Tin Sulfide/Gallium Oxide Heterojunctions for Solar Water Splitting

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Abstract: Tin (II) sulfide (SnS) is a promising semiconductor material for next-generation solar energy conversion due to its favorable bandgap, elemental abundance, low toxicity, and low cost. A major challenge, however, lies in the low open circuit voltages that are typically obtained in SnS-based devices. Herein, a low-cost solution-phase deposition technique is used to prepare SnS thin films and investigate different junction materials (Ga2O3 and In2S3) to improve the photovoltage in SnS-based water splitting photocathodes. Molecular inks are prepared by dissolving SnS powder in solvent mixtures of ethylenediamine and 1,2-ethanediithiol. SnS thin films are then successfully deposited by spin coating the inks onto substrates, followed by a heat treatment at 350°C in an inert atmosphere. With a photoelectrode based on a SnS/Ga2O3 heterojunction, an onset potential of +0.25 V versus reversible hydrogen electrode (RHE) is achieved for photoelectrochemical hydrogen evolution in pH 7 phosphate buffer, which is until now the earliest onset potential (highest photovoltage) among nontoxic replacements to CdS junctions in SnS-based water splitting systems.

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Tin Sulfide/Gallium Oxide Heterojunctions for Solar Water Splitting

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1. Introduction

Photoelectrochemical (PEC) water splitting is a promising route to “green” hydrogen manufacture,[1] which integrates the solar absorber and electrolyzer into a single device and has some advantages over photovoltaic-powered electrolysis, assuming efficiency and stability standards are met.[2–4] To compete economically with hydrogen produced from fossil fuels, the PEC water splitting devices should be scalable and low cost, which implies the use of earth-abundant, nontoxic, and cheap elements, stability against (photo)corrosion, and cost-effective fabrication.

One promising photoabsorber material for large-scale water splitting is tin sulfide, a binary chalcogenide material composed of tin and sulfur, which are both earth-abundant elements. Tin has two main oxidation states of Sn$^{2+}$ or Sn$^{4+}$ and a versatile coordination chemistry from 2 to 9, which results in a variety of phases of tin sulfide compounds.[5,6] Among the rich phase space of tin sulfides, thermodynamically the most stable one is the orthorhombic Pnma phase, referred to as herzenbergite or α-SnS.[7] In this structure, the Sn$^{2+}$ ion coordinates to three $S^{2-}$ ions, with the Sn $S^2$ lone pair occupying the last position of a tetrahedral geometry.[8] The lone-pair electrons on the tin ions repel each other, resulting in a layered structure. The layered structure of SnS results in anisotropic properties of the material. It has a natural p-type conductivity, which is attributed to tin vacancies ($V_{Sn}$) in the lattice.[8]

Various deposition methods have been developed for SnS thin films, including thermal evaporation,[9] chemical vapor deposition (CVD),[10] atomic layer deposition (ALD),[11] sputter deposition,[12] spray pyrolysis,[13] chemical bath deposition (CBD),[14] and electrodeposition.[15] Major developments in the last decade have been mainly driven by the group of Gordon (Table S1, Supporting Information). The record efficiency of 4.36% in solar cells was achieved by the Gordon group in 2014 using an ALD SnS absorber layer and Zn(O,S) junction layer optimized for maximum efficiency.[16] A following study used a faster deposition method, congruent thermal evaporation, and reached 3.88% efficiency in a photovoltaic (PV) configuration.[9] Research on SnS-based PEC cells is a more recent phenomenon. The first demonstration of an SnS-based PEC cell was reported by Gao et al. in 2016.[17] The SnS layer was deposited by spray pyrolysis on FTO followed by an n-type CdS layer to make a p–n junction. Since then a number of works have been published, indicating that interest is increasing for the use of SnS thin films for PEC water splitting (Table S2, Supporting Information).

A promising low-cost deposition method for solar absorber materials is based on molecular inks, which are molecular precursor solutions in the form of a single phase—a homogenous liquid—and are distinguished from slurries, suspensions, or colloidal dispersions of nanoparticles. Using a variety of deposition techniques—spin coating, spray coating, doctor blading, printing—the ink can be loaded as a thin layer on any desired substrate. Upon heating, the solvent molecules evaporate and/or decompose (or precursor molecules can be decomposed) and crystalline chalcogenide films are formed. However, the covalent character of the metal chalcogenides hinders the use of traditional solvents such as water, ethanol, or many other organic solvents. Bruchey and co-workers have shown that a thiol–amine solvent mixture can dissolve many different chalcogenide materials,[18–20] and PV cells prepared using such methods have given promising efficiencies (Table S3, Supporting Information).
Information). In this work, we used molecular inks based on a thiol–amine solvent mixture to fabricate SnS photoabsorber materials and investigated novel junction materials to improve the photovoltage of these devices.

2. Results and Discussion

The SnS ink was prepared by dissolving a commercial SnS powder directly in a thiol–amine solvent mixture under an inert atmosphere (see experimental section for details). With rigorous stirring, the dissolution of the fine powder was on the order of minutes, whereas that of millimeter-sized particles took several hours. The maximum solubility of SnS in the solvent mixture was reported to be 120 mg mL\(^{-1}\) at 25 °C and 1 atm, corresponding to a 0.8 m solution.\(^{[23]}\) In this work, typically 0.5 m inks were prepared, resulting in a transparent solution of slight yellow tint without any visible light scattering by particles. The inks were stable over several months under an inert atmosphere without exhibiting any color change or precipitation. After spin coating the inks on the Au-coated FTO substrates and annealing them at 350 °C, dark gray/black thin films were observed (Figure 1).

The SnS powder was insoluble in en alone and a strong exothermic reaction was observed following the addition of edtH\(_2\). An acid–base reaction between en and edtH\(_2\) results in extensive ion formation. It is proposed that the thiolate anions break down the Sn–S bonds through nucleophilic attack on the tin ions. A \(^{119}\)Sn NMR study revealed that the nature of Sn in the solution is a four-coordinate Sn\(^{2+}\)-thiolate complex.\(^{[18]}\) The Sn\(^{2+}\) are stabilized by thiolate anions, whereas S\(^{2-}\) are counterbalanced with an ammonium cation.

Thermogravimetric analysis (TGA) and differential thermal analysis (DTA) were used to investigate the phase formation of the metal chalcogenide. The TGA curve of the ink showed a two-step mass loss of 84% from 30 to 325 °C (Figure S1, Supporting Information). The first dominant mass loss between 25 and 150 °C coincides with a large endothermic DTA peak, which can be assigned to solvent evaporation and/or decomposition. Also, a transformation of the transparent ink layer to a dark-colored film was observed on the hotplate during a similar temperature range.

Recovery of the SnS orthorhombic \(Pnma\) phase from the ink after annealing at 350 °C was confirmed by XRD measurements (Figure 2). The Tauc plot (Figure S2, Supporting Information) shows a direct bandgap value of 1.05 eV and an indirect bandgap of 1.7 eV, which are in line with the values reported in the literature.\(^{[22,23]}\) Variation in the optical properties of SnS with respect to deposition techniques and conditions has been reported.\(^{[22,23]}\)

The water-splitting capability of the SnS thin film was assessed in a pH 7 phosphate electrolyte solution under simulated AM 1.5G illumination (100 mW cm\(^{-2}\)) after coating the SnS layer with nanoparticulate Pt as a hydrogen evolution reaction (HER) catalyst. Although the electrode generated photocurrents over several mA cm\(^{-2}\) (≈3 mA cm\(^{-2}\) at −0.25 V vs reversible hydrogen electrode [RHE]), a late onset potential around −0.15 V versus RHE indicated a poor photovoltage (Figure S3, Supporting Information). Therefore, we assessed a buried junction approach with charge extraction and corrosion protection layers to improve the photovoltage and stability of the system.

The design of p–n junctions for SnS PEC cells aims at providing a maximum built-in potential. Assuming the recombination at the junction interface is similar, a larger difference between the Fermi levels of each material will favor a larger photovoltage (Figure S4, Supporting Information). In addition, the position of the valence band edge of the n-type material is more positive (on the electrochemical scale) relative to that of SnS. The latter factor is to minimize recombination by forming a barrier for holes to reach the electrolyte solution. A few additional factors were considered in searching for a suitable n-type material for p-type SnS. It should grow well on the SnS layer by easy and inexpensive methods and conditions that do not destroy the SnS film. In addition, it should have good charge transport properties and be robust against photocorrosion. To protect the p–n junction, a conformal layer of 50 nm thick TiO\(_2\) was deposited by ALD. The complete photodegradation fabrication procedure is shown in Figure 3.

We studied two different junction materials: indium sulfide and gallium oxide. Indium sulfide (In\(_2\)S\(_3\)) is an n-type semiconductor with an optical bandgap of 2.1–2.3 eV.\(^{[24]}\) It is considered an effective and nontoxic substitute for CdS and has been applied as a junction layer on CIGS-based solar cells.\(^{[25]}\) The conduction band minimum of In\(_2\)S\(_3\) is expected to fall between those of SnS

![Figure 1](https://www.entechnol.de)

**Figure 1.** a) Ink formulation from the bulk SnS powder to a molecular ink in the ethylenediamine (en) and 1,2-ethanedithiol (edtH\(_2\)) solvent mixture. b) Optical image (sample size 25 mm \(\times\) 25 mm) and an SEM micrograph of the resulting SnS thin film. c) A schematic illustration of SnS thin film deposition on a Au-coated FTO substrate using molecular inks by spin coating.
and TiO$_2$, enabling electron flow to the electrolyte. An In$_2$S$_3$ layer was deposited by CBD. After deposition of the In$_2$S$_3$ layer, a 50 nm layer of TiO$_2$ was deposited by ALD, and Pt was deposited by photoelectrodeposition. The SnS/In$_2$S$_3$/TiO$_2$/Pt photocathode showed an improved onset potential of 0.16 V versus RHE (Figure 4a, red curve).

Gallium oxide (Ga$_2$O$_3$) is mainly studied in the transistor field as a wide-bandgap semiconductor material. The ALD grown Ga$_2$O$_3$ is amorphous with a band gap of 5.25 eV, which makes it attractive as a junction layer from a practical perspective as it is transparent, minimizing the loss of light absorption in the underlying SnS layer. It was first applied in Cu$_2$O-based solar cells as an n-type junction layer, demonstrating high open circuit voltage of the solar cells.

To demonstrate the effectiveness of a Ga$_2$O$_3$ junction layer for SnS-based PEC cells, a thin layer of Ga$_2$O$_3$ was grown by ALD on the SnS thin film. The capability to deposit Ga$_2$O$_3$ by ALD was advantageous because a conformal layer is assured with easy tunability of the film thickness. The Ga$_2$O$_3$ layer 20 nm thick was shown to form an effective p–n junction with SnS. The device fabrication was completed by a subsequent deposition of 50 nm of ALD TiO$_2$ and Pt deposition. An scanning electron microscopy (SEM) cross-sectional image of the SnS/Ga$_2$O$_3$/TiO$_2$/Pt electrode is shown in Figure 3c. The device exhibited...
>350 mV improvement in the onset potential compared to the SnS/Pt PEC cell. This is a further 80 mV photovoltage gain compared to the In$_2$S$_3$ junction layer devices (Figure 4). This could be due to better band alignment with SnS (Figure S4, Supporting Information) and/or elimination of other recombination sources such as surface recombination by passivation of the interface.

To test the stability of the SnS/Ga$_2$O$_3$/TiO$_2$/Pt photoelectrodes, the time dependence of the photocurrent was measured at 0 V versus RHE under 1 sun irradiation, as shown in Figure 5. The photocathodes maintained relatively stable photocurrent with a marginal but continuous decrease in photocurrent over 30 min. The decrease in photocurrent was due to the loss of active catalyst on the TiO$_2$ surface. After redepositing Pt, the lost

Figure 4. $J$–$V$ curves of SnS/In$_2$S$_3$/TiO$_2$/Pt (red) and SnS/Ga$_2$O$_3$/TiO$_2$/Pt (blue) and a schematic diagram of the layered SnS-based photocathode. An optical image of the SnS-based photocathode is shown on a 4 mm × 4 mm grid.

Figure 5. The chronoamperometry of SnS/Ga$_2$O$_3$/TiO$_2$/Pt photoelectrodes at 0 V versus RHE bias in pH 7 phosphate buffer electrolyte under intermittent 1 sun illumination.
photocurrent was regained, indicating that the SnS/Ga2O3/TiO2 junction is stable (Figure S5, Supporting Information). It is suspected that the Pt particles are detached due to bubble formation of hydrogen on the surface.

Although promising photovoltages were obtained with In2S3 and Ga2O3 junction layers, the photocurrent remains rather low compared to the reported photocurrents of SnS-based PV cells. Enhancing the bulk film quality should improve the photocurrent density. The grain size is rather small (Figure 1b) and generating larger grains should minimize interfacial recombination. Furthermore, surface etching or passivation treatment may reduce the surface defects, which we suspect are one of the major sources of recombination.

3. Conclusion

SnS thin films were successfully deposited using thiol–amine-based SnS molecular inks. SnS alone as a photocathode suffered from corrosion and low photovoltage for the PEC hydrogen evolution reaction. In2S3 and Ga2O3 were investigated as novel n-type junction layers for SnS. Both In2S3 and Ga2O3 were shown to form a suitable p–n junction, with SnS exhibiting a further 250–300 mV improvement in the onset potential (photovoltage). The SnS/Ga2O3 junction provided a larger photovoltage than SnS/In2S3. It is speculated that the position of the Fermi level of Ga2O3 relative to the p-type SnS Fermi level is suitable for generating a large photocurrent. Further improvements could potentially be obtained with appropriate interface treatments to minimize recombination. The SnS/Ga2O3 junction was stable over 30 min of operation at 0 V versus RHE. Improving the anchoring of the catalyst on the TiO2 corrosion protection layer is important to maintaining the steady state operation of the device.

4. Experimental Section

SnS Molecular Ink Preparation: Seven fifty-four milligrams of SnS (5 mmol, Sigma-Aldrich, >99.99%, USA) was dissolved in 9.1 mL 1,2-ethylene diamine (Sigma-Aldrich, USA) and 0.9 mL 1,2-ethanedithiol (Sigma-Aldrich, USA) to produce a 0.5 m ink. The solution was stirred at 60 °C overnight and then filtered through a 0.2 μm polytetrafluoroethylene (PTFE) membrane. The inks were prepared either by standard Schlenk line techniques or in a N2-filled glove box.

Substrate Preparation: An FTO on glass window (FTO TEC 15, Pilkinson, Tokyo, Japan) was cut into pieces (2.3 cm x 2.5 cm) and cleaned by sonication in Deconex solution in water (5%), distilled water, acetone, and then EtOH for 10 min each. After drying in a stream of N2, 10 nm of Cr followed by 150 nm of Au was deposited using a Safematic CCU-010 sputter coater. The sputter coater chamber was vented between Cr and Au deposition to change the sputtering target. Before coating, the Au surface was cleaned by O2 plasma for 30 min using a Diener electronic ZEPTO apparatus.

Spin Coating and Annealing: All spin-coating processes were done using a Laurell WS-650Mz-23NPPB spin coater. First 120 μL of the ink was loaded on the substrate and then spin coated at 1250 rpm for 1 min, using an acceleration of 990 rpm s⁻¹. After spin coating, the samples were immediately dried on a hot plate (in the nitrogen-filled glove box) at 180 °C for 3 min and then the temperature was ramped to 350 °C at a ramping rate of 11 °C min⁻¹ and kept for 30 min. After the annealing, the samples were removed from the hotplate and cooled to room temperature, which took ≈1 h. This process was repeated twice to obtain ≈400 nm thick SnS thin films. The samples were then left in ambient air overnight to grow a thin layer of SnOx on the surface.[31]

Atomic Layer Deposition of Ga2O3 Thin Films: All ALD layers were fabricated using a Picosun R200 system. ALD layers of Ga2O3 film were deposited at a reactor temperature of 160 °C using bis(μ-dimethylamino) tetrais(dimethylamino)digallium (STREM, 98%) and H2O as the precursors of Ga and O, respectively. Each cycle consists of 2.5 s of Ga precursor pulse followed by a 7.0 s N2 purge and a subsequent 0.1 s pulse of H2O followed by a 4.0 s N2 purge. Two hundred and fifty cycles were applied to obtain a 20 nm thick Ga2O3 layer.

Chemical Bath Deposition of In2S3: In a 100 mL beaker, 829.4 mg of In2(SO4)3·xH2O (Merck, 99.99%), 567.2 mg of thioacetamide (Sigma Aldrich, ≥99%), and 0.45 mL of acetic acid were mixed first, and then 75 mL of deionized water was added. The solution was sonicated for ≈30 min with intermittent stirring to produce a homogeneous solution, which was clear and colorless. The solution beaker was immersed in a water bath heated at 70 °C. After ≈45 min, once the solution started to turn into pale yellow, the SnS thin films were dipped in the solution. After 15 min of deposition, the samples were taken out and rinsed with deionized water and dried with a gentle stream of N2. The SnS/In2S3 samples were then immediately transferred to the ALD chamber for ALD TiO2 deposition.

Atomic Layer Deposition of TiO2: All ALD layers of TiO2 were deposited with tetraakis(μ-dimethylamido)titanium(IV) (TDMAT) (99.999%, Sigma-Aldrich, Buchs, Switzerland) and Milli-Q water as precursors, and N2 (99.9999%) as carrier gas. The titanium precursor was heated to 85 °C for the deposition process and the water was kept at 25 °C. The substrate and reactor temperature were 120 °C during the deposition. Each cycle consisted of an initial 1.6 s (0.5 s dosing to the reactor, 1.2 s of increased line pressure) of TDMA pulse, followed by a 6.0 s N2 purge. Then a 0.1 s pulse of H2O was used, followed by a 6.0 s N2 purge. Growth of the layers was tracked by inserting a 1 cm × 1 cm piece of silicon wafer into the reactor and measuring the resulting film thickness by ellipsometry. An average growth rate of 0.054 nm cycle⁻¹ was observed.

Photoelectrodeposition of Pt Catalyst: The platinum catalyst was photoelectrodeposited from an aqueous solution of 1 m H2PtCl6 containing 1 M Na2SO4, using a constant current of −0.03 mA cm⁻² for 10 min under 1 sun illumination.

Characterization of SnS Thin Films: The SEM images of SnS thin films were obtained using an SEM, a Zeiss Supra 50VP or Zeiss Gemini 450, which was equipped with an energy-dispersive X-ray spectroscopy (EDX) detector (genesis). X-ray diffraction was performed on a Rigaku SmartLab diffractometer with Cu Kα (λ = 0.15406 nm).

Photoelectrochemical Characterization of SnS Photocathodes: The photoelectrochemical performance of the SnS-based electrodes was evaluated in a three-electrode configuration under simulated AM 1.5 illumination (1 sun) using a Biologic sp-50, sp-200, or sp-300 potentiostat. Pt wire was used as the counter electrode. Experiments performed in aqueous electrolytes typically without purging with N2, as there was no distinctive difference compared to purged electrolytes, were measured against an aqueous Ag/AgCl electrode in saturated KCl. Voltages reported on the RHE scale were obtained by converting the potentials measured with the Ag/AgCl electrode using the Nernst equation, according to the following formula

\[ E_{\text{RHE}} = E_{\text{Ag/AgCl}} + 0.059 \times \text{pH} + E^0_{\text{Ag/AgCl}} \] (1)

The photopotentials were measured under chopped illumination with a scan rate of 10 mV s⁻¹ for the linear sweep voltammetry from positive to negative bias direction.

TGA and DTA: TGA and DTA were performed simultaneously in a Netzsch STA 449 F3 Jupiter instrument. An alumina crucible was used to dry 60 mg of the ink. The sample was heated from 25 to 400 °C at 5 K min⁻¹ and held at this temperature for 30 min.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.
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Conflict of Interest
The authors declare no conflict of interest.

Data Availability Statement
Data available on request from the authors.

Keywords
• gallium oxide, molecular inks, photoelectrochemical water splitting, p–n junctions, tin sulfide

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