Module-1 Unit-4

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 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 used 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 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 acquisition 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

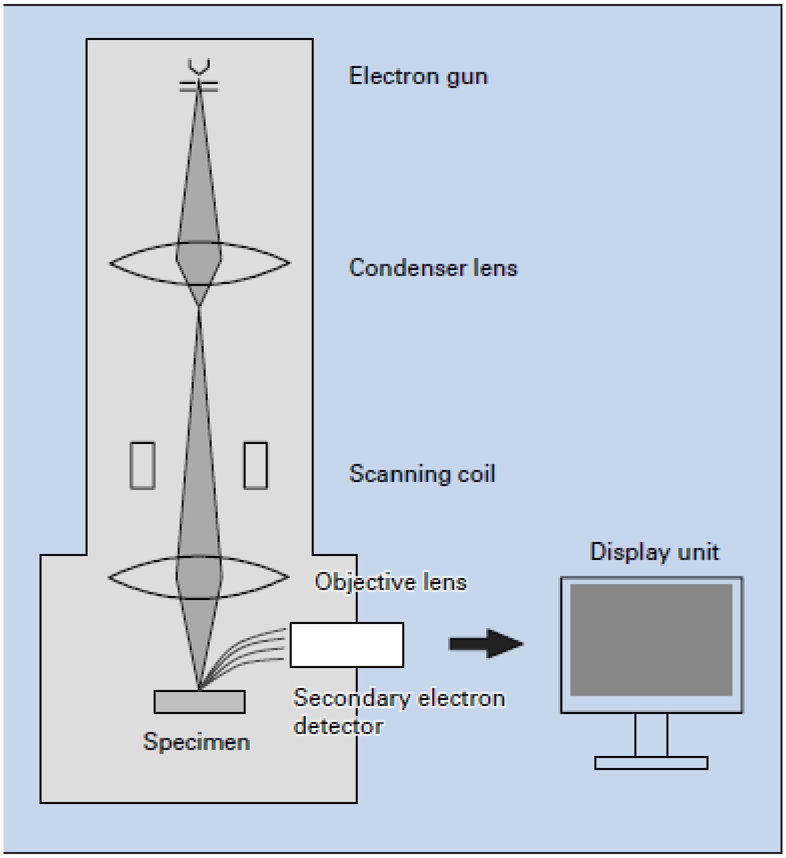

Fig 1: Basic Construction of SEM

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 (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 –

- A thin tungsten wire filament act as cathode to generate thermo electrons by heating the filament at 2800K.
- By applying positive voltage of about 1 to 30 KV 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.
- LaB_6 crystal is used as a cathode. It used to reduce the spot size. It requires high vacuum due to its higher activity.

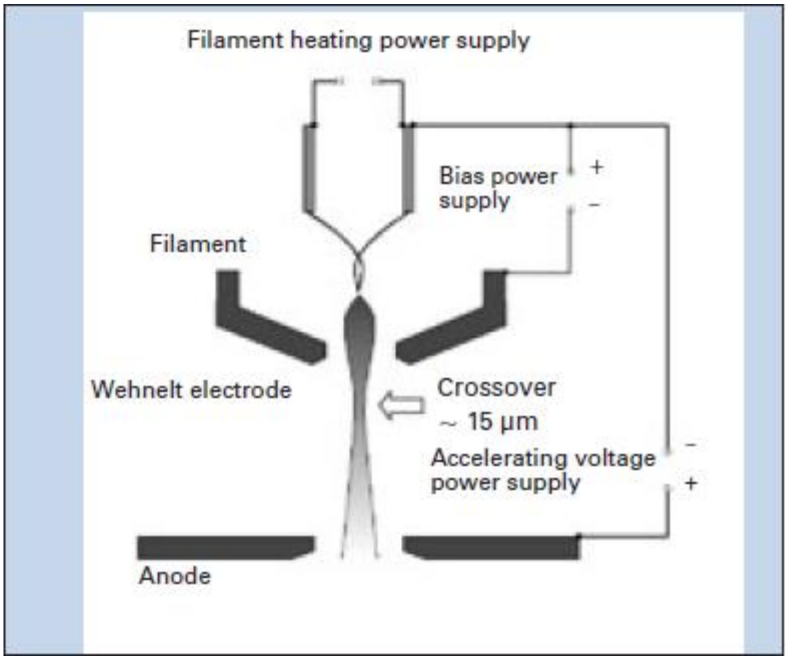

Fig 2: Construction of TE gun

FE (Field- Emission) gun –

- 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 known as emitter.
- Electrons are emitted from emitter through tunneling effect when positive voltage was applied to the extracting electrode.
- Hole 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 5-10nm.
- Requires ultra-high vacuum of the order of 10^{-8} Pa.

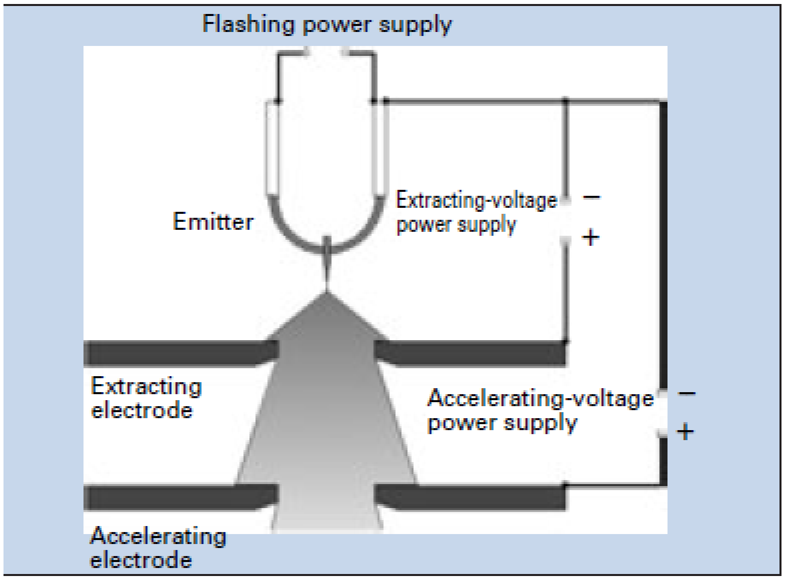

Fig 3: Construction of FE gun

SE (Schottky- Emission) gun –

- 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 acting 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.

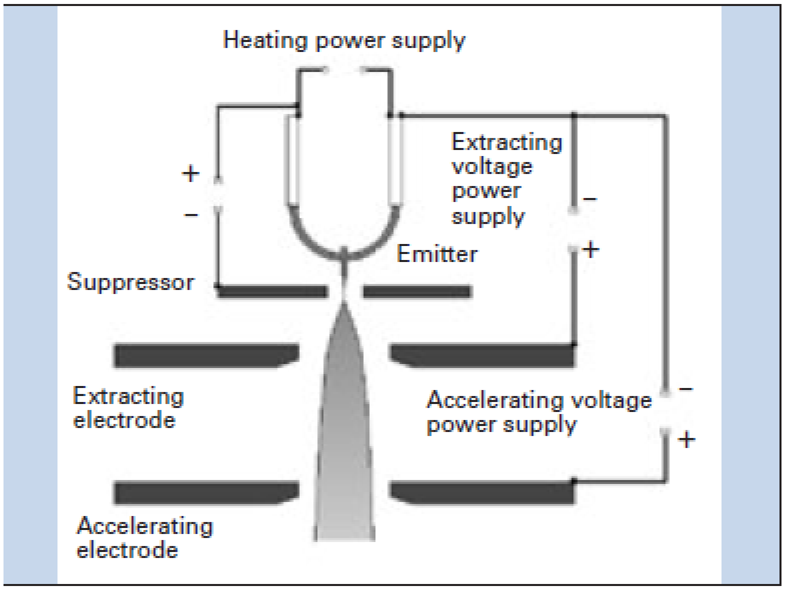

Fig 4: Construction of SE gun

Lenses

To produce finest beam of electron with desired crossoverdiameter therefore two- level lens system used are condenser and objective lens made of metal cylinders with cylindrical hole, which operate in vacuum.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.

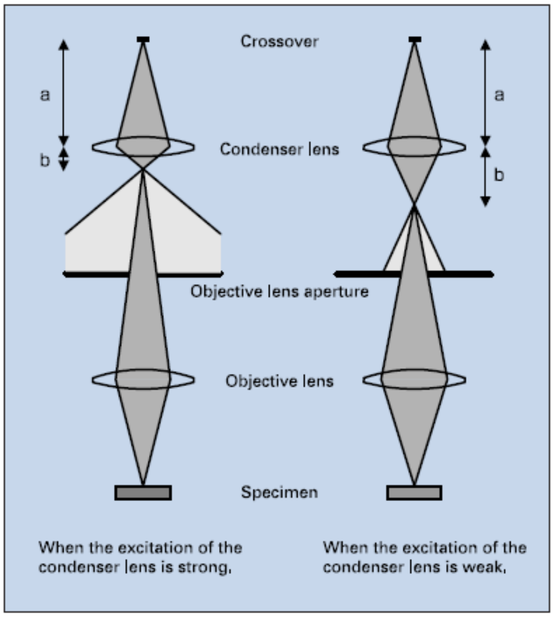

Fig 5: Formation of electron probe by lenses

Role of condenser lens:

Condenser lens action is related to the probe size. If it is strengthened then probe size is narrowed with a smaller ratio of b/a, whereas it is weakened then probe size is broadened. C1 and C2 lenses controls the beam current by varying size and intensity of beam spot. Aperture with a small hole in it made of metal placed between two condenser and objective lensto allow the beam to pass through it to reach the objective lens. 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 single click of mouse.

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 is consisting of a Scintillator coating at the detector tip and high voltage of 10 KV applied to it. The secondary electrons emitted from the specimen gets 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 is fabricated by Everhart and Thornley, therefore named as E-T detector. For higher resolution in some SEM TTL (Through The Lens) detectors are used which is consist of secondary electron detector above the objective lens.

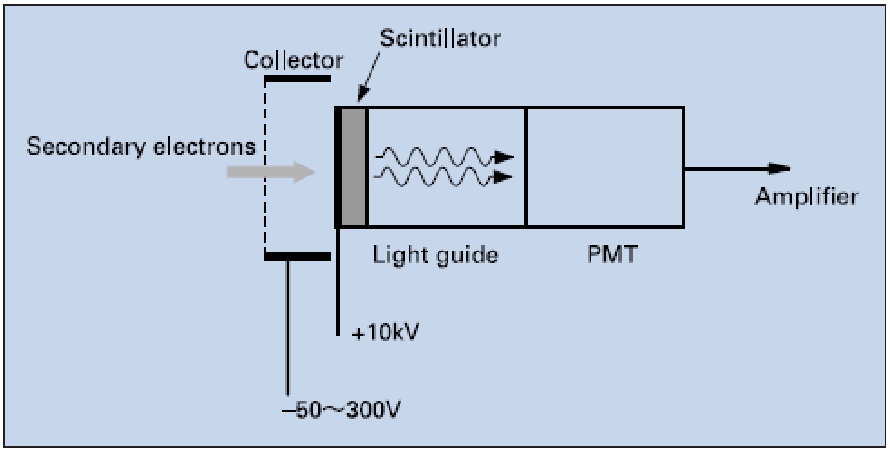

Fig 6: Construction of Secondary electron detector

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 appearing on the monitor screen. Previously, CRT (Cathode Ray Tube) was used as a display unit but these days it is replaced by LCD (Liquid - Crystal Display). Extremely fast scan speed is used while focusing for observation and slow speed used for capturing or saving 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 is ultra-high vacuumed.

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.

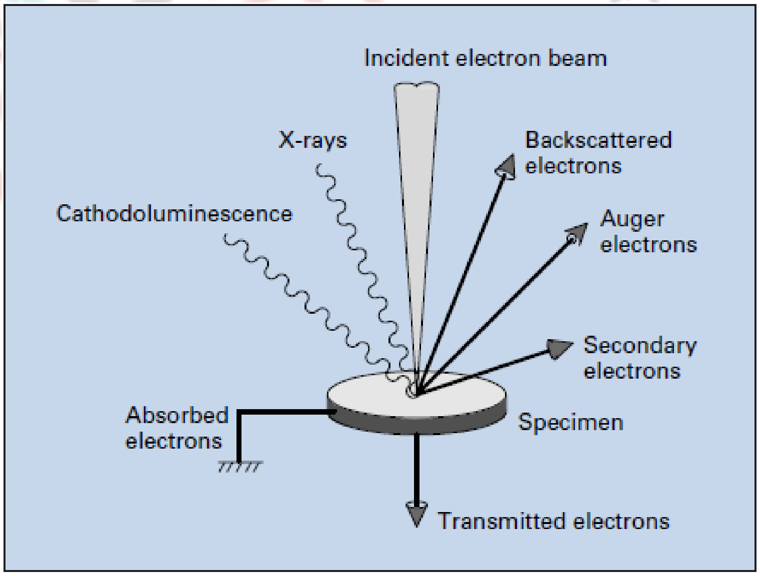

Fig 7: Emission of various electrons and electromagnetic waves from the specimen

- **Interaction of Electrons with Specimen:** Electrons entering specimen gets scattered within it and lose its energy gradually upon getting absorbed within it. Scattering range within specimen depends upon-
    - Energy of electrons – More Energy More Scattering.
    - Elements atomic number (Z) making the sample - More Z Less Scattering.
    - Density of constituent atoms – More Density less scattering.

  Monte Carlo enabled us to understand this phenomenon.

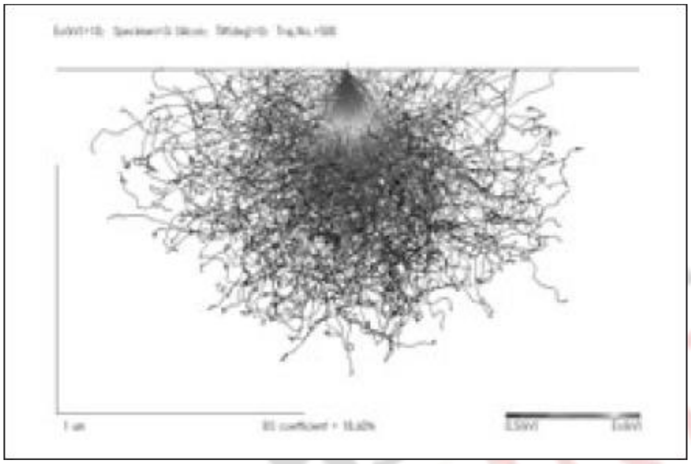

Fig 8: Monte Carlo simulation showing the scattering behavior of electrons within the specimen

Acceleration Voltages effects:

Higher the voltage higher is the penetration depth of the beam within the sample thereby providing the inside information of the specimen but spoiling the specimen surface contrast due to broadening of the spot size inside sample. Therefore, it is suggested to view surface structure lower acceleration voltages is used.

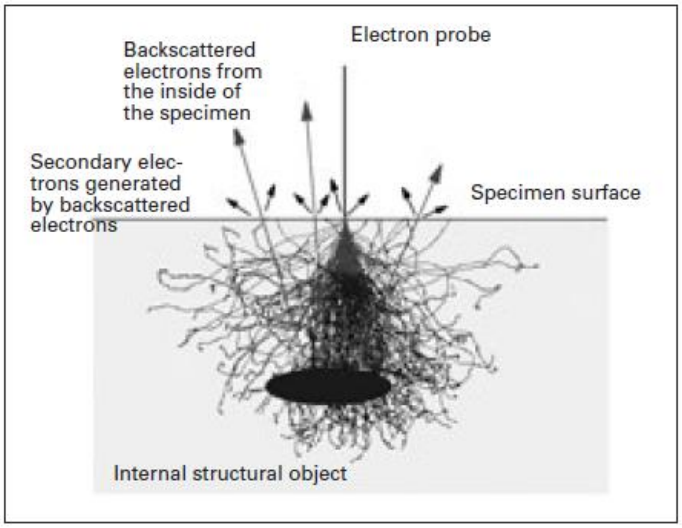

Fig 9: Overlap of the internal structure information on the specimen surface

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:

$M = L_{mon}/L_{spec}$.

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:

a) The aperture size

b) 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 some angle through larger aperture angle then images goes out of focus even though the focus is varied slightly, as shown in fig 10.

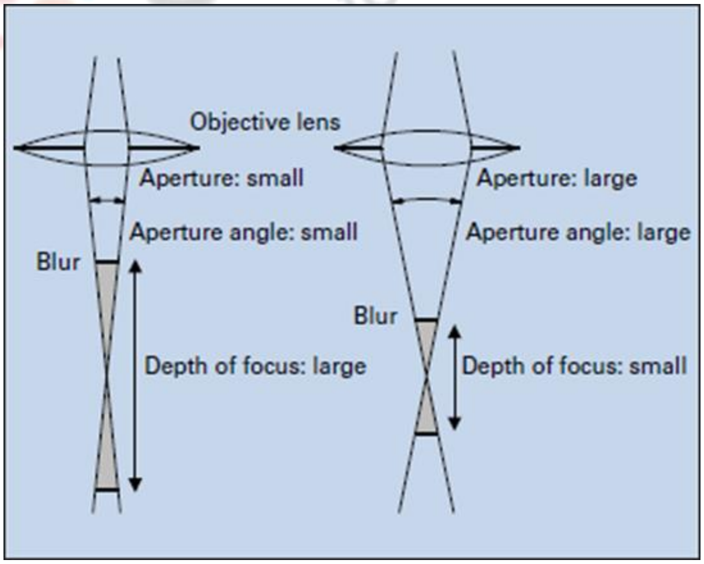

Fig 10: Relation between the aperture angle of the electron probe and the depth of focus

SEM Sample-

Conducting samples provide a path to earth 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:

a) **Charging:** A thin noble 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.

b) **Low accelerating voltage**: Low KV value of about 1KV can even scan insulating samples because number of incident electrons becomes equals to the number of emitted secondary electrons that means sample is not charged.

c) **Tilt Observation:**In this case secondary electrons yield is higher as electron beam is entering at an angle.

d) **Low Vacuum SEM observation:** Ondecreasing the vacuum increases the gas molecules within the sample chamber which gets ionized due to electrons and thus, on reaching the specimen as positive ions neutralizes the charging.

References:

Website site link:

1) http://www.jeolusa.com/RESOURCES/Electron-Optics/Documents-Downloads/EntryId/598.
2) https://link.springer.com/chapter/10.1007%2F978-0-387-49762-4_3
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

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 λ (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)?