ESRF	**Experiment title:** Determination of stacking disorder in colloidal photonic crystals	**Experiment number**: 26-02-421
Beamline: BM26B	**Date of experiment**: from: 10-04-2008 to: 14-04-2008	**Date of report**: 28-05-2008
Shifts: 12	**Local contact(s)**: Dr. Kristina Kvashnina	*Received at ESRF:*

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Report:

Our experiment was scheduled after the spring shutdown of the ESRF. Three of us have arrived earlier and have started preparation for the experiment on Monday, April 7th. We have also made use of the buffer days, April 8th and 9th. In these time the setup was built and aligned together with the beamline personnel. Moreover, Dr. Wim Bras asked to check the functionality of the microradian setup with lenses at a higher photon energy E_{ph}. Since the refraction index contast drops down as $1/E_{ph}^2$, one needs more lenses to focus the x-ray beam at the same distance. An additional set of compound refractive lenses with N=31 lenses was prepared. Test measurements were performed at a photon energy of E_{ph} = 24 keV (λ=0.5 Å) and a sample-detector distance of about 6 metres.

Fig. 1 presents an example of a diffraction pattern measured from a silicon grid (which is usually used as a calibration sample in our experiments). A number of test measurements on several colloidal crystals were also performed. Our results demonstrate that microradian resolution can be achieved at higher photon energies.

However, since the resolution in reciprocal space was compromised at higher energy, the rest of the experiment was performed using E_{ph} = 13.5 keV (λ=0.92 Å), a set of N=7 lenses and a sample-detector distance of 8 metres. The data was collected using the Photonic Science CCD detector (22 microns pixel size) and the SESO camera (7 microns pixel size) was used for alignment.

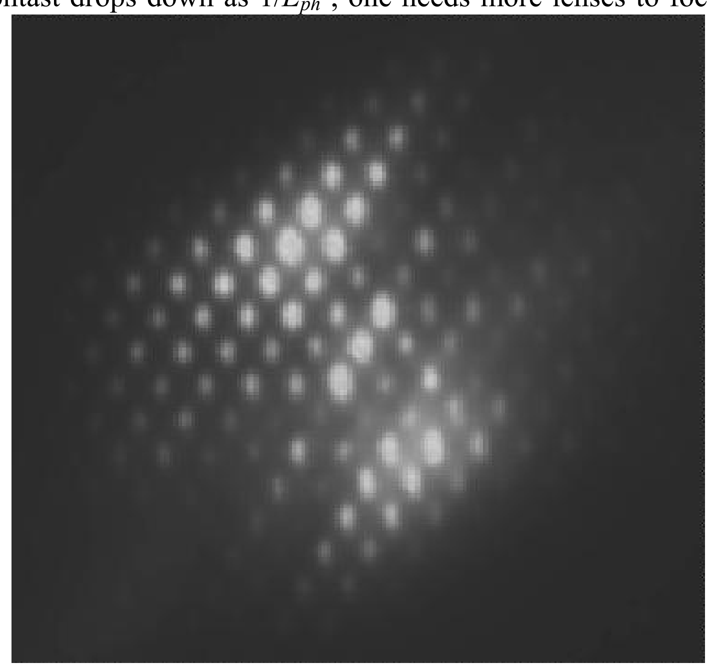

Fig.1 An example of the microradian diffraction pattern from a silicon grid with a hexagonal set of pores. The distance between the pores is 1.6 micron.

In the main experiment we gathered

diffraction patterns of colloidal crystals of silica (SiO_2) colloids, which had been left to sediment in gravity for up to three weeks. Samples had been prepared in rectangular glass capillaries of inner dimensions 100 x 2 x 0.1 mm. After filling the capillaries they were sealed. Next, they were stored in upright position in order for the colloids to sediment and crystallize at the lower ends of the capillaries.

The colloids used were all SiO_2 spheres of similar sizes, around 1 micron, but of different polydispersity: Three different synthesis batches were used in sample preparation, each with a slightly lower polydispersity than the previous batch (3%, 1.5%, and lower than 0.5%).

One type of suspension medium consisted of a mixture of dimethyl sulfoxide (DMSO) and water, which matches the refractive index of the SiO_2 particles, so that their real-space structure can later be studied with confocal microscopy as well. The other type suspension medium was pure ethanol, in which particles are not refractive index matched, but experience a slightly higher gravitational force, due to the lower density of ethanol compared to SiO_2. Also, the boiling point of ethanol is far lower than that of DMSO/water, so a dry sediment can easily be obtained by opening a capillary and letting the ethanol evaporate.

These three polydispersities and two solvent media account for six different types of samples, all of which small angle X-ray diffraction patterns were taken of at different sample orientations and different positions. Since the CCD detector has a limited dynamic range (i.e., the difference between the highest and the lowest intensity, which can be reliably determined from a single diffraction pattern), all the measurements are performed with several exposure times. The shortest exposure data allow to quantify the strongest reflections while longer exposure data can be used for weaker higher-order spots.

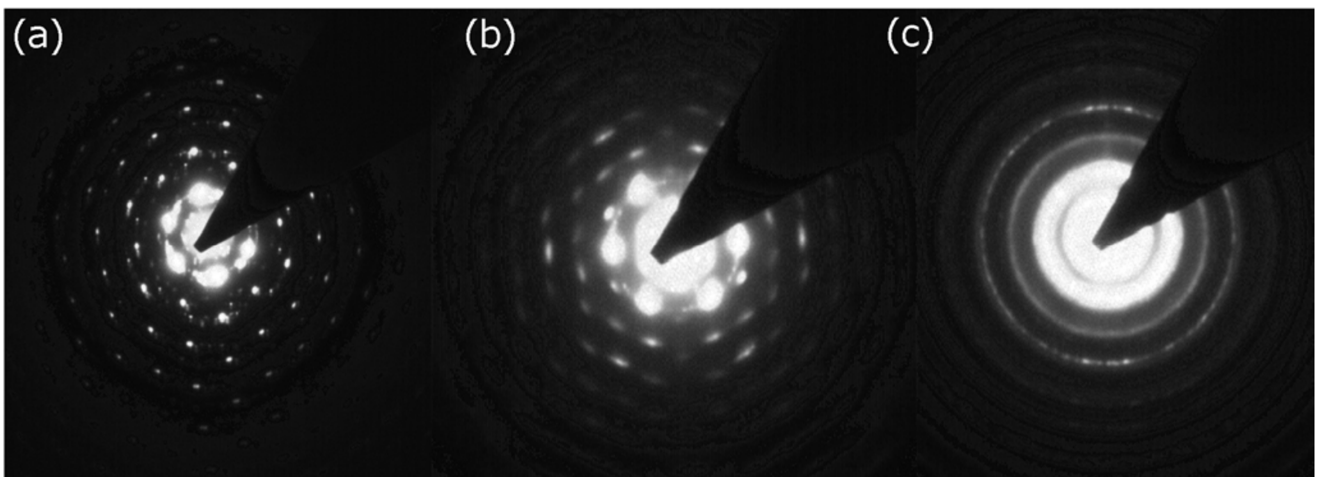

Fig. 2 Diffraction patterns of silica colloid suspensions sedimented under gravity. (a) shows the diffraction pattern of colloids with a polydispersity lower than 0.5%, (b) 1.5% and (c) 3%. Suspension medium was a mixture of dimethyl sulfoxide (DMSO) and water. Exposure time in all images is 1 minute. The black trangle is the beamstop used to block the direct beam.

Figure 2 displays some typical diffraction patterns obtained from the samples suspended in DMSO/water. The particles used to obtain these diffraction patterns have an increasing polydispersity. Less than 0.5% in Fig 2a, 1.5% in Fig. 2b and 3% in Fig. 2c.

Whereas in Fig. 2a many clear sharp diffraction spots can be seen, the spots are much broader and less intense in Fig. 2b and in Fig. 2c only Debye-Scherrer rings are observed. Also visible in Fig. 2a are 'out of place' diffraction spots. The main hexagonal pattern is believed to be caused by a larger crystalline domain of the sedimented colloids, whereas the 'out of place' spots are believed to be caused by a second domain with a slightly different crystal orientation. From the width of the diffraction maxima and rocking curves we hope to quantify the effect of polydispersity on the crystal order in more detail. The results are expecrted to be of crucial imporatnce for the understanding of the influence of the particle polydispersity on the quality of photonic materials, which can be produced using the colloid self-assembly.