DUBBLE	**Experiment title: Accessing disorder in colloidal crystals: Pushing the resolution limits of x-rays**	**Experiment number**: **26-02-384**
Beamline: BM-26B	**Date(s) of experiment**: From: 12-06-2007 To: 15-06-2007	**Date of report**: 9-July-2007
Shifts: 12	**Local contact(s)**: Kristina Kvashnina	
Names and affiliations of applicants (*indicates experimentalists): **A.V. Petukhov*, P.S. Miedema* (van 't Hoff lab, UU); D.C. 't Hart*, J.C.P. Stiefelhagen*, A. Imhof* (scm lab, UU); A. Snigirev**		

Report: (max. 2 pages)

Our experiment was scheduled after a shutdown at the ESRF. For a more effective use of the beamtime we have made use of the buffer days (12th and 13th) for setup construction and alignment. During the buffer days the synchrotron was operating in the usual 2/3 filling mode except for one night, when the current was extremely small. That night was not used for our experiments due to too low flux.

To build up our setup, we have utilised the mechanics pool (in total, 8 stages were borrowed) and the detector pool of the ESRF. Additional equipment was also borrowed form the BM-05 and ID-02 beamlines. The microradian setup consisted of the following components:

(1) The lens goniometer was able to position the beryllium lens (two translation stages for horizontal and vertical displacements) and to properly orient it (two stages to rotate the lens around two axes orthogonal to the beam).

(2) The sample goniometer had the same four computer-controlled degrees of freedom to study different places in the samples and to orient single colloidal crystals.

(3) Two additional detectors were installed on the detector trolley in front of the gas-filled SAXS detector. We have used the Photonic Science CCD detector (4008×2670, 22 micron square pixels at the phosphor screen), which is available at DUBBLE. Most data was collected by this detector. In addition, a high-resolution Sensicam detector (1280×1024, 1.8 micron square pixels) was installed to study beam focusing in greater details. In the last day of the experiment 26-02-376, this detector was replaced by a similar detector borrowed from the BM-05 beamline (1280×1024, 0.67 micron square pixels). Two additional translation stages were used to switch from one detector to the other and to move them away to be able to use the gas-filled detector.

To achieve highest possible angular resolution, we have unbent the second crystal of the monochromator and the mirror. Instead, the beam was focused at the detector position by the Be lens. This allowed us to achieve tighter focusing. Theoretically, the beam size on the detector is determined by the de-magnification ratio of the lens and size of the source (electron beam in the storage ring). The source size of the bending magnet is somewhat smaller in the vertical than in the horizontal direction so that one can expect the focused beam to be slightly smaller in the vertical direction. Indeed, this can be seen in the images of the direct beam taken with short exposures (of the order of a few milliseconds). However, we observe significant fluctuations of the beam position (mostly, in the vertical direction), which spoils our angular resolution to a significant extent.

The fluctuations of the direct beam position on the detector are illustrated in Figure 1. With the exposure time of 100 ms the fluctuations at frequencies higher than ~10 Hz are averaged out. As a result, the beam already appears significantly broadened in the vertical direction. At the same time, significant fluctuations are also present at frequencies below 10 Hz, which led to a remarkable variation of the beam position in between the measurements shown in Fig.1. By collecting data similar to the one presented in Fig. 1 we have also studied the effects of cryotiger (used to cool the second crystal), turbo vacuum pump of the monochromator, pump of the fly-pass tube, and other elements on the beam instabilities.

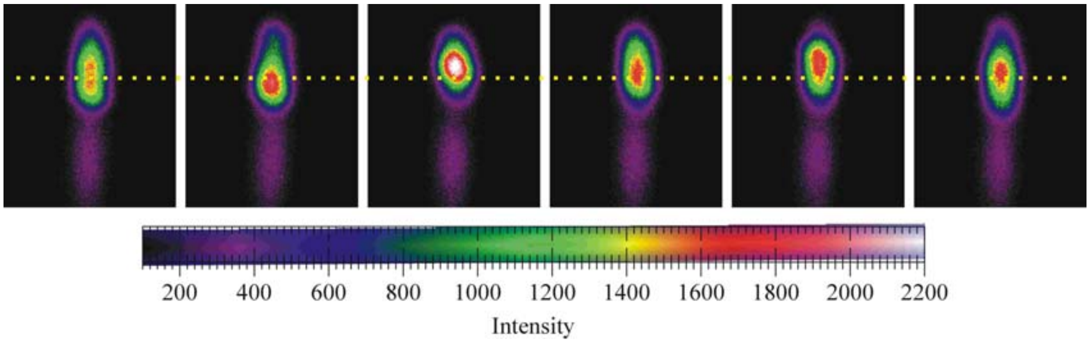

Figure 1. A sequence of 6 subsequent measurements of the focused direct beam using the Sensicam detector from the ESRF detector pool. The exposure time was 100 ms. For every image we have zoomed into exactly the same area of the dimensions (300×360) μm^2 on the detector screen corresponding to (37×45) μrad^2 in angular terms or about (0.0025×0.003) nm^{-2} in the reciprocal space. The dotted yellow line highlights the same vertical position in all the images.

Moreover, Guy Luijckx has made an enquiry on the stability of the electron beam in the storage ring. According to the information provided by the people operating the synchrotron, our problems cannot be caused by the storage ring instabilities and are probably related to the mechanical vibrations at the beamline.

In addition, we have observed another effect, which is also visible in Fig. 1. In addition to the main focused beam, there is a weaker satellite peak (in Fig. 1 below the main beam). The main and the secondary peaks always have practically the same horizontal position while their relative vertical position sensitively depends on the ‘mpitch’, which controls the relative orientation of the first and the second crystal of the monochromator. The secondary peak is usually broader than the main peak. We have seen a similar effect in our experiment in the C hutch of the ESRF beamline ID-10A TROIKA. The only difference was related to the fact that the C end-station uses a monochromator with a horizontally placed channel-cut Si crystal. As a result, the main and the secondary peaks were displaced relative to each other in the horizontal direction. The secondary peak must obviously arise from the monochromator but we do not understand yet its origin.

The horizontal width of the reflections in Fig. 1 is about 40 micron. For the conditions of the present experiment this width corresponds to the angular resolution of about 5 microradians or a resolution of δq = 0.00035 nm^{-1} in the reciprocal space. We also note that the angular resolution in the data collected by the Photonic Science detector is further deteriorated by the detector resolution to about 7 microradian in the horizontal direction. Vertically, the actual resolution of the order 10 μrad is even worse due to the beam instabilities. The setup constructed and aligned in the present experiment was also used in the following experiment 26-02-376.

To summarise, with our microradian setup we are able to reach angular resolution of the order of 5 μrad. However, this achievement is partially spoiled by the detector resolution and the beam instabilities. As a result, the measured peak width in several colloidal systems of our interest is yet instrument-limited. To achieve further progress with the setup, further investigation of the beam instabilities is needed. Moreover, a detector with somewhat higher resolution is needed. The detector pool of the ESRF can only provide us with a detector with a too high resolution, which results in a too small field of view. A new detector with a pixel size of 5-7 microns on the phosphor screen (thus, actual resolution better than 15 microns) is highly desirable.

In addition to the studies of the beam instabilities, we have performed measurements of x-ray diffraction in various colloidal crystals and liquid crystals. Most efforts were invested in hard-sphere silica colloidal crystals with random-stacking hexagonal close-packed structure (rhcp). In particular, we were most interested in the recently discovered in-plane stacking disorder, which leads to broadening of the so-called Bragg scattering rods. In addition, we have performed preliminary measurements of several new colloidal and photonic systems as a preparation for the forthcoming experiments (planned in August 2007). Because of the space limitations, we cannot present more details in this report.

Finally, we would like to express our deep gratitude to our local contact Kristina Kvashnina, to Dirk Detollenaere, who possesses crucial expertise on many aspects related to the beamline operation, and to Guy Luijckx for retrieving the information related to the stability of the electron beam in the storage ring.