Polarization holography for direct surface grating patterning on chalcogenide nanomultilayers

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Abstract. In present work the one-step direct relief formation of diffraction gratings using As$_2$S$_3$–Se nanomultilayers were studied. We have carried out a study of the polarization sensitive recording properties of the chalcogenide glasses in form of nanomultilayers. Nanomultilayers deposition, samples structure and recording process peculiarities were discussed. The surface relief gratings were patterned into nanomultilayers by method of polarization holography. It was shown that surface relief vector holographic gratings formed by two ±45°polarization states and by left-right circular polarizations are phase gratings. The kinetics of diffraction efficiency of both phase and intensity gratings were compared. The possible explanations based on dielectrophoretic forces existing in fluidic systems or H. Fritzche’s model supposing the presence of minimum isotropic volume with the anisotropic structural units are discussed. The method offers the advantage of the fabrication of relief structures with one-step processing, without the necessity for any subsequent processing steps.

1. Introduction

In contrast to the scalar holographic process, in which intensity variations in an interference pattern between identically polarized an object and a reference beams are recorded, polarization holography employs beams with two different polarizations states for hologram recording. In this case, the phase difference between the interfering waves leads to changes only in the polarization state of the resulting light field, i.e., intensity modulation is absent and not visible on photosensitive media. Nevertheless, resulting field of standing waves with spatially variable polarization state can be recorded as hologram.

Use of linearly polarized light results in photo-induced anisotropy in initially isotropic glasses [1, 2] and the effects induced by polarized light are often called vectorial effects.

Vector holograms are holograms recorded by two arbitrarily oriented polarized light waves containing orthogonal components. These arbitrarily polarized light waves create a mixed spatial light field modulation consisting of the polarization modulation produced by the orthogonally polarized components and of the intensity modulation produced by the parallel polarized components [3]. The polarization modulation is obligatory for vector holograms but the intensity modulation which is necessary for scalar hologram recording can be absent. As a result the complex dielectric permeability tensor $\varepsilon_{mn}(\omega, k)$ of the recording material depending on light frequency and wave vector becomes spatially modulated.

The idea to record vector hologram was first expressed by Sh. D. Kakitschashvili in 1972 [4]. He was also the first to realize this idea in 1975 by recording the first vector holograms in AgCl glass. In 1978 Sh. D. Kakitschashvili published the theory of thin vector holograms recorded by polarization
anisotropy. It is based on the generalized Fresnel-Kirchhoff diffraction integral using the formalism of Jones vectors and matrices [5]. He has developed also the thick vector hologram theory but only in the kinematic approximation, i.e., for small diffraction efficiency (DE). This rather complicated theory is still most general because even the development of thick vector grating coupled wave theory like H. Kogelnik’s theory has proven to be too difficult. In 1992 L. Nikolova et al [6] have given the theory of thin amplitude-phase vector gratings recorded by circularly polarized waves.

Conventional recording materials are sensitive only to light intensity. Exposed to such an interference pattern they would be darkened/bleached or change surface topology under volume shrinking/expansion. In order to make use of the polarization modulation it is necessary to use a recording material that has a different response when exposed to light beams with different polarizations and can record the information about the polarization states of light [7]. The earlier works mainly belongs to such materials as polymer films where relations of surface relief and polarization (vector) hologram recording were examined.

Chalcogenide glasses (ChG) are a class of amorphous semiconductors which meet the polarization sensitive requirements too. Amorphous chalcogenides are intrinsically metastable and as such can be easily modified by external actions, by light in particular. The effect of photoinduced structural changes is mainly used for holographic recording in ChG. The theoretical value of DE for vector holograms can be as high as 100%.

Polarization diffraction gratings were shown to be useful elements for the fabrication of polarization-discrimination systems. Indeed, from theoretical considerations [8] it follows that pure polarization gratings can be used for the measurement of Stokes parameters. Now gratings are considered as an essential constituent of a spectrophotopolarimeter. Such holographic elements show less sensitive to angular change of the incident beam and more compact compared with the conventional polarizing systems based, for example, on the birefringence optical crystals.

Holographic recording by polarization states modulation yields the vector hologram as addition to traditional holography which results only in amplitude and phase modulations of recording media. Thus it is considerably increases the optical storage memory capacity. As a result vector diffraction optical elements can perform a full transformation of light wavefronts including their amplitude, phase, wavelength and polarization. In point of fact, polarization anisotropy induced by the vector holographic grating recording is on the one hand a tool for the investigation of glass optical anisotropy on the other hand open way to new practical applications. This is our motivations to study polarization holographic recording of diffraction gratings in ChG.

In spite of the wide variety of applications currently mastered using ChG, this state of matter still represents a complex system with many surprising properties. An important feature of the photoinduced changes in ChG is their high resolution with the presently experimentally recorded limit on the order of a few dozens of nanometers. All the above mentioned processes open up a possibility of ChG application for optical information recording. The various attempts to this end have been and are being made, some of them very successful.

However, many intriguing phenomena in these glasses are not well understood at the microscopic level. For example, there is still a wide divergence of opinions concerning the nature of their sensitivity to the intensity and polarization state of light. Again the understanding of the microscopic mechanisms that are at the origin of the optical properties of chalcogenide glasses thus represents an important scientific and technological challenge.

It should be noticed that the nanometre scale mechanism of light induced changes has long remained unclear and it was not until very recently that in-situ structural studies performed on amorphous selenium unveiled the possible nanometre scale mechanism of reversible photostructural changes.

The importance of recording light polarization state was ascertained as well mass transport role for the process of holographic recording. At the same time, the presence of surface relief formation for As, S and Se contained amorphous chalcogenide films explained by photo-structural transformations are subject of permanent interest. For micro- and nano-relief formation in the ChG films after scalar holographic recording, selective etching is mostly used. The wet etching process requires very strict controlling of many parameters (sophisticated chemical composition, temperature, concentration, etc.).
In addition, often some components of etchant are very toxic. Optical quality of film is difficult to attain by wet processing after recording. Porosity of the film reveals during etching and spoils optical quality of surface. From technological point of view, it is better to have methods to obtain surface relief in one recording step and only by laser irradiation. So the development of methods for one step recording is considered as perspective for practical implementation of planar diffraction optical elements. The transfer of relief pattern from the ChG film onto the polymer film (Al coated) may be done through soft embossing after metal galvanoplastics. In present work research and development of methods and means, based on non-etching surface relief processes, for creating diffraction elements are presented.

The majority of studies of polarization recording in ChG dealt with one-layer chalcogenide glass thin films. In our studies we devote attention to nanomultilayers structures possessing some new optical properties.

Multilayer ChG structures are simplest artificial nanostructures that can be fabricated with controlled geometrical parameters and investigated as thin films. In general, glasses are promising materials for nanostructuring by some reasons.

The study of nanostructures properties could give the information about a glassy structure. Unlike crystalline materials, in which we can prepare atomically controlled surfaces, the amorphous structure is disordered at the atomic level.

The glass nanostructure may yield wider varieties than the crystalline one because bonding constraints of crystals do not exist in glasses. In addition to structural variety the glasses allow us to use diversity compositions to formation nanostructures sensitive to appropriate energy of laser quants coinciding with optical gap of glass. It is need to emphasize the importance of absence any treatment of recording grating.

The surface relief gratings (SRG) patterned on nanomultilayers (NML) As$_2$S$_3$–Se by one-step polarization holographic recording were investigated and compare with amplitude holographic recording. We studied how the polarization states of recording beams and the period of recorded gratings influence on the diffraction efficiency of them.

2. Experiment
NML of ChG were prepared by computer controlled cyclic thermal vacuum successive deposition of Se and As$_2$S$_3$ from two separated boats on continuously rotated glass substrate at room temperature in one vacuum deposition cycle. Deposition of the materials on the polished glass substrate was implemented through two separated windows over boats. The technology allows a films deposition separate control within the whole sample thicknesses in the range from 0.005 up to 3.0µm. The monitoring and determining of the total thicknesses of films of two constituent compositions of NML were carried out during the evaporation by 2 interference sensors at wavelength $\lambda$ = 0.95µm in transmission mode. The thickness of each nanomonolayer was calculated dividing the total measured thickness on a number of complete substrate revolutions. The thicknesses of one layer $d_{As2S3}$ and $d_{Se}$, consequently.

To prevent the crystallization of Se layers, which are rather structurally unstable under heating and/or illumination, we minimized the heating of layers during the deposition by virtue of substrate
rotation and reduced boat temperature. The mask application with windows and sample rotation reduce reciprocal diffusion of NML components which is possible during heating.

Figure 1. (a) The photograph of NML As$_2$S$_3$–Se sample on polished glass substrate (75x75mm$^2$) with the number of nanolayers 100+100; (b) Radial fragment of the cross-section of 4 As$_2$S$_3$ and 4 Se nanolayers formed of nanostructure on the substrate is shown (as an example).

The structural stability at interfaces and in the nanomonolayer is critical for any superlattice including NML. The direct determination of periodicity and roughness of interfaces of NML As$_2$S$_3$–Se deposited by the cyclic thermal evaporation was performed in [9] by Low Angle X-ray Diffraction where the author has shown the good interfaces quality and confirmed the amorphous nature of the NML.

An interferometric method of polarization holographic recording was used to obtain grating on the NML As$_2$S$_3$–Se like described in [10]. The holographic grating (HG) formation experiments were performed using different polarization states of recording beams (Fig.2). The modulation period of the grating $\Lambda=\lambda/(2\sin \alpha)$, where $\lambda$ is the wavelength of laser beam, $\alpha$ is the angle between the incidence laser beams. CW DPSS single mode laser operated at wavelength $\lambda=532$nm and averaged spot power density from 150 up to 350mW/cm$^2$ on the sample was used for recording. The intensity ratio 1:1 of the recording beams was adjusted in order to achieve maximum interference fringes contrast. The holographic gratings with various periods $\Lambda$ were recorded by two symmetrical angled laser beams respecting to the sample surface normal. The angle between the two interfering beams could be set at values providing spatial modulation periods between 1 $\mu$m and 3.3 $\mu$m. The sample is mounted on rotation stage for possibilities recording a number of gratings on the same sample radius (see inset on Fig.2) thereby ensure the equal samples properties.

We distinguish between two forms of recording: scalar process (intensity holography), resulting in intensity gratings and polarization holography (with a periodic variation of the polarization), resulting in polarization gratings recording.

Diffraction gratings in the NML As$_2$S$_3$–Se were recorded in the following beams polarization configurations:

- SS, when two beams were linearly polarized with electric field vectors perpendicular to the incidence plane;
- PP, when two beams were linearly polarized with electric field vectors parallel to the incidence plane;
- $\pm45^\circ$, when two beams were orthogonally linearly polarized at $\pm45^\circ$ with respect to the incidence plane;
- RL, when two beams were orthogonally circular polarized with respect to each other, one is right-circular polarization (R) and the other is left-circular polarization (L).

The interference pattern of two coherent waves with two parallel linear polarizations (SS, PP) has a largest periodically modulated intensity when the contrast of an interference pattern is close to 1, but a polarization state has not spatial modulation in the direction of the resultant electrical vector. In case of orthogonal linear polarizations SP the interference pattern has a constant intensity, but a polarization state on sample surfacethat is periodically modulated. In case of orthogonal linear $\pm45^\circ$ and circular RL polarizations states the interference pattern has a small modulated intensity and is
uniform over the entire exposed area. But these polarizations states (±45° and RL) produce the largest periodically modulation in the resultant electrical field direction on the film surface. The differences in phase of beams lead to sum of polarization states on sample surface periodically variable from linear to circular and conversely. Very small DE and surface relief modulation were obtained for SP polarization states. For last configuration there is no component of resultant electric vector of light along the grating vector direction.

The quarter wave plates were mounted on rotation mounts with angular scales. The phase shift experiments were performed by angular adjustment the quarter wave plates in each optical path; therefore, we can change the polarization state of both recording beams separately. The two interfering beams independently pass through phase turning quarter wave plates to provide a control over the polarization state of the writing beams. Interfering beams with PP, SS, LR or ±45° polarization combinations were used for SRG recording. The experimental set up is sketched in Fig. 2.

![Optical arrangement for holographic grating recording with real-time measurement of diffraction efficiency by photodiodes.](image)

**Figure 2.** Optical arrangement for holographic grating recording with real-time measurement of diffraction efficiency by photodiodes. CW DPSS laser (λ=532 nm, power =100 mW), M, M1,M2–mirrors; BP – right-angle prism with Al coating; BS – beam splitter; LD – laser diode (unpolarized, λ=650 nm) for monitoring of the recording process; P1, P2 - polarization plates; HWP – halfwave plates; PhD₀, PhD₁ – photodetectors.

Nonactinucun polarized light of laser diode was applied for all DE (η) measurements. The DE was controlled in real time by measuring of LD intensity in two first orders (I₁⁺, I₁⁻) in the transmission mode. The DE was determined as \( \eta = \frac{I₁^+ + I₁^-}{I₀} \times 100\% \), where \( I₁^+ \), \( I₁^- \) – intensities of diffractive beams of +1 and -1 diffraction orders, \( I₀ \) – intensity of transmitted beam in zero diffraction order. During holographic recording all the changes in NML volume and surface (like those in absorption and refractive index relief) were controlled by measuring the DE transmission. So the cumulative changes in DE of NML As₂S₃–Se structure were recorded and measured.

3. Results and Discussions
DE recording kinetics for NML As₂S₃–Se with two grating periods \( \Lambda \) is shown in Fig.3. Recording of grating was continued until saturation level of the DE’ value is achieved. The progresses of kinetic curves differ strong for grating periods 1.0 μm and 1.25 μm, and depend on beams polarizations states. For intensity gratings the DE’ results for SS and PP polarizations recording are identical, and we present only PP states. The largest value of DE were obtained for orthogonal linear (±45°) and circular (LR) polarized beams configuration when SRG is recorded. For these configurations there are components of resultant electric vector of light parallel and perpendicular to the grating vector direction. This indicates that the resultant electric field and its direction variation are essential to the formation of SRG on the ChG.

For polarization gratings i.e. recorded at LR and ±45° of polarization states the recording process begins in 1-3 min. after shutter opening the illumination and reach the saturation in 45-50 min. For amplitude gratings (PP polarization states) the recording process begins immediately after shutter...
opening and has maximum in 7-17 min. and then value of DE is drooping. Maximal reached value of DE depends on the recording grating period. In Fig.3 the data corresponding the minimal value of DE for period $\Lambda=1 \mu m$ and the maximal DE for $\Lambda=1.25 \mu m$ are shown. According to results the vector recording in NML As$_2$S$_3$–Se is more efficient than scalar one. It should be emphasized that the best value of DE=45% is obtained for vector recorded gratings and DE≈35% is for scalar one.

Figure 3. The kinetics of DE for three states of polarizations PP, LR and $\pm 45^\circ$ are shown. Results of equal NML thicknesses are shown.

Note that in Fig.3 the DE kinetics of NML As$_2$S$_3$–Se with thicknesses of nanolayers $d=11$ nm for both As$_2$S$_3$ and Se nanolayers is shown. The other nanolayers thicknesses values as $d_{As_2S_3}=9 \mu m$, $d_{Se}=5 \mu m$ give close results to the maximal value of DE for $\pm 45^\circ$ and PP polarization states (see Fig.4).

The diffraction efficiencies kinetics in dependence of the grating periods for vector and scalar HG recordings are compared in Fig.4. Maximal value of DE monotonically increases for scalar HG whereas a maximum around $\Lambda \approx 1.25 \mu m$ takes place for vector HG. Moreover, the evolution of DE is constantly rise until saturation for PP polarization whereas for $\pm 45^\circ$ one can see the slope around 10 min at $\Lambda = 1.7 \mu m$. For periods $\Lambda = 2.0 \mu m$ and $\Lambda = 2.5 \mu m$ we observe the N-form of time dependence of DE. It is evidence of the existing more than one recording process during HG formation.

Periodic sinusoidal gratings can be categorized as either thick or thin [11]. Thick gratings show Bragg diffraction. Their DE can be approximated analytically using Kogelnik’s coupled wave theory [12], which is valid close to the Bragg angle. Thin gratings show Raman-Nath diffraction behavior. They are classically treated with scalar diffraction theory and their far field diffraction efficiencies are approximated using the Fraunhofer approximation [13].

Figure 4. The DE kinetics of vector (on the left side) and scalar (on the right side) recordings for different periods $\Lambda$ of HG.

In order to categorize gratings as either thick or thin for finite values of thickness ($d$), one may look for a parameter that indicates whether the analytical expressions for Bragg and Raman-Nath diffraction result in reasonable approximations. This is done by comparing the grating thickness $d$ and
the wavelength $\lambda$ with $\Delta n$ and/or the average index of refraction $n$ and the grating period $\Lambda$. Most often, the Klein’s parameter $Q$ [14],

$$Q = \frac{2n\mu d}{\Lambda^2},$$

is used for evaluations and a grating is considered thin when value $Q < 1$ and thick when $Q > 10$.

![Graph showing the variation of diffraction efficiency (DE) with grating period for different combinations of Se and As$_2$S$_3$ thicknesses.](image)

**Figure 5.** The kinetics of DE in the first order for ±45° of polarization states of recording beams. Results of thickness combinations of Se and As$_2$S$_3$ nanolayers are shown.

The estimation of $Q$ parameter for the NML As$_2$S$_3$–Se was shown that we can consider the HG as a thin grating for value $\Lambda > 1.4$ µm. The HG with value $\Lambda < 1.4$ µm are intermediate gratings, i.e. neither thick nor thin. In Fig. 5 the kinetic of diffraction efficiency are presented for different periods of grating and two combinations of nanolayers thicknesses. It is seen that really the form of curve is changed at period $\Lambda = 1.4$ µm. We observe the constant rise of DE value up to saturation at $\Lambda$ from 1.0 µm to 1.25 µm. The maximum value of DE is about 45%, which exceeds the theoretical limit for thin gratings 33.9%. It is indicating that these gratings don’t obey the Raman-Nath diffraction law. Beginning from periods at 1.4 µm to 2.5 µm the curves of DE’s kinetics have one or even two (for $\Lambda = 1.4$ µm) maximums, whereas the value of DE’s maximums is less (30-40%) than for smaller periods. It may be explained by appearance of higher diffraction orders which we observed in experiment.

Note that this alteration of DE’s kinetics exists only for polarization gratings recorded at ±45° and LR polarizations states, not for intensity gratings recording. For comparison we present on Fig. 6 the data showing the kinetics of scalar gratings diffraction efficiency for PP polarizations states of recording beams.

From Fig.6 one can see that for all samples under investigation the PP polarization states of recording beams the DE value increases reaching the high value ~45%. Only for value $\Lambda = 2.5$ µm the DE begins to decrease after the time interval around 25-35 min. So the intermediate (between Raman-Nath and Bragg laws) diffraction behaviour presents in NML samples at PP polarizations states up to value $\Lambda = 2.5$ µm.

It is necessary to say that, from a practical point of view, polarization modulation configurations are inevitably accompanied by intensity modulations, except in the case where S and P polarized laser beams are used for exposure create neither intensity nor polarization interference pattern. Note also that in those cases the scalar effects can play a predominant role if the samples are not photodarkened prior to their exposure to the polarization modulated light [15]. Therefore, it is difficult to separate scalar and vector effects.It confirms that the mechanism of relief modulations, in the case when spatially modulated polarization is used for exposure, is pure polarization driven.

There are some concepts to explain the origin of intensity and polarization recording features in ChG. It is known that molecules and nanoparticles can be manipulated in fluidic systems using dielectrophoretic (DEP) forces [16]. A DEP forces generated when a neutral molecule or particle is suspended in a nonuniform electric field. This electric field induces electrical charges within the particle to establish a dipole. If a nonuniform electric field is applied, the ends of the dipole experience unequal Coulomb forces, which result in a total nonzero imposed force. If the particle is less polarisable than the suspending medium, it is repelled from the regions of higher electric field and the motion is called negative dielectrophoresis while the opposite case is referred to as positive dielectrophoresis.
The authors [16] proposed a photo-induced DEP model to explain the photoinduced mass transport in amorphous films. Model is based on the photoinduced softening of the matrix, formation of defects with enhanced or decreased polarizability, and their drift under the electrical field intensity gradient of the light. An interference pattern from the orthogonal ±45° polarizations states can be expressed as a sum of two linearly polarized light components: the electric vector parallel to the grating vector or the P polarization and the electric vector perpendicular to the grating vector or the P polarization. For both components the electric field intensity gradient is parallel to the grating vector. This polarization modulation causes an arrangement of polar photoinduced defects in the film. As a result, their polarizability regarding the direction of electric field intensity gradient has been increased by P electric field component and decreased by P electric field component. Due to the DEP forces material is transferred out of the S polarized light region in to the P polarized region. This model is in well agreement with results obtained for homogenous ChG. In case of NML we obtain the regions with deferent structures and as a consequence different polarizability of nanolayers constituent of total NML.

The other model was proposed by H. Fritzche [17]. Note that all models suppose the creation of anisotropy in the material, but the reason for appearance of this anisotropy is different. These optical anisotropies induced by exposure to polarized light are observed not only in annealed glasses but also, and essentially undiminished, in glasses photo darkened by light exposure with quant energy more than optical band gap.

On the molecular scale, the structure of chalcogenide glasses is strongly anisotropic [17]. Even without specifying the larger anisotropic structural units, such as rings, chains, platelets, or pyramids, which may define the local structure or medium range order of a particular glass composition, it is clear that one needs a volume containing a considerable number of atoms to find the optical isotropy of a macroscopic sample. The minimum isotropic volume will grow with the size of the anisotropic structural units. This minimum volume is optically isotropic only because of the spatial averaging over smaller anisotropic volume elements it contains. Optical anisotropies can arise only when one or both of the photocarriers remain in the absorbing microvolume and recombine nonradiatively. The recombination is called geminate when both carriers remain and recombine in the absorbing microvolume. Accept this model, we can explain the different photoinduced behaviour of As$_2$S$_3$ films and NML As$_2$S$_3$ -Se which we observed in [18].

4. Conclusions

The DE recording kinetics of surface relief grating patterned on NML As$_2$S$_3$ -Se by one-step polarization holographic recording were investigated in dependence of the intensity grating periods. The obtained results were compared with the DE kinetics for the amplitude holographic recording. A strong dependence of surface relief gratings DE on grating period and sample thickness was observed. It has been determined that the highest possible efficiency ~45% of the one-step surface relief holographic recording can be achieved using left-right circular and ±45° polarizations states. The light
intensity contrast of interferograms is very close to zero and the electric field intensity gradient plays the main role for the direct surface relief patterning. The estimation of the Klein’s Q parameter for the NML As$_2$S$_3$–Se was shown that the holographic gratings with period $\Lambda$ more than 1.4 $\mu$m can be considered as thin gratings leading to Raman-Nath diffraction law.

The behaviour of mass transfer and thus the resulting recording surface gratings could be related to interaction between the photo-induced anisotropy in nanolayer with enhanced or decreased polarizability of molecules and their drift under the electrical field intensity gradient.

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