New instruments at IPNS: POSY II and SAD II

R. K. Crawford, G. P. Felcher, R. Kleb, J. E. Epperson, and P. Thiyagarajan
Argonne National Laboratory
Argonne, Illinois
USA

Introduction

Three new instruments are currently in varying degrees of development/construction at IPNS. One of these, the Glass, Liquid, and Amorphous Materials Diffractometer (GLAD) is the subject of a separate paper[1] in these proceedings and so will not be discussed further here. The other two, a second neutron reflectometer (POSY II) and a second small-angle diffractometer (SAD II), are described briefly below.

POSY II

The polarized neutron reflectometer (POSY) has been operating at IPNS since 1984[2]. Recently, it was shown that by utilizing the large difference in scattering from H and D to measure chemical profiles rather than the magnetic profiles for which POSY was designed, such a neutron reflectometer could measure chemical density profiles with resolution unmatched by other techniques[3]. Users in the polymer science community indicated a real need for such an instrument—they could provide enough problems to saturate the time available at POSY. Typical problems include interdiffusion of two similar polymers; concentration gradients for polymers in solutions both at the surface and at solid boundaries; and the mixing of chemically different polymers, or polymeric chains with a head and a tail, such as block copolymers—all over a length scale (1nm) not easily probed by other techniques.

To help satisfy such a greatly expanded user community and because POSY had been conceived and optimized for magnetic studies and, consequently, is not best suited for polymer work, it was decided to split the neutron beam at beam line C2, which serviced only POSY, for use by both POSY and a new instrument (POSY II) that is dedicated totally to polymer research. Figure 1 shows the arrangement of the two instruments on the two beams brought out through the single bulk-shielding penetration. (Beam penetrations in the IPNS bulk shielding are 30 to 35 cm wide, allowing these two narrow beams to achieve an angular separation of 3.7° within the same penetration.) This construction effort, with half of its cost being financed by IBM, is well along, and POSY II and the rebuilt POSY are scheduled for commissioning in Fall 1988. Table I gives some parameters of the two instruments.

In the new arrangement the original POSY will be restructured, implementing modifications previously tested. The sample will not view the direct beam because this will be doubly reflected by a pair of polarizing supermirrors. These mirrors are made of layers of cobalt and titanium of progressive thickness on an anti-reflecting backing of gadolinium and were prepared by Dr. O. Schärpf at Grenoble on accurately polished borosilicate glass. The pair of mirrors are part of a newly

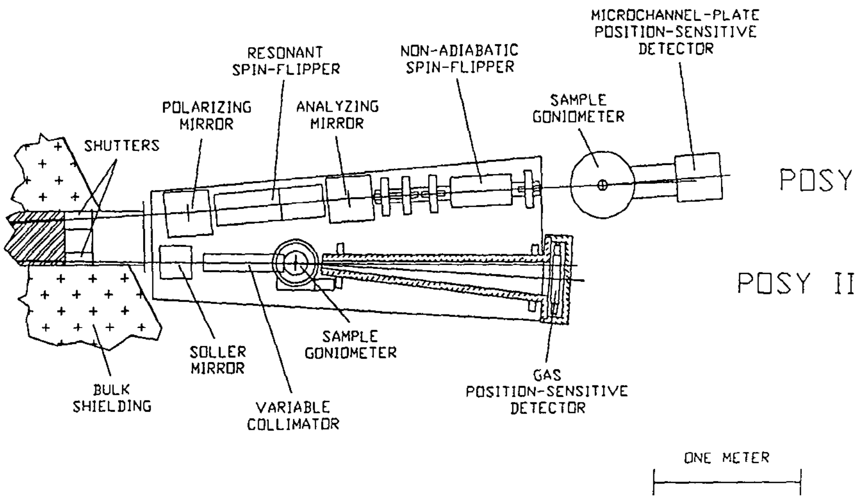

Fig. 1 The reflectometer complex at the IPNS beam line C2. The heavy-duty sample goniometer on POSY can accommodate an electromagnet, a cryostat, or an ultrahigh vacuum chamber, while that on POSY II is a high-precision Huber goniometer to support a temperature-controlled polymer sample cell.

developed "resonant spin flipper" (Fig. 1) to reject neutrons whose wavelengths do not correspond to their nominal time-of-flight, thus sharpening the resolution and improving the background. This device consists of a supermirror spin polarizer, a Drabkin flipper resonantly scanned to flip spins only of the correct wavelength for the current flight time, and a supermirror spin analyzer to reject the unflipped spins[2]. POSY will still use its original microchannel-plate detector. Appropriately placed collimators (not shown in the figure) in the external portion of the incident beam will allow its tailoring to the size of the sample. Samples of magnetic interest are expected to exhibit surfaces of the order of 1 to 2 cm^2.

The design of POSY II reflects the requirements for polymer work. The samples used here have surfaces of several tens of cm^2. The reflectivity will be measured over a wide range of momentum transfers. Because of the increased surface size, slightly relaxed collimation, and absence of polarization analyzers, POSY II should have data rates several times those of POSY.

Table I

	POSY	POSY II
Source-sample distance (cm)	831	623
Sample-detector distance (cm)	60	178
Detector horizontal range (cm)	2.5	20
Detector resolution (mm)	0.5	2
Wave-vector range ($Å^{-1}$)	0.0 - 0.07	0.0 - 0.25
Wave-vector resolution ($Å^{-1}$ FWHM)	~0.003	~0.003
Beam size at moderator (cm^2)	2.6 x 10.3	2.6 x 10.3
Beam size at bulk-shield exit (cm^2)	0.54 x 5.8	0.84 x 6.4

Particular attention has been given to making POSY II easy to operate by external users. Thus, for instance, the counter consists of a linear-position-sensitive detector fixed in a preselected location. This counter has an active area of 5 cm x 20 cm and a one-dimensional position resolution (along the long axis) of 2 mm (Ordela, Inc., Oak Ridge). The detector is of the gas-proportional type, filled with a ^3He-CF_4 mixture, giving an efficiency of 81% at 3 Å.

For this instrument as well, the sample will not be in the direct beam from the source. However, it was desired to eliminate the transmission filter used in the past (beryllium-cooled by liquid nitrogen) to avoid the small-angle scattering from the sintered beryllium and to extend the dynamical range of neutrons to span the maximum neutron flux from the solid moderator ("soller mirrors"). Each of the mirrors will consist of a disk of silicon 10 cm in diameter and 0.4 mm thick. Both faces are covered with 800 Å of ^{58}Ni. The whole soller mirror consists of 24 such disks, enclosed between flat reference plates, and set at an angle with the incident beam to ensure total reflection for $\lambda = 3$ Å.

The portions of the POSY and POSY II flight paths within the collimation in the bulk shielding will be evacuated, while most portions of the external flight paths for both instruments will be He-filled. Both instruments will be equipped with heavy-duty sample goniometers capable of supporting a variety of sample mountings and environments.

SAD II

The Small Angle Diffractometer (SAD) has been operating at IPNS since 1982 and has been in essentially its current configuration since 1984. This instrument has been severely oversubscribed for several years, with proposals exceeding operating time by a factor of two to three. Instrument oversubscription and our experience led us to believe we could now build a significantly improved instrument; therefore, consideration of a second SANS instrument at IPNS was begun in early 1986. Development of the new instrument (currently designated as SAD II) is under way, and the assessment of the final parameters should be completed in 1989. Many of the features of the new instrument will be, at least initially, similar to those of SAD. However, the new instrument is intended to have a much better detector system and more varied collimation options, leading to greater Q range, and is also intended to have a more convenient sample environment than does the present SAD.

In particular, the instrument will occupy the C3 beam line, where it will view the same solid CH_4 (or liquid H_2) moderator viewed by SAD. The moderator-to-sample distance will be 7.5 m, as in SAD; this is roughly the minimum distance that allows room for filters, shielding, interchangeable collimation, etc. The instrument will be, at least initially, a filtered-direct-beam instrument in which the moderator, sample, and detector are in a direct line. A filter (MgO as in SAD, or oriented single-crystal sapphire, probably at 77 K) in the incident beam line will reduce the fast neutron flux by roughly two orders of magnitude, while having a transmission of greater than 50% for the neutrons of interest (~1-14 Å). A second filter of oriented single-crystal bismuth will be used to attenuate the gamma rays from the source, if necessary. An alternate option, a "beam-bender" that would "bend" the trajectories of the long-wavelength neutrons (greater than ~1.5 Å) away from the main beam of fast neutrons and gamma rays, was considered but does not appear to permit transmission to wavelengths as short as those allowed by the filters. The apertures that define the beam from the moderator to the face of the bulk-shielding monolith for the initial filtered-direct-beam instrument have now been installed, and the necessary modifications have been made in the beam gate to accommodate the filter(s).

A two-dimensional position-sensitive gas-proportional counter detector of nominally 40 cm x 40 cm active area with 4 mm x 4 mm resolution (Ordela, Inc., Oak Ridge) was purchased for this instrument and was received in spring, 1988. This detector is filled with ^3He and CF_4 to provide the desired position resolution and neutron efficiency (61% at 2 Å) while having a low gamma sensitivity. (For comparison, the SAD detector has an active area of 17 cm x 17 cm and a resolution of ~4 mm x 4 mm). The position and time-encoding electronics for this detector, as well as the rest of the data acquisition system for this instrument, are similar to those on SAD and have already been acquired. Data acquisition will initially utilize a PDP-11/24 computer, which will be replaced with a microVax when the instrument development is further along.

The remainder of the development of the new instrument is expected to proceed in stages. Temporary beam-transport, collimation, and sample-environment components have been fabricated and will be installed in fall, 1988. These will permit detailed testing of the new detector and will also serve as a testbed for the development of other instrument components. Of primary importance in this regard are shielding to reduce the background to satisfactory levels, the filters discussed above, and the angular collimation system for the incident beam. Design and construction of the final instrument will depend in part on the outcome of the development and testing efforts with this temporary version.

To achieve the necessary angular collimation in the incident beam while utilizing the full source size (~1 cm diameter) requires the use of converging multiple-aperture collimation. If the collimation channels are all focused to the same point on the detector, then a large sample size will not affect Q_{min} or the Q-resolution[4], even if the sample-to-detector distance is short. In SAD such focusing multiple-aperture collimation is done with crossed-converging soller collimators, which define ~400 converging beam channels with essentially no "dead" space between them. This entire collimator system occupies only a distance of ~60 cm along the incident flight path, while providing angular collimation of 0.003 radians FWHM. This system has worked quite well, producing resolutions in good agreement with the calculated values, and the collimators presently in use have produced a cleanly collimated beam. These collimators, and improvements needed in such soller collimator systems, are discussed elsewhere in these proceedings[5]. One of the uses of the initial temporary flight path will be to test (and develop, if necessary) satisfactory collimators to provide even tighter resolution than this.

The practical Q_{min} with the present set of collimators is ~0.005 $Å^{-1}$. With the detector spacial resolution noted above, Q_{min} and Q-resolution down to ~0.002 $Å^{-1}$ can be achieved with the 1.5 m sample-to-detector distance simply by installing different incident beam collimators with the desired angular divergence in each channel. If satisfactory collimators can be produced, several such collimators will be provided in a "cassette" arrangement to permit easy changes in resolution and Q_{min}. A further decrease in Q_{min} and resolution will require moving the detector further from the sample, with a Q-resolution of ~ 0.001 $Å^{-1}$ achievable with this detector resolution and at a sample-to-detector distance of 3 to 3.5 m, if the incident beam collimation can be made to match. (With the moderator and path lengths being considered, the source pulse width is too short to contribute appreciably to the resolution for the cases considered here.) The count rate will fall at least as fast as the square of the resolution, so it will not be desirable to try to push the resolution this far for most cases. Since the multiple-aperture collimation must focus on the detector, a different collimator is needed for each sample-to-detector distance; therefore, there is no point in having a continuously variable sample-to-detector distance in this instrument.

With a filter in the beam, the minimum usable wavelength will be ~1 Å. With the detector at 1.5 m from the sample and centered on the beam, this will yield usable intensity at a Q_{max} of ~0.6 $Å^{-1}$. When the detector is operated with the beam near the edge of its active area, this could be increased to Q_{max} ~1 $Å^{-1}$, at some cost in intensity at the smaller Q values. However, in some cases it is desirable to have Q_{max} even larger than this, and in most cases it is desirable to be able to include measurements over a wide range of wavelengths in the high-Q data, so the ability to cover even higher scattering angles will be incorporated into the instrument. This will be done either by providing a means of rotating the detector about the sample position or by supplementing the main two-dimensional detector by an array of linear position-sensitive detectors (LPSDs) at higher angles, using the technology for position- and time-encoding of such detectors, which has already been developed for GLAD. Roughly 40 LPSDs at 1.5 m from the sample would extend continuous angular coverage out to a scattering angle of 30° and a Q_{max} ~1.6 $Å^{-1}$.

Even without the new enriched-uranium target just installed at IPNS in 1988, the new instrument would have a much higher data rate at high Q than does the present SAD (up to a factor of 10 increase at the highest Q presently obtained on the SAD), and a Q_{max} more than four times that of the present SAD. Under the same conditions, at low Q the new instrument would have a data rate comparable to that of the present SAD at the same resolution. By tightening the collimation or by changing both the collimation and detector position, Q_{min} down to 0.001 to 0.002 $Å^{-1}$ should be achievable, with a corresponding data rate reduction. However, the factor of three increase in source intensity expected from the enriched-uranium target should mean that the time required for such higher-resolution measurements will not be unreasonable in many cases.

Acknowledgments

Work supported by U.S. Department of Energy, BES, Contract No. W-31-109-ENG-38.

References

1. R. K. Crawford, D. L. Price, J. R. Haumann, R. Kleb, D. G. Montague, J. M. Carpenter, S. Susman, and R. J. Dejus, in these proceedings.
2. G. P. Felcher, R. O. Hilleke, R. K. Crawford, J. Haumann, R. Kleb, and G. Ostrowski, 1987, Rev. Sci. Instrum. 58, 609-619.
3. T. P. Russell, A. Karim, A. Mansour, and G. P. Felcher, 1988, Macromolecules 21, 1890.
4. A. C. Nunes, 1974, Nucl. Instrum Methods 119, 291-293.
5. R. K. Crawford, J. E. Epperson, and P. Thiyagarajan, in these proceedings.