

## OPTICAL ELEMENTS FOR IR SPECTRAL REGION ON THE BASE OF ChVS LAYERS

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In this paper, the fabrication of optical elements for the IR spectral region on the base of As-S films is considered. The peculiarities of the selective etching processes of  $As_{100-x}S_x$  films in amine-based etching solutions are considered. The transmitting diffraction gratings with the diffraction efficiency in the first order of  $\sim 23\%$  for a grating period of  $6\ \mu\text{m}$  were obtained. Matrices of refractive microlenses with the diameter of a single lens of  $\sim 12\ \mu\text{m}$  were obtained by the contact printing using halftone masks. The obtained results enable to consider As-S layers as perspective relief forming media for the fabrication of various optical elements in the IR spectral region.

### Introduction

At present, IR technique has found a wide utilization in various areas of human activity. The diverse circle of problems which are solved with the help of IR devices in medicine, ecology, armaments, and machine building determines their intensive use in physics and technology. The achievements of investigations in this direction lead to the creation of a new direction in optoelectronics - IR-optoelectronics - which includes the development of new design-technological principles of the IR systems. This is based on developments of the technology of integrated devices arrays, which combine the functions of information receiving as well as information processing. Such a combination leads to the possibility of signals processing directly in the focal plane with the improved signal-to-noise characteristics. The transition to the new design-technological principles lead to the necessity of development of fabrication methods for optical elements of such systems. Chalcogenide vitreous semiconductors (ChVS) are characterized by a high transparency in the IR spectral region (from visible up to  $15\ \mu\text{m}$ , the band of transmittance is changed with the ChVS composition) and also by high values of the refractive index in this region. Values of the refractive index in the IR region can be changed by the photodoping of ChVS layers with Ag, Cu, etc. This improves the operation characteristics of optical elements, and their fabrication on the base of ChVS was the subject of intense investigations during the last decade [1 - 6]. The presence of various photoinduced effects in ChVS

provides a possibility of their use as registering media in such applications as holography, microlithography, information storage, and optoelectronics. Photoinduced effects observed in ChVS can be used for the fabrication of relief structures and various transmission optical elements operating in the IR region because these materials have excellent transmittance in the IR region.

In the present work, the fabrication of the optical elements (matrices of refractive microlenses, transmitting diffraction gratings) on the base of  $As-S$  layers for the IR spectral region is considered.

### Results and Discussion

The bulk materials of  $As_{100-x}S_x$  ( $x = 71.4; 67; 62; 60; 58$ ) composition were prepared by the direct synthesis according to the conventional melt-quenched method from 5N purity elements. Arsenic and sulfur were weighed and placed in precleaned and outgassed (by heating under vacuum to  $900\ ^\circ\text{C}$ ) quartz ampoules. The synthesis was performed in evacuated (up to a pressure of  $1 \cdot 10^{-3}$  Pa) and sealed quartz ampoules using a rocking furnace at  $700 - 750\ ^\circ\text{C}$  for  $8 - 24$  h. After synthesis, the ampoules were quenched in cold water (temperature  $\sim 15\ ^\circ\text{C}$ , which is equivalent to a cooling rate of the order of  $10\ \text{Ks}^{-1}$ ). As-S films were prepared by the vacuum thermal evaporation ( $P = 10^{-3}$  Pa) from the quartz boats onto glass substrates. The film thickness varied from  $0.4$  up to  $5\ \mu\text{m}$ . The deposition rate was controlled with quartz sensors and consisted  $1.0 - 8.0\ \text{nm/s}$ , the uniformity being provided by the use of a planetary system.

A high level of photoinduced structural changes [7] provides good etching selectivity of unexposed and exposed  $As_{100-x}S_x$  layers in various nonaqueous amine-based solvents. Selective etching is one of the most important steps in the optical element fabrication process, which provides the creation of a relief with the necessary form and depth. The selective etching of As-S layers was carried out using the amine-based solutions. The etching rate of As-S layers in such solutions after exposure is decreased, that is, the case

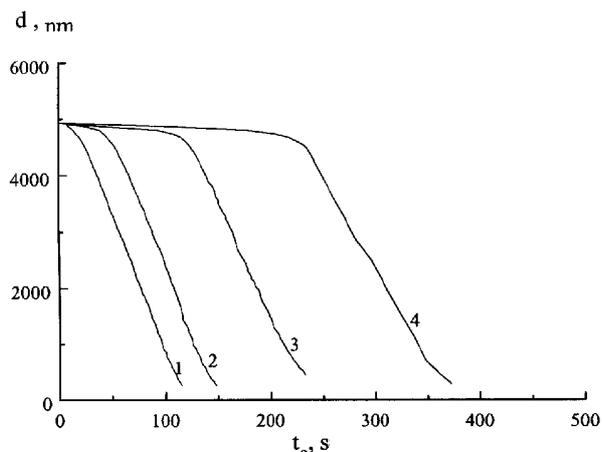


Fig. 1. Dependences of the thickness of  $As_{33}S_{67}$  layers  $d$  on the time of etching  $t_e$  in solutions of triethylamine in dimethylformamide (5 vol.%) for various exposure times. Exposure by a halogen lamp through an IR cutoff filter,  $P = 44$  mW. Exposure time, min: 1 - 0, 2 - 1, 3 - 2, 4 - 4

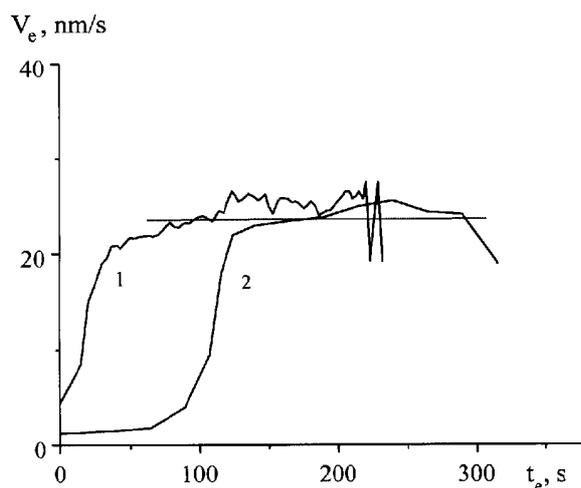


Fig. 2. Dependence of the etching rate  $V_e$  for as-evaporated (curve 1) and exposed (curve 2)  $As_{40}S_{60}$  layers in a solution of triethylamine in dimethylformamide (1 vol.%) on the etching time  $t_e$ . Initial thickness of  $As_{40}S_{60}$  layers  $\sim 5 \mu m$

of negative selective etching is realized. As can be seen from Figs. 1 and 2, such etching solutions provide a high selectivity of etching (the ratio of etching rates of as-evaporated and exposed films can be 10 or more, if necessary). The essential feature of the etching of ChVS layers with a sufficiently big thickness ( $\sim 2 - 5 \mu m$ ) is that the etching selectivity of the film is modified in the subsurface region, the depth of which is determined by the light penetration depth. Most significant photostructural changes occur under ChVS layer exposure in the upper part of this subsurface layer. Their level is characterized by exponential decay (in the absence of interference effects) while going

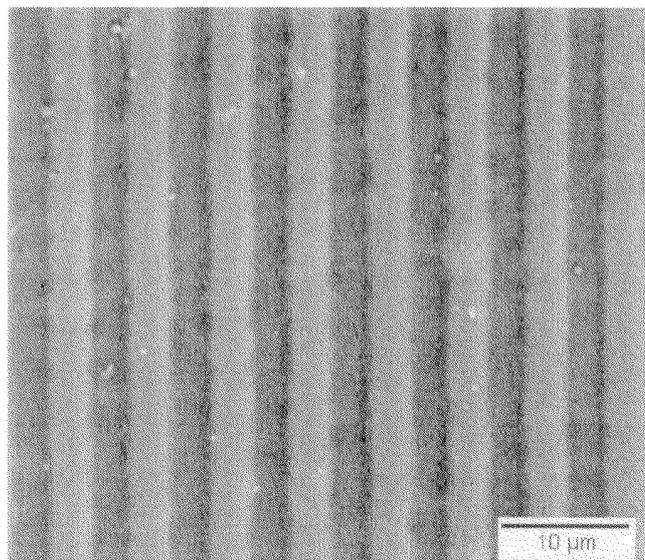


Fig. 3. Diffraction grating on the base of  $As_{33}S_{67}$  layers, obtained by contact printing through a mask. Period  $\sim 6 \mu m$

into the depth of the layer. When the part of the layer with modified selectivity is dissolved, the etching rate becomes near to that characteristic of the as-evaporated layer (Fig. 1). This circumstance should be taken into account in the case of the relief formation for the optical elements with small spatial frequencies and big relief depths ( $D \geq 1 \mu m$ ), because the formation of the necessary relief profile is difficult in this case, underetching occurs, an  $\Omega$ -type relief and other unoptimal forms can be formed, which does not provide the necessary operational characteristics of optical elements.

To obtain transmitting diffraction gratings with the spatial frequencies ranging from 6 up to  $12 \mu m$ , we used binary Cr masks with equal sizes of the open and closed (dark) zones. The samples were exposed through a mask by contact exposure (halogen or xenon lamps, incandescent or natural light). After the exposure, selective etching was carried out in above mentioned amine based solutions. The spectral distribution of the gratings was measured in the 400 - 2000 nm spectral interval with the help of an SPM-2 monochromator. For registration of the diffraction orders in transmission, Si and Ge photodiodes were used as detectors, which were placed at a movable holder and can be shifted along a circle (within the  $-90$  up to  $+90^\circ$  limits). A measured diffraction grating was placed in the center of the circle. The SEM image of a grating with the  $6 \mu m$  period fabricated by contact exposure on the base of  $As_{33}S_{67}$  layers is presented in Fig. 3. The values of diffraction efficiency for the first order of diffraction consisted  $\sim 23\%$ . For the

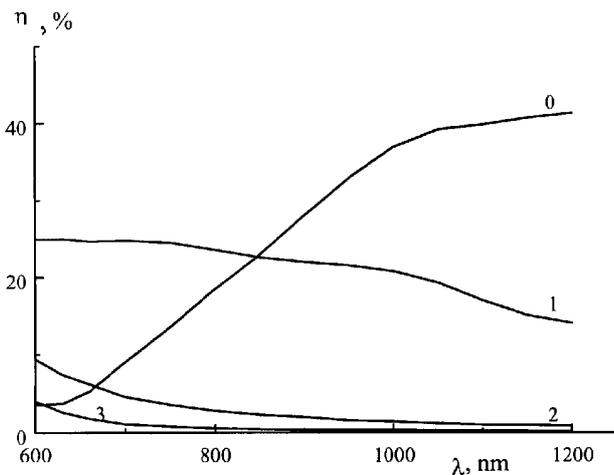


Fig. 4. Spectral distribution of the diffraction efficiency  $\eta$  for various diffraction orders for a grating with 6  $\mu\text{m}$  period on the base of  $\text{As}_{33}\text{S}_{67}$  layers, obtained by contact printing: 0 - zero; 1 - first; 2 - second; 3 - third order of diffraction

second and third orders, they were essentially lower and consisted 10 and 5%, respectively, in the 600 - 1200 nm interval (Fig. 4). The best values of diffraction efficiency for the first order of transmission diffraction gratings on the base of  $\text{As}_{100-x}\text{S}_x$  with periods from 6 up to 12  $\mu\text{m}$  obtained by us earlier using different compositions and etching solutions consisted 20 - 28 % [5 - 6].

Necessary and functionally important elements of integrated optoelectronics devices are matrices of refractive or diffractive microlenses. High transparency of ChVS in the IR region determines their high potential as a material of optical elements in this spectral region. Matrices of refractive microlenses were obtained by us on the base of  $\text{As}_{100-x}\text{S}_x$  layers with the help of halftone masks and contact printing (Fig. 5, 6). The use of the halftone or diffractive masks enables to obtain the necessary light intensity distribution and exposure profile in the registering media for only one exposure and selective etching, which enables to increase the quality of element reliefs, decrease their production costs, and avoid the multistep exposure procedure which is used if the set of binary masks is used. The diameter of a single lens consisted 12  $\mu\text{m}$ . To improve the quality and characteristics of elements, it is necessary to carry out optimization procedures for the exposure conditions and selective etching and to carry on them under conditions corresponding to those existing in the microelectronic industry. The use of photodoping [3] enables to increase the refractive index of media and to decrease, at the same geometric parameters, the focal length value of a single lens.

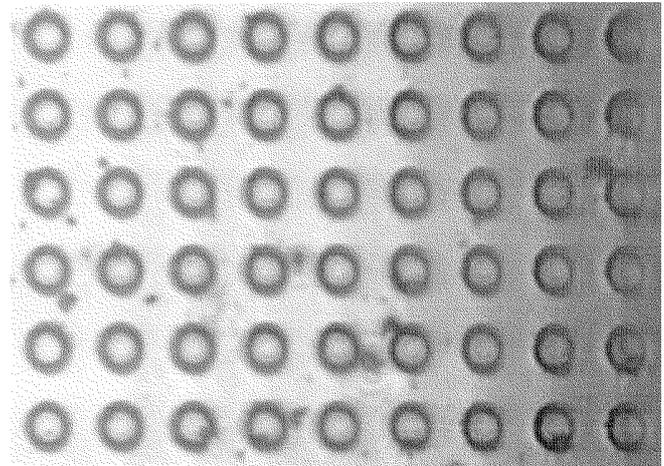


Fig. 5. Microlens matrix, obtained on the base of  $\text{As}_{33}\text{S}_{67}$  by contact printing with the use of a halftone mask. Lens diameter  $\sim 12 \mu\text{m}$

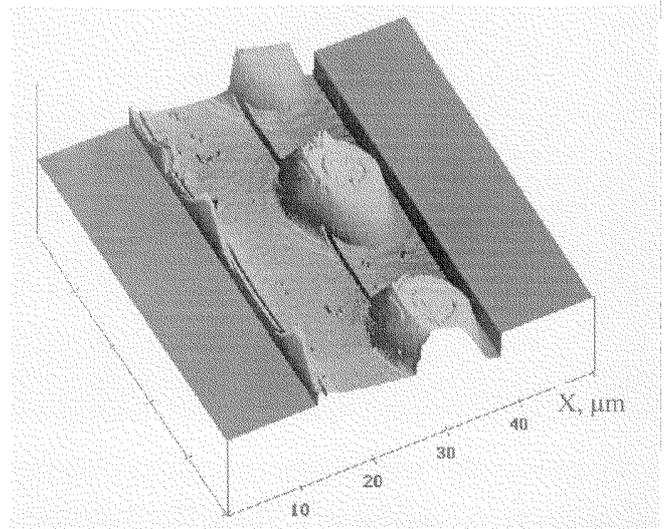


Fig. 6. AFM image of a fragment of a microlens matrix obtained on the base of  $\text{As}_{33}\text{S}_{67}$  layers

## Conclusion

The investigations carried out have shown that  $\text{As}^- \text{S}$  layers can be considered as perspective relief-forming media for the fabrication of various optical elements (diffraction gratings, matrices of microlenses, etc.) with high operational characteristics in the IR spectral region.

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