The effect of confinement on the stability of field induced states and on supercooling in antiferro-ferroelectric phase transitions in chiral smectic liquid crystals

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We investigate both the supercooling and the hysteresis phenomena of the phase transitions between the smectic C^* and the smectic C_A^* phases driven by temperature and electric field, respectively. These two phenomena show similar characteristics for the dependence of transmittance on both the cell thickness and the applied field. The mechanisms for large supercooling and large hysteresis in thin cells are shown to correspond to the suppression of the propagation of solitary wave by the surfaces. Furthermore, these two phenomena are shown to be controlled by a moderate ac field applied across the cell. We present a clear evidence for the existence of at least two field induced subphases (called states here) between the antiferroelectric and the ferroelectric phases. These are found to correspond to the field induced three-layered and four-layered structures through a comparison of experimental results on the tilt angle and its simulation as well as by discrete changes in the texture by increasing the electric field. The correspondence between the thermotropic phases and the field induced states is demonstrated through measurements of the supercooling/superheating and of the hysteresis as a function of the cell thickness. The instability in the field induced states depends strongly on the cell thickness, and the various states are not observed in a cell of 1.6 μm thickness. © *2009 American Institute of Physics*. [doi:10.1063/1.3226865]

I. INTRODUCTION

From the fundamental study of thermodynamics and of electrodynamics of systems with molecular dipoles, it is known that thermal energy gives rise to the molecular thermal fluctuations. An increase in the temperature, therefore, favors a disordered state with a consequential decrease in the order parameter. On the contrary, the electric field aligns the dipoles and leads to an increased order parameter for the aligned state. Despite the intrinsic differences that might exist and the opposite actions caused by the thermal energy and the electrical coupling energy, the two physical stimuli occasionally lead to the same result. This is found to be the case at least for the tilted smectic liquid crystalline materials under discussion here. It is presumably for the reason that the orientational order parameter in the case of field induced phase transitions plays a minor role in chiral smectics as opposed to a reduction in the polar energy with field. Phase transitions are commonly observed in the condensed matter physics including those in liquid crystals. These have been extensively investigated both theoretically and experimentally.[1–3] In thermotropic liquid crystals, a phase transition between the two phases is induced by the thermal energy, that is, by changing the temperature. At the intrinsic phase transition temperature T_C, the free energies of the two phases become the same. However, the actual phase transition may occur at T_{0c} upon cooling, or at T_{0h} upon heating, and $T_{0c} \leq T_C \leq T_{0h}$. For the first order transition, these temperatures are usually different. As the extent of the first order becomes stronger, the difference between these temperatures increases. For the second order phase transition, however, there is no difference between these temperatures. The phenomenon that governs that T_{0c} or T_{0h} is lower or higher than T_C is called the supercooling or the superheating, and usually, the supercooling is larger than the superheating. Theoretically, T_{0c} and T_{0h} are determined to be the temperatures where the energy barrier between the two phases disappears and only a single energy minimum remains.[4] Meanwhile, an application of the electric field also induces a sort of phase (or more appropriately here called a state) transition in the same manner as the thermal energy does to the structure.[5–7] This is because the electrical coupling aligns the polarization vectors in the chiral smectic liquid crystals. Hence the state of the liquid crystals transforms from a less-polar to a more-polar state by applying the electric field. Corresponding to the phenomenon of supercooling in the phase transitions driven by temperature, the hysteresis exists in the field induced states, that is, by increasing the field, the phase transition to a new state occurs at a larger electric field than that on decreasing the field. The hysteresis can well be explained by a similar model where the two energy minima and the energy barrier between these exist, as has been investigated by Qian and Taylor and others.[8–10]

Recently, it was reported by Song *et al.*[11] that the SmC^*-SmC_A^* phase transition occurs rather differently from the usual weakly first order phase transition predicted by the Landau–de Gennes theory. It was found that at this phase transition, the local energy minimum does not disappear, but the two energy minima and the energy barrier between these

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persist over the entire temperature range of the SmC_A^* phase. This means that the phase transition cannot occur intrinsically due to the energy barrier that does exist in between the two phases. However, the propagating solitary wave, which is driven by this energy barrier, can overcome it. As a result, the phase transition occurs despite the existence of a large energy barrier.[12,13] Consequently, the temperature range of only a few degrees is observed in supercooling for a thick cell, and the phase transition is found to be apparently weakly first order. On the contrary, the phase transition in a thin cell shows a large supercooling of several tens of degrees in temperature.[14] Thus, it is obvious that the supercooling phenomenon in this system is controlled by the surface confinement rather than by the free energy of the two minima and the energy barrier between these. The two energy minima and the barrier between the two are the essential parameters for the conventional supercooling model based on Landau–de Gennes theory. We have already suggested that the large supercooling may be due to the suppression of the propagating solitary waves by surfaces.[15] The first objective of this paper is to clarify the mechanism of the large supercooling in a thin cell through a clear experimental evidence being given for its existence. At the same time, the mechanism of hysteresis which is its counterpart phenomenon in the field induced switching of the antiferroelectric cells is also clarified.

The second objective is to clarify the field induced states and the dependence of their stability on the cell thickness. In chiral tilted smectic C phases, several subphases between the SmC_A^* and the SmC^* phases exist over a range of temperatures. Similarly, the field induced subphases (or states) exist on applying the electric field. By applying a large electric field to a nonpolar antiferroelectric phase, a polar ferroelectric phase is generated due to a reduction in the free energy of the system by the coupling of the polarization with the electric field as explained above. For intermediate fields, however, the subphases (or states) between the antiferroelectric and the ferroelectric phases are also observed. The intermediate states are quite similar to the temperature driven subphases observed between the SmC_A^* and the SmC^* phases. This phenomenon of the field induced states was extensively investigated, sequence found and a complete phase diagram was given by Shtykov *et al.*[6] More recently a direct confirmation of this phase diagram at two temperatures using resonant x rays was given by Jaradat *et al.*[16] In this paper, we observe an additional ferrielectric field induced phase, which is proven to be a four-layered structure through simulation of the experimental results. This was not observed in these previously cited works. Recently, Manna *et al.* reported that the stability of the temperature driven subphases is strongly influenced by the surface confinement.[14] Likewise, we can expect that the stability and the existence of the field induced states can also be influenced by the surface confinement and this effect is being investigated experimentally in this paper.

To summarize, we systematically investigate the phenomena of supercooling and of the hysteresis and the dependence of both temperature induced and the field induced
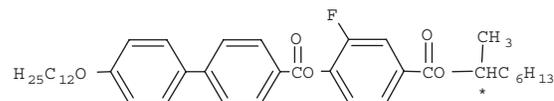

FIG. 1. Chemical structure of 12OF1M7 (Kingston Chemicals, Hull, UK). The phase sequence is SmC_A^* (77.2 °C) $SmC_A^*(1/3)$ (80.2 °C) $SmC_A^*(1/2)$ (82.8 °C) SmC^* (90.2 °C) SmC_α^* (91.8 °C) SmA^*.

states on the cell thickness and explore the similarities between the temperature induced phase transitions with the field induced states.

II. EXPERIMENT AND DISCUSSION

A. The supercooling in the SmC_A^*-SmC^* phase transition driven by temperature

The sample used is a prototype 12OF1M7 antiferroelectric liquid crystalline compound synthesized by Kingston Chemicals, Hull, UK. The chemical structure and the phase sequence are described in Fig. 1. This sample was also used for the studies of the temperature driven phases by Manna *et al.*[14] and the field induced subphases by Shtykov *et al.*[6] Note that 12OF1M7 has stable three-layered and four-layered subphases as are given in the caption in Fig. 1. Here, we use the nomenclature of the subphases introduced first by Isozaki *et al.*[17] They defined the fraction of ferroelectric order in a unit cell as $q_T=[F]/([A]+[F])$, where $[F]$ and $[A]$ are the ferroelectric and the antiferroelectric orderings in a unit cell. Since all of these phases are closely related to SmC_A^*, these are designated as $SmC_A^*(q_T)$. Thus $SmC_A^*(1/3)$ and $SmC_A^*(1/2)$ are the designations for three layer (also called SmC_γ^* or SmC_{FI1}^*) and four layer (called AF or SmC_{FI2}^*), respectively.

The experimental setup of the optical measurements made is shown in the upper part of Fig. 2. A cell of 1.6 µm thick with planar alignment of 12OF1M7 is used. Such a low cell thickness suppresses the subphases between the SmC_A^* and SmC^* phases. Hence, the direct phase transition between the SmC_A^* and SmC^* phases is observed.[14] The cell is placed between the two crossed polarizers and the rubbing direction of the two substrates made parallel to each other is fixed at an angle of 22.5° with the polarizer. In order to induce the collective motion of the liquid crystalline molecules, a very weak (0.03 V/µm) low frequency (30 Hz) square wave field is applied, and the ac part of the optical response is measured. The SmC^* phase shows a very sensitive response to the weak field due to the presence of a strong Goldstone mode. However, the SmC_A^* phase does not show any significant optical response as the macroscopic polarization is almost zero since the spontaneous polarization of each layer is canceled out by those of the adjacent layers. Therefore, there is a clear contrast in the optical response between the two SmC_A^* and SmC^* phases, as shown in Fig. 2. This method gives rather similar results to the dielectric response with a number of known advantages over the latter. The optical signal can be converted into the birefringence, and the experiments were carried out while recording, *in situ*, the texture of the cell mounted on the rotating stage of the polarizing microscope. For frequencies as low as 30 Hz of the applied
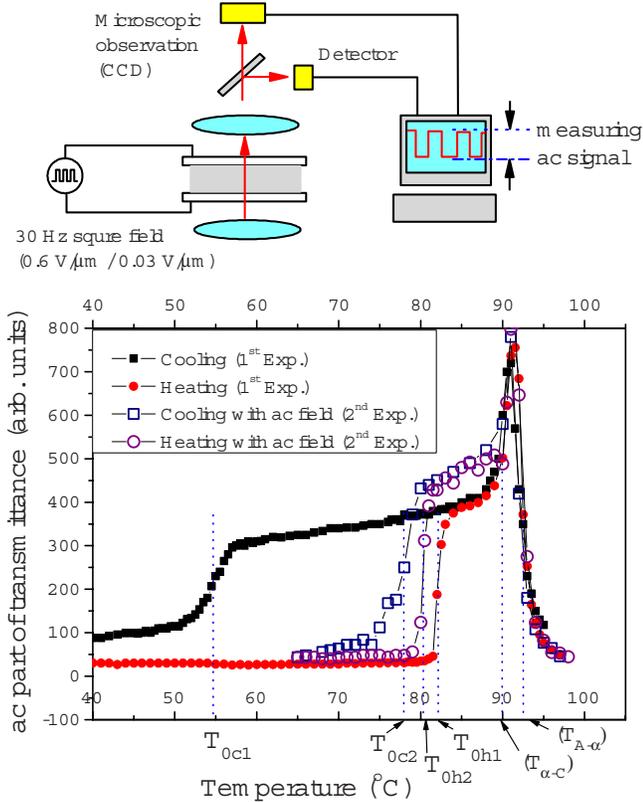
FIG. 2. (Color online) The ac part of the transmittance of the cell subjected to weak ac field (30 Hz, 0.03 V/μm) as a function of temperature (1.6 μm thick, 12OF1M7). T_{0c1} and T_{0h1} were obtained during cooling/heating without additional field, respectively, and T_{0c2} and T_{0h2} were obtained during cooling/heating with additional 0.6 V/μm field applied across the cell.
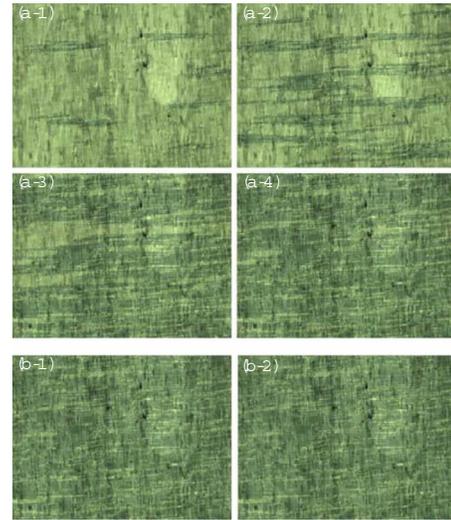

FIG. 3. (Color online) Microscopic images recorded during the cooling experiment without ac field in Fig. 2 (solid square shaped points) on a cell with thickness of 1.6 μm. (a-1), (a-2), (a-3), and (a-4) were recorded at temperatures of 58, 56, 54, and 50 °C, respectively. Images (b-1) and (b-2) were taken at 45 °C promptly and after 5 min had elapsed, respectively.

field, the ionic effect that causes serious noise in the dielectric measurements has negligible effect on the results of the optical experiment.

The first experiment was carried out using a weak signal field (30 Hz, 0.03 V/μm square wave) applied during the measurements for both cooling and heating. $T_{A-\alpha}$ and $T_{\alpha-C}$ in Fig. 2 are the SmA^*-SmC_α^* and the SmC_α^*-SmC^* phase transition temperatures, respectively (a detailed study for the SmC_α^* phase is reported in Refs. 6 and 7, T_{0c1} and T_{0h1} indicate the transition between SmC^* and SmC_A^* upon cooling and heating, respectively. The difference between T_{0c1} and T_{0h1} reaches 27 °C, indicating that the supercooling in the thin cell is very large. Nevertheless, the supercooling in a thick cell is just only a few degrees wide (data not shown here). To investigate the phenomena of large supercooling that might exist in a thin cell, we recorded microscopic observations during the phase transitions. Figure 3 shows the micrograph recorded during the first cooling experiment, where images (a-) 1, 2, 3, and 4 were taken at temperatures of 58, 56, 54, and 50 °C, respectively, while images (b-) 1 and 2 were taken at a temperature of 45 °C promptly after reaching this temperature as well as after an initial time of 5 min had elapsed since (b-1) was recorded. Image (a-1) was recorded at the starting point of the phase transition, where most of the area of the image is in the SmC^* phase and a few dark domains corresponding to SmC_A^* appear. As the temperature decreases, the dark domains for SmC_A^* enlarge at the expense of simultaneous reduction in those of SmC^*. It is

clearly shown that the phase transition occurs through the propagation of the domain boundary, that is through the propagation of solitary waves. This phenomenon and its mechanism has been explained by Song et al.[11,18] The observation of the propagating solitary wave indicates the existence of a nonzero energy barrier between the SmC_A^* and SmC^* phases. Conversely, the existence of an energy barrier confirms that the phase transition can occur only through the solitary wave. Here, we focus the investigation on the range of temperatures over which the propagating solitary waves are observed. In Ref. 11, the temperature induced solitary waves were reported to occur only over a narrow range of temperatures near the transition temperature in a thick cell. However, the solitary waves as shown in Fig. 3 in a thin cell appear over a wider range of temperatures in contrast to the case for a thick cell. Image (b) shows that SmC^* domains still persist even at a temperature of 45 °C, which is 13 °C lower than the transition initially recorded during cooling in this cell. This implies that the surface suppresses the propagation of the solitary wave and, as a consequence, the phase transition itself is suppressed as well. This observation provides a clear evidence for the mechanism of the large supercooling in a thin cell.

In the second experiment, a 30 Hz, 0.6 V/μm square wave field is applied to the cell both during cooling and heating. The field is greater than the field for the optical experiment but is still too weak to induce a phase transition on its own. Only during this optical experiment, the applied field was replaced with a weak probe field of (0.03 V/μm). The idea of this second experiment is to determine whether a moderate ac field during cooling/heating can induce the solitary wave to enable it to overcome the frictional effect of surfaces. Surprisingly, the extent of supercooling has significantly been reduced, as shown in Fig. 2, where T_{0c2} and T_{0h2} are the transition temperatures of cooling and heating, respectively. We note that the difference between T_{0c2} and T_{0h2}
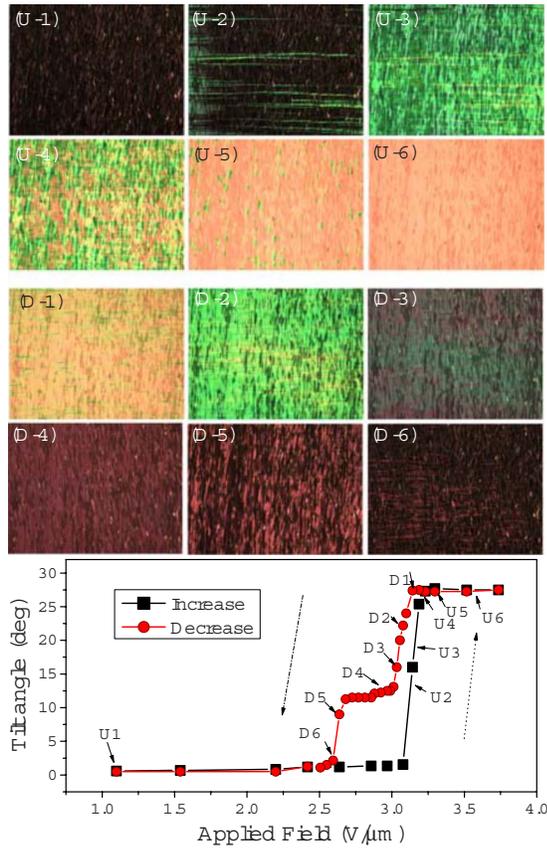
FIG. 4. (Color online) Micrographs taken under the polarizing microscope and the apparent tilt angle as a function of the amplitude of the applied field in homogeneously aligned (12OF1M7) in planar cell with thickness of 9 μm at a temperature of 50 °C. A 30 Hz square wave field is applied across the cell.

is quite small compared to that between T_{0c1} and T_{0h1}. This experimental result clearly shows that the 30 Hz moderate ac field (0.6 V/μm) applied during cooling/heating must enhance both the generation and the propagation of the solitary wave which can easily be understood intuitively. An ac field of 30 Hz makes the liquid crystalline molecules reorient as per the field, and the orientation apparently overcomes the frictional effects of surfaces. The experimental results also clearly indicate that the surface suppresses the generation and the propagation of the solitary wave, and thus, it produces the supercooling in the temperature induced AF-F transition. Moreover, it is quite interesting that the surface induced supercooling can be controlled both by the moderate ac field during cooling/heating and the cell thickness.

B. The field induced states

In this section, we report results on the field induced ferroelectric and ferrielectric states as a function of the cell thickness. Figure 4 shows the micrographs and the measured values of the apparent tilt angle by increasing the electric field across the cell. A 9 μm thick planar cell was prepared, and the apparent tilt angle measured as a function of the applied field using a 30 Hz square wave ac field at a temperature of 50 °C. On increasing the field, the apparent tilt angle suddenly increases which indicates the field induced antiferro- to ferroelectric (AF-F) phase transition occurring

and obtaining the ferroelectric state. By decreasing the field, a two step change in the apparent tilt angle is observed. For each step, the microscopic images are recorded as a function of the field and the tilt angle measured. As shown by the images for the steps observed, the transition between any two states occurs through the solitary wave propagation. That is, the transition from one to the other state is always accompanied by the propagation of the domain boundaries, while the transitions do not occur through a gradual change of color. Each state has its own specific color, indicating that each state has its own specific birefringence. The propagating solitary waves are observed two times on increasing the field, and three times on decreasing the field. The (U-1) micrograph having a dark color corresponds to the AF state, and (U-5) with orange color corresponds to the F state. On increasing the field, green colored domain (U-3) appears in the intermediate field. On decreasing the field, two substates appear between the F and AF states. One state is the green color (D-2) and the other is the brown color (D-4) state. The field induced ferrielectric states are rather stable on decreasing the field, and in particular (D-4) state is observed over a wider range of fields, as shown in the lower part of Fig. 4. (D-2) may correspond to the same state as the (U-3) state, as the color is the same. It was difficult to measure the accurate apparent tilt angle of (U-3) or of the (D-2) state because the state is rather unstable and some part of the cell has a different color than the rest, indicating an existence of the mixture of two states as shown in (U-3), (U-4), or (D-2). Thus, at least, two field induced states are observed here. One corresponds to (D-4) with brown color, which is found to be quite stable on decreasing the field, while it is not observed on increasing the field. The other is the (D-2) or the (U-3) state having green color. This is not stable over a wider temperature range but is observed both on increasing and decreasing the field. Note that the color being a signature of the birefringence of the cell changes suddenly through the growth of domains, and the change is not continuous. This indicates that the states with different colors correspond to the field induced states having different subperiodic structures.

As explained in Sec. I of the paper, the field induced phase transitions are induced by the energy of coupling between the electric field and the apparent polarization. Hence the field induced states having larger apparent spontaneous polarization are more stable for larger applied fields. Intuitively, we can assume the periods of field induced states as illustrated schematically in Fig. 5, suggested by Musevic and Skarabot[19] and more recently by Jaradat et al.[16] To identify the structure of the ferrielectric states, we carried out a simple simulation using the average refractive index approximation.[20] We assume that the field induced ferrielectric states are shown schematically in Fig. 5 as the periodic polar structures. When $\theta = 27°$, $n_e = 1.7$ and $n_o = 1.5$ and $\delta = 0$, the apparent tilt angle of the unwound $SmC_A^*(1/3)$ is calculated to be 15.2°, and those of the four-layered and five-layered structures are calculated as 19.7° and 21.7°, respectively. The apparent tilt angle depends rather weakly on the values of $(n_e - n_o)$ and the angle δ shown in Fig. 5. For example, when $(n_e - n_o)$ changes from 0.20 to 0.15 or from 0.20 to 0.25, the apparent tilt angle changes only by about 0.5°,
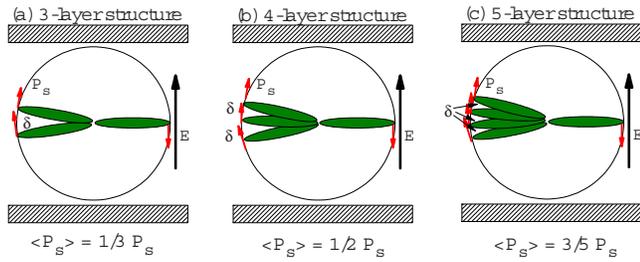
FIG. 5. (Color online) Schematics of the field induced three-layered, four-layered, and five-layered structures. The average apparent polarization $\langle P_s \rangle$ increases as the number of layers in a period increases. δ is the angle between the polarization vectors or the c directors of the closest adjacent layers.
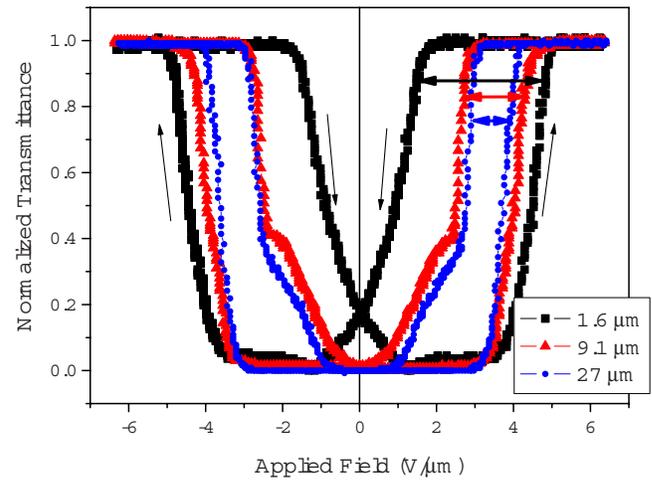

FIG. 6. (Color online) Transmittance as a function of the applied field for the various cell thicknesses of 12OF1M7. The experiment was carried out using the crossed polarizers and a 3 mHz triangular applied field. The thin arrows indicate the sense of the field, that is, on increasing or decreasing the field. The thick black, red, and blue arrows denote the hysteresis for each cell.

and when δ changes from $0°$ to $5°$, the apparent tilt angle changes by only $0.2°$ for the three-layered structure. On considering that the measured apparent tilt angles for (D-4) and (D-2) are approximately $12°$–$13°$, and $20°$–$21°$, respectively. (D-4) state is assigned to be the field induced three-layer structure and (D-2) and/or (U-3) state is assigned to the four-layered structure. A slight difference between the simulated values of ($15.2°$ and $19.2°$) against the measured ones ($12.5°$ and $20.5°$) for the two states might arise from the existence of a deformed helical structure for (D-4) (three-layered structure) and the coexistence of the ferrielectric and the ferroelectric states for (D-2) (four-layered structure). From our observations it is now clear that the field induced phase transition is also accompanied by the solitary wave propagation, and hence the basic mechanism of the field induced transition is the same as that of the temperature driven one. At the same time, the two field induced ferrielectric states are observed at intermediate fields between the antiferroelectric and the field induced ferroelectric states. Note that 12OF1M7 is found to have stable temperature induced three-layered and four-layered subphases that exist in the intermediate temperature range between the SmC_A^* and SmC^* phases.[6] The observation of a four-layered field induced state is additional to that reported by Jaradat *et al.*,[16] as they only reported the field induced three-layered structure. This result is based on a comparison of the observed and simulated tilt angles using the three- and four-layered structures. Our observations on texture also clearly show that there exist at least two apparent intermediate field induced states too. The sequence of the field induced ferrielectric states is found to be the same as the temperature driven phases. The field induced phase sequence is antiferroelectric-three-layered-four-layered-ferroelectric, and in the temperature driven (induced) phase sequence is SmC_A^*-$SmC_A^*(1/3)$ (three-layered)-$SmC_A^*(1/2)$ (four-layered)-SmC^*. Note that the field induced polar state with four layers is also seen indirectly from a change in the slope of the normalized coefficient for the pyroelectric response at ~ 0.5 seen in Fig. 4 curves (b), (c), and (d) of Ref. 6.

Now, we investigate the hysteresis phenomenon in greater detail as a function of the cell thickness. Figure 6 shows optical transmittance as a function of the applied field for various cell thicknesses at a temperature of 50 °C, where a 3 mHz triangular ac field was used. Since the frequency is very low, the electric field acts almost as a dc field. The

tristable switching and the hysteresis characteristics shown in the figure are well known to date.[21] However, it should be noted that the hysteresis for the 1.6 μm thick cell is the largest. As the cell thickness increases, the hysteresis decreases. Thus, the hysteresis depends strongly on the cell thickness as does the supercooling phenomenon, giving a similarity in the field induced phase transition with the phase transition induced by the thermal energy.

To investigate the switching characteristics further, similar tristable switching experiments were carried out by using 30 Hz square wave as a function of electric field, and the results are shown in Fig. 7. The apparent tilt angle of the cell is measured by increasing the amplitude of the ac field. The *abscissas* and the *ordinate* axes on the graphs of Fig. 7 indicate the amplitude of the 30 Hz ac electric field, and the apparent tilt angle of the smectic liquid crystalline material in the cell measured using the polarizing microscope, respectively. Figures 6 and 7 give a number of important results. Firstly, when we compare the switching curves for the 1.6 μm cell in both Figs. 6 and 7, a large hysteresis observed in Fig. 6 is not found in Fig. 7. We can see that the large hysteresis disappears by applying the moderate 30 Hz ac field. Actually, this phenomenon has a close analogy with that found for supercooling, where the extent of the supercooling in terms of the range of temperatures is also diminished by applying the ac field during cooling as explained in Sec. II A. This means that the hysteresis arises from the suppression of the propagating solitary waves by the surfaces. That is, by applying ac field, the solitary wave easily overcomes the frictional effects of surfaces, and the hysteresis disappears. Secondly, the critical field E_c required for the AF state to transit to the F state, is found to depend inversely on the cell thickness, *i.e.* the thinnest cell has the lowest E_c. Similarly, the phase transition temperature between the SmC^* and SmC_A^* phases has been found to decrease by decreasing the cell thickness. The latter can be explained by the model given by Manna *et al.* based on the fact that the sur-
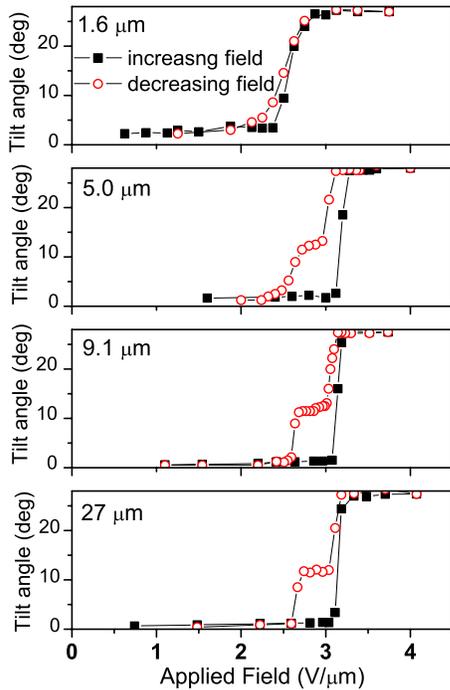
FIG. 7. (Color online) Apparent tilt angle as a function of the amplitude of the applied field for various cell thicknesses in planar alignment (12OF1M7) at 50 °C. A 30 Hz square wave field is applied.

face anchoring energy favors the synclinic ferroelectric state as opposed to the anticlinic antiferroelectric state[14] and the synclinic state continues to be stable with a reduction in temperature for lower cell thicknesses. Similarly, E_c in the electric switching experiment decreases by decreasing the cell thickness. This implies that the energy required for changing antiferroelectric to ferroelectric state is reduced by the effect due to surfaces as the synclinic state is more stable. Thirdly, the intermediate ferrielectric state/s become unstable as the cell thickness decreases, and eventually these disappear in a 1.6 μm cell. As explained in Sec. I of the paper, a very similar phenomenon has been reported for the temperature driven phases, where ferrielectric phases also become unstable as the cell thickness decreases.[14] A similar corresponding behavior is being observed for the field induced ferrielectric states here too. Confinement effects using dielectric spectroscopy on antiferroelectric and ferroelectric liquid crystals in micro- and nano-confinement have been studied qualitatively and reported in the literature.[22–24]

III. CONCLUSION

The mechanism and the correspondence between the dependence of the supercooling phenomenon on the cell thickness on the SmC^*-SmC_A^* phase transition and of the hysteresis in the field induced AF-F switching has been clarified. The phase transitions driven by temperature and electric field are shown to occur through the solitary wave propagation, and the supercooling and the hysteresis are caused by the suppression of the solitary wave by the surfaces. Therefore, both the extent of the supercooling and of the hysteresis

increase by decreasing the cell thickness. We also show that the supercooling and the hysteresis can be controlled by superimposing a moderate ac field. This helps in both the generation and a subsequent propagation of the solitary waves and it reduces both the extent of supercooling and of the hysteresis in the two cases. We also observe the two field induced ferrielectric states, which are proven to be the field induced three-layered and four-layered structures based on a comparison between the simulation and the experimental results of the apparent tilt angle for the two structures. The sequence of the field induced ferrielectric states is found to be the same as of the temperature induced phases. We also find a correspondence in the reduction of the phase transition temperature between SmC^* and SmC_A^* phases and of the critical field to induce SmC_A^* to SmC^* phase transition caused by the surface effects in a planarly aligned thin cell.

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