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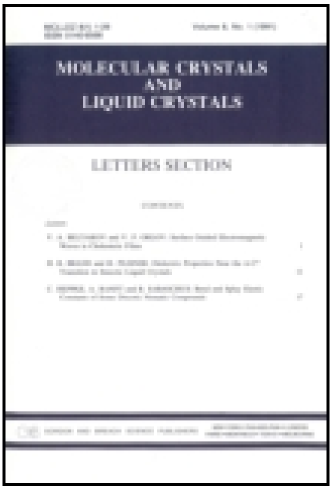

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Diels-Alder Crosslinked Block-Copolymer Gate Dielectrics for Low Voltage Operated Top-Gate Organic Field-Effect Transistors

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Diels-Alder Crosslinked Block-Copolymer Gate Dielectrics for Low Voltage Operated Top-Gate Organic Field-Effect Transistors

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*We report a cross-linkable diblock copolymer at a mild temperature by Diels-Alder click-chemistry for a gate dielectric layer in the top-gate, bottom contact organic field-effect transistors (OFETs). The Diels-Alder reaction between poly[(methyl methacrylate)-*co*-(9-anthracenyl methyl methacrylate)] (P(MMA-AMA)) and 1,1′-(methylenedi-4,1-phenylene)bismaleimide (MPB) enables the preparation of robust cross-linked films at 100 °C annealing for 10 min. Poly(9,9-dioctylfluorene-*alt*-bithiophene (F8T2) or poly(3-hexylthiophene) (P3HT) OFETs exhibited charge carrier mobilities of $3 \sim 4 \times 10^{-3}$ $cm^2\ V^{-1}\ s^{-1}$ with the 250 nm thick crosslinked (P(MMA-AMA)) films at gate voltages of less than −20 V. Moreover, the ratio of MMA and AMA was controlled to observe the effects of crosslinking density on the properties of the gate dielectric layer. When the portion of a crosslinkable anthracenyl unit in (P(MMA-AMA)) is increased to 50 mol.% in the base polymer, the gate leakage current of OFETs decreases below 10^{-8} A at $V_g = -20$ V.*

Keyword Organic field-effect transistors; thin-gate dielectrics; Diels-Alder reaction; crosslinked polymer; conjugated polymer; low power operated transistors; charge carrier mobility

Introduction

Organic field-effect transistors (OFETs) have attracted significant interest for the potential to realize various flexible electronic and opto-electronic devices using cost-effective graphic art printing processes [1–5]. Over the last two decades, impressive progress has been made

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in the development of high performance organic semiconductors, the improvement of the arrangement of semiconducting molecules in an active layer, and the optimization of the process condition [1, 2, 5–8]. However, developments of organic gate dielectric materials for a low voltage operation have been considered less often than the semiconductor materials for high charge carrier mobility. While the charge carrier mobilities of organic semiconductors have surpassed those of amorphous silicon FETs, the common OFETs have operated under a very high gate bias, normally greater than 30 $\sim$ 50 V [1, 2, 5–8]. Most high dielectric constant (κ) materials, which are applied to low voltage driving transistors are based on metal-oxides (such as alumina, titania, and tantalum oxide). They are usually brittle and require a high temperature annealing process [9–12]. Furthermore, the development of solution-processable dielectrics for low-voltage operated transistors has focused mainly on a blend and bilayer system of polymers and inorganic oxide materials [13–15]. or on high permittivity polyelectrolyte dielectrics [16, 17] suffering low-switching speeds.

As the alternative and straightforward approach, reducing the thickness of the gate dielectrics can effectively reduce the operating voltage of the OFETs by increasing the capacitance. However, with conventional polymer insulators such as poly(methyl methacrylate) (PMMA) and polystyrene, without any chemical additives or crosslinking agents it is very difficult to achieve robust dielectric films less than 300 nm without pinholes because the inherent large free volumes lead to a low break-down voltage [18, 19]. Therefore, due to the large free volumes in normal polymer films, cross-linked polymers have been necessary for low operating transistors. Typical examples of crosslinking processes are using Diels-Alder cycloaddition of benzocyclobutene (BCB) precursor [20] or crosslinking between poly(4-vinylphenol) (PVP) with poly(melamine-co-formaldehyde) methylated (as a crosslinking agent) [21] to form robust gate dielectric films. Most of the cross-linkable dielectric polymers, including those above, must be annealed at a high temperature of over 180°C for a long time to obtain complete chemical crosslinking. This means that cross-linkable polymers are difficult to apply with tog-gate and bottom-contact (TG/BC) device geometry as damage can occur to the under-laid organic semiconductors during the annealing process. Therefore, gate dielectric polymers which can be cross-linked at a mild temperature should be developed for TG/BC OFETs.

In this paper, we report a cross-linkable di-block polymer at a mild temperature for a thin gate dielectric layer of TG/BC OFETs. The prime advantage of the polymer is that the crosslinking reaction takes place only at a barely moderate low temperature ($\sim$100°C) and therefore shows a high compatibility with plastic substrates. The Diels-Alder reaction between poly[(methyl methacrylate)-*co*-(9-anthracenyl methyl methacrylate)] (P(MMA-AMA)) and 1,1′-(methylenedi-4,1-phenylene)bismaleimide (MPB) enables robust cross-linked films to be fabricated at 100°C annealing for 10 min. TG/BC F8T2 and P3HT OFETs showed the saturation field-effect mobility (μ_{FET}) of 3.2 $\sim$ 3.8 $\times$ 10^{-3} cm^{2}V^{-1}s^{-1} and 4.2 $\sim$ 4.3 $\times$ 10^{-3} cm^{2}V^{-1}s^{-1}, respectively, with the 250 nm thick crosslinked dielectric layer.

Experimental Section

Preparation of Substrates

Corning Eagle 2000 glass was used as the substrates and cleaned with de-ionized water, acetone, and isopropanol in an ultrasonic bath for 10 min sequentially. The Au/Ni (12 nm/3 nm thick) patterns with channel length (L) 2$\sim$20 μm and width (W) 1 mm for the source/drain electrodes were fabricated on the glass from a conventional lift-off photolithography procedure. Afterwards, the source/drain electrodes were treated with PFDT (1*H*,1*H*,2*H*,2*H*-Perfluorodecanethiol; Sigma-Aldrich Co.) to reduce the contact resistance between the work functions (5.0 eV) of Au and the HOMO level (5.5 eV) of F8T2 (Poly(9,9-dioctylfluorene-*alt*-bithiophene; American Dye Source Inc.). The self-assembly was performed by immersing the substrate in isopropanol solution (0.01 mM) of PFDT for over 12 hours, followed by thorough rinsing with isopropanol and drying in a flow of high-purity nitrogen (N_2) gas. F8T2 and regioregular poly(3-hexylthiophene-2,5-diyl) (rr-P3HT; Sigma-Aldrich Co.) were used as semiconducting polymers without further purification. The semiconducting polymers (F8T2; 7 mg/ml, P3HT; 10 mg/ml) were dissolved in anhydrous *p*-xylene and 1,2,4-trichlorobenzene, respectively. Subsequently, the semiconductor solutions were spin-coated with 2000 rpm for 60 sec in a N_2-filled glove box for F8T2 and inkjet-printed for P3HT in air. The F8T2 and P3HT films were baked at 80°C for 10 min and then annealed at 100°C for 30 min to remove the resident solvents and induce the high molecular ordering.

Insulator Film Preparation

Polymer dielectrics of poly[(methyl methacrylate)-*co*-(9-anthracenyl methyl methacrylate)] (P(MMA-AMA)), with around 30 mol% and 50 mol% (7:3 and 5:5 ratios) of a block of anthracenyl methyl methacrylate (AMA), were synthesized in our laboratory based on a previous report [22]. P(MMA-AMA) with 10 mol% of AMA (9:1 ratio) and 1,1′-(Methylenedi-4,1-phenylene)bismaleimide (MPB) were purchased from Sigma-Aldrich. In order to prepare the solutions of polymer dielectrics with crosslinking agents, P(MMA-AMA) at various ratios were mixed with MPB in a 1:1 volume ratio in 1,2-dichloroethane (DCE) at room temperature and the mixture was then filtered via a 0.2 μm polytetrafluoroethylene (PTFE) filter. The blended solutions (40 mg/ml) were spin-coated at 2000 rpm for 60 sec onto the semiconductors, and annealed at 100°C for 30 min to activate the cross-linking reaction.

Characterization of Films and Devices

The overall film thicknesses were measured using a surface profiler (XP-1; Ambios Technology). The UV/Vis absorption spectroscopy for crosslinked dielectrics was measured using a Perkin Elmer, Lambda 750, and a UV/Vis spectrometer. For the complete top-gate and bottom contact transistors, gate electrodes were patterned by thermal evaporation of aluminum (~30 nm) through a shadow mask. The electrical characteristics of field-effect transistors (FETs) were measured and calculated in the saturation regime using a Keithley 4200-SCS semiconductor parameter analyzer connected to a standard probe station in inert N_2 conditions.

Results and Discussion

Figure 1(a) shows the chemical structure of P(MMA-AMA) (as a host polymer) and MPB (as a crosslinking agent). These materials could react through the Diels-Alder. i.e., click chemistry between anthracenyl side chains on P(MMA-AMA) and maleimido moieties on MPB for crosslinking by simple and mild thermal annealing [22]. From the chemical reaction, the host polymers would be interconnected to form polymer networks, as shown in Figure 1(b). To study the effects of crosslinking density on properties of gate dielectrics and characteristics of OFETs, we synthesize P(MMA-AMA) with various coupling ratios,

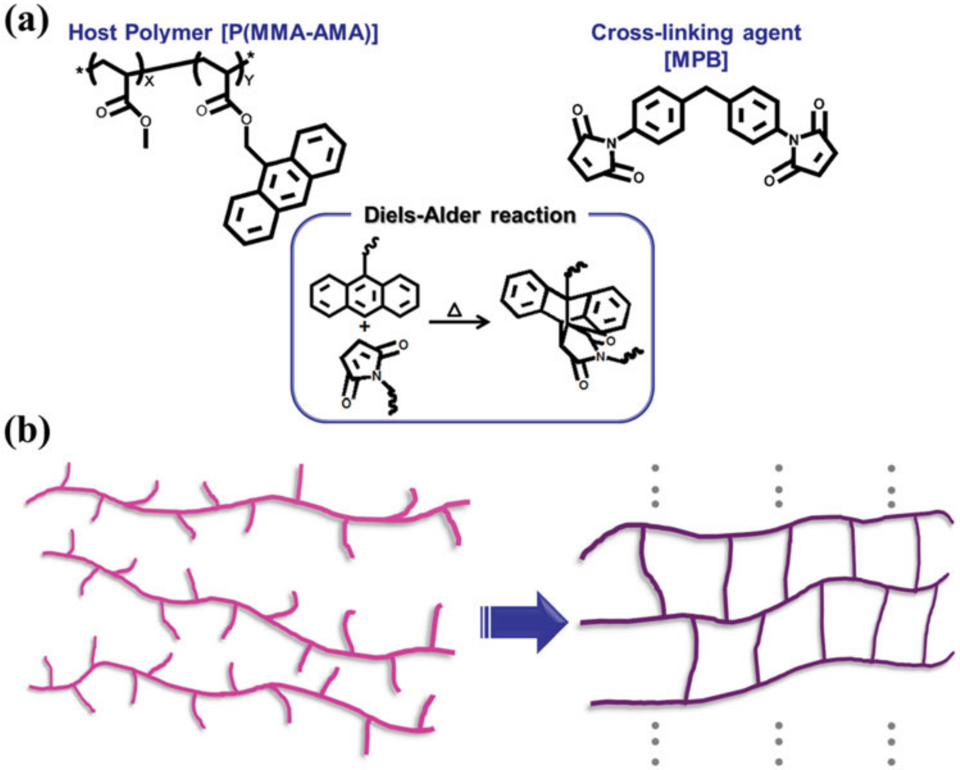

Figure 1. (a) Chemical structure of poly[(methyl methacrylate)-*co*-(9-anthracenyl methyl methacrylate) (P(MMA-AMA)) and 1,1′-(Methylenedi-4,1-phenylene)bismaleimide (MPB) used as a base polymer and crosslinking agent for gate dielectrics. (b) Schematic illustrations of Diels-Alder crosslinking reactions between P(MMA-AMA) and MPB by thermal energy.

5(MMA) : 5 (AMA), 7:3, and 9:1. The highest crosslinking density is expected with P(MMA-AMA) [5:5]. To check the crosslinking temperature, P(MMA-AMA) [9:1] and MPB were dissolved in 1,2-dichloroethane at a concentration of 40 mg/ml, respectively. In addition, the solution was mixed with a 50% volume ratio. Finally, the solution was filtered through a 0.2 μm PTFE filter and spin-coated onto glass substrates at 1000 rpm for 60 s. The films were then dried at various temperatures (30, 70, 90, 110, and 130°C) on a hot plate for 10 min in air. In UV-vis absorption spectra, the pristine sample (baked at 30°C) exhibited four absorption peaks in the range from 300 to 450 nm (Figure 2). These four peaks at 336, 351, 370, and 390 nm are typical absorption peaks which were observed from the anthracene unit [22]. However, these anthracenenyl absorption peaks completely disappeared when the annealing temperature increased to 90°C. This result shows that the maleimido groups in MPB and the anthracenyl groups on P(MMA-AMA) reacted sufficiently to each other, which means the crosslinking reaction occurred at around 90°C. The capacitance per unit area (C_i) of cross-linked polymer dielectric was calculated using ‘$C_i = \varepsilon_0\kappa/d$’, where ε_0 is the permittivity of free space, κ is the dielectric constant of an insulator material, and d is the thickness of the dielectric layer. The resultant capacitances for the cross-linked films of P(MMA-AMA) and MPB were calculated by the dielectric constant value of 3.5 for 10, 30, and 50 mol% AMA blocks.

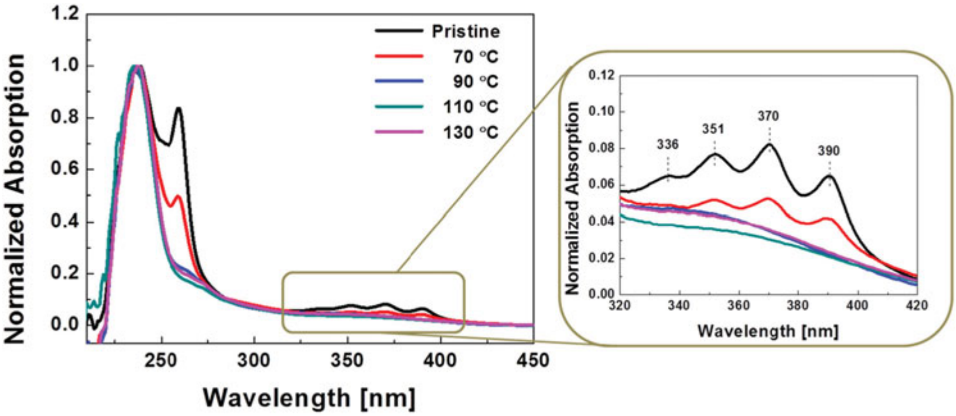

Figure 2. UV-vis absorption spectra of blend films, which were spincoated from the mixed solution of P(MMA-AMA) [9:1] and MPB at a 1:1 volume ratio. The films were annealed at 30 (pristine), 70, 90, 110, and 130°C. In the right panel, the absorption spectra were maximized in the range from 320 and 420 nm.

To fabricate top-gate and bottom-contact polymer FETs (Figure 3(a)), F8T2 or P3HT (Figure 3(b)) were spin-coated or inkjet printed on glass substrates with a pre-patterned Au source-drain electrode, and then solutions of precursor materials (P(MMA-AMA) and MPB) were spin-coated onto the semiconducting films in a nitrogen filled glove box. The DCE solvent for dielectric materials was selected to provide not only a good solubility for the insulating polymers and crosslinking agent but also a completely poor solubility for the

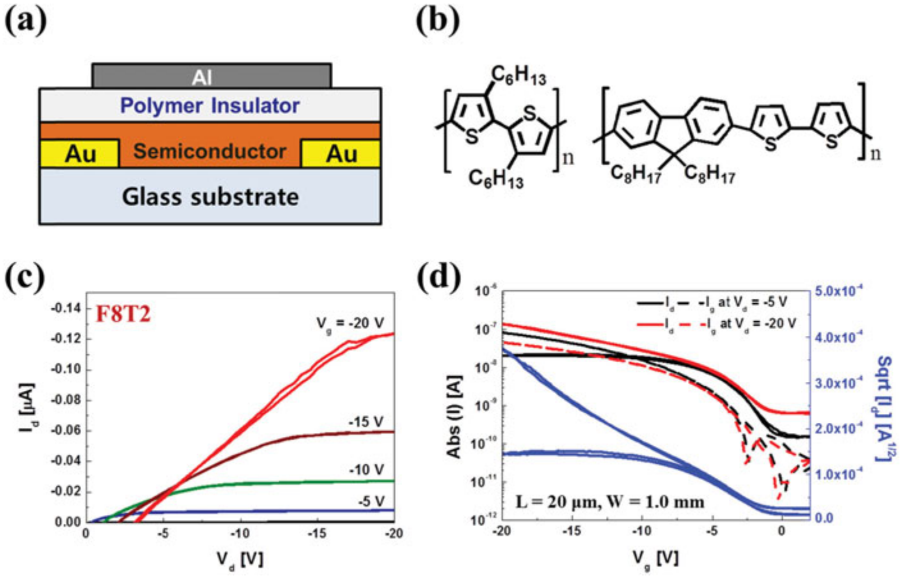

Figure 3. (a) Schematic cross-section of the top-gated polymer transistor used in this study. (b) The chemical structures of P3HT (left) and F8T2 (right) as active layers. (c) Output and (d) Transfer curves of F8T2 OFETs with a crosslinked insulator of P(MMA-AMA) [9:1].

underlying semiconductor films to avoid dissolution or swelling [23]. The corresponding thickness of all dielectric layers was 250 nm and the resultant capacitances of the insulators are approximately 12.4 nF/cm^2. The field-effect mobilities were calculated in the saturation regime ($V_{DS} = -20$ V) of the I_{DS} using the following equation [6].

$$I_D = \frac{\mu C_i W}{2L}(V_G - V_{th})^2 \text{ for } |V_D| > |V_{G-}V_{th}| > 0 \text{ (saturation regime)}$$

where C_i is the capacitance of the dielectric layer, I_{DS} is the source-drain current, and V_D, V_G, and V_{Th} are the source-drain, gate, and threshold voltages, respectively. Current on/off ratios (I_{on}/I_{off}) were determined from the minimum current (I_{off}) and the current at $V_G = -20$ V (I_{on}). As described in Table 1, the base polymer containing various AMA block ratios (9:1, 7:3 and 5:5) were prepared and mixed with MPB as a crosslinker in DCE to check the effects of crosslinking density on the properties of a dielectric layer. All devices were operated below −20 V with saturation field-effect mobility (μ_{FET}) of $3.2 \sim 3.8 \times 10^{-3}$ cm^{2}V^{-1}s^{-1} in the case of F8T2 and $4.2 \sim 4.3 \times 10^{-3}$ cm^{2}V^{-1}s^{-1} for P3HT, which are demonstrated for similar charge carrier mobilities in the previous reports [19]. All device parameters are also summarized in Table 1. These charge carrier mobilities for F8T2 and P3HT are quite uniform at all AMA ratios, implying that the crosslinking density did not affect the charge transport at the interface between the dielectric layer and the semiconductor. Figures 3(c) and 3(d) show the typical transfer and output curves of the F8T2 semiconductor with a crosslinked dielectric of P(MMA-AMA) [9:1]. A high gate-leakage current exists, which is near 10^{-7} A at a −20 V gate voltage (Fig. 3(d)). There might be many leakage-current paths through the dielectric layers due to the pinholes extending through the film and relatively low breakdown voltage strength of the thin dielectric layer (thickness : ~250 nm) [3, 18, 19, 24]. Generally, an ideal transistor device should show zero source-drain currents at V_d and V_g of 0 V. However, as shown in Fig. 3(c), distortion of the drain current curves occurs in the linear region at a low drain voltage owing to the large I_d offset, which is clearly shown up to −20 V of V_g. The increase of positive current at $V_d = 0$ V with increments of V_g is primarily due to the leakage current through the gate insulator layer between source/drain and gate electrode [25]. These results could show that the large I_d offset is closely related to the high leakage current (I_g) induced from the gate voltage for P(MMA-AMA) [9:1].

Table 1. OFET performances[a)] of semiconductor films using the cross-linked polymer dielectrics with various ratios of a crosslinking agent

Condition	F8T2				P3HT			
Ratio[b)]	μ_{FET} [cm^2 V^{-1} s^{-1}]	V_{th} [V]	$S.S$ [V/dec]	I_{on}/I_{off}	μ_{FET} [cm^2 V^{-1} s^{-1}]	V_{th} [V]	$S.S$ [V/dec]	I_{on}/I_{off}
9:1	3.2×10^{-3}	−7.9	2.9	218				
7:3	3.8×10^{-3}	−5.4	1.4	3154	4.3×10^{-3}	5.9	12.6	49
5:5	3.2×10^{-3}	−2.8	1.4	6108	4.2×10^{-3}	7.3	10.7	74

a) The device performances were calculated in the saturation region.

b) This indicates the component ratio between base polymers and crosslinking agents. (ie. polymers : agents)

In the case of P(MMA-AMA) [7:3] (shown in Fig. 4), the transistor of F8T2 also demonstrated the large gate leakage currents, which might affect the distorted I_d offset. This means that the 30 mol% AMA units are still not sufficient in quantity to crosslink all of the films. However, in P3HT OFETs, the drain currents at low drain voltages show almost a coincident onset because the P3HT films were patterned within channel regions by ink-jet printing to reduce gate leakage current paths. By the patterning method for the semiconducting layer, the gate leakage currents were effectively reduced in the range below 10^{-9} A, compared with the spin-coated F8T2 film. Another problem using 250 nm thick polymer layers as the gate dielectrics will be a low on/off drain current ratio resulting from the increased possibility of leakage paths because a high on/off current ratio is essential as a switching function of OFETs.

The gate leakage currents effectively decreased below 10^{-8} A at $V_g = -20$ V when the AMA block ratio ultimately increased to 50 mol% in P(MMA-AMA) [Fig. 5]. Moreover, the on/off source-drain current ratios of F8T2 OFETs with 5:5 ratio increased to 6108 from 218 at 9:1 ratio along with the opposite trend of gate-leakage currents. In addition, the subthreshold swing of F8T2 OFETs was decreased to 1.4 V/dec from 2.9 V/dec. As the portion of the AMA block increases, the possibility and population of crosslinking reaction in dielectric layers would be eventually increased. The μ_{FET} of OFETs depends mainly on the semiconductor itself, and on the morphology and crystallinity of the film, and on the physical and chemical properties of the gate dielectric layer. Based on the many previous reports [1, 2], the roughness and hydrophobicity of gate dielectric layers were the main sources influencing μ_{FET}. In this study, the increase of crosslinking density in P(MMA-AMA) films provides a decrease of air free volume in the gate dielectric films without any

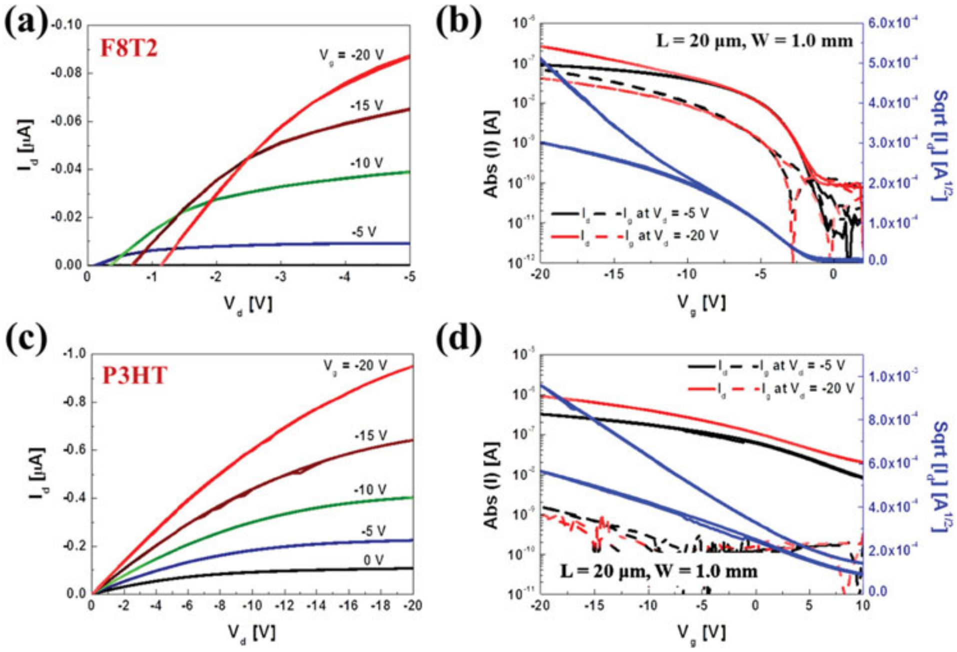

Figure 4. Typical (a,c) output and (b,d) transfer characteristics of (a,b) F8T2 and (c,d) P3HT transistors with 250 nm thick crosslinked dielectric layers of P(MMA-AMA) [7:3] in top-gate and bottom-contact configurations.

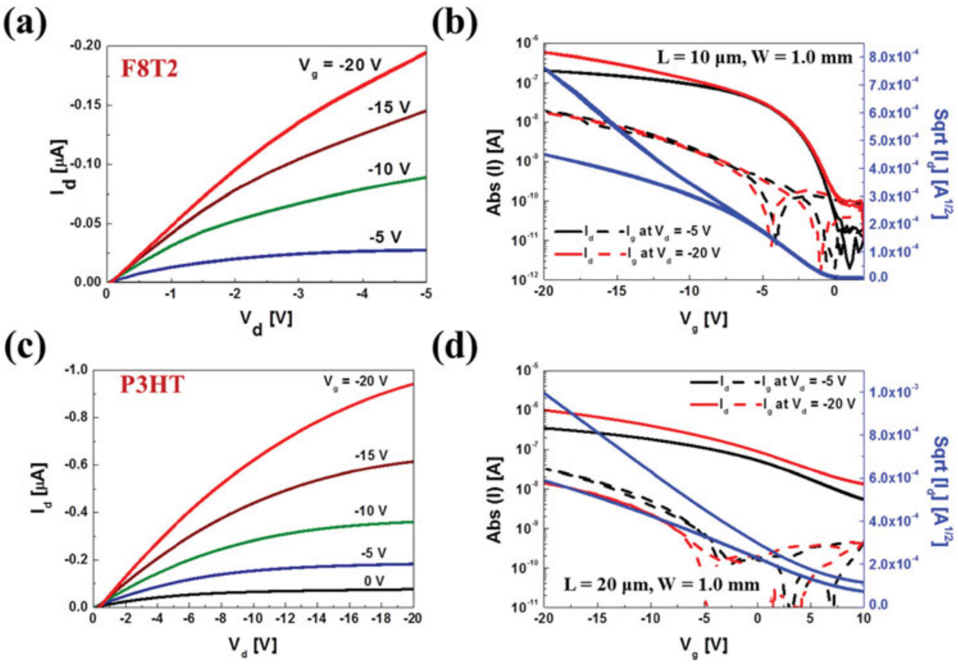

Figure 5. Typical output (a,c) and transfer characteristics (b,d) of F8T2 and P3HT transistors with 250 nm thick crosslinked dielectric layers of P(MMA-AMA) [5:5] in top-gate and bottom-contact configurations.

other changes of physical and chemical properties. Therefore, only the robustness of a gate dielectric film, i.e., the breakdown voltage and the leakage current density, were improved by increasing the crosslinking density of these polymers.

Conclusion

In conclusion, we presented crosslinked P(MMA-AMA) as a gate dielectric layer for solution processed TG/BC OFETs. The Diels-Alder cycloaddition reaction enables crosslinking of the polymers in a mild temperature process (at ∼100°C) and the damage to the lower polymer semiconducting layer could be minimized. In addition, we also controlled the ratio of MMA and AMA in the diblock copolymers to check the effects of crosslinking density on the properties of thin gate dielectric layers. Eventually, the crosslinked copolymer at a 5:5 ratio between MMA and AMA gave the best device characteristics with low gate leakage currents below 10^{-8} A at $V_g = -20$ V.

Acknowledgments

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