Electron beam-specimen interaction

The interaction between the electron probe and a solid surface produces a variety of processes that can be understood by analysing the interactions among the beam electrons and the atoms of the matter and the different ways of energy transfer, until the thermal equilibrium or the reemission of the electron.

The choice of the working parameters (accelerating voltage, probe current, ...) depends critically upon

- what happens when the beam impinges the specimen surface
- the signals produced as effect of the beam-specimen interaction that can be used for images and other information (e.g., composition, ...)
A beam interacts with matter mainly in two ways:

**Elastic scattering**: it consists of a change of propagation direction of the electron without a substantial change of energy, due to an elastic collision in particular with the atomic nuclei of the specimen.

**Inelastic scattering**: it consists of the decreasing of energy without a significant change in the propagation direction of the electron, due to the inelastic collision with the atomic nuclei or with the atomic electron of the specimen.
The elastic scattering can be distinct in:

**Small impact parameter collision** and big deflection angle, with the target atomic nucleus (Rutherford scattering)

If the scattering deflection angle is > 90° the process of backscattering occurs and, if the interaction is near the surface, the backscattered electron energy is similar to the energy of the impinging electron.

**Multiple scattering**, with many large impact parameter collisions, and small deflection angles

It consists of a series of small changes in direction as effect of the Coulomb interaction with the nucleus shielded from the electrons.

The final result can be a change in direction with a big deflection angle and the subsequent emission from the surface as backscattered electron.

It energy can vary in a wide range which depends on the number of collisions and the transferred energy.
As a result of the collisions in the specimen, the probe electrons experience a lateral spread since the electrons are redirected from their original path.

As a consequence the specimen region interacting with the beam is not only a thin layer below the beam-surface contact area, but a bigger volume (of several μm).

The electrons which leave the specimen surface after the multiple elastic scattering (backscattered electrons - BSE) can give interesting information about the analysed sample.

In figure some paths of the beam electrons, which emerge from the surface after the interaction with the bulk sample, are in red.
The probability of elastic scattering increases strongly with the atomic number of the specimen (proportional to $Z^2$), because heavier atoms have a much stronger positive charge on the atomic nucleus, and decreases as electron energy increases (approximately as $1/E^2$).

A mathematical description of this process for an elastic scattering event at angles greater than a specified angle $\phi_0$ is

$$Q(> \phi_0) = 1.62 \times 10^{-20} (Z^2 / E^2) \cot^2 (\phi_0 / 2)$$

$Q(>\phi_0)$ is the cross section (cm$^2$) for elastic scattering (i.e., probability of elastic scattering)
In samples at low Z scattering events at low angle are more probable, and the electrons emerge from the surface after many collisions, so they penetrate more deeply.

In specimens at high Z the probability of great angle scattering increases and, consequently, the probability that the electrons escape from specimen also after a single event. The penetration depth decreases whereas the number of backscattered electrons increases.

Interaction volume as function of the energy of the incident electron beam and of the atomic number of the specimen
In the **inelastic scattering** the electrons transfer their energy to the specimen gradually by a series of processes and propagate through many atomic layers into the specimen before losing (almost) all their energy or escaping from the specimen surface.

The inelastic scattering gives rise to different signals, some of which are useful for the comprehension of the properties of the investigated sample:

- the electron can interact inelastically with the Coulomb field of the atomic nucleus so losing its energy as **Bremsstrahlung X-rays** (continuous emission)

- the electron can transfer kinetic energy to the electrons in the outer shells of the specimen atoms; the atomic electrons can gain enough energy to leave the atom (in a ionized state) or eventually to escape from the sample (**secondary electrons**)

- the electron can interact with the atomic electrons in the inner shells. These electrons escape from the atom; then the atom returns to its fundamental state through an electronic transition with the emission of **characteristic X-rays** or **Auger electrons**
Because of the variety of the involved physical processes, the model describing the energy loss of the beam electron during the interaction with the specimen is complex.

In 1930 Bethe described the rate of energy loss $dE$ with distance traveled $ds$ as:

$$-rac{dE}{dx} \left( \frac{keV}{cm} \right) = 2\pi e^4 N_0 \frac{Z}{A} \frac{\rho \ln 1.166E_m}{E_m}$$

Where:

- $e$ is the charge electron
- $\rho$ is the density (g/cm$^3$)
- $Z$ is the atomic number
- $A$ is the atomic weight (g/mole)
- $N_0$ is Avogadro’s number
- $E_m$ is the electron energy (keV) at any point in the specimen
- $J$ is the average loss in energy per event (eV)

J is given by the expression obtained though the best fit of the experimental data:

$$J = \left(9.76 + 58.8Z^{-1.19}\right)Z \quad (eV)$$

Whereas a simplified expression is

$$J = 11.5Z \quad (eV)$$
The relation \[ -\frac{dE_m}{dx} = 2\pi e^4 N_0 \frac{Z}{A} \frac{\rho}{E_m} \ln \frac{1.166E_m}{J} \]
is often used in the form \[ S = -\frac{1}{\rho} \frac{dE_m}{dx} \]

which represents the energy loss per length unit expressed in density unit. 

\( S \), mass stopping power, doesn’t depend on the density of the specimen.

Since \( \frac{Z}{A} \) is nearly constant and \( J \) increases with \( Z \)

\( \Rightarrow S \) decreases as the mean atomic number of the specimen.

In any case it’s not possible to obtain the electron energy value at a particular depth if the variations of its trajectory, due to the elastic collisions, are not considered.
The behaviour of energy loss (stopping power) as a function of the electron beam energy for different materials is reported in the following diagram:
How far a beam electron travel in the specimen?

By integrating the Bethe expression over the energy range (from the incident energy) an estimate of the total distance an electron can travel in the specimen is obtained:

\[
R = \int_{E_0}^{0} \frac{1}{dE/dx} \, dE \quad \text{Bethe range}
\]

Note: the Bethe range is measured along the trajectory regardless of direction changes caused by elastic scattering.

or similarly

\[
\rho R = \int_{E_0}^{0} \frac{1}{(dE/dx)(1/\rho)} \, dE = \int_{E_0}^{0} \frac{dE}{S} \quad \text{(mg/cm}^2\text{)}
\]
It results that the range increases when the energy increases and the atomic number decreases. The range can be used as a measurement of the interaction volume and of the lateral spread of the electrons in the specimen.

Practically during measurements the interaction volume can be reduced by decreasing the electron beam energy.
Electron spatial distribution in different materials
A comment......

The combined effect of the elastic and inelastic scattering events produces the spread of the beam electrons in a three-dimensional interaction volume of some micrometers.

By using beams with spots of about some nanometers, we would expect reasonably images with a resolution of the same order of magnitude.

In contrast the interaction volume has a size which is some orders of magnitude bigger than the spot size.

This influences the actual resolution of the SEM.

Etching experiment performed in a polymeric material (PMMA) evidencing the interaction volume.
Energetic spectrum of the electrons emitted from a specimen when an electron beam impinges normally the surface. It is possible to distinguish 3 different groups of electrons corresponding to 3 different energy ranges:

- **group 1** corresponds to the elastically (back)scattered electrons which escape from the surface without losing a significant quantity of energy.
- **group 2** presents a series of peaks due to those electrons which have lost discrete quantity of energy during inelastic scattering events near the surface; so they leave the specimen without losing too much energy. The position of the peaks (with respect to $E_0$) depends on the specimen material (not on $E_0$). The other region corresponds to the electrons that had undergone several elastic scattering events (at low scattering angles). The more energetic electrons come from the layers just below the surface, the less energetic ones come from the deeper layers.
- **group 3** includes the less energetic electrons and are classified as secondary electrons.
**Backscattered electrons:** are beam electrons whose trajectories have intercepted the surface, and which thus escape the specimen after one or more scattering events.

The backscattering process is influenced by the specimen atomic number:

- Low Z specimens allow the penetration of the electrons: multiple scattering events take place inside the sample and the number of backscattered electrons is low and it is due only to large angle scattering events.
- In high Z specimens the mean free path between two consecutive events is short so the probability of both single and multiple collisions near the surface increase.

Backscattering is quantified by the backscatter coefficient which is defined as:

\[
\eta = \frac{n_{BSE}}{n_B} = \frac{i_{BSE}}{i_B}
\]

where \( n_B \) is the number of beam electrons incident on the specimen and \( n_{BSE} \) the number of the backscattered electrons.
Variation of the backscattered electron coefficient as a function of atomic number for a value of beam energy of 30 keV and for normal incidence

Some important points about this plot should be noted:

1. The plot shows a general, monotonic increase in the backscattering coefficient with atomic number. The monotonic increase of $\eta$ versus $Z$ forms the basis for atomic number contrast (in SEM images) (also called compositional contrast or Z contrast)

2. The slope of $\eta$ versus $Z$ is initially steep, but decreases with increasing $Z$, becoming very shallow at high $Z$. The practical effect of this behaviour is that atomic number contrast between adjacent pairs of elements is strong at low atomic number and weak at high atomic number.
The curve of $\eta$ versus $Z$ can be conveniently fit with the expression

$$\eta = -0.0254 + 0.016Z - 1.85 \cdot 10^{-4} Z^2 + 8.3 \cdot 10^{-7} Z^3$$
Beam energy dependence of BSE

The size of the interaction volume is seen to be a strong function of beam energy. We might reasonably expect that the backscatter coefficient would also depend strongly on beam energy. However, experimental measurements reveal that this is not the case. As shown in figure there is only a small change, generally less than 10%, in the backscatter coefficient as function of beam energy for the range 5-50 keV, which spans the conventional SEM energy range.
Tilt dependence of BSE

If incidence is not perpendicular, the interaction volume becomes more asymmetrical and flattened under the surface.

If the backscatter coefficient is measured as a function of the tilt angle $\theta$, then a smooth, monotonic increase in backscattering with tilt is found. The slope of the curve is initially shallow, but increases with increasing tilt.
At very high tilt angles, which corresponds to grazing incidence, the value of $\eta$ tends toward unity. If $\eta$ versus $\theta$ is plotted for a range of elements, then at high values of $\theta$ the backscatter coefficients for all elements tend to converge.

An expression suggested by Arnal et al. (1969) gives the backscatter coefficient as a general function of $Z$ and $\theta$:

$$\eta(\theta) = \frac{1}{(1 + \cos \theta)^{9/\sqrt{Z}}}$$
This behavior of $\eta$ versus $\theta$ arises because of the dominant tendency of elastic scattering to be in the forward direction. That is, most elastic scattering events result in relatively small deviation angles, of the order of 5°, so the electron trajectories tend to continue in roughly the same direction after scattering as they were initially traveling.

When the beam is set normal to the specimen surface this tendency for forward scattering means that beam electrons tend to penetrate into the target. Only by the cumulative effects of any small scattering events, and much rarer large-angle events, do some of the electrons trajectories reverse direction and travel back toward the surface to escape as backscattered electrons.

If the tilt angle of the specimen surface is increased sufficiently, the geometry of the situation is such that, despite the tendency for forward scattering, electrons tend to travel near the surface and can escape with less total angular deviation, so $\eta$ increases.
Therefore $\eta$ increases with increasing $\theta$ since the penetration of beam electron along the normal direction decreases and the possibility of escaping from surface increases.

The monotonic rise of $\eta$ with $\theta$ forms the basis for an important component of the mechanism of topographic contrast in the SEM, by which the shape of objects is recognized.
Angular distribution of BSE

A second important consideration is the directionality of the backscattered electrons.

The dependence of $\eta$ upon $\theta$ gives the total number of BSE that emerge at a particular tilt angle of the surface without regard to the trajectories which the BSE follow out of the specimen.

In considering the performance of a BSE detector and in order to interpret the images obtained from the BSE signal, it is necessary to understand relationship of the detector position to the BSE trajectories emitted from the specimen.

Normal beam incidence (0° Tilt)
The angular distribution of BSE is defined relative to the normal to the surface through which the BSE emerge by an angle $\phi$.
The backscatter coefficient at the angle $\phi$, follows a distribution which approximates a cosine expression:

$$\eta(\phi) = \eta_n \cos \phi$$

where $\eta_n$ is the value measured along the normal direction.
Non-Normal beam incidence
When specimen is tilted, the angular distribution changes from the symmetric cosine law. The tendency for forward elastic scattering favors backscattering from the surface in the direction away from the incident beam.

The angular distribution thus becomes asymmetric, with a distinct lobe in the forward scattering direction and the asymmetry is most pronounced at high tilt angles.
Energy distribution of BSE

As the beam electrons travel within the specimen, the various processes of inelastic scattering reduce the electron energy at a rate of roughly 1-10 eV/nm. Individual BSE can follow trajectories which involve different distances of travel in the specimen before escaping. So the energy retained by each BSE depends on the history of the beam electron.

From the energy distribution, it’s possible to evidence several distinct characteristics:

- The energy distribution is a continuum, extending from the incident beam $E_0$ (i.e., a small fraction of beam electrons scatter elastically with a sufficiently large angle immediately upon entering the specimen and backscatter without any significant energy loss) to essentially zero energy (i.e., a small fraction of beam electrons travel so far in the specimen that they lose virtually all their incident energy and reach the surface just prior to being captured by the specimen upon reaching an energy equivalent to the equilibrium thermal energy of the specimen electrons).
the energy distribution shows two distinct regions:

- The uppermost region, denoted I, is the high-energy hump of the BSE that have lost less than 50% of their early energy \( E_0 \) (for most targets of intermediate and high atomic number, the majority of BSE will be found in this region).

- Region II is the broad, gradually decreasing tail, representing those beam electrons that travel progressively greater distances, losing progressively more energy within the specimen prior to backscattering.

the energy distribution shows a peak value, region I, which becomes more distinct for higher Z targets. The peak relative energy \( E_0/E \) increases from approximately 0.8 for copper to 0.95 for gold and the distribution for a light element (such as carbon), is extremely wide with only a broad maximum and no distinct peak.

the energy distribution also depends on the angle above the surface at which the distribution is measured.
In the energy distribution of all the electrons (measured over the range from $E_0$, the incident beam energy, down to 0 keV) most of the contribution is due to BSE, which give rise to region 1 (high peak energy) and 2 (tail from intermediate to low energy).

If this low-energy tail is extrapolated to zero energy, the yield of BSE falls to zero, as expected.

However, at very low energy, below 50 eV, it is found experimentally that the number of electrons emitted from the specimen increases sharply to a level much greater than the expected contribution from BSE alone.

This increase in emitted electrons forms the region 3 and is due to the phenomenon of secondary electron emission.
Secondary electrons are loosely bound outer shell **electrons from the specimen atoms** which receive sufficient kinetics energy during inelastic scattering of the beam electrons to be ejected from the atom and set into motion.

The SE thus created will propagate through the solid, and some will intersect the surface and escape.

SE are defined purely on the basis of their kinetic energy; that is, all electrons emitted from the specimen with an energy less than 50 eV (arbitrary choice) are considered as SE.

Although a tiny fraction of the BSE are obviously included in this energy region and are thus counted as SE, their inclusion in the definition of SE introduces only a negligible effect.

The total SE coefficient $\delta$ is given by:

$$\delta = \frac{n_{SE}}{n_B} = \frac{i_{SE}}{i_B}$$

where $n_B$ is the number of SE emitted from a sample bombarded by $n_{SE}$ beam electrons, and $i$ designates the corresponding currents.
SE have energy less than 50 eV and their energy distribution presents a peak in the range 2-5 eV. Moreover, although there is a probability different from zero, but very small, that some electrons could have energy higher than 50 eV, more than 90 % of SE are emitted with energy less than a 10 eV.
SE yield with primary beam energy

The SE coefficient $\delta$ generally rises as the beam energy is lowered. The table provides experimental data for $\delta$ for Al and Au over a range of energy:

<table>
<thead>
<tr>
<th>Element</th>
<th>5 keV</th>
<th>20 keV</th>
<th>50 keV</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>0.4</td>
<td>0.1</td>
<td>0.05</td>
</tr>
<tr>
<td>Au</td>
<td>0.7</td>
<td>0.2</td>
<td>0.10</td>
</tr>
</tbody>
</table>

Maximum value of $\delta$ (as function of energy) can be lower or higher than unity and it depends on the specimen working function. Generally, it is $>1$ in semiconductors and insulators whereas it is $\leq 1$ in metals.

The decrease of $\delta$ after the maximum is due to the fact that, even if the produced SE increase with beam electron energy, the interaction volume and the diffusion depth increase, too.

The secondary electrons are thus produced at greater depths and, consequently, the probability of escaping from surface decreases.

SE can reach the surface from depths of 2-20 nm in metals and 5-50 nm in insulators.
Relative contributions of $SE_1$ and $SE_2$

In samples there are two sources of SE whose origin are illustrated schematically in the figure. Incident beam electrons generate secondary electrons ($SE_1$) upon entering the sample. Backscattered electrons generate secondary electrons ($SE_2$) while exiting the sample.

In both the processes electrons escape from the surface but only those produced just below the surface can be detected (due to their very low energy).
Anyway, $SE_2$ are distributed over a larger surface.

Experimentalists have been able to distinguish the relative contribution of $SE_1$ and $SE_2$ to the total secondary emission by experiments on thin foils, where the BSE component can be effectively eliminated.

From experiments it results that current associated to $SE_2$ is about 4-6 times higher than current associated to $SE_1$. However, since the current density associated to $SE_2$ is much lower than that associated to $SE_1$, the resolution in morphologic analyses is limited to $SE_1$. 
Spatial profile of SE
The total secondary electron coefficient $\delta$ consists of two components $\delta_1$ and $\delta_2$, corresponding to SE$_1$ and SE$_2$:

$$\delta = \delta_1 + \eta \delta_2$$

The backscatter coefficient $\eta$ multiplies $\delta_2$ because the flux of energetic beam electrons traveling back to the entrance surface is reduced by $\eta$.

The values of $\delta_1$ and $\delta_2$ are not equal, that is an incident beam electron and an average backscattered electron are not equally efficient in generating SE. In general, the ratio $\delta_2/\delta_1$ is of the order of 3-4, that is BSE are significantly more efficient at generating SE than beam electrons.
Specimen composition dependence of SE

Compared to the behaviour of BSE, whose yield increases nearly monotonically with the atomic number of specimen, the SE coefficient is relatively insensitive to Z.

As an example, for a beam energy of 20 keV, $\delta$ has a value approximately 0.1 for most elements.

Two noteworthy exceptions to this average value at 20 keV are carbon, which is anomalously low ($\delta = 0.05$) and gold, which is anomalously high ($\delta = 0.2$).
Specimen tilt dependence of SE

As the angle of the specimen tilt is increased, $\delta$ increases following a relationship reasonably well described by a secant function:

$$\delta(\theta) = \delta_0 \sec \theta$$

where $\delta_0$ is the value of $\delta$ found at $\theta = 0^\circ$ (normal incidence)
The origin of secant function behaviour can be understood with the simple argument illustrated in figure.

Consider that all SE are created along the primary beam trajectory within a distance of $R_0$ of the surface can escape.

When the specimen is tilted to a value of $\theta$, the length $R$ of the primary electron path within a distance $R_0$ from the surface increases as:

$$R = R_0 \sec \theta$$

Since production and escape of SE are proportional to path length, the secondary electron coefficient increases following a secant relationship.

Moreover, since backscattering increases with tilt angle $\theta$, also $SE_2$ contribute to the increase in $\delta$ at high tilt angles.
Images produced with the SE signal will reveal something termed the “edge effect” [6]. Edges and ridges of the sample emit more SEs and thus appear brighter in the image.
Angular distribution of SE

For normal incidence of primary beam, SE emission follows a cosine distribution ($\cos \phi$), where $\phi$ is measured with respect to the surface (similar behaviour of BSE)

When the specimen is tilted, the angular emission behaviours of BSE and SE differ.

For BSE, the angular distribution becomes asymmetric and peaked at a forward scattering direction, for SE the angular distribution remains a symmetric cosine function of $\phi$.

This behaviour is a result of the fact that SE are generated isotropically by the primary electrons regardless of tilt.