

2. INSTRUMENTATION AND SAMPLE PREPARATION

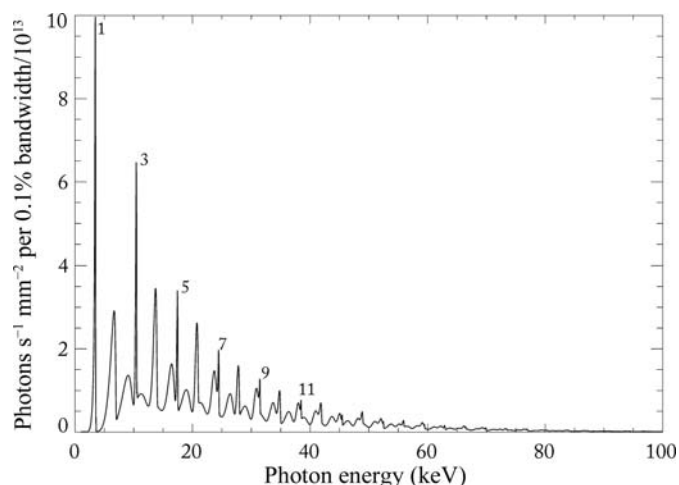


Figure 2.2.6
Photon flux versus energy through a 1-mm² aperture 30 m from the source, 0.1% bandwidth, for an ESRF u35 undulator (magnetic periodicity 35 mm, 1.6 m long, magnetic gap of 11 mm, peak magnetic field $B_0 = 0.71$ T, electron energy 6 GeV, $K = 2.31$, storage-ring current 200 mA). Odd-numbered harmonics are labelled, which are those usually employed for powder-diffraction experiments as they have maximum intensity on axis.

becomes highly collimated in the horizontal and vertical directions. Thus, the radiation from an undulator is concentrated into a central on-axis cone (fundamental and odd harmonics), surrounded by rings from higher-order even harmonics. The flux density arriving on a small sample from this central cone is therefore very high. With high on-axis intensity, it is therefore the undulators that provide the beams with the highest spectral brightness at any synchrotron-radiation source. The interference also modifies the spectrum of the device, which has a series of harmonics derived from a fundamental energy. At a horizontal angle θ to the axis of the insertion device, the wavelength of harmonic n is given by

$$\lambda_n = \frac{1 + (K^2/2) + \gamma^2\theta^2}{2n\gamma^2} \lambda_u,$$

which can be simplified on axis ($\theta = 0$) to

$$\lambda_n [\text{\AA}] = 1.3056 \frac{1 + K^2/2}{nE_e^2 [\text{GeV}]} \lambda_u [\text{mm}]$$

or

$$\varepsilon_n [\text{keV}] = 9.50 \frac{nE_e^2 [\text{GeV}]}{\lambda_u [\text{mm}](1 + K^2/2)}.$$

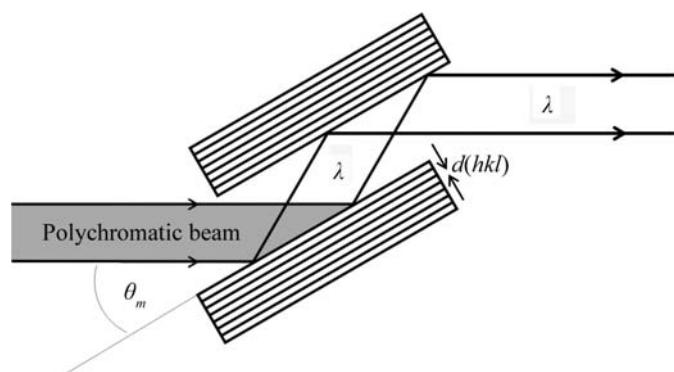


Figure 2.2.7
Double-crystal monochromator arrangement.

On axis, only odd-numbered harmonics are emitted and it is these that are usually employed in a powder-diffraction experiment. The horizontal and vertical divergence of the radiation emerging from an undulator is of the order of $1/[(nN)^{1/2}\gamma]$, where N is the number of magnetic periods making up the device. The spectrum of an undulator at a 6-GeV source with a 35-mm magnetic period is shown in Fig. 2.2.6. By carefully shimming the magnetic lattice so that it is highly regular, the higher-order harmonics persist, allowing the undulator to be a powerful source of high-energy X-rays. Any imperfections in the magnetic periodicity cause the higher-order harmonics to broaden and fade away, reducing the utility of the device at higher energies.

2.2.2.2.3. Tuning

For insertion devices the magnetic field can be modified by changing the vertical distance between the magnetic poles. By opening the gap, the magnetic field and K decrease following

$$B_0 \simeq B_r \exp(-\pi G/\lambda_u),$$

where B_r is proportional to the remanent magnetic field, which depends upon the nature of the magnets used in the insertion device, and G is the magnetic gap. Decreasing K for an undulator means that the energy of the fundamental harmonic increases; however, this is at the expense of the intensities of the higher harmonics. Thus the insertion device can be tuned to produce high intensity at the wavelength most suitable for a particular measurement. The smallest gap possible for a device depends on the design of the storage-ring vacuum vessel in which the electrons circulate. It is difficult to have a vessel smaller than about 10 mm high, and hence for an externally applied field a minimum magnetic gap of about 11 mm is to be expected. For smaller gaps, the magnets must be taken into the vacuum of the storage ring, a so-called ‘in-vacuum’ insertion device.

2.2.3. Optics

The intense polychromatic beam from the source needs to be conditioned before hitting the sample and diffracting. In the simplest experimental configuration, the white beam is used in an energy-dispersive experiment, and conditioning may involve no more than using slits to define the horizontal and vertical beam sizes and suppress background scattering. More usually, monochromatic radiation is employed, and the desired wavelength is chosen from the source by a monochromator. A monochromator consists of a perfect crystal, or a pair of crystals, set to select the chosen wavelength by Bragg diffraction. Additional optical elements can also be incorporated into the beamline for focusing, collimation, or for filtering out unwanted photons to reduce heat loads or remove higher-order wavelengths transmitted by the monochromator.

2.2.3.1. Monochromator

The monochromator is a crucial optical component in any angle-dispersive powder-diffraction beamline, and consists of one or a pair of perfect crystals (*e.g.* Beaumont & Hart, 1974), Fig. 2.2.7, set to a particular angle to the incident beam, θ_m , that transmits by diffraction wavelengths that satisfy the Bragg equation, $n\lambda = 2d(hkl) \sin \theta_m$, where $d(hkl)$ is the lattice spacing of the chosen reflection. Note that photons from higher-order reflections can also be transmitted, corresponding to wavelengths λ/n , depending on the structure factor of the n th-order reflection and its Darwin width, but these can be eliminated by use of a