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Bulk optical properties in binary mixtures of antiferroelectric liquid crystal compounds showing V-shaped switching

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In order to clarify the origin of the V-shaped switching observed in thin cells of antiferroelectric/ferrielectric liquid crystals, bulk properties have been studied by means of helical pitch and conoscope measurements using thick free-standing films of binary mixtures with various mixing ratios. In the temperature range showing V-shaped switching in thin cells, helical structure clearly exists, indicating the existence of ordered phases. Some indistinct phase changes with temperature, coexistence of phases and quasi-continuous phase changes with an applied electric field were observed, suggesting a system with weak inter-layer correlation. By comparing the phase diagrams made using thin homogeneous cells and thick free-standing films, it was found that V-shaped switching occurs in the region where various subphases exist in the bulk. The appearance of many indistinct phases is consistent with the weak inter-layer correlation. In this way, it was concluded that the V-shaped switching occurs in tilted smectic layers, in which the tilt direction is weakly correlated along the layer normal.

1. Introduction

It is well known that ferroelectric and antiferroelectric liquid crystals are characterized by bistable and tristable switching behaviour, respectively [1, 2]. Recently, Inui *et al.* [3] discovered a novel V-shaped switching with neither threshold nor hysteresis. So far, only two mixtures/five compounds (figure 1) showing V-shaped switching have been reported [3, 4]. These materials show antiferroelectric-like or ferrielectric-like liquid crystal phases over a wide temperature range in the bulk states [3-9].

To explain V-shaped switching, Inui *et al.* [3] and Fukuda [5] have suggested, without any experimental evidence, a phase with randomly oriented C-directors due to the reduction of the layer-tilting correlation. However, the existence of helical structure was confirmed in one of the compounds (3 in figure 1) [9] and one of the mixtures (of compounds 1 and 2 in figure 1) [7] in the temperature range showing V-shaped switching. Thus, the phase with randomly oriented C-directors has not been confirmed. Nevertheless, it is reasonable to con-

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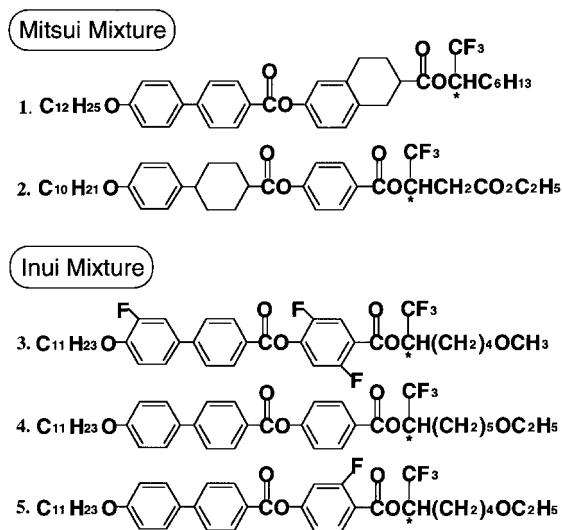


Figure 1. Chemical structures of compounds which show V-shaped switching.

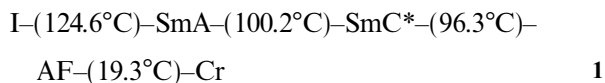
sider that the phase, in which the inter-layer correlation or the tilting correlation is reduced, manifests itself by V-shaped switching. At least in thin homogeneous

cells, the molecular orientational structure (where the molecular tilting direction is confined within the plane parallel to the substrates and the tilting sense is quasi-random) seems to be realized, since the texture in the absence of an applied field is uniformly dark [7, 8]. If there are ferroelectric and/or ferrielectric domains the size of which is larger than the wavelength of visible light, one sees domains with different brightness. Moreover another dark state, different from that appearing during V-shaped switching, was clearly observed [6, 9]. Thus, it is considered that the substrate surface breaks the reduced intrinsic tilting correlation in adjacent layers, and induces quasi-randomization of the tilt sense, resulting in V-shaped switching. Actually, the importance and sensitivity of the surface has been reported previously [8, 10, 11]. It should be noted that proto-type displays using V-shaped switching have been reported [10, 12], even though the origin of V-shaped switching is not clear.

In this paper, we focus on bulk properties in the mixture system showing V-shaped (thresholdless) switching, since otherwise it is impossible to elucidate the origin. We prepared phase diagrams using thin homogeneous cells and thick free-standing films of the mixture system on the basis of pitch, dielectric and electro-optic measurements and conoscopic observation. We report that the thresholdless behaviour appears even in the bulk which has a helical structure, and is an inherent bulk property of special liquid crystal materials with very weak inter-layer correlation.

2. Experimental

The materials used were mixtures of compounds **1** and **2** shown in figure 1, supplied by Mitsui Chemical Co. The phase sequences of the compounds are as follows:



Here AF means the antiferroelectric phase composed of higher superlattice structures different from the two-layer one, SmC_A^* [13]. This assignment was made because two ferrielectric phases appear in the higher and lower temperature regions of this phase in the mixtures of **1** and **2**, as shown later [7]; no ferrielectric phase should appear below the SmC_A^* phase [13]. However, SmC_A^* is also suggested from a short helicoidal pitch in this phase. But we tentatively assign this phase as AF.

Two types of sample, free-standing films and homogeneous cells, were prepared. For the preparation of free-standing films, we used a thin glass substrate with a rectangular hole (8 mm \times 1.5 mm) and suspended a rather thick film. Homogeneous cells were made by sandwiching the sample between two ITO coated glass

substrates separated by 2 μm thick polyester films. The substrates were treated with Toray SP-550 polyimide and only one surface was rubbed unidirectionally.

To construct a phase diagram of the bulk material, transition temperatures were determined and phases were identified by observing the electric field dependence of conoscopic figures, using the method described previously [13–15]. Namely, the centre of the conoscopic figure scarcely shifts in the antiferroelectric phase, unless the electric field exceeds a threshold value for the field-induced phase transition from antiferroelectric to ferrielectric or ferroelectric phase. In the ferrielectric and ferroelectric phases, the centre moves with increased electric field and the two phases are distinguished by whether the biaxial optical plane is parallel (ferrielectric) or perpendicular (ferroelectric) to the field. It has been proved, however, that this feature of conoscopy is unsatisfactory in identifying the phase of liquid crystal phases [16]. Helical pitches determined by transmittance measurements were also used in constructing the phase diagram. To assign the phase, oblique incidence transmittance spectra were also measured.

For phase diagrams in thin homogeneous cells, optical microscopy (Nikon, OPTIPHOT-POL) and dielectric measurements (HP4192A) were made. Electro-optic measurements under crossed polarizers were carried out by applying a triangular electric field with a variety of frequencies and varied temperature. The polarizer axes were set parallel and perpendicular to the smectic layer normal.

3. Results

3.1. Results in thin homogeneous cells

We first show the phase diagram (figure 2) made by observing the texture under the polarizing microscope in homogeneous cells. Although compound **2** did not have the SmA phase, and well aligned homogeneous cells were not obtained, we could see texture changes in the

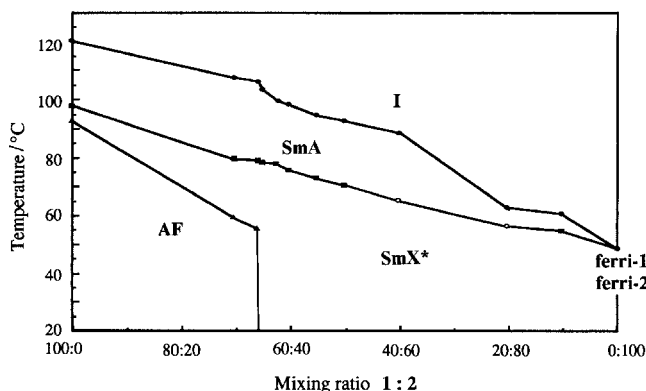


Figure 2. Phase diagram of the binary system of compounds **1** and **2** in thin homogeneous cells.

phase transition between two ferroelectric phases, ferri-1 and ferri-2. In the pure compounds **1** and **2**, the phase sequences coincide with the results in free-standing films, as will be shown. However, phase diagrams obtained in homogeneous cells and thick free-standing films of the mixtures are different from each other. Particularly, an undetermined SmX* phase appears over a wide temperature range in thin homogeneous cells. As shown below the phase diagram is supported by other measurements.

Figure 3 shows the electro-optic (EO) responses observed at various frequencies and temperatures in thin homogeneous cells of five mixtures with the mixing ratios, **1**:**2** = (a) 100:0, (b) 70:30, (c) 50:50, (d) 30:70 and (e) 10:90 by weight. In the pure compound **1**, the EO response showed typical tristable switching at 55°C, as shown in figure 3(a). Even in the AF phase, an anomaly starts to appear when the temperature increases; a new 'tip' at almost 0 V, not seen at lower temperatures, appears at high frequencies and has lower transmittance than the AF phase, as clearly seen in the results at 91°C and 89°C. In the upper temperature phase, which corresponds to SmC* in the bulk state and is called the SmX* phase, quasi-V-shaped switching was observed, though slight hysteresis and a metastable state appeared. In the SmA phase, the electroclinic effect was observed.

In the mixture, **1**:**2** = 70:30, similar behaviour was observed in the AF phase, though stable tristable switching was no longer seen, as shown in figure 3(b). On increasing the temperature in 4°C steps from the AF phase to the SmX* phase, the EO response gradually changed: the threshold disappeared and ferroelectric-like or thresholdless behaviour dependent on frequency appeared. Thus, the phase transition could not distinctly be observed from the EO response. Nevertheless, the existence of the AF phase was clearly recognized from the textures. When the applied electric field was turned off, the molecular arrangement seemed to return to antiferroelectric. This situation is clearly seen in figure 4. We observed tristable switching during the first half positive field period (arrow 1) when application of the triangular wave started from zero field. In the next half negative field period, the switching pattern changed to V-shaped form near zero field (arrow 2), although the switching was associated with the emergence of ferroelectric-like order (arrow 3). This switching in the second half period occurred repeatedly [52°C, 1 Hz in figure 3(b)]. It is also noted that the movement of the domain boundary characteristic of tristable switching [17, 18] disappeared and the texture changed almost uniformly during the whole switching process at 52°C and 1 Hz even in the region of arrow 3. Thus, figure 4 clearly shows that thresholdless switching can be achieved dynamically [9], even if the stable state is the

normal antiferroelectric phase. In the higher temperature range in the SmX* phase, ideal V-shaped switching was realized.

In the mixture **1**:**2** = 50:50, as shown in figure 3(c), the temperature range exhibiting V-shaped switching is expanded, though a slight hysteresis exists. The hysteresis at higher temperatures (60, 50 and 40°C) is abnormal. By abnormal, we mean that the field required to attain the ferroelectric state, $E(\text{to ferro})$, is lower than the field required to start switching from the ferroelectric state $E(\text{from ferro})$, as shown in one of the frames (50°C, 0.1 Hz) of figure 3(c). The direction of loop is opposite to the normal one, where $E(\text{to ferro}) > E(\text{from ferro})$. The hysteresis at low frequencies and at lower temperatures (30 and 24°C) is also abnormal. The change from abnormal to normal hysteresis occurs at higher frequencies as the temperature increases (e.g. 0.5 Hz at 24°C and 1 Hz at 40°C). Hence, hysteresis-free behaviour was obtained at higher frequencies even at 50 and 60°C. The appearance of hysteresis seems to be caused by surface charges due to ferroelectric dipole orientation and the movement of charged carriers in the cells [11]. The effect of charges could be prevented by using materials free from impurity ions and by coating insulating layers onto ITO. It is important to note that the darkest level is quite low. Thus, in this mixture, the switching is not associated with threshold, and V-shaped switching is realized.

Results from mixtures **1**:**2** = 30:70 and 10:90 are shown in figures 3(d) and 3(e), respectively. All the hystereses shown in figure 3(d) are abnormal, as indicated in the response at 50°C and 0.1 Hz. The hysteresis at 25°C of figure 3(e) is normal, while that at 45°C is abnormal. At 35°C, normal hysteresis changes to abnormal between 0.5 and 0.1 Hz, as indicated by arrows. Comparing with figure 3(c), the width of hysteresis in figure 3(e) becomes larger. At 0.1 Hz and 45°C of figure 3(e), even reverse switching occurred before the external field returned to zero, giving a very wide hysteresis. This is explained by a reverse field caused by surface charges and accumulation of ionic carriers. In the mixtures with a larger fraction of **2**, the ferroelectric state is stabilized under an electric field, producing surface charges at the interfaces between the liquid crystal material and the alignment layer, and leading to the subsequent reverse field. This switching is different from typical V-shaped switching not only in the hysteresis but also in the darkest level during the switching—see the results at 25°C of figure 3(e). It should also be noted that domain formation, i.e. boat-shaped domains, is associated with switching particularly in the mixture **1**:**2** = 10:90.

In a different method for detection of phase change, the temperature dependence of the dielectric constant was measured using materials with the mixing ratios

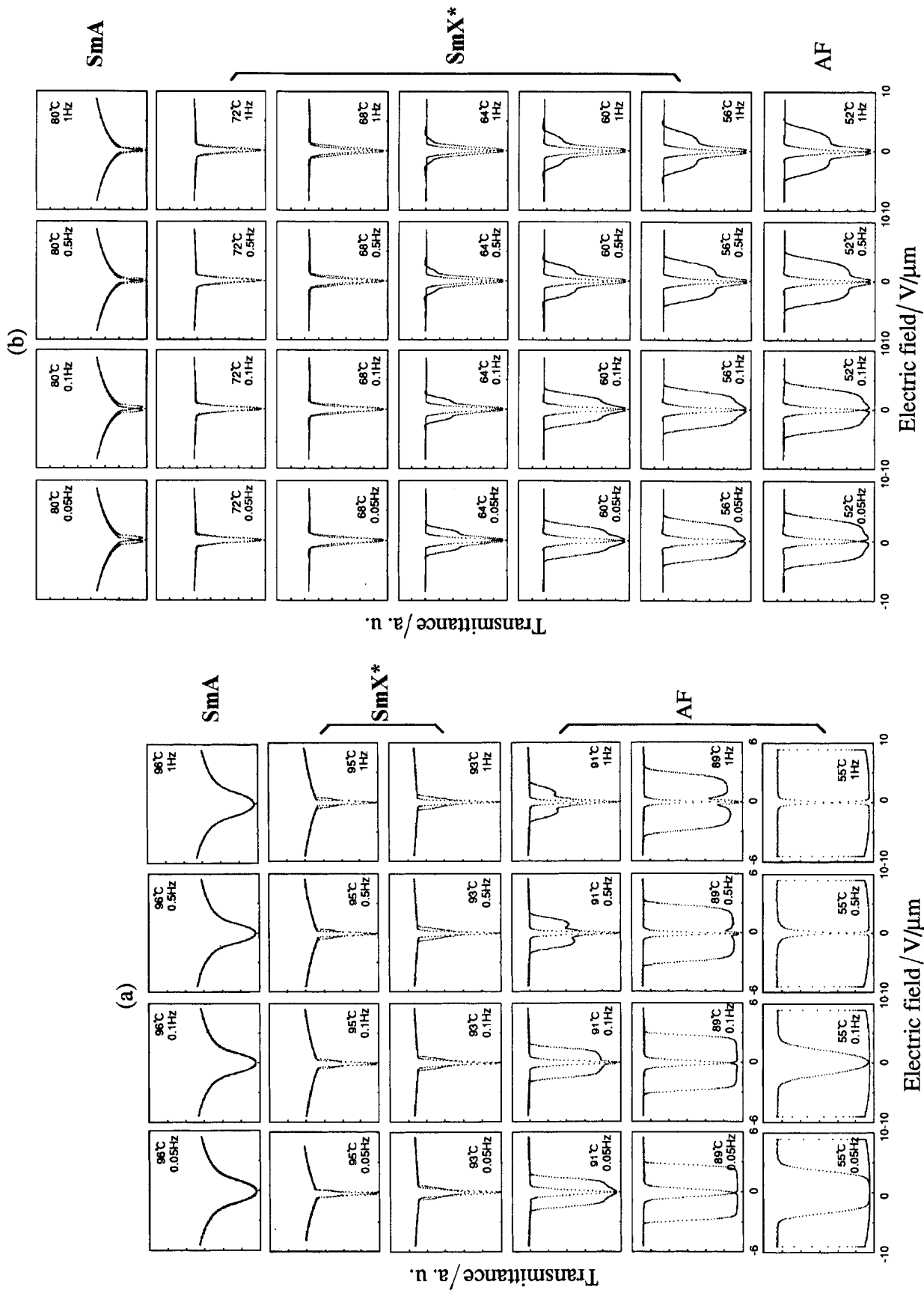


Figure 3. Electro-optic switching at various temperatures and frequencies in mixtures with 1:2 ratios of (a) 100:0, (b) 70:30, (c) 50:50, (d) 30:70 and (e) 10:90 by weight.

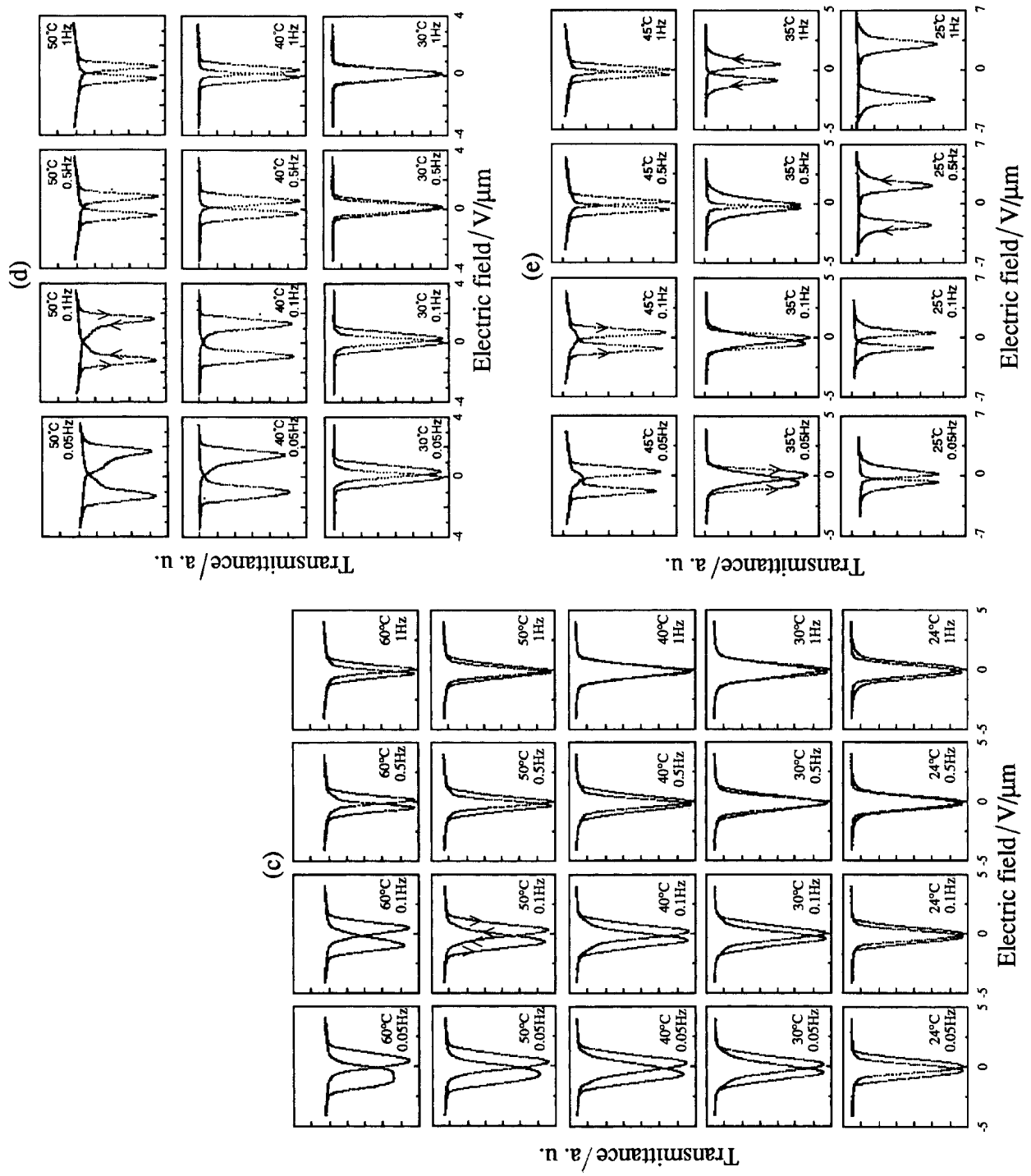


Figure 3. (continued).

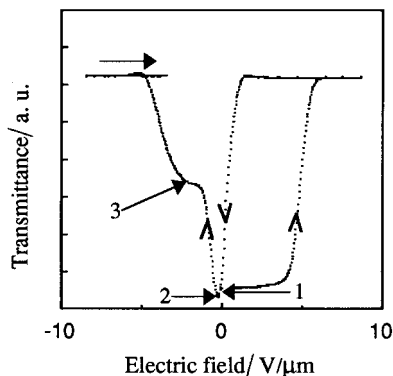


Figure 4. Initial behaviour of electro-optic switching when a triangular wave voltage of 1 Hz is applied from zero voltage in the mixture of 1:2 = 70:30 at 52°C. This figure indicates that the initial state is antiferroelectric.

1:2 = 66:34 and 63:37. The two phases, SmX* and AF, would be distinguished by the existence of the Goldstone mode, since the SmX* phase in this region corresponds to the SmC* phase in free-standing samples. Quite surprisingly, the SmX*–AF phase transition disappears on slightly decreasing the fraction of 1, as shown in figure 5, where results in the mixtures 1:2 = 66:34 and 63:37 are shown. Thus, the AF phase quite suddenly disappears at around 1:2 = 66:34.

3.2. Results in free-standing films

Figure 6 shows the phase diagram of the binary system determined by observing conoscopic figures in free-standing films. In compound 1, the antiferroelectric phase appears similar to the ordinary SmC_A* phase but it is judged to be another antiferroelectric AF phase [13], as mentioned above. Compound 2 does not have the SmA phase but has two ferroelectric phases. In the second ferroelectric phase, we could not observe conoscopic figures, since network-like textures (which appear

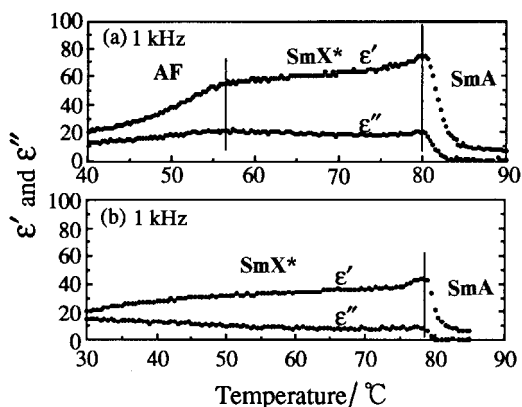


Figure 5. Temperature dependence of dielectric constants performed by applying $7 V_{pp} mm^{-1}$. (a) 1:2 = 66:34, (b) 1:2 = 63:37.

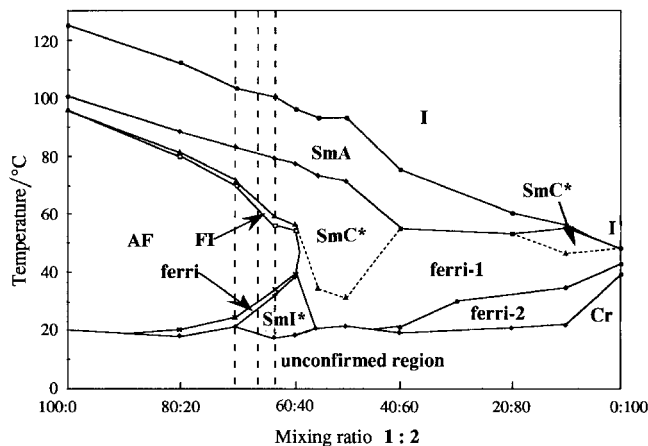


Figure 6. Phase diagram of the binary system in thick free-standing films: mixing ratios by weight.

to be different from the one reported by Gorecka *et al.* [19]) appeared when the phase transition from ferri-1 to ferri-2 took place. The ferroelectric characteristics in the ferri-2 phase were confirmed by conoscopic observation in the mixtures of 1:2 = 10:90 and 30:70, in which the same network-like texture was also observed at the ferri-1 to ferri-2 phase transition but disappeared at lower temperatures.

This phase diagram has three special features. First, with decreasing wt % of 1, the AF phase region gradually becomes narrower and abruptly becomes a ferroelectric phase around 1:2 = 60:40. Secondly, the ferroelectric phase seems to exist in a narrow range around 1:2 = 10:90. Thirdly, although the phase transition from ferroelectric to ferroelectric must be first order, it is impossible to confirm this. The phase diagram shown in figure 6 was made only by conoscopic observation under an applied field. It is noted that the phase boundaries are not distinct, particularly in mixtures in which the proportion of 2 is more than 50 wt %, as will be shown later.

As complementary data to help understand the phase diagram shown in figure 6, we show in figure 7 the temperature dependence of optical pitch, as determined by the dip position of the transmittance spectra due to selective reflection loss. In addition to the ordinary selective reflection band (half pitch band, closed circles), the wavelength of the full pitch band is shown by open circles. The existence of the full pitch band confirms the SmC* or ferroelectric phase; in this the index ellipsoid is tilted with respect to the layer normal, while that in the antiferroelectric structure is along the layer normal [1].

In compound 1, the existence of the SmC* and AF phases can be clearly recognized. The absence of a full pitch band in the AF phase indicates that this is actually antiferroelectric [1]. The rather short pitch of about 700 nm, however, suggests the SmC_A* phase instead of

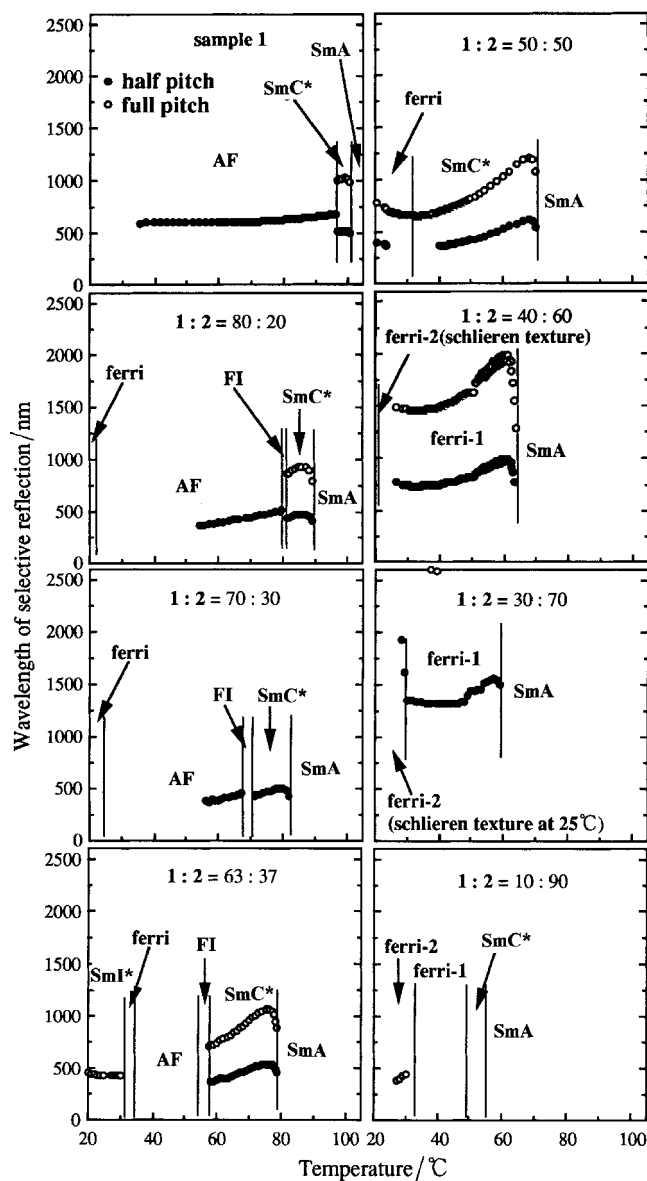


Figure 7. Temperature dependence of helical pitch in several mixtures. The wavelength of the full pitch band in oblique incidence is also shown.

the AF phase, since the subphases between SmC^* and SmC_A^* normally have long pitch because of the competition between SmC^* and SmC_A^* with helices of opposite handednesses. The reason why this phase was assigned to AF is the existence of a ferrielectric phase (designated as ferri in the phase diagram) below this phase [7], as mentioned above. However, the existence of the ferri phase was concluded only by conoscopy; the assignment of AF is therefore still uncertain.

Another ferrielectric phase (designated as FI in the phase diagram) is present between AF and SmC^* , as clearly shown in $1:2 = 80:20$ and $70:30$. The helical

pitch cannot be seen in the measurable wavelength range between 300 and 2500 nm, suggesting a very long pitch. On the other hand, the lack of data points in the AF phase in $1:2 = 63:37$ means a very short pitch below 350 nm, judging from the variation of pitch in the mixtures $1:2 = 100:0$, $80:20$ and $70:30$.

The result of $1:2 = 50:50$ clearly indicates that the AF phase suddenly disappears between $1:2 = 60:40$ and $50:50$. Instead, the SmC^* phase expands. The pitch changes continuously from SmC^* to ferri-1. The assignment to the two different phases, SmC^* and ferri-1, is due only to conoscopy measurements. Moreover, as will be shown later, the difference in conoscopy figures is very small. The temperature dependence of the pitch in $1:2 = 40:60$ is very similar to that in $1:2 = 50:50$. The only difference is in the absolute values of the pitch. Namely, the pitch becomes larger between $1:2 = 50:50$ and $40:60$, although it is almost constant between $1:2 = 100:0$ and $50:50$. This tendency, i.e. the increase of pitch with increasing fraction of 2, continues in 2-rich regions such as $1:2 = 30:70$ and $10:90$.

A further interesting observation may be made on the results of $1:2 = 40:60$ and $30:70$, which are enlarged in figure 8. In both mixtures, the temperature dependence of the pitch shows slight discontinuous changes at 51°C in $1:2 = 40:60$ and at 48 and 53°C in $1:2 = 30:70$, suggesting that the ferri-1 phase consists of multiple phases. Moreover, in $1:2 = 40:60$, two reflections were observed at the higher temperature range. According to the transmittance spectra shown in figure 9, the relative depth of the two dips varies with temperature. The observations shown in figures 8 and 9 suggest that the region which is assigned to SmC^* , ferri-1 and ferri-2 in the phase diagram in thick free-standing films (and coincides with the region assigned to SmX^* in thin homogeneous cells) has ambiguous phase boundaries, and seems to exhibit quasi-continuous phase transitions and the coexistence of two phases.

Lastly we show the result of conoscopy observations. Figure 10 shows the electric field dependence of conoscopy figures at various temperatures in the mixture $1:2 = 50:50$. The variation with field is quite continuous. During the process, ferrielectric-like conoscopy figures can be seen particularly below about 32°C . The angle between the two optic axes increases and decreases and a change to ferroelectric with increasing field, is recognized in the figures between 90 and 240 V mm^{-1} at 26°C . However, the difference between the two types of figures, ferrielectric and ferroelectric, is not large, again suggesting the existence of ambiguous phases.

Using the centre of the conoscopy figures at 28°C , we obtained the apparent tilt angle as a function of applied field; the result is shown in figure 11. The apparent tilt angle shows no threshold field and increases continuously.

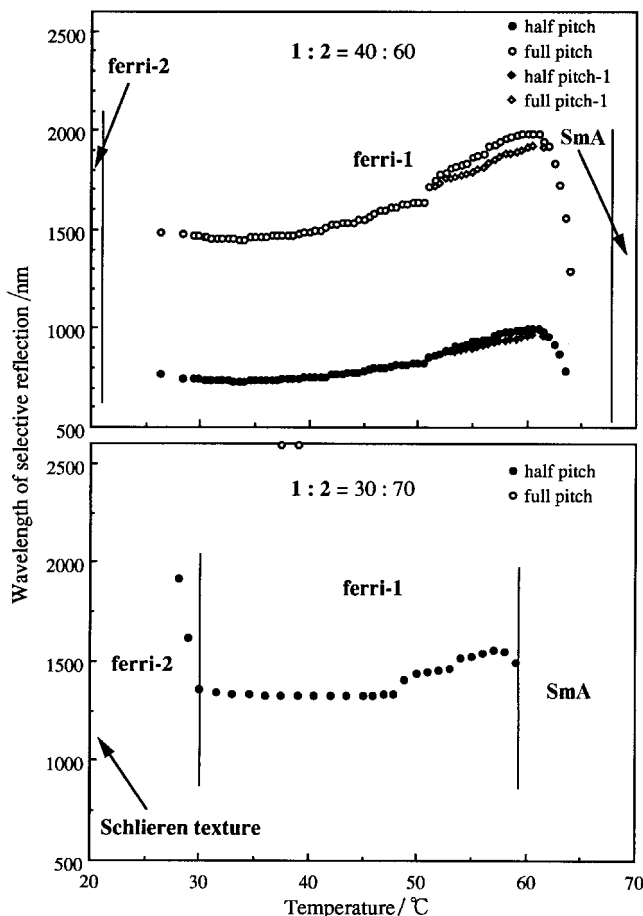


Figure 8. Temperature dependence of helical pitch in (a) 1:2 = 40:60, (b) 1:2 = 30:70, in enlarged scale.

Although the final tilt angle of about 25° was not attained within the experimental observations, the change from ferrielectric-like figure (around $105 \sim 150 \text{ V mm}^{-1}$) to ferroelectric-like figure (at higher field) is included in figure 11. Thus, thresholdless behaviour was confirmed, even in the bulk. We should note the present thresholdless behaviour observed in the bulk state is unique for materials showing V-shaped switching. Actually in MHPOBC, which shows typical tristable switching, the transition from SmC_γ^* to SmC^* is associated with a threshold, and a metastable apparent tilt angle of SmC_γ^* was actually observed in conoscopic observation [14, 15]. In the present case, the apparent tilt angle increases continuously with increasing electric field, although the conoscopic figures change from the ferrielectric to ferroelectric form.

The transmittance T of light for normal incidence, under crossed polarizers which coincide with the layer and layer normal directions, is given by

$$T = \sin^2 \Theta \sin^2(\pi d \Delta n / \lambda) \quad (1)$$

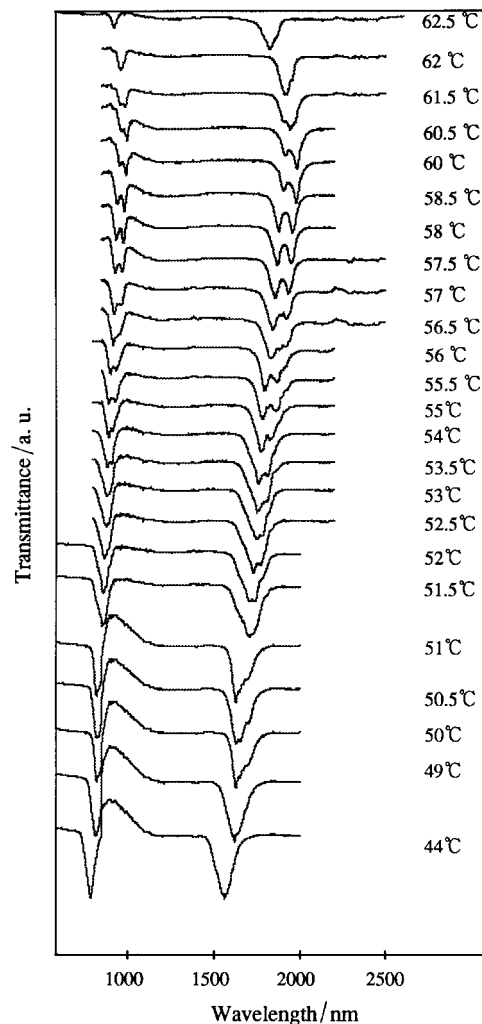


Figure 9. Temperature variation of the transmittance spectra showing selective reflection.

where Θ is the apparent tilt angle, d is the cell thickness, Δn is the anisotropy of the refractive index and λ is the wavelength. In ordinary switching in ferroelectric liquid crystals, Δn is approximately constant during the switching. On the other hand, we have confirmed that Δn depends on the electric field E , as Θ does in V-shaped switching [20]. Hence, it is impossible to predict the electro-optic response curve in homogeneous cells using only Θ -value data.

4. Discussion

As mentioned above, V-shaped switching occurs in materials which have ambiguous phase sequences. A dark state characteristic of V-shaped switching arises even in normal phases such as the AF phase, only in the switching process but not in the static state, as shown in figure 4. Surprisingly, a darker state is achieved in V-shaped switching than in the static AF state. In a

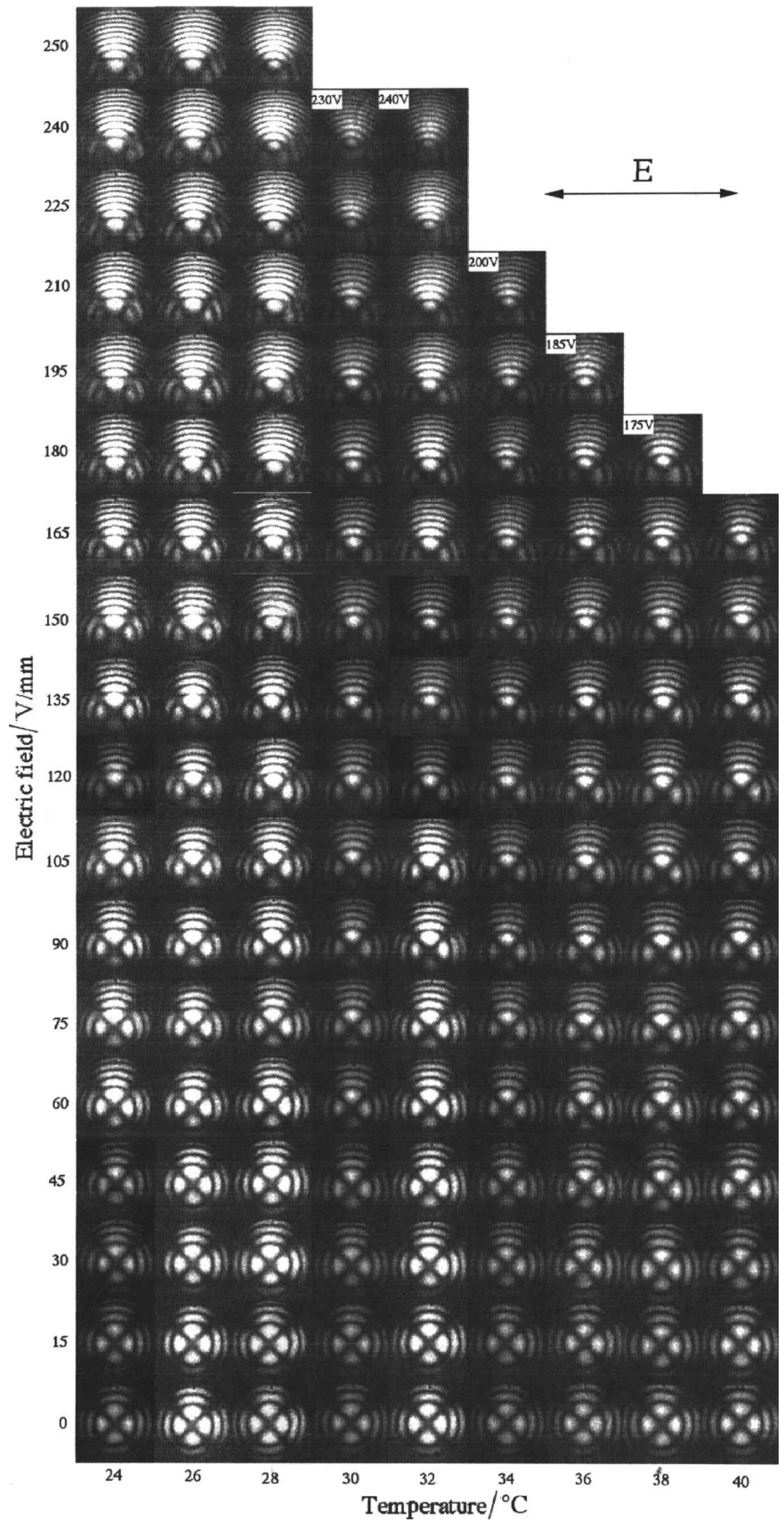


Figure 10. Conoscopic figures at various temperatures and applied fields in 1 : 2 = 50 : 50.

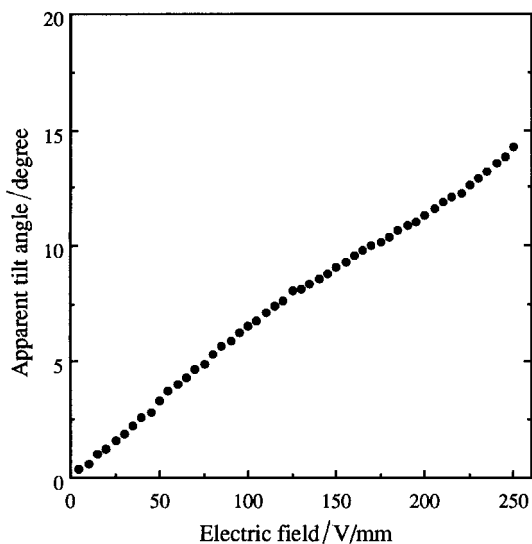


Figure 11. Apparent tilt angle as a function of applied field, determined by observing the conoscopic figures.

region like the SmX^* phase, where ideal V-shaped switching occurs, the brightness change occurs uniformly and remains dark when the field is turned off, though the dark level is slightly higher compared with that in the dynamic process [11].

In antiferroelectric materials, many sub-phases appear [12]. These sub-phases have been explained as appearing as a consequence of the competition between ferroelectricity and antiferroelectricity. We adopt the Ising model rather than xy model; i.e. all the phases are described as regular sequences of right- and left-tilted layers. Let us consider V-shaped switching based on this phase structure, since the phase with randomly ordered C-directors [3], even if such a phase exists, is not realized in the present system.

As mentioned above, V-shaped switching occurs in the region of the temperature/mixing ratio phase diagram where several phases coexist or vary smoothly, with ambiguous phase boundaries depending on the mixing ratio or temperature; this is suggested in figures 7–9. This lack of well-ordered long range orientation must be a consequence of weak inter-layer correlation. The weakness of the inter-layer correlation is also suggested by the strong influence of surface alignment layers on the EO response, the phase diagrams, etc. These details will be reported separately [11].

One can visualize the ambiguous phase sequences and associated weak inter-layer interactions in the phase diagram calculated using the ANNNI (axially next nearest neighbour interaction) model by Yamashita [21]. According to this model, there are four ground states: SmC^* , AF, SmC_γ^* and SmC_A^* . Near the curved critical boundary between ordered and disordered phases,

various subphases appear. It is possible that these phases appear with changing temperature. The temperature dependence of the phases depends on the strength of inter-layer interaction, i.e. (next) nearest neighbour interaction. In the mixtures of balanced compositions, the inter-layer interaction may be weak, since the phases have ambiguous phase boundaries and are located near the critical boundary in the phase diagram due to the ANNNI model. On the contrary, 1-rich and 2-rich mixtures show a rather distinct phase sequence, close to the ground state.

If materials possess the phase behaviour mentioned above and, as a consequence, have weak inter-layer correlation and relatively strong intra-layer correlation, layer-by-layer orientation regularity exists only with weak interaction along the layer normal; it is easily destroyed by applying an electric field or surface constraint. In this case, two-dimensional Langevin-type switching occurs, exhibiting a thresholdless brightness change even in the bulk state. If the materials are sandwiched between substrates whose surfaces exert negligible polar interaction forces on the liquid crystal molecules, and merely forces them to align parallel to the surface, thresholdless V-shaped switching is achieved. In the case of homogeneous cells the rather short range orientational order of the tilt sense along the layer normal cause the molecules to tilt uniformly through the switching associated with stripe domains, the width of which is much less than the wavelength of visible light. Since the width is so thin, an apparently uniform brightness change is observed. When the electric field is turned off, the fraction of layers tilted to the right is the same as that to the left, regardless of the orientational order, as long as the range of order is short compared with the wavelength. Therefore, V-shaped switching appears in ferroelectric and ferrielectric phases as well as in antiferroelectric phases, if the above-mentioned condition, namely weak inter-layer correlation, is satisfied. The switching process can be simulated by a one-dimensional Langevin process, which gives a sharper transmittance change compared with those in two- or three-dimensional Langevin processes. A simulation which includes the birefringence change during switching is now in progress, and will be reported in a separate paper.

5. Conclusions

The physical properties of binary mixtures which show V-shaped switching were studied. The phase diagrams involving temperature and mixture ratio were constructed using thin homogeneous cells and thick free-standing films. In the former case, the EO response and dielectric measurements were used to identify phases and phase boundaries. In the latter case, conoscopy

and helical pitch measurements were carried out. It was found that ambiguous phase sequences are the key for V-shaped switching to be exhibited, and that thresholdless switching occurs even in the bulk state in such phases. Based on these experimental findings, it is concluded that V-shaped switching has its origin in weak inter-layer correlation.

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