DUBBLE	Experiment title: **Synchrotron microradian diffraction study of inverse metallic photonic crystal**	**Experiment number**: **01-02-801**
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Report: (max. 2 pages)

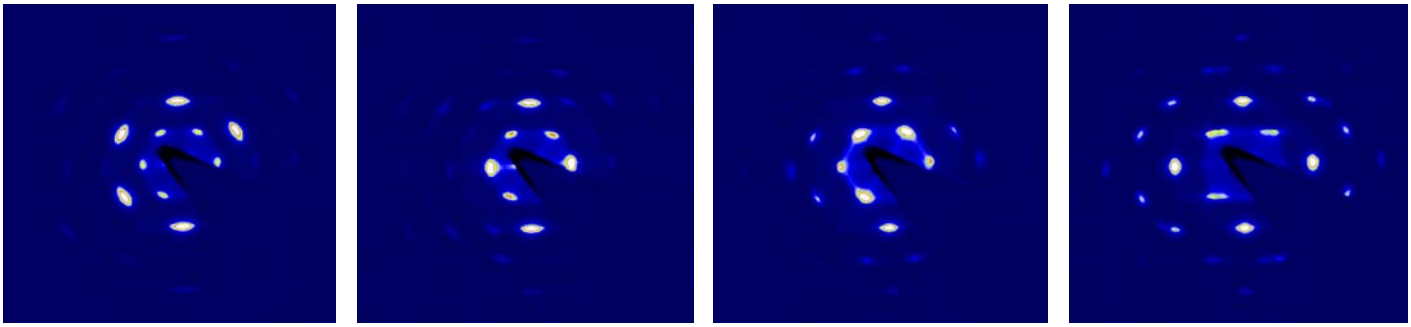
Artificial opals consisting of submicron monodisperse microspheres packed in a face-centred cubic (fcc) cell and materials on their basis are good candidates for the creation of high quality Photonic Crystals (PC). The latter have recently attracted great attention due to their unusual optical properties and promising applications in optical devices. Inverse photonic crystals (IPC) can be synthesized by filling the voids of opal templates and by subsequent removing the initial microspheres to leave three-dimensionally ordered porous materials. It turned out that infiltration of the new material into the structure is one of the crucial steps of the fabrication an IPC, which can lead to significant degradation of the structure (as it is the case of, e.g., chemical vapour deposition [1]). One of the promising alternative methods is the 'wet' electrodeposition enabling for a more delicate infiltration of the voids by a metal with almost 100% degree of filling. The conducting versions of the IPC are of great interest from the point of view of their multi-functionality and interplay between the optical, magnetic, and transport properties.

The synthesis of the metallic PC is based on the deposition of the monodisperse microspheres on a conductive surface followed by the electrodeposition of a metal into the voids of the obtained PC. The important parameters of this technology are the substrate material, the wetting properties, the roughness and the potential of the surface. The aim of the present study was to investigate the structure of the PCs deposited on different conductive substrates and to study the structure of the metallic IPCs obtained on their basis. It is well established that PCs synthesized by the vertical deposition method are normally ordered in an fcc structure. As shown in our recent experiment [2], some stacking disorder is, however, also present. Disorder phenomena can strongly affect the physical properties of IPCs; it is difficult, however, to characterize and manipulate the degree of disorder. A generic goal of this study is to optimize crystal structure and physical properties of the inverse PCs by tuning a set of technologically accessible control parameters.

The microradian diffraction using synchrotron radiation is a very efficient, if not unique, way to determine the structure and to characterize a degree of order of the photonic crystals. It is important to note that in the small angle diffraction geometry the image on the detector is a cross-section of the Fourier image of the crystal structure by the plane perpendicular to the X-ray beam. Thus, by rotating the crystal and recording the patterns with different sample orientations, one can reconstruct the full 3D reciprocal lattice, like in single crystal X-ray diffraction experiment but on a mesoscopic scale.

Our microradian synchrotron diffraction experiments were performed at the DUBBLE beamline (BM26B) on (i) the PCs deposited on mica plates covered by a thin gold layer and (ii) the Ni IPCs. We have found that the two types of the samples yield very similar diffraction patterns implying that electrodeposition of Ni does not destroy the original PC matrix. The results of the experiment with the Ni IPC are shown in Fig.1. The diffraction patterns were recorded at various rotation angles of ω = 0, 19, 35, 55 degrees around the vertical

axis, which coincides with the (2,-2,0) crystallographic direction. Zero angle corresponds to the geometry of the IPC surface perpendicular to the beam. All the reflections can be easily identified as originating from an fcc crystal or as induced by the final width of the crystal (the SEM shows that the sample consists of 8 layers only). Examples of the latter are the peaks of the inner hexagonal rings at ω = 0 (Fig. 1a). Despite of much higher form factor at this value of q, the inner-ring reflections are, however, much weaker than the <2-20> fcc reflections of the second ring. Moreover, stacking disorder-induced Bragg rods are visible in Fig. 1c (see [2] for further detail).



(a) ω = 0 deg (b) ω = 19 deg (c) ω = 35deg (d) ω = 55 deg

Fig. 1. Small angle synchrotron diffraction patterns from the Ni inverse photonic crystal measured at ω = 0 (a) and ω = 19 deg. (b), ω = 35 deg. (c) and ω = 55 deg. (d). These patterns correspond to the cross section of the reciprocal lattice of a thin fcc crystal by (111), (112), (110) and (100) planes, respectively.

Our observations confirm that the electrochemical method of synthesis indeed allows to duplicate structure of artificial opals and to obtain the inverse photonic crystal with fcc structure. One could also note that the visibility of the Bragg peaks up to high diffraction orders demonstrate the high degree of ordering.

In addition, we have performed preliminary measurements of the PCs made of negatively-charged polystyrene spheres deposited on the conductive substrates with various potentials (+0.5 V, 0 V and -0.5 V). It appears that the crystallographic ordering depends on the potential and the crystals obtained on substrates with negative or neutral (zero) potential are much better ordered than that with the positive potential. Thus, the substrate potential seems to be a good candidate for a control parameter tweaking structure and properties of PCs. Unfortunately, due to the time limitation we did not measure the samples synthesized at the potential varied in a wider range of the substrate potentials. We hope to perform such a study in future.

We would also like to note that such an experiment could be done more efficiently having the following improvements done. Firstly, a goniostat with a sphere of confusion better that 100 microns together with a microscope would make sample alignment much easier and precise. A motorized gonio-head (like one of those produced nowadays by HUBER) together with existing φ-axis and ω-arc could be the simplest solution. Secondly, data acquisition could be much faster if it is run in a form of a script synchronizing sample rotation and data recording. This requires the incorporation of the detector control into the SPEC program operated by a LINUX computer. Thirdly, a development of software to reconstruct 3D diffraction image from experimental detector frames is very desirable. We have tested the DUBBLE data with a prototype algorithm realized as a MathCad script (A. Eliseev); the results look very promising.

Finally, we would like to thank DUBBLE and SNBL for the time allocation, and also Dr Kristina Kvashnina and Dirk Detollenaere for their excellent support.

[1] J.H.J. Thijssen et al., Advanced Materials, **18**, 1662 (2006).
[2] Report of the experiment 26-02-392.