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# Fabrication of optical gratings by shrinkage of a rubber material

Teng-Kai Shih <sup>a,\*</sup>, Jeng-Rong Ho <sup>b</sup>, Hung-Yu Liao <sup>b</sup>, Chia-Fu Chen <sup>a,c</sup>, Chueh-Yang Liu <sup>a</sup>

Department of Materials Science and Engineering, National Chiao Tung University, Hsinchu, 300, Taiwan, ROC
Graduate Institute of Opto-mechatronic Engineering, National Chung Cheng University, Chia-Yi, 621, Taiwan, ROC
Institute of Material and system Engineering, MingDao University, ChangHua, 523, Taiwan, ROC

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#### Abstract

Ordered wavy surface structures generated by deposition of a metal thin film on a pre-stretched PDMS plate were fabricated and its potential application for optical gratings was proposed. The orientation of the generated structures was always perpendicular to the pre-stretched direction and the pitch of the structure could be adjusted ranging from  $4.5~\mu m$  to  $6.8~\mu m$  by controlling the strength of the pre-stretched strain and the thickness of the surface metal film. Based on these periodic structures, various optical gratings were demonstrated. With a slight modification of the fabrication scheme, gratings with different orientations can be fabricated on both sides of the PDMS plate, the double-sided gratings, could be fabricated. It is believed the current method has the potential for the fabrication of a large-scale diffractive grating at lower costs. © 2007 Elsevier B.V. All rights reserved.

Keywords: PDMS; Metal films; Wavy structures; Gratings

#### 1. Introduction

When the surface of a rubber material, the polydimethylsiloxane (PDMS), for example, was covered with a thin metallic film, the wavy structures orientated randomly are often formed. These structures are introduced by the compressive stress at the PDMS/metal interface that was induced by the mismatch of the volumetric contractions between the PDMS substrate and the surface metal film [1]. In the literature, two methods have been introduced to modify the degree of orderliness of the wavy structures. One was to deposit the metal film on a pre-patterned substrate [1,2] and the other was on a pre-stretched substrate [3,4]. The idea for the latter technique, on which the present study focused, was first introduced by Lacour et al. [3] and their purpose was to study the special electrical properties introduced by the various types of the wavy surface structure. Based on these structures, our attentions here are, however, placed on their formation mechanism, surface topography as well as the potential application for the diffraction gratings in micro-optics. Compared with the often used techniques, such as the laser or ebeam writing [5] and the interference lithography [6], generation of the desired surface patterns for diffraction gratings using the

E-mail address: lifish20@yahoo.com.tw (T.-K. Shih).

shrinkage from an elastic material is believed to have a greater potential for fabrication of a large-scale grating at lower costs.

In the following, fabrication processes of the diffraction gratings by deposition of metal films on a pre-stretched substrate and tests of their optical performance will be presented. Section 2 shows the detailed processes for fabricating both the single- and the double-sided gratings. In Section 3, we discuss the scheme to control the pitch of the successive structures and the orientation of the generated gratings and present their optical characteristics. Finally, the primary results of this study are concluded in Section 4.

### 2. Experiment

Steps for fabricating diffraction gratings on the surface of PDMS substrate are described as follows.

(a) The liquid silicone pre-polymer, Sylgard 184 from Dow Corning, was mixed in a weight ratio of 10:1 with the curing agent. After mixing, the pre-polymer was poured into a respective mold and cured for 1 h at a temperature of 70 °C. The solidified PDMS plate with 1 mm thick could be easily fabricated. Then, the PDMS plate was stretched by an external, uniaxial tension load and it was extended in the specified direction prescribed by the tension load. The resulting PDMS substrate is shown in Fig. 1(a).

<sup>\*</sup> Corresponding author. Postal address: 1001 Ta Hsueh Road, Hsinchu 300, Taiwan, ROC. Tel.: +886 3 5712121x55346; fax: +886 3 5724727.

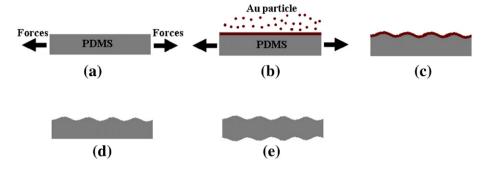


Fig. 1. Schematic diagrams showing the fabrication process of optical gratings on a PDMS substrate.

- (b) A thin gold film with a reasonable thickness was then deposited on the surface of the pre-stretched PDMS substrate in a sputter. The schematic is shown in Fig. 1(b).
- (c) After the thin metal film was deposited, the pre-stretching load on the substrate was released. The elasticity in the PDMS intended to draw the pre-stretched substrate back to its original dimension resulted in the formation of the wavy structures on the PDMS surface. The schematic is shown in Fig. 1(c).
- (d) In order to promote the optical transmittance of the substrate, the surface Au film should be removed. It could be done by dipping the PDMS substrate with the surface Au film, obtained in Step (c), into a chemical solution with the mixture of hydrochloric acid and nitric acid at a volume ratio of 3 to 1 for 30 s. The resulting single-layer gratings are shown in Fig. 1(d).
- (e) Repeating Steps (a), (b) and (c), the second wavy structures could be formed on the other side of the PDMS substrate. After removing the Au films on both sides of the substrate using the same recipe depicted at Step (d), the resulting substrate with double-layer wavy structures, shown in Fig. 1(e), was obtained.

#### 3. Results and discussion

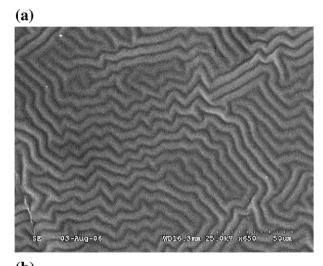
In this section, the surface topographies of the obtained wavy structures are first presented and their corresponding optical performances are then examined and discussed.

# 3.1. Surface topography of wavy structures

Fig. 2(a) shows the SEM (scanning electronic microscope) image of the disordered surface wavy structures on a PDMS plate. This structure could easily be formed from an unstretched PDMS plate covered with an Au film. The formation mechanism of this type of surface structure was due to the different volumetric contraction rates of cooling between the Au film and the PDMS substrate. The coefficients of thermal expansion of PDMS and Au are  $960 \times 10^{-6} / \text{K}$  and  $14 \times 10^{-6} / \text{K}$ , respectively. The larger volumetric contraction rate of the PDMS introduced a drag on the Au film and generated the compressive stress distributed along the Au/PDMS interface that triggered the generation of surface wavy structures. Because there were free of constraints on the plate and the unavoidable non-uniformities, i.e. the thickness of

the Au film was not perfectly uniform, existed in both the Au film and the PDMS substrate, the compressive stresses were unbalanced and thus led to the popped up surface structures orientated randomly [2].

In order to guide the generated wavy structures to arrange regularly, a preloaded tension stress with designated direction and magnitude was considered to be introduced into the PDMS substrate before the Au film was deposited. One of the most



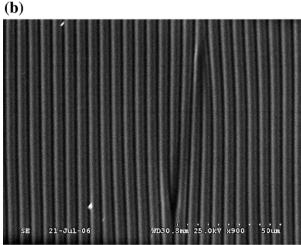


Fig. 2. SEM surface images showing the resulting surface wavy structures formed from two different processes: (a) disordered structures from an unstretched PDMS substrate; (b) ordered structures from a pre-stretched substrate.

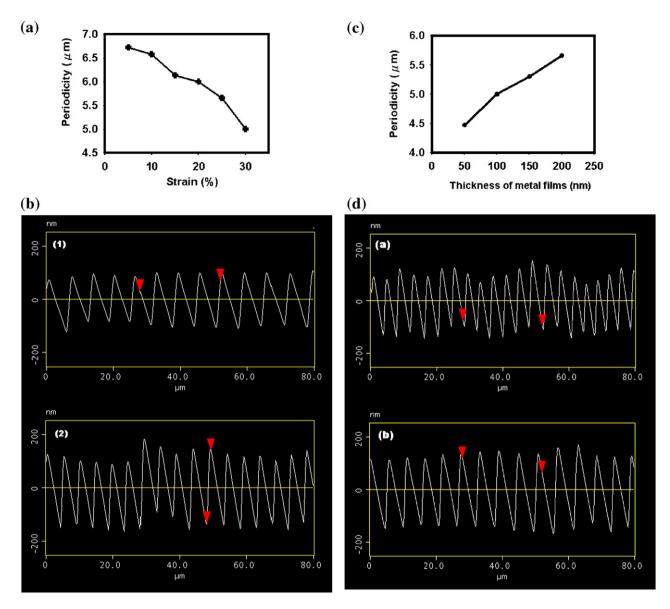


Fig. 3. Effects of strength of the stretched strain and thickness of the metal film on the pitch and the depth of resulting surface wavy structures. (a) The pitch as a function of the strain when film thickness was fixed at 100 nm. (b) The corresponding AFM cross-section images for (a) showing the depths of the wavy structures at strains of 5% (upper) and 30% (lower), respectively. (c) The pitch as a function of the thickness of metal film when the stretched strain was kept at 30%. (d) The corresponding AFM cross-section images for (c) showing the depth of the wavy structures for the film thickness of 50 nm (upper) and 200 nm (lower), respectively.

straightforwardly employable preload was the uniaxial tension. In fabrication Step (b), we observed that there were no wavy structures formed until the preload was released. This was because the magnitude of the compressive stress due to the substrate contraction was smaller than the stretched tension stress introduced by the preload and the substrate was unable to shrink to form any surface wavy structure. Once the preload was released, the elasticity in the rubber PDMS tended to draw the stretched substrate back to its original dimension and led to a uniaxial compression of the substrate. Due to its smaller thermalexpansion coefficient, the surface Au film, however, was not able to contract to the same degree as the substrate did and could only form the wavy structures in order to release the compressive stress from the beneath. These wavy structures were ordered and their orientations were perpendicular to the pre-stretched direction along which the substrate was contracted. Fig. 2(b) shows the SEM image of the formed surface structures after the uniaxial preload tension was released. Each wavy structure there was placed side by side and arranged nearly in parallel. This result demonstrated that a highly ordered surface wavy structure could be achieved by the present method.

An optical grating can be composed of parallel slits with a reasonable distance. As an incident light propagates through a grating, diffraction patterns can be formed. Parameters such as the slit separation (d), the order number of the diffraction pattern (m), the observation angle corresponding to the mth order number of the diffraction pattern ( $\theta_m$ ), and the wavelength of incident light ( $\lambda$ ) are usually employed to characterize the diffraction gratings and they are related through the grating equation given by  $d\sin\theta_m = m\lambda$  [7]. Therefore, the resolving power of the grating increases with the increase of the order number and the number of illuminated slits [7]. Hence, the

parameter, the pitch of the wavy structures, is very important for determining the resolving power. In order to adjust the pitch, two controllable experimental parameters, the magnitude of the pre-stretched strain and the thickness of the Au film, were illustrated in this study. The effect of the stretched strain on the pitch of the structure was first examined and the influence from the thickness of the Au film was then investigated.

A PDMS substrate covered with an Au film with thickness fixed at 100 nm and under a stretched strain ranging from 5% to 30% was investigated. The corresponding pitch of the ordered structure as a function of stretched strain was presented in Fig. 3 (a). It indicated that an enhancement of the stretched strain would result in a reduction in the pitch. Generally speaking, an increase of the stretched strain led to an increase of the recovering uniaxial compressive stress as the preload was released. That would generate more surface wrinkles, corresponding to smaller pitch, and vice versa. After measuring by an atomic force microscope (AFM), the cross-section image of the ordered wavy structures with stains of 5% and 30% were respectively presented in Fig. 3(b) - (1) and (2). Both crosssection profiles were close to the sinusoidal function. As the stretched strain was increased form 5% to 30%, the depth of wavy structures was changed form 195 nm to 262 nm.

The influence of the thickness of the surface Au film on the pitch was investigated based on a PDMS substrate with the stretched strain fixed at 30% but varying the film thickness from 50 nm to 200 nm. The corresponding pitches of the wavy structures as a function of the film thickness were presented in Fig. 3(c). Experiment results demonstrated an increase of the film thickness would result in an increment of the pitch. The increase of the film thickness gave rise to the enhancement of the mechanical strength of the Au film to defend against the substrate contraction. Consequently, the thicker the metal film, the larger the periodicity was formed, and vice versa. The AFM cross-section images for the ordered wavy structures covered with an Au film with thicknesses of 50 nm and 200 nm were presented in Fig. 3(d) - (1) and (2), respectively. As the thickness of the Au film was increased form 50 to 200 nm, the change in the depth of wavy structure was not obvious.

## 3.2. Optical performance of wavy structures

To investigate the optical performance of these wavy structures, an experimental apparatus consisting of a laser diode as the light source, a PDMS plate with the generated wavy structures as the test grating, a screen and CCD camera as the imagine display and recording system was set up. As the laser beam passed through the grating, the generated diffraction patterns were projected on the screen, then they were captured and recorded by the CCD camera.

By this apparatus, two gratings with different periodicities in the wavy structures were examined. Grating 1 was the PDMS plate with the stretched strain of 5% and the thickness of the Au film of 100 nm. For Grating 2, the stretched strain and the thickness of the Au film were 30% and 50 nm, respectively. The diffractive patterns based on Grating 1 and Grating 2 are shown in Figs. 4(a) and (b), respectively. These results showed that

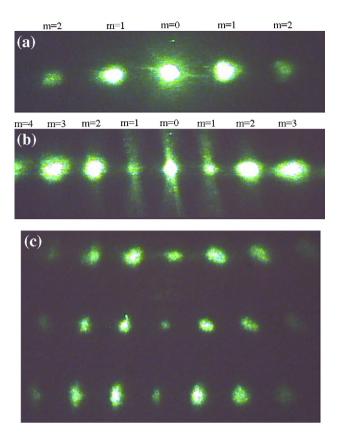


Fig. 4. CCD images showing the diffractive patterns from the single-sided optical grating at differential fabrication conditions, (a) and (b), and from the double-sided optical gratings, (c). The stretched strain and thickness of the surface Au film are respectively 5% and 100 nm for (a) and 30% and 50 nm for (b). The fabrication conditions individually for (a) and (b) are applied respectively for the two gratings of the double-sided gratings.

only the diffraction patterns to the second-order, m=2, could be produced when Grating 1 was examined and the patterns up to the fourth-order, m=4, were still observable if Grating 2 was the test sample. These results clearly demonstrated that gratings with different resolving power could be fabricated by adjusting the stretched strain and the thickness of the deposited Au film.

Because the orientation and periodicity of the wavy structures could be controlled by the direction and the strength of the preloaded strain, following the fabrication steps introduced in Section 2, two gratings with different orientations and periodicities could be fabricated respectively on both sides of a PDMS plate, a double-sided grating plate. Fig. 4(c) shows the SEM image for part of the diffractive patterns from a double-sided grating plate where the orientations of the two-sided gratings were perpendicular to each other. Compared with the diffractive patterns from the single-sided grating, the patterns here were arranged in a two-dimensional array that was the pattern type of a traditional two-dimensional grating generated.

## 4. Conclusions

In this study, we developed both single-sided and double-sided gratings by depositing a metal film on a pre-stretched PDMS substrate. By changing the strength of the stretched strain and the

thickness of the metal film, the periodicity of the gratings could be adjusted in between 4.5  $\mu m$  and 6.8  $\mu m$ . According to our examination, the pitch of the periodic structure on the grating was reduced with the increase of the stretched strain and enhanced with the increase of the thickness of the Au film. Besides, the orientations of wavy structures were always perpendicular to the stretched direction. Optical tests demonstrated that the resolving power of the grating can be easily adjusted by the stretched load and the thickness of the deposited Au film. Consequently, the present proposed method has a great potential for fabrication of a low-cost, large-scale diffractive grating.

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