Mechanistic Analysis of Embedded Copper Oxide in Organic Thin-Film Transistors with Controllable Threshold Voltage

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ABSTRACT: The modulation of threshold voltage ($V_{TH}$) of organic thin-film transistors (OTFTs) was investigated by embedding a thin CuO layer between the two semiconductor layers. The results showed that the $V_{TH}$ of OTFTs with a CuO layer can be effectively tuned by controlling the positive gate-to-source voltage ($V_{GSO}$) under stress of gate-to-source voltages. The $V_{TH}$ shifts from $-3.67$ to $-0.82$ V when the positive $V_{GSO}$ varies from 30 to 50 V. This can be explained by the mechanism of trapping electrons at the interface between the CuO charge-separation layer and the active layer. To confirm the role of the CuO layer acting as the charge-separation source, two organic thin-film diodes, indium-tin oxide (ITO)/tris (8-quinolinolato) aluminum(III) (Alq3)/pentacene/Al (inverted-stack diode) and ITO/Alq3/CuO/pentacene/Al (inverted-stack diode with a CuO layer), were fabricated and their diode current characteristics were measured. For the second device, a large current flow was observed at positive bias on the ITO electrodes, which is ascribed to the internal bipolar charge separation within the added CuO zone.

1. INTRODUCTION

Organic thin-film transistors (OTFTs) have been widely studied over the recent decades due to their low cost, easy processability, flexibility, and applicability in large-area devices. Although sufficient mobility in OTFTs has been achieved for various applications, many factors still restrict the practical applications of OTFTs, such as how to control the threshold voltage ($V_{TH}$) and surface modifying technology. Generally, $V_{TH}$ control techniques are highly important in designing OTFTs with optimal properties and low $V_{TH}$ values. Surface modifying technology and new gate dielectric materials were used for achieving the enhancement of the electrical properties of OTFTs. Recently, high work function (WF) noble metals were applied as gate electrodes to control the $V_{TH}$ of OTFTs. In addition, high WF transition metal oxide (TMO) layers, such as WO3 or MoO3, were also employed as conversing Al gate electrodes to control the $V_{TH}$ for acquiring the low $V_{TH}$ of OTFTs. Copper oxide (CuO) was used for creating efficient hole injection in OTFTs or organic light-emitting diodes (OLEDs). Previous studies mainly focused on the gate electrode or the interface between the semiconductor and the electrodes. All these efforts were aimed at altering the charge distribution in the transistor for achieving low $V_{TH}$ values.

Here, we present an efficient way to control the trap densities in OTFT channel layers by inserting a thin CuO layer between the two semiconductor layers. It is well known that $V_{TH}$ depends on many different factors, such as trap densities in the channel layer. In this paper, a regular positive shift of threshold voltage in OTFTs can be realized by controlling the positive gate-source voltage ($V_{GSO}$) under stress of gate-to-source voltages. The positive shift of the $V_{TH}$ is very useful in reducing the magnitude of $V_{TH}$ and changing the operation mode of p-channel OTFTs. An electric filed assisted bipolar charge separation mechanism within CuO is proposed for demonstrating the previous phenomenon in this paper. To better understand the functionality of the CuO charge separation layer in the OTFT and analyze the appearance of charge production and separation within the CuO zone, we fabricated two simple inverted-stack diodes using conventional materials and tested their current–voltage characteristics. The comparison of current–voltage characteristics of the two devices clearly demonstrates the bipolar charge separation within the CuO zone.

2. EXPERIMENTAL SECTION

Figure 1a shows the schematic structure of the proposed 3 nm-thick CuO-embedded OTFT (CuO-embedded OTFT). Here, the heavily doped n-type silicon wafer with a 300 nm-thick layer and the thermally oxidized SiO2 ($C_i = 10$ nF) is used as the...
gate electrode and the insulator layer, respectively. The pentacene and CuO were vacuum thermally deposited on the insulator layer in turn under a pressure of 3 × 10^{-4} Pa. The CuO layer with a thickness of 3 nm was inserted in the middle of the pentacene layer, which possessed a total thickness of 50 nm. Then, the Cu source and drain electrodes (40 nm-thick) were thermally evaporated. The length (L) and width (W) of the channel were 100 and 10,000 μm, respectively. The electrical properties were characterized by a semiconductor parameter analyzer (Agilent 4155C) at room temperature. To confirm the charge-separation function of CuO, inverted-stack devices A and B were fabricated for comparison. Here, tris(8-quinolinolato) aluminum(III) (Alq₃) and pentacene are used as the electron-transport layer (ETL) and hole-transport layer (HTL), respectively. As shown in Figure 1b, the structure of device A is ITO/Alq₃ (100 nm)/pentacene (120 nm)/Al, whereas the structure of device B is ITO/Alq₃ (100 nm)/CuO (3 nm)/pentacene (120 nm)/Al. All of the layers in the two devices were deposited through the common thermal evaporation under a vacuum pressure of 3.0 × 10^{-5} Pa. The pentacene and CuO layers were thermally deposited on the glass substrate at rate of 0.5 and 0.3 Å s⁻¹, respectively. The 50 nm-thick CuO film was thermally deposited on the glass substrate, and the 1 nm-thick CuO film was thermally deposited on the 50 nm-thick pentacene film and sample D (glass substrate/pentacene (50 nm)/CuO (1 nm)) was obtained. The current properties of devices A and B were characterized by a semiconductor parameter analyzer (Keithley4200) at room temperature. X-ray photoelectron spectroscopy (XPS) characterization of samples C and D was done by multiphoton photoelectron spectroscopy (Kratos AXis Ultra(DLD)).

3. RESULTS AND DISCUSSION

The output and transfer characteristics of the CuO-embedded OTFT are shown in Figure 2a,b, respectively. When a negative gate voltage is applied, the current (−I_D) increases. The CuO-embedded OTFT exhibits good p-type channel field-effect properties with its mobility, V_TH, and current on/off ratio being 0.18 cm² V⁻¹ s⁻¹, -8.0 V, and 1.4 × 10⁴, respectively. However, the mobility and V_TH of the pentacene-based OFET are 0.06 cm² V⁻¹ s⁻¹ and -17.5 V, respectively (as shown in Figure 3). Compared to those of the pentacene-based OFET, the hole mobility of the CuO-embedded OFET is dramatically increased, whereas its V_TH is greatly decreased. Meanwhile, author’s previous work demonstrated that the improvement of the field-effect mobility of the CuO-embedded TFT was attributed to the charge transfer (CT) complexes based on electron transfer from pentacene to CuO. CuO acts as a p-type dopant which could accept electrons from organic materials and generate holes as demonstrated with ReO₃, Cul, and MoO₃. When a CuO film was thermally deposited on...
the surface of the pentacene film in a vacuum chamber, CuO penetrated into the surface of the pentacene film and led to the formation of CT complexes based on an electron transfer from the highest occupied molecular orbital (HOMO) energy level (5.0 eV)\textsuperscript{19} of pentacene to the conduction band energy level (4.7 eV)\textsuperscript{20,21} of CuO due to p-type doping\textsuperscript{22} (as shown in Figure 4a). X-ray photoelectron spectroscopy (XPS) was used to reveal the electron transfer at the interface between organic materials and metal oxide materials.\textsuperscript{23} We tested the X-ray photoelectron spectroscopy (XPS) on samples C (glass/CuO (50 nm)) and sample D (glass/pentacene (50 nm))/CuO (1 nm). As shown in Figure 4b, the Cu 2p\textsubscript{3/2} peak shifts to higher energies in the XPS spectra of sample D than that in the XPS spectra of sample C. Since a negatively charged atom has a higher binding energy, the increase in the binding energy results in a higher shift for the Cu 2p\textsubscript{3/2} peak.\textsuperscript{23,24}

The charge transfer (CT) complexes based on p-type doping can shift the Fermi level of pentacene toward the valence band edge of pentacene\textsuperscript{23,24} which could reduce organic material bulk hole trap density\textsuperscript{23,24} and enhance the pentacene bulk hole carrier injection. Therefore, the thin CuO layer, directly been embedded between the pentacene layer, serves as the hole-injection layer, which is responsible for the improvement of the field-effect mobility of the CuO-embedded TFT.\textsuperscript{19}

For CuO-embedded OTFTs, the gate-to-source voltage (denoted as $V_{GS0} = -50, -30, 30, 50$ V) was constantly applied for 50 s, and the electrical characteristics of the OTFTs were measured (as shown in Figures 5a and 4b) under various $V_{GS0}$ values. As shown in Figure 5 and Table I, when the applied $V_{GS0}$ is changed in the positive range, a positive shift of $V_{TH}$ of the CuO-embedded OTFT is observed. For example, when $V_{GS0}$ varies from 30 to 50 V, the $V_{TH}$ shifts from $-3.67$ to $-0.82$ V (as shown in Table I), the current $I_{on}$ (the source–drain $I_D$ at $V_{GS} = -50$ V and $V_{DS} = -40$ V) increases, and the mobility hardly changes during the shift of $V_{TH}$. The transfer characteristic curves hardly shift under negative $V_{GS0}$ stress (as shown in Figure 5a,b). This shift of the threshold voltage cannot be observed in pure pentacene-based OTFTs (as shown in Figure 6). Thus, it can be concluded that the CuO-embedded layer is very important in modulating the $V_{TH}$ of OTFTs.

As shown in Figure 5a and Table I, the positive $V_{GS0}$ stress (30 or 50 V) are constantly stressed in the CuO-embedded OTFT, the current ($I_D$) is enhanced, and the phenomenon was similar to the result demonstrated with time domain measurement that is used to determine the type of charge trapping in OTFTs.\textsuperscript{25,26} However, the situation with the pentacene-based OTFT is different (see Figure 6). The results from Figure 5 and Table I indicate that the CuO-embedded layer in the OTFT possesses the capability of trapping electrons by applying a positive $V_{GS0}$ and the trapped electrons could induce extra hole accumulation in the OTFT channel, resulting in a positive shift of $V_{TH}$ of the CuO-embedded OTFT (as shown in Figure 5b). The trapped electron population in CuO can be changed by adjusting the positive $V_{GS0}$.

As shown in Table I, the field-effect mobilities of the CuO-embedded OTFT remain unchanged with initial gate voltages ($V_{GS0}$). In this case, the trapped electrons in the CuO-embedded layer cannot be attributed to the formation of charge transfer (CT) complexes based on electron transfer near the point of contact of CuO and pentacene. The process of electron transfer near the point of contact of CuO and pentacene was complete and can be hardly changed with initial gate voltages ($V_{GS0}$). Therefore, it is necessary to illustrate the mechanism of the trapped electrons in the CuO-embedded OTFT with initial gate voltages ($V_{GS0}$).

To illustrate the phenomena in this device, a possible electric-field-assisted tunneling mechanism is shown in Figure 7. The capability of CuO p-dopants in electron accepting could result in electron transfer from pentacene to CuO and form charge transfer (CT) complexes between CuO and pentacene due to p-type doping. The mutual energy level alignment from both sides of the interface based on CuO/pentacene generates the organic–inorganic heterojunction as demonstrated with the interface of MoO\textsubscript{3} and the hole organic transporting.

\[ V_{TH} = -3.67 \text{ V} \]

\[ V_{TH} = -0.82 \text{ V} \]

\[ \Delta V_{TH} = 2.85 \text{ V} \]

\[ \Delta I_{on} = 1.67 \text{ V} \]

\[ \Delta \mu = 0.52 \text{ cm}^2/\text{V}\text{s} \]

\[ \Delta \sigma = 1.23 \text{ V} \]

\[ \Delta \tau = 2.34 \text{ V} \]

\[ \Delta \rho = -0.78 \text{ V} \]

\[ \Delta \epsilon = 1.98 \text{ V} \]

\[ \Delta \delta = 0.12 \text{ V} \]

\[ \Delta \lambda = 1.02 \text{ V} \]

\[ \Delta \mu = 0.34 \text{ V} \]

\[ \Delta \sigma = 1.56 \text{ V} \]

\[ \Delta \tau = 2.89 \text{ V} \]

\[ \Delta \rho = -0.35 \text{ V} \]

\[ \Delta \epsilon = 2.07 \text{ V} \]

\[ \Delta \delta = 1.01 \text{ V} \]

\[ \Delta \lambda = 1.23 \text{ V} \]

\[ \Delta \mu = 0.78 \text{ V} \]

\[ \Delta \sigma = 1.89 \text{ V} \]

\[ \Delta \tau = 3.02 \text{ V} \]

\[ \Delta \rho = -0.56 \text{ V} \]

\[ \Delta \epsilon = 2.17 \text{ V} \]

\[ \Delta \delta = 1.23 \text{ V} \]

\[ \Delta \lambda = 1.45 \text{ V} \]

\[ \Delta \mu = 0.98 \text{ V} \]

\[ \Delta \sigma = 2.08 \text{ V} \]

\[ \Delta \tau = 3.23 \text{ V} \]

\[ \Delta \rho = -0.78 \text{ V} \]

\[ \Delta \epsilon = 2.17 \text{ V} \]

\[ \Delta \delta = 1.45 \text{ V} \]

\[ \Delta \lambda = 1.67 \text{ V} \]
tion layer (CSL), and the separated holes can be injected into the neighboring pentacene HTL and finally flow out to the external electrodes. Electrons are trapped at the interface between the pentacene and CuO layers due to the poor electron-transporting capability of pentacene in the CuO-embedded OTFT and the presence of the injected barrier, and thus the trapped electrons induce free holes in pentacene and finally lead to an increase in the source–drain current ($I_D$) (as shown in Figure 5b and Table 1). The larger the positive $V_{GSO}$ value is used, the more the hole–electron pairs are generated and separated in the CuO zone, leading to an increased population of trapped electrons. Therefore, the trapped electron population in CuO can be controlled by adjusting the positive $V_{GSO}$. When the CuO-embedded OTFT was applied with negative $V_{GSO}$ stress, the pentacene and the thin CuO layer are both hole-transport materials in this case and the embedded CuO layer does not affect the charge injection and transport in the device. Thus, no efficient hole–electron pairs generate and separate in the CuO zone.

The shift of $V_{TH}$ is related to the difference of trapped charges of OTFTs. If the trapped electrons can be controlled, the $V_{TH}$ of OTFTs can also be strictly tuned. So, the $V_{TH}$ of CuO-embedded OTFT depends on the trapped electron population in CuO, which is also changeable with the positive $V_{GSO}$.

Figure 8 shows the voltage–current density characteristics of the two diodes. In device A, in view of the stacking order of pentacene and Alq$_3$, essentially very low current flow is observed in forward bias (ITO positive) (as shown in Figure 8), which is derived from the poor electron-transporting capability of pentacene and the low hole mobility of Alq$_3$. On the other hand, essentially low current flow is observed in reverse bias (ITO negative) in device B (as shown in Figure 8), which is attributed to the presence of a large injected barrier in the pentacene HTL/Al electrode and Alq$_3$ ETL/ITO electrode. For device B, large current flow is observed in

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**Table I. Electrical Parameters of the CuO-Embedded OTFT under Different $V_{GSO}$ Stress**

| $V_{GSO}$ (V) | $V_{TH}$ (V) | mobility (cm$^2$ V$^{-1}$ s$^{-1}$) | $I_{on}$ (A) | current on/off ratio |
|---------------|---------------|-----------------|-------------|-----------------|
| −50           | −8.0          | 0.181           | $1.35 \times 10^{-4}$ | $10^{-3}$       |
| −30           | −8.0          | 0.182           | $1.37 \times 10^{-4}$ | $10^{-3}$       |
| 30            | −3.67         | 0.179           | $1.56 \times 10^{-4}$ | $10^{-3}$       |
| 50            | −0.82         | 0.179           | $1.95 \times 10^{-4}$ | $10^{-3}$       |

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**Figure 5.** (a) $-I_D$ versus $V_{GS}$ and (b) $(-I_D)^{1/2}$ versus $V_{GS}$ for the CuO-embedded OTFT under different $V_{GSO}$ stress: −50, −30, 30, and 50 V.

**Figure 6.** $(-I_D)^{1/2}$ versus $V_{GS}$ for the pentacene-based OTFT with different $V_{GSO}$: 60, 40, 20, and 0 V.

**Figure 7.** Energy level of the heterojunction based on the CuO charge-separation layer (CSL)/pentacene in the proposed electric-field-assisted tunneling model, where $\Phi_i$ is the trap level with respect to the CuO valence band maximum and $\Phi_{fi}$ is the tunneling barrier. Holes and electrons are dissociated upon application of a reverse external electric field (under application of a positive $V_{GSO}$); holes can be injected into the neighboring pentacene and electrons are trapped at the interface between pentacene and CuO. "●" and "○" symbols represent electrons and holes, respectively.

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With a positive $V_{GSO}$ stress being applied to the CuO-embedded OTFT, the pentacene and CuO materials exhibit large bulk resistivity in this case. Therefore, the CuO layer could act as the charge-separation source and generate holes and electrons similar to the MoO$_3$ charge-generation layer. A high external electric field toward the Cu electrodes is dropped in the CuO-embedded layer under positive $V_{GSO}$. As shown in Figure 7, holes and electrons are dissociated upon electric-field-assisted Fowler–Nordheim tunneling in the CuO charge-separation layer (CSL), and the separated holes can be injected into the Cu electrodes in the CuO-electrode layer. Holes and electrons are dissociated upon electric-field-assisted Fowler–Nordheim tunneling in the CuO charge-separation layer (CSL), and the separated holes can be injected into the Cu electrodes in the CuO-electrode layer.
forward bias (as shown in Figure 8). We can notice that the behavior of the current−voltage characteristic of device B is quite similar to that of the conventional double-layer diodes. The only difference between devices A and B is the additional CuO layer inserted in device B. The insertion of extra layers between existing organic layers usually brings about a large decrease of current due to the increase of injection barriers and bulk resistivity. Therefore, the origin of the considerable current enhancement was not ascribed to the increase of the injected current. In device A, when an ITO is biased positively, the inserted layer of CuO acts as the internal bipolar charge−generation source. The holes and electrons are dissociated under the electric field toward the Al electrodes supplied from positive bias (as shown in Figure 7). The dissociated holes can be injected into the adjacent pentacene HTL and electrons can be injected into the adjacent Alq3 ETL. The holes and electrons are transported through organic layers and finally flow out to the external electrodes, resulting in a large current as shown in Figure 8. When an ITO is negative biased, there is no efficient charge generation from the CuO additional layers because of the presence of low mobility and an injected barrier. Thus, it is clear that the origin of large current flow in device B is ascribed to the behavior of charge separation of the extra inserted CuO layer. Thus, the result clearly revealed the bipolar charge separation proposed within the CuO zone.

4. CONCLUSIONS

Regular control of the threshold voltages of CuO-embedded OTFTs is successfully achieved by applying different positive gate-to-source voltage (V_GS) stress. The population of trapped electrons between the CuO layer and the active layer can be changed with positive V_GS. The mobility hardly changes during the shift of V_TH. To illustrate this phenomenon, an electric-field assisted tunneling mechanism within the CuO charge separation zone is proposed. On the basis of the comparison of current−voltage characteristics of two simple inverted-stack diodes, it clearly reveals the presence of bipolar charge separation within the CuO zone. This research could be helpful in fabricating OTFTs with controllable V_TH and offer useful guidelines for the development of other types of organic thin-film devices, such as light-emitting diodes, memory devices, etc.

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Notes
The authors declare no competing financial interest.

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