International Tables for Crystallography (2018). Vol. H, Section 2.1.6.3.4, pp. 44–45.

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/B_4C, Ni/C, Ru/B_4C, Ti/B_4C, V/B_4C, Cr/B_4C and Mo/B_4C. 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.

2.1.6.3.3.2. 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 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 monocapillaries or polycapillaries.

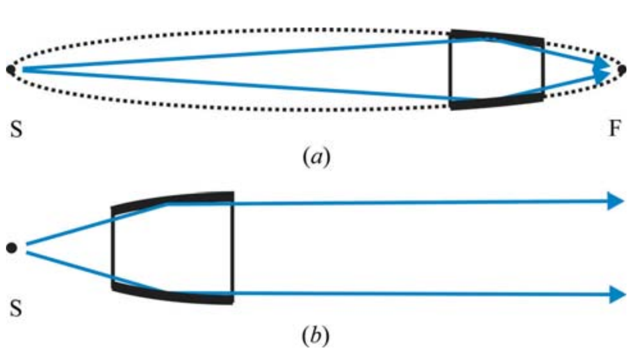

Figure 2.1.21
Schematic of monocapillary optics. (*a*) Elliptical and (*b*) parabolic monocapillary. S = source; F = focal point.

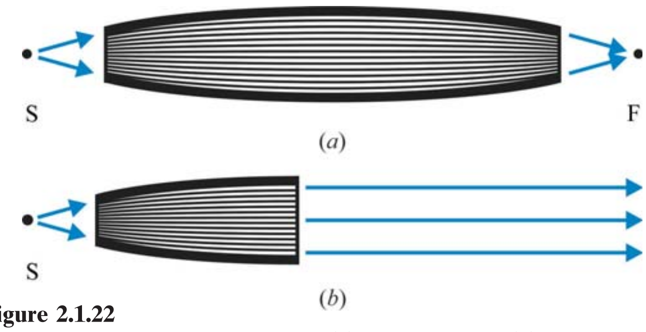

Figure 2.1.22
Schematic of polycapillary optics. (*a*) Focusing and (*b*) parallel-beam polycapillary. S = source; F = focal point.

Monocapillaries 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 monocapillaries are achromatic and almost 100% efficient. Their most important limitations are figure slope errors limiting the spot size. Multi-reflection monocapillaries 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. 2.1.23*a*), the parallel-beam path is blocked by the variable slit. Turning the variable slit parallel to the divergent beam (Fig. 2.1.23*b*) 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

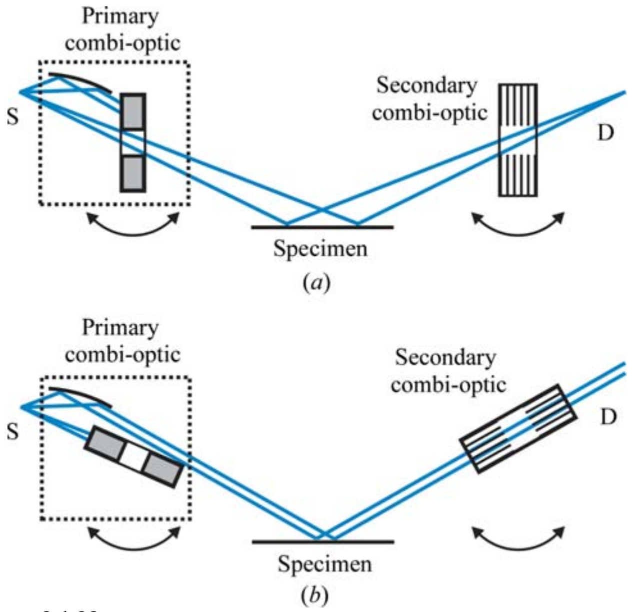

Figure 2.1.23
Incident and diffracted beam combi-optics for switching between (*a*) the Bragg–Brentano geometry and (*b*) the parallel-beam geometry. S: X-ray source; D: detector.

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. 2.1.23*b*). When turning the collimators by approximately 90°, the gap between the two collimators acts as a variable slit enabling a divergent beam (Fig. 2.1.23*a*).

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

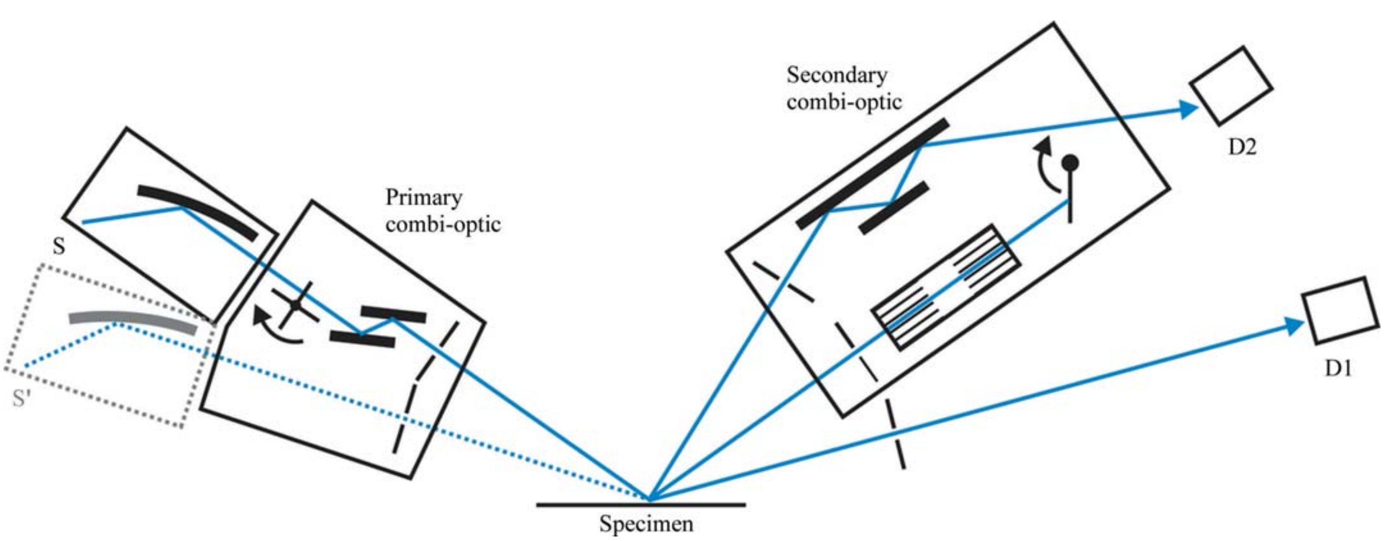

Figure 2.1.24
Example of the use of highly sophisticated incident- and diffracted-beam combi-optics in combination with a rotatable X-ray source and a double detector arm. This setup enables two different incident-beam and four different diffracted-beam paths, and thus provides a choice between eight 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.