Laboratory of Microscopy

Introduction to Scanning Electron Microscopy (SEM)

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Introduction to Scanning Electron Microscopy (SEM)

What is it?

What information does it give?

What samples can be analysed?

What Instrument has to be used?

How is this instrument made?

Introduction to Scanning Electron Microscopy (SEM)

What is it?

Scanning Electron Microscopy is a technique that produces **images** of suitable samples **by scanning** their surface with a **focused beam of electrons**.

The interaction between the **sample atoms** and the electron beam produces **various signals** that contain information about the **sample morphology** and **surface topography** and **composition** of the sample.

Important Keywords/Keyphrases

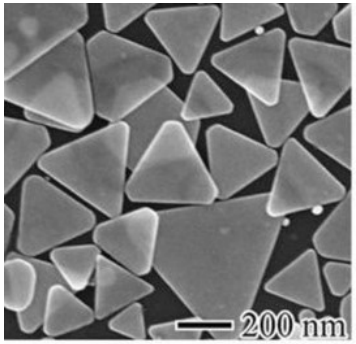
images **focused beam of electrons** **scanning** **various signals** **sample morphology**

surface topography **composition**

We shall deal with each of these keywords/keyphrases to gain the basic understanding of the technique.

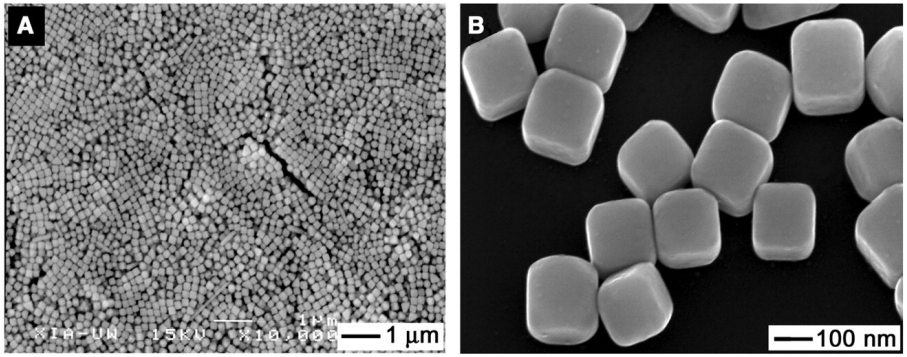
Introduction to Scanning Electron Microscopy (SEM)

images



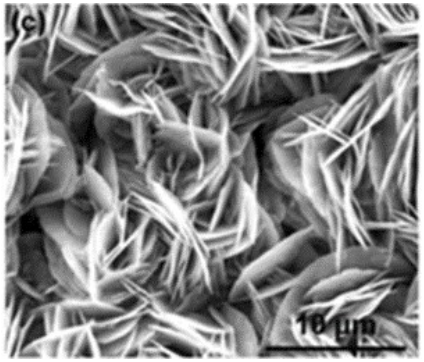
Ag nanoparticles

small 2014, 10, 1430–1437



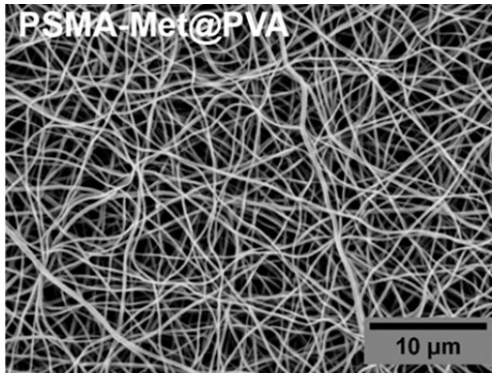
(A) Low- and (B) high-magnification SEM images of silver nanocubes

Science 2002, 298, 2176-2179



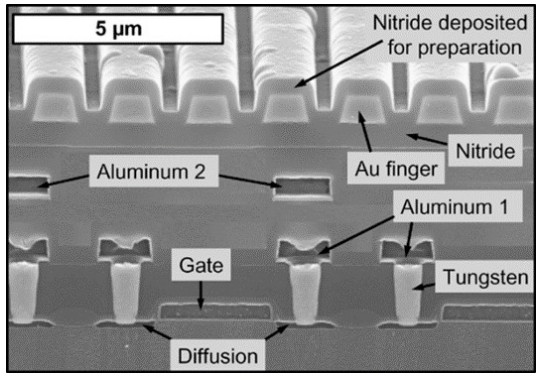
Ag nanosheets

ACS Appl. Mater. Interfaces 2012, 4, 2752–2756



Polymer membrane

ACS Appl. Polym. Mater. 2024, 6, 10436–10451



Integrated circuit

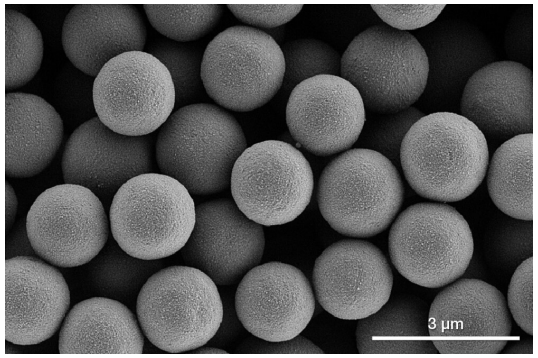
IEEE Journal of Solid-State Circuits, 2004, 39, 2438-2445

Please note the scale bars!

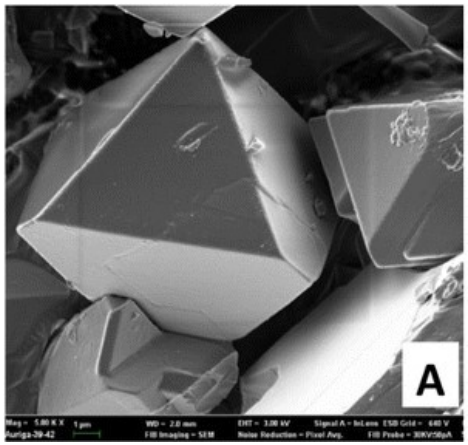
SEM allows analyses of species or features in the 100 nm-10 µm regime.

SEM Images

Morphology means shape



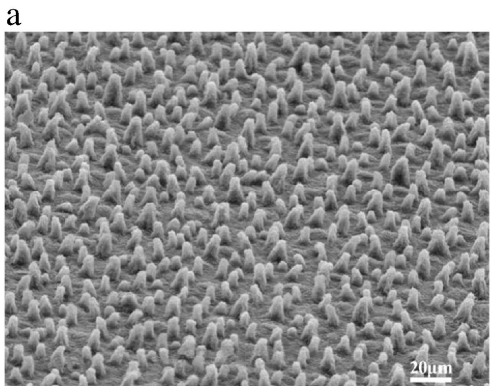
Polymer beads

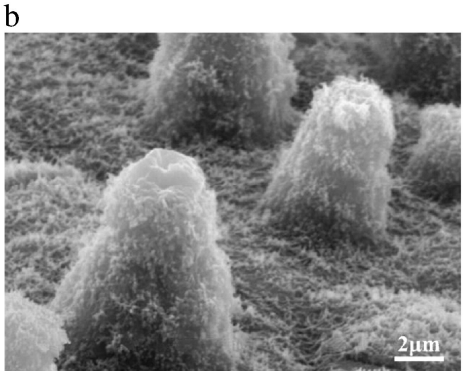


$Cu(BTC)_2$ BTC = benzen-1,3,5-tricarboxylate

RSC Adv., **2023**, *13*, 20816-20829

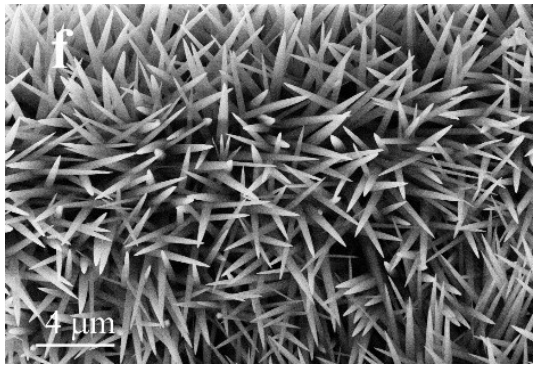
Topopgraphy means ‘surface features’





SEM images of the natural lotus leaves

Thin Solid Films **2011**, *519*, 5523–5527



(Ni/Co_2O_4) nanoneedles on carbon filter paper

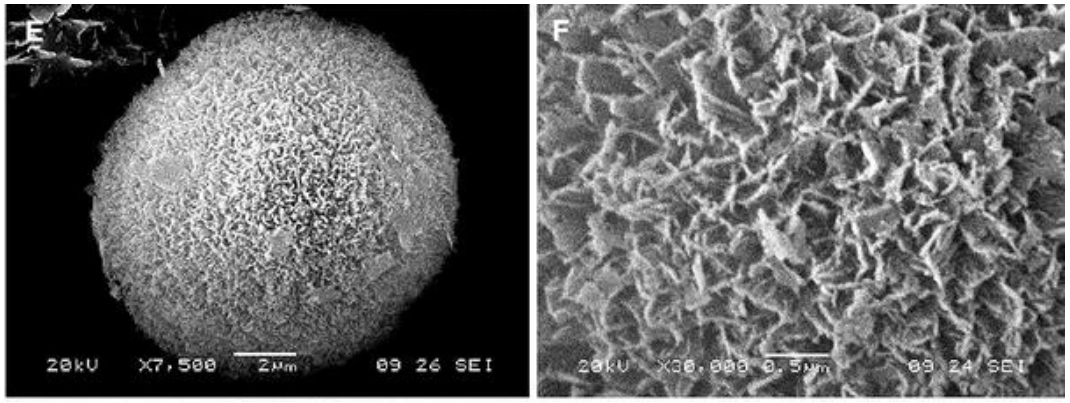
Langmuir **2025**, *41*, 582–596

SEM Images

SEM imaging can be used to achieve magnifications from 10x to 300000x or, for high end instrument up to 1000000x or higher.

For comparison, a TEM can also achieve magnifications in the range 100x to 1000000 and beyond, **but** cannot deal with **thick samples**

For comparison, an optical microscope can achieve magnifications in the range 10x-1000x.



porous microspheres of calcium phosphates

J Porous Mater (2009) 16:683–689

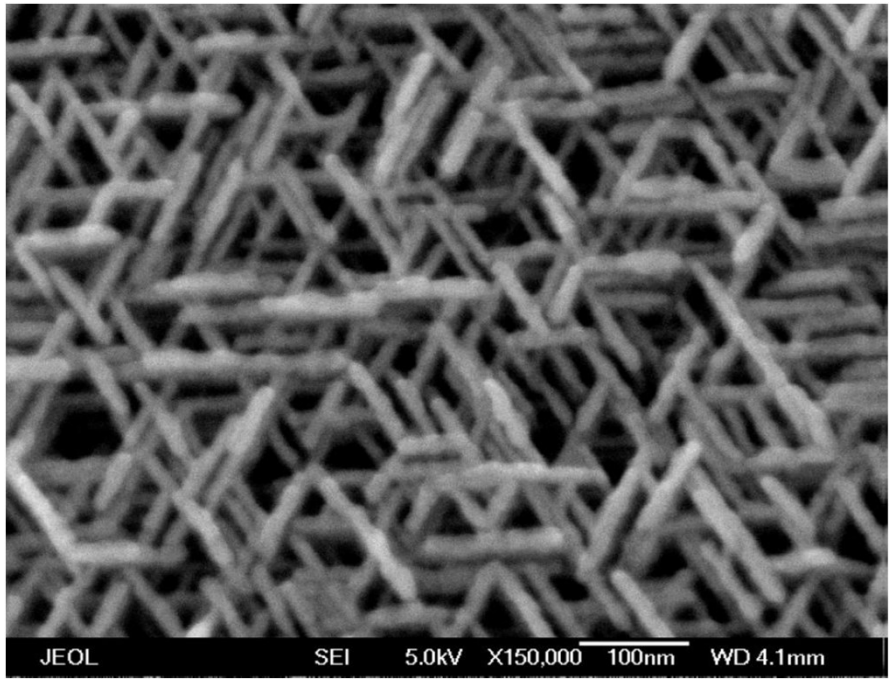
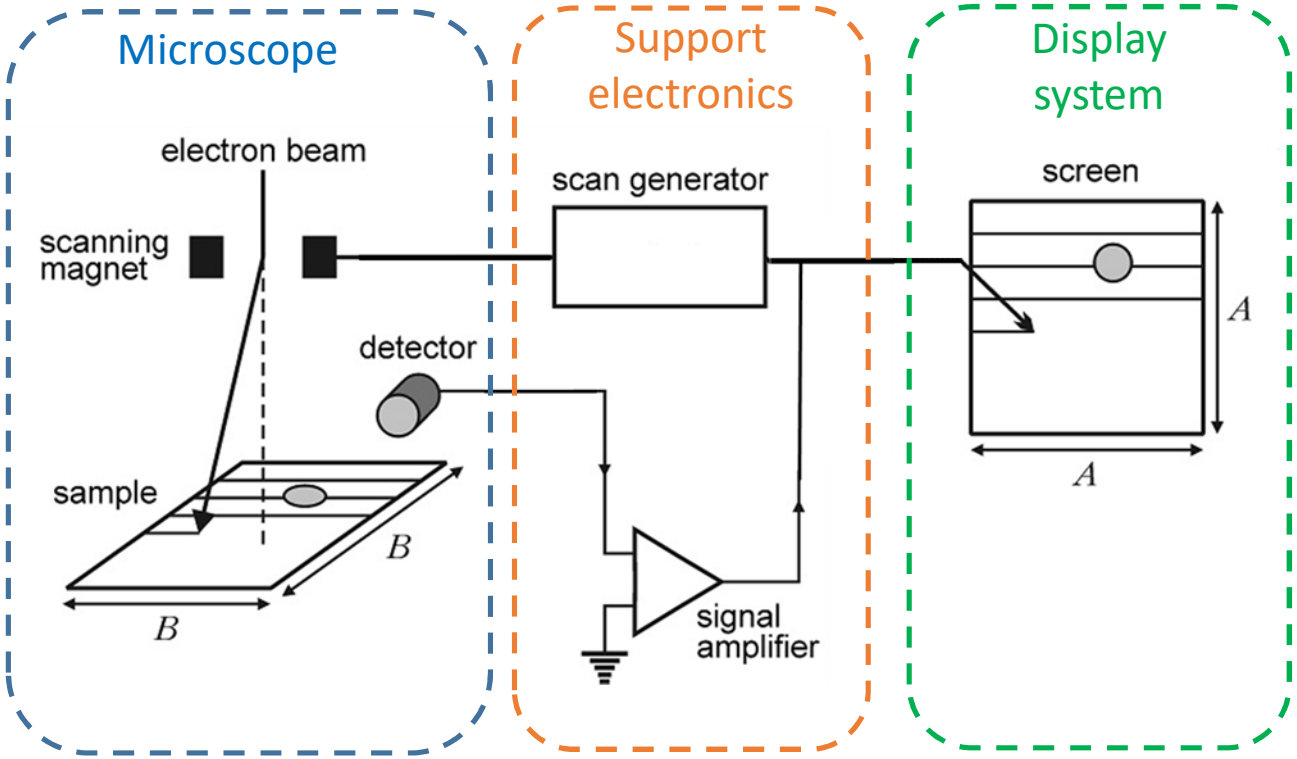


image of Hematite

SEM Images

SEM has some advantages over other EM techniques, as you saw from the preceding slides, for the moment an important one is its ability to image a relatively large surface area of the sample and the possibility to image bulk materials (not just thin films).

SEM Images are **reconstructed** Image is reconstructed pixel by pixel using the 'signals' coming from the sample being scanned by the electron beam. There is no such things as CCD or CMOS cameras for image acquisition in SEM!



Schematic representation of image reconstruction in SEM

SEM Images

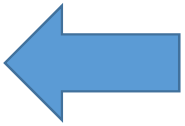
various signals

Fundamental to SEM is the **understanding** of the interaction between an electron beam and an analyte specimen. In particular what events are triggered when an electron beam hits the sample surface.

Several different events may occur, **each one of which brings information** on the sample probed:

Formation of Backscattered electrons (BSE);
Emission of Secondary electrons (SE);
X-ray emission;

Emission of Auger electrons;
Cathode luminescence;
Specimen current;
Transmitted electrons;
…



We shall deal only these three

Secondary electrons

Ideal for gaining information on topography at high resolution.

Backscattered electrons

Useful when relative atomic density information as well as topographical information is needed

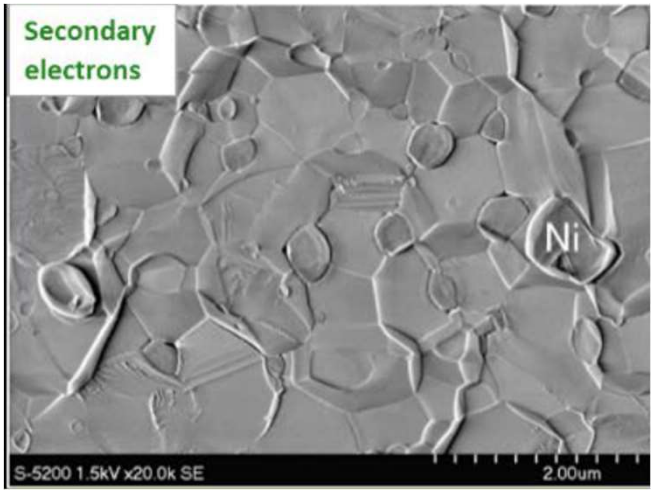
X-ray emission

Information on composition.

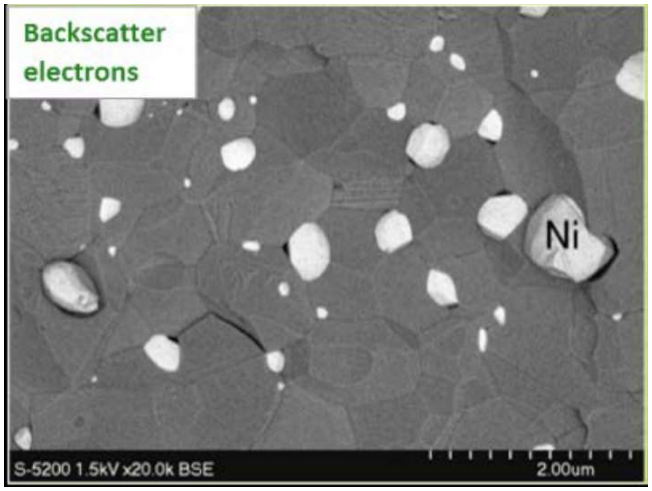
SEM Images

What does ‘density information as well as topographical information is needed’ mean?

In these images, Ni is clearly visible using BSE for image reconstruction while it is difficult to see using secondary electrons for image reconstruction. Topographic features are evident in both images.



Ni on aluminium oxide



Ni on aluminium oxide

This is an example of ‘compositional contrast’ or ‘atomic number contrast’, one of the ways in which contrast can be introduced in SEM images, we will se more afterwards.

Interaction of the electron beams with materials

Some definitions

The region of the analyte that interact with the electron beam is called '**interaction volume**'

The interaction volume may be much larger than the lateral size of the electron beam, this is known as the **spot-size**.

The **spot-size** is in the order of about 10-20 nm but the size of the interaction volume may reach a few microns.

The reason why the interaction volume is larger than the **spot-size** is that electrons in SEM penetrate the sample to a certain depth according to their kinetic energy (due to the accelerating voltage used to produce the beam).

The accelerating voltage is typically around 20-40 kV, (λ 0,01-0,006 nm) frequently lower, rarely higher.

The wavelength of the electrons is given by: $\lambda = \frac{h}{\sqrt{2meE}}$ Remember De Broglie equation: $\lambda = \frac{h}{p}$

The interaction volume is the region of the material where the signals detected (and used for image reconstruction) are generated.

Secondary electrons, Backscattered electrons and X-rays all come from the interaction volume.

Interaction of the electron beams with materials

Inelastic scattering events

"Inelastic" scattering refers to a variety of physical processes that act to progressively reduce the energy of the beam electron by transferring that energy to the specimen atoms through interactions with tightly bound inner-shell atomic electrons and loosely bound valence electrons. These energy loss processes include ejection of weakly bound outer-shell atomic electrons (binding energy of a few eV) to form secondary electrons; ejection of tightly bound inner shell atomic electrons (binding energy of hundreds to thousands of eV) which subsequently results in emission of characteristic X-rays;

Elastic scattering events

Simultaneously with inelastic scattering, "elastic scattering" events occur when the beam electron is deflected by the electrical field of an atom (the positive nuclear charge as partially shielded by the negative charge of the atom's orbital electrons), causing the beam electron to deviate from its previous path onto a new trajectory,

While the average elastic scattering event causes an angular change of only a few degrees, deviations up to 180° are possible in a single elastic scattering event.

Interaction of the electron beams with materials

Energy loss of the incident beam

While energy is lost in these inelastic scattering events, the beam electrons only deviate slightly from their current path. The energy loss due to inelastic scattering sets an eventual limit on how far the beam electron can travel in the specimen before it loses all of its energy and is absorbed by the specimen.

Although the various inelastic scattering energy loss processes are discrete and independent, Bethe (1930) was able to summarize their collective effects into a "continuous energy loss approximation":

$$dE/ds\,(\mathrm{eV/nm}) = -7.85\,(Z\rho/AE)\ln(1.166\,E/J)$$

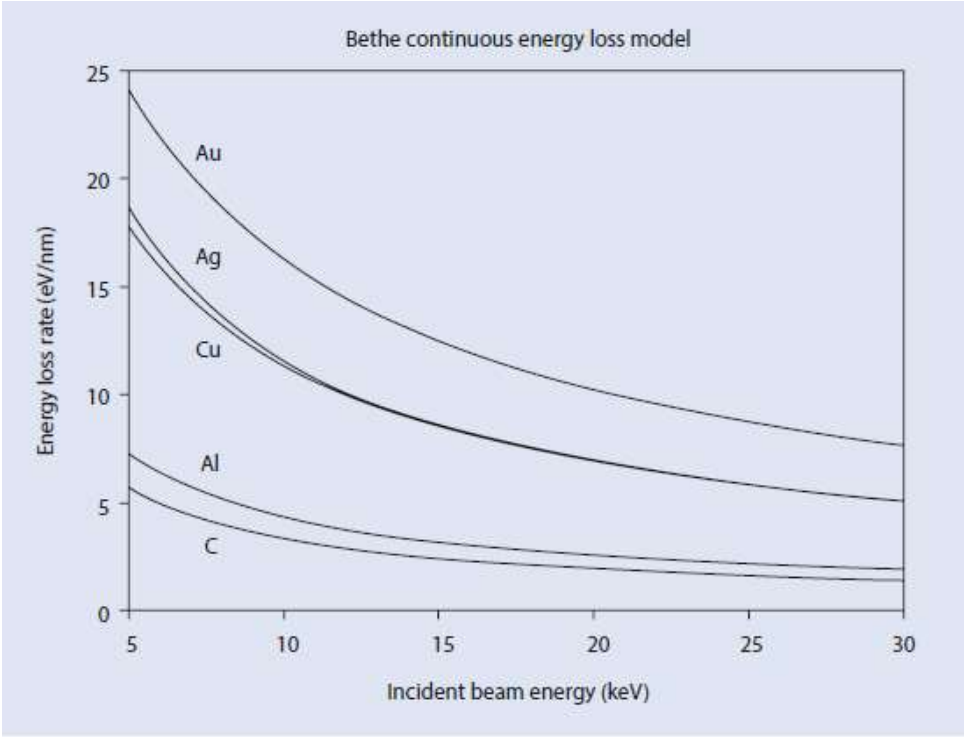
where E is the beam energy (keV), Z is the atomic number, ρ is the density (g/cm^3), A is the atomic weight (g/mol), and J is the "mean ionization potential" (keV) given by

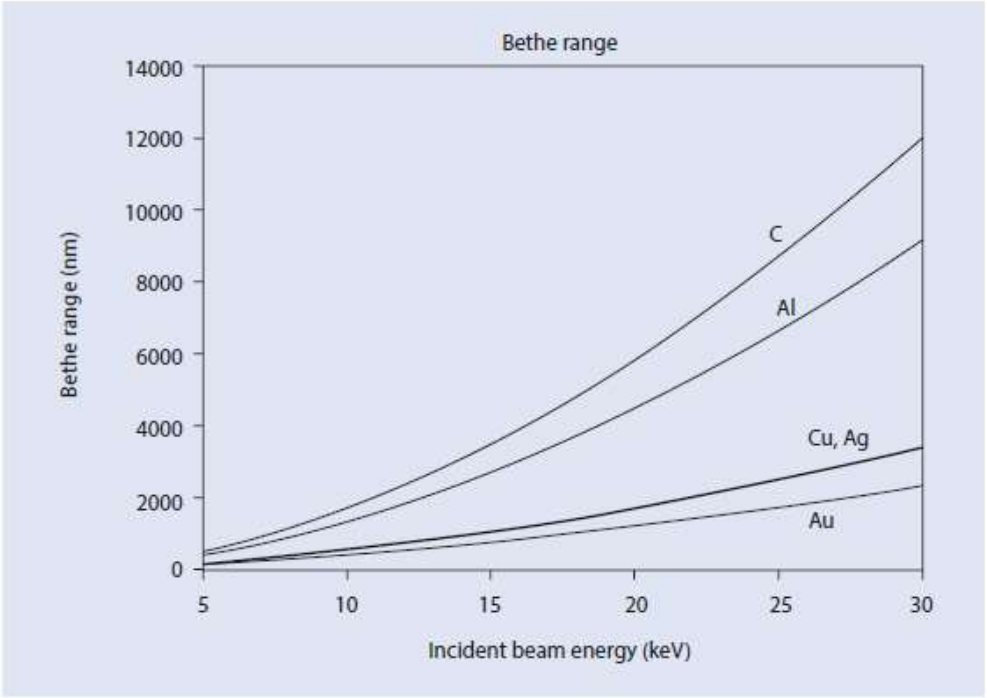
$$J(\mathrm{keV}) = \left(9.76Z + 58.5Z^{-0.19}\right) \times 10^{-3}$$

Interaction of the electron beams with materials

Energy loss of the incident beam

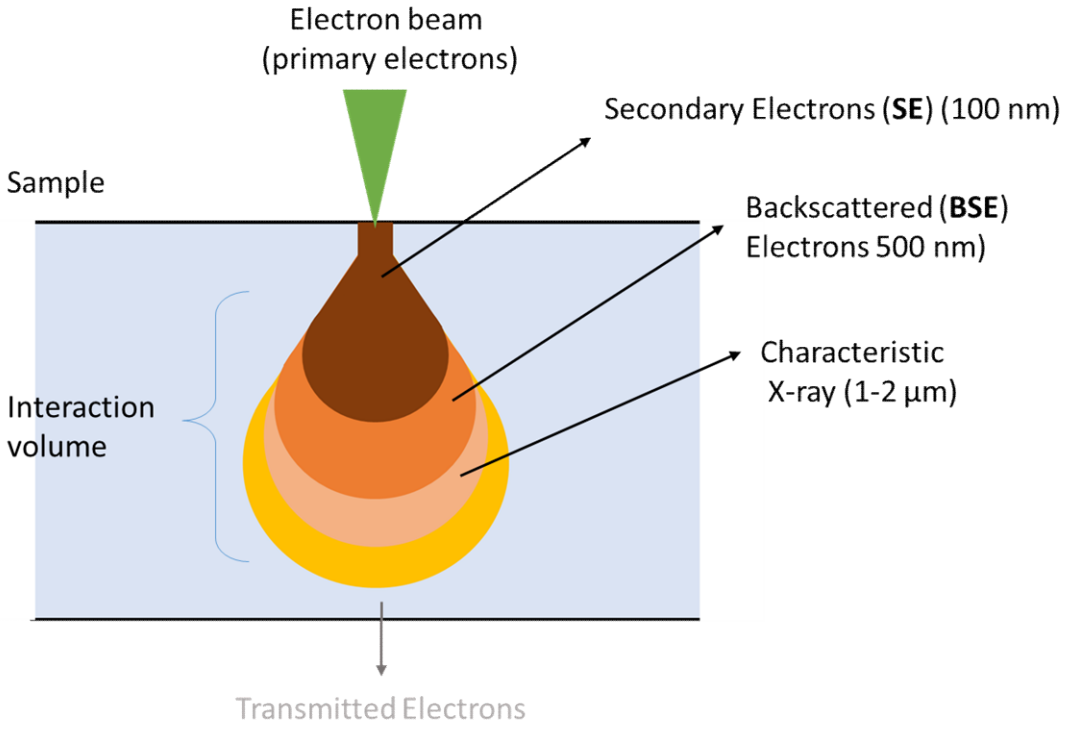
Below, the Bethe expression is plotted for several elements (C, Al, Cu, Ag, Au) over the range of “conventional” SEM operating energies, 5–30 keV. This figure reveals that the rate of energy loss dE/ds increases as the electron energy decreases and increases with the atomic number of the target. An electron with a beam energy of 20 keV loses energy at approximately 10 eV/nm in Au, so that if this rate was constant, the total path traveled in the specimen would be approximately 20,000 eV/(10 eV/nm) = 2000 nm = 2 µm, this is known as ‘Bethe range’





Interaction of the electron beams with materials

The signals from the interaction volume come from **different depths**



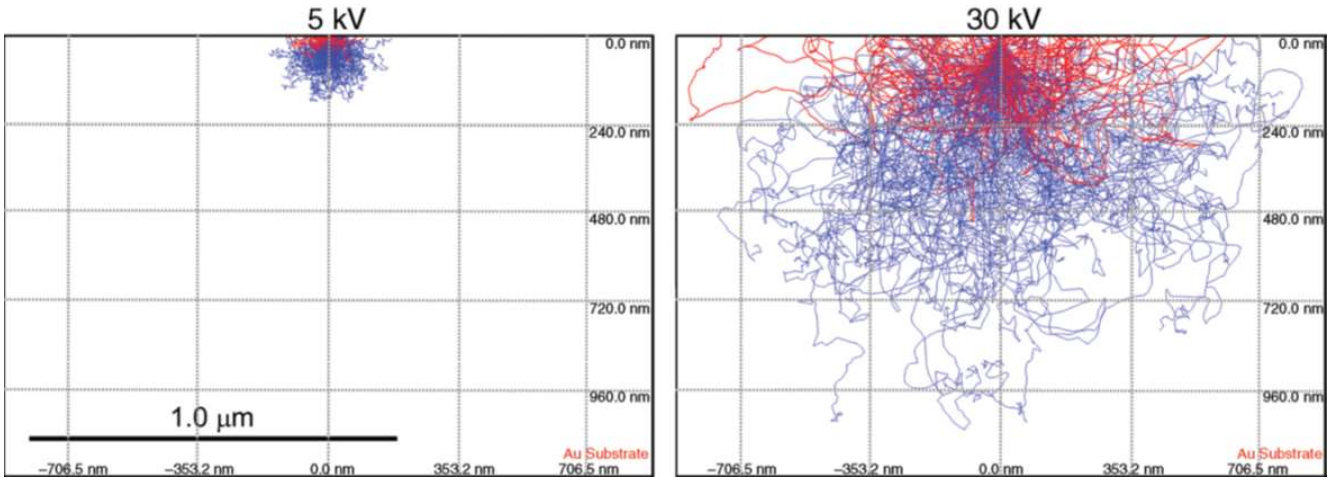
The electrons of the electron beam are known as primary electrons.

The primary electrons entering a sample will hit other particles, electrons or atomic nuclei. This **changes the trajectory** of the primary electron but also its energy. This is known as scattering.
It is the components of the scattering events (not all events involve electrons) that can be detected.

Interaction of the electron beams with materials

The Interaction Volume

The size of the interaction volume is dependent on the acceleration voltage of the primary electrons and the atomic number of the atoms close to the surface. Heavy atoms decelerate the beam more than light elements and therefore reduce the interaction volume.

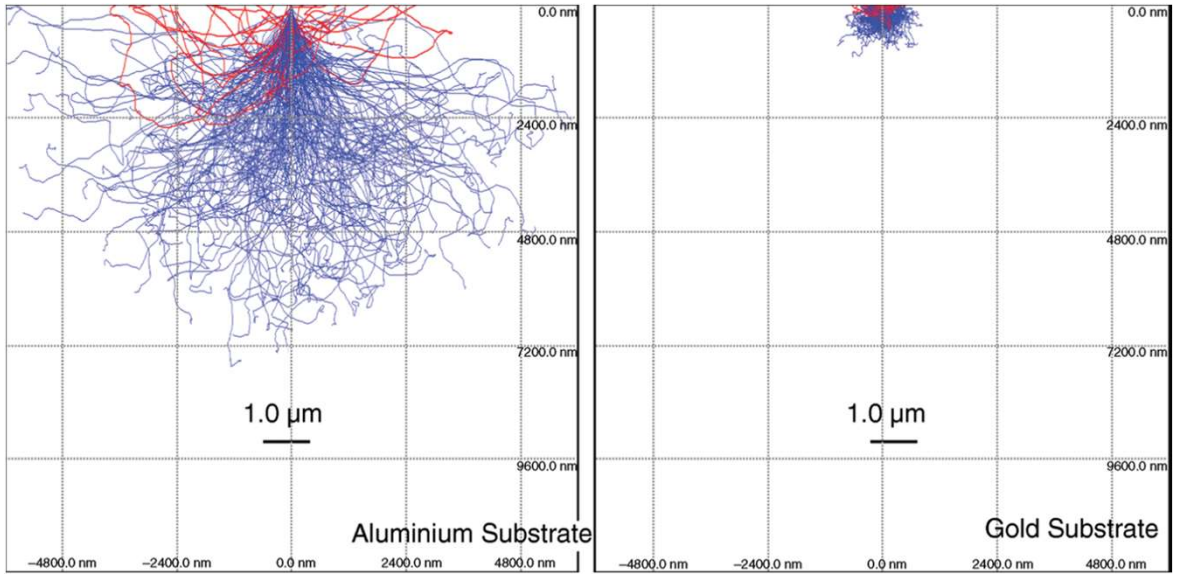


Monte Carlo simulation of electron trajectories in Au, at an incident electron energy of 5 kV and 30 kV and using a 10 nm probe diameter. The red trajectories are for electrons which eventually escaped the sample. The scale bar is identical for both calculations.

Interaction of the electron beams with materials

The Interaction Volume

The interaction volume also depends strongly on the atomic number Z, and its size is drastically reduced with increasing Z



simulation of electron trajectories in Al and Au, at an incident electron energy of 30 kV and using a 10 nm probe diameter. The red trajectories are for electrons which eventually escaped the sample.

Interaction of the electron beams with materials

The size of the Interaction Volume

Kanaya and Okayama (1972) developed a range equation that considered both inelastic and elastic scattering to give an estimate of the interaction volume as the radius of a hemisphere centered on the beam impact point that contained at least 95% of the trajectories:

$R_{K\text{-}O}$ is known as the Kanaya and Okayama range

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

where A is the atomic weight (g/mol), Z is the atomic number, ρ is the density (g/cm3), and E0 is the incident beam energy (keV).

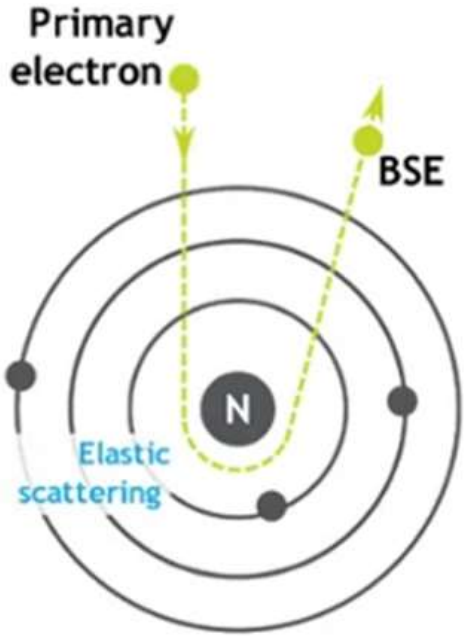
Table 1.1 Kanaya–Okayama range

	5 keV (nm)	10 keV	20 keV	30 keV (μm)
C	450 nm	1.4 μm	4.5 μm	8.9 μm
Al	413 nm	1.3 μm	4.2 μm	8.2 μm
Fe	159 nm	505 nm	1.6 μm	3.2 μm
Ag	135 nm	431 nm	1.4 μm	2.7 μm
Au	85 nm	270 nm	860 nm	1.7 μm

Interaction of the electron beams with materials

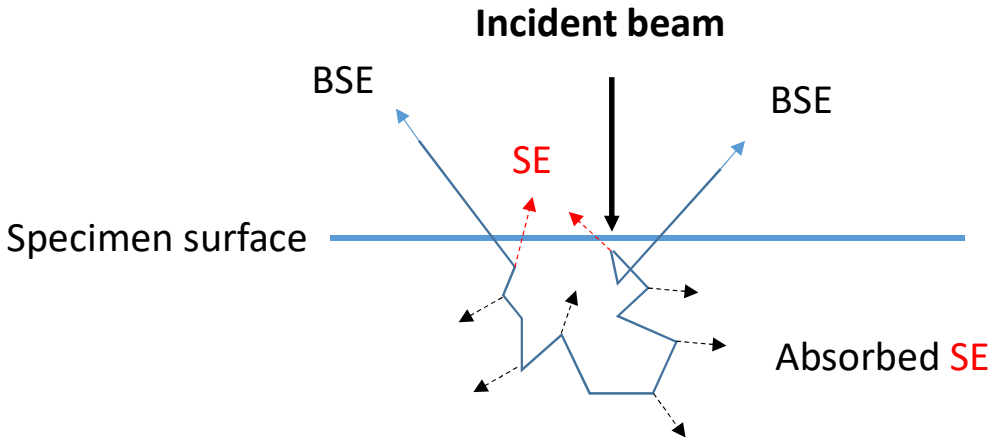
Backscattered electrons (BSE)

Within the sample a primary beam electron may be scattered in such a way that it escapes from the specimen without going through the sample thickness or after having travelled deeper. Backscattered electrons are the original beam electrons and thus, have a high energy level, near that of the gun voltage.



Primary electrons of sufficiently high kinetic energy may be deflected by the nuclei and remerge from the specimen surface. This is an example of Elastic Scattering since only a small amount of energy is lost in the interaction.

Multiple scattering events may take place before a BSE emerges from the surface



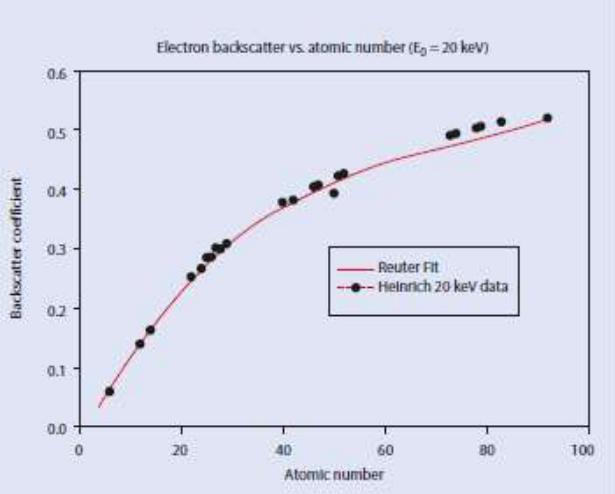
Backscattered electrons (BSE)

The 'yield' of BSE or the backscattered electron coefficient

Backscattered electrons are quantified with the "backscattered electron coefficient," η, defined as: $\eta = N_{BSE}/N_B$

Where N_B is the number of beam electrons entering the specimen and N_{BSE} is the number of electrons emerging as BSE.

The backscattering coefficient η strongly depends on the atomic number of the atoms in the sample and increases with increasing atomic number.



$\eta = -0.0254 + 0.016\,Z - 1.86 \times 10^{-4}\,Z^2 + 8.3 \times 10^{-7}\,Z^3$

The backscattering coefficient η depends only weakly on the accelerating voltage (for E> 10kV)

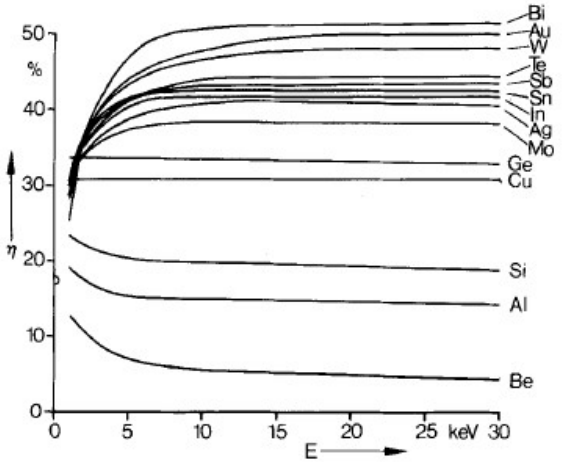


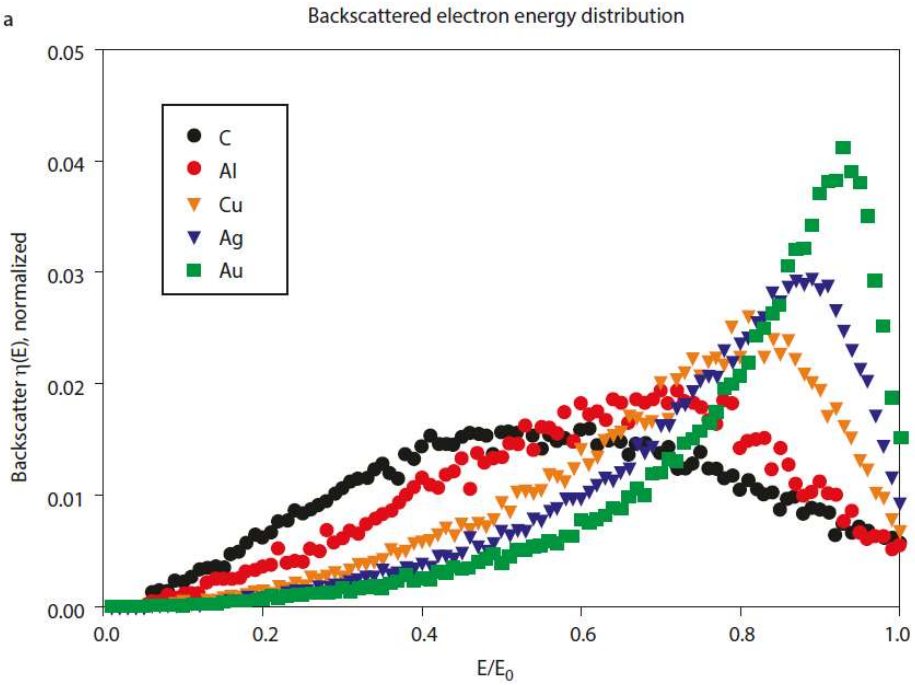
Fig. 4 Backscattering coefficient η as a function of electron energy E for different elements

SCANNING Vol. 3, 35-39 (1980)

Backscattered electrons (BSE)

Energy Distribution of Backscattering

The energy distribution of BSE is seen to extend from the incident beam energy down to zero energy. The energy distribution is sharply peaked at high fractional energy for a strong elastic scattering material such as gold, but the energy distribution is much broader and flatter for a weak elastic scattering material such as carbon.



Simulation of the energy of backscattered electrons for various pure elements at E0 = 20 keV and 0° tilt.

At a given accelerating voltage the BSE coefficient η is higher for heavier elements and consequently, the parts of the material containing heavy elements appear brighter that those part made of lighter elements. This is a form of compositional contrast.