Journal of ELECTRICAL ENGINEERING, VOL. 60, NO. 3, 2009, 166–169

COMMUNICATIONS

PERIODIC STRUCTURES PATTERNED ON METAL AND III–V COMPOUND SURFACES USING TWO–BEAM INTERFERENCE METHOD

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We present an optical interference method as an efficient tool for high quality one- and two-dimensional periodic structure fabrication for photonic applications. Different types of two-dimensional periodic structures are prepared using a two-beam interference technique by double exposure process. also the patterning of an au layer deposited on glass and GaAs substrates is provided using the two-beam interference method. Quite homogeneous and two- dimensional structures with small periodicity were prepared into the photoresist, Au layer and GaAs substrate. the experimentally prepared structures are well in agreement with the theoretically designed ones.

Keywords: periodic structure, interference, holographic lithography

1 INTRODUCTION

Intensive research on photonic crystals with periodic dielectric microstructures has started last years because of their unique optical properties attractive for numerous photonic applications. Periodic structures with a small period have been successfully applied to optoelectronic devices. These can enhance light extraction of light-emitting diodes [1], allow high differential quantum efficiency and output power of laser diodes [2, 3], allow sharp bending and cause clear-cut of waveguides [4, 5].

One of the most promising technologies for periodic structure fabrication is holographic lithography [6–10]. There are different ways for generation of light patterns by holographic lithography using a single exposure of an interference pattern generated by multiple beams [7] or using multiple exposures of an interference optical field produced by two beams [8–10]. This multi-exposure technique is used for fabrication of different one- (1D), two- (2D) and three-dimensional (3D) periodic structures in photoresist materials.

In this paper we present a two-beam interference method as an effective tool for preparation of 1D and 2D periodic structures with a small period. This method in combination with the presented theoretical model allows fabricating different types of periodic structures. The designed periodic structures are patterned in a thin film of a standard photoresist layer as well as in a thin Au layer and GaAs substrate.

2 EXPERIMENTAL

Patterning of periodic structures has been examined using standard positive photoresists AZ 4562 and AZ 5214E. Different set of samples were patterned — a 7 μm thick photoresist layer, an Au layer evaporated on a glass substrate and a GaAs substrate. For patterning of periodic structures the 7 μm thick photoresist film AZ 4562 was spin-coated on a glass substrate with post-baking at 65 °C for 2 minutes and at 103 °C for 3 minutes to remove the solvent. The photoresist has to take up water from the environment (rehydration) for fast and homogeneous development. After exposure the samples were finally developed in AZ 400K developer for 40 seconds, rinsed in DI water and dried with nitrogen.

For the preparation of periodic structures formed into the Au layer a 5 nm thick Ni adhesion layer was deposited on the glass substrate by sputtering followed by a 100 nm thick Au layer. These samples were covered by a 1.5 μm thick photoresist film as a mask. The photoresist film was spin-coated with post-baking at 103 °C for 50 seconds. After exposure and photoresist development, the Au layer was etched off in a solution consisting of KI, I_2 and DI water with etch rate ≈ 1 nm/s, using the patterned photoresist film as a mask.

For the preparation of periodic structures formed into the GaAs substrate, a 700 nm thick AZ 5214E film was spin-coated with post-baking. After exposure and photoresist development, the samples were etched in RIE mode in CCl_4/He based plasma in a ROTH & RAU MICROSYS 350 machine. The chlorine-based plasma was generated by a radio frequency (rf) field at 13.56 MHz supplied via a stainless steel electrode (⌀ 200 mm). The temperature of the electrode was stabilized at 25 °C by He flowing into the chamber at 4 sccm. During etching the flow of CCl_4/He was 6 sccm and the working pressure was 0.8 Pa. Before introduction of CCl_4/He, the chamber was evacuated to a background pressure $< 5 \times 10^4$ Pa.

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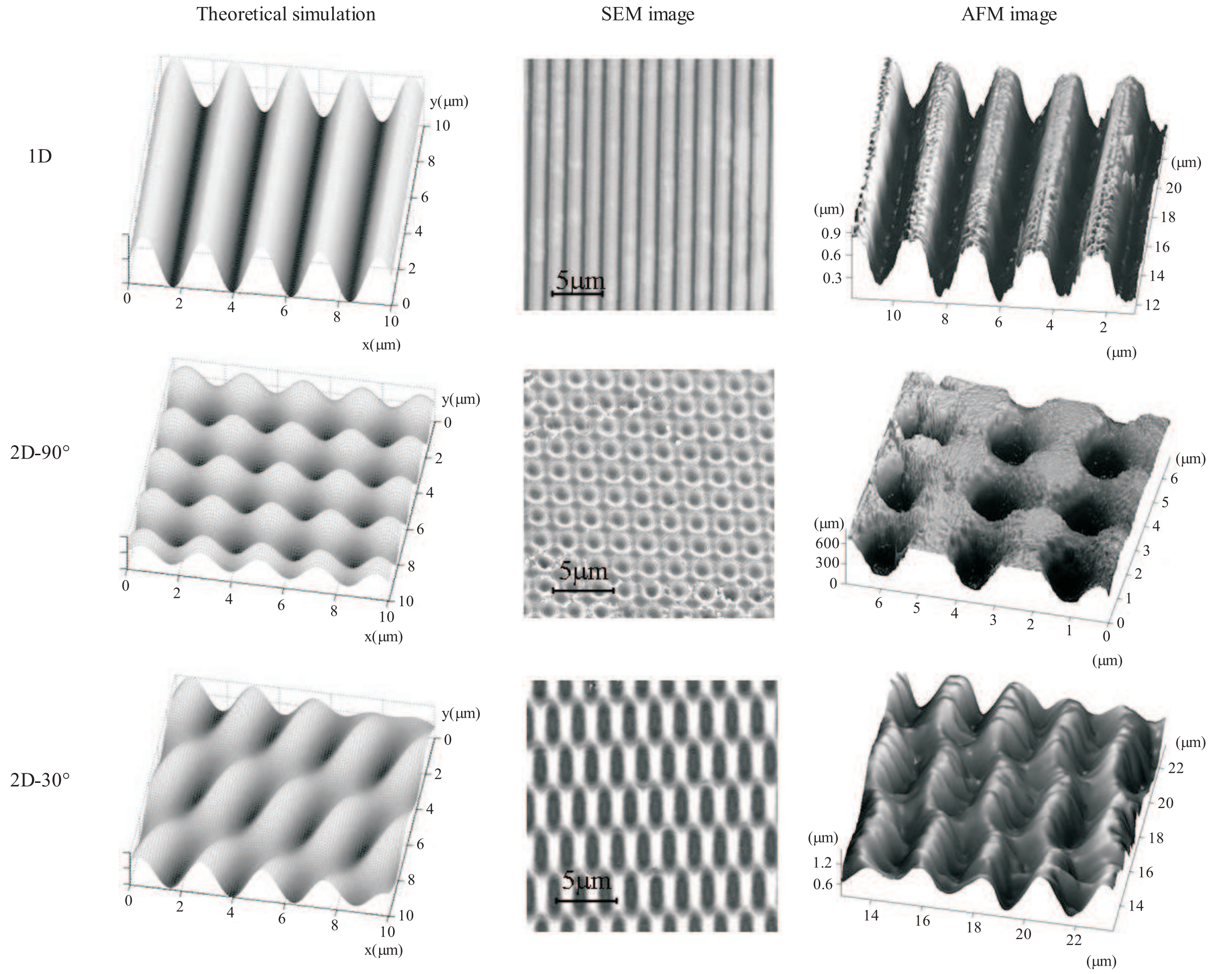

Fig. 1. Simulation, SEM and AFM images of 1D and 2D periodic structures patterned in $7\,\mu$m thick photoresist film

The 1D optical field was formed by an interference of two coherent beams of an argon ion laser operating at 488 nm wavelength. The exposure intensity $20\,\text{mW/cm}^2$ was set in both laser beams. The sample exposure was realized by this periodical optical field using exposition times in the range from 15 to 120 s. Only weak absorption is documented for this type of employed photoresist in the region of the Ar laser wavelength, which affects insufficient polymerization of the photoresist at exposure times < 30 s [11]. The adequate exposure time for this type of photoresist and radiation wavelength was optimized to be 45–90 s [10]. The structure quality has been examined by Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM).

3 EXPOSURE DETAILS

The photoresist layer exposed using the interference optical field pattern forms periodic structures after development. The design of the optical field pattern can be proposed by a simple theoretical approach of the interference of two optical plane waves. The intensity distribution of the interference pattern of the two optical plane waves with the same intensity in the xy plane can be expressed as [8]

$$I_\alpha = 4I_0 \cos^2\big[k \sin\theta (x\cos\alpha + y\sin\alpha)\big]\,, \tag{1}$$

where I_0 is the intensity of the interfering beams, k is the wave number, θ is the semi-angle between the two interfering beams and angle α represents the sample orientation in the xy plane. The optical field periodicity Λ is determined by

$$\Lambda = \frac{\lambda}{2\sin\theta}\,, \tag{2}$$

where λ is the wavelength of the interfering beams. In the case of multiple exposures at different angles α, the exposure dose is accumulated and the total exposure is the sum of the partial exposures. Then the space distribution of the final exposure $I(x,y)$ for the double-exposed process can be expressed as the sum $I(x,y) = I_1(x,y) + I_2(x,y)$, where I_1 and I_2 are the exposure intensities described by eq. (1) for the actual angle α. Two-dimensional periodic structures can be prepared by in-plane rotation of the sample at a definite angle α after the first exposure and then exposed again.

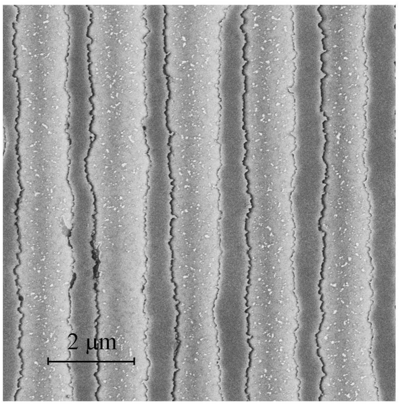

Fig. 2. SEM image of 1D structure patterned in Au layer

4 RESULTS AND DISCUSSION

By applying the two-beam interference method, periodic structures were patterned in the $7\,\mu$m thick photoresist film deposited on the glass substrate, thin Au layer and GaAs substrate. The design of structures was theoretically proposed using eqs. (1, 2) and the quality of the prepared structure has been examined by SEM and AFM.

Using eq. (1) the final profile of various 1D and 2D periodic structures was proposed. Beams intersected at the sample surface seating with definite angle 2θ. The depth profile of 1D structure formed in the $7\,\mu$m thick photoresist is in the range of 0.6–0.7 μm depending on the developing time. The estimated periodicity from SEM and AFM images is in the range $\Lambda = 2.2$–$2.3\,\mu$m and corresponds to the simple approach for interference of two beams $\Lambda = 2.33\,\mu$m containing an angle $2\theta = 12°$ according to eq. (3). The 2D structures were prepared by rotation between single exposures at $\alpha = 30°$ and $90°$. The depth profile can be estimated from AFM analysis to be $0.6\,\mu$m (Fig. 1).

Patterning of Au layer

One-dimensional structures were prepared into the 100 nm thick Au layers sputtered onto glass substrates with 5 nm thick Ni adhesion layer. The period of 1D structures formed into Au layer was set to $\Lambda = 1.75\,\mu$m by choosing the $2\theta = 16°$.

Quite homogeneous periodic structures with periodicity of $\Lambda = 1.8\,\mu$m were revealed from SEM investigations (Fig. 2). Dark stripes in the SEM image correspond to the non-etched Au layers and light stripes represent the Ni adhesion layers after Au removal.

Patterning of GaAs substrate

One-dimensional periodic structures were prepared into GaAs substrate. The period of 1D structures formed into GaAs surface was set to $\Lambda = 2.0\,\mu$m by choosing $2\theta = 14°$. Homogeneous periodic structures of periodicity of $\Lambda = 2\,\mu$m were documented from AFM and SEM analysis (Fig. 3, 4). Quite square, regular and homogenous structures with a depth of 150 nm were obtained using a short time RIE process (90 s) (Fig. 3a). In this phase the GaAs surface is patterned through the open regions of the photoresist mask. By prolonging the RIE process time (150 s), deep structures were formed (500 nm, Fig. 3b) reflecting the sinusoidal shape of the preserved photoresist mask (Fig. 1). This technique usually employs a bottom antireflection coating to eliminate the vertical standing wave patterns. Without antireflection coating the preserved photoresist regions for deep structures were removed in stages reflecting this standing wave pattern (Fig. 4).

Slight irregularities in the wide range images can be found, if this experiment is performed without an expander. These irregularities are caused by the Gaussian shape of the laser beam intensity [10]. This technique using the expander allows optimizing the intensity decrease from the centre to the edge to less than 15 % in the area with a diameter of 5 mm. Such a decrease in intensity is acceptable for implementation of this technique to operations performed on optoelectronic devices.

5 CONCLUSION

The two-beam interference method is an effective tool for fabrication of periodic structures with a small period-

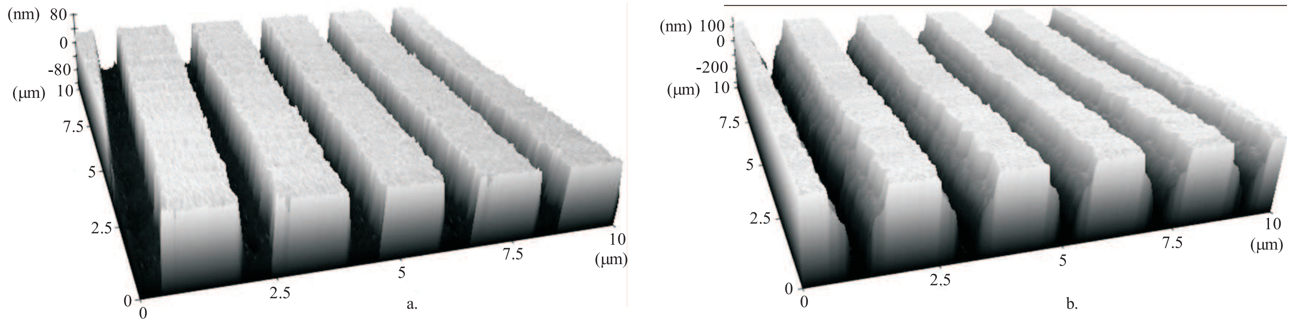

Fig. 3. AFM image of a) shallow (150 nm) and b) deep (500 nm) 1D structure prepared in GaAs substrate

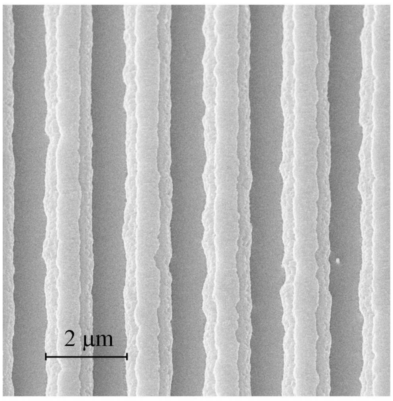

Fig. 4. SEM image of 1D structure patterned in GaAs substrate

icity using a standard photoresist. This method has been successfully examined for preparation of 1D and 2D periodic structures in a thin photoresist film deposited on a glass substrate as well as for patterning of 1D structure into a thin Au layer and a GaAs substrate.

The shape and period of the formed structure can be simply adjusted by the beam geometry as well as by sample rotation between exposures. Variability of the presented interference method opens the possibility to form periodic structures for photonic and optoelectronic applications.

Acknowledgement

Authors would like to thank Professor J. Kováč for cooperation with structure preparation and characterization. This work was done in Center of Excellence CENAMOST (Slovak Research and Development Agency Contract No. VVCE-0049-07) with support of grant VEGA 1/0868/08 and 1/0689/09.

REFERENCES

[1] HSIEH, M. L.—LO, K. C.—LAN, Y. S.—YANG, S. Y.—LIN, C. H.—LIU, H. M.—KUO, H. C. : One-Shot Exposure for Patterning Two-Dimensional Photonic Crystals to Enhance Light Extraction of InGaN-Based Green LEDs, Photonics Technology Letters **20** (2008), 141–143.

[2] ALTUG, H.—VUCKOVIC, J. : Photonic Crystal Nanocavity Array Laser, Optics Express **13** (2005), 8819–8828.

[3] WU, X.—YAMILOV, A.—LIU, X.—LI, S.—DRAVID, V. P.—CHÁNY, R. P. H.—CAO, H. : Ultraviolet Photonic Crystal Laser, Appl. Phys. Lett. **85** (2004), 3657–3659.

[4] MEKIS, A.—CHEN, J. C.—KURLAND, I.—FAN, S. H.—VILLENEUVE, P. R.—JOANNOPOULOS, J. D. : High Transmission Through Sharp Bends in Photonic Crystal Waveguides, Phys. Rev. Lett. **77** (1996), 3787–3790.

[5] DAVID, A.—MEIER, C.—SHARMA, R.—DIANA, F. S.—DenBAARS, S. P.—HU, E.—NAKAMURA, S.—WEISBUCH, C.—BENISTY, H. : Photonic Bands in Two Dimensionally Patterned Multimode GaN Waveguides for Light Extraction, Applied Phys. Lett. **87** (2005), 101–107.

[6] CAMPBELL, M.—SHARP, D. N.—HARRISON, M. T.—DENNING, R. G.—TURBERFIELD, A. J. : Fabrication of Photonic Crystals for the Visible Spectrum by Holographic Lithography, Nature **404** (2000), 53–56.

[7] LIN, Y.—HERMAN, P. R.—ABOLGHASEMI, E. L. : Proposed Single-Exposure Holographic Fabrication of Microsphere-Type Photonic Crystals Through Phase-Mask Techniques, J. Appl. Phys. **97** (2005), 096102.

[8] LAI, N. D.—LIANG, W. P.—LIN, J. H.—HSU, C. C.—LIN, C. H. : Fabrication of Two- and Three-Dimensional Periodic Structures by Multi-Exposure of Two-Beam Interference Technique, Optics Express **13** (2005), 9605–9611.

[9] QUINONEZ, F.—MENEZES, J. W.—CESCATO, L.—RODRIGUEZ-ESQUERRE, V. F.—HERNANDEZ-FIGUEROA, H. —MANSANO, R. D. : Band Gap of Hexagonal 2D Photonic Crystals with Elliptical Holes Recorded by Interference Lithography, Optics Express **14** (2006), 4873–4879.

[10] ŠKRINIAROVÁ, J.—PUDIŠ, D.—MARTINČEK, I.—KOVÁČ, J.—TARJÁNYI, N.—VESELÝ, M.—TUREK, I. : Periodic Structures Prepared by Two-Beam Interference Method, Microelectronics Journal **38** (2007), 746–749.

[11] Bolsen M. AZ® 5200. New Jersey: Hoechst, Sommerville, 1988.

Received 20 October 2008

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