Investigation of Charge Accumulation States in Polymer Solar Cells using Light-Induced Electron Spin Resonance Spectroscopy

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Charge accumulation states in organic solar cells were studied in detail by a microscopic characterization with light-induced electron spin resonance (ESR) spectroscopy during device operation using a typical polymer solar cell of indium-tin oxide (ITO)/poly(3,4-ethylenedioxythiophene):poly (4-styrenesulfonate) (PEDOT:PSS)/regioregular poly(3-hexylthiophene) (P3HT):fullerene [6,6]-phenyl C₆₁-butyric acid methyl ester (PCBM)/LiF/Al. Two light-induced ESR signals with a narrow and broad linewidth were observed where the broad component with a slow accumulation rate clearly correlated with the deterioration of the device performance. From the ESR analysis, the charge accumulation state causing the device deterioration is ascribed to holes at P3HT polymer-chain ends with residual bromines in amorphous regions in the active layer. Preventing the charge accumulation seems indispensable to develop highly durable polymer solar cells with high performance.

Keywords: Organic solar cells, Electron spin resonance (ESR) spectroscopy, Charge accumulation, Device performance deterioration

1. Introduction

Organic solar cells are one of promising next-generation solar cells because of their printable device structure and lightweight using a low-cost and low-temperature device-fabrication process using flexible plastic substrates [1-10]. There has been a significant amount of interest in the high power conversion efficiency (PCE) over 12% for single junction cells [11,12]. In addition to the PCE improvement, device stability is an important issue for the practical use of solar cells. Several irreversible degradation mechanisms due to extrinsic factors such as oxygen and moisture have been discussed [13-15]. Also, an intrinsic reversible deterioration mechanism during device operation has been discussed for a polymer solar cell with blend films of regioregular poly(3-hexylthiophene) (P3HT) and [6,6]-phenyl C₆₁-butyric acid methyl ester (PCBM) [16,17]. The deterioration has been ascribed to an accumulation of photogenerated charges during device operation [16-20].

Elucidation of the intrinsic deterioration mechanism is an important issue because the intrinsic deterioration cannot be prevented by device sealing. In particular, microscopic investigation into the charge accumulation states is indispensable because the information is useful for preventing the charge accumulation by modifying molecular and device structures. Electron spin resonance (ESR) spectroscopy is one of the most suitable techniques for such microscopic investigation because it is capable of directly observing charge accumulation and formation in organic materials and their devices at the molecular level with a high sensitivity and precision [21-24]. This ESR method has clarified the charge formation in pentacene/C₆₀ layered thin-film solar cells during device fabrication [25,26]. For the P3HT:PCBM polymer solar cells, the molecules...
with the charge accumulation during device operation have been identified as P3HT using the ESR method [18-20]. However, the detailed charge accumulation states in P3HT have not yet been completely clarified.

Here we study the charge accumulation states in P3HT:PCBM polymer solar cells in detail. A direct observation of two types of charge accumulation states in P3HT is presented. One is due to charge accumulation in polymers with crystalline lamellar structures and the other is due to charge accumulation at polymer-chain ends with bromines with amorphous nature. The charge accumulation at polymer-chain ends is found to correlate with the performance deterioration during device operation.

2. Experimental

Commercially available P3HT (Luminescence Technology Corp., LT-S909, regioregularity: 93%), PCBM (Frontier Carbon, nanom spectra E100, purity: 99.2%), and a hole-buffer material poly(3,4-ethylenedioxythiophene):poly(4-styrenesulfonate) (PEDOT:PSS) (Clevios P AI4083) were used to fabricate the solar cells. The cell structure is indium-tin oxide (ITO)/PEDOT:PSS (~40 nm)/P3HT:PCBM (230 nm)/LiF (1 nm)/Al (80 nm). The device was fabricated by spin coating PEDOT:PSS and P3HT:PCBM solutions on an ITO substrate, followed by vacuum deposition of LiF and Al on the organic layers to form an electron-collecting electrode. Thermal annealing treatment was carried out at 110 °C for 10 min. For solar cells, only pre-annealing was performed before depositing LiF and Al. For P3HT:PCBM thin films, no annealing was done unless otherwise stated.

3. Results and discussion

3.1. Light-induced ESR signals of polymer solar cells

To directly demonstrate the charge accumulation in the fabricated polymer solar cells during device operation, we measured the light-induced ESR signals under simulated solar irradiation. The ESR spectra of the solar cell under dark conditions and under simulated solar irradiation under short-circuit conditions are shown in Fig. 1(a). The vertical axis is plotted using a unit of peak-to-peak intensity of the ESR spectrum of a standard Mn$^{2+}$ marker sample ($I_{Mn}$). In this experiment, the solar cell was measured under vacuum condition after evacuating the inside of the ESR sample tube down to 1×10$^{-4}$ Pa, followed by sealing the sample tube with a vacuum glass faucet. In this case, air possibly leaks very slightly through the faucet into the sample tube, as discussed later in detail. We mainly observed two ESR signals with $g$ factors of $g = 2.0020$ and $g = 1.9998$. The signal with $g = 2.0020$ is ascribed to P3HT radical cations (positive polarons or holes) [18-20], which increased by simulated solar irradiation. The signal with $g = 1.9998$ is ascribed to PCBM radical
anions [27], which decreased by the irradiation. The observation of the PCBM signal at room temperature has been explained by the formation of PCBM LiF⁺ charge-transfer complexes [27]. The reason for the decrease in the PCBM signal can be explained by the formation of antiferromagnetic spin pairings between PCBM radical anions and photogenerated P3HT radical cations.

The light-induced ESR spectra were evaluated by subtracting the ESR spectrum under dark conditions from that under simulated solar irradiation. Figure 1(b) shows the dependence of the light-induced ESR spectra on the duration of simulated solar irradiation. In addition to the signal with a narrow linewidth with $g = 2.0020$ (the narrow component) mentioned above, the light-induced ESR signal with a broad linewidth (the broad component) was clearly observed. The broad component increased monotonically as the duration of simulated solar irradiation increased, which is ascribed to the charge accumulation in the cell during device operation.

To investigate the origin of the charge accumulation in detail, we performed a spectrum simulation for the observed signals. Figure 1(c) shows the fitting results with a least-squares method using three fitting curves for the light-induced ESR spectrum obtained under 18 h irradiation. The red line shows a fitting curve for the broad component using fitting parameters $g = 2.0032$ and peak-to-peak ESR linewidth ($\Delta H_{pp}$) = 1.52 mT with a Gaussian lineshape. The orange line shows a fitting curve for the narrow component using fitting parameters $g = 2.0022$ and $\Delta H_{pp} = 0.21$ mT with a Gaussian lineshape. The green dotted line shows a fitting curve for the PCBM component using fitting parameters $g = 1.9999$ and $\Delta H_{pp} = 0.33$ mT with a Lorentzian lineshape. The purple line shows the sum of three curves (the total component), which explains the experimental result very well. Note that the observed light-induced ESR spectra show no anisotropy for the direction of the external magnetic field $H$ to the substrate plane, which indicates a random orientation for the charge accumulation states in the cell.

The origin of the charge accumulation is discussed using the obtained $g$ factors. The $g$ factor of 2.0022 for the narrow component is reasonably explained by the average of principal values of the $g$ tensor, $g_x = 2.00310$, $g_y = 2.00152$, and $g_z = 2.00203$, of P3HT radical cations (holes), which have been measured using P3HT crystalline thin-film field-effect transistors where $X$-, $Y$-, and $Z$-axis is parallel to the inter-lamellar direction, the polymer chain, and the $\pi$-stack direction of P3HT, respectively [28]. The average of these principal values gives $g_{ave} = 2.0022$, which is consistent with the observed $g$ factor of 2.0022. Thus, the origin of the narrow component is ascribed to holes in bulk P3HT lamellar structures with random orientation in the cell. However, the $g$ factor of 2.0032 for the broad component cannot be explained by the above $g$ tensor of P3HT radical cations because the broad component shows no anisotropy for the $H$ direction, as shown later. As discussed in the previous ESR studies, this broad component is essential for the deterioration mechanism of the cell, and its origin has been ascribed to the hole accumulation at polymer-chain ends with bromines [19,20], which occurs in amorphous region as discussed later in detail.

### 3.2. Correlation between light-induced ESR and device performance

Investigating the correlation between the light-induced ESR signal and the device performance is important for discussing the deterioration mechanism of the cells from a microscopic viewpoint. To discuss the intensity of the light-induced ESR signal, the number of spins ($N_{spin}$) was evaluated by integrating the light-induced ESR spectrum twice and by comparing the standard Mn²⁺ marker sample. Figure 2(a) shows the transient response of the $N_{spin}$ and short-circuit current density ($J_{sc}$) of the cell upon simulated solar irradiation. In our experiments, we simultaneously observed the light-induced ESR signal and the $J_{sc}$ during device operation, where a continuous-wave method with a modulation frequency of 100 kHz for the $H$, that is, lock-in detection, was used in the ESR experiments [18-20]. Thus, the photogenerated charge carriers with a lifetime of <10 μs in solar cells cannot be observed using the present ESR method [18-20]. Here we mean the term of “lifetime” for the duration of the existence of charge carriers in solar cells. Thus, we could not observe any signals of mobile charges which contributes to generate electricity. The observed light-induced ESR signals are due to photogenerated charge carriers with a lifetime of >10 μs in solar cells, namely, accumulated (or deeply trapped) photogenerated charges [18-20]. Thus, we cannot investigate whether the ESR intensity of mobile charges has a
correlation with the $J_{sc}$ or not using the present ESR method. The $N_{\text{spin}}$ gradually and monotonically increased and the $J_{sc}$ concomitantly decreased as the duration of light irradiation increased. A clear correlation between the increase in the $N_{\text{spin}}$ and the decrease in the $J_{sc}$ was observed. The $N_{\text{spin}}$ includes all contributions from the narrow, broad, and PCBM components shown in Fig. 1(c). However, if we evaluate the $N_{\text{spin}}$ due to the narrow component only, the transient response upon simulated solar irradiation shows a different behavior from that in Fig. 2(a). As shown in Fig. 2(b), the $N_{\text{spin}}$ due to the narrow component showed a rectangular response upon the irradiation, that is, the $N_{\text{spin}}$ rapidly increased and decreased after the irradiation on and off, respectively, showing an almost constant value during the light irradiation. This rapid response is related to the origin of the narrow component which is ascribed to holes in bulk P3HT lamellar structures mentioned above. Similar rectangular response was observed for the transient response of the $N_{\text{spin}}$ due to the PCBM component. These responses are completely different from the monotonic decrease in the $J_{sc}$. Note that the $N_{\text{spin}}$ value due to the narrow component is one order of magnitude smaller than that of the $N_{\text{spin}}$ due to all components. This is because the $N_{\text{spin}}$ is proportional to the square of the $\Delta H_{pp}$; the $\Delta H_{pp}$ of the broad component is much larger than those of the narrow and PCBM components. Thus, the $N_{\text{spin}}$ is mainly determined by the contribution of the broad component, which shows the clear correlation with the decrease in the $J_{sc}$. Therefore, the broad component is found to be essential for the deterioration of the cell during the device operation.

3.3. Annealing effect on charge accumulation

In order to investigate thermal annealing effect on the charge accumulation in P3HT, we investigated a thin film of ITO/PEDOT:PSS/P3HT:PCBM before and after annealing treatment. Figure 2(c) shows the transient responses of light-induced ESR intensity of the thin-film sample on simulated solar irradiation, where blue and red solid circles show the data before and after thermal annealing treatment, respectively. The data before annealing gradually increased as the duration of the irradiation increased, and hardly decreased when the irradiation was turned off. In contrast, the data after annealing showed a smaller value compared with that before annealing, and showed almost rectangular response with quick increase and decrease on the irradiation. This behavior is similar to that observed for the narrow component of the cell shown in Fig. 2(b). Therefore, we conclude that the charge accumulation in the film sample decreases by thermal annealing treatment. The thermal annealing treatment is thought to decrease the charge accumulation in the cell.

3.4. Charge accumulation states analyzed by ESR anisotropy
The charge accumulation states are discussed by analyzing the anisotropy of the ESR signal. First, the results of the thin-film sample of ITO/PEDOT:PSS/P3HT:PCBM are presented. Figure 3(a) shows the angular dependence of the $g$ factor of the ESR signal of P3HT with a narrow linewidth. These data were obtained by using the sample before annealing treatment, and by subtracting the contribution of the ESR signal of PEDOT:PSS. Blue and orange solid circles show the data before and after simulated solar irradiation, respectively. The ESR signal before the irradiation is attributed to holes due to the charge transfer at the interface between PEDOT:PSS and P3HT under dark conditions, which is caused by the difference in work functions between PEDOT:PSS and P3HT [18]. As shown in Fig. 3(a), the anisotropy of the $g$ factor before the irradiation is larger compared with that after the irradiation. This large anisotropy is attributed to the P3HT lamellar orientation. As mentioned above, the $g$ factors of holes in P3HT have been reported as $g_X = 2.00310$, $g_Y = 2.00152$, and $g_Z = 2.00203$, respectively [28]. Thus, in the case of the lamellar structure where the molecular plane is perpendicular to the substrate, the observation of $g_X = 2.00310$ is expected when the $H$ is perpendicular to the substrate. When the $H$ is parallel to the substrate, the average of $g_Y$ and $g_Z$, that is, $g = 2.00178$ is expected to be observed. Actually, the observed $g$ factor before the irradiation in Fig. 3(a) varies between 2.0028 and 2.0017. This result demonstrates that the charge transfer takes place at the interfaces between PEDOT:PSS and P3HT with ordered lamellar structure regions as shown Fig. 4(a), where P3HT seems to have high conductivity due to crystallized structures.

The origin of the ESR signal after the irradiation is ascribed to accumulated (or deeply trapped) photogenerated holes in P3HT after turning off the irradiation. The lifetime of accumulated holes is very long; the number of accumulated holes hardly decreased after one week under dark conditions. In this case, the anisotropy of the $g$ factor is smaller than that before the irradiation, as shown by orange solid circles in Fig. 3(a). This result indicates that holes are accumulated in disordered regions as shown Fig. 4(b). That is, the charge accumulation likely occurs in the disordered regions, which is contrast to the interface charge transfer shown in Fig. 4(a). Here the disordered region means the region where crystallinity of P3HT lamella structures is incompletely preserved. Thus, the increase in ordered regions such as lamellar structures at the interface seems to prevent the charge accumulation in P3HT. In fact, the ESR intensity decreased by thermal annealing as shown in Fig. 2(c). This result indicates that the charge accumulation can be reduced by increasing the ordered regions with the lamellar structures of P3HT.

In contrast to the case of the thin-film sample, the broad component of the ESR signal of the cell after the light irradiation showed almost no anisotropy of the $g$ factor, as shown in Fig. 3(b). This result indicates that the hole accumulation at polymer-chain ends with bromines occurs in amorphous region as shown in Fig. 4(c). Here the amorphous region means the region where no crystallinity of the lamella structures exists. This result will be discussed later in detail.

3.5. Effect of ambient air on the charge accumulation

We investigated the effect of ambient air on the
charge accumulation by changing atmosphere in the ESR sample tube. In the investigation mentioned above, the ESR sample tube was sealed with a vacuum glass faucet after evacuating the inside down to $1 \times 10^{-4}$ Pa. In this case, however, there is a possibility that air leaks very slightly through the faucet into the sample tube. Thus, we measured a P3HT:PCBM solar cell in an ESR sample tube under the condition that air dose not leak at all by sealing the sample tube by melting the edge of the tube after evacuating the inside down to $1 \times 10^{-4}$ Pa, and by refilling helium gas of 100 torr. Figure 5(a) shows the ESR spectra of the cell under short-circuit conditions. The black line shows the ESR spectrum before simulated solar irradiation, and the red line shows that after 27-28 h simulated solar irradiation. In this case, the signal with a broad linewidth increased and the signal due to PCBM radical anions with $g = 1.9999$ decreased by the irradiation, as similarly observed in Fig. 1. However, the signal with a narrow linewidth with $g = 2.0017$ due to P3HT hardly increased by the irradiation. This behavior is different from that shown in Fig. 1. Also, this behavior does not depend on the condition of the device operation. The blue line in Fig. 5(b) shows the ESR spectrum under open-circuit conditions under helium inert-gas condition after additional 8-9 h simulated solar irradiation. This signal is similar to that under short-circuit conditions as shown by the red line in Fig. 5(a).

To study the effect of ambient air on the ESR signal, we performed the ESR measurement using the same cell under ambient-air condition by leaking air. The red line in Fig. 5(b) shows the ESR spectrum after additional 4-5 h simulated solar irradiation. Red line shows the ESR spectrum after additional 4-5 h simulated irradiation under ambient-air condition.
irradiation under ambient-air condition. The signal due to holes in P3HT with a narrow linewidth with \( g = 2.0019 \) increased by the air exposure, which is ascribed to the effect of air containing oxygen and moisture. Thus, the signal due to P3HT with a narrow linewidth can be reduced by helium inert-gas atmosphere. However, the signal with a broad linewidth continued to increase under helium inert-gas condition. Thus, the origin of the signal with a broad linewidth may be due to an intrinsic mechanism in the cell.

3.6. Origin of the ESR signal with the broad linewidth

To clarify the origin of the signal with the broad linewidth, we measured the ESR signal of a P3HT:PCBM film under simulated solar irradiation. Black dotted and red solid lines in Fig. 6(a) show the ESR spectra under dark conditions before light irradiation and under 19-20 h simulated solar irradiation, respectively. Increases in the signals with not only a narrow linewidth but also a broad linewidth were observed for the film by the irradiation.

To confirm the origin of this signal in detail, we performed the spectrum simulation of light-induced ESR spectrum which was obtained by subtracting the ESR spectrum under dark conditions from that under simulated solar irradiation. Figure 6(b) shows the fitting results for the light-induced ESR spectrum. The red solid line shows the fitting result for the signal with a broad linewidth with fitting parameters \( g = 2.0033 \) and \( \Delta H_{pp} = 0.98 \text{ mT} \). The orange solid line shows the fitting result for the signal with a narrow linewidth with fitting parameters \( g = 2.0021 \) and \( \Delta H_{pp} = 0.21 \text{ mT} \). The purple solid line shows the sum of two fitting curves, which explains the experimental result well. The \( g \) factors of the light-induced ESR spectra with the broad and narrow linewidth are nearly same as those of the cell shown in Fig. 1. Thus, each of the signals represents the same component of the cell. In this case, PCBM Li\(^+\) complexes are not formed, and the signal due to PCBM radical anions cannot be observed at room temperature [18,27]. Thus, the signal with the broad linewidth is ascribed to P3HT. This signal has a broader linewidth and a larger \( g \) factor compared with those of P3HT radical cations in polymer chains. Thus, the origin of the signal has been ascribed to P3HT radical cations (holes) at P3HT chain ends with residual bromines, because bromines cause a larger \( g \) shift and make the ESR linewidth broader due to nuclear spin of 3/2 [19,20]. Moreover, P3HT chain ends seem to have amorphous nature, which is consistent with the results shown in Figs. 3(b) and 4(c). Thus, the elimination of residual bromines at P3HT chain ends may reduce charge accumulation and may be useful for further preventing the performance deterioration of polymer solar cells.

4. Conclusion

We have studied the charge accumulation states in the polymer solar cells of ITO/PEDOT:PSS/ P3HT:PCBM/LiF/Al using ESR in detail. The ESR study demonstrates two kinds of the charge accumulation states by observing the light-induced ESR signals with the narrow and broad linewidth, that is, the narrow and broad component. The charge accumulation due to the broad component correlates with the device performance deterioration, which is the main deterioration factor. From the anisotropy in the ESR signals, the narrow component indicates that holes accumulate in
disordered regions of lamellar structure. The broad component is ascribed to hole accumulation in amorphous regions in P3HT, where holes accumulate at P3HT chain ends with residual bromines. The broad component is an intrinsic problem that occurs even under helium inert-gas atmosphere. Thus, the device performance would be improved by improving molecular orientation and by removing residual bromines.

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