Heat treatment for reduction of surface roughness on holographic gratings

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Controlled postdevelopment heat treatment of the photoresist polymer used in the preparation of holographic gratings has been shown to enhance the diffraction efficiency of gratings and reduce the scattering losses. We prove this effect by analyzing the resonant reflection spectra of a waveguide grating and observing the reduction in the arc-shaped light scattering associated with the excitation of waveguide modes. © 2003 Optical Society of America

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Diffraction gratings are a key component in many applications in optics and optoelectronics. They are used as couplers and dispersing elements in integrated optics, spectroscopy, optical communications, and biomedical applications. Reducing the surface roughness is an important aspect of the fabrication process that ultimately enhances the grating's efficiency and performance. The inhomogeneous coating and the limited resolution of the photoresist are the major reasons that the surface roughness is superimposed on the grating's profile. The main goal of the experiment reported in this Letter is to achieve well-controlled heating of the photoresist polymer in which gratings are formed, so that the polymer partially melts and smooths the grating formation. When the polymer starts to melt, we expect that the Fourier spectral components of the roughness will vanish faster than the main Fourier spectral components that represent the grating period; this increases the fraction of the incident power diffracted by the grating.

An SPR505-A photoresist (ethyl lactate, n-butyl acetate, xylene) and a 352 developer (sodium hydroxide), both from Shipley, were used to fabricate the holographic gratings. The grating pattern was made by the interference of two deep-ultraviolet (DUV) beams on the photoresist film. Second-harmonic generation from an Ar ion laser with a 257-nm wavelength was spatially filtered, collimated, and spatially split into two DUV beams. The grating period was controlled by the interference angle of the DUV beams, which was controlled by a computer-controlled rotation stage with 10⁻³-degree resolution. The thickness of the photoresist was in the range 400–500 nm. The pre-exposure sample was soft baked for 90 s at 95 °C. The DUV exposure was in the range 90–120 s with 30 mW of power; the laser beam was expanded to a diameter of 2.5 cm after collimation. The gratings were formed in the photoresist film immediately after the exposed sample was developed for 15 s. Because the melting point of the photoresist must be identified before applying the postdevelopment heat treatment to reduce the surface roughness, the setup shown in Fig. 1 was used. The diffracted power and the temperature inside the heating cavity were measured and recorded. A He–Ne laser with a wavelength of 632.8 nm was used as a probing beam, the diffracted power was read by a photodetector with a lock-in amplifier for noise filtering, and the temperature was read by a thermocouple that was connected to a digital multimeter. The readings of both the temperature and the diffracted power were fed to a computer system. The grating period was designed to be 500 nm to ensure the appearance of the diffracted light when the grating was illuminated by a 632.8-nm-wavelength laser beam. Several runs were performed to study the behavior of the diffracted power when the temperature rose above the melting point. The polymer began to soften at 130 °C, and the melting temperature was ~135 °C. To provide controlled heat treatment, the melting process should be slow and start at the grating's surface rather than in the bulk of the polymer. To achieve this, we suspended the sample in the...
heating chamber so that hot air was the source of the heating. Suspending the sample also provided slower and better control of the heat flow from the main source (i.e., the hot plate), as shown in Fig. 1, where the transient time required for the heating chamber to reach thermal equilibrium was longer than that at the hot plate surface while the temperature was rising.

Figure 2(a) shows the heating process for a sample that was preserved at the melting point. The 20% drop in diffracted power is attributed to a reduction in the grating depth resulting from partial melting of the photoresist. A reduction in the grating depth should not be a problem as long as the surface roughness is reduced. The grating depth can be compensated from the beginning with adjustments to the thickness of the photoresist coating and the exposure dose.

The decrease in the diffracted power while the temperature rises to the melting point is attributed to the dependence of the polymer optical constants on the temperature. This was confirmed when the temperature started to decrease after partial melting took place, as shown in Fig. 2(b).

The waveguide grating was characterized before and after annealing by an analysis of the resonant reflection based on Wood anomalies.3 The roughness reduction was confirmed by two means. First, the absorption and outcoupling losses from the recorded resonant reflection while scanning around the resonance angle were estimated, as shown in Fig. 3. Second, while the guided mode was excited, the power reduction of the arc-shaped scattering associated with the surface roughness2,3 was observed, as shown in Fig. 4.

To estimate the losses, the following relations were used4,5:

\[
\text{FWHM}_\theta = \frac{\alpha_p + \alpha_r}{k \cos \theta},
\]

\[
R_{\text{max}} = \frac{\left| r_f \alpha_p - \alpha_r \right|^2}{\alpha_p + \alpha_r},
\]

where FWHM$_\theta$ is the full width at half-maximum (in radians); $\alpha_p$ is the absorption loss; $\alpha_r$ is the outcoupling loss, which increases with grating depth; $k = 2\pi/\lambda$, with $\lambda$ being the wavelength; $\theta$ is the angle of incidence; $r_f$ is the amplitude of the Fresnel reflection from the waveguide layer; and $R_{\text{max}}$ is the maximum reflection at resonance. Equations (1) and (2) can be solved for $\alpha_p$ and $\alpha_r$, yielding

\[
\alpha_p = \frac{k(\text{FWHM}_\theta) \cos \theta (1 + \sqrt{R_{\text{max}}})}{1 + r_f},
\]

\[
\alpha_r = \frac{k(\text{FWHM}_\theta) \cos \theta |r_f - \sqrt{R_{\text{max}}}|}{1 + r_f}.
\]

Table 1 summarizes the results.

The partial melting of the grating is associated with a drop in diffracted power of as much as 20%, as shown in Fig. 2(b). The outcoupling loss $\alpha_r$ is expected to decrease, but Table 1 indicates an increase in $\alpha_r$ of as much as 11%. The only explanation for this contradiction is that the surface roughness has been reduced. Reducing the surface roughness decreases the scattered power at the moment of coupling and outcoupling from the waveguide. This result was confirmed.
Table 1. Summary of Wood Anomalies Analysis

<table>
<thead>
<tr>
<th></th>
<th>FWHM</th>
<th>$R_{max}$</th>
<th>$\alpha_r$</th>
<th>$\alpha_p$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Without heating</td>
<td>0.46</td>
<td>0.132</td>
<td>85.6</td>
<td>7.0</td>
</tr>
<tr>
<td>With heating</td>
<td>0.26</td>
<td>0.218</td>
<td>51.9</td>
<td>7.8</td>
</tr>
</tbody>
</table>

by the scattered arc-shaped power that appears at resonance: less scattering after heat treatment was observed, as shown in Fig. 4.

To understand this relationship between the scattered power and the surface roughness, we considered the surface roughness to be a random collection of gratings with different periods, orientations, and depths, which can be modeled mathematically by a Fourier transformation of the surface topography. The arc-shaped scattering results from those Fourier components of the surface roughness that outcouple the confined energy from the waveguide and satisfy the waveguide excitation condition

$$n^* = m \frac{\lambda}{\Lambda} \pm \sin \theta,$$

(5)

where $n^*$ is the waveguide effective refractive index, $\lambda$ is the wavelength of the laser beam used for mode excitation, $\Lambda$ is the period of the Fourier component of the roughness, $\theta$ is the outcoupling angle, and $m$ is the order of diffraction.

The total power in the arcs increases with the rms value of the surface roughness, since it represents a deeper random collection of gratings. Thus the decrease in the arc-shaped scattered power is associated with a decrease in the surface roughness.

We have demonstrated that a well-controlled heat treatment of a holographic grating improves the grating efficiency by decreasing the surface roughness. This method, we believe, can be useful for holographic gratings and their many applications, which are in many cases limited by the grating quality.

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References