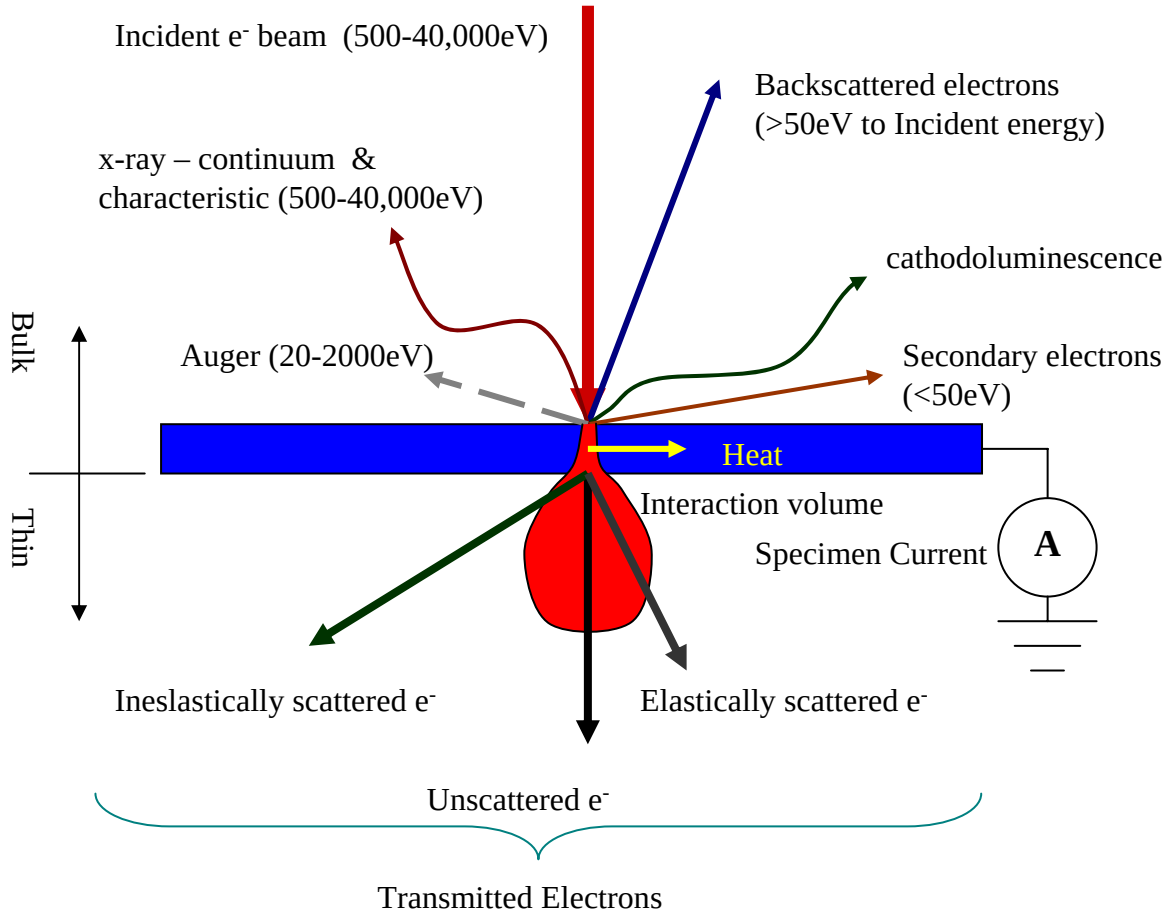
Electron Beam – Specimen Interaction

The interaction of a high energy electron beam with the specimen will produce various effects resulting in a range of signals being emitted. The incident electrons interact with specimen atoms and are significantly scattered by them (rather than penetrating the sample in a linear fashion).

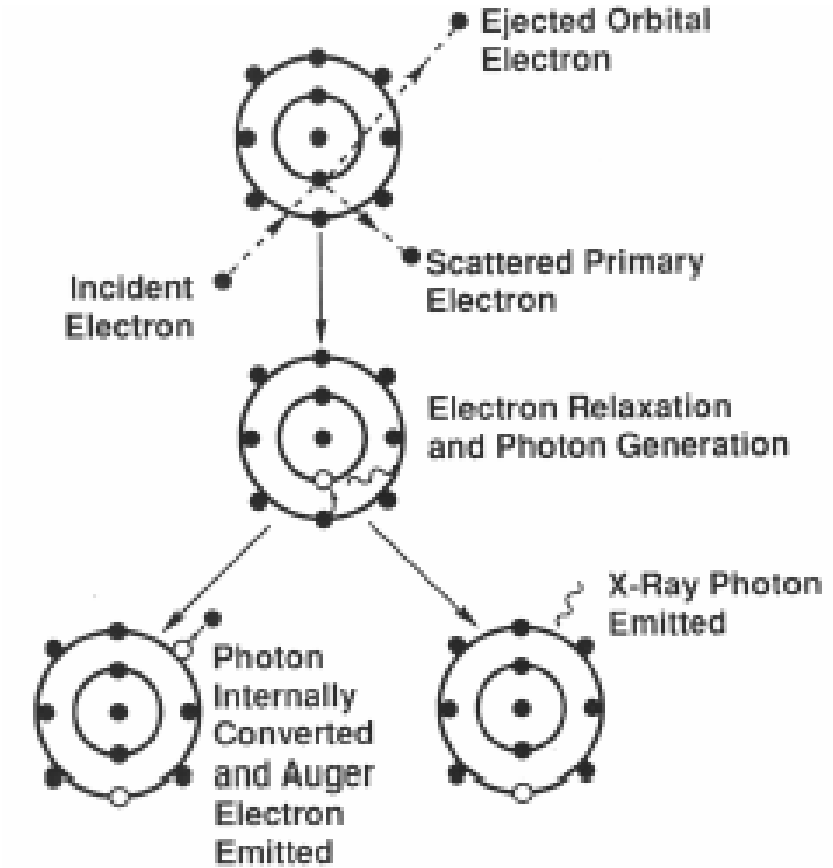
Most of the energy of an electron beam will eventually end up heating the sample (phonon excitation of the atomic lattice); however, before the electrons come to rest, they undergo two types of scattering: ***elastic and inelastic.***



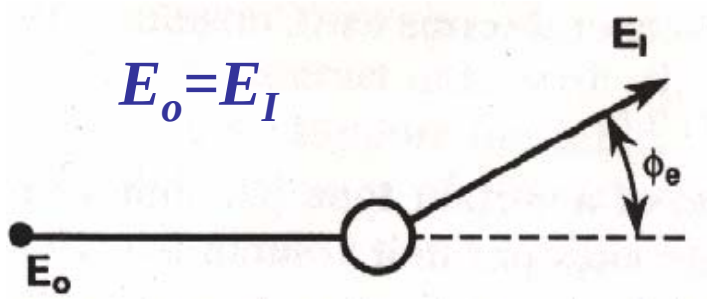
The electrons-specimen interaction can provide information on:

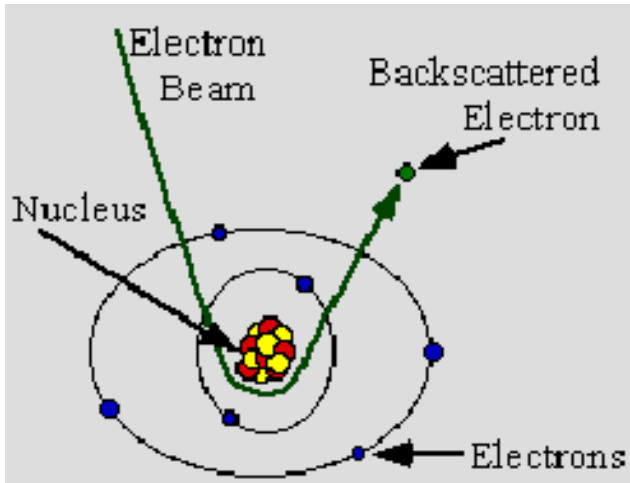
- Specimen composition
- Topography
- Crystallography
- Electrical Potential
- Local Magnetic Field

Secondary electrons: The inelastic interaction is responsible for the production of secondary electrons and it takes place between the incident electrons and the outer (not strongly bound) electrons of the atoms. These outer electrons can be ejected from the atom with energies lower than 50eV. If these “secondary” electrons are produced near the surface, and its energy is higher than the surface energy (~6eV) then, they can escape to the vacuum and reach the detector.

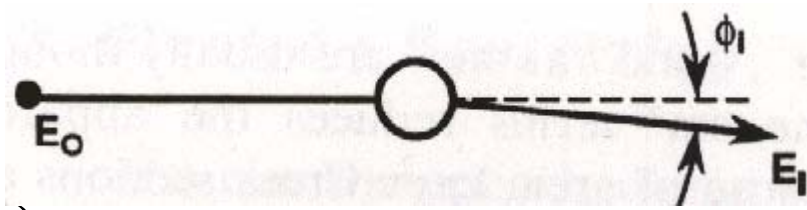


In **elastic scattering**, the electron trajectory changes, but its kinetic energy and velocity remain essentially constant (due to large differences between the mass of the electron and nucleus). This process is known as electron backscattering (although later we will confine the term "**backscattered electrons**" to those scatter out of the sample).



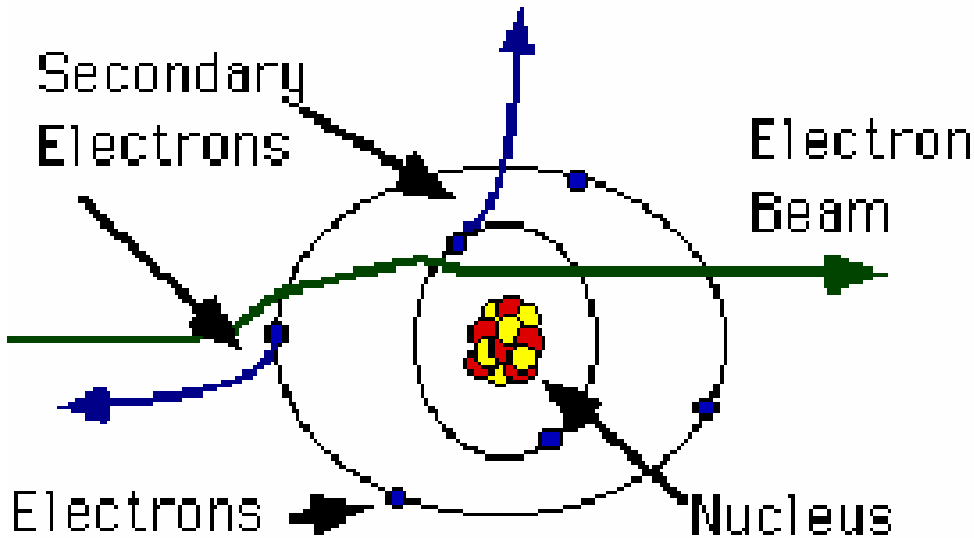


In **inelastic scattering**, the trajectory of the incident electron is only slightly perturbed, but energy is lost through interactions with the orbital electrons of the atoms in the specimen. Inelastic interactions produce diverse effect including:



$E_o > E_I$

- Secondary electrons
- phonon excitation (heating)
- cathodoluminescence (visible light fluorescence)
- continuum radiation (bremsstrahlung or "braking" radiation)
- characteristic x-ray radiation
- plasmon production (secondary electrons)
- Auger electron production (ejection of outer shell electrons)



Interaction Volume

The combined effect of the elastic and inelastic interactions is to limit the penetration of the beam into the solid. The region of interaction between the solid and the beam is known as the interaction volume.

Etching plastic can directly reveal the interaction volume for the low atomic number materials, but it can not do the same for intermediate or high atomic number materials, such as metals.

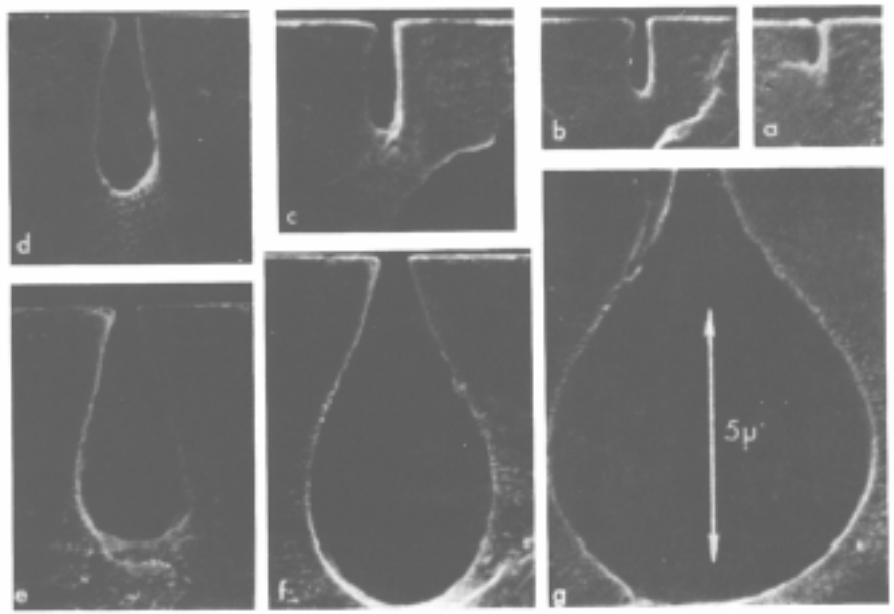


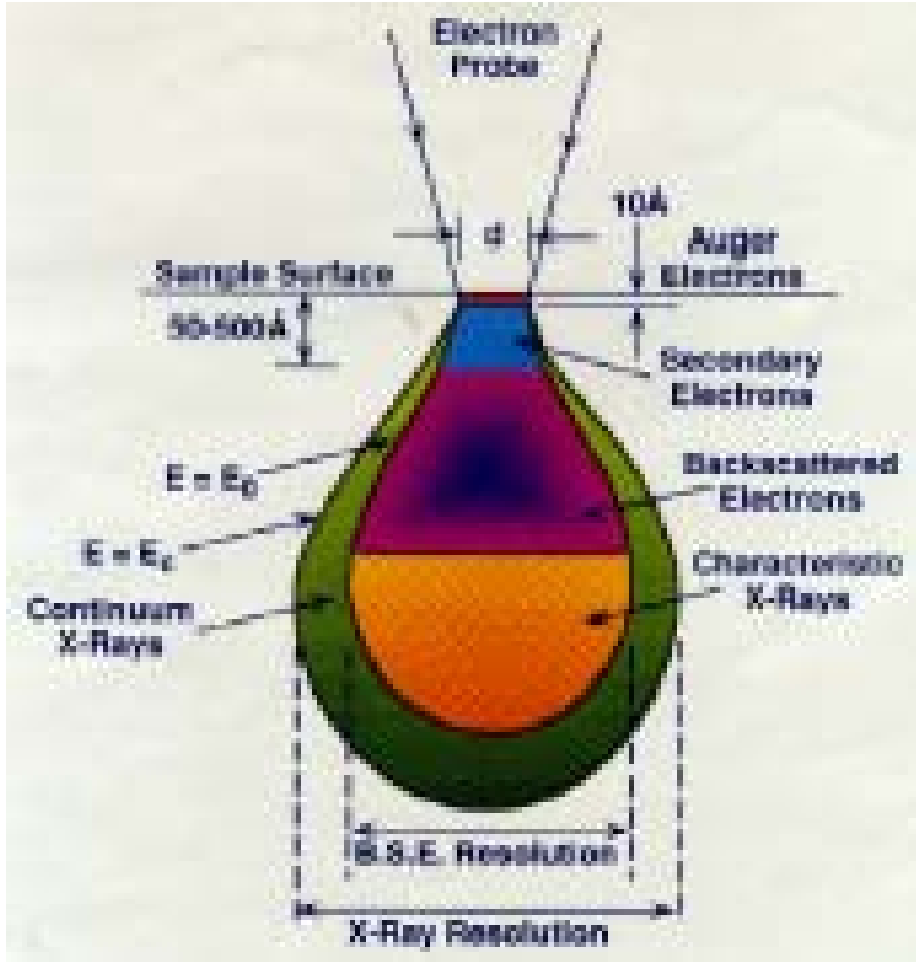
Figure 3.5. Direct visualization of the electron volume in polymethylmethacrylate. In (a) through (g), the electron dose is the same, but the etching time is increased progressively to reveal successively lower energy deposition (radiation damage) levels (from Everhart *et al.*, 1972).

Monte Carlo electron trajectory simulation provides an indirect method to visualize the interaction volume for any material. A large number of trajectories, typically of 10,000 to 100,000 electrons in the beam must be calculated to achieve statistical significance.

Two major factors control which effects can be detected from the interaction volume.

A) A beam of electrons lose energy as they traverse the sample due to interactions with it and if too much energy is required to produce an effect, it will not be possible to produce it from deeper portions of the volume.

B) The degree to which an effect, once produced, can be observed is controlled by how strongly it is diminished by absorption and scattering in the sample.



For example, although secondary and Auger electrons are produced throughout the ***interaction volume***, they have very low energies and can only escape from a thin layer near the sample's surface.

Volume of Excitation

The size and shape of the interaction volume is limited by two factors:
(1) energy loss through inelastic interactions
(2) electron loss or backscattering through elastic interactions.

The resulting excitation volume is a hemispherical to jug-shaped region with the neck of jug at the specimen surface.

The depth of electron penetration of an electron beam and the volume of sample with which it interacts are a function of its angle of incidence, the magnitude of its current, the accelerating voltage, and the average atomic number (Z) of the sample.

Electron penetration generally ranges from 1-5 µm with the beam incident perpendicular to the sample. The ***depth of electron penetration*** (***x***) is approximately (Potts, 1987, p.336):

$$x\ (\mu m) = \frac{0.1\ E_o^{1.5}}{\rho}$$

where E_o = accelerating voltage (keV), and ρ = density (g/cm^3)

The ***width of the excited volume*** (***y***) can be approximated by (Potts, 1987, p. 37):

$$y\ (\mu m) = \frac{0.077\ E_o^{1.5}}{\rho}$$

where E_o = accelerating voltage (keV), and ρ = density (g/cm^3)

Better results are obtained using Monte Carlo approximations

The interaction volume is influenced by:
(a) the beam energy;

(b) the atomic number of the solid;

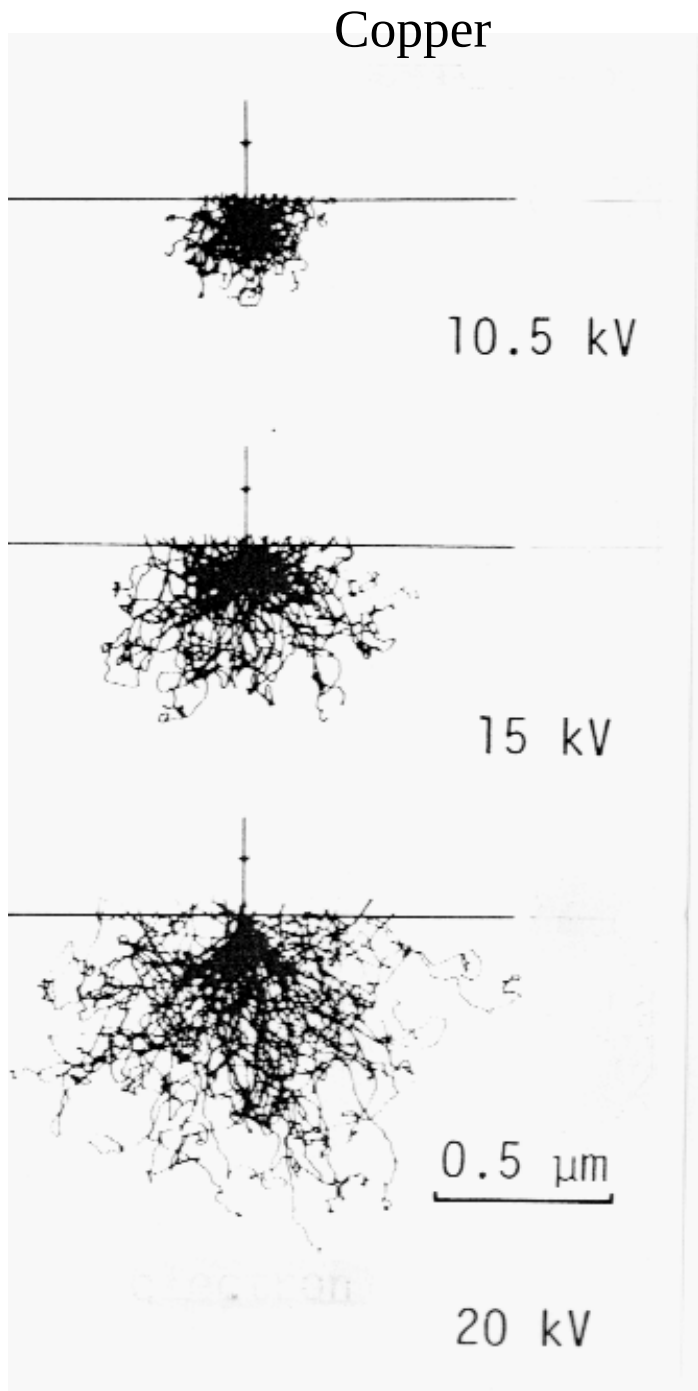
(c) surface tilt and

(d) density of the solid.

Influence of beam energy on the interaction volume:

(1) As the beam energy is increased, the electron beam can penetrate to greater depths.

(2) There is no a significant change in the shape of the interaction volume with beam energy



Influence of the atomic number of the solid in the interaction volume:

(1) In specimens of high atomic number the electrons undergo more elastic scattering per unit distance and the average scattering angle is greater.

(2) The electron trajectory in the high atomic number materials, tends to deviate out of the initial direction of travel more quickly.

(3) the shape of the interaction volume is greatly affected by the atomic number of the specimen

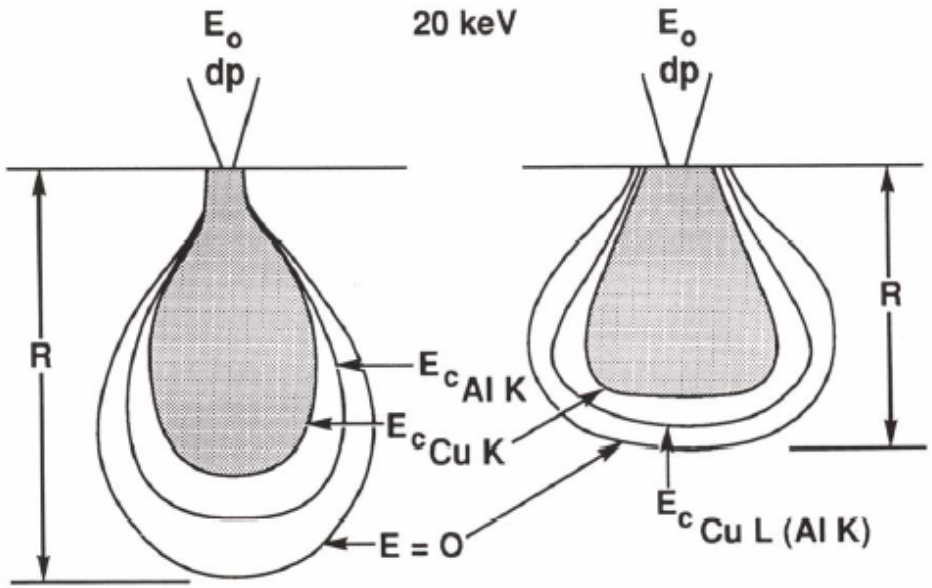
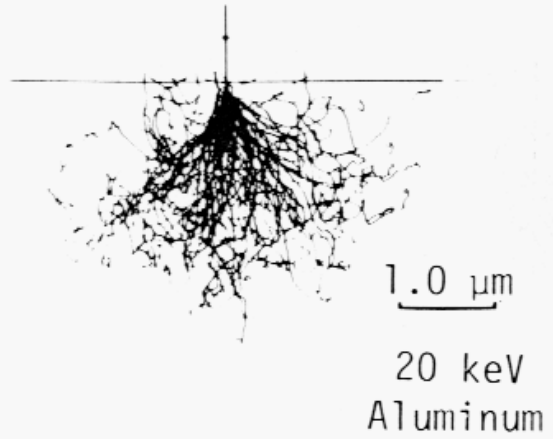
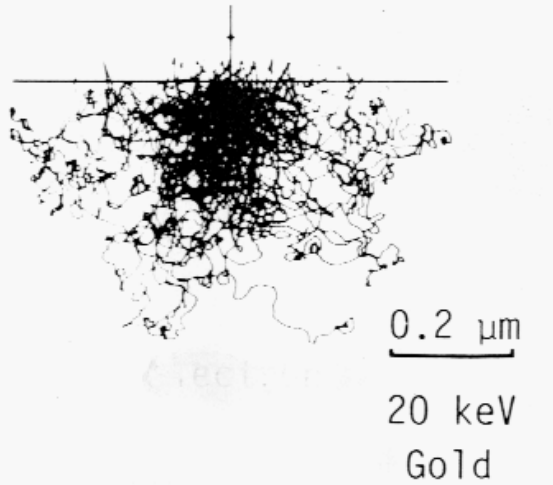
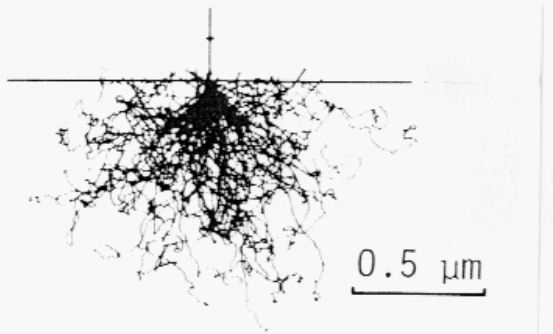


Figure 3.42. Comparison of x-ray production regions from specimens with densities of 3 (left) and 10 g/cm^3 (right).







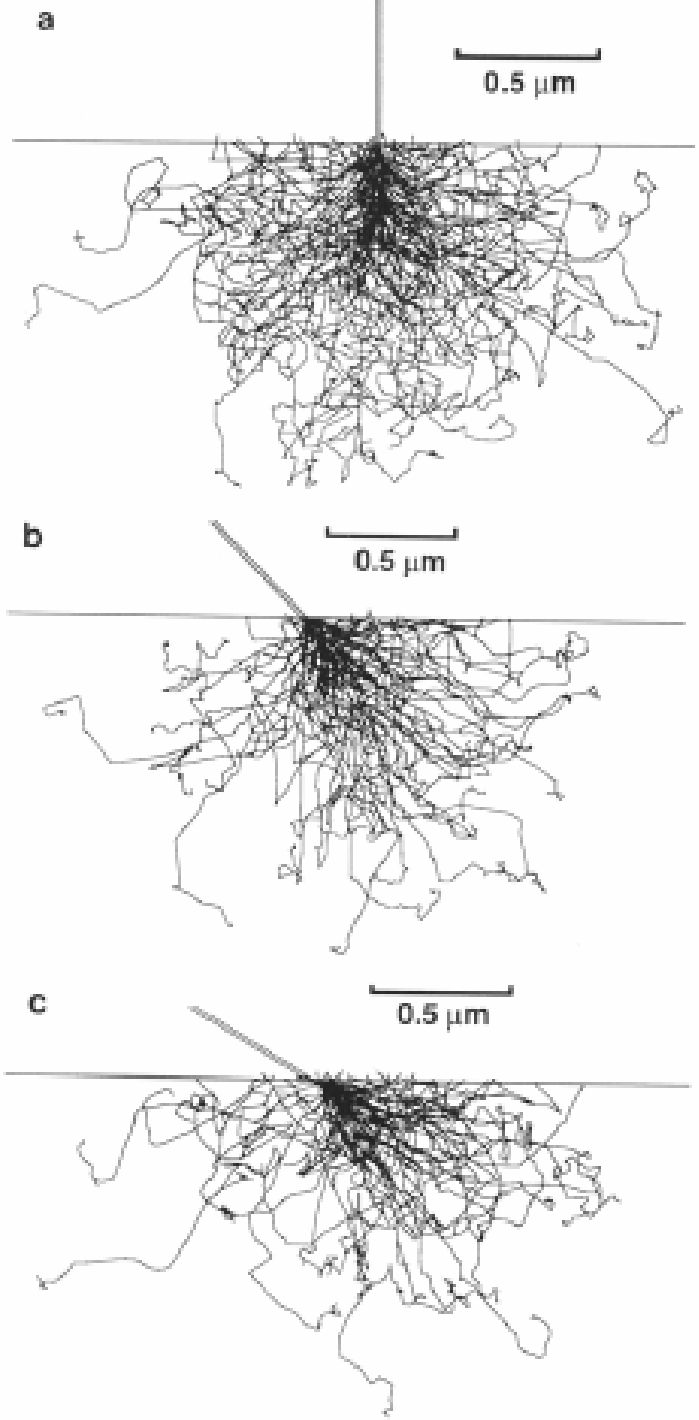


Figure 3.10. Monte Carlo electron-trajectory simulations of the interaction volume in iron at E_0 = 20 keV for various tilts: (a) 0° tilt, (b) 45° tilt, (c) 60° tilt.

Influence of the tilt on the interaction volume:

As the angle of tilt of the specimen surface increases, the interaction volume becomes smaller and asymmetric.

Elastic Scattering - Backscattered Electrons

Scattering of beam electrons produces backscattered and transmitted electrons as the strong electrical field of the specimen's atomic nuclei deflects them; no additional electrons are produced from the sample.

Where transmitted electrons pass completely through the material after interacting with it, backscattered electrons are ejected from the top surface of the specimen at high angles. Transmitted and backscattered electrons can have energies from about 50 eV up to the accelerating voltage ($\boldsymbol{E_o}$). The number of backscattered electron produced from a material may be quantified by its backscattering coefficient, η_b.

$$\eta_b = \frac{n_{BSE}}{n_B} = \frac{number\_of\_electrons\_backscattered}{number\_of\_beam\_electrons\_incident\_on\_specimen}$$

This coefficient depends strongly on a sample's average atomic number, $\boldsymbol{Z}$. Neglecting the effects of $\boldsymbol{E_o}$, the following equation yields a good approximation for the coefficient, η_b (Love & Scott, 1978):

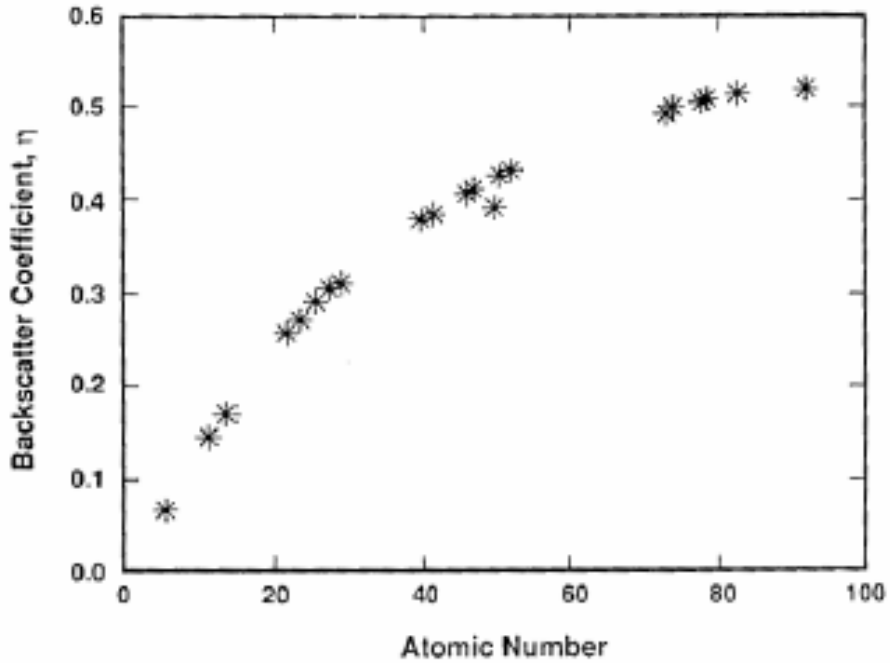


Figure 3.13. Backscattered-electron coefficient η as a function of atomic number at $E_0 = 20$ keV (data of Heinrich, 1966a).

$$n_b = n_{b20}\,[1 + a \ln(E_0/20)]$$

where,

$$a = -0.11128 + 3.0289 \times 10^{-3} Z - 1.5498 \times 10^{-5} Z^2$$

$$n_{b20} = -5.23791 \times 10^{-3} + 1.5048371 \times 10^{-2} Z - 1.67373 \times 10^{-4} Z^2 + 7.16 \times 10^{-7} Z^3$$

Average ***Z*** is calculated using the weight fractions (***w***) of each element:

$$Z = w_1 z_1 + w_2 z_2 + w_3 z_3 \ldots + w_n z_n$$

Thus, for SiO_2, with 0.4674 Si and 0.5326 O by weight:

$$Z_{SiO_2} = 0.4674 \times 14 + 0.5326 \times 8 = 10.8044$$

and n_b***= 0.142***. About 14.2% of incident electrons are backscattered.
About ***48%*** of incident electrons are backscattered by a tungsten target (***Z = 74***), whereas only about ***14%*** are produced by one of sodium (***Z=11***).

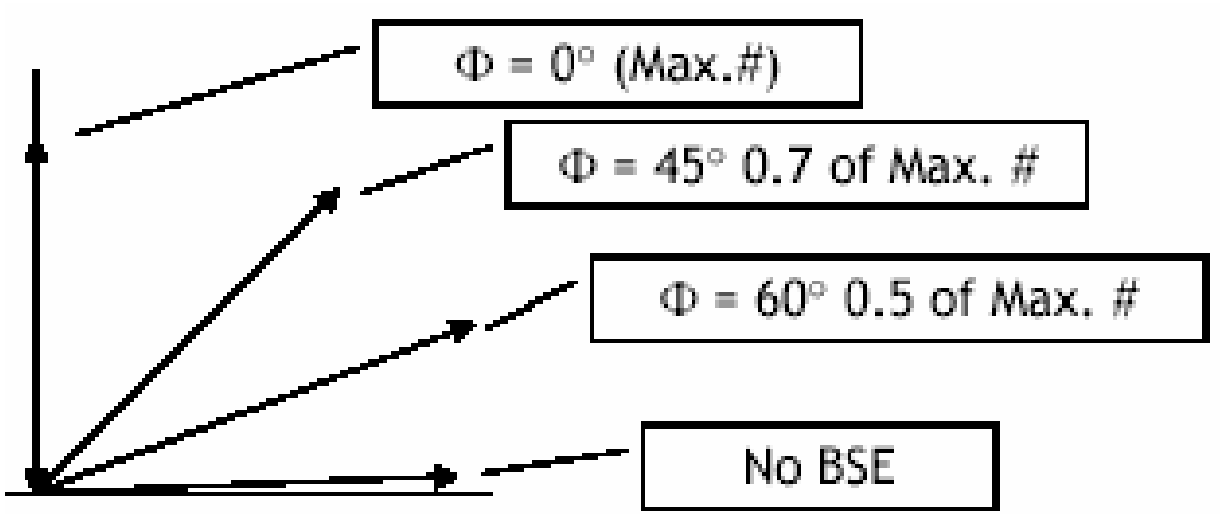
BSE respond to composition allowing for atomic number or compositional contrast.

BSE atomic number dependence: the BSE coefficient increases with increasing atomic number.

BSE beam energy dependence: the BSE coefficient does not depend strongly on beam energy.

BSE tilt dependence: the BSE coefficient increases with tilt as the electrons can escape the surface with less total angular deviation (at very high angles, which correspond to grazing incidence, the value of n_b tends toward unity)

BSE angular distribution: is defined relative to the normal to the surface. It refers to the number of BSE escaping the surface at different angles (ϕ) relative to the normal to the surface.



BSE Lateral Spatial Distribution: beam electrons can travel significant distances laterally from the beam impact point before escaping as backscattered electrons. Consequence: decrease in the capability to resolve fine features.

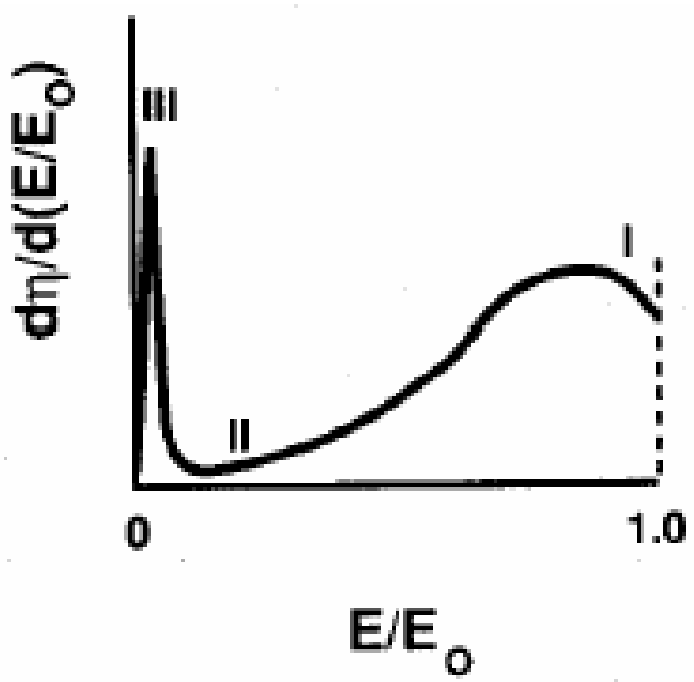
BSE Sampling Depth: the BSE signal can respond to subsurface details of the specimen's structure (as the beam energy decreases the BSE signal becomes much more surface-sensitive).

BSE –SE Energy Distribution:

Region I: represents the high-energy hump of BSE that have lost less than 50% of E_0 (most BSE retain at least 50% of the incident beam energy).

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

Region III: at very low energy, below 50eV, it is found experimentally that the number of electrons emitted from the specimen increases sharply. This is due to the phenomenon of secondary electron emission.



Inelastic Scattering – Secondary Electrons

Secondary Electrons: are electrons of the specimen ejected during inelastic scattering of the energetic beam electrons.
SE are defined purely on the basis of their kinetic energy as all the electrons emitted from the specimen with an energy less than 50eV (an arbitrary choice).

Secondary Electron Coefficient:

n_{SE} = number of secondary electrons
n_B = number of beam electrons incident on the specimen.

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

SE Energy Distribution: SE are produced as a result of interactions between energetic beam electrons and weakly bound conduction-band electrons in metals or outer-shell valence electrons in semiconductors and insulators. More than 90% of the SE are emitted with less than 10 eV of energy (peak at 2-5 eV)

SE specimen composition dependence: in general insensitive to atomic number

SE beam energy dependence: the SE coefficient increases as the beam energy is lowered. The escape depth of SE is small (a few nanometers), so all of the SE created by the beam electrons at greater depths are lost.

SE specimen tilt dependence: as the angle (specimen tilt) is increased, the secondary electron coefficient increases (escape depth).

Cathodoluminescence and Heat

Cathodoluminescence is the emission of visible light from a sample during electron bombardment and may be observed using the light optics of the microprobe. Resolution is about 1000 Å (the same as for X-rays; the diameter of the excitation jug).

This effect is produced in materials with at least some semiconductor properties when incident electrons knock a photoelectron into the "conduction band" of a material resulting in a positively charged "hole." The free electrons recombine with the holes to produce radiated light or heat in the sample.

Cathodoluminescence can be an intrinsic property of a material (e.g., scheelite) or the result of luminescent centers produced by trace impurities (often Mn or rare-earth elements) in a non-luminescent host (e.g., calcite). Minerals that are luminescent include K-feldspar, zircon, fluorite, diamond, apatite, and benitoite (blue); quartz (orange to blue); calcite (red-orange due to Mn^{2+} or Pb^{2+} activator); willemite (green); and enstatite (red due to Mn^{2+} activator or blue with no activator) and dolomite (blue).

Significant amounts of **heat** are produced with a sample because electron excitation of X-rays is not very efficient. Many low energy continuum photons and low-energy inelastically scattered electrons do not escape the sample and their energy is transformed into higher vibrational energies of the bonds (heat). The maximum temperature rise for a material can be expressed as:

$$\Delta T = \frac{4.8\, E_0\, b_i}{C_t\, d_0}$$

where,

E_0 = accelerating voltage (keV)
b_i = beam current (μA)
C_t = thermal conductivity (W/cm·K)
d_0 = beam diameter (μm)

		1 μm diameter spot			5 μm diameter spot		
Material	C_t	**5 nA**	**10 nA**	**25 nA**	**5 nA**	**10 nA**	**25 nA**
Epoxy	0.002	180	360	900	36	72	180
Mica	0.005	72	144	360	14	29	72
Obsidian	0.014	26	51	128	5	10	26
Zircon	0.042	9	17	43	2	3	9
Calcite	0.05	7	14	36	1	3	7
Quartz	0.10	4	7	18	0.7	1	4
Kyanite	0.17	2	4	11	0.4	0.8	2
Periclase	0.46	0.8	2	4	0.2	0.3	0.8