ESRF	**Experiment title:** GIXD Studies of the 2D Pressure Driven Transformation of PbS Nanowires to Nanosheets	**Experiment number**: SC-3878
Beamline:	**Date of experiment**: from: 24 April 2014 to: 29 April 2014	**Date of report**: 29/03/2015
Shifts:	**Local contact(s)**: Oleg Konovalov	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

*Yuval Golan, Materials Engineering Department, Ben-Gurion University, Beer-Sheva, Israel

*Somobrata Acharya, Centre for Advanced Materials, Indian Association for the Cultivation of Science, Kolkata, India (*did not participate in the experiment due to delays in Visa processing)

Jacob Israelachvili, Dept. of Chemical Engineering, University of California, Santa Barbara, USA

*Alexander Rabkin, Materials Engineering Department, Ben-Gurion University, Beer-Sheva, Israel

*Alexander Upcher, Materials Engineering Department, Ben-Gurion University, Beer-Sheva, Israel

*Alexander Pekin, Materials Engineering Department, Ben-Gurion University, Beer-Sheva, Israel

Experiment: PbS nanowires were spread from a dichloromethane suspension onto the air-water interface using a Langmuir trough. Trough enclosure was sealed and purged with He gas in order to minimize collision with air molecules. Multiscale GIXD measurements were carried out using 22 keV radiation with a resolution of 0.053 nm^{-1} at wide angles in a q range of 0.5-53 nm^{-1}, in order to study the correlation with the atomic structure and orientation of the individual nanowires as well as to elucidate the superstructure of the nanowire array. The use of the Pilatus 2D detector allowed for high data acquisition rates.
Measurements were taken prior to compression and at increasing surface pressure values. At pressure values of ~ 30 mN/m the subphase was heated (in a range of 5-45°C), to supply additional energy in order to merge the PbS nanoribbons into sheets, with periodical GIXD measurements to monitor the transformation. Control experiments with bare TOA molecules were carried out in order to isolate and characterize the role of the pure surfactant during the coalescence process. We note that Qz periodicity was observed already after spreading a sufficient amount of the nanowires, before any external compression was applied.

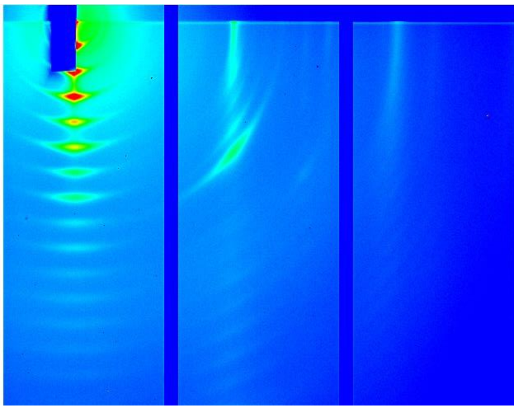

Figure 1: GIXD map obtained from a nanowire sample following compression to surface pressure of 30 mN/m and heating cycles at 24°C, 34°C and 43°C. Snapshot taken at 43°C and 14.4 mN/m.

Results: Figure 1 displays a sample GIXD map. The position of the central beam (overshadowed by the beam stop in the figure) was predetermined, allowing the extraction of values along Qz (vertical axis) and Qxy (horizontal axis). The most noticeable features are the multiple order arcs with serial peak intensities along Qz (Qxy=0) and the bragg rods initiating at the Qz=0 axis. These features appear following initial compression and intensify following additional compression and heating.

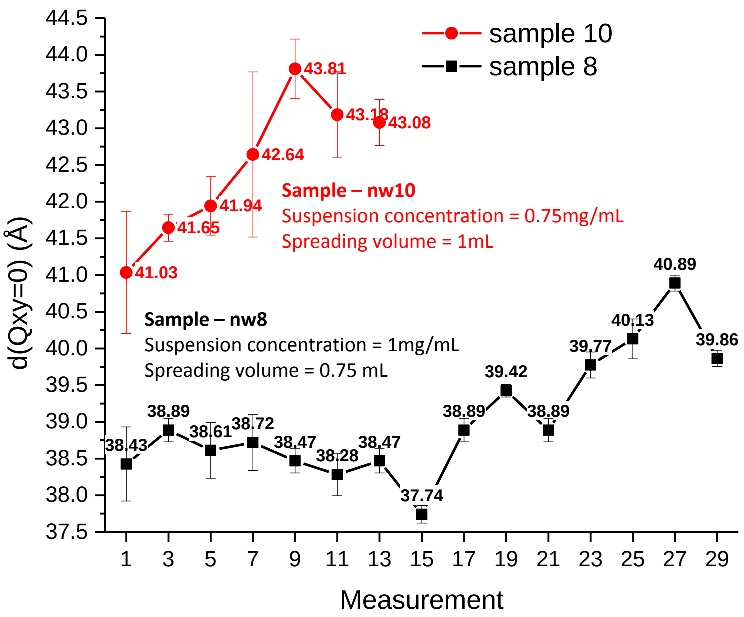

Figure 2: First order interplanar distances of the reflections along the Qxy=0 axis for two of the samples, following varying compression and heating conditions, with measurement 1 indicating initial state without external compression. For sample 10 (marked in red), the measuremets were made maintaining a constant surface pressure, and heating and holding at 34°C for an increasing amount of time. For sample 8 (marked in black), the measurement range of 1-9 was made while increasing the surface pressure, the measurement range of 11-17 was made while maintaining constant surface and decreasing the surface pressure following relaxation, and the following measurements were made while heating and holding at 34°C and 43°C.

Analysis of the arcs with peak intensity along the Qxy=0 axis revealed them to be multiple order reflections of the same distance indicating a highly ordered structure of planes parallel, with a small tilt angle, to the water surface. Extraction of these distances allows monitoring the effect of compression and heating conditions on the interplanar distances of this structure, as can be seen in Figure 2.

In order to correlate the interplanar distances to the sample geometry and structure, transmission electron microscopy (TEM) imaging was carried out at Ben-Gurion University for samples collected from the air-water interface during the experiment following the compression and heating experiment performed, as can be seen in Figure 3.

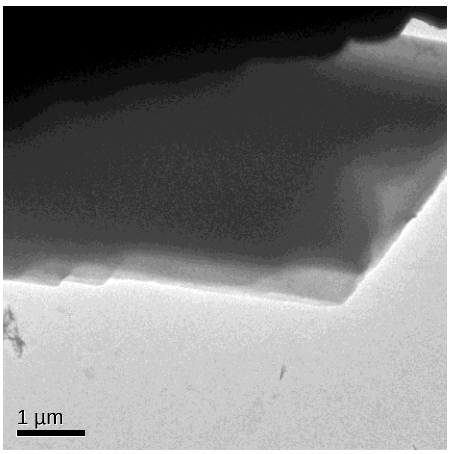

Figure 3: TEM image of a sample collected from the air-water interface following compression and heating experiment, showing formation of multilayered PbS sheets.

The TEM imaging revealed the formation of multilayered PbS sheets. This is likely to be the origin of the arcs in Figures 1 and 2, with the interplanar distance around 4 nm being the distance between two adjacent layers of the sheets. This can be used to describe the sensitivity of these layers to increasing surface pressure and temperature.

Analysis of the observed Bragg rods (at Qz=0, seen as vertical lines in Figure 1), revealed a pattern with the strongest rod located at d_{Qz}=0 of ~6Å. When compared to the Qxy=0 data, this feature has a similar pressure and temperature dependence, as can be seen in Figure 4. Therefore, the reflections could not be related to a particular structural feature of the nanorod arrangement within the air-water interface plane.

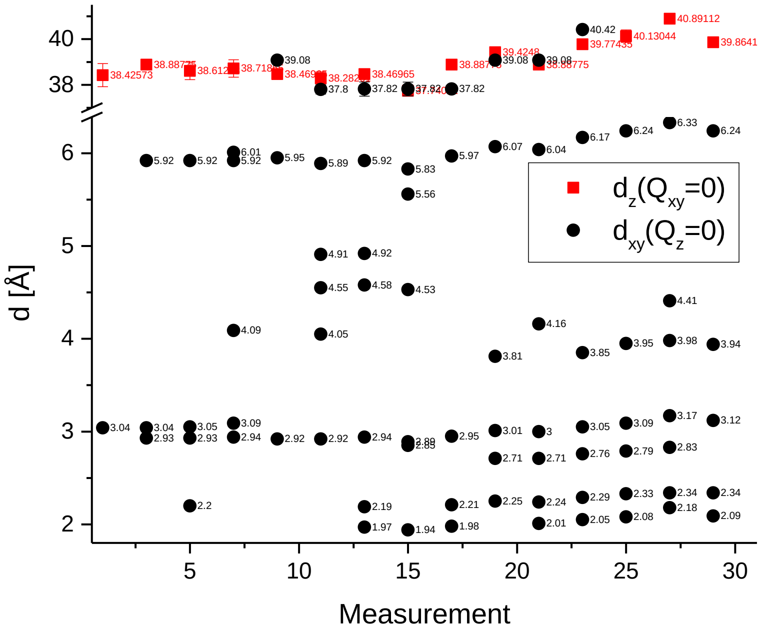

Figure 4: Reflections observed along the Qxy=0 axis for sample 8 (marke in black), following varying compression and heating conditions, with measurement 1 indicating initial state without external compression. The measurement range of 1-9 was made while increasing the surface pressure, the measurement range of 11-17 was made while maintaining constant surface and decreasing the surface pressure following relaxation, and the following measurements were made while heating and holding at 34°C and 43°C. The red dots indicate the first order interplanar distances of the reflections along the Qz=0 axis, as seen in Figure 2.