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Novel Two-Stage Selenization Methods for Fabrication of Thin-Film CIS Cells and Submodules

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> B. M. Basol, V. K. Kapur, A. Halani, C. Leidholm *International Solar Electric Technology Inglewood, California*

NREL technical monitor: H. S. Ullal

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1.0 SUMMARY

This is the Phase I Annual Technical Progress Report of a subcontract titled "Novel Two-Stage Selenization Methods for Fabrication of Thin Film CIS Cells and Submodules". The objectives of the program are the development of a cost effective process for CIS film deposition, optimization of various layers forming the CIS solar cell and fabrication of submodules using these processes and devices.

During this first phase of the program we have completed our 1 $ft²$ size processing capabilities and added to our facilities an in-line sputtering system that can handle up to 1 ft² size substrates. We have optimized the sputtering conditions for the Mo contact as well as the Cu and In films. Thickness uniformity of the Cu and In layers have also been optimized by masking the magnetron cathodes to obtain a variation of 3 % throughout a $ft²$ substrate. Using the resulting films, we have demonstrated our first large area CIS submodules with outputs of about $3W/ft^2$. Addition of a computer controlled mechanical scriber to our fabrication facilities, and optimization of the large area ZnO layers are expected to improve the power output of these submodules to over $5W / ft^2$ shortly.

In addition to the large area submodule work, we have also carried out research aimed at the development of a non-vacuum processing approach for the growth of CIS layers. We have deposited films using this technique, and small area cells with over 10% conversion efficiency have been demonstrated on such CIS layers.

2.0 INTRODUCTION

This is the Phase I Annual Technical Progress Report of a subcontract titled "Novel Two-Stage Selenization Methods for Fabrication of Thin Film CuInSe, Cells and Submodules". The objectives of this program are the development of a cost effective process for CuInSe, (CIS) film deposition, optimization of various layers forming the CIS solar cells and fabrication of 1 ft² size submodules using these approaches.

Recent advances made in CIS and related compound solar cell fabrication processes have clearly demonstrated that these materials and device structures are capable of yielding power conversion efficiencies exceeding 15 % . Thin film CIS solar cells fabricated at ISET employing the two-stage process and the device structure of Fig. 1 have already achieved impressive conversion efficiencies of 12 %-13 % . Electrical characterization of these devices indicated presence of good quality rectifying junctions with diode factors of around 1.5.

ISET's two-stage process for CIS thin film growth involves selenization of Cu-In precursors. ISET, through previous contracts from NREL and various other sources have developed a novel approach to precursor preparation. In this approach the Mo coated glass substrate shown in Fig. 1 is first coated with a thin layer of Te. This step is then followed by the sequential deposition of In and Cu layers. The resulting films are highly alloyed Cu-In precursors which can be repeatably processed into good quality CIS layers. The details of these processes and the results obtained were described in our 1992 Final Report [l] and will not be repeated here.

As part of our previous subcontract with NREL we had also put into commission some hardware in which CIS submodules of 1 ft² area could be processed [1]. This hardware included a metal sputtering system, a selenization reactor, a CdS- deposition system and a MOCVD reactor for ZnO deposition. During the present project period we have concentrated our efforts on improving the large area processing facilities at ISET and fabricating 1 ft² area submodules. Working with large area substrates for the first time gave us valuable information about the issues regarding module manufacturing. Accordingly, certain processing steps were changed and optimized along with some of the 1 ft^2 processing hardware. The following sections of this report will provide information about three major areas of research carried at ISET during the last 12 month period, i.e. large area deposition of films, submodule fabrication and novel processing for CIS solar cells.

Fig. 1 Glass/Mo/CIS/CdS device structure used by ISET.

3.0 TECHNICAL DISCUSSIONS

Details of the selenization technique and the device fabrication steps employed in this program have been previously described [l, 2, 3, 4]. In summary, Cu-In precursors were deposited onto Mo-coated soda-lime glass substrates at room temperature using the evaporation or sputtering methods. For the fabrication of small area films and devices and for specific experiments we used the E-beam evaporation approach. For large area substrates, the sputtering technique was employed. Selenization of the precursors was carried out in a reactor which was kept at around 400 °C. The reactive atmosphere in the selenization chamber contained a mixture of H₂Se gas and Ar. After the selenization step, CIS films were coated with a thin (1000-2000 A) CdS layer using the solution growth technique. This step was then followed by the deposition of a ZnO window layer using the MOCVD method.

3 .1 Large Area Deposition of Films

Large area deposition facilities, i.e. the metal sputtering system, CdS and ZnO deposition

setups and the selenization reactor were described in our last final report. During that program we had set up these facilities and had started to process 1 ft^2 size substrates. During this period we have further improved our 1 ft^2 processing capability and added to the facilities an in-line sputtering machine, and a mechanical scriber for module integration.

Large area Mo, Cu and In depositions were carried out in a sputtering chamber. The most important issue in Mo sputtering is the control of the intrinsic stress which are operative in these layers. In our experiments, we found that the commonly known relationship [5] between the film stress and the sputtering gas pressure was also valid for our Mo films. In other words, the layers deposited at relatively low working gas pressures $(< 10$ mTorr) displayed a compressive stress and low electrical resistivity. The stress changed into tensile in films prepared at Ar pressures higher than 10 mTorr and the resistivity went up. We have optimized our deposition parameters to obtain good quality Mo layers at a pressure of around 5-8 mTorr. These layers adhered well to their glass substrates and they displayed a resistivity of around $2x10^{-5}$ ohm-cm. Resistivity of films prepared at 15 mTorr Ar pressure was as high as $7x10^{-5}$ ohm-cm. These variations observed in the characteristics of sputtered metal films are known to be tied to the changes in the grain structure of such layers. Films grown at low Ar pressures consist of compact filamentary grains, whereas, those obtained at high pressures display a columnar grain structure with a high density of voids.

In our process, the quality of the Cu and In precursors greatly influences the nature of the resulting CIS films and devices. Precursors with proper stoichiometry and morphology tend to selenize into films that yield high efficiency devices. Much of our work during this period of the project was focused on improving the sputtering equipment and the sputtering conditions utilized in preparing the Cu-In precursors. The equipment improvements included the installation of an improved substrate motion apparatus, changes in the gas distribution system, installation of a radiation heater into the chamber, and installation and optimization of cathode shields to improve the overall thickness uniformity of the deposited layers. Improvements have also been made in the manner in which the films were sputtered. Experiments were carried out to find the optimal film stack, sputtering conditions, and substrate speeds which produced precursors with the desired stoichiometry and morphology.

The Cu and In layers were sputtered from individual cathodes in a diffusion pumped vacuum system. A schematic of this system is shown in Fig. 2. The two cathodes were internally-mounted DC-planar magnetrons which could accommodate 17" x *5"* size Cu and In targets with purity levels of 99.99% and 99.999%, respectively. The substrates were vertically mounted in the substrate motion apparatus. This apparatus allowed the substrates to be passed, in either direction, by each cathode at a constant speed. Both the

substrate's speed and its distance to the targets were adjustable. A radiation heater was also mounted above the cathodes to provide substrate heating if desired. Film thicknesses were controlled by adjusting the cathode power, sputtering pressure, substrate-to-target distance, and the speed of the substrate. Relationship between the cathode power level and the coating thickness at a given pressure was found to be linear. Fig. 3 is a plot of the In film thickness at various cathode power levels and it confirms this linear relationship. A similar result was obtained for sputtered Cu layers.

Fig. 2 Sketch of the metal sputtering system used for coating 1 ft^2 size substrates.

To improve the thickness uniformity of the deposited films cathode shields were placed in front of each target. Careful experimentation with different shield shapes yielded improved thickness uniformity over a 1 ft² substrate. Finally, the sputtering gas pressure was maintained at a constant level by a mass-flow controller.

The ultimate output of the CIS module fabricated by the two-stage process depends strongly on the nature and the quality of the Cu-In precursors. We have done a great deal of work to find the optimum sputtering conditions which would produce precursors that would selenize into high efficiency devices. As mentioned before, the uniformity of the Cu/In molar ratio across the ft^2 substrate is of great importance in our process. If certain areas of a module had Cu/In molar ratios outside the range of 0.88 to 1.00, the module's output would be significantly reduced. We have carried out some experiments to determine the stoichiometric uniformity of the Cu-In precursors before they were selenized. In these experiments large area substrates were cut into small pieces and the

Fig. 3 Relationship between the coating thickness and the cathode power for In sputtering.

Cu/In ratio of each piece was determined using Atomic Absorption Spectroscopy. Fig. 4 shows the distribution of Cu/In molar ratios on a 1 $ft²$ area sample which was processed early in the program. It is clear from this figure that certain portions of a module fabricated on such a film after selenization would have stoichiometric ratios very close to or larger than 1.00, and this would limit the performance of the overall device.

Achieving a uniform Cu/in ratio over a ft^2 size substrate requires finding the sputtering conditions and the proper cathode shields which would produce uniform Cu and In film thicknesses throughout this substrate. By evaluating film thicknesses and adjusting the shape of the cathode shields we were able to improve thickness uniformities and hence improve our ratio uniformity. Prior to optimization we were observing a standard

Fig. 4 Cu/In molar ratio at various points on a 1ft^2 substrate coated by DC magnetron sputtering.

deviation in the stoichiometric ratio of \pm 5% across the ft² substrate. After optimization of the shape of the cathode shields we were able to reduce the standard deviation in the ratio to \pm 3%. A plot of the Cu/In ratio distribution across a ft² size substrate after the optimization work is shown in Fig. *5.* Although the overall average ratio of this substrate is slightly low at 0.90, 63% of the module has a ratio between 0.87 and 0.93. If the average ratio were slightly higher, say 0.93 , then 63% of the module would have a ratio between the optimal 0.90 to 0.96 range. This overall decrease in the spread of ratios across the ft^2 size sample helps ensure that the entire module's Cu/In molar ratio is within the required range. This substantial improvement in ratio uniformity has been directly correlated to improved module output as will be reported later.

In addition to the Cu-In thickness uniformity in macro scale, stoichiometric uniformity of CIS films in micro-scale is also of utmost importance for solar cell performance. Cu-rich regions in the films, even though they are on micro-scale, reduce the open circuit voltage values of the devices and give rise to lower module efficiencies.

As stated before, in our method of precursor preparation, the In layer is deposited first on the selected substrate which is previously coated with Te. This step is then followed by the deposition of a Cu film. Therefore, it is expected that the characteristics of the precursor layers obtained by our technique will be affected by the nature of the In films

Fig. *5* Distribution of Cu/In molar ratios across a $ft²$ substrate after the optimization of the sputtering process.

which form the base over which the Cu layer is deposited. When we first initiated the sputtering/selenization process and started to fabricate devices under conditions, which we felt were equivalent to those obtained by the E-beam evaporation/selenization method, we consistently obtained cells with low V_{∞} values. A comparative study of the sputtered and evaporated Cu-In precursor films offered certain clues regarding this problem.

Fig. 6 and Fig. 7 show the SEMs of evaporated and sputter deposited Cu-In precursor films and the CIS layers obtained after selenizing them. It is observed from this data that the E-beam evaporated precursor of Fig. 6a yielded a relatively uniform CIS surface after selenization (Fig. 7a), whereas, the sputtered precursor of Fig. 6b produced a CIS film with a small-grained matrix over which large crystals could be observed (Fig. 7b). An EDAX study of these crystals indicated that they were Cu-rich compared to the underlying small-grained matrix. Since the overall Cu-In ratios of both types of films were in the same acceptable range, the reason for the formation of Cu-rich areas in the sputtered films was investigated by studying the nature of the sputtered In layer.

The SEMs of Fig. 8 show the morphologies of In films sputter deposited on Mo coated glass substrates at two different $(Ar+5\%H_2)$ pressures. The film deposited at low

Fig. 6 (a) E-beam evaporated Cu-In precursor before selenization, (b) after selenization.

Fig. 7 (a) Sputter deposited Cu-In precursor before selenization, (b) after selenization. The In film was deposited at an $Ar + H_2$ pressure of 10 mTorr. Cu-rich large grains are visible in the SEM of (b).

pressure (Fig. 8a) was found to be dark in color and its surface topography was rough. This film also looked discontinuous compared to the In layer of Fig. 8b which was smooth and light silver colored. A comparative microprobe analysis of the two indicated that the deposit of Fig. 8a contained a higher level of oxygen.

Our sequence of In / Cu deposition is intended to cause complete and uniform alloying throughout the precursor film between the low-melting-point In base layer and the Cu film that is deposited over it. Partially oxidized In layers with non-uniform surface morphologies are expected to give rise to non-uniform precursors which in turn may result in the non-uniform CIS film of Fig. 7b. This problem was overcome by optimizing the In deposition parameters and specifically working at high pressures during In depositions.

(a) (b)

Fig. 8 4000 A thick In layers deposited on Mo coated glass substrates. Sputtering conditions were: (a) P= 1kW, $p_{Ar+H2} = 2$ mTorr, (b) P=1kW, $p_{Ar+H2} = 20$ mTorr.

In addition to the Cu and In film thickness uniformity, the repeatability of stoichiometric control from run to run is also crucial especially in a production process. During the last quarter of the project we set up an in-line sputtering facility to improve the reproducability of our process.

The new machine is an MRC sputtering system. It utilizes a vertical sputtering configuration which eliminates virtually all particulate from contacting either the substrate or the target. Any dislodged particulate falls to the chamber floor. The substrate pallet is a vertically oriented plate which translates past stationary targets held in the chamber front plate. Up to three targets may be used in this machine, as well as RF sputtering etch and radiant heating up to 600° C. RF and DC magnetron and RF diode sputter deposition process modes are available in the system. Located on the chamber front plate is the loadlock dome, which acts as both a door and as a separately evacuated chamber. Substrates, mounted on pallets held vertically by the loadlock elevator, move from the loadlock into the main chamber, where they are automatically transferred to the pallet carrier, which translates the full width of the chamber.

The high vacuum pump is a cryogenic closed-cycle helium pump mounted on the chamber back plate, eliminating the possibility of contamination by the b_ackstreaming of pump oils, as in the case of diffusion pumps. We are now in the process of carrying out experiments on the film uniformity in this system. This work will involve cathode masking and optimization of cathode power levels and substrate speed. Since the main deposition chamber of this system remains under vacuum at all times, we expect to achieve better repeatability of film thicknesses compared to the setup depicted in Fig. 2. It should be noted that especially the In target surface gets oxidized every time the deposition chamber is exposed to atmosphere.

3 .2 Module Integration

Modules measuring up to 1 ft^2 area were fabricated on soda-lime glass substrates. The first step in the fabrication process was the formation of the back electrical contact. This was accomplished by sputtering a 1-2 μ m thick Mo layer on a glass sheet. The Mo layer was then laser-scribed to produce 52 electrically isolated segments measuring approximately 0.5 cm x 30 cm, and wide contacting areas at the two ends of the substrate. After cleaning the surface a 100-200 A thick Te film was deposited on the Mo layer by either electrodeposition or evaporation. This was then followed by the deposition of an In and a Cu film using the D.C. magnetron sputtering technique as described before. After the selenization step cell integration was accomplished by a series of film depositions and mechanical scribes. First a 1000-2000 A thick layer of CdS was deposited on the CIS layer using the dip coating method. A 2-3 mil wide channel was then scribed in the composite film to expose the Mo contact along the edges of the Mo segments. A layer of doped ZnO was then applied by the MOCVD technique to form the top electrode, and to make the electrical connection, through the scribes, with the lower Mo contacts. Module integration was completed by making a final isolation scribe through the ZnO/CdS/CIS layers. Both of the latter scribes were made using a hand-operated mechanical scriber. Fig. 9 illustrates an integrated section of a typical CIS submodule.

Fig. 9 A section of the integrated CIS submodule.

We have fabricated several submodules of various sizes in this project. Mechanical scribing step of the module integration process was carried out manually for these early devices. Therefore, the area utilization was quite poor at around 75%. Fig. 10 is an example of the illuminated 1-V characteristics of our early submodules. This specific device had an aperture area of lO"xll" and 42 cells in series and it was measured outdoors in Inglewood California. The measured insolation was 97 mW/cm^2 . The open circuit voltage of the submodule is 15 V corresponding to about 0.36 V per cell. The short circuit current value is 0.32 A. Since the total area per cell is about 14 cm², the short circuit current density in the cell level can be calculated to be about 23 mA/cm^2 . This low value is indicative of the poor area utilization due to manual integration procedures as well as the excessive absorption in the ZnO layer which was estimated to cause a current density loss of about $4-5$ mA/cm². We have fabricated several submodules with performance equivalent to the device of Fig. 10. These module outputs correspond to about 3 W/ft². The highest efficiency we obtained for smaller area submodules so-far is 7.4% for a device with 38 cm² area. It should be noted that the module results presented in this report represent our first attempt to process large area devices using the sputtering technique, and we expect to improve the performance of these devices shortly.

Fig. 10 Illuminated I-V characteristics of a submodule. $A = 710$ cm², $P_{out} = 2.2$ W. Measured under sunlight (97 mW/cm^2) .

3 .3 Novel Cells

During this period ISET has initiated work on a novel approach to CIS solar cell processing. The objective of this effort was to develop a technique that did not use the rather costly vacuum techniques for the growth of CIS films. Furthermore, the aim was to eliminate vacuum processing for all the other layers of the solar cell structure.

Fig. 11 is a SEM of a CIS film prepared by the novel technique. The film has a granular grain structure with an apparent grain size of 1-3 μ m. There are large formations of 10-15 μ m size at the surface of the film. We have looked at the composition of these large formations and compared it to the composition of the background matrix. We have not

Fig. 11 SEM of a CIS layer obtained by ISET's non-vacuum technique.

seen any stoichiometric differences.

We have demonstrated the feasibility of this novel approach by fabricating high efficiency devices on CIS films obtained by the non-vacuum approach. Fig. 12 shows the illuminated 1-V characteristics of two such devices. The area and the efficiency of the device of Fig. 12a is 0.09 cm^2 and 10.3% , respectively. This corresponds to an active area efficiency of about 11.1% . The 1 cm² cell of Fig. 12b is 8.8 % efficient (9.5%) active area efficiency). Prof. Jim Sites of CSU characterized the device of Fig. 12a. The results of his analysis are given in Fig. 13. The current-voltage characteristics in both light (open circles) and dark (filled circles) were measured (Fig. 13a). The solid and dashed lines in this figure are fits for the case of $R=0$ and $r=\infty$. The cell parameters of the device are r_{light} =700 ohm-cm², r_{dark} =1100 ohm-cm², R_{light} =0.2 ohm-cm², R_{dark} =0.2 ohm-cm², $A_{\text{light}} = 1.9$, and $A_{\text{dark}} = 2.0$. The hole density calculated from the capacitance data was $7x10^{16}$. The quantum efficiency data and the photocurrent loss analysis for the cell is shown in Fig. 13b. The window, deep penetrating photon and reflection losses

Fig. 12 Illuminated I-V characteristics of (a) a 0.09 cm^2 device and (b) a 1 cm^2 area cell, fabricated on CIS films obtained by the non-vacuum technique.

Fig. 13 (a) $J+J_L$ vs V data taken from the device of Fig. 12a. (b) J_{sc} loss mechanisms for the same device.

were found to be 2, 3.5 and 3 mA/cm², respectively. There was an unidentified loss of 7 mA/cm²which could partially be attributed to excessive optical absorption in the ZnO layer. Absorption in the ZnO window could not, however, explain by itself this rather large loss in the photocurrent. Further analysis done on EBIC data by R. Matson of NREL shed some light into the loss mechanisms in this device. Fig. 14 shows the SEI and EBIC data taken from a spot on the cell surface. The dark areas in the EBIC data correspond to areas of the SEI data with obvious holes in the ZnO window layer. In fact, EDAX measurements made on these dark areas did showed them to be bare CIS surfaces without any Zn signal. A lower magnification EBIC data indicated a large density of these low-response areas on the surface of the device. Obviously, when ZnO was deposited over the CIS surface and then etched to isolate the device area by photolithographic techniques, it came off the areas with large surface boulders. We were actually able to correlate these low EBIC response areas with the presence of large surface formations.

4.0 Conclusions

As a result of this first year effort ISET has demonstrated its first 1 ft^2 size CIS submodule. Facilities to process such devices have been completed except for an automatic mechanical scriber and a laser scriber. The sputtering process has been optimized to obtain near-uniform coating of Mo, Cu and In films and the sputtering conditions have been studied to determine the deposition parameters necessary to obtain stress-free Mo layers as well as Cu and In films with good morphology and coverage. The early modules fabricated in this program yielded power outputs corresponding to 3 W for an aperture area of 1 ft². Better area utilization and ZnO films with higher optical transmission are needed to improve the conversion efficiencies of these devices.

In addition to the large area submodule development, ISET has also been involved with the development of a novel approach for CIS film deposition. This technique does not use any vacuum process and therefore, is potentially very cost effective. During this period we have demonstrated over 10% efficiency for a cell fabricated on a CIS film grown by the novel approach.

5.0 Acknowledgements

The authors are grateful to R. Matson, A. Franz, A. Mason and K. Emery of NREL for measurements and to Dr. H. Ullal and K. Zweibel for extensive technical discussions.

6.0 Future Plans

Our plans for future development include improving the efficiency of the 1 ft^2 size modules and the novel CIS cells. Efficiency improvement in the large area devices will be partially achieved by setting up a computer controlled mechanical scriber which will allow better area utilization in the integrated modules. Development of this scriber is funded by the California Energy Commission. We have also initiated some work for the improvement of the ZnO layers produced by our large area reactor. Temperature and dopant concentration are the two parameters that are known to affect the optical transmission of ZnO films deposited by the MOCVD method. We will concentrate on these parameters in our exploratory work. Cu/In ratio uniformity is adequate at this time, however, we expect better repeatability from the in-line sputtering system we set up towards the end of the project. We believe that without changing any of the processes in place we can fabricate submodules with over *5* W /ft2 by just improving the scribing and

the ZnO layer.

In addition to the large area module work, our effort on the development of the novel CIS processing approach will continue during the second phase of this project. We will start fabricating small size submodules using this approach. Work will concentrate on the improvement of the surface morphology of these layers which at the present time is very rough.

7 .0 List of Publications

In addition to the list of publications given in our last Final Report [l] the following additional papers were published in the area of thin film solar cells:

- **B.M.** Basol, "Processing high efficiency CdTe solar cells", Int. J. Solar Energy, 1992, vol. 12, p. 25.
- B.M. Basol, V.K. Kapur, A. Halani, C.R. Leidholm and A.J. Minnick, $\ddot{}$ "CIS cells and modules fabricated using the selenization technique", Proc. 11th ECPVSEC, Montreux, Switzerland, 12-16 October 1992.
- B.M. Basol, V.K. Kapur, A. Halani and C. Leidholm, "CIS thin film solar cells fabricated on flexible foil substrates", Solar Energy Materials and Solar Cells, vol. 29, 1993, p. 163.

8.0 References

- 1. B.M. Basol, V.K. Kapur, A. Halani and C. Leidholm, "Low cost CIS submodule development", Final Subcontract Report, July 7, 1990-January 31, 1992, NREL/TP-413-5010.
- 2. **B.M.** Ba§ol, **V .K.** Kapur and A. Halani, "Advances in High Efficiency CIS Solar Cells Prepared by the Selenization Technique", 22nd IEEE PVSC, 1991, p. 893.
- 3. B.M. Ba§ol and V.K. Kapur, "CIS Thin Films and High Efficiency Solar Cells Obtained by Selenization of Metallic Layers", 21st IEEE PVSC, 1990, p. 546.
- 4. B.M. Ba§ol, V.K. Kapur and R.J. Matson, "Control of CIS Film Quality by Substrate Surface Modifications in a Two-Stage Process", 22nd IEEE PVSC, 1991, p. 1179.
- *5.* J.A. Thornton, in "Deposition Technologies for Films and Coatings" Eds. R.F. Bunshah etal., Noyes ,New Jersey, 1982.

Abstract

The objectives of this program are the development of a novel cost effective process for CIS film deposition, optimization of various layers forming the CIS solar cells and fabrication of large area submodules using these processes and devices. During this period of performance these objectives have been mostly met. Specifically, about 1 ft^2 size CIS submodules have been demonstrated using the two-stage selenization technique. Additionally, a non-vacuum CIS processing approach has been developed, and solar cells with over 10% conversion efficiency have been demonstrated using this novel method. Our early large area modules have power outputs corresponding to about $3W/ft^2$.

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