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Molecular Crystals and Liquid Crystals

Publication details, including instructions for authors and subscription information:

<http://www.tandfonline.com/loi/gmcl20>

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Ji Hyuk Im^a, Boknam Chae^b, Seung Hun Lee^a, Jun Bae^a, Hye Mi Seo^a, Jae Hak Jung^a & Seung Woo Lee^a

^a School of Chemical Engineering, Yeungnam University, Gyeongsan,, Korea

^b Pohang Accelerator Laboratory, Beamline Division, Pohang,, Korea
Published online: 06 Dec 2014.

To cite this article: Ji Hyuk Im, Boknam Chae, Seung Hun Lee, Jun Bae, Hye Mi Seo, Jae Hak Jung & Seung Woo Lee (2014) Photosensitive Polyimides with Rigid Side Chain and Their Thermal Stable Liquid-Crystal Alignment Properties, *Molecular Crystals and Liquid Crystals*, 601:1, 20-28, DOI: [10.1080/15421406.2014.940488](http://dx.doi.org/10.1080/15421406.2014.940488)

To link to this article: <http://dx.doi.org/10.1080/15421406.2014.940488>

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Photosensitive Polyimides with Rigid Side Chain and Their Thermal Stable Liquid-Crystal Alignment Properties

JI HYUK IM,¹ BOKNAM CHAE,² SEUNG HUN LEE,¹
JUN BAE,¹ HYE MI SEO,¹ JAE HAK JUNG,¹
AND SEUNG WOO LEE^{1,*}

¹School of Chemical Engineering, Yeungnam University, Gyeongsan, Korea

²Pohang Accelerator Laboratory, Beamline Division, Pohang, Korea

Thermally stable polyimide (PI), which has a photoreactive p-phenylenediacryloyl (PDA) moiety in the main chain with a rigid biphenyl as a side group, was prepared from a poly(amic acid) (PAA) solution using a thermal imidization process. PAA was synthesized successfully with a reasonably high molecular weight and gave high quality films with relatively good thermal stability. The photoreactions and photoalignments of PI were analyzed by ultraviolet-visible spectroscopy, and their liquid crystal (LC) alignment properties were examined. The PI in the film showed good photoreactivity to UV light irradiation. Linearly-polarized ultra-violet light (LPUVL) irradiation induced anisotropic reorientation of the polymer chains in the film due to a direction-selective photoreaction of the PDA moieties. All the films irradiated with LPUVL aligned the LCs in a direction perpendicular to the electric vector of the LPUVL. The pretilt angles of the LC molecules on the LPUVL irradiated film surfaces were controlled by the exposure dose of LPUVL and influenced by the thermal annealing history of the irradiated films. The LC alignments were retained on the films annealed at temperatures up to 210°C.

Keywords Polyimide; high pretilt angle; photoreaction; p-phenylenediacryloyl moiety; nematic liquid-crystal; LCD

Introduction

Liquid crystal (LC) alignment on oriented layered polymeric materials by a rubbing process with fabric velvets has been adopted in the LC display (LCD) industry because this process is simple and enables reasonable control of the alignment of LCs [1–3]. In particular, polyimides (PIs) films are used widely as oriented layered polymeric materials for a LC alignment layer because of their good thermal stability, optical transparency, adhesion, heat and solvent resist, dimensional stability and insulation [4–7]. Recently, new methods for LC alignment on polyimide surfaces using light irradiation have been suggested to avoid

*Address correspondence to Prof. Seung Woo Lee, School Chemical Engineering, Yeungnam University, 280 Daehak-ro, Gyeongsan, 712-749, Korea (ROK). Tel.:(+82)53-810-3972; Fax:(+82)53-810-4631. E-mail: leesw1212@ynu.ac.kr

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some of the problems from the rubbing process, such as difficult to apply to a large area panel, dust generation, electrostatic problems, and poor control of the rubbing strength and uniformity [8, 9].

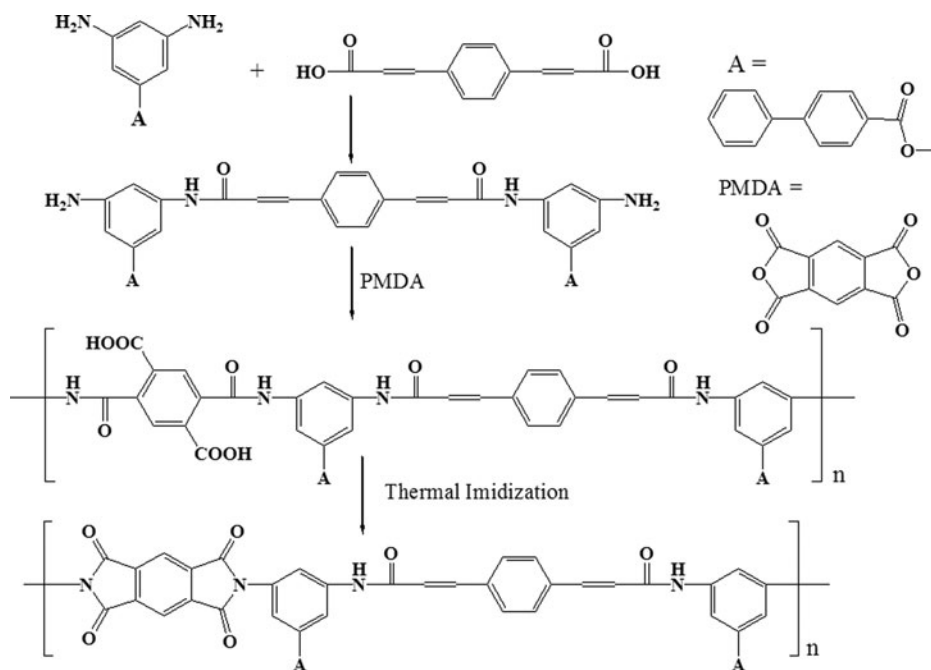
The representative methods are based on light irradiation using polymers with cinnamate groups as the side chain because cinnamate side groups in the polymer undergo dimerization by irradiating with UV light [4, 10]. The other methods use polymers with azobenzene derivatives as side groups because the azobenzene moiety can undergo *cis-trans* isomerization by UV light irradiation [11, 12]. Several polymers containing these photoreactive groups with polyvinyl as the main chains have been reported [10–12]. On the other hand, these polyvinyl-based materials are unsuitable for the LCD industry because of their low thermal stability, low anchoring energy and low pretilt angle [4, 13, 14]. Therefore, developing high performance and temperature polymers for the LC alignment layer using the light irradiation method is still a significant challenge.

In addition, the pretilt angle properties of LC molecules on the surface of LPUVL-irradiated polymers films with photoreactive side groups are not completely understood. Some researchers found that polyimides with long alkyl chains and fluorinated alkyl side chains generate a high pretilt angle of LC molecules via rubbing process [15–17]. They suggested that the high pretilt angles can be attributed to steric interactions between the LC molecules and neatly aligned branched side chains. Therefore, the development of materials for LC molecules with proper pretilt angles for the irradiation of LPUVL in the process of producing LCDs is a challenge.

In this study, a new diamine-containing photo-reactive *p*-phenylenediacryloyl (PDA) moiety in the main chain and a biphenyl moiety in the side group was synthesized. The PIs were prepared by thermal imidization using poly(amic acid)s (PAA), which were synthesized using the newly synthesized diamine and rigid dianhydride. The photoreactivity of PI in the thin films was examined by ultraviolet-visible (UV-Vis) spectroscopy. The alignment behavior of the LC molecules on the linearly polarized UV light (LPUVL)-treated PI films were examined. Moreover, the pretilt angles of the LC molecule at the surface of the PIs films irradiated with LPUVL were determined using the crystal-rotation method. In addition, the pretilt angles of LC molecules on the annealed films were also examined.

Experimental

Synthesis of diamine (BPPDA). First, biphenyl-3,5-diaminobenzoate (BP) was synthesized from a reaction of 5-Di-*tert*-butoxycarbonylaminobenzoic acid (3.5 g, 9.9 mmol), 4-phenylphenol (2.09 g, 11.9 mmol) and DCC (2.5 g, 11.9 mmol) with the DMAP catalyst (0.15 g, 1.23 mmol), and the reactant mixture was stirred at ambient temperature for 12 h [18]. New photoreactive diamine (BPPDA) was synthesized from a reaction of BP and *p*-phenylenediacrylic acid (PDA) using the following process. A mixture of 5 mmol (1.09 g) of PDA, 25 mmol (7.61 g) of BP, 50 mL of NMP/Py (v/v = 4/1), and 10 mmol (3.10 g) of triphenylphosphite (TPP) was stirred at room temperature under nitrogen for 2 h [19, 20]. The reaction mixture was heated to 100°C for an additional 2 h and poured into methanol. The precipitated solids were separated by filtration and washed thoroughly with methanol. The crude product was purified by recrystallization from DMAc/H₂O mixture and drying *in vacuo*. ¹H NMR (DMSO-d₆, δ): 10.16 (s, 2H, -NH), 7.76–7.34 (m, 22H, -ArH-), 7.13 (s, 1H, -ArH-), 6.90 (d, 2H, -Ar-CH=CH-), 5.51 (s, 4H, -Ar-NH₂).



Scheme 1. Chemical structure of PMDA-BPPDA poly(amic acid) and the preparation procedure of polyimide.

Synthesis of poly(amic acid)s. Poly(amic acid) (PAA) was prepared by adding 1 equivalent of pyromellitic dianhydride (PMDA) in a NMP solution and 1 equivalent of BPPDA, which were pre-dissolved in NMP to form a 10.0 wt.% solution, under nitrogen with vigorous stirring (see Scheme 1). Once the addition was complete, the reaction flask was sealed tightly and the mixture was stirred for 48 h to make the polymerization mixture homogeneous and viscous. This solution was filtered through a syringe-driven filter with a pore size of 1.0 μm and stored in a refrigerator until needed. For the synthesized PAA, the inherent viscosity was measured in NMP at 25°C using an Ubbelohde suspended level capillary viscometer according to the method described in the literature [21].

Measurements. The ^1H nuclear magnetic resonance (NMR) spectrum was obtained at room temperature using a Bruker AM 300 spectrometer. The glass transition temperature (T_g) and degradation temperature (T_d) of each film were measured by differential scanning calorimetry (DSC, model DSC-7, Perkin-Elmer, USA) and thermogravimetric analysis (TGA, model TGA-7, Perkin-Elmer, USA), respectively. The UV-visible absorption spectra were obtained as a function of the exposure dose using a Hewlett-Packard 8453 spectrophotometer with or without a dichroic polarizer (Oriel, model 27320). The LC alignment in the cell was examined from the absorption of a linearly polarized He-Ne laser beam (632.8 nm wavelength) as a function of the rotational angle of the cell. The pretilt angle, α , of the LC molecules was measured using a crystal rotation apparatus.

LC cell fabrication. A poly(amic acid) (PAA) solution was spin-cast onto indium tin oxide (ITO)-coated glass for LC cell assembly, followed by drying on a hot plate at 80°C for 1 h. The dried films were imidized thermally in an oven with flowing dry nitrogen gas using

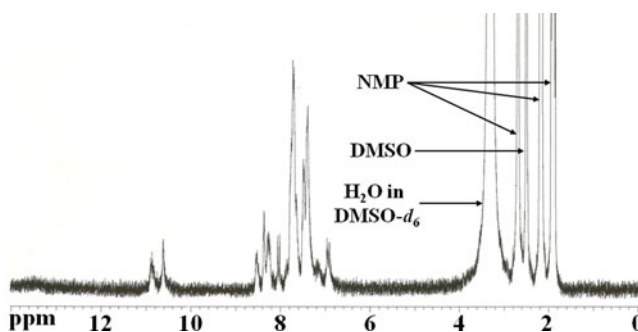


Figure 1. ^1H NMR spectra of PMDA-BPPDA poly(amic acid) including NMP dissolved in dimethyl- d_6 sulfoxide ($\text{DMSO}-d_6$).

a three-step imidization protocol, i.e. $150^\circ\text{C}/60$ min, $200^\circ\text{C}/60$ min, and $250^\circ\text{C}/120$ min, at a ramping rate of $2.0^\circ\text{C}/\text{min}$. The samples were cooled to room temperature at a rate of $10^\circ\text{C}/\text{min}$, producing good quality films. The PI films adhered to the ITO slides were irradiated with the filtered linearly-polarized UV light (LPUVL) at various exposure dose. Some of the PI films irradiated with LPUVL at $1.5\text{ J}/\text{cm}^2$ were annealed thermally in an accumulative step manner from room temperature to 210°C using the regime, $80^\circ\text{C}/10$ min, $120^\circ\text{C}/10$ min, $150^\circ\text{C}/10$ min, $180^\circ\text{C}/10$ min, and $210^\circ\text{C}/10$ min, and then cooled to room temperature. The LC cell assemblies were constructed from the annealed films. Using these polymer films, the LC cells were assembled and filled with 4-pentyl-4-biphenylcarbonitrile (5CB) containing 1.0 wt% of a dichroic dye (Disperse Blue 1) using a capillary technique.

Results and Discussion

To obtain the amide-containing diamine with a photoreactive group in the main chain and a biphenyl moiety as the side group, an excess of BP was condensed with PDA in the presence of TPP and pyridine at 100°C using a TPP-activated amidation reaction [19, 22]. To remove the impurities and produce a diamine monomer, the crude product was obtained by precipitation in excess methanol and purified by repeated recrystallization of the crude precipitates. The BPPDA obtained was characterized by ^1H NMR spectroscopy. The spectroscopy results were coincident with the expected chemical structure.

The prepared PAA with residual DMAc was characterized by ^1H NMR spectroscopy. The two proton peaks at 10.9 and 10.6 ppm were assigned to the amide linkages from the newly formed amide from the reaction of PMDA and BPPDA and the amide linkage of the newly synthesized BPPDA diamine, respectively. PAA revealed aromatic proton peaks at 6.8–8.6 ppm (See Fig. 1). In addition, the amino protons peak at 5.5 ppm originating from the residues of unreacted BPPDA diamine were not detected in the ^1H NMR spectrum. The estimated peak integration corresponded well with the expected chemical structure. For PAA, the measured inherent viscosity was $0.82\text{ dL}/\text{g}$. The ^1H NMR spectrum and inherent viscosity suggest that PAA had been synthesized successfully with a reasonably high molecular weight.

The PI film prepared by the thermal imidization of homogeneous PAA solutions was characterized by FTIR spectroscopy. Figure 2 shows the FTIR spectra of the PI film. The FTIR peaks of the PI and composite films were assigned according to previously reported data [23, 24]. The pure PI film exhibited vibrational absorption bands at 1777, 1719, 1785

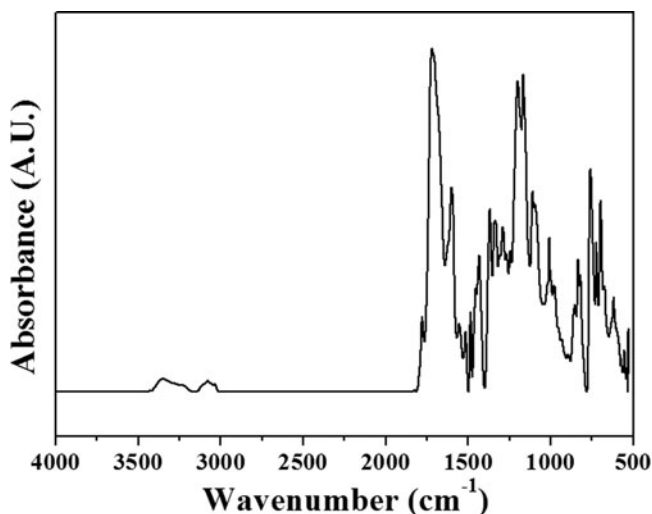


Figure 2. FTIR spectrum of PMDA-BPPDA polyimide film prepared by thermal imidization.

and 1368 cm⁻¹. The two bands at 1777 and 1719 cm⁻¹ were assigned to the symmetric and asymmetric stretching vibrations of the carbonyl group of the imide ring, respectively, which are typical of PIs. The band at 1368 cm⁻¹ corresponds to the C-N stretching vibration in the PI main chain, respectively. These vibrational modes were associated with the polymer main chains.

The thermal stability of the PMDA-BPPDA PI film was examined by TGA. As shown in Fig. 3, the PI films exhibited a two-step thermal degradation pattern. The first weight loss at temperatures of up to 300°C was attributed to the loss of ester groups in the side group and amide groups in the main chain. The second weight loss at ~480°C was assigned to the decomposition of the aromatic component in the PI main chain [24]. In addition, the glass

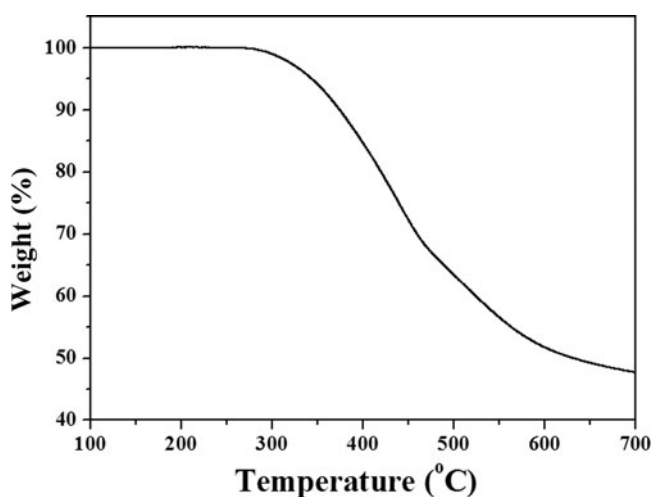


Figure 3. TGA thermogram of the PMDA-BPPDA polyimide film, which was measured at a heating rate of 10°C/min.

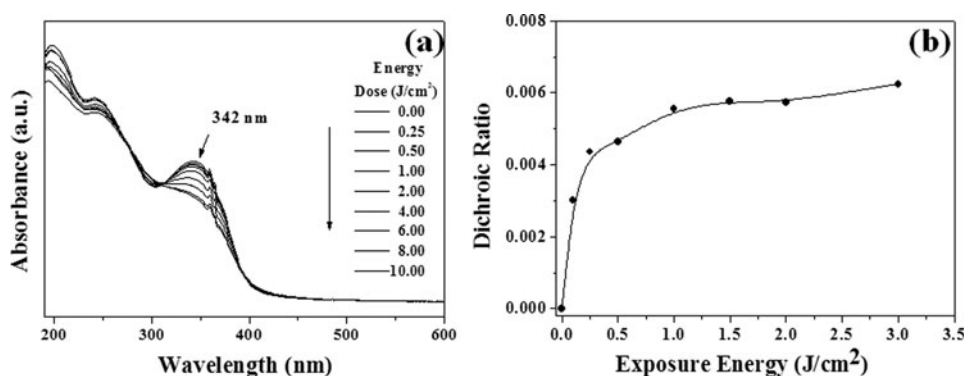


Figure 4. (a) UV spectra of photoreactive PMDA-BPPDA polyimide films irradiated with unpolarized UV light (260–380 nm) at various exposure doses. (b) Dichroic ratios measured from the PMDA-BPPDA polyimide films irradiated with LPUVL at various exposure energies.

transition of the synthesized PI was examined by DSC in a nitrogen atmosphere. The PI film did not show any glass transition over the temperature range, 25–300°C, even after the thermal treatment and repeated scanning. Hence, the PI synthesized has reasonably good thermal

Figure 4(a) shows the UV-vis spectra measured from PMDA-BPPDA PI films irradiated with UV light at various exposure doses. The PMDA-BPPDA polymer exhibited an absorption maximum at 342 nm ($= \lambda_{\max}$), possibly due to the photosensitive PDA group in the polymer. The absorption peak intensity at $\lambda_{\max} = 342$ nm decreased drastically with increasing exposure dose up to 8.0 J/cm² and was relatively constant with further increases in the exposure dose. This might be due to photoreactions, such as [2 + 2] cycloaddition and/or *trans-cis* isomerization of the PDA chromophore in the films.

Lee et al. examined the photochemical reaction of a photosensitive polyester containing PDA chromophores in the main chain in films by NMR spectroscopy, UV-vis spectroscopy, FTIR spectroscopy, and Raman spectroscopy as well as by dissolution testing. They reported that the PDA chromophores of the photosensitive PI underwent both *trans-cis* photoisomerization and [2 + 2] photodimerization, whereas the dimerization product was mostly identified in the films [25, 26]. These results suggest that the decrease in absorption intensity at $\lambda_{\max} = 317$ nm is due mainly to the [2 + 2] photodimerization of cinnamate side groups.

The dichroic ratio [$= (A_{\perp} / A_{\parallel}) / (A_{\perp} + A_{\parallel})$] was determined from the absorbance at λ_{\max} (maximum absorption wavelength) (A_{\perp}), which was measured using a probing UV-Vis light linearly polarized perpendicular to the electric vector of LPUVL and at (A_{\parallel}), which was measured using a probing UV-Vis light linearly polarized parallel to the electric vector of LPUVL. Figure 4b shows the dichroic ratios of the PMDA-BPPDA films determined using this approach. As shown in the figure, all the measured dichroic values were positive over the exposure energy. The dichroic ratio increased sharply with increasing energy dose up to 0.5 J/cm² and was retained with further increases in exposure energy. This suggests that during LPUVL exposure, the PDA chromophores aligned parallel to the polarization direction of the LPUVL were consumed more rapidly than those aligned perpendicular to the polarization direction of the LPUVL. As a result of this directionally selective photoreaction, the LPUVL irradiation of the PMDA-BPPDA PI films orient the polymer

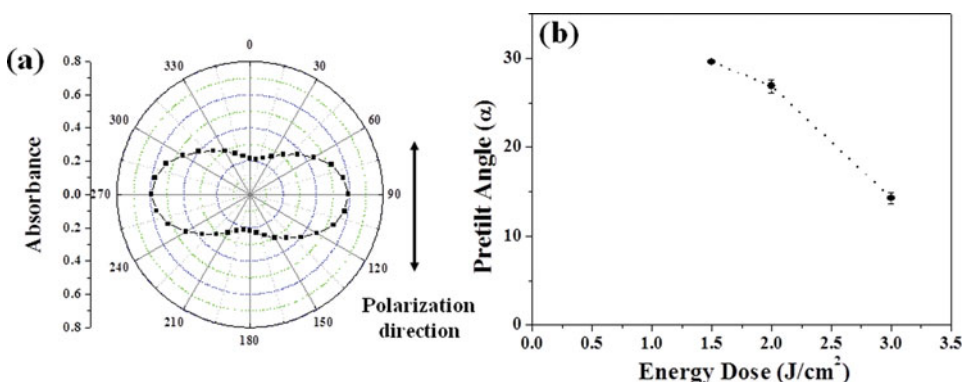


Figure 5. (a) Polar diagrams of the absorption of linearly polarized visible light (632.8 nm wavelength) by LC cells fabricated with PMDA-BPPDA polyimide films irradiated with LPUVL at 1.5 J/cm², as a function of the angle of rotation of the LC cells. (b) Pretilt angle variations of the LCs measured for LC cells fabricated from PMDA-BPPDA polyimide films, which were measured at various LPUVL exposure doses.

chains preferentially and leave PDA chromophores unreacted in the same direction, which is perpendicular to the electric vector of the LPUVL [25].

Optical microscopy showed that all the antiparallel LC cells fabricated using the PI films irradiated with LPUVL at various exposures were homogenous. Figure 5a displays a polar diagram of the absorption of a linearly polarized light probe (wavelength 632.8 nm), which was obtained from measurements of a LC cell fabricated from a PMDA-BPPDA PI film irradiated with LPUVL at 1.5 J/cm². In this polar diagram, the main director of the LC molecules lies along the 270° ↔ 90° direction, which is perpendicular to the polarization direction (0° ↔ 180°) of the LPUVL used in UV-exposure. This means that the LC molecules in contact with the surface are aligned homogeneously at an angle of 90° with respect to the polarization direction of the LPUVL. The direction of the LC alignment

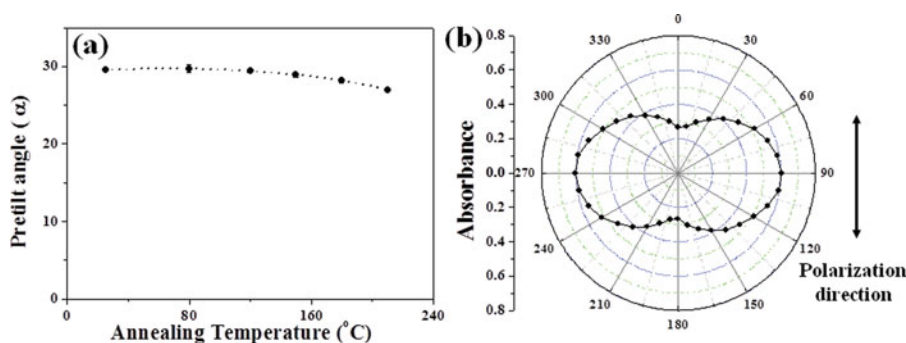


Figure 6. (a) Pretilt angle variations of the LCs measured for LC cells fabricated from PMDA-BPPDA polyimide films. The polyimide films were irradiated with LPUVL at 1.5 J/cm² and then annealed using a stepped heating regime (80°C/10 min, 120°C/10 min, 150°C/10 min, 180°C/10 min, and 210°C/10 min). (b) Polar diagrams of the absorption of linearly polarized visible light (632.8 nm wavelength) by LC cells. Which were fabricated with PMDA-BPPDA polyimide films irradiated with LPUVL at 1.5 J/cm² and then annealed, as a function of the angle of rotation of the LC cells.

was the same for the films irradiated at exposure energies in the range, 0.1 – 3.0 J/cm². This suggests that the unreacted PDA moiety and PI polymer chains oriented anisotropically by LPUVL irradiation play a major role in the alignment of LC molecules in contact with the irradiated film. For the LC cells prepared from the PMDA-BPPDA PI films, an attempt was made to determine the pretilt angle of the LC molecules along the alignment direction of the LC molecules using the crystal-rotation method. The pretilt angle is defined as the angle between the optical principal axis of the LC molecule and the film plane. Figure 5b shows the pretilt angles of the LC molecules at the surface of a series of PMDA-BPPDA PI films irradiated with LPUVL at various exposure doses. The pretilt angle of the LC molecules at the surface of the PI films decreased with increasing exposure dose of LPUVL. Overall, the pretilt angle of the LC molecules was in the range, 25–30°, depending on the irradiation energy of LPUVL. This means that the LPUVL-irradiated PMDA-BPPDA PIs films induce LC molecules to align homogeneously at an angle of 90° with respect to the polarization direction of the LPUVL used in the irradiation process. In addition, the pretilt angles measured are caused mainly by interactions between the rigid ester bond linked biphenyl side groups and the biphenyl mesogen unit of 5CB LC molecules [25]. Consequently, the rigid side groups of the PI play a critical role in the generation of the out-of-plane pretilt angle of the LC molecules in contact with the LPUVL irradiated film surface.

Figure 6(a) shows the pretilt angles of the LC molecules at the surface of the PMDA-BPPDA films irradiated with LPUVL at 1.5 J/cm², followed by annealing using the stepped heating regime: 80°C/10 min, 120°C/10 min, 150°C/10 min, 180°C/10 min, and 210°C/10 min. As shown in the figure, the films annealed at higher temperatures showed a lower LC pretilt angle. This might be due to changes in the out-of-plan orientation of biphenyl side groups of the PIs caused by thermal annealing. On the other hand, the direction of the alignments of LCs on these annealed PMDA-BPPDA films were the same as those for the unannealed PI film, regardless of the annealing history (see Figure 6(b)). This suggests that the oriented polymer chains induced by LPUVL are thermally stable up to 210°C.

Conclusions

A photosensitive polyimide (PI) was prepared by a thermal imidization process from poly(amic acid) (PAA) solutions prepared using pyromellitic dianhydride (PMDA) and newly synthesized diamine with the p-phenylenediacryloyl (PDA) moiety in the main chain with biphenyl groups as a side chain in NMP. Good quality films were obtained by the thermal imidization of PAA. The PI film from PAA showed no glass transition over the temperature range, 25–300°C, and had a high decomposition temperature, suggesting high thermal stability. The polymer film had excellent photoreactivity to UV light. Through the irradiation of LPUVL, the PDA moiety in the polymer main chain undergoes direction-selective photoreaction and induces a reorientation of the polymer main chains. The direction of LC alignment is perpendicular to the LPUVL. These LC alignment and molecular orientation results show that the oriented polymer chains and unreacted PDA chromophores in the irradiated films interact anisotropically with LC molecules to align the LC molecules along their orientation directions. The pretilt angle of the LC molecules ranged from 25° to 30°, depending on the irradiation energy of LPUVL. This means that the rigid side groups of PI play a key role in generating the out-of-plane pretilt angle of the LC molecules in contact with the LPUVL irradiated film surface. The pretilt angles of the LC molecules on the LPUVL irradiated film surfaces were affected by the thermal annealing history of the irradiated films. The LC alignments were retained on the films annealed at

temperatures up to 210°C. In conclusion, this novel PMDA-BPPDA PI is a good candidate material for applications in the LCD industry.

Funding

This study was supported by Yeungnam University research grants in 2011 and a Human Resources Development Program of Korea Institute of Energy Technology Evaluation and Planning (KETEP) grant (No. 20104010100580) funded by the Korean Ministry of Knowledge Economy.

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