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Diffraction gratings in sol–gel films by direct contact printing using a UV-mercury lamp

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Abstract

We report on the fabrication of diffraction gratings in photosensitive sol–gel thin films by direct contact printing using a UV-mercury lamp. Titanium amplitude masks were used to replicate diffraction gratings into photosensitive sol–gel films by contact printing with an incoherent UV-light source. Gratings with 1-μm period were fabricated in sol–gel films. The diffraction efficiencies of each diffracted order were measured and compared to theoretical values. The demonstrated process of contact printing using a regular UV-light source in the optically compatible sol–gel material has potential for large-scale fabrication of submicrometer gratings at very low cost. © 1999 Elsevier Science B.V. All rights reserved.

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1. Introduction

The diffraction grating is an important component for integrated optics technology. It is often used as a fully integrated waveguide coupler or as a Bragg reflector that can be designed for a specific wavelength [1]. Applications of planar waveguide technology into areas such as chemical and biochemical sensing devices will demand processes that can cost effectively mass-produce diffraction gratings and that are fully compatible with the integrated optics platform. The holographic two-beam interference process [1] and the phase mask technique [2] using coherent laser sources and photolithography technology have been widely used for this purpose. Both these methods have also been used to write gratings in sol–gel thin films [3,4], direct electron beam writing has been demonstrated, as well [5].

Sol–gel materials are attractive for integrated optic devices: They are compatible with planar integrated optics technology, they are chemically and mechanically robust, and their optical quality is high and widely tunable [6]. In addition they can be made highly UV-sensitive for microstructure patterning. Indeed, UV-sensitivity has been employed to photo-imprint waveguides and gratings into sol–gel thin films. Submicrometer sol–gel grating fabrication with both the holographic [3] and the phase mask technique [4] has recently been demonstrated. Both these methods have their advantages: the holographic technique is highly flexible and the same setup may be used for different gratings; the phase mask technique is well established and it is widely used to fabricate gratings in optical fibers. On the other hand, there are also disadvantages [7]: the holographic technique requires careful angular alignment and high temporal and spatial stability; the phase mask technique requires high accuracy not only in the grating period but also in the depths of the grating grooves to minimize the unwanted zeroth diffraction order. In addition, both these methods require a coherent monochromatic laser source. Thus there still is a need for a simple and inexpensive sol–gel grating fabrication method for low-cost large-scale manufacturing. This combined with the otherwise simple and versatile sol–gel processing would

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facilitate highly inexpensive manufacturing of a variety of integrated optics devices utilizing gratings.

In this paper, we demonstrate a simple approach to produce photolitographically diffraction gratings in sol–gel thin films. We describe the fabrication of diffraction grating by a standard contact photolithography process in sol–gel films using a conventional incoherent UV-mercury lamp. The process is fully compatible with standard lithography technology and has potential for low-cost large-scale manufacturing of sol–gel integrated optics devices employing submicrometer gratings.

2. Sol–gel glass material

The photosensitive hybrid glass film used here is an organically modified silicate material based on inorganic–organic sol–gel synthesis [6]. Two criteria have to be fulfilled in the design of the glass material. First, the contrast {defined here as $\gamma = [\log_{10}(D^t/D^s)]^{-1}$, where D^t is the dose required for complete crosslinking and D^s is the dose at which the cross linking starts to happen [8]} must be adequate to ensure high differential solubility between the exposed and unexposed regions of the hybrid glass thin film in order to achieve submicrometer feature size. Second, the sensitivity of the material must be sufficiently high to minimize the amount of scattered light beneath the masked regions. Moreover, the sensitivity has to be high enough to meet throughput requirements in mass production [8]. For hybrid sol–gel materials the contrast and the sensitivity are mainly dependent on the molecular weight distribution and the amount of the photo-initiator, respectively.

The synthesis of the photosensitive hybrid glass material is based on the uniform hydrolysis of the trifunctional alkoxysilanes by homogeneous generation of water [9] with acid catalyst. The alkoxysilane is copolymerized with another inorganic network modifier, zirconium alkoxide, in order to increase the refractive index and the mechanical strength. The synthesis of the organically modified trifunctional alkoxysilane (3-methacryloxypropyl trimethoxysilane) is carried out in isopropanol solution in a ratio of 1:3 (molar) with 0.3 M hydrochloric acid. In order to facilitate a hydrolysis reaction the solution is refluxed at 82°C for 4 h so that the system is able to generate a substantial amount of water in solution of varying protonic activity. After the reflux the solution is let to cool and then filtered. Next, the zirconium alkoxide (tetrapropyl zirconate) is synthesized in isopropanol solution with methacrylate acid with a ratio of 1:2:2 (molar), respectively, at room temperature for 1 h. Next this solution is incorporated into the hydrolyzed silane solution. The hydrolyzed silane and zirconium alkoxide solutions are mixed together so that the alkoxide ratio is 4:1 (molar). The copolycondensation of the mixed solution is allowed to occur at 30°C for 12 h in order to obtain a homogeneous, multicomponent, and hybrid sol–gel glass material. Finally, the material is made photosensitive by adding 3% (weight) of photoinitiator into the hybrid sol–gel solution. The photoinitiator is a mixture of two free radical forming compounds: 1-hydroxycyclohexyl phenyl ketone and bis(2,4,6-trimethylbenzoyl)-phenylphosphineoxide that are mixed in a ratio of 1:1 (molar).

3. Grating fabrication

An amplitude mask is required for contact printing with the UV-photolithography. Grating masks with 0.5-μm-feature sizes and free from errors are expensive and available only from a few vendors. Therefore, we chose to fabricate the amplitude masks used in this demonstration ourselves. These masks were fabricated on a glass substrate coated with a 200-nm layer of titanium by sputtering deposition. Next photoresist spin-coating, two-beam interference, and wet chemical etching processes were employed as described in Ref. [10]. The period of the amplitude mask was measured to be 1006 nm. An atomic force microscope (AFM) picture of the mask is shown in Fig. 1a. It is seen that the titanium surface is somewhat rough, probably due to chemical etching effects. The aspect ratio (i.e., the ratio

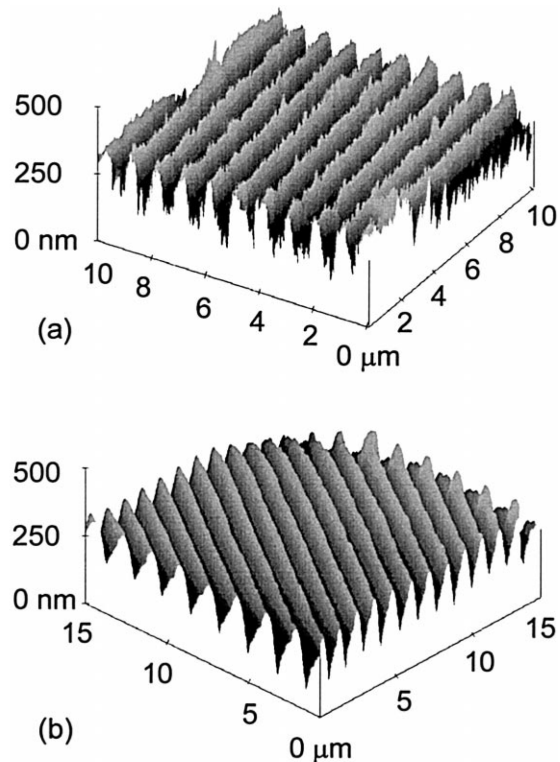

Fig. 1. Atomic force microscope picture of (a) the titanium amplitude mask and (b) the imprinted sol–gel diffraction grating.

Table 1
Measured and modeled diffraction efficiencies

Diffraction order	Measured diffraction efficiency [%]	Modeled diffraction efficiency [%]
T_{-2}	0.34	0.154
T_{-1}	5.3	5.11
T_0	79	85.3
T_{+1}	5.4	6.61
R_{-2}	0.46	0.812
R_{-1}	0.18	1.36
R_0	6.1	0.237
R_{+1}	2.4	0.41
Σ	99.2	99.99

T and R are for transmitted and reflected diffraction order, respectively. The modeling was performed for a grating structure of a triangular shape with 260 nm height and a period of $\Lambda = 984$ nm. The modeling was done for p-polarization using $\lambda = 632.8$ nm.

between the opening and the period) of the grating on the mask was estimated from the AFM picture to be about 0.55 instead of the desired 0.50.

The diffraction gratings in the sol–gel film were fabricated by spin-coating a thin layer of the gel onto a borosilicate glass substrate and by prebaking the film at 90°C for 2 min. Using the amplitude mask in direct contact with the sol–gel thin film, the UV-photolithography process was applied. The sol–gel films were exposed through the titanium mask with a UV-dose of 150 mJ/cm^2 (with peak emissions at 365 and 405 nm wavelengths). As exposed with UV-light the sol–gel material acts as a negative photoresist, i.e., the parts that are exposed remain as the sol–gel is developed in acetone. After the development process the samples were postbaked at 160°C for 1 h in order to densify the material. The measured refractive index of the sol–gel film was $n = 1.52$.

An AFM picture of a typical sol–gel grating is shown in Fig. 1b. The height of the grating structure was estimated from the AFM pictures to be ca. 200 nm and the period of the grating was measured to be 984 nm. The measured period of the sol–gel grating is 2.2% smaller than that of the amplitude mask. This is likely due to the shrinking of the sol–gel material during the postbaking step. The grating was characterized by measuring the diffraction efficiency of each of its orders. The measurement was performed at the first-order-Littrow mounting [10] with a p-polarized helium–neon laser ($\lambda = 632.8$ nm). The diffraction efficiency is defined as the ratio between the power into the diffraction order in question and the incident power onto the sample. The results are given in Table 1. The measured values are also compared with calculated ones. The calculation is based on a coordinate transformation method for diffraction gratings [11]. The model assumes a grating with a triangular profile and uses the structure height as a variable. The best fitting for this variable was calculated for each order individually using a merit-function procedure, then the highest and lowest values were discarded. The average of the remaining values was calculated to be 260 nm. The differences between the measured and modeled diffraction efficiencies are presumed to be mainly due to the triangular approximation in the modeling.

4. Conclusions

The fabrication of gratings with micrometer periodicity in sol–gel thin films by direct UV contact printing using a standard photolithographic mask-aligner with a UV-mercury lamp was demonstrated. The method is extremely simple and has potential to be used for fabrication of gratings in large number at low cost. Here, we employed homemade master masks to demonstrate the approach. The use of high-quality master masks made by electron-beam writing will certainly improve the quality of the replicated sol–gel gratings. Amplitude masks with feature sizes below 0.25 μm are currently commercially available. We believe that the demonstrated technique has potential for fabrication of high-quality glass waveguide Bragg gratings, requiring ~0.5-μm periodicities, for telecommunications applications.

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