ICANS XIII
13th Meeting of the International Collaboration on
Advanced Neutron Sources
October 11-14, 1995
Paul Scherrer Institut, 5232 Villigen PSI, Switzerland

# SURF - A Second Generation Neutron Reflectometer

DG Bucknall[§], J Penfold[§], JRP Webster[§], A Zarbakhsh[§], RM Richardson[‡], A Rennie[‡], JS Higgins[‡], RAL Jones[‡], P Fletcher[‡], RK Thomas[‡], S Roser[‡] and E Dickinson[‡]

§ Rutherford Appleton Laboratory, Chilton, Oxon, OX11 0QX
‡ CRG Member

## Abstract

The SURF neutron reflectometer has been built at the ISIS Facility at the Rutherford Appleton Laboratory. It is a reflectometer optimised for the surface chemistry of soft condensed matter research. The instrument has been built with almost total computer control, allowing a high degree of reproducibility and accuracy of the data. Unique to the instrument is a focusing supermirror which focusses the beam at the sample position. In addition the instrument has a 2D area detector. Some of the results from the commissioning period by RAL staff and the CRG are described.

## 1. Introduction

Over the eight years that the CRISP neutron reflectometer has been in operation it has been applied to numerous but completely varied interfacial and surface studies. This reflectometer is a general purpose reflectometer that is designed to be as flexible and adaptable as possible and can work in the non-polarised and also polarised mode. The instrument is located at the ISIS facility at RAL which provides a white beam of neutrons. The beam on CRISP is inclined at 1.5° to the horizontal to allow the easy application to liquids. The white beam TOF method of reflectivity has several advantages with constant illumination and resolution and due to the white beam provides all the reflectivity curve simultaneously. The motivation for SURF was 2 fold, firstly to make an instrument designed specifically for surface chemistry and secondly to help the pressure of demand of instrument time on CRISP. The basic instrumental requirements were:

- Optimise for surface chemistry
- Provide a large wave-vector transfer, Q
- Ensure a high degree of accuracy and reproducibility on absolute scale
- Provide a good signal to background noise ratio
- Have full control of reflectometer using sophisticated instrumentation
- Have sophisticated and controlled environment
- Increase flux at sample with the capability of relaxed resolution

## 2. SURF Design

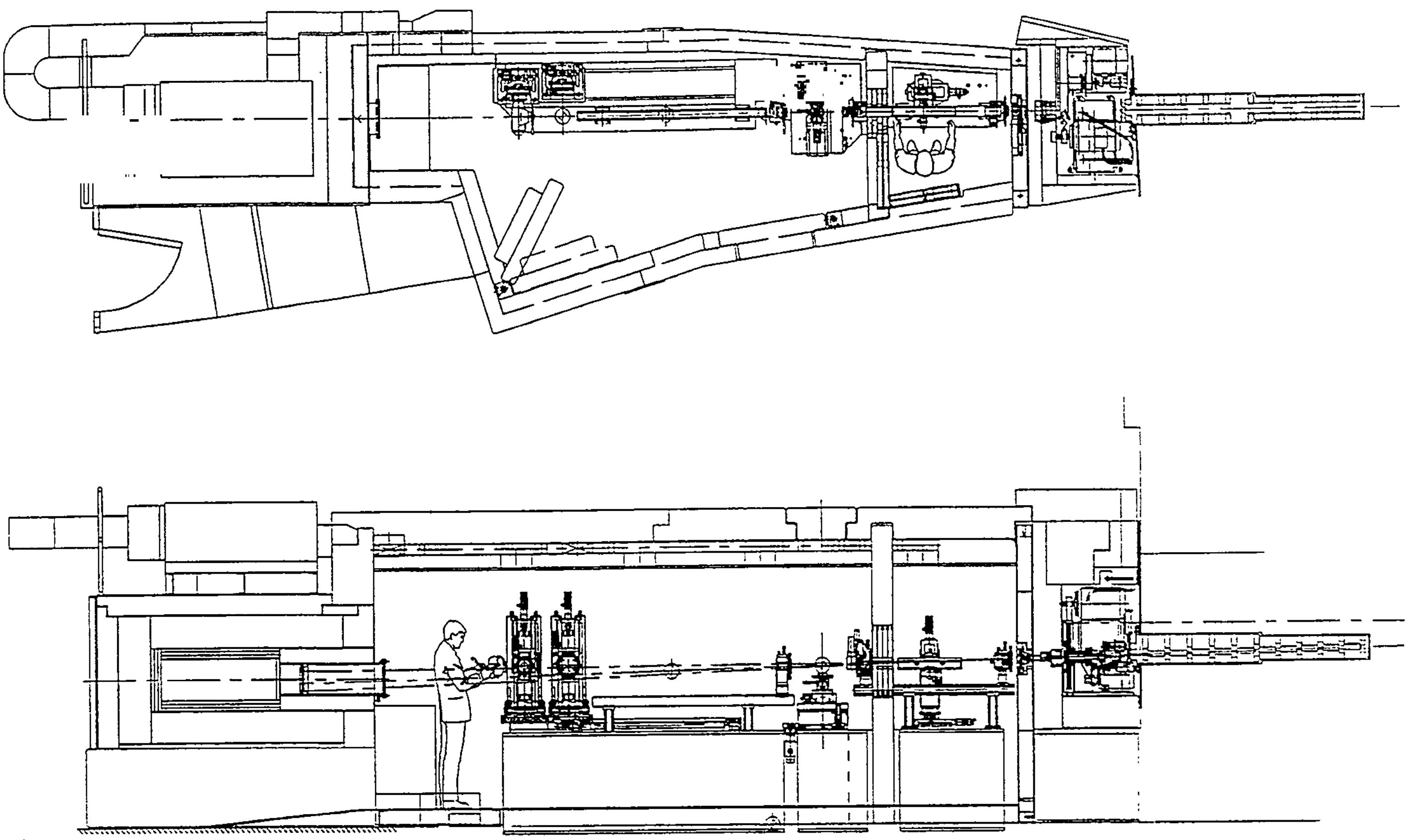

*Figure 1: A schematic layout of the SURF neutron reflectometer in plan and side cross-sections.*

The SURF neutron reflectometer has been installed at the ISIS facility viewing the liquid hydrogen moderator, a schematic layout is shown in Figure 1. The beam is guided down a neutron absorbing shielded collimation tube which defines the 1.5° inclined beam to the horizontal. The beam is defined using a nimonic chopper to remove the fast neutrons and gammas from the beginning of every pulse as well as a wavelength selective double disc chopper. Both choppers are located in a which is well shielded enclosure providing high quality shielding from the target but also from the instruments on either side and the rest of the SURF instrument. The choppers can operate at either 25 or 50 Hz giving a wavelength selection between 0.5 and 13 Å depending on the chopper frequency and disc aperture selected. After further collimation via a pair of coarse collimation jaws a beam of dimension of approximately 60 × 10 mm is obtained, which after passing through a low efficiency scintillator monitor, enters the block house. Great care has been taken in the design of the block house to try and reduce background noise, for this purpose the frame overlap mirrors (FOMs) and focusing supermirror have been separated from the sample area by incorporation of a shielding wall just before the sample position. Accurate computer controlled beam defining slits before the FOMs and also at the sample position allow the illuminated length and resolution to be defined. The sample position has a full set of movements available to it, including height, lateral translation and the possibility of angular motion via 2 perpendicular goniometer arcs. Post sample there is the option of 2 detectors either a single detector or a 2D area detector (to be installed at the end of October 1995). Both detectors are based on a scintillator design. Both these detectors sit on height and rotation stages, and these assemblies are mounted on a translation stage which allows variable sample to detector distances of 1 - 2.43m. At the sample position there is a growing number of sample environment equipment, from the basic goniometer platform, static and dynamic (Nima) liquid troughs, solid sample changer, liquid-solid cells and liquid-liquid cells. The whole instrument enclosure is a controlled environment.

The focusing supermirror is a unique feature of the SURF reflectometer. The mirror has a supermirror coated reflecting surface of 600 x 60 mm, but it has a 2000m radius of curvature. This gives a focusing effect of the beam so that the effective illuminated length is reduced by 3 without loss of neutron flux. (This option has not been installed at time of writing, but the mirror has been purchased and awaits supermirror coating to be deposited.) The supermirror has the option to deflect the beam so that the beam either comes from above but now no longer at 1.5° to the horizontal, or from below. This will allow extreme flexibility of operation for various systems. Without the supermirror there is an approximate seven fold increase in the neutron flux over the reflectometer CRISP. This increase comes from a combination of an increased view of the hydrogen moderator, increased beam size and hence a relaxed resolution specifically for liquids and a shorter moderator to sample distance with respect to CRISP. Optical ray tracing calculations have demonstrated that the use of the focusing mirror will reduce the footprint at the sample by 3 times.

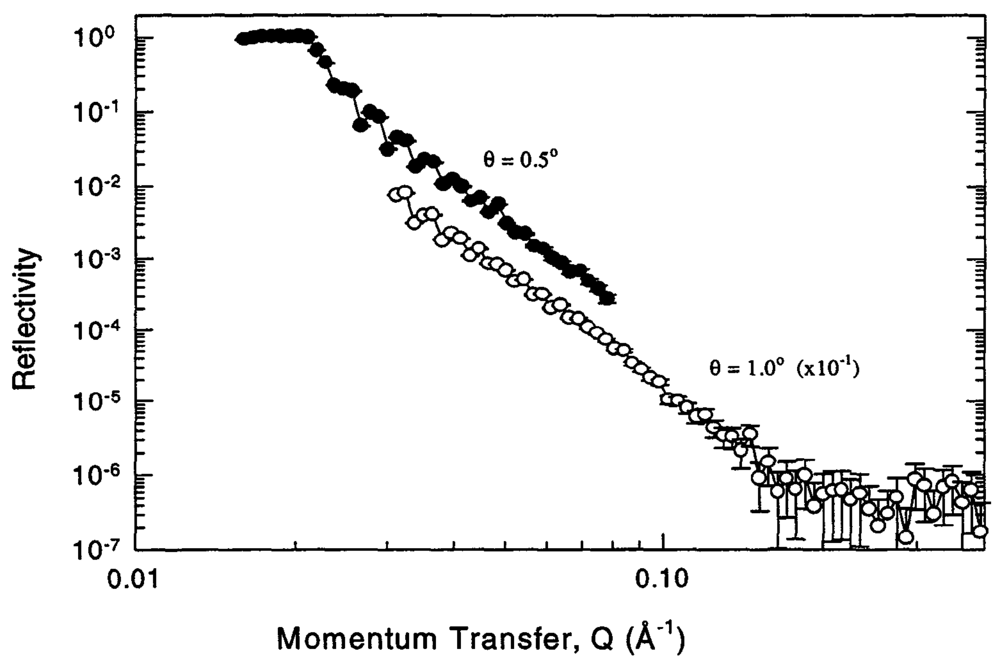


*__Figure 2:__ Reflectivity profile of a NiC film on quartz. The closed circles are the reflectivity profile obtained at an incident angle of θ = 0.5°. The profile obtained at θ = 1.0° is translated by a factor of 2 for clarity.*

## 3. Science on SURF

Although the SURF reflectometer is not fully operational since not all its beamline components installed it has nevertheless been through a vigorous commissioning period during which members of RAL staff and the CRG have been able to push the instrument into areas not previously accessible to the CRISP. Here we present some of the elegant work that has been contributed so far to demonstrate the power of the reflectometer.

Perhaps the place to start is at the beginning with the first data from the reflectometer. It is of a NiC film deposited on quartz. The film is a well characterised sample and shows in Figure 2, the reflectivity profile taken at 2 angles, which are displaced for clarity. The beam defining slits before the sample were varied between the runs to keep a constant illuminated length and resolution. The interference fringes are well pronounced even though the sample was run under a modest resolution of $d\theta/\theta = 5\%$. Although not apparent in this plot the data from both angles overlaps extremely well and indicates the control and precision of the beamline components. Another standard sample used for calibration is the surface of pure $D_2O$, as seen in Figure 3. The open circles is the specular reflectivity of a $D_2O$ using the single detector, showing a background of better than $2 \times 10^{-6}$ in reflectivity. By moving the single detector off the specular position and measuring the off-specular background it is clear that the background observed in the specular reflectivity plot is indeed the true sample dependent background. These data were measured using a resolution of $d\theta/\theta = 10\%$ allowing the data to be collected extremely quickly of the order of 10 of minutes.

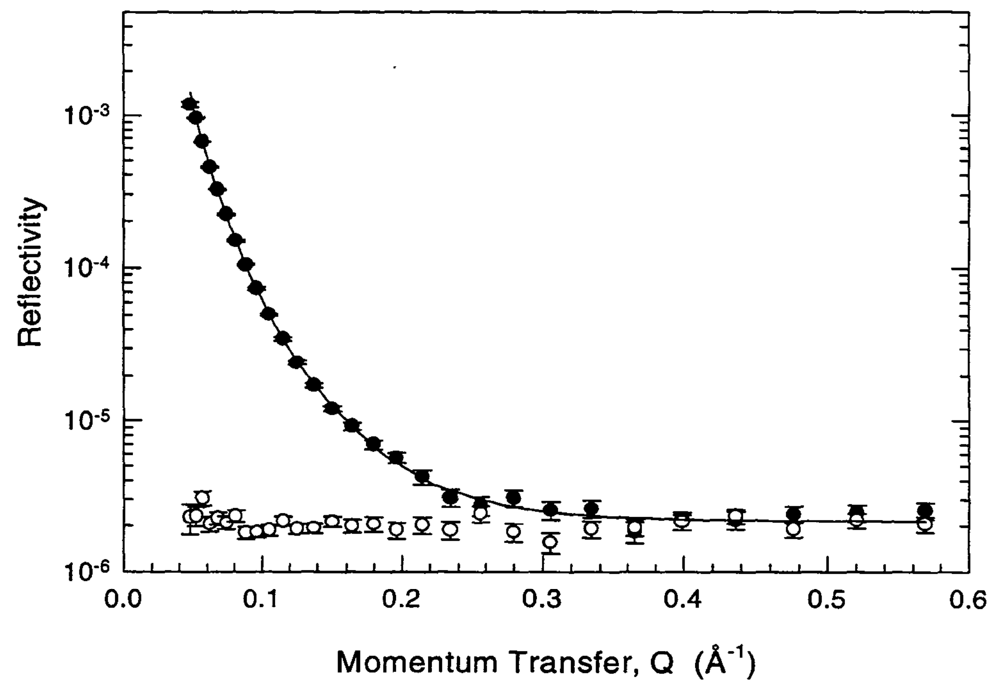


***Figure 3:*** *Plot of the specular reflectivity profile (closed circles) of the surface of D2O with an icident angle of $\theta = 1.5°$. The off-specular profile of the D2O surface obtained by moving the detector off the specular position to 1.65°.*

Instrumental backgrounds have been reduced to the absolute minimum on the reflectometer by careful incorporation of neutron shielding, not only in the design of the blockhouse but also by incorporation of neutron absorbing materials in slits and detector housing. Typically the background for the instrument is below $10^{-7}$ in reflectivity, as can be seen in Figure 4, where the surface of Si has been measured at 3 angles, which have been over plotted. This decrease in background noise has provided a good signal to noise ratio. This has been seen for a hydrogenous thermotropic liquid crystal monolayer on a silicon substrate. The minima of the interference fringe is visible at a reflectivity of approximately $10^{-6}$ which would perhaps not have been possible to observe without all the enhanced signal to noise ratio.

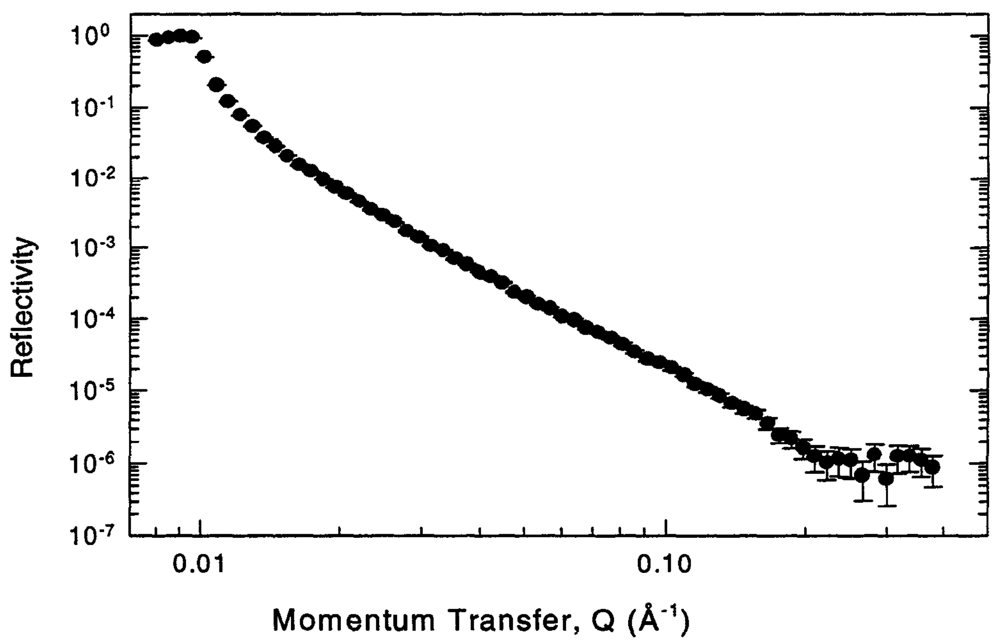


***Figure 4:*** *Reflectivity profile of the surface of Si with its natural oxide in air. The profile was obtained by combining the reflectivity profiles from 3 incident angles of* $\theta$ *= 0.25, 0.7 and 2.0°.*

Of course, the reflectometer is not restricted to relaxed resolution measurements and in fact a large proportion of the data collected so far has used resolution down to $d\theta/\theta = 2$ or 3%. There is of course a flux loss penalty over the relaxed resolution case but this is still a significant flux gain over similar measurements on CRISP. An example of the high resolution work is shown in Figure 5. This is a solid polymer film of polystyrene which incorporates a deuterated end-functionalised telechelic polystyrene. The reflectivity profiles shown are for the unannealed as made sample and also after annealing to equilibrium above the glass transition temperature. The interference fringes are well defined and the fits to the data suggest that there is segregation of the deuterated telechelic polystyrene to both the air-polymer interface as well as the polystyrene-Si substrate interface.

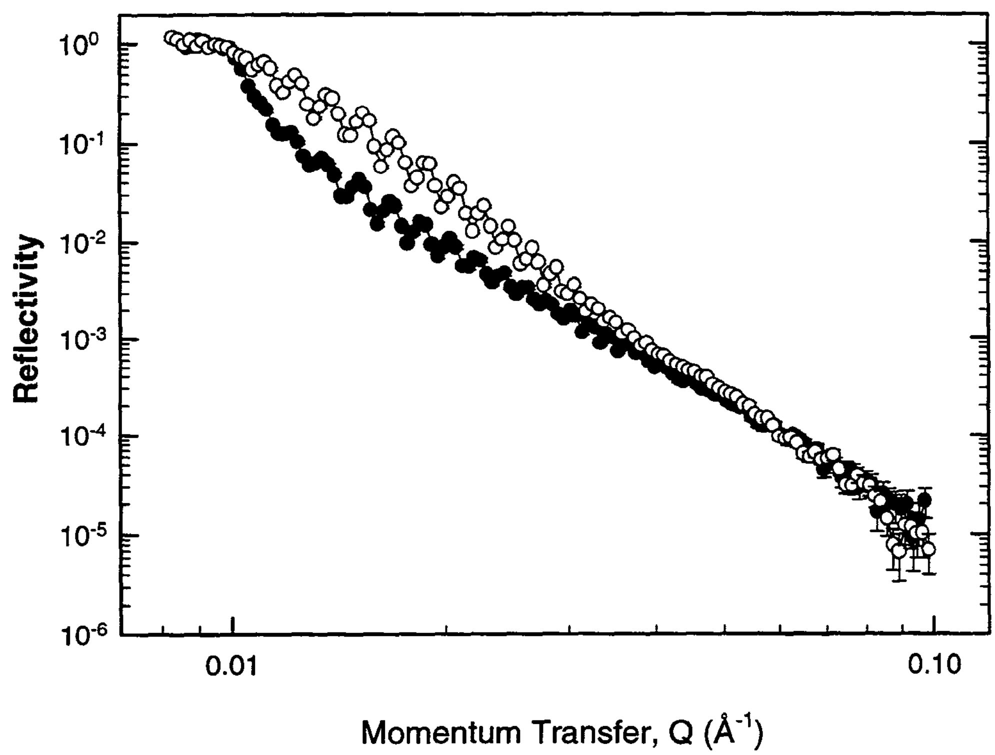


*Figure 5: Two reflectivity profiles over-plotted obtained from a thin polystryrene film with a small percentage of end-functionalised telechelic polymer, in the as made state (closed circles) and after annealing above the glass transition temperature (open circles).*

Perhaps the most stunning result of the commissioning period has been the speed at which meaningful data can now be collected due entirely to the increase in flux. The best demonstrations of this have been using the instrument to its full design potential with wide beam defining slits giving maximum beam dimensions at a relaxed resolution. From work on monolayers on water are 2 examples, which perhaps shows that now truly kinetic effects can be observed. As shown in Figure 6, an insoluble monolayer on $D_2O$ has been measured as a function of time at fixed surface pressure. Each of the lines represents a reflectivity profile collected in 20 minute scans. The obvious change in the reflectivity is due to the monolayer collapsing and from this data it is possible to extract useful data about the layer thickness and density. In an even more stunning example measurements on a different system were allowed the adsorbed monolayer thickness and adsorbed amount to be determined from reflectivity profiles each collected in 5 minutes.

## 4. Future work

The commissioning period of the SURF reflectometer will be completed after the installation of the focusing supermirror (late November 1995) and 2D area detector (in late October 1995). The instrument will become a fully scheduled instrument after at the end of the ISIS 1995

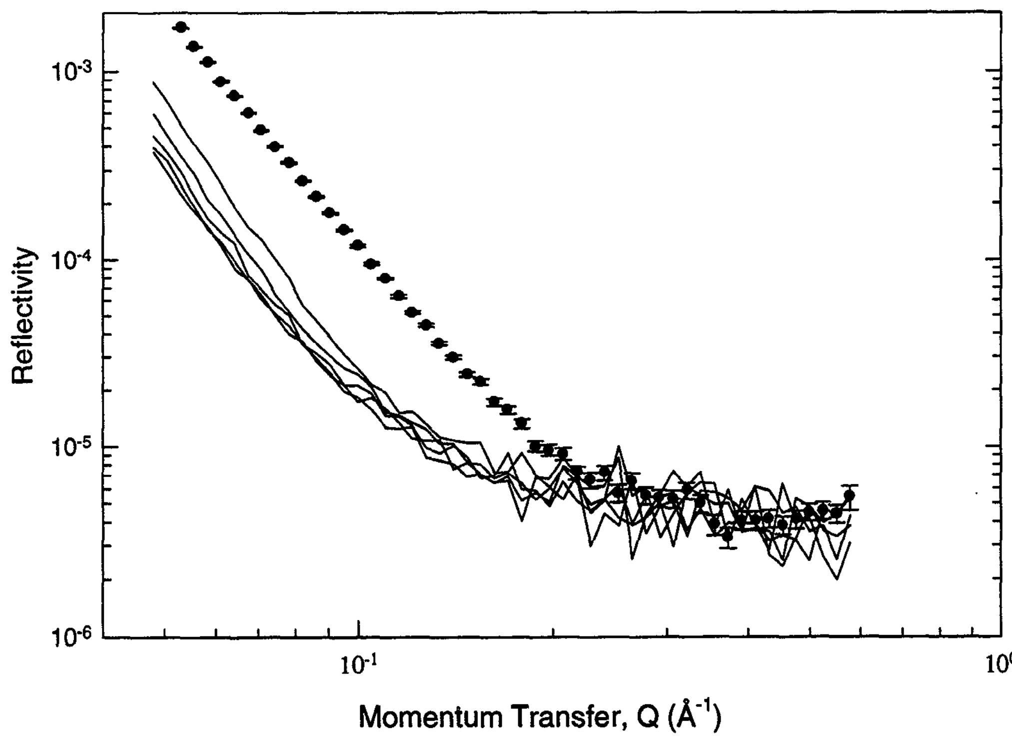


*Figure 6: Kinetic reflectivity profiles obtained from a collasping polymer monolayer (lines) on $D_2O$ (closed circles). Each line represents a reflectivity profile of the monolayer obtained in 20 minute measurements.*

schedule. At the time of writing the instrument is accepting its first set of proposals for scheduling and will become fully incorporated into the suite of instruments at ISIS available to the user community. With the installation of the focusing supermirror it will be possible to push areas not fully exploited as yet. Kinetic studies will now become reality as has already been observed for some systems, but also more exotic systems will be achievable since it will mean smaller sample volumes. In addition the 2D area detector with start to really test some of the newer theories of off-specular scattering which are emerging in literature.

## 5. Acknowledgements

The authors acknowledge the financial backing of the EPSRC for funding the SURF CRG project, as well as additional contributions from ANSTO, Australia. We also gratefully acknowledge the help from the technical staff at ISIS in the design and construction of the reflectometer, especially the project design engineer Richard Coleman. In addition we acknowledge the help in commissioning the instrument from I McLure, A Burgess, E Staples, J White, M James.