MATERIAL CHARACTERIZATION TESTING

OML751 - TESTING OF MATERIALS

VII SEMESTER

[R 2017 - Open Elective Subject]

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OML751 – TESTING OF MATERIALS - SYLLABUS

UNIT IV MATERIAL CHARACTERIZATION TESTING 9

Macroscopic and Microscopic observations, Optical and Electron microscopy (SEM and TEM) - Principles, Types, Advantages and Limitations, Applications. Diffraction techniques, Spectroscopic Techniques, Electrical and Magnetic Techniques- Principles, Types, Advantages and Limitations, Applications.

UNIT-IV : MATERIAL CHARACTERIZATION TESTING

- Macroscopic and Microscopic observations,
- Optical and Electron microscopy (SEM and TEM) –

  Principles, Types, Advantages and Limitations, Applications.
- Diffraction techniques,
- Spectroscopic Techniques,
- Electrical and Magnetic Techniques –

  Principles, Types, Advantages and Limitations, Applications.

Overview

- Material characterization has a wide variety of characterization techniques in the areas of microscopy, spectroscopy and macroscopic techniques which help to increase the different degrees of understanding why different materials show different properties and behavior.
- Materials characterizing are aimed at the features of materials quantitatively, this is often closely related to the analysis, modelling and simulation, and the qualitative characterization of materials through testing.
- *"Characterization describes those features of composition and structure (including defects) of a material that are significant for a particular preparation, study of properties, or use, and suffice for reproduction of the material."*

Material Characterization used for Identification of,

- Contaminants
- Purity
- Active ingredients
- Polymer additives
- Fillers
- Solvents
- Failure analysis
- Material comparisons
- Specialized method development

Methods of Materials Characterization

- Chemical characterization
- Toxicological characterization
- Physical characterization
- Electrical characterization
- Morphological characterization
- Mechanical characterization

Objectives of Materials Characterization

- To measure accurately the physical properties of materials
- To measure accurately the chemical properties of materials
- To determine accurately the structure of a material at atomic and microscopic level structures

Common applications of Materials Characterization

- Surface chemical analysis
- Near surface chemical analysis
- Atomic & Nanoscale chemical analysis
- Surface Imaging
- Defect analysis
- Analytical imaging
- Non destructive Internal Imaging

Classification based on Application

MATERIAL CHARACTERIZATION

Composition and structure

1. Bonding
2. Microstructure
3. Surfaces topography and interfaces

Microscopy Testing & Spectroscopy Testing

PERFORMANCE

a) Materials failure mechanisms

i. Corrosion
ii. Friction and wear
iii. Biogenic impact
iv. Materials environment interactions

b) Performance control by condition monitoring methods

i. Non destructive evaluation
ii. Lifetime predictions
iii. Characterization of safety and reliability

Spectroscopy Testing

Properties

1. Mechanical
2. Thermal
3. Electrical
4. Magnetic
5. Optical

Macroscopic Testing

SCALE

Scale	Range
Nano scale	1 to 100 nanometers
Micro scale (micro-devices and micro systems)	1 to 1000 micro-meters
Macro scale	Millimeter to the kilometer scale

Material Characterization Testing

- Material characterization is the process of measuring and determining physical, chemical, mechanical and microstructural properties of materials.

- Based on scale of testing, there are 2 types,
  1. Microscopy testing → **Some Common Instruments:** Optical Microscopy, SEM, TEM, Atomic force microscope (AFM), X-ray diffraction topography
  2. Macroscopic testing → **Some Common Instruments:** Differential thermal analysis, Dielectric thermal analysis, Impulse excitation technique, Mechanical testing (tensile, hardness, creep, fatigue….)

- Based on testing of composition, there are 2 types,
  1. Spectroscopy &
  2. Nuclear spectroscopy

  → **Some Common Instruments:** UV-visible spectroscopy, thermoluminescence, photoluminescence, energy-dispersive X-ray spectroscopy.

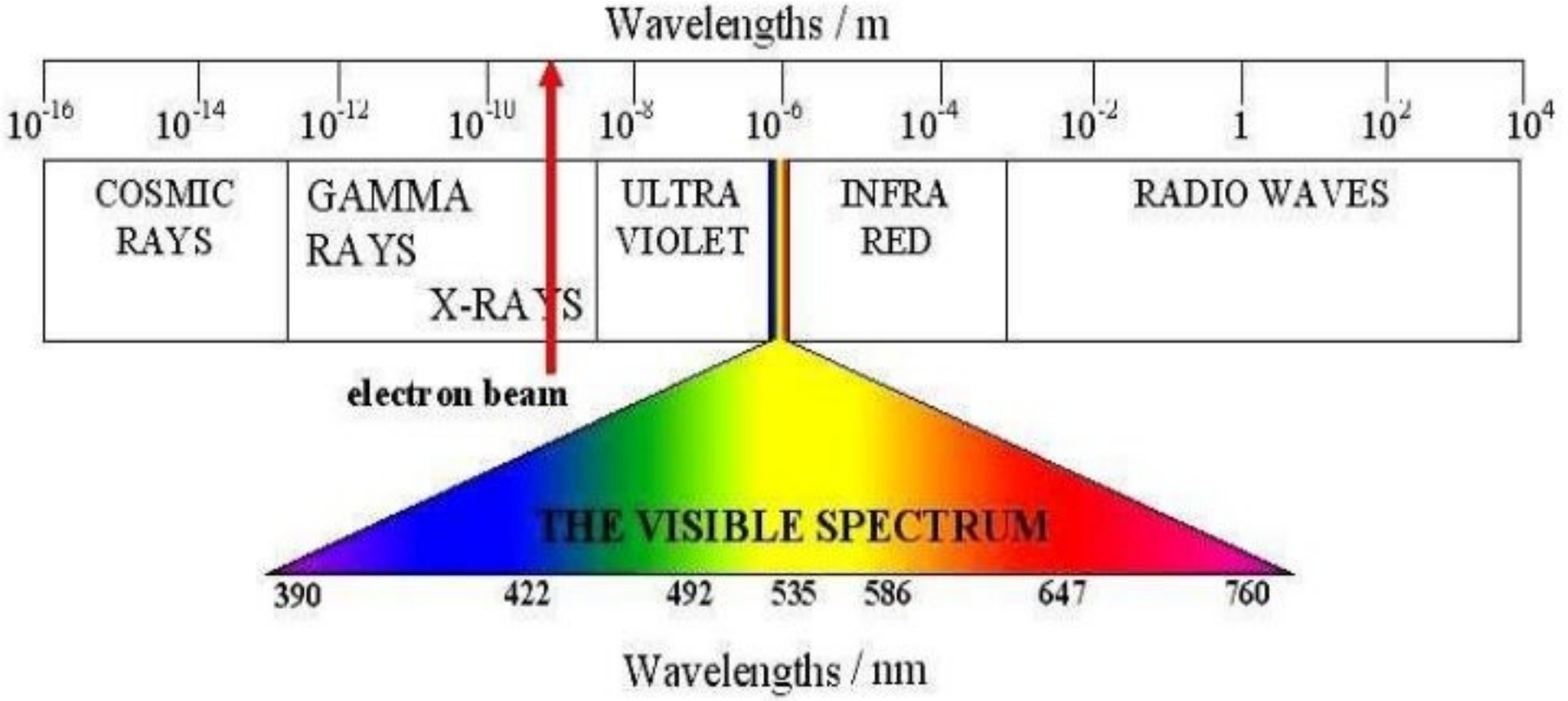
- **Macroscopic Techniques :** https://www.youtube.com/watch?v=fc8zrgYJCJw
- **Microscopic Techniques :** https://www.youtube.com/watch?v=UuHofNW40Yw

BASIC TERMINOLOGY

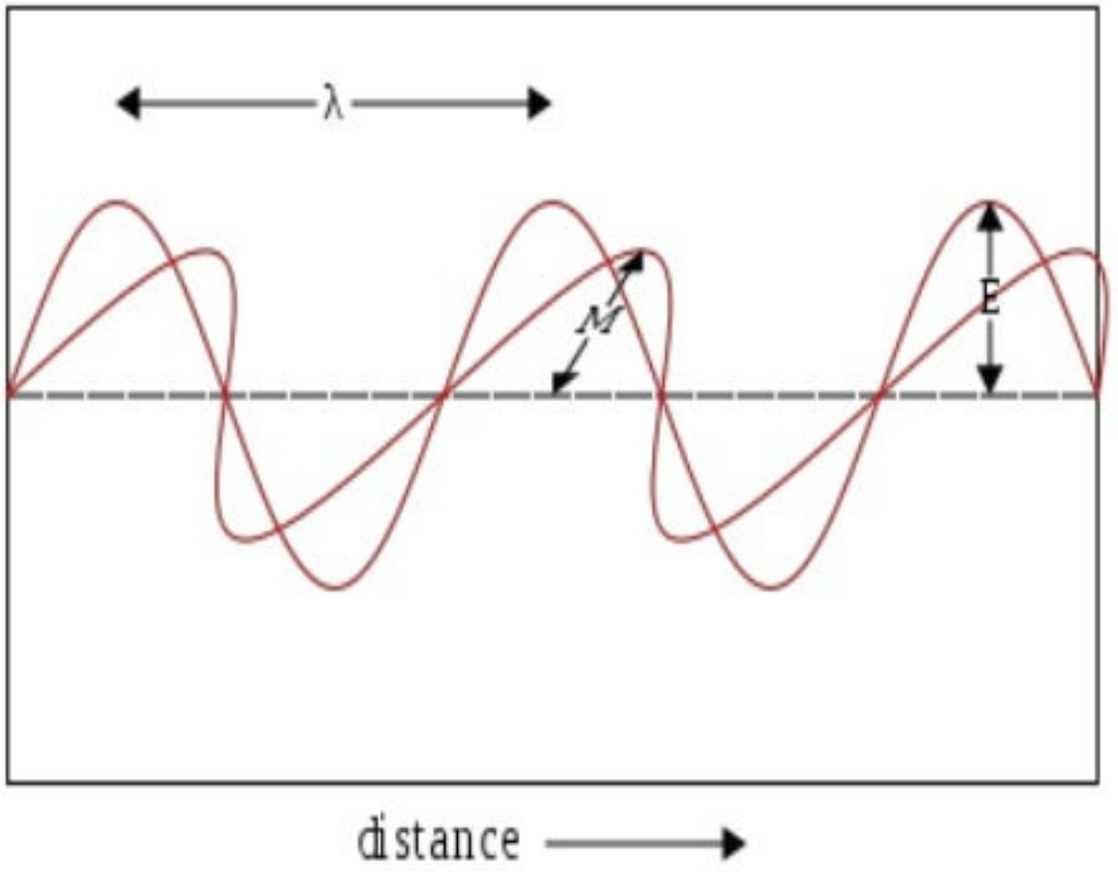
What is a Light?

- **Light** is electromagnetic radiation that is visible to the human eye, and is responsible for the sense of sight.
- Visible light has a wavelength in the range of about 380 nm to about 740 nm – between the invisible infrared, with longer wavelengths and the invisible ultraviolet, with shorter wavelengths.
- Primary properties of visible light are
  - Intensity,
  - Propagation direction,
  - Frequency or wavelength spectrum
  - Polarisation
- Light rays are usually perpendicular to wave front

The electromagnetic spectrum



Light wave



λ = wave length

E = amplitude of electric field

M = amplitude of magnetic field

UNITS OF MEASUREMENT

$1m = 10^3$ mm (millimetres)

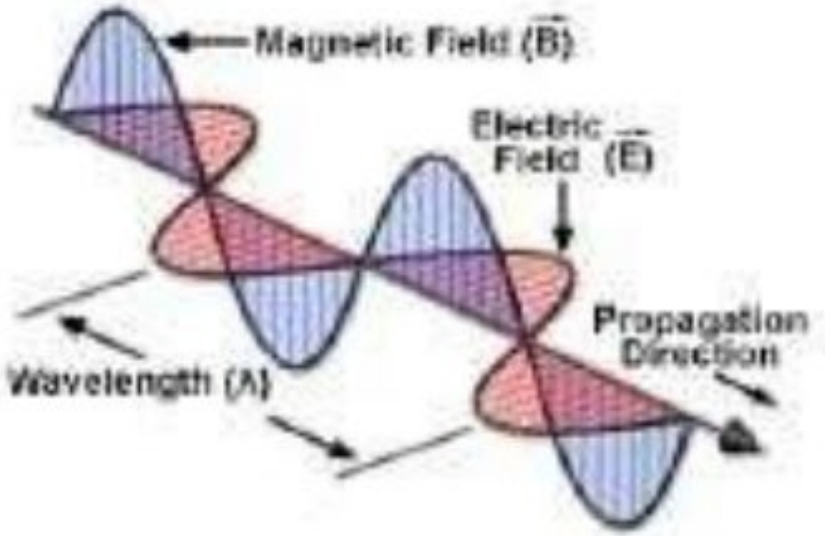
$1m = 10^6$ μm (micrometres)

$1m = 10^9$ nm (nanometres)

Sometimes in old texts Angstroms (Å) are used (the diameter of a hydrogen atom)

$1m = 10^{10}$ Å

Light as an Electromagnetic Wave



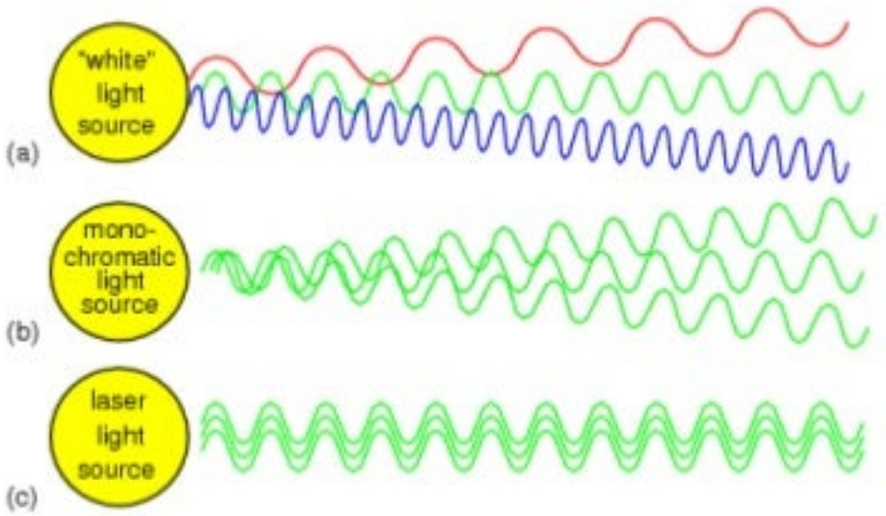
Most matter interacts mostly with the electric field

⇒ We will ignore the magnetic field

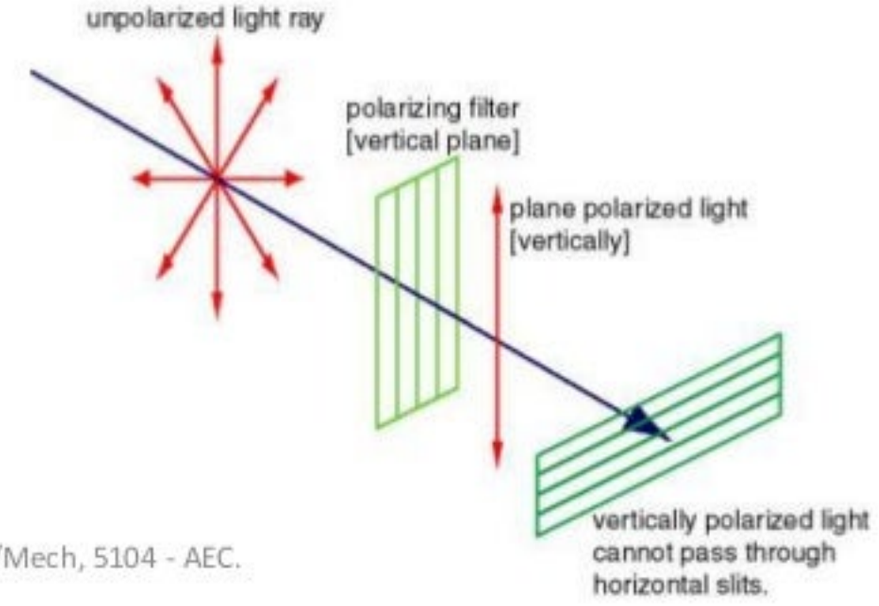
Polarization = direction of electric field

The kinds of light most frequently referred :

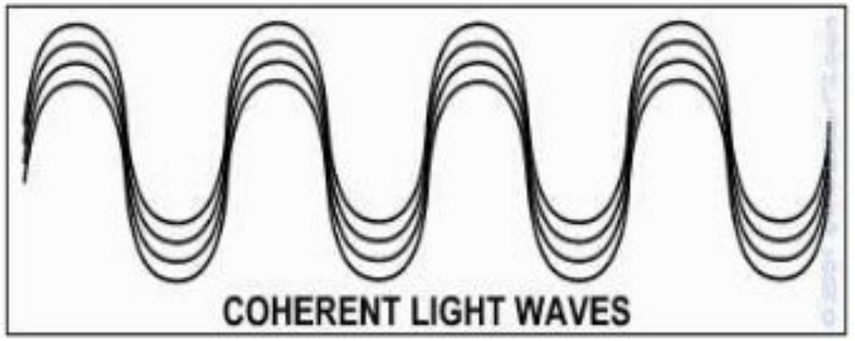
- ***Monochromatic***—*waves having the same wavelength or vibrational frequency (the* same color).



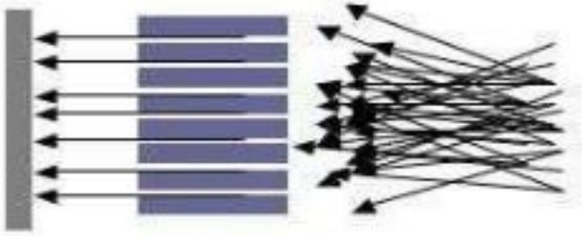
- ***Polarized***—*waves whose E vectors vibrate in planes that are parallel to one another.*



Coherent—*waves of a given wavelength that maintain the same phase relationship* while travelling through space and time (laser light is coherent, monochromatic, and polarized).



- ***Collimated***—*waves having coaxial paths of propagation through space—that is,* without convergence or divergence, but not necessarily having the same wavelength, phase, or state of polarization. The surface wavefront at any point along a cross-section of a beam of collimated light is planar and perpendicular to the axis of propagation.



MAGNIFICATION

- Magnification on a microscope refers to the amount or degree of visual enlargement of an observed object or enlargement of image.

- The magnification of a microscope can be calculated by linear optics.

$$M = \frac{v - f}{f}$$

- f is the focal length of the lens
- v is the distance between the image and lens

- A Higher magnification lens has a shorter focal length.

Magnification

- The two lenses are called:
  - Eyepiece
  - Objective
- The eyepiece has a magnification of "10x"
- The magnification of the objective lenses vary and are marked on the lens

Total magnification = (Eyepiece) X (Objective)

Example: 10 x 40 = 400

In other way, Magnification

- Magnification on a microscope refers to the amount or degree of visual enlargement of an observed object or enlargement of image.
- Magnification is measured by multiplies, such as 2x, 4x and 10x indicating that the object is enlarges to twice as big, four times as big or 10 times as big, respectively.

$$Magnification = \frac{Image}{Object}$$

Magnification	Instrument
1x	Naked eye
2x to 5x	Magnifying glass
10x to 20x	Stereoscopic microscope
50x to 1500x	Upright/ inverted microscope
2000x to 1000000x	Electron microscope

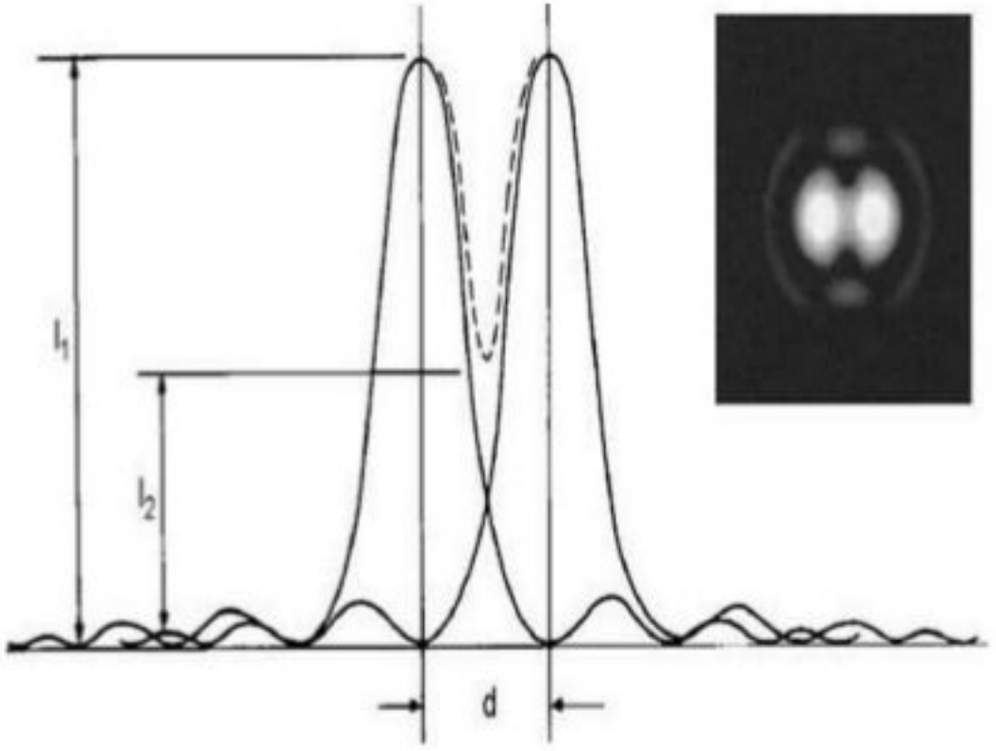
Resolution

- *Resolution – is defined as the ability to distinguish two very small and closely-spaced objects as separate entities.*

- Resolution is determined by certain physical parameters that include the wavelength of light, and the light-gathering power of the objective and condenser lenses.

Resolution

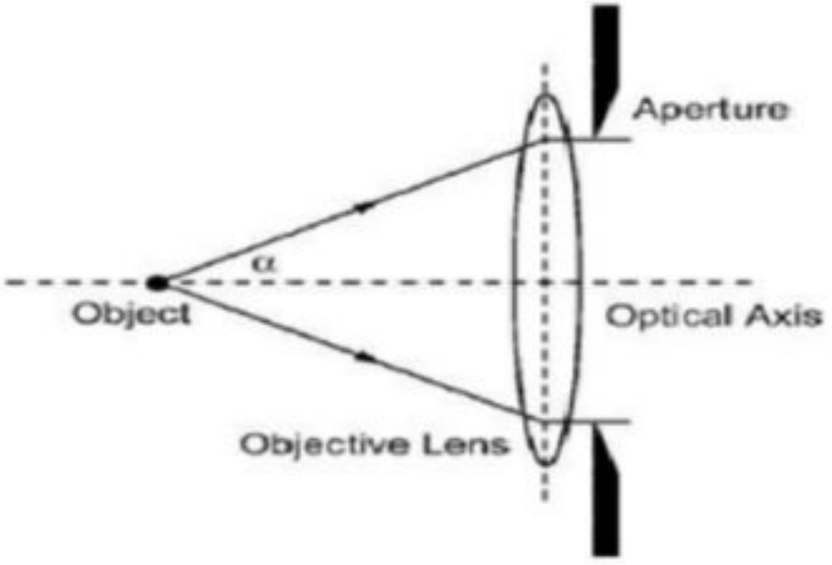
- Resolution refers to the minimum distance between two points at which they can be visibly distinguished as two points.
- Whenever light passes through an aperture, diffraction occurs so that parallel beam of light is transformed into a series of cone, which are seen as circle and they are know as Airy ring.
- *The resolution of a microscope (R) is defined as the minimum distance between two Airy disks that can be distinguished.*



Resolution

- Resolution is a function of microscope parameters.
- Where μ *is the refractive index of the medium between the object and objective lens and* α is the half-angle of the cone of light entering the objective lens.
- The product $\mu \sin \alpha$, *is called the numerical aperture (NA).*
- To achieve higher resolution we should use shorter wavelength light and larger *NA*.

$$R = \frac{d}{2} = \frac{0.61\lambda}{\mu \sin \alpha}$$



LENS

- The observation magnification is the product of the magnifications of each of the lenses. This generally ranges from 10x to 1000x with some models even reaching up to 2000x magnification. Common types of lens include,

- OBJECTIVE LENS: Achromatic lens, Semi-apochromatic lens (fluorite lens), Apochromatic lens, Plan lens, Immersion lens
- OCULAR LENS (EYEPIECE): Huygens lens, Ramsden lens, Periplan lens, Compensation lens, Wide-field lens, Super-field lens
- CONDENSER LENS: Abbe condenser, Achromatic condenser, Universal condenser
- ABERRATION: Chromatic aberration, Spherical aberration

- **Numerical Aperture :** The numerical aperture of a microscope objective is a measure of its ability to resolve fine specimen detail. The value for the numerical aperture is given by,

$$\text{Numerical Aperture (NA)} = n \sin \alpha$$

- **Depth of Field :** Depth of field is the axial depth of the space on both sides of the object plane within which the object can be moved without detectable loss of sharpness in the image, and within which features of the object appear acceptably sharp in the image while the position of the image plane is maintained.
- **Depth of Focus :** Depth of focus is the axial depth of the space on both sides of the image plane within which the image appears acceptably sharp while the positions of the object plane and of the objective are maintained.

OPTICAL MICROSCOPE

PRINCIPLE

- The functioning of the light microscope is based on its ability to focus a beam of light through specimen, which is very small and transparent, to produce an image.
- The image is then passed through one or two lenses for magnification for viewing.
- The transparency of the specimen allows easy and quick penetration of light.

Types of Microscope

1. Bright field microscope
    (a) Simple microscope
    (b) Compound microscope
2. Dark field microscope
3. Polarized light microscope
4. Phase-contrast microscope
5. Fluorescence microscope
6. Digital microscope

Other Microscope Types

i) Stereo microscope

ii) Comparison microscope

iii) Inverted microscope

iv) Epifluorescence microscope

v) Confocal microscope

IMAGE FORMATION

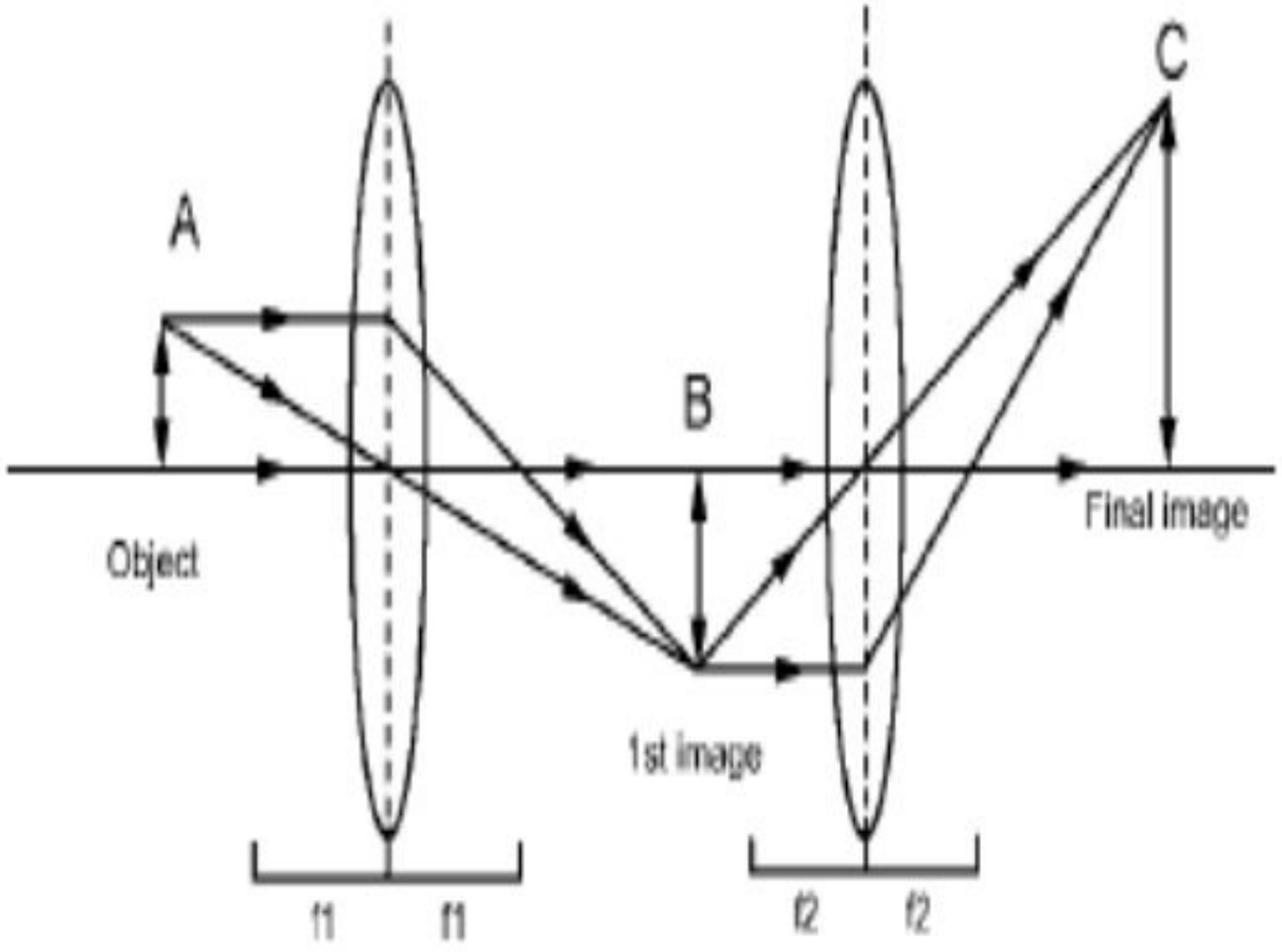
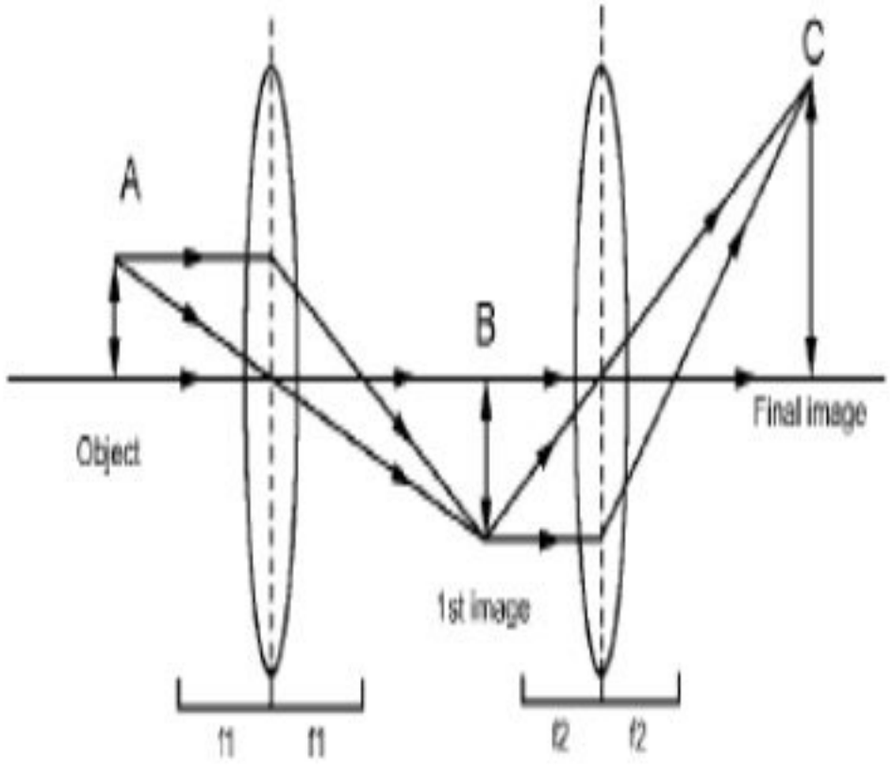
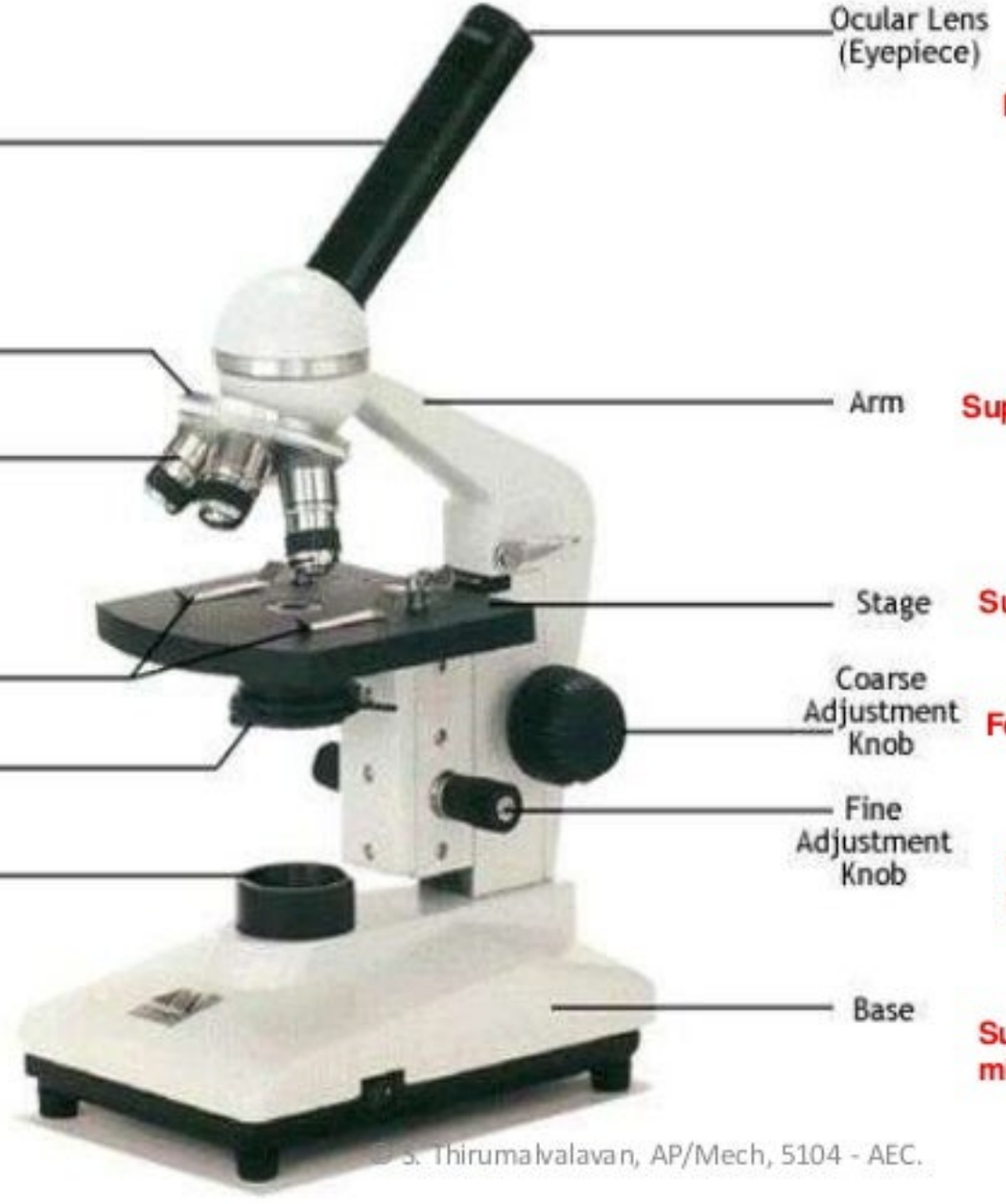


IMAGE FORMATION

- A specimen is placed at a position A where it is between one and two focal length from an objective lens.
- Light rays from the object firstly converge at the objective lens and are then focused at position B to form a magnified inverted image.
- The light rays from the image are further converged by the second lens (*projector lens)* to form a final magnified image of an object at C.



The Parts of the Microscope and Their Function



Maintains proper distance between lenses

Magnification

Hold objectives- rotates to change magnification

Support body tube

Magnification

Supports slide

Holds slide In place

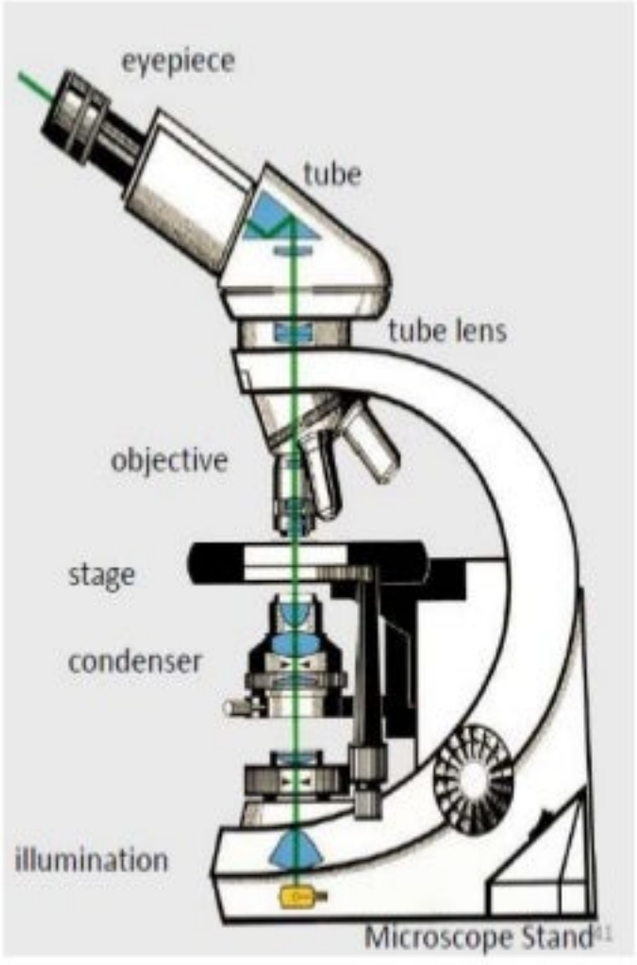
Focuses image

Regulates amount of light

Sharpens the image

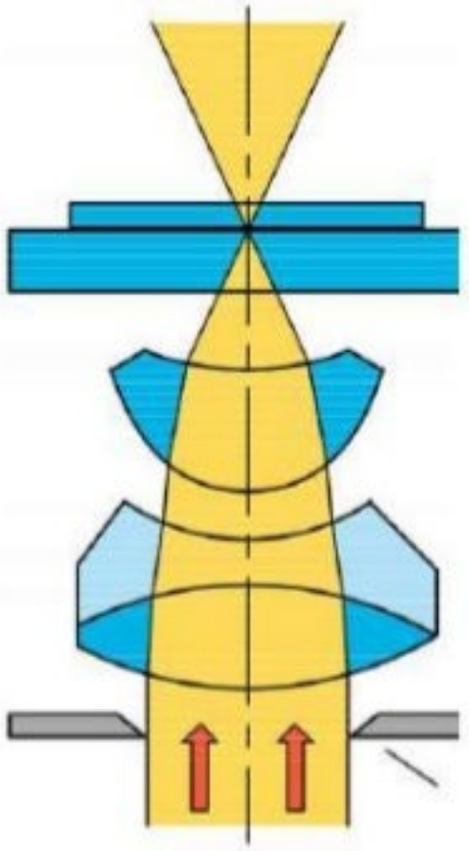
Reflects light towards eyepiece

Supports microscope



Condenser

- Collect illumination light rays and converge them to a focus
- Contains the aperture diaphragm for homogeneous illumination
- Increases resolution, enhances contrast, reduces glare



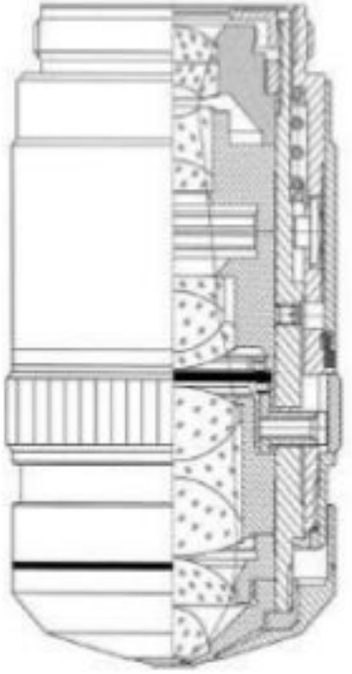
Specimen stages

- Object guide
- x/y - stage
- Scanning stage
- Pol rotary stage
- Heated stage

Objective

- Tubus length 160mm
- different objective classes
- Magnification (1,6x - 100x)
- 4x -red, 10x-yellow, 40x blue, 100x grey
- Different apertures

Objective lens



Cross section of a typical objective

Objective

magnification

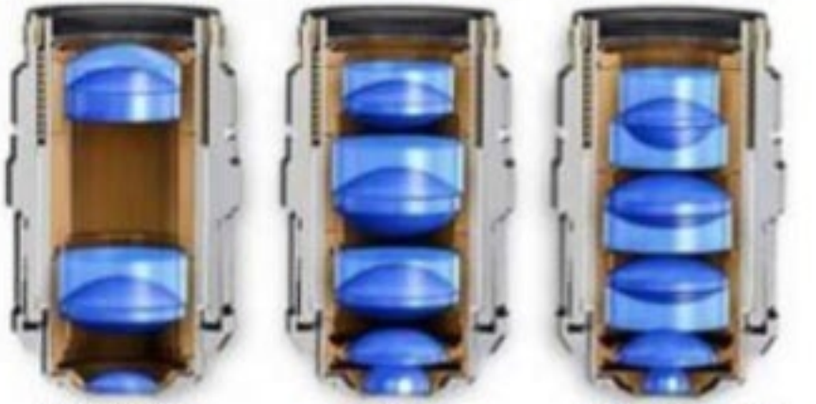
coverglass specification

objective type

Numerical aperture

Correction classes of objectives

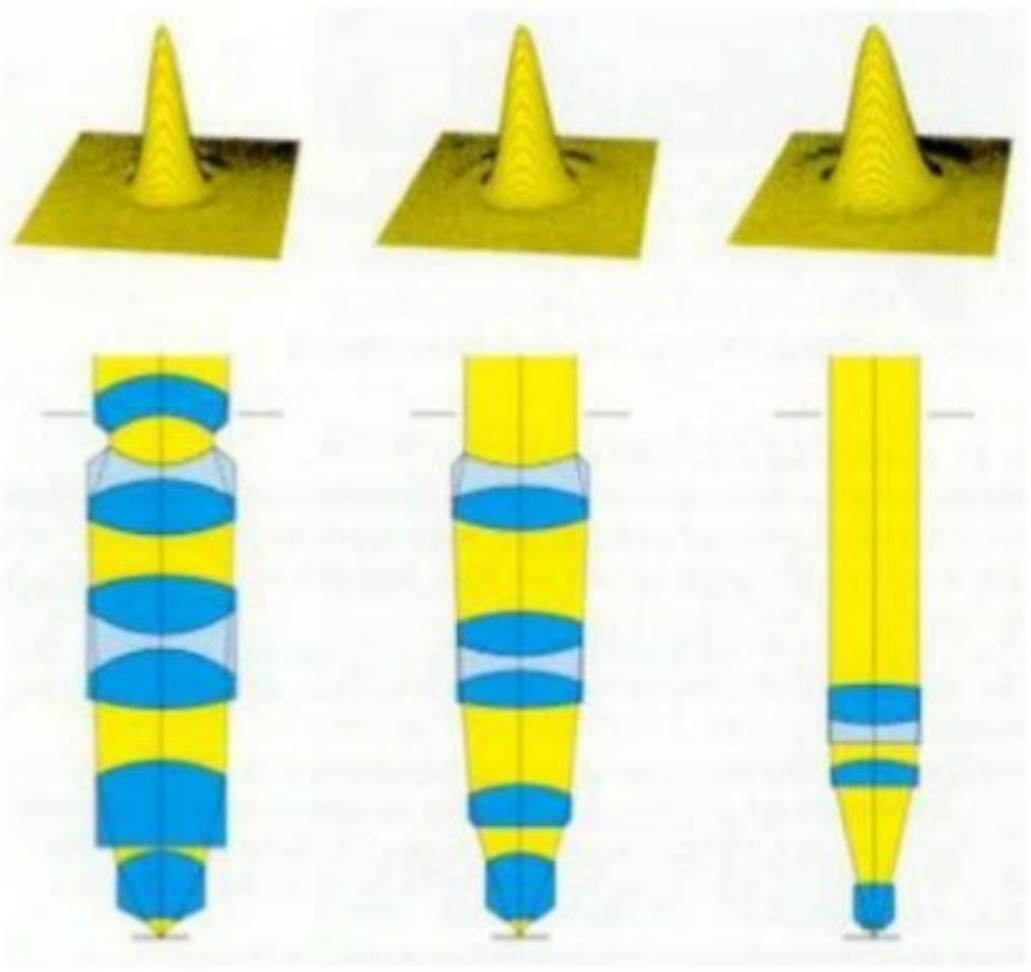
Objectiv class	*Field of view*	*Chromatic correction*
C PLAN	20mm	Achromat
N PLAN	22mm	Achromat
PL FLUOTAR	>25mm	Semi Apochromat
PLAN APO	>25mm	Apochromat
PL APO (CS)	>25mm	Apochromat, smaller tollerances for confocal applications
APO U-V-I	20mm	Apochromat UV, visual light, IR-Transmission



Achromat (cheap)

Fluor "semi-apo" (good correction, high UV transmission)

Apochromat (best correction)



PL APO
20x/0.60

PL FLUOTAR
20x/0.50

N PLAN
20x/0.40

Objective Working and Parfocal Distance

Figure 1

Objective Lens and Eyepiece

- The objective lens generates the primary image of the specimen, and its resolution determines the final resolution of the image.

- Classification of the objective lens is based on its aberration correction capabilities
  - Achromat
    - The achromatic lens corrects chromatic aberration for two wavelengths (red and blue). It requires green illumination to achieve satisfactory results for visual observation and black and white photography.
  - Semi-achromat (also called 'fluorite')
    - The semi-achromatic lens improves correction of chromatic aberration. Its *NA is larger than that of an achromatic lens with the same magnification and produces a* brighter image and higher resolution of detail.
  - Apochromat.
    - The apochromatic lens provides the highest degree of aberration correction. It almost completely eliminates chromatic aberration. It also provides correction of spherical aberration for two colors. Its *NA is even larger than that of* a semi-achromatic lens.

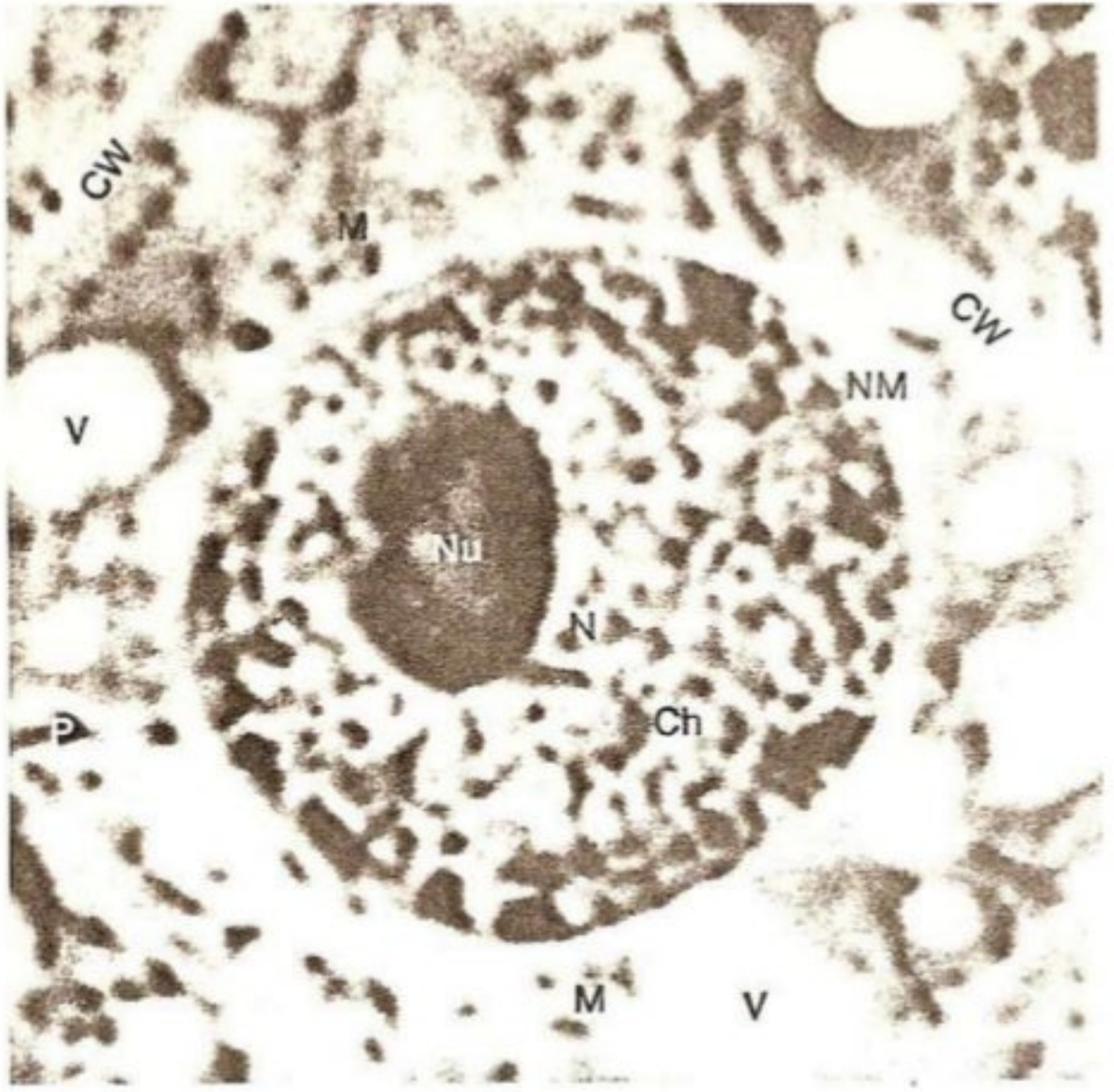
- The characteristics of an objective lens are engraved on the barrel.
  - 'FL', 'FLUOR' or 'NEOFLUOR' stands for 'fluorite' and indicates the lens is semiachromatic;
  - 'APO' indicates that the lens is apochromatic;
  - If neither of the above markings appears, then the lens is achromatic;
  - 'PLAN' or 'PL' stands for 'planar' and means the lens is corrected for curvature of field, and thus generates a flat field of image;
  - 'DIC' means the lens includes a Wollaston prism for *differential interference contrast*;
  - 'PH' or 'PHACO' means the lens has a phase ring for phase contrast microscopy ;
  - 'number/number' indicates *magnification/numerical aperture. Thus, '40/0.75' means the* lens has a magnification of 40×and a numerical aperture of 0.75.

Storing the Microscope

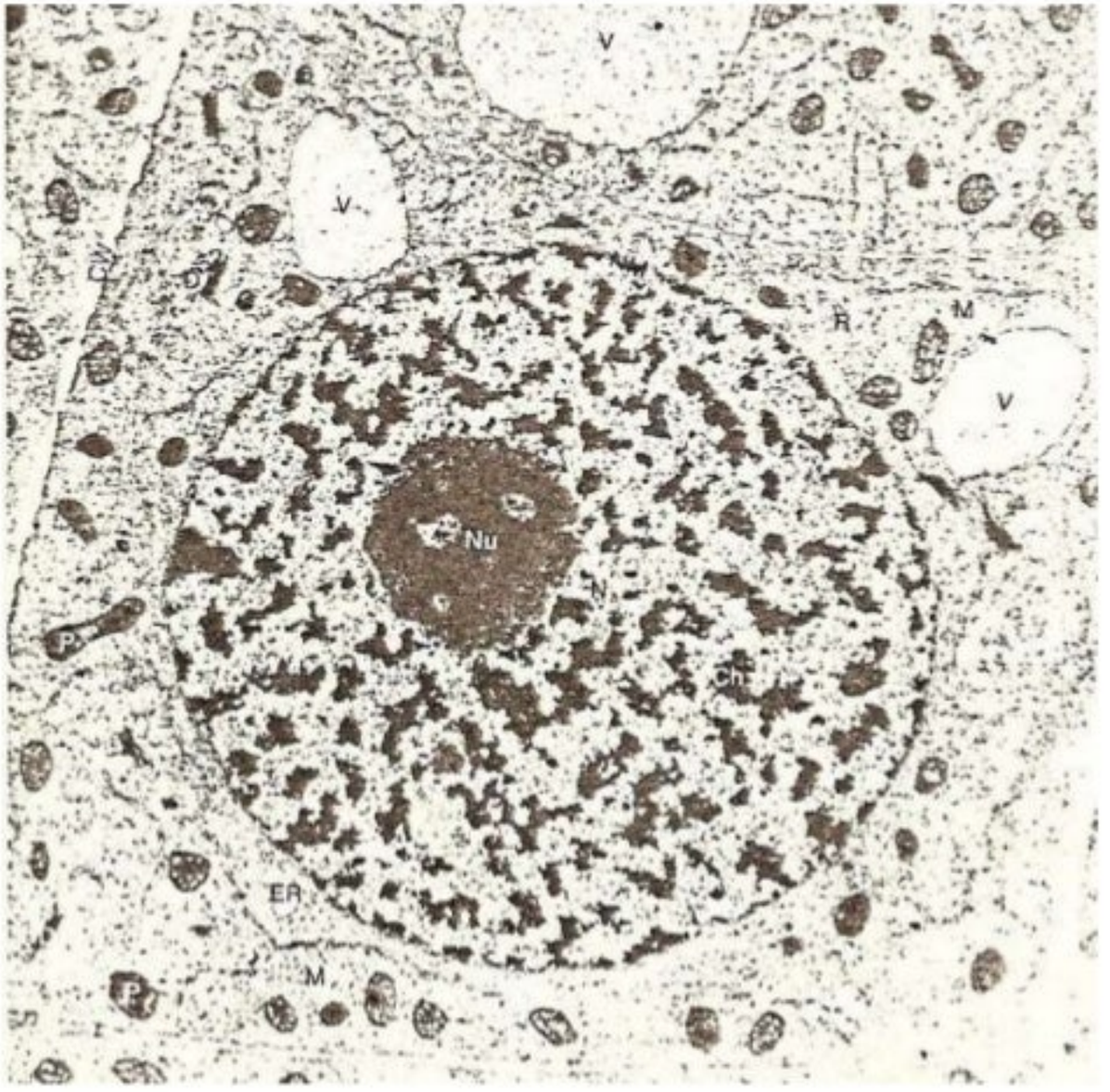
Four steps prepare the microscope for storage:

1. the 10X objective is in place
2. the stage is all the way down
3. the power is off
4. the cord is wrapped around the base

Magnifying using the Light Microscope



Magnifying using the Electron Microscope



Brightness and Contrast

- To make a microscale object in a material specimen visible, high magnification is not sufficient.
- A microscope should also generate sufficient *brightness and contrast of light from the object*.
- Brightness refers to the intensity of light.

– In a transmission light microscope the brightness is related to the numerical aperture (*NA) and magnification (M)*.

$$\text{Brightness} = \frac{(NA)^2}{M^2}$$

– In a reflected light microscope the brightness is more highly dependent on *NA*.

$$\text{Brightness} = \frac{(NA)^4}{M^2}$$

- These relationships indicate that the brightness decreases rapidly with increasing magnification, and controlling *NA is not only important for resolution but also for brightness, particularly* in a reflected light microscope.

– **Contrast** is defined as the relative change in light intensity (*I*) *between an object and its* background.

$$Contrast = \frac{I_{object} - I_{background}}{I_{background}}$$

Instrumentation

- Light microscope includes the following main components:
  - Illumination system;
  - Objective lens;
  - Eyepiece;
  - Photomicrographic system; and
  - Specimen stage.

Illumination System

- The illumination system of a microscope provides visible light by which the specimen is observed.
  - Low-voltage tungsten filament bulbs;
  - Tungsten–halogen bulbs; and
  - Gas discharge tubes.

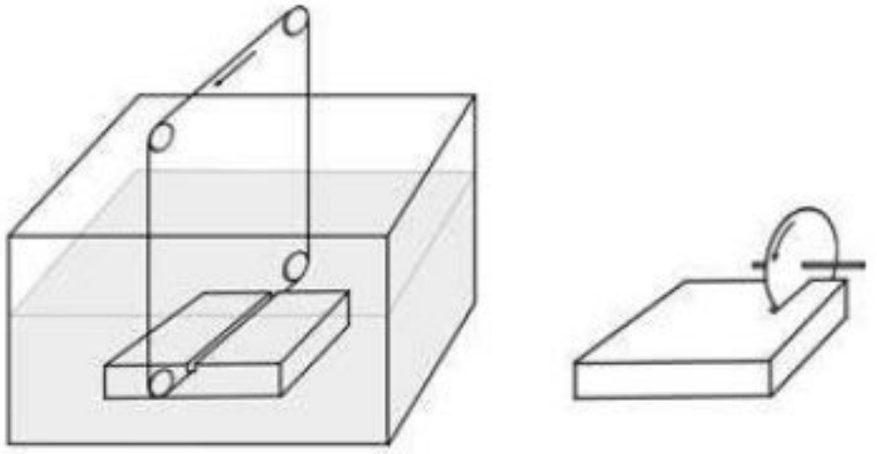
PREPARATION OF SPECIMEN

PREPARATION OF SPECIMEN

1. Sectioning
2. Mounting
3. Grinding
4. Polishing
5. Etching

Sectioning

- Sectioning serves two purposes:
  - generating a cross-section of the specimen to be examined;
  - reducing the size of a specimen to be placed on a stage of light microscope, or reducing size of specimen to be embedded in mounting media for further preparation processes.
- ***Cutting***



(a) (b)

Microtomy

- Microtomy refers to sectioning materials with a knife. It is a common technique in biological specimen preparation.
- It is also used to prepare soft materials such as polymers and soft metals.
- Tool steel, tungsten carbide, glass and diamond are used as knife materials.
- A similar technique, *ultramicrotomy, is widely used for the preparation of biological and polymer specimens in* transmission electron microscopy.

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Mounting

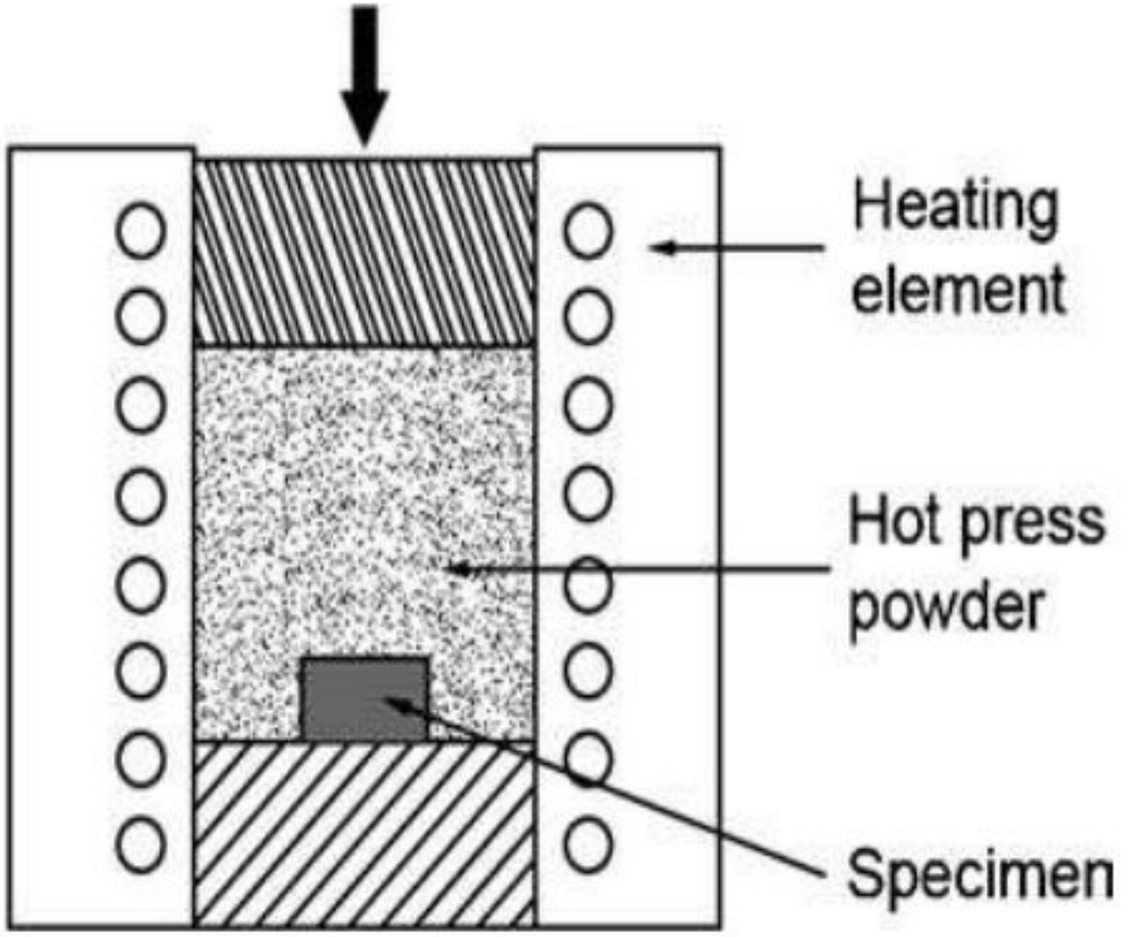
- Mounting refers to embedding specimens in mounting materials (commonly thermosetting polymers) to give them a regular shape for further processing.
- Mounting is not necessary for bulky specimens, but it is required for specimens that are too small or oddly shaped to be handled or when the edge of a specimen needs to be examined in transverse section.
- Mounting is popular now because most automatic grinding and polishing machines require specimens to have a cylindrical shape.
- There are two main types of mounting techniques:

  1. hot mounting

  2. cold mounting.

Hot Mounting

- Hot mounting uses hot-press equipment as shown in Figure.
- A specimen is placed in the cylinder of a press and embedded in polymeric powder.
- The surface to be examined faces the bottom of the cylinder. Then, the specimen and powder are heated at about 150 ◦C under constant pressure for tens of minutes.
- Heat and pressure enable the powder to bond with the specimen to form a cylinder.



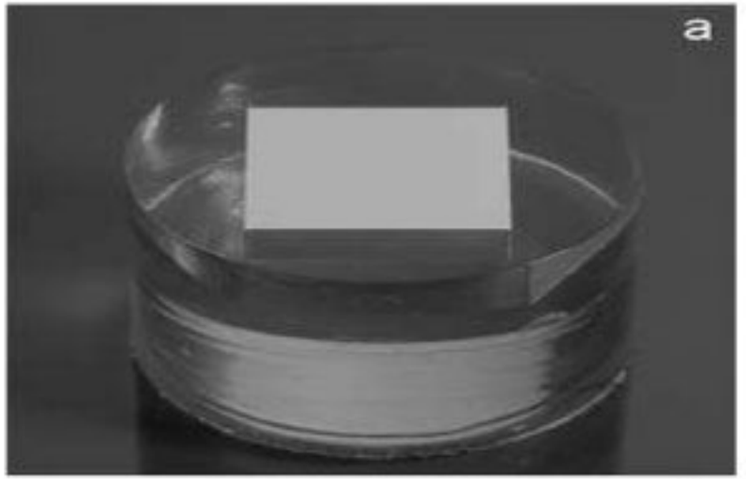
Cold Mounting

- In cold mounting, a polymer resin, commonly epoxy, is used to cast a mold with the specimen at ambient temperature.
- A cold mounting medium has two constituents: a fluid resin and a powder hardener.
- The resin and hardener should be carefully mixed in correct proportion.
- Curing times for mounting materials vary from tens of minutes to several hours, depending on the resin type.

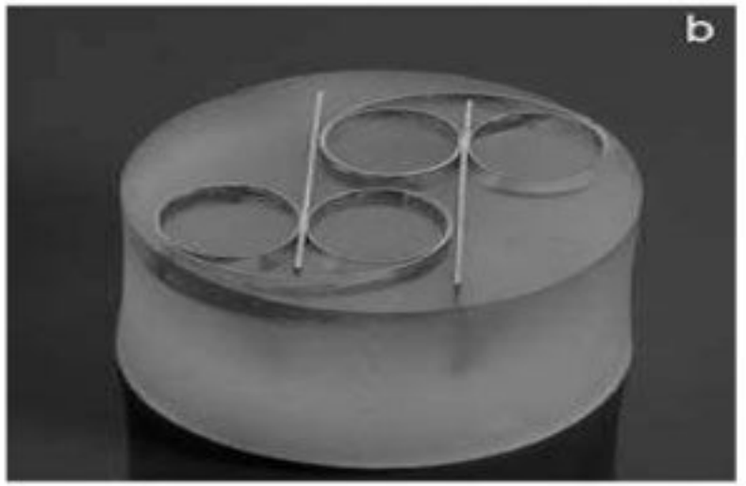
- Cold mounting of specimens:
- (a) place specimens on the bottom of molds supported by clamps

- (b) cast resin into the mold.

- Cold mounted specimens:
- (a) mounted with polyester



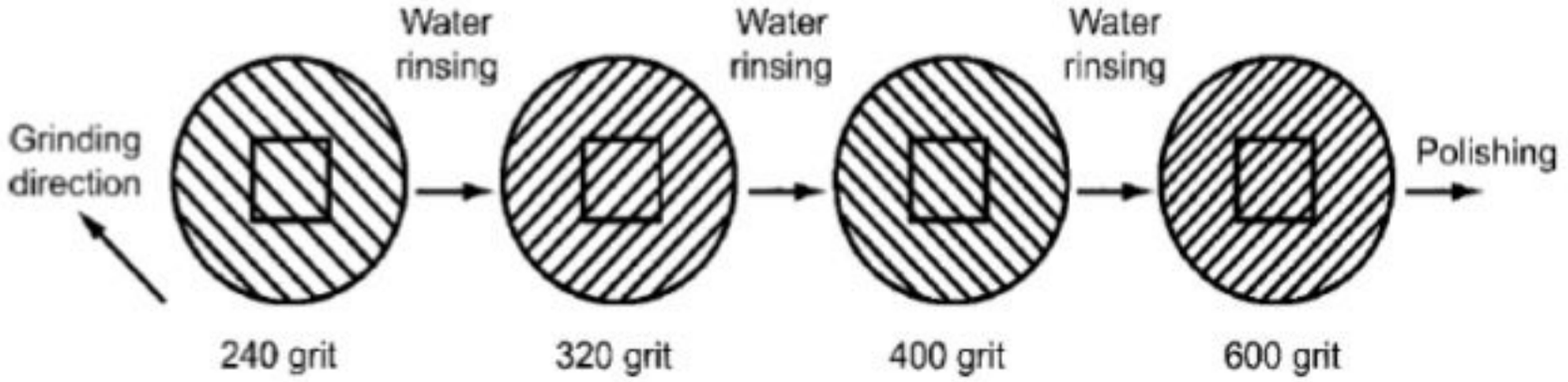
- (b) mounted with acrylic



- (c) mounted with acrylic and mineral fillers.

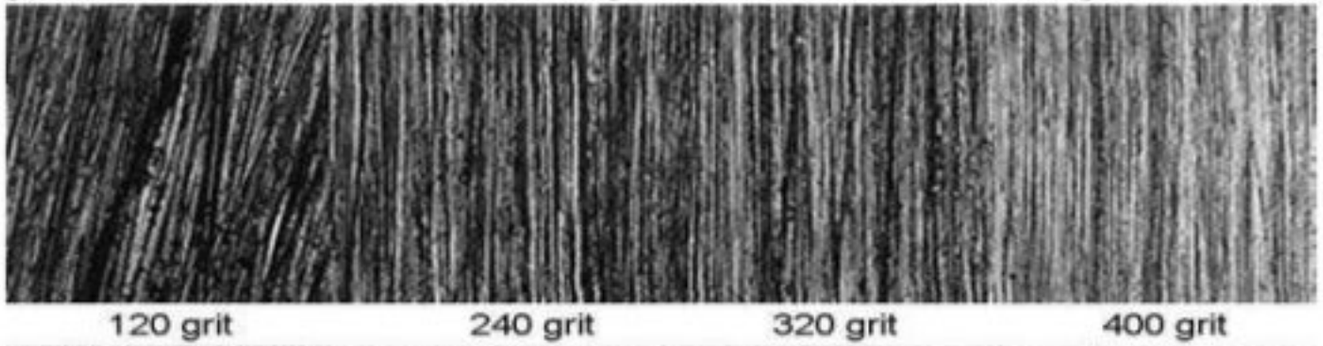
Grinding

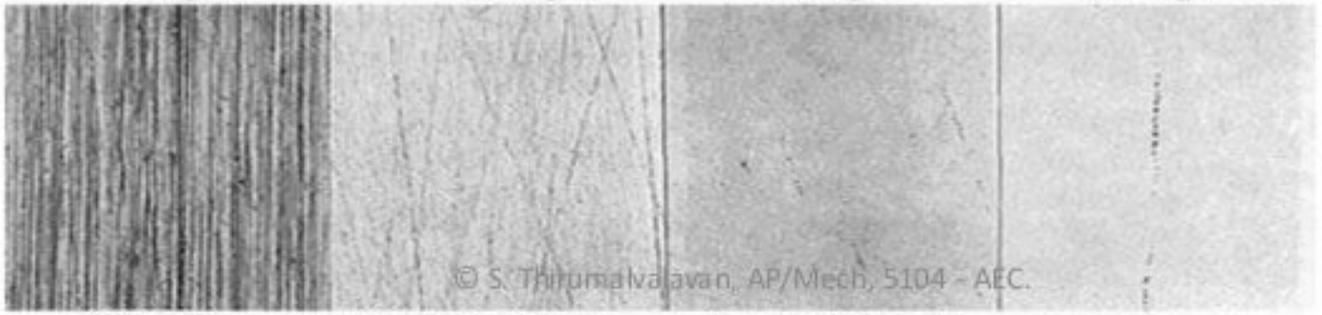
- Grinding refers to flattening the surface to be examined and removing any damage caused by sectioning.
- The specimen surface to be examined is abraded using a graded sequence of abrasives, starting with a coarse grit.
- Abrasive paper is graded according to particle size of abrasives such as 120-, 240-, 320-, 400- and 600-grit paper.
- Running water is supplied to cool specimen surfaces during hand grinding.



Polishing

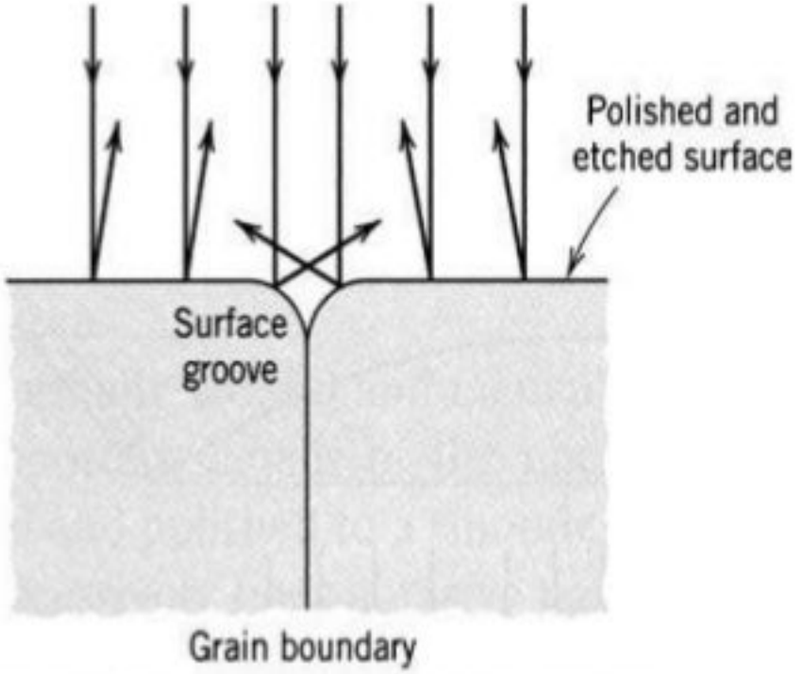
- Polishing is the last step in producing a flat, scratch-free surface.
- After being ground to a 600-grit finish, the specimen should be further polished to remove all visible scratches from grinding.
- Polishing generates a mirror-like finish on the specimen surface to be examined.
- Polishing is commonly conducted by placing the specimen surface against a rotating wheel either by hand or by a motor- driven specimen holder.
- Coarse polishing uses abrasives with a grit size in the range from 3 to 30μm.

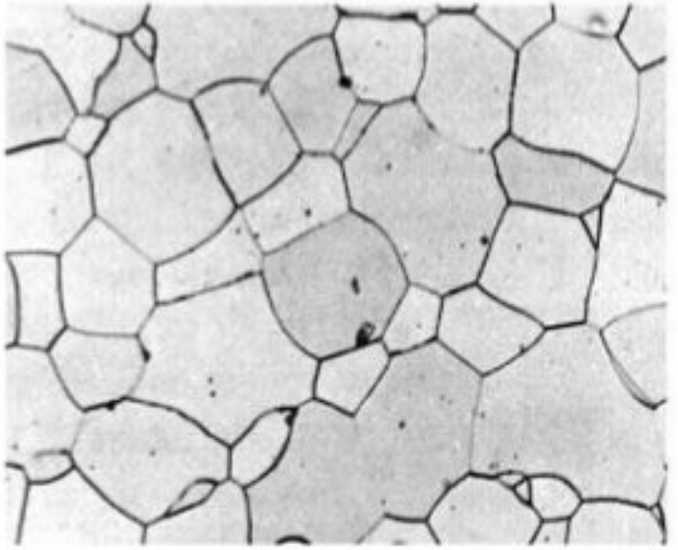




Etching

- Chemical etching is a method to generate contrast between microstructural features in specimen surfaces.
- Etching is a controlled corrosion process by electrolytic action between surface areas with differences in electrochemical potential.
- During etching, chemicals (*etchants) selectively* dissolve areas of the specimen surface because of the differences in the electrochemical potential by electrolytic action between surface areas that exhibit differences.





Optical Microscope - Working

https://www.youtube.com/watch?v=b2PCJ5s-iyk

https://www.youtube.com/watch?v=84hKLaOvumY

https://www.youtube.com/watch?v=kcyF4kLKQTQ

Sample Preparation - https://www.youtube.com/watch?v=4m-6fq5eCxI

https://www.youtube.com/watch?v=VR9d6RnmZww

Visual methods Microscopy

Principe of operation

–Optic or electronic measures

–Two dimensional projection

- Projection screen or circles
- Image analyzing programs

•Measures

– Feret diameters

– Equal circles

•Size range - 0.001-1000 μm

•Gives number average, or area average

Benefits

–"Simple" and spontaneous

–Give shape information

–Reasonable amount of sample

•Drawbacks

– Statistic relevance "tedious" if image analyse can not be used

– Risk for bias interpretation

– Difficult for high concentrations

– Sample preparation might be difficult

Visual
Different types of microscope

- Light microscope (1-1000 μm)
- Fluorescence microscope
- Confocal laser scanning microscopy
- Electron microscope
  - SEM (0.05-500 μm)
  - TEM (Å-0.1 μm)

Advantages

- Measuring microscopes are used for precision measurement
- It is relatively easy to use
- It is small and lightweight
- It offers high levels of observational quality
- It is unaffected by electromagnetic fields
- It does not require radiation to operate.
- It requires very little training.

Disadvantages

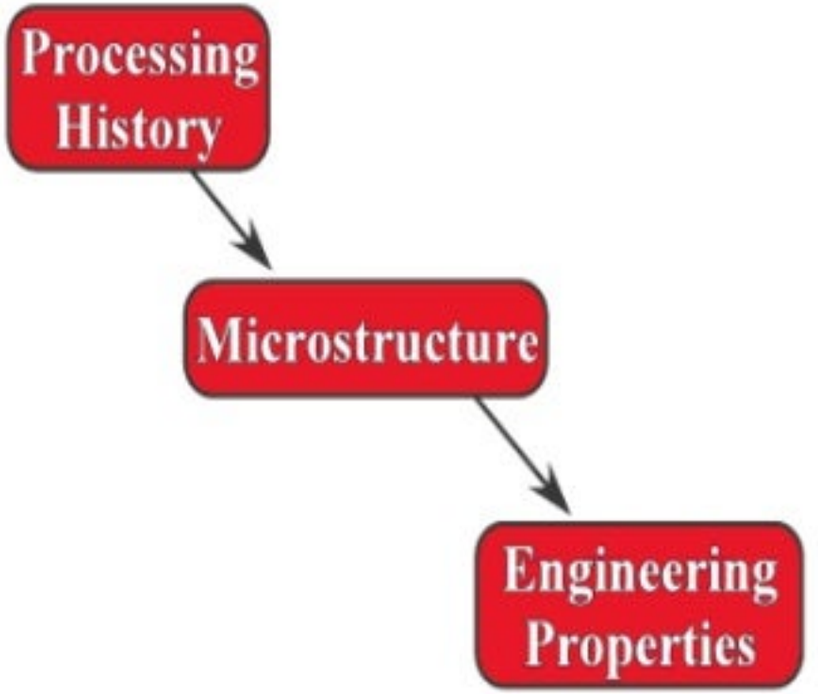
- Resolution limit of optical microscopes.
- Low magnification
- Separate sample preparation
- Poor surface view
- Light microscopes cannot operate in darkness
- Light microscopes cannot provide three-dimensional rendering.

Applications

- Optical microscopy is used for medical diagnosis.
- In industrial use, binocular microscope are common.
- In certain applications, long-working-distance or long-focus microscopes are beneficial.
- An item may need to be examined behind a window, or industrial subjects may be a hazard to the objective.

Microstructure of Engineering Materials

- Microstructure of an engineering material is a result of its chemical composition and processing history . It also determine chemical, physical and mechanical property.
- ***Microstructure*** influences virtually all aspects of the behavior of materials.
- ***Macrostructure*** is the spatial distribution of material in the useful object that is the goal of the endeavor.



ELECTRON MICROSCOPY

Electron Microscopy

- Electron microscopes generate images of material microstructures with much higher magnification and resolution than light microscopes.

- The high resolution of electron microscopes results from short wavelengths of the electrons used for microscope illumination.

- The wave-length of electrons in electron microscopes is about 10,000 times shorter than that of visible light.

There are two main types of electron microscopes:

1. Transmission Electron Microscopes (TEM) &
2. Scanning Electron Microscopes (SEM).

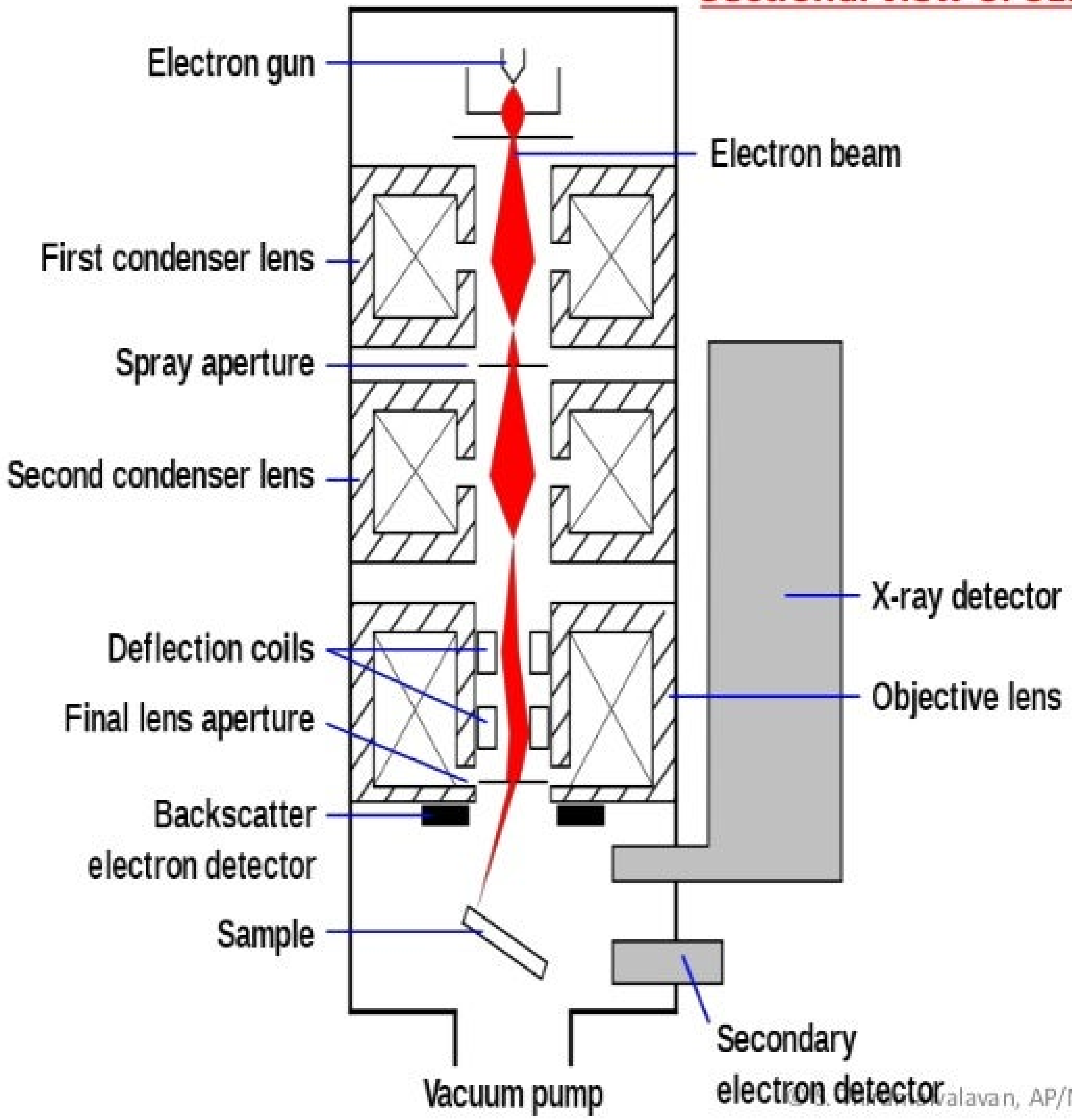
- The optics of the TEM is similar to the conventional transmission light microscope, while that of SEM is more like that of scanning confocal laser microscopes.

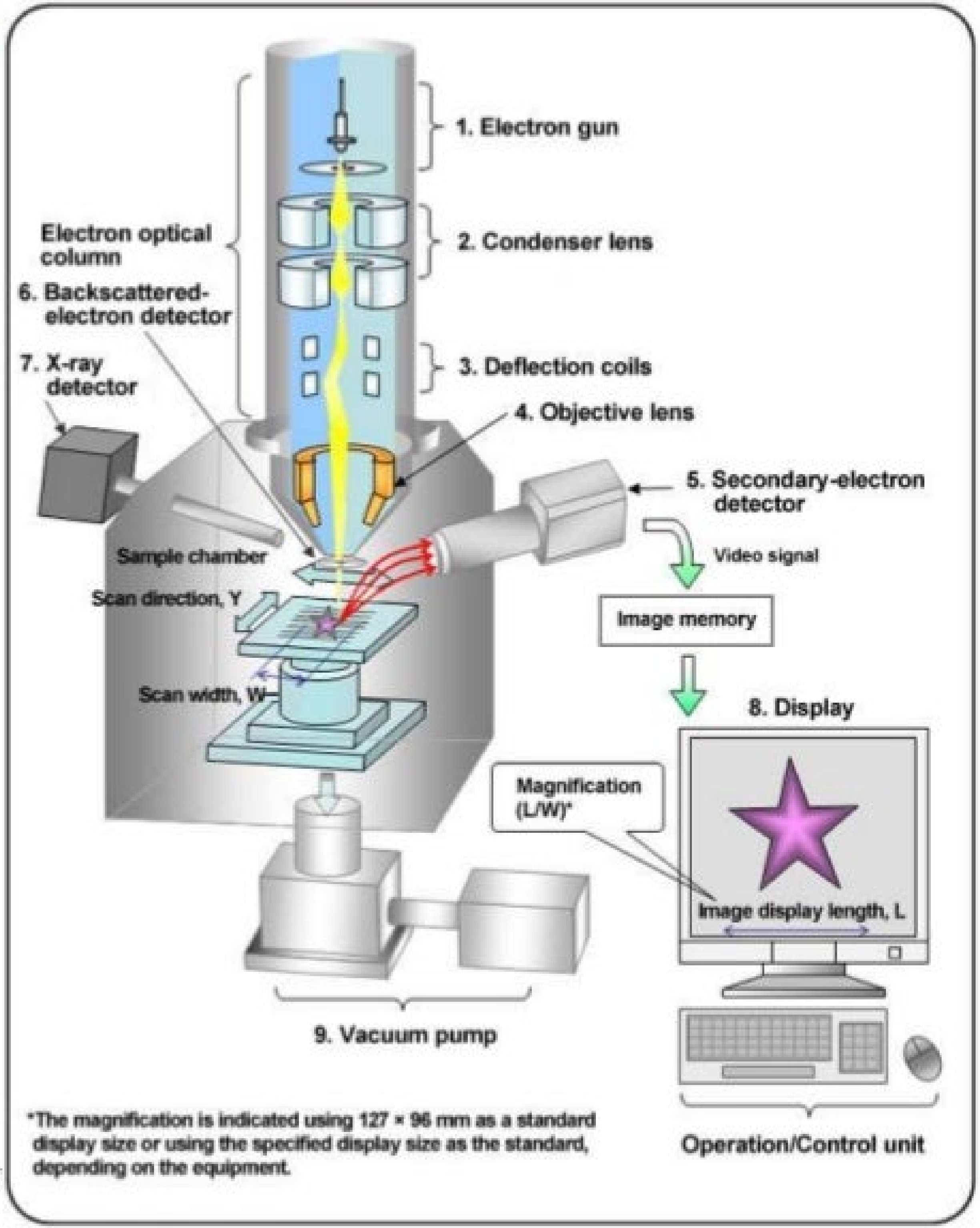
1. Scanning Electron Microscopes (SEM)

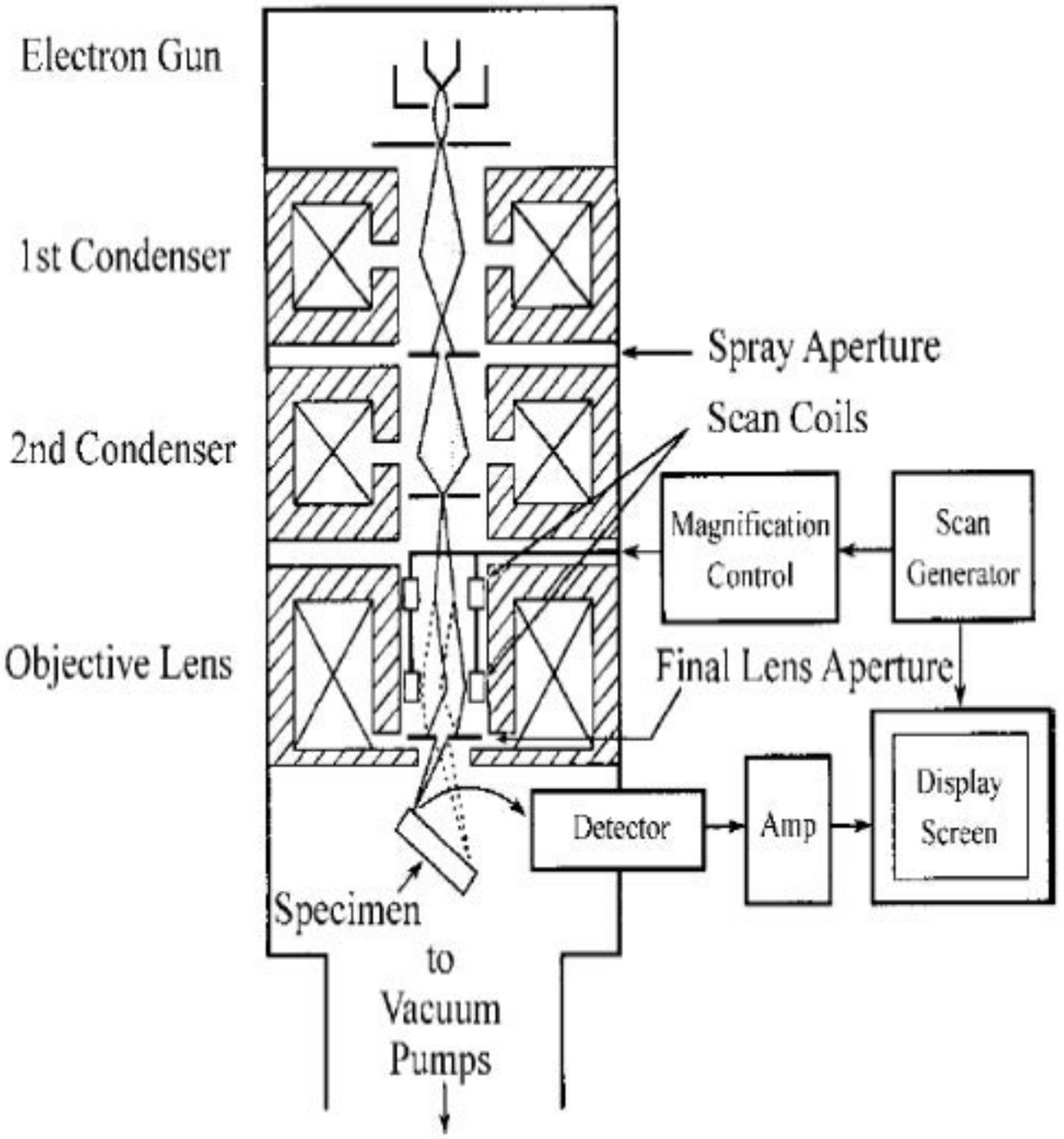
Scanning Electron Microscopes (SEM)

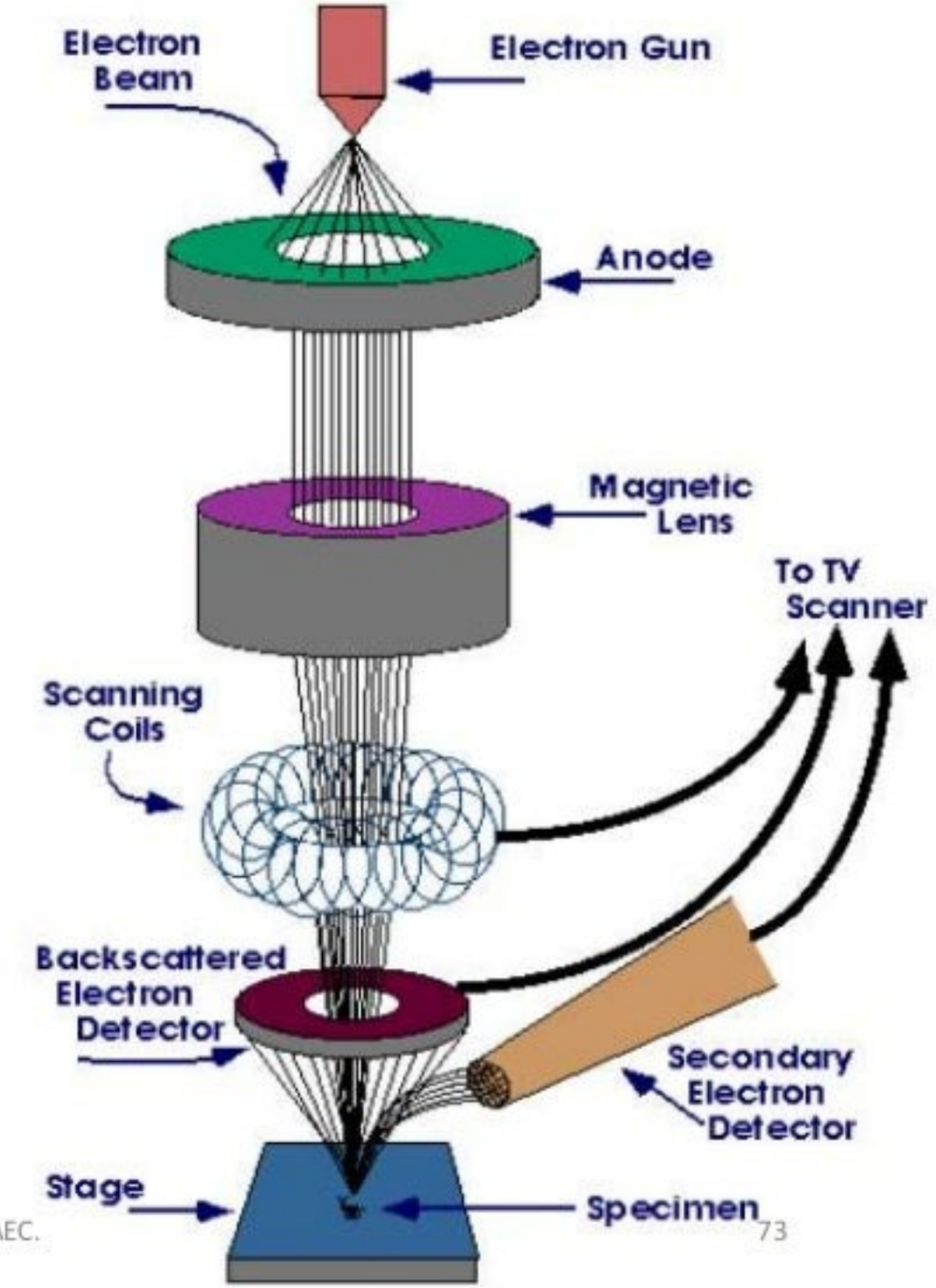
- **PRINCIPLE :** A SEM is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons.
- The electrons interact with atoms in the sample, producing various signals that contain information about the surface topography and composition of the sample.

Sectional view of SEM

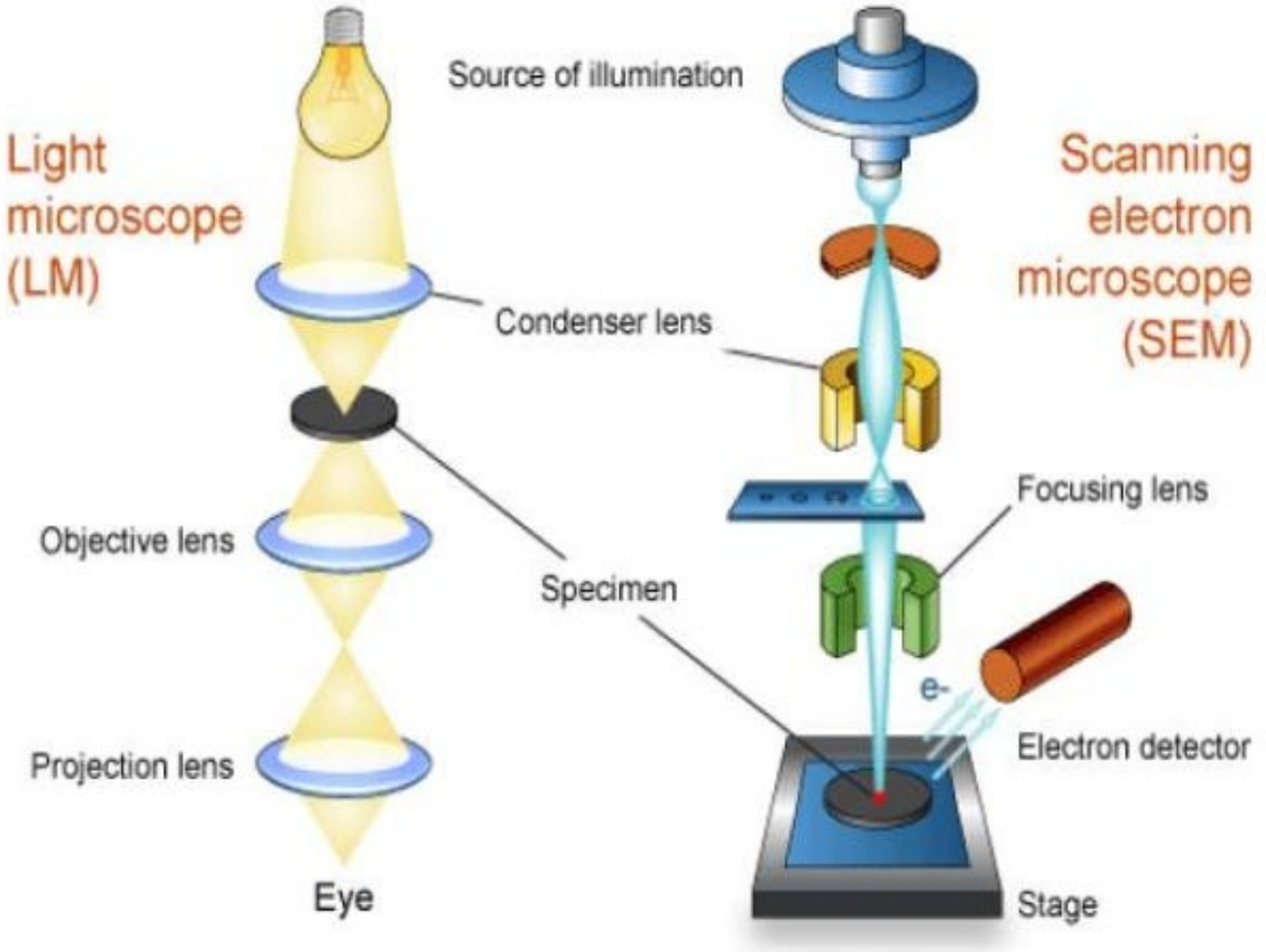





Electron Gun
1st Condenser
2nd Condenser
Objective Lens
Spray Aperture
Scan Coils
Magnification Control
Scan Generator
Final Lens Aperture
Detector
Amp
Display Screen
Specimen
to
Vacuum
Pumps


Electron Beam
Electron Gun
Anode
Magnetic Lens
To TV Scanner
Scanning Coils
Backscattered Electron Detector
Secondary Electron Detector
Stage
Specimen

Light Microscope Vs Scanning Electron Microscope



Components of SEM

- Electron gun
- Condenser lens
- Vacuum chamber
- Deflector coils
- Secondary electron detector
- Image display and recording
- Specimen stage

Specimen Loading Stages

- The specimen must meet the following requirements before it is loaded to the SEM stage,

(i) Surface preparation

(ii) Mounting specimen

(iii) Specimen coating

i) Surface Preparation

- Fracturing
- Cutting
- Mechanical Polishing
- Milling by the ion beam
- Contrast enhancement

ii) Mounting Specimen

- Bulk specimen
- Powders and Particles

iii) Specimen coating

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

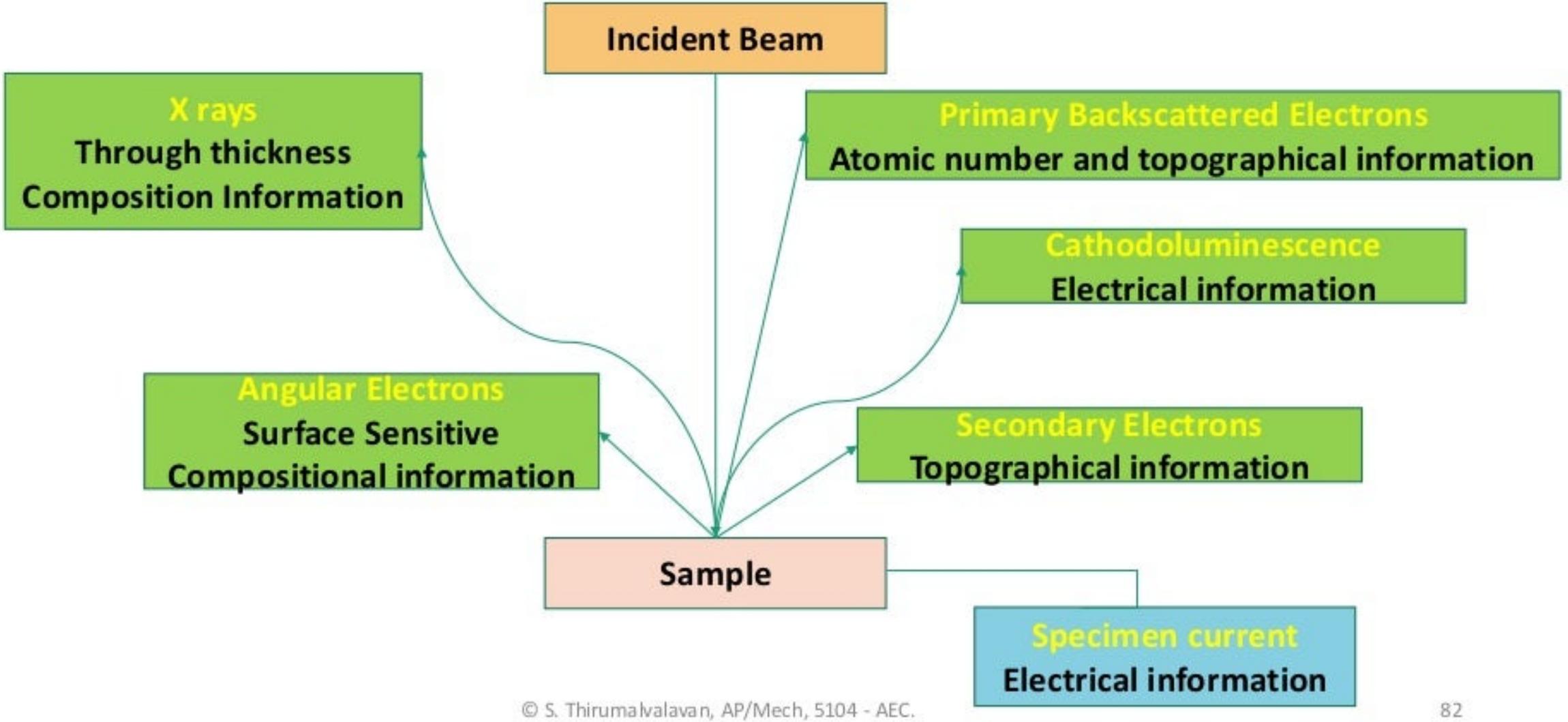
WORKING – SEM

- 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 0.4nm to 5nm 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-ray 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.
- Electron amplifiers of various types are used to amplify the signals, which are displayed as variations is brightness on a computer monitor.

Output - Types Scattered Electrons

- **X-RAYS:** X-rays, emitted from beneath the sample surface, can provide element and mineral information.



- 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.
- 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.

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.

Advantages

- 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

Disadvantages

- Size restrictions may require cutting the sample
- The size is not portable
- SEMs are expensive and large
- Maintenance involves keeping a steady voltage, currents to electromagnetic coils and circulation of cool water.
- 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 of radiation exposure
- Training is required to operate.

Applications

1. The SEM also excels in producing

- Detailed surface topography images
- Failure analysis
- Dimensional analysis
- Process characterization
- Reverse engineering
- Particle identification
- Surface 3D
- Elemental analysis

2. Ideal Uses

- High resolution surface topography images
- Elemental microanalysis and particle characterization

SEM - Working

https://www.youtube.com/watch?v=GY9lfO-tVfE

https://www.youtube.com/watch?v=Mr9-1Sz_CK0

https://www.youtube.com/watch?v=KfQ4VNpWN4M

https://www.youtube.com/watch?v=qExUDTqi8Xc

https://www.youtube.com/watch?v=edk48r78-jY

https://www.youtube.com/watch?v=HpgdP2QEahY

2. Transmission Electron Microscopes (TEM)

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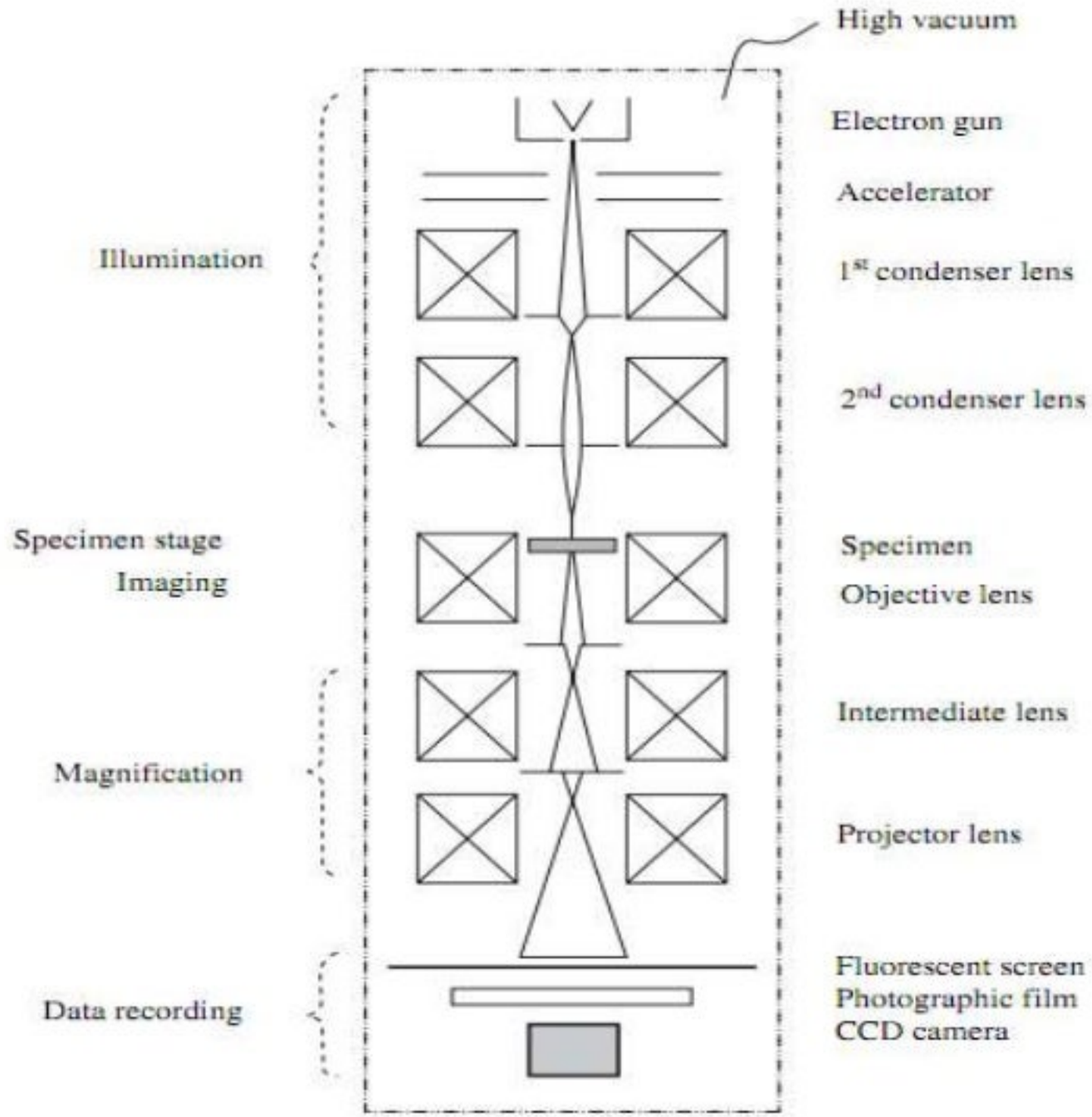
Department of Mechanical Engineering,

E-Mail : thirumalbemech@gmail.com

Transmission Electron Microscopes (TEM)

- **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.



In TEM,

Visible light → Electron ray

Glass lenses → Electromagnetic lens

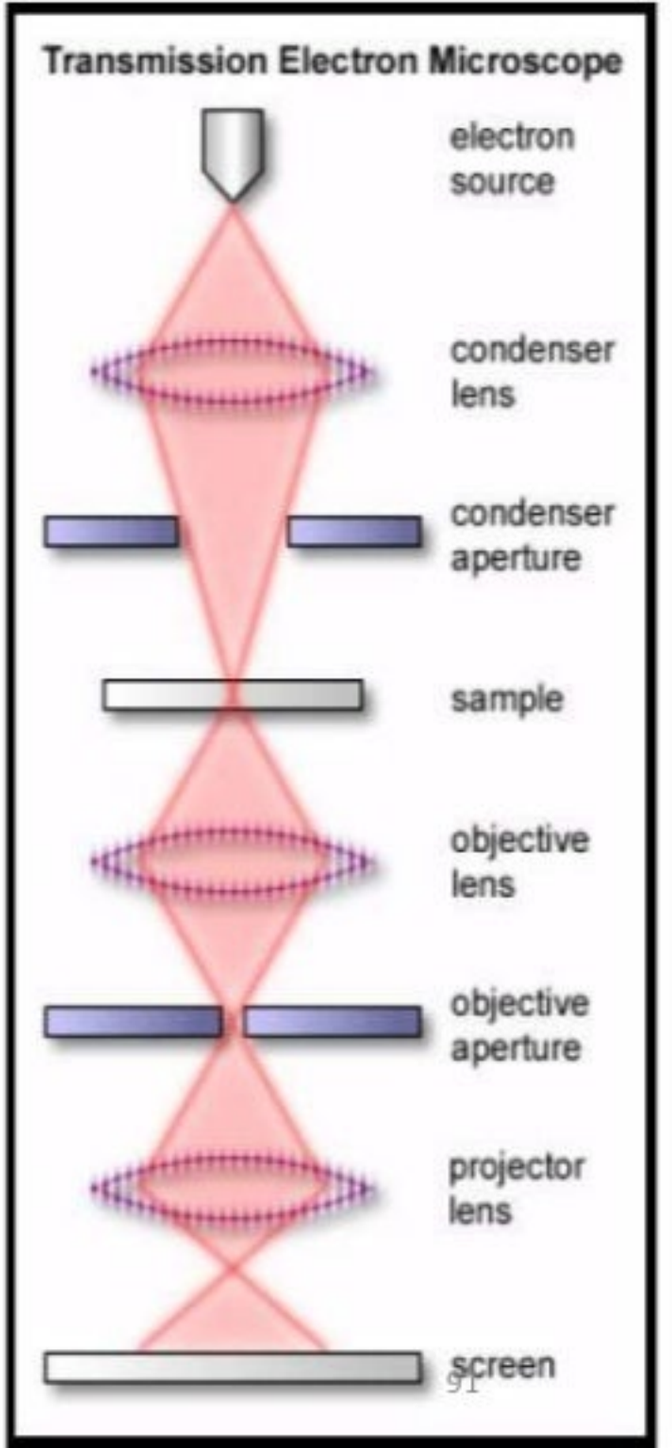
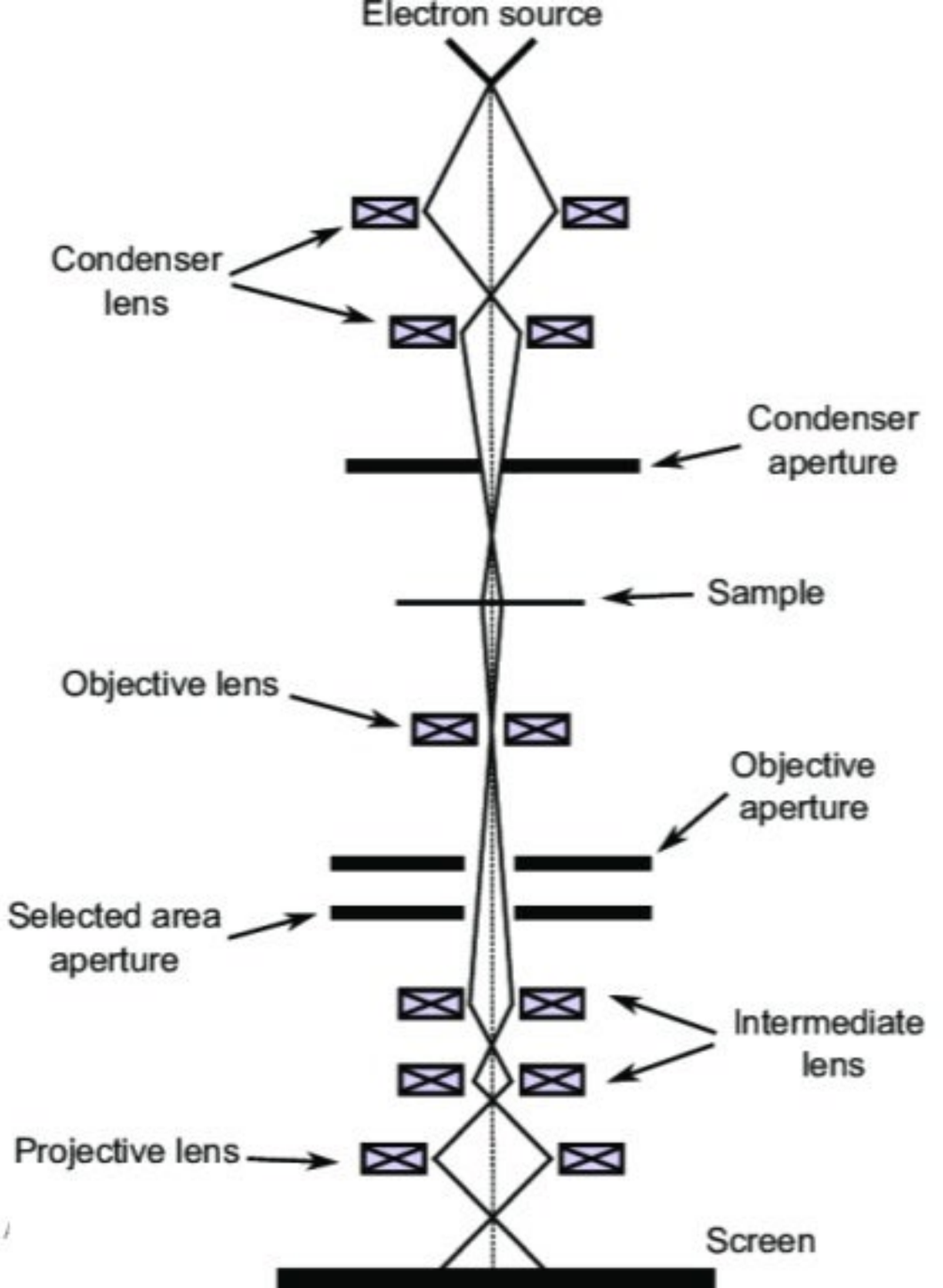


Figure 3.1 Structure of a transmission electron microscope and the optical path.

Components of TEM

- Electron source
- Electromagnetic lenses
- Vacuum chamber
- Condensers
- Sample stage
- Phosphor or fluorescent screen
- Condenser lens
- Objective aperture



Electron Sources

- In a TEM system, an electron gun generates a high energy electron beam for illumination.
- In the electron gun, the electrons emitted from a cathode, a solid surface, are accelerated by high voltage (Vo) to form a high energy electron beam with energy E = eVo.
- To achieve a high resolution, the TEM is usually operated under an acceleration voltage of greater than 100 kV.
- *In practice, 200 kV is commonly used and meets most resolution requirements.*

- ***The general structure of an electron gun is composed of three main parts:***

  Cathode/Electron source,
  Wehnelt electrode &
  Anode, as illustrated in Figure.

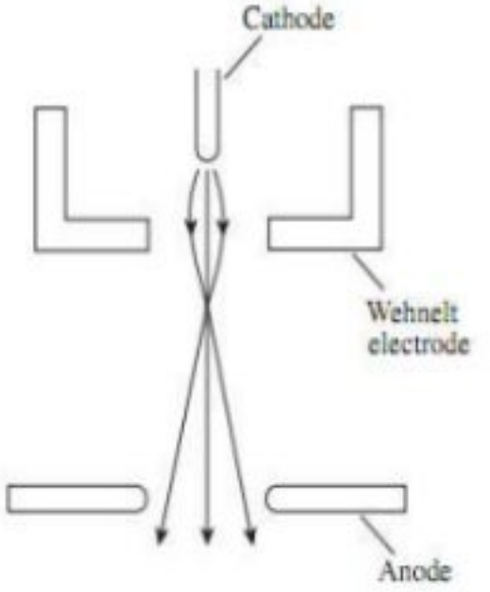


Figure 3.2 General structure of an electron gun.

Electrons are emitted from the surface of the cathode and accelerated by an electric field toward the cathode.
The Wehnelt electrode is placed between the cathode and the anode.

It is biased a few hundred volts negative with respect to the cathode in order to stabilize the electron beam against voltage fluctuation by reducing the electron beam current whenever necessary.

- ***There are two basic types of electron guns:***
  Thermionic emission & Field emission.

Thermionic emission, this is the most common type of electron gun and includes the tungsten filament gun and the lanthanum hexaboride (LaB6) gun. The tungsten gun uses a tungsten filament as the cathode.

Field emission, this type of electron gun does not need to provide thermal energy for electrons to overcome the surface potential barrier of electrons. Electrons are pulled out by applying a very high electric field to a metal surface.

Table 3.3 Comparison of Electron Guns

	Tungsten filament	LaB_6	Field emission	
			Thermal	Cold
Operation temperature (K)	~2800	~1800	1600 ~ 1800	~300
Brightness[a] At 200 kV (A cm^{-2} sr)	$\sim 5 \times 10^5$	$\sim 5 \times 10^6$	$\sim 5 \times 10^8$	$\sim 5 \times 10^8$
Requirement to vacuum (Torr[b])	10^{-4}	10^{-6}–10^{-7}	10^{-9}	10^{-9}–10^{-10}

[a] Intensity emitted per unit cathode surface in unit solid angle.

[b] 1 torr = 133 Pa.

Specimen Preparation

- Preparation of specimens often is the most tedious step in TEM examination. To be electronically transparent, the material thickness is limited.
- We have to prepare a specimen with at least part of its thickness at about 100 nm, depending on the atomic weight of specimen materials.
- For higher atomic weight material, the specimen should be thinner. A common procedure for TEM specimen preparation is described as follows.

Pre-Thinning

- Pre-thinning is the process of reducing the specimen thickness to about 0.1mm before final thinning to 100 nm thickness.
- First, a specimen less than 1mm thick is prepared. This is usually done by mechanical means, such as cutting with a diamond saw.
- Then, a 3mm diameter disc is cut with a specially designed punch before further reduction of thickness.
- Grinding is the most commonly used technique to reduce the thickness of metal and ceramic specimens.

Figure 3.8 Hand-grinding jig for TEM specimen preparation. A disc (S) is glued to the center of the jig and the guide ring (G) controls the amount of grinding.

Final Thinning

Three methods of final thinning are described here: electrolytic thinning for metal specimens that are good electric conductors, ion milling for metal and ceramic specimens, and ultramicrotomy cutting for polymeric and biological specimens.

Electrolytic Thinning

- Electrolytic thinning and ion milling are methods for reducing specimen thickness to the scale of 100 nm.
- These methods create a dimpled area on pre-thinned specimens because it is almost impossible to reduce the thickness of specimens uniformly to the level of electron transparency.
- The dimpled area is likely to have regions of electron transparency as schematically shown in fig.

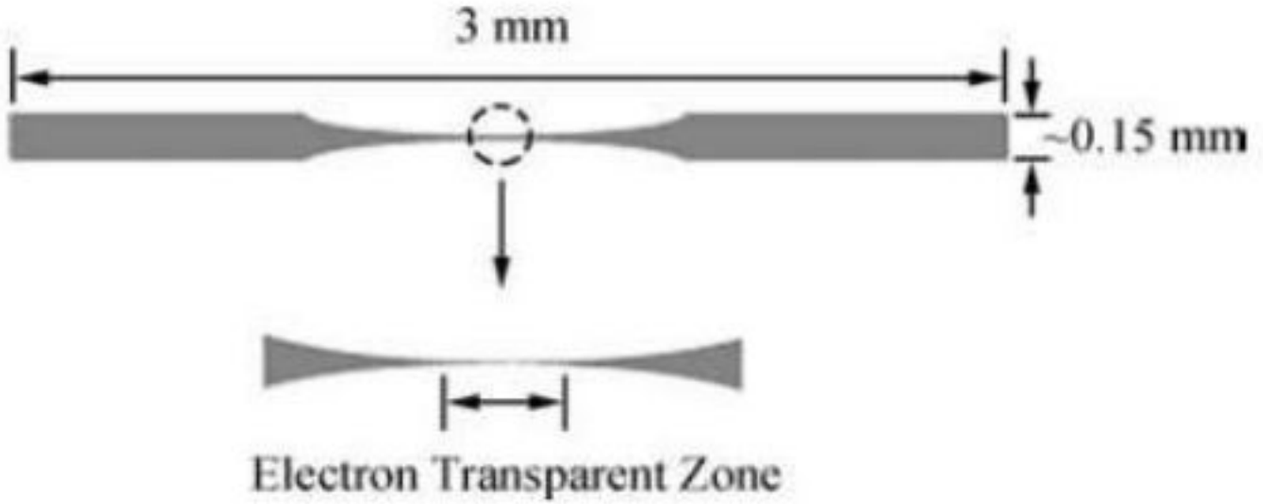
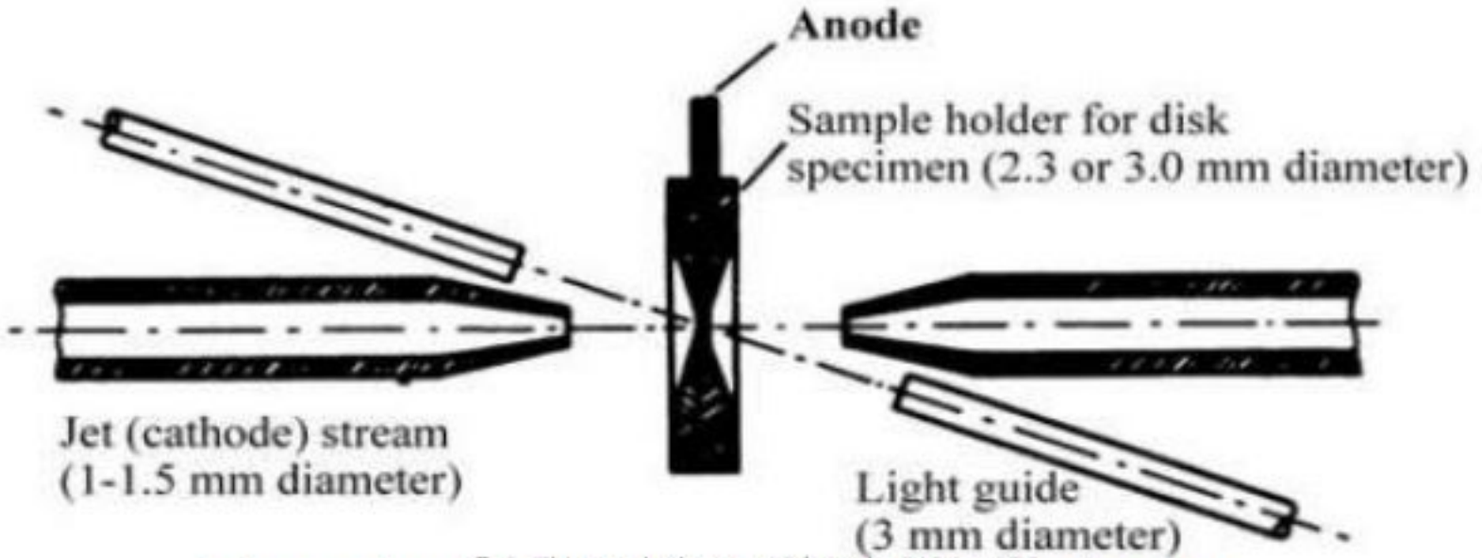


Figure 3.9 A dimple in the center of a specimen disc.

- A specimen is placed in an electrochemical cell with the specimen as anode. A suitable electrolyte (usually an acidic solution) is used to electrochemically reduce specimen thickness.
- A common technique is jet polishing, illustrated schematically in Fig. A specimen is placed in a holder with two sides facing guides of electrolyte jet streams, which serve as cathode.
- The electrolyte jet polishes both sides of the specimen until light transparency is detected by a light detector.
- Catching the precise moment that tiny holes appear is crucial, because only the edge of a tiny hole contains thin sections of electron transparency.
- Electrolytic thinning is very efficient and is completed in only 3–15 minutes.



Electrochemical jet-polishing schematic diagram

Ion Milling

- Ion milling uses a beam of energetic ions to bombard specimen surfaces in order to reduce the thickness by knocking atoms out of a specimen.

- The specimen does not need to be electrically conductive for ion milling. Thus, the technique is suitable for metal, ceramics and other materials, as long as they are thermally stable.

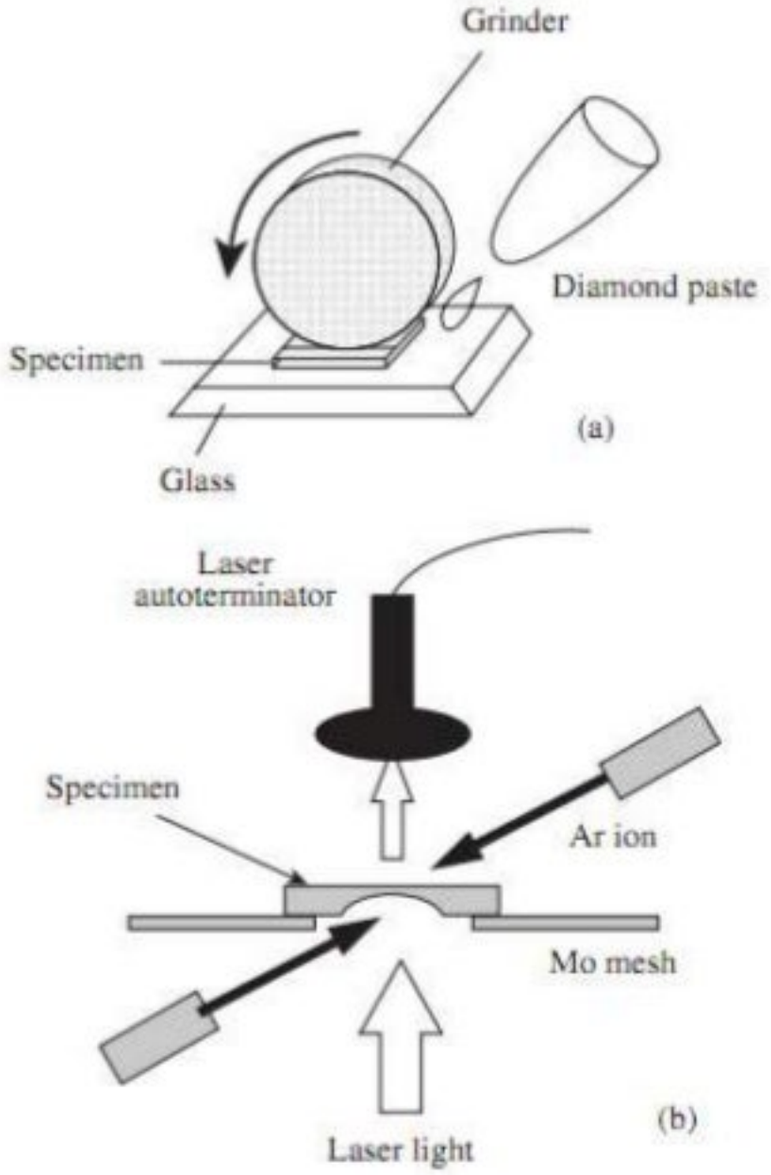
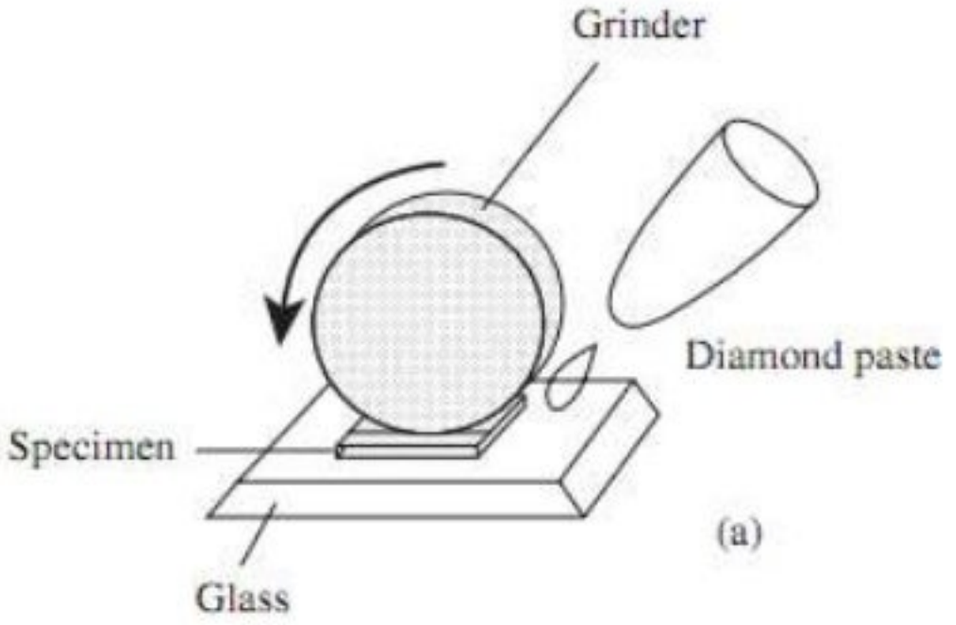
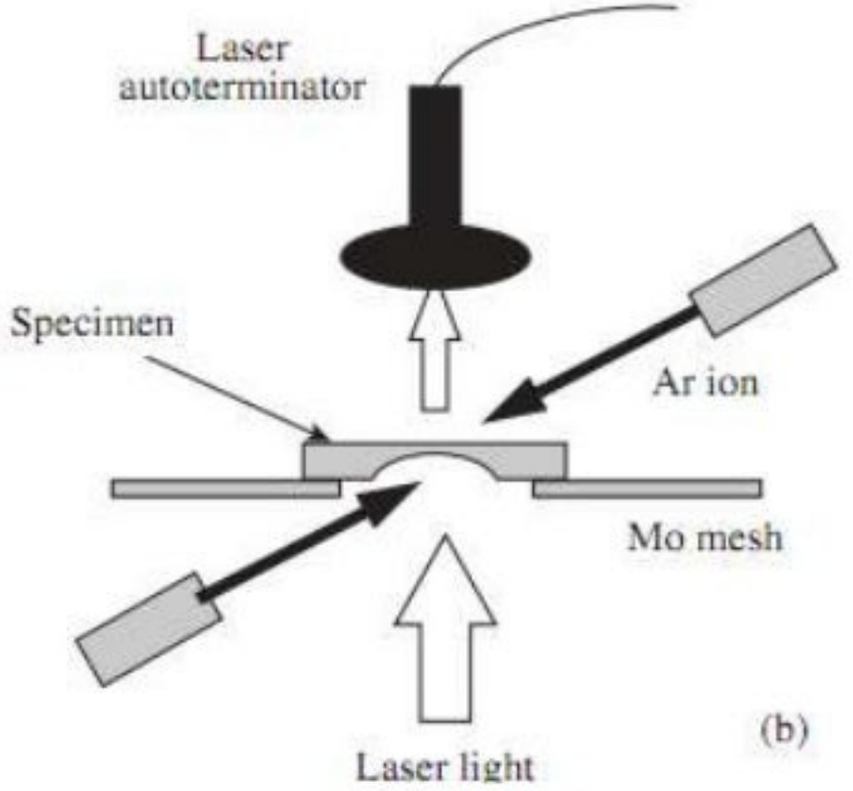


Figure 3.11 Ion thinning process: (a) dimple grinding; and (b) ion milling. (Reproduced with kind permission of Springer Science and Business Media from D. Shindo and T. Oikawa, *Analytical Electron Microscopy for Materials Science*, Springer-Verlag, Tokyo. © 2002 Springer-Verlag GmbH.)

Figure illustrates the general procedure of ion milling. Before ion milling, the specimen is often ground with a dimple grinding device in order to reduce the thickness in the central are of specimen.



- Then, the ground specimen is cut as a 3-mm disc and placed in the ion milling chamber with a geometric arrangement as shown in Figure.



- The edge of the specimen disc may need to be covered by a molybdenum ring in order to strengthen it during ion milling.
  An ion beam with energy of 1–10 keV bombards the specimen.
- The specimen is placed in the center at an angle of about 5–30◦ to the ion beam in order have a high yield of sputtering. Light transparency is detected by a light detector aligned along the vertical direction. The ion beam can raise the temperature of the specimen rapidly and dramatically.
- Thus, cooling the chamber with liquid nitrogen is necessary in order to prevent specimen heating by the ion beam. Even with cooling, organic materials are not suitable for ion milling because the ion beam can cause severe damage to the microstructure of organic specimens such as polymers.

Ultramicrotomy

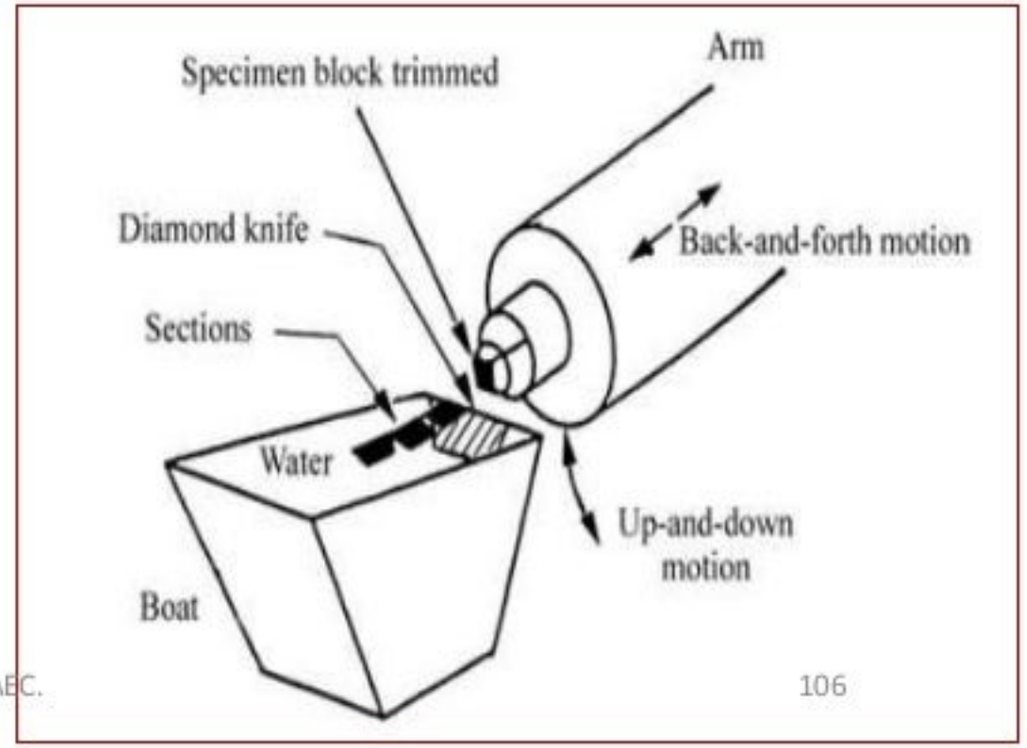
- Ultramicrotomy is basically the same type of method as microtomy for preparing soft specimens for light microscopy.

- However, ultramicrotomy can be used to section a specimen to the 100nm scale. It is commonly used to prepare polymeric or biological TEM specimens.

Figure illustrates the working principles of the instrument.

- A specimen is mounted in a holder against the cutting tool (glass knife or diamond knife).
- The specimen should be trimmed to have a tip held against the knife. The cross-section of the trimmed tip usually is only about 1mm^2 for diamond knife cutting.
- The holder gradually moves toward the knife while it re-peatedly moves up and down.
- The firmly mounted specimen is sectioned as it passes the edge of the knife blade.
- To be sectioned by an ultramicrotome, materials must be

softer than the knife. Even though diamond is the hardest material available, a specimen can still damage the tip of a diamond knife because the extremely sharp tip of a diamond knife makes it very fragile.



In other way – Method of Specimen Preparation

Simply,

- Ultra microtome
- Ultrasonic disk cutting
- Dimpling
- Ion milling
- Mechanical milling
- Chemical etching
- Ion etching
- Replication

Components of TEM

- Electron sources
- Electromagnetic lenses
- Vacuum chamber
- Condensers
- Sample stage
- Phosphor or Fluorescent screen
- Condenser lens
- Objective aperture

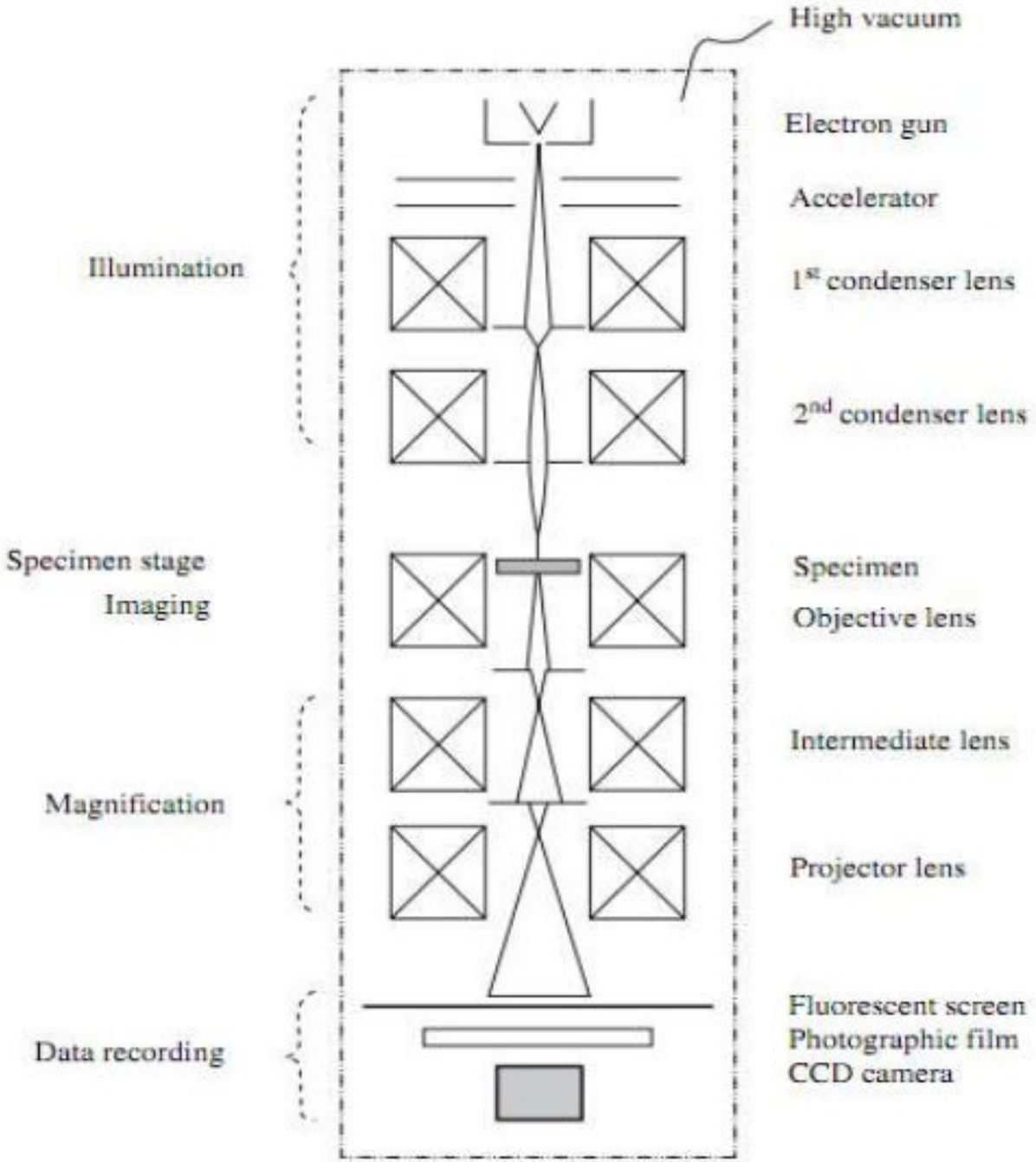


Figure 3.1 Structure of a transmission electron microscope and the optical path.

WORKING – TEM

- TEM employ a high voltage electron beam in order to create an image
- 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 and the electron wither 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 grater 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 scree. 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.

Operation Modes of TEM

- After interaction with the sample, on the exit surface of the specimen two types of electrons exist – Unscattered and scattered electrons.

- The two basic operation modes of TEM

(i) Imaging mode

(ii) Diffraction mode

Resolution - TEM

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

Table 3.1 Comparison Between Light and Transmission Electron Microscopes

	Transmission light microscope	Transmission electron microscope
Specimen requirements	Polished surface and thin section (<100 μm)	Thin section (<200 nm)
Best resolution	~200 nm	~0.2 nm
Magnification range	2–2000×	500–1,000,000×
Source of illumination	Visible light	High-speed electrons
Lens	Glass	Electromagnetic
Operation environment	Air or liquid	High vacuum
Image formation for viewing	By eye	On phosphorescent plate

Advantages

- The highest spatial resolution elemental mapping of any analytical technique (0.2nm (2 Å) 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
- Image are high quality and detailed

Disadvantages

- Significant sample preparation time (1-4 hrs)
- 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 require special training
- Image are black and white

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 nanometer scale
- Catalyst studies
- Nanometer scale elemental maps
- Super lattice characterization
- Energy filtered imaging (EFTEM)
- Colleges and universities can utilize TEMs for research and studies

TEM - Working

https://www.youtube.com/watch?v=DTIlLeCU5JQ

https://www.youtube.com/watch?v=omcxfWGJzsw

https://www.youtube.com/watch?v=ON6MfBBBq6k

Electron microscope | TEM | SEM | Cryo EM - https://www.youtube.com/watch?v=t4hhdgJADE8

THE LIGHT MICROSCOPE Vs THE ELECTRON MICROSCOPE

FEATURE	LIGHT MICROSCOPE	ELECTRON MICROSCOPE
Electromagnetic spectrum used	Visible light 760nm (red) – 390nm Colours visible	Electrons app. 4nm Monochrome
Maximum resolving power	app. 200nm	0.2nm Fine detail
Maximum magnification	x1000 – x1500	x500 000
Radiation source	Tungsten or quartz halogen lamp	High voltage (50kV) tungsten lamp
Lenses	Glass	Magnets
Interior	Air-filled	Vacuum
Focussing screen	Human eye (retina), photographic film	fluorescent (TV) screen, photographic film

THE LIGHT MICROSCOPE Vs THE ELECTRON MICROSCOPE

FEATURE	LIGHT MICROSCOPE	ELECTRON MICROSCOPE
Preparation of specimens	Temporary mounts living or dead	Tissues must be dehydrated = dead
Embedding	Wax	Resin
Sectioning	Hand or microtome slices ≤ 20 000nm Whole cells visible	Microtome only. Slices ≤ 50nm Parts of cells visible
Stains	Water soluble dyes	Heavy metals
Support	Glass slide	Copper grid

DIFFRACTION TECHNIQUES

DIFFRACTION : Diffraction refers to various phenomena that occur when a wave encounters an obstacle or a slit. It is defined as the bending of waves around the concerns of an obstacle or through an aperture into the region of geometrical shadow of the obstacle / aperture.

Fundamentals of Diffraction

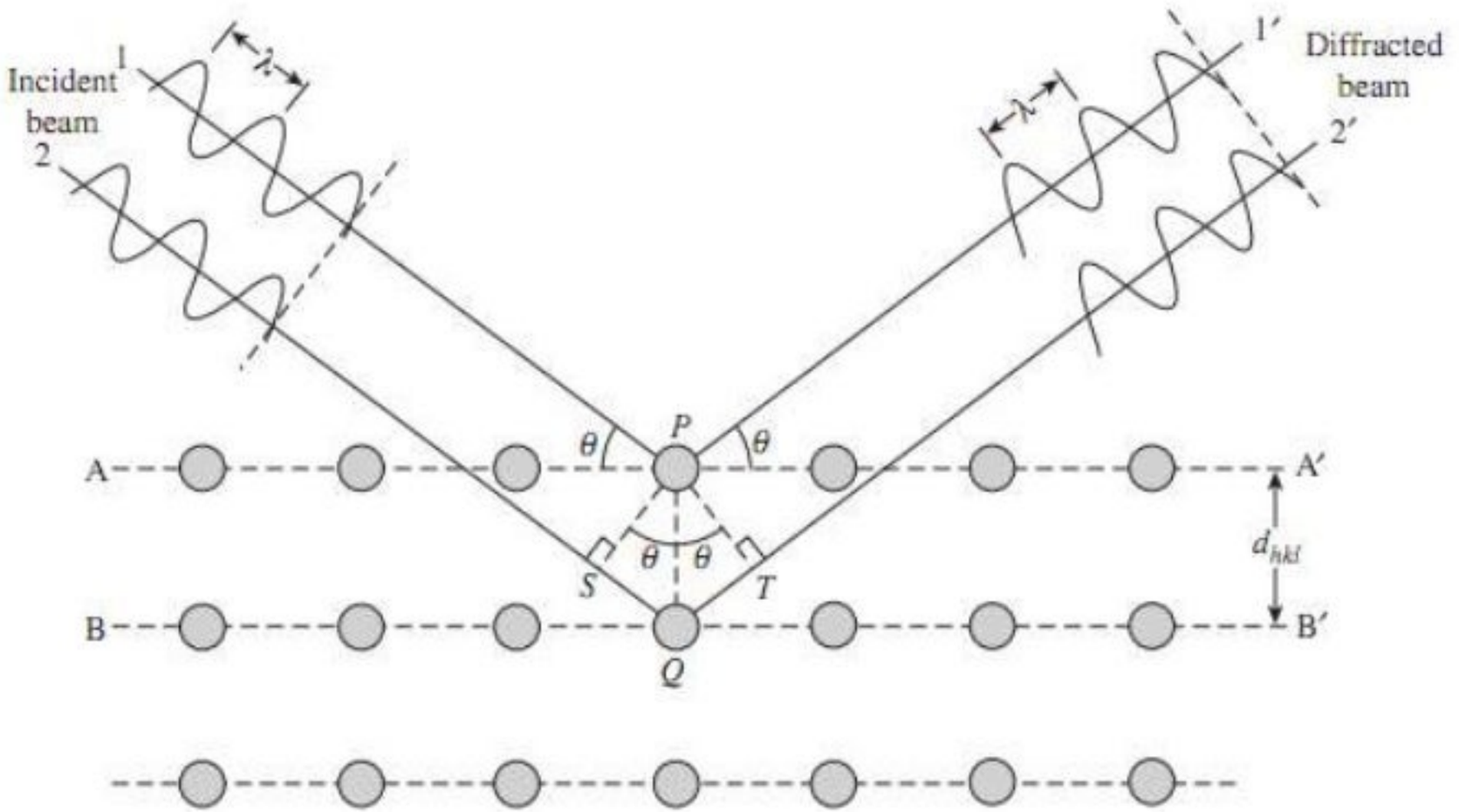
Refraction : The change in direction of a wave passing from one medium to another caused by its change in speed.

Interference : The net effect of the combination of two or more wave trains moving on intersecting or coincident paths. The effect is that of the addition of the amplitudes of the individual waves at each point affected by more than one wave.

Reflection : abrupt change in the direction of propagation of a wave that strikes the boundary between different mediums.

A diffraction grating : is an arrangement equivalent to a large number of parallel slits of equal widths and separated from one another by equal opaque spaces. They are two types reflection and transmission gratings.

Diffraction Principle



$$n\lambda = 2d \sin\theta$$

Bragg's Law

BRAGG'S LAW

- **Bragg's Law** is which determines the angle of coherent and incoherent scattering from a crystal lattice. When X-rays are incident on a particular atom, they make an electronic cloud move just like an electromagnetic wave.

There are two conditions for constructive interference of waves:

1. The angle of incidence must equal the angle of reflection
2. The difference in path length must be an intergral number of wavelengths.

PATTERN OF DIFFRACTION

- **Fresnel's Diffraction** Forms cylindrical wave front with sources of screen at finite distance.
- **Fraunhofer Diffraction** Forms plane wave fronts with observation distance at infinite distance.

Common method of Diffraction

1. Electron diffraction
2. Neutron diffraction
3. X-ray diffraction

Factors Affecting Intensity of Diffraction

1. Structure factor
2. Polarization factor
3. Lorentz factor
4. Multiplicity factor
5. Temperature factor
6. Absorption factor

Advantages

- Data generation is quick
- Testing is cheap but equipment installation is costlier

Disadvantages

- Sample preparation is complex
- My have chance of absorption of radiation
- Source is costlier
- Most of diffraction method need vacuum

Application

- Diffraction methods offer a unique way to measure micro stresses in crystalline materials, because each phase will have its own diffraction pattern giving information on the stresses in that phase.
- It is also the measurement of changes in crystal plane spacing in different directions with respect to the specimen surface.

Spectroscopy Techniques

1. SPECTRUM: Spectrum is a plot of the response as a function of wavelength or more commonly frequency.

2. SPECTROSCOPY: Spectroscopy deals with the production, measurement and interpretation of spectra arising from the interaction of electromagnetic radiation with matter.

Spectroscopic methods are very informative and widely used for both quantitative and qualitative analysis.

Spectroscopy Techniques

3. SPECTROMETRY: It is the measurement of these spectrum responses and an instrument which performs such measurements is a spectrometer or spectrograph.

4. SPECTROPHOTOMETRY: Spectrophotometry is a quantitative approach of measuring the relative energy i.e. emitted, transmitted or reflected in the visible or UV regions as a function of wave length or wave number.

5. ELECTROMAGNETIC SPECTRUM: Electromagnetic radiation is a form of energy that is transmitted through space at enormous velocities. Electromagnetic radiation, or light, is described by the properties of both waves and particles nature.

- In dealing with phenomena such as reflection, refraction, interference, and diffraction, but electromagnetic radiation is conveniently modeled as waves.
- An electromagnetic wave is characterized by several fundamental properties, including its frequency, velocity, amplitude, phase angle, polarization, and direction of propagation.

- The entire electromagnetic spectrum, from the lowest to the highest frequency (longest to shortest wavelength), includes all radio waves (e.g., commercial radio and TV, microwaves, radar), IR, visible light, UV radiation, X-rays and gamma rays.
- Nearly all frequencies and wavelength of electromagnetic radiation can be used for spectroscopy.

6. PRINCIPLE OF SPECTROSCOPY:

- The beam of electromagnetic radiation onto a sample, and observe how it responds to such a stimulus. The response is usually recorded as a function of radiation wavelength. A plot of the response as a function wavelength is referred to as a spectrum.
- The beer-lambert law states that the quantity of light absorbed by a substance dissolved in a fully transmitting solvent is directly proportional to the concentration of the substance and the path length of the light through the solution

7. COMMON METHOD OF SPECTROSCOPY:

- The method of spectroscopy differ with respect to the species to be analyzed (such as molecular or atomic spectroscopy), the type of radiation-matter interaction to be monitored (such as absorption, emission, or diffraction) and the region of the electromagnetic spectrum used in the analysis.
- Spectroscopic methods based on the absorption or emission of radiation in the UV, visible, IR, and radio (nuclear magnetic resonance, NMR)
- Each of these methods is distinct in that it monitors and different types of molecular or atomic transitions.

8. COMMON TYPE OF SPECTROSCOPY:

- Ultraviolet-visible spectroscopy (UV-vis)
- Electron spin resonance spectroscopy
- Atomic spectroscopy
- Infrared spectroscopy and Raman spectroscopy
- Mass spectroscopy
- Nuclear spectroscopy (Nuclear magnetic resonance)

Application of Spectroscopic Analysis

- Understanding constitution of matter from atoms to complex molecules
- Studies on diverse materials existing in nature from deep sea studies to space missions
- Investigations of crime samples
- Studies on environmental sample
- Mineraology

Advantages

- Cure monitoring of composites using optical fibers
- Estimate weathered wood exposure times using near infrared spectroscopy
- Measurement of toxic compounds in blood samples
- Non destructive elemental analysis
- Electronic structure research with various spectroscopes
- Quantitative and qualitative analysis

Disadvantages

- The radiation may be easily contaminated
- Cost of spectroscopy equipment is high
- Not suitable for all kind of material
- Need low working temperature at certain condition

Types of Spectroscopy

1. Atomic spectroscopy (or) Flame Spectroscopy

2. UV / Visible Spectroscopy

1. Atomic spectroscopy (or) Flame Spectroscopy

- Atomic spectroscopy is based upon the absorption or emission of electromagnetic radiation by atomic particles.
- Spectroscopic determination of atomic species can only be performed on a gaseous medium in which the individual atoms or elemental ions.
- This method is widely applied to a wide range of metals and nonmetals.
- Energy transitions of outer electrons of atoms after volatilization in a flame.
- Liquid solution samples are aspired into a burner or nebulizer/ burner combination, desolvated, atomized and sometimes excited to a higher energy electronic state.
- Atomic spectroscopy is the study of the electromagnetic radiation absorbed and emitted by atoms.

PRINCIPLE

- The electrons of the atoms in the atomizer can be promoted to higher orbitals for a short amount of time by absorbing a set quantity of energy (i.e., light of a given wavelength).
- This amount of energy is specific to a particular electron transition on a particular element, and in general, each wavelength corresponds to only one element. This gives the technique its elemental selectivity.

Spectroscopy - TYPES

- **Absorption** – Light of a wavelength characteristics of the element of interest radiates through the atom vapor. The atoms absorb some of the light. The amount absorbed is measured.
- **Emission** – Sample is heated to excitation/ ionization of the sample atoms. Excited and ionized atoms decay to a lower energy state through emission. Intensity of the light emitted is measured.
- **Fluorescence** – A short wavelength is absorbed by the sample atoms, a longer wavelength (lower energy) radiation characteristic of the element is emitted and measured.

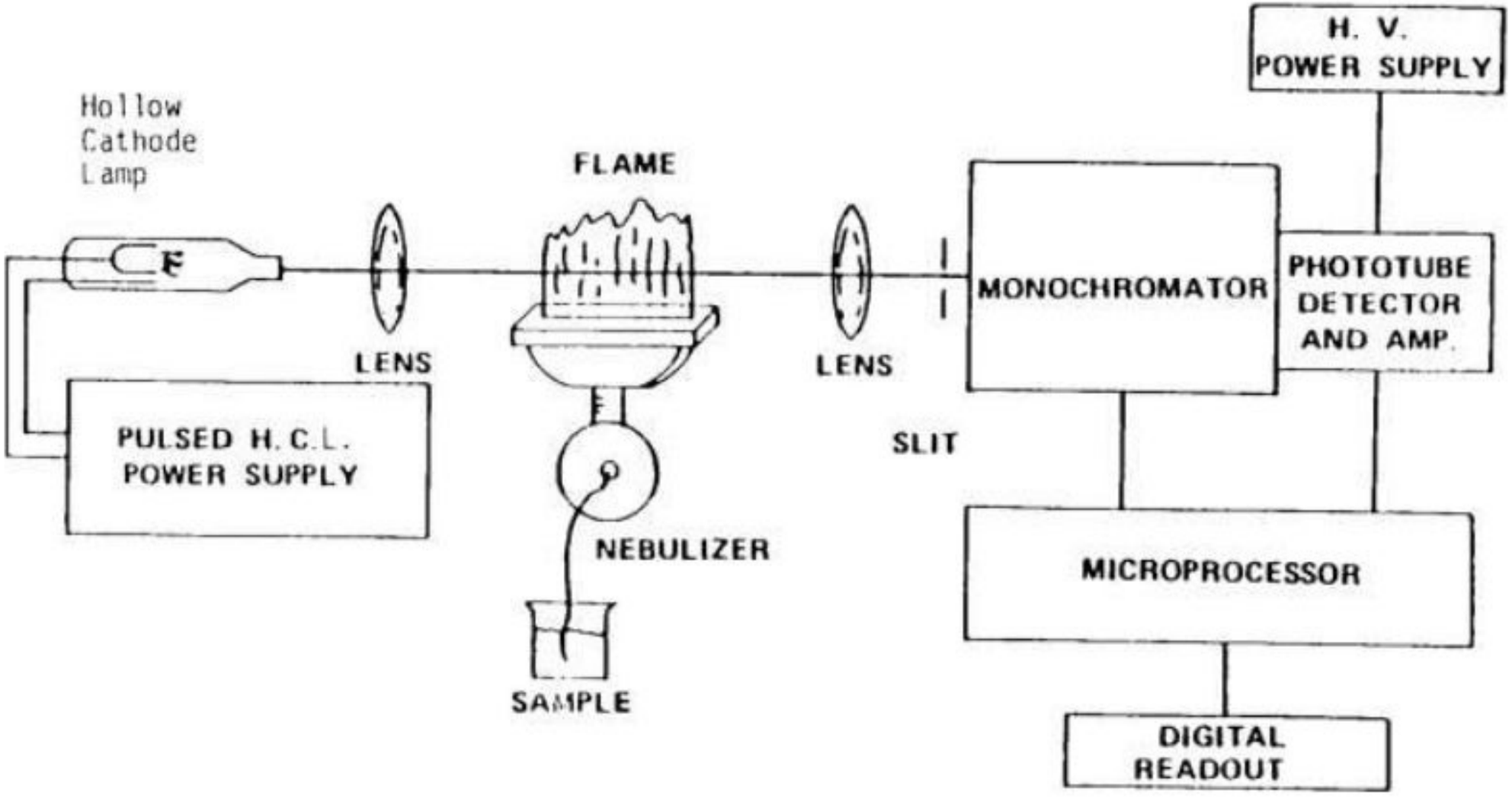
Components

- Lamp source
- Nebulizer
- Atomizer
- Monochromator
- Detector

Construction & Working

- The first step in all atomic spectroscopic procedures is atomization, a process in which a sample is volatilized and decomposed to produce gas phase atoms and ions.
- Atomization is a critical step in all atomic spectroscopy. Several methods are used to atomize samples for atomic spectroscopic studies.

Eg. Inductively coupled plasmas, flames, and electro thermal atomizers; Flames and electro thermal atomizers are widely used in atomic absorption spectrometry, while the inductively coupled plasma is employed in optical emission and in atomic mass spectrometry.



Construction & Working

- In the components of an atomic absorption or flame absorption apparatus, the flame can be considered to be a dilute gaseous solution of the atomized sample held in place by the aspirator-burner.
- Radiation from a suitable source is passed through the atomized sample and into the slit of a photometer or spectrophotometer.
- Radiation of specific wavelength is emitted by the hollow cathode lamp onto the gaseous atoms in the atomizer.
- The monochromator focuses the specific wavelength onto the detector.
- The detector finds the amount of light absorbed.
- The concentration of atoms in the sample is directly proportional to the absorbance.

2. UV / VISIBLE SPECTROSCOPY

- UV-V is spectrometry is based upon absorption of electromagnetic radiation in the visible and ultraviolet regions of the spectrum resulting in changes in the electron structure of ions and molecules.
- The wavelength of UV and visible light are substantially shorter than the wavelength of IR radiation.
- The UV-V is spectrum range from 200 to 700 nm. When a molecule or ion absorbs ultraviolet or visible radiation it undergoes a change in its valence electron transition.

UV / VISIBLE SPECTROSCOPY - PRINCIPLE

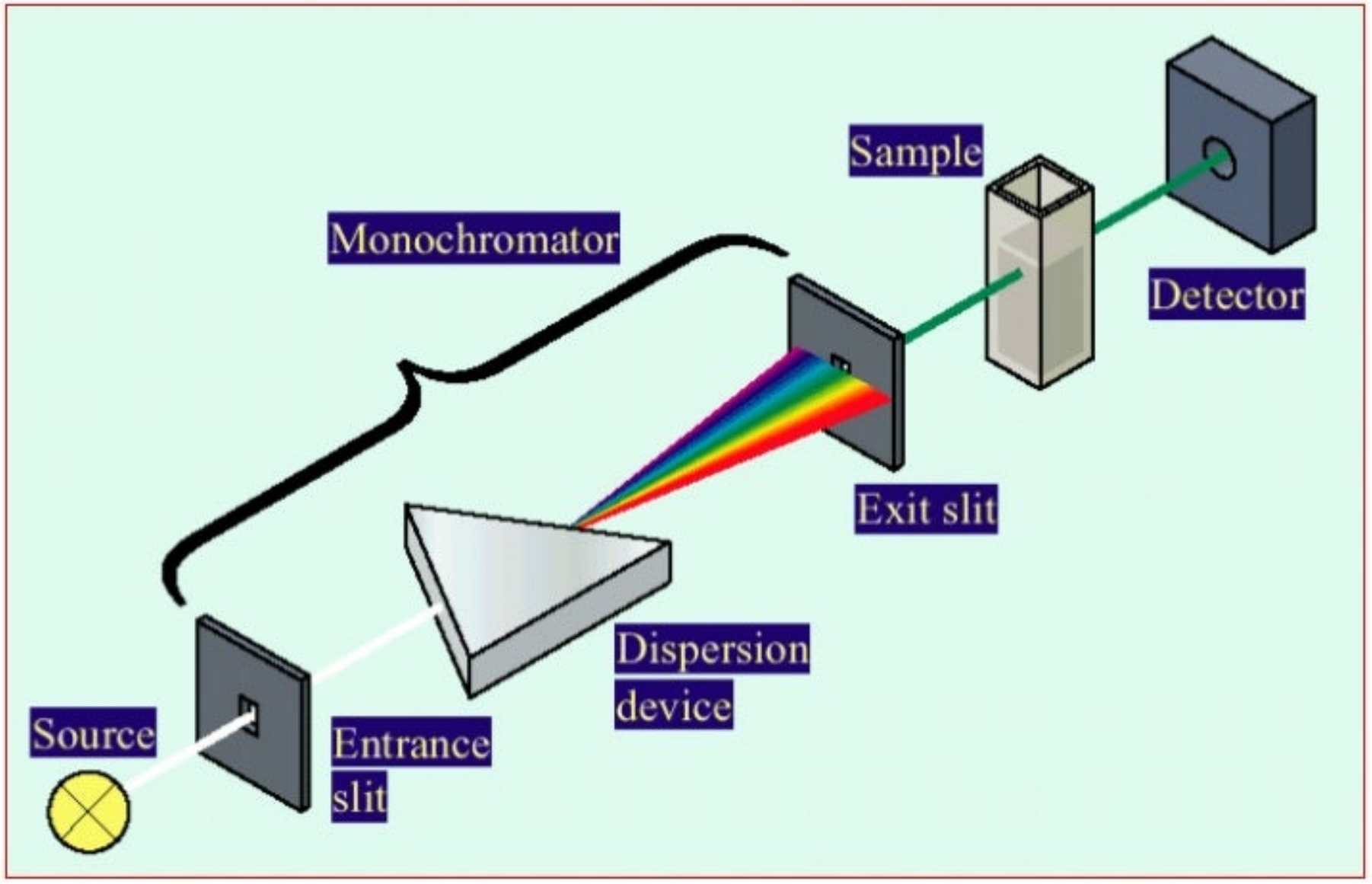
- Diminution of a beam of light after it passes through a sample or after reflection from a sample surface. Absorption measurements can be at a single wavelength or over an extended spectral range.
- Energy transition of bonding and non-bonding outer electrons and molecules, usually delocalized electrons.

Components

- Light source
- Monochromator
- Detector
- Recording devices

WORKING

- Polychromatic light from the source is focused on the entrance slit of a monochromator, which selectively transmit a narrow band of light. This light then passes through the sample area to the detector.
- The absorbance of a sample is determined by measuring the intensity of light reaching the detector without the sample (the blank) and comparing it with the intensity of light reaching the detector after passing through the sample.


Sample
Monochromator
Detector
Exit slit
Dispersion
device
Source
Entrance
slit

Advantages

- Minimum damage to sample
- Better result at lower concentration
- Very rapid calibration
- High sensitivity
- Good accuracy

Disadvantages

- Lack of sensitivity
- Instrument is expensive
- Have limited application to identify the functional group or particular molecule as a result of absorption spectra.

Applications

- Routine qualitative and quantitative measurement
- Used to find relative purity of a solution
- Widely applicability to both organic and inorganic compounds.

ELECTRICAL & MAGNETIC TECHNIQUES

Electrical Techniques

- Electrical properties are a key physical property of conducting materials. It is often necessary to accurately measure the resistivity of materials.

COMMON METHODS

- Dielectric strength
- Electrochemical Impedance Spectroscopy (EIS)
- Arc resistance
- EMF shielding test
- Two-probe method and four-probe method

Electrochemical Impedance spectroscopy

- **Electrochemical impedance spectroscopy** (EIS) is a highly sensitive characterization technique used to establish the electrical response of chemical systems in a nondestructive manner.
- It is an electrochemical technique to measure the impedance of a system in dependence of the AC potentials frequency.

PRINCIPLE

- An electrochemical cell is used to house the chemical reaction and is electrically connected to the electrochemical spectrometer to obtain the electrical response of an electrolytic solution.
- **Electrochemical impedance spectroscopy** (EIS) systems are operated using computer programs specifically designed for EIS testing.
- Therefore, prior to conducting an EIS experiment it is essential that all components of the system be attained.

Components

- Three electrodes

  working electrode,

  counter electrode,

  reference electrode.

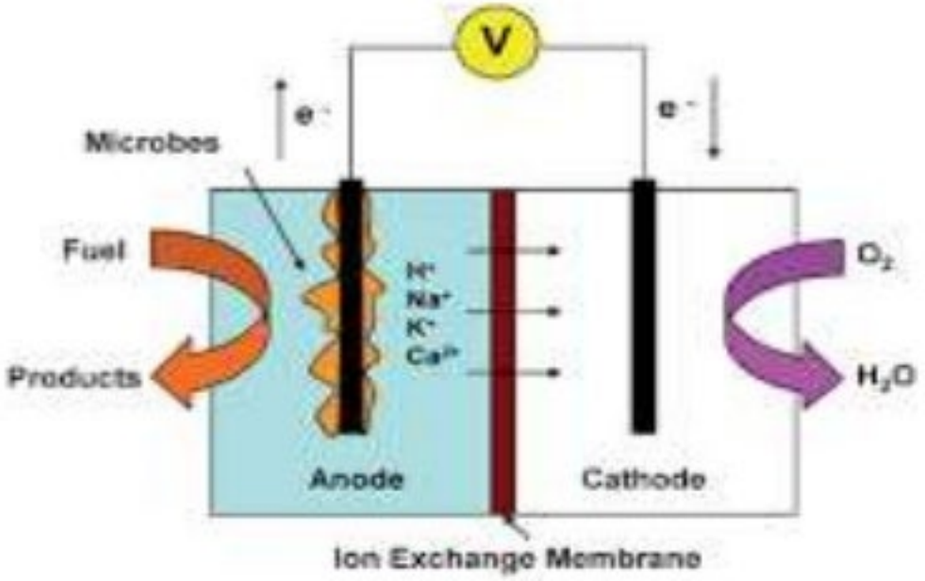
- Electrolytic solution
- Insulating material
- Display unit

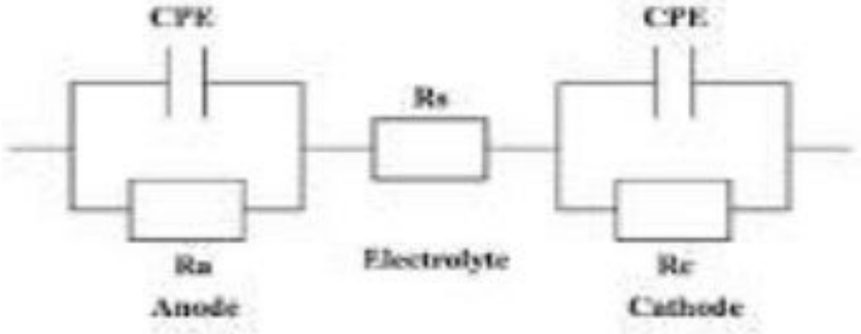
Construction & Working

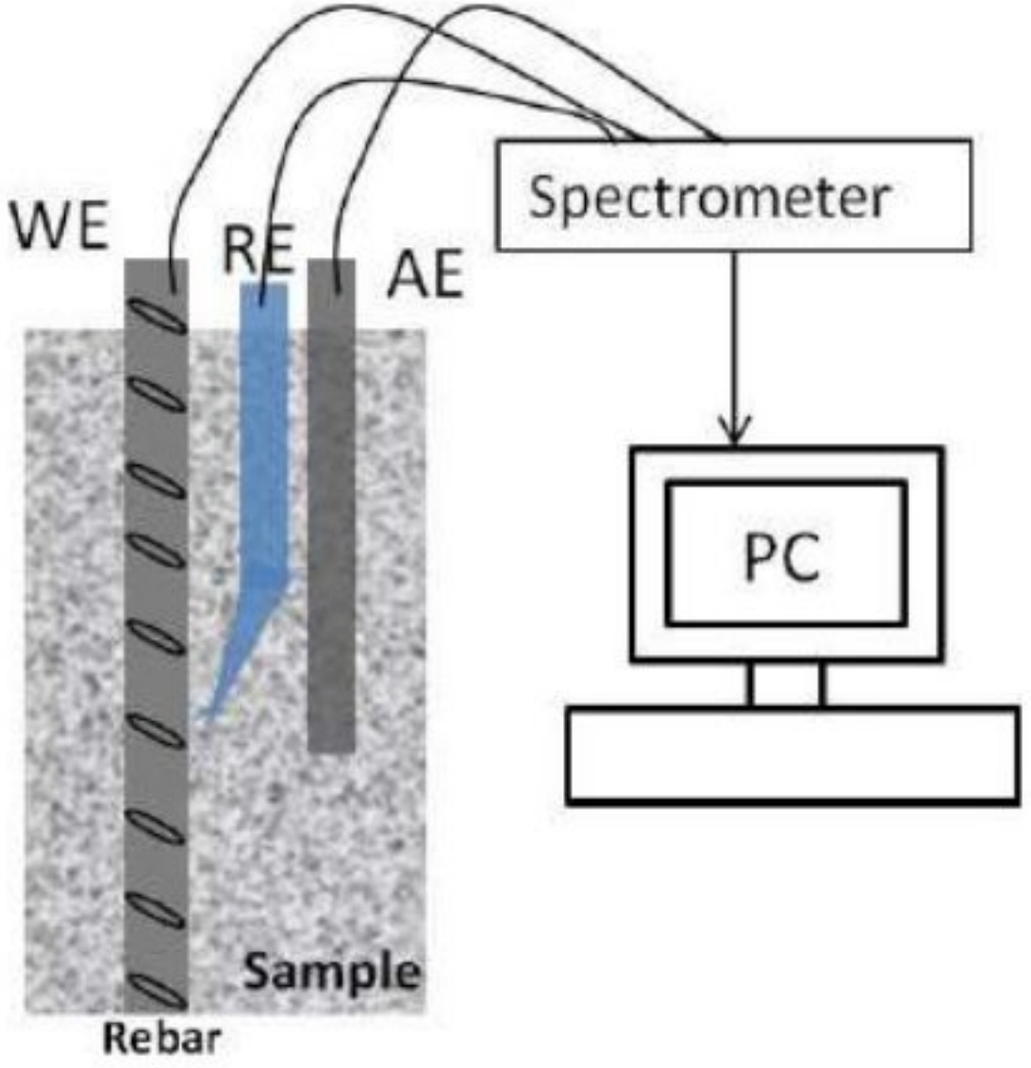
- EIS studies utilize a three electrode mode which is comprised of a working electrode (the sample material), a counter electrode (commonly graphite or platinum) and a reference electrode.
- While electrode geometries may vary the general experimental setup remains similar to the procedure outlined below.
- The three electrodes are mounted on an electrode stage and secured. The electrolytic solution is prepared and transferred to the sample container.

Construction & Working

- A metallic sample container would provide additional pathways for electrons during experimentation leading to a reduction in the EIS current response as electrons move into the metal rather than the reference electrodes.
- Therefore, the sample container should be composed of an insulating material, such as glass or plastic, which will not interface with the transfer of electrons during testing.
- The electrode mount is then placed on the sample container such that a portion of each electrode is submerged in the electrolytic solution.


V
e⁻
e⁻
Microbes
Fuel
Products
H⁺
Na⁺
K⁺
Ca²⁺
O₂
H₂O
Anode
Cathode
Ion Exchange Membrane


CPE
CPE
Rs
Ra
Anode
Electrolyte
Rc
Cathode


WE
RE
AE
Spectrometer
PC
Sample
Rebar

Construction & Working

- Four leads are used to attach the three electrodes to the EIS frequency response analyzer.
- A working lead and a counter lead is used to carry current, whereas the working sense lead and reference leads are used to sense voltage.
- The electrochemical impedance spectroscopy (EIS) working sense lead connects the exposed end of the electrode to the EIS. The reference lead is attached to the reference electrode and the counter lead is connected to the counter electrode.

Construction & Working

- The fourth lead is recommended to ground the system during testing. Once all leads are connected and by stimulus the data is collected from computer generated data.
- The impedance produced during electrochemical experimentation can be valuated through use of one or more equivalent circuits.

Advantages

- Useful on high resistance materials such as paints and coatings
- Time dependent data is available
- Non destructive
- Quantitative data available
- Use service environment

Disadvantages

- Expensive
- Complex data analysis for quantification

Applications

- It provides information about the corrosion kinetics and coatings evaluation.
- It is an accurate and reproducible technique suitable for highly resistive environment.
- It provides data about the electrochemical control mechanism, indicating if corrosion occurs by activation, concentration or diffusion.
- It characterizes the state of the rebar and the morphology of the corrosion.
- It allows for monitoring of the evolution of the passive or active state over time.

Magnetic Techniques

- **Magnetic methods** are potential methods for evaluation of surface manifestation such as microstructural degradation, residual stresses, surface roughness and defect detection in surface coatings of magnetic substrates.

Common Methods

- Magnetic adhesive force method
- Magnetically inductive method
- Magnetic barkhausen emission method

Electromagnetic Techniques

- **Electromagnetic** methods have very high potential for material characterization and well known nondestructive testing.
- Electromagnetic techniques are able to indicate nondestructively and quickly changes of residual stresses, texture, microstructure states, and mechanical properties, and are therefore very useful tools for materials characterization and damage assessment of in-service engineering components.

PRINCIPLE

- Magnetic hysteresis occurs when an external magnetic field is applied to a ferromagnetic such as iron and the atomic dipoles align themselves with it.
- Even when the field is removed, part of the alignment will be retained: the material has become magnetized.

Electromagnetic Methods

- Magnetic Barkhausen noise
- Incremental permeability
    - Non-resonant method (transmission/ reflected method)
    - Resonant method
- Upper harmonics
- Incremental permeability

Advantages

- Nondestructive technique
- Used for Nano material characterization
- Immediate result
- Accuracy in measurement of dielectric losses

Disadvantages

- Limitation of lateral resolution
- Need very thin samples
- Characterization limited to dielectric permittivity
- Multiple steps
- Need technical knowledge
- Presence of air gaps may reduce the accuracy

Applications

- Used for purpose of finding micro structure, texture, hardness depth, phase content, residual stress, aging and grain size.

THANK YOU

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