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LIQUID CRYSTALLINE MATERIAL EXHIBITING MULTIPLE PHASE TRANSITIONS: MORPHOLOGIC PROPERTIES AND OPTICAL TRANSMISSION

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Morphologic and thermo-optical properties of liquid crystalline material exhibiting multiple phase transitions were investigated in this work. Investigations were carried out for both the direct (by heating) and the reverse (by cooling) phase transitions. Thermo-morphologic properties of the heterophase regions of phase transitions were investigated. Temperature dependences of the optical transmission were studied, the mean optical density of crystalline state, smectic C, smectic A and nematic mesophases was estimated, temperature widths of the heterophase regions of *crystal* ↔ *smectic C*, *smectic C* ↔ *smectic A*, *smectic A* ↔ *nematic* and *nematic* ↔ *isotropic liquid* phase transitions were determined with high accuracy.

Keywords: liquid crystals, texture, multiple phase transitions, optical transmission, heterophase regions

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1. Introduction

Due to the physical anisotropic and self-assembling nature, liquid crystalline materials hold an important place in theoretical and experimental physics, and also in various technical devices and technological processes [1–5].

In liquid crystalline materials, between the solid crystal and isotropic liquid one or more liquid crystalline mesophases take place. Between these mesophases, in a definite temperature interval, the first or second order phase transitions can be observed. Availability of a series of phase transitions (multiple phase transitions) distinguishes the liquid crystals from most other materials, which have only a single phase transition [6–11]. In liquid crystals exhibiting multiple phase transitions, differences between various phases and mesophases are defined by differences of the structural organization, spatial symmetry and ordering of these phases and mesophases. Therefore, such liquid crystalline materials are excellent objects for the study of peculiarities for various types of phase transition, and investigation of melting processes and critical phenomena [12–17].

By heating the liquid crystals from a crystalline state to an isotropic liquid state and by cooling from an isotropic liquid state to a crystalline state they can exhibit different mesomorphism degree and display various types of thermotropic phase transitions. At such phase transitions structural transformations of liquid crystalline mesophases, change of space and point-like symmetries of mesophases, change of various physical parameters and appearance of unusual physical properties can be observed. Besides, liquid crystalline materials at the direct and reverse phase transitions can display various types of enantiotropic and monotropic mesophases [18–21]. Therefore, information about the temperature behaviour of physical parameters of liquid crystalline materials at phase transitions is important from a fundamental and an application point of view.

Scientific literature presents rich information about the temperature behaviour of optical parameters of monomorphic and dimorphic liquid crystals at the direct phase transitions in the heating process. Unfortunately, information about the temperature behaviour of optical parameters for the reverse phase transitions in the cooling process and in the circular heating–cooling cycle is insufficient. Besides, a special interest in physics of liquid crystalline materials is shown in topics connected with investigations of polymorphic liquid crystals exhibiting multiple phase transitions.

In polymorphic liquid crystals, unlike monomorphic and dimorphic liquid crystals, successive and unremitting transformations from more structurally ordered to less structurally ordered mesophases occur.

In this work, the morphologic and thermo-optical properties of mesophases were investigated. Peculiarities of the heterophase regions of direct and reverse phase transitions in the heating–cooling processes in a liquid crystal with multiple phase transitions were studied. The optical transmission during the *crystal–smectic C–smectic A–nematic–isotropic liquid–nematic–smectic A–smectic C–crystal* phase transitions was studied and a comparative estimation of the optical density in a crystalline state, smectic C, smectic A and nematic mesophases was carried out.

2. Experiment

In this work, 4-octyloxy-benzoic acid 4′-buthoxyphenyl ether (OBABPE) was the object of our investigations. The structural formula of this material is given in Fig. 1. This liquid crystal was purchased from *Khimreactive Inc.* (Russia). The purity of OBABPE was estimated to be 99.70% by liquid chromatography [22]. OBABPE has a calamitic molecular form, is a polymorphic liquid crystal, displays enantiotropic smectic C, smectic A and nematic mesophases, exhibits multiple thermotropic phase transitions, and is thermally stable and stable to moisture.

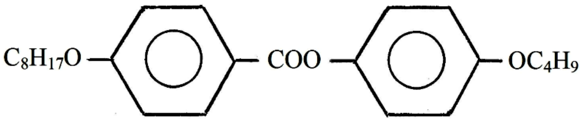

Fig. 1. The molecular structure of OBABPE.

The samples used in this study were the sandwich-cells with a fixed thickness. The reference surfaces of the plane capillaries were optical glass slides. The thickness of a liquid crystalline layer, which was placed between the reference surfaces of the sandwich-cell, was examined by the POM technique and a digital micrometer with an accuracy of ±0.1 μm. The thickness of the liquid crystalline layer was fixed to be 40 μm. OBABPE was filled into the sandwich-cell by the capillary forces at the isotropic liquid state.

Investigations of the thermotropic and thermomorphologic properties of OBABPE were carried out by the crystallo-optics and crystallo-physics methods [23–25]. These methods allow determining the number and orientation of optical axes, the value and optical sign of birefringence, peculiarities of defects and disclinations, etc. in materials with optically anisotropic properties. The classic polarizing optical microscopy (POM) technique was also used in this work. Our POM set-up consists of a trinocular polarizing conoscopic/orthoscopic microscope, Berek and quartz compensators, optical filters and λ-plates from *Olympus Optical Co., Ltd.*, and also a special heater-thermostat with a digital temperature control system, multimeters and power supply. Registration of microphotographs and conoscopic images was carried out by the digital microphotographic system from *Olympus Optical Co., Ltd.*

The thermo-morphologic properties of the heterophase regions of phase transitions were studied by the method of temperature wedge [26] which was modified by us as a capillary temperature wedge (CTW) device [27, 28]. The CTW device provides observations of all thermal states of liquid crystalline materials in a real scale of time and in a wide temperature range, and also provides the calculation of phase transition temperatures and the temperature widths of the biphasic regions with an accuracy not less than 10^{-2} K [27–31].

Temperature dependences of the optical transmission (OT) were measured using the thermo-optical set-up (Fig. 2). This set-up consists of a polarizer, an analyzer, a beam splitter, a special thermostat with a digital control system, detectors, power supply and multimeters. As the light source, the He–Ne laser was used. Temperature dependences of the OT were recorded by a computer. The heating and cooling rate during the thermo-optical measurements was 0.5 K·min^{-1}.

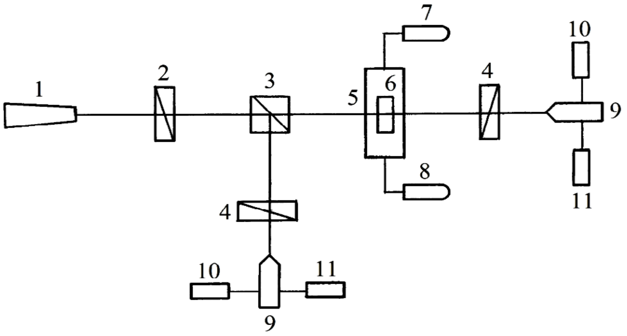

Fig. 2. Schematic representation of the experimental set-up. 1, He-Ne laser; 2, polarizer; 3, beam splitter; 4, analyzers; 5, thermostat; 6, the sandwich-cell; 7, digital temperature control system; 8, power supply; 9, detectors; 10, multimeters; 11, computers.

3. Results and discussion

In OBABPE, the *solid crystal* (Cr) → *smectic C* (SmC) *mesophase* → *smectic A* (SmA) *mesophase* → *nematic* (N) *mesophase* → *isotropic liquid* (I) → N *mesophase* →

SmA *mesophase* → SmC *mesophase* → Cr sequence of the direct and reverse phase transitions takes place. Temperatures of the phase transitions in OBABPE are given in Table 1.

The N mesophase in OBABPE exhibits a marble-like texture (Fig. 3(a)). This texture is a modification of a mosaic texture. Such texture type has been observed by various scientists for the N mesophase in different liquid crystals [32–35]. This texture consists of various uniform regions. In these regions the director $\vec{n}$ has a strong definite direction. The boundaries between these uniform regions are the disclinations. As seen in Fig. 3(a), in this texture the thread-like formations, which have no geometric form, also take place. These formations are the linear disclinations and are typical for the N mesophase. We can say that this texture is classic for the N mesophase. We would like to note that the thread-like formations can also arise in the SmC mesophase. But in the N mesophase the thread formations divide separate uniform regions, which are optically biaxial; in the SmA mesophase the abovementioned formations divide separate uniform regions, which are optically uniaxial.

The SmA mesophase in OBABPE exhibits a confocal texture (Fig. 33(b)). Such type of texture is a classic and specific texture of the SmA mesophase, and is a characteristic type for the $D_{\infty h}$ symmetry [33, 34, 36, 37]. As seen in Fig. 3(b), such texture consists of the groups of confocal formations. Optical investigations showed that these formations have a positive optical sign. The confocal textures are a sufficiently interesting type of polycrystalline textures in liquid crystals and have a complicated topology. The confocal textures can also be displayed as the fan-shaped and polygonal textures [8, 33, 38, 39].

By heating of OBABPE from a crystalline state or by cooling SmA, the texture of SmA transformed to the texture of SmC (Fig. 3(c)). As seen in Fig. 3(c), the texture of SmC consists of broken polygonal and

Table 1. Features of phase transitions in OBABPE.

Temperatures of direct phase transitions, K				Temperatures of reverse phase transitions, K			
Cr–SmC	SmC–SmA	SmA–N	N–I	I–N	N–SmA	SmA–SmC	SmC–Cr
335.4	350.2	355.7	362.1	359.9	354.0	348.1	333.5

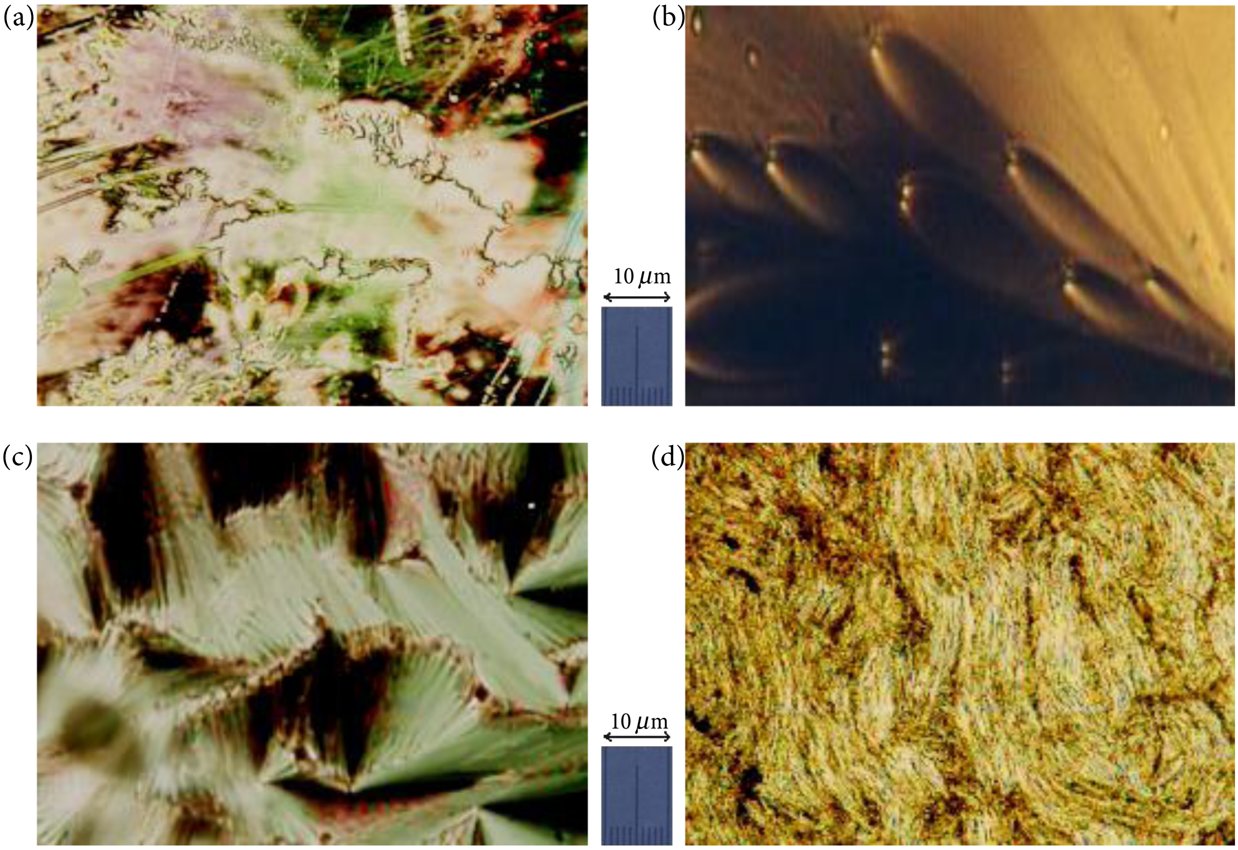

Fig. 3. Microphotographs of the textures in OBABPE (coloured online). Crossed polarizers. Magnification ×100. (a) texture of N mesophase, (b) texture of SmA mesophase, (c) texture of SmC mesophase, (d) texture of Cr state.

fan-shaped formations. Availability of broken polygonal and fan-shaped formations is a typical display of the SmC mesophase [33, 35, 36]. Such type of the texture was presented in [33, 34, 36, 38, 39], is characteristic of the C_{2h} symmetry and corresponds to the SmC mesophase. The crystalline state of OBABPE exhibits the texture which is presented in Fig. 3(d). This texture has no definite formations and uniform regions, and is sufficiently different from the textures of liquid crystalline mesophases.

In this work, the thermo-morphologic properties of the heterophase regions of direct and reverse phase transitions were investigated and the temperature widths of these regions were calculated by the CTW device. The investigations showed that these regions exhibit interesting thermo-morphologic properties (Figs. 4, 5). Namely, as seen in Figs. 4 and 5, in all observed cases there are no sharp boundaries between the neighbouring phases. Besides, as seen in these figures, in the definite temperature interval, as $\Delta T = T^{**}-T^{*}$, a simultaneous coexistence of a low temperature phase and a high temperature phase is observed. At the T^{*} temperature, the low temperature phase is stable, but the high temperature phase is metastable; at the T^{**} temperature, the high temperature phase is stable, but the low temperature phase is metastable. This fact indicates an availability of the heterophase regions of direct and reverse Cr↔SmC, SmC↔SmA, SmA↔N and N↔I phase transitions. In Fig. 6, a schematic presentation of the mesophases and positions of heterophase regions in OBABPE at the direct and reverse phase transitions is presented. Thus, for the abovementioned phase transitions thermal hysteresis takes place. Availability of thermal hysteresis and T^{*} and T^{**} temperatures at the phase transitions in liquid crystals was theoretically predicted in [11, 14, 38, 40–46] and was experimentally observed in [47–56].

The investigations showed that the morphologic properties of mesophases at the direct and reverse phase transitions and the appearance of heterophase regions are different (Figs. 4, 5). Besides, estimation of the temperature widths of heterophase regions, which was done by the CTW, showed that these widths are different when heating OBAPBE from the crystalline state to isotropic liquid and when cooling this liquid crystal from the isotropic liquid to crystalline state. Namely, widening of the temperature width of heterophase regions in the cooling process of liquid crystal under investigation was observed. Thus,

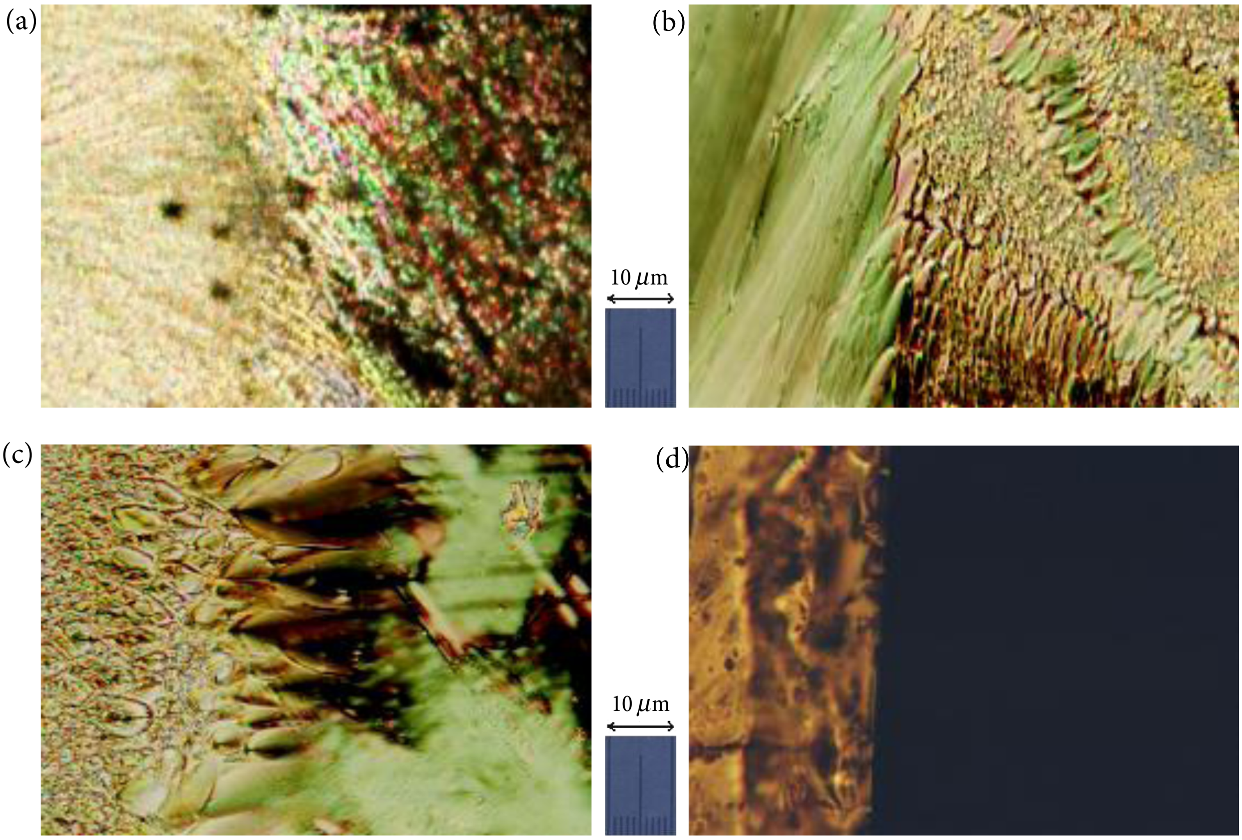

Fig. 4. Microphotographs of the heterophase regions of direct Cr–SmC (a), SmC–SmA (b), SmA–N (c) and N–I (d) phase transitions (coloured online). Crossed polarizers. Magnification ×100.

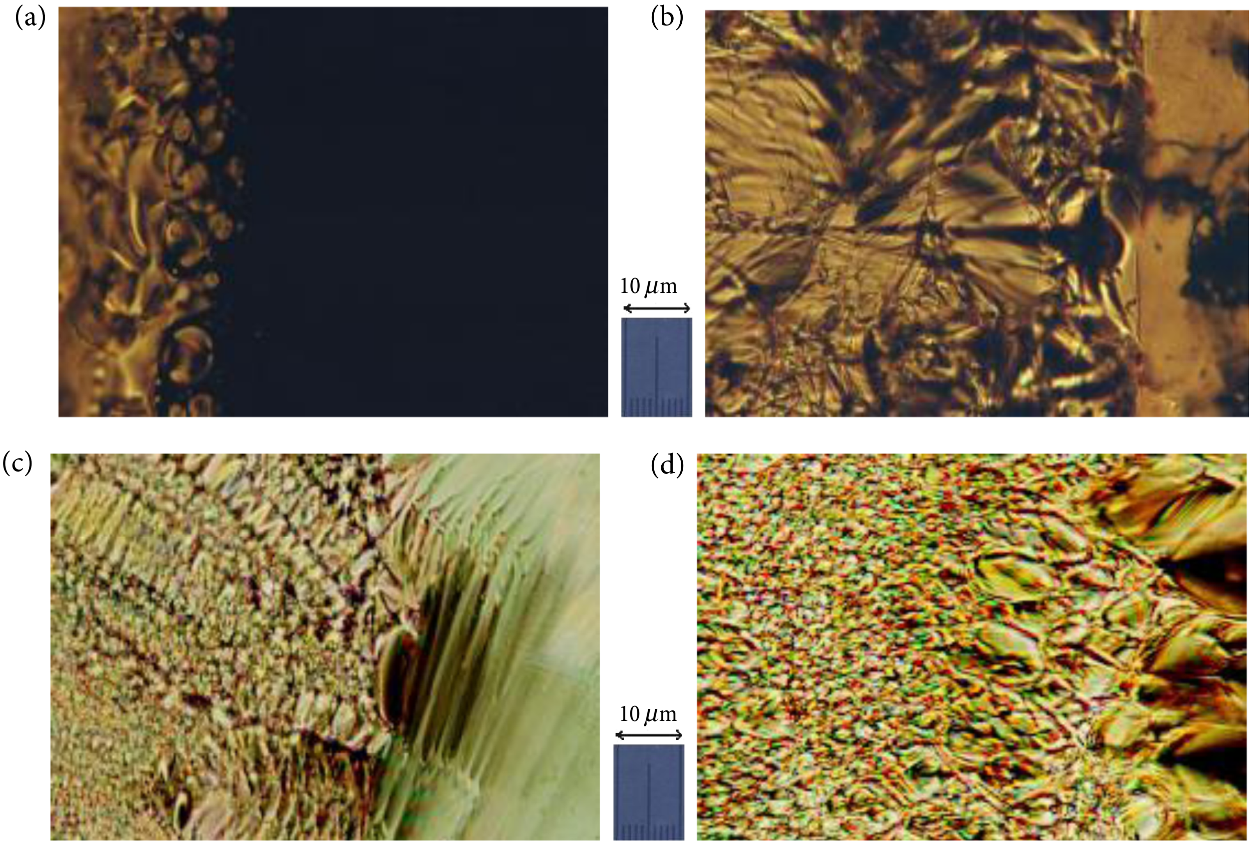

Fig. 5. Microphotographs of the heterophase regions of reverse I–N (a), N–SmA (b), SmA–SmC (c) and SmC–Cr (d) phase transitions (coloured online). Crossed polarizers. Magnification ×100.

the investigations showed that for the temperature widths of the heterophase regions of direct and reverse Cr↔SmC, SmC↔SmA, SmA↔N and N↔I phase transitions thermal hysteresis takes place. The temperature widths of heterophase regions for the abovementioned phase transitions are presented in Table 2.

The phase transition between SmC and SmA mesophases was theoretically investigated in [8, 16, 45, 57–67]. Almost in most of these works the SmC–SmA phase transition appears to be of the second order. But in [8, 16, 57, 64] this transition is presented as the first order and in [45, 66, 67] this transition is considered as both the first and second order transition. Availability of the heterophase regions of SmC–SmA phase transition and availability of thermal hysteresis, which were observed in the present work, are a typical display of the first order transition [8, 11, 13,

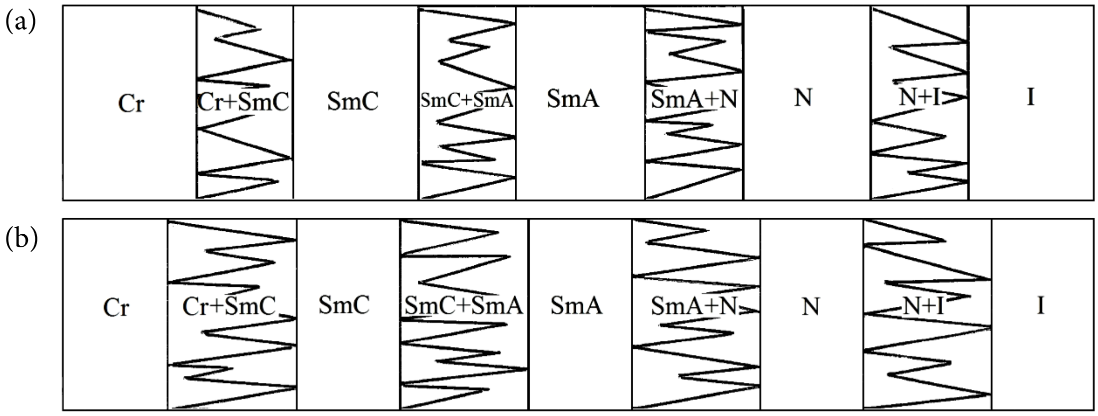

Fig. 6. Schematic representation of mesophases and heterophase regions at the direct (a) and reverse (b) phase transitions in OBABPE.

Table 2. Temperature widths of the biphasic regions of phase transitions in OBABPE.

Temperature widths of the heterophase regions for direct phase transitions, K			Temperature widths of the heterophase regions for reverse phase transitions, K		
SmC–SmA	SmA–N	N–I	I–N	N–SmA	SmA–SmC
1.21	0.83	0.74	0.95	1.22	1.39

55]. We would like to emphasize that the heterophase region and the thermic hysteresis for the SmC–SmA phase transition were also observed earlier for compounds with the alkenic terminal group chain in [49].

The phase transition between SmA and N mesophases has been theoretically investigated in a lot of works, e. g. [8, 10, 11–14, 55, 68–77]. In these works it is suggested that the SmA–N transition could be of the first or the second order. In an elegant description of the SmA–N phase transition [70–72], possibilities for the first order or second order transitions have been considered. In [38, 46, 57, 70–72, 76–78] two cases, when $T_{NA} \ll T_{NI}$ and $T_{NA} \approx T_{NI}$, have been considered. (Here T_{AN} is the SmA–N phase transition temperature; T_{NI} is the N–I phase transition temperature.) For the first case, the translational order parameter $|\psi|$ unremittingly decreases to zero at the SmA–N phase transition, and the orientational order parameter Q exhibits some fluctuation at this transition. This case corresponds to the abovementioned second order phase transition. For the second case, both the $|\psi|$ and Q parameters sharply decrease at the SmA–N phase transition. This case corresponds to the abovementioned first order phase transition. In [70–72] the quantitative criterion for the first order and the second order SmA–N phase transitions, which is connected with the T_{AN} and T_{NI} temperatures, is presented. Namely, for the $\beta = \frac{T_{AN}}{T_{NI}} < 0.87$ correlation this transition must be of the second order, but for the $\beta = \frac{T_{AN}}{T_{NI}} < 0.87$ correlation this transition must be of the first order.

In accordance with the data, which are presented in Table 1, the β_{d} parameter for the direct SmA–N phase transition was determined as $\beta_{\mathrm{d}} = \frac{T_{AN}}{T_{NI}} = 0.98$ and for the reverse N–SmA phase transition the β_{r} parameter was determined also as $\beta_{\mathrm{r}} = \frac{T_{NA}}{T_{IN}} = 0.98$. These values of the β parameter indicate that the SmA↔N phase transition in OBABPE is the first order transition. Furthermore, availability of the thermic hysteresis and the heterophase regions for the direct and reverse phase transition between SmA and N mesophases is an additional fact characterizing the first order of the abovementioned transition in OBABPE.

The phase transition between the N mesophase and isotropic liquid, which has the most common peculiarities, was the object of theoretical studies by various scientists, e. g. [8, 10, 11, 13, 14, 16, 19, 38, 76]. In these works it is shown that the N–I phase transition is the first order transition. This transition is characterized by an existence of the heterophase region with temperature limits as T^{*}_{NI} and T^{**}_{NI}. The T^{*}_{NI} is a low temperature limit; at this temperature the low temperature state (i. e. N mesophase) is stable, but the high temperature state (i. e. isotropic liquid) is metastable. The T^{**}_{NI} is a high temperature limit; at this temperature the high temperature state (i. e. isotropic liquid) is stable, but the low temperature state (i. e. N mesophase) is metastable. In the $\Delta T = T^{*}_{NI}–\mathrm{T}^{*}_{NI}$ temperature interval a simultaneous coexistence of the N mesophase and isotropic liquid takes place.

In this work we are interested in the temperature behaviour of the OT of OBABPE in a large temperature interval for the heating and cooling processes. Investigations showed that such dependences for both the heating and cooling processes exhibit a nonlinear character (Fig. 7). As seen in Fig. 7, these dependences can be considered in some stages. Namely, by heating of the sample a fluent increase of OT in the regions of SmC, SmA and N mesophases was found. At the SmC–SmA, SmA–N and N–I phase transition regions abrupt changes of the OT were observed. A bigger OT was found for the nonordered state of OBABPE, i. e. for the isotropic liquid state. By cooling of the sample a fluent decrease of OT in the regions of N, SmA and SmC mesophases was found. During the cooling process abrupt changes of the OT were observed in the regions of I–N, N–SmA and SmA–SmC phase transition regions. By comparison of the temperature dependences of OT (Fig. 7) with the character of the heterophase regions of the abovementioned phase transitions (Figs. 4, 5) it can be concluded that jumps of the OT correspond to the regions of texture transformations in the heterophase regions of these transitions.

In this work, estimation of the mean optical density (OD) of textures in OBABPE was determined. The OD is an important parameter for the system, which can be used in optical devices. As known, this parameter is connected with the OT and optical absorption. For this estimation, bound values for the full brightness I_{b} (minimum density) and full darkness I_{d} (maximum density) were determined.

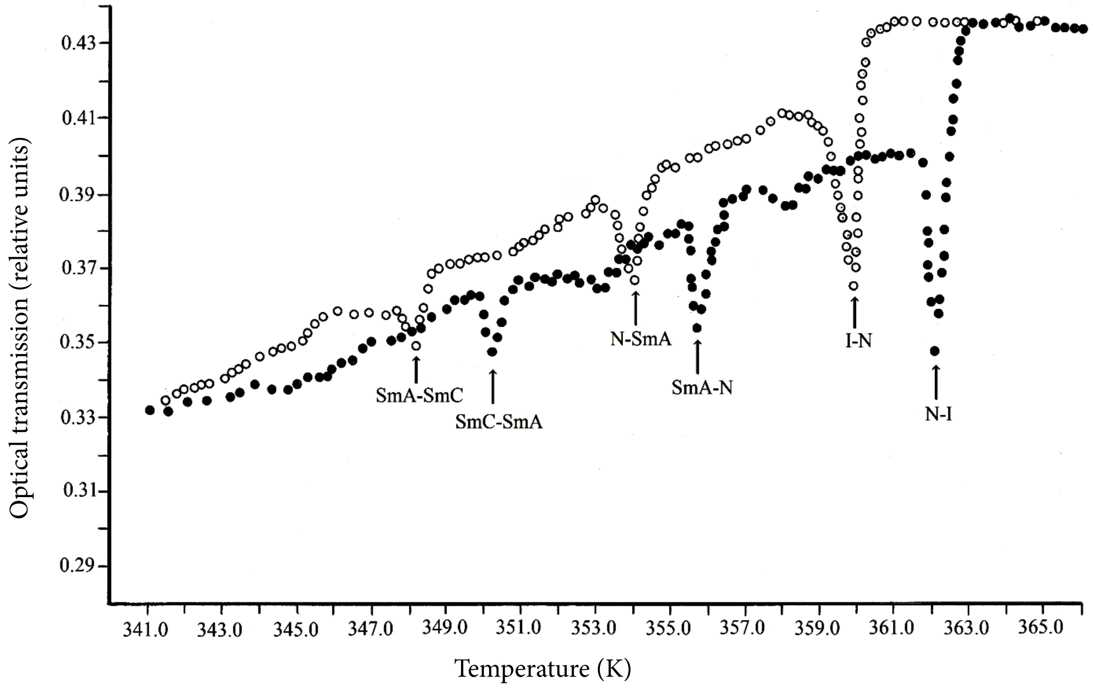

Fig. 7. Dependences of the OT vs temperature. These dependences were obtained without polarizers.

The full brightness was obtained in the situation of the planar aligned sample, which was placed between the crossed polarizer and analyzer in the position of the optical axis of the sample as $\theta = 45°$ to the polarizer and the analyzer (Fig. 8(a)). The full darkness was obtained in the situation with a sample of the isotropic liquid state. The sample in this case was placed between the crossed polarizer and analyzer in the free position (Fig. 8(b)).

The diagram of mean OD for the Cr state and SmC, SmA and N mesophases in OBABPE is presented in Fig. 9. Non-aligned textures of the Cr state, SmC, SmA and N mesophases were used. As seen in Fig. 9, the N mesophase is the least optical dense state and Cr is the most optical dense state in OBABPE. Besides, the SmC mesophase has a greater OD than that in the SmA mesophase. It is interesting to compare the symmetry of the abovementioned states – Cr (G^n_m),

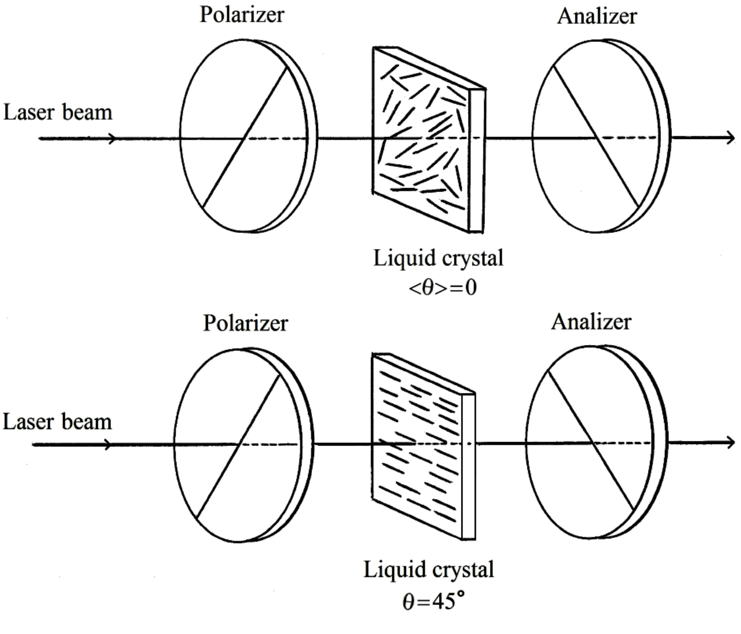

Fig. 8. Schematic representation of the position of the sample for full brightness (a) and full darkness (b).

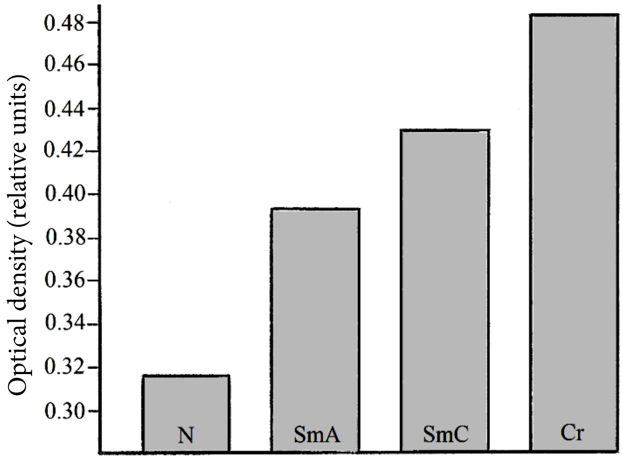

Fig. 9. Diagram of the mean optical density of phase states in OBABPE.

SmC (C_{2h}), SmA ($D_{\infty h}$), N (D_{∞}), I (K_h or K) – with their OD. A correlation of these states can be presented as $OD_I < OD_N < OD_{SmA} < OD_{SmC} < OD_{Cr}$. Thus it is clear that a more uniform and ordered state in OBABPE has bigger values of the OD.

4. Conclusions

In the present research, the thermo-morphologic properties of mesophases and peculiarities of the heterophase regions of direct and reverse phase transitions in 4-octyloxy-benzoic acid 4′-buthoxy-phenyl ether (OBABPE) liquid crystalline material, exhibiting the multiple phase transitions, were investigated. The analysis of the typical textures of SmC, SmA and N mesophases and their temperature transformations is given. The shift of temperatures of the reverse I–N, N–SmA, SmA–SmC and SmC–Cr phase transitions to lower temperatures and thermal hysteresis for the temperature widths of the heterophase regions of direct and reverse Cr↔SmC, SmC↔SmA, SmA↔N and N↔I phase transitions were observed. The shifts of the abovementioned phase transition temperatures were as 1.9 K (for Cr↔SmC transition), 2.1 K (for SmC↔SmA transition), 1.7 K (for SmA↔N transition) and 2.2 K (for N↔I transition).

Temperature widths of the direct and reverse phase transitions were determined with high accuracy by an original method, i. e. the method of capillary temperature wedge.

The temperature dependences of the optical transmission (OT) during *crystal → smectic C → smectic A → nematic → isotropic liquid → nematic → smectic C → smectic A → crystal* phase transitions were studied. Fluent changes of the OT with an increase of temperature in the regions of SmC, SmA and N mesophases were observed. At the SmC–SmA, SmA–N and N–I phase transition regions abrupt changes of the OT were found. Bigger OT was observed for the less ordered state of OBABPE, i. e. for the isotropic liquid state, lower OT was for the more ordered state of OBABPE, i. e. for the SmC mesophase.

The diagram of the mean optical density (OD) for the Cr state and SmC, SmA and N mesophases in OBABPE was estimated. The estimation showed that $OD_I < OD_N < OD_{SmA} < OD_{SmC} < OD_{Cr}$ correlation took place in OBABPE, i. e. more uniform and ordered thermodynamic state in OBABPE had bigger values of the OD.

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