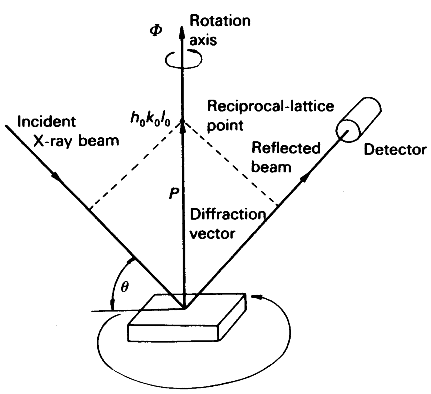
*International Tables for Crystallography* (2006). Vol. C, Figure 5.3.3.7, p. 527.

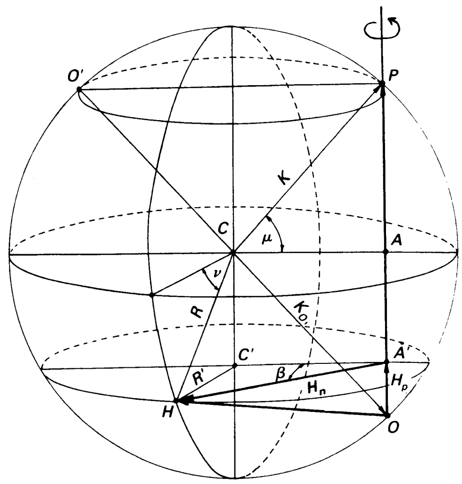


simultaneous reflection or (after Renninger, 1937) ‘*Umweganregung*’, may be observed in both X-ray and neutron experiments. In the first case, it occurs both in methods with counter recording, initiated by Renninger (1937), and in methods with photographic recording in which a highly divergent beam is used (§5.3.2.4.2). The intersections of conic sections encountered in the methods developed by Kossel (1936) and Lonsdale (1947) are the cases of multiple-diffraction phenomena in photographic methods.

Simultaneous reflection, undesirable in some cases (‘forbidden’ reflections in measurements of intensities) can be very useful in others. Its various applications have been reviewed by Terminasov & Tuzov (1964) and Chang (1984). Only the utility of multiple diffraction in lattice-parameter determination will be discussed here.




(*a*)




(*b*)

Fig. 5.3.3.6. Schematic representation of the multiple-diffraction method. (*a*) Experimental set-up (after Cole, Chambers & Dunn, 1962; Post, 1975). (*b*) Geometric representation in reciprocal space.

The principle of Renninger’s (1937) experiment, which is also the basis of the method described by Post (1975), is shown in Fig. 5.3.3.6. The crystal in the shape of a slab is at first set in a position to diffract the primary X-ray beam. A primary reflection whose intensity is very low or which is forbidden by the space group of the crystal is usually selected. Its intensity determines the background intensity of the pattern, which should be low. The detector, with a wide-open window, is situated in the appropriate position and remains fixed throughout the experiment while the crystal is rotated around the axis perpendicular to the crystal planes (and its surface) to record successive reflections.

The multiple-diffraction pattern (an example is shown in Fig. 5.3.3.7) has next to be indexed. The principle of the method of indexing, known as the reference-vector method (Cole, Chambers & Dunn, 1962; Post, 1975; Chang, 1984), is shown in Fig. 5.3.3.6(*b*). Directions of the primary and diffracted beams are marked by vectors $\mathbf{K}_0$ and $\mathbf{K}$. The ends of the vectors lie on the Ewald sphere, the radius of which is equal to $1/\lambda$. The reciprocal vector $\mathbf{P} = h_0\mathbf{a}^* + k_0\mathbf{b}^* + l_0\mathbf{c}^*$, being the difference between the vectors $\mathbf{K}$ and $\mathbf{K}_0$, represents the first diffraction phenomenon, which is observed for setting angles equal to $\varphi_0$ and $\mu$ (usually $\mu = \theta$). Let us assume that the reciprocal vector $\mathbf{H} = h\mathbf{a}^* + k\mathbf{b}^* + l\mathbf{c}^*$, observed for setting angles $\varphi_0 + \beta$ and $\nu$, represents the next diffraction. The vector components of $\mathbf{H}$, parallel and normal to $\mathbf{P}$, are denoted by $\mathbf{H}_p$ and $\mathbf{H}_n$, respectively. For a given wavelength, the lengths $P, H, H_p, H_n$ of respective vectors $\mathbf{P}$, $\mathbf{H}$, $\mathbf{H}_p, \mathbf{H}_n$ are functions of lattice parameters and diffraction indices.

The task is to find the relationship between the difference $\beta$ of angles of rotation (or between two values of the setting angles, $\beta$ and $\nu$) and the lengths of the reciprocal vectors. The following relations result from Fig. 5.3.3.6(*b*):

$$\cos\beta = \frac{(C'A')^2 + H_n^2 - R'^2}{2H_n(C'A')},$$
$$(C'A')^2 = R^2 - P^2/4,$$
$$H_p = P/2 - R\sin\nu,$$
$$R' = R\cos\nu.$$

Taking these into consideration, and remembering that $H^2 = H_p^2 + H_n^2$, we finally obtain

$$\cos\beta = \frac{H^2 - H_pP}{2H_n(R^2 - P^2/4)^{1/2}}. \qquad (5.3.3.34)$$

Since $\cos(-\beta) = \cos\beta$, the appearance of successive reflections does not depend on the direction of rotation. A detailed discussion of (5.3.3.34) is given by Cole, Chambers & Dunn (1962) and Chang (1984). When preliminary values of the lattice parameters are known, (5.3.3.34) can be applied for indexing multiple-diffraction patterns. A computer program (Rossmanith, 1985) can be very useful in rather complicated calculations and in the graphical representation of the multiple-diffraction

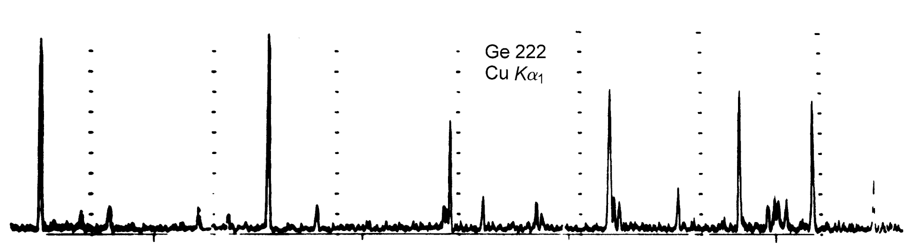



Fig. 5.3.3.7. The multiple-diffraction pattern at the 222 position in germanium (Cole, Chambers & Dunn, 1962).





references