

Experiment title:
Formation of Regular Three-Dimensional Polymeric Structures by Core-Shell Latex Particles as Investigated by SAXS

Experiment number:
SC-594

Beamline:
ID02A

Date of experiment:
from: 27-Nov-99 to: 29-Nov-99

Date of report:
29-Aug-2000

Shifts:
6

Local contact(s):
NARAYANAN Theyencheri

Received at ESRF:

Names and affiliations of applicants (* indicates experimentalists):

Ballauff, Matthias
Dingenouts, Nico*
Rosenfeldt, Sabine*
de Robillard, Quentin*

Address: Polymer-Institut, Universität Karlsruhe, Kaiserstr. 12
76128 Karlsruhe, Germany

Report:

We studied the formation of three-dimensional structures by Latex particles during the process of film formation. As we have shown in our previous work [1], the first step in the process of film formation, the water evaporation, is the most important one for the structure of the final film. So the first step in a film formation study of latex particles should always be the examination of the ordering (fig. 1) achieved in the first step.



Fig.1: Possible Ordering after the first step of film formation

We studied different latex consisting of the same core polystyrene (PS), but varying in the thickness of the shell of poly(methylmethacrylate) (PMMA). Out of the scattering curves of the latex systems in dispersion, we determined the radius of the core to 47 nm, the thickness of the shell layed in between 2 and 10 nm. The polydispersity could be determined out of the scattering data as well, the core has a standard deviation of 6.5%, the total systems one between 5.0% and 6.5%. This number is very small for usual latex systems, so that with these systems, there should be a good possibility to achieve well ordered systems in the dried state.

For the dried latex systems, we changed systematically the velocity of the drying process by changing the temperature and the pressure. With temperatures far away from the glass temperature (PS=100°C, PMMA=110°C) we achieved a wide range of drying times between 2 hours and 30 days. Additionally, we dried some systems at one higher temperature (85°C).

Systems dried at higher temperature show a lower degree of ordering, but all other systems behave similarly, no non-ambiguous effect of the drying velocity was found. All systems were measured at at least 6 different positions to get an impression of the homogeneity of a sample. All of them show areas with a kind of a glassy ordering (fig.2 left), but also, less frequently, areas with crystalline ordering (fig.2 right).

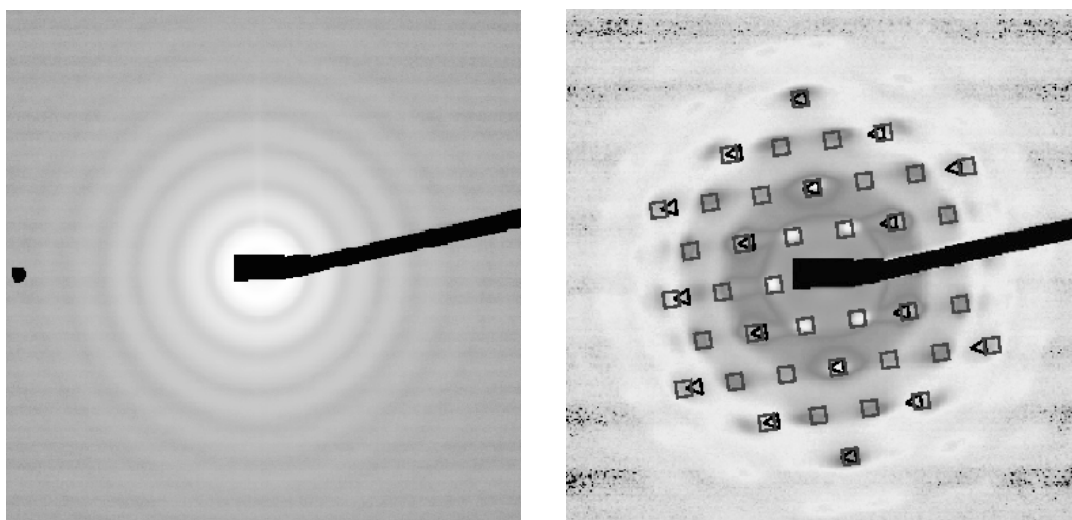


Fig2: 2D-Scattering images from different regions of a dried latex system. Left: glassy region; Right: crystalline region, structure can be described by a combination of two orientations of a fcc-lattice.

Even the regions with a radial symmetric scattering image show a pronounced structure factor. The first peak of $S(q)$ has a height between 5 and 10, so we have an extremely ordered glassy state. All crystalline regions could be described with an fcc-lattice, seen from two typical orientations ($\{111\}$ and $\{1\bar{1}0\}$), (fig. 2)). The size of the crystalline regions we found lies in between 0.2 and 1mm.

We achieved to produce crystalline regions in macroscopic sizes (1mm), but the greater part of the sample shows a glassy ordering. So we still have to improve the ordering in the first state of the film formation process in future, e.g. by lowering the polydispersity or by changing the stabilization of the latex to avoid clustering during the drying process. Another point is to ensure that we have the same structure after deforming the particles. But in conclusion, we can say that it is possible to produce nanostructured materials by film formation of polymeric latexes.

References

- [1] Dingenouts N, Ballauff M (1998) *Macromolecules* **31** 7423