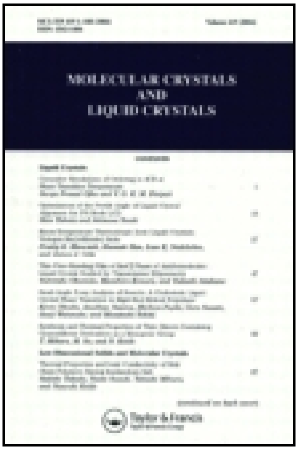

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Polyimide, Polyamide-Imide and Polyamide Alignment Layers for Antiferroelectric Liquid Crystal Display Cells and Their Structural Effect on Hysteresis Behavior

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Polyimide, Polyamide-Imide and Polyamide Alignment Layers for Antiferroelectric Liquid Crystal Display Cells and Their Structural Effect on Hysteresis Behavior[†]

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Polyimide, polyamide-imide and polyamide alignment layers for antiferroelectric liquid crystal cell devices were developed. Good quality alignment with improved electrooptical properties of the cell was observed by using structurally different fluorine and non-fluorine containing polymers as surface alignment layers. The double hysteresis loops characteristic of the antiferroelectricity arising from the chiral smectic phase were found to be different depending on the different molecular structure of polymer alignment layers. The switching between antiferroelectric (AF) and ferroelectric (F) states significantly depended on the molecular structure of the alignment layer and liquid crystals used. The liquid crystal blend (AFLCB-1) used in the experiment exhibited a wide temperature range of the smectic C_A^* phase. In the present paper, we report the broader hysteresis with higher driving margin for the non-fluoro polymer alignment layers as compared to those of the fluoro polymer aligning layers under identical conditions. The driving margin (M) varied from the 0.21 to 2.2 as the alignment layers were changed in the cells. All the alignment layers developed in our laboratory showed superior properties to that of the commercially available alignment layer material (LQ-1800).

Keywords: polyimide, polyamide-imide, polyamide, alignment layers, antiferroelectric, ferroelectric, display cell, driving margin, hysteresis

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[†] Part of this research paper was orally presented at the 4th SPSJ International Polymer Science Conference (29th Nov. to 3rd Dec., 1992) Yokohama, Japan.

INTRODUCTION

Surface-stabilized ferroelectric liquid crystals (SSFLCs) in highly multiplexable bistable switching have attracted the great deal of attention towards the display device application areas over the past decade.[1] The polymer alignment layers within the cell are playing important role to align the smectic (SmC*) phase responsible for the bistability in ferroelectric liquid crystal display devices.[2] The final alignment will result mainly due to the physico-chemical interaction between the surface and the different liquid crystal phases. It has been reported earlier[3] that the molecules in the nematic phase are aligned parallel to the rubbing direction of the rubbed polymeric surfaces which develops the ordering with the polymer chains in the alignment layer and less on the physical deformation of the layer. The occurrence of smectic layers result at the nematic-smectic A transition. Obviously the molecular rearrangement during this phase transition is expected due to the longer axes orientation relative to the rubbing direction as a result the smectic layers will form perpendicular orientation relative to the rubbing direction. However polymer surfaces which exhibit highly ordered nematic phase do not show ordered smectic A layers.[4] Furthermore at the chiral smectic C-smectic A transition, tilting of the molecules will occur within the layers exhibiting few complication despite of the well aligned smectic A phase. On the other hand the formation of the chevron structure in the smectic C phase causes zig-zag defects in the cell.[5] Many publications have appeared about the different physical properties of the polymer alignment layers and their effect on the microscopic texture and bistability of surface stabilized ferroelectric liquid crystals with special interest to the degree of crystallinity,[3,6-10] the crystal structure,[10,11] the polarity of the polymer,[10,12] the surface tension,[10,13,14] the conductivity of the polymer,[10,15-17] the dielectric constant,[10,16,17] the Mark-Houwink coefficient,[10,18] and the layer thickness.[12,15-17] It has also been surveyed that the polymer alignment layers that can form monoclinic or triclinic crystals will exhibit bistability in liquid crystal cells,[10,11] however the bistability is not observed when other crystal classes or amorphous polymers are used.[3] According to Myrvold[18] in the practical ferroelectric liquid crystal displays four parameters are of major importance: (a) the defect density and hence the transmission of the dark state; (b) the cone angle between the two relaxed (no field) states; (c) the stability of the director orientation towards small disturbing fields; and (d) the time taken to switch the cell into one of the stable states is the fourth parameter. The former two factors determine the contrast of the device under memory conditions. The dark state transmission can be controlled by the proper mechanical and thermal treatment of the polymer alignment layer.

In addition to this, much has been reported in regard to the liquid crystal materials and the material constants in the past. Polymeric alignment films have also been reported in order to optimize the suitable material parameters in SSFLC cell to control SmC* liquid crystal phase.[2,3,18,19] In ferroelectric cells however it is difficult to obtain the sufficient bistability for multiplex driving[5] because the smectic layer structure in a homogeneously aligned cell is not like a bookshelf but like a chevron structure and the switching between two twisted states occur mainly under low applied voltage. Another inherent problem is that the previous image remain intact slightly even after removing the applied field. The reason for this so called 'Ghost Effect' is caused by the transfer of impurity ions included in a cell under the internal electric field due to polarization charges.

Recently, the discovery of the antiferroelectric liquid crystals have drawn the significant research focus because of their wide range of expected applications to display devices and their important basic research impact. The tristable switching in the SmC_A^* phase, where the antiferroelectricity in liquid crystals was firstly found which showed fast response, steep threshold and double hysteresis characteristics. The phenomenon is based on the molecular tilting in the neighbouring smectic layers projecting in the opposite directions, leading to an antiparallel orientation of the spontaneous polarization in successive smectic layers. Similarly as in liquid crystalline ferroelectrics the direction of the tilt and the antipolarization slowly precess around the layer normal from one pair of smectic layers to another. After applying an external electric field the antiferroelectric phase changes into a ferroelectric one leading to tristable switching. The tristable switching shows sharp threshold characteristics and a double hysteresis loop which satisfy the driving specification because in the AF-state the polarization charges cancel out between the layers and thus eliminating the 'Ghost Effect.' Thus the tristable switching is considered to be one of the superior methods at present to realize the high resolution ferroelectric liquid crystal display devices.

However, the alignment layers and their effect on the molecular alignment of the liquid crystal in the cell which would determine the switching characteristics have not been extensively studied in regard to the relationship between antiferroelectric liquid crystals and different polymer alignment layers. In 1988, Chandani *et al.*[20] and Hiji *et al.*[21] have reported the new phenomenon and its practical importance in the new electrooptical research field by studying the several type of the antiferroelectric liquid crystals. The tristable switching from an antiferroelectric state to ferroelectric state under an external electric field occurs in the SmC_A^* liquid crystal phase[22] and confirmed by the appearance of double hysteresis loop at the threshold voltage. The two threshold voltages of the field induced AFLC transitions, the threshold voltage (Vth) [Antiferro (AF)—Ferro (F)] can be defined as the absolute value of the voltage where the transition from the antiferroelectric state (AF) or dark state to the ferroelectric state (F) or bright state takes place. Thus the threshold voltage Vth (90) of the transition from the (F) state to the (AF) state, i.e., a bright state to a dark state is defined as the absolute value of the voltage where the transmission decreases to 90%. The threshold voltages obtained according to this definition are shown in Figure 2 as a function of frequency applied and triangular voltage. The antiferroelectric liquid crystal phase is expected to exist either in a helical structure in thick films or in an unwound structure at the substrate molecule interface. The possible expected molecular mechanism is shown in Figure 1.

The application of the polyimide layer as the alignment layer in prototype antiferroelectric liquid crystal display cell has also been reported by our research group.[23-25,26] In this paper, we report the effect of different alignment layers of fluoro and non-fluoro polyimide, polyamide-imide and polyamide on driving margin and double hysteresis behavior. Alignment layer (LQ-1800) was also compared

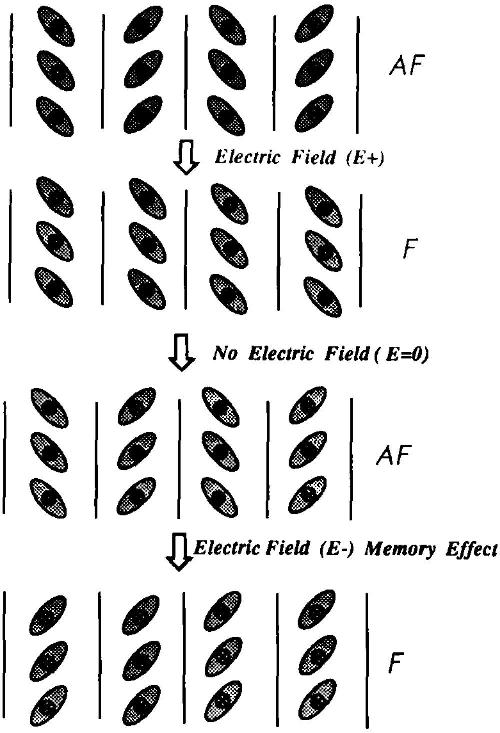


FIGURE 1 Molecular orientation mechanism of ferroelectric and antiferroelectric liquid crystal under an electric field.

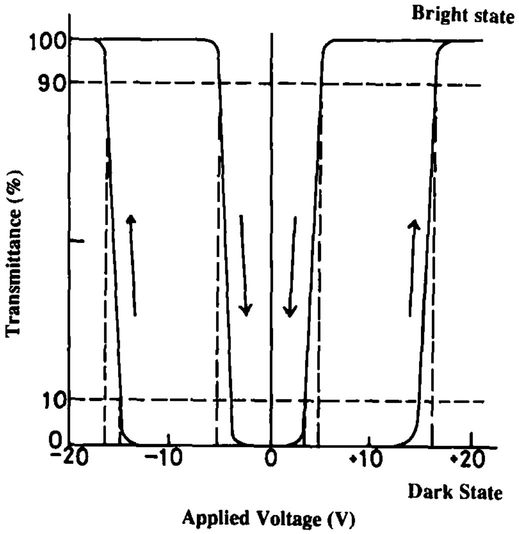


FIGURE 2 A typical electro-optical double hysteresis curve by applying a triangular voltage.

with our laboratory synthesized polymer alignment layers with respect to electro-optical properties.

EXPERIMENTAL

Fluoro and non-fluoro polyimides (PI-1, PI-2, PI-3, PI-4, PI-5, PIF-1), polyamides (PA-1, PA-2, PAF-1, PAF-2) and polyamide-imides (PAI-1, PAI-2, PAIF-1) were synthesized and characterized before using them as the alignment layers.[27,28] The polyimide alignment layer (LQ-1800) for comparative study was purchased from the Hitachi Chemical Co. Ltd., Japan. Each of the polyamide acids dissolved in N,N-dimethylacetamide (DMAc) was spin coated on transparent ITO (Indium Tin Oxide) electrode glass plates by using the spin coater and cured at 100, 200, and 300°C under vacuum for 1 h in each case to conduct the imidization into polyimide and the polyamide-imide thin surfaces. The polyamide coating was cured at 200°C under vacuum. The surface of the resulting low dielectric thin film was rubbed in parallel direction with nonwoven cloth made by nylon or polyester to obtain the orientation control film cell for liquid crystal. The rubbed substrate plates were assembled into a cell with the orientation layer facing to each other in such a manner that the rubbing directions of the upper and lower orientation control films were in parallel direction spaced with the cell gap 2.0 μm. The liquid crystal mixture AFLCB-1[22,26] consisting of three liquid crystals, MHPOBC, TFMHPOBC and TFMHPODBC was used to expand the temperature range of the SmC_A^* in the range of -20°C to ~80°C. The physical parameters and structures are tabulated in Table I. The AFLCB-1 was introduced by thermal heating to convert it into an isotropic liquid crystal phase which on slow cooling at a rate of 1 to 2°C/min was transformed to chiral smectic (SmC*) phase. In order to control the sample temperature, a hot stage (Mettler FP 82) and a central processor (Mettler FP 80) were used. The light transmitted through the sample cell was detected by a photodiode. The probe pulses synthesized in an arbitrary function synthesizer and supplied to the sample cells through a bipolar amplifier. The switching behavior of antiferroelectric liquid crystals in a homogeneous 2.0 μm cell was investigated with a polarizing microscope by applying the triangluar wave voltage. The optical response curves in the SmC* and SmC_A^* phase are presented in Figure 3a and 3b, respectively. The driving margin was calculated (Figure 2) according to the equation shown below:

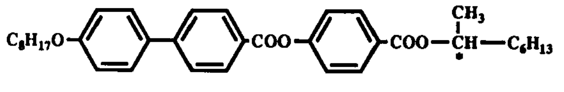
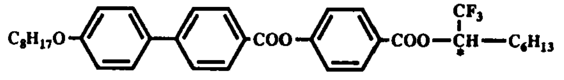
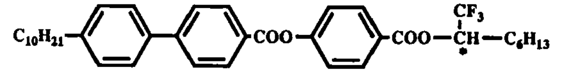
$$\text{Driving Margin (M)} = \frac{V_{\mathrm{th}}(10) - V_{\mathrm{th}}(90)}{V_{\mathrm{sat}}(90) - V_{\mathrm{th}}(10)}$$

Where $V_{\mathrm{th}}(10)$ is an increase of negative or positive voltage at which a transmission is 10%; $V_{\mathrm{sat}}(90)$ is an increase of negative or positive voltage at which a transmission is 90%; and $V_{\mathrm{th}}(90)$ is a decrease of negative or positive voltage at which a transmission is 90%.

Driving margin (M) is a parameter which offers an important guide in driving the element of the present invention as a simple matrix display device. When the

TABLE I

Characteristics of liquid crystals

Physical Parameter	AFLCB - 1
Spontaneous Polarization	218 nC / cm^2
 MHPOBC  TFMHPOBC  TFMHPDBC $\text{Cryst} \underset{-17}{\overset{-28}{\rightleftharpoons}} \text{SmC}_A^* \underset{85}{\overset{82}{\rightleftharpoons}} \text{SmC}^* \underset{89}{\overset{83}{\rightleftharpoons}} \text{SmA} \underset{99\,^\circ\text{C}}{\overset{92\,^\circ\text{C}}{\rightleftharpoons}} \text{Iso}$	

practical value of M > 1 or 2 a high contrast matrix display can be obtained. The relationship of the driving margin calculated from the double hysteresis loop can be explained on the basis of the steepness of the threshold voltage properties arising from the antiferroelectric to ferroelectric transition. The sharp steepness around the threshold voltage and wide hysteresis shape will favour the better driving margin values for the application of the pulse voltage.[29]

RESULTS AND DISCUSSION

Several types of fluoro and non-fluoro polyimides (PI-1, PI-2, PI-3, PI-4, PI-5, PIF-1), polyamides (PA-1, PA-2, PAF-1, PAF-2) and polyamide-imides (PAI-1, PAI-2, PAIF-1) were synthesized with high molecular weight (ηinh greater than 0.50 dL/g) and characterized by conventional analytical techniques in order to use them as the alignment layers for device cells. The micrographs of alignment texture observed in each cell are shown in Figures 4a, 4b and 4c. These micro texture photographs were taken under no field conditions. From the microtextural studies it is clear that the direction of the smectic layer normal closely coincided with the rubbing direction in all the cases. The uniform alignment was obtained in the cell using the non-fluoro polymer layers as the alignment layer while in the cells with other polymers (e.g., fluoro polymers) the multidomain state was formed. Such a difference of the alignment texture probably originates due to the packing arrangement of the polymer molecular structure. The switching behaviour of the AFLCB-1 in homogeneous cell having the PI-1 and PIF-1 was also studied with a polarizing microscope under a triangluar wave voltage, the successive changes in

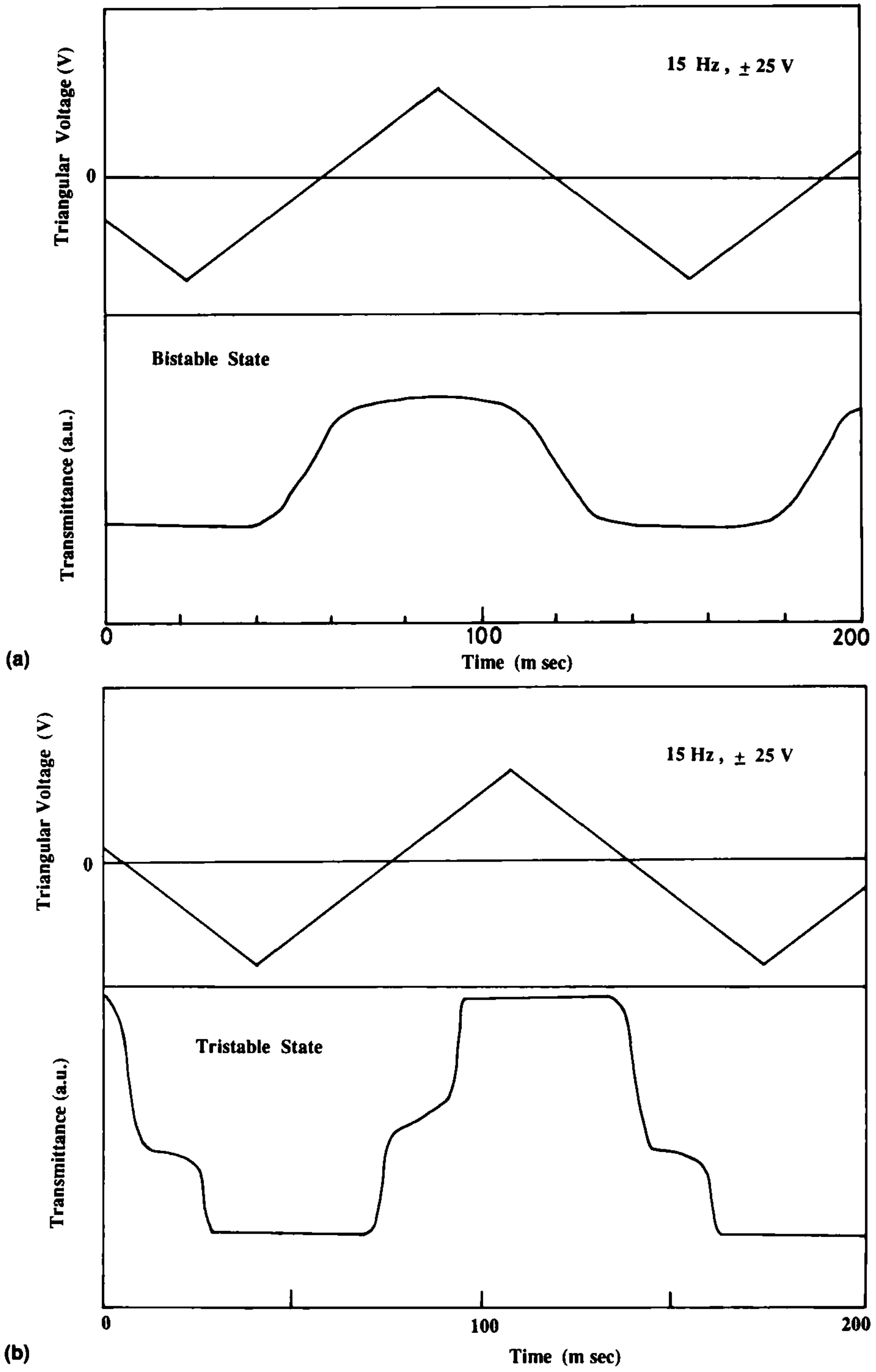


FIGURE 3 (a) Optical response curve in the SmC* phase; (b) optical response curve in the SmC_A^* phase.

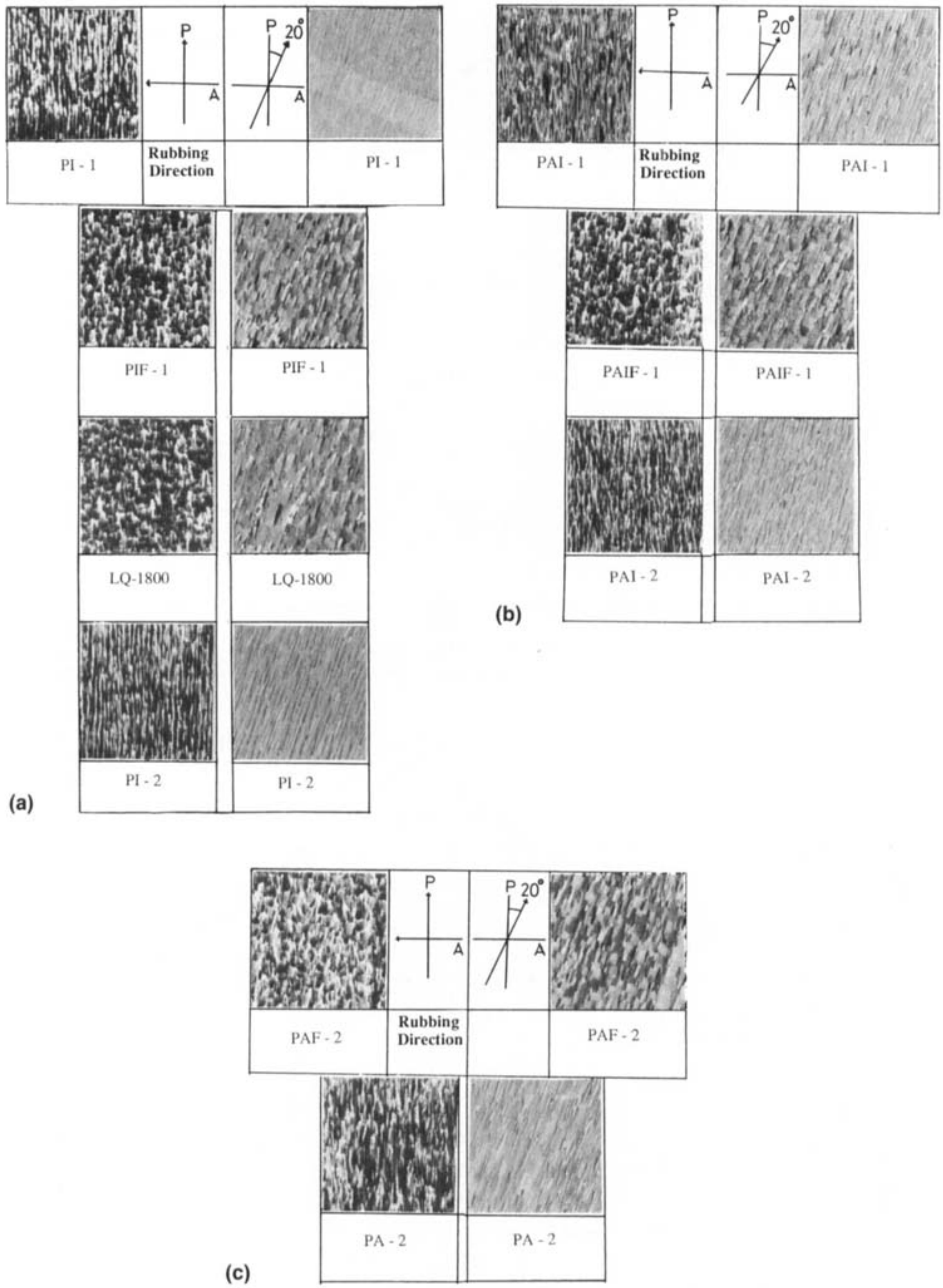


FIGURE 4 (a) Optical micrographs of alignment texture in SmC_A^* phase using polyimide (See Color Plate I); (b) optical micrographs of alignment texture in SmC_A^* phase using polyamide-imide (See Color Plate II); and (c) optical micrographs of alignment texture in SmC_A^* phase using polyamide. (See Color Plate III).

the transmittance was observed because of the transition from AF-state to the F-state as shown in the Figure 5. Similar experimental conditions were adopted for the measurement of the switching properties of the cells. In all the cells switching from AF-state to the F-state took place through two steps.[25] At the first step the change from the initial AF-state to an antiferroelectric intermediate state was observed as the continuous shift of an extinction position. At the second step the Ist state switched to the F-state together with the stripe domains which appeared at random position and grew only along the layer direction (Figure 5). We have also made the comparative study of the different cells having the polymer aligning

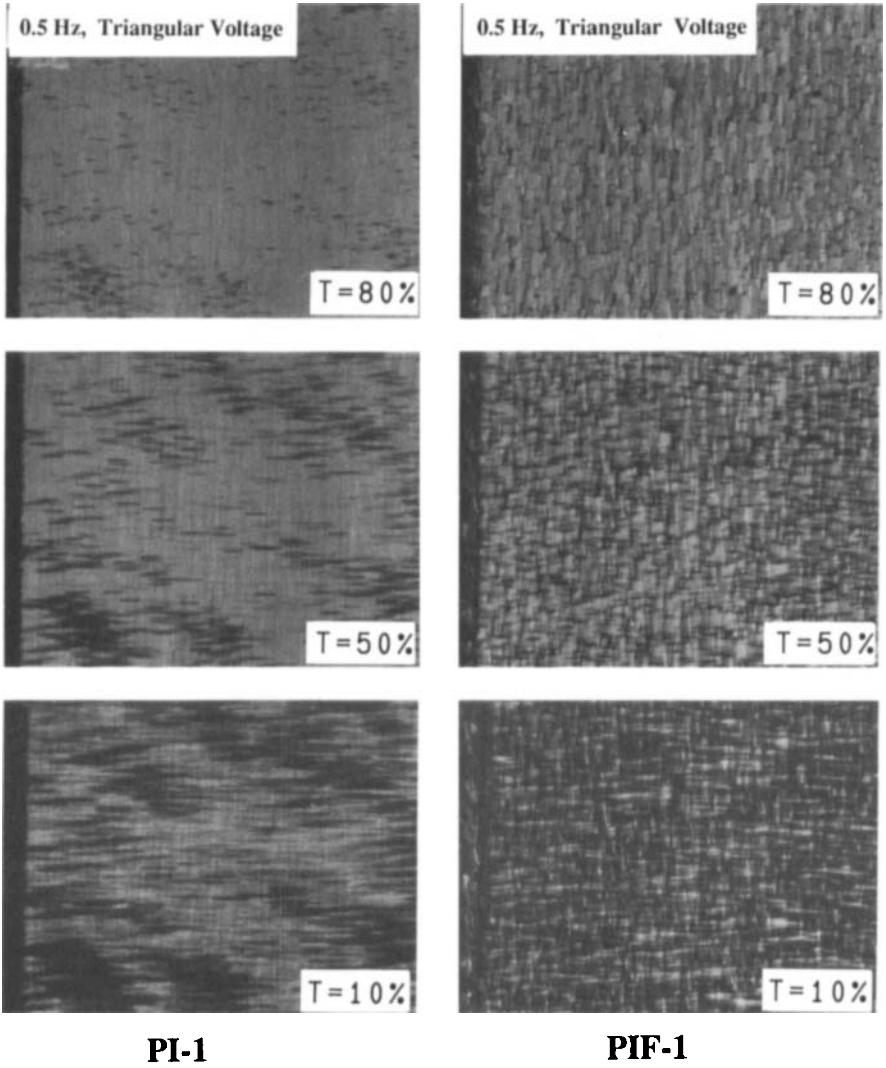


FIGURE 5 Photograph shows switching process under the application of the triangular wave of 0.5 Hz. The top and bottom photographs indicate the different transmission states. The cells are with the PI-1 and PIF-1 as the alignment layers (See Color Plate IV).

layers. In the ferroelectric cell tilted SmC* phases respond to the surface stabilized bistability which is attributed to the single hysteresis loop. Unlike the Smc* phase another phase SmC_A^* phase contributed the double hysteresis loop which has been confirmed as the antiferroelectric liquid crystal phase. In the SmC_A^* phase, the contrast ratio an important display property originated from the third stable state (AF-state) to the upper right or upper left state (F-state) is large enough for the application to display devices. Moreover, this switching exhibits a double hysteresis property after applying the triangular voltage. The shape of the hysteresis varies on changing the temperature, frequency and electric field. The polyimides based on pyromellitic anhydride and different diamine structures in the polymer backbone have shown the different driving margin values as calculated from the equation. Similar results were obtained for the polyamide-imides based on trimellitic anhydride and different diamines. However the polyamides under study exhibited either random alignment or lower values of driving margin. The driving margin varies which of course depend on the polymer structures having the polar groups such as C=O and C—N as in the polyimide structure. The structural symmetry of the polyimide layer is, however, high and the opposite dipoles will also have significant role in aligning the particular phase. On the other hand the polyimides (PIF-1 and PI-5) backbone substituted by hexafluoro or propane moieties have comparatively low structural symmetry due to the sterically hindered diamine units which in turn showed the low value of driving margin or even zero (e.g., LQ-1800). Other polyimide structures gave the driving margin values in the range of 0.90 to 2.2 depending on the polymer structures.

The structural variation in polyamide-imide and polyamide alignment layers with low linearity gave lower values of the driving margin. But the polyamide-imide structures with hexafluoro moiety contributed to the lower driving margins as compared to the non-fluoro counterpart. The changing shape of the double hys-

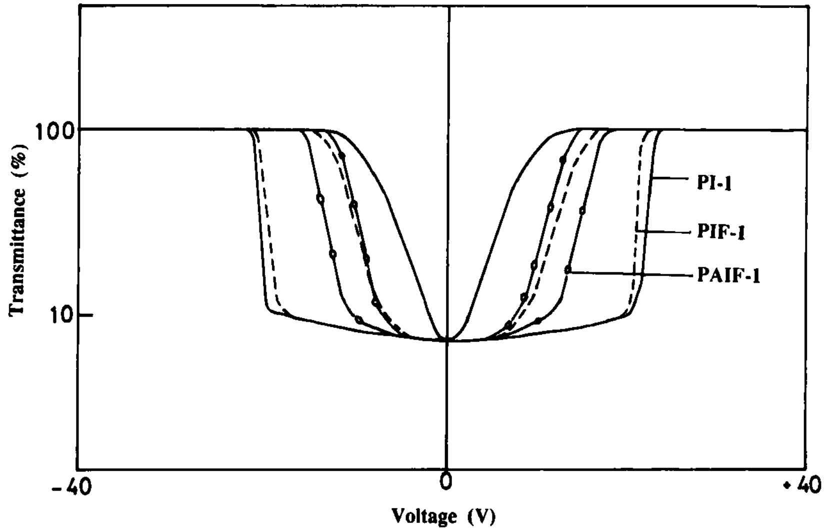


FIGURE 6 Optical hystereis curves measured on applying triangular wave voltage.

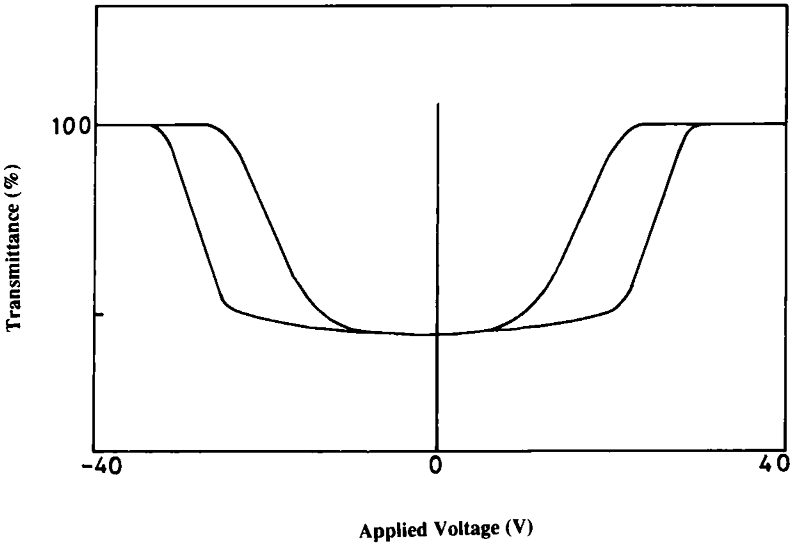
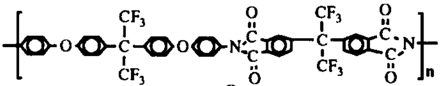
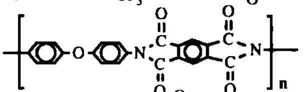
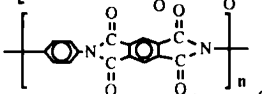
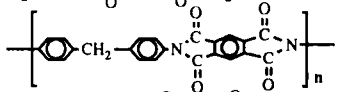
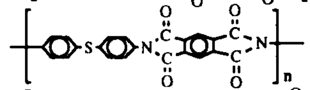
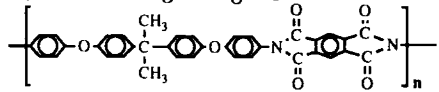
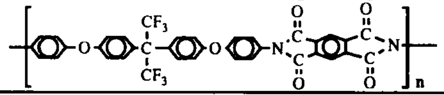
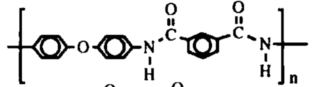
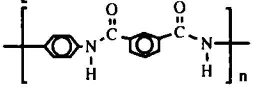
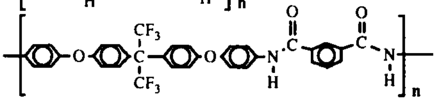
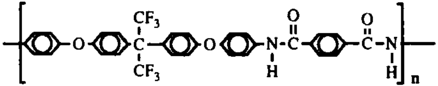
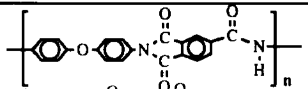
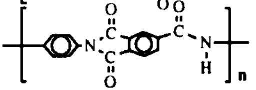
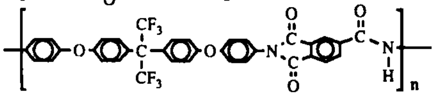


FIGURE 7 Optical hysteresis for LQ-1800.

teresis depended on the molecular structure of the alignment layers. This can be understood as further evidence for the interaction between the polar groups of AFLC and any polymer surface present in the cell. The pre-tilt due to rubbed polymer surface and epitaxial growth are also important factors besides the dipole moment. Both of these effects depend on the properties of both the substrate and the liquid crystal. In case of the polyamide the structural symmetry is lower, because of the meta orientation of the diacid part as a result the dipole moment might be larger hence the possibility of the hydrogen bonding with the neighbouring amide groups and therefore effectively reduce the net dipoles present. This shows the possible difference in different cell surfaces. The high symmetry monomeric units in the polymeric surface layer seem to play an important role in stabilizing AFLCs when high driving margins are to be achieved. The typical electrooptical double hysteresis shown in Figure 2 was utilized for driving a matrix display device. In the multiplex driving, the optical hysteresis in the positive voltage and the negative voltage was used alternatively by applying the offset voltage of $+V_0$, which is set exactly in the middle voltage between the threshold voltage of the Vth and the saturated voltage of V_{sat}. The triangular wave voltage was applied to the cell from external electrical source and voltage vs transmission characteristics were determined. With no voltage applied, the linearly polarized light transmitted through polarizer could not pass through polarizer thus showing an off state. As the plus voltage was applied the direction of the spontaneous polarization was directed along electric field direction. The liquid crystal molecules deviate from the angle of zero from the rubbing direction at this stage the light passed through polarizer rotates the plane of polarization by the action of birefringence of the liquid crystal and resulting the on state. The hystereses behaviour of practical observation is shown in Figure 6 using the alignment control layer PI-1, PIF-2 and PAI-2 in which voltage is plotted as abscissa and the transmission as the ordinate. On comparing

TABLE II

Effect of polyimide, polyamide and polyamide-imide alignment layers on hysteresis and driving margin using AFLCB-1 as liquid crystal mixture

POLYMER CODE	POLYMER STRUCTURE	VISCOSITY dL / gm	Driving Margin	ALIGNMENT with AFLCB-1
LQ-1800 for Comparison		–	0.0	GOOD
PI - 1		1.5	2.0	GOOD
PI - 2		1.0	2.1	GOOD
PI - 3		0.55	0.95	GOOD
PI - 4		0.68	1.60	GOOD
PI - 5		1.76	0.23	GOOD
PIF - 1		0.93	0.21	GOOD
PA - 1		0.90	–	RANDOM
PA - 2		0.80	1.68	GOOD
PAF - 1		1.0	–	RANDOM
PAF - 2		1.0	0.44	GOOD
PAI - 1		1.1	2.0	GOOD
PAI - 2		0.70	2.1	GOOD
PAIF - 1		0.90	0.43	GOOD

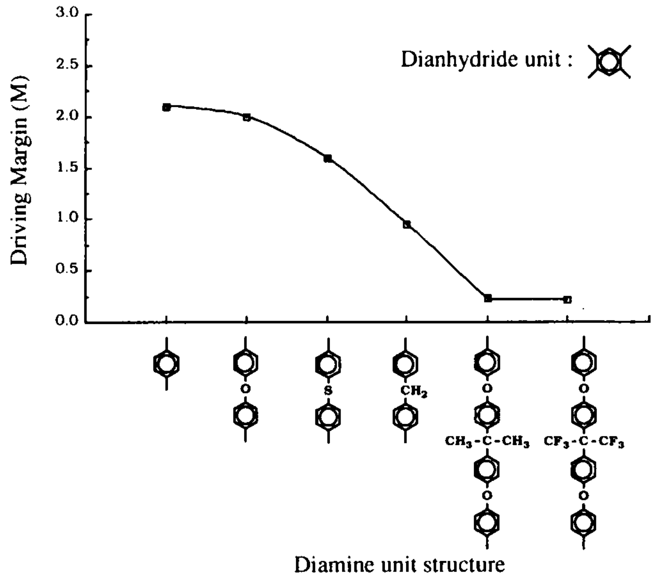


FIGURE 8 Effect of polyimide molecular structure on driving margin (M).

the electrooptical double hysteresis traces of different alignment layer with the commercial LQ-1800 polyimide layer (Figure 7) it is apparent that the alignment layer of the present invention shows fast switching from the off state (dark) to a on state (light). The voltage is increased from zero and exceeds a given level (non-linearity) when this voltage is decreased the on state is switched to a off state.

The Table II shows the experimental results of driving margin calculated from the expression described in experimental part. It is interesting to note that all the polymer alignment layers showed the good alignment except PA-1 and PAF-1 alignment layers. The driving margin results varied depending upon the molecular structure of alignment layers (Figure 8) under identical conditions. Only the cell having the good alignment of liquid crystal is able to display the driving margin. Accordingly, hysteresis shape has the total dependence on driving margin therefore it can be compared by plotting them together. In the Figure 8, molecular structure of polyimides were plotted against the driving margin values. The sterically hindered structure in the polyimide backbone gave the lower values of driving margin than that of the less hindered or linear structures.

CONCLUSIONS

Fluoro and non-fluoro polymer alignment layers used in antiferroelectric liquid crystal display device cells showed driving margin 0.21 to 2.2 depending upon the molecular structure of polymer surface.

Threshold voltages V_{th} (AF) and V_{th} (90) (F) depend on the alignment layers used in all the cells.

Good alignment control layers (PI and PAI types) developed were having the superior properties than that of the LQ-1800 polyimide alignment layer.

Polyimides and polyamide-imides were found to be good alignment layers as compared to polyamide layers in antiferroelectric liquid crystal cells. This difference arises because of the differences in polar character of the imide, amide-imide and amide linkages present in the polymer backbone. The improved alignment of imide-containing polymers can be explained on the basis of altered intermolecular interactions of the surface polar groups in these polymers and the liquid crystal molecules. These polymeric alignment layers are expected to be the promising candidate for AFLC display cell devices as we could successfully develop the prototype 6 inch size flat panel AFLC display device recently by using some of the developed polymeric alignment layers in the laboratory.

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