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THIN FILM CdS/Cu₂S SOLAR CELLS BY CHEMICAL SPRAYING

Final Report

By
John F. Jordan

MASTER

MASTER

December 15, 1976

Work Performed Under Contract No. EY-76-C-04-3579

D. H. Baldwin Company
Cincinnati, Ohio

U.S. Department of Energy

Solar Energy

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D. H. Baldwin Company
1801 Gilbert Avenue
Cincinnati, Ohio 45202

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I. INTRODUCTION

This project involves further work on a process developed by the D. H. Baldwin Company, which indicates that it is eventually and technically feasible to make very thin film CdS/Cu₂S solar cells. The process is adapted to the float glass process and consists of spraying suitable chemicals on a moving ribbon of glass which floats on a bath of molten metal. The spraying produces the necessary thin films (with suitable optical and electrical properties) of SnO₂, CdS and Cu₂S.

The CdS films produced are two (2) to three (3) microns thick, thus conserving the use of cadmium. The estimated cost per peak watt is approximately 7¢ (1975 costs) at 5% efficiency and an output of 37.6×10^6 square meters annually.

The cells which are backwall cells are shown in Fig. 1. A float glass plant modified for the process is shown schematically in Fig. 2.

Contract Objectives

- (1) Life test cells of present 4.5% average efficiency
- (2) Increase average efficiency to 5%
- (3) Life test cells of 5% average efficiencies

Present Status

Cells are now being made by the subject process averaging 4.9% in efficiency with some cells exceeding 5.3% efficiency. Short circuit currents averaging 20 milliamperes per square centimeter are readily obtained. This is a satisfactory value indicating fairly good current collection. Open circuit voltages of our standard cells, however, are lower than they should be, averaging some 395 millivolts whereas they should be about 470 millivolts. Fill factors are about 62%.

Maximum Voc of unelectroded experimental cells = 522 mv

Maximum Voc of electroded experimental cells = 495 mv

However, the currents are lower for these higher Voc cells.

Progress During Report Period

- (1) Average efficiency has been increased from 4.5% to 4.9%.
- (2) Device characterization tests have been devised and the necessary equipment has been set up for J_0 , alpha, and ϕ_B measurements.
- (3) Life tests are in progress, two correctable degradation mechanisms have been identified and remedies devised.
- (4) Accelerated life tests have proved the effectiveness of the above remedies.
- (5) Maximum Voc of experimental cells has been increased to

522 mv for unelectroded cells and to 495 mv for electroded cells.

- (6) Complete removal of residual CdCl_2 has been shown possible.
- (7) Large grain growth in pure CdS and standard films has been obtained as well as in solid solutions of ZnS/CdS.
- (8) Improved process control equipment has been designed, placed in use and preliminary results obtained.
- (9) A joint analytic program between Baldwin and Sandia has been started, and useful data has been obtained.

Efficiency Increase

A moderate increase in efficiency was realized. It was due to:

- (1) Use of an improved plating schedule which gave higher short circuit currents.
- (2) Removal of peripheral Cu_xS from positive electrode which consistently improved our fill factor.
- (3) Better control over residual chlorides in the CdS layers.

The Cu_xS layer at present is made by first dipping the cells in a solution of CuCl , then electroplating Cu, not as an electroding operation, but as a method to increase the Cu_xS thickness. By electroplating at a lower current density and longer times, the cell efficiency has been improved. Yield has been improved since cells previously rejected can be improved by removal of the peripheral Cu_xS not covered by the positive electrode. In many cases, removing the peripheral Cu_xS removes shunting paths, thus increasing the fill factor and the voltage. An IV curve of one of our better cells is shown in Fig. 10.

Improved residual chloride tests have revealed a layer of CdCl_2 present on the CdS after spraying. This layer is normally removed during heat treatment. However, any non-uniformity in the layer may produce non-uniformity in the CdS after heat treatment. Efforts to eliminate and/or make this layer more uniform, have resulted in noticeable increases in yield.

Device Characterization (Physical)

Until very recently, it has not been possible to characterize the films by any simple single test. Recently, with the aid of a field emission SEM, structure in these very thin films has become visible (see Fig. 3). This is a photomicrograph of a cell which has been fractured and the cross section photographed, showing the fracture morphology which we assume is intimately related to the structure. Using this assumption, we see elongated grain sizes in the CdS averaging 0.2 to 0.4 microns. However, much smaller grains (0.03 microns) are also visible.

The granular structure is also indicated by etching. First the CdS surface is converted to Cu_xS . The Cu_xS is then etched away in KCN and the weight loss measured. This shows an apparent Cu_xS thickness of 0.2 to 0.4 microns. The original yellow CdS color returns after this etching, showing no "visible" trace of Cu_xS . However, this remnant CdS is now easily removed by mild abrasion, as with a wet paper towel, rubbing it with hand pressure. (The CdS is not normally attacked by KCN nor is it easily removed by abrasion). The thickness of CdS rubbed off is approximately 2 microns. The only CdS remaining is essentially the first layer, i.e., the layer of CdS containing aluminum. This demonstrates that

penetration of the CuCl dip does proceed down the grain boundaries until stopped by the first CdS layer containing aluminum. Granular structure is also demonstrated by the ease with which pure CdS layers are penetrated by the dip process. It is interesting to note that sprayed Cu_xS does not penetrate even thin films of pure CdS. This is an advantage of the spray process.

With the SEM, it has also been possible to see the effect of heat treatments on our films. See Fig. 19.

The film structure shown by these photos is not optimum; small grain size is suggested as a major reason for the Voc of our cells being 100 mv less than cells made by the evaporation process. In order to study the correlation between film structure and efficiency, a program of electrical parameter studies was started.

Device Characterization (Electrical)

It has been suspected that the relatively low open circuit voltages observed in the sprayed CdS cells are caused by excessive carrier recombination via the interface states at the $\text{Cu}_2\text{S}/\text{CdS}$ junction. An analytical investigation of the current voltage characteristics and their temperature dependence was undertaken to evaluate the diode parameters and to determine the nature of current flow mechanism at the junction. A temperature chamber (range -70°C to 170°C) was set up for this purpose; the cell could be illuminated from a solar simulator placed outside the chamber. A fan blowing right on the cell insured that the cell temperature did not rise appreciably above the chamber temperature during the brief time it was illuminated with "One Sun".

Preliminary results obtained on a single cell (#5/25/76 CID) are reported here. Fig. 4 shows the I-V characteristic of this

cell in dark and under simulated sunlight (total intensity 85 mw/cm²). Open circuit voltage is 390 mv and short circuit current 21 $\frac{\text{ma}}{\text{cm}^2}$. After correcting for the series and shunt resistance, $\log (J + J_L)$ is plotted against the junction voltage in Fig. 5. The single diode equation in the presence of light can be written as:

$$J = J_0(\exp. \alpha V - 1) - J_L$$

where J is current density; V is junction voltage; J_L is the light generated current density and α is diode parameter whose value is determined by the dominant current mechanism at the junction.

$\alpha = \frac{q}{kT}$ for the regular diffusion current across the junction. Assuming $e^{\alpha V} \gg 1$, the slope of the straight line in Fig. 5 yields $\alpha = 16.9$. Intercept on the current axis yields $J_0 = 3.2 \times 10^{-5} \text{ A/cm}^2$.

Fig. 6 shows the logarithmic current voltage plots at different temperatures. These curves are seen to be practically parallel to each other. This means that α is temperature independent, a result which strongly supports a tunneling current mechanism at the $\text{Cu}_2\text{S/CdS}$ junction in our cells. Rothwarf⁽¹⁾ has described the various current flow processes in the thin film $\text{Cu}_2\text{S/CdS}$ cells. Our initial results strongly suggest that one or both of the following processes are dominant:

- (1) Tunneling of electrons from deep levels in CdS to the interface states at the junction, followed by recombination with holes in Cu_2S .
- (2) Tunneling of holes from Cu_2S to the deep levels in CdS followed by recombination with the electrons in CdS.

Both of these processes require a large number of deep level states in CdS. The presently used technique for spraying CdS leaves ample room for impurity and defect centers to provide the deep level recombination centers mentioned above. As our efforts to increase the grain size and improve the crystal structure of CdS are successful, we expect to use diode parameter measurements to give us an understanding of cell operation and correlation with process parameters.

Life Tests

Life tests are in continuous operation and have provided much valuable information. Two degradation mechanisms have been identified and remedies devised to correct them.

The first and the most useful life test is one used by Centre National & Etudes Spatial, 91 Bretigny St Orge, BP No. 4, France, to screen their solar cells. It consists of operating the cells at 100°C under one sun illumination continuously for 100 hours. Both illumination and temperature are closely controlled throughout the test. The voltage of the cells is monitored during the test. The I-V curves are recorded before and after the test. C.N.E.S. has found that this 100 hour test is equivalent to 4,000 hours of continuous sunshine or seven (7) years of continuous operation in the area of Paris, France.

The first degradation mechanism discovered was the effect of residual CdCl₂ in the film. Since the main chemicals used to spray these films are CdCl₂ and Thiourea, it is evident that the pyrolysis reaction is not going to completion. Using a recently developed

wet chemical test for residual chloride, methods of controlling residual chloride were developed. These methods, so far, are:

- (1) Addition of HCl to the starting chemicals reduces chloride residue.
- (2) Variations of spray parameters, such as flow rate and solution concentration, also affect chloride residue.
- (3) Cd to S ratio has some effect on residual chloride, but the effect is more complex than previously thought and is still being studied.

Fig. 7 shows the effect of residual chloride in accelerated life tests.

Another degradation mechanism identified was the effect of that Cu_xS adjacent to the peripheral of the positive electrode which shunts the cell during life tests. Fig. 8 shows the cell construction used for life testing and the structure change when the peripheral Cu_xS is removed. Fig. 9 shows the effect of the peripheral Cu_xS on the I-V curve of a cell having undergone accelerated life testing.

It should be noted here that during life tests, these cells are not protected by an inert atmosphere such as dry nitrogen or vacuum as is usual with other types of $\text{CdS}/\text{Cu}_x\text{S}$ solar cells. The cell is protected on one side by the glass substrate and by the copper-lead positive electrode on the other side. The cells are subjected to uncontrolled humidity.

Cu_xS Removal

A technique for removal of unprotected Cu_xS prior to life testing was developed using KCN as an etchant. These cells were tested

using the CNES accelerated life test. It became evident during testing that on some cells, a new degradation mechanism was operating, shunting the cell. This degradation could be removed by simply soaking the cells in deionized water. Evidently, it is a residue around the edge of the electrode left by the KCN etch. More rigorous rinsing techniques have greatly reduced this failure mode. It should be noted that, in production, the KCN etch will not be used in any event.

The second type of life test ongoing is a real time roof-top life test. Cells have been on roof-top in the southwest U. S. area for over 8,000 hours. Some of these cells have shown no change whether open circuited, short circuited or loaded for maximum power during the entire test duration. Some of the cells have degraded as much as 35%. It should be noted that all of these cells still had unprotected Cu_xS peripheral to the positive electrode. At the end of the report period, no significant real time life test data had been accumulated for cells with peripheral Cu_xS removed prior to testing.

These cells were protected only by a thin layer of plastic .003 inches thick and were mounted in the following configuration:

The cells were directly exposed to rain, snow and dust, the windscreen serving to prevent impingement of high velocity sand and rain.

Voc Increase

Maximum Voc was increased by:

- (1) Heat treatment of CdS films in H_2S .
- (2) Use of chromium as a positive electrode to Cu_xS .
- (3) Revised junction formation procedure.

The H_2S heat treatment times and temperatures are approximately the same as our standard, i.e., $500^{\circ}C/30$ min.

Our ordinary heat treatment in air has three noticeable results:

- (1) The film is made harder and more resistant to CuCl dipping.
- (2) The residual chloride is reduced by half.
- (3) There is visible grain growth in the second layer. The

H_2S heat treatment extends the first two results, e.c., the film is many times more resistant to CuCl dipping and the residual chloride level is reduced to a level undetectable by our tests. There is no visible difference in structure as seen by the SEM between air heat and H_2S heat treatment.

This H_2S heat treated CdS is resistant to grain boundary penetration by the junction formation chemicals. After removing the Cu_xS with KCN, the remaining CdS is still resistant to abrasion. Contrast this with our standard air heated CdS, which loses cohesion after KCN etch removal of the Cu_xS . This indicates that there is much less penetration of cuprous ions along the grain boundaries in our H_2S treated cells.

The residual CdCl_2 is possibly removed by the following reaction:

Chromium has been used as a positive electrode with a thickness of 0.17 microns. A thicker lead layer is used on top. This electrode yields higher V_{oc} than Cu when used with H_2S treated CdS cells. On ordinary standard cells, Cr and Cu produce nearly equivalent cells.

Fig. 20 shows the energy band diagram that can be expected to obtain at the interface between chromium and copper sulfide. It is clear that a Schottky-type contact instead of an ohmic contact is predicted. However, in practice, because of the large conductivity of Cu_xS film, a conducting contact, instead of a barrier, is observed. This can be understood in terms of tunneling across the "thin" depletion region (of width W) at the interface. Assuming a hole density of 10^{19} CM^{-3} in Cu_xS , a rough estimate of W is obtained from Poisson's equation

$$\frac{d^2v}{dx^2} = \frac{qN_A}{\epsilon}$$

$$V_D = \frac{qN_A W^2}{2\epsilon}$$

$$W = \frac{2\epsilon V_D}{q N_A}^{\frac{1}{2}}$$

Where V_D is the diffusion potential at the interface, ϵ is the dielectric constant of copper sulfide, q is the electronic charge and N_A is the space charge density in the depletion layer in copper sulfide. Letting $V_D = 0.7$ volts, $\epsilon = 10$ and $N_A = 10^{19} \text{ CM}^{-3}$, we get $W = 86$ Angstroms.

For such small depletion layer width tunneling, probability can be expected to be quite high so that the electrical contact between vacuum evaporated chromium and Cu_xS film is quite conductive. Chromium, as an electrode for $\text{Cu}_x\text{S}/\text{CdS}$ cells, offers the advantages of stability (not susceptible to oxidation) not chemically reactive to Cu_xS and relatively inexpensive.

The junction formation was revised as follows: The chemical treatment times were shortened while an additional heat treatment at 200°C in air was used between chemical treatment steps.

The currents for these high voltage cells are low, from 7 - 10 ma/cm^2 , compared to approximately 20 ma/cm^2 for our standard cells.

Large Grain Growth

Heat treatments in atmospheres saturated with CdCl_2 and CdS have produced films with much larger grains. See Fig. 21. This work is the result of experiments done in 1973, but the results were not confirmed until the CdS was examined with the SEM just recently placed in service.

With the help of an SEM, a correlation between grain size and structure and CdS processing parameters has been sought. Initial results indicate that CdCl_2 acts to some extent as a catalyst for growth of grains.

Larger crystallites or grains of up to 13 microns in diameter were produced. These grains grow, at the expense of their smaller neighbors, into discreet masses separated by voids extending to the tin oxide layer. See Fig. 22. In order to use such films, a method of either causing the grains to join and fuse into a

monolithic layer or of filling the voids must be found.

Some small areas of monolithic CdS have been observed in films otherwise composed of separated grains. It isn't known what caused these areas to grow. See Fig. 23.

CdS/ZnS Films

The main results are as follows:

- (1) Optically the band gap shifts as a function of ZnS percentage. See Fig. 24.
- (2) The ZnS is very resistant to the dip solution.
- (3) Some films show dendritic forms indicating inhomogeneities. See Fig. 25a.
- (4) When heat treated in air, a ZnO (Ref. 2 phase) becomes visible. See Fig. 25b.

Process Control

Two new devices to improve the film deposition process and thus improve the film itself have been completed and operated. The first is a nozzle to generate uniformly sized droplets. The spraying process used thus far has involved the use of air atomizing nozzles which shear a column of solution containing the active chemicals into droplets which vary in diameter by 100-1 and in volume by a million to one. Previously, high speed photography of the actual spray deposition process showed the need for uniform droplets 50 microns or less in diameter. The present nozzles produce droplets of 125 microns diameter. We have achieved 50 micron droplets in single aperture nozzles and are now working to achieve this size droplet in multi-aperture nozzle.

Fig. 11 shows these nozzles. Although the method of generating the droplets is the same in both cases, (i.e., ultrasonic agitation), the methods of droplet dispersal are different. Droplet dispersal is necessary as an individual stream of droplets, if allowed to hit the hot glass substrate, would overload the reaction causing poor film formation and glass cracking.

The first design disperses the droplets by means of a high voltage electrostatic field between the nozzle and substrate. The second design uses a low flow stream of air to form vortices and disperse the droplets by air velocity variations. The air dispersal nozzle used one-tenth the air flow of an air atomizer nozzle. Previous tests showed the heat loss through the substrate, because of propellant air, was as great as the heat lost by droplet evaporation.

Films produced by these nozzles are smoother and more transparent than usual. Transparency is important since present CdS films transmit only 70% or less of incoming radiation.

SEM photographs by Sandia in Figs. 12 and 13 show the difference in morphology between the two films in surface views and in cross section.

It should be noted that the nodular morphology is caused by the Al inclusion in the first layer. The pure CdS grows into a much smoother film.

The other process control device is a controlled atmosphere spray booth shown in Fig. 14. This booth permits nitrogen to be used both as a propellant and as a means of removing the reaction by-products during film formation. Previous analysis by Sandia

showed oxygen present in the pure CdS. Also Cu_xS is known to be very sensitive to ambient oxygen during formation. Oxygen levels as low as 0.1% can be attained.

Preliminary results obtained so far are:

- (1) CdS containing Al grows much more slowly in low oxygen atmosphere.
- (2) Pure CdS grows at a normal rate but the resultant film reacts more rapidly in the CuCl dip.
- (3) Initial Cu_xS films deposit more rapidly but seem to be less stoichiometric.

Because of differences in geometric layout incorporated into the controlled atmosphere spray booth, a large increase in deposition rate has been observed.

Joint Baldwin-Sandia Analytical Program

Several meetings between Baldwin and Sandia Laboratories have taken place. As a result, a schedule of recommendations for a program of analysis on characterization of films and completed devices has been agreed upon. A complete report of this program will be published by Sandia, but a summary of some key results will be made here.

1. Auger and SIMS have shown that the Al contained in the first layer of the two-layer CdS film is present as an oxide and not as a sulfide or unbound free Al.
2. SEM photographs have shown more clearly the structure of the films.
3. Rutherford Backscattering profiles of the Cu in the C_xS layer have shown a 400° + thick layer at the surface with a high concentration of copper, then a lesser concentration penetrating

at least .5 microns. This surface layer has been tentatively identified as CuO by electrochemical means by Baldwin. Sandia is attempting to verify this. The profile is shown in Fig. 15.

4. Impurity analysis of the film and starting chemicals have been made and are shown in Figs. 16 and 17.

5. Optical analysis of the films have been performed. Typical values of the absorption constant of CdS are shown in Fig. 18.

6. Material studies, in order to improve the substrate, were made. A low iron content glass was proposed and samples sent to Baldwin. This glass transmits 7% more of the solar radiation than does our present float glass substrate.

7. Sandia personnel have studied the optical properties of our films and have observed a correlation between the spectral transmission curve and cell performance. This holds promise of being a valuable analytic tool and quality control aid.

8. Several groups of cells which have been made from CdS sprayed varying stoichiometry or temperature were sent to Sandia Labs for analysis. They will issue a report on this.

- (1) A. Rothwarf in International Workshop on Cadmium Sulfide Solar Cells and Other Abrupt Heterojunctions, University of Delaware, May 1976, Page 15.
- (2) T. Karwan and C. Malinowsky in Thermochemica Acta 17 (1976) 195-206. Grain Size and Grain Shape Effect of Zinc Sulphide Structure and of Oxidizing Process Conditions.

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CROSS SECTION OF BACKWALL SOLAR CELL

SCHEMATIC OF A FLOAT-GLASS
SOLAR-CELL PLANT

Fig. 3

Fig. 4. Dark and Light I-V characteristics of cell # 5/25/76 C1D

RESIDUAL CHLORIDE LEVEL
7 %

RESIDUAL CHLORIDE
LEVEL 1.7%

100° c ONE SUN ACCELERATED LIFE TEST

Figure 7

Figure 8

BEFORE TEST

AFTER TEST

AFTER TEST AND
REMOVAL OF PERIPHERAL
UNPROTECTED Cu_xS

Figure 9

FIG. 10

Uniform
Droplet
Generator

Air
Atomized
Droplets

Surface View of CdS Films

Fig. 12

Uniform
Droplet
Generator

Air
Atomized
Droplets

Cross Section Views of CdS Films

Fig. 13

CONTROLLED ATMOSPHERE SPRAY BOOTH
 O_2 LIMIT .1%

Figure 14

Figure 15

Rutherford Backscattering spectrum showing a determination of the copper profile in the near surface region by subtracting the extrapolated CdS background. The lower trace shows the resulting copper profile.

FILM IMPURITIES AND THEIR SOURCES

FILM IMPURITY	STARTING CHEMICALS	WATER AND SPRAY EQUIPMENT	AIR	SUBSTRATE
O			MAJOR	
Na	X			C
Mg	I	X		
Al	I			
Si	I	X		
Cl	MAJOR			
K	X			C
Ca	X			
Mn		I		
Fe	I	C-M		
Ni	I			
Cu	I	X		
Zn	X			
Sr	I			
Pb			C	

I = PPM

X = TENS OF PPM

C = HUNDRED OF PPM

M = THOUSANDS OF PPM

Figure 16

Constituents in SnO_x Film Sample 5b-1
by AES and SIMS

Major	Medium	Minor
O	Cl	C
Sn	S	Al
Na	F	Si
		K
		Ca
		Fe ?

Figure 17

CdS absorption constant and net absorption

Figure 18

Non Heat
Treated

Heat
Treated

Fig. 19

Fig. 20

Fig. 21

Gaps Between Large Grains

Note CdCl_2 Crystal

Fig. 22

Fig. 23

Fig. 24

(b)

(a)

Fig. 25