The Small-Angle Neutron Scattering Spectrometer at the University of Missouri Research Reactor*

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Abstract

A small-angle neutron scattering (SANS) spectrometer has been constructed at the University of Missouri Research Reactor Facility (MURR). The design of the MURR-SANS is unusual in that the size of the reactor containment building constrains the flight path to be vertical. This is achieved by Bragg scattering upward through 90° from a set of slightly misaligned pyrolytic graphite crystals to provide a neutron beam at 4.75 Å with a wavelength spread of approximately 4.1%. The beam incident on the sample is defined by two matched variable apertures located either 3.0 or 4.5 m apart. The evacuated scattered flight path is designed with removable extensions to match the primary flight path in length. The instrument has an automatic sample handling capability provided by its own dedicated PDP 11/03 computer. The detector is a large assembly of commercially available linear 3He detectors as an economic alternative to a crossed-wire two-dimensional multi-detector. An array of 43 position-sensitive proportional counters, 24 in (609.6 mm) long and 0.5 in (12.7 mm) in diameter, using charge division gives a spatial resolution of 5 x 12.7 mm. The area-averaged detector efficiency is about 84% at a wavelength of 4.75 Å. The range of scattering vectors that can be measured is 0.005 < Q < 0.15 Å⁻¹. The instrument is well suited to a wide variety of experiments on specimens having characteristic dimensions between 30 and 500 Å. MURR-SANS is designed as a user-oriented facility which provides both reasonable resolution and intensity on sample at a modest cost, and forms part of a neutron scattering center.

1. Introduction

Small-angle neutron scattering (SANS) has now become an important technique in the study of metallurgical, polymeric and biological materials (Schmatz, Springer, Schelten & Ibel, 1974). Structural features in the spatial range of 10⁻⁴ to 10⁻¹ Å may be observed by the analysis of the scattering of neutrons in the wave-vector transfer range 10⁻⁴ < Q < 1 Å⁻¹ produced by fluctuations in the nuclear scattering density within the specimen. Excellent reviews are available of the applications of SANS to materials science (Gerold & Kostorz, 1978), polymer science (Higgins & Stein, 1978) and biological science (Stuhrmann & Miller, 1978). In addition, the recent developments in instrumentation and data analysis have been reviewed (Schelten & Hendricks, 1978).

The long-flight-path, long-wavelength neutrons and low scattered intensities characteristic of small-angle scattering experiments make SANS spectrometers appropriate to the 'cold source' beam lines of reactors (Schelten, 1972; Ibel, 1976; Galotto, Pizzi, Walter, Angelastro, Cerullo & Cherubini, 1976), though instruments have been built at reactors without cold sources (Haywood & Worcester, 1973; Mozer, 1977; Schoenborn, Alberi, Saxena & Fischer, 1978; Hofmeyr, Mayer & Tillwick, 1979; Koehler & Hendricks, 1979; Child & Spooner, 1980). In addition, SANS instruments have required the construction of elaborate large-area position-sensitive neutron detectors. We describe a SANS spectrometer with a multidetector installed on a medium flux 10 MW reactor. The MURR-SANS spectrometer has an intensity of 1.4 x 10⁸ m⁻² s⁻¹ for a resolution defined by a minimum scattering vector of 4.4 x 10⁻³ Å⁻¹.

The instrument design combination of a 4.75 Å incident beam and a vertical flight path was chosen to overcome the lack of a cold neutron source and the space limitations of the reactor containment hall. Pyrolytic graphite was chosen as a monochromator since it scatters 4.75 Å neutrons through 90°, and at this wavelength pyrolytic graphite has excellent reflectivity and the MURR reactor has adequate flux. In addition, pyrolytic graphite can be obtained with a mosaic spread appropriate to yield the optimum wavelength spread for this instrument. Advantages of the vertical beam geometry are that the gravity effect on the neutron trajectory is negligible and that liquid samples are more easily accommodated.

We have designed and built our own two-dimensional multidetector rather than buy the sophisticated and costly X-Y detectors offered by the LETI company of Grenoble, France (Allemand et al., 1975), or by Oak Ridge National Laboratory (Borkowski & Kopp, 1975) and TEC Inc. The instrument uses as a
multidetector an array of commercially available linear position-sensitive proportional counters formed into a 540 × 600 mm area detector. The 43 detector array provides a position resolution of 5 × 12.7 mm at data rates greater than 10^4 counts s⁻¹, with an area-averaged detector efficiency for 4.75 Å neutrons of approximately 84%.

2. Spectrometer design

2.1. The monochromator system

Fig. 1 is a schematic view of the MURR-SANS spectrometer. The primary beam for the instrument is obtained from the upper half of a 150 mm diameter beam tube which is shared with the double-crystal monochromator system of another instrument. The primary reactor beam passes through a rectangular collimator so that the entire vertical height of the effective source (the beryllium reflector surrounding the core) is utilized. The source flux of this beam port is estimated to be 10¹⁸ neutrons m⁻² s⁻¹ with a considerable contamination of fast neutrons.

Initial estimates (see Appendix A) indicate that we should expect a flux of 1.1 × 10¹⁴ neutrons m⁻² s⁻¹ at the monochromator assembly. The results of gold foil measurements give a thermal flux of 1.0 × 10¹⁴ neutrons m⁻² s⁻¹ and an epithermal flux of 3.0 × 10¹² neutrons m⁻² s⁻¹. These measurements indicate that the beam-port flux is as expected, though with a somewhat harder spectrum than anticipated.

The size of the reactor containment building constrains the spectrometer axis to be vertical, which is achieved by Bragg scattering upward through 90° from the monochromator assembly. A set of slightly misaligned pyrolytic graphite crystals was chosen as the monochromator in order to maximize reflectivity. The monochromator is composed of nine 12.7 × 44.5 mm crystals with a nominal thickness of 1.6 mm and mosaic width of 0.8°. They are assembled in a cassette in sets of three; the crystals in each set are offset one from the other by 0.4° in the scattering plane, and each set from each other by 0.2° out of the plane (see Fig. 2). This yields a composite crystal with an effective width of 1.93° in the scattering plane, while the spread is 1.01° in the direction perpendicular to the scattering plane. These values were obtained by measurements on another diffractometer (see Appendix B). The reflectivity is about 84.7%, which is somewhat less than that (90.2%) expected for one crystal of that thickness (see Appendix B).

The 38 × 44.5 mm crystal cassette is placed 3-30 m from the effective source plane inside the initial heavy concrete shield and 0.2 m outside the reactor biological shield. The cassette is held in a goniometer so that the crystals can rotate about two perpendicular axes through the plane of the cassette, and the whole goniometer–crystal assembly is held in the shielding to intersect the upper part of the beam. In this way, the monochromator orientation can be optimized by intensity measurements at the sample position with an end window detector.

The wavelength of the spectrometer beam is centered around 4.75 Å with the crystal mosaics matched to the angular aperture of the primary collimation to produce the maximum intensity with a wavelength spread ∆λ/λ (FWHM) of approximately 4.1%, determined by the vertical divergence. Our initial measurements were performed with crystals of 1.2° mosaic which is too wide to diffract maximum intensity onto the target owing to the perpendicular divergence factor, as discussed in
§ 3.1. A better match, and therefore improvement in the instrument intensity, is obtained with 0.4° mosaic crystals, though with the presently available 0.8° mosaic crystals we have optimized the diffracted intensity (see Appendix A).

2.2. Beryllium filter and flux monitor

The monochromated beam passes vertically upward through a beryllium polycrystalline filter cooled to liquid-nitrogen temperature. This filter removes all those neutrons whose wavelengths are below the Bragg cut-off at 4.05 Å, including higher-order Bragg-reflected neutrons and fast neutrons. The beam intensity is decreased by a factor of 1.7 when the filter is at room temperature.

The filter cryostat sits inside the beam-port primary shield immediately above the monochromating crystal assembly. A $^{235}$U fission-chamber proportional counter detector is placed directly above the filter and used as a beam monitor for experiments. It has an efficiency of about $10^{-4}$ and can monitor intensities up to $10^{8}$ neutrons s$^{-1}$. This is used to normalize data on account of the short-term variations in flux from the reactor.

Counting rates with an end window $^3$He detector at the sample position indicate that small-angle scattering in the beryllium filter creates an additional divergence of the beam. Consequently, the filter should be kept as short as possible to limit this beam broadening and allow a maximum flux of 4.75 $\text{nucleons}$ to reach the specimen. This length must be compromised with enough beryllium to remove higher orders of reflection from the graphite and reduce the fast-neutron background. The spectrometer operates adequately with 150 mm of beryllium.

2.3. Flight paths

The key components of the incident and scattered neutron flight paths are illustrated in Fig. 1. The 3 m primary flight path consists of two concentric tubes, with the inner tube evacuated and the annulus filled with borated paraffin. Two tapered pins center the primary flight path above the monochromating crystals, with the weight supported by a platform cantilevered off the reactor biological shield wall. The upper flange of the tube can be adjusted for alignment. A cadmium shielded extension of 1.5 m can be added to the lower-flight-path tube to lengthen the total primary flight path to 4.5 m.

Above the platform is a 6.1 m high tower of aluminum channeling, inside which are four ground steel shafts aligned parallel to each other. The ground shafts form a system of guide rails for the movement of the upper (scattered) flight-path tube along the spectrometer axis. Alignment of the whole spectrometer was accomplished using the spectrometer beam and a detector placed at various heights inside the tower.

The scattered flight path consists of a 2.13 m long pipe in addition to one of two extension tubes, so that the total secondary or scattered flight-path tube length can be matched to the primary flight path of either 3 or 4.5 m. A large square aluminum box housing the 43 linear position-sensitive detectors, which run parallel to the reactor face, is attached to the top of the secondary flight tube. The tube is attached to the ground shafts with four sets of bearings, each of which have adjustments to place the center of the detector coincident with the spectrometer axis. The entire secondary flight path is surrounded by 25 mm of boron carbide as shielding. The whole assembly may be moved vertically inside the tower by the reactor crane to adjust the path lengths or to change the sample environment.

Both the primary and secondary flight paths are evacuated to $\sim 1 \text{ Pa}$ with a mechanical pump. A vacuum of 100 Pa is sufficient to suppress parasitic neutron scattering due to air. The beam intensity at the sample position is decreased by 31% when the 4.5 m lower flight-path tube is not evacuated. The neutron windows of all the flight-path tubes are 2.5 mm thick single-crystal silicon to reduce small-angle scattering. These 50 mm diameter windows are held at their edges on both sides by O rings.

2.4. Sample environment

The primary beam of the spectrometer is defined by two variable-diameter circular apertures located either 3 or 4.5 m apart at each end of the primary flight path tube. A selection of four aperture sizes between 2.5 and 20 mm diameter is provided by a stepper-motor driven wheel which allows the resolution of the instrument to be changed. The first aperture wheel is located immediately above the beryllium filter and fission-chamber monitor, below the entrance to the primary flight path. The second aperture wheel is placed immediately below the sample table. The upper aperture wheel and the sample table are mounted on a plate which may move vertically along the ground steel shafts. Fine lateral positioning of the wheel is accomplished with an $x$-$y$ translation stage. Minor adjustments of the position of the direct neutron beam on the detector can be obtained by shifts in the upper collimating aperture wheel.

The present sample changer consists of a plate which can rotate about a vertical axis and accommodate eight specimens in 50 mm diameter holes on a 280 mm diameter circle. This sample table is stepper-motor controlled and can be placed over the upper aperture. Light emitting/sensing diodes detect when the sample is positioned correctly. The spectrometer design also allows the upper flight tube to be raised so that more complicated specimen environments, such as an oven or a cryostat which are being planned, can be accommodated. In this way, the sample position is uncomplicated and can be held in a helium atmosphere to eliminate air scattering.

2.5. The position-sensitive detector

The overall detector system consists of the proportional counters, their analog/digital electronic modules,
an M6809 microcomputer, a PDP 11/03 computer for real-time analysis and a PDP 11/40 with 15 Mbyte disk capacity for off-line data storage and analysis.

The two-dimensional detector is a large assembly of one-dimensional position-sensitive proportional counters. The detector elements were developed to our specifications by Reuter Stokes, and are coupled to their electronic components to form an effective and economical multidetector (Berliner, Mildner, Pringle & King, 1981). Unlike other detectors made in a single envelope [such as the LETI design (Allemand et al., 1975) used at ILL, Grenoble, at AWRE Aldermaston and at AERE Harwell, or the Borkowski–Kopp design (Borkowski & Kopp, 1968) used at ORNL and at ANL], should one detector element malfunction, the detector or its electronics may be replaced easily with little instrument downtime. In addition, the front wall scattering of a monolithic detector is essentially eliminated through the use of thin [0.010 in (0.254 mm)] stainless-steel walls.

The detector array consists of 43 commercially available cylindrical tubes of 0.5 in (12.7 mm) diameter and 24 in (609.6 mm) sensitive length, with a central 0.0006 in (0.0152 mm) diameter nickel-chrome anode. The detector cells have an $x$–$y$ geometry with a spatial resolution of $5 \times 12.7$ mm. Each tube is filled with 6 atm ($6.078 \times 10^5$ Pa) of $^3$He and 4 atm ($4.052 \times 10^5$ Pa) of argon (including 5% CO$_2$). The area-averaged detector efficiency is about 84%, at a wavelength of 4.75 Å.

Fig. 3 is a view of the multidetector as seen by the beam. Each of the detectors has its active length within the vacuum of the secondary flight tube, but its high-voltage connection outside. This prevents electrical breakdown in a vacuum, but requires many vacuum seals which are accommodated by having two levels of detectors 19 mm apart. The separation of levels has a negligible effect on the solid angle subtended by each detector at the sample for a scattering flight path of 3 m or more. Also shown is one of the two 16 μm thick gadolinium beam stops mounted on a thin aluminum rod that may be rotated under stepper-motor control into the center of the detector box to reduce the direct beam count rate. The beam-stop sizes are commensurate with the two frequently used primary beam resolutions. Since they transmit about $5 \times 10^{-3}$ of the direct beam, they may be used for an easy determination of the specimen transmission.

Each of the 43 linear position-sensitive detectors is serviced by two charge-sensitive preamplifiers, and each counter in the array is serviced by its own dual-amplifier–dual-ADC electronic module. These modules reside in the memory/address space of an M6809 microcomputer so that the detector elements become part of the computer memory. The ADC's digitize the pulses from the amplifiers for any neutron event whose pulse amplitude exceeds the lower level discriminator (LLD) setting, and the binary pulse amplitudes are processed by the M6809 microcomputer to encode the event positions. Upon the receipt of a valid neutron event, the detector tube is disabled until the data are read and its ADC's cleared by the microcomputer. Provision has also been made for the detector to be controlled by an external signal, permitting the instrument to be used for dynamic or 'time slicing' type experiments.

The microcomputer uses the charge division method to calculate the neutron event position from the digitized signals. If $Q_A$ and $Q_B$ are the charges collected at each end of the detector, $A$ and $B$, $l$, the anode wire length, $\rho$, the anode wire resistivity, $x$, the distance between the neutron event and the detector end $A$, and $Z$, the input impedance of each preamplifier, then the position of the event may be determined from the relationship

$$\frac{Q_A}{Q_A + Q_B} = \frac{x\rho + Z}{\rho \rho + 2Z}.$$  (1)

With this technique, greatest sensitivity is obtained when the input impedance, $Z$, of the preamplifiers is small compared to the anode-wire resistance, $\rho \rho$, which is $3900\Omega$ for each linear detector. The variation of the positional sensitivity for different impedances has been analyzed elsewhere (Berliner, King & Mildner, 1978).

The detector array is designed to operate at event throughputs of 10 to 20 K s$^{-1}$. Since the calculation of the neutron event position from (1) is performed...
digitally within the M6809, the digital logic design must accommodate event processing times of 50 to 100 \( \mu \text{s} \) per event. These speeds rule out performing the division by software (\( \sim 1.5 \text{ ms per event} \)), or by hardware floating-point arithmetic (150 to 200 \( \mu \text{s} \) per event). Instead, the quotient \( Q_s/(Q_a + Q_b) \) is formed by multiplication. The sum \( (Q_a + Q_b) \) is used to address a microcomputer memory location which contains the previously computed inverse value \( (Q_a + Q_b)^{-1} \). The digital representation of \( Q_b \) and the value of \( (Q_a + Q_b)^{-1} \) obtained from the look-up table is passed to a hardware multiplier.

In order to improve event processing speed, the detector array is extensively buffered. The design (Berliner, Mildner, Pringle & King, 1981) allows the detector electronics, the microcomputer and the PDP 11/03 to operate independently. It also liberates the PDP 11/03 – which contains the memory image of the detector data – allowing it to be used for data analysis while data collection is proceeding. The PDP 11/03 can transmit its data to a PDP 11/40 for storage and complex data analysis.

### 3. Instrument calibration

#### 3.1. Intensity

The most critical feature of any SANS spectrometer is the magnitude of the product of the source intensity at the sample and the sample area, for a given resolution. The intensity at the sample can be estimated from the Maxwellian source distribution and the spectrometer geometry. The total number of neutrons incident on the sample per second is given approximately by

\[
I = \frac{d^2\phi}{d\omega^2 L_1^2} A_1 A_2, \tag{2}
\]

where the double differential is the flux of neutrons per wavelength per steradian at the effective source plane (the beryllium reflector which surrounds the reactor core), \( A_2 \) is the wavelength spread transmitted by the monochromator, \( A_1 \) is the source aperture, \( A_2 \) is the sample aperture and \( L_1 \) is the distance between the apertures.

It is useful to express the intensity as a function of the Maxwellian distribution by

\[
I = \frac{\phi_0}{4\pi 2} \left( \frac{E}{k_B T} \right)^2 \exp \left( -\frac{E}{k_B T} \right) A_1 A_2 L_1^2 \frac{f_\parallel}{f_\perp} R_0 \beta_\parallel, \tag{3}
\]

where \( \phi_0 \) is the integral of the Maxwellian over all energies \( E, T \) is the effective temperature of the reactor, \( k_B \) is the Boltzmann constant and \( R_0 \) is the maximum reflectivity of the graphite crystals at a wavelength of 4.75 \( \text{Å} \). The factors \( f_\parallel \) and \( f_\perp \) are divergence factors relating the primary beam and mosaic orientation angles in planes parallel and perpendicular to the scattering plane and are given by

\[
f_\parallel = \left( 1 + \frac{\beta_\parallel}{\beta_\parallel} \right)^{-1/2}
\]

and

\[
f_\perp = \left( 1 + \frac{2 \beta_\perp^2}{\beta_\parallel^2} \right)^{-1/2}, \tag{4}
\]

where \( \beta_\parallel \) and \( \beta_\perp \) are the divergences of the primary collimation in the directions in and perpendicular to the scattering plane. The quantities \( \beta_\parallel \) and \( \beta_\perp \) are the rocking-curve widths of the composite crystal in directions in and perpendicular to the scattering plane. These divergence factors are closest to unity when the effective crystal mosaic \( \beta_\parallel \) is largest and \( \beta_\perp \) is smallest. Hence it is best to have many crystals of narrow mosaic, slightly misoriented relative to each other in the scattering plane to increase the effective mosaic \( \beta_\parallel \) in that direction. This increases the reflectivity over the full angular divergence of the wavelength spread \( \Delta \lambda \). The derivation of (3) and estimates of the intensity are found in Appendix A.

We have taken intensity measurements at the sample position by placing an end window BF\(_3\) detector immediately over the upper aperture. The neutron count rate at the sample position depends on the resolution defined by the incident beam. Without a statement regarding the resolution, an intensity number is meaningless. This may be stated with the minimum scattering vector

\[
\vec{Q} = \frac{2\pi}{L_1} (R_1 + 2R_2), \tag{5}
\]

defined by the radii, \( R_1 \) and \( R_2 \), of the source and sample apertures, and the distance \( L_1 \) between them. Thus, the intensity is proportional to \( \vec{Q}^2 \) for a matched stem. We have found that at the 4.5 m position with a \( \vec{Q} = 5.9 \times 10^{-3} \text{ Å}^{-1} \), the intensity is \( 2.5 \times 10^8 \text{ m}^{-2} \text{ s}^{-1} \). This value should be compared to the theoretical intensity of \( 4.2 \times 10^8 \) neutrons \text{ m}^{-2} \text{ s}^{-1} \) for \( \vec{Q} = 0.006 \text{ Å}^{-1} \) derived in Appendix A. The difference is most probably due to losses occurring in the 1 m section containing the liquid-nitrogen-cooled beryllium filter (see Fig. 1). Values of count rate and intensity at the sample position for other

### Table 1. Values of the neutron count rate and neutron intensity at the sample for different instrumental configurations

<table>
<thead>
<tr>
<th>Incident length ( L (\text{m}) )</th>
<th>First aperture radius ( R_1 (\text{mm}) )</th>
<th>Second aperture radius ( R_2 (\text{mm}) )</th>
<th>Minimum scattering vector ( \vec{Q} (\text{Å}^{-1}) \times 10^{-3} )</th>
<th>Neutron count rate ( \times 10^3 )</th>
<th>Neutron intensity ( \text{m}^{-2} \text{ s}^{-1} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.0</td>
<td>5</td>
<td>2.5</td>
<td>4.41</td>
<td>2.81</td>
<td>1.43 \times 10^6</td>
</tr>
<tr>
<td>3.0</td>
<td>10</td>
<td>5</td>
<td>8.82</td>
<td>4.49 \times 10^4</td>
<td>5.72 \times 10^6</td>
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<tr>
<td>4.5</td>
<td>5</td>
<td>2.5</td>
<td>2.94</td>
<td>1.25 \times 10^4</td>
<td>6.4 \times 10^6</td>
</tr>
<tr>
<td>4.5</td>
<td>10</td>
<td>5</td>
<td>5.88</td>
<td>2.00 \times 10^4</td>
<td>2.54 \times 10^6</td>
</tr>
</tbody>
</table>
values of $\bar{Q}$ are given in Table 1. These values are averages of a number of measurements.

### 3.2. Resolution

There are three components to the resolution function for the spectrometer. These contributions arise from: (1) the spread in wavelength delivered to the instrument by the monochromator; (2) the finite sizes of the beam-defining apertures and of the elements of the multidetector; and (3) the digitization effects of the linear position-sensitive detectors. The scattering vector $Q$ associated with the diffraction of a beam of neutrons of wavelength $\lambda$ scattered through an angle $\theta$ is given by $Q = \frac{4\pi}{\lambda} \sin(\theta/2)$ for the small angles used with this instrument ($\theta < 4^\circ$), may be reduced to

$$Q = \frac{2\pi}{\lambda} \theta.$$  \hspace{1cm} (6)

The dependence of the width $AQ$ of the resolution function on the wavelength spread $\Delta\lambda$ and on the angular uncertainty $\Delta\theta$ is given by

$$(AQ/Q)^2 = (\Delta\lambda/\lambda)^2 + (\Delta\theta/\theta)^2. \hspace{1cm} (7)$$

These two terms are considered in turn, and the third component will be considered separately.

#### 3.2.1. Wavelength resolution.

The orientation of the graphite crystals in the cassette is such that they view the full beam-port height at the beryllium reflector in order to maximize the intensity delivered to the sample position. The spectrometer wavelength $\lambda$ is defined by the spacing $d$ of the pyrolytic graphite (002) planes, and the angle $2\theta_B$ through which the neutrons are Bragg reflected. The spread in wavelength $\Delta\lambda$ depends on the range $\Delta\theta_B$ of orientation of the crystals; that is, the full width $\beta_B$ at half maximum of the rocking curve of the crystals in the scattering plane. Hence, the wavelength resolution is given by

$$\Delta\lambda/\lambda = \cot\theta_B \Delta\theta_B = \beta_B,$$  \hspace{1cm} (8)

so that the beam is diffracted through $90^\circ$ so that $\theta_B = \pi/4$ and $\cot\theta_B = 1$. The cassette views the full source height of 136 mm at a distance of 3.3 m from the beryllium reflector so that the primary collimation of $x_B = 0.041$ rad in the scattering plane defines the wavelength spread. The wavelength resolution of the instrument and its contribution to the overall resolution is 4.1\%.

That is, the contribution $AQ_s$ to the width of a peak at a scattering vector $Q$ is linearly dependent on the value of $Q$; it is negligible at the smallest values of $Q$ but is dominant at the highest scattering vectors. This is a penalty paid for attempting to increase the beam intensity of the spectrometer which varies linearly with $x_B$ or $\Delta\lambda$ (see equation 3).

#### 3.2.2. Angular resolution.

The geometric angular contribution to the overall resolution is more complex and depends on the incident and scattered flight path lengths. Optimization of the intensity for a given resolution requires the incident and scattered flight paths to be equal. Optimization of the resolution requires that the various angular contributions to the width of the scattering vector have the same magnitude.

The source and sample apertures are circular whereas the detector elements are rectangular. Combining optimization results for radial and Cartesian geometries (Mildner, 1978), we obtain the resolution

$$\Delta Q_O^2 = \frac{k^2}{12} \left[ 6 \left( \frac{R_1}{L_1} \right)^2 + 6 R_2^2 \left( \frac{1}{L_1} + \frac{1}{L_2} \right)^2 \right] \left( \frac{\Delta X}{2} + \frac{\Delta Y}{2} \right)^2 + \frac{(\Delta X)^2 + (\Delta Y)^2}{L_2^2}. \hspace{1cm} (9)$$

where $L_1$ and $L_2$ are the incident and scattered flight path lengths, $\Delta X$ and $\Delta Y$ are the detector element sizes in directions along and perpendicular to the detector axes and $k$ is the neutron wave vector. Separating this resolution into two orthogonal components gives, with equal flight paths ($L_1 = L_2 = L$),

$$\Delta Q_{O_i}^2 = \frac{k^2}{2L} \left[ R_1^2 + 4 R_2^2 + \frac{1}{3} \left( \frac{\Delta X}{\Delta Y} \right)^2 \right]^{1/2}. \hspace{1cm} (10)$$

The geometric resolution depends on the flight path length $L$ and the radii of the two beam-defining apertures, all of which are discretely variable, and the detector element size which is fixed. The contribution $AQ_o$ to the width of a peak is independent of scattering vector $Q$ and is the major contribution at the smallest scattering vectors. Typical values of the geometric resolution of the instrument and their contributions to the overall resolution are given in Table 2. Though the three terms in (10) cannot be made equal since the detector element size is fixed, it is best approximated by values of $R_1 = 10$ mm and $R_2 = 5$ mm, which were used for one of the detector design goals (Berliner, Mildner, Pringle & King, 1981).

#### 3.2.3. Digital resolution.

An additional source of resolution broadening is created by the digital processing of the charge division position encoding algorithm. A neutron incident at a given position along one of the detector tubes creates an average charge cloud diameter

<table>
<thead>
<tr>
<th>$L$ (m)</th>
<th>$R_1$ (mm)</th>
<th>$R_2$ (mm)</th>
<th>$\Delta X$ (mm)</th>
<th>$\Delta Y$ (mm)</th>
<th>$AQ_s$ ($10^{-3}$ Å$^{-1}$)</th>
<th>$AQ_o$ ($10^{-3}$ Å$^{-1}$)</th>
<th>$AQ$ ($10^{-3}$ Å$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.0</td>
<td>5</td>
<td>2.5</td>
<td>5</td>
<td>12.7</td>
<td>1.68</td>
<td>2.25</td>
<td>2.81</td>
</tr>
<tr>
<td>3.0</td>
<td>10</td>
<td>5</td>
<td>5</td>
<td>12.7</td>
<td>3.48</td>
<td>3.51</td>
<td>4.74</td>
</tr>
<tr>
<td>4.5</td>
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<td>2.5</td>
<td>5</td>
<td>12.7</td>
<td>1.12</td>
<td>1.50</td>
<td>1.87</td>
</tr>
<tr>
<td>4.5</td>
<td>10</td>
<td>5</td>
<td>5</td>
<td>12.7</td>
<td>2.12</td>
<td>2.34</td>
<td>3.16</td>
</tr>
</tbody>
</table>

Table 2. Typical values of the geometric resolution of the instrument and the contributions to the overall resolution

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of 2.5 mm about that position. A consequence of the breadth of the neutron pulse-height spectrum is that the digital charge division algorithm may map the encoded event position into one of three adjacent detector position channels depending on the amplitude of the sum \((Q_a + Q_b)\) pulse, the preamplifier input impedance and the neutron event position along the detector element. The full implications of these digitization errors introduced into positional encoding for linear detectors are discussed more fully elsewhere (Berliner, Mildner & Pringle, 1981).

The method we have used for analyzing the digitization broadening is by computer simulation. A large number of neutrons within a typically measured pulse-height spectrum are introduced at a large number of discrete points along a Debye–Scherrer cone at the detector for a given scattering vector \(Q\). The pulse-height spectrum is digitized in exactly the same way as that done by the detector electronics. For each element in the pulse-height spectrum, a detector position cell is calculated and stored in the memory of the computer using the same algorithm as in an experiment. The scattering angle \(\theta\) is defined by the ratio of the radial distance \(D\) of the neutron event from the beam center at the detector to the distance \(L\) of the detector from the sample; i.e. \(\theta = D/L\). If \(\Delta D\) is a measure of the spread of the delta-function data caused by digitization broadening, then the spread in scattering vector \(\Delta Q\) is given by:

\[
\Delta Q = \frac{2\pi \Delta D}{\lambda}.
\]

(11)

The simulated spectrum is radially averaged and a width \(\Delta Q\) determined. The results of the simulations for various values of scattering vector show that there are no gross systematic variations of \(\Delta D\) with radial distance, so that the results may be taken as typical. The average value of \(\Delta D\) of \(~10\) mm is to be compared with the resolution of each detector element of \(\Delta Y \sim 5\) mm and \(\Delta X \sim 12.7\) mm, or \((\Delta X^2 + \Delta Y^2)^{1/2} \sim 13.6\) mm. It is found that, for the 4.5 m position, \(\Delta Q \sim 0.003\) \(\text{Å}^{-1}\) and, for the 3 m position, \(\Delta Q \sim 0.0045\) \(\text{Å}^{-1}\).

The values of the three contributions to the instrumental resolution as a function of scattering vector are shown in Fig. 4 for the two different flight path lengths and two different sets of apertures. These results have also been measured experimentally with a Xenopus laevis yolk lipoprotein in heavy water (Banaszak, Wrenn & Meininger, 1980) and are shown in Fig. 5. The data were obtained with the multidetector at the 4.5 m position in 15 h with \(R_1 = 5\) mm and \(R_2 = 2.5\) mm, and radially averaged with scattering vector bins of size \(\Delta Q \sim 0.0015\) \(\text{Å}^{-1}\). The data show Bragg reflections which correspond well with X-ray reflections obtained from wet, native polycrystalline samples (Ohlendorf, Collins, Puronen, Banaszak & Harrison, 1975). The width of the prominent peak at a scattering vector \(Q \sim 0.0785\) \(\text{Å}^{-1}\) (corresponding to \(d = 2\pi/Q = 80\) Å, the spacing of the orthorhombic cell 021 reflection which is also most marked in the SAXS data) is about \(\Delta Q \sim 0.0055\) \(\text{Å}^{-1}\). The wavelength contribution \(\Delta Q\) to the width is \(\sim 0.0039\) \(\text{Å}^{-1}\), and for the two apertures used, the angular contribution \(\Delta Q\) is \(\sim 0.0019\) \(\text{Å}^{-1}\), and the calculated digital contribution \(\Delta Q\) is 0.0031 \(\text{Å}^{-1}\). These contributions add in quadrature to give a total width \(\Delta Q\) of \(\sim 0.0053\) \(\text{Å}^{-1}\) which is in good agreement with the measured peak width.

3.3. Background

The inside of the aluminum detector box is lined with 0.5 mm cadmium, and only the flight tube below is open to the detectors. The outside of the detector box is covered by a 50 mm layer of boron carbide on top and a 38 mm layer to the sides; the thickness varies because of the complicated geometry of the detector box inside the tower. Further shielding at the detector level outside the tower cuts down on the large contribution to

![Fig. 4. The values of the three contributions (wavelength \(\lambda\), angular \(\theta\), and digital \(D\)) to the instrumental resolution as a function of scattering vector for different spectrometer configurations (two different flight path lengths, and two different sets of apertures). (a) \(R_1 = 5\) mm, \(R_2 = 2.5\) mm; (b) \(R_1 = 10\) mm, \(R_2 = 5\) mm.](image1)

![Fig. 5. The small-angle neutron scattering from a solution of Xenopus laevis yolk lipoprotein in heavy water (prepared by Banaszak, Wrenn & Meininger, 1980).](image2)
background from the area immediately adjacent to the ends of the detectors. Another 25 mm layer of boron carbide wraps around the outside of the flight tube and its extensions.

Fig. 6 shows that the background level caused by the residual tail of the pulse-height spectrum from γ-rays above a hardware lower level discriminator (LLD) setting, can be improved by raising the software LLD. This reduction occurs at a small expense of neutron count rate, so that the signal-to-noise ratio can be improved. The instrument is currently operating with a software LLD setting of 110 and a background of less than 0.001 counts (resolution element) $^{-1}$ s$^{-1}$. That is, with the reactor operating at a power level of 10 MW, the background count rate is 15 counts m$^{-2}$ s$^{-1}$, whereas with the reactor shut down the count rate is less than 0.5 m$^{-2}$ s$^{-1}$.

4. Applications

In general, small-angle scattering measurements require the system under study to be characterized also by other techniques. Consequently, the experimental program for MURR–SANS is performed in collaboration with many scientific groups, both in universities and industrial and government laboratories. We have started measurements of a number of systems in the fields of biological, polymeric and materials sciences. An example of a biological macromolecule has been given in § 3.2.

Comparison measurements have been made on samples of polyethylene and polystyrene studied earlier at ILL, Grenoble. Fig. 7 shows the data taken on a sample of 100% deuterated polyethylene of low molecular weight which has been studied previously by both SANS and SAXS (King et al., 1979) to examine differences in concentration and density inhomogeneities. Also shown are the data taken on the D11A instrument at Grenoble. These data have been arbitrarily normalized at $Q=0.025$ Å$^{-1}$. It can be seen that the MURR–SANS data follow closely the data taken on the D11A over the range of scattering vectors measured, including the shoulder in the data around $Q \sim 0.01$ Å$^{-1}$.

It has been demonstrated that it is possible to extract the single-chain structure factor from experimental data taken on highly concentrated deuterated polymer samples (Summerfield, King & Ullman, 1978). This is a

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**Fig. 6.** The background γ and detected neutron count rates, and the signal-to-noise ratio as a function of lower level discriminator (LLD) setting, relative to measurements at an LLD setting of 110 channels.

**Fig. 7.** A comparison of small-angle neutron scattering data for a sample of deuterated polyethylene on the D11A instrument at ILL, Grenoble and on the MURR–SANS spectrometer. These data are normalized at $Q=0.025$ Å$^{-1}$, and the curve is a line through the MURR data.

**Fig. 8.** The transmission-corrected data for four samples of polystyrene with weight fractions of deuterated polymer of 1.0, 0.7, 0.3 and 0.0. The spectrometer geometry is $R_1 = 10$ mm, $R_2 = 5$ mm, $\Delta X = 5$ mm, $\Delta Y = 12.7$ mm and $L_1 = L_2 = 4.5$ m.
consequence of the different dependence on concentration for the coefficients of the self and total scattering terms. The precision of the method has been demonstrated by reproducing the radius of gyration results of low-concentration deuterated bulk atactic polystyrene (Tangari, Summerfield, King, Berliner & Mildner, 1980). The transmission-corrected data from four samples with weight fractions of deuterated polymer of 1.0, 0.7, 0.3 and 0.0 are shown in Fig. 8. Each set of data was taken in a 3 h run and radially averaged. This demonstrates the ability of MURR–SANS for determining low-Q data for medium-weight polymer samples.

The data from small-angle neutron scattering measurements performed on pure, hydrogenated and deuterated samples of amorphous silicon (Postol, Falco, Kampwirth, Schuller & Yelon, 1980) indicate structures in the sample of average radius of gyration of approximately 270 Å. Differences between the data for the hydrogenated and deuterated samples are consistent with the existence of voids and/or large areas of grain boundaries on which approximately half the H or D reside. The scattering from the voids surrounded by SiD is found to be less intense than from similar voids in the SiH. This is explained by assuming that large surface areas within the sample are covered with hydrogen (deuterium), so that there is a larger fluctuation in the scattering density for the hydrogenated case.

5. Conclusions

We have constructed a small-angle neutron scattering spectrometer on the 10 MW research reactor at the University of Missouri. This spectrometer, designed to be competitive with all but the most powerful SANS instruments and yet maintain a reasonable cost, is distinguished by two unique features. One is its vertical geometry which makes some sample handling considerations easier, and the other is its multidetector which is composed of a large array of linear position-sensitive detectors. The MURR–SANS spectrometer uses incident neutrons of wavelength 4.75 Å to give a flux of about 10^8 neutrons m^-2 s^-1 on the sample, depending on the instrument configuration and resolution. A range of scattering vectors from 0.005 to 0.15 Å^-1 may be measured. These figures show that the instrument compares favorably with other SANS spectrometers. Preliminary results show that this instrument is suitable for a wide variety of experiments where extremely high flux or resolution are not required. MURR–SANS is designed as a user-oriented facility and forms part of a neutron scattering center.

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APPENDIX A

Calculation of beam intensity

Consider the incident flux \( \varphi(E) \) whose spectral shape may be described by a Maxwellian

\[
\frac{d^2 \varphi}{dE d\Omega} = \frac{\varphi_0}{4\pi (k_B T)^2} \exp\left(-\frac{E}{k_B T}\right),
\]

where \( E \) is the neutron energy, \( T \) the effective temperature of reactor flux, \( k_B \) the Boltzmann constant, \( \Omega \) the solid angle and \( \varphi_0 \) a normalizing constant. Converting this to a flux per unit volume in \( k \) space, we obtain

\[
\frac{d^3 \varphi}{dk^3} = \frac{\varphi_0}{4\pi k_0^3} \frac{2}{(k_B T)^2} \exp\left(-\frac{E}{k_B T}\right),
\]

where \( k_0 \) is the incident wave number. We wish to consider the diffraction of neutrons by the graphite crystals from a volume \( A \) of the primary beam in \( k \) space through an angle \( 2\theta_0 \) to a volume \( A' \) of the scattered beam (see Fig. 9), and analyze the effects of the crystal mosaic and collimation on the flux incident on the sample.

Let \( x \) be the direction of the primary beam and \( z \) be the direction of the scattered beam, with \( y \) the third axis perpendicular to the scattering plane defined by the vectors \( x \) and \( z \). Let \( x_0 \) and \( z_0 \) be the divergences of the primary collimation in directions in and perpendicular

\[
\text{Fig. 9. A schematic drawing of the diffraction of neutrons by the graphite crystals from a volume } A \text{ of the primary beam in } k \text{ space through an angle } 2\theta_0 \text{ to a volume } A' \text{ of the scattered beam.}
\]
to the scattering plane. If we assume that the angular distribution of neutrons from the source plane may be approximated by a two-dimensional Gaussian, then the widths of volume A of the primary beam in \( k \) space are given by

\[
\sqrt{2 \pi W_{\parallel}} = \alpha_{\parallel} k_0 / \sin \theta_B
\]

and

\[
\sqrt{2 \pi W_{\perp}} = \alpha_{\perp} k_0,
\]

where \( \theta_B \) is the Bragg angle. The transmission function of the primary collimation for an off-axis neutron of wavevector \( k(1, \alpha_{\parallel}, \alpha_{\perp}) \) is given by

\[
T_0(\alpha_{\parallel}, \alpha_{\perp}) = \exp \left( -\frac{\Delta \alpha_{\parallel}^2}{2W_{\parallel}^2} \right) \exp \left( -\frac{\Delta \alpha_{\perp}^2}{2W_{\perp}^2} \right),
\]

where \( \Delta \alpha_{\parallel} \) and \( \Delta \alpha_{\perp} \) are components of the angle the incident ray makes with the central ray in and perpendicular to the scattering plane. Similarly, the transmission function of the secondary collimation for an off-axis neutron of wavevector \( k(Ax, Ay, 1) \) is given by

\[
T_1(Ax, Ay) = \exp \left( -\frac{\Delta x^2}{2W_{\parallel}^2} \right) \exp \left( -\frac{\Delta y^2}{2W_{\perp}^2} \right),
\]

where \( \Delta x \) and \( \Delta y \) are the components of the angle the scattered ray makes with the central ray in and perpendicular to the scattering plane. \( W_{\parallel} \) and \( W_{\perp} \) are the Gaussian widths of the scattered beam caused by the secondary collimation alone.

The contributions to the widths of volume \( A' \) of the scattered beam in \( k \) space caused by the effective mosaic spreads of the composite crystal in and perpendicular to the scattering plane are given by

\[
\sqrt{2 \pi W'_{\parallel}} = \beta_{\parallel} k_0 \cos \theta_B / \sin \theta_B
\]

and

\[
\sqrt{2 \pi W'_{\perp}} = \beta_{\perp} G = \beta_{\perp} 2k_0 \sin \theta_B
\]

where \( G \) is the reciprocal-lattice vector for the 002 reflection of pyrolytic graphite, and \( \beta_{\parallel} \) and \( \beta_{\perp} \) are the full widths at half maximum of the rocking curves of the composite crystal in the two orthogonal directions. The reflectivity of the crystal monochromator as a function of mosaic orientation angles \( \Delta \beta_{\parallel} \) and \( \Delta \beta_{\perp} \) is given by

\[
R(\Delta \beta_{\parallel}, \Delta \beta_{\perp}) = \frac{R_0}{\sqrt{2 \pi W'_{\perp}}} \exp \left( -\frac{\Delta \beta_{\parallel}^2}{2W_{\parallel}^2} \right) \times \exp \left( -\frac{\Delta \beta_{\perp}^2}{2W_{\perp}^2} \right),
\]

where \( R_0 \) is the maximum reflectivity. The value of \( R_0 \) is given in Appendix B and is shown to increase with the use of crystals with narrow mosaic.

We need to express the angles \( Ax, Ay \) and \( Az \) (the angle the incident ray makes with the central beam in the \( z \) direction) in terms of the primary beam and mosaic orientation angles; that is,

\[
\Delta x = \Delta \alpha_{\parallel} \cos \theta_B + \Delta \beta_{\parallel}
\]

and

\[
\Delta y = \Delta \alpha_{\perp} + \Delta \beta_{\perp}
\]

so that

\[
\Delta z = \Delta \alpha_{\parallel} \sin \theta_B,
\]

and

\[
\Delta \beta_{\parallel} = \Delta x - \Delta z \cot \theta_B.
\]

The neutron flux leaving the crystal is given by

\[
\phi_1(k) = \frac{d^3 \varphi}{dk^3} \int R(\Delta \beta_{\parallel}, \Delta \beta_{\perp}) T_0(\Delta \alpha_{\parallel}, \Delta \alpha_{\perp}) d(\Delta \alpha_{\perp}).
\]

Define a divergence factor \( f_\perp \) relating the primary beam and mosaic orientation angles in the plane perpendicular to the scattering plane by

\[
f_\perp = \left( 1 + \frac{W_{\parallel}^2}{W_{\perp}^2} \right)^{-1/2} = \left[ 1 + (2\beta_{\perp} \sin \theta_B / \alpha_{\perp})^2 \right]^{-1/2},
\]

which for \( \theta_B = \pi/4 \) becomes

\[
f_\perp = (1 + 2\beta_{\perp}^2 / \alpha_{\perp}^2)^{-1/2}.
\]

Then (A10) may be evaluated to give

\[
\phi_1(k) = \frac{d^3 \varphi}{dk^3} R_0 f_\perp \int \exp \left( -\frac{\Delta \beta_{\parallel}^2}{2W_{\parallel}^2} \right) \times \exp \left( -\frac{\Delta \alpha_{\parallel}^2}{2W_{\parallel}^2} \right) \exp \left( -\frac{\Delta \alpha_{\perp}^2}{2W_{\perp}^2} \right),
\]

where

\[
W_{\parallel}^2 = W_{\alpha_{\parallel}}^2 + W_{\beta_{\parallel}}^2.
\]

The flux incident on the sample is given by

\[
\phi = \int \int \int \varphi_1(k) T_1(Ax, Ay) d(Ax) d(Ay) d(Az) = \frac{d^3 \varphi}{dk^3} R_0 f_\perp \int \int \int \exp \left[ -\frac{(Ax - Az \cot \theta_B)^2}{2W_{\parallel}^2} \right]
\]

\[
\exp \left[ -\frac{(Ax - Az \cot \theta_B)^2}{2W_{\parallel}^2} \right] \exp \left[ -\frac{(Ax - Az \cot \theta_B)^2}{2W_{\parallel}^2} \right]
\]

\[
\times \exp \left[ -\frac{(Ax - Az \cot \theta_B)^2}{2W_{\parallel}^2} \right] d(Ax) d(Ay) d(Az).
\]

It is reasonable to assume that the contribution to the Gaussian width of the scattered beam caused by the scattering collimation is small compared to that from the primary collimation and the mosaic spread; that is,

\[
W_{\alpha_{\parallel}} \leq W_{\parallel} \text{ and } W_{\alpha_{\perp}} \leq W_{\perp}.
\]

Define a divergence factor \( f_\parallel \) relating the primary beam and mosaic orientation angles in the plane parallel to
the scattering plane by
\[ f_\parallel = (1 + W_{\parallel}^2/W_{\perp}^2 \cos^2 \theta_B)^{-1/2} = (1 + \beta_{\parallel}^2/\alpha_{\parallel}^2)^{-1/2}. \]  

Then (A14) may be evaluated to give
\[ \varphi = \frac{d^3 \varphi}{d^2 R_0 f_1 f_2 (2\pi)^{3/2}} W_{\alpha\perp} W_{\beta\parallel} \tan \theta_B. \]  

Now consider the experimental configuration of MURR-SANS (see Fig. 1). If \( L_1 \) is the distance between the lower aperture of area \( A_1 \) and the upper aperture which defines the sample area \( A_2 \), then the secondary collimation is given by
\[ \sqrt{2\pi} W_{\beta\parallel} \beta_{\parallel} k_0 \cot \theta_B. \]  

Then the neutron flux at the sample position defined by the upper aperture is given by
\[ \varphi = \frac{\phi_0}{4\pi} \left( \frac{E}{k_B T} \right)^2 \exp \left( - \frac{E}{k_B T} \right) \frac{A_1}{L_1} f_1 f_2 R_0 \beta_{\parallel}. \]  

The product \( f_1 R_0 \beta_{\parallel} = (0.021) \) of the parallel divergence factor, the peak reflectivity and the effective mosaic width of the composite crystal in the scattering plane defines a figure-of-merit for the instrument. It is equivalent to the product of the wavelength resolution \( \Delta\lambda/\lambda \) of a spectrometer with a velocity selector and its transmission factor.

Notice that the product of the divergence factor \( f_1 \) and the effective mosaic width \( \beta_{\parallel} \) of the composite crystal in the scattering plane can be expressed differently by
\[ f_1 \beta_{\parallel} = f_\parallel \alpha_{\parallel}, \]  

where
\[ f_\parallel = (1 + \alpha_{\parallel}^2/\beta_{\parallel}^2)^{-1/2}. \]  

Then the expression for the neutron intensity at the sample position becomes
\[ I = \frac{\phi_0}{4\pi} \left( \frac{E}{k_B T} \right)^2 \exp \left( - \frac{E}{k_B T} \right) \frac{A_1 A_2}{L_1^2} f_\parallel f_1 R_0 \alpha_{\parallel}. \]  

This form of the intensity equation shows the dependence on the primary collimation in the scattering plane. The value of \( \alpha_{\parallel} \) should be taken as that averaged across the circular source; that is
\[ \alpha_{\parallel} = \frac{\pi D}{4 L_0} \approx 1.86^\circ, \]  

where \( D (= 136 \text{ mm}) \) is the inside diameter of the beam port, and \( L_0 (= 3.3 \text{ m}) \) is the distance from the effective source plane of the beam port to the graphite crystals. Similarly, the value of \( \alpha_{\perp} \) should be taken as its average value of \( 1.86^\circ \), rather than the maximum value of \( 2.37^\circ \). The two divergence factors, \( f_\parallel \) and \( f_\perp \), show explicitly the dependence of the neutron intensity on the crystal mosaic spread, provided that the thickness of the crystal is sufficiently large that the peak reflectivity is close to unity. Fig. 10 shows the dependence of \( f_\parallel \) and \( f_\perp \) on the crystal mosaic spread for a given primary collimation \( \alpha = 1.86^\circ \). These curves show that these divergence factors are closest to unity when \( \beta_{\parallel} \) is as large as possible and \( \beta_{\perp} \) is as small as possible. This can be accommodated by having many crystals of narrow mosaic, placed back to back but slightly misoriented relative to each other, so that the effective mosaic spread in the scattering plane is made large. In this way, the full height of the beam port is utilized without spreading the beam too much in a direction perpendicular to the scattering plane.

We now evaluate the neutron intensity at the sample position by taking all the parameters in (A22) as their full widths at half maximum. The rocking-curve widths, \( \beta_{\parallel} \) and \( \beta_{\perp} \), of the composite crystal are 1.93 and 1.01° respectively. The divergence factors, \( f_\parallel \) and \( f_\perp \), defined by (A21) and (A12), are 0.720 and 0.793, where the primary collimations, \( \alpha_{\parallel} \) and \( \alpha_{\perp} \), are 1.86°. The reflectivity \( R_0 \) at a wavelength of 4.75 A is given in Appendix B as 0.847. For a neutron energy \( E = 3.62 \text{ meV} \), and \( k_B T = 28 \text{ meV} \), \( E/k_B T = 0.1296 \) and \( \exp(-E/k_B T) = 0.878 \). We take the commonly used configuration of the lower aperture radius \( R_1 = 10 \text{ mm}, \)
and the upper aperture radius $R_2 = 5$ mm, placed a distance $L_1 = 4.5$ m apart, so that $A_1 = 100 \pi \text{mm}^2$ and $A_2 = 100 \pi /4 \text{mm}^2$, and the minimum scattering vector defined by the two apertures is $Q = 0.006 \text{Å}^{-1}$ by (5).

The beam port has a nominal source flux of $\varphi_0 = 1 \times 10^{18}$ neutrons m$^{-2}$ s$^{-1}$. Beryllium cooled to liquid-nitrogen temperature has a neutron cross section of about $4.7 \times 10^{-30}$ m$^2$ at a wavelength of $4.75$ Å, so that the transmission through a $150$ mm filter is 0.916. Substituting all these factors into (A22), we obtain for the neutron flux at the sample position a value of $\varphi = 4.2 \times 10^8$ neutrons m$^{-2}$ s$^{-1}$, for $\varphi_0 = 0.006 \text{Å}^{-1}$.

Integrating the product of the flux per unit volume in $k$ space (given by equation A2) and the transmission function of the primary collimation (given by equation A4) over the two primary collimation angles and over the wavevector gives a measure of the flux at the monochromator assembly. That is,

$$\int \frac{d^3 \varphi}{dk^3} T_o(\Delta x_{\parallel}, \Delta x_{\perp})d(\Delta x_{\parallel})d(\Delta x_{\perp})dk = \frac{\varphi_0}{4\pi} \alpha_{\parallel} \alpha_{\perp},$$

(A25)

where $\alpha_1$ is taken as its maximum value. We calculate a value of $1.1 \times 10^{14}$ neutrons m$^{-2}$ s$^{-1}$ for the neutron flux at the monochromating crystals.

**APPENDIX B**

**Measurement of composite crystal mosaic**

The mosaic spreads of the composite crystal in the two directions were measured on a diffractometer at a wavelength of $4.07$ Å. The experimental arrangement is shown in Fig. 11. A tightly collimated beam of neutrons was incident on the crystals which were set at the Bragg condition. A $38$ mm wide detector composed of $40$ atm ($4.05 \times 10^6$ Pa) $^3$He proportional counters was set at the scattering angle $2\theta_B$ at a distance of $178$ mm from the cassette center. Therefore the detector is black at this wavelength and has no efficiency variations across its width. The geometry ensures that all the reflected intensity is detected as the crystals are rocked around the Bragg angle with the detector fixed. An aperture of $12.7 \times 12.7$ mm was placed at the end of the second $10'$ Soller collimator to define the beam.

The results of the rocking-curve measurements, in both reflection and transmission geometries, are shown in Fig. 12. Measurements of the mosaic widths were made for both the parallel and perpendicular conditions. For the parallel geometry, it can be seen that the three crystals combine to give a broad rocking curve, with a peak reflectivity of $80.8\%$ at a wavelength of $4.07$ Å over a range of $1.2^\circ$, and a full width at half maximum of $1.93^\circ$. The rocking curve in the perpendicular direction is more sharply peaked, and the measurements were performed with a $0.4^\circ$ mosaic silicon (111) crystal. The full width at half maximum was measured to be $1.01^\circ$.

The results of these reflectivity measurements at $4.07$ Å must be converted for a wavelength of $4.75$ Å. The peak reflectivity for a pyrolytic graphite crystal with a mosaic spread of $\eta$ is given by (Riste & Otnes, 1969)

$$R_0 = \frac{1}{\sqrt{2\pi \eta \sin \theta_B}} \left[ 1 + \frac{1}{\sqrt{2\pi} \eta \sin \theta_B} \right]^{-1}, \quad (B1)$$

where $t_0$ is the crystal thickness in the direction of the scattering vector, and the crystallographic quantity $Q$ for graphite is given by

$$Q = \frac{\lambda^3}{\sin 2\theta_B} \times 5.65 \times 10^{-3}. \quad (B2)$$

The mosaic spread $\eta$ is related to $\beta$, the full width at half maximum of the rocking curve, by $\eta = 0.424\beta$. It can be...
seen from \((B1)\) that the value of the maximum reflectivity \(R_0\) is increased by the use of the crystals with a narrow mosaic. Knowing the reflectivity at some wavelength allows the reflectivity at another wavelength to be calculated through \((B1)\), assuming that \(t_0\) is some effective crystal thickness. We find that the reflectivity is 84.7% at a wavelength \(\lambda = 4.75\) Å. The theoretical value of the maximum reflectivity for one 0-8° mosaic crystal is 90.25° from \((B1)\).

The width of the composite crystal in the plane perpendicular to the scattering vector for the SANS instrument is evidently not much greater than that for the individual crystals. This is necessary so that the losses due to the spreading of the beam in that direction are minimized. In order to increase the beam intensity, the crystals should be slightly curved in the direction perpendicular to the scattering plane. In practice, we use three sets of three 12.7 mm wide crystals, with the outer sets displaced by an angle 0.2° from the center set (Fig. 2). This displacement angle is determined by the distance (3.3 m) of the crystal assembly from the source plane.

An increase in the width \(\beta_\parallel\) causes a reduction in the average maximum reflectivity \(R_0\). Consequently there is a compromise between \(R_0\) and the parallel divergence factor, \(f_\parallel\), in the intensity (equation 3) delivered to the sample. Differentiating this expression with respect to \(\beta_\parallel\) gives the following optimization condition for the rocking-curve width

\[
\beta_\parallel^2 = \left[\frac{Q t_0}{2\pi \times 0.424 \sin \theta_\parallel}\right]^2.
\]

This expression assumes that the rocking curve may be described by a Gaussian, which is evidently not true from Fig. 12. We have made the rocking-curve width of the composite crystal in the scattering plane (1.93°) well matched to the primary collimation (1.86°).

**References**

