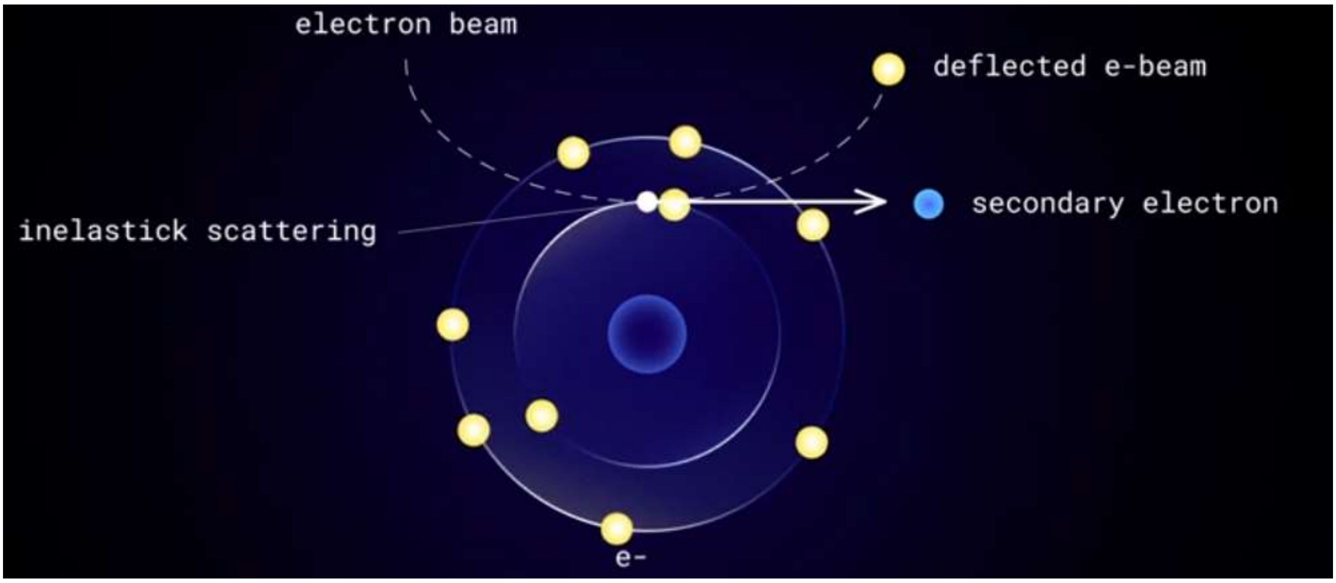
1/ Electron spectroscopy:

The device for producing electrons is called an electron gun. Since even high-energy electrons are absorbed by the air, their production and use takes place in a secondary vacuum enclosure. The electrons are produced by a hot cathode with thermionic emission or a cold cathode with field emission. They are then accelerated by an anode brought to a positive potential with respect to the cathode.
The sample to be studied is bombarded by a beam of primary electrons. The radiation produced during the interaction of the primary beam with the atoms of the sample is analyzed.

2/ Electron-Matter Interactions:

Electrons accelerated onto a material result in a number of interactions with the atoms of the target sample. Accelerated electrons can pass through the sample without interaction, undergo elastic scattering and can be inelastically scattered.



Elastic and inelastic scattering result in a number of signals that are used for imaging, quantitative and semi-quantitative information of the target sample and generation of an X-ray source. As shown in Figure 01, typical signals used for imaging include secondary electrons (SE), backscattered electrons (BSE), Cathodoluminescence (CL), Auger electrons and characteristic X-rays.

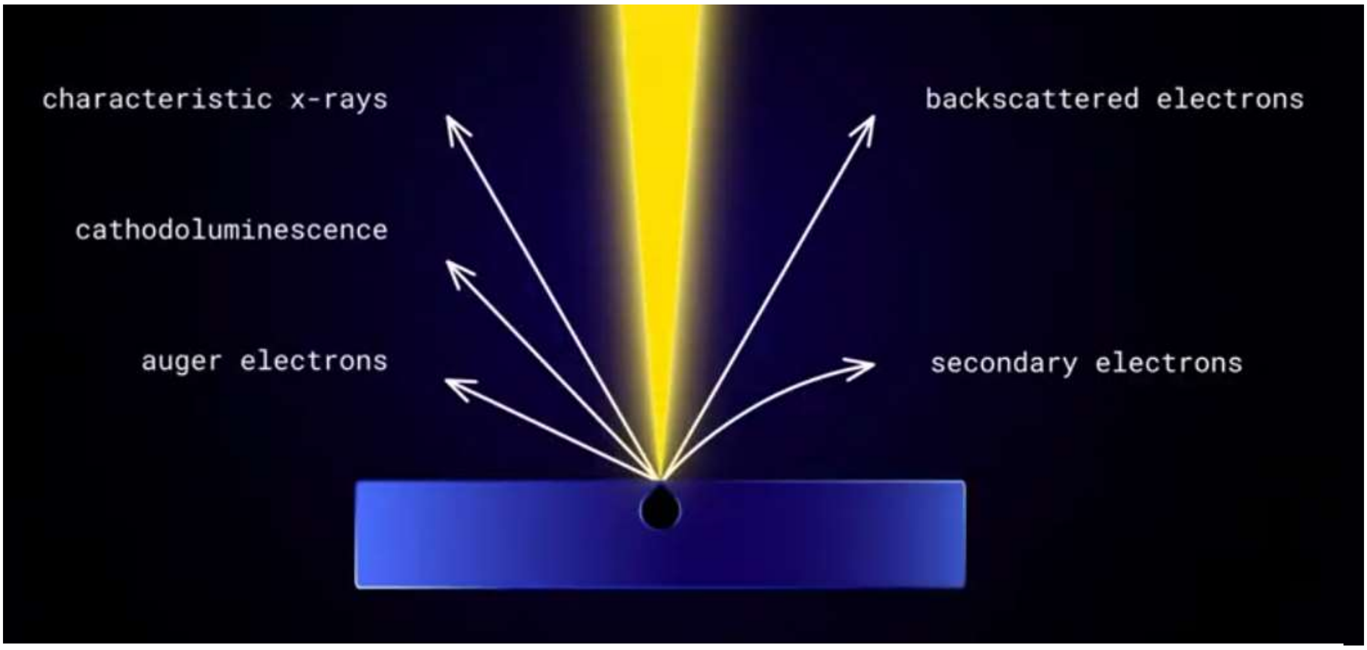
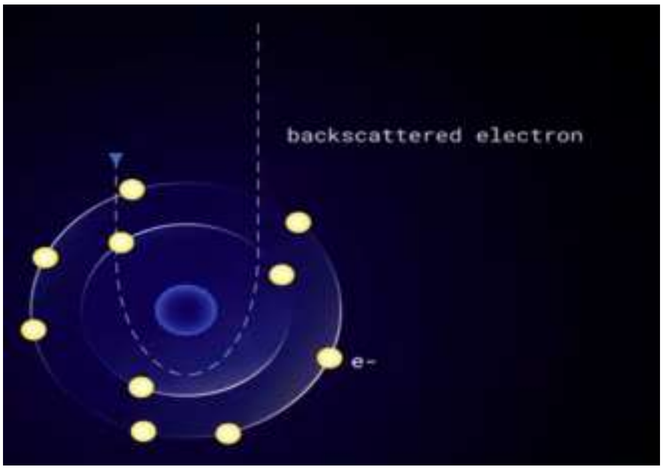


Figure 01

Where an electron beam impinges on a sample, electron scattering and photon- and X-ray-production develops in a volume -Figure 2-(the electron interaction volume) that is dependent on several factors.

These include:

- The energy of the incident beam (accelerating potential) increases the interaction volume, but decreases the elastic scattering (e.g. backscattering).



- The interaction volume decreases as a function of the mean atomic weight.
- Smaller and more asymmetric interaction volumes develop in samples tilted relative to the impinging electron beam.

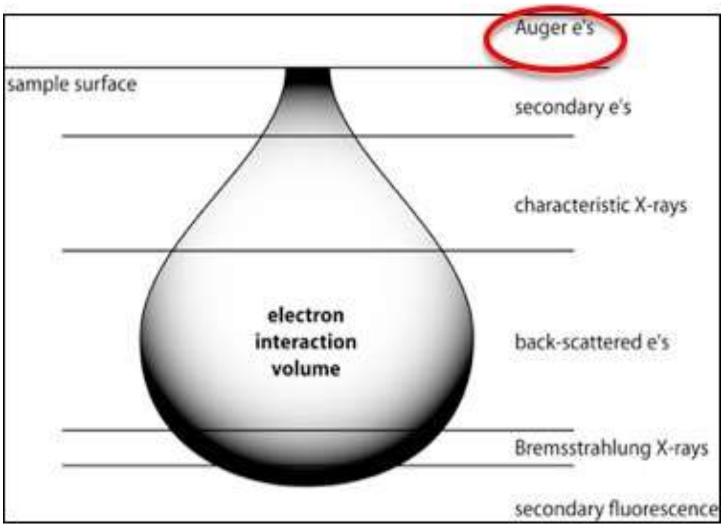


Figure 02: the electron interaction volume

Each of the signals used for imaging or X-ray generation is generated from different electron interaction volumes and, in turn, each of the signals has different imaging or analytical resolution.
Auger and Secondary images have the best imaging resolution, being generated in the smallest volume near the surface of the sample.
Backscattered electrons are generated over a larger volume resulting in images of intermediate resolution.
Cathodoluminescence is generated over the largest volume, even larger than Bremsstrahlung radiation, resulting in images with the poorest resolution.

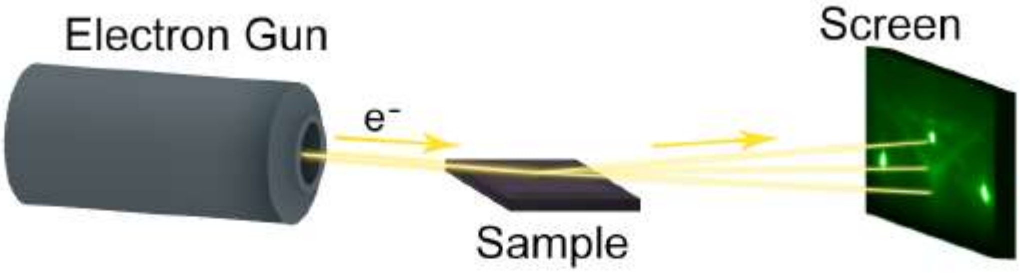
3/ Electron microprobe:

Analysis by electron microprobe, (Electron Probe Micro Analysis), is based on measuring the intensity of the characteristic X-radiation emitted by a given element, when the sample to be studied is subjected to electron bombardment.
During the bombardment, electrons from the incident beam give up part of their kinetic energy to electrons from the deep layers of the atoms in the sample and cause them to be ejected. Atoms that have lost a core electron become de-excited by emitting either an Auger electron or a characteristic X photon.
Electron diffraction is based on the wave nature of electrons and their strong interaction with matter. This technique makes it possible to study the structure and symmetry of surfaces. Depending on the energy of the electrons in the incident beam, three types of electron diffraction can be observed:

a- LEED (Low Energy Electron Diffraction): The surface is irradiated by a beam of electrons with an energy of between 10 eV and 1K eV .

The mean free path is only a few A°. The analysis of the diffraction diagram can provide information on the existence of a clean surface or an adsorbed layer, as well as the arrangement of the surface atoms.

b- RHEED (Reflection High Energy Electron Diffraction):
RHEED uses an electron gun to project electrons at a sample and measures the diffraction of those electrons on a fluorescence screen. Electrons penetrate only a few A° into a sample unlike X-ray diffraction (XRD) which measures the bulk of the material. High energy electrons are more easily focused via electromagnetic fields further making RHEED a prime candidate for thin films and various other surface studies. This technique is widely used to follow the growth of thin layers.



The simplest RHEED set up includes an electron gun, a sample, and a fluorescence screen across from the gun.

c- EBSD (Electron BackScatter Diffraction): It has become an additional characterization technique in the SEM. It allows the characterization of the microstructure and the local crystallographic texture of crystalline materials.

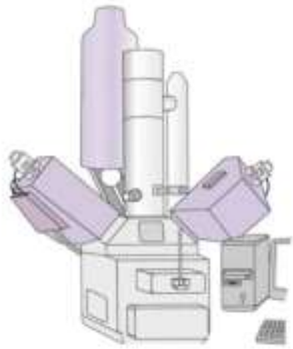
4/ Electron microscope:

To overcome the limitations of human eyesight, the microscope was developed and used as an efficient magnifying tool. Microscope has a much higher resolution and has been used as a powerful tool for studying and characterizing a wide range of materials.

Microscope can be divided into two categories on the basis of source to produce image, optical or light microscope (OM) and electron microscope (EM), both have the same working principle, but the major difference is the source, in Optical Microscope uses visible light as a source while Electron Microscope uses focused accelerated electrons beam.

a- SEM: "Scanning Electron Microscope is a versatile advanced instrument which is largely employed to observe the surface phenomena of the materials. The sample is shot in a SEM using highenergy electron, and the outcoming electrons/X-rays are analysed. These outcoming electrons/X-rays give information about topography, morphology, composition, orientation of grains, crystallographic information, etc. of a material.

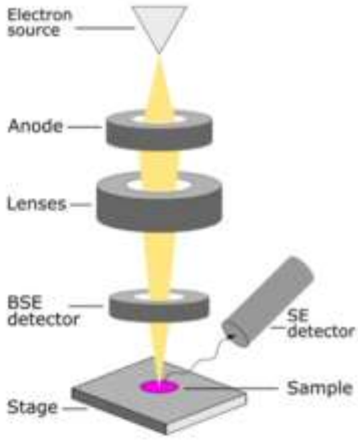
Scanning Electron Microscopy



What is Scanning Electron Microscopy ?

Scanning electron microscopy is a EM technique used to obtain high-resolution images and detailed surface information of samples

It is a type of electron microscopy that uses a focused beam of electrons to scan the surface of a specimen

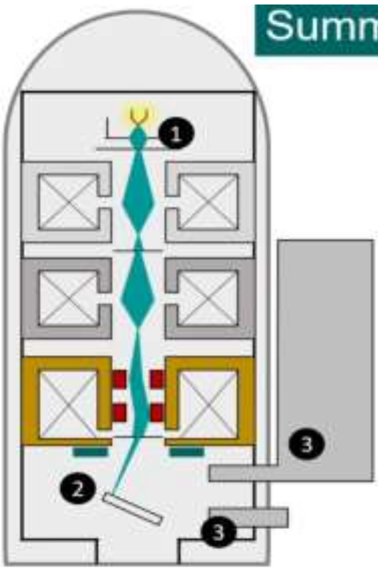


What is resolving power of SEM?

The resolution of SEM instruments can range from < 1 nanometer up to several nanometers.

Morphology indicates the shape and size, while topography indicates the surface features of an object or “how it looks”, its texture, smoothness or roughness. Likewise, composition means elements and compounds that constitute the material, while crystallography means the arrangement of atoms in the materials. SEM is the leading apparatus that is capable of achieving a detailed visual image of a particle with high-quality and spatial resolution of 1 nm. Magnifications of this kind of apparatus can extend up to 300,000 times. Although SEM is used just to visualize surface images of a material and does not give any internal information, it is still considered as a powerful instrument that can be used in characterizing crystallographic, magnetic and electrical features of the sample and in determining if any morphological changes of the particle has occurred after modifying the sample surface with other molecules”[1].

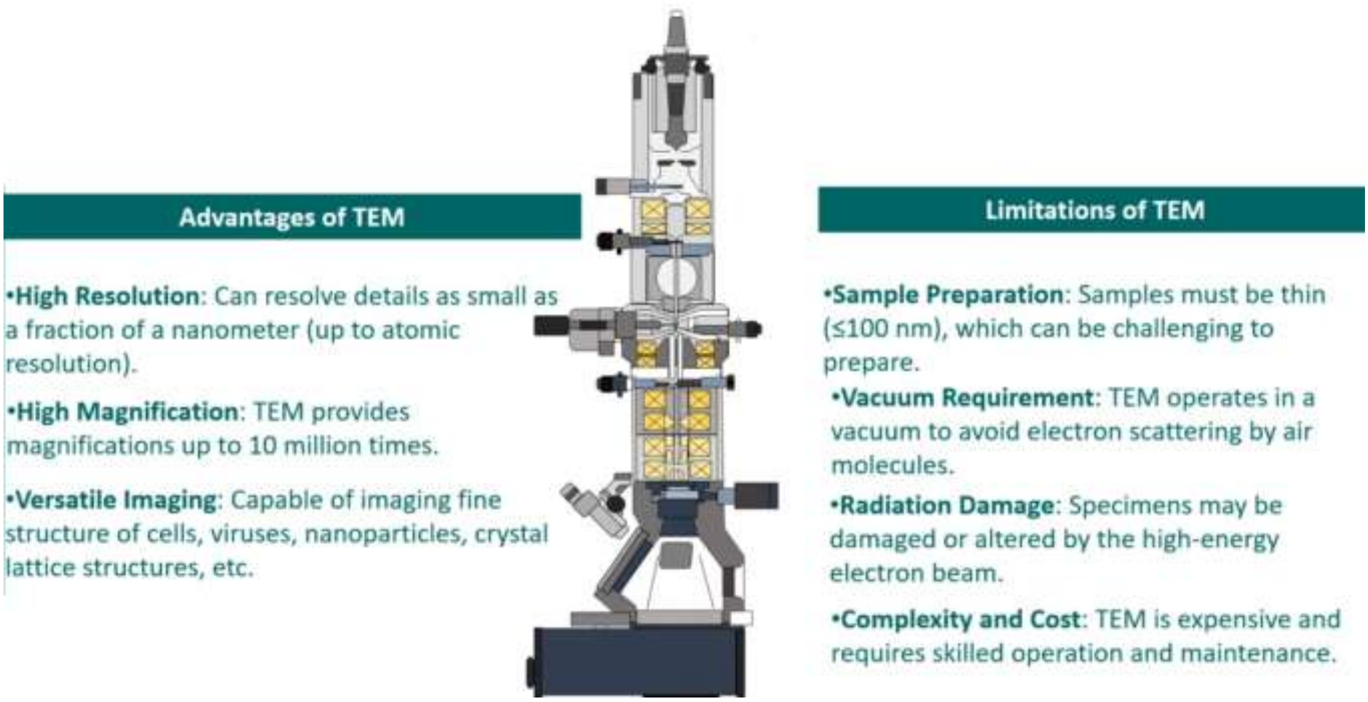
Summary: working principle of the SEM



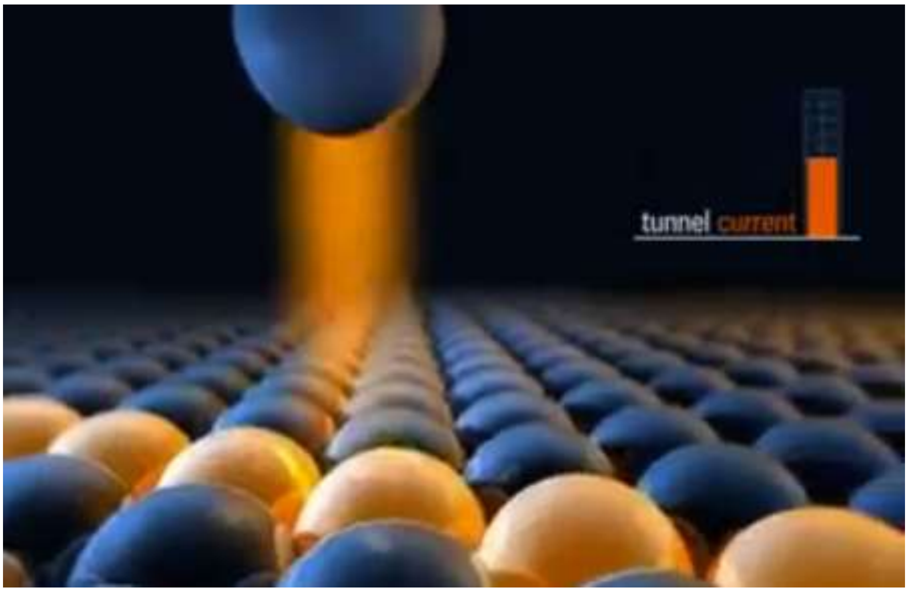
1. **Electron Beam Generation**: Electrons are produced by an electron gun and accelerated towards the sample.
2. **Scanning**: The electron beam scans the sample surface in a raster pattern.
3. **Interaction**: As the beam interacts with the sample, various signals (secondary electrons, backscattered electrons, etc.) are emitted.

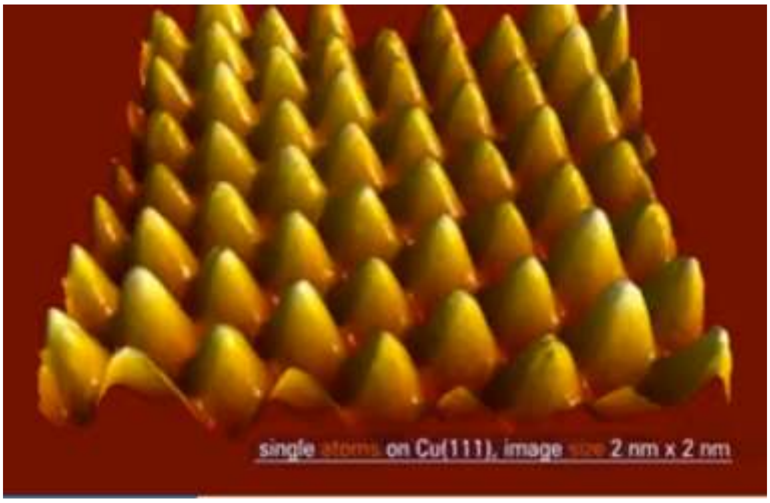
[1] Kalsoom Akhtar and all, Handbook of Materials Characterization, 2018 , Chapter 4, Scanning Electron Microscopy, p 116.

b- TEM: Transmission Electron Microscope provides powerful techniques for understanding various information of materials at very high spatial resolution, including morphology, size distribution, crystal structure, strain, defects, chemical information down to atomic level and so on. All the information that TEM can give to us are from electron-sample interaction. The transmitted electrons that have passed through the thin sample are detected to form images, which is the reason to call it "transmission" electron microscopy. In order to allow electrons to transmit through the sample, TEM sample must be very thin (typically, sample thickness is less than 200 nm, depending on the composition of sample and the expected information from TEM characterization).



c- STM: "In a Scanning Tunneling Microscopy experiment, a sharp metallic tip is separated by a few angstroms from a conductive sample. When a voltage is applied between the tip and the sample, electrons tunnel between them, producing an electric current, which decays exponentially with increasing tip–sample separation. In a standard operation, the current is kept constant during scanning by a feedback circuit, so that the vertical displacement of the scanner reflects the surface topography and gives true atomic resolution. Tip shape and sharpness are the two most important parameters in imaging surfaces, particularly those with significant topography. STM images invariably include contributions from specimen structure and tip geometry. Thus, the study of the tip's geometry is indispensable in distinguishing between the apparent and the true structure, or to establish the relationship among the tip's geometry, the true surface structure, and the STM image"[2].





[2] Chiara Musumeci, Advanced Scanning Probe Microscopy of Graphene and Other 2D Materials, Crystals **2017**, p2.