High Speed Response Time of Nematic Liquid Crystal Mixtures
for LCD Monitor and TV Applications

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Abstract

The most important parameter for TV application of LCD is a fast switching time for the display of moving image. To achieve faster switching time, the novel LC single materials with large dielectric anisotropies (16~20), high clearing temperatures (195.5 ~ 237.4 °C), broad nematic ranges (up to 169.9 °C) and high birefringence (0.254~0.2200) were developed. KUR-series LC mixtures blended these single materials having significantly higher clearing temperatures and dielectric anisotropy values compared with conventional LC mixture. Especially, their clearing temperatures are 10~30 °C higher than their host mixture. These LC mixtures showing about 10ms of high-speed switching time in TV/Monitor of TFT LCD, is short enough to be addressed in a single time frame of 60Hz (16.7 ms). The threshold voltage V_{th} was low enough to operate at a driving voltage of 5 V. The VHR values were found to be high enough for TFT-LCD in wide temperature range. Our novel LC mixtures are suitable materials for the inclusion in to LC mixtures for TV application of TN-LCD

1. Introduction

The response time of a liquid crystal (LC) device affects human perception of the displayed images. For a desktop computer, the displayed images are usually static and the pace of information update is relatively slow. However, for a TFT-LCD TV, the motion pictures changes rapidly. If the LC switching speed is too slow, the images will be blurred. The improvement of switching time is therefore a key technical feature for the TFT-LCD TV. It is highly desirable to have the LC device operate at the true video rate, i.e. less than 16.7 ms regardless of ambient temperature.

Nematic liquid crystals offer several attractive features for display applications, including a low operation voltage, analog gray scales, simple molecular alignment, abundant material selection, and a large cell gap tolerance. Two major drawbacks that demand continuous improvements are the switching time and the viewing angle. Material effects (viscosity, elastic constant, and birefringence), molecular alignment, temperature, voltage and cell gap all play significant roles in determining the LC response time. Dielectric and elastic constants jointly determine the operating voltage, whereas the rotational viscosity and the elastic constant determine the switching time. From the molecular structure viewpoint, the macroscopic properties of phase transition behavior, birefringence, dielectric constant and viscosity are interrelated. If may not be too difficult optimize one parameter may not be too difficult, but to optimize all the parameters simultaneously is considered to be very difficult.

In order to improve panel brightness of LCD TV, stronger backlights are used which can lead to a slightly higher operating temperature of the panel. Therefore, development of LC mixtures has to be focused on high

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clearing temperature of 90 °C. It is important to develop higher clearing points LC mixtures without sacrificing the viscosity value.

Super fluoro-substituted materials (SFM) have very high stability, which has a great advantage in the field of TFT-LCD applications. For this reason, many types of SFM were practically used and their properties investigated. However, they have low dielectric and optical anisotropy, which deter the development of high-speed response and low power consumption. On the other hand, a large variety of liquid crystal compounds with an isothiocyanate terminal group has been widely investigated they impart advantageous properties to LCDs applications [1]. For example, their physical properties are low melting point, wide nematic range, modest viscosity, high dielectric and optical anisotropy.

Recently, we developed a new class of LC by compounds substituting a fluoro-isothiocyanate terminal group, which have features of coexistence of high birefringence and low viscosity. The chemical structure of those novel LC compounds is depicted in Fig. 1.
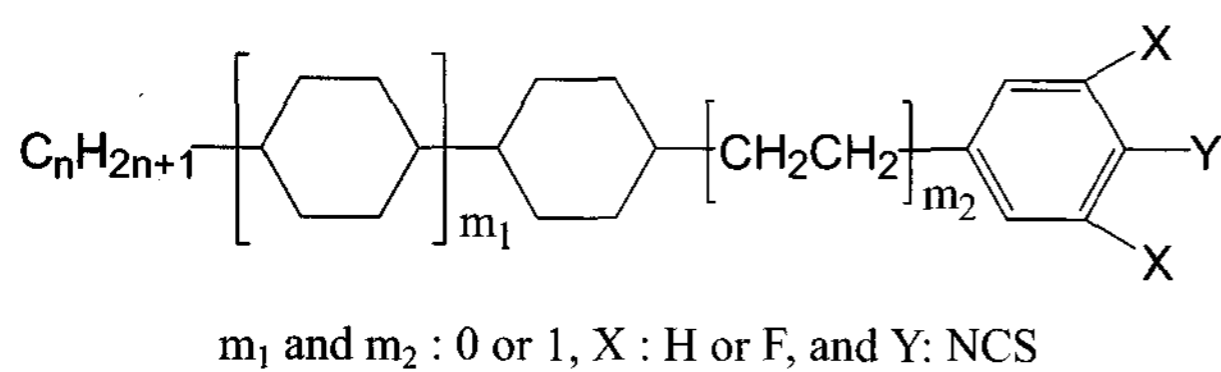

m_1 and m_2 : 0 or 1, X : H or F, and Y: NCS

Fig. 1. Chemical structure of novel LC compounds substituted with the fluoro-isothiocyanate terminal group: nCCetPFSF, nCCPFSF.

In this work, we developed high elastic constant and high clearing point LC components without sacrificing the viscosity. These new compounds have a variety of physical properties and high potentiality to improve characteristics of LC mixture for a high response LCD. In order to achieve LC mixtures to be applicable to LCD TV, we prepared some LC mixtures by blending the fluoro-isothiocyanated LC components.

2. High Birefringence and Elastic Constant LC Mixture for LCD TV Application

From the small pretilt angle approximation [2], the switching on time (τ_{on}) and off time (τ_{off}) of a TN-LCD

are dependent on the cell gap d, rotational viscosity γ_1, elastic constant K_{ii} (i=1,2,3), threshold voltage V_{th}, dielectric anisotropy $\Delta\varepsilon$ and applied voltage V, as follows:

$$\tau_{on} \propto \left(\frac{d}{\pi}\right)^2 \frac{\gamma_1}{K_{eff}} \left(\frac{1}{(V/V_{th})^2 - 1}\right) \qquad (1)$$

$$\tau_{off} \propto \left(\frac{d}{\pi}\right)^2 \frac{\gamma_1}{K_{eff}} \qquad (2)$$

$$V_{th} = \pi \sqrt{\frac{K_{11} + (K_{33} - 2K_{22})/4}{\varepsilon_o \Delta\varepsilon}} = \pi \sqrt{\frac{K_{eff}}{\varepsilon_o \Delta\varepsilon}} \qquad (3)$$

Thus, a low threshold voltage can be obtained by either enhancing the dielectric anisotropy, reducing the elastic constant or a combination of both as exhibited in Equation (3). However, increasing the elastic constant would also speed up the switching off time of the LC device. Due to this unique relationship between K_{eff} and V_{th}, the resulting increase in V_{th} can be compensated by liquid crystal materials with high dielectric anisotropy. K_{eff} is an effective elastic constant of off state.

Normally, the LC switching on time is faster than the switching off time owing to the driving voltage factor, as described by Equation (1). According to these equations, the switching time of a TN-LCD is primarily determined by the cell gap d and the viscoelastic ratio γ_1/K_{eff} [3]. It is clear that a design of thinner cell gap is much more effective than a lowering rotational viscosity γ_1. Because γ_1 is contributed to the switching time in proportion to the first power and d is the second power. The effect of birefringence Δn on the switching time is through the cell gap dependence. High Δn help to shorten the switching time because of the thinner cell gap requirement.

For a transmission type 90° TN cell, the product of the cell gap d and the birefringence Δn needs to satisfy the Gooch-Tarry first minimum condition [4]. Let us consider the free relaxation process, the high elastic constant, while preserving low viscosity materials, become critical. Therefore, small viscoelastic ratio of liquid crystal mixture is necessary to realize very fast switching.

3. Experiments

The flouoro-isothiocyanated LC compounds were

Table 1. Physical properties and molecular parameters of fluoro-isothiocyanate LC compounds.

Abbreviation	n	m_1	m_2	X_1	X_2	μ^c	$\Delta\alpha^c$	$\Delta\alpha/M^c$	$\Delta\varepsilon^a$	Δn^a	η^b (mm^2s^{-1})	T_{CN} $(^\circ C)$	T_{NI} $(^\circ C)$	ΔH_{NI} (kJ/mol)	$\Delta S_{NI}/R$ (J/mol)
3CCPFS	3	1	0	F	H	3.23	229.0	0.638	19.72	0.254	27.90	81.5	237.4	0.98	0.231
3CCPFSF	3	1	0	F	F	3.68	227.5	0.603	20.16	0.242	28.26	65.7	215.7	1.13	0.278
5CCPFS	5	1	0	F	H	3.23	236.3	0.611	16.29	0.252	31.80	56.9	226.8	1.00	0.241
5CCPFSF	5	1	0	F	F	3.75	232.1	0.573	17.60	0.231	30.66	62.9	202.4	0.87	0.220
7CCPFS	7	1	0	F	H	3.45	239.1	0.576	15.89	0.248	34.11	50.6	207.6	1.10	0.275
7CCPFSF	7	1	0	F	F	3.76	238.8	0.552	16.48	0.223	34.57	60.7	195.5	0.48	0.123
3CCetPFS	3	1	1	F	H	3.49	196.9	0.509	16.82	0.246	32.50	72.9	189.0	2.60	0.677
3CCetPFSF	3	1	1	F	F	4.00	193.7	0.478	17.60	0.241	32.89	81.1	175.2	2.31	0.620
5CCetPFS	5	1	1	F	H	3.51	203.3	0.490	16.04	0.246	34.88	60.9	186.0	2.61	0.684
5CCetPFSF	5	1	1	F	F	4.01	200.5	0.463	16.79	0.224	35.10	50.0	175.3	2.24	0.601
3CetPFS	3	0	1	F	H	3.44	169.9	0.557	9.19	0.135	8.68	50.9	•	•	•
3CetPFSF	3	0	1	F	F	3.97	161.4	0.500	9.36	0.130	9.56	33.6	(20.1)	1.23	0.585
5CetPFS	5	0	1	F	H	3.49	157.6	0.473	8.77	0.140	12.55	29.5	44.0	1.54	0.584
5CetPFSF	5	0	1	F	F	3.98	153.5	0.437	8.86	0.139	13.16	9.0	33.2	1.32	0.518

a: Extrapolated values from 15 wt % solutions in the 4-(4-hexylcyclohexyl)-1-isothiocyanatobenzene.
b: Extrapolated values from 15 wt % solutions in the ZKC-1002(Chisso Petrochemical Co.).
c: For the calculations, we decided to use semi-empirical quantum chemical methods(AM1 Hamiltonian in the MOPAC 5.0 package).

synthesized by employing the method previously described [1]. They were produced by carbon-carbon bond formation from carbonyl compound and organolithium reagent generated from metal-halogen exchange reaction at 4-bromo-2-fluoro-N,N-dibenzyl-aniline, followed by dehydration, reduction and isothio-cyanation with thiophosgene. The chemical structures of the products were confirmed by ^1H and ^{13}C-NMR spectroscopy (Bruker DPX-400 MHz Spectroscope) and EI mass spectroscopy (70 eV Hewlett Packard 5972 MSD). Their purity was checked by a GLC analysis (Hewlett Packard 6890 series attached to a 30m HP-5MS column). and were found to be more than 99.9 % pure.

The mesophases were identified by optical microscopy and the phase transition temperatures were determined by differential scanning calorimetery. The Optical birefringence Δn was of the homeotropically aligned sample which was measured using an Abbe refractometer (λ=589 nm). The dielectric anisotropy $\Delta\varepsilon$ was determined by the capacitive method at a frequency of 1 kHz. The physical properties of the single compounds were obtained from 15 % solutions in the host mixture by extrapolation. In order to obtain maximum transmittance for TFT-LCD with a compensation film, it is well known that a total retardation value of LCD cell and compensation film should satisfy the Gooch-Tarry first minimum condition. The retardation of TFT-LCD cell was determined to be

0.36, and the test cells with cell gap of 3.5 and 4.5 μm were used. The switching times were measured by applying a square-wave AC voltage of 5 Volt at 60 Hz. The τ_{on} and τ_{off} were defined as the switching times between 10 % and 90 % transmittance.

4. Results and Discussion

4.1 Molecular parameters and physical properties

The thermodynamic properties for synthesized fluoro-isothiocyanated compounds and isothiocyanated com-pounds (nCCPS) are summarized in Table 1. As can be seen, most of the compounds exhibit enantiotropic nematic phases, whereas 3CetPFS is a monotropic liquid crystal. Only one compound, 3CetPFSF, is a non-mesogenic. No smectic phases were observed for any synthesized fluoro-isothiocyanated compounds.

All the homologues of the nCCPFS, nCCPFSF nCCetPFS, and nCCetPFSF series showed remarkably high nematic-isotropic transition temperature (T_{NI}) points (195.5 ~ 237.4 °C) and wide nematic temperature ranges (up to 169.9 °C for 5CCPFS). In the homologous series, the thermal stabilities decrease with the increasing number of fluorine atoms substituted in the benzene ring, and the nematic ranges shift to a lower temperature. In other words, introducing one fluorine atom induces a

Table 2. The comparison of liquid crystals having different terminal moieties.

Parameter	3CCPN[a]	3CCPS	3CCPFS	3CCPFSF
T_{NI} (°C)	242[b]	248.7	237.4	215.7
L/d (Å)[c]	18.0/4.4	19.0/4.4	19.0/4.6	19.0/4.8
μ (Debye[c])	4.05	2.80	3.23	3.68
Δα (a.u.[c])	128.1	176.2	229.1	228.4

a: The code indicates 4-[trans-4-(trans-4-propylcyclohexyl)-cyclohexyl]benzonitrile.
b: The T_{NI} value is referred to reference [7].
c: For the calculations, we decided to use semi-empirical quantum chemical methods
(AM1 Hamiltonian in the MOPAC 5.0 package).

decrease of 10~25 °C in T_{NI} points. The thermal stability is strongly influenced by the molecular parameters such as rigidity, length-to-breadth ratio (l/d), packing density, polarizability and enhancement of polarity by conjugation. These factors can rationalize the effect of fluoro-substitution on the thermal stability of liquid crystal molecules. The parameters from the molecular modeling calculations are listed in Table 1. As can be seen, the molecular lengths of 3CCPFS and 3CCPFSF are 19.0 Å, but their breadths vary in proportion to the number of fluorine atoms. In this case, other parameters such as the dipole moment (μ) and polarizability anisotropy (Δα) do not seem to be closely related to thermal stability. Thus, introducing a fluorine atom diminisheds the T_{NI} points because of the reduction of the l/d value of the molecule.

To explain this phenomenon, some molecular parameters of 4-[trans-4-(trans-4-propylcyclohexyl) cyclohexyl]-1-iso-thiocyanatobennzene (3CCPS) and 4-[trans-4-(trans-4-propylcyclohexyl) cyclohexyl] ben-zonitrile (3CCPN) are shown in Table 2. The rigidity, linearity (l/d) and size of 3CCPS may be similar to 3CCPN, which forms a dimer, causing the increase in packing density or T_{NI} point. And the dipole moment (μ) of 3CCPS is slightly lower than that of 3CCPN. Considering these factors, the T_{NI} point of 3CCPN may well be higher. However, the polarizability anisotropy (Δα) of 3CCPS is higher than that of 3CCPN because of the isothiocyanato group with three non-bonding electron pairs. This factor enhances the Van der Waal's attraction in the liquid crystal phase [5] so that the T_{NI} point of liquid crystal with the isothiocyanato group as terminal moiety is higher than with the cyano analogues.

The T_{NI} points of 1,2-ethylene-linked nCCetPFS and nCCetPFSF series are lower than those of the

nCCPFS and nCCPFSF series. It is probable that the introduction of the 1,2-ethylene linkage group into a directly bonded molecular core results in a substantial increase in the l/d of the molecule without having to decreasing its linearity of form. In this case, the carbon-carbon bond in the 1,2-ethylene linkage prefers to adopt anti-parallel conformers, which induce the energy minimization and the high molecular packing density. At high transition temperature, however, the proportion of anti-parallel conformers is lower than that of the gauch conformers with non-linear properties, which is not possible for the directly linked analogue. Therefore, the T_{NI} points of 1,2-ethylene linked three-ring compounds are lower than those of the corresponding directly-bonded compounds. Such observation has also been made in butyl linked bicyclohexylene compounds [5].

The magnitude of entropy for the nematic-isotropic transition ($\Delta S_{NI}/R$) values of synthesized compounds is exhibited in Table 1. Generally, the $\Delta S_{NI}/R$ value is 0.10 to 0.68 for low molar mass liquid crystals. With the introduction of 1,2-ethylene as a linkage group into the rigid central core of directly bonded bicyclohexylene compounds (nCCPFS and nCCPFSF), the $\Delta S_{NI}/R$ values of 1,2-ethylene linked three-ring compounds (nCCetPFS and nCCetPFSF) approximately double. In contrast, introducing the fluorine atom into a terminal benzene ring decreases $\Delta S_{NI}/R$ values. It is well known that inserting a 1,2-ehtylene group as the central linkage group affords greater conformational freedom, which could allow the rod-like moieties to align more efficiently with respect to the director, and thus pack more efficiently in the mesophase. Therefore, this increase in the molecular ordering of the mesophase increases the $\Delta S_{NI}/R$ at the transition to the isotropic

phase.

As shown in Table 1, the dielectric anisotropy ($\Delta\varepsilon = \varepsilon_{//} - \varepsilon_{\perp}$) values vary according to core structure, terminal group and alkyl spacer length. This appearance is explained by the Maier-Meier theory [6] based upon the Onsager model, and dielectric anisotropy is given by

$$\Delta\varepsilon = \frac{N_A F h}{\varepsilon_0}\left[\Delta\alpha + \left(\mu^2/2kT\right)\left(3\cos^2\beta - 1\right)\right] \bullet S \qquad (4)$$

where ε_0 is the free space permittivity, N is the number density, F is the reaction field factor, h is the cavity factor, $\Delta\alpha$ is the anisotropy polarizability of the molecule, k_B is the Boltzman constant, S is the order parameter, and μ is the dipole moment forming an angle β with the major inertial axis of the molecule.

The $\Delta\varepsilon$ values of the bicyclohexylene compounds are approximately twice as high as those of the cyclohexylene compounds, and the directly bonded bicyclohexylene compounds show $\Delta\varepsilon$ values slightly higher than the corresponding 1,2-ethylene linked bicyclohexylene compounds. These results show that the introduction of the trans-1,4-cyclohexylene fragment into the molecular core or the augmentation of the molecular rigidity increases the $\Delta\varepsilon$ value due to the enhancement of $\Delta\alpha$ in equation (4).

On the other, in the analogue series with the same core and alkyl spacer length, irrespective of the fact that difluoro-isothiocyanated compounds show slightly lower $\Delta\alpha$ values than monofluoro-isothiocyanated compounds, the $\Delta\varepsilon$ value of isothiocyanated compound substituted by monofluoro atom is larger than difluoro-substituted compounds. These effects can be explained by considering the μ term in equation (4) and dipole moment values depicted in Table 1.

The optical anisotropy values Δn have 0.130 ~ 0.140 for the cyclohexylene LC compounds and 0.223 ~ 0.254 for the bicyclohexylene compounds, as shown in Table 1 and Fig. 2. In spite of the similarity of the $\Delta\alpha/M$ value for all LC compounds, the Δn values of the bicyclohexylene compounds are almost twice as high as the cyclohexylene compounds due to the exhibiting high anisotropy of the molecular polarizability. The bicyclohexylene compounds have large $\Delta\varepsilon$ and high Δn. It is a general behavior for Δn increases to with the increase of $\Delta\varepsilon$ because of the relationship of two parameters.

According to the results on the kinematic viscosity

(v) value presented in Table 1, an increase in the number of trans-1,4-cyclohexylene fragments in the molecular core of the two-ring compounds significantly increases the values. Three-ring compounds indicate a v value that is three times higher than indicated by two-ring compounds. This viscous relationship has also been observed in other liquid crystals.

Fig. 2. Relationship between $\Delta\varepsilon$ and Δn for new liquid crystal substances for the experiments.

4.2 Liquid crystal mixtures for high response TFT-LCD TV

By using the newly fluoro-isothiocyated LC compounds having high potentiality to improve characteristics for a switching speed of TN-LCD, LC mixtures having excellent properties suitable for TFT-LCD TV application were developed. Many researchers have been looking for the liquid crystal materials with the high birefringence, low viscosity and high clearing points for TV/Monitor of TFT-LCD. Especially, in the large size TV applications having a high brightness similar to CRT, stronger backlights are used which heat up most components of the display sometimes up to sometimes more than 50 °C. Hence, development of the liquid crystal mixture with high clearing points is a decisive point for material development. In particular, LC mixture development should be performed for to further reduce switching time so as to display moving pictures at good quality. The viscoelastic ratio γ_1/K_{eff} is directly proportional to the switching time in the LC panel as described by equations (1) and (2). Hence, some focus was laid on the development of LC mixtures with higher clearing temperature of 80 °C or more and lower values of γ_1/K_{eff}. But, there exists an intrinsic trade-off relation

Table 3. Properties of practical LC Mixtures for application of TFT-LCD TV.

Specifications	LC-A	KUR-01	KUR-02	KUR-03	KUR-04
T_{SN}, T_{NI} (°C)	<-30, 70	<-30, 90	<-30, 102	<-30, 83	<-30, 81
Δn (589 nm, 25 °C)	0.077	0.104	0.095	0.102	0.112
$\Delta \varepsilon$ (1k Hz, 25 °C)	3.7	6.8	6.2	6.4	5.8
γ_1 (mPa s)	64.3	-	-	99.2	106.1
$\gamma_1/K_{eff} \times 10^{-10}$ (m^{-2}s)	0.66	0.72	0.69	0.61	0.57
$V_{10,0,25}$ (Volt)	1.5	1.5	1.6	1.4	1.4
$V_{90,0,25}$ (Volt)	2.6	2.6	2.9	2.6	2.5
Cell gap (μm)	3.5	3.5	3.5	3.5	4.5
τ_{on} (ms)	1.4	1.1	2.2	1.6	2.6
τ_{off} (ms)	8.2	9.0	8.6	7.6	11.8
VHR (%, 25 °C/60 °C)	98.0/97.7	97.9/96.4	97.5/96.8	98/96.6	98/97.1

between the clearing point T_{NI} of LC mixture and its corresponding rotational viscosity γ_1. Lower γ_1 values lead to lower clearing points.

As shown in Table 3, we developed several KUR-series TN mixtures by blending new LC substances depicted in Figure 1 and LC-A mixture. KUR-series were designed to have the Δn value of between 0.100 and 0.11 for 3.5 μm cell thickness, respectively

As can be clearly seen in Table 3, our novel LC mixtures have significantly higher clearing points and dielectric anisotropy values compared with LC-A mixture. Especially, their clearing points of them are 10~30 °C higher than their host mixture. Additionally, it was confirmed that their total switching times, $\tau_{on}+\tau_{off}$, were about 10 ms, which are half a magnitude smaller than conventional TN-LCDs and small enough to be addressed in 16.7 ms time frame. It was also confirmed that the threshold voltage V_{th} was low enough to operate at a driving voltage of 5 V.

Comparing the viscoelastic ratio γ_1/K_{eff} of LC-A with KUR-03 shows that the blending of fluoro-isothiocyanated LC compounds lead to a good increase in the effective elastic constant K_{eff} of KUR-03 mixture, despite the increase of γ_1. Therefore, because of the rather large K_{eff} value low γ_1/K_{eff} = 6.1 result which lead to the quick switching time τ_{off} of KUR-03 mixture. This finding is confirmed by the similar viscoelastic ratio data of KUR-01 and KUR-02.

We also measured the temperature dependence of voltage holding ratio (VHR) of the novel LC mixtures. The VHR properties were determined at 60Hz, which is a typical frame frequency of conventional addressing scheme. As listed in Table 3, the VHR values of KUR-series LC mixture were found to be high enough for TFT-LCD in a wide temperature range.

3. Conclusion

The most important requirement for liquid crystal display researchers is the reduction in switching time in LCD. We have succeeded in developing novel LC materials with higher clearing temperatures, higher birefringence and dielectric anisotropies. From the data presented in Table 1, it can be seen that materials, such as nCCPFS, nCCPFSF nCCetPFS, and nCCetPFSF series, show remarkably high clearing points (195.5 ~ 237.4 °C), broad nematic ranges (up to 169.9 °C for 5CCPFS) and high birefringence values of 0.254~0.220. KUR-series LC mixtures blended these single materials having signify-cantly higher clearing points and dielectric anisotropy values compared with conventional LC mixture. Especially, their clearing points of are 10~30 °C higher than their host mixture.

We have achieved LC mixtures showing about 10ms of high-speed switching time that is shorter enough to be addressed in one frame time of 60Hz (16.7 ms). The threshold voltage V_{th} was low enough to operate at a

driving voltage of 5V. The VHR values were found to high enough for TFT-LCD in wide temperature range.

Acknowledgement

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