Thermal stability of spin-coated colloidal PS films

G. Herzog\(^1\), A. Buffet\(^1\), M. M. Abul Kashem\(^1\), V. Körstgens\(^2\), G. Benecke\(^1\), J. Perlich\(^1\), R. Gehrke\(^1\), M. Schwartzkopf\(^1\), A. Rothkirch\(^1\), P. Müller-Buschbaum\(^2\), W. Wurth\(^3\) and S. V. Roth\(^1\)

\(^1\) HASYLAB at DESY, Notkestr. 85, 22607 Hamburg, Germany  
\(^2\) TU München, Physik-Department E 13, James-Franck-Str. 1, 85747 Garching, Germany  
\(^3\) Institut für Experimentalphysik, Universität Hamburg, Luruper Chaussee 149, 22761 Hamburg, Germany

Installation of large scale colloidal nanoparticle thin films is of great interest in sensor technology or magnetic storage [1]. Among the methods used are dip-coating, spin-coating and spray deposition. Often, such devices are operated at elevated environmental or thermal conditions.

In this experiment we investigated in-situ the effect of heat treatment on the structure of spin-coated colloidal solutions of polystyrene nanospheres by grazing incidence small angle x-ray scattering (GISAXS) and optical microscopy. An aqueous solution of polystyrene nanospheres with a nominal diameter of 100 nm was spin-coated onto base-cleaned silicon wafers. At the beamline BW4 [2] at HASYLAB, GISAXS measurements at a wavelength of 0.138 nm were performed at room temperature, at 100 °C, at 110 °C and after cooling the sample down again to 25 °C using a 2D detector. The resulting scattering patterns are shown in fig. 1. Out of such 2D patterns horizontal (out-of-plane) cuts were taken which indicate characteristic lateral length scales on the sample surface and are shown in fig. 2.

As can be seen in fig. 1, annealing leads to an increasing intensity along the vertical \(q_z\) axis while the intensity of the side peaks in horizontal \(q_y\) direction decreases, especially when the sample is heated to 110 °C which is above the glass transition temperature of PS. The vanishing of the peaks in horizontal direction is interpreted as a loss of long range order in the spin-coated films and it is irreversible, i.e. the peaks do not reappear after cooling the sample to 25 °C. Our model uses an increased mobility of the nanoparticles to explain this behaviour [3].

Figure 1: GISAXS images at room temperature (left), at 100 °C (middle) and at 25 °C (right) after heating to 110 °C
Figure 2: Out-of-plane cuts before, during and after heating

References