Sequential Infiltration on Two-Photon Polymerized IP-L 780 3D Microstructures for Photonic Applications

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The process of sequential infiltration synthesis (SIS) is derived from atomic layer deposition (ALD) [1]. SIS utilizes self-limiting alternating half-step reactions at lower reaction temperatures, higher reaction pressures, and longer reaction times to allow precursor gases to infiltrate and react deep within the polymer [2]. Two-photon stereolithographic instruments, such as the Nanoscribe Photonic Professional GT system, which utilize two-photon polymerization (2PP), can create three-dimensional (3D) structures with feature sizes of several hundred nanometers. In this work, we investigate the use of SIS of IP-L 780 resin with zinc oxide (ZnO) to expand the utility of 2PP and present 3D photonic crystals (PhCs) fabricated with 2PP as a further photonic application.

For mechanical testing, 100 µm cubes were fabricated at a 700 nm hatching distance and SIS was performed on developed structures. The ZnO-infiltrated cube was focused ion beam (FIB) half-milled, and energy dispersive spectroscopy (EDS) was performed on the milled structure, showing counts for Zn L-shell, as shown in *Figure 1*, confirming infiltration. To test mechanical changes to the structure, comparisons of elasticity and hardness were made pre- and post-SIS on fabricated structures showing good mechanical stability in *Figure 2*. A comparison of thermogravimetric analysis and differential scanning calorimetry in *Figure 3* shows minimal changes to decomposition and melting points.

3D Photonic Crystals (PhC) can slow the group velocity of light, leading to enhanced light-matter interaction, enabling new applications in spectroscopic sensing systems [3]. Over planar microfabrication techniques, 2PP enables rapid fabrication of PhC, as shown in *Figure 4*. However, the refractive index of ~1.5 for IP-L 780 leads to a very narrow photonic bandgap (PBG). In this work, we use SIS to achieve an enhanced refractive index of 2PP-fabricated PhC, allowing the generation of a wider PBG with applications in spectroscopic sensing.

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[1] Peng, Q. et al (2011) ACS Nana, 5-6, 4600-4606
[2] Ramanathan, M. et al (2013) J. Mater. Chem. 1-11, 2080-2091
[3] Singhal, A. et al (2022) IEEE Sensors J. 22-21, 20126-20137

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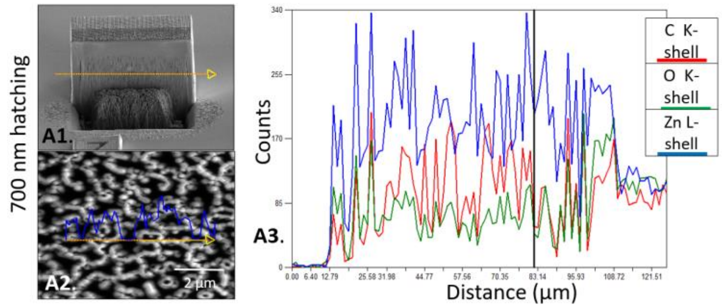

Figure 1: A1: FIB half-milled ZnO-infiltrated IP-L 780 2PP 100 μm cube utilizing 700 nm hatching distance, and e-beam trace shown by the arrow. *A2:* EDS analysis of grass-like structures following FIB milling. The blue line corresponds to the Zn L-shell X-rays originating from the e-beam trace shown by the arrow. *A3:* EDS analysis of milled structure, with violet, blue, green and red liens corresponding to Zn L-shell, Al K-shell, O K-shell, and C K-shell X-rays.

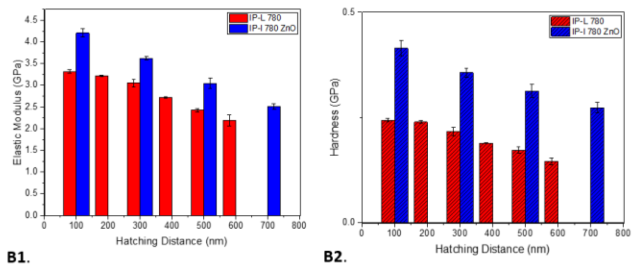

Figure 2: B1: Comparison of the elastic modulus of IP-L 780 and ZnO infiltrated IP-L780. *B2:* Comparison of the hardness of IP-L 780 and ZnO infiltrated IP-L 780.

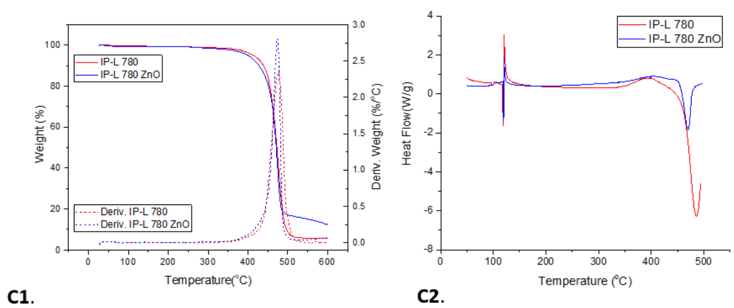

Figure 3: C1: Comparison of thermogravimetric analysis of ZnO infiltrated IP-L 780 and non-infiltrated IP-L 780 structures. *C2*. Differential scanning calorimetric comparison of IP-L 780 and ZnO infiltrated IP-L 780

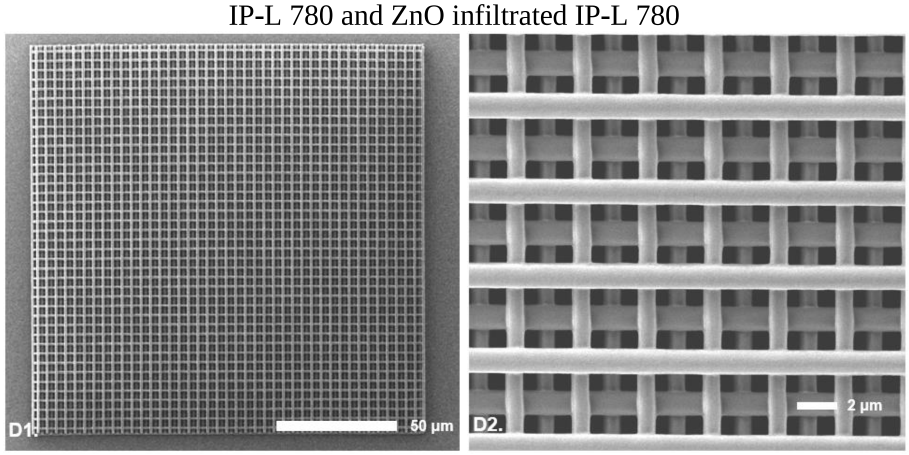

Figure 4: D1: 150 μm X 150 μm PhC fabricated using 2PP from IP-L 780. *D2:* Zoomed in view of PhC showing FCC arrangement confirming good uniformity