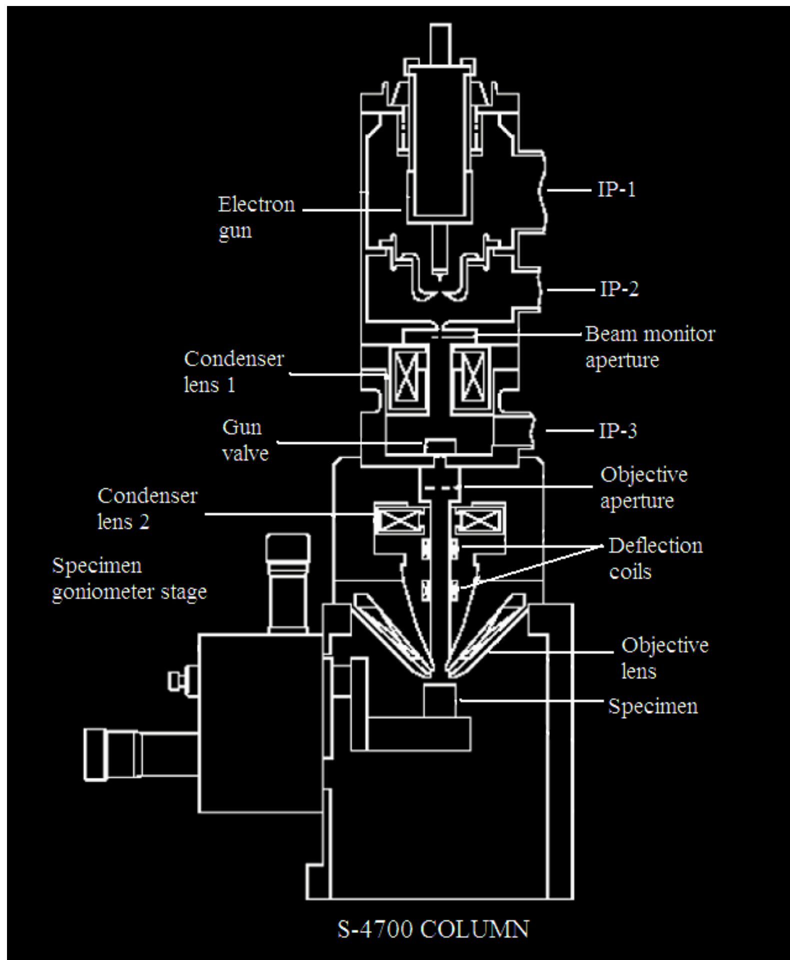
Scanning Electron Microscopy Primer

Bob Hafner

This primer is intended as background for the Introductory Scanning Electron Microscopy training offered by the University of Minnesota's Characterization Facility (CharFac). The primer addresses concepts fundamental to any scanning electron microscope (SEM); it also, where possible, informs the reader concerning specifics of the facility's four SEMs: JEOL 6500; JEOL 6700; Hitachi S-4700; and Hitachi S-900. You must learn this material **prior to** the hands-on training and you will be required to pass a test on it in order to become an independent SEM user at CharFac. A good source for further information is: "Scanning Electron Microscopy and X-Ray Microanalysis" by Joseph Goldstein et al.

The Big Picture

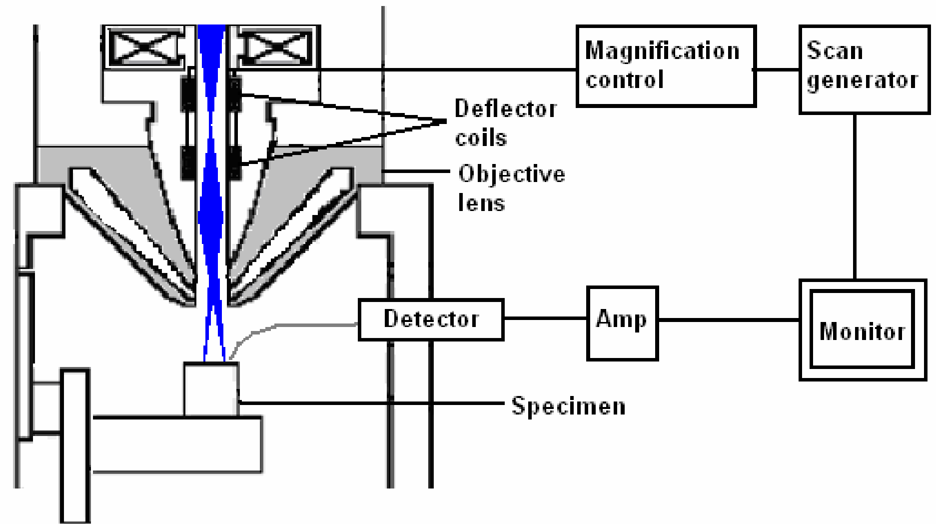
To the right is a picture of our Hitachi S-4700. The microscope column, specimen chamber, and vacuum system are on the left; the computer, monitor, and many of the instrument controls on the right. As an operator you will need to understand what is happening inside the "black box" (microscope column and specimen chamber) when an instrument control is manipulated to produce a change in the monitor image.



A look inside the black box [1] reveals quite a bit of complexity; however, we can simplify at this point. We have:

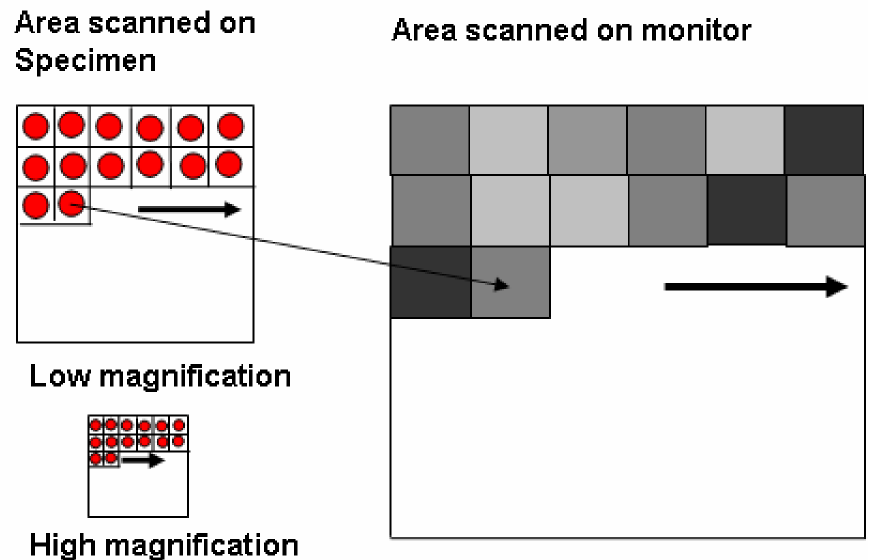
- a source (**electron gun**) of the electron beam which is accelerated down the column;
- a series of **lenses (condenser and objective)** which act to control the diameter of the beam as well as to focus the beam on the specimen;
- a series of **apertures** (micron-scale holes in metal film) which the beam passes through and which affect properties of that beam;
- controls for **specimen position** (x,y,z-height) and orientation (tilt, rotation);
- an area of **beam/specimen interaction** that generates several types of signals that can be detected and processed to produce an image or spectra; and
- all of the above maintained at high **vacuum levels** (the value of the upper column being greater than the specimen chamber).

If we take a closer look at the lower column and specimen chamber, we see the objective lens which focuses the electron beam on the specimen surface. A signal is generated from the specimen, acquired by the detector, and processed to produce an image or spectrum on the monitor display.



We also see a pair of deflector coils, controlled by the Scan Generator, which are responsible for rastering that focused beam across the specimen surface. The size of the rastering pattern is under Magnification Control.

The beam is rastered from left to right and top to bottom. There is a one-to-one correspondence between the rastering pattern on the specimen and the rastering pattern used to produce the image on the monitor. The resolution we choose to image at will obviously affect the number of pixels per row as well as the number of rows that constitute the scanned area.



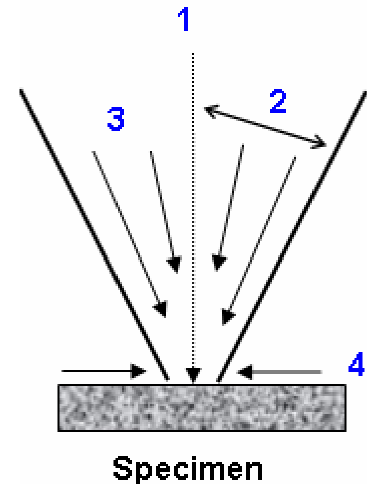
The red dot within each pixel on the specimen represents an area of beam--specimen interaction from which the signal is derived (more on this later). The signal is collected by the detector and subsequently processed to generate the image. That processing takes the intensity of the signal coming from a pixel on the specimen and converts it to a grayscale value of the corresponding monitor pixel. **The monitor image is a two dimensional rastered pattern of grayscale values.**

With the beam focused on the specimen surface, all we need to do to change magnification is to change the size of the rastered area on the specimen. The size of the monitor raster pattern is constant. Magnification will increase if we reduce the size of the area scanned on the specimen. **Magnification = area scanned on the monitor / area scanned on the specimen.**

A knowledgeable SEM operator should have a basic command of the following content areas:

- electron optics;
- beam-specimen interactions;
- signal types and detector characteristics;
- signal quality/feature visibility relations; and
- signal/imaging processing.

In order to better understand the first two of these content areas, it will be useful at this point to define the major parameters associated with the electron beam (probe) at the specimen surface. These parameters are ones that we can control as an operator and define the major modes of imaging in the SEM. They are:



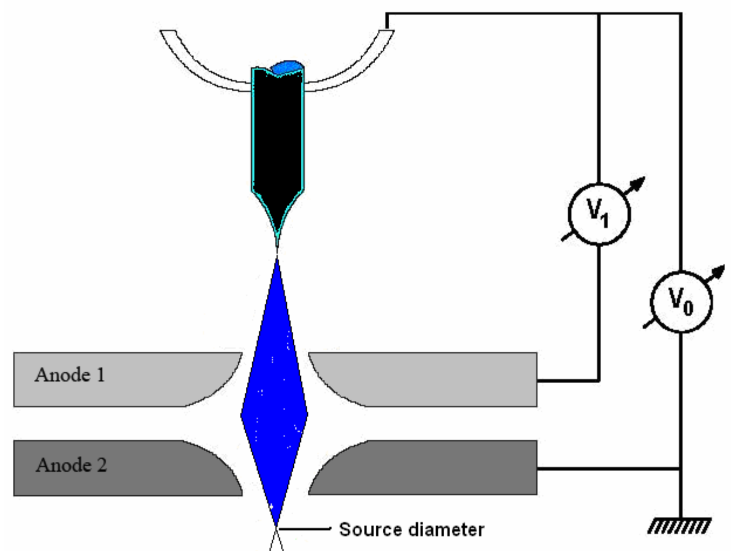
1. **Beam accelerating voltage (kV)**: the voltage with which the electrons are accelerated down the column;
2. **Probe convergence angle (α_p)**: the half-angle of the cone of electrons converging onto the specimen;
3. **Probe current (i_p)**: the current that impinges upon the specimen and generates the various imaging signals; and
4. **Probe diameter or spot size (d_p)**: the diameter of the final beam at the surface of the specimen.

Looking at the diagram it would seem that all we would have to do to maintain adequate probe current in a small probe diameter would be to increase the probe convergence angle. But this is not the case due to aberrations in the optic system (more on this later). A small probe diameter always comes with a decrease in probe current. These parameters are interrelated in other ways. For example, a decrease in accelerating voltage will result in a decrease in probe current as well as an increase in probe size. **"Doing SEM" involves understanding the trade offs** that necessarily occur when we choose operational parameters.

Electron Optics

Electron Guns

The purpose of the electron gun is to provide a stable beam of electrons of adjustable energy. There are three main types of electron guns: Tungsten hairpin; Lanthanum hexaboride (LaB_6); and Field emission. We will concentrate on the latter since the CharFac's four SEMs are all field emission gun (FEG).



The FEG cathode consists of a sharp metal (usually Tungsten) tip with a radius of less than 100 nm. A potential difference (V_1 = extraction voltage) is established between the first anode and the tip. The result is an electric field, concentrated at the tip, which facilitates electron emission (**emission current**).

The potential difference between the tip and the second grounded anode determines the **accelerating voltage** (V_0) of the gun. The higher the accelerating voltage the faster the electrons travel down the column and the more penetrating power they have.

There are two types of FEGs:

- Cold (JEOL 6700; Hitachi S-4700; Hitachi S-900), and
- Thermally assisted (JEOL 6500).

Both types of field emission require that the tip remain free of contaminants and oxide and thus they require Ultra High Vacuum conditions (10^{-10} to 10^{-11} Torr). In the cold FEG the electric field produced by the extraction voltage lowers the work function barrier and allows electrons to directly tunnel through it—thus facilitating emission. The cold FEGS must have their tip "flashed" (briefly heated) periodically to free absorbed gas molecules. The thermally assisted FEG (Schottky field emitter) uses heat and chemistry (nitride coating) in addition to voltage to overcome the potential barrier level.

Although the FEG has a moderate emission current, its **"Brightness"** value is orders of magnitude greater than the thermionic Tungsten and LaB_6 sources (table below). Brightness is the beam current per unit area per solid angle **[$B = 4i_p \ / \ (\pi \ d_p \ \alpha_p)^2$]** and, unlike current, it is conserved down the column. Brightness increases linearly with accelerating voltage.

	Tungsten	**LaB_6**	**Thermal FEG**	**Cold FEG**
Brightness (A/cm^2str)	10^5	10^6	10^8	10^8
Lifetime (hrs)	40-100	200-1000	>1000	>1000
Source Size	30-100 um	5-50 um	<5 nm	<5 nm
Energy Spread (eV)	1-3	1-2	1	0.3
Current Stability (%hr)	1	1	5	5
Vacuum (Torr)	10^{-5}	10^{-7}	10^{-11}	10^{-11}

The high brightness value is due to the fact that a given emission current occurs within a very small source size as the beam exits the gun. This **"Source Size"** for FEGs is on the order of nanometers rather than microns for the other emission sources. **The ability to have enough probe current (and thus potential signal) in a probe of small diameter allows the FEGSEM to obtain the resolution it does**. The ability to achieve a small probe diameter is directly related to the source size or the diameter of the electron beam exiting the gun. An electron beam emanating from a small source size is said to have high **spatial coherency**. Electron beams can also be characterized in terms of **temporal coherency**. A beam with high temporal coherency will have electrons of the same wavelength. In reality there is a certain **"Energy Spread**" associated with the beam. As we will see, lower energy spreads result in better resolution and are particularly important in low accelerating voltage imaging.

These enhanced FEGSEM capabilities come with a cost (literally). Very expensive vacuum systems must be attached to these microscopes to achieve the vacuum levels they require. The advantages of a coherent beam source will be negated if the beam is interacting with molecules on its path down the column. The vacuum at the gun level of the column is kept at 10^{-10} to 10^{-11} Torr; the vacuum in the specimen chamber is in the 10^{-5} to 10^{-6} Torr range [1 Torr = 133 Pa = 1.33 mbar]. The table below is provided simply to give you a feeling for what these vacuum levels translate to inside the microscope.

Vacuum	Atoms/cm3	Distance between atoms	Mean Free Path	Time to monolayer
1 Atm (760 Torr)	10^{19}	5 x 10^{-9} meters	10^{-7} meters	10^{-9} seconds
10^{-2} Torr	10^{14}	2 x 10^{-7} meters	10^{-2} meters	10^{-4} seconds
10^{-7} Torr	10^{9}	1 x 10^{-5} meters	10^{3} meters	10 seconds
10^{-10} Torr	10^{6}	1 x 10^{-4} meters	10^{6} meters	10^{4} seconds

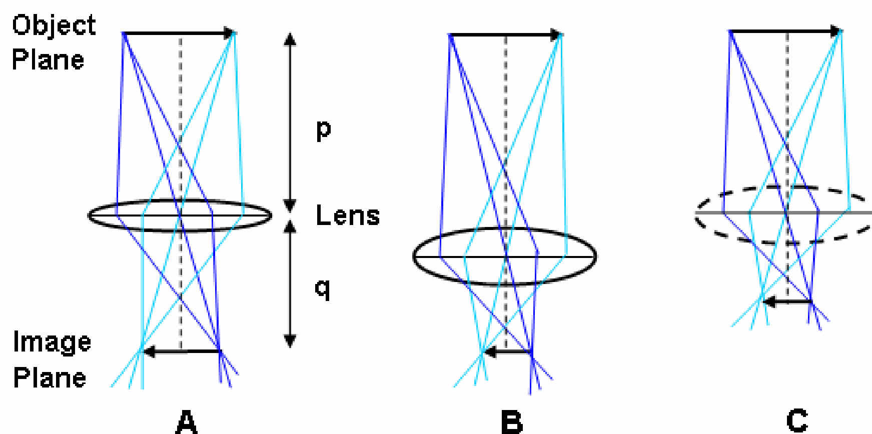
We won't spend time here talking about the various kinds of pumps and gauges associated with the vacuum system since those are maintained by the staff. However, as an operator, **you should be very aware of the vacuum state of the microscope** and ensure that:

- the microscope is at good vacuum level when you begin your session;
- the "Gun Valve" separating the upper column from the rest of the microscope is closed during sample exchange;
- appropriate vacuum levels are achieved prior to engaging the high tension;
- you use gloves when mounting samples and transferring them to the column; and
- your samples are dry and free of excessive outgassing.

Electron Lenses

Electromagnetic lenses are used to demagnify the image of the beam source exiting the electron gun and to focus the beam on the specimen. Condenser lenses are involved in demagnification; the objective lens focuses on the specimen as well as demagnifies. The source size of the FEG is comparatively small so that the amount of demagnification necessary to produce small probe sizes is less than that of other electron sources.

It is useful to reason by analogy with glass lenses used for focusing light to help one understand the operation of electron lenses. Analogies also have regions of non-correspondence which are equally important to understand.



A: a perfect optical lens. The rays emanating from a point in the object plane come to one common well defined point in the image plane. The optical lens has a fixed focal point and the object is in focus at the image plane.

B: a lens of different strength (represented as a thicker lens) and thus focal point. Focusing (changing the height of the lens along the optic axis) the object on the image plane results in a change in magnification.

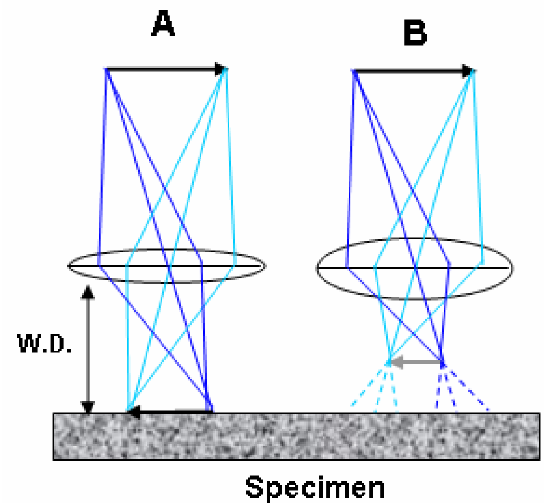
C: a hypothetical fixed position lens of variable strength (symbolized by the dashed lines) that achieves the same magnification change as in **B**.

There are a number of points to emphasize here when thinking about scanning electron microscopy.

1. The object being imaged is the source diameter (Gaussian intensity distribution) of the electron beam as it exits the gun.
2. We are interested in demagnifying (not magnifying) this beam source diameter. The amount of demagnification is simply p/q.
3. The type of lens used in SEMs is represented in **C**. SEMs have stationary electromagnetic lenses which we can vary the strength of by altering the amount of current running through them.
4. SEMs will have more than one electromagnetic lens. Under this circumstance the image plane of the first lens becomes the object plane of the second. The total demagnification is the product of the demagnification of lens one with lens two.

The objective lens is used to focus the beam on the specimen. Coarse focusing of the specimen is done by choosing the working distance (WD = distance between the bottom of the objective lens and the specimen); focusing the objective lens to coincide with this value; and then changing the physical height of the specimen to bring it into focus. Fine focusing is subsequently done solely with the objective lens.

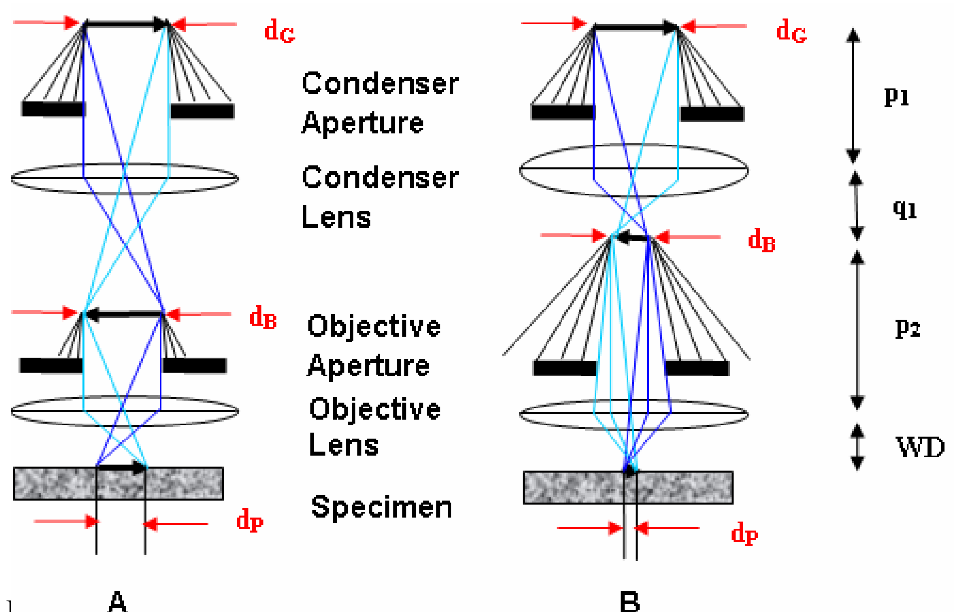
A: specimen in focus.
B: working distance needs to be decreased for specimen to be in focus



The figure below shows a simplified column with one condenser lens, an objective lens, and an aperture for each lens. The portion of the electron beam blocked by the apertures is represented with black lines. Both A and B show the source diameter of the electron beam exiting the gun (d_G) being focused to an electron probe (d_P) of a given diameter at the specimen. The diameter of the probe on the right is smaller. Why is this?

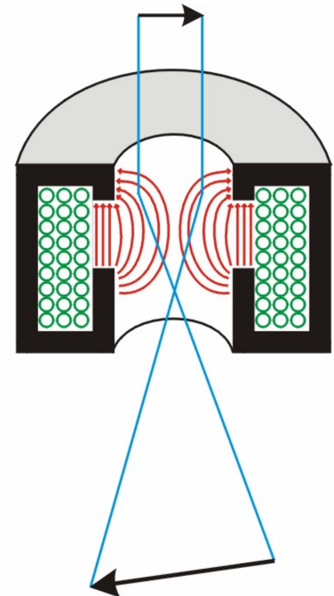
The amount of demagnification of the source diameter to the beam at d_B is p_1 / q_1. So the diameter of the beam at $d_B = d_G / (p_1 / q_1)$.

And likewise, the amount of demagnification of the beam at d_B to the electron probe (d_P) is p_2 / WD. So the diameter of the electron probe at the specimen $d_P = d_B / (p_2 / WD)$.



The strength of the condenser lens is stronger (thicker) in B than A. This results in a smaller diameter d_B and thus a smaller probe diameter on the specimen. A smaller probe diameter will enable better resolution but it comes at a cost. The stronger condenser lens setting in B causes more of the beam to be stopped by the objective aperture and thus a reduction in probe current occurs. Beam current increases to the 8/3 power as probe diameter increases. Adequate current is essential to produce images with the necessary contrast and signal to noise ratio.

An electromagnetic lens [2] consists of a coil of copper wires inside an iron pole piece. A current through the coils creates a magnetic field (symbolized by red lines) in the bore of the pole pieces which is used to converge the electron beam.



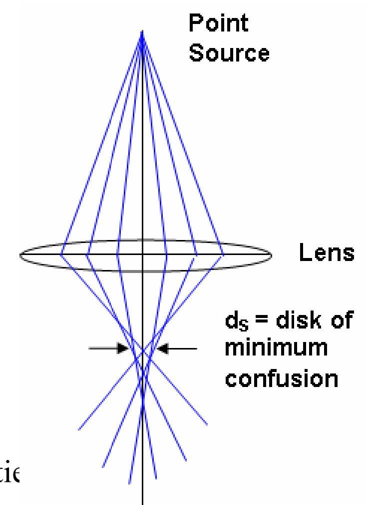
When an electron passes through an electromagnetic lens it is subjected to two vector forces at any particular moment: a force (H_Z) parallel to the core (Z axis) of the lens; and a force (H_R) parallel to the radius of the lens. These two forces are responsible for two different actions on the electrons, spiraling and focusing, as they pass through the lens. An electron passing through the lens parallel to the Z axis will experience the force (H_Z) causing it to spiral through the lens. This spiraling causes the electron to experience (H_R) which causes the beam to be compressed toward the Z axis. The magnetic field is inhomogeneous in such a way that it is weak in the center of the gap and becomes stronger close to the bore. Electrons close to the center are less strongly deflected than those passing the lens far from the axis.

So far we've mentioned that electromagnetic lenses are unlike optical lenses in that they are: stationary; have variable focal points; and cause the image to be rotated. The latter is corrected for in modern SEMs. Electromagnetic lenses also differ in that: the deflection of the electron within the lens is a continuous process (no abrupt changes in the refractive index); only beam convergence (not divergence) is possible; and the convergence angle with respect to the optic axis is very small compared with optical light microscopy (less than one degree!).

Finally, it is important to keep in mind that electron lenses, compared to glass lenses, perform much more poorly. Some have compared the quality of electron optics to that of imaging and focusing with a coke bottle. This is mainly due to the fact that aberrations are relatively easily corrected in glass lenses.

Lens Aberrations

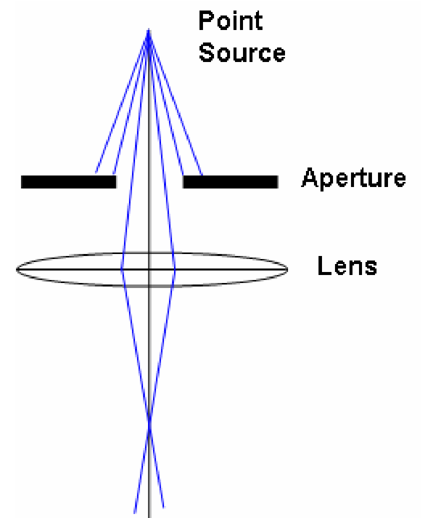
Up to this point, all of our representations depict a perfect lens. That is, all rays emanating from a point in the object plane come to the same focal point in the image plane. In reality, all lenses have defects. The defects of most importance to us are spherical aberration; chromatic aberration and astigmatism. Rather than a clearly defined focal point, we end up with a "disk of minimum confusion" in each instance.



Spherical aberration (d_S): The further off the optical axis (the closer to the electromagnetic pole piece) the electron is, the stronger the magnetic force and thus the more strongly it is bent back toward the axis. The result is a series of focal points and the point source is imaged as a disk

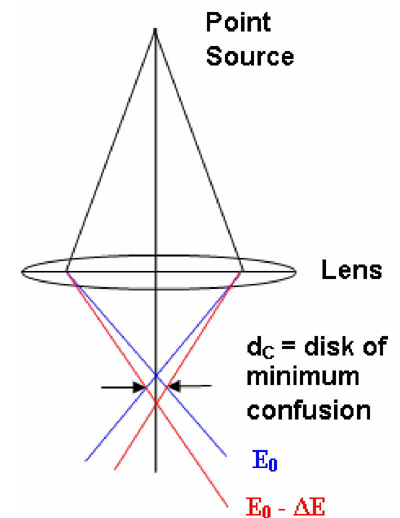
of finite size. Spherical aberration is the principle limiting factor with respect to the resolving power of the SEM

To reduce the effects of spherical aberration, apertures are introduced into the beam path. Apertures are circular holes in metal disks on the micron scale. The net effect of the aperture is to reduce the diameter of the disk of minimum confusion. However, as we mentioned earlier, that positive effect comes at the price of reduced beam current. Also, a very small aperture will display **diffraction effects (d_D)**. The wave nature of electrons gives rise to a circular diffraction pattern rather than a point in the Gaussian image plane. Manufacturers utilize apertures of a diameter that are a compromise of reducing spherical aberration and diffraction effects.



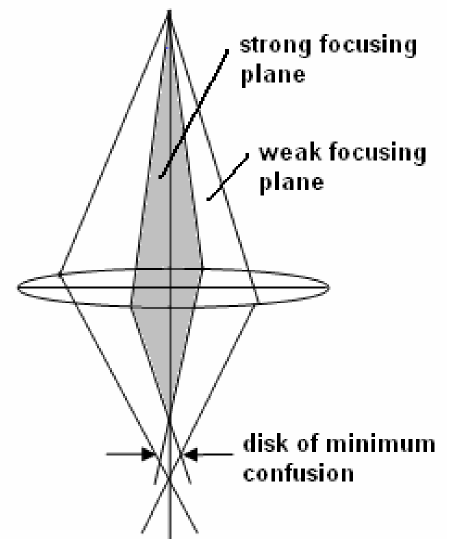
The diameter of the aperture used will also affect the convergence angle of the beam and this in turn will affect image properties such as depth of focus.

Chromatic aberration (d_C): The electron beam generated by the gun will have a certain energy spread. Electrons of different energies at the same location in the column will experience different forces. An electromagnetic lens will "bend" electrons of lower energy more strongly than those of higher energy. As with spherical aberration, a disk of minimum confusion is produced. Chromatic aberration is not something we can do much about as an operator and it becomes particularly problematic when imaging at low accelerating voltages.



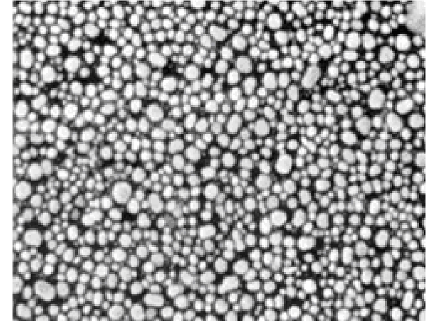
The actual probe size at the specimen = $(d_P^2 + d_S^2 + d_D^2 + d_C^2)^{1/2}$

Astigmatism: Finally, the electromagnetic lenses used in the SEM can not be machined to perfect symmetry. If the fields produced by the lenses were perfectly symmetrical, a converged beam would appear circular (looking down the column). A lack of symmetry would result in an oblong beam: the narrower diameter due to the stronger focusing plane; the wider diameter due to the weaker focusing plane. The net effect is the same as that of the aberrations above—a disk of minimum confusion rather than a well defined point of focus.



Operator Control

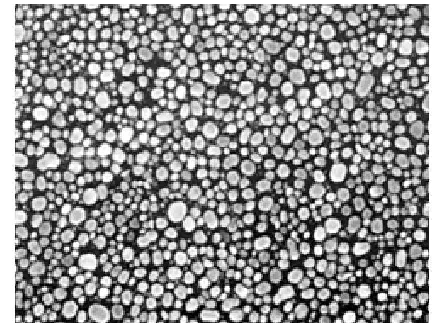
We can begin to talk about the column parameters you can control and what effects altering them will have. A more complete understanding will derive from our subsequent discussion of specimen-beam interactions.



Accelerating voltage: The accelerating voltage can be varied by the operator from < 1 kV to 30 kV on all four SEMs. Increasing accelerating voltage will:

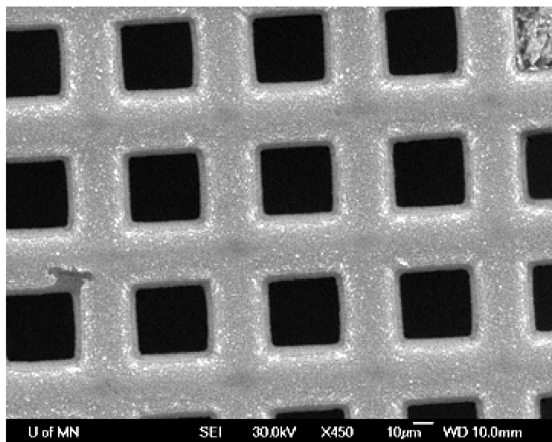
- decrease lens aberrations. The result is a smaller probe diameter (when considering it alone) and thus better resolution. Top right [3]: evaporated gold particles at 5 kV and 36 kX magnification; Bottom right [3]: 25 kV at the same magnification.

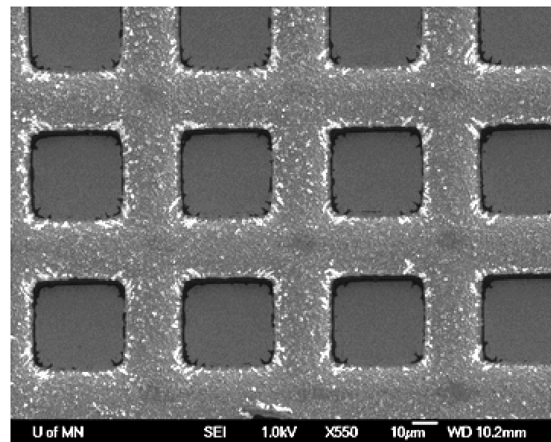
- increase the probe current at the specimen. A minimum probe current is necessary to obtain an image with good contrast and a high signal to noise ratio.



- potentially increase charge-up and damage in specimens that are non-conductive and beam sensitive.

- increase beam penetration into the specimen and thus obscure surface detail (more on this later). Below: 30 nm carbon film over a copper TEM grid. Left: 20 keV; Right: 2 keV. The carbon film is virtually invisible at the higher accelerating voltage.

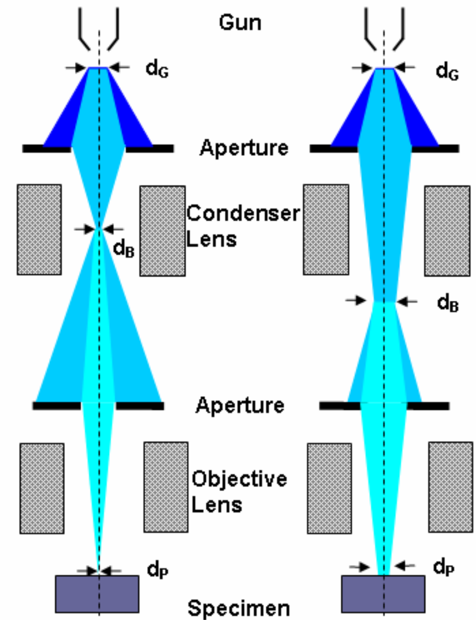




Emission current: The emission current can be varied (to different degrees) on all of CharFac's Cold FEGSEMs. Increasing emission current will:

- increase the probe current at the specimen.

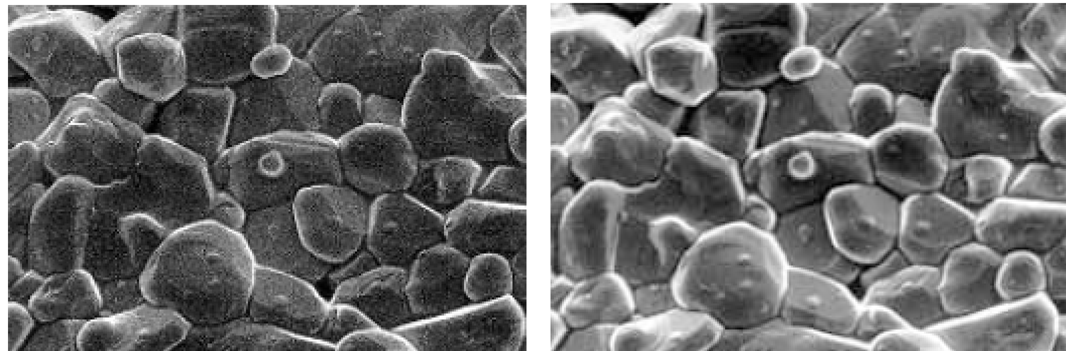
- potentially increase charge up and damage in specimens that are non-conductive and beam sensitive.



Probe diameter: The probe diameter or spot size can be varied on all four FEGSEMs by altering current to a condenser lens. I've redrawn an earlier diagram representing the electron beam with solid colors. Decreasing the probe diameter will:

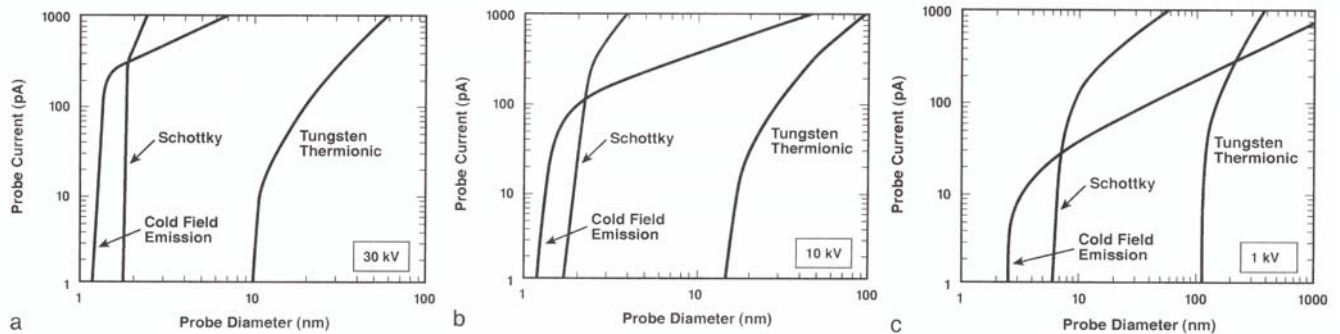
- enable greater resolution. Resolving small specimen features requires probe diameters of similar dimensions.
- decrease lens aberration due to a stronger lens setting.
- decrease probe current.

Bottom left [3]: ceramic imaged with a smaller spot size. Image is sharper but also grainier in appearance due to the lower signal to noise ratios associated with a lower beam current. Bottom right [3]: larger probe size results in a less sharp but smoother image in appearance.

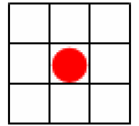


The relation between probe diameter and probe current for the cold FEG, thermally assisted FEG, and Tungsten thermionic gun at different accelerating voltages is shown below [4]. There are a few important points to make/reinforce here:

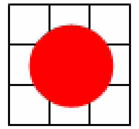
- You will get a smaller probe diameter with higher accelerating voltage for each of the gun types.
- The Tungsten thermionic gun has a larger minimum probe diameter due to the larger source size at the gun. Also, the increase of spot size with increasing probe current is most dramatic for this type of gun.
- The increase of spot size with increasing probe current is relatively small in FEGs until larger current values are obtained. This is particularly true at high accelerating voltages. Spot size will begin to increase at a greater rate at lower current values as the accelerating voltage decreases.



The picture element is the size of the area on the specimen from which the signal is collected. The table below gives the linear dimension of these pixels at various magnifications. **For a given choice of magnification, images are considered to be in sharpest focus if the signal that is measured when the beam is addressed to a given picture element comes only from that picture element.**



The probe diameter can be one of the contributing factors in determining the dimensions of the area on the specimen from which the signal is generated. As magnification increases and pixel dimensions decrease, overlap of adjacent pixels will eventually occur. What is surprising is that the overlap starts occurring at very low magnifications in the 5-30 kV range. For example, a 10 keV beam with a spot size of 50 nm focused on a flat surface of Aluminum will show overlap at 100 x magnification! Gold under the same circumstances will show overlap at 1000 x magnification! We will address the consequences of this later.

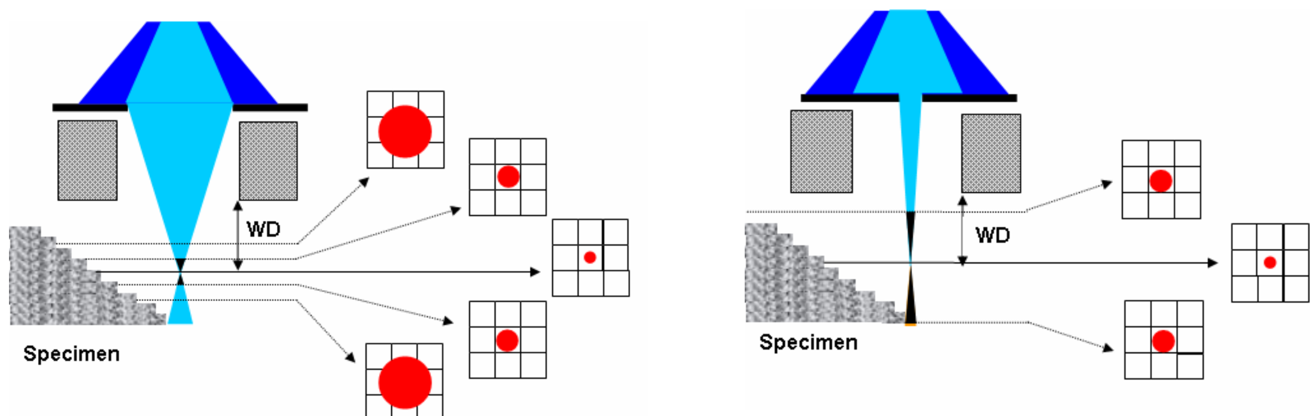


Magnification	Area on Sample (CRT screen: 10 x 10 cm)	Edge Dimension of Picture Element (1000 x 1000 pixel scan)
10	1 cm2	10 um
100	1 mm2	1 um
1,000	100 um2	100 nm
10,000	10 um2	10 nm
100,000	1 um2	1 nm

Objective aperture size: The objective apertures on all four FEGSEMS have a range of sizes that can be selected. Decreasing the diameter of the aperture will:

- decrease lens aberrations and thus increase resolution.
- decrease the probe current.
- decrease the convergence angle of the beam and thus increase depth of focus.

Bottom left: the larger diameter aperture results in a larger beam convergence angle and thus a reduced depth of focus. Sharp focus is obtained when the signal that is measured when the beam is addressed to a given picture element comes only from that picture element. The portion of the electron beam associated with sharp focus is shown in black.
Bottom right: the same working distance but a narrower diameter aperture and thus an increased depth of focus.

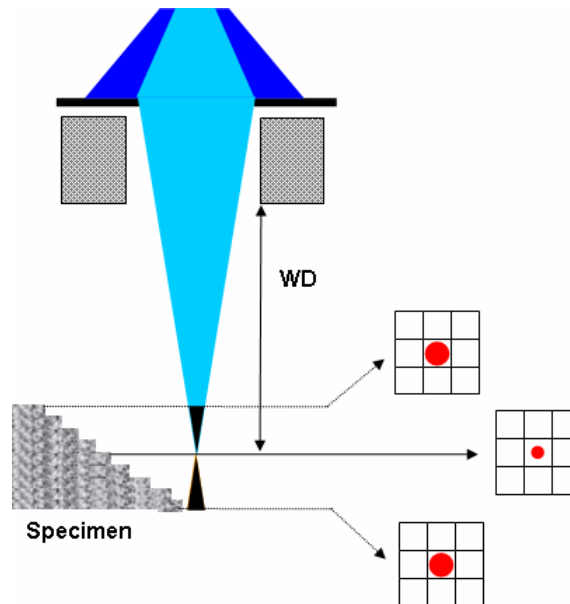


We mentioned earlier that manufacturers utilize apertures of a diameter that are a compromise of reducing spherical aberration and diffraction effects. Thus for the most part as an operator you will not be altering the size of the column apertures (there will be exceptions). But there is another way to increase depth of focus….working distance.

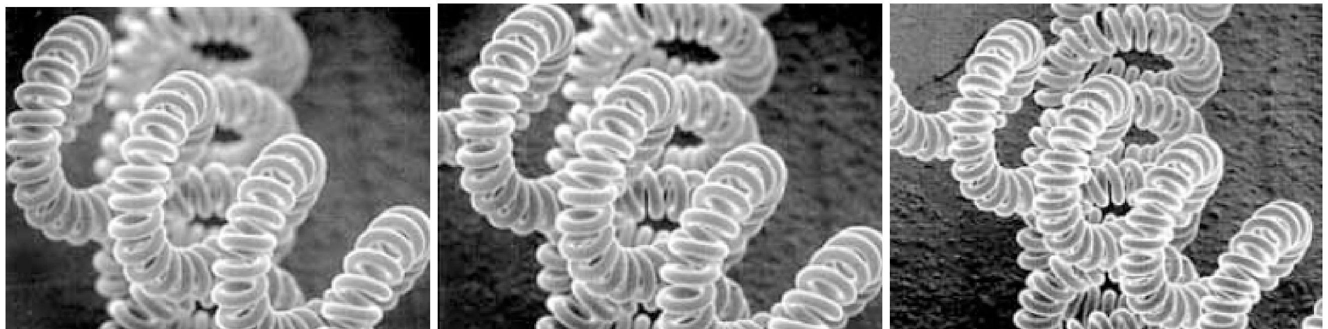
Working distance: The working distance is adjustable on all four FEGSEMs (the Hitachi S-900 has a very small range given its in-lens design). Increasing the working distance will:

- increase depth of focus.
- increase probe size and thus decrease resolution.
- increase the effects of stray magnetic fields and thus decrease resolution.
- increase aberrations due to the need for a weaker lens to focus.

The drawing below shows the larger aperture setup above with a greatly increased working distance. The result is an increased depth of focus but reduced resolving capabilities.

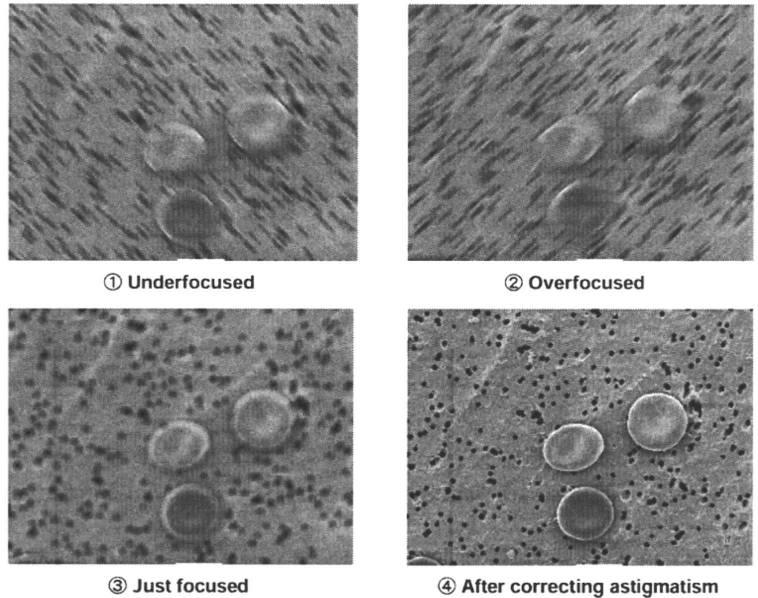


The images below reveal the effects of aperture diameter and working distance on resolution and depth of focus. Left [3]: light bulb coil with a 600 um aperture and 10 mm WD. Middle [3]: 200 um aperture and 10 mm WD. Right [3]: 200 um aperture and 38 mm WD.

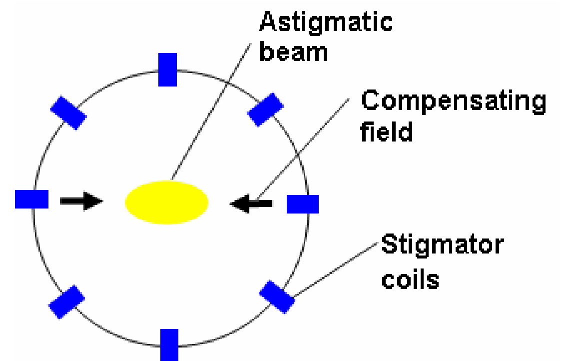


Focus and alignment:
An important aspect of aligning the microscope is ensuring that the apertures are centered with respect to the beam and thus the optical axis of the microscope. If an objective aperture is not centered the image will move when you try to focus it. The way to correct this is to wobble the current to the objective lens and align the aperture to minimize movement in both the X and Y plane. This correction is done at successively higher magnifications—course to fine adjustment.

Equally important is the correction of objective lens astigmatism at increasing magnifications. When extreme, astigmatism shows itself as a streaking of the image on both sides of focus. Even if streaking is not evident, astigmatism still needs to be fine-tuned to obtain quality images [5].



The way an operator compensates for objective lens astigmatism is by applying current differentially to a ring of stigmator coils around the objective lens. The compensating field in the image to the right will cause the beam to assume a more circular (symmetric) form



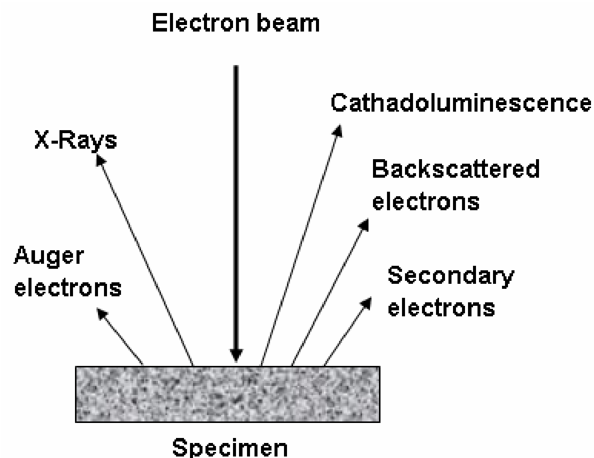
The operator uses the objective lens to focus the specimen. Since an increase in magnification simply corresponds to a smaller rastered area on the specimen, it makes sense to do fine focusing at high magnifications. When you subsequently decrease your magnification the focus will be very good. So, a useful heuristic would be to get a sense of the highest magnification at which you wish to obtain images, double that magnification value, and conduct your final aperture alignment, astigmatism correction, and focus there, then come down to where you wish to acquire images.

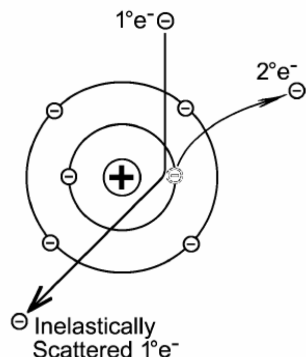
It should be noted that alignment is something you will likely do often. It is necessary when you change accelerating voltage, spot size etc.

Beam-Specimen Interactions

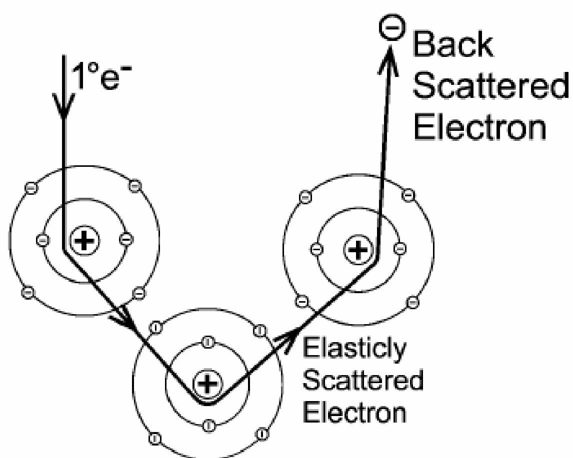
Signals

The beam electron can interact with the coulomb (electric charge) field of both the specimen nucleus and electrons. These interactions are responsible for a multitude of signal types: backscattered electrons, secondary electrons, X-Rays, Auger electrons, cathadoluminescence. Our discussion will focus upon backscattered and secondary electrons.





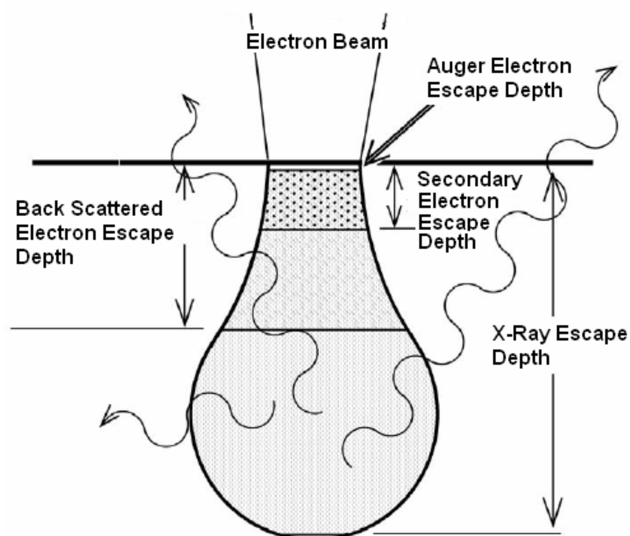
Inelastic events [6] occur when a beam electron interacts with the electric field of a specimen atom electron. The result is a **transfer of energy** to the specimen atom and a potential expulsion of an electron from that atom as a **secondary electron (SE)**. SEs by definition are less than 50 eV. If the vacancy due to the creation of a secondary electron is filled from a higher level orbital, an X-Ray characteristic of that energy transition is produced.



Elastic events [6] occur when a beam electron interacts with the electric field of the nucleus of a specimen atom, resulting in a change in direction of the beam electron **without a significant change in the energy** of the beam electron (< 1 eV). If the elastically scattered beam electron is deflected back out of the specimen, the electron is termed a **backscattered electron (BSE)**. BSEs can have an energy range from 50 eV to nearly the incident beam energy. However, most backscattered electrons retain at least 50% of the incident beam energy.

Interaction volume

The combined effect of the elastic and inelastic interactions is to distribute the beam electrons over a three-dimensional **"interaction volume"** [6]. The interaction volume can have linear dimensions orders of magnitude greater than the specimen surface under the beam foot print.



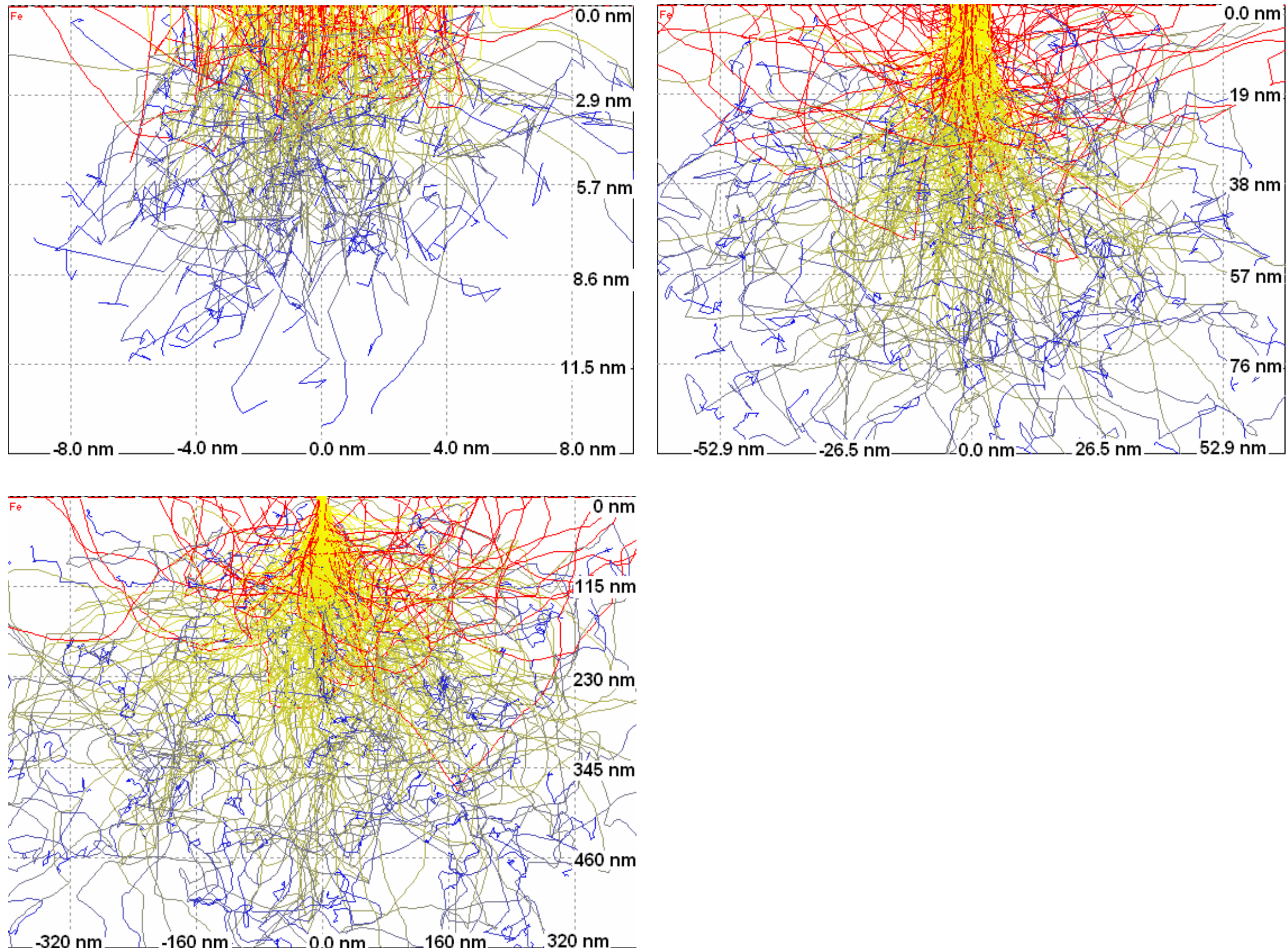
Secondary and backscattered electrons have different maximum escape depths given their energy differences. The escape depth of SEs is approximately 5-50 nm; BSEs can escape from a depth a hundred times greater, and X-Rays greater yet. Since there is a certain symmetry to the

interaction volume, greater escape depths generally translate to wider lateral dimension from which the signal can generate and thus lower potential resolutions.

The actual dimensions and shape of the interaction volume are dependent upon a number of parameters: accelerating voltage, atomic number and tilt. We will use a Monte Carlo simulation (CASINO) [7] of the interaction volume below to demonstrate some of these effects. The backscatter electron signal is shown in red; the energy range of the electron beam within the sample is shown from high (yellow) to low (blue).

Accelerating voltage: The dimensions of the interaction volume will increase with accelerating voltage. As the beam energy increases the rate of energy loss in the specimen decreases and thus the beam electrons penetrate deeper into the sample. Also, the probability of elastic scattering is inversely proportional to beam energy. With less elastic scattering the beam trajectories near the specimen surface become straighter and the beam electrons penetrate more deeply. Eventually the cumulative effects of multiple elastic scattering cause some electrons to propagate back towards the surface -- thus widening the interaction volume.

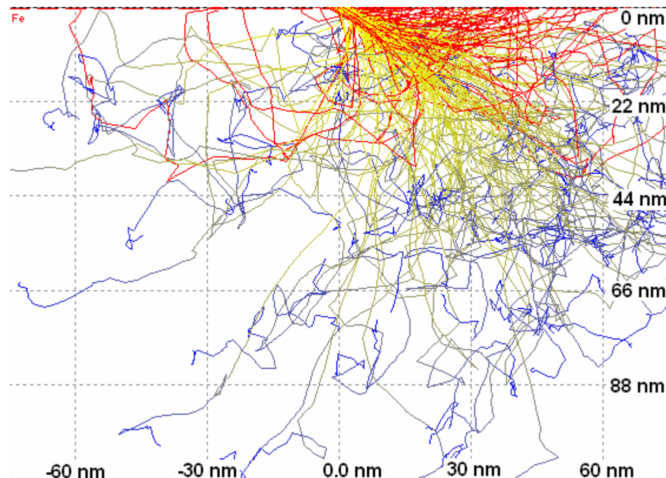
Below left to right and top to bottom: A bulk sample of iron at 1, 5, and 15 kV; 10 nm probe diameter.



Atomic number: The dimensions of the interaction volume will decrease with higher atomic number elements. The rate of energy loss of the electron beam increases with atomic number and thus electrons do not penetrate as deeply into the sample. Also, the probability for elastic scattering and the average scattering angle increase with atomic number—causing the interaction volume to widen.

Below left to right and top to bottom: Bulk samples of Carbon, Iron and Gold at 5 kV; 10 nm probe diameter.

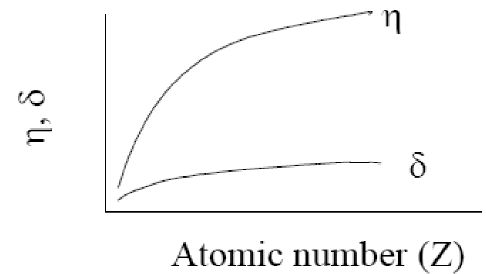
Tilt: The interaction volume becomes somewhat smaller and more asymmetric with the degree of tilt. Below: Iron at 5 kV and 60 degrees of tilt



Backscatter electron signal

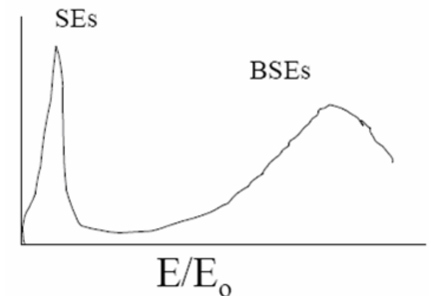
The backscatter coefficient (η) is the ratio of the number of BSEs to the number of beam electrons incident on the sample.

η and atomic number: η shows a monotonic increase with atomic number. The above Monte Carlo simulations of Carbon, Iron and Gold at 5 kV had the following respective η values: 0.08, 0.335, and 0.44. This relationship forms the basis of **atomic number (Z) contrast**. Areas of the specimen composed of higher atomic number elements emit more backscatter signal and thus appear brighter in the image. As we can see from the slope of the line, Z contrast is relatively stronger at lower atomic numbers.

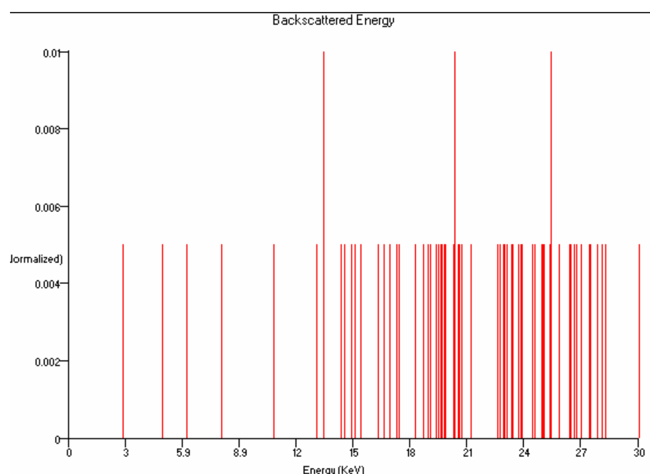


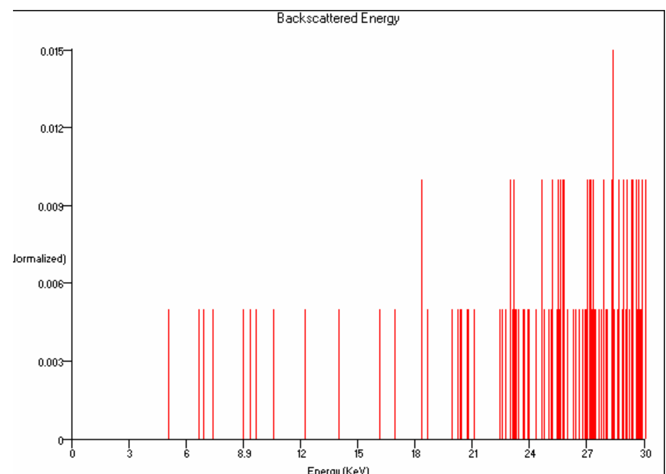
η and accelerating voltage: There is only a small change in η with accelerating voltage (< 10% in the 5-50 keV range). As the accelerating voltage is reduced toward the very lower end (1 keV), η increases for low Z elements and decreases for high Z elements.

BSE energy range: BSEs follow trajectories which involve very different distances of travel in the specimen before escaping. The energy range for BSEs is thus wide (from 50 eV to that of the incident beams energy). The majority of BSEs, however, retain at least 50% of the incident beam energy (E_0). Generally speaking, higher atomic number elements produce a greater number of higher energy BSEs and their energy peak at the higher end is better defined.



Below left: BSE energy distribution for Iron at 30 kV; Below right: BSE energy distribution for Gold at 30 kV

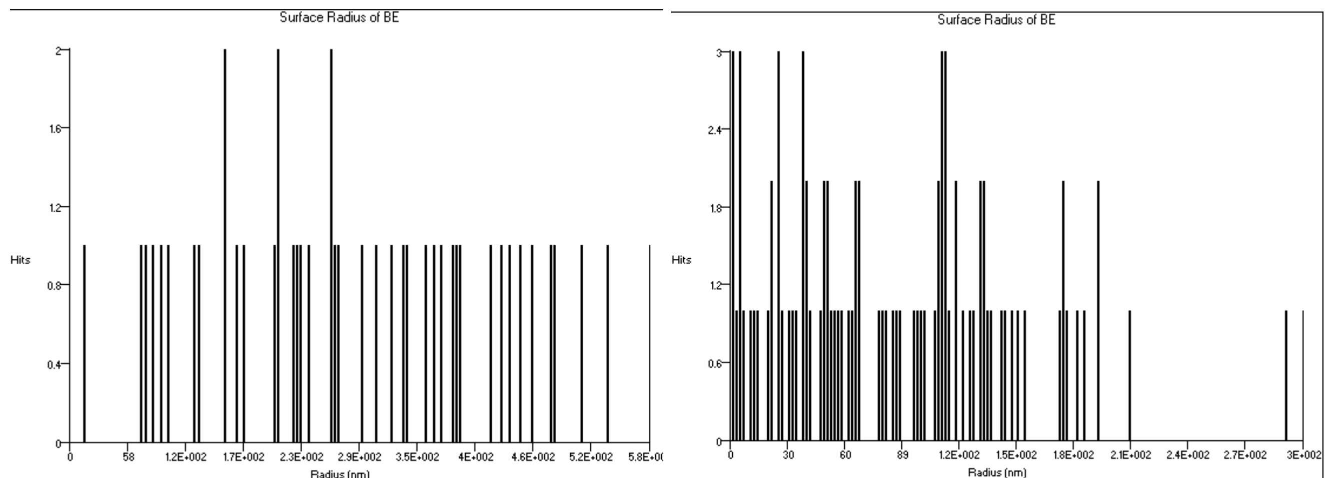




Lateral / depth dimensions: Both the lateral and depth dimensions from which the BSE signal derives increase with accelerating voltage and decrease with atomic number. Some ballpark values are shown in the table below (units are microns; first value is the maximum escape depth; second value is the lateral dimension from which the BSE signal derives).

Specimen	1 keV	5 keV	15 keV	30 keV
Al	0.011 / 0.035	0.11 / 0.4	0.7 / 3.5	2.3 / 10.6
Cu	0.008 / 0.018	0.05 / 0.12	0.28 / 0.94	0.8 / 3.2
Au	0.004 / 0.014	0.034 / 0.09	0.16 / 0.42	0.45 / 1.3

The lateral spatial distribution of the BSE signal resembles a sombrero with a central peak and broad tails. Higher atomic number elements have a larger fraction of their signal derived from the peak and thus have a potentially higher resolution signal. Below left: Iron at 15 kV; Below right: Gold at 15 kV. Y-axis: number of signals; X-axis: distance from the incident electron beam.



Angular distribution: As the surface is tilted, η increases but in a direction away from the incident beam. With no tilt, η follows a distribution that approximates a cosine expression. What this means is that the maximum number of backscattered electrons travel back along the incident beam!

Secondary electron signal

The secondary electron coefficient (**δ**) is the ratio of the number of secondary electrons to the number of beam electrons incident on the sample.

δ and atomic number: **δ** is relatively insensitive to atomic number. For most elements **δ** is approximately 0.1. A couple of notable exceptions are Carbon (0.05) and Gold (2.0).

δ and accelerating voltage: There is a general rise in **δ** as the beam energy is decreased due primarily to the reduction in interaction volume which occurs.

SE energy range: By definition SEs have an energy of less than 50 eV. Ninety % of SEs have energies less than 10 eV; most, from 2 to 5 eV.

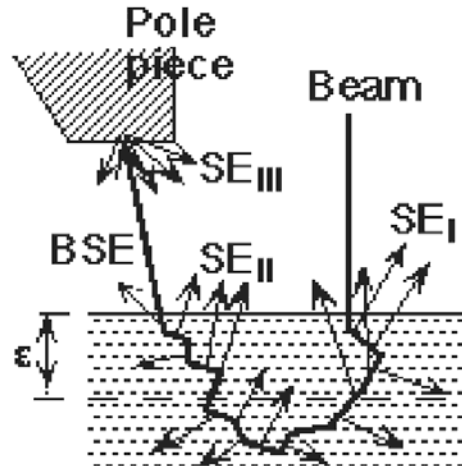
Lateral and depth dimensions: SEs have a shallow maximum escape depth due to their low energy levels. For conductors the maximum escape depth is approximately 5 nm; for insulators it is around 50 nm. Most secondary electrons escape from a depth of 2-5 nm.

SEs are generated by 3 different mechanisms [8]:

- SE(I) are produced by interactions of electrons from the incident beam with specimen atoms. These SEs are produced in close proximity to the incident beam and thus represent a high lateral resolution signal.

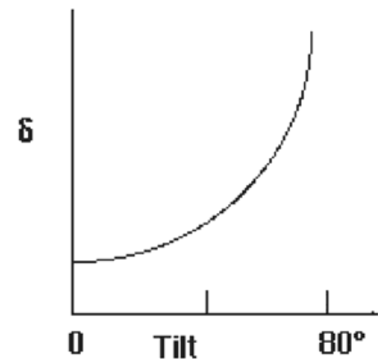
- SE(II) are produced by interactions of high energy BSEs with specimen atoms. Both lateral and depth distribution characteristics of BSEs are found in the SE(II) signal and thus it is a comparatively low resolution signal.

- SE(III) are produced by high energy BSEs which strike the pole pieces and other solid objects within the specimen chamber

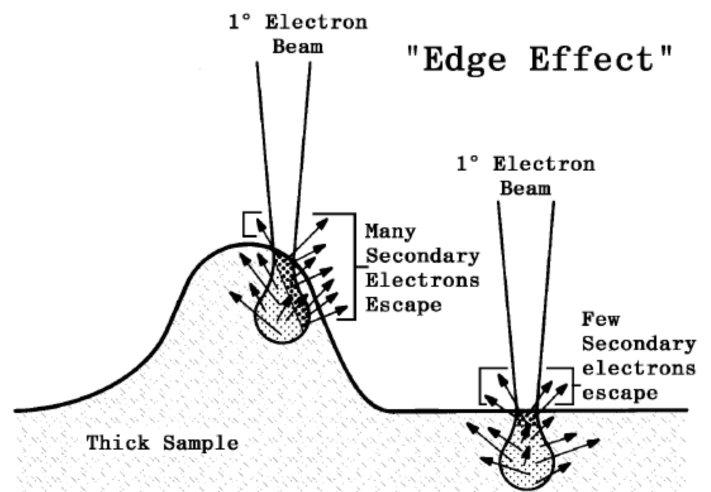


In general the δ of SE(II) / δ of SE(I) is about three. The SE(I) signal dominates for light elements since backscattering is low; the SE(II) signal dominates for heavy elements since backscatter is high.

Tilt: When the sample is tilted the incident beam electrons travels greater distances in the region close to the surface. As a result, more SEs are generated within the escape depth in these areas than in areas which are normal to the beam.

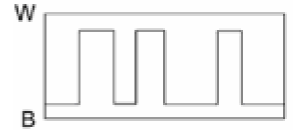


Images produced with the SE signal will reveal something termed the "edge effect" [6]. Edges and ridges of the sample emit more SEs and thus appear brighter in the image.



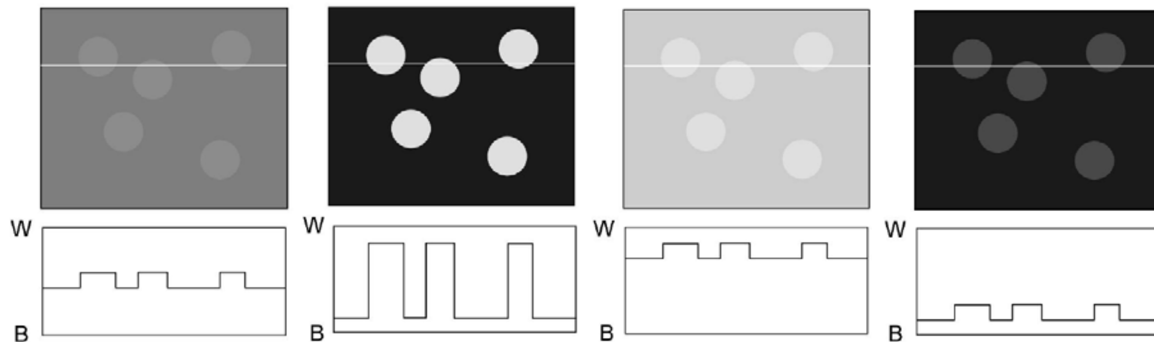
Contrast

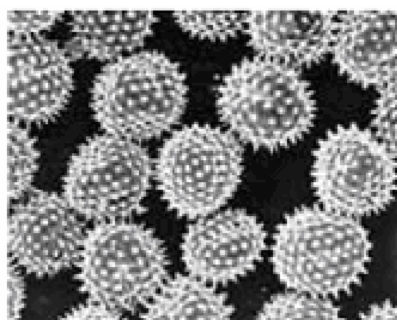
A high resolution, high intensity signal resulting from a scan will reveal nothing if contrast is absent. Contrast can be defined as $(S_2 – S_1) / S_2$ where S_2 is the signal from the feature of interest; S_1 is the background signal; and $S_2 > S_1$. Interpreting grey scale images requires an understanding of the origin of contrast mechanisms. For most users the minimum useful image contrast level is about 5 % (contrast lower than this can be enhanced via image processing).

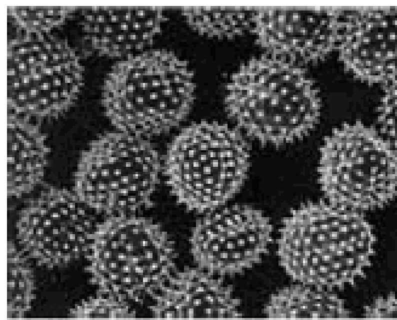


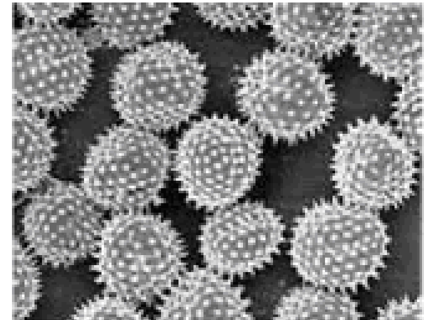
The difference between peak white (W) and peak black level (B) defines the dynamic range of the signal viewed on the SEM monitor. A line scan across a specimen is shown to the right. The X axis corresponds directly to the distance scanned across the specimen; the Y axis represents the dynamic range of the signal. The base level of the signal is the **brightness** or the standing dc level of the amplifier output. The spread of the signal up from this baseline is the **contrast** and is varied with the gain of the amplifier. The contrast should span as much as possible of the dynamic range because this produces the most useful and pleasing images.

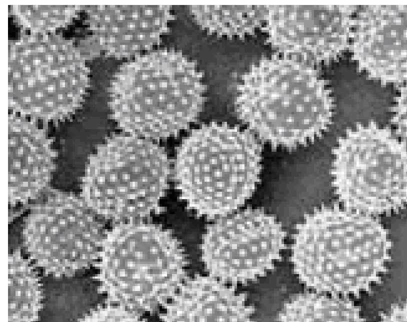
Both the line scan and area scan images [6, 3] below show the relationship between brightness and contrast. Most microscopes have an automatic brightness and contrast control. Manually adjustment involves: turning down the contrast; adjusting brightness so that it is just visible on the screen; and then increasing the contrast to produce a visually appealing image.

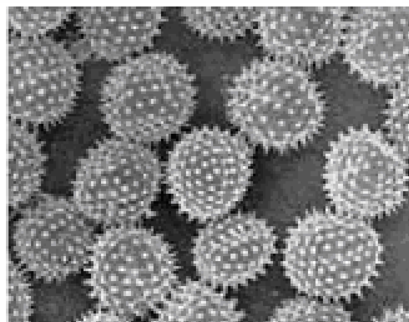



(1) Excessive contrast


(2) Insufficient brightness

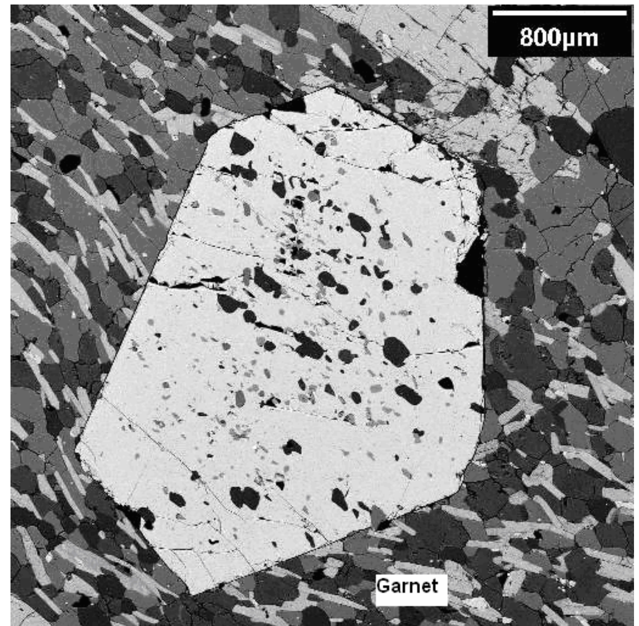

(3) Optimum contrast and brightness


(4) Excessive brightness


(5) Insufficient contrast

Compositional (atomic number) contrast:

Compositional contrast results from different numbers of backscattered electrons being emitted from areas of the sample differing in atomic number. The backscatter coefficient increases with increasing atomic number and so higher atomic number elements will appear brighter in the image. Elements widely separated in atomic number will result in the greatest contrast. For pairs of elements of similar atomic number, the contrast between them decreases with increasing atomic number.



We mentioned earlier when discussing the angular distribution of BSEs that a large percentage travel back along the incident beam. The backscatter detectors we have at CharFac are mounted directly under the objective lens to take advantage of this fact. The detectors are circular and have a hole in the middle to allow the electron beam to pass through.

The JEOL 6500, Hitachi S-4700, and the Hitachi S-900 all have dedicated backscattered detectors. They are designed to preferentially use the higher energy (and thus higher resolution) BSE signal. The 6500 detector can obtain images with accelerating voltages as low as 1 kV; however, the resolution is less than that obtained by the S-4700 and S-900. The S-4700 and S-900 detectors require approximately a minimum of 5 kV to obtain quality images.

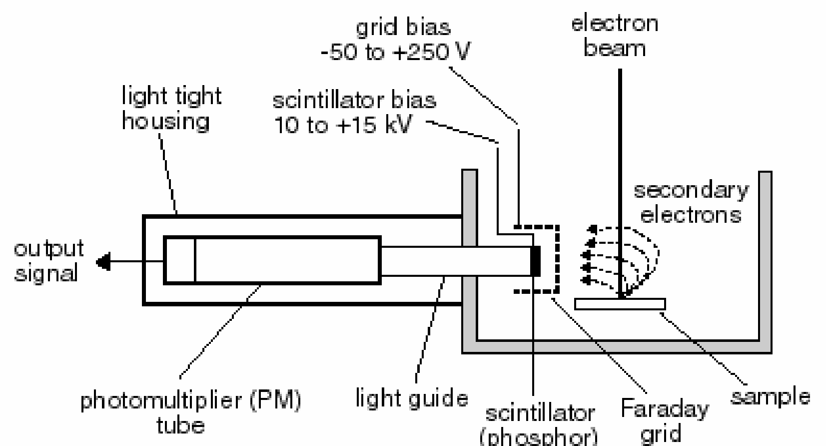
Obviously one would want to use a backscattered detector to assess whether a sample had compositional differences—perhaps as a precursor to determining the specific elements present with Energy Dispersive Spectroscopy. Also, BSE imaging is one strategy to deal with samples that present charging problems (more on this later).

Topographic contrast:

Topographic contrast enables us to image the size, shape and texture of three dimensional objects. It is dependent upon the number of SEs and BSEs being emitted from different areas of the specimen as well as the trajectories they take in relation to the location of the detector.

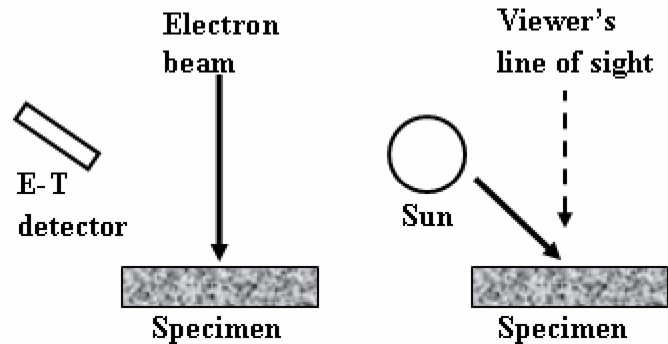
The most common SE detector is the Everhart Thornley (ET) detector. All four of CharFac's SEMs have an ET detector mounted on the side of the specimen chamber.

A Faraday cage over the detector can be variably biased from -50 to +250 V. A small negative bias will exclude low energy secondary electrons but will receive high energy backscattered electrons whose path is



line of sight with the detector. A positive bias to the faraday cage will allow the detector to attract the entire range (SE I--III) of secondary signals as well as the line of sight BSE signal. A positive potential (10-12 kV) is applied to the face of the phosphor coated scintillator to ensure that the electrons accepted through the Faraday cage will be accelerated sufficiently to generate photons. The photons are conducted by a light guide to the photomultiplier where they are converted back to electrons and amplified up to a million-fold. This photomultiplication process provides high gain with little noise degradation.

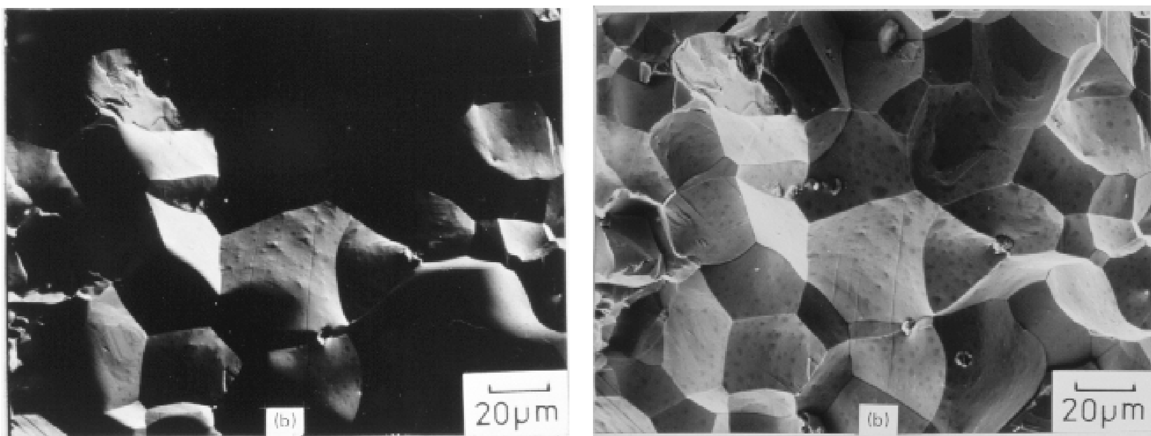
The fact that SEM images can be readily interpreted by viewers derives from the "Light optical analogy". The direction of the detector within the image is analogous to where the "illumination" appears to come from. Since we are used to having the illumination from overhead, we rotate the scanned image so that detector/sun appears to be at top of the image. The viewer's line of sight is analogous to the direction of the electron beam.



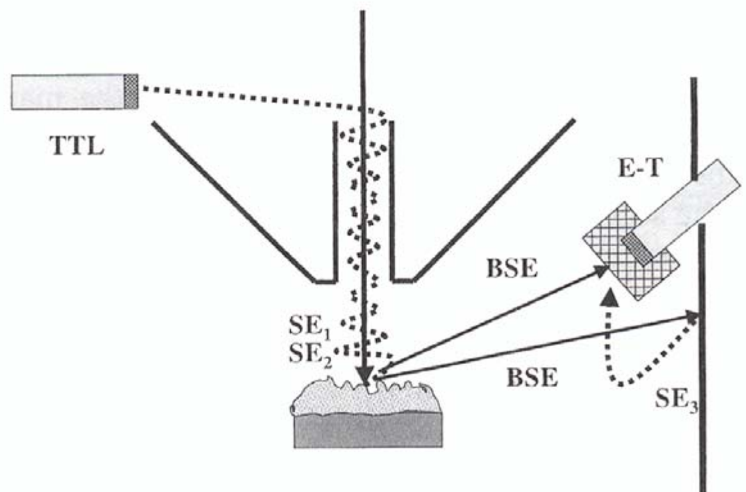
The images below [4] are of polycrystalline iron. Both images were obtained from a side mounted ET detector.

Left image: A small negative bias is applied to the ET detector which excludes low energy SEs. However, high energy BSEs whose path is line of sight with the detector are responsible for the contrast in the image. Compositional contrast would also be apparent in the image if the specimen surfaces responsible for the line of sight BSEs varied in their elemental makeup.

Right image: A small positive bias is applied to the ET detector (this is the normal detector setting). Now the signal source for the image has both a BSE and SE component. BSEs from surfaces oriented toward the detector and whose path is line of sight with the detector contribute to the grayscale image. And again, to the extent that these surfaces are varied in their elemental makeup, there will be a small compositional contrast component to the image. The small positive bias will also attract the entire range (I – III) of low energy SEs to the detector. Surfaces facing the detector will contribute relatively more SEs to the image than those facing away. So, we have a strong numbers and trajectory component to both BSEs and SEs when utilizing the ET detector.



Both the JEOL 6700 and Hitachi S-4700 have an additional detector ("through the lens") positioned up in the vicinity of the objective lens [4]. The magnetic field of the objective lens projects into the sample chamber and high resolution SE Is and SE IIs are trapped by this field and follow spiral trajectories around the lines of magnetic flux up through the lens. Off axis SE IIIs and BSEs are excluded. Shorter working distances are required when using these detectors. The image provided by the upper detector shows sharp edges and clearly defined details at high resolution/magnification. However, there is less topography observed using the upper detector. Specimens prone to charging may be more difficult to image with the upper detector given the reduced BSE signal. It is also possible to image with a mix of lower and upper detectors to achieve a desired effect.



Below left [1]: lower detector; Below right [1]: upper detector.

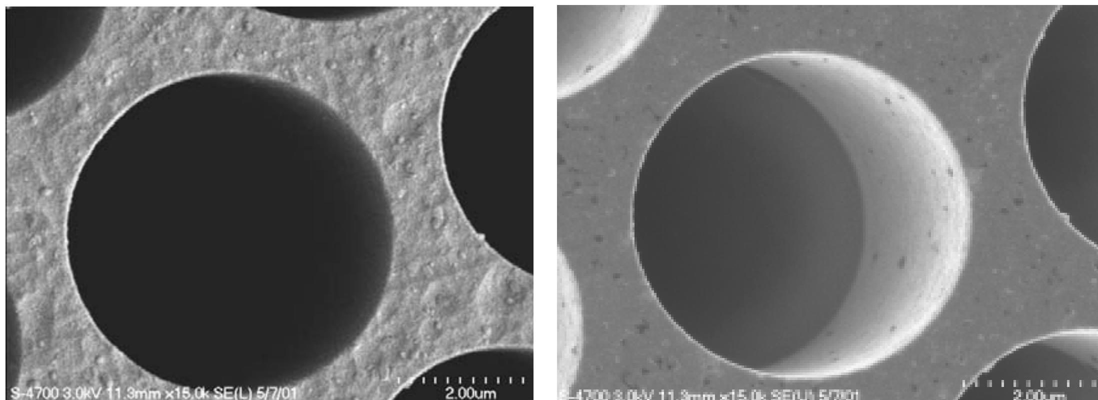
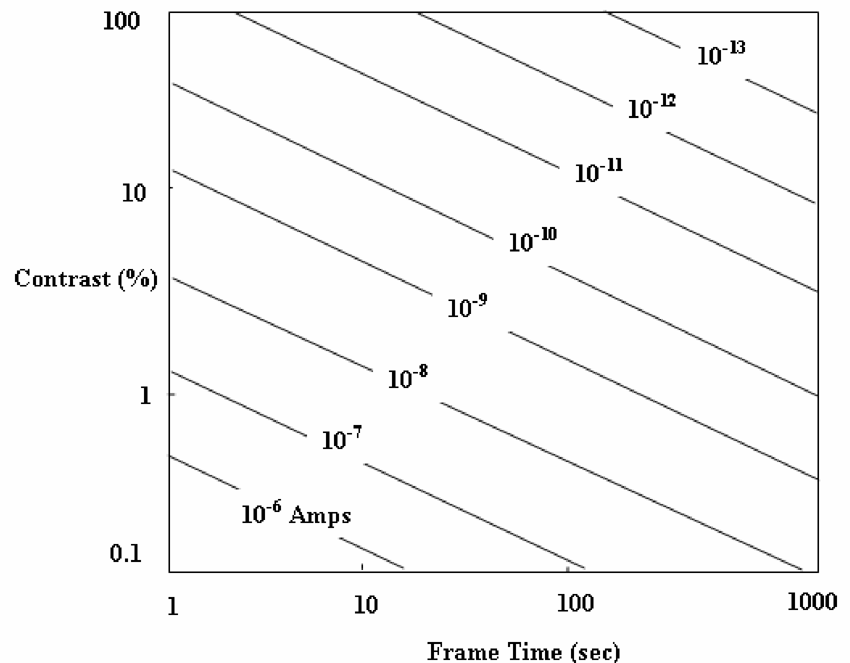


Image quality:

High quality images require a high signal to noise ratio. The signal intensity from multiple scans from a given pixel will display itself as a Gaussian distribution. The mean (n) of that distribution corresponds to the signal (S) and the standard deviation ($n^{1/2}$) corresponds to the noise (N). The signal to noise ratio = $S/N = n / n^{1/2} = n^{1/2}$.

An observer can distinguish small features in the presence of noise provided that the change in signal ΔS is greater than the noise (N) by a factor of 5. Thus the smallest beam current that will permit a given contrast level ($C = \Delta S/S$) to be discerned is: $n > (5/C)^2$.



A perhaps more intuitive way to think about the relationships between contrast, beam current and scan rate is the graph to the right. **It is necessary to use some combination of high beam current and a slow scan speed in order to detect objects of small size and low contrast in an SEM.**

High resolution imaging

The resolution is the minimum spacing at which two features of the specimen can be recognized as distinct or separate. In order to obtain high resolution images we need to adjust the probe diameter to the scale of interest and ensure that a minimum level of contrast exists with the appropriate probe current and scan rate settings.

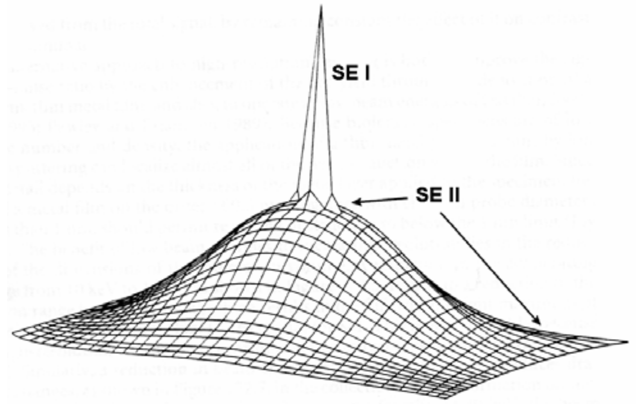
In addition, we now know that the high resolution signal comes from the SE Is that are generated within a few nanometers of the incident beam, and the SE IIs that are generated from BSEs of high energy and thus close proximity to the beam. We also know that, even with small probe diameters, the interaction volume can be quite large and pixel overlap will occur at surprisingly small magnifications.

Given the above, there are two ways to achieve high resolution [4]:

1. separate the high from the low resolution signal (high resolution at high kV).

2. use operating conditions that create similar ranges for the "high" and "low" resolution signals (high resolution at low kV).

High resolution at high kV:

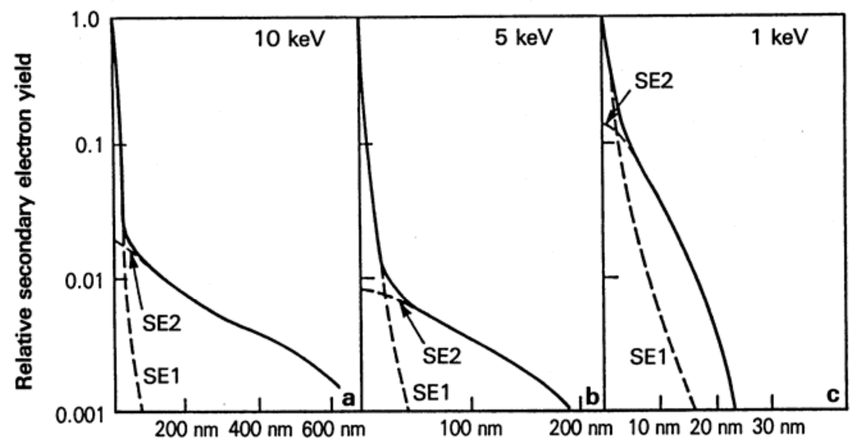


The image to the right [4] shows the SE emission from a 30 keV, 1 nm probe diameter beam focused on a Si target. The FWHM of the SE I signal is 2 nm; the FWHM of the SE II signal is 10 um! Consider what is occurring when scanning at lower magnifications when the pixel size is large. The SE II component of the signal will change in response to features of the field of view on the size scale of one pixel and larger. The SE I signal will also change, but since the region from which it emanates is so small compared to the pixel size, it will register as random discrete noise. Under these conditions we will have a low resolution image.

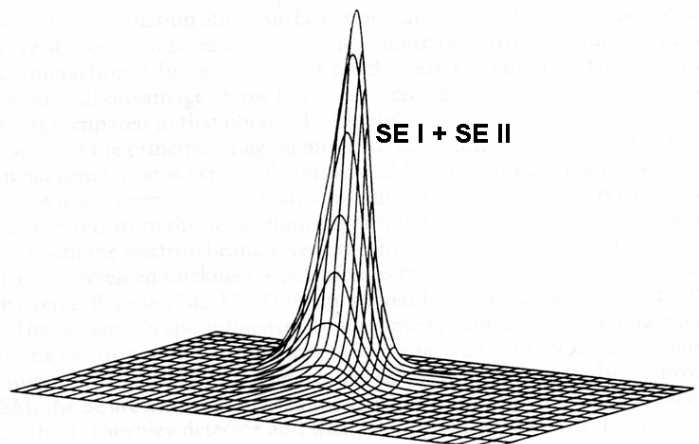
Now consider what happens at high magnification (> 100 kX). Now the field of view is much smaller than the interaction volume. During scanning the effective change in the SE II distribution is small; its magnitude remains relatively constant; and thus its contribution to detail in the image is minimized. The SE II signal does, however, contribute a random noise component to the signal. Thus the changes in the total signal are almost entirely due to the high resolution/information SE I component.

High resolution at low (< 5) kV:

We found earlier that the dimensions of the interaction volume decrease rapidly with incident beam energy. Under these conditions the FWHM dimensions of the SE II signal begins to approach that of the high resolution SE I signal [9].

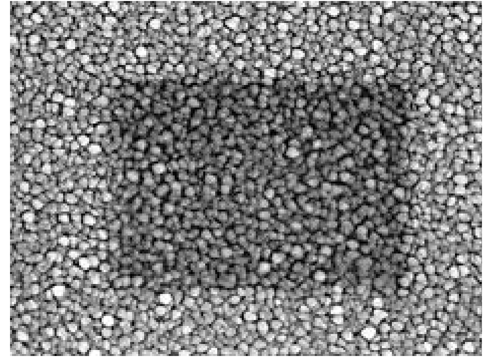


Thus there is no need to separate one signal from the other. At 1 keV the probe diameter is now approximately 3-4 nm, but now the SE IIs are emitted from approximately the same area as SE Is. This also means that dedicated backscattered imaging can begin to approach the resolution of SE imaging at these low accelerating voltages.



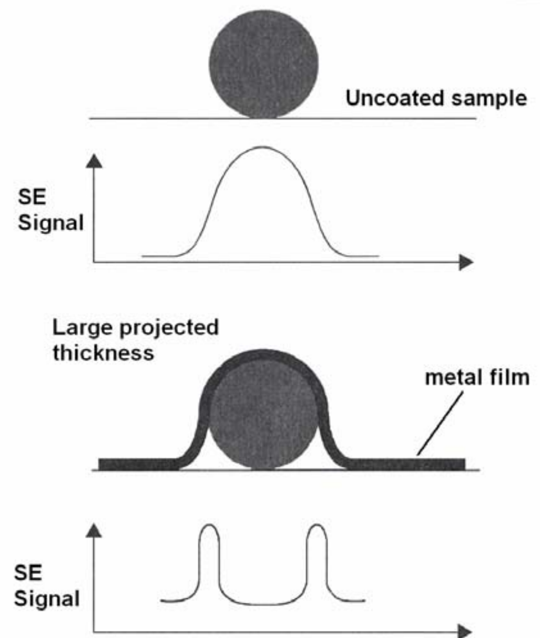
There is a relatively good S/N ratio at these very low accelerating voltages because the probe current decrease due to small probe diameters is offset by the rise of the SE signal with decreasing accelerating voltage. The

downside of low kV imaging is that the performance of the optical system is decreased due to an increase in chromatic aberration and the beam is more susceptible to stray magnetic fields.



In addition, contamination can be a serious limitation to low voltage microscopy [3]. Contamination results from the interaction of the electron beam with residual gases and hydrocarbons on the specimen surface. SE imaging in particular is vulnerable to contamination given their low energy level.

Contamination can be reduced by:
- ensuring the cleanliness of specimens;
- decreasing the probe current;
- avoiding high magnification except where essential;
- aligning the microscope on areas of the specimen not used for imaging;
- using a cold finger (anticontamination trap) in the column if available.



For both methods of high resolution imaging, low atomic number targets can be challenging due to low SE yield and thus poor signal to noise ratio. The SE yield can be increased substantially with the application of an ultrathin metal coating (Platinum for example). Coatings from approximately 1-5 nm are applied. Thicker coatings in this range approximate the escape depth for SEs and thus produce a high SE yield. However, the metal grains in thicker coatings may become apparent in an image above 100 kX. High resolution imaging will thus make use of coatings on the lower end of the range and sacrifice some SE yield. The net result of the thin metal coating is the ability to resolve small features. Right [4]: The uncoated sample appears as a bright blur with no defined edges. In the coated sample, the rapid change in metal thickness that occurs as the beam is scanned across the specimen leads to well defined SE signal maxima which in turn allows the size and shape of the feature to be clearly resolved. Coating is also a remedy for the charging that occurs with non-conductive samples under the beam

Charging

Charging is the condition when a material cannot effectively conduct the beam energy imparted to it. A large fraction of the probe current remains in the specimen as the beam electrons lose all of their initial energy and are captured by the specimen. This charge flows to ground if the specimen is a conductor and a suitable connection exists. If the ground path is broken, even a conducting specimen quickly accumulates charge and its surface potential rises. Non-conducting specimens will obviously accumulate charge. The ensuing image will “glow” or cause streaks or general distortion in the image as electron production is artificially enhanced and the beam is unintentionally deflected.

Both conducting and non-conducting samples are attached to their holders with conductive tapes or paints. Non-conducting samples are also coated with thin conductive films (Carbon, Gold-Palladium, Platinum etc) to facilitate electron flow.

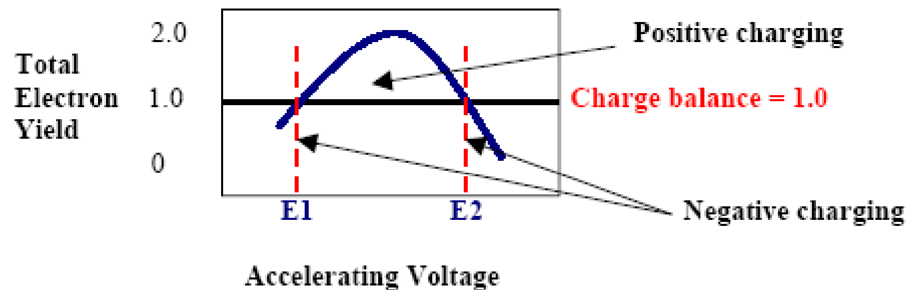
Scan-square method of testing the conductivity of a specimen [1]:

1. Focus on an area at a high magnification.
2. Wait a few seconds, letting the beam irradiate the selected area.
3. Reduce the magnification and observe the sample.
4. If a bright square appears (which may disappear upon going to the lower magnification), negative charging is probable. Therefore, lower the voltage.
5. If a dark square appears, and then quickly disappears, positive charging is probable. Therefore, raise the voltage. Note: If the dark square remains, contamination is most likely the problem.
6. The effect of charging is exacerbated at higher magnifications, so be sure to check these conditions at levels near to or at the highest magnification you intend to use.

In addition to the application of thin metal coatings, the following strategies can be used to reduce charging:

- reducing the probe current;
- lowering the accelerating voltage;
- tilting the specimen to find a balanced point between the amount of incident electrons and the amount of electrons that go out of the specimen;
- imaging with BSEs;
- use rapid scan rates and frame integration;
- find the E2 value for insulators.

E2 Value [1]**:** When the sum of the BSE and SE coefficient is measured at very low kV, it is found that there is an energy range over which this sum is positive. This positive form of charging results from more electrons being emitted from the sample than the primary electron beam provides. Rather than glowing, a dark box (from the scan footprint) will appear as secondary electrons are emitted. Flanking this area of positive charging are voltages at which the sample achieves charge balance: E1 and E2. For each material, E1 and E2 are constants. E2 is the value to seek for uncoated observation since it is found at a higher accelerating voltage, thus utilizing a higher resolution condition of the SEM.



Material	E2 (kV)
Kapton	0.4
Electron resist	0.55—0.7
Nylon	1.18
5% PB7/nylon	1.40
Acetal	1.65
Polyvinyl chloride	1.65
Teflon	1.82
Glass passivation	2.0
GaAs	2.6
Quartz	3.0
Alumina	4.2

Specimen damage by electron beam

The loss of electron beam energy in the specimen occurs mostly in the form of heat generation at the irradiated point. Polymer materials and biological specimens, which are generally not resistant to heat, are easily damaged by the electron beam, because of their low heat conductivity.

The following strategies can be used to avoid this damage:

- decrease the electron beam intensity;
- shorten the exposure time, even though this reduces image smoothness slightly;
- image large scanning areas with low magnifications;
- control the thickness of coating metal on the specimen surface. It is also advisable to adjust beforehand the astigmatism and brightness using another field of view and then image the actual field as quickly as possible.

Summary Table of SEM Information vs SEM Parameters (10)

	Condenser Lens Strength		Accelerating Voltage		Working Distance		Objective Aperture Size	
	Stronger	**Weaker**	**Higher**	**Lower**	**Shorter**	**Longer**	**Smaller**	**Larger**
Spot Size	Smaller	Larger	Smaller	Larger	Smaller	Larger	Smaller	Larger
Sharpness	More	Less	More	Less	More	Less	More	Less
Resolution	More	Less	More	Less	More	Less	More	Less
Probe Current	Less	More	Little effect		Little effect		Less	More
SE Signal	Less	More	Little effect		Little effect		Less	More
BSE Signal	Less	More	More	Less	More	Less	Less	More
X-ray Signal	Less	More	More	Less	More	Less	Less	More
Depth of Field	Little effect		Little effect		Less	More	More	Less
Surface Info	Little effect		Less	More	Little effect		Little effect	
Depth Info	Little effect		More	Less	Little effect		Little effect	
Charging, Beam Damage, Contamination	Less	More	More	Less	Little effect		Less	More

[1] FE-SEM Training Manual, Hitachi Scientific Instruments
[2] http://www.microscopy.ethz.ch/lens.htm
[3] JEOL: A Guide to Scanning Microscope Observation
[4] Joseph Goldstein et al. "Scanning Electron Microscopy and X-Ray Microanalysis".
[5] JEOL 6700 SEM User Manual
[6] http://www.cas.muohio.edu/~**em**fweb/**EMTheory**/OH_Index.html
[7] http://www.gel.usherbrooke.ca/casino/What.html
[8] http://emalwww.engin.umich.edu/courses/semlectures/semlec.html#anchor659909
[9] David C. Joy. "Low Voltage Scanning Electron Microscopy", Hitachi Instrument News, July 1989
[10] JEOL training documents