The GISANS options at ILL for liquid and polymer interfaces: Recent examples and perspectives

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I will present the possibilities and some examples of grazing incidence small angle neutron scattering (GISANS) experiments at the Institut Laue-Langevin (ILL) in Grenoble, France, mainly performed on two instruments: The SANS machine D22 and the neutron reflectometer FIGARO. I will put a particular emphasis on the different options of sample environment available for GISANS. I will focus on interfaces confined at liquid/gas, liquid/solid, solid/solid and polymer/polymer interfaces.

To illustrate the possibilities, I will show recent scientific results obtained by GISANS at ILL. These will include two studies at the solid/liquid interface under *in situ* shear using a cone/plate rheometer mounted on the FIGARO sample stage [1]. In one study, we followed the destruction of lamellae multilayers of an anionic surfactant on sapphire surfaces under shear stress. By a combination of specular neutron reflectometry (NR) and GISANS we observed an alignment of the lamellae parallel to the interface which is either completely lost for steady shear above a threshold shear stress (see Fig. 1) or partially lost for oscillatory shear. In both cases the monolayer in direct contact with the solid surface stays intact [2]. In the second example, using the same sample environment, we observed the inverse effect, namely alignment of peptide nanotubes in water close to a silicon surface above a certain shear rate [3]. In both examples the structural data is complemented with the mechanical response measured with the rheometer *in situ.*

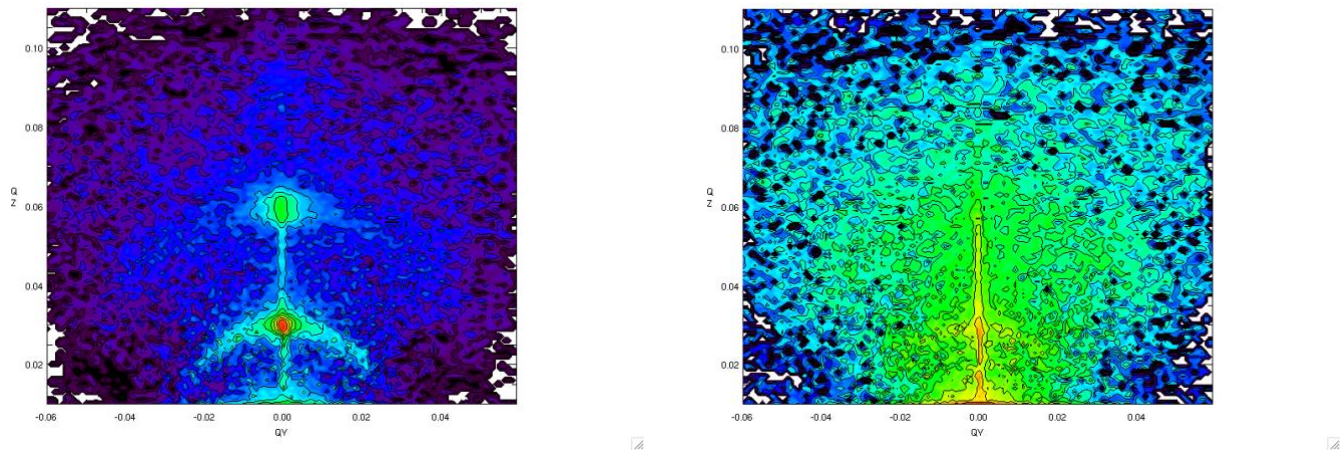

Figure 1: GISANS patterns recorded on FIGARO at θ_i = 1.4° λ = 2-20 Å < λ_C.
Left: Static sample. Right: 2 s^{-1} shear rate.

I will show also recent examples of thin polymer film investigations on polymer multilayers supported by silicon substrates investigated by a combination of NR, off-specular neutron reflectometry, GISANS and transmission SANS. One example will include the determination of the single chain conformation of polyelectrolytes arrested in a polyelectrolyte multilayer assembly prepared by the layer-by-layer technique. The out-of-plane conformation was inferred from NR, whereas the in-plane conformation was measured using (GI)SANS showcasing the necessity of the combination of several techniques to get a full (3D) picture of the system. We confirm the expected flattened coil conformation for poly(styrenesulfonate) (PSS) / poly(allyl amine hydrochloride) (PAH)

multilayers and determine quantitatively the extend of single PSS coils as a function of different preparation techniques, namely dipping, spraying and spin-coating as well as the influence of salt concentration during preparation [4].

In the last part of my talk I will show the current status of GISANS capabilities at the air/liquid interface on FIGARO using Langmuir troughs and an outlook including the development of a confinement cell for GISANS on D22 allowing the nanometric confinement of a liquid sample between two solid surfaces, eventually allowing the application of shear stress as well [5]. The suitability of these sample environments for GISANS will be assessed by using a monolayer of silica nanoparticles as model system.

References

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