

Experimental Report

11/05/2015

Proposal:	9-10-1356	Council:	4/2014	
Title:	TIME RESOLVED STROBOSCOPIC RHEO-REFLECTIVITY MEASUREMENTS OF F127AQ			
This proposal is a new proposal				
Research Area:	Soft condensed matter			
Main proposer:	ADLMANN Franz			
Experimental Team:	ADLMANN Franz WOLFF Maximilian GUTFREUND Philipp KOROLKOVAS Airidas			
Local Contact:	GUTFREUND Philipp			
Samples:	F127			
Instrument	Req. Days	All. Days	From	To
FIGARO User-supplied	4	4	12/11/2014	16/11/2014
Abstract: Pluronic F127 aqueous solutions are widely used self-organizing block co-polymers, that scatters significantly when solved in heavy water and display complex shear and structural behavior during temperature changes and mechanical loading. Using rhe-oreflectometry is a promising way to observe the complex ordering of micelles under different loads. By taking snapshots of the neutron event data during different stages of the oscillatory wave form (stroboscopic mode) and consequent integration of these histograms the structural changes in different steps of the waveform can be highlighted.				

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The aim of the experiment was to extract information from surface scattering of a liquid under shear. Figure A shows the experimental setup where the neutron beam is transduced through a silicon crystal and then scatters at the solid liquid boundary from a liquid under large amplitude oscillatory shear (LAOS). The changes in the scattering pattern during a duty cycle are extracted. To overcome the relatively low flux, the information of consecutive cycles is rebinned and integrated.

To access the deflection of the cone head, i.e. the LAOS duty cycle, this information was connected to one of the BNC ports of the rheometer and then electronically triggered into a TTL signal with an operational amplifier, the RC time constant of the system was set to the needs of mechanical stress. To get rid of noise driven overtriggering in the transition region, an additional algorithm has been applied. The whole process was monitored by using an oscilloscope fed with both signals.

We used a script, priority developed in Uppsala, to read out and visualize the event stream as offered at ILL. The stroboscopic rebinning, summing up, was done corresponding to the trigger signal in the post processing.

As a sample system, we dissolved Pluronic F127 18.5% in heavy water. This composition shows interesting rheological and interfacial properties.

We swept through all LAOS conditions with 1 rad/s 1 Hz and 0.1 Hz for the linear and the nonlinear region and took two different temperature settings, in addition we used two different silicon blocks, one coated with OTS, thus being hydrophobic, and one activated by Piranha solution to ensure hydrophilicity.

The analysis is still ongoing. Figure B shows the integrated Bragg peak's intensity over a half period of the duty cycle, a clear dependence is visible corresponding with the surface order and Figure C depicts a momentum transfer map of one of the slices in the time resolved experiment. For what we have extracted till now the results of the feasibility study from Oak Ridge, were confirmed and reproduced but as applied for in the proposal we were able to do a systematic study of deformations, frequencies and surface coatings at two temperatures. Unfortunately, it turned out that the lower temperature experiments are not influenced by the oscillatory shear at all and the sample does not have the tendency to crystallize under shear under all probed conditions in this phase region, which is interesting, since it is in contradiction with (S Gerth *et al* 2012 *J. Phys.: Conf. Ser.* **340** 012088 [doi:10.1088/1742-6596/340/1/012088](https://doi.org/10.1088/1742-6596/340/1/012088)) and still a publishable but negative result.

For high amplitudes and frequencies issues with counting time did occur, since the rheometer tends to overheat and then deactivate itself, which we are now aware of and will take into account for the next experiments.

In summary, we have shown the feasibility of time resolved reflectivity by using the in situ LAOS set up in combination with list mode data acquisition on FIGARO and had paved the road for future time resolved experiments at the ILL. We successfully used event mode based data acquisition combined with a trigger signal to extract time resolved neutron scattering data at the ILL. We were able to collect data of a block polymer solution of P127 solved in deuterated water at all conditions

proposed in the application. Due to the complex and time consuming rebinning and data analysis, further data analysis is still on the way.

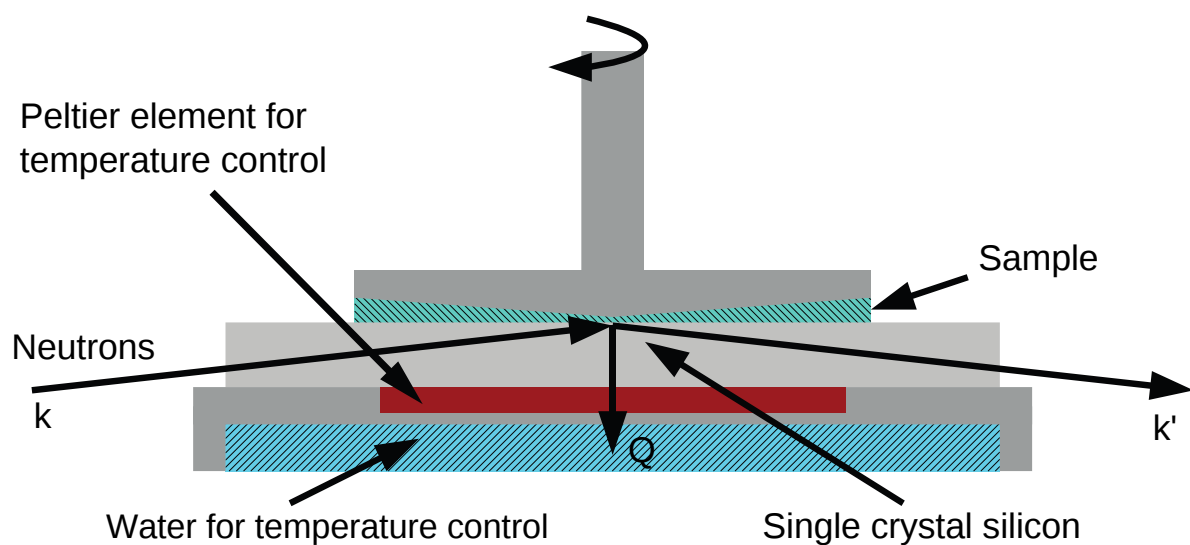


Figure A. Schematics of the setup. The neutron beam is transduced through a silicon block and reflected at the surface of the liquid under mechanical load.

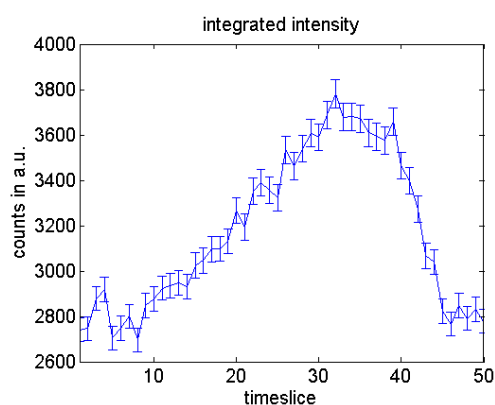


Figure B: The first order Bragg peak intensity plotted over time for a half period of the sinus duty cycle.

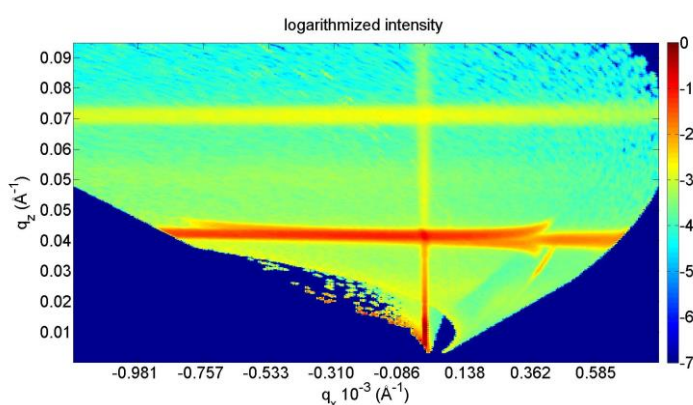


Figure C: Momentum transfer map with two horizontal Bragg sheets and the specular ridge (vertical line).