Effects of Cathode Confinement on the Performance of Polymer/Fullerene Photovoltaic Cells in the Thermal Treatment

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Abstract—Polymeric photovoltaic (PV) cells based on poly(3-hexylthiophene-2,5-diyl):[6, 6]-phenyl C61 butyric acid methyl ester (P3HT:PCBM) with the cathode confinement in the thermal treatment show better performance than the PV cells without the cathode confinement in the thermal treatment. The functions of the cathode confinement are investigated in this paper by using X-ray photoelectron spectroscopy, atomic force microscopy, optical absorption analysis, and X-ray diffraction analysis. It is found that the cathode confinement in the thermal treatment strengthens the contact between the active layer and the cathode by forming Al–O–C bonds and P3HT-Al complexes. The improved contact effectively improves the device charge collection ability. More importantly, it is found that the cathode confinement in the thermal treatment greatly improves the active layer morphology. The capped cathode effectively prevents the overgrowth of the PCBM molecules and, at the same time, increases the crystallization of P3HT during the thermal treatment. Thus, a better bicontinuous interpenetrating network is formed, which greatly reduces the exciton loss and improves the charge transport capability. Meanwhile, the enhanced crystallities of P3HT improve the absorption property of the active layer. All these aforementioned effects together lead to the great performance improvement of polymeric PV cells.

Index Terms—Cathode confinement, contact, morphology, organic solar cells.

I. INTRODUCTION

POLYMERIC photovoltaic (PV) cells are of tremendous interests due to their attractive properties such as flexibility, ease of fabrication, low materials, and energy budget [1]–[8]. However, compared with the inorganic counterparts, the performance of polymeric PV cells is severely limited by the exciton loss and poor charge transport. These challenges can be effectively overcome through the use of the bulk heterojunction structure because it can simultaneously guarantee effective exciton dissociation and carrier transport if a proper bicontinuous interpenetrating network is formed in the active layer [2]–[5]. The detail of the interpenetrating network or, to say, the morphology is essentially important for the performance of polymeric PV cells. In order to achieve an optimal morphology, a thermal treatment is usually utilized in the device fabrication, particularly for the widely used poly(3-hexylthiophene-2,5-diyl):[6,6]-phenyl C61 butyric acid methyl ester (P3HT:PCBM) PV devices [3]–[21]. The thermal treatment can be carried out before [18]–[21] and after [3]–[18] the electrode deposition. Both the methods can greatly improve the device performance [3]–[21]. The functions of the thermal treatment have been extensively investigated, and it has been shown that the morphology will be rearranged through the nanoscale phase separation between donor and acceptor components during the thermal treatment. By carefully optimizing the thermal treatment condition, an optimal interpenetrating network can be formed, which greatly improves the charge transport property. In addition, the thermal treatment can also effectively enhance the crystallization of P3HT [3]–[5], [9]–[11], which will increase the hole mobility and the optical absorption capability. Due to the importance of the thermal treatment for P3HT:PCBM PV devices, great efforts have been devoted into the study of the thermal annealing process in the past few years [3]–[5], [9]–[11]. How the thermal annealing ambient, thermal annealing temperature, and thermal annealing time affect the device performance has been well studied. However, only very few studies paid attention to the role of cathode in the thermal treatment. As is known, the thermal treatment can be done before [18]–[21] and after [3]–[18] the cathode deposition, and both methods can greatly improve the device performance. The unique difference between them is whether there is cathode confinement in the thermal treatment or not. Although most of the previous studies [3]–[18] have tended to use the cathode confinement and carry out the thermal treatment after the cathode deposition [3]–[18], what the functions of the cathode confinement in the thermal treatment are and how they affect the device performance are still not well studied.

In this paper, the effects of cathode confinement on the performance of polymeric PV cells are investigated. It is shown that better device performance can be achieved by using the cathode confinement in the thermal treatment. The
experimental analysis indicates that, by capping the cathode before the thermal treatment, the Al–O–C bonds and P3HT–Al complexes are formed at the interface between the P3HT:PCBM active layer and the cathode, which leads to a better contact and thus improves the charge collection capability. More importantly, the cathode confinement in the thermal treatment greatly improves the active layer morphology. It is shown that the cathode confinement in the thermal treatment can effectively inhibit the overgrowth of the PCBM molecules and, at the same time, increase the crystallization of P3HT. Thus, better morphology is achieved, which effectively reduces the exciton loss and improves the charge transport capability. Meanwhile, the enhanced P3HT crystallites improve the absorption property of the active layer. All these effects contribute to improve the device performance.

II. EXPERIMENTAL

Fig. 1 shows the layer structure of our polymeric PV cells and the chemical structures of P3HT and PCBM. All the devices were fabricated on the indium tin oxide (ITO)-coated glass substrates. Briefly, after being sequentially cleaned with detergent, de-ionized water, acetone, and isopropanol in an ultrasonic bath for about 15 min, the dried ITO glass substrates were treated with oxygen plasma for about 3 min. Then, the filtered poly(3, 4-ethylenedioxythiophene):poly (styrene sulfonate) (PEDOT:PSS) (Baytron P VP AI 4083) suspension (through 0.45-μm filter) was spun coated on top of the ITO surface to form a 100-nm layer under ambient condition and dried at 120 °C in an oven for about 1 h. P3HT:PCBM solution dissolved in 1,2-dichlorobenzene with a weight ratio of 1:0.8 was then spun coated on the PEDOT:PSS layer in the glove box to form a 100-nm blend layer. A 100-nm Al cathode was further thermally evaporated through a shadow mask, giving an active device area of 20 mm². In order to investigate the effects of the cathode confinement on the device performance in the thermal treatment, two different types of devices were investigated: the devices without the cathode confinement in the thermal treatment (anneal the devices before the cathode deposition) and the devices with the cathode confinement in the thermal treatment (anneal the devices after the cathode deposition). The thermal treatment was carried out by annealing the devices in the glove box at the optimized temperature of 160 °C for about 10 min as our previous report [22]. For reference, the devices without any thermal treatment were also fabricated.

III. RESULTS AND DISCUSSION

The current–voltage (J–V) characteristics were measured by a Keithley 2400 source-measure unit under AM 1.5 solar illumination at an intensity of 100 mW/cm² calibrated by a Thorlabs optical power meter. The X-ray photoelectron spectroscopy (XPS) samples consisted of an identical sandwiched structure: ITO-coated glass/P3HT:PCBM(100 nm)/Al (3 nm). Because XPS is a surface chemical analysis technique, the XPS spectra were measured with the samples to the chamber of a Kratos AXIS HSi spectrometer at once. The operating pressure of the analysis chamber was maintained at 8 × 10⁻⁹ torr. A 1486.71-eV monochromatic Al Kα X-ray gun source was used to achieve the Al 2p, O 1s, C 1s, and S 2p spectra. Tapping-mode atomic force microscopy (AFM) measurements were taken with a Nanoscope III A (Digital Instruments) scanning probe microscope. The samples were prepared in the same sequence as the XPS samples. The phase images and the line scanning profiles of the samples were then recorded under air operation. For both the optical absorption study and X-ray diffraction measurement, the thin films of P3HT:PCBM in the same thickness of 100 nm were spun cast on the microscope slides. The optical absorption study was recorded by a Shimadzu UV-3101 PC UV-VIS-NIR scanning spectrophotometer. The X-ray diffraction measurement was carried out by the θ – 2θ scan method with CuKα radiation (λ = 0.1542 nm) operated at 30 kV and 30 mA using a Shimadzu X-ray diffractometer.

Fig. 2 shows the J–V characteristics of the devices with the same configuration of ITO/PEDOT:PSS/P3HT:PCBM/Al. For the device without any thermal treatment, it shows the PV response with the short-circuit current density (JSC) of 5.12 mA/cm², open-circuit voltage (VOC) of 0.58 V, fill factor (FF) of 47.63%, and power conversion efficiency (PCE) of 1.41%. The device performance is greatly improved by the thermal treatment. However, there are obvious differences for the devices with and without the cathode confinement in
TABLE I
SUMMARY OF THE PARAMETERS EXTRACTED FROM THE J–V CURVES SHOWN IN FIG. 2

<table>
<thead>
<tr>
<th>Samples</th>
<th>$V_{OC}$</th>
<th>$J_{SC}$</th>
<th>FF</th>
<th>PCE</th>
<th>$J_0$</th>
<th>$J_{ph}$</th>
<th>$n$</th>
<th>$R_{sh}$</th>
<th>$R_S$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Without thermal treatment</td>
<td>0.58</td>
<td>5.13</td>
<td>47.64</td>
<td>1.42</td>
<td>2.75e-4</td>
<td>5.32</td>
<td>2.32</td>
<td>778.25</td>
<td>29.00</td>
</tr>
<tr>
<td>Without cathode confinement</td>
<td>0.58</td>
<td>7.50</td>
<td>57.13</td>
<td>2.49</td>
<td>4.80e-5</td>
<td>7.62</td>
<td>1.89</td>
<td>575.19</td>
<td>9.34</td>
</tr>
<tr>
<td>With cathode confinement</td>
<td>0.60</td>
<td>8.34</td>
<td>62.25</td>
<td>3.12</td>
<td>3.03e-5</td>
<td>8.40</td>
<td>1.88</td>
<td>617.28</td>
<td>4.43</td>
</tr>
</tbody>
</table>

Units of parameters, $V_{OC}$: $V$; $J_{SC}$, $J_0$ and $J_{ph}$: mA/cm$^2$; FF and PCE: %; $R_{sh}$ and $R_S$: Ω cm$^2$.

the thermal treatment, as shown in Fig. 2 and Table I. For the device without the cathode confinement in the thermal treatment, it shows the performance of $J_{SC} = 7.50$ mA/cm$^2$, $V_{OC} = 0.58$ V, $FF = 57.13\%$, and $PCE = 2.49\%$. However, further performance improvement is observed for the device with the cathode confinement in the thermal treatment, which shows a better performance of $J_{SC} = 8.34$ mA/cm$^2$, $V_{OC} = 0.60$ V, $FF = 62.57\%$, and $PCE = 3.12\%$. It can be seen that the cathode confinement in the thermal treatment effectively increases $J_{SC}$ and $FF$, which makes the overall $PCE$ improved by 25%. This trend was found for a series of cells. Similar results are recently reported by Kim et al. [24], where they also observed that the device with thermal treatment after cathode deposition could show better performance. This further confirms our experimental results.

In order to understand the functions of the cathode confinement in the thermal treatment, the electrical parameters need to be extracted. The $J$–$V$ characteristics of organic PV cells can be approximately described by the Shockley equation

$$J = J_0 \left( e^{\frac{q(V - R_S J)}{n k_B T} - 1} \right) + \frac{V - R_S J}{R_{sh}} - J_{ph}$$

(1)

where $J_0$, $J_{ph}$, $R_s$, $R_{sh}$, $q$, $n$, $k_B$, and $T$ are the saturation current density, photocurrent density, series resistance, shunt resistance, electron charge, ideality factor, Boltzmann constant, and temperature, respectively. By fitting the Shockley equation (Fig. 2), the estimated parameters are extracted and listed in Table I. It is shown that $R_s$ of the device with the cathode confinement in the thermal treatment is greatly reduced, compared with the device without the cathode confinement in the thermal treatment (from 9.34 to 4.43 Ω cm$^2$). $R_s$ can significantly affect the device performance, and reducing the value of $R_s$ is an efficient method to increase PCE [25]. The reduced $R_s$ by using the cathode confinement plays one main role for the significant performance improvement of polymeric PV cells. $R_s$ is directly related to the contacts between the cathode and the active layer. Thus, these contacts were addressed by the XPS measurement.

The interfacial analysis results obtained by XPS measurement are shown in Fig. 3. Each top curve and bottom curve in the Al 2p, C 1s, O 1s, and S 2p core-level spectra graphs are corresponding to the samples with and without cathode confinement in the thermal treatment. As shown in Fig. 3, both samples show the Al 2p spectrum peaks located at the binding energy (BE) of 74.95 and 74.6 eV, which correspond to the Al oxide and Al–O–C bond, respectively, by referring to Table II. The Al–O–C bond is also confirmed by the peaks located at the BE of 286.2 eV in the C 1s spectrum and 531 eV in the O 1s spectrum, as shown in Fig. 3. Studies [27], [32], [33] have indicated that the Al–O–C bond is formed by the reaction of Al atoms and the carbonyl groups in PCBM, and its existence will improve the contact between the polymer and the metal [4] for both samples. However, by using the cathode confinement in thermal treatment, there is an additional shoulder peak at the BE of 76 eV in the Al 2p spectrum, and an additional shoulder peak at the BE of 162.4 eV in the S 2p spectrum.
additional chemical bond. The additional chemical bond signal can also be seen from the S 2p spectrum. Although the typical peaks of P3HT appeared at the BE of 164.1 eV (2p1/2) and 165.3 eV (2p3/2) due to the spin-orbit coupling are observed for both samples in the S 2p spectrum, there is an extra shoulder peak at the BE of 162.4 eV for the sample by using the cathode confinement. Considering the donation of electron density from the Al metal to the thiophene ring of P3HT [29], [32], these additional peaks suggest that the interaction between P3HT and the Al metal occurs by using the cathode confinement in the thermal treatment.

Since the direct reaction between the Al atoms and the sulfur atoms is unlikely to occur because of the inherently high electron density on these sites, it is suggested that the Al atoms form bonds with the carbon atoms on the thiophene ring in the positions adjacent to the sulfur atom and form the P3HT-Al complex. One possible structure of the P3HT-Al complex is proposed in Fig. 4. The formation of the P3HT-Al complex will change the electron density of the sulfur atoms [30], [35]. In the P3HT-Al complex, the overall charge density of the sulfur atoms is smaller than that of the pristine P3HT. Thus, the S 2p peaks located at the BE of 164.1 and 165.3 eV are shifted to the higher BE side at 164.3 and 165.5 eV, respectively, for the sample with the cathode confinement in the thermal treatment. Although the P3HT-Al complex is formed, there is only a slight energy difference (≈0.1-eV shift in BE) in the C 1s spectrum for both samples, as shown in Fig. 3. This is because the C 1s peak is dominated by the aliphatic carbon atoms while the Al atoms preferentially react with the carbon atoms in the conjugated system (thiophene ring of P3HT in this case) [31], [32], [34]. The signal that arose from the interaction between P3HT and Al is too weak to affect the C 1s spectrum of the sample with cathode confinement. This explains why only very small energy difference in the C 1s spectrum is observed. The exact structure of P3HT-Al complex needs to be ascertained by further experiments, and this will be our future work.

It has been reported that Al metal can effectively transfer the electron to the conjugated polymer with the sulfide species, and this feature makes it as a potential cathode for polymeric electronics [27]. Another study [23] also has reported that Cu can react with P3HT and form sulfidelike species. The formed sulfidelike species can improve the solar cell performance. It is believed that the formation of the P3HT-Al complexes will play the same role. With the help of P3HT-Al complexes and the Al–O–C bonds, there is a better contact between the electrode and the active layer. This improved contact effectively reduces $R_s$ and results in the improvement of the device performance.

How $R_s$ affects the device performance is clearly shown in Fig. 5. It is shown that a large $R_s$ will induce the decrease in $F_F$ and $J_{SC}$. By reducing $R_s$, $F_F$ and $J_{SC}$ are increased, and thus, the device performance is improved. At the same time, it is also noted that, although both $F_F$ and $J_{SC}$ can be affected by $R_s$, their dependences on $R_s$ are different. From Fig. 5, it can be seen that $F_F$ can be greatly adjusted by $R_s$ when the value of $R_s$ is just larger than 1.0 $\Omega \cdot \text{cm}^2$. The decrease in $R_s$ from 9.34 to 4.43 $\Omega \cdot \text{cm}^2$ (Table I) should be the main reason for the increase in $F_F$ from 57.13% to 62.25% (Table I) for the sample by using the cathode confinement. However, there is no obvious change of $J_{SC}$ observed until $R_s$ is larger than 25 $\Omega \cdot \text{cm}^2$ (Fig. 5). Since $R_s$ of the two devices are relative low (9.34 and 4.43 $\Omega \cdot \text{cm}^2$, respectively, Table I), it seems that the decrease in $R_s$ is not the main reason for the obvious increase in $J_{SC}$ (from 7.5 to 8.34 mA/cm$^2$, Table I) by using the cathode confinement. This conclusion is also confirmed by the extracted parameter of $J_{ph}$. $J_{ph}$ is mainly determined by the properties of the active layer and only slightly depends on $R_s$ [independent parameters in (1)]. If the cathode confinement in the thermal treatment is only to improve the contact and reduce $R_s$, there should be no such obvious change in $J_{ph}$ from 7.62 to 8.40 mA/cm$^2$, Table I). Thus, there must be other more important factors aside from $R_s$, which lead to the obvious increase in $J_{ph}$. It is well known that $J_{ph}$ is very sensitive to the device morphology and material absorption, and thus, these aspects should be well addressed.

The effects of cathode confinement on the device morphology are first investigated by the AFM measurement. Because the interface between the active layer and the cathode is mainly enriched by PCBM upon thermal treatment [1], [10], [16], the evolution of the surface morphology directly reflects the change in the PCBM domains. As shown in Fig. 6, it is shown that the thermal treatment effectively leads to the growth of the PCBM domains and thus increases the root mean square roughness. However, comparing to the device without the cathode confinement, there is a smoother surface morphology for the device with the cathode confinement. As shown in the AFM phase images [Fig. 6(b) and (c)], there is a smaller island...
size for the sample with the cathode confinement. The profile measurements [Fig. 6(e) and (f)] also show that the average peak-to-peak height and the width of the surface morphology are reduced by 20% and 33% by using the cathode confinement. Since surface morphology change is mainly induced by the aggregates of PCBM [1], [10], [16], the smoother surface morphology means that the cathode confinement can prevent the formation of too large underlying PCBM domains.

It is well known that the main roles of annealing process are to induce the redistribution of PCBM [10], [16], [36], [37] and increase the crystallization of P3HT, so that the bicontinuous interpenetrating networks is achieved, and meanwhile, the optical absorption capability is enhanced. However, a too fast PCBM diffusion will lead to the formation of very large PCBM aggregates and thus destroy the optimal bicontinuous interpenetrating network [9], [16], [38]–[40]. In addition, too large PCBM domains also reduce the interfacial contact area between P3HT and PCBM, and lead to the inefficient exciton dissociation [16], [38]. In order to achieve a high performance, it is required to well control the PCBM domain size. It is shown here that the overgrowth of the PCBM domains in the thermal treatment is effectively inhibited by using the cathode confinement. Thus, better nanoscale morphology control is achieved. A similar metal confinement effect was also demonstrated on the organic surface by using silver cap [41]. The improved morphology will decrease the exciton loss, facilitate the charger transport, and thus increase $J_{SC}$.

$J_{SC}$ is also directly related to the optical absorption of the active layer. In order to investigate the effects of cathode confinement on the optical absorption capability, the UV-Vis absorption spectra of the active layer capped with the Al electrode were measured. Because the annealed metal results in a slight variation of the light absorption, the optical spectra were obtained by subtracting the pure metal spectra. The results are shown in Fig. 7. All the samples show the typical absorption spectrum of the P3HT:PCBM blend film with the absorption peak at the wavelength of 515 nm and shoulders at 550 and 604 nm. Similar with the previous studies [9], [10], the thermal treatment obviously increases the optical absorption of the P3HT:PCBM film. However, there is better optical absorption capability for the sample with the cathode confinement (e in Fig. 7), compared with the sample without the cathode confinement (d in Fig. 7). It is well known that the absorption capability of the P3HT:PCBM system is directly related to the P3HT crystallites [3]–[5], [9], [10]. The crystallization of P3HT was measured by XRD.
TABLE III
SUMMARY OF X-RAY DIFFRACTION PEAKS OF P3HT:PCBM FROM FIG. 8

<table>
<thead>
<tr>
<th>Samples</th>
<th>2θ [°]</th>
<th>Δ2θ [°]</th>
<th>h [counts/s]</th>
<th>L [nm]</th>
<th>d [nm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Without thermal treatment</td>
<td>5.49</td>
<td>0.83</td>
<td>318</td>
<td>9.6</td>
<td>1.61</td>
</tr>
<tr>
<td>Without cathode confinement</td>
<td>5.44</td>
<td>0.61</td>
<td>596</td>
<td>13</td>
<td>1.625</td>
</tr>
<tr>
<td>With cathode confinement</td>
<td>5.44</td>
<td>0.45</td>
<td>617</td>
<td>17.7</td>
<td>1.625</td>
</tr>
</tbody>
</table>

Fig. 8 shows the obtained X-ray diffraction (XRD) measurement results. A characteristic peak around 2θ = 5.4° is observed for all the samples, which is associated with the lamella structure of thiophene rings in P3HT [42]. Based on Bragg’s law [43] and Scherrer relation [44], the lattice constant d and the size of the polymer crystallites L can be determined as

\[ n\lambda = 2d \sin \theta \]

(2)

\[ L = \frac{0.9\lambda}{\Delta 2\theta \cos \theta} \]

(3)

where \( \lambda \) is the wavelength of the X-ray, \( \theta \) is Bragg’s angle, \( \Delta 2\theta \) is the smallest full-width at half-maximum of the peak.

The extracted d and L are listed in Table III. It is shown that all the samples show the lattice constant of 1.62 ± 0.01 nm that represents the P3HT crystallites in α-axis orientation [43]. Thermal treatment increases the crystallization of P3HT. However, the increased magnitudes are different for the devices with and without the cathode confinement. The sample with the help of the cathode confinement in the thermal treatment shows the highest peak. As listed in Table III, the size of the P3HT crystallites (L value of 17.7 nm) is increased by 36% by using the cathode confinement, compared with that without the cathode confinement (L value of 13 nm). The increased crystallite size may come from the effective inhibition of the strong PCBM diffusion by the cathode confinement. It has been shown [9], [45] that a too strong diffusion of PCBM from the P3HT matrix would reduce the P3HT crystallization and optical absorption property. Because of the presence of the cathode in the thermal treatment, the PCBM diffusion is slowed down. Thus, it is easier for P3HT to be crystallized. The increased P3HT crystallites will enhance the active layer optical absorption capability and increase \( J_{SC} \).

IV. CONCLUSION

P3HT:PCBM PV cells with the cathode confinement in the thermal treatment show better performance than the PV cells without the cathode confinement in the thermal treatment. The effects of the cathode confinement on the device performance have been investigated in this paper. According to the XPS results, it is found that the Al–O–C bonds and P3HT–Al complexes are formed at the interface between the active layer and the cathode by using the cathode confinement. These chemical structures effectively reduce the contact resistance and improve the device performance. More importantly, the cathode confinement effectively improves the active layer morphology. According to the AFM, UV-Vis absorption spectra, and XRD measurement results, it is found that the cathode confinement in the thermal treatment not only prevents the overgrowth of the PCBM domains but also increases the crystallization of P3HT. With the help of cathode confinement in the thermal treatment, a better optical absorption and a more ideal bicontinuous interpenetrating network can be obtained at the same time. This will effectively reduce the exciton loss and improve the charge transport capability. Thus, an improved device performance is achieved.

REFERENCES


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