

Enhanced Charge Transport in Polymer Thin-Film Transistors Prepared by Contact Film Transfer Method

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ABSTRACT The charge-carrier transport in the structures formed at the surface of various conjugated polymer films is investigated by constructing organic thin-film transistors using a novel and simple contact film transfer method. Thin-film transistors prepared by this transfer process have higher field-effect mobility values compared with conventional spin-coated devices for all the studied polymers. In contrast to previous reports, the hole mobility in regioregular poly(3-alkylthiophene)s does not depend on the length of the alkyl chain when the contact film transfer method is used. These results suggest that the thiophene rings adopt a highly ordered edge-on orientation and strong interchain π - π interactions spontaneously form at the polymer/air interface during the spin coating.

KEYWORDS: Organic field-effect transistor • film transfer • conjugated polymer • interfacial property

INTRODUCTION

Control of molecular packing and orientation at the interface of semiconducting materials is of high importance for achieving superior performance in organic electronic devices such as thin-film transistors (TFTs) (1), light-emitting diodes (LEDs) (2, 3), and photovoltaic devices (PVs) (4). For example, the carrier mobility of thiophene-based polymers such as regioregular poly(3-hexylthiophene) (RR-P3HT) in TFTs strongly depends on their molecular orientation at the polymer/dielectric interface (5, 6). Therefore, to achieve high mobility in the polymer TFTs, it is often necessary to modify the dielectric surface to induce edge-on close-packing at the interface, which is advantageous for lateral charge transport (7–9). However, even with surface modifications, precise control of interfacial structures is difficult during dynamic solution-based coating processes because of the many variable factors involved, such as the wettability of the dielectric layers, molecular weight of the polymers, viscosity of the solution and identity of the solvents used for casting. As a result, a wide range of mobility values ranging from 1×10^{-4} to $0.1 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ have been reported for RR-P3HT (10–15).

The polymer/air interface formed during the coating process, on the other hand, is another interface that could

be utilized for charge transport. Although contradicting results have been reported (16–19), it has been suggested that the structural order at the surface of RR-P3HT films is high. Hao et al. have reported that RR-P3HT films coated at low spin-coating speeds are highly ordered with the side chains normal to the substrate and strong interchain π - π interactions at the polymer/air interface, based on investigations using a combination of near edge X-ray absorption fine structure spectroscopy (NEXAFS), ultraviolet photoelectron spectroscopy (UPS), and penning ionization electron spectroscopy (PIES) (19). This orientation is possibly because of the low surface energy of alkyl side chains that prefer to migrate to the polymer/air interface to form a thermodynamically favorable structure. Interestingly, this molecular orientation is advantageous for charge transport in the lateral direction. Therefore, if transistors can be constructed using this oriented surface layer, high carrier mobility could be achieved in the transistors. Obviously, fabrication of the top-gate transistors by directly depositing the dielectric layers on top of the polymer films would be a straightforward approach. However, in these processes, the structures of the underlying semiconducting layers could be altered by solvents, vacuum deposition or heat. In addition, the differing properties of the dielectric layers of top-gate and bottom-gate devices make comparison of the transport properties between these two interfaces rather difficult (20).

In this paper, we introduce a novel film transfer approach based on contacting and floating off processes of the films. Organic films having areas of more than several square centimeters can be transferred onto various target substrates

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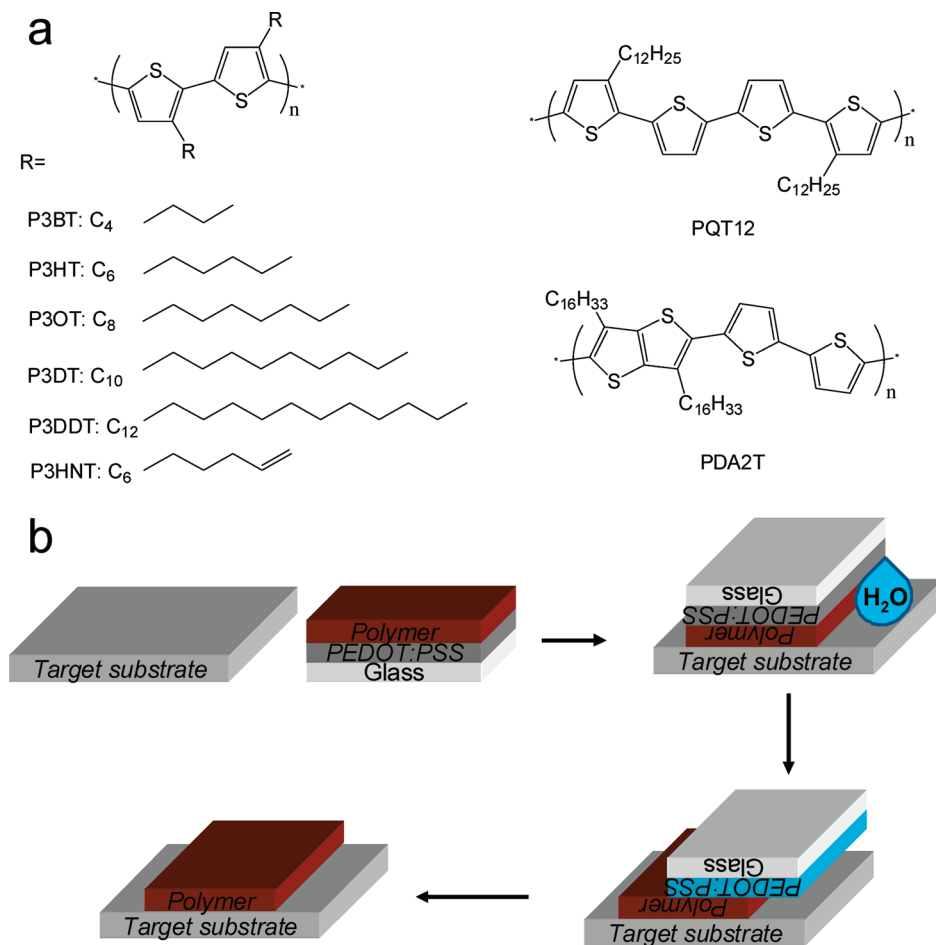


FIGURE 1. (a) Chemical structures of conjugated polymers used in this study. (b) Schematics of transfer process used to deposit polymer film onto target substrate.

without damage. This film transfer process inverts the film geometry; therefore, bottom-gate TFTs fabricated from the transferred films have transport layers originally formed at the polymer/air interface. This fabrication process has advantages over top-gate TFTs for preserving interfacial structures during the transfer process because neither external forces nor thermal treatment is necessary. In addition, because the same materials can be used as the dielectric layers for both the spin-coated and transferred films, comparison of the results is expected to be straightforward. Although several organic film transfer techniques have been reported recently (21–25), there are few reports on the fabrication of TFTs that use these methods (26). Here, we fabricate high-performance polymer TFTs prepared by contact film transfer from various conjugated polymers and compare them with transistors prepared by conventional spin coating.

EXPERIMENTAL SECTION

Chemicals. Two samples of P3HT (Lisicon SP001, Merck Chemicals; 4002EE, Rieke Metals.) were used as received. PQT-12 was purchased from American Dye Source. PDA2T was received from Merck Chemicals. 2,5-Dibromo-3-butylthiophene, 2,5-dibromo-3-octylthiophene, 2,5-dibromo-3-decylthiophene and 2,5-dibromo-3-dodecylthiophene were purchased from

Rieke Metals. Chlorobenzene (C₆H₅Cl, 99.8%) was purchased from Aldrich. BCB (CYCLOTENE 3000) was received from Nissan Chemicals, Japan. Poly(3,4-ethylenedioxythiophene): poly(styrenesulfonate) (PEDOT:PSS; Baytron P) was purchased from H. C. Starck and used as received.

Synthesis. All reagents were used as received from the manufacturers. P3BT, P3HT, P3OT, P3DT, P3DDT, and P3HNT were synthesized by following the method reported by McCullough et al. (27). After the polymerization, the reaction mixture was quenched with 5 M HCl, following the report by Yokozawa et al. (28). The number-averaged molecular weight (M_n) and the polydispersity index (M_w/M_n) of the polymers were determined by gel permeation chromatography (GPC). The regioregularity of the polymers was confirmed by the peak integrations of ¹H NMR spectra.

Device Fabrication and Characterization. Transistors were built on highly doped n-type (100) Si substrates (<0.02 Ω cm) with 300-nm thermally grown silicon dioxide. The SiO₂ surface was passivated with BCB (CYCLOTENE 3000), which was spin coated from a dilute trimethylbenzene solution, and cross-linked at 250 °C on a hot plate for 3 h in a nitrogen-filled glovebox. The capacitance of the gate dielectric was $C_i = 10.7 \pm 0.7$ nF cm⁻², which was measured by using the charging time. Octadecyltrichlorosilane (OTS) modified SiO₂ was prepared by soaking the substrates in a 5 mM toluene solution of OTS for 12 h in a dry N₂-filled glovebox. For the spin-coated devices, chlorobenzene solutions of the polymers (10 mg mL⁻¹ for P3HT, P3OT, P3DT, and P3DDT; 5 mg mL⁻¹ for P3BT, PQT-12, and PDA2T) were directly spin-coated onto

Table 1. Summary of the Molecular Weight (M_n), polydispersity (PDI), Regioregularity (RR) and TFT Characteristics of the Polymers Used in This Study^a

	M_n	PDI	RR	contact film transfer			spin coating		
				μ (cm ² V ⁻¹ s ⁻¹)	V_T (V)	on/off	μ (cm ² V ⁻¹ s ⁻¹)	V_T (V)	on/off
P3BT	18 000	1.4	>98%	0.05 ± 0.003	-13	5 × 10 ⁵	5 ± 0.1 × 10 ⁻⁵	-2	4 × 10 ⁵
P3HT (Rieke)	20 000	1.8	~94%	0.05 ± 0.008	-9	3 × 10 ⁵	4 ± 0.1 × 10 ⁻⁴	1	3 × 10 ²
P3HT (Merck)	20 000	1.4	>98%	0.07 ± 0.011	-1	5 × 10 ⁵	7 ± 1.3 × 10 ⁻⁴	9	6 × 10 ²
P3HT (Home)	32 000	1.1	>98%	0.06 ± 0.002	-10	2 × 10 ⁴	3 ± 0.9 × 10 ⁻⁵	0.1	3 × 10 ⁵
P3HT (Merck, on OTS-SiO ₂)	20 000	1.4	>98%	0.17 ± 0.04	+18	5 × 10 ⁵	^b	^b	^b
P3OT	25 000	1.2	>97%	0.05 ± 0.005	-8	4 × 10 ⁴	2 ± 0.1 × 10 ⁻⁴	5	8 × 10 ²
P3DT	22 000	1.2	>97%	0.05 ± 0.006	-10	8 × 10 ⁵	2 ± 0.3 × 10 ⁻⁴	5	6 × 10 ²
P3DDT	25 000	1.2	>98%	0.05 ± 0.013	-12	8 × 10 ⁵	1 ± 0.2 × 10 ⁻⁴	-2	4 × 10 ²
P3HNT	28 000	1.5	>97%	0.08 ± 0.003	-7	2 × 10 ²	7 ± 0.3 × 10 ⁻³	-8	2 × 10 ⁵
PDA2T	23 500	3.3		0.04 ± 0.003	-17	2 × 10 ⁵	4 ± 0.1 × 10 ⁻³	-2	1 × 10 ⁵
PQT12	21 000	2.2		0.01 ± 0.001	-12	1 × 10 ⁴	3 ± 0.1 × 10 ⁻³	-13	6 × 10 ²

^a The average mobility values and the standard deviations are calculated from 4–8 device data. ^b Because chlorobenzene does not wet the surface of OTS-treated SiO₂, the solution was completely repelled from the surface during the spin-coating and the reference FET device could not be prepared by spin coating.

the dielectric substrates (600 rpm, 30 s). For the transferred films, substrates with the structure of glass/PEDOT:PSS/polymer were prepared by successive spin-coating of an aqueous solution of PEDOT:PSS and a chlorobenzene solution of the polymer. This polymer film was gently brought into contact with the target substrate with the polymer face down. One drop of water was placed on the edge of the two stacked substrates. The PEDOT:PSS layer was selectively permeated by water. After 1–5 min, the water flowed from one side of the substrate to the other, and the PEDOT:PSS layer was completely dissolved. Finally, the glass substrate was easily detached from the organic layer, resulting in the transfer of the polymer film from the glass to the target substrate. The polymer films have thicknesses in the range of 20–50 nm depending on the concentration of the solutions. Gold electrodes were evaporated onto the surface through a metal mask. The electrical characteristics of the transistors were measured using Keithley 2400 and 6430 source/measurement units at room temperature. All the transistors were measured under ambient conditions.

RESULTS AND DISCUSSION

Figure 1a shows the chemical structures of the semiconducting polymers used in this study. Poly(3-butylthiophene) (P3BT), poly(3-hexylthiophene) (P3HT), poly(3-octylthiophene) (P3OT), poly(3-decylthiophene) (P3DT), poly(3-dodecylthiophene) (P3DDT) and poly(3-(5-hexenyl)thiophene) (P3HNT) were synthesized in our laboratory via the McCullough route (see the Supporting Information) (27–29). Commercial samples of P3HT (Rieke Metals and Merck Chemicals), poly(3,3'-didodecylquaterthiophene) (PQT-12) (8) and poly(2,5-bis(2-thienyl)-3,6-dihexadecylthieno[3,2-b]thiophene) (PDA2T) (30) were also used in this study. The number-averaged molecular weight (M_n), polydispersity index (PDI), and regioregularity (RR) of the polymer samples are shown in Table 1. All the synthesized polymers had high molecular weights ($M_n > 18\,000$), narrow PDIs (<1.5), and high RR (>97%).

The schematic drawing in Figure 1b depicts the contact film transfer process developed in the present study. First, a film with a structure of glass/PEDOT:PSS/polymer was

prepared by successive spin coating of an aqueous solution of PEDOT:PSS and a chlorobenzene solution of the polymer. The PEDOT:PSS layer acts as a “sacrificial layer” in the following transfer process. This polymer film was gently brought into contact with the target substrate with the polymer face down. Note that no external pressure was applied between the two substrates. One drop of water was placed on the edge of the two stacked substrates. The PEDOT:PSS layer was selectively permeated by water because of the insolubility of the semiconducting polymers in water. After 1–5 min, the water flowed from one side of the substrate to the other, and the PEDOT:PSS layer was completely dissolved. Finally, the glass substrate was easily detached from the organic layer, resulting in the transfer of the polymer film from the glass to the target substrate. Besides PEDOT:PSS, other water-soluble polymers such as poly(4-styrenesulfonate sodium salt) (PSS) can also be used as the sacrificial layers in the contact film transfer, and there is little difference in the transfer process and the device performance. A video of the contact film transfer process is available in the Supporting Information.

This simple process can be applied to various substrates such as glass, indium tin oxide (ITO), Si/SiO₂, and flexible polymer substrates. As shown in images a and b in Figure 2, the whole area of the P3HT spin-coated film was uniformly transferred onto either a glass or silicone sheet by using this method. Optical microscope images and atomic force microscopy (AFM) height images of a transferred P3HT film on a glass substrate are shown in images c and d in Figure 2, respectively. In the optical microscope images of the transferred films, no cracking, wrinkling, or pinhole formation was observed. The AFM height images of the transferred films indicate that the surfaces were flat with roughness of less than 10 nm.

The films prepared by the contact film transfer were used to fabricate OTFTs. Transistors with a channel length (L) of 50 μm and width (W) of 8 mm were fabricated in a bottom-gate configuration using highly

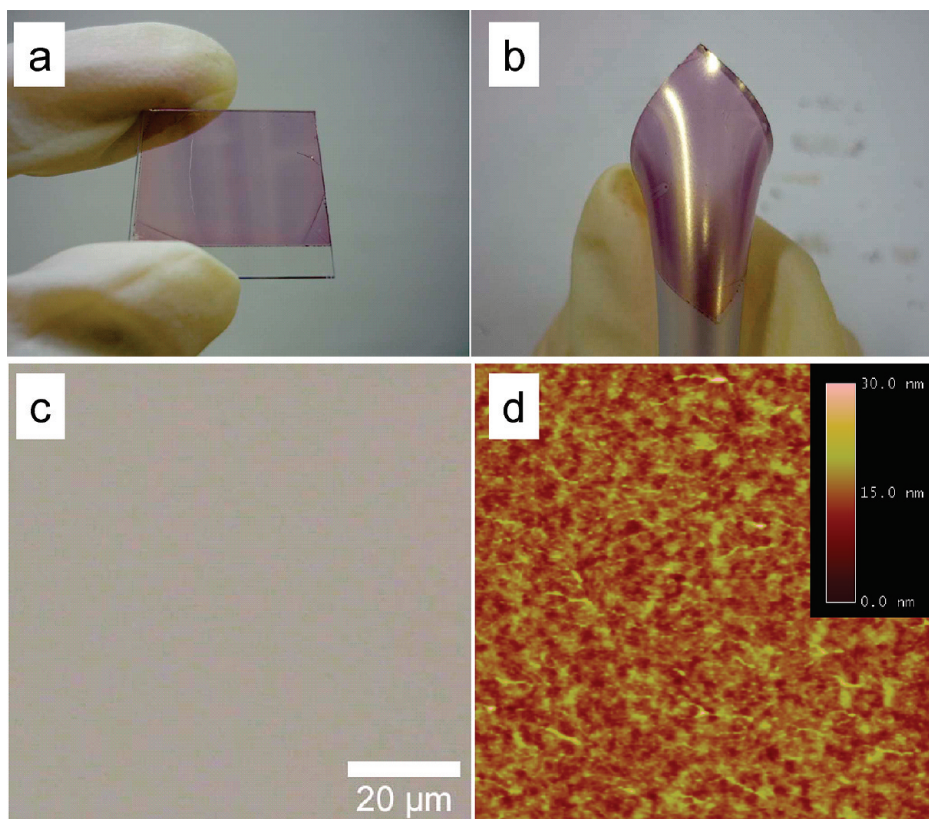


FIGURE 2. (a–d) Photographic images of transferred P3HT films on different substrates: (a) glass substrate and (b) silicone rubber film. (c) Optical microscope image and (d) AFM image ($5 \mu\text{m} \times 5 \mu\text{m}$) of transferred P3HT film on glass substrate.

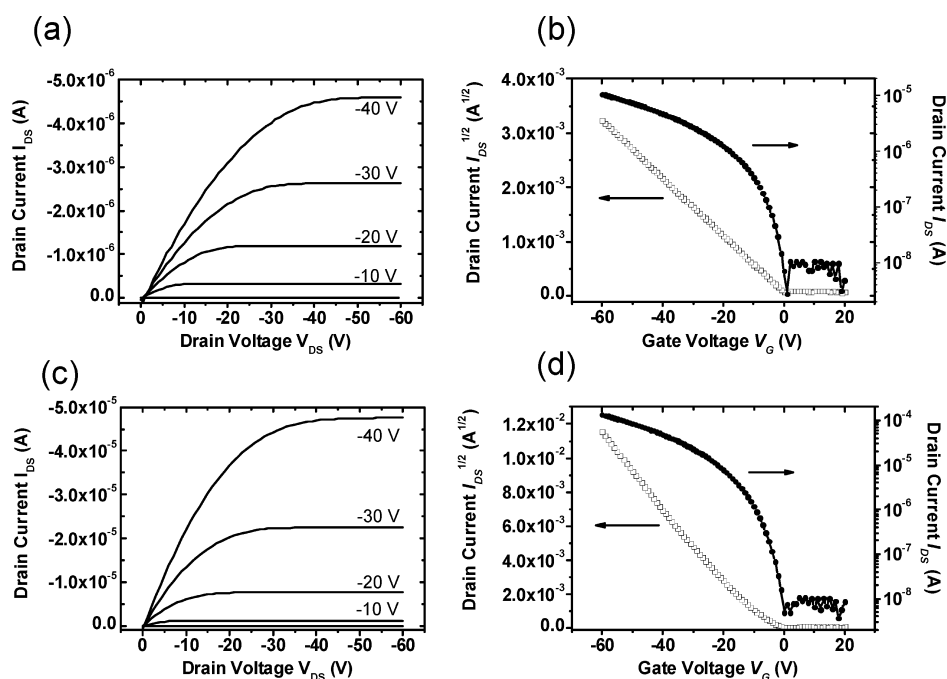


FIGURE 3. OTFT performance of RR-P3HT devices. (a) Output curves and (b) transfer curves of spin-coated P3HT films; (c) output curves and (d) transfer curves of transferred P3HT films on BCB/SiO₂.

doped Si as the gate electrode. A 300 nm SiO₂ layer and an approximately 200-nm divinyltetramethyldisiloxane-bis(benzocyclobutene) (BCB) layer were used as the gate dielectric with a capacitance of $C_i = 10.7 \pm 0.7 \text{ nF cm}^{-2}$.

On the basis of the transfer characteristics measured in the saturation regime ($V_{\text{DS}} = -60 \text{ V}$), we determined the field-effect mobility of each transistor with the following equation

$$\mu_{\text{sat}} = \frac{2L}{WC_i} \frac{I_{\text{DS}}}{(V_{\text{GS}} - V_{\text{T}})^2} \quad (1)$$

First, we compare transistors based on RR-P3HT (synthesized in our lab) films prepared by either conventional spin coating or contact film transfer from the same polymer solution. The output curves and transfer curves are shown in Figure 3. A clear field-effect and well-resolved linear and saturation regions are observed from the output characteristics (Figure 3a and c). For the spin-coated films, the mobility calculated in the saturation region is $4.0 \times 10^{-5} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ with an on/off ratio of 3×10^3 and a threshold voltage of 0.1 V (Figure 3b). However, it should be emphasized that the TFT devices fabricated by contact film transfer of the P3HT film have a field-effect mobility of $0.06 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$, which is 1 order of magnitude higher than that of the devices fabricated by spin coating, with an on/off ratio of 2×10^4 and a threshold voltage of -10 V (Figure 3d). These results clearly show that the carrier transport in the structures formed at the P3HT/air interface is higher than that formed at the P3HT/dielectric interface. The devices fabricated by contact film transfer have off-current values similar to those of the spin-coated devices (smaller than $1 \times 10^{-8} \text{ A}$), indicating that a significant level of unintentional doping of the samples does not occur during the transfer process. The same experiments were performed by using P3HTs from different sources (Rieke Metals and Merck Chemicals). The results are summarized as entries 2–4 in Table 1. The devices prepared by contact film transfer have hole mobilities of one to two orders of magnitude higher than those of the devices fabricated by spin coating for all the polymer samples. When the spin-coated TFTs are compared, the variation in mobility between samples is large (1×10^{-5} to $1 \times 10^{-4} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$). This is possibly because of morphological differences at the dielectric/RR-P3HT interface induced during the spin coating, although a detailed discussion is difficult since numerous factors can affect the structures. In contrast, the devices prepared by contact film transfer have much smaller variation in the mobility values between samples ($0.06\text{--}0.09 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$). This suggests that the surface structure is less sensitive to subtle differences in the polymer properties. These results also show that spin-coated RR-P3HT films form well-ordered edge-on packing structures at the polymer/air interface during the spin coating, which is advantageous for lateral charge transport.

To further confirm our hypothesis regarding the surface structure, we investigated the effect of alkyl side-chain length on the carrier mobility in poly(3-alkylthiophene) films. As shown in Figure 4a, the slope of the transfer curves is smaller for longer alkyl chain lengths in the case of the spin-coated devices. As the result, the mobility values fall from $5 \times 10^{-5} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ for P3BT to $1 \times 10^{-4} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ for P3DDT as side-chain length increases. This dependence of the mobility on the side-chain length agrees with previous reports (31, 32). This result suggests that thiophene rings with longer alkyl side chains adopt a poorer edge-on molecular orientation during the spin coating, which is likely the result of the

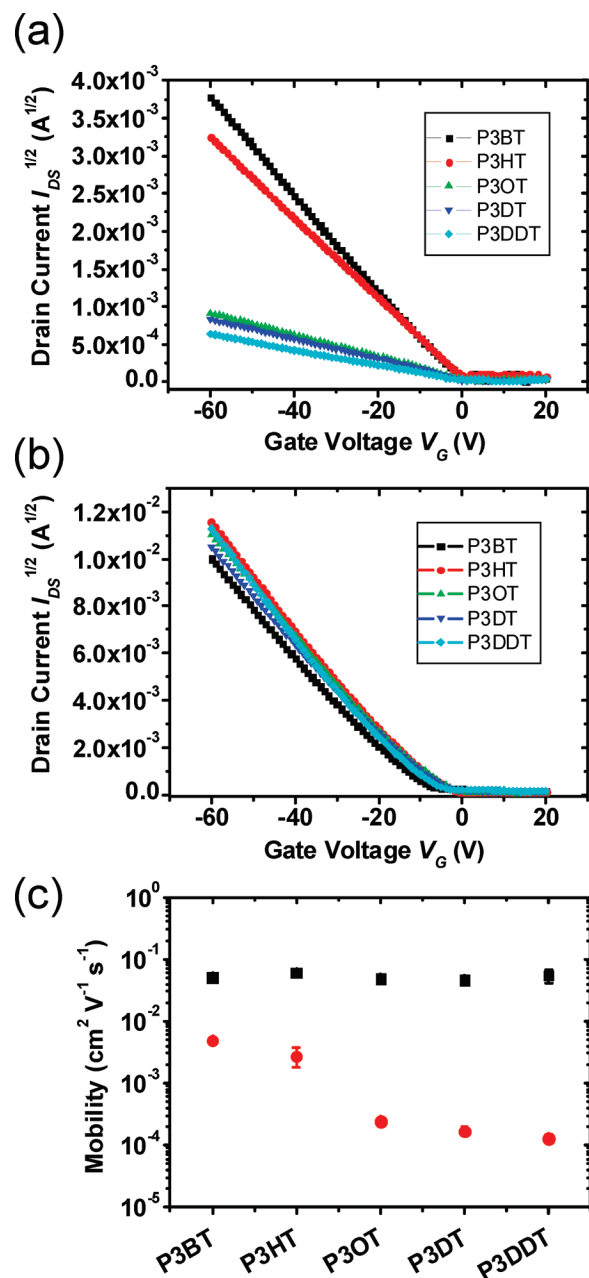


FIGURE 4. Transfer characteristics of poly(3-alkylthiophene) transistors prepared by (a) spin coating and (b) contact film transfer ($V_{\text{DS}} = -60 \text{ V}$). (c) Plots of average mobility for different polymers. Devices were prepared by spin coating (red circles) and contact film transfer (black squares).

structure at the interface forming under kinetic control. Moreover, the flexibility of the long side chains likely hinders well-ordered packing. Since the insulating side chains interfere with the interchain hopping of the charge carriers, the mobility values are expected to drop as the length of the side chains increases. However, all the devices fabricated by contact film transfer have similar threshold voltages and slopes of the transfer curves (Figure 4b; for output curves, see the Supporting Information). As summarized in Table 1, the TFTs prepared by contact film transfer have high mobility values in a narrow range of $0.05\text{--}0.07 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$. This result is in striking contrast with those for the spin-coated devices, as shown in Figure 4c. The independence

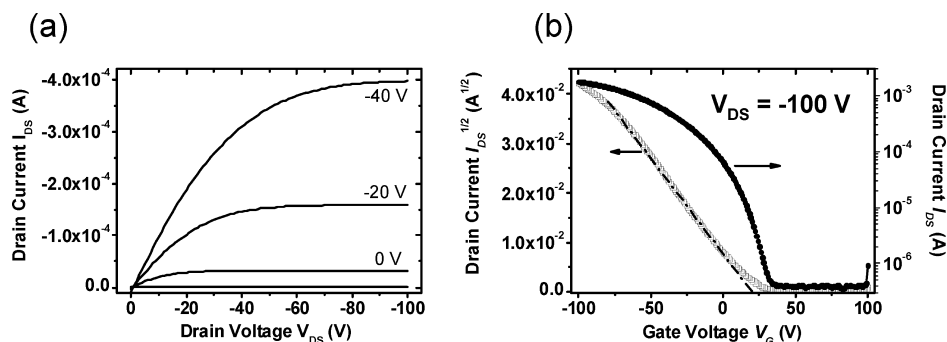


FIGURE 5. OTFT performance of RR-P3HT devices. (a) Output curves and (b) transfer curves of transferred P3HT films on OTS-treated SiO₂.

of the mobilities from the side-chain length suggests that the insulating side chains do not interfere with the charge hopping process in the lateral direction, which supports the hypothesis postulating the edge-on orientation of the thiophene rings in the transferred films. This structure at the surface agrees well with the recent analytical work by Hao et al. (19). They concluded that slow evaporation of the solvent allows sufficient time for the polymer chains at the surface to adopt a nearly equilibrium structure with good edge-on orientation.

It is well-known that the surface treatment of dielectric layer significantly affects the TFT device performance because of the change in the number of trap states and the surface roughness. As a standard surface modification method in TFT fabrication, octadecyltrichlorosilane (OTS) treated SiO₂ gives reproducible results for many organic semiconductors (14). We also successfully prepared RR-P3HT TFT devices by the contact film transfer methods (note that because chlorobenzene does not wet the surface of OTS-treated SiO₂, the solution was completely repelled from the surface during the spin-coating and the reference FET device could not be prepared by spin coating). Figure 5 shows the output and transfer characterizations ($V_{DS} = -100$ V) of the best TFT device prepared by transferring the RR-P3HT film on OTS-treated SiO₂, the mobility calculated in the saturation region is $0.19 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ with an on/off ratio of 5×10^3 and a threshold voltage of +18 V. The improved device performance could be attributed to the flatter surface of OTS-treated SiO₂ compared to BCB/SiO₂. The mobility value of RR-P3HT transistors achieved in this work is comparable with the best devices reported in the literature (9, 33).

To explore the generality of the contact film transfer, we fabricated TFTs based on three other kinds of semiconducting polymers, P3HNT, PQT-12, and PDA2T. As listed in Table 1 (for transfer and output curves, see the Supporting Information), all the transferred films have higher carrier mobility than the spin-coated films when used in TFTs. Thermal annealing before film transfer was carried out for transistors based on RR-P3HT, PQT-12, and PDA2T (PSS was used as the sacrificial layer in these experiments). No significant difference was observed in RR-P3HT TFTs after thermal annealing, but improved performances were achieved in PQT-12 ($0.09 \text{ cm}^2/(\text{V s})$) and PDA2T ($0.08 \text{ cm}^2/(\text{V s})$). This result suggests that contact film transfer is potentially applicable to a variety of solution-processable polymers and could provide a simple and general approach for the con-

struction of high-performance transistors utilizing polymer/air interfaces.

CONCLUSIONS

In summary, we have demonstrated the preparation of high-performance polymer TFTs by a novel contact film transfer process. The TFTs prepared by this transfer process have higher mobility values than those of the conventional spin-coated devices for all the semiconducting polymers studied. This suggests that at the polymer/air interface, the molecular orientation is higher and the interchain π - π interactions are more extensive than those at the polymer/dielectric interface. This simple approach potentially provides a facile and general method for evaluating the electronic properties of semiconducting organic materials. Furthermore, this method can be easily applied to the preparation of multilayered structures free from the constraints of conventional solution processes. The preparation of more complex polymer electronic devices by contact film transfer is now in progress (34).

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Supporting Information Available: Synthesis of the polymers and output characteristics of the transistors prepared by the contact film transfer (PDF). Movie clip (MPG). This material is available free of charge via the Internet at <http://pubs.acs.org>.

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