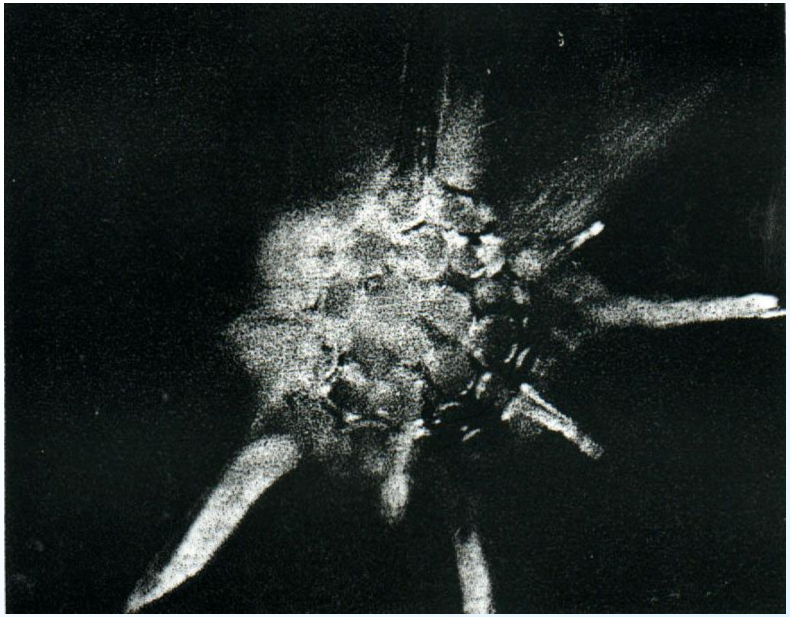
Scanning Electron Microscopy

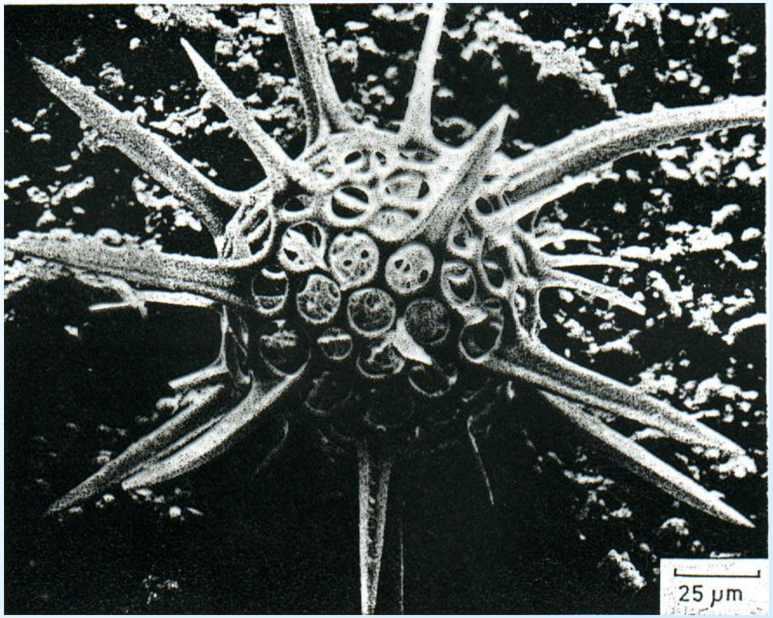
Alfred Gunasekaran

Scanning Electron Microscopy

Image Quality



Optical Microscope



SEM

SEM is very useful for examining objects at a wide range of magnifications, compared to optical microscope.

IfM
Institute for Micromanufacturing
Louisiana Tech University

Electron Microscopy

SEM

FESEM

A standard SEM is typically used for low-to-medium magnification (10-150,000X) imaging of conductive samples, usually metals.

For non-conductive samples, a conductive coating of carbon, gold, chromium, etc. should be applied to avoid charging effects.

A variable pressure SEM (low vacuum or environmental) is used for non-conductive specimens like glass, polymers/paint, and biological materials.

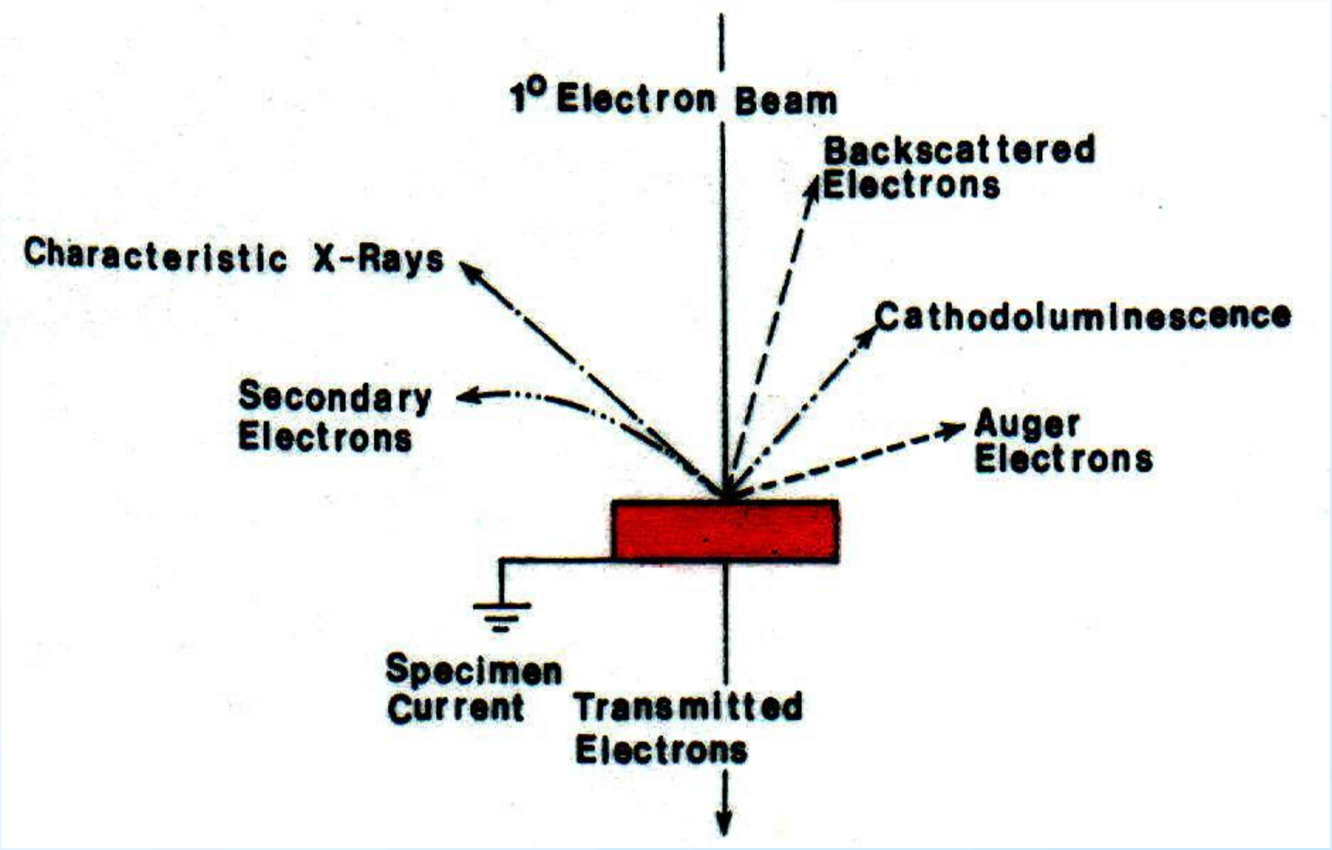
A field-emission SEM (FESEM) is used for medium-high magnification (20,000- 800,000X) and high resolution at low electron beam accelerating voltages (0.5-2 kV) resulting in excellent surface texture images even on non-conductive materials.

Current SEM Facility

Hitachi S-4800 FESEM
Cold-cathode Gun

Amray 1830 SEM
Thermionic Emission

Electron Beam-Sample Interaction

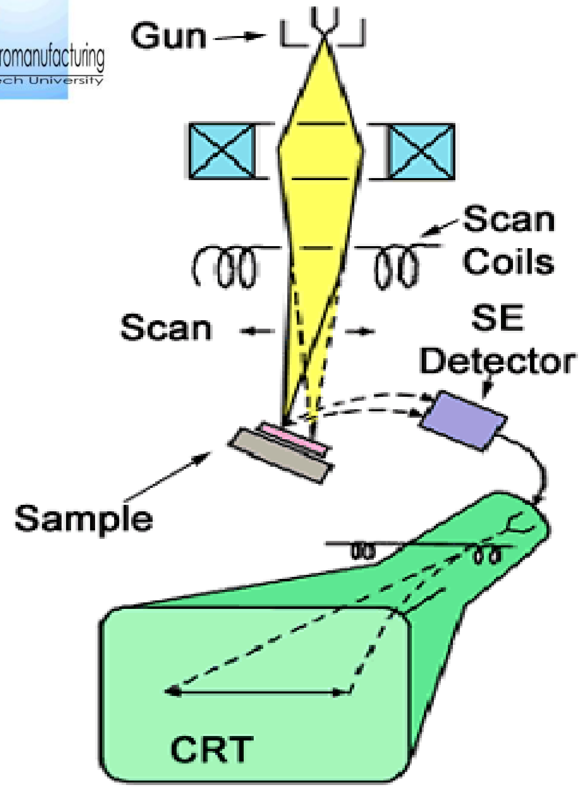


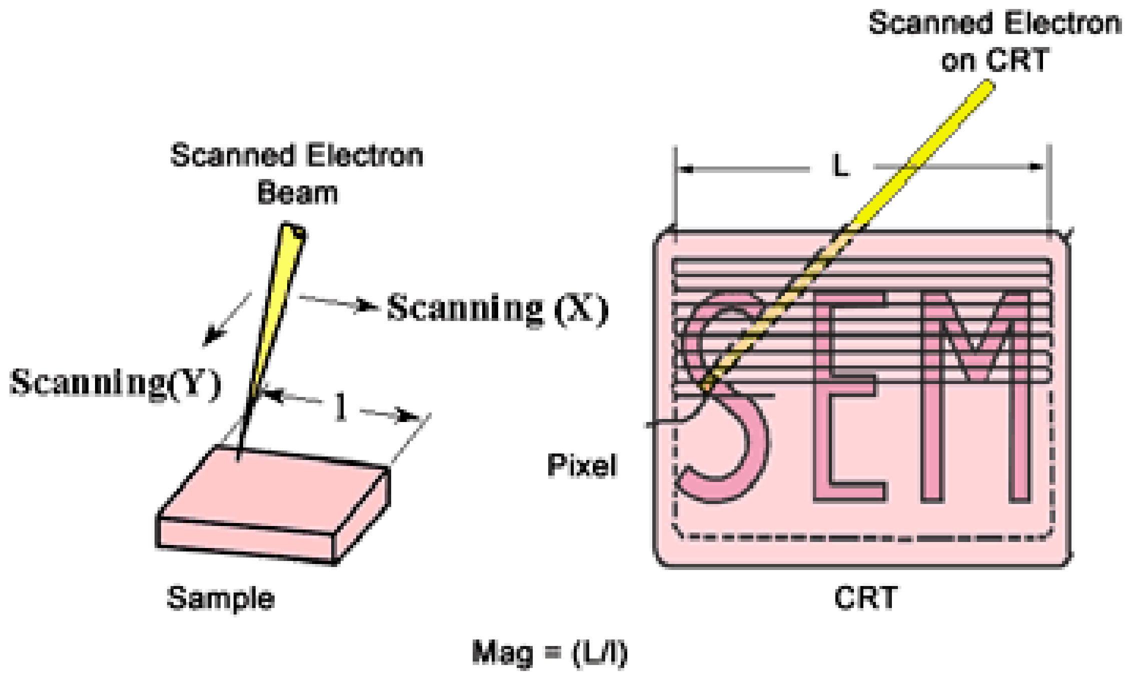
Scanning Electron Microscopy

The variety of electron induced signals that are produced in the SEM can provide

- Morphological (surface topography)
- Composition (chemical information)
- Physical Info (structural, electrical)

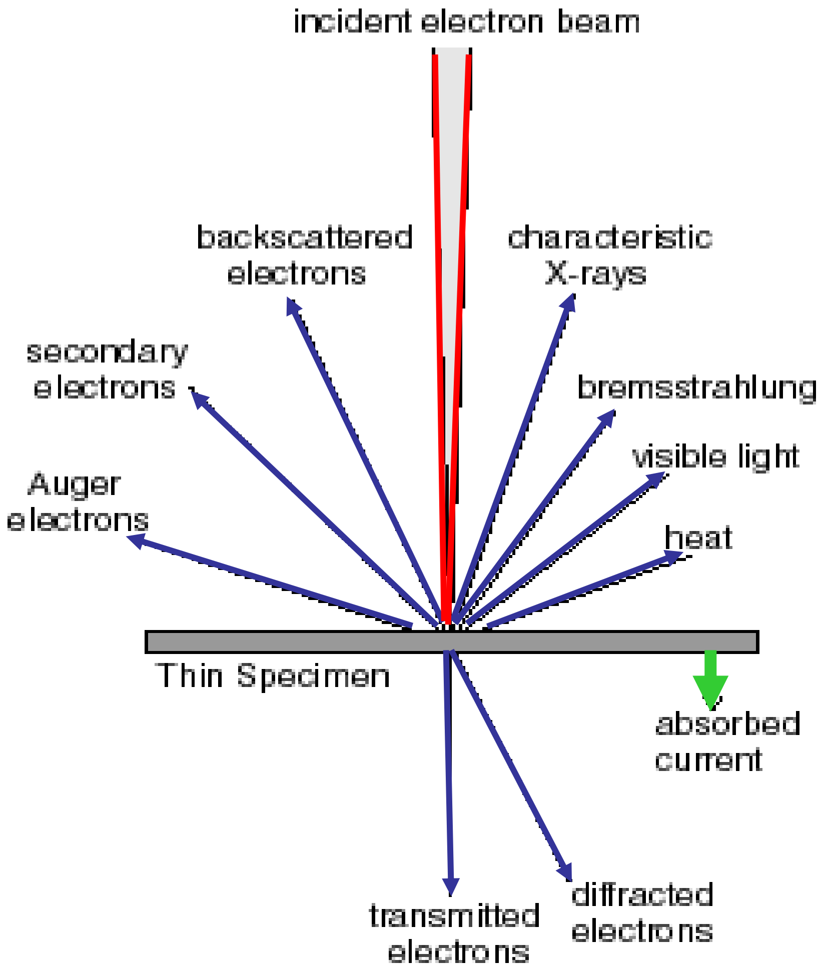
Scanning Electron Microscope

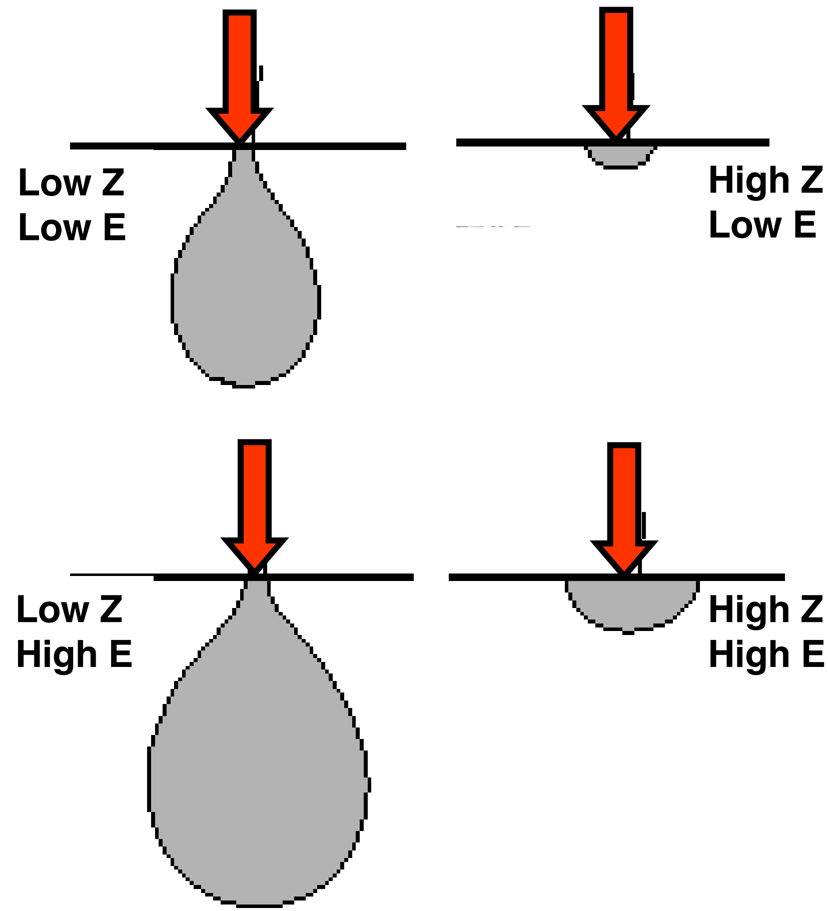




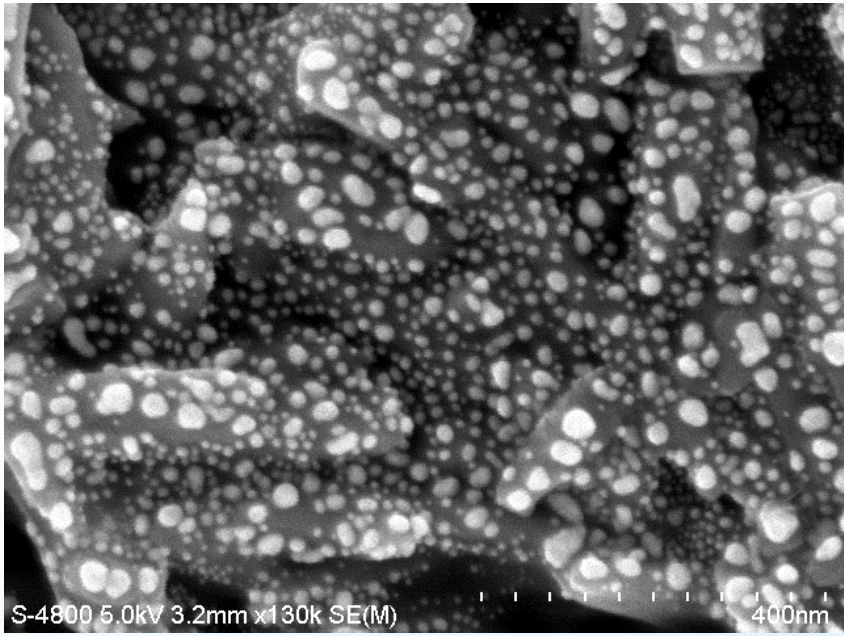
In SEM, an electron beam is focused to a fine probe which is scanned over the sample. As the beam is scanned, the beam interacts with the specimen to create several types of signals which can be detected. The detected signal is displayed on a CRT which is raster scanned in synch with the electron beam thus forming an image on the screen. The magnification of the image is a function of the ratio of the displayed image size over the scanned electron beam image size.

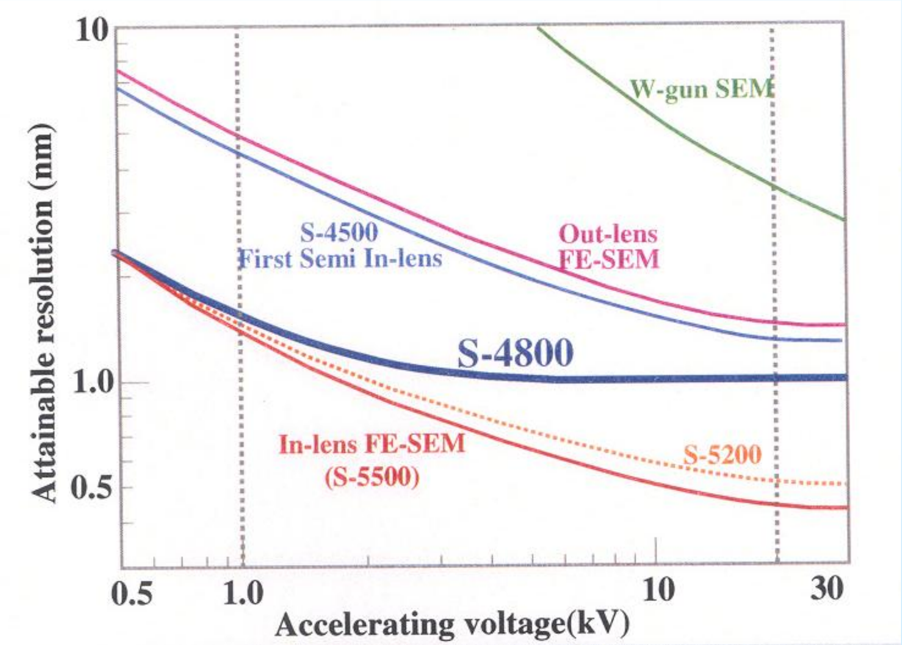
Electron Interaction with Solids





Resolution of SEMs



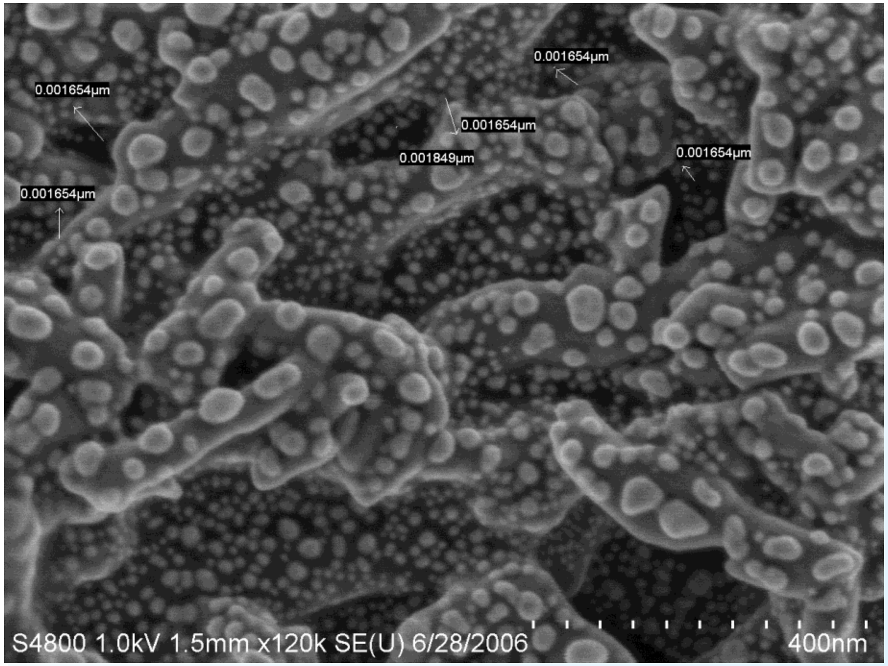


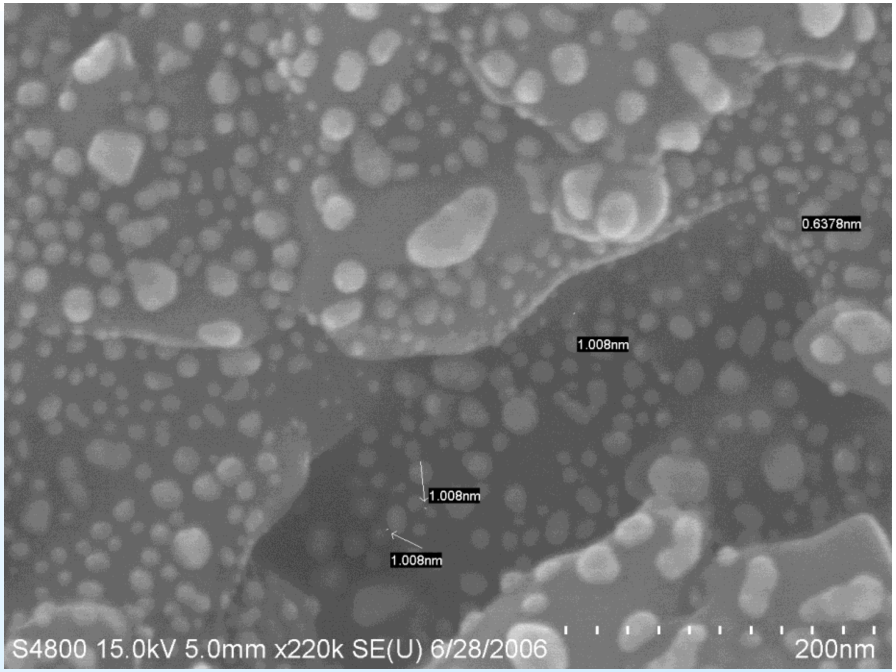
SEM provides high image magnification and resolution!

5.0 nm (50 Å) - common for commercial instruments

2.0 nm (20 Å) - for Advanced Research instruments

FESEM Resolution





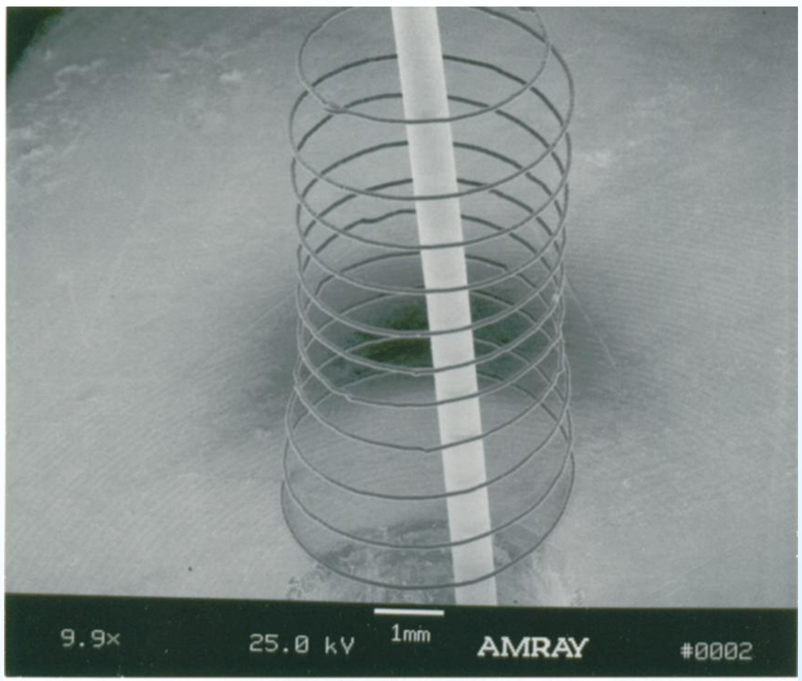
1.0 KV

15.0 KV

Field-emission SEMs are superior!

Resolution of 1.0 nm can be easily attained.

3-D View of SEM Images

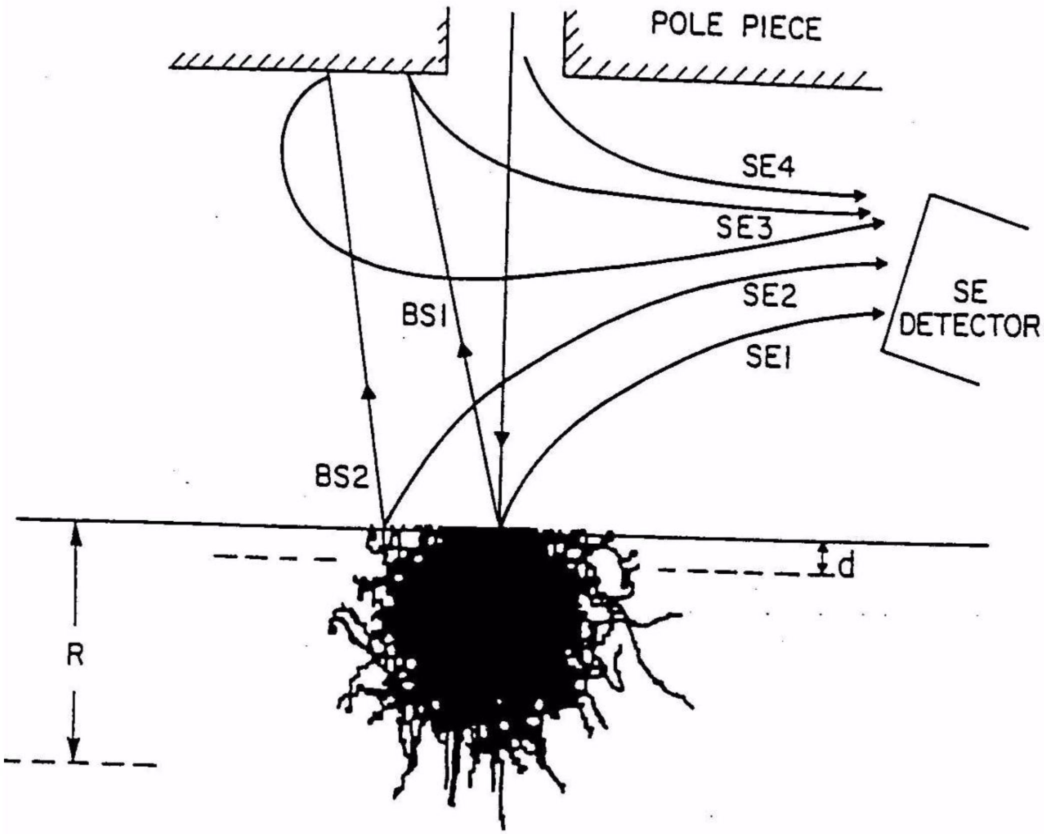


Large DOF provides more info about the specimen, and makes it easy to interpret the image.

Basic Components of SEM

- Electron gun assembly -
  This produces a stable source of primary electron beam.
- Electromagnetic lenses and apertures -
  These focus the electron beam on the specimen.
- Vacuum system -
  This allows the passage of the electrons through the column without the interference of air molecules.
- Electron collector, signal detection and display components, and recording CRT – Imaging.
- Specimen Goniometer Stage -
  moving the sample under the electron beam.

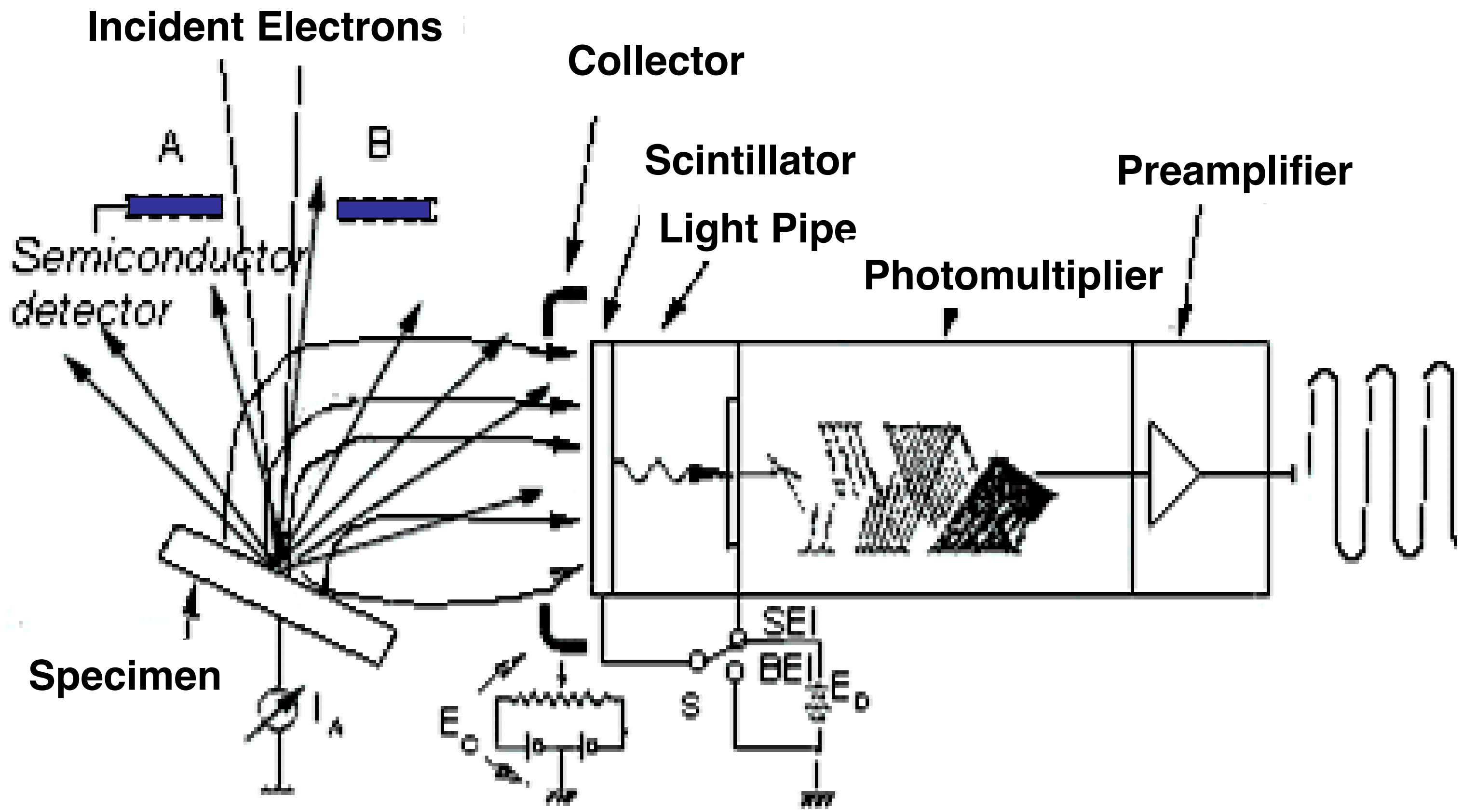
Origin of SE Emission



The size, sensitivity and position of the detector drastically affect its collection efficiency and the appearance of the image, and also the results of any measurements made from the image.

Metrology schemes must take into account the type & characteristics of the detector and their effect on the observed signal.

Secondary Electron Detector



Tuning Electron Current

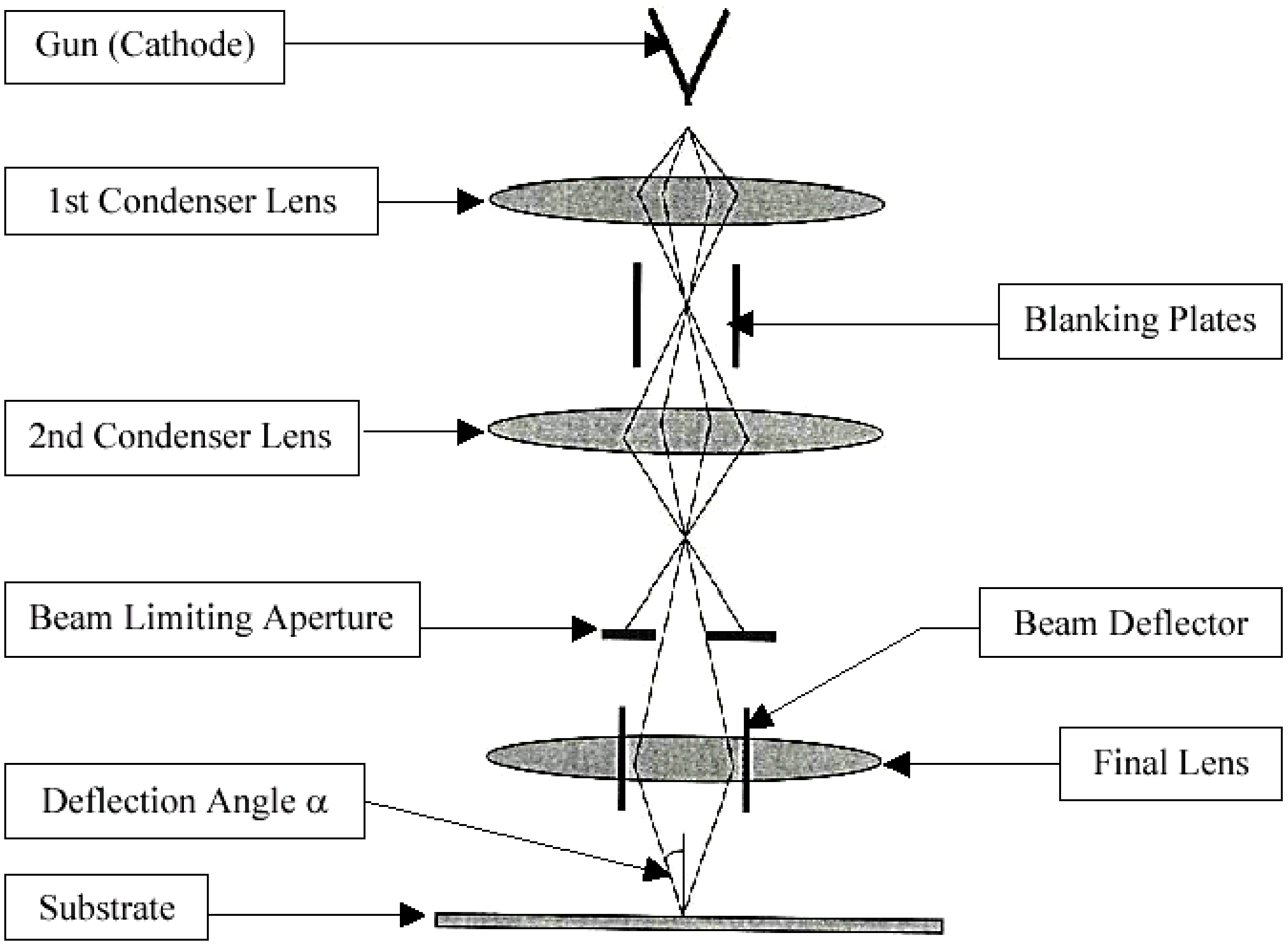
In SEM, a focused beam of high energy electrons scans across the surface of the specimen.

The amount of current in the focused e-beam impinging on a specimen, determines the magnitude of the signals emitted.

The size of the final probe or beam determines the best possible resolution for many of the measured signals.

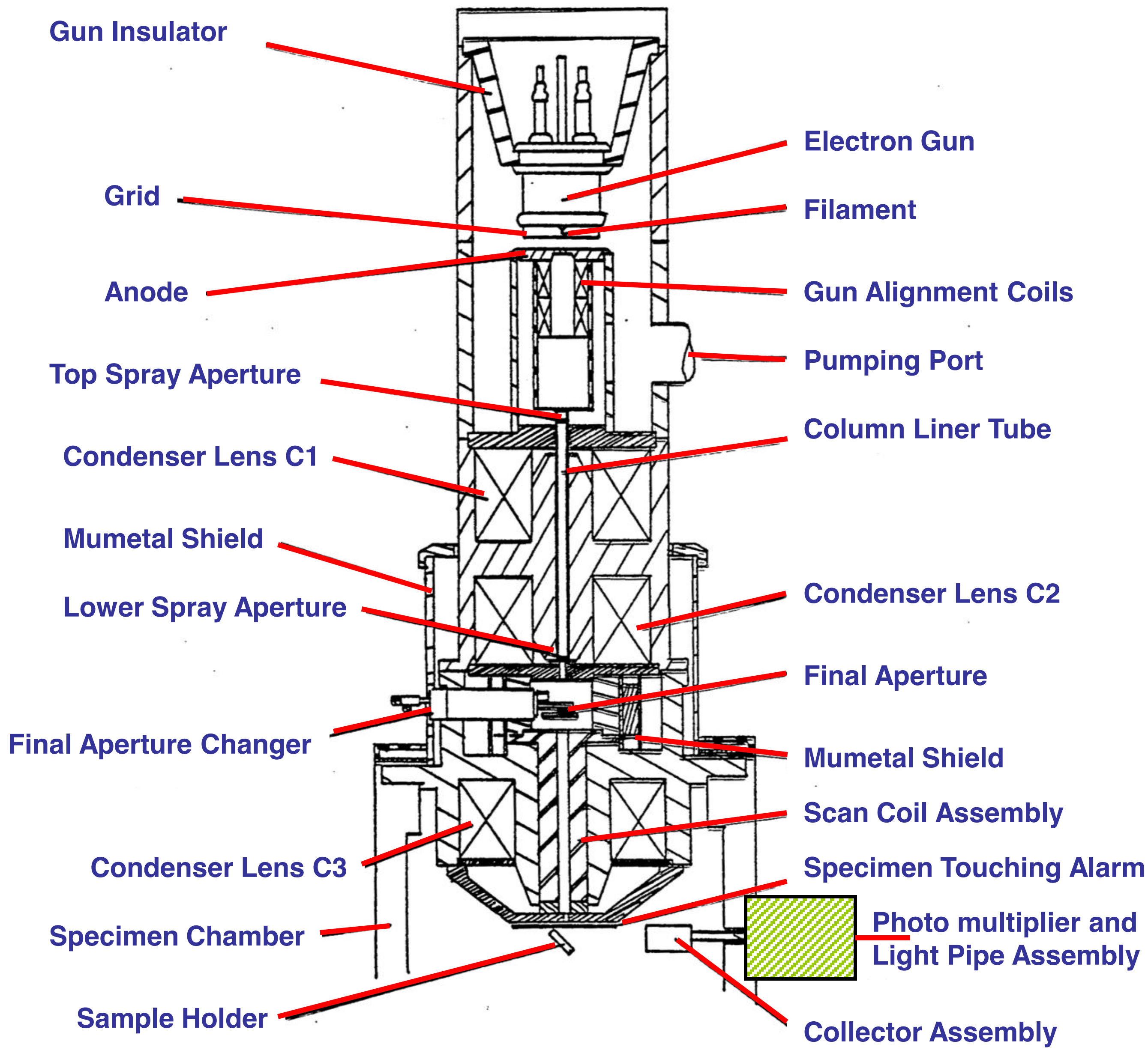
The electron optical system in SEM is designed so that maximum possible current is obtained in the smallest possible electron probe.

Scanning Electron Microscope



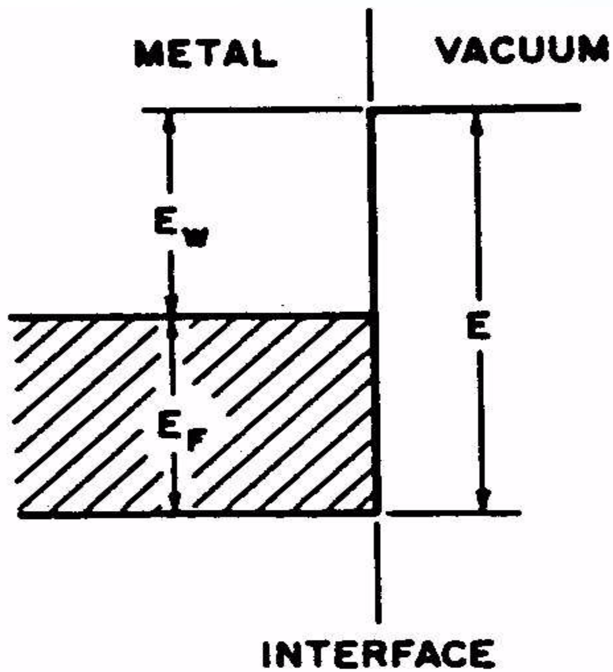
Schematic Representation – Electron Optical Column

Electron Optical Column



Concept of Work Function

Energy Model of Thermionic Emission



$$E_W = E - E_F$$

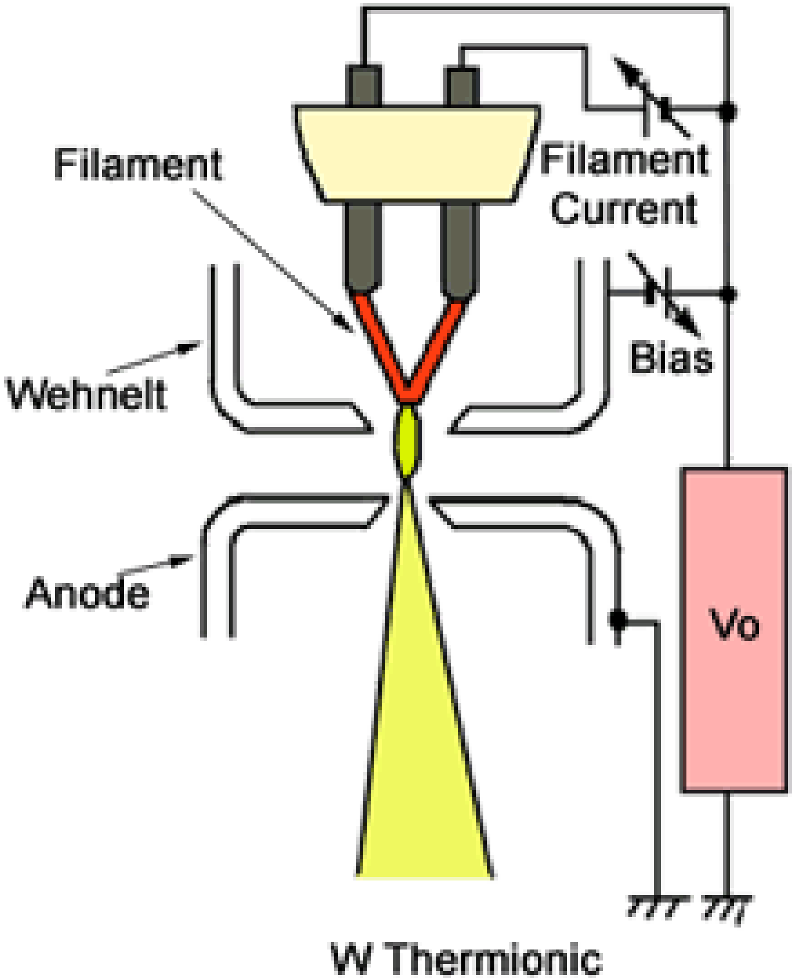
The emission current density is given by Richardson equation

$J_c = A_c T^2 \exp(-E_W/kT)$ A/cm^2

A_c - (A/cm^2K^2); T - (K)

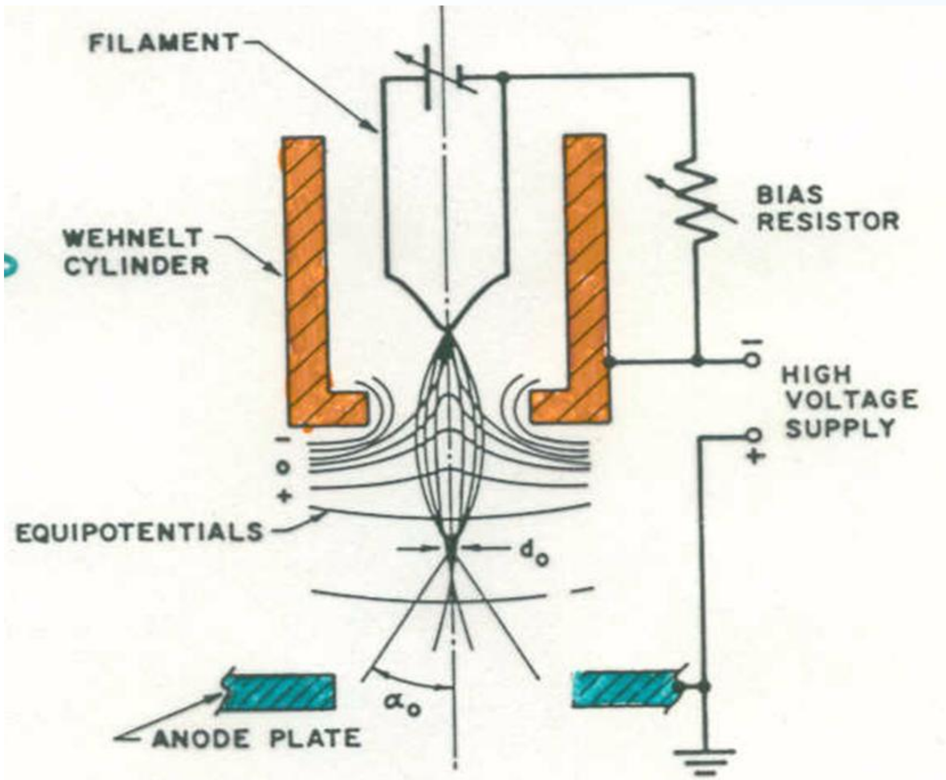
At sufficiently high temperatures, a certain % of the electrons become sufficiently energetic to overcome the work function (E_W) of the cathode material and escape the source.

Thermionic Emission Electron Source

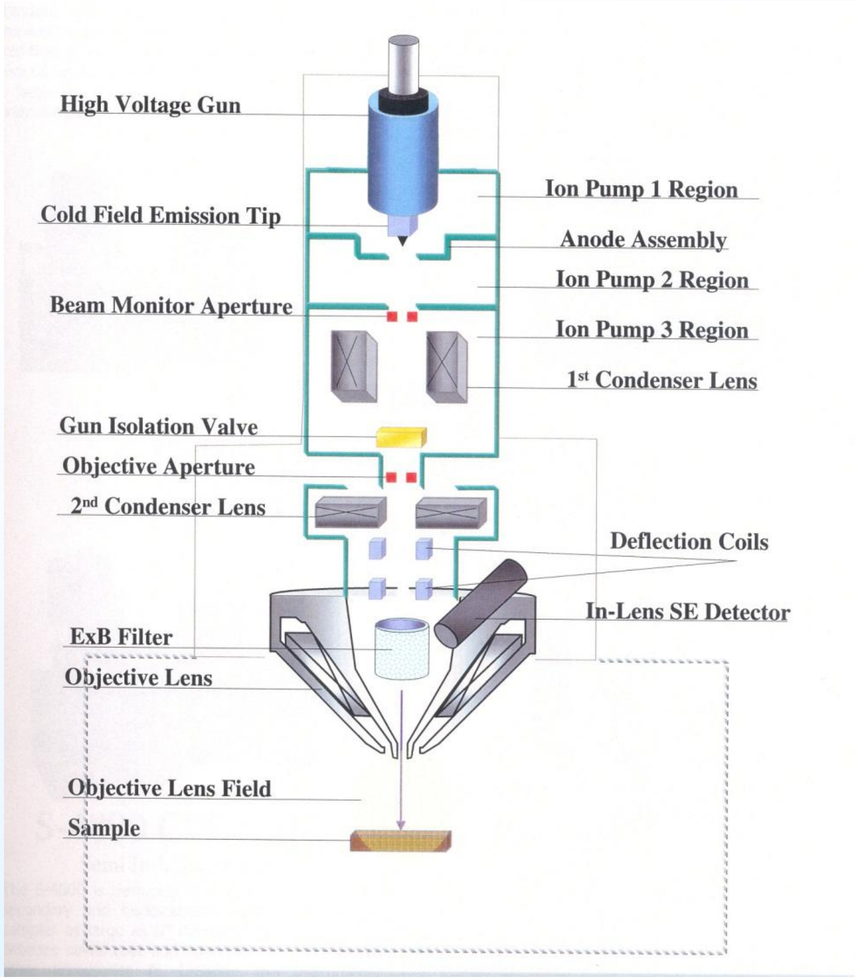


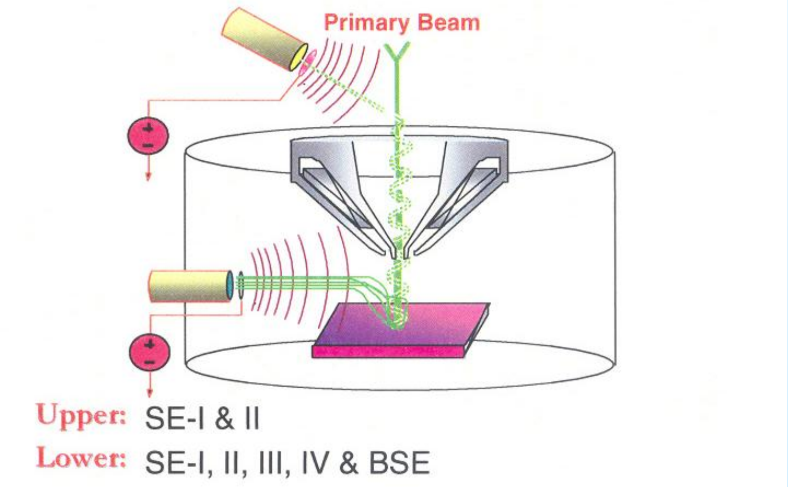
The most common emitter material for the electron beam is tungsten (W) or LaB6. The emitter is located at the top of the SEM column in the Gun assembly. The filament sits inside a housing called the Wehnelt cap. A potential is created between the sample and the gun and when the emitter is heated to a certain temperature electrons will begin streaming from the tip of the emitter and travel down the column. The electron beam is focused and defocused by the use of electromagnetic lenses.

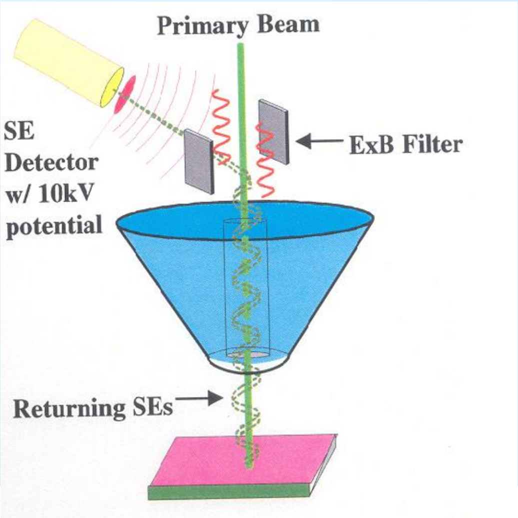
Configuration of Self-biased Gun
Wehnelt Cap Assembly



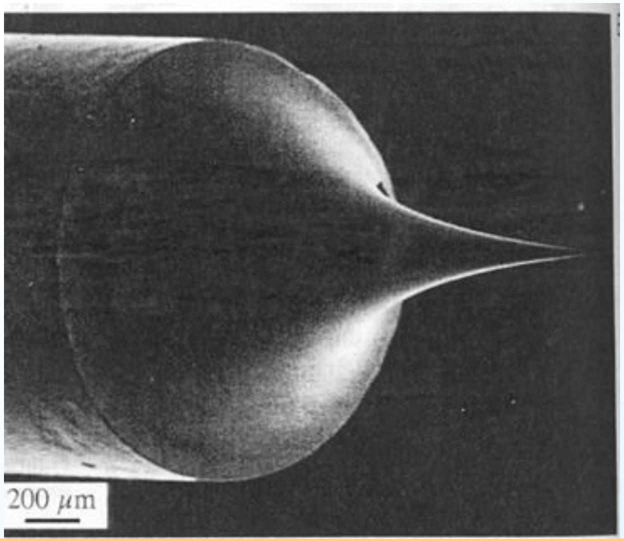
Cold-Cathode Field Emission SEM
S-4800 Column

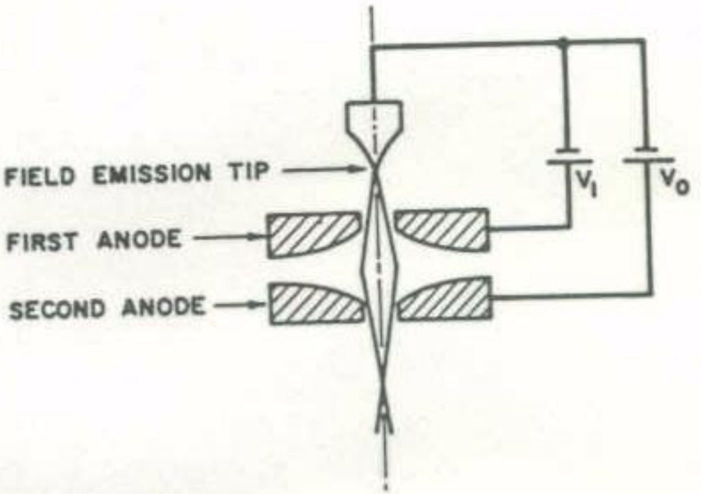






Field Emission Gun

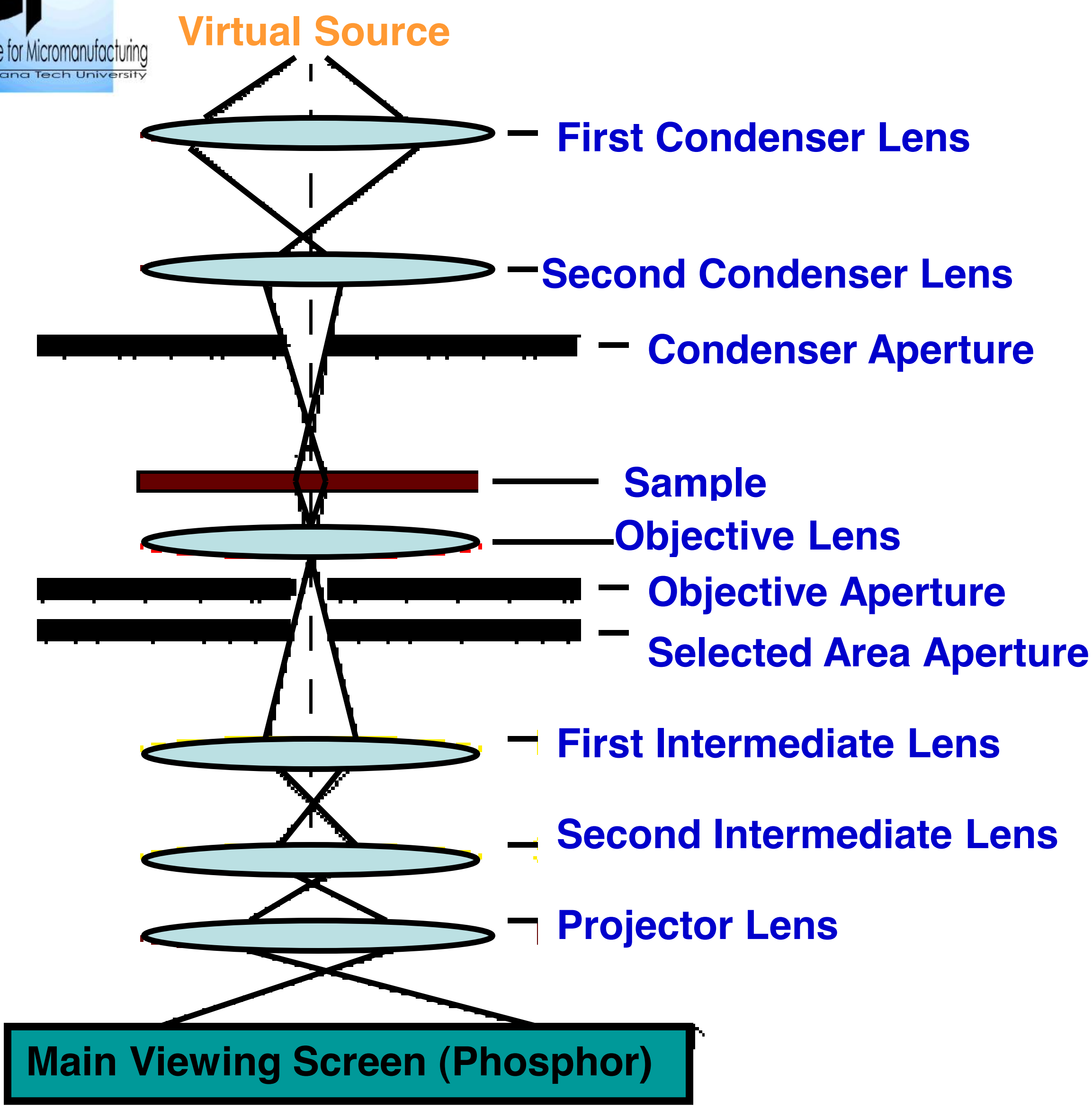




The effective source or crossover size (d_0) of a field emitter is approx. 10 nm, as compared to LaB_6 (10 μm), and W (50 μm).

No further demagnifying lenses are needed to produce an electron probe suitable for high resolution SEM.

Transmission Electron Microscope



A TEM works much like a slide projector. A projector shines a beam of light through the slide. As the light passes through, it is affected by the density of images on the slide. These effects result in only certain parts of the light beam being transmitted. This transmitted beam is then projected onto the viewing screen, forming an enlarged image of the slide. TEMs work in much the same way except that a beam of electrons (like the light) is transmitted through the specimen (like the slide). Whatever part is transmitted is projected onto a phosphor screen for the user to see.

Calculation of Current Density
Tungsten Cathode

The filament current necessary to reach the operating temperature, and to obtain filament saturation decrease with the age of the filament.

$J_c = A_c T^2 \exp(-E_w/kT)$ A /cm^2

If A_c = 60 A/cm^{2}K^2

E_w = 4.5 eV; k = 8.617 x 10^{-5} eV/K

T = 2700 K

J_c = ?

Beam Current Density

The J_c in the e-beam at crossover point represents the current that could be concentrated into a focused spot on the sample, provided there is no aberration in the electron lenses.

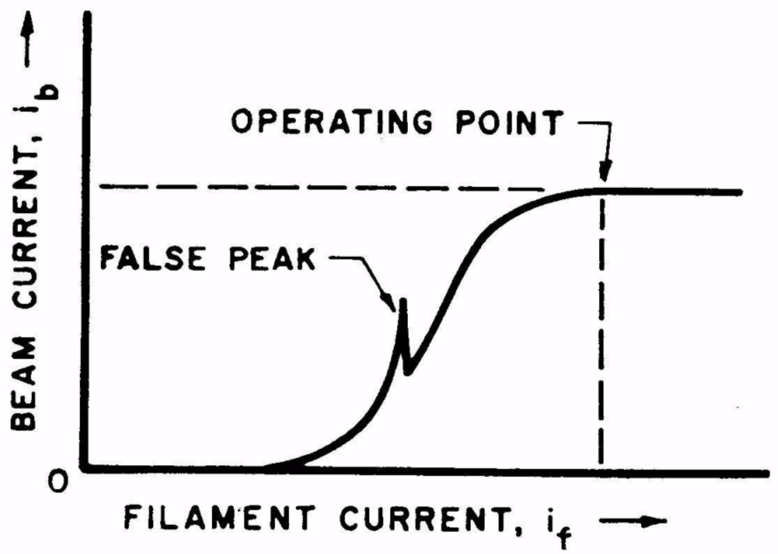
The J_b is the maximum intensity of electrons in the beam at the crossover, can be defined as

$$J_b = \frac{i_b}{\pi(\frac{d_o}{2})^2}$$

i_b- is the total beam or emission current measured from filament

Beam current usually varies between 100-200μA.
In practice, maximum current density in the final image is desired.

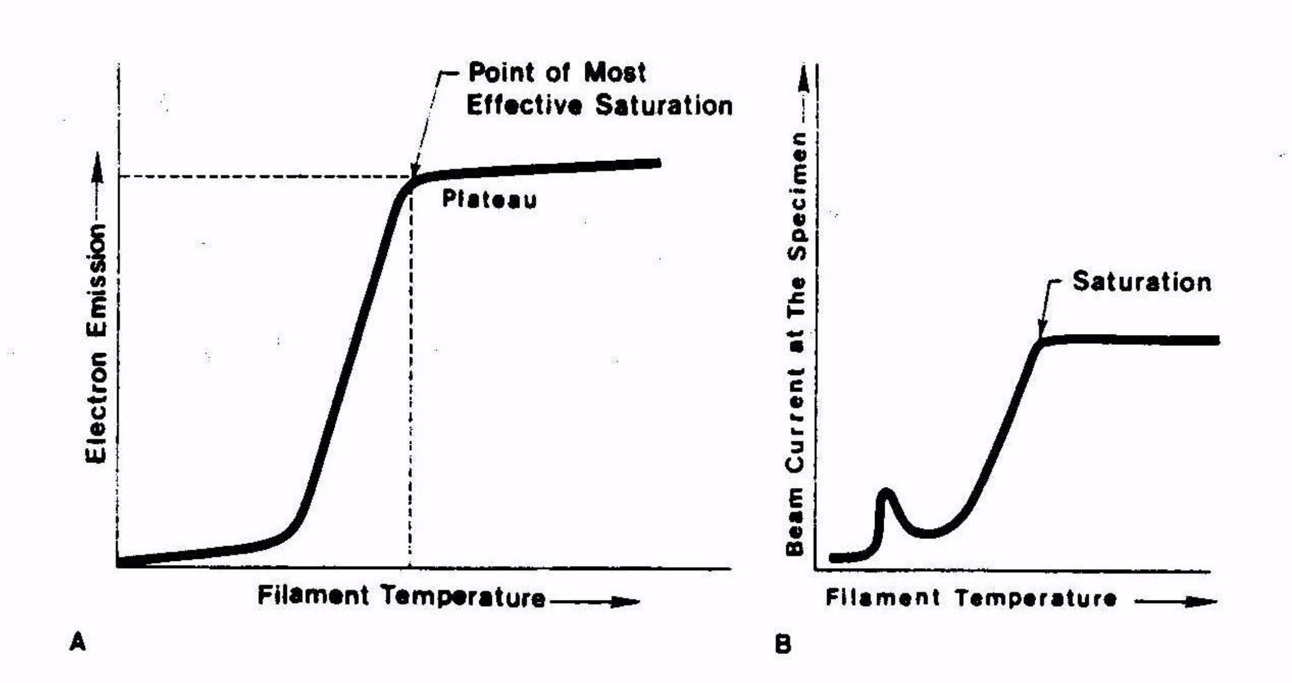
Emission Characteristics of Self-Biased Electron Gun



It is important to obtain a stable well-regulated beam current.
Saturation allows to achieve a self-regulating gun and a stable beam current.

When the i_f is increased above that necessary for emission, the bias voltage also increases causing the negative field gradient around the filament to increase, and limiting the rise in i_b.

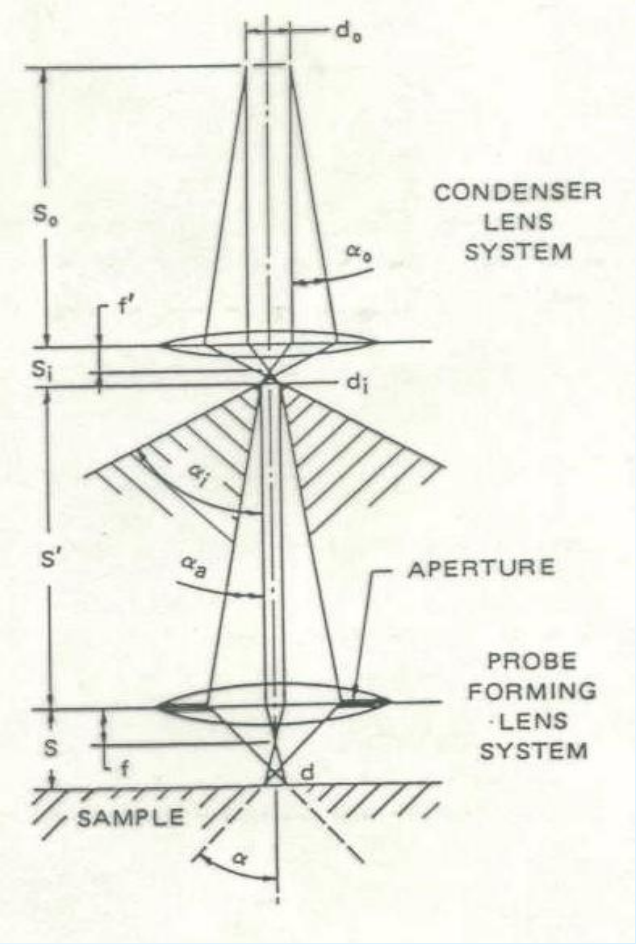
Saturation of Filament Current



At the most effective point of saturation, the highest quantity of electrons are generated for the least amount of current.

As the current flowing through the filament increases, the number of electrons emitted increases up to a specific point called Saturation Point.

Minimizing Spot Size



Reduction of the e-beam diameter at the crossover point to a focused electron probe

As the current in the condenser lens is increased, the magnetic field increases and the focal length of the lens decreases

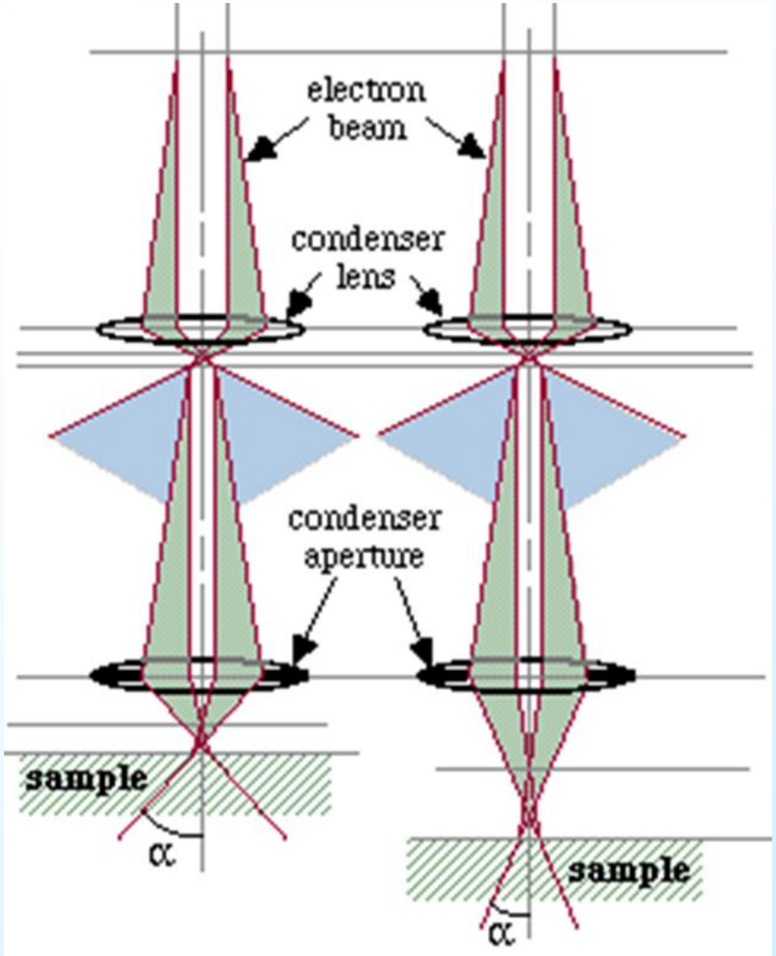
The demagnification of the lens

$M = S_0/S_i = d_0/d_i$

The divergence angle α_i of electrons

$\alpha_i = \alpha_0 . M$

Demagnification as a function of Working Distance



When the working distance (S) increases, the demagnification **(S'/S) decreases, and** the resulting spot size on the specimen will be large.
The divergence angle (α) is also reduced.

Brightness of Electron Beam

The maximum usable divergence angle of the focused electron beam is fixed by the aberrations of the final lens.

The most important performance parameter of the electron gun is the current density per unit solid angle.

The brightness (β) of the e-beam,

$$\beta = \text{Current}/(\text{area})(\text{solid angle})$$

$$\beta = \frac{4i}{\pi^2 d^2 \alpha^2} \quad \text{A/cm}^2\text{Sr}$$

Brightness of the Beam

The brightness of the electron beam has a maximum value, and it can be calculated based on Langmuir equation.

$$\beta = \frac{J_c e E_0}{\pi k T}$$

A/cm^{2}Sr

The brightness of the filament can be increased by increasing either the E_0 (high voltage) or the J_c (the current density at the cathode).

Improved brightness would provide either increased current for the same beam size, or a reduced beam diameter for the same current.

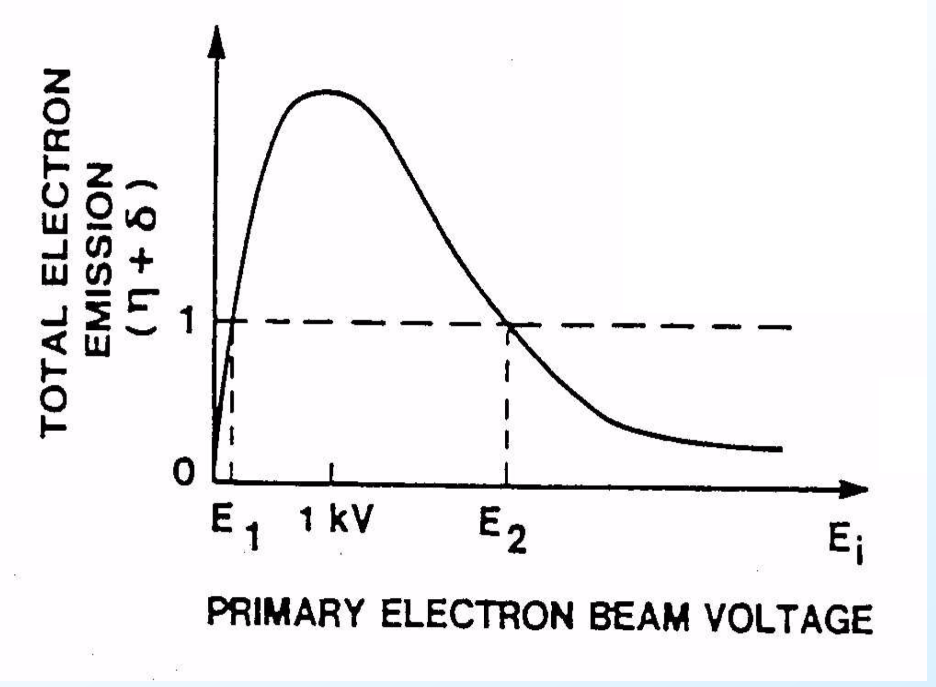
Brightness of the Beam

The brightness can be calculated as

$\beta = 11600\ J_c E_0/\pi T$

where the units for J_c is A/cm^2 and for E_o is V.

Electron Emission Yield



For an insulating sample:

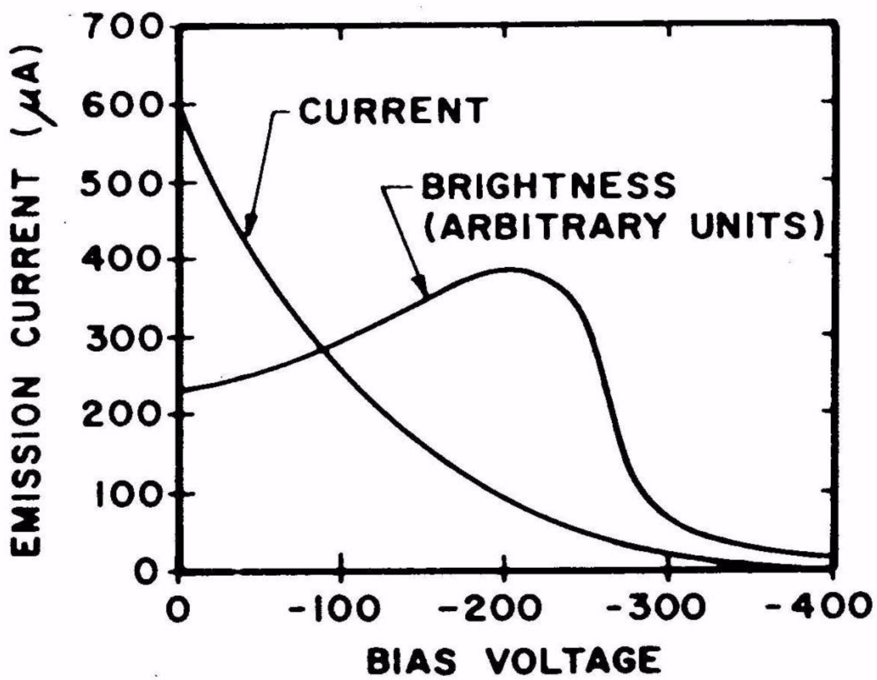
$E_1 \leq E \leq E_2$

$E > E_2$, $i_{sec} < I_{primary}$

Sample becomes negative
This may distort image!

The total electron emission includes both back scattered electrons and secondary electrons.

Relationship of Emission Current and Brightness to Bias Voltage



At low bias, the –ve field gradient is weak, and the e-ns will sense a +ve field or voltage gradient towards the anode. So, the emission current is high. Since little or no focusing occurs at low bias, the crossover dimension is large, and the brightness is not optimum.

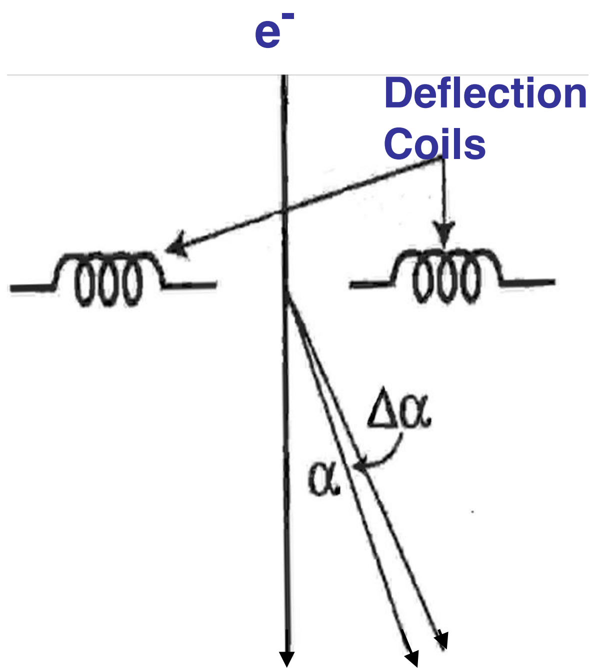
If the filament-Wehnelt cylinder distance can be changed, then the shape of the constant field lines can also be changed.
Bias resistance also influence the field!

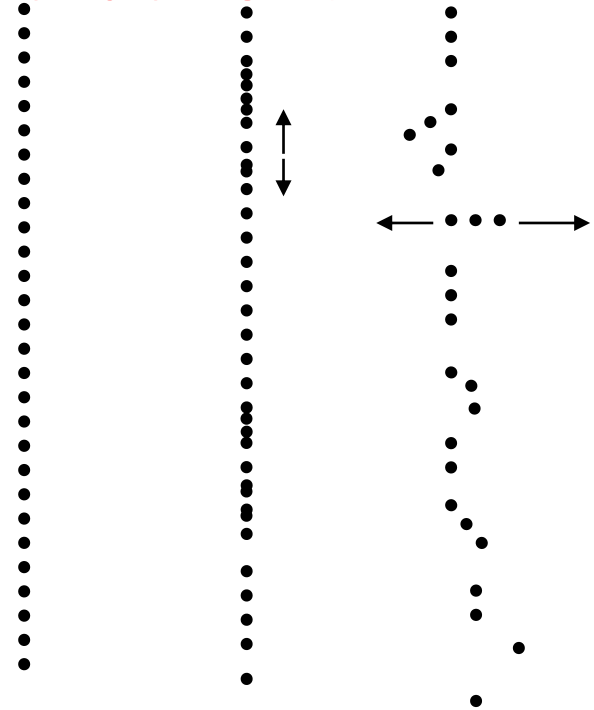
Brightness of the beam from Different Sources

COMPARISON OF ELECTRON SOURCES (AFTER WELTER, 1975)

	Tungsten Filament	Lanthanum Hexaboride	Field Emission
Type of Emission	Thermionic	Thermionic	Field
Operating vacuum (torr)	10^{-4} - 10^{-5}	10^{-6} - 10^{-7}	10^{-9} - 10^{-10}
Brightness (A/cm²•ster)	10^{4} - 10^{5}	10^{5} - 10^{6}	10^{7} - 10^{9}
Source Size (Å)	1×10^{6}	2×10^{5}	$<1 \times 10^{2}$
Energy Spread (eV)	1-5	0.5-3	0.2-0.3
Probe Current Stability (% per minute)	0.1-1.0	0.2-2.0	2-10
Operating Life (hrs.)	>20	>100	>300

Electron – Electron Interactions





Transverse chromatic aberration due to the energy spread of source electrons

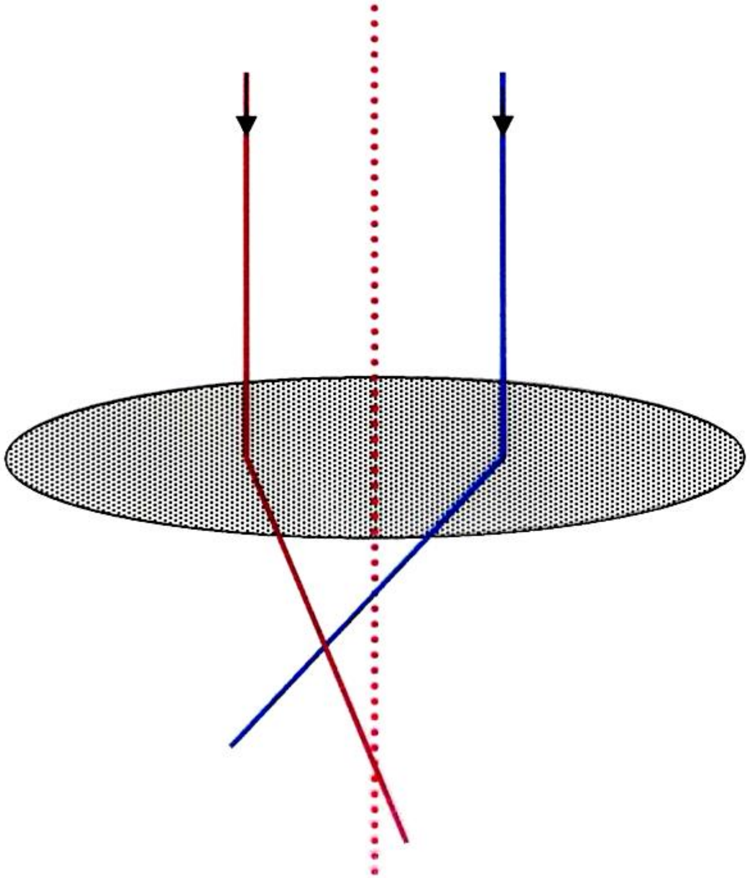
Statistical effects of electron - electron interactions

$$\Delta\alpha = \frac{1}{2}\frac{\Delta V}{V}\alpha$$

Where α = deflection angle

ΔV = Energy spread of electrons which is about 2 eV for tungsten, 1 eV for LaB_6 and 0.2 to 0.5 eV for a field emitter.

Chromatic Aberration



- Faster (higher energy) electrons focused less strongly than slower electrons

$$d_c = C_c \alpha \frac{\Delta V}{V}$$

- Aberration coefficient is roughly equal to the focal length of the lens

d_c = Chromatically limited Beam Diameter

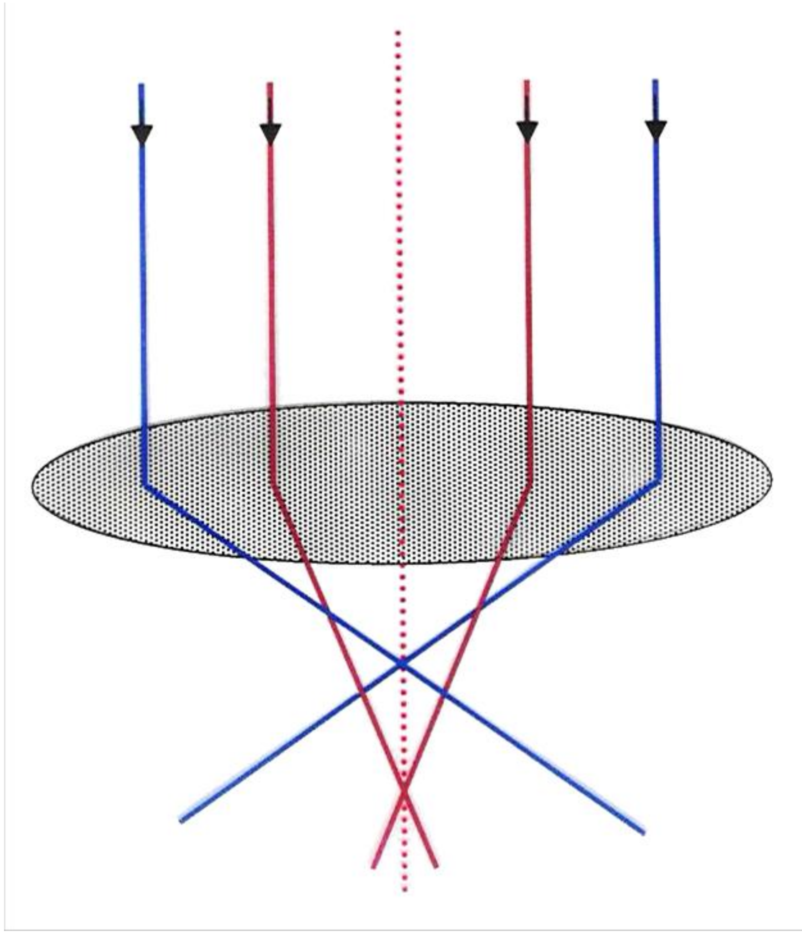
C_c = Chromatic Aberration Coefficient of the Final Lens

α = Convergence Half Angle of the Beam at the target

V = Beam Voltage

ΔV = Energy Spread

Spherical Aberration



- Off-axis electrons focused more strongly

$$d_s = \frac{1}{2} C_s \alpha^3$$

- Aberration coefficient is approximately equal to the focal length of the lens

d_s = Beam Diameter

C_s = Spherical Aberration Coefficient of the Final Lens

α = Convergence Half Angle of the Beam at the target

Advantages and Disadvantages of SEM over Optical Microscope

Advantages

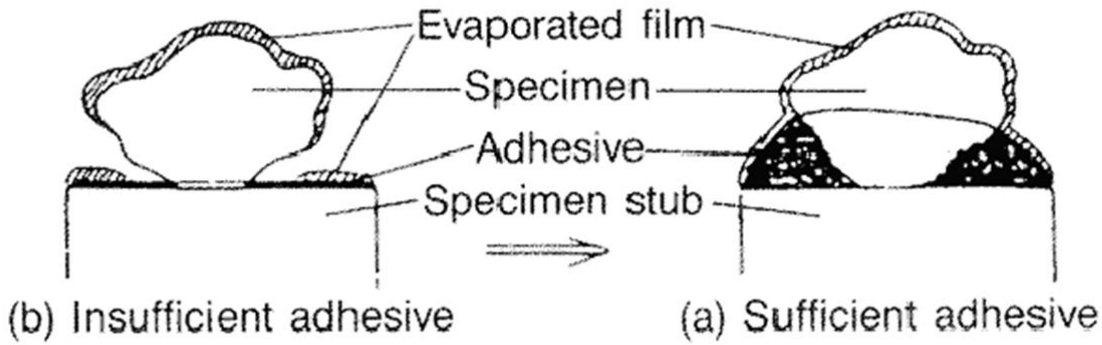
- High Resolution (2-20 nm)
- High Depth of Field
- Flexible Viewing Angles
- X-ray Characterization
- Readily Interpreted Image

Disadvantages

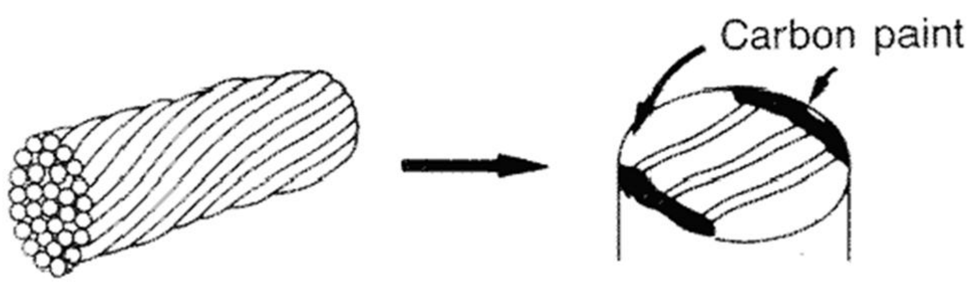
- High vacuum is required
- Lower throughput
- E-beam/sample interactions
- Sample Charging
- Linewidth standard problem
- Expensive Instrumentation

The increased resolution is due to shorter λ of electrons which helps to circumvent the diffraction effects prevalent in the optical microscope.

SEM Sample Preparation

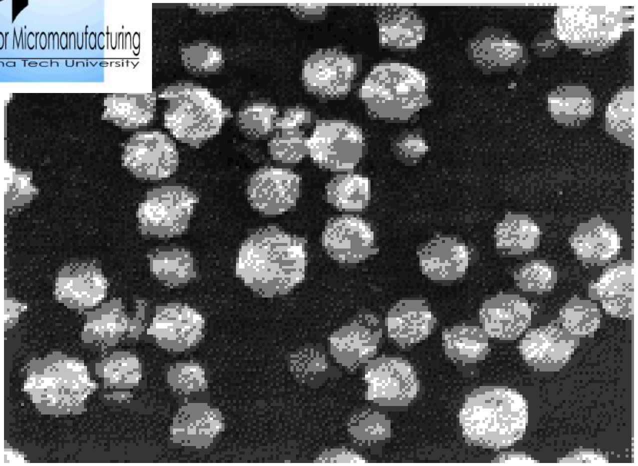


Fixing of a bulk specimen

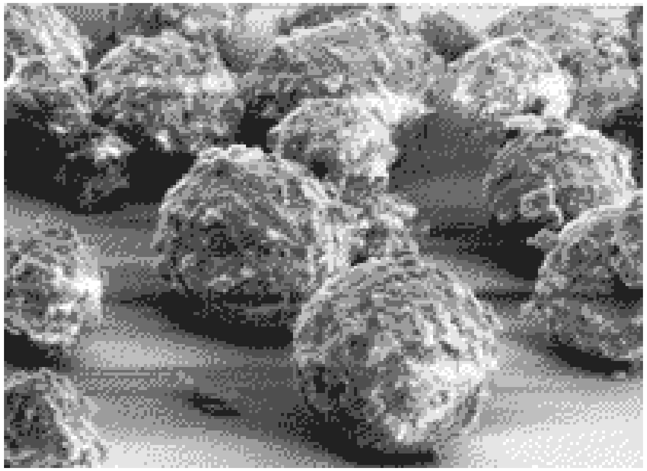


Fixing of fiber

Sample Preparation – Toner Powder

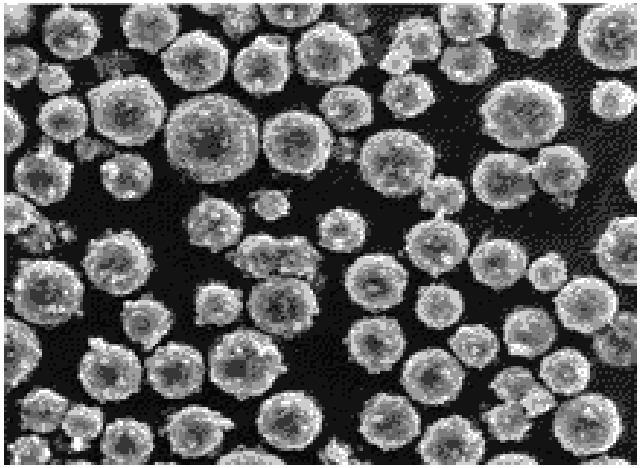


15 kV Tilt = 0 degrees 720 X

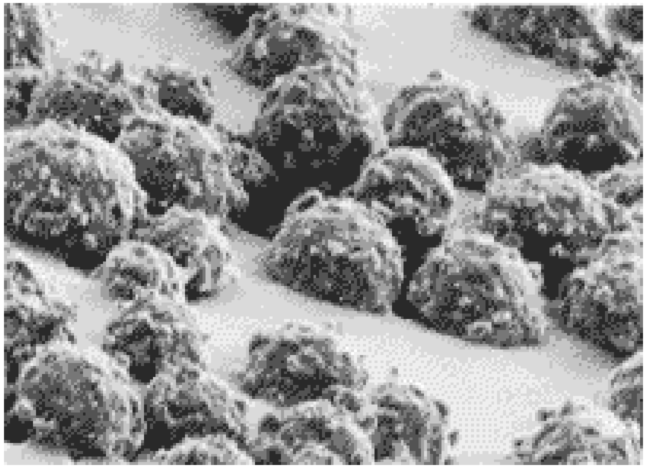


5 kV Tilt = 45 degrees 1,400 X

Sample charging due to insufficient electrical contact to sample holder



5 kV Tilt = 0 degrees 720 X



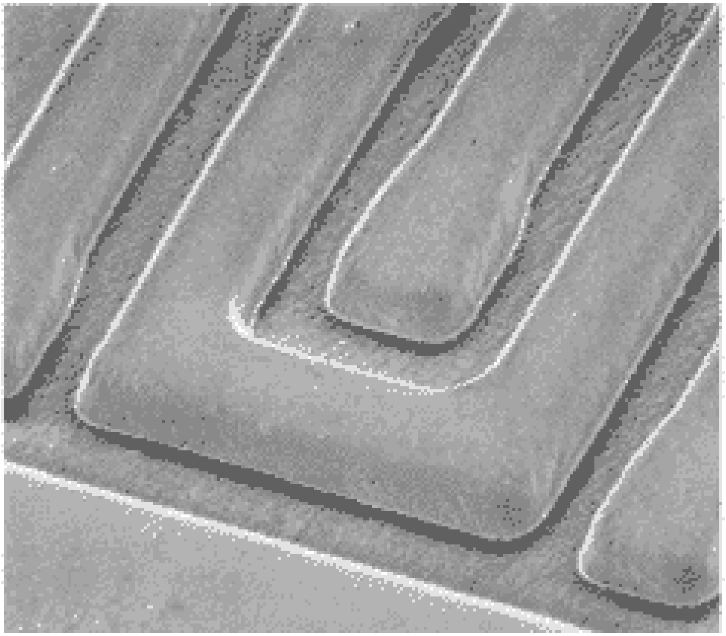
5 kV Tilt = 45 degrees 1,400 X

Properly prepared sample

Influence of Sample Charging on Image Quality

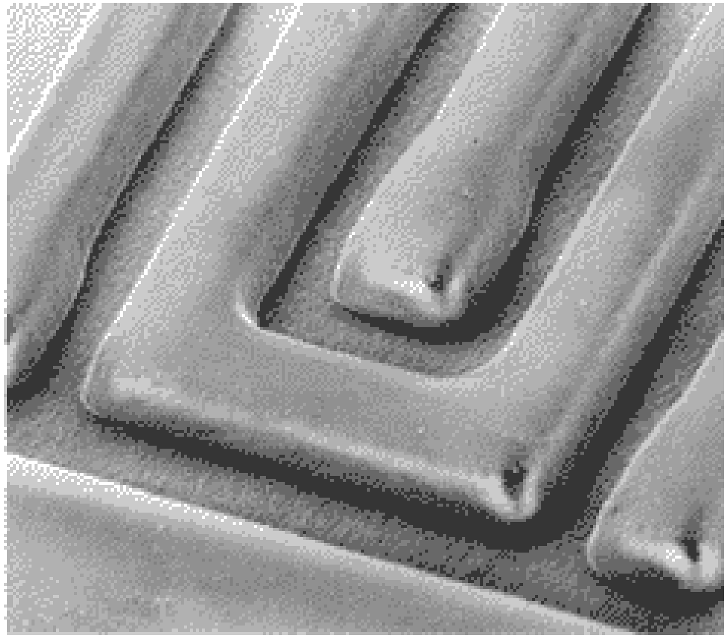
Non-conductive Material

Image with No Surface Charging



1.0 kV 3,200 X

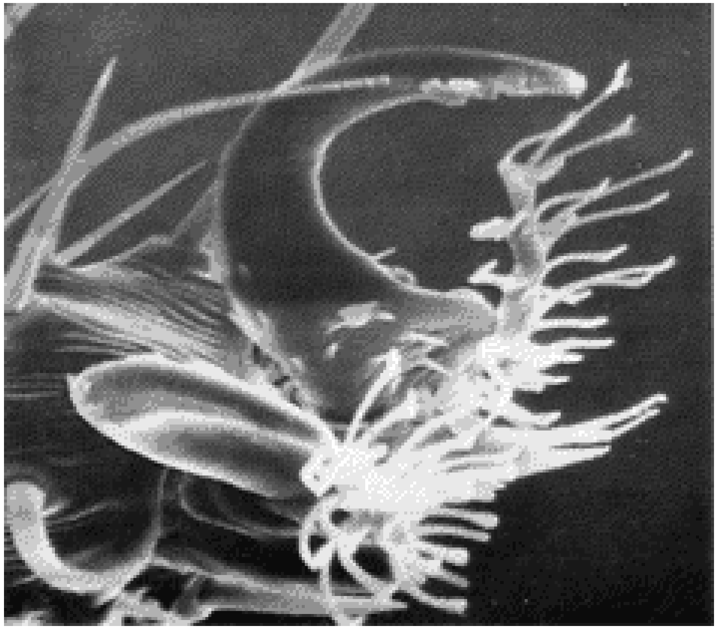
Image with Surface Charging



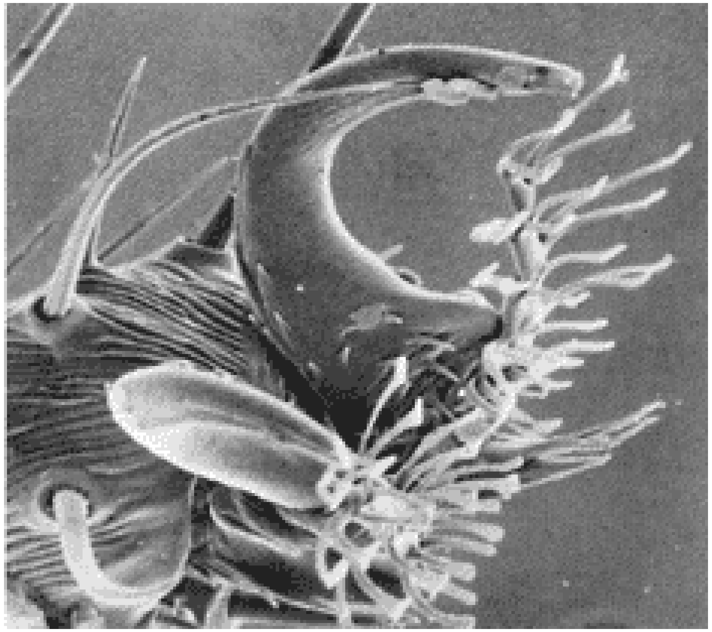
1.3 kV 3,200 X

Biological Samples

Vinegar Fly Foreleg



10 kV

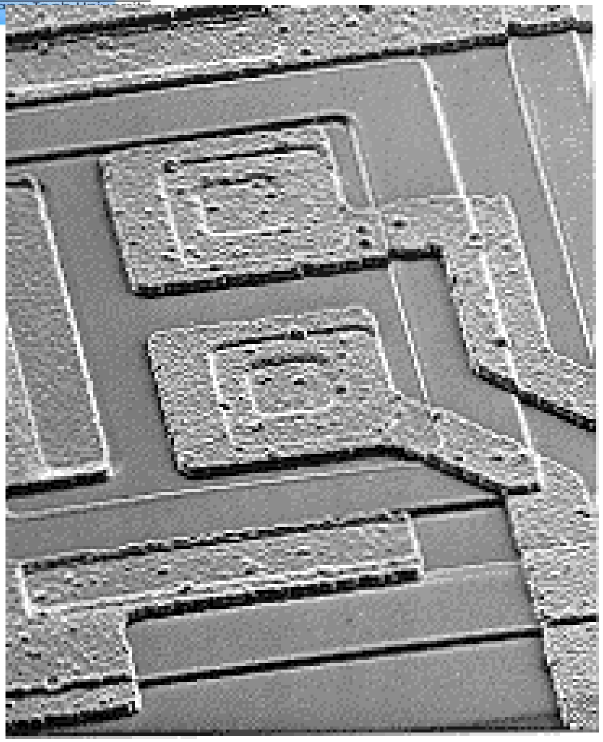


4 kV

Sample charging causes extremes in image contrast at 10kV reducing detail

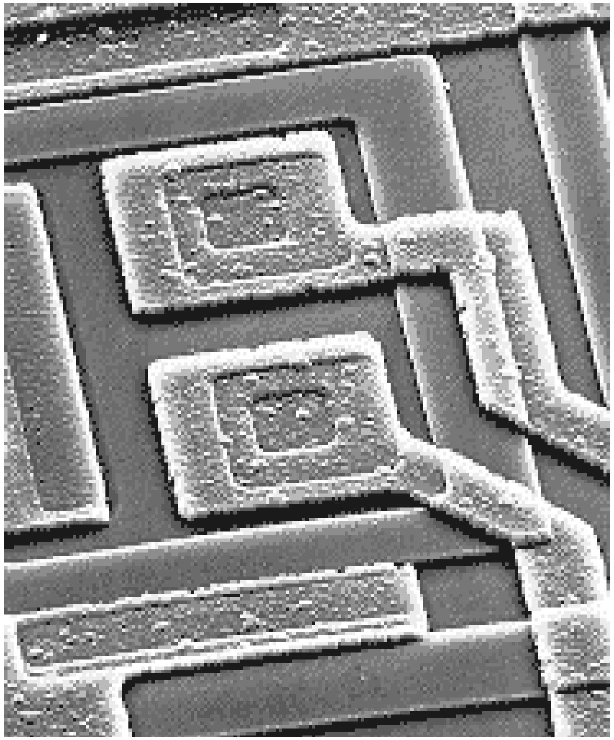
Reducing beam voltage to 4 kV equalizes image contrast and improves detail

Effect of Surface Topography on Observed Image



5 kV 720 X

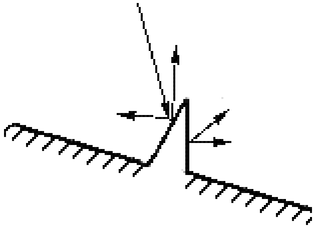
Tilt Angle = 50°



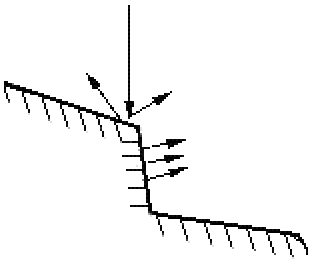
25 kV 720 X

Tilt Angle = 50°

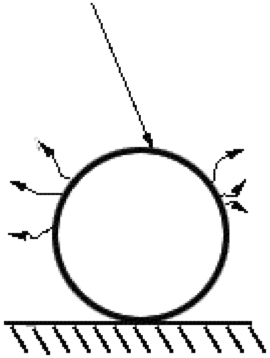
Electron penetration is increased at higher beam energy



(a) Protrusion

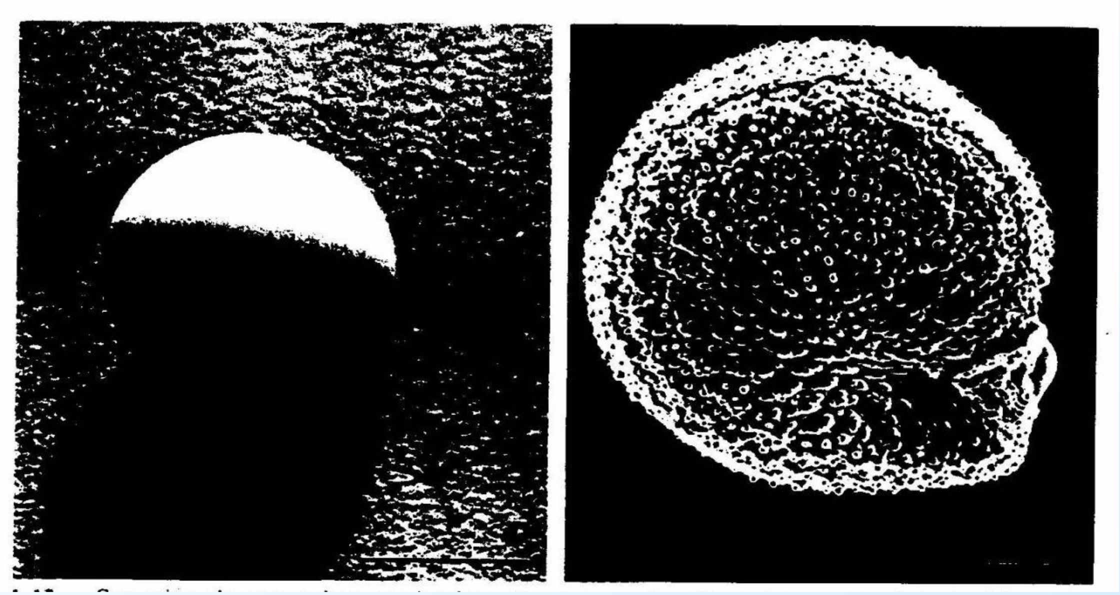


(b) Edge



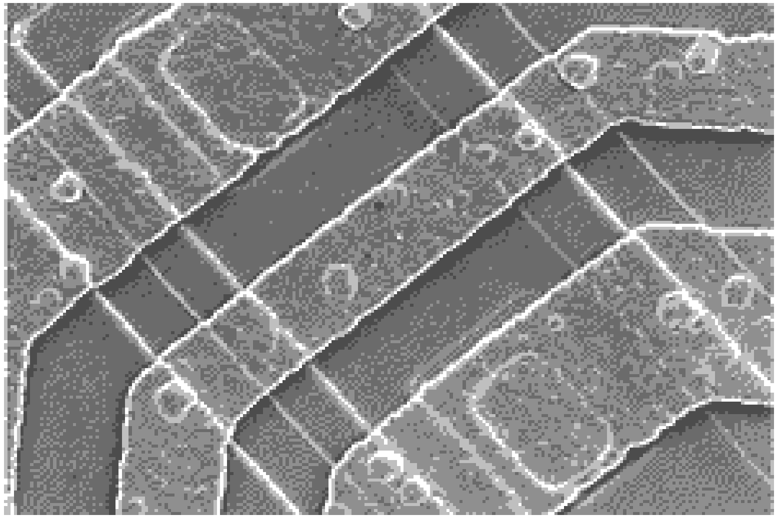
(c) Circumference

Bright and Dark Spots in SEM Images

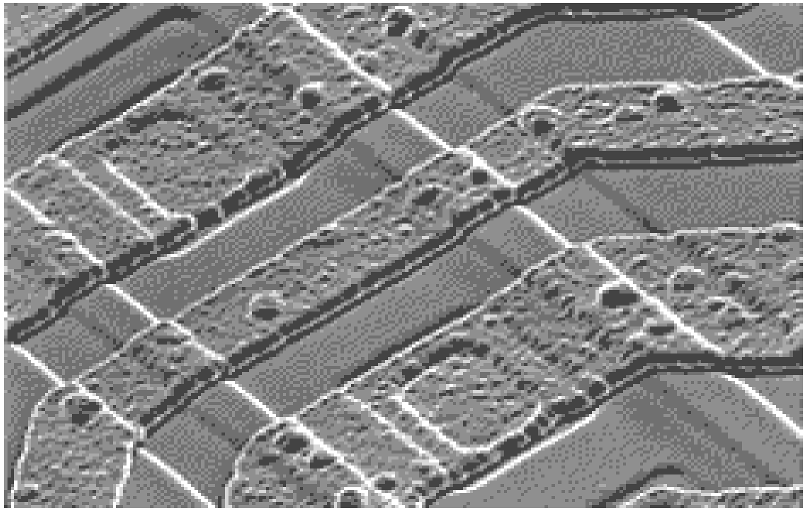


The electrons emitted from the surface which faces away from the detector are partially blocked by the specimen, and the image of such a surface is darker than that of a surface which faces toward the detector.

Effect of Sample Orientation on Observed Image



Tilt Angle = 0^o



Tilt Angle = 45^o

IC Chip

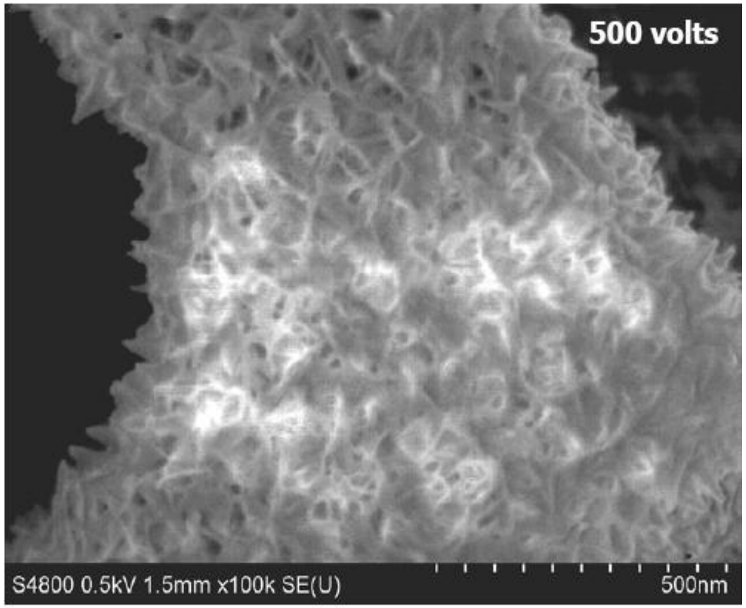
5 kV and 1,100 X Magnification

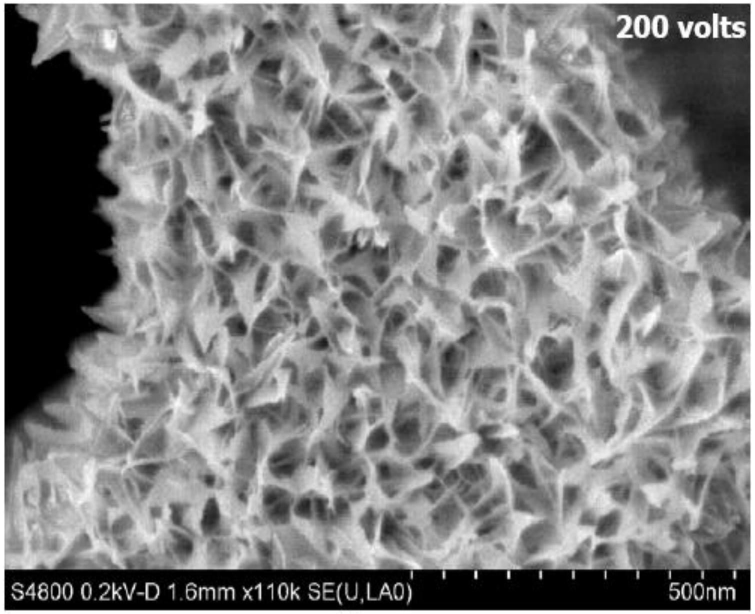
The sides of patterns are viewed by tilting the specimen. The amount of secondary electron electrons that are collected is increased.

Influence of Sample Charging on Image Quality

Institute for Micromanufacturing
Louisiana Tech University

Non-conductive Material

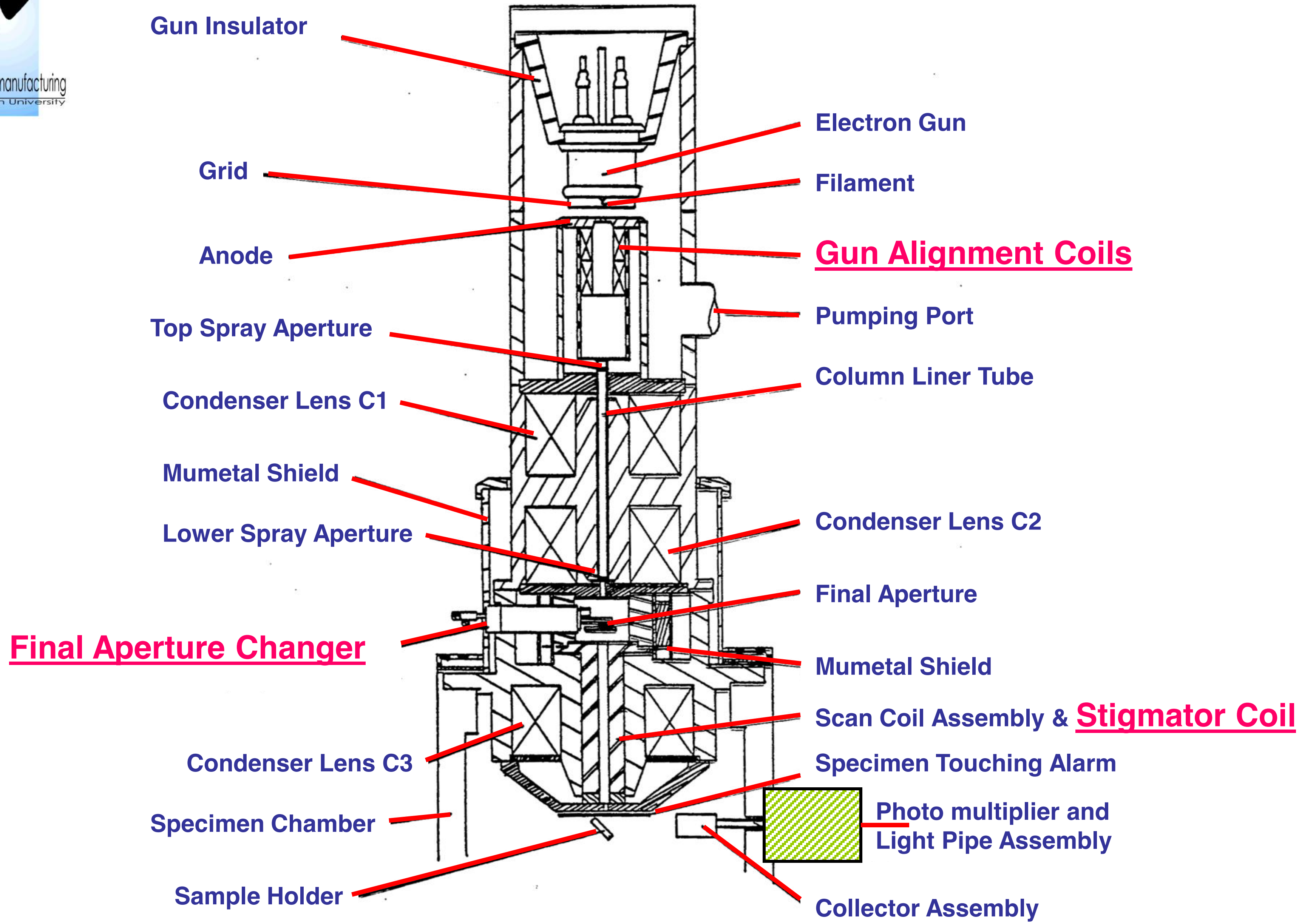




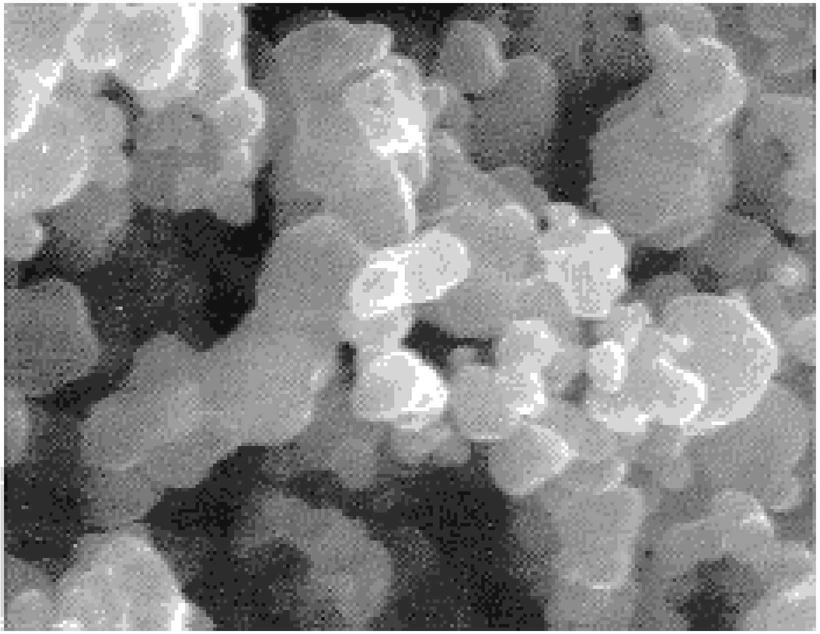
Aluminum Oxide

Imaging at ultra low voltages (100-500 volts) provides the ideal solution. The 200-volt image demonstrates a reduction in charging and enhanced surface information without the additional time needed for coating or a compromise in signal to noise. Charge effects no longer obscure the flake-like surface features and a true feel for the surface structure is understood. With the absence of charge the image is captured using a slow scan, high resolution image collection. Within the ultra low voltage range, specimens that were previously difficult to examine are no longer challenges to image.

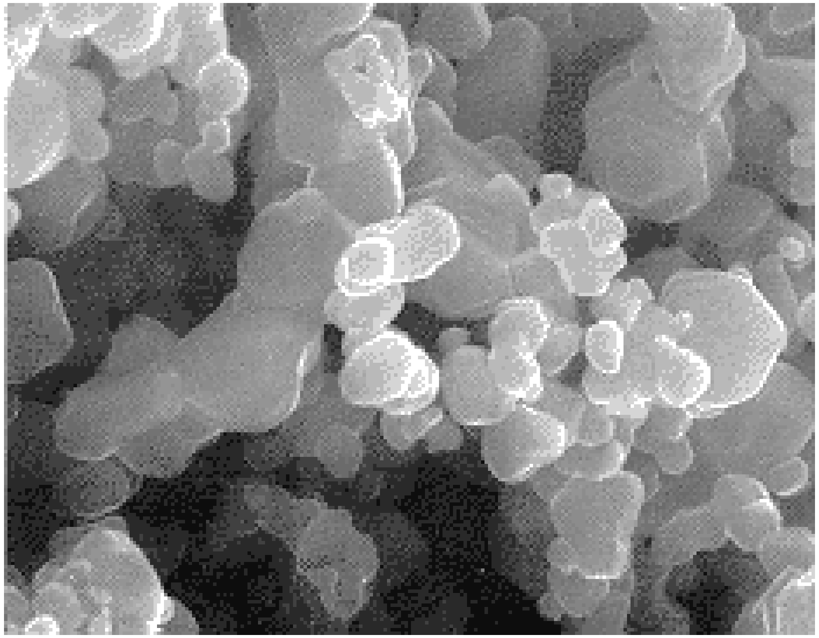
Electron Optical Column – Beam Alignment



Influence of Beam Alignment on Image



Incorrect Alignment

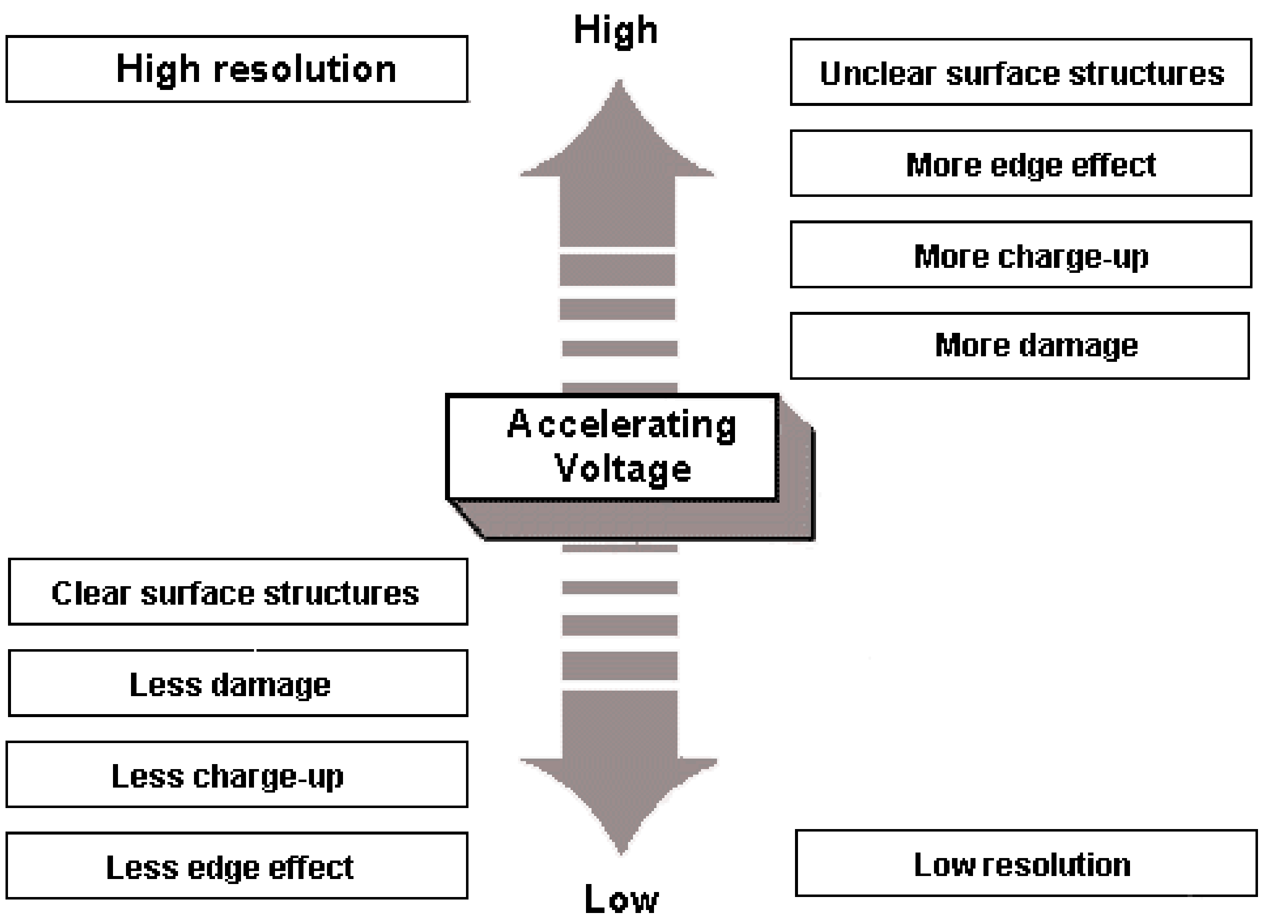


Correct Alignment

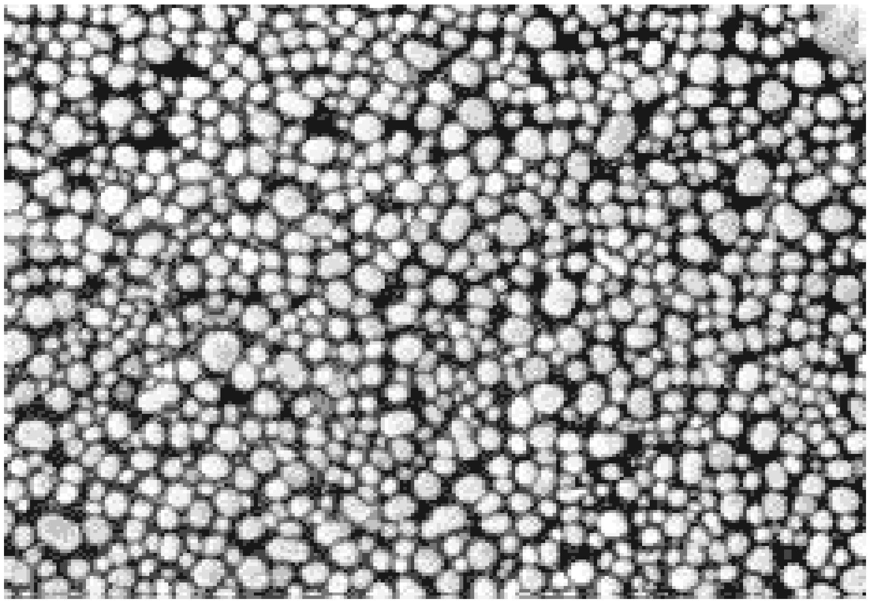
Zinc oxide

25 kV and 21,000 X magnification
Influence of alignment on image quality

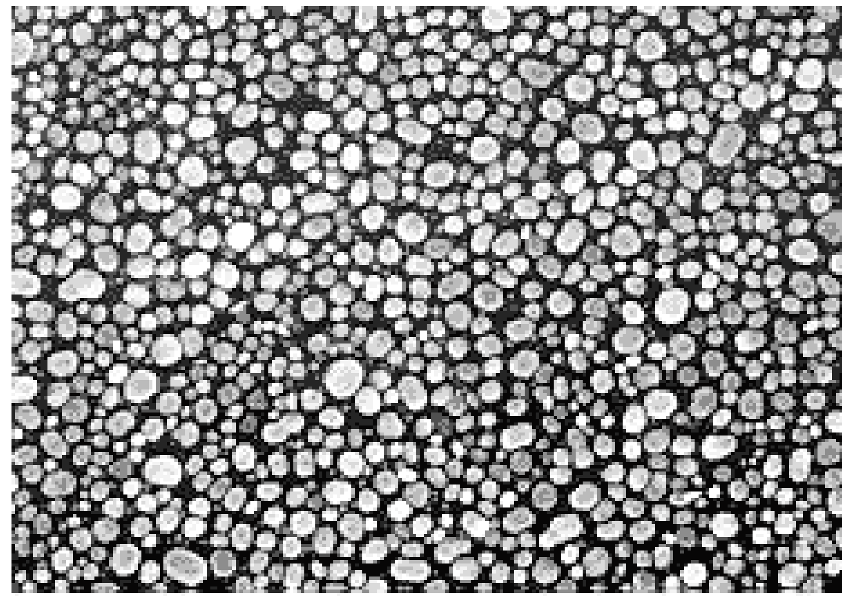
Effect of Accelerating Voltage on Image Quality



Effect of Accelerating Voltage on Observed Image



5.0 kV **36,000 X**

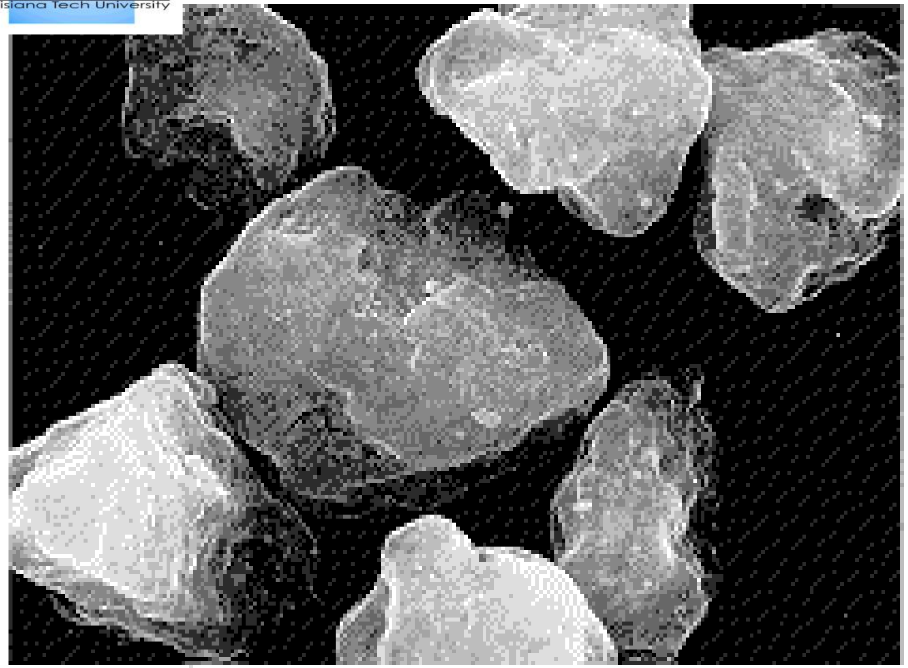


25 kV **36,000 X**

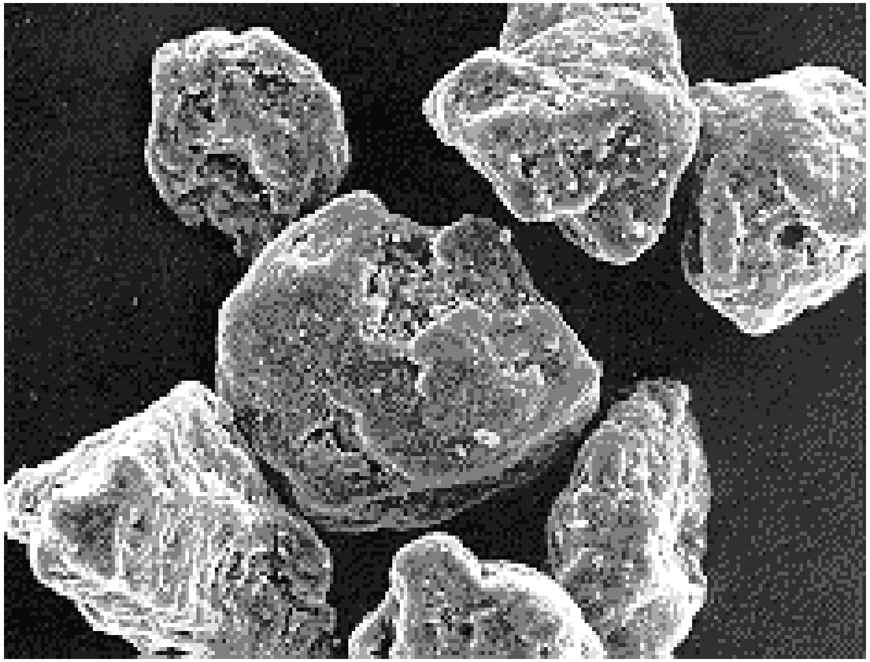
Evaporated Au particles

The image sharpness and resolution are better at the higher accelerating voltage, 25 kV

Effect of Accelerating Voltage on Observed Image



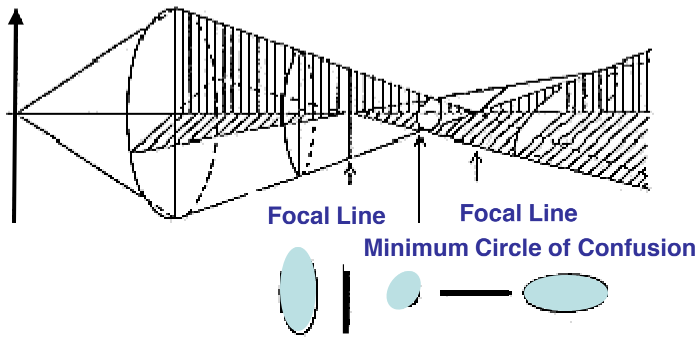
(a) 30 kV 2,500 X



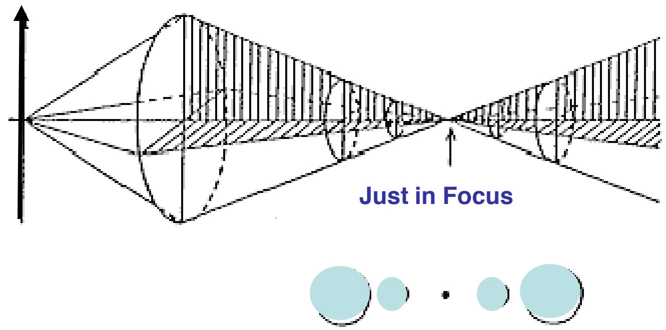
(b) 5.0 kV 2,500 X

When high accelerating voltage is used as at (a), it is hard to obtain the contrast of the specimen surface structure. The specimen surface is also easily charged up. The surface microstructures are clearly seen at (b).

Influence of Astigmatism on Electron Beam Shape



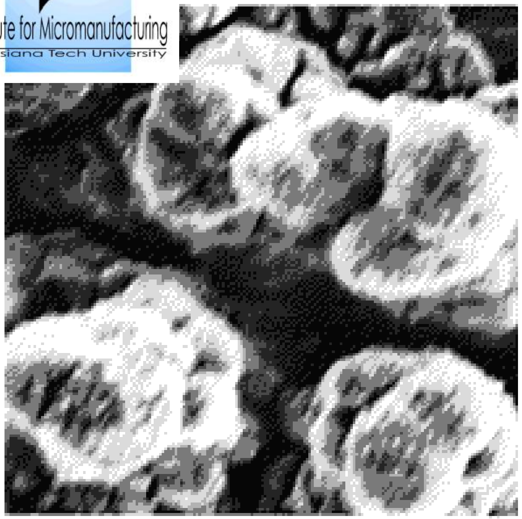
Electron beam shape when there is astigmatism present



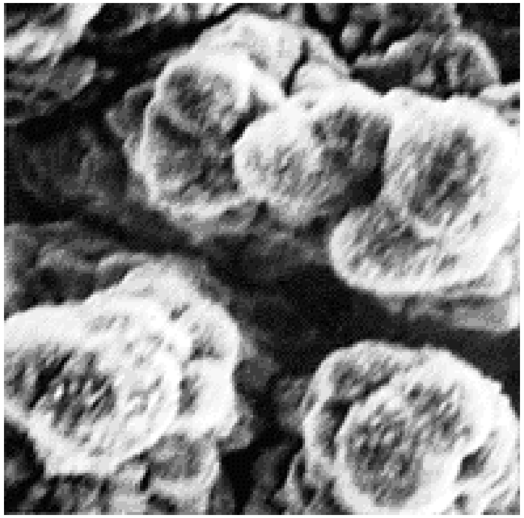
Electron beam shape when astigmatism is corrected

The aberration caused by the machining inaccuracy and material of the pole piece is called "astigmatism." This astigmatism can be removed by adjusting the X and Y correction controls of the stigmator.
An image is judged as astigmatism-free if it has no unidirectional defocusing when the objective lens is changed to under or over-focus at a higher magnification (at about 10,000 X).

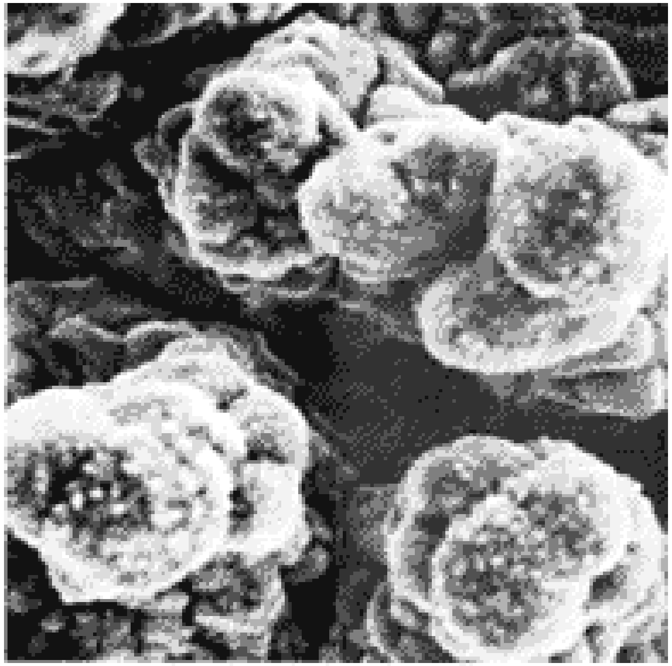
Influence of Astigmatism on Image Quality



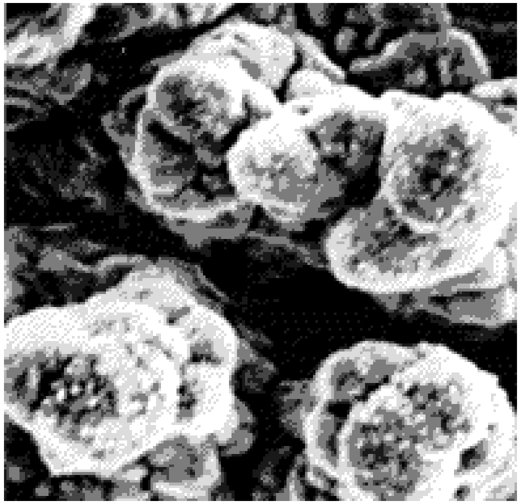
a) Under Focus



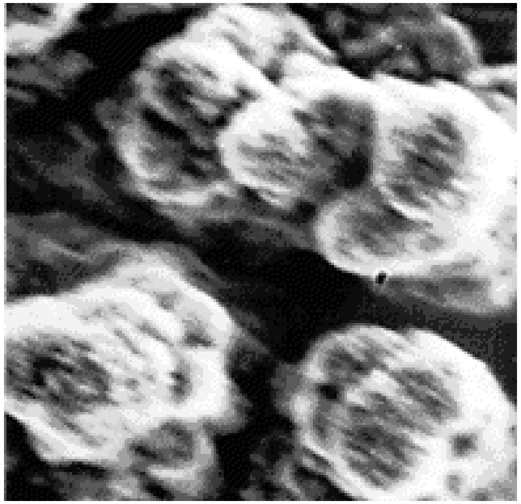
b) Under Focus



c) Just in Focus



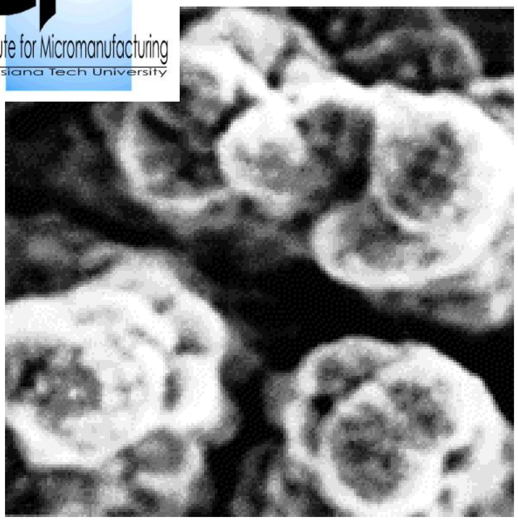
d) Over Focus



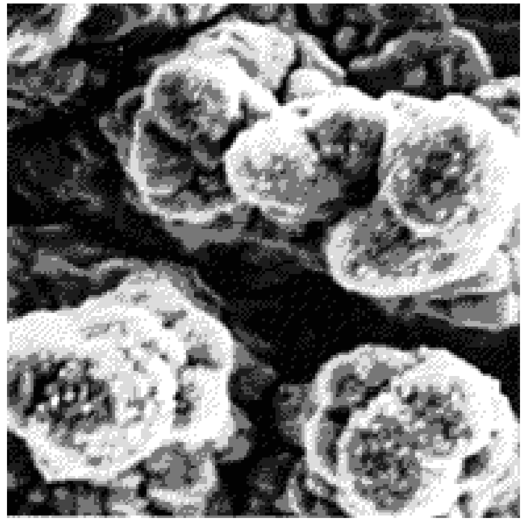
e) Over Focus

Images before astigmatism correction

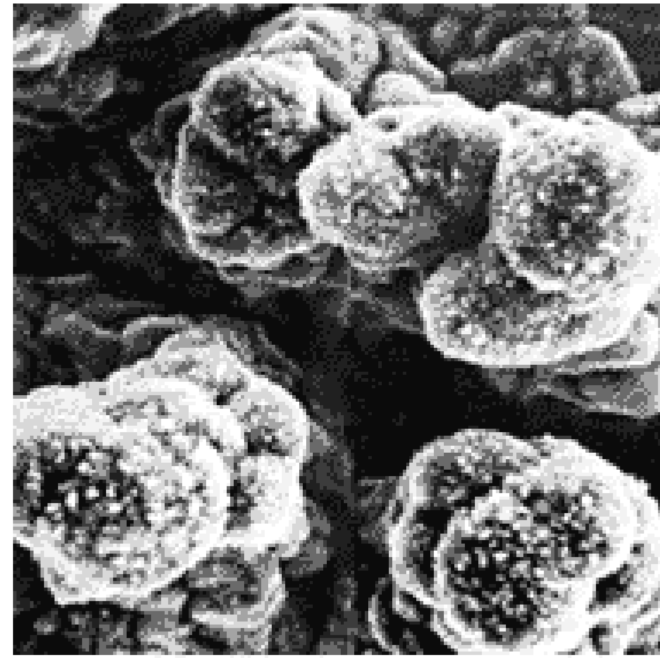
Influence of Astigmatism on Image Quality



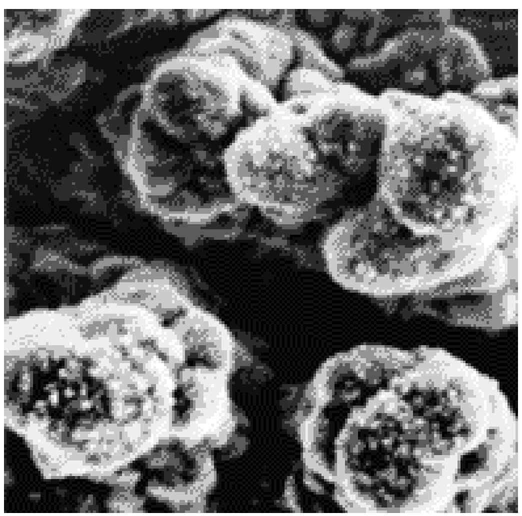
f) Under Focus



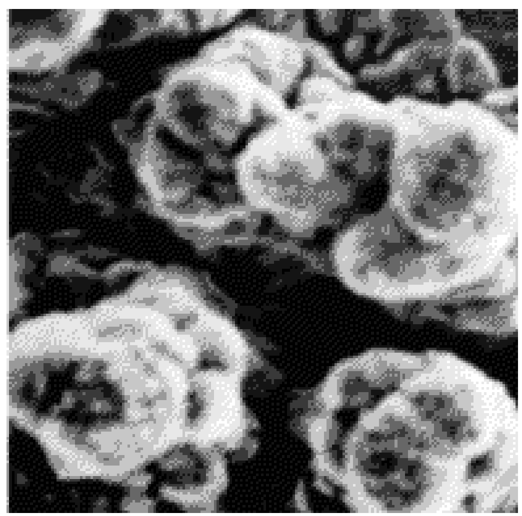
g) Under Focus



h) Just in Focus



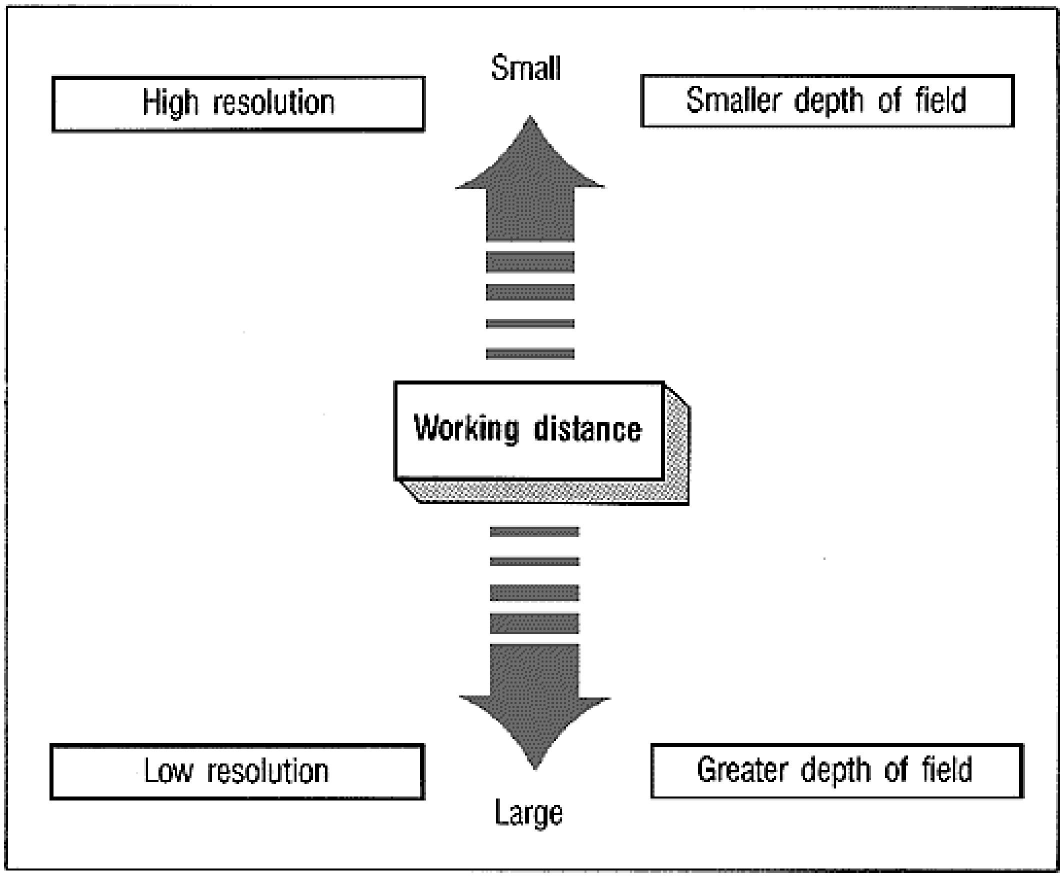
i) Over Focus



j) Over Focus

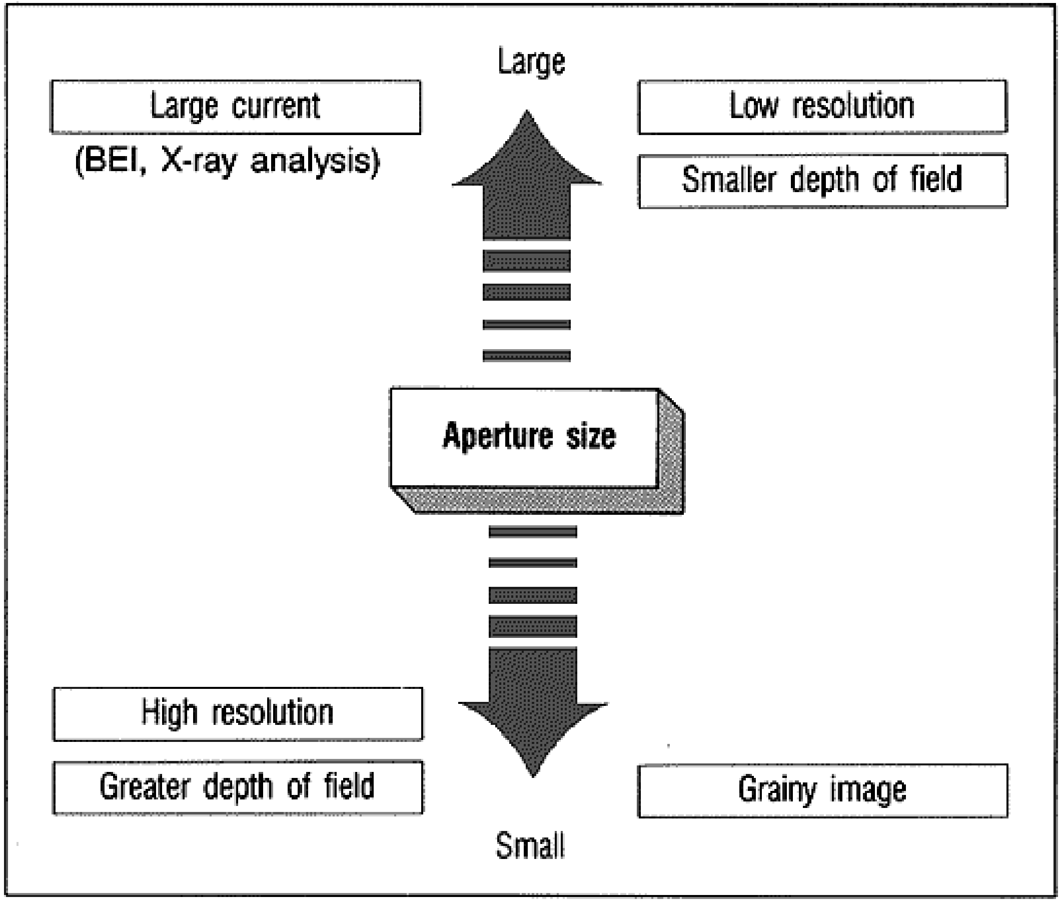
Images <u>after</u> astigmatism correction

Influence of Working Distance (WD) on Images



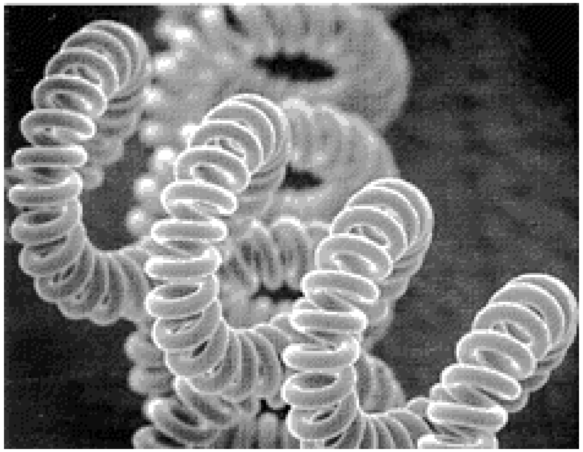
WD is changeable on many available SEM models. The schematic shows what effect is produced on the image when WD is changed with other conditions kept constant.

Effect of Objective Lens Aperture Diameter on Image

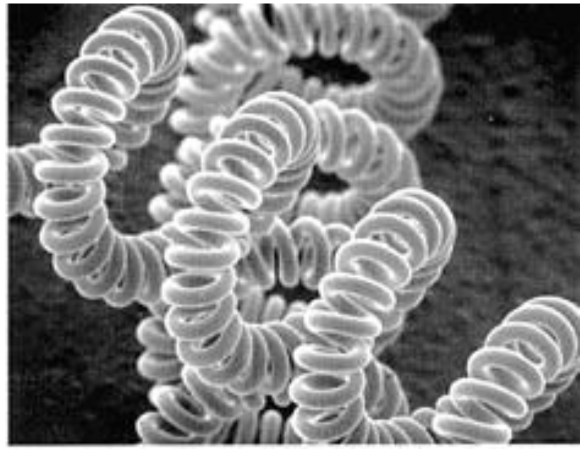


The objective lens (OL) aperture set in the SEM as standard is of the optimum size selected considering various conditions. SEM images require not only a fine electron probe, but also a sufficient amount of signal for forming an image. The aperture cannot be reduced unnecessarily. The OL aperture must be selected with consideration given to the effect on the image.

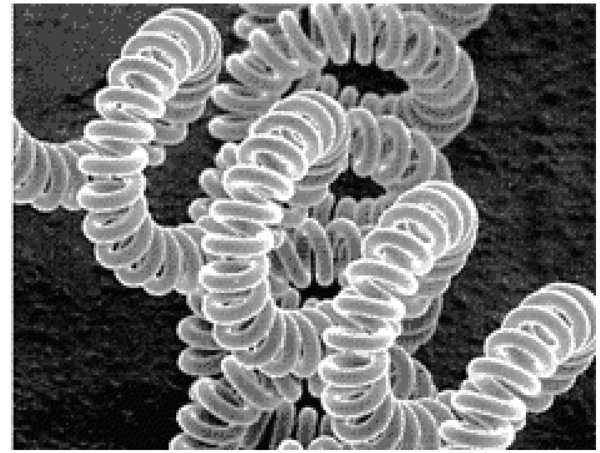
Effect of Objective Lens Aperture Diameter and WD on Image



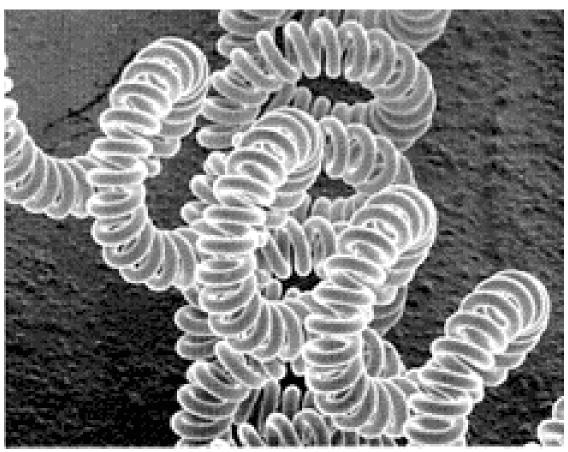
**(a) Aperture diameter = 600 um
Working distance = 10 mm**



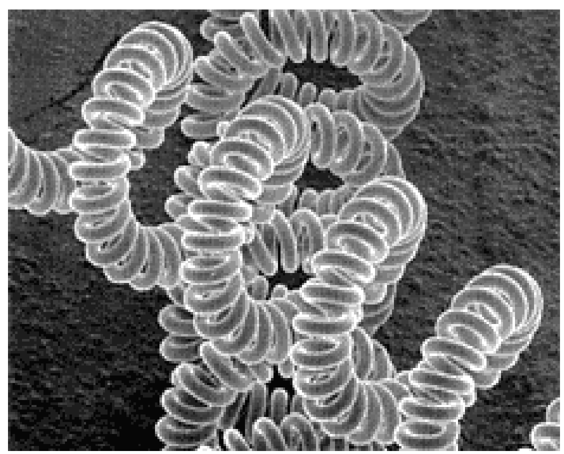
**(b) Aperture diameter = 200 um
Working distance = 10 mm**



**(c) Aperture diameter = 200 um
Working distance = 20 mm**



**(d) Aperture diameter = 200 um
Working distance = 38 mm**

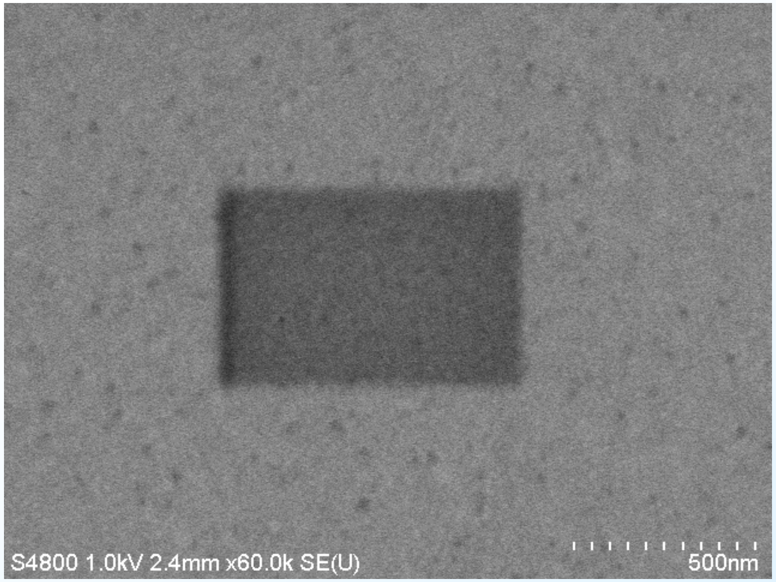


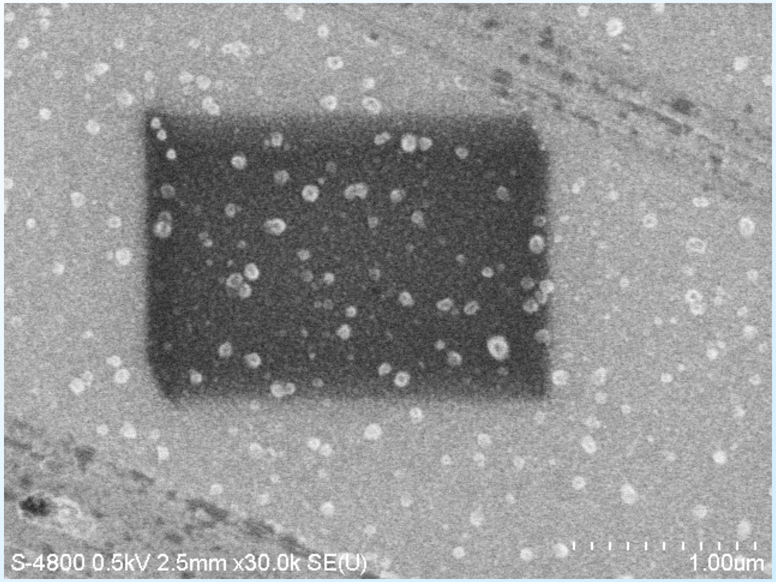
**(e) Aperture diameter = 100 um
Working distance = 38 mm**

Electric light bulb filament

The smaller the objective lens aperture diameter and the longer the working distance (WD), the greater will be the depth of field

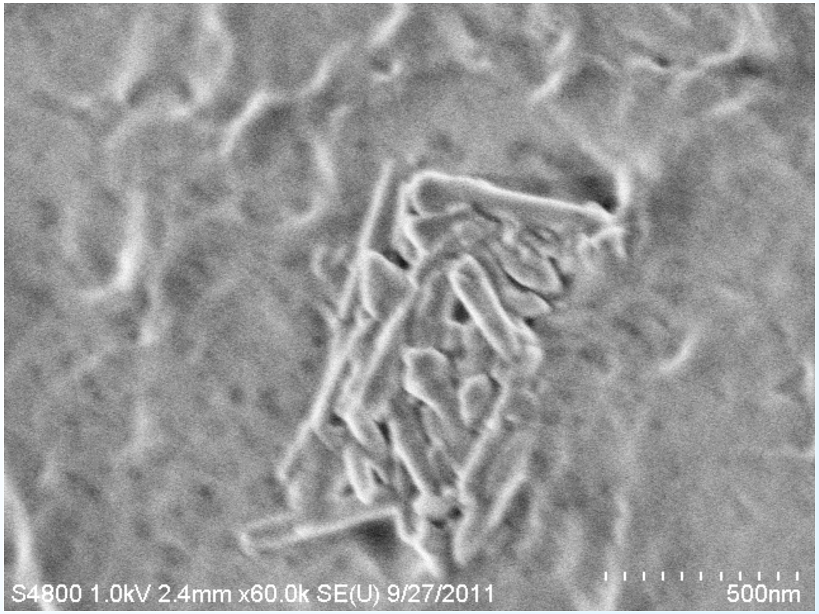
Sample Contamination

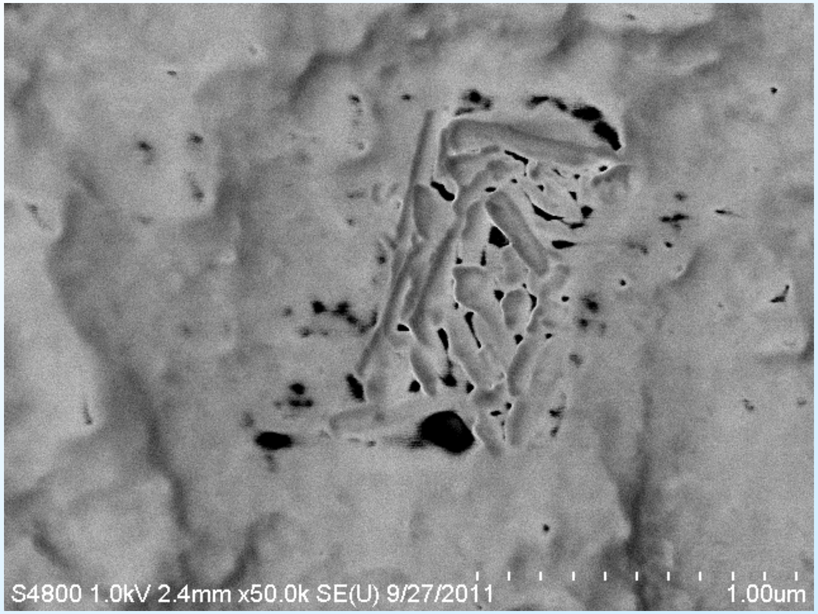




Residual gas or the presence of organic contaminants can affect imaging, reducing the sharpness of the image. Requires careful handling of specimens!

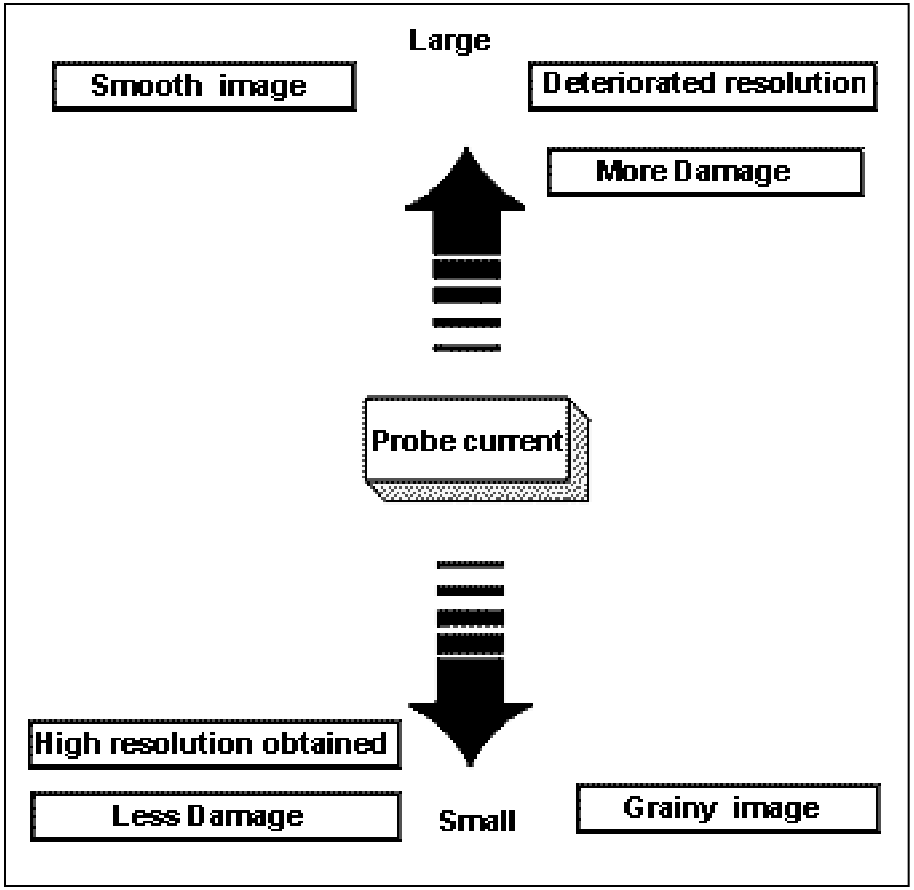
Beam Induced Damage on Polymers





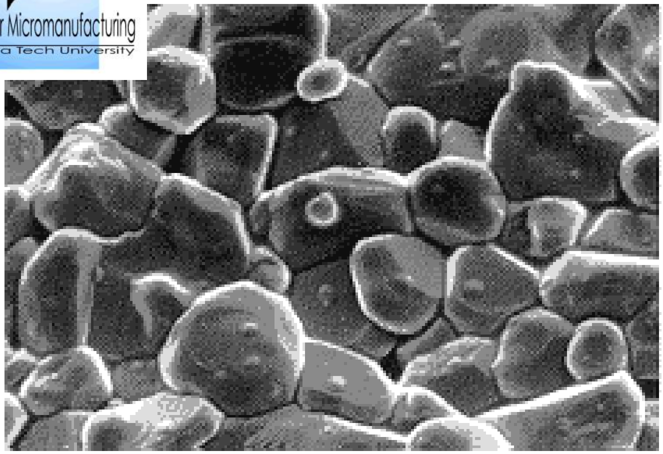
For certain low density polymer materials, scanning and capturing images faster help to minimize beam induced damages on the sample.

Effect of Electron Probe Diameter on Image Quality



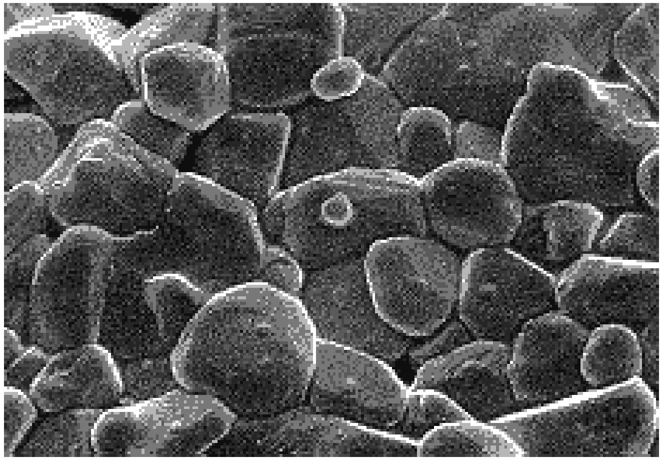
In the SEM, the smaller the electron probe diameter on the specimen, the higher the magnification and resolution. However, the image smoothness, namely, the S/N ratio depends on the probe current.
As the probe diameter is reduced, the probe current is reduced.
It is therefore necessary to select a probe current suited for the magnification and observation conditions (accelerating voltage, specimen tilt, etc.) and the specimen.

Relationship Between Beam Current and Probe Diameter



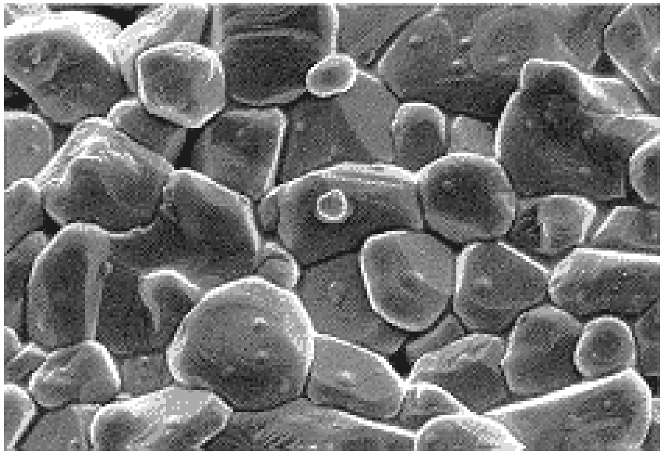
Ceramic

10 kV and 5,400 X

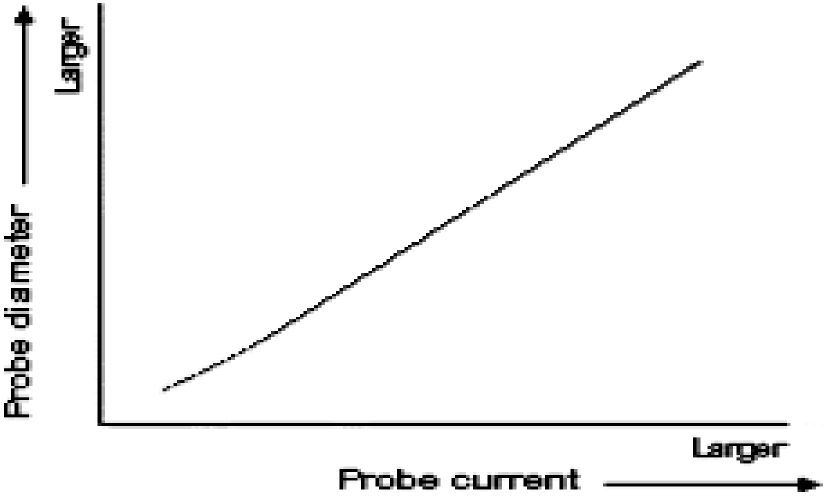


(a) 1 nA

c) 10 pA

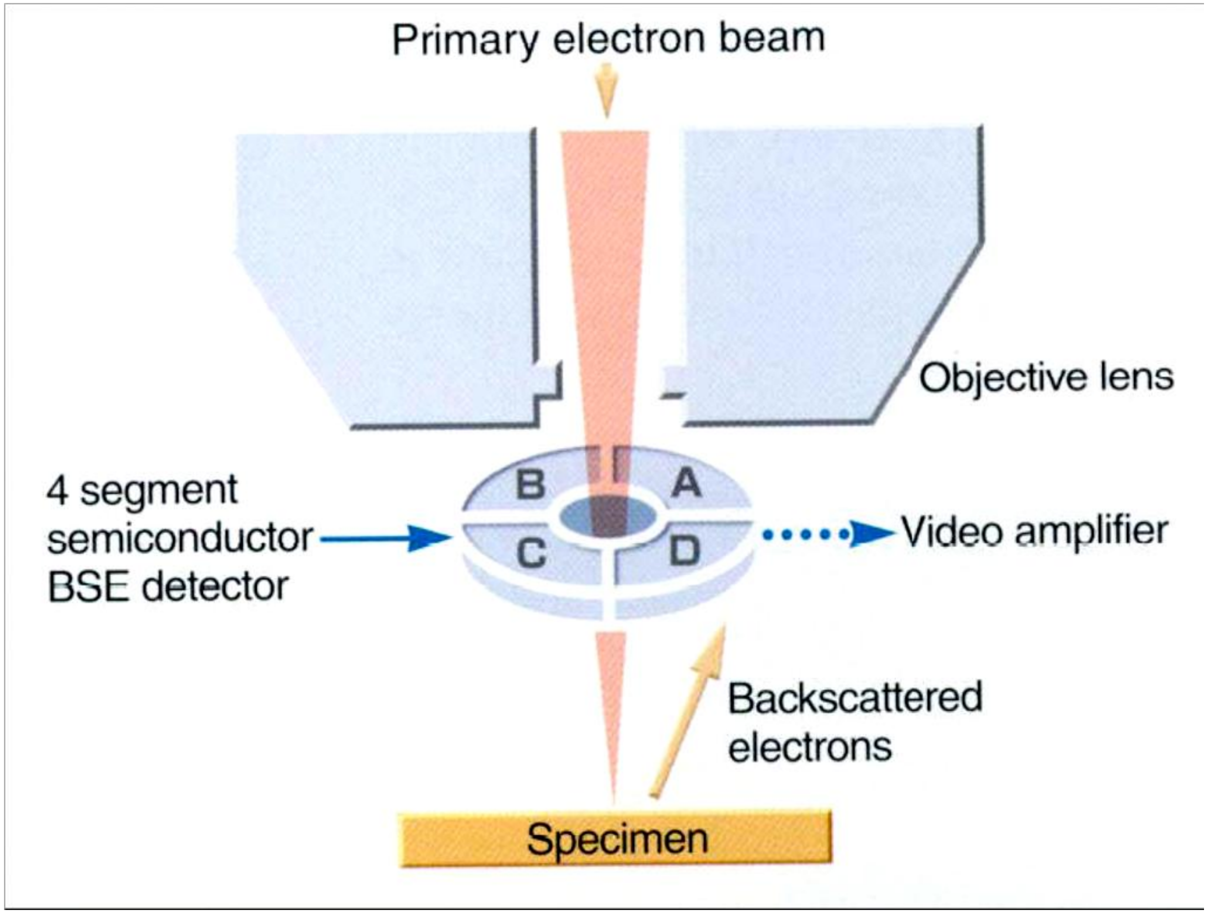


b) 0.1 nA

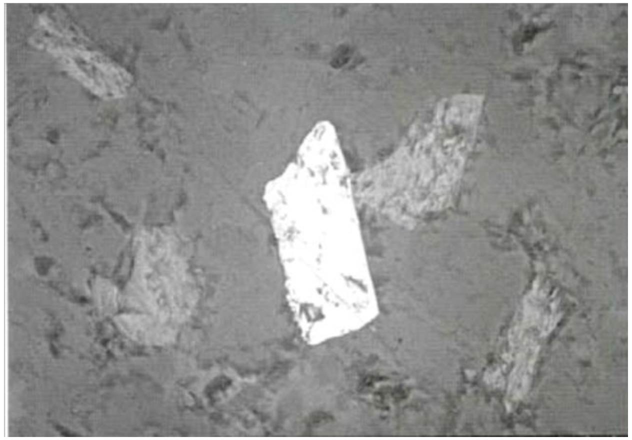


The smaller the probe current, the sharper is the image, but the image becomes grainy.

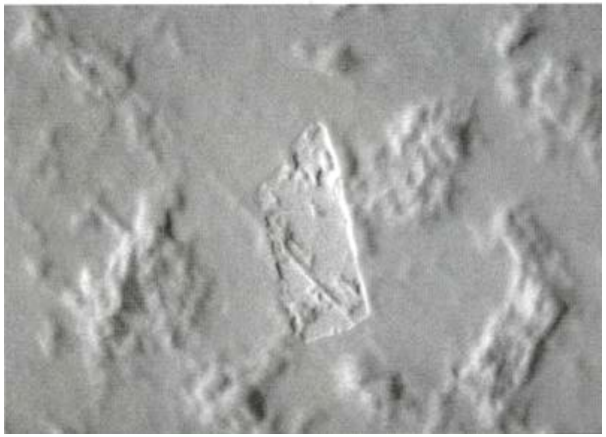
Backscattered Electron Detector



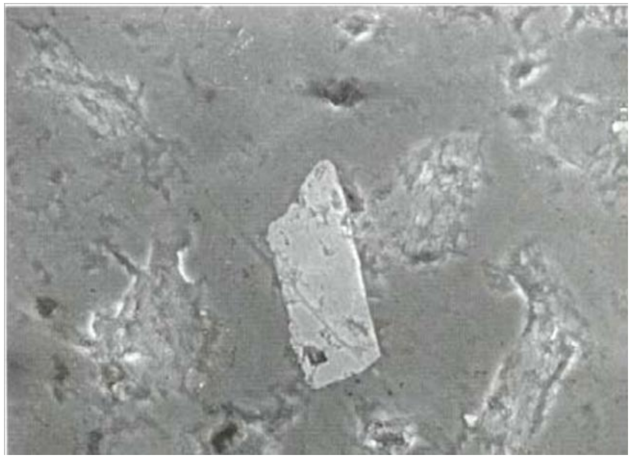
Images Using 4-Quadrant BSE Detector



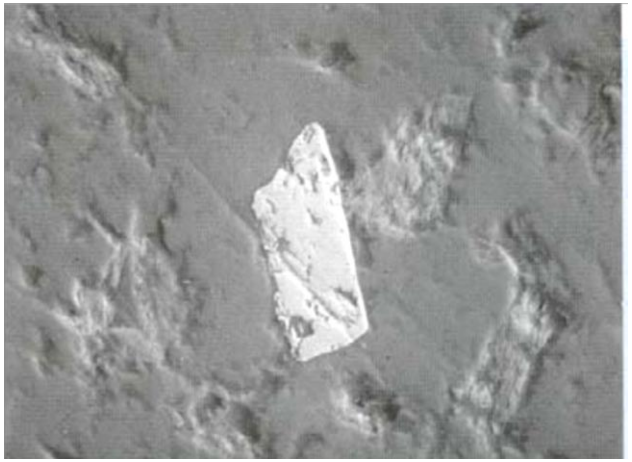
BSE Signal Summed > Composition



BSE Signal Subtracted > Topography

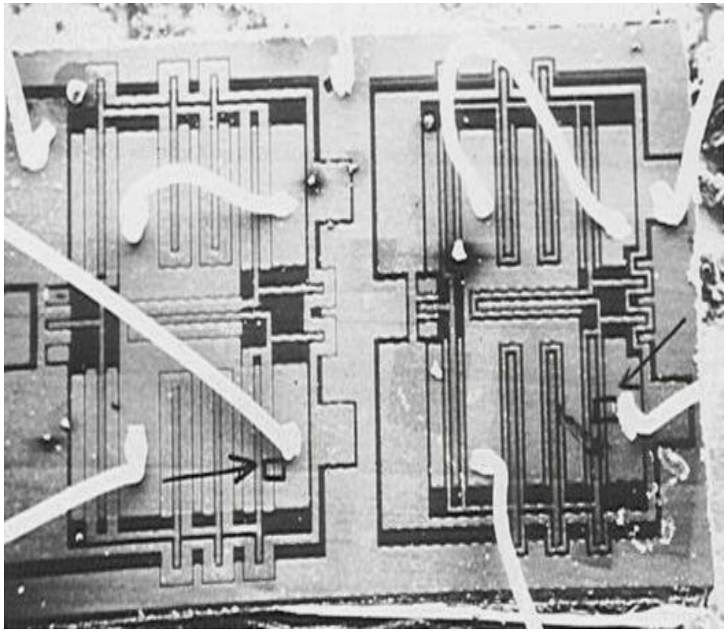


Secondary Electron Image

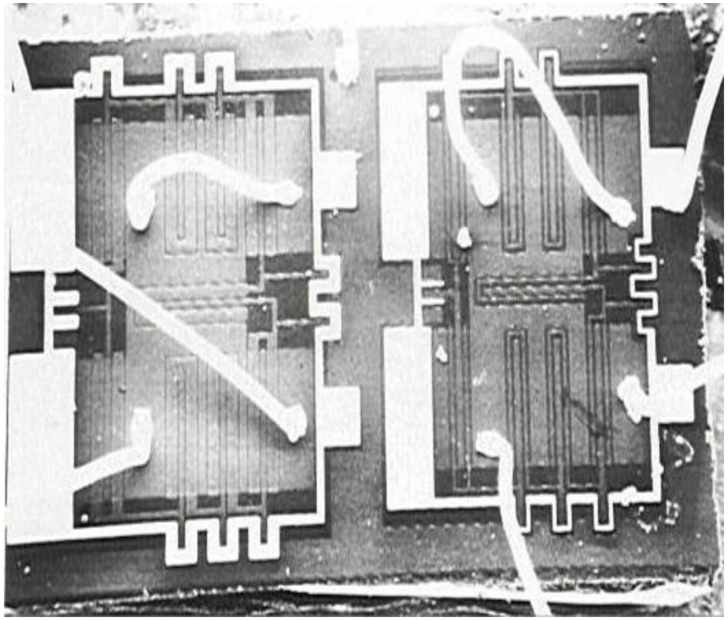


Mixed BSE and SE Image

SEM Voltage Contrast Mode
Semiconductor Chain Oscillator



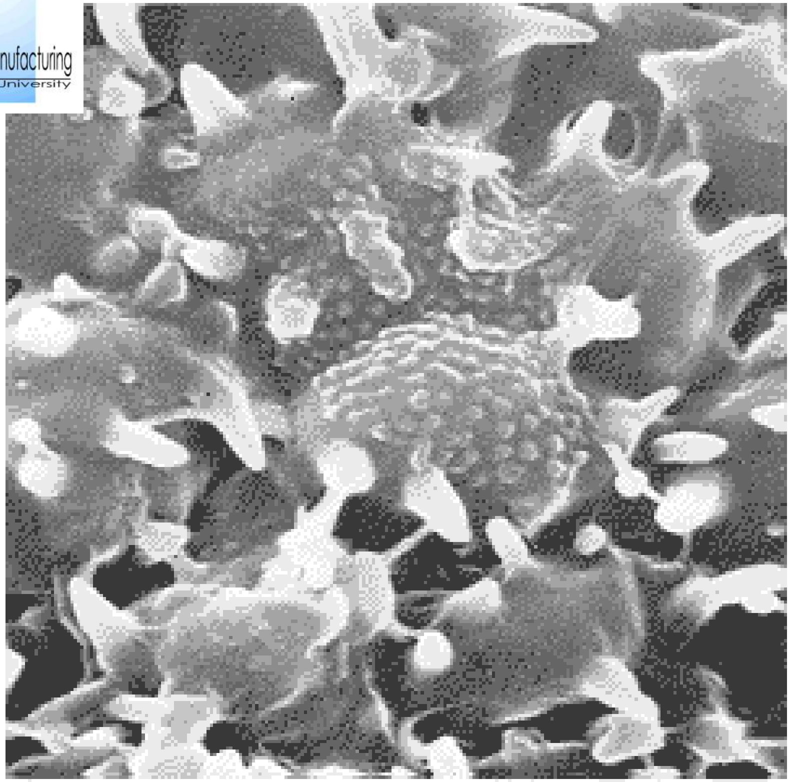
Secondary Electron Emission (Topography)

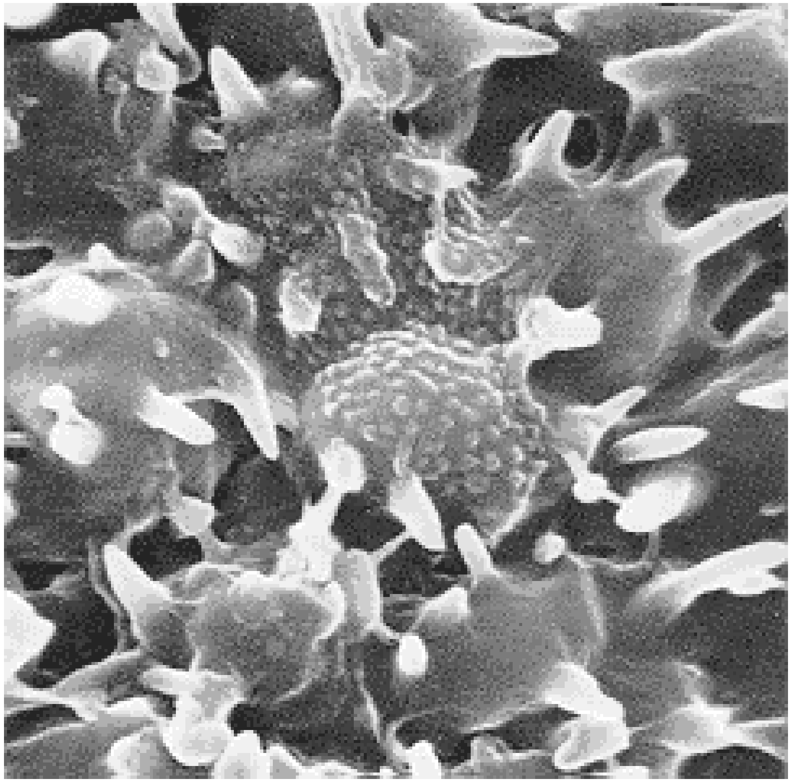


Voltage Contrast (Circuit Continuity)

- 24 Volts Applied to Device

Other Sources of Image Distortion



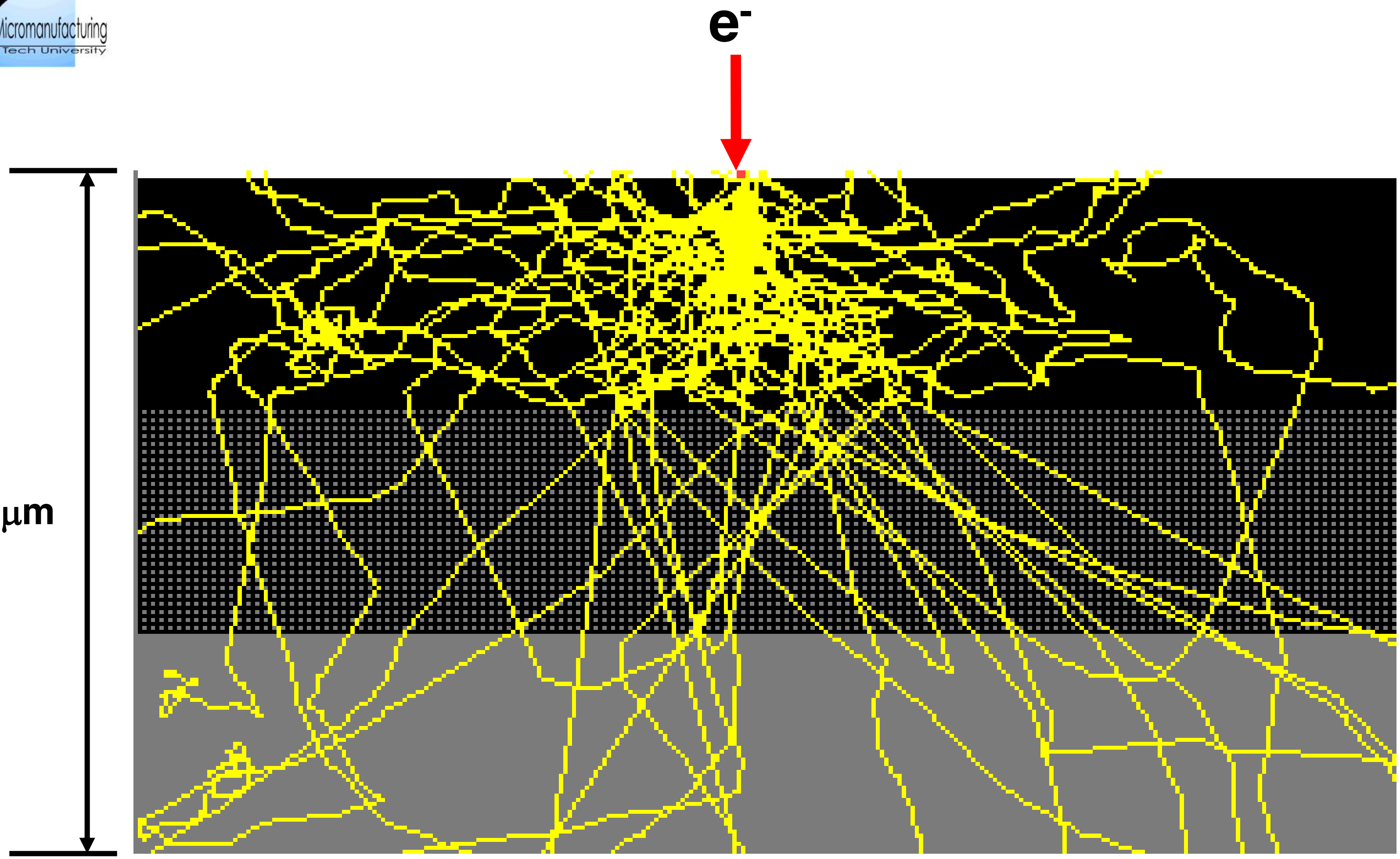


(a) No magnetic field present **(b) Magnetic field is present**

Influence of external magnetic field on image. Compared with (a), (b) is demagnified at the center and magnified at both sides due to magnetic field, 50 Hz.

Electron Beam Interaction with Solids

20 keV Accelerating Voltage and Z = 28 (nickel)



Monte Carlo Simulation of Electron Trajectories

Electron Interaction with Solids

Electron penetration generally ranges from 1- 5 *μ*m with the beam incident perpendicular to the sample. <u>The depth of electron penetration is approximately:</u>

$$x\ (\mu m) = \frac{0.1\ E_o^{1.5}}{\rho}$$

where E_o = accelerating voltage (keV), and ρ = density (g/cm^3)

For example, bombarding a material with a density of 2.5 g/cm^3, which is about the minimum density for silicate minerals, with E_o = 15 keV, gives x = 2.3 *μ*m. <u>The width of the excited volume can be approximated by:</u>

$$y\ (\mu m) = \frac{0.077\ E_o^{1.5}}{\rho}$$

where E_o = accelerating voltage (keV), and ρ = density (g/cm^3)

Electron Interaction with Solids

A theoretical expression for the "range" of an electron, the straight line distance between where an electron enters and its final resting place, for a given E_o is:

$$r\,(\mu m) = \frac{2.76 \times 10^{-2}\, A\, E_o^{1.67}}{\rho\, Z^{0.89}}$$

where ρ = density of the material (g/cm^3),
Z = atomic number,
A = atomic mass,
and E_o = accelerating voltage.

Waveforms Generated from a Single and Dual SE Detectors

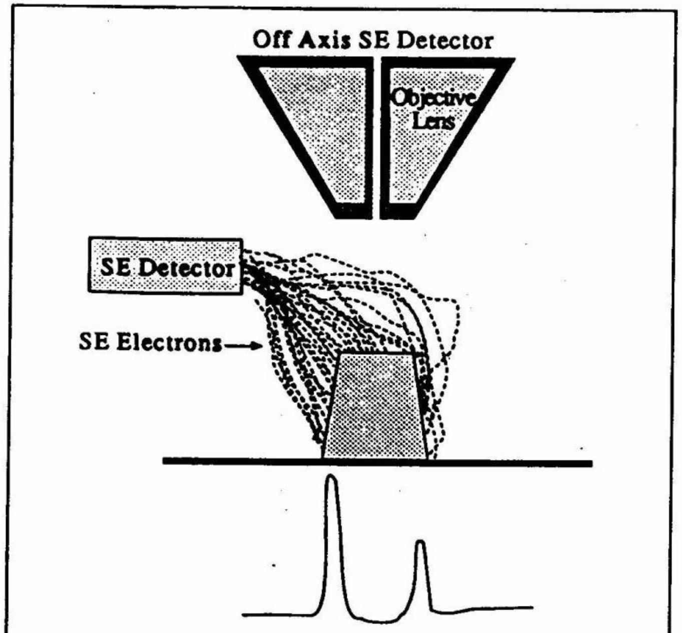


Figure 3. Waveform generated by an off axis SE detector configuration.

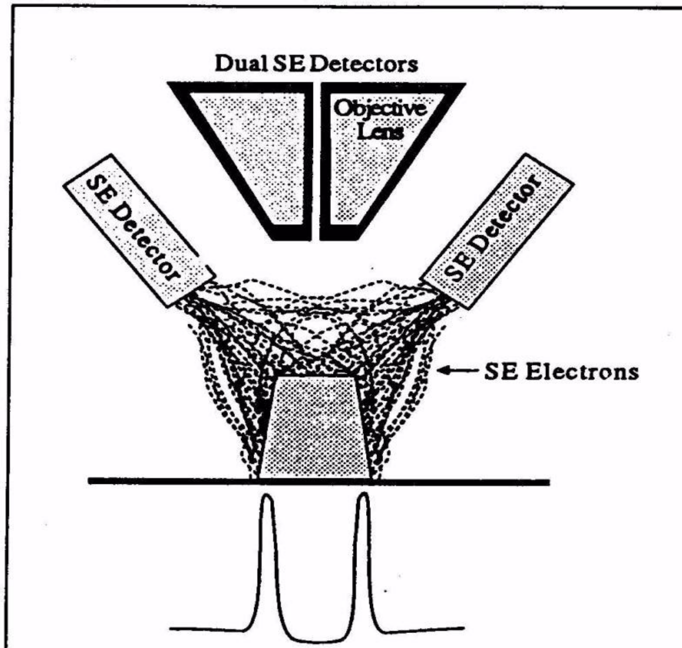
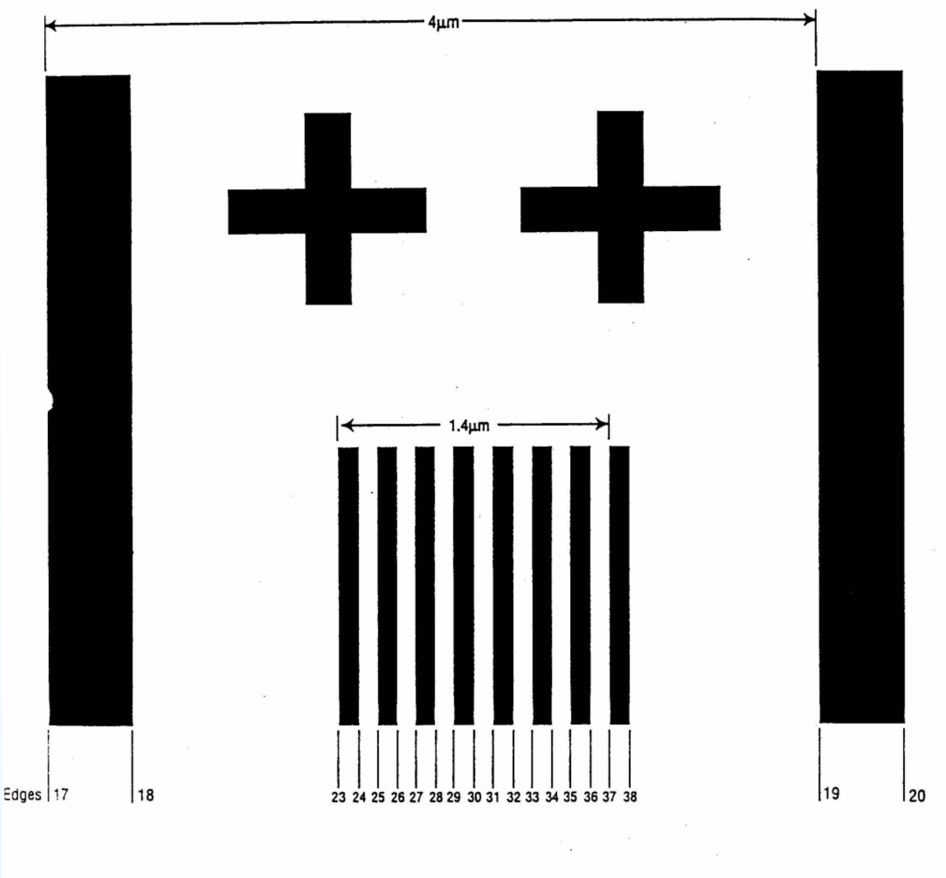


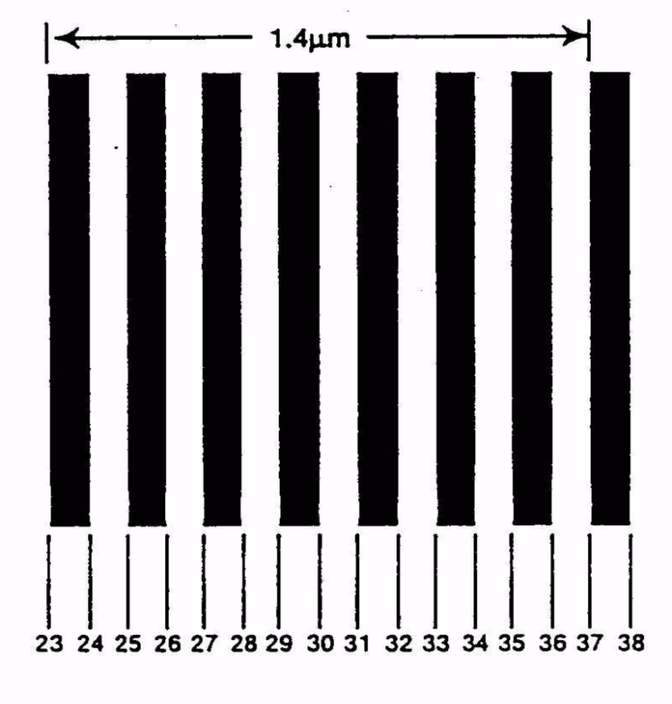
Figure 4. Waveform generated by a dual SE detector configuration.

Uneven sample irradiation or signal collection will affect the measurement results.

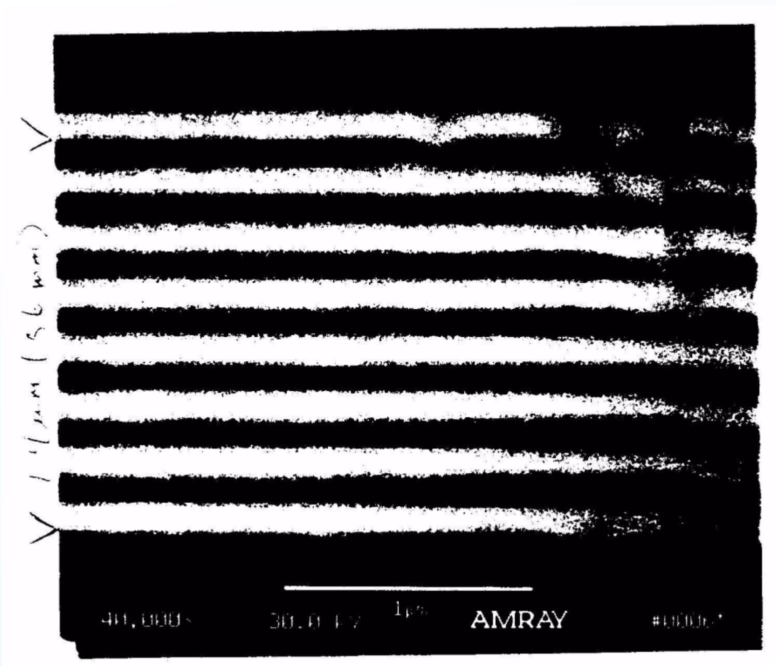
Critical SEM Metrology

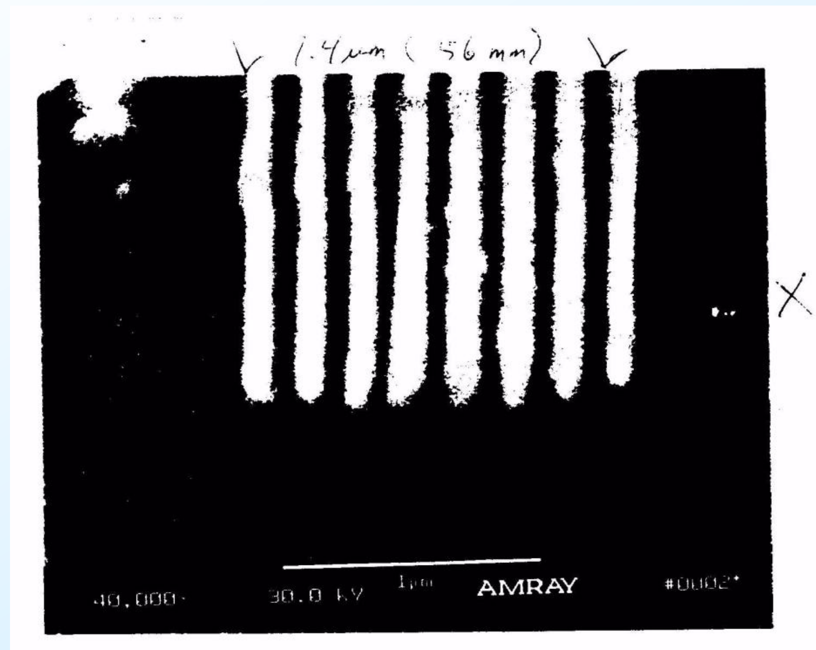
Calibration of Instruments





Checking the Calibration Standard in SEM



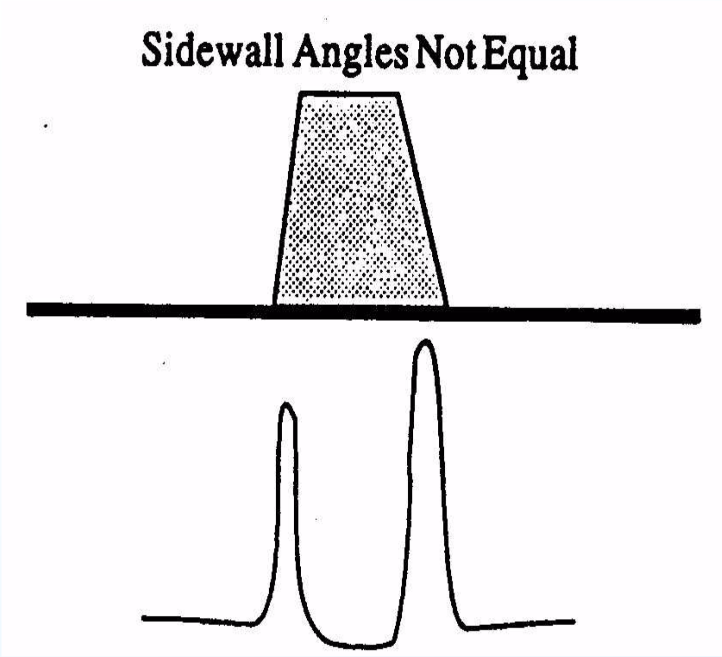


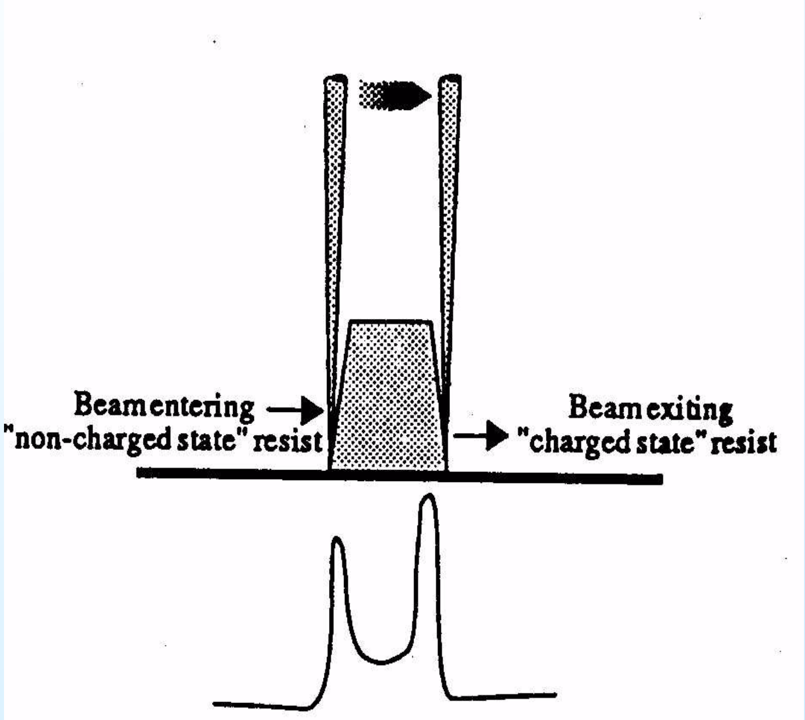
Measured Distance = Actual Distance x Magnification

56 mm = 1.4 micron x 40000

Need to be checked for both X and Y axes!

Asymmetrical Waveforms

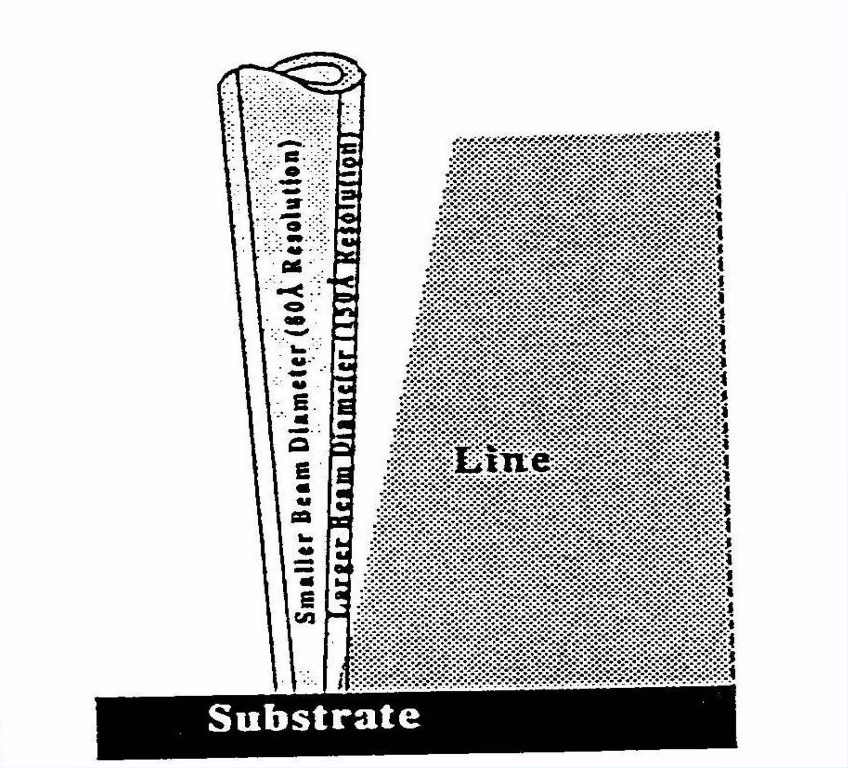




Waveform generated by unequal side-walls and proximity effect

Waveform generated by different SE emission due to non-charged/ charged state.

Beam diameter-Sample Interaction Effects



If the beam diameter is larger, the beam will intersect the area where the edge of the line rests on the substrate before a smaller beam would.

It would seem the line is larger.

References and Supplementary Reading Materials

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3. Electron Microscopy, J.J. Bozzola and L.D. Russell, Second Edition, ISBN: 0-7637-0192-0
4. Submicrometer Microelectronics Dimensional Metrology: Scanning Electron Microscopy, M.T. Postek and D.C. Joy, J. Res. NBS, v. 92, no.3, (1987) p. 205.
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6. **Scanning Electron Microscopy, A Student's Handbook, M.T.** Postek, K.S. Howard, A.H. Johnson, K.L. McMichael.
7. http:/www.mse.iastate.edu/microscopy/