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CAZAC VERONICA

DEVELOPMENT OF DIGITAL HOLOGRAPHIC MICROSCOPE FOR THE INVESTIGATION OF DIFFRACTIVE OPTICAL ELEMENTS RECORDED ON CHALCOGENIDE GLASSES AND AZOPOLYMER THIN FILMS

134.01 – Physics and technology of materials

Abstract of the PhD disertation in physics

CHIŞINĂU, 2023

The PhD thesis was elaborated in the **Laboratory for Materials for Photovoltaics and Photonics, Institute of Applied Physics of Moldova of the Moldova State University**. The doctoral studies were carried out at the **Doctoral School of Nature Sciences at Moldova State University.**

PhD Supervisor:

ACHIMOVA Elena, PhD Habilitate in physical sciences, research associate, Institute of Applied Physics of Moldova State University

Doctoral Commission:

CULIUC Leonid, PhD Habilitate in physico-mathematical sciences, professor, academician, Moldova State University– **president of the DC**

ACHIMOVA Elena, PhD Habilitate in physical sciences, research associate, Institute of Applied Physics of Moldova State University – **PhD supervisor**

SIDORENCO Anatolie, PhD Habilitate in physico-mathematical sciences, professor, academician, Institute of Electronics Engineering and Nanotechnologies „D. Ghițu” of Technical University of Moldova – **official reviewer**

URSACHI Veaceslav, PhD Habilitate in physico-mathematical sciences, professor, research associate, corresponding member of the Academy of Sciences of Moldova, National Center for Materials Study and Testing of Technical University of Moldova – **official reviewer**

CEBAN Victor, PhD in physical sciences, superior scientific researcher, Institute of Applied Physics of Moldova State University – **official reviewer**

The dissertation defense will take place on December 21, 2023, at 15:00, in the meeting of the Doctoral Commission within the Doctoral School of Nature Sciences of Moldova State University, Office – Doctoral School of Nature Sciences, Moldova State University (http://www.usm.md), str. A. Mateevici 60, building no. 4, room 222, MD-2009, Chișinău, Moldova.

The summary of the PhD thesis can be consulted at the National Library of Moldova, Central Library of Moldova State University (MD 2009, mun. Chișinău, str. Alexei Mateevici 60), on the ANACEC website (http://www.cnaa.md) as well as on the website of Moldova State University (http://www.usm.md).
The summary was sent on November 21, 2023

Președintele Comisiei de Doctorat ____________ **CULIUC Leonid**
doctor habilitat în științe fizico-matematice,
profesor universitar, academician

Conducători științifici:

doctor habilitat în științe fizice, ____________ **ACHIMOVA Elena**
conferențiar cercetotor

Autor: ____________ **CAZAC Veronica**

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CONCEPTUAL GUIDLINES OF THE RESEARCH

Actuality and importance of the topic

In the last decade, many hardware and software solutions have been proposed to enhance the digital holographic microscope (DHM) performance with current available digital sensor array dimensions and non-ideal imaging conditions [1-3]. Despite intense research efforts, there still exists an increased demand in the improvement of DHM especially for obtaining high-precision, full-field depth mapping as well as 3D surface and shape measurements of DOEs pattern on photo-sensitive materials. Being a non-destructive and label-free technique, DHM was used to 3D and quantitative phase imaging of live cancer cells dynamics [4], light-induced surface deformation of thin films [5] and many other fascinating implementations [6].

DOEs, as the name suggests, are compact optical components capable of tailoring the light in complicated ways via diffraction phenomenon. Two centuries of research on fabrication of DOEs on optical materials gave rise to several techniques as photolithography [7], focused ion beam writing [8], and holography [9]. Most of these techniques provide good quality DOE, however time-consuming procedures involving numerous processing stages is the main problem, especially when encumbering etching procedures are required.

Polarization holographic recording (PHR) operating with different polarization states of the recording beams, triggers the mass-transport mechanism that leads to deep photo-induced surface-relief formation without additional post-processing stages. The advance of this fabrication technique is important for understanding how light interference can cause macroscopic movements of different types of photo-sensitive optical materials and for its efficient use in photonic applications. For this purpose, new digital recording approaches must be developed along with the analog ones. The design of the PHR setup allows the operation of a SLM, useful for controlling of the geometry and phase of DOEs via computer generated holograms.

The quality of the DOEs is determined not only by the fabrication techniques but also by the properties of optical light-responsive materials. High photo-sensitivity, high-resolution, low cost and wide spectral and frequency range are the key requirements for the optical materials. Furthermore, it is very important that the recording media maintains temporal stability of the obtained DOE. Chalcogenide glasses (ChG) and azopolymer (AP) thin films are optical materials that possess the above-mentioned characteristics including sensitivity to polarization and match the PHR as a patterning technique.

For this reason, with an aim to extend the understanding of ChGs nano-multilayers and AP thin films photo-induced polarization behavior, a part of this thesis is focused on design, fabrication and investigation of DOEs patterned on these materials. In addition, the application of the enhanced DHM technique for quantitative investigations of the designed DOEs provides essential information about anisotropy and birefringence properties of these optical materials.

The aim of the thesis

The main goal of this work is to improve the design and performance of DHM, including its hardware and software components, for an accurate quantitative description of key parameters of different types of DOEs, patterned on ChGs nanomultilayers (NMLs) and AP thin films by polarization holographic recording.

The objectives of the thesis

In order to accomplish the aim of the thesis, it is necessary to fulfill several objectives:

- Develop numerical reconstruction algorithms for accurate phase reconstruction from acquired digital holograms of DOEs recorded on photosensitive ChGs nanomultilayers and AP thin films and subsequent mapping of their surface relief and refractive index mapping.
- Modify the optical part of the DHM in the off-axis and phase-shifting configurations by introducing liquid crystal devices. Phase and polarization calibration of the liquid crystal devices implemented in digital holographic setups. Compare the effectiveness of these approaches.
- The development of analog and digital holographic techniques for one-step polarization holographic recording of different types of DOEs based on ChGs NMLs and carbazole-based APs.
- Apply the developed imaging – DHM - system with all the integrated tools for characterization of the created DOEs.

Scientific research methodology

The theoretical support of the dissertation was carried out based on analysis of relevant literature accessed from the electronic libraries of Moldova State University, Tampere Universities and through online access to scientific journals and open access articles. In order to achieve the objectives of the dissertation, the following methods were used:

1. Analog and digital PHR methods were applied to record different types of DOEs on ChGs nanomultilayers and AP thin films.

2. The DHM in off-axis and phase-shifting configurations were applied for the surface relief and refractive index mapping of DOEs recorded on ChGs NML and AP thin films.

3. Wavefront shaping techniques by digital modulation of light. Measurement and manipulation of the light field have been realized through the utilizations of liquid crystal devices.

4. The variational algorithm Sparse Phase and Amplitude Reconstruction (SPAR) proposed in [10] and Fast Fourier Transform (FFT) methods were adjusted and applied in the numerical algorithms for phase reconstruction from off-axis digital holograms. Both methods were used for calibration of the SLM.

Theoretical significance and applicative value

The presented research is of particular interest because it targets a relatively young field and contains research issues that exist internationally. In recent years, there has been an increasing emphasis on the computerization of scientific equipment and devices, and especially the microscope. For the DHM to become competitive with the atomic force microscope (AFM) or electron scanning microscope, reconstructed results from digital holograms must be viable and the working time with this tool must be reduced. Enhancement of microscopic tools is mandatory in order to support efficient investigations in different fields as photonics, material science, biomedicine, etc. Therefore, the optical system of DHM including hardware and software components was improved for efficient investigation of the DOEs on a nanometer scale.

The capability to create and perform quantitative analysis of multifunctional DOEs make them attractive in a variety of applications, including aberration correction, sensitive microscopy, augmented reality, imaging systems, solar energy and more.

The main scientific results submitted for defense:

1. Analog and digital PHR are direct approaches that can be applied for one-step fabrication of multifunctional DOEs on the surface of ChGs nanomultilayers and azopolymer thin films.

2. Phase imaging enhancement can be obtained by adopting a sinusoidal phase grating mask to the off-axis lens less DHM setup.

3. Integration of liquid crystal devices in digital holographic optical systems presumes the mandatory and important procedure of detailed phase and polarization calibration for each specific configuration.

4. Off-axis and phase-shifting DHM configurations with integrated liquid crystal devices are applicable for the effective investigation of refractive index and surface relief maps of multifunctional DOEs recorded on azopolymers thin films and ChGs NMLs.

The scientific novelty of the research

The work includes several advances that improve upon current state-of-the art. First, the improved version of the DHM software and hardware configuration has been demonstrated as a versatile instrument for the investigation of DOEs on ChG nanomultilayers and AP thin films. Second, analog and digital PHR are new methods that allowed the photo-induced formation of different multifunctional DOEs on ChG nanomultilayers and AP thin films.

The solved scientific problem

As a result of the work, it was shown that the digital holography technique increases both the possibilities of polarization holographic recording of multifunctional DOEs on ChGs NMLs and AP thin films, and the perceptivity of digital approach to research of the obtained DOEs. The current work constitutes an important step towards increasing the availability of computational imaging methods from the hardware-side, which has been typically a bottleneck.

Approval of the results of the thesis and scientific publications

The scientific results were presented and discussed at 11 national conferences, including international conferences that took place in the Republic of Moldova (5), Romania (1), Russia (1), Switzerland (1), Finland (1), Ukraine (1), United States of America (1).

In total, 9 articles as main author and 6 articles (including abstracts, published in the conference proceedings) as co-author were elaborated and published, out of which: 2 single-author articles; 4 articles were published in specialized scientific journals abroad. All 4 journal articles are indexed in Scopus. Scientific results described in this thesis were obtained and implemented in 5 projects, inlcuding one EU-funded international project.

DISSERTATION CONTENT

The thesis has been written in English language and consists of the introduction, 5 chapters, general conclusions and recommendations, and the list of 214 references. The thesis contains 128 pages of basic text, 85 figures and 62 formulas. The results presented in the thesis are published in 15 scientific publications and presented at 11 international conferences.

Chapter one presents a general overview of the existing DHM technologies. The main challenges encountered during digital hologram recording and computational processing are described. Important advantages and limitations of different DHM configurations are highlighted. State-of-the-art methods proposed for improving the resolution and overall DHM performance are discussed. ChGs nanomultilayers and azopolymer thin films are described as photo-sensitive media for DOE recording. Chapter one ends with description of the novelty and contribution of the doctoral thesis.

Chapter two describes the theoretical framework for the improvement of the DHM by computational processing. FFT and SPAR phase reconstruction methods are described for the off-axis digital hologram processing procedure. For the phase-shifting holograms, an algorithm based on the Yamaguchi approach given in detail in [11-13] was designed and adopted. Different phase masks were investigated for improving the DHM performance in both, off-axis and phase-shifting configurations. The phase masks were introduced in the setup for modulating the object wavefront. The success of the experiment was evaluated by calculating the root mean square error for both cases.

Fig. 1. Numerical simulation results of the experiment with a Gaussian phase object. After phase mask rotation the value of $RMSE_{phase}$=0.0243, without phase mask $RMSE_{phase}$=0.0292.

Computational simulations results prove that phase imaging enhancement can be obtained by adopting a sinusoidal phase modulation by a diffraction grating for the off-axis lens less DHM setup. The following optimal phase mask parameters were identified: the period $\Lambda = 44\mu m$, the value of phase modulation was $\varphi = 0.1\pi$ and two positions rotation steps of the phase mask at 0, 90, 180 degrees. Due to the optimized diffraction grating parameters (period, phase and number of rotating steps) more information about the high spatial frequencies of the object spectrum is recorded in the CCD sensor from the diffracted orders. Computational experiments show that with sinusoidal phase modulation RMSE values are decreased about 20%.

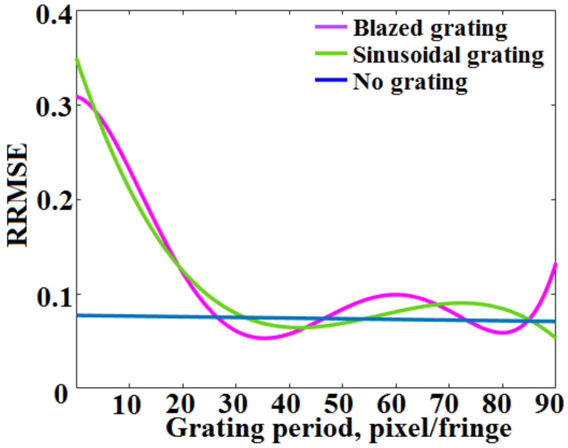


Fig. 2. RRMSE dependence on the sinusoidal and blazed gratings period.

Computational simulations of the phase-shifting DHM configuration prove that by introducing a blazed diffraction grating in the phase-shifting optical setup the precision of the phase reconstruction can be improved by approximately 72%. Particularly, the simulation showed that the best reconstruction can be obtained for the blazed grating with a period equal to 35 and 80 pixels/fringe and an angle $\alpha=90^0$ with respect to the optical axis of the DHM. The results are demonstrated for two phase objects specifically, a Gaussian beam and a phase step that were selected as experimental samples. The obtained results were compared with the experiment without the phase mask. The standard Gaussian noise was considered. Several factors affecting the process of recording holograms were not considered in the numerical simulation. Uncontrollable factors include: the stability of the laser in spatial coherence, the noise of the video camera, the rigidity of the tables, and the scattering of light by micro particles in the air.

In the **third Chapter**, the implementation of the liquid crystal devices in digital holography is discussed. The pixel-wise phase calibration of the SLM is described in the first section of this chapter. Two different algorythms were used for the phase reconstruction, an FFT and a SPAR based methods. In addition, retardance calibration of the SLM is performed in order to understand how the device changes the polarization of the outcoming beam. The calibration of the liquid crystal variable retarder is presented in the second section of this chapter.

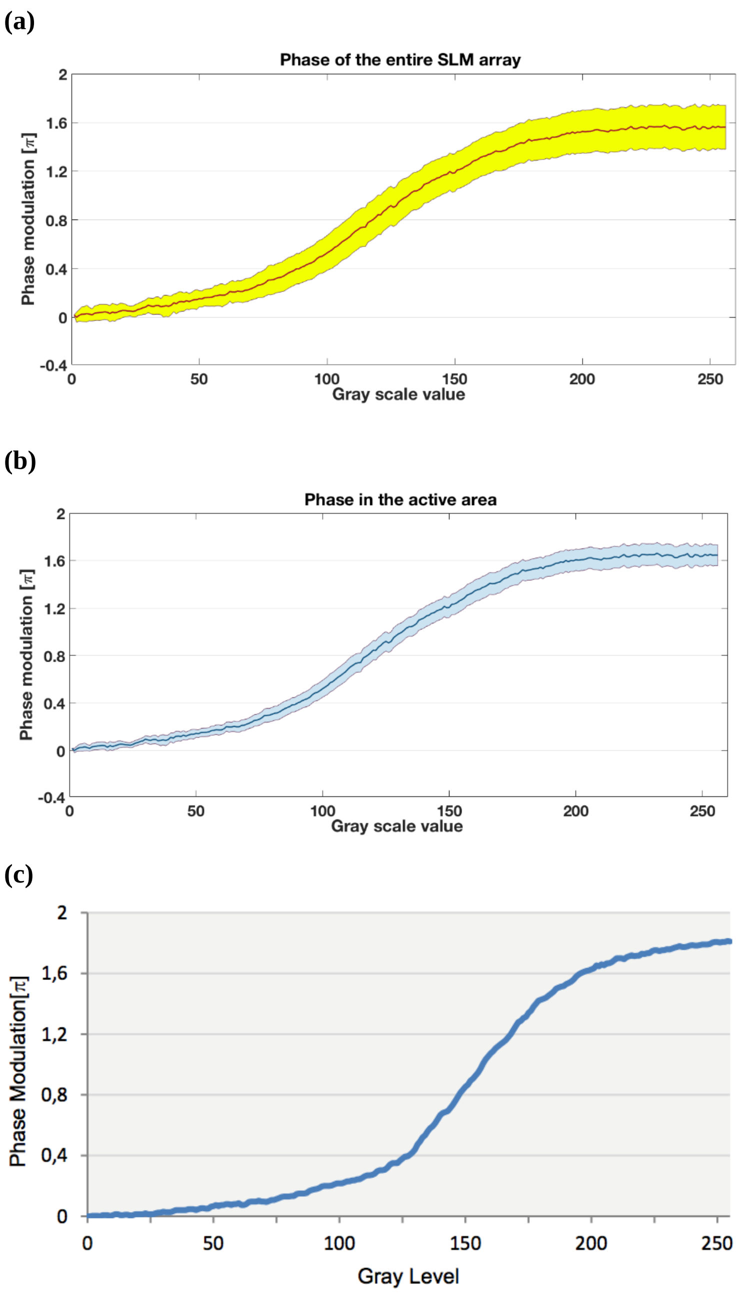


Fig. 3. The calibration curve resulted from application of the SPAR algorithm representing the relationship between the SLM phase modulation (blue and red lines), phase standard deviation (yellow and blue areas around the main curves) and the gray scale of (a) the entire SLM array and (b) in the active area of the SLM pixel for λ=532 nm; (c) the calibration curve given by the manufacturer for λ=543 nm.

From the results obtained by the SPAR based algorithm it can be noticed that in the region of maximum phase modulation, the standard deviation of the active part of the SLM pixel is 0.5 radians smaller if compared to the phase of the entire SLM array, as illustrated in Fig. 3.

This effect could be the result of the “dead zones” of the SLM array, where the polarization of passed laser beam cannot be controlled.

Even though the dependences presented in Fig. 3 show similar tendencies, specific differences can be noticed when comparing the calibration curve given by the manufacturer Fig. 3(c) with the curves resulting from the implemented SPAR algorithm. First, the phase response increases at lower rates of gray scale values using the calibration setup and software approach described in this chapter. Particularly, at the 125-gray scale value, the phase modulation of the SLM active pixel area is π; the entire SLM display modulates 0.9π, for λ=532 nm. Also, the maximal phase modulation of the SLM active pixel area is 1.7π and the entire SLM display is 1.6π.

The phase shifting calibration of the LCVR by taking into account the polarization of the incident beam at a wavelength of 532nm is described. The calibration of the LCVR is a mandatory procedure, since detailed data about this device (on this exact wavelength) is not provided by the manufacturer. The phase shifting produced by the LCVR was done by a preliminary fine adjustment of the fast (slow) axis of the liquid crystal with the polarization plane of the incident beam. To produce accurate all-optical phase shifts, via DHM systems containing an LCVR, a linear approximation of the phase shift in the region 2-3.2V of the applied voltages was build.

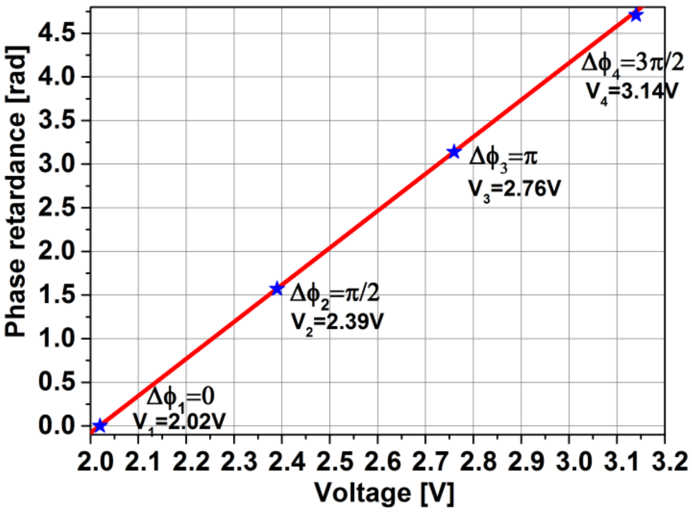


Fig. 4. A linear approximation of the LCVR phase shift in the region 2-3.2V vs. the applied voltages.

Since the DHM system described in the fifth chapter of this thesis requires to produce particular phase shifts equal to $\varphi_1 = 0$, $\varphi_2 = \frac{\pi}{2}$, $\varphi_3 = \pi$, $\varphi_3 = 3\pi/2$ a linear approximation of the LCVR phase shift in the region 2-3.2V vs. applied voltages was carried out. The calibration results are shown in Fig. 4. The initial applied voltage is 2.02 V where the phase shift is considered null. According to the formula $\Delta\varphi_n = \mathrm{a} + \mathrm{b}V_n$, where a=-8.58 and b=4.25 are constants, V_n is the corresponding voltage, and n=1,2,3,4. The phase step is equal to π/2. After calculations, it was determined that the four phase changes pointed out above correspond to the voltages V_1 = 2.02 V, V_2 = 2.39 V, V_3 = 2.76 V, V_4 = 3.14 V, respectively.

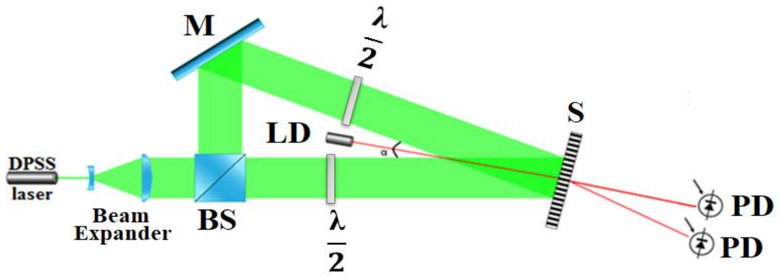
The resulted calibration data shows that non-negligible errors are probable when controlling the phase and polarization of LC devices. For proper application of these devices, prior to use the SLMs, a characterization study must first be performed. The interferometric set-ups presented in this chapter are compatible with in-situ calibration and can be adapted to different optical systems. Furthermore, the software procedures included in the SPAR calibration algorithm compensate the effects of external noise which reduce the requirements for stability of the interferometer. The described procedures for region-by-region calibration of the SLM have valuable implementations in DHM.

Chapter four is dedicated to the design of various DOEs on ChGs nanomultilayers and azopolymer thin films. In the first section, the fabrication technique of photosensitive ChGs NML and azopolymer thin films is described. Nanomultilayers of ChGs were prepared by computer controlled successive cyclic thermal vacuum deposition of bulk As_2S_3 and Se. Azopolymer thin films were synthesized by the polymerization of the poly-n-epoxypropyl carbazole (PEPC) with azo dye Solvent Yellow 3 (SY3) chromophore. For a broad understanding of the photo-sensitivity of this materials analog and digital PHR were exploited. The DE kinetics was monitored and analised for DOEs obtained with different exposure doses and recording approaches.

Analog PHR with S:S, P:P, ±45° and RCP:LCP polarization configurations were implemented on ChGs NML and a series of surface relief and refractive index gratings were obtained. The recording arrangement was located on vibration isolated table in order to be insensitive to the environmental perturbation. In order to investigate the kinetics of the photo-induced gratings patterning, the DE in dependence on the exposure time is measured by a laser diode (LD) and two photodiodes for the intensity measurement at zero I_0 and first I_1 diffraction orders. The highest DE was obtained for DOE recorded by ±45° polarization configuration reaching 31% for As_2S_3-Se NML and 13.5% for As_2S_3 NML. Figure 5 (a) shows the experimental setup used to record sinusoidal gratings on As_2S_3 and As_2S_3-Se NML.

Digital dual-beam PHR was applied on As_2S_3-Se NML producing a complex sinusoidal DOE that generated special multiplexed diffraction maxima. For performing digital PHR of vortex DOEs a SLM was integrated in the recording optical systems. The essence of SLM utilization for PHR is the computer-generated hologram that involves numerical calculation of the pattern rather than producing it photographically. The phase SLM panel acts as a sophisticated diffraction grating which transforms the phase of the transmitted light beam into a specific pattern. The phase modulator, in contrast to amplitude one, is more efficient as it completely redirects the incoming light to the outcome image. The dynamic phase range of the SLM (LC2002 HOLOEYE) used in our experiment depends on the wavelength of incident light and is less than 2π for wavelength of λ=532 nm.

(a)



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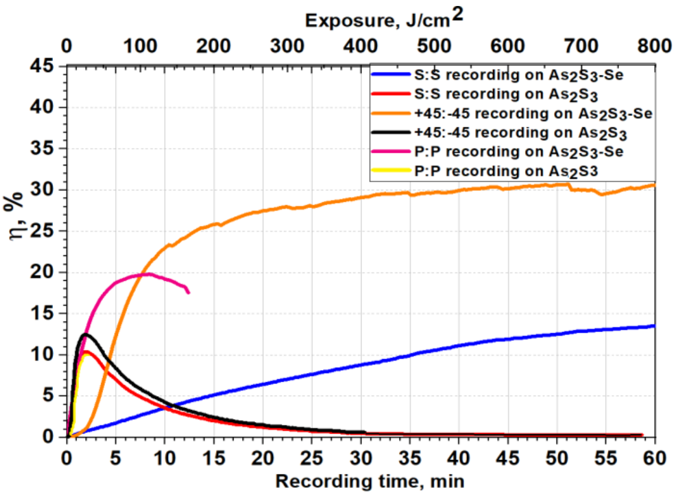
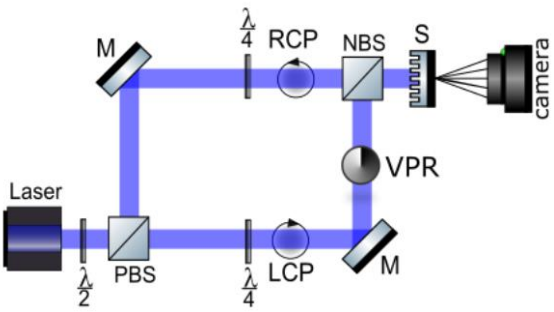


Fig. 5. (a) Analog PHR for patterning on ChGs NMLs: single mode laser DPSS (λ=532nm, 100 mW), beam expander, BS-beam splitter 50/50, PD-photo diodes, M-mirror, LD-laser diode (λ=650 nm, 1 mW), sample of As_2S_3 film or As_2S_3-Se NML, α-angle between incident beams, λ/2-half wave plate on the rotation stage; (b) Kinetics of the first-order diffraction efficiency of the recorded DOEs with different polarizations of the recording beams.

It can be supposed that the recorded DOE consists of two orthogonal surface gratings in the X and Y directions of the sample plane. The grating period on the X-axis resulting from the first recording mode is tuned by the period of the hologram fringes displayed on the SLM. The grating period on the Y-axis resulting from the second recording mode is determined by the interference produced by the reference and the object waves beams of the optical setup. The obtained DE value is about 0.4%, which is a relative value as it is estimated from a single diffraction maximum. The real efficiency is higher if considering all the diffraction maxima.

The designing process of vortex DOEs on carbazole-based azopolymers is described for analog as well as digital PHR. The patterned vortex DOEs are capable of generating single-channel and spatial multiplexed phase singularities. The spiral wavefront generated by analog PHR via VPR produces DOEs that create vortex beams with high diffraction efficiency, however the variability of the spatial distribution of the diffraction pattern is limited. In contrast, the digital generation of the spiral wavefront by SLM introduced in the PHR system is a more flexible source that permits to record DOEs that generate 2D and 3D spatial distribution of the diffraction patterns.

(a)



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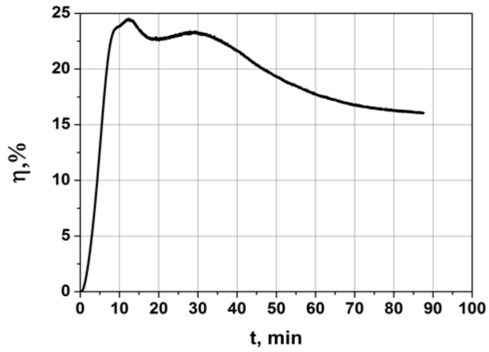

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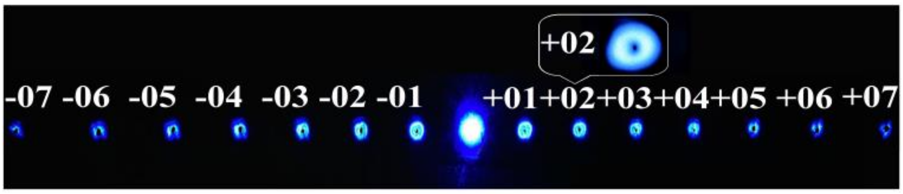


Fig.6. (a) Analog interferometric arrangement for vortex DOE recording via VPR on AP thin films. The components of the setup are: CW DPSS laser (λ=473 nm, power =100 mW), M – mirror, PBS – polarized beam splitter, NBS- non-polarized beam splitter, VPR-vortex phase retarder plate, S – sample; λ/4- quarter wave plate, λ/2- half wave plate, camera - 12MPxs camera; (b) Kinetics of the first-order diffraction efficiency of the recorded vortex DOE obtained by VPR-based PHR; (c) diffraction image of the phase singularities (donuts) displayed in the diffraction pattern.

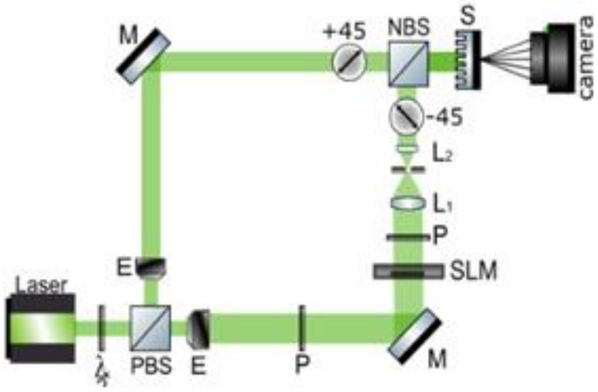
The sketch of the analog PHR setup based on a VPR is illustrated in Fig. 6 (a). The laser beam polarization state is inclined at 45^0 by a half-wave plate. Two quarter-wave plates with fast axes reciprocal angles at 90^0($+45^0$ and -45^0 to vertical axis) create RCP and LCP. The DOE is produced by interference between a spiral wavefront formed after a laser beam passes through the VPR and a plane wavefront of the reference beam. The VPR generates an OAM with a TC equal to 1. Both interfering beams pass through quarter-wave plates for obtaining a polarized DOE. The angle between interfering beams is approximately $\theta \approx 3.6°$. This angle determines the period of the grating on the X-axis of approximately 7.5μm. It can be underlined that the DOEs that are used to create optical vortices are fabricated in a one-step recording process. The resulted diffraction pattern projected on the camera is pictured in Fig.4.13 (b). The collinearly aligned phase singularities can be observed on the diffraction image, shown in Fig. 6 (c).

Digital PHR of vortex DOEs on azopolymer thin films was performed using single-beam and dual-beam PHR. A fork-shaped grating (FSG) is displayed on the SLM panel instead of a vortex phase mask. It is another powerful method for generating beams with a desired singular phase distribution and topological charges TC=1 and TC=2. The two-dimensional intensity distribution, which would be observed as a result of interference was calculated.

The multifunctional vortex DOE was imprinted simultaneously by means of two different recording modes, operating in a parallel regime. The first recording mode functions as the single-beam PHR described previously, with the SLM display located in the object arm of the optical setup, as pictured in Fig. 7 (a). The second recording mode functions as a Mach-Zehnder interferometric recording setup due to the addition of the reference arm to the patterning system. The dual-beam PHR setup is shown in Fig. 7 (a). The vortex DOE is formed when the object beam passing through the SLM and possessing -45^0 polarization and the plane Gaussian reference beam possessing $+45^0$ polarization (with regard to the plane of interfering beams) interfere. In this way, the angle between polarization states of the interfering beams is 90^0. As a result, only the phase pattern was recorded on the thin film, and amplitude was not recorded.

As shown in previous investigations [14] on digital PHR recording on azopolymer thin films, cross-polarized states of light beams controlled by half-wave plates facilitate the maximum surface relief modulation. The half-wave plate placed immediately after the laser controls the polarization direction of the incident beam with regard to the beam splitter. The polarized beam-splitter provides 30:70 intensity ratio with a minimal dependence on the polarization state of the incident light. For covering the entire SLM matrix, the incident beams were expanded. To scale the hologram transmitted through SLM in correspondence to the recording spot on the film, two lenses with different focal lengths were used. The spatial multiplexed phase singularities obtained from the DOE recorded via dual-beam PHR is shown in Fig. 7 (c).

(a)



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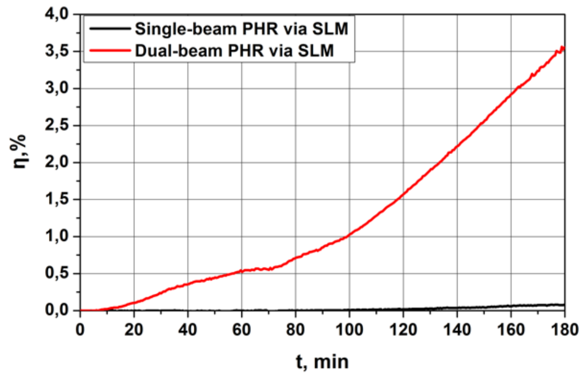

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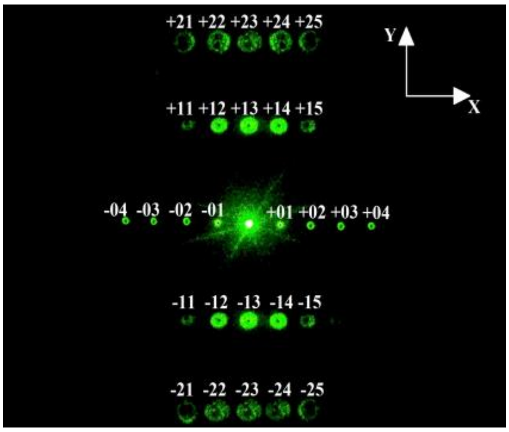

Fig. 7. (a) Digital dual-beam PHR setup for patterning complex vortex DOEs on AP thin films. CW DPSS laser (λ=532 nm, power =2W, TEM00 mode), M – mirror, PBS – polarized beam splitter, NBS- non-polarized beam splitter, E-beam expander, SLM-spatial light modulator LC-2002 with a resolution of 800 × 600 pixels and a pixel pitch of 32 μm, S – sample; λ/2- half wave plate, P-polarizer, L1, L2-lenses, (b) Kinetics of the diffraction efficiency in the +01 order of the recorded vortex DOEs obtained by digital PHR via SLM; (c) The spatial multiplexed diffraction pattern in donut-shape was obtained via dual-beam digital PHR.

The specific advantage of the presented results is the combination of PHR with a new polarization-sensitive azopolymer, which permits to obtain DOEs with a deep surface relief of about 40% with respect to the total thin film thickness. The implemented analog and digital PHR in different optical setups have proven to be an applicable one-step method for patterning vortex DOEs that generate phase singularities. Although it seems that both approaches permit to fabricate DOEs that produce similar fields, the variation of the vortex beams parameters must be considered. The highest diffraction efficiency of 24% is exhibited by the DOE patterned via an analog VPR, while the DOE obtained by the digital single-beam interferometric setup with an embedded SLM produces vortices with a low diffraction efficiency of only 0.1%. It must be taken into account that in the first case the number of observed maxima are 12, while in the second case there are 28 maxima.

The **fifth chapter** presents the developed DHM for the investigation of DOEs obtained on ChGs NMLs and azopolymer thin films. The chapter considers the off-axis configuration for the 3D imaging and characterization of the surface relief and refractive index parameters of the DOEs. The second part of this chapter describes the implemented phase-shifting DHM configuration for the study of DOEs.

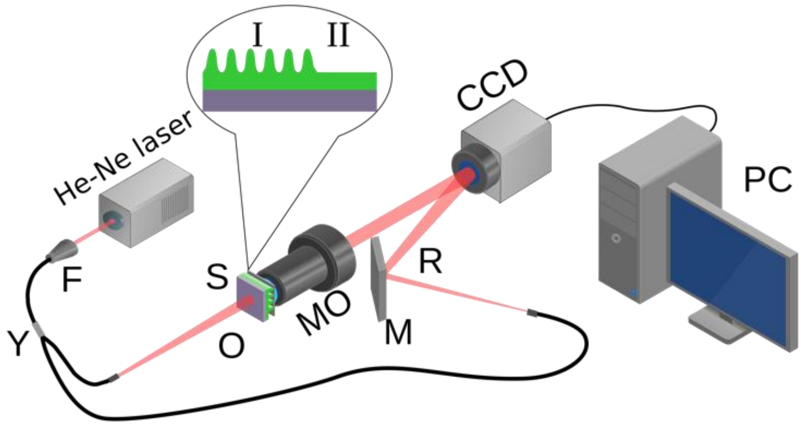

Fig. 8. Setup of the off-axis DHM: He-Ne laser ($\lambda = 632.8\ \mu m,\ power = 10mW$) F- micropositioner for coupling light into a single-mode fiber; Y-2x2 single-mode fiber optic coupler, O-object beam, R- reference beam, S-sample with diffraction grating and coverslip on top with immersion liquid (n=1.51); MO- immersion microscope objective, M- mirror and CCD camera, PC-computer, I, II show two illumination beams positions on the sample, namely I- through the grating and II-outside of the grating.

For analyzing the DOEs imprinted on ChGs an off-axis DHM bright-field configuration in transmission mode was used. The optical system of the off-axis DHM is shown in Fig. 8. For processing the recorded off-axis digital holograms, the iterative SPAR technique is used. It was recently developed in MATLAB software for the reconstruction of the wavefront phase with efficient noise suppression. A more detailed description of the SPAR algorithm is shown in Chapter 2.

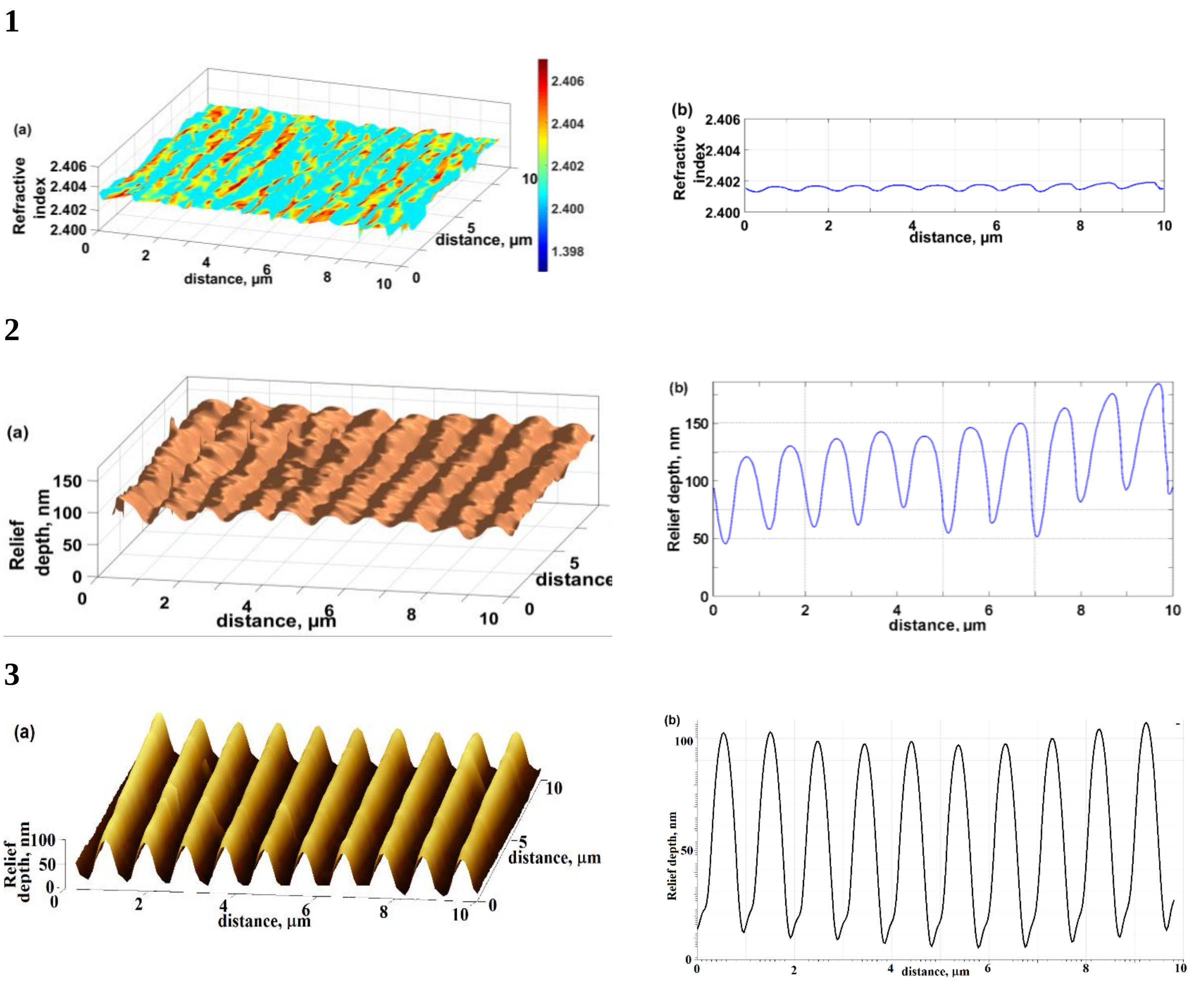

Fig. 9. 1(a) Refractive index map obtained via off-axis DHM and 1(b) mean cross-section for DOE recorded on As_2S_3-Se NML, 2(a) The topography map and 2(b) mean cross-section of DOE formed on NML As_2S_3-Se and obtained by DHM with SPAR technique, 3(a) 3(b) The topography map and cross-section of DOE formed on NML As_2S_3-Se and measured by AFM.

Several adjustments were made in the code according to the experiment features and nature/properties of the investigated DOEs.

First, a spherical reference wavefront as well as a plane one was incorporated in the algorithm contrary to the linear wavefront which is exploited in [10]. Second, the operating window dimensions x_m are 20-by-20 pixels, and the Gaussian standard deviation is 5, which have been tested to provide improved the reconstruction results.

The phase map $\Delta\varphi(x,y)$ of DOEs was determined from the reconstructed wavefront by the following equation

$$\Delta\varphi(x,y)=\varphi_{01}(x,y)-\varphi_{02}(x,y). \quad (1)$$

where φ_{01} is the phase calculated from the reconstructed object hologram of the exposed DOE and φ_{02} is the phase calculated from the reconstructed reference hologram of the unexposed area.

An advantage of the DHM with SPAR for refractive index mapping is that it can be used for the study of small variations of the refractive index variations while ellipsometry is a relatively slow scanning technique, effectuated in reflective mode and involves a cumbersome process.

Figure 9, 1(a) represents the three-dimensional refractive index distribution of the DOE on As_2S_3-Se NML and Fig. 9.1(b) represents the mean cross-section that corresponds to the surface topography shown in Fig. 9.1(a). The topography and cross-section of DOE with the diffraction efficiency of 13.4% obtained by off-axis DHM with SPAR technique Fig. 9.2(a), (b) were compared with measurements done by AFM shown in Fig. 9.3(a) and Fig. 9.3(b). The cross-section mean values of DHM data (Fig. 9.2(b)) match generally with the cross- section mean values of AFM data (Fig. 9.3(b)). The differences are more visible in the topography maps. The noisier 3D map in DHM may be as a result of speckle noise and of different spaces of data presentation in these methods. The difference is caused by type of analysis and the accuracy of AFM depends on the radius of the tip whereas DHM is a contactless optical method if compared to AFM.

From Fig. 9 it can be concluded that the DHM with SPAR reconstruction algorithm can be successfully used as an alternative for the phase reconstruction of SRG and it shows only small visual differences from the AFM cross-sections.

The PS-DHM system was upgraded with the LCVR to perform phase shifts in the reference beam. The microscope was applied for investigating sinusoidal DOEs obtained using different exposure doses and vortex DOEs obtained via analog and digital PHR on azopolymer thin films. The merit of the developed PS-DHM is that LCVR executes optical phase shifting, by driving voltages, with high accuracy. The automatic control of the LCVR adopted in LabVIEW software permits the holograms frame registration within 30 milliseconds. Thus, fast hologram acquisition minimizes the periodic background noise and temperature variation typically influencing the image quality.

The upgraded phase-shifting digital holographic microscope (PS-DHM) with a LCVR for producing all-optical phase-shifts is depicted in Fig. 10. In the experiment, the single-mode laser beam is divided by a non-polarized beam-splitter into the undisturbed reference wave-Rw and the scattering from the vortex DOE- Ow. The laser beam is expanded by a microscope objective and lens L2. Thus, the beam was collimated. The collimated object beam passes through a half-wave plate to adjust the polarization states of the object and reference beams.

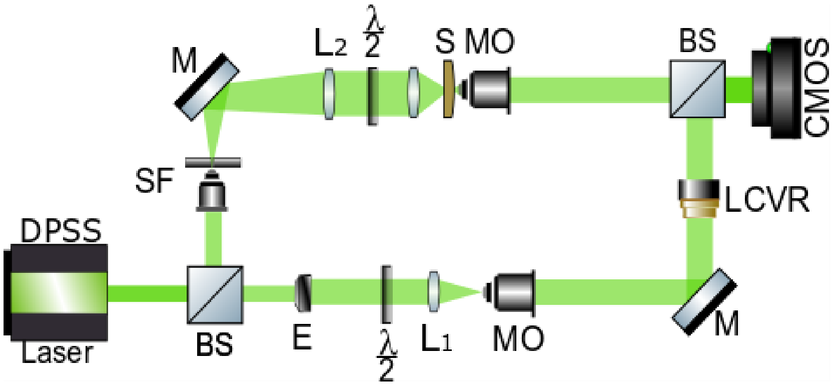

Fig. 10. Phase-shifting DHM setup: CW DPSS laser (λ=532nm, 100 mW, TEM00), BS-non-polarized beam splitter, λ/2- half-wave plates, M-mirror, MO-microscope objective (20x, NA=0.40), S-sample, L1, L2-lenses, CMOS- digital camera "DMK33UX264", resolution 2,448×2,048 (5 MPxs), LCVR- "Meadowlark optics" liquid crystal variable retarder LCVR-100, SF-spatial filter, E-beam expander.

The second lens, as a collimator, focuses light on the sample. The microscope magnification was evaluated by preliminary calibration using a 1 mm linear stage micrometer with 10 μm divisions. The phase of the reference beam is shifted sequentially by the LCVR. Nematic liquid crystals producing the phase retardations exhibit optical birefringence properties. Therefore, half-wave plates are introduced in each arm to maintain similar polarization of both beams and to achieve high-contrast interference patterns. The object wave is superposed with the reference wave after a non-polarized beam splitter, and then the combined beams form the in-line hologram on the CMOS camera.

First, holograms with the object and then without the object are recorded. The holograms without the object carry information of the spherical phase arising from the microscope objective. The phase from the object holograms is calculated by

$$\Phi_O(x, y) = \tan^{-1}\left[\frac{I_1(x,y)-I_3(x,y)}{I_2(x,y)-I_4(x,y)}\right], \tag{2}$$

where, I_1, I_2, I_3, I_4 are the four recorded hologram intensities under corresponding phase shifts of the object under study.

The phase from the reference holograms which contains information about the additional phase term is calculated by

$$\Phi_R(x,y) = \tan^{-1}\left[\frac{R_1(x,y)-R_3(x,y)}{R_2(x,y)-R_4(x,y)}\right], \tag{3}$$

where, R_1, R_2, R_3, R_4 are the four reference intensities under corresponding phase shifts captured in the region without the object. The object phase is quantitatively reconstructed by subtracting the additional spherical phase term from the calculated reference phase using the formula

$$\Delta\Phi_O(x,y) = \Phi_O(x,y) - \Phi_R(x,y). \tag{4}$$

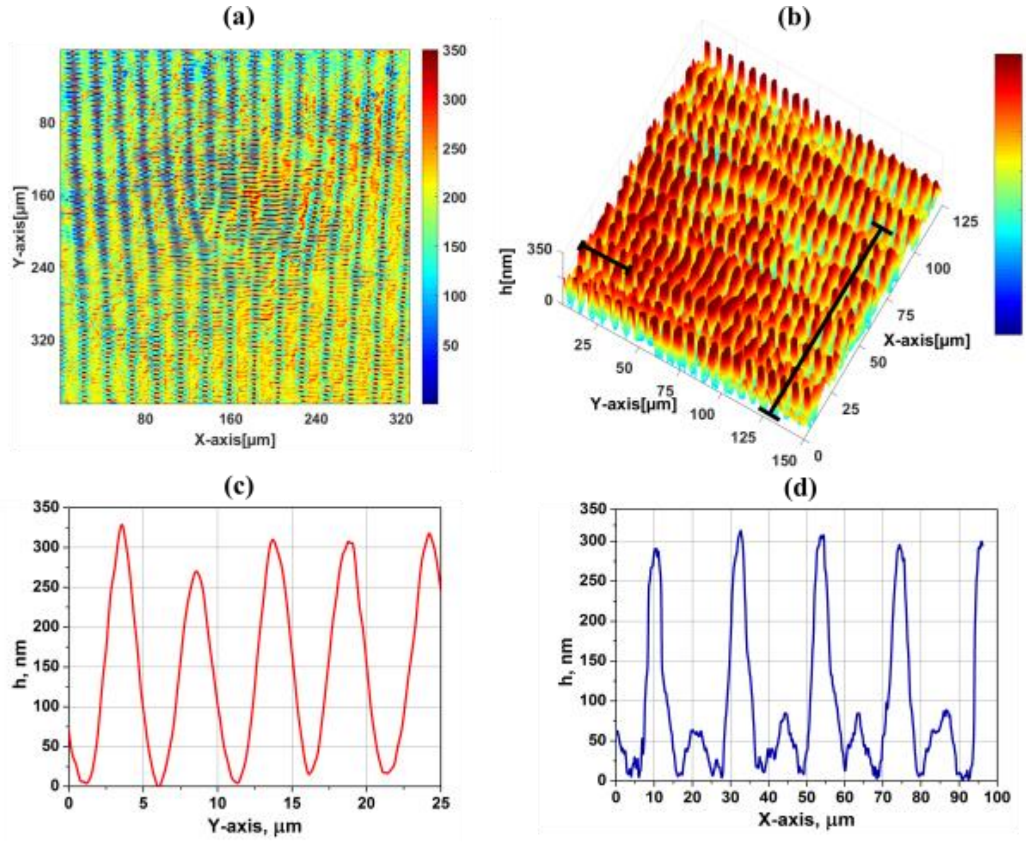

Fig. 11. PS-DHM investigation results. (a) 2D phase image of vortex DOE with TC=2, (b) 3D topography map (c) Y-axis cross-section (d) X-axis cross-section of the complex vortex DOE patterned by digital dual-beam PHR.

The reconstructed full-field surface of the complex vortex DOE inscribed via dual-beam SLM-based setup is pictured in Fig. 11. Two-fold symmetrically spaced fringes along the X- and Y-directions of the relief map result from the one-step PHR of two orthogonal modes. The cross-section built across the Y-axis, shown in Fig. 11 (c), reveals the relief slopes with a height of 300 nm and a frequency of 5.0 μm, created by the intersection of the SLM modulated beam and the object wave with polarization states of $+45^0$: -45^0, respectively. Surface profile extracted across the X-axis presented in Fig. 11 (d) denotes the azopolymer films structured by the digital fork-shaped hologram addressed to the SLM. A grating with two peaks was observed on the X-axis: the major peak with depth about 300 nm, and additional minor peaks with a height of 50 nm. The period of the patterned fringes is 20.0 μm and corresponds to the fork grating displayed on the SLM. The origin of minor peaks could be the result of the refractive index modulation in the azopolymer films.

The PhD thesis ends with the description of the general conclusions and recommendations of the research work.

GENERAL CONCLUSIONS AND RECOMMENDATIONS

The development of digital holographic microscopy as an optical instrument has an important role in the exploration, understanding and advancement/breakthrough of nanotechnology and innovative optical materials. At present, continuous enhancement of microscopic tools is mandatory in order to support the needs of micro-nanotechnology. In this regard, the main focus of this thesis was the improvement of the digital holographic microscope including hardware and software components for quantitative investigation of the diffractive optical elements on a nanometer scale. Another emphasis was the design of complex diffractive optical elements using analog and digital methods for polarization holographic recording on chalcogenide glasses nanomultilayers and azopolymer thin films. For both purposes, new digital devices were introduced in optical setups, thus a detailed calibration of digitally controlled liquid crystal devices was necessary to be implemented. Direct one step recording of DOEs makes it possible to preserve original calculated pattern, in contrast to etching procedures. The plasma and wet etching methods often used to create the relief distort, in one way or another, both the calculated drawing, the grooves form, and the depth of the designed relief. Laser power and the time of direct recording holography are more easily controlled than a number of etching parameters. The notable properties of DOEs recorded by polarization holography are that they diffraction efficiency is higher than ones recorded by scalar holography, and they are ready for use in an optical setup. As analog of free form optics with new, never seen before optical functionalities is a remarkable direction for DOEs in modern optical devices.

In the following paragraphs the main conclusions drawn from the research results are presented:

1. The results of the computational simulations of off-axis DHM configuration have proved that phase imaging enhancement can be obtained by adopting a sinusoidal phase grating mask to the off-axis lens less DHM setup. Due to the optimized diffraction grating parameters (period, phase and number of rotating steps) more information about the high spatial frequencies of the object spectrum is recorded in the CCD sensor from the diffracted orders. The root mean square error values of the phase reconstruction decreased with about 20%. At the same time, the results of the computational simulations of the phase-shifting DHM configuration show that by inserting a blazed diffraction grating in the optical setup the error of the phase reconstruction decrease by approximately 72%. This knowledge and the developed numerical tools could then be applied to record, reconstruct and process digital holograms. (Conclusions driven from the experiments described in Chapter 2 of the thesis)

2. Integration of liquid crystal devices in digital holographic optical systems presume the mandatory and important procedure of detailed phase and polarization calibration for each specific configuration. The calibration of LC devices shows that non-negligible errors are probable when controlling the phase and polarization of LC devices. Furthermore, the software procedures included in the SPAR calibration algorithm applied in this thesis demonstrate a compensation of the effects of external noise which reduce the requirements for stability of the interferometer. (Conclusions driven from the experiments described in Chapter 3 of the thesis)

a. The standard deviation of the phase data from individual pixels and their active area proves the fact that SLMs exhibit spatial variations the standard deviation of the active part of the SLM pixel is 0.5 radians smaller if compared to the phase of entire SLM array. This is the result of the spacing between pixels (the "dead zones") present on the SLM display, where the incident laser beam cannot control the phase modulation. The ad hoc calibration procedure of the SLM polarization modulation was implemented for evaluating the ellipticity and azimuth rotation angle of output beam as a function of gray level addressed to the SLM. The results point that SLM output polarization state is typically, elliptical. Only when 125 gray level blank screen is loaded on the SLM display it keeps the linear polarization of the input light. Also, it shows that the larger voltage applied to the SLM, the smaller becomes the light polarization modulation.

b. The calibration of the LCVR indicates that the phase shifting produced by the LCVR was possible by fine adjustment of the fast (slow) axis of the liquid crystal with the polarization plane of the incident beam without rotating the polarization plane. The obtained phase shift-volt characteristic of the LCVR is nonlinear. To produce accurate all-optical phase shifts, via DHM systems containing an LCVR, a linear approximation of the phase shift in the region 2-3.2V of the applied voltages was build. The advanced PS-DHM with the incorporated LCVR was developed and implemented for non-contact, all-optical investigation of the phase and surface relief transformations on azopolymer thin films and ChG NML.

3. Through analog and digital PHR, the direct (one-step) method of forming multifunctional DOEs on the surface of ChGs nanomultilayers and azopolymer thin films was achieved, which is the result of the mass transfer induced by the vectorial character of holography in polarized light (vectorial). Analog PHR produces DOEs with a high diffraction efficiency with a maximum of 33% achieved in this thesis on azopolymer thin films, while digital PHR enabled designing of DOEs with vortex geometries capable of generating spatially

multiplexed beams with phase singularities, but the diffraction efficiency of these DOEs is not more than 1%. (Conclusions driven from the experiments described in Chapter 4 of the thesis)

4. Off-axis and phase-shifting DHM configurations are applicable for the investigation of refractive index and surface relief maps of various DOEs recorded on azopolymers thin films and ChGs nanomultilayers. A hologram processing algorithm with SPAR reconstruction was developed and applied for the off-axis and phase-shifting digital holographic setups, capable of reconstructing the phase from noisy data (holograms). A liquid crystal variable retarder was integrated in the imaging system of the phase-shifting DHM for the enhancing the performance and stability of the microscope. (Conclusion driven from the experiments described in Chapter 5 of the thesis)

5. The proposed PS-DHM configuration was proven to be successfully used as a versatile metrological tool for 3D quantitative measurements of complex structures of DOEs including transparent surface relief deformations on a nanometer scale. On the bases of these results two assumptions have been made. First, we assume that the variations in the results may be due to the refractive index changes also arising in bulk of azopolymer thin films and ChG NML during exposure. Second, the non-uniform modulations of the relief depth of the DOEs caused by the Gaussian beam distribution makes it difficult to investigate the patterns in the area with maximal depth modulation. Therefore, further investigations in this field would increase the knowledge on the mechanisms of photoinduced phenomena in azopolymer thin films as well as the photonic applications of these materials. (Conclusion driven from the experiments described in Chapter 5 of the thesis)

RECOMMENDATIONS

Considering the conclusions above, the following recommendations for further research are highlighted:

1) Automatization of the phase reconstruction process, by excluding manual/individual introduction of the digital holograms in the MATLAB software and adjustment of algorithm parameters. Implementation of artificial intelligence algorithms is recommended for these procedures.

2) Implementation of multiwavelength DHM for broadening the application of the advanced optical system as it will expand absolute distance/phase measurements and diversify

the objects under study. In addition, multiwavelength DHM allows changing the resolution of microscope.

3) The development of a complex optical system (a combination of DHM with a PHR setup) capable of simultaneous and real time DOE recording and investigation of phase, surface relief growth and diffraction efficiency would facilitate a profound understanding of photoinduced phenomena and increase the accuracy of the investigations. The development of DHM in reflection mode would allow more complete examination of films and opaque objects.

4) Extended research on the input of surface relief and refractive index changes into the phase modulation of the azopolymer thin films and ChGs NML would further facilitate complete understanding of the light-matter interactions as well as the photonic applications of these materials.

5) Development of a compact DHM system for easier transportation and capable of adapting to various measurement tasks. This can be achieved by implementing advanced digital devices as SLM and LCVR (suggested in this thesis) that are capable to efficiently execute tasks and replace analog optical components.

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ANNOTATION

to the thesis „Development of digital holographic microscope for the investigation of diffractive optical elements recorded on chalcogenide glasses and azopolymer thin films", presented by Veronica Cazac for conferring the scientific degree of Ph.D. in Physics, Specialty 134.01 Physics and technology of materials, Chişinau, 2023.

The thesis has been written in English language and consists of the introduction, 5 chapters, general conclusions and recommendations, and the list of 214 references. The thesis contains 128 pages of basic text, 85 figures and 62 formulas. The results presented in the thesis are published in 17 scientific publications and presented at 16 international conferences.

Key words: digital holographic microscope, diffractive optical elements (DOEs), chalcogenide glasses (ChGs), azopolymers (APs), liquid crystal spatial light modulator and variable retarder, phase mask.

The goal: Quantitative analysis of key parameters of different types of diffractive optical elements, patterned on ChGs nanomultilayers and AP thin films by polarization holographic recording via improved hardware and software configuration of digital holographic microscope (DHM).

Research objectives: The development of numerical algorithms for phase reconstruction from acquired digital holograms of DOEs recorded on photosensitive ChGs nanomultilayers and AP thin films and subsequent mapping of their surface relief and refractive index; the modification of the optical part of the DHM in the off-axis and phase-shifting configurations and integration of liquid crystal devices; the calibration of phase and polarization of the liquid crystal devices implemented in digital holographic setups; the development of analog and digital holographic techniques for one-step polarization holographic recording (PHR) of different types of DOEs on nanomultilayers structures from ChGs and carbazole-based APs; the application of the developed DHM with the integrated tools for characterization of the created DOEs.

Scientific novelty and originality of the results: the improved version of the DHM software and hardware configuration has been demonstrated as a versatile instrument for the investigation of DOEs on ChG nanomultilayers and AP thin films; analog and digital PHR are new methods that allowed the photo-induced formation of complex DOEs on ChG nanomultilayers and azopolymer thin films.

The main scientific problem solved consists in elaborating of multifunctional DOEs patterned on ChGs nanomultilayers and azopolymer thin films via analog and digital PHR, and nanoscale investigation of their optical and physical parameters by high performance digital holographic microscope.

Theoretical significance and applicative value: of the work is that continuous enhancement of microscopic tools is mandatory in order to support efficient investigations in different fields as photonics, material science, biomedicine, etc. Therefore, the optical system of DHM including hardware and software components was improved for efficient investigation of the diffractive optical elements on a nanometer scale. The capability to create and perform quantitative analysis of multifunctional DOEs make them attractive in a variety of applications, including aberration correction, sensitive microscopy, augmented reality, imaging systems, solar energy and more.

The implementation of the scientific results: the research presented in this thesis has been successfully implemented in the framework of the EU project nr. 687328 (2016-2018), bilateral moldo-belarous project ANCD 19.80013.50.07.04A/BL (2020-2021), national project ANCD 20.70086.16/COV (2020-2021), national project 20.80009.5007.03 (2020-2023) and international project ANCD 21.80013.5007.1M (2021-2023), and may be further used for educational purposes.

ADNOTARE

la teza „Dezvoltarea microscopului holografic digital pentru investigarea elementelor optice de difracție înregistrate pe straturi subțiri de sticle calcogenice și azopolimeri", elaborată de Veronica Cazac pentru conferirea gradului științific de doctor în științe fizice la specialitatea 134.01 Fizica și tehnologia materialelor, Chișinău, 2023.

Teza este scrisă în limba engleză și constă din introducere, 5 capitole, concluzii generale și recomandări, și bibliografia din 214 titluri. Teza conține 128 pagini de text de bază, 85 figuri și 62 formule. Rezultatele prezentate în teză sunt publicate în 17 lucrări științifice și prezentate la 16 conferințe.

Cuvinte cheie: microscop holografic digital (MHD), elemente optice de difracție (EOD), sticle calcogenice (SC), azopolimeri (AP), modulator spațial de lumină cu cristale lichide și retarder variabil, mască de faza.

Scopul tezei: Analiza cantitativă a parametrilor cheie a diferitor tipuri de EOD, înregistrate pe nanomultistraturi de SC și straturi subțiri de AP utilizând metoda holografică cu lumina polarizată (HLP) cu ajutorul configurației hardware și software îmbunătățite a MHD.

Obiectivele tezei: Dezvoltarea algoritmilor de reconstrucție numerică pentru reconstrucția fazei din holograme digitale ale EOD înregistrate pe nanomultistraturi fotosensibile de SC și straturi subțiri de AP și măsurarea topografică a suprafeței precum și a indicelui de refracție a acestora; modificarea schemei optice a MHD în configurația „off-axis" precum și în configurația „phase-shifting" și integrarea dispozitivelor cu cristale lichide; calibrarea fazei și polarizării dispozitivelor cu cristale lichide implementate în configurații holografice digitale; dezvoltarea tehnicilor analogice și digitale pentru înregistrarea HLP printr-o singură etapă a diferitelor tipuri de EOD pe nanomultistraturi din SC și AP pe bază de carbazol; aplicarea MHD dezvoltat cu instrumentele integrate pentru caracterizarea EOD create.

Noutatea științifică și originalitatea rezultatelor: versiunea îmbunătățită a configurației software și hardware a MHD a fost demonstrată ca un instrument versatil pentru investigarea EOD pe nanomultistraturi de SC și a straturilor subțiri de AP; Înregistrarea HLP analog și digitală sunt metode noi care au permis formarea foto-indusă a EOD complexe pe nanomultistraturi de SC și straturilor subțiri de AP.

Problema științifică soluționată constă în elaborarea EOD multifuncționale înregistrate pe nanomultistraturi de SC și straturi subțiri de AP prin înregistrarea HLP analog și digitală precum și investigarea la scară nanometrică a parametrilor optici și fizici a acestora folosind MHD de înaltă performanță.

Semnificația teoretică și valoarea aplicativă: a tezei de doctor constă în îmbunătățirea continuă a microscopului ca instrument pentru efectuarea cercetărilor eficiente în diferite domenii precum fotonica, știința materialelor, biomedicină etc. În acest scop, sistemul optic al MHD, inclusiv componentele hardware și software, au fost îmbunătățite pentru investigarea eficientă a EOD la scară nanometrică. Posibilitatea de a crea și de a efectua analize cantitative a EOD multifuncționale le face atractive pentru o gamă largă de aplicații, inclusiv corectarea aberațiilor (erorilor), microscopia sensibilă, realitate augmentată, sisteme de imagistică, energie solară și multe altele.

Implementarea rezultatelor științifice: studiile prezentate în această teză au fost cu succes implementate în cadrul proiectului European nr. 687328 (2016-2018), proiectului bilateral moldo-belorus ANCD 19.80013.50.07.04A/BL (2020-2021), proiectului național ANCD 20.70086.16/COV (2020-2021), proiectului național 20.80009.5007.03 (2020-2023) și proiectului internațional ANCD 21.80013.5007.1M (2021-2023), și pot fi utilizate cu scop didactic pentru studenții ciclului universitar și post-universitar.

АННОТАЦИЯ

к диссертации « Разработка цифрового голографического микроскопа для исследования дифракционных оптических элементов на тонких пленках халькогенидных стекол и азополимеров», представленной Вероникой Казак на соискание ученой степени доктора физических наук по специальности 134.01 Физика и технология материалов, Кишинев, 2023

Диссертация написана на английском языке и состоит из введения, пяти глав, общих заключений и рекомендаций, и списка цитируемой литературы из 214 источников. Диссертация содержит 128 страниц базового текста, 85 графиков и 62 формул. Результаты диссертационной работы опубликованы в 17 научных публикациях и представлены на 16 конференциях.

Ключевые слова: цифровой голографический микроскоп (ЦГМ), оптические дифракционные элементы (ДОЭ), халькогенидные стекла (ХС), азополимеры (АП), жидкокристаллический пространственный модулятор света и фазовая пластинка, фазовая маска, цифровая обработка изображений.

Цель диссертации: Количественный анализ ключевых параметров различных типов дифракционных оптических элементов, нанесенных на многослойные наноструктуры ХС и тонкие пленки АП, методом поляризационной голографической записи (ПГЗ) с использованием усовершенствованной аппаратной и программной конфигураций ЦГМ.

Задачи диссертации: Разработка алгоритмов для реконструкции фазы из цифровых голограммам ДОЭ записанных на ХС и тонких пленках АП, и последующего картографирования их поверхностного рельефа и показателя преломления; модификация ЦГС во внеосевой и со сдвигом фазы конфигурациях и интеграция жидкокристаллических устройств; калибровка фазы и поляризации жидкокристаллических устройств; разработка аналоговых и цифровых методов одноэтапной ПГЗ; применение разработанного ЦГС с интегрированными инструментами для иследования харахтеристик созданых ДОЭ.

Научная новизна и оригинальность результатов: усовершенствованная версия программного и аппаратного обеспечения ЦГС была продемонстрирована как универсальный инструмент для исследования ДОЭ, записанных на наномногослойных ХС и тонких пленках АП; аналоговая и цифровая ПГЗ - это новые методы, которые позволили фотоиндуцированное формирование сложных ДОЭ.

Основная научная задача, решаемая диссертацией, заключается в разработке многофункциональных ДОЭ, нанесенных на наномногослойные ХС и тонкие пленки АП с помощью аналоговых и цифровых ПГЗ, и в исследовании их оптических и физических параметров в наномасштабе с помощью высокоэффективного ЦГС.

Теоретическая значимость и прикладная ценность: заключается в том, что постоянное совершенствование микроскопических инструментов является обязательным для поддержки исследований в различных областях, как например фотоника, материаловедение, биомедицина и т. д. Оптическая система ЦГС была усовершенствована для эффективного исследования ДОЭ. Возможность создания и проведения количественного анализа многофункциональных ДОЭ делает их привлекательными для различных применений, включая микроскопию, системы визуализации, солнечную энергетику и многое другое.

Внедрение научных результатов: представленные в настоящей диссертации результаты были успешно применены в рамках Европейского проекта нр. 687328 (2016-2018), в молдавско-белорусском двустороннем проекте ANCD 19.80013.50.07.04A/BL (2020-2021), в национальном проекте ANCD 20.70086.16/COV (2020-2021) и в 20.80009. 5007.03 (2020-2023) и международном проекте ANCD 21.80013.5007.1M (2021-2023).

CAZAC VERONICA

DEVELOPMENT OF DIGITAL HOLOGRAPHIC MICROSCOPE FOR THE INVESTIGATION OF DIFFRACTIVE OPTICAL ELEMENTS RECORDED ON CHALCOGENIDE GLASSES AND AZOPOLYMER THIN FILMS

134.01 –Physics and technology of materials

Abstract of the PhD dissertation in physics

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