CHARACTERIZATION TECHNIQUES

- **Introduction:**
- Characterization refers to the study of material features such as its composition, structure, and its properties like physical, electrical, and magnetic.
- The structure, morphology, and other properties of nanoparticles are characterized by the following techniques:

  (i) X-ray Diffraction (XRD)

  (ii) Scanning Electron Microscope (SEM)

  (iii) Transmission Electron Microscope (TEM)

(i) XRD(X- RAY DIFFRACTION)

XRD : XRD is a technique that uses X-rays to determine the structure of a material.

- It is a powerful technique to identify the crystalline phase present in a material and to measure the phase composition, preferred orientation, grain size, strain size, and defect structure of these phases

Principle:

- The principle of XRD is based on constructive interference by X-rays and the crystalline structure of a material & Bragg's law.

Construction / Experimental arrangement:

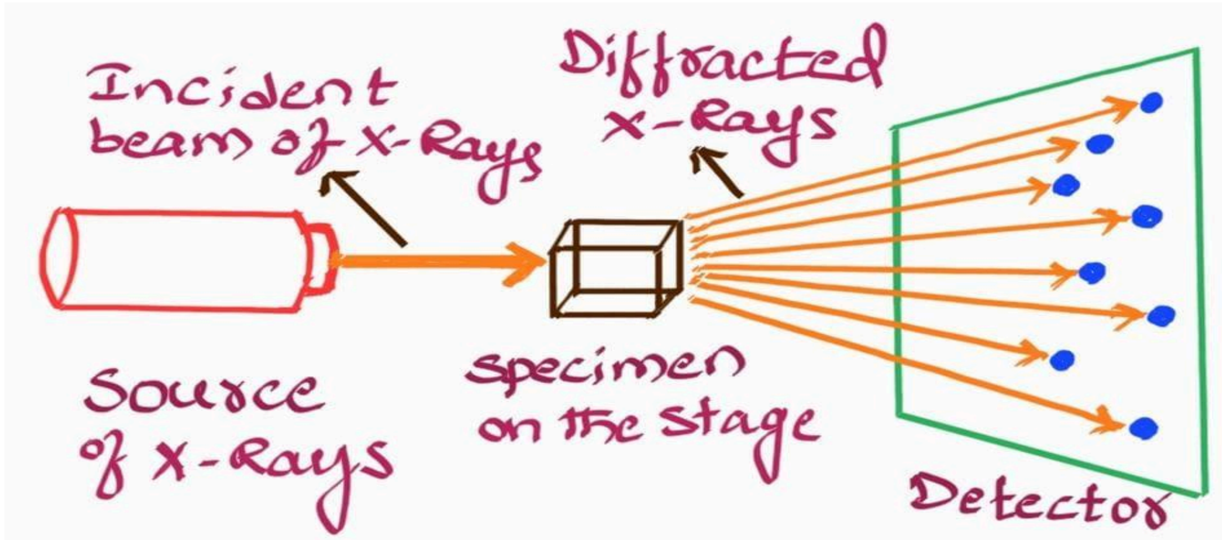
- XRD instrumentation consists of 4 main components such as

  (i) Source of X-rays

  (ii) Specimen stage (crystal)

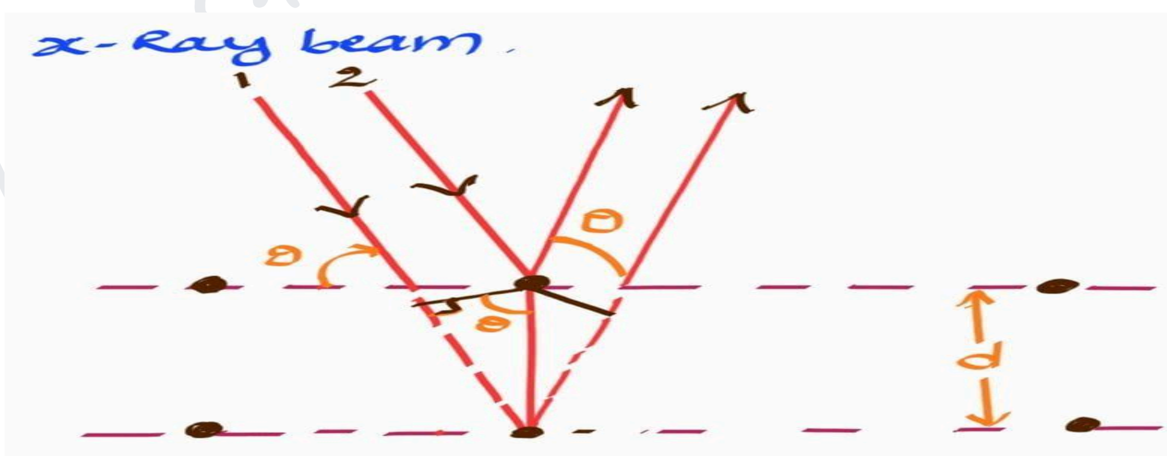
  (iii) Diffracted X-rays and

  (iv) Detector.



Working process:

- A beam of X-rays of wavelength λ is directed to the crystal by the source, at an angle θ to the atomic planes.
- The interaction between X-rays and the electrons of the atoms is visualized as a process of X-rays reflections by the atomic planes.
- The atomic planes allow a part of the X-rays to pass through and reflect the other part, the incident angle θ being equal to the reflected angle called Bragg's angle. There is a path difference between rays reflected from plane 1 and the adjacent plane 2 in the crystal.
- The two reflected rays will reinforce each other only when this path difference is equal to an integral multiple of the wavelength λ.
- If d is the interplanar spacing, the path difference is twice the distance dsinθ.



Analysis: XRD analysis is based on Bragg's Law and Bragg's angle.

- Bragg's Law given by $n\lambda=2d\sin\theta$

Where: $n\rightarrow$ An integer

$\lambda\rightarrow$ wavelength of X-rays

$\theta\rightarrow$ The diffraction angle

APPLICATIONS:

- Phase Identification in nanomaterials, nanoparticles, nanowires and thin films by XRD
- Crystal structure analysis is used to understand the physical, chemical properties of nanomaterials and their behaviour by XRD
- Grain size & Strain analysis of nanomaterials by XRD
- Texture analysis & Preferred orientation of crystalline grains in nanomaterials by XRD
- Quantitative phase analysis of phase abundance in nanomaterials is valuable for assessing phase purity, phase transformation etc by XRD
- Stress & Texture mapping done by XRD techniques, provides local structure property relationships and investigate defects etc
- Catalysis & Energy materials are developed by XRD, in battery electrodes, photovoltaic devices, catalyst nanoparticles etc.

SCANNING ELECTRON MICROSCOPE(SEM)

Introduction:

- In 1938, Von Ardenne described the theoretical principles of SEM but true SEM was first developed by Zworykin, Hillier & Snyder in 1942.

SEM: A Scanning Electron Microscope (SEM) is a type of electron microscope that produces images of a sample by scanning its surface with a high-energy beam of electrons.

Principle:

- The SEM works by scanning the surface of a sample using a high-energy beam of electrons. This interaction generates various signals, such as secondary electrons, backscattered electrons, and characteristic X-rays, which are used to create detailed images of the sample.

Main components of SEM:

1.Electron gun

2.Anode

3.Magnetic lens

4.Scanning coils

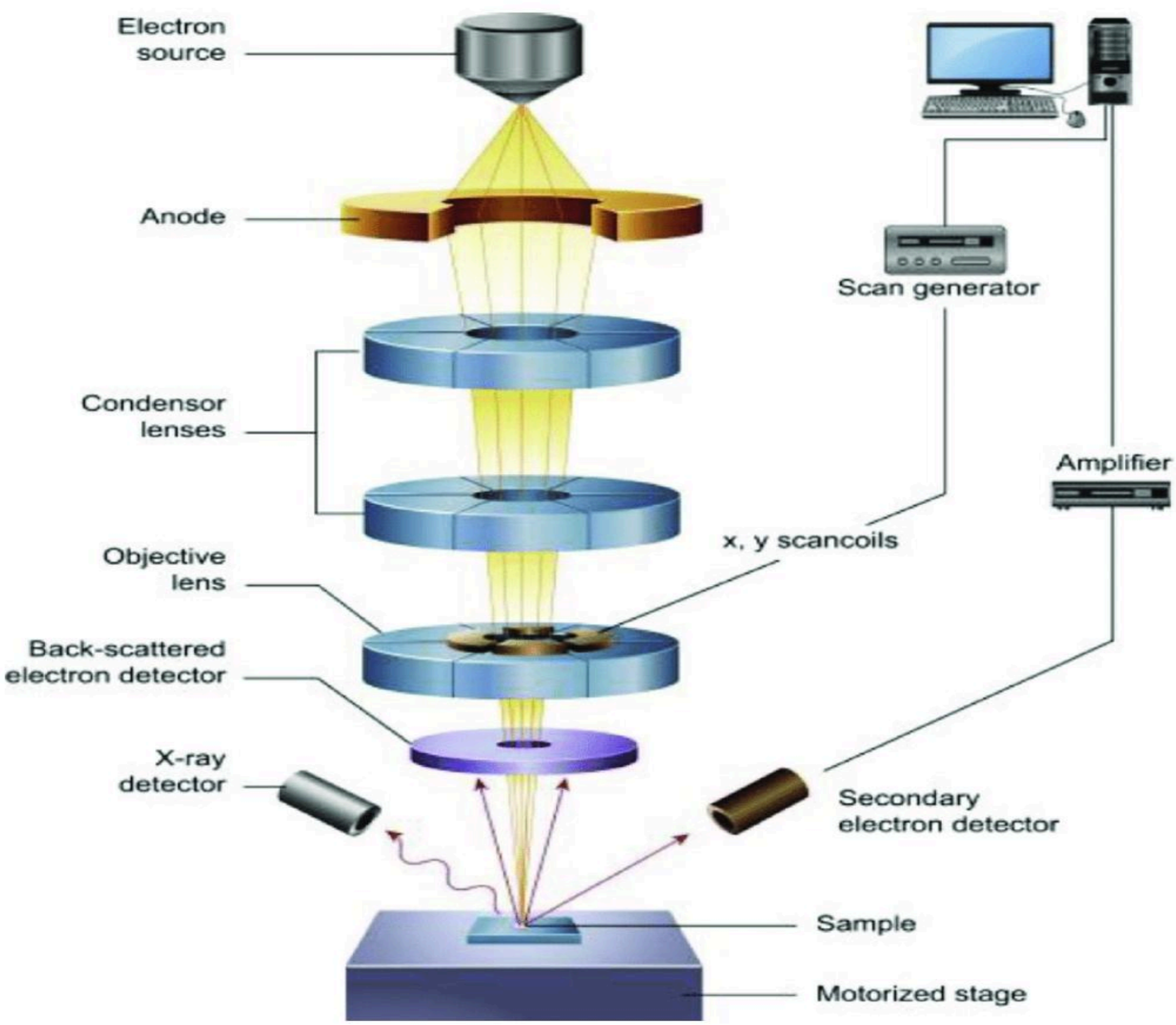
5.Objective lens

6.TV scanner

7.Detectors

8.Specimen stage

Diagram / sketch:



Construction & Working:

- The virtual source at the top represents the **Electron-gun** which produces a stream of high energy monochromatic electrons.
- These electrons are emitted from a filament (cathode) made up of a thin tungsten wire by heating the filament at high temperature.
- Now, these electrons are attracted and travel through **Anode** due to directionality.
- Two magnetic lenses are used as **Condenser lenses** to convert the diverging electron beam into a fine pencil beam and the condenser lens eliminates the high angled electrons from the beam so the electron beam becomes thin and coherent.
- **Scancoils x,y** are used to deflect the electron beam to scan the sample.
- The **Objective lens** is used to focus the scanning beam on a desired spot on the sample.
- When the high energy electron beam strikes the sample, some electrons are scattered due to elastic scattering called **Back scattered electrons**, some electrons are knocked off from the surface called **Secondary electrons** and some electrons penetrate deep into the inner shells of the sample atoms to knock off inner shell electrons due to which X-rays are produced.
- The intensities of secondary electrons, backscattered electrons and X-rays recorded using detectors and the signals are amplified by **Amplifiers** and display the images on a **TV** scanner/monitor.
- This process is repeated several times (up to 30 times per second) to get accurate results.
- To record the SEM image, in the past, the SE image appearing on the LCD was photographed with a camera. But recently, the image has been recorded in a digital format called **electronic file** and it is easier to process and convert to send or receive image information.

Characterization of Sample using SEM:

- Topography: To study surface features & texture
- Morphology: To study the shape, size & arrangement
- Composition: To study the ratio of elements & compounds
- Crystallographic information: Arrangement of atoms & their order in the crystal

Advantages:

- High resolution 3D images
- Chemical analysis
- Versatile information
- Applications in biology, material science, forensics & microchip assembly.

Disadvantages:

- Expensive

- Sample must be solid
- Require strong insulator coating
- Require specimen size to fit in chamber
- Require low pressure / wet organic materials
- Special training is required.
- Require special operating environments like vibration free rooms & electromagnetic elements.

Applications:

- SEMs are versatile instruments used in a wide range of scientific and industrial applications for imaging & analysing the surface morphology of materials at high magnification. Some applications are:

    1.Material science

    2.Nanowire for gas sensing

    3.Semiconductor inspection

    4.Microchip assembly

    5.Forensic investigations

    6.Biological Sciences

    7.Soil & Rock sampling

    8.Medical[1] investigations

TRANSMISSION ELECTRON MICROSCOPE (TEM)

Introduction: In 1931, Ernst Ruska and Max Knoll developed the first Transmission Electron Microscope but high-resolution TEM commercialized by RCA Lab in 1940. In 1986, Ruska was awarded the Nobel Prize in Physics for his work on the TEM.

- TEM is an electron microscope that uses a beam of electrons to create an image of a thin specimen, such as a tissue section or molecule.
- TEM is used to study the physical, chemical, and structural properties of materials at the nanoscale.

Principle:

- It works on the principle that a beam of high-energy (velocity) electrons accelerated under vacuum, focused by a condenser lens onto a specimen, and the emergent electron beam is focused by the objective lens. The final image forms on a fluorescent screen or camera for image viewing.

<u>Main components of TEM</u>

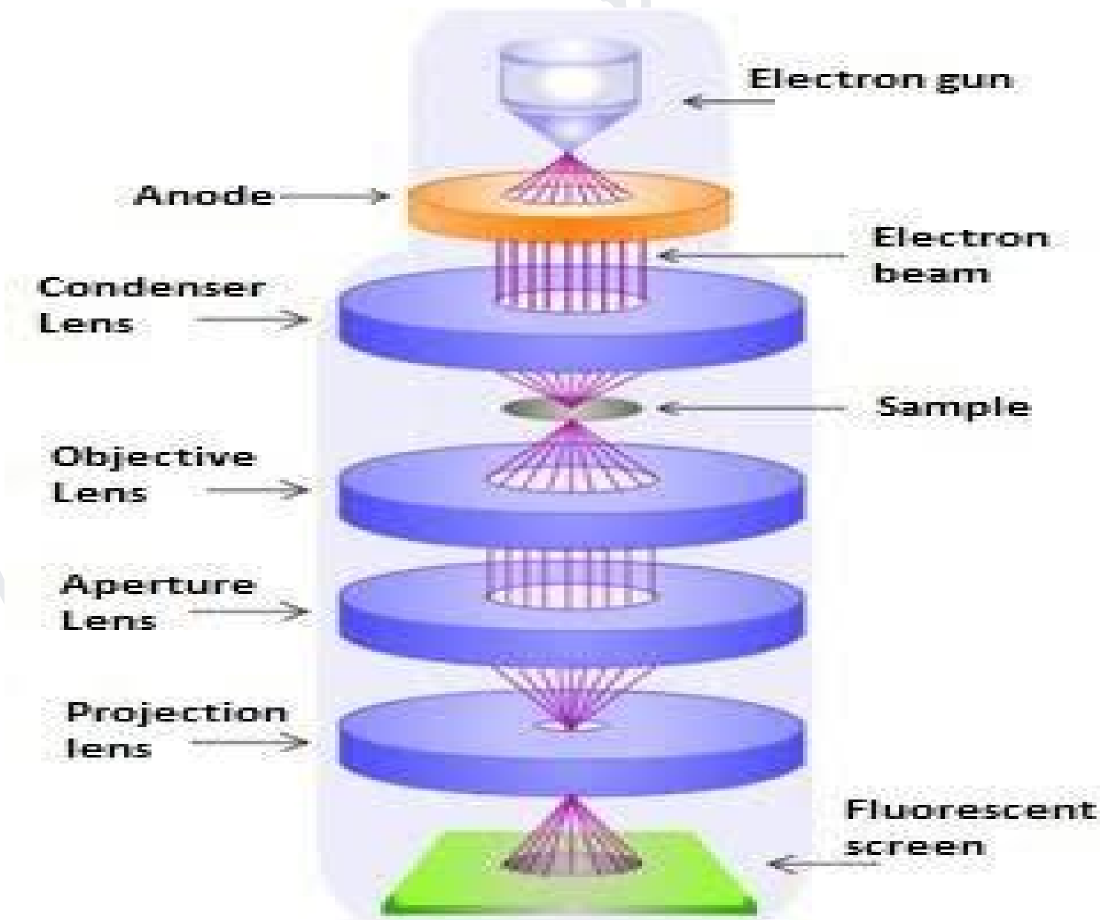
1. Electron gun
2. Anode
3. Condenser lens
4. Scanning coils
5. Specimen sample
6. Objective lens

7.Aperture

8.Projection lense

9. Fluorescent screen

<u>Diagram / sketch:</u>



Construction & Working:

- The virtual source at the top represents the E**lectron gun**, which produces a high energy/velocity electron beam. These electrons are emitted from a small area of filament due to Cathode(-ve potential of the electrode).
- These emitted electrons are attracted and travel through anode, there by directionality (parallel electron beam).
- These **parallel beams** of high energy electrons accelerated under vacuum, focused by **condenser lens** (electromagnetic bending of electron beam) onto specimen/sample.
- The **objective lens** focuses the transmitted electrons from the sample into an **Aperture len**s.
- The aperture lens magnifies and focuses the image or diffraction pattern produced by the objective lens. And forms the magnified image on the object plane of the projector lens.
- The **projection lens** then projects the final image onto the viewing **Fluorescent screen** /camera.

Characteristics of Sample using TEM:

1. Sample used in Transmission Electron Microscope have several characteristics.

2.Thin & Vacuum stable

3.Free of contamination

4.Contrast-enhanced

Advantages:

1.High resolution imaging

2.High magnification

3.Versatile applications

4.Provides structural, morphological, and compositional information

Disadvantages:

1.Sample preparation and Vacuum requirement

2.Image is 2D projection

3.Expensive and requires specialized expertise