MODULE I
SCANNING ELECTRON MICROSCOPE (SEM)
**Scanning Electron Microscope (SEM)**

Initially, the plan of SEM was offered by H. Stintzing in 1927 (a German patent application). His suggested procedure was unable to produce magnified image because the collimated beam with which sample was irradiated was light, X-rays and corpuscles. Then a German electrical engineer named M. Knoll contributed a paradigm of SEM in 1935 where specimen was scanned with electron beam to obtain image. In 1938, Von Ardenne developed SEM with slight modification by introducing DE magnifying lenses called scanning transmission electron microscope to scan thin samples. For scanning bulk samples Zworykin (in 1942) improved SEM with few other alterations. Eventually SEM was commercialized in 1965 with many alterations being done in the R & D of the Oatley Lab.

**Principle:**

**SEM (Figure 1)** belongs to the family of electron microscopes which produce images of an object by scanning its surface with highly focused electron beam. The process involves the interaction of electrons with atoms of an object, creating signals containing information of object’s composition and topography. Arrangement of constituent atoms is studied by 2D beam scanning upon the sample surface and image is produced from collected secondary electrons. Scan pattern is generated by the electron beam and the image is formed by merging beam’s position and the detected signal.

**Instrumentation of SEM**

The basic components used in electron optical system are:

- A source of electrons, called electron gun
- Lenses
- Scanning Coils
- Detectors to collect signals
- Sample Stage
- Display/Data output devices

**Infrastructure Requirement**

- Power supply
- Vacuum system
- Cooling system
- Vibration free floor
- Room free of ambient electric and magnetic fields
Figure 1 Basic Construction of SEM.

Description of Components

1) Electron Beam: It has two variables i.e. energy and current. The voltage is variable from about 1 - 60keV and the current from 1e-7 to 1e-12 A. These values are specific to the instrument model.

2) Electron Gun: It is used to produce fine electron beam (and is also called as electron probe). Several different types of electron guns used are:
   a) TE (Thermionic- Emission) gun
   b) FE (Field- Emission) gun
   c) SE (Schottky- Emission) gun

TE (Thermionic- Emission) gun (Figure 2) –

- A thin tungsten wire filament acts as cathode to generate thermo electrons by heating the filament at 2800K.
- A positive voltage of about 1 to 30 KV is applied to the metal plate acting as anode, in order to collect these thermo electrons.
- By applying negative voltage to the Wehnelt electrode placed between the anode and the cathode, current of the electron beam is adjusted. This electrode also helps in focussing the electron beam.
- Thinnest point of beam known as cross-over (15-20μm Diameter), regarded as actual electron source.
- LaB₆ crystal is used as a cathode. It used to reduce the spot size. It requires high vacuum due to its higher activity.

![Diagram of TE gun](image)

**Figure 2 Construction of TE gun.**

**FE (Field- Emission) gun (Figure 3) –**
- Provides high resolution.
- Works on field-emission effect when high electric field is applied to the metal surface.
- A thin tungsten wire act as cathode wielded to the tungsten single crystal whose tip is curved with the radius of about 100nm, and is known as emitter.
- Electrons are emitted from emitter through tunnelling effect when positive voltage is applied to the extracting electrode.
- Hole is created in the extracting electrode to allow emitted electrons to flow through it. Then electron beam containing some energy is obtained by applying voltage to the accelerating electrode present beneath the extracting electrode.
• In FE gun energy spread is less because no heating is required and also electron beam diameter is 5-10 nm.
• Requires ultra-high vacuum of the order of $10^{-8}$ Pa.

![Figure 3 Construction of FE gun.](image)

SE (Schottky- Emission) gun (Figure 4) –

• Works on Schottky emission effect when high electric field is applied to heated metal surface.
• A tungsten single crystal (tip radius – few hundred nm) coated with ZrO acts as cathode.
• ZrO coating reduces the work function to enhance the emission current at low cathode temperature.
• Thermo electrons are shielded from emitter by applying negative voltage to the suppressor electrode.
• Advantage: electron beam current is highly stable because emitter is placed in ultra high vacuum of the order of $10^{-7}$ Pa.
• Produces larger probe current.
Lenses

To produce finest beam of electron with desired crossover diameter, two-level lens system, i.e., condenser and objective lens, made of metal cylinders with cylindrical hole, operating in vacuum is used. These lenses are located beneath the electron gun. Magnetic field is generated in the inner part of the lenses to focus or de-focus the beam.

Figure 5 Formation of electron probe by lenses.
Role of condenser lens:
Condenser lens affects the probe size. If it is strengthened then probe size is narrowed with a smaller ratio of b/a, whereas if it is weakened then probe size is broadened. C1 and C2 lenses control the beam current by varying size and intensity of beam spot. Aperture is formed by making a small hole in the metal placed between the two condenser lenses and the objective lens to allow the beam to pass through it and reach the objective. Resolution is dependent upon aperture as it controls the spot size.

Role of objective lens:
It is used for focusing and determines the final diameter of probe.

Scanning Coils-
These coils deflect the beam in X or Y directions in order to scan the sample surface in a raster pattern.

Sample Stage-
It is a motorized plate which has movement in three directions X, Y and Z controlled by feeding value in the software. The samples are supported on it and move smoothly in the required direction. X and Y, the two horizontal movement are used to change the field of view whereas Z, the vertical movement is required for image resolution as well as depth of focus. Along with these movements rotation and tilting are also possible. Also, stage movement can be controlled manually through mouse in the user interface of the software.

Detector–
Characteristics of sample are measured at different beam position to form image. Secondary electrons emitted from the sample are measured using secondary electron detector.

- **Secondary Electron Detector:** It includes a Scintillator coating at the detector tip and high positive voltage of 10 KV is applied to it. The secondary electrons emitted from the specimen get magnetized towards this positive voltage, also this secondary electron collection is supported by supplementary electrode (the collector) placed before scintillator by applying few volts to this collector. When they hit the scintillator light is produced which is guided to PMT (Photo multiplier tube) through light guide. Then light is converted to electrons which are amplified as electric signal. This detector was fabricated by Everhart and Thornley, and therefore named as E-T detector. For higher resolution in some SEM TTL (Through The Lens) detectors are used which consists of secondary electron detector above the objective lens.
Display Unit and Recording system-

The output in the form of amplified electronic signal is send to the display unit. To form SEM image, scanning is synchronized with electron beam scan and brightness (which depends upon number of secondary electrons emitted) on the display unit. Originally, CRT (Cathode Ray Tube) was used as a display unit but these days it is replaced by LCD (Liquid - Crystal Display). Usually, very fast scan speed is used while focusing for observation, whereas slow speed is used for capturing or saving the image.

Vacuum System-

The microscope column and the specimen chamber is kept under high vacuum i.e. $10^{-3}$ to $10^{-4}$ Pa. Diffusion pump is used to evacuate these components. For oil-free environment – Turbo molecular pump is used, for FE-SEM – sputter ion pump is used as Fe-SEM requires ultra-high vacuum conditions.

Principle of SEM image formation-

When an electron beam is incident on the sample then many different types of signals are generated which are eventually used to observe or analyze morphology/topology of the sample. SEM is also used for elemental and state analysis. These signals includes: Secondary electrons, Backscattered electrons, Auger electrons, Cathodoluminescence and X-rays.
Interaction of Electrons with Specimen: Electrons entering specimen gets scattered within it and lose their energy gradually upon getting absorbed within the sample. Scattering range within specimen depends upon:

- Element’s atomic number \( Z \) making the sample - More \( Z \) Less Scattering.
- Density of constituent atoms – More Density less scattering.

Monte Carlo simulations are employed to understand the behavior of electrons in the sample (Figure 8).

Figure 7 Emission of various electrons and electromagnetic waves from the specimen.

Figure 8 Monte Carlo simulation showing the scattering behavior of electrons within the specimen.
**Acceleration Voltage effects:**

Higher the voltage, higher is the penetration depth of the beam within the sample, thereby providing the inside information of the specimen. However, it spoils the contrast at the specimen’s surface due to broadening of the spot size inside the sample. Therefore, lower acceleration voltages are used to view surface of the samples.

![Figure 9 Overlap of the internal structure information on the specimen surface.](image)

**Magnification of SEM -**

Magnification is yet another parameter of the performance of SEM. Magnification in a SEM can vary in the range of around 6 orders of magnitude, or from ~10 - 500,000 times. Unlike the optical and transmission electron microscopes, magnification in SEM does not depend on objective lens power. The condenser and objective lenses only focus the beam to a spot, and do not form image of object. Further, SEM can even work without the condenser and objective since the electron gun itself generates a highly focused electron beam, but it may not be able to achieve high resolutions. Like the scanning probe microscopy, magnification in SEM comes from ratio of the dimensions of raster on specimen and display screen. For a fixed display screen, reducing the dimensions of raster on object can lead to higher magnifications, and vice versa. Thus, the magnification can be regulated by the current fed to x, y scanning coils, and voltage applied on x, y deflector plates. The numerical value of magnification is determined by the ratio of the length of the monitor versus the length of the scan on the sample:

\[ M = \frac{L_{\text{mon}}}{L_{\text{spec}}} \]

**Depth of Focus -**

Sometimes when top side of the specimen is focused the bottom goes out of focus. For SEM systems this depth of focus depends upon the two variables which are:

1. **The aperture size**
2. **The working distance**
When aperture or aperture angle is small, then the depth of focus will be larger. On the other hand, when the aperture or aperture angle is larger, the depth of focus will be smaller. The parallel electron probe entering through smaller aperture angle then the image remains focused even though the focus is varied significantly and when electron probe entering is at an angle larger than the aperture angle, then image goes out of focus even if the focus is varied slightly, as shown in Figure 10.

Figure 10 Relation between the aperture angle of the electron probe and the depth of focus.

SEM Sample-
Conducting samples provide a path to ground for the beam electrons, and therefore require no special preparation. Insulating materials, however, require a thin coating of a conductor (often carbon or gold) in order to prevent charging.

Sample Preparation-
It is done in order to eliminate the sample charging few steps are followed:

1. **Charging**: A thin metal coating of about 10nm is done on the sample because metal film is highly stable and its secondary electron yield is higher. Too thin coating is not preferred because continuity is lost.
2. **Low accelerating voltage**: Low KV value of about 1KV can be used to scan insulating samples because the number of incident electrons becomes equal to the number of emitted secondary electrons, implying that the sample is not charged.
3. **Tilt Observation**: In this case secondary electrons yield is higher as electron beam is entering at an angle.
4. **Low Vacuum SEM observation**: Ondecreasing the vacuum, the gas molecules within the sample chamberincreases, which get ionized due to electrons and thus, on reaching the specimen as positive ions neutralize the charging.
Review your learning:

1) Which of the following scientists is credited with the invention of electron microscope and awarded the noble prize for the same?

   a) J. J. Thompson
   b) Ernst Ruska
   c) Louise de Broglie
   d) Otto van Borris

2) Osmium tetra oxide is used in electron microscopy as a

   a) Precipitator
   b) Mordant
   c) Staining agent
   d) Fixing agent

3) All of the following are true for both TEM and SEM both

   a) The illuminating source is electron beam
   b) The microscope is focused using electromagnetic lenses
   c) Can be used to view specimens smaller than 0.2 micrometer
   d) The specimen must be sectioned prior to viewing

4) In its usual mode, the SEM has a magnification that ranges from

   a) 1X To 100X
   b) 10X To 100,000X
   c) 100X To 10,000X
   d) 10X To 10,000X

5) The major attractions of the scanning electron microscope (SEM) include all of the following except:

   a) Its ability to polarize light.
   b) Its high resolution.
   c) Its high magnification.
   d) Its great depth of focus.

True/False:

1) In electron microscopy, electron dense regions of the organisms appear brighter than electron transparent region.
2) Contrast in SEM is a result of sample in-homogeneity.
3) Scanning Electron Microscopy (SEM) is better than standard light microscopy.
4) One advantage of a tungsten (W) filament type electron gun is that it requires no electrical fields in order to form an e-beam.
5) The e- in the SEM beam are of such short $\lambda$ (wavelength) that they do not interact with small gas molecules such as $O_2$ and $N_2$.

Match the following parts of the SEM electron beam as follows:

1) Highest current beam entering anode plate
2) Med. current beam as it strikes the sample
3) Lowest current beam just leaving gun cap

Long type questions:

1) What 2 important things are brought together in the area/volume defined by the working distance?
2) Which SEM control directly affects the scan coils? contrast/ magnification/spot size/ACCV (acceleration Voltage)?

References:

3) https://www.slideshare.net/JessaArio/scanning-electron-microscopy
4) www.ems.psu.edu/~ryba/harbin/SEM.ppt
5) https://nitrkl.ac.in/Institute/SEM/docs/information/Basic%20SEM%20Design.pdf
6) www.understanding-cement.com/sem-introduction.html