

# SEM Imaging Checklist

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## 14.1 Specimen Considerations (High Vacuum SEM; Specimen Chamber Pressure <math> < 10^{-3}</math> Pa)

### 14.1.1 Conducting or Semiconducting Specimens

A conducting or semiconducting specimen must maintain good contact with electrical ground to dissipate the injected beam current. Without such an electrical path, even a highly conducting specimen such as a metal will show charging artifacts, in the extreme case acting as an electron mirror and reflecting the beam off the specimen. A typical strategy is to use an adhesive such as double-sided conducting tape to both grip the specimen to a support, for example, a stub or a planchet, as well as to make the necessary electrical path connection. Note that some adhesives may only be suitable for low magnification (scanned field dimensions greater than  $100 \times 100 \mu\text{m}$ , nominally less than  $1,000\times$  magnification) and intermediate magnification (scanned field dimensions between  $100 \mu\text{m} \times 100 \mu\text{m}$ , nominally less than  $1,000\times$  magnification and  $10 \mu\text{m} \times 10 \mu\text{m}$ , nominally less than  $10,000\times$  magnification) due to dimensional changes which may occur as the adhesive outgases in the SEM leading to image instability such as drift. Good practice is to adequately outgas the mounted specimen in the SEM airlock or a separate vacuum system to minimize contamination in the SEM as well as to minimize further dimensional shrinkage. Note that some adhesive media are also subject to dimensional change due to electron radiation damage during imaging, which can also lead to image drift.

### 14.1.2 Insulating Specimens

For SEM imaging above the low beam energy range ( $E_0 \leq 5 \text{ keV}$ ), insulating specimens must be coated with a suitable conducting layer to dissipate the charge injected by the beam and avoid charging artifacts. Note that after this layer is applied, a connection to electrical ground must be established for the coating to be effective. For tall specimens, the side of the specimen may not receive adequate coating to create a conducting path. A small strip of adhesive tape may be used for this purpose, running from the coating to the conducting stub. Note that for complex shapes, surfaces that do not receive the coating due to geometric shading may still accumulate charge even if not directly exposed to the beam due to re-scattering of backscattered electrons (BSEs).

To optimize imaging, the conductive coating should have a high secondary electron coefficient (e.g., Au-Pd, Cr, platinum-family metals). While thermally evaporated carbon is an effective, tough coating suitable for elemental X-ray microanalysis, the low secondary electron coefficient of carbon makes it a poor choice for imaging, especially for high resolution work involving high magnification where establishing visibility is critical.

The coating should be the thinnest possible that is effective at discharging the specimen, typically a few nanometers or less for ion-sputtered coatings. For high resolution imaging, the coating material should be chosen to have the least possible structure, for example, Au-Pd, which produces a continuous fine-grained layer, rather than pure Au, which tends to produce discontinuous islands.

Uncoated insulating specimens can be successfully imaged with minimum charging artifacts by carefully choosing the beam energy, typically in the range  $0.1 \text{ keV} - 5 \text{ keV}$  with the exact value dependent on the material, specimen topography, tilt, beam current, and scan speed to achieve a charge-neutral condition in which the charge injected by the beam is matched by the charge ejected as backscattered electrons and secondary electrons.

## 14.2 Electron Signals Available

### 14.2.1 Beam Electron Range

Beam electrons penetrate into the specimen spreading laterally through elastic scattering and losing energy through inelastic scattering creating the interaction volume (IV). The Kanaya-Okayama range equation gives the total penetration distance (for a beam incident perpendicular to the specimen surface):

$$R_{K-O}(\text{nm}) = (27.6 A) / (Z^{0.89} \rho) E_0^{1.67} \quad (14.1)$$

where  $A$  is the atomic weight (g/mol),  $Z$  is the atomic number,  $\rho$  is the density ( $\text{g}/\text{cm}^3$ ), and  $E_0$  is the incident beam energy (keV).

### 14.2.2 Backscattered Electrons

BSEs are beam electrons that escape the specimen after one or many elastic scattering events. The BSE coefficient increases with increasing atomic number of the target (compositional contrast) and with increasing tilt of a surface (topographic contrast). BSEs have a wide spectrum of kinetic energy, but over half retain a significant fraction, 50% or more, of the incident beam energy. BSE sample specimen depths as great as 0.15 (high  $Z$ ) to 0.3 (low  $Z$ ) of  $R_{K-O}$  and spread laterally by 0.2 (high  $Z$ ) to 0.5 (low  $Z$ ) of  $R_{K-O}$ . From a flat surface normal to the incident beam, BSEs follow a cosine angular distribution (angle measured relative to the surface normal), while for tilted flat surfaces, the angular distribution becomes more strongly peaked in the forward direction with increasing surface tilt.

### 14.2.3 Secondary Electrons

Secondary electrons (SEs) are specimen electrons that are ejected through beam electron - atom interactions. SE have a distribution of kinetic energy which peaks at a few electronvolts. SEs sample only a few nanometers into the specimen due to this low kinetic energy. SE emission increases strongly

with surface tilt (topographic contrast). SE emission increases as the beam energy decreases. Three classes of SEs are recognized: (1)  $SE_1$  are produced as the beam electrons enter the specimen surface within footprint of the beam, potentially carrying high resolution information, and are sensitive to the first few nm below the surface. (2)  $SE_2$  are produced as beam electrons exit as BSEs and are actually sensitive to BSE characteristics (lateral and depth sampling). (3)  $SE_3$  are produced as the BSEs strike the objective lens and specimen chamber walls, and are also sensitive to BSE characteristics (lateral and depth sampling). SEs are sensitive to electrical and magnetic fields, and even a few volts of surface potential (“charging”) can alter SE trajectories and eventual collection.

### 14.3 Selecting the Electron Detector

#### 14.3.1 Everhart–Thornley Detector (“Secondary Electron” Detector)

Virtually all SEMs are equipped with an Everhart–Thornley detector, often referred to as the “secondary electron (SE)” detector. While SEs constitute a large fraction of the E–T signal, the E–T detector is also sensitive to BSEs directly and indirectly through the collection of  $SE_2$  and  $SE_3$ . The E–T detector is the usual choice for imaging problems involving fine spatial details. The effective collection angle for SEs is nearly  $2\pi$  sr. Some E–T detectors allow user selection of the potential applied to the SE-collecting Faraday cage so that the SE signal can be minimized or eliminated leaving a BSE signal. This BSE signal is collected over a very small solid angle,  $\sim 0.01$  sr.

#### 14.3.2 Backscattered Electron Detectors

Most SEMs are also equipped with a “dedicated” backscattered electron detector which has no sensitivity to SEs. Passive scintillator BSE detectors and semiconductor BSE detectors are typically placed on the bottom of the objective lens above the specimen, giving a large solid angle of collection approaching  $2\pi$  sr. Both types have an energy threshold below which there is no response, the value of which depends on the particular detector in use and is typically in the range 1 keV to 5 keV. Above this threshold, the detector response increases nearly linearly with BSE energy, creating a modest energy selectivity.

#### 14.3.3 “Through-the-Lens” Detectors

Some high performance SEMs include “through-the-lens” (TTL) detectors which use the strong magnetic field of the objective lens to capture SEs. The collection is restricted to the  $SE_1$  and  $SE_2$  signals, with the  $SE_3$  component excluded. Since  $SE_3$  actually carry lower resolution BSE information, excluding  $SE_3$  benefits high resolution imaging. TTL BSE detectors capture the portion of the BSEs emitted into the bore of the lens. Some TTL SE and TTL BSE detectors can energy filter the signal-carrying electrons according to their energy.

### 14.4 Selecting the Beam Energy for SEM Imaging

The optimum beam energy depends on the nature of the imaging problem to be solved. The location of the feature (s) of interest on the surface or within the specimen; the contrast generating mechanism (s), and the degree of spatial resolution to be achieved are examples of factors to be considered.

#### 14.4.1 Compositional Contrast With Backscattered Electrons

Choose  $E_0 \geq 10$  keV: Above 5 keV, electron backscattering follows a nearly monotonic increase with atomic number, resulting in easily interpretable compositional contrast (aka “atomic number contrast”; “Z-contrast”). Because of the energy threshold of the passive scintillator BSE detector and semiconductor BSE detector ( $\sim 1$  keV to 5 keV), by selecting  $E_0 \geq 10$  keV the BSE detector will operate reliably with the energy spectrum of BSEs produced by the specimen. For maximum compositional contrast, a flat polished specimen should be placed at  $0^\circ$  tilt (i.e., perpendicular to the beam).

#### 14.4.2 Topographic Contrast With Backscattered Electrons

Choose  $E_0 \geq 10$  keV: BSE detectors can respond strongly to variations in specimen topography, so the same beam energy conditions apply as for compositional contrast (► Sect. 14.4.1) to assure efficient BSE detector response. Local variations in the specimen surface tilt cause BSEs to travel in different directions. BSE topographic contrast is maximized by a small BSE detector placed on one side of the beam (e.g., Everhart–Thornley detector with zero or negative Faraday cage bias) and minimized by large BSE detectors placed symmetrically around the beam (e.g., large passive scintillator or semiconductor detector).

#### 14.4.3 Topographic Contrast With Secondary Electrons

Choose any  $E_0$  within the operating range: Topographic contrast is usually viewed in “secondary electron” images prepared with the E–T detector, positively biased for SE collection. The E–T detector is designed to efficiently collect and detect SEs, which are produced at all incident beam energies and are maximized at low beam energy.

#### 14.4.4 High Resolution SEM Imaging

Two beam energy strategies optimize imaging fine-scale details by maximizing the contribution of the SE that are produced within the footprint of the focused beam:

### Strategy 1

Choose the highest available beam energy,  $E_0 \geq 25$  keV. The  $SE_1$  component of the total SE signal retains the high resolution information at the scale of the beam entrance footprint. Due to lateral spreading of the interaction volume, the BSE and their associated  $SE_2$  and  $SE_3$  signals actually degrade spatial resolution at intermediate beam energy (e.g., 5 keV to 20 keV). As the beam energy increases, the electron range increases as  $E_0^{1.67}$ , causing the lateral spreading of BSEs to increase. When these signal components are spread out as much as possible by using the maximum beam energy, their contribution diminishes toward random noise, while the high resolution  $SE_1$  contribution remains. Degraded signal-to-noise means that longer pixel dwell will be necessary to establish visibility of weak contrast. An additional advantage is the improvement in gun brightness, which increases linearly with  $E_0$ , so that more beam current can be obtained in the focused beam of a given size.

### Strategy 2

Choose low beam energy,  $E_0 \leq 2$  keV: as the beam energy is reduced, the electron range decreases as  $E_0^{1.67}$ , which collapses the BSE and associated  $SE_2$  and  $SE_3$  signals to dimensions approaching that of the footprint of the focused beam which defines the  $SE_1$  distribution. These abundant BSE,  $SE_2$  and  $SE_3$  signals thus contribute to the high resolution signal rather than degrading it. Although there is a significant penalty in gun brightness imposed by low beam energy operation, the increased abundance of the high resolution signals partially compensates for the loss in gun brightness.

## 14.5 Selecting the Beam Current

### 14.5.1 High Resolution Imaging

Imaging fine spatial details requires a small beam diameter, which requires choosing a strong first condenser lens that inevitably restricts the beam current to a low value. Beam current ( $I_B$ ), beam diameter ( $d$ ), and beam divergence ( $\alpha$ ) are related through the Brightness ( $\beta$ ) Equation:

$$\beta = 4 I_B / (\pi^2 d^2 \alpha^2) \quad (14.2)$$

*Using a small beam for high resolution inevitably restricts the beam current available. An important consequence of operating with low beam current is poor visibility of low contrast features.*

### 14.5.2 Low Contrast Features Require High Beam Current and/or Long Frame Time to Establish Visibility

Contrast ( $C_{tr}$ ),  $C_{tr} = (S_2 - S_1)/S_2$ , where  $S_2 > S_1$ , arises when the properties of a feature (e.g., composition, mass thickness, and/or surface tilt) cause a difference in the BSE ( $\eta$ ) and/or SE ( $\delta$ ) thus altering the measured signal,  $S_{feature} = S_2$ , compared to the background signal,  $S_{background} = S_1$ , from adjacent parts of the

specimen. The visibility of this contrast depends on satisfying the Threshold Current Equation:

$$I_{th} > 4 \text{ pA} / (\delta \text{ DQE } C_{tr}^2 t_F) \quad (14.3a)$$

or in terms of the contrast threshold as

$$C_{th} > \text{SQRT} [4 \text{ pA} / (I_B \delta \text{ DQE } t_F)] \quad (14.3b)$$

where  $\delta$  is the secondary electron coefficient ( $\eta$  if imaging with backscattered electrons), DQE is the detective quantum efficiency (effectively the fraction of the collected electrons—detector solid angle and detection—that contribute to the measured signal), and  $t_F$  is the frame time (s) for a 1024 by 1024-pixel image. Lower values of  $C_{th}$  can be obtained with higher beam current and/or longer frame times. *For any selection of beam current and frame time, there is always a threshold contrast below which features will not be visible.*

## 14.6 Image Presentation

### 14.6.1 “Live” Display Adjustments

After the visibility threshold has been established for a contrast level  $C_{th}$  through appropriate selection of beam current and frame time, the imaging signal must be manipulated to properly present this contrast on the final image display. An image histogram function allows monitoring of the distribution of the displayed signal. Ideally, the signal amplification parameters (e.g., “contrast” and “gain” or other designations) are adjusted so signal variations span nearly the entire gray-scale range of the digitizer (8-bit, 0–255) without reaching pure white (level 255) to avoid saturation or pure black (level 0) to avoid “bottoming”; both conditions cause loss of information.

### 14.6.2 Post-Collection Processing

Provided that the signal has been properly digitized (no saturation or bottoming), various digital image processing algorithms can be applied to the stored image to improve the displayed image, including contrast and brightness adjustment, non-linear expansion of a portion of the gray scale range, edge enhancements, and many others. ImageJ-Fiji provides a free open source platform of these software tools.

## 14.7 Image Interpretation

### 14.7.1 Observer’s Point of View

The SEM image is interpreted as if the observer is looking along the incident electron beam. Your eye is the beam!

### 14.7.2 Direction of Illumination

The apparent source of illumination is from the position of the detector. The detector is the apparent flashlight!

### 14.7.3 Contrast Encoding

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SEM image contrast is carried by number effects (different numbers of electrons leave the specimen because of local properties), trajectory effects (differences in the directions electrons travel after leaving the specimen), and energy effects (some contrast mechanisms are more sensitive to higher energy BSEs).

### 14.7.4 Imaging Topography With the Everhart–Thornley Detector

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We are strongly conditioned to expect “top lighting”; that is, the illumination of a scene comes from above (e.g., sun in the sky, lighting fixtures on the ceiling). The E–T detector (positively biased to collect SE) collects a complex mix of SEs and BSEs, which produces an image of topographic surfaces that is easily interpretable if the effective position of the E–T detector is at the top of the scanned image, achieving top lighting. This condition can be achieved by adjusting the “scan rotation” control to place the E–T detector at the top (i.e., 12 o’clock position) of the scanned image (use a simple object like a particle—ideally a sphere—on a flat surface to establish the proper value of scan rotation). Brightly illuminated features then are those that face upwards. With top lighting, most viewers will properly interpret the sense of topography. Stereomicroscopy techniques can be employed to reinforce the proper interpretation of topography.

### 14.7.5 Annular BSE Detector (Semiconductor Sum Mode A + B and Passive Scintillator)

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Because the BSE detector surrounds the electron beam symmetrically, the illumination appears to be along the viewer’s line-of-sight, much like looking along a flashlight beam. Surfaces perpendicular to the beam appear bright, tilted surfaces darker. These detectors favor number contrast mechanisms such as BSE compositional contrast (atomic number contrast).

### 14.7.6 Semiconductor BSE Detector Difference Mode, A–B

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The difference mode suppresses number effects but enhances trajectory effects such as topography.

### 14.7.7 Everhart–Thornley Detector, Negatively Biased to Reject SE

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E–T(negative bias) collects only BSE within a small solid angle, giving the effect of strong oblique illumination (similar to a scene illuminated with a shallow sun angle and viewed from above, e.g., observer in an airplane at dawn or sunset).

## 14.8 Variable Pressure Scanning Electron Microscopy (VPSEM)

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- Conventional SEM specimen chamber pressure  $< 10^{-3}$  Pa.
- VPSEM chamber pressure: 1 to 2000 pA (upper limit depends on specific VPSEM).

### 14.8.1 VPSEM Advantages

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- Electron beam–BSE–SE interactions with gas atoms create ions and free electrons that discharge insulating specimens, minimizing charging artifacts.
- Water can be maintained in equilibrium (e.g., 750 Pa and 3 °C), enabling observation of biological specimens with minimum preparation as well as water-based reactions.

### 14.8.2 VPSEM Disadvantages

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- The beam loses electrons due to gas scattering, reducing the effective useful signal generated by the electrons remaining unscattered in the focused beam while increasing noise due to scattered electron interactions. Nevertheless, nearly uncompromised high spatial resolution can be achieved. But for high resolution, compensate for loss of current in the beam by using longer frame times. For lower magnifications, compensate by using higher beam current.
- High voltage detectors such as the Everhart–Thornley secondary electron detector cannot operate due to high chamber pressure.
- VPSEM electron detectors: Gas cascade amplification detector (GSED) for SE detects SE<sub>1</sub> and SE<sub>2</sub> but avoids SE<sub>3</sub>; passive semiconductor or scintillator detectors for BSE.