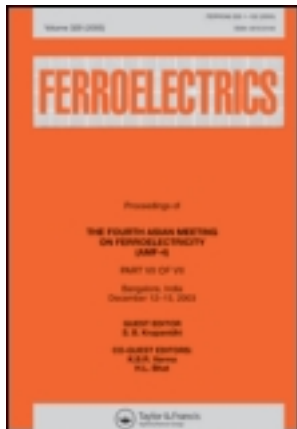


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### Defect-Free FLC'D's with High Optical Quality Based Upon New FLC's

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## Defect-Free FLC D's with High Optical Quality Based Upon New FLC's

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*We have designed new FLC compounds and new FLC materials based upon them, and we have found general criteria of the preparation of FLC D's with uniform and stable orientation of FLC materials [1]. The influence of the molecular structure on the quality of orientation, on thermal and mechanical stability and on the contrast ratio was investigated by using 4 types of aligning materials in the thin and thick testing cells. The correlation between the number, type and amount of achiral and 3-, 4-ring chiral compounds in the mixture and contrast ratio of our FLC D's was found. Finally we optimized the FLC-mixtures, alignment conditions and we prepared the defect-free samples with optical contrast about 700:1.*

**Keywords** Ferroelectric liquid crystalline materials; tilt angle; defect-free

### Introduction

The surface-stabilized ferroelectric LCD's is an attractive device because of its unique characteristics such as bistable memory capability, wide viewing angle and fast response [2]. However, it is hard to fabricate a defect-free SSFLCD's owing to the appearance of the so-called zig-zag defect that degrades memory capability and contrast ratio of the display.

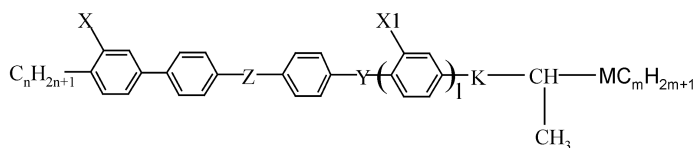
The development described in this paper has solved two fundamental problems of FLC D's. The first one is shock stability and the second one is the gray scale.

Main attention of this research has focused on determining what alignment materials and surface treatments tend to produce defect-free textures. In this work, key technologies were developed in order to overcome these problems. Firstly we suggested that FLC materials may play the main role in the solution of these problems and decided that flexurally rigid rod-like chiral molecules with the definite length, the long helical pitches and a wide temperature range of the SmC\* phase are needed for this. Long helical pitches in SmC\* phases are required in order to yield a high quality of alignment FLC materials. A wide temperature range of SmC\* phase is needed for the extension of the operating temperature range of FLC D's. For this target the new FLC-mixtures base upon 3- and 4-ring chiral compounds with different positions of the lateral substitutes on the central core and with different positions of the bridge fragment were developed [1].

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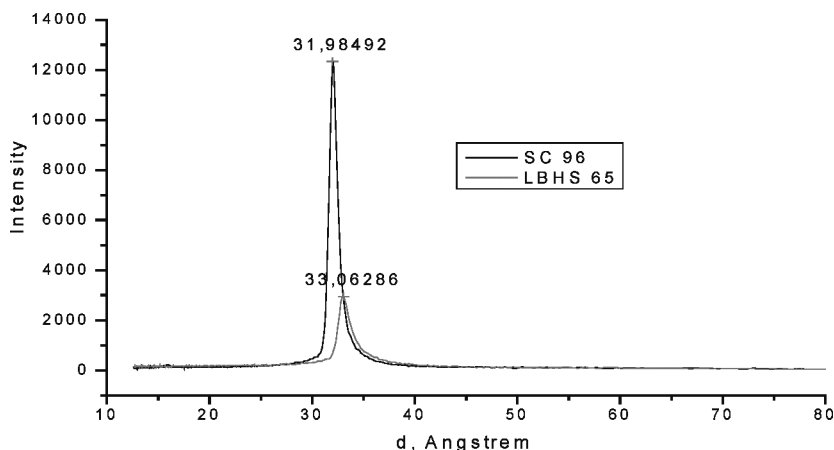
$n = 6-10$ ;  $m = 4-6$ ;  $\text{X}, \text{X1} = \text{CH}_3, \text{Cl}$ ;  $\text{Z}, \text{Y}, \text{M} = \text{COO}$ , single bond;  $\text{K} = \text{O}, \text{COO}$ ;  $l = 0-1$

Secondly we suggested that these molecules can be mixed together with a special mobile and flexible buffers like a spring. In this case the decreasing of the translation order may be observed. The influence of the molecular structure on the quality of orientation, on thermal and mechanical stability and on the contrast ratio of FLCCD's was investigated using 4 types of aligning materials for thick testing cells. In fact, it has been reported before for SSFLCD fabricated using weak-surface-anchoring alignment films. For the understanding of the role of the surface morphology of the rubbed alignment layers we investigated the testing cell with the weak, medium and strong rubbing.

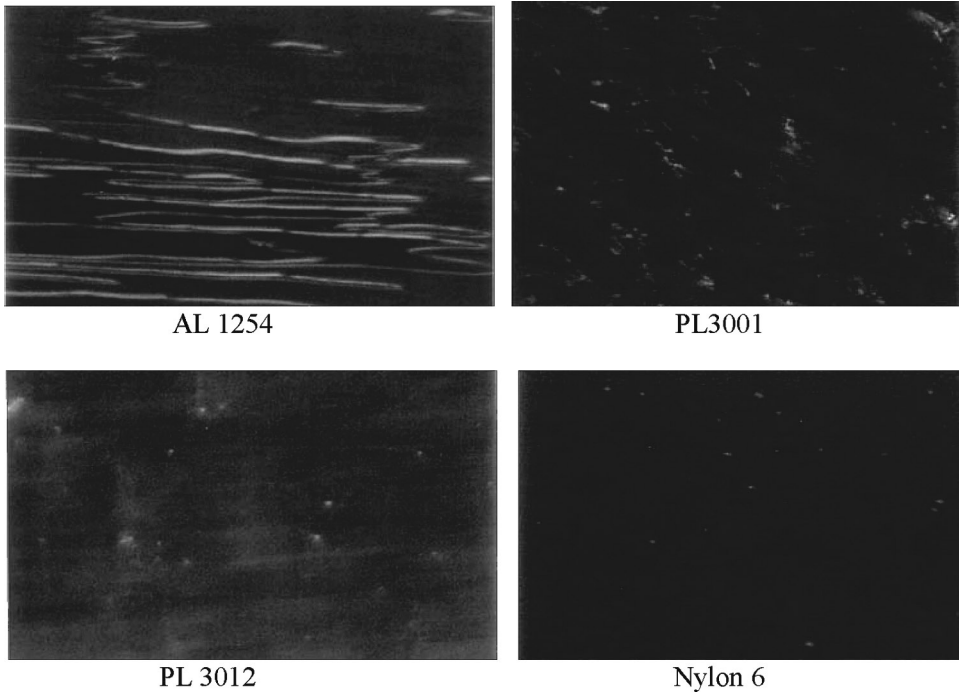
Finally the general criteria of manufacturing the thermal and mechanical stable FLCCD's with high contrast ratio and good memory capability for the different cell-gap (from  $1.5 \mu\text{m}$  till  $20 \mu\text{m}$ ) was evaluated.

## Results and Discussion

For the preparation of the FLC-mixtures we used the new flexurally rigid rod-like chiral molecules with the definite length and wide temperature range of the  $\text{SmC}^*$  phase. These molecules were mixed together with a special mobile and flexible buffers that allow similar to spring to restore instantly of the smectic layers after mechanical pressure. Firstly we optimized the type of the flexible buffer. After that the correlation between the number and amount of the 3- and 4-ring chiral compounds and special dopants was found. In this conditions the smectic layers are not broken under the pressure and only become deformed independently from the force of pressure and value of the shear (geometrical deviation) [3]. Also from XRD data we found that intensity of peak for SC 96 is higher and half-width is smaller than for LBHS-65. It means that translation order for LBHS-65 is smaller. This confirms our suggestions that LBHS-65 is promising for the preparation shock- problem free FLCCD's (see Fig. 1).



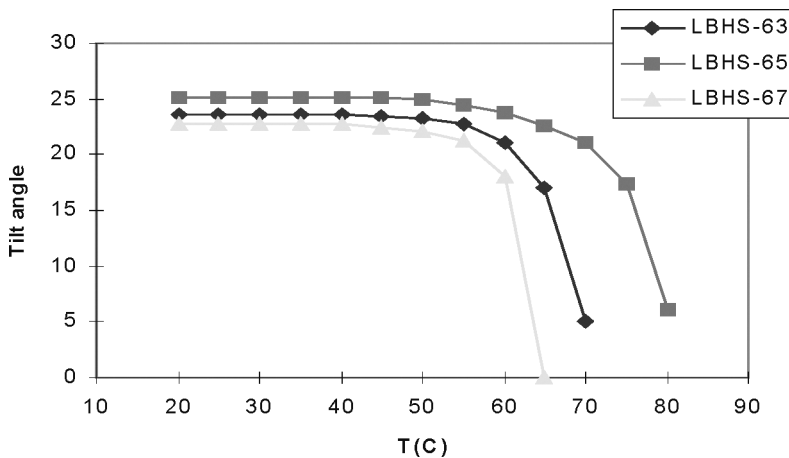
**Figure 1.** XRD pattern for compound SC 96 and mixture LBHS-65.(See Color Plate XXXI)



**Figure 2.** Photographs under polarizing microscope of FLC cell with the different alignment materials. (See Color Plate XXXII)

On the first stage we investigated all our mixtures in the testing cells with cell-gap  $1.5 - 3 \mu\text{m}$  and with the weak, medium and strong rubbing using the different alignment materials (nylon 6, polyimides-Al 1254, PL 3001, PL 3012) (see Fig. 2).

The developed FLC-materials and cells with nylon 6, PL 3001 and medium rubbing possess a wide temperature range of the  $\text{SmC}^*$  phase as well as bistability, gray scale capability, low operation voltage, high optical contrast, temperature independence of the main parameters and therefore they are promising good candidates for practical application (see Fig. 3 and Table 1).

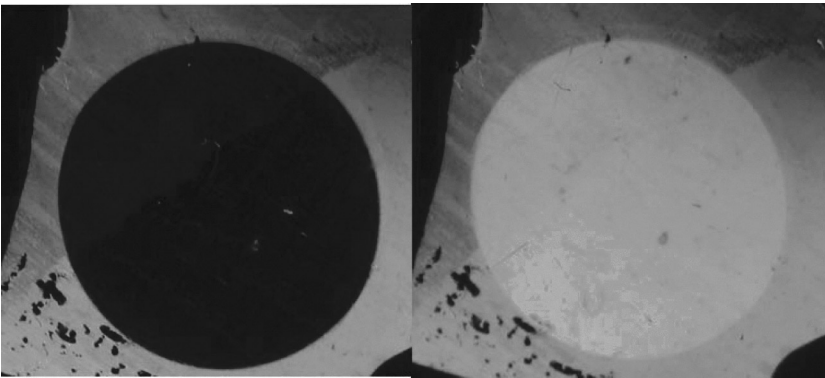


**Figure 3.** Temperature dependence of the tilt angle of FLC mixtures. (See Color Plate XXXIII).

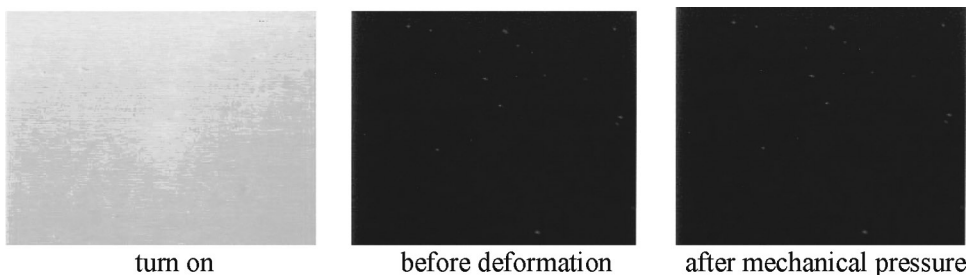
**Table 1**  
Physical and electrooptical properties of FLC mixtures

Mixtures	SmC* temperature range (°C)	Operating voltage (V/ $\mu$ m)	Spontaneous polarization (nC/cm <sup>2</sup> )	Contrast ratio	t <sub>on</sub> (ms)	t <sub>off</sub> (ms)	Tilt angle
SC 96	20< -+117.2	10	112	11:1	2.0	2.1	33.2
SC 106	20< -+98.6	10	107	10:1	1.91	1.95	28.7
LBHS-55	20< -+74.7	10	21	65:1	0.62	0.63	23.9
LBHS-56	20< -+68.3	10	21	52:1	0.6	0.61	23.7
LBHS-59	20< -+99.3	10	31	44:1	0.78	0.83	30.6
LBHS-63	20< -+73.4	10	23	500:1	0.52	0.53	23.8
LBHS-63 (AL-1254)	20< -+73.4	10	21	134:1	0.48	0.49	23.4
LBHS-63 (PL-3001)	20< -+73.4	10	21	77:1	0.43	0.44	23.2
LBHS-65	20< -+74.5	10	21	140:1	0.34	0.35	25.3
LBHS-65 (AL-1254)	20< -+74.5	10	23	125:1	0.32	0.33	24.9
LBHS-65 (PL-3001)	20< -+74.5	10	24	74:1	0.31	0.31	24.7
LBHS-67	20< -+64.5	10	12	690:1	0.48	0.49	22.8
LBHS-69	20< -+88.8	10	25	210:1	0.35	0.36	25.4
LBHS-70	20< -+84.2	10	23	44:1	0.62	0.63	23.2
LBHS-71	20< -+75.1	10	27	170:1	0.28	0.28	25.9
LBHS-72	20< -+60.2	10	25	78:1	0.62	0.63	26.5
LBHS-73	20< -+69.3	10	89	82:1	0.32	0.31	29.7
LBHS-77	20< -+87.9	10	19	44:1	0.52	0.53	27.2
LBHS-81	20< -+91.5	10	149	30:1	0.21	0.21	18.3
LBHS-84	20< -+84.2	10	23	44:1	0.62	0.63	23.2

The maximum contrast ratio 700:1 was achieved for the best testing cell (see Fig. 4). Our first cells were prepared 10 months ago. During this period we checked them on the thermal and mechanical stability. One day in the week we become deformed under pressure our cells when shear (geometrical deviation) was more than 60% (for example: when we pushed on the cells the thickness of them was decreased from 3.5 $\mu$ m till 1.5  $\mu$ m). We did



**Figure 4.** The example of defect-free cells with high contrast ratio (LBHS-67, d = 1.7  $\mu$ m). (See Color Plate XXXIV)



**Figure 5.** Photographs under polarizing microscope of FLC cell before and after strong pressure during long time [when shear (geometrical deviation) more than 60%]. (See Color Plate XXXV)

not find any changes after that, all these cells are defect-free and are characterized the same alignment quality. The thermal stability of the samples is very good also (see Fig. 5).

We did not investigate our cell when shear was more than 60%, because cells were broken under such pressure.

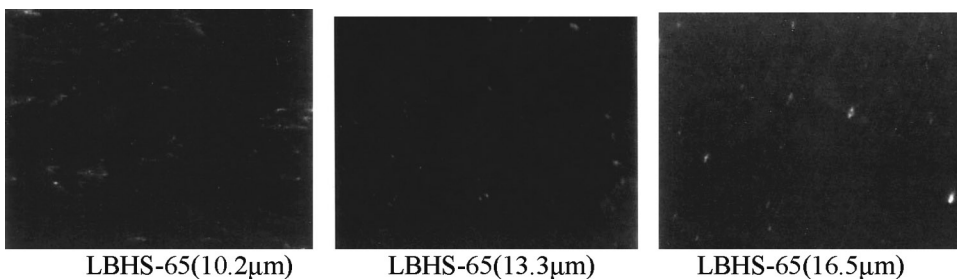
Then we decided to check what is maximum thickness when the cells do not have the shock problems. Our investigations have shown that the structure of the molecules very strongly influences the possibility of good orientation of FLC materials in thick cells also. We found that a special mobile and flexible buffers very strong influence on the maximum oriented cell gap and value of contrast ratio (see Table 2, Figs. 6 and 7).

This is a threshold effect (thickness of the cell when the smectic layers are not oriented). Finally we prepared thermal and mechanical stable samples with a cell-gap of about 21  $\mu\text{m}$  (see Fig. 8).

The first thick cells were prepared 7 months ago. During this period we checked them on the thermal and mechanical stability. One day in the week we pressured our cells when shear (geometrical deviation) was 50–60% (for example: when we pushed on the cells the thickness of them was decreased from 16.5  $\mu\text{m}$  till 8.0  $\mu\text{m}$ ). We did not find any changes for 50% shear and big changes for 60% shear after that. For first case all these cells are defect-free and are characterized the same alignment quality and for the second case contrast ratio decreased only a 10%.

## Experimental

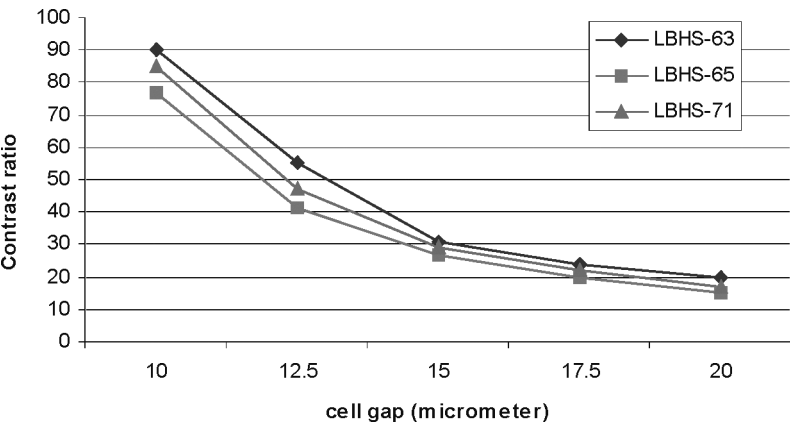
The structures of the prepared compounds were confirmed by  $^1\text{H}$ -NMR and mass spectroscopy. Phase transition temperatures were measured using a Linkam heating stage having a polarizing PZO microscope and also using a Setaram DSC 92.



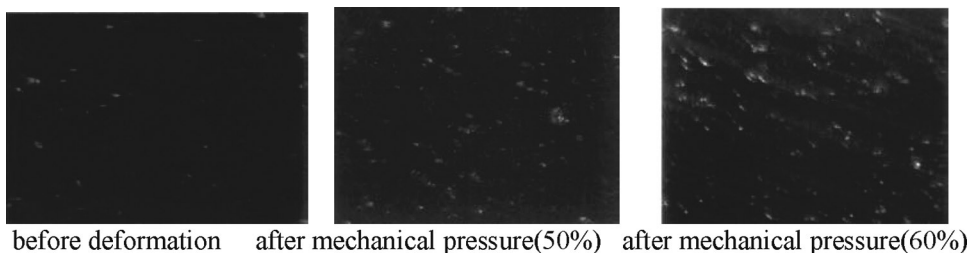
**Figure 6.** Photographs under polarizing microscope of FLC cells with the different thickness. (See Color Plate XXXVI)

**Table 2**  
Physical and electrooptical parameters of the FLC mixtures

Mixtures	SmC* temperature range (°C)	Operating voltage (V/ $\mu$ m)	Spontaneous polarization (nC/cm <sup>2</sup> )	Contrast ratio	t <sub>on</sub> (ms)	Tilt angle	Cell gap ( $\mu$ m)
LBHS-63	20< – +73.4	3	22	92:1	0.53	23.5	9.2
			22	55:1	0.51	23.6	12.2
			21	31:1	0.52	23.8	15.9
			21	20:1	0.52	23.9	20.4
LBHS-63 (AL-1254)	20< – +73.4	3	21	19:1	0.48	23.4	16.1
LBHS-63 (PL-3001)	20< – +73.4	3	21	15:1	0.43	23.4	16.3
LBHS-65	20< – +74.5	3	25	77:1	0.35	25.3	10.2
			25	41:1	0.35	25.3	13.3
			24	27:1	0.36	25.4	16.5
			24	15:1	0.36	25.4	20.8
LBHS-65 (AL-1254)	20< – +74.5	3	25	17:1	0.32	25.2	16.2
LBHS-65 (PL-3001)	20< – +74.5	3	25	12:1	0.31	25.1	16.2
LBHS-65	20< – +75.1	3	27	85:1	0.32	25.8	9.9
			27	47:1	0.32	25.9	12.7
			26	29:1	0.33	25.9	16.2
			26	17:1	0.33	25.9	19.9
LBHS-71 (AL-1254)	20< – +75.1	10	27	19:1	0.31	25.6	15.9
LBHS-71 (PL-3001)	20< – +75.1	10	27	15:1	0.30	25.5	16.2



**Figure 7.** Contrast ratio dependence of the thickness of the cell. (See Color Plate XXXVII)



**Figure 8.** Photographs under polarizing microscope of FLC cell ( $d = 20.8 \mu\text{m}$ ) before and after moderate or strong pressure during long period [when shear (geometrical deviation) less than 50% and more than 60%]. (See Color Plate XXXVIII)

The solution of the alignment materials [nylon 6, polyimides (Al 1254, PL-3001, PL-3012) 80–120 nm] was spun onto the indium-tin-oxid(ITO) substrate (with the receptivity about  $20\text{--}30 \Omega/\text{cm}^2$ ) at 3000 rpm rate and then baked at  $180^\circ\text{C}$  for 1 hour for the imidization to get the polyimide film. Aligning layers were unidirectional rubbed under a velvet-covered cylinder. The thickness of the cells was about  $1.5\text{--}21 \mu\text{m}$  and measured in each case interferometrically. The microscope textures of the SSFLC cells were observed using a polarising microscope. The EO properties of the SSFLC cells were measured between the crossed polarizers using a He-Ne laser. The light transmittance was detected by a photomultiplier and recorded in a digital oscilloscope. The contrast ratio was measured under the application of a  $\pm 10 \text{ V}/\mu\text{m}$ , 10 Hz rectangular voltage. The rise and decay times are defined as the transmittance changes from 10% - 90% and vice versa. The bistability (or the memory capability) was measured using a 1 ms bipolar pulse with a period of 20 ms. During electro-optic measurements the temperature of the cells was controlled with the accuracy  $0.3^\circ\text{C}$ .

XRD measurements were made on STOE Stadi 4 plant with standard 1, 1.5, 2 mm capillaries (from Hilgenberg)

## Conclusion

- Correlation between the chemical structures of molecules and shock-problem free FLCD's was found
- Correlation between the amount of the flexurally rigid rod-like chiral molecules with the definite length, mobile and flexible buffers (spring) and contrast ratio was found
- These results show that the defect-free FLCD's when shear (geometrical deviation) more than 60 % with small thickness ( $< 5 \mu\text{m}$ ) and less than 50% with large thickness ( $> 15 \mu\text{m}$ ) are real and can be practically realized.
- Correlation between the amount and type of mobile and flexible buffers (spring) in the mixture and maximum thickness for defect-free cells was found
- Correlation between the chemical structures of molecules and contrast ratio for thick cells was found

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