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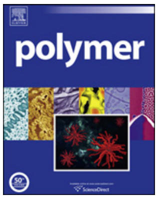

Polyimide blend alignment layers for control of liquid crystal pretilt angle through baking

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ABSTRACT

Polyimide films were used for liquid crystal (LC) alignment layers to control LC pretilt angles over the full range (8°–90°). The pretilt angles could be controlled using polyimide films prepared from polyamic acid for vertical LC alignment and using polyimide blend films prepared from two types of polyamic acids, one for vertical LC alignment and the other for planar LC alignment, by changing the baking times ranging from 40 to 180 min at 230 °C. The polyimide blend film could control the pretilt angle better than the polyimide prepared from just one polymer component. The LC alignment behavior was well correlated with the wettability of the polyimide films due to the fragmentation of the long alkyl side group on the polyimide surfaces by the baking process.

1. Introduction

Continuous control of the pretilt angle of liquid crystal (LC), including intermediate pretilt angle between homogeneous planar and vertical, has been extensively studied due to the scientific and technical interest in liquid crystal displays (LCDs). In particular, it is desired to improve image quality and electro-optical (E-O) performance. Recently, various techniques have been developed to control the continuous pretilt angle of LCs over a wide range on the alignment layer surfaces, so as to be applied in several LCD modes; rubbing of the polyimide surfaces [1,2], photoirradiation of the photosensitive alignment layer [3], ion beam techniques using organic/inorganic alignment layer [4–7], mixing of two kinds of polyimides for planar and vertical LC alignment [8–11], stacking of the alignment layers [12–14], nano/micro-structured patterns using lithographic techniques [15,16], and inkjet printed and air-buffed polyimide surface [17]. Recently, we reported that pretilt angles on polyimide films can be controlled by changing the baking temperature of the polyimide films from 230 to 290 °C, rubbing strength, and composition of the blends [11,18–21].

In this study we found that the pretilt angles could be continuously controlled further by changing the baking time at different baking temperatures. Moreover, changes of the pretilt angles were found to be strongly related to changes of the surface properties. The surface properties of polyimide films giving different pretilt angles were investigated using fourier transform infrared spectroscopy (FTIR), contact angle measurements, electron spectroscopy for chemical analysis (ESCA), and atomic force microscopy (AFM).

2. Experimental section

2.1. Materials

Pre-imidized alignment agents were kindly supplied by Nissan Chemical Industries and a nematic LC (5CB) was purchased from Merck Co. All other reagents and solvents were used as received.

2.2. Film preparation and LC alignment process

RN-1175, SE-1211, and a mixture (75 wt-% of RN-1175 and 25 wt-% of SE-1211) were dissolved in varnish as supplied by the manufacturer to prepare 4 wt-% polymer solutions. The alignment agents were spin coated (1800 rpm, 9 s) onto 1.5 cm × 1.5 cm indium tin oxide (ITO) coated glass substrates. The films were prebaked at 80 °C for 30 min and then were fully baked at 200, 230, or 250 °C for 40–180 min. The polymer films were rubbed using a rubbing machine (RMS-50-M, Nam Il Optical Components Corp.). The rubbing density equation is written as $L/l = N[(2\pi rn/60v)-1]$,

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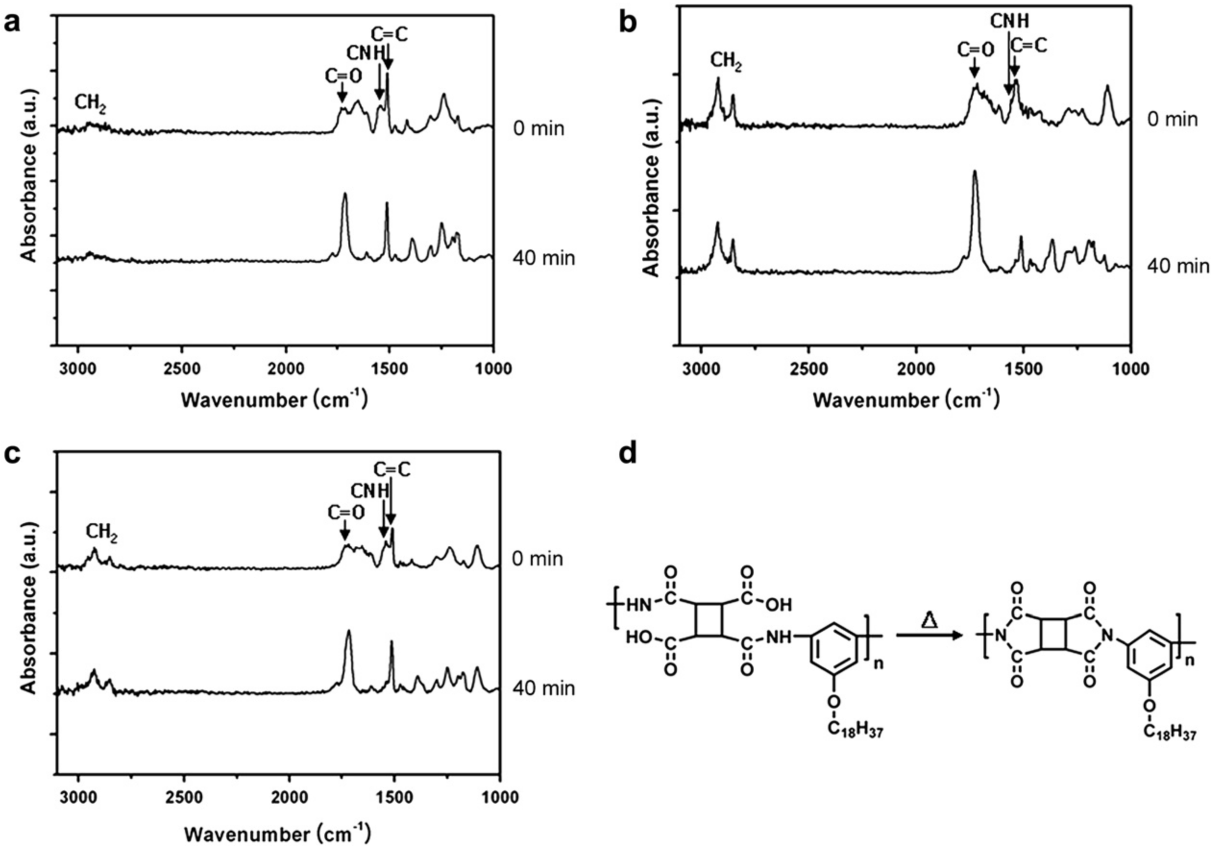

Fig. 1. FTIR spectra of the (a) RN-1175, (b) SE-1211, and (c) polyimide blend films prepared using different the baking times at 230 °C, and (d) the changes of amic acid to imide groups in SE-1211.

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 [22,23]. For this experiment, all of the polyimide films were rubbed with a rubbing density of 100.

2.3. LC cell assembly

Antiparallel LC cells were fabricated using the rubbed polymer films onto ITO coated glass slides substrates. The antiparallel LC cells were constructed by assembling the rubbed polyimide films together antiparallel with respect to the rubbing direction using spacers with thicknesses of 4.5 and 50 μm. The fabricated LC cells were filled with a nematic LC, 5CB (Merck Co., 4-cyano-4′-pentylbiphenyl, $n_e = 1.7360$, $n_o = 1.5442$, and $\Delta\varepsilon = 14.5$, where n_e, n_o, and $\Delta\varepsilon$ represent extraordinary refractive indexes, ordinary refractive indexes, and dielectric anisotropy, respectively), in the isotropic state in order to avoid creating flow alignment by the capillary action. The manufactured LC cells were sealed with epoxy.

2.4. Instrumentation

The polymer films for the transmission, attenuated total reflection (ATR) FTIR (Thermal Electron Corp., Nicolet 5700) measurement were prepared by spin coating (1800 rpm, 9 sec) onto a silicon wafer. The film thickness was measured using a Nanospec/AFT (K-MAC, ST-2000). The film thicknesses were found to be in the range of 2900–3290 Å. FTIR spectra were recorded 4 cm^{-1} resolution and interferograms were accumulated 64 times for transmission mode, 256 times for ATR mode. The static contact angles of distilled water and methylene iodide on polymer films were determined with a Kruss DSA10 contact angle analyzer equipped with drop shape analysis software. The surface energy value was calculated using the Owens–Wendt's equation as

$$\gamma_{sl} = \gamma_s + \gamma_l - 2\left(\gamma_s^d \gamma_l^d\right)^{1/2} - 2\left(\gamma_s^p \gamma_l^p\right)^{1/2}$$

where γ_l is the surface energy of the liquid, γ_{sl} is the interfacial energy of the solid/liquid interface, γ_s is the surface energy of the solid, γ_l^d and γ_l^p are known for the test liquids, γ_s^d and γ_s^p can be calculated from the measured static contact angles [24]. The electron spectroscopy for chemical analysis (ESCA) experiments were performed in an UHV multipurpose surface analysis system (Thermo Scientific, SIGMA PROBE, UK) operating at base pressures $<10^{-10}$ mbar. The photoelectron spectra were excited by an Al K_α (1486.6 eV) anode operating at constant power of 100 W (15 kV and 10 mA). During the spectra acquisition, the constant analyzer energy (CAE) mode was employed at a pass energy of 40 eV and a step of 0.1 eV at a take-off angle of 90°. The binding energy (BE) scale was calibrated from the hydrocarbon contamination using the C_{1s} peak at 285.0 eV. Core peaks were analyzed using a linear-type background, and peak positions and areas were obtained using fitting program (Thermo Scientific, Avantage Data System) by a least-square fitting of model curves (70 % Gaussian, 30 % Lorentzian) to the experimental data. The surface morphology of the polymer films in an area of 3 μm × 3 μm was examined using atomic force microscopy (AFM, PSIA, XE-150) in contact mode (spring constant of the cantilever: 0.6 N/m, scan rate: 1 Hz). The LC alignment direction of the antiparallel LC cells was investigated by

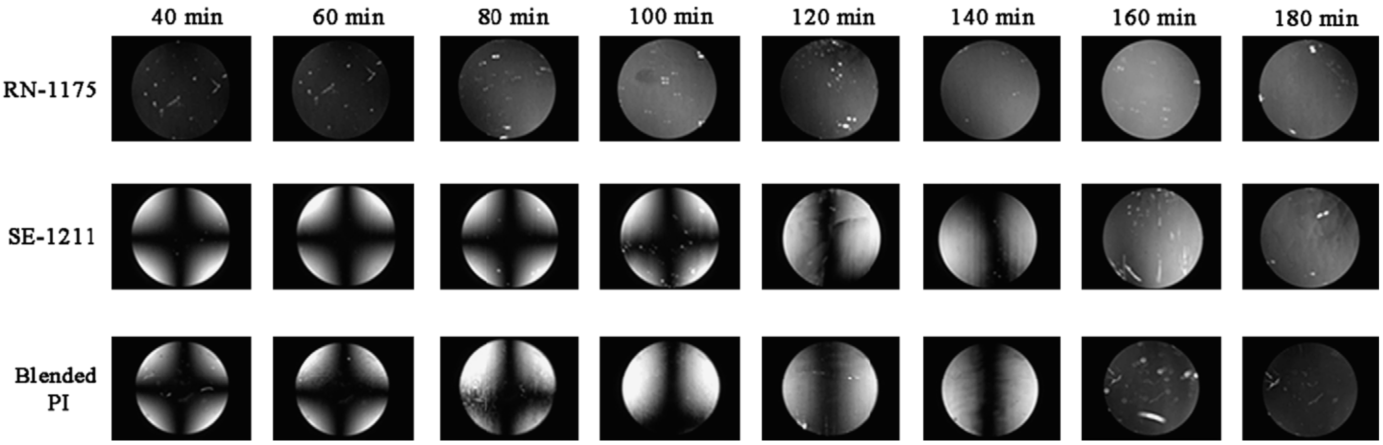

Fig. 2. Conoscopic POM images of the LC cells made from RN-1175, SE-1211, and polyimide blend films baked at 230 °C according to the baking time.

measuring the angular dependence of absorbance of dichroic dye (disperse blue 1, Aldrich), which was dissolved in 5CB at a concentration of 1 wt-%, using optical apparatus equipped with a He–Ne laser, a polarizer, and a photodiode detector as a function of rotation angle of samples [23]. The pretilt angle of LCs with respect to the planar direction in antiparallel LC cell was measured by modified crystal rotation method using PAMS series (Sesim Photonics Technology, Korea), as used in previous several papers [6,25,26]. The cell gap was measured before filling the LCs using a spectrophotometer (Ocean Optics Inc., S2000). The polarized optical microscopy (POM) images of LC cell were observed from an optical microscopy (Nikon, ECLIPSE E600 POL) equipped with a polarizer and digital camera (Nikon, COOLPIX995).

3. Results and discussion

The SE-1211 having a long *n*-octadecyl side group was used for vertical LC alignment layer [27–29], while the detailed chemical structure of the RN-1175 used for homogeneous planar LC alignment layer remains proprietary. In this experiment polyimide blend films were prepared from a mixture of 75 wt-% of RN-1175 and 25 wt-% of SE-1211 because LC alignment had been controlled continuously from vertical to homogeneous planar for the LC cells prepared using the polyimide blend films baked from 230 to 290 °C for 50 min in our previous experiments [21]. This time we found that LCs align vertically, in this work the tilt angle of 0° as planar and 90° as vertical, from the polyimide blend films baked at 230 °C for up to 60 min, while the tilt angle decreases continuously from 90° to about 8°, *i.e.* it becomes more planar, when we increase the baking time from 40 to 180 min as will be shown in the later part of this paper.

Fig. 1 shows FTIR spectra of polymer films prepared from RN-1175, SE-1211, and 3:1 mixture of RN-1175 and SE-1211. The FTIR peaks of the films were assigned with the aid of result of other research groups [10,30]. The IR peaks representing stretching vibrational mode of CNH, C═C, and C═O groups were observed at 1536, 1610, and 1726 cm^{-1}, respectively, from the samples without baking (baking time of 0 min). When the baking time was increased to 40 min, C═C and C═O peak remains while the CNH peak at 1536 cm^{-1} disappears, indicating that the amic acid group is transformed into imide group [10,30]. We tried to observe the changes of attenuated total reflection (ATR) FTIR spectra of polymer films obtained using different baking time, while those of polymer films obtained from baking times of 40–180 min were almost identical for each sample within experimental error. Especially we expected the intensity changes of $\mathrm{CH_2}$ peaks at around 2900 cm^{-1}, because the changes of pretilt angles of polyimide films from SE-1211 were ascribed to the cleavage of the alkyl groups [21]. Possibly the resolution of our ATR FTIR spectra is not sufficient to detect the concentration changes of alkyl groups on the polymer surface. Therefore we could not clarify the effect of cleavage of the alkyl groups on the pretilt angle of LCs using ATR FTIR. However the contact angles and ESCA results shown later in the paper indicate the changes of surface properties of the polymer films prepared using SE-1211 with the changes of the baking time. In particular, polarizing optical microscopy (POM) images of the LC cells prepared from the polymer films containing SE-1211 moiety show the changes of the LC alignment behavior according to the baking times.

Fig. 2 shows the conoscopic POM images of antiparallel LC cells fabricated from the polymer films with different baking times. All LC cells from the RN-1175 films heated at different baking times from 40 and 180 min show the homogeneous planar LC alignment having pretilt angles about 8°, as shown in Fig. 3. On the contrary the LC cells fabricated from SE-1211 and the polyimide blend show the change of LC alignment from vertical to homogeneous planar alignment. For example the pretilt angle of the LC cells from SE-1211 decreases from 90° to 8° when the baking time increases from 40 to 180 min. In particular, a very large decrease from

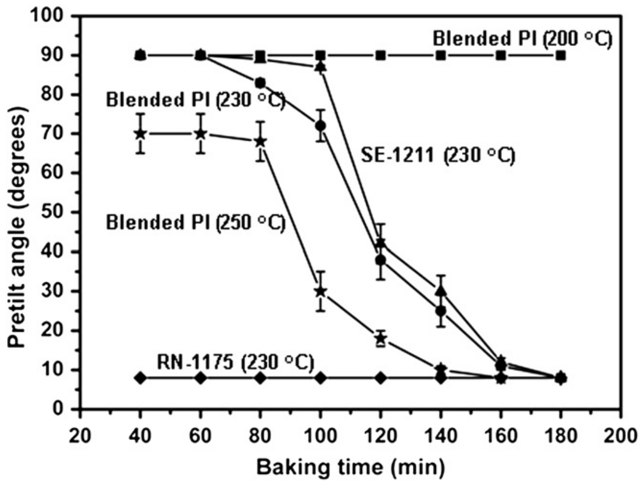

Fig. 3. Pretilt angles of the antiparallel LC cells fabricated with polyimide films as a function of the baking time.

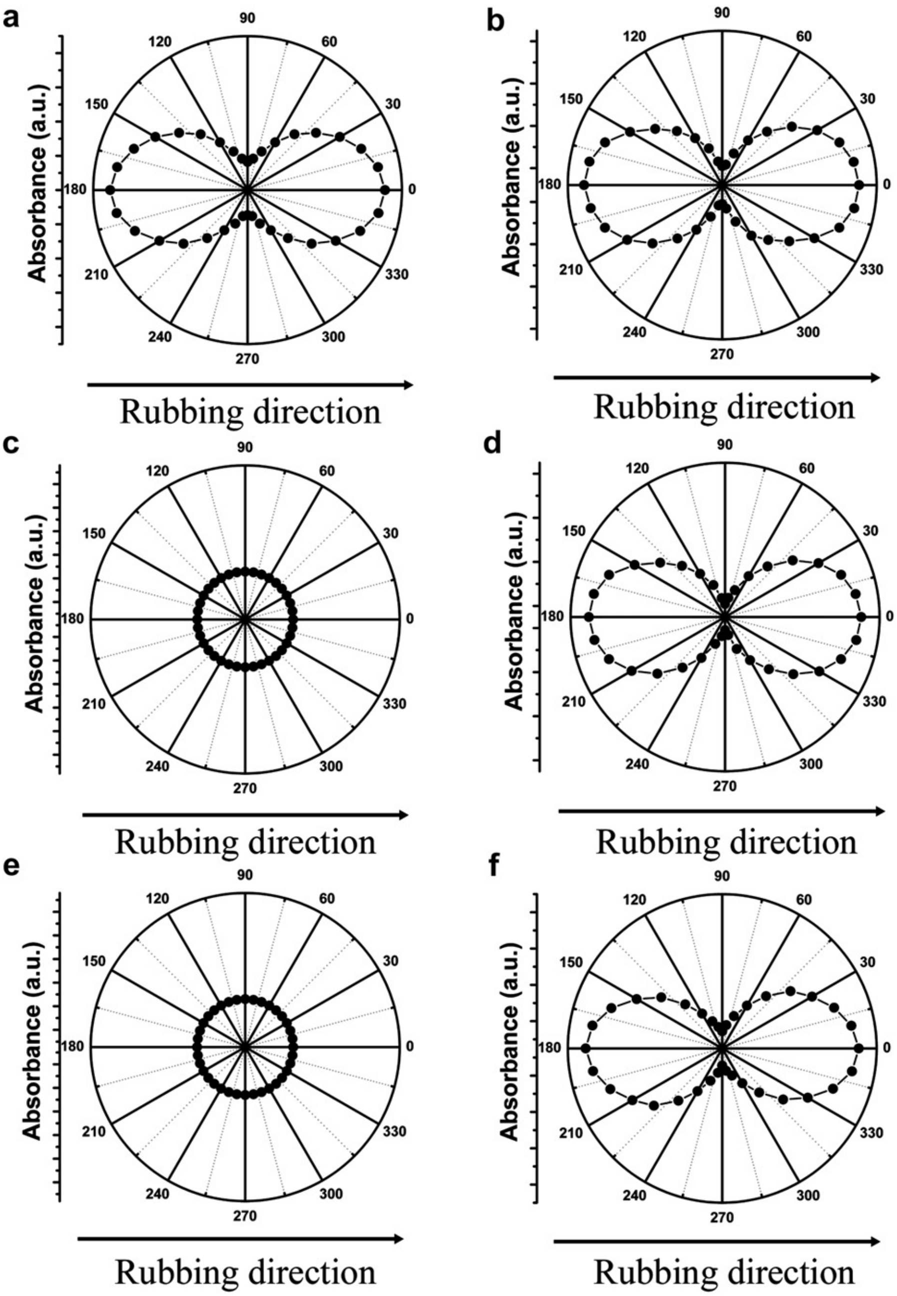

Fig. 4. Polar diagrams of the absorbance of a dichroic dye (disperse blue 1) in the antiparallel LC cells fabricated using the RN-1175 films baked at 230 °C for (a) 40 min, (b) 180 min and SE-1211 films for (c) 40 min, (d) 180 min and polyimide blend films for (e) 40 min, (f) 180 min as a function of rotation angle of the samples.

a pretilt angle of 87° to 42° was observed when the baking time increased from 100 to 120 min. The pretilt angles of the LC cell from the polyimide blend films show a more linear decrease than those from SE-1211; for example the pretilt angles are 83°, 72°, 38°, 25°, and 11° for the baking times of 80, 100, 120, 140, and 160 min, respectively. Therefore the addition of RN-1175 into SE-1211 for the preparation of the alignment layer improves the continuous control of the pretilt angle in the LC cells. We also used the baking temperature of 200 and 250 °C to study the effect of temperature and baking time on the pretilt angle of the polyimide blend films. When the blended film was heated at 200 °C, the vertical alignment having pretilt angle of 90° was maintained from 40 to 180 min of baking times. When the blend film was heated at 250 °C, the pretilt angles decrease from 70° to 8° with increasing the baking time from 40 to 180 min. We could not obtain a continuous pretilt angle having full range with the baking temperatures of 200 and 250 °C, while continuous control of the pretilt angles was possible using the polyimide blend film for baking time ranging from 40 to 180 min at the baking temperature of 230 °C. We also investigated the effect of the composition of blends on the LC orientation. Two polyimide blend films were prepared by mixing RN-1175 and SE-1211 at 50/50 and 25/75 weight ratios, respectively. The changes of the LC orientation with the change of baking time for these two blends were quite similar to those for SE-1211 film; a large change of LC orientation was observed from 100 to 120 min at 230 °C. Therefore 75/25 weight ratio and baking temperature at 230 °C were found to be optimum condition for the continuous control of the pretilt angle.

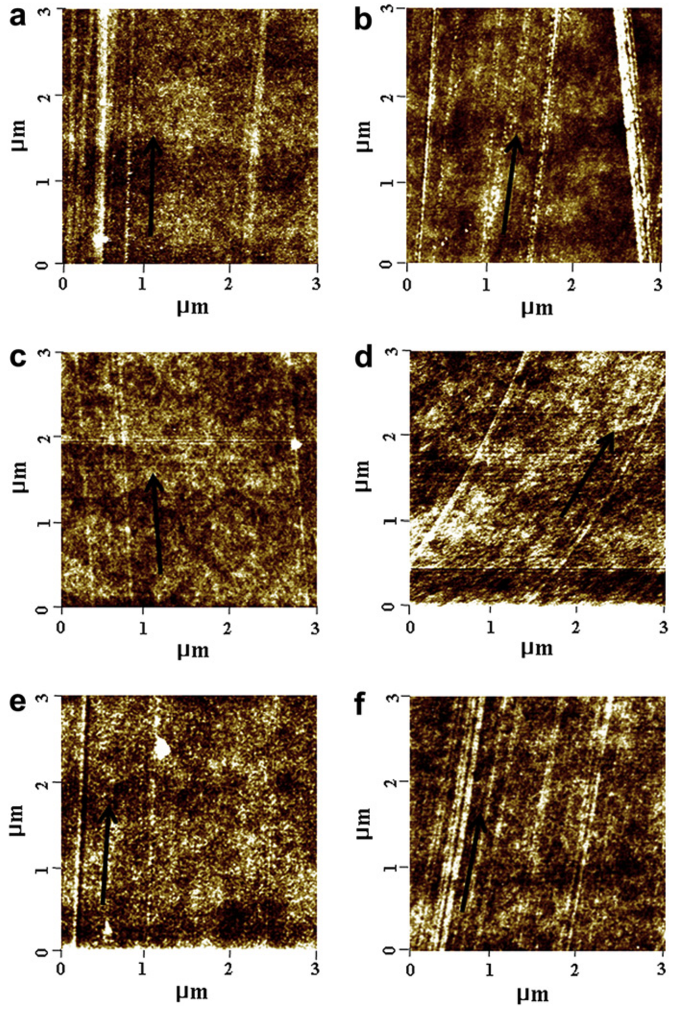

Fig. 5. AFM images of the RN-1175 films baked at 230 °C for (a) 40 min, (b) 180 min and SE-1211 films for (c) 40 min, (d) 180 min and polyimide blend films for (e) 40 min, (f) 180 min. The arrow indicates the rubbing direction.

Fig. 4 shows polar diagrams of the absorbance of a dichroic dye (disperse blue 1) in the antiparallel LC cells fabricated using the polyimide films baked at 230 °C for 40 and 180 min, respectively. LC cells made from the RN-1175 films baked for 40 and 180 min show homogeneous planar LC alignment behavior with the maximum absorbance along the 0° ↔ 180° direction (Fig. 4(a) and (b)), which is parallel with respect to the rubbing direction. The polar diagram of the LC cell made from the SE-1211 film baked for 40 min shows vertical LC aligning behavior (Fig. 4(c)), while that baked for 180 min shows homogeneous planar LC alignment behavior with the maximum absorbance along the 0° ↔ 180° direction, which is parallel with respect to the rubbing direction (Fig. 4(d)). As shown in the polar diagrams of the LC cells made from the polyimide films baked for 40 and 180 min, the LC orientation changed from vertical to planar and parallel LC alignment behavior with respect to the rubbing direction (Fig. 4(e) and (f)).

The surface morphology of the polyimide films baked at 40 and 180 min was obtained using atomic force microscopy (AFM) (Fig. 5). The surface morphology of the rubbed polyimide films having R_a (average roughness) in the range of 0.42–0.74 nm showed submicroscale groove-like structures formed in the parallel direction with respect to the rubbing direction. In particular, the polyimide blend films do not, if any, show any micro/nanoscale phase separation as described in a previous paper [11]. The AFM images make it difficult to distinguish any difference in the submicroscale groove-like structure, which might be responsible for the difference in the LC alignment properties between homo polyimide (RN-1175 and SE-1211) and polyimide blend films baked for different baking times ranging from 40 to 180 min.

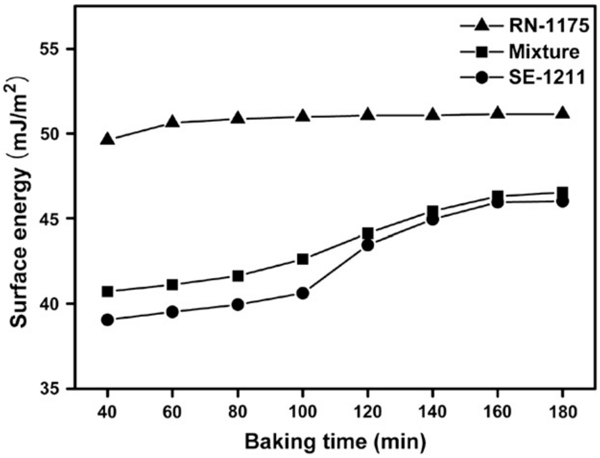

Fig. 6. Surface energy values on RN-1175, SE-1211, and polyimide blend films as a function of the baking time.

The surface energy values on rubbed polyimide films according to the baking time were measured to study the effect of the wettability on the pretilt angle of LCs (Fig. 6). We adopted water and methylene iodide contact angles on the polyimide films in static mode. The surface energy values on RN-1175 films do not change significantly with changes of the baking time, although the surface energy values at 40 min are slightly smaller than others; They are 49.62, 50.63, 50.86, 50.98, 51.07, 51.07, 51.15, and 51.15 mJ/m^2 at 40, 60, 80, 100, 120, 140, 160, and 180 min, respectively. On the contrary the surface energy values on SE-1211 and polyimide blend films decrease with the increase of baking time indicating that the changes of pretilt angles are strongly related with the changes of wettability on polymer films as described by others [7,31,32]. It was also noticed that the surface energy values of the polyimide blend films are slightly higher than those of SE-1211 when they have the same baking time. Although the content of RN-1175 in the blend is larger than that of SE-1211 (75 wt-% of RN-1175 and 25 wt-% of SE-1211), the surface energy values of the blend are close to that of SE-1211 having smaller surface energy values, rather than to that of RN-1175 having larger surface energy values. It has been known that the surface properties of polymer blends are normally governed by the polymer component having lower the surface energy values [33,34].

Electron spectroscopy for chemical analysis (ESCA) study of the polymer films also was carried out in order to investigate the effect of the chemical composition on the LC alignment behavior on the polyimide surfaces as a function of the baking time. Fig. 7 shows the ESCA spectra of C_{1s} recorded at 90° take-off angle. ESCA spectra were fitted to Gaussian-Lorentzian sum functions and the full width at half maximum (FWHM) was kept below at about 1.6 keV. Three signals were observed in the C_{1s} ESCA spectra through curve-fit and were assigned to the component C–C overlapped with $C{=}C$ at 285.0 eV, the component C–N overlapped with C–O induced by the bond-breaking of $C{=}O$ in the backbone at 286.6 eV, and the component $C{=}O$ at 288.8 eV, respectively. As the baking time was increased from 40 to 180 min, the atomic percent of the component C–C overlapped with $C{=}C$ decreased in the SE-1211 and blend polyimide films in C_{1s} ESCA spectra. While, the atomic percent of

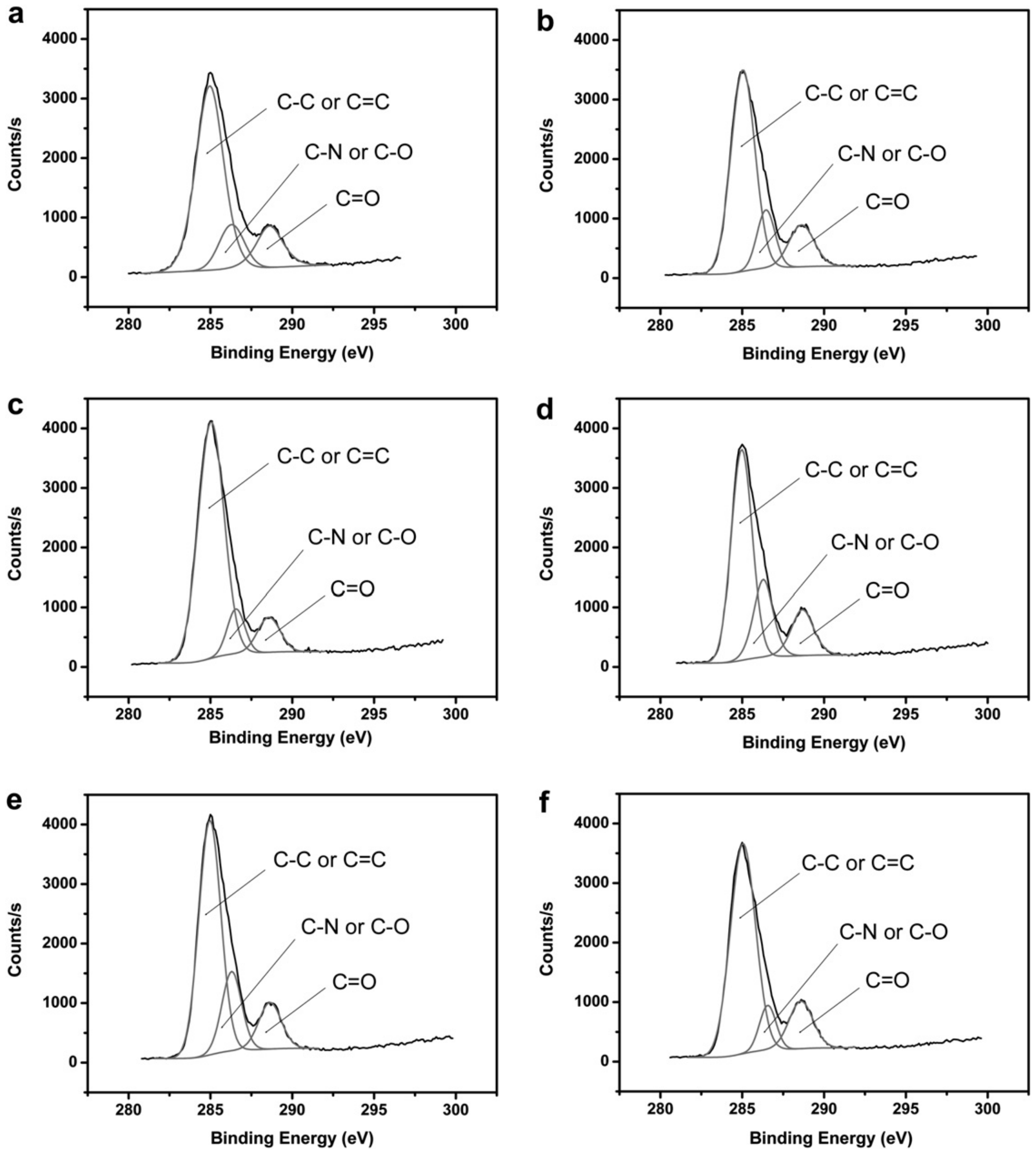

Fig. 7. ESCA spectra of the RN-1175 films baked at 230 °C for (a) 40 min, (b) 180 min and SE-1211 films for (c) 40 min, (d) 180 min and polyimide blend films for (e) 40 min, (f) 180 min in the C_{1s} recorded at 90° take-off angle.

the component C–C overlapped with C═C did not change in the RN-1175 films in C_{1s} ESCA spectra. The one N_{1s} peak in the ESCA spectra was assigned to the component C–N at 399.0 eV. It was found that the atomic percent of the component C–N of the polyimide films at baking time of 180 min was similar to those at baking time of 40 min in N_{1s} ESCA spectra. The two O_{1s} peaks in the ESCA spectra were assigned to the component C═O at 531.9 eV and the component C–O at 533.2 eV, respectively. The carbon to nitrogen ratio (C/N ratio in polyimide films at the take-off angle of 90° calculated from these C_{1s} and N_{1s} peaks in the ESCA spectra) value on the RN-1175 baked at 230 °C for 180 min were found to be very close to that baked at the same temperature for 40 min, indicating that the over baking does not change the surface composition much, if any, as expected from the pretilt angle and surface energy value results (Table 1). However the C/N ratio values of the SE-1211 and polyimide blend films obtained from baking time of 180 min

Table 1
C/N ratio values of polyimide films.

	C_{1s}/N_{1s}[a]
RN-1175 (40 min)	10.84
RN-1175 (100 min)	10.70
RN-1175 (120 min)	10.65
RN-1175 (140 min)	10.62
RN-1175 (180 min)	10.33
SE-1211 (40 min)	13.58
SE-1211 (100 min)	13.30
SE-1211 (120 min)	12.21
SE-1211 (140 min)	11.88
SE-1211 (180 min)	11.39
Blended PI (40 min)	12.88
Blended PI (100 min)	12.43
Blended PI (120 min)	12.05
Blended PI (140 min)	11.65
Blended PI (180 min)	11.23

[a] The C/N ratio values in PI films at the take-off angle of 90° calculated from these C_{1s} and N_{1s} peaks in the ESCA spectra.

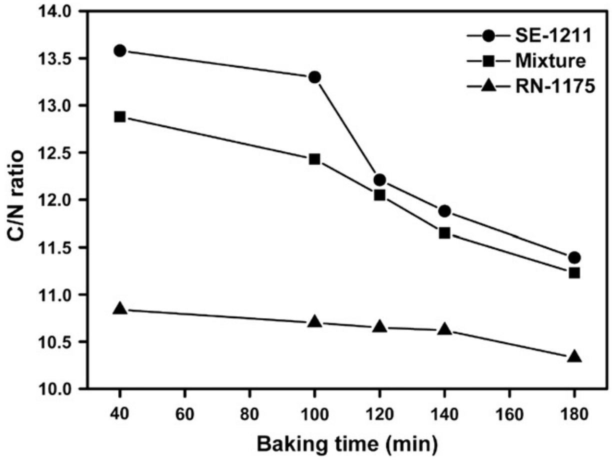

Fig. 8. C/N ratio values of the RN-1175, SE-1211, and polyimide blend films as a function of the baking time.

were found to be smaller than those obtained from baking time of 40 min, indicating that the over baking process reduce the content of carbon component on the polymer surface possibly due to the fragmentation of an *n*-octadecyl side group on the polyimide surfaces. Similarly the fragmentation of alkyl side group on the polyimide surfaces was reported previously from the overbaked polyimide [21] and ion-beam treated polyimide [35]. It was also found that the C/N ratio values of the polyimide blend films are slightly smaller than those of SE-1211 films when they have the same baking time (Fig. 8 and Table 1). The C/N ratio values of the polyimide blend films show a smaller decrease than those from SE-1211 films; for example the C/N ratio values of the polyimide blend films are 12.88, 12.43, 12.05, 11.65, and 11.23 and those of the SE-1211 films are 13.58, 13.30, 12.21, 11.88, and 11.39 for the baking times of 40, 100, 120, 140, and 180 min, respectively. The smaller change of C/N ratio of the blend films compared with that of SE-1211 films with the increase of the baking time should affect the continuous control of the pretilt angle on the blend films. Therefore, the over baking process decreases the C/N ratio values on the surface of the SE-1211 and polyimide blend films, which in turn decreases the pretilt angle and increases the surface energy of these polymer films.

Fig. 9 shows schematic illustration for the changes of the pretilt angle of LC molecules by the over baking process on the alignment layers. The polyimide film having a large content of alkyl side group can produce vertical LC alignment due to the steric repulsion between liquid crystal (5CB) molecules and long alkyl side group in the polyimide. When the polyimide films were heated at 230 °C, the content of alkyl side group on the polyimide surface decreases due to fragmentation, and thus intermediate pretilt angles could be produced due to competition between the dipole–dipole/π–π interaction between LC molecules and polyimide backbone and the steric repulsion between LCs and remaining alkyl side group. When the polyimide films were overbaked to produce the surface having smaller content of alkyl groups, then the dipole–dipole and π–π interaction between LC molecules and polyimide backbone was dominant and produced homogeneous planar LC alignment on the polymer surfaces. Therefore, we could control the pretilt angle due to the polyimide alignment films at appropriate baking temperature by changing the baking time.

4. Conclusions

The pretilt angle of LC was controlled using polyimide alignment layers by changing the baking time. Although the pretilt angle could be controlled using the polyimide films prepared from only one type of polyamic acid having an alkyl side group used for vertical LC alignment, better control was achieved from polyimide blend films prepared by mixing the polyamic acid having an alkyl side group with polyamic acid without an alkyl side group used for homogeneous planar LC alignment. For example, the pretilt angles on the polyimide blend films could be controlled from 90° to 8° by heating at 230 °C by changing the baking time ranging from 40 to 180 min. The change of the pretilt angle according to the baking time was ascribed to the competition between the dipole–dipole/π–π interaction between LC molecules and polyimide backbone and the steric repulsion between LCs and remaining alkyl side group from the surface characterization results.

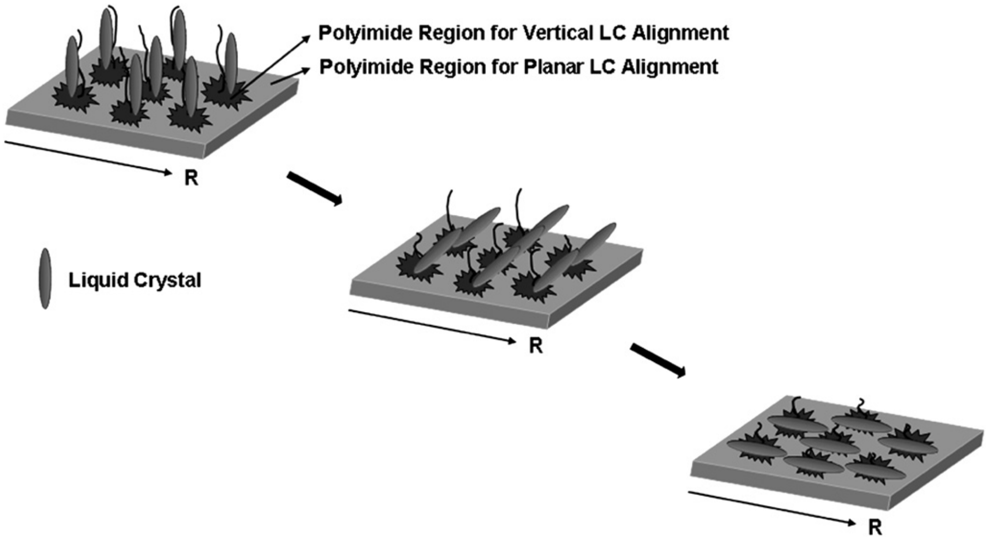

Fig. 9. Schematic illustration on the changes of liquid crystal pretilt angle on polyimide blend alignment layers obtained at 230 °C.

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