Rapid Fabrication of Large-Area Periodic Structures by Multiple Exposure of Two-Beam Interference

Lei Wang, Zhen-Hua Lü, Xiao-Feng Lin, Qi-Dai Chen, Bin-Bin Xu, and Hong-Bo Sun, *Member, IEEE*

***Abstract*—Holographic lithography is a convenient, inexpensive technique to fabricate large-area, periodic biofunctional templates such as antireflection and superhydrophobic surfaces. As opposed to two-beam interference (TBI), which produces grating patterns, interference of three or more beams can create multifarious patterns of symmetry, which have broader range of functions. However, as the number of beams used increases, the configuration gets more complicated, and thus possibly incurs more errors of alignment. To avoid the issue, we employed the technique of multi-exposure of two-beam interference to fabricate two-dimensional periodic structures and quasi-periodic structures. The theoretical simulation of $2n$-beam interference (n is a positive integer) and n exposures of TBI was compared to validate that patterns made by $2n$-beam interference could also be made by n exposures of TBI. Structures with symmetry of different folds were demonstrated not only on the negative photoresist SU-8 based on an add-type fabrication approach but also directly on the surface of the infrared window material zinc sulfide (ZnS) through a subtract-type fabrication approach. A transmittance of more than 90% and a water contact angle of 145° were obtained by three exposures of two-beam interference, and a transmittance of 80% as well as a water contact angle of 126° by ablation of nine exposures of TBI on ZnS substrate.**

***Index Terms*—Antireflection, superhydrophobic, multiple exposure of two-beam interference, periodic structures.**

I. Introduction

SURFACES with periodic structures are widely used by nature and humans due to multifarious possible properties such as anti-reflection [1], [2], super-hydrophobia [3], [4], etc., which lead to a variety of applications from OLED (organic light-emitting diode), solar cells, self-cleaning surfaces, etc. [3]–[7]. Many techniques have been developed to produce these surfaces, including self-assembly [8], direct laser writing [5], [9], [10], photolithography [11], holographic lithography [6], [12], etc. Among these techniques, holographic lithography is a convenient, inexpensive technique used to fabricate large-area, defect-free periodic structures, [13], [14], and many efforts have been made to study the approaches of fabricating miscellaneous periodic structures. In 2009, Jiménez-Ceniceros *et al.* investigated the interference of n (n is a positive integer) beams theoretically [15]. In 2010, Wu *et al.* fabricated superhydrophobic lotus-leaf-inspired micro-pillar arrays using four-beam interference [3]; Mao *et al.* fabricated 2D (two-dimensional) photonic quasi-lattices with high-order symmetry by five-beam interference [16]. As opposed to two-beam interference (TBI), which produces grating patterns, interference of three or more beams can create multifarious patterns of symmetry, which has broader range of functions [17], [12], [18], [19]. However, as the number of beams used increases, the configuration gets more complicated, and thus possibly incurs more errors of alignment [20]. To avoid these issues, it is advisable to use as few beams as possible—for example, two beams—and multiple exposures to maintain the pattern that multi-beam interference (MBI) produces. Besides the simplicity of the configuration, multiple exposures of two-beam interference (METBI) possesses more advantages—for instance, it is easy to alter the period only by varying the intersecting angle of the two beams; the symmetry of structures can be determined by choosing the rotating angle of the substrate between consecutive exposures. Some researchers have demonstrated to fabricate 3D photonic crystal structures in SU8 by multiple exposures of two-beam interference and three-beam interference [21], [22]. In 2011, Bai fabricated varies 2D structures in SU8 by multiple exposure of two-beam-interference by tuning the exposure dose [23]. Danh Bich Do obtained ellipticity-controlled microlens arrays in SU8 by double-exposure of two-beam interference and PDMS transliteration [24]. These works are quite pioneering and promising. However, authors have hinted using multi-exposure of two-beam interference to replace multi-beam interference. Relationship between multiple exposures two-beam interference and multi-beam interference is not discussed. Patterns were limited in photoresists unless by a subsequent transliteration which was time-consuming and inefficient.

In this work, we conducted a systematic work. First we compared 2D patterns of light intensity distribution formed by METBI with by MBI in theory to validate that patterns made by $2n$-beam (n is a positive integer) interference can also be made by n exposures of TBI. Two-dimensional structures with up to 18-fold symmetry were fabricated on the negative photoresist SU-8 by METBI based on an add-type approach. Ablation of METBI directly on the infrared window material zinc sulfide (ZnS) was also experimented to obtain sub-wavelength anti-reflection surface by a subtract-type approach. A transmittance of more than 90% along with a 145° water contact angle was obtained by ablation of three exposures of TBI, and an 80% transmittance as well as a 126° contact angle by ablation of nine exposures of TBI.

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II. Principle

In METBI, the final distribution of exposure intensity involves a vector calculation between the two interfering beams in each exposure of TBI, and a scalar calculation between consecutive exposures; but in MBI, only a vector calculation among the interfering beams needs performing. Taking MBI for example, n beams with the same wavelength, amplitude, phase and intersecting angle between adjacent ones superimpose at a spot on a screen; the wave function of the j^{th} beam can be described by:

$$\begin{aligned}\overrightarrow{U}_j(\overrightarrow{P},t) &= \overrightarrow{A}_j(\overrightarrow{P})e^{-i[w_j t - f_j(\overrightarrow{P})]}\\ &= \overrightarrow{A}_j(\overrightarrow{P})e^{if_j(\overrightarrow{P})} * e^{-iw_j t}\\ & \qquad j = 1,2,3\ldots \end{aligned} \tag{1}$$

where $\overrightarrow{\boldsymbol{U}}$ represents the vector wave, $\overrightarrow{\boldsymbol{A}}$ is the amplitude, ω is the frequency, and φ is the initial phase.

So, the total wave function of N interfering beams is

$$\overrightarrow{U}(\overrightarrow{P}) = \sum_{j=1}^{N}(\overrightarrow{U}_j(\overrightarrow{P},t)), \tag{2}$$

The intensity is

$$\begin{aligned} I_N(P) = &\sum_{j=1}^{N} I_j(\overrightarrow{P})\\ &+ \sum_{j=1}^{N}\sum_{\substack{k=1\\ j\neq k}}^{N}\left(\sqrt{(I_j(\overrightarrow{P})I_k(\overrightarrow{P}))}\cos\delta_{j-k}(\overrightarrow{P})\right) \end{aligned} \tag{3}$$

where

$$I_j(\overrightarrow{P}) = \overrightarrow{U}_j(\overrightarrow{P})\overrightarrow{U}_j^{*}(\overrightarrow{P}) = A_j^2(P) \tag{4}$$

$$\delta_{j-k}(\overrightarrow{P}) = \varphi_j(\overrightarrow{P}) - \varphi_k(\overrightarrow{P}). \tag{5}$$

For N-time exposures, (N is the exposure times) the intensity is only a linear superposition of I_n for different coordinates.

Intensity distribution of MBI is described by (3). When exposure times is N, (6) represents the intensity distribution.

$$I_n^N = \sum_{j=1}^{N}(I_n(P_j)) \tag{6}$$

P_j represents coordinate after rotating j times and is described as (7).

$$P_j = P_1[\cos(\theta_j), \sin(\theta_j)] \tag{7}$$

$$\theta_j = (j-1)(n-1)\frac{180^\circ}{n}. \tag{8}$$

when $n = 2$, (6) represents N times exposure two-beam-interference. When $N = 1$, (6) represents n-beam-interference.

To obtain the light intensity distribution, we used software Matlab to perform the calculation, and the final results are shown in Fig. 1.

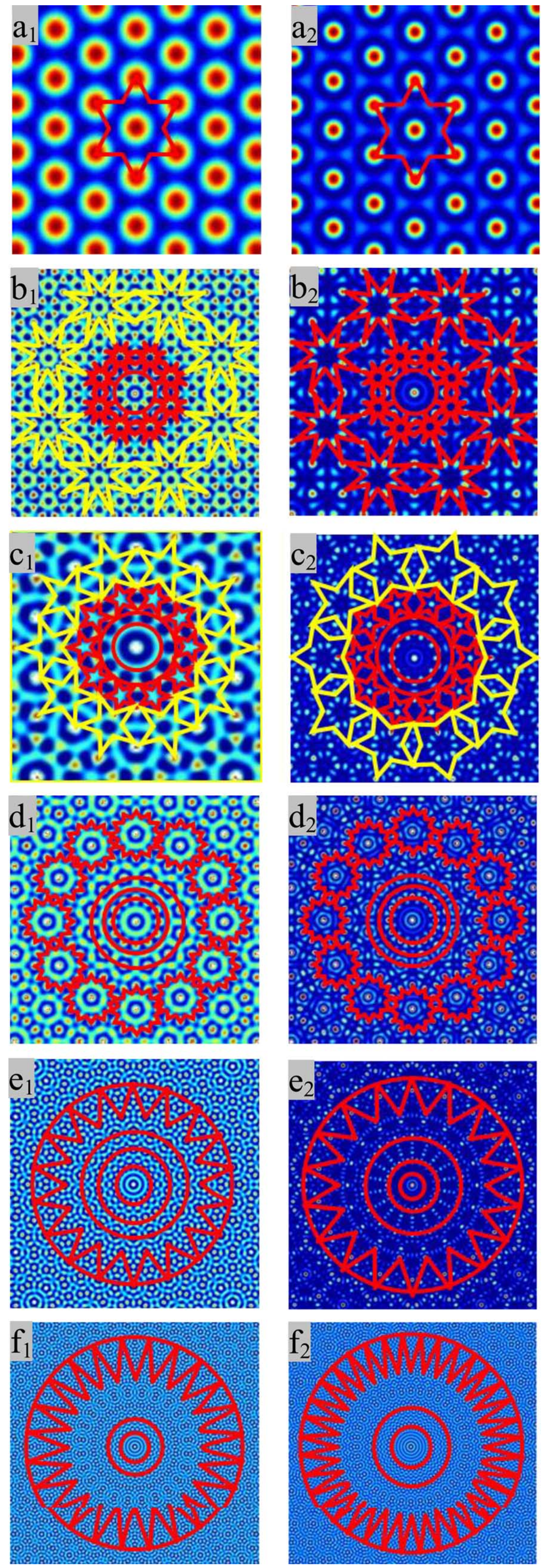

Fig. 1. Shown in the left column are patterns made by 3, 4, 5, 6, 12, 18 exposures of TBI, respectively; shown in the right column are those made by 6-, 8-, 10-, 12-, 24-, 36-beam interference, respectively.

Had carefully compared the image shown in Fig. 1, we concluded that:

1) As in MBI, patterns made by n exposures of TBI are of n-fold symmetry.
2) Maxima and minima intensity in METBI distribute the same as in MBI. Although intensity gradients around maxima in MTBI are much less than those in MBI. Duty ratio of structures fabricated by METBI can be varied by adjusting the exposure power and time in the experiment.
3) A singular point appears in the center of MBI pattern but none in METBI. Energy will be collected to the center if much more beams are used in MBI while a relatively well-proportioned intensity distribution in a large area can be made by METBI.
4) $2n$-exposure MBI can be replaced by n-exposure METBI if large area well-proportioned structures of n-fold symmetry are required.

III. Results and Discussion

A. Add-Type Approach

Periodic structures were made by METBI on negative photoresist based on an add-type approach. The commercial photo-resist SU-8 2025 was used in our work. This photo-resist has been widely used due to near ultraviolet (350–400 nm) processing, low volume shrinkage, good mechanical properties (Young's modulus 4 GPa and biaxial modulus of elasticity 5.2 GPa), superb chemical and temperature resistance [9]. A frequency-tripling, Q-switched, single-mode Nd:YAG laser (Spectra-physics) with an emission wavelength of 355 nm, a frequency of 10 Hz and a pulse duration of 10 ns was employed in the experiment. The power range was 0–1200 mW. Because the maximum intensity at the exposure region in the TBI is four times of one beam intensity, the maximum power density is enough for the beams to penetrate nearly 10 μm deep into SU-8. In the first step, SU-8 was diluted by 50% cyclopentanone; then it was spun on a cleaned glass at a rotational speed of 1000 r/min for 30 s, thereby formed a coating about 10 μm. After a soft bake of 40 min, the sample was exposed by TBI with only one pulse, thus a latent grating took shape in the photoresist layer; then the sample was rotated by a certain angle, and exposed with one pulse again, thus the two gratings formed by the two exposures superimposed, creating a latent 2D structure with four-fold symmetry. In this way, more gratings could be added on to make higher-fold symmetry. In this experiment, the rotation angle should be $180°/n$ where n was the number of rotations. Here only one pulse was used. After n exposures, a 15-min post bake was executed to make molecules of the exposed photoresist crosslink; then the photoresist was immersed in SU-8 developer for 5–6 min, which would dissolve the unexposed and reveal the crosslinked structures.

The atomic force microscopy (AFM; iCON, Veeko) images of the periodic structures are shown in Fig. 2. The hexagonal structures with translational symmetry fabricated by three exposures of TBI are shown in Fig. 2(a). Shown in Figs. 2(b)–(f) are Quasi-periodic structures with long range order but without translational symmetry, which were fabricated by four, five, six, twelve, and even eighteen exposures of TBI, respectively.

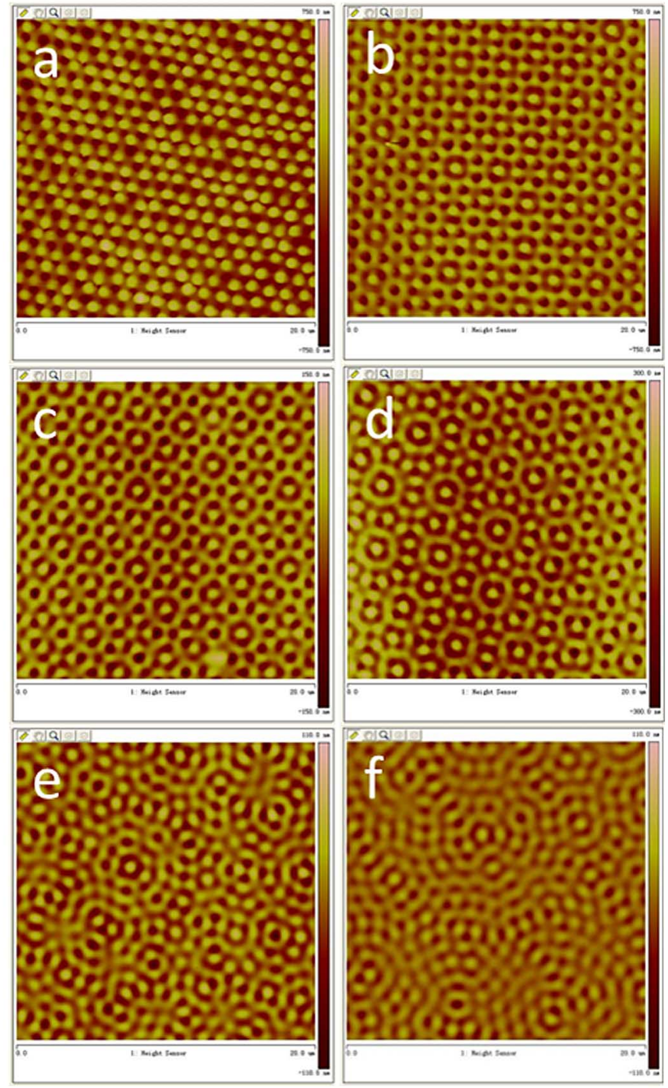

Fig. 2. AFM images of structures made by METBI through (a) 3, (b) 4, (c) 5, (d) 6, (e) 12, and (f) 18 exposures, respectively.

The photo-crosslinking in the negative photoresist SU-8 is actually an accumulation of luminous flux. In the process of METBI, power and exposure time are both variable parameters. In our experiment, however, since only one pulse was used in a single exposure of TBI, power was the only variable. If the power was too high, even the minimum flux accumulation was higher than the threshold after n exposures of TBI, so probably all photoresist would crosslink that no structures would be obtained finally but a flat polymerized SU-8 membrane. On the contrary, if the power was too low that even the maximum flux accumulation was lower than the threshold, no sufficient molecules of SU-8 would crosslink so that the entire sample would be dissolved by the developer. As far as our experiment is concerned, 50 mW was the minimum power that could enable formation of gratings in SU-8; on the other hand, when the power was more than 600 mW, the SU-8 coating would be ablated off. If periodic structures made by n exposures of TBI were needed, the approximate power should be more than an $1/n$ of the minimum power and less than an $1/n$ of the maximum power. The duty ratio of structures can be altered by varying the power in this range. Since the difference of the refractive indices between the crosslinked zone and the non-crosslinked zone was little, it could hardly affect consecutive exposures of TBI. So in this experiment, the power used in each exposure was set the same.

B. Subtract-Type Approach

Periodic structures made by METBI ablation were also demonstrated on the hard material ZnS. ZnS is an excellent widow material for infrared spectra especially in the band of 7 μm–11 μm. In the first step, a ZnS substrate was cleaned by acetone, alcohol, and deionized water in turn. In the ablation of the first exposure of TBI, material under the maximum intensity was ablated off and a grating formed. As in the add-type approach, the structure with multi-fold symmetry was created by rotating the substrate before every exposure. The difference is that in the subtract-type approach, the material was ablated off to form a grating structure in each exposure. The exposure power was all 1100 mW. The diameter of the laser spot was 6 mm. To clean the modified surface, we immersed the sample in H_2SO_4 solution with $\mathrm{pH} > 3$ for one hour. In the process of ablation, the first exposure time was 3 s, and we obtained a grating with a period of 3 μm and a height of about 2.7 μm. Because the absorption of the surface with gratings became larger, the exposure time was reduced so as to ensure the height was identical. Here the best exposure time was 1.6 s, and decreased by 0.1 s in turn every following exposure.

The SEM (scanning electron microscopic, JEOL JSM-6700F) images of structures made by ablation of three, four, five, six, eight, and nine exposures of TBI are shown in Figs. 3(a)–(f), respectively.

In ablation of METBI, the highest pillar of the structure formed in the minimum power density as the material removed was the least. A pattern of METBI in theory is a linear superposition of the patterns of TBI, and the pattern of TBI is sinusoidal. As a sinusoid is the same as its inverse, the pattern fabricated by ablation of METBI is identical to the pattern by METBI in theory. When exposures of TBI are less, for example, four or five, a hole formed by the maximum intensity appears in the center of the period, as shown in the insets of Figs. 3(b) and (c); when the number of rotations is more than five, a pillar formed by the intensity minimum appears, as shown in Figs. 3(d)–(f).

Anti-reflection is an important character of sub-wavelength structure surface (SWSS) [2]. SWSS has a potential application in OLED, solar cells, etc. for reduction of surface Fresnel reflection [1], [2]. Transmittance of ZnS with two-side SWSS made by three, four, five, six, eight, nine exposures of TBI ablation, respectively, is shown in Fig. 4(a). For comparison, the transmittance without SWSS is also demonstrated. A transmittance of more than 90% was obtained through three exposures. Though the transmittance decreases with the number of exposures, it is still higher than 75% in the spectrum of 8.2 μm–12 μm for all numbers of exposures. In addition to anti-reflection properties, SWSS also demonstrates a character of hydrophobia. To investigate it, we modified the surface with fluoroalkylsilane ($CF_3(CF_2)_5CH_2CH_2SiCl_3$) by means of thermal chemical vapor deposition for one hour at 60 °C. Surface energy was greatly reduced due to the introduction of SWSS. The contact angle was 147° on the structure fabricated

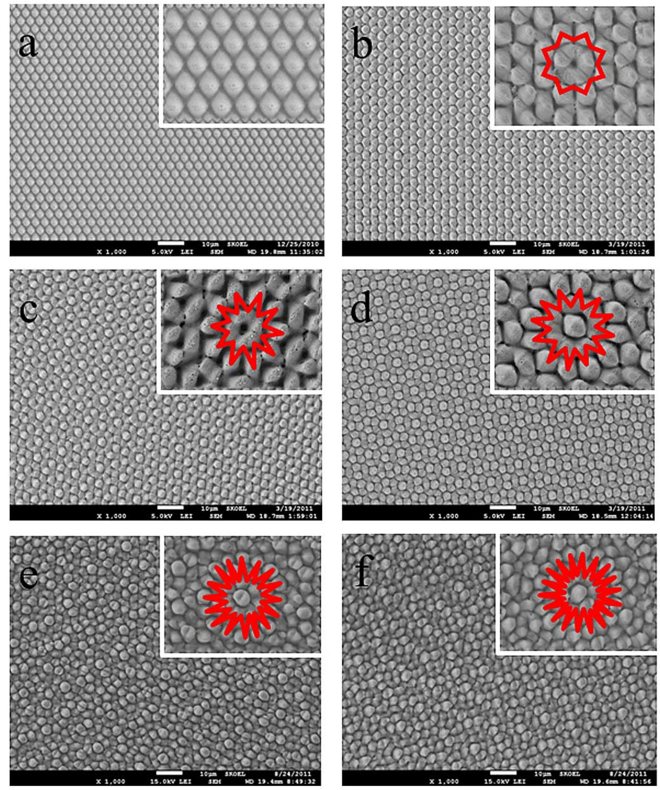

Fig. 3. SEM images of structures fabricated on ZnS by METBI ablation through (a) 3, (b) 4, (c) 5, (d) 6, (e) 8, and (f) 9 exposures.

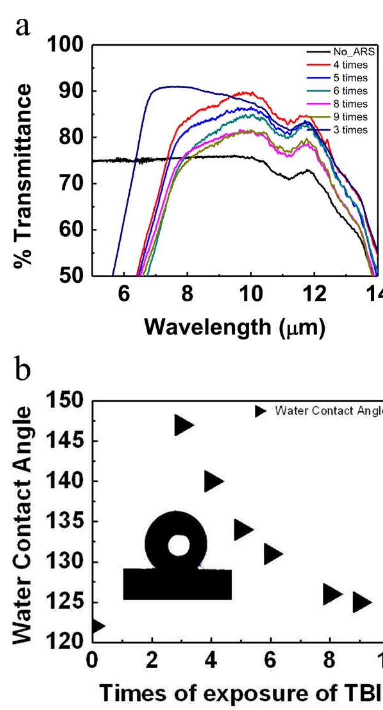

Fig. 4. (a) Transmittance of ZnS with SWSS made by ablation of METBI. (b) Water contact angle of ZnS with anti-reflection structures (ARS) made by ablation of METBI.

by three exposures of TBI, as opposed to 122° on a bare ZnS substrate. The contact angle decreased with the number of exposures because the height of structures became low.

IV. Conclusion

We fabricated periodic structures of various symmetries by METBI. Theoretical simulation of 2n-beam interference and of n exposures of TBI was compared to validate that patterns made by $2n$-beam interference can also be made by n exposures of TBI. Structures with symmetry of different folds were demonstrated not only on the negative photoresist SU-8 based on add-type fabrication but also directly on the surface of infrared window material zinc sulfide (ZnS) through subtract-type fabrication. A transmittance of more than 90% and a water contact angle of 145° were obtained by three exposures of two-beam interference, and an 80% transmittance as well as a 126° water contact angle by ablation of nine exposures of TBI on ZnS substrate. These results show that METBI is a facile, efficient technique of producing periodic structures of various symmetries and that these structures, combining anti-reflection and super-hydrophobia, may find applications where high transmittance and self-cleaning are desirable or necessary, such as windows, OLED, solar cells, etc.

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