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X-ray microscopy of polymer photonic structures

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Over the last two decades, direct laser writing (DLW) has developed into one of the most versatile techniques for the fabrication of nanophotonic structures [1]. DLW employs an ultrafast optical pulse, which is focused tightly in a transparent photoresist. This focus locally induces non-linear absorption that initiates polymerization in the 3D volume of the resist. Continuous development has led to commercial nanofabrication systems with around 100 nm lateral resolution. Inspection of the fabrication results, however, poses a challenge: Readily available diagnostic methods such as scanning electron microscope (SEM) are limited to the exterior of the fabricated structure. A method like Focused Ion Beam (FIB) milling gives access to the interior. However, this method requires physically slicing the structure and hence inhibits subsequent optical measurements on the same structures. Here we argue that X-ray tomography offers a solution. It can be used to quantify the effect of nanofabrication disorder on the optical response of a DLW-written photonic medium. The relation between the nanostructure of a medium and its optical response is of critical importance for security applications that utilize complex media as authentication tokens [2, 3].

In X-ray tomography [4, 5], a focused coherent X-ray beam illuminates the sample. Fresnel diffraction patterns from the structure are recorded at different sample orientations. The diffraction patterns can be analyzed to reconstruct the material (electron) density with spatial resolution of < 50 nm. The advantage of the X-ray tomography method is the non-destructive 3D reconstruction of the nanostructure with deep sub-optical-wavelength resolution. This allows the investigation of the nano-fabricated structure by comparing with the design. We used the commercial DLW platform from Nanoscribe with their photoresist IP-G 780. In our study, we have created cubic disordered polymer rod structures of $(20\ \mu m)^3$ dimensions. Polymer rods stretch randomly from one face of the cube to another. The geometry is optimized to fill the cubic volume with a uniform density [6], while maintaining the rigidity of the structure through optimal interconnections of rods (figure 1). We have taken X-ray microscopy data at a recent run at the European Synchrotron Radiation Facility. Presently we are reconstructing the electron densities from the data.

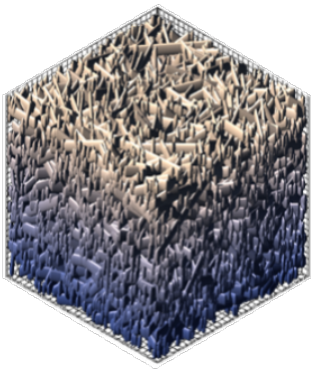

Figure 1: The $(20\ \mu m)^3$ 3D spatial structure of a DLW-written disordered polymer rod sample. The lighter shading indicates the features on top, while the features closer to the substrate are darker.

The reconstructed electron density function using X-ray tomography can be directly compared against the designed density function of the ideal structure. Preliminary qualitative analysis indeed shows fabrication deviations, such as a) missing features that occur during the development of the polymer photoresist, b) collapsed features or other mechanical distortions within the volume, and c) overall shrinkage, a well-known issue in DLW: the polymerized volume will shrink and distort the structure.

We now pursue a quantitative analysis by correlating the design and the reconstructed material density. Several nanofabricated structures of a fixed design will be analyzed. We will compare the optical response of the analyzed structures and quantify the effect of the fabrication disorder on the optical response of the media. By varying the density of rods, the effect of polymer fill fraction on the fabrication process can be analyzed for optimizing feature size and structural stability in the volume of the photonic medium.

Cross-correlation of designed and fabricated density maps will be used to quantify the accuracy of our DLW fabrication process. In future, we aim to predict deviations in the optical response of the nanophotonic structures by using the measured differences in density maps obtained from X-ray data.

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