## Surface morphology of refractive-index waveguide gratings fabricated in polymer films<sup>\*</sup>

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The characteristic modifications are reported on the surface of polymeric waveguide film in the process of volume-grating fabrication. The light from a mode-locked 76 MHz femtosecond laser with pulse duration of 200 fs and wavelength of 800 nm is focused normal to the surface of the sample. The surface morphology modifications are ascribed to a fact that surface swelling occurs during the process. Periodic micro-structure is inscribed with increasing incident power. The laser-induced swelling threshold on the grating, which is higher than that of two-photon initiated photo-polymerization (TPIP) (8 mW), is verified to be about 20 mW. It is feasible to enhance the surface smoothness of integrated optics devices for further encapsulation. The variation of modulation depth is studied for different values of incident power and scan spacing. Ablation accompanied with surface swelling appears when the power is higher. By optimizing the laser carving parameters, highly efficient grating devices can be fabricated.

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In the last decade, two-photon initiated photo-polymerization (TPIP) process of polymer materials has gained increasing interest for the production of individually shaped structures<sup>[1-3]</sup>. One potential application of this technology is to create refractive-index grating structures by special scanning procedure<sup>[4,5]</sup> or holographic interference in polymer film<sup>[6,7]</sup>. The primary mechanism is to create a refractive-index change in the bulk materials by TPIP. In previous work<sup>[8-10]</sup>, we have fabricated refractive-index waveguide grating structures by TPIP in polymer films. Grating structures can be realized deep inside the volume of a polymer waveguide, and an incident laser beam is successfully coupled into the polymethyl methacrylate (PMMA) doped polymer film by coupling gratings<sup>[9]</sup>. Different from polymerization in bulk materials<sup>[11,12]</sup>, process of fabricating grating in waveguide film is more complicated due to the influence of the high intensity inside the laser focus on the surface of film. The interactions of high-peak-power femtosecond laser with organic materials usually lead to rapid and large morphological changes on material surface<sup>[13,14]</sup>. For planar waveguide film, it is essential to know the morphological changes with respect to laser power because of its effect on the performance of the integrated optical devices<sup>[15]</sup>. Especially for grating coupler<sup>[16]</sup>, the influence of the modulation depth of grating on the leakage parameter can not be ignored while the corrugation is deep enough.

In this paper, we report the influence of femtosecond laser pulses on surface morphology of the polymer film during the refractive-index grating fabrication process by means of atomic force microscope (AFM) and scanning electron microscope (SEM). Possible mechanisms responsible for the surface alteration are discussed.

Polymer film, which consists of 70% PMMA (with molecular weight of 120 000), 29% oligomer (Sartomer Product No.CN970E60) and 1% two-photon initiator<sup>[17]</sup>, was prepared by spin coating on quartz substrate and dried for 8 h at room temperature. Thickness of the polymer film is about 2 µm. The mean roughness measured by AFM is between 1.5 nm and 2.0 nm over a 10  $\mu$ m scan length. A mode-locked 76 MHz and ~200 fs pulses Ti:Sapphire laser operating at 800 nm was used as an excitation source. The detail is described in Ref.[9]. The sample was placed on a computer controlled x-ytranslation stage. The surface of sample was positioned perpendicular to the direction of the incident laser beam. Femtosecond laser pulses periodically scanned on the top of the thin film. The scan speed was set to be  $50 \,\mu\text{m/s}$ . All incident powers were measured at the back aperture of objective.

Photograph of the grating was performed on a phase contrast optical microscope (Olympus IX70) and polarized optical microscope (Olympus IX51). Atomic force

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micrographs were taken with instruments from digital instruments (Nano Scope IIIa and Dimension NS3A). SEM image was obtained with a JEOL JSM-6700F microscope (JEOL Ltd) at an acceleration voltage of 5.0 kV.

Under radiation of femtosecond laser pulses, the TPIP of oligomer in the polymer matrix was carried out upon the polymeric threshold of about 8 mW. The incident power was set to be 30 mW, and the grating with period of 2  $\mu$ m was fabricated in the film. The microscopic photo of obtained microstructure from phase contrast optical microscope is shown in Fig.1.



Fig.1 Optical microscopic photo of the volume grating fabricated by TPIP from a phase contrast optical microscope

We pay attention to laser induced morphological changes of the polymer film. Various sinusoidal gratings with different grating amplitudes are generated on the surface of polymer films by applying different laser powers. Fig.2 displays the results of the AFM cross-section analysis of the refractive-index grating. In Fig.2(a), the peak-to-peak modulation depth is about 7 nm at an incident power of 30 mW. The modulation depth is far less than that of ridge gratings<sup>[18]</sup>. An example of a typical surface relief grating at an incident power of 90 mW is shown in Fig.2(b). Compared with the first volume-grating sample produced by 30 mW, the laser-generated grating produced by 90 mW shows a sinusoidal grating surface with homogeneous periodicity. The quality of the groove edges of the surface gratings is high, and the peak-to-peak modulation depth is over 180 nm. It is pertinent to note that TPIP still occurs in this case.





Fig.2 AFM cross-section analysis results of the surface relief grating after being irradiated by laser with power of (a) 30 mW and (b) 90 mW

The femtosecond laser material processing mechanisms can be complex. It is generally accepted that surface swelling and material ablation of polymer occur during the process. The former can be attributed to a photothermal mechanism, and the latter is attributed to a photochemical ablation mechanism<sup>[19]</sup>. Surface swelling can raise the high intensity regions, while the ablation can reduce the thickness of the polymer. In some cases, surface swelling prior to material ablation is observed<sup>[20]</sup>. However, it is difficult to distinguish swelling from ablation from Fig.2, because there is no an initial reference point. The AFM image of this surface is shown in Fig.3. Further investigations show in Fig.3 that the surface of the polymer rises by approximately 180 nm, then dips by approximately 72 nm, rises by 71 nm, and dips by 92 nm again, as we go from the unwritten section to the written section of the same sample. The evidence indicated that the surface is transformed by the swelling of materials. The non-uniform surface relief can be attributed to the slight shake of the translation stage, which can affect the position of the focal spot at the beginning.



Fig.3 AFM micrograph of the film from the unwritten section to the written section of the sample

Obviously, the modulation depth is sensitive to the

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variation of the scan spacing. In the vertical direction, the position of focal spot is independently controlled by a translation stage. Therefore, the height of swelling region for each stripe is identical with the same scan speed and incident power. With the decrease of the scan spacing, the adjacent swelling regions partly overlap with each other, and the modulation depths of the surface are decreased gradually. The modulation depth depending on the overlapping degree is obviously a function of the scan spacing. In Fig.4, the maximum modulation depths of the film are shown against the incident power for two different spacing values of 2 µm and 3 µm, respectively. The depth falls as the spacing decreases from 3 µm to 2 µm for the same condition. Femtosecond pulses do not cause any surface changes at relatively low power. But it is difficult to pinpoint the threshold value for the onset of the morphological changes. After irradiation of femtosecond laser with 30 mW power, surface relief structures with less than 7 nm depth for both spacing values are observed. The morphological changes are very small, so we consider that the effect of the relief structure to the performance of the device can be negligible.



Fig.4 The surface modulation depth versus the incident power for spacing values of 2  $\mu m$  and 3  $\mu m$ 

Due to the Gaussian intensity distribution in the focal spot of beam, the process involves ablation and the resulting composite is partly carbonized while the incident power is high enough. Fig.5 shows the polarized optical microscopic photo of the microstructure irradiated with femtosecond laser at 198 mW. And an SEM images is



Fig.5 Optical microscopic photo of the microstructure from polarized optical microscope

demonstrated in Fig.6. The grating shown in Fig.5 is not totally transparent. The irradiated zones of the gratings are darkened, which indicates that the irradiated region is carbonized. Fig.6 illustrates that there are grooves appearing in the middle of each bulge of the resulting composite. Additionally, it is also obvious that the edges of the irradiated zones are bordered by strong debris formation, yielding a blurred grating.



Fig.6 SEM image of the microstructure

In conclusion, we investigate the morphological change of polymer film in the process of volume grating fabrication. Characteristic microstructure simultaneously appears on the surface of the polymer film with increasing incident power during the TPIP. Both three-dimensional profile and the cross-section of the relief gratings are revealed with AFM and SEM. Surface and volume gratings can be generated simultaneously in polymer film at a relatively high power by multi-photon absorption.

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