	Experiment title: **Influence of substrate modification on the 3D structure of colloidal photonic crystals**	**Experiment number**: 26-02-444
Beamline: BM-26B	**Date(s) of experiment**: 31.08.2008 – 02.09.2008	**Date of report**: -09-2008
Shifts: 6	**Local contact(s)**: **Dr Kristina Kvashnina**	

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Report: (max. 2 pages)

Let us first of all stress that microradian x-ray diffraction experiments are challenging and time-consuming as they require a significant re-building of the setup and careful alignment of the beamline optics to reach the world-best angular resolution. Only this usually requires about 4 shifts of the beamtime. It should be pointed out that it is simply ***impossible*** to perform such a challenging experiment within 2 beamdays, which were assigned to this experiment.

To make at least some work possible, our experiments 26-02-444 and 26-02-448 were scheduled in a row after a shutdown, thanks to the effort of our local contact. In this way we had a chance to make use of the buffer days (August 26^{th} – 28^{th}). A part of the experimental team has arrived to the ESRF already on Monday, August 25^{th}, to start preparation for the experiments.

Unfortunately, technical problems came into our way. Despite hard and busy work of the beamline staff, no beam was available in the experimental hutch till Friday evening, 29^{th} of August. Even then, no proper beam focusing was achieved, which indicated that the phase front of the beam was strongly distorted. Varying the x-ray photon energy to improve the focusing did not help either. The achieved resolution and the beam intensity after the lens were several times worse than usual (see the report of the experiment 26-02-448 for more details). On Friday, late in the evening, we have decided to collect some (low-quality) data instead of loosing the beamtime completely.

The x-ray diffraction experiment was performed for a number of colloidal crystals fabricated by the vertical deposition technique. To study the effect of the substrate modification, the data were collected from crystal grown on insulating glass substrates as well as on conducting Au-covered glass and ITO (tin-oxide) substrates. Figure 1 shows a couple of examples of the measured diffraction patterns obtained from a crystal of silica spheres, which was grown on an non-conductive glass substrate by the vertical deposition method. Although the resolution was sufficient to resolve the diffraction peaks, important details such as the intrinsic width of the reflections is not accessible from these data. A full rotation series was measured, i.e. the patterns were taken as a function of the sample orientation, with the step size of 1 degree.

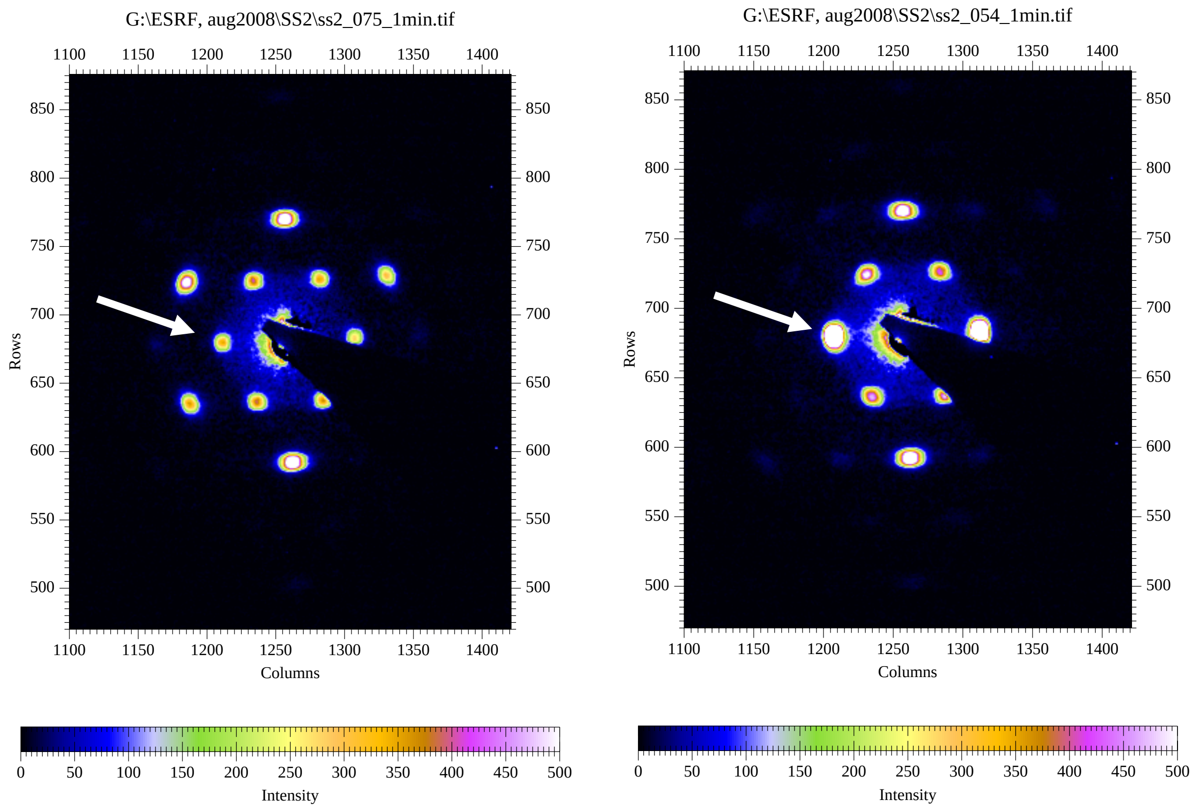

Figure 1. X-ray diffraction patterns measured for a crystal of silica spheres (diameter = 410 nm) at normal incidence (left) and after sample rotation by ω = 20 degrees around the vertical axis (right). The arrows point onto the 't1' reflection (see Fig. 2).

For several samples we have performed the analysis of the dependence of the peak intensity *I* on the sample rotation angle ω. An example of the result this analysis for the 't1' reflection (see Fig. 1) of the crystal of silica spheres is shown in Fig. 2. At the moment we do not yet understand this result. It does not correspond to the face-centred cubic (fcc) structure, which was found in crystals of polystyrene particles [1] in our previous experiment 26-02-392, since the couple of the first maxima is not observed at ±19.5° (expected for fcc) but at about ±24°, which is close to the position expected for a random-stacking hexagonal close packed (rhcp) crystal. At the same time, a too low intensity at ω = 0 excludes an rhcp structure. We were unable to fit the data within Wilson's theory, which describes the intensity profile along the Bragg rods for crystals with an arbitrary percentage of fcc-type planes. We also failed to interpret this dependence as a result of a crystal distortion [2] since the secondary maxima (at |ω| between 55° and 60°) are shifted much less than the primary maxima. The dependence cannot be either understood as an effect of the crystal thickness. At the moment we are left with a dilemma whether a result of this type could have been caused by the problems in the beam.

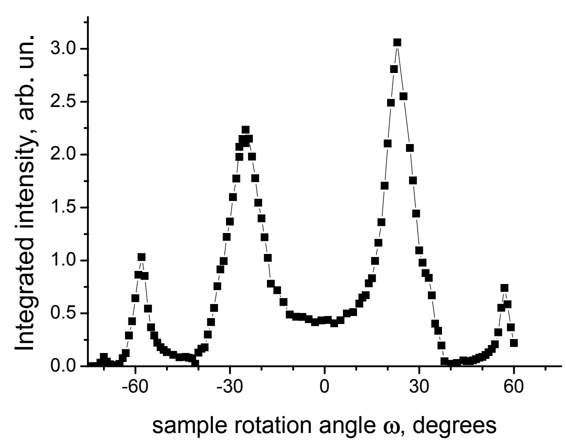

Figure 2. Dependence of the intensity of the 't1' reflection on the rotation angle ω.

[1] J. Hilhorst, V. Abramova, A. Sinitskii et al., submitted.
[2] J.H.J. Thijssen, PhD thesis, Utrecht University (2007); J.H.J. Thijssen et al, to be published.