DUBBLE	**Experiment title:** **Single Scattering Characterization of Inverse Photonic Crystals with a Complete Band Gap**	**Experiment number:** **26-02-181**
Beamline: BM-26B 'DUBBLE'	**Date(s) of experiment:** From: 26-09-2003 To: 29-09-2003	**Date of report:** 3- 11 -2003
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Report: (max. 2 pages)

Photonic crystals are materials in which the refractive index varies periodically on length scales comparable to optical wavelengths [1]. Like for x-rays in atomic crystals, this large-scale periodicity leads to Bragg diffraction in the visible and produces a colourful display similar to that of nacre. Unlike for x-rays, however, the contrast can easily be made quite large leading to a strong wave-lattice coupling. There exist lattices in which light of a certain wavelength range (the photonic band gap) cannot propagate in any direction. Such "semiconductors" of light would be highly useful in optical devices, because they could control light in unprecedented ways: by inhibiting spontaneous emission in the band gap and by forcing light to move in tight places such as in optical integrated circuits. An important area of potential applications is infrared telecommunication, operating at wavelengths of 1.3 and 1.5 μm, where silica fibres display high transport performance.

3D photonic crystals are difficult to fabricate. The method we used is self-organisation of colloidal particles. Spherical particles can be made of the appropriate sizes and can self-assemble into crystals with long-range order. By tuning the interparticle interaction potential, various lattices can be made, such as face centred cubic (fcc), randomly-stacked hexagonal close-packed (rhcp), body-centred cubic (bcc) and body-centred tetragonal (bct) [2]. By filling dried colloidal crystals ("opals") with silicon and etching out the silica, "inverted crystals" of silicon can be made [3], which possess a band gap.

The question we tried to answer in this experiment is: Does the photonic crystal still have the intended structure as well as a high degree of order? Although typical sizes would normally be large enough to see under an ordinary microscope, this has become impossible since the crystal is filled with strongly scattering and (in the visible) absorbing material. X-rays, which are weakly scattered and possess sufficient penetration depth, are one of a few tools, if not the only one, available to elucidate the internal 3D structure and order of such photonic crystals.

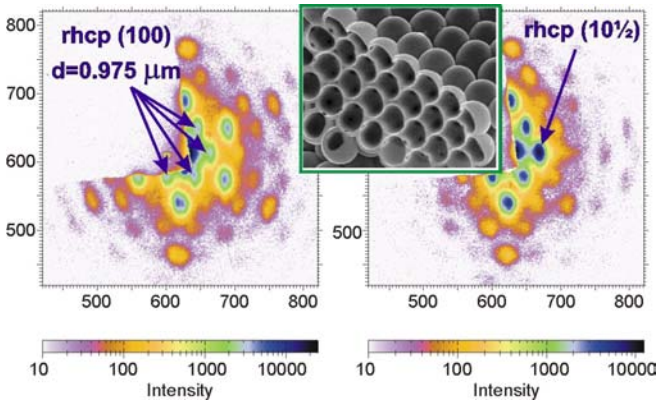

Figure 1: μrad-XRD patterns of a close-packed colloidal crystal filled with silicon obtained with two sample orientations. The inset displays an SEM micrograph of the same sample after removing the silica. Here and in Fig. 2 the x-ray energy was 11 keV (λ = 1.13 Å) and the sample-detector distance was 8 m.

Microradian 3D crystallography

We have applied small-angle x-ray crystallography to photonic crystals. An example of data measured in a silica crystal filled with silicon is shown in **Figure 1**. Well-resolved diffraction peaks can be clearly seen. The crystal had a close-packed lattice while the exact stacking sequence was not known beforehand. By measuring diffraction at different sample orientations [4,5], the 3D rhcp crystal structure was established.

Figure 2 presents diffraction patterns of a wet colloidal crystal with bct structure. The rectangular arrangement of bright reflections in panel c (highlighted by white dotted lines) reflects the bct-stacking of close-packed layers (cf. panel b). This wet and index-matched crystal has also been characterised in real space. However, to make it more strongly photonic, further treatment was necessary. Panel (d) presents a diffraction pattern obtained from a similar bct crystal, which was dried and then filled with silicon. One can clearly see several changes in the diffraction pattern. The rectangular arrangement of the strongest reflections is still recognisable, but other features come up as well. In particular, the arrows in (d) point at greatly enhanced reflections typical for close-packed (fcc, rhcp) structures [4,5]. Thus, although most of the crystal still preserves the bridge-site stacking of the hexagonal planes of the bct structure, the hollow-site stacking of close-packed structures becomes significant as well. Furthermore, one can see a very strong increase of the scattering background in between the Bragg peaks, which indicates creation of defects in the crystal. The Bragg peaks display about 6% shift of their positions indicating a reduction of the average period of the structure. Moreover, one can see broadening of the diffraction peaks, which suggests that the intrinsic 2σ width of the crystal reflection increases to 0.6 μm^{-1} (or, 11 μrad in angular terms).

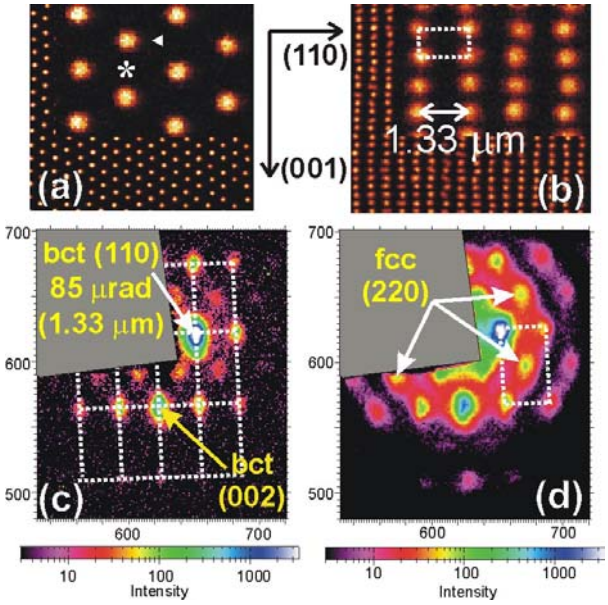

Figure 2: Body-centred tetragonal colloidal crystal: (a) A confocal laser microscope xy-image of a single hexagonal plane. White star and triangle denote a bridge and a hollow site, respectively. (b) A z-average of lateral particle positions in four crystal planes. μrad-XRD patterns of a crystal before (c) and after (d) drying and silicon infiltration.

These results demonstrate that x-ray diffraction at microradian angles (μrad-XRD) is readily achievable at DUBBLE even without a Bonse-Hart camera, which is typically used in an ultra-small-angle x-ray scattering (USAXS) setup. μrad-XRD is shown to yield a wealth of information about photonic structures with spacing above 1 μm. In addition to the 3D crystal structure, one can record degradation of the order in successive steps of the fabrication process. The latter, however, is at the edge of the resolution power of BM26B. As was shown in the report of the experiment 26-02-166, the main limitation arises from beam focusing by an element, which is far upstream from the sample, leading to a degradation of the coherence properties of the beam in the sample. Recent experiments [6] performed at BM5 have shown that even higher resolution can be achieved by using another focusing scheme with a compound refractive lens [7] with a sufficiently short focal length. The latter allows one to focus the beam into much smaller size at the detector position within the experimental hutch. This permitted resolving diffraction at an angle of a few microradian from structures with period larger than 10 microns. We are currently planning application of this technique at ID10 to our 3D photonic crystals, which is believed to yield even more details on the crystal (dis)order. Later one could consider possibilities of building a similar setup at DUBBLE.

References

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