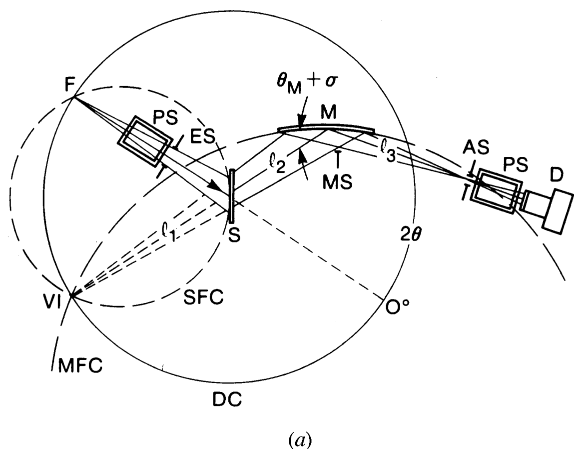
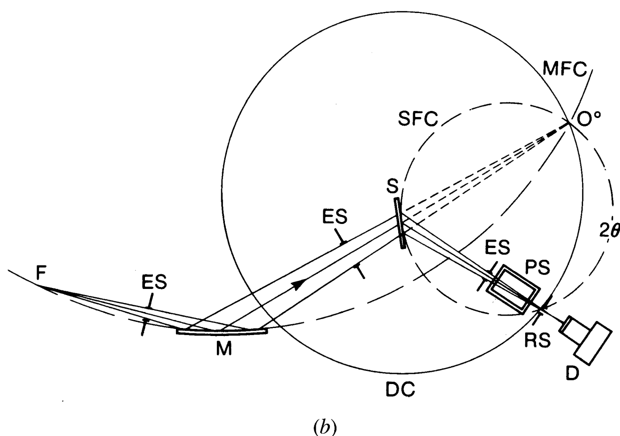
International Tables for Crystallography (2006). Vol. C, Figure 2.3.1.12, p. 51.

diverge after it passes through the specimen and the monochromator is required to refocus the beam, on the detector as shown in Fig. 2.3.1.12 (de Wolff, 1968*b*; Parrish, 1958). The monochromator can be placed before or after the specimen and the position has different effects on the pattern. Using the monochromator in the diffracted beam, the intensity and width of the profiles are determined by the X-ray focal line width and the quality of the bent monochromator rather than the receiving slit which serves as an antiscatter slit. This geometrical arrangement places the virtual image VI of the focal line at the intersection of the focusing circles. After reflection from the specimen, the divergent beam is again reflected by the focusing crystal M and converges on the detector. The pattern is recorded with θ–2θ scanning with the monochromator and detector both mounted on a rigid arm rotating around the diffractometer axis. A beam stop MS can be translated and moved in and out near the crossover point to prevent the primary beam from entering the detector at small 2θ's. To avoid long radii, the crystal surface is cut at an angle σ (about 3°) to the reflecting lattice plane. The distances are related by

$$\begin{aligned}(l_1+l_2)/l_3 &= [\sin(\theta_M+\sigma)]/[\sin(\theta_M-\sigma)]\\ R_{\rm FC} &= [l_1+l_2]/[2\sin(\theta_M+\sigma)]\\ &= l_3/[2\sin(\theta_M-\sigma)],\end{aligned} \quad (2.3.1.16)$$



(*a*)



(*b*)

Fig. 2.3.1.12. X-ray optics of the transmission specimen with asymmetric focusing monochromator and θ–2θ scanning. (*a*) Monochromator in diffracted beam. θ_M Bragg angle of monochromator with surface cut at angle σ to reflecting plane, MS adjustable beam stop, I_1, I_2, and I_3 defined in text and other symbols listed in Fig. 2.3.1.3. (*b*) Monochromator in incident beam, equivalent to Guinier focusing camera.

where θ_M is the Bragg angle of the monochromator for the selected wavelength and the l's are shown in Fig. 2.3.2.12(*a*).

Because the profile shape and the intensity are determined by the monochromator, the crystal quality and the accuracy of the bending are crucial factors in determining the quality of the pattern. A flat thin quartz (101) wafer bent with a special device to approximate a section of a logarithmic spiral has been successfully used (de Wolff, 1968*b*). The curvature can be varied to obtain the sharpest focus. Thin silicon crystals that can be bent are now available, and Johann and Johannsen asymmetric crystals may be used. Pyrolytic graphite monochromators are not applicable; the radii would be longer because graphite is too soft to be cut at an angle, and a receiving slit would be necessary to define the diffracted beam because the monochromator produces a broad reflection.

A polarization factor is introduced by the monochromator,

$$p=(1+k\cos^2 2\theta)/(1+k), \quad (2.3.1.17)$$

where $k=\cos^2 2\theta_M$ for mosaic crystals and $k=\cos 2\theta_M$ for perfect crystals. The value of k is strongly dependent on the surface finish of the crystal and the crystal should be measured to determine the effect. A specimen with accurately known structure factors such as silicon can be used to calibrate the intensities.

The $K\alpha$-doublet separation is zero at the 2θ angle at which the dispersion of the specimen compensates that of the monochromator, *i.e.* the 2θ at which the monochromator is aligned and also depends on the distances. The $K\alpha_1$ and $K\alpha_2$ peaks are superposed and appear as a single peak over a small range of 2θ's. The $K\alpha_2$ peak gradually separates with increasing 2θ but the separation is less than calculated from the wavelengths and the intensity ratio may not be 2:1 until higher angles are reached as shown in Fig. 2.3.1.8.

A larger angular aperture α_T can be used for transmission than for reflection α_R because the specimen is more nearly normal to than parallel to the primary beam:

$$\alpha_T/\alpha_R=2R_D/[1+(R/l_2)L_s], \quad (2.3.1.18)$$

where the diffractometer radius $R_D=l_1$. For $R_D=170$ mm, specimen length $L_S=20$ mm and $l_2=65$ mm; α_T could be 4.7 times larger than α_R but the monochromator length usually limits it to about 3°. The smallest reflection angle that can be measured is

$$2\theta_{\rm min}=\alpha_T[(R_D+l_2)/l_2]. \quad (2.3.1.19)$$

Using $\alpha_T=0.5°$, $2\theta_{\rm min}=1.75°$ and $d=50$ Å for Cu $K\alpha$ radiation.

Specimen preparation is not difficult and the preparation can be easily tested and changed. The specimen must be X-ray transparent and can be a free-standing film or foil, or a powder cemented to a thin substrate. The substrate selection is important because its pattern is included. If both transmission and reflection patterns are to be compared, the substrate should be selected to have a minimal contribution to both. For example, Mylar is a good substrate for transmission but has a strong reflection pattern, and although rolled Be foil has a few reflections it is often satisfactory for both.

The absorption factor is

$$A=(t/\cos\theta)\exp(-s/\cos\theta), \quad (2.3.1.20)$$

where t is the powder thickness and s is the sum of the products of the absorption coefficients and thicknesses of the powder and the substrate. The optimum specimen thickness to give the highest intensity is $\mu t=1$, *i.e.* the specimen should transmit about 38% of the incident $K\alpha$ intensity. The transmission can be

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