DUBBLE	**Experiment title: In-plane order and 3D structure of colloidal crystals and inverse opals prepared by vertical deposition**	**Experiment number:** **26-02-392**
Beamline: BM-26B	**Date(s) of experiment:** From: 23-08-2007 To: 25-08-2007	**Date of report:** 19-09-2007
Shifts: 9	**Local contact(s):** Dr Kristina Kvashnina	
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Report: (max. 2 pages)

To allow for a more effective use of the beamtime, our experiment was scheduled after the shutdown so that we were able to make use of the buffer days for the construction and the alignment of the microradian setup. 13 keV x-rays and a set of 7 compound refractive lenses were used. The second crystal of the monochromator and the mirror were unbent to avoid beam focusing before the experimental hutch. Instead, the beam was focused by the lenses, which were installed just before the sample. The adjustment of the beam focusing was performed by a fine tuning of the photon energy, which affects the focal length of the lens (proportional to the square of the photon energy) with simultaneous recording of the beam profile by a high resolution Sensicam camera (pixel size 0.67 micron) borrowed from BM-05 beamline. The data collection was performed by the Photonic Science camera (pixel size 22 micron) available at DUBBLE. Thanks to the excellent support of the local contact and Dirk Detollenaere we were able to start the data collection already on 23rd, the last buffer day.

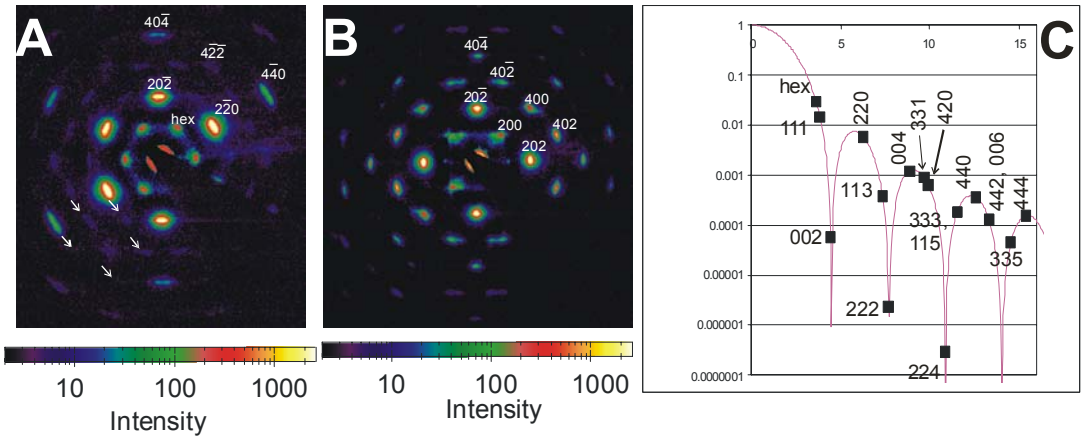

Figure 1 Microradian diffraction patterns from an fcc cubic crystal measured with the beam normal to the substrate (111 crystallographic direction, panel A) and after the sample rotation by 55 degrees around the vertical axis (002 crystallographic direction, panel B). Panel C shows the form factor of uniform spheres (lines) and its value at the position of several Bragg peaks (points). The horizontal axis presents the values of qR (R is the sphere radius) and the vertical axis shows scattering intensity $I(q)$ for a single particle normalized by $I(q \to 0)$. 'hex' in panels A and C denotes the intersection of the lowest-order Bragg rod by the Ewald sphere at normal incidence. White arrows in A point onto examples of intersections of higher-order Bragg rods.

The microradian setup was used to study colloidal crystals fabricated using the vertical deposition technique as well as inverse opals made using various materials. In particular, Figure 1 presents examples of the diffraction patterns measured from a colloidal crystal of polystyrene spheres (diameter 425 nm)

fabricated using the vertical deposition technique at normal incidence (panel A) and after sample rotation by 55 degrees. As will be illustrated below, the crystal possesses face-centred cubic (fcc) structure.

In panel A, the hexagonally arranged set of reflections reflects the 3-fold symmetry along the 111 direction normal to the substrate surface. In addition to the well-pronounced fcc reflection, one can also see additional reflections, which are forbidden for a perfect fcc crystal. They originate from intersections of the Ewald sphere by so-called Bragg rods, which are induced by stacking disorder in the direction normal to the substrate. The intersections of the lowest-order Bragg rods (denoted by 'hex') have the highest form factor and are clearly visible. Some weak higher-order Bragg rods can be also seen (see white arrows). In panel B, a pattern measured after a sample rotation by 55^o around the vertical axis is shown. Here the x-ray beam propagates along the 002 crystallographic direction. The underlying 4-fold cubic symmetry can be easily seen. The relative intensity of various reflections can be qualitatively understood using panel C, which displays the variation of the form factor of the spheres. In this model calculation the spheres were assumed to possess uniformly distributed electron density. For example, the low-order 200 reflection appears very close to the first minimum of the form factor and is indeed very weak in the measured pattern presented in panel B.

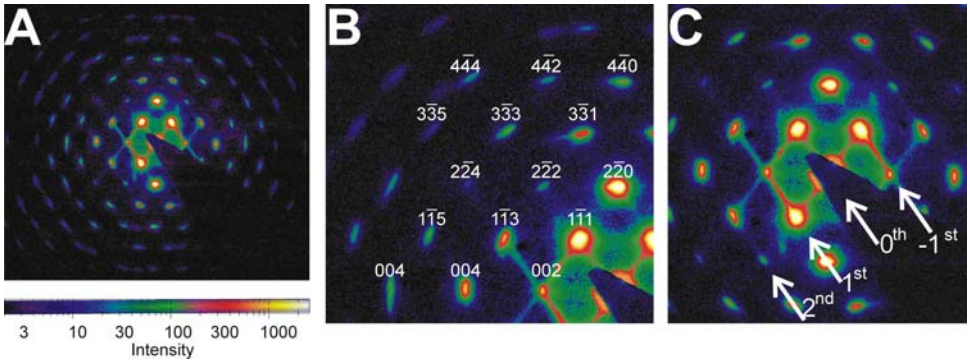

Figure 2. Panel A presents a microradian diffraction pattern measured after sample rotation by 35 degrees around the vertical axis. Panel B shows a zoom into one quarter of the pattern along with assignment of several low-order reflections. Panels C presents the central part of the pattern. Arrows point onto several Bragg rods, making an angle of 70.5 degrees with the substrate normal.

An interesting result was observed at the angle of rotation of 35 degrees when the beam propagates along the 220 crystallographic direction (Figure 2). Panel A displays a full diffraction pattern while a zoom into one quarter is given in Fig. 2B along with the crystallographic assignment of the peaks. Again, the relative weakness of the 002, 222 and 224 reflections can be understood on the basis of Fig. 1C. Moreover, a closer look on the central part of the pattern reveals an unexpected result: presence of well-pronounced lines in between the peaks running in the 1$\underline{1}$1 and 1$\underline{11}$ directions. These lines are the Bragg rods, which are caused by the presence of the stacking disorder. Till now, the stacking disorder in such samples was only detected in the direction perpendicular to the substrate (as seen in Fig. 1A). We now find that in a crystal with predominant fcc structure, some (small) amount of stacking faults is present also in the other 111 directions. Interestingly, the crystal possesses only one stacking direction (e.g., of the ABCABC type) and does not have a twin (stacked ACBACB). A single stacking fault would change the stacking direction (e.g., ABC**A**CBA, where the bold letter **A** denotes a layer with hcp stacking environment). Absence of the twin reflections along the rods shows that only double stacking faults are present (e.g., ABC**AC**ABC; two layers **AC** have hcp environment).

Results presented above illustrate the high potential of the microradian setup at DUBBLE for the characterization of structure and (dis)order in colloidal and photonic crystals. In addition, during the experiment we have proved presence of large domains of cubic colloidal crystals with their 200 face parallel to the substrate. Moreover, we have studied in detail various inverted crystals based on WO_3, TiO_2, SiO_2, Fe_2O_3, Ni, Co. These data will be analyzed to extract information on the structure and order parameters (stacking disorder, positional and bond-orientational correlation lengths) in the samples.

Finally, we would like to thank our local contact Dr Kristina Kvashnina and Dirk Detollenaere for their excellent support.