# X-ray Imaging Non-Destructively Identifies Functional 3-Dimensional Nanostructures

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Three-dimensional (3D) nanostructures are drawing a fast-growing attention for advanced functionalities in Nanophotonics, photovoltaics, and novel 3D integrated circuits and novel flash memories. The functional properties of such nanostructures are fundamentally determined by their complex internal structure that consist of 3D arrangements of structural motifs such as spheres, rods, or pores. As exemplary nanostructures, we study 3D silicon photonic band gap crystals. These crystals are powerful tools to control the propagation and the emission of light on account of their broad complete 3D photonic band gap [1]. Inevitably, any fabricated nanostructure differs from its initial design. Hence, the observed functionality differs from the expected one. It is therefore critical to non-destructively assess the structure of a 3D nanomaterial and verify how well its function matches the design.

In nanotechnology, it is an outstanding challenge to characterize the structure of a real sample, in particular of a 3D one. The challenge notably pertains to nanophotonic materials, whose properties are determined by their complex structure with feature sizes $d$ comparable to or even less than the wavelength $\lambda$ of light ($d \leq \lambda$). These nanomaterials are from the outset highly opaque, thus optical microscopy has insufficient penetration depth, apart from a limited resolution. While scanning electron microscopy (SEM) offers fantastic nanometer spatial resolution, it has a small penetration depth hence only the sample surface is viewed (*cf.* Figure 1), but not the bulk. Although X-ray techniques are very promising tools for Nanophotonics, in view of the excellent penetration depth, and the non-destructive character [2, 3], they are hardly exploited in this field. To achieve nanometer spatial resolution in a nanostructure with thick (millimeter) substrates that do not have to be cut away (as with, *e.g.*, ptychography), we decided to employ X-ray holographic tomography.

The photonic crystals have a diamond-like structure, known as the "*inverse woodpile*" structure [Ho1994]. The material distribution in the crystals is defined by two perpendicular 2D rectangular arrays of pores, with pores running in the Z and X-directions (Figure 1). The crystals are made by CMOS-compatible means using deep reactive ion-etching through tailored etch masks [4]. We performed X-ray holographic tomography studies at the ESRF on beamline ID-16NI: X-rays with a photon energy of 17 keV are focused before the sample. We collect data at four sample-to-detector-distances to cover all spatial frequencies, as we operate in the Fresnel regime [5]. At each distance, 1500 images are recorded while rotating the crystal from 0 to 180°. Phase maps are obtained at every angle, followed by standard tomographic reconstruction based on the inverse Radon transform to obtain the 3D electron density distribution with 20 nm resolution.

Figure 1 shows the reconstructed volume of one crystal. The volume is a cube that contains the 3D photonic crystal structure that is surrounded by bulk silicon below and by the 2D array of deep pores that are etched first. A closer inspection reveals two sets of pores running in the Z and the X-directions, matching the design. The pores etched in the X-direction appear to be much deeper than in the Z-direction. It thus appears that one can realize much greater 3D nanostructures than expected beforehand. Our results also reveal that the structure is periodic over many unit cells with average lattice parameters that match the designed ones very well. New information offered by tomography are the statistical variations of feature sizes, which has consequences for optical functionality. For instance, we observe a variation in pore depths that corresponds to a corrugated crystal-silicon buried interface, causing a vanishing of Fabry-Pérot fringes (due to front-to-backface interference) in optical reflectivity spectra. We conclude that X-ray tomography is a powerful tool for structural characterization of any complex 3D functional nanostructure with arbitrary short or long-range order[6].

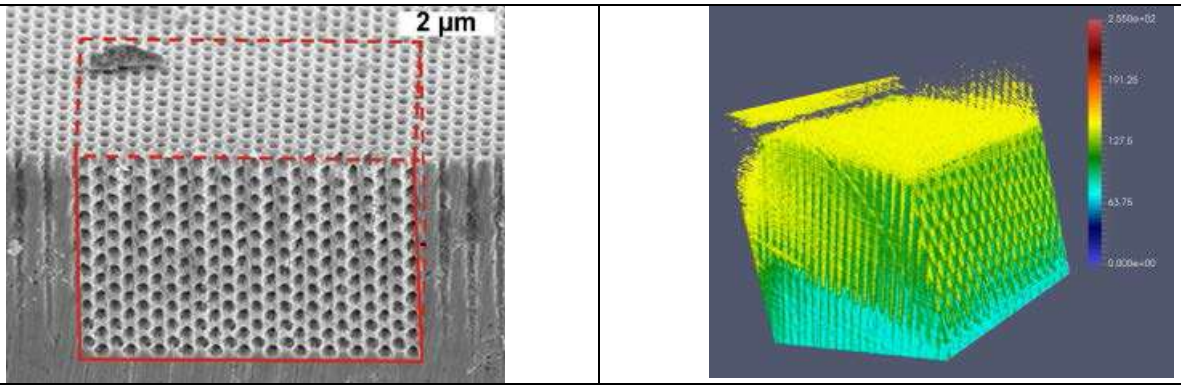


Figure 1. (Left) SEM image of the surface of a 3D Si inverse woodpile photonic crystal. The estimated extent is shown by dashed lines. The 3D crystal is surrounded by a large 2D array of pores that is first etched in the Z-direction. From Ref. [1] (Right) Bird's-eye view of the sample volume of an inverse woodpile crystal reconstructed from X-ray tomography with 20 nm resolution. The color scale is the 3D material density interpolated between air and Si, the latter set to 255.

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