International Tables for Crystallography (2006). Vol. C, Figure 2.3.1.1, p. 43.

diffraction (Reynolds, 1989), instrumentation, specimen preparation, profile fitting, synchrotron and neutron methods. A recent book by Jenkins & Snyder (1996) gives a useful comprehensive account of basic methods and practices in powder diffractometry.

Papers presented at international symposia on powder diffraction describe advances in the field (Block & Hubbard, 1980; *Australian Journal of Physics*, 1988; Bojarski & Bołd, 1979; Bish & Post, 1989; Prince & Stalick, 1992). Papers on the theory and new methods and applications are published in the *Journal of Applied Crystallography*. *Powder Diffraction* started in 1986 and contains powder data and papers on instrumentation and methods. Papers presented at the Annual Denver Conference on Applications of X-ray Analysis have been published yearly since 1957 as separate volumes entitled *Advances in X-Ray Analysis*, by Plenum Press. Volume 37 of the series was published in 1994. These volumes contain papers that are roughly equally divided between X-ray powder diffraction and fluorescence analysis. This extensive source describes many types of instrumentation, methods and applications. The *Norelco Reporter* has been published several times a year since 1954 and contains original articles and reprints of papers on powder diffraction, fluorescence analysis, and electron microscopy. There are other 'house journals' published by Rigaku, Siemens, and other X-ray companies. A history of the powder method in the USA was written by Parrish (1983).

The following description includes only the most frequently used methods. The divergent beam from X-ray tubes is best used with focusing geometries, and synchrotron radiation with parallel-beam optics.

2.3.1. Focusing diffractometer geometries

The critical elements in the basic geometries of the principal focusing diffractometers used with X-ray tubes are illustrated schematically in Fig. 2.3.1.1. The six arrangements are described below in paragraphs (*a*) to (*f*) corresponding to the subdivisions of Fig. 2.3.1.1. The most frequently used methods are illustrated in (*a*) and (*b*). The abbreviations used are *S*(*R*) for the specimen set for reflection, *S*(*T*) for a transmission specimen, and *M* refers to a focusing reflection monochromator. The letters are arranged in order of the beam direction. The detector rotates around the diffractometer axis at twice the speed of the specimen in the θ–2θ scanning used in (*a*) to (*e*). In the Seemann–Bohlin geometry (*S-B*), the specimen is stationary and the detector rotates around the focusing circle in scanning the pattern (*f*). The line focus of the X-ray tube is used in all cases.

The monochromator is either symmetrical, with the lattice planes parallel to the crystal surface, or asymmetric with the lattice planes inclined at a small angle to the surface to shorten one of the focal-length distances. Placing the monochromator in the diffracted beam has the important advantage of eliminating specimen fluorescence. It also simplifies shielding the detector if the specimen is radioactive. The monochromator in the incident beam reduces fluorescence and radiation damage to the specimen by removing the continuous X-ray spectrum. When the diffracted beam is defined by the receiving slit as in (*b*) and (*c*), highly oriented pyrolytic graphite (placed in front of the detector) is generally used to obtain high intensity. In the (*d*) and (*c*) geometries, a high-quality bent crystal such as silicon or quartz is necessary to achieve good focusing.

(*a*) *S*(*R*): The aperture of the incident divergent beam from the line focus of the X-ray tube *F* is limited by the entrance slit ES and the reflection from the specimen converges ('focuses') on the receiving slit RS. The intensity is determined by the ES and RS and the profile width is determined mainly by the RS width. The parallel slits PS in the incident and diffracted beams limit the axial divergence.

(*b*) *S*(*R*)/*M*: Same as (*a*) with the addition of the symmetrical monochromator (usually graphite) to record only the characteristic radiation. ES and RS have the same rôle as in (*a*), and only the incident PS are required.

(*c*) *M*/*S*(*R*): Using an incident-beam monochromator, the slit at *F*′ determines the effective source size and divergence of the beam striking the specimen, and *RS* limits the profile width.

(*d*) *S*(*T*)/*M*: The divergent incident beam continues to diverge after diffraction from the transmission specimen and the asymmetric monochromator focuses the beam on the detector.

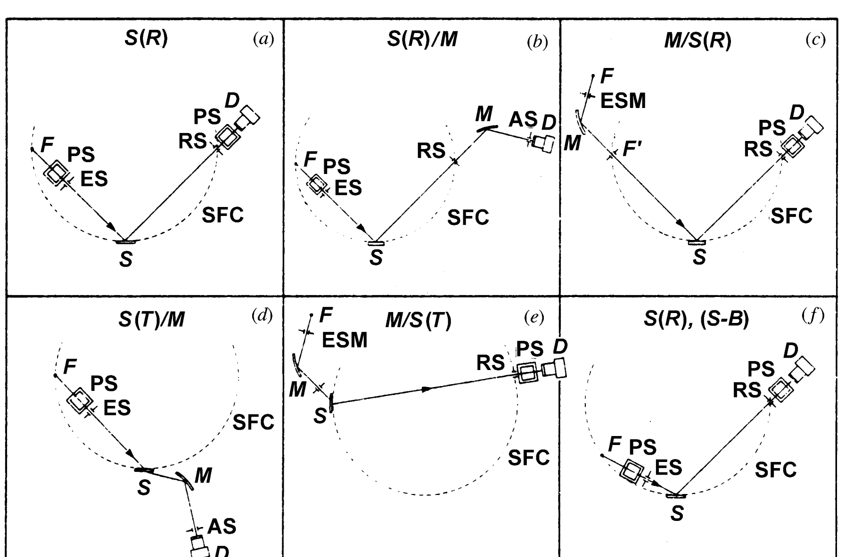

Fig. 2.3.1.1. Basic arrangements of focusing diffractometer methods. Simplified and not to scale; detailed drawings shown in later figures. (*a*)–(*e*) operate with θ–2θ scanning; (*f*) fixed specimen with detector scanning. *F* line focus of X-ray tube (normal to plane of drawings), *F*′ focus of incident-beam monochromator, PS parallel slits (to limit axial divergence), ES entrance (divergence) slit, ESM entrance slit for monochromator, *S* specimen, RS receiving slit, AS antiscatter slit, *D* detector, SFC specimen focusing circle, *M* focusing monochromator. Other symbols described in text.

references