



Sol-gel derived cobalt sulphide as an economical counter electrode material for dye sensitized solar cells



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ABSTRACT

In this study, a solution-derived cobalt sulfide (CoS) was utilized as counter electrode (CE) in dye sensitized solar cell (DSSC). It is shown that high-quality CoS nanocrystals (10 ~ 500 nm) can be formed by post annealing process at temperatures above 500 °C. The deposited CoS nanocrystals effectively replace the commonly used platinum. Photovoltaic measurements revealed that the DSSCs assembled with the CoS CE showed excellent performance with open voltage of around 0.64 V, short circuit current of 7.1 mA/cm² and power conversion efficiency of around 5%.

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1. Introduction

Recently, there have been intensive research interests in the production of low-cost photovoltaic devices for renewable energy applications. The current photovoltaic industry is dominated by crystalline silicon that provides stable performances with good power conversion efficiency [1,2]. However, due to its heavy reliance on its raw material, the cost of crystalline silicon solar cells are vulnerable to price fluctuations of silicon and is still too expensive to replace grid electricity. In order to competitively provide energy from a renewable source, inexpensive photovoltaic technologies must be developed.

Among the available technologies, dyes sensitized solar cells (DSSCs) [3–6] are most promising, due to its reasonable power conversion efficiency and low cost equipment requirements. Typically, a DSSC consists of a (TiO₂)/dye/electrolyte/platinum, sandwiched together between two conductive fluorinated doped tin oxide (FTO) coated glass substrates [7,8]. The main function of the Pt is to accelerate the reduction of I₃⁻ to I⁻, which regenerates the dye molecules. Therefore, a good CE must possess high electrical conductivity and good electrocatalytic activity for electrolyte reduction. Although Pt possesses both of these characteristics, it is not an ideal choice of CE material due to its limited supply and high-cost [9–11]. Consequently, a great deal of research attentions have been focused on carbon-based nanomaterials, such as carbon nanotubes [12], activated carbon [13] and graphene [14]. Carbon-based nanomaterials have been proposed as a potential substitute to Pt due to its chemical stability against electrolytes and large surface area for reaction. In fact, previous studies have demonstrated that the carbon nanomaterials can achieved power conversion efficiency close to that of Pt-based DSSCs [15,16]. Wang et al. have [17] shown that superior DSSCs performances can be achieved by replacing Pt with cobalt sulphide (CoS), due to its ability to enhanced reduction of triiodide to iodide. In their study, the CoS films were synthesized by electroplating of cobalt chloride and thiourea at room temperatures. Subsequently, Lin [18,19] obtained a highly transparent CoS counter electrode that exhibit lower charge transfer resistance and resulted in enhanced power conversion efficiency of 6.01%. In a separate study, Kung [20] demonstrated an impressive DSSCs with power conversion efficiency of 7.67% by adapting CoS nanorod arrays as CE. Extensive literature review have revealed that CoS nanostructures can be deposited via chemical bath deposition

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[21], solvothermal process [22] and electrochemical deposition [23]. So far, there is still lack of studies reported regarding sol-gel deposited CoS CEs. The sol-gel deposition is attractive due to its simplicity and low cost. Therefore, it is of practical importance to assess the feasibility of using the sol-gel technique for CoS production.

In this paper, CoS films were deposited via a simple sol-gel deposition process. The samples were post-annealed at different temperature and characterized by using scanning electron microscopy (SEM), X-ray diffraction (XRD), energy dispersive spectroscopy (EDS) and four probe measurements. It is shown that the resistivity of the CoS film can be effectively reduced by increasing the annealing temperature. The optimized CoS layer was employed in a DSSCs and achieved power conversion of around 5.5% which is comparable with DSSCs with carbon and Pt.

2. Experimental procedure

The precursor solution was prepared by mixing 0.1 M cobalt acetate (Sigma Aldrich 99.999% purity) and 0.1 M thiourea (Sigma Aldrich 99.0% purity) in isopropanol. FTO coated glasses were cleaned using acetone, isopropanol and deionized water using ultrasonic bath before performing the deposition. The stock solution was then magnetically stirred for two hours and left to age for 24 h. The CoS films were deposited by a spin coater at 3000 rpm for 30 s and subsequently post-annealed at temperatures between 400 °C to 550 °C on a hotplate.

For the photoanode preparation, the TiO₂ suspension were prepared by mixing the TiO₂ nanoparticles (Degussa P25) with acetic acid. Photo-anode electrodes were manufactured by using TiO₂ paste onto the FTO glasses. The TiO₂ paste was coated onto FTO/glass substrate (~15 Ω) and heated to 450 °C on a hotplate to yield a thin layer (~15 μm). Then the photo anode electrode was immersed in N719(*cis*-di(thiocyanato)-*N,N'*-bis(2,2'-bipyridyl-4,4'-dicarboxylate)Ru(II)) dye (Eversolar) solution for 24 h to ensure even absorption onto the TiO₂ layer. Counter electrodes was fabricated using chemical bath derived CoS on FTO coated glass. The electrolyte were made from mixture of Li and I (0.3 M to 0.03 M) in ethylene glycol.

X-ray diffraction (XRD) experiments for the CoS thin film on glass substrates were carried out in a Siemens D5000 X-Ray diffractometer using Cu K α radiation. The Scanning electron microscopy (SEM) images were taken by using an FEI Quanta 400 F Environmental Scanning Electron Microscope (ESEM). The elemental composition analyses were measured by the Energy dispersive Spectroscopy (EDS) equipped on the SEM chamber. The electrical resistivity of the CoS films were measured by a four-point probe in Van der Pauw configuration. Cyclic voltammetry (CV) measurements CoS counter electrode were performed between -0.2 and 1.2 V in the aqueous electrolyte at a scan rate of 50 mV s⁻¹ on a CHI 660 electrochemical analyser. Electrochemical

impedance spectroscopy (EIS) measurements were performed in the dark with the same system by applying an AC voltage with 10 mV amplitude in a frequency range from 0.01 Hz to 100 kHz. The photovoltaic performances of the solar cells were evaluated using an Illuminated J-V characteristics were obtained using a solar simulator (Science tech).

3. Results and discussions

Fig. 1(a)–(d) presents the top view of the SEM images for chemically derived CoS film post annealed at different temperatures. It can be observed that the film morphology is strongly influenced by the annealing temperature. Fig. 1(a) shows the CoS films synthesized at 400 °C consist of agglomeration of small grains. As the annealing temperature is increased to 450 °C (Fig. 1(b)) larger grains can be observed, which is an indication of CoS film crystallization. The films underwent further structural changes as function of temperature at 500 °C and 550 °C, (Fig. 1(c) and (d)). It would appear from Fig. 1(c) that samples annealed at 500 °C resulted in merging of the grains and film formation. Fig. 1(d) shows the morphology of the film annealed at 550 °C (Fig. 1(d)) as ridge-like structure is formed. The growth of the CoS thin film can be explained by the Oswald ripening model, where thermodynamically-driven process occurs due to the fact that larger particles are more energetically stable than smaller particles. Therefore, the small clustering of atoms (Fig. 1(a)) will tends to aggregate, attach itself onto the surface of larger particles and eventually result in island formation (Fig. 1(a) and (b)). The ridge-like structure is similar to CoS nanomaterials obtained by other deposition method [19,24,25].

Fig. 2 shows the XRD data as a function of annealing temperature. The CoS films synthesized at 550 °C produced XRD peaks correspond to (100), (101), (102) and (110) hexagonal phase of CoS. Clearly confirming the transformation of amorphous films into nanocrystalline CoS. Fig. 2 also reveals that CoS films sintered below 550 °C are predominately amorphous with no obvious X-ray diffraction patterns. In the past, CoS nanoparticles have been successfully obtained through hot injection of precursors at around 187 °C [26], or even by successive ionic layer adsorption of Co_xO_y precursors in to Na₂S [27]. The difference in crystallization temperature can be attributed to the crystallization of film rather than just nanoparticle synthesis, it is much easier to produce CoS nanoparticles than the whole film.

EDS analysis was used to determine the elemental composition of samples. As shown in Fig. 3(a), the EDS measurements confirm the presence of Co, S and O. The chemically derived CoS films contain a significant amount of oxygen. These data suggests that in addition to CoS other by-product chemical compounds such as cobalt oxide and sulfur oxide may have also been formed. This is consistent with EDS and FTIR data obtained by other study [28], where significant amount of oxygen is present was also detected. Fig. 3(b) shows the extracted chemical composition as a function of deposition temperature. This figure revealed that as the temperature increases the oxygen content in the CoS film decreases, which is probably due to the desorption of water from the film.

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