	Experiment title: SAXS investigations on colloid suspensions confined in periodic microcavities	Experiment number: SI-1064
Beamline: ID02	**Date of experiment**: from: 10.02.2005 to: 14.02.2005	**Date of report**: 31.8.2005
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Report:

In this experiment we measured the confinement effect of colloid solutions in microcavities. In a previous experiment at BM05 we had shown that the experiment was possible, but a different experimental approach using a 2D detector was needed to obtain complete data of the system. We used the setup shown on the left side of Fig.1: several microcavity arays etched in a Si chip were used to confine a colloid of 110 nm-diameter SiO_2 spheres in an alcohol mixture with a concentration of about 10% in volume. A beam of 12.4 keV was

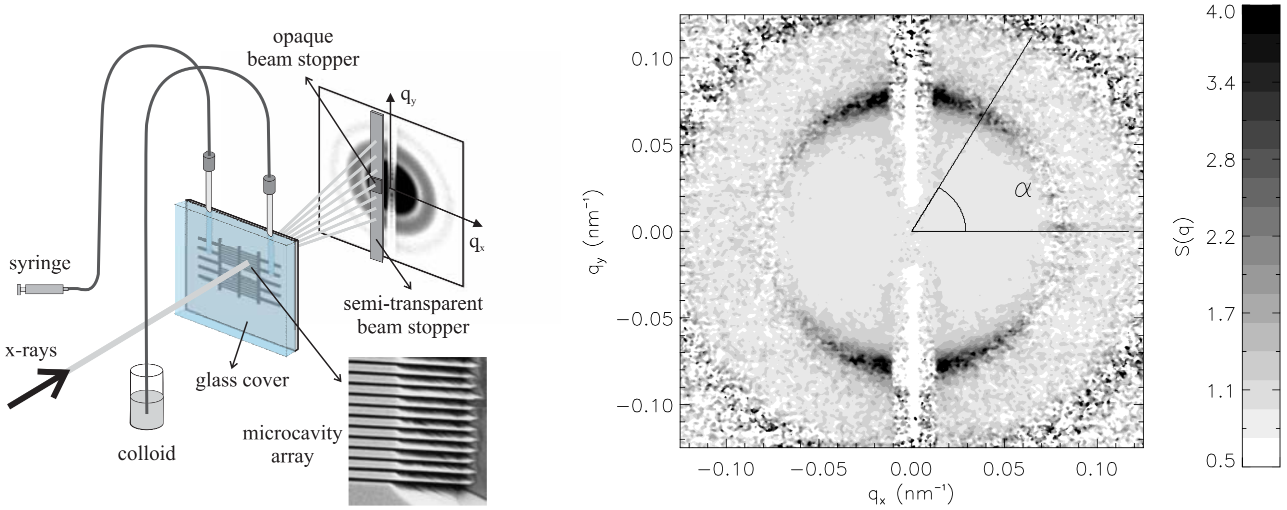

***Fig. 1**: Setup of the experiment with a SEM picture of one microcavity array (left). Measured structure factor of the colloid confined in a microcavity array. The structure factor was obtained by dividing a measurement through an array by another measurement through a big cavity etched in the chip.*

used in a transmission geometry and the scattered intensity was detected with the 2D detector. A semi-transparent stripe beam stop in the vertical direction was used to decrease the intensity from the diffraction peaks of the microcavity arrays, and an opaque central beam-stop was blocking the primary beam. The setup allowed SAXS measurements of the colloid confined in the microcavities, which had gap sizes from 1000 down to 300 nm.

The measured structure factor S(q) of the colloid in confinement is shown on the right side of Fig. 1. The S(q) is circular, as expected for a liquid, but much stronger peaked along the confinement direction. The increasement of S(q) with α is plotted in Fig. 2, where α is the angle shown in Fig. 1. The position of the peak does not change at different directions, but the intensity clearly increases close to the confinement direction (α = 90°). The error bars in the plot arise from the standard deviations of the integration of the

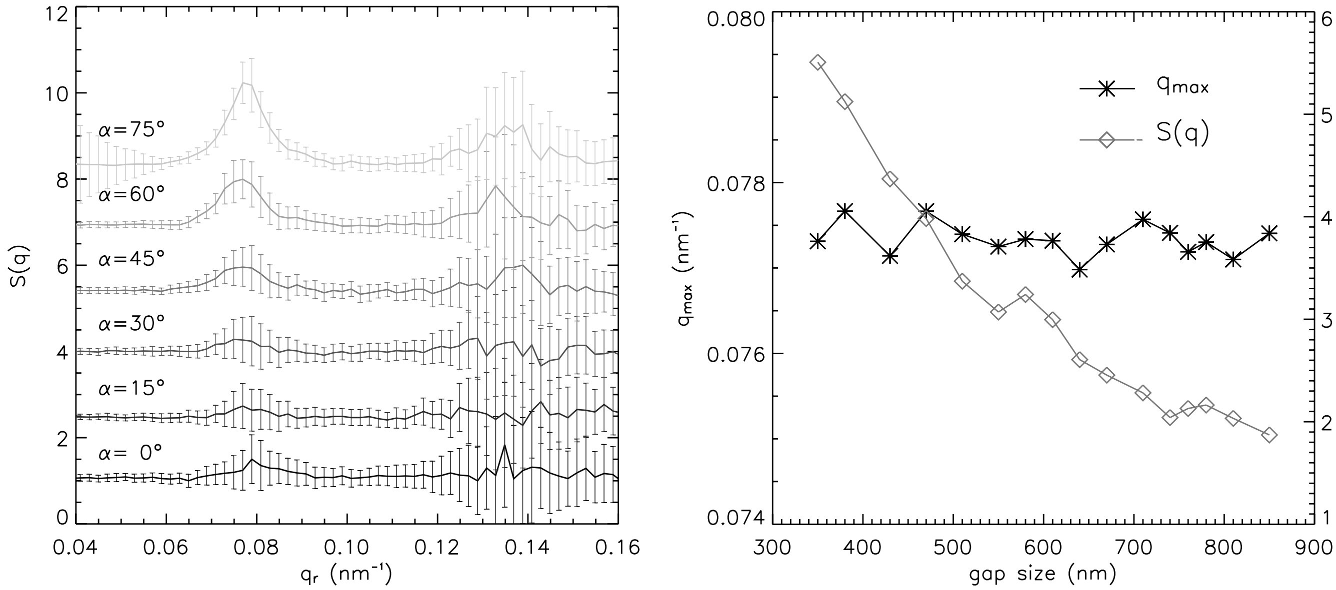

Fig. 2: *S(q) along different directions: α = 90° corresponds to the confinement direction and α = 0° to the in-plane direction.*

signal in angular sectors along the different α. On the right side of Fig.2 we plot the position of the S(q) peak and the peak intensity as a function of the gap size. The peak positions remain approximatelly constant at all gap sizes (q_{max} = 0.078 nm^{-1}) corresponding in real space with √2R, R being the radius of the colloid. On the other hand, the peak intensity increases as the cavity gap size decreases, showing a clear confinement effect.

Our interpretation of the data is that small clusters of colloids are formed next to the confining walls of the microcavities due to a fcc- or hcp-like nucleation of the colloids in a preferred <111> orientation perpendicular to the confining walls (see Fig. 3)

In summary, our experiment at ID2 has provided unique SAXS measurements of a colloid in confinement. Therefor we consider this experiment a complete success. A publication containing the results is presently under preparation. We thank the beam line staff of ID02 for their excellent support.

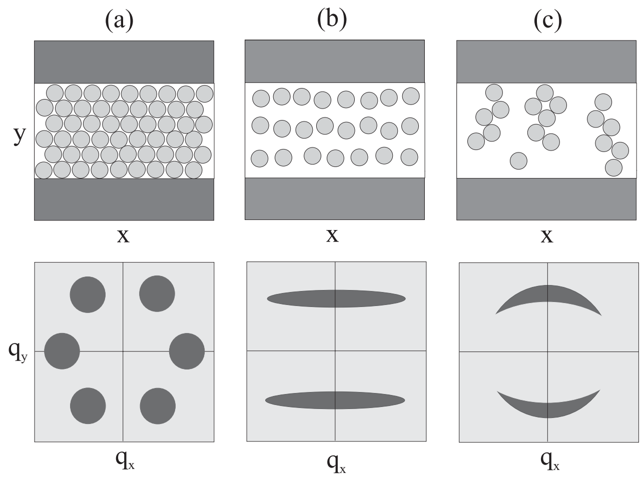

Fig. 3: *Three different models of colloid in confinement and their expected Fourier space map: crystallization (a), layering (b) and nucleation in the confinement direction (c).*