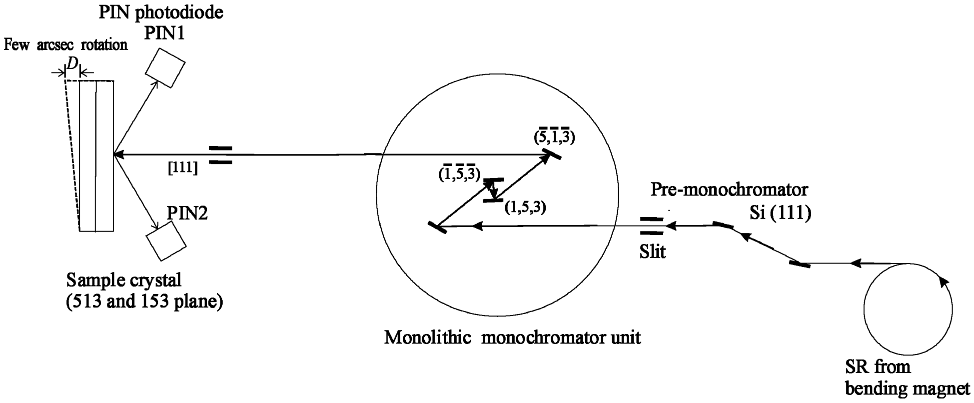

Fig. 5.3.3.19. Synchrotron radiation, SR, from the bending magnet incident on the Si(111) double-crystal monochromator and, after four reflections from the monolithic monochromator (0.1612 nm), impinges on sample Si(153). Two diffractions are recorded at the photodiode detectors, PIN1 and PIN2. The differences between the two peaks' D values are recorded using a Heiden height encoder.

Lipson, 1946; Popović, 1974), in the Weissenberg method (the use of zero-layer photographs), in multiple-exposure cameras (Glazer, 1972; Popović, Šljukić & Hanic, 1974), in standard and special diffractometers (Kobayashi, Yamada & Nakamura, 1963), in double-crystal arrangements with white X-radiation (Okazaki & Kawaminami, 1973*a*,*b*; Okazaki & Ohama, 1979; Ohama, Sakashita & Okazaki, 1979; Okazaki & Soejima, 2001), and in the triple-reflection scheme realized by means of the double-beam technique, proposed by Kovalchuk, Kovev & Pinsker (1975).

Increasing the θ value, in the case of photographic cameras or counter diffractometers, not only reduces the value of $\cot\theta$ in the formulae describing accuracy and precision, but also decreases several systematic errors proportional to $\cos\theta$, $\cos^2\theta$, $\cot\theta$ or $\cot^2\theta$ (Kheiker & Zevin, 1963; Wilson, 1963, 1980). To find the minimum total systematic error, which would occur for $\theta = 90°$ (not attainable in practice), extrapolation of the results is used (Farquhar & Lipson, 1946; Weisz, Cochran & Cole, 1948; Smakula & Kalnajs, 1955; Kobayashi, Yamada & Azumi, 1968; Pierron & McNeely, 1969).

The problem of the choice of suitable reflections for the measurement, calculation, and reduction of systematic errors could be generalized. Not only are such reflections for which θ values are close to 90° desired but also those for which ν tends to 90° in rotating-crystal cameras (Umansky, 1960), high-order Kossel lines in divergent-beam techniques, axial or non-axial reflections in counter-diffractometer methods, *etc.* Zero-layer reflections in Weissenberg photographs or in two-circle ('Weissenberg') diffractometers are preferable to upper-layer ones, because they are less affected by crystal misalignment (Clegg & Sheldrick, 1984) and a larger range of reciprocal-lattice points can be recorded (Luger, 1980); they are not sufficient, however, for the determination of all the lattice parameters in the less-symmetric crystal systems.

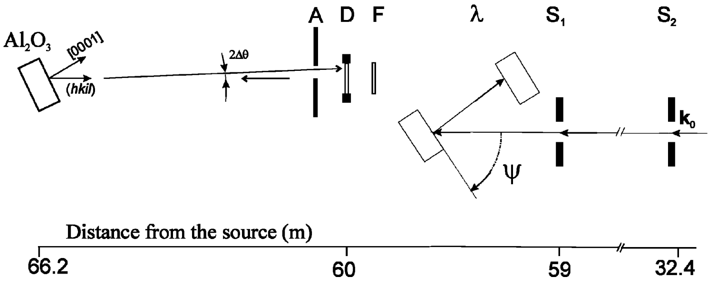

Fig. 5.3.3.20. Experimental set-up for measuring lattice parameters. X-rays after a high-heat-load monochromator (not shown) pass through the vertical slits S_1 and S_2 at a distance of 26.6 m. λ is a 'λ-meter'; F is a ^{57}Fe foil used as a source of the Mössbauer radiation of high brightness; D is a semi-transparent avalanche photodiode with 0.7 ns time resolution; Al_2O_3 is a sapphire single crystal in a furnace on a four-circle goniometer.

Use of the orientation matrix makes possible accurate crystal setting for an arbitrary reflection and identification of recorded reflections not only in the case of the automated four-circle diffractometer or the two-circle diffractometer but also in photographic methods.

For obtaining high accuracy of lattice-parameter determination, systematic errors depending on the radiation, the crystal, and the technique should be known, evaluated, and reduced or corrected. As a rule, those systematic errors whose part in the total systematic error is the most important should be removed first. Looking at the development of the X-ray diffraction techniques, the following remarks can be made.

As far as the photographic methods are concerned, the errors due to the means of recording (film shrinkage, uncertainty of measurement of distances on the film) and camera construction (radius in the moving-crystal methods and the source-to-film distance in divergent-beam techniques) play a major role. They can be reduced to some extent by using the Straumanis mounting or the ratio method, or the resolved doublet $K\alpha_{1,2}$. Various methods have been introduced for reducing the error due to the source-to-film distance in divergent-beam techniques.

In counter-diffractometer methods, which give more accurate determinations of the Bragg angles and intensities, several instrumental and physical factors should be taken into account (Kheiker & Zevin, 1963; Wilson, 1963, 1980; Berger, 1984, 1986*a*; Härtwig & Grosswig, 1989). The effects of some can be diminished by the use of Soller slits (Berger, 1984) and the effects of most can be reduced by the Bond (1960) geometry, in its basic form or in its various modifications (Kheiker, 1973; Mauer *et al.*, 1975; Hubbard & Mauer, 1976; Wołcyrz, Pietraszko & Łukaszewicz, 1980; Kudo, 1982; Lisoivan, 1982; Grosswig *et al.*, 1985), in particular in combination with double- or triple-crystal spectrometers (Kurbatov, Zubenko & Umansky, 1972; Godwod, Kowalczyk & Szmid, 1974; Hart & Lloyd, 1975; Sasvári & Zsoldos, 1980; Fewster, 1982; Pick *et al.*, 1977; Obaidur, 2002). Another arrangement giving a partial reduction of systematic errors is that proposed by Renninger (1937) and developed by Post (1975) and Kshevetsky *et al.* (1979, 1985), in which multiple-diffraction phenomena are applied. In most one- or double-crystal asymmetric spectro-