AIP Applied Physics Letters

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Citation: Applied Physics Letters **93**, 051107 (2008); doi: 10.1063/1.2967872
View online: http://dx.doi.org/10.1063/1.2967872
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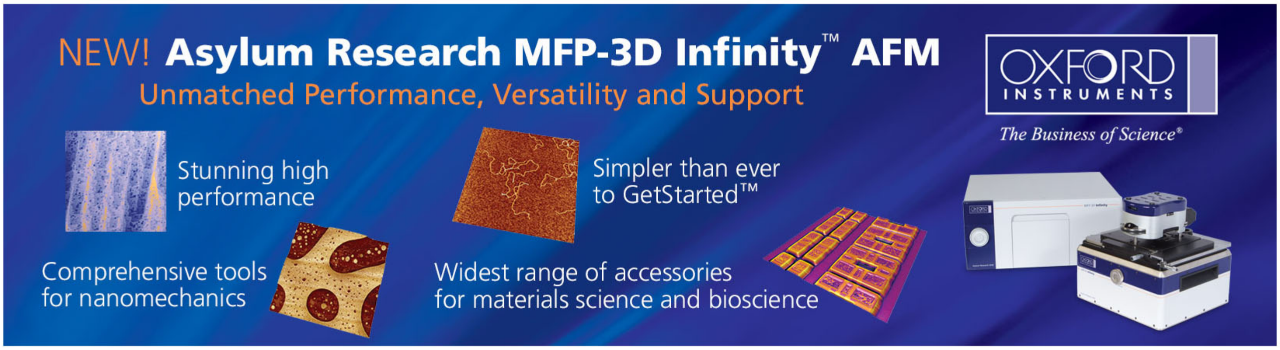

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APPLIED PHYSICS LETTERS **93**, 051107 (2008)

Laser microfabrication and rotation of ship-in-a-bottle optical rotators

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(Received 24 May 2008; accepted 10 July 2008; published online 5 August 2008)

We have fabricated optical rotators inside a silica substrate and rotated them by a laser trapping technique. The fabrication method used was femtosecond laser-assisted etching, i.e., modification of the host material by irradiation with femtosecond laser pulses along a predesigned pattern, followed by selective chemical etching. The rotators, which consist of the same material as the substrate, can move inside the microcavity but cannot get out. The rotation speed was proportional to the trapping laser power, and the maximum achieved was about 100 rpm. Such rotators will be applicable to micro-total-analysis systems and microfluidics. © *2008 American Institute of Physics*. [DOI: 10.1063/1.2967872]

The invention of the laser opened up new fields in optical science and technology. Laser trapping has attracted much attention as a promising laser technique. Ashkin proved that optical pressure generated by an intense laser beam can be used to trap micrometer-scale transparent objects.[1,2] Later, trapped objects were rotated by using the angular momentum of the trapping laser beam,[3] birefringence of the trapped object with a circularly polarized laser beam,[4,5] and shape anisotropy of the trapped object.[6] Higurashi *et al.* have demonstrated that especially designed artificial anisotropic objects can be rotated by laser trapping along the axis of the trapping laser beam.[6] They fabricated such "optical rotators" made of silica using semiconductor technology. Optical rotators were also fabricated with photopolymerizable resin by femtosecond (fs) laser microfabrication.[7,8] In addition, rotation along an axis perpendicular to the optical axis has also been demonstrated.[9]

When such a micrometer-sized rotator is incorporated into micro-total-analysis systems (μ-TAS), it can serve as an active element of devices such as pumps and mixers.[10] In addition, the noncontact rotation would be suitable for active elements working in a closed space. Thus we recently proposed the concept of a ship-in-a-bottle optical rotator.[11] As the name indicates, the object exists inside a cavity that is embedded in a solid substrate, and the object cannot get out of the cavity because it is larger than the cavity's outlet. In this letter we report the fabrication of "ship-in-a-bottle" micro-optical rotators by the femtosecond laser-assisted etching technique. The technique consists of two steps. (i) The region to be etched out is irradiated by focused femtosecond laser pulses. Each pulse modifies a small (on the order of cubic micrometers) volume, and accordingly many continuous spots are irradiated in order to modify the whole region. (ii) The substrate is immersed in an etching solution, then the photomodified region is selectively removed by chemical etching if nature allows. This technique allows us three-dimensional removal processing inside solid materials with a micrometer-order spatial resolution. Kondo *et al.* reported this technique for the fabrication of microchannels inside a photosensitive glass.[12] Marcinkevičius *et al.* reported that this technique can be applied to a normal (nonphotosensitive) material of silica glass.[13] This technique has been applied to several kinds of microstructures, for example, a microdye laser,[14] a microfluidic device with an air- pressure-driven microvalve,[15] and primitive micro-optofluidic devices combined with waveguides that are also written in solid substrates using a femtosecond laser.[16,17] The air-pressure-driven microvalve by Masuda *et al.* is a kind of ship-in-a-bottle structure, but it is too large (on the order of millimeters) to be actuated by laser trapping. Here we demonstrate both the fabrication and actuation of microrotators by these laser techniques.

The samples used in the present study were synthetic silica glass substrates (Shin-Etsu Chemical Co., Ltd.). The substrates, each with a thickness of 0.625 mm, were cut into about 10×10 mm^2 pieces and used for the experiments.

A Ti:sapphire regenerative amplifier (Spitfire, Spectra Physics) was used as the femtosecond light source for photomodification. The femtosecond pulses of 800 nm wavelength were led to an inverted microscope (IX-70, Olympus) and focused by an objective lens [100×, numerical aperture (NA)=1.35]. The typical pulse energy was 75 nJ (measured before the microscope). The sample was translated by a computer-controlled three-axis piezoelectric stage (P-563.3CD, Physik Instrumente), and laser irradiation was controlled by a shutter, so that a cuboid cavity region was photomodified, leaving the region of the optical rotator. The basic design of the optical rotator, a four-wing shape, is almost the same as that by Higurashi *et al.*[6] Its cross section has a fourfold rotational symmetry and an inversion symmetry, but no mirror symmetry. In addition to the cuboid cavity that includes the optical rotator, four paths are also irradiated from the surface to the cavity so that the etching solution can reach the cavity. The spatial distance between adjacent photomodified points along the optical axis, p_z, was fixed to 2.0 μm, and the distance perpendicular to the optical axis, p_x ($=p_y$), was varied among 0.25, 0.5, and 1.0 μm.

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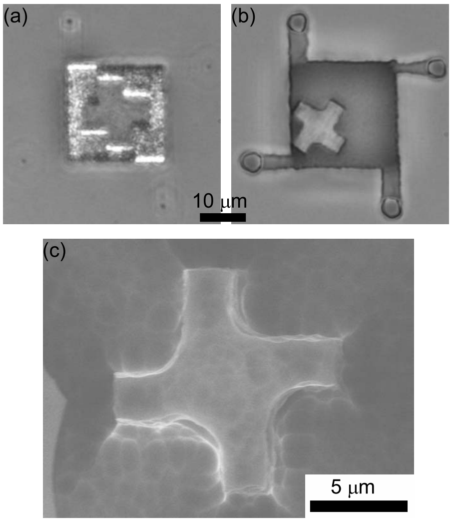

FIG. 1. [(a) and (b)] Optical micrographs of an optical rotator with $p_x = 0.25\ \mu$m (a) before and (b) after etching for 2 h. (c) Scanning electron micrograph of an optical rotator whose bottom is not separated from the substrate.

After the photomodification, the sample was immersed in aqueous solution of 2% hydrofluoric acid for 2 h at room temperature for the selective etching of the photomodified region.

The light source used for laser trapping was a cw neodymium-doped yttrium aluminum garnet laser (Spectra Physics, J20U-S3-CW) with a wavelength of 1064 nm. Laser trapping was carried out on an inverted microscope (Olympus, IX-71).[18] The laser beam was focused by an oil-immersion objective lens of 100× (NA=1.40), and the motion of the trapped rotator was observed through the same objective. A comparative experiment was carried out using a 10× dry objective lens (NA=0.40). The silica substrate, inside which the rotator was fabricated, was put on a coverslip and set on the stage of the microscope. The empty space around the rotator was filled with water. The motion of the rotator was monitored by a charge coupled device camera and recorded by a HDD/DVD recorder, and an image sequence with 30 frames/s was extracted.

Figures 1(a) and 1(b) show the optical micrographs of the rotator before and after etching with $p_x = 0.25\ \mu$m. During etching, the optical rotator became smaller than the unphotomodified region before etching, and was about 12.5 μm in width. This width was still much larger than the diameter of the paths, as can be seen in Fig. 1(b). It is also clear from Fig. 1(b) that the position of the optical rotator changed after etching. In addition, Brownian motion of the rotator was observed when the cavity was filled with water. These results indicated the rotator was separated from its surroundings and thus movable inside the cavity. In other words, a truly ship-in-a-bottle movable structure was obtained. A similar result was obtained for the rotator with $p_x = 0.5\ \mu$m, although the etching was not enough to separate the rotator under the present etching condition for $p_x = 1.0\ \mu$m.

Close-up views of the rotators were obtained by a scanning electron microscope. For this purpose, the region under the bottom of the rotator was not photomodified, so that the rotator was fixed to the substrate; after etching, the top wall of the cavity was removed by mechanical polishing. Figure 1(c) shows a scanning electron micrograph of an optical rotator with $p_{xy} = 0.25\ \mu$m. The rotator has smooth surfaces with submicrometer roughness.

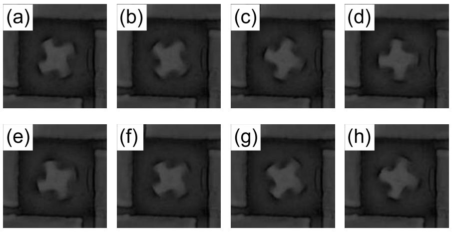

FIG. 2. Series of sequential video frames (30 s^{-1}) of an optical rotator, where a 4 W laser beam was focused by the 100× objective lens.

The fabricated rotators spun under laser trapping [see movie in EPAPS (Ref. 19)]. Figure 2 shows a series of sequential video frames of the trapped rotator on which a 4 W laser beam was focused by the 100× objective lens. The rotator rotated counterclockwise at about 100 rpm, which was calculated from the video frames. This rotation direction coincided with that observed by Higurashi *et al.*[6] When the rotator was reversed, it spun in the opposite direction. These results indicated rotation was induced due to the shape of the rotator.

The trapping laser power dependence of rotation speed is shown in Fig. 3. The rotation speed was almost proportional to the trapping laser power in the measured power range when the 100× objective lens was used. The rotation speed is determined by the balance between torque and viscous drag. The torque is exerted by the optical pressure that originates from the trapping laser beam; correspondingly, the torque is proportional to the laser power. The viscous drag is proportional to the rotation speed. Thus it is reasonable that the rotation speed is proportional to the trapping laser power. For comparison, rotation speed with the 10× objective lens is also plotted at the laser power of 4 W. This revealed that the rotation speed was much lower with an objective lens of smaller NA, indicating that only the light that the side surfaces refract or reflect contributes to the torque.[6,20]

It should be noted that the rotation speed differed from sample to sample, while a linear dependence of rotation speed was confirmed for every sample. This may be because of small differences in the morphology of individual rotators. Thus, a quantitative comparison of rotation speed among samples was impossible at the present stage. All the data plotted in Fig. 3 were obtained from the same rotator. The sample to sample difference in rotation speed was within

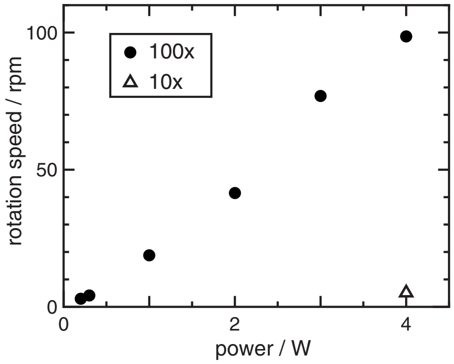

FIG. 3. Laser power dependence of rotation speed.

15%; this value was much smaller than the difference in rotation speed between the two objective lenses used. In addition, an unexpected dependence on the polarization of trapping laser beam was observed in some rotators. Further investigation of the effect of fabrication precision on rotation behavior is necessary.

In the present study, only one ship-in-a-bottle rotator was fabricated and rotated in a microcavity. In principle, however, there is no limit to the number of rotators. Many rotators in microcavities connected by microchannels can be fabricated in batches. Simultaneous spinning of rotators by laser trapping is also possible by using a holographic technique.[21] Integration of microcavities or microchannels with implanted active elements will be advantageous in μ-TAS and microfluidics research.

In conclusion, we have fabricated optical rotators confined in a microcavity inside a silica glass substrate by using femtosecond laser-assisted etching. The rotators were spun by a laser trapping technique. The rotation speed was proportional to the trapping laser power, and the maximum speed observed was about 100 rpm. Such rotators would contribute to a progression of μ-TAS.

This work was partly supported by the Association for the Progress of New Chemistry and the Venture Business Laboratory of the University of Tokushima.

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