

2.1. LABORATORY X-RAY SCATTERING

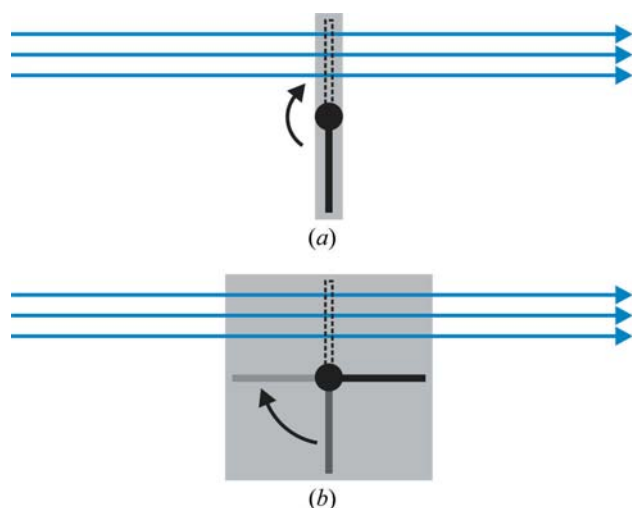


Figure 2.1.15
Motorized switchable (a) and rotating (b) absorbers.

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).

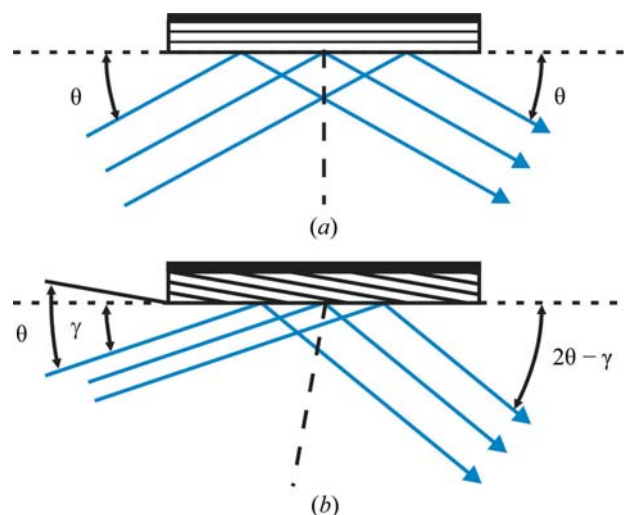


Figure 2.1.16
Illustration of flat single-reflection monochromators. (a) Symmetrically cut crystal, (b) asymmetrically cut crystal with an angle γ between the reflecting lattice planes and the crystal surface.

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

2. INSTRUMENTATION AND SAMPLE PREPARATION

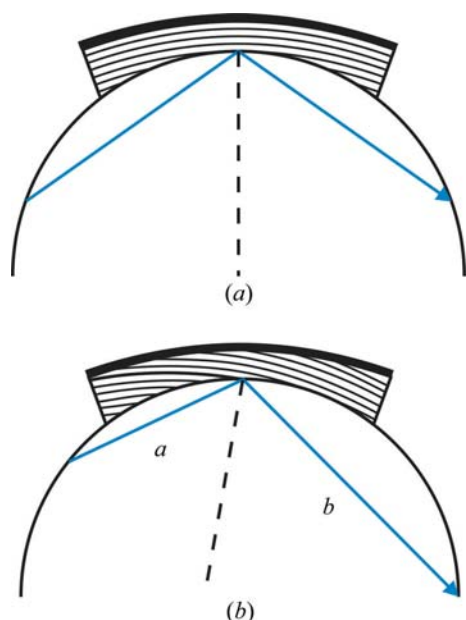


Figure 2.1.17
Illustration of curved and ground single-reflection monochromators. Only the central beam is shown for clarity. (a) Symmetrically cut crystal, (b) asymmetrically cut crystal with two different focal lengths a and b .

sufficiently large, then the instrument geometry can be converted between the Bragg–Brentano and the focusing Debye–Scherrer geometries by shifting the monochromator crystal and the X-ray source along the incident-beam X-ray optical bench (see Section 2.1.4.1 and Fig. 2.1.3).

The most commonly used monochromator crystal materials are germanium and quartz, which have very small mosaic spreads and are able to separate the $K\alpha_1/K\alpha_2$ doublet. In contrast to germanium and quartz crystals, graphite and lithium fluoride have large mosaic spreads and thus high reflectivity, but cannot suppress $K\alpha_2$. In principle, any of these monochromators can be mounted in the incident as well as the diffracted beam; the choice mostly depends on the purpose of the monochromator. Germanium and quartz monochromators are typically used as incident-beam monochromators to produce pure $K\alpha_1$ radiation. Graphite

(focusing geometries) and lithium fluoride (parallel-beam geometry) are often used as diffracted-beam monochromators to suppress fluorescence radiation. Germanium and quartz can also be used as diffracted-beam monochromators, but are usually not because of their lower reflectivity. Where mounting of diffracted-beam monochromators is difficult or impossible, which is specifically true for one- and two-dimensional detector applications, curved graphite monochromators are frequently used as incident-beam monochromators.

The use of diffracted-beam monochromators – at least in powder X-ray diffraction – is declining steeply because of the geometric incompatibility issues with one- and two-dimensional detector systems (which, since 2010, have been sold with more than 90% of all diffractometers; see Section 2.1.3.2). With the recent improvements of energy-discrimination capabilities for silicon micro-strip detectors, the need for diffracted-beam monochromators will further diminish (see Section 2.1.7.2.3).

2.1.6.3.2.2. Multiple-reflection monochromators

Multiple-reflection monochromators can reduce the wavelength dispersion $\Delta\lambda/\lambda$ significantly more than single-reflection monochromators. Multiple-reflection monochromators are often made of monolithically grooved single crystals and are also known as channel-cut monochromators (Bonse & Hart, 1965). In Fig. 2.1.18 an overview is given of the most common channel-cut monochromator types; for a detailed discussion see *e.g.* Hart (1971) and Bowen & Tanner (1998). Successive reflection of the X-ray beam at the channel walls by the same lattice planes causes a strong reduction of the X-ray intensity contained in the tails of the beam. Depending on the number of reflections, multiple-reflection monochromators are denoted as two-bounce, three-bounce *etc.* channel-cut monochromators. The Bartels monochromator (Bartels, 1983) comprises two two-bounce channel-cut crystals. For Cu radiation, such a monochromator results in a wavelength spread which is less than the natural line width of the Cu $K\alpha_1$ line. The most commonly used crystal material is germanium, which delivers higher intensity than silicon, using the 400, 220, or 440 reflections. Crystals may be cut symmetrically or asymmetrically. In Table 2.1.5 several types of

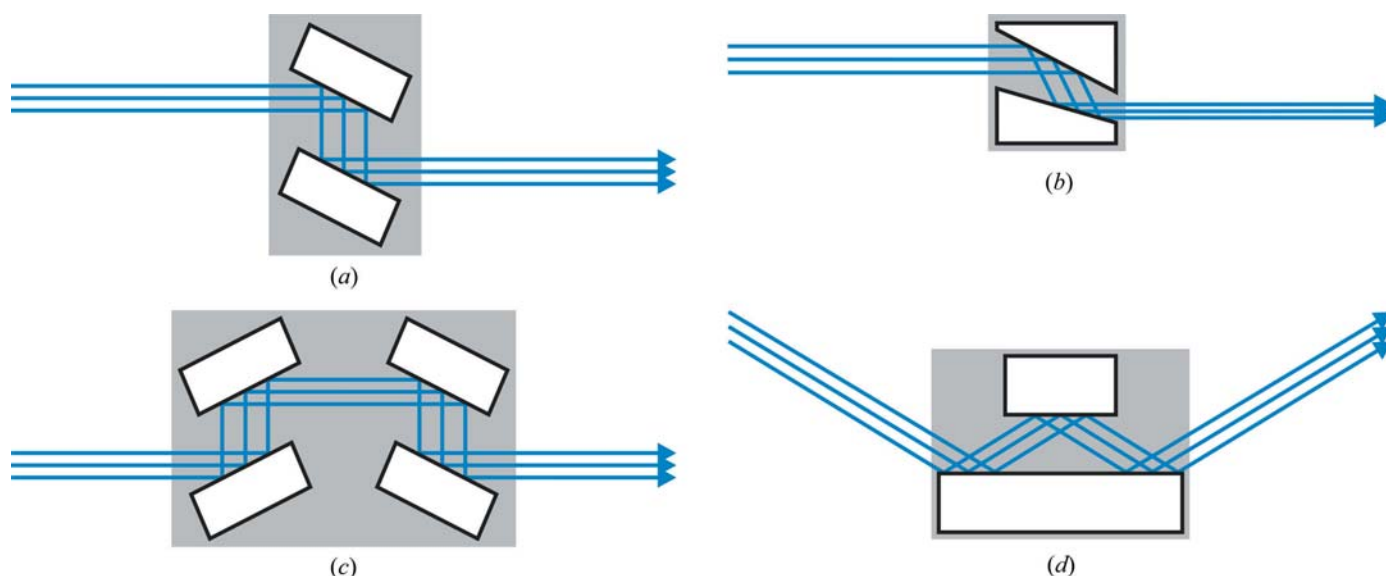


Figure 2.1.18
Illustration of multiple-reflection monochromators. (a) Symmetrically cut two-bounce channel-cut monochromator, (b) asymmetrically cut two-bounce channel-cut monochromator for beam compression, or, if reversed, for beam expansion, (c) symmetrically or asymmetrically cut four-bounce channel-cut monochromator, (d) symmetrically cut three-bounce channel-cut monochromator.

Table 2.1.5

Comparison of divergence and intensity for several types of germanium channel-cut monochromators

In each case, the monochromator is coupled with a graded multilayer providing 3×10^9 counts per second at $<0.028^\circ$ beam divergence. The values in parentheses denote the percentage of intensity diffracted by the respective monochromator crystals.

Type	(<i>hkl</i>)	Divergence ($^\circ$)	Intensity
Two-bounce	220, symmetric	<0.0052	5.0×10^7 (~1.5%)
Two-bounce	220, asymmetric	<0.0085	3.3×10^8 (~10%)
Two-bounce	400, asymmetric	<0.0045	4.8×10^7 (~1.5%)
Four-bounce	220, symmetric	<0.0035	6.5×10^6 (~0.2%)
Four-bounce	220, asymmetric	<0.0080	2.7×10^7 (~1%)
Four-bounce	440, symmetric	<0.0015	2.2×10^5 (~0.075%)

germanium channel-cut monochromators are compared in terms of divergence and intensity.

Switching between different channel-cut monochromators is extremely easy these days and can be accomplished without the need for any tools and without realignment. This is also true for cases where a beam offset is introduced, *e.g.* by switching between two- and four-bounce channel-cut monochromators. In sophisticated instruments such an offset can be compensated fully automatically by a software-controlled motor.

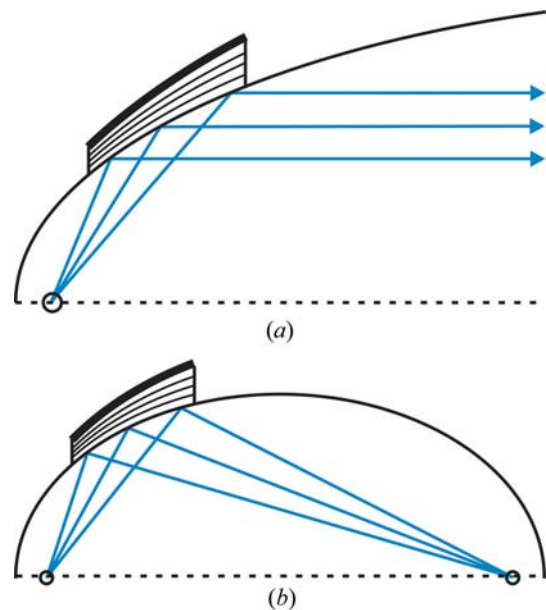
The combination of different types of channel-cut monochromators in both the incident and diffracted beam allows the construction of advanced diffractometer configurations with extremely high resolution capabilities. It should be emphasized that laboratory X-ray diffractometers can have identical optical configurations to diffractometers operated at synchrotron beamlines. The important and obvious difference, however, is the extremely low flux coming from laboratory X-ray sources, which is further diminished by each reflection in a channel-cut monochromator (Table 2.1.5). While such configurations work perfectly for strongly scattering single-crystal layers in thin films, for example, analysis of ideal powders is normally not possible.

2.1.6.3.3. Reflective X-ray optics

2.1.6.3.3.1. Multilayer mirrors

Multilayer mirrors used in laboratory X-ray diffractometers are efficient beam conditioners, using total reflection as well as Bragg reflection on inner interfaces of a multilayer structure to modify beam divergence, cross-section size, shape and – to some extent – spectral bandwidth. A comprehensive description of current mirror designs and important mirror properties is found in the VDI/VDE Guideline 5575 Part 4 (2011).

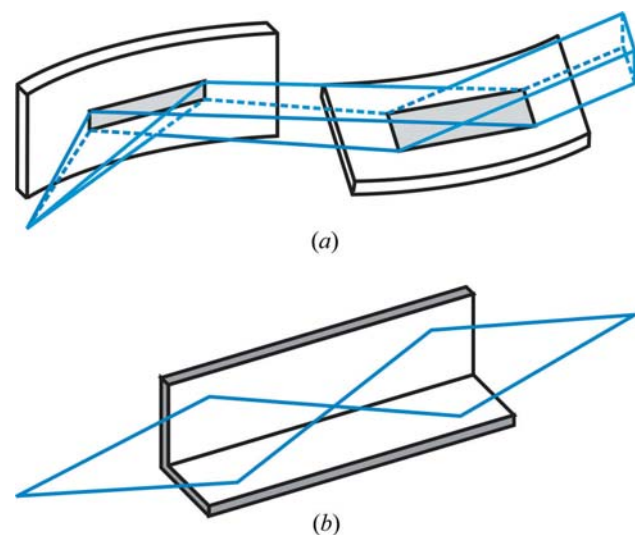
Multilayer mirrors consist of a multilayer coating deposited on a flat or curved substrate. The *imaging* characteristics are mostly determined by the contour of the mirror surface as defined by the substrate surface. The most common contours include planes, ellipsoids, paraboloids, elliptic cylinders or parabolic cylinders. The *spectral reflection* properties are determined by the coating, which may consist of some 10 up to 1000 alternating layers of amorphous low-density ('spacer') and high-density ('reflector') materials, with a period of a few nanometres. The first curved mirrors were produced by depositing the multilayers on a flat substrate that was subsequently bent to the desired contour, yielding typical r.m.s. slope errors of about 15 arcsec. By using prefigured substrates with r.m.s. slope errors below 1.7 arcsec, significantly improved reflectivity and lower beam divergence are obtained. Laterally graded multilayer mirrors (so-called 'Göbel mirrors') have a layer thickness gradient parallel to the surface

**Figure 2.1.19**

Schematic of graded multilayer mirrors. (a) Parabolic mirror for parallelization of a divergent beam, or, if reversed, focusing of a parallel beam. In the latter case the mirror will also filter some specimen fluorescence. (b) Elliptical mirror for focusing of a divergent beam.

(Schuster & Göbel, 1996), which, when combined with a planar, parabolic or elliptic substrate contour, produce a divergent, parallel or focusing beam. Fig. 2.1.19 illustrates graded multilayer mirrors for parallelization and focusing in the plane of diffraction.

For beam conditioning in two perpendicular directions, two perpendicularly oriented curved mirrors may be used, as illustrated in Fig. 2.1.20. In the Kirkpatrick–Baez scheme (Kirkpatrick & Baez, 1948), two mirrors are cross-coupled as shown in Fig. 2.1.20(a). This setup has some issues related to the inherently different capture angles and magnification of both mirrors, resulting in less flux from smaller sources and in different divergences in both directions for elliptical mirrors. The Montel optics (Montel, 1957) shown in Fig. 2.1.20(b) overcome these issues by arranging both mirrors in a 'side-by-side' configuration.

**Figure 2.1.20**

Examples for orthogonally positioned curved mirrors for beam conditioning. (a) Kirkpatrick–Baez scheme employing two parabolic mirrors to create a parallel beam, (b) Montel optics employing two elliptical mirrors side-by-side to create a focusing beam.