

unit-1

Bragg's law of diffraction

=> Bragg's x-ray diffraction

-> Bragg's x-ray diffraction was first proposed by William Henry Bragg and William Lawrence Bragg in 1913.

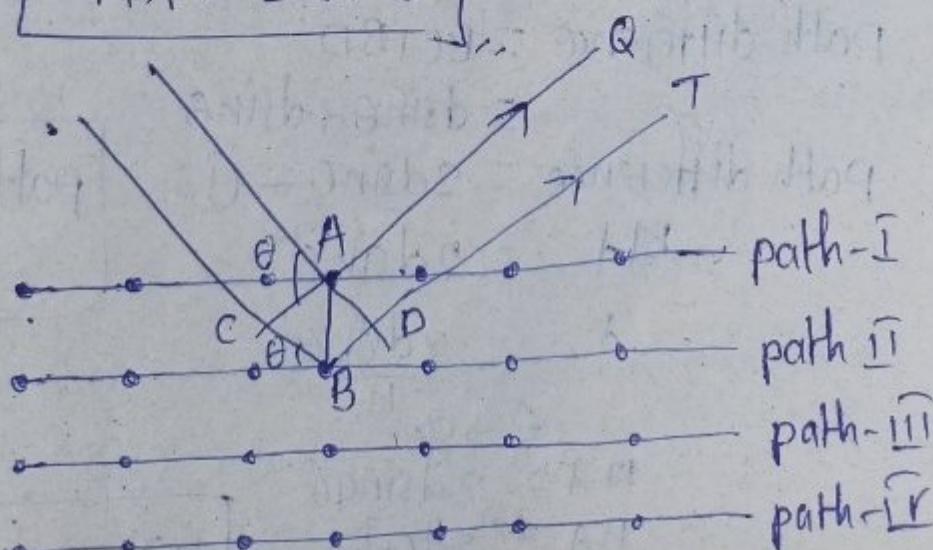
-> Bragg's equation is given by

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

Bragg's Law

Bragg's law is defined as the relation between the wavelength of x-ray beam and incident angle and interplanar spacing is called Bragg's law.

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



planes.

X-ray falls on crystal plane-i at point A

make an angle of incident θ , along the direction of AG.

Another X-ray falls on crystal plane-ii at point B, make an angle of incident θ , along the direction of BT.

The two reflected rays depending upon the difference path pattern in the superimposing medium which can form the diffraction pattern.

Let us draw a normal from A to C and A to D, to calculate path difference BC+BD consider, $\triangle ABC$

$$\sin \theta = \frac{BC}{AB}$$

$$\sin \theta = \frac{BC}{d}$$

$$BC = d \sin \theta$$

$$\text{difference} = BC + BD$$

$$\text{path difference} = d \sin \theta + d \sin \theta$$

$$= 2d \sin \theta - (i)$$

$$- d \sin \theta$$

$$= nA$$

$$\frac{nA}{d}$$

$$= \frac{h\lambda}{d}$$

$$= \sqrt{h\lambda^2} = 2d$$

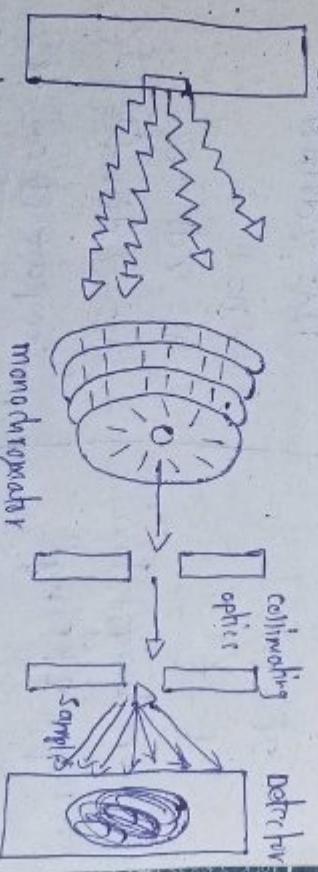
Small-Angle X-ray Scattering (SAXS)

→ Small SAXS is a tool for structural characterization of material by using the X-rays, Neutrons, and laser light.

→ SAXS- small angle X-ray scattering.

→ It is applicable to structural characterization techniques for solids, liquids and gases etc.

→ Block diagram.



→ Block diagram consists of source.

Source : It produces the light on to the monochromator.

Monochromator : It defines the wavelength.

Collimating optics : It determines the angular divergence of beam.

Sample : It is a X-ray generator, detector, collect the radiation scattered by sample.

sample.

→ let us consider equidistance of four crystal planes.

→ X-ray falls on crystal plane i_1 at point A make an angle of incident θ , along the direction of AG.

→ Another X-ray falls on crystal plane i_2 at point B, make an angle of incident θ , along the direction of BT.

→ The two reflected rays' depending upon the path difference in the superimposing medium which can form the diffraction pattern.

→ Let us draw a Normal from A to C and A to D. To calculate path difference BC + BD

consider, ΔABC

$$\sin \theta = \frac{BC}{AB}$$

$$\sin \theta = \frac{BD}{AB}$$

$$BC = ds \sin \theta$$

$$BD = ds \sin \theta$$

Path difference = BC + BD.

$= ds \sin \theta + ds \sin \theta$

Path difference = $ds(2 \sin \theta)$

$n_d = 2 ds \sin \theta$

$\theta \rightarrow 90^\circ$

$\lambda = \frac{2 ds \sin \theta}{n_d} = \boxed{n_d = 2d \sin \theta}$

$n_d = 2d \sin 90^\circ$

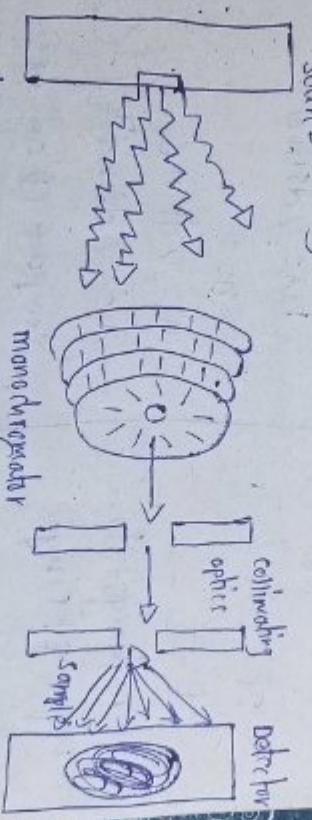
$n_d = 2d$

Small-Angle X-ray Scattering (SAXS)

→ Small SAXS is a tool for structural characterization of material by using the X-rays, Neutrons, and laser light.

→ SAXS - small angle X-ray scattering. It is applicable to structural characterization techniques for solids, liquids and gases etc.

→ Block diagram.



→ Block diagram consists of

Source: It produces the light or to the monochromator; it defines the wavelength.

Monochromator: It determines the angular divergence of beam.

Collimating optics: It determines the angular divergence of beam.

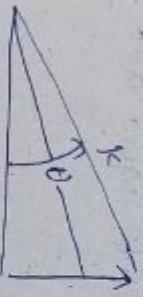
Sample: It is a X-ray generator, detector; collects the radiation scattered by sample.

\Rightarrow S.A.X. related to diffraction

wave vector k :

$$|k| = k = \frac{2\pi}{\lambda}$$

$$k_0$$



Bragg



$$2\theta = \psi$$

$$\theta = \frac{\psi}{2}$$

from Bragg's law

$$nq = 2ks \sin \theta$$

n

$$q = 2k \sin \left(\frac{\psi}{2} \right)$$

$$q = 2 \frac{2\pi}{\lambda} \sin \left(\frac{\psi}{2} \right)$$

$$q = \frac{4\pi}{\lambda} 3 \sin \left(\frac{\psi}{2} \right)$$

$$A = \frac{4\pi}{q} \sin \frac{\psi}{2}$$

$$\boxed{A = \frac{4\pi}{q} \sin \frac{\psi}{2}}$$

Compare ① and ②

$$\frac{4\pi}{q} \sin \frac{\psi}{2} = \frac{k d \sin \frac{\psi}{2}}{l}$$

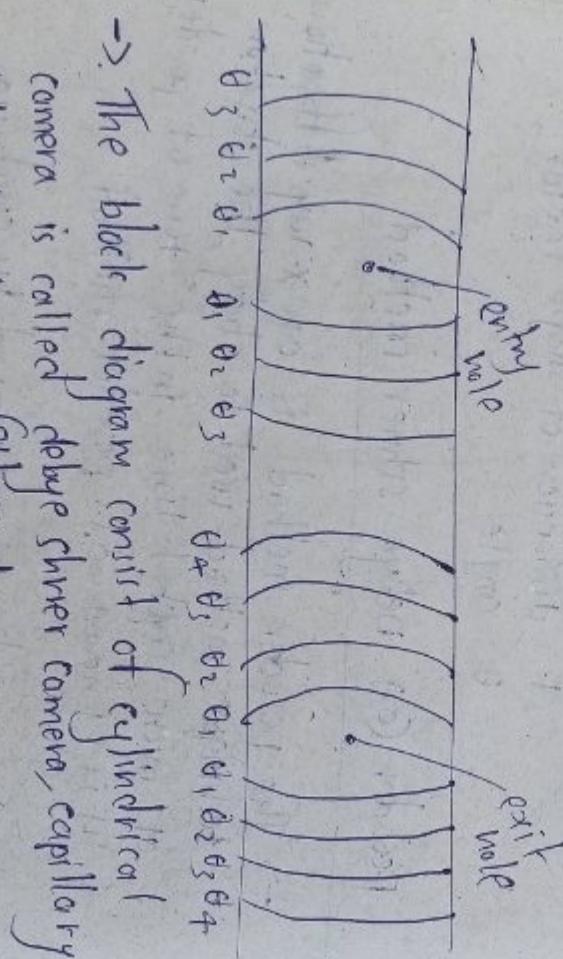
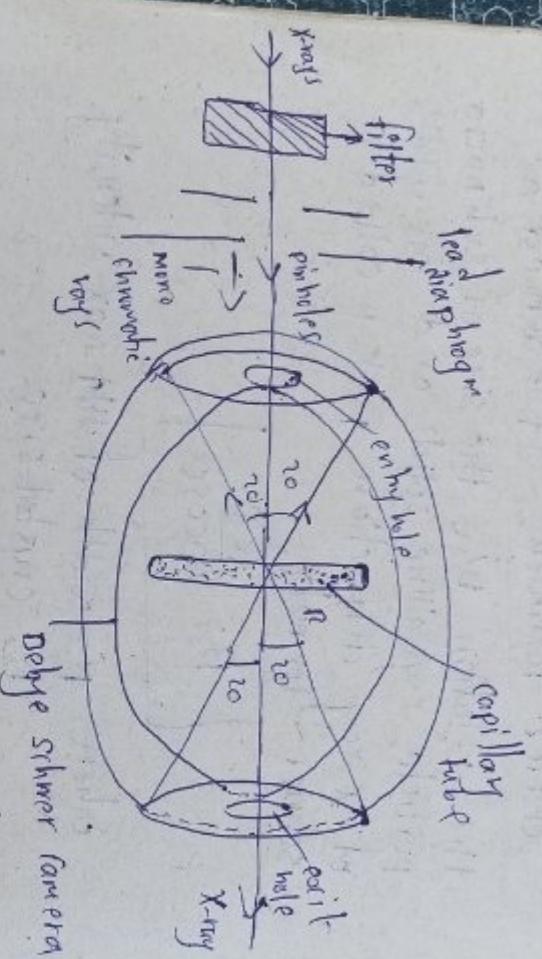
Powder (or) Debye Scherrer method

where $\beta = \text{width of half max intensity}$
 $d = \text{crystal size}$
 $k = \text{dimension of shape factor}$
 $\theta = \text{angle}$

$$\boxed{\beta = \frac{k \lambda}{D \cos \theta}}$$

Scherrer formula: It is defined as the relation b/w the size of sub micro-particle, crystalline solids and line width of X-ray diffraction peak. is called Scherrer formula.

- \rightarrow The powder method is an X-ray diffraction technique method used to study the structure of micro crystal line in the form of powder. It is known as powder method.
- \rightarrow The powder is prepared by crushing of poly crystalline material.
- \rightarrow The crystallization materials are randomly oriented.
- \rightarrow The block diagram of powder method.



- > Monochromatic X-ray beam, passing through the entry hole and falls on capillary tube, to containing of the powder crystal.
- > The powder consist of randomly oriented crystallites, all possible bond values.
- > The debye scherer camera is placed below entry and exit holes.
- > The rays passing through capillary tube and comes out from exit hole, as X-rays.
- > The angle θ corresponding to a particular pair of arc is related to distance s .

$$40 \text{ (radians)} = \frac{s}{R}$$

$$40 \text{ (degree)} = \frac{s}{R} \left(\frac{180}{\pi} \right)$$

$$= \frac{57.295}{R}$$

Factors affecting diffraction intensity

- > factors that affect the diffracted Intensity
- > Structure factor. -> multiplicity factor.
- > polarization factor. -> temperature factor.
- > Lorentz factor. -> absorption factor.

\rightarrow Structure factor: It is defined as the amplitude scattered per unit structure is called structure factor.

$$f_{hkl} = \sum_{n=1}^N f_{n\bar{n}} e^{2\pi i (h\bar{n}_x + k\bar{n}_y + l\bar{n}_z)}$$

f_{hkl} = Amplitude scattered by all atoms in a unit cell

Amplitude scattered by single electron.

\rightarrow Structure factor can be a complex number.

\rightarrow It is useful for mathematical manipulation of complex numbers.

$$\rightarrow I \propto F_{hkl} F_{hkl}^* = |F_{hkl}|^2$$

$$e^{ix} = \cos x + i \sin x$$

$$e^{2\pi i} = e^{3\pi i} = e^{5\pi i} = \dots = +1$$

$$e^0 = e^{2\pi i} = e^{4\pi i} = \dots = +1$$

$$e^{n\pi i} = (-1)^n$$

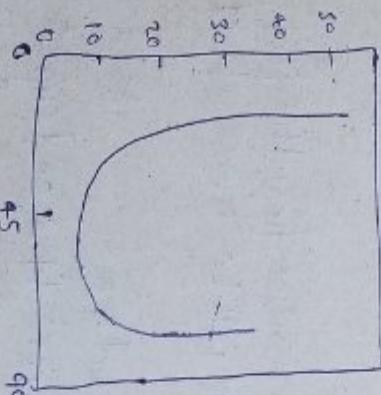
$$e^{n\pi i} = e^{-n\pi i}$$

$$e^{ix} + e^{-ix} = 2 \cos x$$

\rightarrow The Lorentz-polarization factor

The combination of geometric corrections are lumped together into a single Lorentz-polarization factor.

$$\frac{1 + \cos^2 \theta}{2} \left[\frac{1}{\sin \theta} \right] (\cos \theta) \left[\frac{1}{\sin \theta} \right] - \frac{1 + \cos^2 \theta}{2} \sin^2 \theta \tan \theta$$



\rightarrow Lorentz factor

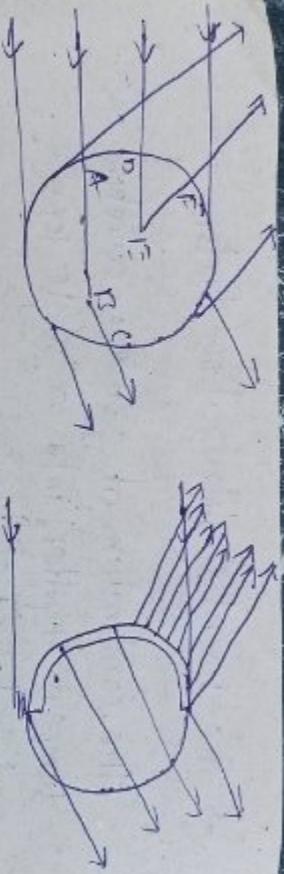
\rightarrow The diffraction beam collected by the detector depends upon diffraction angle.

\rightarrow Absorption factor

\rightarrow Absorption corrections are complicated within the Debye-Scherrer geometry.

→ Temperature factor

→ As atoms vibrate about their equilibrium positions in a crystal, the electron density spread over a larger volume.



→ general case

$$\bar{I}_{\infty} = \frac{\bar{I}_0 A}{2\pi l}$$

→ Highly absorptive specimen

→ Multiplicity factor

→ The multiplicity factor arises from the fact that in general, there will be several sets of hkl -planes.

→ The sign $\pm h$, $\pm k$ and $\pm l$.

→ The value depends upon hkl and crystal symmetry.

→ $100, 100, 010, \bar{0}10, 001, \bar{0}01 P_{100} = 6$

$110, 110, 110, \bar{1}\bar{1}0, 101, \bar{1}01, 10\bar{1}, 011$

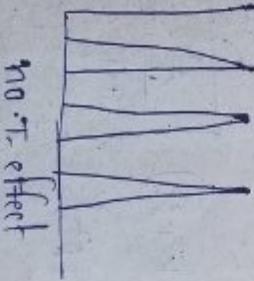
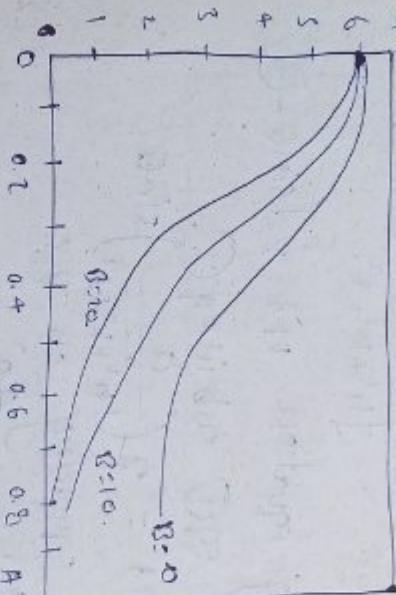
$011, 011, 011$

$$P_{110} = 8/12$$

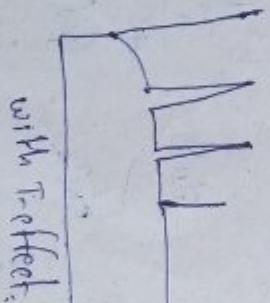
$111, 111, 111, \bar{1}\bar{1}\bar{1}, 101, \bar{1}1\bar{1}, 11\bar{1}$

$$P_{111} = 8$$

$$\text{Temperature factor} = \exp \left[-B \frac{\sin^2 \theta}{\lambda^2} \right]$$



no T. effect



with T. effect

Crystal Structure Determination:

→ For cubic material interplanar material spacing is given by

$$d = \frac{a}{\sqrt{h^2+k^2+l^2}} \quad \textcircled{1}$$

→ Bragg's equation $n\lambda = 2d \sin\theta \rightarrow \textcircled{2}$

$$\textcircled{1} \text{ sub in eq } \textcircled{2} \\ n\lambda = 2 \left(\frac{a}{\sqrt{h^2+k^2+l^2}} \right) \sin\theta$$

squaring on B.S

$$n^2 \lambda^2 = \frac{4a^2}{h^2+k^2+l^2} \sin^2\theta$$

for $n=1$

$$\lambda^2 = \frac{4a^2}{h^2+k^2+l^2} \sin^2\theta$$

$$\sin^2\theta = \frac{\lambda^2}{4a^2} (h^2+k^2+l^2) \quad \boxed{\frac{\lambda^2}{4a^2} = k}$$

$$\sin^2\theta = k(h^2+k^2+l^2)$$

∴

Ex: Hexagonal, Tetragonal, etc

→ consider the plane spacing equation

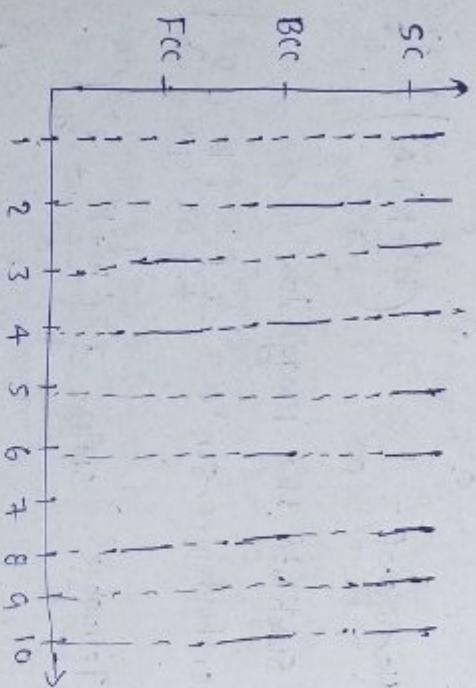
→ allowed reflection of cubic lattices

* simple cubic crystal structure; $h^2+k^2+l^2 =$

$$1, 2, 3, 4, 5, 6, 8, 9, 10, 11, 12, 13, 14, 15, 17, \dots$$

$$* BCC = h^2+k^2+l^2 = 2, 4, 6, 8, 10, 12, 14, 16, 18, \dots$$

$$* FCC = h^2+k^2+l^2 = 3, 4, 8, 11, 12, 16, 19, 20, 24, 27$$



→ For BCC, some lines are missing in diffraction pattern. The Identity lines for BCC is $h^2+k^2+l^2$ is Even.

→ for BCC h, k, l are all even.

→ for FCC h, k, l , value are all odd

$$\rightarrow \sin^2\theta = k(h^2+k^2+l^2)$$

→ many material have crystal structures that are not cubic.

$$\text{Tetragonal} = \frac{1}{a^2} = \frac{h^2+k^2}{a^2} + \frac{l^2}{c^2}$$

$$\text{Hexagonal} = \frac{1}{a^2} = \frac{4}{3} \left(\frac{h^2+k^2+l^2}{a^2} \right) + \frac{l^2}{c^2}$$

Substitute the Bragg's law.

$$\text{orthorhombic } \sin^2\theta = \frac{\lambda^2}{4} \left(\frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2} \right)$$

$$\text{Tetragonal } \sin^2\theta = \frac{\lambda^2}{4} \left(\frac{h^2+k^2}{a^2} + \frac{l^2}{c^2} \right)$$

$$\text{Hexagonal } \sin^2\theta = \frac{\lambda^2}{4} \left(\frac{(4 \cdot h^2 + h(k+k^2))}{a^2} + \frac{l^2}{c^2} \right)$$

Unit - 2

Different types of modes

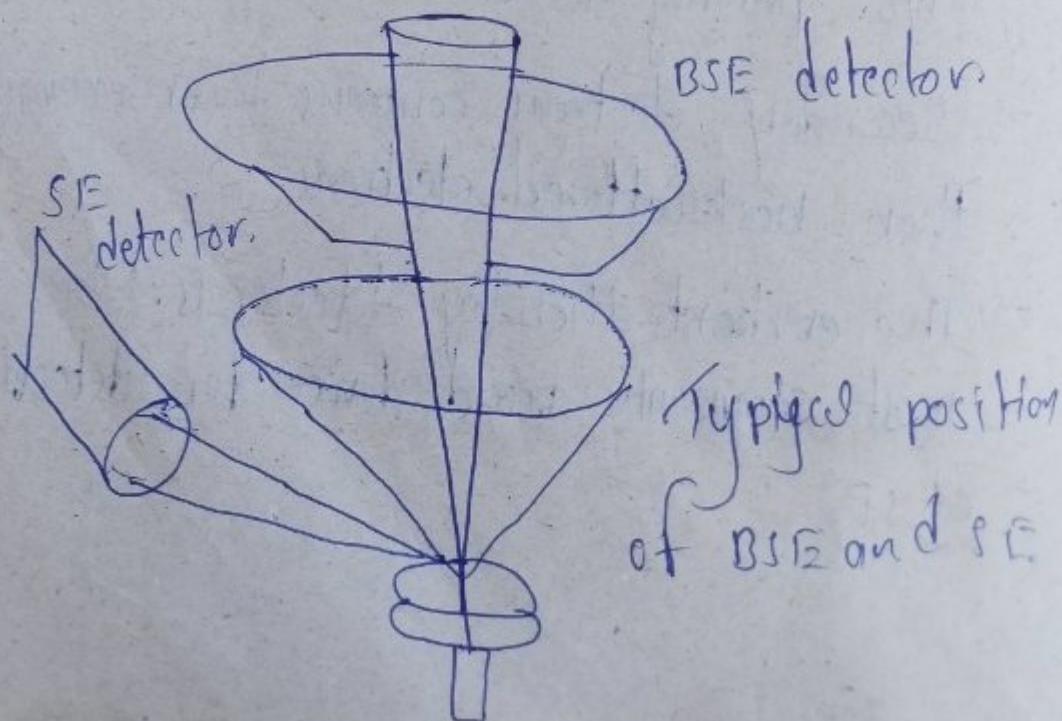
- Electron microscopes are extremely versatile instruments, which can provide various types of information according to user requirement.
- Various types of signals such as.
 - * Secondary electron imaging.
 - * Back scattered electron (BSE)

Secondary electron imaging

- Secondary electrons originate from the surface or near surface regions of the sample.
- They occur due to inelastic interactions b/w primary electron beam and sample.
- Secondary electrons contain lower energy than backscattered electrons.
- The everhart-Thornley detector is the most commonly used device for detection of SE.

Back-scattered electron (BSE) Imaging

- Back-scattered electrons originate from a wide range region within the interaction volume.
- They occur due to elastic collisions of electrons with atoms.
- Imagine electron - atom collision as so called "billiard-ball model".
Here, electrons - tiny particles.
atoms - large particles.
- BSE images can provide beneficial information on topography, crystallography.
- Solid state detectors are most common BSE detectors, which contain p-n junction.



⑤

SEM :-

- The word SEM stands for scanning electron microscope.
- The SEM produces large magnified image by using matter particles instead of light.
- SEM invented by Knoll in the year 1937.

Instrumentation of SEM :-

- The essential components of SEM are :-
 1. Electron gun.
 2. Anode.
 3. Condenser.
 4. Objective lens.
 5. Scan coils.
 6. Sample
 7. Specimen.
 8. Computer system.

1. Electron gun :- A device which produces a narrow stream of electrons from cathode is known as electron gun.

• Thermionic emission gun :- When the sufficient current is subjected to V-shaped metal filament ^{when the filament} heated and electrons are emitted from filament.

• Field emission gun :-

→ Thermionic emission guns are not suitable for image magnification, to overcome this field emission guns are developed.

2. Condenser lens :- It is used to control the intensity and size of electron beam spot.

→ It is used to control the intensity and size of electron beam spot.

objective lens: The objective lens is to focus the electrons onto the sample.

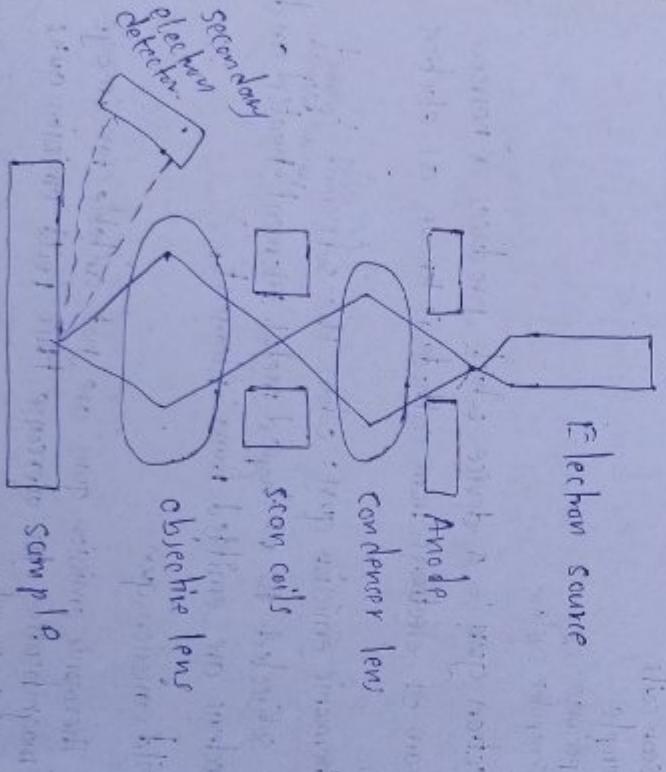
Scan coils: It is used to raster the beam on to the sample.

Sample: The material which requires characterization is sample.

Specimen: Specimen has dimension and made from sample.

Working of SEM

→ The Block diagram of SEM



(3) W-H Method

→ W-H method is defined as to calculate crystallite size and strain from X- RD data using Williamson-Mall plot method in X- RD .

→ Total broadening = Broadening due to crystallite size + Broadening due to strain.

$$\beta_T = \beta_D + \beta_E \quad (1)$$

where β_T = Total broadening

β_D = Broadening due to crystal size.

β_E = Broadening due to strain.

→ From Scherrer equation, we know that, $\beta = \frac{k\lambda}{D \cos \theta}$.

$$\left[\beta_E = \frac{k\lambda}{D \cos \theta} \right] \quad (2)$$

$\lambda = 0.15 \text{ nm}$

$\theta = \text{peak position in radians}$

→ First, The electrons are generated at the top of the column by electron source.

→ The electrons emitted by electron source is accelerated and attracted by anode.

→ The entire electron column is under vacuum.

→ The control lens is used to control the path of the electron beam and projected to objective lens by passing scan coils.

→ The electron beam falls on objective lens focus on sample.

sub (1) and (2) in (1).

$$\beta_T = \frac{k\lambda}{D \cos \theta} + 4\varepsilon \tan \theta$$

$$\beta_T = \frac{k\lambda}{D \cos \theta} + 4\varepsilon \frac{\sin \theta}{\cos \theta}$$

multiply with $\cos \theta$ on B.S

$$\beta_T \cos \theta = \frac{k\lambda}{D} \cos \theta + 4\varepsilon \cdot \frac{\sin \theta}{\cos \theta} \cos \theta$$

$$\beta_T \cos \theta = \frac{k\lambda}{D} + 4\varepsilon \sin \theta \quad \text{---(4)}$$

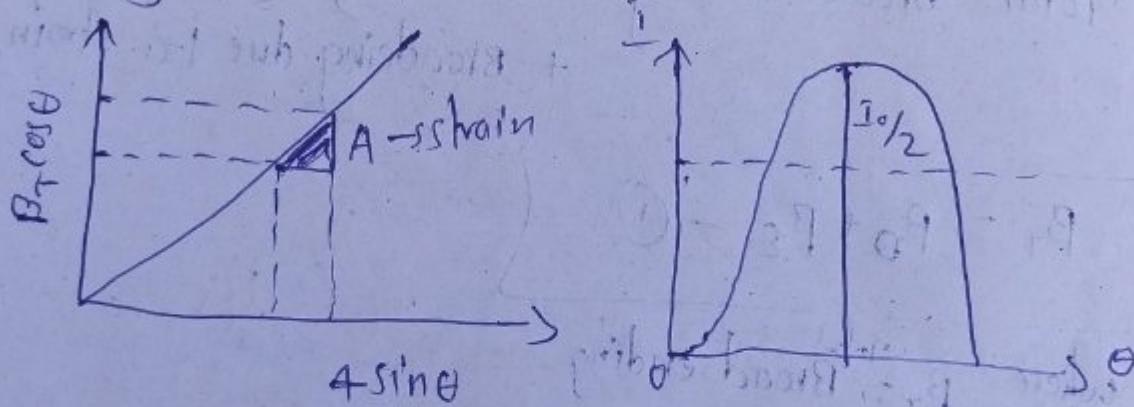
we know that linear equation.

$$y = mx + c \quad \text{---(5)}$$

\rightarrow compare (4) and (5).

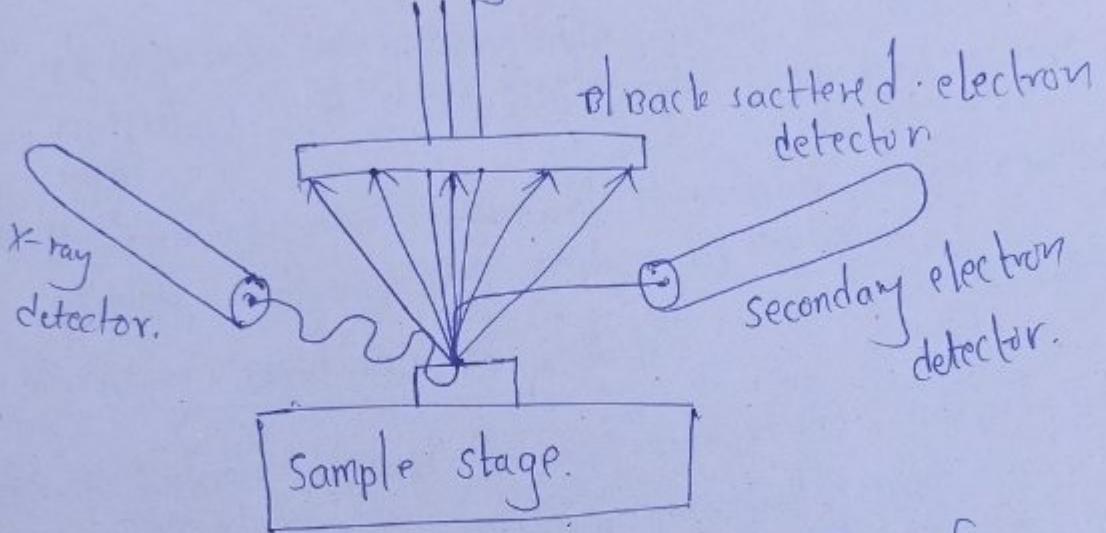
$$y = \beta_T \cos \theta, c = \frac{k\lambda}{D}, m = \varepsilon, x = 4 \sin \theta$$

\rightarrow plotting graphs.



Working of SEM

→ The schematic working diagram of SEM:



→ when the beam touches the surface of the sample, it produces:

- * Secondary electrons (SE)
- * Back scattered electrons (BSE).

* X-Rays.

→ Then the emitted SE is collected by SED, and converted into signal that is sent to a screen which produces final image.

unit-3

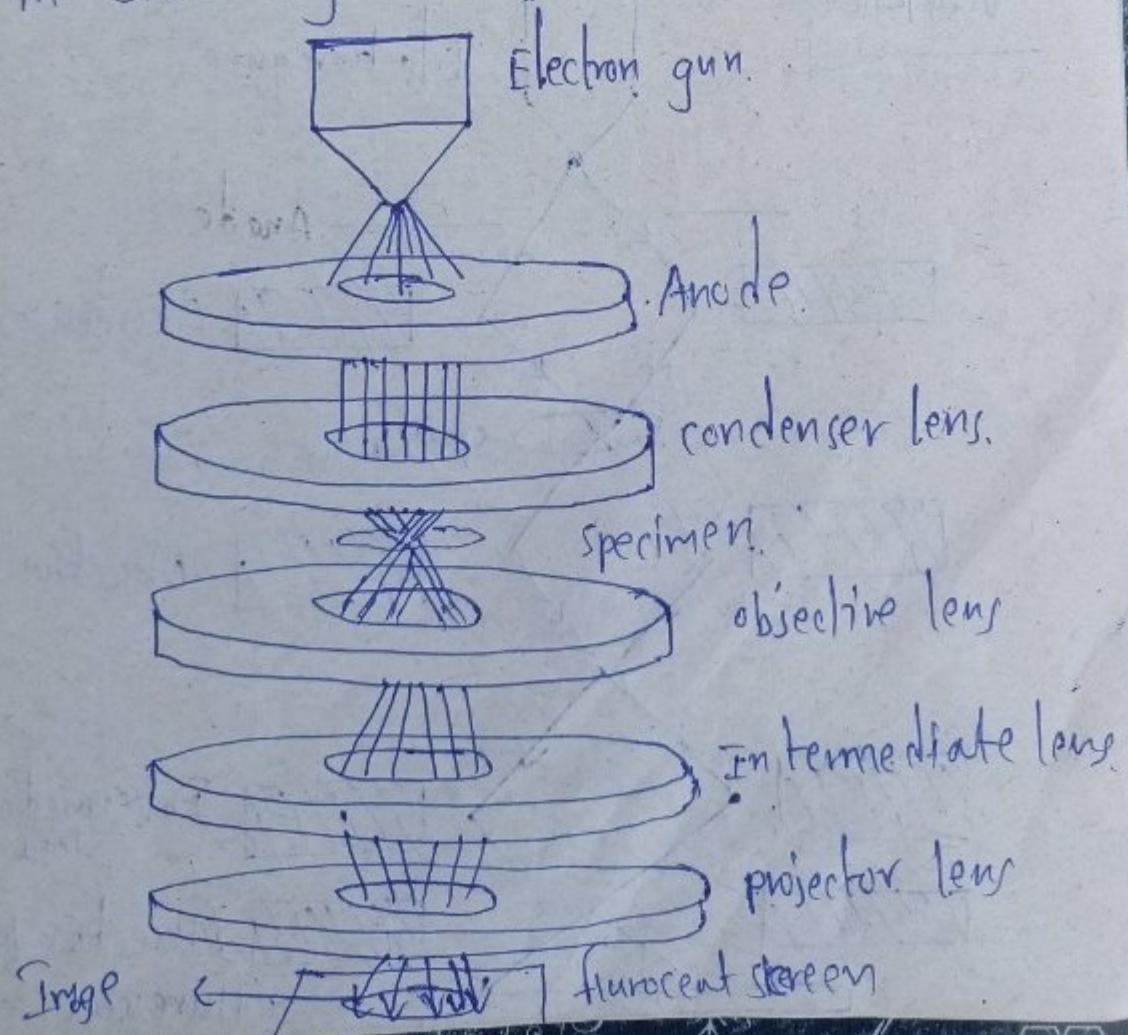
Transmission electron microscope

TEM: Transmission electron microscopy is a microscopy technique in which beam of electrons is transmitted through a specimen to form an image.

→ TEM stands for Transmission electron microscopy.

Construction

→ TEM construction block diagram shown in below figure.



→ Block diagram consist of electron gun, Anode, condenser lens, specimen objective lens, intermediate lens, projector lens, fluorescent screen etc.

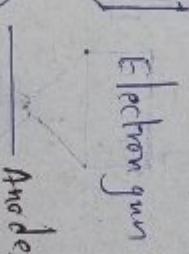
→ It consist of source as electron gun to produce electrons.

→ condenser lens is used to condense the electrons.

→ Specimen is placed below condenser lens and objective lens.

→ Objective lens focuses and magnify the image. The projector lens is placed above the fluorescent screen to achieve higher magnification.

Working



- In order to eliminate diffracted beam the resultant beam passes through objective lens.
- The objective lens and intermediate lenses are part of image producing systems.
- Projector lens provides further magnification.
- Finally image recording system is shown in fluorescent screen.

Advantages

- Three dimensional image obtained gives more information about the specimen.
- very small amount of specimen on sample required for analysis.
- very powerful magnification and resolution.
- TEM have wide-range of applications.
- Images are high quality and detailed.

→ Schematic diagram of TEM shown in above figure.

→ Electron gun produces the stream of electrons and made to fall over anode.

→ Anode which accelerates the electrons to fall on condenser lens.

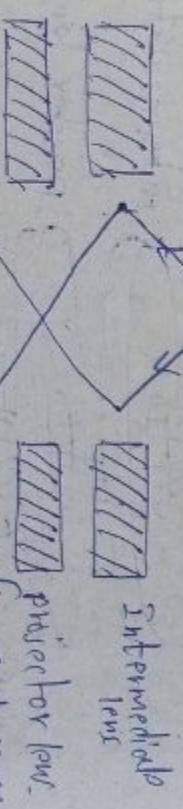
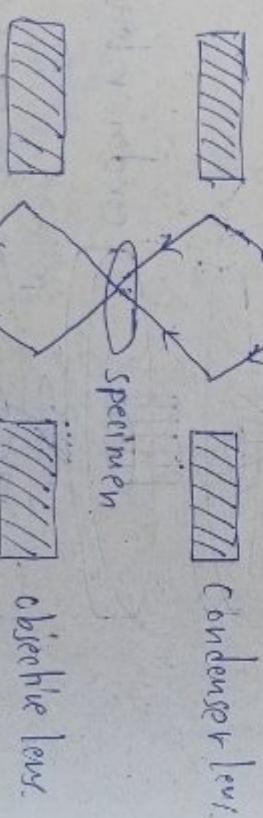
→ Condenser lens which focuses the electrons to fall on specimen or sample.

→ In order to eliminate diffracted beam the resultant beam passes through objective lens.

→ The objective lens and intermediate lenses are part of image producing systems.

→ Projector lens provides further magnification.

→ Finally image recording system is shown in fluorescent screen.



→ Easy to operate.

→ High magnification.

Disadvantages

→ TEMs are large and very expensive.

→ operation and analysis requires special

training.

→ Images are black and white.

→ Requires housing and maintenance.

→ Time consuming.

Applications

→ Nanotechnology

→ medical

→ biological and material research

→ forensic analysis.

→ gemology.

SEM

→ SEM stands for Scanning electron microscopy.

→ SEM is based on scattered electrons.

→ Scattered electrons in SEM are classified into backscattered Secondary scatter electrons.

→ SEM shows the sample bit by bit

→ SEM provides three dimensional image.

→ SEM has 2 million magnification level

→ SEM has 0.4 resolution.

→ It is less expensive.

→ Speed is faster.

→ Image shown on T.V monitor.

TEM

→ TEM stands for Transmission electron microscopy.

→ TEM is based on transmitted electrons.

→ There is no magnification of electrons in TEM.

→ TEM shows the sample as whole.

→ TEM provides two dimensional image.

→ TEM has 50 million magnification level.

→ TEM has 0.5 resolution.

→ It is more expensive.

→ Speed is slower.

→ Image shown on fluorescent screen.

Unit - 4

Fourier Transform Infrared spectroscopy

- FTIR - stands for fourier transform infrared spectroscopy.
- FTIR is analytical technique that is used by material analysis to identify organic compounds.
- Here Infrared radiation is thermal radiation [Heat].
- IR radiation is passed through the sample. Some of the infrared radiation is absorbed by the sample.

Information provided by the FT-IR

- It can identify unknown materials.
- It can determine the quality and consistency of a sample.
- It can determine the amount of components in a mixture.

FTIR Spectroscopy

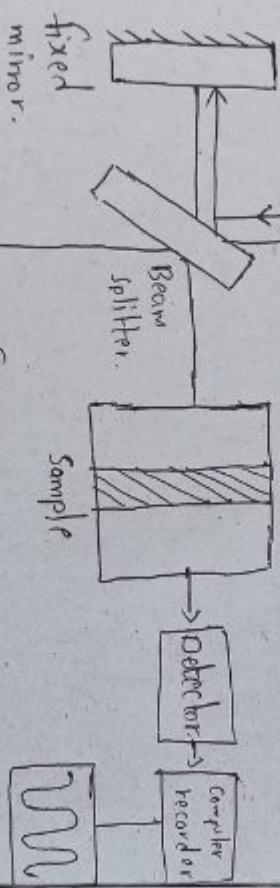
(Introduction in first page)

Construction and working

-> The Block diagram of FTIR spectroscopy.

Movable mirror

fixed mirror

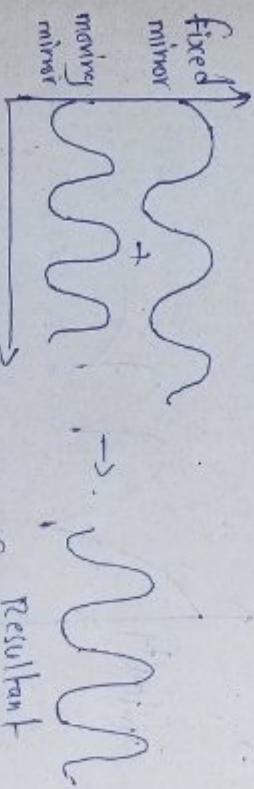


-> From infrared source, global radiation is sent to the beam splitter.

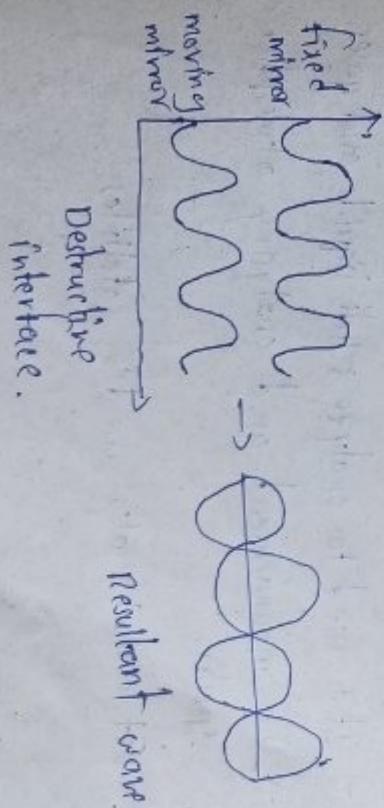
-> Beam splitter splits the half radiation to the movable mirror and another half to fixed mirror.

-> Again radiation back to beam splitter due to mirror reflection.

-> If the fixed mirror and movable mirror are at same distance from beam splitter it called zero path difference. [ZPD]



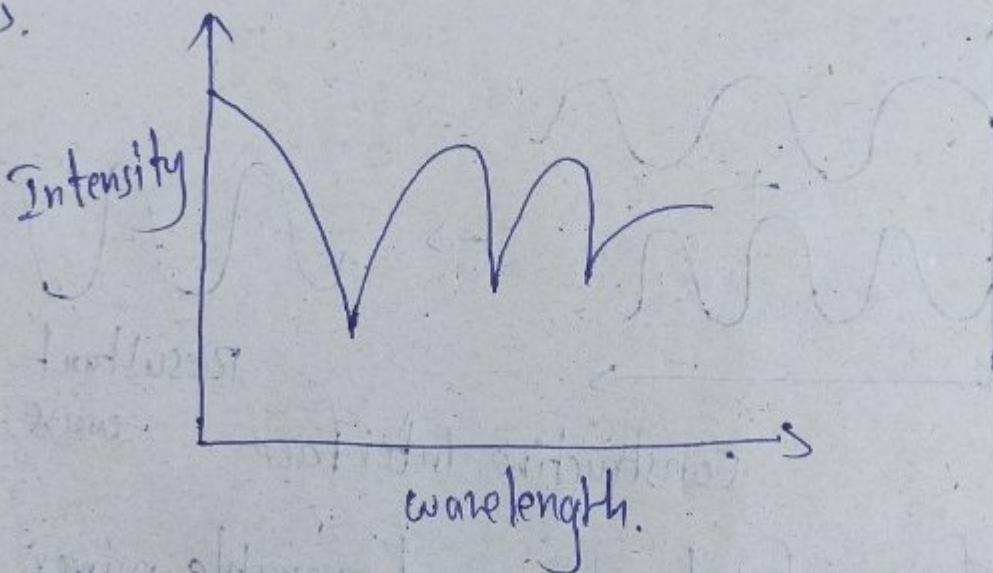
-> If the fixed mirror and movable mirror are at different distance from the beam splitter is called optical path difference. (OPD).



→ Interferometer contains all the radiative energy coming from the source, it has wide range of wavelength.

→ Interferometer contains all the information in one time domain signal.

→



Applications

→ Identification of organic and inorganic compounds.

→ It is used to analyze solids, liquids, and gases.

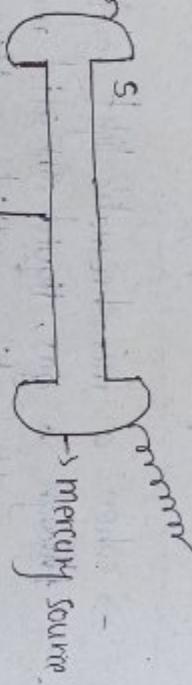
→ In measurement and analysing atmospheric spectra.

→ It can be also used in satellites.

Raman spectroscopy

- > when a monochromatic light ray is passed through a gas, liquid (or) solid material, a small fraction of light scattered in all directions, the scattered light can be seen from the side of the substances.
- > Rayleigh observed that a spectrum of scatter light, there is no change in scattered frequency and incident frequency.
- > This scattered is called Rayleigh scattering
- > In the year of 1928, sir c.v raman observed that change in scattered frequency compares with incident frequency.
- > This scattered spectrum is called Raman spectrum.
- > The lines of greater frequency is called Antistrocle lines and lines of lower frequency is called strocle lines.
- > Block diagram

Diagram



-> Here the source of light should be very strong, otherwise raman lines will be low intensity.

-> Now a days lasers are used for this purpose.

-> The experimental arrangement consist of source, Raman tube, spectrophotov.

-> The mercury light source passes through filter to get monochromatic light i.e beam of single frequency.

-> The monochromatic light passes through open Reflector and falls on Raman tube.

-> The Raman tube consist of a glass tube of above 10 or 2 cm in diameter. 10-15 cm length.

-> one end of the tube has flat glass surface.

-> The other end is draw out into hornshape and black tube.

-> The light tube is surrounded by the water jacket, in which water is circulated to prevent overheating of the sample in the Raman tube.

-> sample is placed in the Raman tube.

principle :-

-> Monochromatic source of light and to photograph scattered light and by means of spectrophotograph, arranged in transverse direction.

Classical Theory of Raman effect

\rightarrow If when atoms or molecules are placed in electric field, the electrons and nucleus are displaced.

\rightarrow Electrons attract towards +ve pole and nucleus attracts towards -ve pole.

\rightarrow More

$$\mu = \alpha_0 E - \beta E \cdot \cos(\omega_0 t)$$

$E = \text{Electric field}$

$$E = E_0 \sin(\omega_0 t - \phi)$$

sub in eq(1)

$$\mu = \alpha_0 E_0 \sin^2(\omega_0 t) - \beta E_0 \sin(\omega_0 t) \cos(\omega_0 t)$$

\rightarrow Effect of vibration of molecule on polarisability

$$\alpha = \alpha_0 + \beta \sin(\omega_0 t) \quad (2)$$

$\alpha = \text{Equilibrium polarisability}$

$$\alpha = \alpha_0 + \beta \sin(2\pi\nu t) \quad (2)$$

sub in eq(2)

$$\alpha = \alpha_0 + \beta \sin(2\pi\nu t) + \frac{A}{2} \sin(4\pi\nu t) \quad (3)$$

$$\alpha = \alpha_0 + \beta \sin(2\pi\nu t) \quad (3)$$

where ν = frequency of vibration molecule.

$$\alpha = \alpha_0 + \beta \sin(2\pi\nu t) \quad (3)$$

$$\mu = \alpha_0 E_0 \sin(\omega_0 t - \phi) \quad (4)$$

sub eq(3) in eq(4).

$$\mu = (\alpha_0 + \beta \sin(2\pi\nu t)) E_0 \sin(\omega_0 t)$$

$$\mu = \alpha_0 E_0 \sin^2(\omega_0 t) + \beta E_0 \sin(\omega_0 t) \sin(2\pi\nu t)$$

$$\mu = \alpha_0 E_0 \sin^2(\omega_0 t) + \frac{1}{2} \beta [\cos(2\pi\nu t) - \cos(4\pi\nu t)] \quad (5)$$

$$\rightarrow \nu_V + \nu = \text{Anti Stokes line}$$

$$\cos(2\pi(\nu_V + \nu)t)$$

$$\rightarrow \nu_V - \nu = \text{Stokes line}$$

$$\nu = \nu_V - \frac{\nu}{2} \sin(4\pi\nu t)$$

$$\nu = 2\nu_V$$

where ν = frequency of rotation

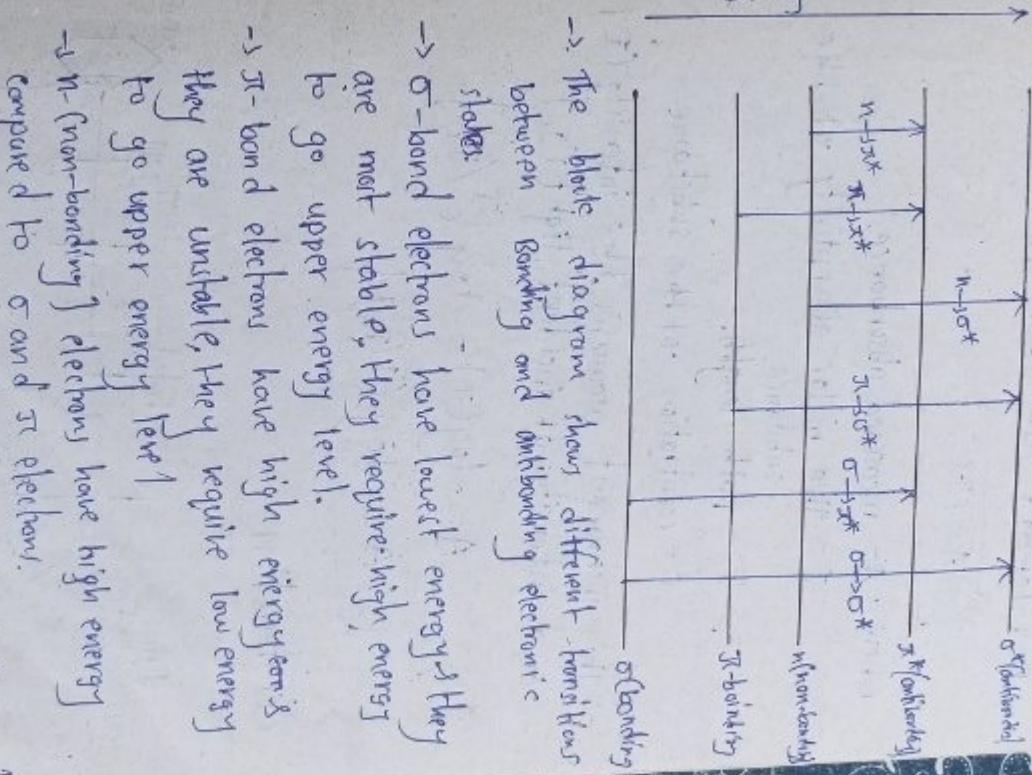
$$\nu = 2\nu_V$$

Advantages

- > simpler mechanical design.
- > universal technique.
- > speed.
- > sensitivity.
- > reliability.

i) U-V visible Spectroscopy

- > ultra-violet visible spectroscopy is an important technique to study optical properties of material.
- > It is also referred to absorption spectroscopy (or) reflectant spectroscopy.
- > In U-V visible spectroscopy the material is exposed to ultra-violet and visible radiation.
- > The radiation of wavelength 200nm to 800nm



Principle:

The basic principle used by UV visible Spectroscopy is transition of π electrons to higher antibonding molecular orbitals by absorbing energy in the form of light.

- > Types of electron orbitals.
 - ii) π -bonding
 - iii) non-bonding or

The UV-visible spectrometer: The instrument used for U-V visible spectroscopy is called UV-visible spectrometer.

- > This instrument is based on Lambert-Beer's law.

Advantages

- > simpler mechanical design.
- > universal technique.
- > speed.
- > sensitivity.
- > reliability.

i. U-V visible Spectroscopy

→ Ultra-violet visible spectroscopy is an important technique to study optical properties of material.

→ It is also refer's to absorption spectroscopy

(or) Reflectant spectroscopy.

→ In U-V visible spectroscopy the material is exposed to ultra-violet and visible radiation.

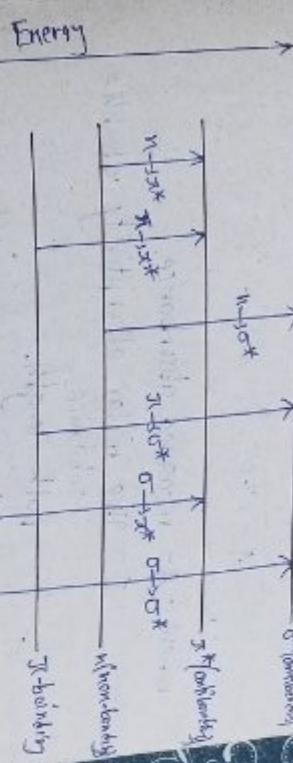
→ The radiation of wavelength 200nm to 800nm

Principle :

→ The basic principle used by UV visible Spectroscopy is transition of π electrons to higher antibonding molecular orbitals by absorbing energy in the form of light.

→ Types of electron orbits.

i σ -bonding ii π -bonding iii non-bonding or lone pair.



→ The block diagram shows different transitions between bonding and antibonding electronic states.

→ σ -bond electrons have lowest energy. They are most stable. They require high energy to go upper energy level.

→ π -bond electrons have high energy. They are unstable. They require low energy to go upper energy level.

→ π -n (non-bonding) electrons have high energy compared to σ and π electrons.

The UV-visible spectrometer : The instrument

used for U-V visible spectroscopy is called UV-visible spectrometer.

→ This instrument is based on Lambert-Beer's law.

→ Beer-Lambert law states that

$$A = \epsilon \tau c$$

where A = measured absorbance.

ϵ = molar absorptivity of the substance

τ = path length.

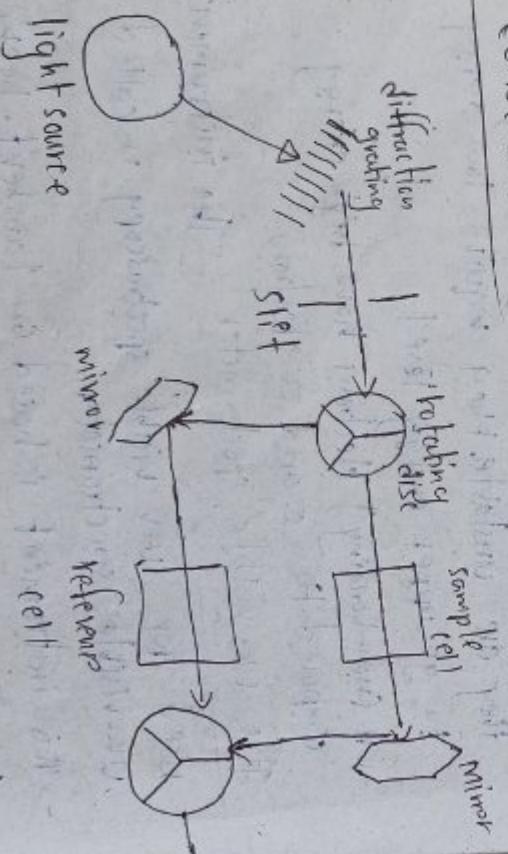
c = concentration of the substance

→ This instrument measures the intensity (τ) and intensity of incident light (I_0).

$$A = -\log(\tau) = -\log\left(\frac{\tau}{I_0}\right)$$

$$A = -\log\left(\frac{I}{I_0}\right)$$

Construction



→ The above block diagram shows schematic diagram of UV-visible spectroscopy.

→ The UV-visible Spectrometer consists four basic components, light source, monochromator, and detector.

→ The UV-visible spectrometer needs light source which is needed one.

→ For this purpose, combination of different lamps are used.

→ Deuterium lamp gives UV part and tungsten

and halogen lamp gives visible part.

→ Recently the light emitting diode (LED) also have been used.

→ The detector is photodiode.

→ The sample holder for UV-visible spectroscopy

is called cell, it contains solvent.

→ Solvents and their absorption wavelengths are given in table.

Solvent	Absorption-wavelength
Water	191
Heptane	201
Methanol	203
Ethanol	204

- Two types of arrangements are used in the U-V visible spectrosocpy.
 - i Single beam.
 - ii Dual beam.
- The black diagram shows dual-beam UV-visible spectrometer. The working of this instrument is very simple.
 - Light source is converted into monochromator by passing through diffraction grating and slit.
 - After slit light source hits rotating disc.
 - Rotating disc consist of three sides.
 - i Black side/minor side in transparent side.
 - ii After rotating disc, monochromator light reaches another rotating disc. by passing sample cells and minor.

→ After rotating disc, light reaches detector and computer after. chart recorder.

Applications

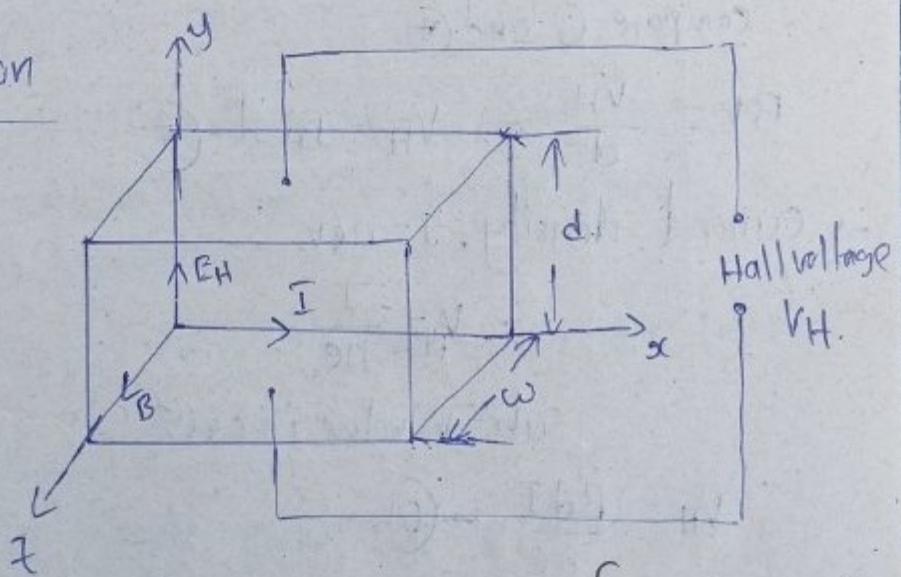
- Measuring the concentration of substances such as protein, DNA, RNA etc.

Unit - 5

Hall Effect

Def: When a current carrying conductor is subjected to a transverse magnetic field, then a potential difference is developed across the semiconductor perpendicular to both current and applied magnetic field:
 → This phenomena is called Hall effect.

Explanation



- Let us consider semiconductor slab of width w and thickness d .
- Current I is flowing along x -direction.
- Hall-voltage is V_H .

Derivation:

→ Force of electric field, $F = eE_H$ - (1)

→ Force of magnetic field, $F = BeV$ - (2)

Compare ① and ②.

$$\cancel{eE_H = B\cancel{eV}}$$

$$\boxed{E_H = BV} - (3)$$

V_H = Hall voltage.

$$E_H = \frac{V_H}{d} - (4)$$

Compare ④ and ④.

$$BV = \frac{V_H}{d} \Rightarrow V_H = BVd - (5)$$

→ Current density, $J = neV$.

$$V_H = \frac{J}{ne}$$

Sub V value in eq ⑤

$$V_H = \frac{BdT}{ne} - (6)$$

$$\boxed{V_H = \frac{B\cancel{dT}}{ne} = \frac{B\Omega}{ne\omega}} - (7)$$

$$R_H = \frac{1}{ne}$$

$$V_H = \frac{BTR_H}{ne\omega} = \boxed{R_H = \frac{V_H\omega}{BT}} - (8)$$

$$\text{conductivity } \sigma = neV, \text{ so } \lambda = \frac{\sigma}{V}, \mu = \frac{\sigma}{R_H}$$

Vibrating sample magnetometer (VSM)

Def → A vibrating sample magnetometer (VSM) is a scientific instrument that measures the magnetic properties, based on Faraday's law of induction.

→ VSM is also referred as Foner magnetometer.

→ Simon Foner invented VSM in 1955 at MIT Lincoln laboratory.

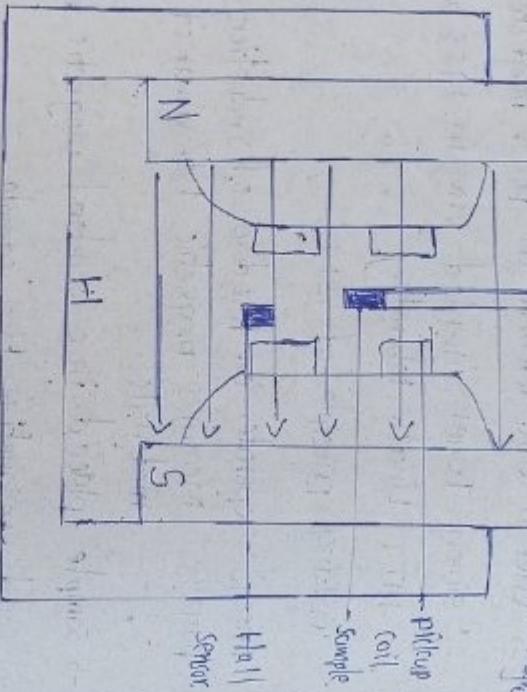
Working principle of VSM:

Principle: operate on Faraday's law of induction
purpose: measures magnetic behaviour of materials

- sample placed in a constant magnetic field.
- Field magnetizes the sample.
- Magnetized sample creates magnetic field.
- pick-up coils sense changing magnetic field.
- Faraday's law induces electric field in coils, changing magnetic field into electric field.
- This makes an electric signal.
- The signal tells us how magnetic material is magnetized.
- It tells us how much sample is magnetized and how it reacts to magnetic field.

Construction

Vibration unit



Advantages

→ The precision and accuracy of VSM's.

Disadvantage

→ VSM's not suited for determining magnetization loop.

Applications

→ For magnetic measurement.

→ Mineral, ores, properties can find.

SQUID

Introduction

→ SQUID stands for superconducting quantum interference device.

→ SQUID act as sensitive magnetometer, used to measure weak magnetic field.

→ SQUIDS are widely used in medical field to estimate low magnetic field produced by various organs of human body

→ The block diagram shows construction of VSM.

→ The block diagram consist of vibration unit, sample holder, pick-up coil, sample, Hall sensor, and an electromagnet.

→ Explain each.

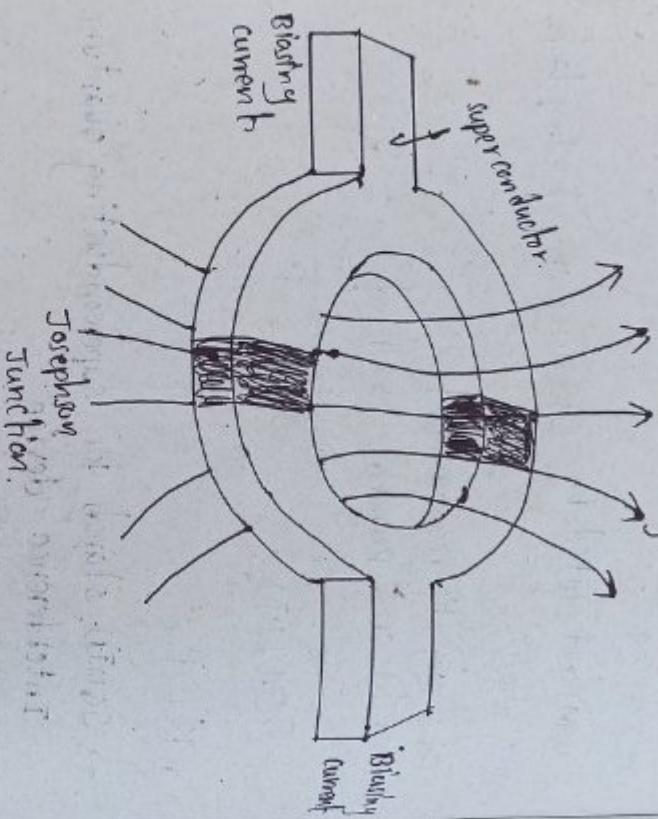
construction

→ A SQUID is constructed by super conducting loop containing one or more Josephson junctions.

→ The essential part of squid are superconducting ring loop.

→ A Josephson junction.

magnetic field.



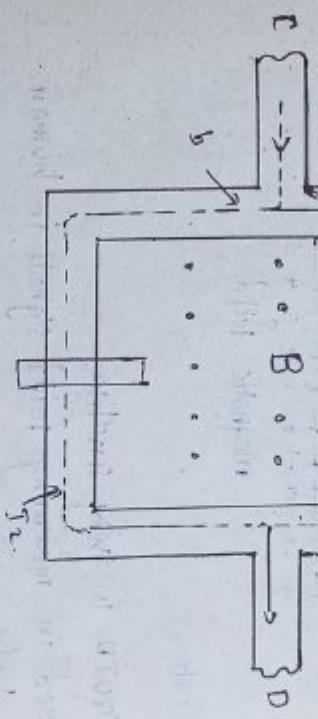
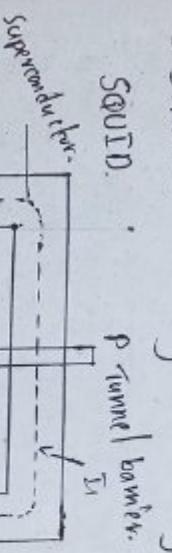
Josephson
Junction

- The above block diagram shows the DC-SQUID with Josephson junction.
- low-temperature SQUID fabricated with low temperature superconducting material such as pure niobium.
- This device is cooled with liquid helium.
- High temperature SQUID fabricated with high temperature superconducting (YBCO).
- This device is cooled with liquid nitrogen.

Working

→ schematic working block diagram of

SQUID



→ when DC current is applied to the SQUID, this current enters into the device and split into two paths a and b as \bar{I}_1 and \bar{I}_2 .

→ wave function $\Psi = \Psi_0 e^{i\phi}$
where Ψ_0 = Amplitude.
 δ = phase value.

→ Total current through Josephson junction

$$\bar{I}_T = (I_0 \sin \delta) \cos \frac{\phi}{\hbar c}$$

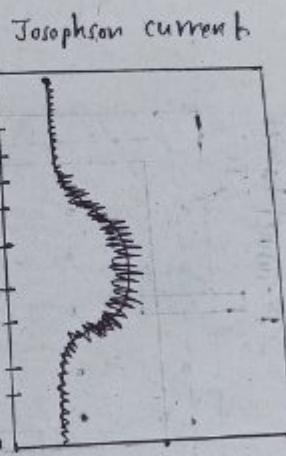
→ graph b/w magnetic field and current.

Electrical properties

- conducting species: The charge carriers which responsible for generation of current are called conducting species

- Electrons

- Holes



Applications

- SQUID is very sensitive.
- used in measuring faint signals in human brain.
- used in "magnetencephalography"

$$I = \frac{dq}{dt}$$

- Electric current: The amount of electricity flowing through material is called electric current

$$V = \frac{dq}{dt}$$

- Electric voltage: The change in potential energy per charge is known as electric voltage.
- electric field: electric field is an invisible force created by attraction and repulsion of electrical charges
- resistance: measure of opposition to current flow in circuit

$$(R = \frac{V}{I})$$

- conductivity: It is the reciprocal of resistivity

-> According to KVL, the algebraic sum of voltages around circuit is zero.

-> Apply KVL for above circuit

$$E_T - IR_{\text{wire}} - IR_{\text{at interface}} - IR_{\text{at sample}} = 0$$

$$IR_{\text{at sample}} = E_T - IR_{\text{wire}} - IR_{\text{at interface}}$$

where,

E_T = Battery tension.

IR_1 = Voltage through wire medium.

IR_2 = Voltage at wire and sample.

IR_3 = Voltage at sample medium.

The electrical properties analysis

AC and DC conductivity

- > The electrical properties such as conductivity, resistivity etc. unknown samples can be determine by applying AC and DC current.
- > The application of alternate current sample gives "AC" conductivity.
- > The application of direct current sample give "DC" conductivity.
- > circuit diagram voltmeter

