

Cite this: *Phys. Chem. Chem. Phys.*, 2012, **14**, 8397–8402

www.rsc.org/pccp

PAPER

Charge dynamics in solar cells with a blend of π -conjugated polymer-fullerene studied by transient photo-generated voltage†

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Received 22nd March 2012, Accepted 17th April 2012

DOI: 10.1039/c2cp40911a

The biphasic feature of transient photo-generated voltage (TPV) is investigated in organic solar cells (OSCs) with a blend active layer of poly(3-hexylthiophene) (P3HT) and phenyl C61 butyric acid methyl ester (PCBM). The positive and negative components in biphasic TPV are explained through PCBM only and P3HT only devices. The negative and positive components are ascribed to the dipole formation at the buried interface of P3HT/indium tin oxide (ITO) and PCBM/ITO respectively. Based on these findings, two fundamental phenomena are revealed as follows: (1) interfacial modification on the buried interface inverts the negative component in biphasic TPV to a positive component, which prevents the leakage current channel in the conventional OSC structure; and (2) the solvent chosen transforms the positive component in biphasic TPV into a negative signal, which blocks the leakage current channel in the inverted OSC structure. Consequently, the study of TPV polarity provides the justification of the interaction at the buried interface. Besides, the decay of TPV is found to be bi-exponential, which can be used as a tool to estimate the degree of charge balance in OSCs.

1 Introduction

The advantages of polymers, namely a wide variety of choices in materials, simple fabrication processes, low cost and compatible with flexible substrates, have allowed the development of organic solar cells (OSCs) to boom in recent years.^{1–5} OSC performances are closely correlated with the four-step charge dynamics including exciton formation through light absorption, charge separation, charge transport and charge collection by electrodes. Various approaches have been explored to optimize each step, for instance (1) synthesizing low band gap polymers to enhance light absorption,^{3,6–8} (2) fabricating bulk heterojunction structure and intermixing electron donors and acceptors to improve the efficiency of charge separation,^{4,5,9} (3) annealing in various solvent vapors to form a phase separation that balances bipolar transport¹⁰ and modifying the surface of the electrode to strengthen charge collection at electrodes.^{11,12} Considering the great significance of the charge dynamics, an effective characterization concurrently indicative of the four steps is highly desirable.

Distinctly different from depositing small molecules by using a thermal evaporation method, the polymer layer formation relies

on printing technology,¹¹ spin coating⁵ or dip coating.¹³ The thickness of the polymer layer is therefore difficult to be finely controlled, which makes the conventional surface analysis techniques, such as ultraviolet photoemission spectroscopy,^{14,15} invalid in the analysis of the buried interface between the polymer layer and the electrode on a substrate. While the buried interface plays a crucial role in charge transport and collection, characterization of the buried interface has proved to be difficult.

Recently, transient photo-generated voltage (TPV) has been reported to be closely related to the four steps in small molecule based organic optoelectronic devices.^{16,17} In this paper, TPV is introduced to study the charge dynamics in the polymer devices. By studying changes of TPV polarity and decaying rate in different polymer devices, the interaction at the buried interface of the blend of poly(3-hexylthiophene) (P3HT):[6,6]-phenyl-C61-butyric acid methylester (PCBM)/indium tin oxide (ITO) and solvent effects on charge balance are revealed. Our study shows that TPV can function as a potential tool to characterize the performance of polymer based optoelectronic devices.

2 Results and discussions

2.1 Biphasic feature of TPV

The polymers of our studied devices mainly are P3HT and PCBM because they are robust and representative materials of OSCs.

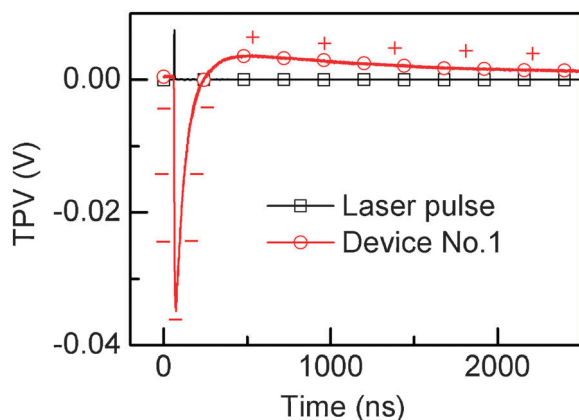
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† Electronic supplementary information (ESI) available. See DOI: 10.1039/c2cp40911a

Table 1 Summary of the device structures

Device no	Structure	Solvent
1	ITO/P3HT:PCBM/Al	CB
2	ITO/PCBM/Al	CB
3	ITO/P3HT/Al	CB
4	ITO/PEDOT:PSS/P3HT:PCBM/Al	CB
5	ITO/P3HT:PCBM/Al	DCB
6	ITO/PEDOT:PSS/P3HT:PCBM/Al	DCB
7	ITO/Ca/P3HT:PCBM/Ca/Al	CB
8	ITO/PEDOT:PSS/P3HT:PCBM/Au/Al	CB
9	ITO/Ca/P3HT:PCBM/Ca/Al	DCB
10	ITO/PEDOT:PSS/P3HT:PCBM/Au/Al	DCB

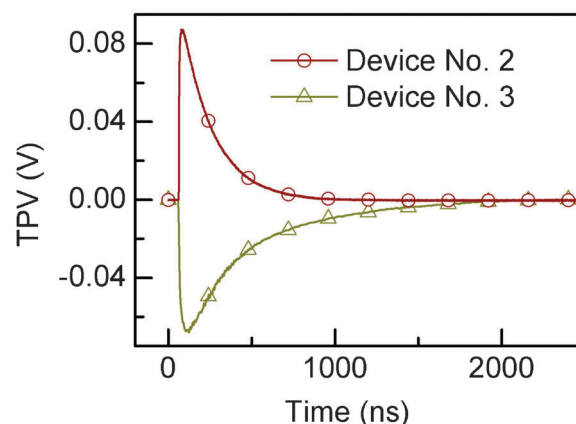
**Fig. 1** Transient photo-generated voltage of Device 1 and transient spectrum of laser pulse.

In the bulk heterojunction of the P3HT:PCBM blend polymer, the active layer consists of an interpenetrative network of P3HT and PCBM which work as an electron donor and acceptor respectively.^{10,18} Ten devices have been designed to study the effect of structures on TPV features. Their structures are listed in Table 1. Other experimental conditions can be found in the experimental section.

Fig. 1 shows the TPV of Device 1 (control device) in Table 1. The laser pulse used as shown in Fig. 1 has a very narrow width of ~ 1 ns. TPV is established once the sample is excited by the laser pulse. For TPV polarity, it consists of positive and negative components (*i.e.* biphasic feature). The negative component initially dominates the TPV signal in the first 240 ns, while the later response of TPV is positive. TPV polarity depends on the internal built-in electric field ($E_{\text{built-in}}$) crossing the active layer. In conventional sandwiched structure, the work function of the two electrodes controls the $E_{\text{built-in}}$ direction, which points from the electrode with low work function to the electrode with high work function. Free charges will move along the direction of $E_{\text{built-in}}$. Generally, TPV should have only one polarity for single polymer based devices. However, the active layer of Device 1 is a P3HT:PCBM blend polymer. The biphasic feature of TPV for Device 1 will depend on the special constituent of the blend. To examine the reasons, PCBM only and P3HT only devices labeled as Devices 2 and 3 are fabricated respectively.

2.2 Separation of negative and positive components from biphasic TPV

Fig. 2 depicts TPVs of Devices 2 and 3. As expected, TPVs of Device 2 or 3 are unipolar. For Device 2, TPV is fully positive,

**Fig. 2** Transient photo-generated voltage of Devices 2 and 3.

while that for Device 3 is negative. Work functions of bare ITO and Al are 4.6 eV¹⁹ and 4.3 eV²⁰ respectively. $E_{\text{built-in}}$ points from Al to ITO in the normal case²¹ and drives electrons and holes to move towards the Al cathode and the ITO anode respectively. The positive TPV signal obtained is regarded as the “Normal Case”, while the negative TPV, whenever happens is considered as the “Abnormal Case”. It has been reported that the interaction between the electrode and the active layer can modify the work function of electrodes. If it is the case in Device 3, the $E_{\text{built-in}}$ direction can be reversed to yield a negative TPV as shown in Fig. 2. Back to the biphasic feature, if P3HT and PCBM coexist at the interface of Device 1, where the dipole forms at electrode/P3HT and electrode/PCBM, the contact of the active layer and electrode will generate two conducting channels, in which P3HT and PCBM contribute to the positive and negative components as shown in Fig. 1. It should be noted that the negative component implies that the channel of the P3HT phase leads to the current leakage in conventional normal structure, which is detrimental for performance improvement.

2.3 Inverting the negative component in biphasic TPV to a positive component by surface modification

In this section, we will investigate the effect of the buffer layer on the dipole-formation interface for eliminating the negative TPV component and the leakage current channel. In Device 1, there are two interfaces, namely, the buried interface and the top interface. The buried interface is between ITO and the active layer, and the top interface is between the active layer and Al. Poly(3,4-ethylenedioxythiophene) poly(styrenesulfonate) (PEDOT:PSS) is chosen as the buffer layer depositing on the surface of ITO. The structure is listed as Device 4 in Table 1. The influence of interfacial modifications on TPV polarity is shown in Fig. 3. The insertion of PEDOT:PSS makes the negative component become positive with the trend marked by an arrow. In contrast, the positive component in Device 1 is almost unaffected.

PEDOT:PSS has been reported to provide the high work function of 5.2 eV and smoothen the surface of ITO.⁴ From our results, another role of PEDOT:PSS can be revealed from the data in Fig. 3. PEDOT:PSS successfully removes the negative component through preventing the contact between ITO and P3HT.

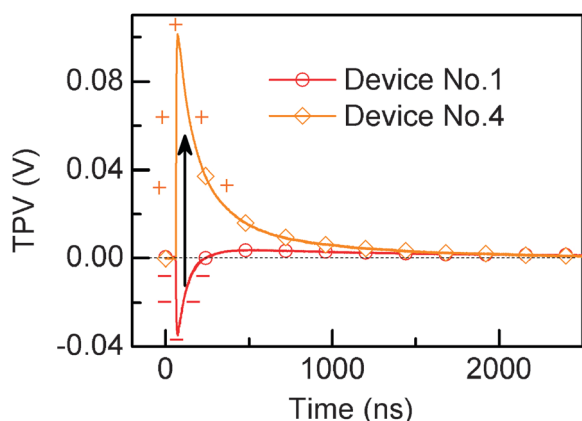


Fig. 3 Transient photo-generated voltage of Devices 1 and 4.

So the interaction at P3HT/ITO can be eliminated. In conjunction with the higher work function PEDOT:PSS, $E_{\text{built-in}}$ crossing the P3HT network is analogous to the “Normal Case”, namely pointing from the cathode to the anode. Consequently, by proper surface modification, the elimination of the negative TPV component implies that the leakage current channel generated at the interaction between P3HT and ITO can be removed. Meanwhile, based on the results of the above three sections, it is confirmed that the origin of the biphasic feature of TPV in Device 1 is the coexistence of P3HT and PCBM at the buried interface.

2.4 Inverting the positive component in biphasic TPV to a negative component by solvent chosen

Even the negative component is diminished by the insertion of PEDOT:PSS, which is good for improving the performance of normal device structure. However, a drawback of this normal device structure is that the ITO/PEDOT:PSS interface is not stable, which leads to the degradation of OSC.^{22,23} One way to solve this drawback is to use inverted OSC structure. In the inverted OSC structure, fully negative TPV is obtained at the buried interface. From our previous results, it is clear that the positive component is from the component of PCBM at the buried interface. To eliminate the positive component, the proportion of PCBM should be as low as possible at the buried interface. We find an interesting phenomenon in fabricating Device 2. Solutions of 20 mg ml⁻¹ PCBM were prepared with two solvents, namely chlorobenzene (CB) or dichlorobenzene (DCB). After stirring for 24 h, we spin coated the two solutions separately on the two pre-cleaned ITO samples. It could easily form the uniform film for the CB solution by spin coating it at the rate of 650 rpm for 30 s. But for DCB solution, even when the spinning speed was tuned in a wide range from 2000 rpm to 400 rpm, the film was not formed yet. The result indicates that for the ITO substrate, PCBM in DCB is less adhesive than that in CB solution. The finding enlightens us that the proper choice of solvents can tune the constituent at the buried interface. We therefore fabricate additional Device 5. The only difference in fabricating Devices 1 and 5 is the solvent.

Fig. 4 shows their TPVs. Unlike the biphasic TPV feature of Device 1, fully negative TPV is observed. The disappearance of

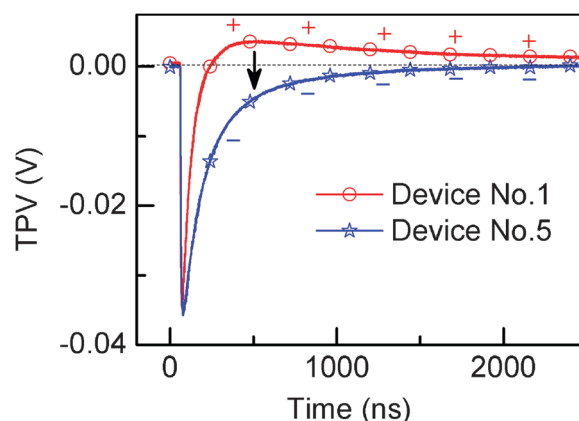


Fig. 4 Transient photo-generated voltage of Devices 1 and 5.

the positive component from biphasic TPV demonstrates the obvious reduction in the PCBM content, and P3HT dominates the composition at the buried interface in Device 5. Meanwhile, the result further confirms that the biphasic TPV feature for Device 1 originates from the coexistence of P3HT and PCBM at the buried interface. The change of TPV polarity in Fig. 4 represents that the constituent at the buried interface can be modulated by proper selection of solvents. Based on the finding in Fig. 4, we have successfully fabricated the inverted OSC, with the simple structure of ITO/P3HT:PCBM (DCB)/MoO₃/Ag. The power conversion efficiency reaches up to 2.5%, while for the CB solvent, the efficiency is around 1.5%. The difference in performance between two devices illuminates that choosing an appropriate solvent for certain structures is critical in achieving high performance. Certainly, the solvent chosen can also cause other changes of device structure, such as morphology. The work here aims at revealing the effect of solvent chosen on the buried interface by using a TPV method since the detailed characterization of buried interface is full of challenge.

Besides that the TPV polarity characterizes the buried interface, the decaying part of TPV is also sensitive to the charge transport in the bulk of the active layer. Device 6 with 20 nm PEDOT:PSS inserted at the buried interface in Device 5 was fabricated. Fig. 5a presents the TPVs for Devices 4 and 5. TPV amplitudes of both devices are almost the same. The establishment of TPV is on the time scale of <1 ns which is consistent with that of charge separation as reported in ref. 24. Therefore, the TPV amplitude can reflect the efficiency of charge separation. The comparable amplitudes of the two devices indicate that the efficiency of charge separation in the bulk is almost unaffected by the chosen solvent of CB or DCB. This is because that no matter spin coated from CB solution or from DCB solution, the donor/acceptor system in the bulk heterojunction structure can efficiently dissociate excitons. As for the decaying part, the decaying rate for Device 6 is greatly different from that for Device 4. In the recent studies on transient photocurrent, Heeger's group suggested that the decaying part could be concurrently dominated by mobilities of electrons and holes.²⁵ The initial decay arises from the faster carrier and the later signal is from the slower carrier. To compare decaying responses in our results, TPVs of both devices are normalized and plotted in Fig. 5b.

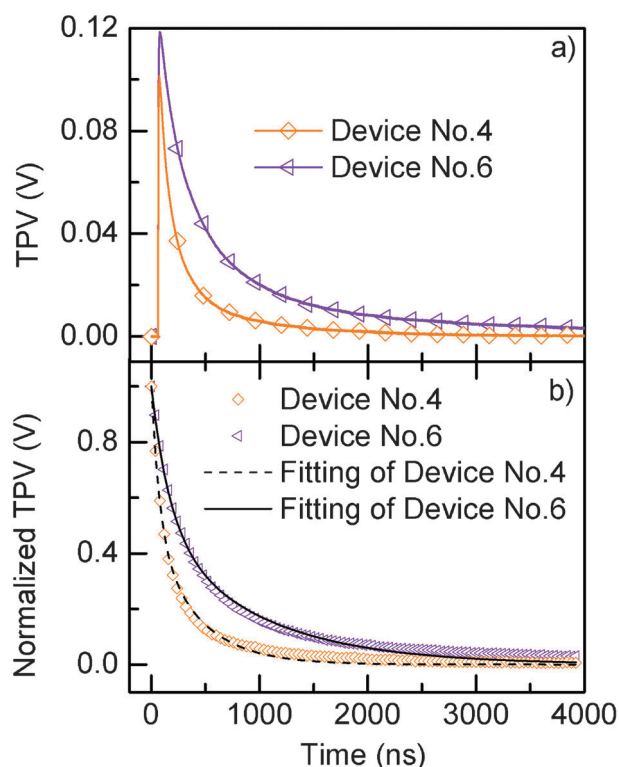


Fig. 5 (a) Transient photo-generated voltage of Devices 4 and 6. (b) Theoretical fitting on normalized transient photo-generated voltage of Devices 4 and 6.

Both the initial decaying and the later response are different for the two devices. In this work, because the decaying response is related to the mobilities of two kinds of carriers, we use a bi-exponential function to fit the transient signal.

$$N(t) = N_1 \exp(-t/\tau_{\text{fast}}) + N_2 \exp(-t/\tau_{\text{slow}}), \quad (1)$$

where $N(t)$ is the charge density in time t after excitation, N_1 and N_2 are the charge densities of two kinds of carriers at $t = 0$. According to the reported theory, τ_{fast} and τ_{slow} are closely related to the mobilities of the fast carrier and the slow carrier.

Therefore, the ratio of $\tau_{\text{fast}}/\tau_{\text{slow}}$ qualitatively reflects the degree of charge balance (mobility balance). For the unbalanced OSC, the photocurrent is space-charge-limited. The fill factor will be low, which diminishes the power conversion efficiency. As a result, charge balance plays a key role in the performance of solar cells. N_1 and N_2 are set to be equal to 0.5. The simplification arises from an ideal assumption that the generated electrons and holes after the exciton dissociation are equivalent. Meanwhile electrons and holes only transport in PCBM and P3HT phases respectively.

Fig. 5b shows the fitting results obtained by using eqn (1). The ratio of $\tau_{\text{fast}}/\tau_{\text{slow}}$ for Device 4 is 10, while for Device 6 the ratio decreases to 3. The optimal balanced solar cell should have to a ratio of one, i.e. $\tau_{\text{fast}} = \tau_{\text{slow}}$. Therefore, Device 6 is more balanced since its ratio of $\tau_{\text{fast}}/\tau_{\text{slow}}$ is closer to one. Noticeably, the bi-exponential decay fitting in the main text is just a simplified calculation to qualitatively reveal the balance ratio between electron and hole transport, which cannot be used to reveal the exact value of charge mobility, because TPV

depends on several photo-physical processes in many solid environments and not only for electron-transfer reactions. A more exact model based on the current steady photon-to-carrier conversion is needed which is discussed in detail in ESI.†

In order to quantitatively confirm that the decaying rate of TPV can characterize the charge balance, we fabricate four single-carrier-dominated devices listed in Table 1, from Device 7 to Device 10, including hole- and electron-dominated devices. Devices 7 and 9 are electron-dominated devices. The Ca on ITO electrode has been demonstrated to modify the work function of ITO for electron extraction.^{26,27}

Injections of holes from both electrodes are blocked due to the high energy barrier caused by low work functions of the two electrodes. The charges in the blend layer are therefore electron-dominated. Devices 8 and 10 are hole-dominated devices. High work functions of PEDOT:PSS(5.2 eV) and Au (4.9 eV)¹² make the injections of electrons from both electrodes blocked due to the high energy barrier. The majority in the blend layer are therefore holes. Fig. 6a shows the absolute $J-V$ curve from -3 V to $+3$ V of the electron-dominated device. The symmetric $J-V$ indicates that work functions at the two electrodes of ITO/Ca and Ca/Al are the same. To study the electron or hole mobility, the range of forward bias from 0 to 3 V is plotted. The right inset of Fig. 5a shows the $\log(J)$ versus $\log(V)$ curve. The current density depends quadratically on the voltage. This behavior is the characteristic of space charge limited current (SCLC).^{28,29} Fig. 6b and c, respectively, show the fitting results of Devices 7 and 8. The simulation process is also the same for the DCB devices. The calculated mobilities of four devices are summarized in Fig. 7. For the CB devices, electron and hole mobilities are $4.1 \times 10^{-3} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ and $3.8 \times 10^{-5} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ respectively. We also cite the data measured by time of flight taken from ref. 18 in Fig. 7 denoted as solid circle dots. The result estimated by SCLC is consistent with that from time of flight methods. The ratio of $\mu_{\text{fast}}/\mu_{\text{slow}}$ is 110. While for the DCB devices, electron and hole mobilities are $4.6 \times 10^{-4} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ and $5.0 \times 10^{-4} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ respectively. The ratio of $\mu_{\text{fast}}/\mu_{\text{slow}}$ is 1.1. Such balanced charge transport has been confirmed to contribute to well-documented device performance.¹⁰ The balance ratio obtained from TPV measurement in Fig. 5b also shows that Device 6 is more charge balanced than Device 4. Therefore the conclusions from TPV and SCLC measurements coincide with each other.

It should be noted that the value of the ratio from the two methods cannot exactly match each other because (1) although τ is found to be in direct proportion to μ , the quantitative relationship between τ and μ needs further study; (2) in the real case, a small amount of electrons can migrate to the P3HT network from PCBM, and holes to the PCBM phase from P3HT. N_1 and N_2 in eqn (1) are not equal to each other in the real case. To get the exact fitting, the above two problems are required to be considered in future.

Consequently, our results show that there are two effects of solvents on the devices: (1) to change the composition near the buried interface, and (2) to tune the charge mobility in the bulk. The decaying rate of TPV is therefore closely related to charge transport in a charge dynamic process.

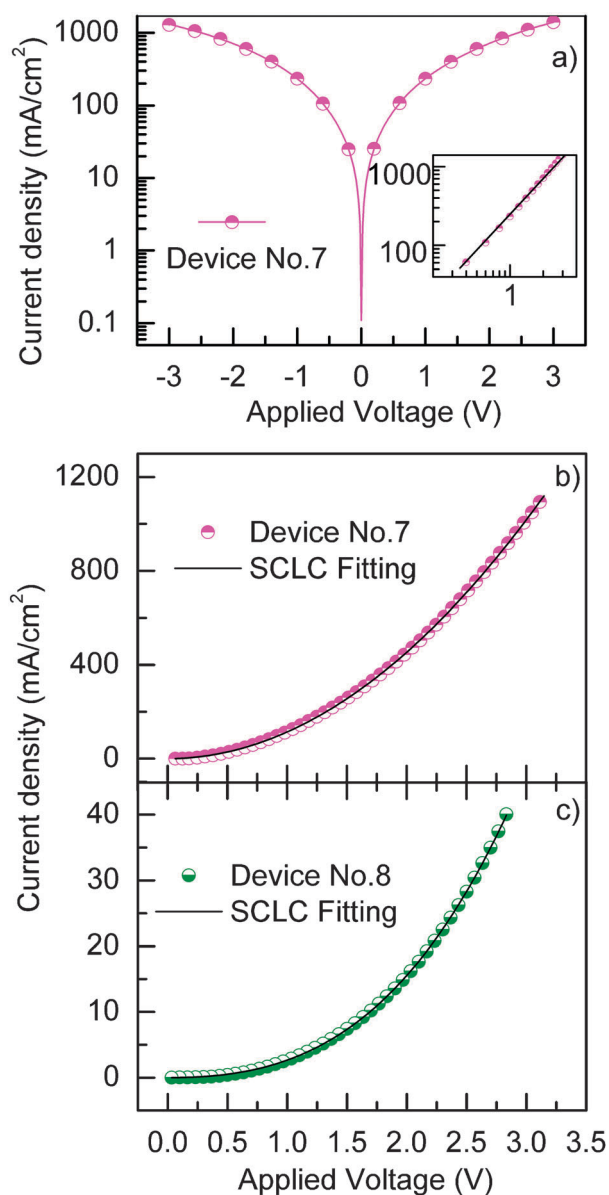


Fig. 6 Mobility measurement by the space-charge-limited-current method. (a) J - V curve of Device 7. The inset plots $\log(J)$ - $\log(V)$ curve from 0 V to 3 V. (b) Fitting to J - V curve of Device 7 from 0 V to 3 V based on a space-charge-limited current method. (c) Fitting to J - V curve of Device 8.

3 Conclusion

In this work, biphasic TPV consisting of both negative and positive components is observed in P3HT:PCBM blend layer based devices. The negative and positive components are studied through P3HT-only and PCBM-only devices. Our results show that the negative and positive components originate from the contact between ITO/P3HT and ITO/PCBM, respectively, at the buried interface. Importantly, the interaction between P3HT and ITO makes the P3HT network function as a leakage current channel in OSCs. By using methods of surface modification and solvent selection, the negative component and the positive component can be converted to each other. For surface modification, the

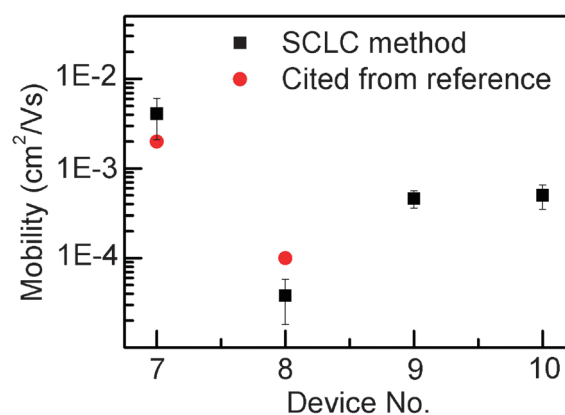


Fig. 7 A summary of electron and hole mobilities for CB and DCB devices estimated by an SCLC method. Solid circle dots are cited from ref. 19.

insertion of PEDOT:PSS at the buried interface can prevent the dipole formation between P3HT and ITO. While for solvent effects, by substituting the CB solvent with the DCB solvent, the content of PCBM at the buried interface is decreased, thus the positive component is transformed to a negative component. Besides the influence of solvent on the constituent of the buried interface, the bi-exponential decaying response of TPV also reveals that an appropriate solvent can balance the charge transport. These results have important implications for the strategies to use TPV as a tool to optimize and characterize OSC performances.

4 Experimental

4.1 Sample preparation

The ITO glass substrates with sheet resistance of 15 ohms per square were cleaned and then treated in a UV-Ozone cleaner for 15 min in an ambient atmosphere under room temperature. The substrates were then transferred into a glove box filled with nitrogen. For a blend of P3HT and PCBM, the solution is prepared by mixing P3HT and PCBM in the CB or DCB solvent with weight ratio of 1 : 1 and density of 20 mg ml⁻¹ for each material. The solution was then stirred for 18 h to dissolve the polymers homogeneously. Polymer films were spin-coated onto glass substrates at a spin rate of 650 rpm for 30 s from the solution under an N₂ atmosphere. Before annealing at 110 °C for 10 minutes on a hot plate, slow growth was employed. The Al cathode was finally thermally evaporated with a device area of 5.77 mm². For the blend active layer, the thickness was about 200–220 nm. For studying solvent effects of CB and DCB, the conditions of fabricating the polymer layer were the same except the solvents.

4.2 Characterization

The schematic diagram of TPV measurement for OSC is shown in Fig. 8(a). In this work, we use the common donor P3HT and acceptor PCBM. For the structure of OSC, there are typically two interfaces, namely the top interface, and the buried interface as shown in Fig. 8(a). The buried interface is defined as the ITO/polymer layer, while the top interface is polymer layer/Al. Laser pulse irradiates the sample from the ITO side.

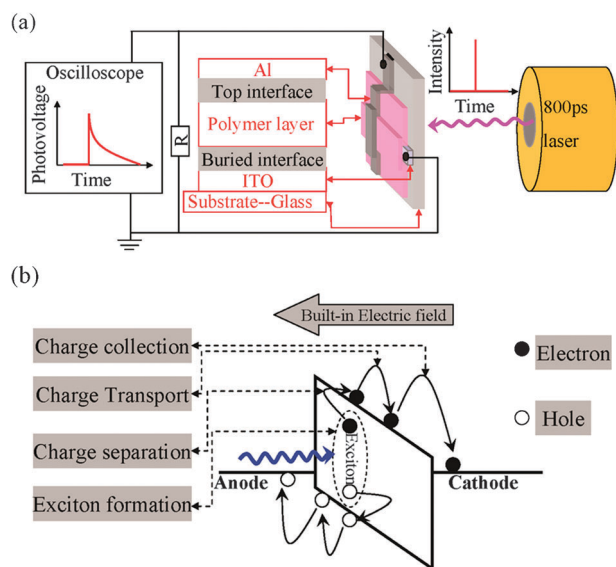


Fig. 8 (a) Schematic view of the setup of transient photo-generated voltage measurement and structure of devices. (b) The four steps in charge dynamics (photon-to-carrier process) of OSCs.

The TPV signal is recorded by a 300 MHz oscilloscope. The laser wavelength is 355 nm, the width of pulse is 0.8 ps, while the power is 40 μ J. Fig. 8(b) illustrates the establishment of general photogenerated voltage, there are four steps in charge dynamics (photon-to-carrier process) of OSCs:^{24,30}

- (1) exciton formation in the polymer layer by light absorption,
- (2) free charge generation in the bulk through the exciton dissociation and charge separation,^{1,31}
- (3) transport of electrons and holes in the opposite direction driven by the built-in electric field,^{16,17,32} and
- (4) charge collection by the anode and the cathode.²⁴

Acknowledgements

This work is supported by the UGC grant (#10401466) of the University of Hong Kong and the General Research Fund (HKU#712010) from the Research Grants Council of Hong Kong Special Administrative Region, China as well as the financial support from Jiawei SolarChina, Co. Ltd. We would like to thank Xiaoyuan Hou and Chee-Leung Mak for their useful discussion and Yiping Sun for her assistance in proofreading the manuscript.

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