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Invited Lecture

Ferroelectric liquid crystalline polymers and related model compounds with a low-moderate degree of polymerization

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Ferroelectric liquid crystalline polymers (FLCPs) with a low-moderate degree of polymerization were synthesized. These had a comb structure; the main chain was polyacrylate and the side chain consisted of a flexible spacer, a core and an optically active chiral end group. They exhibited electro-optic switching times ranging from a few milliseconds to a few seconds in the S_C^* phase. As the molecular weight \bar{M}_n increased, the range of the S_C^* shifted to higher temperatures. At a given temperature, the switching time increased with \bar{M}_n .

Spontaneous polarizations P_s , apparent cone angles 2θ , electro-optic switching times τ and rotational viscosities η of some polyoxyethylene FLCPs and corresponding low molecular weight ferroelectric liquid crystal materials (FLCs) were also measured. This is the first report of polyoxyethylene FLCPs. There was no significant difference in P_s and 2θ , which shows that the arrangement of the side chains in the FLCP is similar to that of the molecules in ordinary FLC. On the contrary, τ and η for the FLCPs were 10^2 - 10^3 times as large as those for FLCs. The rotational viscosity of the FLCP was mainly dependent on the side chain structure rather than on the spacer. Therefore collisions between adjacent side chains cause large η values in FLCPs rather than hindrance to side chain gyration due to the spacer group. By combining FLCPs with ITO-coated plastic substrates, a large area matrix driven display has been made. Although an improvement in switching time is still needed, a flexible and lightweight display like a sheet of paper will be available in the near future.

1. Introduction

Since the discovery of microsecond order, fast electro-optic switching in surface stabilized ferroelectric liquid crystal (SSFLC) cells [1], FLCs have attracted much attention from the viewpoint of display application. Contrary to the eager expectation of high quality display devices, it appears that there remain many difficulties in manufacturing large area FLC cells. One such problem lies in controlling the cell thickness. To realize SSFLCs, the cell thickness is required to be a few microns [1]. The other problem lies in achieving good molecular alignment. Indeed, the layer structure of FLC is too complicated to control [2].

In order to overcome these difficulties, we have polymerized some FLCs [3]. These polymers had a comb shape and contained side chains, each of which consisted of a flexible spacer, a core and an optically active chiral end group. Similar investigations have been reported by Shibaev *et al.* [4, 5], Decobert *et al.* [6-11],

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Hahn *et al.* [12, 13], Zentel *et al.* [14–19] and Keller [20]. As we expected, these polymers show ferroelectricity [3] and have the advantage of easy handling compared with low molecular weight FLCs. We can readily obtain a thin film by coating and achieve good molecular alignment by shearing treatment [21]. Hereafter, we will refer to a ferroelectric liquid crystalline polymer as ‘FLCP’ and distinguish it from an ordinary low molecular weight ferroelectric liquid crystal, referred to as ‘FLC’.

Recently, FLCPs have attracted a good deal of attention from the viewpoint of basic liquid crystal polymer science and its potential for application [22–39]. We have previously reported some electro-optic characteristics of FLCPs such as switching time, tilt angle, spontaneous polarization [3, 21], dependence of helical pitch on molecular structure [40] and smectic layer structure [21]. Recently, we first reported the application of FLCP to display devices [21]. When applied in display devices, the FLCP has the disadvantage of slow electro-optic response, and it is important to know the switching mechanism in FLCPs in order to improve the electro-optic switching time. The mechanism in FLCPs has, however, not yet been clearly elucidated.

In this paper we introduce our synthesized FLCPs and their electro-optic characteristics. In addition, by comparing FLCPs with the corresponding FLCs, we propose a new model for the switching mechanism that can explain the slow response in FLCPs. Lastly, we introduce our attempt to apply FLCPs to display devices.

2. Experimental

2.1. Preparation of FLCPs and FLCs

The molecular structure of FLCP **P0** that we synthesized is given in figure 1. The synthesis was reported previously [3, 40]. In order to investigate the dependence of electro-optic characteristics on molecular weight, we prepared four different polymers **P0** whose number-average molecular weights \bar{M}_n measured by GPC were 3100, 4800, 5900 and 19 000, respectively.

In order to compare FLCPs with FLCs, we prepared the three FLCs **L1** (1MB), **L2** (EL) and **L3** (1MH) shown in figure 1, where the letters in parentheses refer to the corresponding chiral end groups, i.e. 1MB:1-methylbutyloxycarbonyl, EL:ethyl-lactateoxycarbonyl and 1MH:1-methylheptyloxycarbonyl. The three FLCs had the same molecular structure except for the chiral end group. We also prepared the corresponding three polymers **P1** (1MB), **P2** (EL) and **P3** (1MH) shown in figure 1. Using polyoxyethylene as the main chain we succeeded in obtaining a large and stable temperature range of S_C^* phase. This is the first report of a polyoxyethylene FLCP as far as we know. The side chains of the three FLCPs **P1**, **P2** and **P3** had the same molecular structures as the corresponding FLCs **L1**, **L2** and **L3**, respectively.

In this study we chose a biphenyl benzoate core group so as to obtain a large and stable temperature range of the S_C^* phase in both the FLC and the FLCP. In general, a FLC that contains a biphenyl or a phenyl benzoate as a core group exhibits a small S_C^* temperature range that tends to be monotropic. On the contrary, a FLCP that contains such a core group exhibits a rather large stable temperature range of the S_C^* phase, which shows that polymerization stabilizes the S_C^* phase. A core consisting of biphenyl benzoate leads to a large and stable S_C^* temperature range in both the FLC and the FLCP and therefore provides a suitable basis for comparing FLCs with FLCPs. As the electro-optic response of the FLCP depends on \bar{M}_n , we made

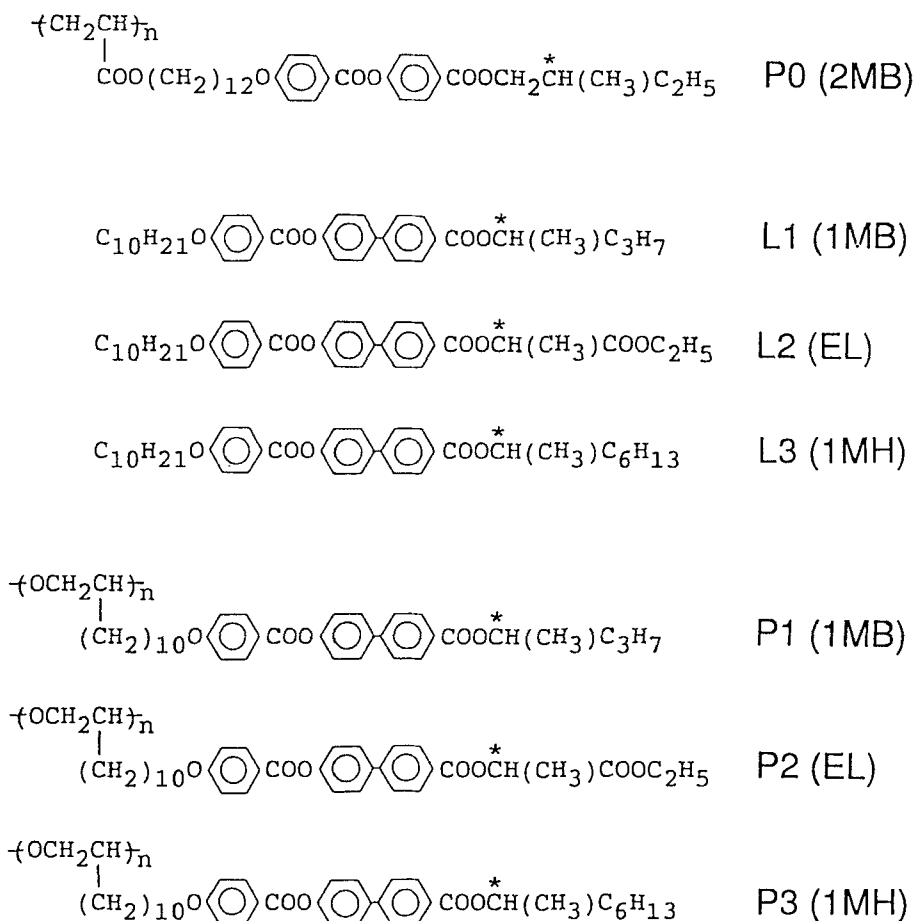


Figure 1. Molecular structures of the polyacrylate FLCs (**P0**), FLCs (**L1**, **L2** and **L3**) and polyoxyethylene FLCs (**P1**, **P2** and **P3**).

sure that **P1**, **P2** and **P3** had roughly the same \bar{M}_n , about 5000 (**P1**: $\bar{M}_n=4600$, **P2**: $\bar{M}_n=4700$, **P3**: $\bar{M}_n=4900$).

The phase transition temperatures of the above compounds are listed in the table.

2.2. Measurement

In order to investigate the electro-optic characteristics of the aforementioned liquid crystal materials, we measured the switching times τ , the apparent cone angles 2θ , where θ is the apparent tilt angle, and the spontaneous polarizations P_s .

The switching time τ is defined as the time for the transmitted light intensity to change from 10 to 90 per cent when the applied D.C. electric field polarity is reversed. A sample of liquid crystal was laminated between two ITO-coated glass plates. The cell thickness was controlled to be about $1.5\ \mu\text{m}$ using glass beads. We obtained molecular alignment by using shearing treatment. The cell was set on a hot stage (METTLER FP 80 and FP 82), to control the cell temperature T , mounted in a

Phase transition temperatures in °C of materials studied, as determined, on cooling, by polarized light microscopy.

		g	S _C *	S _A	I			
P0	($\bar{M}_n = 3100$)	●	-5	●	10	●	52	●
	(4800)	●	-1	●	14	●	61	●
	(5900)	●	9	●	29	●	90	●
	(19 000)	●	16	●	42	●	107	●
P1		●	30	●	130	●	148	●
P2		●	23	●	132	●	152	●
P3		●	22	●	122			●

		C	S ₁	S _C *	S _A	N*	I	
L1	●	52	●		105	●	130	●
L2	●	42	●		100	●	130	●
L3	●	40	●	46	101	●	118	●

g, Glass-like phase below which electro-optic response was not observed. S₁, Unidentified higher order smectic phase.

polarizing microscope (NIKON Optiphot-pol). The transmitted light intensity was detected by a photo-diode attached to the microscope, and the signal was recorded by a digital storage oscilloscope (IWATSU DS-8623). The applied D.C. field intensity E was $\pm 10 \text{ MV m}^{-1}$ except when the dependence of τ on E was required; then we varied E from 2.5 to 25 MV m^{-1} .

In order to determine the apparent cone angle 2θ , we used the same cell and apparatus used for the measurement of τ . We first applied the D.C. field of 10 MV m^{-1} and determined the position of the rotating table of the microscope where we got a certain intensity (~ 50 per cent) of the transmitted light. Then we reversed the field polarity, turned the table and determined the position at which we measured the same intensity. The angle between the two positions corresponds to 2θ .

We measured the spontaneous polarization P_s by using the triangular wave method [41]. The triangular wave generated by a signal generator (NF 1930) was amplified by a power amplifier (NF 4005), then applied to the cell. The cell thickness was $7 \mu\text{m}$ and the applied field peak to peak intensity was 20 MV m^{-1} . The current signal was received by a current amplifier (KEITHLEY 428), recorded by the digital storage oscilloscope and analysed by a computer (HP 9000/300). The frequency of the triangular wave was 50–100 Hz for FLCs and 0.02–2 Hz for FLCs. Because of the slow response of the FLC, the current signal of polarization reversal in the FLC becomes much smaller than that in a FLC, and the signal to noise ratio tends to be worse due to impure ion current. In order to avoid the influence of ion current we made the cell thickness $7 \mu\text{m}$.

In addition we evaluated the rotational viscosity η using the relation [42]

$$\eta = P_s \cdot E \cdot \tau. \quad (1)$$

3. Results and discussion

3.1. Electro-optic switching times and apparent cone angles of the FLCs

Figure 2 shows the temperature dependence of τ for FLC **P0**. For $3100 \leq \bar{M}_n \leq 5900$, an electro-optic switching was detected over a range of about 30°C , where the liquid crystal material was in the S_C* phase. As \bar{M}_n increases, the curve of τ seems

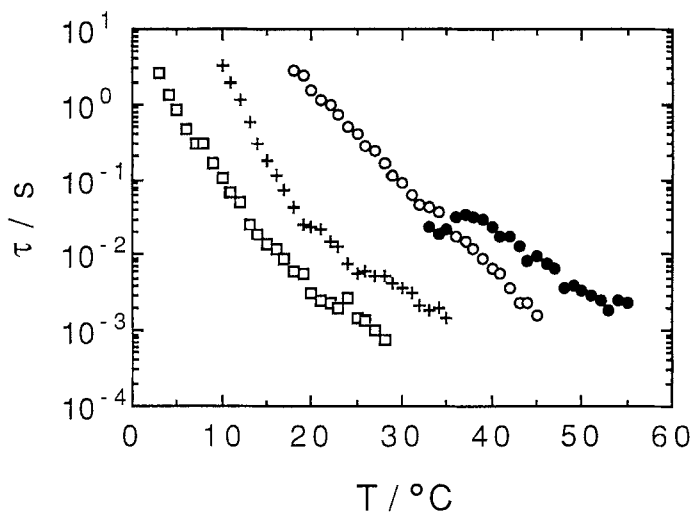


Figure 2. Dependence of τ on T in the case of FLCP **P0** of \bar{M}_n 3100 (\square), 4800 (+), 5900 (\circ) and 19000 (\bullet).

to shift parallel to the temperature axis T . At a given T , τ became larger with \bar{M}_n . The switching time was a few milliseconds at higher T values. As T became lower, τ increased rapidly and ultimately exceeded a few seconds. At low T , where τ was more than a few seconds, the texture of the liquid crystal was 'frozen' in a glassy state and we could no longer detect electro-optic switching. In the case of $\bar{M}_n = 19000$, the behaviour was different. The temperature range wherein switching was detected was 20°C and smaller than in the cases of $\bar{M}_n \leq 5900$. In addition, before τ reached the order of seconds, the texture was 'frozen' and the polymer gave no response. The molecular weight was so high that the polymer could not remain in the S_C^* phase at low T .

Figure 3 shows the field intensity dependence of τ in the case of **P0** ($\bar{M}_n = 5900$). In the case of $T \leq 30^\circ\text{C}$, τ was mostly proportional to E^{-1} , which showed that the electro-optic switching was ferroelectric.

Figure 4 shows the temperature dependence of 2θ in the case of **P0**. For $\bar{M}_n = 5900$, 2θ reached a maximum of 35° at lower T . In the case of \bar{M}_n greater or less than 5900, the maxima of 2θ were smaller than that for $\bar{M}_n = 5900$, which shows that there exists a particular \bar{M}_n for achieving a stable S_C^* phase in the FLCP. In all cases, 2θ sloped down to the region of higher T . The temperature range of this gentle slope was 20°C or more; here S_C^* and S_A phases coexisted. The existence of the S_A phase is also supported by figure 3. In the case of $T \geq 35^\circ\text{C}$, the dependence of τ on E deviated from the relation $\tau \propto E^{-1}$, and the tilt must be attributed to an electroclinic (EC) effect. It is remarkable that the range of the EC effect is so large in FLCP. We suppose that the molecular weight distribution made a transition temperature from S_A to S_C^* imprecise, and hence the coexistence region became broad. In the case of $\bar{M}_n = 19000$, the maximal 2θ was 4° , which was much smaller than for the others. A large \bar{M}_n apparently prevented the side chains from tilting sufficiently.

3.2. Comparison of FLCPs with FLCs

Hereafter, until the end of this section, we will deal with the polyoxyethylene FLCPs. Figure 5 shows the temperature dependence of P_s , (a) in the case of the

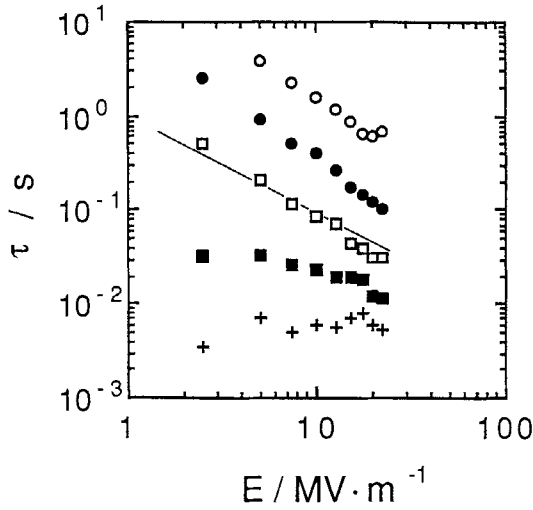


Figure 3. Dependence of τ on E in the case of FLC P0 ($\bar{M}_n = 5900$) at the temperatures 20°C (○), 25°C (●), 30°C (□), 35°C (■) and 40°C (+). For the temperature 30°C a line representing $\tau \propto E^{-1}$ is given.

FLCs and (b) for the FLCs. The value of P_s in the FLCP was mostly at the same level as that in the corresponding FLC, which shows that P_s is not varied through polymerization. In the cases of both FLCs and FLCPs, the values of P_s were arranged in the same order, according to the differences in chiral end group, i.e. $1\text{MB} < \text{EL} < 1\text{MH}$. Because P_s is thought to represent the order of molecular arrangement in a ferroelectric phase, the arrangement of side chains must be similar to that of FLC molecules. Therefore, it seems that the molecular packing, in other words, the distance between adjacent molecules can be the same for FLCP and FLC.

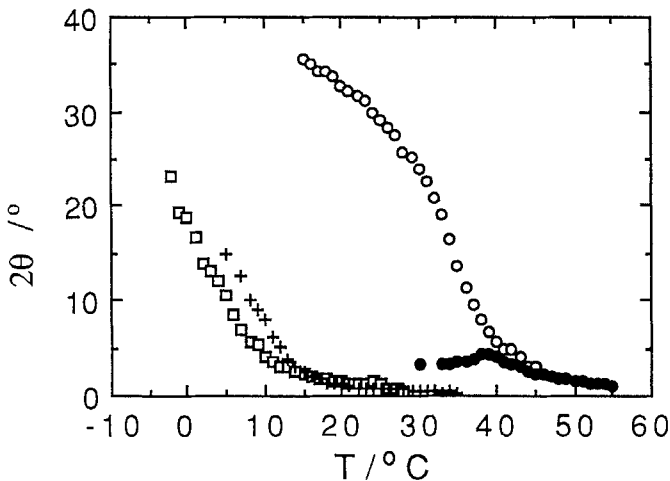


Figure 4. Dependence of 2θ on T in the case of FLC P0 of \bar{M}_n 3100 (□), 4800 (+), 5900 (○) and 19000 (●).

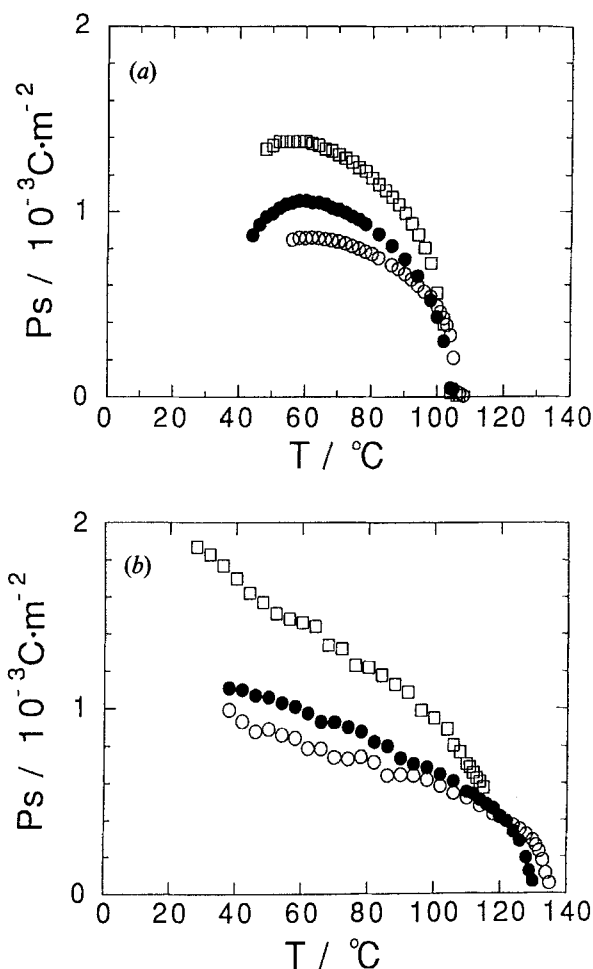


Figure 5. Dependence of P_s on T ; (a) FLCs L1 (1MB) (\circ), L2 (EL) (\bullet) and L3 (1MH) (\square), and (b) FLCPs P1 (1MB) (\circ), P2 (EL) (\bullet) and P3 (1MH) (\square).

As both FLC and FLCP exhibit the same S_C^* phase, we can naturally expect that they are similar in molecular arrangement except for the main chain in the FLCP which may lie somewhere in the plane between each smectic layer.

Figure 6 shows the temperature dependence of 2θ ; (a) is for FLCs and (b) is for FLCPs. Similarly to the case of P_s , 2θ in the FLCP had a similar value to that in the corresponding FLC, which supports the previous assumption that the FLC and the FLCP are similar in molecular arrangement.

From figures 5 and 6, we find that the temperature range of the S_C^* phase in the FLCP is much larger than that in the corresponding FLC. Polymerization is effective in stabilizing the S_C^* phase of a FLC.

Figure 7 shows the temperature dependence of τ . In the case of both FLCs and FLCPs, the values of τ were arranged in the same order according to the differences in chiral end group, i.e. $1\text{MH} < \text{EL} < 1\text{MB}$, the reverse of the order for P_s . The value of τ for the FLCP was, however, 10^2 – 10^3 times larger than that for the corresponding FLC.

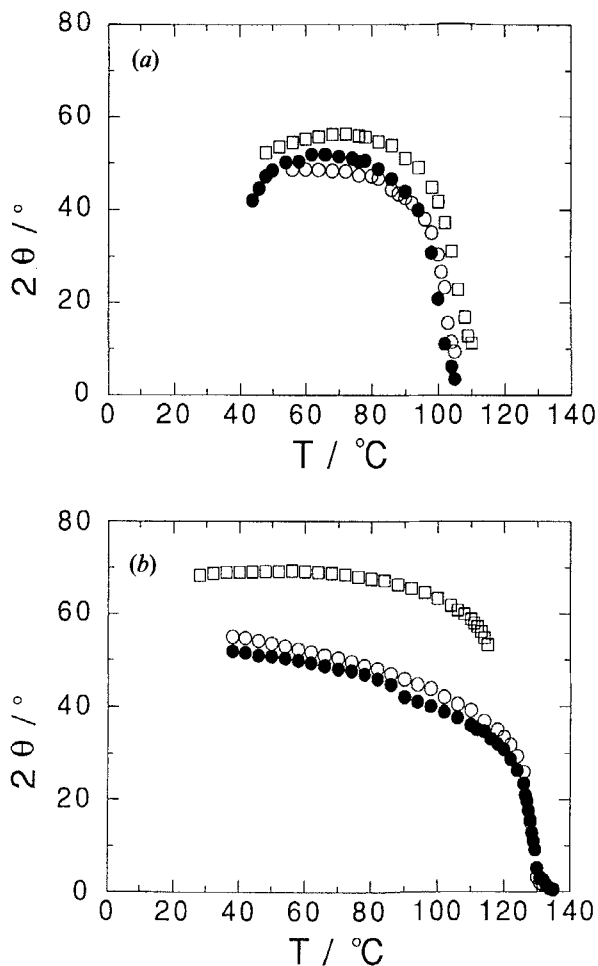
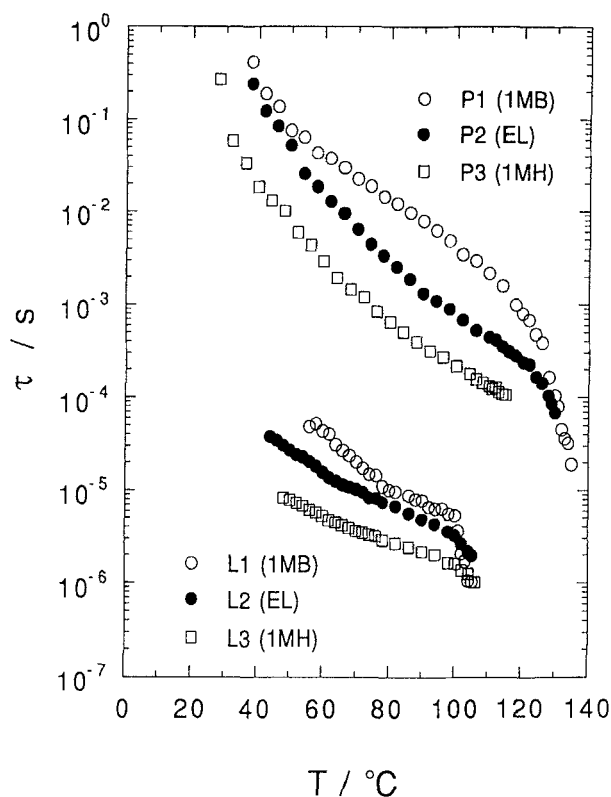


Figure 6. Dependence of 2θ on T ; (a) FLCs L1 (1MB) (○), L2 (EL) (●) and L3 (1MH) (□), and (b) FLCPs P1 (1MB) (○), P2 (EL) (●) and P3 (1MH) (□).

In the case of both FLCs and FLCPs, we observed that τ rapidly decreased at higher T ; for example, τ of P1 rapidly decreased for $T > 125$ °C as shown in figure 7. In this region, the EC effect was dominant in the electro-optic switching.

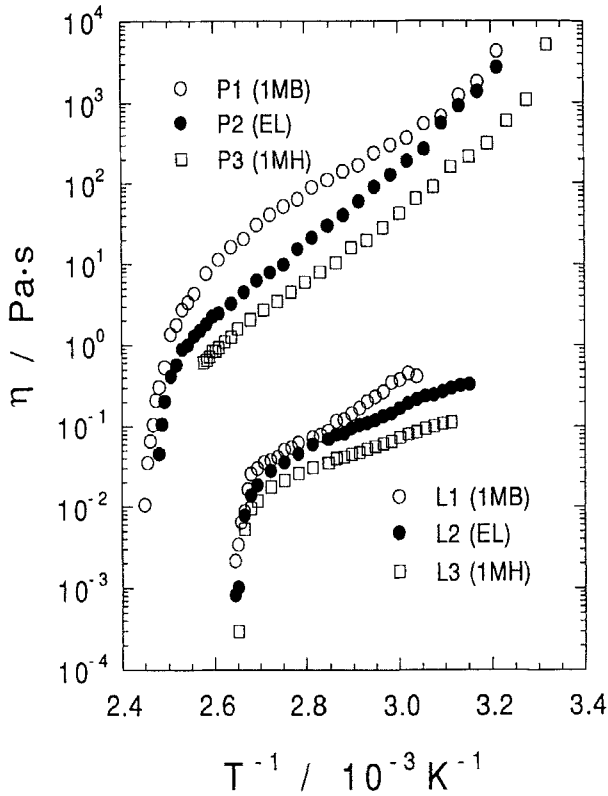
We evaluated η from the measured P_s and τ , according to equation (1). Arrhenius' plots of η are shown in figure 8. In the case of both FLCs and FLCPs, the values of η are arranged in the same order, according to the differences in chiral end group, i.e. $1MH < EL < 1MB$. The value for a FLCP was, however, 10^2 – 10^3 times larger than that for the corresponding FLC, reflecting the slow response of FLCPs. In addition, the temperature dependence of η in the FLCP is much larger than that in the FLC.

Here we discuss the difference of molecular motion between FLCs and FLCPs. A molecule of a FLC is thought to gyrate around the surface of double cones as illustrated in figure 9(a). It is remarked that the centre of gravity of the FLC molecule need not move during switching.

Figure 7. Dependence of τ on T .

Supposing that decoupling due to the spacer in a FLCP is enough to allow the side chains to move freely around the surface of double cones. As the rotational viscosity in a FLCP is much larger than that in a FLC, the large rotational viscosity in the FLCP must be attributable mainly to the viscosity due to spacer coupling. Compared with the huge viscosity due to the spacer, viscosity due to the structure of the mesogenic moiety must be negligible. As a result, polymers **P1–P3** must have roughly the same rotational viscosity. However, as experimental results show, η in FLCPs strongly depends on the structure of the mesogenic unit, especially on the chiral end group. In addition, a CPK model shows that the length of main chain between adjacent side chains or the length of a C_{10} spacer seems not to be enough for side chains to move freely along the surface of double cones. Therefore we need a new model in which η for the FLCP reflects the structure of the mesogenic moiety.

In the case of the FLCP, the opposite end to the chiral end group is pinned onto the main chain by the spacer group. If the pinning effect is strong enough, the side chain must be forced to gyrate around the surface of a single cone as illustrated in figure 9(b). Here the centre of gravity must move. As a result, one side chain exchanges its position for the next one. Requiring that dipole moments should gyrate along with the gyration of P_s , some side chains must gyrate along the upper half surface of the cone, others along the lower half as illustrated in figure 10. Side chains may sometimes come into collision with each other. These collisions cause

Figure 8. Arrhenius' plots of η .

large frictional effects, which are the origin of the large rotational viscosity of FLCs.

According to this model, a slow response seems to be an essential feature of comb-like FLCs. We can indeed obtain faster response FLCs by polymerizing faster response FLCs, but the improvement is limited. In order to improve the switching time of FLCs drastically, some new molecular design to modify the switching mechanism is indispensable.

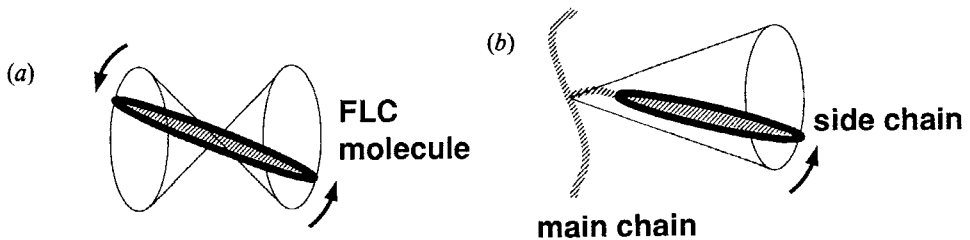


Figure 9. Illustrations of molecular motion. (a) In the case of FLC, (b) in the case of FLCP.

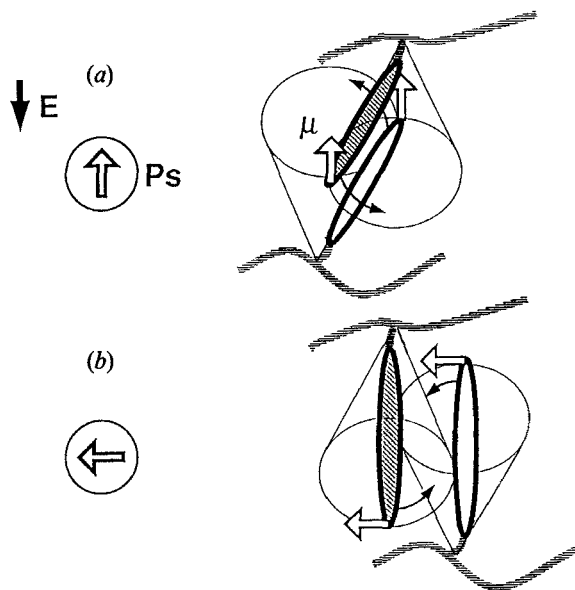


Figure 10. Illustrations of side chain motion in the case of a FLCP, where μ is a dipole moment which is considered to have its origin in a carbonyl group close to the chiral end group and generates P_s . Collisions between side chains take place. (a) At the beginning of the switching process, one side chain overlays the other. One goes upward and the other downward, then they collide with each other. (b) At the middle point of gyration, one side chain is on the top of the cone, and the other is on the bottom. They are arranged in parallel with each other and then collide head-on.

Here, we have to emphasize that the above model oversimplifies the role of the spacer and is still a fairly speculative proposal. The model must be substantiated for higher molecular weight samples and with different spacer lengths.

4. Application of FLCs to display devices

Combining a FLC with ITO-coated plastic substrates, we can easily produce large area display devices. First we coat the FLC on an ITO-coated plastic substrate and laminate it with the other ITO-coated substrate. As we have only to coat and laminate, we can easily make a large area cell. Then we obtain molecular alignment by using a bending procedure. When we bend the plastic substrates, the FLC molecules laminated between two substrates suffer a uniform shear stress and align uniformly. Thus we can easily obtain good molecular alignment without using any aligning layer. It is remarkable that using this method we can also align a FLC which exhibits no nematic phase in its phase transition sequence. In addition, as the FLC has a high viscosity, it can maintain a particular cell thickness without any spacer such as glass beads. Compared with a glass-based process, our procedure has an advantage of easy production. We expect that the process must reduce cost and become suitable for continuous mass production.

Using the process mentioned above, we succeeded in making a large area (15 cm \times 40 cm) static driven display [21]. The display was flexible and lightweight. In addition, the molecular alignment resists considerable mechanical shock. Recently, we have also succeeded in making a dynamically driven, simple, matrix display of



Figure 11. Photograph of a dynamically driven, simple, matrix display (15 cm \times 40 cm, 100 \times 300 pixels).

the same size (see figure 11). However, the switching time remains at the millisecond level. Much improvement in switching time is needed for any application to high definition displays.

5. Conclusion

We have synthesized some polyacrylate FLCs of the same molecular structure, differing only in \bar{M}_n . They exhibit electro-optic switching times ranging from a few milliseconds to a few seconds in the S_C^* phase. As \bar{M}_n increases, the S_C^* temperature range shifts to higher values and the switching time curve also shifts parallel, along the temperature axis. At a given temperature, switching time increases with \bar{M}_n . The apparent cone angle exhibits a maximum at a particular \bar{M}_n ($= 5900$), which shows that there exists a particular \bar{M}_n to obtain a stable S_C^* phase in a FLC.

In addition, we have compared some polyoxyethylene FLCs with the corresponding FLCs. There is no significant difference in spontaneous polarization and cone angle, which shows that the side chains are arranged in the same way as the FLC molecules. On the other hand, the switching time and rotational viscosity in the FLC are 10^2 – 10^3 times larger than those in the FLC. Rotational viscosity in the FLC is mainly dependent on side chain structure rather than on the spacer. Therefore collision between side chains, rather than hindrance by the spacer group of side chain gyration, causes the large rotational viscosity of a FLC.

Combining a FLC with ITO-coated plastic substrates, we succeeded in making a large area, dynamically driven, dot matrix display. Though much improvement in switching time is still needed, a flexible and lightweight display like a sheet of paper will be available in the near future. It will bring us a new life style in information-oriented society.

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