

# Combined Characterization Techniques to Understand the Stability of a Variety of Organic Photovoltaic Devices - the ISOS-3 inter-laboratory collaboration

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## ABSTRACT

This work is part of the inter-laboratory collaboration to study the stability of seven distinct sets of state-of-the-art organic photovoltaic (OPVs) devices prepared by leading research laboratories. All devices have been shipped to and degraded at the Danish Technical University (DTU, formerly RISO-DTU) up to 1830 hours in accordance with established ISOS-3 protocols under defined illumination conditions. In this work we present a summary of the degradation response observed for the NREL sample, an inverted OPV of the type ITO/ZnO/P3HT:PCBM/PEDOT:PSS/Ag/Al, under full sun stability test. The results reported from the combination of the different characterization techniques results in a proposed degradation mechanism. The final conclusion is that the failure of the photovoltaic response of the device with time under full sun solar simulation, is mainly due to the degradation of the electrodes and not to the active materials of the solar cell.

**Keywords:** Polymer solar cells, organic solar cells, stability, metal electrodes, polymer/metal interaction, degradation.

## INTRODUCTION

This work is part of a series of publications from the research collaboration established at the Third International Summit on Organic Photovoltaic Stability (ISOS-3). Seven distinct state-of-the-art OPV devices were prepared by leading research laboratories (NREL, IMEC, HOLST, ISE, RISO and IAPP). The devices were aged under different degradation conditions: accelerated full sun simulation; low level indoor fluorescent lighting and dark storage. Our first publication deals with the overall degradation behaviour of the devices by reporting on the changes observed on power conversion efficiency with aging time.<sup>1-2</sup> The second report deals with the analysis of the degradation of the solar cells by the combination of different imaging characterization techniques like laser beam induced current

(LBIC), dark lock-in thermography (DLIT), electroluminescence (ELI) and photoluminescence (PLI) imaging.<sup>3</sup> In the third publication, we apply the Incident Photon-to-Electron Conversion Efficiency (IPCE) and *in-situ* IPCE techniques as tools to analyze the different degradation paths observed on the OPV devices.<sup>4</sup> The fourth publication deals with the time-of-flight secondary ion mass spectrometry (TOF-SIMS) analysis, a technique producing direct chemical information.<sup>5</sup> The secondary technique is X-ray photoelectron spectroscopy (XPS), which also produces direct (but complementary) chemical information. The basic information of TOF-SIMS is mass spectral information, i.e. chemical information. In this work we present a possible degradation mechanism for the NREL sample degraded under full sun stability test. The results are based on the conclusions from all the analytical methods applied in this collaborative work. Our results indicate that the main cause of device failure comes from the electrodes and not from the active materials of the solar cell.

## CHARACTERIZATION TECHNIQUES

### IPCE and *in-situ* IPCE analyses

The incident photon-to-electron conversion efficiency (IPCE) analyses were carried out in a QE/IPCE measurement System from Newport (300 W Xe light source, digital lock in measurement with NIST calibrated detector) at 10 nm intervals between 300 and 1000 nm. A calibrated photodiode from Hamamatsu (S1227-1010BQ) is also used to calibrate the equipment before each experiment. The results were not corrected for intensity losses due to light absorption and reflection by the glass support. Analyses were carried out in the dark without light bias. For the results, no interference effects were considered. The *in-situ* IPCE analysis refers to the analysis of a sample by IPCE at regular periods of time when the testing atmosphere is changed from ambient air to N<sub>2</sub> atmosphere. The method permits to monitor any changes observed on the solar cell when oxygen is eliminated from the environment. It is also an easy method to identify the materials that are more susceptible to degradation due to the ambient atmosphere. To carry out the *in-situ* IPCE analyses, devices were introduced into a home-made holder designed at CIN2. The CIN2's cell holder has been described before for small devices (5 cm diameter window).<sup>4</sup> Briefly, it consists of a two-piece glass reactor with a cooling jacket for temperature control, with a home-designed o-ring sealed cap. It has ports for thermocouple, inlet and outlets for low pressure gas flow, quartz window and cable connections. Once the device is placed inside the cell holder, the *in-situ* IPCE test was carried out every few minutes at the same time that N<sub>2</sub> was introduced into the cell holder at constant rate. Thus, IPCE curves were recorded from the transition from ambient atmosphere to the inert N<sub>2</sub>. The comparison of the normalized IPCE spectra obtained in air and under N<sub>2</sub> atmosphere gives an indication of the peaks and/or wavelength region affected by the atmosphere. IV curves were carried out at 1000 W m<sup>-2</sup> in a sun simulator KHS Solar Constant 1200 calibrated with both a pyranometer (Kipp & Zonen, CM 4) and a solar reference cell from ABET tech (NIST traceable calibration). The IV curves were recorded with a Keithley 2601 applying the Solar\_Cell\_v4\_SP software.

### Imaging Techniques

Luminescence imaging makes use of the capability of the organic semiconductor or the respective heterojunction to yield radiative de-excitation processes. This method yields the complete image of the device at once, possibly enabling very fast characterization. The challenge lies in the quantitative detection of the generally very weak luminescence signals arising from organic solar cells, which are orders of magnitude lower than for organic light emitting diode (OLED) devices. The experimental setup for performing luminescence imaging is described in.<sup>3</sup>

### TOF-SIMS analyses

The TOF-SIMS analyses were performed using a TOF-SIMS IV (ION-TOF GmbH, Münster, Germany). 25-ns pulses of 25-keV Bi<sup>+</sup> (primary ions) were bunched to form ion packets with a nominal temporal extent of <0.9 ns at a repetition rate of 10 kHz yielding a target current of 0.7 pA. These primary ion conditions were used to obtain mass spectra, ion images, and depth profiles. Depth profiling was performed using an analysis area of 200 × 200 μm<sup>2</sup> centered in a sputter area of 300 × 300 μm<sup>2</sup>. 30 nA of 3-keV Xe<sup>+</sup> was used as sputter ions. An encapsulated IMEC devices was analyzed in a slightly different way. The encapsulation was removed and the Al/Ag/MoO<sub>3</sub> stack was partly removed. 11 × 8 mm<sup>2</sup> surface areas were then imaged each covering four cells on the device. These images were cropped to sizes corresponding to the individual cells (5.2 × 2.7 mm<sup>2</sup>). Depth profiling was performed at various surface locations using an analysis area of 500 × 500 μm<sup>2</sup> centered in a sputter area of 750 × 750 μm<sup>2</sup>. For all analyses electron bombardment (20 eV) was used to minimize charge built-up at the surface. Desorbed secondary ions were accelerated to 2 keV, mass analyzed in the flight tube, and post-accelerated to 10 keV before detection.

## RESULTS AND DISCUSSION

The degradation mechanisms that are involved on the failure of OPVs are many and discrete. Here we describe one possible degradation path (among several within a device) for the inverted Organic solar cell from NREL. Among the solar cells analyzed in this collaborative work, the NREL and IMEC devices, are un-encapsulated inverted OPVs. In both cases, the solar cells have the configuration ITO/ZnO/PCBM:P3HT/HEL/Ag, where HEL means hole extraction layer. In the case of NREL the HEL is a PEDOT:PSS layer, in the case of IMEC the HEL is MoO<sub>3</sub>. In both cases solar cells are located on the right and left side of the substrate (6 solar cells/substrate for NREL and 12 in the case of IMEC). We work with the conclusions observed from our four published works<sup>2-5</sup> as follows:

**Conclusions from the IPCE analyses:** Figure 1 shows the schematic representation of these inverted OPVs, NREL and IMEC, and the respective images of their solar cells. The normalized IPCE spectras in Figure 1 correspond to the NREL (Figure 1a) and IMEC (Figure 1b) devices. Since both devices are very similar, almost the same IPCE peaks can be identified: the active materials P3HT:PCBM blend can be observed by the peaks corresponding to the P3HT at around 490 nm, 520 nm and 600 nm, and the PCBM between 450 and 600 nm. The electron extracting layer (EEL) ZnO is present in both devices as a peak at about 380 nm. In the case of the hole extracting layer (HEL), a peak at 340 nm corresponding to the MoO<sub>3</sub> layer is shown in the IMEC sample (a peak not present in the NREL spectra as expected). Nevertheless, the PEDOT:PSS adsorption for the NREL sample is not clearly observed. PEDOT adsorbs above 600 nm, thus the adsorption of the P3HT must be overlapping the peak at those wavelengths. In the case of the IMEC device the UV region below 400 nm is wider than for the NREL solar cell, due to the absorption of both, the electron extraction layer (EEL) made of ZnO and the hole extraction layer (HEL) made of MoO<sub>3</sub>. Finally, we have observed a peak that appears at 425-440 nm. This peak has been attributed to the interaction between PEDOT:PSS and the metal electrode Ag.

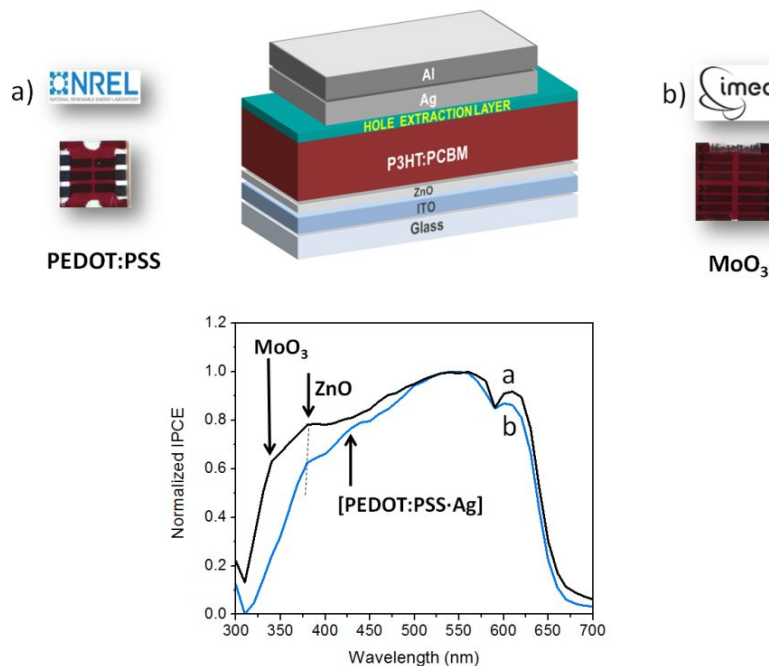


Figure 1. Normalized IPCE analysis of two inverted "air-stable" organic solar cells from NREL (a) and IMEC (b). In both cases the solar cell configuration is Glass/ITO/ZnO/P3HT:PCBM/HEL/Ag/Al. Where HEL means hole extraction layer and is PEDOT:PSS for the NREL sample (a) and MoO<sub>3</sub> layer for the IMEC (b).<sup>3</sup>

Figure 2 shows the normalized IPCE spectra and the corresponding IV-curves for a set of 6 NREL samples (all on the same substrate) degraded under the full sun stability test at T<sub>71</sub>. Since all the solar cells are part of the same substrate, the same degradation level is expected for all the samples. Yet, the inhomogeneous response observed among the devices has been attributed to the position of the solar cell within the substrate, and the effect of the hygroscopic nature of the PEDOT:PSS, as described in our full papers.<sup>4</sup> Moreover, the IPCE spectra of Figure 1a reveals that the adsorption range below 450 nm shows the most drastic changes. This adsorption range correspond to the peak attributed to the [PEDOT:PSS-Ag] interaction and to the ZnO layer. In both cases, these changes are attributed indirectly to the HEL layer (Ag), due to its possible interaction with PEDOT, and the EEL layer (ZnO). The behavior observed on the IV-curves (labeled as 1,2,3) together with the reduction in the V<sub>oc</sub> indicates power losses due to the presence

of shunting resistance on the device. These process are mainly due to manufacturing defects rather than to the active materials of the device.

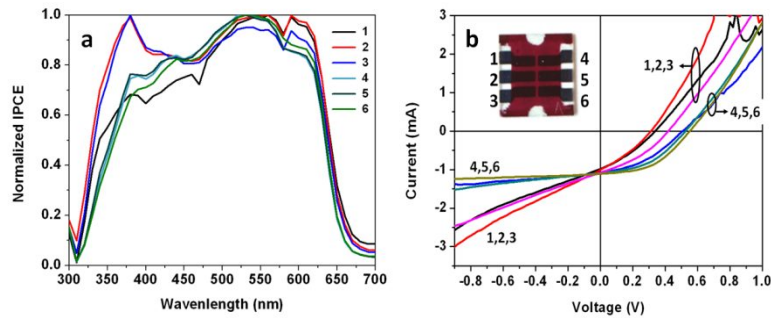


Figure 2. Normalized IPCE spectra of the 6 solar cells of a NREL sample after the Full sun stability test at  $T_{71}$  (a) and the corresponding IV curves (b). Among all solar cells, device No 2 (in red) was the one chosen initially to carry out the stability tests at RISØ-DTU.<sup>4</sup>

**Conclusions from the imaging analyses.** Analysis carried out by the imaging techniques for a NREL sample degraded under the full sun stability test until  $T_{00}$  is shown in Figure 2 (LEFT). The degradation of the device is also monitored by the IV-curves at different stages of the degradation test, Figure 2 (RIGHT). A conclusion from these analyses is the massive parasitic shunting observed at the end of the device lifetime, leaving no room for photovoltaic operation. The response has been attributed to the electromigration of Ag that led to penetration of the 40 nm thin PEDOT:PSS interlayer. The Ag migration finally leads to the formation of silver oxide.

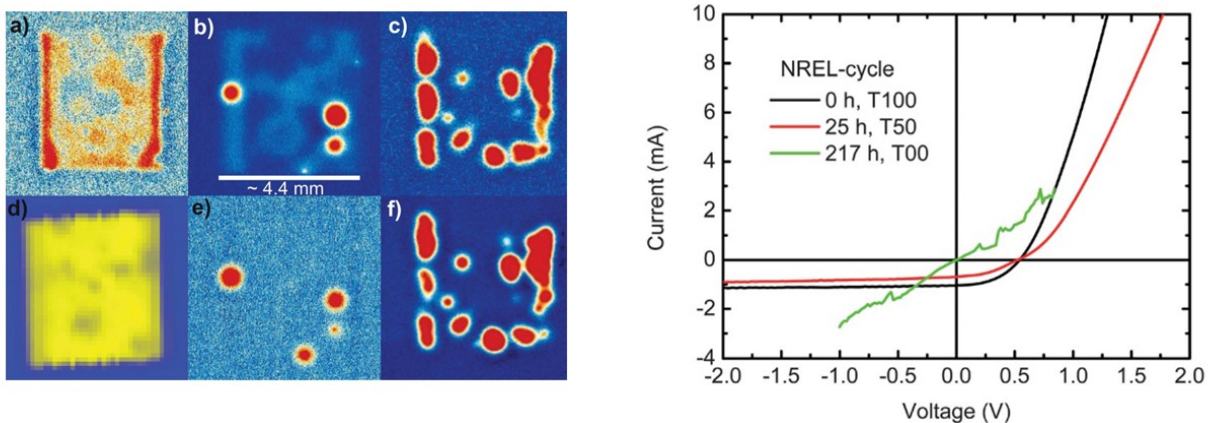


Figure 3. **LEFT:** DLIT characterization of the NREL solar cell from the cycle experiment without encapsulation: initially at nominally  $T_{100}$  (a), after 25 hours at  $T_{50}$  (b, e) and after total 217 hours under full sun simulation at  $T_{00}$  (c, f). The upper row shows the images obtained by 1.5 Volts forward bias excitation, the lower row shows the images obtained at -1.5 Volts reverse excitation and the initial operation ( $T_{100}$ , Risø DTU) detected by LBIC (d). Clearly the features present at  $T_{50}$  and  $T_{00}$  are dominated by parasitic shunting. **RIGHT:** IV-characteristics of the NREL cycle device at initial ( $T_{100}$ ), after 25 hours ( $T_{50}$ ) and after 217 hours ( $T_{00}$ ) under full sun illumination. After 25 hours stressing, the device exhibits initial features of blocking layer formation. At its final stage the device suffered from massive shunting, visible in the  $T_{00}$  IV-curve that crossed the origin of the current-voltage plot. Contacting problems led to the peculiar noisy IV-recording.<sup>3</sup>

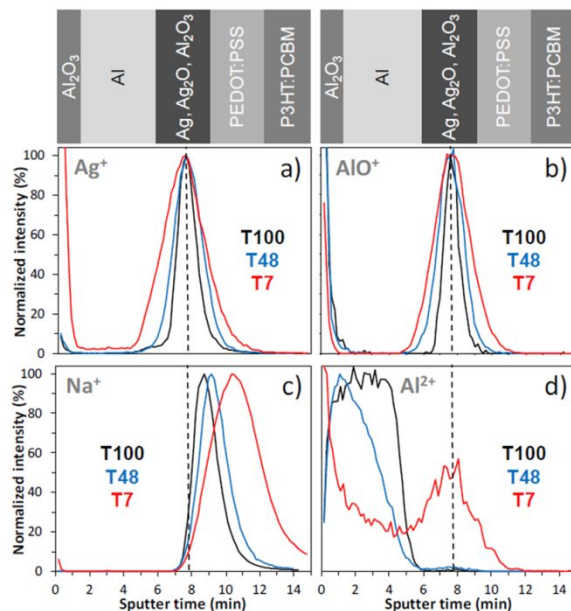
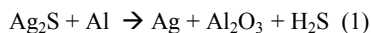


Figure 4. Normalized IPCE analysis of two inverted "air-stable" organic solar cells from NREL (a) and IMEC (b). In both cases the solar cell configuration is Glass/ITO/ZnO/P3HT:PCBM/HEL/Ag/Al. Where HEL means hole extraction layer and is PEDOT:PSS for the NREL sample (a) and MoO<sub>3</sub> layer for the IMEC (b).<sup>5</sup>

**Conclusions from the TOF-SIMS analyses.** The TOF-SIMS analyses reported in our fourth publication allowed the identification of distinctive degradation compounds found at the end of the NREL lifetime under full sun stability test. First of all, the presence of Al<sub>2</sub>O<sub>3</sub> is confirmed. A very interesting observation is that the profiles representing the Ag, Al<sub>2</sub>O<sub>3</sub>, and PEDOT:PSS layers are observed systematically to widen for decreasing cell performance. Moreover, analyses revealed that the Al<sub>2</sub>O<sub>3</sub> from the Al/Ag interface is dissolving in the Ag layer that consequently thickens the Ag layer (Fig. 3a). When Al<sub>2</sub>O<sub>3</sub> expands into the Ag layer the result will be a widening of the AlO<sup>-</sup> profile as shown in Fig. 3b. The Al<sup>2+</sup> T<sub>100</sub> profile shown Fig. 3d is consistent with the expected depth profile for a normal Al electrode. However, the Al<sup>2+</sup> signal is systematically lost in the Al/Ag interface for decreasing cell performance. This supports the proposed phenomenon of Al<sub>2</sub>O<sub>3</sub> and apparently also Al dissolving in the Ag layer. This is not observed for the IMEC device suggesting that PEDOT:PSS is involved in the phenomenon, possibly from migration of water or acid from PEDOT:PSS to the Al/Ag interface.

## 4. CONCLUSIONS

As a summary, the combination of all characterization techniques reveals as the principal cause of device failure, the degradation of the electrodes (and not the active materials of the device). In all cases the oxidation of Al<sub>2</sub>O<sub>3</sub> is directly or indirectly identified by all the techniques.<sup>2-4</sup> The electromigration of Ag into the PEDOT:PSS layer has been reported by the imaging analyses<sup>3</sup> and indirectly observed by an IPCE peak at 430 nm.<sup>4</sup> The migration of metal electrodes, like Ag, into almost any material, is a very common and well-known phenomena in electronics. Nevertheless, the presence of PEDOT:PSS in the NREL solar cells could enhance the metal migration since the S-atoms of polymers like PEDOT or P3HT, are known to interact easily with Ag. This interaction depends strongly on the metal electrode used. For example, Al and Cr reacts with the C-atom adjacent to the S-atom in P3HT, while Cu and Ag interacts with the S-atom of the polymer resulting in the formation of a silver sulfide-type of compound.<sup>6-7</sup> The latter could explain the presence of the IPCE peak observed at 430 nm and attributed to the [Ag-PEDOT:PSS] formation. The TOF-SIMS analyses show that the Al<sub>2</sub>O<sub>3</sub> and Al from the Al/Ag interface is dissolving in the Ag layer. Since the response is not observed in the IMEC device, it has been attributed to the presence of the PEDOT:PSS layer. This last observation can give the evidence for a possible degradation mechanism. Thus, one (among many) possible degradation mechanism that encompass all the above mentioned processes and degradation compounds can be the following: First the Ag metal migrates into the PEDOT:PSS layer. Then the interaction of part of these Ag atoms with the S-atom of PEDOT:PSS can result in the formation of a silver sulfide -type of compound.<sup>6-7</sup> The presence of the Al electrode in direct contact with the silver sulfide-type of compound (and moisture) creates a difference in electronegativity between both materials (a redox pair). The latter provokes a spontaneous reaction where silver sulfide is reduced to Ag and the Al metal electrode is oxidized to Al<sub>2</sub>O<sub>3</sub>. In air, the remaining Ag atoms can easily oxidize to silver oxide:



The later proposed mechanism is similar to the methodology used for the cleaning of silver metal, which is usually tarnished (dark surface) due to its reaction with sulfur from the atmosphere (silver sulfide is dark in color). If this degradation mechanism proposed for the Al/Ag electrode in the NREL is correct, it can explain the presence of Ag, Al<sub>2</sub>O<sub>3</sub> and AgO<sub>x</sub> observed in all the characterization methods reported on our published work.<sup>2-5</sup> Thus we conclude that, in the case of an inverted organic solar cell applying the PEDOT:PSS as hole extracting layer like in the NREL device, the principal cause of failure could come from the electrode degradation and not from the active materials.

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