International Tables for Crystallography (2006). Vol. C, Figure 2.3.1.13, p. 52.

easily measured with a standard specimen set to reflect the $K\alpha$ and the specimen to be measured inserted normal to the diffracted beam in front of the detector. It is not critical to achieve the exact value and a range of ±15–20% of the transmission can be tolerated. This minimizes the effect of the absorption change with 2θ, and corrections of the relative intensities are required only when accurate values are required.

The intensity of the incident beam can be measured at 0° in the same geometry and used to scale the relative intensities to ‘absolute’ values. The flat specimen, transparency, and specimen surface displacement aberrations are similar to those in reflection specimen geometry except that they vary as $\sin\theta$ rather than $\cos\theta$. This is an important factor in the measurement of large-*d*-spacing reflections. The flat-specimen effect is smaller because the irradiated specimen length is usually smaller. The transparency error is also usually smaller because thin specimens are used.

An important advantage of the method is that the specimen displacement can be directly determined by measuring the peak in the normal position and again after rotating the specimen holder 180°. The correct peak position is at one-half the angle between the two values. The axial divergence has the same effect as in reflection. The limitations are that only the forward-reflection region is accessible, and the intensity is about one-half of the reflection method (except at small angles) because smaller specimen volumes are used.

An alternative arrangement for the transmission specimen mode is to use an incident-beam monochromator as shown in Fig. 2.3.1.12(*b*). This is similar to the geometry used in the Guinier powder camera with the detector replacing the film. A high-quality focusing crystal is required. Wölfel (1981) used a symmetrical focusing monochromator with 260 mm focal length for quantitative analysis. Göbel (1982) used an asymmetric monochromator with a position-sensitive detector for high-speed scanning, see §2.3.5.4.1. By proper selection of the source size and distances, the $K\alpha_2$ can be eliminated and the pattern contains only the $K\alpha_1$ peaks (Guinier & Sébilleau, 1952). This geometry can have high resolution with the FWHM typically about 0.05 to 0.07°. The profile widths are narrower for the subtractive setting of the monochromator than for the additive setting.

The pattern is recorded with θ–2θ scanning. The 0° position can be determined by measuring 4θ, *i.e.* peaks above and below 0°, or calibration can be made with a standard specimen. A slit after the monochromator limits the size of the beam striking the specimen. The width and intensity of the powder reflections are limited by the receiving-slit width. A parallel slit is used between the specimen and detector to limit axial divergence.

The full spectrum from the X-ray tube strikes the monochromator and only the monochromatic beam reaches the specimen, so that it is preferred for radiation-sensitive materials. On the other hand, the radiation reaching the specimen may cause fluorescence (though considerably less than the full spectrum) which adds to the background.

2.3.1.3. *Seemann–Bohlin method*

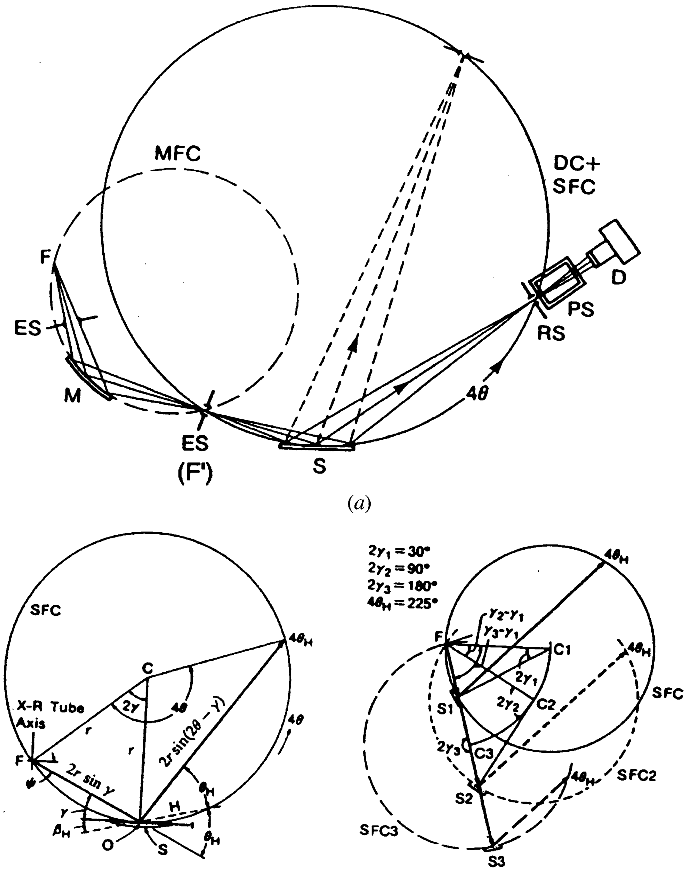
The Seemann–Bohlin (*S–B*) diffractometer has the specimen mounted on a radial arm instead of the axis of rotation and a linkage or servomechanism moves the detector around the circumference of a fixed-radius focusing circle while keeping it pointed to the stationary specimen. All reflections occur simultaneously focused on the focusing circle as shown in Fig. 2.3.1.13(*a*). The method was originally developed for powder cameras by Seemann (1919) and Bohlin (1920) but was not widely used because of the limited angular range and the broad reflections caused by inclination of the rays to the film. The diffractometer eliminates the broadening and extends the angular range. Diffractometers designed for this geometry have been described by Wassermann & Wiewiorosky (1953), Segmüller (1957), Kunze (1964*a*,*b*), Parrish, Mack & Vajda (1967), King, Gillham & Huggins (1970), Feder & Berry (1970), and others.

The geometry is shown in Fig. 2.3.1.13(*b*) (Parrish & Mack, 1967). Reflections occur from lattice planes with varying inclinations β_H to the specimen surface. The reflecting position of a plane H is $\theta_H = \gamma + \beta_H$, where γ is the incidence angle and $4\theta_H$ the reflection angle. The maximum value of β_H is about 45°. It is essential to align the specimen tangent to FC. This is a critical adjustment because even a small misalignment causes profile broadening and loss of peak intensity.

The source may be the line focus of the X-ray tube [F in Fig. 2.3.1.13(*b*)] or at the focus of a monochromator [ES in Fig. 2.3.1.13(*a*)]; in the latter case, the entrance slit at F' limits the divergent beam reaching the specimen. The source, specimen centre O, and receiving slit RS lie on the specimen focusing circle SFC, which has a fixed radius r. The incidence angle γ is given by

$$\gamma = \arcsin(b/2r), \qquad (2.3.1.21)$$

where b is the distance from F or F' to O, or $2r\sin\gamma$. The γ angle determines the angular range that can be recorded with a given r, decreasing γ decreases $2\theta_{\min}$. The relationships of specimen position on the focusing circle and the recording range



(*a*)

(*b*)

(*c*)

Fig. 2.3.1.13. Seemann–Bohlin method. (*a*) X-ray optics using incident-beam monochromator. (*b*) X-ray tube line-focus source showing geometrical relations: γ mean angle of incident beam, β_H inclination of reflecting plane H to specimen surface, θ_H Bragg angle of H plane, t tangent to focusing circle at O. (*c*) Diffractometer settings for various angular ranges.

references