Single-step fabrication of continuous surface relief micro-optical elements in hybrid sol-gel glass by laser direct writing

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Abstract: A negative tone hybrid sol-gel material was applied to the fabrication of continuous surface relief micro-optical elements by laser direct writing lithography. The hybrid sol-gel glass was synthesized as UV photosensitive material and used for the fabrication of micro-optical elements with continuous surface relief profile. The surface profile was directly controlled by the UV exposure dosage and the exposed areas were crosslinked and converted into sol-gel glass with relief structures. The UV exposure dosage was realized by an acousto-optic modulator (AOM) in the laser direct writing system. Characterization results indicated that the thickness of the sol-gel glass has a linear response to the AOM values ranging from 3.25 to 5.5. The sol-gel thin film was measured to have a refractive index of 1.52 and the maximum thickness of 2 µm. For various designs of diffractive optical elements within the visible wavelength, the sol-gel film enables us to achieve an arbitrary phase change between 0 and 2π with the linear AOM exposure range. As an example, a blazed grating with a period of 45 µm and height of 1.17 µm was fabricated in the sol-gel glass using direct laser writing method.

OCIS Codes: (050.1970) Diffractive Optics, (220.4000) Microstructure Fabrication

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1. Introduction

Micro-optical elements have wide applications in many areas such as acousto-optics, integrated optics, spectroscopy, optical interconnects, binary optics and quantum electronics. Continuous 3D surface relief diffractive components are believed to offer better optical quality and higher diffraction efficiency than binary phase-only diffractive optical elements (DOEs). With the advent of advanced microelectronics planar fabrication technologies and new methods for material synthesis, the hybrid sol-gel material has shown a great potential for the fabrication of complex micro-optical components such as phase holograms, kinoform lenses and blazed gratings in a single developing step without etching. In conventional planar lithographic technologies, it is known that the processes involve two main steps, i.e. patterning and developing the micro-optical structure on the photoresist and then etching the structures into the substrate. However, for fabrication of 3D continuous surface relief structures, it is difficult to control the surface profile in the etching process. To achieve a low cost and simple fabrication process, we explore a hybrid sol-gel material that enables the fabrication of surface relief micro-optical components in a single step using laser direct writing.

It is known that the sol-gel material has considerable advantages for the fabrication of micro-optical components due to its good optical properties, cost effectiveness, and process simplicity. Since the physical and chemical properties of the hybrid sol-gel material can be manipulated at the desired molecular level, it provides great flexibility for the synthesis of the material to meet specific requirements, for example, to improve the mechanical properties and to tailor the refractive index. The sol-gel process also offers a low processing temperature route for material synthesis.

Various hybrid sol-gel materials and fabricating methods have been reported in the past decade. The authors of Ref [1-2] presented the silicon zirconium (SiO_2/ZrO_2) material based on the sol-gel method, which was characterized and applied to the fabrication of gratings using contact imprinting and interference techniques, respectively. We have previously reported on the fabrication of surface relief gratings [3] with a period ranging from 0.925 μm to 4.45 μm using the SiO_2/TiO_2 sol-gel glass together with the UV holographic interference technique. The sol-gel material was also used for the fabrication of diffractive microlens using the high-energy beam-sensitive (HEBS) glass gray scale mask [4]. Rantala *et al* demonstrated the fabrication of surface relief diffractive grating in the SiO_2/ZrO_2 sol-gel glass using direct variable-dose electron-beam lithograph [5]. The techniques employed for the sol-gel synthesis of the above SiO_2/ZrO_2 and SiO_2/TiO_2 materials were investigated for the fabrication process with a single developing step. That is, after the UV or e-beam patterning, the unexposed areas were washed away in the development process. A self-processing technique reported by Blanc and Pelissier in Ref [6], where an acrylate-modified silica-titania (SiO_2/TiO_2) sol-gel glass was used to fabricate sub-micron period gratings through a phase mask with an excimer laser at 193 nm. Since the material is self-processing, no further developing and etching steps are required to form a surface relief micro-optical element.

It is known that the HEBS gray scale mask is a promising technology for the fabrication of 3D continuous structures, however due to the nature of using the mask aligner and the cost of the gray scale mask, the HEBS technique is more suitable for high-volume production. For fabrication of prototype structures in the laboratories, UV laser direct writing technology is a contender for fabrication of 3D micro-optical elements with feature size at micron scale. Comparing with the electron beam lithography, which is able to provide fabrication with sub-micron feature sizes, the laser direct writing technique is a cost effective and simple approach. Neumann *et al* fabricated the surface relief structure in dry, self-developing photopolymer films by using the direct laser writing [7].

In this paper, we report on the fabrication and characterization of a blazed grating built in hybrid sol-gel glass by using the laser direct writing technique. The sol-gel glass used in the experiment was an organically modified SiO_2/TiO_2 hybrid glass synthesized at room temperature. The laser source employed in the laser writing system was a He-Cd laser with a wavelength of 325 nm.

2. Fabrication and Characterization

The hybrid sol-gel glass was synthesized from two solutions. Solution I was a silicon oxide network, which was formed by the hydrolysis of prolylmethacrylate-substituted trimethoxysilane and 3-(trimethoxysilyl) propyl methacrylate in isopropanol and acidified water. In this case, the volume ratio is 30 ml: 12 ml: 1 ml. Solution II was a titanium oxide network, which was formed by adding titanium propoxide ($Ti(OCH)_4$) into acetylacetone at a molar ratio of 1:4 in nitrogen environment; and the solution was agitated until homogenization was reached. The two solutions were then mixed with a molar ratio of 4:1 (SiO_2:TiO_2). It should be mentioned that titanium was used here to modify the refractive index of silicon oxide network. The final mixture solution was allowed to age at room temperature for 30 hours with vigorous stirring. This negative-tone silicon titanium material was made UV photosensitive possible by adding a photoinitiator IRGACURE 184 (CIBA) with 4% wt to the sol gel. Large particles in the mixed solution were removed by a 0.1 μm membrane filter attached to a syringe before spin coating on a pre-cleaned BK7 microscope glass slide. A thin film layer of the sol-gel film was spun onto a glass substrate at 1200 rpm/min for 60 secs and a film thickness of more than 2.0 μm could be easily achieved. Before the UV exposure, the sample was pre-baked on a hotplate at 90 ^{o}C for 5 mins to remove the excessive solvent and improve the adhesion of sol-gel film to glass slides. After the exposure, the sample was developed in ethanol for 30 secs to remove the unexposed component and the patterns written by laser writing were clearly observed. Finally, the sample was post-baked on a hotplate at 160 ^{o}C for 30 mins for further solidification. It should be noted that the laser power used in the experiment was a 200 mW He-Cd laser with a wavelength of 325 nm. In the laser direct writing system, an acousto-optic modulator (AOM) was used as a beam blanker to control the 1st diffraction order by making the AOM "on" and "off" for deflection of the beam from the aperture stop. The laser beam was focused onto the sol-gel samples through an optical microscope. The laser writing system has a lateral resolution about 3 μm. Figure 1 shows a schematic diagram of the laser direct writing setup.

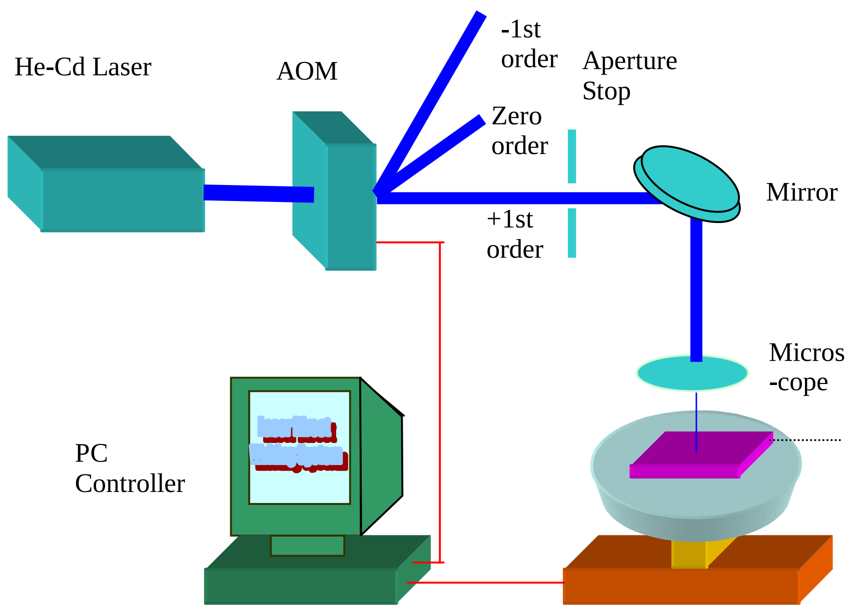

Figure 1. Schematic of the laser direct writing system.

When the hybrid sol-gel thin film was exposed under the UV laser beam, free radicals formed by photoinitiator lead to free-radical cross-linking polymerisation of the unsaturated carbon bonds, namely, the response of the hybrid sol-gel glass to UV exposure is the same as a negative tone photoresist. In the development, the exposed areas in the sol gel were crosslinked and remained on the substrate and the unexposed areas were washed away. The refractive index and thickness of the hybrid sol-gel glass were characterized using a prism coupler (Metricon Corporation). The refractive index and thickness of the hybrid sol-gel glass were measured to be 1.52 and 2.0 μm, respectively.

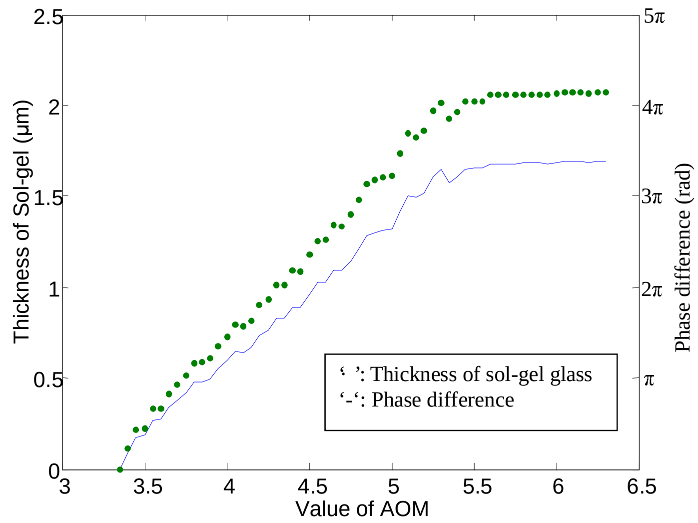

Figure 2. '.' Sol-gel film thickness as a function of AOM values. '-' phase difference caused by the sol-gel film thickness as a function of AOM value for a refractive index of n = 1.52 , and laser wavelength of λ = 0.6328 μm.

The relationship between the polymerized sol-gel film thickness and the AOM value is shown in Figure 2. It can be seen that the thickness of the hybrid sol-gel glass is a function of the AOM values, which is ultimately determined by the laser beam intensities of the UV exposure. Experimental calibrations indicated that the AOM is a nonlinear function of the intensities based on a measurement of the output laser beam intensity modulated by an AOM with a power-meter. The AOM values are governed by

$$AOM = -0.08146 \cdot ln(1.262510 - 16I) + 2.00119 \cdot (I - 0.5)^{0.3189} \qquad (1)$$

where I is the laser intensity in μW.

It is seen in the figure that the thickness of the sol-gel glass has a dynamic linear response with the AOM values of between 3.25 and 5.5. This linear characteristics can be used for the fabrication of DOEs with a particular height.

It is noted that in many designs and fabrications of DOEs, the DOEs are only considered to generate a phase change in the range between 0 and 2π. For example, we can calculate the phase difference caused by the thickness of the hybrid sol-gel glass and the laser wavelength of 0.6328 μm. The following expression is used for this calculation,

$$\Delta\Phi = \frac{2\pi(n-1)h}{\lambda} \qquad (2)$$

where the refractive index is n = 1.52, λ = 0.6328 μm and h is the thickness of hybrid sol-gel glass. It is seen in Figure 2 that the thickness of the sol gel and its linear response to the AOM values between 3.25 and 5.5 will enable us to realize various DOE designs with various height.

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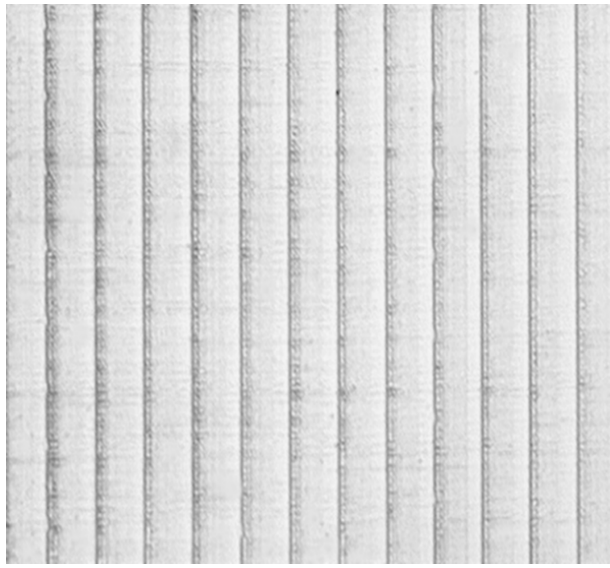

Figure 3. Photograph of the blazed grating.

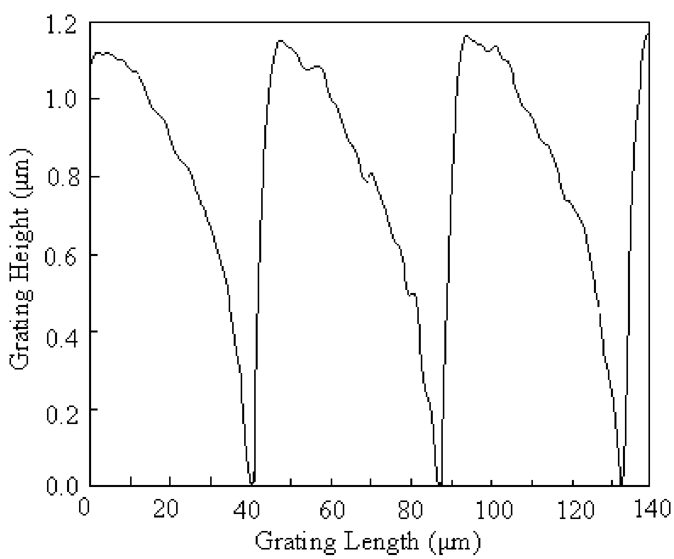

Figure 4. Surface profile of the blazed grating.

With the calibration results, we have designed and fabricated a blazed grating by using laser direct writing lithograph technique. Figure 3 shows a photograph of a blazed grating fabricated using the sol-gel glass. The thickness of the grating was measured by a Dektak surface profiler (Veeco Metrology). Figure 4 shows the surface profile of the blazed grating. The blazed grating has a period of 45 μm and a maximum height of 1.17 μm, which is 0.05 μm smaller than the expected height of 1.22 μm. This discrepancy could be due to the shrinkage of the hybrid sol-gel glass during the post-baking process. In addition, the humps on the grating surface are due to the coarse steps of the AOM values in the fabrication. For better

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surface quality elements, both fine AOM steps and optical proximity effect should be taken into account.

Comparing with the laser direct writing technique, the electron beam lithography (EBL) is able to achieve fabrication of feature sizes as small as 100 nm. Therefore for optical elements with sub-micron sized structures, for example the sub-wavelength sized DOEs, the EBL is the workhorse in the laboratories. However, since the e-beam scanning area is significantly limited by the e-beam deflection angle, the writing field of the EBL is restricted to a small area without involving the filed stitching technology. Consequently, the laser writing method offers an alternative lithographic process with lateral resolution in a few microns. It is a cheaper and faster technique compared to the EBL.

3. Conclusion

We have demonstrated the fabrication of continuous relief structures in hybrid sol-gel glass by the laser direct writing technique. To realize a diffractive optical element with multilevel phase structures for the entire visible wavelength range of 400 – 780 nm, the sol-gel glass with a refractive index of 1.52 is able to produce the phase difference between 0 and 2π. This property will enable us to fabricate complex micro-optical components such as gray scale phase hologram and kinoform lens using the sol-gel method. Comparing with the gray-scale mask technique for the fabrication of 3D structures, we believe that the laser direct writing technique provides a cost-effective and flexible alternative for fabrication of prototype components in the laboratory.

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