A 6,13-bis(Triisopropylsilylethynyl) Pentacene Thin-Film Transistor Using a Spun-On Inorganic Gate-Dielectric

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Abstract—We present the latest results of the use of soluble materials such as organic semiconductors (OSCs) or gate-dielectrics for simplified processing of organic thin-film transistors (OTFTs). In this paper, we described our fabrication of a solution-processed OTFT with 6,13-bis(Triisopropylsilylethynyl) pentacene (TIPS-pentacene) as the OSC and siloxane-based spin-on glass (SOG) as the inorganic gate-dielectric. Also, synthesized TIPS-pentacene and SOG were examined for use as the OSC and gate-dielectric in an OTFT. From electrical measurements, we obtained device performance characteristics such as charge carrier mobility, threshold voltage, current ON/OFF ratio, and subthreshold swing, which were 6.48×10^{-3} cm²/V · s, -13 V, ~ 100 , and 1.83 V/dec, respectively.

Index Terms—6,13-bis(Triisopropylsilylethynyl) pentacene (TIPS-pentacene), organic electronics, organic thin-film transistor (OTFT), spin-on glass.

I. INTRODUCTION

The PERFORMANCE of organic thin-film transistors (OTFTs) has improved remarkably during the last decade. Thus, it is at least the same as that of a hydrogenated amorphous silicon (α -Si:H) thin-film transistor (TFT), which is currently used for the manufacturing of TFT-liquid crystal displays. However, its severe insolubility renders it useless for solution-based fabrication of electronic devices. Solution-based processing is the key to enabling ultralow-cost circuit fabrication. This processing eliminates the need for lithography, subtractive processing, and vacuum-based film deposition. Recently, there has been great interest in the development of printed organic electronics technologies since these technologies allow the use of spin coating, drop casting, ink jet printing, spray coating, nanoimprinting, or the screen printing process [1]–[6]. These technologies are

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expected to be used in low-cost, flexible displays and disposable electronics applications. As a consequence, extensive research by many groups has been actively carried out to improve the charge carrier mobility and the current modulation.

The morphology of the dielectric surface onto which the organic semiconductor (OSC) layer is formed is an important factor in the device performance of OTFTs. It has been reported [7], [8] that a rougher surface leads to more defects and voids that can act as traps at OSC-dielectric interface. The surface roughness also affects the ordering of OSC molecules during an active-layer formation [9], [10]. Therefore, OTFT with low surface roughness film exhibits good electric performance.

In this paper, we utilize a very smooth surface roughness film, spin-coatable inorganic dielectric, less sensitive to moisture, and a siloxane-based spin-on-glass (SOG) as gate dielectric for OTFT. Also, we present the characterization of synthesized 6,13-bis(Triisopropylsilylethynyl) pentacene (TIPSpentacene) as a p-type OSC. We discuss their applications in solution-process-based OTFTs. We obtained the thermal properties, optical properties, and device performances using both TIPS-pentacene and SOG as results. This paper may help us to understand the structural relationships of the materials. It may provide a useful way for the rational design of transistor materials or their applications.

II. MATERIAL AND DEVICE FABRICATION

Functionalized pentacene, TIPS-pentacene, leads to a highly soluble and oxidatively stable OSC as a hole transport channel designed to exhibit enhanced π -stacking interactions. It is easily prepared in near quantitative yield in a one-pot reaction from 6,13-pentacenequinone [7]. TIPS-pentacene [see Fig. 1(a)], synthesized as described elsewhere [11], [12], was synthesized to address precisely these limitations. On one hand, the TIPS side groups make TIPS-pentacene soluble in common organic solvents; on the other hand, these bulky groups also help to disrupt the face-to-edge herringbone packing pattern and to form a regular columnar stacking between the acene planes [11]–[13]. For a solution processable gate dielectric, we utilized a siloxanebased SOG (Honeywell 512B, dielectric constant = 3.1 ± 0.1) that was composed primarily of siloxane that contained CH₃ (15% organic content) groups bonded to Si atoms in the Si-O backbone, as illustrated in Fig. 1(c). Unlike polymer dielectric materials, an inorganic dielectric, SOG would not have the moisture and hysteresis problems of a polymer dielectric [14].



Fig. 1. (a) Chemical structure of 6,13-bis(Triisopropylsilylethynyl) pentacene. (b) Schematic of the inverted staggered OTFT structure on a highly doped Si wafer with a gate electrode. (c) Structure of Accuglass 512B (spin-on glass) composed mainly of siloxane.

The schematic structure of a TIPS-pentacene/SOG-based OTFT is shown in Fig. 1(b). The devices were made by using a heavily doped n-type Si wafer, with an optimized SOG [14], which functioned as the gate electrode and the gate insulator, respectively. The SOG was spin-coated on the HF-treated Si surface at a spin rate of 4000 r/min for 20 s, baked successively at 80 and 250 °C for 1 min each in air, and finally, cured at 450 °C at $1.0 \text{ L} \cdot \text{min}^{-1} \text{ N}_2$ flow. The details about the SOG/Si surface were described in the literature [15]-[17]. Gold (Au, 70 nm thickness) source/drain contacts including titanium (Ti, 30 nm thickness), which acted as an adhesion layer, were deposited by electrobeam evaporation. Also, a shadow mask was used to define the source and drain electrodes. The channel width (W)and length (L) were 500 and 20 μ m, respectively. Next, the TIPS-pentacene film was deposited by a drop casting. Finally, the coated TIPS-pentacene-based device was annealed using an oven at 110 °C for 30 min. After the device fabrication, the transistor characteristics were measured using Keithley SCS/4200 in ambient air.

III. RESULT AND DISCUSSION

The thermal properties of TIPS-pentacene were investigated using thermogravimetric analysis (TGA) (TA Instrument 2050)



Fig. 2. (a) TGA plots with a heating rate of $10 \,^{\circ}$ C /min under nitrogen. The inset shows the DSC traces with a heating (cooling) rate of $10 \,(-10) \,^{\circ}$ C/min under a nitrogen flow. (b) Optical absorption (UV-Vis) spectra of TIPS-pentacene in chloroform.

and differential scanning calorimetry (DSC) (Mettler Toledo DSC821e analyzer), as shown in Fig. 2(a). These techniques were used to characterize the thermal transitions in polymers. TGA is a simple analytical technique that measures the weight retention of a material as a function of temperature. The TGA data for TIPS-pentacene revealed inflections at 388 °C that suggested that a decomposition process occurred. In addition, a significant decomposition-related residue was observed at \sim 498 °C, as shown in Fig. 2(a). By substituting the bulky groups at the 6 and 13 positions of pentacene, edge-to-face interactions are inhibited; it forms molecular crystals with a slip-stacked structure, which resulted in a decomposition process at high temperature. When a high-temperature DCS was run from 50 to 250 °C, two peaks were observed, as shown in the inset of Fig. 2(a). The endothermic peak (heating line) at 123 °C corresponded to the melting of the compound. The exothermic peak (cooling line) at 118 °C indicated a structural phase transition. Fig. 2(b) depicts the optical absorption [ultraviolet-visible (UV-Vis) spectrometry, HP 8453, PDA, type $\lambda = 190-1100$ nm] spectra of the solution of TIPS-pentacene in chloroform. The



Fig. 3. (a) $10 \times 10 \ \mu m^2$ SPM image of the TIPS-pentacene film on SOG. (b) FE-SEM micrograph (with an apparent viewing angle of 12°) of the same TIPS-pentacene film presented in Fig. 1(a). (c) EDX of a siloxane-based spin-on glass film showing a stoichiometric elemental percentage analysis.

absorption maximum peak was at 643 nm with a few absorption peaks between 400 and 600 nm that matched the absorption spectrum reported for TIPS-pentacene in the tetrahydrofuran solution [18]. The functionalization of TIPS-pentacene with electron-rich substituents resulted in oxidatively stable and highly soluble organic active materials that can be engineered to preserve the close π -stacking and increased orbital overlap [19].

Fig. 3(b) shows a cross-sectional field-emission scanning electron microscope (FE-SEM) image presented in Fig. 1(c), which is the image of the TIPS-pentacene layer on the precoated SOG layer on the n^{++} Si substrate. The thicknesses of the TIPS-pentacene and SOG layers were about 800 and 750 nm, respectively. Fig. 3(a) shows the scanning probe microscopy (SPM, XE-100 system) image of TIPS-pentacene coated onto the SOG/Si substrate. The bright area in the image was interpreted as increasing the film thickness in that direction. Fig. 3(a) is a topographic image of the OCS film formed by drop casting from a 2 wt% solution of TIPS-pentacene in toluene. The image demonstrated continuous films, but also showed what appeared to be molecular steps with the rms roughness of 9.835 nm

at 110 °C. The elemental energy dispersive X-ray (EDX, Horiba EX-200) analysis [Fig. 3(c)] was carried out only for siloxanebased SOG. The average atomic percentage ratio of O:Si was 67.97:32.03 [inset table of Fig. 3(c)] showing that the ratio of O to Si in the silicon-oxide formed by the room-temperature reaction was 2.12. This was about the same as that of thermally grown SiO₂ (O/Si ratio of standard SiO₂: 2) [20]. Also, it was known [21] that a low surface roughness was necessary for high mobility. The very smooth surface (rms roughness =0.508 nm while rms roughness of thermally grown $SiO_2 =$ 1.08 nm, not shown here) that the SOG provided must have contributed to the relatively high mobility obtained. It can be seen that a clear interface exists between the gate-dielectric and the OSC film, as shown in Fig. 3(b). If the dielectric film has poor surface roughness, then this roughness leads to valleys in the channel region. These valleys may act as carrier traps with a number of scatterings [22]. To further investigate the device performance, surface properties of dielectric layers were characterized by advancing the water-contact angle measurement (not shown here). SOG was rather hydrophobic surface with a water-contact angle of $\sim 86^{\circ}$ while thermal grown SiO₂, relatively hydrophilic surface with a water contact angle of $\sim 42^{\circ}$ was measured. From these results, it is believed that the relatively large water-contact angle of SOG with nonploar character is attributable to higher density of methyl (CH₃) groups and a near absence of hydroxyl (OH) groups on the surface. Therefore, TIPS-pentacene/SOG-based OTFT has higher performance than that of TIPS-pentacene/SiO₂-based OTFT because of relatively less hydroxyl bindings in SOG dielectric surface related to nearly no hysteresis of device [23]. Fig. 4 shows the electrical characteristics of drop-casted TIPS-pentacene on thermally grown SiO_2/Si wafer. From the electrical output [Fig. 4(a)] and transfer [Fig. 4(b)] curve, TIPS-pentacene/SiO₂-based OTFT had an extracted mobility of up to $4.91 \times 10^{-5} \text{ cm}^2/\text{V} \cdot \text{s}$, threshold voltage of 11 V, current ON/OFF ratio of \sim 100, and subthreshold swing of 3.07 V/dec. All electrical measurements were performed in air ambient at room temperature and in ambient light condition.

The output and transfer characteristics are shown in Fig. 5 for a TIPS-pentacene thin-film transistor with siloxane-based SOG as an inorganic gate dielectric. The high work function of Au was expected to improve the injection of holes into the OSCs. The Au contacts sometimes produced low mobilities ($<10^{-4} \text{ cm}^2/\text{V} \cdot \text{s}$) which improved to about $6.48 \times 10^{-3} \text{ cm}^2/\text{V} \cdot \text{s}$ following annealing at a temperature of 110 °C. Fig. 5(a) shows drain-current (I_D) versus drain-voltage (V_D) of the TIPS-pentacene/SOGbased OTFT as the source-drain electrodes at different gate voltage (V_G) . The curve was typical for *p*-type OTFT working in an accumulation mode. The positive current near the 0 V drain bias (V_D) , increasing with V_G , was the result of the leakage current through the gate-dielectric layer between the source-drain and gate electrodes. To improve the device contact characteristics, the treatment of the interface between the OSC and gate-dielectric layer using a self-assembled monolayer was studied by other groups [24]. The corresponding plots of I_D and $\sqrt{I_D}$ versus V_G for the device are shown in Fig. 5(b). The device had a subthreshold swing of 1.83 V per decade,



Fig. 4. Current–voltage characteristics of a TIPS-pentacene/SiO₂-based OTFT. (a) Drain current (I_D) versus the drain voltage (V_D) characteristic curve where the gate voltage (V_G) varies between 0 and -50 V in steps of -10 V. (b) Log I_D and square root of drain current $(\sqrt{I_D})$ as a function of V_G (from +10 to 50 V) at drain to source voltage (V_{DS}) . The inset shows the schematic of the inverted staggered the TIPS-pentacene/SiO₂-based OTFT structure (W/L = $500 \ \mu\text{m}/20 \ \mu\text{m}$) on a highly doped Si wafer with a gate electrode. The SiO₂, as gate-dielectric, was thermally grown to a thickness of 300 nm. The drop-casted OSC-based device was annealed using an oven at $110 \ ^{\circ}$ C for 30 min.

an extracted mobility of up to $6.48 \times 10^{-3} \text{ cm}^2/\text{V} \cdot \text{s}$, and had a threshold voltage of -13 V [from Fig. 5(b)]. However, the current ON/OFF ratio was relatively small (~ 100). The current ON/OFF ratio was limited by the OFF current $(2.9 \times 10^{-10} \text{ A})$ that was extremely low in our device. This was related to a high material purity without unintentional doping by charge carriers. The distribution of traps was considered to be an important explanation for the high current ON/OFF ratio of OTFTs assumed on amorphous molecular materials [25]. However, our device has lower performance than that of the TIPS-pentacenebased OTFT with carrier mobility >1 cm²/V \cdot s fabricated by other research groups [26], [27]. Because the carrier channel in which the electronic charges were transported in the device was formed in close proximity to the interface, the properties of the OSC/gate-dielectric interface were of critical importance. Hence, the treatment of the interface between the OSC and gatedielectric layer using dry or wet cleaning techniques and surface modification of gate-dielectric using self-assembled monolayer were studied by other groups [26]-[30] to improve the characteristics of the OTFTs.



Fig. 5. Current–voltage characteristics of a TIPS-pentacene/SOG-based OTFT. (a) I_D versus the V_D characteristic curve where the V_G varies between 0 and -50 V in steps of -10 V. The increasing deviations from zero current at zero drain-source voltage with higher gate voltages are caused by leakage through the dielectric. (b) Log I_D and $\sqrt{I_D}$ versus V_G curve that results when the gate voltage is scanned from +10 to -50 V.

IV. CONCLUSION

We have reported a solution-processed OTFT consisting of TIPS-pentacene as an OSC and SOG as a gate-dielectric. Although the device characteristics described here were not well optimized and contacts appeared to limit device performance, our work provided advantages in the areas of solubility, processability, and tunability for organic electronics applications through soluble materials. This paper has provided a possible path to low-cost and large-area organic electronics processing.

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