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CUPROUS OXIDE PHOTOVOLTAIC CELLS

First Quarterly Technical Progress Report, April 9—July 8, 1979

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Solar Energy

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Emerging Materials Systems for Solar Cell Applications

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I. INTRODUCTION

Cuprous Oxide (Cu_2O) is of interest as a material for photovoltaic cells because of its low cost, high availability and inexpensive processing. It is a direct band gap semiconductor with a band gap of 2.0 eV which is in the suitable range for solar cells. It has been produced thus far only as a p-type semiconductor so that p-n junctions have not yet been demonstrated. Thus most of the work on Cu_2O cells has been done with Schottky barriers with some explorations of heterojunctions. This has produced cells with a conversion efficiency of 1%.

The previous work on the project at Wayne State University has been summarized in a Final Report on NSF Grant AER-75-23453 (1) and a Final Report on DOE Contract ET-78-S-02-4726 (2). The work was also reported at ERDA and DOE Program Reviews during 1976-1978 and at IEEE Photovoltaic Specialists Conferences. (3,4)

In this previous work, a variety of Schottky barrier cells with a number of metals as contacts on Cu_2O were studied, and it was found that the measured barrier heights did not show a clear dependence on the work functions of the metals. (1,3) We postulated that this was due to chemical reactions at the Metal/ Cu_2O contacts. This was confirmed, especially for the case of Al/ Cu_2O contacts, by high resolution Auger/ESCA surface studies. (2,4)

The current aim of the research is to avoid or prevent this chemical degradation in order to achieve higher barrier heights and thus higher V_{oc} and improved conversion efficiencies. One possible method of preventing this chemical reaction is to interpose a stable oxide interlayer, e.g., Al_2O_3 or SiO_2 , between the metal

and the Cu_2O . Since this layer must be thin in order to allow tunnelling, the structure becomes an MIS structure, which may provide additional advantages.

Another approach to improved Cu_2O solar cells could be through heterojunctions. In favorable cases, i.e., heterojunctions with other oxides, this could avoid the chemical degradation that some Schottky barrier cells display.

Thus the main directions chosen for research on Cu_2O cells in the current phase of the research are the investigation of MIS cells using mainly oxide interlayers and the exploration of heterojunctions on Cu_2O , especially using other oxides.

Other items in the research plan include the preparation of materials including the doping of copper and cuprous oxide; characterization of Cu_2O samples including measurement of optical absorption, diffusion length, resistivity, and Hall effect; exploration of composite cells with lower band gap materials such as silicon; and the development of models for MIS cells, heterojunctions, and Schottky barrier cells.

II. EXPERIMENTAL APPARATUS AND METHODS

Since the new research on MIS and heterojunction cells would require more extensive and versatile equipment for the deposition of oxides and other compounds, the experimental facilities of our laboratory were substantially expanded. In order to maximize the expansion within the budget limitations, the equipment was largely assembled from purchased or laboratory-built components and fixtures.

A. Ion Sputtering Apparatus

The ion sputtering system was built around a 2.5 cm Ion Tech ion source, using a Varian 6 in oil-pumped vacuum system as a base. The ion gun base plate is mounted on an 18" diameter glass cylinder vacuum chamber. Suitable power supplies have been built or purchased. A plasma bridge neutralizer has been added. A sample holder was constructed and provided with facilities for heating and cooling of samples, application of bias, and measurement of I-V characteristics in the vacuum system. The sputtering targets can be mounted on a four-position carousel holder. A pan type holder allows the use of powders in the target. In addition, metal coating can be done from a shielded resistance-heated source.

This equipment has been used to sputter a variety of materials in argon or oxygen, such as SiO_2 , Al_2O_3 , CdO , ZnO , Al , In_2O_3 , etc.

B. Electron Beam Evaporation Apparatus

The electron beam equipment was installed in a Perkin-Elmer Ultek ultrahigh vacuum system, Automatic Rapid Cycle A-RCS, with an 18" x 30" bell jar. This is a non-oil system and uses an ion pump together with a Cryopump CFR. The E-gun which was installed in this apparatus is an Airco-Temescal 4-pocket turret, Model STIH-270-1. This is powered by a Sloan electron beam power supply of 5 KW and 10 KV, equipped with an X-Y sweep. Appropriate sample holders and a crystal deposition monitor have been installed. A separate resistance-heated source has also been provided.

C. Apparatus for Evaluation of MIS Structures.

The apparatus for measuring cell parameters has also been upgraded and expanded. Two separate sets of equipment have been assembled to measure I-V and capacitance-voltage (C-V) curves. One of the sample holders is shielded and temperature-controlled, so that measurements can be made from 15° C to 90° C. Modifications are being made on the other sample holder to give it similar capabilities. The power supply has been modified to allow it to be bipolar without switching and also to absorb up to 200 mA of current from a photocell without affecting its accuracy. Figure 1 is a block diagram of the experimental setup. The current output of the cell is measured by measuring the voltage drop across a standard resistor in series with the cell. The capacitance is measured by superimposing a small AC voltage on the DC bias voltage. The AC signal developed across the measurement resistor is measured with a PAR Model 124 lock-in amplifier.

The spectral response curves on the cells are measured with essentially the same equipment that has been used for the surface photovoltage measurements. Figure 2 shows a block diagram for this apparatus. The monochromator is a SPEX Model 1704 Spectrometer and has a spectral band pass of 5 Å or less for the slit setting of 0.5 mm or less used in these experiments. For spectral response measurements the photovoltaic cell is placed in the sample holder with the top contact fingers directly contacting a tin oxide coated transparent electrode, whereas for the SPV measurements a transparent dielectric is placed between the top surface of the Cu_2O sample and the top transparent electrode.

Figure 1. Experimental Setup for IV and C-V Measurements on Solar Cells

Figure 2. Experimental Setup for Spectral Response and Surface Photovoltage Measurements

III. ELECTRONIC PROPERTIES OF MIS STRUCTURES.

A. Preparation and Photovoltaic Characteristics

A number of MIS structures were prepared on Cu_2O using various metals as top contacts and several oxides as interlayers. The oxides SiO_2 and Al_2O_3 were examined first in order to select the best candidate for MIS cells. Films of these oxides were deposited on Cu_2O by ion sputtering in the apparatus described above, and the structures were examined by depth profiling using high resolution Auger/ESCA techniques. The $\text{Al}_2\text{O}_3/\text{Cu}_2\text{O}$ structure showed as much as 20% Cu(O) at the interface indicating the presence of the chemical reaction previously postulated, presumably in this case due to some residual reducing action in the AlO_x as deposited. The $\text{SiO}_2/\text{Cu}_2\text{O}$ appeared to be relatively free of Cu(O) but a faint trace may have been present. Thus the SiO_2 deposit was chosen as the best producible by present techniques for use as an interlayer.

A number of MIS cells were prepared on Cu_2O using SiO_2 as the interlayer and using gold, silver, copper and aluminum as top contacts. The thickness of the SiO_2 was varied from 10 Å to 150 Å and greater. Table I shows the types of cells that were prepared and gives the photovoltaic properties. As the table shows, the cell resistance increased with increasing thickness of oxide, and above 100 Å, there was a marked increase. Since tunneling through a smooth uniform interlayer should be effective only below 40 Å, this indicates that the SiO_2 interlayers are non-uniform in thickness, or they contain partial shorts or inclusions of conductive material, or that some conductivity mechanism other than tunnelling is present. Another

TABLE I. Photovoltaic Properties of Cuprous Oxide MIS Cells.

Top Contact	SiO ₂ Thickness Å	Cu ₂ O Resistivity Ω·cm	J _{sc} mA/cm ²	V _{oc} V	Cell Resistance Ω
Gold	10	95	Ohmic Junction		5
	20	200	No Photoresponse		12
	40	480			91
	60	340			96
	80	780			440
	100	890			230
Silver	10	250	0.75	0.055	30
	20	250	0.25	0.025	33
	40	170	0.04	0.025	45
	100	220	0.005	0.004	350
Copper	10	95	1.35	0.08	20
	20	200	0.10	0.04	65
	40	480	0.20	0.03	50
	60	340	0.15	0.08	60
	80	780	Slight Photores.		130
	100	890	Slight Photores.		220
Aluminum	10	250	1.4	0.24	35
	20	200	1.25	0.25	45
	40	170	0.15	0.11	45
	60	250	0.10	0.19	150
	100	220	0.03	0.14	190
	150	250	No Photoresp.		2500
	500	220	No Photoresp.		12,000

result shown in the table is that the V_{oc} increased from Au to Ag to Cu to Al, which is the order of decreasing work function of these metals. Gold, as we had established previously, gave an ohmic contact in these MIS structures, as it does with Au/Cu₂O "Schottky type" junctions. The silver MIS cells also gave the type of performance expected from the corresponding Schottky barrier cell. However, the results for the Cu MIS cells were poorer than expected from a comparison with Schottky cells. This may indicate some difficulty in making good junctions in these MIS cells rather than a degradation due to the insertion of the SiO₂ layer. The aluminum MIS cells were the best of the series but still not up to the performance of the usual Al/Cu₂O cell.

B. Current-Voltage Characteristics

The dark I-V characteristics of these MIS cells are shown as semilogarithmic plots in Figures 3, 4, and 5. Assuming the applicability of the equation

$$J = J_0 [\exp(qV/nkT) - 1]$$

the values of the ideality factor n and the values of J_0 were obtained. Applying the Richardson equation to J_0 , the values of the barrier heights were calculated. The values for n and ϕ_B are shown on the corresponding figures.

The barrier heights for the silver and the copper MIS cells are very similar to each other but considerably lower than the 0.68 eV normally obtained for a Cu/Cu₂O cell. The barrier heights for the two thinner aluminum MIS cells are similar to that for a Cu/Cu₂O cell. These cells may in fact be Cu/Cu₂O Schottky barriers,

Figure 3. Dark J-V Curves for Ag/SiO₂/Cu₂O MIS Cells of Various Oxide Thicknesses

Figure 4. Dark J-V Curves for Cu/SiO₂/Cu₂O MIS Cells of Various Oxide Thicknesses

Figure 5. Dark J-V Curves for Al/SiO₂/Cu₂O MIS Cells of Various Oxide Thicknesses

arising from a chemical reaction of the aluminum with the Cu_2O through pores in the SiO_2 layers.

The semilogarithmic I-V curves for the thicker aluminum MIS cells do not yield linear plots and the extrapolated values of J_0 give considerably higher calculated values of the barrier height. These could be examples of MIS structures in which the n-value varies with applied voltage in a manner similar to the theoretical model of Card and Rhoderick. (5)

C. Spectral Response of the MIS Cells.

The spectral response of a number of the MIS cells was measured. Figure 6 shows the spectral response for a set of $\text{Cu/SiO}_2/\text{Cu}_2\text{O}$ cells of different thicknesses of SiO_2 . Similar results were obtained for the other cells. The peak at 2.6 eV may correspond to a peak in the absorption spectrum as given by Pastrnak (6). However, in some preliminary measurements in our laboratory, we have not confirmed the existence of this peak in the absorption spectrum. While our results agree with Pastrnak's at lower frequencies, the values of the absorption at the expected peak ($\sim 10^5 \text{ cm}^{-1}$) gave too small a light transmission to measure in the spectrophotometer. We are attempting to prepare thinner samples in order to measure the absorption coefficient in this region.

D. Junction Capacitance Measurements

Since the capacitance of MIS structures can be used to determine some features of the junction and its depletion layer as well as the insulating layer, a series of capacitance measurements were made on

Figure 6. Spectral Response of Cu/SiO₂/Cu₂O MIS Solar Cells

the MIS cells prepared for the I-V and spectral response measurements. The apparatus and methods used were described in an earlier section. Some preliminary results are presented here.

The results can sometimes be represented by linear plots of $1/C^2$ vs V . Figures 7 and 8 show the data for Al/SiO₂/Cu₂O and Ag/SiO₂/Cu₂O MIS structures with different thicknesses of SiO₂. The intercepts for the aluminum MIS cells with 10 and 20 Å SiO₂ are about 0.35 to 0.4 V which are similar to those for the Al/Cu₂O Schottky barrier cells. However, for the Al MIS cell with 60 Å SiO₂, the intercept is 1.2 V, which does not appear to be reasonable for the built-in potential for this system. For thick SiO₂ layers, the capacitive reactance of the insulating layer becomes appreciable.

The majority carrier concentrations for the 10 Å and 20 Å SiO₂ cells are about 10^{15} cm⁻³, and the depletion width is 1 μm, which is larger than we had previously assumed.

The junction capacitance of the silver MIS structures was too small to measure accurately in the presence of the large diode leakage currents. Therefore, the data shown for the Ag/SiO₂Cu₂O cells are of a very preliminary nature but they show the same trends in voltage and frequency dependence as those of the aluminum cells.

In the case of the gold MIS structures, it was not possible to measure a depletion layer capacitance. The measured capacitance was independent of voltage and presumably was due to the SiO₂ insulating layer. In the case of gold contacts on Cu₂O, which are ohmic, there is no band bending and no depletion layer.

Figures 9 and 10 show the frequency dependence of the capacitance for aluminum and gold MIS structures. The rapid rise of

Figure 7. Plots of $1/C^2$ vs. Applied DC Bias for Al/SiO₂/Cu₂O MIS Cells of Various Oxide Thicknesses

Figure 8. Plots of $1/C^2$ vs. Applied DC Bias for Ag/SiO₂/Cu₂O MIS Cells of Various Oxide Thicknesses

Figure 9. Frequency Dependence of the Junction Capacitance of an Al/SiO₂/Cu₂O MIS Cell

Figure 10. Frequency Dependence of the Junction Capacitance of an Au/SiO₂/Cu₂O MIS Cell

capacitance with decreasing frequency may be due to the presence of slow traps, possibly both in the insulating layer and at the $\text{SiO}_2/\text{Cu}_2\text{O}$ interface. Future studies of the temperature dependence of the capacitance may help to identify the nature of these traps.

E. Discussion of MIS Properties

For the metals studied, the MIS cells gave increased values of V_{oc} and barrier height in the order of decreasing work function of the metal, i.e., gold, silver, copper and aluminum. Thus it appears that in these cells, the work function of the metal plays a dominant role in determining the degree of band bending and V_{oc} , and that interface states play a secondary role. The results of the capacitance measurements are also consistent with the view that there is no band bending with Au in MIS cells, some with Ag and more with Cu and Al. However, there is an indication that some deep traps are present.

IV. HETEROJUNCTIONS

During this quarter some studies were made of heterojunctions of ZnSe on Cu_2O . More recently work has been initiated on heterojunctions of oxides on Cu_2O . The ZnSe work was a continuation in our laboratory by Mr. L. Papadimitriou of some studies initiated by him in Thessalonika, Greece.

Because of the lack of reliable data on electron affinities, the choices of candidates for heterojunctions on Cu_2O are not obvious. Two possible choices are ZnSe and ZnS. The energy band diagrams of these heterojunctions are shown in Figure 11 for n-ZnSe/p- Cu_2O and in Figure 12 for n-ZnS/p- Cu_2O . The built-in potential for the ZnSe/ Cu_2O junction is estimated to be 0.57 eV and that for ZnS/ Cu_2O junction is estimated as 0.61 eV.

The ZnSe films were prepared by thermal evaporation in the Ultek ultrahigh vacuum system. First, ZnSe films on glass were prepared in order to determine the conditions for producing transparent, low resistance coatings. Studies were made of different rates of deposition, film thickness, and substrate temperature. The resistivity values obtained ranged from $10^{10} \Omega\cdot\text{cm}$ to $10^3 \Omega\cdot\text{cm}$. It was found that the best combination of the above parameters for good transparency and low resistivity was:

Film thickness	$\sim 1500 \text{ \AA}$
Rate of deposition	$\sim 500 \text{ \AA/min}$
Substrate temperature	Room temperature

Figure 13 shows the transmission curves on two ZnSe films of the same thickness and rate of deposition but with different substrate temperatures, room temperature and 150°C . The thickness was $1650 \text{ \AA}$ and the deposition rate was $550 \text{ \AA/min}$. The resistivity

Figure 11. Energy Band Diagram for a ZnSe/Cu₂O Heterojunction

Figure 12. Energy Band Diagram for a ZnS/Cu₂O Heterojunction

Figure 13. Optical Transmission of ZnSe Films Prepared at Different Temperatures

was $4.1 \times 10^4 \Omega \cdot \text{cm}$ for the sample prepared at room temperature and $1.1 \times 10^6 \Omega \cdot \text{cm}$ for the 150°C case.

Heterojunction cells of $\text{ZnSe}/\text{Cu}_2\text{O}$ were prepared by similar methods. The junctions usually possessed rectifying characteristics but the photovoltaic effect was often small or not observable. The series resistance of the cells was usually high. Both the diode and photovoltaic characteristics of the junctions tended to change with time.

Deposition of ZnSe on Cu_2O at room temperature, which produced the lowest resistivity ZnSe films on glass, usually resulted in no photovoltaic effect. Better photovoltaic results were obtained when the ZnSe deposition on Cu_2O was done with a substrate temperature of 150°C . Figure 14 shows the dark and illuminated I-V characteristics of a $\text{ZnSe}/\text{Cu}_2\text{O}$ heterojunction cell prepared at 150°C and I-V curves for the same cell after two months.

These results may be explainable by assuming that a chemical reaction occurs during the deposition at 150°C and that this or other reactions occur after preparation. Thus the photovoltaic effect in these cells as currently prepared may not be due to a $\text{ZnSe}/\text{Cu}_2\text{O}$ heterojunction. One possibility is that a $\text{Cu}/\text{Cu}_2\text{O}$ junction is generated by a chemical reaction during the processing. It is planned to investigate these possibilities by Auger/ESCA studies.

Figure 14. Dark and Light I-V Characteristics of a ZnSe/Cu₂O Heterojunction Cell After Preparation at 150°C and After Two Months

V. SUMMARY

In order to investigate Cu_2O MIS structures and heterojunctions, we have expanded our capabilities to prepare and measure these types of cells. The apparatus for the preparation of thin films includes ion beam sputtering equipment and electron beam evaporation equipment, as well as expanded capabilities for thermal evaporation. The cells can be studied by a variety of techniques including I-V measurements in the dark and under illumination, capacitance-voltage measurements as a function of frequency and temperature, spectral response, surface photovoltage for evaluation of diffusion length, etc. We also have some access to Auger/ESCA equipment for surface analysis.

The MIS structures chosen for initial study were Metal/ SiO_2 / Cu_2O using the metals Au, Ag, Cu, and Al and various thicknesses of SiO_2 . The results indicate that the band bending and V_{oc} depend primarily on the work function of the metal and that interface states play a secondary role. It appears that the preparation of the cells can be further improved upon to secure more definitive results. It is planned to investigate other oxide interlayers. Further Auger/ESCA studies are planned to determine the influence of chemical changes.

Work on heterojunctions on Cu_2O has been initiated. The first system explored was $\text{ZnSe}/\text{Cu}_2\text{O}$. It appears that chemical changes at the interface have an important effect on the properties. In the future, the emphasis will be placed on heterojunctions of Cu_2O with other oxide semiconductors.

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