ESRF	**Experiment title:** Polymeric Membrane Mesophases	**Experiment number**: SC 2143
Beamline: BM26	**Date of experiment**: from: 31 January 2007 to: 03 February 2007	**Date of report**: 27-02-2008
Shifts: 9	**Local contact(s)**: Dr. Kristina KVASHNINA	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Dr Giuseppe Battaglia - University of Sheffield

Mr Tom Smart* - University of Sheffield
Miss Miriam Flores-Merino* – University of Sheffield
Dr Jon Howse* - University of Sheffield

Report:

There is currently a great deal of interest in the self-assembly of amphiphilic block copolymers and the nano-structures formed by them. The aim of this experiment was to map a phase diagram of the structures and mesophases formed with respect to molecular weight and concentration. Eight amphiphilic block copolymers with the formula poly(ethylene oxide)n-b-poly(butylene oxide)m were chosen to have a range in molecular weight whilst all having an ethylene oxide volume fraction (ϕ_E) of ~0.3. The 8 block copolymers used had a range from 2-13 kgmol-1 and each was used to make 10 samples with a concentration range 5-90% w/w polymer in water.

This experiment was highly successful and provided some very interesting results.

Firstly the structural peaks were assesed to obtain the stuctures and mesophases formed with increasing molecular weight and concentration. The results from this are shown in figure 1. This data revealed a novel bicontinuous mesophase of hexagonal perforated lamellar in the 30-40% w/w region of higher molecular weight polymers used.

Following on from constructing the phase diagram, the composition of the concentrated lamellar phases was compared to the dispersed vesicle (lamellar membranes) phases. The SAXS data was analysed to determine the electron density profiles through the layers. Firstly we expected the hydrophobic domains in the vesicle membranes to have the same thickness

as in the lamellar sheets. Secondly we expected to see the hydrophilic domains in the lamellar sheets swell with increasing water content, whilst the hydrophobic domais remained constant.

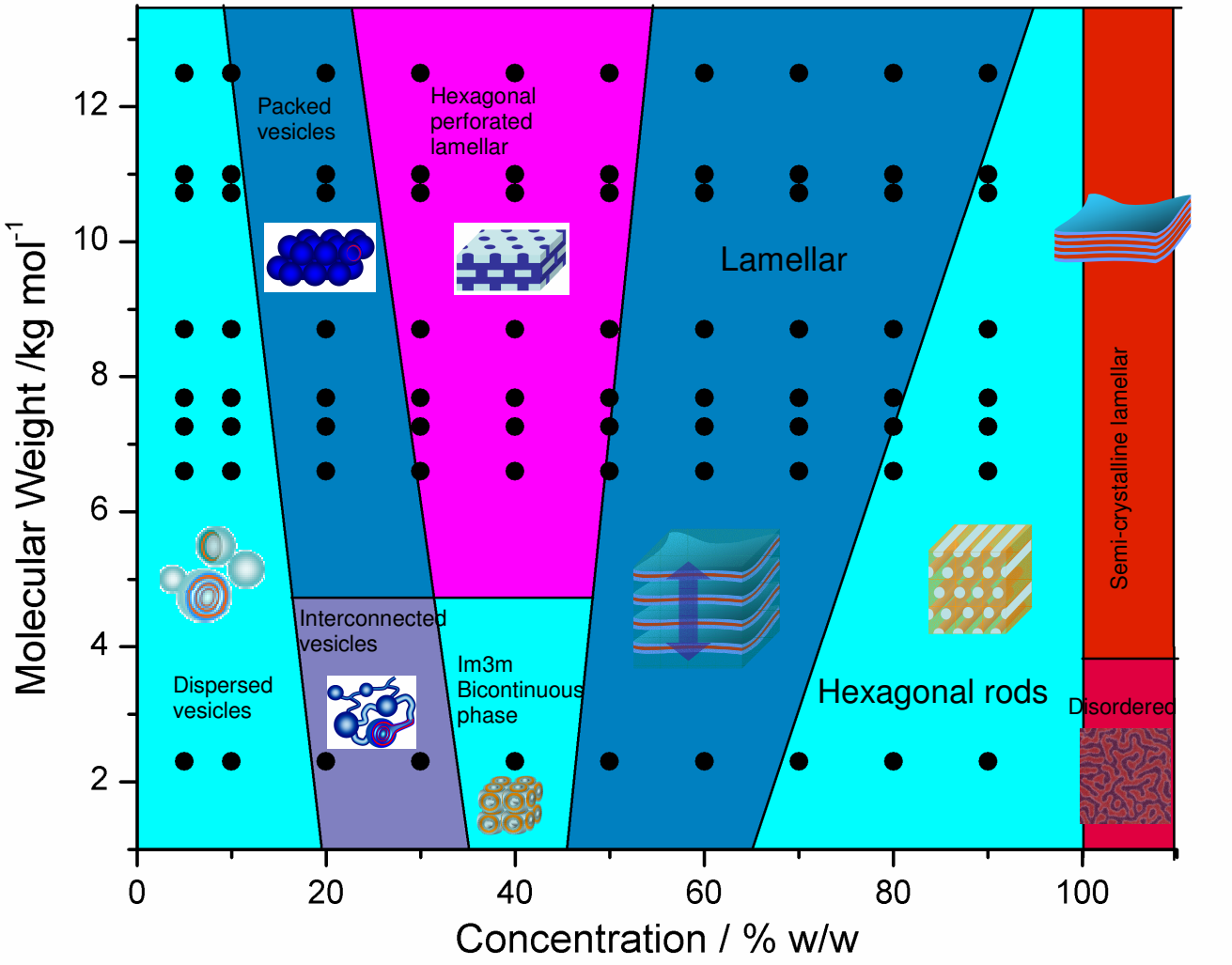

Figure 1: The above phase diagram shows the structures derived from the SAXS data collected. Each black point signifies a sample run at the ESRF. The red section at 100% concentration refers to the solid state structure.

The electron density calculations derived from the SAXS data have provided some excellent results and are currently being writen up for publication.

This experiment, as well as providing very useful information on the position of boundaries between the various mesophases possible, also led to a second project exploring the electron density profiles through the different sructures. This experiment has shown that it will be feasible to study this system on BM26 in the future investigating the controlled movement from one phase to another; primarily the reversible transition from dispersed vesicles to interconnected vesicles by the addition of a homopolymer.