



Solution processed NiO_x hole-transporting material for all-inorganic planar heterojunction Sb₂S₃ solar cells

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ABSTRACT

Light-harvesting material Sb₂S₃ has recently attracted tremendous attention due to its excellent photovoltaic properties. Extensive efforts have been exerted to improve the power conversion efficiency through process innovation, interface modification and band gap engineering. In this study, we report an all-inorganic planar heterojunction Sb₂S₃ solar cell using NiO_x as hole extraction material, which is deposited from preformed NiO_x nanoparticle solution. We demonstrate that the device performance can be significantly enhanced upon O₂ plasma treatment on NiO_x layer. As a result, O₂ plasma-treated NiO_x hole conductor leads to a 43% enhancement in power conversion efficiency when compared to untreated one, delivering an efficiency of 3.51%. The enhancement mechanisms are interpreted in terms of electronic structures and interfacial charge transport properties characterized by synchrotron-based high resolution ultraviolet photoelectron spectroscopy and electrochemical impedance spectroscopy. This work provides a choice of novel inorganic hole-transporting material for the preparation of stable and efficient all-inorganic solar cell device based on Sb₂S₃ as well as Sb₂(S,Se)₃.

1. Introduction

Solar electricity is regarded as one the most promising renewable energies for a sustainable society. In the field of solar cell, researcher have pursued practical solar cell device featured (1) high power conversion efficiency (PCE), (2) low production cost with non-toxic constituents and (3) long device lifetime. Binary compound antimony sulfide (Sb₂S₃) with non-toxic and earth-abundant constituents exists in the form of mineral stibnite in nature. It generally crystallizes in orthorhombic structure and possesses suitable band gap of 1.7–1.8 eV with high light absorption coefficient ($> 10^5 \text{ cm}^{-1}$) in visible region [1,2]. Because of the desirable minority carrier diffusion length of Sb₂S₃ [3], solar cell based on Sb₂S₃ as light absorption material is able to be constructed in either sensitized device configuration or planar heterojunction structure [4–9].

Currently, extensive efforts have been exerted to improve the PCE through innovation of preparation technology, interface modification and band gap engineering [1,4–6,10]. In specific, solid-state Sb₂S₃-based solar cells typically employ sandwiched structure of electron conductor/Sb₂S₃/hole conductor. The frequently applied electron

transporting layer (ETM) is mesoporous TiO₂ layer (mp-TiO₂) deposited onto compact TiO₂ layer (c-TiO₂) [6,11]. On the other hand, organic hole-transporting materials (HTM) are commonly used to extract hole from Sb₂S₃ [6,11–16]. Among these studies, the Seok group reported the highest efficiency of 7.5% in Sb₂S₃-sensitized solar cell using TiO₂ and PEDOT:PSS as ETM and HTM, respectively [6]. Considering the stability of solar cell, by contrast, all-inorganic solar cell device could be more desirable. With selenization treatment, Deng et al. reported an efficient and stable TiO₂/Sb₂S₃/Au planar solar cell with efficiency of 3.2% [17]. Yuan et al. reported the top value of 4.17% in HTM-free planar Sb₂S₃ solar cell with efficiency [18]. In addition, CuSCN and CuI have been applied for Sb₂S₃-sensitized solar cells [1,19–21]. Among them, the highest PCE of 5.7% for Sb₂S₃-sensitized solar cell with inorganic CuSCN was reported [19]. However, the deposition of CuSCN layer has practical difficulty arisen from the inevitably used air-sensitive solvent (thioether) to dissolve CuSCN powder, rendering potential problem in large scale production [22]. What's more, the complete dissolution of CuSCN in propyl sulfide usually requires very long time (3–4 days). In this perspective, searching for alternative air-stable and easy-fabricated inorganic HTM is of great significance for realizing

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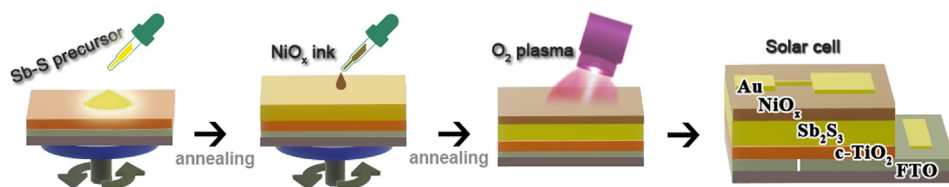


Fig. 1. Schematic flow of fabricating planar heterojunction Sb_2S_3 solar cell using NiO_x hole transporting material.

stable Sb_2S_3 -based solar cell.

In this study, we demonstrate that inorganic p-type NiO_x can act as efficient HTM for planar heterojunction Sb_2S_3 solar cells. Actually, due to its high air stability and hole mobility, NiO_x has been demonstrated to be a very promising HTL for fabricating high efficient inverted organic-inorganic hybrid perovskite solar cell (p-i-n configuration) in which active layer was deposited after NiO_x film [23,24]. Nevertheless, there has been no report on the application of NiO_x in Sb_2S_3 -based solar cell as far as we know. The preparation of NiO_x layer was mostly based on sol-gel process which involved a high temperature annealing step [25–27]. In the current study, as shown in Fig. 1, the Sb_2S_3 light-absorbing layer was prepared by a facile solution method. NiO_x nanoparticle was synthesized by thermal decomposition method and then dispersed in pure water solvent to form a nanoparticle ink. The deposition of NiO_x HTL is then conducted by spin-coating the nanoparticle ink in air followed by a low temperature annealing. Furthermore, we manifest that the photovoltaic performance of planar Sb_2S_3 solar cell can be significantly enhanced by a precise surface treatment of NiO_x nanoparticle film with O_2 plasma. The underlying mechanisms for the efficiency enhancement are interpreted from the perspectives of electronic structures of NiO_x and the interfacial charge transport kinetics in the planar heterojunction solar cells.

2. Experimental

2.1. Preparation of Sb-S and NiO_x solution

The synthesis of Sb-S and Sb_2S_3 was conducted by referring our previously reported method and others with further modifications [8,28]. Herein, we introduce DMSO to the reaction system. In specific, 0.1458 g Sb_2O_3 (purity > 99.99%) was loaded into a 5 mL vial containing 0.375 mL ethanol, 0.625 mL dimethyl sulfoxide (DMSO) and 0.75 mL carbon disulfide (CS_2). Under vigorous magnetic stirring, 0.8 mL n-butylamine was added in dropwise manner to prevent splashing caused by boiling, then tighten the lid. The as-obtained dark yellow solution was continued to be stirred for 30 min in nitrogen-filled glove box. Before spin-coating, the as-prepared solution was filtered with 0.22 μm PVDF filters.

To prepare NiO_x nanoparticles ink, First, 0.05 mol nickel (II) nitrate hexahydrate was dissolved in 10 mL deionized water. Under vigorous magnetic stirring, NaOH solution (10 M) was added drop by drop until the pH value reached 10, a turbid green solution was eventually formed. The precipitation was obtained by suction filtration and washed with deionized water, this procedure was repeated at least four times to get rid of impurity ions. The wet bulk was pre-dried at 80 °C for 4 h and ground into powder. NiO_x nanoparticles was obtained by annealing the hydroxide in air at 270 °C for 2 h. The NiO_x ink was freshly prepared just before spin-coating by dispersing NiO_x nanoparticles in deionized water with suitable concentration.

2.2. Fabrication of solar cell device

Prior to the deposition of the compact TiO_2 layer (c- TiO_2), the Fluorine-doped SnO_2 (FTO) substrate (15 Ω/sq) was etched with zinc powder and diluted HCl (2M) to obtain desired pattern. The etched FTO substrate was consecutively cleaned ultrasonically in acetone, isopropanol and ethanol for 45 min in each step and then treated in O_2

plasma for 20 min. A compact TiO_2 ETM of ca. 40 nm thickness was spin-coated onto FTO substrate from a mixture solution of 140 μL of titanium isopropoxide, 2 mL of ethanol, and 26 μL of HCl (2M) at 2000 rpm for 40 s, followed by annealing at 550 °C for 1 h in a muffle furnace. To deposit Sb_2S_3 light-absorbing layer, 60 μL Sb-S molecular precursor solution was spin-coated on c- TiO_2 at 8000 rpm for 30 s in a nitrogen-filled glove box (O_2 and H_2O < 0.1 ppm), the as-prepared film was pre-annealed at 200 °C for 1 min and further annealed at 300 °C for 2 min on hot plate to form dark brown Sb_2S_3 thin film. HTM was then directly deposited by spin-coating NiO_x nanoparticle ink at 6000 rpm for 40 s in air, and then annealed in a vacuum chamber.

To conduct O_2 plasma treatment, plasma cleaner (Harrick Plasma, PDC-001, equipped with a glow discharge radio frequency generator operated at 13.56 MHz with a maximum power of 30 W), which was connected with high-purity oxygen, was used to perform O_2 plasma treatment on NiO_x HTM. The working power was fixed to 7.16 W (low power level). The schematic illustration of O_2 plasma treatment apparatus can be seen in Fig. S1. Finally, device fabrication was completed by thermally evaporating 80 nm gold using a patterned mask (well-defined cell area of 0.12 cm^2).

2.3. Characterizations

The crystal structure characterization of Sb_2S_3 thin film and NiO_x nanoparticle was conducted by using X-ray diffractometer (Rigaku D/Max-rA) with $\text{Cu-K}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$). The surface morphology of thin films was characterized by field emission SEM (FE-SEM, Sirion 200). The optical absorption spectrum was recorded on a UV-Vis-NIR spectrophotometer (SOLID 3700) in the range of 200–1600 nm. The electrical properties were characterized by Hall Effect measurement (Vander Paw configuration) operated at room temperature, the Au contact with desired pattern was evaporated on the film by using a customized mask (four electrical contacts arranged in a square). XPS measurements were conducted on a Thermo ESCALAB 250 instrument with a monochromatized Al K α . HRTEM observation was performed on JEOL-2011F with an acceleration voltage of 200 kV. The current density-voltage (J-V) characterizations were conducted using Keithley 2400 source measurement unit under simulated AM1.5 irradiation (100 mW/cm^2) provided by a standard xenon-lamp-based solar simulator (Oriel Sol 3 A, USA), the solar simulator illumination intensity was calibrated by a monocrystalline silicon reference cell (Oriel P/N 91150 V, with KG-5 visible color filter) calibrated by the National Renewable Energy Laboratory (NREL). The EQE measurements were carried out on an ORIEL IQE-200™ measurement system. Electrochemical impedance spectroscopic measurements (EIS) were performed using Zahner Zennium PP211 electrochemical workstation with the frequency range from 1 MHz to 1 Hz under -0.4 V bias in the dark, and AC 10 mV perturbation was applied. The obtained impedance spectra were fitted with ZView software (v3.1, Scribner Associates). UPS experiments were performed at the Catalysis and Surface Science Endstation at the BL11U beamline in the National Synchrotron Radiation Laboratory (NSRL) in Hefei, China. The valance band (VB) spectra were measured with a photon energy of 40 eV. A sample bias of -5 V was applied to observe the secondary electron cutoff (SEC). The work function (ϕ) can be determined by the difference between the photon energy and the binding energy of the secondary cutoff edge.

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