

# Platinum and indium sulfide- modified CuInS<sub>2</sub> as efficient photocathodes for photoelectrochemical water splitting

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# Platinum and indium sulfide-modified CuInS<sub>2</sub> as efficient photocathodes for photoelectrochemical water splitting†

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**A highly efficient CuInS<sub>2</sub>-based photocathode for water reduction with a maximum applied bias photon-to-current efficiency of ca. 2% was prepared by using a novel In<sub>2</sub>S<sub>3</sub> surface modifier.**

Hydrogen (H<sub>2</sub>) production by photoelectrochemical (PEC) water splitting is an attractive concept for attaining a sustainable energy source. Since the first report of PEC water splitting over a single crystalline TiO<sub>2</sub> photoelectrode,<sup>1</sup> various semiconductor electrodes have been investigated for this purpose.<sup>2</sup> Meanwhile, Cu-chalcopyrite p-type semiconductors such as CuInSe<sub>2</sub>, CuGaSe<sub>2</sub>, CuInS<sub>2</sub>, CuGaS<sub>2</sub> and their mixed crystals are widely used as photoabsorbers in thin film solar cells.<sup>3</sup> Due to their high absorption coefficients, tunable band gap values (1.0–2.4 eV) by changing In/Ga and/or Se/S ratios, and suitable band alignment for water reduction, these materials are also attractive for use as photocathodes for H<sub>2</sub> production from water.<sup>4</sup>

The insertion of n-type thin layers on photocathodes to improve their performance of PEC water splitting has been studied by several researchers using various p-type materials.<sup>4b–h,5</sup> This modification is widely employed to facilitate efficient charge separation<sup>2</sup> due to the modulated built-in field at the solid–solid interface by the formed p–n junction. For the Cu–chalcopyrite system, surface modification with an n-type CdS layer using a chemical bath deposition (CBD) technique was typically employed; this resulted in appreciable improvement of photocurrent as well as onset potential compared to those of unmodified films. However, due to the high toxicity of CdS, replacement with alternative

environment-friendly n-type layers would be advantageous for practical use.

In the present study, we investigated surface modification of a CuInS<sub>2</sub> thin film with a novel n-type surface modifier, In<sub>2</sub>S<sub>3</sub>, for PEC water splitting. In<sub>2</sub>S<sub>3</sub> possesses an indirect gap characteristic: it exhibits favorable transparency over the solar spectrum due to the weak absorption in the blue region, despite its relatively narrow band gap (2.1 eV) compared to that of CdS.<sup>6</sup> Herein we show that the In<sub>2</sub>S<sub>3</sub>-modified CuInS<sub>2</sub> thin film worked more efficiently than the CdS-modified one as a photocathode for PEC water splitting.

A CuInS<sub>2</sub> thin film was fabricated from electrochemically stacked layers of metallic Cu and In followed by sulfurization. The XRD pattern of the thus-obtained thin film showed typical diffraction peaks assignable to the CuInS<sub>2</sub> film with a chalcopyrite structure without any other phases (Fig. S1, ESI†). The film consists of angular-shaped grains with sizes ranging from 1.5 to 3 μm, as shown in a top-view FE-SEM image (Fig. 1a). When the CuInS<sub>2</sub> thin film was modified with a ca. 50 nm thick In<sub>2</sub>S<sub>3</sub> deposited by a chemical bath deposition method, the smooth surface of each CuInS<sub>2</sub> grain became granular (Fig. 1b). Appreciable lowering of the Cu/In atomic ratio after the In<sub>2</sub>S<sub>3</sub> deposition as confirmed by EDX analysis (Table S1, ESI†) also indicated the overall surface coverage of the CuInS<sub>2</sub> thin film by the In<sub>2</sub>S<sub>3</sub> layer. It should be noted that we could not exclude the possibility of the presence of In(OH)<sub>3</sub> and In<sub>2</sub>O<sub>3</sub> components in

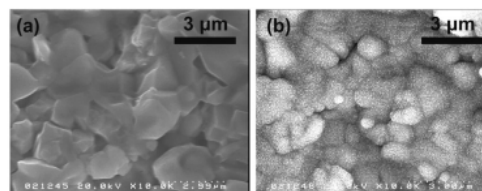


Fig. 1 Surface SEM images of (a) a CuInS<sub>2</sub> thin film and (b) that modified with In<sub>2</sub>S<sub>3</sub>.

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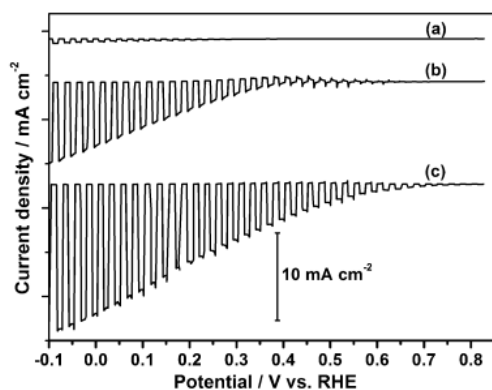


Fig. 2  $J$ - $V$  curves of (a) a  $\text{CuInS}_2$  thin film, (b)  $\text{Pt-CuInS}_2$ , and (c)  $\text{Pt-In}_2\text{S}_3/\text{CuInS}_2$  measured in  $0.1 \text{ M Na}_2\text{SO}_4$  (pH 9) under chopped illumination from AM 1.5 simulated solar irradiation.

the  $\text{In}_2\text{S}_3$  layer because of the observation of appreciably high EDX signals derived from oxygen on the  $\text{In}_2\text{S}_3$ -modified sample.

As reported previously,<sup>4c</sup> a bare  $\text{CuInS}_2$  thin film cannot work as a photocathode for PEC water splitting. As shown in Fig. 2a, a current density-potential ( $J$ - $V$ ) curve of a  $\text{CuInS}_2$  thin film measured in  $0.1 \text{ M Na}_2\text{SO}_4$  (pH 9) under chopped irradiation of AM 1.5 simulated solar light also exhibited almost no photoresponse. Surface modification of Pt deposits is a well-known technique to enhance  $\text{H}_2$  production from water. As expected, the  $\text{CuInS}_2$  thin film modified with Pt deposits ( $\text{Pt-CuInS}_2$ ) exhibited appreciable cathodic photocurrents. By applying the modification of Pt deposits to the  $\text{In}_2\text{S}_3$ -modified  $\text{CuInS}_2$  thin film, a significant increase of cathodic photocurrent was achieved. As shown in Fig. 2c, the  $\text{CuInS}_2$  thin film modified with both  $\text{In}_2\text{S}_3$  and Pt deposits ( $\text{Pt-In}_2\text{S}_3/\text{CuInS}_2$ ) showed higher photocurrent density than that of  $\text{CuInS}_2$  as well as a large shift of the photocurrent onset to the positive region. As illustrated in Fig. S3 (ESI<sup>†</sup>), the p-n junction of the  $\text{Pt-In}_2\text{S}_3/\text{CuInS}_2$  system resulted in the formation of higher built-in potential than that of the semiconductor-electrolyte one of the  $\text{Pt-CuInS}_2$  system.

For quantitative analyses, the applied bias photon-to-current efficiency (ABPE) of  $\text{Pt-In}_2\text{S}_3/\text{CuInS}_2$  was calculated by using the above  $J$ - $V$  curve with an assumption of 100% Faradic efficiency. As a result, the  $\text{Pt-In}_2\text{S}_3/\text{CuInS}_2$  electrode exhibited a photocurrent onset of  $0.73 \text{ V}$  (vs. RHE), and a maximum ABPE of 1.97% was achieved at  $0.28 \text{ V}$  (vs. RHE) (Fig. 3a). When the same analysis was performed for the  $\text{CuInS}_2$  thin film modified with both an n-type CdS layer and Pt deposits ( $\text{Pt-CdS/CuInS}_2$ ) (Fig. 3b), the photocurrent onset potential was estimated to be  $0.65 \text{ V}$  (vs. RHE), and a maximum ABPE of 1.63% at  $0.2 \text{ V}$  (vs. RHE) was obtained. Hence, it is obvious that the present  $\text{Pt-In}_2\text{S}_3/\text{CuInS}_2$  electrode was more advantageous than the  $\text{Pt-CdS/CuInS}_2$  electrode for use as a cathode for PEC water splitting. The results suggest that the  $\text{In}_2\text{S}_3$ - $\text{CuInS}_2$  interface forms a favorable p-n junction for suppressing recombination of photogenerated carriers at the heterointerface compared to that of the  $\text{CdS-CuInS}_2$  interface. A possible explanation is the

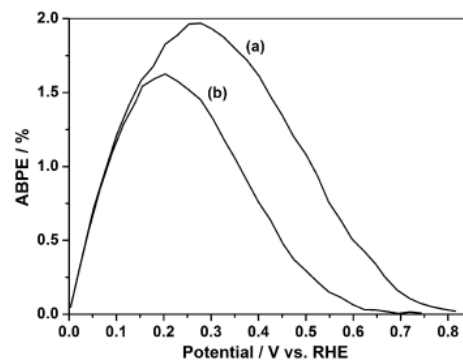


Fig. 3 ABPE curves of (a)  $\text{Pt-In}_2\text{S}_3/\text{CuInS}_2$  and (b)  $\text{Pt-CdS/CuInS}_2$  obtained from  $J$ - $V$  curves of these samples measured in  $0.1 \text{ M Na}_2\text{SO}_4$  (pH 9) under chopped illumination from AM 1.5 simulated solar irradiation.

homogeneous coverage of  $\text{CuInS}_2$  with  $\text{In}_2\text{S}_3$ , leading to efficient utilization of the heterointerface for charge separation. Another possibility is the limited interdiffusion of In and Cu from  $\text{In}_2\text{S}_3$  and  $\text{CuInS}_2$  layers to form a p-n homojunction; this should suppress interface recombination, resulting in higher attainable photovoltage compared to the use of the  $\text{CdS-CuInS}_2$  heterojunction. In order to clarify the interface structure, detailed structural characterization is now underway.

The incident photon-to-current conversion efficiency (IPCE) spectra of  $\text{Pt-In}_2\text{S}_3/\text{CuInS}_2$  and  $\text{Pt-CdS/CuInS}_2$  measured at (vs. RHE) showed the same onset at a photon energy of  $1.45 \text{ eV}$ , which is close to the band gap of  $\text{CuInS}_2$  ( $1.5 \text{ eV}$ )<sup>7</sup> (Fig. S2, ESI<sup>†</sup>). Both the spectra reached around 37–41% in the range of 520–700 nm and then dropped in the relatively large photon energy region. The drop was greater for the  $\text{Pt-CdS/CuInS}_2$  sample. Since this energy region corresponds to the band gap of bulk CdS ( $2.4 \text{ eV}$ ),<sup>8</sup> the observed drop in the spectra of  $\text{Pt-CdS/CuInS}_2$  should be caused by photon absorption of the top CdS layer. The drop was obviously suppressed for  $\text{Pt-In}_2\text{S}_3/\text{CuInS}_2$ , i.e., the sample has a higher IPCE gain at this high photon energy region than that of  $\text{Pt-CdS/CuInS}_2$ . As shown in Fig. S4 (ESI<sup>†</sup>), transmittance spectra of CdS and  $\text{In}_2\text{S}_3$  layers deposited on a fluorine doped tin oxide (FTO) glass substrate indicated appreciably higher transparency of the  $\text{In}_2\text{S}_3$  layer than that of the CdS layer at this energy region. The better transparency of  $\text{In}_2\text{S}_3$  led to the advantageous feature for PEC water splitting.

Fig. 4 shows time course curves of  $\text{H}_2$  and  $\text{O}_2$  evolution over the  $\text{Pt-CdS/CuInS}_2$  photocathode and the Pt counter electrode in the three-electrode system with an applied bias of  $0 \text{ V}$  (vs. RHE) under light irradiation from a 300 W xenon lamp. While a slight induction period was observed for the  $\text{O}_2$  time course curve,  $\text{H}_2$  evolution and  $\text{O}_2$  evolution were monotonically observed close to the stoichiometric ratio. A slight deviation of the amount of  $\text{H}_2$  evolved from half of the electrons passing through the external circuit ( $e^-/2$ , shown as a solid line in Fig. 4) is likely to be due to the occurrence of backward reaction, i.e., water formation from the evolved  $\text{H}_2$  and  $\text{O}_2$ . After 150 min photoirradiation,  $407 \mu\text{mol}$  of  $\text{H}_2$  gas and  $185 \mu\text{mol}$  of  $\text{O}_2$  gas

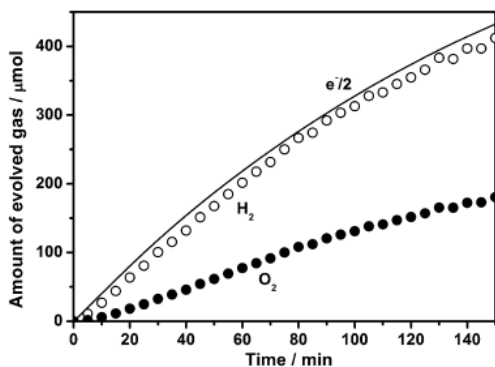


Fig. 4 Time course curves of gas evolution from the Pt-In<sub>2</sub>S<sub>3</sub>/CuInS<sub>2</sub> photoelectrode (H<sub>2</sub>: open circle) and the counter electrode (O<sub>2</sub>: closed circle) in a 0.1 M Na<sub>2</sub>SO<sub>4</sub> solution (pH 9) at 0 V (vs. RHE) under irradiation from a 300 W xenon lamp. The solid line denotes a time course curve of half of electrons passing through the outer circuit ( $e^-/2$ ).

were evolved: the amount of evolved H<sub>2</sub> significantly exceeded the molar amount of CuInS<sub>2</sub> deposited on the sample used with a total area of 0.7 cm<sup>2</sup>, indicating catalytic function of the electrode.

The formation of a p-n junction by surface modification of the CuInS<sub>2</sub> film with an n-type In<sub>2</sub>S<sub>3</sub> layer followed by loading of Pt deposits made it an efficient H<sub>2</sub>-evolving photoelectrode: a maximum ABPE of 1.97% at 0.28 V (vs. RHE) measured at 0.1 M Na<sub>2</sub>SO<sub>4</sub> (pH 9) under simulated AM 1.5 solar irradiation. To our knowledge, the observed PEC water splitting activity is the highest among chalcopyrite-based photocathodes reported so far.<sup>9</sup> This modification also effectively shifted the onset potential to a lower applied bias (ca. 0.78 V vs. RHE), which is promising for a tandem PEC system without any bias voltage. However, one of the critical issues encountered in the present system is stability, e.g., prolonged illumination in a 0.1 M Na<sub>2</sub>SO<sub>4</sub> solution (pH 9) at 0 V (vs. RHE) for 3 h resulted in 63% reduction of photocurrent from the initial amount (Fig. S5, ESI<sup>†</sup>). Thus, further studies to improve it are now in progress.

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