

## 5.3. X-RAY DIFFRACTION METHODS: SINGLE CRYSTAL

shifts to  $\theta_0 - \tan \theta \delta d/d$ . The measurement of this shift rather than the absolute position of the rocking curve is the basis of all the double-crystal methods. An example of the application of a double-crystal spectrometer with photographic recording has been given in §5.3.2.3.5 (Bearden & Henins, 1965).

The basic requirements that should be fulfilled to make the most of the double-crystal spectrometer are: limitation of the primary beam by means of a collimator, parallelism of the two axes [precision as high as  $1''$  obtained by Godwod, Kowalczyk & Szmíd (1974)], and high thermal stability (0.1 K; Godwod, Kowalczyk & Szmíd, 1974). Alignment procedure, errors, and corrections valid for the double-crystal spectrometer have been considered by Bearden & Thomsen (1971).

The double-crystal diffractometer, because of the small width of the diffraction profile, is a very suitable tool for local measurements of lattice-parameter differences, for example between an epitaxial layer and its substrate. Hart & Lloyd (1975) carried out such a measurement on a standard single-axis diffractometer (APEX) to which a simple second axis, goniometer head, and detector were added (Fig. 5.3.3.9). The diffracted beam was recorded simultaneously by three detectors. A *symmetric arrangement* with two detectors,  $D_1$  and  $D_2$ , with no layer present, makes possible the determination of the absolute value of the lattice parameter of the substrate, as in the Bond (1960) method. The third detector makes it possible to record the double-crystal rocking curve, which usually fully resolves the layer and substrate profiles. The changes in the lattice parameter between the two components can be used for determination of strain (at 1 part in  $10^4$ ).

The very important advantage of this method, from the point of view of local measurements, is that single- or double-crystal diffraction can be selected, simultaneously if needed, on exactly the same specimen area. Other examples of strain measurements by means of a double-crystal spectrometer are given by Takano & Maki (1972), who measured lattice strain due to oxygen diffusing into a silicon single crystal; by Fukahara & Takano (1977), who compared experimental rocking curves and theoretical ones computed within the frame of the dynamical theory; and Barla, Herino, Bomchil & Pfister (1984), who examined the elastic properties of silicon.

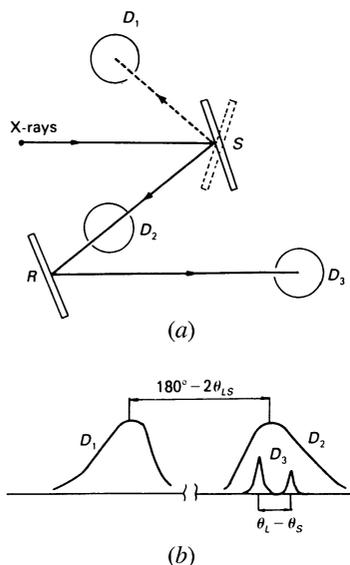


Fig. 5.3.3.9. Schematic representation of the double-crystal arrangement of Hart & Lloyd (1975) for the examination of epitaxial layers. (a) Experimental set-up. (b) Diffraction profiles recorded by detectors  $D_1$ ,  $D_2$ , and  $D_3$ .

The standard double-crystal technique does not allow determination of relatively small strains, *i.e.* ones that affect the lattice parameter by, for example, less than 2–3 parts in  $10^5$ , as in the case of (004) Si reflection and Cu  $K\alpha$  radiation. To overcome this difficulty, Zolotoyabko, Sander, Komem & Kantor (1993) propose a new method that combines double-crystal X-ray diffraction with *high-frequency ultrasonic excitation*. Since ultrasound has a wavelength a little less than the X-ray excitation length, it affects the diffraction profile close to the Bragg position and so permits the detection of very small profile broadenings caused by lattice distortions. With this method, lattice distortion as small as 5 parts in  $10^6$  can be measured.

As has been shown in the case of the device used by Hart & Lloyd (1975), the symmetric arrangement due to Bond (1960) proves to be very useful when the double-crystal spectrometer is to be used for absolute lattice-parameter determination, since such an arrangement combines the high precision and sensitivity of a double-crystal spectrometer with the high absolute accuracy of the Bond method. Other examples of a similar idea are presented by Kurbatov, Zubenko & Umansky (1972), who report measurements of the thermal expansion of silicon; Godwod, Kowalczyk & Szmíd (1974), who also discuss the theoretical basis of their arrangement; Ridou, Rousseau & Freund (1977), who examine a phase transition; Sasvári & Zsoldos (1980), and Fewster (1982). The latter two papers are concerned with epitaxial layers. A rapid method is proposed by Sasvári & Zsoldos (1980) for deconvoluting the overlapping peaks due to the layer and the substrate. A particular feature of the arrangement proposed in the first of these papers (Kurbatov, Zubenko & Umansky, 1972) is the use of a germanium-crystal monochromator with anomalous transmission, to obtain a nearly parallel primary beam (the horizontal divergence is  $28''$  and the vertical  $14''$ ).

The error analyses given by Godwod, Kowalczyk & Szmíd (1974) and Sasvári & Zsoldos (1980) show that systematic errors due to eccentricity, absorption, and zero position are eliminated experimentally, owing to the symmetric arrangement, as in the Bond (1960) method. In contrast, the errors due to crystal tilt, refraction and the Lorentz-polarization factor [their uncertainties in lattice parameters, as evaluated by Sasvári & Zsoldos (1980), are  $10^{-6}$  Å each], axial divergence ( $2 \times 10^{-6}$  Å), angle reading ( $10^{-4}$  Å), and instrument correction and calculations (each to  $5 \times 10^{-5}$  Å) should be taken into account. The effect of absorption, discussed by Kurbatov, Zubenko & Umansky (1972), proved to be negligible. The final accuracy achieved for silicon single crystals by Godwod, Kowalczyk & Szmíd (1974) is comparable with that obtained by Bond (1960).

A specific group of double-crystal arrangements is formed by those in which *white X-radiation* is used instead of characteristic. Such an arrangement makes possible very large values of the Bragg angle (larger than about  $80^\circ$ ), which increases the accuracy, precision, and sensitivity of measurement of the lattice parameters and their change with change of temperature. This task is rather difficult to realize by means of

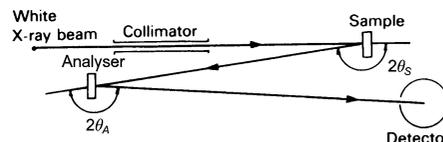


Fig. 5.3.3.10. Schematic representation of the double-crystal arrangement of Okazaki & Kawaminami (1973a); white incident X-rays are used.