International Tables for Crystallography (2006). Vol. C, Section 5.3.3.8, pp. 533–534.

uncoupled). Various applications of such high-sensitivity multiple-crystal X-ray spectrometers for reciprocal-space mapping and imaging (topography), which are outside the scope of the present paper, are reviewed by Fewster (1993, and references therein).

As was shown a few years later by Fewster & Andrew (1995), the device can also be used for absolute lattice-parameter measurements of single-crystal and polycrystalline materials with a relative accuracy of a few parts in 10^6. The authors checked the angular resolution and the sample centring of their instrument, and discussed systematic errors due to refraction, the Lorenz and polarization factor, the diffracting-plane tilt and the peak-position determination.

5.3.3.8. *Optical and X-ray interferometry – a non-dispersive technique*

The accuracy of an absolute measurement can be improved, in relation to that obtained in traditional methods (*cf.* Subsection 5.3.3.5), either if the wavelength of the radiation used in an experiment is known with better accuracy [*cf.* equation (5.3.1.3)] or if a high-quality standard single crystal is given, whose lattice spacing has been very accurately determined (Baker & Hart, 1975; mentioned in §5.3.3.7.3). The two tasks, *i.e.* very accurate determination of both lattice spacings and wavelengths in metric units, can be realized by use of combined optical and X-ray interferometry. This original concept of absolute-lattice-spacing determination directly in units of a standard light wavelength has been proposed and realized by Deslattes (1969) and Deslattes & Henins (1973).

The principle of the method is presented in Fig. 5.3.3.16. The silicon-crystal X-ray interferometer is a symmetric Laue-case type (Bonse & te Kaat, 1968). The parallel translation device consists of the stationary assembly (*a*) formed by two specially prepared crystals, and a moveable one (*b*), to which belongs the third crystal. One of the two mirrors of a high-resolution Fabry–Perot interferometer is attached to the stationary assembly and the second to the moving assembly. A stabilized He–Ne laser is used as a source of radiation, the wavelength of which has been established relative to visible standards. The first two crystals produce a standing wavefield, which is intercepted by the third crystal, so that displacement of the third crystal parallel to the diffraction vector (as suggested by the large arrow) produces alternate maxima and minima in the diffracted beams, detected by X-ray detector (*c*). Resonant transmission maxima of the optical interferometer are detected simultaneously by the photomultiplier indicated at (*d*). Analysis of the fringes (shown in Fig. 5.3.3.17) is the basis for the calculation of the lattice-spacing-to-optical-wavelength ratio (d/λ), which is given by

$$\frac{2d}{\lambda} = \frac{n}{m}\frac{\cos\alpha}{\cos\beta}, \qquad (5.3.3.48)$$

where n and m are the numbers of optical and X-ray diffraction fringes, respectively, and α and β are the measured angular deviations of the optical and X-ray diffraction vectors from the direction of motion. The measurements are carried out in two steps. First, the lattice parameter of silicon along the [110] crystallographic direction was measured in the metric system, independently of the X-ray wavelength used in the experiment. As the next step, a specimen of known lattice spacing, treated as a reference crystal, was used for the accurate wavelength determination of Cu $K\alpha_1$ and Mo $K\alpha_1$. Accuracy better than 1 part in 10^6 was reported (see Section 4.2.2).

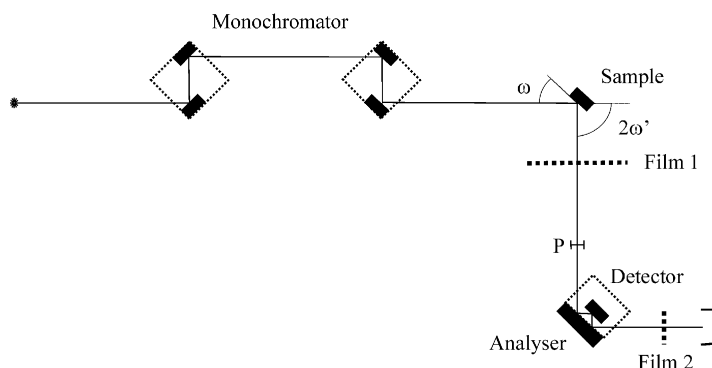

Fig. 5.3.3.15. The geometry of the diffractometer used by Fewster & Andrew (1995). The scattering angle, $2\omega'$, is the fundamental angle for determination of the interplanar spacing and P is the analyser-groove entrance.

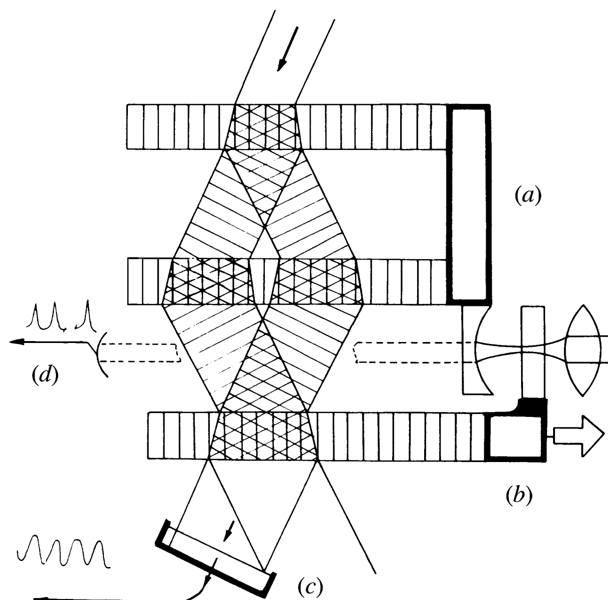

Fig. 5.3.3.16. Optical and X-ray interferometry. Schematic representation of the experimental set-up (after Deslattes & Henins, 1973; Becker *et al.*, 1981).

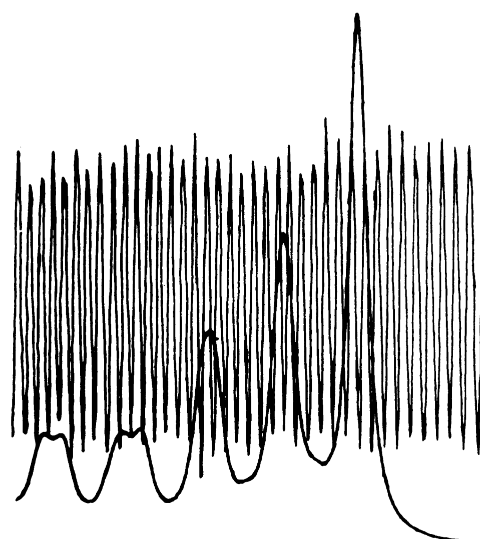

Fig. 5.3.3.17. Portion of a dual-channel recording of X-ray and optical fringes (Deslattes, 1969).

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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), $(\bar{5},\bar{1},\bar{3})$, (1,5,3) and $(\bar{1},5,\bar{3})$. 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 &

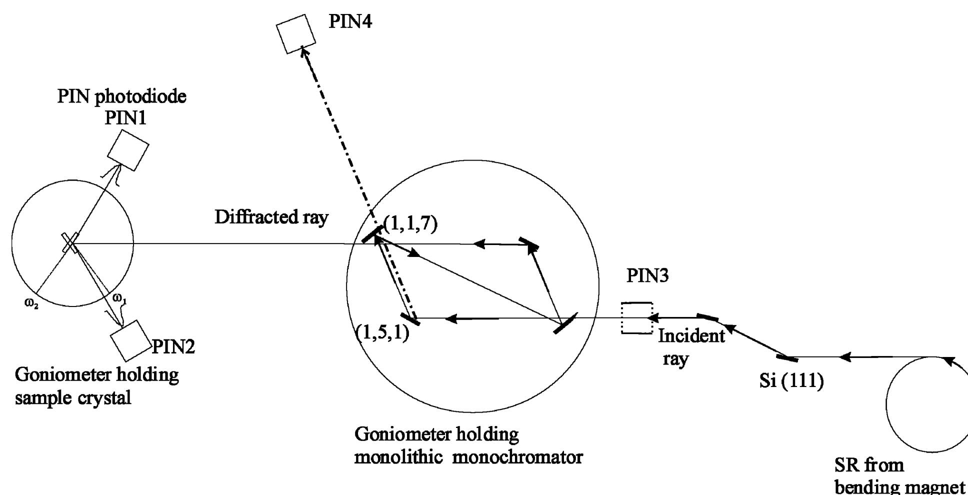

Fig. 5.3.3.18. Synchrotron radiation, SR, from the bending magnet incident on the Si(111) double-crystal monochromator and, after four reflections from the monolithic monochromator (0.1410 nm), impinges on sample Si(444). Two diffractions are recorded at the photodiode detectors, PIN1 and PIN2. The ω_1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

references