ESRF	**Experiment title:** Looking inside three-dimensional (3D) silicon photonic band gap crystals	**Experiment number:** HC-2520
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Shifts: 9	**Local contact(s):** Peter Cloetens (email: cloetens@esrf.fr)	*Received at ESRF:*

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Report:

In nanofabrication, it is a main challenge to characterize the structure of a real sample. This is especially true for nanophotonic metamaterials, whose properties and functionality are determined by their complex structure with features comparable, or even smaller than the wavelength of light. X-ray techniques are ideal to structural characterize opaque metamaterials, as they are non-destructive and provide *nm*-resolution. We first study 3D Si photonic band gap crystals with a diamond-like structure by 3D tomography. The density distribution $\rho_e(X, Y, Z)$ of the structure is defined by two perpendicular 2D arrays of pores (Fig. 1(a)). Both 2D arrays have a centered rectangular structure with lattice parameters (a, c) with $a/c = \sqrt{2}$ (diamond (110)).

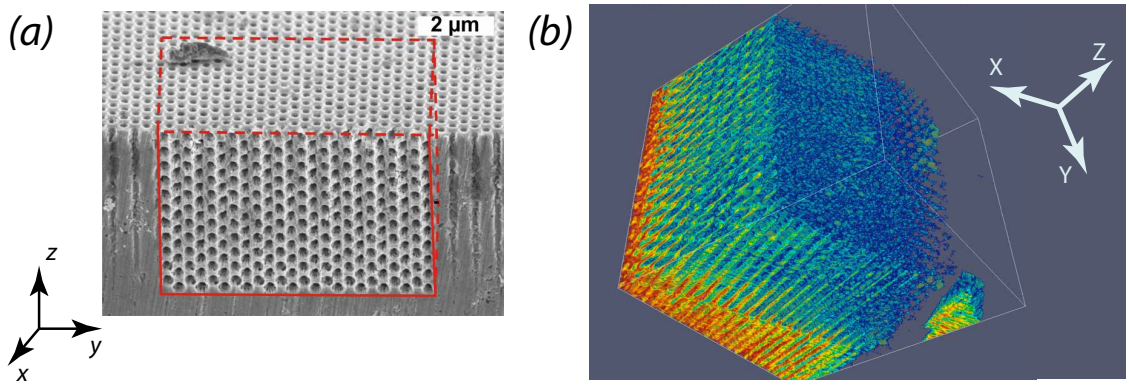

Figure 1: (a) SEM image of the surface of a 3D Si inverse woodpile photonic crystal. The *estimated* extent is shown by the dashed lines. The 3D crystal is surrounded by a large 2D array of pores that are first etched in the Z-direction (see: Leistikow *et al.*, PRL **107**, 193903 (2011)). (b) Bird's-eye view of the reconstructed sample volume. The color scale is the electron density $\rho_e(X, Y, Z)$ interpolated between Si and air, the latter shown transparent (see: Grishina, Cloetens, Harteveld & Vos (2016)).

The alignment of the pores is a crucial step in the nanofabrication that can only be assessed *in situ* by X-ray tomography. Moreover, it is crucial to verify the cylindrical pore geometry, and that pore depth that ultimately limits the crystal size.
Figure 1(b) shows the reconstructed volume of one crystal. Closer inspection indeed reveals two sets of pores running in the Z and the X-directions, matching the design. It appears that the angle between the pore arrays systematically deviates by a few degrees from the 90^o design. This means that the crystal structure is not truly cubic but monoclinic. Our band structure calculations (JAP **105**, 093108 (2009)) reveal that the 3D photonic band gap remains robust: the gap hardly narrows from 24% relative bandwidth to more than 21%, plenty for strong spontaneous emission control.

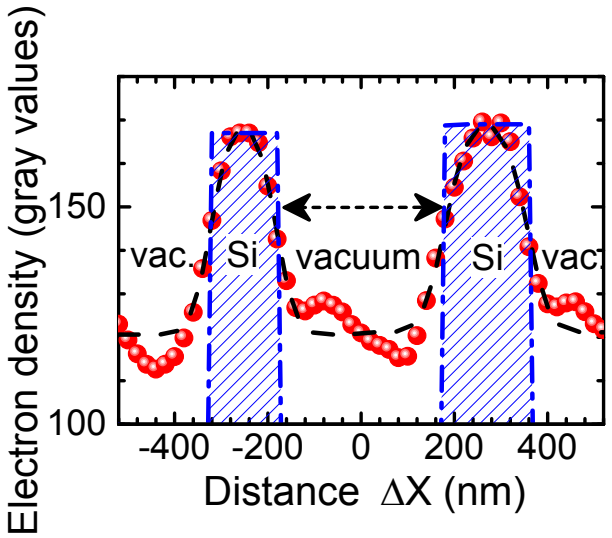

Figure 2: Density profile in the X-direction through pores in Si (red circles). The black dashed curves is the convolution of a square density function (blue hatched bars) and a Gaussian resolution function.

Figure 2 shows a cross-section through pores in the reconstructed volume. To interpret the electron density, we propose a simple model of a binary normalized density function for silicon ($\rho_e = 1$) and vacuum ($\rho_e = 0$) convoluted with a Gaussian resolution function. The adjustable model parameters are the width w of a Si-wall, the Gaussian resolution σ, the amplitude, and a background. The data are well modeled with two Si-walls with widths $w_1 = 140$ nm and $w_2 = 176$ nm. Given the 20 nm voxel size and the resolution, this agrees with the design $w = 156$ nm, hence the structure was faithfully realized. The resolution widths ($\sigma_1 = 32$ nm and $\sigma_2 = 37$ nm) agree well with the measured X-ray beam size. Modeling of the structure offers a straightforward future path to input tomographic structural data into $ab-initio$ numerical codes (FEM, FDTD) to compute nanophotonic properties. This opens the prospect to do*model-free* calculations of metamaterial to predict experiments and device functionality.
At the end of the run we briefly did successful tests of a direct-laser written polymer structure, and of X-ray fluorescence detection of semiconductor quantum dots inside a photonic crystal. We therefore conclude that 3D X-ray tomography has great potential to solve many questions on optical metamaterials for much nanophotonic research and applications, including cavity arrays, physically unclonable functions, and precise localization of light emitters for enhanced efficiency.