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At the National Synchrotron Radiation Research Center (NSRRC) with a 1.5 GeV storage ring, a small-angle X-ray scattering (SAXS) beamline has been installed. The X-ray beam can be selectively monochromatized by a double Si(111) crystal monochromator (DCM) with high energy resolution ($\Delta E/E \approx 2\times10^{-4}$) in the energy range of 5-23 keV or by a double Mo/B_4C multilayer monochromator (DMM) for 10-30 times higher flux (~10^{11} photons/s) in the 6-15 keV range. A plane mirror is especially installed to the beamline to selectively deflect the beam downwards with high precision for grazing-incidence SAXS (GISAXS) with liquid surfaces. Using a grazing incident angle near the critical angle of water with 10 keV X-rays, we have monitored in-situ the growth process of silicate films at the air-water interface. Cetyltrimethylammooium bromide (CTAB) is used as the surfactant template for the tetraethyl orthosilicate (TEOS) in the solution in forming mesoporous silicated films, with the film growth temple controlled by temperature (in the range 25-55 °C) and pH value. The TESO molar ratio is varied from 0.04 ro 0.7 with respect to the molar ratio of H_2O:HCl:CTAB = 100:2-0.5:0.11. At 25 °C, after an induction period the formation of a lamellar phase and its transformation to a hexagonal mesophase of single-crystal-like reflections can be clearly observed. The layering process of the silicate rods, however, can be suppressed at higher temperatures above 45 °C; namely, the hexagonally-packed silicate rods can be formed near the air-water interface without going through the layering process of the rods, which is due presumably to larger thermal fluctuations.[1] Furthermore, transmission SAXS is used to monitor the evolution of the aggregation structure of the silicate/surfactant complex in the bulk solution. During the induction period, there are mainly complex CTAB/TESO rod-like micelles. Later, randomly oriented domains of lamellarly and hexagonally packed rods appear sequentially in the bulk solution. These results imply that the lamellar-to-hexagonal phase transformation can occur both in the bulk and near the air-water interface. Instead of layer-by-layer formation, it is possible that the silicate films may grow via reorientation-and-attachment of ordered silicate domains that adsorb to the air-water interface, while there is a thin surface layer of one or two micelle thickness stabilized at the air-water interface.

[1] S. A. Holt; J. L. Ruggles; J. W. White; R. F. Garrett, *J.Phys. Chem.* **2002**, 106, 2330.

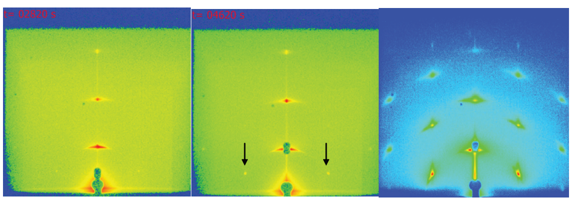

Figure 1. *Representative GISAXS images taken at 2820 s, 4620s, and 4 h, for a highly ordered silicate film in situ formed at the air-water interface at 25 °C.*

Keywords: GISAXS; air-water interface; silicate films

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New bio-SAXS Beamline ID14-3 at the ESRF. Petra Pernot[a], Adam Round[b]. [a]*ESRF, BP220, Grenoble, France.* [b]*EMBL Grenoble Outstation, France.*
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The new small-angle scattering beamline ID14-3 at the ESRF, Grenoble, France, dedicated exclusively to experiments of biological macromolecules in solution, is under user operation from November 2008 (bio-SAXS beamline). Originally running as a protein crystallography beamline, ID14-3 was refurbished, still as a part of the ESRF Macromolecular group (MX), with the main aim to provide a facility with 'quick and easy' access to rapidly growing demands from crystallographers, biochemists and structural biologists. The beamline provide manual and automatic sample loading/unloading, data collection, processing (conversion of a 2-D image to a normalized 1D X-ray scattering profile) and analysis. The users obtain on-line standard data concerning the size (radius of gyration, maximum dimension and volume) and molecular weight of samples which allow on-the fly ab-inito shape reconstruction in order to provide feedback enabling the data collection strategies to be optimized. Automation of sample loading is incorporated on the beamline using a device constructed in a collaboration between the EMBL (Grenoble and Hamburg outstations) and the ESRF. Semi/automated data analysis is implemented following the model of the SAXS facility at X-33, EMBL Hamburg. Future plans extend to allowing remote access, based on the system currently in use on the ESRF MX end-stations.
The photon source consists of three high power undulators shared with three other end-stations: ID14-3 is a fix energy beamline (E = 13.3 keV) as two others and the third end-station is tunable. The flux can consequently vary according to the tunable end-station request on undulator settings. The beamline optics consists of first diamond (111) monochromator in Laue geometry, second germanium (220) monochromator in Bragg geometry and a torodial mirror with the fixed focus spot close to the sample location. The beam defining slit just after the mirror reduces parasitic scattering downstream. An aperture slit is implemented after the 10 m long tube between optics and experimental hutches. A set of guard slits in the experimental hutch defines the illuminated region on the sample, being 0.7 × 0.7 mm^2 with the maximal flux 10^{12}ph/s. The spot on the detector is 1 × 1 mm^2 if 2 m from the sample. The sample station is mounted onto an marble table together with modulable lenght flight tube and Vantec-2000 detector (2D gaz filled detector from Bruker). The premise of the new bio-SAXS facility is to take advantage of having an optimized but simple experimental setup. The representative user experiments and inhouse research examples will be presented. An additional benefit of building this new facility at the ESRF is the collaboration with the already established small angle neutron scattering facility at the ILL, Grenoble. Users will have the possibility to access both facilities for appropriate experiments. This will allow the complementary information provided by neutron and X-ray scattering to be obtained in a single visit to the Grenoble site.

Keywords: proteins in solution; small-angle scattering; synchrotron X-rays