Characterization of nanomaterials

Nano = Size10-9 (extremely small) Particle
= Small piece of matter

Nanoparticle is a microscopic particle whose size is measured in nanometers (nm).

These particles can be spherical, tubular, or irregularly shaped and can exist in fused, aggregated or agglomerated forms.

INTRODUCTION

- Characterization refers to the study of material's features such as its composition, structure,& various properties like physical, electrical, magnetic etc.
- Nanomaterials, dispersed in the form of colloids in solutions, particles (dry powders) or thin films, are characterized by various techniques.
- Although the techniques to be used would depend upon the type of material and information one needs to know, usually one is interested in first knowing the size, crystalline type, composition, thermal, chemical state, and properties like optical or magnetic properties.

- Optical Microscopes
- Confocal Microscope
- Electron Microscopes
  - Scanning Electron Microscope
  - Transmission Electron Microscope (TEM)
- Scanning Probe Microscopes (SPM)
  - Scanning Tunnelling Microscope
  - Atomic Force Microscope
  - Scanning Near-Field Optical Microscope (SNOM)
- Diffraction Techniques
  - X-Ray Diffraction (XRD)
- Spectroscopies
  - UV-Vis-NIR Spectrometer
  - Infra Red Spectrometers
  - Dispersive Infra Red Spectrometer
  - Fourier Transform Infra Red Spectrometer
  - Raman Spectroscopy
  - Optical (Ultraviolet-Visible-Near Infra Red) Absorption Spectrometer
- Magnetic Measurements
  - Vibrating Sample Magnetometer (VSM)
- Mechanical Measurements

VARIOUS CHARACTERISATION METHODS AND THEIR USES

Acronym	Full Form	Uses
SEM	Scanning Electron Microscope	Imaging/ Topology/ Morphology
TEM	Transmission Electron Microscope	Imaging/ particle size and shape analysis
AFM	Atomic Force Microscopy	Imaging/ Topology/ surface Structure
STM	Scanning Tunnelling Microscopy	Topology/ Imaging/ surface Structure
AAS	Atomic Absorption Spectroscopy	Chemical Analysis
XRD	X-Ray Diffraction	Crystal Structure

MICROSCOPY

- What is microscopy?
- "observation and examination of minute objects which will provide a magnified image of an object not visible to the naked eye"
- Types: 3 main types
  - Optical Microscopy
  - Electron Microscopy
  - Scanning Probe Microscopy (SPM)

MICROSCOPES

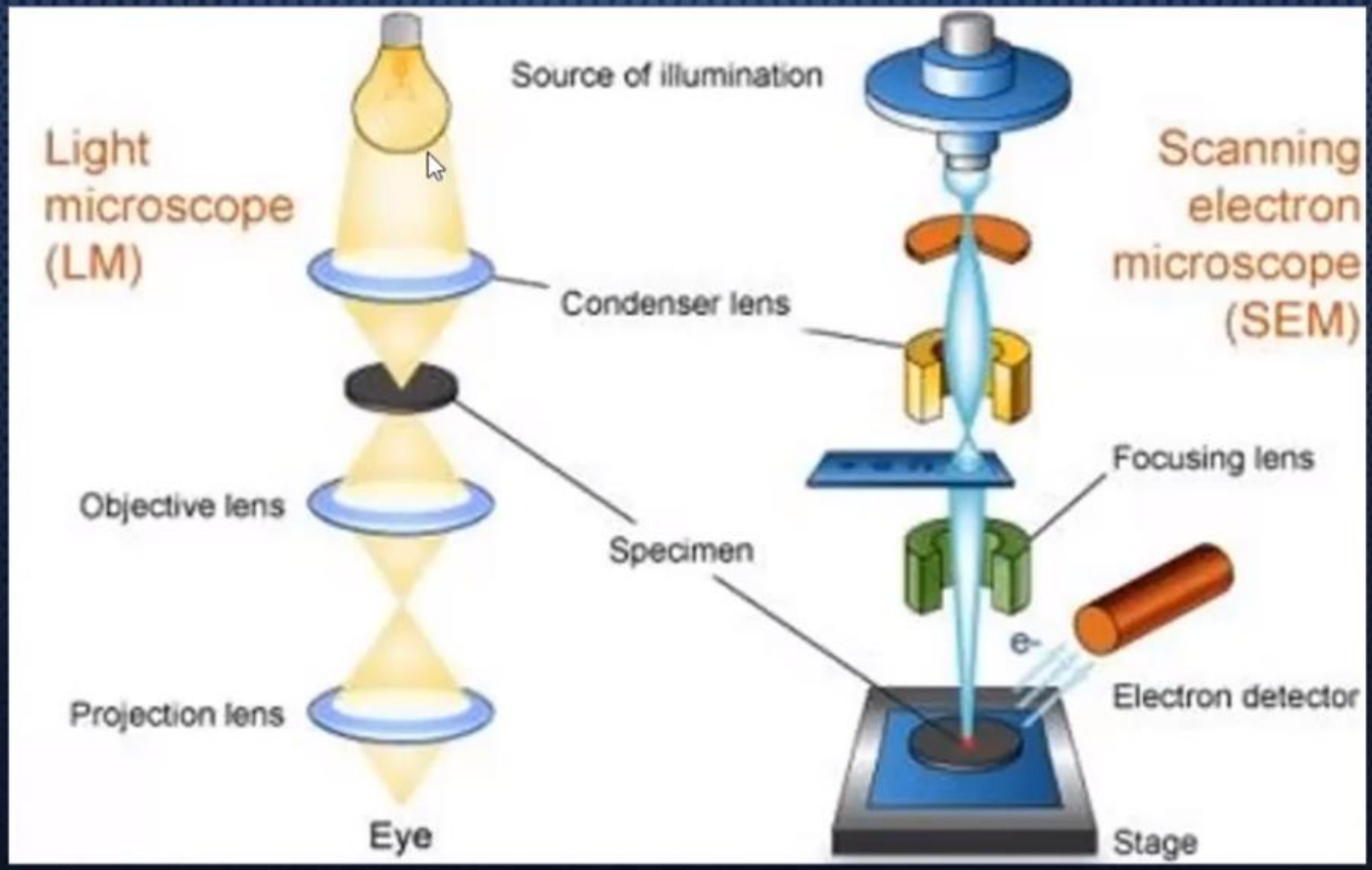
- Low dimensional materials such as quantum dots, quantum wires, quantum wells, self-assembled materials, interactions of small molecules with surfaces, multilayers etc. need special microscopes like follows:
  - Transmission Electron Microscope (TEM),
  - Scanning Tunneling Microscope (STM),
  - Atomic Force Microscope (AFM)
  - Scanning Near-Field Optical Microscope.
- However, in many instances, one inspects the sample, specially thin films, with an optical microscope, to check the quality of samples like presence of cracks, agglomeration at large scale, etc.
- Therefore we shall begin with a simple optical microscope, define some common terminologies used in microscopy analysis work and then discuss other microscopes.

ELECTRON MICROSCOPY

- Electron Microscopes are scientific instruments that use a beam of highly energetic electrons to examine objects on a very fine scale which yield the following information:

1. Topography : The surface features of an object (hardness, reflectivity etc.)
2. Morphology: The shape and size of the particles(ductility, strength, reactivity etc.)
3. Composition: The elements and compounds that the object is composed of and the relative amounts of them.
4. Crystallographic Information: How the atoms are arranged in the object.

COMPARISON OF LIGHT VS ELECTRON MICROSCOPE

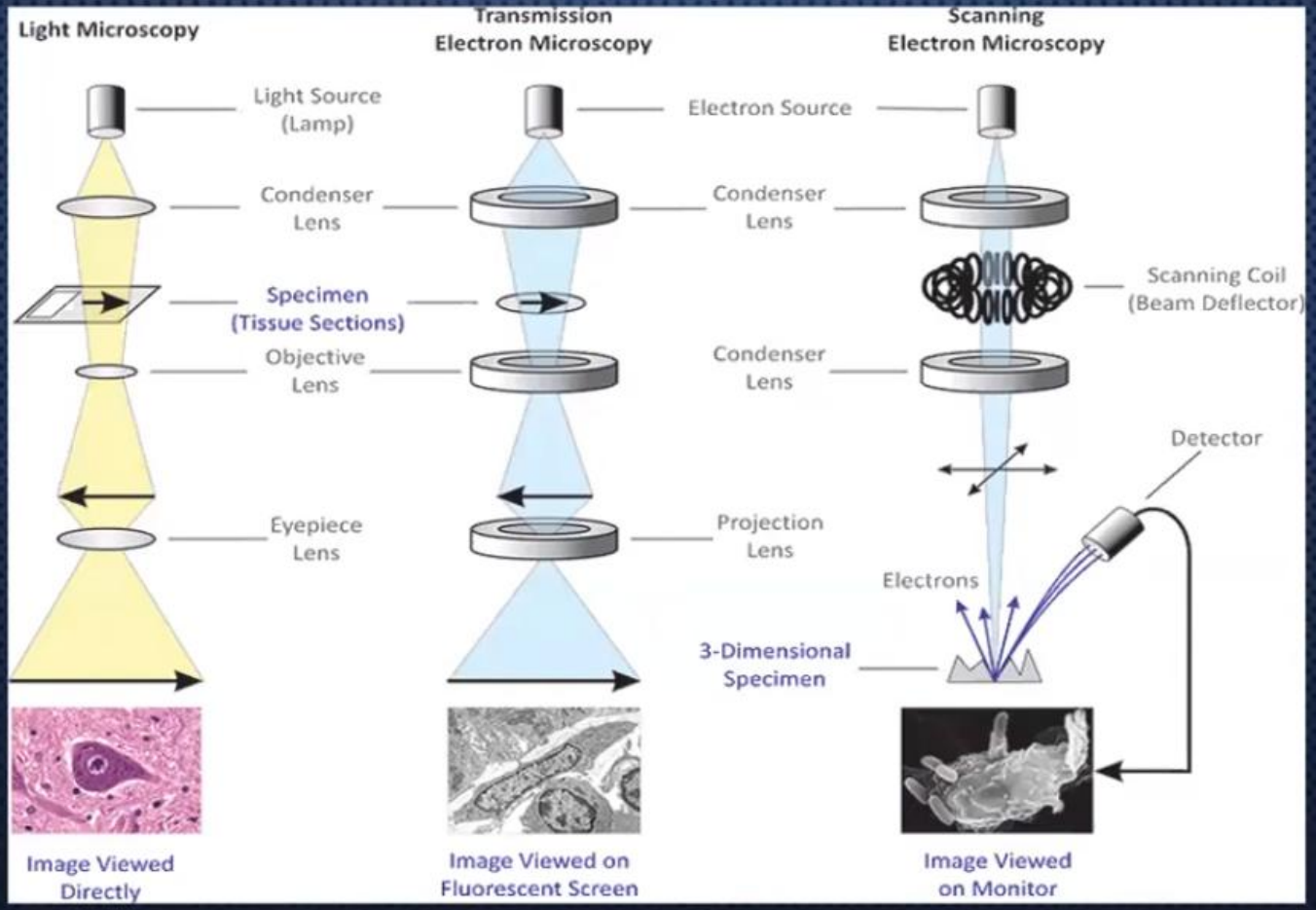


SCANNING ELECTRON MICROSCOPE

- Principle : SEM images the sample surface by scanning with a high energy beam of electrons, under high vacuum.
- Electrons interact with the atoms that make up the sample producing signals that contain information about surface topography, composition, electrical conductivity etc.
- Procedure : Samples are coated with electrically conductive materials like gold, platinum, osmium, graphite etc.
- Coating prevents static electric charge accumulation on specimen during electron irradiation.

SCANNING ELECTRON MICROSCOPE

- Alternative to coating is to increase the bulk conductivity by impregnation with osmium.
- Size range : 1-5nm in size.
- Disadvantages :
  - Sample must be dry.
  - Coating agent may change the morphology and size of the particle.
  - Provides only 2D projection.



Light Microscope:
Optical glass lens, Small depth of Field, lower magnification, do not Require vacuum, Low price.

Electron Microscope: Magnetic lens, Large depth of field, Higher magnification and better Resolution, Operates in high vacuum, High Price.

- (TEM) uses a wide beam of electrons passing through a thin sliced specimen to form an image.
- This microscope is analogous to a standard upright or inverted light microscope.
- Electrons scatter when they pass through thin sections of a specimen
- Transmitted electrons (those that do not scatter) are used to produce image
- Denser regions in specimen, scatter more electrons and appear darker
- Ultrathin sections of specimens
- Light passes through specimen, then an electromagnetic lens, to a screen or film
- Specimens may be stained with heavy metal salts