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Realization of solution processed multi-layer bulk heterojunction organic solar cells by electro-spray deposition

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ABSTRACT

Electro-spray deposition (ESD) was applied to fabricate solution processed donor–acceptor bulk heterojunction organic photovoltaic devices with multi-layer structure. Solvent effect was observed when using different organic solvents. Power conversion efficiency (PCE) of the devices prepared from dichlorobenzene increased dramatically comparing to the ones from chloroform, owing to improved homogeneity of the films. ESD enabled us to fabricate solution processed multi-layer (donor/donor:acceptor/acceptor) devices with simple successive deposition steps. Energy Dispersive X-ray Reflectometry analysis confirmed distinct three layered structure of the active layers. Solar cell device parameters of the trilayer devices were compared to single layer devices and those of spin coated devices with the same donor:acceptor ratio and film thickness. Post-thermal treatment results showed that after annealing at 125 °C, trilayer devices exhibited best performance with the maximum PCE of 2.17%.

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1. Introduction

Organic photovoltaic (OPV) devices have been attracting extensive research attention over the last decade as they can significantly decrease the fabrication cost of solar energy conversion. Chemical designing and device engineering have been developed in parallel to maximize the power conversion efficiency (PCE), reaching published certified record value up to 8.62% [1]. In solution processed devices, donor–acceptor bulk heterojunction concept has been proven to be most successful [2]. A model system, which has been widely studied, is poly(3-hexylthiophene) (P3HT) as donor and [6,6]-phenyl C_{61} butyric acid methyl

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ester (PCBM) as acceptor. Optimization of the device structure and post treatment have continuously increased the PCE [3,4]. OPV devices fabricated by spin coating process have limitations such as small scaling of device area, larger material consumption and usage of high solution concentration. Towards large scale production, novel material growth such as screen printing [5], doctor blading [6], inkjet printing [7], spray deposition were proposed [8,9]. Electrospray deposition (ESD) is another alternative approach with its unique advantages. High applied voltage at the tip creates highly charged droplets, followed by a series of coulomb expulsion assisted by driving gas. Therefore, in electrospray deposition, very efficient separation of solute and solvent can be achieved [10]. Very low solution concentration can be used. This is particularly important for wide range of promising but low solubility polymers for device applications. Moreover, it provides the possibility of replacing environmentally harmful halogenated



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Fig. 1. Chemical structures of (a) P3HT; (b) PCBM; (c) device structure of trilayer bulk heterojunction organic solar cell.

solvents commonly used for polymer semiconductors. Indeed. ESD was used for fabrication of OPV devices. Kim and coworkers [11] obtained PCE up to 0.38% with solution concentration of 1.1 mg/ml of P3HT:PCBM applying very high electric field (15-18 kV). Thermal annealing increased PCE to 1.67%. However, further improvement of PCE to 3.25% involved additional solvent soaking process. Recently, Fukuda et al. reported PCE of 1.9% using mixed solvents where the concentration used was 2.2-1.8 mg/ml [12]. PCE of 2.2% was achieved when acetonitrile was used as additional solvent [13]. In these studies, another advantage of ESD was not utilized, which is the possibility of depositing multi-layer solution processed films. Multilayer structure is a path for further increasing the device efficiency, as a better device configuration should be a multi-layer structure of the type (donor/donor:acceptor/ acceptor) [14,15]. Here we call it trilayer (Fig. 1) bulk heterojunction, which can offer the advantages of both the BHI and the bilaver devices, i.e. an enlarged donor and acceptor interface and a spatially uninterrupted pathway for the transport of charge carriers to their corresponding electrodes. However, such a structure is difficult to be realized through solution processing, as the orthogonal dissolution cannot be avoided using common techniques. Since very efficient separation of solute and solvent can be achieved using electro-spray methods, one is allowed to fabricate such a novel device structure with simple deposition steps. In our recent studies, we demonstrated deposition parameter optimization of organic thin films using ESD method [16,17]. P3HT thin films prepared with optimized growth parameters, i.e., applied voltage of 2 kV and solution concentration of 0.5 mg/ml exhibited predominant flat-on orientation of the molecular lamella plane and good diode rectification [17], which is essential for the fabrication of OPV devices. In this work, we report the application of the ESD for OPV devices using low applied voltage (2 kV), low solution concentration (0.5 mg/ ml) and multi-layer device configuration. Optical absorption was measured to infer structural configuration. Film morphologies were investigated using Atomic Force Microscopy (AFM). Multi-layer device structure was studied using Energy Dispersive X-ray Reflectometry. Efficiencies of OPV devices were presented and compared for two different solvents and device structures as well as spin-coated films following careful thermal treatment.

2. Experimental

The experimental part of the solution effect was carried out at Linz institute for organic solar cells (Austria). Indium tin oxide (ITO) coated glass substrates were cleaned by sonication in 2% Hellmanex solution, deionized water, acetone, isopropanol and deionized water for 20 min each sequentially. The conducting hole transport buffer layer (50 nm) poly(3,4-ethylenedioxythiophene) doped with polystyrene sulfonic acid (PEDOT:PSS) was spin coated on ITO/glass substrates and subsequently dried in air for 30 min at 150 °C. Commercial electronic grade P3HT and PCBM powders (Sigma) without further purification were dissolved in chloroform and dichlorobenzene solutions (0.5 mg/ml). The active layers (blend of P3HT and PCBM), were deposited with weight ratios 1:1 using electrospray at Camerino University (Italy). Details of the home-made setup were reported in previous publications [16,17]. Optimized parameters are voltage of (2 kV) and flow rate of 1.3 µl/s for the films from dichlorobenzene and 2 μ l/s for the films from chloroform. Flow rate was controlled by over-pressurizing the solution bottle with nitrogen gas. Active layers were exposed to air about one week before they reached the lab for characterization. Their average thicknesses are about 120 nm measured by AFM. A 150 nm thick aluminum electrode was thermally deposited under an operating pressure of 10⁻⁶ Torr onto the active layer using a shadow mask to define an active device area of 10 mm² to complete the devices. The optical absorption spectra were measured by means of UV-Vis Varian Carry 3G spectrophotometer. Atomic Force Microscopy studies were carried out using Digital Instruments DIMENSION 3100 in tapping mode. AFM quantitative morphological analysis was performed using WSxM software. Devices were annealed at 150 °C for 5 min in the glovebox. I-V characteristics were measured using a Keithley SMU 236 in dry nitrogen glove box. A Steuernagel solar simulator for AM1.5 conditions was used as the irradiation source with an input power of 100 mW/cm² white-light illumination which was calibrated using a standard silicon diode. The second part of the experiment with multi-layer device architecture was realized at IMS-Bordeaux (France). Commercially available P3HT (Plextronics) and PCBM (Solaris) have been used without further purification. Indium tin oxide (ITO)-coated glass electrodes were cleaned in successive solutions of deionized water, acetone, ethanol and iso-propanol. In each solvent, substrates were immersed for 15 min. Then ITO plates were subjected to UV-ozone treatment for 5 min. About 30 nm thick PEDOT:PSS layer was spin coated and annealed in low vacuum at 110 °C for 30 min. Active layers were prepared using same ESD setup. For single layer and trilaver devices, the same amount of total solutions was used (2 ml). For trilaver devices, the ratio of used total solution for the separate layers of P3HT/P3HT:PCBM/PCBM was 0.3/1:1/0.3. The active layer thickness was about 90 nm. X-ray Reflectometry (EDXR) was used to investigate the layered structure of the films. A non-commercial setup, characterized by a simplified geometry, with neither monochromator nor goniometer, was used. In the ED mode the scan of the reciprocal space is carried out electronically, rather than mechanically as in conventional X-ray experiments, and the setup is kept in static conditions during the measurements [19]. This allow for an accurate sample analysis, since both the scattering volume of the film and the footprint of the X-ray beam on the surface remain constant, avoiding systematic errors induced by repositioning of the diffractometer arms, which can be critical when measurements are performed at grazing incidence, as in the present case. Reference spin coated films were prepared using P3HT:PCBM (1:1) of 40 mg/ml concentration in dichlorobenzene. Spin coating at 2000 rpm/s for 1 min matched the thickness of the film to that of ESD films. Spin coated active layers were not exposed to air. UV-Vis spectra were recorded over the spectral range of 300-800 nm using a SAFAS UVMC spectrometer. Current density-voltage (I-V)characteristics of devices were examined using a Keithley 4200 semiconductor analyzer under illumination of an AM1.5 set at 100 mW/cm² calibrated by an IL1400BL radiometer.

3. Results and discussion

Fig. 2 displays AFM images of active layers prepared with chloroform (a) and with dichlorobenzene (b) (which are most commonly used solvents for P3HT and PCBM in literatures), showing very different morphologies. The root mean square roughnesses are about 20 nm and 2 nm, respectively. Generally, the films prepared with dichlorobenzene show rather smooth surface compared to the ones with chloroform solutions, because of the different boiling



Fig. 2. AFM images in tapping mode (surface scan area: $10\times10\,\mu m^2)$ of P3HT:PCBM active layers deposited from (a) chloroform, and (b) dichlorobenzene.

points of solvents used. In the electrospray process, even if the solute and solvent separation can be achieved very efficiently, high boiling point organic solvents result in a less dry film. Dewetting of the trace solvent is playing an important role in smoothening the film. Much drier polymer blends tend to aggregate to form much rougher surface. Moreover, separate agglomeration of P3HT and PCBM is more prominent in the case of the blends from chloroform. Higher film roughness and poorer percolation of the donor and acceptor materials can be responsible for the lower efficiency of the devices prepared from chloroform as we see later on.

The normalized absorption spectra of P3HT:PCBM blend films from different solvents are reported in Fig. 3. As the main absorption comes from the P3HT component, the absorption spectra of P3HT:PCBM blend films were normalized to the maximum of P3HT absorption region. They show the same prominent vibronic feature at around 600 nm, which can be attributed to the interlayer stacking of P3HT polymer backbone [20]. The more intense vibronic feature in the case of samples prepared from chloroform might be due to the strong aggregation of the donor (P3HT). This also caused relatively higher P3HT to PCBM absorbance ratio. In addition, samples grown from chloroform solution showed sizable Mie scattering in the higher wavelength region due to microsize agglomeration [21]. These observations are in agreement with AFM analysis as discussed previously.



Fig. 3. Normalized absorption spectra of electro-spray deposited P3HT:PCBM (1:1) blend films from chloroform and dichlorobenzene, respectively.

Solar cell devices were fabricated from these two different types of films. The current density–voltage (J–V) characteristics under dark and illumination at AM1.5G (100 mW/cm²) are presented in Fig. 4 (left panel). In the dark, J–V curves showed poor rectification in the case of the devices deposited from chloroform with high leakage current on reverse bias. On the contrary, the devices prepared from dichlorobenzene showed higher rectification, suggesting better diode behavior due to smoother surface (as confirmed from AFM analysis) of the active layer and uniform coverage of the PEDOT:PSS layer resulting in much lower series resistance comparing to the devices from chloroform.

In fact, almost fourfold (from 0.34% to 1.3%) increase in PCE was measured for the devices deposited from dichlorobenzene with respect to the ones from chloroform (as shown in the right panel of Fig. 4). The current density inTable 1

Device performance parameters obtained from *J*–*V* curves for the electrospray deposited solar cell devices from chloroform and dichlorobenzene.

Devices	J _{sc} (mA/ cm ²)	V _{OC} (V)	FF (%)	PCE (%)	$R_{\rm s}$ (Ω cm ²)	$R_{\rm sh}$ (Ω cm ²)
Chloroform	2.15	0.50	31	0.34	225	462
Dichlorobenzene	7.0	0.44	41	1.3	20	207

creased to 7.0 mA/cm² and the FF to 41%, which suggests improved donor/acceptor percolation. Higher open-circuit voltage ($V_{\rm oc}$) in the devices from chloroform can be due to the lower recombination rate coming from larger domains. However poorer percolation of donor/acceptor and high series resistance led to lower current. Thus dichlorobenzene has become the choice of solvent in our further studies of ESD devices. Table 1 summarizes the solar cell parameters of these devices.

In order to further improve the performance of the devices and make use of the unique advantage of ESD method, we fabricated multi-layer devices with thin donor and acceptor layers sandwiching the bulk heterojunction of donor/acceptor layer. Multi-layer structure of the device was investigated using EDXR, which was carried out at a grazing scattering angle of $2\theta = 0.290^{\circ}$ on the trilayer P3HT/ P3HT:PCBM/PCBM film and on the single layer films, allowing a step by step characterization. The results of the experiments are summarized in Fig. 5. From the experimental profiles of the single layer samples (curves a-c) in Fig. 5, the numeric value of the scattering parameter q at the critical reflection edge (q_c) can be obtained by a sigmoidal fitting procedure (as reported in figure). The critical reflection edge is related to film scattering length density $\rho_{\rm e}: q_{\rm c}(A) = 2\sqrt{4\pi\rho_{\rm e}}$. This latter depends, in turn, on the chemical composition of each film and is proportional to the material density in the scattering volume. On the other hand, the roughness of the sample (σ (Å)), contributing to



Fig. 4. Comparison of current density-voltage characteristics for electro-spray deposited devices from chloroform and dichlorobenzene under dark and illumination at AM1.5G (100 mW/cm²).



Fig. 5. Reflectivity patterns collected upon the following films deposited from dichlorobenzene on glass/ITO substrates: (a) P3HT film, $q_c = 0.022 \text{ Å}^{-1}$; (b) PCBM film $q_c = 0.024 \text{ Å}^{-1}$; (c) P3HT:PCBM film $q_c = 0.028 \text{ Å}^{-1}$. The upper profile corresponds to the P3HT/P3HT:PCBM/ PCBM multi-layer.

Table 2

Fitting results of the EDXR data for separate films of P3HT, P3HT:PCBM and PCBM. ρ_e is the scattering length density and σ is the roughness of the sample.

Sample	$ ho_{ m e}$ (Å $^{-2}$)	σ (Å)
Glass/ITO/P3HT	9.65 (5) E-6	25 (1)
Glass/ITO/P3HT:PCBM	1.560(5) E-5	7 (1)
Glass/ITO/PCBM	1.150 (5) E-5	18 (1)



Fig. 6. Absorption spectra of electrospray deposited single layer (P3HT:PCBM) and trilayer (P3HT/P3HT:PCBM/PCBM) film together with spin coated (P3HT:PCBM) film. Solutions are from dichlorobenzene with 1:1 donor/acceptor ratio.

the pattern slope, is obtained by applying the Parratt formula to fit the data [18]. The results of data refinement are reported in Table 2.

The patterns of the intermediate systems, representing each single constituent of the trilayer i.e. respectively: (a) P3HT, (b) PCBM and (c) P3HT:PCBM directly deposited on the substrate, allow to assign the corresponding total reflection edges: (a) P3HT film, $q_c = 0.022$ Å⁻¹; (b) PCBM film, $q_c = 0.024 \text{ Å}^{-1}$; and (c) P3HT:PCBM $q_c = 0.028 \text{ Å}^{-1}$. Furthermore, the pattern of the P3HT/P3HT:PCBM/PCBM multilayer (top curve) is compared with the ones of the intermediate samples. The results show that the P3HT/



Fig. 7. Current density-voltage characteristics of the solar cell devices with spin coated, ESD single layer and ESD trilayer films. The devices were annealed at 120 °C for 10 min.

Table 3

Device performance parameters obtained from J-V curves for the ESD single layer, trilayer as well as spin coated devices after having annealed them at 125 °C for 10 min.

Devices	$J_{\rm SC}~({\rm mA/cm^2})$	$V_{\rm OC}$ (V)	FF (%)	PCE (%)	$R_{\rm s}$ (Ω cm ²)	$R_{\rm sh}$ (Ω cm ²)
Spin coated	6.8	0.42	49	1.4	20	287
ESD single layer	7.8	0.48	44	1.77	26	350
ESD trilayer	8.8	0.56	44	2.17	15	283

P3HT:PCBM/PCBM sample is characterized by the presence of three distinguished edges, perfectly coincident with the contribution of each single film composing the multi-layer. This is a clear demonstration that the trilayer is actually a composition of three single layers with distinct interfaces, individually contributing to the scattered signal.

To avoid thickness effect, we used the same amount of total solvents for single layer and trilayer ESD films. Spin coated samples (P3HT:PCBM = 1:1) with optimum spin speed matched the thickness of ESD films. Comparison of the absorption spectra of these films is shown in Fig. 6. Similar total absorption was achieved. Interestingly, vibronic feature at about 600 nm, which comes from polymer backbone stacking, is stronger in ESD films than in the spin coated film, suggesting that ESD films are better organized than spin coated films due to slow growth process.

Solar cell devices for these three types of films were fabricated and careful post annealing treatment was carried out. At lower annealing temperatures, ESD trilayer device consistently exhibited better performance than ESD single layer devices as well as spin coated devices. Best performance for ESD trilayer devices were obtained at around 125 °C. Comparison of the current density-voltage characteristics after annealing at 125 °C was presented in Fig. 7. Diode characteristics of the devices justifies the direct comparison as they show almost the same leakage current (left panel of Fig. 7). Highest diode rectification was achieved in the case of ESD trilayer device. Derived device parameters (as shown in the right panel of Fig. 7) revealed maximum J_{SC} (8.8 mA/cm²), V_{OC} (0.56 V) and PCE (2.17%). Details of the device performance parameters are summarized in Table 3.



Fig. 8. Various solar cell device performance parameters for spin coated, ESD single layer and ESD trilayer films following thermal treatment. At each temperature the devices were annealed for 10 min.

At lower annealing temperatures, both single layer and trilayer ESD devices show higher J_{SC} than spin coated devices, probably due to better structural order of donor/acceptor in ESD films assisted by slow growth process. Stronger vibronic features in absorption spectra confirm this postulation. Such an enhanced order results in higher electron and hole mobilities, consequently higher current achievable in the short circuit condition. In trilayer devices, charge carriers suffer minimum "dead-ends" due to continuous percolation path provided by pure donor and acceptor layers respectively at anode and cathode. These layers can be understood as transport layers, providing very efficient charge extraction at the interface. Thus, the current is further enhanced comparing to single layer devices.

 V_{OC} is known to scale with the difference between the highest occupied molecular orbital (HOMO) of the donor and the lowest unoccupied molecular orbital (LUMO) of the acceptor [22,23]. Here we have the same donor and acceptor materials, indicating same maximum achievable V_{OC} from these devices. However, V_{OC} is strongly affected by energetic disorder [24]. Higher disorder induces enhanced loss, consequently leading to lower achievable V_{OC} . Therefore, lower V_{OC} of spin coated devices comparing to ESD deposited devices can be attributed to their lower degree of order at lower annealing temperatures. In trilayer devices, interface recombination, which is another loss mechanism, can be minimized. Therefore, highest V_{OC} is achieved in trilayer devices.

However, FF of ESD devices are generally lower than spin coated devices. We suppose this is mainly due to higher trap state density in ESD films coming from morphological defects and partial oxidation. While J_{SC} is less affected by trap states owing to high internal field induced de-trapping, FF is much more sensitive to the presence of trap states. Oxidation can be stronger for donor or acceptor pristine films than donor/acceptor heterojunction films, making trilayers films more susceptible to air exposure than single layer films. Although higher overall carrier mobilities prompted by donor/acceptor transport layers in trilayer devices is expected to yield higher FF, stronger oxidation can compensate this effect, leading to comparable FF of trilayer and single layer devices.

Finally, in Fig. 8, we demonstrate the evolution of all the device parameters following the thermal treatment. Above 130 °C, efficiencies of both single layer and trilayer ESD devices started to decrease, while that of spin coated device continued to increase up until 165 °C, which is consistent with literature reports for the spin coated devices. Such a behavior can be ascribed to the fact that the ESD films were exposed to air for a quite long period, causing oxidization of the film surface, which in turn partially oxidized the Al top contact at higher annealing temperatures. Indeed, we observed the increase of the series resistance in the case of ESD devices above 130 °C, while the contrary was true for the spin coated devices. Continuous increase of V_{OC} of spin coated devices until saturation indicates progressive reorganization happening in the films until high annealing temperatures. However, the saturations of $V_{\rm OC}$ in ESD devices are at rather low temperatures, confirming already well ordered polymer structure in the films. It should be

noted that $V_{\rm OC}$ is not affected by the change in the series resistance following the temperature treatment, as there is no current in open circuit condition. However, the increase of series resistance in ESD devices at higher annealing temperatures seriously affects the $J_{\rm SC}$ and FF of the devices leading to the deterioration of the device performance.

In conclusion, we successfully demonstrated that electrospray deposition technique is a promising deposition process for the fabrication of active layers in solution processed organic solar cells in more complex device configuration. Strong solvent effect was observed with more than four times higher efficiency when using dichlorobenzene comparing to chloroform, which is due to improved film quality in terms of roughness and better percolation of donor/acceptor. Solution processed multi-layer donor/ donor:acceptor/acceptor device structure was realized through simple successive deposition steps. Such a trilayer configuration was confirmed by EDXR measurements. Due to better polymer organization in ESD films under slow growth condition, at lower annealing temperatures (below 130 °C), ESD devices exhibit better performance comparing to spin coated devices with the same thickness and donor/ acceptor ratio. Highest PCE (2.17%) was achieved for trilayer ESD devices, thanks to better charge transport and extraction through the thin layers of pure donor or acceptor at the proper side of charge collecting electrodes. The performance of ESD devices could be further improved by optimizing parameters such as the film roughness, film thickness, decreasing oxidation and the use of other post growth treatments. Another asset of the present method is the use of low concentration solutions, allowing a wide range of polymers with low solubility to be candidates for future device applications. Furthermore the ESD technique might be employed to fabricate tandem solar cells in a more controlled deposition manner.

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