PART 6. RADIATION SOURCES AND OPTICS

Chapter 6.1. X-ray sources

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6.1.1. Overview

In this chapter we shall discuss the production of the most suitable X-ray beams for data collection from single crystals of macromolecules. This subject covers the generation of X-rays and the conditioning or selection of the X-ray beam that falls on the sample with regard to intensity, cross section, degree of parallelism and spectral composition. The conclusions drawn do not necessarily apply to smaller-unit-cell crystals or to noncrystalline samples.

6.1.2. Generation of X-rays

X-rays are generated by the interaction of charged particles with an electromagnetic field. There are four sources of interest to the crystallographer.

(1) The bombardment of a target by electrons in a vacuum tube produces a continuous ('white') X-ray spectrum, called *Bremsstrahlung*, which is accompanied by a number of discrete spectral lines characteristic of the target material. The most common target material is copper, and the most frequently employed X-ray line is the copper $K\alpha$ doublet with a mean wavelength of 1.542 Å. X-ray tubes are described in some detail in Chapter 4.2 of *IT* C (2004). We shall consider only the most important points in X-ray tube design here.

(2) Synchrotron radiation is produced by relativistic electrons in orbital motion. This is the subject of Part 8.

(3) The decay of natural or artificial radioisotopes is often accompanied by the emission of X-rays. Radioactive sources are often used for the testing and calibration of X-ray detectors. For our purposes, the most important source is made from ^{55}Fe, which has a half-life of 2.6 years and produces Mn $K\alpha$ X-rays with an energy of 5.90 keV.

(4) Ultra-short pulses of X-rays are generated in plasmas produced by the bombardment of targets by high-intensity sub-picosecond laser pulses (*e.g.* Forsyth & Frankel, 1984). In earlier work, the maximum pulse repetition frequency was much less than 1 Hz, but picosecond pulses at more than 1 Hz are now being achieved with μm-size sources. The time-averaged X-ray intensities from these sources are very low, so their application will probably remain limited to time-resolved studies (Kleffer *et al.*, 1993).

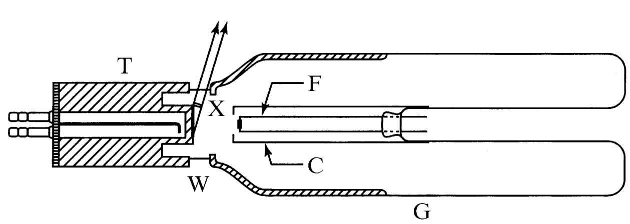

Figure 6.1.2.1
Section through a sealed X-ray tube. G, glass envelope; F, filament leads (at negative high voltage); C, focusing cup; T, target (at ground potential); W, one of four beryllium windows. The electron beam forms a line on the target, which is viewed at a small take-off angle to form a foreshortened effective source X.

X-rays also arise in the form of channelling radiation resulting from the bombardment of crystals, such as diamonds, by electrons with energies of several MeV from a linear accelerator (Genz *et al.*, 1990) and in the form of transition radiation when multiple-foil targets are bombarded by electrons in the range 100–500 MeV (*e.g.* Piestrup *et al.*, 1991). It will be some time before these new sources can compete with the older methods for routine data collection.

6.1.2.1. Stationary-target X-ray tubes

A section through a permanently evacuated, sealed X-ray tube is shown in Fig. 6.1.2.1 The tube has a spirally wound tungsten filament, F, placed immediately behind a slot in the focusing cup, C, and a water-cooled target or anode, T, approximately 10 mm from the surface of C. The filament–focusing-cup assembly is at a negative voltage of between 30 and 50 kV, and the target is at ground potential. The electron beam strikes the target in a focal line, which acts as a line source of X-rays. There are usually two pairs of X-ray windows, W, through which the source is viewed at a small angle to the target surface, thus producing a foreshortened effective source, X, which is approximately square in one plane and a narrow line in the other. Focus dimensions on the target and maximum recommended power loading are shown for a number of standard inserts in Table 6.1.2.1. None of these are ideal for macromolecular crystallography. The assembly of a cathode, anode and windows – the tube insert – is inserted in a shock- and radiation-proof shield which is fixed to the table. Attached to the shield are X-ray shutters and filters, and sometimes brackets for bolting on X-ray cameras. A high-voltage connection is made to the tube by means of a flexible, shielded, shock-proof cable; nowadays, this high voltage is almost invariably full-wave rectified and smoothed DC.

6.1.2.2. Rotating-anode X-ray tubes

The sealed tubes described above are convenient and require little maintenance, but their power dissipation, and thus their X-ray output, is limited. For macromolecular crystallography, the most commonly used tubes are continuously pumped, demountable tubes with water-cooled rotating targets [see the

Table 6.1.2.1
Standard X-ray tube inserts

Focus size on target (mm × mm)	Recommended power loading (kW)
8 × 0.15	0.8
8 × 0.4	1.5
10 × 1.0	2.0
12 × 2.0	2.7

references