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Materials Characterization Methods

Seventh Session (Electron Microscopes)

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Humans use the sense of sight to study the microscopic structure of materials. The human eye can recognize the external structure of materials by recording the reflected light from them, but the eye has only a certain limit of detection and reception in relation to its observations. This limitation can be overcome by using auxiliary interface tools.

One of these tools, first used in the 1940s, is Electron Microscope.



In electron microscopes, electron radiation is used to study the surface and structure of the sample.

While in optical microscopes, the light beam is responsible for this task.



# **Depth of Field**



- Depth of field is the extent to which the focus is in front of or behind the main subject.
- ✓ Depth of field represents the change in depth of the sample to achieve acceptable focus.
- ✓ In electron microscopes at a given magnification, the depth of field is about 500 times greater than in light microscopes.



# **Resolution Limit**



- The Resolution limit is the smallest distance between two points that can achieve acceptable focus.
- The lower the resolution limit, the smaller the dots can be  $\checkmark$ detected at higher magnifications.
- **In Optical Microscope:**

**AR: Resolution Limit** 

**α: Convergence Angle** 

**λ: Wavelength** 



# **Resolution Limit**



**In Electron Microscope:** 

$$\Delta R = \frac{0.61\,\lambda}{\alpha}$$

Because electron microscopes work in vacuum, the refractive index of light is one (n=1).

In electron microscopes, the refractive angle is very small.

#### $\sin \alpha = \alpha$

The wavelength of light is about 500 nm and the electron wavelength is about 0.002 - 0.02 nm.



## **Components of Electron Microscopes**



The most important components of electron microscopes are:

✓ Electronic Gun (Source of Electron Production)
 ✓ Magnetic Lenses and Apertures
 ✓ Pumps and Vacuum System
 ✓ Detectors



- The most common way to separate electrons from a material that can accelerate them toward a sample is thermal emission from a heated element (thermionic sources: electron emission by heat).
- ✓ High melting point tungsten (3660 K) is a suitable material as a source of electron production.
- ✓ Because the current density of the electron source depends on the melting point, at temperatures above 2700 K, a tungsten wire emits large amounts of light and electrons.

#### **Electron Gun**



The tungsten wire used is in the form of a spiral filament with a diameter of about 0.1 mm, a V-shape wire strand or a wire whose end is sharpened to a point.



#### **Electron Gun**

- Tungsten heats up by passing electric current and acts as a cathode and is kept at a negative potential relative to the anode.
- Electrons from the filament
  in high heat effect quickly
  towards the anode are
  accelerated and a beam of
  high energy electrons emits.



## Electromagnetic Lens



- ✓ Electromagnetic lenses are used to focus the electron beam and reduce its diameter.
- These lenses consist of a soft magnetic core with a hole in the middle and a set of copper coils that create a magnetic field in the core by passing current through this coil.
- ✓ The electrons pass through the lens core and are affected by the magnetic field, converging towards the lens axis and concentrated in its focus.

## Electromagnetic Lens









## **Objective Lens and Aperture**



- An objective lens is a lens that is adjacent to a sample and is usually the most powerful microscope lens.
- ✓ The objective lens is used to create a clear image with the desired magnification and the formation of diffraction patterns.
- The aperture is used to reduce or adjust the exposure angle and can be used to control the resolution limit, depth of field and focus of the beam diameter.
- ✓ The aperatures are usually circular and placed inside a metal disk (molybdenum and platinum).

## **Objective Lens and Aperture**





## Pump and Vacuum System



- Electrons are dispersed by atoms, so to prevent them from being dispersed by air molecules or impurities (hydrocarbons and oils), All components such as electron gun, lenses, and sample must be vacuumed.
- ✓ Types of Vacuum:
- Approximate Vacuum: 100 to 10<sup>-1</sup> Pa
- Low Vacuum: 10<sup>-1</sup> to 10<sup>-4</sup> Pa
- High Vacuum: 10<sup>-4</sup> to 10<sup>-7</sup> Pa
- Very High Vacuum: above 10<sup>-7</sup> Pa

#### Pump and Vacuum System



- Mechanical or rotary pumps are used for approximate vacuum.
- Rotary pumps, which are also known as vane pumps, work by applying oil pressure to an offcenter vane that simply moves air by rotating the said vane.



## Pump and Vacuum System



- ✓ In electron microscopes, first mechanical and diffusion pumps and then turbo and ionic pumps are used for different parts.
- ✓ Diffusion, turbomolecular and ion pumps are used for high vacuum.
- Ion pumps are generally turned on after diffusion-type operation and the vacuum reduced to less than 10<sup>-5</sup> Torr and connected directly to the electron gun chamber.





- The diffusion pump is placed directly below the electron microscope column and performs the final step of creating a vacuum with the support of the mechanical pump.
- In diffusion pumps, oil vapor is used to create a vacuum, which leads to some oil vapor remaining in the chamber.





 Turbomolecular pumps are of great importance because they have a high ability to minimize contamination in the electron microscope. This is especially important when high-precision analysis of the elements in the sample is required.

