Optical Materials 47 (2015) 473–477

Contents lists available at ScienceDirect

Optical Materials

journal homepage: www.elsevier.com/locate/optmat

# Interference lithography for the synthesis of three-dimensional lattices in SU-8: Interrelation between porosity, an exposure dose and a grating period

D.S. Isakov [a,*], N.D. Kundikova [a,b], Y.V. Miklyaev [a,b]

[a] South Ural State University (National Research University), Chelyabinsk, Russia
[b] Institute of Electrophysics of Urals Branch of RAS, Ekaterinburg, Russia

ARTICLE INFO

*Article history:*
Received 15 December 2014
Received in revised form 18 May 2015
Accepted 9 June 2015
Available online 22 June 2015

*Keywords:*
Interference lithography
Photonic crystals
Stereolithography
Resolution
Photoresist SU-8

ABSTRACT

Three-dimensional gratings with different periods were synthesized in SU-8 photoresist through its triple exposure to two-wave interference pattern. Interrelation between porosity, an exposure dose and a grating period were investigated. It was found that decreasing porous grating periods require decreasing density of absorbed laser radiation. Moreover, decrease of grating periods leads to a narrow range of radiation dose that allows us to obtain porous structure.



## 1. Introduction

A photonic crystal is a periodically structured material which can be used for control of light propagation, generation and absorption [1–3]. The technical problem of structures fabrication with a period comparable to the light wave-length should be solved in order to demonstrate the properties of photonic crystals. This problem becomes especially urgent in the near infrared and visible ranges. Many methods have been developed for the synthesis of photonic crystals having photonic band gaps in this spectral range [4–7]. A method of interference lithography is one of the most promising techniques for the photonic crystals synthesis. The method includes the exposure of a photoresist layer to interference pattern [8]. This method of synthesis allows it to obtain large structures with ideal periodicity, low defect concentration and high homogeneity. Moreover, the cost of experimental schemes for the photonic structures synthesis is very low.

To produce a three-dimensional lattice, an interference scheme for three-dimensional light distribution in an interference pattern was proposed [8,9]. To form such an interference pattern, four waves were needed. It is also possible to illuminate a photopolymer layer through a diffraction grating on its surfacer [10,11]. Unlike the previous illumination scheme [8,9], this method makes it quite easy to obtain a structure with lattice symmetry corresponding to the diamond lattice.

A three-dimensional periodic structure can be obtained by means of triple photoresist exposure to a one-dimensional interference pattern with three different orientations of the intensity grating vector [12]. As a result, the three-dimensional periodic distribution of the density of absorbed radiation energy is accumulated in a photoresist layer. The important advantage of this exposure technique is capability to change period of the resulting structure continuously using the same wavelength of laser radiation without changing the lattice symmetry [12]. Multibeam interference does not allow to change the grating period and lattice symmetry independently. It was shown theoretically [13] that for the structures fabricated by this method the lowest refractive index of material for band gap opening is equal to 2.14. This refractive index threshold for band gap opening is comparable to the best results obtained in the diamond lattice [14].

Photoresist SU-8 is the most commonly used material for the synthesis of lattices by the interference lithography [15]. The lattices synthesis using different exposure schemes were successfully realized in this photoresist [8,12,16,19,20]. Porous lattices were obtained with spacing in the ranges from 0.5 to 6 μm. Mutual influence of grating spacing, porosity and photoresist exposure has not been investigated yet. Solving this problem can allow us to extrapolate the results to the not yet investigated range of

* Corresponding author.
*E-mail address:* isakovds@susu.ac.ru (D.S. Isakov).

http://dx.doi.org/10.1016/j.optmat.2015.06.023
0925-3467/

spatial frequencies and understand some features of the process of the photopolymer lattice forming. Here we study the mutual influence of grating spacing, porosity and exposure dose using the method of triple exposure.

## 2. Scheme of the exposure

A triple exposure of a photoresist film to the interference pattern of two waves was used to generate a three-dimensional lattice in the photoresist film by the interference lithography method, as it was done in [17,18]. The experimental scheme is shown in Fig. 1.

The sample was exposed to a two-wave interference pattern. It was rotated by 120 degrees around its vertical axis after each exposure. The time of each exposure was the same. All beams were linearly polarized. The plane of polarization was perpendicular to the plane of the incidence. The beams intensities in photoresist after refraction at the boundary between photoresist and air were equal to each other.

The distribution of the absorbed energy in the photoresist film has the following form:

$$I(\vec{r}) = \sum_{i=1}^{N} I_i \cos^2(\vec{b}_i \vec{r} + \phi_i), \tag{1}$$

where $I_i$ is intensity of $i_{th}$ exposure, $\vec{r}$ is a coordinate, $b_i = k_{1i} - k_{2i}, k_{1i}$ , $k_{2i}$ are wave vectors of interfering waves, $i$ is the number of exposure, $\phi_i$ are phases of intensity patterns. We suppose that polymerization takes place only for intensity $I > I_{th}$, where $I_{th}$ is the threshold density of absorbed radiation for polymerization. It is easy to see from Eq. (1) that vectors $b_i$ are the basis vectors of a reciprocal lattice of the synthesized photonic crystal. The angle between the interfering beams in each of the three exposures was the same. It means that basis vectors of the reciprocal lattice have the same length. The angle between the basis vectors was also the same. This case corresponds to the orthorhombic lattice [13]. A structure view can be depicted as the isosurface of the equal density of absorbed radiation. The example of such a structure for the given threshold value of polymerization is shown in Fig. 2.

As was shown in [12], structures corresponding to simple cubic, face-centered and body-centered lattice can be synthesized by the choice of the angle $\beta$ between the interfering waves and the normal to the sample surface (Fig. 1). As it was mentioned above, the value of the refractive index for the band gap formation has the minimum equal to 2.14.

It should be pointed out that the multibeam interference scheme does not allow us to change the lattice period without changing lattice symmetry. If we change the angle between interfering beams, we change the lattice type at the same time. We have more degree of freedom in the triple exposure scheme and can control the lattice symmetry and the grating spacing

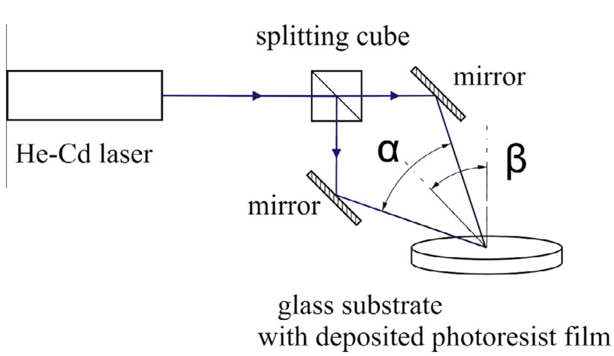


**Fig. 1.** Exposure scheme.

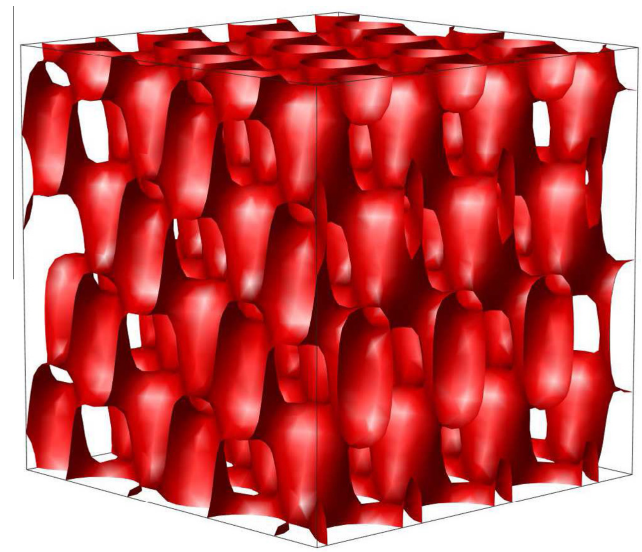

**Fig. 2.** 3D isosurface of equal density of absorbed laser radiation with the following parameters: $\alpha = 14°$, $\beta = 32°$, $\lambda = 442$ nm, $n = 1.67$, $I_{th} = 10$ mW.

independently. To keep the lattice symmetry unchanged, angle $\beta$ should be constant (Fig. 1).

## 3. Description of the experiment

A He-Cd laser with a wavelength of 442 nm, corresponding to the low absorption of the material, was used for the synthesis of the three-dimensional photonic crystal lattices. The output power of the He-Cd laser was 10 mW. Photoresist SU-8 with cationic polymerization mechanism was used as a photomaterial. The photoresist was deposited on the substrate by a two-step spin coating in accordance with the recommendations of the manufacturer. The first step was the photoresist pre-allocation on the sample surface at 500 rev/min within 10 s. The second step was formation of a photoresist layer with the desired thickness at the speed of 3000 rev/min within 30 s.

The described procedure provides the layer thickness of approximately 40–50 μm in accordance with the photoresist specifications. Then the sample was subjected to a two-stage drying. The first stage was drying at 60 °C within 5 min followed by drying at 95 °C within 20 min to remove the solvent. The exposed photoresist was baked at the temperature of 95 °C within 6 min. After baking, the photoresist was placed into developer PGMEA (2-(1-methoxy) propyl) for 5–7 min, and was then fixed with isopropyl alcohol. A layer of the primer had been deposited on the glass surface previously to improve adhesion of the photoresist to the glass to prevent peeling of the lattice from the substrate. The primer is the same photoresist which will be diluted by a solvent. The primer was deposited and processed using the same technology as the base layer, but before baking, the photoresist layer was illuminated all over by a UV lamp for complete polymerization. The quality of the structures was analyzed by using a scanning electron microscope JEOL JSM-6460LV. The 10 nm platinum layers were deposited on the samples surface before investigation.

## 4. Synthesis of three-dimensional lattices photonic crystals

We synthesized and investigated approximately 60 samples with different periods and exposure time. The lattice period was changed by variating angle $\alpha$ between the interfering waves and by changing angle $\beta$ between the bisector of the waves and the