

FLEXIBLE ENCAPSULATION FOR ORGANIC SOLAR CELLS

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ABSTRACT

Efficient organic photovoltaic devices show many interesting properties, but share a common drawback, namely their instability in atmosphere. We report on a shelf lifetime study of solar cells based on blends of two widely used polymeric semiconductors with 1-(3-methoxycarbonyl) propyl-1-phenyl[6,6]C61 (PCBM), encapsulated in a new flexible and transparent poly(ethylene naphthalate) (PEN)-based ultra-high barrier material. The barrier coating is entirely fabricated by plasma enhanced chemical vapor deposition (PECVD). The conjugated polymers used are poly(2-methoxy-5-(3',7'-dimethyloctyloxy)-1,4-phenylene-vinylene) (MDMO-PPV) and poly(3-hexyl)thiophene (P3HT). We have observed in this work that the encapsulation raises the shelf lifetime (50 % of the initial efficiency) from a few hours into the range beyond 3,000 hours for MDMO-PPV based devices. Using the more stable P3HT, the lifetime could be increased to approximately 6,000 hours, or more than eight months.

Keywords: solar cells, photovoltaic, organic semiconductors, encapsulation, PECVD, stability

1. INTRODUCTION

Due to a strong demand for renewable energy¹ and a widespread and growing interest in organic semiconductor based devices,^{2,3} organic solar cells have been the object of increasing development during the last decade.^{4,5} Recently, efficiencies up to 5% have been reported for both conjugated polymer⁶ and small molecule based photovoltaic devices (PV).⁷ Considering their intrinsic physical properties such as light-weight, mechanical flexibility and semi-transparency, these devices may open up new opportunities for applications of PV cells. Besides, conjugated polymers can be made soluble⁸ in organic solvents, so that low-cost production of polymeric PV can be envisaged by applying common printing techniques.⁹ This allows large-scale roll-to-roll processing using flexible substrates. However, to be viable in the market, such products should not only offer high efficiencies and low cost, but also lifetimes (delivering more than 50% of the initial performance level) of at least 10,000 hours.¹⁰

1.1. Degradation of Organic Solar Cells

Conjugated polymers are known to be rather unstable in air,¹¹ being particularly susceptible to photodegradation induced by oxygen and moisture.¹² Especially poly(p-phenylene vinylene) (PPV) and its derivatives are susceptible to degradation in atmosphere. The mechanism involves the binding of oxygen atoms to vinyl bonds, which breaks the conjugation and leads to the formation of carbonyl groups.^{12,13} Spectroscopic ellipsometry studies showed that during simultaneous exposure to air and light, the thickness of the active layer increases, while its refractive index and absorption coefficient drastically decrease.¹⁴ Moreover, the material for the low work function electrode is usually chosen from metals like Al and Ca, in order to minimize the electron injection barrier and to maximize the open circuit voltage (V_{OC}) of the solar cells.¹⁵ These metals undergo rapid oxidation when exposed to air. This leads to the formation of thin insulating oxide barriers,¹⁶ hindering electric conduction and collection of the charge carriers. Besides, it has been reported that water can affect the interface between the metallic contact and organic semiconductors by an electrochemical process that causes delamination of the electrode.¹⁷ In spite of the fact that the degradation effect is significantly decreased when conjugated

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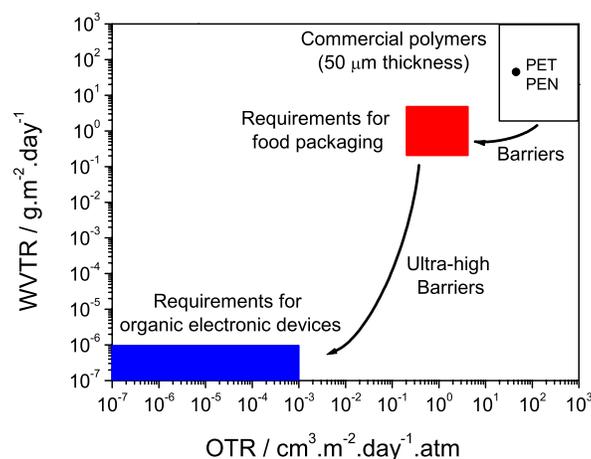


Figure 1. Water vapor transmission rate (WVTR) versus oxygen transmission rate (OTR) for commercial polymers, encapsulations required for food packaging and for organic electronics purposes.

polymers are mixed with fullerenes,¹⁸ PPV based solar cells usually do not operate for more than a few hours in air.¹⁹ Therefore, appropriate encapsulation is mandatory for this type of devices. The ability of oxygen and moisture to cross an encapsulating membrane is expressed by the oxygen transmission rate (OTR) and the water vapor transmission rate (WVTR), respectively. It is generally accepted that the lifetime of organic light emitting diode (OLED) devices above 10,000 hours requires WVTR and OTR of below 10^{-6} g m⁻² day⁻¹ and 10^{-3} cm³ m⁻² day⁻¹ atm⁻¹, respectively.²⁰ These values, which seem to be on the upper limit of organic solar cell requirements, are about six to eight orders of magnitude lower than the corresponding values of commercially available polymer films (Fig. 1). A quite straightforward solution consists of encapsulating the sensitive devices between glass plates,^{21,22} which form an optimum barrier even when the plates are very thin. However, this solution obviously sacrifices flexibility and mechanical robustness of the final device, and therefore is not satisfactory for large-scale, low-cost manufacturing.

1.2. Thin Film Gas Barriers on Polymers

Thin film barrier coatings against permeation of gases and vapors deposited onto flexible polymer substrates have been intensively studied for applications in food and pharmaceutical packaging, where improving the barrier capabilities of the bare plastic films by one to three orders of magnitude is usually considered sufficient (Fig. 1). It has been shown that silicon-based dielectric coatings deposited by plasma enhanced chemical vapor deposition (PECVD) on plastic films can be used as single layer permeation barriers.^{23,24} Permeation through those barrier materials is a defect-controlled phenomenon. It has been observed that sub-micron-size structural defects present in the barrier coating, which are mainly caused by intrinsic or extrinsic surface roughness account for this limited performance of single-layer gas barrier. Theoretical calculations have shown that the total permeation rate through many small pinholes is much higher than that corresponding to the same total pinhole area combined in a few larger defects.²⁵ This can be explained by lateral diffusion and it is of crucial importance when the diameters of defects are small compared with the thickness of the substrate. Finally, it should be noted that deposition of multilayered inorganic films usually improves the barrier performance only slightly,²⁶ mainly because of defect propagation through successive layers during growth. The most common technique used to achieve ultra-high barrier properties is based on alternating organic-inorganic multilayers: Sandwiching inorganic barriers between polymeric buffers has been reported to reduce the number of pinholes significantly.²⁷ This can be explained in terms of smoothing of the coated surface, reduction of mechanical damage and increased thermal stability.²⁰ Moreover, repeating the alternating process yields stacked structures that allow the organic layers to "decouple" the defects from neighboring inorganic layers. The best flexible barrier materials reported so far are based on

polyacrylate/ Al_2O_3 alternating coatings produced in a multi-step process. The organic layer is produced by flash evaporation of an acrylic monomer that is subsequently cured by UV light,²⁸ while the inorganic coating is usually deposited by reactive sputtering. WVTR of about $2 \times 10^{-6} \text{ g m}^{-2} \text{ day}^{-1}$ has been reported,²⁹ yielding 5 mm^2 OLED lifetime of 2,500 hours.³⁰ In the following we report comparative shelf lifetime studies on solar cells based on two different conjugated polymer:fullerene blends, fabricated on, and encapsulated with flexible, transparent ultra-high barrier material produced entirely by PECVD.

2. EXPERIMENTAL

2.1. Plasma Enhanced Chemical Vapor Deposition of Ultra-high Barrier Coatings

The encapsulation films used in this study are poly(ethylene naphthalate) (PEN) substrates coated with ultra-high barrier multilayer coatings deposited by PECVD. The choice of PEN instead of PET is justified by its higher temperature of glass transition that is compatible with production steps such as heat treatment of the final devices. The ultra-high-barrier coatings comprise alternating transparent layers of dense inorganic material and plasma-deposited organic material, the hard inorganic material being the first layer in direct contact with the flexible substrate. Inorganic and organic layers, namely SiO_X and PECVD-deposited organosilicon, are grown sequentially on the PEN film in the same deposition chamber. The former (SiO_X) are produced from a volatile organosilicon precursor under oxidative plasma conditions, while the latter are deposited using similar organosilicon precursor but in a non-oxidizing plasma. The final ultra-high barrier consists of five layers of about 100 nm, resulting in a total thickness of about 500 nm, as measured by spectroscopic ellipsometry. These coatings are transparent with an optical transmission greater 85% over the entire visible spectrum, they show good resistance to common organic solvents and do not deteriorate during washing with isopropanol, acetone, chlorobenzene or toluene. Oxygen- and water vapor permeation measurements performed with MOCON Oxtran and Permatran instruments are below their detection limits which are $0.005 \text{ cm}^3 \text{ m}^{-2} \text{ day}^{-1} \text{ atm}^{-1}$ and $0.005 \text{ g m}^{-2} \text{ day}^{-1}$, respectively. Our barrier material survives an accelerated "Calcium test" (50°C, 85% relative humidity) for more than 1,000 hours, which corresponds to at least 10,000 hours under normal ambient conditions.

2.2. Preparation of the Solar Cells

2.2.1. Solar Cells based on PPV:fullerene blends

First, indium tin oxide (ITO) electrodes were sputtered through a mask onto the $75 \times 42 \text{ mm}^2$ barrier substrates. Adhesion was sufficiently good and the sheet resistance was found to be about $60 \Omega/\text{square}$. The transparent substrate was then coated with a thin ($\sim 90 \text{ nm}$) layer of poly(3,4-ethylenedioxythiophene) doped with poly(styrene sulfonate) (PEDOT:PSS, Baytron, Bayer AG), deposited by doctor blading. On top, a solution of poly[2-methoxy-5-(3',7'-dimethyloctyloxy)-1,4-phenylene-vinylene] (MDMO-PPV, Covion GmbH) : 1-(3-methoxycarbonyl) propyl-1-phenyl[6,6]C61 (PCBM, Nano-C Inc.) with a mass ratio 1:4, was deposited by the same technique; the resulting active layer was 200 nm thick. Finally, an aluminum electrode was thermally evaporated through a second mask. Unlike the conjugated polymer deposition procedures that were done in air, the encapsulation step was carried out in a glove box in pure, dry Ar atmosphere. A two-component epoxy resin was used to seal the device with a top cap made of the same barrier material as the substrate (Fig. 2). After curing the epoxy, the overall structure can still be bent appreciably, as illustrated in Fig. 3. The cells showed a quite low power conversion efficiency under AM 1.5 conditions of about 0.035% (fill factor, $FF = 0.26$; $V_{OC} = 1,100 \text{ mV}$; short circuit current (I_{SC}) = 0.12 mA cm^{-2}). This is explained by the very large surface area ($30 \times 57 \text{ mm}^2$) made of two non-optimized series connected cells, combined with an ITO having a quite high sheet resistance. However, we believe that this drastic limitation due to an overwhelming series resistance does not influence our lifetime study.

2.2.2. Solar Cells based on polythiophene:fullerene blends

The device preparation steps were the same as described above for the MDMO-PPV:fullerene blends. Poly(3-hexyl)thiophene (P3HT) was used as donor component and PCBM as acceptor in the mass ratio of 1:2. The photoactive layer was deposited by doctor blading from a solution in chloroform resulting in a film thickness of $\sim 200 \text{ nm}$. After deposition of the Al top contact, the P3HT based devices underwent a heat treatment at $\sim 110^\circ\text{C}$ for approximately three minutes. The active area of the solar cells was roughly 60 mm^2 ($20 \times 3 \text{ mm}^2$).

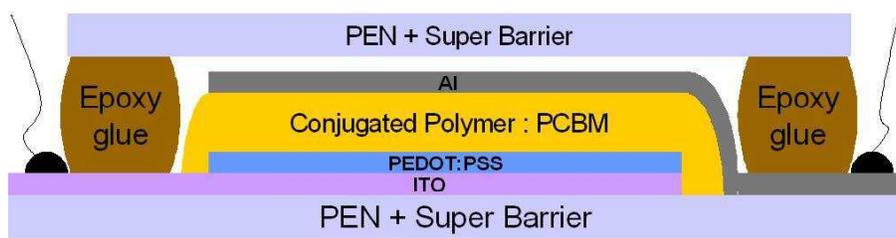


Figure 2. Cross sectional view of the conjugated polymer:fullerene solar cells investigated here.

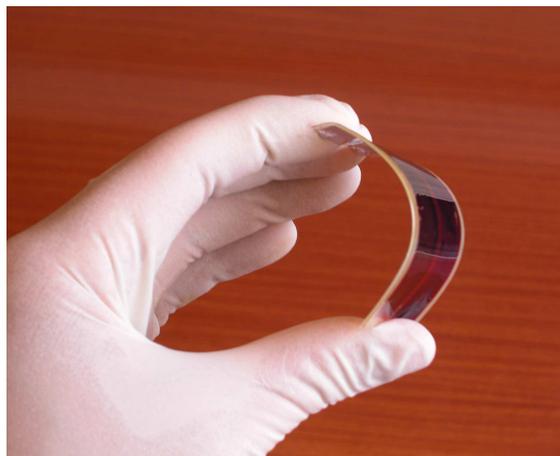


Figure 3. Picture of a bent device.

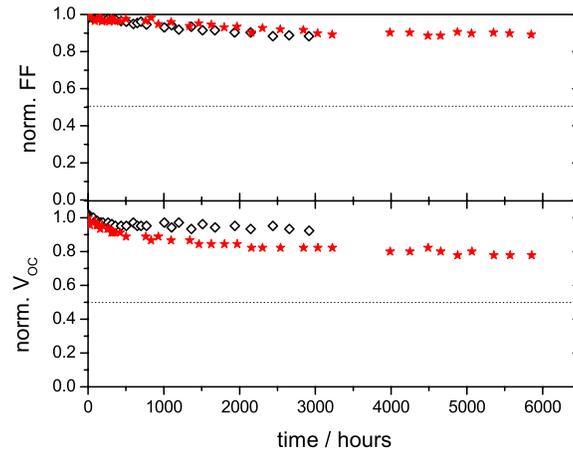


Figure 4. Normalized V_{OC} and FF of solar cells based on MDMO-PPV:PCBM blends (\diamond) and P3HT:PCBM blends (\star) encapsulated with flexible gas barrier material versus storage time in the dark under ambient air.

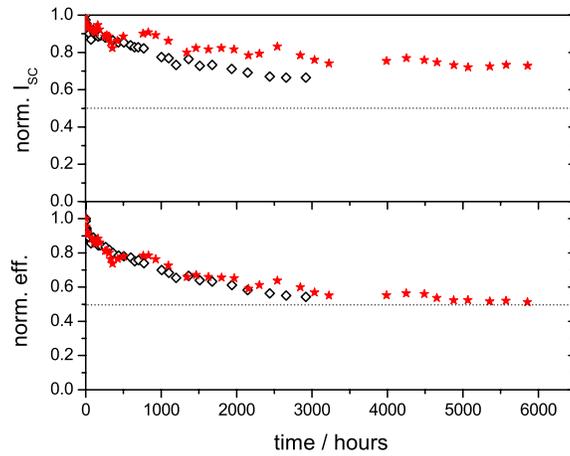


Figure 5. Normalized I_{SC} and efficiency of solar cells based on MDMO-PPV:PCBM blends (\diamond) and P3HT:PCBM blends (\star) encapsulated with flexible gas barrier material versus storage time in the dark under ambient air.

Due to the lower surface area (decreased limitation from the high sheet resistance of ITO) as well as the generally higher power conversion efficiency in P3HT:PCBM composites⁶ compared to MDMO-PPV:PCBM cells,³¹ the efficiency of these cells was $\sim 0.25\%$. Current-voltage ($I - V$) measurements were performed with a Keithley 236 source measure unit under simulated AM 1.5 solar irradiation (Steuernagel Lichttechnik GmbH, 100 mW cm^{-2}). Between successive measurements, the solar cells were stored in the dark under ambient air ($\sim 20\text{-}25^\circ\text{C}$; 35-50% relative humidity).

3. RESULTS

Figure 4 shows the evolution of the normalized V_{OC} and Fill Factor (FF) of typical solar cells versus storage time. We observed that aging of the cells results in the development of a back diode that tends to drastically reduce

FF. However, in the case of MDMO-PPV:PCBM based devices with flexible barrier encapsulation, V_{OC} remains at about 90% of its initial value after 3,000 hours, as does FF. Fig. 5 presents the evolutions of the normalized short circuit current (I_{SC}) and of the efficiency η , calculated by

$$\eta = \frac{V_{OC} \cdot I_{SC} \cdot FF}{P_{in}}, \quad (1)$$

where P_{in} is the incident light power. The difference between their decay rates arises from the fact that η accumulates the decrease of V_{OC} , FF and I_{SC} . After 3,000 hours, I_{SC} and η still possess 67% and 54% of their initial values, respectively, in case of the PPV containing samples. A significant part of the observed performance drop occurs during the first 50 hours of the experiment.

Qualitatively the degradation of P3HT:PCBM solar cells follows a similar behavior, but happens on a longer time scale. The FF is stable over long times, degrading only slowly. While V_{OC} decays faster than in the case of MDMO-PPV based samples, I_{SC} decreases significantly slower from the initial value. η accumulates all these contributions and stays above 50% of its starting value up to 6,000 hours.

The flexible gas barrier encapsulation used in this study drastically increases the lifetimes of conjugated polymer:fullerene solar cells. While a simple PET film provides a shelf lifetime of less than six hours,^{32,33} the permeation barrier material produced by PECVD yields lifetimes exceeding 3,000 hours in MDMO-PPV:PCBM. This is comparable to those reported for the same type of cells encapsulated between glass plates.¹⁸ This suggests that degradation of the device characteristics may not be related to the flexible gas barrier itself, but to intrinsic instability of the photoactive material. This is supported by the significantly longer lifetimes achieved by using P3HT:PCBM blends as active layers of the solar cell devices. The rapid decay observed in both cases during the first 50 hours that accounts for 20% of the overall performance loss is quite intriguing. We believe that it may possibly be explained by two different phenomena: (i) As mentioned earlier, the cells were produced under normal laboratory ambient conditions, no special measures being taken to remove residual oxygen adsorbed on the surface or absorbed in the bulk of the device; (ii) the epoxy glue was used as received without any step of degassing before application, though it might contain some traces of moisture or oxygen. One possible solution to minimize these effects could be to use a getter material for moisture and oxygen.^{20,29} As known from OLED display manufacturing, getter encapsulated together with the device reacts more rapidly with O_2 and H_2O than the active semiconducting polymers. The 6,000 hours of device stability achieved so far in this study is not sufficient for this type of device on the market, yet it represents a critical step in the realization of long-lived flexible plastic solar cells.

4. CONCLUSIONS

A novel type of transparent, flexible ultra-high barrier material, entirely produced by PECVD, has been used to encapsulate MDMO-PPV:PCBM and P3HT:PCBM solar cells. Shelf lifetimes up to 6,000 hours have been realized with the devices based on the polythiophene derivative and above 3,000 hours for the MDMO-PPV containing devices. Bare commercial PET films permit lifetime of less than six hours.³² This extended lifetime equals those previously reported for the same type of devices encapsulated between glass plates. Using more stable materials such as P3HT for the photoactive layer, the shelf lifetime could be pushed to approximately 6,000 hours, equivalent to more than eight months. The combination of less sensitive materials like polythiophenes and this new transparent flexible gas barrier, that is compatible with roll-to-roll processing, may open the way to long-lived flexible plastic solar cells.

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