



## Letter

## Rubrene thin-film transistors with crystalline channels achieved on optimally modified dielectric surface

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## ARTICLE INFO

## Article history:

Received 25 February 2008

Received in revised form 9 July 2008

Accepted 26 September 2008

Available online 2 October 2008

## PACS:

85.30.Tv

81.40.-z

## Keywords:

Organic thin-film transistor

Rubrene

Self-assembled-monolayer

Surface treatment on dielectrics

## ABSTRACT

We report on the fabrication of rubrene thin-film transistors (TFTs) with surface-modified dielectrics adopting several kinds of self-assembled-monolayer (SAM) on SiO<sub>2</sub>/p<sup>+</sup>-Si substrate. With the dielectric of lower surface energy, the crystalline rubrene growth or amorphous-to-crystalline transformation kinetics is faster during in-situ vacuum post-annealing, which was performed after rubrene vacuum deposition. In the present study, hexamethyldisilazane (HMDS) was finally determined to be the most effective SAM interlayer for polycrystalline rubrene channel formation. Our rubrene TFT with HMDS-coated SiO<sub>2</sub> dielectric showed quite a high field mobility of  $\sim 10^{-2}$  cm<sup>2</sup>/V s and a high on/off current ratio of  $\sim 10^5$  under 40 V.

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### 1. Introduction

Rubrene transistors with crystalline channels, as fabricated by solution- or vapor-induced processes, recently attracted much attention, displaying high mobilities exceeding those of pentacene thin-film transistors (TFTs) that usually have vacuum-deposited patterned channels [1–6]. This means that rubrene TFTs may have higher potentials than those of pentacene TFTs even in patterned device form. However, until recent years rubrene TFTs could hardly draw such a desirable device performance from vacuum-deposited patterned channels [7]. It is mainly due to process-dependent difficulties of vacuum-deposition (thermal evaporation) method in obtaining crystalline channels on a dielectric substrate; this vacuum

process could hardly obtain good crystalline rubrene film because the evaporated rubrene molecules could not easily overcome the energy barrier to the planar conformation [8]. In addition, considering the fact that the surface chemical state of the dielectric is important for the formation of crystalline organic semiconductor layers although those studies appeared in lack of systematic understanding or uncertain in terms of device performance [6,9]. Therefore, in order to achieve a high performance rubrene TFT with vacuum-deposited channel pattern, it is very desirable to exploit the previously-found deposition conditions [10] on an optimally-modified dielectric surface. We thus, as the first step, implemented a systematic study for the crystalline growth of vacuum-deposited rubrene film on several dielectric surfaces modified with such a variety of self-assembled-monolayers (SAMs) as Hexamethyldisilazane (HMDS), 7-octenyltrichlorosilane (7-OTS), and Trichloro(1H,1H,2H,2H-perfluorooctyl)Silane (FTS) in the

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order of hydrophobic tendency. Then as the next step of research, we have fabricated channel patterned rubrene TFTs with those surface-modified dielectrics to determine an optimal SAM layer leading to the best performance rubrene TFTs with enhanced mobilities.

## 2. Experimental section

A 200 nm-thick conventional thermal silicon oxide ( $\text{SiO}_2$ ) on  $p^+ - \text{Si}$  was used as substrates (or gate dielectrics). In order to modify the surface states of our dielectrics, HMDS, 7-OTS, and FTS were applied onto the substrates using the following universal methods [11–15]; prior to the surface modification of  $\text{SiO}_2$  substrates, the substrates were cleaned with acetone, methanol, and de-ionized water in that order, and then the substrates reacted with 7-OTS and FTS solution (0.1 wt.%) using hexane as the solvent by immersion for 1 h at room temperature (RT). In HMDS coated sample case, it was coated on the  $\text{SiO}_2$  substrates by spin casting at RT. After that, the SAM-modified samples were dried in the vacuum oven at 100 °C for 1 h.

The rubrene channel layers (Aldrich, ~99.19% purity) were initially deposited on the substrates at 40 °C by thermal evaporation. After deposition, we immediately carried out in-situ post-annealing at 80 °C for 17 h. For source/drain (S/D) of the rubrene TFTs, 100 nm-thick Au was then evaporated onto the rubrene channels at RT. Nominal channel length and width of our rubrene TFTs were 90 and 500  $\mu\text{m}$ , respectively. All electrical characterizations were carried out with a semiconductor parameter analyzer (Model HP 4155C, Agilent Technologies) in the dark. Atomic force microscopy (AFM: model XE-100, PSIA) was carried out on the surfaces of our rubrene channels and SAM-treated dielectrics. Absorption measurements were also performed for HMDS-, 7-OTS-, FTS-functionalized, and unfunctionalized 80 nm-thick rubrene films on Corning glass with a Varian Cary 5G Spectrophotometer.

## 3. Results and discussion

Table 1 shows the information on the de-ionized water contact angles (c.a.) of pristine, HMDS-treated, 7-OTS-treated, and FTS-treated  $\text{SiO}_2$ , listed in the order of dielectric surface energy. In view of AFM image those pristine or modified dielectric surfaces appeared nearly identical (root-mean-square roughness,  $R_{\text{rms}}$ ; 0.2–0.3 nm for all). The information on the c.a. is very important here because only the surface energy may influence on the rubrene channel formation and the effects from surface roughness can be ignored in our experiments.

Fig. 1a–d are the optical micrographs of rubrene surfaces obtained by thermal evaporation of rubrene molecules at a substrate temperature of 40 °C and subsequent

in-situ vacuum annealing at 80 °C for 17 h. [10] on  $\text{SiO}_2$  substrate surfaces modified with various SAMs: (a) pristine, (b) HMDS, (c) 7-OTS, and (d) FTS. In the figures, the photos I, II, and III are  $\times 90$ ,  $\times 140$ , and  $\times 3.5$  times magnified images of rubrene film surfaces, respectively. According to the photo I in Fig. 1a–d, the final radii of crystalline rubrene discs grown in the same post-anneal period were observed to be 300  $\mu\text{m}$ , 400  $\mu\text{m}$ , 500  $\mu\text{m}$ , and more than 500  $\mu\text{m}$  on pristine, HMDS-, 7-OTS-, and FTS-treated  $\text{SiO}_2$ , respectively, along in the inverse order of the hydrophobic tendency of dielectric surfaces. This means that the growth rate of the nuclei becomes faster with the lower dielectric surface energy. These phenomena may be understandable based on a classical nucleation and growth theory as we deploy it in a simplified manner as follows;

$$\Delta G = \pi R^2 d \Delta G_c + \pi R^2 (\gamma_3 - \gamma_1) + 2\pi R d \gamma_2 \quad (1)$$

where  $\Delta G$  is the total free energy necessary for the amorphous-to-crystalline phase transition of rubrene disc,  $R$  and  $d$  are the radius and thickness of the crystalline rubrene disc,  $\Delta G_c$  is bulk free energy for the phase transition,  $\gamma_1$  and  $\gamma_3$  are the interface energies of dielectric/amorphous and dielectric/crystalline rubrene, respectively.  $\gamma_2$  is the interface energy between amorphous and crystalline rubrene. Since we want to derive a critical radius ( $R_c$ ) of crystal rubrene disc, it can be determined from the following Eq. (2) if the equation satisfies the next conditional formula (3).

$$\left(\frac{\partial \Delta G}{\partial R}\right) = 2\pi(d\Delta G_c + \gamma_3 - \gamma_1)R + 2\pi d\gamma_2 = 0 \quad (2)$$

$$\left(\frac{\partial^2 \Delta G}{\partial R^2}\right) = 2\pi(d\Delta G_c + \gamma_3 - \gamma_1) < 0$$

Here,  $\Delta G_c$  and  $\gamma_3 - \gamma_1$  are always negative values because crystalline phase must be more stable than amorphous phase ( $\Delta G_c < 0$ ) and  $\gamma_3$  is usually smaller than  $\gamma_1$  ( $\gamma_3 - \gamma_1 < 0$ ).

Therefore formula (3) is satisfied. We thus could derive  $R_c$  from Eq. (2).

$$R_c = -\frac{d\gamma_2}{(d\Delta G_c + \gamma_3 - \gamma_1)} \quad (3)$$

Here  $\gamma_1$  is probably almost the same for all the SAM-modified  $\text{SiO}_2$  while  $\gamma_3$  should vary with the chemical state of dielectric substrate. According to our experimental results on the growth rate, it is deduced that the  $\gamma_3$  decreases with the hydrophobic tendency of dielectrics and also that with the stronger hydrophobic dielectric surface the magnitude of  $R_c$  becomes smaller. This means that the crystalline rubrene nuclei on the FTS-modified  $\text{SiO}_2$  are relatively easy to go to the stable growth stage due to their small  $R_c$  or small energy barrier conditions compared to those of pristine  $\text{SiO}_2$ . Since the growth of the crystalline disc becomes fast once they overcome the  $R_c$  condition, it is now understandable that why the dielectrics of the low surface energy display large-sized rubrene discs here. Photo II shows a merged form of crystalline nuclei and their grain boundaries.

**Table 1**

Contact angle measured from (a) Pristine, (b) HMDS treated, (c) 7-OTS treated, and (d) FTS treated  $\text{SiO}_2$ .

	Pristine	HMDS	7-OTS	FTS
Contact angle	~40°	~60°	~85°	~117°

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