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# Fabrication of diffractive optical elements inside polymers by femtosecond laser irradiation

Hiroyuki Mochizuki <sup>a,\*</sup>, Wataru Watanabe <sup>a</sup>, Yasuyuki Ozeki <sup>b</sup>, Kazuyoshi Itoh <sup>b</sup>, Katsumi Matsuda <sup>c</sup>, Satoshi Hirono <sup>c</sup>

<sup>a</sup> Photonics Research Institute, National Institute of Advanced Industrial Science and Technology, 1-8-31 Midorigaoka, Ikeda, Osaka 563-8577, Japan

<sup>b</sup> Department of Material and Life Science, Graduate School of Engineering, Osaka University, 2-1 Yamadaoka, Suita, Osaka 565-0871, Japan

<sup>c</sup> Production Method Development Group, Omron Corporation, 2-1-2 Nishikusatsu, Kusatsu, Shiga 525-0035, Japan

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#### ABSTRACT

Bragg-type gratings were prepared by irradiation inside a series of optical polymers with femtosecond laser pulses and the preparation conditions of the grating were examined. Repeated scanning irradiation with femtosecond laser pulses formed gratings due to refractive index changes inside polymers. Among the polymers examined in the present study, polymethylpentene (PMP) showed the highest diffraction efficiency, which was an order of magnitude higher than those of other optical polymers. The density of PMP was the lowest among the polymers evaluated in the present study, and the large volume contraction based on its low density was responsible for the larger refractive index change of PMP. Furthermore, we fabricated large-area diffractive optical elements (DOEs) in PMP measuring  $15 \times 25$  mm<sup>2</sup> by widening the scanning area. © 2009 Elsevier B.V. All rights reserved.

#### 1. Introduction

Polymer optical components have a great deal of promise for use in electro-optical devices, as polymers have a number of advantages, including cost-effectiveness, low weight, and design freedom. Among these polymer optical components, diffractive optical elements (DOEs) have attracted a great deal of attention with regard to their light-solving, light-gathering, and light-splitting capabilities.

There have been a number of reports on DOEs with formation of periodic structures on their surfaces [1]. However, these DOEs have disadvantages including low contact toughness and the need for a complex etching process. On the other hand, DOEs prepared inside polymers have greater toughness and are suitable for photonic three-dimensional device integration.

Femtosecond laser processing allows the preparation of photonic devices in three-dimensions inside transparent materials. Focused femtosecond laser pulses induce structural changes inside transparent materials, such as glass and polymers, due to nonlinear absorption by enormous peak power. Waveguides have been fabricated inside various types of glass by irradiation with femtosecond laser pulses [2,3], and waveguide fabrication within poly(methyl methacrylate) (PMMA) by femtosecond laser pulses irradiation has also been reported [4,5]. DOEs using PMMA plates were fabricated at their surfaces or within them by translating the laser focus area [6,7]. In all

previous studies, the refractive index in the laser focus area was increased compared with that before irradiation. However, effective mechanisms for these positive refractive index changes are not yet clear, and we suggested previously that polymer densities maybe related to their refractive index changes induced by laser irradiation [8].

Here, in fabrication of DOEs within a series of transparent polymers, we discuss the important process factors and the mechanisms responsible for the refractive index changes by femtosecond laser irradiation. Furthermore, we report the fabrication of large-area DOEs inside polymers.

#### 2. Experimental

In the present study, we employed a series of polymers designed for optical use (Fig. 1 shows the chemical structures of the polymers): PMMA (Acrylite L #000; Mitsubishi Rayon Co., Ltd.), polycarbonate (PC, Panlite AD-5503; Teijin Chemical Ltd.), cycloolefin polymer (COP, Zeonex 480; Zeon Corporation Ltd.), polymethylpentene (PMP, TPX RT-18; Mitsui Chemical Inc.), poly(methyl methacrylimide) (PMMI, Pleximid 8805; Degussa Co., Ltd), and polystyrene (PS, GPPS K27; PS Japan Co. Ltd.). All samples were formed with dimensions of 2–  $4 \times 10 \times 10$  mm<sup>3</sup>. The characteristics of all polymers are summarized in Table 1 (data taken from reference [9]).

Fig. 2 shows a schematic representation of the optical setup used for preparing DOEs inside the polymers. Laser pulses of 120 fs with a wavelength of 800 nm were generated by a Ti:sapphire laser system

<sup>\*</sup> Corresponding author. E-mail address: h-mochizuki@aist.go.jp (H. Mochizuki).

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Fig. 1. Chemical structures of the optical polymers used.

 Table 1

 Characteristics of polymers used in the present study.

Grade	PMMA	PC	COP	PMP	PMMI	PS
	Mitsubishi	Teijin	Zeon;	Mitsui	Degussa;	PS
	Rayon; #000	Chemical; AD-5503	Zeonex 480	Chemical; RT-18	8805	Japan; SGP10
Refractive index (n <sub>d</sub> )	1.491	1.585	1.531	1.463	1.526	1.591
Tg (°C) <sup>*</sup>	120	142	140	30 (Tm = 227 <sup>**</sup> )	165	100
LEC <sup>***</sup>	7	8	6	12	5	7
Density (g/cm <sup>3</sup> )	1.19	1.20	1.01	0.83	1.21	1.05

\* Glass transition temperature.

\*\* As PMP is a crystalline polymer, it is valid to describe a melting point (Tm) to compare thermal properties.

\*\*\* Linear expansion coefficient; (1/10<sup>5</sup> °C).

and a repetition rate was 1 kHz. The pulse energy was attenuated by rotating a half-wave plate in front of a polarizer. The laser pulses were focused with a  $5\times$  microscope objective lens (numerical aperture of 0.13), and the focal point was located 500 µm beneath the sample surface. The sample was placed on a computer-controlled translation stage for scanning the focal point. Periodic structures with a period of 10 µm were fabricated inside the polymers.

The diffraction efficiency of each grating was measured with a continuous wave He:Ne laser at a wavelength of 633 nm. The beam from the He:Ne laser was incident upon the grating at the Bragg angle, and the diffracted beam pattern was projected on a screen, which was placed 500 mm from the sample. The first-order diffraction efficiency ( $\eta_1$ ) of each grating was calculated by measuring the beam intensity in the first-order diffraction spot with a power meter (see Fig. 3), and defined by dividing the first-order diffraction beam intensity by intensity of total transmitted light.

The Q value, a parameter used to judge the diffraction type, is given by the equation  $Q = 2\pi\lambda L/n\Lambda^2$ , where  $\lambda$ , *L*,  $\Lambda$ , and n are the wavelength of the beam (633 nm), the thickness of the diffraction grating, the grid interval, and the refractive index of the polymer, respectively. In the present study, we prepared Bragg-type gratings with Q values greater than 1. Therefore,  $\eta_1$  can be written as  $\eta_1 = \sin^2(\pi\Delta nL/\lambda\cos\theta)$ , where  $\Delta n$  and  $\theta$  are the refractive index change and the Bragg angle, respectively [10]. We estimated the refractive index change of the gratings using the above equation.

#### 3. Results and discussion

#### 3.1. Important process factors

The scanning speed and laser energy were important process factors. As a pulse laser was used in the present study, scanning speed over  $\sim 4 \text{ mm/s}$  was too high to continuously modify the polymer, resulting in dot-like modification (see Fig. 4a)). On the other hand, a scanning speed below 0.1 mm/s caused damage to the polymer. Similarly, the laser energy could also affect the resulting sample. A laser energy above 4000 nJ was too high to induce a change in refractive index, causing opaque damage (see Fig. 4b)). On the other hand, no changes were observed at laser energies below 100 nJ. These parameters are independent but their effects can be superposed upon each other. Therefore, the process conditions should be optimized, and the following standard conditions were used in the present study: scanning speed, 1 mm/s; laser energy, 500 nJ.

#### 3.2. Properties of DOEs

Fig. 5 shows microscopic images of irradiated PMP and PMMA plates. Periodic structures with a spacing of 10  $\mu$ m were formed inside the plates from these images. We estimated that the width of the grating line was approximately 1.5  $\mu$ m. As shown in Table 2, the *Q* values of all DOEs prepared were much greater than 1, and the *L* values of all samples were approximately 300  $\mu$ m. Therefore, all sample prepared in the present study were Bragg type gratings as mentioned above.

Fig. 6 shows  $\eta_1$  values of DOEs as a function of the density of the polymer material, where the Bragg angle  $\theta$  was 1.8°. The irradiation energy used for preparation was 500 nJ. As shown in Fig. 6,  $\eta_1$  in PMP was 45.1% and was about an order of magnitude greater than those of the other polymers ranging from 0.3% to 4.6%. Furthermore,  $\eta_1$  in PMP increased up to 65.1% at an irradiation energy of 700 nJ, whereas that in PMMA was only 2.1%, even with irradiation at 1000 nJ. As shown at the top of Fig. 6, PMP also showed the highest  $\Delta n$  value among the polymers examined, as calculated from the above equation. The densities of a number of optical polymers were approximately 1.1 g/ cm<sup>3</sup>, and the values for the polymers used in the present study also ranged from 1.0 to 1.2 g/cm<sup>3</sup> (Table 1). On the other hand, PMP had a density of 0.89 g/cm<sup>3</sup>, which is one of the lowest values among not only the polymers used in the present study but also all transparent polymers reported to date.

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