ESRF	**Experiment title:** "Natural and synthetic structural colour by spinodal phase separation"	**Experiment number**: SC-4386
Beamline: ID02	**Date of experiment**: from: 11/11/2016 to: 14/11/2016	**Date of report**: 11/09/2017
Shifts: 9	**Local contact(s)**: Rajeev Dattani	*Received at ESRF:*

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Report:

The aim of this expeirment was to investigate the dynamics of phase separating media in time. ID02 was used to collect time resolved ultra-small angle x-ray scattering (U-SAXS) data on drying films of a cellulosic polymer which containted various concentrations of a salt capable of inducing phase separation. This sample set was selected because previous work had shown that these solutions produced highly voided phase seperated films as shown in Fig. 1. Additionally, films of these solutions were known to take more than a minute to dry, making the dynamics slow enough to be captured within the capabilities of ID02. The exact mechanism through which these films phase separated was unknown prior to this experiment.

The biggest challenge in doing this experiment was developing a way to remotely drawdown a film, from solution, inside the beamline hutch. The solution was to design a set of rails which could accommodate a drawdown cube. Set into the rails was a moving platform that could swing freely about a single axis. At the top of the moving platform a circular hole where a mica disk was placed as a substrate for the film. At the bottom of the moving platform a screw was placed to hold washers and nuts to act as a counter weight. When the drawdown cube was positioned over the hole in the moving platform the weight of the cube is enough to hold the platform level. However, once the cube has finished drawing a film and passes the edge of the platform, the weight at the bottom of the platform caused it to rotate into a vertical position. Detailed schematics of the set up are shown in Fig. 2a.

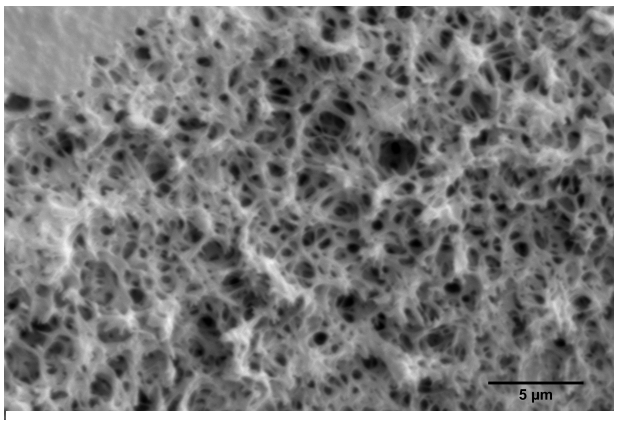

Figure 1: SEM image of a phase separated cellulose film

Once this setup was in place, it was possible to add a solution of interest to the center of the drawdown cube, seal the hutch and then use a linear actuator to push the cube down the rails and have the film rotate into a veritcal position in the path of the beamline.

The results for a film contaning a 2:1 ratio of polymer to salt are shown in Fig. 2b as a 3-D plot showing a the entire background corrected data set from the onset of the measurement until the film had finished drying with each hoizontal line in time being a single scattering pattern. The area of interest for understanding the phase separation mechanism occurs in the region between time 20 and 40 seconds. When the scattering patterns are plotted within this time scale a clear peak showing a characteristic length scale is observed for each patter as seen in Fig. 2c. As time progresses, this peak increases in intensity and the location of q_{max} moves toward lower q values. When the peak positions are plotted in real space versus time, as shown in Fig. 2d, a clear linear relationship is observed. These results clearly indicate that the films are phase separting through the mechanism of spinodal decomposition[1]. This result has particuar importance because the measured films were designed as biomimetic of a structural colour observed in nature and therefore could help reserachers further understand how nature makes it self assembled strucutres. We are in the process of drafting what we hope will be a high impact paper combining our recent tomography data on white beetle scales from ID16B (SC 4363) with this synthetic work and think that the story and science we have discovered makes a really exciting piece of work [2].

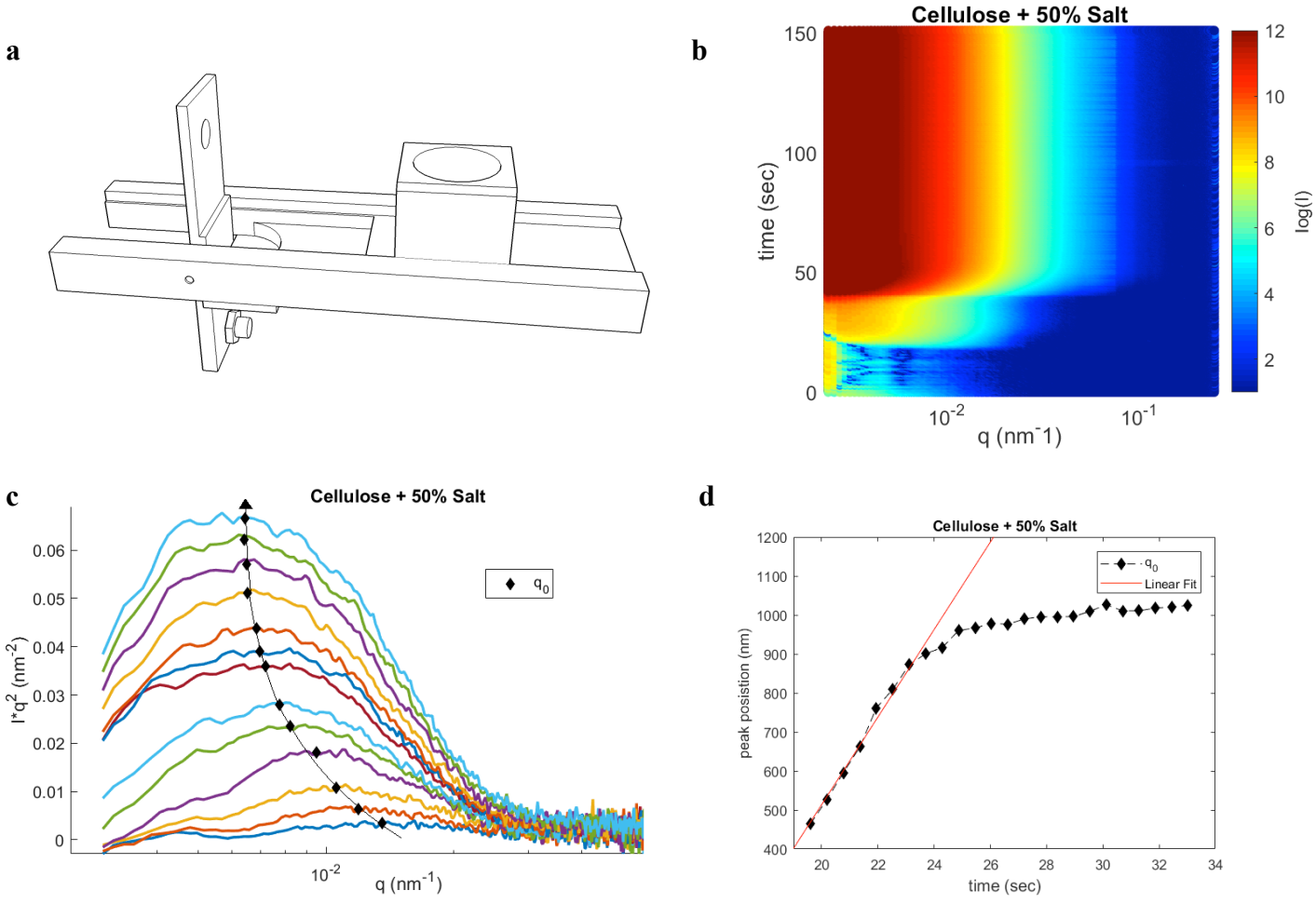

Figure 2: (a) schematic of drawdown system used to measure film dynamics (b) complete dynamics data captured for a phase separating film (c) 1-D U-SAXS patterns showing the peak increasing in intesntiy and moving to lower q in time (d) peak position in real space versus time showing a linear relationship indicative of spinodal decomposition

1. Anderson, V. . & Jones, R. A. . The influence of gelation on the mechanism of phase separation of a biopolymer mixture. *Polymer (Guildf).* **42,** 9601–9610 (2001).
2. Burg *et al*. Structurally isotropic ultra-white beetle scales and a synthetic analogue with similar structure and optical properties produced via spinodal phase separation. (In preparation)