Experimental report

16/05/2020

Proposal: **CRG-2599**

Council: 4/2019

Title: Structure under confinement and shear

Research area: Methods and instrumentation

This proposal is a new proposal

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Samples: Polystyrene
Silica nanoparticles

Instrument	Requested days	Allocated days	From	To
SUPERADAM	5	5	28/01/2020	02/02/2020

Abstract:

The study of soft matter films under mechanical confinement was originally made possible by the emergence of the Surface Force Apparatus and the Atomic Force Microscope. These techniques provide accurate interaction forces at different separations. However, they do not provide structural information. Going beyond measurements of forces and measuring near-surface structures has been a challenge for a number of decades. In this regard, Neutron Reflectometry (NR) offers unparalleled possibilities for extracting structural information of mechanically confined thin films. Recently, a team partly formed by the applicants developed a sample environment for NR studies of mechanically confined thin soft films that has been a breakthrough in confined soft matter investigations. However, there is significant room for improvement. Specifically, the possibility to study shear-induced structural rearrangements would be of great value in lubrication science along with a number of other fields. We have addressed this challenge and designed a new NR confinement cell allowing shearing of two opposing surfaces. Here, we apply for beam time to test the first prototype of this novel sample environment.

Structure under confinement and shear (Proposal CRG-2599)

The confinement cell previously developed in Bristol University was tested in SuperADAM (CRG-2539). Thanks to the Nordforsk project a new prototype that implements shear (rotational and linear) was developed to be tested at SuperADAM. The sample stage of the original confinement cell was modified and two setups were made (figure 1). The shear was implemented using a servo motor control with a PLC (Mitsubishi, figure 2). Pressure control was made using a pressure sensor that measures the pressure inside the confinement cell and with the pressure controller, made by Fluid Controls, Ltd. The results presented here are qualitative in nature, as they were needed to test the functionality of the new confinement cell.

Figure 1. Sample holder modification to implement shear (a) rotational stage (b) linear stage.

Figure 2. Mitsubishi servo motor encoder and PLC suitcase.

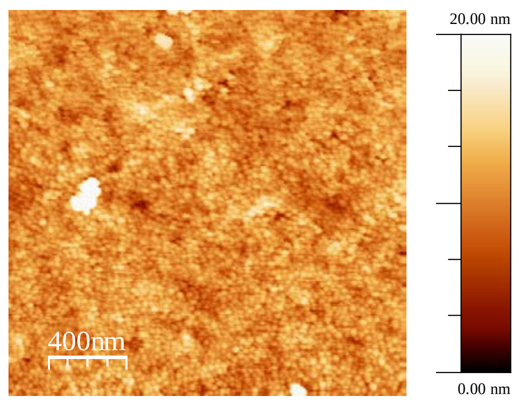

Figure 3. AFM image of the silicon block with the attached nanoparticles.

Test of the confinement-shear cell

During the measuring time, linear and rotational configurations were tested using a sample model, silica nanoparticles (Ludox TM-50 from Sigma Aldrich), on top of the silicon block (FZ, Si 100). The sample was prepared using the same method previously applied at Figaro for Nellie experiments:

- Clean silicon block using RCA procedure
- UV the Surface
- Spin coating with a 100 times diluted solution of silica nanoparticles
- Heat in the oven at 90 Celsius degrees for 10 minutes

Also, the samples were checked using the AFM Dimensions 3100 (Bruker), thanks to the collaboration of the ESRF group formed by Marie Capron, Alain Panzarella and Diego Pontoni.

For the reflectometry experiments, the following schema was followed (direct beams were measured for one block):

Linear Ofelia reflectometry: different pressures and speeds

In the first experiment with linear Ofelia, static and dynamic measurements with the membrane (Melinex 401), silica nanoparticles and the silicon block were performed. Data plotted in figure 5 it shows that regardless the speed, the intensity in the reflectometry curve is preserved. Figure 6 shows that major changes will be made to the reflectivity curve depending on the applied pressure.

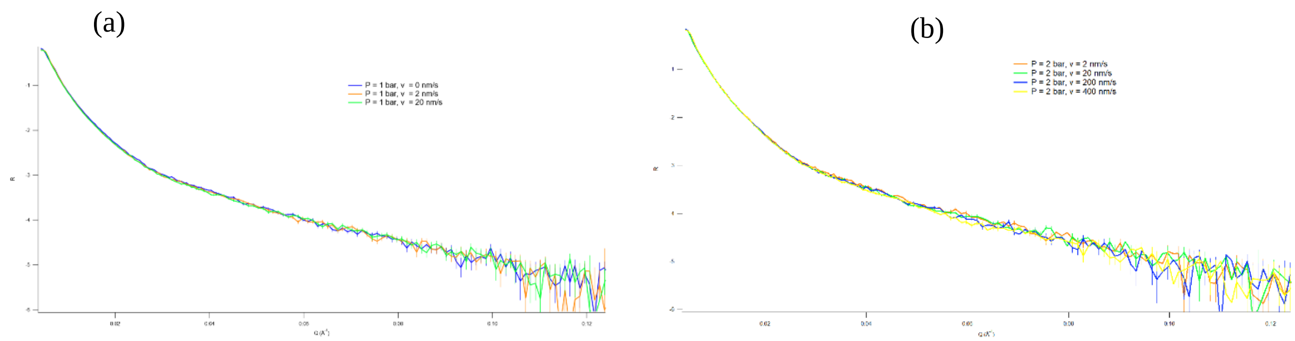

Figure 5. a) P = 1 bar for three different speeds and b) P = 2 bar for four different speeds using the linear Ofelia (Log R vs Q).

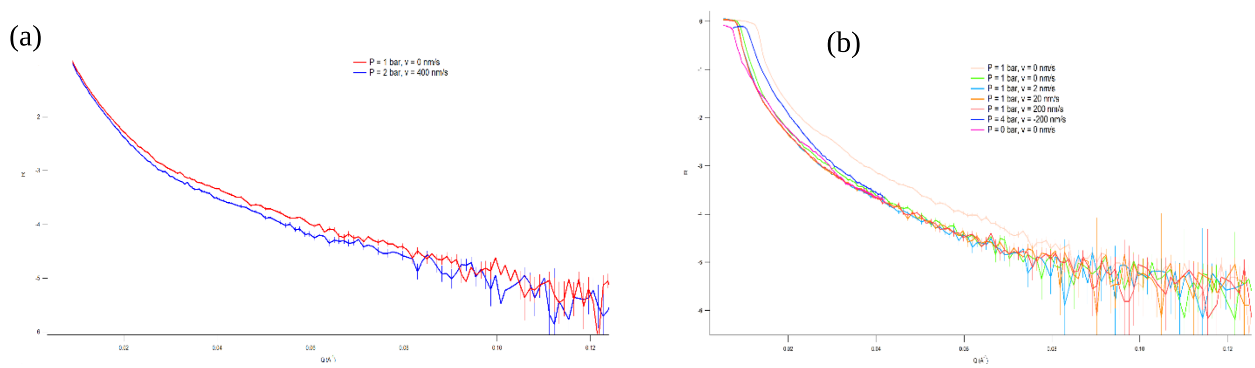

Figure 6. (a) Comparison between the P = 1 bar, v = 0 nm/s and P = 2 bar, v = 400 nm/s. Most of the difference comes from the change of pressure (Log R vs Q). (b) Experiment with D2O added between the silica nanoparticles and the Melinex membrane. The behaviour for large Q was the same as in the previous experiment, but low Q reveals some stabilization problems (Log R vs Q).

In the second experiment, a similar setup was used, but this time using D2O between the membrane and the nanoparticles. As it could be seen in figure, a total reflection appears at the beginning of the curves. Some changes in the curves for different pressures and speeds could be seen, due to stabilization problems (the setup was measured but without stabilization time).

Rotational Ofelia reflectometry: pressure and speeds

For the experiments with rotational Ofelia, and to simplify the analysis, the pressure range was restricted to one bar and two speeds were tested. Also, a stabilization time was left, and data was registered in the meantime. For these experiments, the double critical angle due to Melinex and the deuterated water pockets was observed (figure 8).

After stabilization, two speeds were selected: v1 = 12 µrpm and v2 = 120 µrpm. These speeds were low enough, in principle, to avoid overheating of the motor, as the friction in the rotational model was higher than in the linear model. However, v2 was high enough to increment the torque above the motor limit, thus overheating the motor and stopping it. As the reflectometry signal was registered while the motor stopped, a change could be seen at large Q values. The exact point where this happens is shown in figure 9. Several hypotheses could explain this behavior, being the formation of wrinkles in the Melinex one of the explanations. In fact, after the experiment was finished, and the membrane was taken out, it showed some clear wrinkles in the boundary of the sensing area.

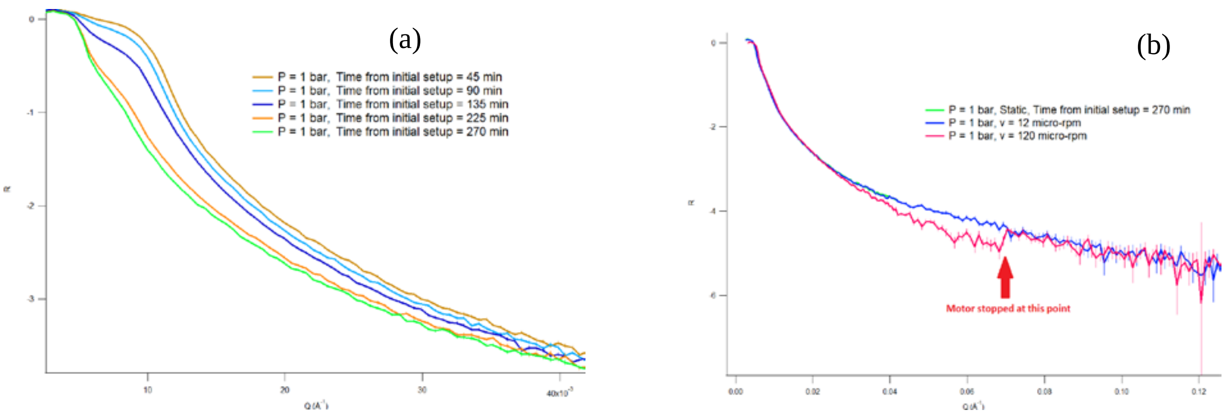

Figure 8. (a) Set of short static measurements during stabilization (Log R vs Q) of rotational Ofelia. (b) Dynamic rotational Ofelia at two speeds (Log R vs Q). Second speed was high enough to overheat the motor, giving the opportunity to register the change in reflectometry at large Q.

Linear Ofelia GISANS: static measurement

As was shown in the previous report, it was probed that Nellie could be used for GISANS configuration (D22 beamtime). During the test beamtime at SuperADAM, and thanks to Alexei Vorobiev (instrument responsible) and Olivier (beamline technician) it was possible to test Ofelia in GISANS static configuration. However, due to the shutdown of the reactor, it was not possible to test GISANS in a dynamic experiment. Figure 10 shows the results for the GISANS static test with the linear Ofelia at SuperADAM. This image probes that Ofelia works as Nellie and that GISANS could be performed. Also, this was the first time that a confinement GISANS measurement was done at SuperADAM. It should also be remarked that the measurement was obtained with a minor alignment and only 10 minutes of exposure.

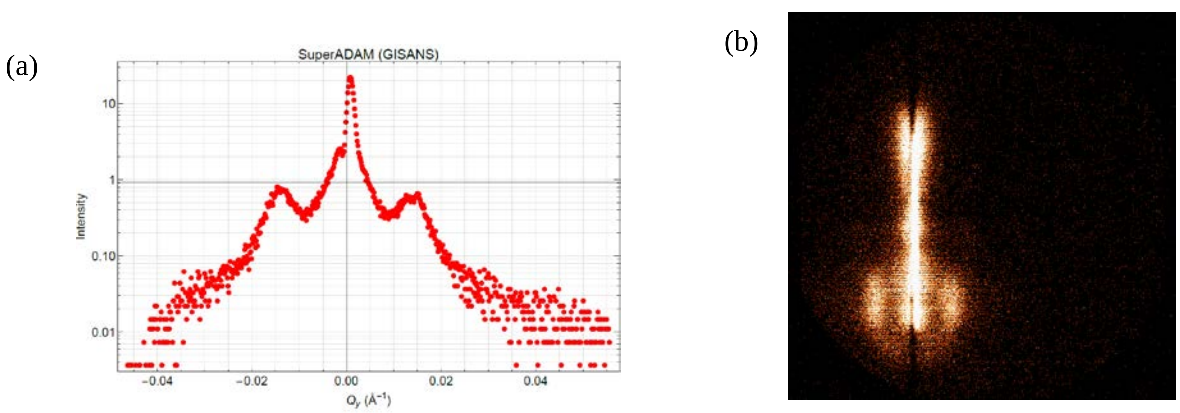

Figure 10. a) GISANS measurements using Ofelia. b) Raw data from GISANS SuperADAM.