

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