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Liquid crystal alignment properties of polystyrene derivatives containing coumarin side groups

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The liquid crystal (LC) alignment properties of LC cells fabricated with films of polystyrene derivatives containing coumarin side groups (P7COU#, where # is the molar content of coumarin side groups in %) were investigated. Good optical transparency of the P7COU# films was observed in the visible light region. LC cells fabricated with rubbed P7COU# films with more than 51 mol.% of coumarin-containing monomeric units exhibited LC alignment perpendicular with respect to the rubbing direction with a high azimuthal anchoring energy (1×10^{-5} – 7×10^{-5} J m⁻²). LC cells made from rubbed P7COU# films with more than 65 mol.% of coumarin-containing monomeric units exhibited a shift of the LC alignment direction by approximately 10° compared with those made from a polystyrene film. The electro-optical performance of the LC cells fabricated with the rubbed P7COU82 film was found to be as good as that of the LC cells fabricated with a rubbed polyimide film.

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

1. Introduction

The uniform alignment of liquid crystals (LCs) on polymeric substrates is very important for the fabrication of stable LC cells in LC devices, including flexible devices (1–6). The electro-optical characteristics and uniform aligning ability of LC cells made from alignment layers are important parameters, which strongly influence the dynamic performance of LC displays (7, 8). Rubbed polyimide (PI) surfaces are the most commonly used polymeric alignment layers to obtain stable LC cells (9–20). 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 the manufacture of flexible plastic LC devices (21, 22). Therefore, an organic soluble polyimide with a baking temperature of about 120°C was developed for flexible LC display applications (23). We believe that a new approach is needed for the development of LC alignment layers with a lower processing temperature for use in various flexible LC display applications.

Polystyrene (PS) surfaces producing homogenous LC alignment perpendicular with respect to the rubbing direction can be prepared at low temperatures suitable to the fabrication of flexible displays (24–31). However, it is generally accepted that the anchoring stability of the LC cells fabricated with rubbed PS films is not good enough to produce reliable LC alignment layers; the homogeneous LC alignment in these LC cells cannot be sustained for

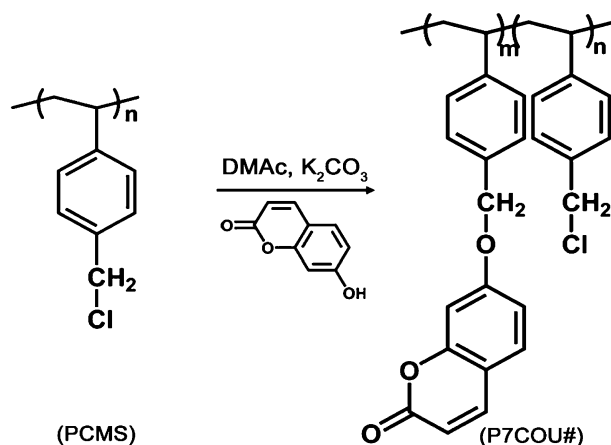
more than one day (29–31). Recently, we found that PS derivatives containing coumarin side groups can produce very stable LC alignment layers through the rubbing process and that these layers can be produced at a low temperature using the casting method (32). In this study, the LC alignment properties and electro-optical characteristics of the LC cells made from these polymers were investigated for possible applications in LC displays.

2. Experimental

Preparation of polystyrene derivatives containing coumarin side groups (P7COU#)

Figure 1 shows the synthetic route to the polystyrene derivatives containing coumarin side groups (P7COU#, where # is the molar content of coumarin side groups in %). Polymers with different degrees of substitution were obtained through the reaction of polychloromethylstyrene (PCMS) with 7-hydroxycoumarin by varying the amounts of 7-hydroxycoumarin in *N,N'*-dimethylacetamide (DMAc). The average molecular weights (M_n) of these synthesised polymers were always larger than 30 000 according to GPC measurements. The maximum degree of substitution of PCMS was 82%, when 180 mol.% of 7-hydroxycoumarin was used at 80°C for 3 h. The physical properties of P7COU# are summarised in Table 1; more detailed information about the synthesis and characterisation is provided elsewhere (32).

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Where # is the molar content of coumarin side groups in %

Figure 1. Synthetic route for the P7COU# films.

Film preparation and LC cell assembly

Solutions of the P7COU#, PCMS and polystyrene in CHCl_3 (2 wt%) were prepared and a polyimide solution (SE-5291, Nissan Chemical Co. Ltd.) was used as received. These solutions were filtered through a PTFE membrane with a pore size of $0.45 \mu\text{m}$. The film thickness was measured using an ellipsometer (Auto gain ellipsometer L116B, Gaertner). UV-visible spectra were acquired using a Perkin Elmer Lambda 20 spectrometer. For the UV-visible spectroscopy of P7COU#, the polymer films were prepared by spin-coating 2 wt% P7COU# solutions in CHCl_3 onto ITO-coated glass substrates at 2000 rpm for 30 s. The film thickness was found to be about 50 nm within an experimental error of $\pm 5\%$. Polarised FTIR measurements of the rubbed polymer films with a rubbing density of 100 were

carried out using a Perkin Elmer Spectrum 2000 spectrometer in transmission mode equipped with a polariser. The polymer films for the FTIR measurement were prepared by spin coating (1000 rpm, 30 s) a 3 wt% polymer solution in toluene onto a silicon wafer. The polymer films were rubbed using a rubbing machine (RMS-50-M, Nam Il Optical Components Corp.). The rubbing density equation is: $L/l = N[(2\pi rn/60v) - 1]$, 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) of the substrate stage (18, 19).

The photoalignment of the films was also carried out by irradiating them under a 200 W high-pressure mercury lamp (66902, ThermoOriental Instruments) equipped with an interference filter at 290 nm and attached to a power supply (69911, ThermoOriental Instruments). A dichroic polariser was used to irradiate the films with linearly polarised UV (LPUV) light. The intensity of the LPUV light was 6 mW cm^{-2} . The exposure dose of irradiated LPUV light was measured with a UV detector (UM-10, Minolta) equipped with a sensor. Twisted nematic (TN) and antiparallel LC cells were fabricated using the rubbed or photoirradiated polymer films in an area of $1.5 \times 1.5 \text{ cm}^2$ ITO-coated glass slides. The TN LC cells were fabricated by assembling the films together orthogonally with respect to the rubbing direction for the rubbed film or the polarisation direction of LPUV light for the photoaligned films using spacers with a thickness of $6.5 \mu\text{m}$. The antiparallel LC cells were constructed by assembling the films together so that they were antiparallel with

Table 1. Reaction conditions and results in the synthesis of polymers and pretilt angle and LC alignment direction of the LC cells made from rubbed or photoirradiated polymer films.

Polymer designation	7-Hydroxy coumarin /mol. %	Molar content of coumarin side groups /%	M_n^a	Pretilt angle / $^\circ$ ^b	LC alignment direction with respect to the rubbing direction or polarisation direction of the incident light / $^\circ$
PS	0	0	37 000	0	$90 \leftrightarrow 270$
PCMS	0	0	34 000	0	$90 \leftrightarrow 270$
P7COU16	30	16	34 000	0	$90 \leftrightarrow 270$
P7COU31	50	31	36 000	0	$90 \leftrightarrow 270$
P7COU51	60	51	35 000	0.1	$95 \leftrightarrow 275$
				0.1^c	$0 \leftrightarrow 180^c$
P7COU65	100	65	38 000	0.1	$100 \leftrightarrow 280$
				0.1^c	$0 \leftrightarrow 180^c$
P7COU82	180	82	41 000	0.1	$100 \leftrightarrow 280$
				0.1^c	$0 \leftrightarrow 180^c$
PI				3.5	$0 \leftrightarrow 180$

^aObtained from GPC using CHCl_3 as solvent with respect to monodisperse polystyrene as standards. ^bMeasured from the antiparallel LC cell fabricated with rubbed or photoirradiated polymer films using crystal rotation method. ^cMeasured from the LC cells fabricated with photoirradiated P7COU# films.

respect to the rubbing direction using spacers with thicknesses of 50 μm and 6.5 μm . These cells were filled with nematic LC, ZLI-5900-000 (Merck Co., $K_{22}=2.3759 \times 10^{-12}$, $n_e=1.6327$, $n_o=1.5011$, and $\Delta\varepsilon=15.5$, where n_e , n_o and $\Delta\varepsilon$ represent the extraordinary refractive indexes, ordinary refractive indexes and dielectric anisotropy, respectively), in an isotropic state, in order to avoid creating flow alignment through capillary action, and were then sealed with epoxy glue. The cell gap was measured before LC filling using a spectrophotometer (Ocean Optics Inc., S 2000).

Characterisation of LC cells

The LC alignment direction of the antiparallel LC cells was investigated as a function of the rotation angle of the samples by measuring the angular dependence of the absorbance of a dichroic dye (Disperse Blue 1, which was dissolved at 1 wt% in ZLI-5900-000) using an optical apparatus equipped with a He-Ne laser, a polariser and a photodiode detector (19). The pretilt angles of the antiparallel LC cells were measured using a crystal rotation method (33). The azimuthal anchoring energy was measured from the TN LC cells using an optical apparatus equipped with a photoelastic modulator (PEM-90 TM, HINDS instruments) with a modulation frequency of 50 kHz, a polariser and analyser pair, photodiode detector, lock-in amplifier (Stanford Research Systems, SR 830 DSP), digital voltmeter (Keithley, Model 2000 multimeter) and stage controller (Mark-202, Sigma Koki Co.) (34).

The response time and voltage-transmittance (V - T) curves were measured from the TN LC cells using the same method as that reported in the literature (8). The threshold voltage (V_{th}) and driving voltage (V_{on}) in the V - T curve are defined as the voltages at which the transmittance is decreased to 90% and to 10% of the initial transmittance value, respectively. The rise (T_r) and fall (T_f) response times for white-to-black and black-to-white changes, respectively, are defined as the time to transition from 10% to 90% transmittance and vice versa (8). For the TN LC cell, they are given by

$$V_{th} = \pi \sqrt{\frac{K}{\varepsilon_0 \Delta\varepsilon}}, \quad (1)$$

$$T_r = \frac{\gamma_1 d^2}{\varepsilon_0 \Delta\varepsilon_{LC} V^2 - \pi^2 K}, \quad (2)$$

$$T_f = \frac{\gamma_1 d^2}{\pi^2 K}, \quad (3)$$

$$K = \frac{4K_{11} + (K_{33} - 2K_{22})}{4}, \quad (4)$$

where γ_1 is the rotational viscosity of the LC, d is the LC cell gap, V is the applied voltage, $\Delta\varepsilon_{LC}$ is the dielectric anisotropy of the LC, and K , the elastic constant of the LC, is a combination of the elastic constants K_{11} , K_{22} , and K_{33} for splay, twist, and bend, respectively.

3. Results and discussion

Figure 2 shows the UV-visible transmittance spectra of the P7COU# films with a thickness of about 50 nm. As the molar content of coumarin side groups increases, the transmittance of the polymer films slightly decreases. For example, as the molar content of coumarin side groups increases from 51% to 82%, the transmittance of the P7COU# films slightly decreases from 96.7% to 94.3% at 500 nm. However, the optical transparency in the visible light region (400–700 nm) of all of the films is still good enough for them to be used as optical materials for flexible LC devices (35).

UV-visible transmittance measurements of the P7COU82 film were performed after heating to temperatures between room temperature and 250°C to investigate the optical stability of the polymer. Figure 3 shows the UV-visible transmittance spectra of the P7COU82 film after heating for a sufficient time (2 h) at various temperatures. The good optical transmittance of the P7COU82 film is maintained when the temperature is lower than 150°C. When the temperature increases to above 200°C, however, a decrease of the optical transmittance is observed. For

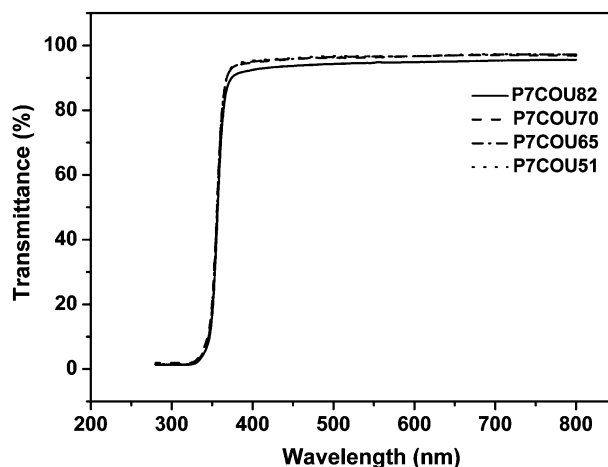


Figure 2. UV-visible transmittance spectra of P7COU# films for various amounts of coumarin side group monomeric units.

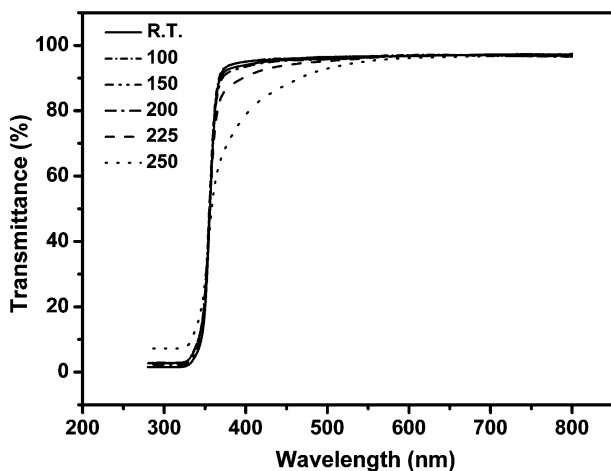


Figure 3. UV-visible transmittance spectra of P7COU82 films after heating the samples at different temperatures for 2 h.

example, the transmittance of the P7COU82 film at 400 nm decreases from 90.4% to 78.7% as the temperature is increased from 200°C to 250°C.

The thermal stability of the LC cells made from the rubbed P7COU82 film was also investigated when they were heated for 10 min at various temperatures. The LC aligning ability of the LC cells made from the rubbed P7COU82 film was sustained at temperatures below 150°C, whereas LC alignment defects were observed when the temperature was higher than 170°C. Therefore, both the optical transparency of the rubbed P7COU82 film and LC aligning ability of the LC cells made from it can be maintained up to 150°C. At room temperature, the aligning ability of the cells made from the rubbed P7COU82 film has been maintained for the more than two years since they were made.

Polar diagrams of the absorbance of a dichroic dye (Disperse Blue 1) in the LC cells fabricated using the rubbed P7COU# films with a rubbing density of 100 and photoirradiated P7COU# films with UV exposure doses of 16 J cm^{-2} were obtained in order to study the effect of the side group content on the LC alignment property (Figure 4). It was found that the maximum absorbances of the LC cells made from the

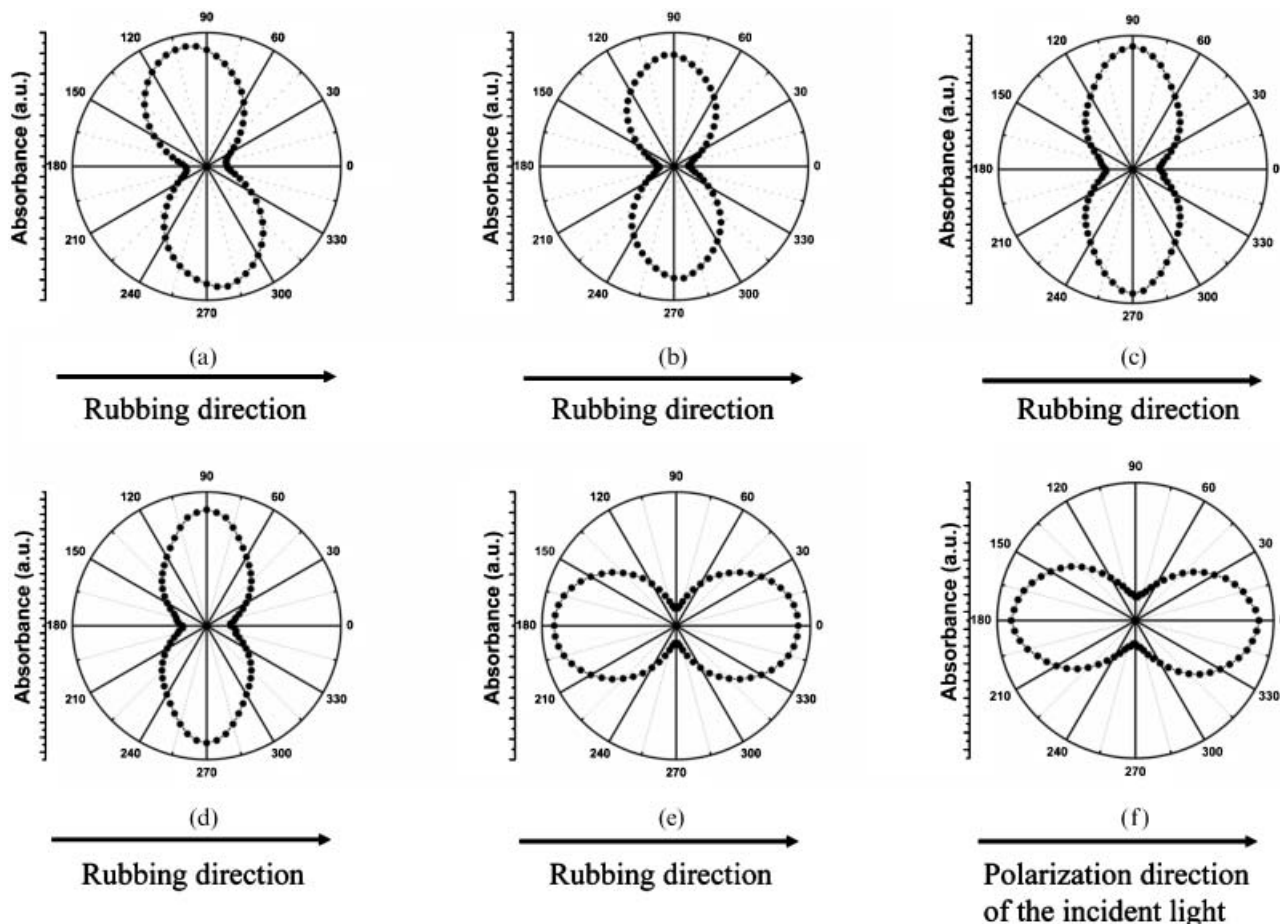


Figure 4. Polar diagrams of the absorbance of Disperse Blue 1 in antiparallel LC cells fabricated with polymer films as a function of angle of the samples: (a) rubbed P7COU82; (b) rubbed P7COU51; (c) rubbed P7COU16; (d) rubbed PS; (e) rubbed PI; (f) photoirradiated P7COU82.

rubbed films of P7COU16, P7COU51 and P7COU82 are observed along the $90^\circ \leftrightarrow 270^\circ$, $95^\circ \leftrightarrow 275^\circ$, and $100^\circ \leftrightarrow 280^\circ$ directions, respectively, indicating that the increase in the molar content of the coumarin side groups tilts the LC alignment direction more. The LC cells made from rubbed films of PS and PI showed maximum absorbance along the $90^\circ \leftrightarrow 270^\circ$ and $0^\circ \leftrightarrow 180^\circ$ directions, respectively.

It has been shown that the LC alignment direction depends on the molecular direction of alignment of the polymer films after the rubbing process (36). Therefore, the LC alignment (maximum absorbance along the $90^\circ \leftrightarrow 270^\circ$) perpendicular with respect to the rubbing direction of the polystyrene film was ascribed to the orientation of the phenyl side groups perpendicular with respect to the rubbing direction and this is confirmed by the FTIR dichroic spectra reported in the literature (29–31). The maximum absorbance direction of $100^\circ \leftrightarrow 280^\circ$ for P7COU82 deviated by 10° from that for the polystyrene film, which indicates that the side chain orientation of P7COU82 is not identical to that of polystyrene. For further comparison, we also measured the polar diagrams of the LC cells made from the photoaligned P7COU# films. The polar diagram of the LC cell made from the photoirradiated P7COU82 showed homogeneous planar LC alignment behaviour with a maximum absorbance along the $0^\circ \leftrightarrow 180^\circ$ direction, which is parallel with respect to the polarisation direction of the incident light, as reported in the literature (37, 38). Although not shown, the LC cells made from photoirradiated P7COU65 and P7COU51 films showed maximum absorbance along the $0^\circ \leftrightarrow 180^\circ$ directions. The LC alignment (maximum absorbance along the $0^\circ \leftrightarrow 180^\circ$) parallel with respect to the polarisation direction of the incident light on the photoirradiated P7COU# films was not changed according to the UV exposure energy. The polar diagrams showing the good orientation of the LCs on the photoirradiated P7COU# films (with a coumarin monomeric unit content smaller than 31 mol.%) could not be measured due to the poor LC alignment. The IR spectrum (Figure 5(a)) of the rubbed P7COU82 film with a rubbing density of 100 on Si wafer, with IR monitoring light along the $30^\circ \leftrightarrow 210^\circ$ and $120^\circ \leftrightarrow 300^\circ$ directions with respect to the rubbing direction, exhibits a dichroic aspect. Figures 5(b) and 5(c) clearly show that the maximum absorbance directions of the C=O (1737 cm^{-1}) and C=C (1614 cm^{-1}) groups are along the $120^\circ \leftrightarrow 300^\circ$ and $90^\circ \leftrightarrow 270^\circ$ directions, respectively. The molecular interactions of the LC and the side groups of P7COU82 having such orientation lead to a tilt of about 10° from the phenyl side group direction of polystyrene.

The pretilt angles of the antiparallel LC cells made from the rubbed or photoirradiated polymer films were measured (Table 1). The pretilt angles of the LC cells made from the rubbed P7COU# films were found to be very low, i.e. about $0\text{--}0.1^\circ$. For comparison, the pretilt angle of the LC cell made from the rubbed polyimide film was found to be approximately 3.5° . We also measured the pretilt angles of the LC cells made from the photoaligned P7COU# films. The pretilt angles of the LC molecules on the photoirradiated P7COU# films (with coumarin monomeric unit content smaller than 31 mol.%) could not be measured due to poor LC alignment. The pretilt angles of about 0.1° on the photoirradiated P7COU# films (with a coumarin monomeric unit content greater than 51 mol.%) were obtained. We believe that these P7COU# films (with a coumarin monomeric unit content greater than 51 mol.%) with a high azimuthal anchoring energy could be a good candidate as an alignment layer for flexible LC display applications, such as IPS (in-plane switching) mode applications, because they have very low LC pretilt angles (39–41). Previously, we measured the azimuthal anchoring energies of the TN LC cells made from the rubbed P7COU# films (32). We found that the azimuthal anchoring energy value increases as the molar content of coumarin side groups increases; they were 5.0×10^{-7} , 9.0×10^{-7} , 1.5×10^{-5} , 4.0×10^{-5} and $7.0 \times 10^{-5}\text{ J m}^{-2}$ for the LC cells made from the rubbed P7COU16, P7COU31, P7COU51, P7COU65 and P7COU82 films, respectively. The anchoring energy of the LC cells made from the photoaligned P7COU82 film, $1 \times 10^{-5}\text{ J m}^{-2}$, was found to be smaller than that of the LC cells made from the rubbed P7COU82 film. We also found that the electro-optical performance of the LC cells made from the rubbed P7COU82 film was better than that of the LC cells made from the photoaligned P7COU82 film, as shown below.

The electro-optical performance of LC cells having the same cell gap of about $7\text{ }\mu\text{m}$ was determined by measuring the V – T curves and response times using the same conditions (see Figure 6 and Table 2). A stable V – T curve was observed for the TN LC cell fabricated with the rubbed PS film. The V_{th} , V_{on} and response time of the rubbed PS film were 1.11 V, 1.71 V and 40 ms, respectively. However, these good electro-optical properties were lost after just one day, possibly due to the low anchoring stability of the rubbed PS films (29–31). Therefore, rubbed PS films cannot be used for practical LC display applications. LC cells made from the rubbed P7COU82 film were found to have good electro-optical properties (Figure 6(b)), which were maintained for at least two years (from the initial time of fabrication of the LC

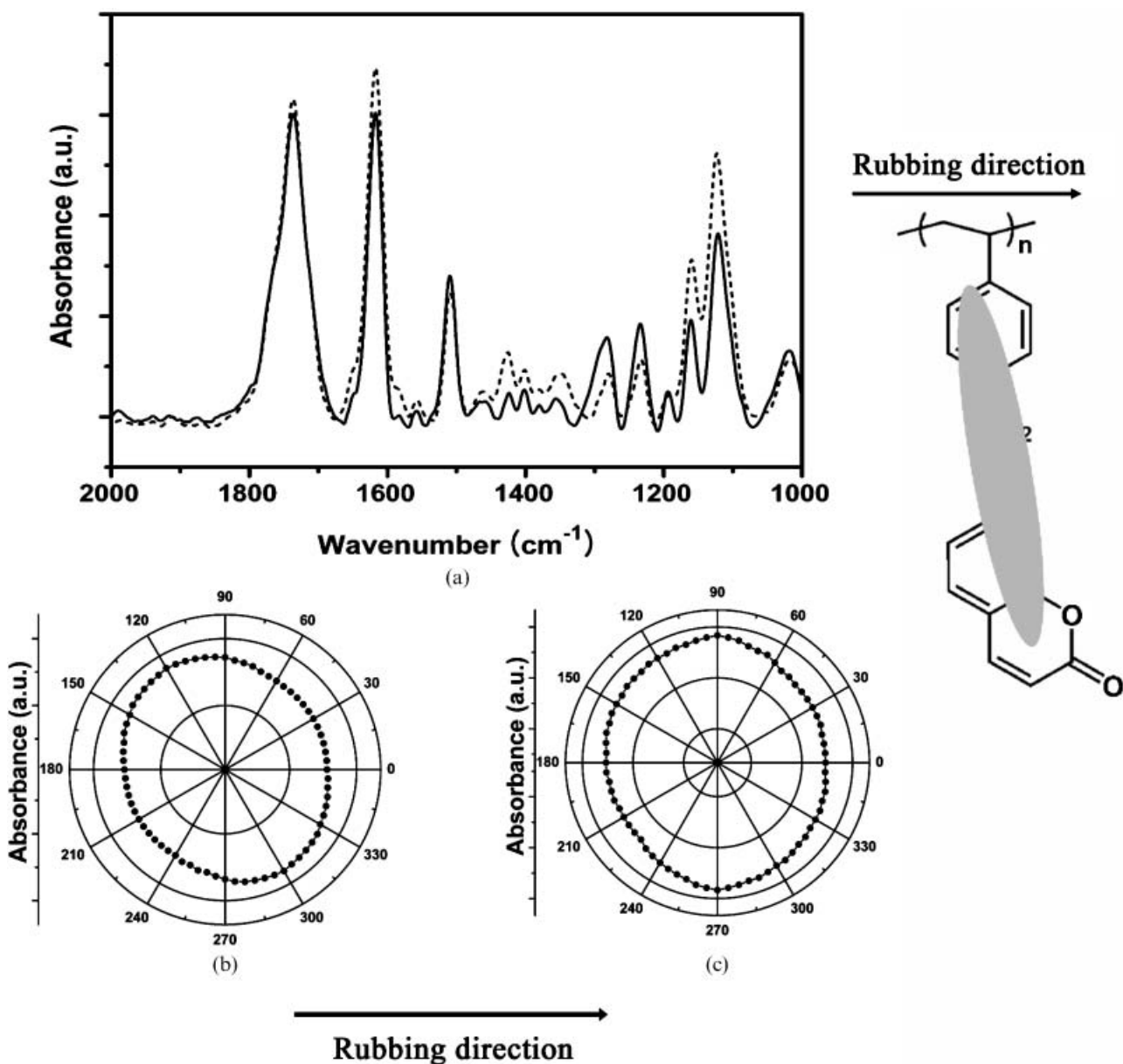


Figure 5. (a) FTIR dichroic spectra of rubbed P7COU82 on Si-wafer film with a rubbing density of 100. Solid and dotted lines indicate the results obtained from the IR source along $30^\circ \leftrightarrow 210^\circ$ and $120^\circ \leftrightarrow 300^\circ$ directions, respectively, with respect to the rubbing direction. Polar diagram of specific vibrational IR peaks of rubbed P7COU82 film measured as a function of rotation angle of polarizer: (b) $\nu(\text{C}=\text{O})$ at 1737 cm^{-1} ; (c) $\nu(\text{C}=\text{C}_{\text{benzene}})$ at 1614 cm^{-1} .

cells from the rubbed P7COU82). The V_{th} , V_{on} , and response time values of the rubbed P7COU82, 1.02 V, 1.62 V and 36 ms, respectively, are close to those of rubbed PI, 0.91 V, 1.52 V and 35 ms, respectively. It has been reported that LC alignment films having larger pretilt angles have faster response times in various LCD modes, such as the TN, OCB (optically compensated bend) and NBB (no-bias-bend) modes (42–44). It has also been reported that LC cells made from an alignment layer having a strong LC anchoring energy have a slow response time (45, 46). Therefore,

the combination of these two factors makes the rubbed P7COU82 and PI films have similar electro-optical properties.

For comparison of the rubbing and photoalignment effects, we also measured the electro-optical performance of the TN LC cell made from the photoaligned P7COU82 film. The LC cell made from the rubbed P7COU82 film has a faster response time and lower operating voltage than those of the LC cell made from the photoirradiated P7COU82 film, as reported by Seo *et al.* (47, 48). For example, the V_{th}

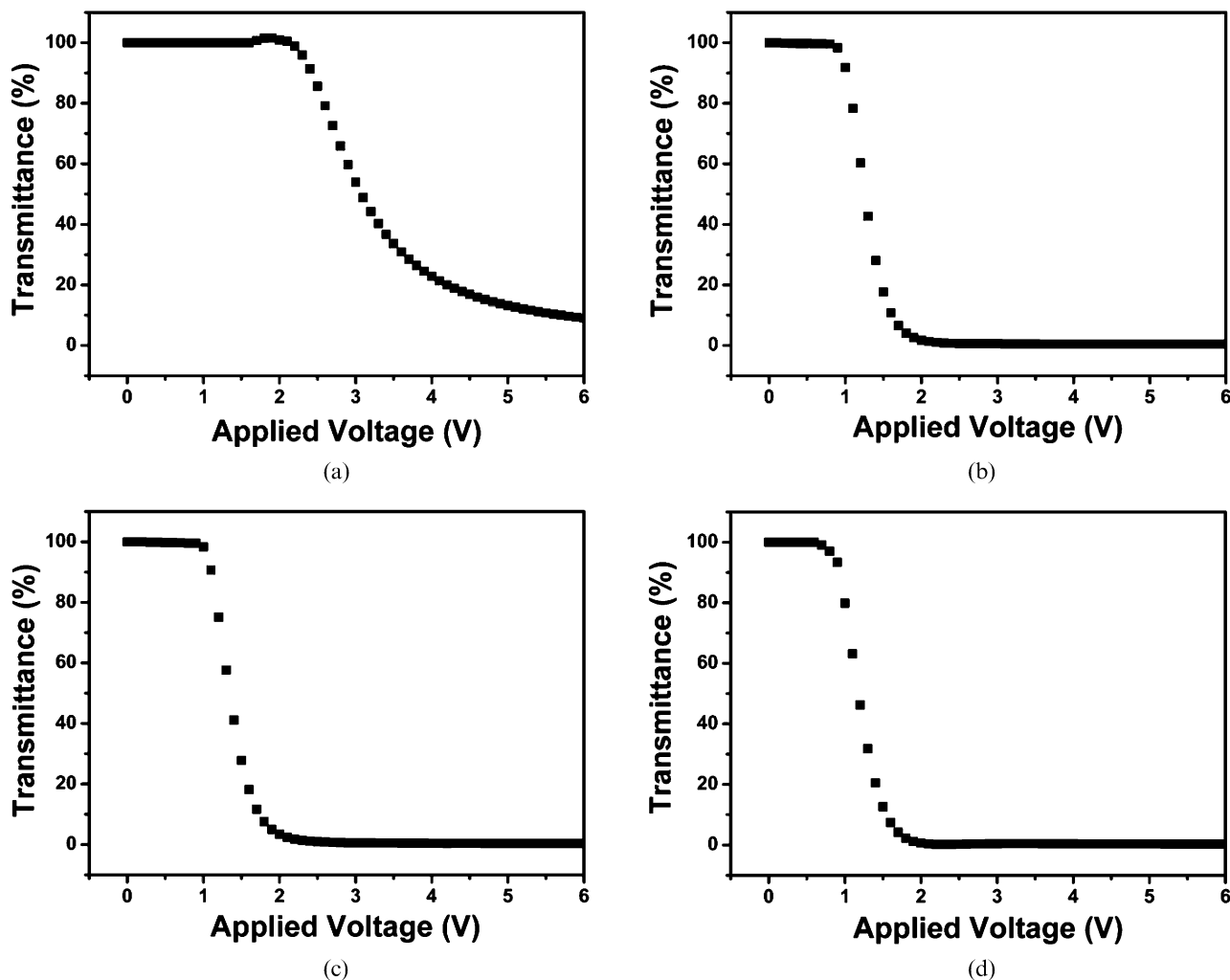


Figure 6. Voltage–transmittance curves of the LC cells fabricated with (a) photoirradiated P7COU82 film, (b) rubbed P7COU82 film, (c) rubbed PS film and (d) rubbed PI film.

and V_{on} values of the LC cell fabricated with the photoirradiated P7COU82 film ($V_{th}=2.42$ V and $V_{on}=5.70$ V) were higher than those of the LC cell fabricated with the rubbed P7COU82 film ($V_{th}=1.02$ V and $V_{on}=1.62$ V). In addition to the operating voltage values, a backflow bounce effect on the LC cell fabricated with the photoirradiated P7COU82 film was observed (49, 50). The backflow bounce effect from the photoirradiated P7COU82 film is probably related to the slower response time

(42, 51); the response times ($T_r=28$ ms and $T_f=58$ ms) for the photoirradiated P7COU82 film are slower than those ($T_r=12$ ms and $T_f=24$ ms) for the rubbed P7COU82 film.

4. Conclusions

The LC alignment properties, such as the LC alignment direction and azimuthal anchoring energy, were found to be affected by the molar content of the

Table 2. Voltage–transmittance and response time values of the LC cells made from alignment films.

Sample	Voltage–transmittance /V			Response time /ms		
	V_{th}	V_{50}	V_{on}	T_r	T_f	Response time
Photoirradiated P7COU82	2.42	3.15	5.70	28	58	86
Rubbed P7COU82	1.02	1.24	1.62	12	24	36
Rubbed PS	1.11	1.34	1.71	15	25	40
Rubbed PI	0.91	1.16	1.52	12	23	35

coumarin side groups in the polystyrene derivatives containing coumarin side groups (P7COU#, where # is the molar content of coumarin side groups in %). For example, as the molar content of coumarin side groups increased from 16% to 82%, the LC alignment direction shifted from an angle of 90° (perpendicular) to an angle of 100° with respect to the rubbing direction, and the azimuthal anchoring energy increased from 10^{-7} – 10^{-8} Jm⁻² to 7×10^{-5} Jm⁻². The electro-optical properties, such as the threshold voltage, driving voltage and response time, of the LC cell fabricated with the rubbed P7COU82 film was as good as that of the LC cell fabricated with the rubbed PI film. In addition, the LC cells made from the rubbed P7COU82 film maintained their aligning ability and electro-optical properties for at least two years (from the time when the LC cells were initially made from the rubbed P7COU82), indicating that these cells can be used for practical LC display applications including flexible displays.

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