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ESRF

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- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** **Spatially-resolved high-resolution small-angle diffraction of colloidal crystals**	**Experiment number**: SC-1418
Beamline: ID-10C	**Date of experiment**: from: 12 May 2004 to: 17 May 2004	**Date of report**: 01.03.2005
Shifts: 15	**Local contact(s)**: A. Moussaid	*Received at ESRF:*

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Report:

The challenge of the application of small-angle x-ray diffraction for self-assembled colloidal crystals [1,2] stems from the 3 to 4 orders of magnitude difference between the typical size of the colloidal particles and the x-ray wavelength. To resolve the diffraction pattern, one needs an angular resolution well beyond 10^{-4} radian. Even more challenging is the determination of the intrinsic width of the diffraction peaks, which requires an angular resolution of the order of 10^{-6} radian.

Since the x-ray source of ID-10 is too large in the horizontal direction (about 1 mm), we have used a rather small opening of the primary slits (SS0) of 30 μm in both the horizontal and the vertical direction as illustrated in Fig. 1. The primary slits were acting as a secondary x-ray source for the rest of the setup. The slits opening was of the order of the transverse coherence length (at the position of the SS0 slits) in the vertical direction (theoretical value), but about one order of magnitude larger than the transverse coherence length in the horizontal direction. We estimated (assuming ideal optics in between the primary slits and the sample) the transverse coherence length at the sample position to be of the order of l_{tr}=100 μm and the beam size of a few hundreds of μm. Thus, we intentionally did not create the condition for fully coherent x-ray illumination to avoid the appearance of speckle patterns. Yet, we have minimised the gap between the coherence length and the beam diameter.

After the sample, the transmitted and diffracted x-ray beams are focused by a compound refractive lens (CRL) [3] positioned just after the sample. The lens position and its focal length are adjusted such that it creates an image of the secondary x-ray source (SS0) at the phosphor screen of the detector. For the given SS0-lens and the lens-detector distances, the zoom factor of the imaging system is about 1:9 such that the CRL should produce an image of about 3.3 μm of the 30 μm-wide SS0 slits. With a lens-detector distance of 3 metres one can therefore expect an angular resolution of the setup of the order of 1 μrad.

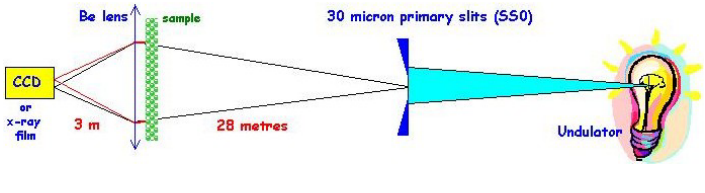

Figure 1. Sketch of the optical scheme used in the experiment. Additional optical elements such as a monochromator and a number of additional protecting slits, in between the primary slits and the sample, are not shown.

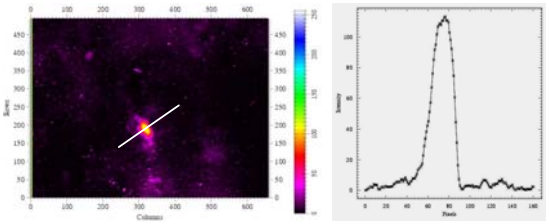

Figure 2. An x-ray film image (corrected for the density-exposure curve) of a (110) Bragg reflection of an rhcp crystal of silica hard spheres (left) and its radial profile (right). The FWHM of the peak is 5.366±0.012 μm.

The ultimate limit of the angular resolution of the setup was tested by recording the direct beam and the diffracted beams on high-resolution x-ray sensitive films. Fig. 2 shows an image of one of the (110) Bragg reflection of a randomly-stacked hexagonal close-packed (rhcp) crystal of hard silica spheres. The full-width at half-maximum (FWHM) of the radial profile through the reflection amounts to less than 6 μm, i.e. the optical setup allows achieving an angular resolution better than 2 μrad.

The vast majority of the diffraction patterns were recorded using a CCD camera (Sensicam) with a pixel size of 6.45 μm. Fig. 3 presents a scattering pattern measured in a cylindrical capillary containing a fluid of PMMA colloidal hard spheres (270 nm diameter). The broad rings in the scattering field originate from the fluid. In addition, x-rays revealed a colloidal crystal had grown at the capillary wall. Later, a careful visual inspection of the sample did allow us to identify a few small crystals displaying Bragg reflections of green light. Analysis of the peak width δq showed that δq = 0.00038 nm^{-1} (i.e., about 1% of the q-value of the lowest-order (100) Bragg rod reflection). In angular terms this corresponds to about 7 μrad and is fully determined by the detector resolution. Although this resolution is less than could be provided by the optics, it is significantly higher than the resolution we were able to reach in our previous experiments without refractive optics.

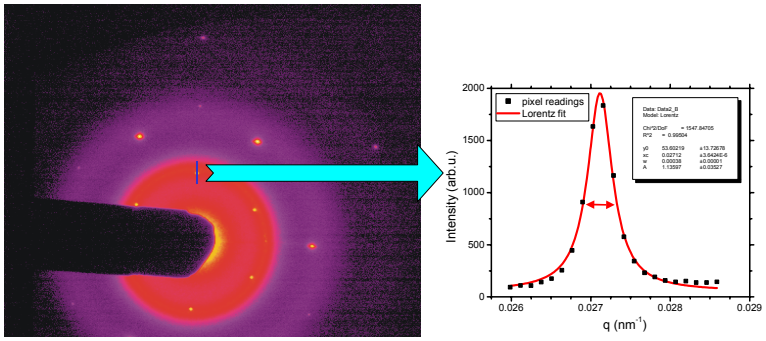

Figure 3. Scattering pattern obtained from a fluid of PMMA colloidal hard spheres and a coexisting crystal grown at the capillary wall (left). The graph on the right displays the profile of a reflection originating from the (100) Bragg rod [2].

Fig. 4 displays examples of the diffraction patterns measured from rhcp crystals of hard silica spheres. For comparison, we also show a similar pattern recorded in the same system earlier (end 2001) with a gas-filled detector at the beamline BM-26 DUBBLE. We present diffraction patterns measured from a well-ordered crystal and a strained crystal yielding azimuthally-broadened reflections. It is clear that the distinction between the two crystals would not have been possible with the resolution we had before. Furthermore, during the experiment an extensive study of order parameters of photonic crystals [4] was performed

It is our pleasure to acknowledge Federico Zontone, Henri Gleyzolle, Patrick Feder, Andrei Fluerasu and Anders Madsen for the excellent support, Marc Diot and Cyril Ponchut for the detector.

References

[1] A.V. Petukhov *et al.*, Phys. Rev. Lett., **88**, 208301 (2002).
[2] A.V. Petukhov *et al.*, Phys. Rev. Lett., **90**, 028304 (2003).
[3] M. Drakopoulos *et al.*, Appl. Phys. Lett., **86**, 014102 (2005).
[4] A.V. Petukhov *et al.*, ESRF Newsletter, **38**, 19 (2003).

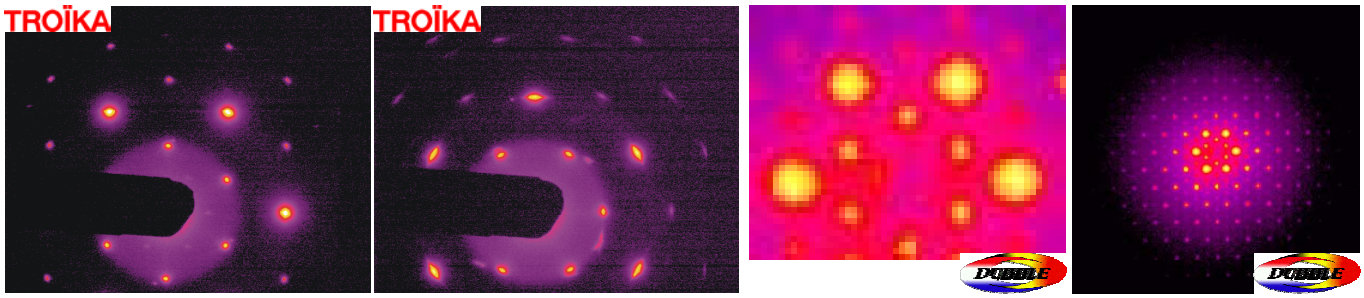

Figure 4. Patterns measured during the experiment SC-1418 in a well ordered (a) and a strained (b) rhcp crystal of silica hard spheres. For comparison, we show a pattern (from Ref. [1]) measured earlier at BM-26 DUBBLE: the full view (d) and a zoom (c) into a region similar to the one in panel (a).