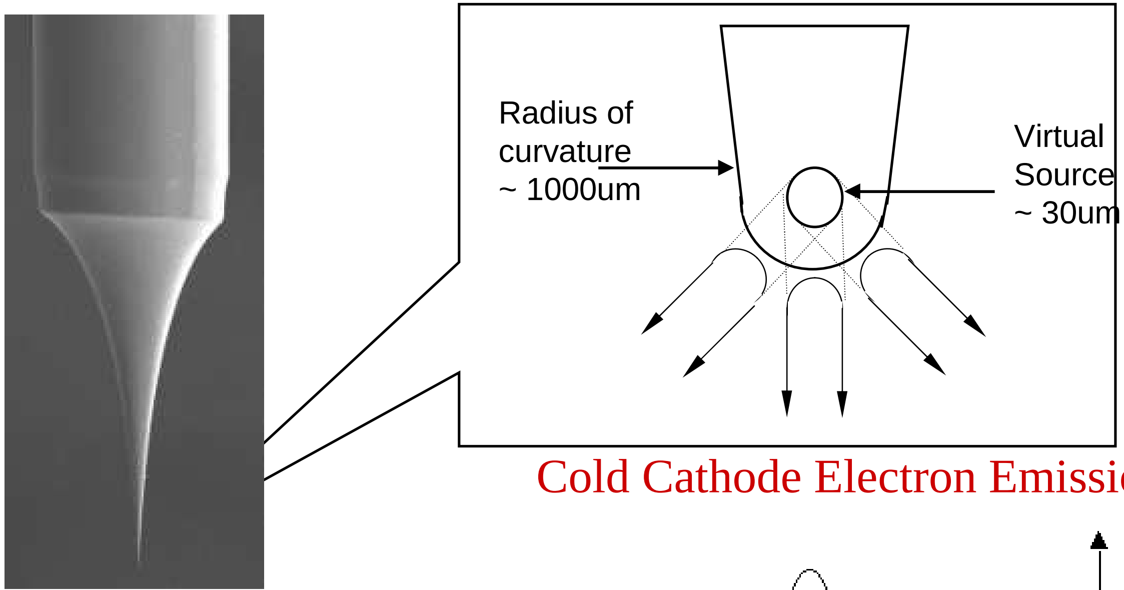
30 - 50Å
Beam Diameter
Ambient Temp.
1000x Brightness
>10000 hrs. Life

10^{-11} Torr Vacuum

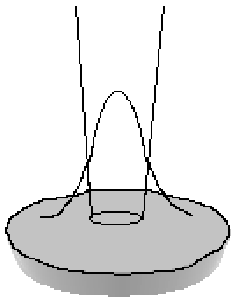
What is Field Emission?

- Work Function Barrier is narrowed
  - **Electrons ‘tunnel’ through the barrier**
- External Potential Applied Against Source
  - **Voltage controls the emission current without heating the source**
- Electrons “Extracted”
  - **Simple Control Of Emission Levels**
  - **Emission diameter is constant, irrespective of emission current set by operator**

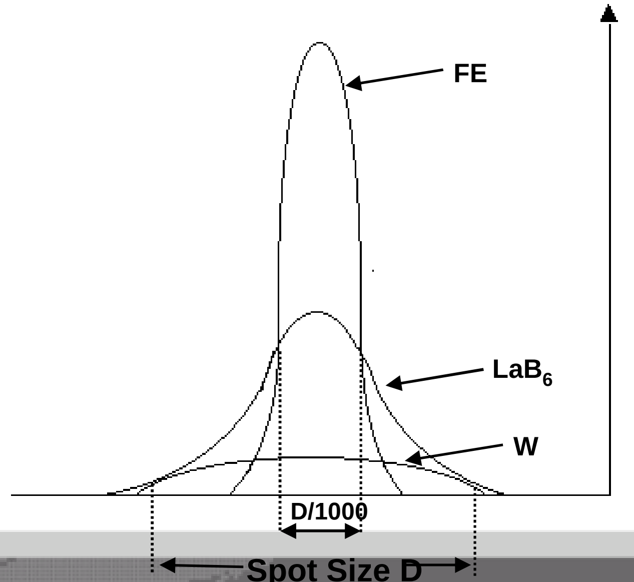
Field Emission Tip



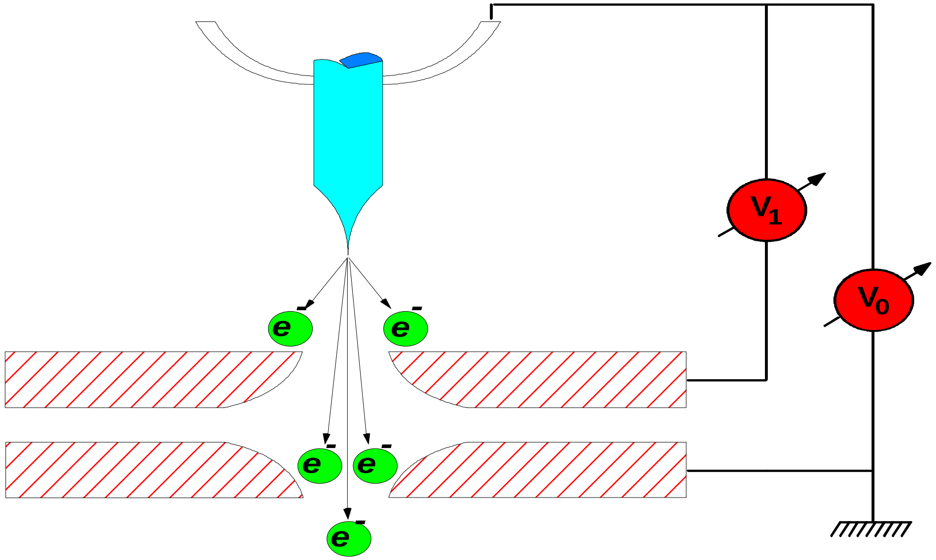
Cold Cathode Electron Emission



Spot Size



Intensity
B X 1000

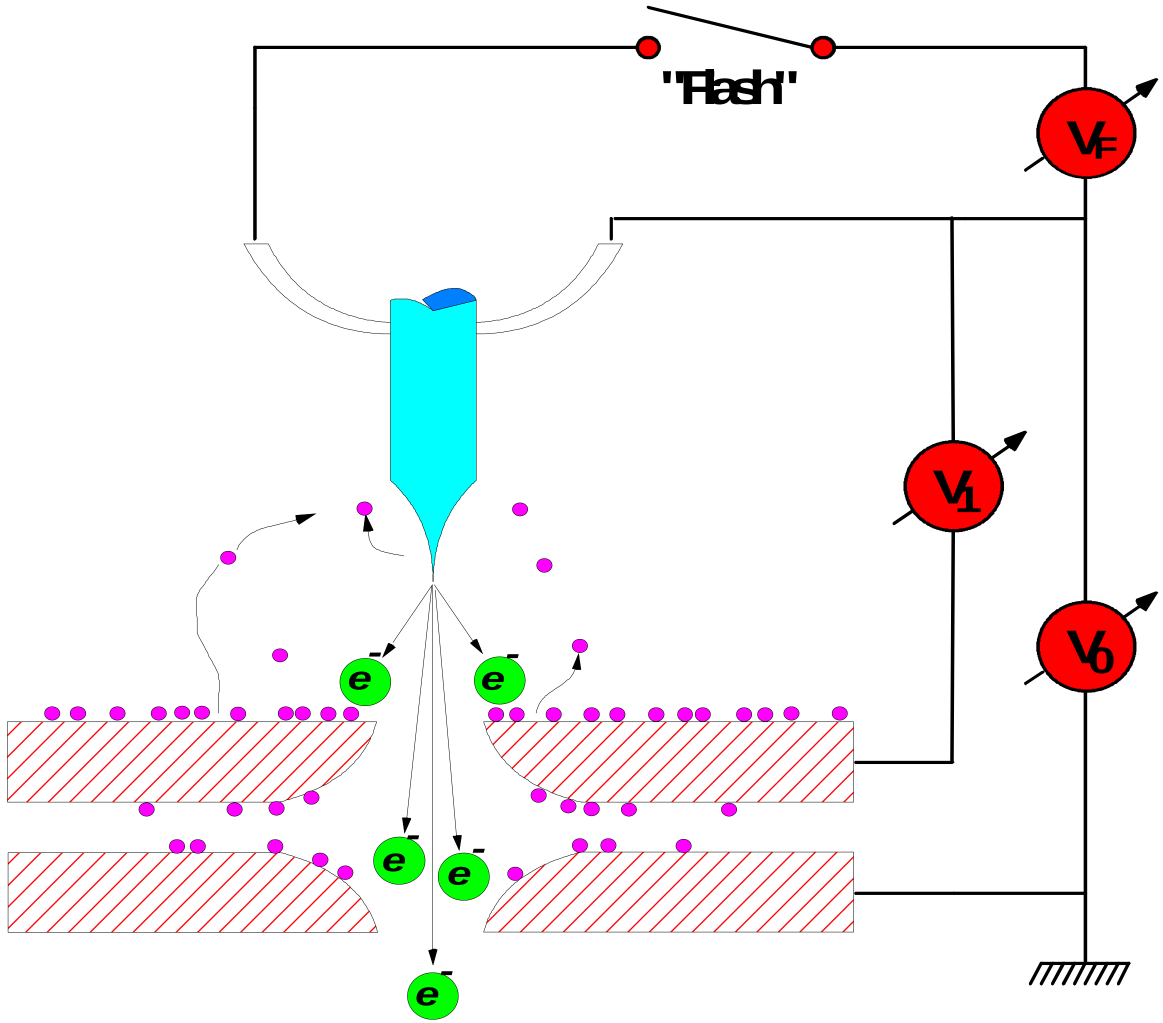


V_0: Accelerating Voltage

- **Computer First Raises This To The Selected Level**

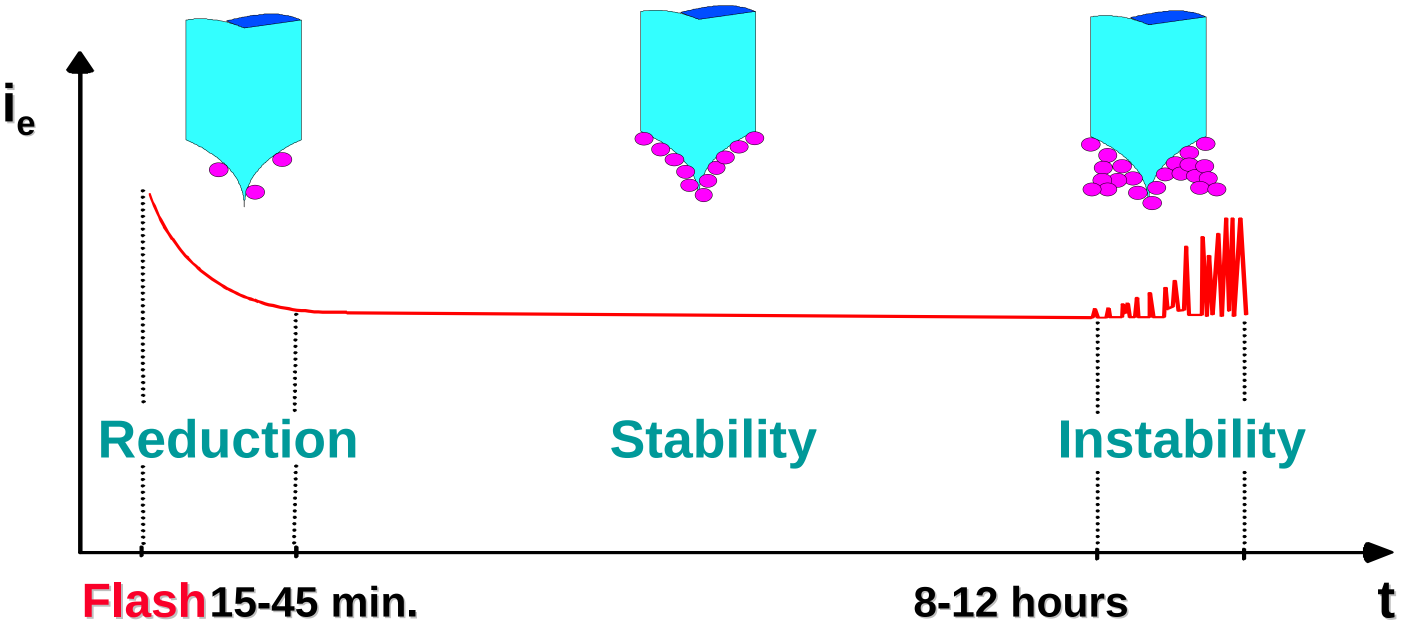
V_1: Extraction Voltage

- **This Voltage Is Raised Until The Desired Amount Of Emission Current Is Obtained, Then It Is Scaled Against V_0 To Deliver The Requested Electron Speed**



V_F=*Flashing* Voltage

CFE "Flashing" Circuit



Reduction Phase	Initial operation after tip flashing... gas molecules begin to coat the tip
Stability Phase	Long-Term Stable Operation... emitter is uniformly coated by gas molecules
Instability Phase	Period Where Instability Begins... **tip noise begins** - automatically corrected

Flashing Should Be Done Now

Cold Field Emission Daily Operation Cycle

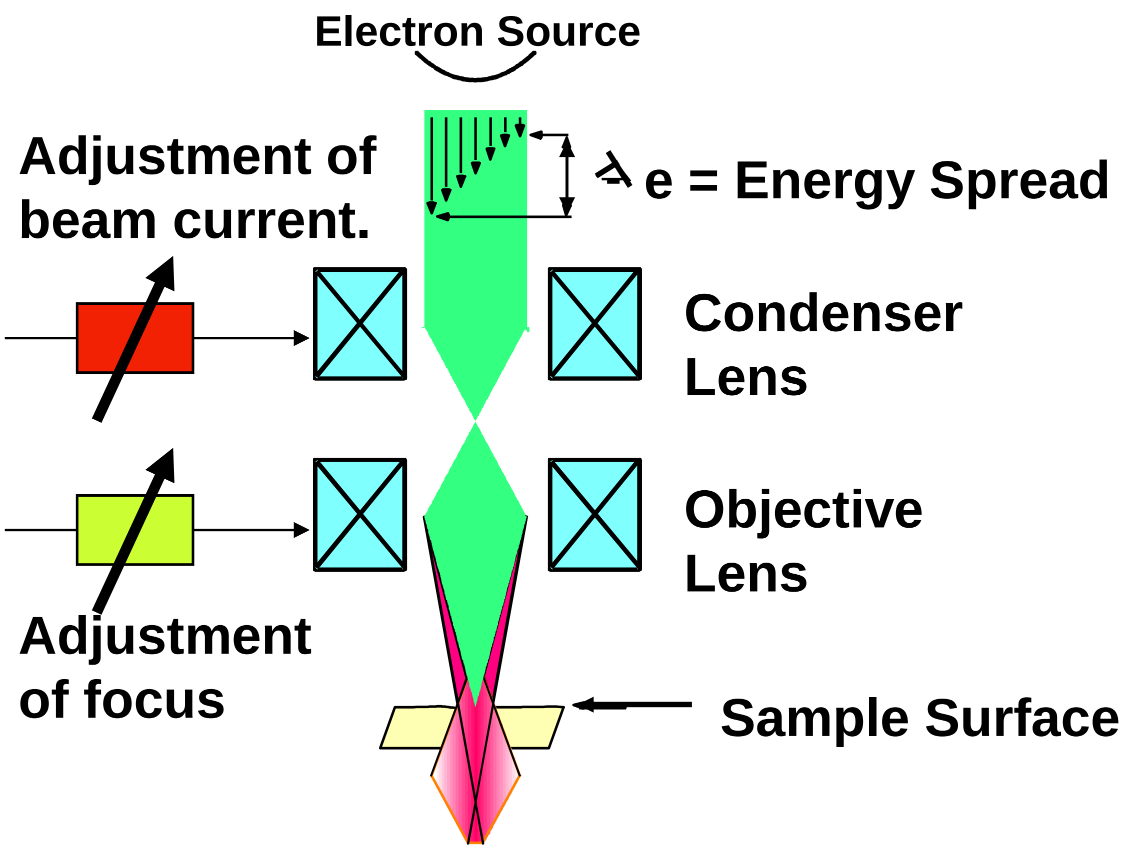
Beam Formation: Purpose of the lenses

To **demagnify** “the crossover” of the electron beam as it exits the Gun by as much as 10,000 times.

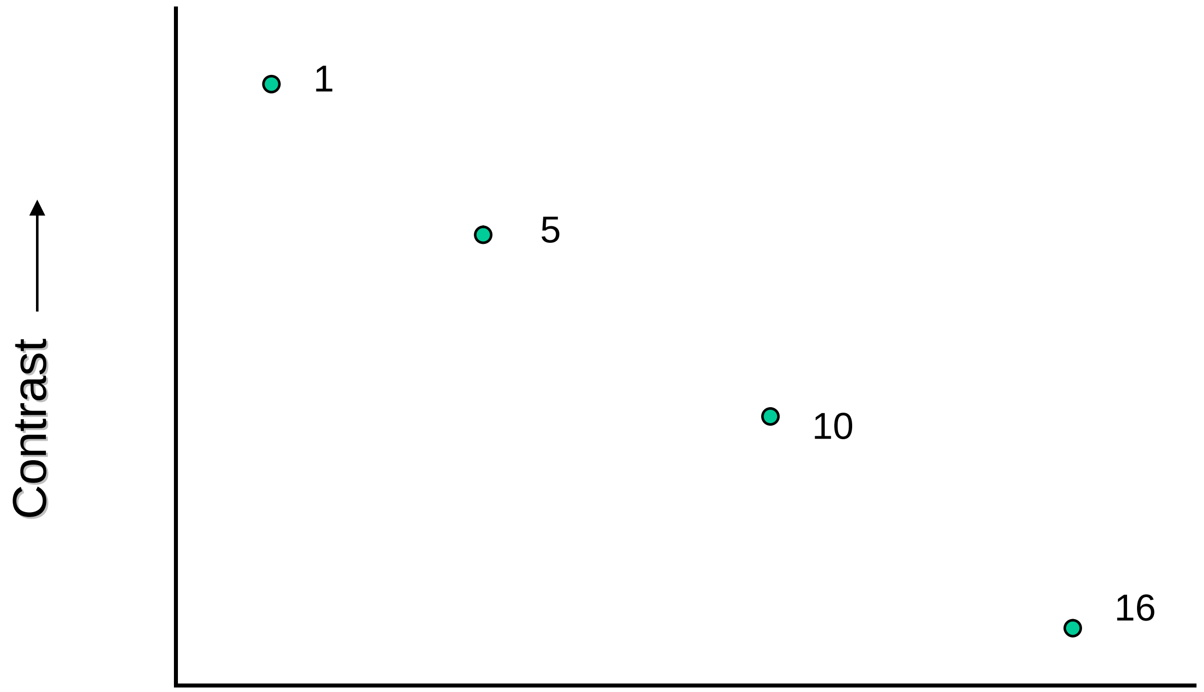
The **condenser lens** adjusts the spot size and beam current impinging on the sample.

The **objective lens** adjusts the beam focus on the sample.

Basic lens system



The Column – Probe Current/Spot Size



Resolution →

Largest spot = 1

Smallest spot = 16

Beam Formation: Lens aberrations

Spherical Aberration

Electrons off axis of the centre of the lens, also causes astigmatism

Chromatic Aberration

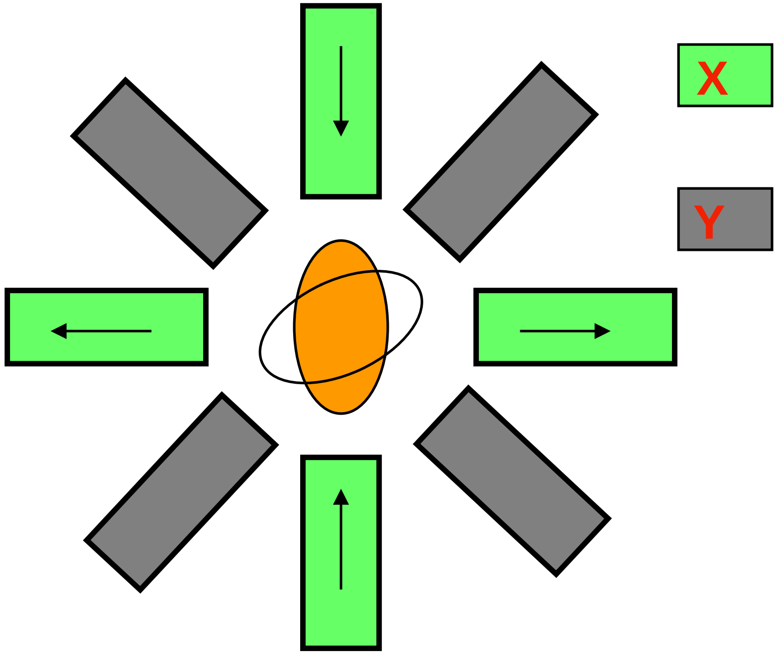
Electrons of various energies become focused at different plains

Astigmatism

Beam from source not spherical, machining of parts not absolutely symmetrical

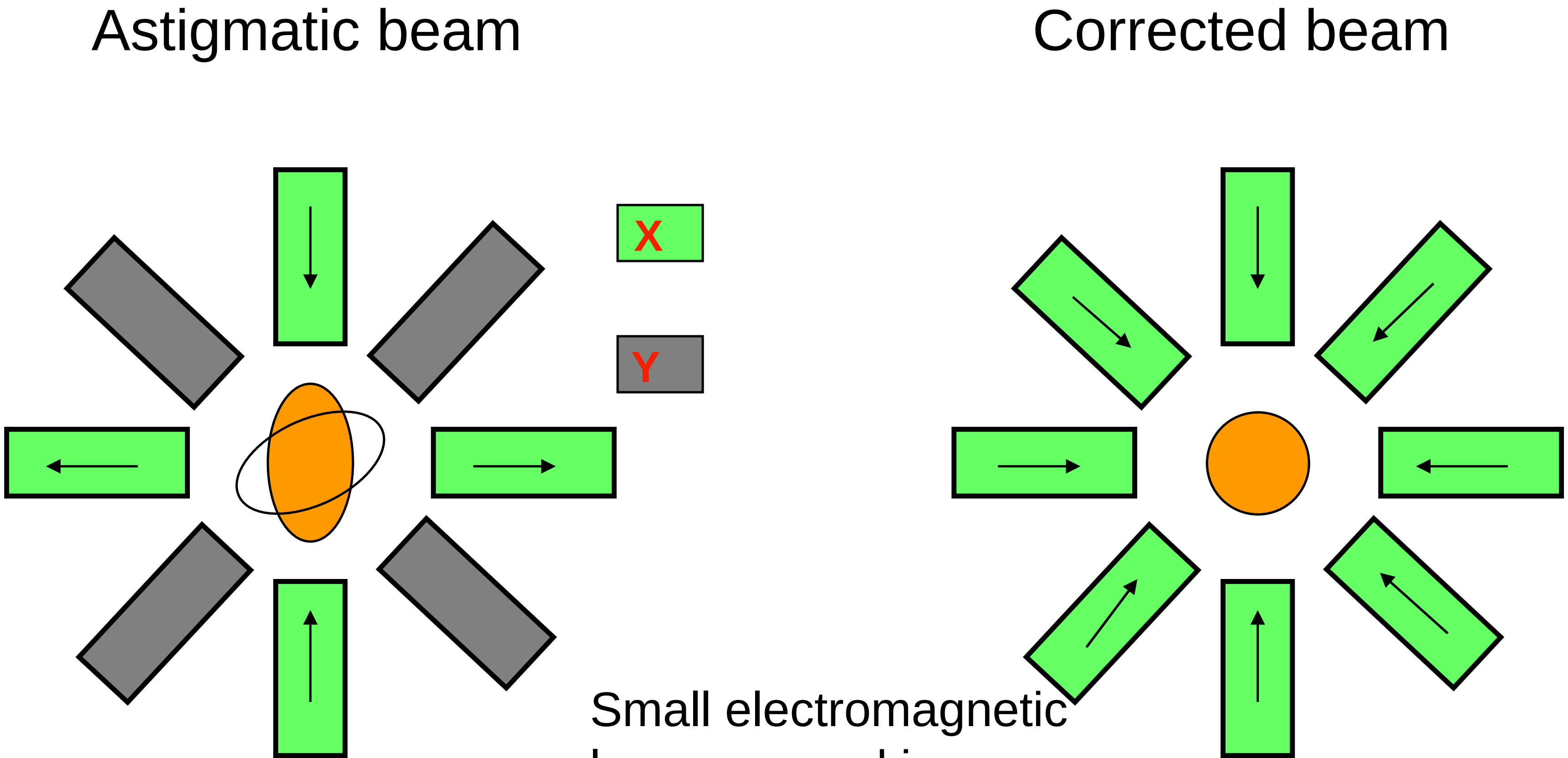
Beam Formation: Correcting Astigmatism

Astigmatic beam



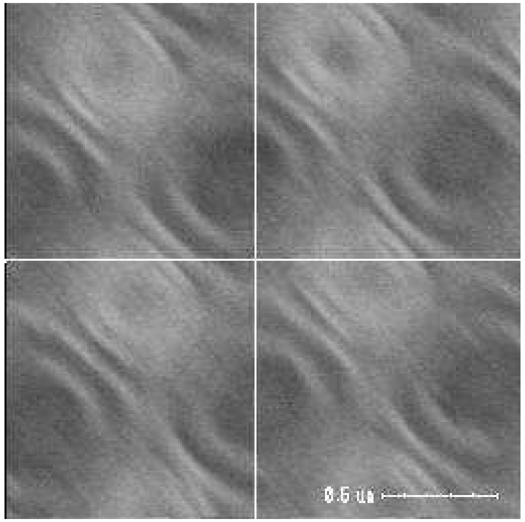
Small electromagnetic
lenses wound in
opposing directions

Beam Formation: Correcting Astigmatism

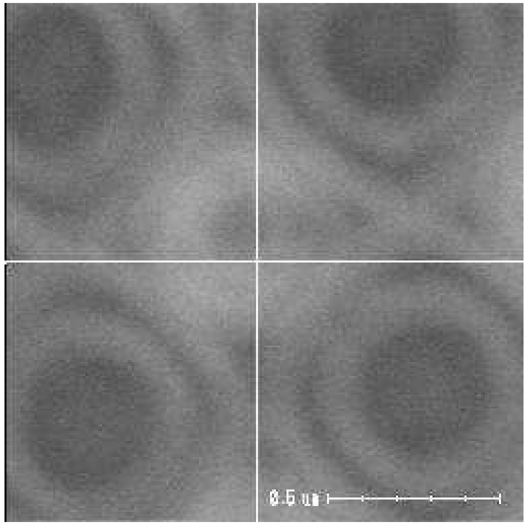


Small electromagnetic lenses wound in opposing directions

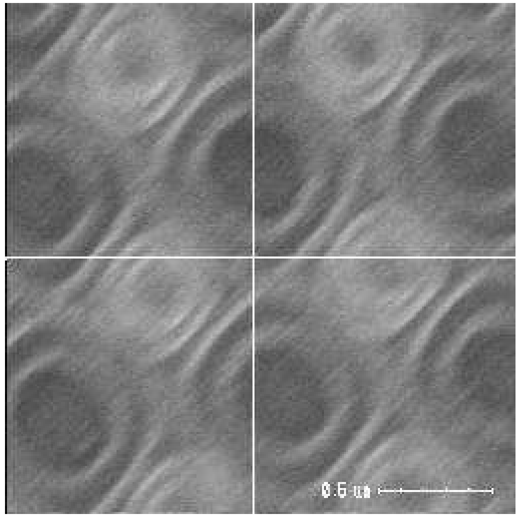
Beam Formation: Correcting Astigmatism



UNDER FOCUS CONDITION

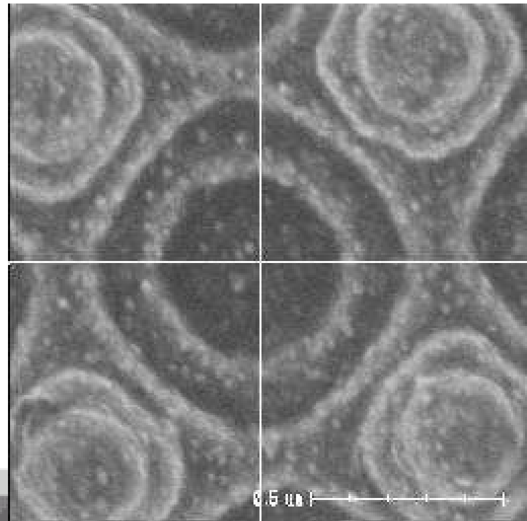


CENTRE OF FOCUS



OVER FOCUS CONDITION

YOU MUST FIND CENTER OF FOCUS FIRST,
<u>THEN</u> ADJUST STIGMATORS ONE AT A TIME, x then y

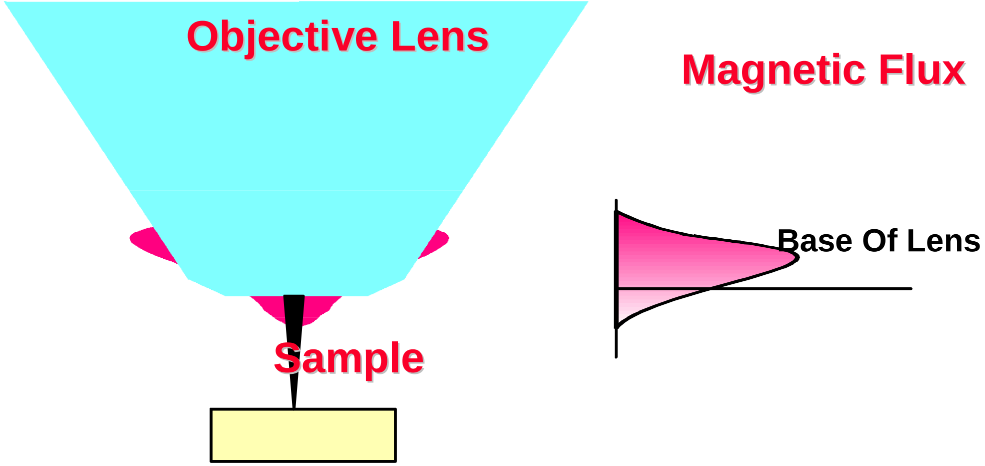


CORRECTLY STIGMATED

NOTE: VERTICAL AND HORIZONTAL EDGES ARE NOW IN FOCUS

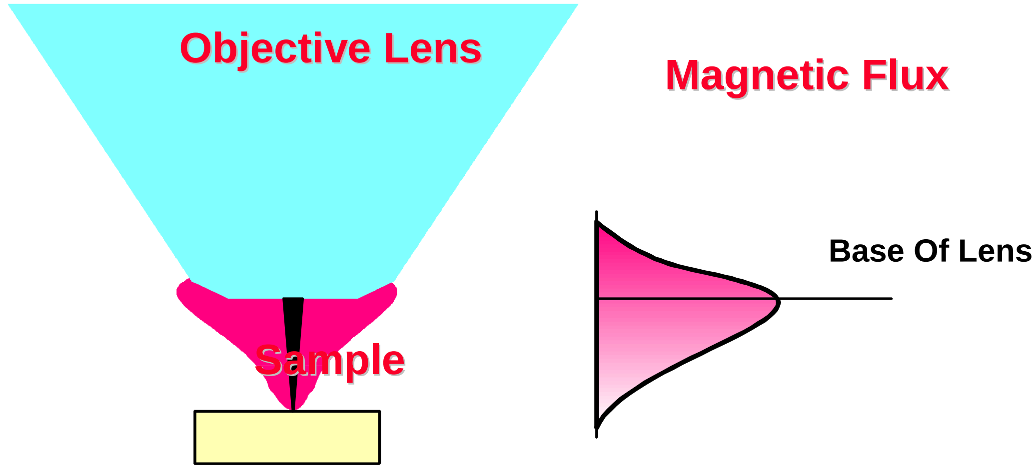
Hitachi Single-Pole Snorkel Lens

Conventional lens



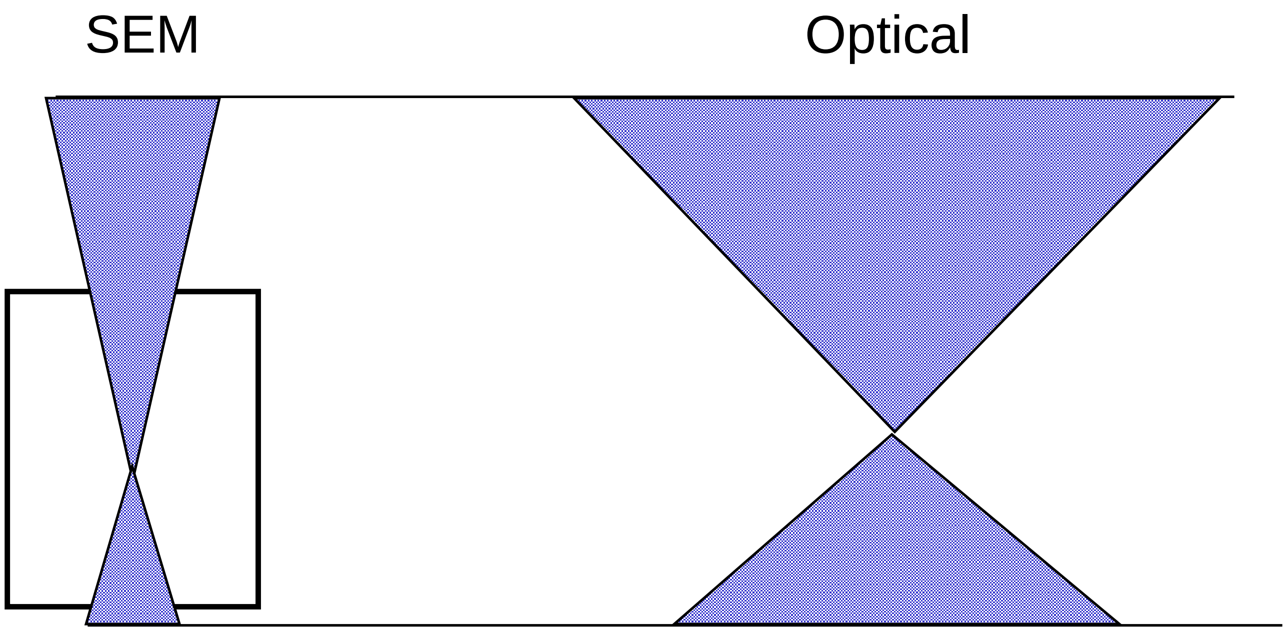
Objective lens field is contained within the pole piece leaving exiting primary and subsequent image electrons vulnerable to local fields and distortion

Snorkel lens



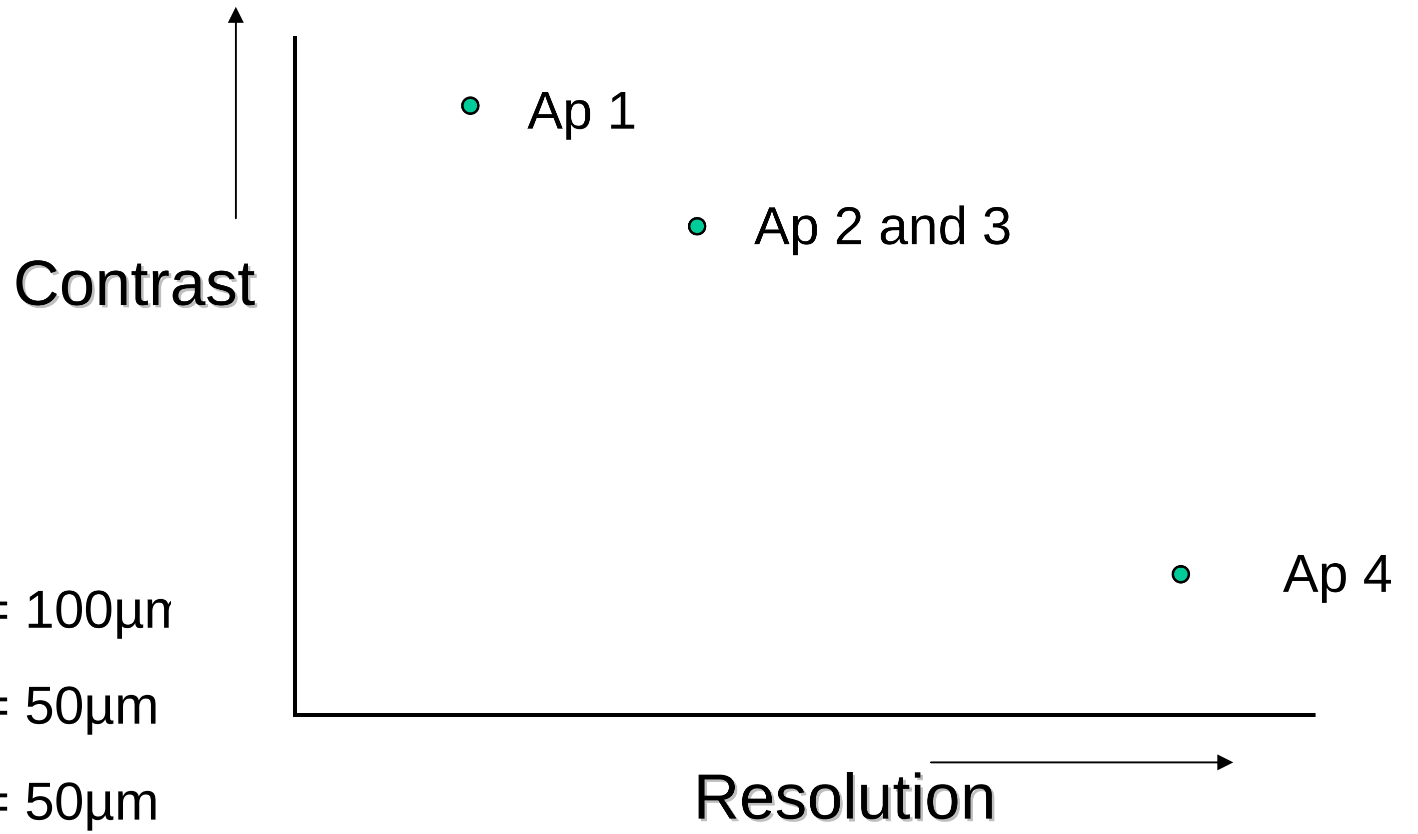
Snorkel lens field is projected down into the chamber, shielding against distortion and promoting High Efficiency collection

Beam Formation: Depth of Field



SEM Depth of Field ~500-1000 better than Optical microscope

The column – Objective Aperture



Ap 1 = 100µm

Ap 2 = 50µm

Ap 3 = 50µm

Ap 4 = 30µm

Beam Formation: Resolution limits

What limits SEM resolution?

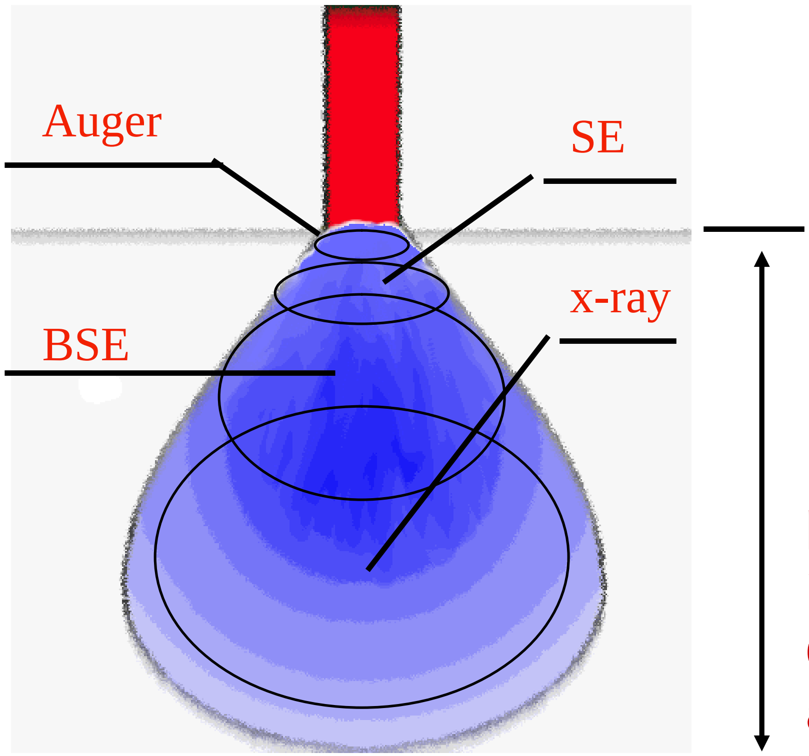
- The physics which may set the limit to the resolution of the image
- The instrument which may not be properly operated and optimised
- The operator who must use the microscope to its full extent

Beam-Sample Interactions

There is a wide variety of interactions which may occur between the beam electrons and the specimen. The exact interactions which occur are dependent on the sample material and beam conditions

- Secondary Electrons
- Backscattered Electrons
- X-Rays
- Auger Electrons
- Light - Cathodoluminescence
- Lattice Phonons / Heating
- Absorbed electrons or EBIC

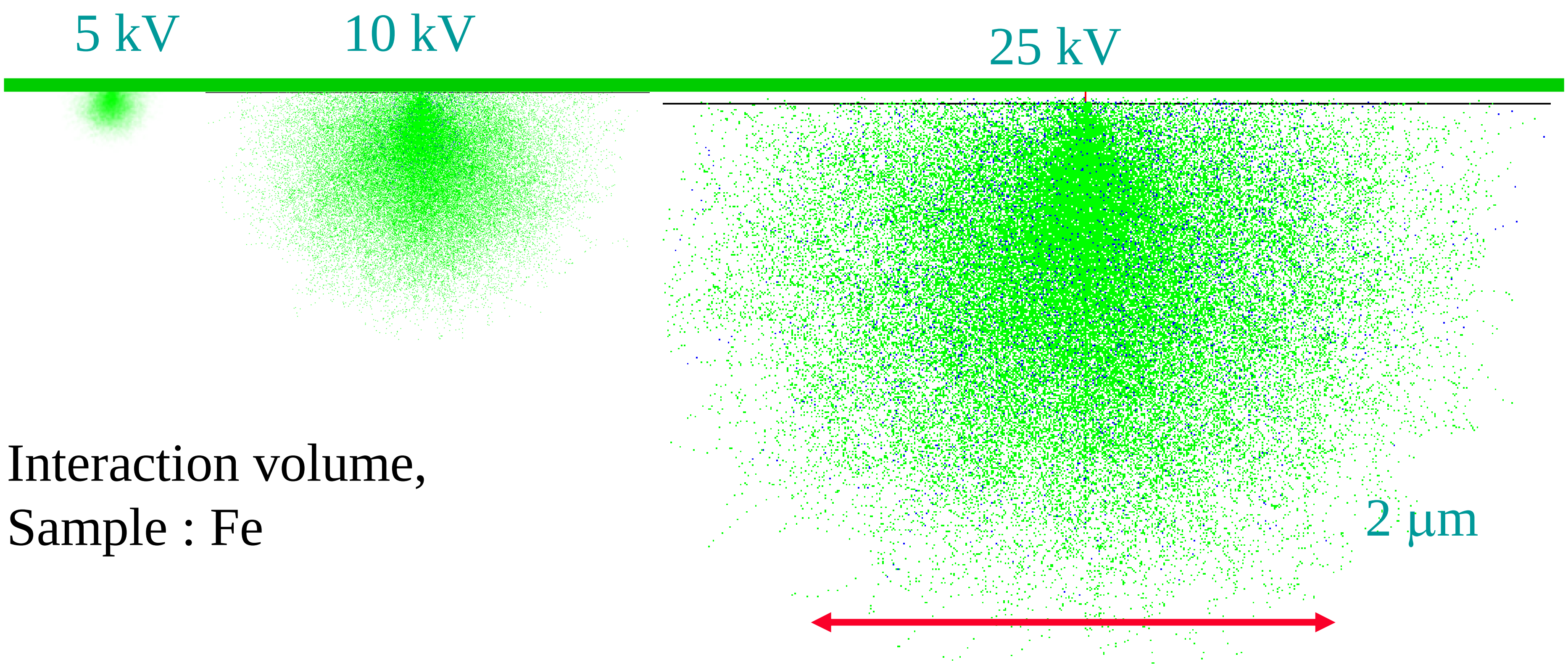
Beam-Sample Interactions: Interaction volume



Dependent on AccV.
As much as 5 um
in light elements
@ 30kV

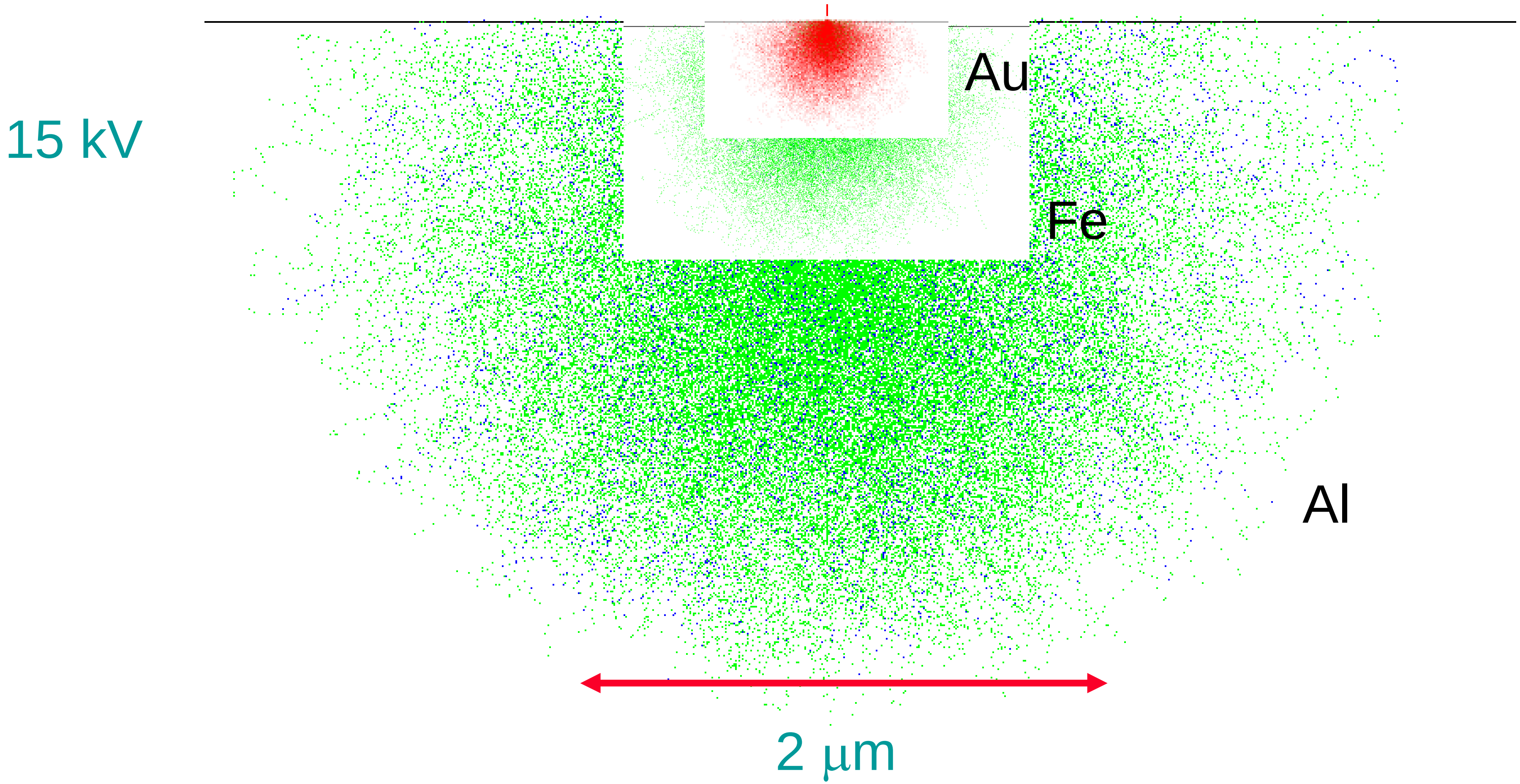
Depth of penetration = n microns, depending on kV and density of material

Beam-Sample Interactions: Examples of interaction volume

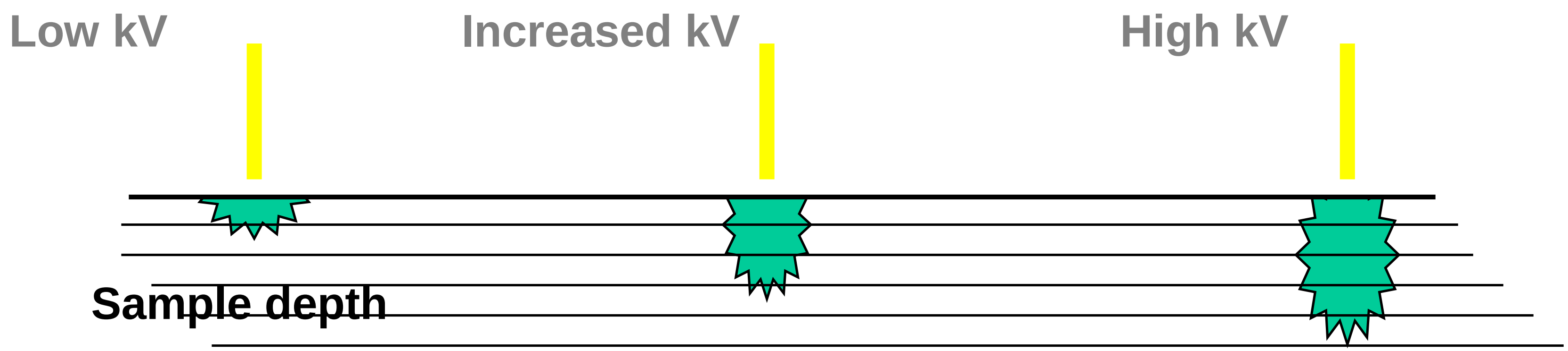


Interaction volume,
Sample : Fe

Beam-Sample Interactions: Interaction volume is density dependent



Beam-Sample Interactions: Interaction volume summary

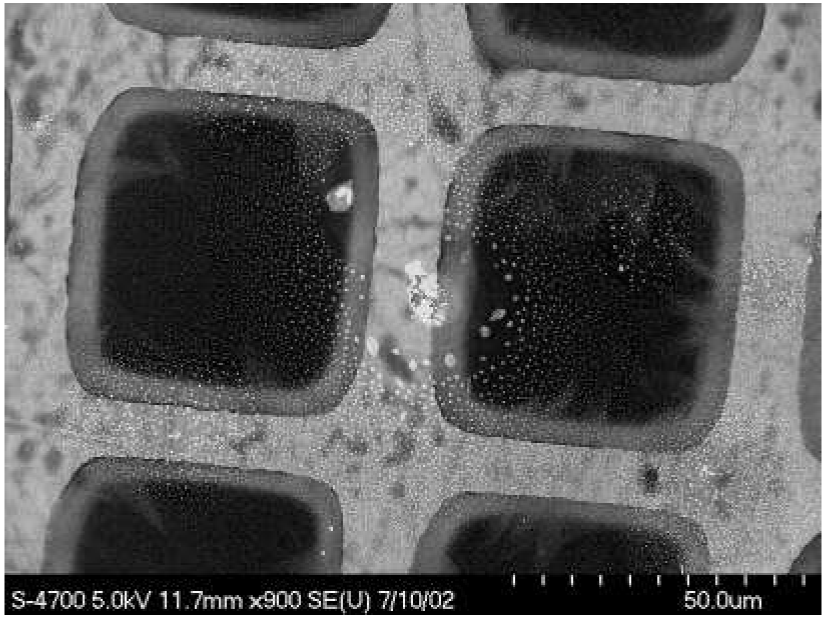


- Excellent surface info
- Difficult x-ray analysis
- Less resolution
- Good BSE info but very difficult to collect
- Very low damage to sample

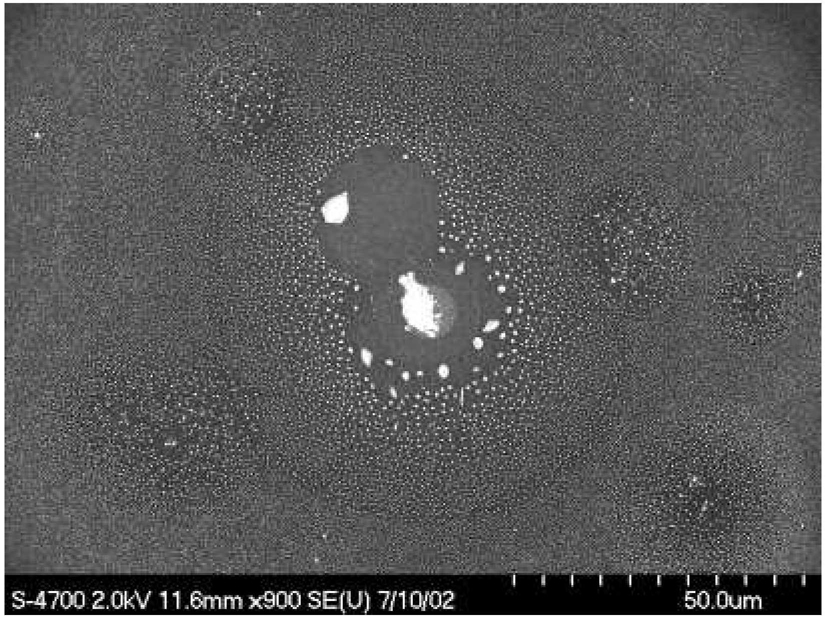
- Poor surface info
- Large x-ray content
- High resolution
- BSE from below the surface, but high energy ,easy to collect
- Possible damage

Beam-Sample Interactions: Interaction volume examples

Thin film on a TEM grid

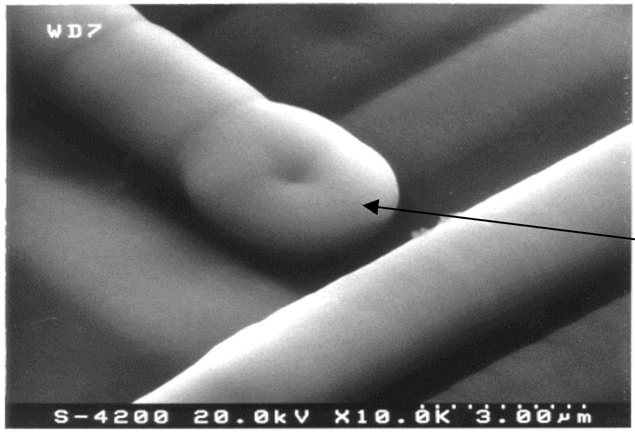


5kV mag x 900

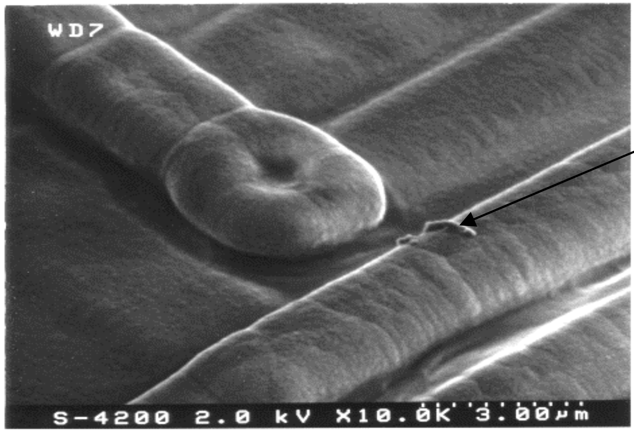


2kV mag x 900

Beam-Sample Interactions: Interaction volume



At 20 kV no discernible image information can be seen from the surface

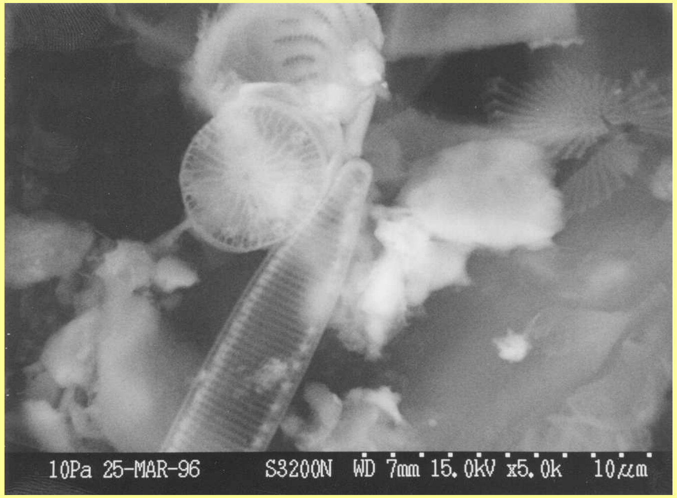


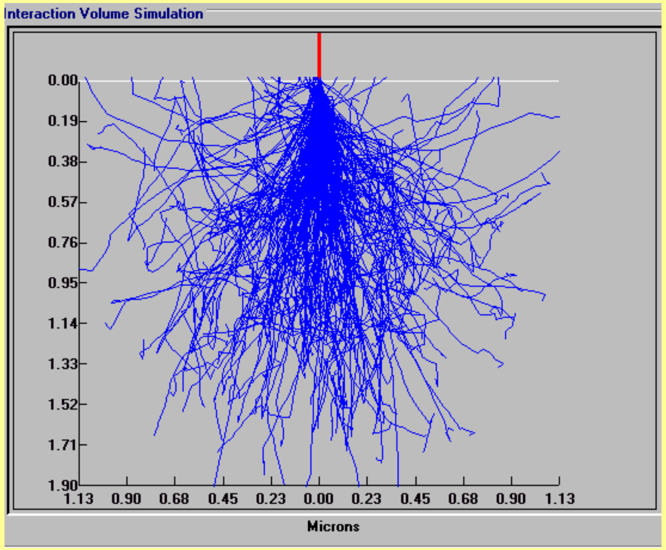
At 2kV a particle that was missed at 20 kv can now be seen and investigated.
The lower the kV the more surface information is detected

Significance of imaging at low acc. voltage

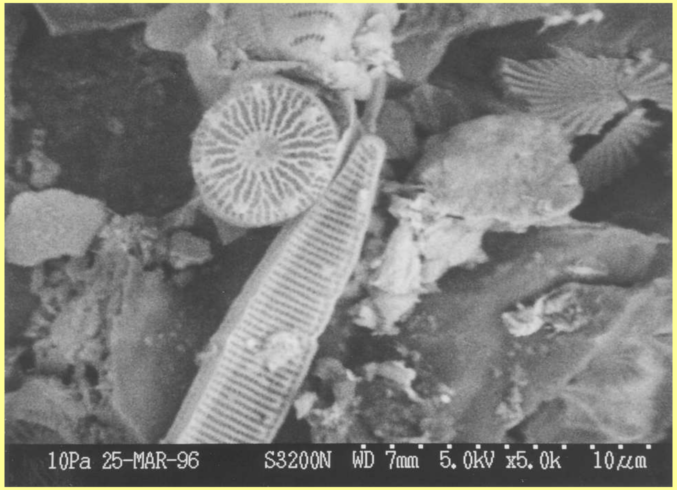
Sample : Diatom

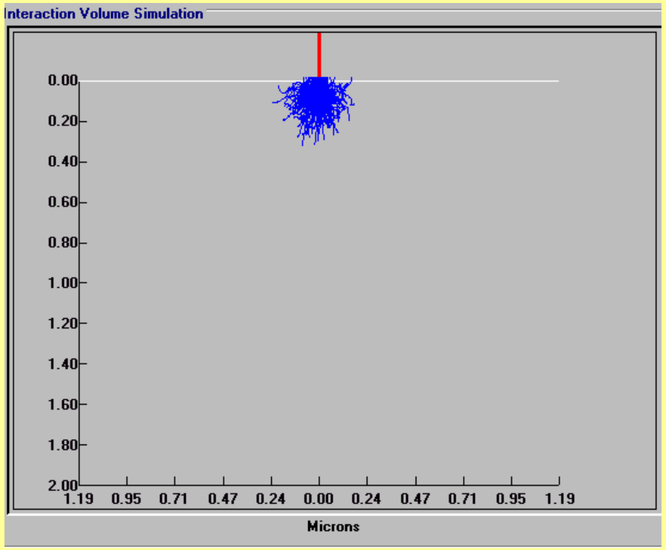
15kV



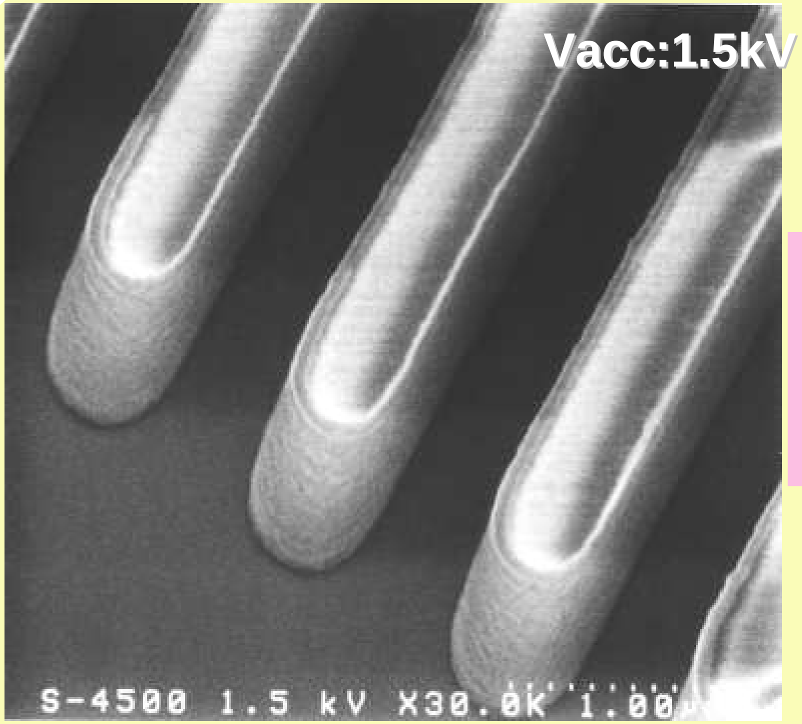


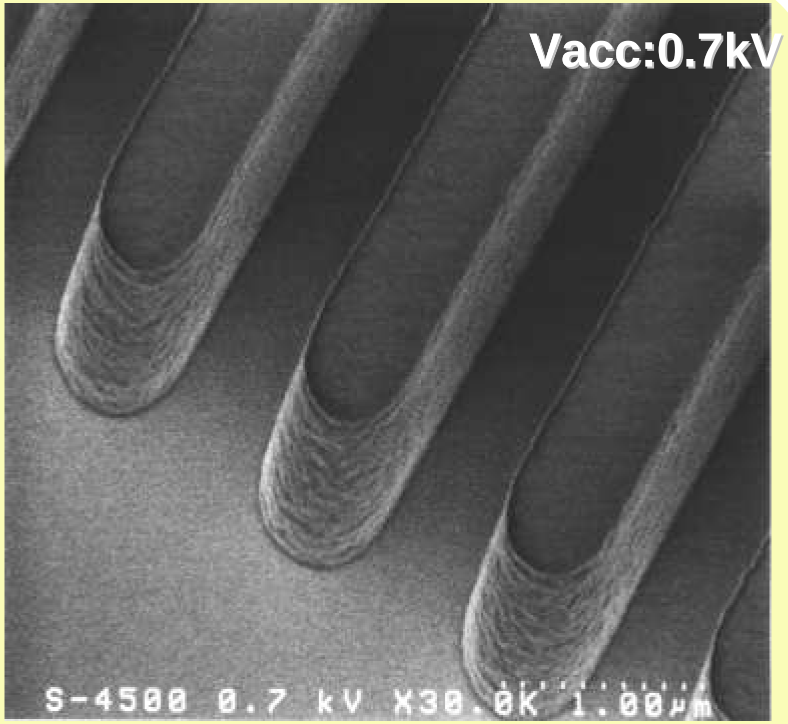
5kV





Observation at low accelerating voltages



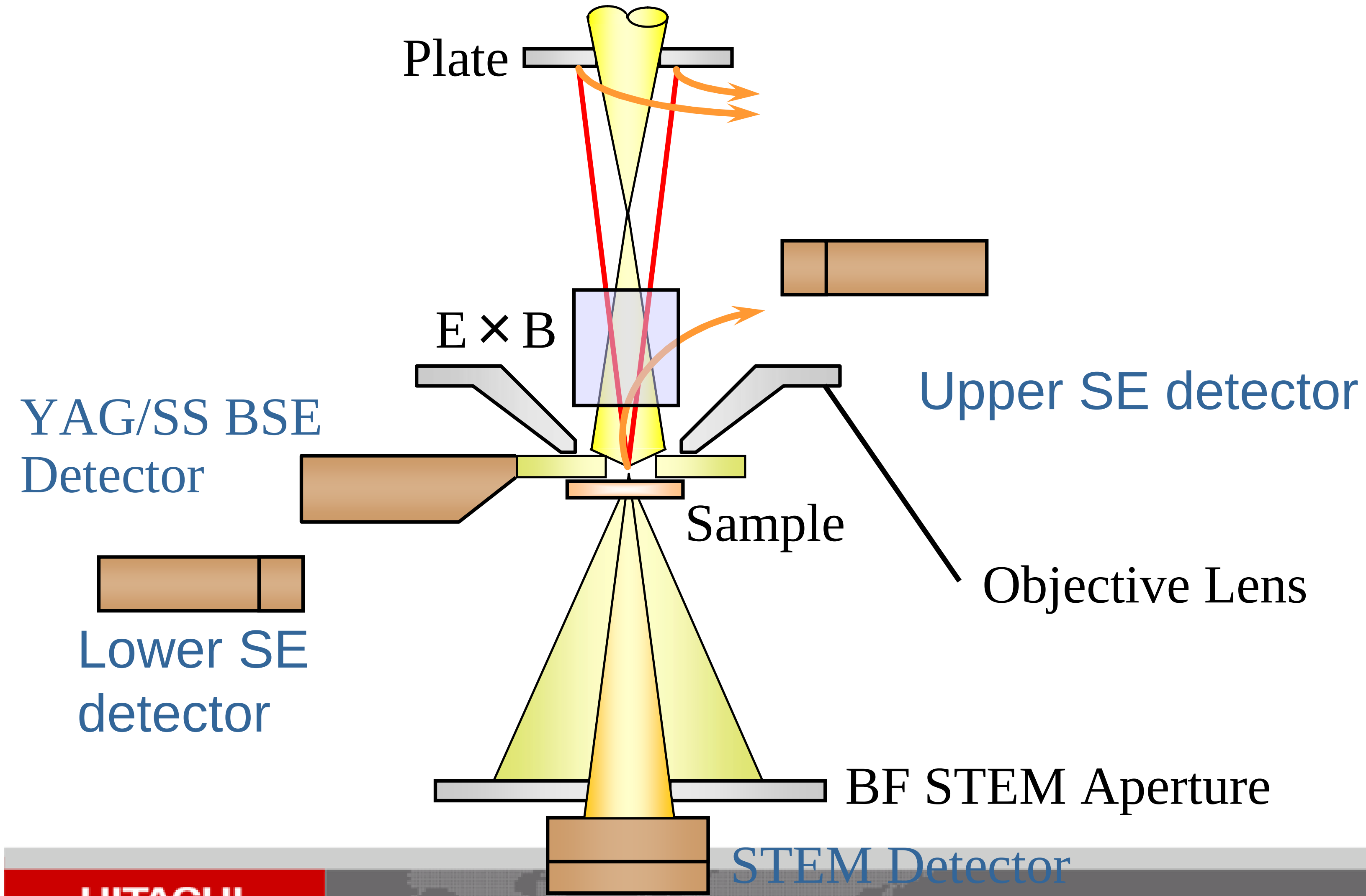


Charge-up phenomena

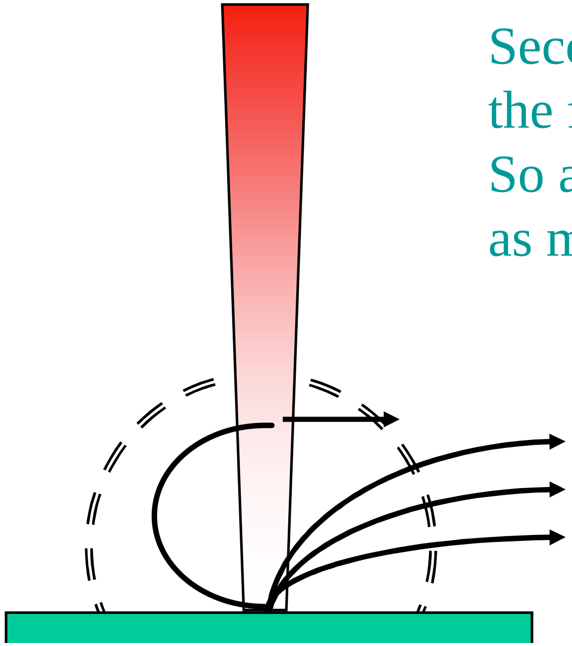
Eliminate Charge-up phenomena using lower kV

Specimen : SiO_2 on Photo Resist Line Pattern

Flexible detection system



Beam-Sample Interactions: Signal Collection, SE



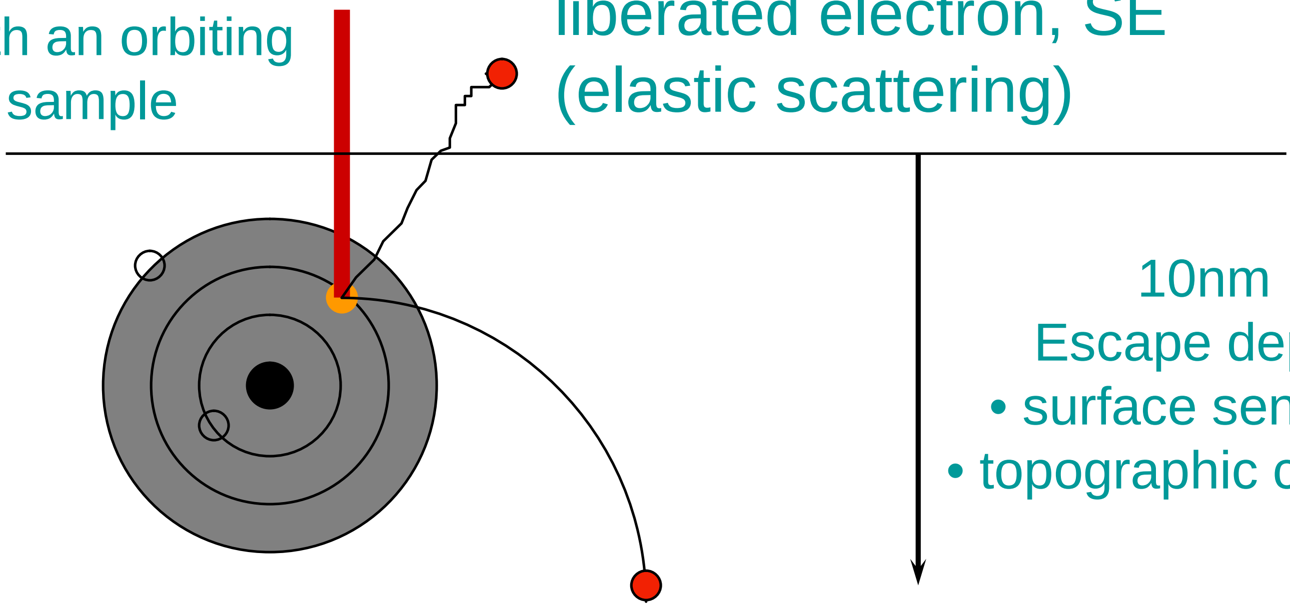
Secondary electrons can even be guided from the far side of the beam.
So a detector can be placed
as much as 150mm away from the sample.

SE Detector

Beam-Sample Interactions: Secondary Electrons

Probability,
Incident beam electron
will interact with an orbiting
electron in the sample

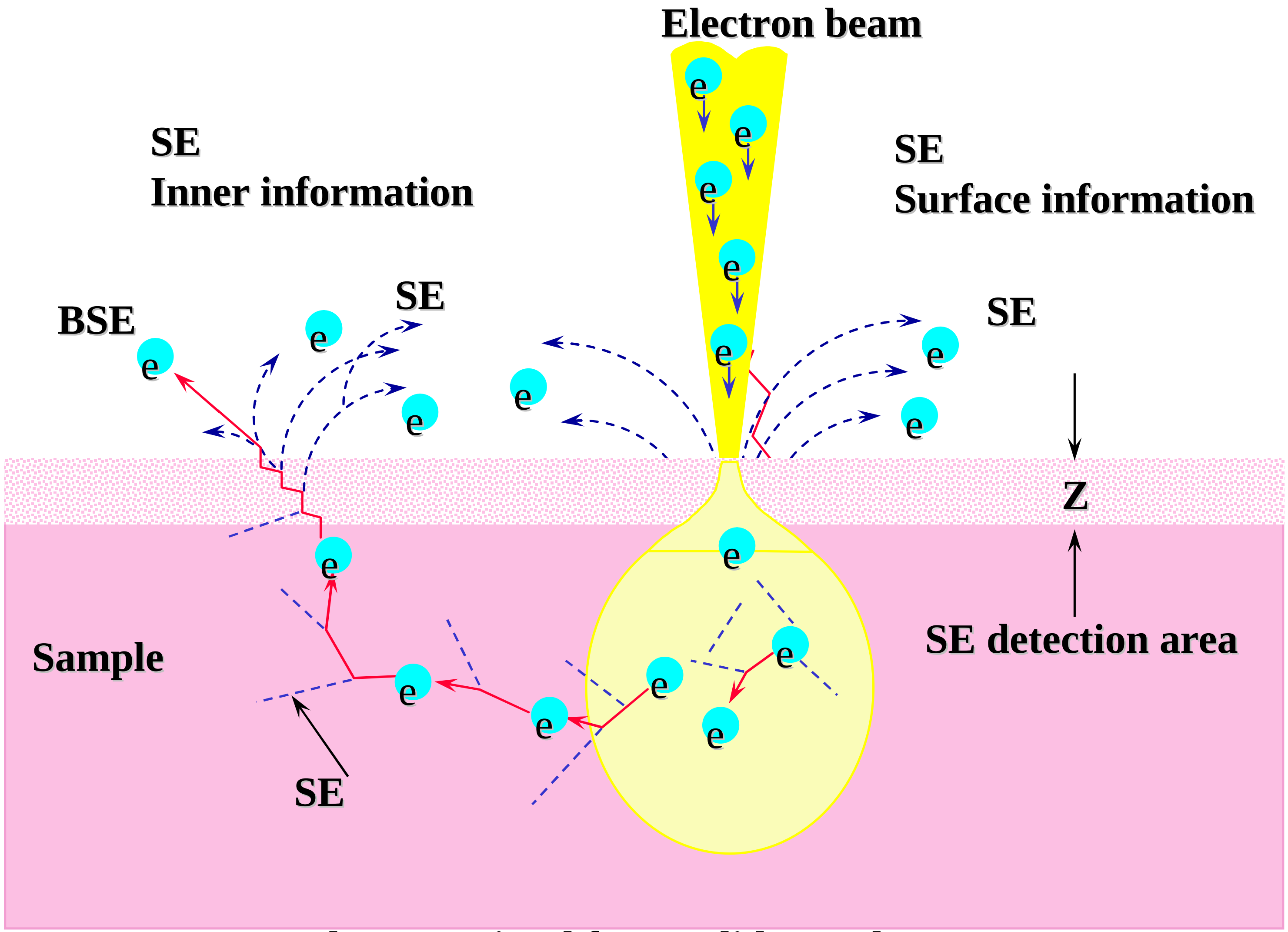
liberated electron, SE
(elastic scattering)



10nm
Escape depth:
• surface sensitive
• topographic contrast

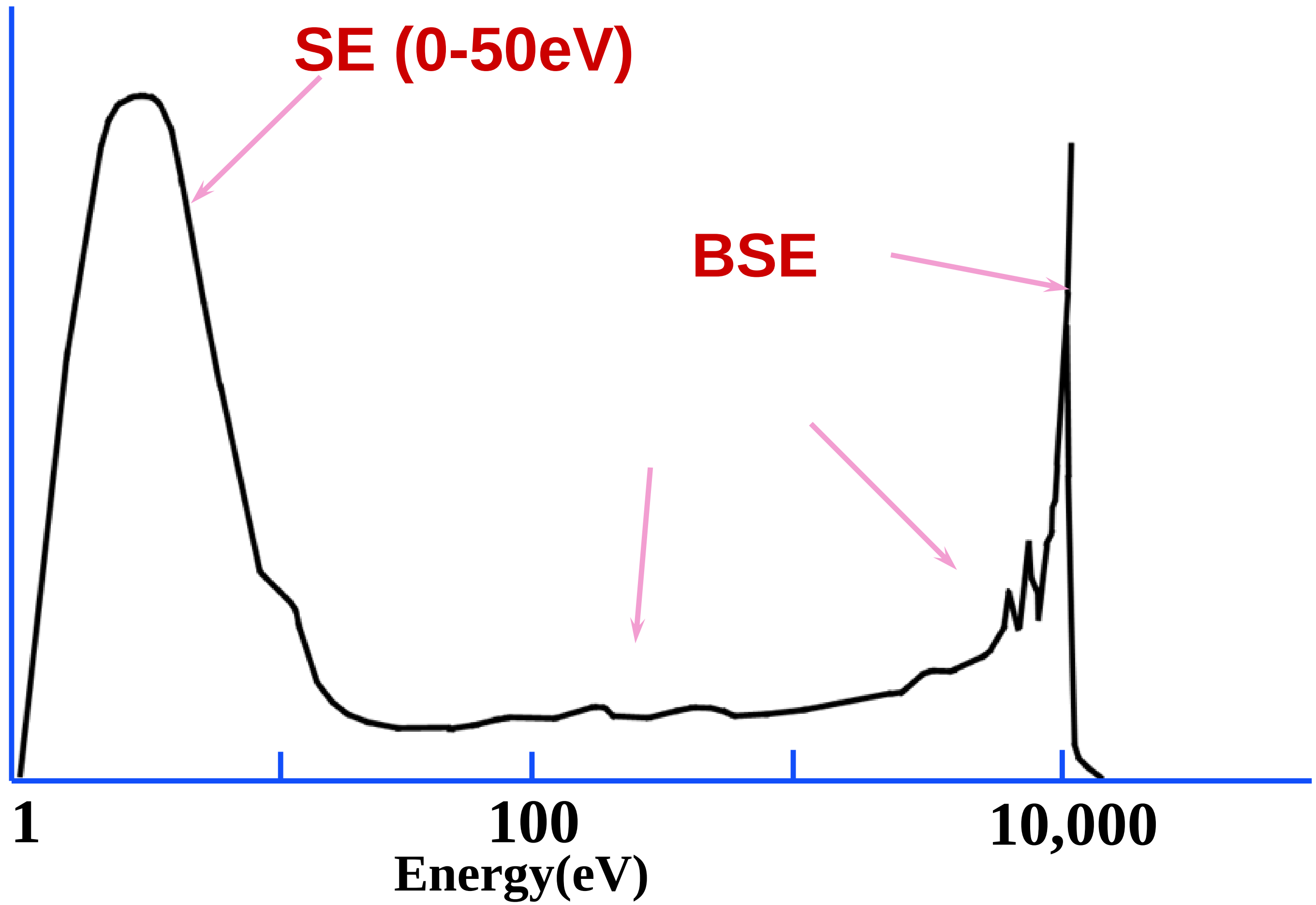
inelastic electron
scattered into the sample

Beam/Specimen Interactions

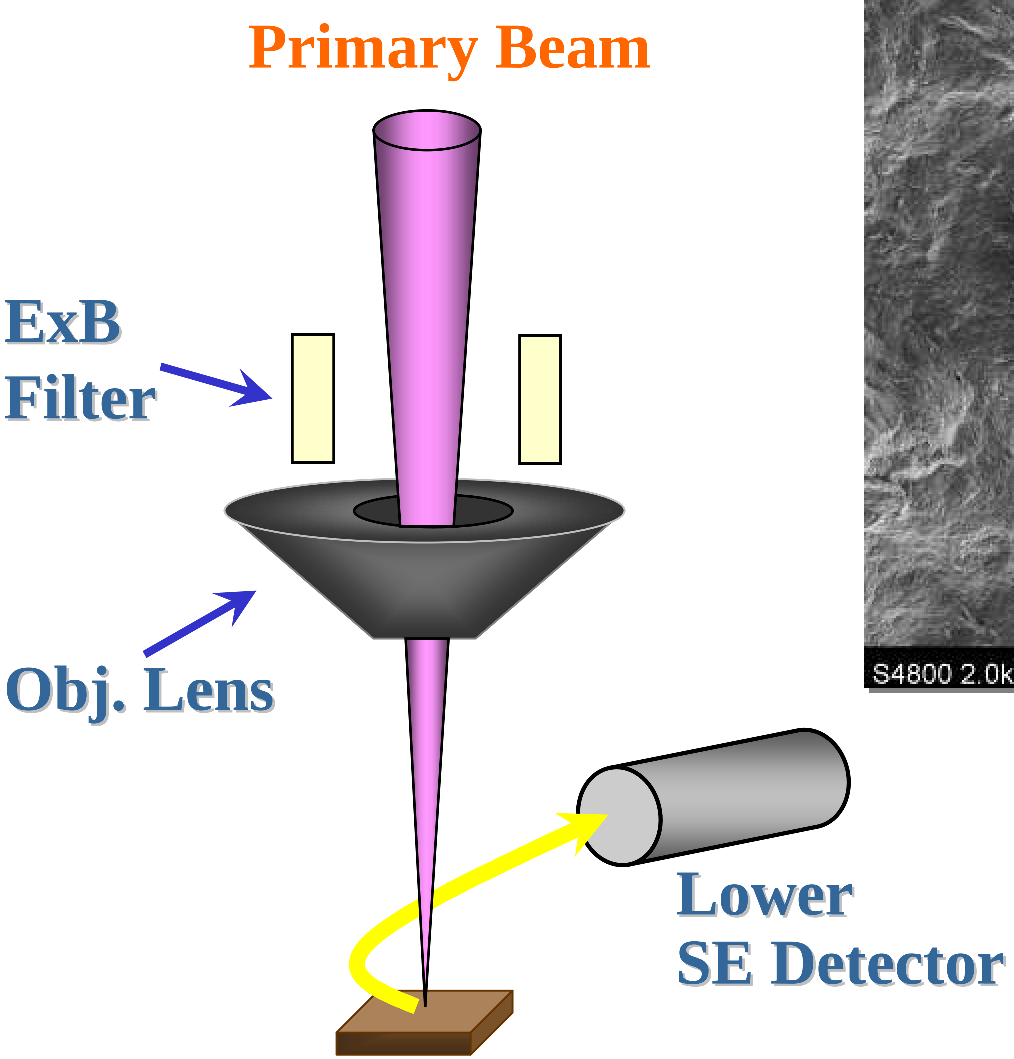


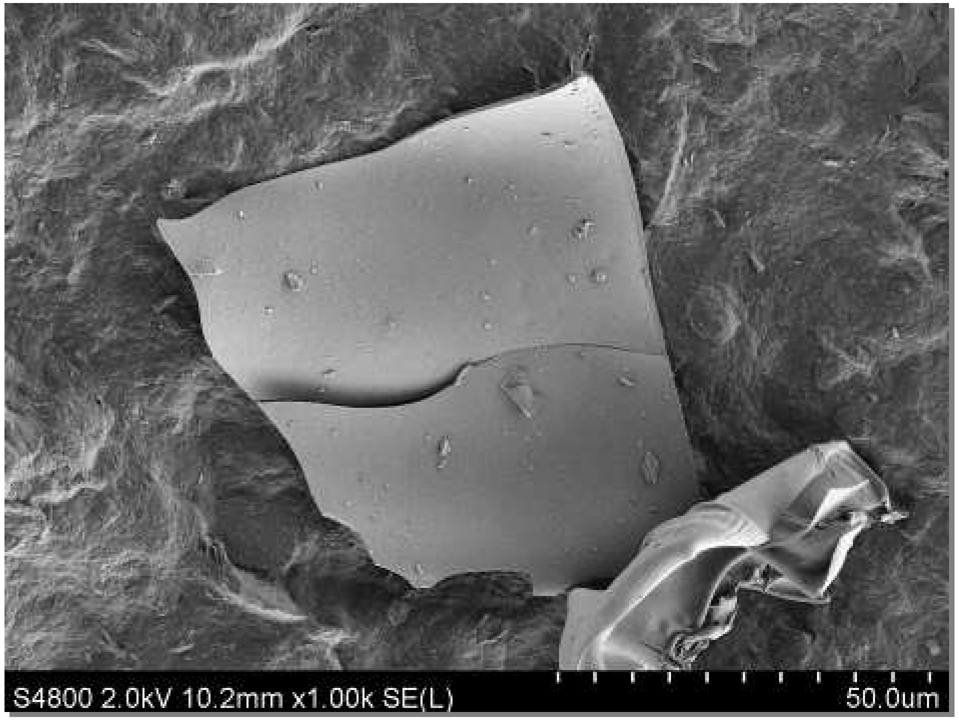
SE and BSE emitted from solid sample

Energy spectrum of the electrons emitted from a specimen



Lower SE Detector

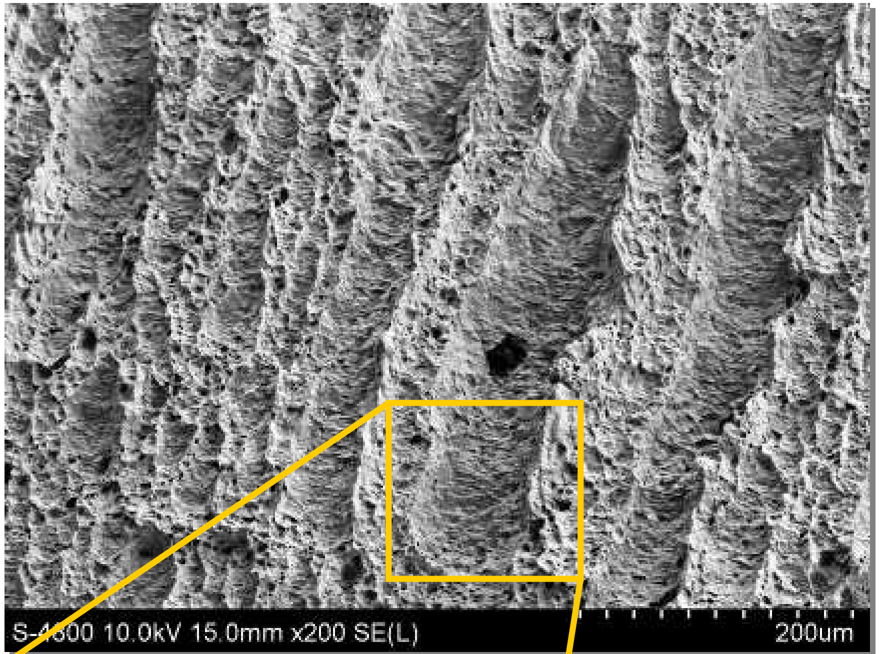


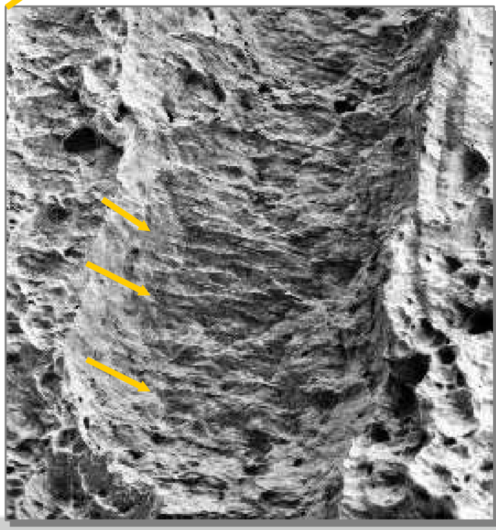


Secondary electrons collected in the chamber provide images with enhanced topographic effects, minimal charging, and good depth of field (SE+BSE)
High efficiency at long working distances required for analysis

Metal Fracture

The fracture pattern of this automotive part tells the story of how the failure happened. The peaks and valleys of the fracture surface are enhanced by the direction nature of the lower detector.



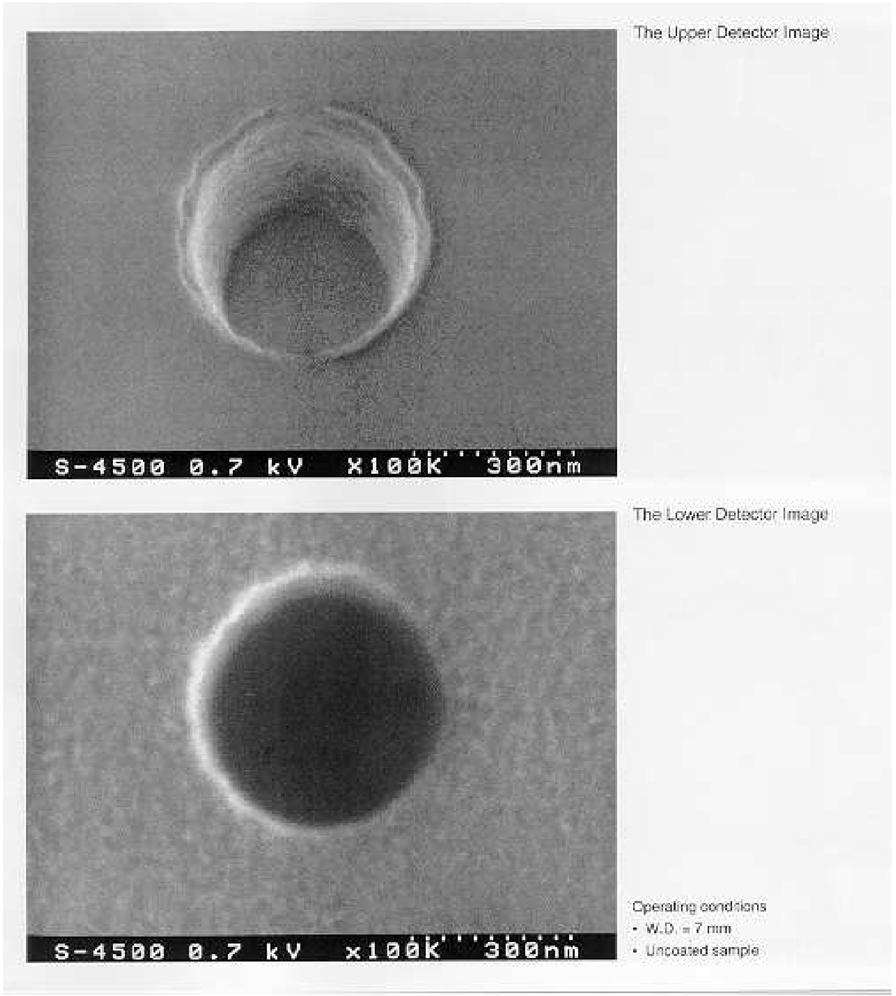


High signal to noise and and good resolution make the secondary fracture plane, in the horizontal direction, stand out even at low magnifications.

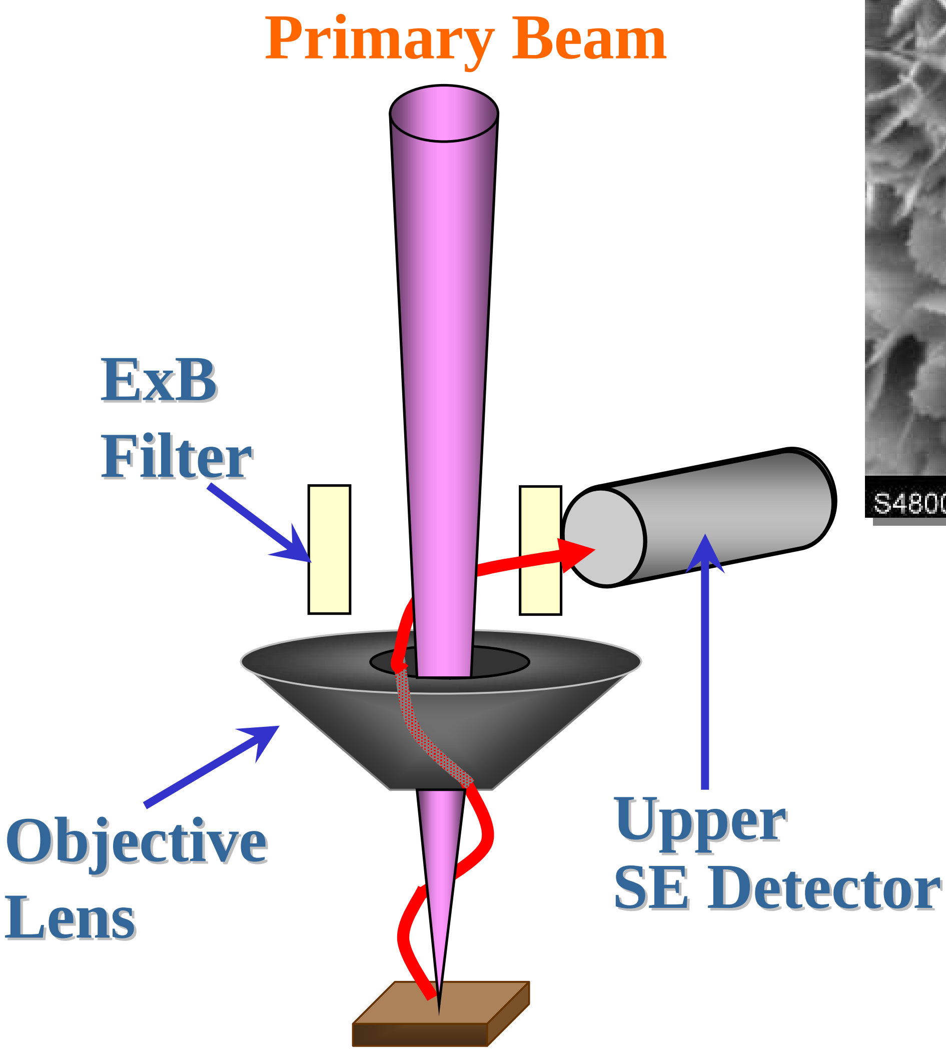
Comparison of Upper and Lower SE detectors

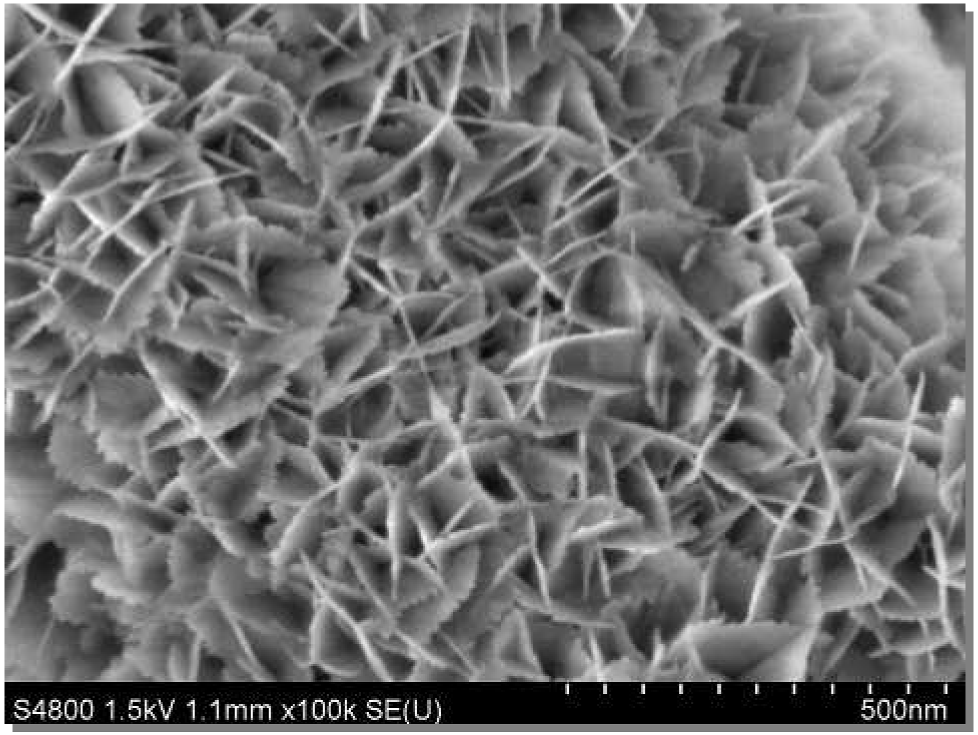
Upper SE detector is annular, positioned over sample (top down type of view)

Lower SE detector is offset, a standard Everhart Thornley configuration for directional contrast



Upper SE Detector





Secondary electrons are efficiently collected through-the-lens to provide images with extremely high resolution.

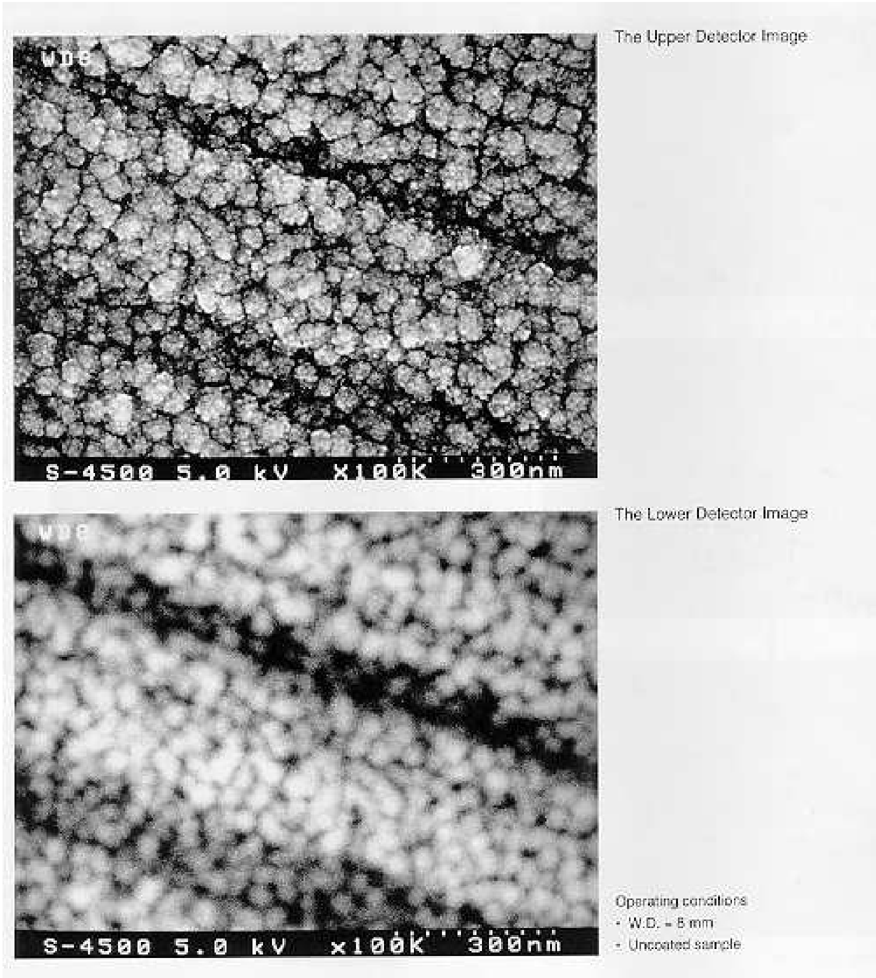
Three main modes of operation utilising the patented Super ExB filter:

1. Pure SE(I)/(II) or
2. SE+Low Angle BSE (Variable ratio) or
3. High Angle BSE

Comparison of Upper SE and Lower SE detectors

Upper SE annular detector for WD 1.5mm – 15mm

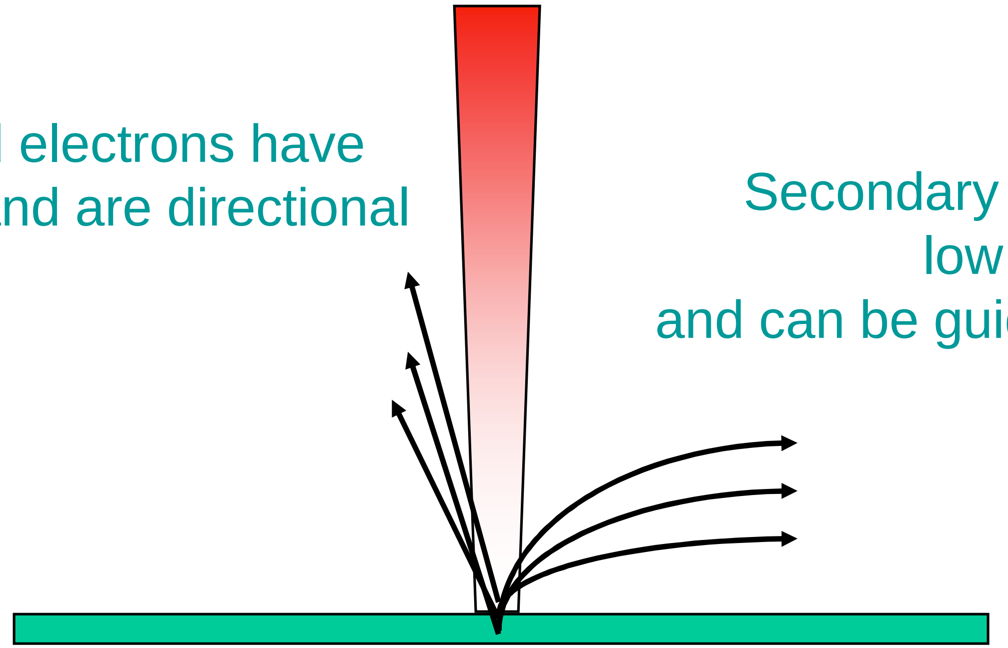
Lower SE detector for WD 7mm – 40mm



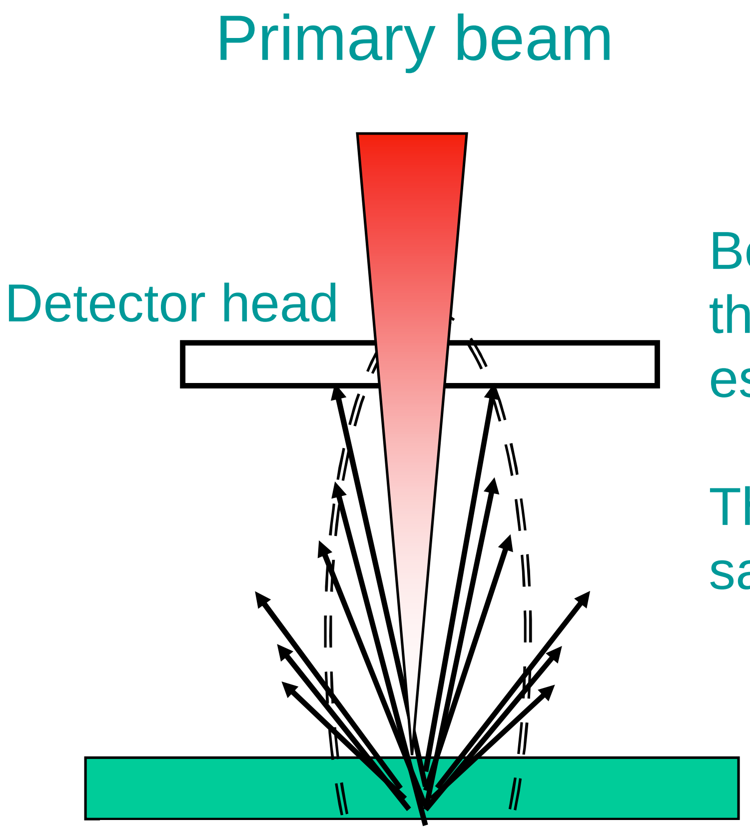
Beam-Sample Interactions: Signal Collection

Backscattered electrons have
high energies and are directional

Secondary electrons have
low energy
and can be guided to the detector



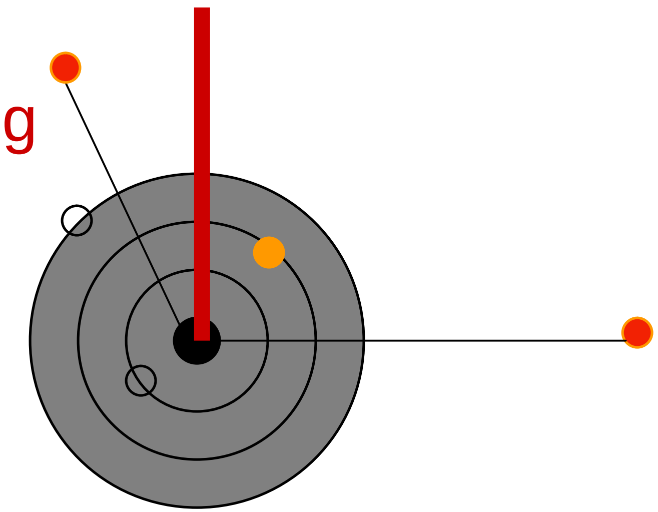
Beam-Sample Interactions: Signal Collection, BSE



Because Backscattered electrons are directional the detector must be placed in the path of the escaping electrons.

The detector must be placed very close to the sample to get the best results, 8-25mm.

Backscattered Electrons



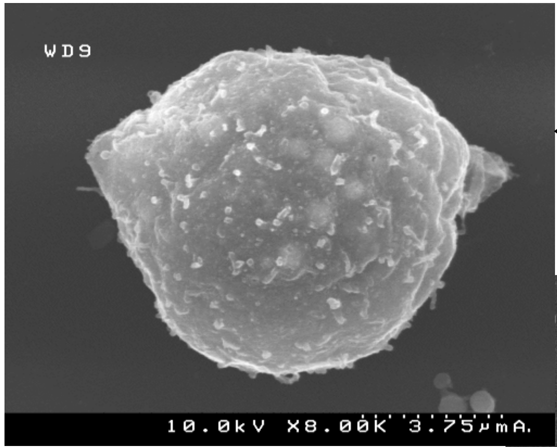
Nucleus of atom retards the incident beam energy and deflects the beam.
This retarded electron may go on to have many collisions with other atoms or electrons

The elastic scattered electron may be backscattered without loss of energy

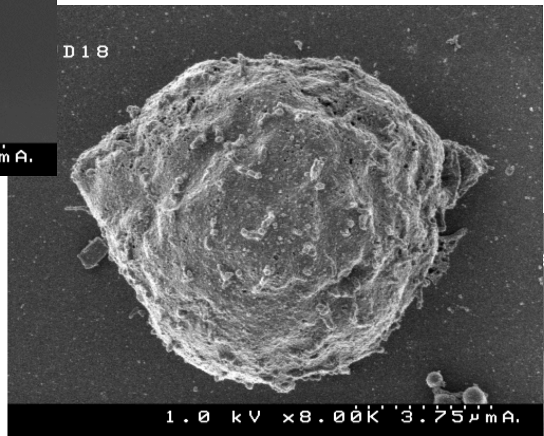
Inelastic scattering - electron may also have further collision events with other nuclei or orbiting electrons before breaking surface

Beam-Sample Interactions: Signal Collection

Image information from different detectors

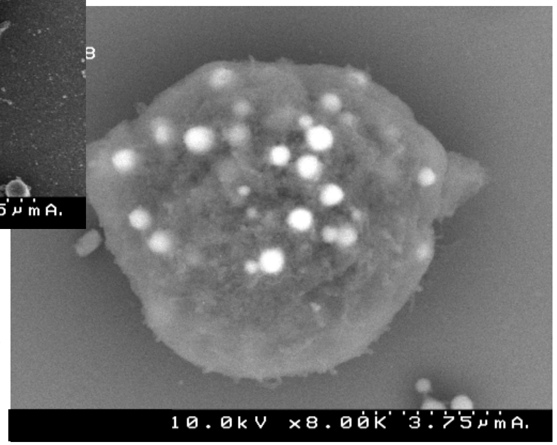


SE image at 10kV



SE image at 1kV

BSE image at 10kV



Beam-Sample Interactions: Signal Collection

SE detectors are of the scintillation type.
- Used with low and high AccV.

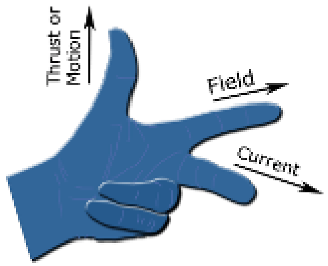
BSE detectors can be of scintillation type or solid state type.
- Normally used for kVs of 5kV plus.

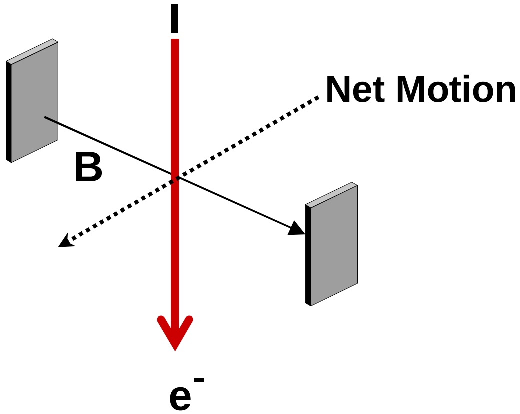
BSE electrons can be converted to SE's and collected using a secondary electron detector.
- Used for low AccV BSEs.

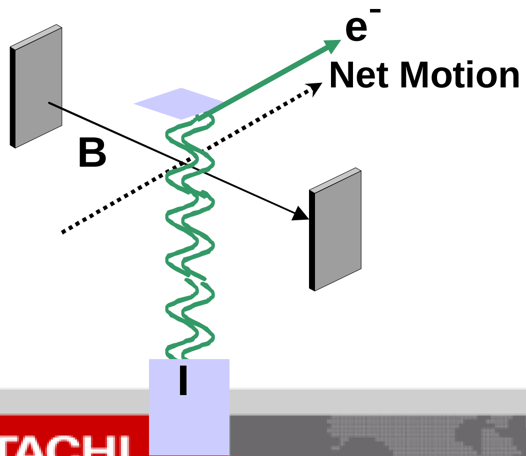
Low kV BSE's can be collected using a channel plate detector.

Hitachi's Patented ExB Filter

Fleming's Left Hand Rule







Upper SE detector

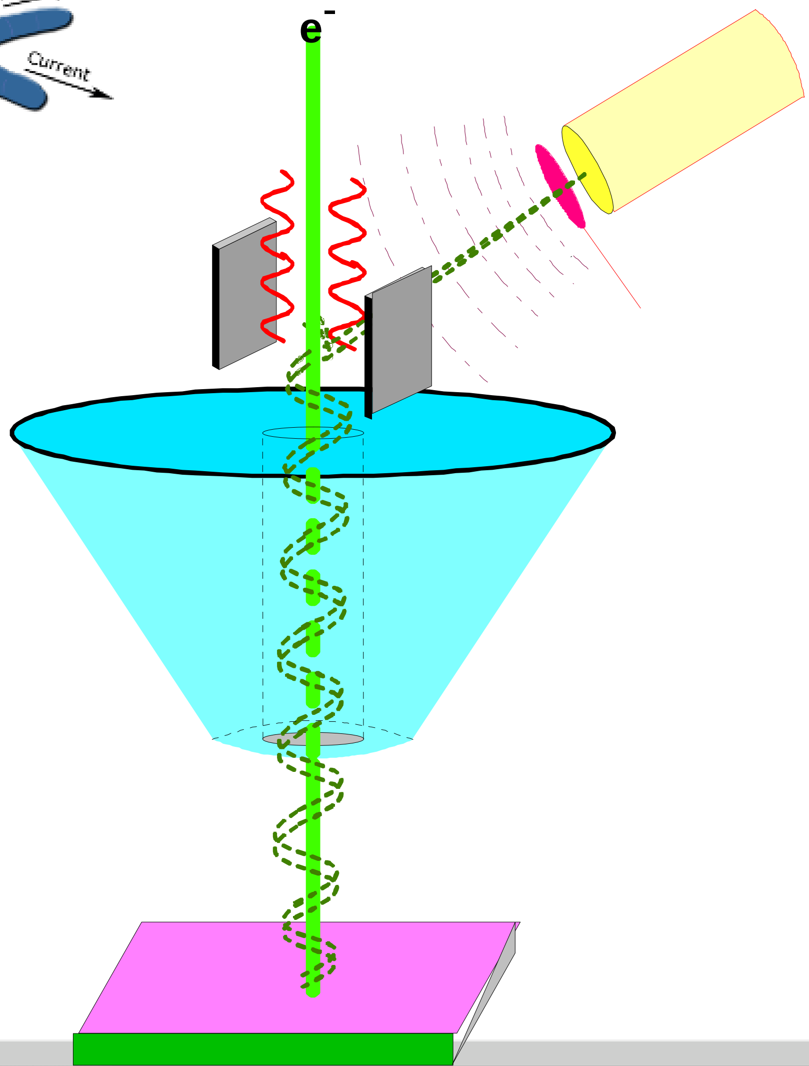
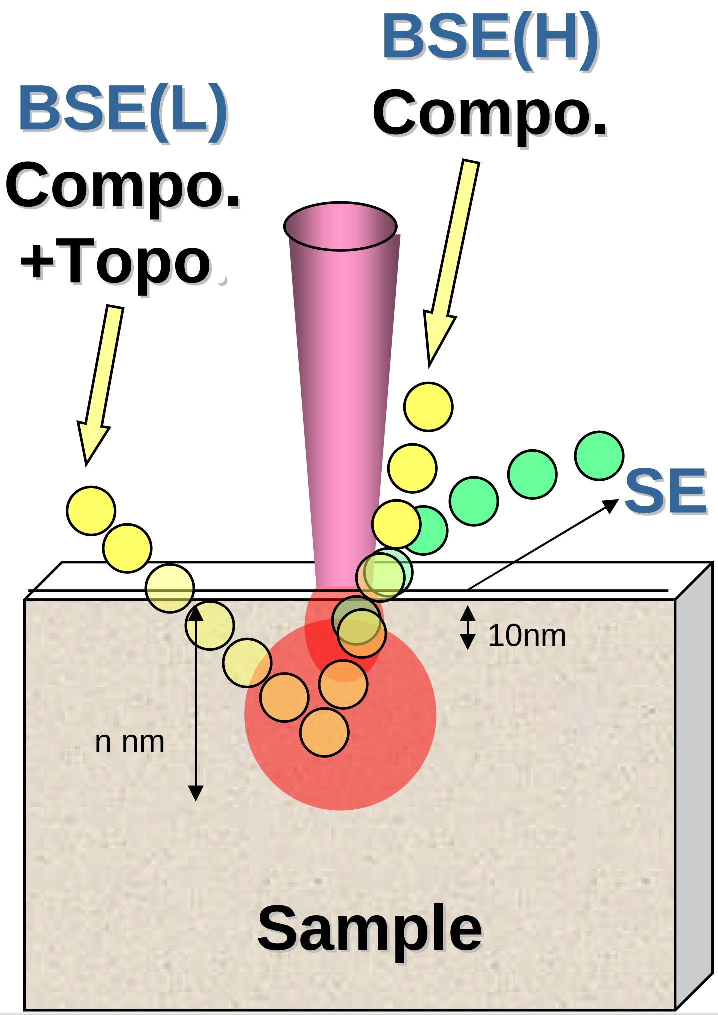


Image information: SE & BSE



Secondary Electrons

Topmost surface information

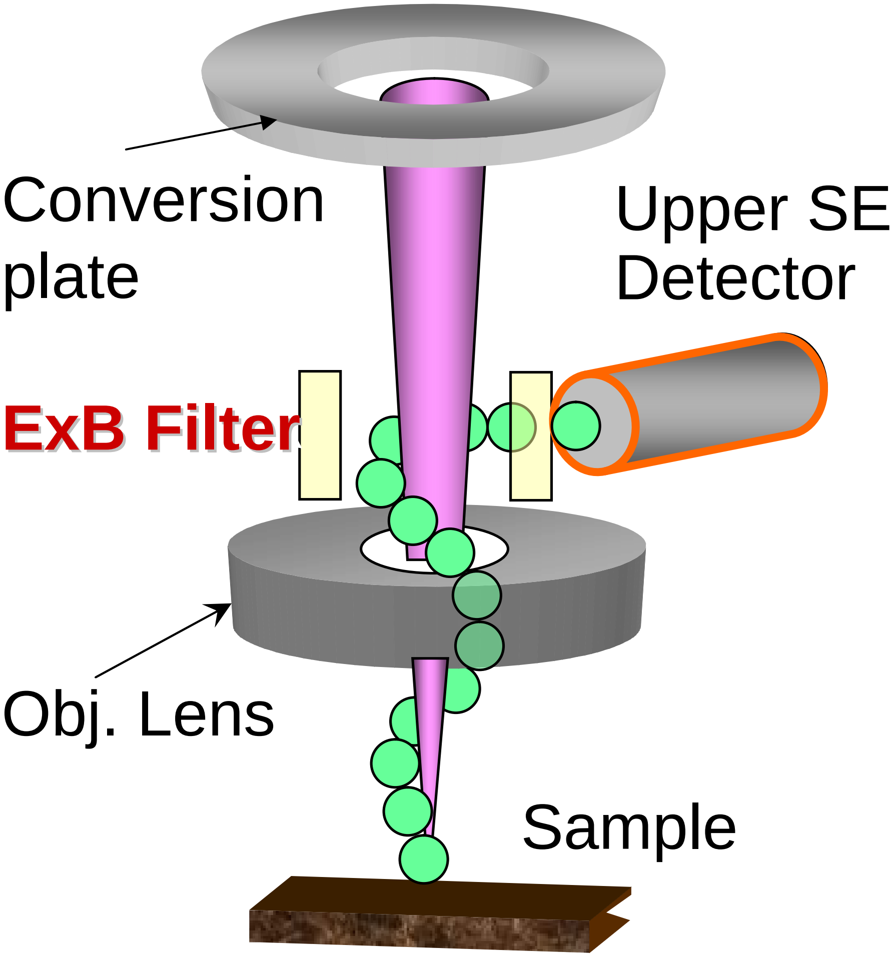
- Electric Potential contrast
- Energy of Electron : <60eV

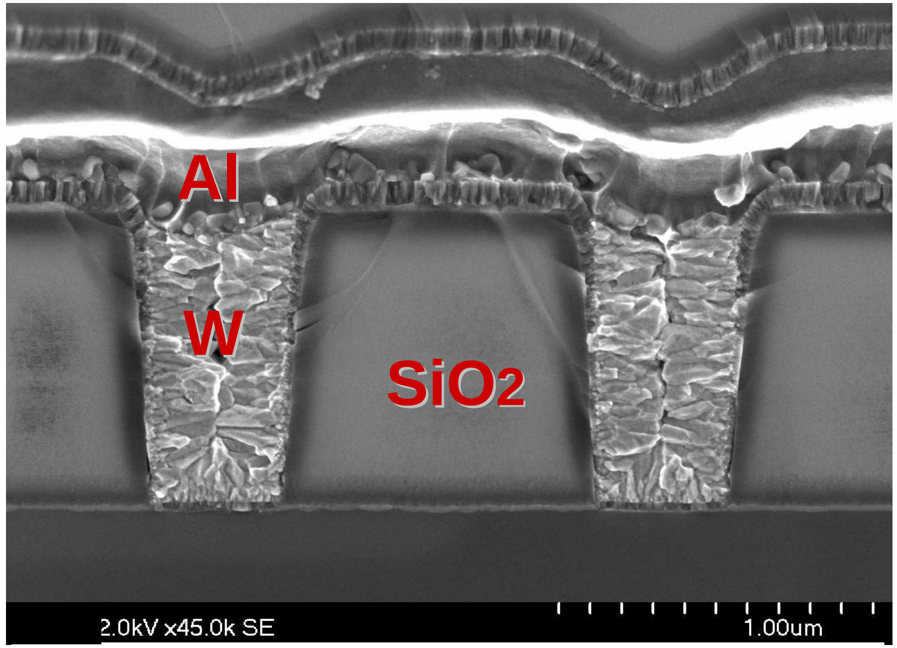
Backscattered electrons

Composite information

- Less charge up
- Less edge contrast
- Topography (Low angle BSE)
- Energy of Electron ～ incident beam

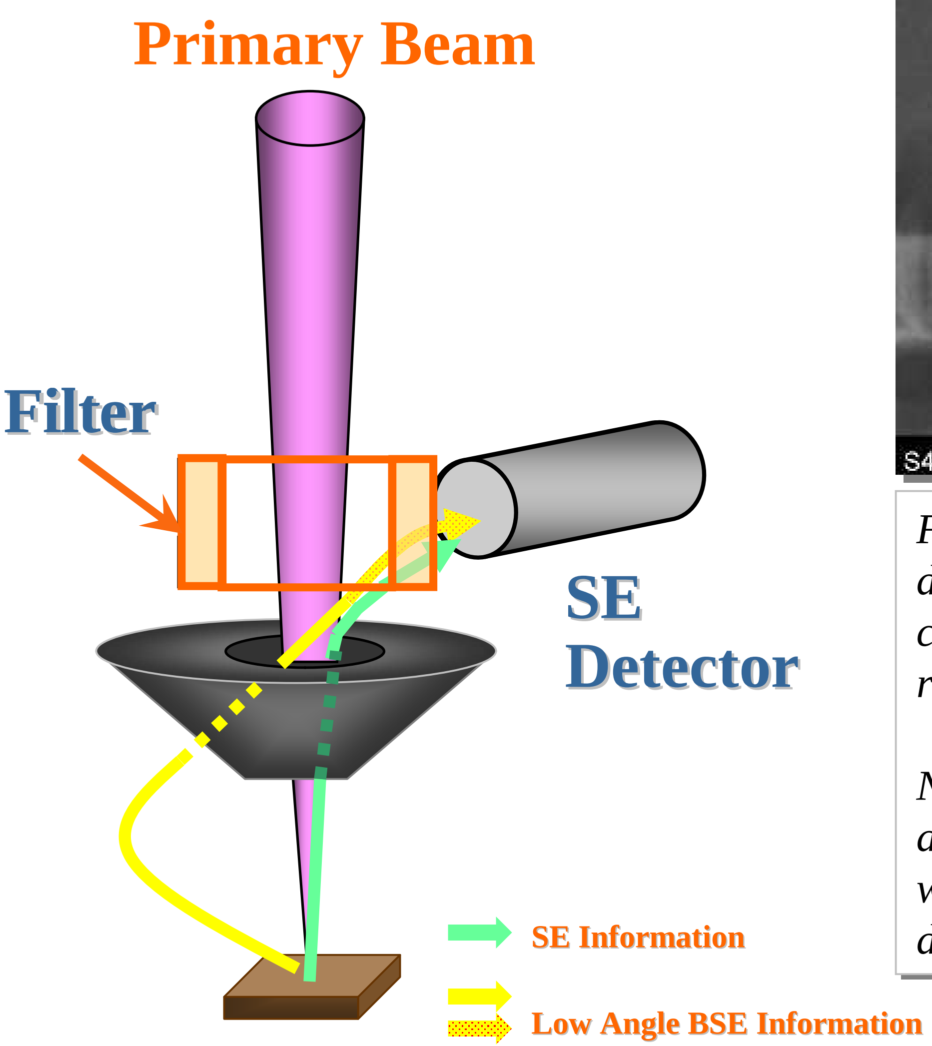
Pure SE information using the ExB filter (no BSE)

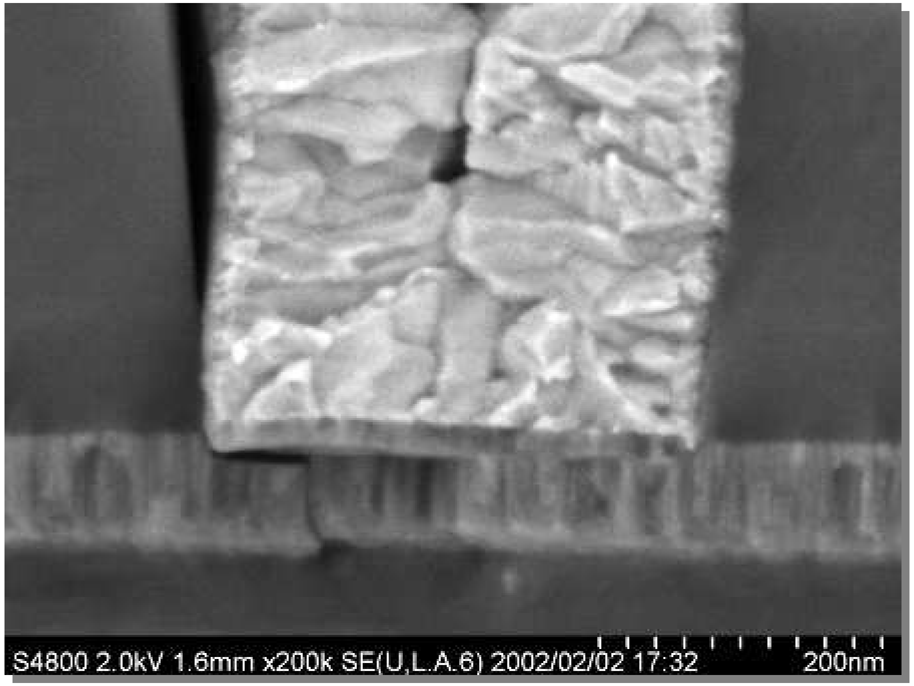




Topographic contrast

SE + BSE Information

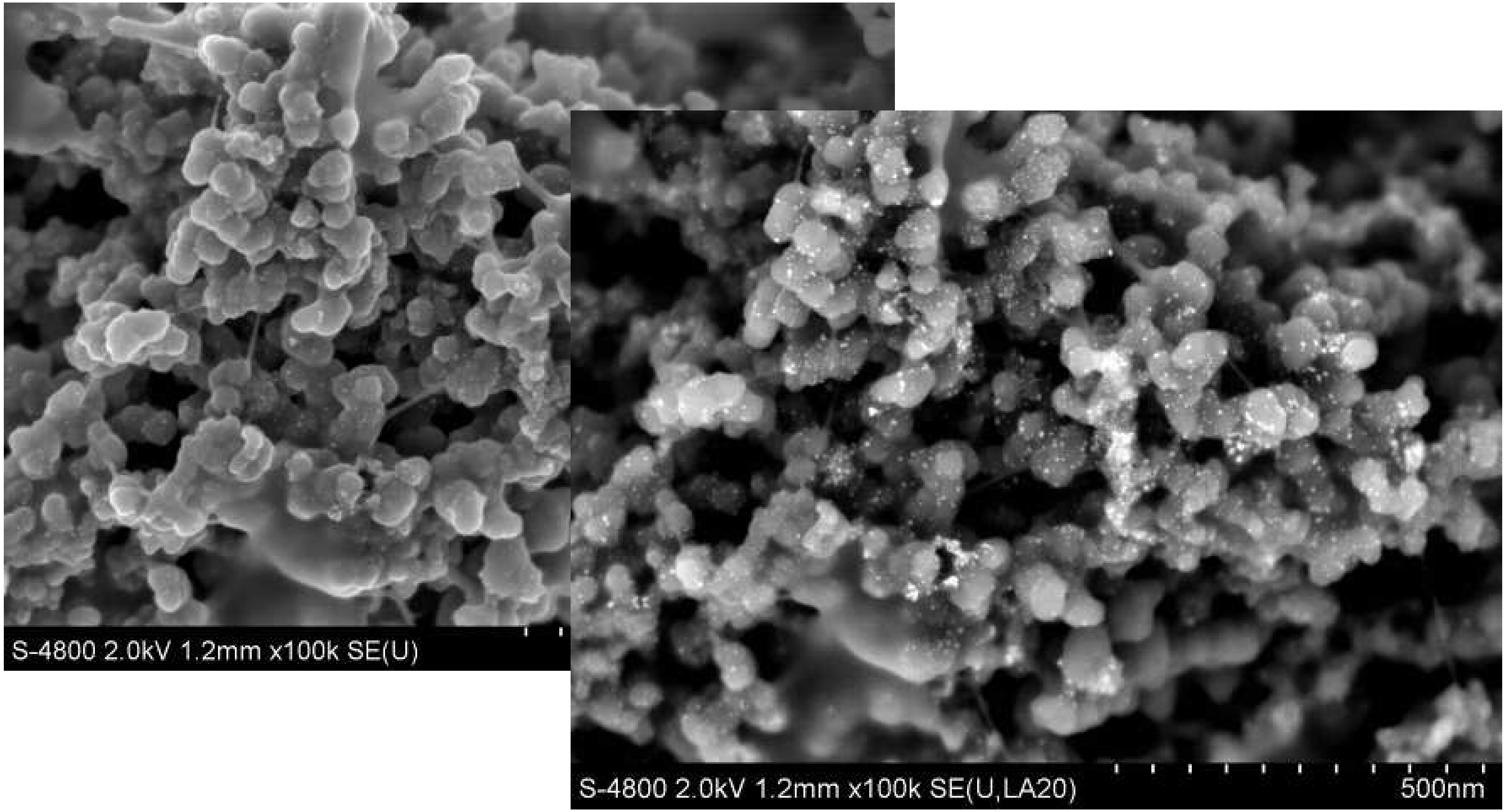




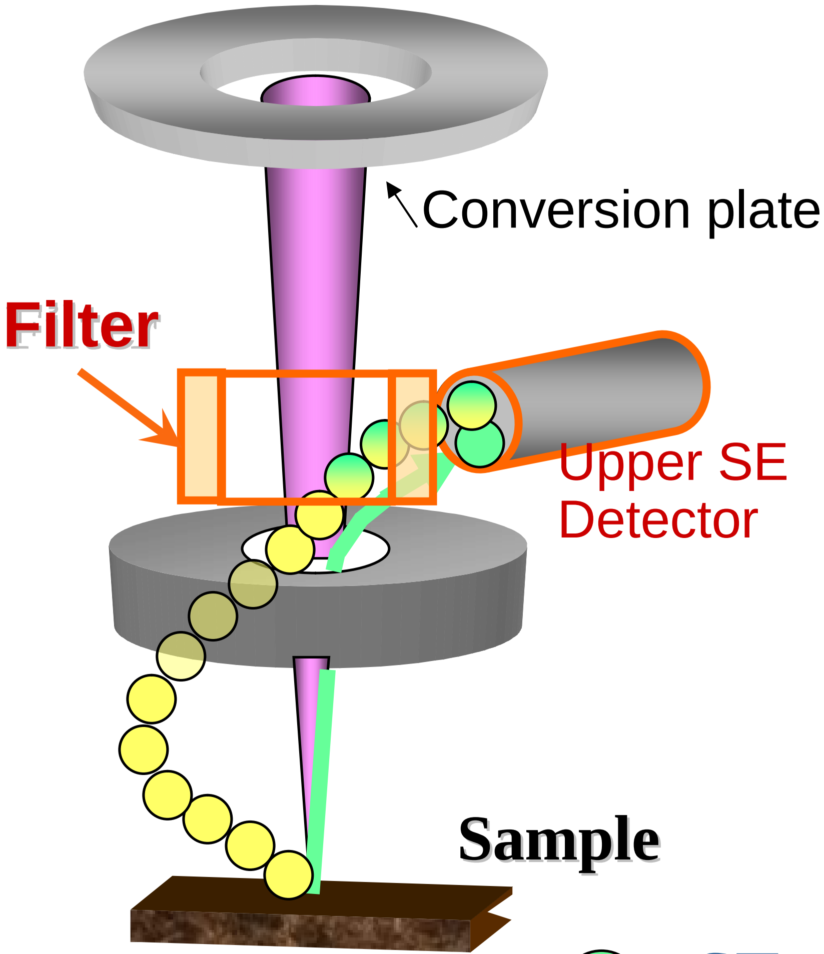
Filtering SE and BSE information to the upper detector provides images with compositional contrast, surface detail, reduced charging, and reduced contamination.

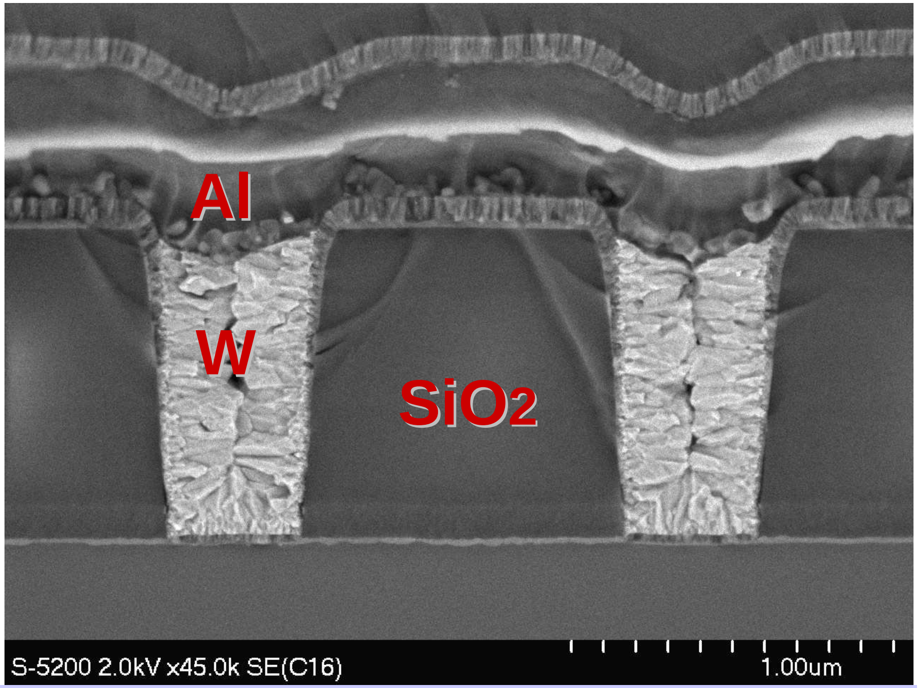
N.B. Enables BSE imaging even at very low accelerating voltages which were not possible with conventional scintillator or solid state BSE detectors (e.g. BSE imaging at 500V)

Catalyst (Pt/C)



SE + BSE(L) Information using ExB filter





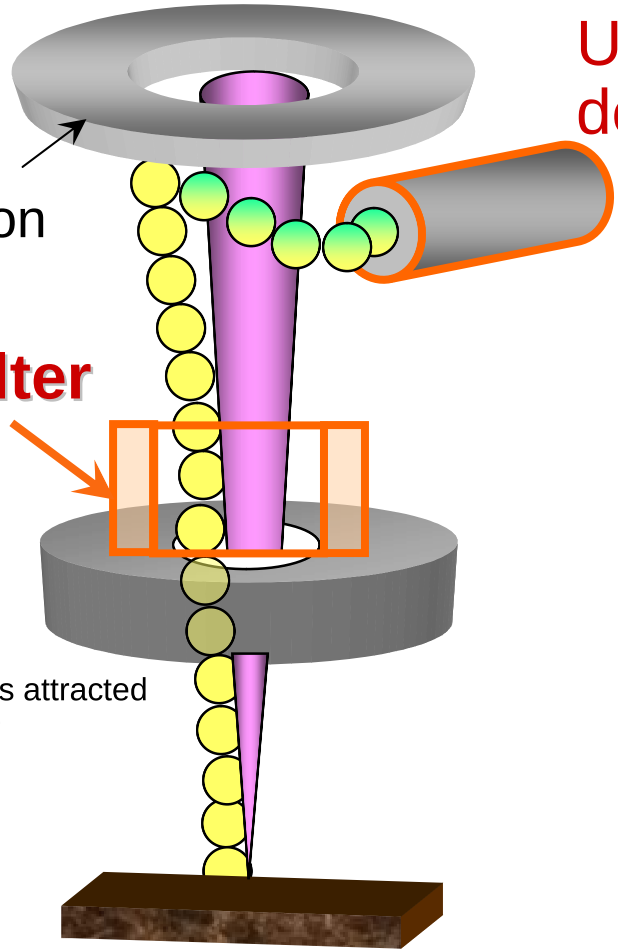
Topographical contrast +

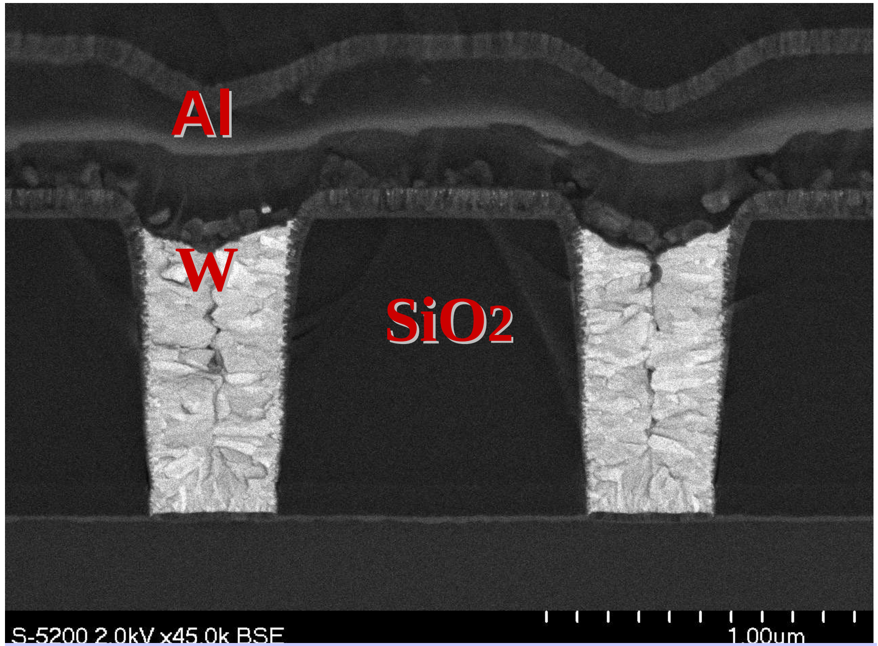
Compositional Contrast

: SE　: BSE(L)

: SE generated by BSE(L)

BSE(H) Information only (no SE)



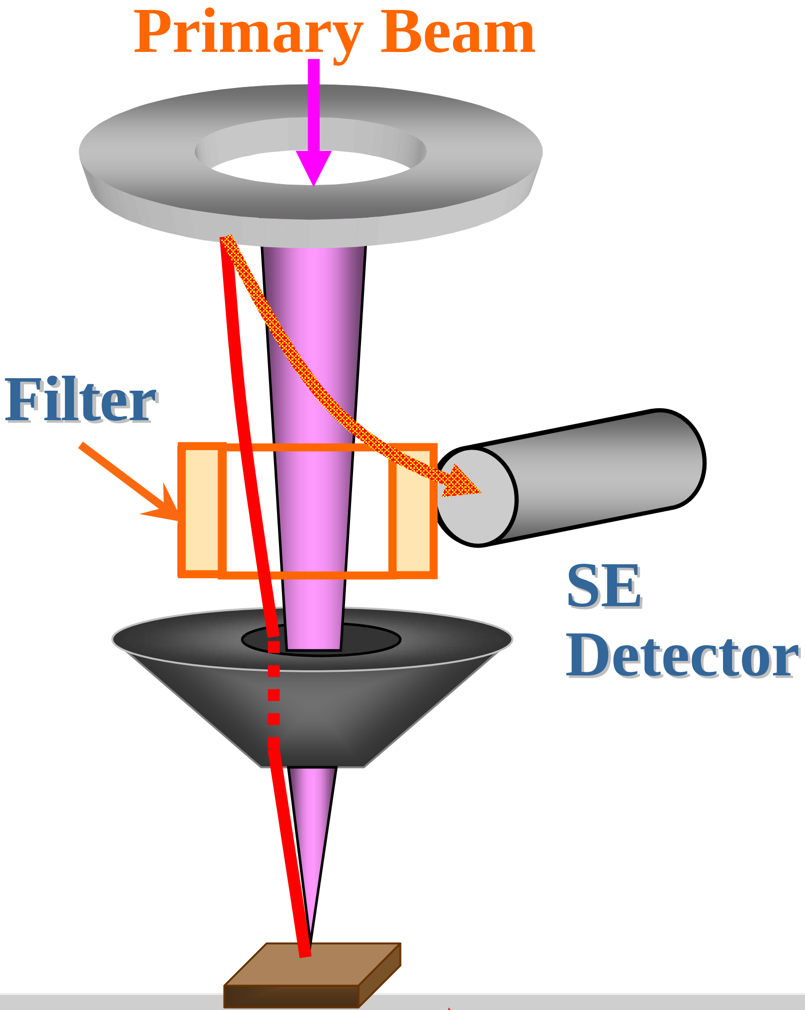


Compositional contrast

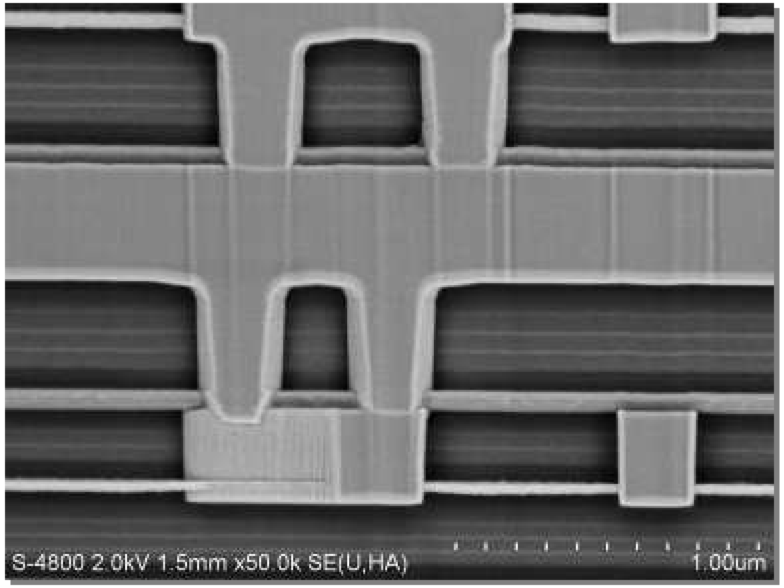
: BSE(H)

: SE generated by BSE(H)

BSE Information



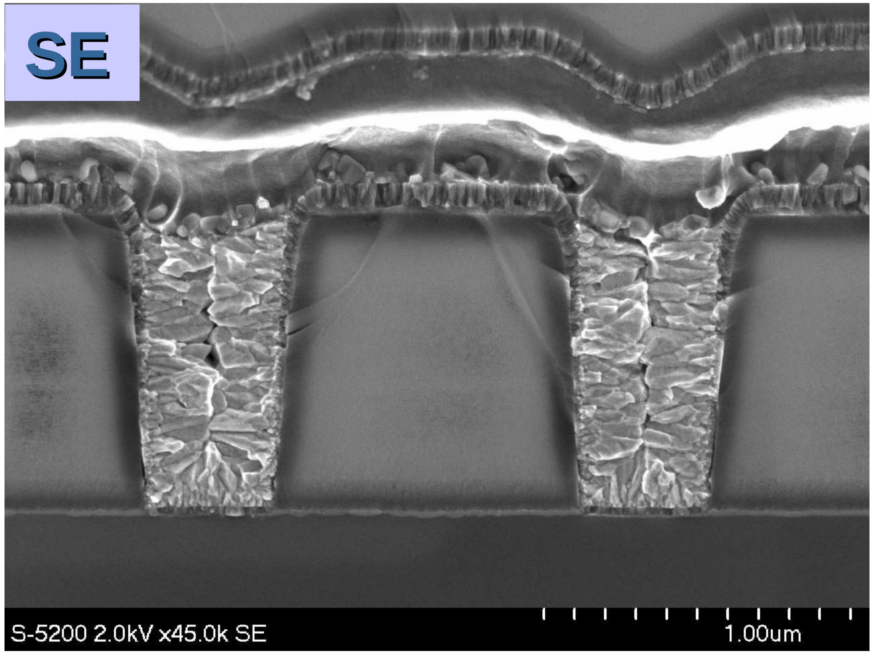
High Angle BSE Information

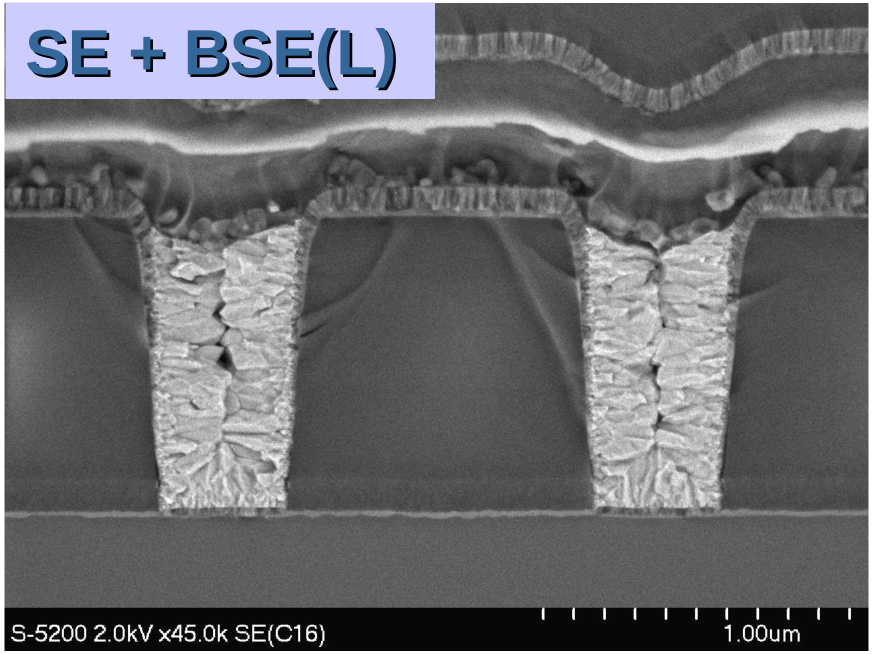


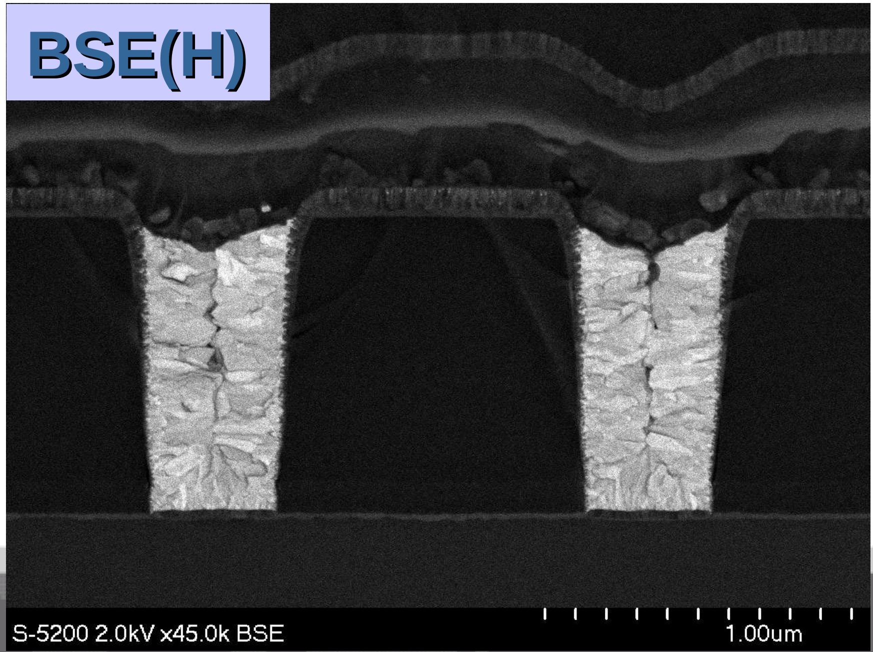
High angle backscattered electrons produce strong compositional contrast.

High energy backscattered electrons strike a conversion plate in the SEM column. This event generates secondary electrons that are then collected by the upper SE detector.

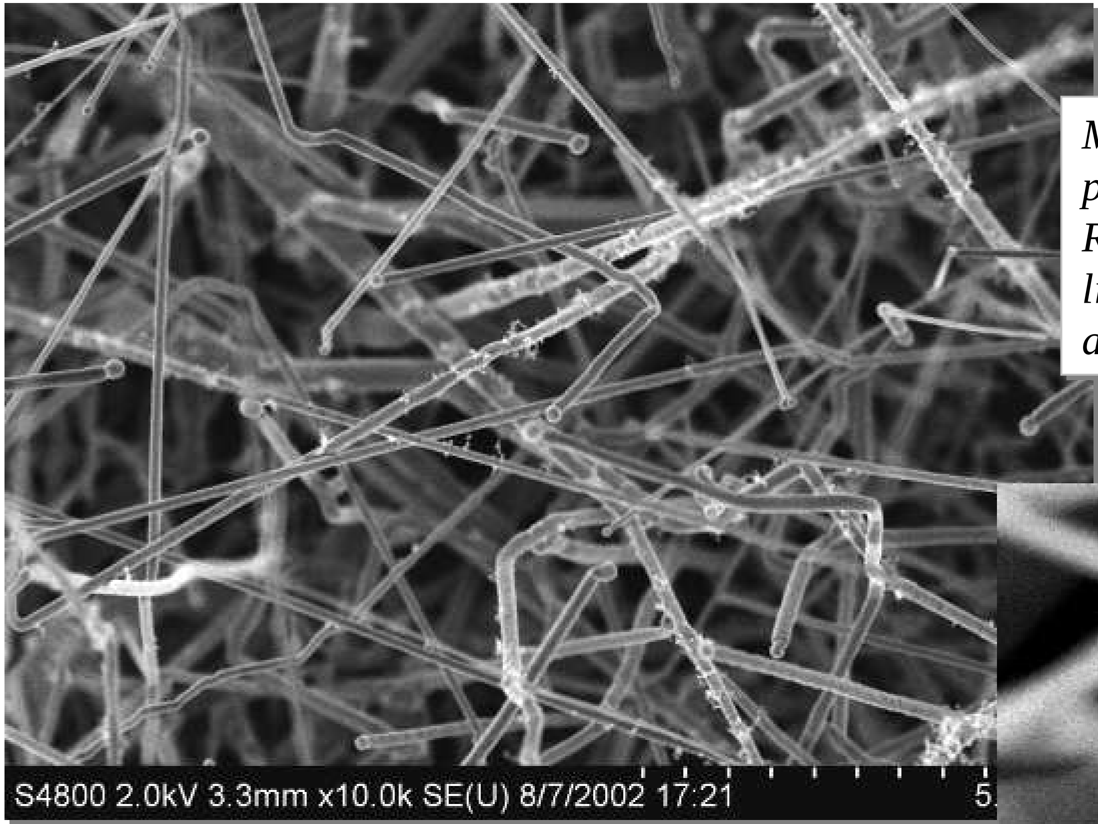
N.B. Enables BSE imaging even at very low accelerating voltages which were not possible with conventional scintillator or solid state BSE detectors (e.g. BSE imaging at 500V)


SE
S-5200 2.0kV x45.0k SE
1.00um

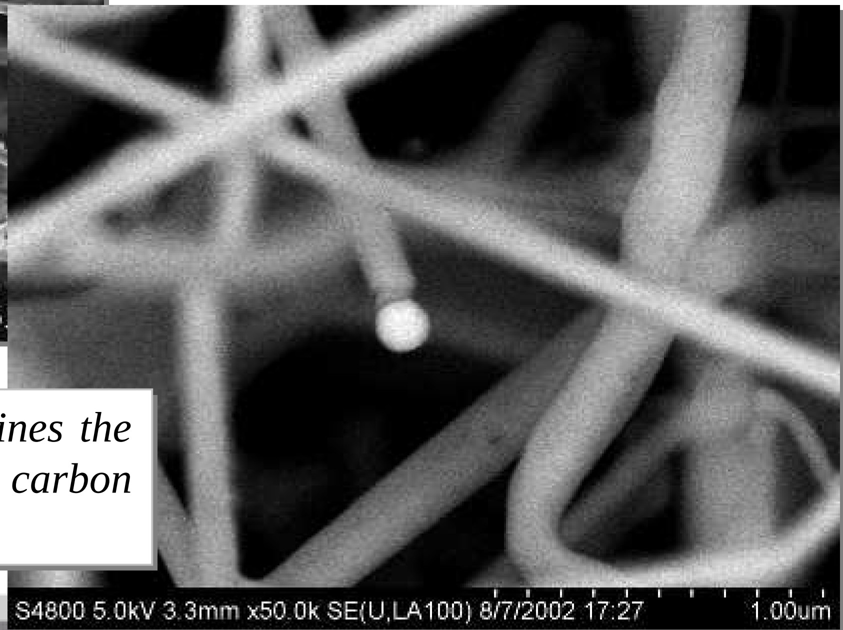

SE + BSE(L)
S-5200 2.0kV x45.0k SE(C16)
1.00um


BSE(H)
S-5200 2.0kV x45.0k BSE
1.00um

Nano-wires



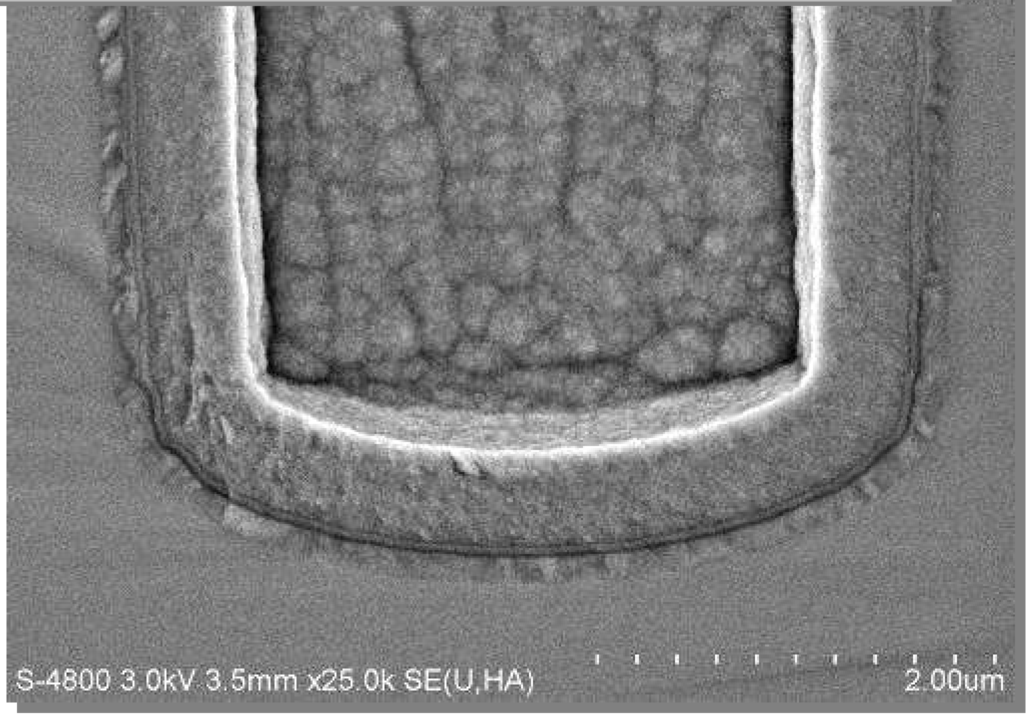
Metallic spheres are the initiation points of the nano-wires' growth. Researchers are interested in the lines and angles of the wires, as well as the position on the metal spheres.

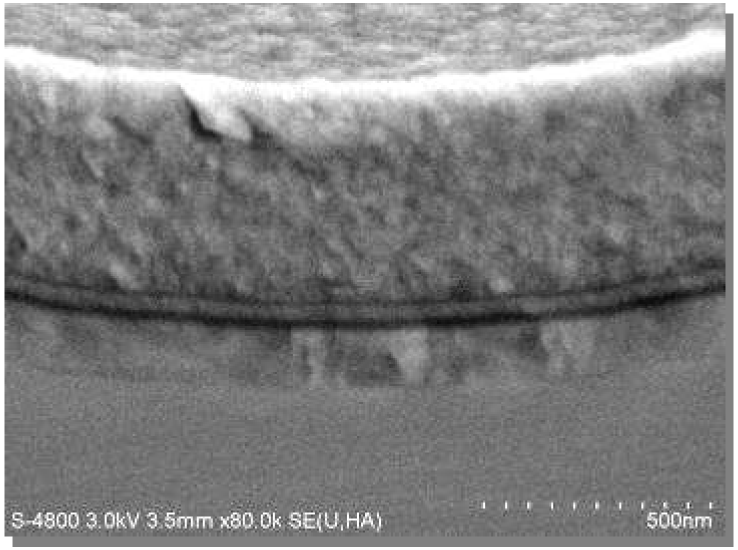


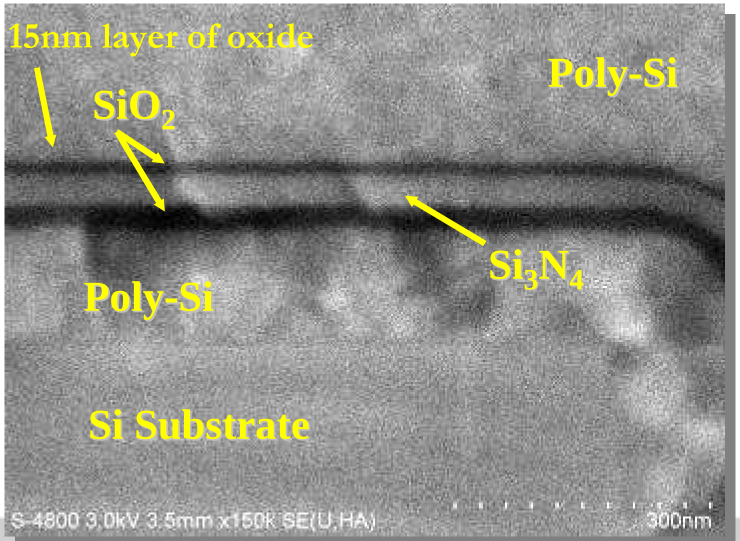
Backscatter imaging clearly defines the point of connection between the carbon tube and the metal particle.

MEMS Application

Good depth of field makes imaging the surface of this x-section and the deep-trench walls possible.

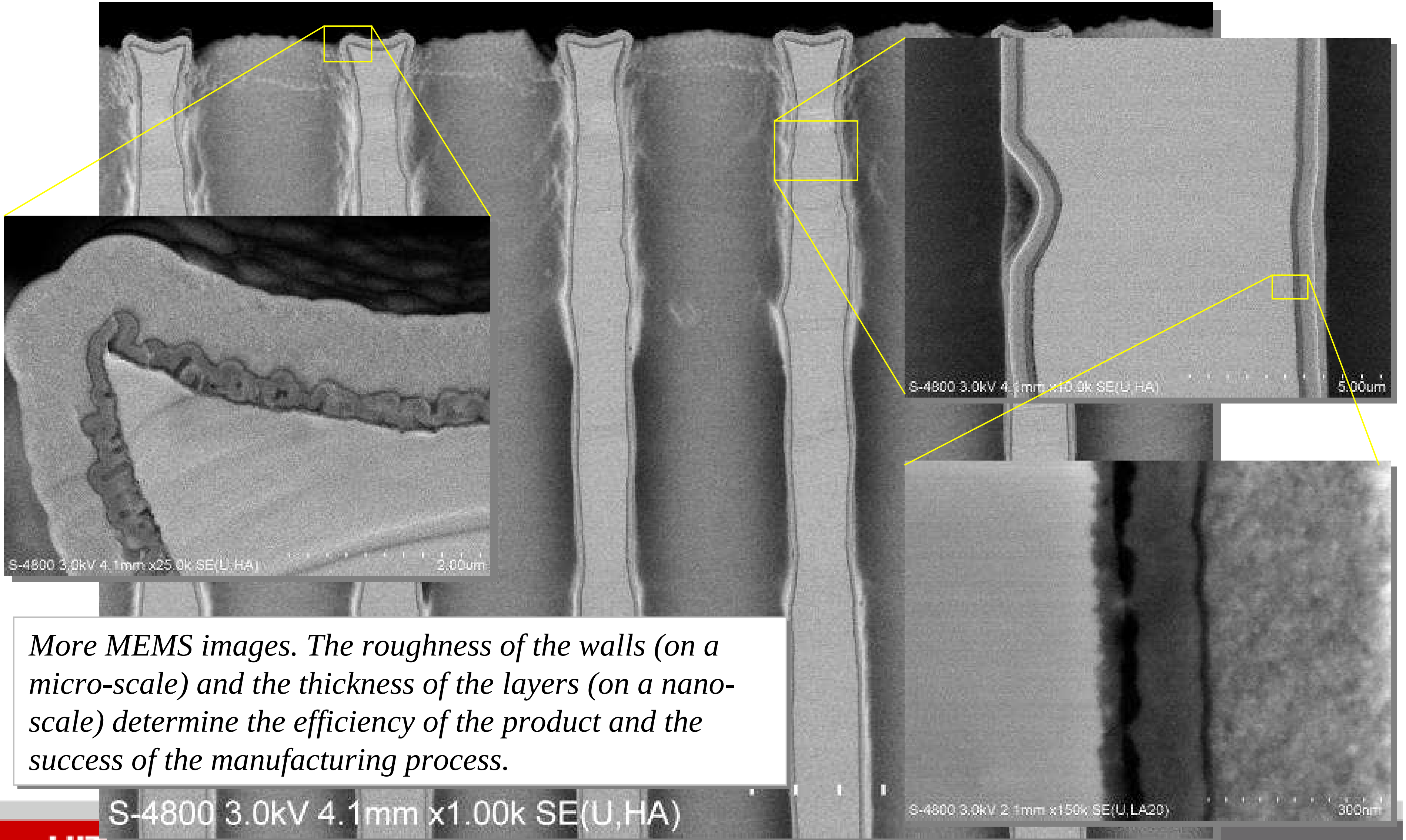






The HA backscattered resolution provides accurate measurements of the various silicon layers and clear interface locations of layers. Notice the beautiful granular structure of the polysilicon as well.

MEMS Application



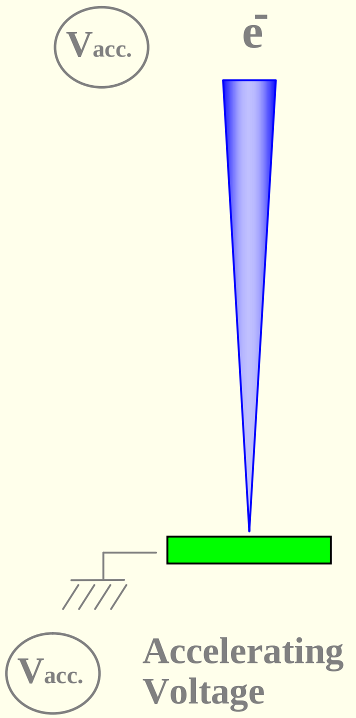
More MEMS images. The roughness of the walls (on a micro-scale) and the thickness of the layers (on a nano-scale) determine the efficiency of the product and the success of the manufacturing process.

Why Low Voltage SEM?

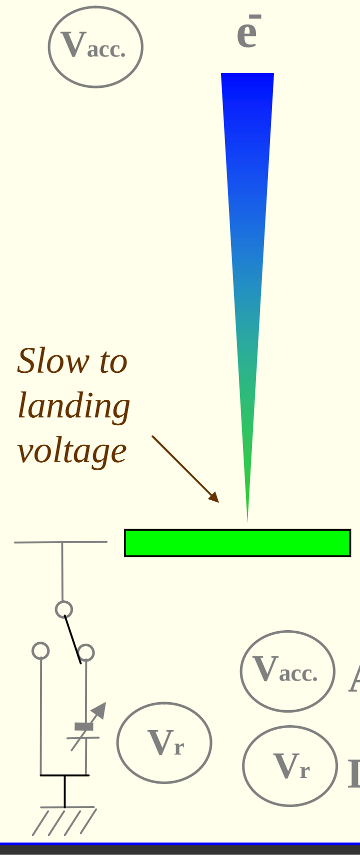
- Better image quality (minimum edge effect, visibility of surface details...)
- minimum beam damage artefacts
- observation of non-conductive material (plastics, ceramics...) without preparation artefacts
- saving of sample preparation time/technique
- lateral EDX resolution below 100nm

Beam Deceleration

No Beam Deceleration (Normal Condition)



Beam Deceleration



By applying a voltage (Vr) to the stage the primary beam is "decelerated". Benefits of this technique include improved resolution at lower kVs and truer surface imaging.

$V_{acc.}$ − V_r = Landing Voltage

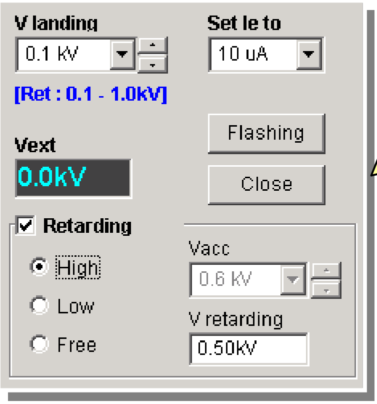
2.0kV – 1.5kV = 500V

For Example: an accelerating voltage of 2kV in combination with a deceleration voltage of 1.5kV results in a landing (imaging) voltage of 500 volts with a resolution similar to a 2kV image.

$V_{acc.}$ Acceleration Voltage

V_r Deceleration Voltage

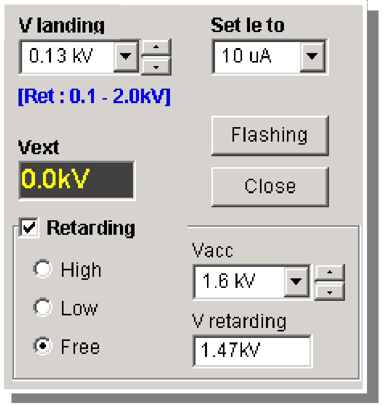
S-4800 Retarding Mode Operation

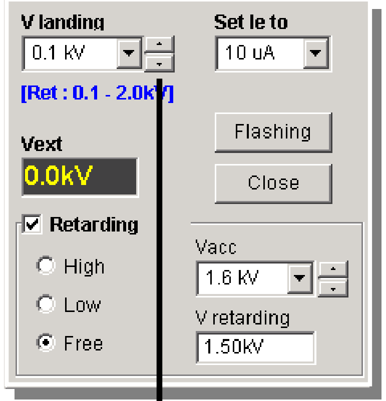


By selecting landing voltage, both accelerating voltage and retarding voltage are set automatically.
Voltage combination is different in High and Low mode as shown below.

V landing = 0.1 - 1.0kV (100V step)

Free





10V setting

Free mode is available for free combination of accelerating voltage and retarding voltage. Select landing voltage and accelerating voltage. Retarding voltage is set automatically. In the Free mode, 10V step is available.

V landing = 0.1 - 2.0kV (10V step)

High

High mode Vacc(kV)	High mode Vr(kV)	High mode Vr/Vacc	High mode Vl(kV)
0.6	0.5	0.83	0.1
1.3	1.1	0.85	0.2
1.8	1.5	0.83	0.3
1.9	1.5	0.79	0.4
2.0	1.5	0.75	0.5
2.1	1.5	0.71	0.6
2.2	1.5	0.68	0.7
2.3	1.5	0.65	0.8
2.4	1.5	0.63	0.9
2.5	1.5	0.60	1.0

Low

Low mode Vacc(kV)	Low mode Vr(kV)	Low mode Vr/Vacc	Low mode Vl(kV)
0.5	0.4	0.80	0.1
0.6	0.4	0.67	0.2
0.7	0.4	0.57	0.3
0.9	0.5	0.56	0.4
1.2	0.7	0.58	0.5
1.4	0.8	0.57	0.6
1.0	0.3	0.30	0.7
1.2	0.4	0.33	0.8
1.3	0.4	0.31	0.9
1.5	0.5	0.33	1.0

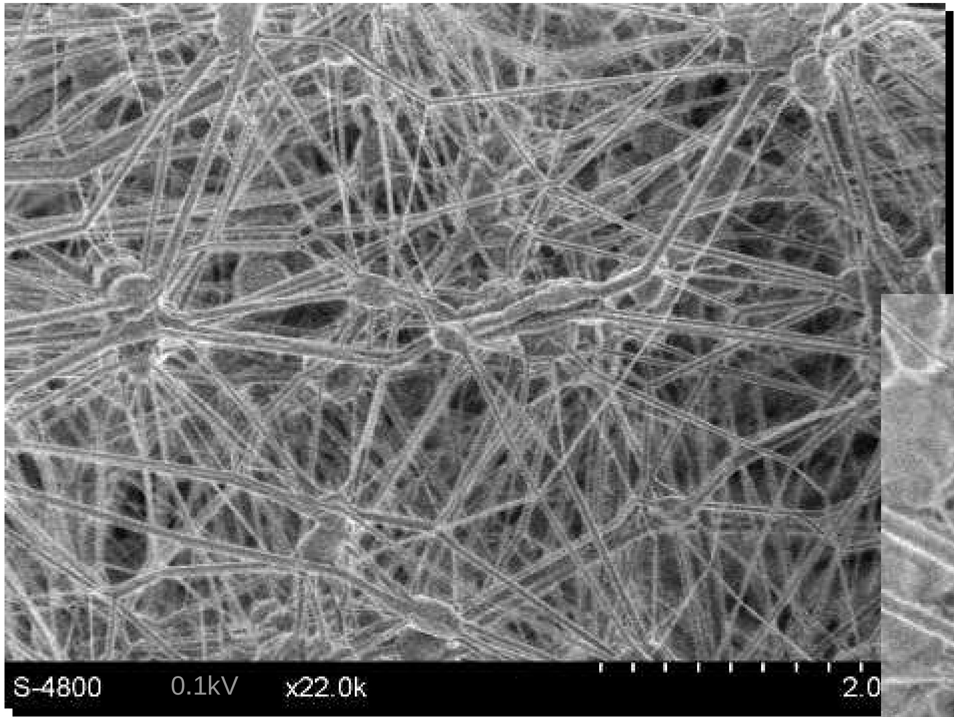
The higher retarding voltage, the higher resolution. In case of heavy charging sample, higher retarding voltage may not be effective to reduce the charge-up. In that case, Low mode works better.

Data Display at Retarding Mode

S4800 0.2kV-R

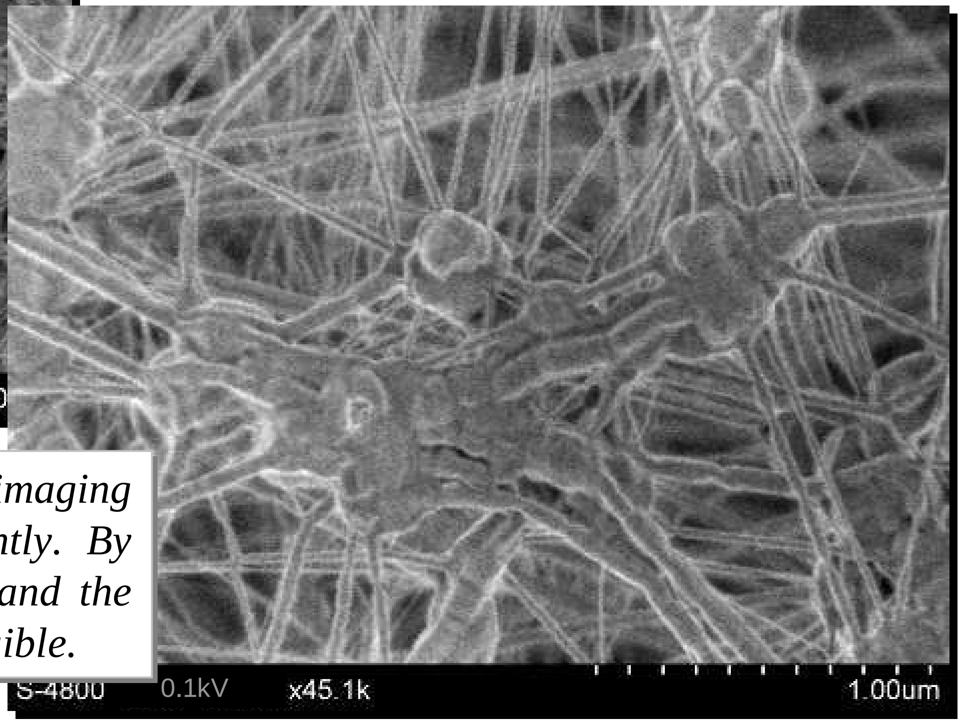
S4800 0.21kV-R

Membrane Filter



Observation at 100V

Deceleration ON 1600 – 1500 = 100V

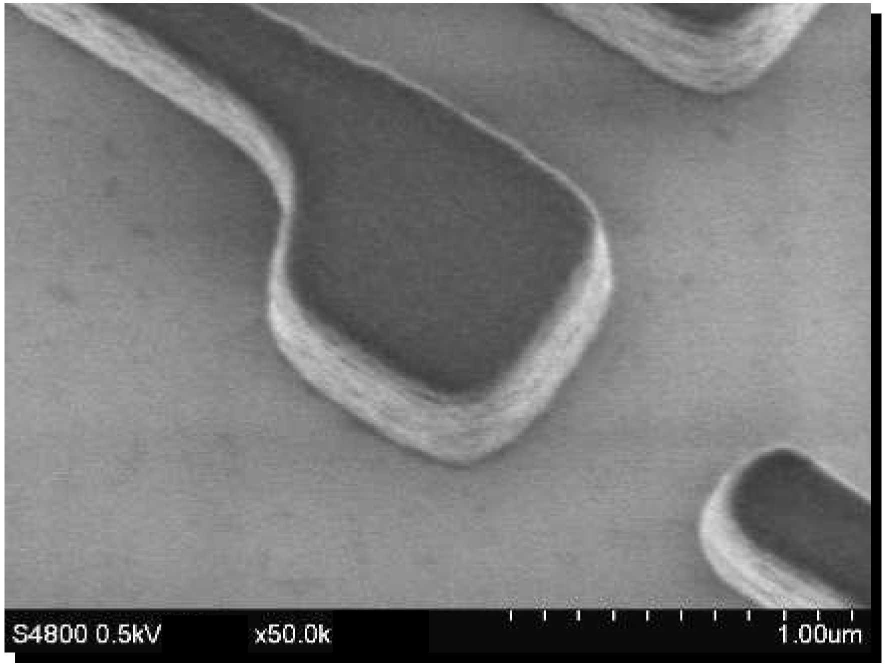


This membrane filter is uncoated. Under normal imaging conditions the sample would charge significantly. By imaging at 100 volts charging does not occur and the ribbed surface structure of the fiber clusters is visible.

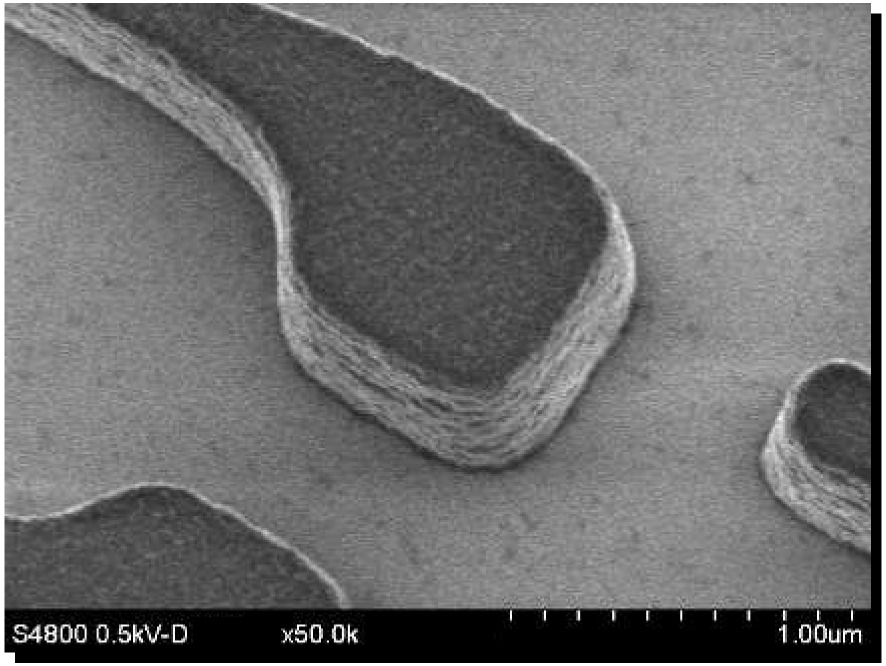
Photo Resist

Hitachi's Beam Deceleration technology enhances fine surface features and nano-size details as seen in this comparison of photo resist with Beam Deceleration OFF and ON.

Observation at 500V



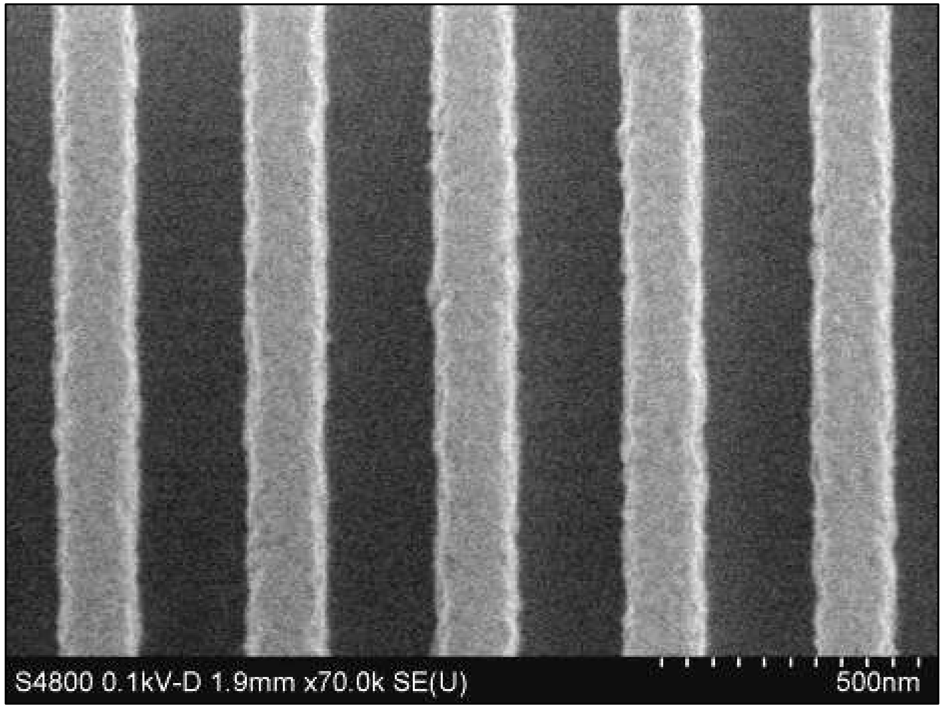
Beam Deceleration ***OFF***



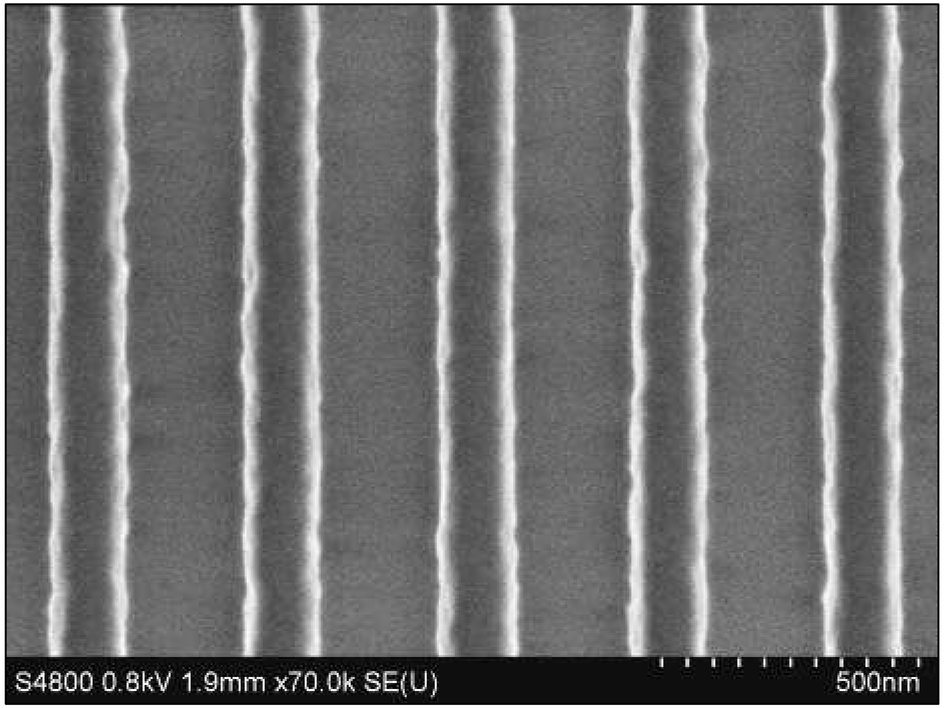
Beam Deceleration ***ON***

ArF Resist

Comparing a 100 volt image to the typical 800 volt image one can see more surface detail and clear definition between the lines and trenches.



Landing voltage : 100V



Accelerating voltage : 800V

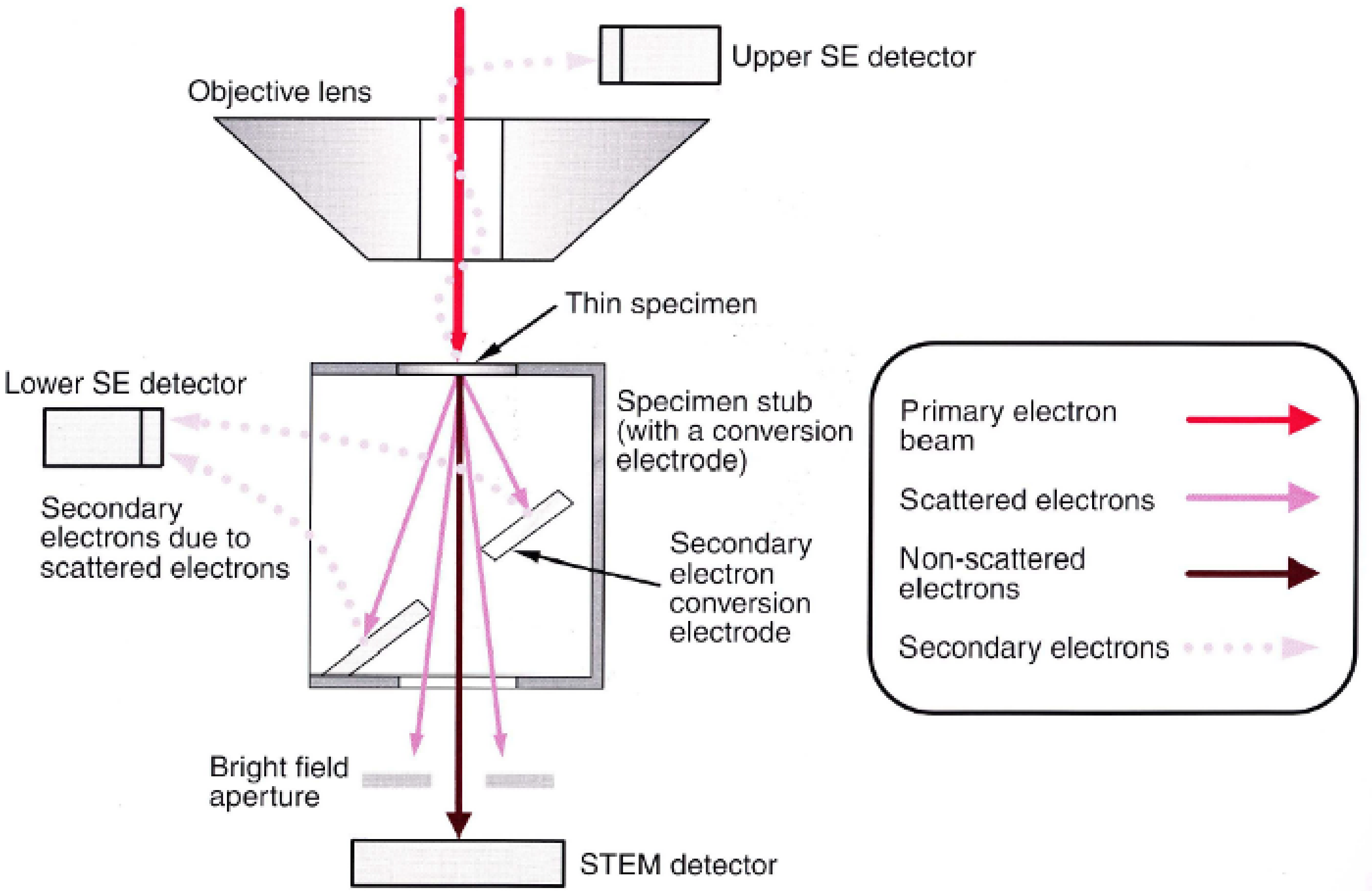
Sample courtesy of Canon Inc.

Probe current : 6pA, Magnification : 70kX

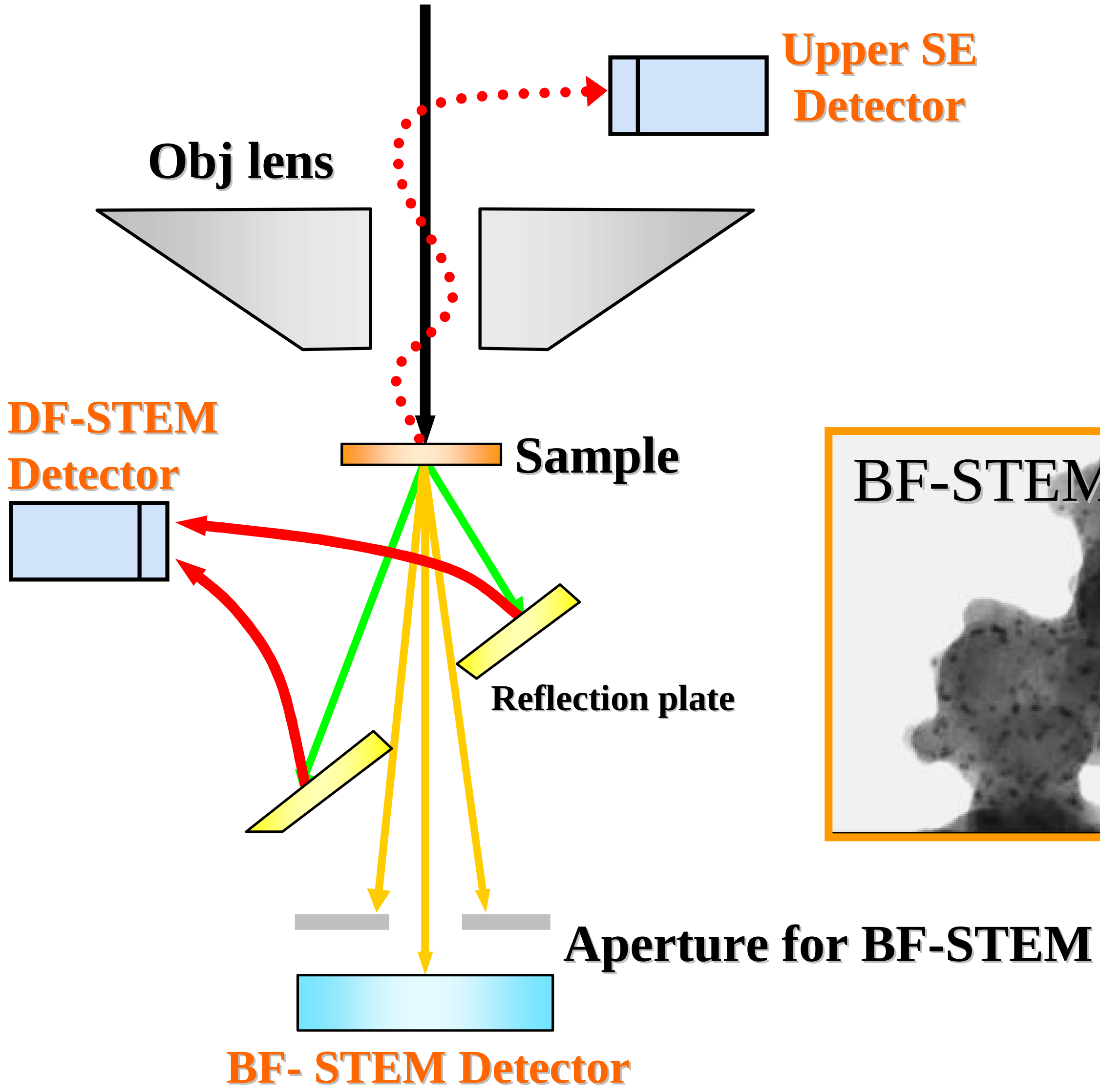
Why the increase in SEM-based STEM imaging?

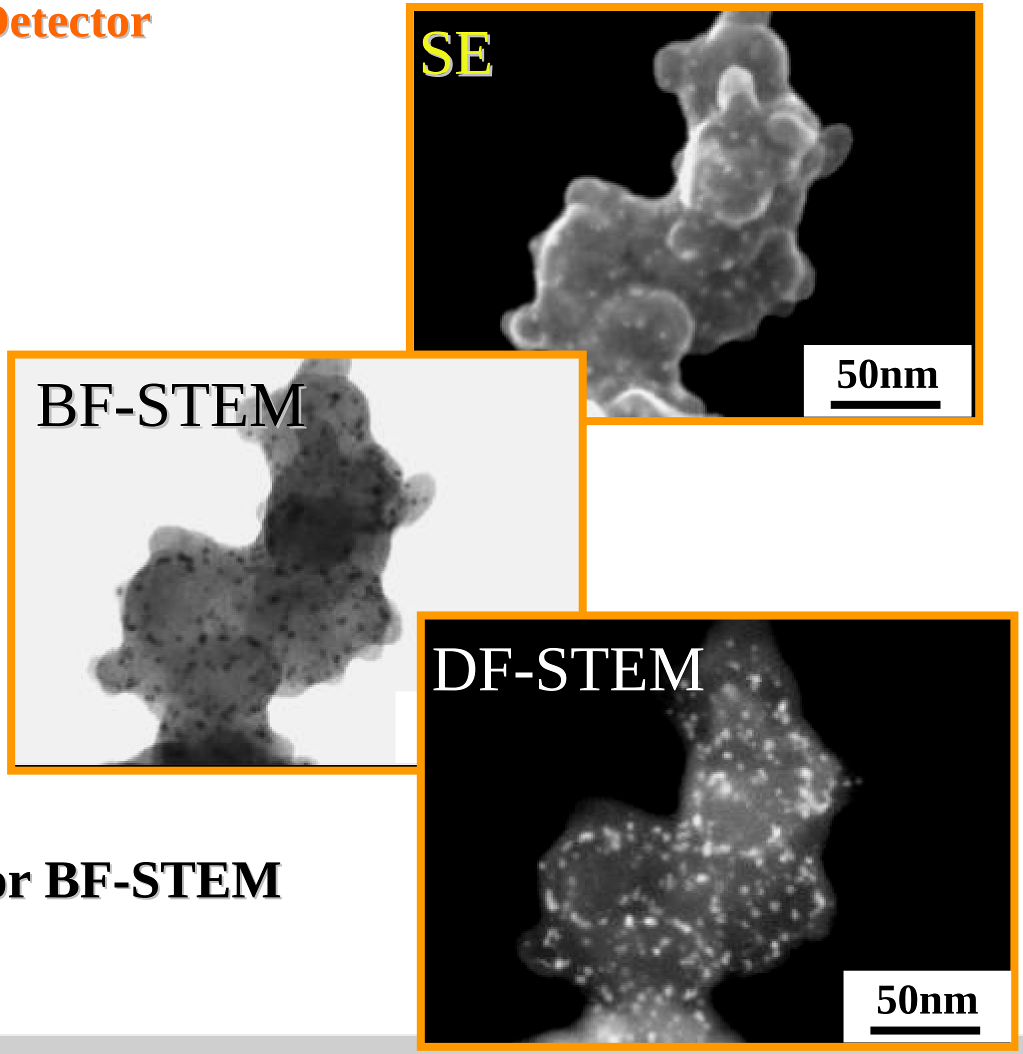
- Enabling technologies like FIB have made thin-section preparation of bulk materials quick and easy
- Research into new nano-materials has demanded high throughput but detailed structural investigation i.e. not just near-surface information from SE and BSE
- The increased use of C-based nano-materials has led to an understanding that high kV is not always required (light C-based material is easily penetrated by less energetic electrons)
- STEM of thin specimens has some potential resolution advantages over SE of thicker specimens as the broadening effect of the interaction volume is reduced. For certain samples moving from SE to STEM gives a quick and simple step-change in resolution (e.g. from 1nm to 0.5nm for in-lens systems)
- The latest ultra-high resolution in-lens SEMs provide <4Å resolution which has pushed the capability of SEM-based STEM closer to that of TEM.

BF/DF STEM (1): A simple STEM configuration

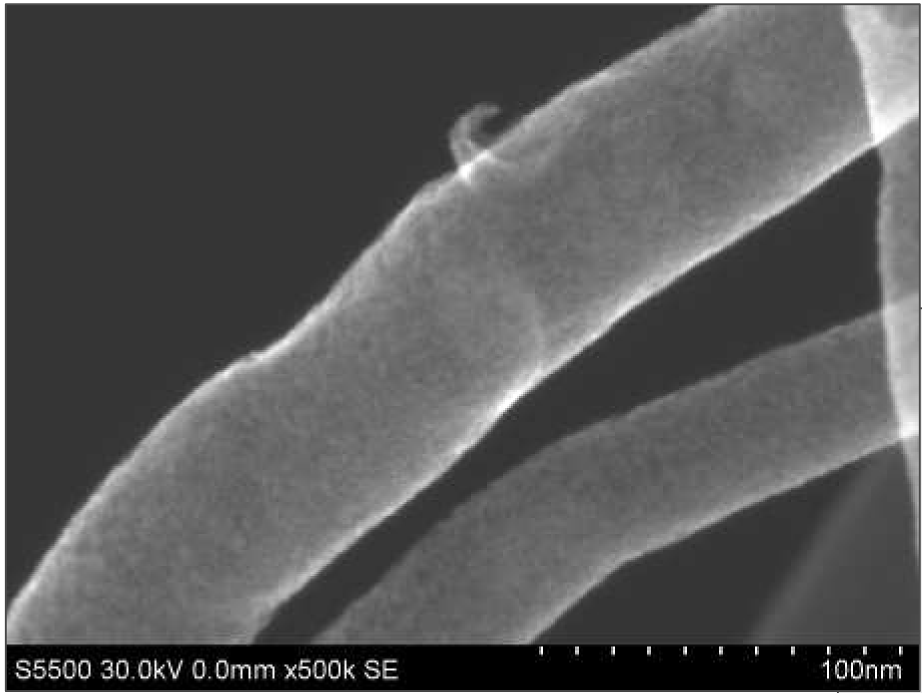


Simultaneous STEM Imaging: BF and DF



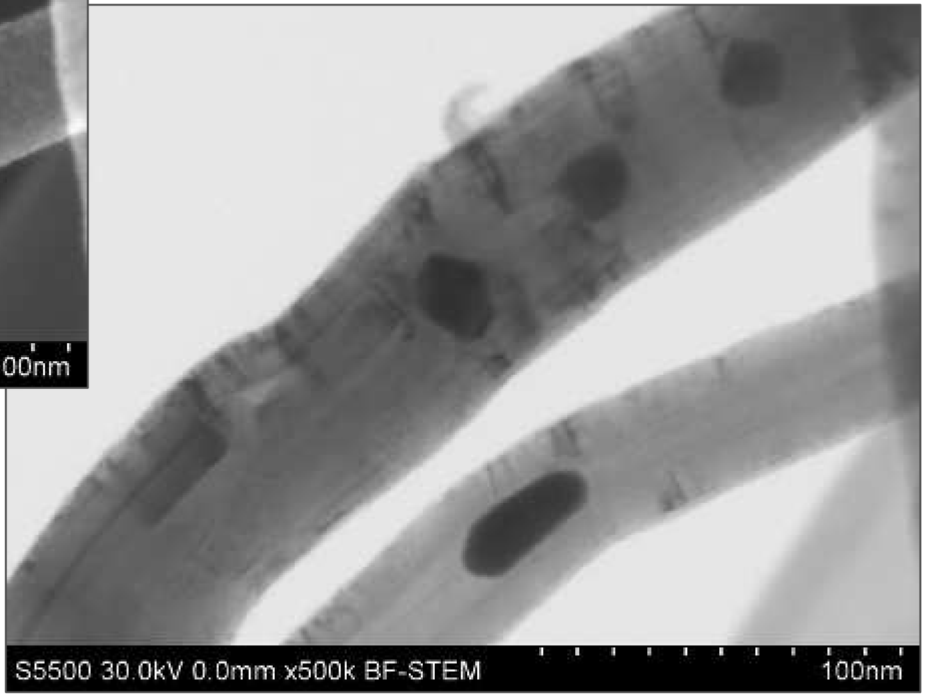


SE and STEM for Nano-materials

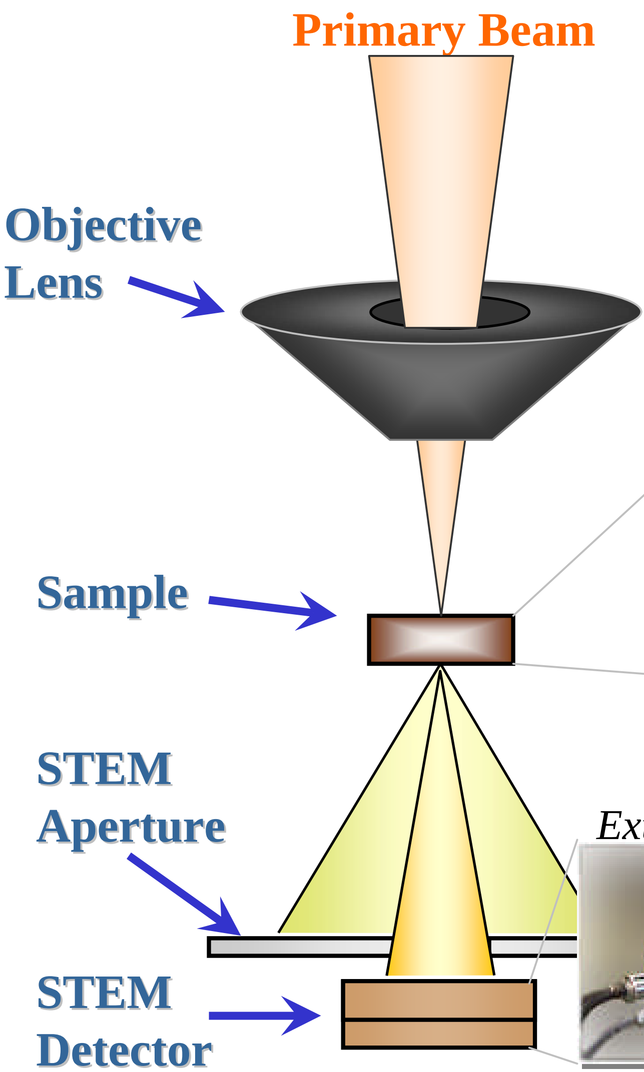


CNT- SE Image

CNT- BF Image

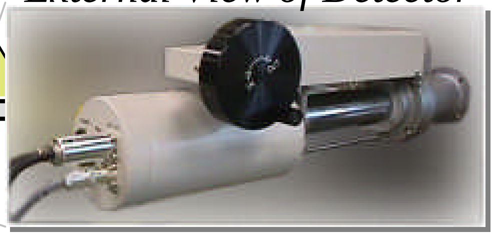


STEM Imaging: Brightfield



Actual STEM Holder

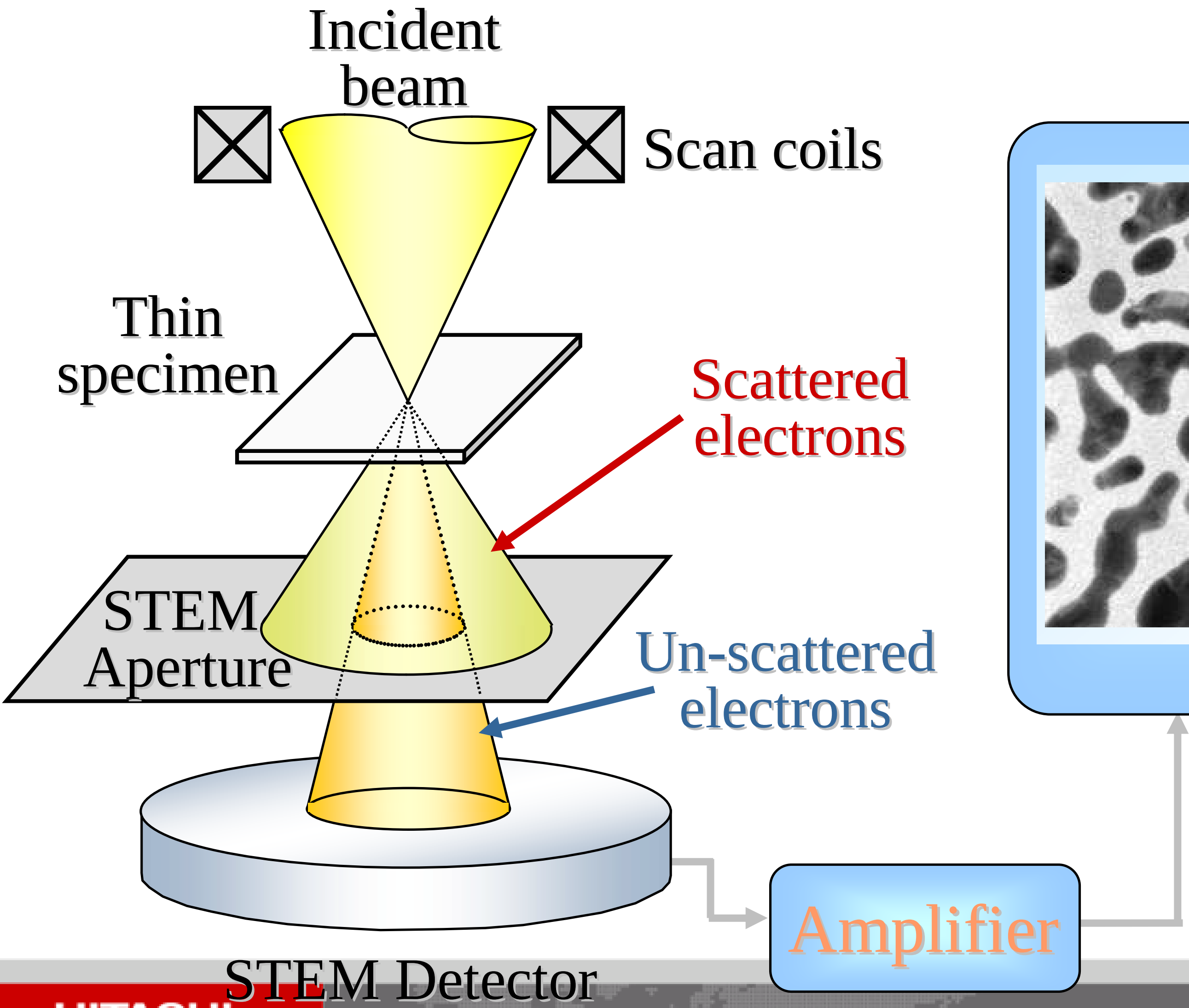
External View of Detector

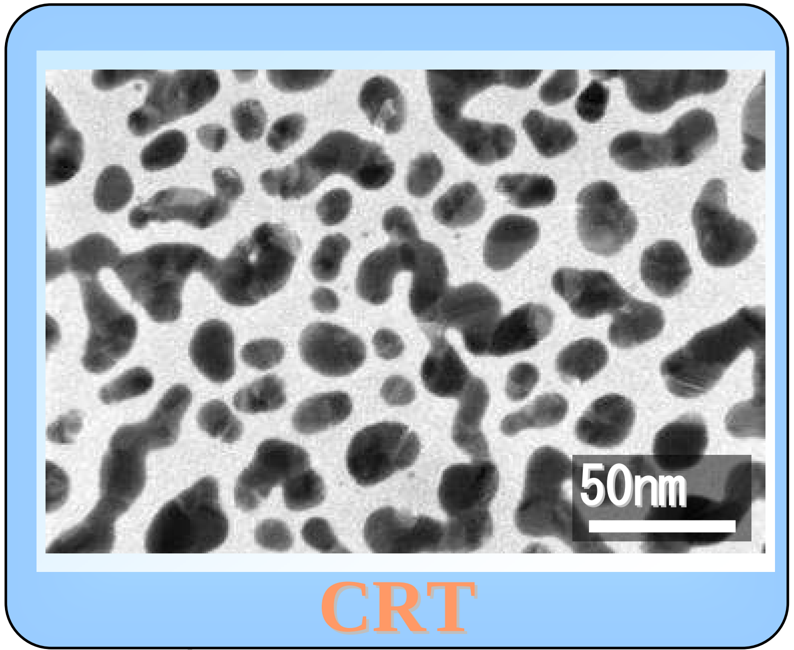


Low voltage STEM imaging at 30kV in an SEM can provide high contrast on low atomic number materials. STEM images of various sample types is possible, from semiconductors to powders to biological samples.

The BF-STEM detector is always mounted to the chamber so it is easy to switch between STEM imaging from other imaging modes. The majority of the following examples have both SE and STEM images so that comparisons can be made.

STEM : Bright Field Imaging

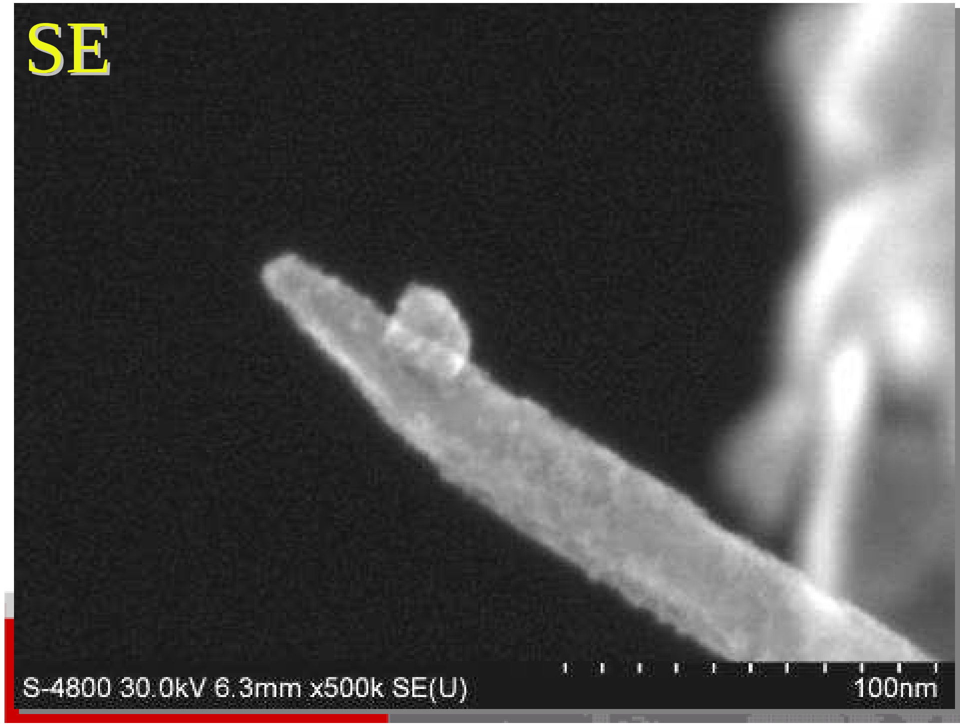


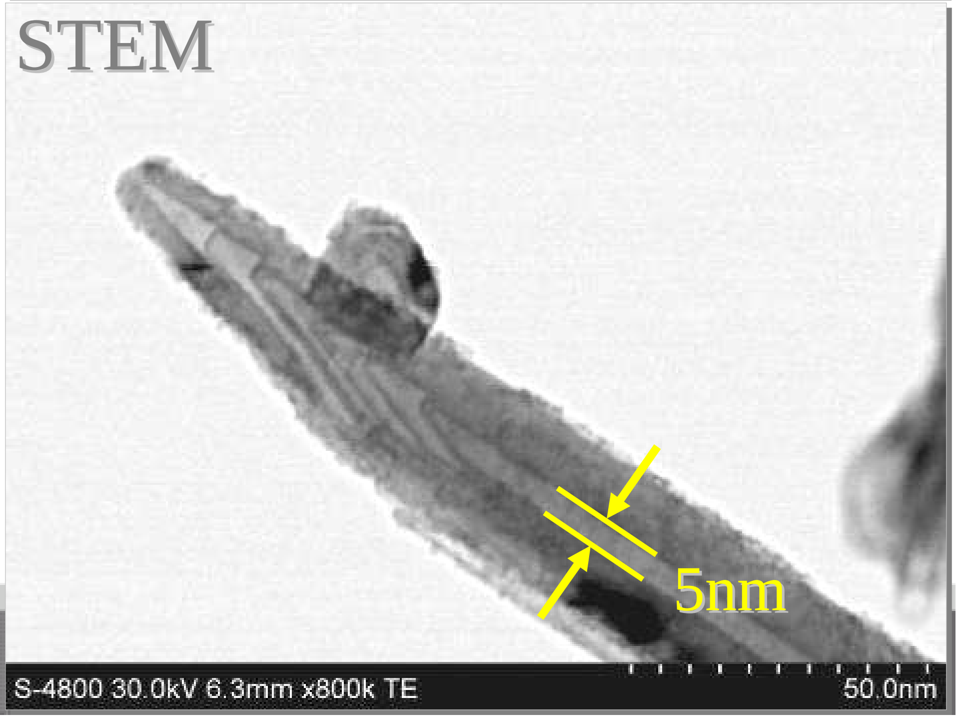


Specimen :
Au islands on C film
Vacc. 30kV

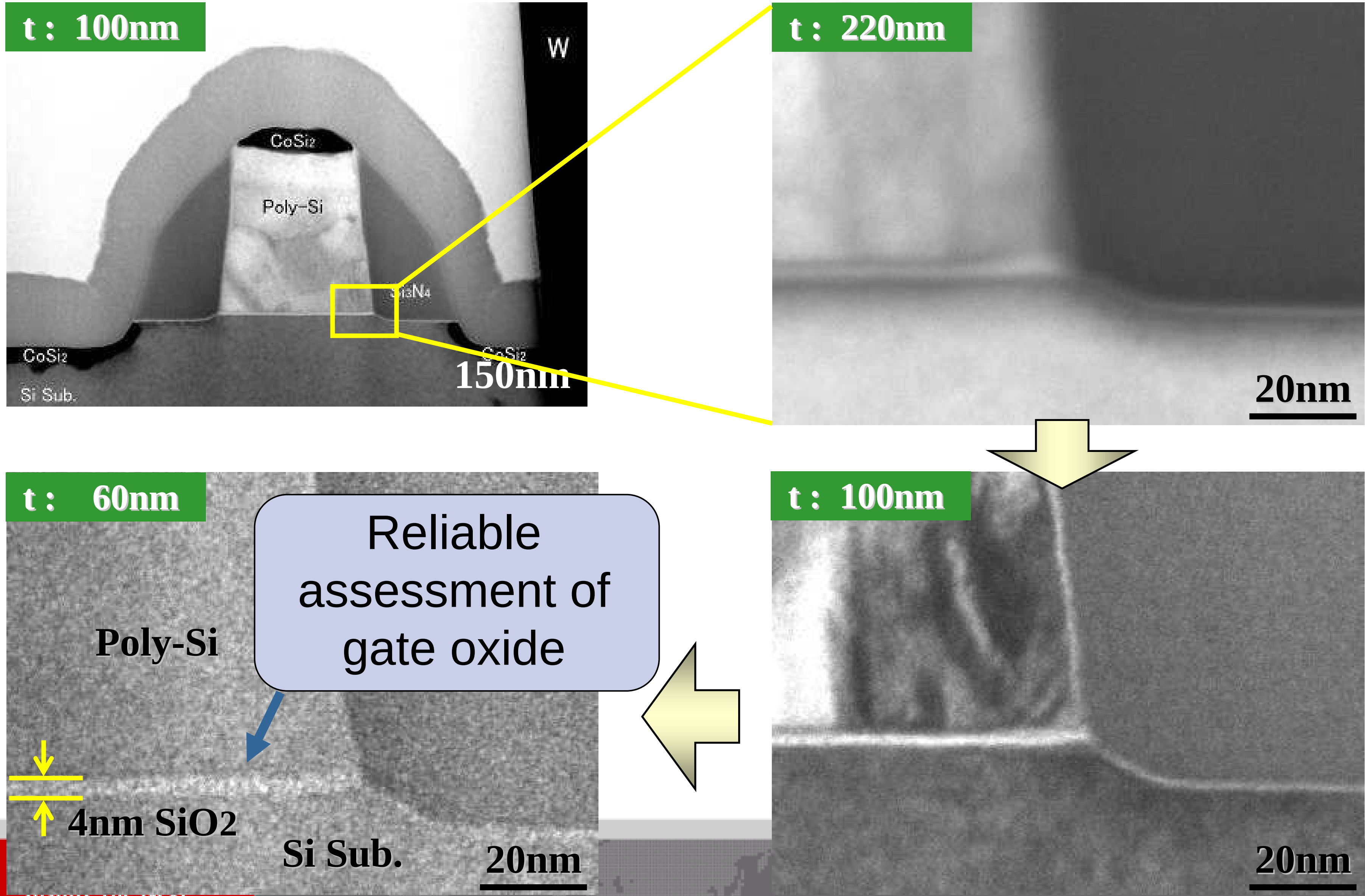
Carbon Nanotube

The SE image below shows excellent surface structure. The structure is also visible in STEM mode. At 800,000x the internal growth structure of the nanotube and internal tube diameter can be accurately measured.

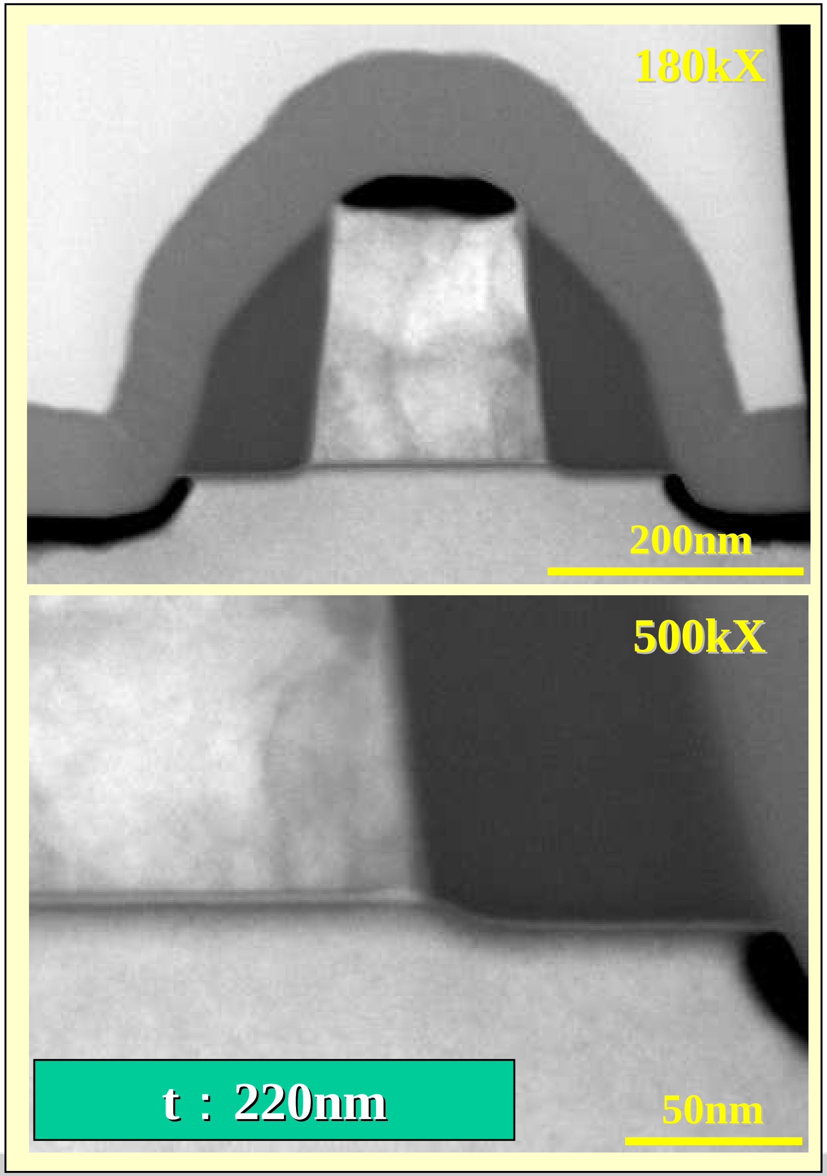


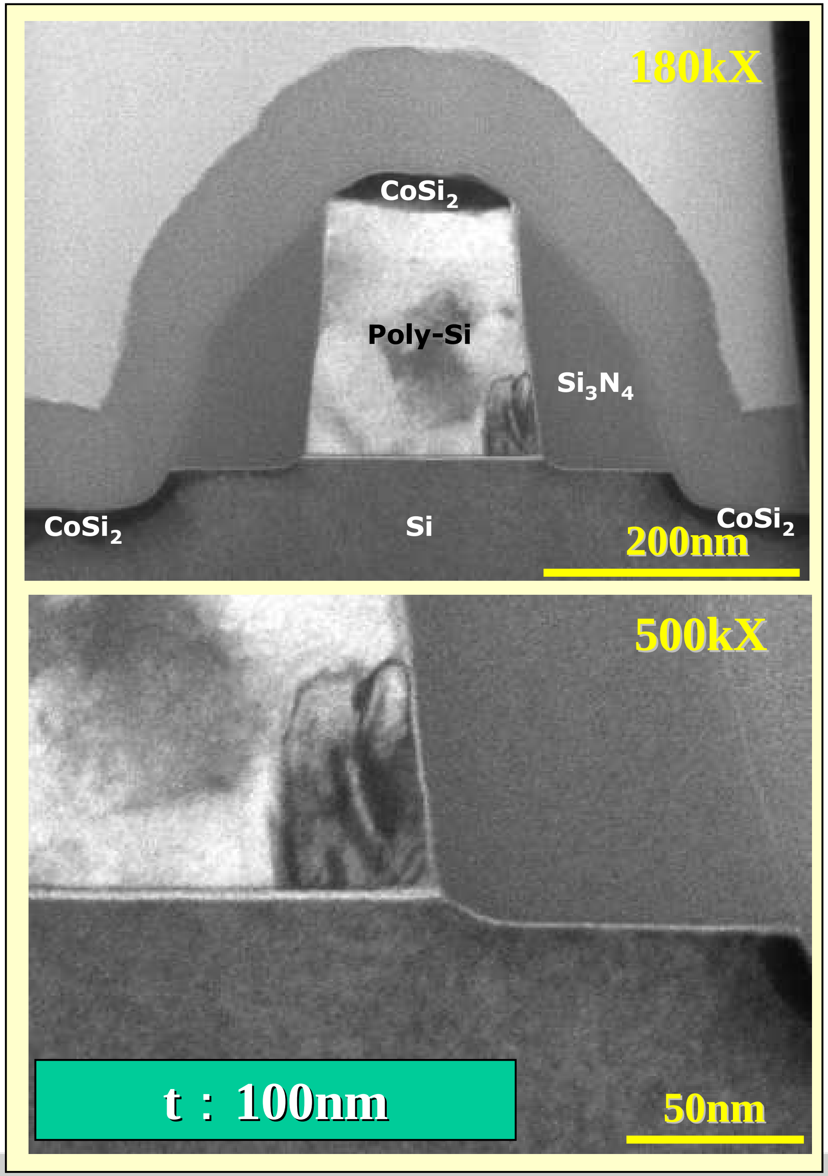


STEM of Semiconductors



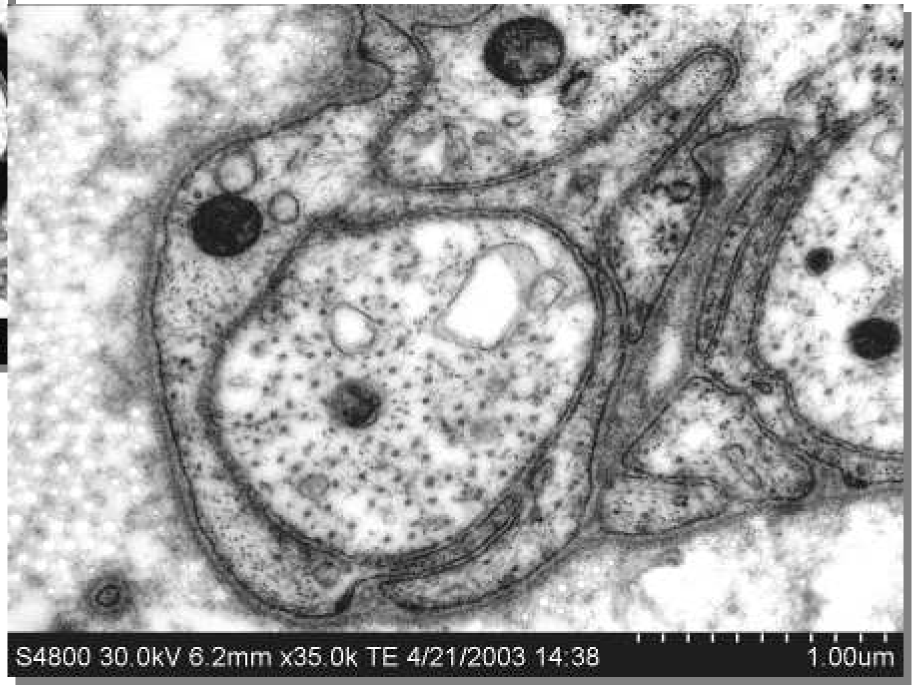
STEM of Semiconductors



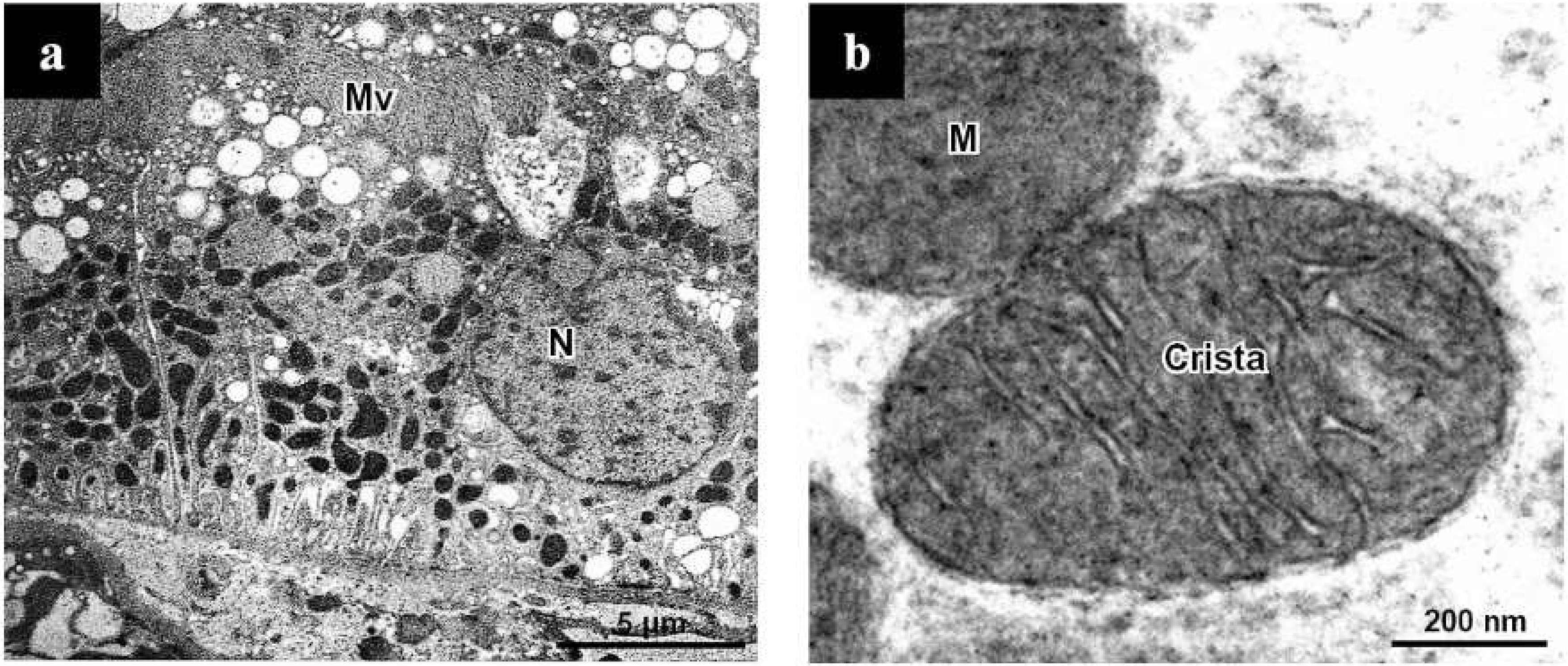


Human Nerve Tissue

Biological thin section samples imaged with the S-4800 dedicated STEM detector show great contrast and detail. Samples are easily mounted as TEM grids onto the STEM holder. The images here of stained human nerve tissue clearly show the mitochondria, cell walls, and plasma.



STEM of biological sections

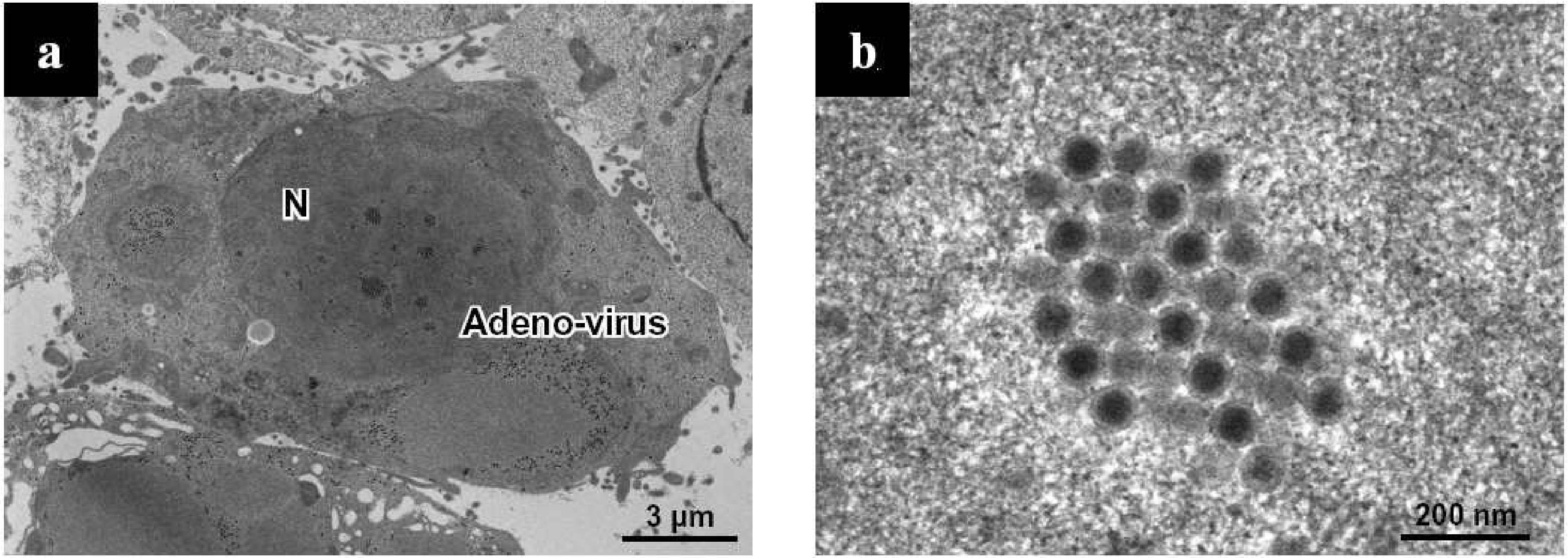


(a) Magnification: ×5,000 (b) Magnification: ×80,000

Fig. 6 Uriniferous tubule of unstained human nephritis kidney, sectioned at about 0.1 µm and recorded at 15 kV

Detailed structure of Cristæ is clearly observed in this mitocondrion

STEM of virus particles

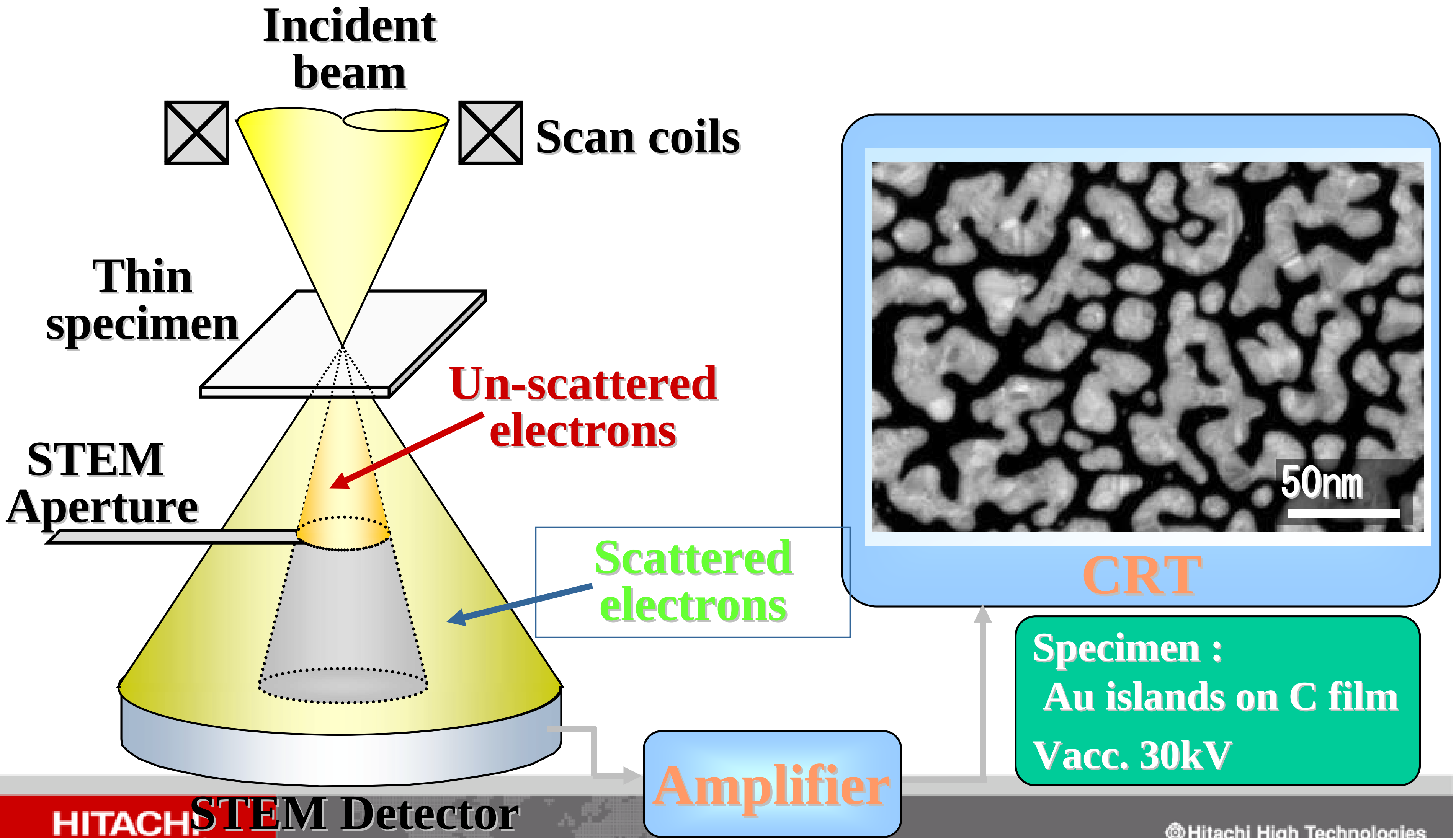


(a) Magnification: ×6,000 (b) Magnification: ×100,000

Fig. 5 A propagating condition of cancer cells in liver, sectioned at about 0.1 μm and recorded at 30 kV

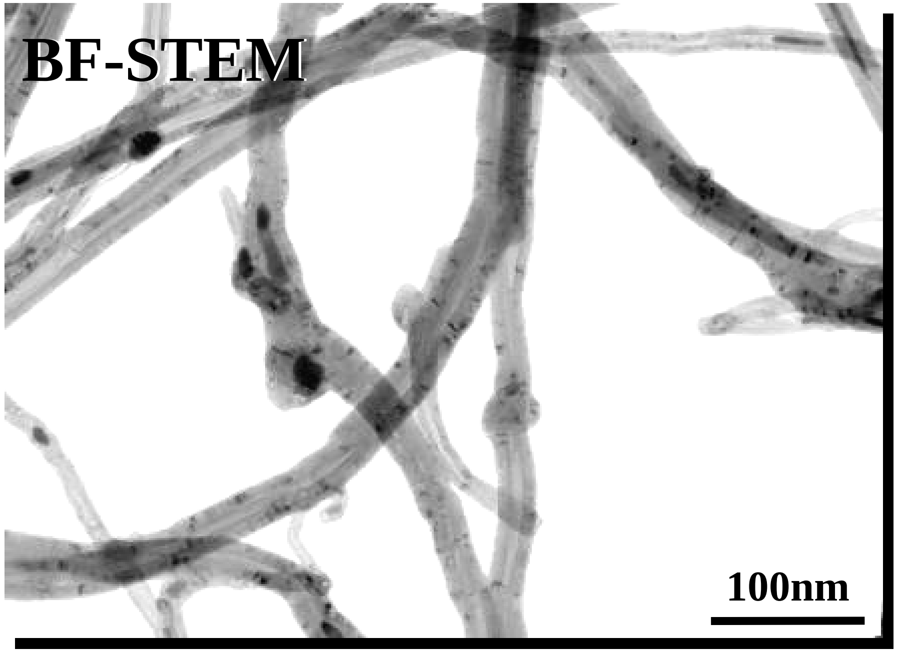
Adenovirus has potential gene-therapy uses for a number of genetic disorders or cancer cells. In this case the propogation of adeno-virus within the cancerous cell is easily monitored without the need for dedicated TEM.

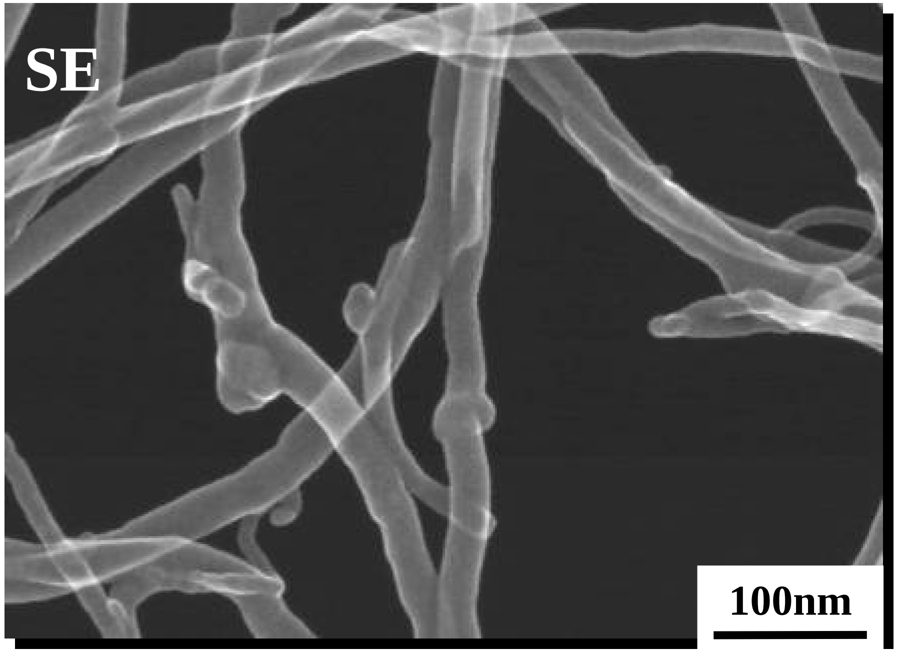
STEM : Dark Field Imaging

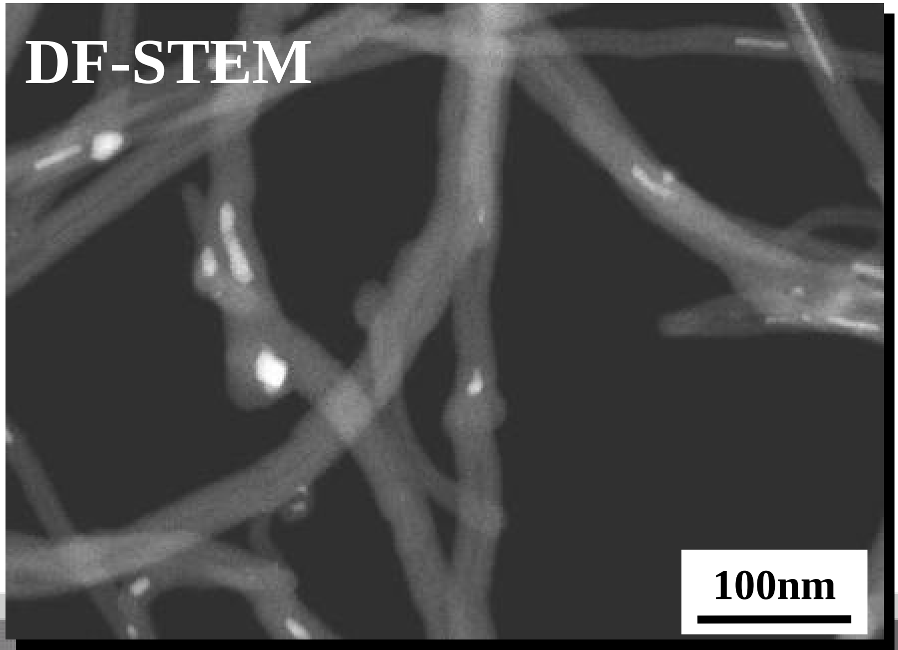


Carbon Nanotubes

Simultaneous collection of BF, DF, and SE imaging is unique to Hitachi's S-4800. This feature utilizes both SE detectors and our dedicated TE detector to provide a variety of high resolution information at one time.







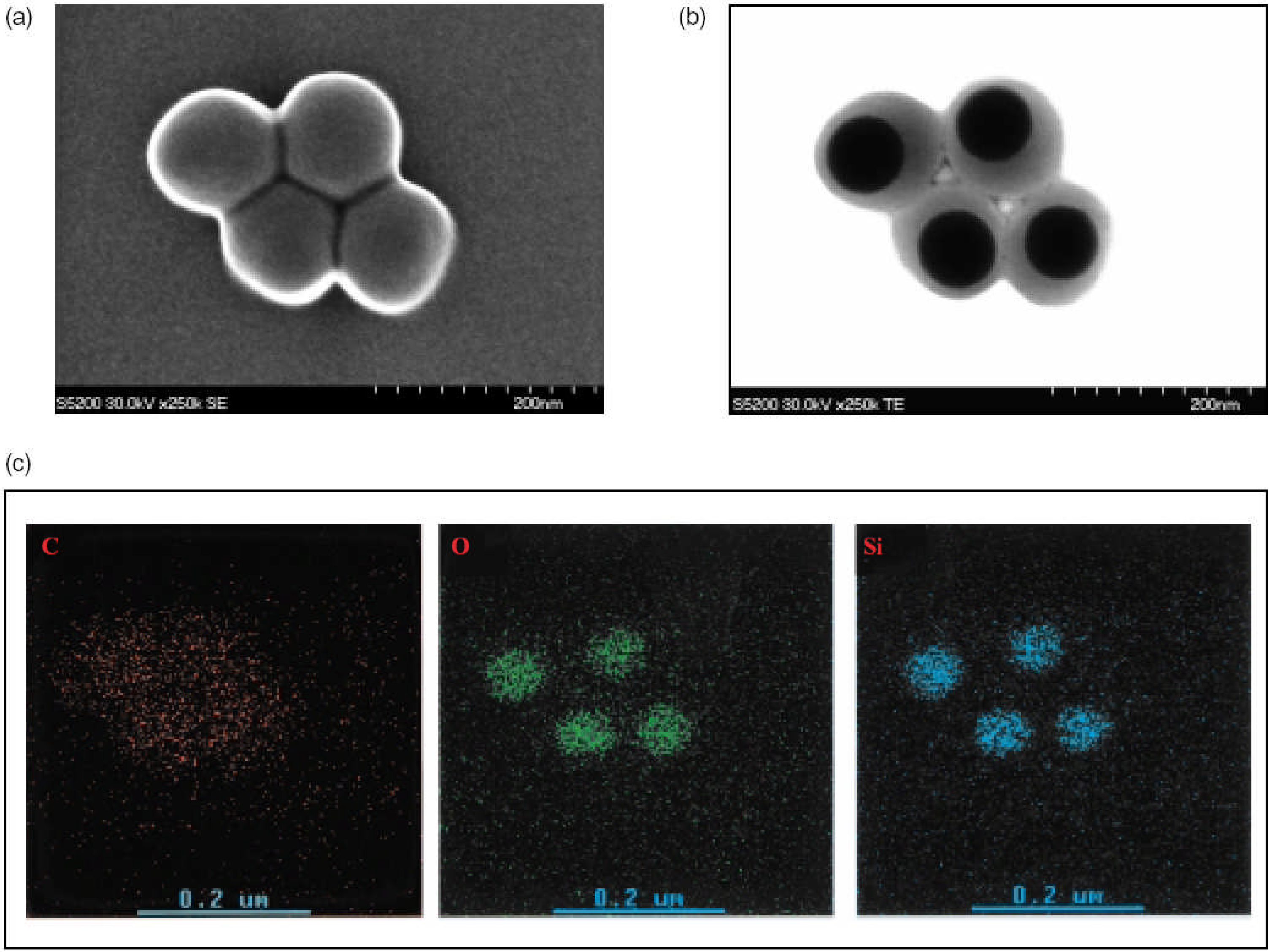


Fig. 4 Microscopy of polymers
(a) SEM image
(b) STEM image
(c) X-ray mapping image

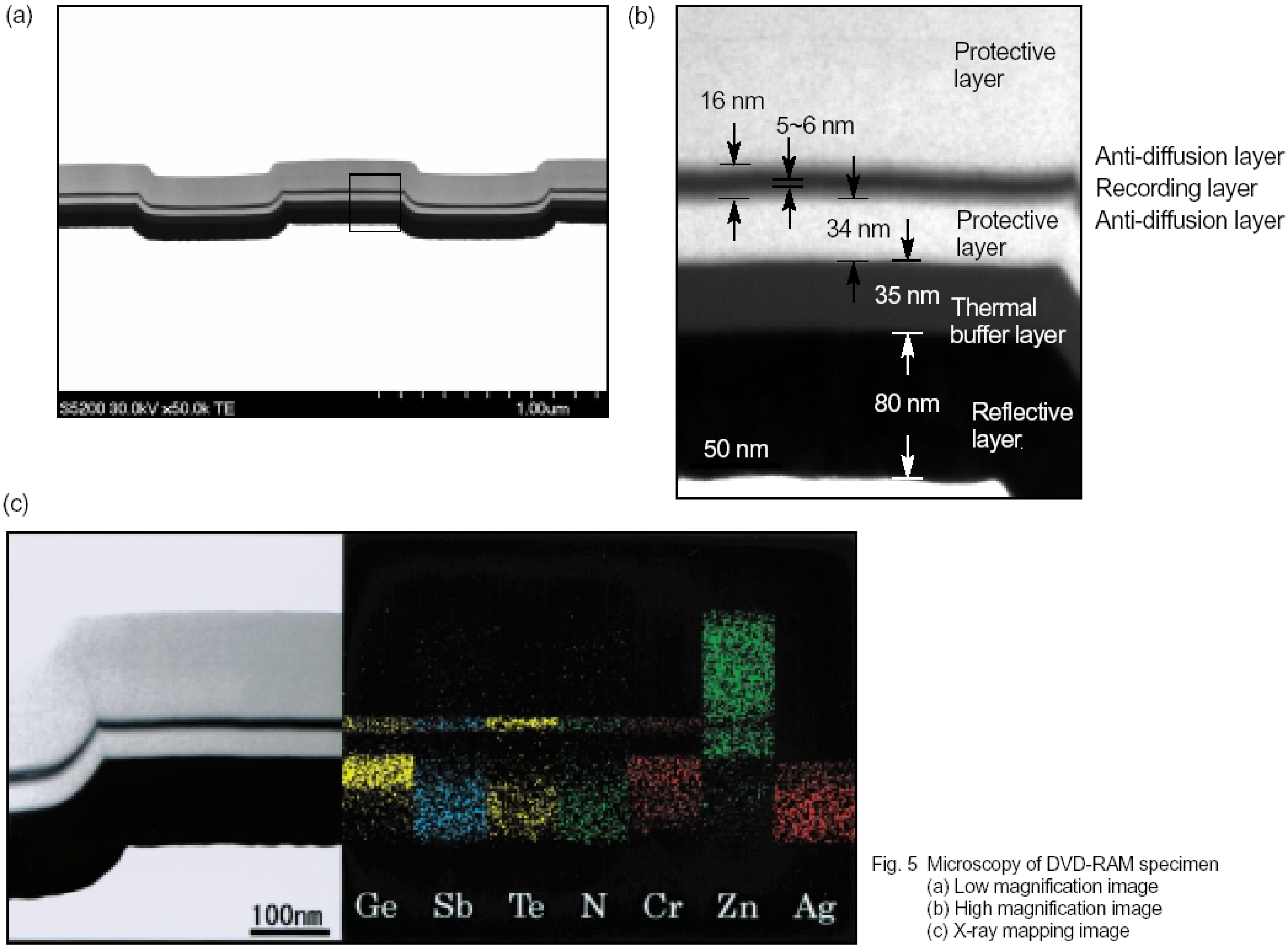


Fig. 5 Microscopy of DVD-RAM specimen
(a) Low magnification image
(b) High magnification image
(c) X-ray mapping image

Summary

SEM-based STEM imaging is developing an increasingly wide range of applications in material and biological sciences

While the technique should not be seen as a replacement for high energy TEM for all applications (due to its lower resolution and lower penetration capabilities) it nonetheless has its own strengths (imaging of low density materials, ability to relate surface and bulk properties, cost, ease of use etc.)