

# Photo-enhanced Polymer Memory Device Based on Polyimide Containing Spiropyran

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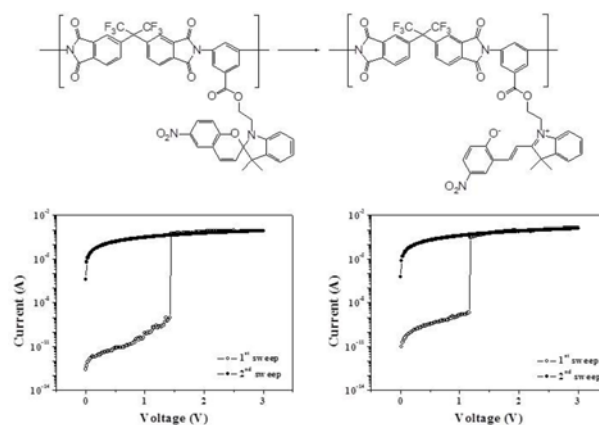
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This paper reports the synthesis of a new polyimide (PI) containing a spiropyran moiety in the side chain and its applications to the switchable polymer memory before and after UV exposure. UV exposure allows memory using spiropyran-based PI as an active layer with a higher current and lower switching-ON voltage compared to the unexposed device due to the structural changes in the spiropyran moiety after UV exposure. In addition, this study examined the effects of UV exposure on the performance of the memory containing spiropyran-based PI using the UV-Vis absorption spectra and space-charge limited conduction (SCLC) model.

**Keywords:** polyimide, spiropyran, memory device, photo response polymer



## 1. INTRODUCTION

In recent years, there has been significant progress in organic and polymer materials for applications to electronic devices as a possible alternative to traditional inorganic semiconducting materials because of their low-cost fabrication, solution processability, and molecular and electronic modification by synthesis.<sup>[1]</sup> Among the several types of organic electronic devices, such as organic light-emitting diodes (OLEDs),<sup>[2]</sup> organic thin-film transistors (OTFTs),<sup>[3]</sup> and organic photovoltaic (OPVs),<sup>[4]</sup> considerable attention has recently been paid to the use of electrically bistable resistive switching organic materials in the fabrication of memory devices. Therefore, the synthesis of various novel

organic and polymer semiconducting materials and high-grade memory devices have been investigated intensively.<sup>[5-13]</sup> In particular, polymer materials have a great advantage over small organic materials, considering the easy fabrication of the three-dimensional (3D) multi-stack layer structures required for high density memory devices. Hence, there has been significant research effort on the development of polymer switching materials with properties and processability that meet the requirements of memory devices.<sup>[12]</sup>

Aromatic polyimides (PIs) have excellent thermal stability, dimensional stability, mechanical properties, optical transparency, adhesion and chemical resistance.<sup>[14,15]</sup> In addition, they can be fabricated easily as films by conventional, simple spin-, bar-, roll- or dip-coating processing of their solutions in organic solvents. For these reasons, the development of highly stable memory devices based on dimensionally and thermally stable polyimides have been reported.<sup>[16-18]</sup> Recently, the spiropyran moiety is frequently

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introduced in the several organic compounds because it can be act as a photoactive functionality that takes advantage of the molecular conformational changes, electronic structural changes, and dipole moment changes as a result of photochromic conversion.<sup>[19]</sup> The photosensitivity of spiropyran has allowed several types of devices (e.g., photodetector and organic flash memories, and photoswitchable OFETs) to operate successfully in response to external inputs, such as light.<sup>[19-23]</sup> Therefore, it is interesting to develop a PI-containing spiropyran moiety as a photosensitive polymer memory and to characterize the memory performance depending on light exposure. Although spiropyran-based PI has been used for switching polymer memory, the role of spiropyran is limited to controlling the optical recording behavior, rather than the memory performance.<sup>[23]</sup>

In this study, a new PI containing a spiropyran moiety in the side chain was synthesized for applications as a material for switchable polymer memory materials. By introducing the spiropyran unit to the side chain of PI, a photo-induced reaction was observed between two isomers with the UV-Vis absorption spectra: colorless, nonplanar, close spiropyran form, and colored, planar, open zwitterionic merocyanine form. In particular, the ring-opened form of spiropyran by exposure to UV enabled the formation of dipolar charges in the backbone, as well as a planar structure that can make an extended conjugation system. Therefore, it is expected that such a structural feature would make it possible to improve the performance of write-once-read-many-times (WORM) memory based on PI. To characterize the effects of the UV-induced structure changes on the performance of the WORM memory-containing spiropyran-based PI, current-voltage ( $I$ - $V$ ) measurements and the space-charge limited conduction model were used to examine the memory depending on the UV exposure.

## 2. EXPERIMENTAL PROCEDURE

### 2.1 Materials

2,2-Bis-(3,4-dicarboxyphenyl)hexafluoropropane dianhydride (6F) was supplied by Chriskev Company (Kansas, USA) and purified by recrystallization from acetic anhydride. 3,5-Diaminobenzoic acid (DABA) was purchased from Aldrich and purified by vacuum drying at 100 °C. 2,3,3-Trimethyl-3H-indole, 2-bromoethanol, 5-nitrososalicylaldehyde, dimethylpyridine (DMAP), 1-ethyl-3-(3-dimethylaminopropyl) carbodiimide (EDAC), chloroform, ethyl acetate, and methylene chloride (MC) were acquired from Aldrich and used as received. *N*-Methyl-2-pyrrolidone (NMP), the polymerization solvent, was purchased from Aldrich and distilled over calcium hydride under reduced pressure.

### 2.2 Synthesis of soluble polyimide

A soluble polyimide, 6F-DABA PI, was prepared from 6F

and DABA as follows.<sup>[24]</sup> Equimolar amounts of 6F (4.00 g, 9.00 mmol) and DABA (1.37 g, 9.00 mmol) were dissolved together with a catalytic amount of isoquinoline in dry NMP. The solution was heated gently with stirring at 80 °C for 2 h and then refluxed with stirring at 240 °C for 12 h. The reaction solution was then poured into a mixture of methanol with vigorous stirring, giving the polymer product in precipitated powder. The precipitated powder was filtered, washed several times with the mixture solvent, and dried in a vacuum oven at 50 °C. The obtained PI product was dissolved in dimethyl-*d*<sub>6</sub> sulfoxide (DMSO-*d*<sub>6</sub>) and characterized by proton nuclear magnetic resonance (<sup>1</sup>H NMR) spectrometry (Bruker, model Aspect 300 MHz): <sup>1</sup>H NMR ( $\delta$ , DMSO-*d*<sub>6</sub>), 10.35-10.15 (s, 1H, ArOH), 8.25-8.00 (d, 2H, ArH), 8.00-7.80 (s, 2H, ArH), 7.80-7.55 (d, 2H, ArH), 7.50-7.25 (d, 1H, ArH), 7.15-6.95 (d, 2H, ArH).

### 2.3 Synthesis of spiropyran derivative

Spiropyran derivative, SP-OH, was prepared by modified according to the previous literature.<sup>[25,26]</sup> A mixture of 2,3,3-trimethyl-3H-indole (6.00 g, 37.68 mmol) and 2-bromoethanol (6.12 g, 48.99 mmol) was heated to 240 °C for 6 h under N<sub>2</sub>. After cooling to room temperature, the crude product was suspended in chloroform and the mixture was sonicated and filtered. The precipitation was recrystallized from ethyl acetate, and dried to give 1-2-(hydroxyethyl)-2,3,3-trimethyl-3H-indolium bromide (9.50 g, 88.7% yield), which was then used in the next step without purification. A solution of 1-2-(hydroxyethyl)-2,3,3-trimethyl-3H-indolium bromide (8.00 g, 28.15 mmol) and KOH (3.60 g, 64.18 mmol) in distilled water was stirred at room temperature for 1 hr and extracted with Et<sub>2</sub>O. The organic layer was collected, dried with MgSO<sub>4</sub>, and concentrated under reduced pressure to afford 3,3-dimethyl-1-(2-hydroxyethyl)-indoline (4.72 g, 82.4% yield) as a yellow oil. A mixture of 3,3-dimethyl-1-(2-hydroxyethyl)-indoline (4.00 g, 19.68 mmol) and 5-nitrososalicylaldehyde (3.41 g, 19.68 mmol) in ethanol was heated for 3 h under reflux in a N<sub>2</sub> atmosphere. After cooling to room temperature, the precipitation was filtered, washed with ethanol, and dried to give 2-(3',3'-dimethyl-6-nitrospiro [chromene-2,2'-indolin]-1'-yl)ethanol, SP-OH, as a purple solid (5.58 g, 74.65 yield). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): 1.21 (s, 3H), 1.31 (s, 3H), 3.32-3.38 (m, 1H), 3.44-3.51 (m, 1H), 3.71-3.85 (m, 2H), 5.88 (d, 1H,  $J = 10.4$  Hz), 6.67 (d, 1H,  $J = 8.0$  Hz), 6.76 (d, 1H,  $J = 8.8$  Hz), 6.90 (m, 2H), 7.10 (d, 1H,  $J = 7.6$  Hz), 7.19 (t, 1H), 8.00 (m, 2H).

### 2.4 Synthesis of polyimide with spiropyran as a side group

A soluble polyimide with spiropyran as a side group, 6F-DAB-SP, was synthesized from the reaction of 6F-DABA and SP-OH.<sup>[27]</sup> A mixture of 6F-DABA, SP-OH, EDAC, and DMAP (1:3:3:3 in molar ratio) in methylene chloride (total

amount of the reactants was 10 wt. %) was stirred at room temperature for 24 h under  $N_2$ . The reaction mixture was precipitated in methanol with vigorous stirring at room temperature, filtered, washed several times with methanol, and then dried in a vacuum oven at 80 °C to give 6F-DAB-SP:  $^1H$  NMR (400 MHz,  $CDCl_3$ ): 1.21 (s, 3H), 1.31 (s, 3H), 3.32-3.38 (m, 1H), 3.44-3.51 (m, 1H), 3.71-3.85 (m, 2H), 5.88 (d, 1H,  $J = 10.4$  Hz), 6.67 (d, 1H,  $J = 8.0$  Hz), 6.76 (d, 1H,  $J = 8.8$  Hz), 6.90 (m, 2H), 7.10 (d, 1H,  $J = 7.6$  Hz), 7.19 (t, 1H), 8.00 (m, 2H).

## 2.5 Fabrication of a memory device

To fabricate the polymer memory devices, homogenous solutions of polymer were prepared by dissolution in cyclopentanone. The resulting solutions were filtered through PTFE-membrane micro filters with a pore size of 0.22  $\mu m$ . The polymer solutions were spin-coated onto pre-cleaned glasses coated with indium tin oxide (ITO) with a thickness of ca. 30 nm at 2000 rpm for 60 s. The films were then baked at 80 °C for 12 h in a vacuum. The thicknesses of the PI films were determined by spectroscopic ellipsometry (model M2000, Woollam). Al top electrodes with a thickness of 30 nm were deposited onto the polymer films through a

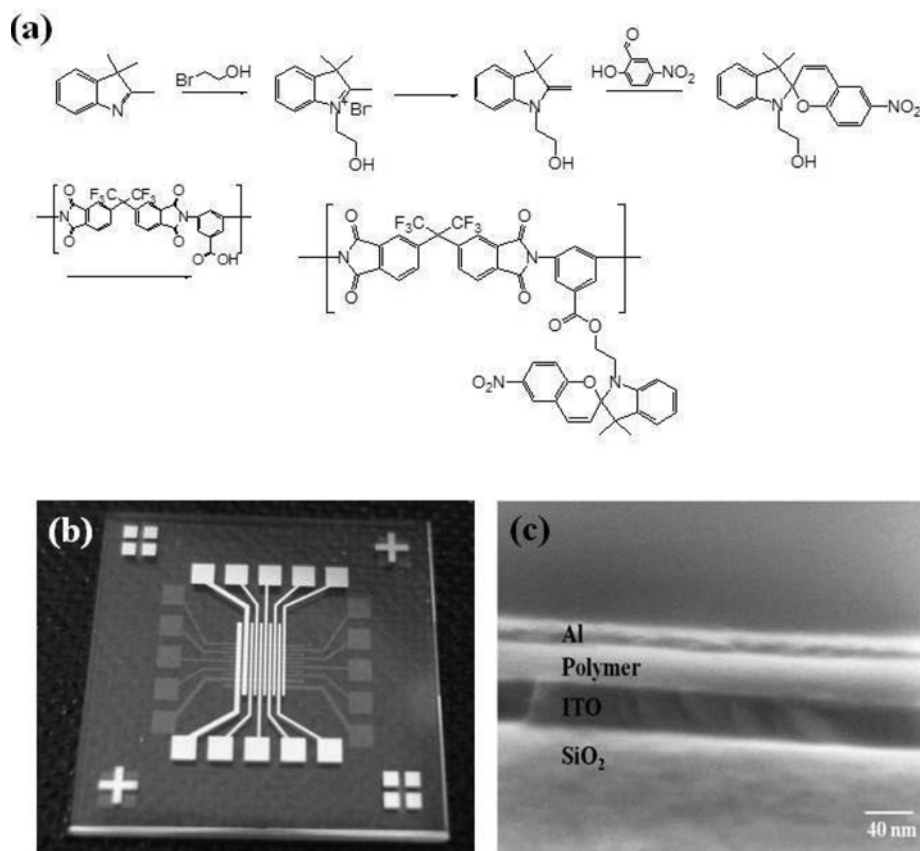
shadow mask by thermal evaporation. All electrical experiments were conducted without device encapsulation either in air or in a nitrogen atmosphere. The current-voltage ( $I$ - $V$ ) measurements were carried out using a Keithley 4200-SCS semiconductor analyzer and a probe station equipped with a heating stage. In all cases, the bias voltage was applied with respect to the bottom electrode.

## 2.6 Measurements

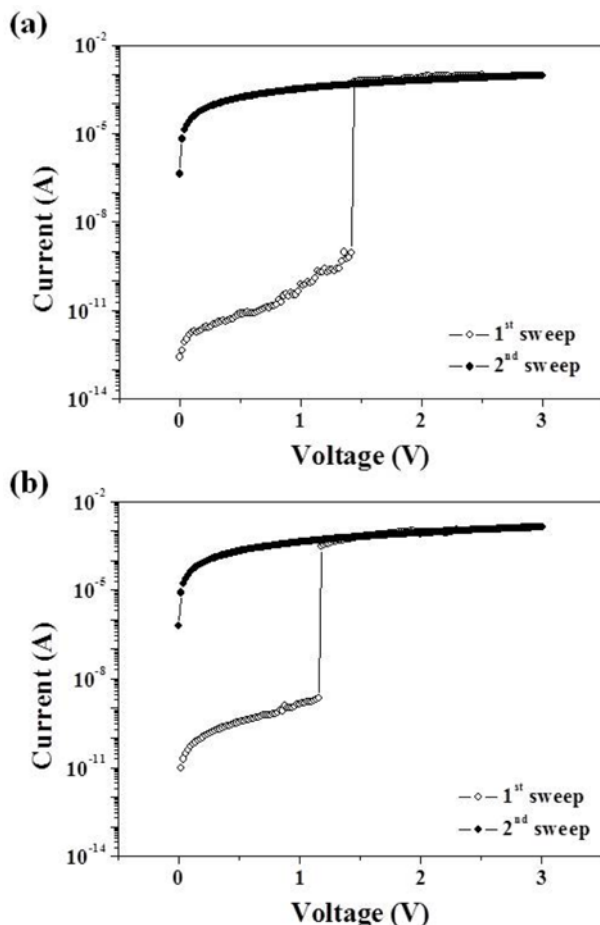
The  $^1H$  nuclear magnetic resonance (NMR, Bruker AM 300) spectra were obtained at room temperature. The ultraviolet-visible (UV-vis) absorption spectra were obtained using an UV-vis spectrophotometer (UV-1800, Shimadzu). For the photoreactivity of the 6F-DAB-SP films, UV light irradiation was conducted with and without a linear dichroic polarizer (Oriel) using a high-pressure Hg lamp system (1.0 kW, Altech, South Korea) equipped with an optical filter (Milles Griot), which transmits a band beam with a wavelength of 260-380 nm.

## 3. RESULTS AND DISCUSSION

The memory effect of the synthesized polyimide with a

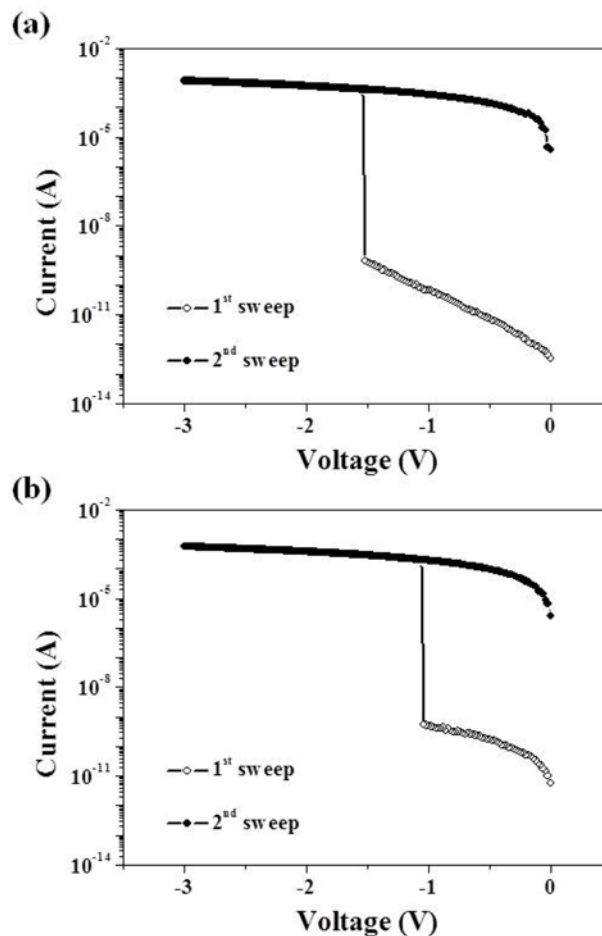


**Fig. 1.** (a) Chemical structure of 6F-DAB-SP polymer (b) photograph of the ITO/6F-DAB-SP/Al, organic memory device, (c) Cross-sectional SEM image of the device.



**Fig. 2.** Typical  $I$ - $V$  curves of the ITO/6F-DAB-SP (30 nm thick)/Al devices, which were measured with a compliance current set of 0.1 A. The applied voltage was swept from 0 V  $\rightarrow$  +3.0 V. The electrode contact area was  $0.1 \times 0.1 \text{ mm}^2$ . (a) unexposed device, (b) exposed device.

spiropyran side group (6F-DAB-SP) film was examined. Figure 1a presents the synthetic procedure and chemical structure of 6F-DAB-SP. Figure 1b shows a fabricated diode-structured device (of Al/6F-DAB-SP/ITO). The vacuum-deposited Al top electrode was stacked on the 30 nm-thick 6F-DAB-SP layer, which had been deposited on the ITO/glass substrate (Fig. 1c). Figure 2 shows typical current-voltage ( $I$ - $V$ ) characteristics of the 30 nm thick 6F-DAB-SP film with Al top and ITO bottom electrodes. The diode devices were operated from 0 V to +3.0 V using a semiconductor parameter analyzer with a compliance current of 0.01 A. In particular, to measure the memory behavior of 6F-DAB-SP device in response to light, the device was exposed to UV light ( $\lambda = 260\text{-}380 \text{ nm}$ ) at  $160 \text{ mJ/cm}^2$  in ambient air. Although both unexposed and exposed 6F-DAB-SP devices showed successful memory characteristics (Fig. 2a and b), there was a significant difference in the switching-ON voltages and OFF-current level. First, the



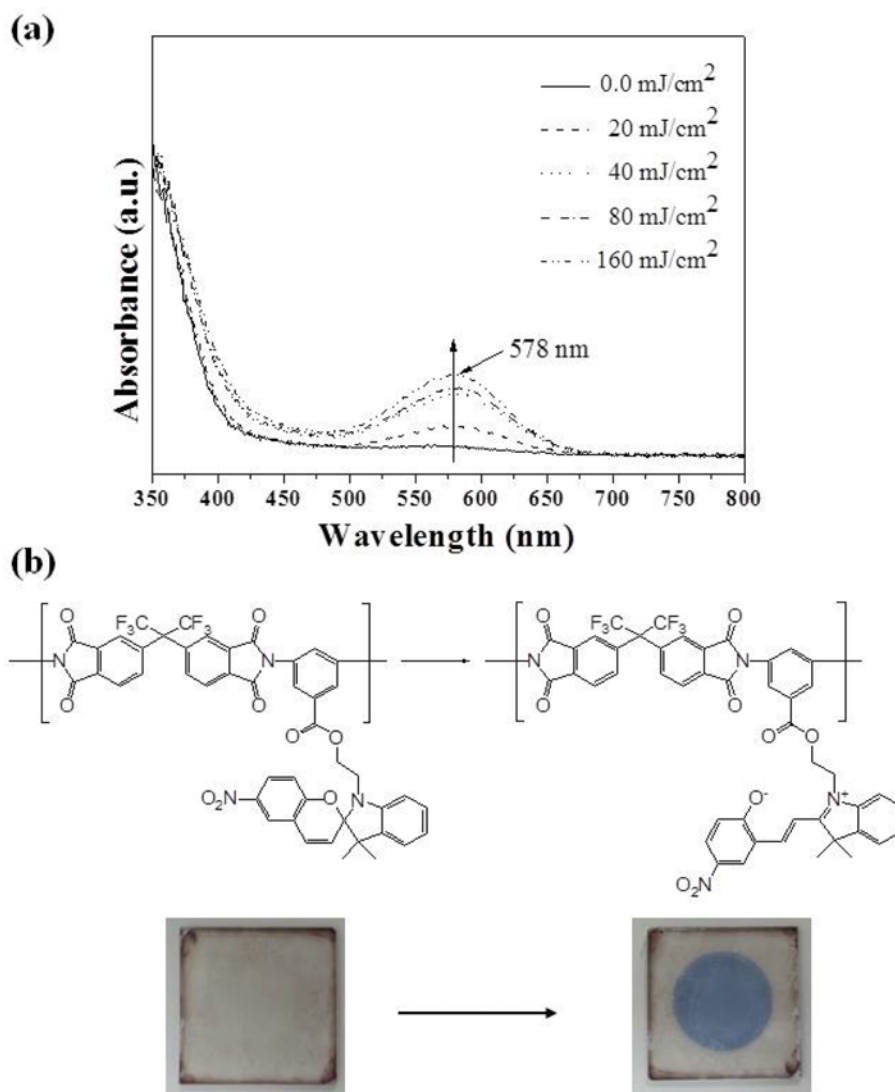
**Fig. 3.** Typical  $I$ - $V$  curves of the ITO/6F-DAB-SP (30 nm thick)/Al devices, which were measured with a compliance current set of 0.1 A. The applied voltage was swept from 0 V  $\rightarrow$  -3.0 V. The electrode contact area was  $0.1 \times 0.1 \text{ mm}^2$ . (a) unexposed device, (b) exposed device.

unexposed devices were initially in the OFF-state, where the current level is below  $9.1 \times 10^{-10} \text{ A}$ , before applying an external voltage to the devices (Fig. 2a). In the first sweep of an operating voltage range (from 0 V to +3 V), an abrupt increase in the current occurred at an input voltage of approximately 1.42 V (which corresponds to the switching-ON threshold voltage), indicating that the film undergoes an electrical transition from the OFF-state to the ON-state. This electrical transition can serve as the 'writing' process in a memory device. This ON-state was maintained even after the input voltage was turned off or during the reverse and forward voltage sweeping with a current compliance of 0.1 A or higher. Similar switching-ON behaviors were observed for the devices when they were swept with a negative voltage (Fig. 3a). These results suggest that the 6F-DAB-SP device exhibits excellent unipolar WORM memory behavior without light exposure. Figures 2b and 3b show the typical  $I$ - $V$  characteristics for UV exposed 6F-

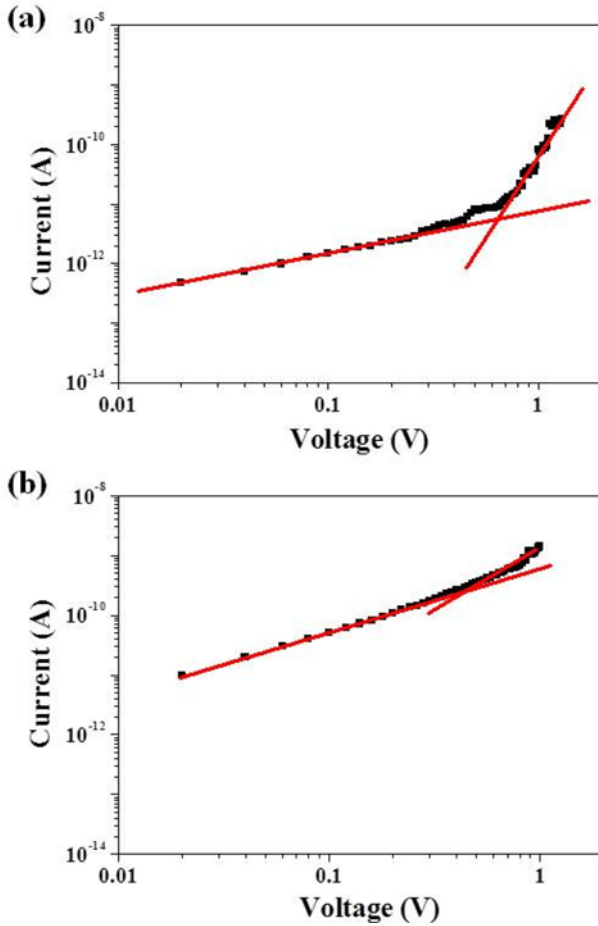
DAB-SP devices, according to the positive and negative sweeping operation, respectively. The UV exposed devices also exhibit WORM memory behavior, regardless of the sweep direction of the applied voltage, which is similar to the unexposed ones; however the electrical performance of the devices before and after UV exposure were somewhat different. When comparing positive voltage swept curves of these devices, the switching-ON voltages and OFF-current level of the exposed device were 1.14 V and below  $2.0 \times 10^{-9}$  A, respectively, whereas the corresponding values for the unexposed device were 1.42 and  $9.1 \times 10^{-10}$  A (Fig. 2a and b). Both the current level and switching-ON voltage of organic WORM device can be affected considerably by two factors, such as the device structure (including electrode combination, roughness, and thickness of active polymer layers) and the molecular structure of the active polymer. In

particular, the electrical and optical properties of memory device can be subject to the HOMO-LUMO band gap controlled by the polymer molecular structure.

UV-Vis spectroscopy of the film was performed to examine the effects of UV exposure on the molecular structure of 6F-DAB-SP. The UV-Vis spectrum for a 6F-DAB-SP indicates a new absorption band in the visible region ( $\lambda_{\max} = 578$  nm) (Fig. 4a). In fact, the spiroopyran moiety triggers a photochromic reaction between the colorless, nonplanar close form and colored, planar open zwitterionic merocyanine form, as shown in Fig. 4b.<sup>[23]</sup> The UV absorption of spiroopyran at  $\lambda_{\max} = 365$  nm allows its structure changes from the close form to the open form (Fig. 4a). The resulting zwitterionic merocyanine can then absorb visible light ( $\lambda_{\max} = 578$  nm) due to the extended conjugation system stemmed by its planar structure<sup>[23,27]</sup> (Fig. 4b). In



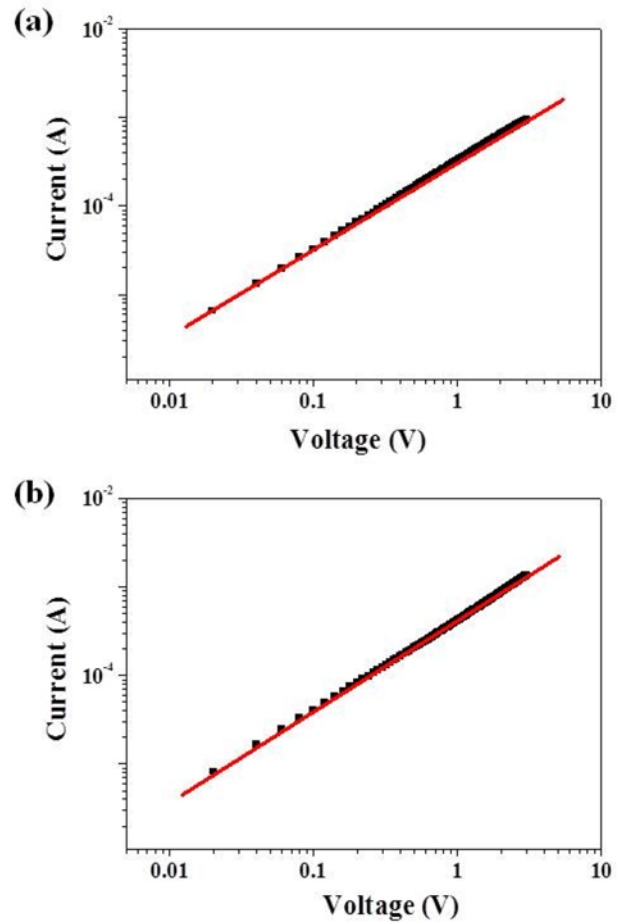
**Fig. 4.** (a) UV-Vis spectrum of a 6F-DAB-SP (b) Structural change in 6F-DAB-SP according to photochemical reaction.



**Fig. 5.** Experimental and fitted  $I$ - $V$  curves for the ITO/6F-DAB-SP (30 nm thick)/Al device: OFF-state with a combination of the space-charge-limited conduction model, (a) unexposed device, (b) exposed device.

particular, the extended  $\pi$ -conjugation open-ring system of the open zwitterionic form leads to a decrease in the bandgap, which can make the polymer more conducting. Therefore, the UV-exposed device yielded a higher OFF-current and lower switching-ON voltage than the unexposed one.

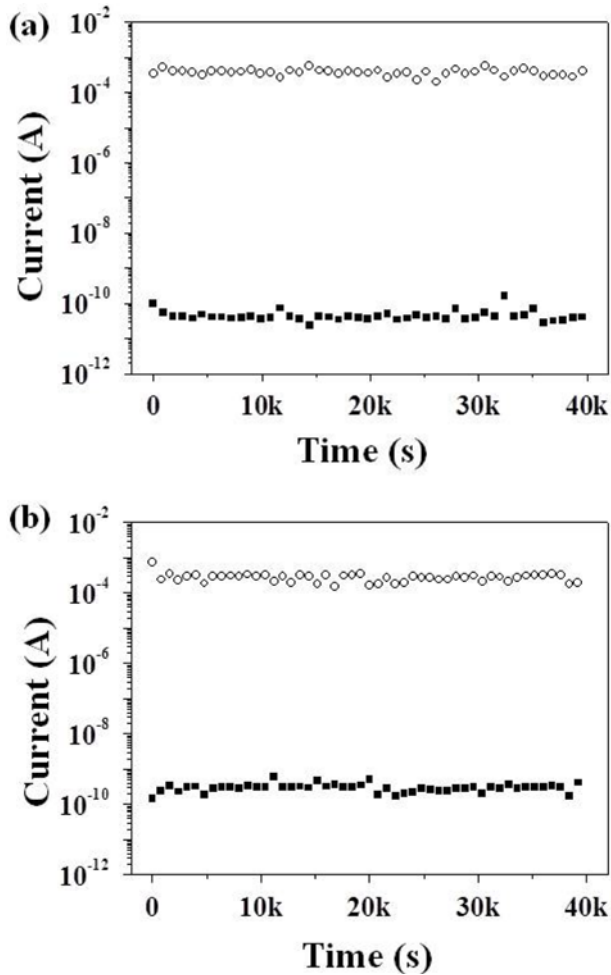
To further examine these memory behaviors, this study analyzed the  $I$ - $V$  characteristics in detail with the space-charge limited conduction (SCLC) model, which has been used to examine the trap states in organic molecules and polymers.<sup>[28]</sup> The SCLC model was found to match the  $I$ - $V$  data for the OFF-state of devices treated with/without UV exposure (Fig. 5). From the data, both the  $I$ - $V$  data of the OFF-state (Fig. 5a, b) could be fitted by the trap-limited SCLC model (a trap-limited SCLC model following the equation:  $I = 9A\epsilon\epsilon_0\mu V^2/8d^3$ , where  $\epsilon\epsilon_0$  is the absolute permittivity of the active layer,  $d$  is the thickness of the polymer film, and  $\mu$  is the carrier mobility). This means that a logarithmic plot of the  $I$ - $V$  results contains two linear



**Fig. 6.** Experimental and fitted  $I$ - $V$  curves for the ITO/6F-DAB-SP (30 nm thick)/Al devices: ON-state with a Ohmic current model, (a) unexposed device, (b) exposed device.

regions based on the specific range. In particular, the exposed device showed higher OFF current density of  $2.45 \times 10^{-9} \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$  at near 1 V than that of the unexposed device ( $5.83 \times 10^{-10} \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ ), as well as the former indicated smaller difference of slopes for two linear lines than the latter (see red lines in Fig. 5a and b). This is due to the extended conjugation system of the open zwitterionic merocyanine form of 6F-DAB-SP after UV exposure, namely the open form has a lower trap density than the closed form, which is consistent with the UV-vis spectrum. The trap density of a polymer memory material is a critical factor determining the switching-ON voltage of the device. When a bias is applied to the film, these trap sites become filled with injected charge carriers, and at the critical voltage (i.e., the switching-on voltage) the trapped charges flow through the trapped sites as a result of a hopping process. Therefore, the switching-ON voltage of the exposed device is lower than that of the unexposed one. In addition, the ohmic contact model matched the  $I$ - $V$  data for the ON-state of both devices. The ohmic conduction model is  $J = qn\mu V/d$ ,





**Fig. 7.** Retention times of the ON- and OFF-states of the ITO/6F-DAB-SP (30 nm thick)/Al device, as probed with a reading voltage of +0.5 V: (a) unexposed, (b) exposed device.

where  $q$  is the electronic charge,  $n$  is the charge barrier density,  $\mu$  is the carrier mobility, and  $d$  is the film thickness. As shown in Fig. 6, the logarithmic plot of the  $I$ - $V$  data for the ON-state contains a linear region with a similar slope of ca. 1.0 (0.99 for devices before UV exposure (a) and 1.01 for cells after UV exposure (b)), which implies that ohmic conduction is dominant in the ON-state of all two devices.

To further investigate the stability of the WORM memory characteristics, ON/OFF current ratio and retention time were measured. Figure 7 a and b show the retention times of the ON- and OFF-states of the ITO/6F-DAB-SP (30 nm thick)/Al devices before and after UV exposure, respectively. Both devices showed that the OFF-state was retained without any degradation. After being switched on by applying a voltage of 2.0 V, and the ON-state retained stable at a voltage of +0.5 V without any degradation for at least  $10^4$  s.

## 4. CONCLUSIONS

A new polyimide containing a spiropyran moiety in the side chain (6F-DAB-SP) was synthesized, and unipolar WORM memory device using spiropyran-based PI was demonstrated to be an active layer. The 6F-DAB-SP-based memory devices showed good memory performance regardless of UV-exposure. On the other hand, the memory device after UV exposure yielded a higher current and lower switching-ON voltage than the unexposed device because of the structural change in the spiropyran moiety after UV exposure: from nonplanar close form to colored, planar open zwitterionic merocyanine form. The open form of the spiropyran moiety induced an extended conjugation system, leading to a lower trap density than the unexposed device. Therefore, the exposed device showed a higher current and lower switching-on voltage.

## ACKNOWLEDGMENTS

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