DUTCH-BELGIAN BEAMLINE
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Experiment Report Form

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- fill in a separate form for each project or series of measurements.
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- 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.

(next page)

DUBBLE	**Experiment title:** **Single Scattering Characterization of Inverse Photonic Crystals with a Complete Band Gap**	**Experiment number**: **26-02-253**
Beamline: BM-26B 'DUBBLE'	**Date(s) of experiment**: From: 04-11-2004 To: 08-11-2004	**Date of report**: 8- 12 -2004
Shifts: 12	**Local contact(s)**: dr. Florian Meneau	

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Report: (max. 2 pages)

Photonic crystals (PCs) are 3D structures in which the refractive index varies periodically throughout space [1,2]. In a suitable 3D lattice, with an index contrast above the threshold (~3), a photonic band gap can be opened up. This means that light of a certain frequency range cannot propagate through the PC in any direction for any polarization, allowing one to manipulate photons similar to semiconductors manipulating electrons. Applications include telecommunications at infrared wavelengths (1.3 or 1.5 μm).

Fabrication of PCs with a band gap is challenging. A promising route towards PCs is through self-assembly of colloidal particles [3,4]. As demonstrated in Figure 1, the colloidal crystals can be used as templates for silicon chemical vapor deposition (CVD) [3]. These (silica) templates can be characterized by confocal microscopy after refractive-index-matching. However, after infiltration, index matching is no longer possible. As x-rays interact relatively weakly with matter, Small-Angle X-ray Scattering (SAXS) forms an excellent tool to probe the internal structure of these inverse PCs. The main objective of the recent measurements was to study the internal 3D structure and order of inverse photonic crystals with lattice spacings on the order of 1 μm. Various colloidal crystals have been characterized, both before and after silicon infiltration.

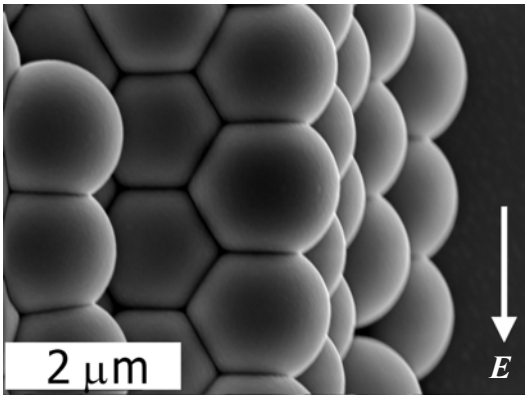

Figure 1: Scanning Electron Microscopy (SEM) image of the first 5 layers of a 7-layer, silicon-infiltrated, colloidal crystal. The image clearly shows the bottom two layers being close-packed, which is due to the infiltration process; the remaining crystal layers are still bct-stacked.

In our previous experiment at DUBBLE, in September 2003, we have performed similar measurements on colloidal crystals [5] with an angular resolution of approximately 20 μrad. This resolution was sufficient to resolve x-ray diffraction from our sample. However, the determination of the intrinsic width of the Bragg peak was impossible in most cases. Further resolution improvement can be attained with a new setup exploiting a compound refractive lens (CRL), as demonstrated in our recent ESRF experiment at the TROIKAIII station (ID10A) in May 2004. We achieved a resolution of ~7 μrad in digital data, which was mostly limited by the resolution of the CCD-camera. As determined by x-ray photographic films, the optical setup allowed for a resolution of 2 μrad.

In the present experiment at DUBBLE we have built a similar setup using a CRL. The main advantage of the DUBBLE beamline is the length of the experimental hutch, which allows for sample-detector distances as large as 8 m (instead of 3 m at TROIKAIII). This allowed us to relax the requirements on the detector resolution. No beam focusing was used *before* the sample, which resulted in a much larger transverse coherence length of the beam [5]. Instead, using the CRL, much tighter focusing was used *after* the sample. To calibrate the setup, we used a silicon grid with a lattice spacing as large as 4.2 μm (Fig. 2A). Diffraction peaks separated by as little as $\Delta(2\theta)$=23 μrad are clearly resolved. Gaussian fits

through the data reveal that the 2σ-width of the peaks in the horizontal direction is about 4 μrad (or, full-width-at-half maximum is smaller than 5 μrad, see Fig. 2B). It is important that this result is achieved at DUBBLE, which uses a bending magnet source instead of a (much brighter) undulator source, as at ID10A. Yet, the resolution at DUBBLE in the vertical direction is worse, presumably due to mechanical instabilities in the optics hutch.

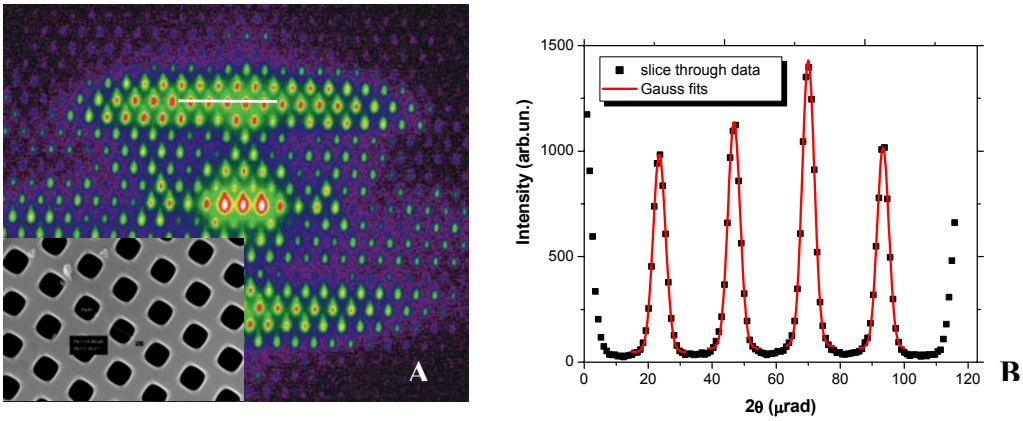

Figure 2: Normal-incidence, x-ray diffraction image of a silicon grid with a lattice spacing of 4.2 μm (A). The diffraction peaks coming from the grid can easily be resolved. The inset shows an electron microscope image of the silicon grid. In order to obtain the resolution of the setup, a line profile (B) through 5 peaks was taken along the white line in image A. A Gaussian fit yielded a resolution of approximately 4 μrad in the horizontal direction.

The extremely high resolution of the setup was then used to accurately probe the internal 3D structure of inverse photonic crystals with lattice spacings of about 1 μm. Furthermore, a smaller beam stop allowed us to record a larger portion of the diffraction pattern at very small angles (see Fig. 3A). In our previous experiments, we observed that silicon-infiltrated, bct crystals showed fcc-like diffraction peaks and increased background scattering (Fig. 3B). Apparently, the infiltration process leads to an increase in the number of defects and to close-packing of the bottom layers due to surface adhesion (Fig. 1). We have since adapted the crystal growth procedure such that the crystal is better able to withstand the silicon-infiltration process. Indeed, as can be seen in Fig. 3A, the fcc-like peaks (green encircled) are now much weaker relative to the bct peaks (red encircled). This indicates that the bct structure is now much better preserved during silicon CVD. Moreover, the diffraction peaks are much better resolved.

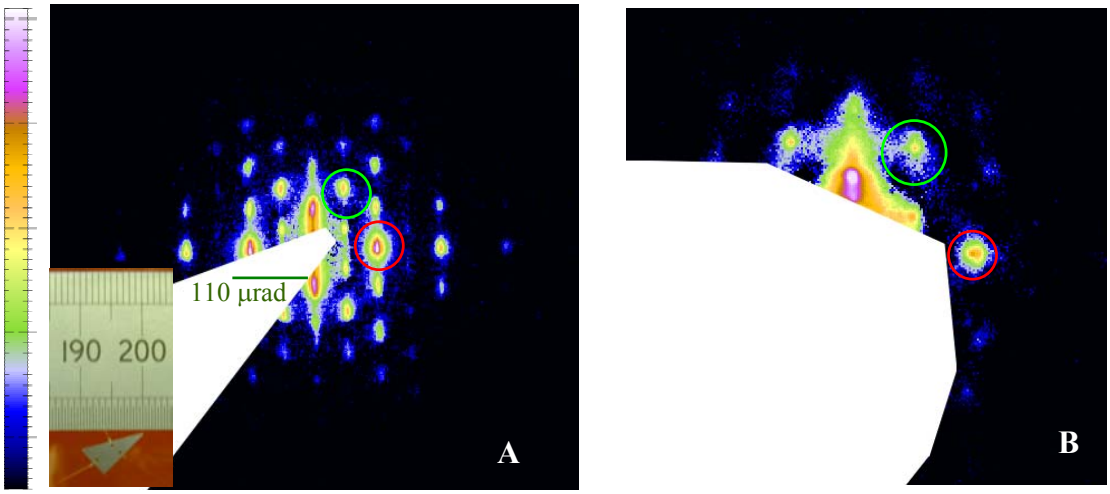

Figure 3: Normal-incidence, x-ray diffraction pattern of a 25-layer, silicon-infiltrated, colloidal bct crystal (A) of 1.1 μm diameter silica spheres taken at DUBBLE in November 2004. The inset shows a photo of the new beamstop (white polygon), which was designed for this experiment. For comparison, image B shows an older diffraction pattern of a similar crystal consisting of 1.4 μm diameter spheres and having only 7 layers, taken at TROIKA in May 2004.

References

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[3] Vlasov *et al.*, Nature (London), **414**, p.289 (2001)
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