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ARTICLE

Reversible degradation in ITO-containing organic photovoltaics under concentrated sunlight

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Stability of ITO-containing and ITO-free organic solar cells were investigated under simulated AM 1.5G illumination and under concentrated natural sunlight. In both cases ITO-free devices exhibit high stability, while devices containing ITO show degradation of their photovoltaic performance. The accelerated degradation under concentrated sunlight (up to 20 suns) in ITO-containing devices was found to be reversible. Dark exposure of degraded samples can partly restore performance. A possible underlying mechanism for such phenomenon is discussed.

1. Introduction

Organic photovoltaics (OPV) constitute promising low-cost approach for solar energy conversion. In order to reach an impact on the high power market on the longer term, OPV should provide high power conversion efficiency, low production costs and long term operational stability. The low cost potential is based on the use of low-cost materials and substrates and the very high production speeds which can be reached by roll-to-roll printing and coating techniques¹⁻⁷. Removal of expensive indium tin oxide (ITO) from the architecture of organic solar cells contributes towards further decreasing the manufacturing cost. Substitution of ITO by composite electrode, containing highly conductive PEDOT:PSS and current collecting grids⁸⁻¹¹ was demonstrated to produce large area devices without efficiency losses and to allow solution processing of bottom electrode. The latter is more cost-efficient in comparison with vacuum deposition of ITO¹².

Further steps towards the development of large-area OPVs require highly stable devices with long lifetime. Earlier, it has been shown that ITO-free devices exhibit higher stability compared to identical devices with ITO-electrode¹³. However, the origin of the degradation was not discussed. Several attempts have been made on comparison of ITO-free devices

with different architectures¹⁴. The understanding of degradation mechanism in the devices with different architecture is a first step to get stable OPV devices and modules.

In order to get the understanding of degradation mechanism in ITO-containing and ITO-free devices, we compare stability of two types of organic solar cells (ITO-containing and ITO-free) under simulated "1 sun" illumination (1 sun = 100 mW/cm²) and accelerated degradation of these devices under concentrated natural sunlight¹⁵⁻¹⁹.

2. Experimental

The series of ITO-based and ITO-free devices were prepared on glass substrates. The devices had following layer sequences:

glass/ITO/PEDOT:PSS/P3HT:PCBM/LiF:Al/encapsulation;

glass/Ag-grids/h.c.PEDOT:PSS/P3HT:PCBM/LiF:Al/encapsulation.

Schematic illustration of the devices is shown in Fig. 1a. The materials used for the device preparation were the following: Clevios P VP AI 4083 PEDOT:PSS from Heraeus; high conducting Orgacon PEDOT:PSS from Agfa-Gevaert; Poly(3-hexylthiophene) (P3HT) from Plextronics and [6,6]-phenyl-C61-butyric acid methyl ester (PCBM) from Solenne BV. The devices were prepared as reported earlier^{11, 20-22}. All devices were encapsulated either with thin-film encapsulation²³ or with metal lid using Huntsman Araldites 2014-1 sealer. To prevent side leakage, prior to encapsulation, the organic layers were structured via laser ablation. The active area of the devices was 3.76 cm².

The one sun lifetime test was done by measuring the performance of the devices over time. In between the measurements the samples were light soaked with constant illumination of a G2 light engine combined with a S-bulb

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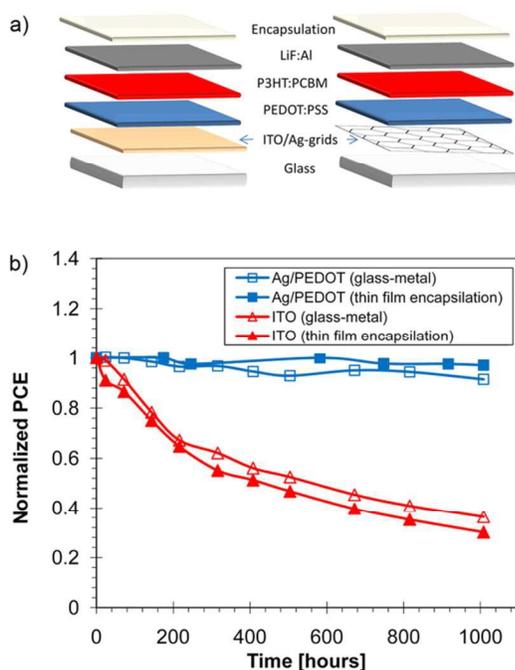
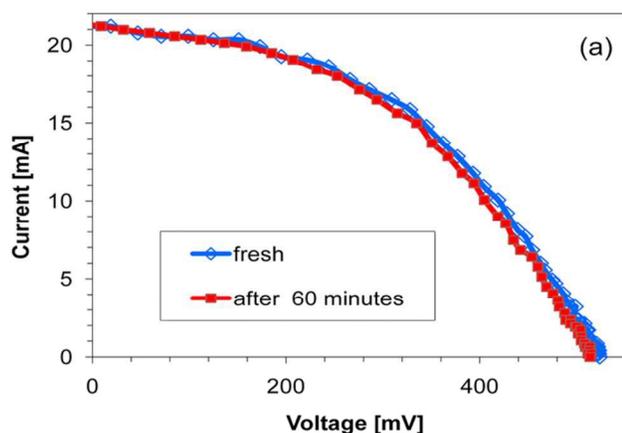


Fig. 1. (a) - Schematic illustration of device architectures; (b) - Relative efficiency change in the devices under 1 sun illumination, at 45°C.

(Solaronix), which provides simulated 1.5AM condition, although with a lower UV and IR content. The temperature of the samples was 45°C during light soaking. Current–voltage (I–V) curves were measured using simulated AM 1.5 global solar irradiation (100 mW/cm²), using a WXS-300S-50 solar simulator (WACOM Electric Co.)

The cell exposure by concentrated sunlight (20 suns) was performed in Sede Boker (desert Negev, Israel). Natural sunlight was concentrated using dual-axis tracking flat heliostat that reflects light into the laboratory, where a flat mirror (with a hole at its center) tilted at 45° redirects the light upward to a 526 mm-diameter paraboloidal dish of numerical aperture of 0.4, whose focal plane is just below the tilted mirror^{24, 25}. The light intensity was moderated by a louvered shutter between the heliostat and the flat indoor mirror. It should be noted that the spectrum measured at 'noon time ± 2-3 hours' at Sede Boqer (Lat. 30.8°N, Lon. 34.8°E, Alt. 475 m) is very close to the AM



1.5G spectrum²⁵. Flux uniformity in both systems was achieved using kaleidoscopes placed between the paraboloid focal point and the cell. The incident power of concentrated sunlight was measured with a pyrometer with 5% accuracy.

I–V measurements of the cells before and after their exposure by concentrated sunlight were performed with 1.5G spectrum by opening a shutter above the cell and illuminating the cell during I–V tracing only (< 1 sec) to avoid excessive degradation and temperature variations. In addition to the conventional PV characterization under 1 sun, we performed I–V measurements with varied sunlight intensities (up to 5 suns). EQE spectra were measured using of a PV spectral response measurement system (“TECHNOEXAN” Ltd, Ioffe Physical-Technical Institute, Russia).

For accelerated lifetime testing using concentrated sunlight only devices with thin film encapsulation were selected. Devices with metal lid cannot be vastly cooled down during concentrated sun illumination because of the space between the glass substrate with OPV stack and metal lid. The nitrogen space (devices were encapsulated in nitrogen atmosphere) between front and back sides of the sample slow down the cooling process during illumination. In such sample increasing of illumination doze will significantly increase the temperature. Thin-film encapsulation allows to make very thin and flat stack which can be cooled down to 30°C using integrated cooling system even at 20 suns illumination.

3. Results and discussion

All devices show very high stability in the dark at room temperature. The combination of “1 sun” simulated light and elevated temperature (45°C) shows a very rapid degradation of the ITO-based devices (Fig. 1b). Efficiency of the ITO-free devices remains almost unchanged during 1000 hours. The same trend is observed for both devices with thin film and metal lid encapsulation. It indicates degradation of ITO-based devices does not associated with the type of encapsulation type, but has intrinsic nature.

Identical devices were exposed to concentrated sun light. Fig. 2 presents the I–V curves and EQE spectrum of the ITO-free devices before and after the illumination by 20 suns for 60 minutes. One can see no degradation of EQE and short-circuit current (I_{sc}) and very slight degradation of the open-circuit voltage (V_{oc}) and the full-factor (FF). Further illumination of the ITO-free cell by 20 suns for two successive periods of 60

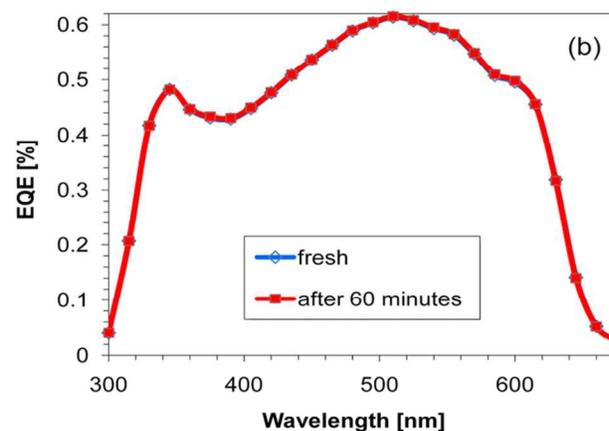


Fig. 2. I–V curves measured at 1 sun (a) and EQE spectra (b) for the ITO-free cell measured before and after the illumination at 20 suns for 60 minutes.

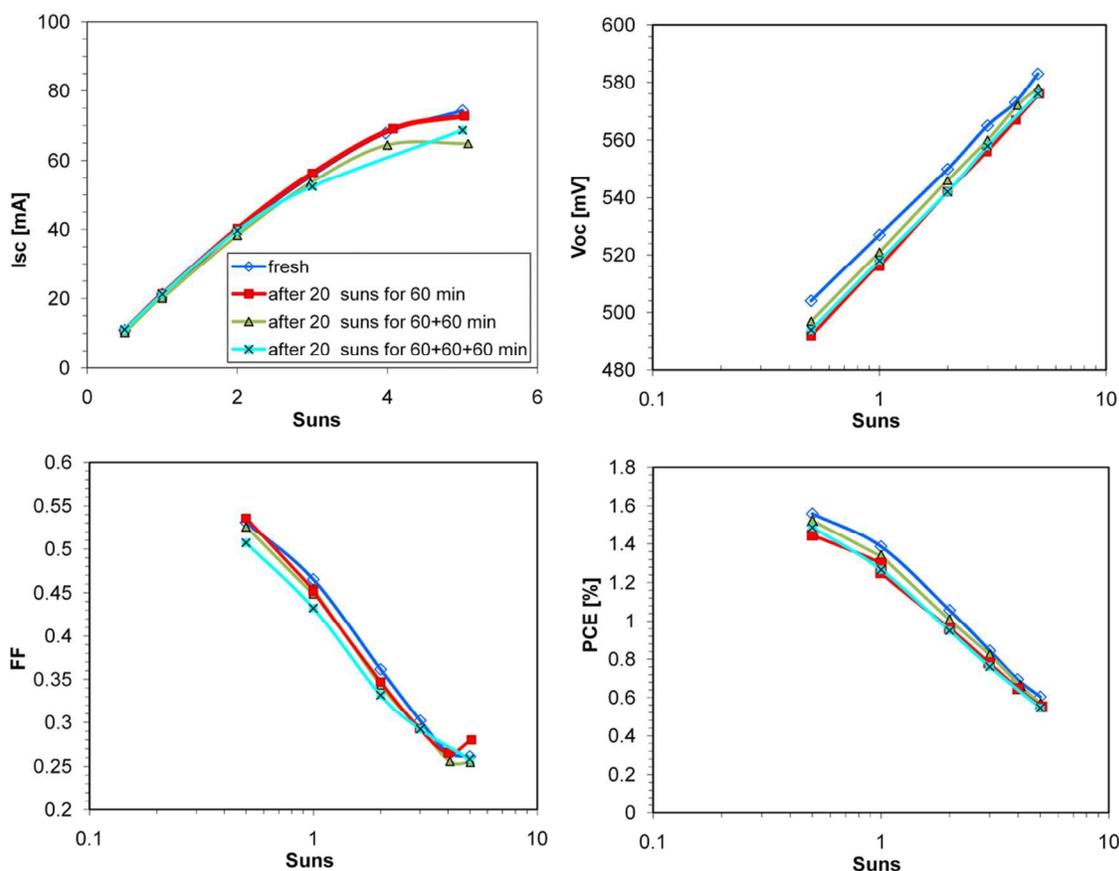


Fig. 3. Light intensity dependences of the ITO-free device principal photovoltaic parameters before and after illumination by 20 suns for three successive periods of 60 minutes.

minutes was shown to result in similar behavior. Fig. 3 depicts light intensity dependence of the device principal photovoltaic parameters measured in this experiment.

The I_{sc} of fresh cell exhibit linear light intensity dependence up to ~ 3 suns (Fig. 3a). However, under higher solar concentration (above 3 suns) the sub-linear dependence of I_{sc} on light intensity is in evidence. The FF decreases monotonically with light intensity (Fig. 3c). These results are in agreement with our previous observations¹⁹. In inorganic PV

such behavior is attributed the cell series resistance losses that become more pronounced at high light intensity²⁶. In OPV, in addition to the conventional series resistance limitation, a number of underlying mechanisms for the sub-linear photocurrent can be suggested: enhancement of the voltage dependence of photocurrent²⁷, space charge limitation²⁸⁻³⁰, unbalanced transport of holes and electrons, also known as “ $\mu\tau$ -limited” process²⁹ and bimolecular recombination³¹⁻³³. One cannot distinguish these mechanisms just by the analysis

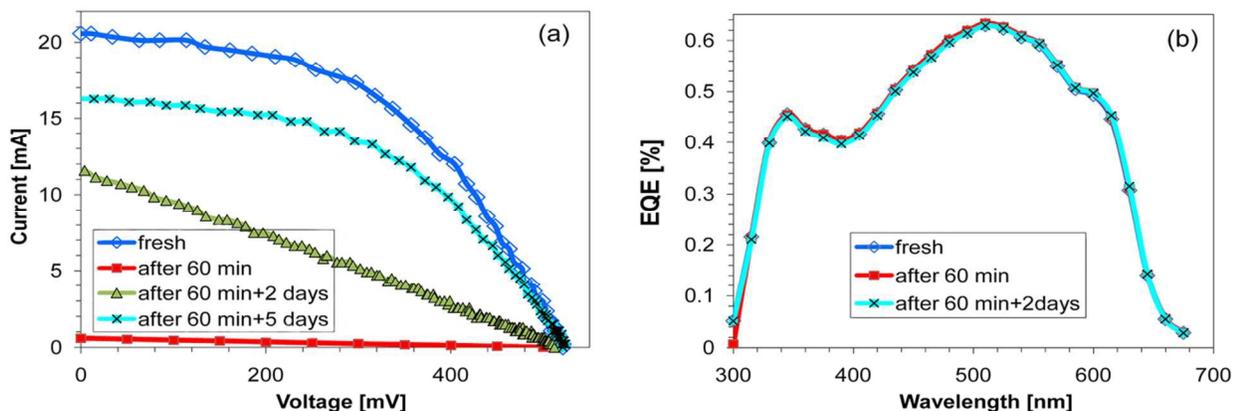


Fig. 4. I-V (a) and EQE (b) curves for an ITO-containing cell measured before and after the illumination by 20 suns for 60 minutes as well as after 2 and 5 days of keeping the cell in the dark after the exposure.

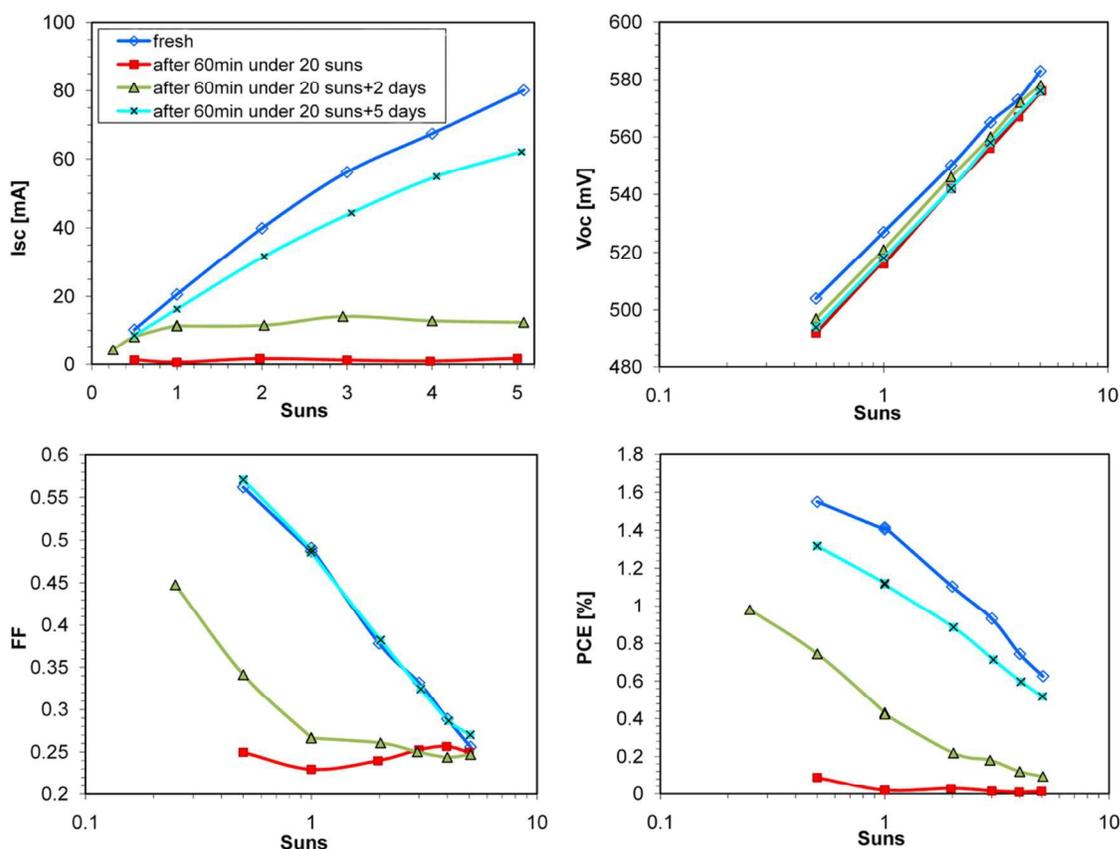


Fig. 5. Light intensity dependences of the ITO-containing device principal photovoltaic parameters measured before and after the illumination by 20 suns for 60 minutes as well as after 2 and 5 days of keeping the cell in the dark after the exposure.

of I-V curves and dependencies of the PV parameters on light intensity. We will use the term “charge collection limitation” to refer such characteristic behavior.

Exposure of the ITO-free cell by 20 suns did not affect a general trend of light intensity dependences of its PV principal parameters (Fig. 3). Slight intensity-independent decrease in Voc and FF was observed. The Isc showed no any degradation up to ~ 3 suns. Above ~ 3 suns the cell's Isc degradation was recorded and the sub-linear dependence of Isc on light intensity was found to be more pronounced.

On the other hand, the same light exposure (20 suns, 60 minutes) of an ITO-containing cell resulted in significant degradation of its PV performance (Fig. 4a). In particular, very strong decrease in FF and Isc was observed after the exposure. After the light exposure, the cell was stored in the dark and measured after two and five days. I-V measurements showed that during the dark storage the cells were partially recovered. It suggests that the observed degradation is (at least partly) reversible.

It should be noted, however, that the exposure did not affect the cell EQE spectrum (Fig. 4b). We explain the apparent contradiction between the EQE stability and Isc and FF substantial degradation by postulating that no photocurrent degradation occurred under the sunlight exposure. The Isc (and FF) degraded due to the cell series resistance increase during the exposure. Indeed, the light intensity in EQE setup is by ~ 3 orders of magnitude lower than that solar light even at 1 sun. The series resistance losses are neglected under low light intensity (in the EQE measurements), while they become

significant under solar irradiation (I-V measurements under 1 sun and higher solar concentrations). Our hypothesis is supported by I-V measurements with varied light intensities (Fig. 5).

The difference between two types of the investigated cells is only in the front contacts (ITO/PEDOT:PSS vs Ag/PEDOT:PSS). This fact brings us to conclusion that the degradation of ITO-containing cell is related to the degradation of ITO layer or/and its interface with the PEDOT:PSS and the corresponding increase in the cell series resistance.

In order to check whether the ITO resistance and transmittance changes due to light exposure, a non-encapsulated ITO-coated glass (the same as used for OPV cells) was irradiated for 60 minutes under 50 suns with the same cooling conditions as in the cells experiment. Prior and after the light exposure, the absorption and resistance of the ITO film was measured (the resistance was measured between two contacts ~ 2 cm distanced). The absorption results are given in Fig. 6.

The light dose in the ITO sample exposure was greater than that for the ITO-containing cell exposure by a factor of 2.5. Nevertheless, no absorption degradation was recorded, except of a minor decrease in the UV part. The resistance measurements did not show any changes due to the light exposure. We can conclude that the ITO by itself is stable even though it was not encapsulated.

Although bare ITO demonstrate high electrical and optical stability at concentrated sun light, there are many publications which point on instability on the ITO/active layers interface³⁴.

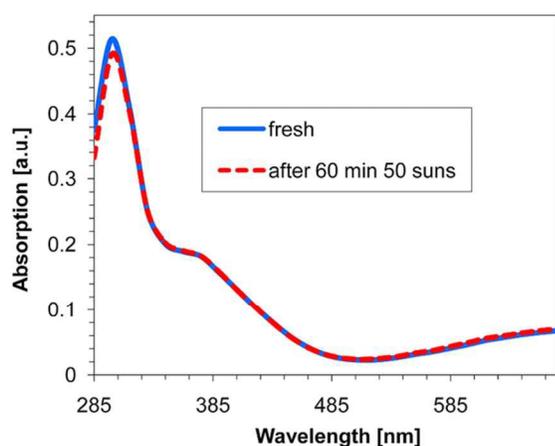


Fig. 6. UV-Vis spectra of an ITO film (not encapsulated) measured before and after the solar exposure by 50 suns for 1 hour.

³⁵ and instability of ITO/PEDOT:PSS interface in particular ^{36, 37}. We speculate that the cell's degradation can be explained by the interaction of ITO with the PEDOT:PSS at their interface. The interaction is attributed to the acidity of the PEDOT:PSS which facilitate the etching of ITO. The etching of ITO is accompanied by the transport of etch products through the PEDOT:PSS film ^{38, 39}, so called indium migration. Interaction of ITO and PEDOT:PSS in non-accelerated condition leads to slow degradation of ITO electrode and reduction of OPV performance. However, in accelerated lifetime test the interaction between ITO and PEDOT:PSS occurs much faster than the transport of interaction products from the interface can occur. Thus, just after accelerated degradation the most of the etch products still stay at the interface, and such surface modification of ITO impairs hole injection mechanism from the anode through the interface layer to PEDOT:PSS. Later, the etched products diffuse into the bulk of PEDOT:PSS and photoactive layer. The contact between ITO and PEDOT:PSS is improving and organic solar cells show reanimated performance.

We can suggest the following mechanism for the conductivity loss at the ITO interface. The low resistivity of ITO film is believed to be due to the large free carrier density, which is in the range of $1020/\text{cm}^3$ to $1021/\text{cm}^3$. It is generally accepted that these free carriers are generated by two processes ^{40, 41}: (1) Sn atom substitution of In atom and giving out one extra electron, and (2) oxygen vacancies acting as two electron donors. Due to its complicate crystal structure (80 atoms in an In_2O_3 unit cell), the conducting mechanism of ITO is still not fully understood ⁴⁰. Decomposition of ITO due to interaction with PEDOT:PSS leads to decrease of oxygen vacancies at the ITO interface, resulting in conductivity loss. However, migration of In (and probably Sn) ions from the interface restore oxygen vacancies at ITO interface and the reanimated current collection in OPV devices. It is important to mention that fill factor and J_{sc} did not reach initial values because resistance of ITO electrode was increased due to partial decomposition of ITO.

The interaction of ITO and PEDOT:PSS at non-accelerated condition is intensively described in the literature ^{38, 42, 43}. The main conditions influencing the interaction are acidity of PEDOT:PSS ⁴³, humidity level (especially for non-encapsulated samples) ^{38, 42}, processing and curing condition of PEDOT:PSS

(which is responsible for the amount of residual water in PEDOT:PSS) ⁴⁴, surface treatments of ITO ⁴⁵, etc. It is obvious from our study, that concentrated sun light also accelerate the degradation of the devices. Most likely the degradation occurs at ITO/PEDOT:PSS interface. As shown above there is no effect of concentrated sun light on pure ITO has been observed. Thus, the effect of concentrated sun light on the PEDOT:PSS layer is of great interest.

Effect of UV-light irradiation on the conductivity and work function of PEDOT:PSS have been intensively investigated ⁴⁶⁻⁴⁸. However, in most of the publications the UV treatments were performed in air or by applying UV-ozone. Nagata et al. ⁴⁸ have clarified the respective roles of UV light irradiation and exposure to ozone gas on the properties of PEDOT:PSS. They have found that the main role of UV light is to decompose the chemical bonds in the PEDOT:PSS, resulting in a decrease of conductivity, while the ozone and atomic oxygen are absorbed and oxidize the surface, leading to an increase of work function. Xing et al. ⁴⁹ describe a simple method to increase the conductivity of PEDOT:PSS film by expose to UV light in vacuum. The difference in the results of Nagata et al. and Zing et al. (decrease and increase of conductivity, respectively) can be explained by presence or absence of oxygen during irradiation. From both studies it is clear that 254 nm UV light can break chemical bonds such as C-C, C-H, and C-OH. For PEDOT:PSS film, PSS is more detrimental under 254 nm UV exposure then PEDOT because the conjugated parts in PEDOT chain has a larger bonding energy then single bonds in in PSS chain ⁴⁸. Hong et al. ⁵⁰ have reported that there are two kinds of bonds in PSS can be broken under UV exposure: the cleavage of a α -hydrogen bond generates polystyrene radicals, and the second cleavage happens between sulfonic acid group and phenyl group.

We speculate that decomposed products of PEDOT:PSS and especially sulfonic acid derivatives etch ITO much faster than non-decompose PEDOT:PSS. The etch products, which are mostly In and Sn ions, will migrate to the bulk. However, as discussed above, after the concentrated sunlight exposure, which accelerate decomposition of PEDOT:PSS and therefore accelerated interaction with ITO, most of etched In and Sn ions are still located at the ITO/PEDOT:PSS interface and create effective barrier for the charges. Organic solar cells at this point look completely degraded. Solar cells still show reasonable voltage, but fill factor and current are significantly reduced, indicating the degradation of the electrodes. However, exposure in the dark and migration of etch products from the interface can restore the device performance.

4. Conclusions

Operational stability of ITO-containing and ITO-free organic solar cells was compared. ITO-containing OPV cells exhibited much faster degradation of their PV performance under exposure of simulated one sun light and natural concentrated sunlight. The accelerated degradation under concentrated sunlight was found to be reversible and can be attributed by the increase in the cell series resistance limitation. The ITO/PEDOT:PSS interface is suggested to be responsible for the degradation. The reversible degradation effect under concentrated sunlight is attributed to a slow migration of the etch products from the interface. Dark expose provides sufficient time for the etch product to migrate to the bulk, and thereby reanimate PV performance.

It is worth to remember that operational lifetime in real conditions consists of alternating illumination and dark exposures during day and night time, respectively. Performance of the cell can degrade under illumination. However, during the night the reaction products can migrate to the bulk, thereby restoring the device performance. This fact should be taken into consideration when the indoor lifetime tests are performing with constant illumination under one sun or accelerated conditions. This problem should be addressed in further detailed study comparing the results of outdoor and indoor stability assessments of OPV cells with various device architectures.

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