*International Tables for Crystallography* (2018). Vol. H, Section 2.1.6.2.2, pp. 37–39.



**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<br>Nb | 0.6889591 (31)<br>0.6531341 (14) |
| Ag | 0.5638131 (26) | 0.55942178 (76) | 0.4976977 (60) | 0.4970817 (60) | Rh<br>Pd | 0.5339086 (69)<br>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

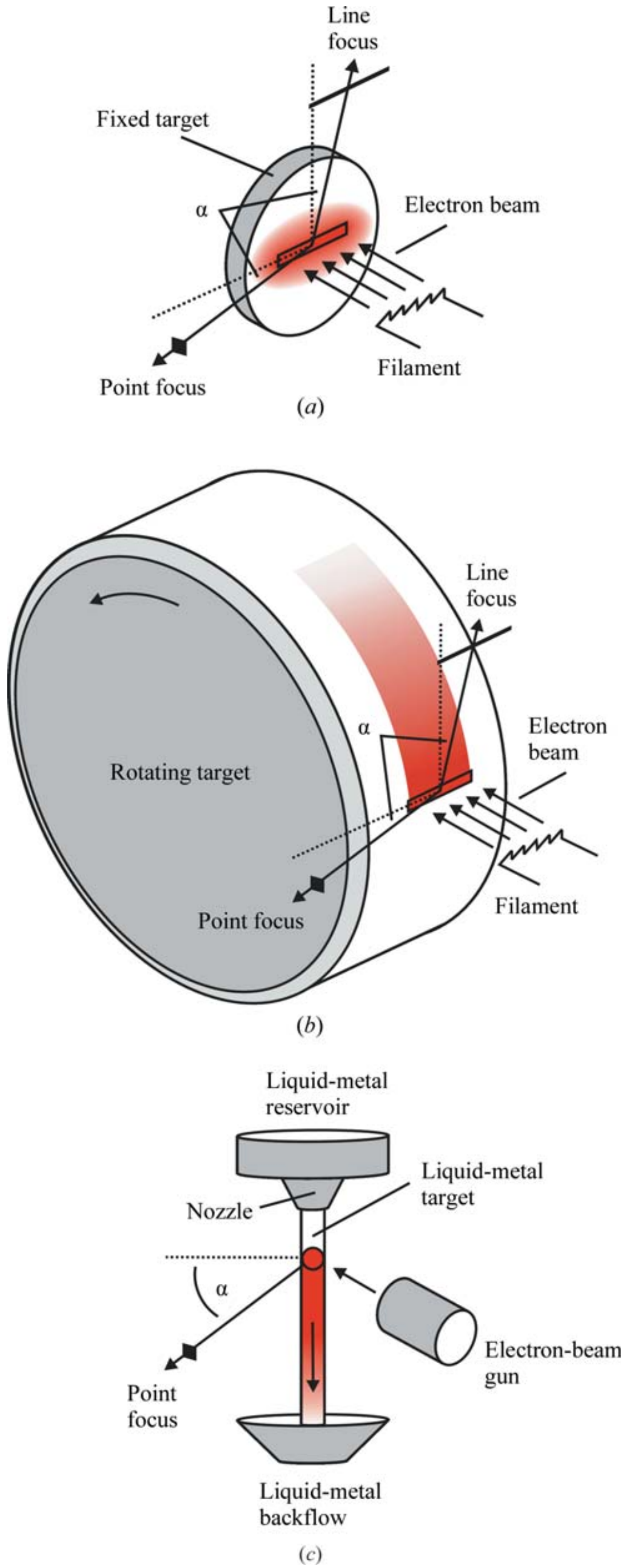


**Figure 2.1.13**
Illustration of the working principle of laboratory X-ray sources: (*a*) fixed target, (*b*) rotating target, (*c*) liquid-metal jet. $\alpha$: take-off angle. For fixed targets (*a*) the heat mainly flows towards the cooled back end of the target. For moving targets (*b*, *c*) cold parts of the target are moved into the electron beam continously, providing an extremely large effective cooling efficiency.

width of the focal spot to increase resolution. On the other hand, it cannot be made arbitrarily small to avoid self-absorption by the metal target due to the finite depth in which the X-ray radiation is produced. The higher the tube voltage the larger the take-off angle should be to avoid intensity losses by self-absorption.

In the history of laboratory X-ray source development, most effort has probably been concentrated on techniques for removing the heat from the metal target as efficiently as possible, as illustrated in Fig. 2.1.13, leading to two different categories of X-ray sources for laboratory use: fixed- and moving-target X-ray sources.

#### *2.1.6.2.2.1. Fixed-target X-ray sources*

Fixed-target X-ray sources are used in more than 90% of all X-ray diffractometer installations (Fig. 2.1.13*a*). Electrons are generated by heating a filament (cathode) and accelerated towards the metal target (anode) by means of a high potential, typically of the order of 30–60 kV.

In conventional X-ray sources the electrons are focused by an electrostatic lens onto the anode to form the focal spot. Typical power ratings range from several hundred watts up to about 3 kW. The anode is water-cooled from the back. Focal spots are of rectangular shape, and can be viewed at the two long and the two short faces, giving two line and two point foci, respectively. This allows up to four instruments to be operated with a single X-ray source. However, the vast majority of all today's X-ray diffractometers are equipped with an individual X-ray source (and sometimes two, see Section 2.1.5.3.1). This significantly eases alignment as there is no need to align the instrument with respect to the X-ray source, and allows instrument configurations with moving X-ray sources. Modern X-ray-source stage designs allow switching between point and line focus by rotating the X-ray source 90° without alignment and even without the need to disconnect the powder cables and water supply.

Conventional X-ray sources have long and wide electron beams so that a large area of the target is heated (Fig. 2.1.13*a*). The heat generated in the middle of this area can mainly flow in just one direction: towards the water-cooled back of the anode. Heat flow parallel to the surface is minimal, thus limiting the cooling efficiency. It is for this reason that conventional X-ray sources achieve the lowest brilliance of any laboratory X-ray source. Conventional X-ray sources are usually coupled with relatively simple optics and are cheap compared to moving-target systems. In addition they are maintenance-free, apart from periodic changes of the X-ray source owing to ageing.

'Micro-focus' X-ray sources represent another category of X-ray source and are characterized by very small focal spot sizes ranging from a few µm up to about 50 µm. In this type of X-ray source, the improved focusing of the electron beam is achieved by very fine electrostatic or magnetic lenses. Power requirements are significantly less than conventional X-ray sources, ranging from a few watts up to some hundred watts, depending on focal spot size; water cooling is frequently not required. Again, there is no maintenance required beyond periodic tube changes.

As the focal spot area is very small, heat can also flow sideways, improving the thermal cooling efficiency and thus allowing this type of X-ray-source tube to achieve significantly higher brilliance than conventional X-ray sources. To benefit from this increased performance, relatively large optics of the reflective type (see Section 2.1.6.3.3) are required, making micro-focus X-ray source systems significantly more expensive than conventional systems.

The lifetime of a fixed-target X-ray source depends on many factors, of which operation of the source within specifications (such as specific loading and cooling) is particularly important. The 'useful' lifetime may be significantly shorter, even though the X-ray source still operates. Deposition of tungsten from the 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. 2.1.13*b*). 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. 2.1.13*c*), 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-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.

**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<br>0.3 × 3<br>0.2 × 2<br>0.1 × 1 | 18<br>5.4<br>3<br>1.2 | 3.6<br>6<br>7.5<br>12 |
| Micro-focus rotating anode (Cu) | 0.1 | 2.7 | 27 |
| Liquid-metal jet (Ga) | 0.02 × 0.02 | 0.2 | >500 |

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