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TECHNIQUE FOR PRODUCTION OF HOLOGRAPHIC DIFFRACTION GRATINGS BASED ON INORGANIC VACUUM PHOTORESISTS

An innovative project has been implemented to develop a technique for manufacturing the holographic diffraction gratings, which allows the engineers to produce high-quality diffractive elements with spatial frequencies from 600 to 3600 mm^{-1} for spectral devices. The guidelines for the implementation of this method have been elaborated and the pilot samples have been produced. The characteristics of pilot samples of holographic diffraction gratings manufactured under this project have been established to meet the specifications and the government standard 3-6128-86.

Key words: diffraction grating, chalcogenide photoresists, and spectral devices.

For many years, the holographic diffraction gratings have been widely used as dispersing elements in different spectral devices for physical, astronomical, chemical, biological, and other studies in various industries (metallurgy, food, chemical, and engineering etc.), as well as for the purposes of medicine and environmental protection. In addition, the diffraction gratings are used to enhance the capacity of fiber-optic networks in integrated optical devices and lasers with variable frequencies. Unfortunately, neither diffraction gratings nor spectral devices based on them have been produced in Ukraine. However, there is an urgent need for such devices, in particular, for those applied to biomedical research and analysis (especially, for diagnosing infectious diseases at early stages), spectral analysis in the steel industry, environmental research and analysis, and so on.

The main objective of the project was to develop an innovative technique for manufacturing holographic diffraction gratings in a wide range of spatial frequencies using the vacuum photoresists based on layers of chalcogenide glasses. These photoresists are developed and patented by the V.Ye. Lashkarev Institute of Semiconductor Physics of NASU and have a number of unique characteristics, including a resolution of 1 nm. As compared with existing analogues the above mentioned chalcogenide photoresist is characterized by thermal stability (300 °C), no shrinkage effect during post-exposure baking, good mechanical strength and chemical resistance. Chalcogenide photoresists can be applied with the help of thermal deposition in vacuum both on the flat substrates and on the products of complex shape. Therefore, the photoresists are very promising for recording the holographic diffraction elements, including the diffraction gratings.

The development of processes for manufacturing the holographic gratings includes the development and installation of optical circuits, optimization of vacuum deposition of chalcogenide photoresist, exposure, post-exposure baking, and application of reflective and protective coatings. To optimize the parameters and modes of these processes, in addition to experimental studies, it is necessary to theoretically calculate and numerically simulate the formation of grating profile which provide the required spectral distribution of diffraction efficiency.

DEVELOPMENT OF EQUIPMENT AND OPTICAL CIRCUITS FOR RECORDING OF HOLOGRAPHIC GRATINGS AND OPTIMIZATION OF CHALCOGENIDE PHOTORESIST VACUUM APPLICATION

The interference pattern of holographic optical elements, including diffraction gratings, can be made only if the optical circuit is stable during recording. To ensure the thermal and mechanical stability the optical circuits were mounted in the basement on antivibration concrete slabs weighing 1.5 tons, which, in their turn, were placed on the massive foundations (having a mass of 40 tons) isolated from the building foundation and installed on damping pads. The basement is equipped with a ventilation system and meets all engineering requirements.

The optical circuit for holographic recording is showed in Fig. 1. The record is as follows. The laser beam using a spatial filter and a spherical mirror is transformed into an expanded beam with a flat wave front. Then a beam of light is directed to the divider, which is divided into two parts, the power at a ratio of about 1 : 1. Both parts of the beam using flat mirrors are sent on a plate with photoresist on the surface which is the result of superposition of coherent light fronts formed by the interference field.

Fig. 2 shows the optical circuit mounted on antivibration slab in a room with controlled temperature and humidity. The circuit is based on an argon laser LH-503 with an operating wavelength of 514.5 nm. The laser spectral range and output power (1 W) are optimal if inorganic photoresists based on chalcogenide glasses are used as a recording medium.

The main method for chalcogenide photoresist application is thermal evaporation in vacuum. It is a known fact that their resist properties (type of solubility, etch rate selectivity) depend not only on the composition of chalcogenide and etchant, but also on the conditions of deposition (substrate temperature and deposition rate of chalcogenide film on it) [1—3]. In order to reproduce the properties and characteristics of photoresist layers the layers are apply under the following conditions:

- The chalcogenide is evaporated at a residual pressure in the vacuum chamber of at least 10^{-3} Pa;
- The substrate temperature during the deposition of chalcogenide layer is equal to the ambient temperature;
- The deposition rate ranges within 1—2 nm/s.

The layer thickness and rate of deposition were controlled during the deposition by quartz thickness meter (QTM-1) or by optical interferometry

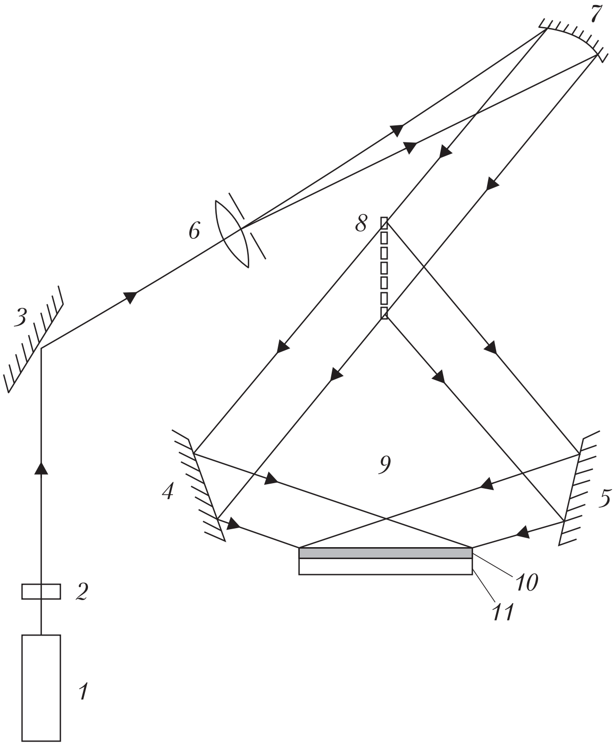

Fig. 1. Optical circuit for holographic recording: *1* – laser, *2* – gate, *3, 4, 5* – flat mirrors, *6* – spatial filter, *7* – spherical mirror, *8* – light beam divider, *9* – interference field, *10* – photoresist layer, and *11* – substrate

Fig. 2. Optical circuit for holographic recording mounted on antivibration foundation

method. The measurement of absolute thickness of deposited layers and control of their homogeneity were conducted using MIM-4 micro-interferometer, *Talystep* profile meter, and LEF-3M ellipsometer. Equipment for applying the chalcogenide photoresist on glass plates by evaporation in vacuum has been designed and manufactured.

In order to optimize the deposition of chalcogenides in vacuum the photoresist thickness distribution on the plate has been simulated with the use of computer. Using the simulation results and optimized modes of thermal evaporation the chalcogenide photoresist layers having a thickness from 200 to 600 nm have been deposited on the plates for holographic recording with a working area of 50×50 mm, with inhomogeneity of thickness less than 5%.

The thickness of chalcogenide photoresists was optimized for recording the gratings with different spatial frequencies allowing for the etchant selectivity. To ensure good adhesion of the photoresist film and to prevent multiple reflection of light from the back surface of the substrate a chromium layer having a thickness of 30–80 nm was deposited before applying the chalcogenide layer.

The developed technological process includes four elements: *the vacuum* (deposition of functional layers), *the holographic* (recording of interference pattern), *the chemical* (washing and selective etching), and *the optical* (control of characteristics) sections. In addition, the morphology of the grating surface relief is controlled using an atomic force microscope.

When implementing the project a plant for measuring the spectral dependence and angular distribution of diffraction efficiency of holographic gratings in the visible spectrum was designed and produced. The diffraction efficiency η is defined as ratio of light intensity I_1 diffracted in a given order of diffraction to light intensity I_0 falling on the structure, i.e. $\eta = 100\%\ I_1/I_0$. The effectiveness was measured for a configuration very similar to the Littrow configuration. The angle between the incident and the diffracted beams ranged from 7 to 10°.

OPTIMIZATION OF EXPOSURE, POST-EXPOSURE BAKING, AND APPLICATION OF REFLECTIVE AND PROTECTIVE COATINGS

An important step in the development of holographic recording technique is to optimize the conditions of exposure and processing. During the exposure the chalcogenide photoresists undergo photo-induced structural transformations of light-sensitive layer. As a result, the optical and chemical properties (dissolution rate in selective etchants) in the exposed areas change [1—4]. The level of these transformations is determined by light energy absorbed in local volume of chalcogenide film. Therefore, a 3D intensity distribution of light field during interferential exposure of such thin-film structure was calculated to correctly describe the process of exposure. The value of exposure is defined as exposure intensity multiplied by exposure time. The energy absorbed at a given point of photoresist layer is proportional to the product of exposure at a given point by absorption coefficient. It determines the degree of photo-induced transformations in the photoresist layer and, consequently, the change in its solubility in selective etchants. In the case of exposure to monochromatic radiation the absorption coefficient is constant, with the change in photoresist solubility depending on light intensity distribution.

Thus, the rate of chalcogenide selective etching after interference exposure is determined mainly by two factors: the distribution of light intensity in the layer and the dependence of rate of chalcogenide photoresist dissolution in etchant on the exposure. This dependence for each photoresist is measured experimentally [5]. The obtained experimental and theoretical dependences (distribution of light intensity during exposure of thin-film structure and dissolution rate of photoresist) were used for numerical simulation of forming a diffraction grating on chalcogenide photoresist layers. The simulation is based on the assumption that in the course of etching each point of contour describing the profile of grating relief structure shifts along the normal line to the surface at a rate that is proportional to the rate of etching, with the shape of contour and, consequently, the directions of normal lines changing continuously.

Using the described algorithm a software package has been developed for the numerical simulation of chalcogenide photoresist selective etching after interferential exposure, i.e. the formation of holographic diffraction gratings. Using the numerical simulation one can observe the formation of relief and determine on which factors (exposure, time of etching, selectivity) the obtained grating parameters mainly depend.

These numerical calculations were carried out for chalcogenide photoresists based on the following glass composition: $As_{40}S_{60}$, $As_{40}Se_{60}$, $As_{40}S_{20}Se_{40}$, $As_{40}S_{40}Se_{20}$, and $Ge_{25}Se_{75}$. The patterns obtained were used for optimizing the technological processes of manufacturing the holographic diffraction gratings based on chalcogenide photoresists [6—13].

On the basis of computer simulation results the composition of selective etchants was chosen to obtain the necessary parameters of micro-relief (during etching), exposure, and etching. Depending on the technological conditions of deposition (substrate temperature, deposition rate) and post-exposure baking (etchant compounding, temperature of annealing of chalcogenide layer) the resist chalcogenide layers can show both negative and positive photo-induced changes in solubility. When choosing the etchants for resist chalcogenide layers the main criteria was sufficiently high selectivity and good quality of etched surface. In general, they are not interrelated, with their priority depending on specific practical application. For example, when using a chalcogenide layer to form a protective mask in optical micro-lithography (production of microelectronic devices) the etchant selectivity plays an important role inasmuch as it determines the thickness of resist layer remaining on the substrate after etching. For laser and holographic lithography (making of original optical track record or marking structure of holographic diffraction gratings, integrated optics elements, etc.) the quality of resist layer surface after etching is of paramount importance in-

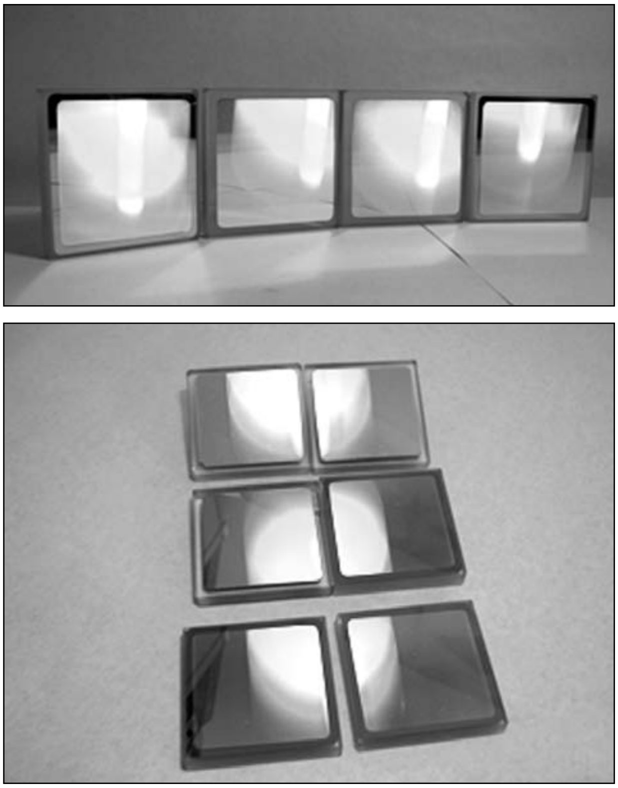

Fig. 3. Pilot samples of diffraction gratings

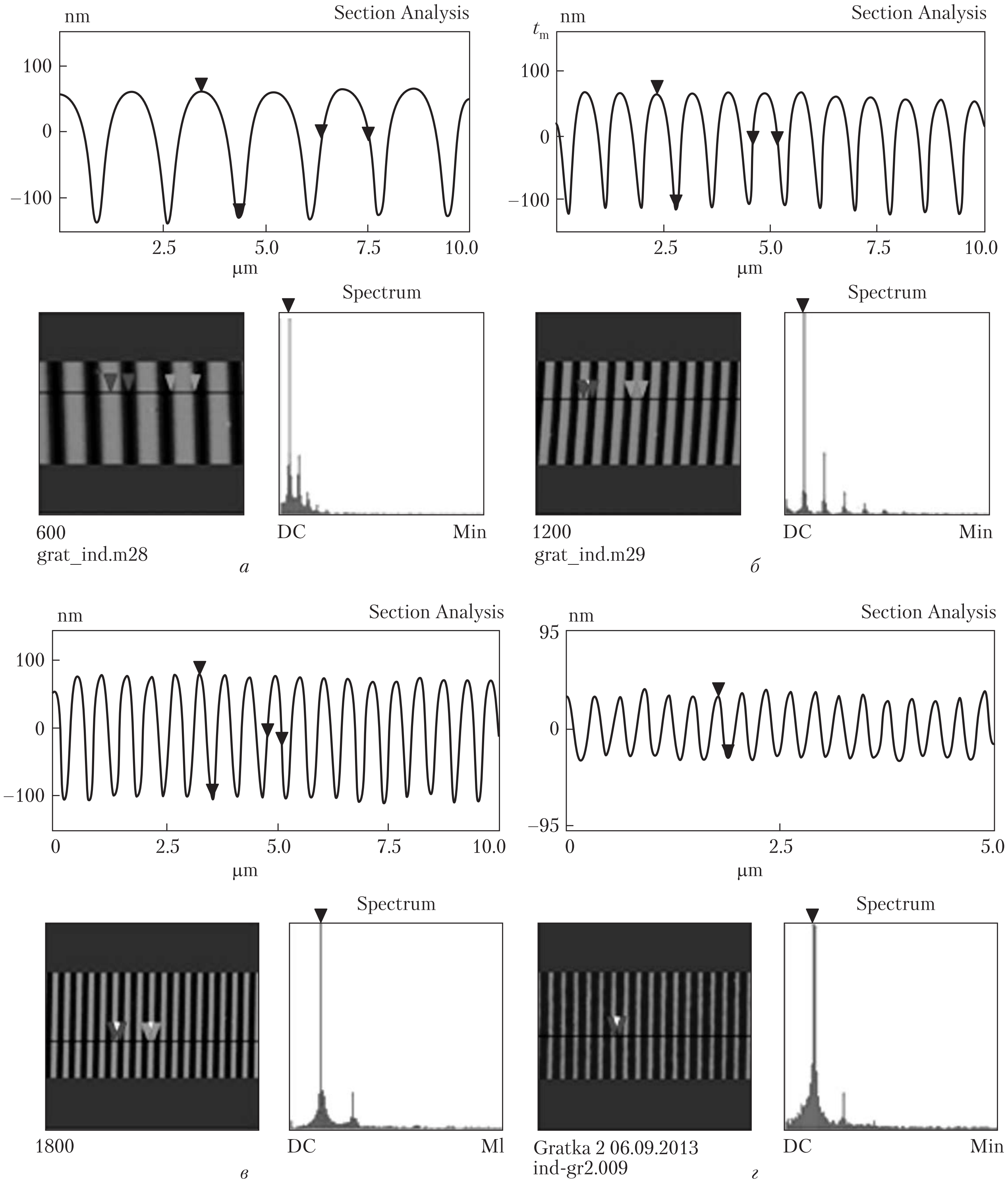

Fig. 4. AFM image of the profile shape of grooves of holographic gratings with periods: 1667 nm (*a*), 833 nm (*b*), 555 nm (*c*), and 278 nm (*d*)

asmuch as it defines the performance of products (signal/noise and scattered light ratios).

In previous research of negative etching of resist chalcogenide layers the solutions of inorganic and organic reagents were used [2, 14, and 15]. Among the typical shortcomings of the former there are low selectivity and heterogeneity of etching and the presence of alkali metal ions in them, as a result of which they cannot be used in the manufacture of microelectronic devices. The etchants based on organic reagents, including ammonia and its derivatives (amines) are more suitable. They give high selectivity and homogeneity of etching. To a large extent, the action of these etchants is caused by organic solvent reagent which not only significantly affects the selectivity, but also can lead to inversion of photo-induced changes in the solubility of the same chalcogenide. For example, for $As_{40}S_{60}$ layers the solutions of dimethylamine in water and isopropyl alcohol [16] are positive etchants, while the solutions in dimethyl sulfoxide or benzonitrile [17] are negative etchants. On the basis of study of photo-induced changes in solubility of resist layers of $As_{40}S_{60-x}Se_x$, where $x = 0; 20; 30; 40$, two-component selective etchants of negative type have been developed. For binary composition $As_{40}S_{60}$ this etchant is ammonia hydroxide solution in dimethylketon. For the layers of triple chalcogenide structure $As_{40}S_{60-x}Se_x$, where $x = 20; 30; 40$, the most suitable solutions are those containing ethylenediamine and dimethylketon.

Within the framework of this project, the etchants based on butylamine, propylamine, diethylamine, dimethylamine, ethylenediamine and others have been studied. The experiments have showed that the best parameters (selectivity of 10–20, linear dependence of relief height on exposure, homogeneity of treatment, etc.) are given by an etchant based on ethylenediamine ($NH_2CH_2CH_2NH_2$) solved in dimethyl sulfoxide.

The optimal time of selective etching is chosen experimentally allowing for the results of computer simulation and the conditions for maximum diffraction efficiency η. The dependence of η on time of treatment is a curve with maximum. This is caused by the fact that the optimal selectivity is not an absolute value and the ratio of dissolution rates of exposed and unexposed areas of XC layer is 10–20. In the course of etching the value of η was controlled by irradiating with non-photoactive light of helium-neon laser (wavelength of 632.5 nm). As the value of η reaches its maximum the etching of sample stops.

The optimum exposure value or time of exposure to radiation at a constant intensity is also chosen experimentally for specific parameters of light-sensitive layers based on the conditions of maximum diffraction efficiency η. The maximum η is achieved with such exposure when in the areas corresponding to the maxima of interference pattern the photo-structural transformations approach saturation. Insofar as the optical circuits forming the interference pattern are not perfect (the modulation of interference pattern through the thickness of photoresist layer is not 100%) after reaching the saturation at the maxima the diffraction efficiency decreases.

The reflective and protective coatings are also applied using thermal deposition in vacuum. The aluminum layers having a thickness of 50–150 nm are used as reflective coating depending on the spatial frequency and depth of modulation of the grating. In some cases, for the gratings operating in the infrared region, golden layers can be used as reflective coating.

For the gratings working in wet or other adverse environment the application of protective coatings is required. Within this project a manufacturing process of vacuum deposition of SiO_x and SiO_2 as protective layers for holographic gratings has been developed. These layers are transparent within the working spectral range, waterproof, and chemically stable. The application of these layers by using thermal evaporation of granular SiO or powdered SiO_2 in vacuum from tantalum evaporators. When depositing SiO, as a result of full oxidation with residual gases, a film of SiO_x, where $x > 1$, is obtained. The rate of deposition is chosen so that the value of x is equal

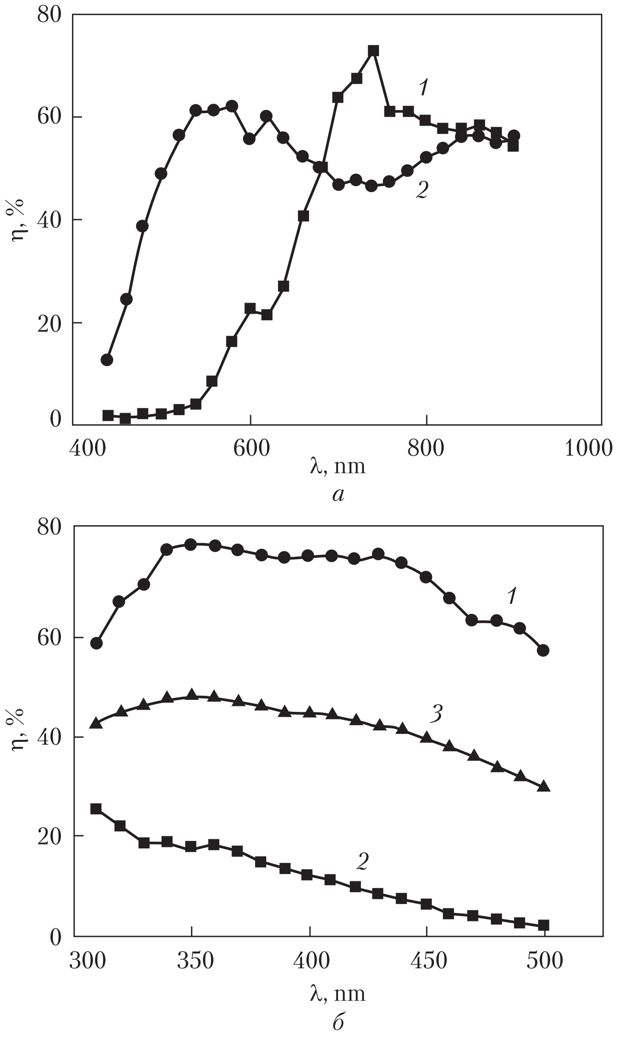

Fig. 5. Spectral distribution of diffraction efficiency of the gratings with spatial frequencies of 1200 (*a*) and 3600 mm^{-1} (*b*) formed on the $As_{40}Se_{60}$ film: curves 1 and 2 correspond to the perpendicular and the parallel alignment of light wave electric vector with respect to the grooves; curve 3 is for the unpolarized light

to, at least, 1.5, which is necessary to ensure transparency in the visible spectrum.

MANUFACTURE OF PILOT SAMPLES AND STUDY OF THEIR CHARACTERISTICS

Using the developed and optimized manufacturing processes, pilot samples of holographic diffraction gratings with spatial frequencies of 600, 1200, 1800, 3600 mm^{-1} and working area of 50×50 mm^2 were made in accordance with the terms of reference (see Fig. 3).

To study the profile shape of holographic grating grooves a *Dimension 3000 Scanning Probe Microscope* AFM by *Digital Instruments* was used.

Figs. 4 (*a-d*) show the images of cross sections of pilot grating samples with spatial frequencies specified in the terms of reference and ranging from 600 to 3700 mm^{-1}. The images were obtained using the atomic force microscope. The samples were prepared by sequential thermal evaporation in vacuum at a pressure of 2×10^{-3} Pa on the substrates of adhesive Cr layer, as well as $As_{40}Se_{60}$ (*a*, *b*), and $Ge_{25}Se_{75}$ (*c, d*) layers. The 10 mm thick polished glass plates were used as substrates. The layer thicknesses were controlled during deposition using a quartz thickness meter (QTM-1). They were equal to 30 nm for Cr and 300–800 nm (depending on the spatial frequency of the grating) for the photoresist.

Having been exposed to periodic light generated by a laser (LGN-503 with a wavelength of 476.5 nm) with spatial frequencies of 600–3600 mm^{-1}, the samples were treated with a selective etchant. This process lasted for 1.5–2 minutes at a temperature of 22 °C. The resulting relief-phase diffraction gratings were covered with a reflective layer of Al having a thickness of 50–80 nm (the sample with a spatial frequency of 3600 mm^{-1} was covered with a 50 nm thick layer, while the rest was covered with a layer of 80 nm).

Figure 4 shows that the shape of the groove profiles is close to a sine wave (or cycloid) depending on spatial frequencies and modes of exposure and treatment. The modulation depth (i.e. the ratio of the groove depth to the grating period) is 0.2–0.3, which is optimal for obtaining high diffraction efficiency and low level of scattered light.

The project also included the study of diffraction efficiency of pilot gratings. The angular $\eta(\varphi)$ (φ is angle of beam incidence on the grating) and the spectral $\eta(\lambda)$ (λ is beam's wavelength) dependence of diffraction efficiency of the obtained gratings were measured for two directions (*p* and *s*) of incident light polarization. The spectral measurements were performed for the first-order diffraction within the range of 310–900 nm.

Figure 5 shows the spectra of diffraction efficiency of the gratings with spatial frequencies of 1200 (*a*) and 3600 mm^{-1} (*b*) formed on the $As_{40}Se_{60}$ film. Curves 1 and 2 correspond to the perpendicular and the parallel alignments of light wave electric vector with respect to the grooves. As one can see from the figure, the diffraction efficiency for the polarized light reaches 80–85%, which indicates a sufficient depth of the modulation. For the unpolarized light it accounts for 40–60% in the working area of the spectrum, which corresponds to the terms of reference and GOST 3-6128-86.

The level of scattered light was measured for the pilot samples of holographic diffraction gratings. The study was performed using a helium-neon laser ($\lambda = 632.8$ nm); the circuit also included a goniometer, a modulator, and a receiver unit with an amplifier. For the samples studied the scattered light level at an angular distance of 1° from the laser beam was measured to range within 2×10^{-6}—4×10^{-6}, which corresponded to the TOR and GOST 3-6128-86.

The radiation strength of holographic gratings was determined by measuring the laser power density at which the sample was destroyed (visible traces of the laser beam were observed on the surface of the grating). The optical circuit included a single-mode solid laser with a Q-factor modulator, a dividing wedge, an optical wedge, a focusing lens and an IMO-2M energy meter. The study was conducted at two wavelengths: 1.06 and 0.53 microns. As a result of the study, the radial strength of the samples was established to range within 2–4 MW/cm^2.

The pilot samples were tested with respect to the effect of high and low temperature and high humidity. Neither visually noticeable defects nor deterioration of spectral characteristics of pilot holographic gratings were observed.

CONCLUSIONS

1. An optical circuit holographic recording has been designed and assembled, which makes it possible to obtain the samples with spatial frequencies from 600 to 3600 mm^{-1}.

2. Using the theoretical and numerical simulation and the experimental results the application of photoresist chalcogenide layer, the interferential exposure, the selective etching, and the deposition of reflective and protective coatings have been optimized.

3. The main advantages of the developed technique as compared with the existing ones is that it makes it possible to use the photoresists based on germanium chalcogenides which are more environmental friendly as compared with the conventional chalcogenide photoresists based on arsenic compounds. In addition, the diffraction gratings can be manufactured on the annealed chalcogenide layers, which reduces the surface roughness after selective etching.

4. On the basis of optimization results the guidelines for manufacturing the holographic diffraction gratings using inorganic chalcogenide photoresists have been developed.

5. The pilot samples of holographic diffraction gratings have been manufactured; their characteristics, including the shape of groove profiles, the spectral and angular dependences of diffraction efficiency, the level of scattered light and radial strength have been studied.

6. The characteristics of pilot samples of holographic diffraction gratings have been established to meet the specifications and GOST 3-6128-86.

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ТЕХНОЛОГІЯ ВИРОБНИЦТВА ГОЛОГРАМНИХ ДИФРАКЦІЙНИХ ҐРАТОК НА ОСНОВІ НЕОРГАНІЧНИХ ВАКУУМНИХ ФОТОРЕЗИСТІВ

Виконано інноваційний проект з розробки технологічного методу формування голограмних дифракційних ґраток, який дає можливість виготовляти високоякісні дифракційні елементи з просторовими частотами від 600 до 3600 мм$^{-1}$ для спектральних приладів. Розроблено технологічні інструкції по реалізації цього методу та виготовлено експериментальні зразки. Встановлено, що характеристики виготовлених в рамках даного проекту експериментальних зразків голограмних дифракційних ґраток відповідають технічному завданню та ДСТ 3-6128-86.

Ключові слова: дифракційні ґратки, халькогенідні фоторезисти, спектральні прилади.

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ТЕХНОЛОГИЯ ПРОИЗВОДСТВА ГОЛОГРАММНЫХ ДИФРАКЦИОННЫХ РЕШЕТОК НА ОСНОВЕ НЕОРГАНИЧЕСКИХ ВАКУУМНЫХ ФОТОРЕЗИСТОВ

Выполнен инновационный проект по разработке технологического метода формирования голограммных дифракционных решеток, позволяющий изготавливать высококачественные дифракционные элементы с пространственными частотами от 600 до 3600 мм$^{-1}$ для спектральных приборов. Разработаны технологические инструкции по реализации этого метода и изготовлены экспериментальные образцы. Установлено, что характеристики изготовленных в рамках данного проекта экспериментальных образцов голограммных дифракционных решеток соответствуют техническому заданию и ГСТ 3-6128-86.

Ключевые слова: дифракционные решетки, халькогенидные фоторезисты, спектральные приборы.

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