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Low glass transition temperature hole transport material in enhanced-performance solid-state dye-sensitized solar cell

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Prof. Gregory L. Baker passed away on Oct. 18. 2012. We dedicate this work as a memorial to him.

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1. Introduction

Solid-state dye sensitized solar cells (ss-DSSC) suffer from incomplete pore-filling of the hole transport materials into the porous TiO₂, which leads to poor dye-regeneration and inhibits cell performance. Using spiro-MeOTAD (2,2',7,7'-tetrakis-(N,N-di-p-methoxyphenylamine) 9,9'-spirobifluorene) as the hole transport material (HTM) have achieved a cell efficiency of over 10% in ss-DSSC [1,2]. Compared to I^-/I_3^- liquid electrolytes, a solid state HTM may avoid the issues of solvent leakage and evaporation. The solid-state cells have the potential for greater stability and longer cell lifetime, making them more suitable for buildings and other outdoor applications. However, ss-DSSCs are currently limited by poorer photocurrent due to the higher internal resistance of the

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ABSTRACT

A new triarylamine-based hole transport materials (HTM) with branched side chain giving low glass transition temperature (T_g) is synthesized and incorporated into a solid state dye sensitized solar cell. This designing of molecular structure of HTM for lowering the T_g along with viscosity and surface tension of the casting solution effectively increases the porefilling fraction (PFF) as the cell is heated during the fabrication, leading to an 8 fold increasing in cell efficiency over cells without heat treatment. We relate the cell performance improvement not only due to the PFF of TiO₂ by the (HTM), but also because of morphological and thickness changes in the hole transport material (HTM) capping layer. © 2014 Elsevier B.V. All rights reserved.

hole-conducting polymer as measured against the ionic conductivity of the liquid cells. Alternatives to spiro-MeO-TAD, such as polymers [3–5] or oligomers [6–8] with higher hole mobility, have been applied to ss-DSSC, but their performance does not rival that of spiro-MeOTAD in ss-DSSC because the larger size of these polymer or oligomer structures inhibits their penetration into mesoporous TiO₂. Indeed not only charge mobility but also interfacial contact resistance will limit performance of ss-DSSC. When only limited amounts of HTM penetrate into the mesoporous TiO₂ there are gaps between the dye-coated TiO₂ and the conductive polymer that result in poor contact between dye molecules and spiro-MeOTAD, inhibiting dye regeneration [9,10].

Grätzel [6,9] and his coworkers were the first to study the pore-filling of spiro-MeOTAD relating to the power conversion efficiency of ss-DSSC. They suggested that both the device performance and conductivity went down with a thicker film device [9,10]. Balancing light absorption against spiro-MeOTAD penetration, they obtained

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maximum cell efficiency with a device having $2-3 \,\mu$ m thickness of TiO₂ which had been treated with an amphiphilic dye that made the spiro-MeOTAD 'wet' the TiO₂ pore [11–13] Subsequently, McGehee's group [14–16] extensively studied and quantitatively determined the pore-filling fraction (PFF) in mesoporous TiO₂ films with spiro-MeOTAD. They showed that as the PFF of spiro-MeOTAD increased from 26% to 65%, the cell efficiency increased by nearly 300%. Very recently, Snaith's group studied the PFF in ss-DSSC via optical reflectometry, which did not require any assumptions on material properties, and re-estimated the PFF of ss-DSSC at about 80% [17].

One approach of increasing the PFF is to change the surface properties of the TiO₂ pores so that the spiro-MeOTAD will easily pass through the 'wet' surface. Using an amphiphilic dye such as Z907 dye [12] or C220 dye [18], enhances the hydrophobility of the TiO₂ surface, and improves the infiltration of the spiro-MeOTAD because of the strong hydrophobic-hydrophobic interaction between the chlorobenzene solution of spiro-MeOTAD and longer alky chain from the dye. Another approach to optimize the pore filling is to tune the deposition parameters of spiro-MeOTAD, such as the spin-coating rate, the concentration of spiro-MeOTAD [14] during the spin-coating process, or deposition techniques by doctor-blading instead of spin-coating [15]. Later, Sellinger et al. designed new organic HTMs (AS 37 and AS 44) with low melting point (T_m) , low glass transition temperature (T_g) and a 2 fold higher solubility which allows facile interpenetration into the TiO₂ pore structure [19]. The AS 37 and AS 44 based ss-DSSC reached efficiencies of 3%, rivaling that of conventional spiro-MeOTAD based cells.

In contrast to these previous methods, here we present a simple heat-assisting method to induce our low T_g HTM $(T_g = 20 \text{ °C})$ into the TiO₂ pores. A remarkable increase in cell performance is obtained especially the photocurrent after the heat treatment. The improved performance is attributed to more efficient dye regeneration. This simple strategy with a unique HTM reduces the surface tension of the HTM in the TiO₂ pores by elevating the temperature of both the HTM solution and deposition TiO₂ substrate. We calculate the PFF and conduct an SEM analysis that reveals a uniform HTM thin layer coating the TiO₂. This thin layer serves as a blocking layer, preventing shortcircuits between the counter electrode and TiO₂, which can lead to charge recombination. This observation is also consistent with the observed increase in open circuit voltage in the ss-DSSC after heat treatment as well.

2. Experimental

2.1. Materials

Fluorine doped Tin Oxide (FTO) Glass purchased from Hartford was pretreated by Alconox, distilled water, acetone and UV–ozone before use. Low T_g HTM was synthesized through a previous literature [19]. TiO₂ paste (Ti-Nanoxide T) was from Solaronix. Gold used as evaporation source for counter electrode was from Kurt J. Lesker. All other chemicals were purchased from Sigma Aldrich and were used as receive unless mentioned otherwise.

2.2. Instruments

The capping layer on TiO₂ was imaged by a Hitachi S-4700 II field-emission scanning electron microscope (FESEM). The cross-sectional structure of solar cell was determined by a Carl Zeiss Auriga Dual Column FIB SEM. The morphology of capping layer was obtained using a Veeco Dimension 3100 scanning probe microscope. Contact angles were measured using a VCA 2000 Video Contact Angle Surface Analysis system. Absorption spectra were obtained from Varian Carey Model 50 UV–Vis Spectrophotometer. Current–voltage measurements and open circuit voltage decay measurements of ss-DSSC were performed using a Xe Arc Lamp and CHI-650D Electrochemical work station. An AM 1.5 solar filter and neutral density filter were used to simulate sunlight at 100 mW cm⁻². A mask with an area of 0.25 cm² was used.

2.3. Solar cell fabrication and preparation

Devices of ss-DSSC were fabricated according to a published procedure [9,20]. Briefly, a pre-cleaned FTO glass was put on a hot plate with a temperature of 500 °C for 30 min before any deposition. A 200 nm thickness of TiO₂ compact layer on FTO was formed by home-made spraypyrolysis set-up using air as the carrier gas and titanium di-isopropoxidebis (acetylacetonate) as the precursor. A 2.5 µm thickness of mesoporous TiO₂ film was deposited by doctor-blading TiO_2 pastes with scotch tape as a spacer. The thickness was measured by profilometer and confirmed by a cross-section SEM image. After baking the TiO₂ paste with a programmable heating procedure up to 450 °C, we let the substrate cool to 80 °C before absorbing it into a 0.3 mM Z907 dye solution for a minimum of 16 hours, which was followed by rinsing with ethanol and acetonitrile. A heated chlorobenzene solution of HTM was used for spin-coating on heated dye-coated TiO₂ substrates. The concentration of hole conductors was 9 wt% or 15 wt% in total with additives of lithium bis(trifluoromethylsulfonyl)imide (LiTFSI) and 4-tert butylpyridine (4-tBP). A 36 µL electrolyte solution was first applied to the substrate for one minute, and then was spin-coated with 2000 rpm for 30 s in air. The cell was stored under dark overnight before depositing the gold electrode. A 30 nm thickness of gold was applied by thermal evaporation as back contact.

3. Results and discussions

A new type of hole transport material is developed and synthesized by tethering 2-ethyl hexyl chains onto fluorine, leading to a low T_g of 20 °C (Scheme 1 and a NMR spectrum and DSC curve shown in Figs. S5 and S6). A longer chain allows a more flexible structure at temperature over T_g [19,21]. A further reason for introducing a branched chain such as the 2-ethyl hexyl group is to minimize intermolecular hydrophobic–hydrophobic interaction, which may cause voids generating a large bulk resistance in solar cell [22]. A UV–vis spectrum of new synthesized HTM is also provided in the supporting

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