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Formation of composite organic thin film transistors with nanotubes and nanowires

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Abstract

Plastic electronics is a rapidly expanding topic, much of which has been focused on organic semiconductors. However, it is also of interest to find viable ways to integrate nanomaterials, such as silicon nanowires (SiNWs) and carbon nanotubes (CNTs), into this technology. Here, we present methods of fabrication of composite devices incorporating such nanostructured materials into an organic matrix. We investigate the formation of polymer/CNT composites, for which we use the semiconducting polymer poly(3,3^{'''}-dialkyl-quaterthiophene) (PQT). We also report a method of fabricating polymer/SiNW TFTs, whereby sparse arrays of parallel oriented SiNWs are initially prepared on silicon dioxide substrates from forests of as-grown gold-catalysed SiNWs. Subsequent ink-jet printing of PQT on these arrays produces a polymer/SiNW composite film. We also present the electrical characterization of all composite devices. © 2007 Elsevier B.V. All rights reserved.

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1. Introduction

In recent years, plastic electronics based on organic semiconductors has achieved significant progress, which is expected to revolutionize the electronics industry [1–9]. Organic thin film transistors (OTFTs) have the advantage of lightweight, bendable features along with cost effectiveness and low temperature processing which allows the use of flexible plastic substrates for fast and inexpensive coverage of large areas [1,2]. Novel applications that cannot be pursued using conventional silicon chips, because of their rigidity and limited size, are rapidly becoming commercially feasible [1]. Organic semiconductors offer promise in terms of properties, processing and cost effectiveness and they can be used in flat panel

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displays, imagers, smart cards, inventory tags and large area electronic applications [7,8]. Ink-jet printing is a very promising technique for inexpensive large area fabrication of electronic and optoelectronic components on plastic substrates [2]. Organic semiconductors have attracted much research interest by virtue of their compatibility with plastic substrates, along with their routine solution processability [10]. Organic TFTs have been successfully fabricated by a variety of methods, including by ink-jet printing [2,11–17]. While organic TFTs often exhibit reasonably good ON/OFF current ratios (up to 107) [13], attempts to improve their low mobilities (typically < $0.1 \text{ cm}^2/\text{V}$ s) [10], poor air stability and limited lifetime [10], remain a subject of ongoing research.

These present drawbacks of stability and performance associated with organic materials make it attractive to pursue other materials for use in plastic electronics. Onedimensional nanostructures, such as carbon nanotubes

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(CNTs) and silicon nanowires (SiNWs), may be feasible for electronics fabricated on flexible substrates while still retaining mechanical flexibility, whether as the sole material in a device structure, or as a compliment to organic semiconducting material. Several groups have recently considered CNTs for fabrication of TFTs, using various fabrication methods, including spin coating, drop casting, and stamp printing, to make devices [18–24]. Inkjet printing holds greater promise as a means of fabrication, as it offers targeted film deposition, and is suitable for industrial-scale production. We have recently demonstrated the viability of using ink-jet printing to fabricate CNT TFTs, which opens the possibility of depositing any type of nanostructured materials for the fabrication of devices using industrial ink-jet technology [25].

Here, we investigate means of incorporating nanostructured materials into organic devices. Techniques for the fabrication polymer/CNT bi-layer composite and polymer/ SiNW bi-layer composite TFTs by ink-jet printing are explored. The dispersion of CNTs in solutions suitable for ink-jet printing is investigated, as well the fabrication of sparse arrays of parallel oriented SiNWs prepared on silicon dioxide substrates from forests of as-grown goldcatalysed SiNWs [26–28]. The performance of these polymer/CNT and polymer/SiNW integrated OTFT devices are also characterized.

2. Experimental

Single-wall HiPCO CNTs (Carbon Nanotechnologies, Inc.) are dispersed in *N*-methyl-2-pyrrolidone (NMP), which has been observed experimentally to be a good solvent for CNTs [29-32]. The HiPCO CNTs used are typically on the order of 1 µm in length and 1 nm in diameter [33]. Typical CNT concentrations prepared are on the order of 0.1-0.2 mg/mL. Sonication is carried out in a 200 W, 20 kHz sonication bath (Bioruptor; Diagenode) for 1 h, before intense ultra-centrifugation for 30 min at 30,000 rpm (Beckman, Optima MAX-E). The top fraction $(\sim 50\%)$ is carefully removed using a pipette, and the rest of the solution is discarded. Scanning electron microscopy (SEM) is carried out using a Philips XL30 SFEGSEM. Prior to ink-jet printing, solutions are filtered using 0.7 µm filter paper (Whatman), in order to ensure that very large aggregates of CNTs are not used in the ink-jet nozzles.

SiNWs are produced using a method previously described [26,27]. An alumina boat of silicon powder is placed at the centre of a furnace and the temperature is set at 1200–1250 °C during growth. Typically, growth is performed for 10 min with nanowires collected downstream at the cooler end of the furnace where the temperature is 800–850 °C. The aligned nanowires are assembled onto Si/SiO₂ or Si/SiN_x substrates by means of a dry shear-sliding assembly method [34,35].

Prior to ink-jet deposition, substrates are immersed in a solution of octadecyltrichlorosilane (OTS) in toluene for 20 min at 60 °C. They are then immersed in a solution of

octanethiol (Aldrich) in toluene for 20 min at ambient temperature in order to prepare a hydrophobic selfassembled monolaver of OTS on the surface [13]. Ink-jet printing is performed using an Autodrop ink-jet dispensing system featuring piezoelectric dispenser heads with 50 µm diameter nozzles (Microdrop GmbH). The ink-jet nozzles are piezodriven, from which drops are deposited by a voltage pulse. The voltage level we typically use is on the order of 150–200 V, with pulse duration of 37 µs. Drops are generally dispensed at a frequency of 50 Hz. These parameters are chosen in order for a single droplet without any smaller satellite droplets to be deposited from the nozzle. Solutions are dispensed onto substrates in ambient conditions where the solvent is allowed to evaporate slowly. Samples are then dried in a vacuum furnace for 3 h at 80 °C, after an annealing step carried out at 140 °C for 30 min.

Electrical contact patterns are defined by UV photolithography and Cr/Au contact electrodes are thermally evaporated. Electrical transport in devices is investigated using a Keithley 4200-SCS parameter analyser. Device preparation and electrical measurements are performed at ambient conditions. Medium integration and a delay of 200 ms are used to ensure no transient instabilities in the current.

3. Results and discussion

3.1. CNT/PQT devices

For CNTs to be effectively deposited by ink-jet, they need to be extremely well dispersed in an appropriate solvent. We have previously discussed a means for achieving this, and calculating the concentration of CNTs in solution (in this case, 0.003 mg/mL) [25,32]. Several techniques have been reported for successful organization of 1-D NWs and nanotubes, one of which involves modification of the surface to promote guided assembly [36]. This is the same method used to align organic semiconducting molecules, and so is employed in this work. A hydrophobic self-assembled monolayer is made from OTS, which is typically used in the alignment of organic semiconducting molecules [37-40]. PQT/CNT composite devices are fabricated in a two-stage process whereby CNTs, dispersed in NMP, are dispensed by ink-jet onto modified gate dielectric surfaces bearing gold electrodes, followed by deposition of poly(3,3"'-dialkyl-quaterthiophene) (PQT) in dichlorobenzene on the CNT films, also by ink-jet.

Fig. 1(a) shows the output characteristics of an ink-jet printed PQT/CNT bi-layer device for different gate voltages. The characteristics clearly resemble those of a p-type field effect transistor (FET), where drain current increases and saturates as the negative drain voltage is increased. Fig. 1(b) shows a logarithmic plot of the drain current as a function of gate voltage for different drain biases. The effective mobility of this device is extracted



Fig. 1. (a) Output characteristics of ink-jet printed CNT/PQT TFT for different gate biases. (b) Transfer characteristics of the same TFT for $V_{\rm D} = -4$ and -40 V.

from the slope of the current voltage characteristics according to

$$\mu_{\rm FE} = \frac{L}{WC_{\rm i}V_{\rm D}} \frac{\mathrm{d}I_{\rm D}}{\mathrm{d}V_{\rm G}},\tag{1}$$

where *L* is the channel length of the device, *W* is the channel width, and C_i is the gate dielectric capacitance. PQT/CNT bi-layer devices exhibit current drive of 1 µA, an ON/OFF current ratio up to 10⁵, with mobility on the order of 0.003 cm²/V s. These results are inferior to those reported in literature for spin-coated PQT films [13]. However, they are comparable to our ink-jet printed pristine polymer TFTs, for which similar mobilities of ~0.003 cm²/V s are observed, with slightly higher ON/OFF current ratios on the order of 10⁶.

From this data, it appears that addition of CNTs in PQT matrix at present leads to a degradation of drain current,

mobility and ON/OFF ratio. One possible explanation for the inferior performance of composite devices is solvent incompatibility between NMP (the solvent for CNTs) and dichlorobenzene (the solvent for PQT) in bi-layer films. Subsequent inspection of CNT films by AFM, before and after annealing at 210 °C, reveals a 75% reduction in the thickness of films. Therefore, when the PQT layer is deposited prior to the annealing step, there is still residual NMP present in the CNT films, which are not completely dried in ambient conditions. This could lead to possible undesirable interaction with dichlorobenzene, and indeed aggregation of CNTs in composite films has been observed even using optical microscopy.

Once this issue of CNT aggregation is addressed, ascertaining an optimal concentration of CNTs in the composites that will increase mobility while retaining desirable device characteristics is of great importance. There will inevitably be metallic CNTs in the composite matrix, which is an obvious drawback to employing CNTs in TFTs. However, ongoing research into the separation of fractions of metallic and semiconducting CNTs holds out the prospect of CNTs playing a significant role in future TFT research [41]. Future experiments will hopefully delineate the role of CNT concentration in the composite network, and its effects on device characteristics.

Nonetheless, our work demonstrates the viability of fabricating composite CNT/PQT TFTs using an ink-jet technique. The potential for this technique should also extend beyond transistors to other devices such as photovoltaic films, OLEDs, etc., and may also be implemented with other nanostructured materials such as semiconducting NWs.

3.2. SiNW/PQT devices

SiNWs are grown thermally in a tube furnace [26,27] (see Fig. 2). An approach for the parallel assembly of 1-D NWs is explored whereby NWs are aligned by a dry shear-sliding method (see Fig. 3(a)) [34]. Arrays of SiNWs are usually assembled by sliding a strip of as-grown SiNW forest on another fixed and flat silicon substrate. It is found that during rubbing, the vertical contact force influences the surface coverage of oriented SiNWs. Experiments carried out for different contact forces (manually controlled) show that heavy contact force causes poor NW alignment, not only breaking the SiNWs, but also damaging the substrate surface (see Fig. 3(b)). Gentler contact improves the quality of NW arrays (see Fig. 3(c)). In addition, the degree of alignment can be controlled by the relative position of the two strips, i.e., the blank strip and that containing the asgrown forest of SiNWs. When the strip with the SiNW nanowire forest is applied face down, a lot of debris and short broken NWs randomly transfer to the surface of the opposite flat substrate during rubbing (see Fig. 3(d)). In contrast, better oriented NWs are transferred in the opposite case, in which the forest is facing upwards (see Fig. 3(e)).



Fig. 2. (a) Schematic drawing of the silicon nanowire synthesis system. (b) SEM images of a silicon nanowire forest produced by this method.

A typical example of parallel assembly of NWs shows that virtually all the NWs are aligned along one direction, i.e., the shear-sliding direction, even though there are still some small deviations with respect to the rubbing direction, which could be achieved by an improved experimental setup (see Fig. 4). Examination of numerous SEM images of assembled NWs taken across the substrate shows that alignment can extend over hundreds of microns. Indeed, alignment of the NWs has been found to extend up to millimetre length scales and seems to be limited only by the size of the substrate.



Fig. 3. (a) Schematic illustration of the shear-sliding process. Shear-sliding assembly. (b) Poorly oriented NWs via heavy contact force and (c) better assembled NWs by gentle contact force. (d) Shear-sliding assembly where NW forest is facing down during transfer, and (e) NW forest is facing up during transfer, following which the average length of aligned NWs is $10-15\,\mu m$.

Fig. 5 illustrates our bi-layer NW/polymer OTFT fabrication process. SiNWs have a core-shell structure, with a single crystal core surrounded by an amorphous



Fig. 4. Highly oriented silicon nanowire arrays. The top-contact electrodes above the NWs were fabricated by photolithography, evaporation and lift-off processes.

silicon oxide shell of 1–3 nm thickness. Synthesized SiNW forests are placed facing upward under another bare substrate, producing an oriented NW thin film by shear-sliding (a and b). The transferred NW thin film consists of an assembly monolayer of NWs oriented in parallel with an average NW spacing of 500-1000 nm. Varying the horizontal rubbing speed and vertical contact force affects the NW spacing. Standard photolithography followed by Cr/Au metallization is used to define source and drain electrodes with the conducting channel parallel to the wire orientation. The orientation of these parallel NWs is not disturbed during the photolithography and lift-off processes (c and d). Furthermore, the dielectric surface and Au electrodes are treated, respectively, with OTS and octanethiol self-assembled monolayers to improve alignment of the organic semiconducting molecules following ink-jet deposition. By means of ink-jet printing, a thin layer of an organic semiconductor polymer is deposited onto the NWs and electrodes (e and f). Fig. 5(g) is a magnified SEM image from which we can



Fig. 5. Illustration of fabrication steps for producing ink-jet printed OTFTs containing parallel-aligned nanowires.



Fig. 6. Schematic cross-section of the OTFT structure employed in this study.

confirm that these NWs still retain their orientation under the polymer layer. Fig. 6 is a schematic of the device structure that results from the process described in Fig. 5.

Fig. 7 illustrates the transfer characteristics of a bi-layer SiNW/PQT OTFT on a SiO₂ gate dielectric. The mobility extracted from transconductance (dI_D/dV_G) measurements in the linear regime $(V_{DS} = -5 \text{ V})$ is $3.81 \times 10^{-4} \text{ cm}^2/\text{V} \text{ s}$ and the ON/OFF current ratio is 1.93×10^4 . In the saturation regime $(V_{DS} = -40 \text{ V})$, the effective field-effect mobility is derived from the transfer curves by using the equation

$$I_{\rm D} = \frac{W}{2L} \times \mu_{\rm sat} \times C_{\rm i} \times (V_{\rm G} - V_{\rm T})^2, \qquad (2)$$

where I_D , C_i , V_G , and V_T represent drain current, insulator capacitance, gate voltage and threshold voltage, respectively. The mobility is $6.62 \times 10^{-4} \text{ cm}^2/\text{V}\text{ s}$ and the ON/OFF current ratio is 1.26×10^4 . The threshold voltage is around -11 V (see Fig. 7(b)). The output characteristics show well-defined saturation behaviour, as illustrated in Fig. 7(a). Most bi-layer SiNW/PQT OTFTs display similar current–voltage characteristics.

The impact of parallel oriented NWs on OTFT performance can be examined by comparison of the electrical performance of SiNW/PQT OTFTs with different SiNW orientation, i.e., the average saturation field-effect mobilities and average ON/OFF current ratios. The average mobility extracted from the devices containing aligned SiNW arrays parallel to the channel between drain and source is slightly higher than the mobility for the devices containing aligned NW arrays perpendicular to the channel. It has also been reported very recently that the mobility of aligned F8BT active semiconducting polymer was enhanced when the polymer chains were aligned to the transport direction by nanoimprinting [42]. Therefore, it is quite possible that the aligned NW arrays affect the orientation of polymer chains. The order of the ON/OFF current ratio is as follows: perpendicular NW/PQT>pristine PQT>parallel NW/PQT. This indicates that the direction of aligned NW affects the electrical characteristics, i.e., for NWs aligned parallel to the channel



Fig. 7. (a) Transfer characteristics $I_{\rm D}-V_{\rm GS}$ of a bottom-gate bottomcontact SiNW/PQT OTFT. W and L are 34 and 12 mm, respectively. (b) A linear plot of $I_{\rm D}^{\rm D2}-V_{\rm GS}$ in the saturation regime. (c) Output characteristics $I_{\rm D}-V_{\rm DS}$ of the same device.

direction, the lower the ON/OFF current ratio; for NWs aligned in a perpendicular direction, the higher the ON/OFF ratio.

To achieve high mobility, it is crucial for the active organic semiconductor polymer to assemble into a highly ordered molecular structure [39,40,43]. It is expected that the parallel-aligned SiNWs may be able to play the role of either assisting polymer stacking or enhancing conductivity, or both. However, the electrical performance of the SiNW/PQT composite devices presented here is lower than typical literature reports, where the mobility is of the order of 10^{-1} - 10^{-2} and the ON/OFF ratio is of the order of 10^6 – 10^8 [13,37,38]. It is still necessary to optimize fabrication conditions for this NW/PQT OTFT architecture, including the orientation of aligned NWs, interaction at the interface between NW and polymer, ink-jet printing parameters (drop shape, driving voltage, and substrate temperature), and annealing conditions.

4. Conclusions

Different techniques for fabricating composite TFTs comprised of organic semiconducting material and nanostructured conductors such as CNTs and SiNWs are demonstrated. The first of these is the fabrication of TFTs comprised of CNTs and semiconducting polymer by means of ink-jet printing. Stable solutions of CNTs are formed, which can be dispensed using an ink-jet printing technique without clogging the nozzles. CNTs are incorporated into an organic semiconducting matrix, and bilayer composite TFTs are fabricated. Also, a simple technique for large-scale parallel assembly of 1-D nanostructures is demonstrated. The dry shear-sliding deposition method of 1-D nanostructures is flexible in terms of the processing sequences and in choice of the materials. These parallel-assembled SiNW arrays are employed in the fabrication of bi-layer SiNW/PQT OTFTs. The electrical measurements of these OTFTs show that the NW alignment direction corresponding to the current flow direction between drain and source influences the field effect mobility and ON/OFF current ratio. However, optimized fabrication conditions have to be found in order to understand the role of parallel-aligned NWs in an organic material. It is assumed that with proper optimization of the printing parameters, such as improved nanowire alignment and density, higher uniformity, and improved ink-jet printing process, the alignment method should be applicable to a wide range of active organic semiconducting polymers to improve device performance in OTFTs.

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