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Charge carrier mobility study of a mesogenic thienothiophene derivative in bulk and thin films



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ARTICLE INFO

Article history:

Received 31 October 2013

Received in revised form 13 January 2014

Accepted 15 January 2014

Available online 10 February 2014

Keywords:

Thienothiophene

Ordered mesophase

Charge transport properties

Hole mobility

Time-of-flight

Organic field-effect transistor

ABSTRACT

A novel mesogenic 2,5-bis-(5-octylthiophene)-thieno[3,2b]thiophene (TT) derivative has been synthesized. The fused-ring thiophene, end-capped with two octylthiophenes, exhibits ordered lamellar mesophases which were characterized by polarizing optical microscopy, differential scanning calorimetry and small-angle X-ray diffraction at various temperatures. The charge transport properties were investigated by time-of-flight technique as a function of temperature. On cooling from isotropic phase, a maximum hole mobility value of $0.07 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ was measured in the highly ordered mesophase of the bulk films. Field-effect transistor experiments on both solution and vacuum deposited thin films have also been performed. The solution-processed films exhibit charge carrier mobilities several orders of magnitude lower than values extracted from bulk time-of-flight curves and from vacuum deposited thin film transistors. This work provides evidence that the melt-processing route is an efficient alternative to commonly used solution-processing for fabrication of charge transporting layers from liquid crystalline semiconductors, with performances comparable to evaporation techniques.

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1. Introduction

Since the discovery of conducting properties in π -conjugated polymers [1], π -conjugated systems have attracted

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much attention as futuristic materials for the development and production of the next generation of electronic and optoelectronic devices [2] such as organic solar cells [3–6] or organic field-effect transistors [7,8] (OFETs). The fabrication of commercial OFET devices by a solution process is generally preferred considering its potential for large-area applicability and low cost production. However, the charge carrier mobility of OFET semiconducting active

<http://dx.doi.org/10.1016/j.orgel.2014.01.017>

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layers prepared by such a process is usually lower than that of devices fabricated by vacuum deposition. From a general point of view, that is due to the inevitable loss of long-range scale order and to the formation of “grain boundaries” defects inherent to polycrystalline structures formed during the solvent evaporation stage.

Among all the π -conjugated architectures, liquid-crystalline semiconductors have emerged as promising materials for organic electronics and appear as appropriate candidates to avoid these deleterious effects due to their dynamic and tunable supramolecular organizing and self-healing abilities [9–12]. Recent studies performed on semiconducting calamitic liquid crystals showing highly ordered smectic mesophases have validated this approach and high charge carrier mobilities up to $10^{-1} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ were extracted from time-of-flight (TOF) experiments and OFET measurements [13–17].

Recently, a smart melt-processing route has been reported to fabricate charge-transporting layers from liquid crystalline organic semiconductors. This approach appeared as an efficient alternative to commonly used vacuum or solution processing for the elaboration of well-structured polycrystalline organic films [18–20]. Indeed, this route, which takes advantage on the self-healing abilities of mesophases, allows to control the polycrystalline film morphology on cooling from isotropic state, and can lead to significant improvement of the layer charge transport properties.

Fused thiophene, in particular thieno[3,2-*b*]thiophene is a very attractive and important chemical moiety for the design of organic semiconductors with improved air-stability and relative good transport performances for both low-molecular weight systems [21] and polymers [22]. Indeed, due to the central cross-conjugated double bond, this planar and stable aromatic heterocycle can form a fully conjugated pathway and preserve the close π - π intermolecular distances necessary for high charge carrier mobility when incorporated in π -conjugated systems. Thus, a large number of semiconducting molecules or oligomers based on thieno[3,2-*b*]thiophene unit was recently prepared and successfully studied in evaporated thin-film transistor configuration [23–26]. Moreover, McCulloch et al. have demonstrated the possibility to confer mesomorphic properties to the fused thiophene moiety by synthesizing a semiconducting main-chain liquid-crystalline thieno[3,2-*b*]thiophene polymer. This polymer exhibited enhanced charge-carrier mobility (up to $0.6 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$), when crystallized from the mesophase, due to the formation of large, well organized lamellar domains [27,28]. However, no studies on the influence of the polymorphism on the charge transport properties of mesogenic thieno[3,2-*b*]thiophene based small molecules have been reported so far.

In this context, our main objectives were to design a mesogenic thieno[3,2-*b*]thiophene molecular derivative and to demonstrate that the smart melt-processing route can be used to enhance the morphology and the charge transport properties of organic films based on this kind of material. For this purpose, we prepared a novel mesogenic compound, 2,5-bis-(5-octylthiophene)-thieno[3,2-*b*]thiophene (**TT**). First, we fully studied its thermal and self-

organization behavior by polarizing optical microscopy (POM), differential scanning calorimetry (DSC) and temperature-dependent small-angle X-ray scattering (SAXS). Second, we investigated the hole transport properties of this material using the TOF technique in order to correlate self-organization and mobility in melt-processed bulk samples. Finally, we prepared thin films on SiO_2/Si substrates by solution and vacuum deposition techniques for evaluation of charge transport in OFET configuration, to compare and discuss the experimental results on TOF and FET mobilities of the polycrystalline thin films.

2. Results and discussion

2.1. Material design and synthesis

The **TT** calamitic semiconductor consists of an elongated aromatic core associated with electronic charge carrier transport and peripheral alkyl chains which control the solubility and the self-organization of molecules into layered structure. The synthetic route is shown in Scheme 1. The mesogenic thieno[3,2-*b*]thiophene derivative was synthesized by Stille cross-coupling reaction between the dibromo[3,2-*b*]thienothiophene and the 2-stanyl-5-octylthiophene, in the presence of catalytic amounts of $\text{Pd}(\text{PPh}_3)_3$ and DMF as solvent, with a yield of 91% [29]. The catalyst was prepared *in situ* via reaction of the Pd (acetate) and PPh_3 . The dibromo[3,2-*b*]thienothiophene precursor was prepared via bromination of thieno[3,2-*b*]thiophene with NBS at room temperature in dichloromethane [25]. The 2-stannyl-5-octylthiophene was synthesized from the reaction of the commercially available 2-*n*-octylthiophene with the tributyltin chloride in THF as solvent [30]. All these compounds were purified with column chromatography on silica gel and the final molecule **TT** was also purified with several additional crystallizations in ultra-pure solvents. The products were characterized by ^1H and ^{13}C NMR.

2.2. Cyclic voltammetry, absorption and emission spectra measurements

Absorption and emission spectra of the **TT** molecule in chloroform are shown in Fig. 1a. The absorption spectrum shows a peak with a maximum at 382 nm while the emission spectrum presents two vibronic peaks centered around 430 and 455 nm as well as a shoulder close to 480 nm. The optical bandgap (E_g) of **TT** can be approximated by determining the intersection of its absorption and emission spectra [31], which is around 418 nm. Thus, the E_g was estimated to be 2.96 eV (see experimental Section 4.2).

The cyclic voltammetry (CV), typically used to evaluate the energy levels of organic semiconductors in solution, were carried out to determine the highest occupied molecular orbital energy (E_{HOMO}) of **TT** compounds. The electrochemical potential was calibrated using the ferrocene/ferrocenium (Fc/Fc⁺) couple as an external standard which was measured under same condition before and after the measurements of the **TT** sample. The energy level of the

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