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# First science on the new horizontal reflectometer FIGARO

The ILL's new horizontal neutron reflectometer FIGARO was launched on 15 April 2009 and throughout the year has delivered a packed schedule of 34 user experiments. FIGARO is a versatile instrument with features including a choice of six chopper pairs to balance resolution and flux, deflector mirrors that direct the beam upwards or downwards towards the reflecting surface and a 2D detector to image off-specular scattering. A range of sample environments exists on the instrument including a Langmuir trough for studying compressed surface films at the air/liquid interface, adsorption troughs for studying molecules at the air/liquid interface, and a number of sample cells for studying phenomena at the solid/liquid and liquid/liquid interfaces. Two of the first experiments on the instrument using a range of these configurations are described in this report.

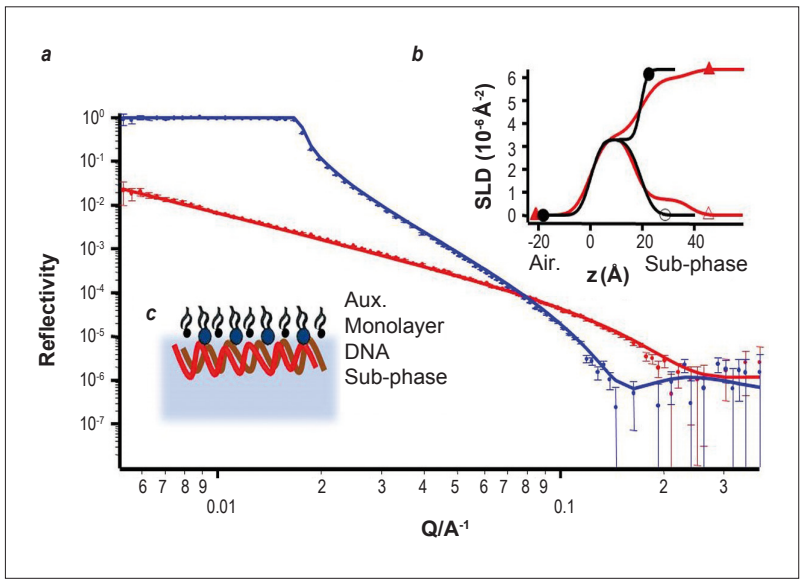


***Figure 1: (a)*** *NR profiles of DDAB:DOPE (1:1 molar ratio) monolayers at the air/water interface at a surface pressure of 30 mN/m. The markers indicate experimental data while the lines are the fitted models for $d_{74}$DDAB:DOPE on $D_2O$ containing 0.067 mg/mL of DNA [blue], $d_{74}$DDAB:DOPE on acmw containing 0.067 mg/mL of DNA [red].* ***(b)*** *The scattering length density (SLD) profile in the z-direction. The SLD was obtained from the fitting for all the contrasts: $d_{74}$DDAB:DOPE on $D_2O$ [●], $d_{74}$DDAB:DOPE on null reflecting water (NRW) [○], $d_{74}$DDAB:DOPE on $D_2O$ containing 0.067 mg/mL of DNA [▲], $d_{74}$DDAB:DOPE on NRW containing 0.067 mg/mL of DNA [Δ].* ***(c)*** *A depiction of the modelled structure at the surface when DNA was present in the sub-phase.*

Understanding the relationship between the molecular structure of a gene vector and its ability to elicit a therapeutic response is critical for effective gene therapy. Prof. Jayne Lawrence's group (King's College London) used FIGARO to study the interaction of a lipid mixture known to effect gene delivery, composed of a cationic lipid, dimethyldioctadecylammonium bromide (DDAB) and a neutral 'helper' lipid, dioleoylphosphatidylethanolamine (DOPE) with DNA. A neutron reflectivity (NR) study of the monolayers formed by this mixture has proven almost impossible due to the differential solubilities of the lipids in the water sub-phase, which results in unstable monolayers in the presence of vibrations. The careful design of FIGARO's sample table enables the formation of stable mixed lipid monolayers. **Figure 1** shows experimental and fitted NR profiles of DDAB:DOPE monolayers with and without DNA. The results revealed that although DNA formed a layer 19 Å thick, directly below the lipid monolayer, it did not alter the packing of the mixed lipid film. This study shows the potential of FIGARO for the study of complex monolayers, hitherto impossible to perform.

Dr. Karen Edler's group (University of Bath) is currently researching the formation and mesostructure of surfactant-templated polymer films at the air/water interface. These films are promising materials for structurally controlled sensors as they form spontaneously upon mixing of polymer and surfactant solutions and exhibit three-dimensionally ordered phases. The group used the adsorption troughs to control the surfactant imparted mesostructure on the films through surfactant/polymer interactions using glucose responsive polymers containing phenylboronic acids.

FIGARO was chosen for this experiment primarily for the horizontal sample configuration, time-of-flight data collection and 2D detector. The troughs were ideal as they have an automated optical alignment system directed through tilted quartz windows that saves valuable beam time. The group found it gratifying to gain high statistics in the data whilst recording sub-minute NR scans for the first time, an example of which is shown in **figure 2**. They have previously not achieved such time-resolution on these systems when using neutrons or X-rays, and the data collected will assist greatly in understanding the mechanism for the formation of the films.

Other experiments during 2009 have included a climate change investigation by Dr. Christian Pfrang (Reading University) into the destruction of organic monolayers at the air/liquid interface by nitrous oxide gases, a structural study of surfactants at the oil/water interface by Prof. Ali Zarbakhsh (University of London), and measurements of the conformation of modified cyclodextrins in mixed membranes formed on solid supports by Prof. Jean Daillant (Saclay).

As 2009 draws to a close, all the essential features of FIGARO have been commissioned by the instrument team (**figure 3**). During the long shutdown in 2010, FIGARO will undergo a number of important upgrades, including the installation of an *in situ* Brewster angle microscope, a kinetic data-acquisition mode for automatic data histogramming, and a stopped-flow system and an HPLC pump for remote sample injections.

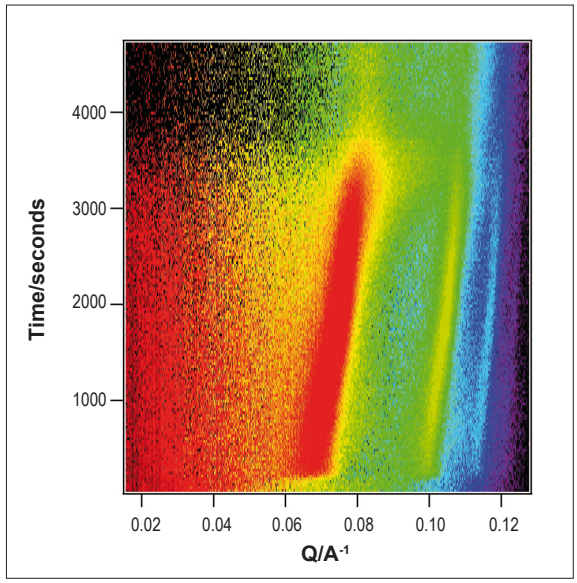


***Figure 2:*** *2D image showing film formation data on a solution of molar ratio 7CTAB:3SDS, 0.05 M total surfactant concentration with 0.1wt% polyacrylamide contining phenylboronic acid, showing a Pn3m (double diamond) cubic phase film structure collected at 30s time resolution. The 110, 211 and 222 reflections (left to right) show that the cubic phase is fully visible at 390s with a unit cell size of 330Å which decreases at a rate of 0.07Å$s^{-1}$ to 140Å before the film macroscopically roughens.*

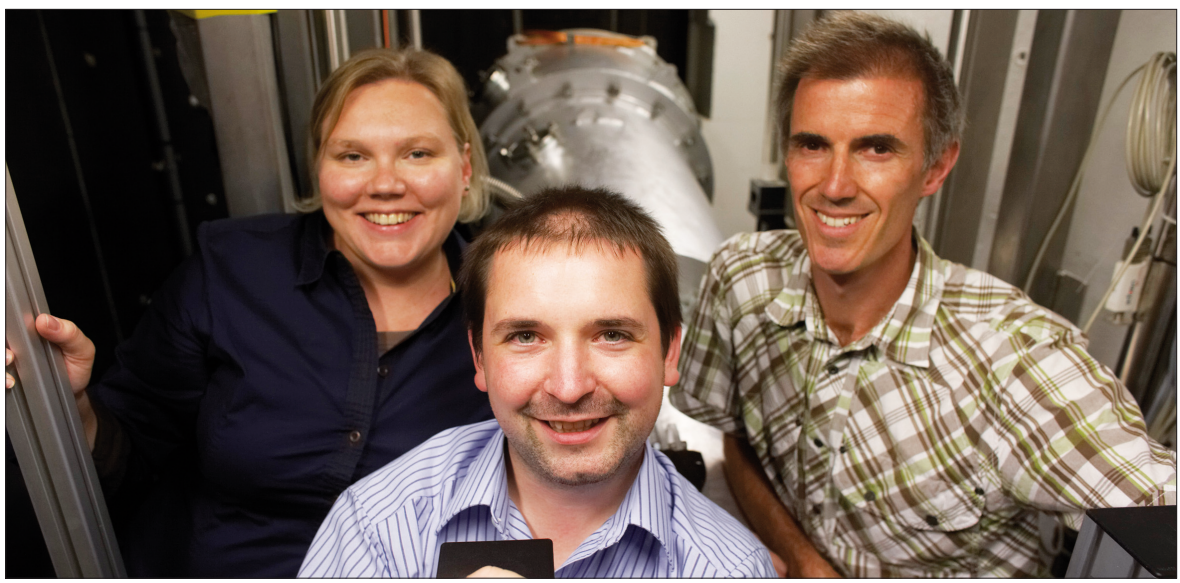

***Figure 3:*** *Photo of the instrument running team, Hanna Wacklin (left), Richard Campbell (centre) and Simon Wood (right), taken in the FIGARO sample area with a view of the detector flight tube.*