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High-resolution periodical structure on polycarbonate using holographic interferometry and electroforming process

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A procedure for prototyping a periodic structure on polycarbonate at submicron order using holographic interferometry and electroplating metal molding processes is demonstrated. The holographic interference using a He-Cd (325nm) laser was first used to create periodic line structure on an i-line sub-micron positive photoresist film. Pattern was then transferred to a metal mold using Nickel-Cobalt electroforming. Final line pattern on a polycarbonate thin film was formed from the metal mold using a hot press machine. The technique allows for accurate control for the transfer of a grating period and depth. The grating pattern on the polycarbonate polymer produced by the metal mold gives an average error of less than 1.7% in the grating period and an average error of 5.7% for depth reproduction.

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1. Introduction

Gratings are widely used in the integrated photonic devices for purposes such as spectral filtering, multiplexing, routing, fan-out, focusing, collimating, etc. [1-6]. Among all the material used, polymer gratings are of great interest for their low cost and easy fabrication. Recently, demands of low cost and high-resolution lithographic technique have attracted a considerable interest in the pursuit of new ways to create a more efficient grating structure. Typical techniques for patterning gratings on polymers include holographic surface relief grating [7-9], electro-beam (e-beam) lithography [10], laser-beam direct writing [11], X-ray mask technology [12], and phase mask lithography [13-14]. Although these tools provide high precision in manufacturing, it often limited by its size of the device which it can actually be manufactured. These mentioned tools are also hard to get and relatively expensive. On the other hand, the unique capacity of holographic lithography for high-resolution and large-area pattern transfer offers significant advantages over the other techniques. The holographic interferometrical technique provides flexibility in the grating period and depth by changing the incident beam angles and the irradiation time. In addition, the theoretical limit of the frequency of the interference pattern produced by two intersecting beams is half of the wavelength of the incident beam. Thus, the grating period is only limited by the wavelength of the light source.

The advanced technology of LIGA-like process eliminates the problem of tool wear and material machine-ability, and can achieve sub-micro accuracy. There are many replication processes that are simple and involve fairly easy fabrication; such as hot-embossing [15], UV-embossing [16], and micro-transfer molding method [17,18]. If these molding processes are combined with a LIGA-like process that can produce a mold for the subsequent molding processes, it may have great potential for mass production [19]. Converting the plastic mold into the metallic mold is the basic concept of a LiGA-like process. There are two issues of electroforming to make micro-molds with enough thickness (at least 2 to 3 μm) for practical molding process; one is the deformation of electroforming molds after a length time, the other is the low growth rate in electroforming itself. Even thought some additives are used to control the deformation induced by internal stress in electroforming process, real time monitoring the use of this additives is no sophisticated, the deformation of micro-molds after electroforming is still unpredictable [20]. In here, we have used two steps to improve the problems. First step is to control the electroforming thickness under 300 μm with less internal stress and no deformation. The second step is to apply a mold plate enough thick for supporting molding pressure.

By using the holographic interferometry, electroforming and stamping process, this work demonstrates a new fabricating technique of period structure on polycarbonate thin film. Based on the preliminary experimental results, it is found that it is possible to replicate grating period and depth with high precision. The metal mold also has the ability to show more reliable and reproducible results.

2. Methodology and results

The technique of forming grating patterns on the polycarbonate thin film involved three processing steps: First, a grating pattern is holographically exposed using two-beam interference pattern on a positive photoresist film. This produces a master that can be subsequently transferred to a Nickel-Cobalt (Ni-Co) metal mold. The metal mold then is used to transfer the final gratings pattern onto a polycarbonate using a hot press process.

2.1 Holographic grating

The master grating patterns on a positive photoresist (Ultra123, Microchem. Corp. MA) were holographically exposed using a two-beam interferometer technique [21]. Based on the results, we found that the grating period and the corresponding depth of the grating pattern can be accurately controlled down to an error rate of less than 1%. We also found that a high aspect ratio of almost 1:1 between the depth and the period of the grating structure could be obtained using this process. The profiles of the grating were measured by atomic force microscope (AFM) and shown in Fig. 1.

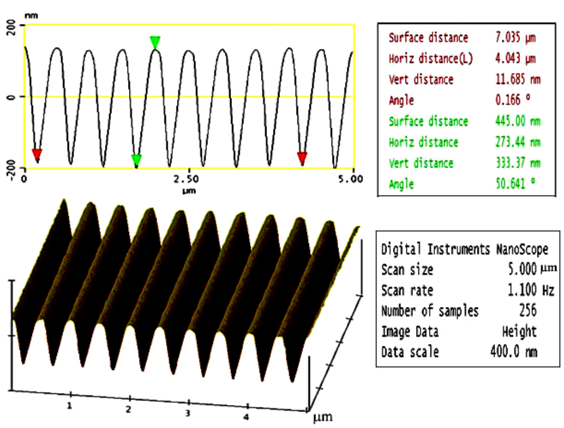


Fig. 1. The AFM picture and measurement result for the grating on Ultra123 positive photoresist (505 nm grating period and 333 nm grating depth).

2.2 Molding process

Electroplating process using nickel-cobalt alloy was applied to create the metal mold. To increase the adhesion, a 200 nm nickel thin film first sputtered onto the positive photoresist mold to serve as a seed layer for the subsequent electroplating of Ni-Co alloy. The sample is then immersed into a tank filled with solution of nickel and cobalt salt diluted in boric acid. Bars of nickel and cobalt hang in the tank served as anodes to keep the solution in balance. An electrical current passed through the solution, caused atoms from each element to form a metal on the being plated. For our setting, Boric acid: 30 – 40 g/l. and Co concentration between 1 to 20 g/l. and 70 g/l were used. The pH level was kept around 4.0 and temperature was maintained near 55 ^{o}C, and the solution was agitated by a Magnetic stirrer (see Table 1).

Table 1. Ni-Co alloy electrolyte composition.

Ni concentration	70 g/L
Co concentration	1~20% (w/o) in sol.
Boric acid	30~40 g/L
Current density	1~10 ASD (Adm^{-2})
PH	4.0 ± 0.5
Temperature	55 ± 1ºC
Agitation	Magnetic stirrer

In order to improve the thickness uniformity of electroplating mold, which is usually in concave shape, a secondary cathode was applied. The secondary cathode was placed at a specific distance (less than 2.5 mm) from the primary cathode during the electroplating process. Two power supplies are used, one is for the primary cathode, and the other one is for the secondary electrode. Both cathodes share with the same anodes, and the applied current density is the same for both cathodes. The purpose of the second cathode was to reduce the local ion concentration of the double layer surface plating area. This process hindered the grow rate on the edges of the deposited mold. When high deposition rate occurs on the edges of the plating field, the secondary cathode becomes a frame shaped like the plating area, which adapts to locally reduce the deposition rate on the primary cathode.

After 15 hours of continuous deposition at a deposition rate of 15.6 μm/hour, a 234 μm thick layer of Ni-Co metal is deposited above the surface of the positive resist mold. Basically, the mold was formed, but it still not thick enough for subsequent molding process. A thick nickel plate was clamped onto the metal mold surface for continuous electroplating. After 30 hours, the thick plate bonded onto the Nickel mold and formed into a piece. This enables a 5 mm thick metal mold to be formed. The positive photoresist was then stripped away using acetone. The metallization process is depicted in Fig. 2. Picture taken from the scanning electron microscope indicated that the grating pattern structure of the metal mold was well transferred (see Fig. 3). Based on the measurement taken from atomic force microscope (AFM) and SEM, the gratings on the metal mold transferred from a master grating were slightly off from the original dimension. For example, the grating period was off by 2 nm and the depth was reduced by 26 nm for a profile of 505 nm period and 333 nm depth, while the grating period was off by 2 nm and the depth was reduced by 34 nm for a profile of 703 nm period and 399 nm depth.

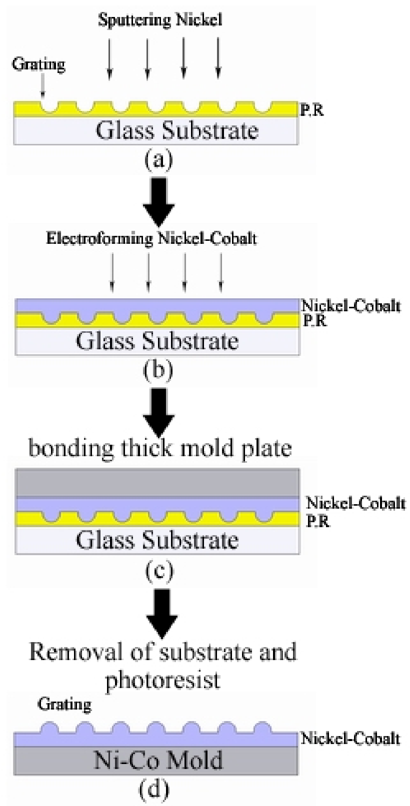


Fig. 2. Electroplating process for Ni-Co grating mold.

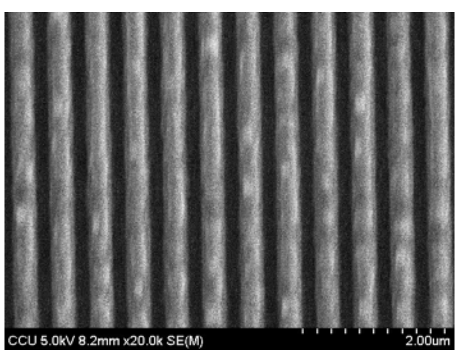
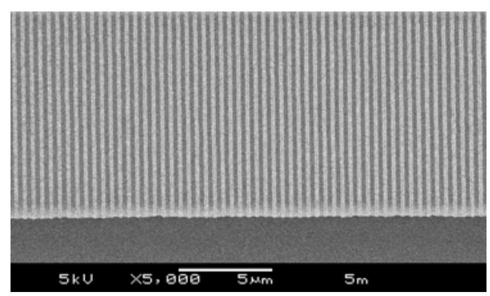
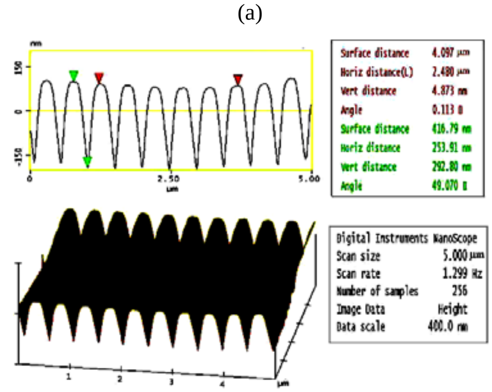


Fig. 3. The SEM picture of gratings on Ni-Co mold (503 nm grating period and 307 nm grating depth).

The patterned metal mold was used as a master mold to transfer the grating pattern onto a polycarbonate (PC) material thin film (supplied by Hsintou Company, Taiwan) using typical thermal pressing technique. The PC is a suitable material for optical devices due to its good physical and optical properties. A process similar to injection molding was used. Metal mold was placed on a stainless substrate in a hot pressing machine. Two processing parameters including working temperature and pressure were investigated. One test we conducted was adjusting pressure in the fabrication process between 5 to 20 kg/cm ^{2} with a holding time of 180 seconds. However, no significant difference was found in the gratings formation at different pressures. However, the temperature appeared to be critical in the process. Our result indicated that the grating pattern could not be formed on the polymer when the temperature was less than 130 ^oC. When the temperature was over 145 ^oC, tiny bubbles started to develop in the samples. The out-gassing caused surface damage on the sample. Therefore, the idea operating temperature was found to be between 135 ^oC to 140 ^oC. The thickness of the PC sample was about 2 mm. The profiles of the gratings on PC were measured using an AFM. Based on the measurement, when the depths of the gratings were less than 370 nm, grating patterns transferred quite accurately from the metal mold. Comparing the two SEM micrographs of PC and Nickel metal mold (grating period of 494 nm) indicated that the patterns were successful (Fig. 4(a) and (b)). The AFM micrograph result shows that the gratings on a PC substrate transferred from a metal grating, a profile of 503 nm period and 307 nm depth, are slightly off from the metal mold dimension (Fig. 4(b)). The grating period was off by 9 nm while the depth was reduced by 15 nm. Based on the results from three different metal molds, which are 701 nm, 610 nm, and 503 nm periods with 365 nm, 343 nm, and 307 nm depth respectively, the reduction shows an average of 1.7% or 10 nm average reductions in the periods. The depths on the other hand were reduced by as much as 5.7% or 20 nm on average.



(a)



(b)

Fig. 4. The SEM and AFM micrographs of gratings on PC polymer (a) SEM (b) AFM. (494 nm grating period and 292 nm grating depth).

In order to investigate the performance of the diffraction gratings, the first order diffraction efficiencies, η, of the gratings with period of 494 nm and depth 292 nm printed on the PC polymer were measured by the He-Cd laser at a wavelength of 325 nm. The first order diffraction efficiency of 18.51% was attained during the irradiation.

3. Conclusion

In conclusion, we have successfully created a process for rapid production of submicron range gratings by using both electroplating-molding and holographic interference techniques. A large aspect ratio on the grating pattern could be obtained, and reproduction of the grating on a polycarbonate polymer could be achieved. The grating period and depth on the polymer gratings exhibit small difference from the original designed grating pattern. This process shows great potential for mass production of any period of grating structure.

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