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Liquid crystal alignment properties of 2-naphthoxymethyl-substituted polystyrenes

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The liquid crystal (LC) alignment properties of LC cells fabricated with films of 2-naphthoxymethyl-substituted polystyrenes with different contents of naphthoxymethyl side groups were investigated. The polymer films exhibited good optical transparency in the visible light region (400–700 nm). The LC cells made from the unrubbed films of polymers having more than 57 mol% of 2-naphthoxymethyl containing monomeric units showed home-otropic LC alignment with a high pretilt angle of about 90°. Good electro-optical characteristics, such as the threshold voltage, response time, voltage holding ratio and residual DC voltage were observed for the LC cells fabricated with the polymer having 100 mol% of 2-naphthoxymethyl containing monomeric units as an LC alignment layer.

Keywords: liquid crystal; alignment; electro-optical property; polystyrene

1. Introduction

The alignment technologies of liquid crystal (LC) molecules on alignment layer surfaces are very important for the fabrication of stable LC cells in LC devices (1-8). Homeotropic LC alignment methods on alignment layer surfaces have been known to produce LC cells with better electro-optical (E-O) performance, such as a faster response time and higher contrast ratio, than those prepared using homogeneous planar LC alignment methods (9, 10). The homeotropic alignment mode using multi-domain vertical alignment substrates such as protrusion and fringe-field vertical alignment was reported by Takeda *et al.* (11)to improve the image quality and E-O performance in the liquid crystal display (LCD) industry.

Polyimide derivatives with long alkyl or alkyloxy groups have been synthesised in order to use them as homeotropic LC alignment layers (12-15). For example, the homeotropic LC alignment of the LC cells made from films of semi-flexible copolyimides containing *n*-octadecyl side groups (12) and polyimides with (n-decyloxy)biphenyloxy or (n-dodecyloxy)biphenyloxy side chains (14) can be observed. Hard baking processes are used to produce polyimide alignment layers and the conventional hard baking temperature of polyimide films is usually over 200°C, which is too high for manufacturing flexible plastic LC devices (16, 17). Therefore, ion (18, 19) and electron (20) beam exposure methods using inorganic substrates and nanotechnologies using nanomaterials (17, 21, 22), nanopatterns (7, 23), and nanostructures (24, 25) were developed to produce homeotropic LC alignment layer surfaces under milder conditions.

Recently, we found that polystyrene surfaces producing homogeneous planar and homeotropic LC alignment layers can be prepared at low temperatures (26-30). For example, the LC cells fabricated with films of 2-naphthoxymethyl-substituted polystyrene exhibited homeotropic LC alignment behaviour with a high pretilt angle of approximately 90° through a non-rubbing process, and these alignment layers having good solubility in many organic solvents can be produced at a low temperature using the spin casting method (28). In this paper, the LC alignment properties and E-O characteristics of the LC cells made from 2-naphthoxymethyl-substituted polystyrenes were investigated for possible applications in LCDs.

2. Experimental

2.1 Film preparation and LC alignment process

Solutions of the 2-naphthoxymethyl-substituted polystyrenes (P2NAP# shown in Figure 1) in toluene (2 wt%) were prepared. These solutions were filtered through a polytetrafluoroethylene (PTFE) membrane with a pore size of 0.45 μ m. Thin films of the polymers were prepared by spin-coating (2000 rpm, 30 seconds) them on to 1.5 cm × 1.5 cm indium tin oxide (ITO)coated glass substrates and 2.5 cm × 2.5 cm patterned vertical alignment (PVA) substrates, which were kindly supplied by Samsung Electronics. The polymer films were rubbed using a rubbing machine (RMS-50-M, Nam II Optical Components Corp., Incheon, Korea). The rubbing density equation is written as

$$L/l = N[(2\pi rn/60\nu) - 1],$$

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where L is the total length of the rubbing cloth (mm), l is the contact length of the circumference of the rubbing roller (mm), N is the cumulative number of rubbings, n is the speed (rpm) of the rubbing roller, r is the radius (cm) of rubbing roller, and v is the velocity (cm s⁻¹) of the substrate stage (31, 32).

2.2 LC cell assembly

Antiparallel LC cells were fabricated using the unrubbed or rubbed polymer films on the ITO-coated glass slides. The antiparallel LC cells were constructed by assembling the polymer films together antiparallel with respect to the rubbing direction using spacers with thicknesses of 6.5 μ m and 50 μ m. The LC cell was constructed by assembling the polymer films on to the PVA substrates using spacers with a thickness of 6.5 μ m for the measurement of the voltage-transmittance and response time values. The fabricated LC cells were filled with a nematic LC, MLC-7026-000 (Merck Co., $n_{\rm e} = 1.5577$, $n_{\rm o} = 1.4755$ and $\Delta \varepsilon = -3.7$, where $n_{\rm e}$, $n_{\rm o}$ and $\Delta \varepsilon$ represent the extraordinary refractive indexes, ordinary refractive indexes and dielectric anisotropy, respectively), in the isotropic state in order to avoid creating flow alignment by capillary action. The manufactured LC cells were sealed with epoxy.

2.3 Instrumentation

The optical transmittance of the P2NAP# films was obtained using UV-Vis spectroscopy (Perkin Elmer Lamda 20 spectrometer). For the UV-Vis spectroscopy of P2NAP#, the polymer films were prepared by spin-coating P2NAP# in toluene (2 wt%) on to ITO-coated glass substrates at 2000 rpm for 30 seconds. The contact angles of distilled water and methylene iodide on the polymer films were determined with a Kruss DSA10 contact angle analyser equipped with drop shape analysis software. The Owens-Wendt's equation was applied to calculate the surface energy values from the static contact angles (33). The LC alignment direction of the antiparallel LC cells was investigated by measuring the angular dependence of the absorbance of a dichroic dye (disperse blue 1, Aldrich), which was dissolved in MLC-7026-000 at a concentration of 1 wt%, using an optical apparatus equipped with a He-Ne laser, a polariser and a photodiode detector as a function of the rotation angle of the samples (32). The pretilt angle of the LCs of the antiparallel LC cell using spacers with a thickness of 50 μ m was measured by the crystal rotation method (34). The cell gap was measured before filling the LCs using a spectrophotometer S 2000 (Ocean Optics Inc., Dunedin, FL). The polarised optical microscopy (POM) images of the LC cells were observed using

an optical microscopy (Nikon, ECLIPSE E600 POL) equipped with a polariser and digital camera (Nikon, COOLPIX995). The voltage holding ratio (VHR) was measured using a VHR measurement system (autronic-MELCHERS, VHRM 105). The pulse width, frame frequency and data voltages were 64 μ s, 60 Hz and 1.0 V, respectively. The measurement temperatures were 25°C and 60°C. The residual DC voltage (R-DC) value was evaluated using a capacitance-voltage (C-V) hysteresis method, which is used by Nissan Chemical Industries, Ltd. The response time and voltage-transmittance (V-T) were measured from the LC cells using the same method as that reported by others (35). The threshold voltage $(V_{\rm th})$ and driving voltage (V_{on}) in the V-T curve are defined as the voltages at which the transmittance was decreased to 90% and 10% of the initial transmittance value, respectively. The rising (T_r) and falling (T_f) response times for the white-to-black and black-towhite changes, respectively, are defined as the time to transition from 10% to 90% transmittance and vice versa (35).

3. Results and discussion

Figure 1 shows the chemical structure of the 2-naphthoxymethyl-substituted polystyrenes (P2NAP#, where # is the molar content of 2-naphthoxymethyl side groups in %). These polymers with different degrees of substitution were obtained through the reaction of



Figure 1. Chemical structure of P2NAP# film.



Figure 2. UV-Vis transmittance spectra of P2NAP# films according to the monomeric units of 2-naphthoxymethyl side groups.

polychloromethylstyrene with 2-naphthol by varying the amount of 2-naphthol in N,N'-dimethylacetamide. More detailed information about the synthesis and characterisation was reported previously (28).

Figure 2 shows the UV-Vis transmittance spectra of the P2NAP# films. The transmittance value of the P2NAP# film increases as the content of 2-naphthoxymethyl side group increases and it is slightly smaller than that of bare ITO. For example, as the molar content of 2-naphthoxymethyl side groups increases from 20% to 100%, the transmittance of the P2NAP# films decreases from 86.9% to 84.6% at 550 nm, and that of bare ITO is 87% at 550 nm. However, the optical transparency of all the films in the visible light region (400–700 nm) is still good enough for them to be used as optical materials for flexible LCD devices (36).

At first, the LC alignment behaviour of the LC cells fabricated with the unrubbed polymer films was investigated. Homeotropic LC alignment was observed from the unrubbed P2NAP100, P2NAP79 and P2NAP57 films. For example, the POM image (Figure 3(a)) and polar diagram (Figure 4(a)) of the LC cell made from P2NAP100 film clearly show homeotropic LC alignment behaviour. Furthermore, these LC cells showed very good room temperature stability for a duration of at least 2 years. Figure 4(d)–(f) clearly show that the very nice homeotropic LC aligning ability of LC cells fabricated from the unrubbed P2NAP100, P2NAP79 and P2NAP57 films showing good polar diagrams have been maintained for 24 months since we first made the LC cell. However we could not obtain any good polar diagrams showing orientation from the LC cells fabricated using the unrubbed P2NAP40 and P2NAP20



Figure 3. Polarised optical microscopy images in dark and conoscopic (inlet) images of the liquid crystal cells made from unrubbed, (a) P2NAP100, (b) P2NAP79, (c) P2NAP57, (d) P2NAP40 and (e) P2NAP20, and rubbed, (f) P2NAP100, (g) P2NAP79, (h) P2NAP57, (i) P2NAP40 and (j) P2NAP20, P2NAP# films with a rubbing density of 150.

films, because they induced partial homeotropic LC alignment and random planar LC alignment behaviours, as shown in Figure 3(d) and (e), respectively.

All the LC cells fabricated from the rubbed P2NAP# films with a rubbing density of 150 show homogeneous planar and perpendicular LC alignment with respect to the rubbing direction, as in the case of the rubbed polystyrene film (37-39). This homogeneous planar LC alignment behaviour was confirmed by the POM images as shown in Figure 3(f)–(j), while the homogeneity was lost after a few days, producing a totally random planar orientation of the LCs. We believe that the instability of the LC cells made from the rubbed P2NAP#s is caused by their low anchoring properties.

We found that the unrubbed polymer (P2NAP100, P2NAP79 and P2NAP57) films producing homeotropic LC alignment behaviour have lower surface energy values than those (P2NAP40 and P2NAP20) producing random LC orientation behaviour; the surface energy values of P2NAP100, P2NAP79 and P2NAP57 are 45.9, 46.4 and 47.0 mJ m⁻², respectively, and those of P2NAP40 and P2NAP20 are 47.9 and 48.7 mJ m⁻², respectively (Table 1). It has been known that the high pretilt angle of LCs on alignment layer surfaces could be ascribed to the low surface energy values of the alignment layer (40–44). In our case, the polymer films having surface energy values smaller than about 47.0 mJ m⁻² could induce homeotropic LC alignment behaviour with a pretilt angle of about 90°.

The pretilt angles of the antiparallel LC cells fabricated with the P2NAP# films were measured in order to determine the effect of the rubbing density on the LC alignment direction (Figure 5). The pretilt angles of the LCs on the rubbed P2NAP20 and P2NAP40 films with a rubbing density of <50 could not be measured, due to the poor LC alignment. Low



Figure 4. Polar diagrams of the absorbance of disperse blue 1 in the antiparallel liquid crystal cells fabricated from unrubbed polymer films as a function of the rotation angle of the samples. (a) P2NAP100, (b) P2NAP79 and (c) P2NAP57 were obtained just after the liquid crystal cells were prepared and (d) P2NAP100, (e) P2NAP79 and (f) P2NAP57 were obtained after 24 months.

Table 1. Surface energy and liquid crystal alignment property of P2NAP# film.

Polymer designation	Contact angle (o) ^a				
	Water	Methylene iodide	Surface energy (mJ m ⁻²) ^b	Homeotropic liquid crystal aligning ability ^c	Pretilt angle (o) ^d
P2NAP20	81	18	48.7	Х	_e
P2NAP40	81	21	47.9	Δ	_e
P2NAP57	81	24	47.0	0	90
P2NAP79	81	26	46.4	0	90
P2NAP100	80	28	45.9	0	90

^aMeasured from static contact angles.

^bCalculated from Owens-Wendt's equation.

 $^{\circ}$ O: good homeotropic LC alignment; Δ : partial homeotropic LC alignment; X: random planar LC alignment.

^dMeasured from the antiparallel LC cells made from unrubbed polymer films.

"The pretilt angles of LCs on the unrubbed polymer films could not be measured due to poor LC alignment.

pretilt angles of approximately 0° on the rubbed P2NAP20 and P2NAP40 films were obtained at a rubbing density of >50. For the LC cells made from the P2NAP100, P2NAP79 and P2NAP57 films, the pretilt angles of about 90° for the unrubbed films decrease slightly as the rubbing density increases from 0 to 75. For example, the pretilt angles of the rubbed P2NAP100 and P2NAP57 films with a rubbing density of 75 are 85° and 84° , respectively. When the rubbing density of the P2NAP100, P2NAP79 and P2NAP57 films was increased to 100, the pretilt angles decreased abruptly to about 0° , indicating that the LC alignment directions on the P2NAP# change to homogeneous planar LC alignment. Therefore, a rubbing

density between 75 and 100 is a critical value for changing the LC alignment direction on the P2NAP# films having a 2-naphthoxymethyl content larger than 57 mol% from homeotropic to homogeneous planar LC alignment. The surface energy values of the rubbed P2NAP# films were also measured in order to investigate the effect of wettability on the pretilt angles of the LCs on the rubbed P2NAP# surfaces as a function of the rubbing density. The surface energy values of the rubbed P2NAP# films were found to be almost identical to those of the unrubbed P2NAP# films, regardless of the rubbing density. Therefore, we believe that the changes of the pretilt angles of the LCs after the rubbing process are not



Figure 5. Pretilt angles of the liquid crystal cells fabricated with unrubbed or rubbed P2NAP# films as a function of the rubbing density.

caused by the change of surface energy value, but by that of the molecular orientation on the polymer surfaces. The change of the molecular orientation on the P2NAP100 surfaces caused by the rubbing process was confirmed by the Fourier transform infrared dichroic spectra, as described in a previous paper (28).

Figure 6 shows the POM images of the LC cells fabricated with the P2NAP100 films heated for 10 minutes at various temperatures (room temperature, 125°C, 150°C, 200°C and 250°C). The homeotropic LC alignment behaviour of the LC cell was found to be maintained when it was heated for 10 minutes at 125°C (Figure 6(b)). Since the glass transition temperature of P2NAP100 is about 67°C (28), 125°C is well above the temperature at which the molecular motions start (45). Therefore, the molecular motion on the surface of P2NAP100 heated at 125°C for 10 minutes is not enough to disturb the orientation of the LCs However, defects of the LC alignment were observed in the LC cells heated at 150°C and 200°C, as shown in Figure 6(c) and (d), respectively, and totally random LC orientation was observed in the LC cell heated at 250°C for 10 minutes (Figure 6(e)).

Therefore, the processing temperature of P2NAP100 for the LCD application should be below 125°C.

The E-O performance of the LC cell fabricated with the unrubbed P2NAP100 film was measured for possible practical LCD applications (Figure 7 and Table 2). The LC cell exhibited a VHR of about 98% at 25°C and this value was maintained at 60°C. It is sufficiently high for practical applications as the LC alignment layer in thin-film transistor addressed LCDs (1). The R-DC of the LC cell measured using the C–V hysteresis method was found to be very low, about 38 mV. This value is lower than that of commercial polyimides (1). The V-T and response time values were determined in order to investigate the driving property of the LC cell. The angular dependence of the transmitted light intensity of an LP He-Ne laser through the LC cell between cross polarisers with increasing applied voltage was investigated by applying 1 kHz AC voltages. The transmitted light intensity of the LP He-Ne laser through the LC cell remains constant as the applied voltage is increased from 0 to 2.2 V, then starts to increase as the applied voltage is further increased from 2.2 V to 3.2 V, indicating that the LC orientation changes from homeotropic to homogeneous planar alignment, and the maximum transmittance is observed when the applied voltage is 3.2 V. Figure 7 clearly shows that the LC cell made from the P2NAP100 film has good E-O properties with a good contrast ratio. The $V_{\rm th}$, $V_{\rm on}$ and response time values of P2NAP100 were 2.34 V, 3.02 V and 32 ms, respectively, which are close to those of PI reported by others (Table 2) (46, 47).

Recently, considerable effort has been expended in an attempt to develop plastic substrates for flexible LCDs (48). We found that the LC cells fabricated using the unrubbed films of P2NAP57, P2NAP79 and P2NAP100 on plastic (PET, polyethylene terephthalate) substrates show good homeotropic LC alignment behaviour. Therefore, P2NAP# films might be candidates for use as LC alignment layers for flexible LCDs, such as in vertical alignment mode applications. Further research on the application of P2NAP#s for flexible LCDs is in progress.



Figure 6. Polarised optical microscopy images of the liquid crystal cells made from unrubbed P2NAP100 films annealed for 10 minutes at (a) room temperature, (b) 125°C, (c) 150°C, (d) 200°C and (e) 250°C.



Figure 7. Angular dependence of the transmitted light intensity of an LP He-Ne laser through a liquid crystal cell made from P2NAP100 film between cross polarisers. Applied voltages are 3.2 V (closed squares), 2.8 V (open circles), 2.5 V (open squares) and 0 V (closed circles), respectively.

Table 2. Voltage holding ratio, residual DC voltage, voltage-transmittance and response time value of the liquid crystal cell made from P2NAP100 film.

			Voltage- transmittance (V)			Response time (ms)		
Sample	Voltage holding ratio (%)	Residual DC vol- tage (mV)	V _{th}	V ₅₀	Von	$T_{\rm r}$	T_{f}	$T_{\rm t}$
P2NAP100	98	38	2.34	2.64	3.02	11	21	32

4. Conclusions

The LC alignment properties of 2-naphthoxymethylsubstituted polystyrenes (P2NAP#, where # is the molar content of 2-naphthoxymethyl side groups in %) were found to be affected by the molar content of the 2-naphthoxymethyl side groups. When the molar content of 2-naphthoxymethyl containing monomeric units in the P2NAP# was larger than 57 mol%, homeotropic LC alignment behaviour was observed, which was ascribed to the low surface energy (<47.02 mJ m⁻²). Good E-O properties were observed for the LC cell made from the P2NAP100 film. For example, the VHR, R-DC, $V_{\rm th}$, $V_{\rm on}$ and response time of the LC cell fabricated with the P2NAP100 film were 98%, 38 mV, 2.34 V, 3.02 V and 32 ms, respectively. In addition, the LC cells made from the P2NAP# films maintained their homeotropic LC aligning ability for at least 2 years (from the time when the LC cells were initially made from the P2NAP#), indicating that these LC cells can be used for practical LCD applications including flexible displays.

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