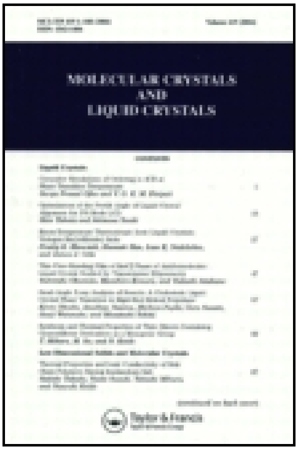

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Relation Between the Supermolecular Structure and Optical Properties of Liquid Crystal - Photopolymer Composite Films

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Composite materials with spatially alternating polymer and nematic liquid crystal layers have been obtained. The tetraacrylate pentaerrythritol monomer, methylene blue and nematic liquid crystals were used as the pre-polymer composition. Scanning electron microscopy results show that the supermolecular structure holographically formed in the film depends on the relation between the nematic liquid crystal and dye concentrations in the pre-polymer composition and the curing energy. This system allows preparation of electrically switchable transmission gratings with first-order diffraction efficiencies up to 30–55%. The formed gratings could be switched from diffracting to transparent state at 4.5–7.5 V/μm. The turn-on and turn-off times were 200–300 μs and 1.2–3 ms, depending on the particular composition of the initial composition. Stability of grating characteristics was examined over a 1.5-year period of grating storage.

Keywords: nematic liquid crystal – photopolymer composite; ordered periodic structure; switchable transmission gratings

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INTRODUCTION

Presently, holographic photopolymer materials with nematic liquid crystals (NLC) keep attracting considerable interest. This interest is related with the possibility of using such materials in thin-film displays, optical switches, diffractive optical elements, etc. [1–4]. The distinguishing feature here is the possibility to form a spatially periodic structure in a thin film of such a material (from several micrometers to several tens of micrometers) using spatially anisotropic photopolymerization (the so-called holographic technique). The spatially periodic structure results in refraction-index modulation between the polymer- and NLC-rich planes in the volume of the film, and the film therefore can be considered and used as a phase-type diffraction grating possessing a certain spatial period, or spacing, and some diffraction efficiency. Such a film, sandwiched between two plane-parallel glass substrates covered with conducting layers (ITO), constitutes an experimental cell. An external electric field applied to the cell will reorient NLC molecules in conventional planes, thus switching the system to the transparent state.

The characteristics of diffraction gratings prepared in this manner depend on many factors. The studies reported by many authors show that among critical factors here are the types of components used to prepare the initial composition, energy characteristics of the curing radiation, and the material morphology [1,3,5–12]. In spite of the great body of reported studies in which holographically formed polymer dispersed liquid crystals (H-PDLC) were examined, the noise figures of such materials still remain a scantily investigated matter. In H-PDLC, light is scattered both by the material microstructure in individual layers and by the holographically formed diffracting structure. To reduce scattering-induced noise, one has to reduce the size of structural formations in the NLC-rich planes whose surface scatters light. Yet, downsizing of NLC droplets, which represent the most frequent type of structural formations in H-PDLC, require higher switching field to be used [13]. Recently, the authors of [9,11,12] showed it possible to obtain diffraction gratings composed of alternating polymer planes and planes formed by a uniformly oriented NLC phase. Since the individual NLC layers in such gratings are structurally uniform, the losses for scattering within the layers turn out to be minimum and the switching field, low (3–8 V/μm). On the other hand, in the gratings prepared at elevated temperatures, detrimental effects become more pronounced due to shrinkage and mutual diffusion of components at the cooling stage, resulting in deterioration of the final structure of the formed grating and worsened performance characteristics [9].

The purpose of the present study was to prepare a diffraction grating at room temperature from a model composition made up by a monomer, a photo-initiator, and an NLC (a), to study the effect due to the proportion between the components in the initial mixture, and the effect of curing energy on the film morphology (b), to analyze the influence of the morphology on the diffraction efficiency and time characteristics of obtained films (c).

FILM FORMATION AND EXPERIMENTAL METHODS

The initial pre-polymer composition contained an NLC, a multifunctional acrylate monomer and a photo-initiating system. The NLC was a commercially available eutectic mixture of cyanobiphenyls and cyclohexacarbon acid ethers ($n_0 = 1.52$, $n_e = 1.69$, $\Delta n = 0.17$, $\Delta\varepsilon = 9.9$), and the monomer, pentaerythritol tetraacrylate (produced by "Aldrich"). N-vinylpyrrolidynon produced by the same firm was used both as a cross-linking component and as a solvent for the dye and co-initiator. To prepare gratings, compositions with the photo-initiating systems were used in which the dye and the co-initiator were respectively methylene blue and triethanolamine. The NLC concentration in the composition varied from 0 to 50 wt.%. The dye concentration was 0.1–0.4 wt.%. The choice of the components, motivated by the desire to obtain a composition with a maximum photosensitivity in the spectral region 650 to 680 nm, was made considering the relatively high mutual room-temperature solubility. The chosen range of dye concentration was such that to allow preparation of composite materials with various degrees of structuring, largely defined by the polymerization rate of the initial composition.

The photopolymer composition was prepared as a homogeneous solution. The thickness of experimental cells was of 10 μm. Anisotropic photopolymerization was initiated by light with a periodic intensity distribution. The light source was an ML120G laser diode with the wavelength of $\lambda_r = 658$ nm. The curing energy was of 200–2700 mJ/cm^2. The interference of two plane waves produced a periodic light intensity distribution in the sample. The spatial period of the interference pattern was 1.1 μm.

To accomplish the polymerization after the laser curing, all cells were treated with a daylight lamp. All fabrication steps were carried out at room temperature.

The switching tests were performed using voltage pulses of amplitude up to 200 V. The tests were performed under strictly fulfilled Bragg condition, with a He-Ne laser as a light source (the reading wavelength was $\lambda = 632.8$ nm). The obtained angular dependencies of light transmission enabled estimation of the spatial period of the gratings, their diffraction efficiency, the refractive index modulation amplitude, and the scattering induced noise [14]. The first-order diffraction efficiency of the gratings used here, η_1, is defined as $\eta_1 = I_1/I_{inc}$, where I_{inc} and I_1 are respectively the intensity of the incident probing beam and that of the first–order diffraction beam. The zero–order efficiency η_0 (i.e., zero-order transmittance) was found as the ratio between the non-diffracted transmitted intensity I_0 and the incident intensity I_{inc}. The reorientation field and the switching times, namely, the turn-on time (on application of a voltage) and the turn-off time (on removal of the voltage) were found from the measured electro-optical characteristics. The contrast ratio was determined as the ratio between the maximum zero-order diffraction efficiency in the transparent state and the diffraction efficiency in the off-state. For the first diffraction order, the contrast ratio was defined as a reciprocal quantity, i.e., as the ratio between the maximum diffraction efficiency in the off-state and the minimum diffraction efficiency in the transparent state [15].

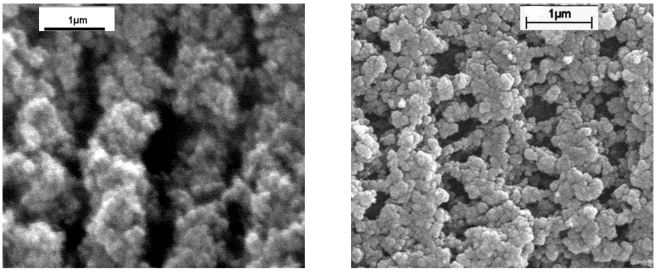
The morphology of the formed gratings was examined with the help of a scanning electron microscopy (LEO 1430 and REM-100U). The preparation of samples included their treatment in hexane, aimed at removing the NLC out of the photopolymer matrix, and their subsequent annealing at 80°, followed by shadowing of the samples with 150–200 nm of Au.

EXPERIMENTAL RESULTS

It was established in previous studies that diffraction gratings with different characteristics could be formed in a narrow range of NLC concentration in photopolymer composition under study [14]. In this connection, in the present paper we report comparative characteristics of the gratings prepared from compositions with different NLC concentrations (37 or 50%) and different dye concentrations (0.1 or 0.4%) exposed to different curing energies.

Effect of the Initial Composition of Mixture and the Curing Energies on the Grating Morphology and Diffraction Efficiency

Figures 1 (a) and 2 (a) show the polymer structures of gratings prepared from mixtures with different NLC concentrations. The curing conditions and the dye concentrations were identical. It is seen from the figures that at 37% of NLC, a diffraction grating with a clearly

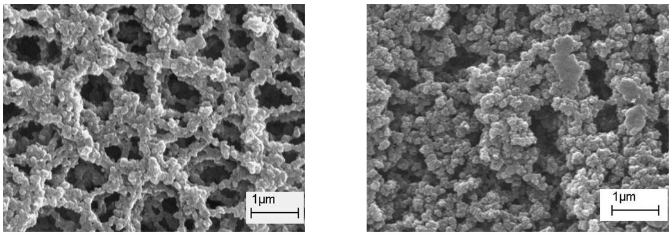


(a) (b)

FIGURE 1 SEM images of polymer structures. The light and dark strips are respectively the polymer layers and the regions with removed NLC. The initial composition contained 0.1% (a) and 0.4% (b) of dye and 37% of NLC. The curing energy was 900 mJ/cm^2.

defined periodic structure was formed. Less pronounced degradation of the grating structure was observed with dye concentration increased to 0.4% in the composition with 37% of NLC (Fig. 1 (b)). It is evident that the dye concentration is accompanied by a higher polymerization speed, which does not allow the full separation of the polymer and NLC phases and therefore violates the periodic structure. This results in the decreased diffraction efficiency.

The first-order diffraction efficiency (η_1) in the obtained gratings also depends on the curing energy. For the gratings prepared from the compositions with 37% NLC and 0.1% dye, the maximum efficiency η_1 was achieved at 600–1000 mJ/cm^2 (Fig. 3, curve 1). Nevertheless, with the same curing conditions, an increase in the dye concentration to 0.4% resulted in a 20–30% decrease of η_1 (Fig. 3, curve 2). Irrespective of curing conditions and dye concentration, an



(a) (b)

FIGURE 2 SEM images of polymer matrices. The curing energy was 900 (a) and 1800 (b) mJ/cm^2. The initial composition contained 0.1% of dye and 50% of NLC.

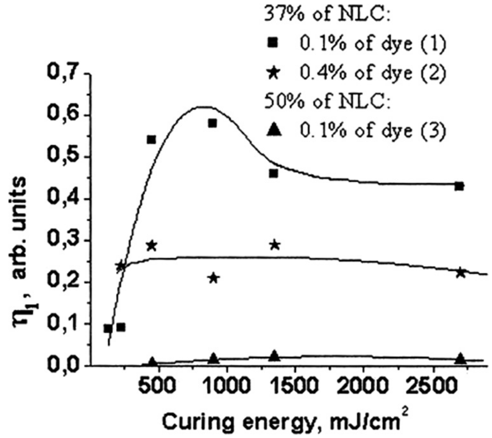


FIGURE 3 First-order diffraction efficiency (η_1) of gratings versus curing energy for different dye and NLC concentrations: 0.1% (1) and 0.4% (2) of dye, composition with 37% of NLC; 0.1% of dye (3), composition with 50% of NLC.

increase in the NLC concentration to 50% had a more pronounced effect on η_1 (Fig. 3, curve 3). In this case, the efficiency η_1 was low, about 1–2%. The latter may result from the absence of clearly defined spatially periodic structure in the film (Fig. 2 (a) and (b)).

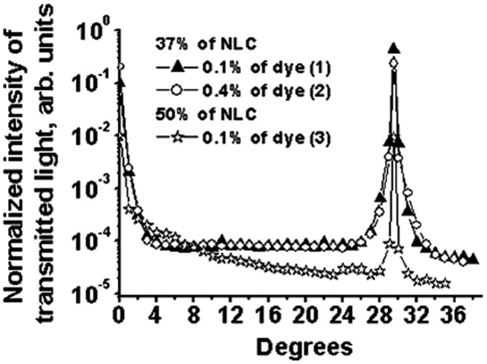


FIGURE 4 Effect of NLC and dye concentrations on the angular dependences of normalized transmitted light intensity: (1) 37% of NLC, 0.1% of dye; (2) 37% of NLC, 0.4% of dye; (3) 50% of NLC, 0.1% of dye. The curing energy was 1350 mJ/cm^2.

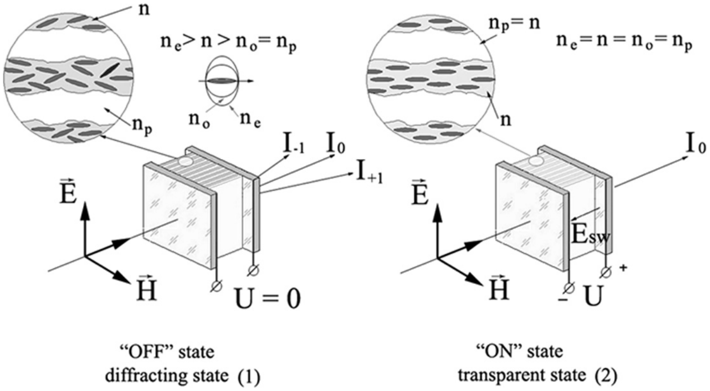
Thus, only a restricted interval of curing energies can be used to prepare the gratings with high diffraction efficiencies from the initial photopolymer mixture with the certain NLC and dye concentration.

The angular dependence of the normalized transmitted light intensity for the gratings obtained from the compositions with different NLC concentrations was examined (Fig. 4, curves 1 and 3). It is possible to control the noise figures of the gratings by varying the NLC concentration in the initial photopolymer composition. However, the variation of the dye concentration (at NLC concentration kept unchanged) does not affect the noise figures of the gratings (Fig. 4, curves 1 and 2).

Electro-Optical Characteristics of the Gratings

To examine the field modulation of the diffraction efficiency, we tested gratings prepared with curing energies 900–1350 mJ/cm^2. In this range of curing energies, gratings with the highest first-order diffraction efficiency for the system under study could be formed.

Figure 5 shows a schematic illustrating the electrical switching of an experimental cell between the diffracting (1) and non-diffracting (2) states. With no voltage applied, the cell is operated in the diffraction mode (with maximum first-order diffraction efficiency for the p-polarized beam), presenting therefore a transmission-type phase



"OFF" state
diffracting state (1)

"ON" state
transparent state (2)

FIGURE 5 Schematic of the experimental cell with a written grating and its operating principle: (1) diffracting state; (2) non-diffracting (or transparent) state. The upper part of the figure schematically shows the structure of the diffraction grating formed by alternating NLC and polymer layers and the effect due to reorientation of NLC molecules in NLC-rich layers.

grating. If the ordinary refraction index n_0 of the NLC matches the refraction index n_p of the polymer, the cell can be switched into a transparent state by applying an electric field across the cell (if, like in the present study, an NLC with positive dielectric anisotropy $\Delta\varepsilon$ is used). The applied voltage should be sufficiently high to reorient the NLC molecules in conventional planes of the grating. In this state (2), the refractive index modulation in the bulk of the film almost vanishes for a probing laser beam incident onto the cell at the Bragg angle. This means that the first-order diffraction efficiency is minimum in this state. Simultaneously, the zero-order transmittance increases to its maximum value. Thus, the grating can be switched into the transparent, or non-diffracting, state.

The performed study showed that the electro-optical parameters of the gratings (switching voltage, contrast ratio, and turn-on and turn-off times) depend on the initial mixture composition.

Figure 6 shows the zero-order transmittance (η_0) and the first-order diffraction efficiency (η_1) versus the applied voltage. These data prove the refractive-index modulation Δn to exist in the volume of the film under the applied electric field. With the dye concentration increased from 0.1 to 0.4%, the refractive index modulation effect becomes less pronounced (see Fig. 6 (a)) in the grating prepared from compositions with 37% of NLC, presumably because of the less degree of phase separation between the NLC and polymer phases resulting from the higher polymerization rate. It was shown in [14,16] that the maximum value of Δn ($\Delta n \approx 0.015$) without an electric field could be achieved in the gratings with a spatial period of 1.1 μm prepared from the mixture of 0.1% dye concentration. The dye concentration being increased to 0.4%, Δn reduces to 0.009. Accordingly, the range in which η_1 varies from its maximum to minimum value under the applied electric field also becomes narrower. As a consequence, the contrast ratio (CR) found from the field dependencies of η_1, decreases. In this case, the CR decreases from 11:1 to 7:1 as the dye concentration increases to 0.4%. For η_0, the effect is unnoticeable. With increasing dye concentration, the corresponding value CR = 6:1 remains unchanged. This observation calls for further study. It is also seen from Fig. 6 (a) that an increased concentration of dye raises the reorientation field from 4.5 to 7.5 V/μm. Apparently, the formation of the gratings at room temperature (i.e., well below the isotropic – nematic transition) gives rise to a domain structure of NLC layers. In the samples with the increased dye concentration, somewhat smaller domains form, and the range of switching voltages increases.

A gradual increase of the switching voltage in time after initial fabrication of gratings from a composition with 0.1% of dye was observed.

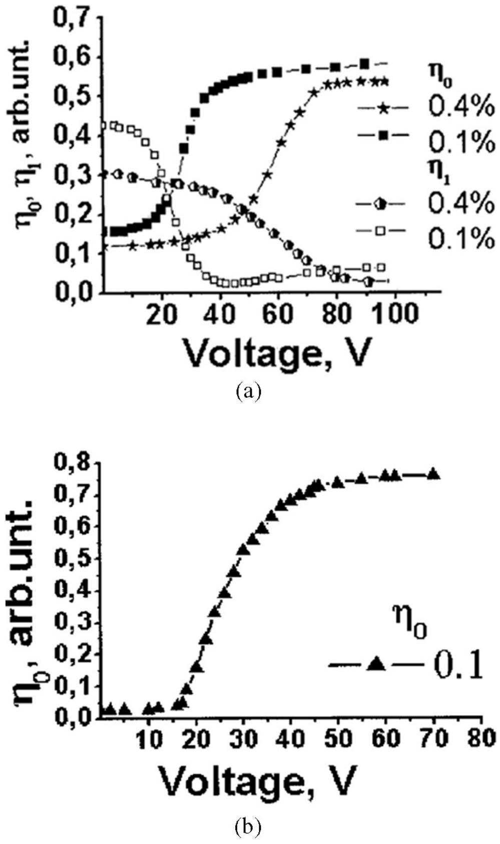
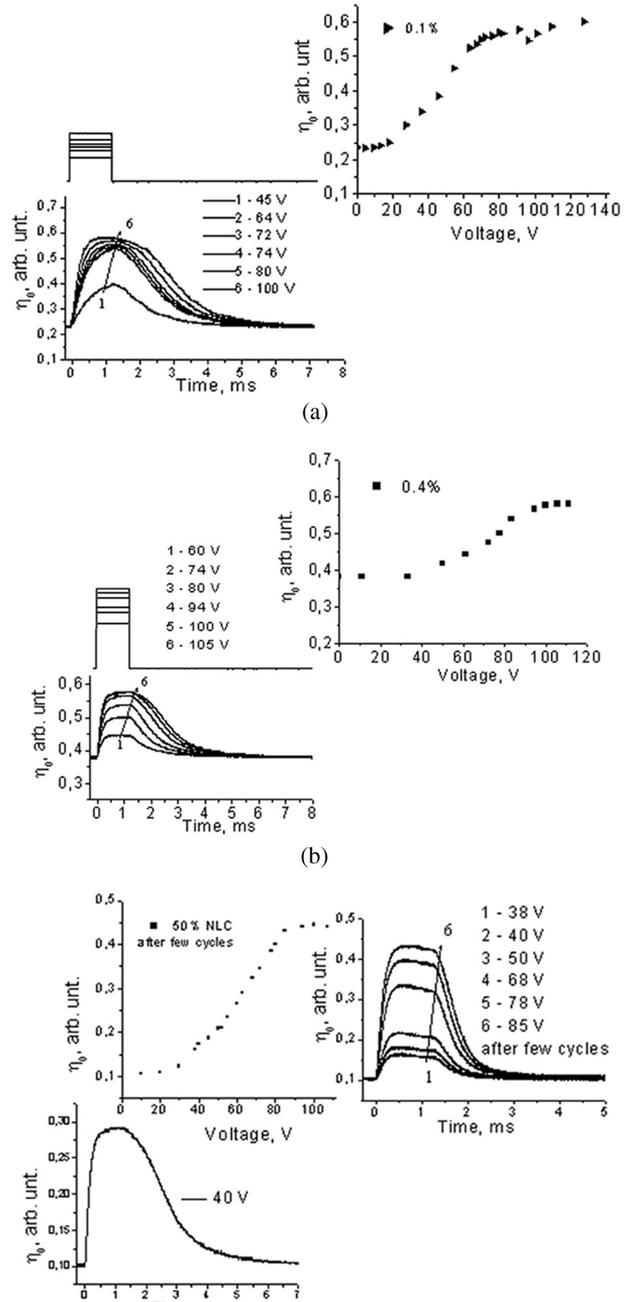


FIGURE 6 Effect of NLC and dye concentrations in the initial compositions on the field dependences of η_1 and η_0: (a) 37% of NLC, (b) 50% of NLC. The dye concentration is indicated in the graphs. The curing energy was 900 (a) and 1350 (b) mJ/cm^2.

For the gratings under study, the switching voltage increases approximately by 160% over a five-month period due to continuing polymerization (i.e., post-polymerization) after the grating formation. It was observed that the final switching voltage does not change over a 1.5-year period (see the insert to Fig. 7 (a)). In the case of gratings prepared from compositions with 0.4% of dye, the switching voltage rises by about 25% to remain unchanged during 1.5-year storage as well (see the insert to Fig. 7 (b)). Such a behavior of the electro-optical characteristics of this sample can be attributed to faster polymerization accomplished in a shorter time interval.


0.1%
η0, arb. unt
Voltage, V
1 - 45 V
2 - 64 V
3 - 72 V
4 - 74 V
5 - 80 V
6 - 100 V
η0, arb. unt.
Time, ms
0.4%
η0, arb. unt.
Voltage, V
1 - 60 V
2 - 74 V
3 - 80 V
4 - 94 V
5 - 100 V
6 - 105 V
η0, arb. unt.
Time, ms
50% NLC
after few cycles
η0, arb. unt.
Voltage, V
1 - 38 V
2 - 40 V
3 - 50 V
4 - 68 V
5 - 78 V
6 - 85 V
after few cycles
η0, arb. unt.
Time, ms
40 V
η0, arb. unt.
Time, ms

(a)

(b)

(c)

Irrespective of the dye concentration, with the NLC concentration increased to 50% the values of η_0 and η_1 at $U = 0\,V$ decrease to 1–2% because the grating periodic structure degrades substantially. Thus, such gratings can only be operated in one diffraction order, namely, in the zero diffraction order. A typical field dependence of η_0 for a grating prepared from a composition with 0.1% of dye is shown in Figure 6 (b). As is seen from Fig. 6 (a) and (b), for the gratings written with the identical dye contents (0.1%), an increase in NLC concentration makes the efficiency in the zero diffraction order η_0 vary in a broad range under applied electric field. In this case, the switching fields remain almost unchanged, whereas the CR increases to 38:1.

For gratings with the 50%-NLC, the time evolution of the electro-optical characteristics turns out to be weaker compared to the case of gratings prepared from compositions with 37% of NLC. This finding can be attributed to the fact that, as the NLC concentration rises, the polymerization and phase separation processes finish faster, with almost no post-polymerization observed. It should be noted that in this case the ordering of the NLC in the proper domains of the film structure is less defined than in the NLC-layers of the periodic structure prepared from 37%-NLC compositions. However, as the time passes, some partial NLC-ordering occurs, and the zero-order diffraction efficiency η_0 at 0 V rises to 8–10%, resulting in a lower contrast ratio. It should be noted that the first-order diffraction efficiency does not increase either, remaining at the level of 1–2%. Here, the reorientation field remains unchanged after the first switching (as previously, the saturation is observed at about 40 V), and then, after several switching cycles the reorientation voltage increases to 80 V (see the insert to Fig. 7 (c)).

Thus, more pronounced phase separation of the polymer and NLC in the film volume results in an increased switching field for all the gratings after storage during several months. Yet, the field modulation of the diffraction decreases in all the gratings after the longer storage time of 1.5 years. Very probably, the latter is related with slow degradation of the grating periodic structure and reduced refraction index modulation between the polymer and NLC layers for all gratings

FIGURE 7 Effect of NLC and dye concentrations in the initial compositions on switching times after1.5-year storage of the gratings: (a) 0.1% of dye, 37% of NLC; (b) 0.4% of dye, 37% of NLC; (c) 0.1% of dye, 50% of NLC. The switching times are presented for the zero-order transmittance η_0. The curing energy was 1350 mJ/cm^2. The pulse duration was 1.24 ms. Inserts show the field dependences of η_0 after 1.5-year storage. Figure 7 (c) shows switching times and the field dependence of η_0 measured after several switching cycles.

examined in the present study (indeed, the grating is spatially nonuniform, and the interfaces between the polymer and NLC layers are not sharp).

Switching Times of the Gratings

The effect due to the layered periodic structure is also manifested in the switching times of the samples. Following two months after the preparation, the gratings show a turn-on time of 200–300 μs and a turn-off time of 1.2–3 ms depending on the particular composition of the initial mixture with 37% of NLC. With the NLC concentration increased to 50%, the turn-on and turn-off times increase to 500 μs and 5 ms, respectively. As it was noted above, the latter effect can be attributed to the degradation of the periodic structure for this concentration of NLC.

The switching times were also measured after 1.5-year storage of the gratings (Fig. 7). As it was noted above, Figure 7 shows the field dependences of the zero-order diffraction efficiency η_0 (inserts to Fig. 7 (a), (b), (c)). Similarly to other parameters, we observed changes in the turn-on and turn-off times of the gratings due to post-polymerization, and also slow time changes of NLC domain sizes, domain shape anisotropy, and NLC ordering in domains. For the gratings prepared from compositions with 0.1% of dye, the turn-on times increase to 800 μs, the turn-off times, to 5 ms, and for the gratings prepared 0.4-% compositions, these times increase to 500 μs and 3.5 ms, respectively (see Fig. 7 (a) and (b)). When the pulse with the amplitude of 76 and 84 V is off, one can observe a memory effect in the gratings prepared from compositions with 0.1 and 0.4% of dye. The memory effect is typical of composites prepared from compositions containing the multifunctional acrylate monomer [7,13]. In the case of gratings prepared from compositions with 50% of NLC, the turn-on times are 600–800 μs, and turn-off times are 5.5 ms (Fig. 7 (c)) after the first application of the electric field. The turn-off time, equal to about 5.5 ms at the first switching, decreases after several switching cycles and then relaxation to the initial state takes place in about 1.5 ms (see the insert to Fig. 7 (c)). The effect seems to result from charging processes proceeding during the application of high-intensity electric fields (8–10 V/μm) to the gratings. It should be noted that in the case of gratings written in compositions with 37% of NLC, the effect due to induced field is almost unnoticeable even with high-intensity electric fields applied to the samples.

Taking into account the specific features of the structure of the formed gratings (alternating NLC and polymer layers), and also the domain structure of NLC layers, we can conclude that the switching times are largely defined by the rotational viscosity of NLC and by the characteristic size of domains. The performed measurements of switching times showed that these times are determined by the total effect of the surface and bulk rotational viscosity of NLC (gentle sloping and sharply increasing portions of the time characteristic). The surface viscosity is much greater than the bulk viscosity due to the proximity of the polymer boundary at which the polymer partially fixes the NLC molecules (the grating front is smooth). Thus, it is the balance between the "fast" and "slow" components that defines the turn-on and turn-off times of the grating. It should be noted that the switching times substantially depend on the duration and amplitude of applied electric pulses. A study of time characteristics of formed gratings is under way.

It can be concluded from the obtained data that the "lifetime" of gratings prepared from initial compositions with 37% of NLC is longer, and stability of their electro-optical characteristics, better than those of gratings prepared from initial compositions with 50% of NLC.

CONCLUSIONS

This study has demonstrated a relation existing between the supermolecular structure and the optical properties of a periodically structured composite material obtained by spatially non-uniform photopolymerization. The tetraacrylate pentaerrythritol monomer, methylene blue and nematic liquid crystals were used as the pre-polymer composition. Experimental results show that variation of the concentrations of the components in the starting mixture affects the morphology of the material. There exists a certain restricted interval of curing energies suitable for fabrication of gratings from compositions with different NLC and dye concentrations. The structure of the constituent layers determines the switching voltage, the switching times, and the noise figures of the gratings. The examined system allows one to prepare electrically switchable volume phase gratings with first-order diffraction efficiencies up to 30–55%. The gratings could be switched from diffracting to transparent state by an electric field of 4.5–7.5 V/μm. Two months after the preparation, the turn-on and turn-off times were 200–300 μs and 1.2–3 ms, depending on the particular composition of the starting mixture. The diffraction properties have been preserved over a 1.5-year period, but the driving voltage and the switching times have increased. Stimulation of the phase separation of the NLC and polymer, and also adjustment of the polymerization rate through optimization of the initial composition of the starting photopolymer mixture and parameters of the curing process will make it possible to improve electro-optical parameters of the gratings, namely, to rise their diffraction efficiency and to reduce the reorientation field and switching times.

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