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Doped conducting-polymer–semiconducting-polymer interfaces: Their use in organic photovoltaic devices

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We report a study of the interface between poly(*p*-phenylene vinylene) (PPV) and poly(ethylene dioxythiophene) doped with polystyrene sulfonic acid (PEDOT:PSS). We find from measurements of optical absorption and conductivity that PSS dopes PPV during the sample preparation. In addition, the photoluminescence efficiency of PPV is strongly affected by the presence of PSS which we attribute to the effect of chemical doping. We further studied the interaction between PSS and PPV by measurements of the photovoltaic response, spectrally resolved, of a number of diode structures. By forming the interface between a layer of PEDOT:PSS and a layer of PPV precursor prior to the thermal conversion we obtained an interpenetrating interface with large surface area between the photoresponsive and the charge collecting polymers. For devices made with PEDOT:PSS as top electrode and aluminum as the bottom electrode the short-circuit external quantum efficiency is 4%. [S0163-1829(99)11327-4]

I. INTRODUCTION

In the field of organic photovoltaic devices, the separation of charges following a photoexcitation and the balance between the transport of holes and electrons is crucial for the device performance. Therefore, the properties of the interface between the materials are very important. Organic optoelectronic devices are commonly built using a sandwich architecture, starting from a glass substrate covered with transparent, electrically conducting indium-tin oxide (ITO), and then forming the active layer (or layers) either by sublimation or spin-coating techniques. The final step is the formation of a second electrode, usually an evaporated film of a low work-function metal.[1–7] However, using organic materials we have a large freedom of choice when it comes to fabrication methods and device designs. For instance, it has been shown for polymer light-emitting diodes that crucial properties such as luminous efficiency and lifetime have been improved when the ITO is coated with doped conducting polymers such as doped poly(aniline) or poly(ethylene dioxythiophene) (PEDOT) prior to the formation of the luminescent layer.[8,9] For polymeric photovoltaic devices, the open-circuit voltage and fill factor improve when PEDOT is used as electrode[10] and the external quantum efficiency increases when using PEDOT as an intermediate layer between a semiconducting polymer layer and ITO.[11] Using a polymer electrode it is possible to envision flexible photodiodes[12–14] and to change the fabrication sequence, allowing for different types of interfaces to be created and studied. This has been the background for the present work, where we have compared devices made the conventional way with devices in which the metal contact is evaporated on the glass substrate and a polymer electrode is used as top contact.

II. EXPERIMENTAL METHODS

A. Fabrication and characterization of the interfaces

We have studied the interface between poly(*p*-phenylene vinylene), (PPV) and poly(ethylene dioxythiophene) doped with polystyrene sulfonic acid, PEDOT:PSS (from here on referred to as PEDOT), in two different configurations. In the first configuration, films of PEDOT (commercially available from Bayer AG, Leverkusen, Germany) were deposited by spin coating from a water solution (1.2%) onto a glass substrate. Subsequently PPV films were prepared via the sulfonium salt precursor route,[15] by spin coating films of the precursor polymer from a methanol solution on top of the PEDOT film. Conversion to PPV was achieved by heating the samples *in vacuo* at 220 °C for 10 h. In the second configuration, we spin coated PPV precursor on top of the glass substrate and subsequently PEDOT on top of the PPV precursor before the thermal conversion of the precursor to PPV. We spin the PEDOT film on top of the precursor rather than the converted PPV film for an entirely practical reason. It is very difficult to spin from a water solution on top of the hydrophobic PPV surface, compared to on the more hydrophilic surface of the precursor. In addition, conductivity and work-function measurements show that the PEDOT film is very stable to the heat treatment, since the conductivity and the work function remain virtually unaffected.[16] We would like to point out that in contrast to a previous report from our group,[17] we used the PEDOT solution as received from Bayer, without any modifications. Optical absorption was measured with a Perkin-Elmer λ9 spectrometer and a Hewlett Packard 8453 UV-Vis spectrophotometer. The morphology was investigated using two techniques: scanning force microscopy (SFM) and optical microscopy. SFM images were taken using a NanoScope IIIa Dimension 3100 (Digital Instruments Inc., Santa Barbara, CA) run in tapping mode. Optical imaging of the films was done with a fluorescence microscope (Vickers Photoplan M41). Photoluminescence (PL) efficiency measurements were performed in an integrating sphere coupled via a liquid light-guide to an Oriel InstaSpec IV spectrograph.[18] The excitation source was the 488-nm line from an argon ion laser.

Photothermal deflection spectroscopy (PDS) spectra were measured using a setup similar to that described by Jackson *et al.*[19] The sample was illuminated using a combination of a Light Support MKII 100 W Xenon arc source and a CVI

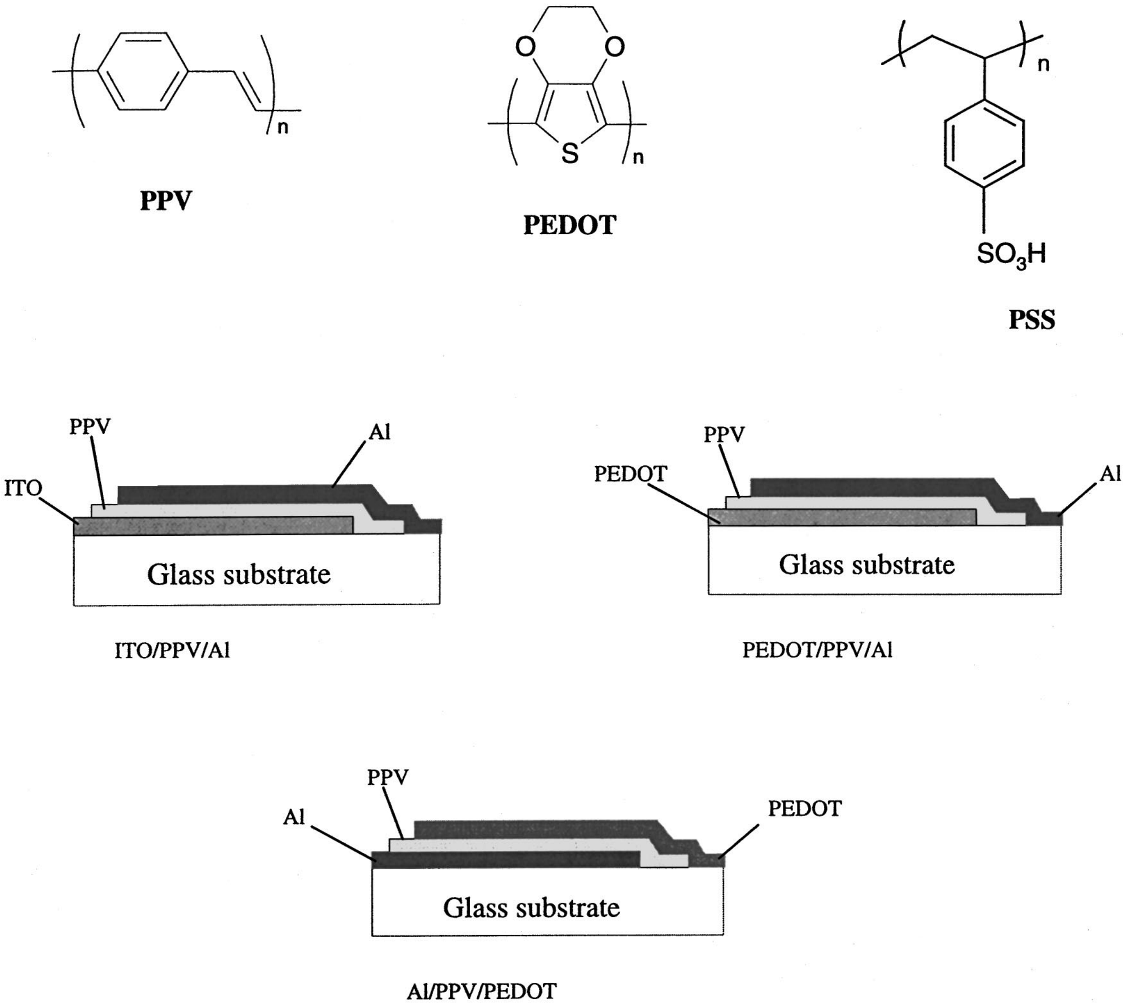


FIG. 1. Schematic sandwich structure of the photodiodes used and chemical structure of PPV, PEDOT, and PSS. PPV is the photoresponsive material, Al is the electron-collecting electrode, and PEDOT is the hole-collecting electrode.

DK240 monochromator. The probe beam was supplied by a Point Source 670-nm fiber coupled diode laser with temperature stabilizer for reduced beam pointing noise. Beam deflections were measured using a differentially amplified quadrant photodiode and a Stanford Research SR830 lock-in amplifier. Throughout the measurement, the samples were held in a hermetically sealed fused silica cuvette filled with Fluorinert FC-104 (3M Corporation, St. Paul, MN) as a deflection medium.

B. Fabrication and characterization of photovoltaic devices

In order to investigate the photoresponse of these materials, three different types of photovoltaic devices were fabricated all using a single layer of PPV (150-nm thick) as photoresponsive material. The first type of device is the standard device made with the usual sandwich structure: glass/ITO/PPV/Al. In the second type, the same structure is used, but ITO is replaced by PEDOT. In the third type of device, PEDOT was used as the top contact and Al as the bottom contact, forming an inverted structure, which we refer to as ‘‘upside-down’’ structure.[20] The structures of these devices are shown in Fig. 1 together with the chemical structures of the conjugated polymers. Aluminum contacts were deposited by thermal evaporation, under vacuum at a pressure of 10^{-6} mbar. PEDOT and PPV films were prepared as described above. In order to measure the photocurrent spectra, the devices were illuminated by a tungsten lamp, dispersed by a Bentham M300 single-grating monochromator. To correct for the optical throughput of the illumination system, the setup was calibrated using a silicon photodiode (Hamamatsu S5106). Open circuit voltages were measured at or close to the peak of the PPV photocurrent output, and with an illumination intensity of 40 μW/cm^2. The devices were measured in air and under vacuum, and had an active area of 16 mm^2. In all experiments, the thickness of the polymer layers were 150 and 200 nm for PPV and PEDOT, respectively.

III. RESULTS

We found the PL efficiency of PPV converted on glass to be 0.30, which is similar to values reported by Greenham *et al.*[21] We also found that when PPV is converted on top of ITO, the PL efficiency is reduced to 0.15 and when PPV is converted on top of PEDOT, the PL efficiency is 0.06. However, when PPV is converted with PEDOT on top, the quenching of the photoluminescence is even stronger, down to about 0.02.

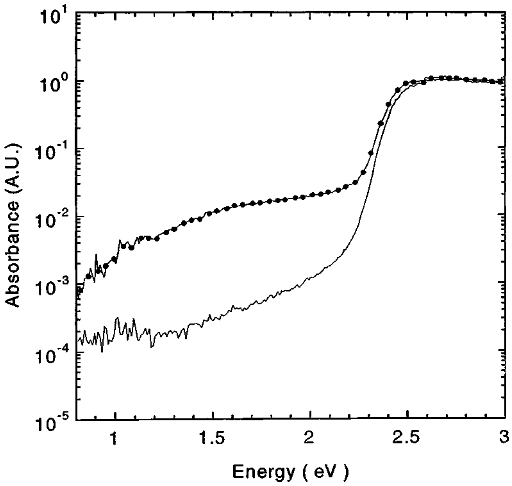


FIG. 2. Photothermal deflection spectroscopy spectra of PPV (solid line) and PPV converted on top of PSS (full circles).

Fou *et al.* suggest that PSS can dope PPV if present during the conversion or during the operation of light-emitting diodes, reducing the luminescence of these devices.[22] Similarly, it could be expected that the presence of the PSS at the PEDOT/PPV interface could lead to a partial doping of the PPV layer, thereby quenching the photoluminescence. Figure 2 shows the PDS spectra of PPV, and of PPV converted on top of a PSS film. It can be seen that the presence of PSS causes the appearance of an absorption band in the subgap region of the PPV absorption spectrum. This can be associated with the presence of charged species such as polarons or bipolarons resulting in a peak near 1.6 eV.[22] We also found that PPV films converted on top of PSS show higher conductivities (about 10^{-4} S/cm) than PPV converted on top of glass.

Figure 3(a) is an optical microscopy image of a PPV/PEDOT film in which PEDOT was deposited on top of the PPV precursor, taken from the PPV side. It clearly shows spots in which the PEDOT (darker) reaches or approaches the interface between the PPV and substrate. Figure 3(a) also suggests that in some areas the PPV is thinner, showing more of the darker color of the PEDOT.

Figures 3(b) and 3(c) show topography and phase SFM images of the surface of the PPV side from a PPV/PEDOT film. The phase detection mode of the SFM is particularly useful to detect where there are different materials present, for instance in a polymer blend. Here we see that there are domains of a second material (PEDOT) reaching the surface (mainly PPV). Both optical and SFM images show that these domains are about 2.5–4 μm in size. Figure 3(d) shows a schematic representation of the interface, inferred from the SFM images.

The short-circuit photocurrent action spectrum of ITO/PPV/Al devices, under illumination through the ITO, consists of a very narrow peak located at the low-energy tail of the PPV absorption spectrum. This response is shown in Fig. 4, where the external quantum efficiency (EQE) of the device is plotted together with the absorption spectrum. However, when the device is illuminated through a semitransparent Al electrode, the spectral response is broader and follows the basic shape of the absorption. Our results for ITO/PPV/Al devices are in agreement with the results reported previously for 150-nm-thick single layer PPV photodiodes[3] and the devices exhibit an open circuit voltage of 0.8 V and a fill factor of 0.23. The fill factor gives an indication of how well a device is suited as a current source and is defined as the maximum electrical power $(IV)_{\max}$ divided by the product of the short-circuit current I_{sc} and maximum open-circuit voltage V_{oc}:

$$f_F=\frac{(IV)_{\max}}{I_{sc}V_{oc}}. \tag{1}$$

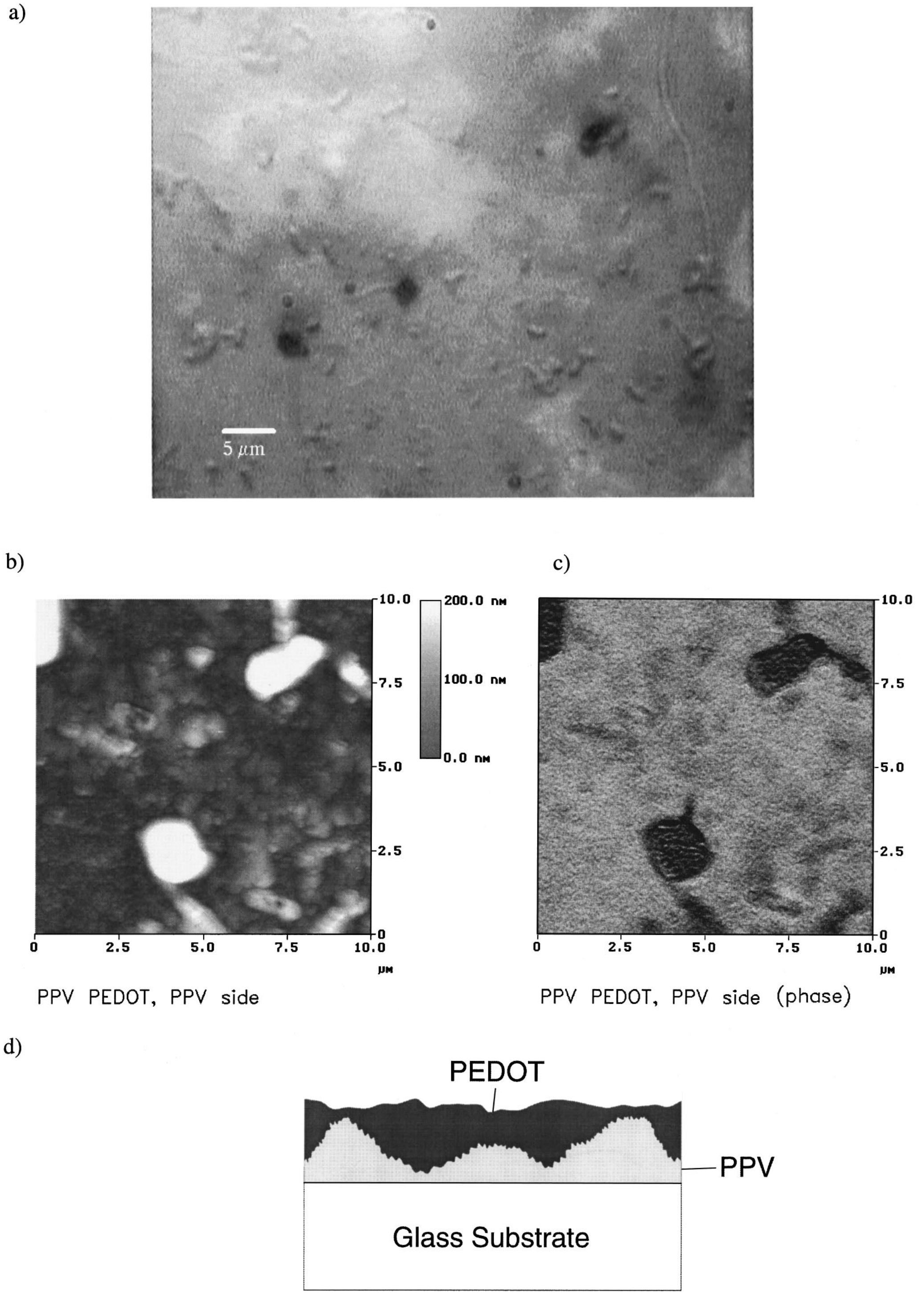
Replacing the ITO contact by PEDOT results in similar efficiencies, but the shape of the EQE action spectrum is different. Figure 5 shows that when the device is illuminated through the PEDOT contact, the external quantum efficiency increases rapidly at the onset of the PPV absorption, and as the absorption coefficient increases, the EQE remains constant. When the device is illuminated through the Al electrode, the EQE reaches higher values than when illuminated through the anode and follows the shape of the absorption curve in a similar fashion to the ITO samples. The open circuit voltage of these devices is 1.0 V and the fill factor is 0.23.

Using upside-down devices (glass/Al/PPV/PEDOT), we notice an increased photocurrent and an improvement of the EQE. The photocurrent spectra of upside-down devices, expressed as EQE, are shown in Fig. 6. The absorption spectrum of PPV is shown for comparison. Both curves in Fig. 6 were obtained when illuminating through the PEDOT contact. When the device is measured in air, the EQE response follows the absorption very closely. However, when the same device is measured under vacuum, the maximum of the EQE drops reversibly from 4% to less than 0.5%, and the peak shifts to lower energies where the absorption is smaller. Under reverse bias, the quantum efficiency rises rapidly, reaching 12% when -1 V is applied and 16% at -1.5 V. The device is rectifying both in the dark and when illuminated, but under illumination the current increases under both forward (PEDOT is the positive contact) and reverse bias, and an open-circuit voltage of 0.6 V is observed. The fill factor calculated for these devices is about 0.23, which is similar to other single layer PPV devices.[3]

IV. DISCUSSION

A. Electronic properties of PPV in contact with PSS

The use of a doped conjugated polymer as the hole-injecting layer in polymer light-emitting diodes is widely reported, and is considered to give important improvements in performance, both efficiency of operation and durability. This is reported for devices made with directly soluble polymer semiconductors, such as soluble dialkoxy-PPV's,[8] and also with precursor-route PPV.[23] The reasons for its effectiveness are not well understood, although it is certainly desirable to use electrodes with very high work functions for hole injection, in order to match the position of the valence-band edge in the semiconducting polymer, and doped poly-



PPV PEDOT, PPV side

PPV PEDOT, PPV side (phase)

FIG. 3. (a) Optical microscopy image of PPV/PEDOT film, taken from the PPV side. The darker contrast comes from the PEDOT. (b) Scanning force microscopy topography image of a film prepared in the same way as in (a). Tapping mode, scan size 10×10 μm (c) Phase detection mode image of the scan in (b), clearly showing the two different phases. (d) Sketch of possible cross section.

mers such as PEDOT:PSS do show work functions above 5 eV. It is also generally desirable to separate the underlying ITO from the semiconducting layer since ITO is not a well-controlled material, exhibiting a work function that varies considerably with surface treatment.[17]

We note, however, that doped conducting polymer layers can interact with semiconducting polymer layers in complex ways, and the results that we have presented here provide clear evidence for such interactions for the case of PEDOT:PSS in contact with PPV formed via the sulfonium precursor route. The virtue of using this polymer is that the high temperatures involved in the conversion of the precursor polymer allow interactions with the PEDOT:PSS to proceed towards completion [in contrast to the use of directly soluble polymers such as the poly(2,5-dialkoxy)-PPV's that are processed at room temperature].

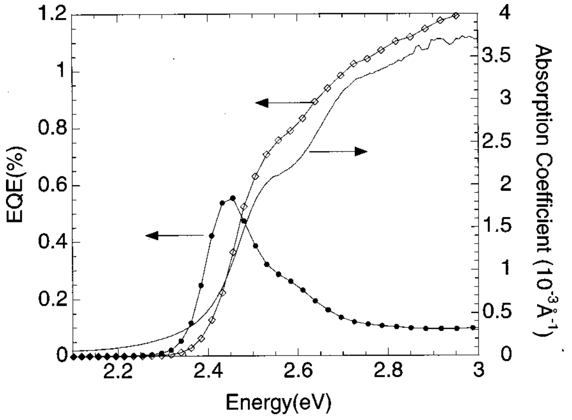


FIG. 4. Short-circuit external quantum efficiency spectrum of ITO/PPV/Al device, with illumination through ITO (full circles), and through a semitransparent Al (open diamonds). The absorption coefficient of PPV film is shown for comparison (solid line).

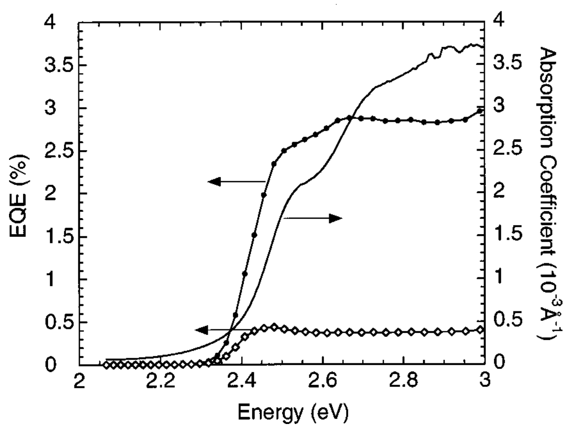


FIG. 6. Short-circuit external quantum efficiency spectrum of an Al/PPV/PEDOT device, with illumination through the PEDOT contact. The full circles represent measurement in air and the open diamonds represent measurement under vacuum.

The most direct interaction between doped polymer and semiconducting polymer is the movement of the ‘‘dopant’’ from the former to the latter, giving rise to partial doping of the semiconducting polymer. This may be very desirable for the purpose of hole injection, since it may be possible to form a ‘‘graded’’ dopant region, which allows easy passage of holes from highly doped to undoped polymer. We have used PEDOT:PSS here since this is a favored combination, and we note that the ‘‘dopant’’ is a strong (protonic) polymeric acid, PSS. Since it is a polymer its ability to diffuse between layers is limited, and we would expect interactions to be confined to the surface layers at the interfaces formed between the doped and semiconducting polymers. The actual mechanism of the ‘‘doping’’ reaction involving the acid is not as clearly understood as is the case for doping using oxidizing agents. Han and Elsenbaumer[24] have discussed the possible mechanisms and consider that there is protonation at a carbon site involved in the delocalized π-electron system, and that this introduces a net charge (hole). Values of conductivity are generally lower for doping of this type than for oxidative doping, as we find here for the case of PPV. The clearest evidence for doping is from the optical absorption spectrum shown in Fig. 2 measured for PPV formed on a layer of PSS. We note that the subgap absorption band attributed to polaron or bipolaron formation has a peak intensity near 1.6 eV about 1% of the π-π^* absorption band. This indicates that the level of doping is relatively low, of the order of 1%, and is consistent with the level of dc conductivity achieved (10^{-4} S/cm). This level of doping is much lower than for the PEDOT, and is presumably attributable to the higher value of ionization potential for PPV.

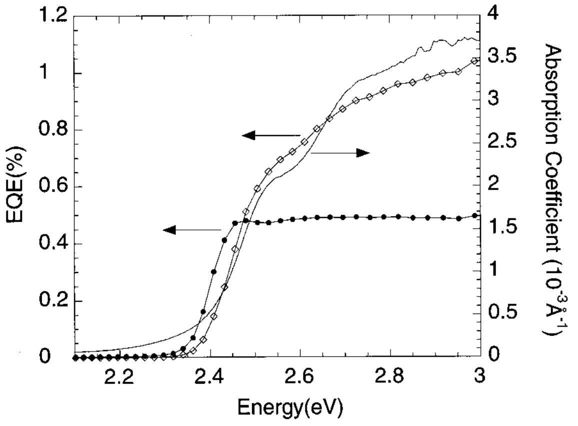


FIG. 5. Short-circuit external quantum efficiency spectrum of PEDOT/PPV/Al device, with illumination through PEDOT (full circles), and through a semitransparent Al (open diamonds). The solid line is the absorption coefficient of PPV.

We have also found that the integrity of the layer structure is disrupted by the interactions between PPV and PEDOT:PSS, and we discuss below how this alters the photoconductive and photovoltaic behavior of a range of diode structures. We note that it is in principle very desirable to be able to process a transparent electrode on top of the semiconductor layers, allowing the fabrication of device on nontransparent substrates. This has been achieved by direct deposition of ITO onto PPV.[20] It would be very useful to use a polymer system such as PEDOT:PPS in this role, and we have therefore examined such ‘‘upside down’’ structures here. As discussed below, we find that the layer structure is heavily disrupted by the processes involved in forming this structure.

B. Photovoltaic results

In order to study the influence of different types of interfaces in exciton dissociation and current generation in photovoltaic devices, we use the EQE action spectra of the different types of devices as our main evaluation tool. The shape of the EQE action spectrum provides information about which parts of a photovoltaic device are active and where the dominating exciton dissociation takes place.[7] When an ITO/PPV/Al device is illuminated through the ITO contact, in the range where the absorption coefficient is small, the incident light penetrates deep in the device and can reach the Al/PPV interface. Since PPV transports holes better than electrons,[25–27] the excitons dissociated at this interface contribute efficiently to the current because the electrons are close to the Al contact and holes are transported through the PPV film to the ITO contact. In Fig. 4, we can see that the EQE has a peak at the onset of the absorption spectrum. In the energy range where the absorption coefficient is higher, most of the incident light is absorbed close to the ITO/PPV interface and by the bulk of the polymer. If excitons are dissociated near the ITO/PPV interface, holes are easily collected by the ITO contact, but electrons have to travel through the PPV film to be collected by the aluminum electrode. Because PPV is a poor electron transport material, the probability that electrons are trapped and undergo recombination before collection is very high. As Fig. 4 shows, the EQE falls in this range to a low value. Such behavior is known as filter effect, because the bulk of the film acts as an optical filter.[28] However, when the device is illuminated through the semitransparent Al contact, the filter effect is not observed, since the EQE spectrum follows the absorption. In this case, most of the incident light is absorbed close to the Al/PPV interface and excitons are dissociated near this interface contributing to the current. When the absorption coefficient increases, even more photons are absorbed near the same interface resulting in a larger current and consequently higher quantum efficiency.

On replacing ITO with PEDOT we observe a different shape of the EQE (see Fig. 5). When the device is illuminated through the PEDOT, we observe that the EQE increases rapidly at the onset of the absorption. However, when the absorption coefficient increases the EQE remains constant and the filter effect observed in devices made with ITO does not occur. In this context, we also note that when PPV is converted on top of PEDOT, the photoluminescence efficiency of PPV is quenched more than when PPV is converted on top of ITO. The strong reduction in the photoluminescence indicates that interfacial reactions between PPV and PEDOT are occurring during the PPV conversion. The fact that the PPV layer is doped (see Fig. 2) corresponds well with the photoluminescence measurements. It is well known that either introducing charges in the form of dopant ions or using field effect devices strongly quenches the PL.[29–31] Apart from quenching the PL, the doping also results in an increased conductivity of the polymer. Compared to the ITO/PPV devices where the electrons have to be transported through the poorly conducting film to the aluminum electrode, even a slight doping of the PPV facilitates this transport in the PEDOT/PPV devices. We also believe that the doping could improve exciton dissociation in these devices. Therefore, when the device is illuminated through the PEDOT electrode, charges are collected independently of where in the device the incident light is absorbed, resulting in a flat EQE action spectrum, as observed in Fig. 5. The absence of a filter effect indicates that excitons dissociated close to both interfaces contribute to the current.

In upside-down devices, the shape of the EQE spectrum is similar to the device with PEDOT as a bottom contact but shows higher values. From optical microscopy and SFM images in Fig. 3, it is clear that the thickness of the PPV film varies considerably. This means that at the end of the conversion we obtain a very large interfacial area between the two polymers. In some parts of the device the PEDOT is close to the aluminum contact, resulting in a very thin PPV layer, whereas in other parts the PEDOT does not penetrate so much, which gives a thicker layer of PPV. We also observed a stronger quenching of the PL efficiency of PPV when converted with PEDOT on top, than when PPV is converted on top of PEDOT. This effect is in agreement with the increased interfacial area between PPV and PEDOT leading to a larger portion of the PPV film being exposed to PSS. The large interpenetrating interface between PPV and PEDOT is likely to improve the collection of electrons, which need to be transported through the PPV film to reach the Al electrode. As in some parts of this device the PPV layer is very thin, the probability of an electron to be trapped or to recombine before collection is smaller compared to the standard devices. The increased doping also improves the transport through the film. Moreover, where the PPV layer is very thin, light can reach the Al/PPV interface, even when the absorption coefficient is fairly high, and both interfaces contribute to the exciton dissociation. Consequently, devices made with this structure show improved external quantum efficiencies of up to 4% when illuminated with a light of intensity of 40 μW/cm^2. These values are almost an order of magnitude higher than for the first two types of devices made under the same conditions, which show a peak EQE of 0.5%. Comparing the results obtained in air and under vacuum, we can conclude that the presence of oxygen increases the exciton dissociation. The PEDOT/PPV interface is the least protected interface and most vulnerable to attacks from oxygen and water in the ambient atmosphere.

Another fact to note is that the open circuit voltage is lower for the upside-down device compared to the PEDOT/PPV/Al device (0.6 V compared to 1.0 V, respectively). One possible reason for this could be that the conversion of PPV precursor to PPV involves the release of hydrochloric acid, which conceivably could etch the aluminum electrode and thereby change its work function. However, Kelvin Probe measurements show that this is not the case, and that the work function of the aluminum remains the same after conversion and subsequent removal of the polymer film. Reasons for the decreased open-circuit voltage therefore have to be sought elsewhere, such as the formation of interfacial layers and possibly also a different interaction between the metal and the polymer depending on the fabrication sequence. It has been shown that aluminum evaporated onto PPV or PPV-related materials form covalent bonds, thereby modifying the π-electron structure of the conjugated molecules.[32,33] The interface formed when PPV is deposited on top of aluminum remains to be investigated and understood.

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