

## 2.3. POWDER AND RELATED TECHNIQUES: X-RAY TECHNIQUES

$$R_{\text{SFC}} = R_{\text{DC}}/2 \sin \theta. \quad (2.3.1.1)$$

The specimen holder is set parallel to the central ray at  $0^\circ$  and the gears drive the RS-detector arm at twice the speed of the specimen to maintain the  $\theta$ - $2\theta$  relation at all angles. The source  $F$  is the line focus of the X-ray tube viewed at a take-off angle  $\psi$ . The actual width,  $F'_w$ , is foreshortened to

$$F_w = F'_w \sin \psi. \quad (2.3.1.2)$$

In a typical case,  $F'_w = 0.4$  mm and, at  $\psi = 5^\circ$ ,  $F_w = 0.03$  mm and the projected angular width is  $0.025^\circ$  for  $R = 185$  mm. The angular aperture  $\alpha_{\text{ES}}$  of the incident beam in the equatorial (focusing) plane is determined by the entrance slit width  $\text{ES}_w$  (also called the 'divergence slit' since it limits the divergence of the beam) and its distance  $D_1$  from  $F$ :

$$\text{ES}_\alpha = 2 \arctan[(\text{ES}_w + F_w)/2D_1]. \quad (2.3.1.3)$$

Because the beam is divergent, the length of specimen irradiated  $S_l$  in the direction of the incident beam normal to  $O$  varies with  $\theta$ :

$$S_l = [\alpha(R - D')]/\sin \theta, \quad (2.3.1.4)$$

where  $\alpha$  is in radians and  $D'$  is the distance from  $F$  to the crossover point before ES and is given by  $F_w D_1 / (F_w + \text{ES})$ . The approximate relation

$$S_l = \alpha R / \sin \theta \quad (2.3.1.5)$$

is close enough for practical purposes (Parrish, Mack & Taylor, 1966). The intensity is nearly proportional to  $\text{ES}_\alpha$  but the maximum aperture that can be used is determined by  $S_l$  and the smallest angle to be scanned  $2\theta_{\text{min}}$ , as shown in Fig. 2.3.1.4. The entrance-slit width may be increased to obtain higher intensity at the upper angular range; for example,  $\text{ES} = 1^\circ$  for the forward-reflection region and  $4^\circ$  for back-reflection.

Some slit designs are shown in Fig. 2.3.1.5. The base is machined with a pair of rectangular shoulders whose separation  $A$  is the sum of the diameters of the two rods ( $a$ ) or bar widths ( $b$ ) and the central spacers on both ends that determine the slit opening. The distance  $P$  between the centre of the slit opening and the edge of the slit frame is kept constant for all slits to avoid angular errors when changing slits. The rods may be molybdenum or other highly absorbing metal and are cemented in

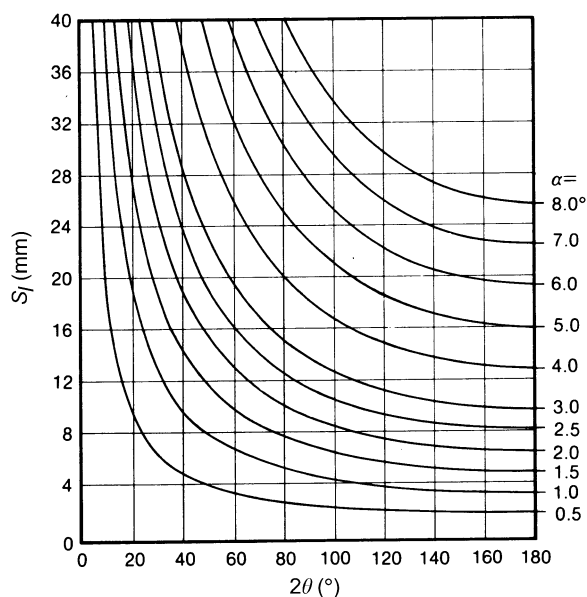


Fig. 2.3.1.4. Length of specimen irradiated,  $S_l$ , as a function of  $2\theta$  for various angular apertures.  $S_l = \alpha R / \sin \theta$ ,  $R = 185$  mm.

place. It may also be made in one piece ( $c$ ) using machinable tungsten (Parrish & Vajda, 1966).

A variable ES whose width increases with  $2\theta$  so that the irradiated length is about the same at all angles has been described by Jenkins & Paolini (1974). It is called a  $\theta$ -compensating slit in which a pair of semicircular cylinders with a fixed opening is rotated around the axis of the opening by a linkage attached to the specimen shaft of the diffractometer to vary the aperture continuously with  $\theta$ . The observed intensities must be corrected to obtain the relative intensities and the angular dependence of the aberrations is different from the fixed aperture slit.

Another way to irradiate constantly the entire specimen length is to use a self-centring slit which acts as an entrance and antiscatter slit (de Wolff, 1957). A 1 mm thick brass plate with rounded edge is mounted above the centre of the specimen and is moved in a plane normal to the specimen surface so that the aperture is proportional to  $\sin \theta$ . It can only be used for forward reflections.

Owing to the beam divergence, the geometric centre of the irradiated specimen length shifts a small amount during the scan (see also §2.3.5.1.5). It is generally advisable to centre the beam at the smallest  $2\theta$  to be scanned. Below about  $20^\circ$ , the irradiated length increases rapidly and it is essential to use small apertures and to align the entrance and antiscatter slits carefully. Failure to do this correctly could cause ( $a$ ) errors in the relative intensities owing to the primary beam exceeding the specimen area, ( $b$ ) cutoff by the walls of the specimen holder for low-absorbing thick specimens, and ( $c$ ) increased background from scattering by the specimen holder or the primary beam entering the detector. The transmission specimen method (Subsection 2.3.1.2) has advantages in measuring large  $d$ 's.

The beam converges after reflection on the receiving slit RS, whose width defines the reflection and profile width. Only those rays that are within the  $\theta$ - $2\theta$  setting are in sharp convergence, *i.e.* 'in focus'. The reflections become broader with increasing distance from the RS, and, therefore, this method is not suited for position-sensitive detectors. The RS aperture

$$\alpha_{\text{RS}} = 2 \arctan(\text{RS}_w/2R) \quad (2.3.1.6)$$

is the dominant factor in determining the intensity and resolution. For  $\text{RS}_w = 0.1$  mm and  $R = 185$  mm,  $\alpha_{\text{RS}} = 0.031^\circ$ .

Antiscatter slits AS are slightly wider than the beam and are essential in this and other geometries to make certain the detector

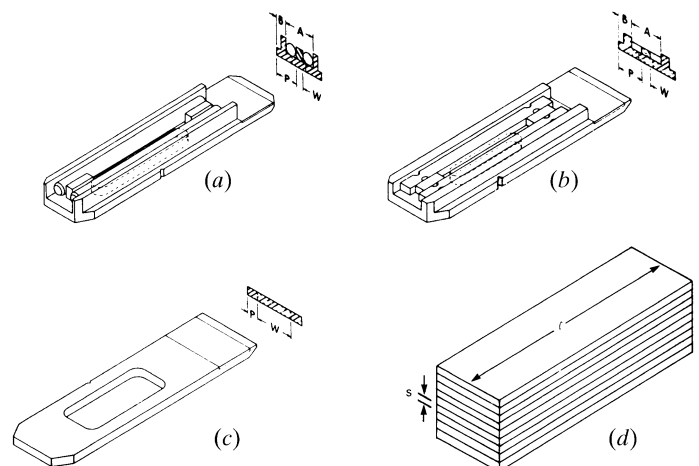


Fig. 2.3.1.5. Slit designs made with ( $a$ ) rods, ( $b$ ) bars, and ( $c$ ) machined from single piece. ( $d$ ) Parallel (Soller) slits made with spacers or slots cut into the two side pieces (not shown) to position the foils.