are illustrated in Fig. 2.3.1.13(*c*). To change the range requires rotation of the X-ray tube axis or the diffractometer around *F*. The detector must also be repositioned. For forward-reflection measurements, γ is usually $\leq 10^\circ$. Extreme care must be used in the specimen preparation to avoid errors due to microabsorption (particle-shadowing) effects, which increase with decreasing γ. The 0° position cannot be measured directly and a standard is used for calibration. The range from 0° to about $15^\circ 2\theta$ is inaccessible because of mechanical dimensions. At $\gamma = 90^\circ$, only the back-reflection region can be scanned.

The aperture of the beam striking the specimen is

$$\alpha_{SB} = 2\arctan(ES_w/2a), \qquad (2.3.1.22)$$

where ES_w is the entrance slit width and *a* the distance between *F* or *F′* and the slit. The irradiated specimen length *l* is constant at all angles, $l = 2\alpha r$. A large aperture can be used to increase intensity since the specimen is close to *F*. However, the selection of α is limited if γ is small, and also because of the large flat-specimen aberration.

The receiving-slit aperture varies with the distance of the slit to the specimen

$$\alpha_{RS}(^\circ 4\theta) = 2\arctan RS_w/[2r\sin(2\theta - \gamma)]. \qquad (2.3.1.23)$$

Consequently, the resolution and relative intensity gradually change across the pattern. The *S*–*B* has greater widths at the smaller 2θ's and nearly the same widths at the higher angles compared with the θ–2θ diffractometer. The aperture can be kept constant by using a special slit with offset sides (to avoid shadowing) and pointing the opening to *C* while the detector remains pointed to *O* (Parrish *et al.*, 1967). The slit opening is tangent to FC and inclined to the beam and rotates while scanning. The constant aperture slit has

$$\alpha_{RS}(^\circ 4\theta) = 2\arctan(RS_w/2r). \qquad (2.3.1.24)$$

The axial divergence is limited by parallel slits as in conventional diffractometry and the effects are about the same. The equatorial aberrations are also similar but larger in magnitude. The specimen-aberration errors are listed in Table 5.2.7.1. The flat specimen causes asymmetric broadening; the shift is proportional to α_{ES}^2 and increases with decreasing θ. It can be eliminated by making the specimen with the same curvature as $r = FC$. In this case, one curvature satisfies the entire angular range because the focusing circle has a fixed radius. However, the curvature precludes rotating the specimen. The specimen transparency also causes asymmetric broadening and a peak shift that increases with decreasing θ. For $\mu h \rightarrow 0$, the geometric term is the same as for specimen displacement (Mack & Parrish, 1967).

The diffracted intensity is proportional to $I_0 A(\mu h) TB$, where I_0 is the incident intensity determined by α, δ, and the axial length *L* of the incident-beam assembly, $A(\mu h)$ is the specimen absorption factor, *T* the transmission of the air path, and *B* the length L_{RS} of the diffracted ring intercepted by the slit. The X-rays reflected at a depth *x* below the specimen surface are attenuated by

$$\exp\{-[\mu x\,\mathrm{cosec}\,\delta + \mu x\,\mathrm{cosec}\,(2\theta - \delta)]\}, \qquad (2.3.1.25)$$

where μ is the linear absorption coefficient. The asymmetric geometry causes the absorption to vary with the reflection angle. The air absorption path varies with the distance *O* to RS and reaches a maximum at $180^\circ + 2\gamma$. The expression for air transmission includes the radius of the X-ray tube R_T, which is needed only for the case where the X-ray tube focal line is used as *F*. In a typical instrument with X-ray tube source *F* and $r = 174$ mm, the transmission of Cu $K\alpha$ decreases from 90% at $40^\circ 4\theta$ to 62% at 210°, and Cr $K\alpha$ from 73 to 23% at the same angles.

Some of the advantages of the method include the following: (*a*) the fixed specimen makes it possible to simplify the design of specimen environment devices; (*b*) a large aperture can be used and the intensities are higher than for conventional diffractometers; (*c*) the flat-specimen aberration can be eliminated by a single-curvature specimen; (*d*) a small γ angle can be used to increase the path length *l* in the specimen, and hence the intensity of low-absorbing thin-film samples ($l = t/\sin\gamma$ and for $\gamma = 5^\circ$, $l = 11.5t$); (*e*) the method is useful in thin-film and preferred-orientation studies because about a 45° range of lattice-plane orientations can be measured and compared with conventional patterns. The limitations include (*a*) the more complicated diffractometer and its alignment, (*b*) limited angular range of about 10 to $110^\circ 2\theta$ for the forward-reflection setting, (*c*) extreme care required in specimen preparation, and (*d*) larger aberration errors.

2.3.1.4. *Reflection specimen, θ–θ scan*

In this geometry, the specimen is fixed in the horizontal plane and the X-ray tube and detector are synchronously scanned in the vertical plane in opposite directions above the centre of the specimen as shown in Fig. 2.3.1.14. The distances source to *S* and *S* to RS are equal to that the angles of incidence and diffraction and a constant $d\theta/dt$ are maintained over the entire angular range. A focusing monochromator can be used in the incident or diffracted beam. High- and low-temperature chambers are simplified because the specimen does not move. The arms carrying the X-ray tube and detector must be counterbalanced because of the unequal weights. The method has advantages in certain applications such as the measurement of liquid scattering without a covering window, high-temperature molten samples, and other applications requiring a stationary horizontal sample (Kaplow & Averbach, 1963; Wagner, 1969).

2.3.1.5. *Microdiffractometry*

There are two types of microdiffraction: (*a*) only a very small amount of powder is available, and (*b*) information is required from very small areas of a conventional-size specimen. Small-volume samples have been analysed with a conventional diffractometer by concentrating the powder over a small spot centred on a single-crystal plate such as silicon (510) or an

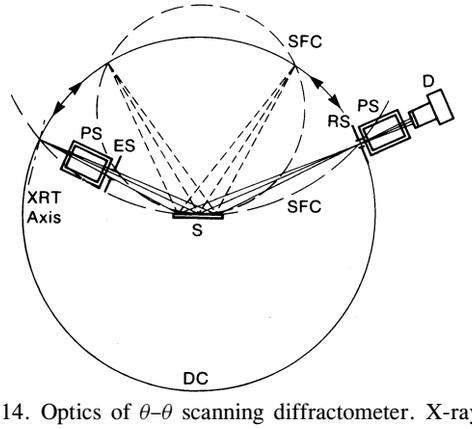

Fig. 2.3.1.14. Optics of θ–θ scanning diffractometer. X-ray tube and detector move synchronously in opposite directions (arrows) around fixed horizontal specimen. A focusing monochromator can be used after the receiving slit.

references