# The good, the bad, the ugly: Looking inside 3-D silicon nanostructures without leaving a trace

December 23 2019, by Diana Grishina

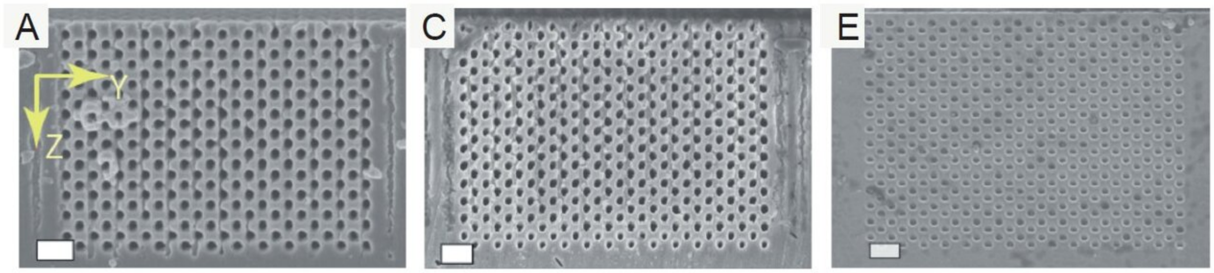



Figure 1. Electron microscope images of three 3D photonic crystal nanostructures fabricated in the same way. Each external surface looks closely matches the design and looks like the other, but the functionality and internal structure will appear to be completely different. Credit: Diana Grishina

Scientists from the University of Twente and the European Synchrotron Radiation Facility in France have discovered a new method to non-destructively look inside three-dimensional (3-D) nanostructures without breaking them. The study is timely since 3-D nanostructures are drawing fast-growing attention for their advanced functionalities in nanophotonics, photovoltaics, 3-D integrated circuits and flash memory devices. The traditional way to look inside 3-D nanostructures is to cut them from their substrate or slice them and view each slice separately. The new method allows researchers to view the complete 3-D structure of the whole nanodevice without cutting or breaking. Hence, this traceless method preserves the device after the 3-D image is made. The breakthrough results will soon appear in *ACS Nano*.

The world around us is truly three-dimensional. As a child exploring surroundings, you learn that you do not want to break things. The same holds for scientists: Once you manage to fabricate a new experimental sample, you do not want to destroy it while investigating whether it behaves as expected. This holds even more so in the world of nanotechnology, where functional properties of nanostructures are essentially defined by the 3-D arrangement of the inner material. Therefore, nanotechnologists carefully design 3-D nanostructures to achieve a specific functionality. And in real life, new samples never match the initial design perfectly, so it is crucial to investigate how the actual sample turns out.

A simple approach to figure out if your fabricated nanostructure matches the design is to study it under the microscope. To illustrate the challenges in such an approach, try this quiz: Consider the three samples shown in Fig. 1 that are fabricated in the same way. Can you tell by the appearance in the electron microscope images whether they are, indeed, the same inside and have the same functionality?

Obviously, a microscope image only gives information about the outside of the sample, whereas knowledge of the internal structure is needed. Traditionally, this challenge is solved by slicing the sample into thin layers and studying them one by one to learn about the internal structure. These widespread, yet destructive methods have an obvious disadvantage: You might find that the sample was made according to specifications, but alas, you have destroyed it. Suitable techniques to look inside 3-D structures include X-ray methods. But even in recent X-ray work, samples are typically cut out from the device or substrate to achieve sufficient transmission and contrast.

In their new paper, the team demonstrates traceless X-ray tomography (TXT). One major step forward is the much greater feasible thickness of the sample of more than a millimeter of silicon due to the much higher X-ray photon energy. Grishina says, "In modern nanotechnology, this is plenty sufficient to image through wafers. Indeed, all silicon device remained untouched and 'as-is' during the study." Moreover, the team's method greatly improved the total sample size to field-of-view ratio, allowing them to zoom in on the desired region.

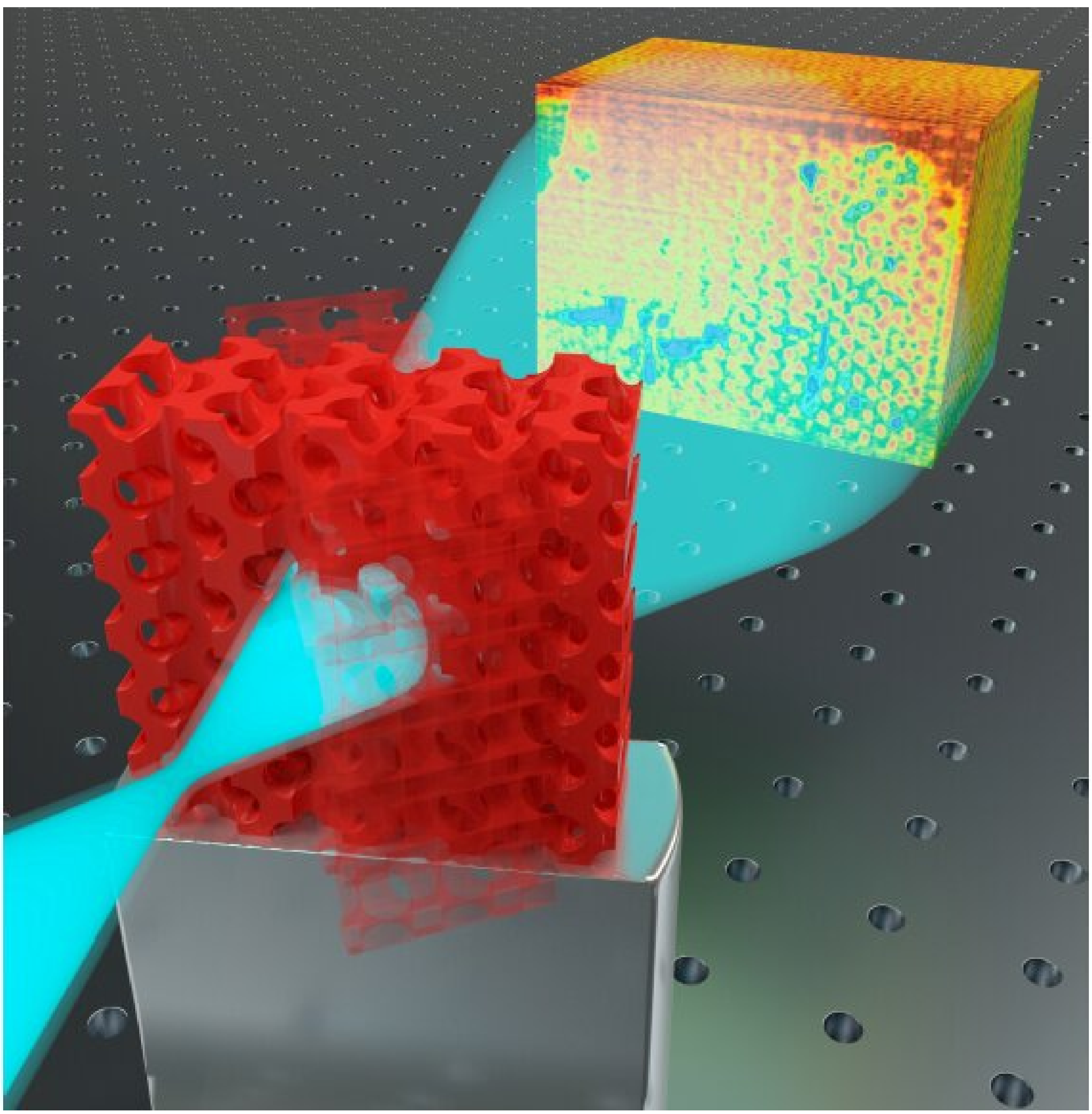
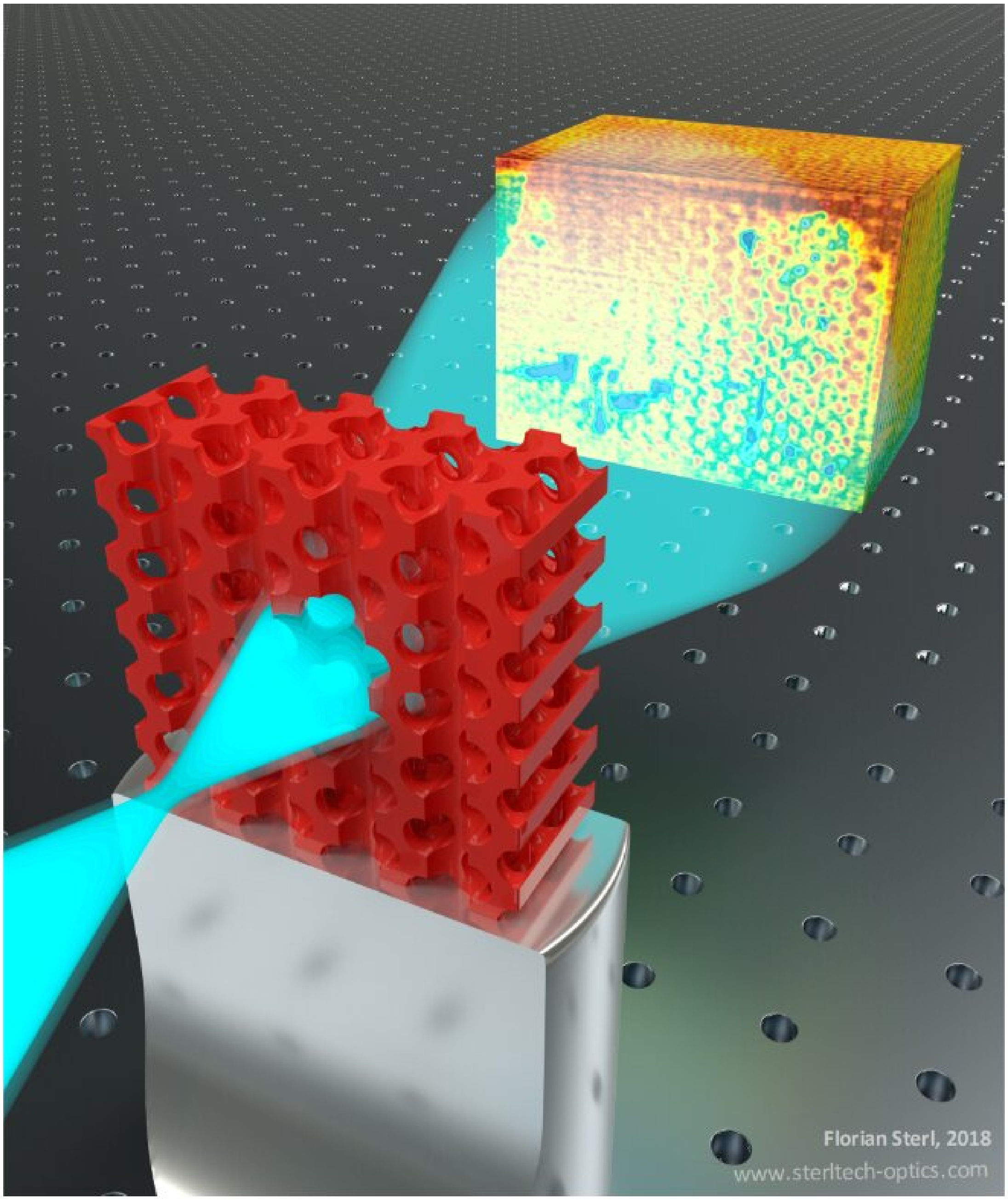


Figure 2. Animation sequence showing that TXT data are recorded while rotating the sample (three orientations are shown). From the recorded radiographs, the tomographic reconstruction is derived that is shown in the background. Credit: Diana Grishina

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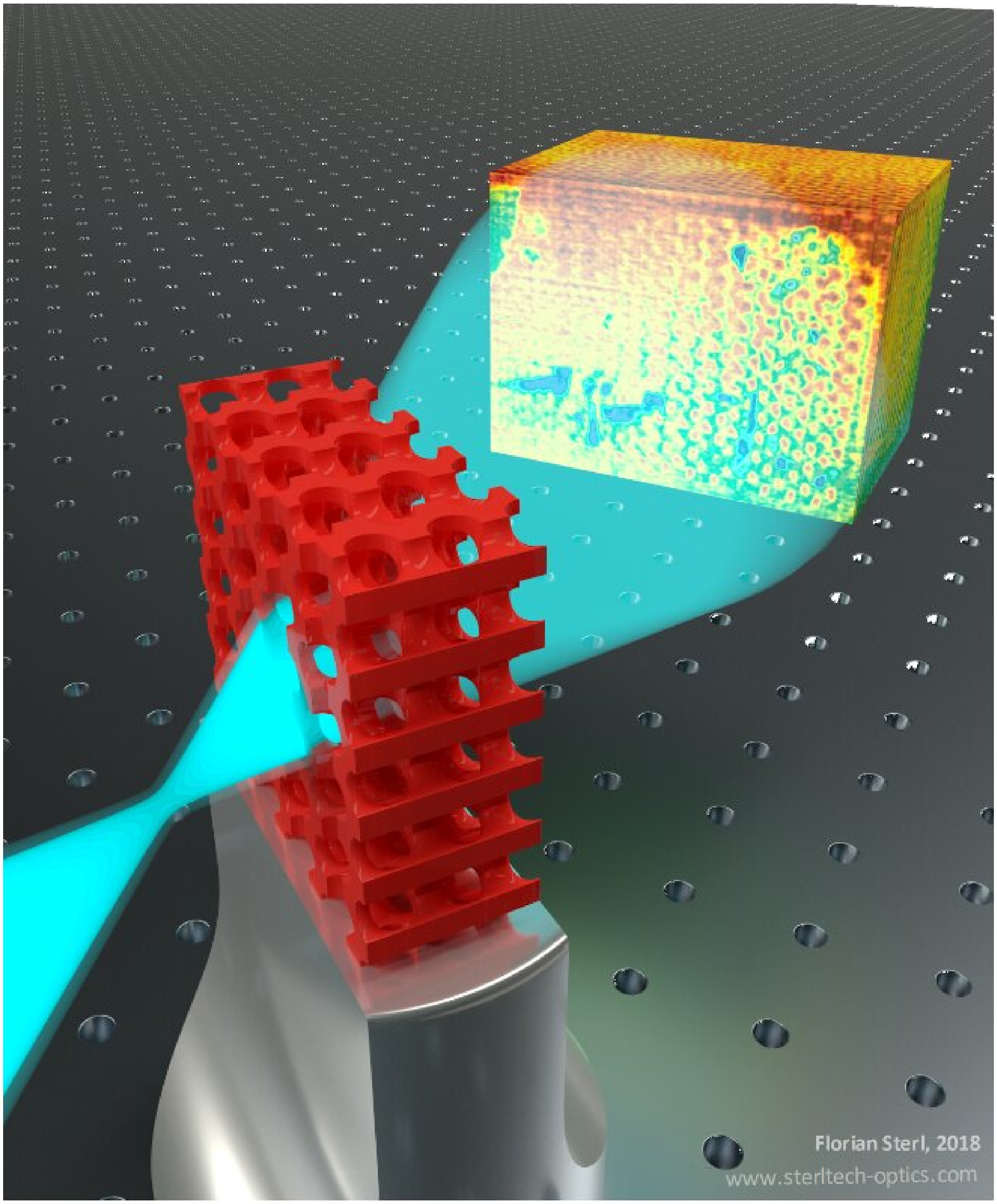


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Holographic tomography experiments were performed at the European Synchrotron Radiation Facility (ESRF). In the process, the hard X-ray beam with 17 keV photon energy propagates in the z direction and is focused with multilayer coated Kirkpatrick-Baez optics to a tiny, 23 x 37-nm focus. Cloetens says, "One key feature of our TXT study is the use of X-rays with a much higher photon energy than before. Therefore, the attenuation length for silicon is 640 μm, which is nine to 20 times greater than before, and sufficient to traverse wafer-thick silicon substrates."

The sample is placed at a small distance downstream from the focus and the detector is placed further downstream. At each distance, 1500 images were recorded while rotating the sample about the Y–axis, as shown in Figure 2. The data processing is an intensive two-step procedure consisting of a phase-retrieval step followed by a tomographic reconstruction.

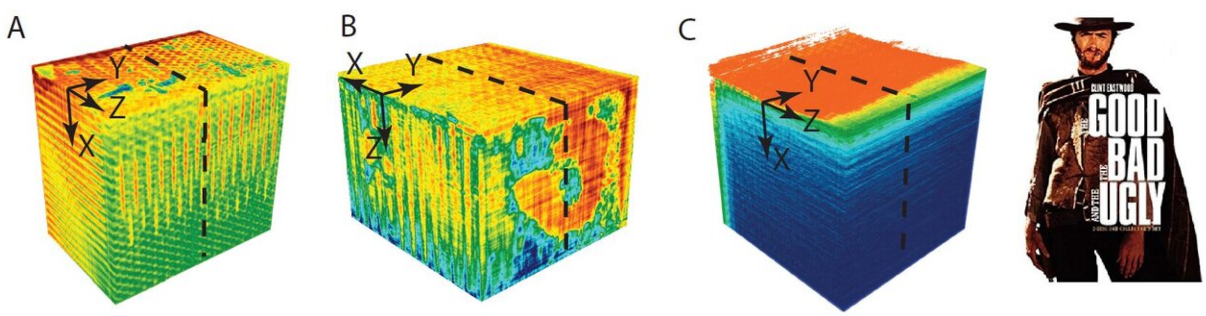


Figure 3. Birds-eye view of reconstructed 3D silicon nanostructures shown in the SEM images in Fig. 1. (A) 3D photonic crystal that reveals a broad photonic gap in agreement with theory: “the Good”. (B) 3D photonic crystal that reveals a large void due to stiction resulting from violent liquid evaporation in the pores:

“the Bad”. (C) Sample that shows shallow pores due to a fabrication error: “the Ugly”. The nicknames are inspired by a famous “spaghetti western”. Credit: Diana Grishina

The team studied exemplary 3-D photonic band gap crystals made from silicon via CMOS-compatible means (a breakthrough a few years ago). They obtained real space 3-D density distributions with 55-nm spatial resolution. TXT identifies why nanostructures that look similar in electron microscopy (see Figure 1) have vastly different nanophotonic functionality: One "good" crystal with a broad photonic gap reveals 3-D periodicity as designed (see Figure 3); a second "bad" structure without gap reveals a buried void; a third "ugly" one without the gap is shallow due to errors during the fabrication. Vos says, "TXT serves to nondestructively differentiate between the possible reasons of not finding the designed and expected performance. This is why we think that TXT is an original and powerful tool to critically assess 3-D functional nanostructures. Judging from responses at conferences, it seems that our colleagues agree. Or is it perhaps because they like spaghetti western movies?"

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**More information:** Diana A. Grishina et al. X-ray Imaging of Functional Three-Dimensional Nanostructures on Massive Substrates, *ACS Nano* (2019). DOI: 10.1021/acsnano.9b05519