

Optimization of 6,13Bis(triisopropylsilylethynyl)pentacene (TIPS-Pentacene) Organic Field Effect Transistor: Annealing Temperature and Solvent Effects

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Abstract

Abstract: In this contribution, we report on the effect of solvents with different boiling points and annealing temperature on the performance of TIPS-pentacene transistors. Several solvents have been used for TIPS-pentacene thin film processing: toluene, chlorobenzene and tetrahydrofuran. To study the influence of solvent and temperature; the electrical parameters of TIPS-pentacene field effect transistor were measured. The highest values of mobilities were 7.1×10^{-3} cm²·V⁻¹·s⁻¹, 4.5×10^{-3} cm²·V⁻¹·s⁻¹ and 1.43×10^{-3} cm²·V⁻¹·s⁻¹ respectively for TIPS-pentacene field effect transistor using chlorobenzene, toluene and tetrahydrofuran and annealed respectively at 120°C, 150°C and 120°C. We have correlated these electrical performances with AFM images in order to point out the role of morphological properties. It is found that the grain size, and roughness highly affect the electrical parameters.

Keywords

Tips-Pentacene, Transistor, Solvent, Annealing Temperature

1. Introduction

Organic field-effect transistors (OFETs) have gained recently a lot of attraction in organic electronics. Since they achieve a good performance comparable to amorphous silicon (a-Si-H), organic transistors play a key role in next generation of electronic devices. Organic materials such as polymers and small molecules have potential advantages as active layers in the field-effect transistors (FETs) due to processable solution that allows them low-cost, large area and compatibility with flexible substrates [1] [2] [3]. Recently, many efforts have been done to making crystalline organic semiconducting thin films from solution for the use of organic field-effect transistor [4] [5]. Small molecule like 6,13-Bis(triisopropylsilyl)pentacene (TIPS-Pentacene) has gained a lot of consideration due to its environmental stability [6], its solubility in organic solvents [3] and its high field effect mobility higher than 1 $\text{cm}^2 \cdot \text{V}^{-1} \cdot \text{s}^{-1}$ [7] [8]. Solution-processed organic semiconductor active layers are highly interesting mostly in low-coast manufacturing approaches are needed. Hence several works have been done on TIPS-pentacene as active layer of electronics devices. Several studies were achieving to improve the performance of TIPS-pentacene organic field-effect transistors [8] [9] [10] [11] [12]. From those researches, one can identify the influence of process parameters in the performances of organic transistors. Even spin coating [13], dip coating [14] and Ink-jet printing [15] are currently the most widely used solution processing method in organic electronics, many other technics have been used to improve the crystalline film growth [8] [16]. Previous studies show that charge transport properties of organic semiconductor are widely dependent on their crystal structure and morphology [17]-[24]. In solution process, most of organic semiconductors require dissolution in organic solvent. Consequently, various works have done to explore the effect of annealing temperature [21] [25] [26] [27] [28] [29] and solvent influence on the thin film morphology and crystallinity [30]-[35]. It is well known that processing conditions impacted the electrical performances and especially when solvents and temperature are involved.

In this paper we study the influence of solvent and annealing temperature in TIPS-pentacene organic transistor. For that purpose three different solvents, chlorobenzene (CB), toluene and tetrahydrofuran (THF), have been used to dissolve the organic material. The temperature for post fabrication annealing was also chosen from non-annealing (as-prepared) to temperature above the boiling point of all solvents. In order to understand the electrical parameters change, we have correlated the electrical performances to thin film morphology by using atomic force microscopic (AFM) It turns out that the highest value of mobility was obtained with Toluene and chlorobenzene solvents at $T = 150^{\circ}C$ and $120^{\circ}C$ respectively.

2. Experimental

We used a prefabricated bottom-gate/bottom-contact (BG-BC) structure (as seen in **Figure 1**) from Fraunhofer IPMS. N-doped silicon (doping at wafer surface $n\sim3.10^{17}$ cm⁻³) are used as gate electrode with 230 nm of SiO₂ (thermal oxidation) as dielectric. 30 nm Au with 10 nm high work function adhesion layer



Figure 1. (a) schematic structure of Bottom gate-Bottom contact OFET and (b) image of BG-BC OFET.

(ITO) was deposited on wafer for source and drain contacts. All characterized OFETs present a channel length (L) of 20 µm and a width (W) of 10 mm. The devices fabrications begun by the classic steps of cleaning of substrates: sonication in detergent (15 min), acetone (15 min) and isopropyl alcohol (15 min) and deionized water. The substrates were then dried in a UV-Ozone for 15 min to remove organic residuals.

The organic material 6,13 bis(triisopropylsilylethynyl)-pentacene (TIPS-pentacene) was purchased from OSSILA. Three solutions for different solvents were prepared. 15 mg of TIPS-pentacene was dissolved in 1 ml of each solvent (CB, toluene and THF). Each solution was stirring for 24 h at 40°C to dissolve completely the organic material. The hexamethyldisilazane (HMDS) treatment is performed first to ensure a uniform adhesion of the film and solution dewetting on the substrate. 70 µl of TIPS-pentacene solution is deposited on Si/SiO₂ substrate by spin coating to form thin film in two steps: the first one at 2000 rpm for 120 s and the second one at (2500 rpm for 60 s). And then samples are annealed for 10 min before characterization. The chosen annealing temperature was 50°C, 80°C, 100°C, 120°C and 150°C, although we only showed the electrical characteristics of transistor devices for three different temperatures. The thickness of each TIPS-pentacene film was determined by a surface profiler BRUKER DektakXT. Optical proprieties are investigated by using a UV-visible spectrophotometer Perkin Elmer (150 mm InGaAs sphere). The morphology of thin film deposited on SiO₂ was studied by using an XE-100 Atomic Force Microscopic (AFM)electrical characterization was performed by using KEITHLEY 4200-SCS semiconductor characterization system in a glovebox with controlled atmosphere.

The mobility μ was extracted from the saturation region of the transfer curves with the equation:

$$I_{D,sat} = \frac{W}{2L} \mu C_i \left(V_G - V_{Th} \right)^2 \tag{1}$$

where $I_{D,sat}$ is drain current in the saturation regime, W/L is the width to length ratio, C_i is the capacitance per unit area, V_G the gate voltage and V_{Th} the threshold voltage.

3. Results and Discussion

3.1. Effect of Solvent and Temperature on Optical Properties

Optical experiment provides a good way to examine the properties of semiconductors. Measuring the absorption for various wavelength gives information about the band gap of the material, which is important for understanding the optical (electrical) properties of the semiconductors. Measurement are performed at room temperature on 60.5 nm, 88.56 nm and 250.0 nm of TIPS-pentacene film respectively from toluene, chlorobenzene (CB) and tetrahydrofuran (THF) as solvent.

Figures 2(a)-(c) show the absorption spectra of TIPS-pentacene thin film respectively for toluene, CB and THF as solvents.

The absorption curves shows that the absorption rate is higher in TIPS-pentacene thin film cast from tetrahydrofuran film (Figure 2(c)) than TIPS-pentacene film from toluene and chlorobenzene as solvent.

The absorptions spectra show intense peaks in redshift between 660 nm and 680 nm. These peaks are attributed to electronic transition between the HOMO and the LUMO states and correspond to optical band gap wavelength [36]. From the **Figure 2**, there is no shift in the absorption spectra. It is just observed a small enhancement of the absorption rate in the case of toluene as solvent before and after thermal annealing. The increase in the absorption could be recognized as a better molecular organization in the annealed samples [28] which is confirmed by our AFM images of thin films from the three solvents.

Table 1 shows the optical band gap values of TIPS-pentacene for different annealing temperatures and for the three using solvents. By estimating the wavelength at the absorption edge in the absorption spectra, it is possible to calculate the optical band gap of TIPS-pentacene.

Optical band gap energy is obtained from the wavelength of the most intense peak by using Planck's equation:

$$E_g = \frac{1240}{\lambda(nm)} \tag{2}$$

where λ correspond to the threshold absorption wavelength in the spectra and E_g the optical band gap.

Vibronic bands in absorption spectra of TIPS-pentacene films show the influence of solvent, with relative intensities of the bands varied depending on the film thickness and the surface of the film. For example, films deposited with toluene as solvent, the absorption increase by increasing the annealing temperature, which correlates the absorption and the morphology. TIPS-pentacene films cast from toluene as solvent are better organized when the temperature increase. In contrast, the influence of temperature in THF as solvent in TIPS-pentacene thin films absorption is more visible compare to other solvents. Indeed one can see that the film's absorption increase as well as the temperature increases. These results suggest that the absorption depends only on film structure and



Figure 2. Absorption spectra of TIPS-pentacene film cast: (a) from toluene, (b) from Chlorobenzene (CB) and (c) from Tetrahydrofuran (THF).

Solvents	Toluene	СВ	THF	
T (°C)	Eg (eV)	Eg (eV)	Eg (eV)	
50	1.68	1.69	1.67	
80	1.67	1.67	1.68	
100	1.69	1.70	1.67	
120	1.72	1.72	1.69	
150	1.68	1.72	1.67	

 Table 1. Optical band gap of TIPS-pentacene thin film obtained from different temperatures and solvent.

morphology in samples using toluene as solvent while in THF solvent, the annealing influence may be considered. This study shows that all values of the optical band gap are close to 1.6 eV or 1.81 eV [37] [38] [39] and less than that (1.91 eV) given by Saeed *et al.* [40].

3.2. Influence of Solvent and Annealing in Morphology and Electrical Performance

The AFM results of the non-annealed and annealed samples have been investigated to obtain a better insight into the topographical changes as a result of the thermal annealing treatment and solvent effect. The AFM images of the TIPS-pentacene thin film deposited by toluene, chlorobenzene and tetrahydrofuran are respectively shown in **Figure 3**. It shows 1 μ m × 1 μ m AFM topographic images of the TIPS-pentacene thin films before and after post fabrication thermal annealing at 120°C and 150°C.



Figure 3. AFM images of TIPS-pentacene film cast: (a) from toluene, (b) from Chlorobenzene (CB) and (c) from Tetrahydrofuran (THF).

From the AFM images of Figures 3(a)-(c), it is clearly observed the effect of postfabrication thermal treatment. The grain size in toluene's case increases with temperature: 72 nm for non-annealed 70 nm for heated at 120°C and 151 nm for annealed at 150°C. The grain size of TIPS-pentacene in CB is 191 nm, 61 nm and 96 nm for as-prepared, 120°C and 150°C. And for THF, the grain sizes are 29.9 nm, 49 nm, and 82 nm respectively for non-annealed, 120°C and 150°C. The non-annealed film exhibits many small projections and has a grain size of less than 30 nm (Figure 3(c): non-annealed). The largest of crystal grain suggesting that during thermal annealing, some adjacent TIPS-pentacene grains in the non-annealed or annealed at 120°C film join together via a recrystallization process. Several works show postfabrication thermal annealing influence in grain size that could improve electrical performance [21] [25] [41] [42]. The roughness (RMS) of spin-coated film from toluene decreased when increasing postfabrication annealing temperature. Figure 4 shows the output and transfer characteristics of a Bottom-Gate/Bottom-Contact (BG/BC) TIPS-pentacene FETs cast respectively from toluene, chlorobenzene and tetrahydrofuran as solvent. The organic transistors operate in the accumulation mode since the gate electrode is biased negatively with respect to the grounded source electrode. Drain current (I_D) is almost linear with drain voltage at low V_D, whereas it tends to



Figure 4. Output characteristics of TIPS-pentacene FETs: (a) Non-annealed, 120° C and 150° C for toluene used as solvent; (b) For non-annealed, 120° C and 150° C respectively for Chlorobenzene used as solvent; (c) Using tetrahydrofuran as solvent respectively for no-annealed, 120° C and 150° C.

saturate at higher drain voltage due to the pinch off of the accumulation layer. For all electrical output characteristics (**Figure 4**) we clearly observed a linear and saturation regime confirming a field effect behaviour despite the contact resistance effect appears at the origin when temperature increases.

In the case of toluene as solvent, the field effect mobility was 1.2×10^{-3} cm²·V⁻¹·s⁻¹, 1.5×10^{-3} cm²·V⁻¹·s⁻¹ and 4.5×10^{-3} cm²·V⁻¹·s⁻¹ respectively for no-annealed and for thermal annealed at 120°C and 150°C. The saturation mobilities were 2.1×10^{-5} cm²·V⁻¹·s⁻¹, 7.1×10^{-3} cm²·V⁻¹·s⁻¹, 1.34×10^{-4} cm²·V⁻¹·s⁻¹ and 1.41×10^{-4} cm²·V⁻¹·s⁻¹, 1.43×10^{-3} cm²·V⁻¹·s⁻¹, 5.13×10^{-4} cm²·V⁻¹·s⁻¹ respectively for CB and THF at non-annealed, 120°C and 150°C. The slight increase of the mobility could be explained by thermal organisation of film morphology after annealing. The field-effect mobility increases with decreasing the surface roughness (RMS 16.65 nm at 150°C for toluene, 27.51 nm at 120°C for CB and 37.08 nm at 120°C for THF) (**Table 2**).

From this table one could observe the evolution of the surface roughness with grain size depending on the annealing temperature. From Figure 4(b) and Figure 4(c), one could observe the instability of device performance with decreasing of drain current when the annealing temperature is above 120°C. As it is shown by Kim et al. [30] this instability is induced by solvent impurities. The presence of impurities or additional ions at the semiconductor/dielectric interface might induce positive threshold voltage shift observed in devices with CB and THF. This phenomenon is commonly observed in solution processed p-type OFETs in which absorbed water molecules could influence the charge transport. The TIPS-pentacene devices using toluene present negative threshold voltage. At high temperature-temperature above the boiling point of those solvents-the polar atom of the solvent could be ionised and affect the interface of conduction channel. The drain current decrease could also be explained by the contact resistance effect appearing at the origin of the characteristic. The solvent could also influence the field-effect mobility. Kim et al. [32] showed that the field-effect mobility increases when the solvent polarity is increased. Many studies showed

Solvents	Temperature	$\mu (10^{-3} \text{ cm}^2 \cdot \text{V}^{-1} \cdot \text{s}^{-1})$	$\boldsymbol{V_{Th}}\left(\mathrm{V}\right)$	I_{on}/I_{off}	RMS (nm)	Grain size (nm)
Toluene	Non-annealed	1.2	-2.5	8·10 ³	20.41	72
	120°C	1.5	-6.2	10 ³	22.4	70
	150°C	4.5	-4.3	7.10^{2}	16.65	151
СВ	Non-annealed	0.021	6.8	10^{4}	57.79	191
	120°C	7.1	-2.4	10 ³	27.51	61
	150°C	0.13	-2.5	18.7	34.01	96
THF	Non-annealed	0.14	6.1	2.2·10 ³	13.67	29.9
	120°C	1.4	1.1	1.2·10 ³	37.08	49
	150°C	0.51	2.2	50	63.07	82

Table 2. Electrical parameters of OFETs and film morphological properties.

how annealing could affect the morphology of film by creating cracks when solvent is removed consequently the charge carries transport is affected [10] [43]. With a boiling point of 66°C for THF, film obtained from THF used as solvent is rough and disorganized with small grain size. This behavior could be attributed to a rapid solidification of the deposited film. In contrast to high boiling point solvent, the material crystallizes better when increasing temperature with well-ordered film. Toluene and chlorobenzene have respectively 111°C and 132°C as boiling point. So the film has sufficient time to be well organized. This could explain the highest value 7.1×10^{-3} cm²·V⁻¹·s⁻¹ obtained for OFETs fabricated from chlorobenzene annealed at 120°C and 4.5×10^{-3} cm²·V⁻¹·s⁻¹ at 150°C for the toluene using as solvents.

4. Conclusion

In summary, we report solvent and temperature effects on performance of TIPS-pentacene usingtoluene, chlorobenzene and tetrahydrofuran with different boiling points. The optical band gap of TIPS-pentacene is not affected by the annealing temperature and solvent. In contrast the electrical parameters are highly affected by the solvent and annealing temperature. We found that increasing annealing temperature led to increase the field effect mobility. This observation suggests a crystalline structure organisation in TIPS-pentacene semiconductor depending on temperature and used solvent. The processing conditions being highly crucial for device performance, it is well desired in these conditions to take into account solvent and temperature annealing in solution-processed organic transistor. This study confirms and points out the role of slow evaporation rate in solvent and annealing temperature which are of outmost prominence to improve molecular organisation and promote charge transport in organic semiconductor. Transistor devices using Toluene and chlorobenzene as solvents exhibited better performances.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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