

## 5. DETERMINATION OF LATTICE PARAMETERS

The application of the double-beam technique makes it possible to realize a triple-reflection scheme for comparing lattice parameters on the basis of a double-axis spectrometer. The arrangement proposed by Ando, Bailey & Hart (1978), shown in Fig. 5.3.3.13, consists of a sample and a reference crystal, which are made from the same material but differ in purity (or strain, stoichiometry, vacancy concentration, *etc.*). The angular difference  $\Delta\theta$  in the Bragg angles of the sample and the reference crystal,  $\theta_S$  and  $\theta_R$ , respectively,

$$\Delta\theta = \theta_S - \theta_R, \quad (5.3.3.45a)$$

is measured as the sample angle  $\Delta\omega$  between the double-reflected peak  $D$  and the triply diffracted peak  $T$ :

$$\Delta\theta = \Delta\omega, \quad (5.3.3.45b)$$

$$\Delta\omega = \omega_D - \omega_T. \quad (5.3.3.45c)$$

Assuming that  $\Delta\theta$  is entirely due to changes  $\Delta d$  in atomic spacings, the authors use the following relation for determination of the latter:

$$\Delta d/d = -\cot\theta\Delta\theta. \quad (5.3.3.46)$$

The experimental requirements are simple and inexpensive, owing to simple shapes of both the reference crystal and the sample crystal, so that the measurement can be made quickly. By combining the two reference crystals into a single monolithic reference crystal, excellent stability, difficult to achieve with triple-axis arrangements (*cf.* §5.3.3.7.2), is obtained at the same time. The disadvantage of the method is that it covers a smaller range of lattice parameters than the other double-beam methods (Hart, 1969; Larson, 1974) described in §5.3.3.7.3. A new version of the double-crystal triple-reflection scheme (Häusermann & Hart, 1990) allows one to achieve a precision of 1 part in  $10^8$  in 2 min of measurement time, which includes the data analysis; 30 min are needed to change the sample. Errors due to the crystal tilt and thermal drifts are considered.

Another example of the triple-reflection scheme realized by means of the double-beam technique has been presented by Kovalchuk, Kovev & Pinsker (1975), who realized the triple-crystal arrangement on the basis of a double-crystal spectrometer by parallel mounting of the two crystals to be compared (the sample and the reference crystal) on one common axis. The advantage of this system is that Bragg angles as high as  $80^\circ$  are available. The device can be applied in studies of the real structure of a single crystal.

High-sensitivity ( $\Delta d/d$  up to  $\pm 3 \times 10^{-8}$ ) lattice-parameter-comparison measurement over a wide range of temperatures can

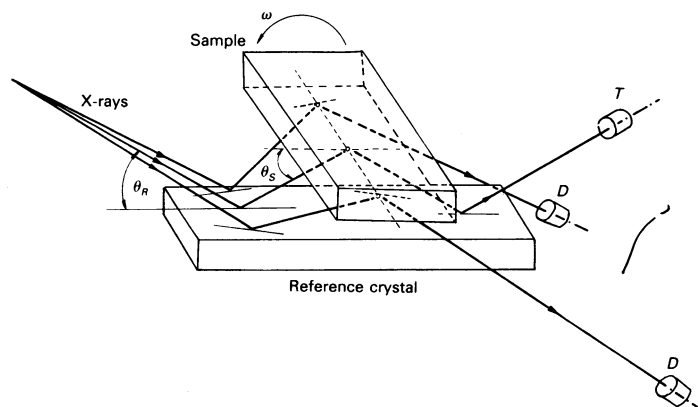


Fig. 5.3.3.13. The double-axis lattice-spacing comparator of Ando, Bailey & Hart (1978); a triple-diffracted beam is used.

be performed by means of the triple-crystal (more accurately, triple-axis) X-ray spectrometer realized by Buschert, Pace, Inzaghi & Merlini (1980). The arrangement (Fig. 5.3.3.14) consists of four crystals. The first is used for obtaining a very wide but extremely parallel exit beam, which is incident on both the standard crystal  $S$  and an unknown crystal  $X$ , placed side by side on a common axis in the cryostat. The reflected beams from  $S$  and  $X$  are recorded by partially transmitting detectors  $DA_2$  and  $DB_2$ , so that the beams reflect from the third crystal and are detected by the counters  $DA_3$  and  $DB_3$ . There is a small, sensitive, angle adjustment to rotate the crystal  $X$  with respect to the standard  $S$  and it is used to bring the peaks of  $S$  and  $X$  into approximate coincidence. The angular difference in the peak positions on the third axis is used for determination of lattice-parameter changes from (5.3.3.46), so that

$$\Delta\theta = \Delta\theta_3/2 - \Delta\theta_2, \quad (5.3.3.47)$$

where  $\Delta\theta_2$  and  $\Delta\theta_3$  are the differences in peak positions at axes (2) and (3), respectively. The device was used, for example, to study the effect of isotope concentration on the lattice parameter of germanium perfect crystals (Buschert, Merlini, Pace, Rodriguez & Grimsditch, 1988). The measured differences in the lattice parameter, of the order of 1 part in  $10^5$ , were compared with those evaluated theoretically, and a very good agreement was obtained.

Another variant of a multiple-beam arrangement, based on a triple-crystal spectrometer, was proposed by Kubena & Holý (1988). The authors compared the distances of lattice planes in a direction perpendicular to the surface of the sample while studying the growth striations. One well collimated and monochromated beam coming from the first crystal was directed into the sample, and then two beams – one transmitted and one diffracted in the sample – diffracted in the reference crystal. Intensities of the diffracted beams were measured by two detectors. The difference of lattice spacings of the sample and the reference crystal was determined from the difference in positions of respective peaks. The accuracy of the lattice-spacing comparison of 2 parts in  $10^7$  and the precision of 1 part in  $10^7$  were obtained.

A four-crystal six-reflection diffractometer (Fewster, 1989) was built to study crystals distorted by epitaxy and defects in nearly perfect crystals. Fig. 5.3.3.15 is a schematic diagram of this device. The two-crystal four-reflection Bartels monochromator (Bartels, 1983) defines a narrow reflectivity profile. The analyser selects the angular range diffracted from the sample. The device may be used for recording both near-perfect rocking curves from distorted crystals (when rotations of the sample and the analyser are coupled) and a diffraction-space map for studying the diffuse scattering (when the two rotations are

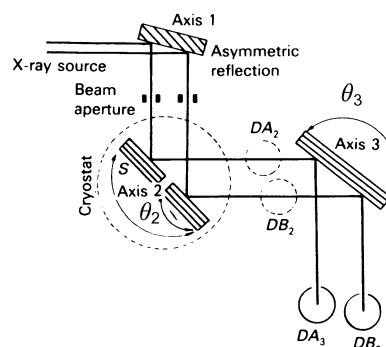


Fig. 5.3.3.14. Schematic representation of the double-beam triple-crystal spectrometer of Buschert *et al.* (1980).