ESRF	**Experiment title:** **Vertical-beam Rheo-SAXS on colloidal platelets and complex polymers**	**Experiment number**: 26-02 763
Beamline: BM26B	**Date of experiment**: from: 20/01/2016 to: 02/02/2016	**Date of report**: 31/03/2016
Shifts: 13	**Local contact(s)**: Daniel Hermida Merino	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

M. P. Lettinga[1], O. Korculanin[3]*, F. Westermeier[2]*, H. Graafsma[2], G. Baeza[4]*, D. Vlassopoulos[4], G. W.M. Peters[5]

[1]Department of physics and astronomy, KU Leuven, Belgium [2]Photon Science Detector Systems, DESY, Germany [3]Institute for Complex Systems 3, FZJ, Germany [4]Laboratory Foundation for Research & Technology Polymers Science Group Institute of Electronic Structure & Laser Vassilika Vouton Crete, Heraklion, Greece [5]Laboratory Eindhoven University of Technology Department of Mechanical Engineering, Eindhoven, Netherlands

Report: Instrumentally, the main focus of this beam time has been to extend the previously used mobile vertical beam RheoSAXS setup. [1, 2, 3] This mainly consisted of commissioning a new rheometer that was designed such that both SAXS and WAXS are feasible and exchangeable.

In previous works we always used dispersions of Gibbsite in glycerol and obtained relatively high ordering, although in marked differences were noted between orientation at the wall and in the middle of the cell, which we could observe due to the used geometry, which allows for scanning the gap. This beam time we used dispersions of Gibbsite in water at middle (nematic) and high (columnar) concentrations. We mainly focused on obtaining the flow curve, where the orientation and ordering of the platelets is followed as a function of shear rate and compare it to the stress (data not shown). Contrary to our expectations, we observed that the ordering generally was almost independent of shear rate, as well as the angle of the director. The main difference between the columnar phase and the nematic phase is that the ordering $<P_2>$ is distinctly higher in the columnar phase, see fig 1 and 2. We did see a correlation between the stress response after flow reversal and this angle, which was more pronounced in the nematic as compared to the columnar phase especially for lower shear rates. The results hint to a pronounced influence of the solvent viscosity and the concentration on the wall anchoring of this platelet system. As this is of importance for the use of these systems in many industrial applications like the use as drilling mud or the formation of tough films, it is important to know how this can be tuned. In further experiments we therefore plan to use different mixtures of platelets and scan the gap in order to obtain local orientations.

The main body of our beam time was used to commission the WAXS in combination with rheology. We used a newly designed set up by the group of Prof. Peters from the TU Eindhoven in collaboration with our group in KU Leuven and FZ Juelich. This set up allowed to measure angles up to 30 nm^{-1}. The unique feature of the set up is that polymer melts can be tested rheologically while checking for features like crystallization and in that sense it is unique. The CIT group from KU Leuven used it to measure WAXS in couette geometry. This exemplifies the very flexible use of the set up. Moreover, an oven was build-in which can go up to 270 ºC, which is more than sufficient for most polymer melts.

Here we show the response in time of a random T4T (“hard”) / pTHF (“soft”) multiblock copolymers going up and down in temperature and thus melting and freezing the sample. Indeed all the expected features are found, combined with the rheological response.

It should be noted that we did encountered a few issues that should be solved for next experiments. The main issue is maybe that the transition from SAXS to WAXS should be improved. Changing to the WAXS took about 12 hours, but we have evaluated the set up using our experience from last beam time and developed a new scheme of how to switch between the two geometries.

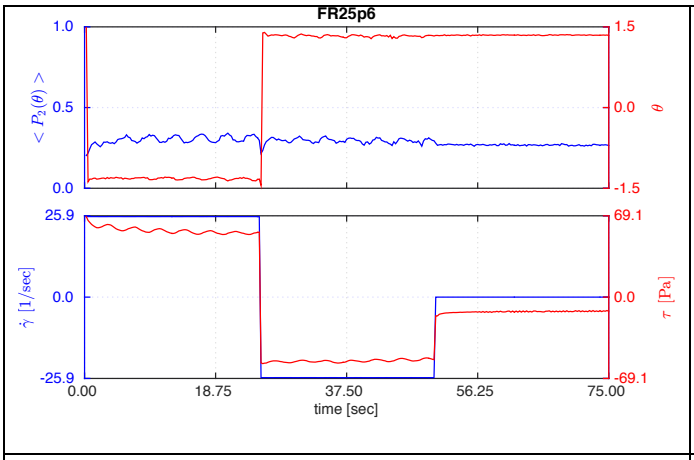

Figure 1: Flow reversal of nematic Gibbsites dispersed in water at 25.6 1/sec shear rate. Where P2 is the orientational order parameter and theta is the azimuthal orientation of the nematic director.

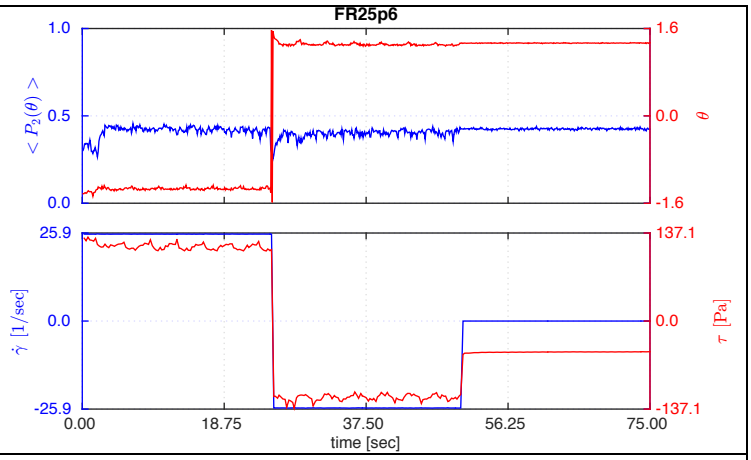

Figure 2: Flow reversal of columnar Gibbsites dispersed in water at 25.6 1/sec shear rate.

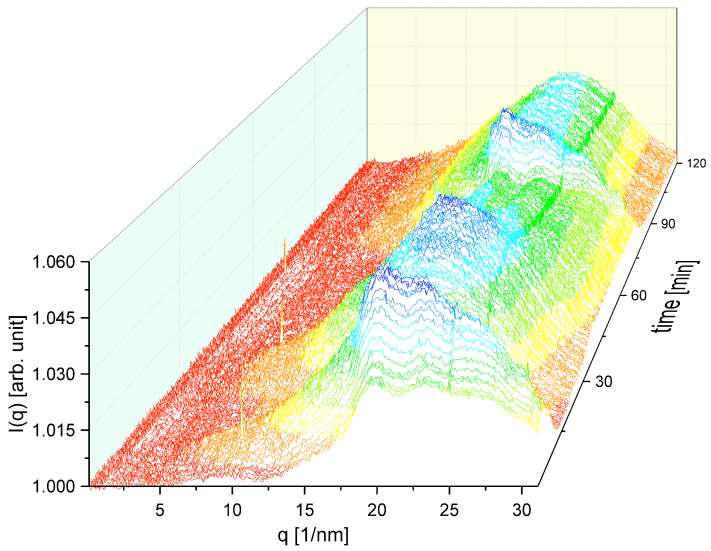

Figure 3: Crystallization of the T4T units in industrial multi-block copolymers.

References

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