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**CADMIUM SULFIDE COPPER TERNARY HETEROJUNCTION
CELL RESEARCH**

Annual Report for September 30, 1977—September 30, 1978

By
D. L. Fleming

October 1978

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Sperry Univac
St. Paul, Minnesota

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Solar Energy

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Cadmium Sulfide Copper Ternary Heterojunction Cell Research

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D. L. Fleming
Principal Investigator

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1.0 Abstract

Work on the first year of a project to develop the technology to produce efficient, large area CdS/CuInSe₂ heterojunction solar cells is described.

Two methods of fabricating the CuInSe₂ were investigated; flash evaporation and dual source, CuInSe₂ and Se, coevaporation. Data on film structure using x-ray diffraction, film stoichiometry using x-ray fluorescence, and grain size measurements using transmission electron microscopy are presented for each method of fabrication.

Preliminary 0.45% efficient CdS/CuInSe₂ devices are described.

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4.0 Discussion

4.1 Introduction

A number of copper ternary semiconductors have been investigated and their properties reported in the literature. A review of much of this work is given in Shay & Wernick, "Ternary Chalcopyrite Semiconductors/Growth, Electronic Properties and Applications," Pergamon Press, 1975. Of these, CuInSe_2 is the preeminent candidate for a heterojunction cell with CdS. Preliminary investigations of this system, both single-crystal^{1, 2, 3} and polycrystalline^{4, 5} have been encouraging. CuInSe_2 has a band gap of 1.0 eV which is within the range of 1-2 eV expected to give best efficiency⁶ in solar photovoltaic devices. CuInSe_2 has a smaller lattice mismatch with CdS (~1%) than any other copper ternary. This leads to a smaller defect density at the junction, which in turn reduces the junction current and makes the external current larger, o.e.p. Single crystal $\text{CuInSe}_2/\text{CdS}$ cells have achieved 12% efficiency⁷, and polycrystalline cells have achieved an efficiency of 6.7%⁶. CuInS_2 , the only other copper ternary to have been used in a polycrystalline device with CdS, has achieved an efficiency of only 2.9%.

In order to achieve the goal of developing a Cadmium Sulfide/Copper Ternary heterojunction solar cell which can be reproducibly manufactured with an air mass-1 conversion efficiency of no less than 8% (as measured between 20°C and 30°C over cell areas of at least 4 cm²), with a maximum electrical output degradation of 5% over 20 years, this research program has been undertaken. The program involves physical vapor deposition of CdS and CuInSe_2 films and fabrication of devices. The dependence of material parameters on

deposition conditions is being investigated along with the effect of fabrication procedures on device efficiency and stability so as to establish a technology capable of producing cells meeting the above goal.

The work by L.L. Kazmerski⁸, on CuInSe_2 cells has revealed a number of problems associated with this system and has indicated some avenues of approach. The most important problem to be confronted is the control of the stoichiometry of the ternary CuInSe_2 film over larger areas. Other potential problems include shorting of junction through pin holes, the diffusion of Cd in the CuInSe_2 and obtaining low resistivity CdS on the CuInSe_2 film.

Several methods of deposition, p type CuInSe_2 films are being investigated. Principally these methods are the flash evaporation of p-type (Se rich) CuInSe_2 powder and a dual source, CuInSe_2 and Se, coevaporation process utilizing Electron Impact Emission Spectroscopy (EIES) to monitor and control the deposition process. Flash evaporation is an attractive process for depositing compound semiconductors. By keeping the source sufficiently hot, any tendency for the CuInSe_2 to dissociate can be ameliorated. It is a simple procedure having few process parameters to control. The deposition of p type CuInSe_2 films using flash evaporation has been reported.⁵ The dual source deposition of CuInSe_2 for photovoltaic applications is a proven technique. Utilizing EIES to monitor and control the deposition should provide the process control required to turn it in to a reliable manufacturing process.

4.2 CuInSe₂ Preparation

For both the flash evaporation and the dual source coevaporation process, CuInSe₂ is a required source material. The synthesis of the starting CuInSe₂ powder has been an ongoing process. Stoichiometric (or nearly stoichiometric, for flash evaporation a .1% or 1% excess of Se was used) amounts of Cu, In, and Se are sealed in an evacuated quartz tube. The tube was heated inside a second evacuated tube to approximately 1000°C for nominally a day.

Upon cooling the CuInSe₂ is crushed and examined for extraneous phases using x-ray diffraction. Due to the high temperature used to form the CuInSe₂, other phases have not been observed. A Cu K α diffraction pattern is shown indexed in Figure 1. X-ray fluorescence pattern have also been taken of the stoichiometric powders as a reference standard to judge deposited films with. Such a fluorescence pattern is shown in Figure 2 with the characteristic In, Cu and Se peaks.

4.3 Flash Evaporated CuInSe₂

CdS/CuInSe₂ devices made in a single pump down should minimize the inclusion of contaminants in the junction. A flash evaporation system having a second source for CdS deposition was set up to accomplish this. The tooling for the system is shown in Figure 3. CuInSe₂ powder is dropped from a hopper into a resistance heated, ceramic coated, tungsten crucible. A second ceramic coated tungsten crucible with a baffled Mo cap is used to deposit the CdS films. Quartz lamps, controlled by a variac are used as substrate heaters with the temperature being monitored by a thermocouple mounted in

Figure 1.

X-RAY DIFFRACTION PATTERN CuInSe_2 POWDER

XRAY FLUORESCENCE OF CuInSe_2 POWDER

Figure 2.

FLASH EVAPORATION SYSTEM

Figure 3.

the substrate holder backing plate. The substrate holder is fitted with a moveable mask to allow the deposition of CuInSe_2 films at one end of 1"x3" substrates, CdS at the other ends, and heterojunctions in the middle.

Figure 4 shows the typical devices and associated films produced in a run. A dog bone probe, a van der Pauw probe, and a gap in a metal film bridged by the semiconductor films are seen at each end. Approximately 1 inch square devices are formed in the center. Typical film thicknesses are 1μ for the CuInSe_2 and 10μ for the CdS films. A series of cells and/or CuInSe_2 films were deposited at substrate temperatures from 50°C to 640°C . CdS depositions were made at a substrate temperature of 200°C . No photovoltaic effect was observed for these junctions. The CuInSe_2 films have been examined using x-ray diffraction, electron diffraction, x-ray fluorescence, transmission and scanning electron microscopy, and optical transmission.

Initial CuInSe_2 films were nonstoichiometric. X-ray fluorescence spectroscopy of a film deposited at 200°C , Figure 5, shows approximately a Cu deficit (compare Figure 2). The build up of a Cu residue in the flash evaporation source indicated that dissociation of the source material was the cause of the Cu deficit in deposited films. By raising the temperature of the flash evaporation source to an estimated 1100°C , stoichiometric films can be produced. Figure 6 shows 3 x-ray fluorescence spectra taken at different points (separated by approximately 1 inch) on a CuInSe_2 film. They show that stoichiometric films with good

(a)

(b)

(c)

Figure 4.

Cu DEFICIENT CuInSe_2 FILM

Figure 5.

STOICHIOMETRIC CuInSe_2 FILMS

Figure 6.

uniformity over the substrate can be deposited using flash evaporation. The CuInSe_2 must still dissociate with the higher temperature source, however the vapor stream then has the stoichiometric composition and the CuInSe_2 reacts on the substrate.

Another possible cause of nonstoichiometry for CuInSe_2 films deposited on metal electrodes is the diffusion of Cu into the electrode. This is particularly evident for films deposited above 400°C . One such CuInSe_2 film was removed from the metal film (2000 Å gold on 400 Å chromium) on which it was deposited. The x-ray fluorescence spectra of this electrode, Figure 7, shows the presence of the diffused Cu. At lower temperatures this is less significant and it could be prevented by depositing on a Au electrode having an equilibrium concentration of Cu alloyed with it.

X-ray diffraction of the flash evaporated CuInSe_2 films shows them to be single phased. Figure 8 and Figure 9 show diffraction patterns of CuInSe_2 films deposited at 200°C and 640°C respectively. They are both indexable as CuInSe_2 . The film deposited at 200°C shows preferential orientation of grains.

The grain sizes for our flash evaporated CuInSe_2 films have been measured by depositing a series of CuInSe_2 films onto carbon coated glass substrates. These films were deposited to a thickness of approximately 1000 Å. They were then removed from the substrates using an HF etch and photographed with a transmission electron microscope. Figure 10 and 11 show films deposited at 200°C and 600°C respectively. The growth of grain size with temperature and the extremely small

AN ELECTRODE UNDERLYING CuInSe_2 FILM

Figure 7.

X-RAY DIFFRACTION CuInSe₂ FILM - 200°C

Figure 8.

X-RAY DIFFRACTION $\text{CuInSe}_2 - 640^\circ\text{C}$

Figure 9.

←————→
1 μ

Flash Evaporated CuInSe_2 200°C

Figure 10.

Figure 11.

ELECTRON TRANSMISSION MICROGRAPH

CuIn Se₂ Film 600°C

size of the grains are apparent. Over this temperature range grain sizes for flash evaporated films vary from one hundredth of a micron to several tenths of a micron.

Figures 12 and 13 show electron diffraction patterns from the 200°C and 600°C films respectively. These verified that the grain photos are of CuInSe_2 films and the growth of the grain size over that temperature range. L. L. Kazmerski reports grain sizes over 1μ for films deposited at 220°C using dual source evaporation from heated crucibles. The smaller grain size for flash evaporated films is perhaps due to high instantaneous deposition rates with the flash evaporation system. The small grain sizes and (associated small minority carrier diffusion length) is responsible for the absence of photovoltaic response in the junction we have fabricated.

4.4 Dual Source CuInSe_2 Films

A dual source, CuInSe_2 and Se, coevaporation system has been utilized to fabricate efficient CdS/CuInSe_2 heterojunctions.⁸ The selenium source supplies the excess Se required to obtain p type films. The control of the selenium rate relative to the CuInSe_2 deposition rate is required to obtain reproducible properties for the CuInSe_2 films. The use of Electron Impact Emission Spectroscopy is an attractive method of supplying the required deposition control.

A schematic block diagram of the EIES system is shown in Figure 14. At the heart of the system is the Sentinel 200 (made by Inficon, including optical processor and sensor) which permits monitoring of two species simultaneously in the deposition vapor stream. The sensor stimulates characteristic optical

e^- Diffraction CuInSe_2 200°C
Figure 12.

e-Diffraction CuInSe_2 600°C
Figure 13.

EIES DUAL SOURCE SYSTEM

Figure 14.

transistions in the evaporant using e-beam excitation. The emitted light is filtered and detected in the optical processor. This deposition rate information can then be used to regulate the power supplied to the sources and thereby control the film composition.

We had planned on measuring the Cu rate with one channel and using that information to control the CuInSe_2 source while measuring Se rates with the second channel and thereby control the Se source. A number of problems have been encountered in implementing this system. Chief among these has been the difficulty of obtaining emission from Se vapor. Scans from 185nm to 500nm for our initial Se source, a resistance heated ceramic coated tungsten crucible, showed no emission from the Se.

The emissions obtained from an element can be influenced by the molecular binding for that element as it occurs in the vapor stream. Se is evaporated in ring molecules¹⁰ of various number dependent on source temperature and pressure. Another Se source was made in an effort to change the molecular composition of the Se vapor stream and thereby, obtain the desired Se emission at 470nm. This source is shown schematically in Figure 15. The temperature of the resistance heated source ($\sim 250^\circ\text{C}$) is used to control the Se deposition rate. The vapor leaving the source passes through a quartz tube with a heating filament along its axis. This is heated to $\sim 800^\circ\text{C}$ to break the Se into diatomic molecules. No emission from the Se has been seen with this source either. This source has been used to deposit the CuInSe_2 films discussed below. Crystal rate monitors were used for deposition control. Recent work on a bottle type source had indicated a Se

Diatomic Se Source

Figure 15.

line at 198nm. Efforts will be made to utilize the EIES system on that line in the coming year.

A number of CuInSe_2 films have been deposited using the dual source system with the CuInSe_2 crucible under manual control and the Se crucible controlled to give a set Se crucible temperature. Stoichiometric, single phased CuInSe_2 films are readily obtained by this method. Figure 16 shows the x-ray fluorescence pattern of a CuInSe_2 film deposited at a substrate temperature of 400°C . CuInSe_2 films have been deposited at substrate temperatures from 200°C to 500°C . As with flash evaporated CuInSe_2 films, films deposited at lower substrate temperatures have a preferred orientation. Figure 17 shows the x-ray diffraction pattern for a CuInSe_2 film deposited at 250°C , and Figure 18 shows the electron diffraction pattern of the initial 1000 Å of that same film. It indicates that the preferred orientation develops as the film grows in thickness.

Grain sizes have been measured for a number of CuInSe_2 films deposited on carbon coated glass slides to a thickness of ~1000 Å. Grain sizes from .1 μ to 1 μ were seen for films deposited at temperature of 200°C to 600°C . Figure 19 and 20 show transmission electron micrographs of one such film deposited at 450°C . Figure 20 shows a thicker region of the film shown in Figure 19. The growth of the grain size with film thickness is quite apparent.

Resistivity and Hall voltage measurements have been made on a number of CuInSe_2 films. The results are rather variable with resistivities of .001 Ωcm to 20 Ωcm , mobilities of .1 $\text{cm}^2/\text{v-sec}$ to 10 $\text{cm}^2/\text{v-sec}$,

Figure 16

Xray Diffraction CuInSe_2
Dual Source 250°C
Figure 17.

e Diffraction

CuInSe_2 (15-8) 250°C

Figure 18.

← 1 μ →

CuInSe₂, 450°C

Figure 19.

1/4
 CuInSe_2 , 450°C

Figure 20.

and carrier concentrations of 10^{17} cm^{-3} to 10^{21} cm^{-3} . (The higher concentrations are due probably to the presence of both n & p type carriers while using a single carrier type analysis). This great variability in electrical properties is believed to be caused by inadequate control of the selenium rate.

4.5 Device Studies

A number of $\text{CuInSe}_2/\text{CdS}$ heterojunctions have been fabricated. The best, a 6 cm^2 area device, had an efficiency of 0.4% in simulated 100 mw/cm^2 illumination using ELH lamps. Dark and illuminated I-V curves for this device are shown in Figure 21. The device had an open circuit voltage of nearly 0.4 volts which is not unreasonable, however the short circuits current density of $\sim 3 \text{ ma/cm}^2$ needs to be an order of magnitude larger. Indium fingers, 10 mils wide on 100 mil centers, were vacuum deposited to grid this device. This is probably inadequate and accounts partly for the poor collection efficiency. The I-V curves for a second device are shown in Figure 22. Analysis of these curves indicates the shunt resistance, is 3000Ω , series resistance 30Ω , ideality of 2.3 and dark saturation current of $3 \times 10^{-6} \text{ ma/cm}^2$.

4.6 Related Studies

A preliminary investigation of sputtered CuInSe_2 films has been carried out. Single phased, stoichiometric, n type films were rf sputtered from a powder target. Substrate temperatures were quite high so that films showed no preferential orientation. N type films are to be expected due to the high temperatures and lack of excess Se but sputtering in H_2Se may yield p type films. This will be attempted during the next report period.

CdS/CuInSe₂
Illumination 100 mW/cm²
Area 6 cm²

Figure 21.

Figure 22.

The CdS conductivity (carrier concentration) plays an important role in determining device efficiency. Resistivities of 1 Ω -cm or less are required to achieve efficiencies of 9% or better.⁹ We have measured CdS resistivities and Hall voltages for a range of substrate temperatures and deposition rates as well as doping with In. These results are summarized in Figure 23. It is seen that doping with Indium gives the carrier concentration and conductivity required for efficient devices. In doped CdS films will be used to make devices during the next report period.

5.0 Summary

We have looked at several methods of fabricating the CuInSe_2 films. Flash evaporation is an attractive approach as p type CuInSe_2 films have been reported by this method. It is a relatively simple method adaptable to large scale fabrication of devices. Our work has indicated that single phased, stoichiometric, CuInSe_2 films are obtainable using flash evaporation. Film conductivity type control was not good and grain sizes of the films obtained over a wide range of temperatures were too small to be useful for devices. Annealing possibly can be used to obtain larger grains and control film type. This added processing reduces the attractiveness of flash evaporation.

Dual source evaporation systems with a CuInSe_2 source and a Se source have been shown capable of fabricating useful CuInSe_2 films. The control of the Se source aids the deposition of p-type films. We have studied the use of Electron Impact Emission Spectroscopy for monitoring the rate of Se and Cu simultaneously and incorporating it in a feedback loop to control the deposition

CDS ELECTRICAL PROPERTIES

	50 $\overset{0}{\text{Å}}/\text{SEC}$	170 $\overset{0}{\text{Å}}/\text{SEC}$	50 $\overset{0}{\text{Å}}/\text{SEC}$ CDS .5 $\overset{0}{\text{Å}}/\text{SEC}$ IN
150°C	$1.5 \times 10^{16} \text{ cm}^{-3}$ $3 \text{ cm}^2/\text{V-SEC}$ $120 \Omega\text{-cm}$	$1 \times 10^{17} \text{ cm}^{-3}$ $2 \text{ cm}^2/\text{V-SEC}$ $30 \Omega\text{-cm}$	
200°C	$3 \times 10^{15} \text{ cm}^{-3}$ $12 \text{ cm}^2/\text{V-SEC}$ $188 \Omega\text{-cm}$	$1 \times 10^{17} \text{ cm}^{-3}$ $2 \text{ cm}^2/\text{V-SEC}$ $40 \Omega\text{-cm}$	$3 \times 10^{19} \text{ cm}^{-3}$ $70 \text{ cm}^2/\text{V-SEC}$ $.003 \Omega\text{-cm}$
300°C	$6,500 \Omega\text{-cm}$	250°C $204 \Omega\text{-cm}$	

Figure 23.

process. The spectra observed for any element depends on the molecular arrangement of that element in the vapor stream. Initial sources have not given measurable emissions from Se at expected wavelengths so that it has not been possible to utilize this technique for controlling the Se deposition. Preliminary results on our most recent sources have indicated a possible emission due to Se. Further work on this will be carried out.

A dual source system using temperature of the Se source to control rate of Se deposition has been used to deposit a number of CuInSe_2 films. The grain sizes of these films have been measured using TEM microscopy. These measurements indicate $\sim \frac{1}{2}\mu$ grains for films $\sim 1500 \text{ \AA}$ thick. Evidence of grain size growth with increasing film thickness has been seen. Preferential orientation of these films is seen particularly for films deposited at substrate temperatures less than 600°K .

Electrical properties of these films have been measured and devices fabricated. The best device, 6 cm^2 has had an open circuit voltage of 0.4 volts, short circuit current density of 3 ma/cm^2 , and fill factor of 0.4 under 100 mw/cm^2 tungsten illumination. The low device efficiency of 0.45% is attributable to poor collection efficiency. The shunt resistance of $3,000 \Omega$ for this 5 cm^2 area is reasonable while the observed series resistance of 30Ω may indicate a problem with poor gridding or high CdS resistivity.

Resistivity and Hall measurements have been made on a number of CuInSe_2 and CdS films. A wide range of resistivity, carrier concentrations, and mobility have been obtained for CuInSe_2 films. This, it is believed, is indicative of poor control of the Se source. A number of changes have been initiated in the Se source to give uniform controlled Se rates. More work will be done in that area. CdS resistivities have generally been in the range 10 to 100 $\Omega\text{-cm}$. By doping with indium resistivities of .003 Ωcm and mobilities of 70 $\text{cm}^2/\text{v-sec}$ have been obtained.

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