

## 6.1. X-RAY SOURCES

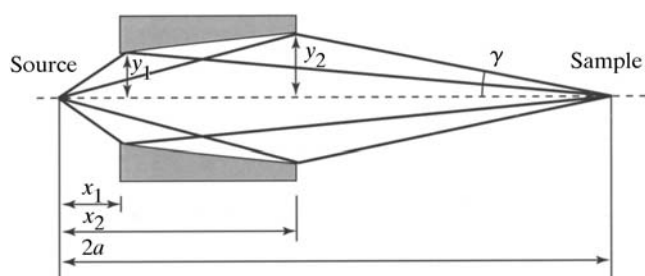


Fig. 6.1.4.6. Ellipsoidal mirror for use with a microfocus X-ray tube, where  $x_1$  is  $\sim 15$  mm. The major axis,  $2a$ , may be up to 600 mm, whereas the exit aperture,  $2y_2$ , lies in the region 0.8–1.4 mm. The angle  $\gamma$  determines the cross fire on the sample and is less than 1 rad.

For X-rays of wavelength  $\lambda$ , measured in Å,

$$\theta_c \simeq 2.32 \times 10^{-3} (Z\rho/A)^{1/2} \lambda,$$

where  $Z$  is the atomic number,  $A$  is the atomic mass and  $\rho$  is the specific gravity of the reflecting surface.

Thus, for Cu  $K\alpha$  radiation and a gold surface,  $\theta_c \simeq 10$  mrad. The reflectivity of the mirror surface is strongly dependent on the surface roughness; for the reflectivity to be more than 50%, the r.m.s. roughness must not exceed 10 Å.

It is not possible to design a reflecting collimator with a planar angle of collection greater than about  $3\theta_c$ . For the shorter wavelengths, in particular, variable-spacing multilayer mirrors (Schuster & Göbel, 1997) hold considerable promise. If the spacing at the upstream end of the mirror is 30 Å, the largest angles of incidence will be 26 and 17 mrad for 1.54 and 1.0 Å X-rays, respectively. By comparison, the critical angles at a gold surface for these radiations are 10 and 6.5 mrad, respectively.

## 6.1.4.2. Focusing collimators for microfocus sources

In most arrangements that include conventional X-ray tubes, the planar angle of collection is very small. A more efficient use is always made of the radiation from the target by a focusing collimator, which forms an image of the source on the sample (Fig. 6.1.4.6). The angle of collection should be as large as possible, while the cross fire, *i.e.* the angle of convergence, is kept small, say, at about  $10^{-3}$  rad. It is possible to design focusing collimators based on gold-surfaced toroids of revolution (Elliott, 1965), which afford a planar angle of collection of about three times the critical angle for total external reflection, that is, about  $30 \times 10^{-3}$  rad.

Consequently, the mirror should *magnify* about 30 times, and if the image diameter, determined by a typical sample size, is to be 300  $\mu\text{m}$ , the size of the focus should be about 10  $\mu\text{m}$ . The solid angle of collection of such an imaging toroid is about  $8 \times 10^{-4}$  steradians, that is, more than 1000 times greater than the solid angle of a simple non-imaging collimator. The averaged mirror reflectivity achieved at present is about 0.3, so the microfocus tube and toroidal mirror combination produces a similar intensity at the sample as the conventional tube with a non-focusing collimator at about 300 times the power. Future increases of the reflectivity are likely as the surface roughness of the mirrors is improved.

A suitable microfocus tube has been described by Arndt, Long & Duncumb (1998); mirrors used with this tube were discussed by Arndt, Duncumb *et al.* (1998). The tube design allows the distance between the source and the mirror to be as little as 10 mm in order to achieve the necessary magnification without making the distance between the tube and the sample inconveniently long.

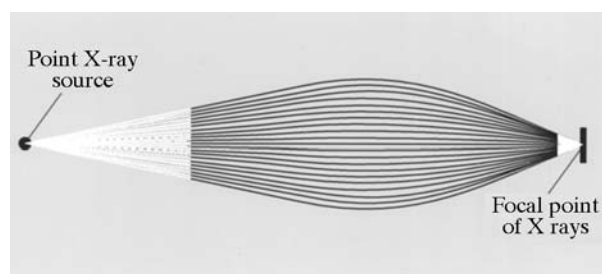


Fig. 6.1.4.7. A polycapillary collimator (after Bly & Gibson, 1996).

## 6.1.4.3. Other focusing collimators

There has been very active development in recent years of tapering capillaries for focusing X-rays, either as individual capillaries (see the review by Bilderback *et al.*, 1994), or in the form of multicapillary bundles. The latter were first described by Kumakhov & Komarov (1990); since then, they have undergone great improvements in the form of fused bundles (Bly & Gibson, 1996) (Fig. 6.1.4.7). Single capillaries have found the greatest use as X-ray concentrators, where a larger-diameter beam of X-rays enters the large end of a tapered capillary and is concentrated to a diameter of a few  $\mu\text{m}$ . Fused polycapillary bundles have been employed as focusing collimators for protein crystallography (MacDonald *et al.*, 1999). Both types of capillary optics are usually designed as multi-bounce devices, in which the X-rays undergo several, or many, reflections at the walls of the capillary; consequently the cross-fire half-angle at the output end has a value about equal to the critical angle for reflection at a glass surface or, perhaps, 4 mrad. This is sometimes too great for producing diffraction patterns with an optimum signal-to-background ratio.

Other methods of focusing X-rays, such as zone plates (Kirz, 1974) and refractive optics, are being investigated, but at present none of them can compare with toroidal reflectors for data collection from single crystals of macromolecules.

## 6.1.4.4. Crystal monochromators

When the X-rays from the tube target are specularly reflected by a mirror, the spectrum is cut off for X-rays below the shortest wavelength for which the critical angle is equal to the smallest angle of incidence on the mirror. For a typical mirror designed for Cu  $K\alpha$  radiation, this cutoff wavelength might be about 0.75 Å, and the harder X-rays can be further attenuated by a  $\beta$ -filter. Of course, the more nearly monochromatic the radiation falling on the sample, the lower the radiation damage and the higher the spot-to-background ratio in the recorded patterns.

White radiation is almost completely eliminated by reflecting the primary X-ray beam using a natural or artificial (multilayer) crystal. The most commonly used type of plane monochromator for macromolecular crystallography is a single crystal of graphite. This material (HOPG, or highly ordered pyrolytic graphite) has a relatively large mosaic spread, typically about  $0.4^\circ$ , and it cannot separate the  $K\alpha$  doublet. This separation is essential in most small-molecule investigations, but is unnecessary for macromolecular crystals, which rarely diffract beyond 1.5 Å, and disadvantageous where a high intensity of the beam reflected by the monochromator is the main consideration.

The intensity of the diffraction pattern obtained with a graphite monochromator is only about two or three times lower than that resulting from a  $\beta$ -filtered pinhole-collimated beam. The situation is different at synchrotron beam lines, which must incorporate a monochromator in order to select the desired X-ray energy band. Curved focusing crystals collect X-rays over a relatively large horizontal angular range and thus produce a beam with a horizontal

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convergence angle of up to several milliradians. Much more nearly parallel beams are produced by reflection at several crystals in tandem, often in the form of monolithic channel-cut monochromators. In present-day storage rings, the power density at the first optical element is of the order of  $10 \text{ W mm}^{-2}$  at wiggler and undulator beam lines. This amount of power can be dissipated by careful design of water-cooling channels (Quintana & Hart, 1995; van Silfhout, 1998). In addition, the monochromator crystal, usually

of silicon or germanium, may be profiled to minimize distortions as a result of thermal stresses.

The next generation of insertion devices will subject the optical elements to loads of several hundred  $\text{W mm}^{-2}$ . Possible engineering solutions to the very severe heat-loading problem include the use of diamond crystals as reflecting elements. This material has a very high thermal conductivity, especially at low temperatures.