Beamline Name: **INTER**

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Summary:

In the last fifteen years or so neutron reflectometry has been established as a powerful and in many cases essential technique for studying surfaces and interfaces [1]. One of the particular strengths of the technique is that it can be applied to ‘wet’ interfaces attaining information at better than molecular resolution [2, 3]. These properties of neutron reflectivity allied with the ability to advantage of hydrogen/deuterium exchange make it especially suitable to the study of interfaces in soft condensed matter systems. To this end the SURF [4] and CRISP [5] reflectometers have been established as world leading reflectometers.

INTER will improve upon the capabilities of current reflectometers providing a pre-eminent facility for the study of interfacial structure in soft matter by neutron reflectometry. It will provide access to a range of interfaces (air/liquid, air/solid, liquid/liquid and liquid/solid), enable the use of smaller samples, allow studies of and access to a wide range of experimental parameters and expand the time scales that are observable for dynamic studies. These features will provide exciting new opportunities in soft matter research and related areas.

Science Case:

The current pulsed neutron sources have been optimised primarily for thermal and epithermal applications. Experience has shown that reflectometry is best performed with cold neutrons. Even though the current ISIS target station is not optimally suited to reflectometry the range and impact of the science that has been carried out on both the SURF and CRISP reflectometers is impressive and world leading. The development of a source that has characteristics optimised for reflectometry can only be expected to take this technique into new and exciting territory. The design of the Second Target Station will be optimised for the production of a high cold neutron flux with a relaxed time structure and broad wavelength range.

The INTER reflectometer will view the grooved surface of the cold coupled methane moderator giving the best possible flux gains without compromising resolution or signal to noise. The lower repetition rate of the source will result in a wider range of neutron wavelengths being available within each pulse, greater than double that from the current target station. It will therefore be possible on INTER to measure a wider simultaneous Q range in a shorter time than is currently achievable on either SURF or CRISP for a reduced Q range. Conservative estimates indicate that the improved performance will be greater than fifteen times.

In addition to allowing a wider parametric space to be explored in shorter time, the increased flux will inevitably lead to a smaller sample size. Such flux-limited experiments are currently confined to samples with high reflectivities ($>10^{-3}$) at Q values close to total reflection. One of the current restrictions in sample preparation, namely that the interface should be atomically 'flat' over an area of typically 1000 mm^2 can be relaxed. This dramatically opens up the range of substrates and experimental conditions that can be employed. It will be possible to probe unique domains of the interface, making measurements with lateral spatial resolution. This could have a particular impact in surface ageing studies where the regenerated surface characteristics depend critically on the spatial separation from the regeneration point, for example, protein folding at the air-water interface in which the interface is regenerated by means of an 'overflowing weir' arrangement.

The benefits outlined above may be attained in isolation or by combining a number of these together. This will lead to the application of reflectometry to more complex and multi-component systems, enabling the investigation of systems and materials more closely aligned with that found in industry or nature. It may well obviate the need in many circumstances to develop model systems which mimic 'real world' applications, for example adsorption at mineral interfaces, influencing processes such as flotation, will become possible on the minerals themselves rather than needing to develop a model interfacial structure.

Kinetics:
Polymer interdiffusion driven either thermally or under pressure by super-critical solvents, placticiser ingress into polymer films, diffusion limited adsorption processes at the air-liquid and solid-liquid interfaces, Slow reaction kinetics at interfaces, e.g. hydrolysis in surfactant films or esterification in biomineral templates.

Biological Membranes:
A growing area of interest in reflectometry will benefit greatly from increased flux and bandwidth allowing smaller samples to be employed in fixed experimental geometries, greatly simplifying sample preparation and increasing the breadth of systems accessible to neutron reflection.

Liquid-Liquid Interfaces:
The increased flux may allow studies wherein a substantial thickness of the upper, oil phase is traversed. At present this thickness is limited to less than 10 microns in order to accommodate the high attenuation of the neutron beam in any liquid and this places severe constraints on the experimental methodology and sample environment.

Air-Liquid Interface:
Although the incident angle at this interface is adjustable using supermirror optics the concomitant loss of bandwidth transported to the sample and the necessity to realign the sample means this is not ideal. INTER's increased bandwidth will allow lower Q ranges to be explored with a fixed sample geometry. The shift of the peak flux to longer wavelengths on the Second Target Station also allows for a larger fixed 'natural' incident angle at this interface. At 2.3° on

INTER we will probe the same maximum Q as SURF at 1.5° but with a much lower minimum Q. At this angle the peak moderator flux is matched to the region of interest for most thin films (surfactants, polymers) at the air-water interface. This increase in incident angle also gives rise to an additional increase in flux since the resolution of the experiment will be governed by the $\cot(\theta).\delta\theta$ term. Thus we gain of order 1.5 in flux and 2.3 in bandwidth over the equivalent experiment on SURF. This flux gain is entirely geometric. The total expected flux gain is an order of magnitude greater than SURF.

Small Samples:
The tighter beam collimation and improved flux will result in many small samples, which are at present impractical for neutron reflectometry, becoming routine systems. In many systems particularly biological where there is very little material available the volume of sample required can often select or determine the types of experiments that can currently be attempted. Additionally a small beam can be used to selectively probe localised regions of heterogeneous surfaces as is generally encountered on technologically relevant metal and metal oxide coatings.

Dilute Systems:
Reflectometry is a methodology that generally probes weakly scattering systems, with the improved statistics we will be able to investigate systems of even lower concentrations, or examine deutero/hydro mixtures at interfaces where one component is in excess.

Complex Environments:
The penetrating power of neutrons make them a valuable tool for examining buried interfaces and interfaces within sample environments where the pressure or temperature is regulated. In-situ studies of electrochemical systems will also become more amenable to reflectometry, for example the improved time resolution will enable the study of dynamics of solvent transfer and film structural evolution in response to an electrochemical stimulus.

Solid/Liquid Interfaces:
At present the solid/liquid interface is studied by passing the neutron beam through a solid substrate, usually silicon (although quartz and sapphire have also been used). Silicon though is not always the substrate or support material of choice. Reflectometry at the second target station offers the possibility of working with substrates with a transmission of less than 5 % while still retaining measurements times similar to that of the CRISP and SURF reflectometers. This will open up a wide area of science not currently tractable by reflectometry.

Parametric Studies:
The flux gains will enable many measurements to be made much faster. This is particularly important in many of the complex multi-component systems where a large parameter space, or a number of different contrasts, need to be measured.

Resolution:
The improved signal to noise ratio and incident flux will enable more detailed isotopic labelling schemes to be envisaged, resulting in improved optical resolution of the technique. The intrinsically lower background of INTER compared to SURF and CRISP will enable higher values of Q to be reached, also improving the spatial resolution.

Specific Areas of Science:

It is anticipated that the following scientific areas will benefit from the construction of the reflectometer INTER on the Second Target Station.

Soft condensed Matter:

- Interfacial studies: the self-assembly, ordering, competition between components and displacement in complex mixtures of surfactants, polymer and proteins at interfaces will become more accessible.
- It will be possible to work with more complicated systems enabling better models of real world systems to be employed.
- Kinetics of multicomponent mixtures at technologically relevant interfaces (liquid-liquid and liquid-solid) will benefit from the improved speed and real space resolution that will be achieved on INTER.

Polymer Systems:

- The ability to deuterate polymers means that neutron reflectometry offers a powerful means to study processes at interfaces between what may otherwise be identical or nearly identical polymers. It will be possible with appropriate deuteration schemes to define interfacial widths and shapes on an unprecedented scale.
- The data collection speed on INTER will mean that diffusion at polymer interface will be tractable to real time studies in a variety of systems. This will also include the ingress of small molecules into polymer matrices.

Advanced Materials:

- The ability to collect data more quickly and use smaller substrates will enable real time investigation of sensors. The penetration of the neutron beam through the complex sample environment that surrounds sensors designed for use at extreme temperatures or pressures will provide a powerful method for understanding and optimising sensor performance.
- Self-assembly of templated films at an interface has the potential to produce film structures containing mesoscopic pores which are either randomly oriented or in ordered arrays parallel to or perpendicular to the interface. Neutron reflectometry will be an excellent method to follow the development of the interfacial structure in these high surface area materials in real time.

Biomolecular Sciences:

- Much progress has been made recently on the production of supported fully hydrated phospholipid films, providing a more realistic model of cell membranes. It is anticipated that this will lead to a significant increase in the range and relevance of biological experiments. The competitive adsorption of proteins or antibiotics onto and into these membranes, and at other interfaces will be much more readily accessible on INTER.
- The temperature induced denaturation of proteins can be studied at solid/liquid and air/liquid interfaces on CRISP or SURF to arrive at interfacial unfolding energies, typically lower than that measured in the bulk solution. Performing these types of experiments on INTER would also enable the kinetics of the unfolding to be studied as a function of temperature.

Food technology:

- The production and stability of many ‘new’ food products is predominantly a colloid science problem, related to emulsion stability. The adsorption of proteins to oil-water, air-water, air-oil interfaces and the stability of that interface are important parameters in food stability. INTER will enable studies of more complex, more dilute food related systems than has been possible in the past.

- Protein fouling of process equipment can be a major problem in many food production industries. This effect can be caused by very dilute components in the mixture, for example the interaction of different genetic variants of the same protein can result in drastically different fouling rates. The spatial resolution and low background of INTER will be required to study these systems under realistic conditions.

Outline Design Specification:

Front End Optics:

INTER will benefit from an optimised guide section consisting principally of an m=4 bender section allied to straight m=3 supermirror guides. Figure 1 shows a possible configuration. This will allow the transport of a 60mm wide beam of bandwidth 1Å ≤ λ ≤ 16Å to the sample position. The increased angle to the horizontal and the moderator to detector distance will result in the instrument detectors will be out of line-of- sight of the moderator, reducing intrinsic background. *Detailed design parameters for the guide system are currently being evaluated with Monte-Carlo type simulations of beam transport.* The possibility of beam-cramping in the final post-guide section of beam collimation is also being investigated. This would reduce the width of the beam to match the sample size resulting in an increased flux at the expense of lateral resolution. For purely specular measurements intensity is integrated across the beam profile and the resolution in that direction is therefore unimportant.

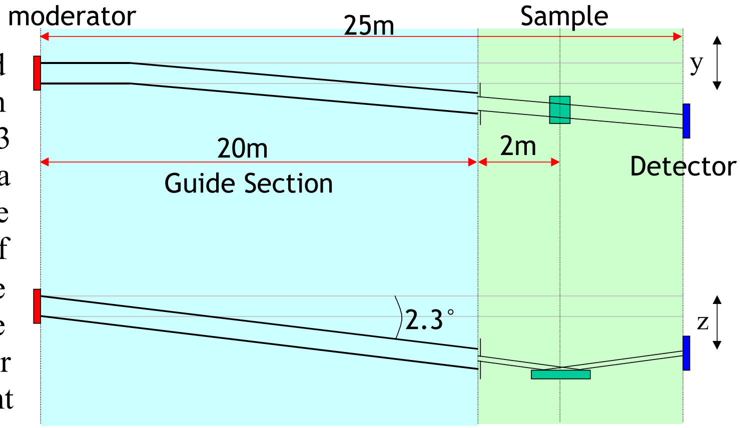

Figure 1: Illustration of the probable guide configuration for INTER

Background Reduction:

In conjunction with the reduced background from the source INTER will also employ either evacuated or Argon gas filled flight paths from the final guide section to sample position and from sample position to detector, further reducing the instrument background due to parasitic scatter. With the longer flight path than is currently the case on SURF we also anticipate significant gains in the intensity at the detector. Such flight paths will be designed to allow ready access to the final optical elements.

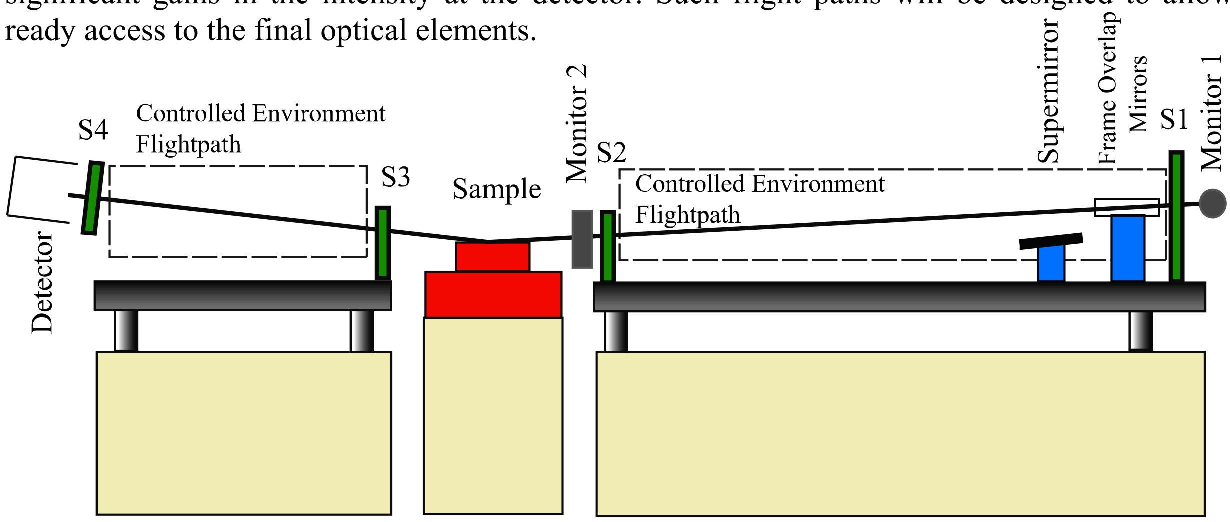

Figure 2: Schematic layout of the INTER reflectometer on Target Station II, not to scale.

Detectors:
Figure 2 shows a schematic of a possible beam layout. An optimised ^3He single detector will still be required for measurements with high count rate and where a high signal to noise ratio is required. Off specular measurements will require a 200 x 200 mm detector with a resolution of the order of $1mm^2$. The combination of the source characteristics and front end optics of INTER will result in a significantly lower flux of high energy neutrons entering the instrument blockhouse, helping to reduce detector saturation when making transmission measurements. This will open up the possibility of routinely measuring both the transmitted and reflected beams using either the area detector alone or by combined use of the two detectors. In combination with multidetector measurements this will help diagnose and characterise 'off specular' scatter where the source can be small-angle scattering or in-plane correlations. There may be a strong case for having a detector dedicated to measuring the transmitted beam.

Signal to Noise:
It is proposed that significant gains in signal to noise can be achieved by increasing the sample to detector distance over that currently employed on SURF. The predicted gains arise not only from improved background reduction but also from the improved discrimination of coherent, specular scattering over incoherent scattering (see figure 3). The incoherent scattering is propagated into all directions equally from the sample. Increasing the sample to detector distance with the slits set at the same resolution therefore gives rise to this enhanced discrimination.

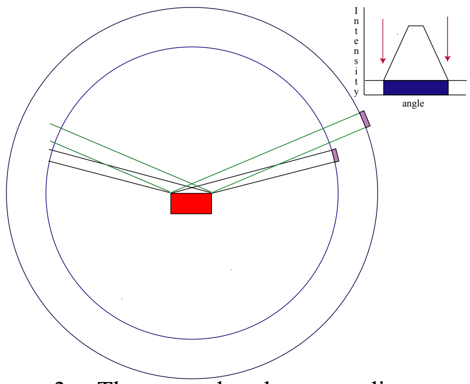

Figure 3: The sample detector distance is increased by 20% yielding a 1.5x gain in signal to noise over SURF

Bandwidth:
The proposed moderator to detector distance is 25 metres. The installation of optimised guide optics will transport neutrons with a wavelength of 1 to 16 Å into the instrument blockhouse. This results in an effective bandwidth ***gain of 1.5x*** over that transported to SURF, experimentally this will result in an improved performance of about ***1.5x.(e.g. figure 4)***

Flux:
The following flux comparisons can be made between a reflectometer situated at Target Station One running at 200μA viewing a H_2 moderator and one at Target Station Two viewing a grooved coupled methane moderator. There are gains to be made from the optimised moderator, target station, incidence angle, bandwidth, and the optics, with a slightly negative impact of lower power dissipation in the target.

At 1 in 5 pulses from ISIS at 300 μA. Cold neutron flux /μA	=	15~20 x
Power ratio 48 kW / 160kW	=	0.3 x
Increased angle of incidence 2.3° (spectral peak from 2.4 to 4A)	=	1.7 x
Increased Q range (from bandwidth)	=	1.5 x
Improved optics (supermirror guides)	=	1.5 x

TOTAL GAIN* of *17* to *23 over SURF on Target Station One.

Figure 4 gives an indication of the improved capabilities of INTER compared to SURF. The left hand panel shows the reflectivity as a function of wavelength, SURF and INTER have different incident angles, hence the different fringe minima position. It is immediately obvious that INTER will cover about twice the Q range in a single measurement. It can be seen on the right hand side that INTER as well as covering a much wider Q range per measurement will also offer improved resolution as evidenced by the clearer Keissig fringes shown. The improved flux on INTER is evidenced by the much poorer counting statistics in the SURF simulation

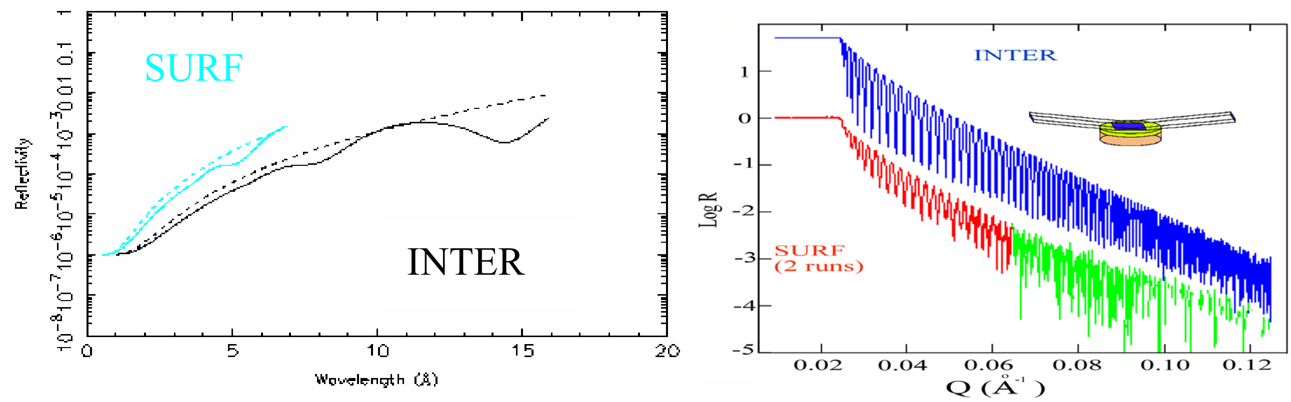

Figure 4. Reflectivity Simulations. Left Hand Panel, Air/D_2O interface with and without (dashed lines) a 200Å deuterated layer. Right Hand Panel, a 4000Å polymer film showing INTER's superior resolution and flux.

Sample Environment and Combined techniques:

Sample Environment:

The sample environment requirements for INTER will be more demanding than that on SURF, reflecting the greater range of systems that will be amenable to study. There will be more space around the sample position to allow for complex sample environment equipment to be positioned, allied with this overhead crane access will also be possible. To cope with the size and range of sample environment equipment and the smaller beam sizes that will be required in some instances the load carrying capability and positioning precision of the sample stage will be improved accordingly.

Combined Techniques:

INTER represents the next generation of neutron reflectometers, allowing for higher resolution and enhanced measurement speed. It will therefore to attract new neutron reflectometry users, many of whom may wish to incorporate their laboratory based methods into the neutron reflectometry experiments where possible. The increasing use of INTER to study dynamic processes means that there will be a greater demand to incorporate other techniques for simultaneous measurement so that the results can be compared directly. The increased length of the beamline compared to CRISP or SURF will mean that there will be more space, particularly laterally, at the sample position.

X-ray Reflectivity:

The provision of an in-situ x-ray reflectometer operating in parallel with INTER would immediately open up the possibility of collecting datasets with two contrasts at air/liquid and air/solid interfaces. This would be a system that is without peer on any source in the world. Mounted independently from the neutron optics this facility will provide an added dimension for some air-liquid and air-solid interface experiments. In particular studies of Langmuir films will benefit from in-situ studies particularly when one is following the growth of a new phase with time. Not all soft-condensed matter systems lend themselves to specific deuteration. For example fluorinated surfactants and polymers, or samples in which the organic chemistry is not sufficiently tractable. X-ray reflectometry would provide an additional contrast whilst ensuring that the structure probed is identical to that seen by the neutron experiment.

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