V. The Scanning Electron Microscope

A. The instrument

The most common form of electron microscope is the scanning electron microscope (SEM), which was pioneered in the late 1940s by Charles Oatley and co-workers at Cambridge University. In these microscopes the lenses are placed before the sample, allowing electrons to be focused onto a small spot that is then scanned across the surface to build up a 2D image. The ability of the scanning electron microscope to image bulk samples makes it extremely versatile. Whereas the resolution of the transmission electron microscope is limited by the wavelength of the electrons and the quality of its lenses, the resolution of the scanning variety is limited by the relatively large interaction region between the beam and the sample.

The electron source in an SEM is typically a tungsten filament, although field emission guns (FEGs) are increasingly being used for higher resolution. The electrons are accelerated to an energy which is usually between 20 - 30 keV. Two or three condenser lenses then demagnify the electron beam until, as it hits the specimen, it may have a diameter of only 2-10nm. The diagram above is a simplified plan of a microscope like the JEOL 6300. In reality, the objective lens shown is part of the condenser system and a separate objective lens is located just below the scan coils, which are themselves split into two parts. On other microscopes, the diagram is accurate, but the aperture is placed below the scan coils.

On older instruments, the fine beam of electrons is scanned (rastered) across the specimen by the scan coils, while a detector counts the number of secondary or backscattered electrons. At the same time, a CRT is scanned and the signal is modulated by the amplified current from the detector. In more modern instruments, the same effect is achieved by digitally controlling the beam position on the sample, and the resultant image is displayed on a computer screen.
The magnification mechanism is very simple and involves no lenses at all. The raster scanned by the electron beam is simply made smaller than the raster on the CRT or computer monitor. For example, if the electron beam is made to raster a 10µm x 10µm area on the specimen, and the CRT is 100mm x 100mm, the linear magnification is 10,000x.

B. Detecting Electrons

1. Everhart-Thornley (SEI) detector

The Everhart-Thornley detector is a scintillator-photomultiplier which is normally sited to the side of the specimen chamber in the SEM. Low energy secondary electrons are attracted to the small positive voltage on the screen (+200V, giving a field strength of only +10V/mm). Once they pass through the screen they are accelerated by the much higher voltage (+10,000V) to impact on the scintillator with high energy causing light emission. The light emitted is detected by the photo-multiplier outside the microscope column. The Faraday cage shields the microscope (and electron beam) from the high voltage in the detector and improves the collection efficiency by attracting electrons which weren’t initially moving towards the detector. For flat specimens, nearly all the secondary electrons can be collected in this way.
2. Backscatter electron (BEI) detectors

Backscattered electrons travelling in the appropriate direction will also hit the Everhart-Thornley detector and contribute to the secondary electron image; therefore, the secondary electron signal always contains some backscattered component as well. If the scintillator is switched off or given a small negative bias, then secondary electrons will be excluded from the detector (their low energies cannot excite the scintillator without the help of the bias field) and a pure backscattered signal is obtained; however, only those electrons travelling along the direct line of sight towards the detector will be collected, making the efficiency very poor for geometrical reasons. This method is rarely used now.

Purpose-built backscattered electron detectors are usually placed just below the final aperture in the SEM, where the collection efficiency is greatest. Scintillators like the Robinson detector, which operates on the same principle as the Everhart-Thornley SEI detector, can be used in this geometry; but they are bulky, restrict the working distance of the microscope, and may need to be retracted if, for example, it is necessary to detect x-rays.

Most modern detectors consist of an annular solid state $p$-$n$ diode which creates a cascade of electrons when the backscattered electron enters. The response time of the diode is relatively slow so that slow beam scan rates are normally required.
The number of backscattered electrons produced depends strongly on the atomic number of elements in the specimen. The number arriving at the detector will also depend on the shape of the surface. **For good composition contrast images, a polished specimen is usually required.**

C. Optics

A typical SEM contains two lenses, a **condenser** lens and an **objective** lens, as well as scan coils. The condenser lens performs the same function here as in a TEM, that is, to reduce (demagnify) the diameter of the beam (source image).
If the diameter of the filament is \( d_0 \) and it is placed a distance \( u_1 \) from the condenser lens, then the diameter of the demagnified filament image \( d_1 \), which forms at a distance \( v_1 \) on the other side of the condenser lens, is:

\[
d_1 = d_0 \times \frac{v_1}{u_1}
\]

From this equation it is apparent that the stronger the condenser lens (the shorter \( v_1 \)), the smaller will be the beam diameter \( d_1 \).

Unlike in a TEM, the objective lens in an SEM is used to further demagnify the beam, not for magnification of the image. The diameter of the final probe, \( d \), on the specimen is:

\[
d = d_1 \times \frac{v_2}{u_2} = d_1 \times \frac{WD}{u_2}
\]

where \( u_2 \) is the distance between the demagnified source image (cross-over below the condenser lens with a diameter \( d_1 \)) and the objective lens and \( v_2 \) is the working distance (WD), defined as the distance between the objective lens and the specimen.

The physical distance between condenser and objective lenses \( (v_1 + u_2) \) is obviously a constant for a given instrument, so that a strong condenser lens (small \( v_1 \)) inevitably results in a larger \( u_2 \), which in turn results in a smaller \( d \). The probe size in an SEM is decreased by either increasing the strength of the condenser lens or decreasing the working distance.

As in TEM, in order to decrease spherical aberration, an objective aperture is used to exclude off-axis rays. If the semi-angle of the condenser and objective lenses are \( \alpha_0 \) and \( \alpha_1 \), respectively, then the current in the final probe is:

\[
I_1 = I_0 \times \left(\frac{\alpha_1}{\alpha_0}\right)^2
\]

The current decreases as \( \alpha_0 \) increases (stronger condenser lens) or \( \alpha_1 \) decreases (smaller objective aperture).

**D. Performance**

1. **Pixels**
   Unlike in a TEM, images are built up in an SEM from picture elements (pixels). A typical pixel on a compute screen or CRT is about 100 \( \mu \text{m} \) across, and each one will correspond to a pixel on the specimen (specimen pixel size) with a diameter \( p \) of:

\[
p = \frac{100 \mu \text{m}}{M}
\]

where \( M \) is the magnification. This diameter is the effective diameter of the interaction volume. In order to be resolved, two features must occupy two separate pixels; therefore, the resolution of the SEM can never be better than the specimen pixel size (or the effective diameter of the
interaction volume). If the probe size is larger, then information from adjacent pixels is merged and resolution degraded. If the probe size is smaller, then the signal is weak and possibly noisy. It is usually best to use a probe diameter equal to the specimen pixel diameter. In practice, this means that the probe size should be reduced as magnification increases.

2. Depth of field
If a beam is focused on a specimen, the beam diameter defocus, $s$, over a vertical distance, $h$, will be less than:

$$s = h\alpha$$

If the beam diameter is not greater than a specimen pixel diameter, then the image will remain in focus and, still assuming a pixel size of 100 $\mu$m:

$$h = \frac{s}{\alpha} = \frac{0.1\text{mm}}{M\alpha}$$

From the figure above, it is clear that:

$$\alpha \approx \tan \alpha = \frac{A}{2WD}$$

Now, combining terms we obtain:

$$h = \frac{0.1\text{mm}(2WD)}{M\frac{A}{AM}} = \frac{0.2WD}{AM}\text{mm}$$

So, the depth of field is increased by increasing the WD or decreasing the size of the objective aperture, both of which simultaneously degrade resolution in the SEM. A third way is by decreasing the magnification.

E. Resolution

In the scanning electron microscope, resolution is limited by the source brightness.

$$d_b = \frac{\pi}{2\alpha} \left( \frac{I_c}{\beta} \right)^{1/2}$$

where $\beta$ is the source brightness (controlled by the electron gun), and $I_c$ is the minimum beam current required to provide sufficient contrast for a feature to be seen above the signal noise level. So, to improve the resolution of an SEM it is necessary to use a large aperture, small probe size, and a bright electron gun; hence, for high resolution work, an SEM with a field emission electron gun (FEG SEM) is typically used.
1. Probe size
We have already seen that the probe size can be decreased by increasing the strength of the condenser lens or decreasing the working distance; however, decreasing the working distance necessarily increases $\alpha$, which has the effect of increasing spherical aberration (see Ch. 1). The minimum probe diameter, which corresponds to the best resolution in an SEM, is given by:

$$d_{opt} = K\lambda/C_s$$

where the constant $K$ is typically around 1.219. This equation is essentially identical to that given for TEM in Ch. 1. For a typical SEM operating at 20 kV ($\lambda = 0.08589$ Å) with $C_s = 20$ mm, $d_{opt} = 2.3$ nm.

As the probe diameter decreases, its current also decreases. The relationship can be quantified for thermionic emission by the Pease-Nixon equation:

$$d = d_{opt} \left[ 7.92 \times 10^9 \left( \frac{IT}{j} \right) + 1 \right]^{1/6}$$

where $I$ is the probe current [A], $T$ is the absolute temperature of the filament, and $j$ is the current density at the filament surface [A/cm$^2$].

2. Beam current
In order to resolve two points on a specimen, there must be a visible difference in the signal generated from them. If the average number of electrons detected from one point is $\bar{n}$, then statistically that number will vary by up to $\sqrt{\bar{n}}$ about the mean.

Natural contrast, $C$, is defined as the normalized difference between the signals from two adjacent points on the specimen ($0 < C \leq 1$):

$$C = \frac{\bar{n}_1 - \bar{n}_2}{\bar{n}_1} = \frac{\Delta\bar{n}}{\bar{n}_1} \quad (\eta_1 > \eta_2)$$

It has been experimentally determined by Rose that the eye can only distinguish between two points if:

$$\bar{n}_1 > \bar{n}_2 + 5\sqrt{\bar{n}_1} \quad : \quad \Delta\bar{n} > 5\sqrt{\bar{n}_1}$$

i.e., signal 1 is greater than signal 2 by five times the noise in signal 1. Now, combining equations yields the minimum observable level of contrast:

$$C > \frac{5\sqrt{\bar{n}_1}}{\bar{n}_1} = \frac{5}{\sqrt{\bar{n}_1}} \quad : \quad \bar{n}_1 > (5/C)^2$$
Given $10^6$ pixels on a 1000 x 1000 pixel screen, a frame scan time $F$ (a complete scan takes $F$ seconds), and a beam current $I$, the number of electrons which enter the specimen at a given pixel is:

$$n_0 = \frac{It}{e} = \frac{IF \times 10^{-6}}{e}$$

The number of electrons actually detected, $n$, depends on the beam-specimen interactions and the efficiency of the detector. We can write

$$n = qn_0$$

where $q$ is the product of detector efficiency and electron yield ($0.1 \leq q \leq 0.2$ for secondary electrons).

Now, by combining equations we see that the critical current, $I_c$, which is required to see a contrast level $C$ between two adjacent pixels is:

$$I_c > \frac{4 \times 10^{-12}}{qFC^2} A$$

There is a minimum current required to observe a given contrast level. Increasing the scan time will decrease the critical current, and a naturally high-contrast specimen will require a lower $I_c$. The contrast actually observed in the image will increase as $\bar{n}$ increases, which is achieved in practice by either increasing the beam current or increasing the scan time (which decreases $I_c$); however, neither of these methods alters $C$, the natural contrast level.

This expression can be substituted into the one for $d$ above in order to predict the effect of contrast on the required probe size (and therefore resolution). In general for secondary electrons, the required probe size decreases as either natural contrast or scan time increases. When microscope manufacturers specify an ultimate resolution for an instrument, this value typically corresponds to an ideal high-contrast specimen. For samples with low contrast, the larger probe sizes necessary may result in much poorer resolution.

**F. Topographical images**

As we have already seen, both the secondary electron coefficient, $\delta$, and the back-scattered electron coefficient, $\eta$, are minimised when the sample is perpendicular to the beam. As the sample is tilted, electrons are increasingly likely to be scattered out of the sample rather than further into it; therefore, tilting the specimen towards the detector by 20-40° is common to maximise the detected signal. In particular, $\delta$ varies with tilt angle $\theta$ as:

$$\delta = \frac{\delta}{\sin \theta}$$
Quantitative information about topography can be obtained using **stereomicroscopy**. In this technique, two images are taken of a specimen, one tilted 10-15° with respect to the other. The two images are then viewed simultaneously, most successfully with the aid of a stereo viewer. Some microscopes are capable of displaying both images simultaneously as an anaglyph, one in red and the other in green. In this case, the three-dimensional image can be revealed by wearing appropriately coloured glasses. The vertical height difference between two points on the image is given by:

\[
h = \frac{p}{2M \sin(\theta/2)}
\]

where \( p \) is the relative lateral displacement in different parts of the images caused by parallax, \( M \) is the magnification, and \( \theta \) is the tilt or parallax angle.

It is also possible to obtain topographical information from back-scattered electrons, but doing so is problematic. As only line-of-sight backscattered electrons are detected, a small backscattered detector would make a rough surface appear with more shadows than it would when viewed with secondary electrons; however, as most backscattered electron detectors are annular, this effect is cancelled out. It is highly unusual to see topographical information presented with backscattered electron images.

**G. Compositional images**

Unlike the secondary electron coefficient, the backscattered electron coefficient is a function of atomic number. Heinrich used a simple third-order polynomial curve fit to show that, for an untilted sample, \( \eta_0 \) is approximately independent of primary electron energy in the range 5 - 100 keV and varies as:

\[
\eta_0 = -0.254 + 0.016Z - 1.86 \times 10^{-4}Z^2 + 8.31 \times 10^{-7}Z^3
\]

Like the secondary electron coefficient, \( \eta \) varies with sample tilt, but in a more complicated way:

\[
\eta = 0.89(\eta_0 / 0.89)^{\cos \phi}
\]

This form of contrast is typically weak and can be easily drowned out by topographical effects. For this reason, it is best practice to polish flat (and sometimes etch) specimens for backscattered analysis. The backscattered contrast can be calculated as:

\[
C = \frac{\eta_1 - \eta_2}{\eta_1} \quad (\eta_1 > \eta_2)
\]

So, phases with similar values of \( \eta \) will result in very weak contrast; therefore, they will require large critical currents, \( I_c \), and probe sizes, \( d \), to see and consequently result in poorer resolution images. For example, while Al and Pt have sufficiently different values of \( \eta \) to produce a natural
contrast of \( C = 0.685 \) and require a beam diameter of just 3.6 nm to image (assuming \( F = 100 \) s, \( q = 0.1 \), and \( E = 20 \) keV), \( \alpha \)-brass and \( \beta \)-brass (both alloys of Cu and Zn) have \( \eta \) values of 0.305 and 0.306, respectively, resulting in a contrast value of only \( C = 0.003 \) and a beam diameter (resolution) of 269 nm.

H. Crystallographic information

1. Channelling
The backscattered electron coefficient, \( \eta \), is a function of crystal orientation as a result of diffraction within the specimen, and this effect is called *electron channelling*. When an electron beam is incident on a crystalline specimen, the density of atoms which it encounters will vary with crystal orientation. Along certain directions, paths of low atomic density might be found, the co-called *channels*, which permit some of the beam intensity to penetrate more deeply into the specimen before beginning to scatter. When electrons scatter deeper in the specimen, they are less likely to be able to escape from the surface, so the backscattered coefficient is lower in these cases. For other orientations, the atom density might be higher and so these electrons start scattering almost immediately, resulting in a relatively higher backscattered coefficient. The maximum difference in \( \eta \) may only be about 5%, but it is sufficient to provide crystallographic information in some cases.

Channelling is strong (likely) when the Bragg equation is satisfied for just one set of planes. The stronger the diffraction from a particular grain, the darker that grain will appear in backscattered imaging (this is the same contrast one obtains in the TEM of strongly diffracting grains in BF imaging).

This contrast mechanism \((0.0005 < C < 0.05)\) is much weaker than atomic number contrast and requires:

1. A high brightness electron gun (FEG is recommended)
2. A small beam semi-angle \((\alpha < 10 \text{ mrad})\)
3. A backscattered electron detector with a large solid angle of collection
4. A clean, smooth sample surface free of preparation artefacts such as those introduced by mechanical polishing. A surface that is optically smooth will not work if the near-surface crystallinity has been disturbed with dislocations. Electropolishing often works.
5. A sufficiently high probe current to resolve the channeling contrast above the detector noise ($I > 1.5 \text{ nA}$)

As we have already seen, the backscattered coefficient, $\eta$, increases with tilt angle, so high tilts are sometimes used to maximise $\eta$, which also increases the channelling contrast.

Channelling contrast can detect small orientation changes in specimens, and even dislocations can be revealed by their effect on the planes around them. It is currently used on metallurgical and geological samples.

The spatial resolution of channelling is better than for typical backscattered electron images and is optimum for low accelerating voltages and large atomic numbers.

2. Electron backscatter diffraction (EBSD)

EBSD is a technique which allows crystallographic information to be obtained from samples in the scanning electron microscope (SEM). In EBSD a stationary electron beam strikes a tilted (~70°) crystalline sample and the diffracted electrons form a pattern on a fluorescent screen. This pattern, like others we've seen in this course, is characteristic of the crystal structure and orientation of the sample region from which it was generated. The diffraction pattern can be used to measure the crystal orientation or grain boundary misorientations, discriminate between different materials, and provide information about local crystalline perfection. When the beam is scanned in a grid across a polycrystalline sample and the crystal orientation measured at each point, the resulting "texture" map will reveal the constituent grain morphology, orientations, and boundaries.

The principal components of an EBSD system are:
- A sample tilted at 70° from the horizontal.
- A phosphor screen which is fluoresced by electrons from the sample to form the diffraction pattern.
- A sensitive charge coupled device (CCD) video camera for viewing the diffraction pattern on the phosphor screen.
For EBSD, a beam of electrons is directed at a point of interest on a tilted crystalline sample in the SEM. The mechanism by which the diffraction patterns are formed is complex, but the following model describes the principal features. The atoms in the material inelastically scatter a fraction of the electrons with a small loss of energy to form a divergent source of electrons close to the surface of the sample. Some of these electrons are incident on atomic planes at angles which satisfy the Bragg equation. These electrons are diffracted to form a set of paired large angle cones corresponding to each diffracting plane. When used to form an image on the fluorescent screen the regions of enhanced electron intensity between the cones produce the characteristic Kikuchi bands of the electron backscatter diffraction pattern.
The centre lines of the Kikuchi bands correspond to the projection of the diffracting planes on the phosphor screen. Hence, each Kikuchi band can be indexed by the Miller indices of the diffracting crystal plane which formed it. Each point on the phosphor screen corresponds to the intersection of a crystal direction with the screen. In particular, the intersections of the Kikuchi bands correspond to the intersection of zone axes in the crystal with the phosphor screen. These points can be labelled by the crystal direction for the zone axis.

The true symmetry of the crystal is shown in the diffraction pattern. For example, four-fold symmetry is shown around the [001] direction by four symmetrically equivalent <013> zone axes.
The width, $w$, of the Kikuchi bands close to the pattern centre is given by:

$$w = 2L\theta = \frac{nL\lambda}{d}$$

where $L$ is the distance from the sample to the screen (camera length). Hence, planes with large $d$-spacings give thinner Kikuchi bands than closely-spaced planes. For example, the (200) planar spacing is wider than the (220) spacing, so the Kikuchi bands from (200) planes are narrower than those from (220) planes.
I. Electron beam induced current (EBIC)

Every incident electron knocks out hundreds or thousands of electrons from their atoms, leaving positively-charged holes behind. Normally, these electron-hole pairs recombine extremely quickly; however, if a bias voltage is applied to the specimen, they can be made to separate and form a current which can be measured. Using this current to form images is the basis of the electron beam induced current (EBIC) technique. Contrast in such images corresponds to differences in conductivity, electron-hole pair lifetime, and electron-hole mobility. These parameters make this a very powerful technique for studying semiconductors.

J. Cathodoluminescence

Cathodoluminescence (CL), already discussed in Ch.2, is a kind of fluorescence; but instead of being excited by photons (as in the case of XPS), it is excited by the incoming electrons. Electrons in the primary beam knock out outer electrons from atoms in the specimen, and the consequent relaxation mechanism causes the release of a photon of light. This mechanism is identical to that of EDS except that an outer electron is involved in CL and an inner one in EDS, resulting in high-energy photons (x-rays) in EDS and low-energy ones (visible light) in CL. Certain semiconductors and insulators will emit ultraviolet or visible light when bombarded with high-energy electrons. The intensity of these emissions is modified by the presence of impurities or defects like dislocations. This imaging technique is used extensively to examine defects in semiconductors like GaAs and AlN.

Because these photons can be ejected from anywhere within the sample interaction volume, spatial resolution is normally fairly poor in CL images, but it can be improved by using low voltages or narrower beams such as produced by a FEG.

The peak in photon energy detected corresponds to the band gap of the specimen, and the width of the peak decreases with temperature such that, at liquid helium temperatures, very small local alterations in band gap due to composition can be detected and related to levels of impurity < 0.01 ppm , making CL several orders of magnitude more sensitive than EDS or other x-ray techniques.

K. Sample preparation and charging

As there are no lenses below the specimen, there is a great deal of room in an SEM available to accommodate even large bulky samples; however, specimens must normally be conductive (or must be made conductive). When the surface is bombarded with electrons, both secondary and backscattered electrons will be produced, but in different amounts. At voltages of 1 – 5 keV, it is possible to produce as many (or more) electrons as are incident on the surface. At higher voltages, the yield is lower, leaving an excess of electrons on the surface. If these electrons were not conducted away, they would pile up on the surface and eventually start to deflect the incoming beam, thus distorting the image. This process is called charging. Of course, charging is not an issue when studying conductive samples like most metals, provided they are mounted so as to provide a conductive path to earth; however, insulators like ceramics, polymers, or
biological materials require coating with a thin (~10nm) conductive film (usually carbon or gold). Such coatings are easily created by *sputter coating*.

An alternative method of avoiding charging is to operate the microscope at low voltages where the electron yield is near unity, so that no charging occurs (the number of electrons arriving at the surface is balanced by the number of electrons leaving it).

![Graph showing total electron yield versus accelerating voltage](image)

If the electron yield is greater than unity (as it is for voltages between \( V_1 \) and \( V_2 \)), the sample charges positively, increasing the potential difference between the filament and the sample and thus shifting the beam voltage towards \( V_2 \). This self-compensation stabilises the beam voltage close to \( V_2 \).

Certain non-conducting specimens, particularly polymers and biological materials, may present additional problems like degradation by beam heating, radiation damage, or volatility.

**I. Environmental scanning electron microscopy (ESEM)**

Many wet or volatile samples are inappropriate for use in typical SEMs due to the high vacuum maintained in them; however, ESEMs maintain three separate vacuum conditions in the gun, column, and sample chamber. A high vacuum is kept around the gun, a lower vacuum is maintained in the column, and the sample chamber itself can be kept at 1-10 torr (0.01 - 0.001 atm).

Both secondary and backscattered electrons can be used in an ESEM; however, the Everhart-Thornley secondary electron detector cannot be used, as its very large bias voltage would cause electrical breakdown at the relatively poor vacuum of the specimen chamber. Gas-phase detectors analogous to the spectrometers used for WDS, have been developed for SEI. There is inevitably some loss of resolution at high pressures due to elastic scattering of the electrons off gas molecules. It is also possible to image uncoated non-conducting samples in an ESEM, as any accumulated charge is dissipated by electron-gas interactions.