4.4 ELECTRON MICROSCOPY

- ❖ An electron microscope is a microscope that uses a beam of accelerated electrons as a source of illumination.

- ❖ As the wavelength of an electron can be up to 100,000 times shorter than that of visible light photons, electron microscopes have a higher resolving power than light microscopes and can reveal the structure of smaller objects.

TYPES OF ELECTRON MICROSCOPE

- Transmission Electron Microscope
- Scanning Electron Microscope

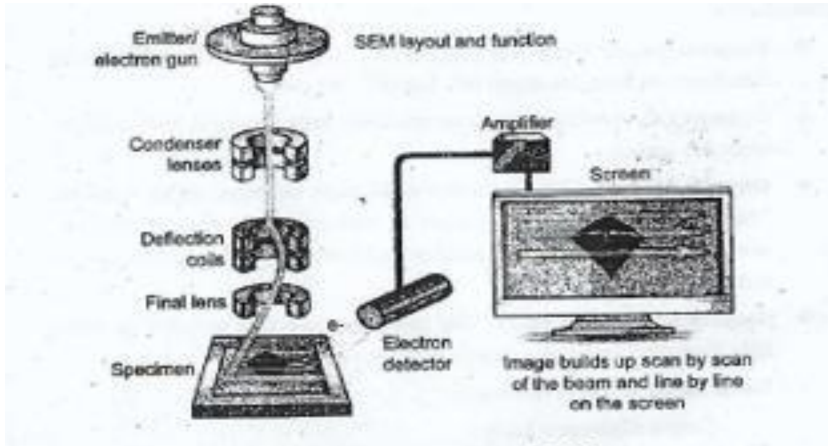
4.4.1SCANNING ELECTRON MICROSCOPE (SEM)

A scanning electron microscope (SEM) uses a focused electro probe to extract structural and chemical information point by point on the specimen. It use wide range of scale from nanometre to micrometre.

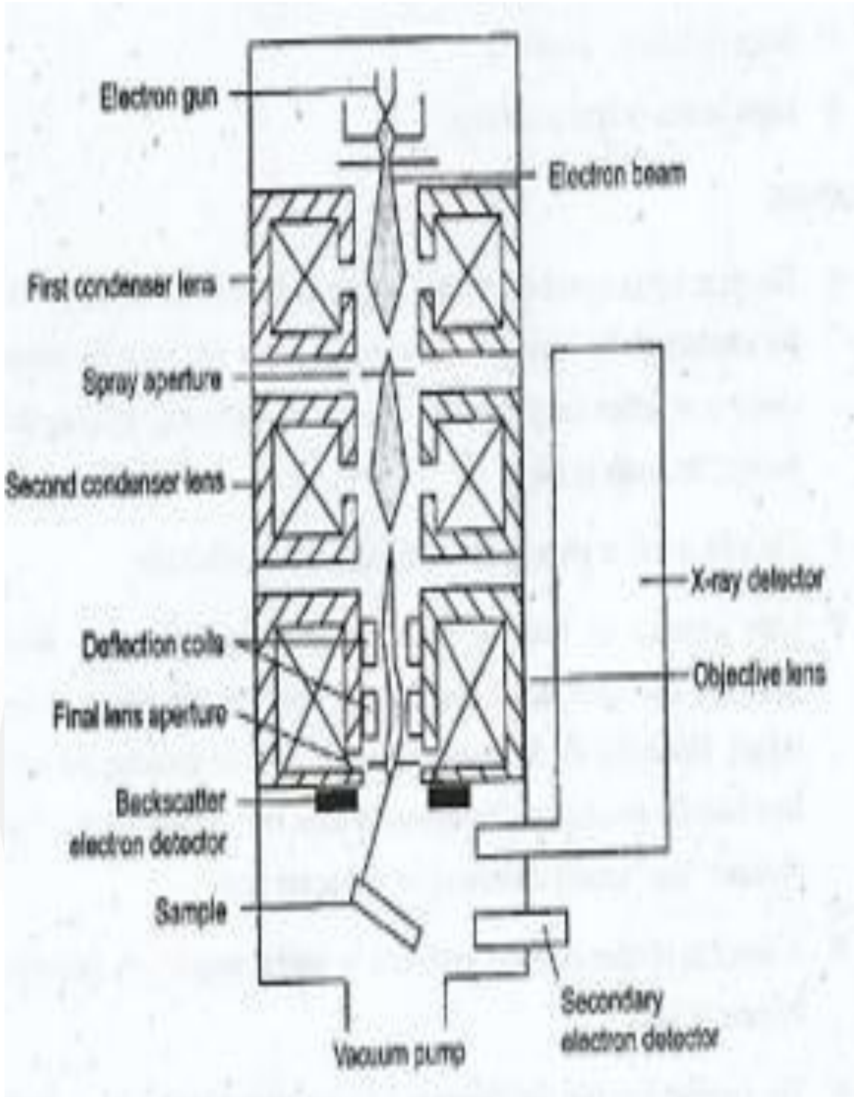
1. PRINCIPLE

- ❖ A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons.

- ❖ The electrons interact with atoms in the sample, producing various signals. that contain information about the surface topography and composition of the sample.



Working nature of SEM



Sectional view of SEM

2. COMPONENTS OF SEM

- **Electron gun**: It produces the high energy electron. Tungsten is normally used in guns because it has the highest melting point thereby allowing it to be electrically heated for electron emission, and its low cost.

- **Condenser lens**: It placed below electron gun. It is used to adjust the width (intensity) of the electron beam as per requirement. The main purpose is focusing the electron beam.

- **Vacuum chamber**: SEMs require a vacuum to operate. Without a vacuum, the electron beam generated by the electron gun would meet at constant interference from air particles in the atmosphere. The specimen chamber must be kept at a high vacuum of 10-3 to 10-4 Pa.

- **Deflector coils**: The scanning coils deflect the electron beam horizontally and vertically over the specimen surface. This is also called restoring.

- **Secondary electron detector**: A fluorescent substance (scintillator) is coated on the tip of the detector and a high voltage of about 10 kV is applied to it. The secondary electrons from the specimen are attracted to this high voltage and then generate light when they hit the scintillator. Then, the light is converted to electrons, and these electrons are amplified as an electric signal.

- **Image Display and Recording**: The output signals from the secondary electron detector are amplified and then transferred to the display unit

- **Specimen stage** - The platform on which a specimen sits while being imaged.

3. CONSTRUCTION

It consists of an electron gun. A magnetic condensing lens is used to condense the electron beam. The scanning coil is arranged in-between magnetic condensing lens and the sample.

4. SPECIMEN LOADING STAGES

- The specimen must meet the following requirements before it is loaded to the SEM stage:
    - Surface preparation
    - Mounting specimen
    - Specimen coating

(a) SURFACE PREPARATION

- Fracturing- When a specimen is a structural object, such as semiconductor device fracturing the specimen in this specific direction enables you to obtain a flat cross section
- Cutting- If a specimen is soft like a polymer, it can be cut.
- Mechanical polishing- For many metal or mineral specimens, mechanical polishing is applied.
- Milling by the ion beam- A focused ion beam (FIB) system enables you to obtain a cross section with a high positional accuracy of a few hundreds of nanometres
- Contrast enhancement- Surfaces of cross sections are chemically or physically etched to form irregularity on the surface and internal structures are observed using secondary electron images.

(b) MOUNTING SPECIMEN

- Bulk specimens- Bulk specimens are fixed to the specimen mount by conductive paste or conductive double-sided adhesive tape. If a bulk specimen has a relatively uniform shape, it is clamped with an exclusive specimen holder.

- Powders and particles- These specimens are dusted on conductive paste or double-sided adhesive tape

(c) SPECIMEN COATING

- If a specimen is nonconductive, its surface needs to be coated with a thin metal film so that the surface has conductivity.

5. WORKING OF SEM

- In SEM an electron beam is emitted from an electron gun fitted with a tungsten filament cathode.
- The electron beam, which typically has an energy ranging from 0.2 keV to 40 keV, is focused by one or two condenser lenses to a spot about 0.4 nm to 5 nm in diameter.
- The beam passes through pairs of scanning coils or pairs of deflector plates in the electron column, which deflect the beam in the x and y axes so that it scans in a raster fashion (rectangular area) of the sample surface
- When the primary electron beam interacts with the sample, the electrons lose energy by repeated random scattering and absorption.
- The energy exchange between the electron beam and the sample results in the reflection of high-energy electrons by elastic scattering, emission of secondary electrons, backscattered electrons and characteristic X-rays by inelastic scattering and the emission of electromagnetic radiation, each of which can be detected by specialized detectors.
- The beam current absorbed by the specimen can also be detected and used to create images of the distribution of specimen current.

- ❖ Electronic amplifiers of various types are used to amplify the signals, which are displayed as variations in brightness on a computer monitor.

6. OUTPUT-TYPES SCATTERED ELECTRONS

(a) X-rays

- ❖ emitted from beneath the sample surface, can provide element and mineral information.

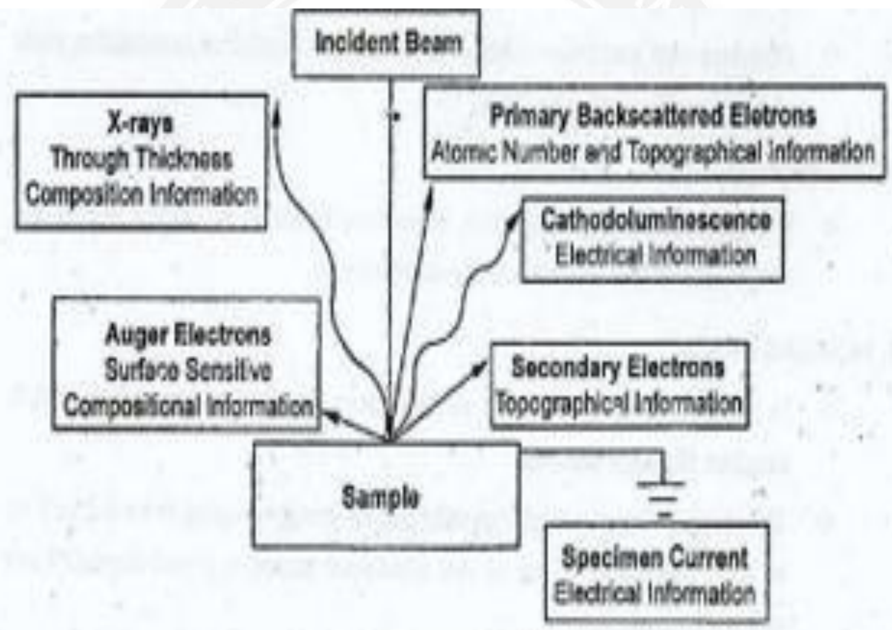


Fig. 4.5. Scattering Electrons

(b) SECONDARY ELECTRONS

- ❖ When the incident electron beam enters the specimen, secondary electrons are produced from the emission of the valence electrons of the constituent atoms in the specimen.
- ❖ Secondary electron image information used for surface morphology

(c) BACKSCATTERED ELECTRONS

- Backscattered electrons are those scattered backward and emitted out of the specimen, when the incident electrons are scattered in the specimen.
- This feature can be used to observe the topography of the surface.

7. MAGNIFICATION

- Magnification in an SEM can be controlled over a range of about 6X orders of magnitude from about 10 to 3,000,000 times.
- Magnification is therefore controlled by the current supplied to the scanning coils, or the voltage supplied to the deflector plates, and not by objective lens power.

8. APPLICATIONS

1. THE SEM ALSO EXCELS IN PRODUCING

   - Detailed surface top.graphy images
   - Failure analysis
   - Dimensional analysis
   - Process characterization
   - Reverse engineering
   - Particle identification

- Surface 3D
- Elemental analysis

2. **IDEAL USES**

- High resolution surface topography images.

9. ADVANTAGES

- Elemental microanalysis and particle characterization
- Rapid, high-resolution imaging
- Quick identification of elements present
- Excellent depth of field (~100X that of optical microscopy)
- Versatile platform that supports many other analysis techniques
- Low vacuum mode enables imaging of insulating and hydrated samples

10. LIMITATIONS

- Size restrictions may require cutting the sample.

- ❖ The size is not portable.
- ❖ SEMs are expensive and large.
- ❖ Maintenance involves keeping a steady voltage, currents electromagnetic coils and circulation of cool water. to
- ❖ SEMs are limited to solid, inorganic samples small enough to fit inside the vacuum chamber that can handle moderate vacuum pressure.
- ❖ SEMS carry a small risk adiation exposure
- ❖ Training is required to operate.

4.4.2 TRANSMISSION ELECTRON MICROSCOPY (TEM)

A Transmission Electron Microscope (TEM) utilizes energetic electrons to provide morphologic, compositional and crystallographic information on samples. The transmitted electrons that have passed through the thin sample are detected to form images, which is the reason to call it "transmission" electron microscopy

1. PRINCIPLE

An image is formed from the interaction of the electrons with the sample as the beam is transmitted through the specimen. The image is then magnified and focused onto an imaging device, such as a fluorescent screen, a layer of photographic film, or a sensor such as a scintillator attached to a charge-coupled device.

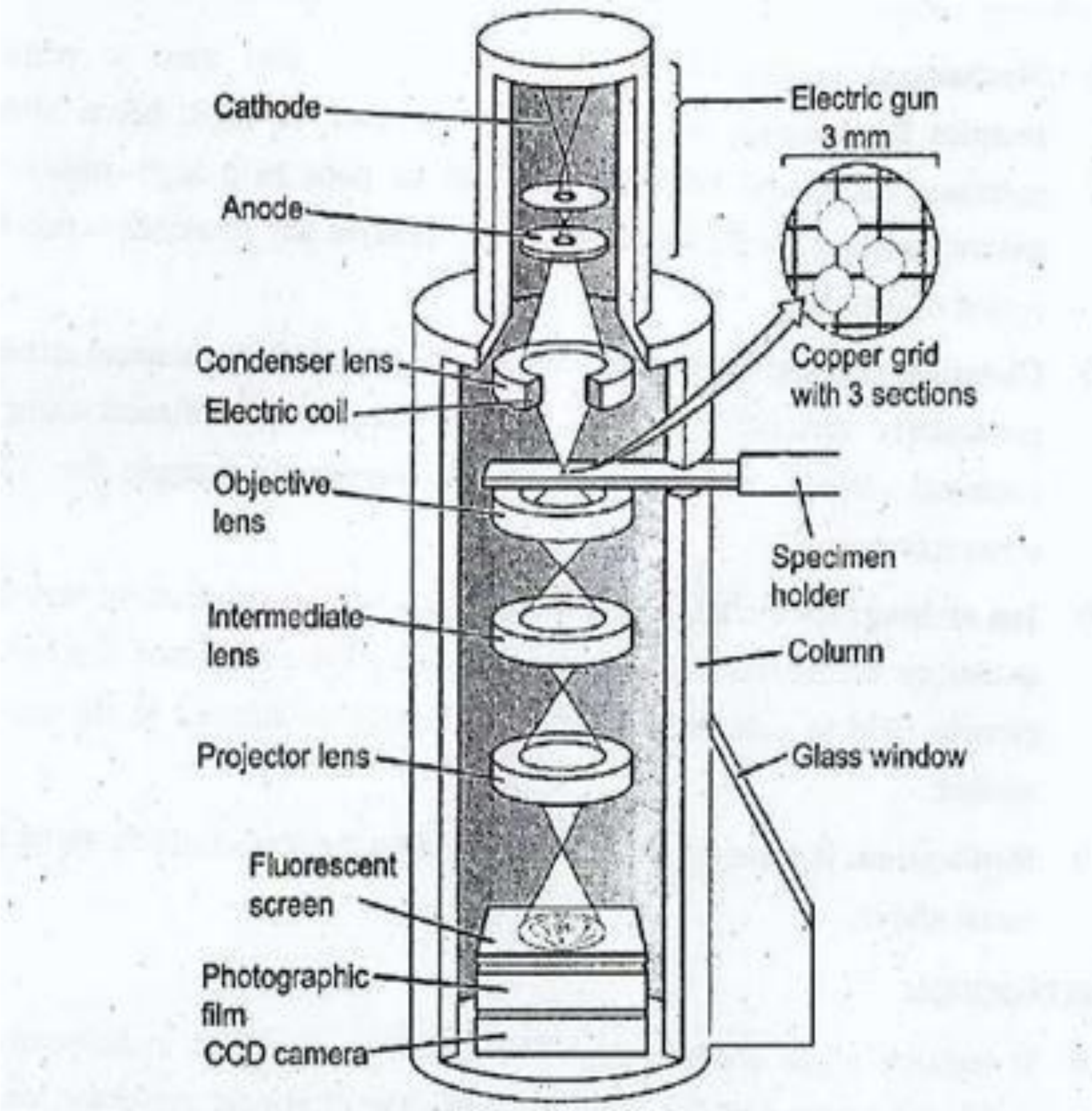


Fig. 4.6. Working of TEM

2. METHOD OF SPECIMEN PREPARATION

- **Ultra microtome**: Specimens must be very thin so that electrons are able to pass. This may be done by cutting very thin slices of a specimen's using an ultra-microtome.

- **Ultrasonic disk cutting**: For most electronic materials, it is a common sequence of preparation technique.
- **Dimpling**: Dimpling is a preparation technique that produces a specimen with a thinned central area and an outer rim of sufficient thickness to permit ease of handling.
- **Ion milling**: Ion milling is traditionally the final form of specimen preparation. In this process, charged argon ions are accelerated to the specimen surface by the application of high voltage. The ion impingement upon the specimen surface removes material as a result of momentum transfer
- .**Mechanical milling**: Mechanical polishing is also used to prepare samples for imaging on the TEM. A diamond, or cubic boron nitride polishing compound Polishing needs to be done to a high quality, to ensure constant sample thickness and to remove any scratches across the region of interest.
- **Chemical etching**: Certain samples may be prepared by chemical etching, particularly metallic specimens. These samples are thinned using a chemical etchant, such as an acid, to prepare the sample for TEM observation.
- **Ion etching**: lon etching is a sputtering process that can remove very fine quantities of material. Ion etching uses an inert gas passed through an electric field to generate a plasma stream that is directed to the sample surface.
- **Replication**: It common use is for examining the fresh fracture surface of metal alloys.

3. CONSTRUCTION

It consists of an electron gun. The specimen is placed in between the condensing lens and the objective lens. The magnetic projector lens is placed above the fluorescent screen.

4. COMPONENTS OF TEM

- **Electron Source** : The emission source or cathode, which may be a tungsten filament or needle. The gun is connected to a high voltage source (typically -100-300 kV) and it emit electrons either by thermionic or field electron emission into the vacuum.
- **Electromagnetic lenses -** Electron lenses are designed to act similar like optical lens, by focusing parallel electrons at some constant focal distance. These focuses selected magnetic properties, such as magnetic saturation, hysteresis and permeability.
- **Vacuum chamber**: To increase the mean free path of the electron gas interaction, it is evacuated to low pressures, typically on the order of 10-4 Pa

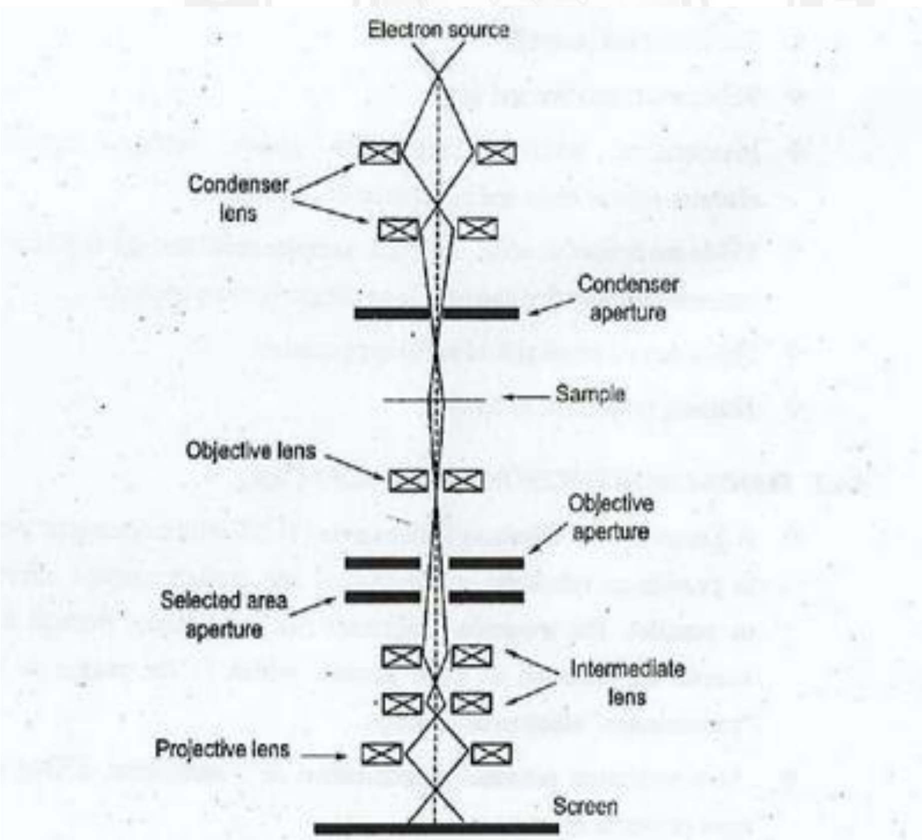


Fig. 4.7. Cross sectional view of TEM

- **Condensers**: Condensers consists of condenser lenses, the objective lenses, and the projector lenses. The condenser lenses are responsible for primary beam formation, while the objective lenses focus the beam that comes through the sample itself. The projector lenses are used to expand the beam onto the phosphor screen or other imaging device, such as film.
- **Sample stage** - Stage designs include specimen holder infs the vacuum with minimal loss of vacuum in other areas of the microscope.
- **Phosphor or fluorescent screen** - Imaging systems in a TEM consist of a phosphor screen, which may be made of fine (10- μm) particulate zinc sulphide, for direct observation by the operator and optionally an image recording system such as photographic film
- **Condenser lens** - The first electromagnetic lens that the electron beam encounters. Focuses the electrons onto the specimen.

5. WORKING

- TEMs employ a high voltage electron beam in order to create animage.
- An electron gun at the top of a TEM emits electrons that travel through the microscope's vacuum tube.
- Rather than having a glass lens focusing the light, it employs an electromagnetic lens which focuses the electrons into a very fine beam.
- This beam then passes through the specimen, which is very thin (typically, sample thickness is less than 200 nm, depending on the composition of sample and the expected information from TEM characterization) and the electrons either scatter or hit a fluorescent screen at the bottom of the microscope.

- During transmission, the speed of electrons directly correlates to electron wavelength; the faster electrons move, the shorter wavelength and the greater the quality and detail of the image.
- An image of the specimen with its assorted parts shown in different shades according to its density appears on the screen. The image becomes visible when the electron beam hits a fluorescent screen at the base of the machine. This is analogous to the phosphor screen at the front of an old fashioned TV.

6. OPERATION MODES OF TEM

- After interaction with the sample, on the exit surface of the specimen two types of electrons exist - unscattered (which will correspond to the bright central beam on the diffraction pattern) and scattered electrons (which change their trajectories due to interaction with the material).
- The two basic operation modes of TEM
  - Imaging mode
  - Diffraction mode

7. RESOLUTION

TEMs can produce images with resolution down to 0.2nm. This resolution is smaller than the size of most atoms and therefore shows the true structural arrangement of atoms in the sample material.

8. LIMITATIONS

- Significant sample preparation time
- Small sampling volumes and samples are typically ~100nm thick.

- ❖ Some materials are not stable in the high energy electron beam
- ❖ TEMs are large and very expensive
- ❖ Laborious sample preparation
- ❖ Operation and analysis requires special training
- ❖ Samples are limited to those that are electron transparent, able to tolerate the vacuum chamber and small enough to fit in the chamber
- ❖ Images are black and white
- ❖ Electron microscopes are sensitive to vibration and electromagnetic fields and must be housed in an area that isolates them from possible exposure.
- ❖ A Transmission Electron Microscope requires constant upkeep including maintaining voltage, currents to the electromagnetic coils and cooling water.

9. ADVANTAGES

- ❖ The highest spatial resolution elemental mapping of any analytical technique (0.2nm (2A) image resolution)
- ❖ Small area crystallographic information

- ❖ Strong contrast between crystalline vs amorphous materials without chemical staining.
- ❖ TEMs offer the most powerful magnification, potentially over one million times or more.
- ❖ TEMS have a wide-range of applications and can be utilized in a variety of different scientific, educational and industrial fields
- ❖ TEMS provide information on element and compound structure.
- ❖ Images are high-quality and detailed
- ❖ TEMs are able to yield information of surface features, shape, size and structure

10. APPLICATIONS

- ❖ Metrology at 0.2nm resolution
- ❖ Identification of nm-sized defects on integrated circuits, including embedded particles and via residues
- ❖ Determination of crystallographic phases at the nanometre scale
- ❖ Catalyst studies
- ❖ Nanometre scale elemental maps
- ❖ Super lattice characterization

- Crystal defect characterization (dislocations, grain boundaries, voids, stacking faults)
- Microstructure and nanostructure: size and morphology
- Crystal structure determination through electron diffraction
- Chemical information composition and bonding (EDS, EELS) from single points, line scans or maps
- Energy filtered imaging (EFTEM)
- TEMs can be used in semiconductor analysis and production and the manufacturing of computer and silicon chips.

4.4.3 COMPARISON BETWEEN SEM AND TEM

Category	SEM	TEM
Source electrons	Scattered electrons	Transmitted electrons
Process of working	Scattering absorption	Diffraction
Energy	1-30kV	60-300kV
Environment	Air/vacuum	Vacuum
Specimen thickness	Any thickness	Typically less than 150nm
Output	3D image formation	2D image projection of inner structure

Magnification	2 million level magnification	50 million level magnification

Field of view	Large	Limited
Optimum resolution	0.4 nanometre	0.5 nanometre
Operation	Little sample preparation	Laboratory sample preparation
Amount of sample	Huge	Minimum
Cost	Cost is low	Cost is 2 or 3 times higher than SEM
Sample usage	Invasive	Non-invasive