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Effect of Absolute Configuration on the Formation of TGB_A^* Phase in Liquid Crystallines Containing Two Stereogenic Centres

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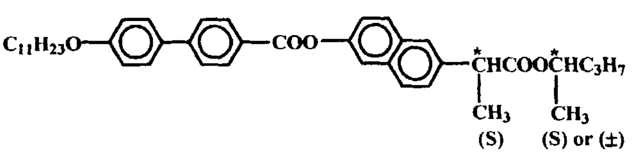
Abstract Our previous work has shown that a homologous series of chiral materials containing two sereogenic centres, (R)-2-pentyl (S)-2-(6-(4-(4-alkoxyphenyl)benzoyloxy-2-naphthyl)propionates (R,S)PmPBNP (m=7-14), exhibited TGB_A^* phase. To understand the effect of relative configuration of two stereogenic centres at chiral tail on the appearance of TGB_A^* phase, two stereoisomers of (R,S)P11PBNP, (S,S)P11PBNP and (±,S)P11PBNP were synthesized and their mesophases investigated. The results showed that both isomers also possessed TGB_A^* phase with the phase sequence of I-N^*-TGB_A^*-S_A^*-S_C^*-C. The temperature range of TGB_A^* phase in (R,S)-isomer was significantly larger than that in (S,S)-isomer, indicating the stability of TGB_A^* phase was affected by the relative spatial configuration of the second chiral centre at chiral tail. In addition, the temperature range of TGB_A^* phase in (±,S)-isomer was larger than (S,S)-isomer, suggesting that an introducing methyl branched group at the external side of mono chiral centre could cause a weakening of smectic layer order and result in an occurrence of TGB_A^* phase. The measured Ps values in the S_C^* phase from the isomers, also indicated the absolute configuration of second chiral centre plays an important role in determining the magnitude of spontaneous polarization.

INTRODUCTION

The optically active mesophases such as the blue phase[1], ferroelectric[2], ferrielectric[3] and antiferroelectric[4] S_C^* phases, and twisted grain boundary (TGB; TGB_A^*, TGB_C and TGB_C^*) phases[5-8] have been found in the liquid crystals system after the discovery of the first optically active N^* phase in 1888.[9] The investigation in the relationship between the chirality and liquid crystals, especially the TGB phases, has become one of most important research areas in this field. For examples, the systemic investigations of propionates[5-8, 11-12] were obtained certain structural relationships on the formation the TGB phases and reviewed.[10] These factors divided according to the molecular structure of mesogen were the length of nonchiral terminal alkyl chain[5,6], the length of external side terminal alkyl chain at chiral centre[10], the effect of lateral substituted group on the core[11] and the linking groups[10,12] etc. However, only a few investigations[13] had been put forward to the effect of numbers of chiral centers and their absolute configurations on the occurrence of these mesophases. It should be noted that it is well known in the ferroeletric S_C^* phase. Our previous study[14] in a series homologous of materials, (R)-2-pentyl (S)-2-(6-(4-(4-alkoxyphenyl)benzoyloxy)-2-naphthyl)propionates, (R,S)PmPBNP (m=7-14), containing two sereogenic centres showed an existence of TGB_A phase with the phase sequence: I-N^*-TGB_A^*-S_A^*-S_C^* for m=7-9 and I-N^*-TGB_A^*-S_C^* for m=10-14. In order to understand the effect of the relative spatial configuration of the chiral tail on the formation of TGB_A^* phase, two stereoisomeric compounds of (R,S)P11PBNP, (S)-2-pentyl (S)-2-(6-(4-(4-undecyloxyphenyl)benzoyloxy)-2-naphthyl)propionates, (S,S)P11PBNP, and 2-pentyl (S)-2-(6-(4-(4-undecyloxyphenyl)benzoyloxy)-2-naphthyl)propionates,(±,S)P11PBNP, were synthesized for the study. The relationship between the absolute configurations



on the appearance of TGB_A^* phase was established in terms of the mesomorphic properties of diastereomeric mixtures of (R,S)- and (S,S)- isomers. In addition, the physical properties of ferroelectric S_C^* were also measured and discussed.

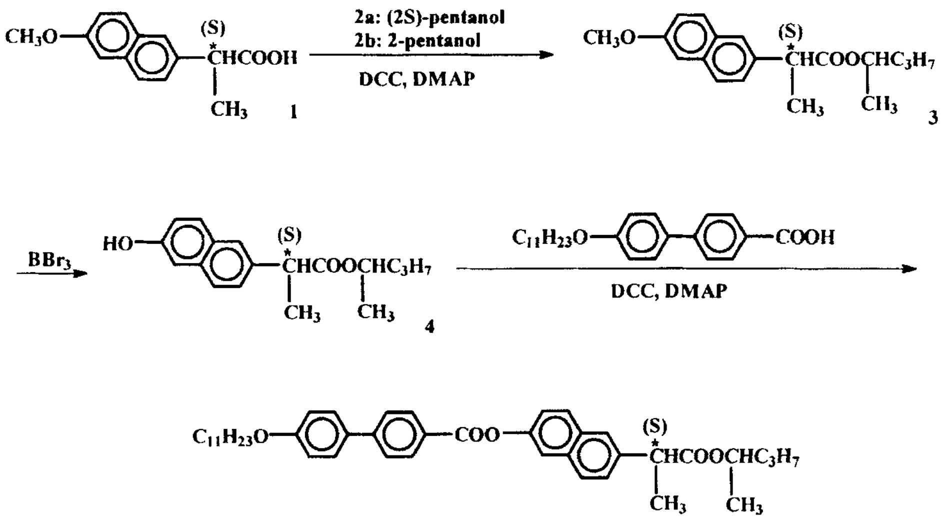
CHARACTERIZATION AND PREPARATION OF MATERIALS

The chemical structures for intermediates and target compounds were analyzed by nuclear magnetic resonance spectroscopy using a JEOL EX-400 FT-NMR spectrometer. The purity was checked by thin-layer chromatography and further confirmed the purity of final products were done by the elemental analysis using a PERKIN-ELMER 2400 spectrometer. The magnitudes of the specific rotation were measured in dichloromethane using a JASCO DIP-360 digital polarimeter.

Mesophases of diastereomers and mixtures were principally identified by observing the textures in the glass slides, the homeotropic alignment cell with 7.5μm thickness purchased form Linkam Instr. Ltd and homogeneously alignment cell with 2μm thickness produced by the E.H.C. Co. using a NIKON MICROPHOT-FXA optical microscopy under crossed polarizers with a METTLER FP82-HT hot stage in connection with a METTLER FP80-HT heat controller. The ferroelectric phase was further identified by switching behaviour and dielectric property measurements[15] in parallel alignment liquid crystal cells with 2μm and 25μm thickness purchased from E.H.C. Co.

Synthesis

The synthetic procedures for preparing chiral materials (S,S)P11PBNP and (±,S)P11PBNP were the same as that previously described for (R,S)P11PBNP[14] and were outlined in Scheme 1. The acid **1** was esterified with alcohol **2** in the presence of N,N'-dicyclohexyl carbodiimide (DCC) and 4-dimethylaminopyridine (DMAP) to produce an intermediate ester **3**. The methoxy group of this ester was demethylated by the treatment of tribromoborane (BBr_3), and the resulting hydroxy group of chiral compound **4** was subsequently esterified with 4-(4-undecyloxyphenyl)benzoic acid by the treatment of DCC and DMAP to produce the target compounds.



Scheme 1 Synthetic procedures for chiral materials (S,S)P11PBNP and (±,S)P11PBNP

<u>(S)-2-Pentyl (S)-2-(6-methoxy-2-naphthyl)propionate 3a, (S,S)PMNP</u>

(S)-2-Pentanol **2a** (27.5mmol), DCC (27.5mmol) and DMAP (2.5mmol) were added to a solution of acid **1** (25mmol) in 100ml dry dichloromethane. The reaction mixture was stirred continuously at room temperature for 3 days. The precipitates were filtrated and wash with dichloromethane. The filtrate was successively washed with 5% acetic acid, 5% aqueous sodium hydroxide and water and then dried by anhydrous magnesium sulfate. The product **3a**, (S,S)PMNP, was isolated by column chromatography over silica gel (70-230 mesh) with dichloromethane as eluent and dried in vacuo with the yield of 82% and used directly for the follow-up synthesis without purification. ^{1}H-

NMR (400MHz, $CDCl_3$): δ 0.7-1.7 (m, 13H, R**CH$_2$CH$_3$**), 3.87 (s, 3H, O**CH$_3$**), 3.9-4.2 (q, 1H, Arc**H**COO), 4.9-5.1 (m, 1H, COOC**H**), 7.1-7.8 (m, 6H, Ar**H**).

2-Pentyl (S)-2-(6-methoxy-2-naphthyl)propionate **3b**, (±,S)PMNP

The synthetic procedures were the same as that for **3**a. Yield: 90%. ^{1}H-NMR (400MHz, $CDCl_3$): δ 0.7-1.7 (m, 13H, R**CH$_2$CH$_3$**), 3.9 (s, 3H, O**CH$_3$**), 3.8-4.0 (q, 1H, Arc**H**COO), 4.9-5.0 (m, 1H, COOC**H**), 7.0-7.8 (m, 6H, Ar**H**).

(S)-2-Pentyl (S)-2-(6-hydroxy-2-naphthyl)propionate **4**, (S,S)PHNP

Tribromoborane (2.5ml) was added to a solution of **3**a (0.015mol) in 57ml dry dichloromethane at -20°C and further stirred for 5min. The reaction was then stirred 50min at 0°C. After diluting with dichloromethane (114ml), the solution was poured into a mixture of saturated ammonium chloride (57ml) and ice (57g). The organic layer was separated from aqueous layer and washed twice with brine-ice. The crude product **4**a (S,S)PHNP was dried over anhydrous sodium sulfate and concentrated in vacuo. The product was purified twice by column chromatography over silica gel (70-230 mesh) with dichloromethane as eluent and then collected after recrystallization from hexane and dried in vacuo with the yield of 64%. ^{1}H-NMR (400MHz, $CDCl_3$): δ 0.7-1.7 (m,13H, R**CH$_2$CH$_3$**), 3.8 (q, 1H, Arc**H**COO), 4.9-5.0 (m, 1H, COOC**H**), 5.26 (s, 1H, O**H**), 7.0-7.7 (m,6H, Ar**H**). Elemental analysis: Calc.: C, 75.50%; H, 7.74%. Found: C, 75.41%; H,7.70%.

2-Pentyl (S)-2-(6-hydroxy-2-naphthyl)propionate **4b**, (±,S)PHNP

The synthetic procedures were the same as that for **4**a. Yield: 70%. ^{1}H-NMR (400MHz, $CDCl_3$): δ 0.7-1.7 (m,13H, R**CH$_2$CH$_3$**), 3.8 (q, 1H, Arc**H**COO), 4.9-5.0 (m, 1H, COOC**H**), 6.3 (s,1H, O**H**), 7.0-7.7 (m,6H, Ar**H**). Elemental analysis: Calc.: C, 75.50%; H, 7.74%. Found: C, 75.62%; H,7.72%.

(S)-2-Pentyl (S)-2-(6-(4-(4-undecyloxyphenyl)benzoyloxy)-2-naphthyl)propionate, (S,S)P11PBNP

compound **4a** (S,S)PHNP (1.05mmol) was reacted with 4-(4'-undecyloxyphenyl)benzoic acid (1.16mmol) by the treatment of DCC (1.26mmol) and DMAP (0.1mmol) in dry tetrahydrofuran (4ml) at room temperature for 5 days. After the work-up procedures, the product (S,S)P11PBNP was purified twice by column chromatography with dichloromethane as eluent and then collected after recrystallization from ethanol and dried in vacuo with the yield of 84%. ^{1}H-NMR (400MHz, $CDCl_3$): δ 0.8-1.9 (m, 40H, R**CH_2CH_3**), 3.8 (q. 1H, Arc**H**COO), 4.0 (t, 2H, O**CH_2**), 4.9 (m, 1H, COOC**H**), 6.9-8.2 (m,6H, Ar**H**). Elemental analysis: Calc.: C, 79.61%; H, 8.61%. Found: C, 80.35%; H, 8.75%. Specific rotation $[\alpha]_D^{30}$(0.6g/100ml): +17.41°.

2-Pentyl (S)-2-(6-(4-(4-undecyloxyphenyl)benzoyloxy)-2-naphthyl)propionate, (±,S)P11PBNP

The synthetic procedures were the same as that for (S,S)P11PBNP. Yield: 90%. ^{1}H-NMR (400MHz, $CDCl_3$): δ 0.1-1.8 (m, 40H, R**CH_2CH_3**), 3.8-3.9 (q. 1H, Arc**H**COO), 4.1 (t, 2H, O**CH_2**), 4.9-5.0 (m, 1H, COOC**H**), 7.1-8.2 (m,6H, Ar**H**). Elemental analysis: Calc.: C, 79.61%; H, 8.61%. Found: C, 79.58%; H, 8.68%. Specific rotation $[\alpha]_D^{30}$(0.6g/100ml): +5.63°.

Preparation of diastereomeric mixtures

Diastereomeric mixtures, except (±,S)-form, were prepared by mixing (R,S)-form and its diastereomeric compound (S,S)-form into a vial, adding 2-5ml dry dichloromethane into the vial and vigorously agitating the solution for about 2 min, and then dried in vacuo.

RESULTS

Mesomorphic properties

The samples sandwich-packed in two untreated glass slides were conducted in the heating and cooling runs at 2°C/min scanning rate for the microscopic observation. The mesophases and their corresponding phase transition temperatures principally identified by observing textures of the materials under crossed polarizing microscope were summarized in Table 1.

The mesophases of the (±,S)- and (S,S)- isomers were enantiotropic and possessed the same phase sequence: I-N*-TGB$_A^*$-S$_A^*$-S$_C^*$-C. The TGB$_A^*$ phase of (±,S)- and (S,S)-isomers was mediated between the N* and the S$_A^*$ transition unlike that of (R,S)-isomer was an intermediary phase between the N* and the S$_C^*$ transition. Examples of microscopic textures

Table 1 Phase transition temperatures (°C) of binary mixture of (S,S)- and (R,S)-isomers obtained by the microscopic observation on cooling

wt% of (S,S)-form	phase sequence											
	I		N*		TGB$_A^*$		S$_A^*$		S$_C^*$		C	mp
0.00	•	137.1	•	134.2	•		-	116.3	•	55.8	•	86.0
2.86	•	136.8	•	134.0	•	129.3	•	116.8	•	65.4	•	85.7
5.62	•	136.9	•	133.9	•	129.2	•	115.0	•	60.6	•	85.2
10.57	•	136.6	•	133.8	•	128.2	•	115.4	•	62.8	•	84.9
31.03	•	136.9	•	133.4	•	129.3	•	113.5	•	62.8	•	83.2
50.00	•	135.3	•	131.5	•	128.6	•	110.7	•	65.3	•	81.1
71.29	•	134.8	•	130.9	•	128.5	•	107.9	•	64.4	•	80.2
85.12	•	132.5	•	129.6	•	127.3	•	107.3	•	65.1	•	83.1
100.00	•	129.4	•	127.2	•	126.1	•	106.8	•	73.6	•	87.1

observed from (±,S)-isomer were displayed in Plates 1(a)-(d). The texture of N^* phase was characterized by the paramorphotic and planar texture (Plate 1(a)). Cooling to the TGB_A^* phase, the paramorphotic texture of N^* phase changed to the filament texture and the planar texture altered to more viscous and blurred planar texture (Plate 1(b)). When cooling to S_A^* phase, the focal-conic and homeotropic texture coexisted with filament texture (Plate 1(c)), appeared to be resembled to that observed by Bouchta et al.[12] Continuously lowing the temperature to the S_C^* phase, the disclination lines appeared in the focal-conic region and the filament texture disappeared and converted to the iridescent planar texture (Plate 1(d)). Similar mesomorphic textures were also observed in the (S,S)-isomer and the diastereomeric mixtures.

Mesophases of (±,S)-isomer were also characterized by the textures (Plates 2(a)-(d)) in the cells with parallel alignment layer. The microscopic texture exhibited significantly colored Bragg-like reflection with shorter selective reflecting wavelength of light in the N^* phase and longer reflecting wavelength of light in the TGB_A^* phase as shown in Plates 2(a) and 2(b), respectively. When the temperature closed to the TGB_A^*-S_A^* transition, the selectively reflecting phenomena were no longer observed due to the variation of helical pitch in the TGB_A^* phase. Upon further cooling, a poorly aligned texture (Plate 2(c)) of S_A^* phase resembled to that observed by Nishiyama et al.[16] was found. This poor alignment of the S_A^* phase was attributed due to the twist ordering of the molecules in the TGB_A^* phase. The textures of S_C^* phase obtained from (±,S)- and (S,S)- isomers (Plate 2(d)) were different with respect to the parquet texture (Plate 3(a)) obtained from (R,S)-isomer. The parquet texture could be gradually altered to the surface-stabilized state when the electric field was steadily and increasingly applied as shown in Plates 3(b) at 9.5V, 3(c) at 14.2V and 3(d) at -14.2V. The fieldinduced texture was irreversible upon removal of electric field. The threshold hold voltages for the (R,S)-, (±,S)- and (S,S)- isomers were 6.5V, 4.9V and 3.8V, respectively.

Miscibility study

Further investigation on the effect of absolute configuration of the second chiral centre on the occurrence of TGB_A^* phase was conducted by investigating the mesomorphic properties of diastereomeric mixtures. The mesophases and corresponding phase transition temperatures were also summarized in the Table 1. The phase diagram of the mixtures was plotted in Figure 1. It showed that the clearing points were

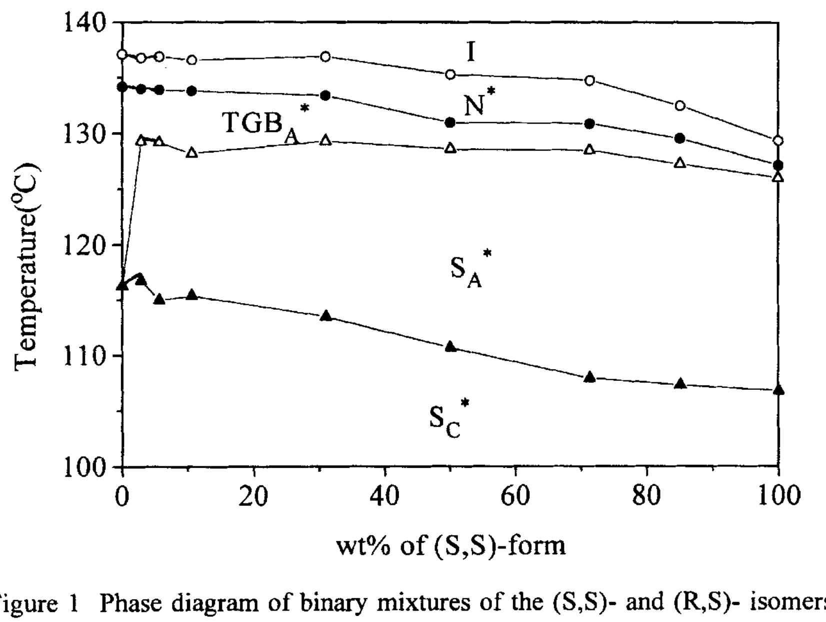


Figure 1 Phase diagram of binary mixtures of the (S,S)- and (R,S)- isomers.

decreased as the increasing concentration of (S,S)-isomer. Similar trends were also observed for all transition temperatures. The figure also indicated that the N^*, TGB_A^* and S_C^* phases were continuously miscible in the phase diagram, suggesting that the helix senses of these phases in (S,S)- and (R,S) -isomers were the same. Furthermore, it was worth to note that the most striking feature was the temperature range of TGB_A^* phase dramatically and sharply suppressed by adding a small quantity of (S,S)-isomer into the (R,S)-isomer.

Spontaneous Polarization of S_C^* phase

The physical properties of the ferroelectric S_C^* phase such as direction and magnitude of spontaneous polarization (Ps) for (R,S)-isomer and its diastereomers were measured as a function of temperature on cooling in the 2μm thickness homogeneously aligned cells. The directions of spontaneous polarization for all isomers were all negative (-) detected by the switching studies.[17] All stereoisomers showed the similar tendency in temperature dependence of spontaneous polarization. The magnitudes of spontaneous polarization as a function of reduced temperature form the Curie point (Tc) shown in Figure 2, indicated an order of (S,S)- > (±,S)- > (R,S)- isomers.

DISCUSSION

The experimental results indicated that the temperature range of TGB_A^* phase of (R,S)-, (±,S)- and (S,S)- isomers were 17.9°, 2.9° and 2.1°C, respectively. Since the formation and stability of TGB_A^* phase have been reported to be chirality dependent[18], the (R,S)-isomer should possess a larger degree of molecular chirality than its diastereomers (S,S)-isomer. Moreover, considering the pre-transitional effect caused by the underlying S_C^* phase[19], the stability of TGB_A^* phase of (R,S)-isomer should be greater than that of (S,S)-isomer. This result was the same as those of the phenyl-

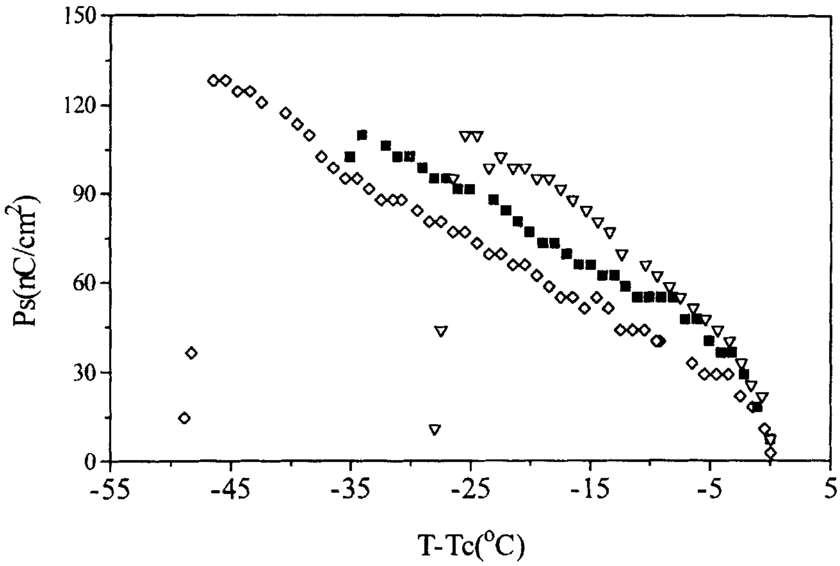


Figure 2 Temperature dependence of spontaneous polarization (Ps) of (R,S)-; ○, (±,S)-; ■ and (S,S)-; ▵.

propionate esters derived from isoleucine.[13] However, as comparing the temperature range of TGB_A^* phase between (±,S)-isomer and (S,S)- isomer, the chirality might not be the only cause for the occurrence of TGB_A^* phase. It was apparent that an introducing methyl branched group at the external side of the first chiral centre could increase the steric hindrance[10] to affect the molecular packing such that weaken the smectic layer order and form a TGB_A^* phase. Furthermore, a sharply decreasing the TGB_A^* temperature range as a small quantity of (S,S)-isomer was added to the (R,S)-isomer, demonstrated that the stability the TGB_A^* phase strongly depended on the relative spatial configuration and optical purity of the second chiral centre at the chiral tail.

The direction of Ps predicted by using the Boulder model[20], as shown in

Figure 3, suggested that the direction should be negative (-), in agreement with the

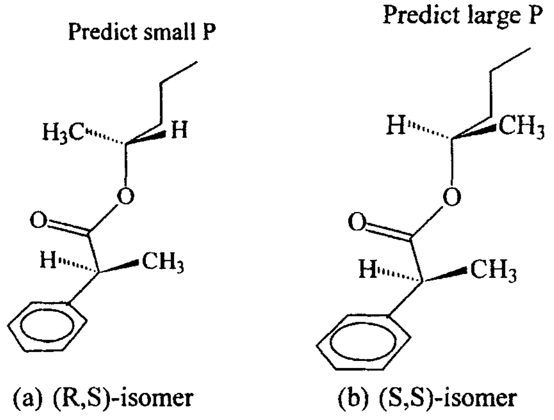


Figure 3 Boulder model for the predication of the relative spatial configuration on the diastereomeric compounds (a) (R,S)- and (b) (S,S)-isomers

measured results. Moreover, this figure also showed an obvious difference for diastereomers in relative spatial configuration at chiral tail, such that the effective transverse dipole moment produced by two methyl groups and dipolar carbonyl group[21] for the (R,S)-isomer was smaller than that for (S,S)-and (±,S)- isomers in consistent with the measured magnitudes of Ps values. This was to suggest that the absolute configuration of second chiral centre might not affect, to some degree, the magnitude of transverse dipole moment .

CONCLUSION

We have demonstrated that the formation and the stability of the TGB_A^* phase remarkably depended on the relative spatial configuration and optical purity of the chiral materials containing two stereogenic centres. Moreover, an introducing methyl branched group to the external side of the first chiral centre at the chiral tail could cause a weakening of the smectic layer order and result in the formation of TGB$_A^*$ phase.

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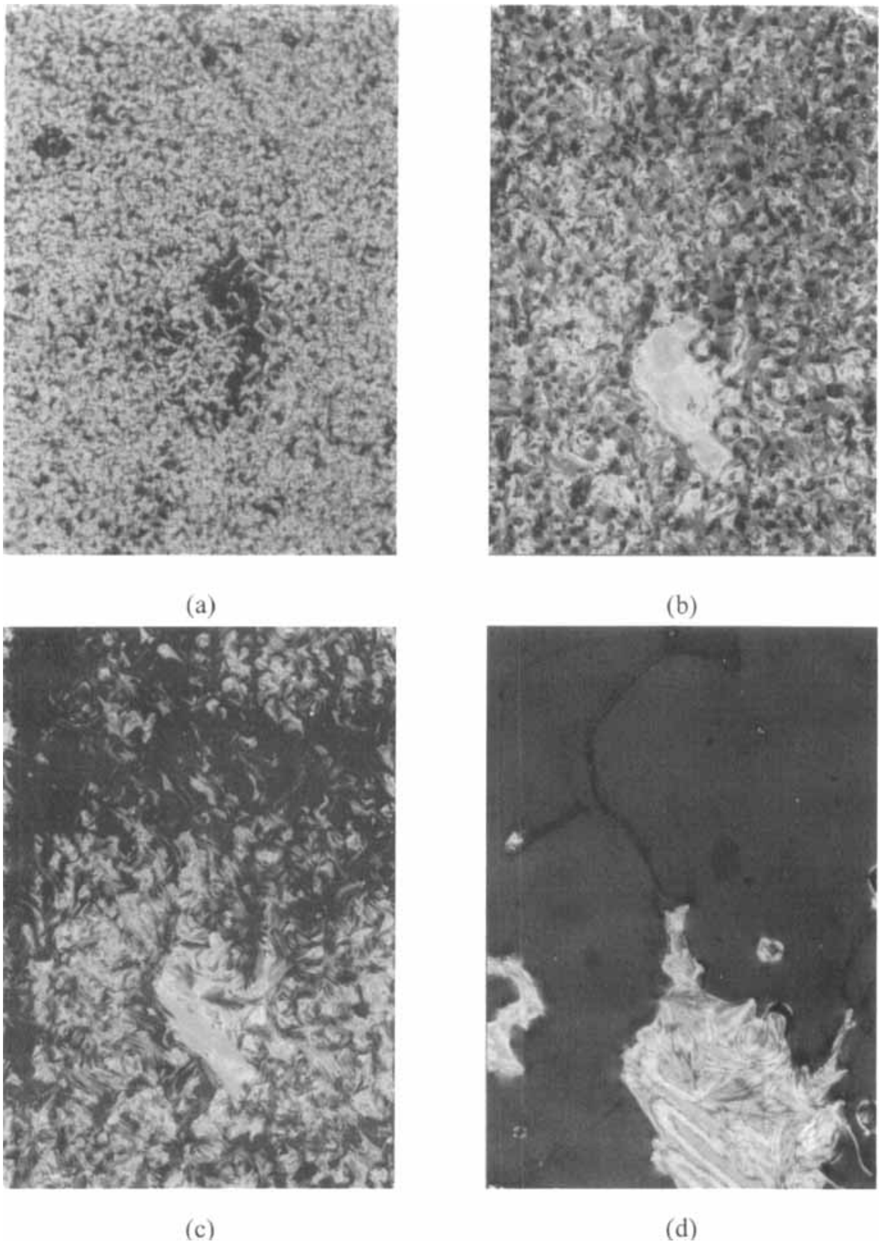
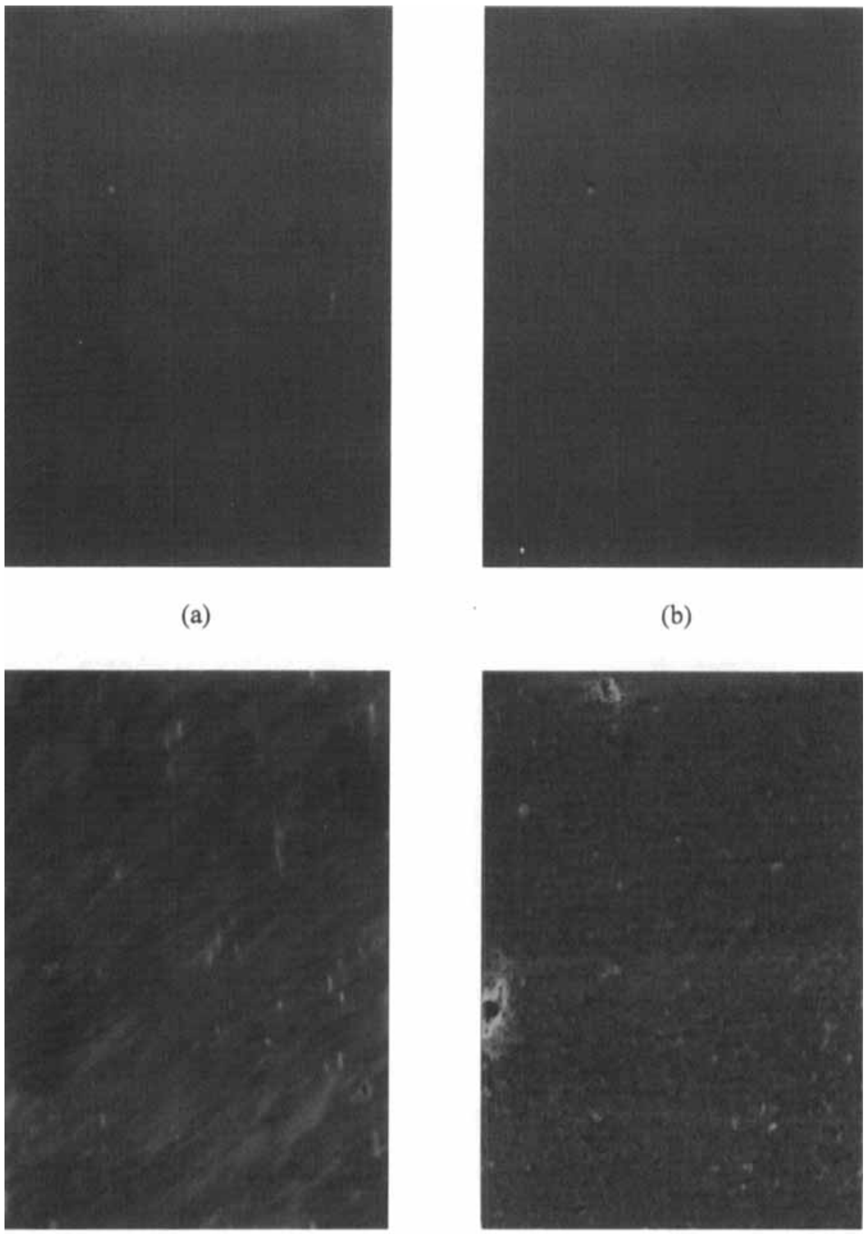


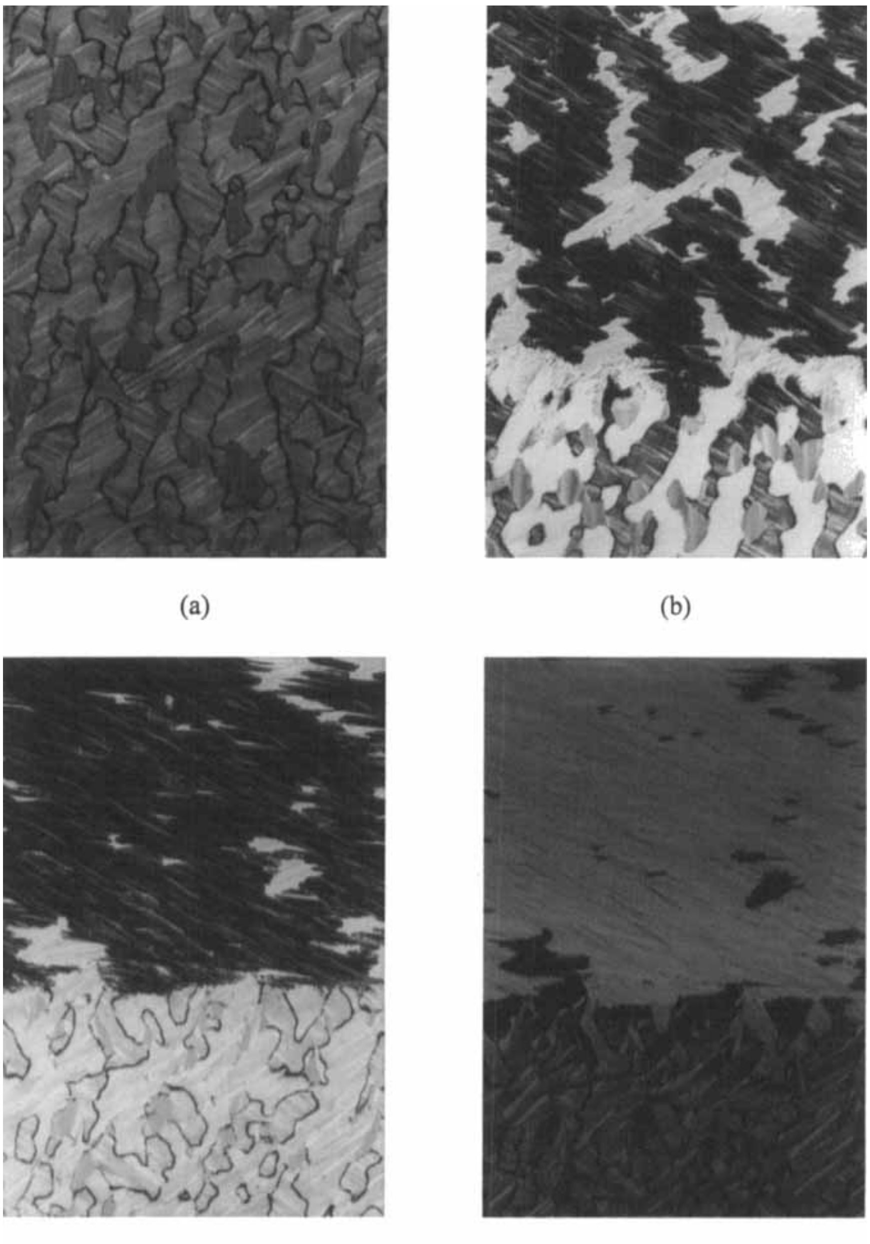
Plate 1 (a) The paramorphotic and Grandjean texture of N* phase at 133.8°C (X100), (b) the Grandjean and filament texture of TGB$_A$* phase at 129.7°C (X200), (c) the focal-conic and homeotropic texture of S$_A$* phase at 127.9°C (X200) and (d) the broken focal-conic with striated lines and planar texture of S$_C$* phase at 80.3°C (X400) of (±,S)-isomer in slides under crossed polarizers microscope. (See Color Plate IV).



(a) (b)

(c) (d)

Plate 2 The mesophase texture of (±,S)-isomer observed in the cell with homongeously aligned layer under crossed polarizers microscope. (a) N^* phase at 130.8°C (X100), (b) TGB_A^* phase at 128.5°C (X100), (c) S_A^* phase at 126.2°C (X100) and (d) S_C^* phase at 94.3°C (X100). (See Color Plate V).



(a) (b) (c) (d)

Plate 3 The texture of S_C^* phase of (R,S)-isomer with (upper part) and without (bottom part) electrode under applied voltage at T-Tc=-5°C. (a) 0V, (b) 9.5V (c) 14.16V and (d) -14.16V. (See Color Plate VI).