

## APPLICATION OF AMORPHOUS CHALCOGENIDE FILMS FOR RECORDING OF HIGH-FREQUENCY PHASE-RELIEF DIFFRACTION GRATINGS

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The investigation results of the research on high-frequency holographic diffraction gratings recorded by helium-cadmium and argon laser radiations are presented. For decreasing grating period glass prisms were used. Inorganic chalcogenide glasses photoresist treated by the newly developed selective etching solution was chosen as a registering media. High-quality gratings and their polymeric copies with spatial frequency up to  $6000 \text{ mm}^{-1}$  and diffraction efficiency up to 44% were obtained.

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### 1. Introduction

High-frequency phase-relief gratings are used for input-output light coupling in optical sensors [1,2], at various surface subwavelength structures (zero-order diffractive elements)[3] etc. For manufacturing of such gratings costly electron beam lithography or ultraviolet lithography with use of phase-shifting masks would be required. More simple and cheap method is holographic recording of interferential pattern formed by two, or more coherent beams on a photoresist. Such method allows to receive high-quality and sizable gratings.

As it was shown earlier, vacuum-evaporated layers of chalcogenide glasses (ChG) (for example  $\text{As}_2\text{Se}_3$ ,  $\text{As}_2\text{S}_3$ , or As-S-Se composition) are good registering media for diffraction optical elements [4-7]. Such chalcogenide media are characterized by high resolution capability, optical uniformity, sensitivity to the irradiation of available lasers, absence of shrinkage during postexposure treatment. Using chalcogenide resist the high-quality holographic gratings were obtained with spatial frequencies in the range of  $600$  to  $3600 \text{ mm}^{-1}$  and diffraction efficiencies up to 80 % in polarized light [4].

In this paper we report the results got during the investigation of high-frequency gratings recording onto ChG photoresists deposited by thermal vacuum evaporation. Gratings and their polymeric copies with spatial frequency up to  $6000 \text{ mm}^{-1}$  were produced and their properties were investigated.

### 2. Experiment

The ChG layers for holographic diffractive grating recording were deposited on high-quality polished glass substrates or glass prism surface using thermal vacuum evaporation at the pressure  $10^{-3} \text{ Pa}$ . To eliminate the interference effects connected with reflectance from the substrate back surface, a chromium layer was deposited on the glass before photoresist deposition. The ChG layer onto prism surface were deposited without chromium. The layer thickness was controlled during

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deposition process by the quartz-crystal-oscillator monitoring system (KIT-1) and reached 60 nm for chromium layer and 400 nm for photoresist ( $As_{40}S_{60}$ ) layer.

The prepared samples were exposed by interferential pattern that was generated by an argon laser (wavelength of 458 nm), or helium-cadmium laser (wavelength of 440 nm) using the holographic setup assembled by the wave-amplitude division method and angle  $120^\circ$  between laser beams. For decreasing of grating period glass prisms were used. The expose values varied from 0.05 to  $0.5 \text{ J/cm}^2$ .

Fig. 1 (a, b) shows two methods of grating recording using glass prism. In the first method the ChG photoresist was deposited directly onto prism, in the second – the prism was applied to substrate with ChG layer using immersion liquid. Both methods allow to increase spatial frequency of grating in  $n$  time, where  $n$  - index of refraction of prism (in our case  $n$  was equal 1.5).

After exposure, the samples were chemically treated in non-water alkaline organic solutions to form a relief pattern. The duration of etching process was chosen equal to the time of complete dissolution of unexposed photoresist (negative etching). The obtained relief-phase gratings were used for replication of photopolymer copies. The fabricated gratings and copies were covered in vacuum with reflective thin-film ( $\sim 30 \text{ nm}$ ) Al layer.

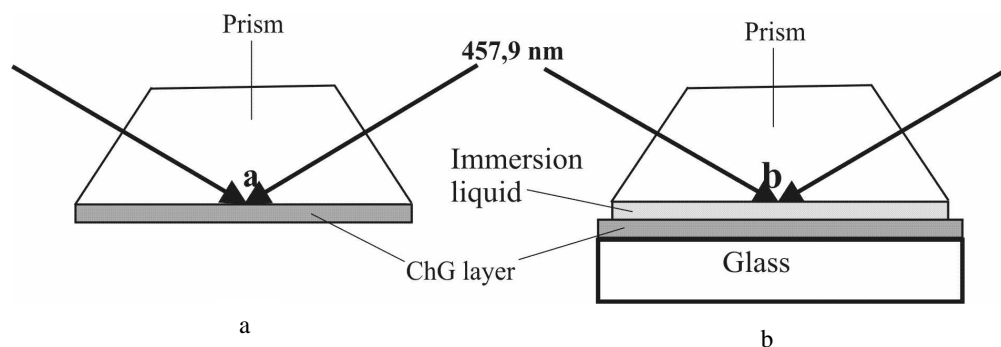


Fig. 1. High-frequency gratings recording using glass prism: (a) – ChG layer deposited onto prism side, (b) – plate with ChG photoresist was exposed through prism and immersion liquid.

Spectral dependencies of the diffraction efficiency  $\eta$  for the first order of diffraction were measured for nonpolarized light. The  $\eta(\lambda)$  measurements ( $\lambda$  - wavelength) were carried out in the setup close to the Littrow scheme and the angle between incident and diffracted beams was 4 deg. The grating diffraction efficiency was taken as the ratio of intensity of light diffracted into the first order to the intensity of incident light reflected from Al layer. The surface patterns of the gratings and copies were examined with a Dimension 3000 scanning probe microscope (Digital Instruments) in the AFM tapping mode.

### 3. Results and discussions

In our previous investigations [4] it was established that groove profiles of gratings (and its optical characteristics) depend on exposure, selectivity of etchant and postexposure etching time. We have developed high-selective etchant for  $As_2S_3$  layers, based on non-water alkaline organic solutions. Exposure time and etching time are chosen experimentally, and the  $\eta$  value of the grating is used as the criterion. Maximal diffraction efficiency can be obtained for gratings with groove profiles close to sinusoid. Under- or overexposure results in cycloid form of grooves.

The optimal exposure for recording of high-frequency gratings using glass prism (for both methods shown on Fig. 1) was determined experimentally also, but as the criterion we have used both  $\eta$  value and AFM scanning results. For  $\lambda = 458 \text{ nm}$  optimal exposure is  $0.3 \text{ J/cm}^2$  and for  $440 \text{ nm}$  -  $0.1 \text{ J/cm}^2$ .

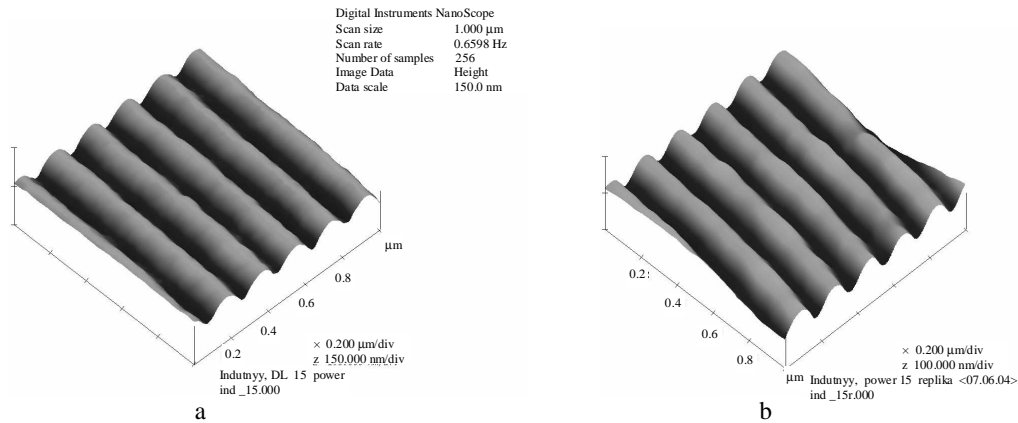


Fig. 2. Relief of grating produced on the 400 nm  $\text{As}_2\text{S}_3$  layer (a), and its photopolymer copy (b).

Fig. 2 shows surface of the grating (a) obtained using  $\text{As}_2\text{S}_3$  inorganic photoresists, and its photopolymer copy (b). This grating were recorded using argon laser (wavelength of 458 nm), with exposure equal  $0.3 \text{ J/cm}^2$ .

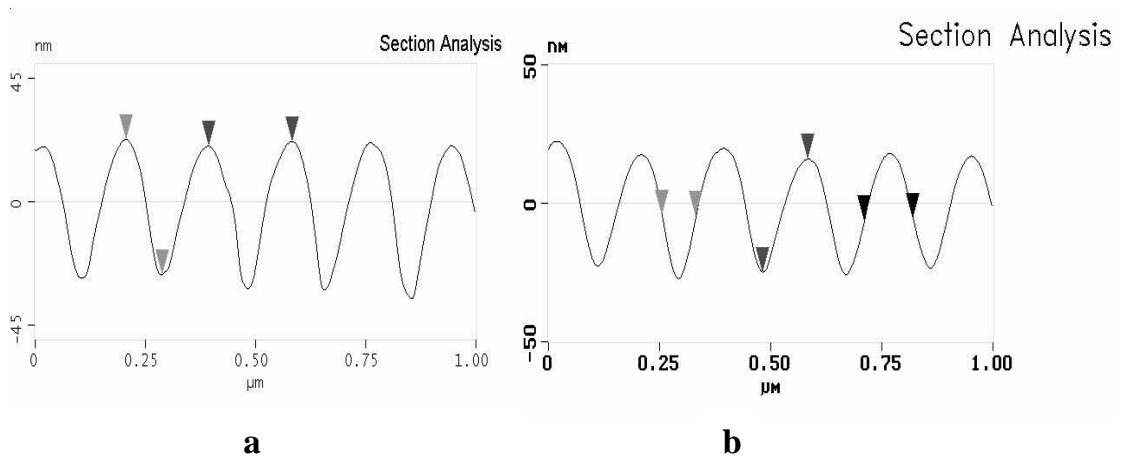


Fig. 3. Groove profile of grating produced on the 400 nm  $\text{As}_2\text{S}_3$  layer (a), and its photopolymer copy (b).

The groove profile of the replica is identical to the relief profile of the master grating and is close to sinusoid for both gratings, as seen from Fig.3. The grating period ( $d$ ) is 187 nm (spatial frequency  $\nu = 5350 \text{ mm}^{-1}$ ), relief depth  $h$  of the original grating is 50 nm, the modulation depth ( $h/d$ ) reached 0.27. Thus, in accordance with [9], we have obtained high-modulation gratings. For photopolymer copy we obtained some reduction of relief depth ( $h = 44 \text{ nm}$ ) which can be connected with shrinkage of photopolymer.

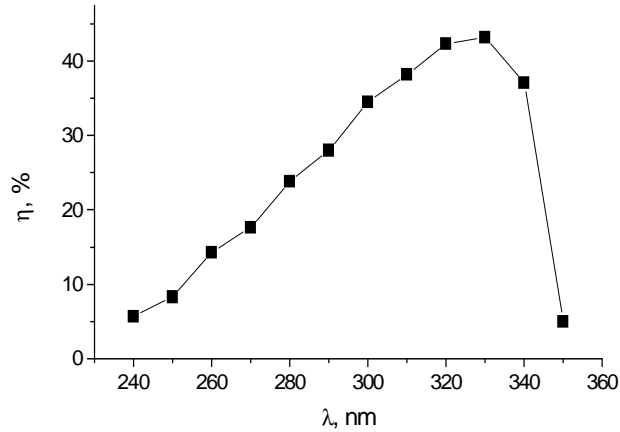


Fig. 4. Spectral dependence of diffraction efficiency  $\eta$ . The grating was recorded on  $\text{As}_2\text{S}_3$  layer by He-Cd laser ( $\lambda = 440$  nm), spatial frequency  $5750$   $\text{mm}^{-1}$ , nonpolarized light.

Fig. 4 shows the  $\eta$  spectrum for nonpolarized light of grating with  $\nu = 5750$   $\text{mm}^{-1}$  produced on  $\text{As}_2\text{S}_3$  layer by He-Cd laser using second method from Fig. 1. The maximum value is 44%, and shape of spectral distribution is typical for high-frequency grating with sinusoid groove profile.

#### 4. Conclusion

The optimal exposure for recording of high-frequency gratings with sinusoidal groove profile on the  $\text{As}_2\text{S}_3$  layers are determined ( $0.3$   $\text{J}/\text{cm}^2$  for 458 nm and  $0.1$   $\text{J}/\text{cm}^2$  for 440 nm). The grating samples and their polymer replicas with the sizes  $20 \times 30$   $\text{mm}^2$  and spatial frequencies up to  $6000$   $\text{mm}^{-1}$  are obtained.

#### References

- [1] I. Szendro, Proc. SPIE **4284**, 80 (2001).
- [2] J. Voros, J. J. Ramsden, G. Csucs et al., Biomaterials **23**, 3699 (2002).
- [3] M. Pfeffer, Europhotonics **8**(6), 34 (2003).
- [4] I. Z. Indutnyi, A. V. Stronski, S. A. Kostioukevich et al., Optical Engineering **34**, 1030 (1995).
- [5] J. Teteris, M. Reinfelde, J. Optoelectron. Adv. Mater. **5**, 1355 (2003).
- [6] M. Vlcek, P. J. S. Ewen, T. Wagner, J. Non-Cryst. Solids 227-230, 743 (1998).
- [7] V. I. Min'ko, P. E. Shepeliavyi, V. A. Dan'ko, et al., Semicond. Phys., Quant. Electron. & Optoelectron. **7**, 88(2004).