Photoelectronic properties of composite films based on conductive polymer PIDT-BT and single-walled carbon nanotubes

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Abstract

We investigated photoelectronic properties of composite films based on semiconducting polymer poly(indacenodithiophene-co-benzothiadiazole) (PIDT-BT) and single-walled carbon nanotubes (SWCNTs). It was found that the absorbance spectra of PIDT-BT and PIDT-BT/SWCNT (1:0.3) films were in the spectral region from 200 to 800 nm, with an absorbance edge at ca. 1.71 eV. Adding of ca 30 wt% of SWCNT to the PIDT-BT polymer matrix resulted in a decrease in absorbance by a factor of 1.5. We made a hole-only device based on PIDT-BT lightly doped with carbon nanotubes. Current–voltage characteristics (I – V s) of PIDT-BT/SWCNT (1:0.3) composite films were measured within temperature interval from 300 to 77 K in the dark and under illumination by a solar simulator. I – V s demonstrated an increase in photocurrent under illumination at 300 K by factor of ca. 1.4–1.7 with respect to dark I – V s. The dark I – V s were investigated in the temperature interval from 300 to 77 K. It was found that the temperature dependence of the resistivity of PIDT-BT/SWCNT (1:0.3) films followed a power law in this temperature region. It has been established that the main charge carrier transport mechanism is the space charge limited current mechanism in the dark and the space charge limited photocurrents under illumination.

Keywords Semiconducting polymers · Single-walled carbon nanotubes · Charge transport · Temperature dependence of resistivity · Photoelectronic properties

1 Introduction

Recent developments of semiconducting polymer–inorganic hybrid nanocomposites have attracted the interest of scientists from both fundamental and applied points of view, since such nanocomposites are used in various photoelectronic applications [1]. This is due to the unique properties of such hybrid nanocomposites, namely flexibility, adaptability, the ability to print over a large area and their lightweight, along with noticeable electrical conductivity, low thermal conductivity, which can facilitate their use in solar cells, photodetectors and field-effect

transistors. It has been shown that the introduction of single-walled carbon nanotubes (SWCNTs) into the conducting polymer matrix improves the electrical conductivity, as well as the mechanical properties of the neat polymers [2]. SWCNTs have a unique structure, narrow distribution in size, high available surface area, low resistivity and high stability [3]. Recent research on CNTs indicates it is a potential material for the organic and hybrid solar cells [4, 5]. Namely, CNTs have a remarkably high level of electronic mobility (much higher than the hole mobility of semiconducting polymers) and work function similar to that of the electrodes (Au or ITO) which makes them an

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ideal candidate for the usage in the hole-extraction layer (or hole transporting layer) in organic photovoltaics [6, 7]. Investigation of SWCNT–polymer solar cells has been conducted toward developing alternative, lightweight, flexible devices for space power applications [5]. Benzothiadiazole (BT)-based polymers such as PIDT-BT with indacenodithiophene (IDT) as the donor (*D*) and BT as the acceptor (*A*) unit have been recognized as one of the first successful BT-based *D*–*A* copolymers with a mobility exceeding $1\text{ cm}^2/\text{Vs}$ [8–10]. Thin films of PIDT-BT exhibited low crystallinity with a large π -stacking distance and face-on orientation. A high hole mobility up to $1.25\text{ cm}^2/\text{Vs}$ was achieved in bottom-contact/top-gate field-effect transistors [8]. Photoelectronic properties of both, PIDT-BT and SWCNTs, were investigated well during the last years [2, 3, 8–10]. Both carbon nanotubes and semiconducting polymers, such as PIDT-BT, possess conjugated π -systems, and the nature of their electronic interaction is anticipated to occur via π – π stacking. At the same time, the use of hole-only devices such as PIDT-BT lightly doped with CNT with gold electrodes is of important for mobility measurements. However, the doping effect of PIDT-BT semiconducting polymer films by the introduction of SWCNTs has not yet been investigated. In addition, the electronic properties and the transport mechanism of composite films based on the PIDT-BT semiconducting polymer weakly and medium doped of SWCNT have not been studied in detail at temperatures up to 77 K.

In this work, we obtained films with a hole-only conduction based on the PIDT-BT semiconductor polymer, with weak and intermediate doping with single-walled carbon nanotubes, and investigated their optical and electrical properties in order to clarify the mechanism of charge carrier transport in such composite films. The obtained films were characterized by absorbance, atomic force microscope and current–voltage characteristics (*I*–*V*s) techniques. Adding of ca. 30 wt% of SWCNTs to the PIDT-BT matrix resulted in a decrease in absorbance by factor of 1.5. The results of measurements of the *I*–*V*s of PIDT-BT/SWCNT composite films (1:0.3) in the dark and under illumination at temperatures of 300–77 K show that the charge carrier transport is determined by the mechanism of the space charge limited currents in the dark and by the space charge limited photocurrents under illumination, and the temperature dependence of the resistivity of such films obeyed a power law.

2 Experimental

To prepare samples, the conjugated polymer PIDT-BT with a band gap $E_g \approx 1.71\text{ eV}$ [10] and SWCNTs (obtained from Sigma-Aldrich with the following parameters: 98%

semiconducting, 2% metallic, with the length ca. 0.3–5 μm , diameter of ca. 1.2–1.7 nm, a density of ca. $1.7\text{--}1.9\text{ g/cm}^3$) and used without additional treatment. Both components were dissolved and dispersed separately in chloroform. Subsequently, the solutions were mixed with relative amounts of 1:0.1 and 1:0.3 and subjected to ultrasonic treatment for 10 min on a Bandeline Sonopuls HD 2070 ultrasonic mixer (at a frequency $f \approx 20\text{ kHz}$). The resulting solution was applied by drop-cast technique at a temperature of 300 K in an atmosphere of N_2 on glass substrates with thermally evaporated gold (Au) electrodes on top. (We used the usual lithography technique to obtain an electrode spacing of $\approx 15\text{ }\mu\text{m}$.) Afterward, the films were dried at 100 °C under N_2 for 10 min and kept in vacuum desiccator (MB SANPLATEC Corp.) until measurements. Thus, in our study, we are dealing with films with hole-only conductivity based on PIDT-BT, with weakly and intermediate doped with SWCNT, which are referred to as composite films in the text. The morphology of the PIDT-BT/SWCNT composite films was investigated by atomic force microscope (AFM)—Solver P47-NT-MDT. The thickness of the films obtained by drop casting onto quartz substrates was $\approx 1\text{ }\mu\text{m}$, according to the AFM data. The chemical structure of PIDT-BT/SWCNT and the schematic morphology of the PIDT-BT/SWCNT nanocomposite films are shown in Fig. 1a–c, respectively. Absorbance spectra of the films deposited by drop-casting technique onto quartz substrates were studied using a Cary-50 (Varian) spectrometer. The DC *I*–*V*s of the PIDT-BT and PIDT-BT/SWCNT films were measured in nitrogen atmosphere in the dark and under solar simulator illumination at temperatures from 300 to 77 K using a sample holder of a liquid N_2 cooled optical cryostat optCRYO198 with temperature stabilization and DC electronic computer-controlled measuring system based on a Keithley 6487 picoamperemeter/voltage source. The DC voltages were varied from -10 to $+10\text{ V}$. The electrical contacts for the samples were made with a silver wire, which was fixed to the metal electrodes with a silver paste.

3 Results and discussion

The results of AFM investigations of the PIDT-BT/SWCNT (1:0.3) film are shown in Fig. 2a–c. As can be seen from these figures, the surface of PIDT-BT/SWCNT nanocomposite films was moderately rough. We found that the overall height variation was about 50–100 nm, exhibiting steps of several nm (Fig. 2b), which may reflect SWCNTs and their agglomerates covered with the PIDT-BT polymer. It was found that the average roughness and root-mean-square (RMS) values of the PIDT-BT/SWCNT film (1:0.3) as shown in Fig. 2a–c are about 8.8 nm and 11 nm, respectively. These

Fig. 1 Chemical structure of PIDT-BT polymer, $R=4$ -*n*-hexylphenyl, $C_{16}H_{33}$ (a), SWCNT (b) and schematic drawing of the morphology of the PIDT-BT/SWCNT nanocomposite film (c)

parameters are higher than that of a pure PIDT-BT film, where the average roughness and root mean square are 4 nm and 5 nm, respectively (not shown). As can be seen from our AFM measurements, the modification of the PIDT-BT polymer with SWCNT led to a relatively smooth surface of the PIDT-BT/SWCNT composite films.

Figure 3 shows typical absorbance spectra of neat PIDT-BT (1) and PIDT-BT/SWCNT (1:0.3) films (2) deposited onto quartz substrates. As can be seen from Fig. 3, the absorbance spectra of PIDT-BT and PIDT-BT/SWCNT (1:0.3) films were in the spectral region from 200 nm to 800 nm. It was found that the absorbance onset for PIDT-BT and PIDT-BT/SWCNT (1:0.3) films is about 725 nm, which corresponds to 1.71 eV, which correlates well with the band gap of the PIDT-BT polymer: $E_g \approx 1.71$ eV [8]. As evident from Fig. 3, adding of about 30 wt% of SWCNTs to the PIDT-BT polymer matrix resulted in a decrease in absorbance by a factor of 1.5 without changing of absorbance maxima (at ~ 688 nm and ~ 417 nm) and absorbance edge position.

The typical I -Vs of PIDT-BT/SWCNT (1:0.3) composite film measured in the dark and under illumination by solar simulator at 300 K are shown in Fig. 4a, b. It is found that photocurrent of PIDT-BT/SWCNT (1:0.3) film at 300 K increased by factor ≈ 1.5 – 1.7 under solar simulator illumination with respect to dark I -Vs. There was some small hysteresis of I -Vs of composite film at 300 K in the dark probably due to the influence of shallow traps in the polymer matrix. However, as can be seen from Fig. 4a, b, this small hysteresis of I -Vs disappeared for negative voltages under and after solar simulator illumination since all traps were populated (Fig. 4a, curves 2 and 3). This reproducible hysteresis of I -Vs in PIDT-BT/SWCNT films is very similar to that observed at 300 K in composite films based on SWCNTs embedded in poly(methyl methacrylate) (PMMA) [11]. As in the case of PIDT-BT/SWCNT films, a stable hysteresis loop along anticlockwise direction at positive and negative biases was observed in PMMA/SWCNT films and the width of the hysteresis was reduced or quenched when the film was exposed to light [11]. By analogy with PMMA/SWCNT composite films, the observed I -Vs hysteresis effects of the PIDT-BT/SWCNT films have been attributed to the injection of electrons from the electrodes to the trap sites located in the PIDT-BT/SWCNT layer.

Inset 1 in Fig. 4b shows $\lg$ - $\lg$ plots for positive voltages for the same sample before and under illumination. As can be seen from Inset 1, the dark I -Vs at low voltages are linear, but not ohmic, which is characteristic of the mechanism of space charge limited currents (SCLC) [12, 13]. The same behavior was found for the I -Vs of this sample when illuminated by a solar simulator, which means that the mechanism of space charge photocurrents is the main transport mechanism in lighting [14]. For comparison, we measured the I -Vs of a neat PIDT-BT polymer film at 300 K deposited onto a glass substrate under the same conditions as the PIDT-BT/SWCNT (1:0.3) film. Inset 2 in Fig. 4b shows I -Vs of neat PIDT-BT film in the dark and under solar simulator illumination. As evident from Inset 2 in Fig. 4b, the DC conductivity of the neat PIDT-BT polymer film was lower by two orders of magnitude compared with PIDT-BT/SWCNT (1:0.3) film. The dark current of pure PIDT-BT is very low, but it increases by two orders of magnitude after illumination with a solar simulator. Higher, but also very low DC conductivity was also found for PIDT-BT/SWCNT (1:0.1) composite films (not shown).

Figure 5 presents I -Vs of the PIDT-BT/SWCNT (1:0.3) composite film measured in the dark at different temperatures within temperature interval from 300 to 77 K. As evident from Fig. 5, the magnitude of DC current went down gradually as the sample temperature decreased from 300 K down to 77 K. The resistivity at each temperature was estimated from the linear regime of the I -Vs at voltages varied from +1 to -1 V. It should be noted that, from Inset 1 in

Fig. 2 Results of AFM investigations of the surface of PIDT-BT/SWCNT (1:0.3) films

Fig. 3 Absorbance spectra of pure PIDT-BT (1) and PIDT-BT/SWCNT (1:0.3) (2) films

Fig. 4b, in fact, the linear I - V mode in a device with one type of carrier is not ohmic and obeys the dependence $I \sim V^x$, where $x > 2$, which is characteristic of the SCLC mechanism [15, 16]. The temperature dependence of the resistivity $\rho(T)$ of the PIDT-BT/SWCNT (1:0.3) nanocomposite film is shown in Fig. 6. Inset in Fig. 6 demonstrates the $\rho(T)$ vs. $10^3/T$ dependence for the same sample (curve 1) and for the sample PIDT-BT/SWCNT (1:0.1) (curve 2). We could not measure the $\rho(T)$ dependence of the pure PIDT-BT film due to the very high resistance of the sample and insufficient sensitivity of our equipment. As can be seen from Fig. 6 and the inset, the $\rho(T)$ dependence of PIDT-BT/SWCNT (1:0.3) film has the nature of an activated process but did not exhibit a constant activation energy within the entire temperature interval. Unlike the PIDT-BT/SWCNT sample (1:0.3), the $\rho(T)$ dependence for the PIDT-BT/SWCNT sample (1:0.1) (curve 2 in Fig. 6) is very strong and shows only constant activation energy. In fact, for temperatures from 300 to 280 K for the sample 2 and from 300 to 250 K for the

Fig. 4 **a** I - V s of PIDT-BT/SWCNT (1:0.3) nanocomposite film measured in the dark before illumination (curve 1), under illumination (curve 2) and after illumination (curve 3) at 300 K; **b** $\lg I$ vs. V for the same sample before and under illumination. Inset 1 in **(b)** shows the log-log graphs for positive voltages for the same sample at 300 K. Inset 2 in **(b)**: I - V s of neat PIDT-BT film at 300 K under the same conditions

sample 1, the behavior can be described by an exponential dependence:

$$\rho(T) = \rho_0 \exp(E_{\text{act}}/k_B T) \quad (1)$$

where E_{act} is the activation energy, T is the temperature, and k_B is the Boltzmann constant. E_{act} can be calculated from the following equation:

$$E_{\text{act}}(\text{meV}) = (200 \Delta \lg \rho) / (\Delta 1000/T) \quad (2)$$

According to our estimates, E_{act} for the PIDT-BT/SWCNT (1:0.1) film at $T=300$ – 280 K and for the PIDT-BT/SWCNT (1:0.3) film at $T=300$ – 250 K were found to be $E_{\text{act}} \approx 1.95$ eV and $E_{\text{act}} \approx 62$ meV, respectively. However, at lower temperatures, a transition of $\rho(T)$ to a power law regime $\rho(T) \sim T^{-m}$ for the PIDT-BT/SWCNT (1:0.3) film was observed. One can expect a transition in $\rho(T)$ to a variable-range hopping

Fig. 5 I - V s of the PIDT-BT/SWCNT (1:0.3) nanocomposite film measured in the dark at different temperatures within the temperature interval from 300 to 77 K at $T[K] = 300, 280, 260, 240, 220, 200, 180, 160, 140, 120, 100, 90, 85, 80, 77$

Fig. 6 Temperature dependence of the resistivity of the PIDT-BT/SWCNT (1:0.3) nanocomposite film. Inset: $\rho(T)$ vs. $10^3/T$ dependences for the same PIDT-BT/SWCNT (1:0.3) sample (curve 1) and for PIDT-BT/SWCNT (1:0.1) (curve 2) nanocomposite film

(VRH) transport regime described by the dependence: $\rho(T) = \rho_0 \exp(T_0/k_B T)^p$.

In our opinion, in the case of the PIDT-BT/SWCNT (1:0.1) nanocomposite film, the main transport mechanism is the thermal activation of carriers from HOMO to LUMO, which can also be expected in the film of pure PIDT-BT due to the observed $E_{\text{act}} \approx 1.95$ eV which is of the same order as the energy gap of the PIDT-BT polymer (but not exactly the same). Thus, we can assume that the $\rho(T)$ dependence of the pure PIDT-BT film should be somewhat similar to the dependence obtained for the PIDT-BT/SWCNT film (1:0.1). However, in case of PIDT-BT/SWCNT (1:0.3)

nanocomposite films, the transport mechanism is more complex, consisting of several mechanisms dominating at different temperature intervals. In particular, transport should involve a thermally activated hopping mechanism at high temperatures. At intermediate temperatures, the effect of barriers to charge injection can be significant. It is worth noting that we used gold electrodes to inject holes into our device with one type of charge carriers. Since the HOMO energy level is deeper than the work function of Au, it can be expected that the energy barriers to charge injection are about 100 meV or more. The observed activation energy of the $\rho(T)$ dependence correlates with this energy [17]. A VRH transport regime might be possible at temperatures below 77 K [18–20]. The low-temperature charge transport in single SWCNT is the subject of intensive discussion [21]. However, such low-temperature experiments require particularly careful sample preparation. Usually, the VRH model is valid mostly at low temperatures. Thus, one may suggest that the tunneling model and its derivatives are more appropriate in the case of low-temperature transport of PIDT-BT/SWCNT composite films below 77 K.

Figure 7a, b demonstrates the I - V s of PIDT-BT/SWCNT (1:0.3) composite film measured in the dark and under illumination by a solar simulator at 77 K. As evident from Fig. 7a, b, the photocurrent of the PIDT-BT/SWCNT (1:0.3) film at 77 K increased by factor of ca. 1.4 under illumination by a solar simulator with respect to dark I - V s, similar to the behavior observed for the same sample at room temperature. As shown in Inset 1 in Fig. 7b, I - V s at 77 K at low voltages are not ohmic; they follow the dependences $I \propto V^x$, where $x > 2$ in the dark and under illumination, which is typical for the SCLC mechanism [15, 16] similar to I - V s for the same sample measured at 300 K. In contrast to our results at 300 K, there is no hysteresis I - V s in the dark and under illumination at 77 K, so when the PIDT-BT/SWCNT composite film (1:0.3) cools down to $T \approx 77$ K, the transport mechanism responsible for the hysteresis I - V s is suppressed (this may be traps and the ionic conductivity typical of semiconductor polymers at room temperature [18]) in the PIDT-BT polymer matrix (probably due to $R = C_{16}H_{33}$), achieving a rather balanced charge transport in hybrid PIDT-BT/SWCNT structure. One can conclude that photoelectronic properties of composite films based on conductive polymer PIDT-BT modified with SWCNT remained similar (in the dark and under solar simulator illumination) within the temperature range from 300 to 77 K and the transport mechanism is determined by SCLC when measured in the dark and by space charge limited photocurrents when measured under illumination.

The details of charge carrier transport in the Au/PIDT-BT/SWCNT/Au structures can be understood from the energy diagram as shown in Fig. 8. (This energy diagram is based on results published in references [6, 20].) As evident from

Fig. 7 I - V s of PIDT-BT/SWCNT (1:0.3) nanocomposite film measured in the dark and under illumination at 77 K. Inset shows the $\lg$ - $\lg$ plots for positive voltages for the same sample at 77 K

Fig. 8 Band diagram of the Au/PIDT-BT/SWCNT/Au structure

Fig. 8, excitons are generated in the PIDT-BT polymer, and holes are collected at the Au contact. Electron transfer is possible through the conduction band of the semiconducting SWCNTs. In our case, one may suggest that the

semiconducting SWCNTs played a role as an electron-conducting network. However, as evident from our estimation of the activation energy, PIDT-BT/SWCNT (1:0.3) film at temperatures 300–250 K, the obtained value of $E_{\text{act}} \approx 62$ meV is much lower than the one expected for semiconducting SWCNTs ($E_{\text{act}} \approx 600$ meV [22]). This may indicate the influence of injection barriers to the charge transport in such systems. It should be noted that for experiments on charge carrier transport at low temperatures, we used gold electrodes in our single-charge carrier devices. However, for device applications, other electrode materials such as ITO, PEDOT:PSS, Al and Ca will be selected in our next research.

4 Conclusions

We investigated the effect of SWCNT doping of semiconducting polymer PIDT-BT films. It was shown that adding of 30 wt% of SWCNT to the PIDT-BT polymer matrix resulted in a decrease in absorbance by a factor of 1.5 without changing of the absorbance edge position. I - V s of PIDT-BT/SWCNT (1:0.3) nanocomposite film measured at temperatures from 300 to 77 K in the dark and under illumination by a solar simulator, demonstrated an increase in photocurrent under illumination at 300 K by factor of ca. 1.4–1.7 with respect to dark I - V s. It was found that the temperature dependence of the resistivity of a lightly doped PIDT-BT/SWCNT nanocomposite film (1:0.1) is determined by the transport of charge carriers in which the thermal activation mechanism prevails; however, for PIDT-BT/SWCNT with intermediate doping (1:0.3), the $\rho(T)$ dependence of the films follows a power law and the mechanism of injection of holes from gold electrodes into our single-carrier device prevails. It has been established that the main mechanisms of charge carrier transfer in such films are SCLC then measured in the dark and photocurrents limited by the space charge under illumination in the temperature range 300–77 K. The obtained nanocomposite films are expected to be useful for various nanotechnology-based hole-only devices.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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