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D17: the new reflectometer at the ILL

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Abstract. The reflectometer D17 has been operational for a year and has already proved to be an excellent tool for investigating surfaces and interfaces in the realms of physics, biology and chemistry. The instrument has two modes of operation, time-of-flight and monochromatic, the latter incorporating the polarised-neutron option. Both modes are flexible in the wave-vector-transfer (q) resolution. The loosest resolution required to resolve the sample structure can be chosen (and hence the highest flux), enabling the lowest reflectivities and hence the widest q-range to be measured.

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Surface and interface science has been a growing area for many years and particularly in the areas of magnetism, surfactant chemistry and model biological membranes [1], the demand for neutron reflectometry time has exceeded the supply. The ILL has had two reflectometers, ADAM [2] and EVA [3] but these, being funded by external institutes, can give only 30% of their time to ILL users. It was thus decided to rebuild D17 to become a dedicated ILL reflectometer. The instrument was to be as flexible as possible without the complication of non-optimised options. Starting with a vertical guide implied that, with the aid of focusing, the instrument would only be optimised for a horizontal reflection plane (vertical samples). This ruled out free liquid surface experiments which were reserved for another new reflectometer which is presently under consideration [4]. Due to the focusing the white beam flux at the sample position is 9.6×10^9 n/s/cm^2. The complication of having both monochromatic and TOF modes was justified by the specific advantages of each case and that neither option would adversely affect the other. TOF allows continuously variable resolution and as an entire order of magnitude in q can be measured simultaneously in less than a minuite, kinetic studies are possible. On the other hand a monochromatic beam is far easier for magnetic experiments (no q-dependent polarisation efficiency) and is the most efficient in that the peak flux is used for each point in q. Figure 1 shows a simplified layout of the instrument.

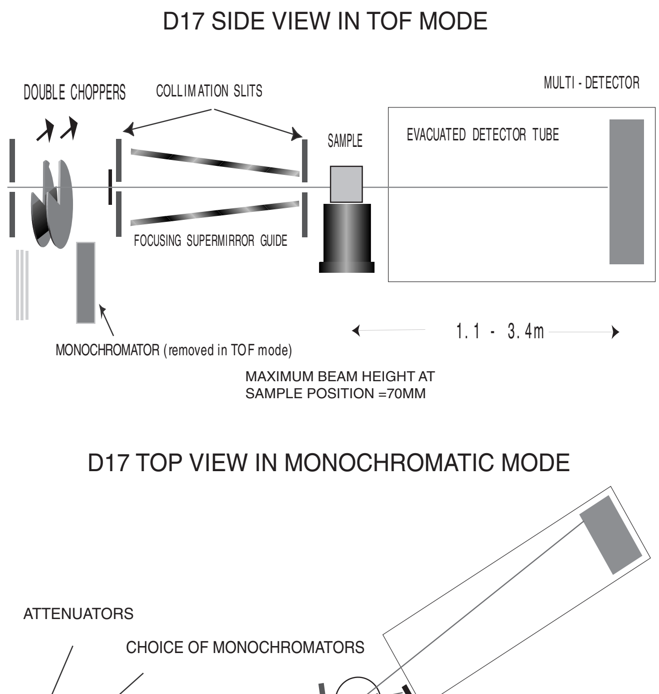

Fig. 1. Layout of D17 showing the two modes of operation

1 Time-of-flight mode

The pulsing of the beam is realised by a double chopper system with variable separation (1–11 cm) and projected sector opening (0–45°). Both choppers spin in the same direction

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and at the same speed of 1000 rpm. This chopper period is set just above the TOF of the longest available wavelength neutron of 30 Å and leads to the approximate relation

$$\frac{dT}{T}(q) \cong \frac{C}{D} + \frac{\phi}{2\pi}\left(\frac{q}{q_{\min}}\right) \tag{1}$$

where C is the inter-chopper distance, D is the TOF distance, T is the TOF, dT is the neutron pulse duration, $q_{\min}$ is the minimum q measurable at a given reflection angle and ϕ is the projected opening of the two choppers in radians. Thus when ϕ is zero the resolution is independent of q and mimics that found at ISIS with the SURF reflectometer [5] but can vary between 0.1 and 2%. A typical reflectivity measurement will involve a q-range of 0.005–0.3 Å^{-1} and can be completed in 1.2 hours for a 10 cm^2 sample. This can be achieved by joining together two measurements with a reflection angle, θ of 0.7 and 3°. The first angle can be measured with $\phi = 0$ and the second with $\phi \sim 3°$ which leads to only a small resolution jump in the overlap region and dT/T rising linearly to $\sim 10\%$ at the maximum q. For the first angle and a typical sample size the collimation is sufficiently tight for under-illumination that the direct beam flux can be measured on the detector without the need for dead-time corrections. For the second angle this is not a case as in keeping $d\theta/\theta$ constant the collimation is much looser coupled with opening ϕ means the flux cannot be measured directly on the detector. In this case an oscillating horizontal slit is vertically scanned over the beam at the sample position. This provides a wavelength independent attenuation of the direct beam and the joining together of the reflectivities measured at two angles involves an easily determined constant. The useful wavelength range is from 2 to 18Å, giving a q-range for specular reflectivity of 0.004–4 Å^{-1}. The q-range for in-plane structure is much lower at 2×10^{-5}–3×10^{-3} Å^{-1}. Figure 2 shows an example of neutron reflectivity at a solid-liquid interface measured with TOF.

2 Monochromatic mode

At present there are two prototype monochromators situated on a changer. One is a Fe/Si multilayer for polarised neutrons with a resolution of 5% working at 6 Å. The other is a Ni/Ti multilayer producing a non-polarised beam at 5 Å with the same resolution. The collimator arm is rotated 4° to capture the monochromated beam and a Sollar collimator plus a Ni coated Si stack is translated into the beam to remove long wavelength contamination. In addition two flippers, before and after the sample, coupled with a 'V' shaped polarising super-mirror can be placed in the beam for polarisation analysis. At present both prototype multilayers only have a reflectivity in the Bragg peak of 0.2. With new multilayers the reflectivity should be close to 1 giving a gain of 5 over the present flux of 5×10^4 n/s at 1 mrad angular divergence in the collimation. A reflectivity measurement typically involves a $\theta - 2\theta$ scan keeping $\delta\theta/\theta$ constant and approximately equal to 5%. The direct beam flux for each point in q is determined using perspex attenuators in a separate measurement. Figure 3 shows an example of reflectivity measured in this mode.

3 Detector

The detector is two-dimensional of size 250×500 mm and resolution 2.3×3.5 mm especially suited for off-specular studies (it is shorter and has better resolution in the horizontal

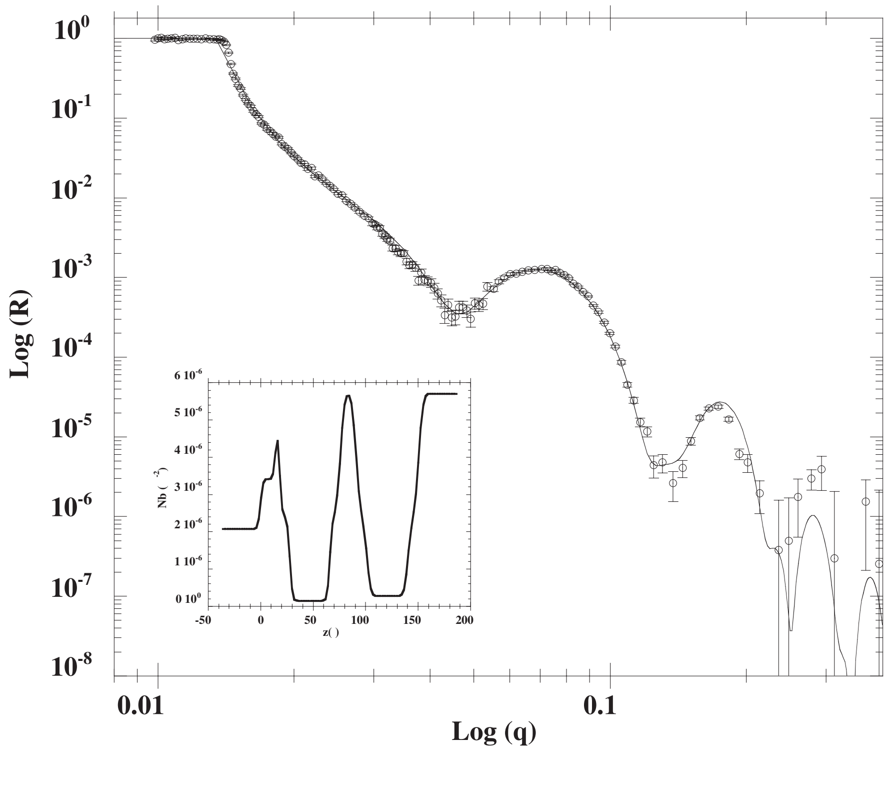

Fig. 2. Reflectivity from a double lipid bi-layer adsorbed at a Si-D_2O interface. Measurements were taken at $\theta = 0.7°$ and $\theta = 4°$ for a total time of 2 hours. The *solid line* is a fit to the data derived from the scattering length density profile in the *insert*. The structure of the two bi-layers is resolved including the head, tail groups and a water layer between the bi-layers [1]

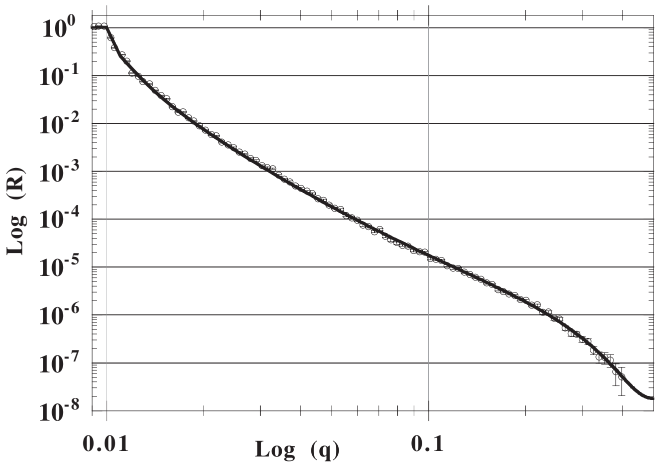

Fig. 3. Reflectivity of a 40 cm^2 Si single crystal measured in monochromatic mode. The *solid line* is a fit to the data with an oxide layer 14 Å thick and a surface roughness of only 2 Å. The surface was polished by the ESRF optics laboratory

direction). The resolution was the fitted FWHM of the beam on the detector extrapolated to an infinately thin beam. The detector is of a delay-line type made by A. Gabriel [6] which has a very low dead-time of 350 ns achieved by fast current amplifiers and real time signal processing. No measurable miss-addressing of the detected neutron position occurs even when a 300 kHz, 2 mm wide beam is measured directly on the detector. The background with the beam off and the reactor on is 1.5 c/s. The detector can be translated from 3.4 to 1m from the sample position and rotated up to 50° from the direct beam axis. The time binning is such that 1 X-pixel corresponds to 1.04 mm. As the amplifiers are current rather than charge one would not have expected the rejection of counted gamma-rays to be efficient. However the measured efficiency for counting gammas is less than 10^{-7}.

4 Sample environment

D17 has its own 2.5 T horizontal cryomagnet with a sample temperature range of 1.5–310 K. In addition there is a 1.7 T electromagnet which is only available for room temperature studies at present. A sample changer will presently be in use for up to 6 separate samples and a study is in progress for an automatic liquids changer for solid-liquid experiments. Sample environment includes a Langmuir trough optimised for preparing Langmuir-Blodgett films on large substrates.

5 Conclusions

The D17 instrument has finished the period of commissioning and is operating with scheduled experiments. The ability to explore low reflectivities (hence a wider q-range) with flexible resolution and the possiblity of kinetic experiments provides the opportunity for new science in the areas of solid/solid and solid/liquid interfaces.

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