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CADMIUM SULFIDE/COPPER SULFIDE HETEROJUNCTION CELL RESEARCH

Technical Progress Report No. 1, September 30—December 31, 1977

By
J. R. Szedon
F. A. Shirland
W. J. Biter

July 31, 1978

Work Performed Under Contract No. EG-77-C-03-1577

Westinghouse Electric Corporation
Research & Development Center
Pittsburgh, Pennsylvania

MASTER

U.S. Department of Energy

Solar Energy

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CADMIUM SULFIDE/COPPER SULFIDE
HETEROJUNCTION CELL RESEARCH

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Westinghouse R&D Center
1310 Beulah Road
Pittsburgh, Pennsylvania 15235

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Cadmium Sulfide/Copper Sulfide
Heterojunction Cell Research

by

J. R. Szedon, F. A. Shirland and W. J. Biter

ABSTRACT

An approach for the research effort required to meet the program objective is described, leading to an outline of nine tasks comprising the planned program. Results of effort applied to tasks during the first quarter effort are described. Highlights include fabrication and evaluation of cells on Mo foil substrates, a new chemical etch method which may allow easy, rapid determination of CdS grain diameters in films, and preparation and preliminary evaluation of cell structures on single crystal CdS substrates. Several problems will require special effort in the future: poor adhesion of CdS film to Zn-plated Cu foil substrates and a high incidence of film defects attributed to particle spatter from the CdS evaporation source.

Cadmium Sulfide/Copper Sulfide Heterojunction Cell Research

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1. SUMMARY

During the first quarter of this program, emphasis has been on establishing a base-line capability in $\text{Cu}_2\text{S}/\text{CdS}$ thin film cells after an interval of some 15 months during which repetitive processing was not done. Problems have been countered in obtaining good adhesion of CdS films to Zn-plated Cu foil substrates. Preliminary measures to correct the situation have not been successful. The suspected source of the problem is the zinc fluoborate for the plating bath, which is not within specification as regards pH and zinc content. New material of proper composition will be used in future work. In the meantime, CdS depositions on molybdenum substrates have been successful. Cells prepared on these films have been below 3% in conversion efficiency. This is attributed to shunting paths associated with particles spattered from the evaporation source. Some improvement has been achieved and more is projected with accumulation of experience in preparing the sources and executing evaporations. A new etching method has been developed which may be useful in clearly delineating CdS crystallites for size determination by conventional optical microscopy rather than using SEM methods on textured surfaces.

Single crystal CdS substrates are proposed as a method of preparing simpler cell structures more appropriate for compositional analysis and photovoltaic evaluation than are the thin film structures. Acceptable methods for surface preparation of the crystals have been established. A method for forming localized Cu_2S layers using photoresist masking has been successfully demonstrated.

2. INTRODUCTION

2.1 Objective

The objective of this program is to conduct research on solar cells of the copper sulfide/cadmium sulfide and copper sulfide/(cadmium sulfide-zinc sulfide alloy) types with the goal of producing thin film cells capable of 10% AM1 conversion efficiency with less than 5% degradation in performance over a use period of 20 years.

2.2 Background

Thin film copper sulfide/cadmium sulfide solar cells have exhibited reasonably attractive conversion efficiencies using low cost materials prepared, for the most part, with conventional evaporation equipment. Low material cost projections are based, in part, on the fact that the active conversion region of the cell is a very thin ($\sim 1000\text{\AA}$) copper sulfide layer, which is relatively easily formed by treatment of the cadmium sulfide. The high optical absorption of the copper sulfide and the immediately adjacent carrier collection arrangement provided by the copper sulfide/cadmium sulfide heterojunction provide the basis for respectable photovoltaic performance. Another positive contribution to the performance of state-of-the-art $\text{Cu}_2\text{S}/\text{CdS}$ thin film cells is the light-trapping arrangement of the textured surface produced by etching prior to formation of the Cu_2S layer. This and the penetration of that layer into the boundaries between adjacent CdS crystallites produce a complex topography for the active region of such thin film cells.

The extreme thinness of the copper sulfide layer and the geometric complexity of the heterojunction surface in real cells make analysis of important physical, electrical, and chemical parameters of the active cell region very difficult. Furthermore, they make simple applications of theoretical models less useful for understanding detailed

cell mechanisms and for guiding geometric or material improvements. These structural matters have presented formidable obstacles to improving CdS/Cu₂S solar cell performance and to appreciating the practical limit to which these cells may be brought by development.

2.3 Approach

The main thrust of the present approach is to control the material and geometric conditions of the copper sulfide layer and of the heterojunction region. This will be done for two purposes: (1) to allow use of appropriate analytical and evaluation methods in characterizing the optical, compositional and electrical properties of the important parts of the cell, preferably using single crystal substrates, and (2) to permit extension of these results to cells on polycrystalline substrates of controlled grain size with ultimate application to state-of-the-art cells.

Better insight into cell mechanisms obtained from the single crystal or large crystallite results and from improved modelling should result in improved state-of-the-art cells. When the degree of improvement has been preliminarily assessed the matter of degradation effects will be addressed. If the analytical and evaluation methods proposed for the work are valid and if the control of important cell parameters can be realized, changes related to degradation should be more clearly identifiable. More accurate prediction of degradation in improved devices should be possible.

With better understanding of the important parameters controlling cell performance, as developed on the program, a realistic projection of improved performance should be possible, together with estimates of the costs associated with production of cells of the required characteristics.

2.4 Program Plan

The program planned to execute this work involves nine tasks:

1. CdS preparation, evaluation, and control.
2. Cu₂S preparation and evaluation.

3. Prototype cell fabrication and evaluations.
4. $\text{Cu}_2\text{S}/\text{Zn}_x\text{Cd}_{1-x}\text{S}$ studies.
5. Model development and refinement.
6. Copper migration, oxygen, and heat effect studies.
7. Design, fabrication and evaluation of improved cells.
8. Assessment and extrapolation of program results.
9. Cell processing cost analysis.

In the discussion of progress on the program which follows, individual technical topics will be referred back to these tasks.

3. PROGRAM PROGRESS

3.1 CdS Film Preparation and Cell Fabrication

In earlier work at the Westinghouse Research and Development Center, state-of-the-art $\text{Cu}_2\text{S}/\text{CdS}$ thin film solar cells were produced. During a period of about 15 months, regular production of such cells was not pursued; instead selected pieces of apparatus were redesigned and upgraded in anticipation of work similar to that involved for the current program. Reactivation of the procedures for thin film cell fabrication has revealed a problem in CdS film adhesion to zinc plated copper foil substrates. Adhesion was so poor that no cells could be fabricated from the films. Two possible causes of poor adhesion were identified: contamination of the vacuum evaporation chamber with oil from the pumps and contamination or improper formulation of the zinc plating bath.

A thorough clean-up of the vacuum evaporator eliminated oil vapors in the vacuum chamber. The zinc-plating bath was treated with activated charcoal to remove all traces of organic matter and re-filtered to remove the activated charcoal. These actions had no significant effect on film adhesion. A new lot of zinc fluoborate solution used to prepare the present zinc plating bath was suspect. Analysis of this material showed it to be out of specification as regards both pH and zinc concentration. New baths were formulated with this taken into account, but the adhesion problem persisted. A new supply of zinc fluoborate has been ordered. On delivery during the next quarter, its properties will be verified as regards specification before additional attempts are made to produce CdS films on Zn-plated copper. For the interim, CdS film depositions have been made on molybdenum foil substrates. These do not require zinc plating and there are no adhesion problems with this substrate if normal cleaning procedures are used.

Nine CdS film depositions were carried out in December. Foil substrates were 7" x 7" in area. Four evaporation sources were used in the standard process as evolved earlier and described in detail in the final report on the earlier NSF contract.¹ Some general results of the evaporation runs are summarized in Table 1. The remainder were on molybdenum foil substrates. The molybdenum foil was obtained from the AMAX Company, Cleveland, Ohio.

As mentioned above, the CdS films peeled badly from the zinc-plated copper foil substrates, while good adhesion was obtained on the molybdenum foil substrates. The molybdenum substrates were prepared for CdS film deposition as follows: first they were hand scrubbed with a soft bristle brush using dilute Alconox solution to remove any possible traces of oil from the rolling operation; then they were thoroughly rinsed in deionized water and their surfaces oxidized in concentrated HNO_3 (1:1); the black oxidized surface was loosened in concentrated HCl and removed by agitation and rinsing in deionized water. Finally the substrates were rinsed separately in acetone and absolute methyl alcohol.

As noted in Table I, good CdS film adhesion was obtained on all the molybdenum substrates. However, examination with an optical microscope revealed irregularities in the surface of the CdS films which was attributed to spattering of solid particles from the CdS evaporation sources. On HCl etching of the CdS films for texturing prior to formation of the Cu_2S layer, these spattered regions produce pinholes, resulting in partial or complete shorting of the finished cells. In part, this spatter problem can be reduced by modifying the quartz wool packing in the evaporation sources and by reducing the evaporation rate. Improvements in the former area were made for the last CdS film listed in Table I. Reproducible results are expected as the operator gains more experience with such details.

Table II summarizes the data on the cells fabricated from selected CdS films of Table I. For most runs the cells were so badly shorted that no attempt was made to characterize the cells in detail. Of the remainder, many cells were partially shorted, resulting in low

TABLE I - CdS FILM EVAPORATION RUNS
(December 1977)

Run No.	Substrate	Adhesion	General Film Quality	General Cell Quality	Comments
489	.0014" Cu	Half of area peeled	Badly spattered	Not usable for cells	-
490	.0014" Cu	Very poor	-	Not usable for cells	-
491	.002" Mo	Good	Badly spattered	Cells badly shorted	-
492	.001" Mo	Good	Spattered	50% badly shorted	See Table II
493	.001" Mo	Good	Spattered	Partially shorted	See Table II
494	.001" Mo	Good	Very badly spattered	Badly shorted	Collimation cone off one source during run
495	.001" Mo	Good	Film thickness uneven Badly spattered	Badly shorted	Slower evaporation rate used. Collimation cone off one source during run
496	.001" Mo	Good	Badly spattered	All shorted	-
497	.001" Mo	Good	Noticeably less spattered	Usable for cells	See Table II

TABLE II - RESULTS FOR CELLS FABRICATED (December, 1977)

Cell No.	V _{OC} (volt)	J (mA/cm ²)	η (%)	Fill Factor	R _{series} (ohms)	R _{shunt} (ohms)
492-1A	Shorted	-	-	-	-	-
-1B	.370	5.1	-	-	-	-
-2	.390	11.3	1.8	-	-	-
-4B	.22	4.5	-	-	-	-
-5B	.42	3.4	0.7	0.50	4.2	-
-7A	Shorted	-	-	-	-	-
-7B	"	-	-	-	-	-
-8A	"	-	-	-	-	-
-8B	"	-	-	-	-	-
493-9	.41	4.1	1.0	0.62	2.5	1400
-10	.44	8.1	2.2	0.63	1.8	700
-15	Shorted	-	-	-	-	-
-18	"	-	-	-	-	-
-20	"	-	-	-	-	-
-22	"	-	-	-	-	-
497-2	.434	10.7	1.9	0.40	1.1	90
-3	.458	11.5	2.9	0.55	2.7	240
-3' (*)	.463	12.3	2.7	0.47	1.4	260
-4	.452	12.0	2.7	0.50	2.6	150
-4' (*)	.460	13.1	2.9	0.48	1.8	140

*Cut to 1 cm² area

open circuit voltage and poor conversion efficiency. Only the last run of the month, No. 497, gave open circuit voltage approaching an acceptable level for $\text{Cu}_2\text{S}/\text{CdS}$ cells. It gave lower than expected short circuit current, efficiencies less than 3.0%, and poor fill factors.

The work described above relates primarily to Tasks 1 and 3 of the Program Plan in Section 2.4.

3.2 CdS Crystallite Delineation

An important aspect of the physical evaluation of CdS films for cell fabrication or detailed studies is obtaining information on the crystallite size distribution as associated with substrate and processing details. Inferences of distributions can be made from SEM evaluations of CdS films after texturing by an HCl etch, but this approach is time consuming. A convenient method for clearly delineating crystallite boundaries, without significant texturing is important to the present study as regards crystallite size effects on cell performance.

Work during this quarter may be useful in providing a technique for determination of grain size and grain size distribution. A chromic acid etching solution was prepared by adding 0.8g of Cr_2O_3 to a solution of 10 ml H_2SO_4 per liter of H_2O . Etching was carried out at 80°C . Film samples of CdS were etched in this solution at 80°C . After ten minutes of etching, there was no texturing of the CdS film surface, but fissures were produced delineating regions having sizes expected of the CdS crystallites ($\sim 1 \mu\text{m}$ across). These features were quite evident using optical microscopy. Typical examples of SEM micrographs before and after 10 minutes etching are shown in Fig. 1.

Attempts were also made to see if this etch would delineate structural defects at the surface of individual grains. At 55,000X magnification using the SEM there was indication of localized point etching. More work would be needed to determine whether these features have significance as regards defects. Pursuit of this at some time in

Fig. 1 Scanning electron micrographs at 20,000X magnification of CdS film surfaces.

TOP: As evaporated films. Note the absence of features delineating the grain boundaries.

BOTTOM: After 10 min. of etching using the method described in the text, the dimensions of the top surfaces of crystallites can be appreciated.

the future might provide a useful tool in measuring the effect of various film formation conditions on the structural quality of the CdS films.

3.3 Apparatus for High Temperature Deposition of CdS Films

Prior to the beginning of this program, a hot wall evaporation apparatus was designed, built and preliminarily tested on Westinghouse funding. For the current program some changes in the equipment are being made. The new system will permit deposition of CdS films at temperatures higher (to $\sim 600^{\circ}\text{C}$) than in the system used for state-of-the-art CdS film preparation. Such a facility is required for the production of CdS crystallites greater than $1\text{ }\mu\text{m}$ in diameter (Section 2.4, Task 1).

Earlier work on CdS films deposited by the hot wall inner chamber technique was carried by Shirland, et al.² with substrate temperatures of 600°C . Films of up to 5 microns thickness were grown using In_2S_3 -doped CdS starting powder material. Film resistivities of 10 to 100 ohm-cm and grain sizes of about 5 to 10 microns were obtained. Cells made from those films gave conversion efficiencies up to 3.3%, but the conditions were not optimized.

There were difficulties during the earlier work in controlling the doping of the CdS films and in nonuniform doping profiles through the thickness of the films. These are believed to be a result of using a single evaporation source and the consequent segregation of dopants due to difference in vapor pressure between CdS and the dopant. Therefore, in this equipment, provisions were made for the use of up to three separate sources for evaporation of CdS and dopants or for the evaporation of Cd, S and dopant. The details of the apparatus are shown in Fig. 2. The chimney is Vycor tubing, 5-1/2" in diameter by 7" long. In it, threads were ground about 1/64" deep, eight to the inch. The chimney was wound with 0.020" diameter tungsten wire and then wrapped with unsized Fibrefrax (Al_2O_3 matting) insulation. The insulation was held in place by an 0.001" thick tantalum foil heat shield. A well was provided midway up the outside of the chimney for a thermocouple.

Fig. 2 Tooling for Inner Chamber, Hot Wall Evaporation.

The evaporation sources are quartz crucibles, positioned beneath the chimney with their orifices projecting through openings of a specially machined molybdenum support base. The chimney rests on the molybdenum support base. Cartridge heaters were buried in the base and provision for monitoring the base temperature was made by a thermocouple embedded in the base. The three sources were arranged with 120° symmetry about the axis of the chimney.

Molybdenum foil was planned for the substrates for these experiments. Molybdenum foil, 0.002" thick stress relieved material, was obtained from the Amax Company. Substrates are mounted in a copper wire frame (for cleaning and handling purposes). The substrate and frame are placed on top of the chimney to completely cover the top opening. The substrate is heated by a radiation heater positioned approximately 0.5 inches above it. Substrate temperature is measured using a fine (0.001" junction) thermocouple in pressure contact with the back surface of the substrate foil. A number of test runs were carried out and films of CdS having good appearance and adhesion were obtained on the molybdenum foil substrates.

Some modifications to the apparatus are needed, however. First it was found that the 0.020" diameter tungsten wire used to heat the chimney was not satisfactory. The brittleness of the tungsten caused breakage at the terminals in handling during loading and unloading operations. Provisions for stress relief at the terminals help somewhat but difficulties were still experienced. Also, the high currents needed to reach temperatures over 400°C could not be handled by the 0.020" tungsten winding. Therefore, the chimney was rewound using 0.025" diameter molybdenum wire. It was also discovered that the cartridge heaters used to heat the chimney base support were not able to operate at temperatures in excess of about 265°C. Special high-temperature heaters of increased wattage were secured for this purpose.

3.4 Surface Preparation of Single Crystal CdS

Single crystals of CdS will be used to prepare samples for electrical, compositional and other studies (Section 2.4, Tasks 2, 4, and 6)

related to Cu S/CdS solar cells which are precluded by the polycrystalline material in thin film solar cells. Single crystals of CdS have been obtained from two different vendors: Eagle-Pitcher Co. of Miami, OK and Cleveland Crystal Co. of Cleveland, OH. The crystals are sliced 90° to the C-axis and are 2 mm thick. The resistivities are as grown and range from 1 to 10 ohm-cm.

The slices are received unpolished with the expectation that in-house surface treatment will give tighter control over the process. A number of polishes for CdS have been described in the literature. These usually include a final HCl chemical polish to maintain a good finish on the Cd face. Sullivan and Bracht³ used 30% concentrated HCl while Pritchard and Wagner⁴ used dilute, buffered HCl polish. The latter report evaluation of the surface by means of photoluminescence using a 457.99 nm laser. The absence of surface damage is inferred for good response, considering that the absorption depth in CdS for this wavelength is about 0.1 μm .

It is not clear what minimal surface quality of the CdS is required for the $\text{Cu}_2\text{S}/\text{CdS}$ cell. Since the Cu_2S forms topotaxially into the CdS, shallow surface damage should not affect the junction. Damage could affect the Cu_2S layer itself. It is well known that the conversion of thin film CdS to Cu_2S proceeds faster along grain boundaries. Similar behavior might be expected in damaged material. In addition, damage might affect carrier transport in both the CdS and Cu_2S portions of cells.

The following procedure was found to give acceptable results in the work to date. The sample is polished on a glass plate. American Optical M320 (5 μm grain size) abrasive is used initially, followed by Linde A. Linde B(.05 μm) abrasive is then used with a Pellon cloth on a glass plate. Polishing is done until there are no remaining marks visible on the sample when examined under a microscope. The final chemical polish is a free etch using buffered HCl (.5 ml HCl, 13.5 gr. KCl and 1000 ml of H_2O).

In initial studies a chemical polish using a pad was tried, however, this produced pitting of the sample surface. A 4 to 8 hour free etch produces a very good surface, although it is not featureless. However, part of this problem is believed to be due to defects in the CdS. The surface of one of the CdS crystals was examined (after the etch) with a reflection electron microscope. From the pattern, surface damage was inferred to be slight, extending less than 100Å into the CdS. A longer etch should eliminate this remaining damage layer, if such action is necessary.

3.5 Cu₂S/CdS Cells based on Single Crystal Material

For state-of-the-art thin film solar cells of the Cu₂S/CdS type, the major contribution to photocurrent collection is from the low bandgap, highly absorbing Cu₂S layer. In the thin film structure the Cu₂S layer is not readily analyzed independently of its effect on cell performance because of the surface irregularity of the underlying polycrystalline CdS. For this reason single crystal CdS substrates are being used on the current program. In addition to permitting more meaningful characterizations of Cu₂S layers with regard to their thickness, planar single crystal-based structures are more amenable than state-of-the-art film structures to micro-analysis in terms of composition profiles and film chemistry using ESCA, Auger or similar techniques.

A number of single crystal CdS cells were prepared to date. The light generated current and OCV were reasonably high although no effort has yet been made to optimize the Cu₂S layer. Most of the work has involved designing of tooling needed to fabricate and test the single crystal cells.

The present cell fabrication technique used photolithographic methods to define the area of the Cu₂S region. First, both sides of the CdS are polished and chemically etched as described in Section 3.4. Indium is evaporated through an aperture mask onto an area of the front surface of the CdS for convenience in contacting the CdS region with a probe. Photoresist (Shipley AZ-1350H) is applied, exposed and developed

to delineate the region for preparation of the Cu_2S layer of the cell. The resist can be stripped in acetone if needed and appears to have no effect on cell performance. The Cu_2S layer was formed by dipping for 20 seconds in a CuCl solution at 100°C , carefully rinsing and then annealing in air at 250°C for 2 minutes. Cells with reasonable values of J_{SC} (10 mA/cm^2) have been obtained. Further measurements will be made on these cells to determine the losses. Evaluations of cells operating in both frontwall and backwall modes are planned as soon as tooling is finished for improved methods of probe contacting.

Development of a method for the preparation of the Cu_2S layer of the cell. The resist can be stripped in acetone if needed and appears to have no effect on cell performance. The Cu_2S layer was formed by dipping for 20 seconds in a CuCl solution at 100°C , carefully rinsing and then annealing in air at 250°C for 2 minutes. Cells with reasonable values of J_{SC} (10 mA/cm^2) have been obtained. Further measurements will be made on these cells to determine the losses. Evaluations of cells operating in both frontwall and backwall modes are planned as soon as tooling is finished for improved methods of probe contacting.

4. PLAN FOR NEXT QUARTER

During the next quarter of this program (January 1, 1978 to March 31, 1978) effort is planned on the first six tasks listed in Section 2.4. Specifically the following items will be addressed.

1. Extend the evaluation of CdS films on various substrates.
2. Subject Cu_2S /single crystal CdS structures to compositional analysis and to electrical/optical evaluations aimed at measuring minority carrier properties of the Cu_2S layer.
3. Continue efforts to resolve the problem of CdS film adhesion on Zn-plated Cu foil substrates and evaluate thin film cells to establish a base-line capability.
4. Prepare (ZnCd)S films on single crystal CdS substrates by evaporation and assess preliminary results.
5. Begin modelling efforts for cell structures, as appropriate to deal with experimental results.
6. Initiate studies to evaluate and differentiate between heating, oxidation and copper migration influences.

6. ACKNOWLEDGMENTS

The authors are happy to acknowledge the technical support of J. A. Stoll in CdS film preparation and cell fabrication and of D. R. Detar for the SEM work involved in the report. They are appreciative of the masks made available by T. A. Temofonte for fabrication of single crystal devices.

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