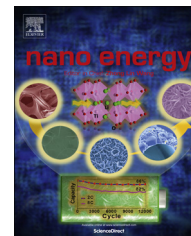




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## FULL PAPER

# The incorporation of thermionic emission and work function tuning layer into intermediate connecting layer for high performance tandem organic solar cells



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Tandem organic solar cell;  
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S-shape;  
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### Abstract

There are two conventional methods to eliminate counter-diode in intermediate connecting layer (ICL) for tandem organic solar cell (OSC), namely forming tunneling junction by using heavily doped electron (ETL) and hole transport layer (HTL) and establishing recombination center of metal layer in between ETL and HTL of ICL. Here, a new method to solve the typical S-shape current density ( $J$ )-voltage ( $V$ ) characteristics of tandem OSC resulted from quasi-Fermi level splitting in ICL has been proposed and demonstrated. Poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate) (PEDOT:PSS) and amino-functionalized alcohol soluble conjugated polymer (PF6N25Py) are effective HTL and ETL respectively in single OSC. Their direct combination as ICL would result a typical S-shape  $J$ - $V$  curve due to mis-aligned quasi-Fermi levels. Our results show that workfunction of PEDOT:PSS can be widely tuned by changing thickness of as-prepared zirconium acetylacetonate (a-ZrAcac). With an 11.1 nm thickness of a-ZrAcac, the WF of PEDOT:PSS has been considerably decreased from 5.0 eV to 3.49 eV. Based on PEDOT:PSS/a-ZrAcac/PF6N25Py ICL, a high efficiency tandem OSC with 8.21% PCE, ignorable  $V_{oc}$  loss and no S-shape  $J$ - $V$  curve has been achieved. The removal of S-shape in  $J$ - $V$  curve is also demonstrated when a-ZrAcac is inserted between PEDOT:PSS/UV-inactivated zinc oxide (ZnO) as ICL in

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tandem OSC. Consequently, this method contributes towards a general strategy to develop other qualified ICL with different HTL and ETL for tandem OSC.  
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## Introduction

Tandem organic solar cell (OSC) using high efficiency photoactive materials with complimentary absorption spectra has the potential to further boost the efficiency of OSC [1–12]. A qualified intermediate connecting layer (ICL) has been recognized to play an important role for the high performance tandem OSCs [9,13,14]. There are three essential requirements for the qualified ICL: (1) optically, the ICL should be transparent so that light loss by the absorption of ICL is minimized; (2) electrically, the ICL should form ohmic equivalent contact so that there is no counter-diode induced  $V_{oc}$  loss and S-shape formation in current density ( $J$ )-voltage ( $V$ ) characteristics; (3) the ICL should be robust enough to protect bottom cell from dissolved by the solvent of top cell.

The efficient combination of electron transport layer (ETL) and hole transport layer (HTL) as qualified ICL has been limited to poly(3,4-ethylenedioxythiophene): poly(styrenesulfonate) (PEDOT:PSS)/ aluminum-doped zinc oxide (AZO), UV-activated titanium dioxide ( $TiO_2$ ) or zinc oxide (ZnO), PEDOT: PSS/ ethoxylated polyethylenimine (PEIE) and molybdenum trioxide  $MoO_3$ /Ag (Al)/ $TiO_2$  (ZnO) so far in literature, which meets the above three requirements simultaneously [5,9,15–17]. The appearance of S-shape in tandem  $J$ - $V$  curve is the major cause of the limitation. In single OSC, the major origin of S-shape has been reported in the literature resulted from imbalanced carrier mobility, accumulated charge carrier, interface barrier and undesirable dipole formation [18–21]. In tandem OSC, constructed from non S-shape single subcells, the S-shape  $J$ - $V$  curve in tandem is mainly caused by the interface barriers in ICL resulted from counter-diode formed by carrier transport layers with mis-aligned quasi-Fermi level [14,22,23].

To better align quasi-Fermi level in ICL, such as PEDOT: PSS/AZO (or UV-activated  $TiO_2$  or ZnO), both the heavily doped HTL and ETL are essential to form tunneling junction to eliminate the counter-diode in ICL, which largely limits the selection of different ETL and HTL materials working as qualified ICL. It is reported that in inverted single OSC structure, when PEDOT:PSS and ZnO are combined as equivalent ETL, a pronounced S-shape appears, which reduce FF,  $V_{oc}$  as well as  $J_{sc}$  dramatically [23]. By replacing ZnO with AZO, the S-shape disappears and the device shows comparable photovoltaic performance compared to the device with single AZO as ETL. The S-shape resulting from PEDOT:PSS/ZnO and PEDOT:PSS/ $TiO_2$  as ICL has also been reported [24,25]. After irradiation of UV light, the S-shape disappears by increasing the doping level of ZnO and  $TiO_2$  to form tunneling junction. However, the UV irradiation, which has to propagate through the organic materials of active layer before reaching and activating the oxide layer in ICL, would exacerbate the organic materials and

device degradation in long term [26]. Another strategy to eliminate the S-shape is to insert a thin layer of Ag or Al as recombination layer between HTL and ETL. More specifically, metal oxides based HTL and ETL, such as  $MoO_x$  and  $TiO_2$  or ZnO, have been used as part of ICL, between which Ag or Al nanoclusters, thermally evaporated under high vacuum condition, act as recombination center to better align metal oxides quasi-Fermi level [16,17]. In this case, when the recombination rate is higher than carrier generation rate due to illumination, the quasi-Fermi level alignment is achieved [27]. However, this metal oxide/metal/metal oxide ICL usually results in some small amount of  $V_{oc}$ ,  $J_{sc}$  and FF loss due to the incomplete separation of ETL and HTL, decreased ICL optical transmittance and high series resistance respectively. Thus, broadening the efficient combination of HTL and ETL as qualified ICL with the features of all-solution process, UV-soaking free and good electrical properties with ignorable  $V_{oc}$  loss and S-shape free  $J$ - $V$  characteristics is highly desirable for the evolution of tandem OSC.

In this work, an all-solution processed, UV-soaking free, multi-layered effective ICL composed of HTL/as-prepared zirconium acetylacetonate (a-ZrAcac)/ETL has been proposed and demonstrated with ignorable  $V_{oc}$  loss and no S-shape formation in  $J$ - $V$  curve. The ICL has a structure of PEDOT:PSS (HTL)/a-ZrAcac/PF6N25Py (ETL). The S-shape formed between PEDOT:PSS and PF6N25Py has been successfully removed by incorporation of a-ZrAcac. Meanwhile, the processing temperature of the ICL is under 100 °C, which is of great benefit for deposition of photoactive layer to avoid high temperature detriments. In addition, the ICL is all-solution, room temperature process, which is suitable for large area solution processes such as roll-to-roll printing, spray, and spin-coating. Furthermore, this PEDOT: PSS/a-ZrAcac/PF6N25Py ICL shows very good  $J$ - $V$  characteristics. Through studying the  $J$ - $V$  characteristics at different low temperatures, it is shown that thermionic emission within a-ZrAcac accounts for the effective recombination of electrons and holes in ICL. We demonstrate a 1.20 V  $V_{oc}$  and 3.51% PCE tandem OSC consisting of two identical subcells (homo-tandem) from poly (3-hexylthiophene) (P3HT): [6, 6]-phenyl C61-butyric acid methylester ( $PC_{60}BM$ ). The high efficiency tandem consists of P3HT based subcell and low bandgap poly(4,8-bis(5-(2-ethylhexyl)-thiophene-2-yl)-benzo [1,2-b:5,4-b']dithiophene-alt alkylcarbonylthieno[3,4-b]thiophene) (PBDTTT-C-T) based subcell shows 8.21% PCE with 1.60 V  $V_{oc}$ . In addition, the removal of S-shape in  $J$ - $V$  curve resulted from ICL is also demonstrated when a-ZrAcac is inserted between PEDOT:PSS and UV-inactivated ZnO as ICL in tandem OSC. As a result, this new ICL strategy contributes towards a general strategy for combining different HTL and ETL as qualified ICL with features of UV-soaking free, room temperature and all-solution process in tandem OSC.

## Experimental section

### Material synthesis and preparation

PEDOT:PSS PH4083 was purchased from Clevious, Inc. PF6N25Py, the amino functionalized water/alcohol soluble conjugated polymer, was provided by Huang's group [28]. P3HT, PC<sub>60</sub>BM, ICBA and PBDTTT-C-T were purchased from Solarmer Energy, Inc. PC<sub>70</sub>BM was purchased from Nano-C. Zirconium acetylacetonate and Triton X-100 were purchased from Sigma Aldrich, Inc.

### Fabrication of single junction OSC and tandem OSC

ITO glass with a sheet resistivity of  $15 \Omega \text{ square}^{-1}$  was cleaned with detergent, acetone, ethanol in sequence followed by 15 min UV-ozone treatment. After that, a thin layer of PF6N25Py was spin-casted at 3000 rpm for 40 s and then vacuumed for 10 min. PF6N25Py was first dissolved in methanol ( $2 \text{ mg ml}^{-1}$ ) with a small amount of acetic acid ( $2 \text{ ul ml}^{-1}$ ), after which the solution was further diluted in butanol (1: 9 vol%). P3HT:PC<sub>60</sub>BM (1:1) in 1,2-dichlorobenzene (DCB) with a concentration of  $20 \text{ mg ml}^{-1}$  was spin-coated at 670 rpm for 40 s followed by 1 h slow growth within a petri dish. The samples were then post-annealed at  $100^\circ\text{C}$  for 10 min. For fast growth, P3HT:PC<sub>60</sub>BM with the same concentration in DCB was spin-coated at different speed and annealed at  $100^\circ\text{C}$  for 10 min immediately. P3HT:ICBA (1: 1) in 1,2-dichlorobenzene (DCB) with a concentration of  $18 \text{ mg ml}^{-1}$  was spin-coated at 800 rpm for 40 s followed by 1 h slow growth within a petri dish. The sample was then post annealed at  $120^\circ\text{C}$  for 10 min as well. After that, a modified PEDOT:PSS was spin-coated on top of P3HT:PC<sub>60</sub>BM and P3HT:ICBA active layer with 2000 rpm and followed by  $100^\circ\text{C}$  annealing for 10 min. The modified PEDOT:PSS was mixed with Triton 100 (500: 1 vol%) and stirred overnight before usage. After that, 100 nm Ag was thermal evaporated to complete the device. PBDTTT-C-T:PC<sub>70</sub>BM (1: 1.8) in chlorobenzene (CB) with a concentration of  $10 \text{ mg ml}^{-1}$  and a 3% volume ratio of 1,8-diiodooctane (DIO) was spin-coated at 1500 rpm for 40 s followed by 1 h vacuum treatment. After that, 10 nm MoO<sub>3</sub> and then 100 nm Ag were thermal evaporated to complete the device.

For tandem OSC fabrication, ITO glass with a sheet resistivity of  $15 \Omega \text{ square}^{-1}$  was cleaned with detergent, acetone, ethanol in sequence followed by 15 min UV-ozone treatment. After that, a thin layer of PF6N25Py was spin-casted at 3000 rpm for 40 s and then vacuumed for 10 min. For P3HT:PC<sub>60</sub>BM homo-tandem OSC, P3HT:PC<sub>60</sub>BM in DCB was spin-coated at 2500 rpm for 40 s followed by  $100^\circ\text{C}$  post annealing for 10 min. Modified PEDOT:PSS was then spin-coated at 2000 rpm for 40 s on top of photoactive layer and post annealed at  $100^\circ\text{C}$  for 10 min. Then a-ZrAcac, dissolved in isopropanol (IPA) with different concentration, was spin-coated above the photoactive layer at 3000 rpm. After that, a thin layer of PF6N25Py was spin-coated atop. Then P3HT:PC<sub>60</sub>BM in DCB was spin-coated at 3000 rpm on top of ICL followed by  $100^\circ\text{C}$  annealing for 10 min. Then modified PEDOT:PSS is spin-coated atop at 2000 rpm for 40 s and annealed at  $100^\circ\text{C}$  post annealing for 10 min. The device was finalized by thermalization of 100 nm Ag. For high efficiency tandem OSC, to control the thickness to tune the short circuit current of first cell, a varied concentration of P3HT ( $16\text{--}18 \text{ mg ml}^{-1}$ ) was used. P3HT:ICBA in DCB was

first spin-coated at 900 rpm for 40 s followed by 1 h slow growth in petri dish and then post annealing at  $120^\circ\text{C}$  for 10 min. After the deposition of PEDOT:PSS/a-ZrAcac/PF6N25Py ICL, PBDTTT-C-T:PC<sub>70</sub>BM in CB was spin-coated at 1500 rpm for 40 s followed by 1 h slow vacuum treatment. The high efficiency tandem OSC device was finalized by thermal evaporation of 10 nm MoO<sub>3</sub> and 100 nm Ag as well.

### Characterizations

J-V characteristics of OSCs were measured by using Keithley 2635 source meter and ABET AM 1.5 G solar simulator with a light intensity of  $100 \text{ mW cm}^{-2}$ . The ICL film thickness was measured by spectroscopic ellipsometry (J.A. WOOLLAM CO. INC.). The photoactive film thickness was measured by a Dektak alpha-step profile. To evaluate the absorption of the device, the reflection of the device was measured with a goniometer combined with a CCD spectrometer and integrating sphere. The absorption of the devices was obtained by  $(100-R)\%$ . For tandem EQE measurement, a 532 nm and 730 nm light bias were selected to light bias the front and rear cells to measure the EQE of the P3HT:ICBA and PBDTTT-C-T:PC<sub>70</sub>BM respectively. The shunt resistances of tandem OSC is as high as  $3.21 \times 10^5 \Omega \text{ cm}^2$ , derived from its dark J-V curve. Thus voltage bias is not necessary to avoid over-estimation of EQE. UPS measurement was performed using a He I discharge lamp (Kratos Analytical) with energy of 21.22 eV and a resolution of 0.15 eV. A 10 V bias was applied on samples to enhance the measured signals.

## Results and discussion

### ICL properties

#### Equivalent Ohmic contact of ICL in Single OSC

To demonstrate the PEDOT:PSS/a-ZrAcac/PF6N25Py working as qualified ICL in tandem OSC, we will first investigate the single unit OSC performance with PF6N25Py as ETL and PEDOT:PSS as HTL. In order to increase the wettability of the hydrophilic PEDOT:PSS on the hydrophobic photoactive layer, such as the P3HT:PC<sub>60</sub>BM, a modified PEDOT:PSS with Triton X-100 surfactant is used as described in *Experimental section*. PF6N25Py, an amino functionalized water/alcohol soluble conjugated polymer, has previously been reported as ETL in organic solar cell and its chemical structure is shown in Figure 1b [28]. The single P3HT:PC<sub>60</sub>BM OSC with structure of ITO/PF6N25Py/ P3HT:PC<sub>60</sub>BM/ PEDOT:PSS/Ag (Device A) has been fabricated as control device. Its J-V characteristics and photovoltaic parameters are shown in Figure 1c and Table 1 respectively. The good photovoltaic performance of the control device with  $0.60 \text{ V } V_{oc}$ ,  $9.60 \text{ mA cm}^{-2} J_{sc}$ , 65.5% FF and 3.77% PCE demonstrates that PF6N25Py and PEDOT:PSS are effective ETL and HTL in single OSC. For tandem OSC, efficient combination of ETL and HTL working as equivalent ohmic contact shall show the same functionality as the used single ETL layer (PF6N25Py) in inverted single unit device. Thus single P3HT:PC<sub>60</sub>BM with structure of ITO/PEDOT:PSS/PF6N25Py/P3HT:PC<sub>60</sub>BM/ PEDOT:PSS/Ag (Device B) has been fabricated and its J-V characteristics and photovoltaic parameters are shown in Figure 1c and Table 1 as well. Interestingly, the device shows a pronounced S-shape J-V curve with  $0.59 \text{ V } V_{oc}$ ,  $5.75 \text{ mA cm}^{-2}$

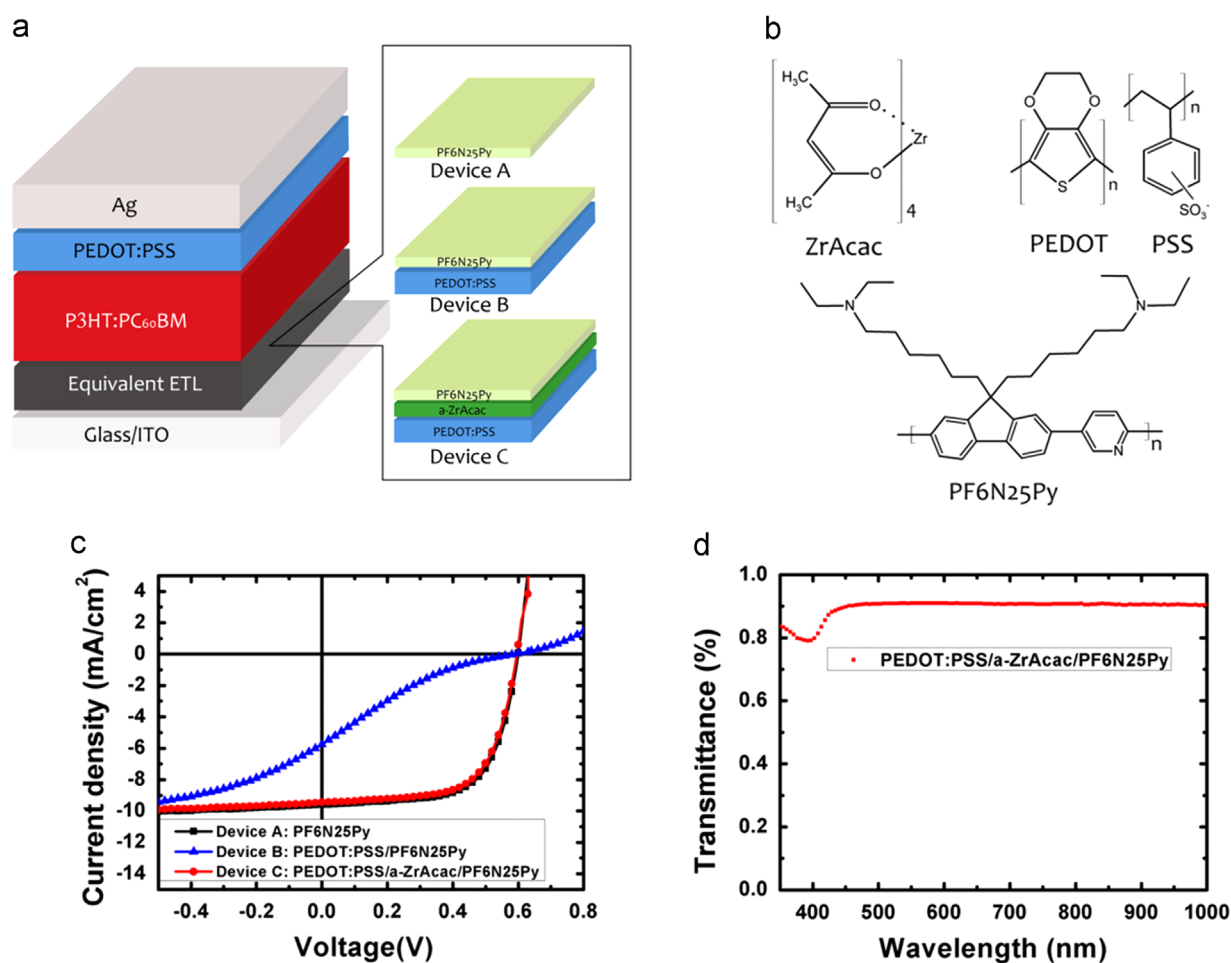


**Figure 1** (a) Device structure of P3HT:PC<sub>60</sub>BM single OSC with different equivalent ETL. (b) The chemical structure of PEDOT:PSS, PF6N25Py, a-ZrAcac. (c) *J*-*V* characteristics of P3HT:PC<sub>60</sub>BM single OSCs employing different ETL. (d). Transmittance of PEDOT:PSS/a-ZrAcac/PF6N25Py.

**Table 1** Photovoltaic performance of P3HT:PC<sub>60</sub>BM single OSCs employing different ETL.

| Device | ETL                         | $J_{sc}$ [mA cm <sup>-2</sup> ] | $V_{oc}$ [V] | FF [%] | PCE [%] |
|--------|-----------------------------|---------------------------------|--------------|--------|---------|
| A      | PF6N25Py                    | 9.60                            | 0.60         | 65.51  | 3.77    |
| B      | PEDOT:PSS/PF6N25Py          | 5.75                            | 0.59         | 17.54  | 0.59    |
| C      | PEDOT:PSS/a-ZrAcac/PF6N25Py | 9.45                            | 0.60         | 64.63  | 3.66    |

$J_{sc}$ , 17.54% FF and 0.59% PCE as compared to device A. This S-shape is very common in tandem OSC, indicating the existence of the non-equivalent ohmic contact resulted from the counter-diode formed between PEDOT:PSS (HTL) and PF6N25Py (ETL) [16,17,23-25,28].

To address the issue, we incorporate a thin layer of a-ZrAcac to prevent the formation of counter-diode between PEDOT:PSS and PF6N25Py, acting as a novel qualified ICL in tandem OSC. Recently, a-ZrAcac has been reported as effective ETL in OSC with normal structure [29]. In single OSC, the work of using multi-layered interface layer to improve the carrier extraction has been well studied. However, for tandem OSC, the work on the three layered, especially featured in solving the key electrical issue of

quasi-Fermi level splitting in ICL with S-shape *J*-*V* curve in ICL of tandem OSC by using a-ZrAcac has rarely been reported. In this work, we introduce and demonstrate a-ZrAcac's work function tuning ability on top of PEDOT:PSS in the three layered ICL structure in tandem OSC. The chemical structure of a-ZrAcac is shown in Figure 1b. The device with structure of ITO/PEDOT:PSS/a-ZrAcac/PF6N25Py/ P3HT:PC<sub>60</sub>BM/PEDOT:PSS/Ag (Device C) as shown in Figure 1a, has been fabricated. The a-ZrAcac was simply prepared by spin-coating its isopropanol (IPA) solution on top of PEDOT:PSS at room temperature without thermal annealing or any other post-treatment as described in Experimental section. As shown in Figure 1c and Table 1, the Device C shows comparable *J*-*V* characteristics and

photovoltaic parameters as the control Device A, with 0.60 V  $V_{oc}$ , 9.45 mA cm<sup>-2</sup>  $J_{sc}$ , 64.6% FF and 3.66% PCE. Importantly, the insertion of a-ZrAcac has successfully eliminated the S-shape  $J$ - $V$  curve formed between PEDOT:PSS and PF6N25Py. The transparency of a-ZrAcac layer is highly desirable in tandem OSC to minimize light absorption of ICL. The transmission spectrum of PEDOT:PSS/a-ZrAcac/PF6N25Py is shown in Figure 1d, with above 90% transmittance at wavelength longer than 450 nm. There is a small valley of 77% at the wavelength of 410 nm, which is mainly due to the 400 nm absorption peak of PF6N25Py [28]. Thus, both of the electrical and optical properties indicate that PEDOT:PSS/a-ZrAcac/PF6N25Py is a qualified ICL in tandem OSC.

#### Thickness effects of a-ZrAcac in ICL for tandem OSC

In order to investigate the PEDOT:PSS/a-ZrAcac/PF6N25Py role as qualified ICL in tandem OSC and the electrical role of a-ZrAcac in the ICL, a series of P3HT:PC<sub>60</sub>BM homo-tandem OSC has been fabricated with different thicknesses of a-ZrAcac. The device performances are shown in Figure 2b and Table 2. When

no a-ZrAcac is inserted between PEDOT:PSS and PF6N25Py, the  $J$ - $V$  curve shows a consistent pronounced S-shape as device B, with 1.18 V<sub>oc</sub>, 3.43 mA cm<sup>-2</sup>  $J_{sc}$ , 39.41% FF and 1.60% PCE. The counter-diode formed between PEDOT:PSS and PF6N25Py results in reduced  $V_{oc}$ , FF and PCE. Interestingly, when the

**Table 2** Photovoltaic performance of P3HT:PC<sub>60</sub>BM tandem OSCs with different thicknesses of a-ZrAcac in PEDOT:PSS/a-ZrAcac/PF6N25Py ICL.

| a-ZrAcac thickness [nm] | $J_{sc}$ [mA cm <sup>-2</sup> ] | $V_{oc}$ [V] | FF [%] | PCE [%] |
|-------------------------|---------------------------------|--------------|--------|---------|
| 0                       | 3.43                            | 1.18         | 39.41  | 1.60    |
| 1.5                     | 3.81                            | 1.19         | 54.01  | 2.45    |
| 3.1                     | 4.05                            | 1.20         | 56.82  | 2.76    |
| 11.1                    | 4.53                            | 1.20         | 64.61  | 3.51    |
| 20.7                    | 4.43                            | 1.19         | 63.08  | 3.33    |
| 26.9                    | 4.37                            | 1.19         | 62.86  | 3.27    |

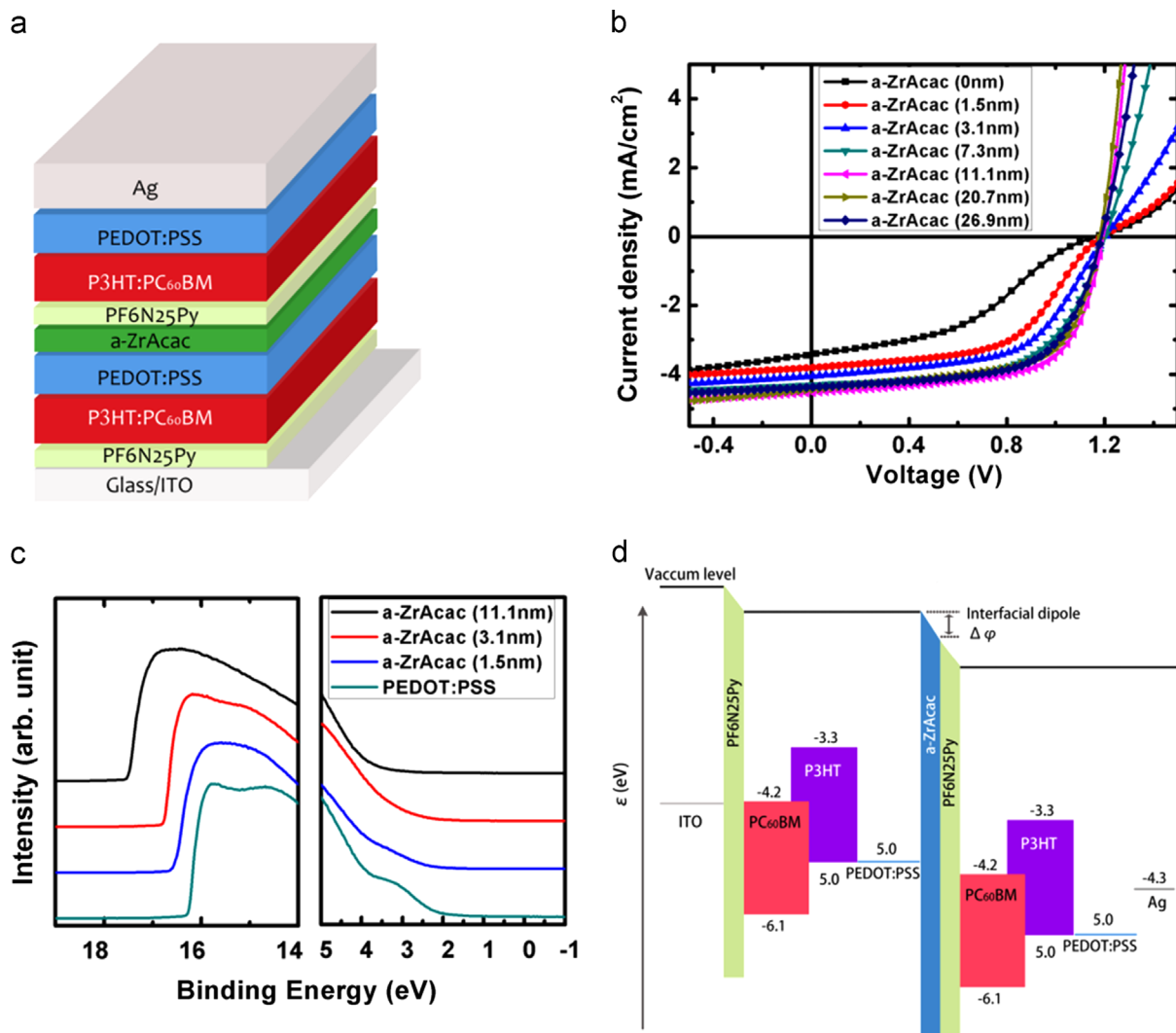


**Figure 2** (a) Device structure of P3HT:PC<sub>60</sub>BM tandem OSC. (b)  $J$ - $V$  characteristics of P3HT:PC<sub>60</sub>BM homo-tandem OSCs with different thicknesses of a-ZrAcac based on PEDOT:PSS/a-ZrAcac/PF6N25Py ICL. (c) UPS spectra of different a-ZrAcac thicknesses on top of PEDOT:PSS. (d) Band diagram of P3HT:PC<sub>60</sub>BM tandem OSC with PEDOT:PSS/a-ZrAcac/PF6N25Py ICL.

thickness of a-ZrAcac increases from 1.5 nm to 11.1 nm, the S-shape weakens gradually and finally disappears. As a result, the device parameters are dramatically improved with FF increased from 39.41% to 64.61%,  $J_{sc}$  from  $3.43 \text{ mA cm}^{-2}$  to  $4.53 \text{ mA cm}^{-2}$ , PCE from 1.60% to 3.51%. Further increase of a-ZrAcac thickness lowers FF majorly due to the increased series resistance in ICL. The optimized thickness of a-ZrAcac is 11.1 nm for P3HT:PC<sub>60</sub>BM homo-tandem OSC with 1.20  $V_{oc}$ ,  $4.53 \text{ mA cm}^{-2}$   $J_{sc}$ , 64.61% FF and 3.51% PCE.

To understand the energy-level alignment of a-ZrAcac with different thicknesses on top of PEDOT:PSS, we conduct ultra-violet photoemission spectroscopy (UPS) measurement, which is a well-established analytical technique for obtaining the energy-level alignment in organic thin films [30–36]. The UPS results are shown in Figure 2c. Together with band diagram in Figure 2d, the performance improvement and elimination of S-shape characteristics can be described by the WF tuning of PEDOT:PSS by the a-ZrAcac dipole. The difference  $\Delta$  of high binding energy cutoff ( $E_{cutoff}$ ) in 12–18 eV between a-ZrAcac and PEDOT:PSS indicates the shift of vacuum level ( $E_{vac}$ ). The deposition of a-ZrAcac on top of PEDOT:PSS results a higher  $E_{cutoff}$  with increasing thickness from 0 to 11.1 nm, showing a stronger positive dipole formation from a-ZrAcac to PEDOT:PSS. The highest occupied molecular orbital (HOMO) level of a-ZrAcac is extracted from the onset of UPS in low energy. Ionization potentials ( $I_p$ ) is extracted with equation [37]:

$$I_p = 21.22 - (E_{cutoff} - \text{HOMO}) \quad (1)$$

The absorbance of a-ZrAcac is shown in Figure S1 with high transparence after 340 nm in the visible wavelength range. The absorption peak of acetylacetonate located around 300 nm attributes to the n-p\* and p-p\* intra-ligand electronic transitions [38]. By combination of  $I_p$  and 3.65 eV  $E_{opt}$  (derived from absorbance 340 nm onset as shown in Figure S1), the lowest unoccupied molecular orbital (LUMO) of a-ZrAcac can be obtained. Table 3 summarizes the variables of  $E_{cutoff}$ ,  $I_p$ ,  $E_A$ ,  $E_f$ ,  $\phi_{hole}$ ,  $\phi_{electron}$  and  $\Delta$ . Consequently, the deposition of a-ZrAcac with different thicknesses from 0 nm to 11.1 nm produces a strong dipole, tuning the WF of PEDOT:PSS in the top subcell. With an 11.1 nm thickness of a-ZrAcac layer, the WF of PEDOT is decreased from 5.0 eV to 3.49 eV to facilitate electrons from the second cell to recombine with holes from first cell.

### Temperature effects of ICL in tandem OSC

In order to further investigate the carrier recombination mechanism in this ICL, the J-V characteristics of PEDOT:PSS/a-ZrAcac/PF6N25Py ICL based P3HT:PC<sub>60</sub>BM homo-tandem OSC at different temperatures is shown in Figure 3a. When temperature decreases from 300 K to 240 K,  $J_{sc}$ , FF and PCE decrease while  $V_{oc}$  increases. In conjugated polymer, carrier transport is majorly dominated by thermally assisted hopping and current is negatively affected by recombination via trap,

both of which would lead to increase of current when temperature increases [39,40]. The  $V_{oc}$  shows almost linear dependence on temperature, which is consistent for inorganic solar cell theory [41]. When temperature further decreases from 240 K to 60 K,  $J_{sc}$  continues to decrease and  $V_{oc}$  continues to increase. Meanwhile, a pronounced S-shape appears in the J-V curve, indicating the formation of barrier in the interface of P3HT:PC<sub>60</sub>BM homo-tandem OSC. The appearance of S-shape J-V curve of tandem OSC at low temperature indicates the existence of counter-diode in ICL, which prevents efficient recombination of holes from PEDOT:PSS of first cell and electrons from the PF6N25Py of second cell.

In order to investigate the origin of S-shape is whether from the ETL, HTL or a-ZrAcac in ICL of the tandem OSC, single P3HT:PC<sub>60</sub>BM OSC with PEDOT:PSS as HTL and PF6N25Py as ETL is studied as shown in Figure 3b. When temperature decreases from 300 K to 60 K,  $J_{sc}$  decreases and  $V_{oc}$  increases with no appearance of S-shape. Since both the single and tandem OSCs use the same ETL and HTL, the results exclude the possible origin of S-shape from neither the HTL (PEDOT:PSS) nor the ETL (PF6N25Py) but rather from the a-ZrAcac in the ICL of tandem OSC. From Figure 3a, the results can be explained that when the temperature decreases, the reduction of kinetic energy of carriers prevents the carriers from jumping over the barrier at the interfaces of a-ZrAcac existed at low temperature, resulting in S-shape J-V curves. Therefore, we can understand the results as thermionic emission within a-ZrAcac for helping electrons and holes to recombine for PEDOT:PSS/a-ZrAcac/PF6N25Py function as effective ICL.

### UV-soaking free property of ICL in tandem OSC

It has been reported that UV soaking is needed for some common ETL such as metal oxides of TiO<sub>2</sub> and ZnO for promoting trap filling and eliminating the S-shape characteristics in tandem OSC [24,25]. In our result, the UV-soaking free feature of PEDOT:PSS/a-ZrAcac/PF6N25Py ICL is demonstrated in P3HT:PC<sub>60</sub>BM homo-tandem OSC as shown in Figure 4a. We use UV light with 400 nm cutoff filter (a-ZrAcac absorbance onset is 340 nm as shown in Figure S1) to investigate the UV soaking effect. It is observed that with (Curve B) and without the UV light pre-illumination (Curve A), the J-V curves are almost identical without formation of S-shape. These results confirm that PEDOT:PSS/a-ZrAcac/PF6N25Py ICL is UV-soaking free compared to PEDOT:PSS/TiO<sub>2</sub> or ZnO ICL, which would benefit tandem OSC device performance without UV induced degradation in long term.

Using a-ZrAcac to remove S-shape in J-V curve based on PEDOT:PSS/UV-inactivated ZnO ICL has also been demonstrated in Figure 4b. First, PEDOT:PSS/ZnO ICL based P3HT:PC<sub>60</sub>BM homo-tandem OSC's J-V curve has been measured under UV light with 400 nm cutoff filter. When no UV light pre-illumination is used, the result shows a distinct S-shape, indicating the

**Table 3** Summary of results from UPS measurement for a-ZrAcac with different thicknesses on top of PEDOT:PSS.

| a-ZrAcac thickness [nm] | $\phi_{electron}$ [eV] | $\phi_{hole}$ [eV] | $E_{cutoff}$ [eV] | $\Delta$ [eV] | WF [eV] | $I_p$ [eV] | $E_A$ [eV] |
|-------------------------|------------------------|--------------------|-------------------|---------------|---------|------------|------------|
| 1.5                     | 1.04                   | 2.61               | 16.61             | 0.31          | 4.59    | 6.91       | 3.26       |
| 3.1                     | 0.61                   | 3.04               | 16.78             | 0.48          | 4.42    | 6.99       | 3.34       |
| 11.1                    | 0.07                   | 3.58               | 17.71             | 1.41          | 3.49    | 7.11       | 3.46       |

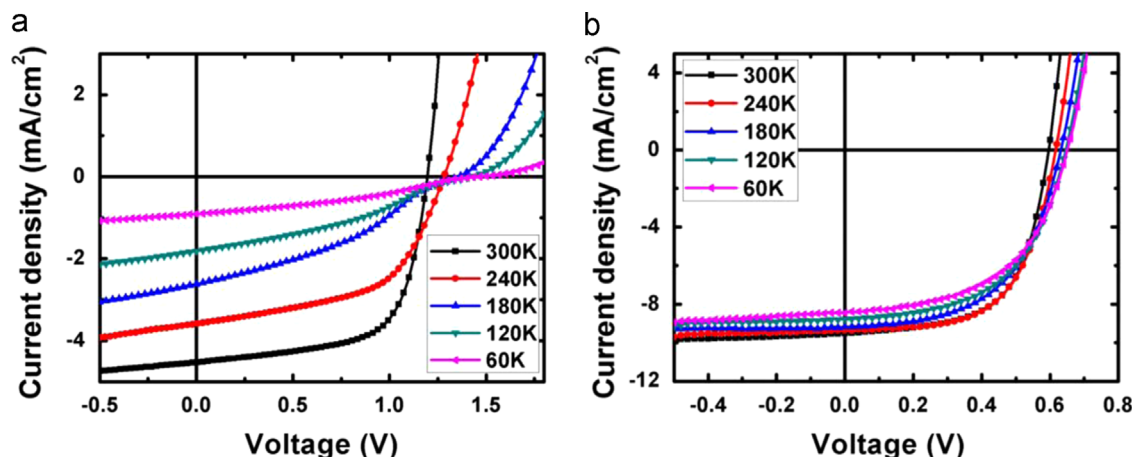


**Figure 3**  $J$ - $V$  characteristics at different temperatures for (a) P3HT:PC<sub>60</sub>BM homo-tandem with PEDOT:PSS/a-ZrAcac/PF6N25Py ICL and (b) single P3HT:PC<sub>60</sub>BM OSC with PEDOT:PSS as HTL and PF6N25Py as ETL.

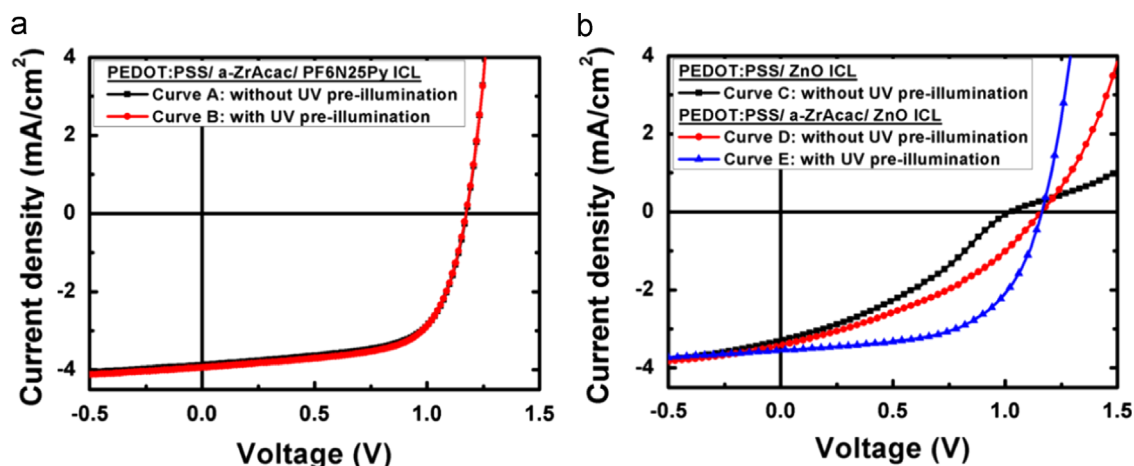


**Figure 4**  $J$ - $V$  characteristics measured with and without UV light pre-illumination for ICL of (a) PEDOT:PSS/a-ZrAcac/PF6N25Py and (b) PEDOT:PSS/ ZnO and PEDOT:PSS/z-ZrAcac/ZnO. All the devices are measured under UV light with 400 nm cutoff filter.

existence of counter-diode (Curve C). Secondly, PEDOT:PSS/a-ZrAcac/ZnO ICL based P3HT:PC<sub>60</sub>BM homo-tandem OSC's  $J$ - $V$  curve has also been measured under UV light with 400 nm cutoff filter. When no UV light pre-illumination is used, the device shows a poor FF without S-shape formation in  $J$ - $V$  curve (Curve D), indicating no interface barrier in the ICL. We can understand the poor FF results from low carrier density in ZnO. When UV light pre-illumination is used, the  $J$ - $V$  curve shows increased FF (Curve E), which is mainly due to the increased carrier density in ZnO after UV light pre-illumination activation. Thus, the incorporation of a-ZrAcac between PEDOT:PSS and UV-inactivated ZnO to solve the key ICL electrical issue of quasi-Fermi level splitting resulted S-shape  $J$ - $V$  curve provides another fact that our multi-layered strategy of ICL is a general strategy to develop other qualified ICL for tandem OSC.

## Tandem OSC device performance

### P3HT:PC<sub>60</sub>BM tandem OSC

The optimized P3HT:PC<sub>60</sub>BM homo-tandem OSC based on PEDOT:PSS/a-ZrAcac/PF6N25Py ICL has achieved 3.51% PCE with doubled 1.20 V  $V_{oc}$  and its  $J$ - $V$  characteristics and photovoltaic performance are shown in Figure 5 and

Table 4 respectively. The device fabrication details are described in *Experimental section*. To balance the photon absorption in the subcells of P3HT:PC<sub>60</sub>BM homo-tandem OSC, thin first and second photoactive layers have been formed by fast spin-coating without slow growth process. Thus for a more objective comparison of  $J_{sc}$  enhancement, a fast growth single P3HT:PC<sub>60</sub>BM OSC has been optimized as control device, which has 0.60 V  $V_{oc}$ , 7.22 mA cm<sup>-2</sup>  $J_{sc}$ , 60.98% FF and 2.64% PCE. It can be observed from Table 4 that the total sum of  $J_{sc}$  of the two subcells in P3HT:PC<sub>60</sub>BM homo-tandem OSC is 9.06 mA cm<sup>-2</sup>, which is 25.48% enhancement compared to optimized fast growth single P3HT:PC<sub>60</sub>BM OSC. In addition, P3HT:PC<sub>60</sub>BM homo-tandem OSC shows 5.95% higher FF (from 60.98% to 64.61%) compared to the optimized single P3HT:PC<sub>60</sub>BM OSC, which can be explained by the stronger carrier extraction in thinner photoactive layer. As a result, the combined improvement of  $J_{sc}$  and FF in P3HT:PC<sub>60</sub>BM homo-tandem OSC results 32.95% PCE enhancement from 2.64% to 3.51% compared to its single OSC.

The total absorption of P3HT:PC<sub>60</sub>BM single and homo-tandem OSCs are shown in Figure 5b. It is observed that the major absorption enhancement of P3HT:PC<sub>60</sub>BM homo-tandem OSC covers the wavelength ranging from 350 nm to 450 nm and

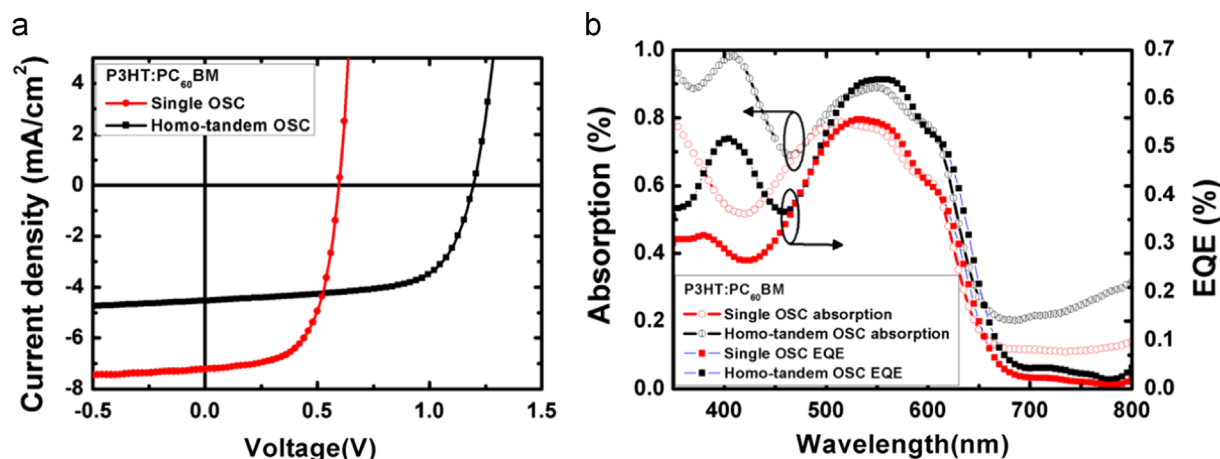


Figure 5 (a)  $J$ - $V$  characteristics and (b) device absorption and EQE for fast growth P3HT:PC<sub>60</sub>BM single and homo-tandem OSCs.

Table 4 Photovoltaic performance of fast growth P3HT:PC<sub>60</sub>BM single and homo-tandem OSCs.

| Device | Structure                                | $J_{sc}$ [mA cm <sup>-2</sup> ] | $V_{oc}$ [V] | FF [%] | PCE [%] |
|--------|------------------------------------------|---------------------------------|--------------|--------|---------|
| D      | P3HT:PC <sub>60</sub> BM single OSC      | 7.22                            | 0.60         | 60.98  | 2.64    |
| E      | P3HT:PC <sub>60</sub> BM homo-tandem OSC | 4.53                            | 1.20         | 64.61  | 3.51    |

490 nm to 600 nm. For external quantum efficiency (EQE), a calculated approach is used assuming that the subcells of homo-tandem OSC has the same internal quantum efficiency (IQE) of its single OSC since measuring EQE by light biasing for homo-tandem OSC's is not feasible [42,43]. The EQE of measured single OSC and calculated homo-tandem OSC EQE are shown in Figure 5b as well. P3HT:PC<sub>60</sub>BM homo-tandem OSC shows almost identical EQE to single OSC above 650 nm. For wavelength shorter than 650 nm, especially from 350 nm to 450 nm and 490 nm to 600 nm, an obvious EQE enhancement, which is mainly due to the increased photon absorption, is achieved in P3HT:PC<sub>60</sub>BM homo-tandem OSC compared to its single OSC.

#### P3HT:ICBA and PBDTTT-C-T:PC<sub>70</sub>BM tandem OSC

Tandem OSC based on P3HT: indene-C60bisadduct (ICBA) and PBDTTT-C-T: [6, 6]-phenyl C71-butyric acid methyl ester (PC<sub>70</sub>BM) subcells has been used to demonstrate PEDOT:PSS/a-ZrAcac/PF6N25Py ICL for high efficiency tandem solar cell. The single optimized P3HT:ICBA with structure of ITO/ PF6N25Py/ P3HT:ICBA/PEDOT:PSS/Ag and the single optimized PBDTTT-C-T: PC<sub>70</sub>BM with structure of ITO/PF6N25Py/PBDTTT-C-T:PC<sub>70</sub>BM/ MoO<sub>3</sub>/Ag have been fabricated and its  $J$ - $V$  characteristics and photovoltaic parameters are shown in Figure 6a and Table 5. The reason for using MoO<sub>3</sub> instead of PEDOT:PSS on top of PBDTTT-C-T:PC<sub>70</sub>BM acting as HTL is that PEDOT:PSS would introduce large surface recombination atop low bandgap material, resulting in reduced  $J_{sc}$ , FF as well as  $V_{oc}$  [42]. The P3HT:ICBA single OSC shows 0.84 V  $V_{oc}$ , 10.34 mA cm<sup>-2</sup>  $J_{sc}$ , 65.31% FF and 5.67% PCE. The PBDTTT-C-T:PC<sub>70</sub>BM single OSC shows 0.77 V  $V_{oc}$ , 14.62 mA cm<sup>-2</sup>  $J_{sc}$ , 63.58% FF and 7.16% PCE. The device fabrication details are described in Experimental section. An optimized tandem OSC consisting of P3HT:ICBA and PBDTTT-C-T:PC<sub>70</sub>BM as its subcell based on PEDOT:PSS/a-ZrAcac/PF6N25Py ICL has achieved 1.60 V  $V_{oc}$ , 7.58 mA cm<sup>-2</sup>  $J_{sc}$ , 63.69% FF and 8.21%

PCE. The tandem  $V_{oc}$  is almost equal to the sum of its subcell with an ignorable loss, which indicates PEDOT:PSS/a-ZrAcac/ PF6N25Py as qualified ICL for high efficiency tandem OSC. Figure 6b shows the EQE of P3HT:ICBA and PBDTTT-C-T:PC<sub>70</sub>BM subcell. Integration of EQE gives  $J_{sc}$  of 8.54 mA cm<sup>-2</sup> and 7.76 mA cm<sup>-2</sup> for P3HT:ICBA and PBDTTT-C-T:PC<sub>70</sub>BM subcell respectively in its tandem OSC. In series connected tandem OSC, the total  $J_{sc}$  is decided by the smallest  $J_{sc}$  of its subcells. Thus the bottom cell PBDTTT-C-T:PC<sub>70</sub>BM with  $J_{sc}$  of 7.76 mA cm<sup>-2</sup> determines the tandem  $J_{sc}$ , which is quite close to 7.58 mA cm<sup>-2</sup>. The optimized P3HT:ICBA and PBDTTT-C-T:PC<sub>70</sub>BM tandem OSC shows 14.66% (from 7.16% to 8.21%) and 44.80% (from 5.67% to 8.21%) PCE enhancement compared to their optimized single cell. The PCE improvement of tandem OSC compared to its optimized single P3HT:ICBA and PBDTTT-C-T:PC<sub>70</sub>BM comes from both enhanced photon absorption and minimized thermalization loss.

## Conclusion

A room temperature and all-solution processed multi-layered ICL, featured in a-ZrAcac film sandwiched between PEDOT:PSS and PF6N25Py, has been demonstrated in tandem OSC. The incorporation of a-ZrAcac has successfully removed the typical S-shape  $J$ - $V$  curve for tandem OSC based on PEDOT:PSS/PF6N25Py ICL by tuning the WF of PEDOT:PSS with its thickness. Through low temperature  $J$ - $V$  characteristics measurement, we show that the thermionic emission within a-ZrAcac accounts for the effective recombination of electrons and holes in ICL. Based on that, tandem OSCs with ignorable total  $V_{oc}$  loss and no S-shape formation in  $J$ - $V$  curve have been achieved. A homo-tandem based on P3HT:PC<sub>60</sub>BM shows 1.20 V  $V_{oc}$  and 3.51% PCE (32.95% enhancement from its single OSC) has been achieved and a high

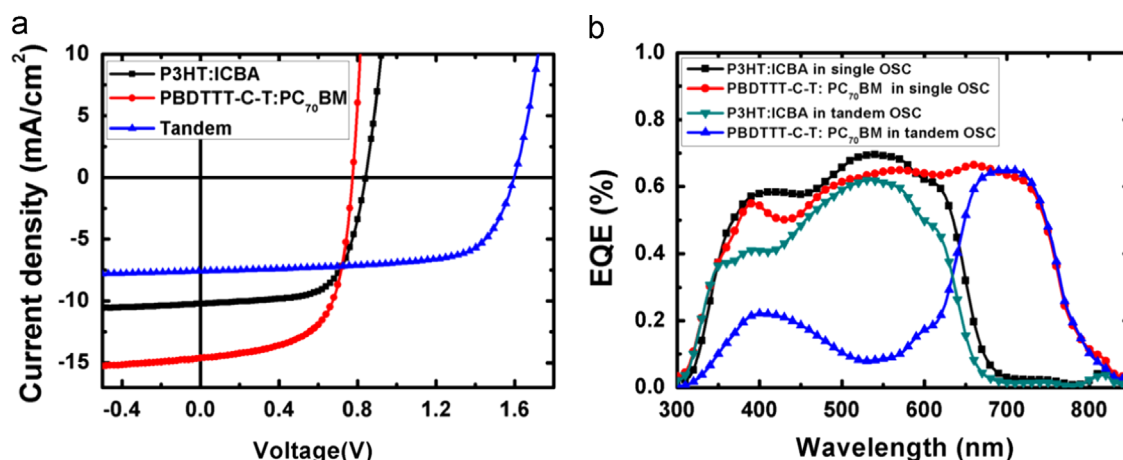


**Figure 6** (a) *J-V* characteristics and (b) device absorption and EQE for P3HT:ICBA, PBDTTT-C-T:PC<sub>70</sub>BM single and tandem OSCs.

**Table 5** Photovoltaic performance of P3HT:ICBA and PBDTTT-C-T:PC<sub>70</sub>BM single and tandem OSCs.

| Device | Structure                                 | $J_{sc}$ [mA cm <sup>-2</sup> ] | $V_{oc}$ [V] | FF [%] | PCE [%] |
|--------|-------------------------------------------|---------------------------------|--------------|--------|---------|
| F      | P3HT:ICBA single OSC                      | 10.34                           | 0.84         | 65.31  | 5.67    |
| G      | PBDTTT-C-T:PC <sub>70</sub> BM single OSC | 14.62                           | 0.77         | 63.58  | 7.16    |
| H      | Tandem OSC                                | 7.58                            | 1.60         | 67.73  | 8.21    |

efficiency tandem OSC based on P3HT:ICBA and PBDTTT-C-T:PC<sub>70</sub>BM subcells with 1.60 V  $V_{oc}$  and 8.21% PCE has been realized. In addition, the removal of S-shape in *J-V* curve by a-ZrAcac in ICL has also been demonstrated when a-ZrAcac is inserted between PEDOT:PSS/UV-inactivated ZnO as ICL in homo-tandem OSC. Consequently, this HTL/zirconium acetylacetonate/ETL ICL contributes towards a new strategy to develop other qualified ICL with different HTL and ETL for tandem OSC.

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## Appendix A. Supplementary material

Supplementary data associated with this article can be found in the online version at <http://dx.doi.org/10.1016/j.nanoen.2016.01.002>.

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