5. DETERMINATION OF LATTICE PARAMETERS

traditional methods, in which both the wavelengths and the lattice parameters are fixed, and it is difficult to find a suitable combination of their values.

The principle of the method presented by Okazaki & Kawaminami (1973*a*) is shown in Fig. 5.3.3.10. The first crystal (the specimen to be measured) remains fixed during a single measurement, the second (the analyser) is mounted on the goniometer of an X-ray diffractometer and can be operated with either an ω or a θ–2θ scan. As diffraction phenomena appear for both the specimen and the analyser (in general of different materials) whose interplanar spacings are equal to d_s and d_A, respectively, the following relation results from Bragg's law:

$$d_s \sin \theta_s = d_A \sin \theta_A, \qquad (5.3.3.38)$$

where θ_s and θ_A are the respective Bragg angles. Since d_A and θ_s are kept constant, a change in d_s as a function of temperature is determined from a change in θ_A. The relative error $\delta d/d$ resulting from (5.3.3.38) with $\theta_A \approx 90°$ is

$$\frac{\delta d_s}{d_s} = \cot \theta_A \, \delta\theta_A = \tan (\pi/2 - \theta_A) \delta\theta_A$$

$$\approx (\pi/2 - \theta_A) \delta\theta_A. \qquad (5.3.3.39)$$

The method initiated by Okazaki & Kawaminami (1973*a*) has been developed by Okazaki & Ohama (1979), who constructed the special diffractometer HADOX (the positions of the specimen and the analyser were interchanged) and discussed systematic errors. Precision as high as 1 part in 10^7 was reported. Examples of the application of such an arrangement for measuring the temperature dependence of lattice parameters were given by Okazaki & Kawaminami (1973*b*) and Ohama, Sakashita & Okazaki (1979). Various versions of the HADOX diffractometer are still reported. By introducing two slits (Soejima, Tomonoga, Onitsuka & Okazaki, 1991) – one to limit the area of the specimen surface to be examined and the other to define the resolution of 2θ – it is possible to combine ω and 2θ scans and obtain a two-dimensional intensity distribution in the plane parallel to the plane of the diffractometer, and to determine the temperature dependence of lattice parameters on a selected area of the specimen (avoiding the effects of the surroundings). The HADOX diffractometer may work with both a rotating-anode high-power X-ray source (examples reported above) and a sealed-tube X-ray source. In the latter case (Irie, Koshiji & Okazaki, 1989), to increase the efficiency of the X-ray tube, the distance between the X-ray source and the first crystal has been shortened by a factor of five. As is implied by (5.3.3.39), one can increase the relative precision of the method by using the analyser angle close to $\pi/2$. This idea has been realized by Okazaki & Soejima (2001), who achieved the relative accuracy of determination of lattice-parameter changes as high as 1 part in 10^9–10^{10} by extending the Bragg angle from 78° (previous versions) to 89.99° and by elimination of systematic errors due to crystal tilt, crystal displacement, temperature effects and radiation damage.

An original method for the measurement of lateral lattice-parameter variation by means of a double-crystal arrangement
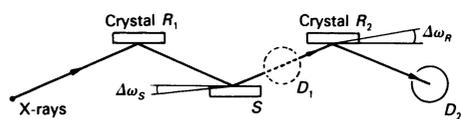
with an oscillating slit was proposed by Korytár (1984). This method permitted simultaneous recording of two rocking curves from two locations on a crystal. Precision of 3 parts in 10^7 was reported. The method has been applied for the measurement of growth striations in silicon.

The main disadvantage of double-crystal spectrometers, in their basic form (Fig. 5.3.3.8), is that they cannot be used for measurements on an absolute scale. Combination of the double-crystal arrangement with the system proposed by Bond (1960) makes it possible to recover the origin of the angular scale and thus such an absolute measurement, but the reported precision is rather moderate.

There are two other ways to overcome this difficulty in pseudo-non-dispersive methods: addition either of a third crystal (more accurately, a third reflection) (§5.3.3.7.2) or of a second source (a second beam) (§5.3.3.7.3). Such arrangements require additional detectors. Combinations of both techniques are also available (§5.3.3.7.4).

5.3.3.7.2. *Triple-crystal spectrometers*

Higher precision than that obtained with the double-crystal arrangements (§5.3.3.7.1) can be achieved by means of triple-crystal diffractometers. Arrangements specially designed for the determination of lattice-parameter changes are described by Buschert (1965) and Skupov & Uspeckaya (1975), and reviewed by Hart (1981).

The principle of the triple-axis spectrometer is shown in Fig. 5.3.3.11. The arrangement consists of one standard crystal S, ultimately replaced by the sample under investigation, and two reference crystals R_1 and R_2. The principle of the measurement is as follows. First, the crystals S, R_1, and R_2 are set to their diffraction (peak) positions using two detectors D_1 and D_2. Then the standard crystal S is replaced by the sample and the new peak position is found by means of D_1 when the sample is turned from its original position to its reflecting position. The angle of rotation of the sample $\Delta\omega_s$ depends on the lattice-parameter difference Δd between the sample and the standard. The relation is given by (Hart, 1981)

$$\Delta\omega_s = -\tan\theta \, \Delta d/d. \qquad (5.3.3.40)$$

Next, the second reference crystal R_2 is turned through the angle $\Delta\omega_R$ to its diffracting position, the intensity being controlled with the second detector D_2. From the geometry of the arrangement,

$$\Delta\omega_R = 2\Delta\omega_s. \qquad (5.3.3.41)$$

Because the origin of the ω_s scale is lost during the crystal exchange, this second angle of rotation ($\Delta\omega_R$) is used to determine Δd rather than the first one ($\Delta\omega_s$), by using (5.3.3.41) and (5.3.3.40).

The diffraction profiles observed in the second detector, described by Hart (1981),

$$h(\theta)_R = \int_{-\infty}^{\infty} R^2(\theta') R(\theta' - \theta) \, \mathrm{d}\theta, \qquad (5.3.3.42)$$

are not symmetric but can be as narrow as 0.1–1″, so that a precision of 2 parts in 10^8 is possible.

The main experimental problem here is to adjust the tilts of the crystals. The errors resulting both from the crystal tilts and from the vertical divergence were discussed by Skupov & Uspeckaya (1975).

Triple-crystal spectrometers are often applied as lattice-spacing comparators, when very small changes of lattice parameters ($10^{-8} \leq |\Delta d|/d \leq 10^{-6}$) are to be detected, in

Fig. 5.3.3.11. Schematic representation of the triple-crystal spectrometer developed by Buschert (1965) (after Hart, 1981).