Laser Fabrication of Ship-in-a-bottle Microstructures in Sapphire

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In this paper we propose the concept of a ship-in-a-bottle optical rotator, which is a microscale object, rotatable by a laser manipulation technique, inside a transparent solid. Femtosecond laser-assisted etching of sapphire was used to fabricate this microstructure. We report our attempts at fabrication, and discuss the strategy for completing the fabrication.

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The technique of laser trapping (or laser manipulation, laser tweezers) was proposed by Ashkin more than 30 years ago [1]. This technique made the three-dimensional (3D) positioning of small objects possible using a tightly focused laser beam [2]. The fields of application of laser manipulation are diverse and include biology, physics, and micromachining, to name a few.[3,4]

In addition to the control of position (momentum), the laser trapping technique can simultaneously control the rotation (angular momentum) of the object in some cases. Control of angular momentum is achieved in several ways. In one, the angular momentum of the laser beam is used [5-7]. A photon of a circularly-polarized laser beam has an angular momentum of $|\boldsymbol{\Omega}| = h/2\pi$ where h is the Planck's constant. When a circularly-polarized (or elliptically-polarized) laser beam is used to trap a birefringent object, the angular momentum of the laser beam will be transferred to the object trapped, thus causing it to rotate.

The second possible way of controlling the angular momentum of the laser-trapped object uses the torque which originates from the trapped object's asymmetrical shape. An object with rotational symmetry about the optical axis but without reflection symmetry about the plane which includes the optical axis can be rotated by laser trapping. Such a structure is called an "optical rotator." Higurashi et al. proposed a four-blade-shaped optical rotator, fabricated one by reactive ion-beam etching of SiO_2, and observed rotation during laser trapping [8]. Furthermore, optical rotators with other shapes have been reported by Galajda et al. [9] (whose rotator was made of photopolymers by two-photon lithography) and by Harada et al. [10] (using a bio-material).

Here we report an attempt to fabricate a ship-in-a-bottle optical rotator, i.e., an optical rotator confined in a microcavity inside a transparent solid substrate. The optical rotator is separated from the substrate; thus it is free to move inside the microcavity. The channel from the surface to the microcavity is narrower than the optical rotator; thus the rotator cannot move out of the substrate. Such an optical rotator may act as a micro pump and micro mixer.

A schematic view of the intended structure is shown in Fig. 1. The design of the four-blade optical rotator is similar to that of Higurashi et al [8].

To realize such an optical rotator within a microcavity, a 3D microfabrication technique for the removal process is required. We adopted a femtosecond (fs) laser-assisted etching technique [11-16], which consisted of two steps. The first step was the irradiation of focused fs pulses to a sample substrate along the pre-designed pattern of an empty space, so that the laser-irradiated focus spots will be modified. The second step was a wet etching of the substrate. If the laser-modified spots were selectively etched out, we could obtain 3D microstructures inside the substrate. Because of this unique capability for 3D microstructuring inside a solid substrate, the fs laser-assisted etching technique is suitable for the present purpose. Both photosensitive [11-13] and non-photosensitive [14-16] materials have been subjected to the fs laser-assisted etching technique. In the present study, sapphire (Al_2O_3 crystal) was used as a sample material, because of its high etching selectivity ($>10^4$ [15], defined as the ratio of the etching rate in the laser-irradiated region to that in the un-irradiated region). A high selectivity enables us to process finer structures.

An fs Ti:Sapphire regenerative amplifier was used as a light source. Focused fs pulses with a central wavelength of 800 nm were irradiated to the sample of sapphire substrate. The sample position was controlled by a Piezoelec-

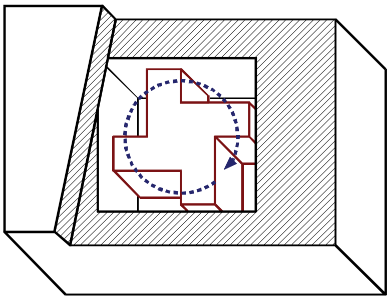

Fig. 1 Design of the "ship-in-a-bottle" optical rotator (local sectional view). The broken arrow indicates the direction in which the rotator will rotate under laser trapping.

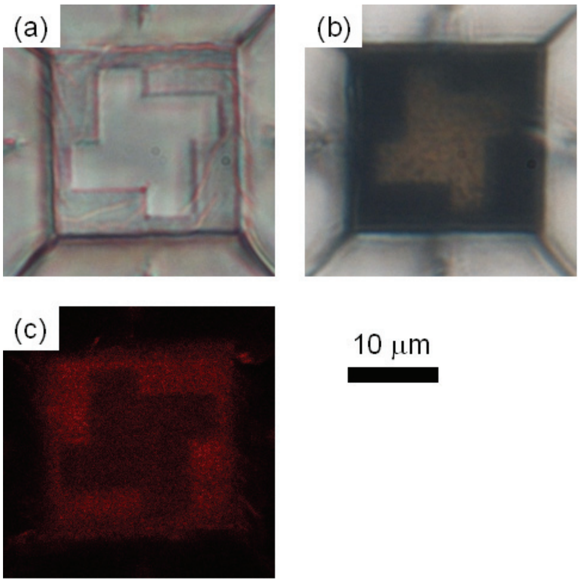

Fig. 2 Optical micrographs of the fabricated “ship-in-a-bottle” optical rotator (top views). (a) Bright-field image after laser irradiation (before etching). (b) Bright-field image after etching. (c) Confocal image after etching.

tric Transducers (PZT) stage synchronized to the laser pulse, so that single pulses were irradiated to the individual spots. The in-plane (direction perpendicular to the surface normal) distance between the spots was 0.25 μm, and the plane-to-plane distance was 1.0 μm. An objective lens of 100x, NA=1.35 was used. The typical pulse energy was 45 nJ. The designed width of the blade of the optical rotator was 9 μm, and the height was 15-20 μm. The microcavity that confined the optical rotator was a cuboid shape, located at a depth of 10 μm subsurface. In addition, four lines were also irradiated from the surface to the microcavity, which will constitute microchannels and bring the etchant to the microcavity. The etchant used was a 10 % aqueous solution of hydrofluoric acid, and the typical etching span was several days.

Figure 2 shows optical micrographs of the fabricated optical rotator with a designed height of 20 μm. Figure 2(a) is the bright-field image before etching. The shape of the optical rotator is recognizable. The region around the optical rotator darkened after wet etching as shown in Fig. 2(b). Darkening is observed when the laser-irradiated region is etched out [16]. Thus this result suggested successful fabrication of the object. To confirm the formation of empty space, confocal microscopy observation was also carried out. Before the confocal observation, the sample was immersed in an aqueous solution of a luminescent dye, in order to fill the etched region with the dye solution. Then the sample was picked up, and a 3D luminescence image was obtained using a confocal microscope. As shown in Fig. 2(c), luminescence was observed from the region surrounding the optical rotator. This result confirmed the formation of continuous empty space in the laser-irradiated region.

Although these optical observations suggested successful fabrication of the optical rotator, we could not make the rotator move. To determine the reason for this, we mechanically polished the sample and examined the etched region directly by scanning electron microscopy (SEM). Then, we found mesh-like un-etched residues at the region where the solid materials should have been removed, as shown in Fig. 3. The pitch of the mesh was 0.25 μm, which is equal to the in-plane distance of the laser-irradiated spots. This result indicates that there remain unetchable regions between the adjacent laser-modified spots. Because of the small pitch, the residue was not recognizable using optical microscopies. Such a mesh-like structure may be useful as a submicrometer-scale sieve or heat exchanger, but was not suitable for the present purpose.

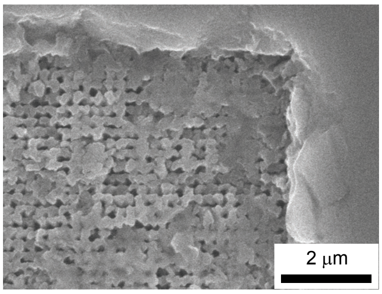

Fig. 3 SEM image of mesh-like residues.

The critical difference between the present fs laser-assisted etching and that reported in previous studies is the volume of the etched-out region. Previous studies mainly focused on the fabrication of microchannels (the removal of a thin line region). The present result indicates that the removal of a region with a large volume, which is required for the fabrication of the optical rotators, is more challenging than the formation of microchannels.

To improve the etching of large-volume regions, we tried several different irradiation geometries by varying the pitch of the laser-modified spots and/or the relative positions of the spots between the adjacent planes. In some cases the etching was improved, but an improvement significant enough to allow us to make a freely movable structure was not attained. In sapphire, it was reported that the irradiation of a focused fs pulse amorphized the focus region, and only the amorphized region was etched out in an aqueous solution of hydrofluoric acid [15]. In addition, irradiation of multiple pulses re-crystallized the amorphized sapphire [15]. The re-crystallization is one of the factors that hinders complete etching.

Another strategy for reaching the goal is to find a new combination of host material and etchant. Sapphire and an aqueous solution of hydrofluoric acid were used in the present study because of their high etching selectivity. However, as a consequence of the high selectivity, the etchable region is strictly confined to the laser-modified region. Thus, un-etched residues are likely to remain unremoved. A combination of host material and etchant in which the etching rate of the un-modified host material is low but not zero (e.g., an etching length of a few hundred nanometers within the etching span) is preferable.

In summary, we have proposed the concept of a ship-in-a-bottle optical rotator. We have tried to fabricate such a structure by the femtosecond laser-assisted etching of sapphire. However, we were not able to complete the fabrication due to un-etched residues. The strategy for fabricating a freely movable optical rotator was discussed.

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