

## 4.2. X-RAYS

significantly reduced. More importantly, the mirrors can be fashioned into shapes that enable the emitted radiation to be brought to a focus. Ellipsoidal, logarithmic spiral, and toroidal mirrors have been manufactured commercially for use with laboratory X-ray sources. Since the X-rays are emitted isotropically from the anode surface, it is important to devise a mirror system that has a maximum angle of acceptance and a relatively long focal length.

At synchrotron-radiation sources, the high intensities that are generated over a very broad spectral range give rise to significant heat loading of subsequent monochromators and therefore degrade the performance of these elements. In many systems, mirrors are used as the first optical element in the monochromator, to reduce the heat load on the primary monochromator and to make it easier for the subsequent monochromators to reject harmonics of the chosen radiation. Shaped mirror geometries are often used to focus the beam in the horizontal plane (Subsection 2.2.7.3). A schematic diagram of the optical elements of a typical synchrotron-radiation beamline is shown in Fig. 4.2.5.2. In this, the primary mirror acts as a thermal shunt for the subsequent monochromator, minimizes the high-energy component that may give rise to possible harmonic content in the final beam, and acts as a vertical collimator. The radii of curvature of mirrors can be changed using a mechanical four-point bending system (Oshima, Harada & Sakabe, 1986). More recent advances in mirror technology enable the shape of the mirror to be changed through use of the piezoelectric effect (Sussini & Laberge, 1995).

## 4.2.5.2.2. Capillaries

Capillaries, and bundles of capillaries, are finding increasing use in situations where a focused beam is required. The radiation is guided along the capillary by total external reflection, and the shape of the capillary determines the overall flux gain and the uniformity of the focused spot. Gains in flux of 100 and better have been reported. There is, however, a degradation in the angular divergence of the outgoing beam. For single capillaries, applications are laboratory-based protein crystallography, microtomography, X-ray microscopy, and micro-X-ray fluorescence spectroscopy. The design and construction of capillaries for use in the laboratory and at synchrotron-radiation sources has been discussed by Bilderback, Thiel, Pahl & Brister (1994), Balaic & Nugent (1995), Balaic, Nugent, Barnea, Garrett & Wilkins (1995), Balaic *et al.* (1996), and Engström, Rindby & Vincze (1996). They are usually used after other monochroma-

tors in these applications and their role as a low-pass energy filter is not of much significance.

Bundles of capillaries are currently being produced commercially to produce focused beams (ellipsoidally shaped bundles) and half-ellipsoidal bundles are used to form beams of large cross section from conventional laboratory sources (Peele *et al.*, 1996; Kumakov & Komarov, 1990).

## 4.2.5.2.3. Quasi-Bragg reflectors

For one interface, the reflectivity ( $R$ ) and the transmissivity ( $T$ ) of the surface are determined by the Fresnel equations, *viz*:

$$R = |(\theta_1 - \theta_2)/(\theta_1 + \theta_2)|^2, \quad (4.2.5.3)$$

and

$$T = |2\theta_1/(\theta_1 + \theta_2)|^2, \quad (4.2.5.4)$$

where  $\theta_1$  and  $\theta_2$  are the angles between the incident ray and the surface plane and the reflected ray and the surface plane, respectively.

If a succession of interfaces exists, the possibility of interference between successively reflected rays exists. Parameters that define the position of the interference maxima, the line breadths of those maxima, and the line intensity depend *inter alia* on the regularity in layer thickness, the interface surface roughness, and the existence of surface tilts between successive interfaces. Algorithms for solving this type of problem are incorporated in software currently available from a number of commercial sources (Bede Scientific, Siemens, and Philips). The reflectivity profile of a system having a periodic layer structure is shown in Fig. 4.2.5.3. This is the reflectivity profile for a multiple-quantum-well structure of alternating aluminium gallium arsenide and indium gallium arsenide layers (Holt, Brown, Creagh & Leon, 1997). Note the interference maxima that are superimposed on the Fresnel reflectivity curve. From the full width at half-maximum of these interference lines, it can be inferred that the energy discrimination of the system,  $\Delta E/E$ , is 2%. The energy range that can be reflected by such a multilayer system depends on the interlayer thickness: the higher the photon energy, the thinner the layer thickness.

Commercially available multilayer mirrors exist, and hitherto they have been used as monochromators in the soft X-ray region in X-ray fluorescence spectrometers. These monochromators are typically made of alternating layers of tungsten and carbon, to maximize the difference in scattering-

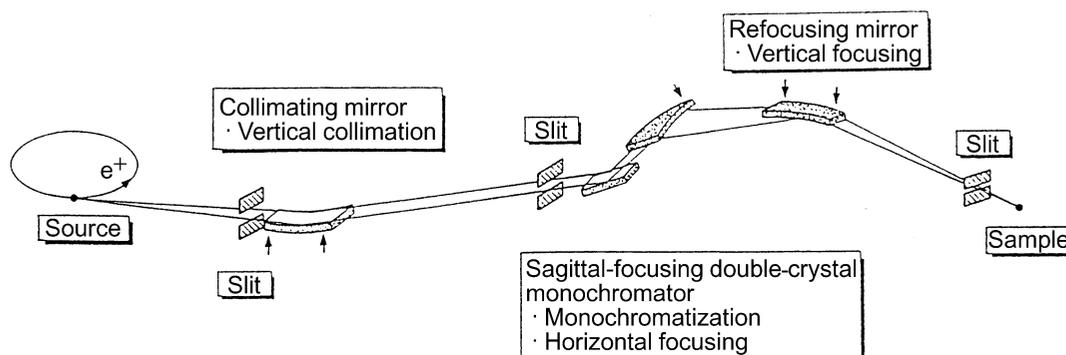


Fig. 4.2.5.2. The use of mirrors in a typical synchrotron-radiation beamline. The X-rays are emitted tangentially to the orbit of the stored positron beam. They pass through a beam-defining slit onto a mirror that serves three purposes, *viz* energy discrimination, heat absorption, and focusing, by means of a mechanical four-point bending system. The beam then passes into a double-crystal monochromator, which selects the desired photon energy. The second element of this monochromator is capable of being bent sagittally using a mechanical four-point bending system to focus the beam in the horizontal plane. The beam is then refocused and redirected by a second mirror.

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length density at the interfaces. Whilst the energy resolution of such systems is not especially good, these monochromators have a good angle of acceptance for the incident beam, and reasonably high photon fluxes can be achieved using conventional laboratory sources.

A recent development of this, the Goebel mirror system, is supplied as an accessory to a commercially available diffractometer (Siemens, 1996a,b,c; OSMIC, 1996). This system combines the focusing capacity of a curved mirror with the energy selectivity of the multilayer system. The spacing between layers in this class of mirror multilayers can be laterally graded to enhance the incident acceptance angle. These multilayers can be fixed to mirrors of any figure to a precision of  $0.3'$  and can therefore be used to form parallel beams (parabolic optical elements) as well as focused beams (elliptical optical elements) of high quality.

### 4.2.5.3. Filters

It is usual to consider only the cases where a quasimonochromatic beam is to be extracted from a polychromatic beam. Before discussing this class of usage, mention must be made of two simple forms of filtering of radiation.

In the first, *screening*, a thin layer of absorbing foil is used to reduce the effect of specimen fluorescence on photon counting, film and imaging-plate detectors. A typical example is the use of aluminium foil in front of a Polaroid camera used in a Laue camera to reduce the *K*-shell fluorescence radiation from a transition-metal crystal when using a conventional sealed molybdenum X-ray source. A 0.1 mm thick foil will reduce the fluorescent radiation from the crystal by a factor of about five, and this radiation is emitted isotropically from the specimen. In contrast, the wanted Laue-reflected beams are emitted as a nearly parallel beam, and the signal-to-noise ratio in the resulting photograph is much increased.

The second case is the ultimate limiting case of filtering, *shielding*. If it is necessary to shield an object completely from a polychromatic incident beam, a sufficient thickness of absorbing material, calculated using the data in Section 4.2.4, to reduce the beam intensity to the level of the ambient background is inserted in the beam. [The details of how shielding systems are designed are given in reference works such as the *Handbook of Radiation Measurement and Protection* (Brodsky, 1982).] In general, the use of an absorber of one atomic species will provide insufficient shielding. The use of composite absorbers is necessary to achieve a maximum of shielding for a minimum of weight. This is of utmost importance if one is designing, say, the shielding of an X-ray telescope to be carried in a rocket or a balloon (Grey, 1996). To produce shielding that satisfies the requirements of minimum weight, good mechanical rigidity, and ability to be constructed to good levels of mechanical tolerance, shielding must be constructed using a number of layers of different absorbers, chosen such that the highest-energy radiation is just stopped in the first layer, the *L*-shell fluorescent radiation created in the absorption process is stopped in the second, and the lower-energy *L* and *M* fluorescent radiation is stopped by the next layer, and so on until the desired radiation level is reached.

In the usual case involving filters, the problem is one of removing as much as possible of the *Bremsstrahlung* radiation and unwanted characteristic radiation from the spectrum of a laboratory sealed tube or rotating-anode source whilst retaining as much of the wanted radiation as is possible. To give an example, a thin characteristic radiation filter of nickel of appropriate thickness almost completely eliminates the

*Bremsstrahlung* and *K $\beta$*  radiation from an X-ray source with a copper target, but reduces the intensity in the Cu *K $\alpha$*  doublet by only about a factor of two. For many applications, this is all that is necessary to provide the required degree of monochromatization. If there is a problem with the residual *Bremsstrahlung*, this problem may be averted by making a second set of measurements with a different filter, one having an absorption edge at an energy a little shorter than that of the desired emission line. The difference between the two sets of measurements corresponds to a comparatively small energy range spanning the emission line. This *balanced-filter* method is more cumbersome than the single-filter method, but no special equipment or difficult adjustments are required. In general, if the required emission is from an element of atomic number *Z*, the first foil is made from material having atomic number *Z* - 1 and the second from atomic number *Z* + 1. A better balance can be achieved using three foils (Young, 1963). The use of filters is discussed in more detail in §2.3.5.4.2. Data for filters for the radiations in common use are given in Tables 2.3.5.2 and 2.3.5.3. The information necessary for choosing filter materials and estimating their optimum thicknesses for other radiations is given in Sections 4.2.2, 4.2.3, and 4.2.4.

It should be remembered that filtration changes the wavelength of the emission line slightly, but this is only of significance for measurements of lattice parameters to high precision (Delf, 1961).

### 4.2.5.4. Monochromators

#### 4.2.5.4.1. Crystal monochromators

Even multifoil balanced filters transmit a wide range of photon energies. Strictly monochromatic radiation is impossible, since all atomic energy levels have a finite width, and emission from these levels therefore is spread over a finite energy range. The corresponding radiative line width is important for the correct evaluation of the dispersion corrections in the neighbourhood of absorption edges (§4.2.6.3.3.2). Even Mössbauer lines, originating as they do from nuclear energy levels that are much narrower than atomic energy levels, have a finite line width. To achieve line widths comparable to these requires the use of monochromators using carefully selected single-crystal reflections.

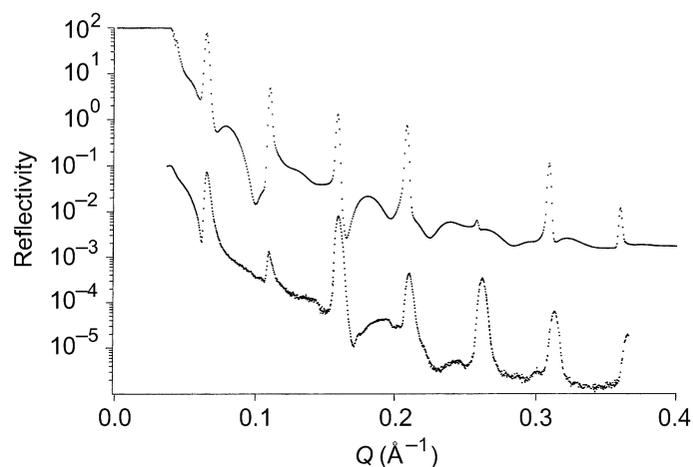


Fig. 4.2.5.3. The reflectivity of a multiple-quantum-well device is shown. This consists of 40 alternating layers of AlGaAs and InGaAs. Shown also, but shifted downwards on the vertical scale for the purpose of clarity, is the theoretical prediction based on standard electromagnetic theory.