International Tables for Crystallography (2012). Vol. F, Section 6.2.2, pp. 172–174.

collection rate and accuracy (Schoenborn *et al.*, 1985, 1986; Schoenborn, 1992*b*).

A concept related to the MWPC is the micro-strip gas chamber (MSGC). With the MSGC, the general principles of gas detection and amplification apply; however, the anode is deposited on a suitable substrate (Oed, 1988, 1995; Velletaz *et al.*, 1997). The MSGC can potentially improve the performance of the MWPC in some applications, particularly with respect to spatial resolution and count-rate capability.

6.2.1.4.2. Image plates

The principles underlying the operation of an image plate (IP) are presented in detail in Chapter 7.2. Briefly, the important difference between an IP for X-ray and neutron detection is the presence of a converter (either Gd_2O_3 or ^6Li). The role of the converter is to capture an incoming neutron and create an event within the IP that mimics the detection of an X-ray photon. For example, neutron capture in Gd produces conversion electrons that exit the Gd_2O_3 grains, enter neighbouring photostimulated luminescence (PSL) material and create colour centres to form a latent image (Niimura *et al.*, 1994; Takahashi *et al.*, 1996). A neutron IP may have a virtually unlimited area and a shape limited only by the requirement to locate the detection event in a suitable coordinate system. With a neutron-detection efficiency of up to 80% at ~1–2 Å, a dynamic quantum efficiency of ~25–30% can be obtained. The dynamic range is intrinsically $1{:}10^5$. The spatial resolution is primarily limited by scattering processes of the readout laser beam, and measured line spread functions are typically 150–200 µm. The γ sensitivity is high and may restrict the application to instruments with low ambient γ background.

Neutron IPs are integrating devices well suited to data-acquisition techniques with long accumulation times, such as Laue diffraction (Niimura *et al.*, 1997) and small-angle scattering. On-line readout is a distinct advantage (Cipriani *et al.*, 1997).

6.2.1.5. Instrument resolution functions

For accurate data collection, the instrument smearing contribution to the data must be known with some certainty, particularly when data are collected over an extended range with multiple instrument settings. A balance must be struck between instrument smearing and neutron flux at the sample position; however, careful instrument design can produce: (i) a good signal-to-background ratio, thereby partially offsetting the flux limitation, and (ii) facilities and procedures for determining the instrument resolution function (Johnson, 1986).

As an example, instrumental resolution effects in the small-angle neutron scattering (SANS) technique have been investigated in some detail. A 'typical' SANS instrument is located on a cold neutron source with an extended (and often variable) collimation system. The sample is as large as possible and the detector is large with low spatial resolution. The instrument is best described by pin-hole geometry. Three major contributions to the smearing of an ideal curve are: (i) the finite λ, (ii) $\Delta\lambda/\lambda$ of the beam and (iii) the finite resolution of the detector. Indirect Fourier transform, Monte Carlo and analytical methods have been developed to analyse experimental data and predict the performance of a given combination of resolution-dependent elements (*e.g.* Wignall *et al.*, 1988; Pedersen *et al.*, 1990; Harris *et al.*, 1995).

6.2.2. Spallation neutron sources

Another phenomenon, quite different from the fission process (Section 6.2.1), that will produce neutrons uses high-energy particles to interact with elements of medium to high mass numbers. This process, called spallation, was first demonstrated by Seaborg and Perlman, who showed that the bombardment of nuclei by high-energy particles results in the emission of various nucleons. The nuclear processes involved in spallation (Prael, 1994) are complex and are summarized in Fig. 6.2.2.1. These processes have been investigated in some detail, and excellent background information is available (Hughes, 1988; Carpenter, 1977; Windsor, 1981). Present-day spallation sources typically use high-energy protons from an accelerator to bombard a heavy-metal target, such as W or U, and come in two types, using either a pulsed proton beam (*e.g.* ISIS or LANSCE) or a 'continuous' proton beam (SINQ).

The high-energy neutrons produced by spallation are moderated in a reflector region to intermediate energies and then reduced to thermal energies in a hydrogenous medium called the moderator (Russell *et al.*, 1996). These thermal neutrons are then extracted *via* beam pipes. A typical layout of a target system with reflectors and moderators is shown in Fig. 6.2.2.2.

The neutrons produced by the proton pulse travel along beam pipes as a function of their velocity, proportional to their energy.

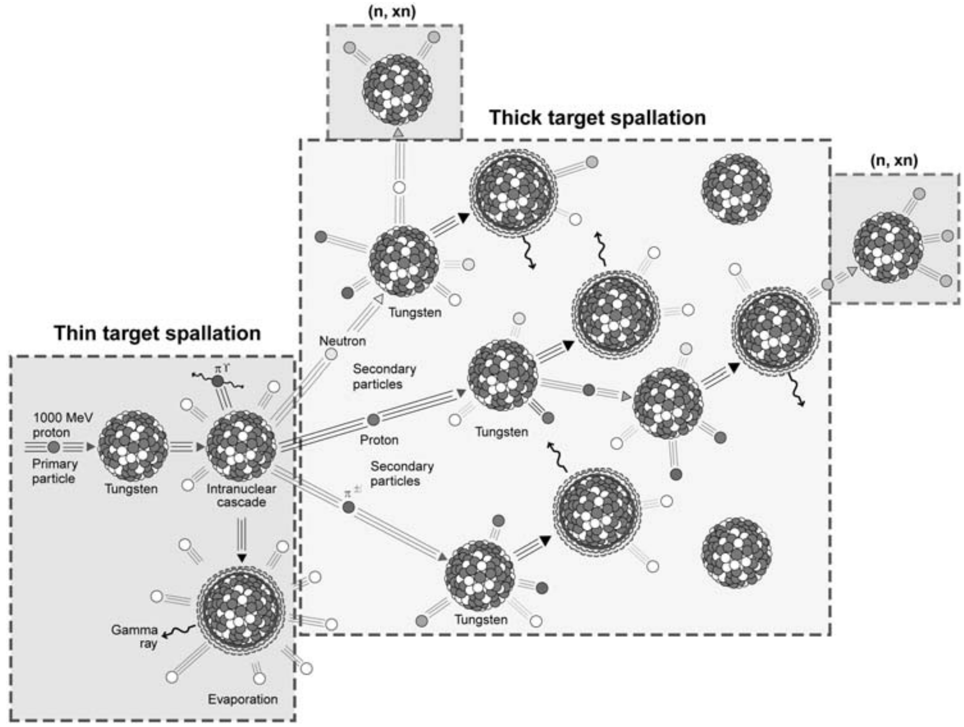

Figure 6.2.2.1
Schematic presentation of the various nuclear processes encountered in spallation. The numerical analysis of these processes is carried out by two Monte Carlo-based codes – the *LAHET* code models the higher-energy nuclear interactions, while the *HMCNP* code models the thermal interactions and the transport of neutrons to the sample.

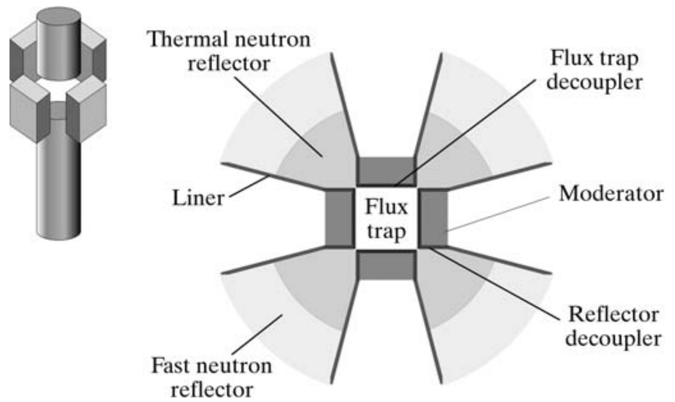

Figure 6.2.2.2
Schematic diagram of a spallation target module depicting the inner Be and outer Pb reflector. Moderators are positioned close to the flux trap, which is between the upper and lower tungsten targets. This schematic includes the location of the various decoupling agents (Cd) for fully decoupled moderators. For the partially coupled moderator system being fabricated for a protein-crystallography station at Los Alamos, the depicted decouplers affecting the given moderator are removed and replaced by a single decoupling layer placed between the outer Pb and the inner Be reflector. This can be done with a split-target arrangement that utilizes a two-tiered moderator system, permitting coupled moderators to be on one tier and decoupled moderators on the other.

At a given distance from the target, neutrons of different energies are observed to arrive as a function of time, with the short-wavelength neutrons arriving first, followed by the longer-wavelength neutrons. Diffraction experiments are therefore carried out with pulsed ‘polychromatic’ neutron beams as a function of time; a time-resolved Laue pattern results. Clearly, the energy resolution of these beams depends on the volume of the neutron source. To achieve high energy resolution, the volume from which thermal neutrons are extracted is limited to the moderator by suitable use of liners and poisons (Fig. 6.2.2.2) that prevent thermal neutrons produced in the reflector from streaming into the beam pipe (decoupled moderators). The use of liners and poisons achieves high energy resolution, but at the expense of flux. The omission or reconfiguration of liners and poisons allows higher flux, but results in lower energy (wavelength) resolution (see Section 6.2.2.2).

6.2.2.1. Spallation neutron production

Pulsed spallation neutrons are produced by protons generated by a particle accelerator (linac) with a frequency typically in the range 10 to 120 Hz. The proton pulses are often shaped in compressor rings to shorten the pulses from the millisecond range to less than a microsecond in duration, with currents reaching the sub-milliampere range at energies of 800 MeV or higher. The planned new spallation source at the Oak Ridge National Laboratory will have a proton energy of 1.2 GeV with a power of 1 MW and a repetition frequency of 60 Hz. The high-energy protons hit a cooled target (typically W or U) and produce high- and medium-energy neutrons in equivalent bursts. The fast neutrons are moderated in the surrounding reflector and returned to the moderator (Fig. 6.2.2.2). In addition to direct collision interactions, the high-energy neutrons also produce low-energy neutrons *via* (n, *x*n) reactions in the Pb and Be reflectors. Final moderation to the thermal energies used for diffraction experiments is completed *via* interactions with light elements, such as H_2O or liquid H_2, in the moderator module.

6.2.2.2. Moderators

Moderators for pulsed spallation neutron sources are nearly always composed of hydrogenous material of about 1 l in volume. Either a thermal or fast reflector surrounds the moderator. Reflectors composed of materials with strong neutron slowing-down properties, such as Be or D_2O, are called thermal reflectors; fast reflectors are composed of materials with weaker slowing-down powers, such as Pb or Ni. In order to retain a narrow pulse width in time, thermal neutrons produced in the reflector region are prevented from reaching the moderator module by judicious use of liners and poisons (typically Cd or Gd) that allow transmission of fast (high and intermediate energy) neutrons, but are opaque to thermal neutrons. Such a moderator arrangement is said to be decoupled, and all thermal neutrons extracted by the beam pipe originate in the moderator itself. For a 0.25 μs-long proton pulse, the target (W or U) produces a fast neutron burst of about 0.5 μs in duration. These very high energy neutrons are slowed down in the reflector and are reflected back into the moderator to produce a thermal neutron pulse of about 1 μs duration. Since thermal neutrons produced in the reflector are prevented from reaching the moderator by the use of liners and poisons, the experiment sees only thermal neutrons originating in the moderator.

By moving the decoupling and poison layers away from the moderator and into the reflector, one can redefine how actively a reflector communicates *via* neutrons with a moderator and how some of the thermal neutrons produced in the reflector are extracted (Schoenborn *et al.*, 1999). The result is an increase in flux, both peak- and time-integrated, but at the expense of the sharply defined time distribution. With the elimination of all liners and poisons, a fully coupled system is obtained with a flux gain of about 6× but with poorer wavelength resolution. The wavelength distribution at a given distance from the moderator is shown in Fig. 6.2.2.3(*a*) for the fully decoupled case and in Fig. 6.2.2.3(*b*) for the fully coupled case. For a decoupled moderator, the slowing-down power of the reflector is not as critical as it is for the coupled one. In the coupled moderator, it is beneficial to use a thermal reflector in the volume immediately surrounding the moderator because this enhances the peak thermal neutron flux. The decay constant of the neutron pulse can be tailored to match the diffractometer resolution by using a composite reflector composed of an inner thermal reflector and an outer fast reflector. The outer reflector can have a moderate thermal neutron absorption cross section, or the inner reflector can be decoupled from the outer reflector in the same manner that a moderator is decoupled from a reflector. The decay constant can then be varied by simply adjusting the size of the inner reflector (Russell *et al.*, 1996). The wavelength or energy distribution of thermal neutrons produced in the moderator is dependent on the temperature of the moderating medium, as described in Section 6.2.1.2.

For neutron protein crystallography, a moderator with an intermediate temperature between a cold and thermal moderator would be most appropriate. This can be achieved with a composite moderator composed of a thermal and a cold moderator in a symbiotic configuration, or a cold methane system.

6.2.2.3. Beamline optics

A chosen wavelength band (say, 1 to 6 Å) is selected by the use of rotating disks (called choppers) composed of neutron absorbing and transparent material. These choppers are synchronized to the proton pulse. The T0 chopper can open the

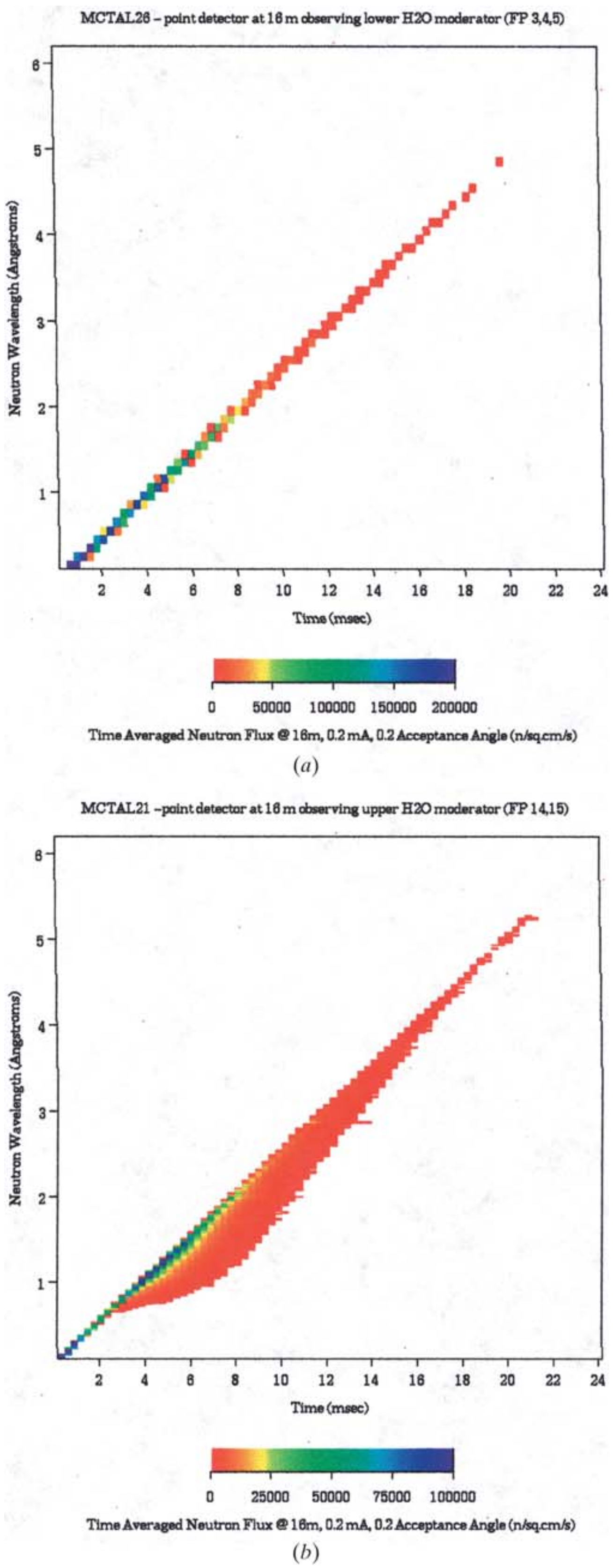

Figure 6.2.2.3
Neutron flux given as a time–wavelength spectrum for (*a*) a fully decoupled system and (*b*) for a fully coupled system. Both spectra are based on Monte Carlo codes (*LAHET* and *HMCNP*) and are calculated for a target-to-sample distance of 10 m. Comparison of such Monte Carlo results calculated using the geometry of an existing beamline shows agreement with measured values to within 10%.

beam a short time after the impact of the proton pulse and stop the high- and intermediate-energy radiation from reaching the sample and the detector. The T1 chopper can select the long-wavelength edge and prevent frame overlap. Since the T0 chopper is designed to stop the initial γ and high-energy neutron radiation, it is usually made of thick (30 cm) blades of Ni, while the T1 chopper is simpler in construction since it is designed to stop only thermal neutrons.

The flight-path lengths of relevant spallation neutron instruments are quite long; the Los Alamos Spallation Neutron Source has a 28 m path length for its protein crystallography station on a partially decoupled moderator. For a fully decoupled system, a flight-path length of 10 m would provide adequate energy resolution.

For protein crystallography, a beam divergence matched to the mosaicity of the crystal provides the best peak-to-background ratio. For such cases, a beam divergence of $\pm 0.1^\circ$ can be achieved using circular collimating disks of Boral or Boron-Poly to form a cone that views most of the moderator (typically 12×12 cm) and channels the neutron beam onto the detector with a final aperture of millimetre dimensions. Another approach uses focusing mirrors, and calculations show that toroidal geometry will produce a gain in intensity of 1.5 to 2 times, depending on flight distance and beam divergence (Schoenborn, 1992*a*).

6.2.2.4. *Time-of-flight techniques*

Because of the time structure inherent at a spallation source, diffraction experiments are carried out as a function of time and use a large part of the neutron energy spectrum. For protein crystallography, this wavelength range might cover from 1 to 5 Å, depending on the unit-cell size and the moderator used. This is particularly advantageous and allows the collection of data in a quasi-Laue fashion (Schoenborn, 1992*a*) without the drawback of spot overlap normally encountered in Laue patterns. Data are collected in a stroboscopic fashion, synchronized to the pulsed nature of the source, with each separately recorded time frame producing a Laue pattern from a narrow, gradually increasing wavelength band. The summation of all time frames will produce a true Laue pattern. The collection and analysis of these quasi-Laue patterns (time frames) will eliminate spot overlap and yield a greatly improved peak-to-background ratio, since the integrated background is produced only by the small wavelength band responsible for a particular diffraction peak.

6.2.2.5. *Data-collection considerations*

Single-event-counting multiwire chambers with centroid-finding electronics (introduced in Section 6.2.1.4) are well suited for the type of time-sliced data collection that is mandatory for spallation neutron instruments. For large, high-resolution, multi-segmented detectors collecting about 100 time slices per cycle, data memories in the order of 100 million pixels are required. The number of time slices that needs to be collected to produce the optimum peak-to-background ratio depends on the characteristics (wavelength bandwidth) of the coupled or decoupled moderator.

Data-integration techniques are similar to those for the classic reactor case (Section 6.2.1.5), but contain a time (wavelength) dimension and no crystal stepping. The crystal is stationary and the reflection is 'scanned' as a function of time by the wavelength band. Time-dependent reflection overlap, caused by long pulse decay (particularly observed in fully coupled moderators), can be a problem. Such overlaps can be minimized by using a partially coupled moderator (Schoenborn *et al.*, 1999).