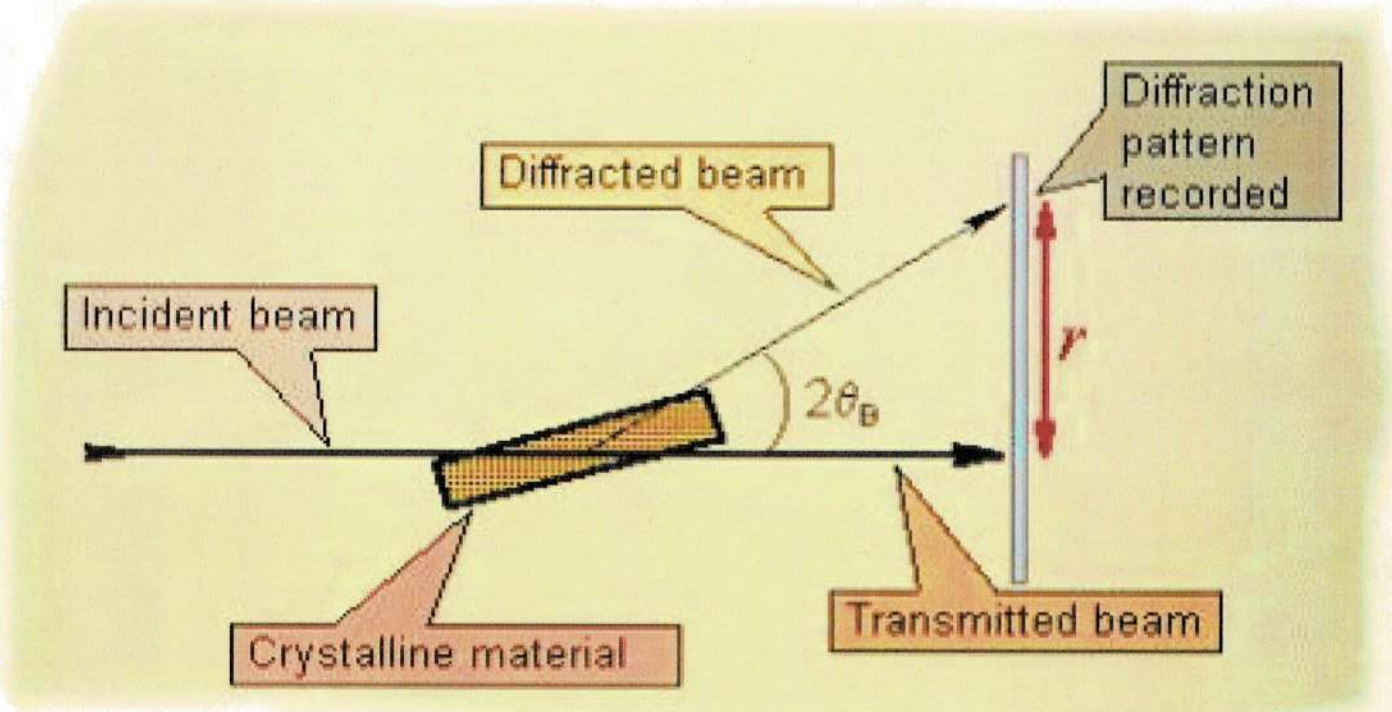
X-RAY DIFFRACTION:



- 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.

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

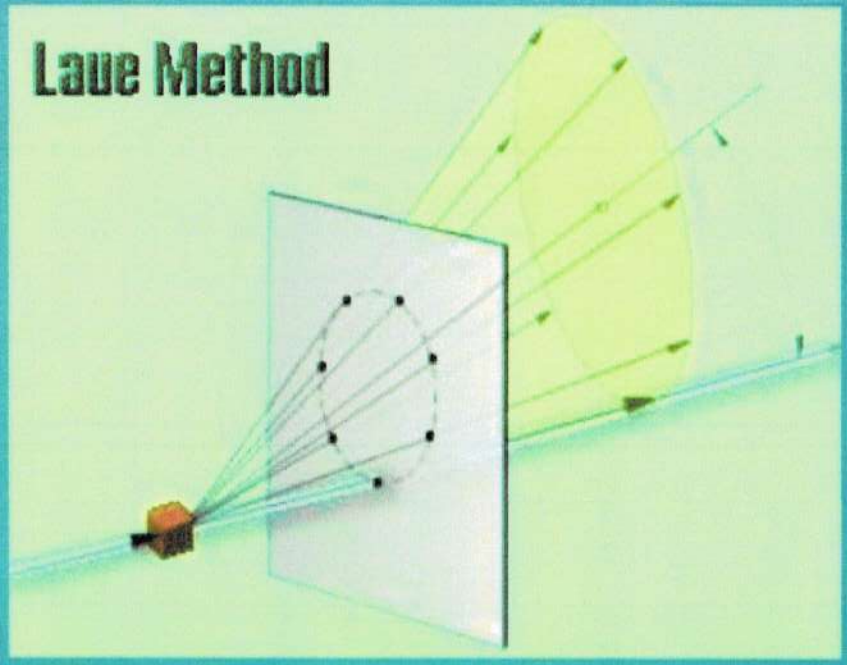
b)Back reflection method

2.Bragg's X-ray spectrometer method

3.Rotating crystal method

4.Powder method

a) Transmission Laue method



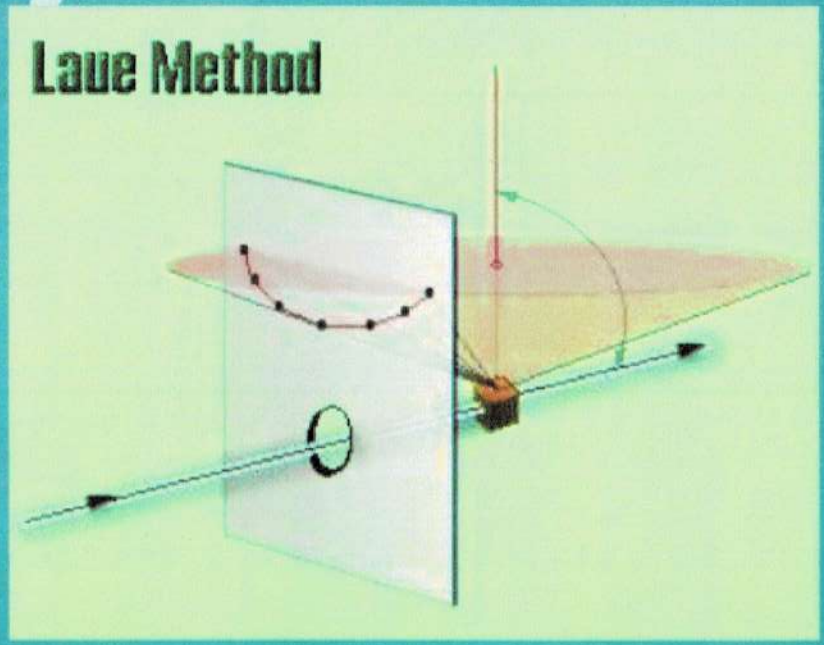
In the transmission Laue method, the film is placed **behind** the crystal to record beams which are transmitted through the crystal.
One side of the cone of Laue reflections is defined by the transmitted beam. The film intersects the cone, with the diffraction spots generally lying on an ellipse.

•Can be used to orient crystals for solid state experiments.

•Most suitable for the investigation of preferred orientation sheet particularly confined to lower diffraction angles.

•Also used in determination of symmetry of single crystals.

b)Back-reflection method



- In the back-reflection method, the film is placed **between** the x-ray source and the crystal. The beams which are diffracted in a backward direction are recorded.
- One side of the cone of Laue reflections is defined by the transmitted beam. The film intersects the cone, with the diffraction spots generally lying on an hyperbola.

- This method is similar to Transmission method however, black-reflection is the only method for the study of large and thick specimens.

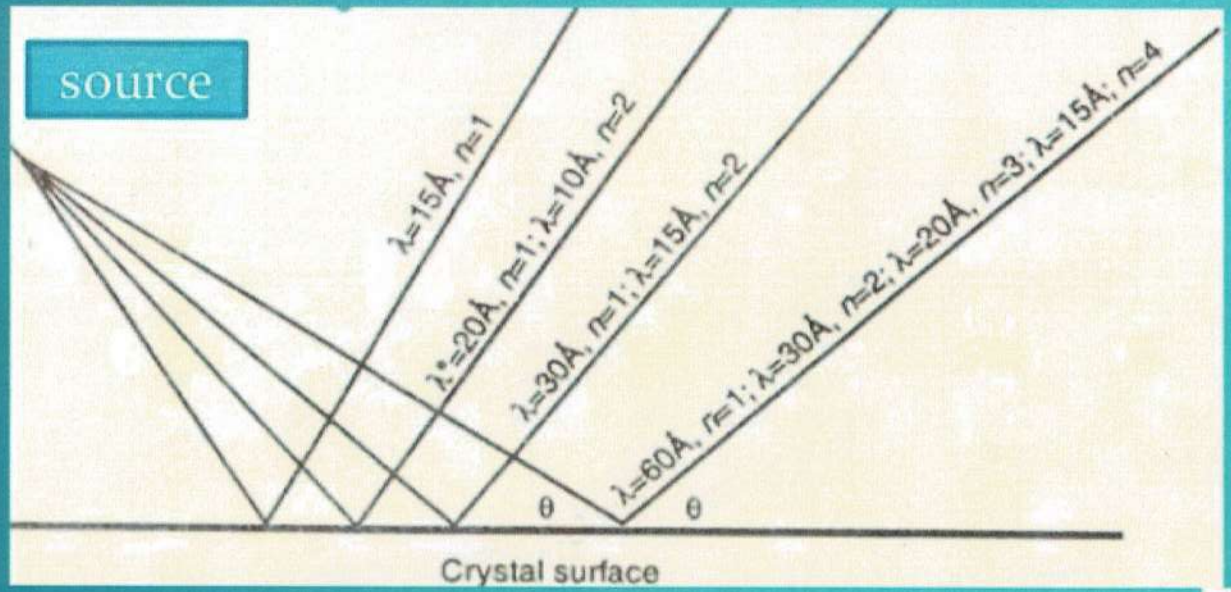
➢ **Disadvantage:**

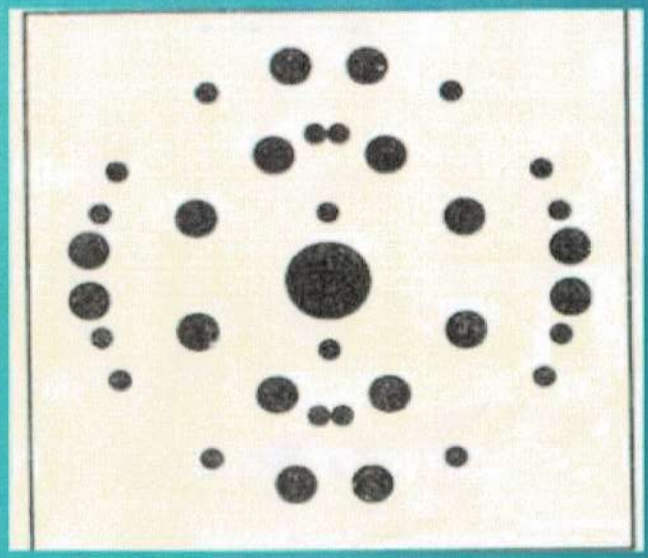
- Big crystals are required

- Crystal orientation is determined from the position of the spots. Each spot can be indexed, i.e. attributed to a particular plane, using special charts.
- The Greninger chart is used for back-reflection patterns and the Leonhardt chart for transmission patterns.
- The Laue technique can also be used to assess crystal perfection from the size and shape

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

- 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:

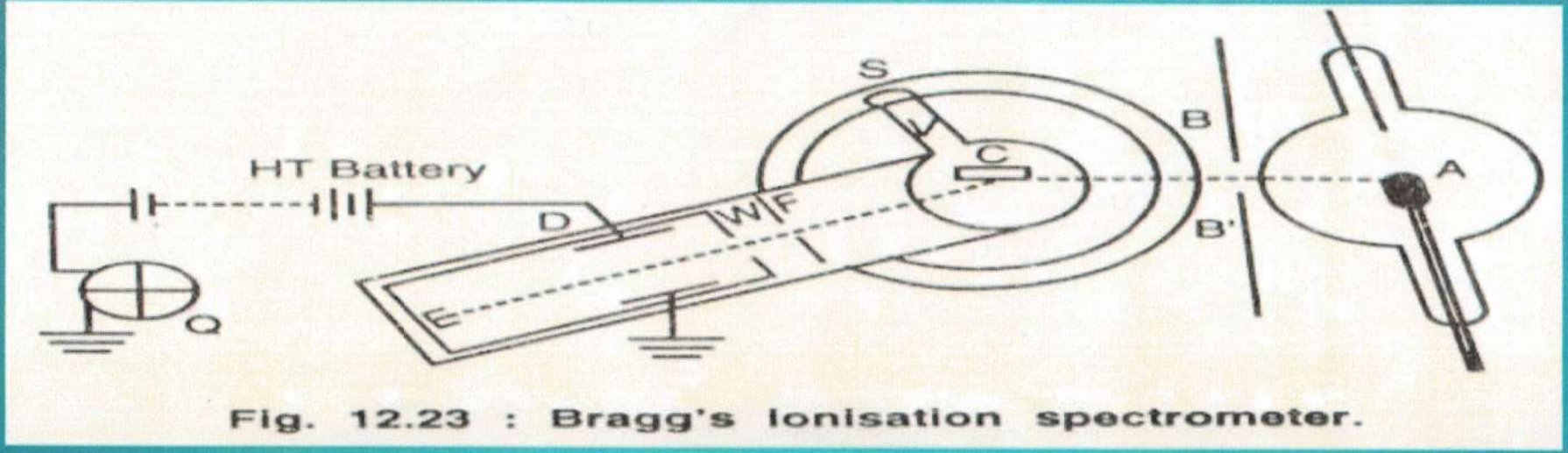


Fig. 12.23 : Bragg's Ionisation spectrometer.

- A-anti cathode
- B-B' – Adjustable slits
- C-crystal
- E-ionization chamber
- One plate of ionization chamber is connected to the positive terminal of a H.T Battery , while negative terminal is connected to quadrant electrometer(measures the strength of ionization current)

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.