The above experiment was a turning point in accurate measurements of both wavelengths and lattice parameters. Owing to the idea of Deslattes & Henins, it became possible to determine the wavelength in nanometres rather than in troublesome XU or Å* units (*cf.* §4.2.1.1.1). However, the results obtained and the method itself needed verification and some adjustments. These were performed by another group of experimenters with a similar but different measuring device (Becker, Seyfried & Siegert, 1982, and references therein; Siegert, Becker & Seyfried, 1984).

5.3.3.9. *Lattice-parameter and wavelength standards*

An extended series of measurements performed by means of the optical and X-ray interferometry (*cf.* §5.3.3.8) led, among other things, to evaluation of the lattice spacing of a highly perfect silicon sample WASO 4.2.A (Becker *et al.*, 1981). Such silicon samples may be used as reference crystals in successive lattice-spacing comparison measurements – with a double-source double-crystal spectrometer (Windisch & Becker, 1990), for example. The latter measurements provided new excellent lattice-spacing standards (WASO 9, for example) of the well known lattice-parameter values. As shown by the authors, the differences in lattice parameters of different samples of float-zone silicon (due to oxygen or carbon content) were not greater than a few parts in 10^8. Finally, the lattice parameter of silicon, $a = 5.43102088(16)$ Å, has been accepted as the atomic scale length standard (Mohr & Taylor, 2000).

Another reference material reported is crystals of pure rhombohedral corrundum (α-Al_2O_3), *i.e.* of ruby or sapphire (Herbstein, 2000, and references therein; Shvyd'ko *et al.*, 2002).

With silicon standards, measurements or remeasurements of $K\alpha_{1,2}$ and/or $K\beta_{1,3}$ X-ray emission lines and absolute wavelength determinations of most of the 3*d* transition metals (Cr, Mn, Fe, Co, Ni and Cu) have been performed [Härtwig, Grosswig, Becker & Windisch, 1991; Hölzer, Fritsch, Deutsch, Härtwig & Förster, 1997 (see §4.2.2)].

The standard crystals may also be used for determination of such physical quantities as the Avogadro constant (Deslattes *et al.*, 1994; Deslattes, Henins, Schoonover, Caroll & Bowman, 1976). The single accurate wavelength values, on the other hand, may be used both in simple measurements of lattice parameters [based directly on the Bragg law, equation (5.3.1.1)] and for accurate scaling of the wavelength spectra, in order to use them, for example, in high-accuracy lattice-parameter measurements based on complete convolution models [*cf.* §5.3.3.3.1, point(ii)].

Unlike the X-rays emitted from an X-ray tube, for which the spectral line and the characteristic wavelength are known, there are no such characteristic features in synchrotron radiation. Therefore, special energy-selective monochromators should be applied in relative lattice-spacing measurements using synchrotron radiation. Obaidur (2002) proposes two measurement schemes, using two types of high-resolution channel-cut monolithic monochromators. The first scheme (see Fig. 5.3.3.18) is a modification of the Bond method. The second one (see Fig. 5.3.3.19) uses the simultaneous Bragg condition for the indices (5,1,3), $(\bar{5},1,\bar{3})$, (1,5,3) and $(1,\bar{5},\bar{3})$. The lattice-spacing differences in Si wafers were determined in the sub-parts in 10^6 range of 0.6 parts in 10^6 (in the first scheme) and of 0.2 parts in 10^6 (second scheme).

Recently, a new atomic scale wavelength standard was proposed by Shvyd'ko *et al.* (2000), instead of the wavelength of the Cu $K\alpha_1$ emission line or of the lattice parameter of a silicon standard. It is the wavelength, λ_M, of the ^{57}Fe Mössbauer radiation, *i.e.* of γ radiation of natural linewidth from nuclear transitions. It has been measured to the sub-parts in 10^6 accuracy: $\lambda_M = 0.86025474(16)$ Å (relative accuracy 0.19 parts in 10^6). Its advantage, in relation to the previous standards, is the high spectral sharpness of the Mössbauer radiation of 3.5×10^{-13} in relative units, which makes its wavelength λ_M extremely well defined. This standard wavelength value, which lies a little outside of scope of the present review (X-ray methods), was next used for the lattice-parameter determination of sapphire single crystals with a relative accuracy of about 0.5 parts in 10^6 (Shvyd'ko *et al.*, 2002). Fig. 5.3.3.20 is a diagram of the measurement arrangement.

5.3.4. Final remarks

Let us review the most important problems concerning accurate and precise lattice-parameter determination.

The first, commonly known, requirement for obtaining the highest accuracy and precision is the use of high-Bragg-angle reflections. The tendency to obtain, record, and use in calculation such reflections can be met in rotating-crystal cameras in which Straumanis mounting is applied (Farquhar &

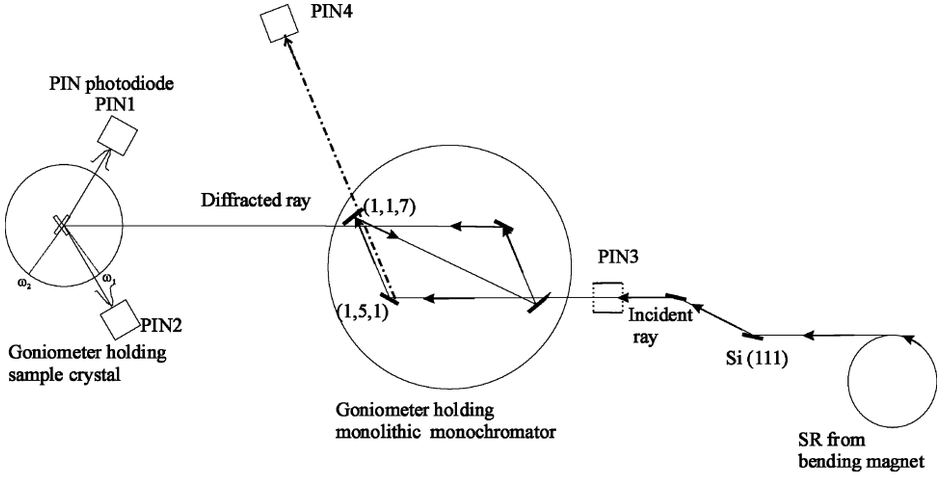

Fig. 5.3.3.18. Synchrotron radiation, SR, from the bending magnet incident on the Si(111) double-crystal monochromator and, after four reflections from the monolithic monochromator (0.1410 nm), impinges on sample Si(444). Two diffractions are recorded at the photodiode detectors, PIN1 and PIN2. The ω_1 and ω_2 values of the crystal positions are recorded using a Heiden height encoder.

references