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$CuInS_2$ nanoparticles: Microwave-assisted synthesis, characterization, and photovoltaic measurements

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ABSTRACT

For the first time, (1,8-diamino-3,6-dioxaoctan)copper(II) sulfate, $[Cu(DADO)]SO_4$, and bis(propylenediamine)copper(II) sulfate, $[Cu(pn)_2]SO_4$, complexes as copper precursors have been used to prepare $CuInS_2$ (CIS) nanoparticles in the presence of microwave irradiation. $InCl_3$ anhydrous, thioacetamide (TAA), and propylene glycol were used as indium source, sulfur precursor, and solvent, respectively. Additionally, sodium dodecyl sulfate (SDS) was used as a capping agent. In this method, microwave irradiation created the activation energy for dissociating the precursors and led to the formation of $CuInS_2$ nanoparticles. The effect of preparation parameters such as microwave power, irradiation time, and type of copper precursor on the particle size of the products was studied. To fabricate a solar cell, CdS film was directly deposited on top of the CIS film through the chemical bath deposition method. The as-deposited $CdS/CuInS_2$ films were used for the photovoltaic measurements. According to *I–V* curves, it was found that the CIS nanoparticles synthesized by $[Cu(DADO)]SO_4$ complex as precursor was better for solar cell applications.

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1. Introduction

Multinary chalcogenides, $CuXY_2$ (X=In, Ga and Al, Y=Se and S), have been very attractive materials for applications of high efficiency thin film solar cells [1,2]. $CuInS_2$ as a semiconductor exhibited a direct band gap of about 1.52 eV, which was very close to the theoretical value of 1.5 eV, and an absorption coefficient of $10^5\ cm^{-1}$ in the visible spectrum range [3,4]. Up to now, different synthesis methods have been investigated for the preparation of $CuInS_2$ nanostructures. Solid state reaction [5], various solution-based routes such as hot injection methods [6], solvothermal and hydrothermal routes [7–10], and single-source precursor methods [11,12] have been applied to fabricate CIS nanostructures. In the recent years, colloidal synthesis routes by using suitable capping agents [13–15] have been heavily investigated. There are also several reports based on microwave heating for the preparation of $CuInS_2$ nanoparticles. In one method, CuO as copper precursor was used [16], and in another procedure, copper iodide as copper source was utilized [17]. Furthermore, the preparation of $CuInS_2$ thin films has attracted considerable attention as a suitable material to fabricate solar cells [18–20].

In the past two decades, the use of microwave energy to heat chemical reactions has attracted a considerable amount of attention due to its many successful applications in peptide, synthesis polymer chemistry, material science, nanotechnology and biochemical processes [21–26]. There are many advantages in the microwave-assisted approaches: the reaction time is very short, heat

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transformation is faster rather than that of reflux process in an oil bath and hydrothermal/solvothermal routes, local overheating can be avoided, and the reaction temperature is controllable.

In addition, metal-organic precursors and organometallic compounds have been widely used for the preparation of various nanostructures to control shape and size distribution, because they represent an important interface between synthetic chemistry and materials science. The available molecular precursors developed include metal acetylacetonate (acac) [27], metal cupferronate [28], metal benzenedicarboxylates [29–31], metal oxalates [32–34], metal dimethylglyoximate [35], metal format [36], metal carbonyl [37], metal 2-hydroxyacetophenato [38], and other new precursors [39–41]. In this work, a facile microwave-assisted process is introduced to fabricate $CuInS_2$ nanoparticles by using (1,8-diamino-3,6-dioxaoctan)copper(II) sulfate and bis(propylenediamine)copper(II) sulfate as copper precursors. Besides copper precursor, $InCl_3$ and thioacetamide were used as indium and sulfur sources, respectively. To further study, the effect of microwave power and irradiation time on the morphology of the products was investigated. To measure the photovoltaic properties of the as-produced $CuInS_2$ nanoparticles, CdS film was directly deposited on top of the CIS film through chemical bath deposition. The as-deposited $CdS/CuInS_2$ films were applied for the photovoltaic measurements.

2. Experimental

2.1. Materials

Propylenediamine, 1,8-diamino-3,6-dioxaoctan, $Cu(SO_4)_2 \cdot H_2O$, methanol, $InCl_3$, thioacetamide, sodium dodecyl sulfate (SDS), propylene glycol were purchased from Merck Company with analytical grade.

Table 1
Preparation conditions of samples A1–A15 and samples B1–B6.

Sample no.	Microwave power (W)	Irradiation time (min)	Particle size (nm)
A1	900	12	20–22
A2	900	10	~20
A3	900	8	18–20
A4	900	6	15–20
A5	900	4	15–17
A6	750	12	23–25
A7	750	10	20–25
A8	750	8	~18
A9	750	6	18–20
A10	750	4	13–15
A11	600	12	20–22
A12	600	10	18–22
A13	600	8	14–16
A14	600	6	~15
A15	600	4	12–14
B1	900	12	85–90
B2	900	10	28–30
B3	900	6	~20
B4	750	12	43–45
B5	750	10	20–22
B6	750	6	~10

Precursors of series A and B were $[Cu(DADO)]SO_4$ and $[Cu(pn)_2]SO_4$, respectively.

2.2. Characterization

The reagents of experiments were exposed to microwave irradiation in a home microwave oven (DG68-00025A, SAMSUNG). Fourier transform infrared (FT-IR) spectra were recorded on a Magna-IR, spectrometer 550 Nicolet in KBr pellets in the range 400–4000 cm^{-1}. The XRD patterns were collected from a diffractometer of Philips Company with X'PertPro monochromatized

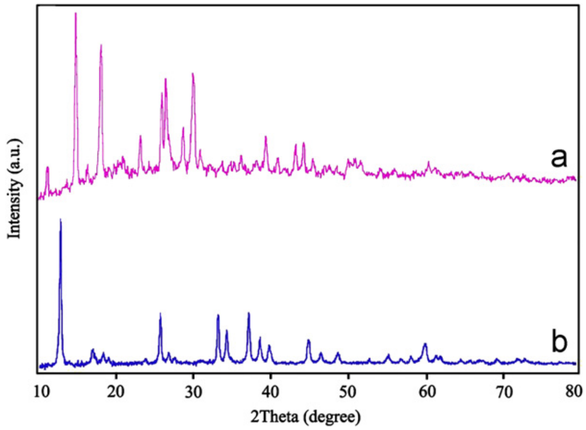

Fig. 1. XRD patterns of $[Cu(pn)_2]SO_4$ (a) and $[Cu(DADO)]SO_4$ (b).

Table 2
XRD data of $[Cu(DADO)]SO_4$ and $[Cu(pn)_2]SO_4$ precursors.

$[Cu(pn)_2]SO_4$			$[Cu(DADO)]SO_4$		
No.	Position (2θ)	FWHM (2θ)	No.	Position (2θ)	FWHM (2θ)
1	16.2192	0.1771	1	12.9325	0.2362
2	18.5164	0.5904	2	17.0803	0.2362
3	19.6555	0.2362	3	18.4406	0.2952
4	21.0357	0.1771	4	19.1867	0.1771
5	22.6884	0.2952	5	23.8603	0.3542
6	25.4043	0.2362	6	25.9092	0.2362
7	27.4209	0.2362	7	26.9887	0.2362
8	30.0096	0.1771	8	27.7822	0.2952
9	30.5175	0.2362	9	33.3415	0.2066
10	32.5795	0.1771	10	34.4953	0.2066
11	33.8146	0.2952	11	37.2879	0.2362
12	34.6421	0.2952	12	38.7550	0.2066
13	37.1854	0.4723	13	40.0464	0.3542
14	39.5524	0.2362	14	45.0589	0.3247
15	41.3518	0.5904	15	46.6772	0.2362
16	42.5611	0.2952	16	48.8915	0.2952
17	43.9791	0.2952	17	52.9791	0.2362
18	46.1433	0.2362	18	55.4346	0.3542
19	47.0995	0.1771	19	57.0889	0.3542
20	48.2169	0.3542	20	58.3445	0.2362
21	52.3936	0.3542	21	60.2257	0.5314
22	53.9732	0.4133	22	61.5158	0.2362
23	56.3365	0.3542	23	62.1177	0.2362
24	62.0936	0.4320	24	64.7982	0.4723
			25	65.9939	0.3542
			26	67.3800	0.7085
			27	69.5214	0.4723
			28	72.2102	0.4320

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