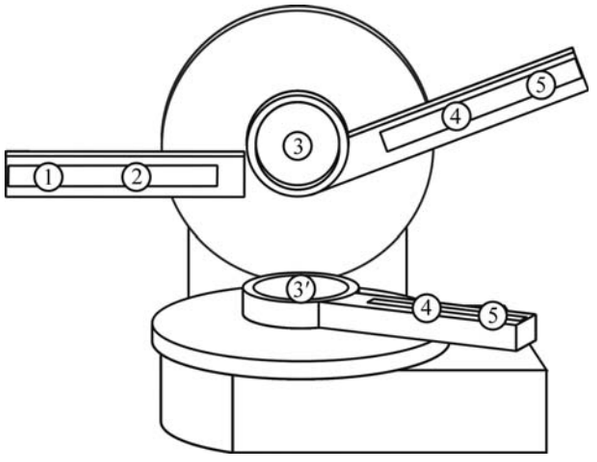

Figure 2.1.12
Sophisticated IP-GID implementation by placing two goniometers vertically with respect to each other, allowing simultaneous coplanar and in-plane measurements using two independent scattered-beam optical X-ray benches (compare with Fig. 2.1.2). The sample stage may be mounted at position 3 or 3′.

(IP-GID) may be measured at angles $2\theta_{\text{IP-GID}}$ in the non-coplanar direction at an exit angle α_F.

There are two principal instrument designs implementing coplanar and in-plane data collection. Firstly, as is obvious from Fig. 2.1.11, a dual-goniometer system may be employed. The most sophisticated implementation has two goniometers placed vertically one above the other, allowing simultaneous coplanar and in-plane measurements using two independent scattered-beam optical X-ray benches as shown in Fig. 2.1.12. Alternatively, the second goniometer may be integrated into the scattered-beam optical X-ray bench, allowing sequential coplanar and in-plane measurements. As a further alternative, a single goniometer may be used, with a Eulerian cradle mounted at the detector position, allowing the detector to be moved around the specimen to perform in-plane measurements. Secondly, a single goniometer equipped with a Eulerian cradle may be used, where the specimen is simply turned by 90° in ψ. As line focus is usually employed for IP-GID measurements, the X-ray source is also turned by 90° to increase the flux.

For all systems, the diffracted-beam optical X-ray benches may be equipped as for multiple beam-path systems, as described in Section 2.1.5.3.1, providing extremely high flexibility. The choice of the most appropriate design depends on issues such as specimen size and weight, the weight of any components in the diffracted-beam path, related spheres of confusion, and the potential need to measure the specimen in a horizontal position.

2.1.6. X-ray sources and optics

This section covers both the generation as well as the conditioning of X-ray beams. All types of X-ray sources, whether laboratory or synchrotron sources, emit a wide range of wavelengths with a characteristic beam divergence and with an intensity related to the power load applied. The function of the incident- and diffracted-beam X-ray optics is to condition the emitted beam in terms of desired wavelength spread, divergence, cross-section size, and shape, and to conserve as much intensity as possible. To achieve maximum performance in terms of intensity and angular resolution, it is essential to design the X-ray optics so that their properties match the characteristics of the X-ray source. Important parameters are the X-ray source beam size and shape, as well as the acceptance angle of the optics given by their design and the distance to the X-ray source.

The optimum choice of an X-ray source and the X-ray optics always depends on the properties of the specimen and the requirements of the applications. Applications requiring high spatial resolution (*e.g.* small single crystals or microdiffraction) or low-angle scattering (*e.g.* thin-film analysis or SAXS) usually require parallel and narrow beams, while diffraction by ideal powders usually works best with larger and slightly divergent beams. As X-ray sources are hardly ever used without X-ray optics, all the components should be seen as one unit determining the beam characteristics at the specimen and eventually at the detector position.

2.1.6.1. *X-ray beam quality measures*

An X-ray beam is characterized by its intensity, wavelength spread, divergence, cross-section size, homogeneity and shape. Simple means for quantifying the quality of an X-ray beam are often useful, and can be used to design an optimal measurement setup by appropriate choice of a combination of X-ray source and X-ray optics. The quantities that are typically used are flux, flux density, brightness and brilliance, all within a 0.1% bandwidth represented by a wavelength range, $\Delta\lambda$, centred around a specific wavelength λ, *i.e.* $\Delta\lambda$ is equal to 1/1000 of λ. While flux, flux density, brightness and brilliance are inter-related, they are distinct and one thus has to consider all of these when comparing X-ray beam characteristics.

Flux represents the integrated intensity of an X-ray beam and is defined as the number of X-ray photons emitted per unit time. The unit for flux is photons per second (p.p.s.).

Flux density is defined as the flux passing through a unit area. The unit is p.p.s. mm^{-2}. Flux density is an appropriate parameter for measuring local counting rates and is synonymous to the term 'intensity' as used in colloquial speech.

Brightness takes the beam divergence into account, and is defined as the flux per unit of solid angle of the radiation cone. The unit is p.p.s. mrad^{-2}. Brightness is an appropriate parameter to use when comparing two X-ray sources with identical focal spot size, as the definition does not contain a unit area.

Brilliance additionally takes the beam dimensions into account and is defined as brightness per mm^2. The unit is p.p.s. mm^{-2} mrad^{-2}. Brilliance is maximized by making the beam size and divergence as small as possible, and the photon flux as large as possible. Two X-ray beams may have the same flux density but different brilliance if the two beams have different beam divergence. Brilliance is thus an appropriate parameter to use when comparing two X-ray sources with different focal spot sizes.

Note that the X-ray source brilliance is an invariant quantity, *i.e.* the brilliance at the specimen position cannot be improved by any optical techniques, but only by increasing the brilliance of the X-ray source. This is a consequence of Liouville's theorem, which states that phase space is conserved. Accordingly, focusing the beam to a smaller size by means of any diffractive or reflective optics will necessarily increase the flux density and the divergence of the X-ray beam, and *vice versa*. Additionally, any diffractive or reflective optics lose flux owing to their reflectivity, which usually is $\leq$90%. Apertures such as slits can help to reduce beam size and divergence, but only at the expense of flux.

Brilliance is more important than flux for experiments with small specimens (*e.g.* single crystals) or small regions of interest (*e.g.* microdiffraction), where it is generally desirable to work

Table 2.1.3
Characteristic wavelengths and absorption edges of metal filters in common use

These data are taken from *International Tables for Crystallography* Vol. C (2004). Metal filters are discussed in Section 2.1.6.3.1.2.

Anode material	$K\alpha_2$	$K\alpha_1$	$K\beta_3$	$K\beta_1$	Metal filter	K absorption edge (Å)
Cr	2.2936510 (30)	2.2897260 (30)	2.0848810 (40)	2.0848810 (40)	V	2.269211 (21)
Co	1.7928350 (10)	1.7889960 (10)	1.6208260 (30)	1.6208260 (30)	Fe	1.7436170 (49)
Cu	1.54442740 (50)	1.54059290 (50)	1.3922340 (60)	1.3922340 (60)	Ni	1.4881401 (36)
Ga†	1.3440260 (40)	1.3401270 (96)	1.208390 (75)	1.207930 (34)		
Mo	0.713607 (12)	0.70931715 (41)	0.632887 (13)	0.632303 (13)	Zr Nb	0.6889591 (31) 0.6531341 (14)
Ag	0.5638131 (26)	0.55942178 (76)	0.4976977 (60)	0.4970817 (60)	Rh Pd	0.5339086 (69) 0.5091212 (42)

† Currently used with dedicated Montel optics only.

with a beam of low divergence and to match the incident beam size to the size of the specimen or the region of interest.

The illumination of larger specimen areas is particularly important for any applications involving polycrystalline specimens, where focusing of the diffracted beam has an advantage over parallel-beam optics in terms of higher beam flux and divergence in that the angular resolution in the diffraction pattern increases. Using an X-ray beam with too small a cross section and/or divergence will result in a smaller or even too small number of diffracting crystallites. This will generally lead to a loss in the diffracted intensity, and may additionally lead to an inhomogeneous intensity distribution in space, leading to random and uncorrectable intensity errors (known as 'particle statistics error', 'spottiness error' or 'granularity error'), and needs to be avoided by all means.

The combination of an appropriate X-ray source with appropriate X-ray optics thus depends on the properties of the specimen and the requirements of the application, and contributes most to the attainable data quality. This is in full agreement with the statement made earlier that there are only a few instrument configurations that will be ideal for any two application areas, or every conceivable sample within a single application area. While changes of most X-ray optics are extremely easy these days, changing between different types of X-ray sources may require significant effort. The choice of the most appropriate X-ray source therefore requires, at the time of instrument acquisition, careful consideration of the types of specimen in relation to the analyses to be conducted.

2.1.6.2. *X-ray sources*

In this section the general concepts of the commonest types of X-ray sources will be described. The physics of X-ray generation and the properties of X-rays have been extensively covered in the literature. More detailed information can be found in, for example, *International Tables for Crystallography* Vol. C (2004) as well as in the textbooks by Pecharsky & Zavalij (2009), Clearfield *et al.* (2008), Jenkins & Snyder (1996), and Klug & Alexander (1974).

2.1.6.2.1. *Generation of X-rays and the X-ray spectrum*

In laboratory X-ray sources, X-rays are produced by a multi-keV electron beam impinging on a metallic target. The X-ray spectrum that is obtained is characterized by a broad band of continuous radiation, accompanied by a number of discrete spectral lines characteristic of the target material. The continuous part of the spectrum ('*Bremsstrahlung*') is generated by the rapid deceleration of the electrons within the target, ranging from lowest energies as a result of gradual deceleration through to a cutoff wavelength whose energy corresponds to the initial kinetic energy of the electron, as a result of instantaneous deceleration. The discrete spectral lines ('characteristic radiation') are the result of electrons knocking out core electrons from the target material. This results in emission of 'fluorescent' X-rays when the perturbed atom relaxes to its ground state by filling up the energy levels of the electrons that have been knocked-out by means of electron transitions from higher electron shells. The energy of the fluorescent radiation is characteristic of the atomic energy levels of the target material. The most commonly used characteristic radiation is that of $K\alpha$, representing the transition of a $2p$ electron (L shell) filling a hole in a $1s$ (K) shell.

The target materials that are commonly in use strongly depend on the application and the type of X-ray source used. The most commonly used target materials range from Cr through to Co, Cu, Mo and Ag. With the recent introduction of liquid-metal targets, see Section 2.1.6.2.2.2(*b*), Ga will find increasing use in applications requiring the smallest spot sizes and highest brilliance. A list of characteristic wavelengths and absorption edges of commonly used metal ($K\beta$) filters is given in Table 2.1.3.

Today's laboratory X-ray sources can be classified as shown in Table 2.1.1, and are described in Section 2.1.6.2.2. For performance considerations see Section 2.1.6.2.3.

2.1.6.2.2. *Types of X-ray sources*

The performance of X-ray sources is usually characterized *via* brilliance as a measure for the quality of the emitted X-rays. The brilliance of an X-ray source is determined by several factors such as electron power density and the take-off angle.

The electron power density is the most important factor. Only a small fraction of <1% of the applied electron energy is converted into X-rays, so most of the incident energy is dissipated within the target as heat. The maximum power density and thus brightness of the X-ray source is limited by the melting or evaporation temperature of solid or liquid metal targets, respectively, and the efficiency with which the heat is removed from the area on which the electrons impact.

The take-off angle describes the angle under which the focal spot is viewed, and typically ranges from 3° to 7°, but may be up to 45°, depending on the type of X-ray source. The actual take-off angle that is chosen represents a compromise. On the one hand, it should be as small as possible to minimize the effectively seen