Rapid phase segregation of P3HT:PCBM composites by thermal annealing for high-performance bulk-heterojunction solar cells

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Abstract The performances of bulk-heterojunction (BHJ) solar cells are investigated for time-dependent thermal annealing with different morphology evolution scales, having special consideration for the diffusion and aggregation of fullerene derivative molecules based on blends of poly(3hexylthiophene):[6,6]-phenyl-C₆₁-butyric acid methyl ester (P3HT:PCBM). Meaningfully, rapid formation of dot-like and needle-like crystalline PCBM structures of a few micrometers up to 60 µm in size is obtained with thermal annealing treatment from 2 to 15 min, which dynamically reflects a fast process of PCBM molecule and cluster aggregation. Upon ultrasonic-assisted processing and annealing treatment, the scale of P3HT crystals is drastically increased in view of X-ray diffraction (XRD) patterns, leading to a high hole mobility. And, the P3HT domains can be gradually converted into larger P3HT crystals approved by the decreased full width at half-maximum in the XRD patterns. Corresponding current-voltage curves are measured in quantity and we propose a model to explain the effect of the crystalline degree of P3HT domains and aggregation of PCBM molecules and clusters on the phase segregation, expressing a viewpoint towards high performance of BHJ solar cells.

1 Introduction

In the past years, bulk-heterojunction (BHJ) solar cells based on organic materials have become very popular, since they have many advantages such as cheapness, light weight, flexibility, and ease of fabrication of large areas to harvest the Sun's energy [1-4]. Recently, single solar cells with a considerable power conversion efficiency (η) beyond 6% have been reported [5, 6]. However, poly(3hexylthiophene) (P3HT) is still one of the most efficient systems in power conversion efficiency. In this system, controlling phase segregation of poly(3-hexylthiophene):[6,6]phenyl-C₆₁-butyric acid methyl ester (P3HT:PCBM) blends is critical for optimizing the power conversion efficiency of BHJ solar cells [7-9]. On the one hand, favorable phase segregation can lead to higher interfacial area between domains of the electron donor P3HT and acceptor PCBM (D/A), enhancing exciton dissociation. On the other hand, high crystalline scale of P3HT induces benefits to increasing the mobility of dissociated holes [10], greatly improving the ability of carrier collection by electrodes. If serious phase segregation occurs, however, it is abundant to contact crystalline P3HT and PCBM molecules and clusters, and charge recombination will dominate in turn [11, 12]. And, because of the aggregation of PCBM clusters to form dot-like and needle-like PCBM crystalline structures, these devices will suffer a decrease in efficiency [13]. Therefore, there exists an optimal scale of phase segregation for P3HT and PCBM domains for achieving the highest solar cell performances. However, the mechanism influencing the solar cell performances by phase segregation is not clear yet. Previous works have reported the formation of dot-like or needle-like crystalline PCBM structures [13–19]. The growth mechanism of the PCBM structures within the film is described in terms of diffusion of PCBM towards the PCBM crystals,

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leaving highly crystalline P3HT behind in the surrounding matrix [16]. A recent finding by Yao et al. illuminated the PCBM clusters of about 200 nm, which were discovered with transmission electron microscopy (TEM) to be a direct result of the 'intelligent' phase separation. The PCBM clusters not only provide a percolation pathway for better electron transport in the PCBM phase, but also enable better hole transport in the P3HT phase [20]. Afterwards, Kim et al. obtained P3HT nanowires (NWs) with a uniform diameter of 20-25 nm by the ultrasonic-assisted self-assembly method [21]. A stronger diffraction peak induced by the selfassembled P3HT NWs with ultrasonic treatment than that of the spin-cast film without ultrasonic treatment was obtained, which benefited to increasing the short-current density (J_{sc}) . Thus, we consider that PCBM clusters, P3HT crystals, and the micro-aggregation of PCBM co-determine the scale of phase segregation.

To explain the performance variation according to phase segregation, we prepared different degrees of phase segregation of P3HT:PCBM blends by controlling the annealing time. In our study, evolved morphologies and structures of P3HT:PCBM blends are strongly influenced by the short-time annealing treatment. The crystalline degree of P3HT domains and aggregation of PCBM clusters were investigated and the results were correlated with the performances of the devices. Based on the above results, we supply a route to understand how to achieve high performances by phase segregation, which is influenced by the morphologies and structures of P3HT:PCBM blends.

2 Experimental details

2.1 Solar cell device fabrication

A blend solution was prepared by dissolving P3HT (Rieke, regioregularity: 95%) and PCBM (Nano-C) in chlorobenzene (CB). The weight ratio of these materials was P3HT:PCBM = 1.0:1.0 in chlorobenzene (1.0 ml). To fabricate the BHJ solar cells (see Fig. 1), a molybdenum oxide (MoO₃) layer of about 10 nm was deposited on fluorinated tin oxide (FTO) substrates at the substrate temperature of 100°C by radio frequency (RF) magnetron sputtering from a Mo metal target. The MoO₃ thin films were deposited at the relative oxygen partial pressure $O_2/(Ar + O_2)$ of 50%. All the films were deposited at a total pressure of 0.5 Pa and a RF power of 130 W. Then, the P3HT:PCBM active layer was deposited by spin coating at 1000 rpm on the MoO₃ film.

Before spin coating the active layer, the solution was put under ultrasonic treatment in 100 W for 120 s, which produces a uniform P3HT nanowire [21], avoiding the effect of inhomogeneous P3HT aggregation on PCBM diffusion



Fig. 1 Device fabricated in this work: FE-SEM image (*bottom*) shows a tilted cross section of P3HT:PCBM solar cells (note that the interface between active layer and FTO is less clear owing to a thin thickness)

and aggregation. Next, these samples were put into a vacuum chamber to deposit Al electrodes (100-nm thick) via thermal evaporation at the pressure of 10^{-5} Pa. Finally, the devices were annealed at 150°C from 2 to 15 min in a dry argon glove-box (<1 ppm O₂ and <1 ppm H₂O).

2.2 Characterization

The film thickness was measured by a surface profilometer (Talysurf Series II). Film crystal structure was characterized by X-ray diffraction (XRD, Bruker AXS, D8 Advance) using CuK α radiation at 40 kV and 40 mA. The spectral transmittances of the P3HT:PCBM layers were recorded using a UV-VIS-NIR spectrophotometer. Optical microscope (OP) images were measured by a Leica MPS 30 microscope. The morphology of the P3HT:PCBM active layer was characterized by atomic force microscopy (AFM, SPM-9500J3, Shimadzu, Japan) and field emission scanning electron microscopy (FESEM, FEI XL-30). Photo-energy conversion characterization was recorded by a source meter (model 2400, Keithley Instruments Inc., USA) under an illumination intensity of 100 mW/cm² (Oriel 91192, AM 1.5, Global). From this, the performance parameters of the device were calculated.

3 Results and discussion

3.1 Optical and structural properties

Figure 2 shows the UV-visible absorption spectra of the P3HT:PCBM active layers with different annealing times. In the spectra, the position of the PCBM absorption peak at



Fig. 2 The UV-visible absorption spectra of the P3HT:PCBM active layer without annealing and annealed with various annealing times from 2 to 15 min



Fig. 3 The XRD results of the P3HT:PCBM films without annealing treatment and annealed with different annealing time

330 nm does not shift in wavelength with the increase of annealing time. In addition, with additional annealing, the absorption intensity of P3HT:PCBM between 300 and 650 nm gradually increases, and the spectrum becomes broader as well as more vibronic and red shifted from 485 nm to 505 nm, due to a change in the state of P3HT aggregation from amorphous to crystalline. At the same time, the interaction between the P3HT molecules becomes stronger, thus improving the optical absorption in the low photon energy region [22, 23].

Figure 3 shows changes of the relative XRD intensities at the specific position $2\theta = 5.4^{\circ}$, corresponding to the *a*-axis orientation of the P3HT crystals at various annealing times. The P3HT:PCBM composite films prepared from CB solution are almost amorphous. Upon thermal annealing, the scale of P3HT crystals is increased, resulting in a stronger interchain interaction [24] and an enhanced hole mobility [25]. In fact, it comes from the improved dielectric constant of the surrounding medium, ε_r , which determines the efficient dissociation of electron-hole pairs by decreasing their mutual Coulomb attraction, V:

$$V = \frac{e^2}{4\pi\varepsilon_0\varepsilon_r r} \tag{1}$$

where *e* is the charge of an electron, ε_0 is the permittivity of vacuum, ε_r is the dielectric constant of the surrounding medium, and *r* is the electron–hole separation distance [26]. The crystallinity of P3HT domains is determined by the peak (100) intensity, and the size of P3HT crystallite is determined mainly by the full width at half-maximum (FWHM). In Fig. 3, the peak intensity of P3HT (100) gradually increases as the FWHM of the peak decreases, indicating not only the formation of more ordered crystalline P3HT domains, but also the achievement of larger size of P3HT crystals. The crystal size can be calculated with Scherrer's equation:

$$D_{hkl} = \frac{0.89\lambda}{\beta_{hkl}\cos\theta_{hkl}} \tag{2}$$

where λ is the X-ray wavelength, θ_{hkl} is the Bragg diffraction angle, and β_{hkl} is the FWHM in radians of the main peak in the XRD patterns. The mean crystallite sizes are distinctly found to be 14.8, 17.8, and 23.8 nm in the films corresponding with 5-, 10-, and 15-min thermal treatment, respectively. The role of different crystalline sizes will be discussed in the following. In the annealing treatment, there is a fast formation of crystalline P3HT, which might drive the diffusion and aggregation of PCBM molecules in a drastic process, subsequently pushing PCBM clusters away from the preferred locality to accelerate the formation of large PCBM aggregation.

3.2 P3HT:PCBM film morphology

Figure 4 shows optical micrographic images of the P3HT:PCBM films in the devices without annealing treatment and with annealing treatment from 2 to 15 min. It is clearly observed that large micro-aggregation structures of PCBM appear everywhere in active layers after annealing treatment, and grow from dot-like crystals to needlelike crystals with the increase of annealing time from 2 to 15 min. Previous works have also reported the similar structures of the PCBM aggregation, which extremely depends on regioregularity of P3HT [14], the quality of the solvent [15], the annealing temperature [16], and the P3HT:PCBM composite ratios [17], etc. Similar dot-like and needle-like crystalline PCBM structures by thermal annealing were also observed by Miyanishi et al. [13] after a longer annealing time (>4 h) at 150°C than ours, which might be due to their lower concentration of the solution, leading to forming crystalline PCBM clusters in a harder fashion by a longer evaporation of solvent. In Fig. 4, upon thermal annealing for

Fig. 4 OP and SEM images of P3HT:PCBM blend films without thermal annealing (**a**) and annealed with different annealing times for 2 min (**b**), 5 min (**c**), 10 min (**d**, **f**) and 15 min (**e**). Scale bar: 50 µm



Fig. 5 AFM images of P3HT:PCBM films spin-coated from chlorobenzene without annealing (**a**) and annealed for 2 min (**b**), 5 min (**c**), 10 min (**d**), 15 min (**e**). AFM height images of devices showing a 5 μm × 5 μm surface area

2 min, PCBM crystalline clusters join together to form dotlike PCBM aggregation structures. When providing a driving force with additional annealing, many larger needle-like PCBM structures (Fig. 4d and e) appear, due to the combination of mutual dot-like PCBM crystals and local PCBM clusters. Therefore, the optical micrographs present the diffusion of PCBM towards the PCBM crystal aggregation in the P3HT:PCBM matrix, which would induce an extreme phase segregation with sufficient annealing.

To investigate the morphological change in smaller scale, AFM measurements were carried out. Figure 5 shows the corresponding surface topography of the P3HT:PCBM films without annealing and with the various annealing times. A relatively rough surface (see Fig. 5a) with a root-mean

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square (RMS) of 1.34 nm is observed for the as-prepared devices without annealing. Upon thermal annealing in argon, however, the RMS increases to 1.62, 3.65, 3.85, and 4.16 nm for 2, 5, 10, and 15 min, respectively. It has been considered that a larger RMS is a 'signature' of high-efficiency solar cells in 'thermal annealing' [20]. In Fig. 5, we observe that highly improved RMS occurs at 5 min, which might illuminate that higher performances are achieved after 5-min annealing. With additional annealing, the RMS increases. It quite probably will degenerate the performances, since excessive aggregation of PCBM needle structures would take up larger effective active areas of the devices [13]. In additional annealing exhibit more serious aggregation of PCBM

clusters in the bright domains, showing larger scale of phase segregation.

3.3 Device performance

Figure 6 shows the performances of solar cells fabricated without annealing treatment and with various timedependent annealing treatments. The performance parameters of solar cells are summarized in Table 1. More than 50 devices were fabricated and measured to confirm the trends in each performance parameter. As one can clearly see from the results, devices fabricated without annealing treatment show relatively poor performances: the open-circuit voltage is 0.58 V, the short-circuit current density is 2.70 mA cm $^{-2}$, the fill factor (FF) is 0.42, and η is 0.66%. Upon annealing treatment, the performances of the devices have a larger evolvement, depending on the crystalline scale of the P3HT and morphologies as observed in XRD, OP, and AFM images. The parameters, such as J_{sc} , FF, and η , all remarkably increase with additional annealing from 2 to 10 min, originating from the enhanced absorption of the incident light and the higher-ordered P3HT crystals. Upon annealing for 15 min, however, it in turn degrades the device performances even with the increased absorption and ordering of P3HT domains. In that way, what induces the performance degradation?



Fig. 6 The current–voltage curves of solar cell devices without annealing and annealed with various annealing times from 2 to 15 min

 Table 1
 Summary of the performance parameters of solar cell devices

 without annealing and with various annealing time from 2 to 15 min

Device	$V_{\rm oc}$ (V)	$J_{\rm sc}~({\rm mA/cm^2})$	FF	η (%)
a (0 min)	0.58	2.70	0.42	0.66
b (2 min)	0.56	6.21	0.54	1.87
c (5 min)	0.56	9.40	0.56	2.94
d (10 min)	0.56	10.53	0.61	3.60
e (15 min)	0.56	8.77	0.58	2.84

3.4 Influencing factors on cell performance

As previously mentioned, thermal annealing treatment usually induces two evolution processes: (i) crystallization or chain stacking of P3HT and (ii) diffusion and aggregation of PCBM molecules into the blend [27]. In our study, due to the sufficient crystallinity of P3HT domains upon 10-min annealing, it is not likely that the hole mobility will be sequentially dominant to the device performances. The crystalline scale of P3HT domains will be quenched into stability and cannot be remarkably improved upon 10-min annealing. This can be concluded from the peak-intensity variation in XRD (see Fig. 3). Therefore, the second process will determine the solar cell performances. However, there are still several questions remaining unanswered: (i) what is the function of the diffusion and aggregation of PCBM molecules and clusters? (ii) How does they determine the device performances during the thermal annealing process? (iii) Can we find new factors which also affect the performances?

In previous reports, Orimo et al. validated the fact that PCBM is dominant at the Al interface replacing P3HT in post-annealing measured by X-ray photoelectron spectroscopy [28]. The aggregation of PCBM molecules can serve as a hole-blocking layer at the Al interface to affect the device performances. Kumar et al. also demonstrated that the thermal annealing step increases the PCBM concentration in the top few nanometers of the active layer due to PCBM diffusion [29]. After thermally evaporating an about 7-nm PCBM thin film on the P3HT:PCBM layer, a great increase in efficiency was found due to the enhanced electron transport and extraction near the Al cathodes. Our work also illustrates that a longer and larger needle-like aggregation of PCBM as well as a surface with a gradually improved RMS upon additional annealing have been formed, indicating that more PCBM molecules and clusters have participated in the PCBM aggregation structures. Recently, nondestructive techniques, such as neutron reflectivity (NR) and small angle neutron scattering (SANS), were used to probe the vertical morphology of the P3HT:PCBM films [30-32]. In NR measurements, it is found that the freshly cast films have a surface layer that is relatively depleted in PCBM, while being relatively rich in PCBM at the film-substrate interface. However, thermal annealing can increase the PCBM concentration at the surface to a level found in the blend film. The PCBM-depleted layers found in 'as-cast' P3HT:PCBM thin films are partly responsible for their relatively poor performance in BHJ solar cells, and thermal annealing of BHJ solar cells improves device efficiency not only by the creation of a percolated PCBM network, but also by improving electron extraction via increasing the PCBM concentration next to the device cathode [30]. Moreover, using SANS, P3HT:PCBM film morphology can also be demonstrated. It



is shown that thermal annealing has the effect of increasing the PCBM agglomerate size and volume fraction, suggesting a coarsening of the phase-separated morphology. The PCBM will accumulate at the air interface creating an improved pathway for electrons to leave the device through the deposited metal electrode. Compared with the as-cast sample, the thermally annealed sample is a substantially better solar cell with improved short-circuit current, fill factor, and efficiency [32]. Combining all the above and our mentioned results, it is possible to illustrate the origin of deteriorated performances in the process of extreme phase segregation by the following model, as sketched in Fig. 7. After 10-min annealing, a richer PCBM distribution at the top is formed in the P3HT:PCBM matrix (see Fig. 7a). Upon annealing for 15 min, the aggregation of mutual clusters occurs and induces a decrease of PCBM molecule layer and PCBM clusters (see Fig. 7b). As an interface of exciton separation and a channel of dissociated electrons away from the interface to the cathode, the decrease of the PCBM molecule layer and clusters will seriously decrease the separation interface of a charge-transfer (CT) exciton, thus deteriorating the solar cell performances.

Moreover, the phase segregation with PCBM aggregation in the P3HT:PCBM matrix is usually related to exciton diffusion, which is short lived and has a typically ~ 10 nm diffusion length [33, 34]. When a singlet exciton is excited via absorbing photons, it can diffuse to the donor-acceptor interface to form an interfacial charge-transfer state with an electron-hole separation distance r (the thermalization length). In the latter, dissociation occurs if the electronhole pairs are sufficient to overcome the energetic driving force, i.e., the offset in the lowest unoccupied molecular orbitals (LUMOs) or the highest occupied molecular orbitals (HOMOs) at the D/A interface [35]. Onsager theory predicts that the thermalization length will be critical to the full CT exciton dissociation into the free-charge carriers. Extreme phase segregation induces an increase of P3HT crystallite size from 17.5 to 23.8 nm in our study. If the crystallite sizes are too large, the limited diffusion length of the photoinduced excitons means that some excitons will not reach an interface within their lifetime [26]. Previous works reported that the crystallite sizes corresponding to high performances are typically below 20 nm [21, 22]. Therefore, we consider that the length of exciton diffusion is sensitive to the mean crystallite size. Large crystallite size provides a 'stumbling block' for singlet exciton diffusion to the P3HT:PCBM interface (see Fig. 7b), and even the formation of a bound at the interface, stronger Coulomb attraction induced by the shorter thermalization length cannot overcome the energetic driving force, the offset in PCBM LUMOs and P3HT HO-MOs at the D/A interface. Thus, the CT exciton cannot be dissociated effectively and will recombine mostly, leading to a relatively poor performance.

Finally, this degradation in device performances is partially caused by micro-aggregation of PCBM, possibly deteriorating the interface contact of the active layer and the Al electrodes [13]. Thus, high-performance BHJ solar cells by annealing treatment can be obtained by controlling the crystalline size of P3HT domains and avoiding extreme aggregation of PCBM molecules and clusters.

4 Conclusion

The influence of the scale of phase segregation of P3HT:PCBM blends on the performances of the BHJ solar cells was investigated. By controlling thermal annealing times, rapid phase segregation was induced in a short time. The UV-visible absorption and XRD results demonstrate that the crystalline degree of P3HT domains is improved with increasing annealing time. From the optical microscopy analysis, increasing the annealing time results in the formation of dot-like and needle-like crystalline PCBM structures of a few micrometers up to 60 μ m in size, which illuminates that more PCBM molecules and clusters have participated in the micro-aggregation structures. In AFM images, the mutation of surfaces with an improved RMS

occurs at 5-min annealing, which also supports the aggregation process and shows that favorable phase segregation was formed with 5-min annealing. When thermal annealing for 10 min was applied, the optimized performance of the devices was achieved with a maximum η of 3.60%. If providing the additional annealing treatment, however, it in turn degrades the device performances even with the increased absorption and ordering of P3HT domains, due to not only a decrease of PCBM molecule layer and PCBM clusters induced by extreme phase segregation, but also a 'stumbling block' provided by the larger PCBM crystallite sizes for singlet exciton diffusion to the P3HT:PCBM interface. Therefore, the crystalline scale of P3HT domains and the aggregation of PCBM clusters should be considered a universal feature of high performances of BHJ solar cells. These results also suggest that moderate crystalline size of P3HT domains and aggregation of PCBM molecules and clusters have an important role to improve the BHJ solar cell performance.

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