particular for the examination of a correlation between lattice parameter and the dopant or impurity concentration (Baker, Tucker, Moyer & Buschert, 1968). Such an arrangement can also be a very suitable tool in deformation studies, since it allows the separation of the effect of deformation on the Bragg angle from that due to lattice-parameter change (Skupov & Uspeckaya, 1975).

The basis of the accurate lattice-parameter comparison proposed by Bowen & Tanner (1995) is the use of a high-purity silicon standard (*cf.* §5.3.3.9 below) with a well known lattice parameter. To compensate an error that may result from a slight misalignment of crystal planes in relation to the axes of the instrument, the authors recommend a twofold measurement of the diffraction-peak position of the reference crystal (for a given diffraction position and after rotating the specimen holder through 180° about the axis normal to its surface) and a similar twofold measurement of the diffraction-peak position of the sample – after replacing the reference crystal by the sample. The mean positions of the reference crystal and of the sample are used in calculations of the Bragg-angle difference and then of the unknown interplanar spacing. The method uses a standard double-crystal diffractometer fitted with a monochromator (therefore, a third crystal), which provides a well defined wavelength, and with a specimen rotation stage. The measurement is accompanied by a detailed error analysis. The accuracy of absolute lattice-parameter determination as high as a few tens of parts in $10^6$, and a much greater relative sensitivity are reported.

By combining a triple-axis spectrometer with the Bond (1960) method, the device can be used for absolute measurements (Pick, Bickmann, Pofahl, Zwoll & Wenzl, 1977). The device described in the latter paper is an automatic triple-crystal diffractometer that permits intensity measurement to be made in any direction in reciprocal space in the diffraction plane with step sizes down to $0.01''$ and therefore can be used for very precise measurements [see also §5.3.3.4.3.2, paragraph (5)].

### 5.3.3.7.3. *Multiple-beam methods*

The other possibility of recovering the crystal-angle scale in differential measurements with a double-crystal spectrometer (*cf.* §§5.3.3.7.1, 5.3.3.7.2) is to obtain reflections from two crystal planes [for example, from $(hkl)$ and $(\bar{h}\bar{k}l)$ planes] by means of a double-beam arrangement and to measure them simultaneously.

The second X-ray beam may come from an additional X-ray source (Hart, 1969) or may be formed from a single X-ray source by using a beam-splitting crystal (Hart, 1969, second method; Larson, 1974; Cembali, Fabri, Servidori, Zani, Basile, Cavagnero, Bergamin & Zosi, 1992). In particular, two beams with different wavelengths ($K\alpha_1, K\beta_1$) separated with a slit system can be used for this purpose (Kishino, 1973, second technique). The principle of the double-beam method is shown in Fig. 5.3.3.12. The beams are directed at the first crystal (the reference crystal) so that the Bragg condition is simultaneously fulfilled for both beams, and they then diffract from the second crystal (the specimen). As the second crystal is rotated, a double-crystal diffraction profile is recorded first in one detector and then in the other. The angle $\Delta\theta$ of crystal rotation between the two rocking curves is given by (Baker & Hart, 1975):

$$\Delta\theta = (\theta_1 - \theta_2) = \tan\theta\,\Delta d/d. \qquad (5.3.3.43)$$

This formula leads to the lattice-parameter changes $\Delta d$.

A double-beam diffractometer can be used for the examination of variations in lattice parameters of about 10 parts in $10^6$ within a sample in a given direction. An example was reported by Baker, Hart, Halliwell & Heckingbottom (1976), who used Larson's (1974) arrangement for this task.

The highest reported sensitivity (1 part in $10^9$) can be achieved in the double-source double-crystal X-ray spectrometer proposed by Buschert, Meyer, Stuckey Kauffman & Gotwals (1983). The device can be used for the investigation of small concentrations of dopants and defects.

The method can also be applied for the absolute determination of a lattice parameter, if that of the reference crystal is accurately known and the difference between the two parameters is sufficiently small. Baker & Hart (1975), using multiple-beam X-ray diffractometry (Hart, 1969, first technique), determined the $d$ spacing of the 800 reflection in germanium by comparing it with the $d$ spacing of the 355 reflection in silicon. The latter had been previously determined by optical and X-ray interferometry (Deslattes & Henins, 1973; the method is presented in Subsection 5.3.3.8).

In the case of two different wavelengths and diffraction from two different diffraction planes $(h_1k_1l_1)$ and $(h_2k_2l_2)$, the lattice parameter $a_0$ of a cubic crystal can be determined using the formula (Kishino, 1973)

$$a_0 = \tfrac{1}{2}\{(L\lambda_1)^2 + [(M\lambda_2 - L\lambda_1\cos\theta_{1-2})/\sin\theta_{1-2}]^2\}^{1/2}, \qquad (5.3.3.44)$$

where $L = (h_1^2 + k_1^2 + l_1^2)^{1/2}$, $M = (h_2^2 + k_2^2 + l_2^2)^{1/2}$, and $\theta_{1-2}$ is the difference between the two Bragg angles for the specimen crystal, estimated from the measurement of $\Delta\theta = |\theta_{1-2} - \theta'_{1-2}|$ if the difference $\theta'_{1-2}$ for the first (reference crystal) is known beforehand. The idea of Kishino was modified by Fukumori, Futagami & Matsunaga (1982) and Fukumori & Futagami (1988), who used the Cu $K\alpha$ doublet instead of $K\alpha_1$ and $K\beta_1$ radiation. Owing to the change, they could use only one detector (Kishino's original method needs two detectors), but a special approach is sometimes needed to resolve two peaks that relate to the components of the doublet. A similar problem of separation of two peaks (recorded by two detectors) is reported by Cembali *et al.* (1992). By introducing a computer simulation of the reflecting curves (using a convolution model), the authors managed to determine the separation with an error of $0.01''$ and to achieve a precision of some parts in $10^7$. The same precision is reported by Fukumori, Imai, Hasegawa & Akashi (1997), who introduced a precise positioning device and a position-sensitive proportional counter to their instrument.

As in the other multiple-crystal methods, the most important experimental problem is accurate crystal setting. Larson (1974), as a result of detailed analysis, gave the dependence between the angular separation of two peaks and angles characterizing misalignment of the first and second crystals.

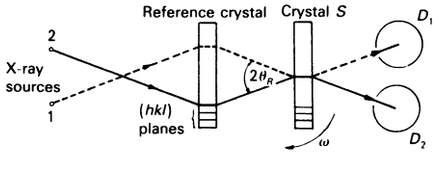


Fig. 5.3.3.12. Schematic representation of the double-beam comparator of Hart (1969).

### 5.3.3.7.4. *Combined methods*

The idea of multiple-beam measurement (§5.3.3.7.3) can be applied to other arrangements that combine the features of the double-beam comparator with those of the triple-crystal spectrometer; there are additional advantages in such a system.





references