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Morphological, optical and electrical characterization of titania-nanotubes-based dye-sensitized solar cells

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Abstract

Self-ordered, vertically aligned, titanium oxide (TiO_2) nanotubes were grown on an electropolished titanium foil by anodic oxidation in a viscous organic electrolyte. We chose two different anodization times to obtain nanotubes of approximately 9 and 17 μm in length. Some of the TiO_2 nanotubes were post-treated with $TiCl_4$ or hydrothermally with TiO_2 P25. Their structure and morphology were investigated with field-emission scanning electron microscopy (FSEM) and transmission electron microscopy (TEM). To improve the sensitizing of the high-aspect-ratio nanotubes with dye we used a new approach in which the TiO_2 arrays were immersed into the dye solution at elevated temperature in an autoclave. The current–voltage characteristics of the assembled DSSCs gave an average energy-conversion efficiency of 5.7% for the untreated nanotubes.
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1. Introduction

High-efficiency, dye-sensitized, solar cells (DSSCs) were first reported in 1991 by Grätzel and O'Reagan. Since then they have received considerable attention because of their low price and relatively easy manufacturing process. The efficiency so far achieved of 12.3% was reported by Yella et al. (2011), who used a Co-based redox electrolyte in conjunction with zinc poriphirin dye as the sensitizer. A DSSC consists of a working electrode, an electrolytic solution, and a counter electrode. One of the most investigated parts of the DSSC is the working electrode, where the active part is a wide-band-gap semiconductor oxide, e.g., TiO_2, ZnO (Rensmo et al., 1997; Jana et al., 2014), SnO_2 (Ferrere et al., 1997), or Nb_2O_5 (Sayama et al., 1998). Among those materials, the most frequently used is TiO_2. Depending on the cell's construction TiO_2 can be used in various morphological forms, such as nanoparticles (O'Reagan and Grätzel, 1991), nanotubes (Macak et al., 2007; Paulose et al., 2006), nanorods (Park et al., 2013), nanowires (Adachi et al., 2004; Boercker et al., 2008), nanoleaves (Dhas et al., 2011), a mesosponge layer (Kim et al., 2009) or as various combinations of these (Alivov and Fan, 2009; Xuan et al., 2011).

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The first reports on application of anodized TiO_2 nanotubes arrays for DSSC devices were published or presented by Macak et al (2005). It was proposed that such ordered nanotube nanostructures have the potential to improve the electron transport in comparison to porous layers composed of TiO_2 nanoparticles (Zhu et al., 2007). Krivec et al. (2013) investigated the interface between titanium substrate and the titania nanotube film grown by anodic oxidation and found out that it is very stable, what is the most important for the electron transfer from titania nanotubes to titanium sheet in DSSC solar cells. Self-organized TiO_2 nanotube arrays are usually grown by anodic oxidation. With this relatively simple process it is possible to optimize the nanotube morphology by adjusting the anodization processing conditions. The most important parameters are the anodization voltage, the time of anodization, the electrolyte composition and its temperature during the anodization. The influence of these parameters on the nanotube's length, diameter, wall thickness and other morphological characteristics has been well investigated (Mor et al., 2006b; Hwang et al., 2011).

The power-conversion efficiency (PCE) of TiO_2 nanotube-based DSSCs as well as TiO_2 nanoparticle-based DSSCs can be further improved by different surface treatments of TiO_2. Two effective treatments have been reported so far: the first is to expose the TiO_2 photoanodes to an O_2 plasma (Kim et al., 2008), while the other is to immerse them into a $TiCl_4$ solution (Nair et al., 2012; Sommeling et al., 2006). The combination of both approaches can be used as well (Wang and Lin, 2009).

DSSCs that have the nanotubes grown on a Ti foil require back-side illumination, which results in significant optical losses. Because of this there were many attempts to avoid the reflection and absorption of light by the counter electrode and electrolyte. There are two possible ways to fabricate front-side-illuminated DSSCs with TiO_2 nanotube arrays. The first one is to transfer the nanotube array to FTO-coated glass (Lei et al., 2010), whereas the second one is to anodize a titanium film sputtered onto FTO coated glass (Mor et al., 2006a).

In the present work, the photovoltaic properties of the back-side illuminated DSSC devices were studied using TiO_2 nanotube array photoanodes with different nanotube lengths. Two post treatments of nanotube arrays were applied. The first one was to treat the nanotube arrays in a $TiCl_4$ solution, while for the second post-treatment we used a not well investigated approach consisting of the hydrothermal treatment of nanotube arrays in a P25 suspension (Hu et al., 2011). The optical losses due to the absorption and reflection of the incident light on the counter electrode and the redox electrolyte were also investigated. In order to determine the DSSCs' main characteristics (energy-conversion efficiency, short-circuit current density, open-circuit voltage and fill factor) the solar cells' electrical properties were characterized by current–voltage measurements.

2. Materials and methods

Highly ordered TiO_2 nanotube arrays were grown using the anodic oxidation of 120-μm-thick 99.6% pure titanium foil. Prior to the anodization the titanium foil was electropolished with Struers A3 electrolyte. The voltage and temperature during the electropolishing were set to 35 V and 22 °C, with the polishing time set to 12 s. Thus, the obtained smooth surface of the titanium foils was ultrasonically cleaned in acetone and ethanol. The electropolished titanium foils were then subjected to potentiostatic anodization in a two-electrode electrochemical cell using a platinum foil counter electrode. The anodization voltage was kept constant at 60 V using a DC power supply (Toellner). The anodized area was set to 0.3 cm^2. All the experiments were carried out at room temperature. The electrolyte was prepared by dissolving 0.3 wt.% of ammonium fluoride (NH_4F, Kemika d.d., Zagreb, Croatia) in ethylene glycol (99.5%, Carlo Erba Reagents, Val de Reuil, France) with the addition of 2 vol.% of distilled water. The anodization time was 3 h and 5 h, respectively.

Altogether, six differently prepared nanotube arrays were produced, three of them with the same length of TiO_2 nanotubes. For each anodization time one set was left untreated. The other two sets with identical TiO_2 nanotube lengths were annealed in air for 30 min at 450 °C with heating and cooling rates of 5 °C/min. After annealing, the TiO_2 arrays were additionally treated in 0.2-M $TiCl_4$ (99.9%, Acros Organics, New Jesey, USA) at 50 °C for 30 min or in a 0.1% aqueous solution of TiO_2 P25 (Degussa). The $TiCl_4$ solution was prepared following the same procedure as described by Clabaugh et al. (1956). The treatment using the P25 solution was carried out in an autoclave at 100 °C for 1 h. The anodized titanium foil was immersed in 50 ml of P25 solution in an 85-ml Teflon vessel. Both the treated and untreated TiO_2 arrays were finally annealed in air at 450 °C for 3 h in order to obtain crystalline TiO_2 nanotubes. After cooling to approximately 70 °C the TiO_2 nanotube arrays were immersed in a 0.3-mM solution of N719 dye (Dyesol) in acetonitrile/tert-Butyl alcohol (volume ratio 1:1) in a 50-ml Teflon vessel. The vessel was held for 24 h at 70 °C in an autoclave. Thus prepared working electrodes were assembled into back-side-illuminated DSSCs. To prepare a counter electrode, a hole was drilled in the fluorine-doped tin oxide (FTO) coated glass (Solaronix) with a sheet resistance of 7 ohm/square. The counter electrodes were then prepared by the brush painting of H_2PtCl_6 (Sigma–Aldrich) onto the FTO-coated glass followed by firing at 450 °C for 15 min to form an activated platinum layer. The two electrodes were separated by a 60-μm-thick hot-melt film (Meltonix 1170-60, Solaronix) that was cut into a gasket. The solar cell was sealed by heating at 120 °C for a few seconds. Then, the commercially available electrolyte EL-HPE (Dyesol) was introduced into the cell through the hole in the counter electrode via vacuum backfilling. EL-HPE

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