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in which n is the number of the layer line and ν is the angle between the directions of the primary and diffracted beams.

The angle ν is determined from the measurement of the distance l_n between two lines corresponding to the same layer number n from the equation

$$\tan\nu = l_n/R, \quad (5.3.2.2)$$

where R is the camera radius.

All the lattice parameters may be determined from separate photographs made for rotations of the crystal along different rotation axes, *i.e.* the system axis, plane and spatial diagonals (Evans & Lonsdale, 1959), without indexing the photographs. In practice, however, this method is rarely used alone and is most often applied together with other photographic methods (for example, the Weissenberg method), but it is a useful preliminary stage for other methods. In particular, the length of a unit-cell vector may be directly determined if the rotation axis coincides with this vector.

Advantages of this method are:

(*a*) simple equipment (only rotation of the crystal is required, since the film is stationary);

(*b*) immediate determination of direct-cell parameters (photographs obtained with other cameras afford information about reciprocal-lattice parameters only);

(*c*) indexing of the photographs is unnecessary.

Drawbacks of the method are:

(*a*) poor precision and accuracy of the measurement ($|\delta d|/d \approx 10^{-2}$);

(*b*) small amount of information from a single photograph (one parameter only);

(*c*) necessity of taking several photographs in the case of a lower-symmetry system if this method is the only one used.

5.3.2.3.2. *Moving-film methods*

A two-dimensional picture of a reciprocal cell from one photograph can be obtained by the methods in which rotation of the crystal is accompanied by movement of the film, as in the Weissenberg, the de Jong–Bouman, and the Buerger precession techniques. These methods give greater precision ($|\delta d|/d \approx 10^{-4}$) than the previous one (§5.3.2.3.1).

The advantages of the Weissenberg method in relation to the other two are:

(*a*) a simpler camera;

(*b*) a larger range of reciprocal-lattice points recorded on one photograph (larger range of θ angles, up to 90° for the zero layer).

On the other hand, the disadvantage, in contrast to the de Jong–Bouman and the Buerger precession methods, is that it gives deformed pictures of the reciprocal lattice. This is not a fundamental problem, especially now that computer programs that calculate lattice parameters and draw the lattice are available (Luger, 1980). In lattice-parameter measurements, both the zero-layer Weissenberg photographs and the higher-layer ones are used. The latter can be made both by the normal-beam method and by the preferable equi-inclination method. Photographs in the de Jong–Bouman and precession methods give undeformed pictures of the reciprocal lattice, but afford less information about it than do Weissenberg photographs.

5.3.2.3.3. *Combined methods*

The most effective photographic method of lattice-parameter measurement is a combination of two techniques (Buerger, 1942; Luger, 1980), which makes it possible to obtain a *three-dimensional picture* of the reciprocal lattice; for example: the rotation method with the Weissenberg (lower accuracy); or the precession (or the Weissenberg) method with the de Jong–Bouman (higher accuracy).

A suitable combination of the two methods will determine all the lattice parameters, even for monoclinic and triclinic systems, *from one crystal mounting*. This problem has been discussed and resolved by Buerger (1942, pp. 388–390), Hulme (1966), and Hebert (1978). Wölfel (1971) has constructed a special instrument for this task, being a combination of a de Jong–Bouman and a precession camera.

5.3.2.3.4. *Accurate and precise lattice-parameter determinations*

To measure with a precision and an accuracy better than is possible in routine photographic methods, additional work has to be performed. The first methods allowing precise measurement of lattice parameters were photographic powder methods (Parrish & Wilson, 1959). Special single-crystal methods with photographic recording to realize this task (earlier papers are reviewed by Woolfson, 1970, Chap. 9) combine elements of basic single-crystal methods (presented in §§5.3.2.3.1 and 5.3.2.3.2) with ideas more often met in powder methods (asymmetric film mounting). A similar treatment of some systematic errors (extrapolation) is met in both powder and single-crystal methods.

(i) The relative accuracy $\Delta I/I$ of the identity period I in the rotating-crystal method, estimated by differentiation of formula (5.3.2.1), is given by

$$\Delta I/I = -\cot\nu\Delta\nu. \quad (5.3.2.3)$$

This formula shows that the highest accuracy is obtained for ν tending to 90°. Since reflections with large values of ν are difficult to record in commonly used cameras, a special camera may be used for this task, in which a flat film is placed perpendicular to the rotation axis, or a different one, whose axis coincides with the primary beam (Umansky, 1960). The accuracy achieved with these improvements is still no better than 5 parts in 10^3.

(ii) The asymmetric film mounting proposed by Straumanis & Ieviņš (1940) in the case of powder cameras can also be used in a simple oscillating camera (Farquhar & Lipson, 1946). In particular, this idea can be realized in a precision Debye–Scherrer camera adapted to single-crystal measurements by mounting in it a goniometer head (Popović, 1974). The Straumanis mounting allows the recording of the high-angle reflections close together on the film, thus reducing the effect of film shrinkage and making it possible to measure the effective camera radius.

(iii) Sometimes, to eliminate systematic errors (uncertainty of the camera radius), the separations resulting from the wavelength differences of the $K\alpha_1$ and $K\alpha_2$ doublet are measured rather than the absolute distances on the film (Main & Woolfson, 1963; Alcock & Sheldrick, 1967). The first reference related to the zero-layer normal-beam photograph, the second to higher layer lines (in the equi-inclination method also) and oscillation photographs.

(iv) Systematic errors connected with film shrinkage can also be eliminated by means of the *ratio method*, introduced by Černohorský (1960) for powder samples and adapted by Polcarová & Zůra (1977) for single crystals. In this method, pairs of reflections that differ from one another in wavelength and/or in *hkl* indices are used and the ratio of the two diameters of the diffraction rings corresponding to these reflections is taken into account. The accuracy of the method is about 1 part in 10^4

references