

Characterization of solution processed, p-doped films using hole-only devices and organic field-effect transistors

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ABSTRACT

We report a solution processed, p-doped film consisting of the organic materials 4,4',4''-tris(3-methylphenylphenylamino)triphenylamine (MTDATA) as the electron donor and 2-(3-(adamantan-1-yl)propyl)-3,5,6-trifluorotetracyanoquinodimethane (F3TCNQ-AdI) as the electron acceptor. UV–vis–NIR absorption spectra identified the presence of a charge transfer complex between the donor and acceptor in the doped films. Field-effect transistors were used to characterize charge transport properties of the films, yielding mobility values. Upon doping, mobility increased and then slightly decreased while carrier concentration increased by two orders of magnitude, which in tandem leads to conductivity increasing from 4×10^{-10} S/cm when undoped to 2×10^{-7} S/cm at 30 mol% F3TCNQ-AdI. The hole density was calculated based on mobility values extracted from OFET data and conductivity values extracted from bulk *I*–*V* data for the MTDATA: *x* mol% F3TCNQ-Sdl films. These films were then shown to function as the hole injection/hole transport layer in a phosphorescent blue OLED.

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1. Introduction

Organic semiconductors can be p-doped with an electron accepting material or n-doped with an electron donating material. Conductivity doping into charge transport layers in OLEDs has increasingly gained attention from both academic and industrial research, owing to its benefit of enabling greater film thickness and thereby a wider process window and a higher manufacturing yield [1]. These conductivity dopants generate mobile carriers by the interaction of electron acceptors or donors with either hole transport molecules or electron transport molecules respectively [1,2]. Traditionally, doped films have been characterized using time-of-flight [3], Hall effect measurements [4], or Seebeck effect measurements [5] to obtain mobility, number of carriers, and/or conductivity. Single carrier devices with a structure of metal/doped organic

material/metal have also been used to show conductivity increase in organic films when a conductivity dopant is added [6,7]. The charge mobility in undoped organic films, on the other hand, are often characterized using field-effect transistors [8–10], which has also been used as a tool to characterize doped organic films [11,12].

Solution process such as spin coating, screen printing and inkjet printing is a class of low-cost fabrication method scalable for large-area and high-throughput manufacturing. In particular solution process is well suited to large-scale manufacturing of OLEDs for display and lighting applications, owing to the flexibility of the materials used in OLEDs. In light of this benefit and the advantage of conductivity doping, demonstration and characterization of solution-processed charge transport layer with varying doping level are important to the study of OLEDs and their wide adoption into the market.

We characterize solution processed, p-doped films consisting of the hole transport/electron donor material 4,4',4''-tris (3- methylphenylphenylamino)triphenylamine

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(MTDATA) and the electron accepting p-dopant 2-(3-(adamantan-1-yl)propyl)-3,5,6-trifluorotetracyanoquinodimethane (F3TCNQ-Adl). Poly (N-vinylcarbazole) (PVK) is added as a matrix element to aid in obtaining good films cast from solution (see Supporting Information for further discussion on the use of PVK as a matrix element). F3TCNQ-Adl is a derivative of 2,3,5,6-tetrafluoro-7,7,8,8-tetracyanoquinodimethane (F4TCNQ) that was developed in our laboratories as a model for functionalizing F4TCNQ [13]. F3TCNQ-Adl showed better stability in solution in comparison to F4TCNQ, owing to the anchoring property of the -adl moiety, as evidenced by the longer duration of charge transfer color upon adding a dopant (either F3TCNQ-Adl or F4TCNQ) into a solution containing the hole-transport host such as MTDATA. The MTDATA: x mol% F3TCNQ-Adl films are characterized by UV-vis-NIR absorption, single carrier hole-only devices, and field-effect transistors. These films are then shown to function well as solution processed, p-doped hole injection/hole transport layer in a phosphorescent organic light emitting device (OLED).

In particular, we show that single carrier hole-only devices can be used to measure the conductivity of the doped films and that field-effect transistors can be used to obtain the charge mobility through the film. Using the mobility and conductivity, we calculate the charge density of these MTDATA: x mol% F3TCNQ-Adl films and find it increased from $1 \times 10^{13} \text{ cm}^{-3}$ when undoped to $4 \times 10^{17} \text{ cm}^{-3}$ at 30 mol% F3TCNQ-Adl.

2. Experimental

A 23 mg/mL stock solution of 80 wt.% MTDATA and 20 wt.% PVK was made in *o*-dichlorobenzene (i.e. 18.4 mg MTDATA and 4.6 mg PVK in 1 mL of solvent). The amount of F3TCNQ-Adl needed to equal 10, 20 and 30 mol% (0.35 mg, 0.71 mg, and 1.06 mg) F3TCNQ-Adl with respect to the number of moles of MTDATA in 350 μL of the stock solution was weighed and placed in clean glass vials and dissolved in 50 μL of acetone. 350 μL of the MTDATA:PVK stock solution were added to each of the F3TCNQ-Adl solutions to generate the doped HTL solution used for spin-coating. For the 0 mol% F3TCNQ-Adl solution, 50 μL of acetone was added to 350 μL of stock solution to ensure the same dilution factor. Films of the solutions were spin-coated onto a variety of substrates (glass, glass/ITO or Si/SiO₂ wafer) at 1000 rpm for 90 s. After spin-casting, the films were cured in a vacuum oven starting at room temperature and then heated up to 100 °C, where the temperature for was held constant for 20 min. The oven was then allowed to cool to room temperature overnight. The resulting films were uniform with thickness of 600 Å as measured by ellipsometry.

Indium tin oxide (ITO) on glass was used for single carrier devices and OLEDs (purchased from Colorado Concept Coatings with sheet resistance of 15 Ω/sq and thickness of 1400 Å). For thin film field-effect transistors, heavily doped n-type Si (0.01–0.02 $\Omega \text{ cm}$) with thermally grown 2000 Å thick SiO₂ served as the gate and gate dielectric (purchased from Silicon Quest International). These substrates were

cleaned by a sequence of aqueous detergent solution, deionized water, organic solvents (hot trichloroethylene, acetone, and 2-propanol), and UV ozone (UVO-Cleaner, Jelight Co. Inc, at 15 mW/cm² for 20 min) prior to spin-coating the PVK:MTDATA:F3-TCNQ-Adl HTL film. After curing the film, the substrates were loaded into a nitrogen glove box (<1 ppm H₂O, <1.5 ppm O₂) coupled to a multi-chamber vacuum deposition system, for subsequent fabrication of the devices. For single carrier devices and thin-film field-effect transistors, the electrodes were defined through 1 mm diameter shadow mask and interdigitated shadow mask respectively by thermally evaporating metal (Au, Al, and Ag) in high-vacuum (base pressure below 10⁻⁸ Torr). For OLEDs, sequential organic layers were thermally evaporated in high vacuum (base pressure below 3 $\times 10^{-7}$ Torr) and then the cathodes were defined through a shadow mask with 1 mm diameter holes by thermally evaporating LiF/Al in high-vacuum (base pressure below 10⁻⁸ Torr). Organic layers were deposited at rates 1–2 Å/s, Au was deposited at a rate of 0.5 Å/s, while Al and Ag were deposited at 3–4 Å/s. Quartz crystal oscillators were used to monitor *in situ* the deposition rate and film thickness, which were calibrated *ex situ* using ellipsometry or profilometry.

Resultant devices were tested in air. The device structures used in this work are shown in Fig. 1. For single carrier devices and OLEDs, electrical contact was made using a tungsten probe tip on the ITO anode and a 0.002 in. diameter gold wire directly probing the patterned electrode. For thin-film field-effect transistors, the heavily-doped n-type Si gate was exposed by diamond-pen scratching and contacted by an alligator clip, while the inter-digitated source and drain electrodes were contacted by Au wire. Current-voltage characteristics were measured with an Agilent Technologies 4155B semiconductor parameter analyzer. The light output was detected using a 1 cm² Si photodetector placed behind the OLED coupled directly to a Newport multifunction optical meter. No corrections were made for light wave guided in the organic thin films or the substrate.

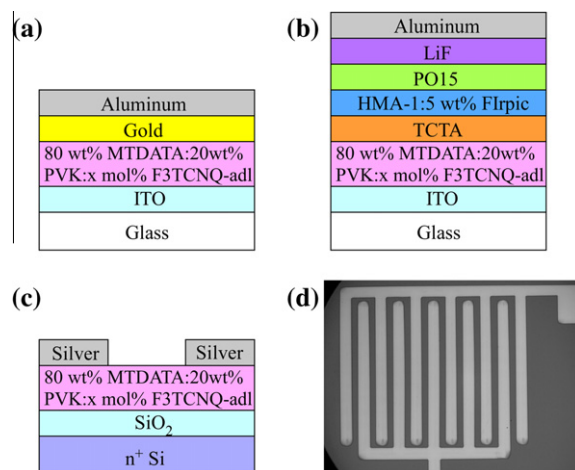


Fig. 1. Device structures used in this work: cross-section diagrams of (a) single-carrier hole-only device, (b) field-effect transistor, (c) and blue OLED, and (d) optical image of the interdigitated source/drain electrodes.

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