Scanning Electron Microscope

The Scanning Electron Microscope (SEM) is a microscope that uses electrons rather than light to form an image. There are many advantages to using the SEM instead of a light microscope.
A Brief Introduction

Electron microscopy takes advantage of the wave nature of rapidly moving electrons. Where visible light has wavelengths from 4,000 to 7,000 Angstroms, electrons accelerated to 10,000 KeV have a wavelength of 0.12 Angstroms.

Optical microscopes have their resolution limited by the diffraction of light to about 1000 diameters magnification. Electron microscopes, so far, are limited to magnifications of around 1,000,000 diameters, primarily because of spherical and chromatic aberrations. Scanning electron microscope resolutions are currently limited to around 25 Angstroms, though, for a variety of reasons.
A Brief Introduction

The scanning electron microscope generates a beam of electrons in a vacuum. That beam is collimated by electromagnetic condenser lenses, focused by an objective lens, and scanned across the surface of the sample by electromagnetic deflection coils.

The primary imaging method is by collecting secondary electrons that are released by the sample. The secondary electrons are detected by a scintillation material that produces flashes of light from the electrons. The light flashes are then detected and amplified by a photomultiplier tube.

By correlating the sample scan position with the resulting signal, an image can be formed that is strikingly similar to what would be seen through an optical microscope. The illumination and shadowing show a quite natural looking surface topography.
A Brief Introduction

There are other imaging modes available in the SEM. Specimen current imaging using the intensity of the electrical current induced in the specimen by the illuminating electron beam to produce an image. It can often be used to show subsurface defects. Backscatter imaging uses high energy electrons that emerge nearly 180 degrees from the illuminating beam direction. The backscatter electron yield is a function of the average atomic number of each point on the sample, and thus can give compositional information.

Scanning electron microscopes are often coupled with x-ray analysers. The energetic electron beam - sample interactions generate x-rays that are characteristic of the elements present in the sample. Many other imaging modes are available that provide specialized information.
Advantages of SEM

- The SEM has a large depth of focus, which allows a large amount of the sample to be in focus at one time.
- The SEM also produces images of high resolution, which means that closely spaced features can be examined at a high magnification.
- Preparation of the samples is relatively easy since most SEMs only require the sample to be conductive.

The combination of higher magnification, larger depth of focus, greater resolution, and ease of sample observation makes the SEM one of the most heavily used instruments in research areas today.
The first, true scanning electron microscope (SEM) was developed and described in 1942 by Zworykin. The instrument described eventually consisted of an inverted column (electron gun at the bottom), three electrostatic lenses and electromagnetic scan coils placed between the second and third lenses. A photomultiplier tube detected the scintillations on a phosphor screen caused by the secondary electron emissions.

At Cambridge University in 1948, C. W. Oatley began construction of an SEM based on Zworykin's. Graduate student D. McMullan described this work in a doctoral dissertation where they had claimed a resolution of 500 Angstroms. Further work, reported by K. C. A. Smith, made large changes to the electron optics. The electrostatic lenses were replaced with electromagnetic coils, a double deflection scanning system was added as were stigmator coils.
History of SEM

The next major improvements were made to the signal collection process. The original phosphor screen-photomultiplier used by Zworykin was improved by the addition of a light pipe. The light pipe allowed for direct optical coupling between the scintillator and the photomultiplier tube, greatly improving efficiency. This was accomplished by T. E. Everhart and R. F. M. Thornley, and the detector arrangement carries their names as the Everhart-Thornley detector.

All of these improvements were combined in one instrument. Once again at Cambridge University, R. F. W. Pease and W. C. Nixon created the SEM V, utilizing the inverted column, electromagnetic lenses, double deflection scan system, stigmation coils and the Everhart-Thornley detector. This instrument became the basis for the first commercial SEM, the Cambridge Scientific Instruments Mark I, first available in 1965.
Structure of a SEM

- Electron gun
- Gun alignment control
- Pneumatic air lock valve
- Condenser lens
- Objective aperture
- Scanning coil
- Objective lens
- Motorized stage
- Sample Chamber

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Structure of a SEM

THE SCANNING ELECTRON MICROSCOPE

- Electron Gun
- Lens 1
  - Beam Current & Spot Size
- Lens 2
  - Focus
- Specimen Position & Working Distance
- Specimen
- Detector
- Frame Speed
- Magnification
- Scan Coils
- Scanning Generator
- Cathode Ray Tube
- Contrast
- Brightness
- Video Amplifier
Optical Microscope -vs- TEM -vs- SEM
The Electron Source

The electron gun in a scanning electron microscope is the source for the electron beam used to probe the sample. Electrons are emitted from a cathode, accelerated by passage through electrical fields and focused to a first optical image of the source.

The size and shape of the apparent source, beam acceleration and current are the primary determining factors in the performance and resolution of a scanning electron microscope.
The Electron Source

Semiconductor, materials, and life sciences electron beam applications require high spatial resolution, rapid data acquisition, and reliable operation. Achieving these results requires an electron source with the following ideal properties:

- Small source size
- Low electron emission energy spread
- High brightness (beam current per solid angle)
- Low short-term noise and long-term stability
- Simple and low-cost operation.
The Electron Source

The electron beam comes from a filament, made of various types of materials. The most common is the tungsten hairpin gun. Other examples of filaments are LaB$_6$ filaments and field emission guns.

- tungsten hairpin gun
- LaB$_6$ gun
- cold FE gun
- thermal (Schottky) FE gun
Thermionic Gun

Thermionic electron gun is used in most electron microscopes. It is robust, relatively cheap and does not require an ultra high vacuum.

In the thermionic electron gun, electrons are emitted from a heated filament and then accelerated toward an anode.

A divergence beam of electrons emerges from the anode hole.
Thermionic emission is the escape of electrons from a heated surface. Electrons are effectively evaporated from the material. To escape from the metal, electrons must have a component of velocity at right angles to the surface and their corresponding kinetic energy must be at least equal to the work done in passing through the surface. This minimum energy is known as the 'work function'. If the heated surface forms a cathode, then at a given temperature $T$ (° K) the maximum current density emitted is given by the Richardson/Dushman equation.
Thermionic Gun

Richardson/Dushman equation:

\[ J = A T^2 e^{-B/T} \]

where A and B are constants. The most important parameter for thermionic emission is that the work function should be as low as possible to use a cathode at an acceptable temperature.
Thermionic Gun

In a diode structure, electrons leaving the cathode surface lower the electric field at the surface. A stable condition exists when the field is zero as any further reduction would repel electrons back to the cathode. This stable regime is known as 'space-charge-limited emission' and is governed by the Child Langmuir equation.
Thermionic Gun

Child-Langmuir equation:

\[ J = PV^{\frac{3}{2}} \]

where \( P \), a constant which is a function of the geometry of the system. However, if the voltage becomes sufficiently high, the Richardson limit for current is reached when the emission becomes temperature limited.
Tungsten Hairpin Gun

A bent tungsten wire filament, with a diameter of around 100 micrometers, is spot welded to metal posts. These posts are embedded in a ceramic holder and extend out the other side to provide electrical connections.
Tungsten Hairpin Gun

In operation, the filament will be heated by passing an electrical current through it. Optimum filament temperature for the thermionic emission of electrons is around 2700 degrees Kelvin.

The anode, which is positive with respect to the filament, forms powerful attractive forces for electrons. This causes electrons to accelerate toward the anode.
The accelerating voltage, generally between -500 Volts and -50,000 Volts DC, is applied to the Wehnelt cylinder. The anode is connected to electrical ground. Resistive self-biasing is usually used where an adjustable bias resistance connects the filament to the accelerating voltage. The biasing brings the filament slightly more positive than the Wehnelt.
Tungsten Hairpin Gun

The left figure depicts the equipotential lines between the various parts of an electron gun. Electrons leaving the filament will be accelerated along the gradient towards the most positive area, the anode.

This beam of electrons will be focussed by the shape of the field gradient to a cross-over just before the anode, forming the first optical image of the source and ensuring that a larger percentage of the electrons will pass through the aperture of the anode.
Without the Wehnelt and anode, electrons emitted from the filament would tend to stay in the area of the filament. This forms a 'space charge' or a cloud of electrons whose mutual repulsion resists any further emission from the filament. The anode, being at ground potential, is more positive than the filament and attracts the electrons away from the filament - providing the primary acceleration for the electron beam.
By adding the grid, or Wehnelt, we have a way of controlling the space charge of the filament, shaping the beam and increasing the beam current.

Adjusting the bias resistance, and thereby the voltage differential between the filament and the grid, allows the beam current to be adjusted from a small de-focused beam current, through a focussed maximum current, to cut-off.

Cut-off is that point at which the more strongly negative fields of the grid prevent any electrons from reaching the anode by reversing the gradient completely around the filament.
LaB₆ Gun

LaB₆ cathodes are becoming quite common. They use a single crystal LaB₆ rod, of approximately 1 mm in diameter. This cathode can not be heated as directly as a tungsten filament, so a special mount or separate heater is used to provide the 1700 - 2100 degrees Kelvin required. The tip of the rod is polished to a point, then a small angled flat is usually polished at the point. The flat provides a defined area for emission. Without the flat, or if the cathode material evaporates past the flat, emission occurs from a broad undefined area around the point and resolution is decreased.
LaB$_6$ Gun

A LaB$_6$ electron gun uses the same basic cathode / grid / anode configuration used in a tungsten wire gun. Because of the high reactivity of the borides at the cathode temperatures used and their brittle nature, the mounting and heating of the cathode is crucial.
LaB$_6$ Gun

Two large posts compressively hold pyrolytic graphite blocks which in turn hold a piece of LaB$_6$ single crystal. The posts are made of a molybdenum-rhenium alloy which maintains a high modulus of elasticity at high temperatures. This means that the clamping force will not relax during operation.
The graphite blocks both mechanically support and resistively heat the LaB$_6$ crystal. Electron emission results from passing an electrical current through the posts and graphite blocks. Because of the inherent characteristics of pyrolytic graphite, very little heat is conducted to the supporting posts.
LaB$_6$ Gun

The standard LaB$_6$ single crystal has a 90° conical tip with a flat emitting surface of several microns in diameter at the apex. This tip shape provides the optimum performance and lifetime. Generally, a sharper cone angle and smaller flat provides higher brightness but shorter lifetimes.
Field Emission Gun

Electron paths from a field-emission source

tip size: 100 ~ 1000 Å
Field Emission Gun

In the field emission gun, a very strong electric field \((10^9 \text{ V/m})\) is used to extract electrons from a metal filament.

Temperatures are lower than that needed for a thermionic gun.

This gives a much higher source brightness than in thermionic guns, but requires a very good vacuum.
Source Brightness

In electron microscopy, the source brightness is defined as beam current density per unit solid angle.

\[
\text{Brightness } \quad B = \frac{j_c}{\pi \beta^2}
\]

\( j_c \) is the current density (in A/cm\(^2\)) and \( \beta \) is the convergence angle (in degrees). The unit of brightness are thus A/cm\(^2\)sr, where sr is a steradian.
Field Emission Gun

The application of a high voltage between a fine point cathode and a contract surface can, by a tunneling effect, give sufficient energy to an electron so that it escapes from the surface. This phenomena is known as high-field or Fowler/Nordheim emission. It should not be forgotten that the electric field around a point is greatly enhanced relative to the apparent average electric field between the electrodes.
Field Emission Gun

The current density ($A/m^2$) emitted by such a point is given by

$$J = 1.54 \times 10^{-10} \frac{E^2}{\Phi} \cdot e^{-6.83 \times 10^9 \Phi^{3/2} k / E}$$

Where $E$ is the electric field at the emitter, $\Phi$ the work function and $k$ a constant approximately equal to 1.

With fields of the order of $10^9$ V/m, current densities can attain $10^{12}$ A/m$^2$ but the actual current is quite small due to the small surface of the emitter.
Field Emission Gun

Field emission results from electrons 'tunneling' past the work function of the metal tip helped by the high electrical field gradients.
Field Emission Gun

While electrons are emitted from the surface, their apparent source is a single point beneath the surface. That is, because of the electrical fields present, electrons tend to be emitted tangential to the surface which, in a hemispherical tip, results in an apparent source at the focus of the hemisphere. This apparent source will actually not be a point because of thermally caused differences in the electron's momentum.
Field Emission Gun

It was first suggested in 1954 that a heated tungsten point, rather than a bent tungsten wire, might produce a smaller source size and higher brightness. This cathode actually incorporates both thermionic and field emissions and is referred to as a 'Schottky' or T-F (thermal - field) cathode.
Field Emission Gun

The emission process itself depends on the work function of the metal, which can be affected by absorbed gases. This is the reason a very high vacuum is required.

Refresh (flash heating with a large current) is required for a cold field emission gun when absorbed gases prevent further emission.

Sustaining high electrical field gradients is also essential to emission, so a tip that is well worn might not emit electrons at all.
Field Emission Gun

As with the tungsten filament gun, the voltage difference or bias between the first anode and the accelerating voltage on the cathode determines the emission current. The second anode is at ground potential and the voltage difference from here to the cathode determines the acceleration given to the electrons. The shape of the anodes is carefully selected to minimize aberrations.
## Comparison Among Electron Sources

<table>
<thead>
<tr>
<th></th>
<th>SCHOTTKY</th>
<th>COLD FIELD</th>
<th>LaB$_6$</th>
<th>TUNGSTEN</th>
</tr>
</thead>
<tbody>
<tr>
<td>Source Size (nm)</td>
<td>15</td>
<td>3</td>
<td>10$^4$</td>
<td>&gt;10$^4$</td>
</tr>
<tr>
<td>Energy Spread (eV)</td>
<td>0.3 - 1.0</td>
<td>0.2 - 0.3</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>Brightness (A/cm$^2$SR)</td>
<td>5 x 10$^8$</td>
<td>10$^9$</td>
<td>10$^7$</td>
<td>10$^6$</td>
</tr>
<tr>
<td>Short-Term Beam</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Current Stability</td>
<td>&lt;1</td>
<td>4 - 6</td>
<td>&lt;1</td>
<td>&lt;1</td>
</tr>
<tr>
<td>(% RMS)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Typical Service Life (hrs)</td>
<td>&gt;1 year</td>
<td>&gt;1 year</td>
<td>1000 hrs</td>
<td>100 hrs</td>
</tr>
</tbody>
</table>

- **expensive**
- **cheap**

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Advantages of Schottky Emitters

The Schottky emitter combines the high brightness and low energy spread of the cold field emitter with the high stability and low beam noise of thermal emitters. As its emitting area is approximately 100 times larger than that of the cold field emitter, it can deliver the much higher probe currents necessary for analytical applications. Furthermore, it achieves a similar low energy spread as the CFE source, but at an emission current level more than 50 times higher.

Because of their many advantages, Schottky emitters are increasingly used for high precision imaging and measurement.
When the electron beam strikes the sample, both photon and electron signals are emitted.

Incident Beam

X-rays
Through Thickness
Composition Information

Primary Backscattered Electrons
Atomic Number and Topographical Information

Cathodoluminescence
Electrical Information

Auger Electrons
Surface Sensitive
Compositional Information

Secondary Electrons
Topographical Information

Sample

Specimen Current
Electrical Information

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Electron-Specimen Interaction

At 20 KV Accelerating Voltage and Z=28
Scattering of Electrons

Inelastic Scattering

During inelastic scattering, energy is transferred to the electrons surrounding the atoms and the kinetic energy of the energetic electron involved decreases. A single inelastic event can transfer a various amount of energy from the beam electron ranging from a fraction to many kiloelectron volts. The main processes include phonon excitation, plasmon excitation, secondary electron excitation, continuum X-ray generation, and ionization of inner shells. In all processes of inelastic scattering, energy is lost, though different processes lose energy at varying rates.
Scattering of Electrons

Elastic Scattering

As the name implies, elastic scattering results in little (<1eV) or no change in energy of the scattered electron, although there is a change in momentum. Since momentum, \( p=mv \), and \( m \) do not change, the direction of the velocity vector must change. The angle of scattering can range from 0-180 degrees, with a typical value being about 5 degrees.
Secondary Electrons: Origin

Secondary electrons are specimen electrons that obtain energy by inelastic collisions with beam electrons. They are defined as electrons emitted from the specimen with energy less than 50 ev.

Secondary electrons are predominantly produced by the interactions between energetic beam electrons and weakly bonded conduction-band electrons in metals or the valence electrons of insulators and semiconductors.

This is an image of the broken surface of a piece of metal, formed using secondary electron imaging.
Secondary Electrons: Origin

There is a great difference between the amount of energy contained by beam electrons compared to the specimen electrons and because of this, only a small amount of kinetic energy can be transferred to the secondary electrons.

After undergoing additional scattering events while traveling through the specimen, some of these ejected electrons emerge from the surface of the specimen.
Arbitrarily, such emergent electrons with energies less than 50 eV are called secondary electrons; 90% of secondary electrons have energies less than 10 eV; most, from 2 to 5 eV.

The fraction of secondary electrons produced $\delta = N_{se}/N_{beam}$ is relatively independent of the atomic number of the scattering atoms (unlike the situation for backscattered electrons).

For carbon, $\delta \approx 0.5$; for Au, $\delta \approx 2.0$; for most other elements, $\delta \approx 0.1$, although some compounds show anomalous values.
SE: Escape Depth

• Because of their low energies, secondary electrons generated more than a certain distance, called the 'maximum escape depth' \( d \) below the surface cannot escape from the specimen.

• Most secondary electrons that do escape are produced within a distance of 2-5 nm of the surface.

\[
\text{metals, } d \approx 5 \text{ nm} \\
\text{insulators, } d \approx 50 \text{ nm}
\]
**SE: Contaminations**

**SE(I)** are produced by interactions of electrons from the incident beam with specimen atoms.

**SE(II)** are produced by interactions of high energy BSE with specimen atoms.

**SE(III)** are produced by high energy BSE which strike pole pieces and other solid objects near the specimen.
SE: Sample Tilt

Because incident beam electrons travel greater distances in the region close to the surface of the specimen in areas where the surface is tilted relative to the incident beam, more SE are generated within the escape depth in these areas than in areas which are normal to the beam.

Thus, the value of $\delta$ increases strongly with surface tilt. In addition, SE can escape from both sides of ridges and edges.

These effects cause tilted surfaces to appear brighter than flat surfaces, and edges and ridges to be markedly highlighted, in images formed with secondary electrons.
Secondary Electron Detection

An electron detector is used with the SEM to convert the radiation of interest into an electrical signal for manipulation and display by signal processing electronics, which to you and me is much like a television. Most SEM's are equipped with an Everhart-Thornley (E-T) detector.
SE Detector: E-T Detector

The detector used for SE usually consists of a photomultiplier tube in combination with a light guide that is coated with a phosphor. The phosphor is coated with a thin layer of Al and a potential of about 10 kV is applied to it to attract the SE and give them enough energy to activate the phosphor.

The +10 kV that is applied to the detector is sufficient to attract SEs from virtually any point where they may be formed, and so images formed with SE generally do not show deep dark areas of the kind found in some BSE images.
Characteristically, SE images appear as though the surface is being viewed from above under diffuse illumination, with the added effects of edge enhancement.
SE Detector: Contaminations

Ideally, images formed with secondary electrons should not show significant compositional contrast, because the emission of SEs does not show a strong dependence on atomic number.

However, the +10 kV collection voltage that is applied to the phosphor of the common SE detector collects all SE produced in the general neighborhood of the sample, including not only the SE(I) electrons, but also the SE(II) and SE(III) types.

The SE(II) and SE(III) type SEs are produced by BSE. The number of BSE produced is strongly dependent on Z. Therefore, the SE(II) and SE(III) electrons will reflect this dependence and bring some atomic number contrast into secondary electron images.
Backscattered Electrons: Origin

Elastic scattering occurs between the negative electron and the positive nucleus. This is essentially Rutherford scattering. Sometimes the angle is such that the electron comes back out of the sample. These are backscattered electrons.

Produced by elastic interactions of beam electrons with nuclei of atoms in the specimen.

- energy loss less than 1 eV, $E_e \approx E_0$
- scattering angles range up to 180°, but average about 5°

Many incident electrons undergo a series of such elastic event that cause them to be scattered back out of the specimen.
Backscattered Electrons: Yield

The fraction of beam electrons backscattered in this way varies strongly with the atomic number $Z$ of the scattering atoms, but does not change much with changes in $E_0$.

$$\eta = \frac{N_{\text{BSE}}}{N_{\text{beam}}}$$
BSE: Atomic Number Contrast

Because of this dependence of $\eta$ on atomic number, images produced using BSE show characteristic atomic number contrast. That is, features of high average $Z$ appear brighter than those of low average $Z$.

The left is an image of an aluminum copper alloy formed using backscattered electron imaging. The light area is mostly aluminum and the dark area is mostly copper.
BSE: Escape Depth

Because the backscattered electrons are usually produced by multiple scattering events, they travel considerable distances within the specimen during the backscattering process.

As a rough approximation, BSEs can be considered to be produced from a hemispherical region whose radius is

\[ R_{BSE} = \frac{0.007 AE_0^{1.67}}{Z^{0.9} \rho} \text{ in } \mu\text{m}. \]

- \( A \) = the atomic weight in gm/mol
- \( Z \) = the atomic number
- \( E_0 \) = incident beam energy in keV
- \( \rho \) = density in gm/cm³
BSE: Escape Depth

Some rough values of $R_{\text{BSE}}$ are:

<table>
<thead>
<tr>
<th>Spec.</th>
<th>5 keV</th>
<th>10 keV</th>
<th>20 keV</th>
<th>30 keV</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>0.08</td>
<td>0.25</td>
<td>0.80</td>
<td>1.6</td>
</tr>
<tr>
<td>Cu</td>
<td>0.03</td>
<td>0.08</td>
<td>0.25</td>
<td>0.5</td>
</tr>
<tr>
<td>Au</td>
<td>0.02</td>
<td>0.04</td>
<td>0.13</td>
<td>0.3</td>
</tr>
</tbody>
</table>

This effect limits the resolution in images produced with BSE to a value that is of the order of $2R_{\text{BSE}}$, regardless of how small the actual diameter of the incident electron beam may be.

It also means that the intensity of the BSE signal will be influenced to some extent by subsurface inhomogeneities in specimen composition occurring within the hemispherical region of BSE generation.
For values of tilt angle $\tau$ up to 70°, the depth of penetration can be calculated approximately using:

$$R_{BSE}(\tau) = R_{BSE} \cos \tau$$

When the surface is normal to the incident beam (i.e. $\Psi = 0$) the BSE are distributed symmetrically around the incident beam in a manner described approximately by the cosine function:

$$N(\Psi) = N_n \cos \Psi$$
BSE: Sample Tilt

For small tilt angles the distribution of BSE remains roughly symmetrical around the surface normal.

For large angles of surface tilt the distribution of BSE becomes asymmetric with respect to the surface normal, developing a distinct lobe caused by a predominance of forward scattering.

These effects cause the contrast in images formed with BSE to show a strong dependence on surface topography, and on the location and characteristics of the BSE detector used.
BSE Detectors

A backscattered electron detector is used with the SEM to convert backscattered electron signals into an electrical signal for manipulation and display by signal processing electronics.

On the far left of the backscatter detector is the lens, in the center is the secondary detector. To collect electrons, the BSE detector moves under the lens so the electron beam can travel through the hole in its center.
BSE Detectors: Scintillator-Light Guide-PMT Detector

BSEs strike the phosphor, producing photons which are conducted to the PMT by the light guide.

Small signal acceptance angle, so signal is inherently small.
BSE Detectors: Scintillator-Light Guide-PMT Detector

- Response time is fast enough for display at TV scan rates.
- Images show starkly strong topographic contrast, with only moderate compositional contrast - changes with specimen tilt.
BSE Detectors: Robinson Detector

- Very large acceptance angle gives high signal levels, which are enhanced by the amplification of the PMT.
- Response fast enough for display at TV scan rates.
- Very effective in producing compositional contrast.
- Topographical contrast is modest, can be varied by tilting specimen.
BSE Detectors: Channel Plate Detectors

- Capillary holes are coated with a material that emits several electrons for each incident electron. Applied kV produces cascade effect.
- Particularly well suited for producing BSE images when working at low electron accelerating voltages.
- Image contrast characteristics depend on detector size & location.
BSE Detectors: Solid State Diode Detector

BSE with energies above about 3 keV produce electron-hole pairs in a junction diode. These charge carriers are collected by an applied voltage and amplified to produce image signal.

Thin wafer, usually placed just below the lens pole piece so that specimen can be brought close to the detector, giving very large acceptance angle and area.

Detector response is too slow to give good images at fast scan rates.
BSE Detectors: Solid State Diode Detector

Detectors are usually segmented to allow contrast enhancement by signal mixing.

Special amplifiers are used to mix signals from different segments.
A beam of electrons is generated in the electron gun, located at the top of the column, which is pictured to the left. This beam is attracted through the anode, condensed by a condenser lens, and focused as a very fine point on the sample by the objective lens. The scan coils are energized (by varying the voltage produced by the scan generator) and create a magnetic field which deflects the beam back and forth in a controlled pattern. The varying voltage is also applied to the coils around the neck of the Cathode-ray tube (CRT) which produces a pattern of light deflected back and forth on the surface of the CRT. The pattern of deflection of the electron beam is the same as the pattern of deflection of the spot of light on the CRT.
The electron beam hits the sample, producing secondary electrons from the sample. These electrons are collected by a secondary detector or a backscatter detector, converted to a voltage, and amplified. The amplified voltage is applied to the grid of the CRT and causes the intensity of the spot of light to change. The image consists of thousands of spots of varying intensity on the face of a CRT that correspond to the topography of the sample.
Increasing the current in Lens1 decreases its focal length. This decreases $Y_1$, decreases $S_1$ and increases $X_2$.

Both of these changes act to decrease the diameter of the spot at the specimen $S_2$. 
It is also possible to decrease $S_2$ by decreasing $W$, the working distance between the specimen and Lens 2.

Thus, when working at high magnifications to obtain high resolution, a small value of $S_2$ is needed; operationally this is achieved by:

- using a high current in Lens 1
- using a small working distance
Increasing the current in Lens 1 decreases $Y_1$. This increases $X_2$ and increases $\beta$, both of which strongly decreases $i_2$.

Obviously, increasing the size of the aperture in Lens 2 will allow a larger fraction of the beam from Lens 1 to pass through to specimen, increasing $i_2$. 

$$i_2 = \frac{a}{X_2 \beta}$$
Electron Optics

There are a number of differences between light and electron optics. As we've seen, light optics relies on the refraction (or reflection) of light. In electron optical systems, we use electrostatic or magnetic fields to influence the trajectories of beams of electrons. There is no definite interface in the refracting medium of fields as there is in the solid lenses of light optics. Additionally, electrons in the influence of a magnetic field will also rotate around the optical axis, an effect not seen in light optics.
Electron Optics

Moving electrons in a vacuum will travel in straight paths as does light, but where there are a number of electrons moving together, their electrical mutual repulsion will cause the beam to diverge. In practical instruments used today, electrostatic lenses are used only in the electron gun, while magnetic lenses are used through the rest of an instrument.
Electron Optics

- Electrostatic lenses require conducting surfaces very close to the path of an electron beam in order to produce an electrical field of high intensity. These surfaces must be accurately formed, extremely smooth and are easily contaminated.

- Magnetic fields, on the other hand, are usually formed by solenoid coils that are located completely outside of the vacuum system. Therefore, they suffer none of the contamination problems inherent in electrostatic lenses.

- Electrostatic lenses, however, can be made extremely small and are capable of producing much faster response for beam deflections.
Electrostatic Lens

Electrical fields can also be used for focusing a beam of charged particles. In the figure below, a series of three cylinders with their axis coincident with the beam axis, demonstrate the principal of electrostatic focusing (please note that the cylindrical shape of the electrodes is merely representative - they could be the inner surfaces of apertures in disc shaped electrodes).
Electrostatic Lens

Electrons entering into the system from the left will be affected by the electrical field formed by the large voltage difference between the first two cylinders. The electrons will be given both an impulse towards the cylinder axis and a boost in velocity by the increasingly negative field. As the electrons move into the second cylinder, they receive an impulse towards the walls of the cylinder, but since they are now closer to the axis and traveling faster, the change in direction will be less than from the first impulse.
Electrostatic Lens

Entering the second field, the electrons will once again be given an impulse towards the cylinder walls. As they pass through the field, though, they will be de-accelerated by the increasingly positive field. Finally, they will again receive an impulse towards the cylinder axis. When exiting the system, the electron beam will again have it's original velocity, and there will be a net impulse towards the cylinder axis, resulting in a focusing of the beam. If the center cylinder were at a positive voltage in relation to the other two, it would create a diverging lens system.
Magnetic fields can also be used as lenses for beams of charged particles. In an SEM, electro-magnetic lens are used almost exclusively for the condenser and objective lenses. Electro-magnetic lenses used in commercial SEMs have always been solenoid coils.
Magnetic Lens

A solenoid is a cylindrical coil with circumferentially wound wire. In SEMs, the lens is completely encased in a ferromagnetic shroud forming a thick walled cylinder, with a small gap around the inner circumference. The gap confines the external magnetic field to a small area and may be located anywhere on the inner surface.
Magnetic Lens

The strength of the magnetic field generated is proportional to the number of turns of wire multiplied by the current passing through the coil ($N \times I$ where $N$ = number of wire turns, $I$ = current). Relatively large fields are required for focusing electron beams of the accelerating voltages found in SEMs (up to 50KV).
Magnetic Lens

This figure represents a number of features of a magnetic lens. Magnetic field lines are shown, as are equipotential lines. These give a visible description of the shape of the magnetic field. The light blue lines represent two possible electron paths.
Magnetic Lens

The inset gives an approximation of the magnetic field strength along the optical axis and perpendicular to the optical axis. What can not be faithfully represented in a two dimensional image is the rotation of charged particles about the axis in a uniform magnetic field.
Forces in a Cylindrical Magnetic Lens

Electron Beam

θ is a direction in the plane, \( V_L \) is \( \perp \) to the plane.

Electrons spiraling down the axis

Nonaxial electrons will experience a force both down the axis and one radial to it. Only electrons traveling down the axis feel equal radial forces from all sides of the lens. The unequal force felt by the off-axis electrons causes spiralling about the optic axis.

Two components to the \( B \) field:

- \( B_L \) = longitudinal component (down the axis)
- \( B_R \) = radial component (perpendicular to axis)
Magnetic Lens

In a uniform magnetic flux density \( B \), a moving charged particle will follow a curved path. For a point charge, like an electron, the force imparted on it by magnetic fields is found in the vector equation \( \mathbf{F} = q \mathbf{v} \times \mathbf{B} \). As an electron comes down the optical path, it first encounters the horizontal component (\( H_r \)) of the magnetic field. This causes the electron to begin a rotation about the axis.
Magnetic Lens

The strengthening of the vertical field component ($H_Z$) gives the electron an impulse towards the axis. Finally, as the electron emerges through the bottom horizontal field component, it receives an impulse that stops its rotation about the axis. Note that unlike the case of electrostatic lenses, no change in velocity takes place along the optical axis.
Magnetic Lens

- Magnetic lens
  - Electron source
  - Soft iron pole pieces
  - Copper coils
  - Image is inverted and rotated

- Optical lens
  - Light source
  - Image is inverted
Objective lens
The beam in the SEM is moved over the specimen in a raster pattern in synchronization with the beam in the CRT. Thus, for each point on the specimen there is a conjugate point on the CRT.

Image Formation

![Diagram of Image Formation](image)

- X: Specimen
- D: Detector
- Amp: Amplifier
- Cathode ray tube
- C: CRT
Image Formation

The video amplifier modulates the intensity of the beam in the CRT so that the intensity of the spot on the CRT is proportional to the signal detected from its conjugate point on the specimen.

It is interesting that no lens is directly involved in the image forming process.
Topographical Contrast
Magnification Relationship

The magnification is then simply the ratio of the length of the scan $C$ on the CRT to the length of the scan $x$ on the specimen.

For a CRT screen that is 10 cm square,

$$M = \frac{C}{x} = \frac{10cm}{x}$$

Changing magnification does not involve changing any lens current, only changing the current in the scan coils, and so:

- focus does not change as mag is changed
- the image does not rotate with mag change

<table>
<thead>
<tr>
<th>Mag</th>
<th>x</th>
</tr>
</thead>
<tbody>
<tr>
<td>10X</td>
<td>1 cm</td>
</tr>
<tr>
<td>100X</td>
<td>1 mm</td>
</tr>
<tr>
<td>1000X</td>
<td>0.1 mm</td>
</tr>
<tr>
<td>10 kX</td>
<td>10 µm</td>
</tr>
<tr>
<td>20 kX</td>
<td>5 µm</td>
</tr>
<tr>
<td>50 kX</td>
<td>2 µm</td>
</tr>
</tbody>
</table>
The Pixel

The unaided human cannot reliably resolve features smaller than about 0.1 mm (100 µm), and so the diameter of the beam in the CRT need not be made smaller than this. Thus, the diameter of an image point on the CRT is \( D = 100 \mu m \).

The conjugate point on the specimen from which the image signal is produced, which is called the 'pixel' \( P \), will have a smaller diameter, depending on the magnification, of:

\[
P = \frac{D}{Mag} = \frac{100 \mu m}{Mag}
\]

<table>
<thead>
<tr>
<th>Mag</th>
<th>( P )</th>
</tr>
</thead>
<tbody>
<tr>
<td>10X</td>
<td>10 µm</td>
</tr>
<tr>
<td>100X</td>
<td>1 µm</td>
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<tr>
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<td>0.1 µm</td>
</tr>
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<td>10 kX</td>
<td>10 nm</td>
</tr>
<tr>
<td>20 kX</td>
<td>5 nm</td>
</tr>
<tr>
<td>50 kX</td>
<td>2 nm</td>
</tr>
</tbody>
</table>
Focus

For the image to be in sharp focus it is clear that the area sampled by the incident electron beam must be at least as small as the pixel diameter for the magnification being used.

Because the beam electrons scatter around as they enter the specimen the signal electrons are actually generated from an area that is somewhat larger (perhaps 1.5 to 2 times) the actual beam diameter.

Thus, to obtain sharp images the beam must be focused to a diameter that is of the order of 0.5 to 0.7 of the diameter of the pixel for the desired magnification.
Useful Magnification

With electron microscopes it is so easy to increase the magnification (you simply press a control key), that inexperienced microscopists tend to work at much higher magnifications than they should.

Each specimen will have certain smallest relevant features, of size $F$, which can be seen clearly when the magnification is sufficient to make them visible to the unaided eye. The use of higher magnifications will not increase the detail that can be seen, but will markedly decrease the area observed. Thus, useful magnification is as following,

$$M_u = \frac{100 \mu m}{F}$$
Useful Magnification

Also, it is important to recognize that for a particular set of instrument operating conditions there is a limit to the magnification at which sharp images can be obtained.

This occurs when the pixel diameter becomes significantly less than the area from which the signal is produced (perhaps 1.5 to 2 times the beam diameter).

If, for example, an SEM is being operated under conditions that give a focused beam diameter of 50 nm, signal electrons will probably be produced from a region at least 75 nm in diameter, and

\[
M_u = \frac{100 \, \mu m}{0.075 \, \mu m} \approx 1300 \, X
\]

At much higher magnifications the signal electrons will be generated from a region that overlaps adjacent pixels and the image will appear blurred.
Image Contrast

Contrast C involves the signals produced by the detector for two points A & B on the sample, which depend on:

1. number of signal electrons emitted from the sample
2. how readily these electrons reach the detector
3. the efficiency of the detector in recording the arriving $e^-$

$$C = \frac{S_A - S_B}{S_A} = \frac{\Delta S}{S_A}$$

Signal display in the CRT or line scan mode
Image Contrast

Subsequent amplification and processing of the signal produced by the detector can change the appearance of the image, but cannot alter its information content.
**Image Quality**

- The quality of the image produced in an SEM depends basically on the signal-to-noise ratio $S/N$.
- Noise involves the statistically random fluctuations in signal that are inherent in the processes that produce the signal electrons, plus similar fluctuations introduced by amplification, and is analogous in character and effect to 'snow' in a weak TV image.
- Signal generation is a random process that follows Poisson statistics, for which the standard deviation equals the square root of the mean, i.e.

  $$\sigma = \sqrt{n} = \text{noise} \quad \text{while} \quad \text{signal} = S = \bar{n}$$

  and so

  $$\frac{S}{N} = \frac{\bar{n}}{\sqrt{n}} = \sqrt{n}$$
Image Quality

High quality images require a high S/N ratio. Operationally this can be achieved in two ways:

(1) by using a high beam current, and

(2) by using a slow scan rate so that the beam spends a longer time on each pixel
Detectable Contrast

For two small objects to be detected against a background of random noise, studies show that their signal difference must be at least five times the noise level, i.e.

\[ \Delta S = S_A - S_B \geq 5\sqrt{n} \]

The minimum contrast required then is

\[ C_{\text{min}} = \frac{\Delta S}{S_A} \geq \frac{5\sqrt{n}}{n} = \frac{5}{\sqrt{n}} \]
Detectable Contrast

The signal current is

\[ I_s = \overline{n} e / \tau \]

\( \tau \) is the time the beam spends on each pixel, given by the frame time \( f \) (sec) divided by the number of pixels per frame \( N_p \approx 10^6 \).

\[ \tau = \frac{f}{N_p} \]

Assuming, on the average, each beam electron produces about 0.25 signal electrons, gives

\[ I_s \approx 0.25 I_{beam} \geq \frac{25e}{\pi C^2} \]

\[ I_{beam} > \frac{1.6 \times 10^{-11}}{C^2 f} \text{ Ampere} \]
The Implications

Since the actual value of the beam current is seldom known, the numerical values here are not as important as the basic concept underlying this equation; namely,

(1) In order to detect objects of small size and low contrast in an SEM it is necessary to use a high beam current and a slow scan speed.

(2) The fact that certain expected features do not show up in an SEM image does not necessarily mean the features are not there (particularly when working at high magnifications where beam currents must be very low to give the small beam diameter needed for good resolution). It may simply be that under the operating conditions being used they cannot be detected - it may be somewhat like trying to photograph a snowball against a white sheet in the moonlight.
Working Distance

The decrease in demagnification is obtained when the lens current is decreased, which in turn increases the focal length $f$ of the lens. The resolution of the specimen is decreased with an increased working distance, because the spot size is increased. Conversely, the depth of field is increased with an increased working distance, because the divergence angle is smaller.
Depth of Focus

One of the great advantages of SEM images is the unusually great depth of focus they exhibit. This makes it possible to examine surfaces much rougher, and at much higher magnifications, than is possible with light microscopes. The reason for this great depth of focus arises from the geometry of the beam optics.
Depth of Focus

The final lens of the SEM focuses the electron beam to a 'crossover' at the plane of best focus.

The beam diameter increases as the beam converges and diverges above and below this plane.
Depth of Focus

At some distance D/2 above and below the focus plane the diameter of the beam becomes twice the pixel diameter for the mag being used, whereupon the signals from adjacent pixels overlap enough to cause the image to appear blurred.

Over the distance D between these limits, however, the image will appear to be in acceptably sharp focus, and so this distance is called 'the depth of field' or 'the depth of focus'.
Depth of Focus

The factors determining the depth of field can be derived as follows,

\[ \tan \alpha = \frac{A/2}{W} = \frac{P}{(D/2)} \]

noting that for small angles in radians, \( \tan \alpha = \alpha \)

and \( P = 100 \ \mu m / Mag \)

then \( \alpha = \frac{(100 \ \mu m / Mag)}{(D/2)} \)
Depth of Focus

Solving for D, and adding conversion factors to follow the common practice of expressing the aperture diameter A, the pixel diameter P, and the depth of field D in µm while the working distance between the lens and the specimen W is expressed in mm, gives:

\[
\text{depth of focus} \approx D = \frac{4 \times 10^5 W}{A \times \text{Mag}} \mu m
\]
Depth of Focus

This is a very useful equation, because it shows the operational variables that determine the depth of field. Namely, to increase the depth of field you must either:

- decrease the size of the aperture in the final lens $A$
- decrease the magnification $M$ being used, or
- increase the distance $W$ between the specimen and the lens

$$D = \frac{4 \times 10^5 W}{A \times Mag} \ \mu m$$
Depth of Focus

Increasing the working distance reduces the divergence angle of the beam. This increases the distance over which the beam diameter stays smaller than the pixel size.

Decreasing the size of the final lens aperture has the same effect.

\[ \alpha = \left(\frac{100 \text{ } \mu m}{\text{Mag}}\right) / (D / 2) \]
Depth of Focus

The following table gives calculated values of $D$ for several different magnifications and common aperture sizes, assuming a working distance of $W = 10$ mm.

These calculations show quite clearly why the SEM is much preferred over the light microscope for studying fracture surfaces, insects, pollen grains, mineral particles, and similar rough and uneven objects.

<table>
<thead>
<tr>
<th>Mag</th>
<th>$A = 100 \mu m$</th>
<th>$200 \mu m$</th>
<th>$400 \mu m$</th>
<th>Light Micros.</th>
</tr>
</thead>
<tbody>
<tr>
<td>50X</td>
<td>800</td>
<td>400</td>
<td>200</td>
<td>100</td>
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<td>0.6</td>
</tr>
<tr>
<td>5000X</td>
<td>8</td>
<td>4</td>
<td>2</td>
<td></td>
</tr>
<tr>
<td>10,000X</td>
<td>4</td>
<td>2</td>
<td>1</td>
<td></td>
</tr>
</tbody>
</table>

$\alpha < 1^\circ$
Accelerating Voltage

When theoretically considering the electron probe diameter alone, the higher the accelerating voltage, the smaller is the electron probe. However, there are some unnegligible demerits in increasing the accelerating voltage. They are mainly as follows:

1. Lack of detailed structures of specimen surfaces.
2. Remarkable edge effect.
3. Higher possibility of charge-up.
4. Higher possibility of specimen damage.
Accelerating Voltage

In SEM, finer surface structure images can generally be obtained with lower accelerating voltages. At higher accelerating voltages, the beam penetration and diffusion area become larger, resulting in unnecessary signals (e.g., backscattered electrons) being generated from within the specimen. And these signals reduce the image contrast and veils fine surface structures. It is especially desirable to use low accelerating voltage for observation of low-concentration substances.
Accelerating Voltage

Incident electrons

[Low acceleration voltage]

[High acceleration voltage]

[Low atomic number] [High atomic number]
Accelerating Voltage

High resolution

Unclear surface structures
More edge effect
More charge-up
More damage

Clear surface structures
Less damage
Less charge-up
Less edge effect

Low resolution

Accelerating Voltage
Accelerating Voltage: Comparison

Specimen: Toner

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

Specimen: Evaporated Au particles

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

(a) 5 kV  x 36,000
(b) 25 kV  x 36,000
Accelerating Voltage: Comparison

Specimen: Filter paper

At 5 kV the microstructures of the specimen surface are clearly seen as the penetration and diffusion area of incident electrons is shallow.

(a) 5 kV  x 1,400  (b) 25 kV  x 1,400
Accelerating Voltage: Comparison

Specimen: Sintered powder

At low accelerating voltage, while surface microstructures can be observed, it is difficult to obtain sharp micrographs at high magnifications. In such a case, clear images can be obtained by shortening the WD or reducing the electron probe diameter.
Accelerating Voltage: Comparison

Specimen: Paint coat

When a high accelerating voltage is used, more scattered electrons are produced from the constituent substances within the specimen. This not only eliminates the contrast of surface microstructures, but produces a different contrast due to backscattered electrons from the substances within the specimen.

(a) 5 kV  
(b) 25 kV × 2,500
Probe Current And Probe Diameter

In the SEM, the smaller the electron probe diameter on the specimen, the higher the magnification and resolution. However, the image smoothness, namely, the S/N ratio depends on the probe current. The probe current and the probe diameter are in the relation shown below.
Probe Current And Probe Diameter

As the probe diameter is reduced, the probe current is reduced. It is therefore necessary to select a probe current suited for the magnification and observation conditions (accelerating voltage, specimen tilt, etc.) and the specimen.
Probe Current And Probe Diameter

The smaller the probe current, the sharper the image, but the surface smoothness is lost.

(a) 1 nA
(b) 0.1 nA
(c) 10 pA
Edge Effect

Among the contrast factors for secondary electrons, the tilt effect and edge effect are both due to the specimen surface morphology. Secondary electron emission from the specimen surface depends largely on the probe's incident angle on the specimen surface, and the higher the angle, the larger emission is caused.
The degree of the edge effect depends on the accelerating voltage. Namely, the lower the accelerating voltage, the smaller the penetration depth of incident electrons into the specimen. This reduces bright edge portions, thus resulting in the microstructures present in them being seen more clearly.

(a) 5 kV  x720  Tilt angle: 50°  (b) 25 kV  x720  Tilt angle: 50°
Edge Effect

Normally, secondary electron images contain some backscattered electron signals. Therefore, if the tilt direction of the specimen surface and the position of the secondary electron detector are geometrically in agreement with each other, more backscattered electrons from the tilted portions are mixed, causing them to be seen more brightly due to synergism.
Use of Sample Tilt

Specimen tilt is aimed at:

1. Improving the quality of secondary electron images.
2. Obtaining information different from that obtained when the specimen is not tilted, that is, observing topographic features and observing specimen sides.
3. Obtaining stereo micrographs.
Use of Sample Tilt

The sides of patterns are viewed by tilting the specimen. The amount of signals is increased. The above photos were taken at a tilt angle of 0° (a) and a photo taken at 45° (b). Their comparison shows that the latter is of smoother quality and steroscopic as compared with the former.

(a) Tilt angle: 0°

(b) Tilt angle: 45°
Use of Sample Tilt

When the specimen is tilted, however, lengths observed are different from their actual values. When measuring pattern widths, etc., it is necessary to measure without specimen tilting or to correct values obtained from a tilted state.

(a) Tilt angle: 0°  
(b) Tilt angle: 45°
Stereo Images

With SEM images it is sometimes difficult to correctly judge their topographical features. In such cases, observation of stereo SEM images makes it easier to understand the structure of the specimen. What is more, stereo observation allows unexpected information to be obtained even from specimens of simple structure.
In stereo observation, after a field of interest is photographed, the same field is photographed again with the specimen tilted to 5° to 15°. Viewing these two photos using stereo glasses with the tilting axis held vertically, provides a stereo image.

Specimen: back sides of oleaster leaves.
More information is obtained from stereo-photos
Detector Position

The amount of secondary electrons produced when the specimen is illuminated with an electron beam, depends on the angle of incidence theoretically. However, there arises a difference in the image brightness depending on whether the tilted side of the specimen is directed to the secondary electron detector or the opposite side. With a long specimen, for example, the brightness differs between the side facing the detector and the opposite side. In such a case, directing the longitudinal axis of the specimen to the detector makes the brightness uniform.
Detector Position

Directing the longitudinal axis of the specimen to the secondary electron detector makes the right and left sides equally bright.
When a nonconductive specimen is directly illuminated with an electron beam, positive charges collect locally (specimen charge-up), thus preventing normal emission of secondary electrons. This charge-up causes some unusual phenomena such as abnormal contrast and image deformation and shift.

Usually, the surface of a nonconductive specimen is coated with some conductive metal prior to observation. Rough surfaced specimens must be evenly coated from every direction. Recently, however, a method has been employed to observe specimens without coating, in order to know their true surface state.
Generally, the following methods are used to reduce specimen charge-up.

1. Reducing the probe current
2. Lowering the accelerating voltage
3. 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 (this point varies with the specimen).
Charge up can be properly prevented by selecting the appropriate accelerating voltage.
Charge Up

(a) 4 kV

(b) 10 kV

foreleg of vinegar fly

Charge up can be properly prevented by selecting the appropriate accelerating voltage.
Figs. (a) and (b) show toner powder dispersed on double-sided tape and a little pressed. It is seen that no charge-up took place even when high accelerating voltage was used. Figs. (c) and (d) show toner powder fixed with manicure liquid. It is seen that charge-up is caused as the powder is not sufficiently fixed.
Beam Damage

The loss of electron beam energy in the specimen occurs mostly in the form of heat generation at the irradiated point. The temperature increase at an irradiated point is dependent on:

1. The electron beam accelerating voltage and dosage.
2. Scanning area.
3. Scanning time.
4. Heat conductivity of the specimen. 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.
Beam Damage

To avoid this damage, the following should be taken into consideration:

1. To use low accelerating voltage.
2. To decrease electron beam intensity.
3. To shorten exposure time, even though this reduces photograph smoothness slightly.
4. To photograph large scanning areas with low magnifications.
5. To 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 photograph the actual field as quickly as possible.
When a specimen area is irradiated with an electron probe for a long time at high magnification, it may be damaged as shown in Fig. (b).
Contamination

When the electron probe is irradiated on a specimen portion for a long time, its image may lose sharpness and become dark.

Specimen: ITO. A x18,000 photo taken after a long-time electron probe scanning at x36,000. As compared with the clear image of peripheral region, the middle region shows reduced contrast and lacks image sharpness.
Contamination

This is caused by the residual gas in the vicinity of the specimen being struck by the electron probe. This phenomenon is called specimen contamination. The conceivable residual gases in the specimen chamber, which cause contamination are:

1. Gas caused from the instrument itself.
2. Gas that specimens bring into the instrument
3. Gas that the specimen itself gives off.
Contamination

To prevent specimen contamination, special attention must be paid to the following matters:

1. Use the minimum amount of double-sided adhesive tape or conductive paint, and completely dry it before putting the specimen in the instrument.

2. In some cases, contamination can also be reduced by drying the adhesive with a drier or the like.

3. Use the smallest possible biological specimens.

4. Since some embedding agents and resins give off a large amount of gas, they need to be selected carefully. Also since organic gas is given off when the resin surface is irradiated with an electron probe, irradiate the smallest possible surface area or coat the surface with a conductive material.
WD is changeable on many recently available SEM models. The figure below shows what effect is produced on the image when WD is changed with other conditions kept unchanged.
Objective Aperture Diameter

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

Electric light bulb coil, 5 kV x540

- The smaller the OL aperture diameter and the longer the WD, the greater the depth of field.
Astigmatism

The aberration caused by the machining accuracy and material of the polepiece is called "astigmatism." This astigmatism can be removed by adjusting the stigmator.

Although blurring is noticed before and after the just-focus image, no unidirectional defocusing is seen in images with corrected astigmatism.

Images with stigmatism exhibit less sharpness.
Astigmatism

An image is judged as astigmatism-free if it has no unidirectional defocusing when the objective lens is changed to under or over-focus at a little high magnification (about x10000).

Shape changes in electron beam when there is astigmatism

Shape changes in electron beam when astigmatism is corrected
Images With Astigmatism

(a) under focus  (b) under focus  (c) just focus
(d) over focus  (e) over focus
After Astigmatism is Corrected

(f) under focus  (g) under focus  (h) just focus

(i) over focus  (j) over focus
Optimum Contrast And Brightness

A good SEM microscope is sharp, noiseless and provides optimum contrast and brightness. Optimum contrast and brightness can be adjusted automatically or by built in controls.

In some cases, however, the contrast and brightness are adjusted for the portion of interest only, and not for the image overall.
An Example

- Excessive contrast
- Optimum contrast and brightness
- Excessive brightness
- Insufficient brightness
- Insufficient contrast
External Disturbances

External disturbances such as a stray magnetic field, mechanical vibrations, etc. can cause image distortion, jagged edge lines and other phenomena. Often disturbances in SEM images are caused by structural or installation conditions such as:

1. Leakage:
   - Magnet field from distribution board
   - High-tension line located too close to the instrument
2. Low floor strength
3. Improper grounding

Installation conditions should be carefully checked in advance to avoid any problems after SEM installation.
An Example

Distorted images due to leakage of external magnetic field.
An Example

(a) shows jagged edge lines due to external mechanical vibration. (b) has no mechanical vibration.
Image Distortion

If correct deflection magnification is lost horizontally or vertically, it results in image distortion. In some cases, latex particles must be used for checking purposes.

(a) Normal image

(b) Horizontally distorted image
The image is horizontally demagnified by $1/\cos 45^\circ$ after specimen tilt.
Influence of external magnetic field on image. Compared with (b), (a) is demagnified at the center and magnified at both sides, because of an intense magnetic field, 50 Hz.
When the column is disassembled for cleaning or when the electron beam is lost, an operation called "alignment of the column" allows for the electron beam from the filament to be most effectively collected onto the specimen surface by means of mechanical and electrical alignment. In this operation, the electron beam from the gun should first be aligned using a tilting correction knob, then the objective aperture should be adjusted to make the electron beam pass the objective lens center.
Column Alignment

After replacing or cleaning the objective aperture, it is necessary to adjust the objective aperture position. It is ideal to set the objective aperture at the center of the objective polepiece. When the aperture shifts from this position, however, the astigmatism of the image becomes extremely high, making it impossible to obtain high-resolution images.
Column Alignment

(a) Incorrect alignment

(a) Correct alignment

Zinc Oxide, x21000
The secondary electron detector and CRT are supplied with a high voltage, 10 kV. The poor connection of cables, exfoliation of the fluorescent paint, and the presence of dust on the metal ring and fluorescent plane of the detector can all cause discharges.
Discharge of Detector and CRT

The images below show images under a 10 kV discharge. The effect appears to be similar to those of unusual accelerating voltage, unsatisfactory gun emission and specimen charge-up. The difference from their effect is that only brightness is changed, with no defocusing, image cut and image shift observed.

(a) Discharge of a detector  
(b) Discharge of CRT
The photo on the upper left shows the sample chamber located at the base of the column. The upper right photo shows the lens and detectors located inside the sample chamber. The lower right is the top view of a chamber with various detectors.
Sample Stage

A prepared sample is mounted on a specimen stub and placed on the stage.

The operator can move or rotate the sample automatically or manually.
Specimen Preparation

The regular SEM requires a conductive sample. **Three requirements** for preparing samples for a regular SEM are:
1) Remove all water, solvents, or other materials that could vaporize while in the vacuum.
2) Firmly mount all the samples.
3) Non-metallic samples, such as bugs, plants, fingernails, and ceramics, should be coated so they are electrically conductive. Metallic samples can be placed directly into the SEM.

An environmental SEM, so called ESEM, can be used to examine a non-conductive sample without coating it with a conductive material.
Mounting the Sample

Any specimen, whether it has been sputter coated or is naturally conductive, must be firmly attached to the specimen support before being viewed in the SEM. Attention to detail in the mounting procedure is very important if a researcher desires a quality result.
Mounting Non-conductive Samples

Make sure the support (specimen stub) is clean before use and also check to make sure the stub you are using is compatible with the stage of the SEM you will be using. Place the specimen on the stub before the sputter coating procedure. This will increase the conductivity and therefore the quality of your results.
Mounting Conductive Samples

The specimen stub you choose should be of a material that will not interfere with the backscattered-electron and x-ray signals that will be emitted from the specimen. Again, make sure the stub is clean and compatible with the SEM you are using.
The sputter coater is used to coat non-metallic samples (ceramics, bugs, plants, human hair, etc.) with a thin layer of gold, platinum or carbon. This makes them conductive, and ready to be viewed by the SEM.
Prevention of charge-up by sampling

Biological, cloth, and powder specimens cannot often be photographed clearly, with some portions looking too bright and some too dark. This is because those specimens are partly charged up. To prevent this, it is necessary to give specimen surfaces uniform conductivity as follows.
Prevention of charge-up by sampling

- When fixing the specimen on a specimen stub, apply conductive paint (carbon paint or the like) to specimen portions which are hard to coat.

![Diagram of specimen fixation](image)

(b) Insufficient adhesive  (a) Sufficient adhesive

Fixing of a bulk specimen
Prevention of charge-up by sampling

- In the case of powder, if its particles are piled on each other, charge-up easily takes place, causing them to move during observation. To prevent this, after the adhesive for fixing the power is dried, blow the piled particles using a hand blower.
The development of the ESEM has filled the longtime desire of scientists to view specimens and processes in their natural state.

The ESEM allows the examination of specimens surrounded by a gaseous environment. This means that a specimen viewed in the microscope does not need to be conductive and therefore does not need to be coated with a conductive material. Even liquids can be viewed in the microscope.
How does ESEM work?

The primary electron beam hits the specimen which causes the specimen to emit secondary electrons. The electrons are attracted to the positively charged detector electrode.
How does ESEM work?

As they travel through the gaseous environment, collisions occur between an electron and a gas particle results in emission of more electrons and ionization of the gas molecules. This increase in the amount of electrons effectively amplifies the original secondary electron signal.

The positively charged gas ions are attracted to the negatively biased specimen and offset charge-up effects.
How does ESEM work?

As the number of secondary electrons varies the amplification effect of the gas. If a large number of electrons are emitted from a position on the specimen during a scan, there is a high signal.

If only a small amount of electrons are emitted the signal is less intense. The difference in signal intensity from different locations on the specimen allows an image to be formed.
Energy Dispersive Spectroscopy

When the sample is bombarded by the electron beam of the SEM, electrons are ejected from the atoms comprising the sample's surface. A resulting electron vacancy is filled by an electron from a higher shell, and an x-ray is emitted to balance the energy difference between the two electrons.
Energy Dispersive Spectroscopy

The X-ray is called characteristic because its energy equals the energy difference between the two levels involved in the transition and this difference is characteristic of the element.

The technique utilizes x-rays that are emitted from the sample during bombardment by the electron beam to characterize the elemental composition of the analyzed volume. Features or phases as small as about 1µm can be analyzed.
Energy Dispersive Spectroscopy

The EDS x-ray detector measures the number of emitted x-rays versus their energy. The energy of the x-ray is characteristic of the element from which the x-ray was emitted. A spectrum of the energy versus relative counts of the detected x-rays is obtained and evaluated for qualitative and quantitative determinations of the elements present in the sampled volume.
Energy Dispersive Spectroscopy

The left figure shows an X-ray detection system attached with a scanning Electron Microscope. The detector is located at the end of the cylindrical liquid nitrogen Dewar.
Energy Dispersive Spectroscopy

The X-ray signal from the sample is picked-up by a solid-state Detector. A series of amplifiers in combination with the detector are then used to convert the X-ray signal into a voltage signal and to amplify it. The final signal is sent to a computer for processing such as peak identification or quantification and for display of the data in the form of a spectrum of intensity versus emission energy. The energy peaks are fingerprints of the specific elements in a specimen.
The window used determines the analysis range. A Be window enables detection from $^{11}_{\text{Na}}$ to $^{92}_{\text{U}}$, while a newly-introduced organic ultra-thin window enables detection from $^{4}_{\text{Be}}$ to $^{92}_{\text{U}}$. 
X-Ray Detector

When the Si(Li) detector is hit by X-ray quanta with energy $E$, electron-hole pairs were generated. In low temperature, the energy used is $\varepsilon=3.8$ eV per electron-hole pair. Therefore, the number of electron-hole pairs is $E/\varepsilon$. For example, 1550 electron-hole pairs can be generated by one Mn K\(\alpha\) photon, whose energy is 5.895 keV.

The pulse height of detector output depends on the number of electron-hole pairs.
EDS Qualitative Analysis

EDS qualitative analysis is based on Moseley formulism of characteristic X-rays.

\[ \sqrt{\nu} = K(Z - \sigma) \quad (\lambda = \frac{c}{\nu}) \]

\( \nu \) is the frequency of characteristic X-rays, \( Z \) is the atomic number, \( K \) and \( \sigma \) are constants, and \( c \) is speed of light in vacuum.
EDS Qualitative Analysis

- The wavelength (energy) of characteristic X-rays varies monotonically with atomic number Z.
- The energy of the X-rays does not change with accelerating voltages of the incident electron beam.
Qualitative Analysis

The vertical axis is the number of pulses (the number of photons detected), the horizontal axis is the pulse height (the energy of photons).

The number of pulses depends on the concentration of the elements, while the pulse height is determined by the atomic number of the corresponding elements.
Quantitative Analysis

Quantitative results are readily obtained without standards. The accuracy of standardless quantitative analysis is highly sample dependent. Greater accuracy is obtained using known standards with similar structure and composition as the unknown sample.

One critical step of quantitative analysis is fit and subtraction of background.
Qualitative Analysis

$C_A$, the concentration of element A, is proportional to its characteristic X-ray intensity: $C_A \propto I_A$.

If the intensity of the same X-ray from element A in an unknown sample and a standard sample is measured, at the same experiment parameters, $C_A$ of the sample is determined as

$$C_A = C_A^{Std} \frac{I_A}{I_A^{Std}}.$$
Operation Mode

Point analysis: Analysis is performed when electron beam is fixed at the point of interest.
Operation Mode

Line profile: Analysis is performed when electron beam is moving along a certain line to obtain composition variation along the line. Combined with secondary electron or backscattered electron images, an intuitive picture can be achieved.
**Operation Mode**

**Elemental mapping**: Analysis is performed when electron beam is rastered on the sample surface. Qualitative results of composition distribution on the surface is obtained.

The SE images are form with SI signals, while the elemental mapping is formed with X-ray signals.
Advantages of EDS

- High speed analysis: all elements are simultaneously detected.
- High efficiency of signal collection: the detector is put closely beside the sample, favorable to analyses of rough surfaces and powders.
- Low beam damage: high beam current is not necessary, favorable to biological samples.
Disadvantages of EDS

- Poor resolution: ~ 130 eV
- Low Peak/Background ratio
- Overlap of peaks
- Require low temperature
Wavelength Dispersive Spectroscopy

WDS differentiates between x-ray photons based on their *wavelength*, hence the name. The analyzer is set up to diffract the x-ray's coming from the sample off of one of several crystals and onto a suitable detector. While some spectrometers are fixed to detect a single element, many offer several crystals and can be driven through a wide range of angles to detect virtually all elements heavier than boron.
Characteristic X-rays are photons with characteristics wavelength. Using proper crystals with known lattice spacing \( d \), with the help of \( 2d \sin \theta = \lambda \), photons with different wavelength are dispersed in different \( 2\theta \).
Wavelength Disperse

The distance between the sample and crystal $L=2R\sin\theta$. $R$ is the radius of the Rowland cycle. Thus, $L=(R/d)n\lambda$.

$R$ and $d$ are constants. When the crystal moves along $L$, the detector collects signals with different $\lambda$. 
Structure of WDS
## Crystals Used in WDS

<table>
<thead>
<tr>
<th>Name</th>
<th>2d, Å</th>
<th>Lowest atomic number diffracted</th>
<th>Resolution</th>
<th>Reflectivity</th>
</tr>
</thead>
<tbody>
<tr>
<td>α-Quartz(1011)</td>
<td>6.687</td>
<td>( K_{\alpha 1} ) 15-P, ( L_{\alpha 1} ) 40-Zr</td>
<td>High</td>
<td>High</td>
</tr>
<tr>
<td>KAP(1010)</td>
<td>26.632</td>
<td>( K_{\alpha 1} ) 8-O, ( L_{\alpha 1} ) 23-V</td>
<td>Medium</td>
<td>Medium</td>
</tr>
<tr>
<td>LiF(200)</td>
<td>4.028</td>
<td>( K_{\alpha 1} ) 19-K, ( L_{\alpha 1} ) 49-In</td>
<td>High</td>
<td>High</td>
</tr>
<tr>
<td>PbSt PET</td>
<td>100.4</td>
<td>( K_{\alpha 1} ) 5-B, ( K_{\alpha 1} ) 13-Al, ( L_{\alpha 1} ) 36-Kr</td>
<td>Medium</td>
<td>Medium</td>
</tr>
<tr>
<td>RAP</td>
<td>26.121</td>
<td>( K_{\alpha 1} ) 8-O, ( L_{\alpha 1} ) 33-As</td>
<td>Medium</td>
<td>Medium</td>
</tr>
</tbody>
</table>
Detector

gas-flow proportional counter
Advantages

Great resolving power: helps greatly in resolving nearby peaks. Whereas an EDS system might specify a resolution of 130 eV for a 5.9 KeV peak, WDS would yield a resolution of much less than 10 eV for the same peak.

High peak-to-background ratio: serves to lower the detection limit of an element and also results in clean (i.e., low background) x-ray maps.
Disadvantages

It is a sequential technique. While EDS collects and displays the x-ray spectrum for the whole energy range in parallel, WDS can only measure at a single wavelength at a time. Therefore, it takes much longer to survey the entire spectrum.

Also, WDS is not as efficient in detecting x-rays as is EDS. A given beam current will generally result in a higher count rate on an EDS system than on a WDS system. The high currents needed for decent WDS performance lead to a larger beam size and a loss of spatial resolution. It can also lead to specimen damage.
## Comparison of WDS and EDS

<table>
<thead>
<tr>
<th></th>
<th>WDS</th>
<th>EDS</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>analysis range</strong></td>
<td>$^4\text{Be}$ – $^{92}\text{U}$</td>
<td>$^4\text{Be}$ – $^{92}\text{U}$</td>
</tr>
<tr>
<td><strong>analysis speed</strong></td>
<td>slow</td>
<td>fast</td>
</tr>
<tr>
<td><strong>energy resolution</strong></td>
<td>high (~ 5eV)</td>
<td>low (~130 eV)</td>
</tr>
<tr>
<td><strong>analysis limit</strong></td>
<td>$10^{-2}%$</td>
<td>$10^{-1}%$</td>
</tr>
<tr>
<td><strong>efficiency</strong></td>
<td>low</td>
<td>high</td>
</tr>
<tr>
<td><strong>P/B ratio</strong></td>
<td>10</td>
<td>1</td>
</tr>
</tbody>
</table>
Electron Backscatter Diffraction

Electron backscatter diffraction (EBSD), or electron backscatter pattern (EBSP) is one of the most exciting techniques in scanning electron microscopy. EBSD can be used to examine a wide range of crystalline materials and to measure microstructure, orientation, texture (microtexture) and boundary properties. It can also be used in conjunction with chemical analyses to identify unknown phases. In short, EBSD can provide a complete overview of a material's physical properties at a microstructural level.
Backscattered Kikuchi Pattern

The EBSD technique (also known as Backscattered Kikuchi Diffraction, BKD) was first developed by Alam and co-workers in 1954, who described some diffraction patterns and called them "wide-angle back-scatter Kikuchi patterns", in recognition of related diffraction phenomena reported by Kikuchi in the 1920's. However, it was not until the 1970's that Venables and co-workers applied EBSD to metallurgical microcrystallography, paving the way for a more widespread application of EBSD to the materials sciences in the ensuing 20 years.
Backscattered Kikuchi Patterns

Backscattered Kikuchi patterns are generated in the SEM by illuminating a highly tilted sample (70°, for example) with a stationary electron probe.

The electron beam is directed at the point of interest on the sample surface: initial elastic scattering of the incident beam causes the electrons to diverge from a point just below the sample surface and to impinge upon crystal planes in all directions.
Backscattered Kikuchi Pattern

Wherever the Bragg condition for diffraction is satisfied by a family of atomic lattice planes in the crystal, two cones of diffracted electrons are produced. These cones are produced for each family of lattice planes.

Two flat cones of higher electron intensity which when imaged on a phosphor screen appear as pairs of parallel nearly straight lines. There are pairs of lines due to interactions with either the front or the back of the atomic planes.
Backscattered Kikuchi Pattern

These cones of electrons can be imaged using a phosphor screen attached to a sensitive camera. The camera is usually positioned horizontally, so that the phosphor screen is close to the sample in order to capture a wide angle of the diffraction patterns. Where the cones of electrons intersect with the phosphor screen, they appear as thin bands. These are called "Kikuchi bands", and each one corresponds to a family of crystal lattice planes. The resulting EBSP is made up of many Kikuchi bands.
Backscattered Kikuchi Pattern

$2 \theta \approx 1 \text{ degree}$ so cones intersect the screen as nearly straight lines.

formation of Kikuchi bands
Backscattered Kikuchi Pattern

EBSD software automatically locates the positions of individual Kikuchi bands, compares these to theoretical data about the relevant phase and rapidly calculates the 3-D crystallographic orientation.

The whole process from start to finish can take less than 0.05 seconds.
Backscattered Kikuchi Pattern

Kikuchi patterns change with the change of grain orientation.

alumina trigonal, R3c
Backscattered Kikuchi Pattern

Kikuchi patterns change with the change of grain orientation.

alumina trigonal, R3c
Backscattered Kikuchi Pattern

Kikuchi patterns change with the change of grain orientation.

alumina trigonal, R3c
Kikuchi patterns change with the change of grain orientation.
Backscattered Kikuchi Pattern

Kikuchi patterns change with the change of grain orientation.

lead titanate tetragonal, P4mm

Dr. Di Wu, Nanjing University
Kikuchi patterns change with the change of grain orientation.

lead titanate tetragonal, P4mm
Backscattered Kikuchi Pattern

Kikuchi patterns change with the change of grain orientation.

Lattices with less symmetry have more complex patterns.

augite monoclinic, C2/c
Backscattered Kikuchi Pattern

Kikuchi patterns change with the change of grain orientation.
Lattices with less symmetry have more complex patterns.
Backscattered Kikuchi Pattern

Kikuchi patterns change with the change of grain orientation.

Lattices with less symmetry have more complex patterns.
The left figure shows an EBSD camera attached on a SEM.
Effect of Sample Tilt

- The larger the tilt angle, the stronger the electron signals.

- However, the smaller the tilt angle, the better the resolution perpendicular to the tilt axis.
EBSD Application Example I

---- to determine grain orientation population of PZT films

This secondary electron micrograph shows the general microstructure of the PZT thin film. Large grains, often with regular forms (e.g. star shaped), are isolated in a matrix of much smaller grains. These shapes may be explained by interpenetrative twinning, resulting in regular twin domains.
EBSD Application Example I

---- to determine grain orientation population of PZT films

Lead zirconium titanate (PZT) thin films are important electronic materials, which have a wide range of potential applications in non-volatile random access memories, piezoelectric actuators, transducers, pyroelectric detectors, and etc.

PZT is a ceramic material. The grain orientation has a great effect on the electrical properties. Thus, this example gives an analysis on grain orientation by EBSD.
In one of the large star-shaped grains, it is clear that the EBSPs 1 and 3 are identical, as are the EBSPs 2 and 4. Indexing these EBSPs as tetragonal (pseudo-cubic) PZT confirms that the orientation pairs are twin related, with a 60° rotation about a <111> axis.
EBSD Application Example I

Small orientation maps were collected of individual large grains or clusters of large grains. It is clear that all the large grains are twinned. The twinning characteristics vary from grain to grain, but typically they have 2-fold or 3-fold structures.
The microstructure of the matrix was characterized using automated EBSD mapping. These images show the results of a typical EBSD analysis. (a): EBSP quality map, the scale bar = 50 µm. (b): Orientation map of the same area, with colors corresponding to the Euler angles. The wide range of colors indicates that there is almost no texture in this sample.
Grain size histogram for the matrix area. The mean grain size is 7.5 µm.
Conclusions can thus be made:

1. Two contrasting grain types have been identified on the surface of the silicon single crystal wafer: A matrix population of small grains (mean < 8 µm), with no texture and very few twin boundaries. Isolated large grains, typically 100-250 µm diameter, all characterized by distinctive twinning. These twins typically form two-fold or three-fold domains.

2. It is likely that some form of abnormal grain growth has occurred in order to produce these large twinned grains. The EBSD data show that they do not have any preferred orientation.

3. In summary, EBSD analyses have identified contrasting microstructural characteristics of two grain populations in these samples: this could have a damaging effect on the electrical properties of the PZT film.
EBSD Application Example II

---- to identify the various phases in a complex alloy system

This application considers recent work on alloy development using diffusion multiples to study complex multi-component alloy phase equilibria.

The left figure is a schematic of an actual diffusion multiple used to explore the phase equilibria in the Cr, Nb, Si, Ti alloy space.
EBSID Application Example II

---- to identify the various phases in a complex alloy system

A diffusion multiple brings into one specimen a multiple of diffusion couples as well as complex regions of contact between three or even four phases.

A diffusion couple is simply two phases of known composition and structure, often pure elements, held together at constant temperature. After they have reached equilibrium, the reacted couple is analyzed, revealing both the equilibrium phases between the end members and the diffusion paths between them.
The left figure is a backscatter electron image from one region circled in the figure of the last page. The region contains several phases. Electron probe microanalysis has been used to determine the compositions of the phases in quilibrium with one another. The traces of the microprobe analysis are faintly visible in the image (vertical lines ~40 µm apart) due to carbon contamination.
The compositions measured in the diffusion multiple can be plotted on a constant temperature phase diagram as is shown in this figure. As there are several known phases in the binary systems, a number of possible ternary phases and significant solid solubility, composition alone is not enough to unambiguously determine what phases are present.
This figure shows one of the as-captured and as-analyzed diffraction pattern pairs from the diffusion multiple region shown in the last page. The phase identified is the ternary phases, orthorhombic \((\text{Cr,Nb})_{11}\text{Si}_8\).
This figure shows another of the as-captured and as-analyzed diffraction pattern pairs from the diffusion multiple region shown in the page before the last. The phase identified is the ternary phases, orthorhombic (Cr,Nb)$_6$Si$_5$. 
This table lists all of the phases used for the EBSD analysis of the Nb, Cr, Ti, Si diffusion multiple and their structure type.

<table>
<thead>
<tr>
<th>Phase</th>
<th>Space Group</th>
<th>symmetry</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nb</td>
<td>229</td>
<td>Cubic</td>
</tr>
<tr>
<td>Cr</td>
<td>229</td>
<td>Cubic</td>
</tr>
<tr>
<td>Ti</td>
<td>229</td>
<td>Cubic</td>
</tr>
<tr>
<td>Ti</td>
<td>194</td>
<td>Hexagonal</td>
</tr>
<tr>
<td>Si</td>
<td>227</td>
<td>Cubic</td>
</tr>
<tr>
<td>Nb$_2$Si$_2$</td>
<td>181</td>
<td>Hexagonal</td>
</tr>
<tr>
<td>Nb$_5$Si$_3$</td>
<td>193</td>
<td>Hexagonal</td>
</tr>
<tr>
<td>CrSi$_2$</td>
<td>180</td>
<td>Hexagonal</td>
</tr>
<tr>
<td>CrSi</td>
<td>198</td>
<td>Cubic</td>
</tr>
<tr>
<td>Cr$_5$Si$_3$</td>
<td>140</td>
<td>Tetragonal</td>
</tr>
<tr>
<td>Cr$_3$Si</td>
<td>223</td>
<td>Cubic</td>
</tr>
<tr>
<td>Cr$<em>2$Nb(C$</em>{14}$)</td>
<td>194</td>
<td>Hexagonal</td>
</tr>
<tr>
<td>Cr$<em>2$Nb(C$</em>{15}$)</td>
<td>227</td>
<td>Cubic</td>
</tr>
<tr>
<td>(Cr,Nb)$_{11}$Si$_8$</td>
<td>62</td>
<td>Orthorhombic</td>
</tr>
<tr>
<td>(Cr,Nb)$_6$Si$_5$</td>
<td>72</td>
<td>Orthorhombic</td>
</tr>
<tr>
<td>NbCrSi</td>
<td>189</td>
<td>Hexagonal</td>
</tr>
</tbody>
</table>
The following conclusions can be drawn:

EBSD is an essential tool, along with compositional microanalysis by electron microprobe, for determining multi-component phase diagrams from diffusion multiples. The inherent automation and high rate of analysis in EBSD work can be very beneficial for these kinds of combinatorial studies. Diffusion multiples are extremely useful for probing multi-component phase equilibria as they cover a broad range of compositional space and are well suited to automated microanalysis.
EBSD Application Example III

---- grain size, grain boundary and texture analysis of a Cu thin film

There are more than 1 million circuit elements are placed on a single chip of ultra-large scale integrated circuits (ULSICs). Recently, copper has gained interest as an interconnect material for ULSICs due to both its low resistivity and its low electron migration resistance when compared to currently used Al-alloys.

The electrical properties of a thin film are influenced considerably by the grain size, the grain boundary characteristics and the texture.

In this example, EBSD is used to analyze an as-deposited Cu thin film in order to determine the influence of the microstructure on the material's electrical properties.
The left figure shows the variation in diffraction pattern quality across the scanning area, illustrating the general microstructural characteristics of the sample. The right figure shows the corresponding orientation map, with colors corresponding to the crystallographic orientation of each point. The inset shows the inverse pole figure for this area (normal direction).
This figure gives a grain boundary map of the analysis area. General high angle (> 10°) grain boundaries are shown in black, other boundaries in color. The $\Sigma 3$ boundary (rotation of 60° about <111>) in red is the predominant boundary type.
The frequency distribution of all boundary types, with the colors corresponding to the figure in the last page.
This figure shows an orientation map in which the different colors represent different texture components. Yellow shows grains with a [100]-fibre texture ([100] || normal direction), green represents gamma-fibre texture ([111] || normal direction) and purple represents [110]-fibre texture ([110] parallel || normal direction).
The grain sizes in this thin film are dependent on the texture characteristics. Those grains contributing to the [100]-fibre texture have a larger grain size than grains contributing to either the gamma-fibre or the [110]-fibre textures.
This single analysis shows that:

- The sample has a strong crystallographic texture with two principle components ([100] and gamma fibres).
- The $\Sigma 3$ twin boundary is the predominant boundary type in the microstructure (accounting for up to ~ 42% of all high angle boundaries).
- The grain size distribution is strongly dependent on the texture, with grains having [100]-fibre orientations over twice the size of grains with gamma or [110]-fibre orientations.

These observations allow manufacturers to amend the formation process for Cu thin films in order to enhance the electrical properties of the material and to improve their application in ULSICs.
The End