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Improving stability of organic devices: a time/space resolved structural monitoring approach applied to plasmonic photovoltaics



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ABSTRACT

An unconventional approach is applied for the first time to study the effect of silver nanoparticles incorporation in plasmonic organic photovoltaic devices. The incorporation of silver nanoparticles in the photoactive film results in enhanced PV performance and stability with respect to the reference device. The role of the local morphology in improving the plasmonic device properties is addressed by time-resolved high spatial resolution X-ray diffraction investigations. TR-HRXD was performed in-situ on an integrated OPV device incorporating silver nanoparticles in the photoactive layer during annealing (simulating the working conditions). Such time-space resolved method allowed tracking the modifications of the structural properties at each layer and interfaces. Remarkably, it is demonstrated that it is possible to track the variation of the plasmonic spatial distribution inside the device over time, a factor strongly influencing the photovoltaic performance.

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1. Introduction

In photovoltaics, performance and stability improvements are recognized as fundamental aspects in the search of novel active materials and cell architectures for next generation devices [1,2] and in recent literature improvements were obtained in particular for dye-sensitized solar cell devices [3]. The study of organic photovoltaic (OPV) devices, a promising flexible and low cost alternative to silicon-based photovoltaic devices, represents an exciting research field [4–6]. Although the power conversion efficiency (PCE) is now over 10% [7–10], the full potential of these systems is still unexploited.

One crucial point is that the restriction imposed on the organic active layer thickness limits the absorbance of OPV devices. A challenging approach for light harvesting is the exploitation of the capability of incorporating metallic nanomaterials of various types and configurations to improve optical absorption [11–16]. Actually, the possibility to incorporate plasmonic silver (Ag) nanoparticles (NPs) in different places throughout the OPV device structure was addressed [17] and enhanced optical and electrical properties

were obtained incorporating gold NPs at the bulk-heterojunction (BHJ) buried interface with the anode buffer layer [18].

Enhanced optical absorption is obtained via metal NPs doping of the active layers of OPV cells, due to a dual beneficial effect: the localized surface plasmon resonance interaction (LSPR) of the smaller size NPs surface electrons and the light field [19] along with multiple scattering by the larger size NPs [20]. Finally, it was recently demonstrated that BHJs doped with NPs show enhanced structural stability [21] under illumination, a central aspect for the development of durable OPVs.

Still, in order to take full advantage of the enormous potential of plasmonic OPVs a vital aspect is the understanding of the relations between the local properties (structure/morphology of the active layers and the NP size and distribution inside the organic layers) and the output of large area devices.

A key role in this contest is played by the development of in-situ diagnostics enabling to monitor structural and interface characteristics at the nanoscale; a far from being easy task.

In this work, a vertical stratigraphy of the integrated OPV device, from one electrode to the other, by means of a nanometer size synchrotron radiation X-ray probe, was performed which allows resolving the contribution of each of the constituent layers. By acquiring a sequence of patterns during in-situ annealing in the

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experimental operating conditions (a temperature of about 70 °C) of the OPV device, the time-dependence of the structural and interface properties of each layer was determined.

2. Experimental

2.1. Materials and devices

2.1.1. Ag NPs generation

The generation of NPs was achieved by ultrafast laser ablation of metallic targets (Ag/99.98%). This technique provides the possibility of producing a large variety of NPs that are free from both surface-active substances and counter-ions [22]. The targets were placed into a Pyrex cell and covered by a layer of absolute ethanol. A femtosecond (pulse duration ≈ 100 fs and repetition rate ≈ 1 kHz) laser beam was focused onto the target through the ethanol layer. The cell was mounted on a computer-driven XY stage and translated during laser exposure. More experimental details can be found elsewhere [23]. Laser irradiation gives rise to a high temperature gradient in the metal bulk and to melting of a thin layer of the target. A fraction of the molten layer is dispersed into the liquid as NPs. The NPs average size was 20 nm with quite a broad size distribution (see TEM analysis in Fig. S1 in the Supporting Information).

2.1.2. OPV device fabrication and parameters

Regioregular P3HT and PCBM were purchased from Rieke Metals and Nano-C, respectively. They were dissolved in dichlorobenzene (*o*-DCB) in a 1:1 ratio and stirred overnight at 75 °C. Then, the metallic NPs were blended into the P3HT:PCBM solution. All photoactive layers were subsequently deposited by spin-coating the blend solutions at 1000 rpm for 120 s. The BHJ average thickness was 230 nm, as determined by Energy-Dispersive X-ray Reflectivity (EDXR) measurements (see Fig. S2 in the Supporting Information). The OPV devices reported were fabricated on 20 mm \times 15 mm indium tin oxide (ITO) glass substrates with a sheet resistance of $\sim 20 \Omega/\text{sq}^{-1}$. As a buffer layer, poly(ethylene-dioxythiophene) doped with poly(4-styrenesulfonate) (PEDOT:PSS), purchased from Heraeus, was spin-cast from an aqueous solution on the ITO substrate, the average thickness the layer was 40 nm, followed by baking for 15 min at 120 °C in ambient

conditions. For PCE experiments complete PV cells were obtained by thermally evaporating aluminum cathodes through a shadow mask. For the annealing process the films were thermally treated at 75 °C for 15 min followed by a post fabrication annealing for another 5 min at 160 °C in nitrogen atmosphere. The current density–voltage (*I*–*V*) measurements were performed in all devices inside a nitrogen-filled glove box using an Agilent B1500A Semiconductor Device Analyzer. For PV characterization the devices were illuminated by 100 mW cm^{-2} power density white light using an Oriel solar simulator (active area = 4 mm²) with an AM1.5 filter through the glass/ITO side. The stability tests were performed under inert atmosphere at 70 °C. Incident photon-to-current efficiency (IPCE) curves were recorded for the reference device and devices with the optimum blend ratio, using an integrated external quantum efficiency system (ENLI Tech.). The light intensity was calibrated using a Si photodetector.

2.2. TR-HRXD setup

Time-resolved high spatial resolution X-ray diffraction (TR-HRXD) experiments were performed on the ID11 beamline at the ESRF, Grenoble, France. An X-ray energy of 35 keV was selected using the Laue-Laue monochromator of the beamline and planar silicon lenses were used to produce a vertical line focus [24] which was aligned parallel to the center of the X-ray beam by a translation and tilt stage. The X-ray line width was defined by slits placed upstream of the lenses. A 1 mm pinhole was placed between the lenses and the sample to remove the background scattering from the optical elements.

The measurements were carried out using a fiber optic coupled CCD area detector (50 μm pixel size [25]) and a high-resolution Sensicam X-ray camera (1.3 μm pixel size) was used for alignment purposes. A four quadrant diode was used to correctly place the sample in the focal depth and a fluorescence detector was employed to track the various elements composing the device for the spectromicrographic analysis. The fluorescence signal from the indium in the ITO thin film was utilized to verify the X-ray probe characteristics. The details of setup, sample preparation positioning and alignment procedures are described elsewhere [26].

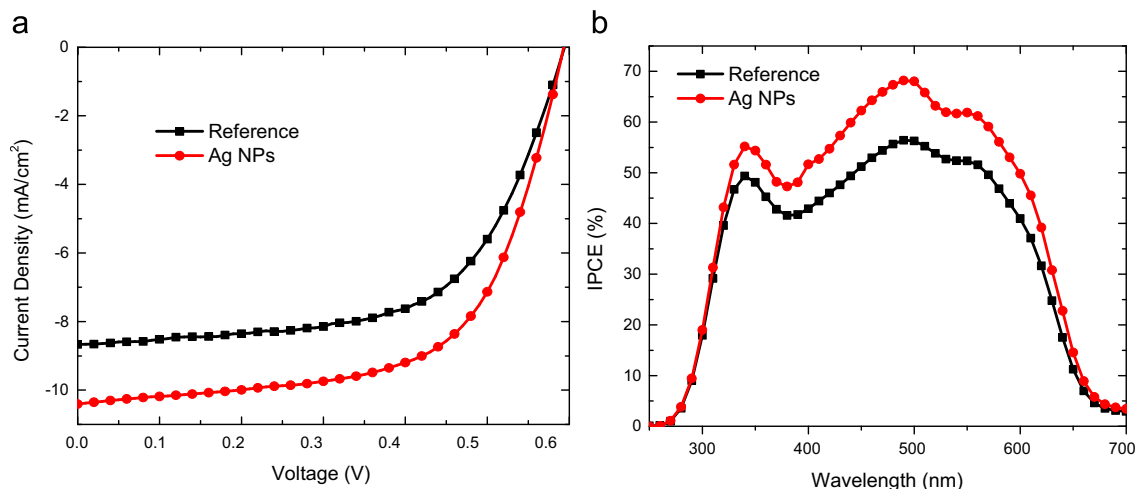


Fig. 1. (a) Current–voltage characteristics of the PV output of the reference P3HT:PCBM (black curve) and P3HT:PCBM-AgNP (red curve) devices. (b) IPCE spectra of the reference (black curve) device and of the device incorporating Ag NPs (red curve). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

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