

## 2. INSTRUMENTATION AND SAMPLE PREPARATION

(2006), Fewster (2003), Bowen & Tanner (1998), Jenkins & Snyder (1996), Klug & Alexander (1974), and Peiser *et al.* (1955). An extensive discussion of the principles of combining X-ray optics to optimally suit a wide range of different powder diffraction as well as thin-film applications has been given in the textbook by Fewster (2003).

## 2.1.6.3.1. Absorptive X-ray optics

## 2.1.6.3.1.1. Apertures

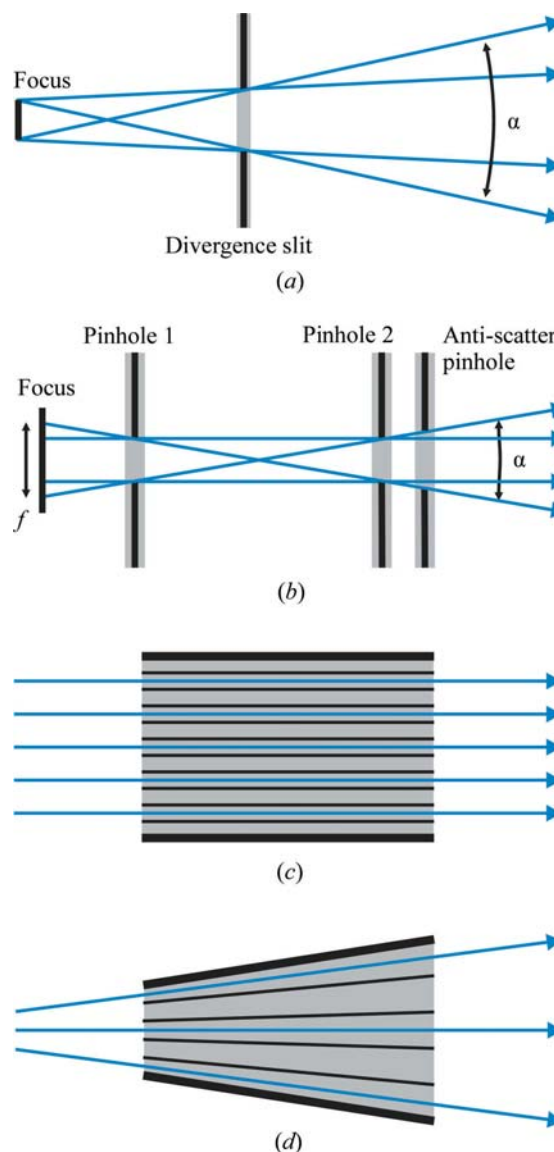
The simplest way of beam conditioning is to place apertures such as slits (line focus) or pinholes (point focus) into the incident and/or diffracted beam to control beam divergence and shape, and to reduce unwanted scattering from air or any beam-path components. Apertures are 'shadow-casting' optics and thus cannot increase flux density. Reducing beam divergence and beam dimensions by means of apertures invariably results in a loss of intensity that is inversely proportional to the slit aperture.

The principles are shown in Fig. 2.1.14. The divergence of a beam is established by the dimensions of the focal spot as well as the aperture and the distance of the aperture from the source (Fig. 2.1.14*a*). The divergence in the diffraction plane is usually called 'equatorial divergence' and the divergence in the axial direction 'axial divergence'. Apertures can be of the plug-in type requiring manual changes of the aperture to obtain different divergence angles, or – usually only for equatorial divergence slits – motorized. Motorized slits are mostly used in the Bragg–Brentano geometry to limit equatorial divergence, which can be arbitrarily chosen and either be kept constant to keep the diffracting specimen volume constant (as is invariably the case with plug-in slits), or varied as a function of  $2\theta$  to keep the illuminated specimen length constant. Typical aperture angles range from  $0.1$ – $1^\circ$ .

To provide additional collimation, a second aperture may be placed at some distance away from the first (Fig. 2.1.14*b*). When using the same aperture, an almost-parallel beam may be obtained from a divergent beam at the cost of high intensity losses. A third aperture is often used to reduce scattering by the second slit. In laboratory X-ray diffractometers dedicated for SAXS analysis such collimation systems may reach lengths of more than 1 m.

Another way to parallelize radiation is to use a parallel-plate collimator (PPC), which is manufactured from sets of parallel, equally spaced thin metal plates, as shown in Fig. 2.1.14(*c*). Each pair of neighbouring plates works like a double-aperture arrangement as shown in Fig. 2.1.14(*b*). In contrast to simple slits and pinholes, PPCs do not change the shape of the beam. PPCs arranged parallel to the diffraction plane are usually called 'Soller slits' and are used to control axial divergence. Such devices can be used for focusing as well as parallel-beam geometries with typical aperture angles ranging from  $1$ – $5^\circ$ . Soller slits are usually mounted on both the incident- and diffracted-beam sides of the specimen. PPCs arranged parallel to the diffraction plane are specifically used in parallel-beam geometries to minimize equatorial beam divergence, with typical aperture angles ranging from  $0.1$ – $0.5^\circ$ .

The ways in which the diffracted beam can be conditioned are limited when employing one- or two-dimensional detectors. A particular issue related to these types of detectors is unwanted scattering from air or any beam-path components. Ideally, a closed, evacuated or He-flushed beam path will be used, but this is often not feasible owing to collision issues. For smaller detectors it is possible to place the anti-scatter aperture closer to the



**Figure 2.1.14**

Apertures used for beam collimation.  $\alpha$ : divergence angle,  $f$ : virtual focus. (*a*) Single slit or pinhole, (*b*) parallelization through double slits or pinholes, (*c*) parallelization through a parallel-plate collimator, (*d*) a radial plate collimator.

specimen surface. Alternatively, a knife edge may be placed on top of the specimen. As knife edges may interfere with divergent beams at higher  $2\theta$  angles, it is necessary to move them away from the specimen at higher  $2\theta$  angles. Another possibility, limited to one-dimensional detectors, is to use radial Soller slits as shown in Fig. 2.1.14(*d*).

## 2.1.6.3.1.2. Metal filters

Metal filters are the most frequently used devices for monochromatization of X-rays in laboratory diffractometers. Metal filters represent single-band bandpass devices where monochromatization is based on the  $K$  absorption edge of the filter material to selectively allow transmission of the  $K\alpha$  characteristic lines while filtering white radiation,  $K\beta$  radiation (hence they are frequently known as ' $K\beta$  filters'), and other characteristic lines.

A properly selected metal filter has its  $K$  absorption edge right between the energies of the  $K\alpha$  and  $K\beta$  characteristic lines of the source. As a rule of thumb, this is achieved by choosing an element just one atomic number less than the X-ray source target material in the periodic table. For heavy target materials such as