

## 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

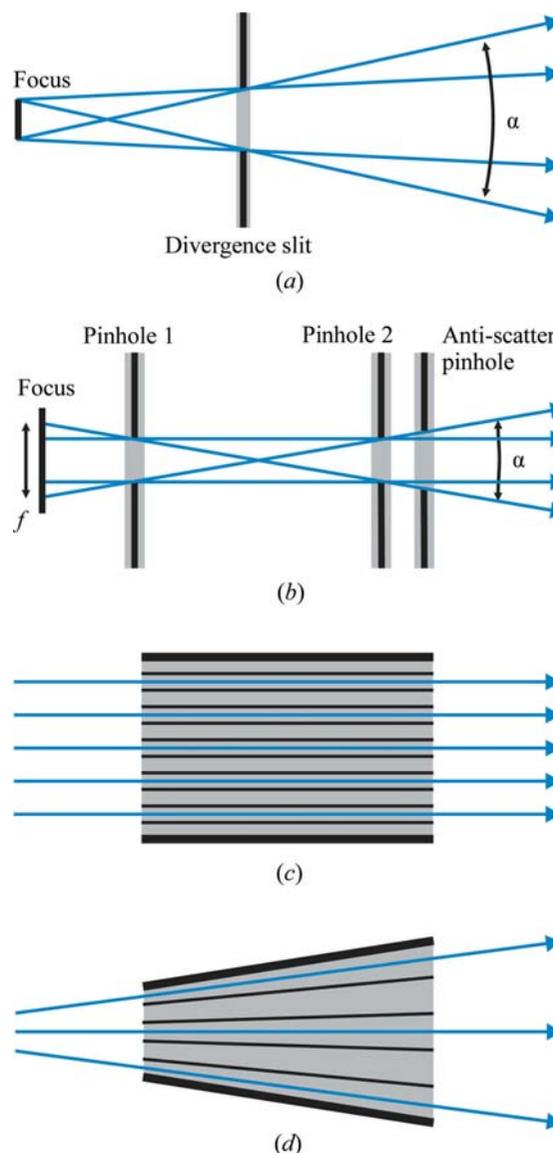
The simplest way of beam conditioning is to place apertures such as slits (line focus) or pinholes (point focus) into the incident and/or diffracted beam to control beam divergence and shape, and to reduce unwanted scattering from air or any beam-path components. Apertures are ‘shadow-casting’ optics 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.

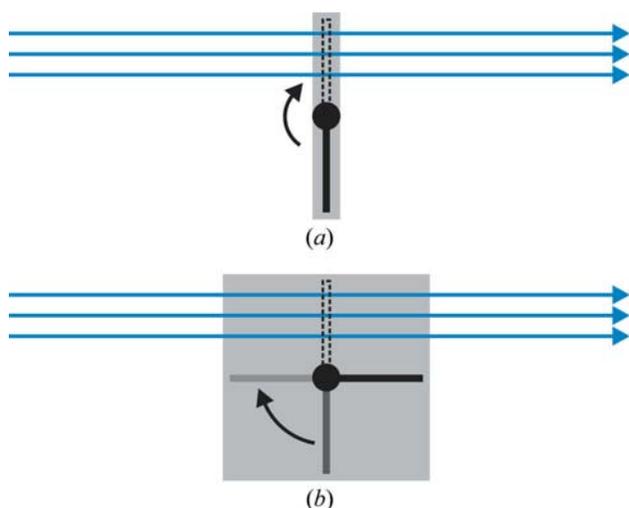
specimen surface. Alternatively, a knife edge may be placed on top of the specimen. As knife edges may interfere with divergent beams at higher  $2\theta$  angles, it is necessary to move them away from the specimen at higher  $2\theta$  angles. Another possibility, limited to one-dimensional detectors, 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

## 2.1. LABORATORY X-RAY SCATTERING



**Figure 2.1.15**  
Motorized switchable (a) and rotating (b) absorbers.

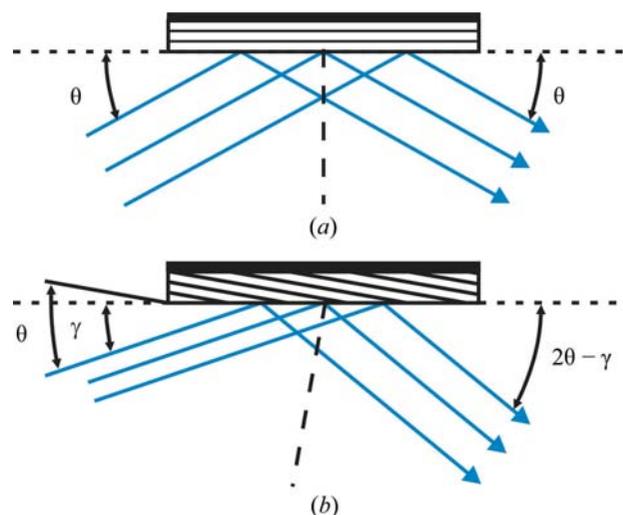
Mo or Ag, this rule can be extended to two atomic numbers. A list of metal filters suitable for the most commonly used target materials is given in Table 2.1.3.

A major disadvantage of metal filters is that they cannot completely eliminate  $K\beta$  radiation at bearable intensity losses. In addition, they introduce absorption edges at the high-energy (low-angle) side of diffraction peaks, the magnitudes of these being dependent on the wavelength as well as on the filter material and its thickness. While for point detectors absorption edges are usually obscured by counting statistics, they are much more readily visible to position-sensitive detectors owing to the high number of counts that are typically collected.

Positioning of the metal filter does not make a difference in terms of filtering of  $K\beta$  or white radiation, but can in the case of specimen fluorescence. Placing the metal filter in the diffracted beam can filter some fluorescence radiation, unless the specimen contains the same element as the metal filter. Taking Cu radiation as an example, most fluorescence radiation excited by Ni in the specimen will pass through a diffracted-beam Ni filter. In this instance, the  $K\beta$  filter should be mounted in the incident beam to suppress Cu  $K\beta$  radiation, which is very efficient at exciting Ni fluorescence. Balanced-filter techniques, employing two (or more) filters that have absorption edges just above and below  $K\alpha$ , are no longer in use as the resulting bandpass is still much wider than that of crystal monochromators at even higher intensity losses.

Metal filters are generally supplemented by some energy discrimination by the detector to remove the high-energy white radiation from the X-ray source. The effectiveness of this white-radiation removal depends upon the energy resolution of the detector, and is discussed in Section 2.1.7 for the different detector technologies currently in use. Recent improvements in the energy-discrimination capabilities for silicon strip detectors now even allow filtering of  $K\beta$  radiation, completely eliminating the need for metal filters (see Section 2.1.7.2.3.2). As a consequence, the use of metal filters is likely to decline.

Another type of metal filter is represented by absorbers, *e.g.* Cu foils, which are used at high intensities to avoid detector saturation or even damage. Absorbers can be motorized and switched in and out automatically depending on the actual count rates that are detected (Fig. 2.1.15a). Several absorbers with different thickness may be combined in the form of motorized rotating absorbers (Fig. 2.1.15b).



**Figure 2.1.16**  
Illustration of flat single-reflection monochromators. (a) Symmetrically cut crystal, (b) asymmetrically cut crystal with an angle  $\gamma$  between the reflecting lattice planes and the crystal surface.

### 2.1.6.3.2. Diffractive X-ray optics

Single crystals or highly textured polycrystals (mosaic crystals) represent effective beam conditioners by allowing the spectral bandwidth as well as the X-ray beam divergence to be modified. When they are placed at a specific angle with respect to the incident and diffracted beams, according to Bragg's law, only a small spectral bandwidth will be transmitted depending on the divergence of the incident beam and the rocking angle (mosaic spread) of the crystal. Higher harmonics ( $\lambda/2$ ,  $\lambda/3$ , ...) are diffracted as well, but can be successfully suppressed by using materials with small higher-order structure factors and *via* energy discrimination by the detector. Depending on the application, a crystal monochromator can be either used as a spectral filter ('monochromator'), typically used in the incident beam, or as an angular filter ('analyser'), typically used in the diffracted beam to restrict the angular acceptance of the detector.

It is likely that all monochromators currently employed in laboratory X-ray diffractometers are of the reflective type ('Bragg geometry'). Transmission-type monochromators ('Laue geometry') play no role in laboratory powder diffraction. Two designs are in common use and are described below: (a) single-reflection monochromators and (b) multiple-reflection monochromators.

#### 2.1.6.3.2.1. Single-reflection monochromators

The most common types of single-reflection monochromators are illustrated in Figs. 2.1.16 and 2.1.17. Flat crystals (Fig. 2.1.16) are used in parallel-beam geometry and curved crystals in focusing geometries (Fig. 2.1.17). A beam reflected from a flat crystal with the reflecting lattice planes parallel to its surface (symmetric cut) is nearly parallel (Fig. 2.1.16a). If the crystal is cut at an angle to the reflecting lattice planes (asymmetric cut), then the beam will be expanded (Fig. 2.1.16b), or compressed if reversed (Fankuchen, 1937). Monochromators can be curved (Johann, 1931) or curved and ground (Johannsson, 1933), and may be cut symmetrically (Fig. 2.1.17a) or asymmetrically (Fig. 2.1.17b). The latter has the particular advantage of providing different focal lengths for the incident and diffracted beam. A shortened incident beam allows the monochromator to be mounted closer to the X-ray source to capture a larger solid angle of the emitted beam. If the diffracted-beam focusing length is