International Tables for Crystallography (2006). Vol. C, Figure 2.3.2.2, p. 54.

AT-cut quartz plate, or on Mylar for transmission. It is essential to rotate the specimen and increase the count time. A Gandolfi camera has also been used for very small specimens (see Section 2.3.4). A high-brilliance microfocus X-ray source has been used with a collimator made of 10 to 100 µm internal-diameter capillary tube. An *X–Y* stage is used with an optical microscope to locate selected areas of the specimen.

A microdiffractometer has been designed for microanalysis, Fig. 2.3.1.15 (Rigaku Corporation, 1990). It has been used to determine phases and stress in areas $< 10^4\,\mu m^2$ (Goldsmith & Walker, 1984). The key to the method is the use of an annular-ring receiving slit, which transmits the entire diffraction cone to the detector instead of a small chord as in conventional diffractometry, thereby utilizing all the available intensity. The pattern is scanned by translating the ring and detector along the direct-beam path so that

$$2\theta = \arctan(R_{\rm RS}/L), \qquad (2.3.1.26)$$

where $R_{\rm RS}$ is the radius of the ring slit and L the distance from the fixed specimen. For $R_{\rm RS} = 15$ mm, L varies from 171 to 9 mm in the transmission range 5 to $60^\circ 2\theta$; a 50 mm diameter scintillation counter is used. A doughnut-shaped proportional counter (3/4 of a full circle) is used for the 30 to 150° reflection specimen mode. The slit width is 0.2 mm and the aperture varies with 2θ. The intensities fall off at the higher 2θ's because of the small incidence angles to the slit. An alternative method uses a position-sensitive proportional counter. Steinmeyer (1986) has described applications of microdiffractometry.

By using synchrotron radiation (Section 2.3.2), single-crystal data for structure determination can now be obtained from a microcrystal about 5–10 µm in size; see Andrews *et al.* (1988), Bachmann, Kohler, Schultz & Weber (1985), Harding (1988), Newsam, King & Liang (1989), Cheetham, Harding, Mingos & Powell (1993), Harding & Kariuki (1994), and Harding, Kariuki, Cernik & Cressey (1994).

2.3.2. Parallel-beam geometries, synchrotron radiation

The radiation from the X-ray tube is divergent and various methods can be used to obtain a parallel beam as shown in Fig. 2.3.2.1. Symmetrical reflection from a flat crystal is the usual method. An asymmetric reflecting monochromator with small incidence angle and large exit angle expands the beam, or in reverse condenses it (§2.3.5.4.1). A channel monochromator has the advantage of not changing the beam direction. A receiving slit or preferably Soller slits can be used to define the diffracted beam. A graphite monochromator in the diffracted beam or a solid-state detector eliminates fluorescence. The incident-beam parallel slits limit vertical divergence. However, all the methods result in a large loss of intensity compared with conventional focusing. In contrast, the storage ring produces a virtually parallel beam with very small vertical divergence of about 0.1 mrad, and the monochromator is used only to select the wavelength. The rest of this section assumes a synchrotron-radiation source.

Storage-ring X-ray sources have a number of unique properties that are of great importance for powder diffraction. The advantages of synchrotron powder diffraction have been described by Hastings, Thomlinson & Cox (1984), Parrish & Hart (1987), Parrish (1988), and Finger (1989). Excellent patterns with high resolution and high peak-to-back ground ratio have been reported. These include the orders-of-magnitude higher intensity and nearly uniform spectral distribution compared with X-ray tubes, the wide continuous range of selectable wavelengths, and the single profile that avoids the problems caused by $K\alpha$ doublets and β filters. Owing to major differences in the diffractometer geometries, comparisons of intensities with X-ray tube focusing methods cannot be predicted simply from the number of source photons.

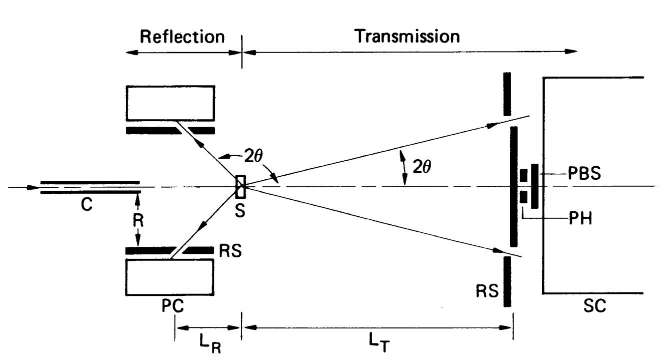

Fig. 2.3.1.15. Rigaku microdiffractometer for microanalysis. *C* collimator, PC ring proportional counter, RS ring slit with radius *r*, *S* specimen, SC scintillation counter, PBS primary beam stop, PH pinhole for alignment, *L* specimen-to-receiving-slit distance.

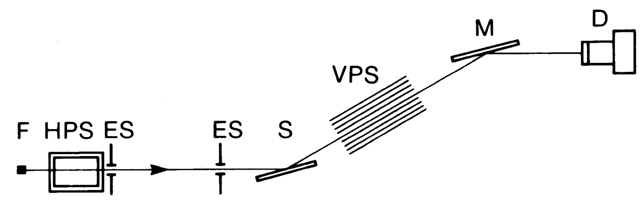

Fig. 2.3.2.1. Method to obtain parallel beam from X-ray tube for powder diffraction. HPS parallel slits to limit axial divergence, ES entrance slits (can be replaced by pair of flat parallel steel bars), *S* specimen, VPS parallel slits to define diffracted beam, *M* flat monochromator (can be omitted). *D* detector. See also Fig. 2.3.2.4(*a*).

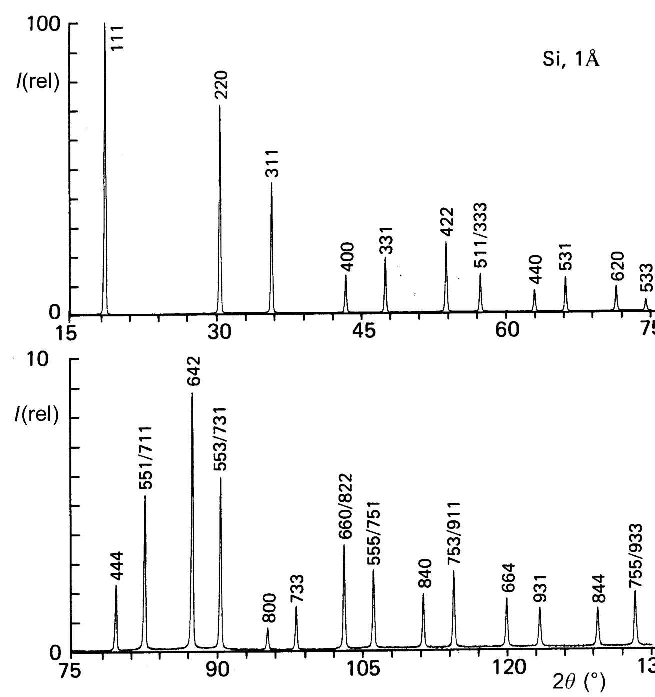

Fig. 2.3.2.2. Silicon powder pattern with 1 Å synchrotron radiation using method shown in Fig. 2.3.2.4(*a*). The 444 reflection is the limit for Cu $K\alpha$ radiation.

references