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Optical lithography of three-dimensional magnetophotonic microdevices

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Abstract. We have recently demonstrated a simple and low-cost fabrication technique, called low one-photon absorption direct laser writing, to realize desired polymeric microstructures. We present the use of this technique for fabrication of three-dimensional magnetophotonic devices on a photocurable homogeneous nanocomposite consisting of magnetite (Fe_3O_4) nanoparticles and a commercial SU8 photoresist. The fabricated magnetophotonic microstructures show strong response to an applied external magnetic field. Thus, various three-dimensional submicromechanical magnetophotonic devices, which can be mechanically driven by magnetic force, are designed and created. Potential applications of these devices are also discussed. © 2018 Society of Photo-Optical Instrumentation Engineers (SPIE) [DOI: [10.1117/1.OE.57.4.041406](https://doi.org/10.1117/1.OE.57.4.041406)]

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1 Introduction

Photopolymerizable resins have been widely used in the fabrication of microstructures and microdevices.¹ However, in many practical applications, functional materials with optical, electric, magnetic, and mechanical properties, which common polymers do not exhibit, are highly desirable. Therefore, functional nanocomposites began to be designed by doping special substances, such as metallic,² semiconductor,³ or magnetic⁴ nanoparticles (NPs), into the common polymers to exploit the hybrid properties. Particularly, magnetopolymeric nanocomposites have attracted great attention due to their multifunctionality for a variety of applications, including data storage,⁵ sensors,⁶ actuators,⁷ biomedicine,⁸ etc. Nowadays, microdevices and micromachines with moving parts have been utilized for environmental applications,⁹ microfluidics,¹⁰ *in vivo* sensing, monitoring,¹¹ and even inside the human body.¹²

Unlike photonic crystals that are merely three-dimensional (3-D) periodic structures,¹³ micromachines and devices generally consist of complex 3-D components, and therefore are more difficult to fabricate. Moreover, micromachines need mechanical movement of individual parts for an operation, which requires not only well-defined, robust microcomponents but also an appropriate machine-driving mechanism is highly desired. Several actuation methods have been proposed, such as an electric field actuation,¹⁴ piezoelectric actuation,¹⁵ thermal actuation,¹⁶ and light-driven actuation.¹⁷ These methods have some challenges, especially for *in vivo* applications. For example, light-driven¹⁷ is an effective method for manipulating micromachines, but it suffers from high laser intensity, which is not suitable to operate inside the human body. The magnetic force-driven technique

would be an ideal method for remote control due to its simple, safe, and noncontact properties.¹⁸

Due to the reasons stated, magnetopolymeric nanocomposites could be a promising candidate for the fabrication and manipulation of micromachines and devices. A number of synthesis and fabrication methods have been proposed to obtain desired magnetic microdevices, for instance, template-assisted method,^{19,20} self-scrolling technique,²¹ lancing angle deposition method,²² and direct laser writing (DLW).^{23–25} Among those, DLW has been the most widely used technique due to its flexibility and capacity of fabricating arbitrary complex 3-D structures. It is a well-developed optical lithography technique based on two-photon absorption (TPA) mechanism, in which, a laser beam is tightly focused into a photosensitive material, called photoresist, to locally induce a polymerization effect. By moving the focusing spot, where the light intensity is high enough to initiate the polymerization, following a programmed trajectory, and after washing away the unexposed area with the developer solvent, the desired polymeric structures are obtained. It is worth mentioning that while the TPA-based DLW technique is very powerful for fabrication of desired structures, it requires the use of a pulsed femtosecond or picosecond laser and a complicated optical system,^{23–25} making it a rather expensive fabrication technique. To overcome this drawback, a simple and low-cost method called low one-photon absorption (LOPA) DLW,^{26,27} which has advantages of both OPA and TPA DLW, has recently been demonstrated. This technique enables the fabrication of 1-D, 2-D, and 3-D structures by using a simple continuous-wave laser at 532 nm with only a few milliwatts.

In this work, we demonstrated the use of LOPA-based DLW as a simple and robust technique for the fabrication of desired multidimensional micromagnetophotonic devices.

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A uniformly dispersed magnetopolymeric nanocomposite is used to fabricate free-floating microstructures and to demonstrate the manipulation of these structures by applying an external magnetic field. A variety of desired magnetophotonic structures and devices with complex 3-D components are also realized.

2 Experimental Setup of LOPA-Based DLW

Figure 1(a) shows the experimental setup of the LOPA-based DLW system. In this system, a continuous-wave laser operating at 532 nm is used. The laser power is monitored by a combination of a half-wave plate ($\lambda/2$) and a polarizer. The laser beam is directed and collimated by a set of lenses and mirrors. In order to realize mapping and fabrication, samples are moved in 3-D by using a 3-D piezoelectric translator, which is controlled by a LabVIEW program. A quarter-wave plate ($\lambda/4$) placed in front of the objective lens (OL) can be rotated to generate a circularly polarized beam for mapping and fabrication. A high numerical aperture (NA) oil-immersion objective was used and placed beneath the glass coverslip to tightly focus the excitation laser beam into the sample. The fluorescence signal emitted by the samples is collected by the same objective, filtered by a 580-nm long-pass filter, and detected by an avalanche photodiode (APD).

To fabricate microstructures, we used the magnetic nanocomposite consisting of magnetite (Fe_3O_4) NPs and a commercial SU8 photoresist. The nanocomposite was synthesized by incorporating magnetic NPs (MNPs) into SU8 matrix. It is important to note that due to the magnetic force (mainly interparticle interactions), MNPs tend to form large agglomerations, causing difficulties in achieving a homogeneous distribution of MNPs in polymer matrix. Hence, different concentrations of Fe_3O_4 MNPs and various

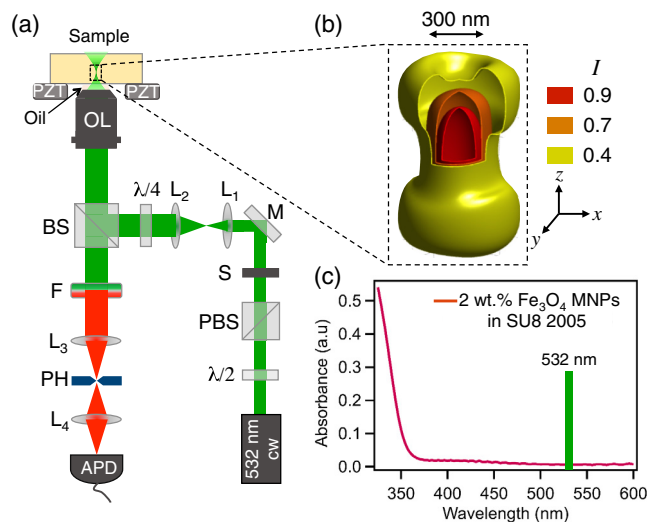


Fig. 1 Experimental setup of the LOPA-based DLW technique. PZT, piezoelectric translator; OL, oil immersion microscope objective; $\lambda/4$, quarter-wave plate; $\lambda/2$, half-wave plate; BS, beam splitter; PBS, polarizing beam splitter; M, mirror; S, electronic shutter; L_i ($i = 1, 2, 3$, and 4), lenses; PH, pinhole; F, 580 nm long-pass filter; APD, avalanche photodiode. (b) Simulation result of intensity distribution at the focus region of a high NA OL. Three surfaces correspond to three isointensities: 0.4, 0.7, and 0.9, respectively. The optical axis of the OL is z axis. (c) Absorption spectrum of the magnetic nanocomposite.

types of SU8 photoresist with different viscosities have been investigated to obtain an excellent dispersion of MNPs in the polymer environment. Finally, the best compromise was achieved by using SU8 2005 photoresist, with a moderate viscosity, and an MNP concentration of 2 wt. %, which is low enough to achieve a homogeneous nanocomposite and high enough to give strong response to external magnetic field.²⁸ We found experimentally that the absorption of the magnetic nanocomposite [shown in Fig. 1(c)] is very similar to that of pure SU8 photoresist, as a result of low concentration of Fe_3O_4 MNPs (2 wt. %). The ultralow absorption at excitation wavelength of 532 nm indicates that the DLW used for the Fe_3O_4 /SU8 2005 nanocomposite operates in the LOPA regime. By using a high NA OL (oil immersion, $\times 100$, NA = 1.3), this low absorption is compensated by a highly focused intensity at the focusing spot. Figure 1(b) shows the simulation result of the light intensity distribution at the focusing region of the OL. The maximal intensity was normalized and three isointensities are shown. This suggests the evolution of the structure size and shape, which is linearly dependent on the exposure doses (excitation power and exposure time) and can be controlled by applying an appropriate dose.²⁷ By scanning the focusing spot, we can obtain desired multidimensional structures, which are similar to those realized by the commercial TPA-based DLW.^{2,3}

3 Magnetic Field-Driven Magnetophotonic Devices

Reversible magnetic field-driven motion of magnetophotonic structures for remote actuation in biomedical applications has recently attracted a growing attention.²⁹ With the aid of an external magnetic field, it is possible to control the displacement of magnetic structures in 3-D as desired. The challenge is to create small magnetic structures that are adaptable to small targets. In order to prove the response of magnetic structures to a magnetic field, arrays of microswimmers were fabricated as an example for demonstration. Figure 2(a) shows the fabrication and demonstration of magnetic field-driven processes. First, an array of microswimmers was fabricated. Unlike other fabrication process where good attachment of structures to the substrate is required, the microswimmers were created at a midway position in order to assure the free-floating ability afterward. After that, a developer solvent was dropped directly on the substrate, washing away all the unexposed areas, leaving the microswimmers, which are free to move inside the liquid. A magnetic field generated by a permanent magnet was then applied to investigate the magnetic field response of the magnetic microswimmers. Figure 2(b) shows SEM image of the microswimmers and the zoomed image of one pattern, fabricated at a power of 9 mW. We note that these microswimmers were fabricated attached to the glass substrate for SEM imaging. Figure 2(c) shows a series of screenshots at 0, 5, and 20 s of a video captured by an optical microscope to illustrate the whole process from the structural development to the movement toward higher gradient of the external magnetic field. Obviously, all of the microswimmers quickly moved toward the magnetic tip, confirming the presence of Fe_3O_4 MNPs inside the structures and their response to the applied magnetic field.

Since the magnetophotonic microstructures respond strongly to the external magnetic field, it is possible to fabricate magnetophotonic micromachines and microdevices

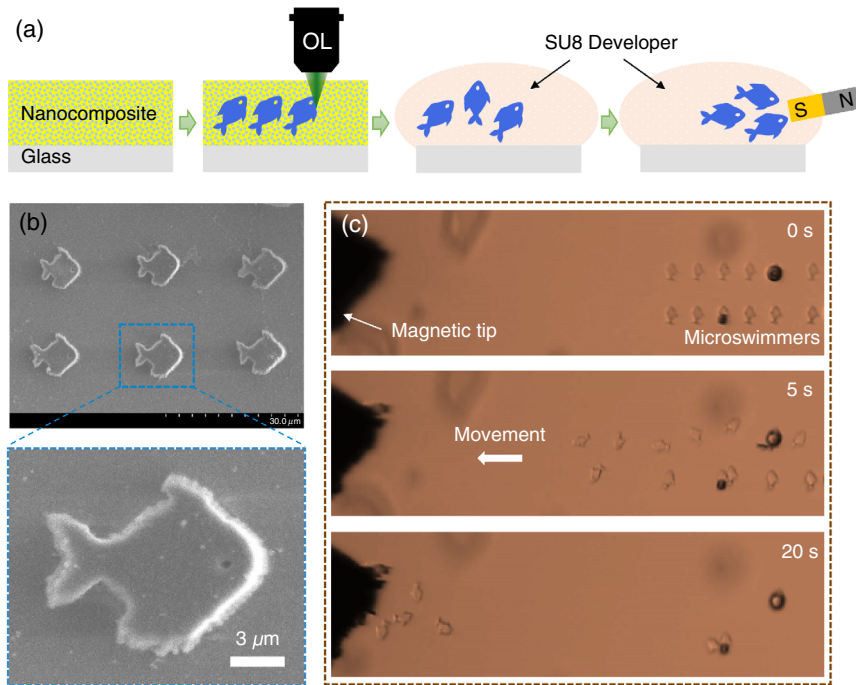


Fig. 2 (a) Illustration of the fabrication and development processes of the microswimmers. (b) SEM images of the microswimmers fabricated by 9 mW. (c) Screenshots showing the movement of microswimmers toward the magnetic tip.

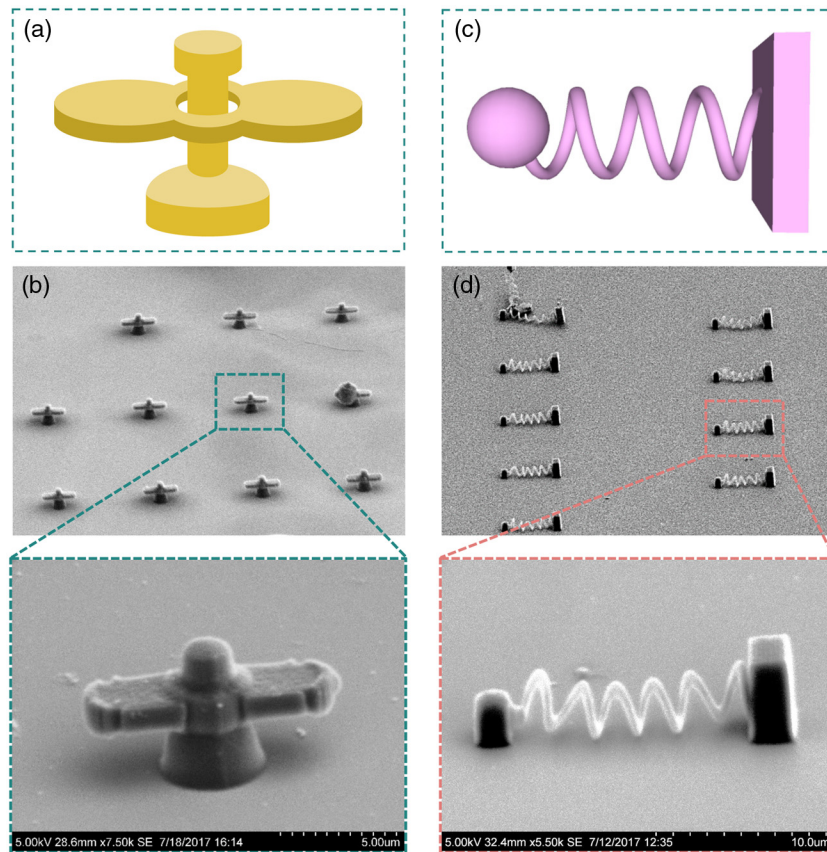


Fig. 3 (a) Design of a microfan. (b) SEM image of an array of microfan and a zoomed image of a microfan (side view). The microfans were fabricated at the power of 8 mW. (c) Design of a microspring. (d) SEM image of an array of micro-oscillator and a zoomed image of a micro-oscillator (side view). The micro-oscillators were fabricated at the power of 8 mW.

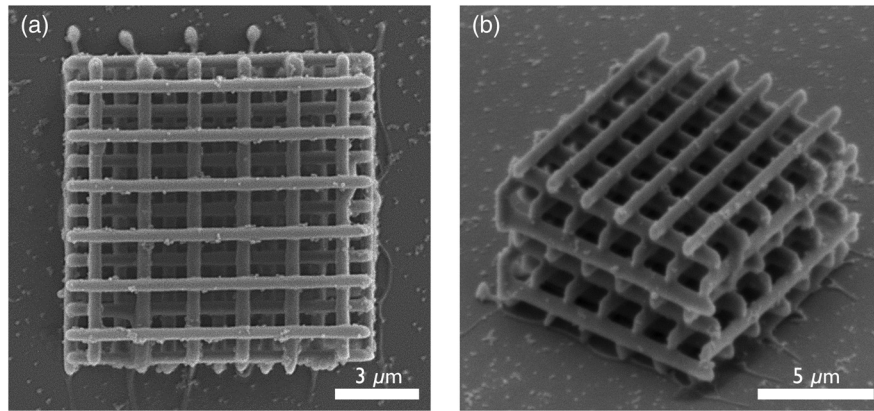


Fig. 4 SEM images of a magnetophotonic submicrometer woodpile structures fabricated by the LOPA-based DLW technique (a) top view and (b) side view.

with complex components, which can be mechanically driven by magnetic force. In the next section, the realization of such microdevices by LOPA-based DLW is demonstrated.

4 Realization of 3-D Magnetophotonic Devices

The great advantage of the DLW method is that it allows the realization of arbitrary structures on demand. The LOPA-based DLW technique has just been demonstrated to be able to pattern magnetic structures from the magnetopolymeric nanocomposite, which show strong response to an external magnetic field. Magnetic micromachines and microdevices have shown their potential for applications in various fields, for example biological, medical fields,^{23,30} and microfluidics,³¹ due to the biocompatibility and flexibility of the magnetic actuation. Figure 3(a) shows a designed model of a microfan which consists of two parts: the central pillar part with two large ends and two blades. The central pillar prevents the microfan from moving away during the development and allows the fan to freely rotate around it like a turbine. External magnetic field can be applied to manipulate the rotation of the fan, making it a potential candidate to be incorporated into a microfluidic system as an active part. Figure 3(b) shows SEM images of the fabricated microfans, which are about $9\ \mu\text{m}$ in length and $5\ \mu\text{m}$ high. Shown in Fig. 3(c) is a model of a microspring, one end of the spring is fixed to a cubic anchor attached to the substrate, and the other end is affixed to a freely moving part. Figure 3(d) shows SEM images of the microsprings fabricated using LOPA DLW. These springs were created with the beads attached to the substrate to keep their true form for SEM imaging. In practice, these beads are free to move. The investigation on the movement of these micromachines needs further testing and are out of scope of this paper. It is also possible for LOPA DLW to fabricate high quality magnetophotonic crystals.³² Figure 4 shows SEM images of a woodpile structure [top view (a) and side view (b)], which is made of 10 alternating layers separated from each other by $1\ \mu\text{m}$. The distance between two lines in x - and y -directions is $2\ \mu\text{m}$. These photonic crystals made by materials possessing magnetic property will allow to control and modify the propagation and polarization of the light since the refractive index and the photonic crystals periodicity are now magnetic field dependent. An external magnetic

field thus allows ability to obtain a tunable photonic bandgap.

All of these experimental results confirm that any magnetophotonic microstructures or devices can be realized using the LOPA-based DLW on this magnetopolymeric nanocomposite. It opens a possibility to go further on development of magnetic nanodevices and microrobotic tools for a wide range of applications.

5 Conclusions

In this work, we have successfully demonstrated the fabrication of magnetophotonic microstructures and devices on demand utilizing the LOPA-based DLW technique. A homogeneous composite of SU8 photoresist and Fe_3O_4 MNPs was used and enables the fabrication of high-quality magnetic microstructures. Free-moving microswimmers with strong response to an applied external magnetic field were fabricated and demonstrated. The LOPA-based DLW technique was also successfully employed to realize desired 3-D magnetophotonic crystals and microdevices with complex 3-D components. The strong and controllable magnetic field response of the fabricated magnetophotonic structures is very promising for different applications, such as tunable photonic structures based on magneto-optical effect, and microrobotic tools for transportation for biological and medical fields.

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