Morphological Characterization

- Scanning Electron Microscopy (SEM): Scanning Electron Microscopy (SEM) is an electron-beam-based imaging technique used to study the **surface morphology, topography, and microstructure** of materials.
- Transmission Electron Microscopy (TEM): Transmission Electron Microscopy (TEM) is a high-resolution imaging technique in which a beam of high-energy electrons is transmitted through an ultra-thin specimen to produce an image.

Scanning Electron Microscopy (SEM)

- Scanning Electron Microscopy (SEM) is an **electron-beam based imaging technique** used to study the **surface morphology, topography, particle size, shape, and distribution** of materials at micro- and nanometer scales.
- In nanomaterials research, SEM is mainly used to:

✓Observe particle shape and surface texture

✓Estimate particle size and agglomeration

✓Examine nanostructures such as rods, sheets, fibers, tubes, and porous networks

Principle of SEM

- SEM works by scanning a **focused beam of high-energy electrons** over the surface of a specimen.
- When the electron beam interacts with the sample, several signals are produced:

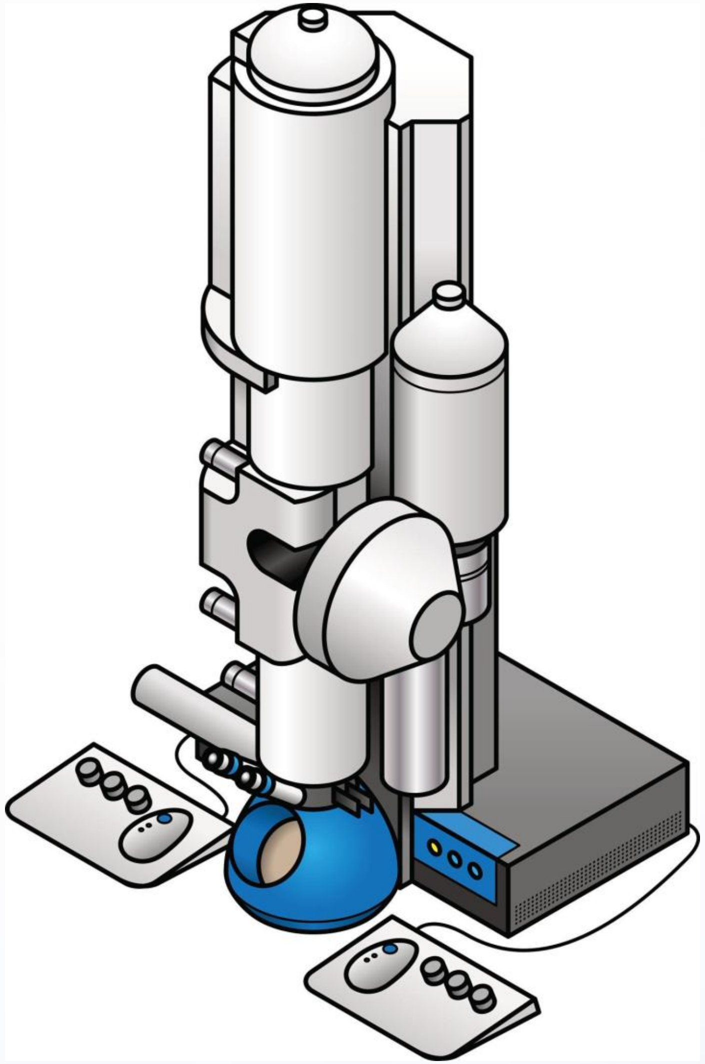
✓ Secondary electrons (SE)
✓ Backscattered electrons (BSE)
✓ Characteristic X-rays

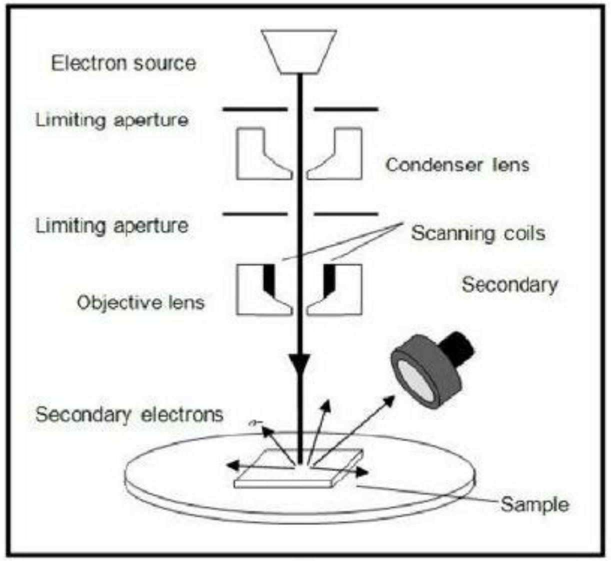
- The most commonly used signal for imaging is **secondary electrons**, which originate from the near-surface region.
- These electrons are collected by a detector and converted into an image.

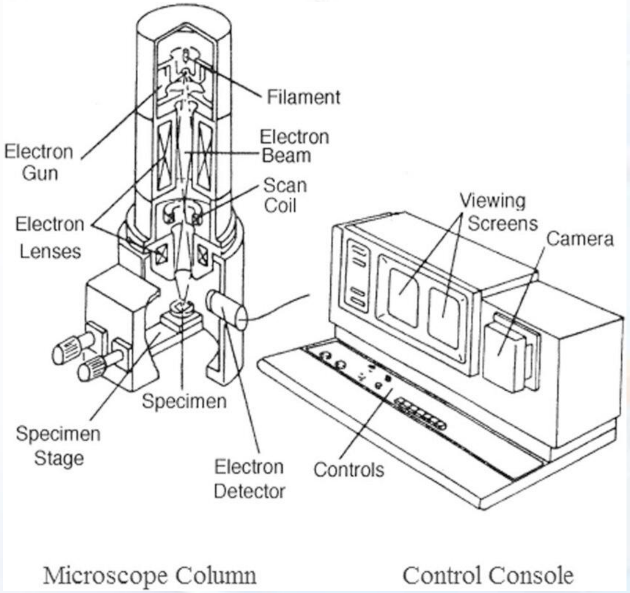
Basic SEM Instrumentation (Schematic View)

Main Components

1. Electron gun
2. Electromagnetic condenser lenses
3. Objective lens
4. Scanning coils
5. Sample chamber & stage
6. Detectors (SE, BSE, EDX)
7. Vacuum system
8. Computer & display system







Electron Gun

Generates electrons by thermionic or field emission.

Types:

- Tungsten filament
- LaB_6 filament
- Field Emission Gun (FEG)

Function: Provides a stable, high-energy electron beam.

Typical accelerating voltage: 1–30 kV

Electromagnetic Lenses & Scanning Coils

- Condenser lenses focus the beam
- Objective lens forms a fine probe
- Scanning coils move beam in raster pattern

Function: Controls beam diameter and scanning area.

Detectors

(a) Secondary Electron Detector (SED)

- Produces topographic images
- High surface sensitivity

(b) Backscattered Electron Detector (BSE)

- Provides compositional contrast
- Heavy elements appear brighter

(c) Energy Dispersive X-ray Detector (EDX)

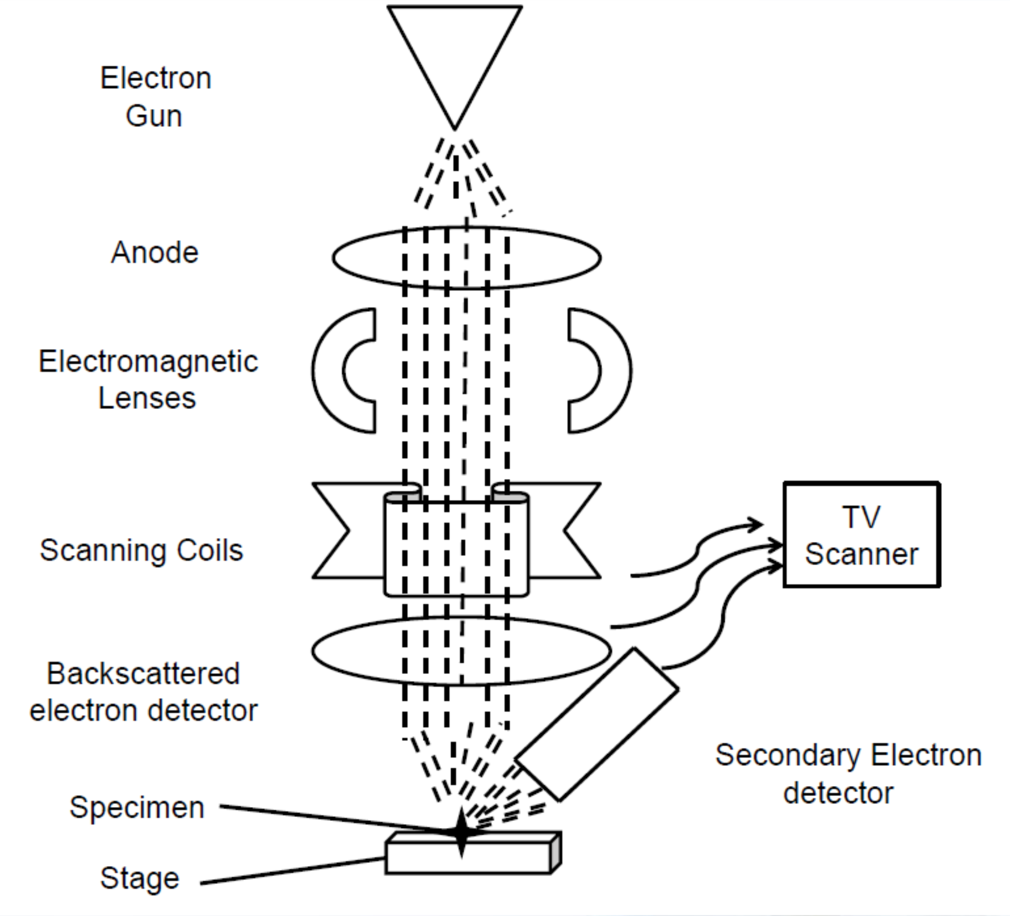
- Gives elemental composition

Sample Chamber and Stage

- Holds solid samples
- Allows movement in X, Y, Z directions
- Sample must be vacuum compatible

Working of SEM (Stepwise)

1. Electron gun emits electrons
2. Beam accelerated and focused
3. Focused beam scans sample surface
4. Secondary electrons emitted
5. Detector collects electrons
6. Signal converted to image



SEM Images of Nanomaterials

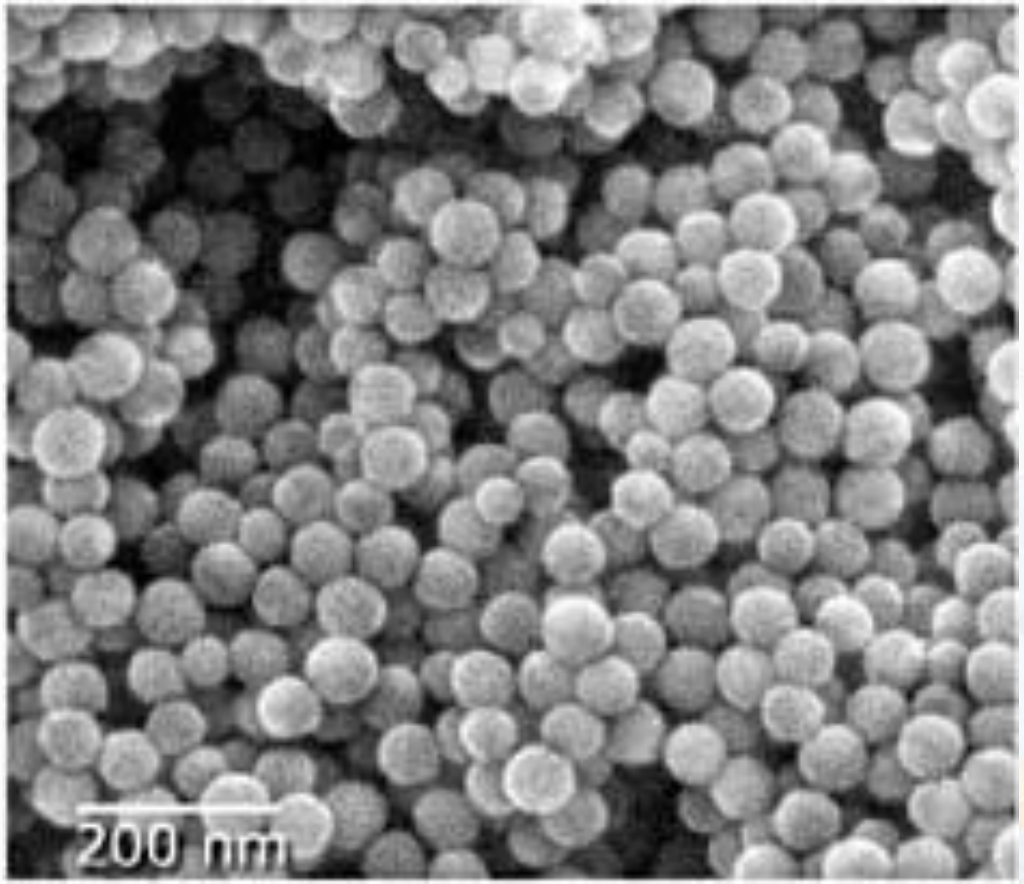
From SEM images we can observe:

•Particle shape (sphere, rod, sheet, flower-like)

•Surface roughness

•Degree of agglomeration

•Porosity



Information Obtained from SEM

✓ Surface Morphology

- Smooth, rough, porous, dense

✓ Particle Shape

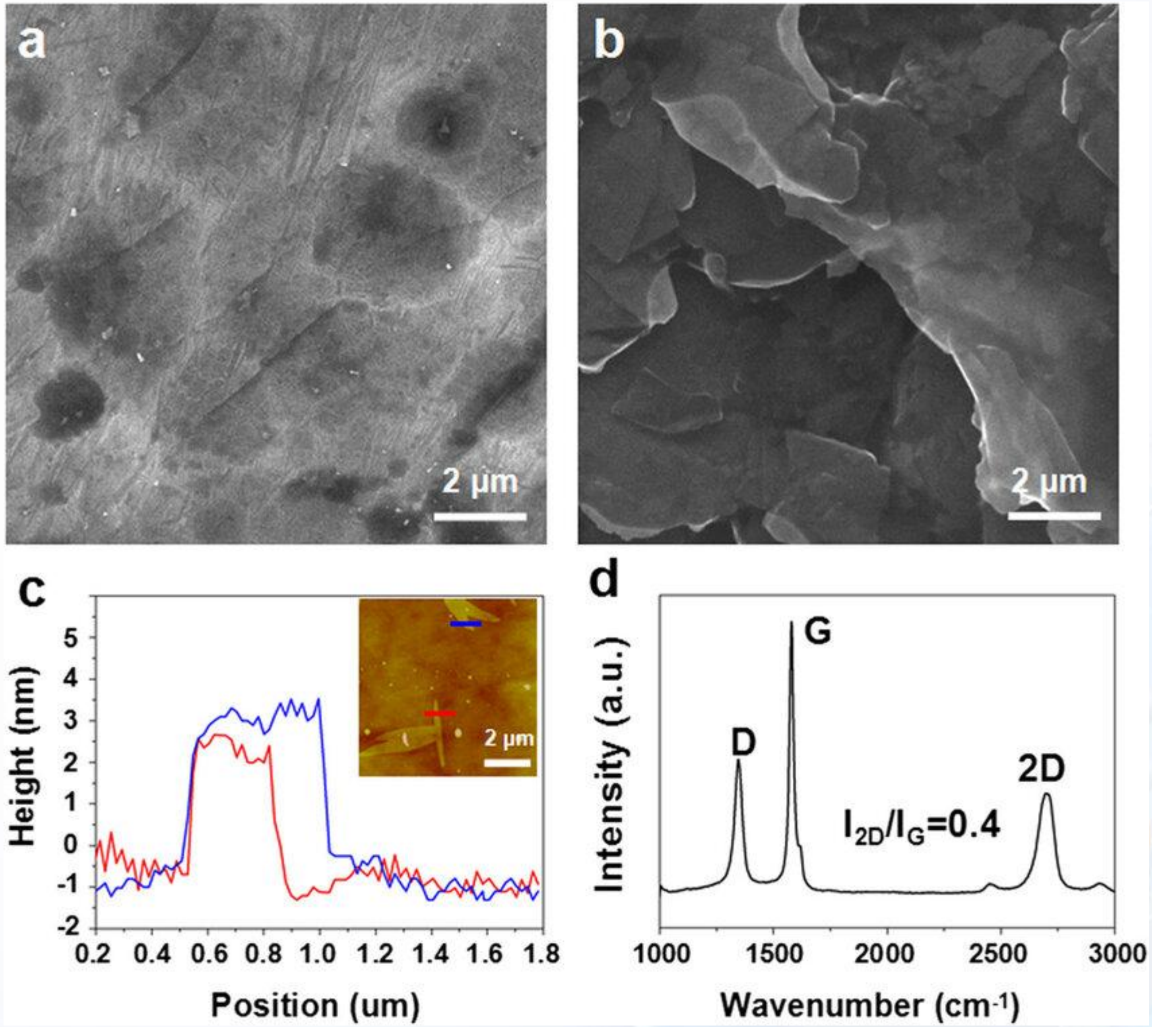
- Spherical, cubic, rod-like, tubular, plate-like

✓ Particle Size (Approximate)

- Measured using scale bar from SEM image.

✓ Agglomeration State

- Whether particles are isolated or clustered



Advantages of SEM

- Easy operation
- High depth of field
- Large area scanning
- Provides real-space images

Limitations of SEM

- Vacuum Requirement
- Lower resolution than TEM
- Cannot see internal structure
- Conductive coating may alter surface

TRANSMISSION ELECTRON MICROSCOPY (TEM) FOR NANOMATERIALS

- Transmission Electron Microscopy (TEM) is a high-resolution imaging technique that uses a high-energy electron beam transmitted through an ultra-thin sample to obtain structural and morphological information.
- TEM is one of the most powerful tools for nanomaterials because:

✓ Resolution up to ∼0.1–0.2 nm
✓ Direct visualization of nanoparticles
✓ Atomic lattice imaging possible
✓ SAED patterns available

Basic Principle of TEM

- A high-energy electron beam (100–300 kV) is generated.
- The beam is focused using electromagnetic lenses.
- Electrons pass through an ultra-thin sample (<100 nm).
- Transmitted and scattered electrons form an image.
- Since electrons have very short wavelength (de Broglie wavelength), they provide extremely high resolution.

Why TEM is Ideal for Nanomaterials

- Nanoparticles typically range from **1–100 nm**.
- TEM allows:

✓ Direct measurement of particle size
✓ Observation of shape (sphere, rod, sheet, tube)
✓ Study of agglomeration
✓ Core–shell structure identification
✓ Lattice fringe visualization

- Unlike XRD (which gives average crystallite size), TEM shows **individual particles**.

Instrumentation

1. Electron Gun

- Tungsten filament / Field emission gun (FEG)
- Produces high-energy electron beam

2. Condenser Lens

- Focuses electron beam onto sample

3. Sample Holder

- Holds ultra-thin specimen
- Sample must be electron transparent

4. Objective Lens

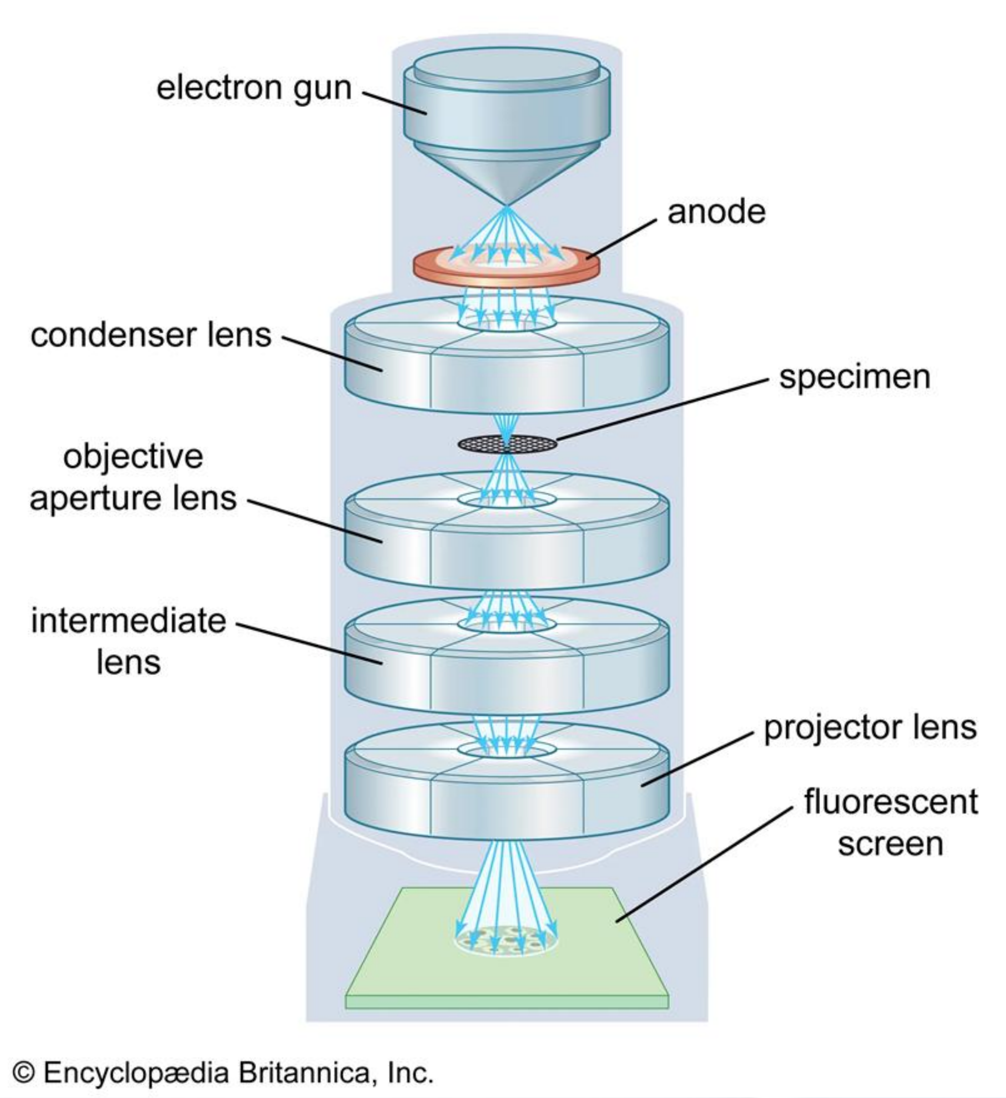
- Forms primary image
- Most important lens in TEM

5. Intermediate & Projector Lenses

- Further magnify image

6. Fluorescent Screen / CCD Camera

- Converts electron image into visible image



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Working Step-by-Step

1.Electron beam generated

2.Beam focused by condenser lens

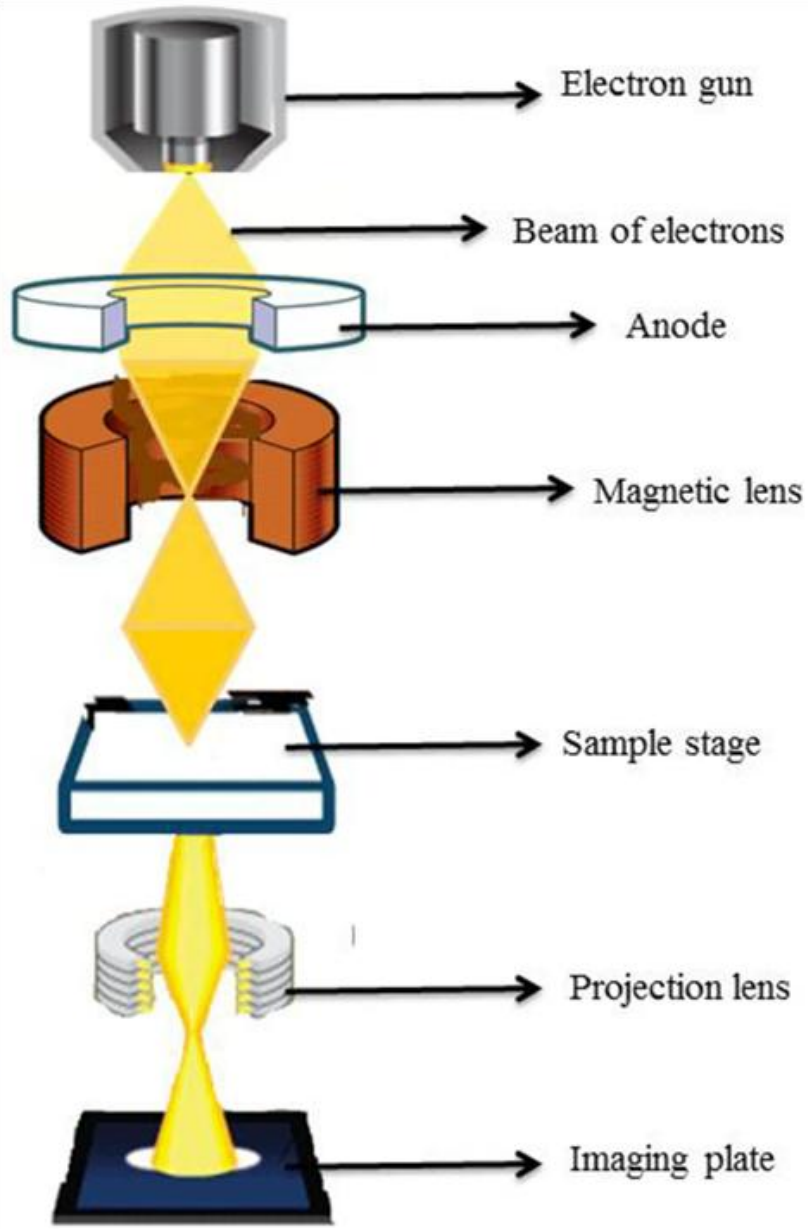
3.Beam strikes thin sample

4.Some electrons transmitted

5.Some electrons scattered

6.Objective lens forms image

7.Image magnified and detected



Types of Images in TEM

(A) Bright Field Image

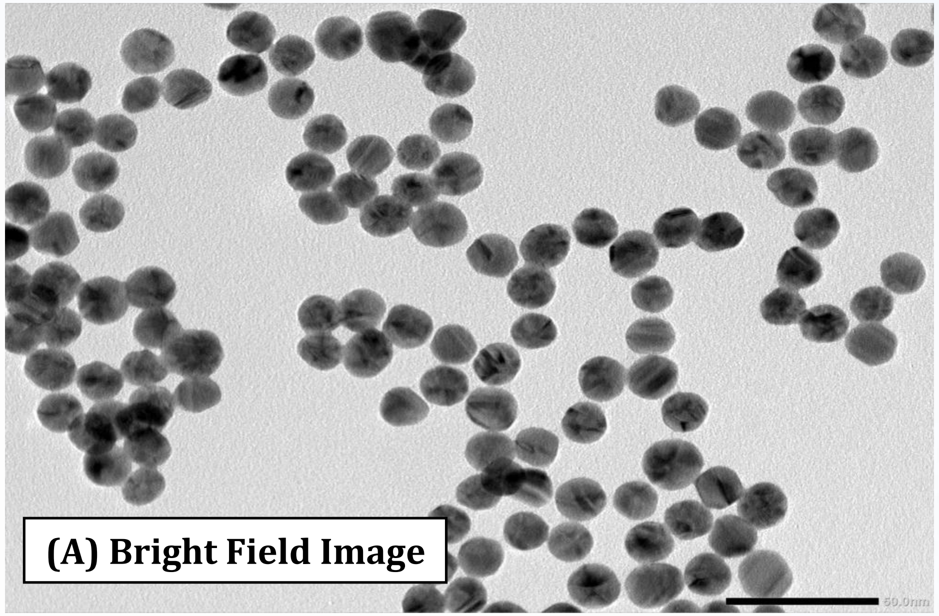
- Formed by transmitted electrons
- Dense regions appear dark

(B) Dark Field Image

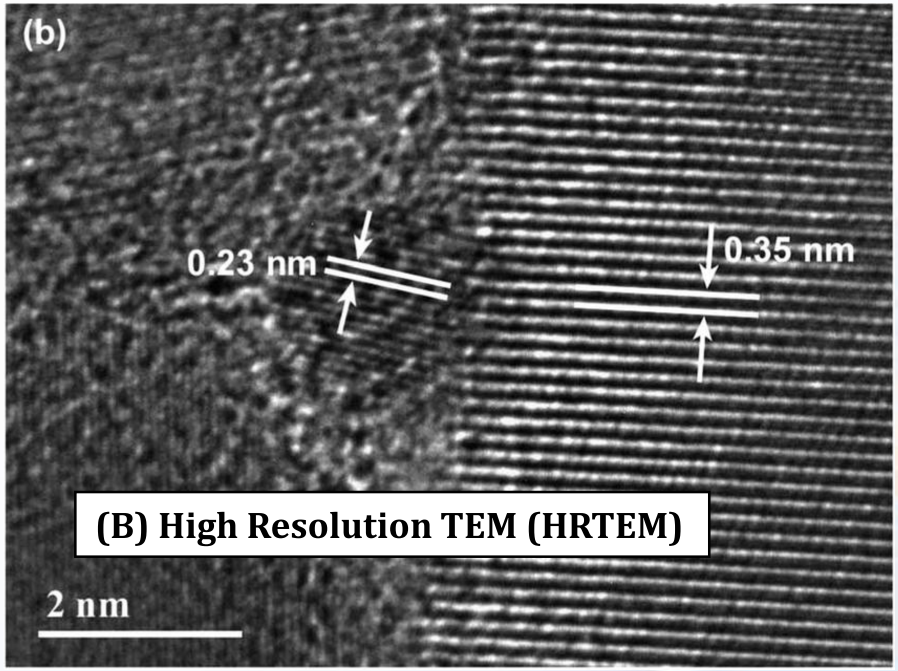
- Formed using scattered electrons
- Highlights crystalline areas

(C) High Resolution TEM (HRTEM)

- Shows lattice fringes
- Interplanar spacing can be measured
- Confirms crystallinity



(A) Bright Field Image



(B) High Resolution TEM (HRTEM)

Advantages of TEM

- Atomic Level resolution
- Direct Visualization
- SAED Possible
- Size Distribution Analysis
- Core-shell Confirmation

Limitations

Expensive Instrumentation

Complex Sample preparation

Small Sample area

Possible Beam Damage