Development of electron microscope

1897: Thompson describes the existence of negatively charged particles (electrons)

1925: De Broglie theorised that electrons have wave-like characteristics, addressing the wave/particle duality

1927: Thompson and Reid demonstrated the wave nature of electrons by diffraction experiments

1931: Ruska et al. build the first electron microscope (Nobel prize in 1986)

1949: Philips commercialises the EM100....
SCANNING ELECTRON MICROSCOPE (SEM)

The basic steps involved in all Electron Microscopy are:

- A stream of electrons is formed by the electron source (*electric gun*) and accelerated toward the specimen using a positive potential.
- This stream of electron is focused using apertures and magnetic lenses into a thin, focused, monochromatic beam.
- This beam is focused onto the specimen using a magnetic lens.
- Interactions occur inside the specimen, affecting the electron beam. These interactions are detected and transformed into an image.

SEM components

1. **Microscope column:**
   - Electron gun (source of electrons)
   - Electromagnetic lenses
   - Apertures
   - Scan coils (used to adjust beam position)
2. **Specimen stage (chamber)**
3. **Vacuum pumping system** *(keep clean, filament oxidation, air will scatter e’s)*
4. **Secondary electron and backscattered electron detectors** *(receive signals used in imaging)*
5. **X-ray detector** *(EDX or EDS)* – used to detect x-rays for chemical analysis
6. **Electronic control and imaging system:** to control the SEM, including focusing, magnification and imaging
SEM PRINCIPLES

- The SEM uses electrons instead of light to form an image.
- A beam of electrons is produced by the electron gun.
- An anode attracts the electrons and directs them towards the sample.
- A series of electromagnetic lenses focuses the beam.

- One type of lens is the condenser lens, which demagnifies the beam to decrease the divergence angle.

- A second type of lens is the objective lens, which further narrows the beam to focus it on the sample.
• Once the beam hits the sample, several electrons are ejected from the sample. These include:

- Secondary Electrons (SE)
- Back-Scattered Electrons (BSE)
- X-rays
- Auger Electrons
- Cathodoluminescence (CL)

• Detectors collect these secondary and back-scattered electrons and convert them into a signal that is sent to a viewing screen similar to a TV, thus producing an image.

1. TYPES OF ELECTRON GUNS (electron source)

- Tungsten hairpin Filament (most commonly used)
- Lanthanum Hexaboride (LaB₆)
- Field Emission (FE) (W or C cathode)

• Electrons are produced from the filament by thermionic emission.
• Current is passed through the filament in order to heat it sufficiently to a T = 2700 K.
• Heating is accomplished by running a 3-4 amp current through the filament.
• There is a large spread in electrons from the filament tip.
• The filament is at a large negative potential.
The electrons are initially focused by the Wehnelt grid cylinder “cap” which is biased negative at -200 to -300 V with respect to the filament.

The Wehnelt reduces the space charge (suppresses emission from the filament except at its tips).

The electrons are accelerated towards the anode (10-100 μm in diameter)

The electrons converge at a point between the Wehnelt cap and anode. This point is called the “cross-over” and is the location of the effective electron source.
- The anode has a hole in its centre and is biased from 1 to 50 keV with respect to the filament-Wehnelt.

- This bias accelerates the electrons and forms the beam.

- The electron flux is minimal until the temperature of the filament is approximately 2500 K.

- Above this temperature it is predicted that the electron flux will increase with increasing temperature, until the filament melts.

- In practice, however, the electron emission reaches a plateau termed "saturation", due to the self-biasing effects of the Wehnelt cap.

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**Figure 7.35:**
(A) The diagram shows rise of brightness as the filament current is raised and the filament heated. A "bake peak" is encountered at point A, followed by a drop in the brightness at point B, and a second drop at point C, before reaching the saturation point D. Beyond the saturation point, the filament may be damaged. The horizontal filaments may be damaged within minutes. A curve would not be at the four points on the abscissa curve shown in the top diagram.

(B) Bent filament taken from an XRF. The bent filament shows a "bake peak" and a second drop in brightness. A second drop in brightness indicates the filament while the properties from the filament are pronounced indicates evaporation of the filament due to a vacuum leak.
**ELECTRON BEAM LOCATION**
- Filament position is very important
- Correct position optimises the electron flow
  - Less heat required
  - Extended filament life

**FILAMENT LIFE**
- Maintenance of high vacuum
- Cleanliness of the electron gun
- Filament current

**ACCELERATING VOLTAGE**
- Voltage varies between 2 - 30 KV
- Electrons have energy of the accelerating voltage
  - at 25 KV, the energy of the electrons = 25 eV.

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2. **ELECTROMAGNETIC LENSES**

- Electrons are negatively charged particles
  - Path can be changed by a magnetic field

- Electron microscope lenses are essentially a coil of wire around a metal cylinder, called *solenoid*.

- The electron path diversion depends on:
  - magnetic field
  - velocity
  - direction
2.1. CONDENSER LENSES

- The electron beam is divergent after passing through the anode and must be collimated by the condenser lens(es) and apertures into a relatively parallel beam.

- The electron beam converges and passes through a focal point (which moves up and down with the strength of the magnetic field).

- The condenser lens controls the amount of current that passes down the rest of the microscope column (controls the intensity or brightness of the electron beam).

2.2. OBJECTIVE LENSES

- The objective lens is located at the base of the column just above the sample.

- This lens focuses the electron beam onto the sample and controls its final size and position.

3. SCANNING COILS

- The scanning coils are housed inside the objective lens.

- They cause the beam to scan over the specimen surface in an adjustable way.
4. ASTIGMATISM

- Astigmatism enlarges the effective beam size (spot size)
- The spot size may not be clear due to:
  - aberrations
  - aperture contamination
  - column contamination

- Stigmator coils housed within the scanning coils consist of a set of electromagnetic lenses that control the final beam shape by applying a magnetic field to make the beam circular.

- They also correct for any dirty column or apertures
4. APERTURES
- Apertures are used to
  - reduce or exclude extraneous electrons
  - reduce aberrations

SPECIMEN CHAMBER
- Houses the specimen and the various detectors (SE, BSE, EDX, …)

SPECIMEN STAGE
- There are five types of motion (TXYZR)
  - Tilt, X, Y, Z, & Rotation

Large SEM specimen chamber
MAGNIFICATION

- The mechanism by which the image is magnified is very simple and does not involve any lenses.
- The CRT area is fixed, and only the area scanned varies.
- The magnification depends on the
  - Spot (beam) size
  - Area scanned

- The magnification $M = \text{the side length of the CRT} / \text{side length of the scanned area}$.
- Magnification is proportional to the applied voltage ($M \uparrow$ with $\uparrow V$)
- Magnification is inversely proportional to the working distance (WD) ($M \propto 1/WD$)
Electron Beam – Specimen Interaction

- As the beam strikes each point on the specimen, various responses are produced due to specimen - electron interactions.

- In almost all types of electron microscopes primary electrons enter the specimen and the same or different electrons leave it again to form the image.

- Several of these responses can be collected by suitable detectors, amplified and displayed in the CRT to form an image (or for chemical analysis).

The types of signals produced

- **X-rays**
  - Through thickness
  - Composition info

- **Primary backscattered electrons (BSE)**
  - Atomic number
  - Topographical/Imaging

- **Cathodoluminescence**
  - Electrical information

- **Auger electrons**
  - Surface sensitive
  - Composition information

- **Secondary electrons (SE)**
  - Topographical/Imaging

- **Specimen current**
  - Electrical current
Interaction Volume

- Electron penetration volume and depth depend on:
  - **Electron beam energy**
    - More energy, deeper penetration and larger interaction volume
  - **Atomic number (Z) of specimen**
    - Higher Z, lower penetration and volume

Effect of accelerating voltage on size of interaction volume specimen

Voltage decreases
Effect of atomic number on size of interaction volume specimen

- High voltage:
  - Low atomic number
  - Medium atomic number
  - High atomic number

- Low voltage:
  - Low atomic number
  - High atomic number
Types of Electron Scattering

There are two types of electron scattering when the electron beam strikes the specimen:

1. Elastic Scattering
   - Electrons retain all of their energy - backscattered electrons (BSE)

2. Inelastic Scattering
   - Electrons lose energy by interacting with the specimen – Secondary electrons (SE) and X-rays

SECONDARY ELECTRONS (SE)

- SE have very low energy (< 50 eV) compared to 30 KV of the electron beam
- They are travel very slowly as they leave the specimen
- The SE are created throughout the primary excitation volume, but if they are near the surface, they can escape
- Since they have low energy, deeper ones are easily absorbed (other interactions)
SECONDARY ELECTRONS DETECTION

- The SE are detected by a detector known as the Everhart-Thornley detector (1960).
- **Scintillator**
  - When the e- strike the scintillator, it produces light
- **Light guide**
  - Transport light to PMT
- **Photomultiplier (PMT)**
  - Amplify light and
  - Convert it to electrical signal

Number of SE that escape from the sample depends on the sample surface
**BACKSCATTERED ELECTRON (BSE) DETECTION**

- The BSE have energies > 50 eV
- They travel in straight lines
- They have weaker signal than SE
- They are detected by using purpose built backscattered electron detectors.

**BSE image:** Provides the atomic number contrast (also lower resolution of topographic contrast).
EFFECT OF MICROSCOPE VARIABLES

High resolution
High
Accelerating Voltage
Clear surface structures
Less damage
Less charge-up
Less edge effect
Low
Unclear surface structures
More edge effect
More charge-up
More damage
Low resolution

Metal

Polymer
EFFECT OF BEAM (SPOT) SIZE ON IMAGE QUALITY

Better Resolution

Bigger spot (Beam) size

Smaller spot (Beam) size

Figure 5.22 Spot size and resolution. The diameter of the beam of electrons as it scans the sample, called the spot size, is directly related to the resolution. In general, the smaller the spot, the greater the resolution.
**Means To Improve Resolution:**

(i) reducing spot size by using different types of filament.
(ii) using optimum accelerating voltages.
(iii) changing the angle of tilt (i.e. emissive area hence number of SEs).
(iv) using high beam current
(v) short working distance
(vi) using secondary electron (SE) image, as the optimum resolutions are:

- 5 nm for SE
- 25 nm for BSE and 2 μm for X-ray mapping.

(vii) long exposure times, leading to more electrons being detected.
(viii) reducing lens aberrations to minimum.
Higher voltage will provide better resolution (since high voltage generates shorter wavelength of electrons)

However, higher voltage also causes an increase in the interaction volume: this will reduce resolution
Smaller electron beam angle increases the interaction volume: thus reducing resolution.

DEPTH OF FIELD (DOF)

- The DOF is increased:
  1. By reducing the final lens aperture
  2. By increasing the working distance (lower the specimen: increase the distance between the final lens and the specimen)
Increasing the DOF by reducing the final lens aperture

Aperture size vs. Resolution and DOF

Increasing the DOF by increasing the WD

WD vs. Resolution and DOF
Means to improve depth of field:

(i) using large spot size.
(ii) using long working distance.
(iii) using small final aperture size.

VARIABLE PRESSURE DETECTOR (VPSE)

- Variable pressure technology enables investigating non-conducting specimens without prior preparation.
Non-conducting material: charging effect, difficult to image

Variable Pressure Technology:
The VP system operates to neutralise the surface by emitting a gas into the sample chamber

SEM with VPSE detector
SPECIMEN PREPARATION FOR SEM

- Image quality can be limited by specimen preparation
- There are 3 requirements for preparing specimens for SEM

1. Remove all water, solvents, or other materials that could evaporate while in the vacuum
2. Firmly mount the specimen
3. Non-metallic samples (non-conductive) should be coated so they are electrically conductive. Metallic samples can be directly placed into the SEM. (non-conducting materials will lead to charging: deflection of SE, beam deflection and SE bursts)

Note: conditions 1 & 3 are valid for conventional SEM's only