Liquid Crystals
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713926090

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Online publication date: 06 August 2010

To cite this Article Minato, T. and Suzuki, K.(1997) 'Orientation of temperature gradient-cooled ferroelectric and antiferroelectric liquid crystals in restricted rectilinear space', Liquid Crystals, 23: 1, 43 — 50
To link to this Article: DOI: 10.1080/026782997208659
URL: http://dx.doi.org/10.1080/026782997208659

LIQUID CRYSTALS, 1997, VOL. 23, NO. 1, 43–50

Orientation of temperature gradient-cooled ferroelectric and antiferroelectric liquid crystals in restricted rectilinear space

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(*Received 23 December 1996; accepted 1 February 1997*)

The authors have developed a method for producing a large-size liquid crystal cell to which top and bottom substrate stripe barriers (viewed from above, barriers appear as a series of stripes) adhere completely. Ferroelectric or antiferroelectric liquid crystals were confined to a long narrow rectilinear space 300 μm in width and cooled from one end to the other under a temperature gradient. Smectic layers were found to bend in the direction of molecular movement due to volume contraction of bulk liquid crystal, with decrease in temperature. No zigzag or line-like defects were found with antiparallel rubbing or in C2 orientation with parallel rubbing. Thus, regarding the orientation of the smectic layers, constituent molecules must be moved only in one direction perpendicular to smectic layers.

1. Introduction

Ferroelectric liquid crystals (FLCs) [1] show ultra fast response times compared to conventional nematic liquid crystals. They also possess bistability and these important features make them useful in surface stabilized liquid crystal display devices without active elements such as thin film transistors [2]. Antiferroelectric liquid crystals (AFLCs) should have similar application [3]. AFLCs are superior to FLCs since they exhibit grey levels and double hysteresis curves with tristable switching [4]. The liquid crystal phases that respond to an external electric field are the chiral smectic C (SmC*) phase in FLCs and the chiral smectic C_A (SmC_A^*) in AFLCs. Both have the same smectic layer structure. In display devices, the layer structure must be held by a pair of glass substrates. The cell filled with the liquid crystal is gradually cooled from the higher temperature liquid phase so that the smectic layer structure is produced without defects. With cooling, new orientation defects characteristic of the smectic layer structure develop. These defects are 'zigzag loop' in shape and vary in number and size [5, 6]. They make clarification of the electro-optical properties difficult and lessen the picture quality of display devices and thus should be eliminated.

Another problem is that the layer structure is not capable of sufficient self-recovery, in contrast to nematic liquid crystals. For example, when liquid crystals flow locally because of changes in the cell gap, the layer structure cannot resume its original organization. The layer structure is destroyed by a very weak external impact. These are the reasons why display device production and appearance on the market, using FLCs and AFLCs, were late in coming about. Thus, a method for producing a rigid cell with sufficient ability to withstand external impacts is desirable.

The inner structure of a liquid crystal cell which permits the bending of smectic layers in only one of two possible directions was developed in the present study. Such a structure should make possible sufficient anti-shock ability and impact resistivity.

2. Background

A FLC or AFLC is sandwiched between a pair of glass substrates separated by 1·4∼1·8 μm. The inner sides of the substrates are coated with a rubbed thin polyimide layer. A suitable liquid crystal and polyimide layer comprise a fairly oriented smectic layer structure, for which there is only one layer normal direction throughout the layers. Rieker and Clark [7] showed smectic layers not to have the bookshelf structure but the so-called 'chevron' structure in which layer bending occurs in the layer normal direction at the middle of the cell gap, as shown in figure 1. There are two opposite bend directions nearly parallel to the layer normal.

The direction of bend is relative to the rubbing direction. In parallel rubbing, the rubbing direction for both glass substrates is the same, as shown in figure 2 (*a*). There are two possible orientations, C1 and C2 [8]. In the C2 orientation, the bend direction is the same as the rubbing direction. In the C1 orientation, these directions are opposite. These two orientations differ from each

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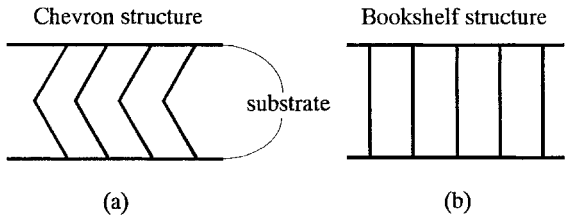


Figure 1. Layer structure of the smectic phase: (*a*) chevron; (*b*) bookshelf.

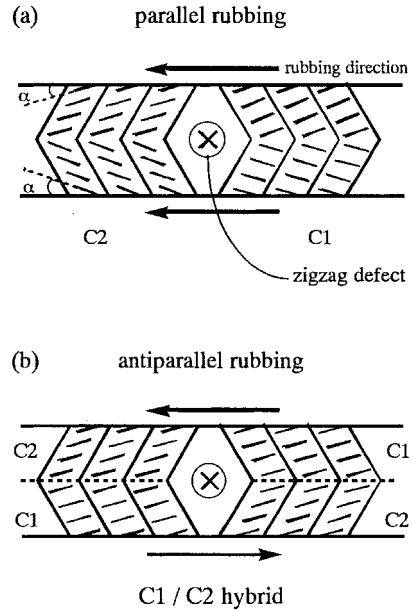


Figure 2. Relation between rubbing direction and bend direction: (*a*) parallel rubbing, (C2) rubbing direction same as that of a layer bend, (C1) rubbing direction opposite that of a layer bend; (*b*) antiparallel rubbing, orientation of the upper half different from that of the lower half. (→): rubbing direction, ⊗: zigzag defect.

other under the condition that the constituent molecules are aligned at a finite angle α to the substrates. Angle α is the pre-tilt angle determined mainly by chemical interactions between the polyimide layer and the liquid crystal molecules. In the case of antiparallel rubbing, as shown in figure 2(*b*), the rubbing directions of the top and bottom substrates are opposite. The two orientations with opposite bend directions cannot be distinguished, as apparent from figure 2(*b*). Thus, in antiparallel rubbing, maintaining only one bend direction is quite difficult because there is an equal probability for each orientation with the opposite bend direction.

Zigzag defects occur along the boundary of two macroscopic domains, as in the case of C1 and C2, having opposite bend directions [5]. If the bend directions are the same throughout the smectic layers, there are no such defects. There is also a defect, line-like in shape [6]. The alignment control means that the smectic layers are bent arbitrarily in one of two possible directions, irrespective of the polyimide resin. Rubbing a polyimide layer appears at present to be the only means for alignment control of FLCs and AFLCs. Rubbing may not necessarily determine, in all cases, the direction of layer bend.

Single bend direction occurs if the liquid crystal molecules in the middle of the substrates move slightly in only one direction along the layer normal and the other molecules, near the substrates, are rendered almost immobile. Such molecular movement may be induced through control of the direction of volume contraction that occurs with a decrease in temperature.

An attempt was thus made to confine the liquid crystals in a closed, long, rectilinear space or 'tunnel'. This tunnel is then cooled under a temperature gradient from one end to the other. The direction of movement is made parallel to the tunnel. In SmA or SmC* phases, constituent molecules may move toward the region that cools first owing to volume contraction. The direction of the temperature gradient along the tunnel uniquely determines the direction of molecular movement. This makes possible a comparison of the two orientations in the one cell.

The temperature dependence of the density of the AFLC 4-(1-methylhepthyloxycarbonyl)-3-fluorophenyl 4′-dodecylbiphenyl-4-carboxylate(MHFPDBC), was evaluated by Uehara *et al.* [9], who found the linear dimension to shrink by 0·5 per cent and 1·5 per cent for a temperature decrease of 10°C and 20°C, respectively, from the SmA–isotropic transition temperature. Volume contraction of liquid crystals is generally greater than that of the tunnel containing liquid crystals. However, if the top and bottom substrates do not adhere completely, that is, if the rectilinear space is not completely closed except at the inlet and outlet, constituent molecules may not move together in the direction normal to the smectic layers. Whether molecular movement becomes sufficient to induce a layer bend is not clear. This may possibly depend on the cross-section and length of the rectilinear space, expansion coefficient of the glass substrate, liquid crystals and the temperature range of the SmA phase.

The orientation of the AFLC is not as good as that of a FLC since the former does not have the long pitch cholesteric phase which promotes formation of the well-oriented SmA phase. Typical zigzag defects are in general buried in other miscellaneous defects. The correct choice of polyimide material, or one-sided or cross rubbing, should make possible completion of the smectic layer structure, with consequent elimination of miscellaneous defects and actualization of zigzag defects. Layer structure completion and zigzag defect elimination should be achieved at the same time. Molecular movement in

rectilinear space may play an important role in both respects.

3. Experimental

By photolithography, a closed rectilinear space between the glass substrates can be created rather easily as follows [10]. Soda-lime and alkali-free glasses (7059, Corning) were prepared; the latter mainly for examination of AFLCs. The polyimide solution was spin coated on the clean-washed glass substrates and dried at 200°C. The polyimide layer was rubbed several times in the same direction. As typical polyimide resins, HL1110 and SE610 from Hitachi Kasei Kogyo and Nissan Kagaku Kogyo, respectively, were used. The photoresist solution (MP1400, posi-type, Shipley), was spin-coated over the polyimide layer and dried at 90°C. The photoresist was exposed to UV light through a mask having a stripe pattern with an appropriate pitch and developed with alkaline developer (MF312, Shipley). Post-baking was conducted again at 140°C for 1 h. In this manner, stripe barriers parallel to the rubbing direction could be attached to the substrates.

The two substrates with or without barriers were made to adhere as follows. Following alignment correction, air in the tunnels was removed, causing the barriers to come into close contact with the corresponding polyimide layer. Such a situation was placed in an air oven and the temperature maintained at 150°C for 1 h, followed by gradual cooling. The substrates completely adhered to the stripe barriers and the single space between the substrates was partitioned into many long narrow tunnels as shown in figure 3. The photoresist did not function as an adhesive agent at room temperature. At 160°C, a specially selected photoresist supported adhesion. A semi-flexible material, such as photoresist, is preferable to a hard material which, in some cases, becomes separated from the substrates due to an expansion coefficient mismatch under a temperature gradient. The barrier width, height, pitch and length were set at 30 μm, 1·7 μm, 330 μm and 28 cm, respectively. There were 640 tunnels partitioned by stripe barriers between the substrates. The tunnel cross-section, quadrilateral in shape, was 300 μm in width and 1·6 μm in height.

Liquid crystals were introduced into the cell by a suitable method, followed by immersing the cell in hot water or an air bath with the temperature controlled to within ±0·1°C and usually set 5°C above the isotropic–chiral nematic transition temperature of the FLC, and the isotropic–SmA transition temperature of the AFLC. The surface of the water was covered with a thick insulation layer having a slit through which the cell could be moved up and down. This insulation thus prevented water vapour from adhering to the surfaces of the cell and calmed the waves on the water surface due to stirring. The immersed cell was removed from the water into the air at 2 mm min^{-1}, while maintaining a 90° angle between the surface of water and the tunnel. Air was applied to both surfaces of the cell by ventilators. A specific temperature gradient, determined by the difference in temperature between the water bath and the air, was applied parallel to the tunnels. For liquid crystals with a clearing point above 90°C, an air bath was used. The FLCs used are listed in table 1. As a typical AFLC, CS4000 (from Chisso Corp. [11]), and the special AFND-1 mixture (supplied by Denso Corp.), were used to observe the orientation.

4. Results

The orientation of the smectic layers obtained by temperature-gradient cooling was compared with that for uniform cooling in air or the water bath. The results for the FLCs, in the case of antiparallel rubbing, are discussed in the following. With cooling, there were no zigzag or line-like defects in any tunnel of the standard width 300 μm. A completely defect-free C1/C2 hybrid orientation was attained regardless of the liquid crystals used or the direction of molecular movement. The direction of molecular movement was the same as the

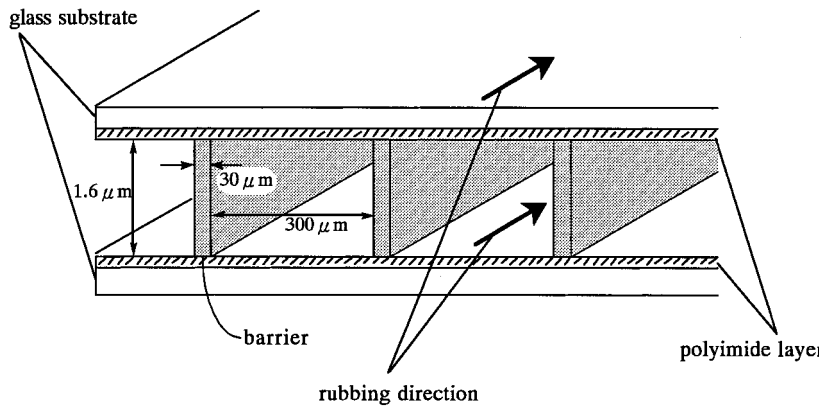


Figure 3. Oblique cross-sectional view of inner structure of the cell showing the top and bottom substrates to which the photoresist barriers completely adhere.

Table 1. Liquid crystals used.

	Transition temperature/°C							
FLC	SmC*		SmA		N*		I	
CS1013[a]	●	63	●	70	●	80	●	
CS1014	●	54	●	69	●	81	●	
ZLI3774[b]	●	62	●	76	●	80	●	
SCE8[c]	●	59	●	79	●	100	●	
SCE9	●	61	●	91	●	115	●	
Felix4851[d]	●	63	●	69	●	73	●	
K24[c]			●	34	●	41	●	
AFLC	SmC_A^*		SmC*		SmA		I	
CS4000[a]	●	83	●	84	●	101	●	
AFND-1[e]	●	94		—	●	114	●	

[a] Chisso Corp.
[b] Merck.
[c] BDH.
[d] Hoechst.
[e] Denso Corp.

direction of motion of the cell. The smectic layers were found to be bent in the same direction as the molecular movement. The determination of bend direction is generally difficult by microscopic observation of the texture of C1/C2 hybrid orientation. The bend direction was determined from the shape of small tear-like zigzag defects produced by pin-holes in the polyimide layer or contamination in the tunnels. For tunnel widths greater than 600 μm, zigzag defects occurred in nearly all cases. With uniform cooling in air or the water bath, in contrast to temperature-gradient cooling, only zigzag defects were detected in all the liquid crystals, except Felix-4851 which showed zigzag or line-like defects.

In parallel rubbing, the orientation of the smectic layers obtained by temperature-gradient cooling differed depending on the direction of molecular movement. When the direction of motion of the cell was the same as the rubbing direction, defect-free C2 orientation was possible for all the FLCs, except ZLI3774 which showed zigzag and line-like defects. When the tunnel width exceeded 600 ~ 800 μm, C2 orientation was accompanied by line-like defects. The critical width appeared dependent on the particular liquid crystal/polyimide resin combination. When the direction of motion of the cell was opposite to the rubbing direction, three different orientations were noted: a defect-free C1 orientation for SCE8, SCE9, and ZLI3774, a C2 orientation with line-like defects, and an ordinary orientation with C1 and C2 domains exhibiting line-like defects. For uniform cooling in the air oven, orientation was essentially the same as the third orientation. A defect-free orientation was never attained. It should be noted that the C1 orientation is not accompanied by line-like defects. In figure 4, the orientation for opposite directions of molecular movement is compared and was found not to be influenced by the polyimide resin. The results are summarized in table 2.

Table 2. Results of orientation (parallel rubbing).

	Direction of molecular movement		
FLC	C2	C1	ΔSmA*[a]
CS1013	○	×	7
CS1014	○	×	15
ZLI3774	×	○	14
SCE8	○	○	20
SCE9	○	○	30
Felix4851	○	×	6

○: Completely defect-free orientation; ×: defects present;
[a] temperature range of chiral SmA phase.

For CS4000, one-sided rubbing gave a better orientation than the parallel or antiparallel rubbing in the previous study. Temperature-gradient cooling was thus conducted. The cell built with soda-lime glass substrates did not show an improved orientation. The number of zigzag or line-like defects remarkably decreased with the 7059 glass substrate at a tunnel width less than 150 μm, as shown in figure 5, irrespective of the direction of molecular movement. Two orientations using AFND-1 are shown in figure 6, with soda-lime glass substrates, a cell gap of 1·7 μm, antiparallel rubbing, and a tunnel width of 240 μm. Figures 6 (*a*) and (*b*) show the orientation with temperature-gradient cooling, and (*c*) and (*d*), the same orientation as that with uniform cooling in the

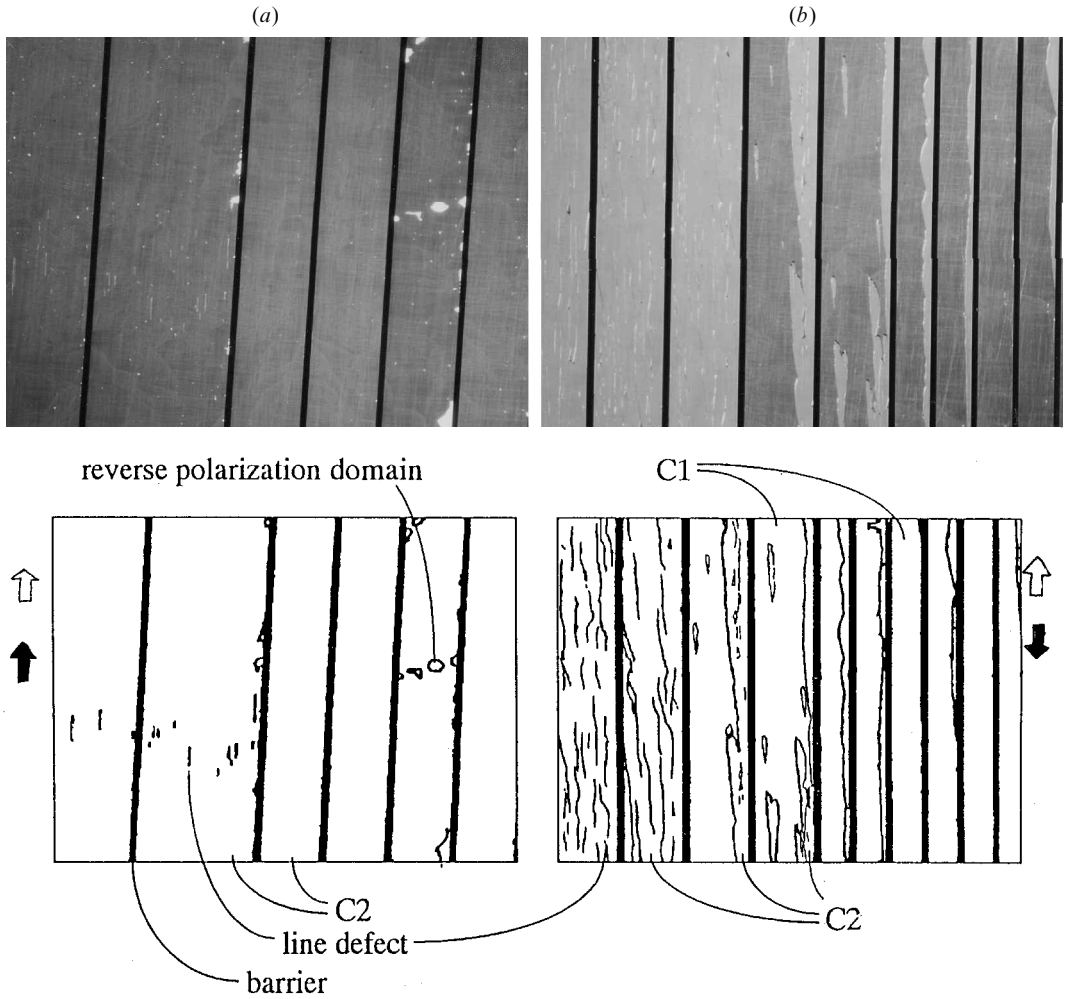


Figure 4. Comparison of the orientation in Felix-4851 with parallel rubbing as determined by the direction of molecular movement: (*a*) movement to C2 direction; (*b*) movement to C1 direction. Orientation is C2 in (*a*). The white spots in (*a*) are domains of reverse polarization. The orientation in (*b*) is very complicated and was observed for different tunnel widths for direct comparison of the width effect: tunnel width; 600, 300 and 150 μm from broad to narrow. : rubbing direction. : direction of temperature gradient.

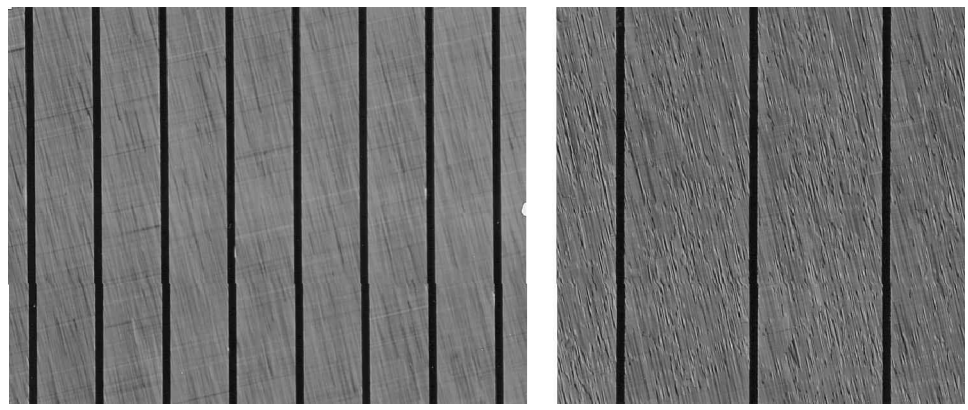
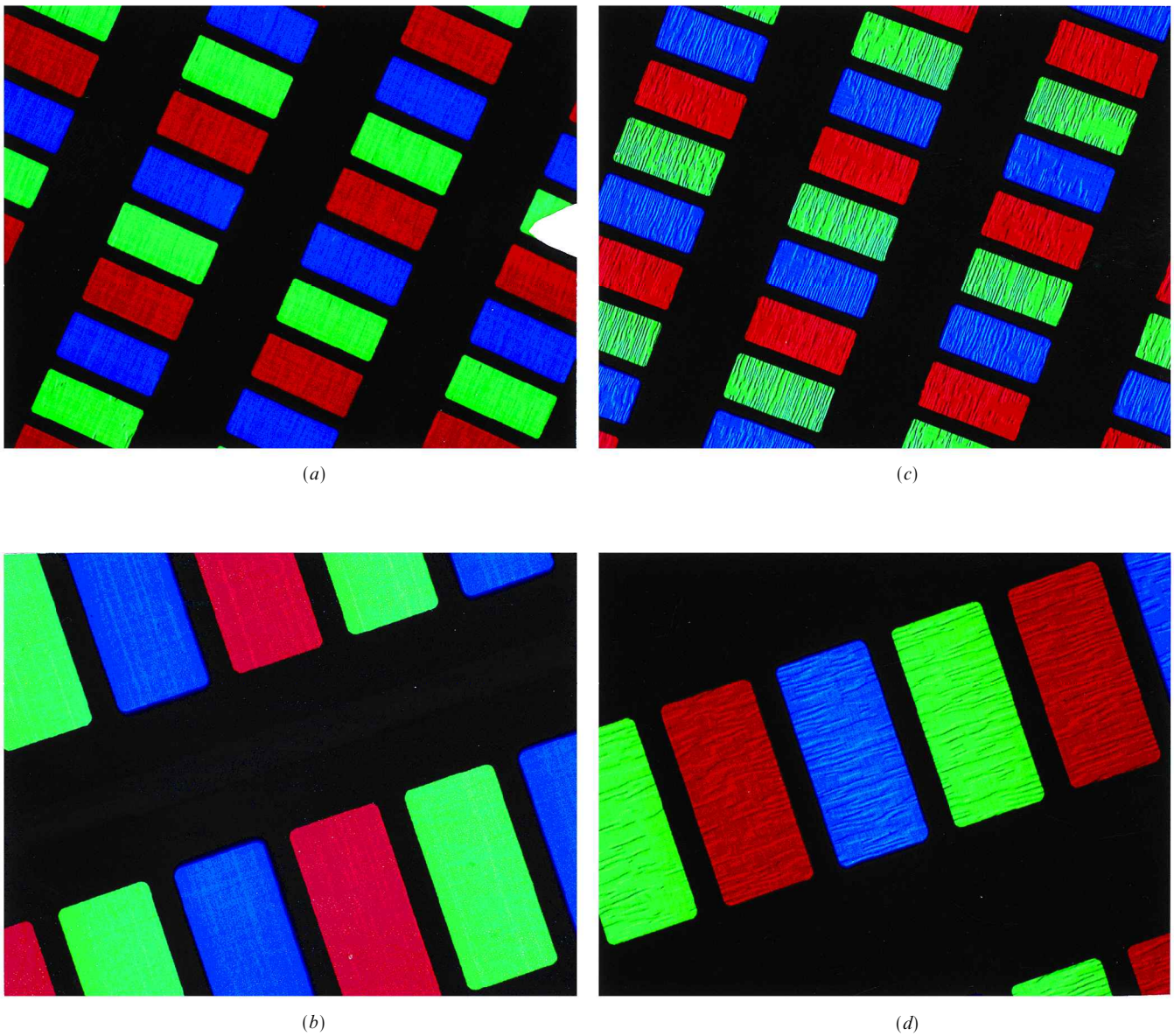


Figure 5. Orientation of the AFLC (CS4000) in the same cell for one-sided rubbing: the 7059 glass substrate and tunnel widths (*a*) 150 μm; (*b*) 300 μm. Effects of temperature-gradient cooling depend strongly on tunnel width.



(*a*) (*c*)

(*b*) (*d*)

Figure 6. Orientation of the AFLC (AFND-1) in the cell with colour filters: (*a*) and (*b*) temperature-gradient cooling; (*c*) and (*d*) uniform cooling in an air bath. Nearly all defects have disappeared by temperature-gradient cooling. Barriers run straight in the centre of the black matrix from bottom to top. Rubbing is antiparallel. Pixel size: 160 μm × 75 μm. Magnification (*a*), (*c*): × 90, (*b*), (*d*): × 180.

air bath. Temperature-gradient cooling eliminated many defects to give orientation comparable to that of FLCs.

When the glass substrate of the filled cell is pushed with a force greater than 30 N cm^{-2}, the SmC* phase showed zigzag or line-like defects. At less than 70 ~ 100 N cm^{-2}, many defects disappeared immediately after the release of the force on the glass substrate [12].

5. Discussion

When the direction of the temperature gradient is in the C2 direction, defect-free C2 orientation is obtained, whereas line-like defects occur in C2 orientation when the direction of the temperature gradient is in the C1 direction. Complex structural changes in SmA and SmC* phases would be the cause of this. Nearly all the FLCs, including those in table 1, initially take on C1 orientation at the SmA–SmC* transition point [8] and then C2 orientation. The real layer structural change is not SmA→C1 or SmA→C2 but SmA→C1→C2. The SmA phase assumes C1 orientation possibly because of the bend being similar to that of C1. The gradual increase in the tilt angle in the smectic layers below the SmA–C1

transition temperature causes C1 to be unstable, with a consequent reversal of the bend direction of the smectic layers. This reversal, C1→C2, is not sudden, occurring over several degrees. It is important whether this change is complete or incomplete. If incomplete, both domains co-exist and the boundary shows zigzag defects. If complete, the low temperature phase of C2 occurs. The degree of reversal may be determined by various factors, such as baking temperature of the polyimide layer, tunnel cross-section and length and temperature range of the SmA phase. But in any case, the C2 domain will have line-like defects, possibly due to collision between C2 domains within the C1 domain. The common boundaries produced by the joining of any two C2 domains remained in some cases. The formation of line-like defects in the C2 domain occurs via the transient C1 phase. There are no line-like defects in the C2 domain following removal of the cell in the C2 direction, since the route to C2 from the SmA phase bypasses the C1 phase. To obtain a defect-free SmC* phase, the smectic layers must be made to bend in only one direction by some external force. Structural change, such as reversal of bend direction, must be suppressed. Layer bending is allowed to occur only once.

In parallel rubbing, (*a*) C2 orientation should be induced directly from the SmA phase, not C1, and (*b*) the smectic layers should be bent and maintained in the C1 direction. If the stable C2 orientation is induced directly from the SmA phase, line-like and zigzag defects never occur. Case (*a*) is easily achieved by a temperature-gradient cooling of liquid crystals confined to the tunnel. SmA layers may undergo, not C1, but C2-like deformation in a temperature-gradient cooling. Under a temperature gradient, each phase co-exists as shown in figure 7 (*a*). Effective volume contraction occurs first in the SmA phase, at low temperature in the tunnel. Molecular movement occurs in response to this volume contraction. Since the liquid crystal molecules are drawn in the direction of volume contraction, they move toward the low temperature side, with the layer structure maintained, as shown in figure 7 (*b*); layer bend occurs in the direction toward the decrease in temperature. Opposite movement never occurs in the tunnel. It is difficult to observe (by microscope) bend of the SmA layers in the tunnel at room temperature. The orientation of the liquid crystal K24 (from Merck) was confirmed to depend on the direction of the temperature gradient [12]. K24 showed the SmA phase with a chevron structure in a thick cell at room temperature [13]. The C1 phase is not stable and changes easily to the C2 phase and thus fails to occur in some cases. C1 orientation showed no defects in SCE8 or SCE9 in which the temperature range of the SmA phase is relatively wide

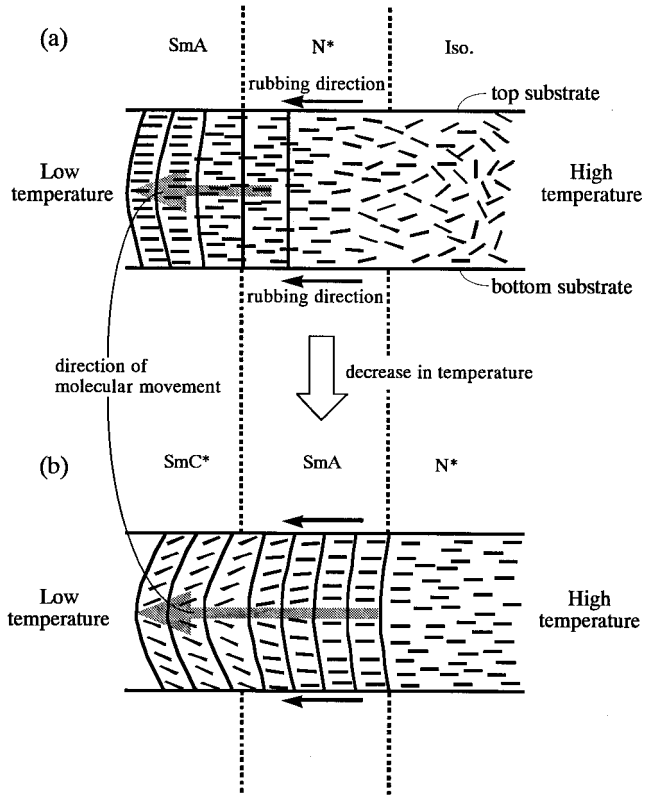


Figure 7. Movement of molecules confined to the tunnel under the temperature gradient: each phase initially existed as (*a*); with further decrease in temperature, each phase becomes (*b*). The arrow (←) signifies the direction of molecular movement due to volume contraction.

as shown in table 2. There may not necessarily be thermodynamic stability of C1 orientation obtained.

In antiparallel rubbing, the bend direction must be the same throughout the smectic layers. The present study demonstrates, for the first time, a defect-free C1/C2 hybrid orientation to be possible in a FLC with a greatly improved AFLC orientation. Though reversal of the bend direction may be possible for FLCs, as found in Felix-4851, this reversal would be easily suppressed by optimizing the tunnel cross-section, cooling conditions and the polyimide layer. Antiparallel rubbing may be useful for display devices owing to electro-optical properties between those of C1 and C2. In the matrix drive of AFLCs, the SmC_A^* phase in antiparallel rubbing is more stable than in parallel rubbing [14].

Temperature-gradient cooling has a very strong effect only on a tunnel whose width is less than the critical width for a height of 1·4∼1·8 μm. At greater widths, there is no effect. Thus, molecular movement along the tunnel depends inversely on width. Fortunately, this width is greater than the standard pitch of the transparent electrodes in display devices. A critical width is 600–800 μm for liquid crystals exhibiting good orientation.

Good orientation means that there are only zigzag and line-like defects. The initial AFLC orientation is not good owing to the absence of the cholesteric phase. The initial orientation can be improved by having liquid crystal molecules flow through the tunnel. For this purpose, glass substrates, with low expansion coefficients, should be used, and the width should be minimal. The expansion coefficient of the 7059 glass is about half that of soda-lime glass. This glass substrate with 150 μm width was used for CS4000 in this study, since its initial orientation is inferior to that of AFND-1.

The importance of width demonstrates that various defects can never be eliminated from a cell with an unpartitioned single space. In the past, even with parallel rubbing, zigzag defects could not be completely eliminated owing to a two-step layer structural change. Basic geometrical considerations [8] indicate that a high pre-tilt can stabilize only the C1 orientation. However, at least in the case of large-size display devices, C1 orientation may be disturbed by molecular movement due to volume contraction, with consequent zigzag defects. C2 and C1/C2 hybrid orientations are also required for display devices. Control of the pre-tilt angle would never permit such an orientation without defects.

In temperature-gradient cooling, no defects were found in the low temperature SmC* phase, such as C2. For ZLI3774, there were no defects in the C1 phase, indicating this to be the low temperature SmC* phase. For ZLI3774, SmA→C2→C1 may occur in contrast to the usual SmA→C1→C2. Such a material with a high pre-tilt angle may ensure a stable C1 orientation with very high contrast.

6. Conclusions

The polyimide layer is not able to completely regulate formation of smectic layers due to liquid crystal volume contraction with decrease in temperature. With the conventional liquid crystal cell, the direction of volume contraction perpendicular to the smectic layers is not regulated. To eliminate zigzag defects completely and improve the initial AFLC orientation, liquid crystal molecules must be moved smoothly in only one direction by volume contraction. The liquid crystal molecules must be confined to a narrow long rectilinear space and be allowed to cool under a temperature gradient to ensure sufficient molecular movement.

It is possible to make a liquid crystal cell to whose top and bottom glass substrates stripe barriers of photoresist adhere completely. When the rectilinear space width is less than 600–300 μm, the smectic layers can be bent arbitrarily in one of the two possible directions by the temperature-gradient cooling. C1/C2 hybrid and C2 orientations are possible without defects but defects may occur in C1 orientation.

In the present cell structure, problems due to fragility of the smectic layers have been eliminated and mass pumping phenomena [15] and vibration of substrates [16] induced by an a.c. voltage may be remarkably suppressed.

The authors express their appreciation to Dr Y. Yamada, Mr K. Maekawa, and Mr K. Miyashita, Research Laboratories of Denso Corp. for valuable comments on AFLCs and permission to use the photographs in figure 6. Thanks are also due to Dr K. Yoshida, Vice President of Technical Research Institute of Toppan Printing Co. Ltd., for continuous encouragement. The careful editing of the manuscript by Robert A. Hargis (Zama-shi, Kanagawa) is greatly appreciated.

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