

2. INSTRUMENTATION AND SAMPLE PREPARATION

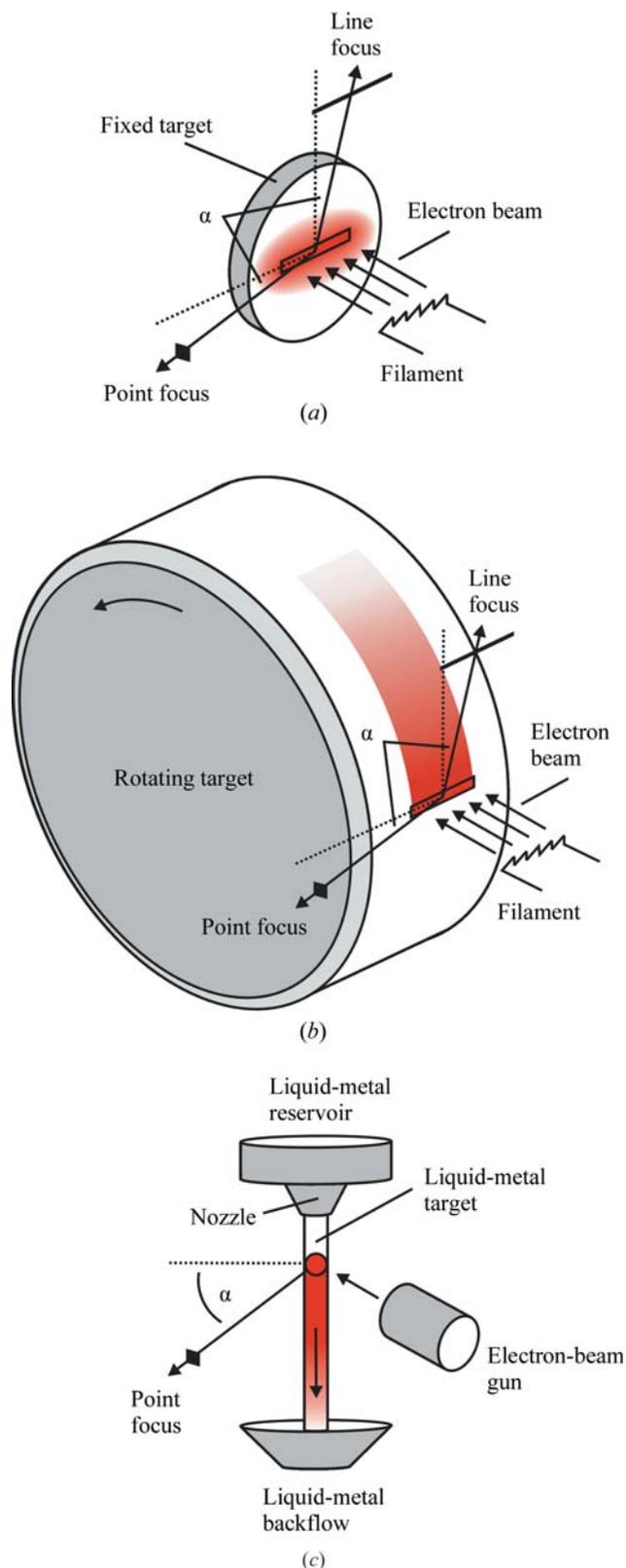
**Figure 2.1.13**

Illustration of the working principle of laboratory X-ray sources: (a) fixed target, (b) rotating target, (c) liquid-metal jet. α : 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 continuously, 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. 2.1.13a). 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. 2.1.13a). 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