POWDER METHOD OR DEBYE SHERRER METHOD OF XRD

❖ Introduction:

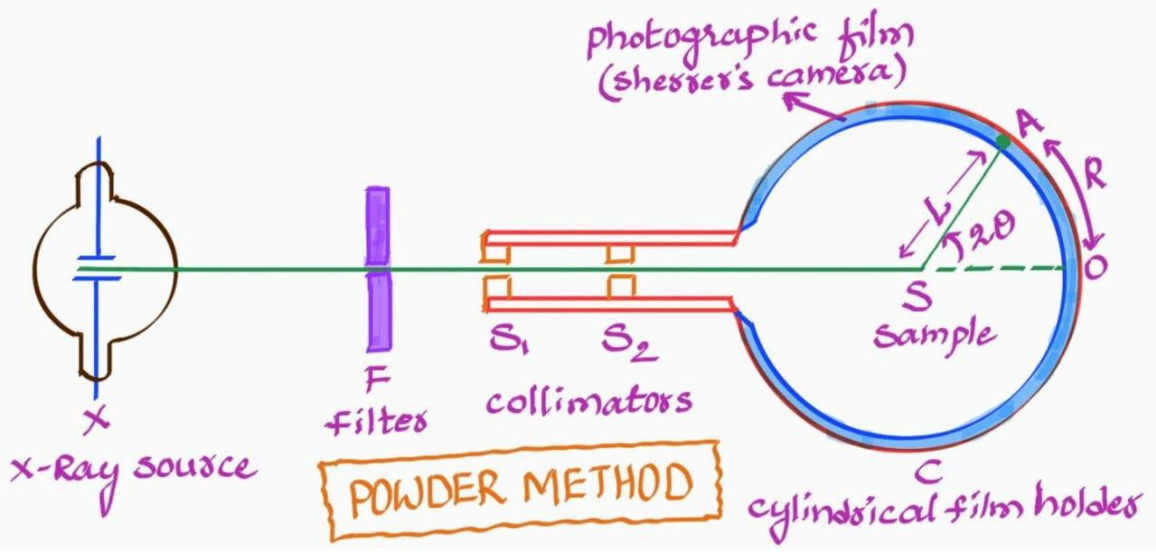
- ❖ The Powder Method, also called the Debye–Scherrer Method, is a widely used X-ray diffraction technique.
- ❖ The powder method was developed by Debye and Scherrer in 1916 in Germany.
- ❖ It is used to investigate crystal structures when single crystals of sufficient size are not available.
- ❖ It is suitable for studying polycrystalline or powdered samples (microcrystalline materials).

❖ Working Principle:

- ❖ This method works on the principle of X-ray diffraction by randomly oriented microcrystals in a powdered sample. When monochromatic X-rays strike the powdered specimen, diffraction occurs at specific angles (**θ**) that satisfy Bragg's law($2d \sin\theta = n\lambda$).

❖ Main components- Setup or Experimental setup:

- ❖ **X-ray source (X):** Produces X-rays of known wavelength.
- ❖ **Filter(F):** Provides a narrow monochromatic X-ray beam by removing unwanted radiations.
- ❖ **Collimators (S1 & S2):** Makes the X-ray beam narrow and directs into a fine parallel beam.
- ❖ **Specimen (S):** Finely powdered sample placed in a thin glass capillary tube.
- ❖ **Cylindrical film holder (C):** Photographic film is mounted on the inside surface of the cylinder to record diffraction patterns.



❖ Working:

- ❖ The specimen is taken in the form of a powdered sample in a thin glass capillary tube and fixed along the axis of the cylindrical film camera.
- ❖ X-rays from the source are filtered and collimated into a narrow beam, which falls on the powdered specimen.
- ❖ When this incident X-ray beam strikes millions of randomly oriented tiny crystals, all values of glancing angles are obtained (because each tiny crystal has a different orientation).
- ❖ For a given wavelength (λ) and spacing (d), only one value of θ will satisfy the Bragg's condition i.e., **$2d \sin\theta = n\lambda$**
- ❖ These diffracted rays are captured on the photographic film mounted inside the cylindrical camera, producing circular diffraction rings called Debye–Scherrer rings.
- ❖ Let **L** be the radius of the cylindrical camera and **R** be the distance from the point where the direct beam strikes the film **O** to the point where a diffracted spectrum is recorded **P**.
- ❖ The glancing(Bragg's) angle θ can be calculated using the relation **$\theta = R/2L$**

❖ Applications:

- ❖ Determination of crystal structures of polycrystalline materials.
- ❖ Measurement of interplanar spacing (d).
- ❖ Identification of unknown substances (phase identification).
- ❖ Study of metals, alloys, carbon, fluorescent powders, ceramics, etc.

❖ Advantages:

- ❖ Works even when single crystals cannot be prepared.
- ❖ Simple method for studying fine powders and polycrystalline materials.
- ❖ Requires only a small quantity of sample.

❖ Limitations:

- ❖ Cannot provide a full 3D crystal structure as single-crystal methods do.
- ❖ Information may overlap when many diffraction rings are close together.