



Electrophoretic deposition of crack-free magnesium oxide-coated tin oxide film and its application in dye-sensitized solar cells



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ABSTRACT

Magnesium oxide-coated tin oxide aggregates were prepared by a facile method. The aggregates consisted of nanoparticles which provided a large surface area of $74.27 \text{ m}^2 \text{ g}^{-1}$. Crack-free MgO-coated SnO_2 porous films were prepared using electrophoretic deposition method. The causes which prevent the forming of cracks were also discussed. For the application in dye-sensitized solar cells, the electrophoresis conditions, such as the amount of MgO loading and deposition duration, were optimized. The MgO-coated SnO_2 photoanodes showed excellent photovoltaic response in the long wavelength region between 550 nm and 700 nm. The optimized MgO-coated SnO_2 photoanodes showed an overall power conversion efficiency above 7.0% under AM 1.5G illumination (100 mW cm^{-2}), having a short circuit current density, open circuit voltage, and fill factor of 20.0 mA cm^{-2} , 0.618 V and 0.57, respectively.

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1. Introduction

Dye-sensitized solar cells (DSSCs) have attracted great scientific and technological interest because of their simple preparation process and comparatively high photoelectric conversion efficiency of up to 12.3% [1]. Nanocrystal photoanodes for DSSCs can be prepared by several methods, such as sputtering, electrospun deposition, doctor blading, screen printing, spray deposition and electrophoretic deposition (EPD) [2–5]. The EPD technique is widely used to prepare semiconductor nanocrystal films due to several advantages, such as high deposition rate, controllable film thickness, no requirement for binder and few restrictions of the substrate shape. Though the EPD technique shows many advantages compared with other processes, there is no binder in the obtained nanoparticle films. As a result, the films are easy to display cracks which seriously affect the properties of photoanodes.

S. Guldin et al. [6] avoided the formation of cracks in the thick films by the predeposited TiO_2 layer ($465 \pm 30 \text{ nm}$). B. Tan et al. [7] found that the presence of TiO_2 nanowires helped to preserve the crack-free films after sintering. S. Nakade et al. [8] avoid cracks in the deposited films by mixing 200 nm TiO_2 particles in the colloidal suspension. H.W. Chen and W.H. Chiu et al. [2,3] found that a large amount of cracks existed in the electrophoretically deposited nanocrystalline films. They also proposed some efficient methods, such as multiple electrophoretic deposition and post-compression

to solve this problem. W. Jarernboon et al. [9] investigated the effects of the deposition voltage and deposition duration on the morphology of cracks. It should be exciting for the improving of the photoelectric conversion efficiency of the photoanode if we can decrease, or even avoid, the cracks in the electrophoretically deposited thick films.

SnO_2 is a wide band-gap (about 3.6 eV) n-type semiconductor, and its application in DSSCs may reduce the effect of UV light in sunlight on their performance and improve their long term stability [10]. However, the open circuit voltages (V_{OC}) and fill factors (FF) were low, as a result of the low conduction band energy of SnO_2 . Coating SnO_2 with thin layers of other oxides, such as MgO, ZnO, Al_2O_3 or TiO_2 , is an efficient way of solving these problems [10–13]. After surface modification, SnO_2 photoanodes have shown high photoelectric conversion efficiency and excellent stability in TCO-glass-based DSSCs.

In this paper, we proposed a method to prepare MgO-coated SnO_2 films with no cracks using electrophoretic deposition (EPD) method. The MgO-coated SnO_2 photoanode preparation process, such as the MgO loading, deposition duration, had been optimized on fluorine-doped tin oxide (FTO) glass substrates. The DSSCs showed an optimum overall conversion efficiency above 7.0% under AM 1.5 G one sun illumination (100 mW cm^{-2}).

2. Experimental

2.1. Preparation and characterization of MgO-coated SnO_2 films

A 10 mL colloidal SnO_2 solution (Alfa Aesar, USA, 15 wt.% in H_2O , particle diameter $\approx 7 \text{ nm}$) was ground with magnesium acetate

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Table 1
The deposition conditions of the Samples.

Samples	Adopted colloid	Magnesium acetate content	Deposition time
Sample I	Colloid A	0.6 g	1 min
Sample II	Colloid B	0.6 g	1 min
Sample III	Colloid C	0.6 g	1 min
Sample IV	Colloid C	0.6 g	2 min
Sample V	Colloid C	0.6 g	3 min
Sample VI	Colloid C	0.6 g	4 min
Sample VII	Colloid C	0.6 g	5 min
Sample VIII	Colloid C	0.0 g	3 min
Sample IX	Colloid C	0.2 g	3 min
Sample X	Colloid C	0.4 g	3 min
Sample XI	Colloid C	0.8 g	3 min
Sample XII	Colloid C	1.0 g	3 min

tetrahydrate (Sinopharm Chemical Reagent Company, China) in an agate mortar. The amount of magnesium acetate tetrahydrate ($\text{Mg}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$) additive was changed from 0.2 to 0.4, 0.6, 0.8 and 1.0 g. The mixture was centrifuged at 16000 RPM for 5 minutes. The sediment was resuspended in 100 mL ethanol and magnetically stirred for 1 h, followed by centrifugation and dispersion in 100 mL acetone. The suspension was centrifuged again. Three kinds of colloids were prepared as follows: (i) The sediment (1.0 g) obtained from the suspension was directly added to a mixture of 8 mL butanol, 4 mL isopropanol, and 2 mL ethanol, followed by magnetically stirring for 30 min and ultrasonically agitating for 10 min to form colloid solution (Colloid A); (ii) The sediment was dried in a vacuum oven at 40 °C for 10 h. The resulting powder was ground in an agate mortar and calcined in air at 550 °C for 1 h. The calcined powder (0.25 g) and the sediment (0.5 g) used in Colloid A were dispersed in the mixture of 8 mL butanol, 4 mL isopropanol and 2 mL ethanol to form Colloid B; (iii) The calcined MgO-coated SnO_2 powder (0.5 g) was dispersed in the mixture of 8 mL butanol, 4 mL isopropanol and 2 mL ethanol to form another colloid solution (Colloid C). An FTO glass ($1 \times 2 \text{ cm}^2$, $15 \Omega \text{ sq}^{-1}$, Opvtech) was used as the cathode and another FTO glass was used as the anode during the EPD process. The distance between the two electrodes was maintained at 1 cm and the DC power supply was set at 48 V. The EPD processes were all carried out in colloid C except those specifically mentioned. The deposition duration was fixed at 1, 2, 3, 4 and 5 min to obtain MgO-coated SnO_2 films with different thicknesses. The preparation condition of the samples was shown in Table 1. The deposited films were air-dried at room temperature, then, sintered at 550 °C for 1 h in air.

The crystalline phase of the samples was characterized by a DX-2700 X-ray diffractometer (XRD) with a monochromatized Cu K irradiation ($k=0.154145 \text{ nm}$). The morphology was studied using a JSM-7001F field emission scanning electron microscope (FE-SEM) and a JEM 2100 transmission electron microscope (TEM). Energy dispersive spectroscopy analysis (EDS) was obtained from Bruker-ASX (Model Quan-Tax 200). The specific surface area was measured by single-point nitrogen adsorption, using a specific surface area analyzer (JW-DA). The dye absorption amount was measured by a UV–vis–NIR photospectrometer (Varian Cary 5000). The Fourier Transform infrared spectra (FT-IR) were measured with a Nicolet AVATAR 360 FT-IR spectrophotometer. Zeta potential measurements were made on a Zetasizer NanoZS instrument (Malvern Instruments). The samples were suspended in alcohol (1 mg/mL) and dispersed by sonication before measurement.

2.2. Fabrication and characterization of dye-sensitized solar cells

For DSSCs fabrication, the samples were immersed in a $5 \times 10^{-4} \text{ mol dm}^{-3}$ ethanol of N719 dye (Cis-diisothiocyanato-bis(2,2'-bipyridyl-4,4'-dicarboxylato)ruthenium(II)bistetrabutylammonium) for 24 h. A transparent Pt counter electrode was

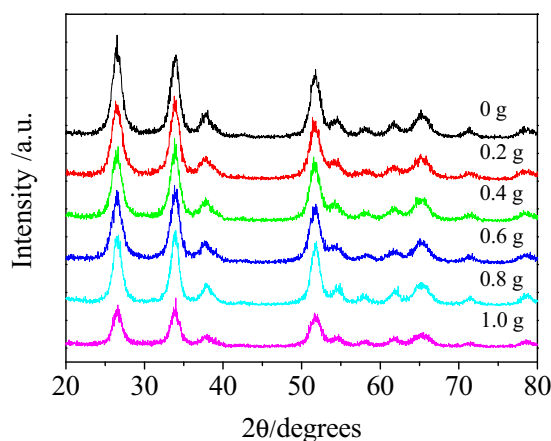


Fig. 1. X-ray diffraction patterns of pure SnO_2 and MgO-coated SnO_2 powder (with different addition of $\text{Mg}(\text{CH}_3\text{COO})_2$).

prepared by spreading 5 mmol dm^{-3} H_2PtCl_6 aqueous solution on an FTO glass substrate and pyrolyzed at 390 °C for 15 min. A mixture of 0.6 mol dm^{-3} dimethylpropylimidazolium iodide, 0.1 mol dm^{-3} iodine, 0.5 mol dm^{-3} 4-tertiarybutylpyridine, and 0.1 mol dm^{-3} lithium iodide in methoxy acetonitrile was used as the DSSCs electrolyte.

Photocurrent density–voltage (J - V) characteristics were measured using a Keithley 2440 Source Meter under AM 1.5G illumination from a Newport Oriel Solar Simulator with an intensity of one sun. The incident light intensity was calibrated with a standard Si solar cell provided by Newport Oriel. The active cell area of the assembled DSSCs was 0.25 cm^2 . The IPCE was measured as a function of the wavelength from 350 to 800 nm using an IPCE system designed especially for DSSCs (Crowntech, Inc.). A 150 W tungsten halogen lamp was used as the light source to generate a monochromatic beam. A silicon solar cell was used as the standard during calibration. IPCE values were measured using a Keithley model 2400 source meter. 5 mL NaOH solution (0.1 mol L^{-1}) was used to desorb the dye from MgO-coated SnO_2 electrodes. The amount of dye adsorption was calculated from absorption spectrum measurement of the desorbed dye solution by a UV–vis–NIR photospectrometer (Varian Cary 5000). The electrochemical impedance spectra (EIS) were also carried out with an electrochemistry workstation (CHI660d). The impedance measurement of DSSCs was recorded at the open circuit voltage under light illumination over a frequency range of 0.1–1 MHz with an AC amplitude of 10 mV.

3. Results and Discussion

3.1. XRD, EDS and TEM characterization of MgO-coated SnO_2 powder

Fig. 1 shows the XRD patterns of the uncoated and MgO-coated SnO_2 powder annealed at 550 °C for 1 h. All of the diffraction peaks in the XRD patterns were indexed as the pure cassiterite SnO_2 phase in good agreement with the reported data (JCPD card, NO. 41-1445). After modification, no MgO peak was observed, which was similar to bare SnO_2 particles. This indicated that the modification had no influence on the crystalline phase of the SnO_2 particles. The absence of the MgO peak may be due to the low MgO loading. But after coating with the MgO layer, all diffraction peaks of SnO_2 were slightly suppressed. The crystalline particle size (D) was estimated from the 101 diffraction peak using the Scherrer equation (Eq.1)[14].

$$D = 0.9\lambda / \beta \cos \theta \quad (1)$$

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