

5. DETERMINATION OF LATTICE PARAMETERS

pattern. Formula (5.3.3.34), since $C'A' = R \cos \mu$, can be presented in another form:

$$R = \frac{H^2 - H_p P}{2H_n \cos \mu \cos \beta}, \quad (5.3.3.35)$$

where two setting angles, μ and β , are taken into account. When the indices are known, both (5.3.3.34) and (5.3.3.35) can be used for the determination or refinement of lattice parameters.

Another analytical method for indexing multiple-diffraction patterns, based on the determination of the Lorentz point, has been described by Kshevetsky, Mikhailyuk, Ostapovich, Polyak, Remenyuk & Fomin (1979).

Formulae (5.3.3.34) and (5.3.3.35) are valid for all crystal systems. In practice, however, the rather complicated method is used mainly for cubic crystals, and a special approach proved to be needed in order to adapt the method to other (rectangular) systems (Kshevetsky, Mikhalchenko, Stetsko & Shelud'ko, 1985). In the case of a cubic lattice, it is convenient to substitute

$$R = a/\lambda \quad (5.3.3.35a)$$

into (5.3.3.34) and (5.3.3.35) rather than $R = 1/\lambda$ used in the general case, so that the lengths of the reciprocal vectors, being now functions of the indices only, are:

$$P = (h_0^2 + k_0^2 + l_0^2)^{1/2}, \quad (5.3.3.35b)$$

$$H = (h^2 + k^2 + l^2)^{1/2}, \quad (5.3.3.35c)$$

$$H_p P = \mathbf{HP} = h_0 h + k_0 k + l_0 l. \quad (5.3.3.35d)$$

The lengths of the components of \mathbf{H} can be determined from (5.3.3.35b,c,d) taking $H_p = H_p P/P$ and $H_n = (H^2 - H_p^2)^{1/2}$. After introducing the alterations [equation (5.3.3.35a) and the resulting equations (5.3.3.35b,c,d)], (5.3.3.35) now describes a simple dependence between the ratio a/λ , the indices, and the setting angles.

The accuracy of the lattice-parameter determination resulting from (5.3.3.35) in the cubic case can be assumed to be:

$$\frac{\Delta a}{a} = \tan \mu \Delta \mu + \tan \beta \Delta \beta; \quad (5.3.3.36)$$

this thus depends on the values of the setting angles β , μ and their accuracies $\Delta \beta$, $\Delta \mu$. The latter depend on various systematic errors.

Since the differences between the two angular settings at which a given set of planes diffracts are measured rather than their absolute values, the systematic errors due to absorption, specimen displacement, and zero-setting are eliminated. In contrast, errors due to vertical divergence, refraction and the change of wavelength of the incident radiation (when it enters the crystal), alignment, and dynamical effects should be taken into account. In the case described by Post (1975), when a fine focus (effective size 0.4×0.5 mm) and collimation limiting the beam divergence to $2'$ were used, the vertical divergence causing the relative error in d of about 5×10^{-8} could be ignored.

The errors due to the real structure (inhomogeneity, mosaicity and internal stress) were discussed by Kshevetsky *et al.* (1979).

The accuracy possible by this method (from 1 to 4 parts in 10^6) is comparable with that obtained with the Bond (1960) method. The advantages of this method from the point of view of lattice-parameter determination are as follows:

(a) a large number of reflections can be measured without realigning or removing the crystal;

(b) all the lattice parameters can be determined and not only one, as in the Bond (1960) method;

(c) the narrow diffraction profiles can be located with very high accuracy and precision;

(d) the arrangement makes it possible to remove some systematic errors;

(e) the high accuracy resulting from (a)–(d), which is comparable with that obtained by means of the Bond (1960) method;

(f) the high precision that results from (a) and (c).

A disadvantage, on the other hand, is the complicated interpretation (indexing) of multiple-diffraction patterns, so that this method is less popular than the Bond (1960) method.

The Post (1975) method has been applied to the accurate lattice-parameter determination of germanium, silicon, and diamond single crystals (Hom, Kiszénick & Post, 1975).

5.3.3.7. Multiple-crystal – pseudo-non-dispersive techniques

5.3.3.7.1. Double-crystal spectrometers

Detailed information concerning the double-crystal spectrometer, which consists of two crystals successively diffracting the X-rays, can be found in James (1967, pp. 306–318), Compton & Allison (1935), and Azároff (1974). This device, usually used for wavelength determination, may also be applied to lattice-parameter determination, if the wavelength is accurately known. The principle of the device is shown in Fig. 5.3.3.8. The first crystal, the monochromator, diffracts the primary beam in the direction defined by the Bragg law for a given set of planes, so that the resulting beam is narrow and parallel. It can thus be considered to be both a collimator (or an additional collimator, if the primary beam has already been collimated) and a wavelength filter. The final profile $h(\theta)$, obtained as a result of the second diffraction by the specimen when the first crystal remains stationary and the second is rotated, is narrower than that which would be obtained with only one crystal. The final crystal profile $h_c(\theta)$ [cf. equation (5.3.1.6)] is due to both crystals, which, if it is assumed that they are cut from the same block, can be described by the autocorrelation function (Hart, 1981):

$$h_c(\theta) = K \int_{-\infty}^{\infty} R(\theta') R(\theta' - \theta) d\theta', \quad (5.3.3.37)$$

where $R(\theta)$ is an individual reflectivity function of one crystal and K is a coefficient of proportionality. Its half-width is 1.4 times larger than that related to only one crystal. In spite of this, the recorded profile can be as narrow as, for example, $2.6''$ (Godwod, Kowalczyk & Szmíd, 1974), since the profile due to the wavelength $h_i(\theta)$, modified by the first crystal, is extremely narrow. Additional advantages of the diffraction profile are: its symmetry, because $h_c(\theta)$ is symmetric as an effect of autocorrelation, and smoothness, as an effect of additional integration. The profile can thus be located with very high accuracy and precision.

When there is a small difference in the two lattice spacings, so that one has a value d and the other $d + \delta d$, if $\delta \lambda / \lambda$ is small enough, it can be assumed that the profile does not alter in shape but in its peak position [cf. §5.3.3.4.3.3, paragraph (3)]. If for two identical crystals this were located at θ_0 , the peak position

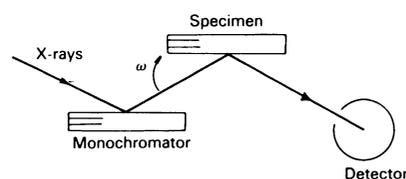


Fig. 5.3.3.8. Schematic representation of the double-crystal spectrometer.