Fully-sprayed flexible polymer solar cells with a cellulose-graphene electrode

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ABSTRACT

Organic photovoltaic (OPV) technology provides energy where conventional photovoltaics are difficult to implement. The rise of efficiency due to the introduction of new polymers and the definition of strategies for the scale-up push OPV devices towards large-scale manufacturing. Here, spray coating has been employed as an easy and versatile scalable technique to deposit all the layers of flexible polymer solar cells starting from PET/ITO/Ag/ITO substrates. A foldable nanocomposite based on cellulose and sprayed graphene nanoplatelets has been applied as top electrode through lamination. The overall fabrication process has been conducted in air by using commercial materials. A significant power conversion efficiency higher than 3% has been achieved and the high quality of the lamination process has been demonstrated by bending and adhesion tests. Such photovoltaic devices are the first fully-sprayed prototypes on plastic substrate and the novel structure has also been effective for devices with active area up to 0.75 cm².

1. Introduction

Organic photovoltaics (OPV) have emerged as the technology able to extend the area of application of conventional photovoltaics, based on inorganic thin films and crystalline silicon, as a result of the versatility in terms of semitransparency, color rendering properties and shape [1–3]. Polymer solar cells (PSCs) have achieved power conversion efficiencies (PCEs) of about 12% in laboratory devices [4,5] and reliable and cost-effective strategies have been developed to scale-up the technology [6–8]. Flexible PSCs have attracted even more interest in light of the appealing mechanical properties and high power-to-weight ratio that render them an attractive solution for the ubiquitous energy demand (e.g. solar-powered consumer electronics). Several printing and coating techniques have been investigated in literature to fabricate flexible organic photovoltaic cells and modules, also in roll-to-roll configuration [9–12]. Spray coating is a versatile technique suitable for depositing different layers on virtually any kind of substrate with a remarkable conformability, difficult to achieve with other techniques [13,14]. Moreover, spray painting is realized with low cost instrumentation and easy processing and it is heavily exploited in the industry (e.g. automotive) since it guarantees large-scale production. This process has already been successfully applied to fabricate OPV with polymer:fullerene [15–17] and all-polymer [18,19] photoactive systems on glass substrate but it needs a decisive development on flexible substrate.

Besides the choice of the proper deposition technique, a critical aspect in OPV is the realization of a solution-processed top electrode. The classical structure of evaporated molybdenum oxide and silver for inverted devices is not scalable and suffers mechanical stability under thermal aging [20]. Several approaches and materials have been investigated comprising the employment of conductive inks composed of PEDOT:PSS, silver nanowires or carbon nanotubes, and of chemical vapor deposited graphene, directly
deposited on the top of the samples or laminated [21–25]. In particular, lamination is the most promising way to produce devices in large volumes with high throughput allowing the simultaneous encapsulation [26,27]. Despite these advantages, drawbacks could rise related to the durability of the solar cell under mechanical stresses (i.e. under repeated bending cycles) and in terms of adhesion of the laminated conductive materials on the cells. Systematic quantitative studies, which correlate the laminated top electrodes adhesion on the cell with bending stability and durability, are not present in literature.

Cellulose is emerging as an abundant, renewable and flexible material to be applied in photovoltaics [12,28–30]. Few/multi layers of graphene nanoplatelets (GnPs) are suitable candidates to provide conductivity to the cellulose in order to act as a flexible electrode, considering the electrical conduction and flexibility of GnPs coupled with their moderate price and large availability [31–34]. In this work, a spray manufactured foldable nanocomposite based on cellulose impregnated with biopolymer-GnPs ink is laminated on top of polymer solar cells fabricated by spraying all the layers in air starting from PET/ITO/Ag/ITO foils. Such production method enables to achieve a PCE of over 3%.

An estimation of the adhesion force of the laminated top electrodes on the solar cell is performed, justifying the durability results obtained with the bending tests. The PSCs presented in this work are the first fully-sprayed photovoltaic devices on plastic substrate and demonstrate the successful implementation of spray coating in OPV.

2. Methods

2.1. Realization of conductive cellulose top electrode

GnPs with average thickness of ≈9 layers (see the Raman spectra in Fig. S1) and lateral size in the range of hundreds of nanometers [35,36] were donated by Directa Plus (grade Ultra G-1). Such GnPs were solvent processed creating a conductive ink in chloroform (Sigma-Aldrich) coupled with Mater-Bi biodegradable thermoplastic (license number 14) obtained from Novamont S.p.A. For each 30 ml of solvent, 0.35 g of both bioplastic and GnPs were employed. To ensure a sufficient dispersion, a probe sonication process (750 W, 20 kHz, 40% amplitude) of 1.5 min was performed using a Sonics & Materials, Inc. (Model Num. VCX750). The spray coating (Paasche air brusher, 0.73 nozzle, 2.2 bar, 18 cm) was performed on both sides of a cellulose substrate (30 μm thick) acquired from Korff (Art. Num. 60,285) of approximately (5 × 5) cm² using a Paasche air brusher. Such nanocomposite was finally hot-pressed (≈60 bar, 30 min, 175 °C) using a Specac-Atlas Power Presses T8 in order to impregnate the conductive ink into the fiber network and obtain isotropic electrical conduction [32]. The Mater-bi was acting as binder between the cellulose and the GnPs. Teflon anti-attachment films (Advent Research Material, Art. Num. FP823338) were employed during the hot-pressing process to prevent sticking of the material on the hot-press plate. A comprehensive study on the electrical, mechanical and chemical properties of the cellulose-GnPs nanocomposite was reported in Ref. [32].

2.2. Preparation of inks

Zinc oxide (ZnO) nanoparticles dispersion was purchased by Sigma Aldrich and diluted 1:100 in ethanol. The ZnO particle size is <130 nm. Polyethyleneimine, 80% ethoxylated solution (PEIE) from Sigma Aldrich was dissolved in ethanol to get a concentration of 0.1 wt% [37]. The active layer comprised poly[5,6-difluoro-2,1,3-benzothiadiazol-4,7-diyli-alt-(3,3′-di(2-octyldodecyl)2,2′;5′,2′;5′,2′‴-quaterthiophen-5,5‴-diyli)] (PFBT4T-2OD) and PC70BM, both purchased by Cal-Os Inc. They were dissolved in ortho-xylene in the ratio 1:1.2 to obtain a total concentration of 17.6 mg/ml. A small amount (3% v/v) of 1,8-diiodooctane (DIO) from Sigma Aldrich was added to the solution, which was stirred for 5 h at 110 °C. Vanadium (V) oxytriisopropoxide from Sigma Aldrich was used as precursor for vanadium oxide (V₂O₅): the sol-gel was prepared by dissolving 0.02 ml of precursor in 3 ml of 2-propanol and by stirring it for 2 h. PEDOT:PSS formulation was composed of Clevios™ HFC Solar filtered through a 0.45 μm pore-size PVDF filter and water in the ratio 1:1. A concentration of 3.75% of D-sorbitol was added to obtain the typical doping and adhesive effect [38,39].

2.3. Solar cell fabrication and electrical testing

Polyethylene terephthalate (PET) foils coated with a transparent conductive ITO/Ag/ITO multilayer as dielectric/metallodielectric (DMD) scheme (Solutilia, -8 & 1/2) were patterned by wet etching in hydrobromic acid at room temperature and then cleaned by ultrasonic bath with detergent, water and isopropanol (10 min each step). The spray deposition of the inks was performed under chemical hood by using a dual action commercial airbrush (Mecafer) supplied by compressed air with pressure set at 2 bar for all the layers [40]. The ambient conditions during the process were temperature of 18 °C, fluorescent light of 6500 K and 3250 L m and humidity value of 28%. Firstly, ZnO and PEIE were deposited at substrate-nozzle distance = 10 cm, flow rate = 4 ml/min, spraying time = 3 and 2 s, respectively. The active layer formulation was sprayed at substrate-nozzle distance = 5 cm, flow rate = 1 ml/min, ink volume = 400 μl V₂O₅ was sprayed at substrate-nozzle distance = 10 cm, flow rate = 2 ml/min, spraying time = 3 s. PEDOT:PSS formulation was sprayed at substrate-nozzle distance = 15 cm, flow rate = 4 ml/min, ink volume = 2 ml. It should be noted that for very thin layers (ZnO, PEIE and V₂O₅) the parameter that controls the thickness is the spraying time (in s) whereas for thick layers (active layer and PEDOT:PSS) such parameter is the ink volume (in ml) loaded in the reservoir of the airbrush. The nanocomposite was applied to the deposited stack through lamination at 110 °C and 0.3 bar by using a flat-bed heat press machine.

A custom-made experimental set-up composed of a source-meter (Keithley 2612A) and a monochromator (Newport 74,000) was used to acquire the External Quantum Efficiency (EQE) of the photovoltaic cells. The spectrum of the reference lamp was measured by a calibrated photodiode. Current density–voltage (J–V) curves of the devices were evaluated both in dark condition and under light set at AM1.5 G and 100 mW cm⁻² by using a class A solar simulator (ABET Technologies, Sun2000). Before the measurement, the irradiation was calibrated by means of a pyrometer (Eko, MS-602). The devices under light testing were covered with a shadow mask to selectively irradiate the active area of the cell. The devices were measured without any encapsulation.

2.4. Characterization

Raman spectra of the carbon based nanofillers were measured with a Horiba HR800UV, LabRam 600 spectrometer (excitation wavelength of 632.8 nm HeNe laser, diffraction grating of 600 lines per mm, maximum power 20 mW). A 90° Polarization test was performed on devices deposited on a glass substrate, in order to evaluate the adhesion strength of the graphene electrode. The sample was held horizontally by the bottom sleg, one end was gently lifted and mounted in the upper clamp. The clamp was displaced upward while the sleg, connected through a pulley to the upper clamp, followed with a perpendicular movement, so that the pulling angle was constant. A scheme of the setup is reported in Fig. 1. The displacement rate was 3 mm/min, the sample width
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