

2.1. LABORATORY X-RAY SCATTERING

filament on the anode and on the inner beryllium window surfaces leads to spectral contamination and substantial loss of intensity with time. Increasing deterioration of the filament may change its position relative to the electrostatic lens used for focusing and result in beam inhomogeneity and additional intensity losses. Further intensity losses and beam inhomogeneity may arise from pitting of the anode surface as a result of the intense electron flux on the anode surface.

2.1.6.2.2.2. Moving-target X-ray sources

(a) *Rotating-target X-ray sources.* Rotating-target X-ray sources are able to remove heat more efficiently than fixed-target sources, and can thereby sustain higher fluxes of X-ray photons (Fig. 2.1.13b). This is achieved by rotating a cooled anode, with a typical diameter ranging from about 10 to 30 cm, at about 6000–12 000 revolutions per minute. The maximum power loads depend on the focal spot size, and can range up to 18 kW for conventional rotating-target X-ray sources, and 3 kW for micro-focus rotating-target X-ray sources. Rotating-target X-ray sources are thus inherently more brilliant, and gain up to an order of magnitude in brilliance compared to their respective fixed-target counterparts.

Rotating-target systems do require routine maintenance such as periodic anode refurbishment and changes of the filament, bearings and seals. The maintenance requirements of micro-focus systems are significantly lower than those of conventional rotating-target systems because of the lower total power loading.

(b) *Liquid-metal-jet X-ray sources.* A very recent development is that of liquid-metal-jet micro-focus X-ray sources (Fig. 2.1.13c), where a jet of liquid metal acts as the electron-beam target (Hemberg *et al.*, 2003). A thin (<100–225 μm) high-speed (>50 m s^{-1}) liquid-metal jet is injected into vacuum by applying a backing pressure of about 200 bar and is targeted by a focused electron beam with a beam power of up to 200 W and a focal spot size of down to 6 μm . The focal spot is viewed at a take-off angle of about 45° to obtain a symmetric beam usually coupled into Montel optics. (Montel optics are described in Section 2.1.6.3.3.1.)

Ideal materials for use in liquid-jet anodes are electrically conductive to avoid charging and have low vapour pressure to simplify vacuum operation. Among a few materials currently being evaluated, Galinstan (a eutectic mixture of 68.5% Ga, 21.5% In and 10% Sn by weight) is particularly suited for laboratory X-ray analyses, as it is liquid at room temperature (melting point 254 K), with the most intense Ga $K\alpha$ line at 9.25 keV, and less intense In $K\alpha$ and Sn $K\alpha$ lines at 24 and 25.3 keV, respectively.

The obvious advantage of a metal-jet anode is that the maximum electron-beam power density can be significantly increased compared to solid-metal anodes and thus the brilliance can be increased by up to an order of magnitude.

2.1.6.2.3. Performance of X-ray sources

The single most important property of an X-ray source is its brilliance, which is proportional to the maximum target loading per unit area of the focal spot, also referred to as the specific loading.

In Table 2.1.4 the maximum target loading and specific loading (relative brilliance) for some typical sealed tubes and some rotating-anode sources with a Cu target are compared. Also listed are data for the liquid-metal jet with Ga as a target. Micro-

Table 2.1.4

Maximum target loading and specific loading for some selected fixed- and moving-target X-ray sources

X-ray source	Focal spot (mm^2)	Maximum load (kW)	Specific loading (kW mm^{-2})
Fixed target			
Broad focus (Cu)	2×10	3	0.15
Normal focus (Cu)	1×10	2.5	0.25
Long fine focus (Cu)	0.4×12	2.2	0.5
Micro-focus (Cu)	0.01–0.05	<0.05	5–50
Moving target			
Rotating anode (Cu)	0.5×10	18	3.6
	0.3×3	5.4	6
	0.2×2	3	7.5
	0.1×1	1.2	12
Micro-focus rotating anode (Cu)	0.1	2.7	27
Liquid-metal jet (Ga)	0.02×0.02	0.2	>500

focus fixed-target X-ray sources have up to two orders of magnitude higher specific loadings compared to conventional fixed target tubes, and even 2 to 5 times higher specific loadings compared to conventional rotating-anode systems. In contrast to fixed-target micro-focus X-ray sources, where the specific loading can only be increased by reducing the source size, moving-target X-ray sources are also made brighter by increasing the speed of the target relative to the electron beam. Moving-target X-ray sources are thus inherently brighter than stationary targets. The liquid-gallium jet has a higher (by a further order of magnitude) specific loading than the most brilliant rotating-anode systems, and now rivals the intensity of second-generation synchrotron beamlines.

2.1.6.3. X-ray optics

The purpose of X-ray optical elements is to condition the beam emitted by an X-ray source in terms of desired wavelength spread, divergence, cross-section size and shape, and to conserve as much intensity as possible. X-ray optics currently employed in laboratory X-ray diffractometers may be classified as absorptive, diffractive and reflective, as shown in Table 2.1.1.

Absorptive and diffractive X-ray optics represent selective beam-conditioning techniques, where parts of the beam are eliminated to achieve a particular wavelength distribution and divergence. In contrast to this, reflective optics modify the beam divergence to direct the full beam to the specimen or to the detector. The extremely large number of X-ray optical elements available allows for an enormous range of incident and diffracted beam-path configurations. Choosing the most appropriate X-ray optics and X-ray optics combination for a particular experiment is a challenge for the user. The general rule to be obeyed in order to obtain the best data quality is that the beam dimension, wavelength distribution and divergence should compare to the specimen dimension and angular spread of the structural features to be resolved.

In this section the most common features of X-ray optics in current use will be discussed. A comprehensive survey cannot be given, since there exists an incredible multitude of variants of the basic X-ray optic types listed in Table 2.1.1. X-ray optics have been extensively covered in the literature, for example in *International Tables for Crystallography* Vol. C (2004) and in the textbooks by He (2009), Pecharsky & Zavalij (2009), Paganin

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(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 2θ to keep the illuminated specimen length constant. Typical aperture angles range from 0.1 – 1° .

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° . 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° .

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

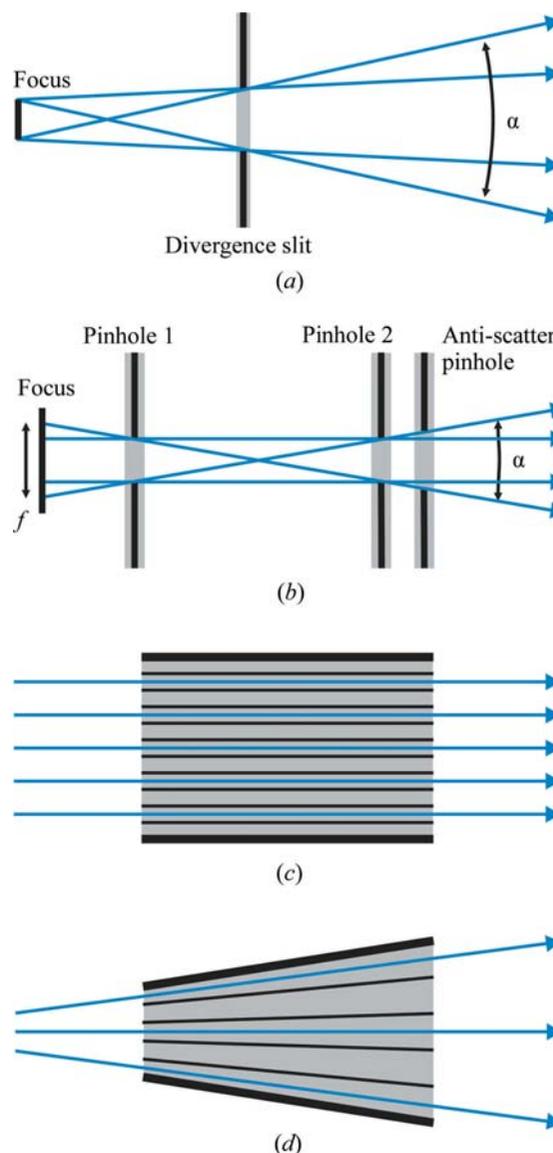


Figure 2.1.14

Apertures used for beam collimation. α : 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 2θ angles, it is necessary to move them away from the specimen at higher 2θ 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

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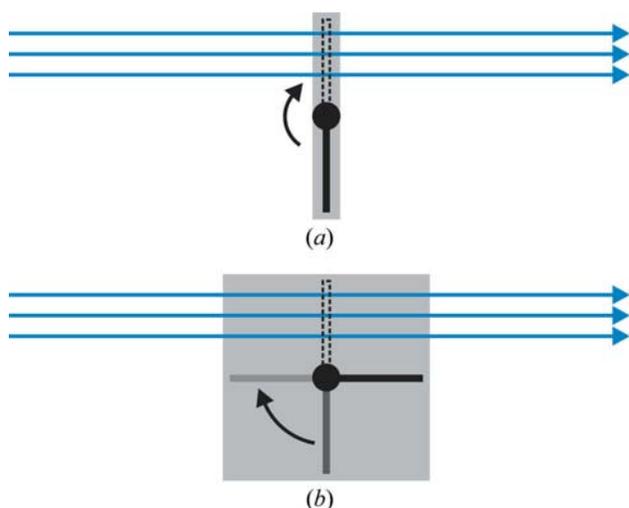


Figure 2.1.15
Motorized switchable (a) and rotating (b) absorbers.

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).

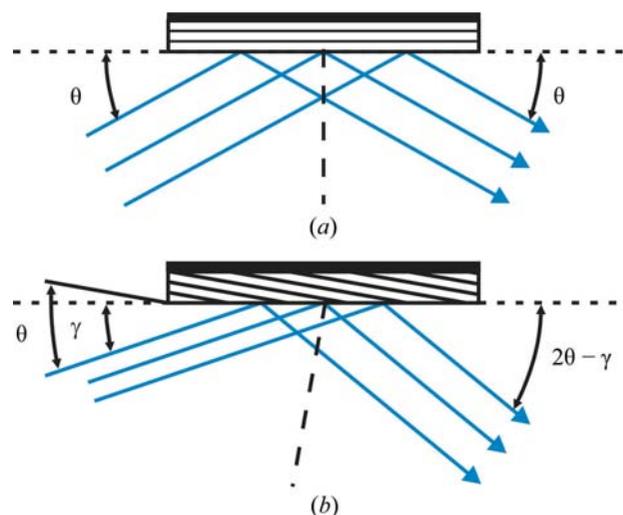


Figure 2.1.16
Illustration of flat single-reflection monochromators. (a) Symmetrically cut crystal, (b) asymmetrically cut crystal with an angle γ between the reflecting lattice planes and the crystal surface.

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

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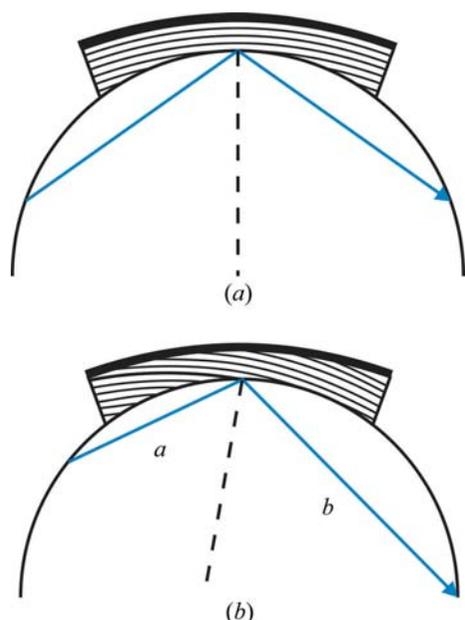


Figure 2.1.17
Illustration of curved and ground single-reflection monochromators. Only the central beam is shown for clarity. (a) Symmetrically cut crystal, (b) asymmetrically cut crystal with two different focal lengths a and b .

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

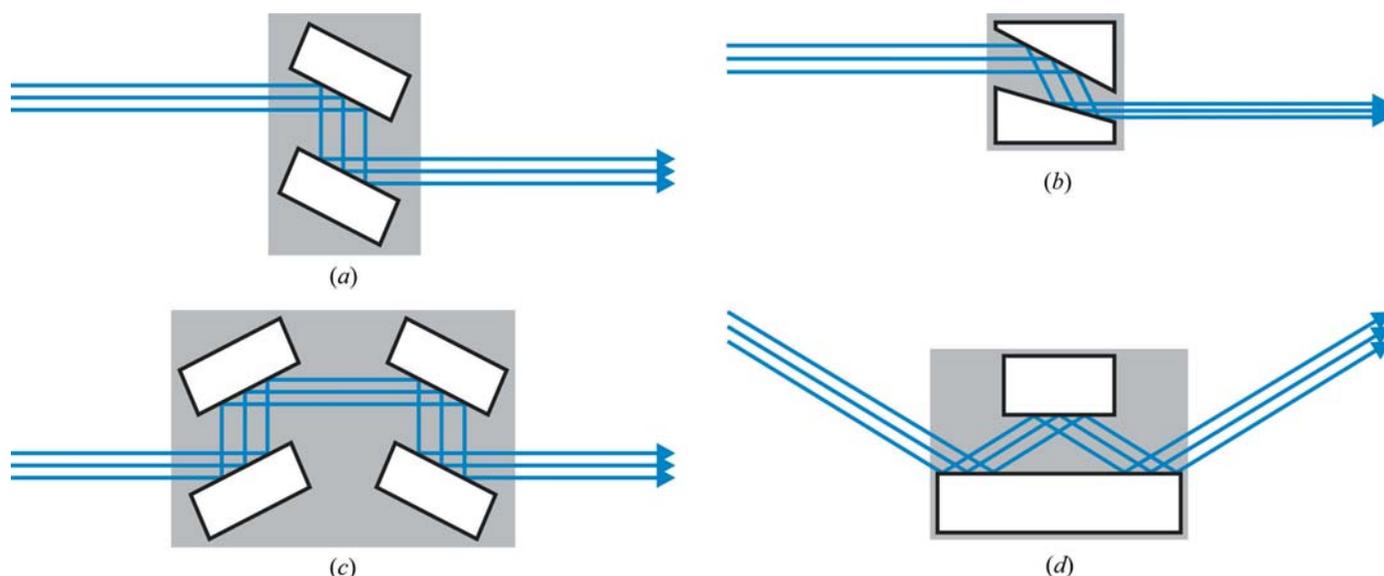


Figure 2.1.18
Illustration of multiple-reflection monochromators. (a) Symmetrically cut two-bounce channel-cut monochromator, (b) asymmetrically cut two-bounce channel-cut monochromator for beam compression, or, if reversed, for beam expansion, (c) symmetrically or asymmetrically cut four-bounce channel-cut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

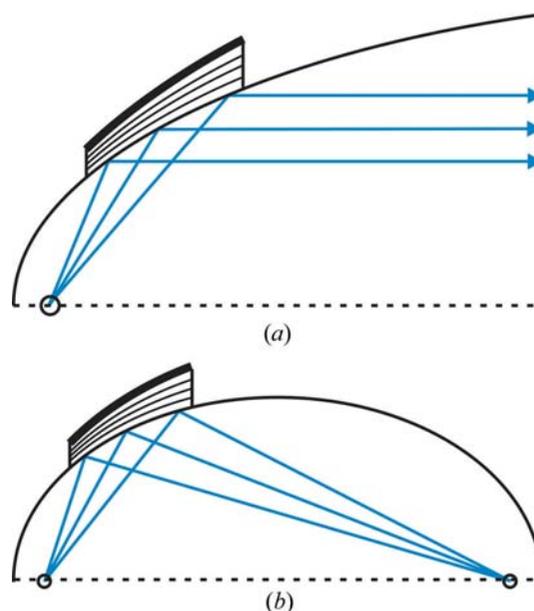
The combination of different types of channel-cut monochromators in both the incident and diffracted beam allows the construction of advanced diffractometer configurations with extremely high resolution capabilities. It should be emphasized that laboratory X-ray diffractometers can have identical optical 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).

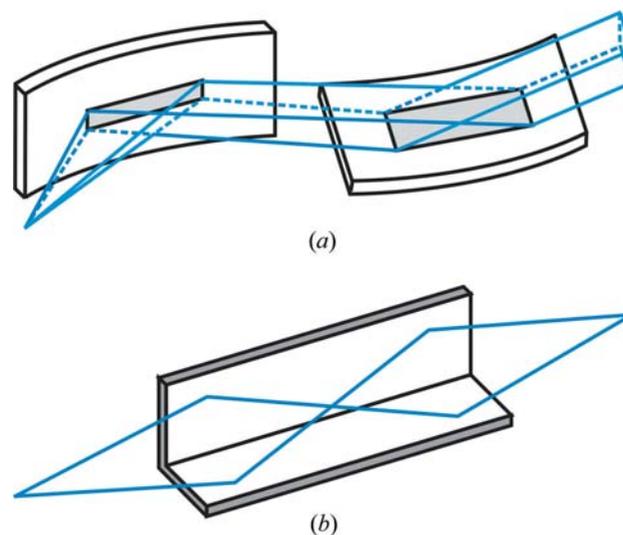
Multilayer mirrors consist of a multilayer coating deposited on a flat or curved substrate. The *imaging* characteristics are mostly determined by the contour of the mirror surface as defined by the substrate surface. The most common contours include planes, ellipsoids, paraboloids, elliptic cylinders 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.

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Mirrors are available for all characteristic wavelengths used in laboratory X-ray powder diffractometers. A wealth of different materials are being used as double layers (reflector/spacer), including but not limited to W/Si, W/B₄C, Ni/C, Ru/B₄C, Ti/B₄C, V/B₄C, Cr/B₄C and Mo/B₄C. The double-layer materials may be selected according to the energies of their absorption edges to make the mirror act as a filter as well. While none of these mirrors is strictly speaking a monochromator, appropriate selection of the double-layer materials, depending on the wavelength used, will allow monochromatization of the radiation to $K\alpha$ while $K\beta$ and *Bremsstrahlung* are suppressed.

Within the past two decades mirror systems have become invaluable for all applications requiring a small and/or highly parallel beam. In particular, coupling of a parallel-beam mirror with multiple-reflection channel-cut monochromators allows the use of a wider solid-angle range of the X-ray source and a gain of nearly two orders of magnitude in intensity (Schuster & Göbel, 1995). For applications requiring ideal powders, however, too-small as well as too-parallel beams may result in too small a number of diffracting crystallites, which will generally reduce the diffracted intensity, and may additionally lead to particle statistics errors (see also Section 2.1.6.1).

Today, advanced sputtering techniques allow the fabrication of a wealth of different multilayer optics with virtually arbitrary beam divergences to generate focusing, parallel and divergent beams, for both point- and line-focus applications. The most comprehensive overview of currently available mirrors and up-to-date specifications will be found in manufacturers' brochures.

2.1.6.3.3. Capillaries

X-ray capillary optics are finding increasing use in applications where a small focused beam with high intensity is required. Their design, important properties and applications are discussed by e.g. Bilderback (2003), He (2009), and the VDI/VDE Guideline 5575 Part 3 (2011).

X-ray capillary optics employ total external reflection by the inner surface of hollow glass tubes to guide and shape X-ray radiation. For incidence angles lower than the critical angle of total reflection the X-ray radiation is guided through the optics at very low losses. The transmission efficiency depends upon the X-ray energy, the capillary materials, reflection surface smoothness, the number of reflections, the capillary inner diameter and the incident beam divergence, and is thus determined by the particular design of the given optics. Generally, the transmission efficiency decreases with increasing X-ray energy owing to the decreasing critical angle of total reflection. The role of X-ray capillary optics as energy filters is insignificant, therefore capillaries are usually used in combination with monochromatization

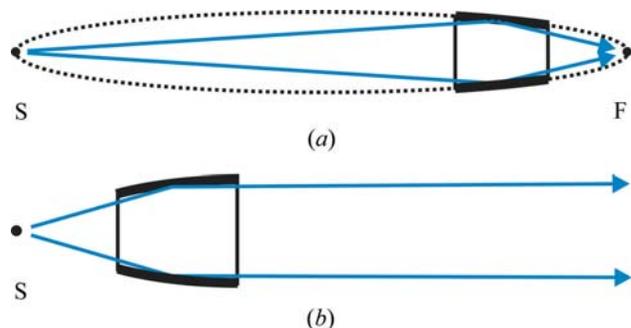


Figure 2.1.21 Schematic of monocapillary optics. (a) Elliptical and (b) parabolic monocapillary. S = source; F = focal point.

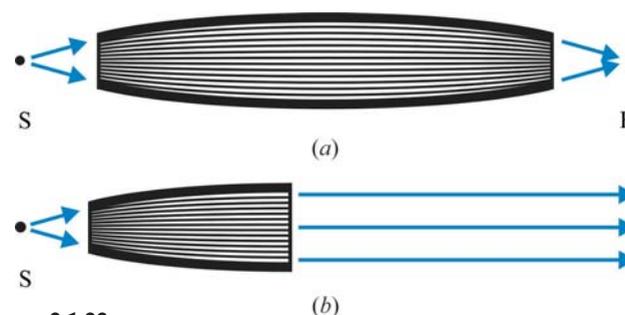


Figure 2.1.22 Schematic of polycapillary optics. (a) Focusing and (b) parallel-beam polycapillary. S = source; F = focal point.

devices such as metal filters, incident-beam graphite monochromators or graded multilayers. Gains in flux density of more than two orders of magnitude compared to pinhole systems have been reported. The most common X-ray capillary optics currently used in laboratory X-ray powder diffractometers can be categorized as either monocabillaries or polycapillaries.

Monocabillaries consist of ellipsoidal or paraboloidal capillaries for focusing or parallelizing X-rays by means of single or multiple total reflections, as illustrated in Fig. 2.1.21. The exit-beam divergence is controlled by the capillary diameter and length as well as the critical angle of total reflection; typical spot sizes range from some 20 mm down to less than 1 μm . Single-reflection monocabillaries are achromatic and almost 100% efficient. Their most important limitations are figure slope errors limiting the spot size. Multi-reflection monocabillaries can have the smallest spot sizes, which do not depend on the source size. An important drawback is that the beam is smallest at the capillary tip. In order to obtain the smallest possible spot size the sample has to be positioned to within 10–100 times the diameter of the tip exit size, e.g. 10–100 μm for a capillary with a 1 μm tip exit size.

Polycapillaries (e.g. Kumakhov & Komarov, 1990) are monolithic systems of micro-structured glass consisting of thousands up to several millions of channels, which are tapered at one or both ends to form desired beam profiles as illustrated in Fig. 2.1.22. A single channel can efficiently turn an 8 keV beam by up to 30° by multiple total reflections. Polycapillaries can collect a very large solid angle up to 20°, resulting in very high intensity gains. Typical spot sizes range from some 20 mm down to about 10 μm and are energy dependent, getting larger at lower energies.

2.1.6.3.4. Combi-optics

The steadily growing trend towards multipurpose instrumentation has led to a multitude of X-ray optics combined in single modules to eliminate reassembling and realignment. Such 'combi-optics' are usually motorized and allow a fully automatic, software-controlled switch between different beam paths to switch between different instrument geometries or to optimize beam conditioning (e.g. high flux *versus* high resolution).

A frequent requirement is the ability to switch between the divergent-beam Bragg–Brentano and parallel-beam Debye–Scherrer geometries, which can be achieved by two combi-optics as illustrated in Fig. 2.1.23. In this example, the incident-beam combi-optics consist of a variable slit and a Göbel mirror. When operating as a variable slit (Fig. 2.1.23a), the parallel-beam path is blocked by the variable slit. Turning the variable slit parallel to the divergent beam (Fig. 2.1.23b) enables the parallel beam and blocks the divergent beam. The diffracted-beam combi-optics consist of a set of two parallel-plate collimators, which are

2.1. LABORATORY X-RAY SCATTERING

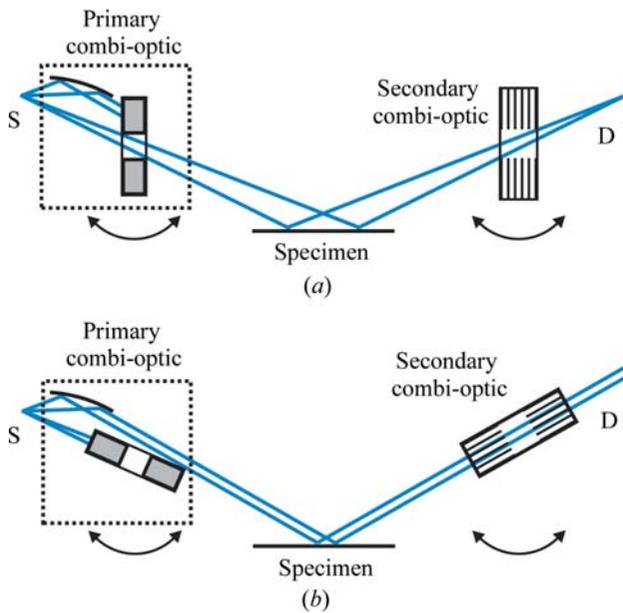


Figure 2.1.23 Incident and diffracted beam combi-optics for switching between (a) the Bragg-Brentano geometry and (b) the parallel-beam geometry. S: X-ray source; D: detector.

separated by a small gap. When turning the two parallel-plate collimators into the beam direction, only those diffracted rays running parallel to the collimator plates will reach the detector (Fig. 2.1.23b). When turning the collimators by approximately 90°, the gap between the two collimators acts as a variable slit enabling a divergent beam (Fig. 2.1.23a).

Significantly more sophisticated combi-optics are used in X-ray diffractometers that are mostly used for thin-film analysis. In Fig. 2.1.24 an example for two different incident-beam and four different diffracted-beam paths is shown, providing the choice between eight different beam paths depending on the properties of the specimen and the application requirements. The incident beam path is characterized by a fixed-target X-ray source equipped with a Göbel mirror, attached on a motorized mount. By rotating this arrangement by about 5°, the beam travels either through a rotary absorber followed by a two-bounce channel-cut monochromator and a slit (upper beam path, high-resolution

setting), or just through a single slit (lower beam path, high-flux setting). The diffracted beam path represents a double-detector setup, typically consisting of a point detector (D1) and a position-sensitive detector (D2). For the point detector three different beam paths can be chosen by means of a switchable slit, which either sends the beam through a three-bounce channel-cut analyser, or through the same two-parallel-plate-collimator arrangement already discussed in Fig. 2.1.23, either acting as a parallel-plate collimator or a variable slit. A fourth beam path without any diffracted-beam X-ray optics allows use of the position-sensitive detector.

2.1.7. X-ray detectors

The general concepts of X-ray detectors are described here with the focus on practical aspects. The physics of X-ray detection and the individual detector technologies are extensively covered in the literature. He (2009) gives a comprehensive discussion that also includes the most recent detector technologies. Additional detailed descriptions are found in *International Tables for Crystallography* Vol. C (2004), as well as in the textbooks by Pecharsky & Zavalij (2009), Clearfield *et al.* (2008), Paganin (2006), Jenkins & Snyder (1996), and Klug & Alexander (1974).

2.1.7.1. Detector parameters

There are many ways to characterize the properties and performance of an X-ray detector.

Ideally, in a given detector operated under appropriate conditions, (1) each photon will produce a detectable signal and (2) the signal recorded is proportional to the number of photons detected. If both conditions are fulfilled then the detector has unit *quantum efficiency*. The *detective quantum efficiency* (DQE) may be defined as the squared ratio of the output signal-to-noise ratio to the input signal-to-noise ratio, expressed as a percentage. A detector's DQE is generally less than 100% because there is always detector noise and not every photon is detected. The DQE thus depends on the characteristics of the detector (*e.g.* transmission of the detector window, count rates and dead time, *etc.*) and varies with the X-ray energy for the same detector.

The *detector linearity* determines the accuracy of intensity measurements and depends on the ratio between the photon

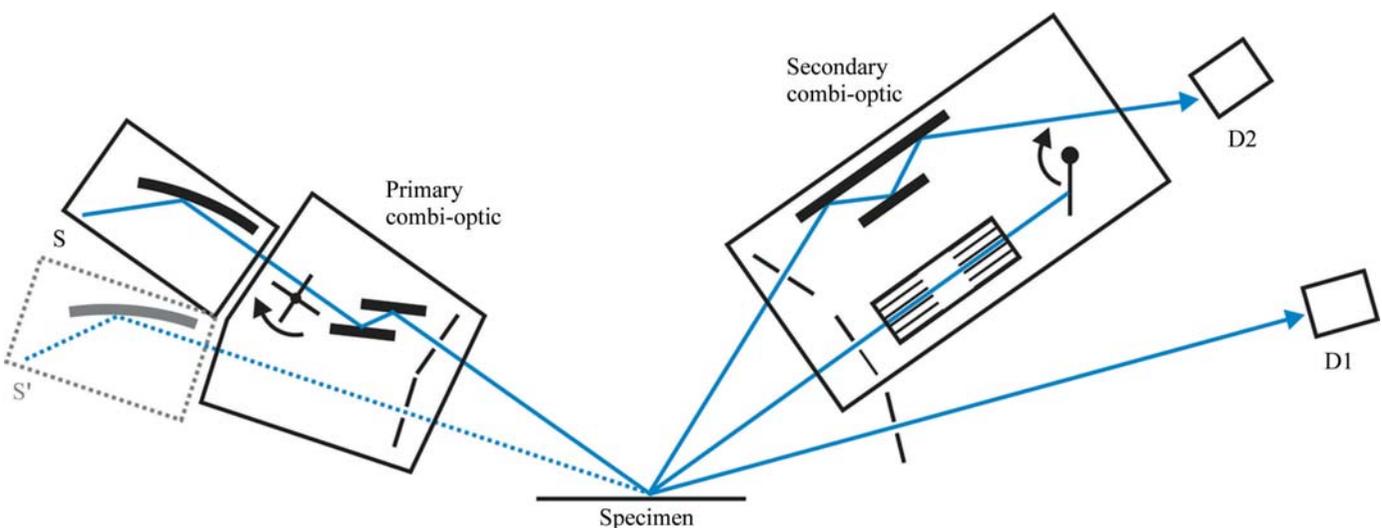


Figure 2.1.24 Example of the use of highly sophisticated incident- and diffracted-beam combi-optics in combination with a rotatable X-ray source and a double detector arm. This setup enables two different incident-beam and four different diffracted-beam paths, and thus provides a choice between eight different beam paths, depending on the properties of the specimen and the requirements of the application. S: X-ray source, S': X-ray source rotated by about 5°, D1, D2: detectors.