Selective Morphology Control of Bulk Heterojunction in Polymer Solar Cells Using Binary Processing Additives

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Supporting Information

ABSTRACT: We report the effect of binary additives on the fabrication of polymer solar cells (PSCs) based on a bulk heterojunction (BHJ) system. The combination of 1,8-diiodooctane (DIO), a high-boiling and selective solvent, for fullerene derivatives and poly(dimethylsiloxane) (PDMS) precursor, a nonvolatile insulating additive, affords complementary functions on the effective modulation of BHJ morphology. It was found that DIO and PDMS precursor each play different roles in the control of BHJ morphology, and thus, the power conversion efficiency (PCE) can be further enhanced to 7.6% by improving the fill factor (FF) from 6.8% compared to that achieved using a conventional device employing only a DIO additive. In the BHJ of the active layer, DIO suppressed the large phase separation of PBDTTT-CF and PC71BM while allowing the formation of continuous polymer networks in the donor polymer through phase separation of the PDMS precursor and BHJ components. The appropriate amount of PDMS precursor does not disturb charge transport in the BHJ despite having insulating properties. In addition, the dependence of photovoltaic parameters on different light intensities reveals that the charge recombination in the device with DIO and PDMS precursor decreases compared to that achieved using the device with only DIO.

KEYWORDS: polymer solar cells, bulk heterojunction, PDMS resin, DIO, binary additives

INTRODUCTION

In bulk heterojunction (BHJ) polymer solar cells (PSCs), the morphology of the photoactive layer influences the separation of excitons and transport of separated charge carriers to their respective electrodes. Therefore, morphology control is a crucial factor that determines device performance.1-2 The BHJ consisting of electron donor and acceptor materials should form nanoscale interpenetrating networks due to exciton diffusion length within 10 nm.3 To control the nanoscale morphology of the active layer, various methods have been used such as thermal annealing, solvent annealing, and introduction of interfacial layer and solvent additives.4-6 Among the methods to control the morphology of active layer, the introduction of solvent additives is not only very effective but also one of the simplest. Various additives such as 1,8-octanediol (ODT), chloronaphthalene (CN), and 1,8-diiodooctane (DIO) have been employed to improve phase separation and to control the morphology by enhancing the crystallinity and by suppressing oversized phase separations.7-9 In many cases, solvent additives are essential, for example, for the BHJ-containing a poly(thieno[3,4-b]thiophene/benzodithiophene) (PTB7) system because the large phase separation between the donor and acceptor materials results in poor device performance.10 DIO in particular has been used as a solvent additive to prevent large phase separation between the donor and acceptor while significantly changing the morphology of the active layer by enhancing the intercalation of [6,6]phenyl C71 butyric acid methyl ester (PC71BM) into PTB7.11-13 Also, the insulating additives such as poly(dimethylsiloxane) (PDMS),14,15 (3-chloropropyl)trimethoxysilane (CP3MS),16 and poly(methyl methacrylate) (PMMA)17 have been used to decrease the film roughness and to induce a more homogeneous morphology with fewer vacancies and less charge recombination.

Even though introducing additives is an attractive method to improve device performance, a single additive dominantly influences one phase, making detailed control of the morphology difficult. Recently, binary additives have been introduced to further optimize and to more finely tune the morphology.18 When binary additives are introduced, the...
appropriate combination of additives should be carefully considered to improve device performance.\textsuperscript{19,20} Liu et al. have investigated the effect of binary additives in PSCs based on a low band gap polymer by changing the ratio of CN and ODT. The crystallinity of the donor polymer when the optimum ratio of CN and ODT was applied facilitated charge transport and led to high short-circuit current ($J_{sc}$) and fill factor (FF).\textsuperscript{21} Therefore, the introduction of well-chosen binary additives has the potential to further optimize device performance.

Herein, we fabricated PSCs with a poly[4,8-bis-substituted-benzod[1,2-b:4,5-b’]dithiophene-2,6-diy1-alt-4-substituted-thieno[3,4-b’][thiophene-2,6-diy1]fluorine (PBDTTT-CF):PC$_{71}$BM BHJ and various mixtures of the binary additives, DIO and PDMS precursor. The device performance is enhanced from 6.8\% to 7.68\% when fabricated with binary additives because the addition of DIO and PDMS precursor further optimized the morphology of the BHJ active layer. DIO suppressed the large phase separation of PBDTTT-CF and PC$_{71}$BM, and PDMS precursor induced the formation of continuous polymer networks in PBDTTT-CF through phase separation. In addition, we found that charge recombination in the device fabricated with DIO and PDMS precursor is reduced compared to that in the device fabricated only with DIO because of the dependence of photovoltaic parameters on different light intensities.

\section*{EXPERIMENTAL METHODS}

The cleaned ITO/glass substrates were treated under UV/O$_3$ for 15 min. The PEDOT:PSS (Clevious P VP Al 4083, Heraeus) was deposited by spin coating at 5000 rpm for 40 s on the ITO substrates. Then, all substrates were annealed at 150 °C for 10 min in air. To prepare the BHJ active layer, PBDTTT-CF (1-material, PDI = 2.2, molecular weight = 58 000), PC$_{71}$BM (Nano-C), and PDMS precursor (Dow Corning Sylgard 184) were used. To optimize the performance of the device, we fabricated devices with various combinations of donor and acceptor materials. For the most optimized device, PBDTTT-CF:PC$_{71}$BM (9 mg:18 mg) was dissolved in 1 mL of chlorobenzene with 5 vol % DIO and then was deposited on the substrates by spin-coating at 1500 rpm for 40 s. For devices using PDMS precursor, 20 mg of PDMS precursor was dissolved in 1 mL of chlorobenzene and stirred at 60 °C for 24 h. The PDMS precursor solution was then blended from 1 vol % to 5 vol % in the same solution used for the optimized device described above. The resulting solution with PDMS precursor was then deposited by spin-coating at 1500 rpm for 40 s on the PEDOT:PSS substrate. Finally, a 20 nm layer of Ca (1–2 Å/s deposition rate) and a 100 nm layer of Al (4–7 Å/s deposition rate) were deposited through a shadow mask (with an active area of 4.64 mm$^2$) by thermal evaporation. The current density–voltage ($J$–$V$) of each device was measured using a Keithley 2400 digital source measurement unit under 100 mW cm$^{-2}$ irradiation with a simulated AM 1.5 G spectrum. This measurement system is calibrated by standard Si solar cell certified by International System of Units (SI) (SRC 1000 TC KG5 N, VLSI Standards, Inc.). The external quantum efficiency (EQE) spectrum was measured by a QEX-7 PV Measurements Inc. spectral response system. To measure the mobility of the hole and the electron, a hole only device with an ITO/PEDOT/active layer/MoO$_3$/Ag architecture and an electron only device with an Al/active layer/Al architecture were also fabricated. The surface morphology of the fabricated devices was imaged using transmission electron microscopy (TEM) (Technai G2 S-Twin 3000 keV electron microscope) and atomic force microscopy (AFM) (Veeco Dimension 3100 atomic force microscope) using tapping mode. The atom concentration was analyzed by X-ray photoelectron spectroscopy (XPS) using a monochromatic Al Kα light source (AXIS NOVA, Kratos). The interface films were measured by a contact angle measurement system (Phoenix-300 Touch, SEO Co., Ltd., Republic of Korea).

\section*{RESULTS AND DISCUSSION}

Figure 1a presents the structural formulas of the photoactive materials in the BHJ and of the additives used in this study, with a schematic structure of the PSC provided in Figure 1b. We used a new type of insulating additive, PDMS precursor, with DIO to create a new binary additive for fabrication. In this BHJ system, the DIO additive is essential to prevent large aggregates of PC$_{71}$BM from forming.\textsuperscript{10} The selective solubility of DIO in PC$_{71}$BM and its high boiling point (332.5 °C at 760 mmHg) allow favorable intercalation of PC$_{71}$BM acceptor into the donor polymer domains, improving the surface area of the interfaces between the donor polymer and PC$_{71}$BM.\textsuperscript{11,12} This DIO-assisted BHJ morphology leads to efficient exciton dissociation and charge transport, thereby resulting in high performing PSCs. On the other hand, the PDMS precursor consists of silicone elastomers for the polymerization of polydimethylsiloxane (PDMS). Previous studies have shown that PDMS induces the fine and continuous interpenetrating network in small molecule based organic solar cells.\textsuperscript{22–24} Also PDMS precursor facilitates the formation of networked conjugated polymer pathways and exhibits improved mobility in the organic field effect transistor and thus is an attractive candidate to control the morphology of the BHJ active layer.\textsuperscript{25} On the basis of these results, we tried to further optimize the BHJ morphology using these complementary properties of the binary additives.

The PSC devices with active layers based on PDBTTT-CF and PC$_{71}$BM with binary additives were fabricated as follows. To finely control the amount added, 20 mg of PDMS precursor was dissolved in 1 mL of chlorobenzene for 24 h at 60 °C, and various volumes of that solution from 1 vol % to 5 vol % were blended into the active solution. Figure 2 provides...
representative current density–voltage ($J$–$V$) curves for a PSCs fabricated from PBDTTT-CF:PC$_{71}$BM and our binary additives by comparing the conventional DIO additive. Table 1 also summarizes the average and the upper limit of the characteristics of the devices. When the 3 vol % PDMS precursor solution is added, the device shows the best performance; the average power conversion efficiency (PCE) increased from 6.59% to 7.28% while enhancing FF from 63.13% to 68.39%. The best of these devices exhibits a $V_{oc}$ of 0.73 V, a $J_{sc}$ of 14.87 mA/cm$^2$, a FF of 70.04%, and a PCE of 7.68%. From the device characteristics, we confirmed that the main factor that enhances the device performance is an increase in FF. The FF of device with added PDMS precursor exhibited a value of 68% on average and an upper limit of 70%. There is negligible deviation in the external quantum efficiency (EQE) intensity between devices with only DIO and with DIO and PDMS precursor as shown in Figure 2b, which indicates that the absorption and the charge extraction abilities of device are not influenced by the insulating PDMS precursor additive. This result is in good agreement with the value obtained from the $J$–$V$ curve showing values of 14.68 mA/cm$^2$ and 14.64 mA/cm$^2$ for devices with only DIO and with DIO and PDMS precursor, respectively.

In order to understand why the device performance improved, transmission electron microscopy (TEM) measurements were carried out to investigate the morphology of the active layer as shown in Figure 3. Figure 3a and Figure 3b show

Table 1. Device Performance of PSCs with Various Amounts of PDMS Precursor

<table>
<thead>
<tr>
<th>condition</th>
<th>$V_{oc}$ (V)</th>
<th>$J_{sc}$ (mA/cm$^2$)</th>
<th>FF (%)</th>
<th>PCE (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ref</td>
<td>0.72 (0.73)</td>
<td>14.38 (14.13)</td>
<td>63.13 (65.78)</td>
<td>6.59 (6.80)*</td>
</tr>
<tr>
<td>1% PDMS precursor</td>
<td>0.71</td>
<td>14.94</td>
<td>64.03</td>
<td>6.88</td>
</tr>
<tr>
<td>3% PDMS precursor</td>
<td>0.74 (0.73)</td>
<td>14.34 (14.87)</td>
<td>68.39 (70.04)</td>
<td>7.28 (7.62)*</td>
</tr>
<tr>
<td>5% PDMS precursor</td>
<td>0.73</td>
<td>14.15</td>
<td>68.09</td>
<td>7.08</td>
</tr>
<tr>
<td>7% PDMS precursor</td>
<td>0.74</td>
<td>14.29</td>
<td>63.79</td>
<td>6.81</td>
</tr>
<tr>
<td><em>The best PCE.</em></td>
<td></td>
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</table>

Figure 2. (a) $J$–$V$ curves and (b) external quantum efficiencies (EQE) of the PSCs based on PBDTTT-CF:PC$_{71}$BM with DIO and with DIO and PDMS precursor additives.

Figure 3. The first row contains the TEM images of PBDTTT-CF:PC$_{71}$BM (a) without DIO and PDMS precursor, (b) with DIO and without 3% PDMS precursor, (c) without 5% DIO and with PDMS precursor, and (d) with 5% DIO and with 3% PDMS precursor. The second row shows the TEM images of PBDTTT-CF (e) without DIO and PDMS precursor, (f) with DIO and without 3% PDMS precursor, (g) without 5% DIO and with PDMS precursor, and (h) with 5% DIO and with 3% PDMS precursor.
the blended film of PBDTTT-CF:PC$_{71}$BM without additives and with only the DIO additive. As shown in Figure 3a, PC$_{71}$BM has large aggregates with grains of around 200–300 nm in diameter, which suppressed charge separation in the donor and acceptor interface. The large domains of PC$_{71}$BM disappeared, and the donor and acceptor are evenly blended with added DIO as shown in Figure 3b. When PDMS precursor is added, the size and shape of PC$_{71}$BM domains do not change, and continuous bold lines appear behind the grains of PC$_{71}$BM indicating that the PDMS precursor influenced the donor polymer more selectively as shown in Figure 3c and Figure 3d. To further clarify the impact of PDMS precursor on the donor polymer, we examined the TEM images of donor polymer without the PC$_{71}$BM acceptor. Figure 3e shows a contrast difference dependent on the charge density despite having a film of pure PBDTTT-CF with darker networks of polymer, suggesting a highly aggregated polymer. The networks observed in the pure PBDTTT-CF film are untangled by the addition of DIO as shown in Figure 3e and Figure 3f. The polymer networks become more obvious and continuous with the PDMS precursor additive and do not disappear with the addition of DIO as shown in Figure 3g and Figure 3h. Surface phase images of the PBDTTT-CF obtained from atomic force microscopy (AFM) are consistent with the morphologies observed in TEM images as shown in Figure S1 in Supporting Information. Unlike finely dispersed morphology of film with only DIO, continuously connected network morphology is observed in film with DIO and PDMS precursor as shown in Figure S1, parts b and d. However, we could not observe distinct difference between films with and without PDMS precursor from GIWAXS and UV–vis spectra as shown in Figure S2, which is similar to results from previous papers. In the GIWAXS analysis of PTB base polymer:PC$_{71}$BM films with and without DIO, the orientation and crystallinity have almost same property despite the significant change of BHJ morphology. Likewise we anticipate the PDMS precursor modified the BHJ morphology inducing aggregation of polymer; however it did not affect the crystallinity and orientation morphology. To distinguish the effect of PDMS precursor on the donor and acceptor, TEM images of the PC$_{71}$BM acceptor without the donor polymer were measured as shown in Figure S3. Compared to the donor polymer film with PDMS precursor, the networks are not observed in the PC$_{71}$BM film with PDMS precursor. From these results, we confirmed that DIO suppresses the aggregation of PC$_{71}$BM and PBDTTT-CF whereas PDMS precursor induces the formation continuous networks in the donor polymer. The optimized morphology of the active layer should have good interpenetration of the donor and acceptor materials for efficient exciton separation and an effective continuous pathway for charge transport. The addition of binary additives better matches the above criteria, and as a result, it generated improved FF and PCE.

To investigate the presence and the distribution of PDMS precursor in the device, X-ray photoelectron spectroscopy (XPS) was employed. Table 2 shows the distribution of atomic concentrations in the top and bottom regions of the films with only DIO and with DIO and PDMS precursor. We identified that PDMS precursor is a nonvolatile additive and is mainly distributed in the top region of the film. The Si atom concentration of the film without PDMS precursor is zero, while in the film with PDMS precursor, the concentration of Si atoms is 6.14% in the top region and 1.48% in the bottom region. Most of the blended PDMS precursor migrates to air leading to vertical phase separation of the active layer. The vertical phase separation could be induced by the surface energy difference of the PDMS precursor with the BHJ consisting of the donor and acceptor materials. PDMS precursor has a relatively low surface energy, which is more hydrophobic compared to the donor and acceptor materials. The film with PDMS precursor has a higher contact angle compared to the film without PDMS precursor as shown in Figure S4, which means that the surface becomes more hydrophobic with the addition of PDMS precursor. During spin coating, the blended PDMS precursor becomes excluded from the polymer due to this surface energy difference and facilitates the refinement of the polymer, leading to continuous polymer networks through polymer aggregation. In addition, Moule et al. reported that the addition of poor solvent in P3HT:PCBM solution causes the P3HT aggregate and partial crystallinity. It is possible that the PDMS precursor can act as poor solvent to PBDTTT-CF:PC$_{71}$BM and induce it to increase aggregation.

To investigate the effect of an insulating additive on the charge transport, we calculated the charge mobility of the films with only DIO and with DIO and PDMS precursor using space charge limited current (SCLC) measurements as shown in Figure 4. The values of the electron mobility are 1.42 × 10$^{-4}$ cm$^2$ V$^{-1}$ s$^{-1}$ and 1.62 × 10$^{-4}$ cm$^2$ V$^{-1}$ s$^{-1}$, and the values of the hole mobility are 2.65 × 10$^{-4}$ cm$^2$ V$^{-1}$ s$^{-1}$ and 2.67 × 10$^{-4}$ cm$^2$ V$^{-1}$ s$^{-1}$ for devices with only DIO and with DIO and PDMS precursor, respectively. There are no significant changes to the electron and the hole mobility despite the blending of the insulating additive into the active layer. These results show that the blending in an insulating additive of adequate amount does not disturb the electrical properties of the device.

In order to further understand the characteristics of the device fabricated using binary additives, the electron−hole recombination mechanism needs to be investigated. The photocurrent density can be expressed as follows:

$$I_{ph} = qL G(I) P_{(I,V)}$$  \(1\)

where $q$ is elemental charge, $L$ is the thickness of active layer, $G(I)$ is the generation rate of bound electron−hole pairs per unit volume, and $P_{(I,V)}$ is the charge collection probability. Figure 5a and Figure 5b show a set of normalized photocurrents characteristic of devices with only DIO and with DIO and PDMS precursor under different irradiation intensities, which shows $P_{(I,V)}$ on voltage and light intensities. From the $J$–$V$ curves shown in Figure 5a and Figure 5b, it can be presumed that $I_{ph}$ is saturated at ~0.5 V, $P_{(I,V)} = 1$ because all the charge carriers are swept out with this large reverse bias. The $P_{(I,V)}$ is strongly dependent on light intensity, indicating that geminate and nongeminate recombination dominate in the

<table>
<thead>
<tr>
<th>atom</th>
<th>with DIO</th>
<th>with DIO and PDMS precursor</th>
</tr>
</thead>
<tbody>
<tr>
<td>F</td>
<td>1.31</td>
<td>0.93</td>
</tr>
<tr>
<td>O</td>
<td>11.27</td>
<td>11.11</td>
</tr>
<tr>
<td>C</td>
<td>83.35</td>
<td>78.35</td>
</tr>
<tr>
<td>S</td>
<td>4.06</td>
<td>3.47</td>
</tr>
<tr>
<td>Si</td>
<td>0</td>
<td>6.14</td>
</tr>
</tbody>
</table>

Table 2. Atomic Concentrations in the Top and Bottom Regions of Films with DIO and with DIO and PDMS Precursor.
device with only DIO as shown in Figure 5a. Conversely, the $P_c(I,V)$ is independent of light intensity from $-0.5$ to $0.4$ V, indicating that the efficiency loss in this range is dominated by geminate recombination. A distinct discontinuity is observed from $0.4$ to $V_{oc}$ indicating that nongeminate recombination dominates in this region for a device with DIO and PDMS precursor, suggesting that nongeminate recombination is reduced by the PDMS precursor from $-0.5$ to $0.4$ V. Moreover, under the open-circuit condition, the recombination mechanism of BHJ PSCs can be explored in detail because all photogenerated carriers recombine at this point. When the dominant loss mechanism is nongeminate recombination, $V_{oc}$ can be simply described as

$$V_{oc} = \frac{kT}{q} \ln(P_{light}) + \text{constant}$$

where the slope of $V_{oc}$ vs $\ln(P_{light})$ will be $kT/q$. In contrast, when the geminate recombination is dominant, the slope will increase to $2kT/q$. The slopes of $V_{oc}$ vs $\ln(I)$ are $1.27kT/q$ for the device with DIO and $1.50kT/q$ for the device with DIO and PDMS precursor as shown in Figure 5c. From these results, we would expect that the devices have nongeminate and geminated recombination; however, the geminate or trap-assisted recombination decreases with the addition of PDMS precursor to active layer. The continuous network of the donor polymer reduces the trap sites by forming a favorable percolation pathway for charge transport. Also, a great gap filling property of PDMS precursor due to its molecular flexibility might have suppressed the formation of trap sites and facilitate the charge transport. These data clearly show that the enhanced device performance with PDMS precursor is caused by reduced charge recombination.

$\Box$ CONCLUSIONS

In this work, we improved the performance of a BHJ PSC device using the binary additives DIO and PDMS precursor. The blended binary additives each have a different influence on the morphology of the BHJ film. DIO suppresses the formation of large domains of PC$_7$BM with fine untangled morphology, while the PDMS precursor induces the formation of the continuous networks in donor polymer selectively upon phase separation. From the electron and hole mobility measured by SCLC, we confirmed that the blended PDMS precursor does not disturb the electronic properties of the device and that the mobility of the hole and the electron are well balanced. In addition, measurements of the dependence of photovoltaic
parameters on light intensity revealed that nongeminate and geminate recombination decreases for the device with binary additives compared to a device using only one additive. Further optimization of the film morphology and the reduction of charge recombination via binary additives enhance the device performance from 6.59% to 7.28%, and the best device showed up to 7.62% with a high FF. These results demonstrate that the introduction of binary additives could be a promising method to further optimize the morphology of the BHJ and further enhance device performance.

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**Notes**

The authors declare no competing financial interest.

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