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# **Report on Neutron Reflectometry for the Australian Replacement Research Reactor**

**Neutron Reflectometry Workshop**

**Australian Nuclear Science and Technology Organisation.**

**8<sup>th</sup> – 9<sup>th</sup> of May, 2001.**

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# Report on Neutron Reflectometry for the Australian Research Reactor

Prepared by participants at the Neutron Reflectometry Workshop, held at ANSTO on the 8-9<sup>th</sup> of May, 2001.

Edited: Michael James

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## Executive Summary

There is a clear need for at least one neutron reflectometer at the Australian Replacement Research Reactor when it commences operation in 2005. The participants at the reflectometry workshop have identified that the neutron reflectometer to be built at the Australian Replacement Research Reactor must be capable of the study of:

- *Specular scattering from air/solid, solid/liquid and in particular 'free liquid' samples; and*
- *'Off-specular' scattering from the above sample types.*
- *Kinetics phenomena on a minute or slower time scale;*
- A range of samples of differing thicknesses, ranging from ultra-thin films to thousand angstrom thick films. In order to achieve this the reflectometer should have *the capacity to vary its resolution.*

Interest was also expressed at the ability to conduct *glancing-angle and wide-angle scattering* studies for the investigation of short length scale, in-plane structures. There was little interest expressed by the workshop participants for polarised neutron reflectometry.

This report contains a scientific case for a neutron reflectometer to be built at the Australian Replacement Research Reactor on a cold neutron guide, which is based on the areas of scientific research expressed by the workshop participants. In addition, trends in neutron reflectometry research conducted at major overseas neutron facilities are noted.

The new neutron Reflectometer should:

- Be based on the *Time-of-Flight* method;
- Have a *vertical scattering plane* (i.e. operate for horizontal samples);
- Be located *on the end of a cold neutron guide*, or be built off the guide axis using a *bender*;
- Have a *position sensitive area detector*;
- Be similar in spirit to the new D17 reflectometer at the ILL.<sup>1</sup>

Basic aspects of a reflectometer design are discussed which meet the above-stated scientific criteria and include a preliminary list of instrument specifications, capabilities and ancillary equipment requested by the workshop participants. A preliminary instrument design is recorded that includes the major necessary components.

Finally, the report lists a number of questions that require further calculation over the next 12 months in order to optimise different aspects of the design of the reflectometer.

## 1. Introduction

On the 8-9<sup>th</sup> of May 2001, a workshop to define the neutron reflectometry requirements for Australia's Research Reactor was held at ANSTO, Lucas Heights. At present it is considered that there will be one dedicated neutron reflectometer at the reactor when it commences operation in late 2005.

The workshop attracted 49 participants from Australia, New Zealand, France, Germany and the United States from diverse scientific fields such as organic, physical and biochemistry, polymer and colloid sciences, physics, engineering, steel processing, petroleum geology, industrial chemical and chemical engineering, and materials science as well as neutron optics. More than 25 other researchers expressed an interest in attending the workshop, but were unable to attend due to other commitments.

The purpose of the workshop was to:

- promote the techniques of neutron and X-ray reflectometry to the Australian Scientific community
- identify the future needs and opportunities in this area, and
- specify instrument requirements based on the future needs.

*This document represents the accumulated views of the participants at the workshop.*



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**Front Row (left to right):** M. Middleton, S. Bandyopadhyay, D. McKenzie, F. Ott, R. Cubitt, J. White, J. Ankner, M. James, I. Snook, J. Foster, I. Gentle, R. Robinson.

*A detailed list of Workshop Attendees and affiliations is in Appendix A.*

## 2. Scientific Case for a Neutron Reflectometer at the RRR.

### Areas of Scientific Study using Neutron Reflectometry

Neutron reflectometry is used to probe the structure of surfaces, thin-films or buried interfaces and processes occurring at surfaces and interfaces such as adsorption, corrosion, adhesion and interdiffusion. In particular, recent years have seen an explosion of interest in the biosciences as well as the emerging field of nanotechnology. Table 2.1 lists a number of systems that can be examined using reflectometry.

**Table 2.1 Systems applicable to be studied using reflectometry.**

<b>Soft-Matter Systems</b>	<b>Hard-Matter Systems</b>	<b>Magnetic Systems</b>
Phase separation in polymer films	Photosensitive films	Multilayer materials (eg GMR's)
Interlayer diffusion and roughness	Quantum dots	Magnetic monolayers
Inorganic templating at air/water interfaces	Catalytic surfaces	Depth-dependent domain imaging
Complex fluids under flow	Electrochemistry	Superconducting thin films
Vesicles and gels	Polymer coatings	Superparamagnetic monolayers
Reaction Kinetics	Self-assembled monolayers	Exchange-biased interfaces
Surfactants at interfaces	Non-linear optical systems	Magnetic tunnel junctions
Interfacial structure in drug delivery systems	Glancing Angle Diffraction	Hard/soft magnetic multilayer combinations
Membranes and their intermolecular interaction		
Protein adsorption		
Critical phenomena in fluid systems		
Biocompatibility and sensors		

### Basics of the technique

Specular neutron reflectometry probes the neutron *Scattering Length Density* (SLD) normal to the surface at depths of up to several thousand Å, with an effective depth resolution of a few Å. What is measured is the profile of reflectivity as a function of angle beyond the critical angle for total external reflection (Figure 2.1). The sample must thus present a smooth, flat surface, preferably several cm<sup>2</sup> in area.

*Specular Reflectivity* is measured when the angle of the neutrons incident onto the surface ( $\theta_i$ ) is equal to the angle of the reflected neutrons ( $\theta_r$ ). The reflectivity ( $R = I_r/I_i$ ) is the number of neutrons reflected from a surface divided by the number incident onto the surface. Typically the reflectivity is displayed as a function of  $Q$ .

$$Q = \frac{4\pi \sin\theta}{\lambda}$$

(where  $\lambda$  is the wavelength of the neutrons and  $\theta$  is the incident angle).

The *scattering length density (SLD)* for neutrons is the number density of an atom or molecule (N) times the neutron scattering length (b) of the atom or molecule.

i.e.

$$\text{SLD} = N_A \frac{\sum_i \rho_i b_i}{M_i}$$

( $N_A$  = Avogadro's number,  $\rho_i$  is the density of the element  $i$  with molar mass  $M_i$  and neutron scattering length  $b_i$ ).

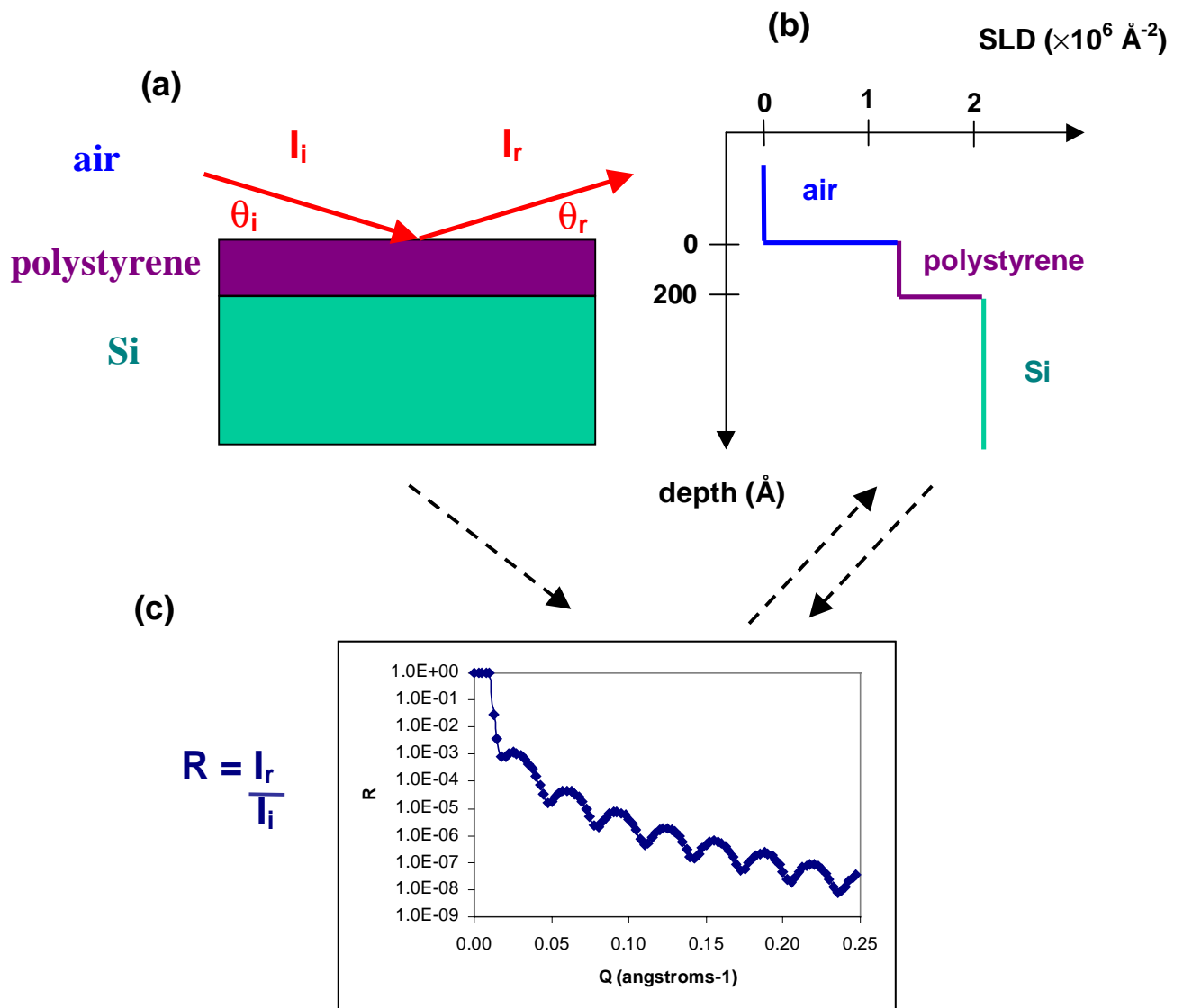
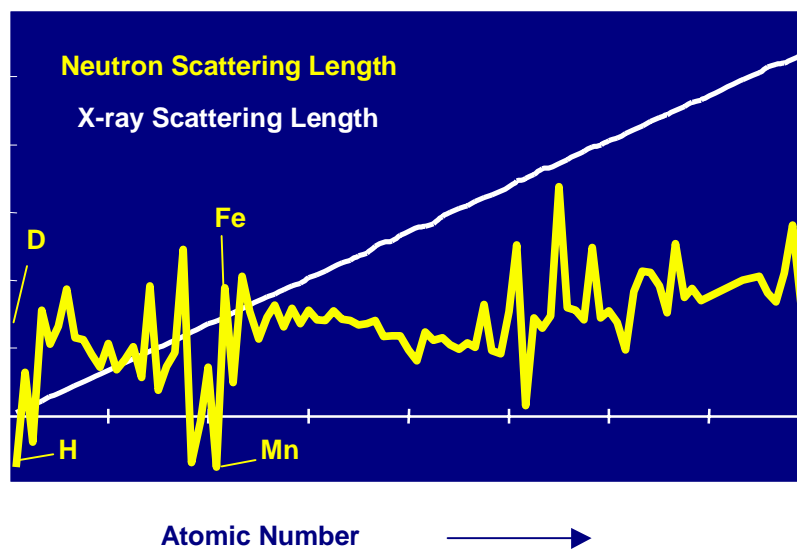


Figure 2.1 (a) A schematic indicating reflection from a 200 Å thick polystyrene film on a Si substrate; (b) A Scattering Length Density Profile for this film; (c) The Simulated Reflectivity Profile calculated from (b).

Uniqueness of the neutron as a structural probe of surfaces and interfaces.

- Neutrons are uncharged and are therefore highly penetrating, which gives them advantages over X-rays for the study of reflectometry in a number of important ways:
  - (i) Neutrons can penetrate into the bulk of samples which enable the study of buried interfaces.
  - (ii) Neutrons can penetrate a number of materials such as silicon, quartz, and sapphire with little attenuation. These materials can act as both substrates for samples and windows for solid/liquid and electrolytic cells. Similarly neutrons can penetrate materials such as aluminium and alumina that are used in the construction of closed-cycle refrigerators, cryostats and furnaces.
- The scattering length of neutrons varies in a random fashion between elements as well as between different isotopes of the same element. On the other hand, X-rays vary monotonically with increasing atomic number. (See Figure 2.2)
  - (i) Neutrons can scatter from adjacent elements such as iron and manganese with high contrast; whereas X-rays show very little contrast.
  - (ii) Neutron scatter differently from hydrogen and deuterium, which enables contrast matching and selective structural labelling in aqueous and soft matter systems.
  - (iii) Light elements (such as hydrogen, carbon, nitrogen and oxygen) may be readily observed in the presence of heavy elements (such as barium, lanthanides and actinides).



**Figure 2.2 Relative scattering powers of neutrons and X-rays.  
(The white line indicates X-ray scattering lengths and the yellow line indicates neutron scattering lengths).**

It is precisely these differences in scattering between X-rays and neutrons that provides complementarity between the techniques. In particular, numerous polymer, surfactant and biological systems rely on the combination of both X-ray and neutron reflectometry measurements in order to solve complex surface and interfacial problems.

- In addition, neutrons have a magnetic moment and polarised neutrons can be produced. This provides a unique tool for the structural study of magnetic and superconducting systems.

Based on a demonstrated need by Australian researchers

**Table 2.2 Expressed scientific interests by workshop participants**

Adsorption Kinetics  
Adsorption of Polymers, Polymer Colloids, Micelles and Surfactants  
Aligned Carbon Nanotube Thin Films  
Biosynthetic-Polymer Composites  
Carbon/Diamond-like films on Surfaces  
Coatings and Adhesion – Paints and Polymers  
Conducting Polymer Thin Films  
Corrosion Studies  
Diffusion Studies Across Interfaces  
DNA modified interfaces and surfaces  
Electro-, Gas and Biological Sensors  
Electrochemical Deposition and Electrokinetics at Solid/Liquid Interfaces  
Ferroelectric and Metal Oxide Thin Films  
Hard Nitride Coatings  
Hydration of Biological Membranes  
In situ Structural Modifications  
Ion implantation in Polymer Films  
Ionic layers at Quartz/Water Interface  
Metal-Ceramic Interfaces  
Molecular Self-Assembly and Templating  
Mono- and Multilayer Adsorption at Air/Liquid Interfaces  
Multilayers for Neutron Optics  
Organic and Inorganic Polymer Films  
Organically Modified Metal Oxides  
Porphyrin-based Photo-voltaic films on Substrates  
Protein Conformations and Kinetics at Liquid Surfaces  
Sol-Gel and ALCVD Thin Films  
Study of Buried Interfaces  
Thin-film Magnetism  
Ultra-thin Multilayers and Integrated Circuit Manufacture  
X-ray and Neutron Mirrors

Based on Overseas Trends at Modern Neutron Research Facilities

- At the NIST Centre for Neutron Research approximately 15% of 420 research proposals carried-out in 2000 were neutron reflectometry based experiments.
- At the ISIS Facility approximately 11% of proposals in 2000 were neutron reflectometry experiments
- At the LLB Facility 150 user days were allocated for each of the EROS and PRISM reflectometers in the second half of 2000. Users requested 300 days on EROS and 200 days on PRISM.

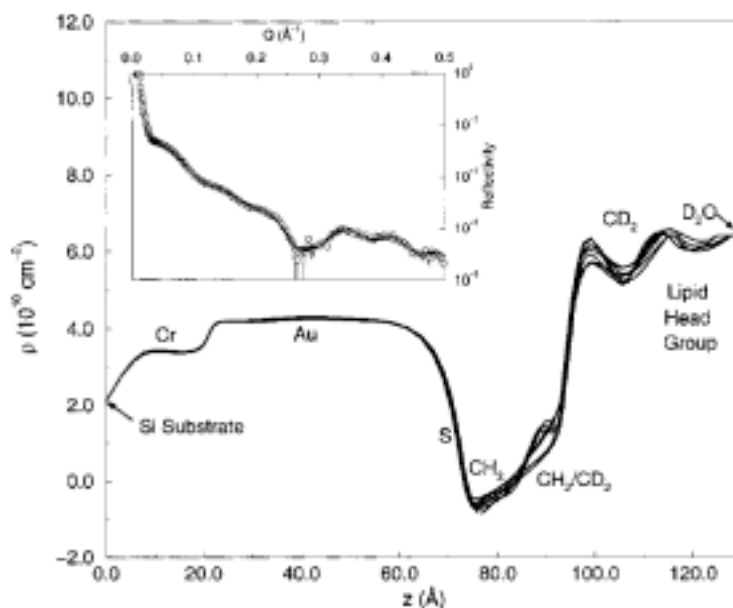


### 3. Recent Developments in Reflectometry Science

#### Model-Independent Data Analysis (C. F. Majkrzak, N. F. Berk, S. Krueger)<sup>2</sup>

Until recently reflectivity profiles have been fitted by comparing data simulated from a model with experimentally observed points. Due to the inability of determining the phase of these data, there might be several different structural models that generate reflectivity profiles that fit the simulated data equally well. Thus the ability to refine structures from reflectometry data has relied heavily on an accurate knowledge of the system under investigation.

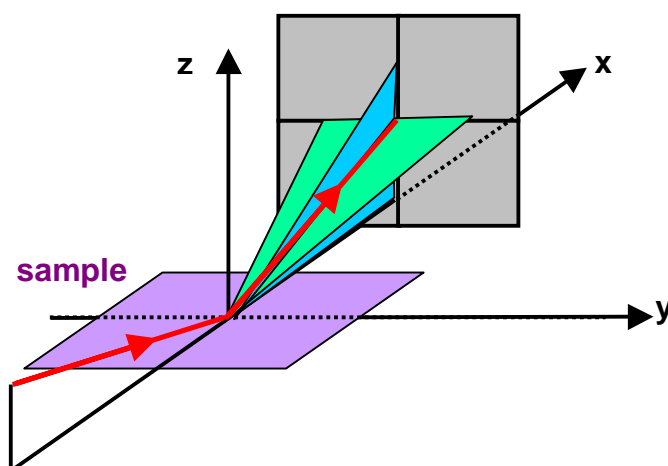
Recent developments in experimental techniques have led to a phase-sensitive data analysis technique that does not assume a structural model, but it still able to produces a family of scattering-length density profiles. The example shown in Figure 3.1 is that of a tethered lipid bilayer in contact with a D<sub>2</sub>O solution containing the peptide toxin melittin.<sup>2,3</sup>



**Figure 3.1** A family of neutron Scattering Length Density profiles obtained from Phase-Sensitive Data Analysis of a tethered lipid bilayer in contact with a D<sub>2</sub>O solution containing the peptide toxin melittin. Each SLD profile represents an equally good fit to the reflectivity data.<sup>2</sup>

#### Off-Specular Reflectometry and Glancing-Angle Diffraction (F.Ott and R. Cubitt)<sup>4</sup>

*Specular reflectometry* (red) measures the intensity reflected for a scattering vector perpendicular to the sample plane (i.e along z). In-plane structures such as surface roughness will give rise to off-specular scattering. Off-specular scattering can occur in the incidence plane (blue) as well as in the plane perpendicular to the incidence plane (green). The latter gives information about structures along the y-direction.<sup>4</sup>

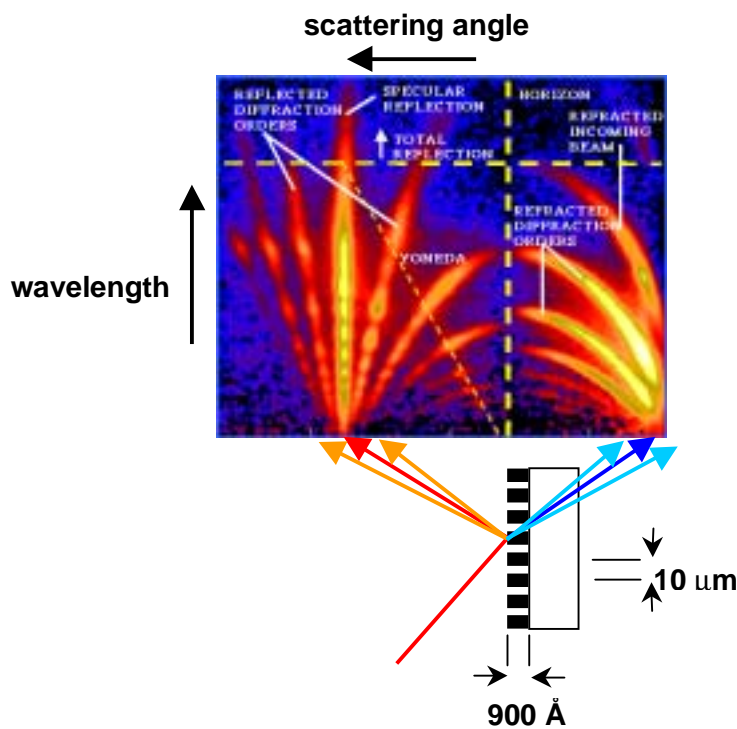


**Figure 3.2** Specular (red) and off-specular (green and blue) scattering.

There are 3 in-plane length scales accessible:

- Off-specular scattering in the incidence plane (blue) 1 - 50  $\mu\text{m}$
- Off-specular scattering perpendicular to the incidence plane (green) 10 - 500 nm
- Surface diffraction at large angles perpendicular to the incidence plane 1 - 5  $\text{\AA}$

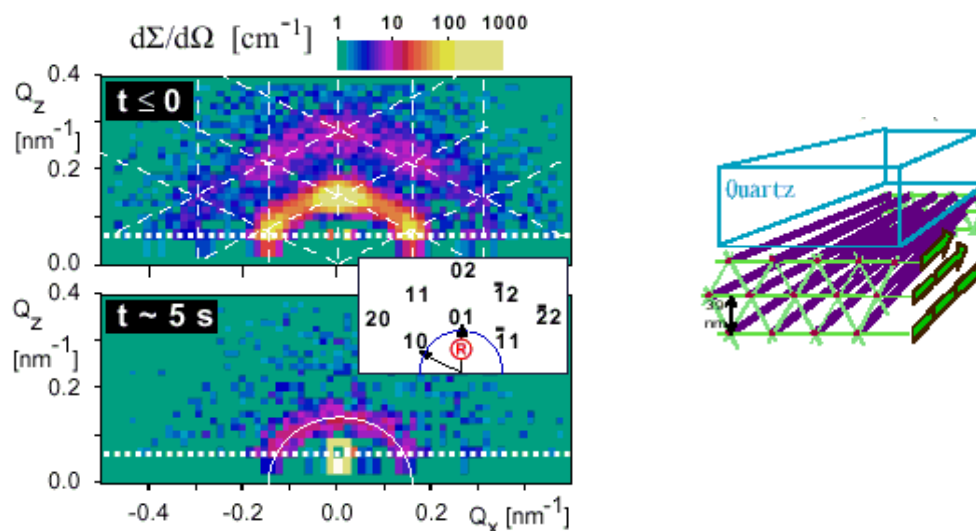
Off-specular scattering can also result from, *low angle diffraction*. Figure 3.3 shows off-specular scattering from a 10  $\mu\text{m}$  nickel diffraction grating. As the incoming beam strikes the grating surface at a glancing angle then many diffraction orders can be seen in both the reflected and transmitted beams at measurable angles of deflection. In addition to information on the stripe separation, the depth profile (consistent with 900 $\text{\AA}$ ) is revealed in the ripples of intensity found running along the specular line and the various reflected diffraction orders. The diagonal line of intensity coming from where the specular line just totally reflects is a Yoneda wing and is a consequence of the roughness along the surface of the nickel strips.<sup>5</sup>



**Figure 3.3** Glancing angle diffraction and off-specular scattering of a 900  $\text{\AA}$  thick Ni diffraction grating on a glass substrate, with 10 $\mu\text{m}$  between strips. The red arrow indicates the reflected beam and the blue arrow the refracted beam. The orange and light blue arrows indicate the diffraction orders of the reflected and transmitted beams respectively (F. Ott, A. Menelle and R. Cubitt).<sup>5</sup>

Near Surface Small Angle Neutron Scattering (W. A. Hamilton)<sup>6</sup>

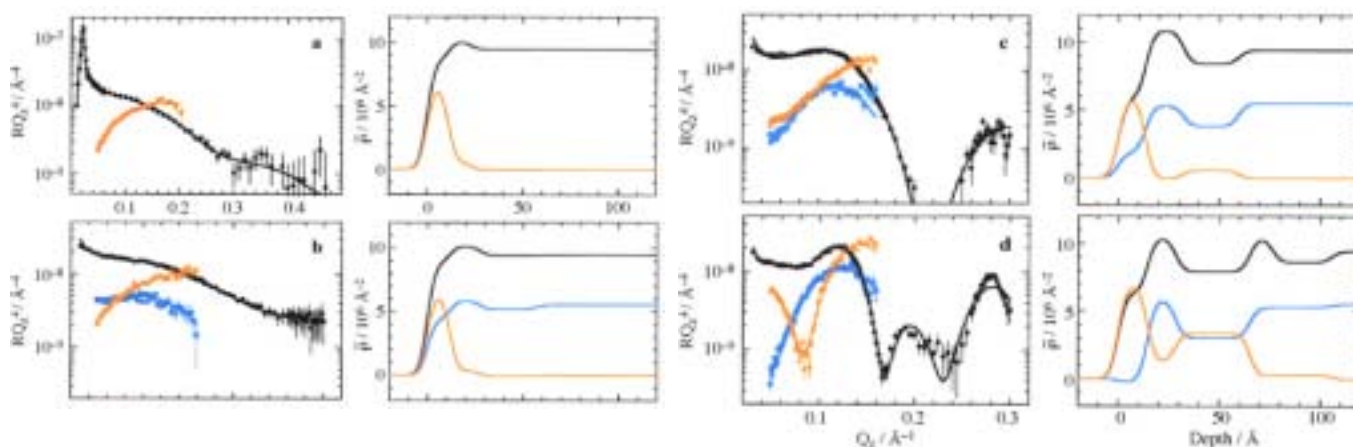
A neutron reflectometer may also be used to examine near-surface small angle scattering. Under shear flow, flexible micelles have been shown to form a 'crystalline' hexagonal surface phase near a quartz interface. Examination of the relaxation processes in this system reveals that the surface order disappears on the time scale of 5 seconds.<sup>6</sup>



**Figure 3.4 Relaxation of a shear induced crystalline surface phase.<sup>6</sup>**

Combined X-ray and Neutron Reflectometry Studies of Complex Interfacial Systems (J. White)<sup>7</sup>

The growth of thin silicate-organic films at the air-water interface of surfactant solutions has been studied *in situ* by x-ray and neutron reflectivity to a resolution of  $\sim 5 \text{ \AA}$ . Surfactant in the solution and the air-water interface itself are involved in directing the growth and final structure of the films. *In situ* x-ray and neutron reflectivity measurements at an early stage of film growth show a slow development of structure in the top 100  $\text{\AA}$  of the solution which is consistent with a monolayer of tilted surfactant molecules at the air-water interface, a layer of partly silicated material and an interdigitated surfactant bilayer or layer of cylindrical micelles oriented with their long axes parallel to the surface. Following this induction period a rapid crystallisation occurs to give a structure with a crystallographic repeat distance of 45  $\text{\AA}$  perpendicular to the surface and composed of alternating layers of mainly surfactant, and then mainly silicate material. The very narrow diffraction peaks observed indicate that the final silicate film is highly ordered.<sup>7</sup>



**Figure 3.5.  $RQ_z^4$  vs  $Q_z$  (left) and model SLD profiles (right) for (a) surfactant-only solutions. (b),(c) and (d) surfactant-silicate solutions after 25%, 75% and 90% of the time required for diffraction peaks to appear. Black lines refer to  $h_{33}\text{-C}_{16}\text{TAC}$  in  $\text{H}_2\text{O}$  x-ray data, orange lines to  $d_{33}\text{-C}_{16}\text{TAB}$  in ACW neutron data, and blue lines to  $h_{33}\text{-C}_{16}\text{TAC}$  in  $\text{D}_2\text{O}$  neutron data.<sup>7</sup>**

## 4. Aspects of Instrument Design

Choice of General Instrument Type – (Presented by Dr John Ankner and discussed by participants).

There are two choices of sample orientation (horizontal or vertical) and two incident beam types (monochromatic or broad bandwidth). The following table discusses the strengths and weaknesses of each combination.

**Table 4.1 Instrument Type Matrix**

<p><b>Horizontal Sample / Monochromatic</b> (e.g. V6 – BENSC; NG-7 – NIST)</p> <p><i>Strengths</i></p> <ul style="list-style-type: none"> <li>• Both liquid and solid surfaces</li> <li>• Polarisation is straight forward</li> </ul> <p><i>Weaknesses</i></p> <ul style="list-style-type: none"> <li>• Crystal monochromator may limit Q-range</li> <li>• Large cryostats &amp; magnets are non-standard</li> <li>• Long <math>\lambda</math> contamination if using multilayer monochromators</li> <li>• Kinetic studies are difficult</li> </ul>	<p><b>Vertical Sample / Monochromatic</b> (e.g. NG-1 – NIST; ADAM – ILL, PRISM - LLB)</p> <p><i>Strengths</i></p> <ul style="list-style-type: none"> <li>• Optimised for solid and solid/liquid surfaces</li> <li>• Polarisation is straight forward</li> <li>• Simplest and cheap to build</li> <li>• Can easily accommodate large cryostats &amp; magnets</li> </ul> <p><i>Weaknesses</i></p> <ul style="list-style-type: none"> <li>• Air/liquid studies not possible</li> <li>• Large cryostats &amp; magnets are non-standard</li> <li>• Long <math>\lambda</math> contamination if using multilayer monochromators</li> <li>• Kinetic studies are difficult</li> </ul>
<p><b>Horizontal Sample / Time-of-flight</b> (e.g. AMOR – SINQ, EROS, LLB)</p> <p><i>Strengths</i></p> <ul style="list-style-type: none"> <li>• Both liquid and solid surfaces</li> <li>• Well suited to kinetics studies</li> <li>• Well suited to off-specular studies</li> </ul> <p><i>Weaknesses</i></p> <ul style="list-style-type: none"> <li>• Polarisation is more difficult than monochromatic</li> <li>• Large cryostats &amp; magnets are non-standard</li> <li>• Requires an end-of-guide position</li> </ul>	<p><b>Vertical Sample / Time-of-flight</b> (e.g. D17 – ILL)</p> <p><i>Strengths</i></p> <ul style="list-style-type: none"> <li>• Optimised for solid and solid/liquid surfaces</li> <li>• Can easily accommodate large cryostats &amp; magnets</li> <li>• Well suited to kinetics studies</li> <li>• Well suited to off-specular studies</li> </ul> <p><i>Weaknesses</i></p> <ul style="list-style-type: none"> <li>• Air/liquid studies not possible</li> <li>• Polarisation is more difficult than monochromatic</li> <li>• Requires an end-of-guide position</li> </ul>

The Workshop participants concluded that the neutron reflectometer must be able to study the following types of systems:

- ‘free-liquid’ samples,
- solid/liquid and air/solid interfaces (including ‘ultra-thin films’),
- kinetic experiments and
- ‘off-specular’ measurements.

A few workshop participants viewed polarisation measurements as important; although this was not reflected in the interests of the majority of participants.

**Examination of the above matrix leads to the conclusion that the best instrument to suit this community’s needs is a horizontal sample (vertical scattering-plane) / time-of-flight neutron reflectometer with a 2-dimensional area detector for ‘off-specular’ measurements.**

## 5. Requested Performance Specifications

Section 4 of this Report identified the scientific needs that must be met by this instrument. Given this basis, the instrument will require the following capabilities to meet these needs:

- (i) In order to probe length scales from a few Å to hundreds of nanometres, a high flux of long wavelength neutrons ( $> 3 \text{ Å}$ ) will be required as the appropriate probe. To achieve this, the instrument will have to be **located on a cold neutron guide** at the Replacement Research Reactor.
- (ii) In order to study 'free liquids', solid/liquid and air/solid systems, the instrument will require a **vertical scattering geometry**. It must be possible to adjust the angle of incidence of the neutron beam onto the sample. A *high precision 2-circle goniometer* will also be required for the alignment of and variation of incident angles with respect to solid/liquid and air/solid samples.
- (iii) A further requirement for the study of free liquids is that the **instrument has adequate vibration isolation**. Without adequate vibration damping (particularly at the sample position) capillary waves on the liquid's surface will ruin the experiment. To help achieve this, *the sample position should not be mechanically coupled to other parts of the instrument and the entire instrument should be based upon an adequate vibration isolation base such as granite*.
- (iv) In order to conduct kinetic experiments high neutron flux is required. This may be achieved by using a broadband wavelength spectrum and the **time-of-flight** method. To facilitate this, the instrument will require an *End-of-Guide position* and a flight-path of at-least 15m from the guide. A time-of-flight instrument will require a **double disc chopper system** in order to convert the continuous wavelength spectrum into a pulsed beam.
- (v) In order to optimise the study of a range of samples from free liquids to ultra-thin solid films the instrument should operate with **a range of different resolutions available**. Free liquids and ultra-thin solid films only require low resolution; while high resolution is needed to study thicker layers. The ability to vary the resolution of the instrument to suit a given experiment will be crucial in order to allow the maximum incident flux onto the sample. Variable resolution may be achieved by changing the *phasing and separation between discs in a double disc chopper*.
- (vi) In order to conduct 'off-specular' measurements the instrument *should have a wide-angle, position sensitive, 2-dimensional area detector that can be moved in the vertical direction*. *High-speed detector electronics are recommended* to avoid detector saturation and also enable the adequate binning of time-of-flight events. The area detector should have *high resolution position sensitivity* ( $\sim 2 \text{ mm}$ ) in the vertical scattering plane. The type of area detector recommended to achieve these parameters is a  *$^3\text{He}$  wire detector with delay-line electronics*.
- (vii) In order to obtain low minimum reflectivities ( $R_{\min} \sim 10^{-8}$ ) the instrument must have a **low background radiation environment**, particularly at the sample and detector positions. In order to achieve this, the instrument should be:
  - located in the guide hall and as far away from monochromator, velocity selector or sample positions of neighbouring instruments as possible
  - built off the axis of the neutron guide so as to minimise fast neutrons and  $\gamma$ -rays
  - heavily shielded from the disc chopper system

A number of basic instrument specifications are given in Table 5.1.

**Table 5.1 Instrument Specifications**

<b>Instrumental Parameter</b>	
Scattering Plane	vertical
Wavelength Range (tof) ( $\text{\AA}$ ) (to be determined)	3 – 15 or (2 - 25)
Q-range ( $\text{\AA}^{-1}$ ) (liquids)	0.005- 0.5
(solids)	0.005 – 2.2
$\Delta t/T$ ( $\Delta\lambda/\lambda$ )	1 - 10 %
$\Delta\theta$	0.02 – 0.06°
$\Delta Q/Q$	2 – 20%
$R_{\min}$	$10^{-8}$
Off-Specular Scattering	Yes
Polarised Reflectometry	not in initial design
Minimum Feature Resolution	< 10 $\text{\AA}$
Minimum Data Acquisition Time	1 min
Typical Data Acquisition Time	4-5 hr
White Flux at Sample ( $\text{ncm}^{-2}\text{s}^{-1}$ )	$\sim 10^9$
Sample Size	> 1 $\text{cm}^2$
Sample Thickness	< 2000 $\text{\AA}$
Beam Size	(0.05 – 20 mm) x 50 mm
Flight Path (collimation system)	~ 3m
(sample – detector)	2 – 4 m
Detector Type	$^3\text{He}$ wire – Delay Line
Detector Area	250 mm x 500 mm
Detector Resolution (In Reflection Plane)	2 mm
(Perpendicular to Reflection Plane)	5 mm
Detector Count Rate	3 MHz
Time Bin Width	100 $\mu\text{sec}$
Background Count Rate (shutter closed)	< 1 count/s over whole detector

## 6. Preliminary Outline of Possible Instrument

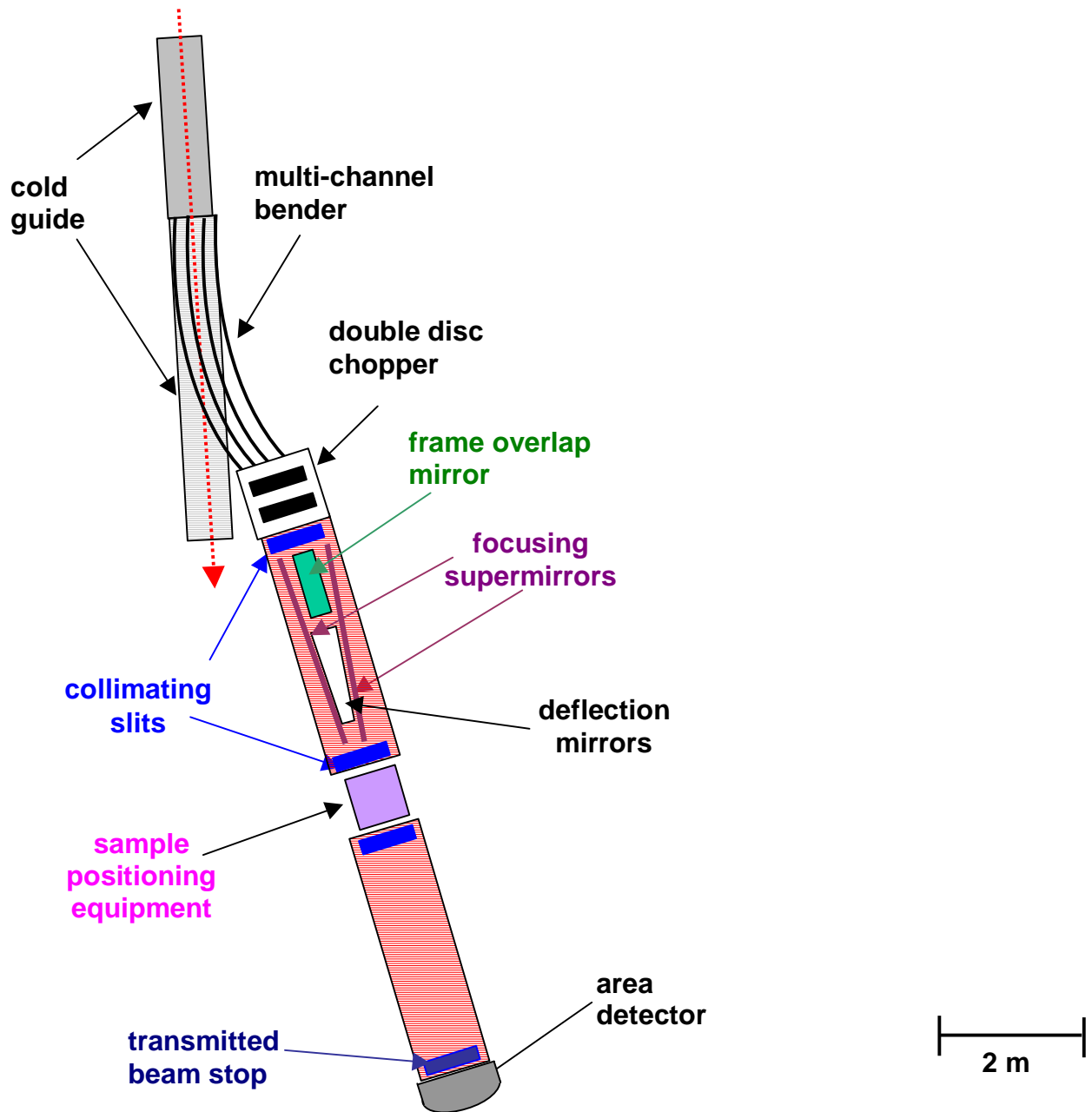


Figure 6.1 Schematic of Reflectometer showing major components.

### Major Instrument Components

#### Multi-channel bender (~5-8 m)

A time-of-flight instrument will require an end of guide position. Should the end of CG3 be unavailable due to issues of timing or design requirements of other instruments, an alternative may be to bend a portion of the beam away from the line of the guide using a bending device.

### Double Disc Chopper (~1.5 m)

In order to provide the reflectometer with a flexible resolution, a double disc chopper system would require variable phasing and variable disc separation. Such a system will require substantial shielding to reduce the instrumental background.

### Collimation System (~ 3 m)

To be housed in an evacuated vessel, along with the following components:

- *Beam defining slits* at either end (typical separation ~4m)
- A *Frame Overlap Mirror* to remove slow, long  $\lambda$  neutrons from one pulse interfering with the fast, short  $\lambda$  neutrons from the next (Cut-off at 15, 20 or 25 Å ?).
- *Focusing Supermirrors* would focus the beam from 50 mm to ~20-30 mm in order to increase the beam intensity.
- Deflection mirrors would be required to direct the beam down onto the surface of liquid samples at 2 or 3 different incident angles.
- Substantial shielding will be required to capture divergent neutrons as well as those removed by the Frame Overlap Mirror. This will add significant weight to the collimation system.

### Sample Positioning Equipment (~0.5 m)

Should not be mechanically linked to other parts of the reflectometer in order to minimise vibrations. Sample positioning equipment should include:

- A *z-stage* to change the height of samples with respect to the incident beam (0.01 mm resolution)
- A 2-circle goniometer to align solid and solid/liquid samples with respect to the beam (0.001° resolution). For solid samples to reach  $Q > 0.5 \text{ \AA}^{-1}$  (up to  $Q \sim 2.2^{-1}$  for Si(111) Bragg peak).
- A rotation stage and a translation stage to move different samples into position (~1 mm resolution)
- An anti-vibration stage for the mounting of free liquid troughs.

### Post-Sample Flight Path (Up to 4 m)

Should be evacuated to minimise air scattering. A slit should immediately follow the sample with a beam stop prior to the detector to reduce the intensity of the non-reflected beam. This flight path should be as short as possible in order to reduce the size of the detector and the effect of gravity on the neutrons. It should be large enough to provide adequate resolution in both the horizontal and vertical directions.

### 2-Dimensional Area Detector (~ 250 mm × 500 mm)

Size is defined by the focusing system and the sample – detector distance

- The detector will require substantial shielding to reduce background counts.
- The detector will have to be of sufficient area to accommodate the measurement of off-specular scattering ( $4^\circ$ ), as well as the horizontal divergence.
- The detector should be able to be raised vertically in order to count the specular signal from solid and solid/liquid samples at very high  $Q$  ( $> 0.5 \text{ \AA}^{-1}$ ), as well as to access off-specular scattering in the plane of incidence.
- Provision may be made to allow the detector to move horizontally in an arc about the sample position. At modest angles ( $< 20^\circ$ ), off-specular scattering perpendicular to the plane of incidence may be obtained. In order to achieve this the horizontal divergence will need to be reduced (losing the flux gains obtained by focusing).
- A back-up single  $^3\text{He}$  tube should be available for specular scattering measurements if the area detector is out of commission.

### Length of Instrument (Estimated total length of instrument - ~ 15 m)



## 7. Calculations to Optimise Design Characteristics

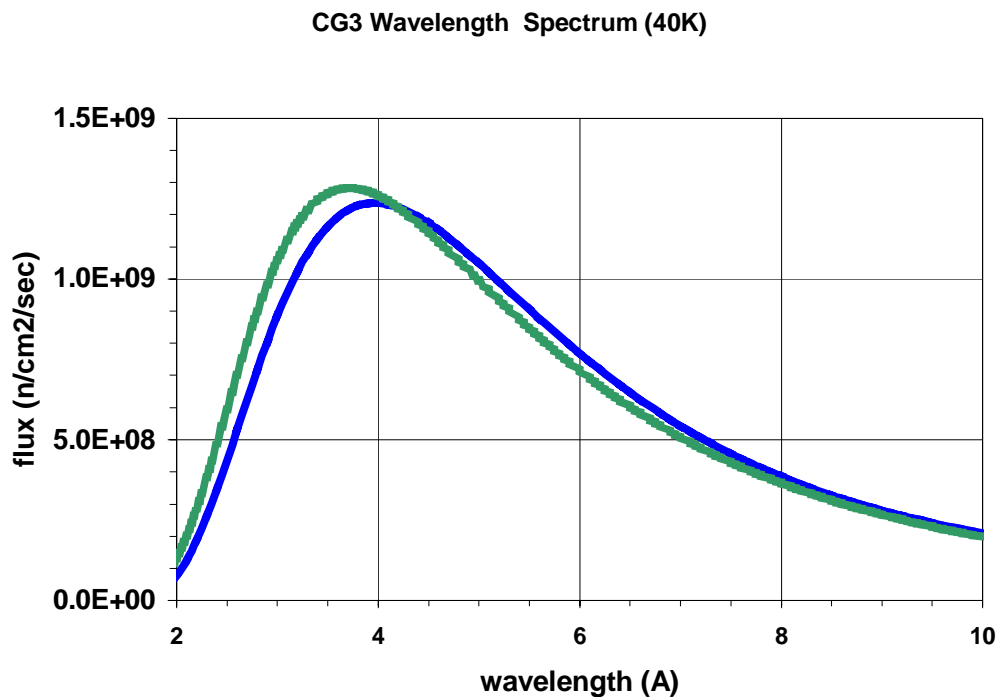
### (i) Source profile from neutron guide

This calculation is required as the foundation of a number of other calculations.

#### *Our current profile estimates*

- White beam flux:  $\sim 6 \times 10^9$  n/cm<sup>2</sup>/s
- ( $\lambda = 3.9$  Å) In divergence range: Horizontal 1.5°, Vertical 2.4°:
- Beam height at guide 1.56 m
- Beam profile in guide 200 mm high, 50 mm wide.

Figure 7.1 indicates current estimates for the neutron flux in CG3 with the above divergences. The actual useable flux in the instrument will be lower and shifted to shorter wavelengths due to much lower vertical collimation.



**Figure 7.1 Estimated Cold Neutron Spectrum in Cold Guide 3 at point where the reflectometer might be situated. Both curves were calculated for guides with  $m=3$  supermirrors on top and bottom. The blue curve has  $m=2$  on both vertical sides, while the green curve has  $m=2.5$  on the inner side and  $m=1.5$  on the outer side. The expected spectrum should be a combination of both curves.<sup>8</sup>**

#### *Calculation Outputs:*

- Intensity vs Position in the Guide
- Intensity vs Bandwidth in the Guide
- Bandwidth vs Position in the Guide
- Horizontal and Angular Divergences
- Will want to account for effect of gravity on long  $\lambda$  neutrons.

(ii) Experimental Time for Time-of-Flight vs Monochromatic Instruments.

Using the Source Profile calculations, we will calculate the efficiency of running experiments in either time-of-flight or monochromatic modes for a range of different sample types. These calculations should account for variation in resolution due to the monochromator and chopper systems.

- It is difficult to achieve the low resolution ( $> 10\%$ ) necessary for air/liquid systems using a monochromator.
- Even if monochromatic measurement is faster, the sample may change between the measurement of  $Q_{\min}$  &  $Q_{\max}$ . Tof has advantage of averaging such changes across the whole pattern.

(iii) Instruments Location on Guide to Provide Low Background

Detector must avoid line of sight to avoid fast neutrons and  $\gamma$ -rays

Detector must be placed well away from monochromators and velocity selectors of other instruments.

*Calculation Outputs:*

- Estimate of background in the detector and impact on  $R_{\min}$ .

(iv) Beam Transport Through a Bender System

- Source profile estimates will be used to design and simulate a bender system to deflect the beam away from the guide path and away from the centre of the guide hall.
- The supermirror type used ( $m = 2$  or  $3$ ) will depend on the low  $\lambda$  neutron cut-off ( $2 - 3 \text{ \AA}$ ).  $m=3$  supermirror will be required on the top and bottom of the bender.

*Calculation Outputs:*

- Single or Multi-Channel (will depend on space available)  
(Multi-channel may give a more homogenous beam)
- Bender material (100 % glass to reduce activation and  $\gamma$  background)
- Transmission through bender (as a function of  $\lambda$ )
- Optimum number of channels and dimension of channels
- Radius of Curvature, length and transverse separation from main guide
- Divergences at end of bender
- Efficiency at peak flux
- Beam profile at end of bender
- Shielding requirements
- Option to deflect beam downwards (up to  $\sim 7^\circ$ )
- Option to include focusing (from 50 mm width to 25-30 mm)

(v) Resolution and Design of Double Disc Chopper System

Source profile estimates will be used to design and simulate a double-disc chopper system.  
(Direct enquires to Delft Group or Astrium)

*Basic chopper requirements:*

- Variable distance between blades for variable resolution:  
( $10\% \Delta\lambda/\lambda$ , will need  $\sim 1.2$  m between blades for a chopper – sample distance of  $\sim 4$  m)
- Variable phasing (with at least  $0.1^\circ$  phasing accuracy)
- The phase of the chopper needs to be changed for different incident angles. (Time to re-phase  $\sim$  a couple of minutes)
- Radius of chopper (will need to be  $\sim 5x$  the width of the beam -  $\sim 50 - 60$  mm)
- Typical sector angle required  $< 10^\circ$
- Vibration isolation required for blades, motor and vacuum equipment

- Chopper speeds of up to ~2000 r.p.m.
- Separate vacuum for chopper system and collimation system

*Calculation Outputs:*

- Effect of different disc materials on blocking the beam & gamma production.  
Gd discs vs B+epoxy
- Radii of discs and size of window
- Transmission factors and transmitted bandwidth
- Effect of variation in speed and phasing of discs
- Effect on resolution due to variation in spacing between discs
- Benefits of operating in vacuum / What pressure is required ?
- Vibrations produced by motors, discs and linkages
- Shielding requirements

(vi) Length, Position and Composition of Frame Overlap Mirror

Depends on cut-off  $\lambda$  and beam height

Top & bottom of guide surrounding Frame Overlap Mirror should be covered with B<sub>4</sub>C or Boroflex to capture scattered or divergent neutrons

*Calculation Outputs:*

- Location between collimating slits
- Surface Properties
- Orientation with respect to beam
- Transmission factors
- Impact of one mirror, or two mirrors in a wedge shape

(vii) Deflection Mirrors and Q-range

An option is to have deflection mirrors located inside the collimation system between the beam defining slits in order to deflect the beam downwards up to ~7°.

If wavelength band is 3 – 15 Å, **3 different angles** will be required for **Q of 0.005 – 0.5 Å<sup>-1</sup>**.

$\theta_1 = 0.34^\circ$	Q: 0.005 – 0.025 Å <sup>-1</sup>
$\theta_2 = 1.44^\circ$	Q: 0.021 – 0.105 Å <sup>-1</sup>
$\theta_3 = 6.86^\circ$	Q: 0.1 – 0.5 Å <sup>-1</sup>

Based on wavelength band of 2 – 25 Å

$\theta_1 = 0.56^\circ$	Q: 0.005 – 0.061 Å <sup>-1</sup> .
$\theta_2 = 4.56^\circ$	Q: 0.040 – 0.500 Å <sup>-1</sup> .

*Calculation Outputs:*

- Ability to cover Q range of 0.005 – 0.5 Å<sup>-1</sup> for a given source spectrum
- Estimate of the time to conduct an experiment based on 2 or 3 angle changes.
- Impact of deflection mirrors on beam spectrum (efficiency, divergence)
- Length of deflection mirrors
- Shielding requirements
- Option to include focusing (from 50 mm width to 20-30 mm)

(viii) Effect of Horizontal Focusing (from 50 mm to 20 - 30 mm)

Calculation Outputs:

- Optimum location of focusing elements (Integration into bender or between collimation slits with FOM)
- Optimum size and type of focusing guide ( $m \geq 3$  supermirror guide)
- Gains in intensity versus increases in horizontal divergence.
- Detector size necessary to capture increased divergence

(ix) Flight Path Between Sample and Detector

- What is the optimum sample – detector distance ? (This will depend on resolution requirements, amount of focusing and the size and resolution of the detector).
- Should the instrument have a variable sample – detector distance ?
- Should the flight path be lined with supermirrors to capture any horizontal divergence losses ? (Recommended but not for *off-specular scattering* - would need to be removable)

(ix) Detector Type and Optimisation of Design

- Detector type to investigate:
  - Microstrip – possibly too small (currently ~150 x 150 mm in size)
  - $^3\text{He}$  using charge-division electronics – slow (but with good  $\gamma$  discrimination)
  - $^3\text{He}$  using 'delay-line' electronics.
- Require sufficient size to capture all of horizontal divergence for specular scattering  
Detector will have to be larger if strumpet is used to focus incident beam.
- Require sufficient size to capture off-specular signals.  
In the horizontal plane should require ~6-7°.
- Detector Size 250 mm x 500 mm ?
- Advantages to counting the background over large solid angles
- Will need to move the detector vertically to catch high Q reflectivity for solid samples.
- On-board high speed pulse recognition electronics ('Overbin' in time 100  $\mu\text{s}$ )
- $\lambda$  calibration is vital for a tof system
- Need to be able to measure direct beam on detector. If is too high attenuate.  
This is vital in order to be able to correct scaling of data.

Calculation Outputs:

- Detector size
- Detector Resolution (eg. 2 mm in vertical and 5 mm in horizontal dimension)
- Optimum  $^3\text{He}$  pressure (depends on the wavelengths used in the instrument).
- Width and type of detector window (Single or double pressure windows).  
(10 mm Al windows can give a high background & will affect off-specular measurements)

## 8. Summary

The workshop participants came to the following conclusions:

1. That the neutron reflectometer be capable of studying “free liquid” samples as well as air/solid and solid/liquid interfaces. The implication from this conclusion is that this reflectometer must operate with a *vertical scattering plane*.
2. That the neutron reflectometer be capable of measuring the kinetics of a range of systems. The implication from this conclusion is that the reflectometer must operate in a ‘broadband’ wavelength mode. The instrument will have to operate using time-of-flight methods and will have to be located at the end-of-guide position. The pulsed neutron beam required for time-of-flight operation will be generated by a disc chopper system.
3. That the neutron reflectometer has flexible resolution in order to study a wide range of structural features. This may be achieved using a double disc chopper with variable phasing and variable disc separation.
4. That the neutron reflectometer be capable of measuring “off-specular” scattering; an area that was viewed by the participants as growing in popularity. The implication from this conclusion is that this reflectometer must have a suitable wide-angle area detection system.
5. There was little demand for polarised neutron reflectometry from the participants at this workshop.

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## Appendix A - Workshop Attendees and Interested Persons

### Workshop Attendees

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## **Appendix B - Invited Presentations**

Dr Robert Robinson (ANSTO)

- *An Overview of Instrument Opportunities at the Australian Replacement Research Reactor*

Dr Ian Gentle (University of Queensland)

- *An Introduction to Reflectometry*

Dr John Ankner (Oak Ridge National Laboratory, USA)

- *Scientific Opportunities for Neutron Reflectometry* and  
- *Design Considerations for Reactor-Based Reflectometers*

Dr Frederic Ott (LLB, France)

- *Scientific Opportunities for Neutron Reflectometry II*

Dr Michael James (ANSTO)

- *Design Consideration for the RRR Neutron Reflectometer*

Professor John White (Australian National University)

- *The Study of Molecular Self-Assembly using Neutron and X-ray Reflectometry*

Dr Robert Cubitt – (ILL, France)

- *The D17 Reflectometer at ILL*

Dr Christophe Barbe (ANSTO)

- *Thin Ceramic Films at ANSTO*

Dr Shane Kennedy (ANSTO)

- *Neutron Guides and the Cold Neutron Source at the RRR*

## Appendix C: Neutron Reflectometers at Reactor Sources Worldwide

Institute	NIST	NIST	ILL	ILL	LLB	LLB	SINQ	ORNL	BENSC
Name	NG-1	NG-7	ADAM	D17	PRISM	EROS	AMOR	MIRROR	V6
Webpage	a	a	b	c	d	d	e	f	g
Guide	cold	cold	cold	cold	cold	cold	cold	cold	cold
Geometry <sup>1</sup>	H	V	H	H	H	H	V	H	V
Mono	PG	PG	PG	Multi ToF	Multi	ToF	Multi ToF	PG	PG
$\lambda$ (Å)	4.1	4.1	4.4	6.0	4.0			5.0	4.8
$\Delta\lambda$ (Å)				3-25		3.5-22	1.3-13		
$Q_{max}$ (Å <sup>-1</sup> )		0.4	5.4	2	2.7		0.7	1.0	0.54
(liquids)		0.4							0.11
$R_{min}$	10 <sup>-8</sup>	10 <sup>-6</sup>	10 <sup>-8</sup>	10 <sup>-8</sup>	5x10 <sup>-6</sup>	5x10 <sup>-6</sup>		10 <sup>-8</sup>	2x10 <sup>-5</sup>
Flux (ncm <sup>-2</sup> s <sup>-1</sup> )	1.3x10 <sup>4</sup>		2x10 <sup>6</sup>	10 <sup>10</sup>	5x10 <sup>5</sup>		1.3x10 <sup>5</sup>	10 <sup>6</sup>	3x10 <sup>4</sup>
Detector	<sup>3</sup> He	<sup>3</sup> He	19x19	25x50	<sup>3</sup> He	<sup>3</sup> He	17x19	5x10	48- <sup>3</sup> He
Resolution (mm)				2					
Polarisation	Y		Y	Y	Y		Y		Y

<sup>1</sup> Geometry Refers to Scattering Plane, not sample orientation.

V = vertical scattering plane for horizontal samples such as 'free liquids'

H = horizontal scattering plane for vertical samples

a <http://rrdjazz.nist.gov/>

b <http://www.ill.fr/YellowBook/ADAM/>

c <http://www.ill.fr/YellowBook/D17/>

d [http://www-llb.cea.fr/index\\_e.html](http://www-llb.cea.fr/index_e.html)

e <http://sinq.web.psi.ch/sinq/instr/amor.html>

f <http://neutrons.ornl.gov/NSatHFIR/HFIRNSFac.HTML>

g <http://www.hmi.de/bensc/instrumentation/instrumente/v6/v6.html>

## Appendix D - Estimates of Component Costs and List of Suppliers

These are estimates only. Accurate estimates will depend on the result of component simulations, detailed design considerations and quotations from suppliers.

**Table D.1**

<b>Component</b>	<b>Possible Procurement Options</b>	<b>Cost (AUS\$)</b>
Multi-channel bender to gain free space ~5m, 3 channels, m=3 supermirror	In-house conceptual design Specify performance-Contract out <i>Swiss Neutronics, Cilas, Mirrortron</i>	500,000
Frame Overlap Mirror	< 25 Å cut-off	10,000
Double Disc Chopper Variable phase Variable Separation (to 1m) ~2000 rpm	Motors, discs, control system, vacuum housing, shielding, power supplies, labour <i>Austrim, Mirrortron?</i>	450,000
2 D Area Detector + Electronics On-board high speed pulse recognition system	<i>EMBL, Risø, ORNL</i> Care should be taken with detector design	500,000
Detector Movement System ~1.5 m travel, 0.1mm steps		100,000
Slit System (x3) + <sup>6</sup> Li Beam Stop	<i>Newport, SNI, Kozhu, MTF</i>	100,000
Sample Stages Liquids: z-stage, vibration isolation Solids: 2-circle goniometer Rotation and Translation stages	<i>Huber, SNI, JJ X-ray</i> Encoders, motors, electronics, software	200,000
Deflection Mirrors (x2) m=3 supermirrors, ~60 cm long (trapezoidal if focusing is used)	<i>Swiss Neutronics, Mirrortron, Cilas</i> Encoders, motors, electronics, software	80,000
Horizontal Focusing m=3 supermirrors, 6 m	<i>Swiss Neutronics, Mirrortron, Cilas</i>	25,000
Vacuum System for Flight Path 5 single crystal windows	Lined with B <sub>4</sub> C plates to stop vertical divergence ( <i>Eagle-Picher</i> )	100,000
Cabling for ~25 motors		50,000
Control Cards & Computers		65,000
Shielding Concrete, lead, B <sub>4</sub> C, B-polyethylene, <sup>6</sup> Li, Boroflex	(B-PE) <i>King Plastic Corp.</i> ; (Boroflex) <i>AZ-Systemes</i>	200,000
<b>Total</b>		<b>2,400,000</b>

**Table D.2 Ancillaries Requested by Workshop Participants**

It is not envisaged that ANSTO or the Neutron Reflectometry Project will fund all of the ancillary equipment listed below. Demand for these and other ancillaries will have to be assessed and balanced with budgetary constraints due to the reflectometer itself. Some of these items may be provided by users for minimal cost, while others may be acquired using funds from competitive government grants. Further discussion will be necessary with the User community to prioritise, resource and acquire ancillary devices for the neutron reflectometer.

<b>Ancillary</b>	<b>Cost Estimate</b>
<b><i>X-ray Reflectometer<sup>a</sup></i></b>	<b><i>~\$200,000</i></b>
Furnace (up to 1000 C)	
Gas extraction for toxic samples	
Controlled atmospheres (gas types and humidity)	
Sample preparation facility – Laminar flow cabinet	
High pressure facility (pressure not specified)	
Closed cycle refrigerator (down to 5 K)	
Langmuir-Blodgett film balance	\$20,000
Anti-vibration stage for liquids	\$15,000
Hydrothermal facilities	
Thermostatic Bath	
Temperature Controlled Cell	
Solid/Liquid cell (with flow-through facility to change liquid)	
Electrochemical cell	
Shear Cell	
Space for a 4 x 4 sample changer	
Brewster Angle Microscope (for simultaneous measurement)	

<sup>a</sup> A number of X-ray reflectometry facilities currently exist in Australia, including those at:

- the Research School of Chemistry, Australian National University;
- the Department of Applied Physics, University of Technology Sydney; and
- one soon to be installed at the School of Chemistry, University of Queensland.

ANSTO does not currently have the facility to conduct X-ray reflectometry experiments.

Options to access X-ray reflectometry facilities include:

- Use of the above facilities (if still available)
- Upgrade of existing ANSTO X-ray powder facilities to allow reflectometry
- Purchase of a dedicated X-ray reflectometry instrument to be sited at ANSTO

**Table D.3 Other Facilities Requested by Workshop Participants**

Computer controlled slits
Real time data display
Multiple computers: Data acquisition and data analysis

## Appendix E - Instrument Design and Experimental Capability.

(Presented by Dr Michael James and discussed by participants).

### Choice of incident wavelength spectrum

#### (a) Access to critical edges

The critical angle below which total external reflection occurs is given by the expression:

$$\theta_c \sim \lambda(\mathbf{Nb}/\pi)^{1/2}$$

where  $\lambda$  (Å) is the neutron wavelength and  $\mathbf{Nb}$  is the Scattering Length Density (SLD) (Å<sup>-2</sup>).

For  $\lambda = 4$  Å,  $\theta_c$  is typically between 0.1° and 0.4° for many materials.

(It should be noted that materials such as H<sub>2</sub>O and Ti have negative SLD's and do not display a critical edge).

#### (b) Cold or Thermal Guide ? - (Relative intensities of long $\lambda$ neutrons)

At  $\lambda = 4$  Å we expect  $\sim 5.4 \times 10^9$  n/cm<sup>2</sup>/s in the cold guide (at 25 K) and  
 $\sim 2.9 \times 10^8$  n/cm<sup>2</sup>/s in the thermal guide (at 323 K)

By using a cold guide we expect approximately **19 times** the flux of 4 Å neutrons than is available in a thermal guide.

#### (c) Broadband spectrum available to the instrument ?

The spectrum available to the instrument will affect key aspects of its design. The broadband flux available on a cold guide may be between wavelengths of 2 Å and 25 Å.

A frame overlap mirror filters the long wavelength neutrons of a pulse so that they do not contaminate the signal of the following pulse. The detailed design of a frame overlap mirror will depend on the quantity of long wavelength neutrons in the broadband spectrum.

Key aspects of the incident beam optics such as a multi-channel bender, focusing optics or deflector mirrors will depend on the quantity of short wavelength neutrons.

#### (d) The effect of gravity.

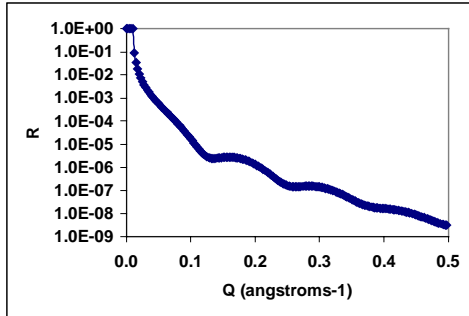
Neutrons fall under the effect of gravity, especially long wavelength neutrons. As the instrument will be built with a vertical scattering plane and given that a broadband spectrum is envisaged, the effect of gravity must be accounted for in the calibration of the detector. Simulations will indicate the magnitude of this effect on the longest  $\lambda$  neutrons.

Q range and Q resolution to observe particular film features

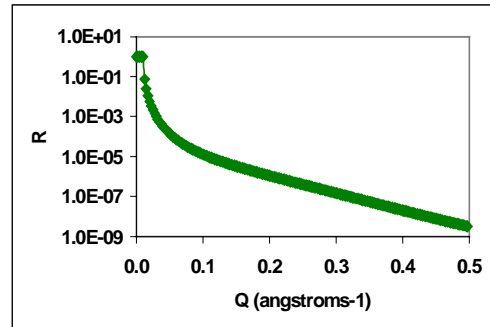
The spacing between fringes in a reflectometry profile  $\Delta Q_f \sim 2\pi/d$  (where d = the film thickness)

(a) Ultra-thin films or high precision requires large Q

Instrument design would aim to maximise flux at large Q.



(a) 50 Å thickness



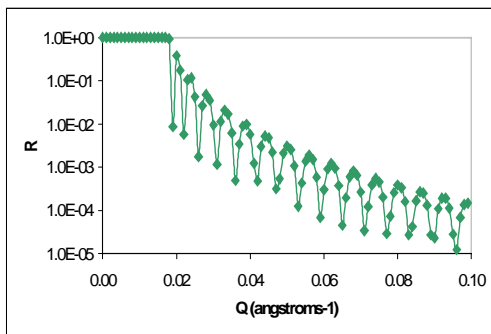
(b) 10 Å thickness

Figure E.1 Calculated reflectivity profiles of SiO<sub>2</sub> film on Si.

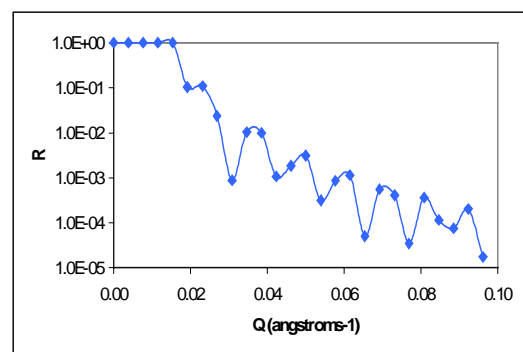
The participants recommended that the instrument should be capable of resolving film features smaller than 10 Å.

(b) Thick films requires high resolution (small ΔQ)

If insufficient resolution is available on the instrument, measurements will not be able to resolve features in the reflectometry profile. The step size between measurements  $\Delta Q_s$  needs to be sufficiently small to allow **at-least 5 steps per fringe** (10 steps per fringe is ideal).



(a)  $\Delta Q_s = 0.001 \text{ \AA}^{-1}$



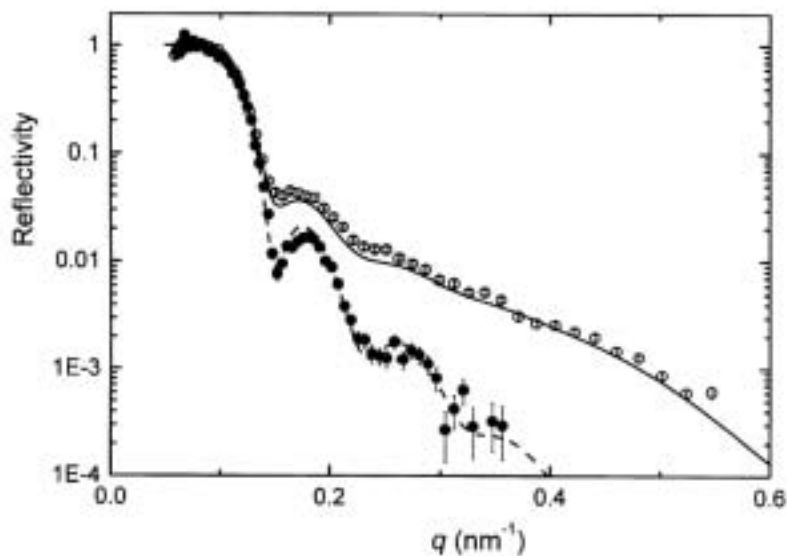
(b)  $\Delta Q_s = 0.004 \text{ \AA}^{-1}$

Figure E.2 Calculated reflectivity profiles of 1000 Å d-polystyrene on Si.

The participants recommended that the instrument should be capable of measuring films with thicknesses of at-least 2000 Å.

## Appendix F - Potential Pitfalls Identified During Workshop

Multilayer monochromators suffer from long wavelength contamination that leads to vast deviations in reflectivity data at high Q. An example is shown below for a 30 nm Ni film on glass in a study by van Well et al.<sup>9</sup>



**Figure F.1 Neutron reflectometry curves for a 30nm Ni film on glass, collected by Time-of-flight (closed circles) and multilayer monochromator (open circles) methods.<sup>9</sup>**

The above results tell us that if a multilayer monochromator is to be used then care must be taken to filter-out long wavelength contamination. A number of strategies have been suggested to achieve this, including filtering using a frame-overlap mirror or using a double monochromator.

There is no real reason to consider a combination of time-of-flight / multilayer monochromator instrument unless contemplating to carry-out Polarised Neutron Reflectometry.