

BRIEF INTRODUCTION TO SCANNING ELECTRON MICROSCOPY (SEM)

Summary

The quality and resolution of SEM images are function of three major parameters: (i) instrument performance, (ii) selection of imaging parameters (e.g. operator control), and (iii) nature of the specimen. All three aspects operate concurrently and neither of them should or can be ignored or overemphasized.

Image formation in the SEM

One of the most surprising aspects of scanning electron microscopy is the apparent ease with which SEM images of three-dimensional objects can be interpreted by any observer with no prior knowledge of the instrument. This is somewhat surprising in view of the unusual way in which image is formed, which seems to differ greatly from normal human experience with images formed by light and viewed by the eye.

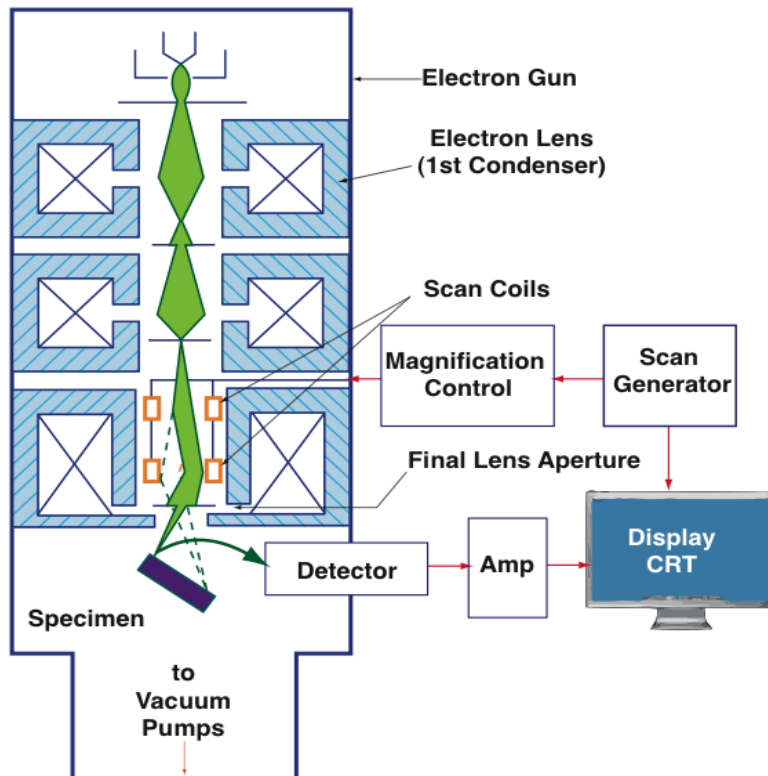


Figure 1

The main components of a typical SEM are electron column, scanning system, detector(s), display, vacuum system and electronics controls (fig. 1).

The electron column of the SEM consists of an electron gun and two or more electromagnetic lenses operating in vacuum. The electron gun generates free electrons and accelerates these electrons to energies in the range 1-40 keV in the SEM. The purpose of the electron lenses is to create a small, focused electron probe on the specimen. Most SEMs can generate an electron beam at the specimen surface with spot size less than 10 nm in diameter while still carrying sufficient current to form acceptable image. Typically the electron beam is defined by probe diameter (d) in the range of 1 nm to 1 μm , probe current (i_b) – pA to μA ; and probe convergence (α) – 10^{-4} to 10^{-2} radians.

In order to produce images the electron beam is focused into a fine probe, which is scanned across the surface of the specimen with the help of scanning coils (fig. 1). Each point on the specimen that is struck by the accelerated electrons emits signal in the form of electromagnetic radiation. Selected portions of this radiation, usually secondary (SE) and/or backscattered electrons (BSE), are collected by a detector and the resulting signal is amplified and displayed on a TV screen or computer monitor. The resulting image is generally straightforward to interpret, at least for topographic imaging of objects at low magnifications.

The electron beam interacts with the specimen to a depth approximately $1\ \mu\text{m}$. Complex interactions of the beam electrons with the atoms of the specimen produce wide variety of radiation. The need of understanding of the process of image formation for reliable interpretation of images arises in special situations and mostly in the case of high-magnification imaging. In such case knowledge of electron optics, beam-specimen interactions, detection, and visualization processes is necessary for successful utilization of the power of the SEM.

Lenses in the SEM

The purpose of the electron lenses is to produce a convergent electron beam with desired crossover diameter. The lenses are metal cylinders with cylindrical hole, which operate in vacuum. Inside the lenses magnetic field is generated, which in turn is varied to focus or defocus the electron beam passing through the hole of the lens.

SEMs employ one to three condenser lenses to de-magnify the electron-beam crossover diameter in the electron gun to a smaller size. The first and second condenser lenses control the amount of demagnification. Usually in the microscopes this is a single control labeled, “spot size”, “condenser”, “C1” or “resolution”. The final lens in the column sometimes inappropriately called “objective” focuses the probe on the sample. The design of the lenses often incorporates space for the scanning coils, the stigmator, and the beam limiting aperture. Since the high currents flowing through the lenses generate excessive heat, they are cooled usually by circulating water.

There are three basic designs of the final lens: the first is called pinhole or conical lens, where the specimen is outside the lens and its magnetic field; the second is for specimens small enough (just several mm across) to be placed inside the lens - called immersion lens, and the third one is a snorkel lens, where the specimen is outside the lens but inside its magnetic field.

Typical focal lengths for the pinhole final lens are between 5 to 40 mm. There are two main operational advantages of the pinhole lens. First, specimen size is limited by the size of the specimen chamber and not by the lens. Second the variable working distance allows control over the depth of field and size of the imaged area.

The immersion lens has a very short focal distance range of 2 to 5 mm. A small specimen is placed directly inside the lens gap. Because lens aberrations scale with focal distance, this type of lens yields the lowest aberrations, the smallest probe size, and the highest image resolution. Secondary electrons spiral upward in the strong magnetic field of the lens and are collected by the detector positioned above the lens. This lens generally is not suitable for detecting BSE.

The snorkel lens combines the best features of both the pinhole and the immersion lenses. Its magnetic field extends outside the lens and reaches the specimen. This design

puts restrictions for imaging of magnetic specimens, which could be drawn inside lens with negative consequences for the samples and the SEM. The advantage is that it has low aberrations, and the sample size is not limited by the lens gap.

The aberrations of the final lens and consequently the resolution are controlled by a final lens aperture, which affects the beam convergence angle.

The final lens aperture has three important effects on the final probe. First there is an optimum aperture angle that minimizes aberrations. Second, the current in the final probe is controlled by the size of the aperture. And third, the probe convergence angle α controls the depth of field. Smaller α produces greater depth of field. Spot size in SEM is minimized at the expense of current.

The general approach in SEM is to minimize the probe diameter and maximize the probe current. The minimum probe diameter depends on the spherical aberration of the SEM electron optics, the gun source size, the electron optical brightness and the accelerating voltage.

The probe size, which directly effects resolution can be decreased by increasing the brightness. The electron optical brightness β is a parameter that is function of the electron gun performance and design. For all types of electron guns, brightness increases linearly with accelerating voltage, so every electron source is 10 times as bright at 10 kV as it is at 1 kV. Decreasing the wavelength and the spherical aberration also decreases the probe size.

The maximum beam current depends on several parameters. We can increase the brightness by increasing the accelerating voltage, the limiting tendency here is that increasing the accelerating voltage too much increases the volume of generation of X-rays and thus limits resolution. As a consequence increasing the accelerating voltage can be done successfully up to about 30 kV, above that the resolution is effected negatively.

Decreasing the spherical aberration is another way of increasing the maximum current. This is achieved more successfully in the immersion type lenses. The above considerations for maximum probe current and minimum probe diameter ignore the contribution of chromatic aberrations. These cannot be ignored when operating at low accelerating voltages in the range of 2 kV or less. The dependence of the probe current becomes much more complex due to the chromatic aberration term.

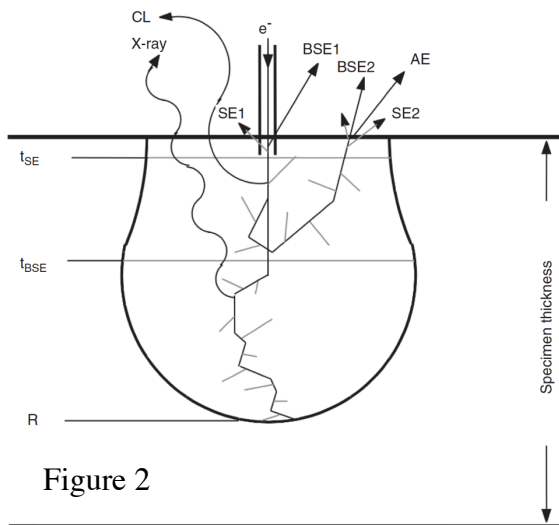


Figure 2

Interaction Volume

The concept of interaction volume of the primary beam electrons and the sampling volume of the emitted secondary radiation are important both in interpretation of SEM images and in the proper application of quantitative X-ray microanalysis. The image details and resolution in the SEM are determined not by the size of the electron probe by itself but rather *by the size and characteristics of the interaction volume.*

When the accelerated beam

electrons strike a specimen they penetrate inside it to depths of about $1\ \mu\text{m}$ and interact both elastically and inelastically with the solid, forming a limiting interaction volume from which various types of radiation emerge, including BSE, SE, characteristic and brehmsstrahlung x-rays, and cathodoluminescence in some materials.

The combined effect of elastic and inelastic scattering controls the penetration of the electron beam into the solid. *The resulting region over which the incident electrons interact with the sample is known as interaction volume.* The interaction volume has several important characteristics, which determine the nature of imaging in the SEM.

The energy deposition rate varies rapidly throughout the interaction volume, being greatest near the beam impact point. The interaction volume has a distinct shape (fig. 2). For low-atomic-number target it has distinct pear shape. For intermediate and high-atomic number materials the shape is in the form of hemi-sphere.

The interaction volume increases with increasing incident beam energy and decreases with increasing average atomic number of the specimen. For secondary electrons the sampling depth is from 10 to 100 nm and diameter equals the diameter of the area emitting backscattered electrons. BSE are emitted from much larger depths compared to SE.

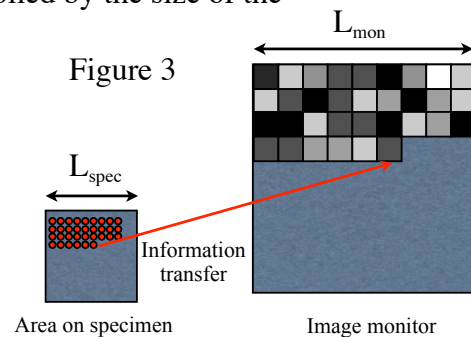
Ultimately the resolution in the SEM is controlled by the size of the interaction volume.

Image formation

The SEM image is a 2D intensity map in the analog or digital domain. Each image pixel on the display corresponds to a point on the sample, which is proportional to the signal intensity captured by the detector at each specific point (fig.3). Unlike optical or transmission electron microscopes no true image exists in the SEM. It is not possible to place a film anywhere in the SEM and record an image. It does not exist. The image is generated and displayed electronically. The images in the SEM are formed by electronic synthesis, no optical transformation takes place, and no real or virtual optical images are produced in the SEM.

In an analog scanning system, the beam is moved continuously; with a rapid scan along the X-axis (line scan) supplemented by a stepwise slow scan along the Y-axis at predefined number of lines. The time for scanning a single line multiplied by the number of lines in a frame gives the frame time. In digital scanning systems, only discrete beam locations are allowed. The beam is positioned in a particular location remains there for a fixed time, called dwell time, and then it is moved to the next point.

When the beam is focused on the specimen an analog signal intensity is measured by the detector. The voltage signal produced by the detector's amplifier is digitized and stored as discrete numerical value in the corresponding computer registry. Typically, the intensity is digitized into 8 bits (256 levels), 12 bits (4096) or 16 bits (65,536). The digital image is viewed by converting the numerical values stored in the computer memory into an analog signal for display on a monitor.



Magnification in the SEM

No optical transformation is responsible for image magnification in the SEM. Magnification is achieved by scanning an area on the specimen, which is smaller than the display. Since the monitor length is fixed, increase or decrease in magnification is achieved by respectively reducing or increasing the length of the scan on the specimen. For accurate magnification measurements a calibration is necessary.

Magnification in the SEM depends only on the excitation of the scan coils and not on the excitation of the objective lens, which determines the focus of the beam. The magnification of the SEM image is changed by adjusting the length of the scan on the specimen (L_{spec}) for a constant length of scan on the monitor (L_{mon}) (fig. 3), which gives the linear magnification of the image (M). The numerical value of magnification is determined by the ratio of the length of the monitor versus the length of the scan on the sample:

$$M = L_{\text{mon}}/L_{\text{spec}}.$$

Image Quality

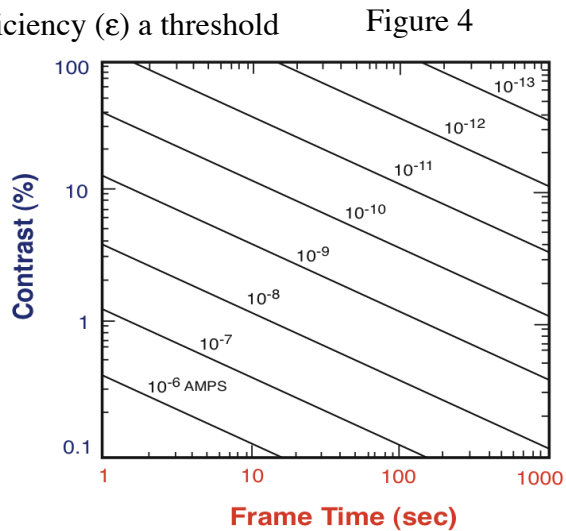
The SEM imaging process involves measurement of discrete events collected at the detector. Measuring the signal (S) consists of counting the number of electrons (n), at the detector. Since the presence of noise degrades the signal, the signal quality is expressed as the signal-to-noise ratio (S/N).

As the mean number of counts (electrons) increases, the signal quality improves because the random fluctuations become less significant fraction of the total signal. Contrast is defined as

$$C = \frac{(S_2 - S_1)}{S_2},$$

where $S_1 < S_2$ and both represent signals detected at any two arbitrary points in the scan raster. By definition contrast is always positive and varies from 0 to 1. Considering electrons as signal carriers, then in terms of beam current, frame time, and signal collection efficiency (ϵ) a threshold beam current (i_b) can be defined.

Objects that do not produce the threshold contrast cannot be distinguished from the noise of background fluctuations. A useful way to understand the relationships of the beam current, frame time and contrast level is a graphical plot (fig. 4). The plot assumes signal collection efficiency is 25%. If we want to obtain an image with 10% contrast and frame time of 100 s, a beam current in excess of 10^{-11} A must be supplied. If the sample produces contrast of only 0.5% then at 100 s per frame a current of 10^{-8} A is necessary. For a specific beam current there is always a level of contrast below which nothing will be visible. Once we know the



current required to image a specific contrast level, we can calculate the probe size that can be obtained with this current, taking into account all aberrations.

Practically in the SEM at a given accelerating voltage and electron beam parameters, the desired contrast is achieved by adjusting the frame time, which is controlled by setting the scanning speed.

Also image quality depends on the operating environment, e.g. the mechanical stability of the instrument and the level of external electromagnetic interferences that can reduce the performance of the instrument.

Signals and Imaging modes in the SEM

The primary signal carriers in SEM used to form images are secondary (SE) and backscattered electrons (BSE). SE are low energy particles with typical energy of about 5 eV. BSE are high energy particles with energies approaching the energy of the incident electron beam, e.g. several thousands eV.

Compositional contrast

BSE. Compositional contrast or atomic number contrast arises because the intensity of the signal generated from areas with different composition is proportional to the difference in the average atomic number of the respective areas. The most efficient way to image compositional contrast is by using backscattered electrons because of the nearly monotonic increase of the BSE coefficient η with atomic number (fig. 5). Regions of high average atomic number will appear bright relative to regions of low atomic number. The magnitude of the atomic-number contrast can be predicted. If we use a detector sensitive to the number of backscattered electrons, then the detected signal is proportional to the backscatter coefficient. The contrast between two regions 1 and 2 can be calculated as the difference between the backscatter coefficients (η_1 and η_2).

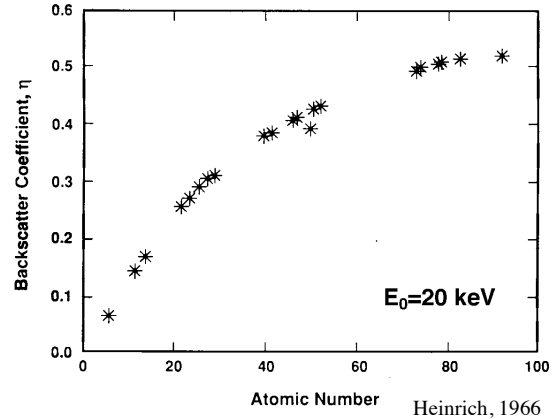
$$C = \frac{(\eta_1 - \eta_2)}{\eta_2}$$

Elements separated by one unit of atomic number produce low contrast; e.g., Al and Si yield contrast of only 6.7%. For pairs widely separated in atomic number, the contrast is much larger; e.g., Al and Au produce contrast of 69%.

Contrast above 10% is relatively easy to image in the SEM, contrast in the range 1 –10% requires careful strategy, and contrast <1% requires extreme measures to successfully image it.

Topographic contrast. Topographic contrast includes all effects by which the shape and morphology of the specimen can be imaged. Vast majority of applications of the SEM involve studying shapes. Topographic contrast is the most important imaging mechanism. Topographic contrast arises because the number and trajectories of BS electrons and the number of SE depend on the angle of incidence between the beam and the specimen's surface. The angle of incidence varies because of the local inclination of the specimen. At

Figure 5



each point the beam strikes, the number of BSE and SE detected gives direct information on the inclination of the specimen. The interpretation of the images is intuitive and no knowledge of the mechanism of image formation is necessary.

The topographic contrast actually observed depends on the detector used and its placement relative to the specimen and on the exact contribution BSE and SE detected.

Defects in SEM Imaging

Contamination

The term contamination describes the collective phenomena by which the surface of a specimen undergoes deposition of a foreign substance, generally a carbonaceous material derived from the breakdown of hydrocarbon. The effect of this surface deposit is generally observed as a “scan square” when the magnification is reduced while centered on the previous field of view. Contrast arises in such image because of the change in the SE coefficient caused by the deposition of foreign material. This can be avoided by starting imaging at low magnifications and gradually increasing it.

Contamination can affect the signal at high-resolution imaging. Hydrocarbon molecules are attracted to the beam location and form build-up, which obscures the fine detail features.

Contamination can be reduced in first place by improving the vacuum in the specimen chamber. Also anti-contamination devices can be employed, which cool area in the close vicinity of the specimen so that hydrocarbon migration is reduced. This can be achieved also by cooling directly the sample. Another solution is exposure of the specimen to an intense ultraviolet light prior to SEM imaging. The UV light can cross-link a surface contamination layer and fix it at its place, so later build-up is minimized.

Charging

When the beam interacts with the sample part of the incident electrons are emitted back, but larger fraction remains in the specimen as the beam electrons lose their initial energy and are captured by the specimen. This charge flows to ground if the specimen is conductor and a suitable connection exists to conduct away the charges. If the ground path is broken, even conducting specimen quickly accumulates charge and its surface potential rises.

Charging is more often observed whenever a specimen or portion of it is an insulator. The charges injected by the beam cannot readily flow to ground. The resulting accumulation of charge is a complex, dynamic phenomenon. The specimen is in a continually changing state of surface potential due to the accumulation and discharge of electrons.

Charging manifests itself in images in a variety of ways. When local charging alters the surface potential, the field lines of the detector potential, which exist around the sample are disrupted, and collection of SE is greatly altered. A contrast mechanism develops known as voltage contrast in which the potential distribution across the surface is imaged. Some areas appear extremely bright because they are negative relative to the E-T detector and enhance SE collection. Other areas appear black because they charge positively and suppress the collection of SE. The difficulty arises because the contrast due to surface potential becomes so large that it overwhelms the contrast from the true features of the sample.

Effects due to charging can be minimized by coating the sample with conductive film, by utilizing rapid scanning, by imaging with BSE, and/or low accelerating voltage.

Sample requirements

Since the SEM is operated under high vacuum the specimens that can be studied must be compatible with high vacuum ($\sim 10^{-5}$ mbar). This means that liquids and materials containing water and other volatile components cannot be studied directly. Also fine powder samples need to be fixed firmly to a specimen holder substrate so that they will not contaminate the SEM specimen chamber.

Non-conductive materials need to be attached to a conductive specimen holder and coated with a thin conductive film by sputtering or evaporation. Typical coating materials are Au, Pt, Pd, their alloys, as well as carbon.

There are special types of SEM instruments such as variable-pressure SEM (VP-SEM) and environmental SEM (ESEM) that can operate at higher specimen chamber pressures thus allowing for non-conductive materials (VP-SEM) or even wet specimens to be studied (ESEM).