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Electro-optics and structure of polymer dispersed liquid crystals doped with nanoparticles of inorganic materials

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Abstract

Electro-optic characteristics and structural peculiarities of polymer dispersed liquid crystals (PDLC) doped with small amount ($\leq 3 \text{ vol.}\%$) of inorganic nanoparticles (NP) are considered. These composites were formed by the photoinduced phase separation of the LC–NP–Prepolymer mixtures. It is established that NP do not change essentially PDLC morphology of LC–Polymer samples. In the course of the phase separation, they are mainly involved with polymer, serving as building blocks for the polymer matrix. The large and/or strongly aggregating particles strengthen light scattering in the polymer that may enhance electrooptic contrast. In turn, the small and weekly aggregating particles modify the effective refractive index of the polymer phase without producing any noticeable optical inhomogeneity. The latter is used to tune the refractive index ratio of LC and polymer in order to meet conditions for the substantial reduction of off-axis haze in the field-on state.

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1. Introduction

Liquid crystal (LC) composites with the electrically controlled intensity of light scattering form real alternative to oriented LC layers widely used as basic elements of LCD and optical switches. The operation in the scattering mode gives a number of advantages. These devices do not set strong demands for the cell gap uniformity that is especially important for the large-area and flexible substrates. They do not need alignment layers, sets of polarizers and retardation films. Avoiding polarizers, scattering type devices demonstrate high brightness especially needed for the projection type LCD.

The well-known examples of light scattering LC composites are polymer dispersed LC (PDLC) [1-3] and filled LC [4–6]. The main reasons for light scattering in these sys-

tems are different. The PDLC composites scatter light because of the refractive index mismatch between LC and polymer, two adjacent droplets and within the droplets of LC [3]. The strong decrease of the light scattering in the field-on state is caused by the unidirectional alignment of LC within the drops and matching the refractive indices of LC and polymer. In contrast, the intense light scattering in filled LC is mainly caused by the orientational defects generated by the particles of filler in LC media. In the field-on state these defects practically disappear that results in a strong increase of sample transmittance. The advantages and disadvantages of PDLC and filled LC from the viewpoint of applications have been widely discussed [1–10].

In the present work we consider some hybrid of these systems—three-component composite consisting of LC, polymer (P) and nanoparticles (NP). Ternary phase diagram of LC–P–NP composites is presented in Fig. 1. The segments PNLC (polymer network liquid crystal [7]),

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Fig. 1. Scheme showing concentration ranges of LC–P, LC–NP and LC–NP–P composites.

PDLC and LC–NP (filled LC) schematically represent the concentration ranges of the two-component systems earlier studied. The two-dimensional areas NP–PNLC and NP–PDLC selected in Fig. 1 represent most interesting ranges of three-component samples. The area NP–PNLC corresponds to the polymer network liquid crystals filled with NP. The other concentration area, NP–PDLC, represents NP doped PDLC.

The NP–PNLC systems were earlier studied in [8–10]. It was shown that polymer network stabilizes LC–NP suspensions preventing phase separation especially active under the electric switching. This increases system's reliability and electro-optic contrast and thus excites application interest.

In the present research we are focused on the NP–PDLC samples. We show that doping with NP may substantially improve electro-optic properties of PDLC so that NP–PDLC samples can be regarded as robust materials for LC applications. We also propose the model of phase separation in the LC–P–NP system that provides good explanation of the electro-optic data.

2. Materials and methods

As liquid crystal and polymer, we used nematic LC mixture E7 from Merck and UV curable adhesive No65 from Norland Inc. This pair of materials was widely used before to prepare PDLC composites [11,12].

As NP filler we used two types of colloidal monodispersed NP: silica (SiO₂) NP with the sizes of 10–20 nm and antimony pentoxide (Sb₂O₅) NP with the sizes of 7– 11 nm. The SiO₂ and Sb₂O₅ NP were chosen, because their refractive indices match well ordinary and extraordinary refractive indices of LC E7. They were obtained from Nissan Chemicals Industries Ltd. as colloidal dispersions in methanol with a particles' content of 30 wt.% and 35 wt.%, respectively. Additionally we used TiO_2 nanoparticles with the size of 25–50 nm in the form of powder obtained from Nanophase Inc.

For the composite preparation we mixed LC, prepolymer composition and colloid (powder). The intensive mixing caused uniform dispersion of NP in LC and evaporation of alcohol. The complete removing of alcohol was achieved by applying vacuum to the suspension at 10^{-2} Torr over 0.5 h.

We operated with volume concentrations of NP, $\varphi_{\rm NP}$, and polymer, $\varphi_{\rm P}$, for the reasons discussed in the subsequent section. The $\varphi_{\rm NP}$ and $\varphi_{\rm P}$ concentrations were calculated as $\varphi_{\rm NP} = \frac{m_{\rm NP}/\rho_{\rm NP}}{m_{\rm NP}/\rho_{\rm NP}+m_{\rm P}/\rho_{\rm P}+m_{\rm LC}/\rho_{\rm LC}}$ and $\varphi_{\rm P} = \frac{m_{\rm P}/\rho_{\rm P}}{m_{\rm NP}/\rho_{\rm NP}+m_{\rm P}/\rho_{\rm P}+m_{\rm LC}/\rho_{\rm LC}}$, where $m_{\rm NP}$, $m_{\rm P}$ and $m_{\rm LC}$ are the masses, while $\rho_{\rm NP}$, $\rho_{\rm P}$ and $\rho_{\rm LC}$ are the densities of corresponding phases. In the present experiments, the concentration of NP was kept relatively low ($\varphi_{\rm NP} = 1-3$ vol.%), while the polymer concentration was varied within a wide range ($\varphi_{\rm P} = 5-50$ vol.%) to optimize conditions for PDLC morphology.

The drop of mixture was placed between two glass slides having transparent ITO electrodes from the inner side. Then the cell was pressed and glued with an epoxy glue. The cell thickness was set by 20 μ m spacers. Finally, the cells were irradiated with a full spectrum of the high-pressure mercury lamp (100 mW cm⁻², 20 min), to provide photopolymerization and corresponding structuring of polymer phase.

The electro-optic characteristics were measured by the home-made measuring system previously described in Ref. [9]. In this set up, LC cell is powered by a sine like voltage signal 0–200 V synthesized by computer and, subsequently, amplified. The system allowed us to vary intensity U and frequency f of the applied voltage. The optical part of the set up consisted of laser ($\lambda = 635$ nm, polarized irradiation) and photodiode detecting light transmitted through the sample within the cone of 2°. Using this set up one can determine the sample transmittance $T = \frac{I_{\text{out}}}{I_{\text{in}}}$ (I_{in} and I_{out} are the intensities of the probe beam before and after passing through the sample) as a function of the applied voltage U. The T vs. U curves showed saturation. The saturation transmittance was measured as a function of the light incidence angle. The polarization of laser beam was in the incidence plane (p-polarization) to maximize dependence of light transmittance on the incidence angle. The reflection loses were estimated by measuring the intensity of the reflected light for various angles of light incidence.

3. Experimental results and discussion

3.1. Morphology

As it was mentioned above the morphology of LC–P composites strongly depends on the concentration of polymer phase. In result of the phase separation in the LC–Prepolymer mixture with $\varphi_{\rm P} = 1-10$ vol.%, polymer forms network structure (PNLC morphology) [7]. At $\varphi_{\rm P} = 30$ –

50 vol.%, polymer forms porous matrix filled with LC (PDLC morphology) [1–3].

In the LC-P-NP composites with a small content of polymer, the polymer network is also formed. As we mentioned before, it prevents intensive aggregation of NP common for filled LC. Below we are focused on the LC-NP-P composites with a polymer content corresponding to PDLC morphology. The amount of NP is chosen to be low ($\varphi_{NP} = 1-3 \text{ vol.}\%$) to minimize deformation of PDLC structure formed by P and LC. Our study of the morphology of LC-P-NP composites is aimed to elucidate two questions: (1) influence of NP on the morphology of LC-P composites; (2) distribution of NP in LC-P-NP composites.

To clear these questions, the morphology of LC-P and LC-P-NP samples was studies by SEM method. The glass substrates were carefully removed and the remained composite layer was kept in hexane to dissolve and remove LC. The solid remainder obtained after extraction of LC was studied by means of SEM method using S-2600N device from Hitachi. The SEM images of PDLC and NP-PDLC samples are shown in Fig. 2. It is evident that NP do not distort notably PDLC morphology. Besides, the cavities, which are the traces of LC droplets, do not contain nanoparticles or their aggregates. Together with thickening of polymer matrix this may imply that NP are mainly involved with polymer in the course of phase separation in LC-P-NP mixture. Possibly, they serve as nucleuses of polymer phase. This conclusion is also confirmed by other investigations. First of all, we examined the content of the phase extracted from the composite. The drop of this phase was placed on the glass slide, kept in atmosphere over 1 h to evaporate hexane and thereafter observed in polarizing microscope. The signs of NP have not been detected. Besides, the biggest drops, sometimes formed in the periphery part of our samples, have also been viewed in polarizing microscope. We have observed the non-distort bipolar LC drops in PDLC and NP-PDLC samples as well. This altogether gives us a ground to suggest that NP are mainly presented in polymer matrix.

Thus, roughly speaking, by insertion of NP, LC-P system is transformed to the LC-P system, where P is a polymer modified by NP. The properties of polymer P may substantially differ from the properties of pure polymer depending on particle's material, size and aggregation rate. What is most important for the present research, embedded NP may modify optical properties of polymer P.

The calculations show that when the size of particles or its aggregates is above 100 nm the polymer loses optical uniformity, i.e. it becomes opaque. By contrast, for the size smaller than 50 nm, even 15 vol.% NP loading does not affect polymer's uniformity [13]. To check the optical conditions of polymer matrix in our samples, the P-NP composites with varying content of NP were prepared. It was obtained that light scattering in both P-SiO₂ NP and P-Sb₂O₅ NP probes can be neglected up to the 5 vol.% loading that implies low aggregation rate of NP. In con-

(a)

Fig. 2. SEM images of PDLC polymer matrix: (a) pure PDLC $(\varphi_{\rm P} = 40 \text{ vol.}\%)$, (b) PDLC doped with Sb₂O₅ NP $(\varphi_{\rm P} = 38 \text{ vol.}\%)$, $\varphi_{\rm Sb_2O_5} = 3 \text{ vol.}\%$).

trast, P–TiO₂ NP samples considerably scatter light even at $\varphi_{\rm NP} = 0.5$ vol.%. This is mainly caused by the intense aggregation of the powdered TiO₂ NP confirmed by microscopic observations.

Even though the uniformity of polymer is preserved, the imbedded NP modify refractive index of polymer. In a good approximation, refractive index of modified polymer P linearly depends on NP concentration:

$$n_{\widetilde{\mathbf{p}}} = (n_{\mathrm{P}}\varphi_{\mathrm{P}} + n_{\mathrm{NP}}\varphi_{\mathrm{NP}}) \tag{1}$$

where $\varphi_{\rm P}$ and $\varphi_{\rm NP}$ are the volume fractions of polymer and NP [13,14]. This method is frequently used to enhance refractive index of polymer, which typically does not exceed 1.7. Inserting NP with a high $n_{\rm NP}$ enables us to increase this value by factor two and more.

For PDLC samples, modification of $n_{\rm P}$ may result in the substantial modification of scattering characteristics, mainly because of the variation of the refractive index mismatch between LC and polymer. Thus, adding NP modifies $n_{\rm P}$ and, as a result, scattering properties of PDLC.



Since the refractive index of modified polymer is a linear function of φ_{NP} independently on the kind of particles, we operate with the volume concentrations of components. Taking into account that NP are mainly involved with a polymer phase, serving as building blocks for polymer skeleton, we compare only samples with equal volume fraction of \tilde{P} and equal volume fraction of LC. The Fig. 2 shows that these samples are of very similar morphology.

3.2. Electro-optic performance

Fig. 3 shows T vs. U curves for the LC–P (1) and LC–P–NP (2,3) composites of PDLC morphology. By adding NP, the initial transmittance trends toward the slight increase, but this dependence is not very clear. At the same time, it is clearly seen that adding NP may considerably reduce the sample transmittance in the saturation state, T_s , making samples hazy. This effect is stronger for Sb₂O₅ NP. Fig. 4 shows that T_s monotonically decreases with the NP concentration.

The results for the T_s can be easily explained in terms of the refractive index mismatch. When LC within the drops is aligned along the electric field (normally to the cell plane) and light passes through the sample in the same direction, the refractive index mismatch between LC and P is a decisive factor of light scattering. For the convenience sake, let us consider the refractive indices diagram of the components of our composites. The diagram presented in Fig. 5 neglects dissolubility of LC and polymer. First, second and third columns in this diagram refer to LC, P (P) and NP. It is seen that the ordinary refractive index of E7 $(n_{\rm LC}^{\rm o} = 1.522)$ is very close to the refractive index of pure polymer $n_{\rm P} = 1.524$. This explains highly transparent state of LC-P composites in the field-on state at the normal viewing. Adding NP to the polymer phase enhances mismatch between the refractive indices of LC, $n_{\rm LC}^{\rm o}$, and the modified polymer, $n_{\widetilde{P}}$. As a result, T_s decreases. The more



Fig. 3. *T* vs. *U* curves for the non-doped and NP doped PDLC composites. $1-\varphi_P = 40 \text{ vol.}\%$, $\varphi_{NP} = 0 \text{ vol.}\%$, $2-\varphi_P = 39 \text{ vol.}\%$, $\varphi_{NP} = 1 \text{ vol.}\%$ (SiO₂ NP), $3-\varphi_P = 39 \text{ vol.}\%$, $\varphi_{NP} = 1 \text{ vol.}\%$ (Sb₂O₅ NP).



Fig. 4. T_s vs. φ_{NP} curves for NP doped PDLC. $\varphi_P + \varphi_{NP} = 40$ vol.%. 1—SiO₂ NP, 2—Sb₂O₅ NP. The lines are guide to the eye.



Fig. 5. The refractive indices diagram showing the refractive index mismatch of the components of LC–NP–P composites.

particles are added, the larger is the distance between $n_{\rm LC}^{\rm o}$ and $n_{\rm P}^{\rm o}$ levels in the diagram that explains the concentration behavior of the field-on transmittance presented in Fig. 4.

Fig. 6 presents data for the contrast ratio $CR = \frac{T_s}{T_0}$ in the LC–P and LC–P–NP series of composites. In these series, the concentration of polymer is varied to determine the proportion of components corresponding to maximal CR. The particle concentration is 1 vol.% for LC–P–SiO₂ NP and LC–P–Sb₂O₅ NP series and 0.5 vol.% for LC–P–TiO₂ NP series. One can see that CR vs. φ_P curves for all series are non-monotonic and go through the maximum, which is mainly determined by the minimization of T_0 . Poor contrast at low φ_P is explained by the non-developed PDLC structure more similar to PNLC (LC–P samples) or PNLC filled with small amount of NP (LC–P–NP samples). In turn, abrupt decrease of CR at $\varphi_P > 50$ vol.% may be caused by the reduction of size and concentration of LC drops and lowering the refractive index mismatch



Fig. 6. CR vs. $\varphi_{\rm P}$ curves for LC–P and LC–NP–P composites. 1— $\varphi_{\rm NP} = 0$ vol.%, 2— $\varphi_{\rm Sb_2O_5} = 1$ vol.%, 3— $\varphi_{\rm SiO_2} = 1$ vol.%, 4— $\varphi_{\rm TiO_2} = 0.5$ vol.%. The concentrations of NP are optimized for maximal contrast and sample uniformity. The lines are just for guidance.

between LC and $P(\tilde{P})$, because of enhanced content of LC in polymer and vice versa.

The CR vs. φ_P curves for the LC–P–NP series are shifted to the lower concentrations as compared to the curve for the LC–P series. This confirms that NP serve as building blocks for the polymer phase thus increasing the total volume of the modified polymer, \tilde{P} .

The maximal value of CR for LC–P–NP series is just slightly smaller than for LC–P series, because of small NP content and so slight modification of polymer matrix. Formula (1) allows us to estimate the increment of the refractive index of polymer. For example, in case of LC– P–Sb₂O₅ NP composites with maximal CR ($\varphi_P = 33 \text{ vol.}\%$ and $\varphi_{NP} = 1 \text{ vol.}\%$), formula (1) yields $n_{\tilde{P}} - n_P = 0.005$. This is, of course, superior limit, based on the complete absorption of NP with polymer and neglect of the fraction of large aggregates.

The highest maximum value of CR is obtained for the LC–P–TiO₂ NP samples. One can explain this by the considerable light scattering in polymer matrix \tilde{P} , as discussed in Section 3.1. The matrix's scattering slightly reduces T_s and, more importantly, T_0 that substantially increases CR value. This result suggests new way for the contrast enhancement in PDLC.

The light scattering due to the refractive index mismatch of \tilde{P} and LC phases of NP–PDLC also influences the sample transmittance in isotropic phase, T_i . Moreover, for the lack of LC orientational defects, this is a decisive factor of light scattering in isotropic phase, same as in the field-on state in the nematic mesophase. The mechanism discussed naturally explains difference between T_s and T_i values of the samples. For instance, for pure PDLC ($\varphi_P = 40 \text{ vol.}\%$), $T_s = 95\%$ and $T_i = 70\%$, while for the PDLC doped with Sb₂O₅ NP ($\varphi_P = 39.4 \text{ vol.}\%$, $\varphi_{Sb_2O_5} = 0.6 \text{ vol.}\%$), T_s and T_i values are estimated to be 85% and 70.5%, respectively. One can see that difference between T_s and T_i values for the Sb₂O₅ doped PDLC is considerably smaller than for the pure PDLC. This can explain refractive index diagram in Fig. 5. For pure PDLC, $n_P \cong n_{LC}^o$ so that T_s value approaches 100%, while $T_i < T_s$. For Sb₂O₅ doped PDLC, $n_{\widetilde{P}}$ approximates to $\langle n_{LC} \rangle$ and so values of T_s and T_i become closer to each other. For higher NP concentration this mechanism is masked by the unnegligible scattering of polymer matrix \widetilde{P} .

Finally, let us consider angular characteristics of T_s . It is well known that the conventional PDLC samples, transparent in the field-on state at the on-axis viewing, become hazy at the oblique incidence [15]. Similarly to the effects discussed above this problem known as the off-axis haze is also connected with the refractive index mismatch. As the light incidence angle α increases, the refractive index of LC changes from $n_{LC}^{\circ} \approx n_P$ at $\alpha = 0^0$ to n_{LC}^{e} at $\alpha \rightarrow 90^{\circ}$. Thus the mismatch between n_{LC} and n_P grows with growing of α . According to the Lambert–Beer law, light scattering is a decaying exponential function of the refractive index mismatch and the optical path length. Consequently, light scattering increases with α being evident in the increasing off–axis haze.

Several methods are proposed to reduce off-axis haze of PDLC. The simplest way consists in setting of polarizer in front of PDLC film with polarization direction perpendicular to the light incidence plane [16]. Indeed, light of this polarization (*s*-polarization) is insensitive to incidence angle. However, polarizer decreases brightness of PDLC film at least in twice that makes this method not very attractive. The other solution [17] suggests to replace isotropic polymer matrix by anisotropic one with the ordinary and extraordinary refractive indices close to n_{LC}^{o} and n_{LC}^{e} , respectively. The challenge of this method is matching of two anisotropic phases. Finally, the most common way consists in selection of isotropic polymer with the refractive index satisfying inequality [15,18]

$$n_{\rm LC}^{\rm o} < n_{\rm P} \ll n_{\rm LC}^{\rm e} \tag{2}$$

When (2) is true, then peak of maximal transmittance shifts away from normal incidence so that range of viewing angles with sufficiently high on-state transmittance becomes broader. The reduction of off-axis haze is accompanied by increasing of on-axis haze. This means that acceptable viewing angle characteristic is a result of thorough optimization of the refractive indices of LC and P. Typically, the optimized value of $n_{\rm LC}^{\rm o} - n_{\rm P}$ does not exceed 0.01 [15,18].

Fig. 7 represents T_s as a function of the incidence angle α of the testing laser beam measured for our composites. It is obvious that for PDLC and SiO₂-PDLC composites T_s rather steeply drops with α . A similar trend is observed for TiO₂-PDLC samples having scattering polymer matrix. In contrast, doping of PDLC with NP Sb₂O₅ flattens angular dependence of T_s at rather high level that evidences for the reduced off-axis haze. Simultaneously, this doping process slightly reduces value of T_s at $\alpha = 0$ for the reason explained above.

The $T_s(\alpha)$ characteristics of PDLC and NP-PDLC samples with transparent polymer phase (SiO₂-PDLC and



Fig. 7. The on-state transmittance T_s of PDLC and NP–PDLC samples as a function of angle of the light incidence α . The light is p-polarized. 1— $\varphi_{NP} = 0$ vol.%, 2— $\varphi_{Sio_2} = 1$ vol.%, 3— $\varphi_{Sb_2O_5} = 1$ vol.%. $\varphi_P + \varphi_{NP} = 40$ vol.%. The lines are guide for eye.

Sb₂O₅–PDLC samples) can be explained considering refractive index differences of \tilde{P} and LC. According to the refractive index diagram in Fig. 5, for pure PDLC $n_{\rm P} \approx n_{\rm LC}^{\rm o} < n_{\rm LC}^{\rm e}$ so that off-axis haze occurs. Adding SiO₂ particles has almost no effect on $T_{\rm s}(\alpha)$ characteristic because $n_{\rm P} \approx n_{\rm LC}^{\rm o}$. Doping PDLC with Sb₂O₅ particles leads to an increase in the refractive index of the polymer phase \tilde{P} and so fulfillment of the condition (2) required to lower off-axis haze. Simultaneously, according to Fig. 6, contrast ratio changes non-essentially. Using this method it is not necessary to select polymer with $n_{\rm P}$ satisfying the inequality (2); by adding NP one can adapt the polymer suitable for the formation of PDLC structures with the given LC.

4. Conclusions

In summary, NP doped PDLC represent a new class of LC composites with the considerable application potential. Retaining electro-optic contrast typical for PDLC, these composites may show substantially improved angular characteristic of light transmittance in the field-on state. It is realized by modifying the polymer refractive index with inorganic NP mainly absorbed by polymer phase in the course of the phase separation of LC–P–NP mixtures. This effect implies week aggregation of NP in polymer. By contrast, the essential aggregation of absorbed particles enhances light scattering in polymer phase that, in turn, may result in the considerable increase of the electro-optic contrast.

We believe that, except lowering of off-axis haze, doping of polymer phase with inorganic NP may lead to other improvements of PDLC, such as enhancement of thermal and mechanical stability of polymer matrix [19], reduction of LC content in polymer etc. Well known that inorganic dopant may also share other properties with polymer phase, e.g. photoconductivity [20] and photochromism [21,22]. In case of doping PDLC one can yield composites with the distinctly new properties. This altogether creates an interesting field for future studies.

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