Cross-Over Temperature for Distinct Low-Frequency Modes in Ferro Electric Liquid Crystalline Phases

S. Sreehari Sastry, L Tanuj Kumar
Department of Physics,
Acharya Nagarjuna University,
Nagarjunanagar -522510, India.

D. M. Potukuchi
Department of physics,
Jawaharlal Nehru Technological University-Kakinada,
Kakinada-533003, India.

Ha Sie Tiong
Department of Chemical Science,
Faculty of Science, Universiti Tunku Abdul Rahman,
Jalan University, Bandar Barat, 31900
Kampar, Perak, Malaysia

***Abstract*— Phase transitions and relaxation behaviour are investigated in the low frequency region (1Hz-1MHz) for two ferroelectric liquid crystal compounds viz., (S)-(-)-2-Methylbutyl4´-(4´´-n-alkanoyloxybenzoyloxy) biphenyl-4-carboxylates (S-MB-nB-BC), for n=16 and 18, which exhibit enantiotropic Smectic A and Smectic C* phases. Phase transition temperatures are determined from LF dielectric method that are in agreement with the results obtained from concurrent microscopic textural (POM) and DSC studies, Dielectric relaxation behaviour is also investigated for SmecticA and SmecticC* phases. Dielectric dispersion has inferred the temperature variation for relaxation frequency, dielectric strength, loss maxima and degrees of freedom. Arrhenius shift has indicated the activation energies in higher range. Dielectric loss maxima in SmC* has exhibited a cross-over temperature that suggested the presence of two distinct modes of relaxation in SmC* phases. LF Relaxation in SmC* has behaved in a similar way of Curie -Weiss law in ferro electrics.**

Keywords---Ferro electric liquid crystals; Smectic A & C* phases; dielectric parameters; cross over temperature; Goldstone mode.

1. INTRODUCTION

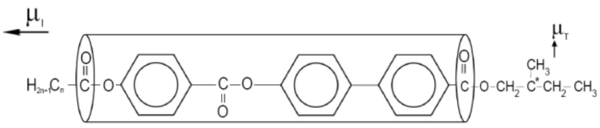
Thermotropic liquid crystal (LC) phase structures [1] such as Nematic and Smectics owe to their rich electro optic response [2,3]. Existence of ferroelectric (FE) response in tilted liquid crystals is reported [4,5] firstly by Meyer in 1975. LC molecules, those possessing chiral centre in their tilted phase structures like in SmC*, Sm I* and Sm F* phases are found[6,7] to exhibit ferroelectric response. LC SmC* phases have been attracted greater attention for studies due to their least viscous state and readily alignable stablized structure surface [8] geometry. Potential applications for [9] fast-switching, high-contrast, large viewing angle devices are noteworthy. The molecular director possesses helicoidal structures in the layered SmC*, and a constant tilt angle is maintained [10,11]. The coupling between applied electric field at given molecular tilt and its electro-clinic response[12,13] in these ferroelectric liquid crystalline materials become interesting[7] on observing fast electro-optic response at sub microsecond. Investigations on low frequency (LF) dielectric response [14,15] in LCs will help to get important information that can be used in making of electro optic devices. Determining the properties like relaxation frequency (f_R), strength $\Delta\varepsilon$, distribution parameter-α and activation energy (E_a) in LCs corresponding to the LF relaxation behaviour would help to optimize their utility. Although LF dielectric response is initially reported [16] in nematics (N) with sluggish millisecond response, the investigations on FELC SmC* phases has resulted in useful information [17, 18] with regard to collective and independent molecular processes. The reported gold stone mode and soft mode, Curie Wiess behaviour and other orientational response modes in SmC* phase in low frequency (in few Hz to KHz) region have attracted the attention for studies on fundamental reorientation in the Quasi-2D crystalline soft condensed matter systems. The information for the reorientation response would be optimized during their utility in devices. Keeping in view the requirement of the ambient FELC SmC* phase structures, being exhibited [20] by LCs preferably for esteric moieties, structures, the effort is made have to bring out the dielectric dispersion properties in FELC materials S-MB-16B-BC and S-MB-18B-BC.

II. MATERIALS AND EXPERIMENTS

a. Materials

The compounds (S)-(-)-2-Methyl butyl4´-(4´´-n-alkanoyloxy benzoyloxy) biphenyl-4-carboxylates for *n*= 16 and 18) mesogens [22] were synthesized.

The molecular structure for the chosen FELC compounds (S)-(-)-2-Methylbutyl 4′-(4′′-*n*-alkanoyloxybenzoyloxy) biphenyl-4-carboxylates (where *n*= 16 and 18) in conjunction of chiral centre (*) presented in Template -1.



Template 1: Molecular structure of S-MB-*n*B-BC, where *n*=16,18

b. *Measurements*

Measurements for textures and phase transition temperatures [1] were carried out by using polarizing optical microscope (POM), coupled with hot stage of Meopta DRU 3 model (Meopta Global Manufacturers, Hauppauge, NY, USA) and Canon EOS Digital REBEL XS/ EOS1000D to record textural images at given POM crossed polar configuration. The FELC compounds [22] were filled through capillary action method in LC1 ITO coated liquid crystal cells of 6 µm space that are received from Instec, (USA). The temperature and frequency variation of dielectric response were at a low frequency range for 1Hz to 1 MHz measured by LCR Meter(Model PSM1700 , Newton's4thLtd., Loughborough.UK). The sample was initially heated to isotropic state and kept it until thermal equilibrium attained. Through capacitance and loss factor, the dielectric response was measured against an input 1Vp-p oscillating signal. The accuracy for dielectric constant and loss values is 1% and 2%, respectively. The accuracy for temperature variation is ± 0.1^{o}C. Phase transition temperatures were determined from the studies to temperature variations on POM textures, capacitance C(ω) and loss factor TanΔ(ω). The off centred dielectric dispersion behaviour [23, 24] was investigated based on temperature and frequency variations of capacitance and loss factor.

c. Computational Details

The dielectric dispersion was measured for variation in capacitance C (T) and loss factor Tan Δ(T) at specified different temperatures for different LC phases during cooling scan. The observed variation of C(ω) and TanΔ(ω) is presented in figure-1 and figure-2. In the wake of the temperature invariant capacitance being exhibited by the empty cell (~38.99pF) for the frequency range 1Hz – 1 MHz, the relative permittivity of 100 KHz ε′ (or ε_r) is estimated by

$\varepsilon^*(\omega) = \varepsilon'(\omega) - j\,\varepsilon''(\omega)$ --------------------------------- (1)

$\varepsilon_r(\omega) = \varepsilon'(\omega) = C(\omega)/(38.99$------------------------------- (2)

where C (ω) is the observed capacitance of the LC cell at a specific temperature in any LC phase corresponding to the frequency , ω= 2πf of the input ac signal.

The dielectric loss ε″(ω) is estimated by

$\varepsilon''(\omega) = \varepsilon_r(\omega) * \tan\Delta(\omega)$ ---------------------------------- (3)

where TanΔ is the observed loss factor exhibited by the LC phase structure at a specified temperature corresponding to

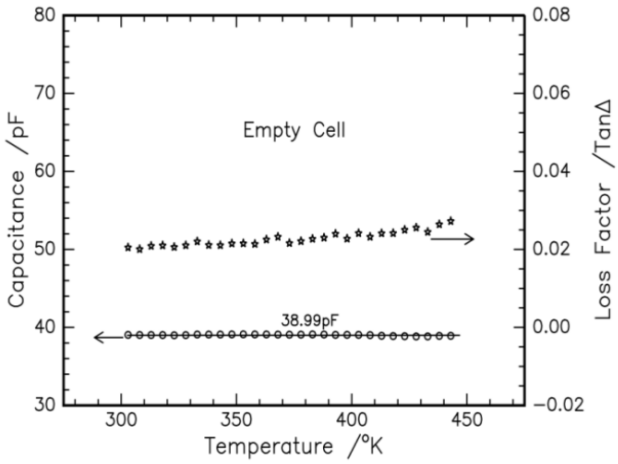


Figure-1: Variation of capacitance and loss factor of empty cell with temperature

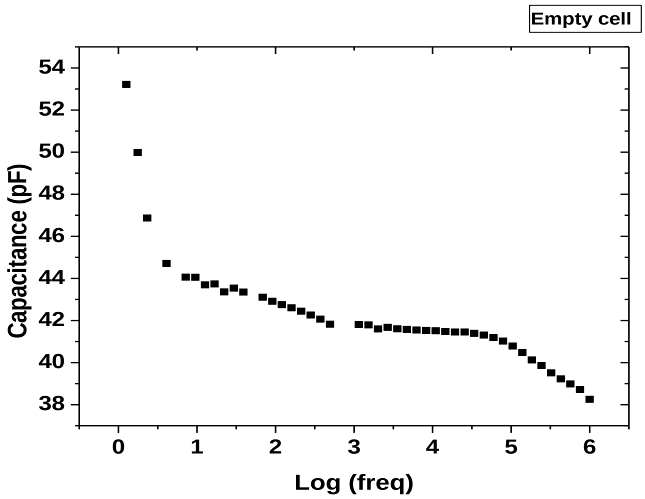


Figure-2: Variation of capacitance for empty cell with frequency

the frequency of input ac signal. The dielectric dispersion is given [14-15] by

$\varepsilon^*(\omega) = \varepsilon_\infty + \{(\Delta\varepsilon)/(1 + [j\omega\tau]^{1-\alpha})\}$ ---------------------- (4)

where, $\Delta\varepsilon = [\varepsilon_o - \varepsilon_\infty]$ is the dielectric strength, estimated by extrapolating ε″ on to the ε_r axis through Cole Cole plots

τ, the relaxation time given by $1/f_R$, where f_R corresponding to the frequency at which ε″ exhibits maximum value

ω for 2πf where f is the frequency applied ac E field, α for the distribution parameter reflecting upon the degrees of freedom exhibited by the phase in any LC phase structure.

III. RESULTS AND DISCUSSION

i) Textures and Phase Transition Temperatures:

Enantiotropic phases and transitions temperatures exhibited by the liquid crystalline SmA and SmC* phases are initially determined by POM. The POM textures shown by (S)-(-)-2-Methylbutyl4´-(4´´-n-alkanoyloxybenzoyloxy) biphenyl-4-carboxylates(S-MB-*n*B-BC for *n* = 16 and 18) for SmA and SmC* phases are shaped in focal conic fan (plate-1) and arced focal conic (plate-2) respectively. The

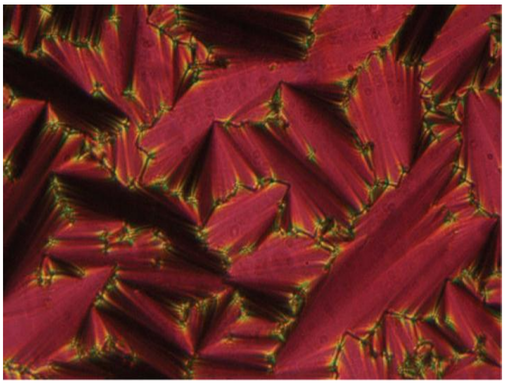


Plate 1: Focal conic fan texture of SmA phase for S-MB-18B-BC at 380.5 K

transition temperatures T_{IA} and T_{AC*} are in agreement with POM and DSC [21] data reports. The observed phase transition temperatures determined by POM (in heating and cooling cycles) are presented in Table-1. The thermal span for heating scan periods of LC phases has differed slightly from those observed for cooling scan. However, the hierarchy in occurrence has remained invariant.

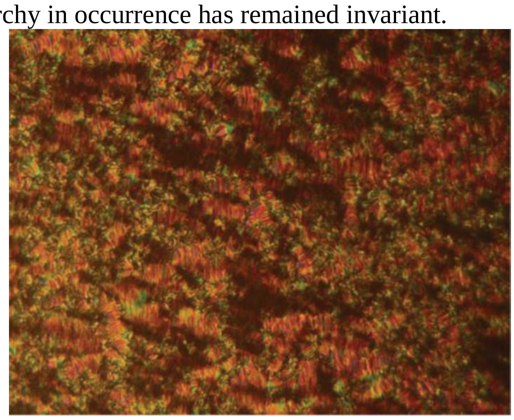


Plate 2: Arced focal conic texture of SmC* phase for S-MB-16B-BC at 350.5 K

Table1: Phase transition temperatures T_c by POM, DSC, LF Dielectric methods for S-MB-*n*B-BC for *n*= 16 and 18

n=	Method		Data of Phases, Transition Temperatures (T_c in K)	Ref
16	POM	H	Cr—337→SmC*—385.4 → SmA—426.2→ Iso	present
		C	Cr_1←—325.4—Cr_2←333.7—SmC* ←375.4—SmA←425.8—Iso	
	POM /DSC	H	Cr—337→SmC*—385.4 → SmA—426.2→ Iso	[21]
		C	Cr_1←—325.4—Cr_2←333.7—SmC* ←375.4—SmA←425.8—Iso	
	LF Dielectric	C	Cr_1←—325.4—Cr_2←337.0—SmC* ←379.5—SmA←424.8—Iso	present
18	POM	H	Cr—346.3→SmC*—385 → SmA—425→ Iso	present
		C	Cr_1←—335.9—Cr_2←341.5—SmC* ←377.5—SmA←424.8—Iso	
	POM /DSC	H	Cr—346.3→SmC*—385 → SmA—425→ Iso	[21]
		C	Cr_1←—335.9—Cr_2←341.5—SmC* ←377.5—SmA←424.8—Iso	
	LF Dielectric	C	Cr_1←—335.9—Cr_2←341.5—SmC* ←379.0—SmA←422.7—Iso	present

H: HEATING, C: COOLING

ii) Phase Transitions by Temperature variation of Dielectric constant $\varepsilon_r(T)$ and Loss Factor $Tan\Delta(T)$:

The S-MB-*n*B-BC cell is connected to the LCR meter which is operated at fixed 100 kHz frequency and triggered by 1Vp-p oscillating signal. The observed temperature variations in dielectric constant $\varepsilon_r(T)$ and loss factor $Tan\Delta(T)$ exhibited for cooling run of FELC samples are presented in figure-3 and figure-4 for *n*=16 and 18, respectively.

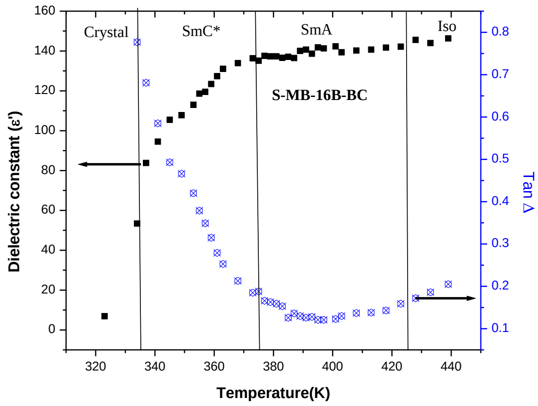


Figure 3 Temperature variations of dielectric constant and loss factor Tan Δ(ω) for S-MB-16B-BC

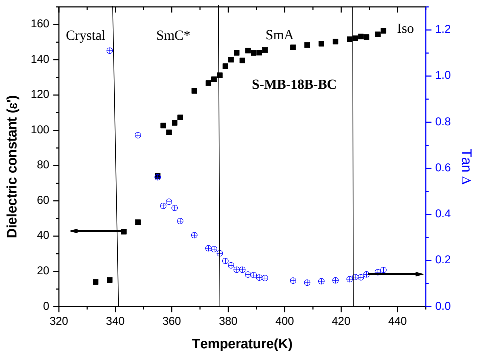


Figure - 4 Temperature variations of dielectric constant and loss factor Tan Δ(ω)for S-MB-18B-BC.

The ε_r in cooling run has exhibited peaks, where as TanΔ (T) displayed dips in the vicinity of phase transition. The increase of ε_r(T) with the decrease of temperature has indicated the increasing dipole correlation due to that LC phase structures grew with higher order. As the reason of change in ε_r(T)and TanΔ(T) is apparently marginal, the derivative curve is drawn for the observed temperature variations of ε_r(T) in figure-5 and figure-6 for *n*= 16 and 18, respectively. The data for transition temperatures determined from LF dielectric and microscopic observation (Table-1) are in agreement with the reported [22] data.

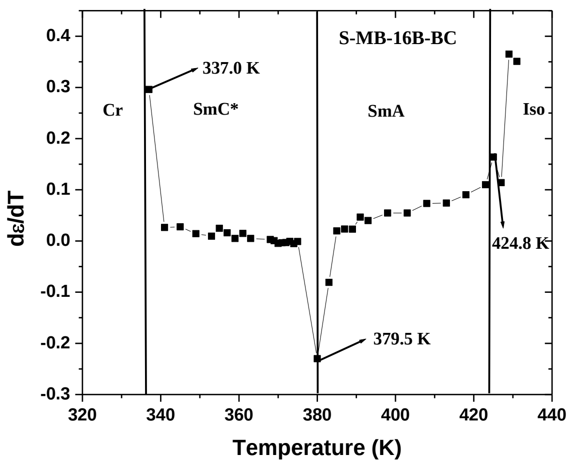


Figure – 5 Temperature variation of differential dielectric constant ε′ (T) for S-MB-16B-BC

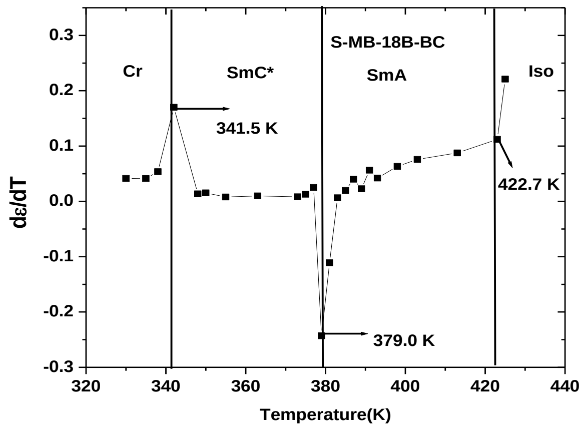


Figure - 6 Temperature variation of differential dielectric constant ε′ (T) for S-MB-18B-BC

The AC* transition noticed here which was not reported [22] for any enthalpy in DSC studies, so that it is considered as a second order transition. However, an anomalous behavior is observed to ε_r(T)and dε_r(T)/dt across AC* transition. In view of these, the LF dielectric method is a capable method to detect second [25] order transitions also.

iii) Dielectric Dispersions:

The LF dielectric dispersion i.e., frequency variation of capacitance C (ω) , loss factor TanΔ(ω) are recorded at different temperatures for SmA and SmC* phases exhibited by FELCs. The dielectric constant (i.e., $\varepsilon_r = \varepsilon'$) at different frequencies is estimated using equation-2. From the data for capacitance variation ε′(ω) of the FELC compounds are presented in figures -7, -8, -9 and-10 at specified temperatures for SmA and SmC* phases. Both compounds have shown decreasing trend of ε′ with increasing frequency.

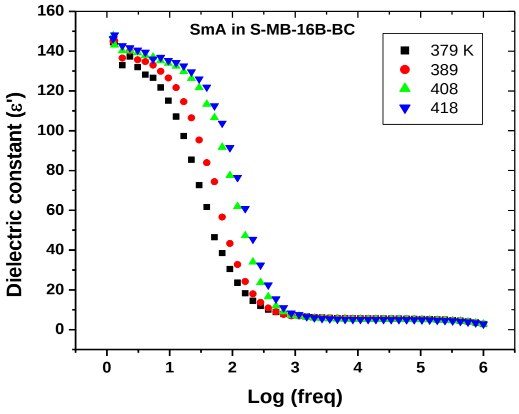


Figure – 7 Frequency variation of dielectric constant ε′ (ω) in SmA of S-MB-16B-B

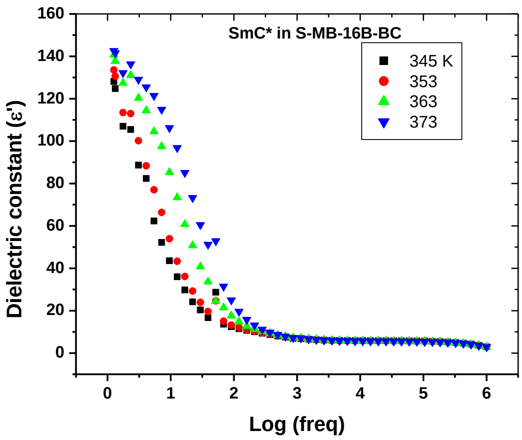


Figure – 8 Frequency variation of dielectric constant ε′ (ω) in SmC* of S-MB-16B-BC

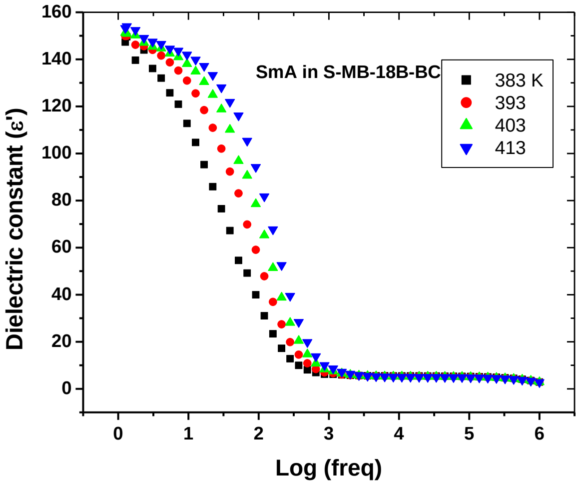


Figure – 9 Frequency variation of dielectric constant ε′ (ω) in SmA of S-MB-18B-BC

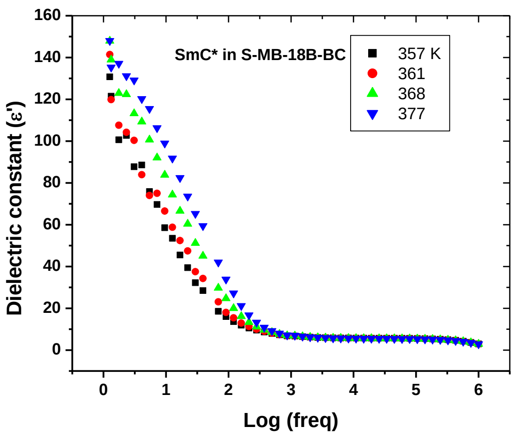


Figure – 10 Frequency variation of dielectric constant ε′ (ω) in SmC* of S-MB-18B-BC

The steep fall in ε′ is noticed in lower frequency region, and it is marginal at higher frequencies. Steep fall of ε′ at lower frequency (Few kHz) range would indicate the predominant response of LC molecules during the dipolar orientational process. It is also noticed that dielectric constant ε′ is increased with increasing temperatures in LC phases. At higher frequency, the values of dielectric constant are lower, that might reflect the lesser contributions of LC molecules dipole moment to the orientational mechanism. The higher value of ε_r at lower frequencies is attributed to the response of large polarization in LC compound.

From the observed data of capacitance C and loss factor TanΔ the dielectric loss ε′′(ω) exhibited by the FELC in the SmA and SmC* LC phases are estimated by the equation-3 in view of the response of empty cell. The loss spectrum ε′′(ω) exhibited by FELC in their SmA and SmC* phases at specified temperatures is presented in figures -11, -12 and -13, -14 for n=16 and 18, respectively.

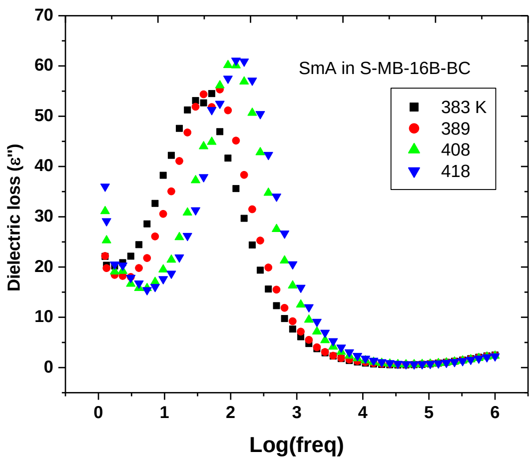


Figure – 11 Frequency variation of dielectric loss ε′′ (ω) in SmA of S-MB-16B-BC

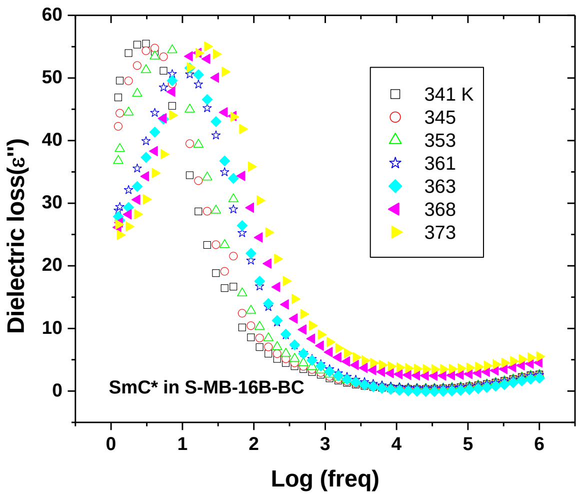


Figure – 12 Frequency variation of dielectric loss ε′′ (ω) in SmC* of S-MB-16B-BC

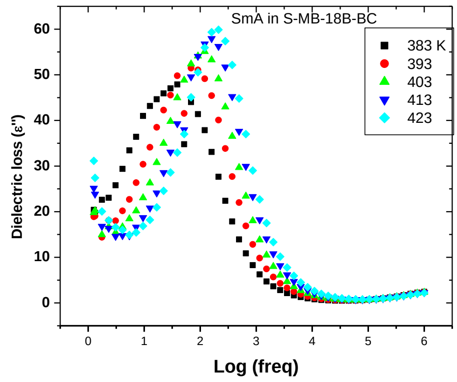


Figure – 13 Frequency variation of dielectric loss ε″ (ω) in SmA of S-MB-18B-BC

iv) Activation Energy:

Variation of f_R(T) and (ε″) $_{max}$ in SmA and SmC* phases exhibited by FELCs is presented in Table-2. The data for f_R have shown decrease trend with the decreasing temperature in both SmA and SmC* phases. This trend is attributed to increasing viscosity on cooling LC phase structure. The value of f_R in good agreement with the reported values for other FELC compounds [17-19, 25]. From data of f_R(T), the Arrhenius plots are drawn (figure -15 and -16) for SmA and SmC* phases.

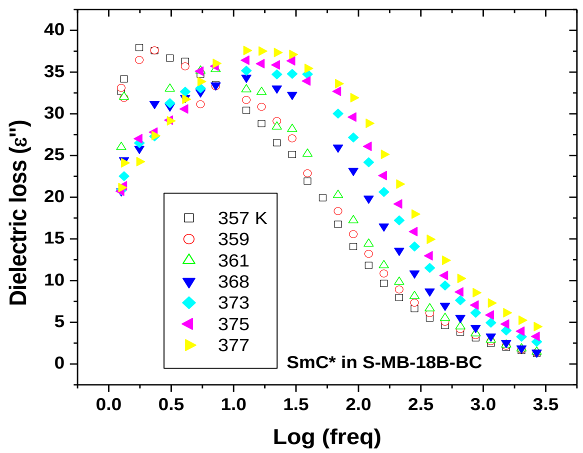


Figure – 14 Frequency variation of dielectric lossε″ (ω) in SmC* of S-MB-18B-BC

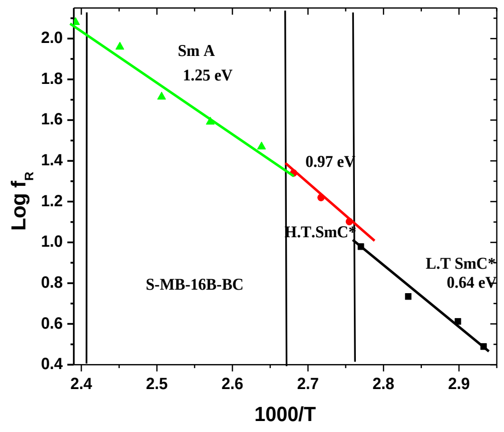


Figure-15 Reduced Temperature plots in different phases of S-MB-16B-BC

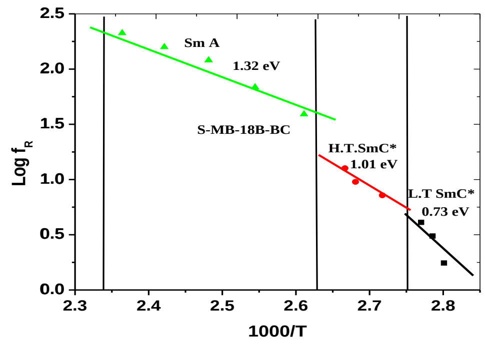


Figure-16 Reduced Temperature plots in different phases of S-MB-18B-BC

The estimated activation energy E_a (Table-2) for LC SmA and SmC* phases are recorded relatively with lower values that would reflect lower potential barrier for the process to re-orient the tilted SmC* phases, than the orthogonal SmA phase structures.

Table 2: Data of dielectric parameters in SmA and SmC* phases of S-MB *n*B-BC for *n*= 16and18

n	Phase	Tempe-rature (K)	Relaxation frequency (f_R) (Hz)	Dielectric strength Δε	Max. loss $(ε'')_{max}$	Distribution Parameter *α*
16		418	2.080	113.05	61.96	0.174
	SmA **[1.25 eV]**	408	1.959	113.60	52.04	0.226
		399	1.714	115.239	50.63	0.261
		389	1.591	116.86	47.08	0.296
		379	1.469	117.95	44.25	0.349
		373	1.340	140.36	56.24	0.191
	SmC*(HT) **[0.97 eV]]**	368	1.220	144.65	55.34	0.209
		363	1.102	152.53	54.62	0.244
		361	0.979	144.30	52.28	0.139
	SmC*(LT) **[0.64**	353	0.734	147.95	55.62	0.157

	eV]					
		345	0.612	150.29	57.29	0.191
		341	0.489	152.38	59.01	0.209
		423	2.326	120.03	49.39	0.209
18	SmA [1.32 eV]	413	2.200	121.33	47.48	0.226
		403	2.080	121.98	46.21	0.244
		393	1.836	123.28	44.29	0.261
		383	1.591	124.59	39.82	0.279
		377	1.224	152.38	47.29	0.08
	SmC*(HT) [1.01eV]	375	1.102	154.47	46.28	0.122
		373	0.979	157.26	43.89	0.157
		368	0.857	160.73	42.67	0.174
		361	0.612	162.82	41.89	0.244
	SmC*(LT) [0.73 eV]	359	0.489	165.6	44.86	0.261
		357	0.244	168.99	45.85	0.296

An overview of data of E_a in SmA and SmC* phases has revealed the higher magnitudes than the reported [26] values. The higher value of Ea is attributed to the additional contribution of transverse dipole moment (μ_t) to the tilted chiral structure.

v) Cross-Over of temperature for LF modes in SmC phase:*

The temperature variation of loss maximum $(\varepsilon'')_{max}$ in SmC* phase presented in figure-12 and-14 has indicated that it is accompanied by a reversal of trend (figure -17) $\varepsilon''_{max}(T)$ at a particular temperature.

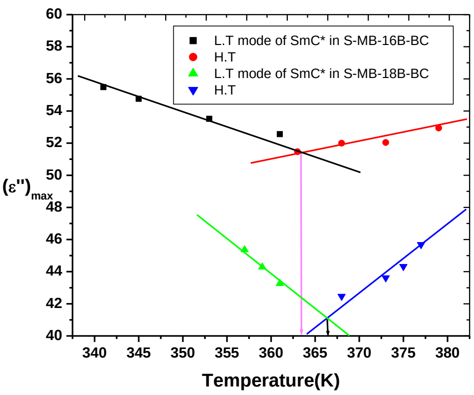


Figure – 17 Variation of Dielectric maxima with temperature for S-MB-16B-BC and S-MB-18B-BC

For a decreasing temperature $(\varepsilon'')_{max}$ the loss decreased initially, but started to increase from a characteristic temperature T_{CO}., Since the $(\varepsilon'')_{max}$ reflected the loss of energy corresponding to a dielectric medium, the reversal trend is related to the contribution made from a different and distinct modes in SmC*phases. As a result,a possible distinction should be present to differentiate the high temperature (HT) mode (figure -18 and -19) from low temperature mode (LT) in the SmC* phase.

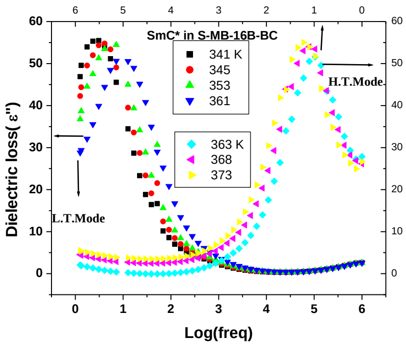
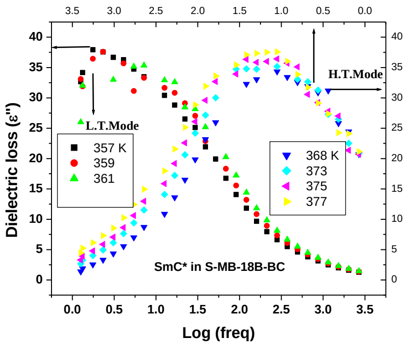


Figure – 18 Cross over temperature modes in SmC* phase for S-MB-16B-BC



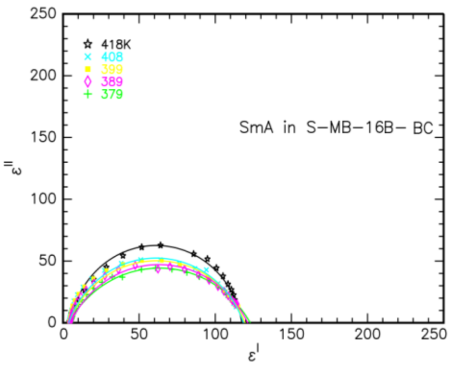
Figure–19 Cross over temperature modes in SmC* phase for S-MB-18B-BC

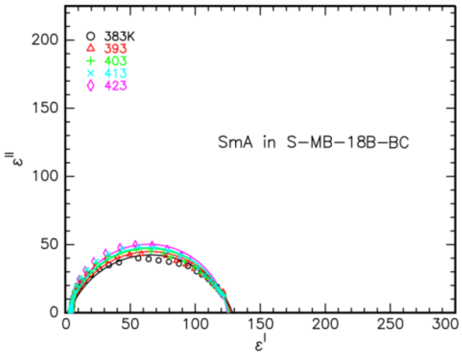
The mode cross- over temperature in SmC* lower for lower end-chain compound FELC i.e., for n=16. The observation of T_{CO} also has implied a lower energy configuration during the reorientation process corresponding to distinct modes

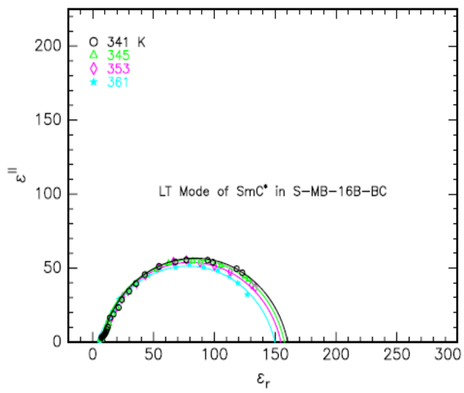
vi) Cole Cole plots:

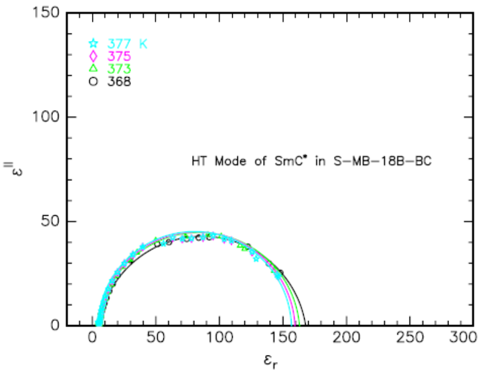
The dispersions of, $\varepsilon'(\omega)$ and $\varepsilon''(\omega)$ are seemed to be asymmetric about ω, where in $\varepsilon''(\omega)$ has shown maximum in both SmA and SmC* phases. The fall of ε'' with frequency is not symmetrical about the $(\varepsilon'')_{max}$. Hence, such off-centered dielectric dispersion [24] exhibited by the SmA and SmC* phases. In order to analyze the observed low frequency (LF) dielectric response and also the distinct (time scale wise) mode of relaxation behavior, the data for ε'' and ε_r , LF dielectric dispersion to all LC phases are received by computation using equation-4. The derived dielectric dispersions are presented as Cole-Cole plots in figure -20 (a) to -20 (f).

The data for LF dielectric parameters corresponding to the distinct modes, such as relaxation frequency f_R, loss maximum ε''_{max}, dielectric strength $\Delta\varepsilon$, distribution parameter-α and activation energy E_a are estimated from Cole-Cole plots drawn for S-MB-*n*B-BC for *n*=16 and -18 and presented in Table-2. In order to determine $\Delta\varepsilon$, ε_o and ε_∞ from Cole-Cole plots the value of ε'' is extrapolated on to the ε_r axis. The extrapolated point towards the lower frequency side is read as ε_o and for higher frequency side as ε_∞. An overview of the Cole-Cole plots for LF relaxations has shown a greater temperature ε_r shift in the ε_o end for all phases. However its shift towards high frequency ε_∞ end, remains constant almost. The observed large temperature ε_o shift informed the relative dielectric susceptibility of FELC in the LF (KHz) region.









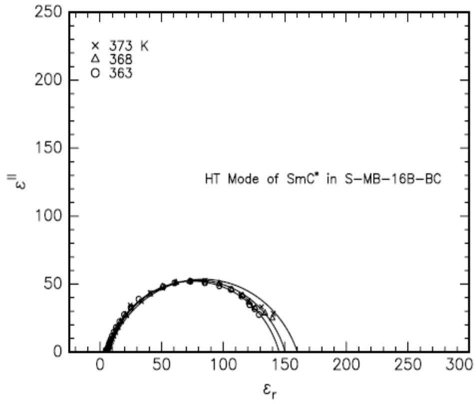


Figure – 20 Cole- Cole plots for SmA and SmC* phases in S-MB-16B-BC and S-MB-18B-BC

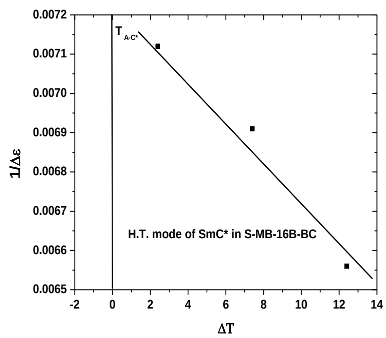
The trend for α-parameters (Table-2) of SmA and SmC* phases as shown the increasing degree of freedom for decreasing temperatures. Increase in α-parameter values are due to increasing restrictions on rotational freedom for longitudinal dipole moment (μ_l).

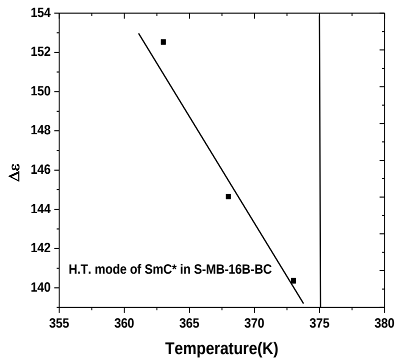
vii) Curie Weiss behavior in FELC in SmC phase:*

The data for temperature variation (Table-2) of LF frequency dielectric strength Δε pertaining to the HT LF mode in the SmC* phase are fitted to the equation given by

$\Delta\varepsilon \;\alpha\; 1/(\Delta T)^{\gamma}$ -- (5)

The data for Δε fitted (Figure – 20 and 21) is shown with the solid line. The estimated exponential value (γ) is 1.0846±0.001 for *n*=16, and for *n*=18 it is1.04±0.001. The value γ - exponent is in agreement with reported [27] the expected Curie wises behavior for FE SmC* phase.





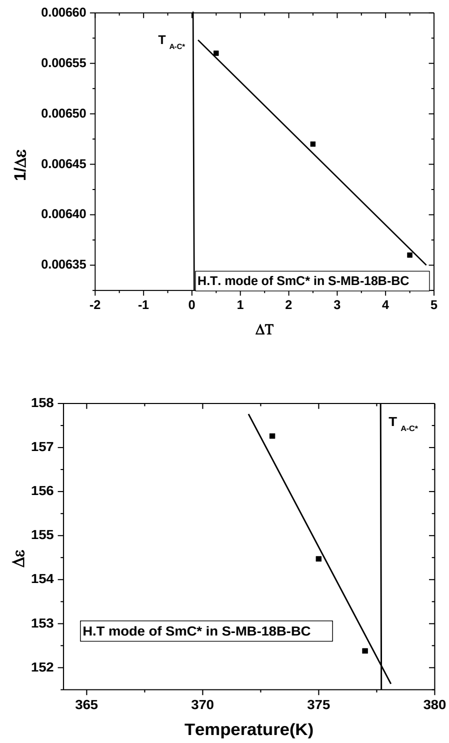


Figure – 21 Curie wises behavior exhibited by dielectric increment relevant to H.T mode of SmC* phase for S-MB-16B-BC and S-MB-18B-BC

IV. CONCLUSIONS

The following conclusion are drawn from the present LF dielectric study

1. LF Dielectric studies can be confidently used to determine the phase transitions temperature in FELCs for involving second order transitions.
2. The temperature variation in f_R (shifting to the lower frequency side) has inferred high activation energy in SmA and SmC* phases that suggested for greater strength of the potential barrier.
3. The Dielectric loss spectrum accompanied by a cross-over temperature for SmC* phase would helps to resolve distinct modes.
4. For decreased temperatures, the value α increased that has reflected upon the relatively more dipole moment in the LC phase.
5. The increase dielectric strength (Δε) in SmA and SmC* phases for the decreased temperature, would suggest that ε_r is more susceptible to lower frequency field.

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