Structuring of the Surface Layer of Polycarbonate Film upon Interaction with Nematic Liquid Crystal

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Received September 29, 2017

Abstract—The structure formation observed in the surface layer of polycarbonate which proceeds when nematic liquid crystal is deposited on a film is studied. Films prepared from polycarbonate solutions in dichloromethane, chloroform, or pyridine and dried in thermobalance at different temperatures and different drying times are studied. Textures are prepared in the process of domain growth. The effect of the orientation of the disclination lines in a magnetic field and the memory effect of molecular ordering after removal and redeposition of the liquid crystal layer are observed. The process of structure formation is analyzed on the basis of the model of cooperative interaction of liquid crystal molecules with polymer chains taking into account the adsorption mechanism.

INTRODUCTION

It is known that, in polymers with regular structure, proper conformation, and segmental mobility of macromolecules under appropriate thermodynamic conditions, supramolecular structures are formed, which are characterized by stacking of polymer chains in packets, ribbons, lamellas, crystallites, fibrils, or spherulites. These structures are particularly diverse in polycarbonates [1−3]. Varying the chemical composition and conditions to prepare polymers, one obtains fine crystalline, polycrystalline, and highly organized spherulite structures [3]. The structures can be studied by optical methods and observed by using a polarization microscope. In addition, the surface properties of polymers usually differ significantly from their bulk properties [4]. Ordering of polymeric chains with a planar or homeotropic orientation may appear near the polymer surface. The orientation order is determined by the tendency of the bulk and surface regions of the polymer to minimize the interfacial energy and depends on the mobility of molecular chains. When the polymer interacts with an external gaseous, liquid, or solid medium, supramolecular globular, fibrillar, or spherolite-like structures can form on its surface [5]. Along with this, the surface layer of the polymer film prepared from the solution is capable of creating a potential barrier at the interface, which prevents the solvent from being released [6].

In practice, sandwich materials are often used, which are layers of liquid crystal bounded by profiled or smooth polymer films [7]. Profiling the surface of the film leads to the formation of grooves where the liquid crystal molecules are laid, and owing to the long-range orientation order, the planar surface orientation is transferred to the bulk of the liquid crystal over considerable distances. On a smooth substrate, in the absence of interaction between the liquid crystal and the surface, either a threadlike texture or a schlieren texture are formed [8, 9]. These textures are conditioned by the properties of the liquid crystal molecules to be packing into wedge-shaped, radial, or circular configurations. However, in study of liquid crystals on smooth polymeric surfaces in [9−11], under certain conditions providing the preparation of a sandwich, effects were found which could only be explained by taking into account the interaction between the liquid crystal and polymer. Thus, when the temperature was varied in the smectic and nematic phases of the liquid crystal, a memory effect was observed consisting in the repeated reproduction of various textures after their destruction [9]. The memory effect was essentially dependent on the type of solvent used to prepare the polymer films and was explained by the plastic interaction of the liquid crystal with the polymer surface. When the nematic was cooled from the isotropic phase in magnetic [10] or electric [11] fields, the effect of orientation ordering memory was observed, which consisted in preserving the orientation of the director of the liquid crystal after the field was switched off. The effect was explained by the interaction of nematic molecules with polymer

chains using the adsorption mechanism [11, 12]. The memory effect was also manifested when studying liquid crystals on photoinduced polymers in an electric field and was interpreted within the framework of the model of cooperative reorientation of the director and polymer fragments [13, 14] based on the adsorption mechanism. Later, the memory effect of orientational ordering was observed in liquid crystal deposited on a film of a gel-like polymer obtained by evaporating solvent from solution in the presence of a magnetic field [15] and was interpreted taking into account adsorption.

Recently, we observed the memory effect of domain ordering of nematic liquid crystals on polycarbonate films formed from solutions [16]. It was shown that the appearance of the domain structure could be explained only when considering the interaction between liquid crystal molecules and polymer chains. However, the process of structuring the polymer surface under the influence of liquid crystals systematically, taking into account existing models and mechanisms, has not been studied.

The purpose of this work is to study the structure formation in the surface layer of polycarbonate (PC) films obtained from solutions with various solvents under different conditions of preparation.
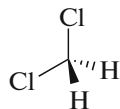
EXPERIMENTAL

Commercially available polycarbonate based on bisphenol A [1, 2] with $M \sim 45 \times 10^3$ synthesized as granules (Sigma-Aldrich) was used for the studies. The chemical structure of the monomer unit
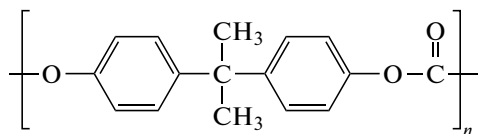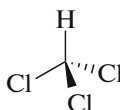

was identified by IR spectroscopy. As the liquid crystal, we used widely known nematic 4-methoxybenzylidene-4'-butyl aniline (MBBA) [17]
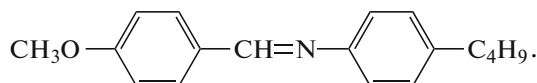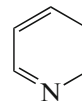

Dichloromethane (CH_2Cl_2)

chloroform ($CHCl_3$)

and pyridine (C_5H_5N)

were used as solvents.

Granules of PC were dissolved in one of the solvents in a concentration of 2 wt % to obtain the samples. A small amount of the solution was deposited on the glass plate by watering or centrifugation at room temperature (24°C). After a few seconds, a polymer film with a thickness from 100 nm to several microns was formed on the glass surface. The film was dried in a thermobalance at a temperature of 120, 50, or 24°C for different time intervals, after which it was cooled to room temperature. The film was taken out of the thermobalance and liquid crystal was deposited immediately on the surface as a drop using a pipette. The thickness of the liquid crystal layer fell in the range from 10 to 100 μm. In experiments with the study of orientation effects during the formation of textures of the liquid crystal, a constant magnetic field $H = 2 \times 10^3$ A/m was applied to the sample using an electromagnet. The liquid crystal textures formed on the PC surface were studied by using a polarization microscope.

RESULTS AND DISCUSSION

Figure 1 shows the time dependences of dichloromethane weight p evaporated from the PC film in the thermobalance at different temperatures. The same dependences $p(\tau)$ were obtained using other solvents. Curve *1* refers to a temperature of 120°C. When the PC film was dried at higher temperatures, up to the glass transition temperature of the polymer $T = 141$°C, the dependence $p(\tau)$ coincided with curve *1*. In this connection, the horizontal section of curve *1* was adopted as the zero reference line for $p(\tau)$. The values of $\Delta p_2 = 30$ μg and $\Delta p_1 = 90$ μg which correspond to distances from the zero reference line to the horizontal sections of curves *2* and *3* corresponding to temperatures of 50 and 24°C, respectively, are the amounts of the solvent remaining in the PC film.

The textures that appeared when the liquid crystal was deposited on films dried from PC solutions in dichloromethane, chloroform, or pyridine for $\tau = 0–30$ min at 120°C are shown in Fig. 2. When dichloromethane was used as a solvent at $\tau < 15$ min, a texture with weakly expressed domains with dimensions up to 100 μm appeared (Fig. 2a). In the case of chloroform and pyridine, a polydomain texture (Fig. 2b) and a small-domain texture without distinct interfaces (Fig. 2c) appeared, respectively. At $\tau > 15$ min, a threadlike texture was observed on the PC film for all types of solvents (Fig. 2d). Since such a texture is formed in the absence of interaction between the liquid crystal and the surface [8], PC films prepared
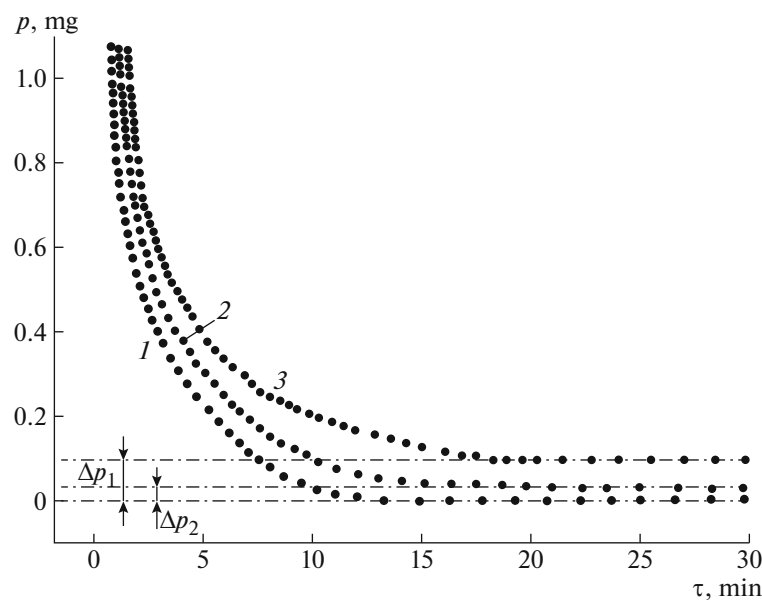
Fig. 1. Change in weight of dichloromethane p in the PC film in the course of drying in thermobalance at a temperature of (*1*) 120, (*2*) 50, and (*3*) and 24°C.
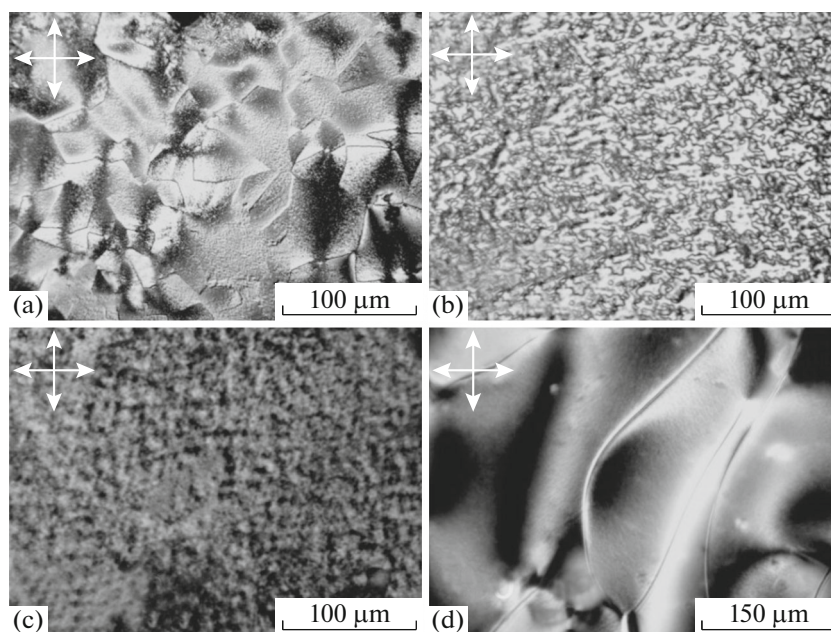

Fig. 2. Microphotographs of textures obtained by MBBA deposition on the PC film dried at a temperature of 120°C for (a–c) 5 min and (d) 30 min from the polymer solution in (a, d) dichloromethane, (b) chloroform, and (c) pyridine. Here and in Figs. 3–5, the directions of light polarization are shown by intersecting arrows.

under these conditions can be considered completely dried.

Figure 3 shows the textures that appeared when the liquid crystals were deposited on PC films dried at 50°C from polymer solutions in dichloromethane, chloroform, and pyridine. Regardless of time τ, when using dichloromethane, chloroform, and pyridine as the solvent, a texture with highly organized domains with dimensions from 50 to 150 µm (Fig. 3a), a texture with weakly expressed domains (Fig. 3b), and a fine-grained texture (Fig. 3c) were obtained, respectively.

Textures obtained by depositing liquid crystals on PC films dried at 24°C from solutions in different solvents are shown in Fig. 4. Using dichloromethane, a
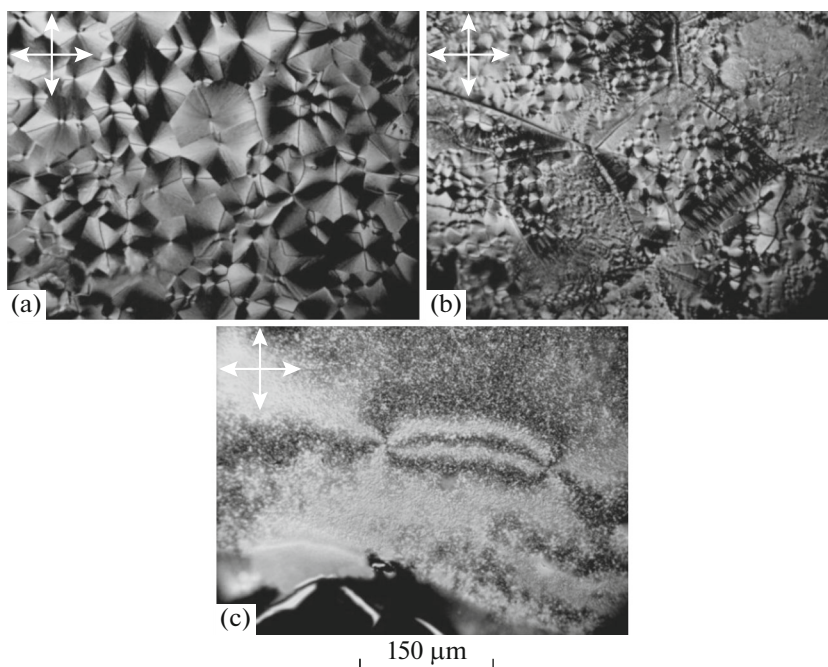
Fig. 3. Photomicrographs of textures obtained by MBBA deposition on the PC film dried at a temperature of 50°C for 15 min from the polymer solution in (a) dichloromethane, (b) chloroform, and (c) pyridine.
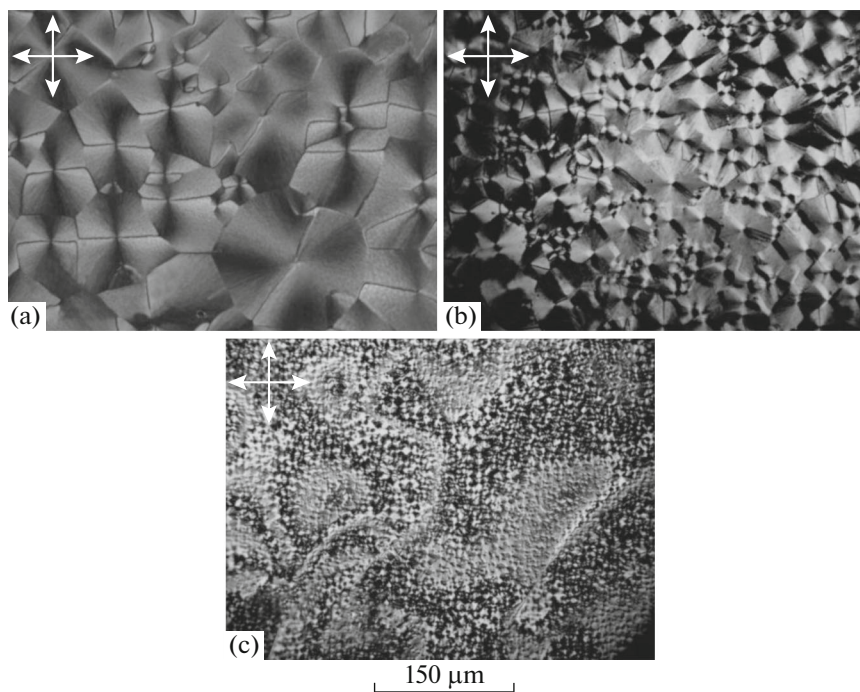

Fig. 4. Photomicrographs of textures obtained by MBBA deposition on the PC film dried at a temperature of 24°C for 15 min from the polymer solution in (a) dichloromethane, (b) chloroform, and (c) pyridine.

similar domain structure as present in Fig. 3a but more pronounced is observed (Fig. 4a). A similar texture but with rather small domains and high dispersion in size was found for chloroform (Fig. 4b). Even smaller and less pronounced domains arose on the PC film when pyridine was used as the solvent (Fig. 4c). In general, as shown by additional experiments, all textures were observed irrespective of the drying time of the film τ
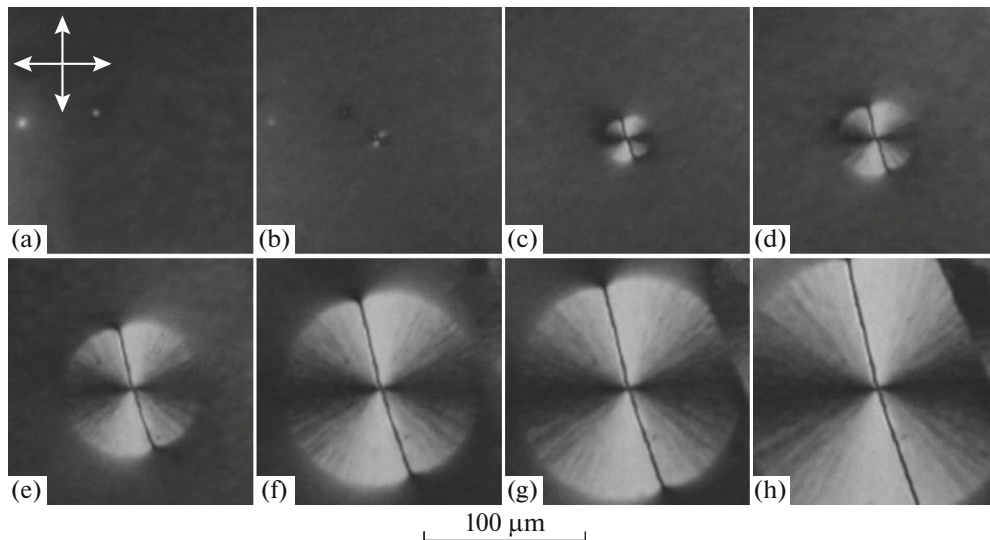
Fig. 5. Photomicrographs of the domain during its growth in a drop of MBBA on the PC film formed from the polymer solution in dichloromethane at a temperature of 24°C and a humidity of 60%. The growth time is (a) 0.5, (b) 1.0, (c) 2.0, (d) 5.0, (e) 6.0, (f) 12.5, (g) 14.0, and (h) 16.0 min.
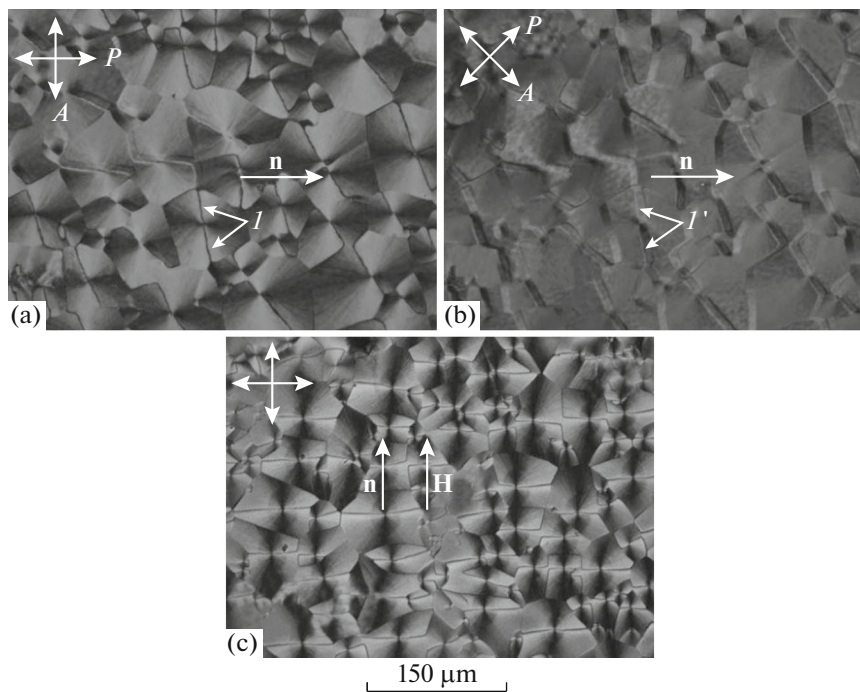

Fig. 6. Photomicrographs of textures containing domains with disclination *l*, *l'* obtained by deposition of MBBA on the PC film dried for 30 min at a temperature of 24°C from the polymer solution in dichloromethane. The axes of analyzer *A* and polarizer *P* (a) are arranged vertically and horizontally and (b) are synchronously rotated by an angle of 45°, and (c) the structure is formed in magnetic field $H = 2 \times 10^3$ A/m.

and found throughout the temperature range of the existence of the nematic MBBA phase (22–47°C).

The domain textures shown in Fig. 4a have a number of features of formation. One of them is that the domains appear over a period of time rather than immediately after the deposition of liquid crystals on

the PC surface, depending on the temperature and humidity of the environment and independently of the shape of the surface of the liquid crystals and polymer film. Figure 5 shows the successive development (growth) of the domain in the MBBA drop on the surface of the PC film. The domain arises from the
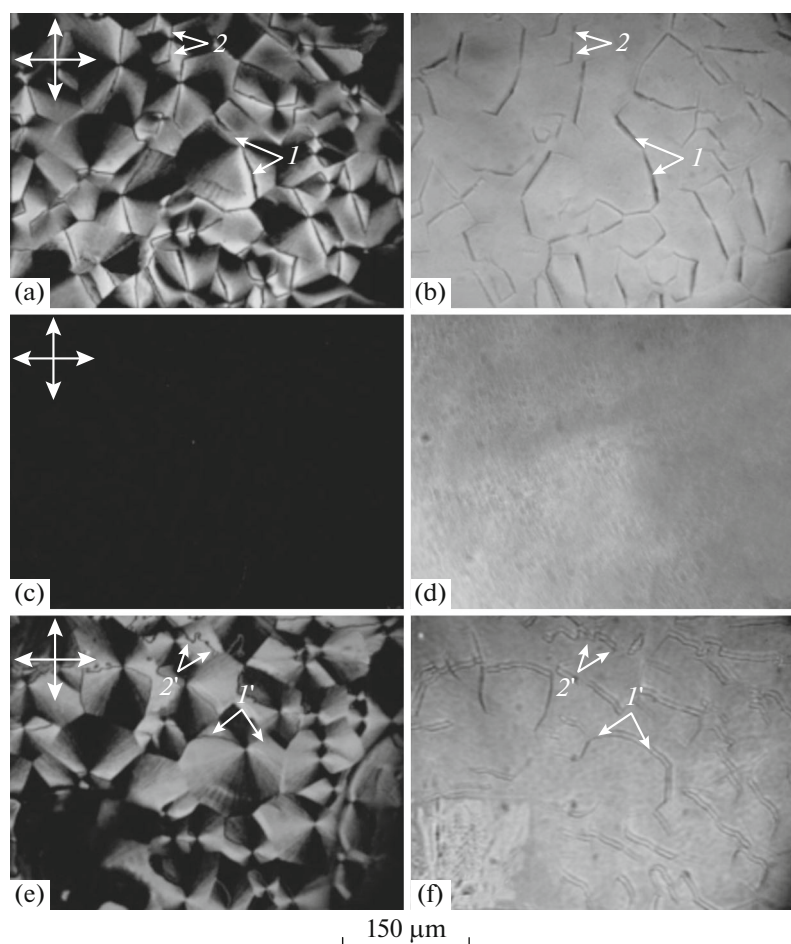
Fig. 7. Photomicrographs of the PC film dried for 5 min at 24°C from the polymer solution in dichloromethane: (a, b) the structure with domains and disclination lines *1, 2* in a drop of MBBA deposited on the film surface; (c, d) the film surface after removal of the droplet; (e, f) the newly formed structure with domains and disclination lines *1', 2'* after redeposition of the drop; (a, c, e) views in crossed polarizers; (b, d, f) views in the absence of polarizers.

nucleus and its diameter *d* increases linearly with time. In crossed polarizers of the microscope, it is observed in the form of a disk with two dark absorption bands. A disclination line emanates from the center of the domain along its radius which is bordered with a circle.

The width of the disclination line depends on the light polarization directions. Thus, Fig. 6a shows the texture obtained by MBBA precipitation on the PC film dried for 30 min at 24°C from a solution in dichloromethane; disclination line *1* in the domain is narrow. When the crossed analyzer and polarizer are synchronously rotated through an angle of 45° (Fig. 6b), it becomes visible as broad band *1'*. In magnetic field $H = 2 \times 10^3$ A/m applied to the sample during its formation parallel to the PC film, the disclination lines are oriented perpendicular to the direction of *H* and are always narrow.

Another peculiarity of domain textures is that, after removal of droplets of liquid crystals from the surfaces of PC films, domain textures are newly formed on them. Thus, Fig. 7a shows a domain texture formed on

the PC film dried for 5 min at 24°C from a solution in dichloromethane in crossed polarizers. The disclination lines *1* and *2* of two separate domains are approximately straight. Figure 7b shows the same texture observed in the absence of polarizers. It also clearly shows the disclination lines. After removal of a drop of liquid crystal from the surface of the PC film, the latter becomes dark in crossed polarizers (Fig. 7c) and looks like an even light surface when the polarizers are removed from the optical path (Fig. 7d). When the droplet of the liquid crystal is redeposited on the surface, the domain texture appears again (Figs. 7e, 7f) with the same anchoring point of the nucleus, but with a different arrangement of the extinction bands and the disclination lines *1'* and *2'* in domains. In this case, some disclination lines (*2'*) become twisting.

The effect of PC film drying time on the formation of textures indicates that the solvent plays an important role in this process. Apparently, the surface layer of the PC film creates a potential barrier that prevents the solvent from being released. It can be assumed that,

when a drop of liquid crystal is applied to the PC surface, nematic molecules destroy the potential barrier and begin to interact with solvent molecules during extraction [18]. This assumption is completely justified, since it is known [19] that dichloromethane used by us as the solvent effectively extracts molecules of the nematic liquid crystal from droplets emulsified in the polymer matrix. We believe that the extraction of solvent molecules by liquid crystal molecules creates an excess of solvent in the surface layer and causes the mobility of the polymer chains (Fig. 8).

The structuring of the PC surface layer can be considered in the following ways. First, we can assume that an increase in the mobility of polymer chains causes PC crystallization, and the liquid crystal promotes the visualization of its supramolecular structure in the same way as, for example, it facilitates the visualization of domains on the surface of the solid crystal [20−22]. Indeed, the textures shown in Figs. 2−7 are comparable with spherulitic and fine-crystalline structures observed on various PC samples [3]. Moreover, the formation of textures with highly organized domains is carried out in much the same way as the growth of highly organized spherulites from nuclei [1, 3]. However, the absence of texture patterns when removing the layer of liquid crystals from the surface of the polymer after domains are formed when observed by optical (Figs. 7c, 7d) and electron microscopes does not allow us to interpret the results obtained.

Secondly, it can be assumed that a three-component system is formed in the surface layer of the polymer film, which contains the polymer, liquid crystal, and common solvent, evaporation of which during the phase separation results in a drop of the liquid crystal being formed with a radial structure [23]. However, this process requires the amount of a solvent in the polymer film much higher than its residual quantity shown in Fig. 1. In addition, within the framework of this assumption, it is difficult to explain the memory effect and the presence of disclination lines, which must be subject to topological laws.

Thirdly, the memory effect of structural ordering, as the effect described earlier [9−11], should be related to the mechanism of adsorption of liquid crystals on the polymer surface. This is evidenced by the stability of the highly organized domain textures shown in Figs. 3−7 and our previous studies [16], in which it was shown that these textures arise against a background of the threadlike texture or schlieren texture destroying them in the course of formation.

Fourth, it is clear that the reasons for the formation of structures cannot be identified using only the adsorption mechanism. The explanation of the growth of domains, the growth and straightening of disclination lines, and the ability of disclination lines to be oriented perpendicular to the director of the liquid crystal and parallel to the magnetic field requires consideration of the interaction between liquid crystal mole-
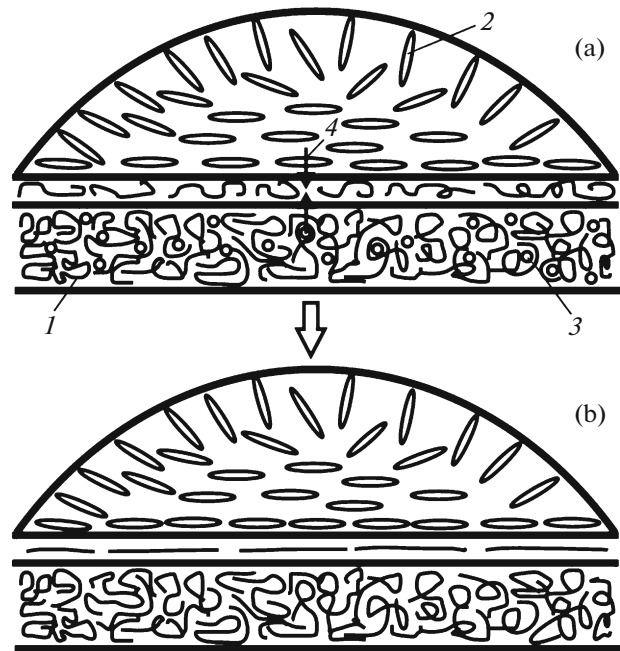

Fig. 8. Schematic representation of the formation of the liquid crystal structure in the PC surface layer (a) during the deposition of a droplet of the liquid crystal on the surface and (b) after completion of the growth of domains: (*1*) polymer chains; (*2*) molecules of a liquid crystal; (*3*) solvent molecules; (*4*) directions of movements of molecules of the liquid crystal and solvent.

cules and polymer chains. Further research will be conducted according to this consideration.

Structuring of the PC surface suitable to retain the domain configuration of the liquid crystal can be estimated using energy E consisting of the characteristic energy of the interaction between polymer and solvent E_{ps}, attraction energy of the polymer units E_{pp}, and interaction energy of the polymer units with nematic molecules E_{plc}: $E = E_{ps} + E_{plc} − E_{pp}$. To estimate E_{ps}, the Huggins constant was calculated for the systems under study according to the equation $\eta_{sp}/c = [\eta] + K_H [\eta]^2 c$ [1], where c is the concentration of the solution, $\eta_{sp}/c = (\eta − \eta_0)/\eta_0 c$ is the reduced viscosity of the solution, η and η_0 are the viscosity of the solution and that of the solvent, and $[\eta]$ is the intrinsic viscosity. By measuring η_{sp} and c and using the standard calculation procedure with extrapolation of η_{sp}/c to zero polymer concentration [24], we obtained $K_H = 0.43$, 0.3, and 0.13 for PC solutions in dichloromethane, chloroform, and pyridine, respectively. When the PC film is dried at a temperature of 120°C, the amount of solvent contained is not sufficient to convert the polymer surface to the state of a dilute solution with separate macromolecules. Nevertheless, the high K_H value and, consequently, high E_{ps} at $E_{ps} + E_{plc} \gg E_{pp} > kT$ for the PC solution in dichloromethane lead to the formation of a structure with weakly expressed domains
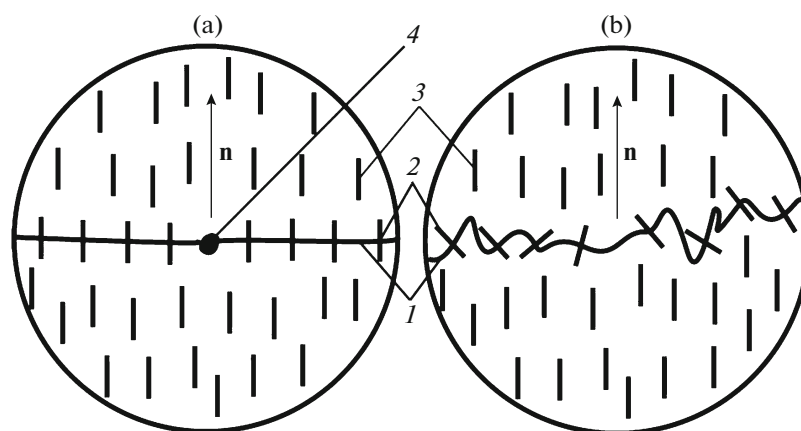
Fig. 9. Schematic representation of the distribution of molecules in the liquid crystal domain on the PC surface: (*1*) polymer chain, (*2*) liquid crystal molecules adsorbed on the polymer chain, (*3*) liquid crystal molecules in the drop volume, (*4*) polymer coil; **n**—director of the bulk layer of the liquid crystal; (a) molecular configuration with a straightened polymer chain, (b) configuration with a twisted chain.

(Fig. 2a). In the case of the PC solution in chloroform with a lower K_H value but high E_{ps} providing $E_{ps} + E_{plc} \gg E_{pp} > kT$, a polydomain texture is visualized on the surface of the PC film (Fig. 2b). At the same time, using pyridine, which has low values of K_H and E_{ps} insufficient for deformation of the polymer chain, leads to the formation of a small-domain texture without distinct interfaces (Fig. 2c). When the PC film is dried for a long time ($\tau > 15$ min) at 120°C, complete evaporation of the solvent occurs, the mobility of the polymer chains in the surface layer becomes low, they do not interact with the molecules of liquid crystals, and a threadlike texture is formed on the polymer surface (Fig. 2d). When the PC film is dried at a temperature of 50°C, the amount of residual solvent is sufficient for allowing mobile parts to appear in the surface layer of the PC film. High K_H values lead to structures with domains when dichloromethane (Fig. 3a) and chloroform (Fig. 3b) are used as solvents, whereas a low K_H value characteristic of pyridine results in a fine-grained texture (Fig. 3c). When the PC film is dried at room temperature, the amount of residual solvent in it becomes sufficient, and textures with domains are visualized using liquid crystals on its surface with higher quality as compared with those prepared by previous methods of solvent evaporation, but also depending on the value of K_H (Fig. 3).

The growth process (Fig. 5) and the effect of the repeated formation of LC domains on the PC film after its washing (Fig. 7) allows us to make the following assumption. The disclination line arises in the domain in the presence of moving parts of polymer chain *1* with molecules of liquid crystal *2* adsorbed perpendicularly (Fig. 9). Adsorbed molecules tend to orient the polymer chain perpendicular to the director **n**, which is the average orientation of the molecules of the planar bulk layer of liquid crystals *3* in the drop, in

order to lower the free energy. The free polymer chain certainly has a curved section that collapses into a coil, resulting in the chain being straightened, since the configuration in Fig. 9a is more energetically favorable than that shown in Fig. 9b. The region of the polymer chain stranded in coil *4* will be the orienting center, around which the molecules of the liquid crystal begin to order, forming a radial structure. This process will occur slowly because of the molecular interactions between the liquid crystal and the polymer surface. Owing to adsorption of nematic molecules on the PC film, the coil will be fixed on the surface. Adsorbed molecules of the liquid crystal visualize polymer chains. If they are oriented in the polymer chains in the same direction as those of director **n** and the analyzer or polarizer, the domain will be narrow when observed in the polarization microscope (Fig. 6a), otherwise it will be wide (Fig. 6b). Magnetic field **H** applied along the PC film orients the molecules of the liquid crystal in the volume of the droplet, which entraining adsorbed molecules orient the polymer chains perpendicular to the director **n** (Fig. 6c). Since the directions of **H**, **n**, and adsorbed molecules coincide with each other and with the direction of light polarization, the disclination lines are narrow in the magnetic field. When liquid crystal is removed from the PC film, the polymer chains are released, but the adsorbed nematic molecules and the attachment points of the coil remain attached to the surface, and the memory effect of the structural ordering remains. However, when molecules of the bulk layer of liquid crystal are removed owing to external forces, the polymer chains are stretched and remain twisted when the nematic is redeposited, as can be seen from Fig. 7 and is presented in Fig. 9b.

CONCLUSIONS

Currently, supramolecular structures of polymers have been studied; their surface properties as well as properties of liquid crystals in contact with polymeric surfaces are actively investigated. However, there are a small number of studies dealing with the structure of polymers formed using liquid crystals. Here, the structure formation in the polymer surface layer is studied upon deposition of the MBBA nematic on the surface of the PC film dried in thermobalance at different temperatures and drying times. Various textures of liquid crystals have been studied, the most attractive of which are textures with highly organized domains. The domains are formed during growth and contain disclination lines that are oriented perpendicular to the director of liquid crystal and to the magnetic field applied in the plane of the PC film. The width of the disclination lines depends on the direction of light polarization. A memory effect is found consisting in the repeated formation of the domain texture after the removal of the layer of liquid crystals. We have failed in comparing the textures with supramolecular structures of the polymer. Therefore, the structure formation in the PC surface layer has been estimated using the intermolecular interaction energies of the polymer, liquid crystal, and solvent. The relationship between the amount of solvent and the mobility of polymer chains is determined. The process of structure formation is considered using a model that takes into account the cooperative interaction of nematic molecules with polymer chains using the adsorption mechanism. On the basis of the same model, we have analyzed the formation of the radial configuration of the director of the nematic, as well as the development, straightening, and ordering of the disclination lines in the domains. We believe that the results of this work will expand the variety of surface properties of polycarbonate and probably other polymers.

ACKNOWLEDGMENTS

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Translated by V. Avdeeva