JIWAJI UNIVERSITY GWALIOR (M.P.)

(SOS INDUSTRIAL CHEMISTRY)

TOPIC- X- ray diffraction

SUBMITTED BY –
Ragini Tomar
Guest Faculty in Industrial Chemistry Department

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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

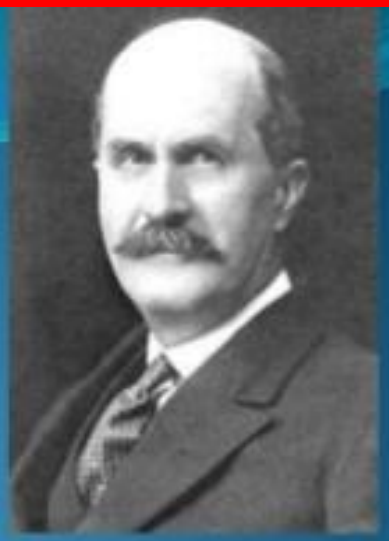
X-RAYS

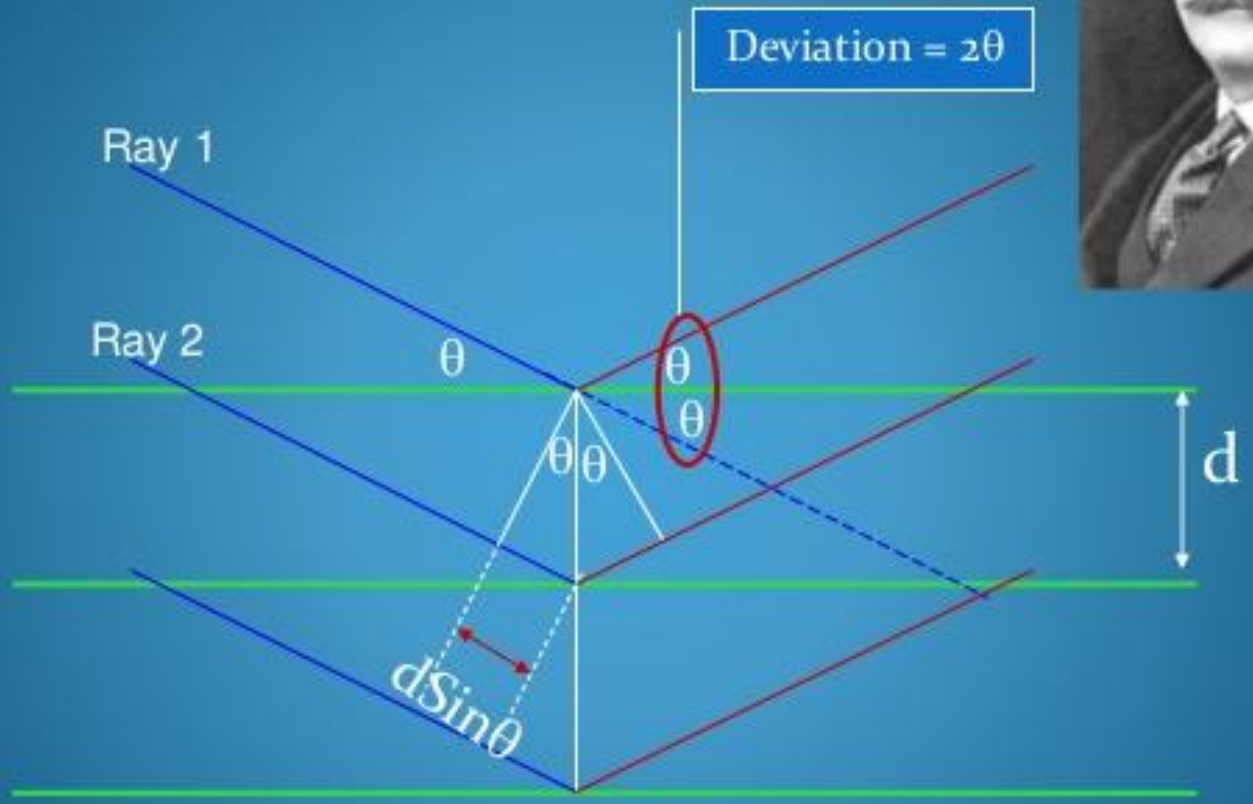
1. X-rays are short wave length electromagnetic radiations produced by the deceleration of high energy electrons or by electronic transitions of electrons in the inner orbital of atoms
2. X-ray region 0.1to100 A°
3. Analytical purpose 0.7 to 2 A°

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
- For constructive interference: $n\lambda = 2d\,Sin\theta$

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,

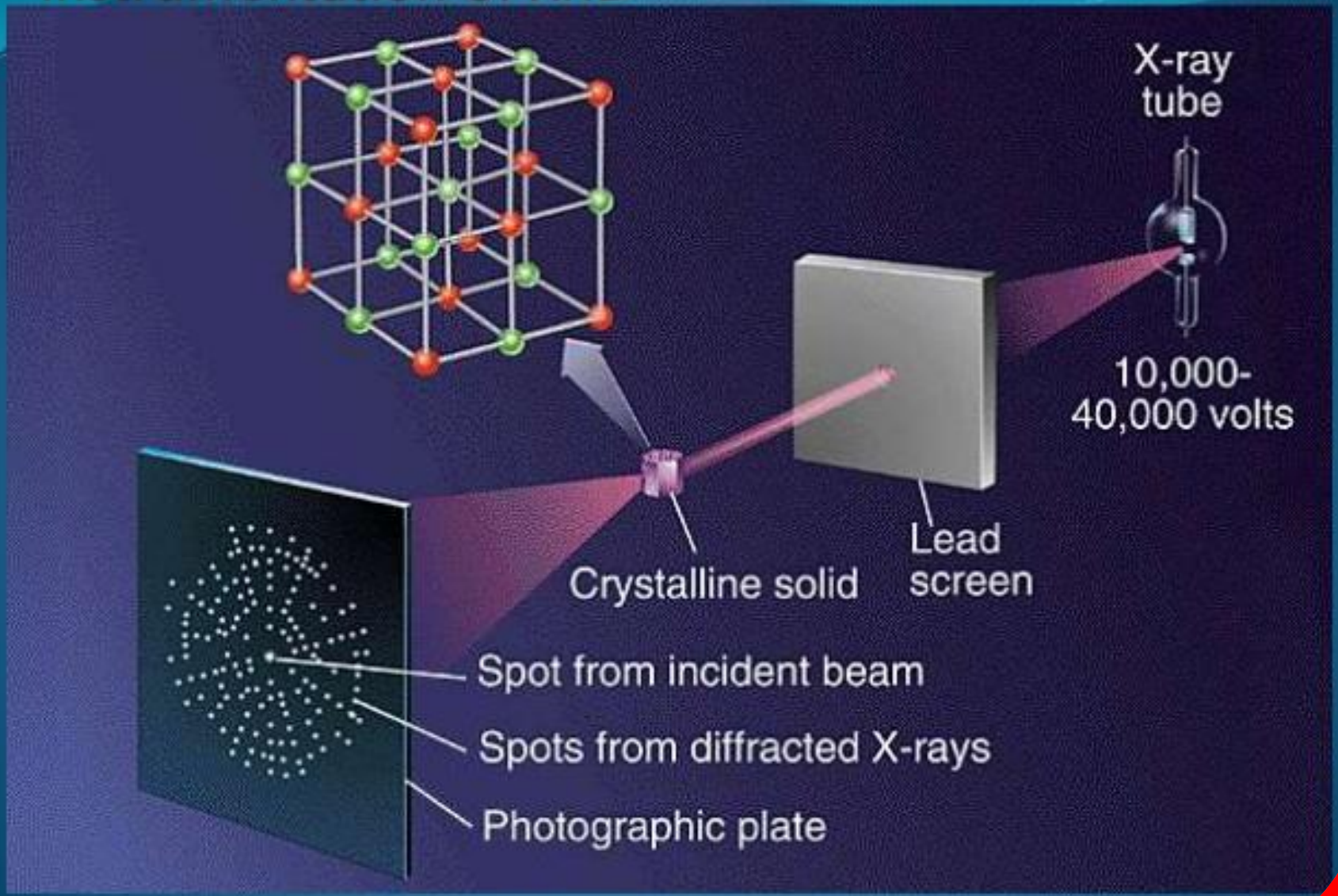
$$n\lambda=2d\sin\theta$$

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

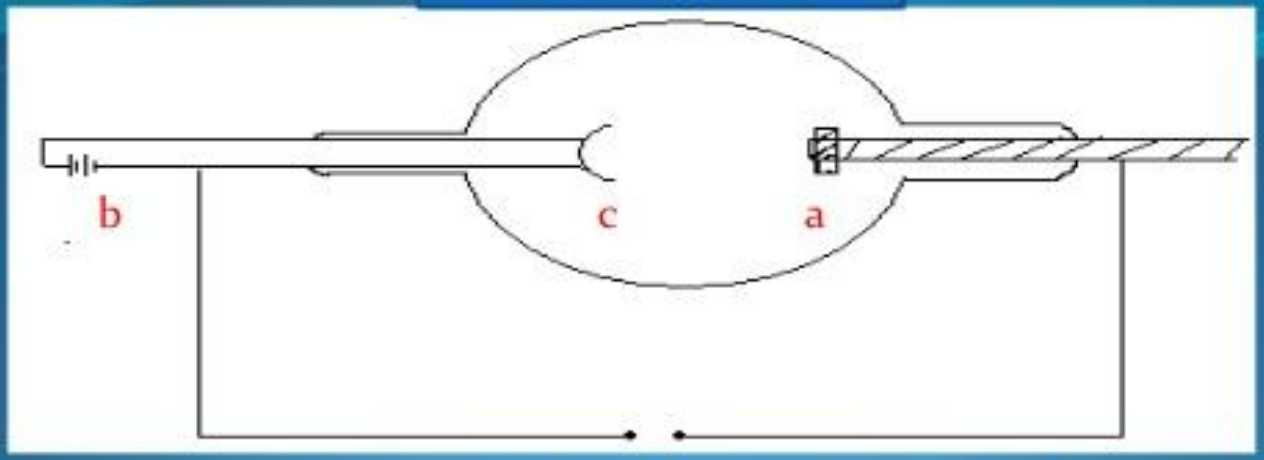
Instrumentation of XRD



PRODUCTION OF X-RAYS:

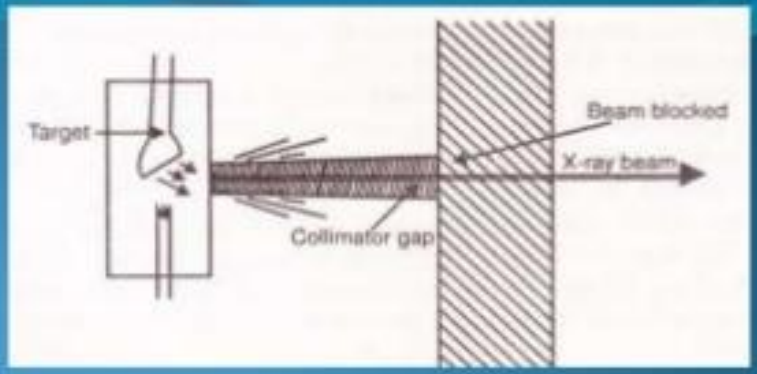
- X-rays are generated when high velocity electrons impinge on a metal target.
- Approximately 1% of the total energy of the electron beam is converted into x-radiation.
- The remainder being dissipated as heat.
- Many types of x-ray tubes are available which are used for producing x-rays.

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

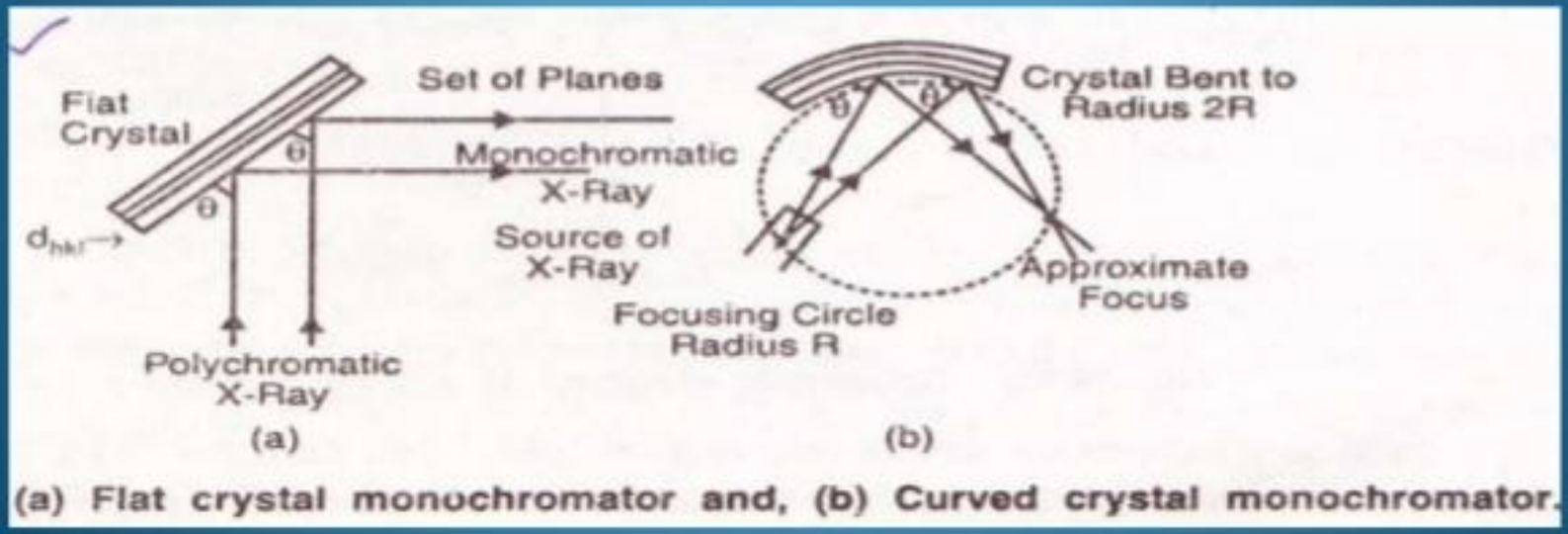
Materials used-Nacl,quartz etc,.

- 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.

- Photographic method: To record the position and intensity of x-ray beam a plane or cylindrical film is used
- The film after exposing to x-ray is developed
- The blackening of the developed film is expressed in terms of density units D given by

$$D=\log I_0/I$$

I_0- incident intensities

I- transmitted intensities

D-Total energy that causes blackening of the film

D is measured by densitometer

The photographic method is mainly used in diffraction studies since it reveals the entire diffraction pattern on a single film .

Dis advg: time consuming and uses exposure of several hours

COUNTER METHODS:

- a) Geiger - Muller tube counter

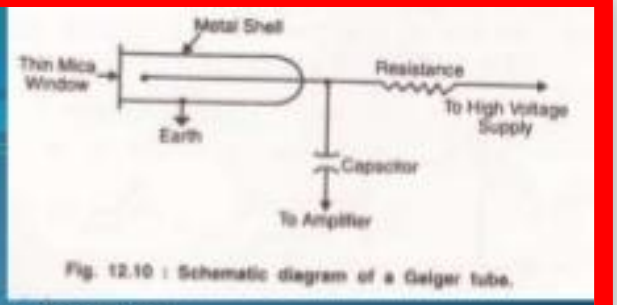
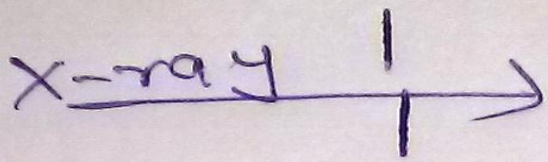


Fig. 12.10 : Schematic diagram of a Geiger tube.

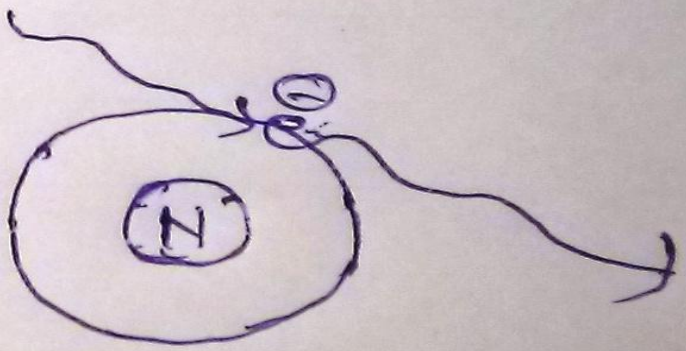
- Geiger tube is filled with inert gas like argon
- Central wire anode is maintained at a positive potential of 800 to 2500V .

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


X-ray


X-ray
ZnS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Thanks