International Tables for Crystallography (2006). Vol. C, Figure 2.3.1.3, p. 44.

M and D rotate around S in θ–2θ scanning and the profile width is determined by the monochromator. Only the forward-reflection region can be recorded.

(*e*) $M/S(T)$: This is the diffractometer equivalent of the Guinier camera. A symmetric or asymmetric monochromator is used in the incident beam and the profile width is determined by the *RS*. The incident-beam divergence is limited by ESM.

(*f*) $S(R)$,$(S$–$B)$: The reflections are focused on a fixed-radius circle which measures 4θ. A linkage moves the detector around the focusing circle and always points it to the fixed specimen. The angular range is limited (normally 30–240°4θ) and can be changed by moving the specimen and diffractometer to different positions. The profile width is determined by ES and RS. The same geometry is used with an incident- or diffracted-beam focusing monochromator.

The interaction of the X-ray beam with the specimen varies in different geometries and this may have important consequences on the results, as will be described later. When a reflection specimen is used in θ–2θ or θ–θ scanning, only those crystallites whose lattice planes are oriented nearly parallel to the specimen surface can reflect (Fig. 2.3.1.2) (Parrish, 1974). A transmission specimen in θ–2θ scanning permits reflections only from planes nearly normal to the surface. In the *S–B* case, reflections can occur from planes inclined over a range of about 45° to the surface. Transmission specimens must, of course, be mounted on X-ray-transparent substrates. Jenkins (1989) has reviewed the instrumentation and experimental procedures.

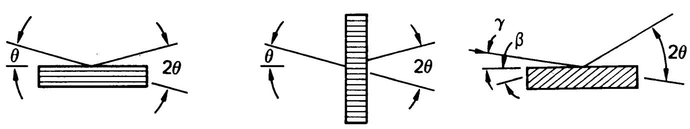

Fig. 2.3.1.2. Specimen orientation for three diffractometer geometries. With θ–2θ scanning, diffraction is possible only from planes nearly parallel to the reflection specimen surface (left), and from planes nearly normal to the transmission specimen surface (middle), and from planes inclined different amounts to the specimen surface in Seemann–Bohlin geometry (right).

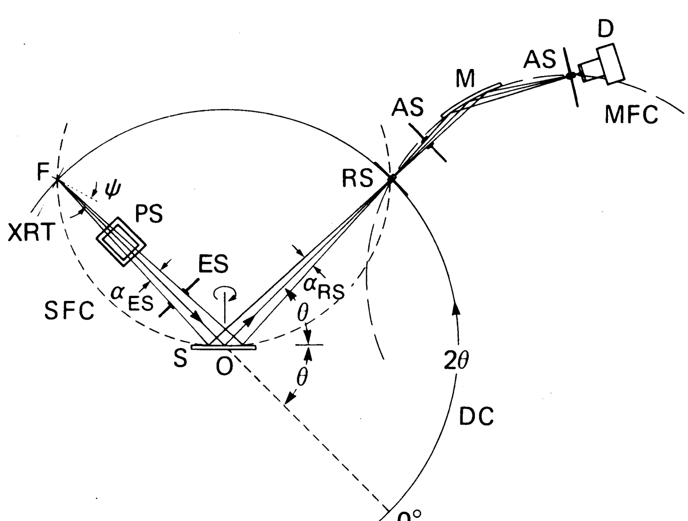

Fig. 2.3.1.3. X-ray optics in the focusing plane of a 'conventional' diffractometer with reflection specimen, diffracted-beam monochromator, and θ–2θ scanning: ψ take-off angle, DC diffractometer circle, MFC monochromator focusing circle, α_{ES} and α_{RS} entrance- and receiving-slit apertures, θ Bragg angle, 2θ reflection angle, O diffractometer and specimen rotation axis; other symbols listed in Fig. 2.3.1.1.

2.3.1.1. *Conventional reflection specimen, θ–2θ scan*

The reflection specimen with θ–2θ scanning in the focusing arrangement shown in Fig. 2.3.1.3 is the most widely used powder diffraction method. It is estimated that about 10 000 to 15 000 of these diffractometers have been sold since they were introduced in 1948, which makes it the most widely used X-ray crystallographic instrument. Some authors have called it the Bragg–Brentano parafocusing method (Bragg, 1921; Brentano, 1946), but the X-ray optics (described below) are significantly different from the methods and instruments described by these authors.

The X-ray tube spot focus was first used as the source and gave broad reflections. A narrow entrance slit improved the resolution but caused a large loss of intensity. Early diffractometers were described by LeGalley (1935), Lindemann & Trost (1940), and Bleeksma, Kloos & DiGiovanni (1948); see Parrish (1983). The use of the line focus with parallel slits to limit axial divergence was developed in the late 1940's and gave much higher resolution. A collection of papers by Parrish and co-workers (Parrish, 1965) and Klug & Alexander (1974) describe details of the instrumentation and method.

2.3.1.1.1. *Geometrical instrument parameters*

The powder diffractometer is basically a single-axis goniometer with a large-diameter precision gear and worm drive. The detector and receiving-slit assembly are mounted on an arm attached to the gear in a radial position. The specimen is mounted in a holder carried by a shaft precisely positioned at the centre of the gear. 2/1 reduction gears drive the specimen post at one-half the speed of the detector. Some diffractometers have two large gears, making it possible to drive only the detector with the specimen fixed or *vice versa*, or to use 2/1 scanning. Synchronous motors have been used for continuous scanning for ratemeter recording and stepping motors for step-scanning with computer control.

The geometry of the method requires that the axis of rotation of the diffractometer be parallel to the X-ray tube focal line to obtain maximum intensity and resolution. The target is normal to the long axis of the tube; vertically mounted tubes require a diffractometer that scans in the vertical plane while a horizontal tube requires a horizontal diffractometer. The X-ray optics are the same for both. The incident angle θ and the reflection angle 2θ are defined with respect to the central ray that passes through the diffractometer axis of rotation O.

The axis of rotation of the specimen is the central axis of the main gear of the diffractometer, as shown in Fig. 2.3.1.3. The centre of the specimen is equidistant from the source F and receiving slit RS. The instrument radius $R_{DC} = F - O = O - \mathrm{RS}$. The radius of commercial instruments is in the range 150 to 250 mm, with 185 mm most common. Changing the radius affects the instrument parameters and a number of the aberrations. Larger radii have been used to obtain higher resolution and better profile shapes. For example, the asymmetric broadening caused by axial divergence is decreased because the chord of the diffraction cone intercepted by the receiving slit has less curvature. However, if the same entrance slit is used, moving the specimen further from the source proportionately increases the length of specimen irradiated and decreases the intensity.

The imaginary specimen focusing circle SFC passes through F, O and the middle of RS and its radius varies with θ:

references