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Liquid Crystals

Publication details, including instructions for authors and subscription information:

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Online publication date: 19 May 2010

To cite this Article Kundu, S. , Ray, T. , Roy Corresponding author, S. K. and Dabrowski, R.(2004) 'Experimental characterization of hexatic smectic phases through electro-optic studies and dielectric relaxation spectroscopy', *Liquid Crystals*, 31: 1, 119 – 125

To link to this Article: DOI: 10.1080/02678290410001637158

URL: <http://dx.doi.org/10.1080/02678290410001637158>

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Experimental characterization of hexatic smectic phases through electro-optic studies and dielectric relaxation spectroscopy

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(Received 14 July 2003; accepted 15 September 2003)

The electro-optic and complex dielectric behaviour of an antiferroelectric liquid crystal 4-(1-methylheptyloxycarbonyl)phenyl 4'-(*n*-butanoyloxyprop-1-oxy)biphenyl-4-carboxylate, having chiral SmC_A^* and hexatic smectic phases, have been investigated. Complex dielectric permittivities were measured as a function of frequency, d.c. bias field and temperature. Spontaneous polarization was measured by the current reversal technique; tilt angle was measured under a polarizing microscope using a low frequency electric field. The electro-optic properties and dielectric behaviour of the material are compared with results obtained by DSC and polarizing optical microscopy. Dielectric relaxation processes in SmC_A^* and hexatic smectic phases were determined. The dielectric strength at the SmC_A^* to hexatic smectic phase transition is discussed in terms of coupling between the long range bond orientational order and smectic C director. It seems from the results of spontaneous polarization and dielectric relaxation spectroscopy that the material might possess an additional phase between the SmC_A^* and hexatic smectic I^* phases.

1. Introduction

Since the discovery of antiferroelectricity in the compound MHPOBC [1], a number of antiferroelectric liquid crystals (AFLCs) have been synthesized and investigated from the fundamental and applications points of view [2, 3]. Three subphases, the SmC_α , SmC_β and SmC_γ , between the paraelectric SmA and antiferroelectric SmA^* phases have been identified in MHPOBC [2] and distinguished by DSC and conoscopic observation [4–7]. At lower temperatures some tilted smectic liquid crystalline phases exhibit long range bond orientational order, but lack positional ordering; these are known as hexatic smectic phases, e.g. SmI , SmJ , SmF [8, 9]. The chiral tilted hexatic phases give rise to pyroelectric properties [10] and macroscopic spontaneous polarization in surface-stabilized cell structures [11]. Chandani *et al.* [2] reported a highly ordered SmI_A^* phase below the SmC_A^* phase in MHPOBC. Later Goodby *et al.* [12] also synthesized an antiferroelectric material similar to MHPOBC and identified a highly ordered hexatic phase below the SmC_A^* phase. It has

been earlier reported [13] that the SmI^* and SmI_A^* are the hexatic versions of SmC^* and SmC_A^* phases, respectively. Moreover, during cooling the SmI^* phase emerges below the SmI_A^* phase [14]. Selinger and Nelson proposed a theory for the hexatic to hexatic phase transition [15]. So far, few literature data are available on the electro-optic and dielectric behaviour of hexatic phases [16–19]. In the present paper we report the electro-optic and detailed dielectric studies of the SmC_A^* and chiral hexatic smectic phases of an AFLC material.

2. Experimental

The material under investigation, 4-(1-methylheptyloxycarbonyl)phenyl 4'-(*n*-butanoyloxyprop-1-oxy)biphenyl-4-carboxylate, has been synthesized by Drzewinski *et al.* by a synthetic procedure described elsewhere [20]. The structure and phase sequences of the material are shown below in figure 1; they depend strongly on the purity of the sample. The phase sequence and transition temperatures were checked by differential scanning calorimetry (DSC) and polarizing optical microscopy (POM). The DSC scan of the material, as observed by Dabrowski, is shown in figure 2; by POM

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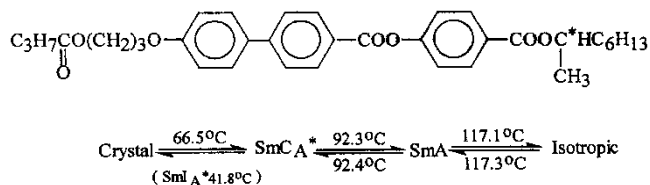


Figure 1. Structure and phase sequence of the test compound.

we have observed a slight change of texture at 54°C in addition to that observed by the DSC scan.

The sample was aligned by applying a square wave voltage of frequency around 2 Hz to 15 MHz from a home-made linear voltage amplifier and a HP 33120A function generator. At the time of alignment the sample was being cooled at a very slow rate ($\sim 0.2^{\circ}\text{C min}^{-1}$). The dielectric measurements in the frequency range 10 Hz to 13 MHz were recorded using a computer controlled impedance analyzer (HP 4192A). Measurements of spontaneous polarization (P_s) monitored the total induced polarization when the sample was switched to a saturated state. The measurements were carried out using a planar aligned cell by applying a low frequency triangular wave; spontaneous polarization was determined by a straight forward current peak integration method.

3. Results and discussion

3.1. Electro-optic properties

Switching current measurements were made during cooling in the SmC_A^* phase as well as in the highly ordered monotropic SmI^* phase. Polarization response currents in these phases at different temperatures are presented in figure 3.

In the SmC_A^* phase two current peaks were observed at 4 Hz as well as at a higher frequency (30 Hz),

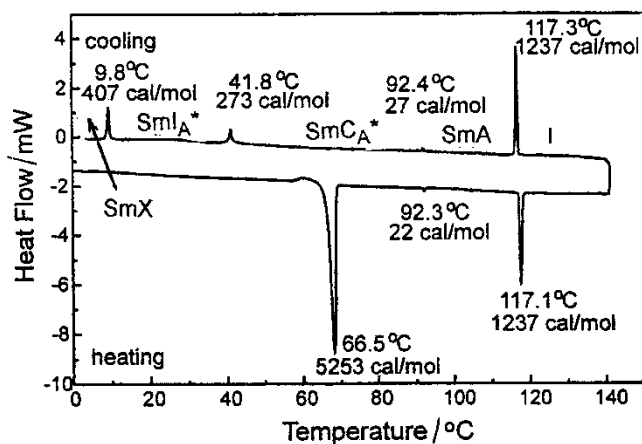


Figure 2. DSC curve of the pure sample.

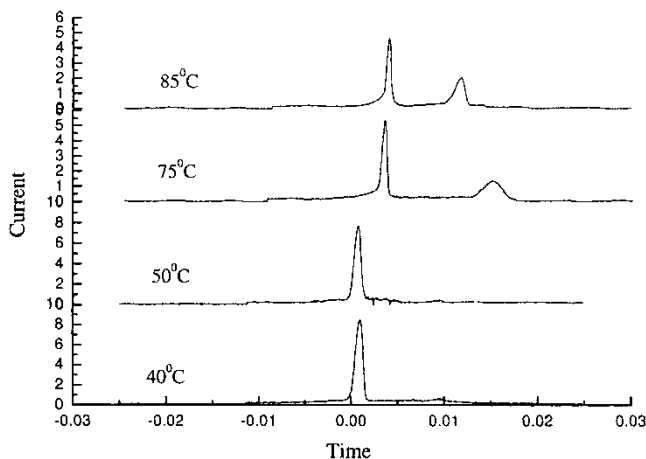


Figure 3. Polarization response current peaks obtained on cooling in the SmC_A^* and SmI^* phases.

indicating three stable states in this particular phase. The two peaks are well separated at the middle of the SmC_A^* phase, then they merge to a single peak at a supercooled temperature. The change in the orientational molecular arrangement in tristable switching takes place from one of the ferroelectric states to the other via an antiferroelectric structure [21, 22].

In the supercooled SmC_A^* phase, as well as in the SmI^* phase, the switching characteristics were changed from tristable to bistable where two current peaks merge to a broader single peak. This generally indicates that the anchoring effect between the molecules and the cell surface lessen in this particular molecular arrangement. The spontaneous polarization of this material was determined by the current peak integration method. Temperature dependence of the spontaneous polarization (P_s) and tilt angle are shown in figures 4 and 5, respectively. According to the extended mean-field

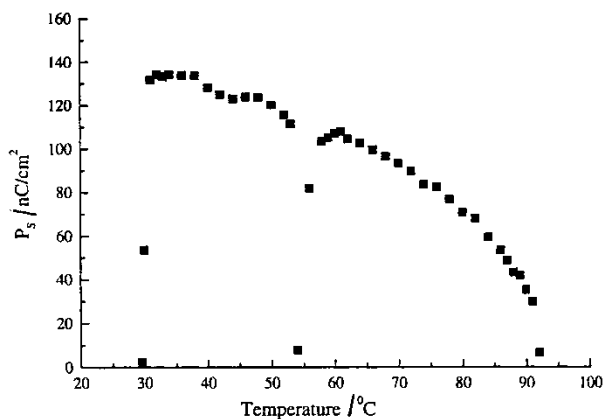


Figure 4. The change of spontaneous polarization with temperature.

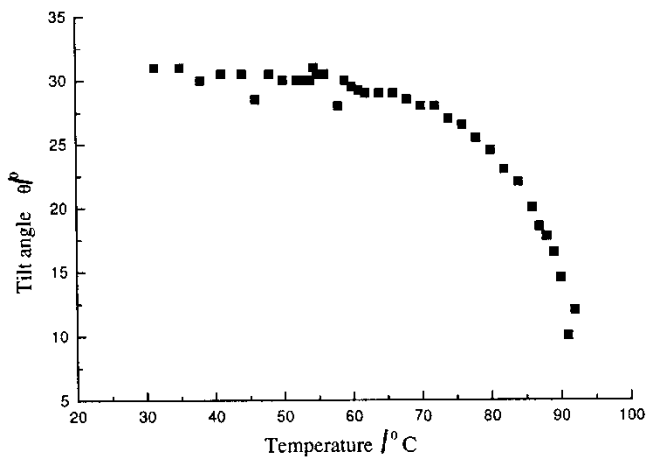


Figure 5. The change of tilt angle with change of temperature.

theory [23] of second order phase transition, the spontaneous polarization \mathbf{P}_s should obey the square root law

$$\mathbf{P}_s = \mathbf{P}_0(T_C - T)^\beta \quad (1)$$

with critical exponent $\beta = 0.5$.

It has been observed that the spontaneous polarization of the material during cooling, continuously increases from the SmA–SmC_A^{*} transition temperature (T_C) down to 30°C in the supercooled SmI_A^{*} phase, as shown in figure 4. Two discontinuities, however, are observed in the temperature dependence of the polarization curve (figure 4), a sharp fall of \mathbf{P}_s at 54°C and a faster rise at 43°C. The temperature dependence of the tilt angle also showed two discontinuities at 54 and 43°C. This may be due to molecular rearrangements connected with the formation of new phases at the above-mentioned temperatures. The DSC curve, however, shows a single peak around 43°C (figure 2) below the SmC_A^{*} phase, which was assigned as the SmI_A^{*} phase [20]. The critical exponent determined after fitting the temperature dependence of the \mathbf{P}_s data in the range T_C to 54°C to the above expression (1) is found to be ≈ 0.3 , which is much less than the theoretically predicted value for a second order phase transition [23]. It was earlier observed by Bahr and Heppke [24] that in case of a first order phase transition, tilt and spontaneous polarization show a discontinuous decrease at the phase transition; whereas in case of a second order phase transition tilt and \mathbf{P}_s decrease continuously to zero when approaching the transition temperature from the low temperature side. Our experimental results of \mathbf{P}_s and tilt also showed some discontinuity at the phase transition. Moreover, after fitting the temperature dependence of the \mathbf{P}_s data, the critical exponent is found to be 0.3, indicating that the

SmC_A^{*}–SmA phase transition may be of first order type. The \mathbf{P}_s data for the whole temperature range could not be fitted to the above expression due to discontinuities in the temperature dependence of the polarization curve.

3.2. Dielectric properties

To ascertain the phase sequence below the supercooled SmC_A^{*} phase and to understand the dielectric behaviour of the different phases, particularly of hexatic smectic phases, we have studied the molecular dynamics of the material by dielectric relaxation spectroscopy. The complex dielectric constant of the material was calculated in the frequency range 10 Hz to 13 MHz as a function of temperature and bias electric field. The experiments were performed during a cooling cycle since the material possesses monotropic phases in the supercooled state, apart from the SmC_A^{*} phase. Experimental results were analysed using a generalized Cole–Cole expression, and characteristic dielectric parameters such as relaxation frequency and dielectric strength were obtained by fitting the data in the Cole–Cole function for single and double relaxation processes:

$$\varepsilon_\perp^* = \varepsilon_{\perp\infty} + \frac{\Delta\varepsilon_1}{1 + (i\omega\tau_1)^{1-\alpha_1}} + \frac{\Delta\varepsilon_2}{1 + (i\omega\tau_2)^{1-\alpha_2}} \quad (2)$$

where ε^* is the complex dielectric constant, ε_∞ is the dielectric constant in the high frequency limit, and $\Delta\varepsilon$, τ and α are the dielectric increment, relaxation time of the various modes and distribution parameter.

Experimental data of the frequency dependence of the imaginary part ε'' of the complex dielectric constant is fitted with equation (2) into single and double relaxation modes in this particular case. The temperature dependence of the dielectric strength $\Delta\varepsilon$ and relaxation frequency $f = 1/(2\pi\tau_1)$ of the modes obtained by Cole–Cole fittings for this compound are shown in figure 6 and 7. In the vicinity of the SmA→SmC_A^{*} phase transition a strong soft mode was observed in the paraelectric SmA phase due to fluctuation of the tilt angle θ . The temperature dependence of the critical frequency and inverse of the dielectric strength of the soft mode (figure 8) obey the Curie-Weiss law [25].

Just inside the SmC_A^{*} phase, one dielectric mode was observed in a narrow temperature region and the mode was softened near T_C , i.e. the relaxation frequency of the mode converges and the dielectric strength diverges while approaching T_C (figures 6 and 7): a ferroelectric soft mode-like behaviour. A similar behaviour of the mode just below T_C in the SmC_A^{*} phase was observed earlier [26, 27] when a SmC_A^{*} phase is preceded by a SmA phase; the authors assigned it as ferroelectric soft

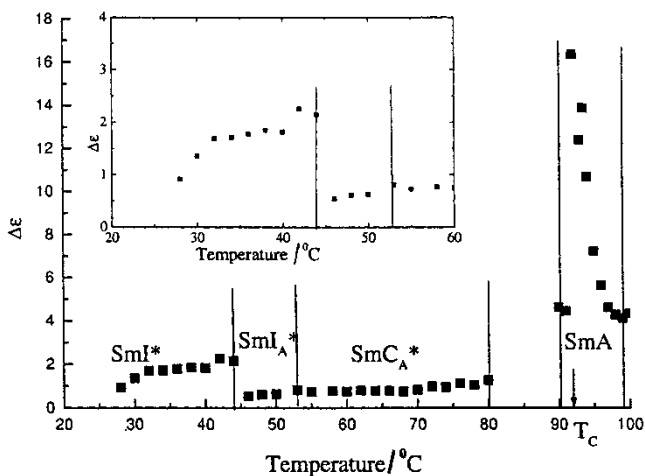


Figure 6. Dielectric strength ($\Delta\epsilon$) vs. temperature curve for the whole temperature range. The inset shows the variation of $\Delta\epsilon$ in the range 28–60°C in enlarged form, and shows the discontinuities of $\Delta\epsilon$ at 43 and 54°C.

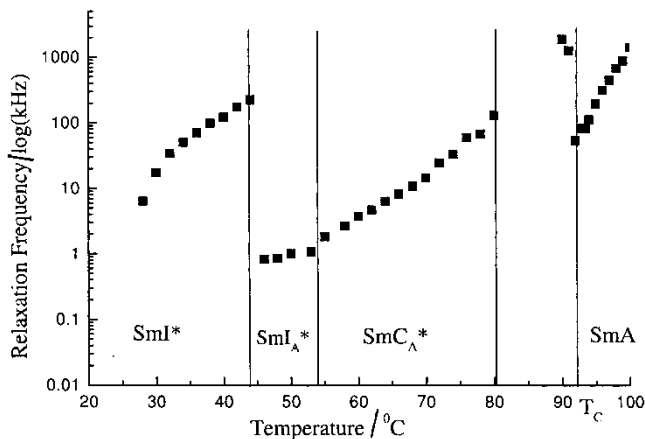


Figure 7. Behaviour of relaxation frequency with temperature in all the phases without application of a d.c. bias field.

mode in antiferroelectric ordering. Apart from soft mode, other dielectric modes in the SmC_A^* phase arise due to coupling between the net polarization with the oscillating electric field. Due to the helical superstructure the angle between the directors in adjacent layers in a SmC_A^* phase is not exactly 180° [28]. Hence the local polarization between adjacent layers makes some angle and is not cancelled completely; a small net polarization thus appears. Surprisingly we could observe no dielectric mode within a few degrees Kelvin below T_C in the temperature range 90–80°C in the SmC_A^* phase. The absence of any dielectric mode was also observed earlier [29] in a short temperature range below T_C in the SmC_A^* phase.

The non-appearance of the mode a little below the $\text{SmA} - \text{SmC}_A^*$ transition temperature within the temperature interval 90–80°C can only be explained if we consider that the helical pitch in that region becomes very large, and net polarization between the adjacent layers is assumed to be negligibly small. The dielectric modes usually observed in the SmC_A^* phase arise due to collective in-phase and anti-phase fluctuations of the molecules in the adjacent layers [30], as a result of coupling between the oscillating field and the net polarization. Sometimes it is termed a non-cancellation mode [31]. If the polarization becomes too small, the permittivity contribution to this mode may be undetectable with dielectric relaxation spectroscopy. At lower temperatures, however, the pitch becomes finite and net polarization becomes significant. Due to this net polarization a single relaxation mode was however observed below 80°C in the SmC_A^* phase and it survived down to a hexatic smectic phase at supercooled temperatures.

Both the dielectric strength and relaxation frequency of the mode in the SmC_A^* and hexatic phases decrease with decrease of temperature (figures 6 and 7). There are, however, discontinuous jumps of relaxation frequency and dielectric strength at 54 and 43°C. In the temperature range 54–43°C there is a slower rate of decrease of dielectric strength and relaxation frequency. It seems that the dielectric mode in the SmC_A^* phase has been extended to 43°C in the supercooled state after a discontinuous decrease at 54°C, as shown in the inset of figure 6. A similar dependence of relaxation frequency on decrease of temperature in a SmI_A^* phase, followed by a SmC_A^* phase of an antiferroelectric liquid crystal, was earlier observed by Glogorova *et al.* [18].

3.3. Dielectric behaviour under bias electric field

Dielectric studies under a bias electric field is a good technique for identifying the different phases by comparing with other experimental results. By using a 5 V d.c. bias soft mode dielectric response, a paraelectric SmA phase was observed over a larger temperature interval, as shown in figure 8. Due to the electroclinic effect [32] an induced tilt as well as \mathbf{P}_s is developed in the SmA phase and the coupling of the induced tilt with the oscillating field is responsible for the appearance of a soft mode over a larger temperature interval. By applying the same bias electric field in the SmC_A^* phase a dielectric mode was found in the temperature range 90–80°C, which was otherwise absent.

The bias voltage can give a small net polarization which will respond to the a.c. field resulting the appearance of the dielectric mode in the range 90–80°C,

which was not observed without the bias field. Dielectric studies were also made under a bias electric field at 35°C, to identify the phase below 43°C. It was observed that the relaxation frequency of the mode at 35°C diverges and dielectric strength decreases with the application of the bias electric field (figure 9). This is a characteristic Goldstone mode-like behaviour generally observed in the SmC* phase, due to unwinding of the helix. The SmC*-like dielectric behaviour suggests that the phase below 43°C should be the hexatic version of the SmC* phase which is known as the SmI* phase [13].

The single antiferroelectric mode observed in the SmC_A* phase near the T_C of the presently studied material has a close resemblance to the mode no. 6 [27]

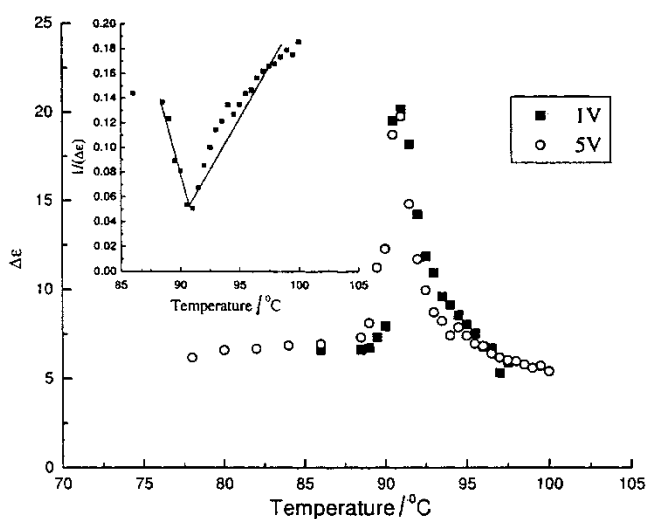


Figure 8. The behaviour of $\Delta\epsilon$ with temperature on application of 1V and 5V d.c. bias fields. Inset figure shows the $(1/\Delta\epsilon)$ vs. temperature curve that follows the Curie-Weiss law.

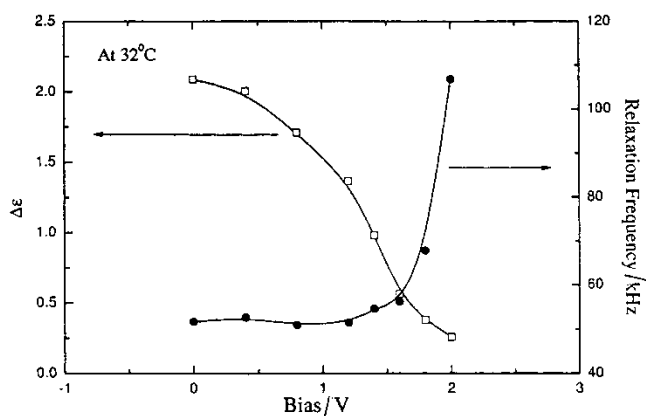


Figure 9. Dielectric strength ($\Delta\epsilon$) and relaxation frequency behaviour with change of d.c. bias field in the SmI* phase.

which was assigned as a ferroelectric soft mode in antiferroelectric ordering. But the mode observed away from T_C at lower temperatures behaves like a non-cancellation mode (NCM) [31], arising due to antiferroelectricity; at higher temperatures the NCM coincides with the antiferroelectric soft mode near T_C . The curves of temperature dependence of spontaneous polarization and tilt angle, as well as the dielectric spectrum, showed two discontinuities at 54°C and 43°C below the monotropic SmC_A* phase.

It has also been observed that the relaxation frequency in the temperature range 90–43°C can be fitted to a bilinear curve with distinctly different slopes (figure 10). The activation energy was determined from an Arrhenius plot (figure 10) and is found to be 20.5 kcal mol⁻¹ in the temperature range 90–54°C, and 4 kcal mol⁻¹ in the range 54–43°C. The experimental results indicate that there may be two successive changes of phases, one at 54°C and another at 43°C. The change of phase around 43°C was also detected at 41.8°C by DSC scan. This slight difference is due to a gradient between the observed temperature and the actual temperature inside the cell. The phase change at around 54°C was not observed by DSC studies. The change of enthalpy at the 54°C phase change may be so small that the heat flow peak is too small to be observable by DSC (figure 2). The discontinuities in the results of the spontaneous polarization, tilt and dielectric permittivity at the lower temperature end of the SmC_A* phase suggest that there may be a phase change at 54°C, apart from that observed at 43°C, but a.c. calorimetric and X-ray studies are needed for confirmation. The phase sequence as observed by dielectric relaxation spectroscopy and from the

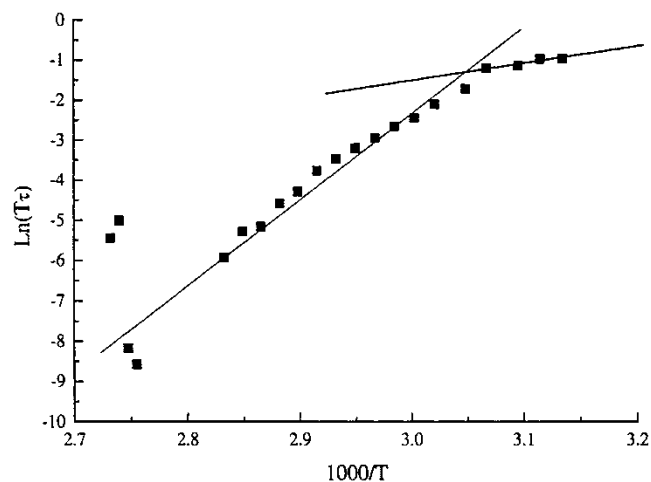


Figure 10. Arrhenius plotting from which a change in activation energy with change of phase can be seen.

measurement of spontaneous polarization and tilt angle has been shown to be as follows:

SmX \leftarrow phase II \leftarrow 43°C phase I \leftarrow 54°C SmC $_A^*$.

It was discussed above how the temperature dependence of the dielectric behaviour of the mode both in the SmC $_A^*$ phase and in phase I are very similar. Moreover phase I emerges below the SmC $_A^*$ phase. The SmC $_A^*$ -like dielectric behaviour of phase I suggests that it should be the hexatic version of the SmC $_A^*$ phase, since it was reported earlier by Fukuda *et al.* [21] that the SmI $_A^*$ phase is a hexatic version of the SmC $_A^*$ phase and appears below the SmC $_A^*$ phase. Therefore the phase that appears below 54°C should be the SmI $_A^*$ phase. The other phase II that appears below 43°C may be suggested as a SmI* phase since this phase normally appears below the SmI $_A^*$ phase [21]. The dielectric properties of hexatic phases may be explained in terms of a collective reorientation of the \hat{c} director in hexatic smectic phases, which are influenced by coupling between the spontaneous polarization and the electric field.

In the SmC $_A^*$ phase the tilt direction is intrinsically locked in a certain position on the local tilt cone. The forces stabilizing the tilt direction can be large in comparison with the weak elastic force and surface interaction [19]. The goldstone mode thus becomes undetectable; only the ferroelectric soft mode in antiferroelectric ordering is observed. In the SmC $_A^*$ – SmI $_A^*$ transition a long range bond orientational order is assumed to be established in addition, and due to this additional ordering the collective tilt fluctuation of the director is significantly restricted, which is reflected as a discontinuous decrease of dielectric strength and relaxation frequency in the dielectric spectra at 54°C, as shown in figures 6 and 7. On decreasing the temperature in the SmI $_A^*$ phase, if the coupling between the bond orientational order and the local tilt direction is assumed to be stronger, then the tilt fluctuation of the collective director mode (antiferroelectric soft mode) should gradually decrease on cooling; this was observed in the curve for temperature dependence of dielectric strength (figure 6). On approaching the SmI $_A^*$ \rightarrow SmI* phase transition on cooling, the long range bond orientational order remains unchanged but the direction of local tilt changes from layer to layer, since the SmI* phase is assumed to take a SmC*-like structure with a long range bond orientational order [13]. Thus the coupling between the bond orientational order and local tilt director weakens, allowing the reorientation of \hat{c} director, i.e. a collective director phase fluctuation. Contributions of the collective director phase fluctuation to the complex dielectric permittivity in tilted smectic phases are designated as Goldstone mode. The Goldstone mode contribution

in the SmI* phase is found to be much smaller than in SmC* phase. The lower dielectric strength Goldstone mode in the SmI* phase might be due to the following reasons.

1. The SmI* phase is a hexatic version of the SmC* phase with additional long range bond orientational ordering; the additional ordering restricts the collective phase fluctuation of the c -director due to its coupling with the bond orientational order.
2. The higher visco-elastic force at lower temperatures also restricts the phase fluctuation of the c -director, i.e. the Goldstone mode contribution in the SmI* phase.

4. Conclusion

The temperature dependence of both the spontaneous polarization and tilt angle curves, as well as the dielectric strength, showed two discontinuities below the supercooled SmC $_A^*$ phase, corresponding to two successive phase transitions before reaching the crystal-like SmX phase. From the results of dielectric relaxation spectroscopy the phases may be identified as SmI $_A^*$ and SmI* in order of decreasing temperature. The phase transition at 54°C could not be detected by DSC studies, which showed a phase transition only at 41.8°C, below the SmC $_A^*$ phase, before reaching the SmX phase. The phase was therefore assigned as SmI $_A^*$ in the DSC spectrum. Collective director reorientation in the vicinity of the SmC* \rightarrow SmI $_A^*$ transition is explained by considering the coupling between the long range bond orientational order and tilt order. The results suggest that the coupling between long range bond ordering and the c -director is strong in the vicinity of the SmC $_A^*$ \rightarrow SmI $_A^*$ phase transition, but the bond-tilt coupling is rather weak in the hexatic SmI $_A^*$ \rightarrow hexatic SmI* phase transition.

The authors wish to thank Professor N. V. Madhusudana for fruitful discussions.

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