

which any of these planes in a crystal reflect an X-ray beam of wavelength λ may be calculated by inserting the appropriate value of d into the Bragg equation.

To ensure that Bragg's law is satisfied and that reflections from various crystal planes can occur, it is necessary to provide a range of either θ or λ values. The various ways in which this can be done leads to the standard methods of X-ray diffraction, namely: (i) the Laue method and (ii) the powder method.

5.3.3 X-ray diffraction methods

5.3.3.1 Laue method

In the Laue method, a stationary single crystal is bathed in a beam of 'white' radiation. Then, because the specimen is a fixed single crystal, the variable necessary to ensure that the Bragg's law is satisfied for all the planes in the crystal has to be provided by the range of wavelengths in the beam, i.e. each set of crystal planes chooses the appropriate λ from the 'white' spectrum to give a Bragg reflection. Radiation from a target metal having a high atomic number (e.g. tungsten) is often used, but almost any form of 'white' radiation is suitable. In the experimental arrangement shown in Figure 5.9, either a transmission photograph or a back-reflection photograph may be taken, and the pattern of spots which are produced lie on ellipses in the transmission case or hyperbolae in the back-reflection case. All spots on any ellipse or hyperbola are reflections from planes of a single zone (i.e. where all the lattice planes are parallel to a common direction, the zone axis) and, consequently, the Laue pattern is able to indicate the symmetry of the crystal. For example, if the beam is directed along a $[1\ 1\ 1]$ or $[1\ 0\ 0]$ direction in the crystal, the Laue pattern will show three- or fourfold symmetry, respectively. The Laue method is used extensively for the determination of the orientation of single crystals and, while charts are available to facilitate this determination, the method consists essentially of plotting the zones taken from the film onto a

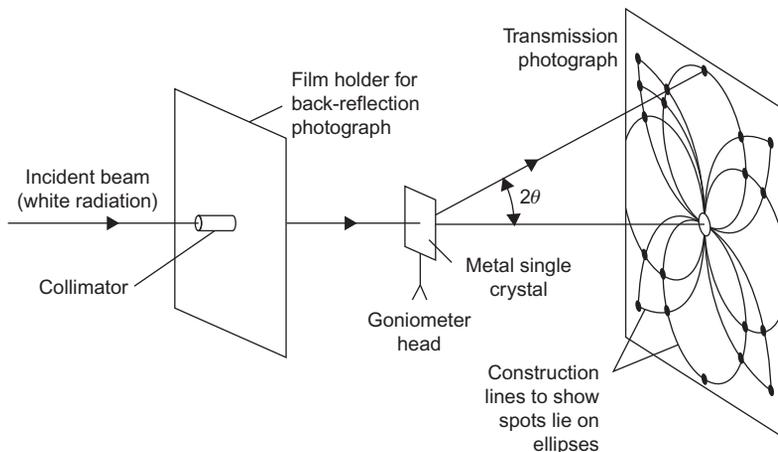


FIGURE 5.9

Laue method of X-ray diffraction.

stereogram, and comparing the angles between them with a standard projection of that crystal structure. In recent years the use of the Laue technique has been extended to the study of imperfections resulting from crystal growth or deformation, because it is found that the Laue spots from perfect crystals are sharp, while those from deformed crystals are elongated. This elongated appearance of the diffraction spots is known as asterism and it arises in an analogous way to the reflection of light from curved mirrors.

5.3.3.2 Powder method

The powder method, devised independently by Debye and Scherrer, is probably the most generally useful of all the X-ray techniques. It employs monochromatic radiation and a finely powdered, or fine-grained polycrystalline, wire specimen. In this case, θ is the variable, since the collection of randomly oriented crystals will contain sufficient particles with the correct orientation to allow reflection from each of the possible reflecting planes, i.e. the powder pattern results from a series of superimposed rotating crystal patterns. The angle between the direct X-ray beam and the reflected ray is 2θ , and consequently each set of crystal planes gives rise to a cone of reflected rays of semi-angle θ , where θ is the Bragg angle for that particular set of reflecting planes producing the cone. Thus, if a film is placed around the specimen, as shown in Figure 5.10, the successive diffracted cones, which consist of rays from hundreds of grains, intersect the film to produce concentric curves around the entrance and exit holes. Figure 5.11 shows examples of patterns from bcc and fcc materials, respectively.

Precise measurement of the pattern of diffraction lines is required for many applications of the powder method, but a good deal of information can readily be obtained merely by inspection. One example of this is in the study of deformed metals, since after deformation the individual spots on the diffraction rings are blurred so much that line broadening occurs, especially at high Bragg angles. On low-temperature annealing, the cold-worked material will tend to recover and this is indicated on the photograph by a sharpening of the broad diffraction lines. At higher annealing temperatures the metal will completely regain its softness by a process known as recrystallization (see Chapter 11), and this phenomenon is accompanied by the completion of the line-sharpening process. With continued annealing, the grains absorb each other to produce a structure with an overall coarser grain size and, because fewer reflections are available to contribute to the diffraction cones,

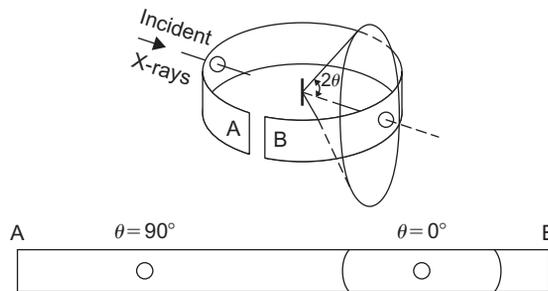


FIGURE 5.10

Powder method of X-ray diffraction.

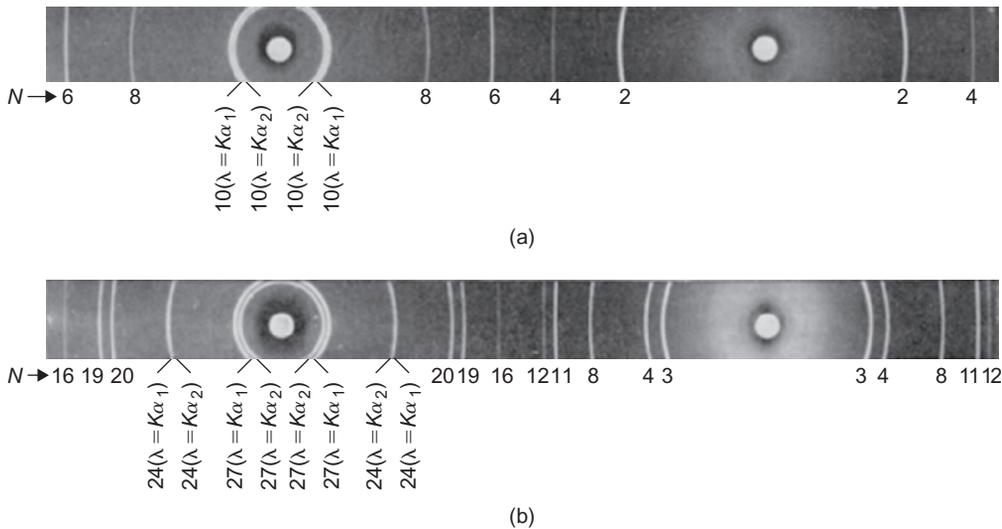


FIGURE 5.11

Powder photographs taken in a Philips camera (114 mm radius) of (a) iron with cobalt radiation using an iron filter and (b) aluminium with copper radiation using a nickel filter. The high-angle lines are resolved and the separate reflections for $\lambda = K\alpha_1$ and $\lambda = K\alpha_2$ are observable.

the lines on the powder photograph take on a spotty appearance. This latter behaviour is sometimes used as a means of determining the grain size of a polycrystalline sample. In practice, an X-ray photograph is taken for each of a series of known grain sizes to form a set of standards, and with them an unknown grain size can be determined quite quickly by comparing the corresponding photograph with the set of standards. Yet a third use of the powder method as an inspection technique is in the detection of a preferred orientation of the grains of a polycrystalline aggregate. This is because a random orientation of the grains will produce a uniformly intense diffraction ring, while a preferred orientation, or texture, will concentrate the intensity at certain positions on the ring. The details of the texture require considerable interpretation and are discussed in Chapter 11.

WORKED EXAMPLE

A Debye–Scherrer pattern of an unknown powder showed diffraction peaks using copper K_{α} X-rays ($\lambda = 1.54 \text{ \AA}$) at the following Bragg angles: 19.25° , 22.45° , 32.7° , 39.3° and 41.4° . (i) Confirm that the powder is cubic in structure and (ii) determine its lattice parameter. Suggest what the powder might be.

Solution

Bragg's law: $\lambda = \frac{2a}{\sqrt{N}} \sin \theta$, where $N = \sqrt{h^2 + k^2 + l^2}$.