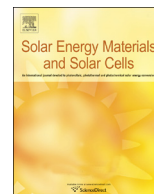




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Annealing-free solution-processed tungsten oxide for inverted organic solar cells



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ABSTRACT

Solution processing is mandatory to continue the development of large scale and low cost organic solar cells. Tungsten oxide (WO₃) has shown good performance when used as a hole transporting layer in organic solar cells. Here this oxide has been deposited by a sol-gel technique on top of the active layer in an inverted structure device. This layer has been characterised by XPS and UPS to understand the effect of the stoichiometry and of the energy levels on the device performance. In the optimised structure, an average power conversion efficiency (PCE) of 3.5% has been achieved, which is comparable to devices with vacuum deposited transition metal oxide using poly(3-hexylthiophene):[6,6]-phenyl-C₆₁ butyric acid methyl ester (P3HT:PCBM) as an active layer. Moreover in our study no annealing is needed after the deposition of the sol-gel processed oxide, which is really promising for the development of industrial roll-to-roll production on flexible substrates.

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1. Introduction

Organic solar cells have been widely studied since photoconductivity has been observed from anthracene at the beginning of the 19th century [1] and the first heterojunction polymer solar cell in [1,2]. However, research efforts are required to optimize the structure of the cells, using new active and interlayer materials to enable cost-effective, large area organic solar modules commercialization. The inverted structure is promising due to the extended stability in air [3]. However, finding the appropriate hole transporting layer is a challenge. Transition metal oxides (TMO) are good candidates to replace poly(3,4-ethylenedioxythiophene) doped with poly(styrene sulfonate) (PEDOT:PSS) which has already shown its limits in terms of stability. Due to its hygroscopic and acidic nature, PEDOT:PSS is responsible for degradations at the interface with the active layer [4–6]. Indeed TMOs exhibit energy levels matching the active layer and electrodes, in order to enhance the collection of holes and to efficiently block electrons. Most of studies deal with the use of molybdenum oxide (MoO₃) [7], nickel oxide (NiO) [8], vanadium oxide (V₂O₅) [9], but only a few papers are reporting integration of tungsten oxide (WO₃) [10,11] as a

hole transporting layer in organic solar cells, although this TMO offers the great advantage not to be toxic for both human and environment (Table 1), which is a key factor for future large scale industrialization.

Nowadays, the solution process deposition of materials is a way to be considered in order to reduce large area production cost, to lead organic electronics to industrialization using printing processes. Transition metal oxide nanoparticles present the advantage to be soluble in ethanol or isopropanol, which are orthogonal solvents to that of the active layer (dichlorobenzene). Moreover, solution deposition of tungsten oxide has been widely studied in the field of electrochromism [12], and recently efficient solution deposition of this oxide using dispersed nanoparticles has been shown [13,14]. Our work is based on the development of a new method to deposit WO₃ from solution, through the use of a sol-gel precursor of WO₃ in inverted solar cells. The deposition using sol-gel ink is a way to be considerate since precursors are significantly less expensive than nanoparticles. Moreover effects of nanoparticles on the human body are still not well known. This solution processed tungsten oxide does not require any annealing process, which represents a great advantage for further industrial production such as roll-to-roll techniques.

2. Materials and method

Solar cells with an inverted structure (Fig. 1) were prepared with the following procedure. ITO coated glass substrates have

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Table 1
Hazard statement of transition metal oxides used in organic solar cells (MSDS data from Sigma-Aldrich).

Hazard statement		MoO ₃	WO ₃	NiO	Cu ₂ O	V ₂ O ₅
H302	Harmful if swallowed		X		X	X
H315	Causes skin irritation		X			
H317	May cause an allergic skin reaction			X		
H319	Causes serious eye irritation	X	X			
H332	Harmful if inhaled					X
H335	May cause respiratory irritation	X	X			X
H341	Suspected of causing genetic defects					X
H350i	May cause cancer by inhalation			X		
H351	Suspected of causing cancer	X				
H361d	Suspected of damaging the unborn child					X
H372	Causes damage to organs through prolonged or repeated exposure			X		X
H410	Very toxic to aquatic life with long lasting effects				X	
H413	May cause long lasting harmful effects to aquatic life			X		
H441	Toxic to aquatic life with long lasting effects					X

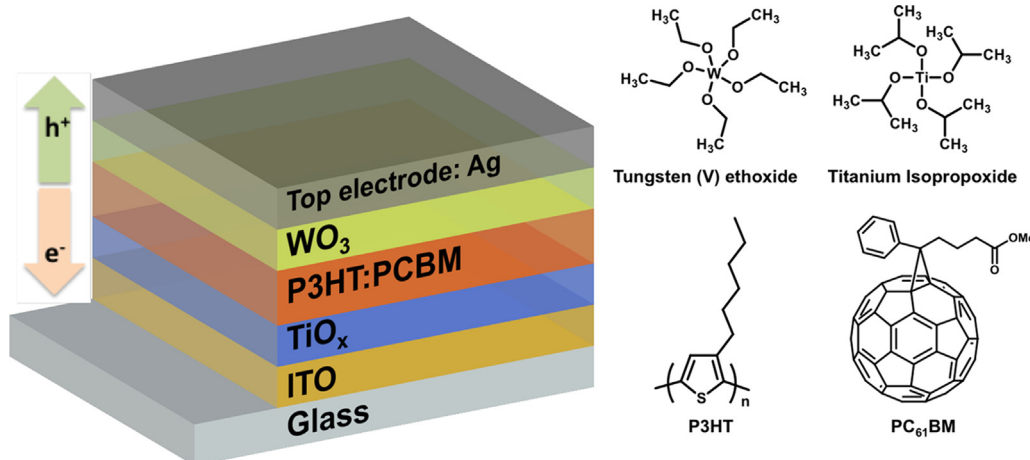


Fig. 1. Structure of an inverted solar cell using tungsten oxide as a hole-transporting layer. Chemicals used in this solar cell. Sol-gel precursors for TiO_x and WO₃.

been cleaned using an ultrasonic bath in successive solutions of acetone, ethanol and isopropanol. Then a UV-ozone plasma treatment was performed to increase the hydrophilic nature of the surface and to remove traces of organic compounds. Then a sol-gel solution of titanium oxide (TiO_x) was prepared using a recipe previously described in 2012 by our group [15]. The resulting layer is 20 nm-thick. The active layer consists of the well-known donor-acceptor system poly(3-hexylthiophene):[6,6]-phenyl-C₆₁ butyric acid methyl ester (P3HT:PCBM) [16]. The solution, composed of 20 mg/mL of P3HT (Plextronic) and 20 mg/mL of PC₆₀BM (Solaris) in 1,2-dichlorobenzene (Aldrich, purity: 99%, anhydrous), was spin-coated in a nitrogen filled glove box to achieve a 200 nm-thick active layer. The resulting layers were left to self-organize in covered petri dishes using the solvent annealing process until complete drying. Tungsten oxide was deposited either under vacuum using an electron-beam evaporation chamber (granules from Neyco, purity: 99.99%) or by spin-coating from a sol-gel solution of tungsten oxide. This solution was prepared by mixing tungsten (V) ethoxide 1.48 vol% (Alfa Aesar), acetic acid 0.1 vol% (Aldrich, purity: 99.8%), in isopropyl alcohol 98.42 vol% (Aldrich, purity: 99.5%, anhydrous). 60 to 80 nm-thick silver electrodes were successively evaporated under secondary vacuum (10⁻⁶ mbar) on the oxide layer through a shadow mask to define a 8.6 mm² active area.

Experiments were repeated on eight individual cells to evaluate error bars. Devices were characterized under a *K.H.S. SolarCelltest-575* solar simulator with AM1.5G filters set at 100 mW/cm² with a

calibrated radiometer (IL 1400BL). Labview controlled *Keithley 2400* SMU enabled to measure current density-voltage (*J-V*) curves. After the TiO_x deposition deposited in air, further fabrication steps and characterizations were conducted under inert atmosphere in a set of gloveboxes (O₂ and H₂O < 1 ppm). In the case of the sol-gel deposited WO₃, the active layer was exposed to ambient atmosphere during the deposition of this interlayer. Thicknesses were measured using an AlphaStep IQ profilometer. Conductivity of e-WO₃ and s-WO₃ has been measured using Al/WO₃/Al sandwich structure. For e-beam deposited WO₃, the conductivity is (1.40 ± 0.04) × 10⁻⁵ S/m. Which is in the same order of magnitude to the one of sol-gel WO₃, with a value of 0.83 ± 0.19 × 10⁻⁵ S/m.

For the X-ray photoelectron spectroscopy (XPS) measurements an unmonochromatized Mg K α line at 1253.6 eV (15 keV with 20 mA anode current) and an analyzer (Leybold EA-11) pass energy of 100 eV, giving a full width at half maximum, FWHM, of 1.3 eV for the Au 4f 7/2 peak, were used. The analyzed area was approximately 4 × 7 mm² rectangle positioned near the geometric center of each sample. XPS analysis was carried out at 0 degrees take-off angle (normal to the sample surface). In all XP spectra the binding energy (BE) of the predominant aliphatic contribution to the C 1s peak at 284.8 eV, was used as a measured BE reference. For the Ultra Violet Photoelectron Spectroscopy (UPS) measurements a He I (21.2 eV) excitation line was used and a voltage of 12.23 eV was applied to the specimen in order to separate the high binding energy cut-off from the analyzer.

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