Basics and Application Of XRay Diffraction

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CONTENTS

- INTRODUCTION
- GENERATION OF X-RAYS
- PRINCIPLE
- INSTRUMENTATION
- METHODS
- APPLICATIONS
- CONCLUSIONS
- REFERENCES

INTRODUCTION:

Max Von Laue

X-rays were discovered by Wilhelm Roentgen who called them x-rays because the nature at first was unknown so, x-rays are also called Roentgen rays. X-ray diffraction in crystals was discovered by Max von Laue. The wavelength range is 10^{-7} to about 10^{-15} m.

The penetrating power of x-rays depends on energy also, there are two types of x-rays.

i) **Hard x-rays**: which have high frequency and have more energy.

ii) **soft x-rays**: which have less penetrating and have low energy

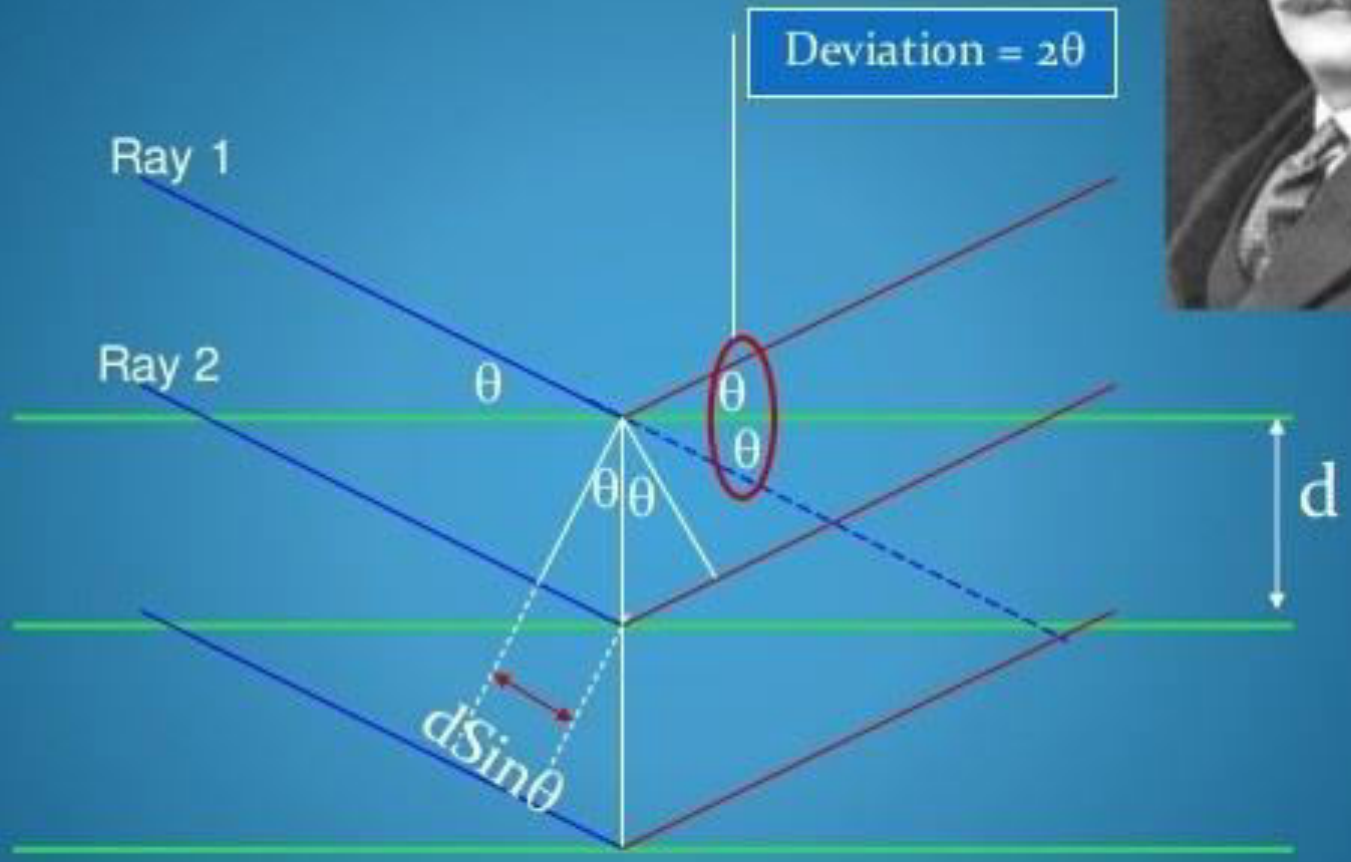
X-RAYS

1. X-rays are short wave length electromagnetic radiations produced by the deceleration of high energy electrons or by electronic transitions of electrons in the inner orbital of atoms
2. X-ray region 0.1to100 A°
3. Analytical purpose 0.7 to 2 A°

PRINCIPLE

X-ray diffraction is based on **constructive interference** of monochromatic x-rays and a crystalline sample. These x-rays are generated by a cathode ray tube, filtered to produce monochromatic radiation ,collimated to concentrate and directed towards the sample. The interaction of incident rays with the sample produces constructive interference when conditions satisfy **Bragg's law.**

BRAGG's EQUATION



- The path difference between ray 1 and ray 2 = 2d Sinθ

Constructive interference of the reflected beams emerging from two different planes will take place if the path lengths of two rays is equal to whole number of wavelengths".

for constructive interference,

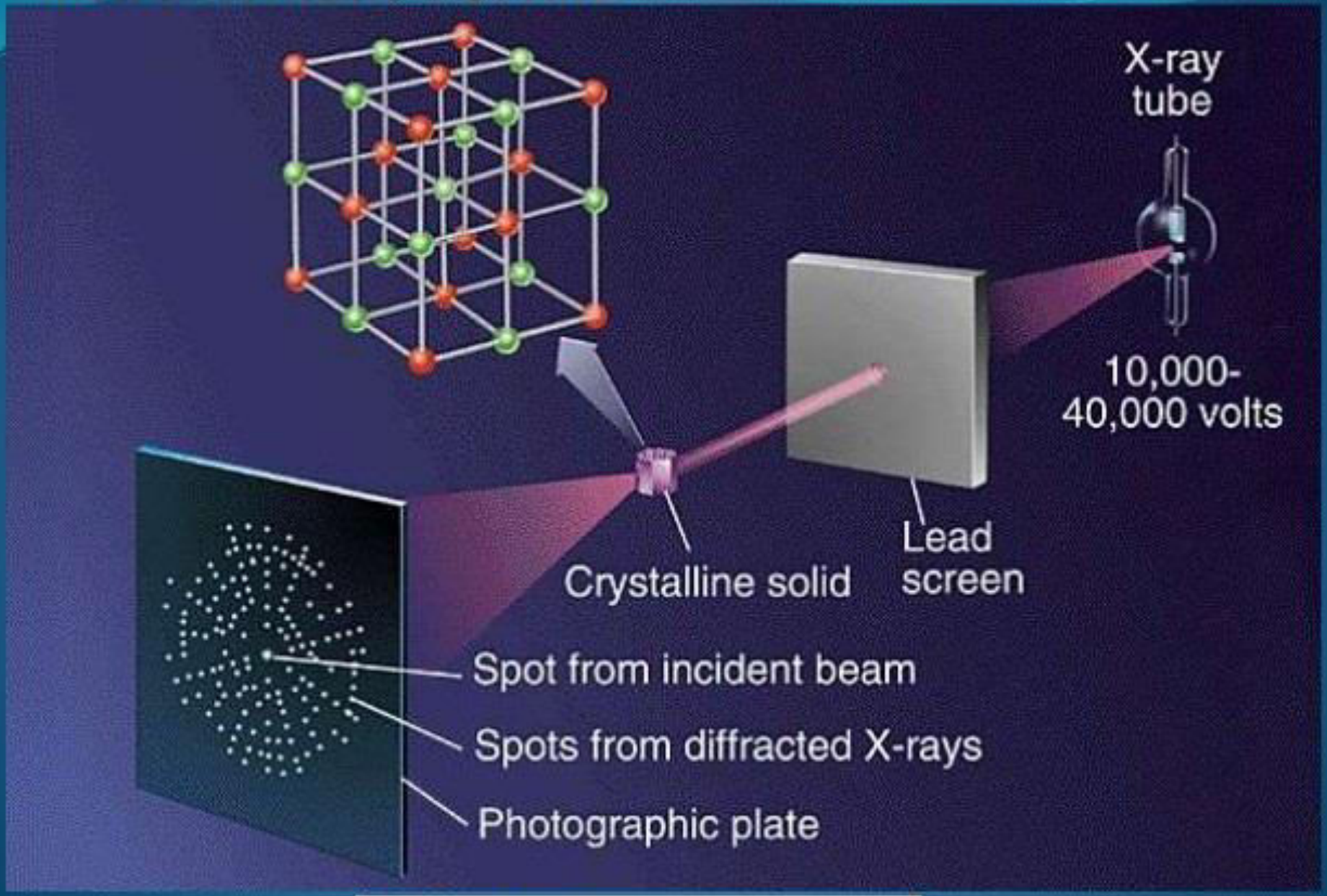
$$n\lambda = 2d\sin\theta$$

this is called as BRAGG'S LAW

INSTRUMENTATION

- Production of x-rays
- Collimator
- Monochromator
  - a.Filter
  - b.Crystal monochromator
- Detectors
  - a.Photographic methods
  - b.Counter methods

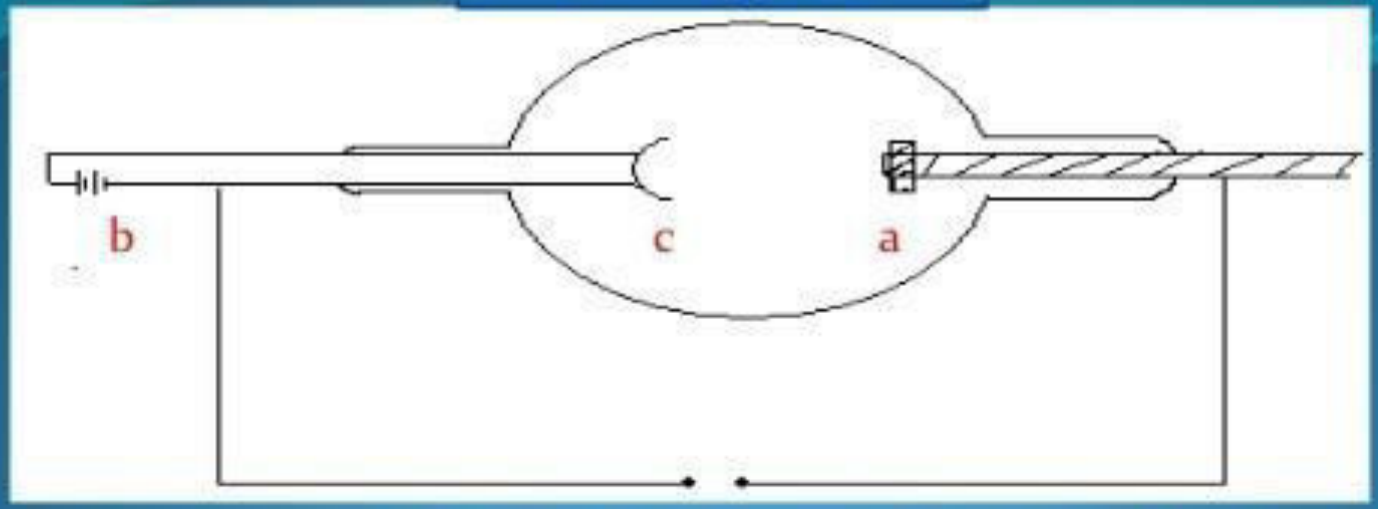
Instrumentation of XRD



PRODUCTION OF X-RAYS:

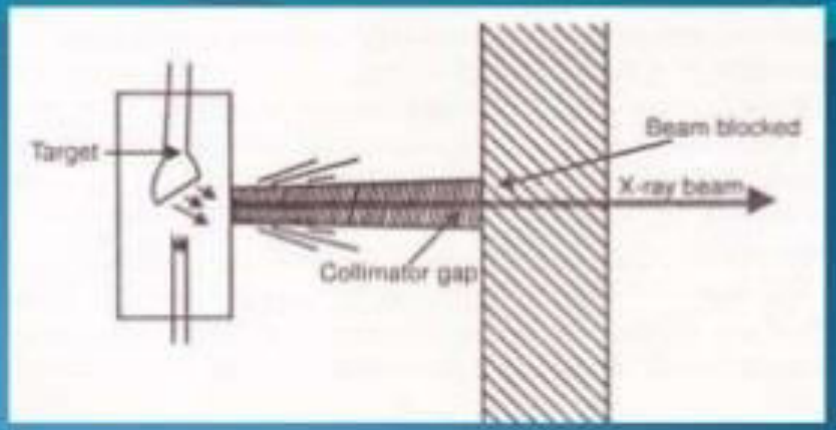
- X-rays are generated when high velocity electrons impinge on a metal target.
- Approximately 1% of the total energy of the electron beam is converted into x-radiation.
- The remainder being dissipated as heat.
- Many types of x-ray tubes are available which are used for producing x-rays.

Coolidge tube



- a . Positive voltage in the form of anode having a target a
- b . Battery to emit thermoionic electrons
- C. Cathode –filament of tungsten metal
- The electrons are accelerated towards the target a
- On striking the target the electrons transfer their energy to its metallic surface which gives off x-ray radiation

COLLIMATOR:



- In order to get a narrow beam of x-rays, the x-rays generated by the target material are allowed to pass through a collimator which consists of two sets of closely packed metal plates separated by a small gap.

- The collimator absorbs all the x-rays except the narrow beam that passes between the gap.

TYPES OF MONOCHROMATORS

In order to do monochromatization,2 methods are available

1.Filter

2.Crystal monochromator

a)Flat crystal monochromator

b)Curved crystal monochromator

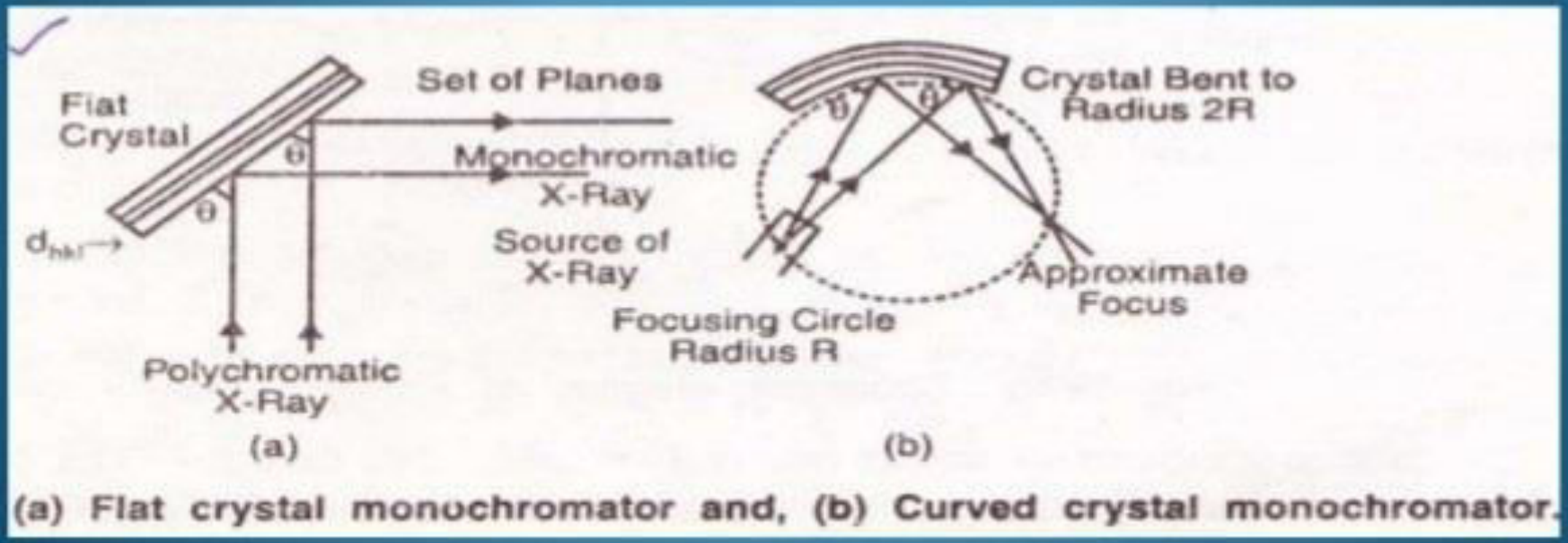
Materials used-Nacl,quartz etc,.

- A.FILTER: X-ray beam may be partly monochromatized by insertion of a suitable filter
- A filter is a window of material that absorbs undesirable radiation but allows the radiation of required wavelength to pass

•2)CRYSTAL MONOCHROMATOR : Crystal monochromators is made up of suitable crystalline material positioned in the x-ray beam so that the angle of reflecting planes satisfied the Bragg's equation for the required wavelength

the beam is split up into component wavelengths

crystals used in monochromators are made up of materials like Nacl, lithium fluoride , quartz etc.



(a) Flat crystal monochromator and, (b) Curved crystal monochromator.

DETECTORS

- The x-ray intensities can be measured and recorded either by
- 1)Photographic methods

- 2)Counter methods
- a) Geiger - Muller tube counter
- b) Proportional counter
- c) Scintillation detector
- d) Solid state semi conductor detector
- e) Semi conductor detectors

- Both these types of methods depends upon ability of x-rays to ionize matter and differ only in the subsequent fate of electrons produced by the ionizing process.

- COUNTER METHODS:
- a) Geiger - Muller tube counter

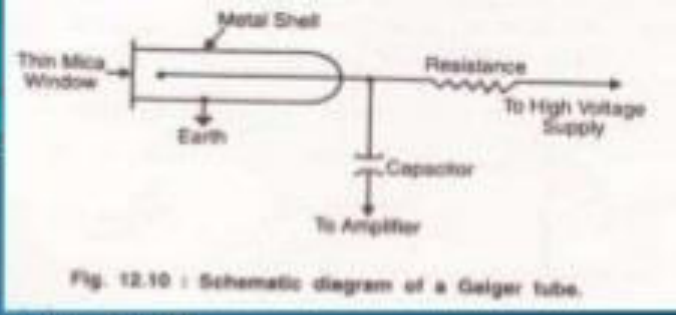


Fig. 12.10 : Schematic diagram of a Geiger tube.

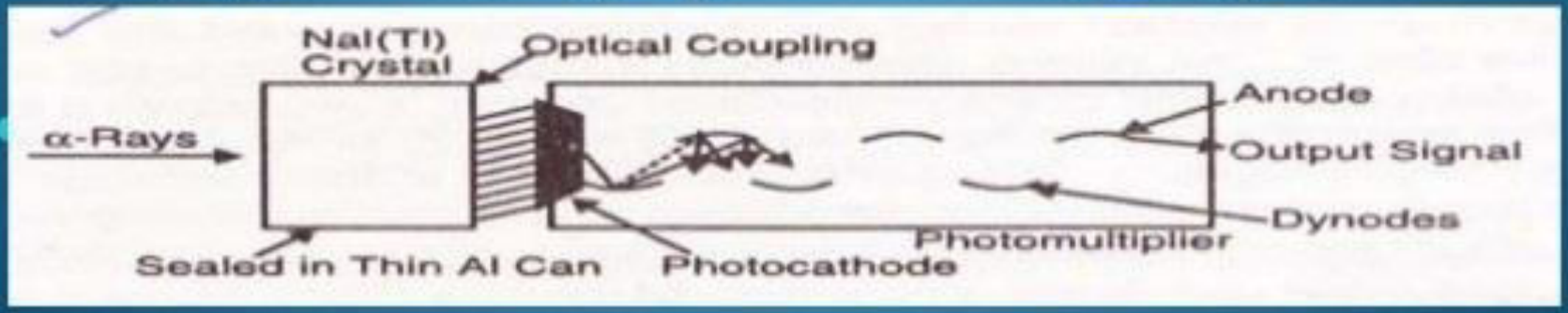
- Geiger tube is filled with inert gas like argon
- Central wire anode is maintained at a positive potential of 800 to 2500V .

X-RAY ⟹ Collision with filling gas ⟹ Production of an ion pair → Electon-central anode; Positive ion-moves to outer electrode

- The electron is accelerated by the potential gradient and causes the ionisation of large number of argon atoms ,resulting in the production of avalanche of electrons that are travelling towards central anode

C)SCINTILLATION DETECTOR:

- In a scintillation detector there is large sodium iodide crystal activated with a small amount of thallium
- When x-ray is incident upon crystal , the pulses of visible light are emitted which can be detected by a photo multiplier tube
- Useful for measuring x-ray of short wavelength
- Crystals used in scintillation detectors include sodium iodide , anthracene ,napthalene and p-terphenol



d)Solid state semi-conductor detector

- In this type of detector ,the electrons produced by x-ray beam are promoted into conduction bands and the current which flows is directly proportional to incident x-ray energy
- Dis advantage:
- Semi – conductor device should be maintained at low temperatures to minimize noise and prevent deterioration

X-RAY DIFFRACTION METHODS

These are generally used for investigating the internal structures and crystal structures of various solid compounds.

They are

1.Laue's photographic method

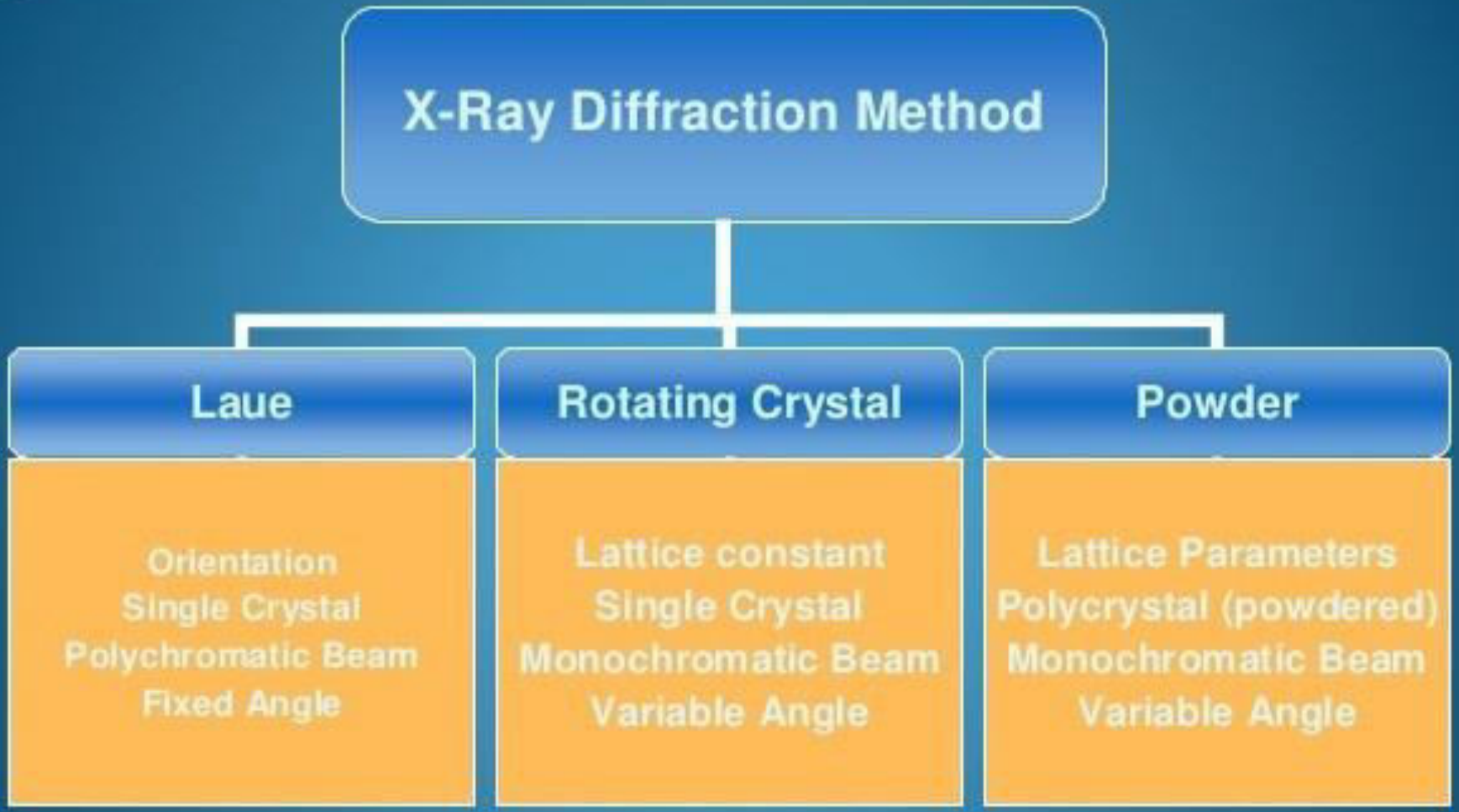
a)Transmission method

b)Back reflection method

2.Bragg's X-ray spectrometer method

3.Rotating crystal method

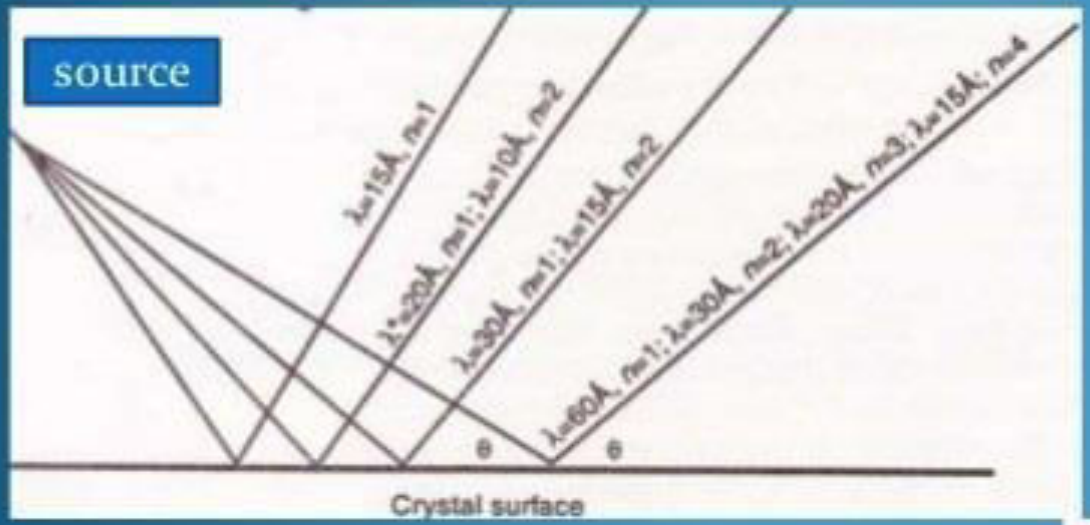
4.Powder method

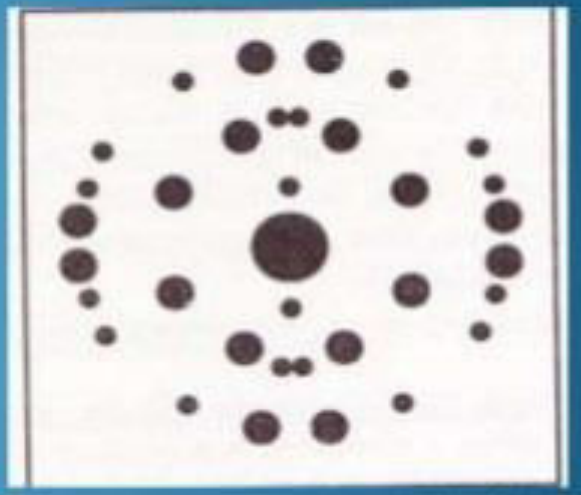


X-RAY DIFFRACTION

The Bragg's x-ray spectrometer method:

- Laue-beam of x-ray-crystal-emitted x-ray obtained on photographic plate-using photograph-brag analysed structures of crystals of Nacl,Kcl,and Zns-brags equation





Diffraction pattern of a single crystal of an inorganic salt.

- Single plane generates several diffraction lines-sum tot of diffraction lines gives diffraction patterns-from the pattern we can deduce different distances between planes-angle between planes in each of three dimensions

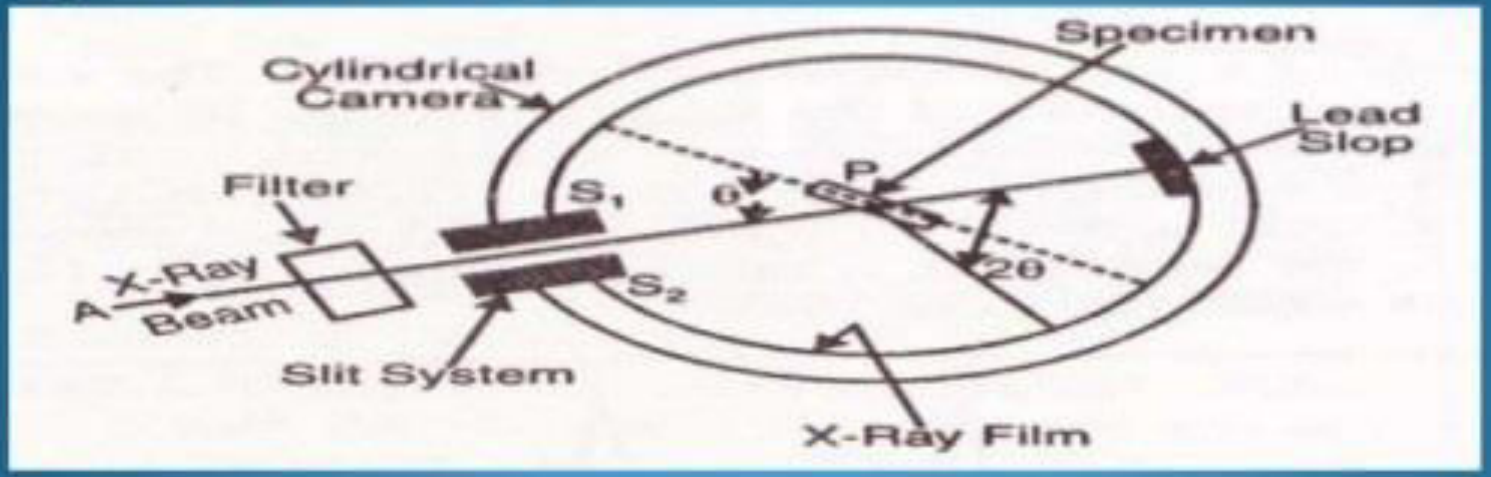
The Bragg's x-ray spectrometer method

Working:

- Crystal is mounted such that θ=0° and ionization chamber is adjusted to receive x-rays
- Crystal and ionization chamber are allowed to move in small steps
- The angle through which the chamber is moved is twice the angle through which the crystal is rotated
- X-ray spectrum is obtained by plotting a graph between ionization current and the glancing angleθ
- Peaks are obtained.peaks corresponds to Bragg's reflection
- Different order glancing angles are obtained with known values of d and n and from the observed value of θ , λ can be measured.

POWDER CRYSTAL METHOD:

X-ray powder diffraction (XRD) is a rapid analytical technique primarily used for phase identification of a crystalline material and can provide information on unit cell dimensions. The analyzed material is finely ground, homogenized, and average bulk composition is determined.

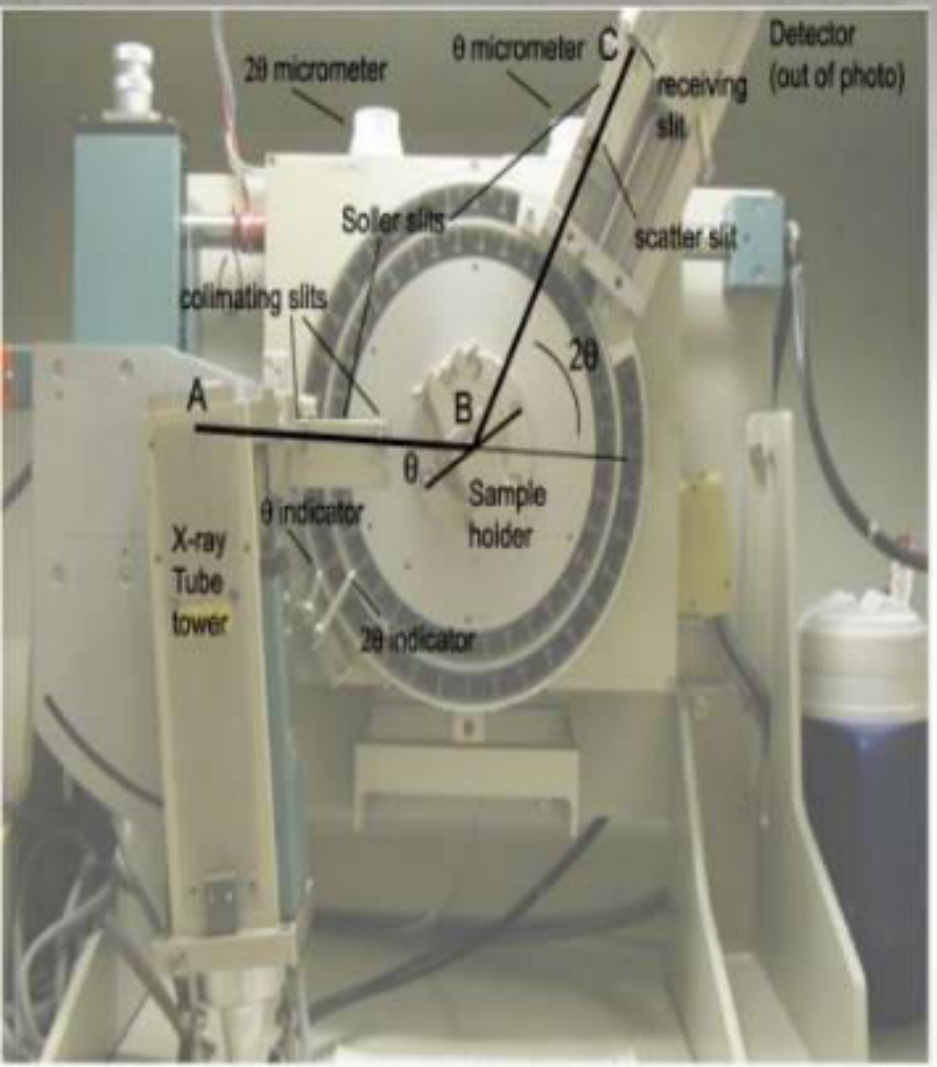


Fine powder is struck on a hair with a gum ,it is suspended vertically in the axis of a cylindrical camera

- When monochromatic beam is allowed to pass different possibilities may happen

1. There will be some particles out of random orientation of small crystals in the fine powder
2. Another fraction of grains will have another set of planes in the correct positions for the reflections to occur
3. Reflections are possible in different orders for each set

X-RAY DIFFRACTION


2θ micrometer
θ micrometer
C
Detector
(out of photo)
receiving
slit
Soller slits
scatter slit
collimating slits
2θ
A
B
θ
Sample
holder
θ indicator
X-ray
Tube
tower
2θ indicator

- If the angle of incidence is θ then the angle of reflection will be 2θ
- If the radius is r the circumference 2πr corresponds to a scattering angle of 360°

$$\Theta = 360 * 1/\pi r$$

- From the above equation the value of θ can be calculated and substituted in bragg's equation to get the value of d

- **Applications**
- Useful for determining the complex structures of metals and alloys
- characterization of crystalline materials
- identification of fine-grained minerals such as clays and mixed layer clays that are difficult to determine optically
- determination of unit cell dimensions
- measurement of sample purity

APPLICATIONS OF XRD

1. Structure of crystals
2. Polymer characterisation
3. State of anneal in metals
4. Particle size determination

a) Spot counting method
b) Broadening of diffraction lines
c) Low-angle scattering

5. Applications of diffraction methods to complexes

a) Determination of cis-trans isomerism
b) Determination of linkage isomerism

6. Miscellaneous applications

THANK YOU