2. INSTRUMENTATION AND SAMPLE PREPARATION
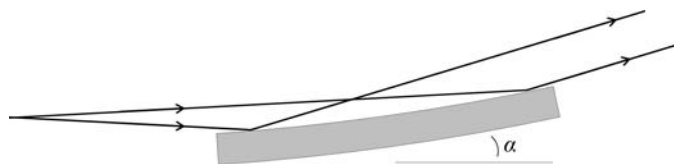

Figure 2.2.8
Curved mirror set to collimate the beam.

Figure 2.2.9
Schematic diagram of a set of refractive lenses.

curvature of the mirror caused by its own weight. Even then, very careful mounting and precise mechanics are required to achieve this level of accuracy. If placed in the polychromatic beam directly from the source, cooling of the mirror will be necessary.

Other mirror arrangements can be employed, such as a horizontal and vertical pair of focusing mirrors in a Kirkpatrick–Baez (Kirkpatrick & Baez, 1948) arrangement. Such a device might be used to produce a small focal spot for powder-diffraction measurements from a sample in a diamond anvil cell. Multilayer mirrors can also be found in service on certain beamlines.

2.2.3.3. *Compound refractive lens*

The refractive index n of a material for X-rays is given (Gullikson, 2001; Spiller, 2000) by

$$n = 1 - \delta - i\beta = 1 - \frac{r_e}{2\pi}\lambda^2 \sum_n N_n f_n,$$

where $f_n = f_1 + if_2$ is the complex scattering factor for forward scattering for atom n and N_n is the number of atoms of type n per unit volume. δ and β are known as the refractive index decrement and the absorption index, respectively, and vary with photon energy depending on the proximity of an absorption edge. The real part of the refractive index is therefore slightly less than 1, with δ typically of the order 10^{-6}–10^{-9} depending on the energy. Thus a hole drilled in a piece of metal can act like a conventional convex lens, as the hole has a higher refractive index than the surrounding metal. With such a small difference in n between hole and metal, the focusing power is very slight; however, a series of holes (Fig. 2.2.9) can be used to focus the X-ray beam over a reasonable distance (Snigirev *et al.*, 1997, 1998). For a series of cylindrical lenses, the focal length, f, is given by $f = r/2N\delta$, where r is the radius of the hole and N is the number of holes.

Note that further away from the axis of the device the X-ray beam must pass through increasing amounts of material which absorb the radiation. Hence, only relatively small holes and apertures are possible (a maximum of a few mm in diameter) and weakly absorbing metals such as Be and Al are preferred. With hard-energy photons, Ni lenses are possible, and indeed the construction of such a device is a compromise between refractive power, absorption, aperture and the desired focal length. Such devices can be placed in the monochromatic beam or in a polychromatic beam with cooling.

Many variants of the basic scheme exist, with lenses pressed from foil with a parabolic form to eliminate spherical aberrations, with axial symmetry to focus in both the horizontal and vertical simultaneously (Lengeler *et al.*, 1999), etched *via* lithography from plastic or other material, or with a more complex profile to minimize the amount of redundant material attenuating the transmitted beam by absorption and so allowing a larger aperture. A 'transfocator' can be constructed whereby series of lenses can be accurately inserted or removed from the beam path, thus allowing the focusing power to be adjusted depending on the
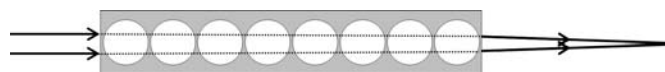
desired focal distance and the wavelength of the experiment (Vaughan *et al.*, 2011).

2.2.4. Diffractometers

Most powder-diffraction beamlines are angle dispersive, operating with monochromatic radiation. When scanning a detector arm or employing a curved position-sensitive detector (PSD), detection is normally in the vertical plane because the polarization of the radiation in the plane of the synchrotron orbit means there is very little effect on the intensities due to polarization. By contrast, if diffracting in the horizontal plane, the projection of the electric vector onto the direction of the diffracted beam means that the intensity is reduced by a factor of $\cos^2 2\theta$, going to zero at $2\theta = 90°$, and so horizontal detection is less useful unless working at hard energies when 2θ angles are correspondingly small. In addition, for the highest angular resolution, the natural beam divergence in the vertical plane is usually lower than in the horizontal plane, particularly if the instrument has a bending magnet or wiggler as its source.

In general, diffractometers are heavy-duty pieces of equipment and are designed to have excellent angular accuracy while working with substantial loads. A high degree of mechanical accuracy is required to match the high optical accuracy inherent in the techniques employed. The calibration of the incident wavelength and any 2θ zero-point error is best done by measuring the diffraction pattern from a sample such as NIST standard Si (640 series), each of which has a certified lattice parameter (see Chapter 3.1). It is also good practice to measure the diffraction pattern of a standard sample regularly and whenever the instrument is realigned or the wavelength changed, to be sure that everything is working as expected.

Monochromatic instruments can have an analyser crystal or long parallel-foil collimators in the diffracted beam (a so-called parallel-beam arrangement), or can scan a receiving slit, or possess a one- or two-dimensional PSD, similar to Debye–Scherrer or Laue front-reflection geometry. Instruments equipped with a PSD can collect data much faster than those with a scanning diffractometer, so are exploited especially for time-resolved measurements. They may also have advantages for rapid data collection if the sample is sensitive to radiation, or be helpful if the sample is prone to granularity or texture to assess the extent of the problem.

Instruments can also be equipped with a sample changer, allowing measurements on a series of specimens, perhaps prepared by systematically changing the conditions of synthesis or the composition in a combinatorial approach. The use of beam time can be optimized with minimal downtime due to interventions around the instrument, and with the possibility to control the data acquisition remotely if desired.

2.2.4.1. *Parallel-beam instruments*

Cox *et al.* (1983, 1986), Hastings *et al.* (1984) and Thompson *et al.* (1987) described the basic ideas behind these instruments *via* their pioneering work at CHESS (Cornell, USA) and NSLS (Brookhaven, USA). The highly collimated monochromatic