

## 6. RADIATION SOURCES AND OPTICS

## References

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 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.

- Arndt, U. W., Duncumb, P., Long, J. V. P., Pina, L. & Inneman, A. (1998). *Focusing mirrors for use with microfocus X-ray tubes*. *J. Appl. Cryst.* **31**, 733.
- Arndt, U. W., Long, J. V. P. & Duncumb, P. (1998). *A microfocus X-ray tube used with focusing collimators*. *J. Appl. Cryst.* **31**, 936–944.
- Arndt, U. W. & Stubbings, S. J. (1988). *Miniature ionisation chambers*. *J. Appl. Cryst.* **21**, 577.
- Bailey, R. L. (1978). *The design and operation of magnetic liquid shaft seals*. In *Thermomechanics of Magnetic Fluids*, edited by B. Berkovsky. London: Hemisphere.
- Beuville, E., Beche, J.-F., Cork, C., Douence, V., Earnest, J., Millaud, D., Nygren, H., Padmore, B., Turko, G., Zizka, G., Datte, P. & Xuong Ng, H. (1997). *Two-dimensional pixel array sensor for protein crystallography*. *Proc. SPIE*, **2859**, 85–92.
- Bilderback, D. H., Thiel, D. J., Pahl, R. & Brister, K. E. (1994). *X-ray applications with glass-capillary optics*. *J. Synchrotron Rad.* **1**, 37–42.
- Bly, P. & Gibson, D. (1996). *Polycapillary optics focus and collimate X-rays*. *Laser Focus World*, March issue.
- Buras, B. & Tazzari, S. (1984). Editors. *European Synchrotron Radiation Facility*. Geneva: ESRP.
- Elliott, A. (1965). *The use of toroidal reflecting surfaces in X-ray diffraction cameras*. *J. Sci. Instrum.* **42**, 312–316.
- Forsyth, J. M. & Frankel, R. D. (1984). *Experimental facility for nanosecond time-resolved low-angle X-ray diffraction experiments using a laser-produced plasma source*. *Rev. Sci. Instrum.* **55**, 1235–1242.
- Fourme, R., Ducruix, A., Ries-Kautt, M. & Capelle, B. (1995). *The perfection of protein crystals probed by direct recording of Bragg reflection profiles with a quasi-planar X-ray wave*. *J. Synchrotron Rad.* **2**, 136–142.
- Franks, A. (1995). *An optically focusing X-ray diffraction camera*. *Proc. Phys. Soc. London Sect. B*, **68**, 1054–1069.
- Genz, H., Graf, H.-D., Hoffmann, P., Lotz, W., Nething, U., Richter, A., Kohl, H., Weickenmeyer, A., Knüpfer, W. & Sellschop, J. P. F. (1990). *High intensity electron channeling and perspectives for a bright tunable X-ray source*. *Appl. Phys. Lett.* **57**, 2956–2958.
- Green, M. (1963). *The target absorption correction in X-ray microanalysis*. In *X-ray Optics and X-ray Microanalysis*, edited by H. H. Pattee, V. E. Cosslett & A. Engström, pp. 361–377. New York and London: Academic Press.
- Green, M. & Cosslett, V. E. (1968). *Measurements of K, L and M shell X-ray production efficiencies*. *Br. J. Appl. Phys. Ser. 2*, **1**, 425–436.
- Hofmann, A. (1978). *Quasi-monochromatic synchrotron radiation from undulators*. *Nucl. Instrum. Methods*, **152**, 17–21.
- International Tables for Crystallography* (2004). Vol. C. *Mathematical, Physical and Chemical Tables*, edited by E. Prince. Dordrecht: Kluwer Academic Publishers.
- Ishimura, T., Shiraiwa, Y. & Sawada, M. (1957). *The input power limit of the cylindrical rotating anode of an X-ray tube*. *J. Phys. Soc. Jpn.* **12**, 1064–1070.
- Kirkpatrick, P. & Baez, A. V. (1948). *J. Opt. Soc. Am.* **56**, 1–13.
- Kirz, J. (1974). *Phase zone plates for X-rays and the extreme UV*. *J. Opt. Soc. Am.* **64**, 301–309.
- Kleffer, J. C., Chaker, M., Matte, J. P., Pépin, H., Côté, C. Y., Beaudouin, Y., Johnston, T. W., Chien, C. Y., Coe, S., Mourou, G. & Peyrusse, O. (1993). *Ultra-fast X-ray sources*. *Phys. Fluids*, **B5**, 2676–2681.
- Kohra, K., Ando, M., Natsushita, T. & Hashizume, H. (1978). *Nucl. Instrum. Methods*, **152**, 161–166.
- Kumakhov, M. A. & Komarov, F. K. (1990). *Phys. Rep.* **191**, 289–350.
- Lemonnier, M., Fourme, R., Rousseaux, F. & Kahn, R. (1978). *X-ray curved-crystal monochromator system at the storage ring DCI*. *Nucl. Instrum. Methods*, **152**, 173–177.
- MacDonald, C. A., Owens, S. M. & Gibson, W. M. (1999). *Polycapillary X-ray optics for microdiffraction*. *J. Appl. Cryst.* **32**, 160–167.
- Milch, J. R. (1983). *A focusing X-ray camera for recording low-angle diffraction from small specimens*. *J. Appl. Cryst.* **16**, 198–203.
- Montel, M. (1957). *X-ray microscopy with catamorphic roof mirrors*. In *X-ray Microscopy and Microradiography*, edited by V. E. Cosslett, A. Engstrom & H. H. Pattee Jr, pp. 177–185. New York: Academic Press.
- Müller, A. (1929). *A spinning target X-ray generator and its input limit*. *Proc. R. Soc. London Ser. A*, **125**, 507–516.
- Müller, A. (1931). *Further estimates of the input limits of X-ray generators*. *Proc. R. Soc. London Ser. A*, **132**, 646–649.

## 6.1. X-RAY SOURCES

- Nagel, D. J. (1980). *Comparison of X-ray sources*. *Ann. N. Y. Acad. Sci.* **342**, 235–247.
- Nave, C., Clark, G., Gonzalez, A., McSweeney, S., Hart, M. & Cummings, S. (1995). *Tests of an asymmetric monochromator to provide increased flux on a synchrotron radiation beam line*. *J. Synchrotron Rad.* **2**, 292–295.
- Oosterkamp, W. J. (1948a). *The heat dissipation in the anode of an X-ray tube*. *Philips Res. Rep.* **3**, 49–59.
- Oosterkamp, W. J. (1948b). *The heat dissipation in the anode of an X-ray tube*. *Philips Res. Rep.* **3**, 161–173.
- Oosterkamp, W. J. (1948c). *The heat dissipation in the anode of an X-ray tube*. *Philips Res. Rep.* **3**, 303–317.
- Osmic Inc. (1998). Sales literature. Osmic Inc., Troy, Michigan, USA.
- Padmore, H. A., Ackermann, G., Celestre, R., Chang, C. H., Franck, K., Howells, M., Hussain, Z., Irick, S., Locklin, S., MacDowell, A. A., Patel, J. R., Rah, S. Y., Renner, T. R. & Sandler, R. (1997). *Submicron white-beam focusing using elliptically bent mirrors*. *Synchrotron Radiat. News*, **10**, 18–26.
- Phillips, W. C. (1985). *X-ray sources*. *Methods Enzymol.* **114**, 300–316.
- Piestrup, M. A., Boyers, D. G., Pincus, C. I., Harris, J. L., Maruyama, X. K., Bergstrom, J. C., Caplan, H. S., Silzer, R. M. & Skopik, D. M. (1991). *Quasimonochromatic X-ray source using photoabsorption-edge transition radiation*. *Phys. Rev. A*, **43**, 3653–3661.
- Quintana, J. P. & Hart, M. (1995). *Adaptive silicon monochromators for high-power wigglers; design, finite-element analysis and laboratory tests*. *J. Synchrotron Rad.* **2**, 119–123.
- Schuster, M. & Göbel, H. (1997). *Application of graded multi-layer optics in X-ray diffraction*. *Adv. X-ray Anal.* **39**, 57–71.
- Schwinger, J. (1949). *On the classical radiation of accelerated electrons*. *Phys. Rev.* **75**, 1912–1925.
- Silfhout, R. G. van (1998). *A new water-cooled monochromator at DORIS III*. *Synchrotron Radiat. News*, **11**, 11–13.
- Smither, R. K. (1982). *New methods for focusing X-rays and gamma rays*. *Rev. Sci. Instrum.* **53**, 131–141.
- Winick, H. (1980). *Properties of synchrotron radiation*. In *Synchrotron Radiation Research*, edited by H. Winick & S. Doniach. New York: Plenum.
- Yoshimatsu, M. & Kozaki, S. (1977). *High brilliance X-ray source*. In *X-ray Optics*, edited by H.-J. Queisser, ch. 2. Berlin: Springer.