Grain size adjustment in organic field-effect transistors for chemical sensing performance improvement

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ABSTRACT Various nanostructures of the organic semiconductor (OSC) films have been reported to enhance the organic field-effect transistors (OFETs) sensing performance. However, complicated fabrication processes hinder their applications. In this work, we have effectively enhanced the sensitivity of the OFET-based sensors only by adjusting substrate temperature in OSC preparation and surface treatment of the dielectric layer. The relative sensitivity of the device can be enhanced by 5 times. The flexible sensors with polymer dielectric also exhibit high sensitivity because the less smooth surface of the polymer provides the OSCs with smaller grain size. Therefore, this work reveals the trade-off effects of the OSCs grain size on both transistor characteristic and chemical sensing performance, and provides a simple and extensively applicable strategy for OFETs sensitivity improvement.

Keywords: grain size, organic semiconductor, OFET, chemical sensor, performance improvement

INTRODUCTION

Chemical sensors based on organic field-effect transistors (OFETs) possess advantages including portability, flexibility and low-cost fabrication, but inherently suffer from limited sensitivity [1–7]. Analyte molecules have to diffuse through the organic semiconductor (OSC) films before they interact with the charge carriers in the conductive channel at the scale of a few molecular layers at the bottom of the OSC film near the OSC/dielectric interface [8,9]. OSC films, including micro/nano wires and micro/nano porous OSC films, can expose the conduction channel to the analyte atmosphere, and thus can improve the OFET sensing performance [10–16]. For example, we reported the porous OFETs fabricated by a porous OSC film using a polystyrene template, showed a relative sensitivity (RS) as high as 340% ppm^{−1} upon exposure to

10 parts per billion (~10 ppb) NH₃ [12]. However, fabrication processes of such nanostructures are rather complicated and often inapplicable for the large amount of the OSC variety.

Grain size of OSC films has significant effect on OFET characteristic [9,17–20]. Generally, larger grains lead to better OFET performance because the transportation of charge carriers is facilitated by less grain boundaries [21,22]. Accordingly, fabrication parameters of OSC films have been optimized to form large grains to improve the OFETs performance. For instance, the substrates are often heated in the thermal evaporation process of the OSC films, and the surfaces of the dielectric layers are normally treated with self-assembled monolayer (SAM) such as octadecyltrichlorosilane (OTS) and so on [23,24]. For the application of chemical sensors, however, the OFET characteristic is not the key factor as compared with sensitivity. The effect of the grain size on the sensitivity of the device should be investigated, which can provide novel optimized fabrication parameters for OFET-based sensors, and thus improve the chemical sensing performance [25].

In this work, we use a stable and high-mobility OSC, dinaphtho [2,3-b:2',3'-f] thieno [3,2-b] thiophene (DNNT), to fabricate a series of films with different grain sizes by adjusting the substrate temperature (T_{sub}) in thermal evaporation process. It is found that OFETs with smaller grain size fabricated at lower T_{sub} exhibited lower drain source current (I_{DS}), but much better chemical sensing performance. The surfaces of the silica dielectrics were also treated with or without OTS SAM, resulting in OFETs with large and small grains, respectively. As the same with aforementioned, larger grains lead to lower sensitivity, which confirms the effect of the grain size. Smaller grain gives more interval space in the OSC films

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for the analyte molecules diffusion, and thus provides the devices with higher sensitivity. By adjusting the common fabrication parameters, the RS of the device can be enhanced by 5 times. Polymer dielectric and substrate is applied to fabricate flexible device, and the DNTT film deposited on the polymer membrane presents decent grain size, leading to the flexible sensor with good sensitivity. Therefore, this work provides a simple and extensively applicable strategy for enhancing sensitivity of OFETs.

EXPERIMENTAL SECTION

Materials preparation

The OSC DNTT and OTS were purchased from Sigma-Aldrich, and DNTT was further purified by vacuum sublimation. Polylactide (PLA) was purchased from Natureworks Company and then recrystallized by ethyl acetate.

Devices fabrication

Heavily n-type doped Si wafers with 300 nm of SiO₂ layers were used as substrates. They were first cleaned by sonication in acetone and isopropanol alternatively for 1 h, and then rinsed with deionized water and ethanol, dried by nitrogen flow. Only for OTS-OFETs, the wafers were immersed in OTS toluene solution (1.2 vol.%) at room temperature for 3 h, followed by washed with toluene and ethanol. Then, 60 nm DNTT film was deposited at the rate of 0.3 Å s⁻¹ by vacuum thermal evaporation under $T_{\text{sub}} = 25, 60, 80, 100^{\circ}\text{C}$, respectively ($P \sim 5 \times 10^{-4}$ Pa). 80 nm gold source-drain electrodes were thermally evaporated through a shadow mask to form top-contact OFET devices. For flexible OFETs,

(tridecafluoro-1,1,2,2-tetrahydrooctyl) trichlorosilane (FOTS) chloroform solution (1 vol.%) was used to modify the Si wafers by spin coating at 3,000 rpm for 20 s, followed by sonication cleaning in chloroform for 30 min. With the FOTS release layer, the devices could be peeled off from the wafers easily after the fabrication process was completed. 80 nm gold gate electrodes were thermally evaporated onto the FOTS-treated wafers. PLA dielectric films were made by dip-coating 50 g L⁻¹ PLA chloroform solution at a speed of 20 μm s⁻¹ (Dip Coater, Shanghai SANYAN SYDC-100H), and then dried at 60°C in air overnight. The average thickness of PLA dielectric is around 3 μm. After that, DNTT and gold source-drain electrodes were deposited as aforementioned.

Device characterization

The surface morphologies of the DNTT films were observed using atomic force microscope (AFM, SEIKO SPA-300HV) operated in tapping mode. All electrical characterizations of the OFETs were measured by using a Keithley 4200-SCS semiconductor characterization system (Keithley Instrument) connected to a homemade vapor testing chamber. Flexible performance of the OFET-based sensors was measured while the devices were tightly bent around cylinders with radius at 5 mm.

RESULTS AND DISCUSSION

Fig. 1 presents the molecular structure of the OSC DNTT and the OFET with a bottom gate top contact configuration. Heavily doped silicon wafers with 300 nm SiO₂ dielectric layers were used as substrates. A part of the wafers were covered by OTS SAM. DNTT were then thermally evaporated on the substrates under different T_{sub} ranging from 25 to 100°C, followed by evaporation of

Figure 1 Schematic illustration of the OFETs with different OSC grain sizes for chemical sensing: (a) molecular structure of the OSC DNTT, (b) OFET with rather small grain size of OSC film, (c) OFET with rather big grain size of OSC film, 3D AFM topography images of the DNTT films with (d) rather small grain size and (e) rather big grain size.

Figure 2 Evaporation of DNTT films with different grain sizes by controlling T_{sub} and the transistor performances of the OFETs with different grain sizes: AFM topography images of DNTT films evaporated under T_{sub} of (a) 25°C, (b) 60°C, (c) 80°C and (d) 100°C, (e) $I_{\text{DS}}-V_{\text{DS}}$ curves of the OFET with DNTT film evaporated under T_{sub} at 25°C, and (f) $I_{\text{DS}}-V_{\text{GS}}$ curves of OFETs with DNTT films evaporated under different T_{sub} .

gold drain/source electrodes. The different fabrication parameters provide the OFETs with different grain sizes, as shown in the AFM images, which possess significant influence on the OFETs sensing performance. In the chemical sensing application, gas analytes firstly diffuse through the OSC films before interacting with the OFETs charge carriers [9].

The morphologies of the DNTT films deposited on silicon wafer evaporated under different T_{sub} were observed using AFM. Fig. 2 shows AFM topography images of the series of DNTT films, and the corresponding phase

images are shown in Fig. S1. Apparently, the DNTT film fabricated under T_{sub} of 25°C presents rather small grains and the crowded grains connected with each other to form a continuous film (Fig. 2a). Higher T_{sub} leads to larger and less crowded grains for the DNTT films. When the T_{sub} reaches up to 100°C, the grains are very large, but the DNTT film is not continuous (Fig. 2d). As shown in Fig. 2e, the OFET fabricated under T_{sub} of 25°C displays obvious linear and saturation regimes in the output curves (drain-source current *versus* drain-source voltage, $I_{\text{DS}}-V_{\text{DS}}$). The transfer curve (drain-source current *versus*

Figure 3 Sensing performances of the OFETs with different grain sizes of DNTT films upon NH_3 analytes: $I_{\text{DS}}-V_{\text{GS}}$ curves upon NH_3 analytes with different concentrations of the OFETs with DNTT film evaporated under T_{sub} at (a) 25°C , (b) 60°C and (c) 80°C , (d) I_{DS} , (e) μ and (f) V_{th} changes of the different OFETs exposed to NH_3 analytes as compared to nitrogen background. The error bars in d, e and f represent standard errors from 5 different measurements.

gate-source voltage, $I_{\text{DS}}-V_{\text{GS}}$) difference for the OFETs fabricated under different T_{sub} (Fig. 2f) demonstrates that higher T_{sub} leads to higher I_{DS} and mobility (μ) for the device, for instance, μ for OFETs fabricated under T_{sub} at 25, 60 and 80°C are 0.01, 0.27 and $0.56\text{ cm}^2\text{ V}^{-1}\text{ s}^{-1}$, respectively. OFETs with these DNTT films exhibit well transistor characteristics, which is consistent with previous report [18]. The on-off ratios of the OFETs are around 1,000, relatively low, because the SiO_2 dielectrics of the OFETs do not undergo surface treatment. The device fabricated with T_{sub} of 100°C does not work due to the discontinuous DNTT film.

Chemical sensing performances of the OFETs were evaluated using ammonia (NH_3) as the analytes. NH_3 detection is promising and important in industrial security, environment monitoring and biomedical applications, and the NH_3 analytes would induce the charge trapping and quenching effects to the charge carriers conducting in the DNTT [12]. The OFETs were measured in a homemade test chamber under nitrogen (N_2) atmosphere and then NH_3 vapors were injected at different

concentrations of 1 and 5 ppm (v/v). Fig. 3a–c show the transfer characteristic curves of the series of OFETs upon exposure to NH_3 at different concentrations. The I_{DS} of all the OFETs decreased after the devices were exposed to NH_3 vapor. The OFET fabricated under lower T_{sub} exhibited more significant response than that evaporated under higher T_{sub} at all NH_3 concentrations. It should be noted that the sensing performances of the OFETs could also be affected by their different charge mobilities. Therefore, normalized currents and mobilities were calculated to evaluate the influence of the grain size on the sensing performances. Fig. 3d–f present the changes of I_{DS} ($V_{\text{DS}}=-40\text{ V}$ and $V_{\text{GS}}=-40\text{ V}$), μ and threshold voltage (V_{th}) for the different OFETs exposed to NH_3 vapor as compared to N_2 background. In all the figures, OFET fabricated under the lowest T_{sub} shows the highest parameter changes. If we define RS as the percentage of I_{DS} change per ppm analyte concentration, the RS of the OFET fabricated under 80°C exposed to 1 ppm NH_3 vapor is $4.5\%\text{ ppm}^{-1}$, while that of the device evaporated under 25°C is $22\%\text{ ppm}^{-1}$ (Fig. 3d). By adjusting T_{sub} , the

Table 1 Summarized I_{DS} , μ and RS of the different OFETs exposed to NH_3 vapor in N_2 background with various concentrations

	$T_{sub} = 25^\circ C$			$T_{sub} = 60^\circ C$			$T_{sub} = 80^\circ C$		
	0	1 ppm	5 ppm	0	1 ppm	5 ppm	0	1 ppm	5 ppm
I_{DS} (μA)	1.94	1.52	1.11	39.7	34.9	27.1	112	107	94
μ ($cm^2 V^{-1} s^{-1}$)	0.0097	0.0079	0.0069	0.274	0.264	0.242	0.564	0.55	0.517
RS ($\% ppm^{-1}$)	-	22	8.6	-	12	8.6	-	4.5	3.2

RS of the OFET to NH_3 vapor can be enhanced up to 5 times. Detailed I_{DS} , μ and RS of the different OFETs exposed to NH_3 with different concentrations were summarized in Table 1. It should be noted that the T_{sub} almost have no effect on the crystalline feature of the DNTT films according to the previous literature [26]. Therefore, the OFET fabricated under lower T_{sub} possesses smaller grain size, and thus gives more interval space in the DNTT films for the NH_3 molecules to diffuse through, resulting in higher sensitivity. Moreover, the error bars in Fig. 3d–f are with limited length, indicating high stability of the OFET-based sensors.

The OFETs studied in this work were fabricated with the silica dielectric coated by OTS SAM. Fig. 4a and b show AFM topography images of the DNTT films deposited on silica dielectric with and without OTS surface treatment, and the corresponding phase images are shown in Fig. S2a and b (T_{sub} at $25^\circ C$). The OTS SAM gives the dielectric a high-quality and smooth surface, which is beneficial for DNTT crystallization [27]. The

grain size of the DNTT film deposited on OTS SAM is larger than that on silica dielectric directly (Fig. 4a and b). As shown in Fig. 4c, the I_{DS} of the OFET with OTS SAM (OTS-OFET) is larger than that of the device without OTS (Fig. 3a), which is consistent with the aforementioned situation. Similar results were also found when T_{sub} was at $80^\circ C$. The grains of the DNTT film deposited on OTS SAM are very large, but the DNTT film is discontinuous, making the corresponding OTS-OFET failed (Fig. S2c–f). The chemical sensing performance of the OTS-OFETs fabricated under T_{sub} at $25^\circ C$ was further evaluated and compared with that of OFETs without OTS. Fig. 4d–f show the changes of I_{DS} ($V_{DS} = -40$ V and $V_{GS} = -40$ V), μ and V_{th} for the different OFETs exposed to NH_3 vapor as compared to N_2 background. Although the OTS-OFETs exhibited high response to NH_3 vapor, the RS of the devices were obviously lower than that of OFETs without OTS. These results confirm that the effect on OFET sensitivity is from the grain size of the OSC film.

Figure 4 Effect of OTS treatment on the grain sizes of the DNTT films and the OFETs sensing performances: AFM topography images of DNTT films evaporated on silicon wafer (a) without and (b) with OTS treatment (T_{sub} at $25^\circ C$), (c) I_{DS} - V_{GS} curves of the OTS treated OFET upon NH_3 analytes with different concentrations, (d) I_{DS} , (e) μ and (f) V_{th} changes of the OFETs with and without OTS treatment exposed to NH_3 analytes as compared to nitrogen background. The error bars in d, e and f represent standard errors from 5 different measurements.

Figure 5 Real-time detection of the OFETs to NH_3 analytes: (a) normalized I_{DS} variation of the OFETs with DNTT films evaporated at different T_{sub} upon nitrogen and NH_3 analytes at different concentrations, and (b) normalized I_{DS} variation of the OFETs with and without OTS treatment upon nitrogen and NH_3 analytes at different concentrations ($V_{DS}=-40$ V and $V_{GS}=-40$ V).

To further demonstrate and compare the chemical sensing performance of the OFETs, the I_{DS} changes of the devices at fixed V_{DS} and V_{GS} were monitored along with time during the NH_3 vapor sensing processes ($V_{DS}=-40$ V and $V_{GS}=-40$ V). As shown in Fig. 5a, the OFET with T_{sub}

at 25°C exhibits 20% I_{DS} decrease rapidly right after 1 ppm NH_3 was injected, followed by a rapid 42% I_{DS} decrease for 5 ppm NH_3 vapor. For OFET with T_{sub} at 80°C, the I_{DS} decrease for 1 and 5 ppm NH_3 were 5% and 12%, respectively. Higher T_{sub} (larger grain size) leads to lower I_{DS} decrease. Similarly, OTS-OFETs with larger grain size showed lower I_{DS} decrease too, as compared with the OFETs without OTS SAM (Fig. 5b). On the other hand, smaller grain size also leads to faster response speed. When 1 ppm NH_3 was injected, the OTS-OFET exhibited $t_{90}=12$ s, which means 90% of the I_{DS} decrease happened in 12 s and then reached a relatively steady value. Such a response time is much shorter than $t_{90}=45$ s for the OFET without OTS SAM. These results demonstrate that our simple and widely applicable strategy is effective for improving the chemical sensing performance of OFETs.

To fully utilizing the advantages of OFETs, flexible sensors were fabricated using a biopolymer, PLA, as the dielectric and substrate simultaneously to construct a device array structure, as shown in Fig. 6a. Fig. 6b presents a photograph of the OFET-based sensors array,

Figure 6 Flexible OFET sensor array and its real-time detection performance: (a) schematic of using an OFET sensor array to detect NH_3 , (b) a photo of the flexible OFET sensor array, (c) AFM topography image of the DNTT membrane evaporated on a PLA film and (d) normalized I_{DS} variation of the flexible OFET upon nitrogen and NH_3 analytes under flat and bent conditions ($V_{DS}=-40$ V and $V_{GS}=-40$ V).

which appears very flexible and ultralight. DNTT film evaporated on PLA exhibits relatively small grain sizes because the surface of the PLA membrane can be less smooth than that of silica (Fig. 6c). When applying the flexible OFET-based sensor in NH_3 detection, the devices exhibited over 20% I_{DS} decrease after 1 ppm NH_3 was injected, and recovered to the baseline when the atmosphere changed back to N_2 background (Fig. 6d). The chemical sensing performance of the flexible sensor was well maintained under bent condition, demonstrating a great mechanical flexibility for the device.

CONCLUSIONS

In summary, we have revealed the significant effect of the grain size of OSC films on the chemical sensing performance of OFETs. The sensitivity of OFETs can be effectively improved only by adjusting common parameters in general device fabrication processes. DNTT films with different grain sizes were firstly fabricated by adjusting the substrate temperature in thermal evaporation process and the surface treatment of the dielectric layer. The OFETs with smaller grain size gives more interval space in the OSC films for the analyte molecules diffusion, and thus provides the devices with higher sensitivity. The relative sensitivity of the device can be enhanced by 5 times by adjusting the grain size. The OFET-based sensor array with PLA dielectric and substrate exhibits relatively small DNTT grain sizes because the surface of PLA membrane can be less smooth than that of silica, and the device thus presented high sensitivity and great mechanical flexibility. Therefore, this work gives a simple and widely applicable strategy for improving the chemical sensing performance of OFETs.

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调节晶粒尺寸来提高有机场效应晶体管的化学传感性能

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摘要 基于有机场效应晶体管(OFET)的化学传感器, 分析物在与位于有机半导体(OSC)薄膜底部的导电沟道发生相互作用之前需要扩散通过整层OSC, 从而大大地限制了器件的灵敏度. 虽然通过设计各种纳米结构OSC薄膜来提高器件传感性能的方法见诸报道, 然而, 这些纳米结构的制备过程复杂且不能广泛适用于种类繁多的OSC材料. 本文首先研究了OSC晶粒尺寸对OFET化学传感器灵敏度的影响, 结果表明更小的晶粒尺寸能为化学分析物的扩散提供更多的空间间隙, 从而有利于提高器件灵敏度. 基于此, 我们通过简单地调控OFET的制备参数, 包括OSC薄膜蒸镀过程中的基板温度, OFET基底的表面处理程度等参数, 将传感器的相对灵敏度提高了5倍. 基于聚合物介电层的柔性传感器也表现出较好的灵敏度. 该工作揭示了OSC晶粒尺寸对器件晶体管性能和传感器性能的互不相同的影响, 并且为提高OFET化学传感器性能提供了一种简便且广适性的策略.