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# The annealing effect on structural, optical and photoelectrical properties of $CuInS_2/In_2S_3$ films

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#### 1. Introduction

CuInS<sub>2</sub> can be used to reach high conversion efficiencies due to its large absorption coefficient and direct band gap of 1.5 eV, which means it absorbs most of the solar spectrum [1]. Various techniques have been developed for the preparation of CuInS<sub>2</sub> films such as elemental chemical vapor deposition [2], sulfurization of metallic precursor [3], spray pyrolysis [4], electro-deposition [5], co-evaporation [6], chemical bath deposition (CBD) [7] and chemical route [8]. CuInS<sub>2</sub>/In<sub>2</sub>S<sub>3</sub> thin films are used for photovoltaic applications. Most of the efficient CuInS<sub>2</sub>-based solar cells were prepared using CdS as the buffer layer [9-13]. Best efficiency has been found to be 12.5% (active area efficiency) for the CuInS<sub>2</sub>/CdS/ZnO cell configuration with MgF<sub>2</sub> antireflection coating reported by Klaery et al. [14]. Recently, many researchers have focused on the effective replacement of CdS. The motivation behind this is not only to eliminate toxic cadmium but also, to improve light transmission in the blue wavelength region by using a material having band gap wider than that of CdS. One such possible replacement of CdS is In<sub>2</sub>S<sub>3</sub>. In<sub>2</sub>S<sub>3</sub> thin films are the effective materials as a window layer for the solar cell structures, because of its a wide and direct band-gap. The wide band gap (> 2.5 eV) of  $In_2S_3$  thin films suggests that it can act as a better buffer layer than CdS having a band gap of 2.4 eV [15]. Conversion efficiency has been achieved up to 15.7% with cadmium-free  $In_x(OH,S)_v$  as buffer layer for a Cu(In,Ga)Se<sub>2</sub>-based solar cell [16].

#### ABSTRACT

Successive lonic Layer Adsorption and Reaction (SILAR) technique was used to deposit the CuInS<sub>2</sub>/In<sub>2</sub>S<sub>3</sub> multilayer thin film structure at room temperature. The as-deposited film was annealed at 100, 200, 300, 400 and 500 °C for 30 min in nitrogen atmosphere and the annealing effect on structural, optical and photoelectrical properties of the film was investigated. X-ray diffraction (XRD) and optical absorption spectroscopy were used for structural and optical studies. Current–Voltage (*I–V*) measurements were performed in dark environment and under 15, 30 and 50 mW/cm<sup>2</sup> light intensity to investigate the photosensitivity of the structure. Also, the electrical resistivity of the film was determined in the temperature range of 300–470 K. It was found that annealing temperature drastically affects the structural, optical and photoelectrical properties of the CuInS<sub>2</sub>/In<sub>2</sub>S<sub>3</sub> films.

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For CuInS<sub>2</sub>, with the same buffer layer, efficiency has been achieved up to 11.4% [17]. Other materials such as ZnO, ZnSe, Zn (Se,O), In<sub>2</sub>S<sub>3</sub> and InSe were also used as alternatives to CdS with considerable conversion efficiencies [18–22].

The aim of this study is to examine and the effect of annealing on structural, optical and photoelectrical properties of  $CuInS_2/In_2S_3$  films and improve the crystal quality of  $CuInS_2$ . The results of the experiments support this aim. In the present paper, the  $CuInS_2$  thin film used for the photovoltaic applications was grown by the SILAR method. In order to improve the crystal quality of  $CuInS_2$ , it was annealed at different temperatures and grown onto the  $In_2S_3$  crystal film instead of amorphous glass substrate. Since indium was evaporated from the films as a result of annealing, to support decrease in the amount of indium,  $CuInS_2$  film was grown on the  $In_2S_3$  films. Besides, there is a lattice match between  $CuInS_2$  and  $In_2S_3$ , so  $CuInS_2$  films have a better crystal quality than those grown on other substrates. Its structural, optical and photoelectrical properties were investigated as a function of annealing temperature.

#### 2. Experimental details

Semiconductor thin films can be fabricated by several methods used both in gas and in liquid phases. In comparison with gas phase methods, liquid phase methods have some advantages of providing thin films for large area at low cost but, they lead to imprecise control of the crystal film structure. One of the liquid phase methods for deposition of thin films is the SILAR method. The SILAR method was developed by Nicolau [23] for the



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Fig. 1. XRD patterns of as-deposited thin films annealed at 100, 200, 300, 400 and 500  $^\circ$ C.

deposition of zinc and cadmium chalcogenides thin films 25 years ago. In this method, thin films are fabricated by dipping into two different solutions of each precursor ion for compound semiconductors. Therefore, the control of the film thickness becomes feasible by changing the number of the dipping cycles. Furthermore, atomically controlled multilayer thin films or super lattices can possibly be fabricated by changing precursor solutions. Since the SILAR method depends only on immersing the substrate into the solutions, the deposition of films with a large area can be achieved at low cost.

The glass substrate materials were cleaned with acetone and water-ethanol (50:50) solution in an ultrasonic bath and dried with nitrogen. For the deposition of the In<sub>2</sub>S<sub>3</sub> thin film. 0.1 M indium trichloride (InCl<sub>3</sub>) solution ( $pH \sim 5.5$ ) was used as the cationic precursor and 0.05 M Na<sub>2</sub>S solution (pH~11.5) as the anionic precursor. The well-cleaned glass substrate was immersed in the cationic precursor solution (InCl<sub>3</sub>) for 30 s in order to adsorb the indium ions on the surface of glass substrate. Then, the substrate was rinsed doubly in distilled water for 50 s to prevent homogenous precipitation. It was followed by immersion in the anionic precursor solution  $Na_2S$  (pH $\sim$ 12) for 30 s. Sulfide ions react with the adsorbed indium ions on the glass substrate resulting in the formation of In<sub>2</sub>S<sub>3</sub> layer. The substrate was rinsed doubly in distilled water for 50 s. This was the last step for one SILAR cycle. The thicknesses of the films were calculated within the limit of experimental errors by using the optical interference method [24]. It was seen that the thicknesses are increasing with number of SILAR deposition cycles. It was found that the optimum SILAR cycle is 75 from the absorbance and XRD measurements. Thus, SILAR cycle was repeated 75 times and In<sub>2</sub>S<sub>3</sub> layer film was grown at room temperature (27 °C). The thickness of the films has been calculated as 330 + 14 nm. After then, the CuInS<sub>2</sub> film was grown onto the  $In_2S_3$  film, CuCl<sub>2</sub> · 2H<sub>2</sub>O, InCl<sub>3</sub> and Na<sub>2</sub>S were used in the deposition of the CuInS<sub>2</sub> thin film. The mixed cationic precursor solutions consist of cupric chloride and indium chloride as the reagents, whose pH value was adjusted to 5.5 by using hydrochloric acid. The source of sulfur ions was 0.05 mol/L Na<sub>2</sub>S (pH $\sim$ 12). The prepared solutions were percolated and taken into 100 mL beakers before deposition.

The deposition was carried out at room temperature (27  $^{\circ}$ C). There are four steps in one cycle dipping:

- (1) The substrate was immersed in the mixed cation precursors for 30 s.
- (2) The substrate was rinsed with doubly distilled water for 50 s to remove the unattached ions.
- (3) The substrate was immersed in 0.05 mol/L sodium sulfide solution for 30 s.
- (4) The substrate was rinsed with doubly distilled water for 50 s to remove the unreacted ions.

The adsorbed  $Cu^{2+}$  and  $In^{3+}$  ions on the substrate react with the  $S^{2-}$  ions in anion precursor solution to form the sulfide compound of CuS and  $In_2S_3$  according to Eqs. (1) and (2). By repeating such SILAR deposition cycle 75 times,  $CulnS_2/In_2S_3$  thin film was grown. The reaction for the formation of stoichiometry  $CulnS_2$  is given in Eq. (3).

 $CuCl_2 + Na_2S \rightarrow CuS + 2NaCl$ (1)

 $2InCl_3 + 3Na_2S \rightarrow In_2S_3 + 6NaCl \tag{2}$ 

$$2CuS + In_2S_3 \rightarrow 2CuInS_2 + S \tag{3}$$

The as-deposit film was annealed at 100, 200, 300, 400 and 500 °C for 30 min in Nitrogen atmosphere. For structural studies, a Rigaku 2200D/Max, X-ray diffractometer of Cu K $\alpha$  ( $\lambda$ =1.5405 Å) radiation with 2 $\theta$  of 20–70° was used. The band gap value of the

films was found via the absorption measurements using a PerkinElmer UV/VS Lambda 2S Spectrometer with a wavelength resolution better than  $\pm$  3 nm at room temperature. All electrical measurements were performed with two steps. In the first step, in order to investigate photoelectrical properties of CuInS<sub>2</sub>/In<sub>2</sub>S<sub>3</sub>, I-V measurements were done in dark environment and under 15, 30 and 50 mW/cm<sup>2</sup> light intensity. CuInS<sub>2</sub>/In<sub>2</sub>S<sub>3</sub> thin films were illuminated by different three tungsten lamps (Philips 230 V, 150 W, 300 W and 500 W). To prevent heating of samples from tungsten lamp, digital temperature controller (Lake Shore) based on PT-100 sensor of accuracy  $\pm 1$  °C was used and the temperature of sample was set to room temperature. Secondly, the electrical resistivity  $(\rho)$  of the films deposited with different annealing temperatures has been measured in the temperature range 300–470 K by the two dc probe method. All I-V measurements were performed by the Keithley 2400 source meter and Keithley 487 Pico-ammeter.

#### 3. Result and discussion

#### 3.1. XRD analysis

Fig. 1 provides the comparison of typical XRD spectra for CuInS<sub>2</sub> thin films annealed at different temperatures. As shown in Fig. 1(a) and (b), both as-deposited and annealed films at 100  $^{\circ}$ C

#### Table 1

d values and (h k l) miller indices of annealed CulnS<sub>2</sub>/In<sub>2</sub>S<sub>3</sub> thin films for different annealing temperatures.

Annealing temperature (°C)	$2\theta$ (deg.) Observed	d (Å) Observed	d (Å) Standard	hkl
200	27.81	3.205	3.196	112 (CuInS <sub>2</sub> )
300	27.81	3.205	3.196	112 (CuInS <sub>2</sub> )
	32.57	2.747	2.762	200 (CuInS <sub>2</sub> )
	48.25	1.885	1.905	440 (In <sub>2</sub> S <sub>3</sub> )
400	27.81	3.205	3.196	112 (CuInS <sub>2</sub> )
	32.57	2.747	2.762	200 (CuInS <sub>2</sub> )
	48.25	1.885	1.905	440 (In <sub>2</sub> S <sub>3</sub> )
500	38.41	2.338	2.308	025 (CuS)
	44.61	2.029	2.029	110 (CuS)



Fig. 2. Grain size values versus annealing temperature for annealed  $\mbox{Culn}S_2/\mbox{ In}_2S_3$  films.

have an amorphous structure. As the film annealed at 200 °C, the found peak is reported as the characteristic peak for CuInS<sub>2</sub> with the preferred orientation of (1 1 2) at  $2\theta = 27.81^{\circ}$  as shown in Fig. 1(c). By increasing the annealing temperature from 200 to 300 and 400 °C, two additional peaks at  $2\theta = 32.57^{\circ}$  and  $48.18^{\circ}$  for CuInS<sub>2</sub> (200),  $In_2S_3$  (4 4 0) reflections are clearly observed and the intensity for each peak also increases, respectively. These results agree with the literature [4,8,25] and for [CPDS no. [27-0159, 65-0459, 65-7111] (see Fig. 1(d) and (e)). (1 1 2) and (2 0 0) peaks of CuInS<sub>2</sub> and (4 4 0)peak of In<sub>2</sub>S<sub>3</sub> completely disappeared after annealing at 500 °C and instead of these, (025), (110) peaks of CuS appeared. It is shown that multilaver structure film converts CuInS<sub>2</sub>/In<sub>2</sub>S<sub>3</sub> to CuS film due to the high annealing temperature. This result can be interpreted as follows: as the CuInS<sub>2</sub>/In<sub>2</sub>S<sub>3</sub> thin film annealed at 500 °C, because the vapor pressure of indium is lower than that of copper, indium is evaporated from the substrate and the excessive copper occurs in the CuInS<sub>2</sub>/In<sub>2</sub>S<sub>3</sub> film. Thus, as shown in Fig. 1(f), the structure is converted from CuInS<sub>2</sub>/In<sub>2</sub>S<sub>3</sub> to CuS. This result is supported by the higher mobility of copper ions and CuS segregation [26,27].

All the results indicate that the crystallinity of these thin films improved with increase in annealing temperature. The lattice parameters of the films are given in Table 1 for  $CuInS_2$ ,  $In_2S_3$  and

CuS thin films. Since  $CuInS_2$ ,  $In_2S_3$  and CuS thin films have some defects, such as crystallite boundary discontinuities, lattice mismatch and strain effects; the observed 'd' values are a little different from standard 'd' values as seen in Table 1.

The crystallite size of thin films is calculated using Scherer's formula:

$$D = K\lambda/\beta\cos\theta \tag{4}$$

where *D* is the crystallite size,  $\lambda$  is the X-ray wavelength used,  $\beta$  is the angular line width of half maximum intensity,  $\theta$  is Bragg's

#### Table 2

Annealing temperature and optical band gap values for annealed  $\text{CuInS}_2/\text{In}_2\text{S}_3$  thin films.

Annealing temperature (°C)	Band gap energy (eV)	
200	2.48	
300	2.49	
400	2.51	
500	2.69	



Fig. 3. Variation of absorbance with wavelength and graphs of  $\alpha^2$  versus energy for thin films annealed at 200, 300 400 and 500 °C.

diffraction angle and *K* is a constant. The average crystallite size is calculated by resolving the greatest peak intensity. As seen in Fig. 1(f), the (1 1 0) peak of CuS is very strong at the annealing temperature of 500 °C. As can be seen in Fig. 2, the grain size increases with increase in annealing temperature thus, crystallization of films improves; this result agrees with the XRD pattern. The grain size increases from 25.32 to 52.48 nm with the increase in annealing temperature.

#### 3.2. Optical studies

The band gap energy of the films is calculated before and after each annealing experiment with the help of absorption spectra. First absorption data are converted into the absorption coefficient as reviewed in our earlier reports [24]. The band-gap calculation was done via the  $(\alpha)^2$  versus  $(\hbar\omega)$  plot, where  $\alpha$  is the absorption coefficient and  $\hbar\omega$  is the photon energy. The absorption coefficient  $\alpha$  is proportional to:

Instead of 
$$\alpha(\hbar\omega) = B(\hbar\omega - E_{\sigma})^n$$
 (5)

where *B* is a constant and *n* is an index (n=1,2,3,...).

These plots  $[(\alpha)^2$  versus  $(\hbar\omega)]$  have been given in Fig. 3 and the band-gap energies of thin films were determined by the extrapolation of the linear regions on the energy axis. Absorption edge is seen to be shifted to shorter wavelengths for annealed films as shown in Fig. 3, which is a result of the conversion from CuInS<sub>2</sub> to CuS. As seen in Fig. 3, the band gap has been found to be 2.48 eV for the thin film annealed at 200 °C and a correlation has been found between annealing and band-gap such that increase in annealing temperature results in an increase in the band-gap up to 2.69 eV, which is the bandgap energy of CuS film (see Table 2). Both absorption and XRD findings showed that the annealing at 500 °C of the grown multilayer film structure converts the structure to CuS film. The band gap energy values for 200, 300 and 400 °C annealing temperatures belong to CuInS<sub>2</sub> whereas, annealing at 500 °C lead to obtain the band gap energy of CuS. Because the band gap energy value of  $In_2S_3$  thin film (2.9 eV, [28–30]) is greater than that of CuInS<sub>2</sub>, the incident light is absorbed by the CuInS<sub>2</sub> film. Consequently, the band gap value of CuInS<sub>2</sub> can be found via the optical absorption method. It is mentioned previously that, because the vapor pressure of indium is lower than that of



Fig. 4. Photocurrent versus light intensity for annealed CuInS<sub>2</sub>/In<sub>2</sub>S<sub>3</sub> films.

copper, copper stays inside the structure, while indium evaporates as a result of annealing process in the film that leads to an increase in the band gap energy value. These values are greater than the reported values of 1.5 eV for single CuInS<sub>2</sub> crystals. This can be interpreted either by poor crystallinity or by deviations from stoichiometry that give rise to defect states, which increase the band gap energy. As seen in XRD measurements (Fig. 1), the structure of the films is converted from CuInS<sub>2</sub>/In<sub>2</sub>S<sub>3</sub> to CuS when the film is annealed at 500 °C. Consequently, energy value of band gap (2.69 eV) is in good agreement with the CuS thin film prepared by chemical bath deposition (CBD) and SILAR [31–33].

#### 3.3. Electrical properties

#### 3.3.1. Photoconductive properties of $CuInS_2/In_2S_3$ thin films

Various crystallinity imperfections in the film, such as vacancies, dislocations and grain boundaries act as trapping or recombination centers of the carriers and play an important role in photoconduction. These traps act as localized positive potential centers for electrons and negative potential centers for holes.



Fig. 6. In Photocurrent versus annealing temperature for annealed CuInS<sub>2</sub>/In<sub>2</sub>S<sub>3</sub> films.



Fig. 5. Photocurrent versus voltage for annealed CuInS<sub>2</sub>/In<sub>2</sub>S<sub>3</sub> films.



Fig. 7. (a) Photosensitivity versus light intensity and (b) Photosensitivity versus annealing temperature for annealed CuInS2/In2S3 films.



**Fig. 8.**  $\ln \rho$  versus 1/T (K) for annealed CuInS<sub>2</sub>/In<sub>2</sub>S<sub>3</sub> films.

Therefore, some localized discrete energy levels are formed in the band gap, in the vicinity of the conduction and valence bands. Fig. 4 shows the variation of photocurrent with light intensity in CuInS<sub>2</sub>/In<sub>2</sub>S<sub>3</sub> film for different annealing temperatures. The variation of photocurrent with applied voltage in CuInS<sub>2</sub>/In<sub>2</sub>S<sub>3</sub> films is shown in Fig. 5. It is clear that the photocurrent increases with increase in voltage. The photocurrent is found to increase with higher annealing temperature and light intensity. As annealing temperature of the film increases, the crystallinity grain size increases and this helps in the improvement of photocurrent. The increase in photocurrent is attributed to an increase in the majority carrier concentration and/or an increase in impurity centers acting as traps for minority carriers as a result of annealing. Especially at 400 °C annealing temperature, this increase in photocurrent compared with other samples is much more. This result is supported by Fig. 6. In this figure, logarithmic photocurrent versus annealing temperature is shown, in order to show the photocurrent of all samples in the same graph.

Photosensitivity is the ratio of the increase in conductivity of the material in the presence of light to the conductivity in darkness and is given by the following relation [34]:

$$S = (I_{\rm photo} - I_{\rm dark})/I_{\rm dark}$$
(6)

where  $I_{\rm photo}$  and  $I_{\rm dark}$  are the currents, measured under illumination and in the dark, respectively. Fig. 7(a) and (b) shows the plot of photosensitivity versus light intensity and photosensitivity versus annealing temperature, respectively. As can be seen in Fig. 7, the value of photosensitivity has a maximum at the 400 °C annealing temperature. As a result of annealing at 400 °C, some transitions which create additional free carriers effectively increase the free life time, which leads to an increase in photosensitivity of the material. This result is supported by Figs. 5 and 6. As a result, it can be concluded that the optimum annealing

#### Table 3

Resistivity values of annealed  $\mbox{Culn}S_2/\mbox{In}_2S_3$  thin films for different annealing temperatures.

Resistivity ( $\Omega$ cm)	
2322.26	
214.69	
91.06	
118.10	



Fig. 9. Variation of resistivity as a function of annealing temperature.

temperature for the  $CuInS_2/In_2S_3$  thin films is 400 °C. These results agree with the literature [35,36].

#### 3.3.2. Electrical resistivity

The electrical resistivity ( $\rho$ ) of the films deposited with different annealing temperatures has been measured in the temperature range 300–470 K by the two dc probe method.

Plots of  $\ln(\rho)$  versus 1/T for different annealing temperatures are shown in Fig. 8. From the slopes of these plots, the values of activation energies for low and high temperature linear regions are calculated. It is observed that the resistivity changes nonlinearly with the increase in temperature, which is a semiconducting behavior. Formation of the film with discontinuous clusters and ionizing impurity levels are the two reasons of this non-linear behavior. The electrical resistivity values, which are calculated at 300 K are given in Table 3. There is a huge decrease in the resistivity values as the annealing temperature increases from 200 to 500 °C (Table 3 and Fig. 9). The predicted reasons for high resistivity are: nanocrystalline nature, crystallite boundary discontinuities and presence of defect states. The decrease in resistivity may be attributed to the presence of high conductivity CuS phase segregation in the grain boundaries (from XRD results) and high mobility of copper ions as mentioned above [26,27,37].

#### 4. Conclusion

CuInS<sub>2</sub>/In<sub>2</sub>S<sub>3</sub> thin films were prepared on glass substrate by Successive Ionic Layer Adsorption and Reaction method and annealed at 100, 200, 300, 400 and 500 °C for 30 min. It is found that the annealing temperature is crucial for structural, optical and photoelectrical properties. As the annealing temperature increases from 200 to 400 °C, the CuInS<sub>2</sub>/In<sub>2</sub>S<sub>3</sub> films show a wide range of resistivity decrease from 2322.26 to 91.06  $\Omega$  cm. Photosensitivity has maximum value and that of resistivity hass minimum value at the 400 °C annealing temperature. Also, from the XRD measurements, the best quality crystallinity is shown at 400 °C annealing temperature for CuInS<sub>2</sub>/In<sub>2</sub>S<sub>3</sub> films. When the annealing temperature is 500 °C, the structure of film is converted from CuInS<sub>2</sub>/In<sub>2</sub>S<sub>3</sub> to CuS. After all these results, it can be concluded that the annealing temperature has an important role in CuInS<sub>2</sub>/In<sub>2</sub>S<sub>3</sub> multilayer films' optical, structural and electrical properties. Furthermore, 400 °C is found to be the optimum annealing temperature for these properties.

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