

5.3. X-RAY DIFFRACTION METHODS: SINGLE CRYSTAL

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 10⁶. 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

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

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 $\text{Cu } K\alpha_1$ and $\text{Mo } K\alpha_1$. Accuracy better than 1 part in 10⁶ was reported (see Section 4.2.2).

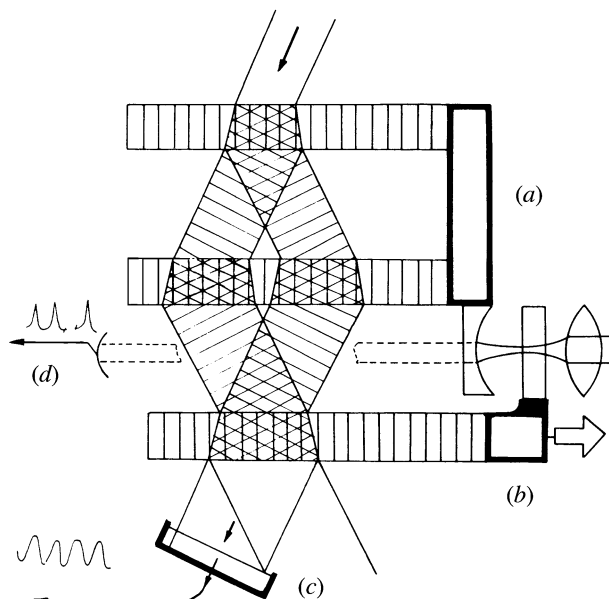


Fig. 5.3.3.16. Optical and X-ray interferometry. Schematic representation of the experimental set-up (after Deslattes & Henins, 1973; Becker *et al.*, 1981).

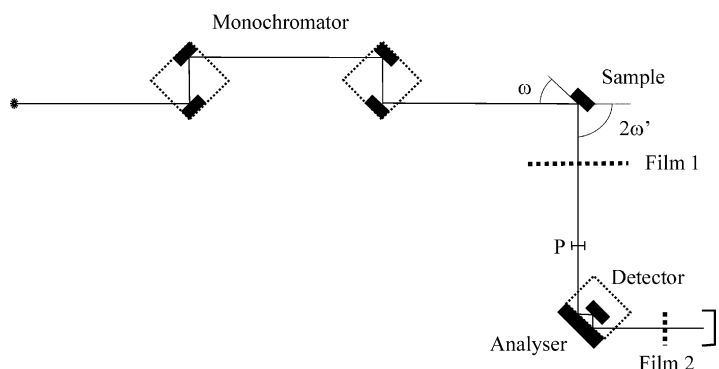


Fig. 5.3.3.15. The geometry of the diffractometer used by Fewster & Andrew (1995). The scattering angle, $2\omega'$, is the fundamental angle for determination of the interplanar spacing and P is the analyser-groove entrance.

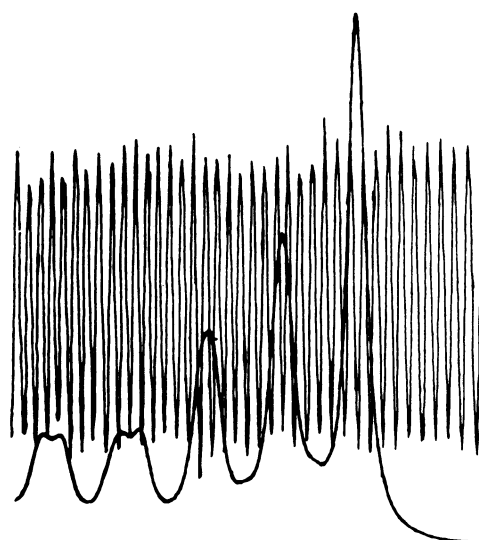


Fig. 5.3.3.17. Portion of a dual-channel recording of X-ray and optical fringes (Deslattes, 1969).