CAD-integrated system for automated multi-photon three-dimensional micro- and nano-fabrication

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ABSTRACT

Multi-photon three-dimensional micro-/nano-fabrication (3DM) is a powerful technique for creating complex 3D micro-scale structures of the type needed for micro-electromechanical systems (MEMS), micro-optics, and microfluidics. In 3DM high peak-power laser pulses are tightly focused into a medium which undergoes a physical or chemical change following multi-photon excitation at the focal point. Complex structures are generated by serial 3D-patterned exposure within the material volume. To further the application of 3DM to micro-component engineering, we are developing a fully automated and integrated 3DM system capable of creating complex cross-linked polymer structures based on patterns designed in a CAD environment. The system consists of four major components: (1) a femtosecond laser and opto-mechanical system; (2) 3-axis micro-positioner; (3) a computer-controlled fabrication interface; and (4) software for fabrication-path planning. The path-planning software generates a 3DM command sequence based on an object-design input file created using standard commercial CAD software. The 3DM system can be used for start-to-finish design and fabrication of waveguides, 3D photonic crystals, and other complex micro-structures. These results demonstrate a technological path for implementing 3DM as a tool for micro- and nano-optical component manufacture.

Keywords: Multi-photon three-dimensional microfabrication, photolithography, micro-electromechanical systems (MEMS), micro-fluidics, micro-optical systems.

1. MULTI-PHOTON 3D MICROFABRICATION

1.1. 3D-confined multi-photon excitation

Multi-photon three-dimensional micro-/nano-fabrication (3DM) is a photolithographic technique that enables topologically complex 3D micro-structures with feature size as small as 1 μm or less to be generated in a single exposure step by nonlinear photo-patterning in a material.[1-10] The material may be a glass, a polymerizable resin, or even a heterogeneous composite, such as a resin containing dispersed nano-particles. Such 3DM is possible because under tight focusing, two-photon excitation (2PE) and the subsequent material transformation are confined at the focus within a volume of $\sim(\lambda/n_0)^3$, where λ and n_0 are the vacuum wavelength of the exciting radiation and the refractive index of the material, respectively. Degenerate 2PE is a resonant third-order nonlinear optical process in which a species is promoted to an excited electronic state by the simultaneous absorption of two photons of equal energy. Relative to conventional one-photon excitation (1PE), 2PE is typically achieved using longer-wavelength radiation (often red or near infrared), for which the combined energy of two absorbed photons is sufficient to promote the species into one of its lower lying excited electronic states (Fig. 1). The region of high excitation, and thus material

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Micromachining Technology for Micro-Optics and Nano-Optics III, edited by
Eric G. Johnson, Gregory P. Nordin, Thomas J. Suleski, Proceedings of SPIE Vol. 5720
(SPIE, Bellingham, WA, 2005) 0277-786X/05/$15 · doi: 10.1117/12.601187

transformation is tightly confined in 3D-space about the focal point (Fig. 1) due to (1) the geometric narrowing of the beam waist lateral to the propagation axis and (2) the rapid decrease in intensity along the propagation axis (longitudinal) with distance from the focal plane. The optical parameters of the excitation geometry and the physical and chemical response of the material to multi-photo excitation determine the dimensions of the smallest volume element, or voxel, that can be generated and thus the limiting resolution of 3DM.[8]

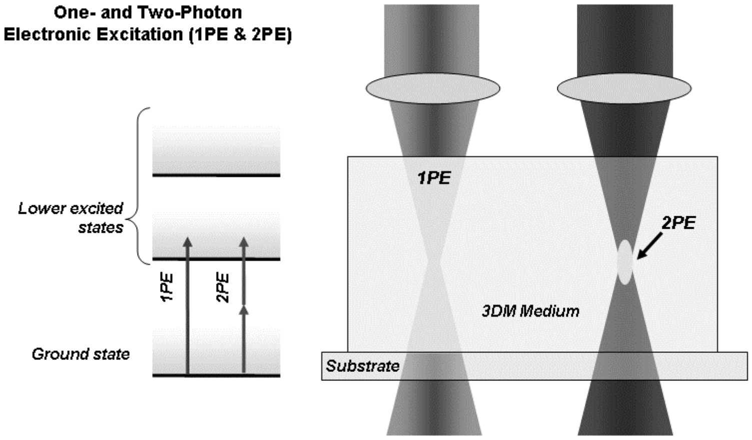

Figure 1. (Left) Jablonski representation of electronic one- and two-photon excitation (1PE and 2PE, respectively). (Right) Illustration of a 3DM medium undergoing 1PE and 2PE using focused short- (left) and long-wavelength (right) radiation, respectively. Under 1PE the medium is excited throughout the length of the interaction volume. Under 2PE the excitation is confined in three dimensions to a small region around the focal point.

1.2. Multi-photon 3D microfabrication process

The most commonly used and arguably the most convenient and reliable excitation source for 3DM is the continuous-wave (CW) mode-locked titanium sapphire (Ti:S) laser. Its emission spans the range of 700 - 1050 nm, making it particularly well suited for two- and three-photon excitation. Ti:S lasers routinely produce sub-100 fs pulses at high repetition rates (~80 MHz) with single-pulse energies, E_p, of ~1 nJ, which corresponds to peak powers of ~10^4 W. For higher peak powers, amplified femtosecond (AFS) lasers can be used which can produce ~100 fs pulses with E_p ~ 1 mJ or higher at repetition rates of 1 - 100 kHz. Given that a continuous microfabricated structure should be generated from partly overlapping voxels, a low repetition rate reduces the speed with which the laser beam may be scanned within the material. Recently, some researchers have turned their attention toward identifying compact solid-state turn-key lasers that could be more convenient and cheaper alternatives to the complex systems described above. A commercial Nd:YAG micro-laser and a mode-locked erbium-doped fiber laser have both been shown to be satisfactory for 3DM in certain acrylate-based media.[11, 12]

The simplest form of 3DM involves patterning a micro-structure within a material using a single tightly focused laser beam that can activate the medium or one of its constituents by 2PE (Fig. 2). The 2PE initiates an irreversible change in the material within the focal volume. The material response depends upon the particular system and is most often photochemical, such as a polymerization reaction, but it can also be photophysical, such as a phase change. The pattern of the target structure is impressed point by point as the focus is translated within the volume of the material. For some applications the final 3D micro-structure is obtained when the unexposed material surrounding the photo-patterned regions is removed, either by a chemical or physical process. When the target structure consists of the photo-transformed material supported within the matrix of the unexposed media, the latter does not have to be removed in a post-exposure process. An example of this case would be the 3DM of optical waveguide circuits embedded within a host slab.

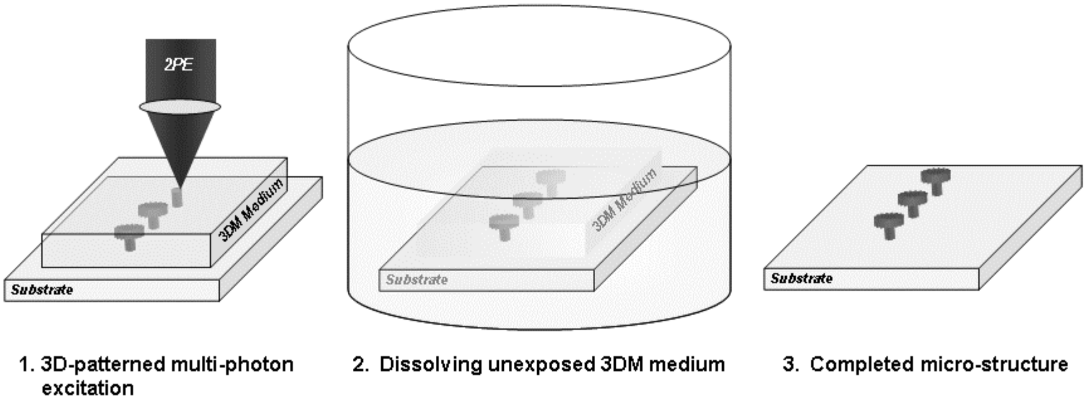

Figure 2. General scheme for single-beam 3DM based on 2PE. (**1. 2PE patterning**): The 3DM medium is translated in 3D-space relative to a tightly focused laser beam which transforms the material only in the region around the focal point. (**2. Development**): In cases where the final structure is needed free from the unexposed material, the sample is treated with a developer that removes the unexposed material. (**3. Completed structure**): Following removal of the unexposed material, the final 3D structure is obtained free-standing on the substrate.

1.3. Polymer systems for multi-photon 3D microfabrication

Cross-linked polymer networks can be generated using multi-functional acrylates and methacrylates or (meth)acrylate-functionalized oligomers and polymers. With sufficient exposure the starting material is converted to less soluble long polymer chains or densely cross-linked polymer networks. The photo-patterned structure can then be "developed" by immersing the sample into a solvent that removes the unexposed material, leaving behind a free-standing structure that is a replica of the photo-pattern (Fig. 2). Several different commercial (meth)acrylate systems have been used successfully for 3DM, such as Nopcocure 800 (San Nopco),[13] and SCR 500 (blend of urethane acrylate monomers and oligomers, Japan Synthetic Rubber Co.).[14] Researchers have also successfully developed custom formulations using blends of commercially available monomers, oligomers, and polymer additives to create resins with tailored physical, chemical and optical properties.[6, 15-17] 3DM based on epoxide polymerization has also been demonstrated using commercial resins including cross-linkable small molecular weight monomers and epoxide oligomers such as SU-8,[18] first developed by IBM. Relative to (meth)acrylates, epoxides shrink substantially less upon polymerization, leading to less distortion of the micro-structure after development. One disadvantage is that epoxide photo-patterning often requires the additional step of a post-exposure bake to accelerate the ring-opening chemistry.

The material systems discussed thus far are "negative-tone" media. This means that only the exposed regions of the material remain after the post-exposure development, so the final structure is a replica of the exposure pattern. In contrast, "positive-tone" media are solid-state systems for which the exposed regions become soluble in a developer.[19-21] The final structure is then the inverse of the exposure pattern. Positive-tone 3DM is desirable for a number of applications. It should be simpler and more expeditious to construct micro-fluidic devices or hollow-channel waveguides by excavating material, rather than building up the walls of each micro-channel in the structure using a negative-tone resist. Perry, Marder, Ober, and collaborators designed a positive-tone material system for 3DM (Fig. 3) based on a high-sensitivity two-photon-activatable photoacid generator and a chemically amplified resist. Using this medium, it was possible to fabricate sub-surface micro-channel structures and micro-gratings using moderate laser scan speeds (50 μm s^{-1}) and exceptionally low average laser power (40 μW).[18, 22]

2. DEVELOPEMENT OF A CAD/CAM SYSTEM FOR 3D MICROFABRICATION

2.1. Opto-mechanical system

Our current experimental set-up for multi-photon 3DM is illustrated in Fig. 4. It can be subdivided into two main sections. The first part consists of a femtosecond Ti:S laser system followed by shutter and a real-time continuous power adjusting system (PAS). The latter is comprised of a half-wave plate, a Glan-Thompson polarizer cube and a silicon photodiode, which provides power feedback for real-time adjustments. Both power management systems (shutter and PAS) are computer controlled and give the flexibility to translate the sample without writing or to create voxels at controlled laser power.

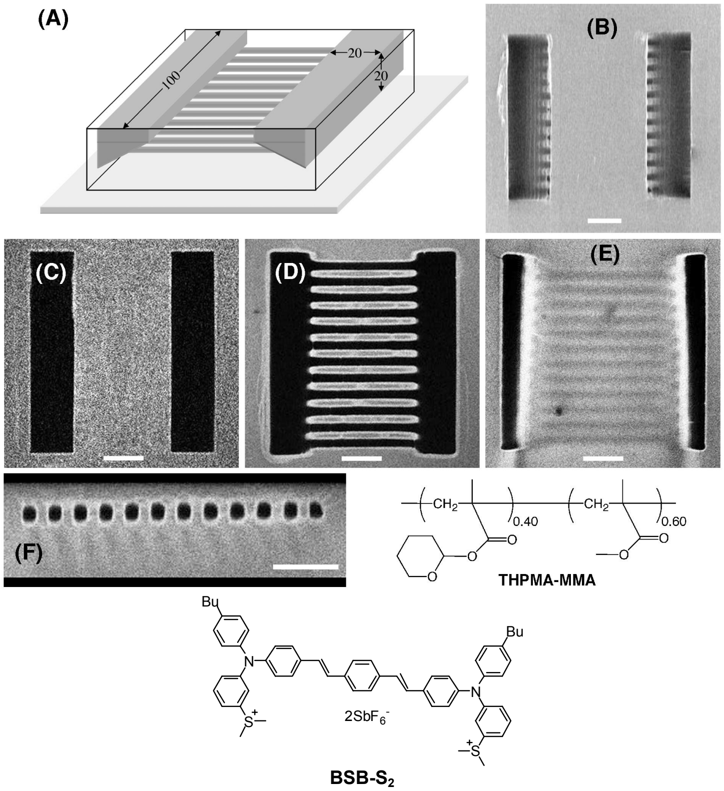

Figure 3. A 3D micro-channel structure fabricated by 2PE-3DM using the positive-tone chemically amplified resist **THPMA-MMA** incorporating 1 wt-% of the two-photon-activatable photoacid generator **BSB-S$_2$**. (**A**) Schematic of the target structure. The dimensions are in units of micrometers. (**B**) Scanning electron micrograph of the final structure, viewed normal to the substrate. (**C** to **E**) Two-photon-fluorescence images of the final structure (viewed normal to the substrate at different depths). (**F**) Two-photon-fluorescence cross-sectional image of the buried channels. The scale bar in **B** to **F** corresponds to 20 μm (W. Zhou *et al.*, *Science*, 2002, vol. 296, pp. 1106-1109).

The second part of the system is a scanning confocal microscope system. It provides a convenient platform for single-beam 3DM. The nano/micro-positioner, which translates the sample relative to the focal spot, is directly mounted on the sample stage of the microscope. The fabrication process is automated as a computer controls the sample translation coordinates and the exposure conditions for each volume element. The confocal system itself enables the medium and the structure to be imaged *in situ*. A 3D representation of the structure can be generated using confocal reflectance or fluorescence imaging. If the material is fluorescent under multi-photon excitation, a 3D image can be obtained by scanning multi-photon fluorescence microscopy. These imaging modes facilitate 3DM by providing a means for locating the focus at an absolute position within the photo-active medium.

The photoactive polymer-coated substrate is affixed to the 3-axis nano/micro-positioner suspended above the objective (in the case of an inverted microscope system). The interior of the photo-active material is patterned during the exposure by translating the sample relative to the focus of the laser beam and shuttering the beam as needed. This configuration is well suited for patterning solid or semi-solid photo-media. An index-matching fluid must be used with high-NA objectives to achieve the smallest focal spot size. If the photopolymer does not dissolve in the fluid, then the two can be placed in direct contact. If the photopolymer is a liquid, a coverslip or other thin transparent barrier must be placed between the index matching fluid and the photopolymer so that the latter does not become contaminated.[17]

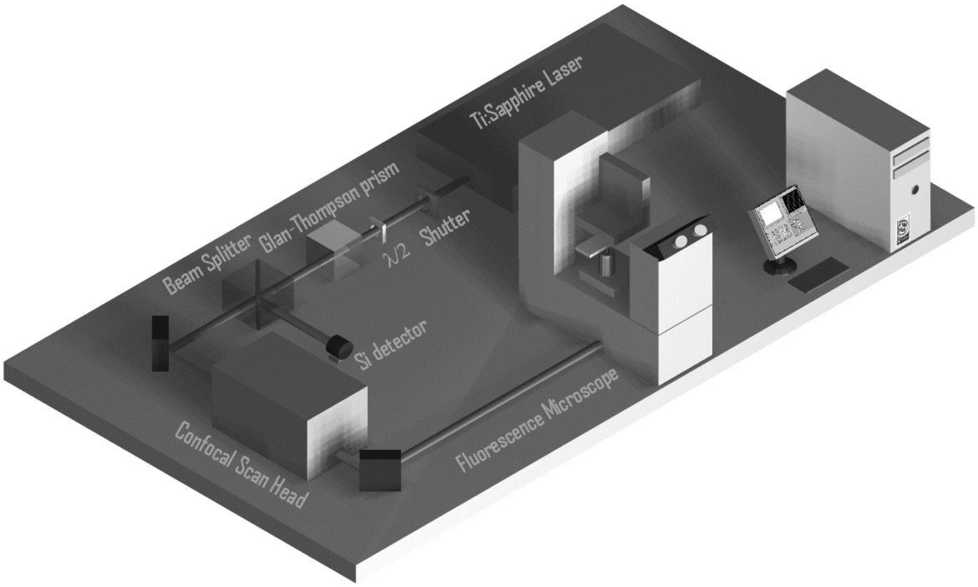

Figure 4. Experimental set-up for multi-photon 3D microfabrication.

3DM can also be realized by moving the focused beam relative to a fixed sample. One convenient implementation utilizes galvo-driven mirrors to scan the beam in planes parallel to the substrate (*xy*-plane). Motion in the z-direction is accomplished by raising and lowering the objective relative to the substrate. The galvo-driven mirrors may be those already present in a scanning confocal microscope system, or they may be part of a separate scan-head dedicated to 3DM. A galvo-based system typically enables faster fabrication, but may not afford motion as precise as that obtained using a multi-axis nano-positioner and limits the patterning area to the image area of the objective.

2.2. Integrated CAD/CAM software interface

We are presently developing an integrated software package that provides an interface for computer-assisted design and manufacture (CAD/CAM) of structures by multi-photon 3DM. The software consists of a Microfabrication Path Planner (MPP) written in ANSI C++ and an Automated Control System (ACS) written in LabView 7.1. A target microstructure is first designed using industry standard CAD software. The CAD design is then exported in the stereolithography file format (STL). In the STL format, the structure volume is defined by a closed, bounding surface comprised of edge-sharing triangles.[23] The STL file is loaded and processed in the MPP, which uses a ray-triangle intersection algorithm to generate a grid of parallel line segments that map the interior volume of the target structure. By convention, the line-segments lie parallel to the *y*-axis, and are spaced in the *x*- and *z*-axes by an interval defined by the user, based on the target resolution and the voxel dimensions (see Fig. 5). The voxel dimensions are determined for a given material prior to fabrication based on a set of laser irradiation and post-exposure developing protocols. The sorted line-segment grid defines the 3DM path. The path definition is exported as a 3DM control file that contains information about the starting and ending coordinates for each line segment, the shutter status during translocation of the sample, and the relative power at which voxels are to be written. The control file serves as the command input for the ACS.

Prior to fabrication a leveling procedure built into the ACS is used to determine the angular tilt of the sample relative to the coordinate system of the translation stage. The photopolymer resin is doped with a multi-photon-excitable fluorescent dye that enables optical identification of the interior volume of the resin and the interface between the resin and the supporting substrate. Multi-photon-induced fluorescence is collected as a function of distance from the objective along the *z*-axis (which is approximately normal to the substrate) at three positions in the *xy*-plane. The plane of the substrate is located using the three coordinates defined by the *z*-position of the sample/substrate interface at each *xy*-position. As microfabrication proceeds, the line segment coordinates are translated into the reference frame of the sample plane in real-time. This procedure eliminates misalignment of the microstructure with respect to the supporting substrate. We are presently optimizing the MPP and ACS software and exploring their use for design and preparation of 3D microstructures.

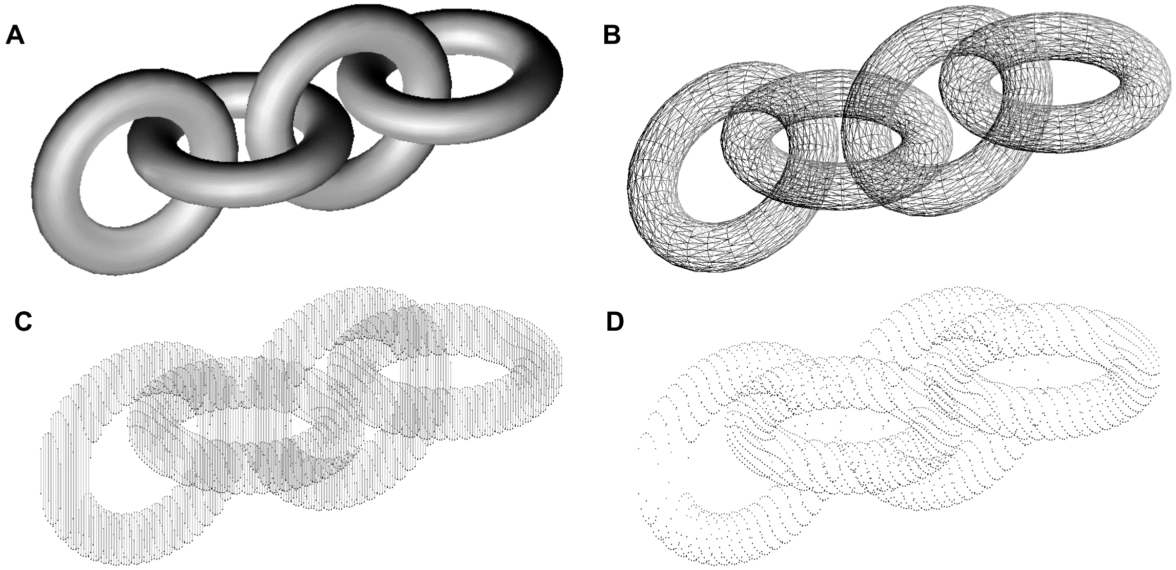

Figure 5. (**A**) Plot of a microstructure design generated using commercial CAD software. (**B**) Export of the CAD design in STL format shown as a "wire-frame" polyhedral surface. (**C**) 3DM path-plan created from the STL input file of (B) using the Microfabrication Path Planner (MPP) software. The vertical lines are the volume-filling line segments along which the focused laser beam would be traced to define the interior volume of the object. (**D**) Starting and ending coordinates for the 3D-microfabrication path-plan of (C), shown with the volume filling-line segments removed.

3. SUMMARY

Multi-photon 3DM offers great promise as a tool for generating micro-devices, such as MEMS, micro-fluidics, and micro-optical components. New frontiers in the technology of such devices require that complex 3D structures, featuring topologically complex shape, high interconnectivity, and extreme under-cut, can be produced in one or more materials that are optimized for a target application in terms of physical, chemical, and mechanical properties. For example, structures with freely movable parts, like those needed for MEMS, are difficult to obtain by other fabrication techniques, but can often be made by 3DM in a single exposure step. Rapid and versatile fabrication is also essential. Creating automated systems that integrate multi-photon 3DM with standard engineering tools, such as CAD/CAM, should help facilitate wider use of this new approach to microfabrication. We have described a first step in this direction that involves the development of software interfaces for CAD-based microstructure design and automated LabView-based control of the multi-photon 3DM process.

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