



	Experiment title: Molecular criteria for aqueous boundary lubrication: <i>An XRR study of surfactant layer structures at mica-water interface</i>	Experiment number: SC2456
Beamline: ID10B	Date of experiment: from: 19 Mar 2008 to: 26 Mar 2008	Date of report: 30 Aug 2008
Shifts: 18	Local contact(s): Dr. Oleg Konovalov	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): Dr Wuge H. Briscoe* , University of Bristol and University of Oxford Dr Robert Jacobs* , University of Oxford Dr Robert K. Thomas , University of Oxford Prof Jacob Klein , University of Oxford and Weizmann Institute of Science, Israel Mr Kevin Mutch* , University of Bristol Mr Neale Harvey* , University of Oxford		

Report:

This has been a very successful and highly productive experiment in several aspects, as summarised below.

- 1) We designed and constructed two liquid cells to house our samples, the main body for which is shown in Fig. 1. They will be used for future related measurements, and could also be adapted to neutron reflectometry experiments. We intend to report this in a manuscript to be submitted to Rev. Sci. Instr..
- 2) We set out to study both the out of plane structures and in plane structures of surfactant bilayers on mica under water using XRR and GID respectively. However, the time restriction means that we were only able to carry out the XRR measurements. However, we still consider the experiment a success. It is the first time that XRR has been applied to mica surface under water, and the reflectivity curves are very convincing. Some examples are shown in Fig. 2, for a semifluorinated surfactant (F8H6). Distinct Keissig fringes are evident, and both the reflectivity and the fringe spacing vary as the bulk concentration of the surfactant increases from 0.1 cmc to 2.5 cmc, indicating a gradual build up of the surface layer. Such information has not been previously available. In total we have performed XRR measurements on 5 semifluorinated and 5 hydrogenated surfactants, and they have all produced distinct Keissig fringes.

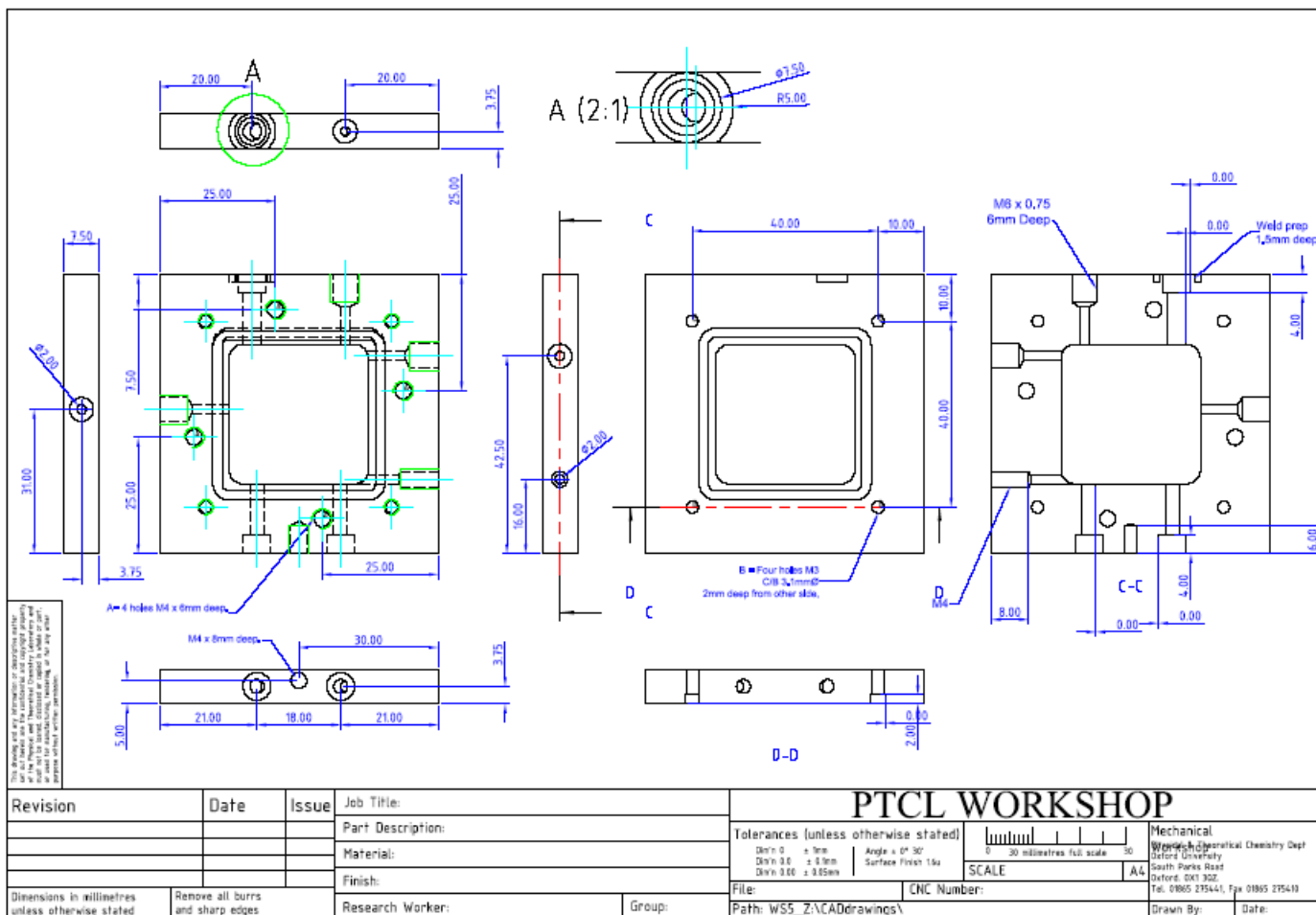


Fig. 1. Mechanical drawing for the main body of a liquid cell designed for ID10B beam line.

3) We found that our local contact, Dr Knovolav, extremely knowledgeable and helpful. The beam alignment was non-trivial due our gently curved mica surface geometry. Dr Knovolav was very careful and patient with us. In addition, he also promptly modified a software package to allow us to convert our data to a portable format.

4) The data analysis is rather challenging, due to the fact that a) it is the first such experiment, and b) the molecular architecture of the semifluorinated surfactants means there is an internal structure in the layer. So far, we have not been able to use the software packages available in the public domain to analyse our XRR data, e.g. Parrott. However, Dr. Robert Thomas (Oxford) is currently modifying his reflectivity fitting software

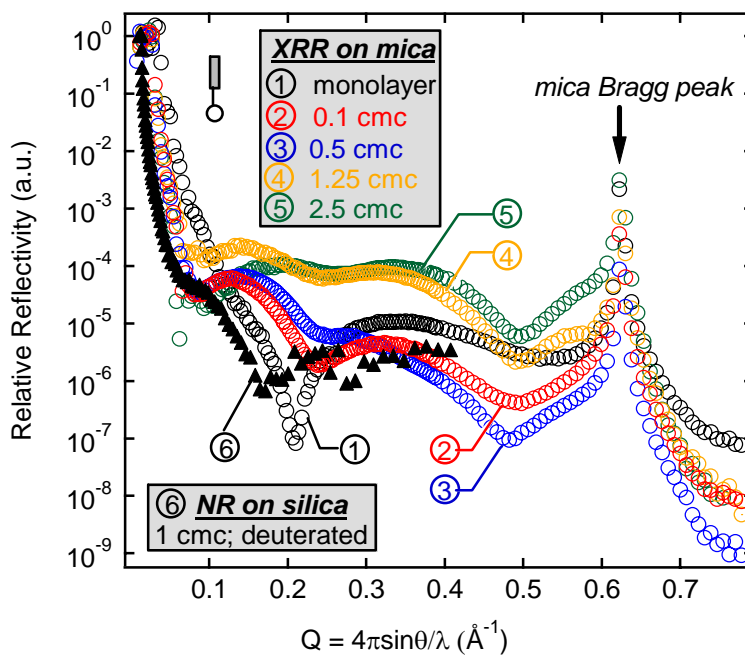


Fig. 2. Example XRR curves from a semifluorinated surfactant, F8H6, on mica under water.

originally devised for neutron reflection so that it could be applied to our XRR data.

- 5) Overall, we anticipate 4 manuscripts to be submitted as a result of this single experiment.**
- 6) Given that we didn't manage to perform the GID measurements as propose, we will intend to seek further beam time.**