Surface modifications of chalcopyrite CuInS2 thin films for photochatodes in photoelectrochemical water splitting under sunlight irradiation

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Surface modifications of chalcopyrite $CuInS_2$ thin films for photochatodes in photoelectrochemical water splitting under sunlight irradiation

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Abstract. Copper chalcopyrite semiconductors include a wide range of compounds that are of interest for photoelectrochemical water splitting which enables them to be used as photochatodes for H_2 generation. Among them, $CuInS_2$ is one of the most important materials due to its optimum band gap energy for sunlight absorption. In the present study, we investigated the application of $CuInS_2$ fabricated by electrodeposition as photochatodes for water splitting. Thin film of $CuInS_2$ chalcopyrite was formed on Mo-coated glass substrate by stacked electrodeposition of copper and indium followed by sulfurization under H_2S flow. The films worked as a H_2 liberation electrode under cathodic polarization from a solution containing Na_2SO_4 after loading Pt deposits on the film. Introduction of an n-type CdS layer by chemical bath deposition on the $CuInS_2$ surface before the Pt loading resulted appreciable improvements of H_2 liberation efficiency and a higher photocurrent onset potential. Moreover, the use of In_2S_3 layer as an alternative *n*-type layer to the CdS significantly improved the H_2 liberation performance: the $CuInS_2$ film modified with In_2S_3 and Pt deposits worked as an efficient photocathode for photoelectrochemical water splitting.

1. Introduction

The shortage of fossil sources has made an effort to get alternative energies such as hydrogen through its conversion to energy by direct combustion in conventional engines or reaction with pure oxygen in a fuel cell. Hydrogen can be produced by natural gas reforming, electrolysis, biomass, thermochemical, thermophysical, photoelectrochemical (PEC) methods, and photo-biological process [1]. Hydrogen (H_2) production by PEC water splitting is considered to be an attractive in view of energy and environmental issues.

Since the first report of a TiO_2 thin-film photoelectrode [2], a variety of semiconductor electrodes and devices have been investigated. To date, performances with conversion efficiency as high as 12.4% have been demonstrated for electrodes based *p*-$GaInP_2$/GaAs cells grown by atmospheric-pressure organometallic vapor-phase epitaxy [3]. However, due to limited corrosion resistance in aqueous electrolytes and expensive cost for production of these electrodes, these systems are not feasible for practical applications. Meanwhile, corrosion-resistant and inexpensive transition metal oxides are well-studied, but conversion efficiencies for these electrodes are not sufficiently high yet

due to the lack of optical and photoelectrochemical properties required for realizing high photocurrents and H_2 evolution rates.

Cu-chalcopyrite type semiconductors such as $CuInSe_2$, $CuGaSe_2$, $CuInS_2$, $CuGaS_2$ and their mixed crystals are used as absorber layers in thin film solar cells. Due to their high absorption coefficient and tunable band gap values (1.0-2.4 eV) by changing the In/Ga and/or Se/S ratios, these materials are also attractive use as photocathodes for H_2 production [4-6]. Electrodeposition is one of the promising technologies for fabrication of low–cost Cu-chalcopyrite films because of the low cost equipment, scalability of large scale, and good control over film composition and morphology [7].

The insertion of n-type thin layers on photocathodes to improve the photo response had been done by reseachers using different absorbers, which are widely employed to fabricate *p-n* junctions for chalcophyrite and kseterite based solar cells. To date the surface modification of calcopyrite and kseterite [8] for photoelectrochemical water splitting had been done using n-type CdS by chemical bath deposition (CBD) and resulted apreciable current densities compared to the bare or Pt-thus films [9-15]. Meanwhile CdS is a toxic compound that should be avoided. Therefore in this reseach we tried to use In_2S_3 by CBD alternative to CdS as *n*-type thin layer on $CuInS_2$ photocathodes.

2. Experiment methods

Synthesis of the $CuInS_2$ thin film

The $CuInS_2$ thin film was synthesized by electrodeposition of Cu and In layers followed by sulfurization. Electrodeposition was carried out potentiostatically using a Hokuto Denko HSV-110 potentiostat-galvanostat under N_2 atomosphere without stirring. A vertical three-electrode setup consisting of an Ag/AgCl reference electrode, a Pt wire counter electrode, and an Mo-coated glass substrate (Mo/glass) as a working electrode (area of the working electrode being 0.7 cm^2) was employed. The electrolyte solution for Cu deposition consisted of 50 mM $CuSO_4$, 150 mM trisodium citrate, and 242 mM citric acid. The solution was adjusted to pH 2.4 using H_2SO_4. For In deposition, the electrolyte solution consisted of 30 mM $InCl_3$, 242 mM citric acid, and 36 mM trisodium citrate. Potentials used for Cu and In deposition were -0.2 V and -0.76 V (*vs*. Ag/AgCl), respectively. Electric charges of Cu and In deposition were fixed at 0.73 C and 0.84 C, respectively: this resulted in the composition ratio of Cu and In (Cu/In) in a Cu and In stacked layer of 1.3. The Cu/In stack as-deposited was then heated at 160 °C for 30 min under Ar flow (200 mL min^{-1}), followed by sulfurization at 560 °C under H_2S flow (5% H_2S in Ar, 200 mL min^{-1}) for 10 min in a glass tube furnace. Thus-obtained $CuInS_2$ films were then etched by immersion in an aqueous KCN solution (10%) for 2 min to remove excess Cu_xS components.

Surface modifications of the $CuInS_2$ thin film

A $CuInS_2$ thin film was added to an aqueous solution containing 12.5 mM $CdSO_4$, 0.22 M thiourea, and 11 M of ammonia; deposition of CdS was performed at 60 °C for 7 min. For In_2S_3 deposition, the $CuInS_2$ thin film was immersed in an aqueous solution containing 25 mM $In_2(SO_4)_3$, 0.1 M thioacetamide and 0.1 M acetic acid at 65 °C for 15 min. Loading of platinum deposits on bare $CuInS_2$ and these modified-$CuInS_2$ thin films was conducted by photoirradiation of the film in an N_2-saturated 0.1 M Na_2SO_4 solution containing 1 mM H_2PtCl_6 (with pH adjusted to 4.0) at -0.1 V (vs. Ag/AgCl) for 10 min. An Asahi Spectra HAL320 solar simulater was used as a light source.

PEC measurements

PEC water splitting was also performed using the three-electrode setup in an aqueous solution of 0.1 M Na_2SO_4 with pH adjusted to pH 9.0 under N_2 or Ar. Transient photocurrents were obtained by scanning applied potentials with a scan rate of 10 mVs^{-1} under chopped illumination of simulated AM 1.5 solar irradiation from the above solar simulator. Applied bias photon-to-current efficiency (ABPE) for photocathode was calculated from the data according to the following equation:

$$ABPE\ [\%] = J \times V_b \times 100 / P_{AM1.5},$$

where J (mA cm^{-2}) is the measured photocurrent density, V_b (V) is the bias voltage measured by the reversible hydrogen electrode (RHE) scale (V_{RHE}, see below), and $P_{AM1.5}$ is the photon flux of the AM 1.5 simulated sunlight (100 mW cm^{-2}). Incident photon-to-current conversion efficiency (IPCE) spectra of Pt-In_2S_3/$CuInS_2$ and Pt-CdS/$CuInS_2$ were also calculated using the following equation:

IPCE [%] = 1240 × J × 100 / (λ × P_i),

where J (mA cm^{-2}) is the measured photocurrent density, λ is the wavelength of incident photons and P_i is the incident light intensity (mW cm^{-2}). The measurement was performed at -0.73 V (vs. Ag/AgCl) by the lock-in technique using an NF LI5630 digital lock-in amplifier. For the measurement, photoirradiation was performed by chopping at 10 Hz of monochromatic light, which was obtained by passing light from the solar simulator through a monochromator. The number of incident photons was determined by an OPHIR Orion Laser power meter equipped with a photodiode. A PEC cell connected to an online gas chromatography system (INFICON 3000 Micro GC Gas Analyzer equipped with an MS-5 A column and a thermal conductivity detector) was used to detect H_2 and O_2 during the PEC water splitting. The PEC cell was immersed in a water bath to maintain the reaction temperature at 288 K, and photoirradiation was performed at -0.73 V (vs. Ag/AgCl) by using a Cermax LX-300F 300 W xenon lamp. The potentials in each measurement measured vursus Ag/AgCl electrode ($E_{Ag/AgCl}$) were converted into E_{RHE} using the following equation:

$$E_{RHE} = E_{Ag/AgCl} + 0.059 \times pH + 0.199.$$

Characterizations

Crystal structures were measured by X-ray diffraction (XRD) using a Rigaku MiniFlex X-ray diffractometer (Cu Kα, Ni filter). Surface morphologies were examined using a Hitachi S-5000 FEG field emission scanning electron microscope (FE-SEM). Elemental compositions of thus-obtained films were determined using a Hitachi TM3000 scanning electron microscope equipped with a Swift ED3000 energy dispersive X-ray spectrometer (EDX).

2. Results and Discussion

In this study, a $CuInS_2$ was fabricated from electrochemically stacked layers of metallic Cu and In followed by sulfurization. The composition ratio of Cu and In (Cu/In) in metallic precursor was fixed at a Cu-rich composition (Cu/In = *ca.* 1.3) compared to the stoichiometric ratio of the final film. It is known that a high Cu/In ratio is desirable for obtaining a $CuInS_2$ film with high crystallinity and large grain sizes, due to the crucial role of Cu_xS impurity phase(s) to accelerate the growth of $CuInS_2$ crystallinity during the sulfurization process. The stacked metallic precursor film obtained on a Mo/glass substrate showed good uniformity and adherence. After sulfurization, the semi-glossy film with a white color became non reflective. A slightly bluish gray color of the resulting film suggests surface coverage of Cu_xS components that dissapeared after KCN etching, The elemental compositions of the film measured by EDX were 27.473, 25.087 and 47.439 % for copper, indium and sulfur, respectively (data not shown). The XRD pattern (Figure 1) of thus-obtained film showed typical difraction peaks assignable to the $CuInS_2$ crystal with a chalcopyrite structure, in addition to peaks due to the Mo substrate. The as-deposited film shows several diffraction peaks assignable to Cu, In and CuIn alloy. It is noted that we performed preheating under Ar to the as-deposited bilayer film before sulfurization to form the Cu-In alloy to suppress the formation of structural void on the fabricated $CuInS_2$ film [16]. XRD pattern of the sulfurized film showed typical diffraction pattern of the chalcopyrite $CuInS_2$ crystal without any impurity elements [17].

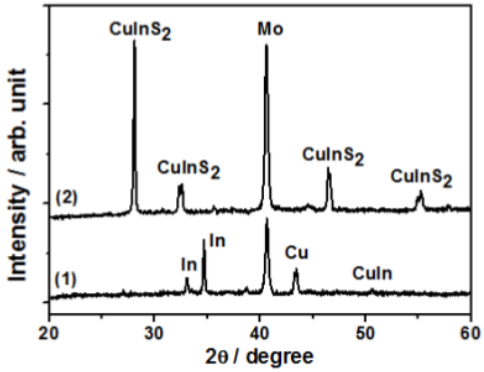

Figure 1. XRD patterns of as-deposited CuIn (1) and $CuInS_2$ (2) on Mo substrate.

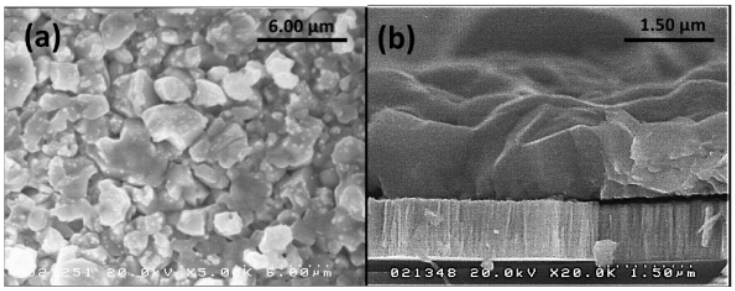

Figure 2. SEM images of Sulfurized samples ($CuInS_2$).

Figure 2 Shows the scanning electron microscope (SEM) image of the $CuInS_2$ film after KCN etching. The top view SEM image indicates that the film consists of compact agglomerates of angular-shaped crystallite (Figure 2a). Cross sectional SEM image of the corresponding sample in Figure 2b also shows regularly shaped crystals with grain sizes ranging from 0.5 to 1.5 μm.

Figure 3 shows Current-potential curves for bare-$CuInS_2$, Pt-$CuInS_2$, Pt-In_2S_3/$CuInS_2$, and Pt-CdS/$CuInS_2$ under 0.1 M Na_2SO_4 at pH 9 under AM 1.5 chopped Xe lamp illumination. Figure 3A shows the bare-$CuInS_2$ film showed little photocurrents due to recombination and leakage [18], whereas the Pt-$CuInS_2$ film exhibited appreciable increases in photocurrents, indicating the effectiveness of loading Pt deposits as promoters for H_2 production and act as activation sites for evolution of hydrogen and improve the kientics of reaction, thereby reducing the bias voltage and enhanching IPCE at low bias [19]. The photocurrent values at 0 V *vs*. RHE were 0.51, 7.36, 15.16 and 15.12 mA cm^{-2} for bare-$CuInS_2$, Pt-$CuInS_2$, Pt-In_2S_3/$CuInS_2$, and Pt-CdS/$CuInS_2$ respectively. The open circuit potential (V_{oc}) or onset potensial of the Pt-$CuInS_2$ electrode was estimated to be 0.40 V *vs*. RHE (Figure 3A). The highly negative V_{oc} suggests that there is a significant potential barrier that hinders electron transfer to the surface loaded Pt deposits. In order to improve the photoresponse of the Pt-$CuInS_2$ electrode, before Pt deposition an n-type thin layer of In_2S_3 was inserted by using a CBD technique to form Pt- and In_2S_3-modified electrode (Pt-In_2S_3/$CuInS_2$). As a result, a significant improvement of cathodic photocurrents was observed. Moreover, these modifications resulted in appreciable positive shifts of V_{oc}, i.e. *ca*. 0.82 V (*vs*. RHE) indicating considerable facilitation of efficient charge separation by introduction of the p-n junction. This is due to good transparancy of In_2S_3 over the solar spectrum [20] and less absorption of blue photons [21], and may be the formation of *p-n* homojunction $CuInS_2$. We also compared In_2S_3 to CdS-modified it was found that introduction of a thin layer of In_2S_3 as an alternative n-type layer resulted comparable photocurrent than that of CdS and seems both had reached saturation at -0.2 V *vs* RHE, although CdS-modified one had lower

V_{oc} than In_2S_3 namely 0.61 V. To estimate solar-to-hydrogen conversion efficiency (STH) we used the applied bias photon-to-current efficiency (ABPE) [22]. The Pt-In_2S_3/$CuInS_2$ electrode gave maximum ABPE (Figure 3B) of 1.9% at 0.27 V (*vs.* RHE) higher than CdS that of 1.6% at 0.18V). Through the surface modifications, both the cathodic photocurrent and the V_{oc} of In_2S_3-modified $CuInS_2$ were increased significantly 29 times and 0.4 V respectively. This increase may be caused by formation of a built-in potential (V_{bi}) in the photoelectrode and asymetric distribution of V_{bi} across the junction which separates photogenerated charge carriers [23].

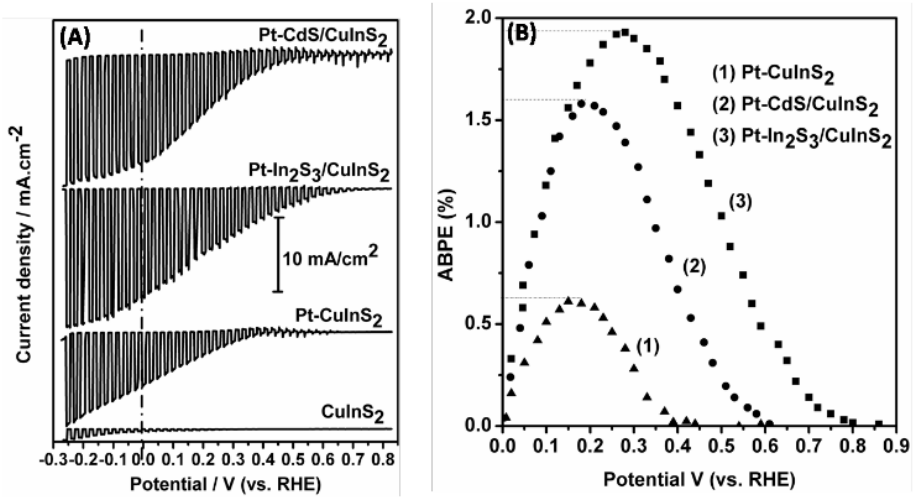

Figure 3. Current-potential curves (A) and APBE (B) of bare-$CuInS_2$, Pt-$CuInS_2$, Pt-In_2S_3/$CuInS_2$, and Pt-CdS/$CuInS_2$ electrodes in 0.1 M Na_2SO_4 at pH 9 under AM 1.5 G chopped Xe lamp illumination.

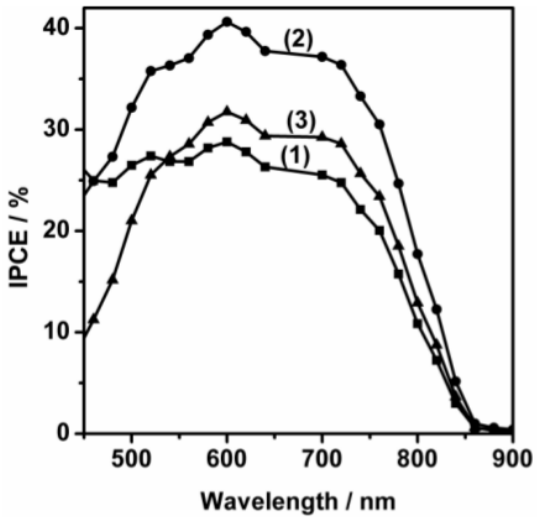

Figure 4. IPCE spectra of Pt-In_2S_3/$CuInS_2$ electrodes at In_2S_3 deposition of (1) 5, (2) 15 and (3) 30 min measured at 0 V (*vs.* RHE) in 0.1 M Na_2SO_4 at H 9.

For quantitative evaluation of photochatodes, the incident photon-to-current efficiency (IPCE) was measured (Figure 4). The measured IPCE values of Pt-In_2S_3/$CuInS_2$ photochatode at potentials of 0 V *vs* RHE. IPCE values were increased significantly under a negative electrode potential. All electrodes showed no photoresponse to the light with wavelength longer than 820 nm because the prepared $CuInS_2$ had a band gap around 1.5 eV, which corresponds to the onset of light absorption of 820 nm. The IPCE spectrum of Pt-In_2S_3/$CuInS_2$ at 15 min In_2S_3 deposition showed a rise at wavelengths ranging from 400 to 500 nm and it reached IPCE as high as 40.8% at wavelength 550-750 nm. Photocathode of Pt-In_2S_3/$CuInS_2$ obtained by In_2S_3 CBD for 15 min showed the higher IPCE than In_2S_3 deposited at 5 and 30 min at those wavelengths. However, at wavelength shorter than 560 nm (the absorption edge of In_2S_3) samples of 15 and 30 min CBD had much lower efficiency than that of 5 min. Therefore photon absorbed by In_2S_3 was forced to recombine [19].

Finally we examined the stability of Pt-In_2S_3/$CuInS_2$ and compared to Pt-$CuInS_2$ and Pt-CdS/$CuInS_2$ as can be seen in Figure 5 CdS modification tends to degrade, although it has the similar initial current density as In_2S_3. On the other hand In_2S_3 was a little bit stable for 1 h. And it seems it has similar trend as Pt-$CuInS_2$. However the protecting agent should be added to the surface to protect the degradation of the photocathode for longer examination.

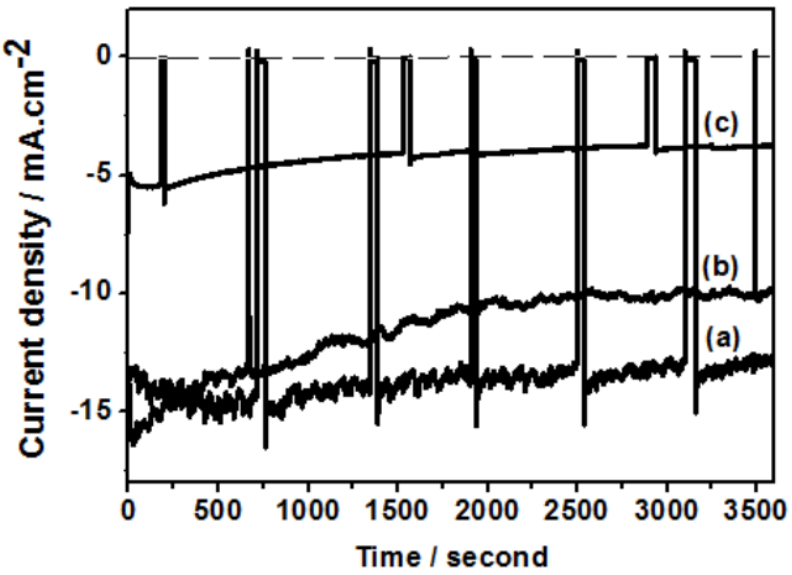

Figure 5. Current-time curves for (a) Pt- In_2S_3/$CuInS_2$, ((b) Pt-CdS/$CuInS_2$, (c) Pt-$CuInS_2$ electrodes in 0.1 M Na_2SO_4, pH 9 at 0 V *vs* RHE under Xe lamp illumination.

Figure 6 shows a time course curve of H_2 and O_2 evolution on Pt-In_2S_3/$CuInS_2$ electrode under 300 W Xe lamp illumination with applied bias of 0 vs RHE using three electrode system namely, photochatode, platinum wire and Ag/AgCl electrodes as working, counter and reference, respectively. Evolved H_2 and O_2 gases were close to stoichiometric ratio (2.2:1). Within 130 min was evolved 406.7 μmol H_2 and 185.1 μmol O_2 that exeeded the molar amount of $CuInS_2$ (ca. 1.6 μmol) these amount demonstrated the catalytic function of the electrode. The H_2 evolved was in agreement with with half of the electrons passing through the outer circuit ($e^-/2$), denoted by unfilled black square, showing that the efficiency of the water splitting is almost 100%. According to my knowledge the H_2 evolved at the same period gave the the highest result [9,23].

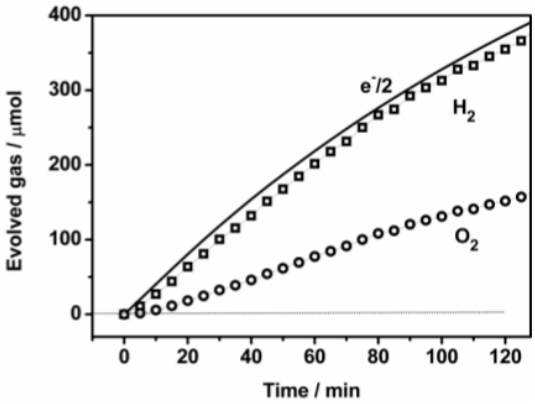

Figure 6. A time course curve of H_2 and O_2 evolution on Pt-In_2S_3/$CuInS_2$ electrode (0.7 cm^2) in 0.1 M Na_2SO_4, pH 9 at 0 V *vs* RHE under 300 W Xe lamp illumination. A black line denotes a time course of e^-_{half} during the H_2 evolution.

3. Conclusions

The formation of heterojunction by surface coverage of the $CuInS_2$ films with n-type buffer layers of In_2S_3 followed by loading of Pt deposits made them efficient H_2-evolving photoelectrodes. For the In_2S_3 modified sample, IPCE of more than 40.8% were obtained at wavelength ranging from 550 to 750 nm by applying a potential at 0 V vs RHE at pH 9. PEC water splitting under teresterial light was demonstrated using Pt-In_2S_3/$CuInS_2$ under external bias potential of 0 V vs RHE. Although the materials used in the present study were not examined for longer time with protecting agent to avoid photocathode corrosion. Thus, studies along these line are now in progress.

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