High efficient inverted polymer solar cells with different annealing treatment

Xiaowei Xu a, Fujun Zhang a,⁎⁎, Jian Zhang b,⁎⁎, Hui Wang c, Zuliang Zhuo a, Yang Liu a, Jian Wang a, Zixuan Wang a, Zheng Xu a

⁎ Key Laboratory of Luminescence and Optical Information (Ministry of Education of China), Beijing Jiaotong University, Beijing 100044, PR China
⁎⁎ State Key Laboratory of Catalysis, Dalian Institute of Chemical Physics CAS, 457 Zhongshan Road, Dalian 116023, PR China
a Key Laboratory of Photochemical Conversion and Optoelectronic Materials, Technical Institute of Physics and Chemistry CAS, Beijing 100190, PR China

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Inverted polymer solar cells (IPSCs) were fabricated with cesium carbonate (Cs2CO3) modified indium tin oxide (ITO) substrates as the electrode and molybdenum trioxide (MoO3) modified Al as the anode. The Cs2CO3 dissolved in 2-ethoxyethanol was spin-coated on ITO substrates, showing snowflake-like morphology characterized by the scanning electron microscope (SEM). The absorption, X-ray diffraction as well as the morphology of the active layer were measured before and after annealing treatment. The IPSCs with annealing treatments on the active layers and MoO3 layers exhibited the maximum power conversion efficiency (PCE) approaching to 2%, with open circuit voltage (Voc) of 0.57 V, short circuit current density (Jsc) of 8.8 mA/cm2 and fill factor (FF) of 38.7%. The performance of IPSCs was dramatically decreased by annealing treatment after deposition of Al cathode, which may be due to the diffusion of Al atom crossing the MoO3 layer forming new channels for charge carrier collection. However, the new channels are not beneficial to the charge carrier collection, which is demonstrated from that the Jsc of IPSCs was evidently decreased from 8.8 to 4.6 mA/cm2 by annealing treatment after deposition Al layer. The annealing treatment after deposition of MoO3 could improve the interfacial contact to aid in electron extraction.

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1. Introduction

Polymersolar cells (PSCs) have attracted more and more attention due to its multi-advantages, such as light weight, low cost, ease of large scale manufacture compatibility with flexible substrates [1,2]. As a choice for inexpensive clean and sustainable renewable energy source, the power conversion efficiency (PCE) of PSCs draws much concentration and has been rapidly increased to more than 7% [3-5]. The PCE of PSCs has reached the value for commercialization, while the stability and lifetime of PSCs are another two thorny problems for its commercial applications. In a conventional PSCs, the hole transporting layer, poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate) (PEDOT:PSS) is spin-coated on the ITO substrate to enhance the work function to aid in hole extraction, however, PSS causes corrosion of ITO and degrades the cell performance due to its strong acidity. The low work function metal as top cathode of conventional PSCs is easily oxidized in ambient conditions, resulting in reduced device lifetime [6]. In order to improve the stability and lifetime of PSCs, a novel structural PSCs use high work function metal as the top anode and low work function metal or metal oxide as the bottom cathode was proposed and named as inverted configuration polymer solar cells (IPSCs) [7]. As concern as the IPSCs, PEDOT:PSS is replaced by Ca [8], titanium dioxide (TiO2) [9] and zinc oxide (ZnO) [10], high work function materials are used as top anode for better hole extraction and stability of cells, such as molybdenum trioxide (MoO3) [11], vanadium oxide (V2O5) [12] and PEDOT:PSS [13]. Recently, Liao et al. reported that the work function of cesium carbonate (Cs2CO3) could be changed from −3.45 eV to −3.06 eV under low temperature (about 150 °C) annealing treatment and obtained a high PCE of IPSCs with Cs2CO3 modified ITO as cathode [14]. Lee et al. reported that the PCE of IPSCs arrives to 4.01% by optimizing the thickness of MoO3 layer. The stability of IPSCs could be effectively improved compared with conventional configuration PSCs [15]. In our pervious works, we have reported a kind of inverted configuration organic small molecule solar cells, which use Ca modified ITO as the electrode and MoO3 modified Ag as the anode. A maximum PCE of 0.64% was obtained by optimizing the thicknesses of active layer and MoO3 layer [16]. Recently, Chu et al. reported high PCE of 6.7% IPSCs based on a blend of low band gap alternating copolymer of 4,4-bis[2-ethylhexyl]-dithieno[3,2-b:20,20-d]-silole and N-octylthieno[3,4-c]pyrrole-4,6-dione (PDTPSTP) and PCBM as an active layer [17]. The IPSCs is highly stable, retaining 85% of its original efficiency after being stored in air for 32 days even without encapsulation. Chen et al. also reported that high efficiency IPSCS with P3HT:PCBM as the active layer with liquid-functionalized carbon nanoparticles...
modified ITO as cathode was demonstrated, which is comparable to PCE of a reference P3HT:PCBM-based solar cell having a conventional structure [18].

The polymer poly(3-hexylthiophene) (P3HT) blended with the fullerene derivative phenyl-C61-butyric acid methyl ester (PCBM) has been demonstrated to be a high efficiency system [6,19]. It is worthwhile to mention that annealing treatment was found to be an effective way to improve the performance of PSCs, the potential influence of annealing treatment on the performance of PSCs could be summarized as the followings: i) increase the phase separation between P3HT and PCBM domains [20]; ii) increase the interfacial contact between the metal electrode and the active layer, resulting in the increase of FF [21]; iii) increase P3HT crystallization, including its interchain and intrachain orders [22,23]; iv) enhance P3HT absorption intensity and range [24,25].

Karagiannidis et al. demonstrated the vertical phase separation of P3HT:PCBM blend after thermal annealing (P3HT enrichment at the film-air interface and abundance of PCBM at the bottom), this type of vertical phase separation of P3HT:PCBM blend after thermal annealing is not appropriate for charge carrier collection in conventional organic solar cells, but beneficial for the charge carrier transportation in IPSCs [26,27]. In this paper, the effect of annealing treatment on the IPSCs performance was discussed based on different annealing treatment processes on the active layer and MoO3 layer. The optimized performance of IPSCs was obtained based on the cells with annealing treatments on the active layer and MoO3 layer.

### 2. Experimental details

#### 2.1. Preparation of substrates

The indium tin oxide (ITO) glass substrates were purchased from Foshan Junyiming Class & Mirror Co., LTD. The thickness of ITO films is about 135 ± 15 nm and its sheet resistance is about 15 Omega/Square. The ITO substrates were cleaned consecutively in ultrasonic baths containing acetone, ethanol and de-ionized water and dried by high pure nitrogen gas. The cleaned ITO substrates were treated by UV-ozone for 10 min for further clean and improvement of work functions. The Cs2CO3 (purchased from Aldrich Sigma) was dissolved in 2-ethoxyethanol or de-ionized water with the concentration 1 mg/ml (~0.1 wt.%) and then spin-coated onto the ITO substrates at the speed of 4000 rpm for 40 seconds. The ITO glass substrates coated with Cs2CO3 thin films were transferred to a hot plate and annealed at 150 °C for 20 min in a clean room conditions. The thickness of prepared Cs2CO3 thin films is about 1 nm, which is similar with the result of Ref. [28].

#### 2.2. Fabrication of inverted polymer solar cells

Polymer P3HT and PC60BM (purchased from Nichem Fine Technology co. Ltd.) were dissolved in chloroform with concentration 20 mg/ml, respectively and then blended together with the same
weight ratio. The blended solution was spin-coated on Cs$_2$CO$_3$ coated ITO substrates at the speed of 2000 rpm for 30 s. The thickness of P3HT:PCBM is about 180 nm under the fabrication conditions. The substrates coated with active layers were transferred to a vacuum chamber, a 8 nm thickness layer of MoO$_3$ was deposited on the active layer under $5 \times 10^{-3}$ Pa vacuum. The film thickness was monitored by a quartz crystal microbalance. The aluminum (Al) cathode with the thickness of 100 nm and the active area 0.09 cm$^2$ defined through foursquare shadow mask was evaporated under $5 \times 10^{-3}$ Pa vacuum.

The configuration of the IPSCs is ITO/Cs$_2$CO$_3$/P3HT:PCBM/MoO$_3$ (8 nm)/Al. All the annealing treatments were carried out in the room conditions.

2.3. Characterization of inverted polymer solar cells

The absorption spectra of the blend thin films were measured by Shimadzu UV-3101 PC spectrometer. The current–voltage characteristics of IPSCs were measured by a Keithley source meter 2410 in dark and under
illuminated at 100 mW/cm² by using a 150 W Xenon lamp. The morphology of thin films was characterized by scanning electron microscope (SEM, XL30 S-FEG). The elemental analysis of P3HT:PCBM films before and after annealing treatment was made with the same SEM, equipped with a mode for in situ energy dispersive X-ray (EDX) spectra. The morphology of films was recorded by atomic force microscope (AFM), using a Multimode Nanoscope IV (Veeco) operated in Tapping Mode. The thickness of blended thin films was measured by surface profiler XP-2 (Ambios Technology), which was also marked by cross section of SEM. X-ray diffraction (XRD) patterns were recorded using a Bruker D8-Focus equipped with Goebel mirror using Cu-Kα radiation. Data were recorded in the 2θ range of 4 – 15° with step of 0.02°. All measurements were carried out at room conditions. The schematic diagram of IPSCs and energy level of used materials, as well as our designed test box for solar cells are and organic light emitting diodes shown in Fig. 1.

3. Results and discussion

The morphology of Cs₂CO₃ films prepared from de-ionized water solution and 2-ethoxyethanol solution were investigated by SEM and are shown in the Fig. 2. It could be observed from the Fig. 2-a) that ITO surface could not be fully covered by Cs₂CO₃ dissolved in de-ionized water; showing different size of Cs₂CO₃ particles on ITO substrate. The large size of Cs₂CO₃ particles may be attributed to the Cs₂CO₃ agglomeration during the slow volatilization of water from the films. The IPSCs with Cs₂CO₃ dissolved in de-ionized water modified ITO surface does not exhibit obvious photovoltaic performance. The Cs₂CO₃ film prepared from 2-ethoxyethanol solution exhibits snowflake-like morphology and mostly covers the ITO surface, as shown in the Fig. 2-b) and c). The 2D snowflake-like morphology of Cs₂CO₃ film changes into 3D island structure by annealing treatment at 150 °C for 20 min, which could be observed from Fig. 2-c) and d). Liao et al. had reported that the work function of Cs₂CO₃ can be modified from −3.45 to −3.06 eV by a low temperature annealing treatment, verified by ultraviolet photoelectron spectroscopy [14]. Therefore, annealing treatment on Cs₂CO₃ coated ITO substrates should be beneficial to the electron collection by Cs₂CO₃ coated ITO substrates due to the decrease of work function from −4.9 eV to −3.06 eV. Meanwhile, the contact angle increases along with the annealing temperature when the temperature is less than 170 °C. The increase of the contact angle indicates a surface property transition from hydrophilic to hydrophobic upon annealing of the Cs₂CO₃ layer, which is beneficial for polymer film growth on it [14]. In our experiments, the IPSCs fabrication on Cs₂CO₃ films with annealing treatment show better photovoltaic performance compared with the IPSCs based on Cs₂CO₃ films without annealing treatment.

Various annealing treatments on the organic active layers and organic electronic devices were carried out to enhance device’s performance [19,20,29-31]. Here, we also investigated the effect of annealing treatment on the optical and morphology characteristics of P3HT:PCBM layer. The absorption spectra of P3HT, PCBM and P3HT:PCBM films before and after annealing treatment were measured and are shown in the Fig. 3. The absorption peaks and intensity of pure P3HT and PCBM films don’t show distinguished changes before and after annealing treatment, indicating that the film already had a high degree of ordering [32]. However, the absorption peak of blended P3HT:PCBM films shows a 25 nm red-shift from 478 nm to 503 nm and absorption intensity is increased in the longer wavelength range after annealing treatment. It means that the more excitons could be generated due to its more photon harvesting in the active layer after annealing.
treatment, resulting in the increase of $J_{sc}$. The absorption spectral change of P3HT:PCBM thin films should be attributed to the enhanced absorption at the longer wavelength of the ordered phase P3HT induced by annealing treatment, which could be further demonstrated by the X-ray diffraction (XRD) of P3HT:PCBM with different temperature annealing treatments, as shown in the Fig. 4.

The structural analysis of the P3HT:PCBM films with different temperature annealing treatments was characterized by XRD. Fig. 4 illustrates the effect of different annealing temperature on the structure of the P3HT:PCBM thin films. All the XRD images show a peak at 5.3°, which is associated with the (100) reflection of the P3HT. This peak corresponds to a-axis orientation, with the main polymer chain parallel and the side chains perpendicular to substrate. The intensity of this diffraction peak at 5.3° arrives to the maximum after annealing treatment at 120 °C for 10 min, indicating an improvement of the degree of crystallization of P3HT [20]. The increase of crystallization degree of P3HT along (100) orientation suggested that the vertical phase separation extent should be increased, especial for the top surface of P3HT:PCBM films, which will be beneficial to the hole transporting and collections [33]. The decrease of crystallization degree of P3HT along (100) orientation may be due to the oxidation of P3HT or PCBM under higher annealing temperature 150 °C for 10 min.

The cross-section SEM images of P3HT:PCBM films before and after annealing treatment are shown in the Fig. 5, the thickness of blend films was decreased from 193 nm to 117 nm after annealing treatment. The decrease of blend film thickness may be due to quick volatilization of solvent during annealing treatment, resulting in the more compact of films. Meanwhile, the morphology of P3HT:PCBM surface becomes more rough, as shown in the Fig. 5. This phenomenon accords with the change of root means square roughness of the active layer from 0.38 to 1.74 nm before and after annealing treatment, which was calculated according to the AFM images. Fig. 6 shows the surface morphology of P3HT:PCBM before and after annealing treatment measured by AFM. It is apparent that the surface roughness of P3HT:PCBM was increased due to the vertical phase separation during annealing treatment, the PC 60BM molecular could easily penetrate into the bulk of active layer and left some hollows on the film surface during annealing treatment, as shown in the Fig. 6-b.

In order to confirm the effect of annealing treatment on the vertical phase separation of P3HT:PCBM film, energy-dispersive X-ray analysis (EDX) was carried out on the same P3HT:PCBM film before and after annealing treatment. The elements associated with characteristic peaks have been identified for clarity. The characteristic element is sulfur to identify P3HT due to the absence of sulfur in PCBM molecule. The chemical structures of P3HT and PCBM are shown in Fig. 7. The weight and mol percent of elements C, O and S was measured and summarized in the Table 1. The weight and mol percent of C and S were decreased due to the oxidation of P3HT and PCBM during the annealing treatment process. Therefore, the content of O was slightly increased after annealing treatment. It is worthwhile to mention that the mol content ratio of carbon to sulfur is about 198.5 before annealing treatment, which was decreased to 195.5 after annealing treatment based on the same sample. It means that the content of P3HT or sulfur element in the top surface of P3HT:PCBM film was increased after annealing treatment, which further demonstrates the vertical phase separation [33-35]. The EDX results accords with that of XRD changing along with the annealing treatment. The vertical phase separation of P3HT:PCBM should be beneficial to the charge carrier’s collection at their individual electrode in this kind of inverted polymer solar cells.

<table>
<thead>
<tr>
<th>Elements</th>
<th>C (wt%)</th>
<th>O (wt%)</th>
<th>S (wt%)</th>
<th>C:S (mol%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Before annealing</td>
<td>93.32</td>
<td>5.41</td>
<td>1.26</td>
<td>198.7</td>
</tr>
<tr>
<td>After annealing</td>
<td>95.36</td>
<td>4.15</td>
<td>0.48</td>
<td>195.5</td>
</tr>
</tbody>
</table>

Table 1  
Element analysis of P3HT:PCBM thin film before and after annealing treatment by EDX method. (wt.%: weight percent; Am%: elemental mol percent; C:S: mol percent).
All the IPSCs have the same cell configuration ITO/Cs2CO3/P3HT:PCBM/MoO3/Al and the only difference is the annealing treatment processes. The J–V curves of IPSCs with different annealing treatment processes under illumination 100 mW/cm² are shown in the Fig. 8. The IPSCs without any annealing treatment on the active layer and MoO3/Al layer was named as cell A. The other cells were annealed after spin-coating the active layer, which have the once annealing treatment after spin-coating active layer, named as cell B. It is apparent that the performance of Cells B is much better than that of Cell A, which should be attributed to the decrease of shunt resistance and series resistance of Cell B induced by vertical phase separation. Cells C were annealed again after deposition MoO3 layer, and cells D had been the third times annealing treatment after deposition Al layers. It should be noted that the transmission of a 8 nm thick MoO3 layer is between 80% and 90%, therefore about 100 nm thick Al layer on MoO3 layer could reflect the irradiant light in the active layer to obtain the second absorption [36,37]. The key parameters, including Voc, Jsc, FF, PCE, series resistance and shunt resistance of our fabricated IPSCs are summarized in the Table 2. It is known that series resistance (Rs) of solar cells depends on resistance of the semiconductor bulk, the metal electrodes, and the metal/semiconductor interfaces. The shunt resistance (Rsh) is due to recombination of charge carriers at the donor/acceptor interface and near electrodes [38]. According to the J–V curve, the value of Rs and Rsh could be simply calculated by

$$ R_s = -\left( \frac{dV}{dJ} \right)_{V=V_{oc}}, \quad \text{and} \quad R_{sh} = -\left( \frac{dV}{dJ} \right)_{J=J_{sc}} $$

The cells A show the largest Voc and the smallest Jsc compared to the cells with annealing treatment. The limited Jsc are mainly attributed to the weak absorption and charge carrier transporting in the active layer and poor the interfacial contact. The larger Voc should be attributed to the poor interfacial contact between the active layer and MoO3 layer along with the larger shunt resistance (13,420 Ω/cm²). Wang et al. also reported that the Voc could be sharply increased from 0.6 V to 1 V when the interfacial contact changed from Ohmic to Schottky contact [16,39]. Carriers can gain or lose energy during their transport and collected by individual cathode due to the band bending of the electrostatic potential at the interface between active layer and cathode layer. It is generally believed that the difference in potential between the LUMO of acceptor and the HOMO of donor affects Voc when the contact is perfect Ohmic contact (non-injection in the reverse bias mode). This perfect Ohmic contact, however, cannot be achieved in practical cells. And the difference in work function between the electrodes has the major effect on the Voc [40]. Scharber et al. reported that the Voc of a cell based on a blend of donor polymer and PC60BM is dictated to the following relation: [41]

$$ V_{oc} = \frac{1}{e} \left( E_{LUMO} - E_{HOMO} \right) - 0.3 $$

The ideal Voc of OSCs based on the P3HT:PCBM system should be 1.2 V, 0.3 V is an empirical data. However, most of reports give the Voc between 0.58 and 0.67 V, which are less than the theoretical value [30,42-45]. The losses are mainly attributed to the interfacial contact and intra-resistance of cells.

Fig. 9 shows the J–V characteristic curves of the four kinds of cell in dark. The cell A shows an almost similar current dependence on the driving voltage under the forward and reverse bias. Under the same driving voltages, the current density of cell A is less than the other cells and the cell C shows the best diodes performance.

For the IPSCs with annealing treatment on the active layers, the Jsc had apparent increase compared with cells A. The optimal performance of cells C by both annealing treatment on the active layer and MoO3 layer have been achieved, resulting in the Voc of 0.57 V, Jsc of 8.8 mA/cm² and FF of 38.7% and the maximum PCE of 1.96%. The annealing treatment on the MoO3 layer is beneficial to the interfacial contact between active layer and MoO3 layer and the increase of FF. The increase of FF was originated from the decrease of series resistance (Rs) after annealing treatment resulting from the improvement of interfacial contacts between active layer and MoO3 layer. This reduction in Voc and the increase in Jsc provide confirmation that the photo-induced electrons and holes are efficiently separated within the composite and collected by the contact electrodes. For the cell D with annealing treatment

![Fig. 8. Current density versus voltage (J-V) curves of cells with different annealing treatment processes under illumination 100 mW/cm².](image)

![Fig. 9. Current density (mA/cm²) versus voltage (V) characteristic curves of cells with different annealing treatment processes.](image)

### Table 2

Photovoltaic parameters of inverted polymer solar cells with different annealing treatments.

<table>
<thead>
<tr>
<th>Number</th>
<th>Voc (V)</th>
<th>Jsc (mA/cm²)</th>
<th>FF (%)</th>
<th>PCE (%)</th>
<th>Rs (Ω/cm²)</th>
<th>Rsh (Ω/cm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>0.77(±0.02)</td>
<td>1.3(±0.5)</td>
<td>32.2(±0.5)</td>
<td>0.32(±0.2)</td>
<td>13420</td>
<td>3116</td>
</tr>
<tr>
<td>B</td>
<td>0.59(±0.02)</td>
<td>6.5(±0.3)</td>
<td>37.5(±0.2)</td>
<td>1.44(±0.2)</td>
<td>3525</td>
<td>480</td>
</tr>
<tr>
<td>C</td>
<td>0.77(±0.02)</td>
<td>8.8(±0.2)</td>
<td>38.7(±0.2)</td>
<td>1.96(±0.1)</td>
<td>3044</td>
<td>358</td>
</tr>
<tr>
<td>D</td>
<td>0.51(±0.01)</td>
<td>4.6(±0.2)</td>
<td>29.9(±0.1)</td>
<td>0.70(±0.2)</td>
<td>2337</td>
<td>954</td>
</tr>
</tbody>
</table>

Numbers in parentheses are standard deviation for 12 individual devices, each device has 8 cells with the active area about 0.09 cm².
after deposition Al cathode, the PCE was dramatically decrease from 1.96% to 0.7% along with the decrease of $V_{oc}$, $J_{sc}$, and FF. This reduction of photovoltaic performance could be attributed to two terms: i) the energy level alignment variation of MoO$_3$/Al induced by annealing treatment; ii) the diffusion of Al atoms crossing the MoO$_3$ thin layer during the annealing treatment, resulting in Al cathode directly contacting with active layer. It is known that the post annealing treatment (after deposition of Al cathode) on the conventional PSCs exhibited the improved photovoltaic performance when the annealing treatment is less than 150 °C, it means that the Al layer could not be destroyed during the lower temperature annealing treatment [46,47]. Therefore, the diffusion of Al atoms crossing the MoO$_3$ thin layer forms the new channels for collecting charge carriers. The work function (4.2 eV) of Al cathode is much lower than the energy level (5.2 eV) of MoO$_3$/Al layer, which decrease the intra-electric field strength adjusted by the both electrodes and is not beneficial to the charge carrier’s collection. The $J_{sc}$ of IPSCs was evidently decreased from 8.8 to 4.6 mA/cm$^2$ by annealing treatment after deposition Al layer, which could further support the understanding about the effect of annealing treatment after deposition Al layer on the performance of IPSCs.

4. Conclusions

A series of IPSCs with Cs$_2$CO$_3$ modified ITO as the electrode and MoO$_3$ modified Al as the anode was fabricated and then carried out several step annealing treatments. The Cs$_2$CO$_3$ dissolved in 2-ethoxyethanol modified ITO exhibited snowflake-like morphology and were annealed at 150 °C for 20 min to adjust the work function for the better electron collection. The IPSCs with twice annealing treatments on the active layer and MoO$_3$ layer show the maximum PCE approaching to 2%, with $V_{oc}$ of 0.57 V, $J_{sc}$ of 8.87 mA/cm$^2$ and high FF of 38.7%. However, the photovoltaic performance of the IPSCs was dramatically decreased due to the further annealing treatments after deposition Al layer, which may be due to the diffusion of Al atoms crossing the MoO$_3$ thin layer during annealing treatment and then forming new channels for charge carrier collection. The new channels are not beneficial to the charge carrier collection. EDX and XRD results directly co-demonstrated the vertical phase separation of P3HT:PCBM induced by annealing treatment. The inverted configuration of cells could take full advantage of vertical phase separation to improve the performance of polymer solar cells. The inverted configuration could also improve the stability and lifetime of cells in the air without encapsulation [48]. The IPSCs may be the idealized devices for the application of polymer solar cells.

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