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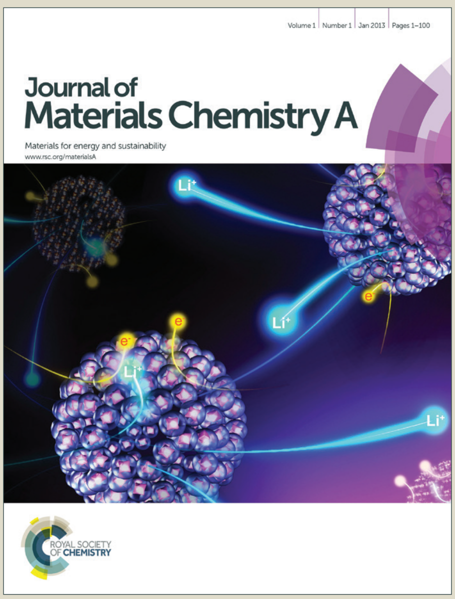

Journal Name

ARTICLE

Schottky Contact of Artificial Polymer Semiconductor Composed of Poly(dimethylsiloxane) and Multiwall Carbon Nanotube

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Organic semiconductors have attracted great attention for offering an attractive alternative to conventional inorganic semiconductors due to lower costs, simpler synthesis and well flexibility. In this work, we fabricated a flexible organic semiconductor by incorporating multiwall carbon nanotubes (MWNTs) into insulating poly(dimethylsiloxane) (PDMS) rubber and researched the contact properties in the metal/composite junctions. The results reveal that the contact properties depend largely on the work function of the metals and the MWNT loadings of the composites. To realize the performance variations of copper/composite junctions characterized by important parameters, Schottky Barrier Heights (SBHs) were measured with various MWNT loadings of the composites. The SBH decreased with the increase of the MWNT loading, exceeded 0.783 eV for 0.35 wt % composite and shared the same changing trend with the composite Coulomb band gap as a function of the MWNT loading. A quantitative analysis of photo-voltages by the photovoltaic tests was used to verify the reliability of these SBHs. The stability test provides direct evidence that the composites possess a good durable performance at ambient temperature. This work shows that the contact properties in the metal/composite junctions cannot be neglected in the application of the composites on flexible electronics.

Introduction

Semiconductors, the core units of most electronic products, have been studied extensively for several decades for their excellent mechanical, electronic, photonic, thermal and electrochemical properties.[1-3] Currently, tremendous efforts have been made in researches of low dimension semiconductor materials, such as carbon nanotube (CNT), and organic semiconductor materials for their unique properties and great potential applications.[4] CNTs are hollow, cylindrical nanostructures composed of one or up to dozens of graphitic shells. They are a promising candidate for flexible electronics like thin-film transistors due to their high conductivity, superior charge carrier mobility, excellent flexibility and compatibility with low-cost, large-scale production.[5-7] Organic semiconductors, which promise mechanical flexibility and large-area manufacturing at ambient conditions at low cost, represent excellent potential for commercial applications such as displays, sensors, logic circuits and solar cells.[8-13] Recently, new opportunities are sought in order to scale up the applications of organic-based semiconductors and enhance their performance.[14-17] Blending organic polymers with low dimension conductive materials seems an interesting approach in view of increasing the conductivity and tunability of organic electronics as well as introducing other feathers.[18-21]

In our previous work, we have demonstrated some semiconductor behaviors of the artificial composites fabricated by incorporating multiwall carbon nanotubes (MWNTs) into insulating poly(dimethylsiloxane) (PDMS) rubber. Coulomb gap variable range hopping (CG VRH) model could satisfactorily explain the semiconductor behaviors in low MWNT-loading composites, and field effect transistors prepared using 0.35 wt % composite showed a typical P-type behavior with a high effective mobility of 1.98 $cm^2/V \cdot s$.[22] These results suggest that low MWNT-loading composites can be treated as P-type semiconductor materials and used in flexible electronics for the feature of flexibility. Here, flexibility means that the composites have the advantages of light weight, low cost and simple technology, and they are portable, bendable, convenient and easily processed. In most cases, the contact type in metal and semiconductor junction may have a significant impact on the device performance.[23,24] For instance, there is a strong relationship between the photocatalytic efficiency and the metal/semiconductor (M/S) junction contact type in the field of photocatalysis.[25] Therefore, the investigation of the metal/MWNT-PDMS composite contact is of crucial importance to the utilization of the composites.

There exist two types of M/S contacts, Ohmic contact and Schottky contact. Herein, we analyze the contact types between MWNT-PDMS composites and three kinds of metals (gold, copper and aluminium). The contact properties of the metal/composite junctions depend largely on the work function of the metals and the MWNT loadings of the composites. We further analyze the Schottky barrier heights (SBHs) of the copper (Cu)/composite contacts and find out that the variation trend of the SBH with MWNT loading is the same

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as that of the composite Coulomb band gap. Photovoltaic samples based on the Schottky junction of the 0.35 wt % composite were fabricated to verify the SBHs of the metal/composite contacts. The durability test reveals that the composite-based devices can maintain a good performance for a long-term duration at room temperature. These results imply that the MWNT-doped insulating polymer composites, which have characteristics of cheap raw materials, simple synthesis, high mechanical flexibility and easy tuned physical properties, can be used as Schottky diodes and may have potential applications in electronic devices.

Experimental

Sample Fabrication

The PDMS rubber used here was a transparent flexible insulator (10^{14} S/m) polymerized by two different parts (part A and part B) with weight ratio 1:1. MWNTs used here were synthesized by chemical vapor deposition (commercial MWNTs, purity>95 %, mean diameter~10 nm and length~10 μm).The MWNT-PDMS composite was fabricated by a three-step method. Firstly, the MWNTs were dispersed into part A by intensive ultrasonic waves (200 W) for 20 min with the aid of ethyl acetate as a dilute solvent. Secondly, part B (1:1) was added into the mixture with manual stirred for 10 min after evaporating the solvent. At last, the mixture was poured onto a polytetrafluoroethylene substrate and cured at 80 °C for 4 hours. Samples for contact tests, with a size of 10 mm in diameter and 1 mm in thickness, were prepared by curing the composite between two metal plates as electrodes (metal/composite/metal). The MWNT loadings in this study were in the range of 0.35-2 wt %. This lower limit is the percolation threshold of the composites (0.35 wt %).

By using tweezers to pull out a CNT bundle, the width of which was determined, from the super-aligned CNT (SACNT) arrays, we could get a continuous ribbon whose width was equal to the initial bundle picked.[26] Repeating the process above, a uniform two-layer SACNT film was obtained with a square resistance of 200 /□. The photovoltaic samples with a single layer structure were fabricated in two steps. First, spin coated (2000 rpm for 60 s) the pre-curing 0.35 wt % MWNT-PDMS composite onto the SACNT film and cured the film at 80 °C in a vacuum oven for 4 hours. Then, a metal (copper or aluminium) top electrode was evaporated on the composite under high vacuum with a thickness of 100 nm. We chose SACNT films as the anode for its flexibility, high transparency, low resistance and well directionality.

Measurements

The microstructure of the samples was probed by scanning electron microscopy (SEM) (Sirion 200, resolution 1.0 nm). All current (*I*)-voltage (*V*) curve measurements were made with a Keithley 2410 source meter at room temperature. The photovoltaic samples were tested under dark and nominal AM 1.5 illumination with an intensity of 1000 W/m^2 (Zolix Solar Simulator SS1000A) conditions through the SACNT side with a property measurement system (Keithley 2410 source meter) at room temperature.

Results and Discussions

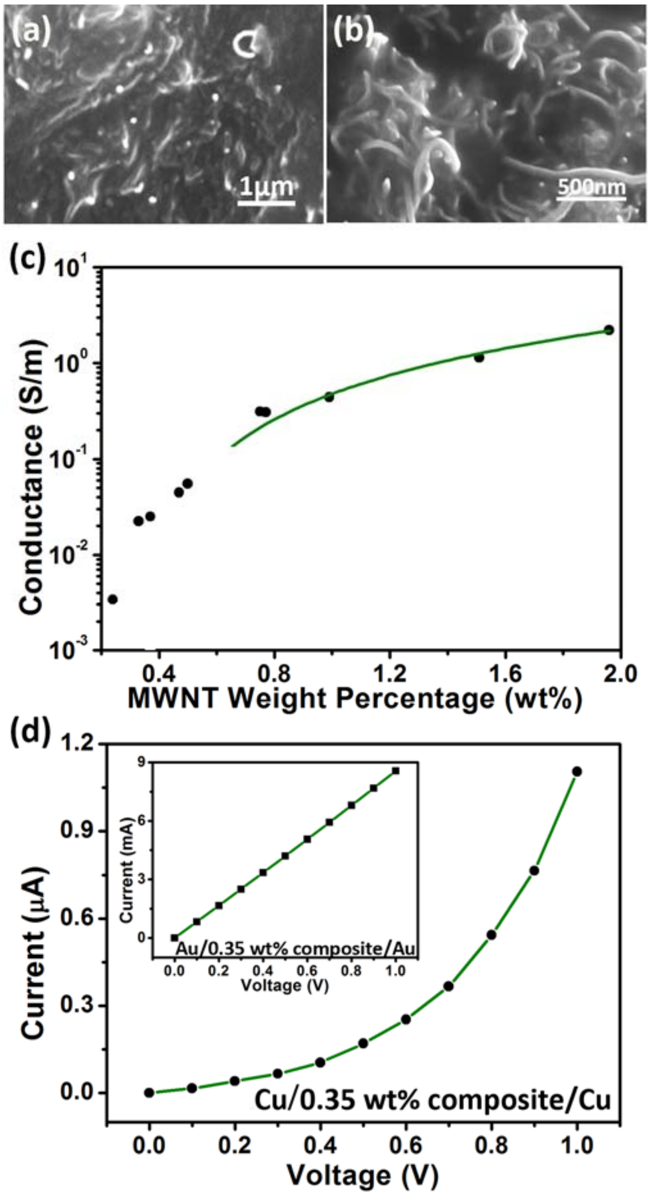

Fig. 1 The SEM images of the fractured surface of the 0.35 wt % (a) and 2 wt % (b) MWNT loading composites. (c) Electrical conductivities of the composites as a function of MWNT loadings. (d) *I-V* characteristics of the 0.35 wt % composite with copper plates (the main panel) and gold plates (the inset) as electrodes.

The SEM images (Fig. 1a and 1b) showed a top view of the 0.35 wt % and 2 wt % MWNT-PDMS composites and revealed that the MWNTs were randomly and well dispersed in the PDMS matrix. The bright filaments were MWNTs. The electrical conductivity measurements were made using the standard four-probe method with a Keithley 2410 source meter at room temperature. The conductivity can be improved by several orders of magnitude with a little increase of the MWNT loading. The dependence of conductivity versus MWNT loading demonstrated a percolation behavior[27], and the percolation threshold was about 0.35 wt % for our composites (Fig. 1c). The contact type (Schottky or Ohmic) of an M/S junction is generally determined by traditional *I-V* tests for bulk semiconductor. Fig. 1d shows the *I-V* curves of the Cu/0.35 wt % composite/Cu sample and gold/0.35 wt % composite/gold (Au) sample. *I-V* curve shows an excellent Ohmic contact in the Au/0.35 wt % composite/Au sample (the inset), while a Schottky contact is found in the Cu/0.35 wt % composite/Cu sample (the main panel).

Above result can be well explained with the band gap model. The work function of Au, Cu and P-type CNT are 5.1 eV, 4.6 eV and 4.6-4.9 eV[28] respectively. The Fermi level of Cu possibly lies in the gap of the composite, hence, a Schottky junction with a built-in potential which is favorable for charge carrier separation may be generated between the composite and Cu. Whereas, the work function of Au is much larger than that of the P-type CNT. Thus, the Fermi level of Au lies under the top of composite valence band, forming an Ohmic contact.

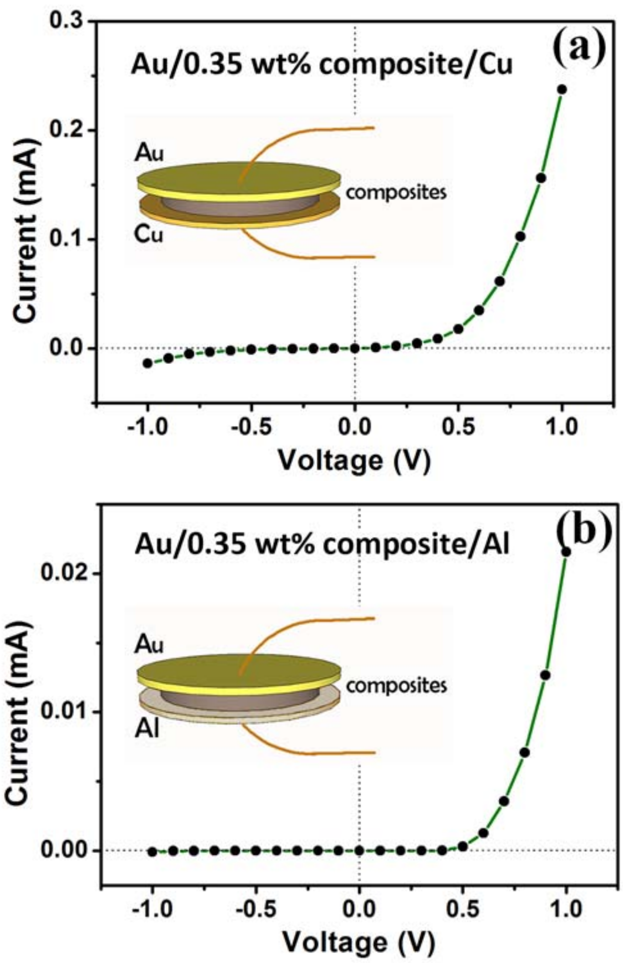

Fig. 2 The *I-V* curves of Au/0.35 wt % composite/Cu (a) and Au/0.35 wt % composite/Al (b) Schottky diodes. Inset: Sketch of the Schottky diode.

The two different contact modes of the Au/composite sample and the Cu/composite sample enlighten us on using Cu as Schottky contact electrode and Au as Ohmic contact electrode to prepare a Schottky diode. The inset of Fig. 2 shows the structure of the Schottky diode. The *I-V* curve of the Au/0.35 wt % composite/Cu sample displays the characteristics of a typical M/S Schottky rectification effect (Fig. 2a). Based on Bethe's thermionic-emission theory[29,30], the relationship between current and voltage is fitted with the formula,

$$I = AA^{*}T^{2}\exp\left(-\frac{\Phi_b}{kT}\right)\left[\exp\frac{e(V-IR_s)}{nkT}-1\right]. \qquad (1)$$

Here, A is the contact area, A^* the Richardson constant, T the absolute temperature, e the electron charge, k the Boltzman constant, n the ideality factor, R_s the series resistance and Φ_b the SBH. Fitting the *I-V* curve with the formula, the SBH of the Schottky diode could be obtained. With this method, we calculated the SBH of the Au/0.35 wt % composite/Cu Schottky diode and got a value of 0.783 eV. By replacing the Cu electrode with aluminium (Al) from the Schottky diode mentioned above, a more significant Schottky rectification effect was generated (Fig. 2b). The contact in Al/0.35 wt % composite junction possesses a higher SBH of 0.95 eV. These results confirm that, for P-type semiconductors, the lower the work function of the contact electrode, the higher the formed SBH. Some SBHs of previously published metal/composite junctions with the similar structure are listed in Table S1 in the supplementary information. Based on the results in Table S1, the SBH of the Al/0.35 wt % composite junction in our work is much larger than that of most previously reported literatures regarding the similar structure, even their substrates are conductive polymer materials and their fillers are single wall CNTs (SWNTs) or few wall CNTs (FWNTs).

CG VRH model can perfectly explain the semiconductor behaviors of the composites. It is well-known that the movement of charge carriers is confined along the tube axis of a CNT. On the basis of percolation theory, the MWNTs form a 3-dimentional conductive network in the MWNT-PDMS composite. So when a carrier transports from one MWNT to the neighboring one, it will get through MWNT/PDMS interfaces and a thin PDMS layer, which means a lot of nanometer-sized barriers exist in the carrier transport path. In this case, the Coulomb charging effect is generated and becomes remarkable, thus opens the Coulomb band gap at the Fermi level. The values of the composite Coulomb band gaps can be determined through this model. Fig. 3a shows that the composite Coulomb band gap increases with decreasing MWNT loading at room temperature, the greatest value is 0.36 eV for the 0.35 wt % composite. We further analyzed the relationship between the MWNT loading of the composite and the formed SBH of the Cu/composite contact. Different MWNT-loading Schottky diodes using Au and Cu as contact electrodes were fabricated. The SBHs of the Schottky diodes (in the range of 0.35-2 wt %) were calculated with the method above-mentioned. The dependence of SBH versus MWNT loading is shown in Fig. 3b. With the MWNT loading increasing from 0.35 wt % to 2 wt %, the SBH decreases monotonically from 0.783 eV to 0.632 eV. The highest value is 0.783 eV at 0.35 wt % composite (the corresponding ideality factor is 2.0), indicating that the highest SBH appears at the percolation threshold. It is evident from Fig.3 that the changing trend of variations in the Cu/composite SBHs with MWNT loadings is the same as that of the Coulomb band gaps. The trend remains the same for repeat measurements. That is, the Cu/composite SBHs and Coulomb band gaps of the composites depend largely on the MWNT loading and can be easily tuned by the mass fraction of MWNTs.

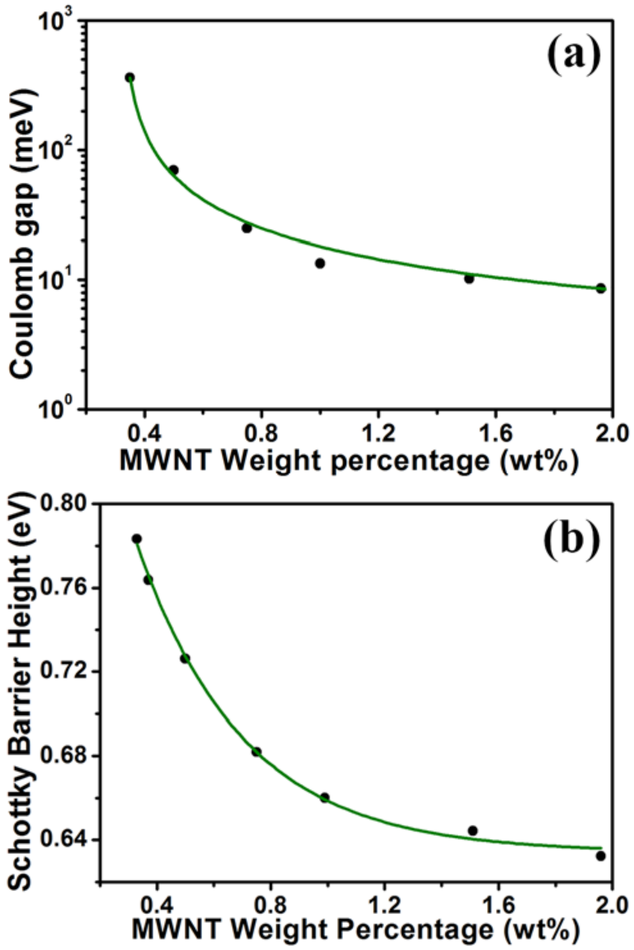

Fig. 3 Coulomb band gaps (a) and SBHs (b) of the composites versus the mass fraction of MWNTs.

As discussed above, the 0.35 wt % composite displays a 0.36 eV Coulomb band gap and a 0.783 eV SBH contacted with Cu at room temperature, which may be verified by the Schottky junction photovoltaic effect. Photovoltaic parameters, such as open-circuit voltage (V_{oc}), short-circuit current density (J_{sc}) and fill factor (*FF*), can be estimated theoretically through the general method mentioned in Jenny Nelson's book[31]. The V_{oc} can be obtained as

$$eV_{oc} = E_{HOMO(D)} - E_{LUMO(A)} - 0.5E_B \, . \qquad (2)$$

Here, e is the electron charge, $E_{HOMO(D)}$ is the highest occupied molecular orbital energy level of the donor material (the composite), $E_{LUMO(A)}$ is the lowest unoccupied molecular orbital energy level of the acceptor material (Cu), E_B is the exciton binding energy of the donor material. In the energy band theory[30], $E_{HOMO(D)}$ satisfies the equation $E_{HOMO(D)}= E_{LUMO(D)} - E_{tran}$, in which E_{tran} is the Coulomb band gap (E_{CG}=0.36 eV). For Cu electrode, the acceptor material, $E_{LUMO(A)}$ is the Fermi level (E_F) of the metal. In view of the Schottky's law, the SBH (Φ_b) of the Cu/composite contact can be expressed as $\Phi_b=E_{LUMO(D)} - E_F$. Thus, Eq. 2 can be simplified into

$$eV_{oc} = \Phi_b - E_{CG} - 0.5E_B \, . \qquad (3)$$

The mathematical expression of E_B is $E_B=E_{HOMO(D)} - E_{LUMO(D)} - E_{opt}$. E_{opt} is the optical band gap of the composite and is determined by the optical absorption edge of the composite. Since CNTs have a strong absorption of infrared light, there does not exist a sharp optical absorption edge in the spectrum of the composites, so it's hard to get the exact value of E_{opt}. Thus, figuring out the accurate theoretical value of V_{oc} is almost impossible. However, the value of the E_{opt} is quiet certain for a specific composite. For this reason, we fabricated two kinds of samples with the same 0.35 wt % composite, the Cu sample and the Al sample, for photovoltaic testing. The V_{oc} of the Cu sample, recorded as V_{ocCu}, meets the equation $eV_{ocCu}=\Phi_{bCu} - E_{CG} - 0.5E_B$, while the V_{oc} of the Al sample, V_{ocAl}, satisfies the similar equation $eV_{ocAl}=\Phi_{bAl} - E_{CG} - 0.5E_B$. Subtracting the two equations, the following equality is tenable.

$$e(V_{ocAl} - V_{ocCu}) = \Phi_{bAl} - \Phi_{bCu} \qquad (4)$$

Here, $\Phi_{bAl} - \Phi_{bCu}$ =0.95 eV – 0.783 eV=0.167 eV. That is, if the measurements of V_{ocCu} and V_{ocAl} make the value of $V_{ocAl} - V_{ocCu}$ approximately equal to 0.167 V, the aforementioned discussions on the metal/composite Schottky contact and the photovoltaic properties of the MWNT-PDMS composite should be credible.

The cross-sectional diagram in Fig. 4a shows the configuration of the photovoltaic samples, whose anode material is SACNT film, photosensitive material is the 0.35 wt % composite with a thickness around the micron order and an active area of 0.5×0.5 cm^2, cathode material is Cu and Al respectively. The samples were tested under dark and illuminated conditions at room temperature. From the *I-V* curves showed in Fig. 4b and 4c, we can see the samples perform a diode behavior in the dark and a photovoltaic behavior in the sunlight. For the Cu sample, we obtained V_{ocCu}=0.237 V and *FF*=0.42; for the Al sample, we obtained V_{ocAl}=0.398 V and *FF*=0.47. The photovoltaic performance of the Al sample is evidently superior to that of the Cu sample. The J_{sc} in the test, which might be severely influenced by the device assembling parameters such as excessive thickness of the samples, is not the concern point of this study. Our concern is on the V_{oc} values. The V_{oc} value difference for the two samples, i.e., $V_{ocAl} - V_{ocCu}$, is 0.161 V. Thus, $e(V_{ocAl} - V_{ocCu})$=0.161 eV. This value basically equals to the difference between the SBHs of the Cu sample and the Al sample, 0.167 eV. These measurements confirm that the above discussions about the metal/composite Schottky contact are reliable.

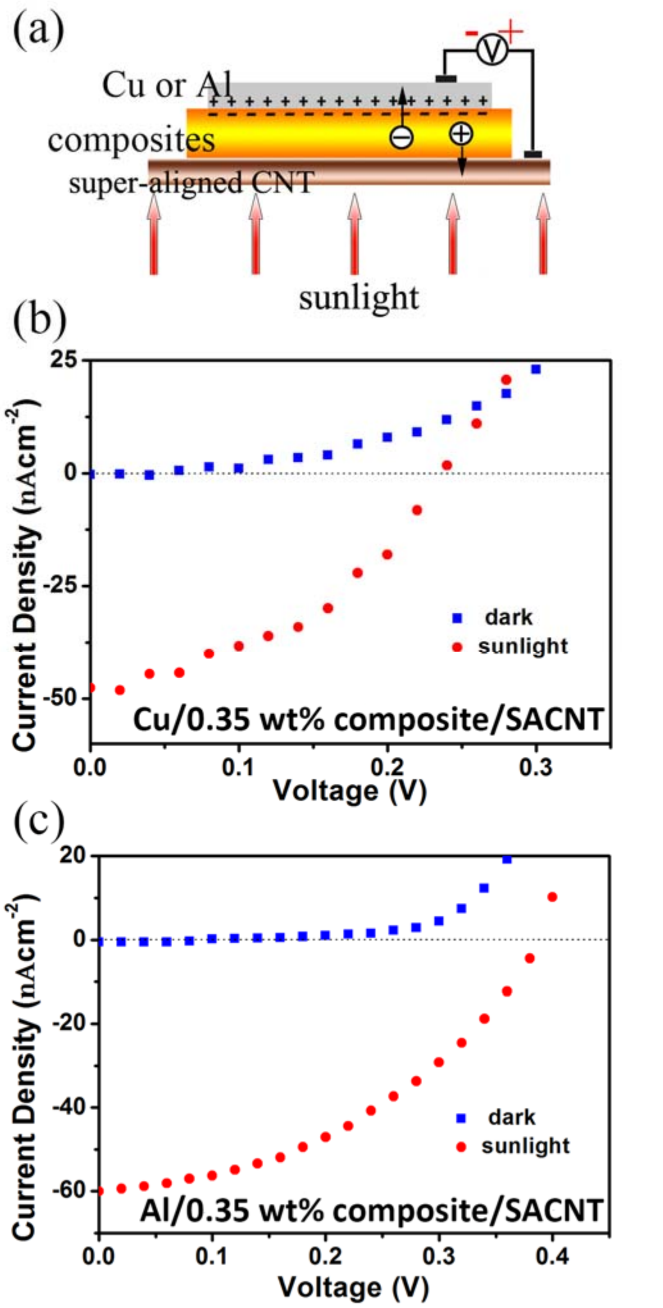

Fig. 4 (a) Cross-sectional diagram of the photovoltaic samples. *I-V* curves of the Cu/0.35 wt % composite/SACNT (b) sample and the Al/0.35 wt % composite/SACNT (c) sample under dark and illuminated conditions.

We tested the durability of the Au/0.35 wt % composite/Cu Schottky diode at room temperature over 12 months. The conductivity and SBH of the diode were measured with the aforementioned method after storing it in the dark. Fig. 5 shows the conductivity and SBH as a function of duration for this stability test. Changing trend of the conductivity is similar to that of the SBH. The conductivity of the composite decreased from 0.0225 S/m to 0.0164 S/m in the first three months, but became stabilized afterwards. The SBH of the diode presented a slight decrease at the beginning and then remained almost constant. After a year, the Schottky diode retained 98 % of the original SBH. These results imply that the Schottky diode can be employed continuously without performance degradations for an extended period.

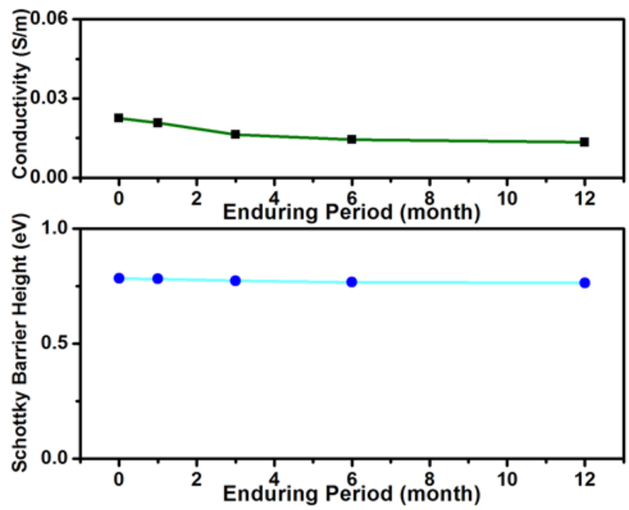

Fig. 5 Evaluation of stability over 12 months for the Au/0.35 wt % composite/Cu Schottky diode at ambient temperature: conductivity and Schottky barrier height.

Conclusions

In summary, we presented a flexible artificial polymer semiconductor composed of PDMS and MWNTs, and provided an overall research on the contacts in the metal/composite junctions for the first time. Different types of contacts can be formed in the junctions of the composite and different metals. The contact properties have a close relationship to the work function of the metals and the MWNT loadings of the composites. The SBHs of the Schottky diodes using Au and Cu as contact electrodes were calculated. The values of the SBHs exceed 0.783 eV for the 0.35 wt % composite and can be easily tuned by the MWNT loadings. The reliability of the aforementioned investigation has been verified by the photovoltaic tests based on the Schottky junctions of 0.35 wt % composite and low work function metals (Cu and Al). The stability test indicates that the performance of the flexible composites will remain stable for a long-term duration. These results suggest that insulating polymers with a proper MWNT loading may provide an optional choice for constructing flexible, low costing and easy tunable Schottky diode-based devices in the future.

Acknowledgements

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Table of contents entry

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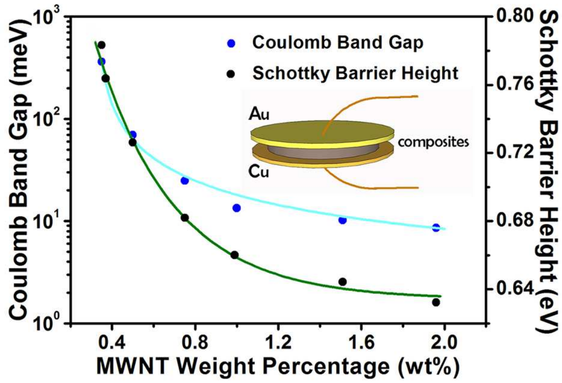

Text:

The Coulomb band gap and Schottky barrier height of the MWNT-PDMS composite can be tuned by the MWNT loading, leading to performance variation on flexible electronics.