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3D Printing Functional Nano-Photonic Devices by Multi-Photon Lithography

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ABSTRACT

Multi-photon lithography (MPL) remains among the handful of practical techniques that can be used to fabricate truly three-dimensional (3D) micro- and nanometer-scale structures with few processing steps. Although photopolymers remain the primary material system for MPL, others have been developed for creating functional structures in chalcogenide glasses and polymer-composites. Post-exposure processes have been developed for transforming a structure created by MPL into another material, such as a metal, semiconductor, or oxide glass. MPL has been used to create a wide range of functional nanophotonic devices. The full potential of MPL is apparent in its use to create spatially-variant lattices (SVLs). SVLs are a new class of nanophotonic device that is engineered to direct and control the flow of light in 3D. The devices are based on unit cells that control the propagation of light through the self-collimation effect. These unit cells are spatially varied in orientation throughout an SVL so that light flows along a prescribed path within the device. The geometry and patterns of the unit cells within the lattice can also be varied to control other key properties, such as phase and polarization. SVLs and their fabrication by MPL opens a new route to 3D integrated photonics, and a myriad of other applications.

Keywords: Multi-photon lithography, 3D patterning, nanophotonics, integrated optics, imaging.

1. THE TECHNIQUE OF MULTI-PHOTON LITHOGRAPHY

Multi-photon lithography (MPL) is a rapidly expanding method for laser-based, three-dimensional (3D) printing of micro- and nano-scale structures. In MPL, a femtosecond-pulsed laser beam is tightly focused into a suitable material to activate a chemical or physical change in the vicinity of the focal spot. The focal spot is then translated within the material to define a photo-pattern. After patterning, the sample can be "developed" or etched to either remove unexposed material, or to remove photo-exposed material. Which occurs depends upon how the material responds to photo-excitation and the nature of the etchant. After developing, a free-standing 3D structure remains on the substrate that is a replica of the photo-pattern. Several authors have reviewed MPL,[1-4] focusing on the technique,[5, 6] material systems,[7-12] means for improving resolution,[13] and its applications for creating functional micro- and nano-scale

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structures.[14] A brief account is given here of the key attributes of MPL.

MPL bears many similarities to other methods for 3D printing, particularly stereolithography ("SL" and "SLA"), with certain key distinctions.[15] In MPL, 3D resolution is achieved through nonlinear excitation, ideally working synergistically with a physical or chemical response that increases nonlinearly with the locally absorbed dose. Conditions can often be found in which a given material system can be promoted to an excited state by multi-photon excitation (MPE). This can be achieved by using a laser source that is sufficiently detuned from conventional absorption bands, so that the energy gap between the ground and excited state is only bridged when the material absorbs two or more photons. When the material does not have a real eigenstate at intermediate energies, the excitation proceeds through virtual states, and the multiple photons must be absorbed nearly simultaneously. Consequently, the cross-sections for multi-photon absorption are typically much smaller than those for linear absorption, but the probability for MPE can be increased significantly by using tightly focused, femtosecond laser pulses.

Many material systems used for MPL have been patterned using the output of titanium:sapphire lasers or certain fiber oscillators, which produce ~ 100 fs pulses at high-repetition rate (> 10 MHz) with $\lambda_0 > 700$ nm. The laser pulses are focused as tightly as possible, often using optics having $NA \sim 1.4$, to achieve the smallest, diffraction-limited focus. Under these conditions, MPE is only efficient near the focal spot, where the local irradiance is highest. As a result, the beam can penetrate deep into the material, without loss from linear absorption, and efficient excitation becomes limited around the focal spot in a region that is tightly confined both laterally and axially. When the material responds to the local irradiance nonlinearly, as in a photo-chemical polymerization, the process of MPL exhibits an irradiance threshold that tightly confines the material transformation to the focal volume. Structures can be patterned readily with lateral resolution as small as 100 nm. The axial extent of the focused irradiance profile is larger than the transverse width, so the patterned volume element, or "voxel," has an axial-to-transverse ratio that is typically 3:1 or larger. This focal spot can nonetheless be translated along adjacent and partly overlapped paths to define structures having other ratios of axial-to-transverse dimension. Individual features with smaller transverse dimensions can be obtained by reducing and carefully controlling the focused average power.[16, 17] Such structures are highly susceptible to shrinkage or other forms of post-exposure distortion. As a result, large-foot-print, free-standing 3D structures comprised entirely of sub-100-nm features have not been created without supporting the structure with a monolithic exterior frame or employing more complex methods to either shape the excitation profile[13, 18-20] and/or develop the structures following photo-patterning.[21]

2. MATERIAL SYSTEMS FOR MULTI-PHOTON LITHOGRAPHY

MPL is most often performed using photopolymers. Photopolymerization is a rich and diverse field unto itself, so here only a brief overview of the principles is given to facilitate discussion of its use for MPL. Detailed treatments of polymerization can be found elsewhere.[22] In its simplest form, a photopolymer is a mixture of one or more components called "monomers," along with a "photoinitiator." Monomers are molecules that can chemically link to one another through bond formation. A photoinitiator is a molecule that upon excitation activates chemical linking of monomers. The structural portions of the monomer that are involved in the linking chemistry are called functional groups. Linking a large number of monomers together forms a polymer. When the monomer molecule has only one functional group, the polymer will be a linear structure. A linear polymer will have very different physical properties from the monomer, such as high viscosity and higher melting point, but the switch in properties is generally not large enough to support fabrication of a free-standing 3D structure. In contrast, multi-functional monomers can be used, which contain two or more functional groups per molecule. Multifunctional monomers will then polymerize forming a complex network of linear chains and cross-links between them. Cross-linked polymers are usually stiffer than their linear-polymer made of equivalent mono-functional monomers, and they are typically insoluble in common solvents, making them better suited for MPL of 3D structures.

Multi-functional monomers based on the acrylate or methacrylate chemical group are widely used for MPL for several reasons. Readers will likely be familiar with poly(methyl methacrylate), or "PMMA," which is a linear polymer of mono-functional methyl methacrylate and a useful structural material. This hard, transparent, and comparatively inert material exemplifies many of the useful properties that are common among photopolymers used for MPL based on multi-functional acrylates and/or methacrylates. An immense range of acrylates and methacrylates is commercially available, and their chemical and physical properties are well documented. Acrylates can be readily photopolymerized at room temperature under ambient conditions, and they can be blended to tune physical properties of the pre- and post-polymerized material.

Several different chemistries have been employed for forming a cross-linked polymer in MPL. Acrylates and methacrylates are usually linked together in MPL by photoinitiated free-radical polymerization. But multi-functional epoxides, like the commercially available material "SU-8" have also been used.[23] Epoxides are cross-linked by a process called cationic ring-opening polymerization. In the case of SU-8, the material patterned by MPL is a solid glassy film. The photo-patterning creates a latent image of Brønsted acid, which is the chemically active species. A post-exposure bake is needed to activate cross-linking of SU-8 and convert the sample from a latent photo-image to a rigid 3D replica of the photo-pattern in cross-linked epoxide polymer.

A wide range of photoinitiators have been developed for free-radical and epoxide polymerization. They are typically optimized for single-photon absorption in the UV or at short visible wavelengths. Nonetheless, some of these can also be activated by MPE, when the correct wavelength, pulse-duration, and focusing conditions are selected.[24] Michler's ketone is but one example of a photoinitiator that was developed for conventional single-photon activation, but which can also undergo MPE and be used for MPL.[25, 26] The novel science of photochemical structure, MPE properties, and how these may be exploited to engineer photoinitiators for MPL has been widely explored[7, 12, 27-30] and continues to be a fruitful area of research and development.[31]

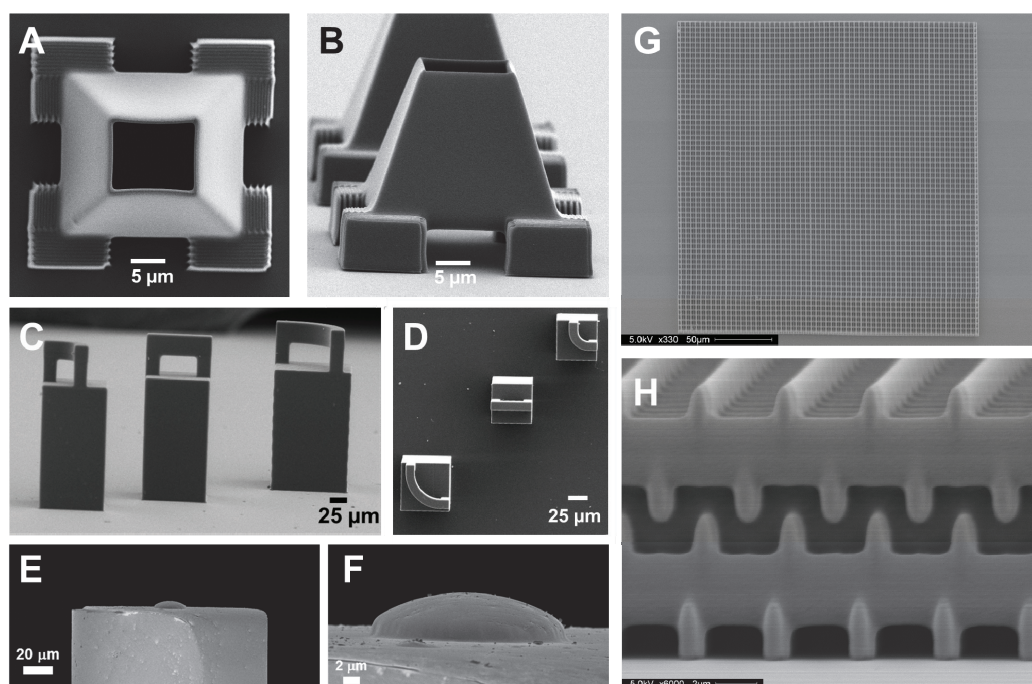


Figure 1. Scanning electron microscopy images showing examples of optically functional devices created by the authors by MPL using photopolymers. (A and B) Top-down and side-views of light-cones. (C and D) Straight and bent wave guides. (E and F) Convex lens fabricated atop an optical fiber. (G and H) Top- and side-views of a face-centered tetragonal photonic crystal.

Polymers satisfy a surprisingly wide range of requirements for applications of MPL. A wider range of properties can be accessed by formulating photopolymer composites MPL containing dyes, nanoparticles, or other additives that imbue the MPL structural material with the required physical or chemical properties.[9, 32-34] Alternatively, post-fabrication processes have been used to modify, chemically transform, or functionalize the surface of 3D structures,[35-39] to deposit other materials onto the polymeric scaffold,[35-37] or to use the polymeric 3D structure as a scaffold into which other materials can be backfilled to get to a targeted form in the required material system.[40] A recent review[8] describes how these approaches have been used to create 3D microstructures in ceramics, composites, semiconductors, metals, and metallodielectrics.

3. FABRICATION OF NANOPHOTONIC DEVICES BY MULTI-PHOTON LITHOGRAPHY

MPL has been used to create a wide range of 2- and 3D structures with unique optical function. Some examples are shown in Fig. 1. Early in its inception, the full power of MPL as a fabrication technique was demonstrated by creating *periodic* 3D photonic crystals.[41-43] In short order, several groups showed that MPL could be used to create polymeric 3D photonic crystals with partial band gaps (band gaps for propagation of light in one or more directions).[44-46] A wide range of structures were created including lattices with symmetries that were simple cubic, face-centered cubic, tetragonal, body-centered cubic, hexagonal, and also chiral or complex spiral and gyroid structures that exhibit properties for circularly polarized light.[47-50] Structures exhibiting complete photonic bandgaps were created by MPL in thermally deposited arsenic sulfide, which is a high-refractive index network solid with transparency in the near infrared.[51] Waveguides, gratings, lenses integrated onto optical fibers, and a range of other optically functional devices have been created.[52-65]

4. SPATIALLY-VARIANT LATTICES

Spatially-variant lattices (SVLs) are a new class of *aperiodic* nanophotonic device that are engineered to direct and control the flow of electromagnetic radiation in 3D.[66-69] SVLs are based on unit cells that control the propagation of light through the self-collimation effect.[70-72] In the most basic form the unit cells in an SVL are spatially varied in orientation throughout the structure so that radiation flows along a prescribed path within the device. The structure of the unit cells within the lattice can also be varied to control other key properties, such as phase and polarization.

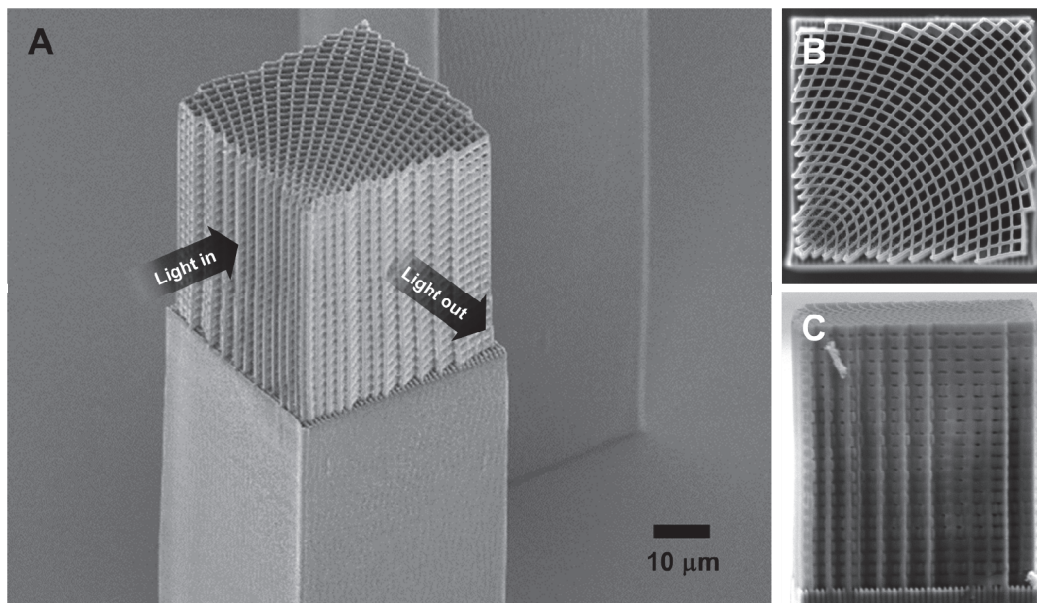


Figure 2. Scanning electron microscopy images of a spatially variant lattice (SVL) configured as a beam bender and fabricated by the authors by MPL in SU-8.[73] (A) Perspective view of the SVL. The lattice spacing is 2 μm . (B) Top-down and side-view and (C) side-view of the beam bending SVL.

MPL was used to fabricate the first example of an SVL that controls the propagation of electromagnetic radiation at optical frequencies.[73] Figure 2 shows an example of an SVLs that was designed to bend a light beam with a vacuum wavelength of $\lambda_0 = 2.94 \mu\text{m}$ through an abrupt turn. These devices were fabricated by MPL in the photopolymer SU-8. Viewed from above, the device has a footprint of $40 \mu\text{m} \times 40 \mu\text{m}$. The SVL consists of a simple-cubic unit cell with period of $\sim 2 \mu\text{m}$ that is spatially varied in a plane parallel to the supporting substrate. Beam-bending SVLs having unit cells with a fill-factor close to 45% were found to strongly self-collimate, and so light incident on the input face is forced to follow the path of spatial-variation through the device to the exit face.

Full characterization of beam-benders like that in Fig. 2 has been reported elsewhere.[73] The key takeaway is that the SVLs can direct light through a turn with a radius as small as $7\lambda_0$, which is a much more abrupt turn than can be achieved via refraction or other means in dielectric media. Other approaches for beam bending based on graded refractive index requires features sizes that are much smaller than λ_0 , and are consequently much more difficult to fabricate. Approaches based on plasmonics can achieve tight beam-bending, but they are inherently lossy. The advantages of SVLs and the new capabilities they afford open many new possibilities for 3D integrated photonics, and a myriad of other applications.

5. CONCLUSION

MPL has grown from an intellectual curiosity to a widely applied method for the fabrication of functional 3D structures. The expansion of the range of material systems available for MPL has supported its use for creating devices that have applications in optics, MEMS, microfluidics, and bio-engineering. Photopolymer materials have supported MPLs immediate use for creating transparent optical devices with novel function. Photonic crystals and other devices that are exceptionally difficult to create by other means were brought into the reach of investigators by MPL. This has supported fundamental and applied studies of 3D photonic crystals and other integrated photonic devices. More recently, the full potential of the method has been illustrated in the fabrication and characterization of functional SPVs, which are aperiodic nanophotonic devices that as yet do not appear to be fabricatable by other means. SVPs offer tremendous scope for application, particularly in the areas of 3D integrated photonics, and potentially new forms of imaging and sensing. MPL will support further development of SVLs and other research in integrated photonics. But the field is wide open for additional contributions in the refinement of MPL, methods for improving resolution, expansion of the range of usable material systems, and improvements in photosensitivity that could help expand its use in research and help move MPL toward the manufacturing floor.

6. ACKNOWLEDGMENTS

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