X-RAY DIFFRACTION METHODS

The phenomenon of x-ray diffraction is employed to determine the structure of solids as well as for the study of x-ray spectroscopy. The underlying principle in both the cases is the Bragg's law as given by $2d\sin\theta = n\lambda$. Considering only the first order reflections from all the possible atomic planes, real or fictitious, the Bragg's law may be written as

$$2d\sin\theta = \lambda \qquad (1)$$

The reflections take place for those values of d, θ and λ which satisfy the above equation. For structural analysis, x-rays of known wavelength are employed and the angles for which reflections take place are determined experimentally. The d values corresponding to these reflections are then obtained from Eq. (1). Using this information, one can proceed to determine the size of the unit cell and the distribution of atoms within the unit cell. In the x-ray spectroscopy, x-rays are incident on a particular cleavage surface of a single crystal so that the interplaner spacing d is known. The angle for which reflections take place are determined experimentally. The wavelength λ of the incident x-rays is then obtained from Eq. (1).

Laue's Method

An experimental arrangement used to produce Laue's patterns is shown in fig. 1. It consists of a flat plate camera which contains a collimator with a fine hole to obtain a very fine beam of X-rays. The sample is placed on a goniometer which can be rotated to change the orientation of the single crystal. Two flat photographic films are used, one for receiving the transmitted diffracted beam and the other for receiving the reflected diffracted beam for back reflection experiments. Such experiments are performed particularly when there is excessive absorption of x-rays in the crystal.

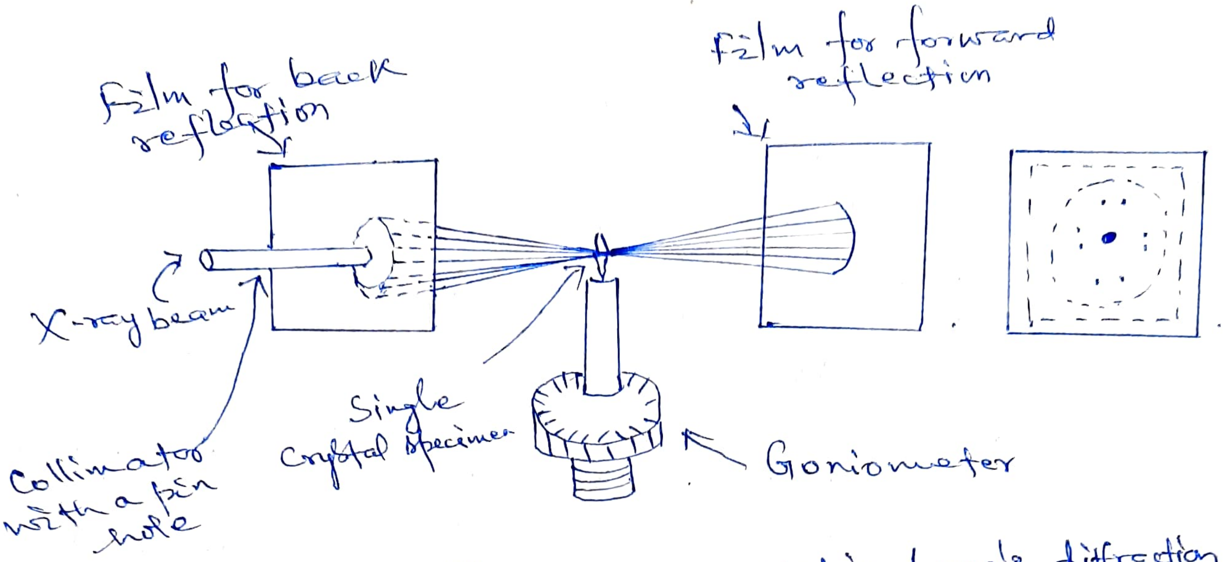

Fig. 1. A flat plane camera used in Laue's diffraction method

Initially, a single crystal specimen having dimensions of the order of 1mm×1mm×1mm is held stationary in the path of white X-rays having wavelengths ranging from 0.2 to 2 A°. Since the crystal contains a number of sets of parallel atomic planes with different interplanar spacings, diffraction is possible for certain values of λ and d which satisfy the Bragg's condition. Thus diffraction spots are produced on the photographic film as shown in fig. 1. The crystal can be rotated with the help of goniometer to change its orientation with respect to the incident beam. By doing so, the diffraction condition may be satisfied for a new set of atomic planes and hence a different type of pattern may be obtained on the photographic film. The symmetry of the crystal is, however, reflected in each pattern.

The Laue's method is mostly used to determine the crystal symmetry. For example, if a crystal having four-fold axial symmetry is oriented so that its axis is parallel to the beam, the resulting Laue's pattern also exhibits the four-fold symmetry. The symmetry of the pattern helps to determine the shape of the unit cell. It is, however, not practicable to determine the structure of the crystal by this method.

It is because a number of wavelengths may be reflected from a single plane in different orders and may superpose at a single point resulting in the loss of a number of reflections. The symmetry of the Laue's pattern also helps to orient the crystals for various solid state experiments. Another application of the Laue's method is the determination of imperfections or strains in the crystal. An imperfect or strained crystal has atomic plane which are not exactly plane but are slightly curved. Thus instead of sharp diffraction spots one gets streaks in the Laue's pattern. This type of streaking on Laue's photographs is called asterism.