ESRF	**Experiment title:** **Rheo-SAXS on thermotropic and lyotropic liquid crystals using a vertically deflected X-ray beam**	**Experiment number**: 26-02 655
Beamline: BM26B	**Date of experiment**: from: 06/02/2014 to: 10/02/2014	**Date of report**: 01/03/2014
Shifts: 12	**Local contact(s)**: Giuseppe Portale	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

M. P. Lettinga[1]. B. Struth[2]*, D. Pennicard[2] H. Graafsma[2]*

[1]Department of physics and astronomy, KU Leuven, Belgium

[2]Photon Science Detector Systems, DESY, Germany

Report: *In situ* structure determination of the structural response in complex fluids to shear flow by small angle X-ray scattering has proven to be a successful methodology[1]. Recently we extended the possibilities of this technique by building in Desy, Hamburg, a set-up where a vertically deflected X-ray beam is passed through a plate / plate or couette geometry of a rheometer [2, 3]. One of the main advantages of this set-up, namely the possibility to probe the flow-gradient plane, has thus far not been exploited. We installed a next generation mobile version of the vertical detection line at BM26B. The installation was performed during two machine days for building the set-up and 1.5 days of beam time during which the beam was aligned and directed to a small beam stop just in front of the camera close to the ceiling of the hut. This beam time was successful because of the insights we gained in the experimental set-up itself and because of the new insights we obtained on the flow behavior of Gibbsite, which was one of the three proposed systems.

The experimental lessons concerned the two main aspects. First, the high quality of the beam confronted us with an unexpected problem that we had not encountered with the set-ups at Desy. It turned out that the motor that we employed to scan the crystal that reflects the beam is rolling to some extent, namely about a hundredth of a degree. This is, however, sufficient to totally lose Bragg conditions in the case of a very clean and low-divergent beam. In order to understand this effect we first tested three different crystals, germanium as well as silicon, which all showed the same problem. A second issue is the shape of the beam and how to best send it through the rheometer. Since the beam is rectangular this should be done with the long axis pointing in the flow direction. This was not possible with the geometry we used, which could just accommodate the beam in the gap without reflections and loss of intensity. For future experiments we will adapt our geometries, so that the beam can be only squeezed in one direction, thus increasing the resolution without losing too much intensity.

The experimental learning process described above required in total two days of beamtime, such that we could not follow up on the proposed measurement schedule. We mainly focussed on the Gibbsite system, both in glycerol and in water; see Figure 1 for an example of a scattering pattern. In this report we focus on the response of nematic Gibbsite dispersions Large Amplitude Oscillatory Shear flow. Previous experiments revealed a rich dynamical response of these systems, where at low amplitudes symmetry break is observed and at high amplitudes a fliping motion of the director at the moment that flow is reversed[2]. Due to the used geometry, however, the reflections of the nematic could not be observed and the angle of the director with respect to the gradient direction was not known. Moreover, the experiment was strain-controlled, as is the case for most *in situ* experiments.

During this beam time we were able to detect the angle θ of the director with respect to the gradient direction, since we used a couette geometry where we directed the beam along the vorticity direction. We could confirm that there is symmetry breaking at low stress amplitudes, since θ does not change sign despite of the fact that flow is reversed while the orientational ordering remains unchanged, see Figure 2 bottom. At higher stress amplitudes, where the system yields during the oscillation, the θ does change sign, although this is accompanied with very irregular behaviour where also the orientational ordering is partially lost. This most likely complies with the flipping behaviour we observed earlier[2]. Thus, the experiment successfully completed the physical picture we had of the dynamic response of nematic Gibbsite dispersions to Large Oscillatory Shear flow.

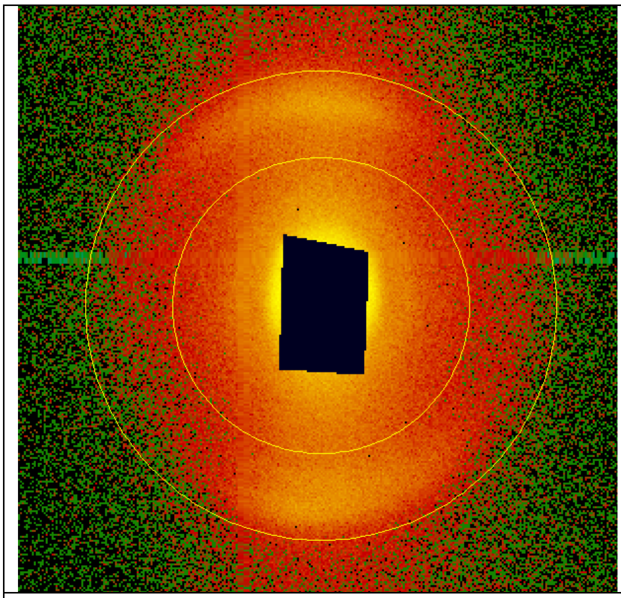

Figure 1: Scatter pattern of Gibbsite platelets in the nematic phase during Large Oscillatory Shear Flow.

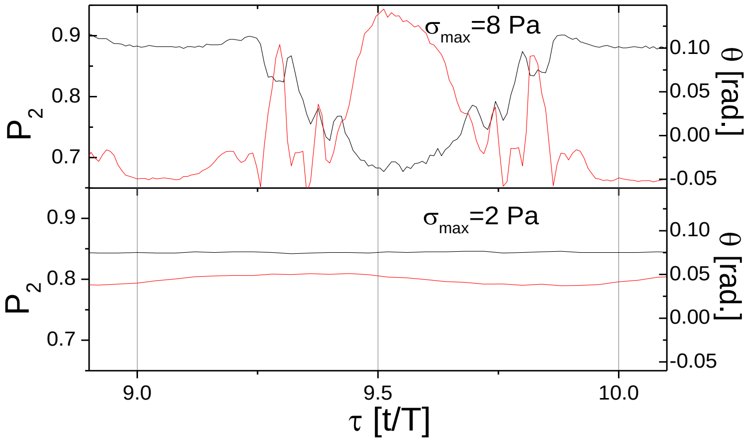

Figure 2: The response of the angle of the director (θ) and the orientational ordering given by the average second Legendre polynomial <P_2> for two different applied amplitudes, as indicated, at a frequency of 0.04 Hz.

References

1. Panine, P., M. Gradzielski, and T. Narayanan, *Combined rheometry and small-angle x-ray scattering.* Review of Scientific Instruments, 2003. **74**(4): p. 2451-2455.
2. Lettinga, M.P., et al., *Nonlinear Behavior of Nematic Platelet Dispersions in Shear Flow.* Phys. Rev. Lett., 2012. **109**: p. 246001.
3. Struth, B., et al., *Observation of New States of Liquid Crystal 8CB under Nonlinear Shear Conditions as Observed via a Novel and Unique Rheology/Small-Angle X-ray Scattering Combination.* Langmuir, 2011. **27 %6**: p. 2880-2887 %&.