



	Experiment title: Kinetics of multiscale self-organisation via a pressure jump	Experiment number: SC-4987
Beamline: ID02	Date of experiment: from: 30-09-2020 to: 3-10-2020	Date of report:
Shifts:9	Local contact(s): Theyencheri Narayanan, Thomas Zinn	<i>Received at ESRF:</i>
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Report:

The objective of this experiment was to use pressure jumps to induce phase changes in a rich self-assembling system of crystalline bilayers. This system, which consists of a 1:2 molar ratio of SDS and beta-cyclodextrin which form host-guest complexes, self-assembles into crystalline bilayers that in turn form superstructures such as multiwalled microtubes of up to 100s μm in length. In previous synchrotron studies (in particular SC-4382) we have demonstrated that the self-assembly process follows a kinetics-dominated mechanism of successive nucleation events occurring in tandem with cylinder closure events. (J. Landman et al. Science Advances 2018).

The experimental plan for this experiment was changed due to the limitations inherent in remote-access experiment due to COVID-19 restrictions. Instead of the originally- planned pressure jump, a temperature- and concentration-dependent rheo-SAXS study was performed, which was easier to do with limited manpower.

While currently we are working on fitting procedures for rheo-SAXS experimental results, we have initial fitting results for a range of concentration-dependent controlled melting experiments that were part of the dataset recorded during this experiment. Fitting of the low-q range profiles shows that pre-assembled samples of multiwalled microtubes, when slowly heated, follow essentially the reverse of the formation mechanism elucidated in experiment SC-4382 – Multiwalled microtubes melt from the inside out. The innermost membranes are wound up in a tighter, less favourable radius and as such have a lower melting temperature. Figure 1 shows the mean radius of the microtubes as a function of temperature, for a range of different concentrations. We see that the mean radii trace out an envelope of a universal curve that relates a preferred radius to the temperature, independent of concentration.

Preliminary measurements inside a pressure cell show us that close to this melting temperature, changes in mechanical pressure absolutely affect the structure of the crystalline bilayers. Figure 2 shows how an 8 wt% sample of SDS/beta-cyclodextrin is brought from atmospheric pressure up to 177 bar of overpressure. From the 1D scattering profile, it is clear that the superstructure disappears quickly. Releasing the pressure and recording the recovery of structure, we see the structure returning slowly, over the course of several minutes –

a significantly slower process than when induced through a temperature jump. However, higher concentrations can lead to faster kinetics.

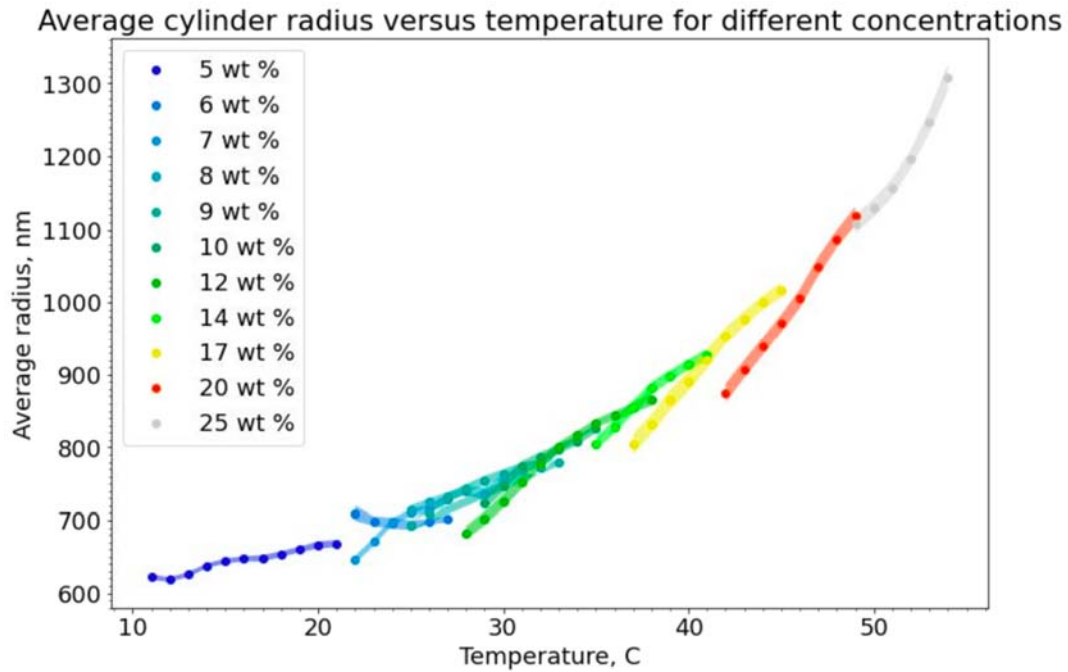


Figure 1: Mean SDS/beta-cyclodextrin cylinder radii as function of temperature, showing the envelope of a universal curve describing preferred mean radius as a function of temperature.

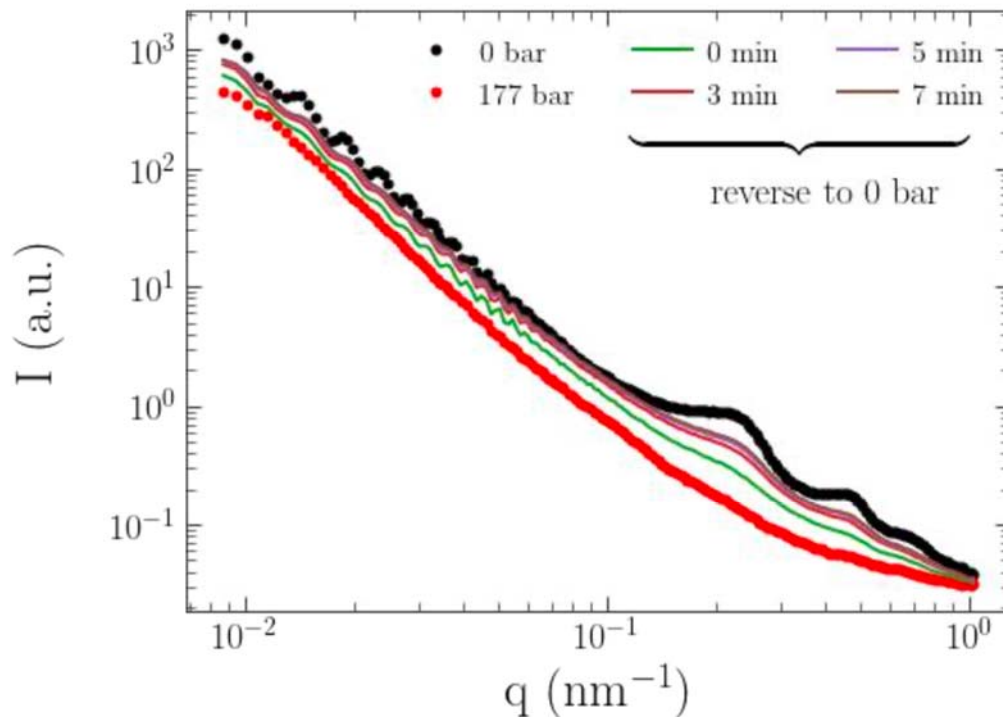


Figure 2: 1D scattering profiles of an 8wt% SDS/beta-cyclodextrin sample at room temperature, before, during and after applying 177 bar of overpressure.