

7. MEASUREMENT OF INTENSITIES

references, see Thomlinson & Williams, 1984; Brown & Lindau, 1986).

A position-sensitive detector can replace the receiving slit when a reciprocal space is scanned. TV area detectors with an X-ray-to-visible light converter and two-dimensional CCD arrays have moderate resolution and efficiency, but they work in the current mode and do not provide pulse discrimination on the basis of the photon energy. One- and two-dimensional proportional chambers have a spatial resolution of the order of 0.1 mm, and the relative energy resolution,  $\Delta E/E \approx 0.2$ , is sufficient for rejection of some of the parasitic scattering.

The NaI(Tl) scintillation counter is used most frequently as the X-ray detector in crystallography. It has 100% efficiency for the commonly used wavelengths, and the energy resolution is comparable to that of a proportional counter. The detector has a long life, and the level of the low-energy noise can be reduced to about 0.1 counts  $s^{-1}$ .

The Ge and Si(Li) solid-state detectors (SSD) have an energy resolution  $\Delta E/E = 0.01$  to 0.03 for the wavelengths used in crystallography. The relative Compton shift,  $\Delta\lambda/\lambda$ , is  $(0.024 \text{ \AA}/\lambda) \times (1 - \cos 2\theta)$ , where  $2\theta$  is the scattering angle, so that even this component can be eliminated to some extent by a SSD. These detectors have been bulky and expensive, but new

constructions that are suitable for X-ray diffraction have become available recently. The effects of the detector resolution are shown schematically in Fig. 7.4.4.5 for a scintillation counter and a SSD.

Crystal monochromators placed in front of the detector eliminate all inelastic scattering but the TDS. The monochromator must be matched with the preceding X-ray optical system, the sample included, and therefore diffracted-beam monochromators are used in powder diffraction only (see Subsection 7.4.4.4).

7.4.4.4. Powder diffraction

The signal-to-background ratio is much worse in powder diffraction than in single-crystal diffraction, because the background is proportional to the irradiated volume in both cases, but the powder reflection is distributed over a ring of which only the order of 1% is recorded. The phase-space diagrams of a typical measurement are shown in Fig. 7.4.4.6. The Johansson monochromator is matched to the incident beam to provide

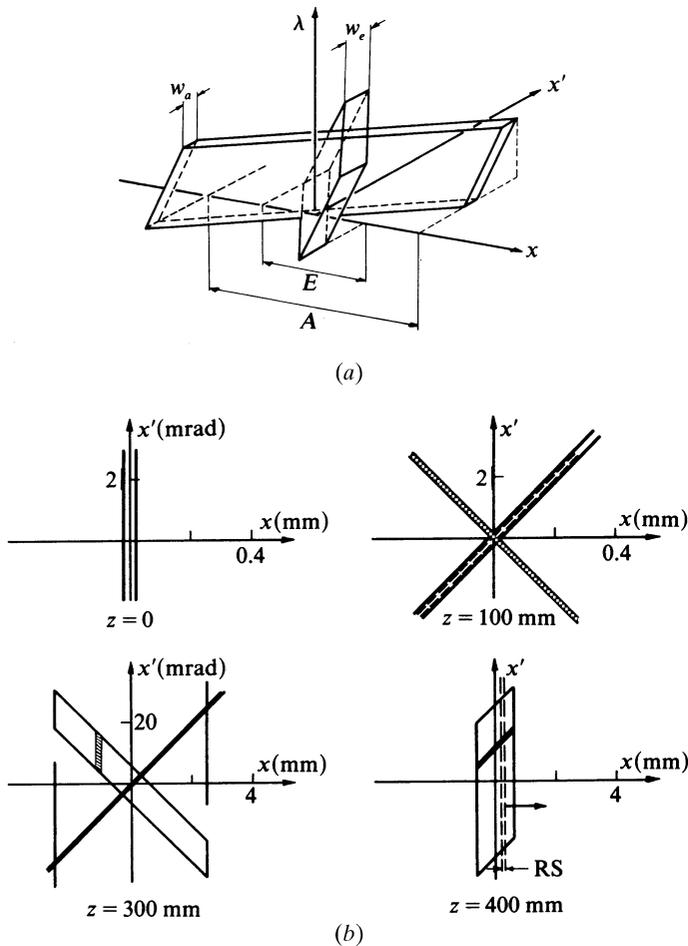


Fig. 7.4.4.6. Equatorial phase-space diagrams for powder diffraction in the Bragg-Brentano geometry. (a) The acceptance and emittance windows of a Johansson monochromator; (b) the beam in the  $\lambda = \lambda_1$  plane: the exit beam from the Johansson monochromator is shown by the hatched area ( $z = 100$  mm), the beam on the sample by two closely spaced lines, the reflectivity range of powder particles in a small area of the sample by the hatched area ( $z = 300$  mm, note the change of scales), and the scan of the reflected beam by a slit RS by broken lines ( $z = 400$  mm, at the parafocus).

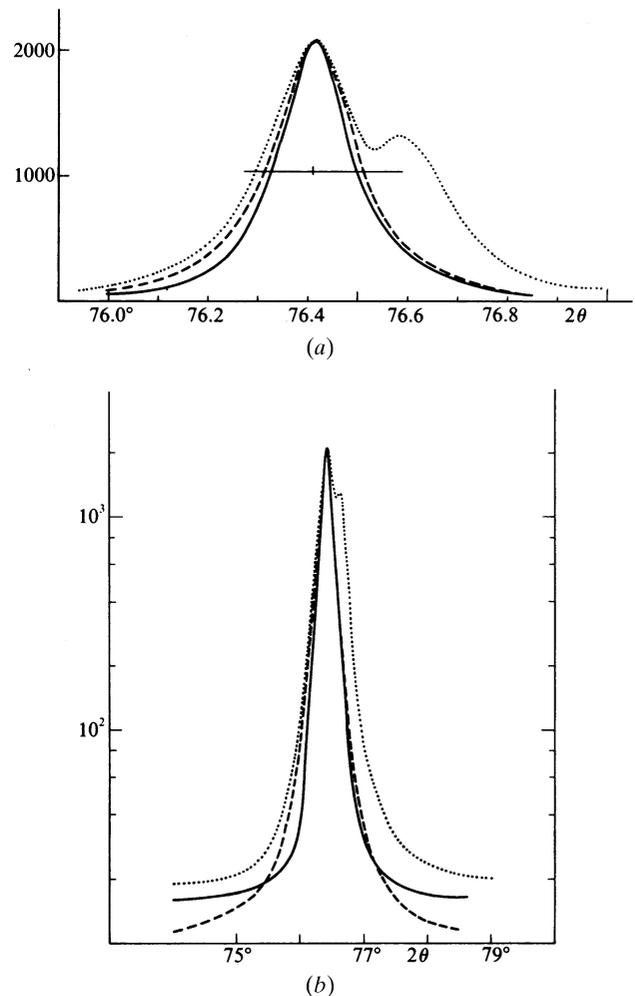


Fig. 7.4.4.7. Three measurements of the 220 reflection of Ni at  $\lambda = 1.541 \text{ \AA}$  scaled to the same peak value; (a) in linear scale, (b) in logarithmic scale. Dotted curve: graphite (00.2) Johann monochromator, conventional 0.1 mm wide X-ray source (Suortti & Jennings, 1977); solid curve: quartz (10.1) Johansson monochromator, conventional 0.05 mm wide X-ray source; broken curve: synchrotron radiation monochromatized by a (+, -) pair of Si (111) crystals, where the second crystal is sagittally bent for horizontal focusing (Suortti, Hastings & Cox, 1985). The horizontal line indicates the half-maximum value. In all cases, the effective slit width is much less than the FWHM of the reflection.