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Novel Ways of Depositing ZnTe Films by a Solution Growth Technique

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SUMMARY

A. Objectives

The objective of this research is to develop novel processes for the deposition of thin (<500Å) ZnTe layers suitable as transparent ohmic contacts for CdS/CdTe solar cells. The processes developed for ZnTe deposition may also be applied to deposition of other II-VI semiconductor layers for polycrystalline thin film solar cells.

B. Discussions

The objectives of this research were fulfilled by the Institute of Energy Conversion through the performance of the following research tasks: 1) ZnTe film deposition from aqueous solutions by electrodeposition, exchange reaction with CdTe, and solution growth; 2) Doping the ZnTe films with Cu during deposition; 3) Structural, optical and electrical characteristics of the ZnTe films; and 4) CdS/CdTe/ZnTe solar cells fabrication and characterization.

Of the three approaches investigated for the formation of the ZnTe films, we were only successful in forming the ZnTe films using the electrochemical method. The films were deposited from an aqueous bath containing 0.1 M $ZnCl_2$ and about 10^{-4} M TeO_2 with the substrate externally short circuited to a Zn counter electrode, forming a galvanic cell. Controlling both the pH and Te^{+4} ion concentration in the solution were vital for the formation of single phase ZnTe films. Controlled p-type doping during ZnTe film growth was achieved by adding an aqueous solution of $CuCl_2$ and triethanolamine (TEA) to the ZnTe bath. The properties of the films were characterized, and CdS/CdTe/ZnTe solar cells were fabricated.

C. Conclusions

Cu-doped ZnTe films, <1000Å, were reproducibly deposited for the first time by an electrochemical method. A CdTe/CdS/ITO/glass substrate is externally short circuited to a zinc counter electrode in an aqueous bath consisting of $ZnCl_2$ and TeO_2 to complete an electrochemical cell. Control of both pH and TeO_2 concentration was necessary to deposit single phase ZnTe films. A copper complex was added to the bath to control the ZnTe conductivity and dope the films p-type. CdTe/CdS solar cells using the ZnTe:Cu as the primary contact to the CdTe have achieved efficiencies of ~9%.

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SECTION 1.0

INTRODUCTION

1.1 BACKGROUND

CdTe/CdS thin film solar cells have been shown to be a promising candidate for photovoltaic power modules (1,2). Small area devices have been made by a variety of techniques with efficiencies of about 10%, of which several methods are believed to be easily scaled for large area cell production. One approach uses a n-i-p cell structure which takes advantage of the intrinsic nature of CdTe and solves the contact problem to the CdTe by using a p+ ZnTe:Cu layer deposited by vacuum evaporation (1). Further, CdTe based cells are candidate wide bandgap cells for tandem structures using $CuInSe_2$ based cells (3). However, in the n-i-p CdTe cells, the absorption in the CdTe cell with the heavily doped ZnTe layer is about 60% from 400 nm to 1300 nm. This reduces the transmission through the device and limits the short circuit current of the narrow bandgap cell. It is thus necessary to prepare ZnTe having the required electronic properties but which are continuous and thin (<500Å) to minimize the absorption. This approach has the additional advantage of permitting alternative CdTe device designs which incorporate optical enhancement. It is not possible to deposit continuous thin ZnTe films by vacuum evaporation. Thus, there is a need to develop an alternative deposition process to deposit thin ZnTe films which can improve device performance, and possibly be more cost effective for commercial applications. The objective of the research described in this report is to develop novel processes for deposition of semiconductor window/contact layers for thin film polycrystalline solar cells, specifically a method of depositing thin semitransparent ZnTe films for CdTe solar cells.

1.2 TECHNICAL APPROACH

In order to achieve the objectives, the Institute of Energy Conversion carried out a program of investigation and analysis to deposit ZnTe films according to the following research tasks:

a. IEC investigated methods for depositing II-VI films using solution growth and electrochemical techniques. The processes investigated included direct ZnTe film formation on the CdTe surface and an exchange reaction with CdTe surface forming a ZnTe layer where Cd is replaced by Zn. Doping of the ZnTe film with Cu to control the resistivity was an integral part of the process.

b. IEC characterized the structural, optical and electrical properties of the ZnTe films using established techniques of x-ray diffraction and total reflection and transmission measurements. Emphasis was placed on obtaining single phase films and on minimizing sub bandgap absorption.

c. IEC fabricated superstrate CdS/CdTe/ZnTe solar cells to characterize the contact properties of the ZnTe films on a working device. The properties of the devices using the ZnTe contact were compared to devices using Au or Cu/ITO as the CdTe contact. Emphasis was placed on total transmission through the device and resistivity losses due to the contact.

1.3 OUTLINE OF REPORT

In this report we describe an electrochemical method for depositing ZnTe films doped with Cu onto a ITO/CdS/CdTe cell and device result using the ZnTe contact. Also included in the report is a brief summary of other unsuccessful approaches that were investigated for depositing ZnTe films. In Section 2.0 the growth and characterization of ZnTe film deposited by an electrochemical method are discussed. In Section 3.0 the device performance of CdTe cells using ZnTe as the primary contact is presented. In Section 4.0 other approaches for depositing ZnTe films from solution are described and difficulties with growth of the films are discussed. Finally in Section 4.0, future work on transparent window/contacts is presented.

SECTION 2.0

ELECTRODEPOSITION OF ZnTe FILMS

ZnTe films were deposited electrochemically from an unstirred aqueous bath containing 0.1 M $ZnCl_2$ and about 10^{-4} M TeO_2. The TeO_2 was brought into solution by dissolving it in a solution of NaOH. The pH of the bath was adjusted to 3 to 4 using dilute HCl. A bath temperature of 50-60°C was maintained during the deposition. A conductive substrate, in most cases a 7059/ITO/CdS/CdTe sample, and a zinc electrode were externally short circuited to form an electrochemical cell as shown in Figure 2-1. A similar method has been used by Bhattacharya et al. (4) and Murali et al. (5) for depositing CdTe and CdSe thin films respectively. The Zn electrode acts as both a source of electrons for the cathodic reaction and as a source of Zn^{+2} ions via the following reaction:

$$Zn \rightarrow Zn+2+2e^-;\ E^{\circ}_{ox}=0.76V,$$

where E°_{ox} is the oxidation potential with respect to the standard hydrogen electrode. The TeO_2 was brought into solution by dissolving it in a solution of NaOH, forming Na_2TeO_3. Once the bath pH is reduced with HCl, $HTeO_2^+$ (tellurous acid) may form. In either form, the Te is in the +4 oxidation state from which it must be reduced by 4 electrons. In this report we refer to the dissolved Te forms collectively as "Te^{+4} ions" to simplify the discussion. Depositions were performed at pH from 2.0 to 5.0 of the solution and for Te^{+4} ion concentration from 10^{-3}M to 10^{-5}M to obtain the optimum condition for growth of ZnTe films. The deposition rate was monitored to determine when the Te^{+4} ions were exhausted.

Figure 2-2 shows the time dependence of the ZnTe film deposition onto a 7059/ITO/CdS/CdTe substrate. The bath consisted 0.1 M $ZnCl_2$ and about 10^{-4} M TeO_2 with a total volume of 50 mℓ, the bath temperature was 50-60°C, the area of the substrate was ~1.9 cm^2, the bath pH was 3.1 to 3.3, and the Zn electrode area was 6 cm^2. A linear growth rate was observed till about 1 hour of deposition after which it tended to saturate, suggesting exhaustion of the Te^{+4} ions in solution. On addition of TeO_2 to the solution and adjusting the pH, growth of ZnTe would proceed in a similar fashion confirming that the growth was limited by the availability of Te^{+4}. It can be observed from Figure 2-2 that about 0.3 μm ZnTe film could be grown on a ~1.2 x ~1.6 cm substrate from a single deposition within a period of about 90 minutes using a solution volume of 50 mℓ.

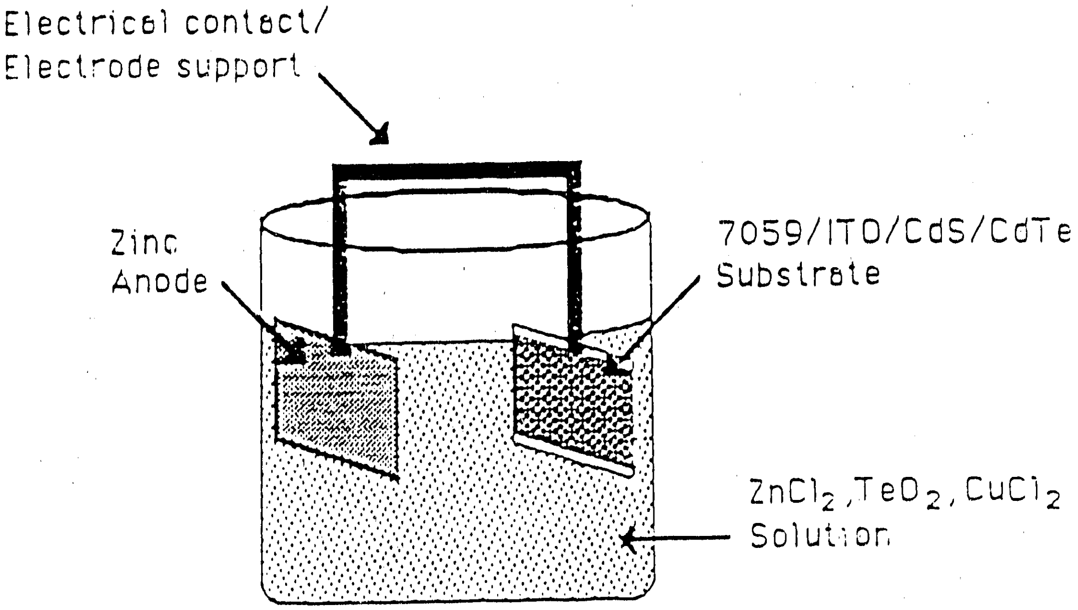

Figure 2-1. Schematic diagram of electrochemical cell used to deposit ZnTe films on a conductive substrate.

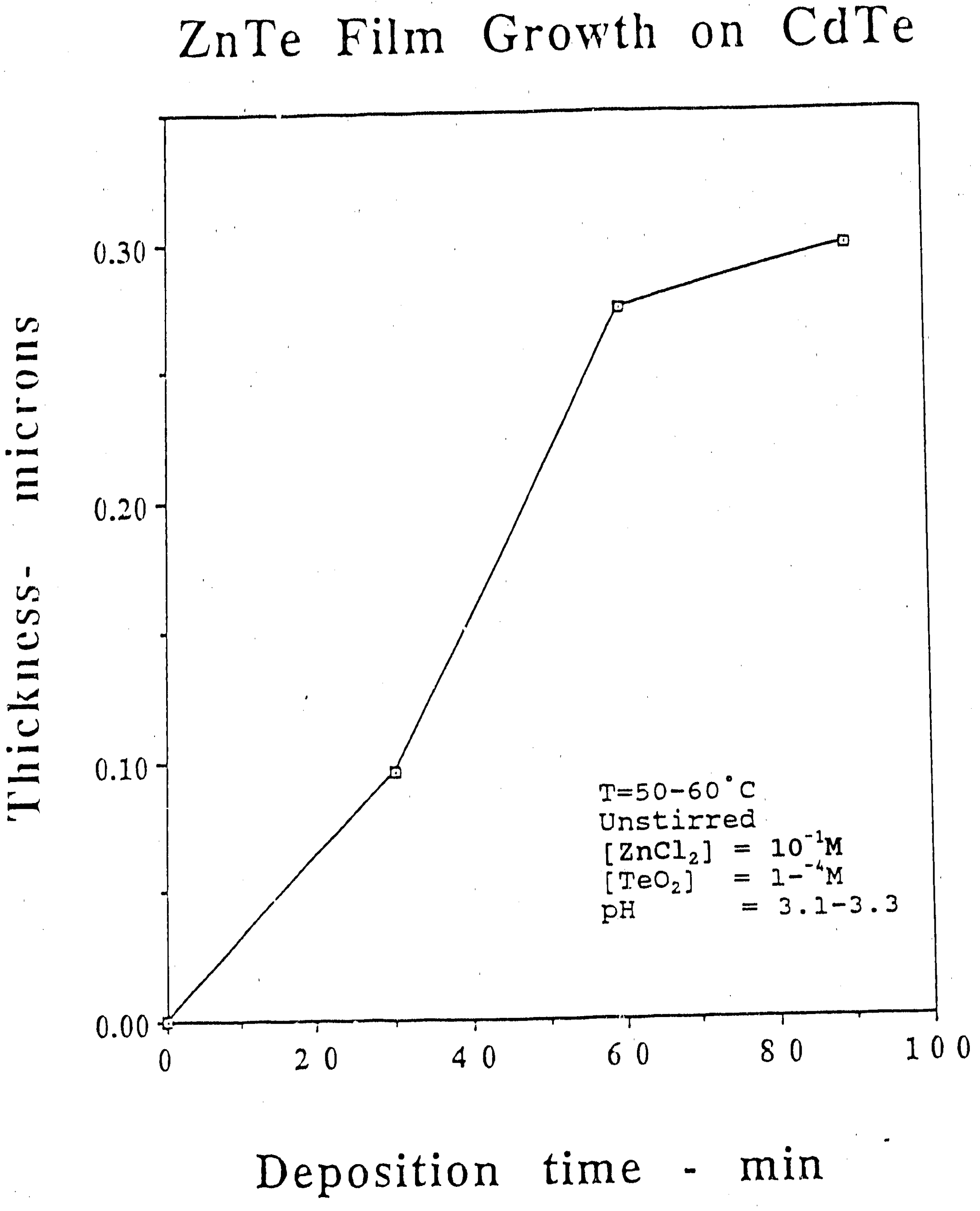

Figure 2-2. **ZnTe film deposited as a function of deposition time.**

Controlling both the pH and Te^{+4} ion concentration in the solution were vital for the formation of the ZnTe films. A pH range of 3 to 4 and a Te concentration of 10^{-4}M or less were found to yield single phase ZnTe films. At lower pH, free Te deposited and above pH=5, the solution became cloudy, probably due to formation of $Zn(OH)_2$. Increasing the Te^{+4} ion concentration in solution increased the deposition rate, however, mostly Te was found to deposit instead of ZnTe. Since Zn is more electropositive than Te, the Zn would be less likely to deposit as the element from its ions in an electrochemical bath than Te from its ions. Hence, low concentration of the Te^{+4} ions in solution must be maintained compared to the Zn^{+2} ions, so that a diffusion-controlled process takes place for the Te^{+4} ions to reach the cathode.

To confirm that single phase ZnTe was directly formed during the growth, films 200-300 nm thick were deposited and analyzed by X-ray diffraction. The X-ray diffraction pattern of a typical ZnTe film deposited on 7059/ITO/CdS/CdTe is shown in Figure 2-3. The sharp (111) peak at $2\theta=25.19^\circ$ establishes the formation of a highly oriented cubic phase ZnTe film. The other peaks are due to CdTe, CdS, and ITO. The x-ray diffraction measurement, while indicating the presence of a well oriented ZnTe film, does not rule out the existence of a small quantity of excess Te or Zn. The presence of excess Zn is unlikely in the presence of Te^{+4} ions since it is more electropositive and less likely to deposit. Excess Te, on the other hand, is possible at higher TeO_2 concentrations, but is detectable at low levels by reduced optical transmission in the films. Such reduction was not observed for undoped ZnTe films.

P-type doping of the ZnTe film with Cu was achieved by adding a copper complex to the ZnTe bath. For this, an aqueous solution of $CuCl_2$ and triethanolamine (TEA) was added to the ZnTe bath to give Cu concentrations from 10^{-5} to 10^{-3} M. For Cu concentrations outside this range either no Cu was incorporated into the film or other compounds such as Cu_2Te were formed. Figure 2-4 shows the time dependence of Cu-doped ZnTe growth on a 7059/ITO/CdS/CdTe substrate. The bath consisted 0.1 M $ZnCl_2$, about 10^{-4} M TeO_2 and 10^{-4} M of $CuCl_2$ in TEA with a total volume of 50 mℓ, a pH of 3.1 to 3.3, a substrate area of ~2.5 cm^2 and the same Zn electrode described previously. The ZnTe:Cu films grow in a similar manner as the undoped films. The electrical properties of the ZnTe:Cu films were not directly measured but were inferred from cell results where fill factors were as high as 74%. The Cu serves two purposes; 1) to dope the ZnTe and 2) to provide a source of Cu to dope the CdTe p-type during a subsequent 150°C air anneal used in the cell optimization process.

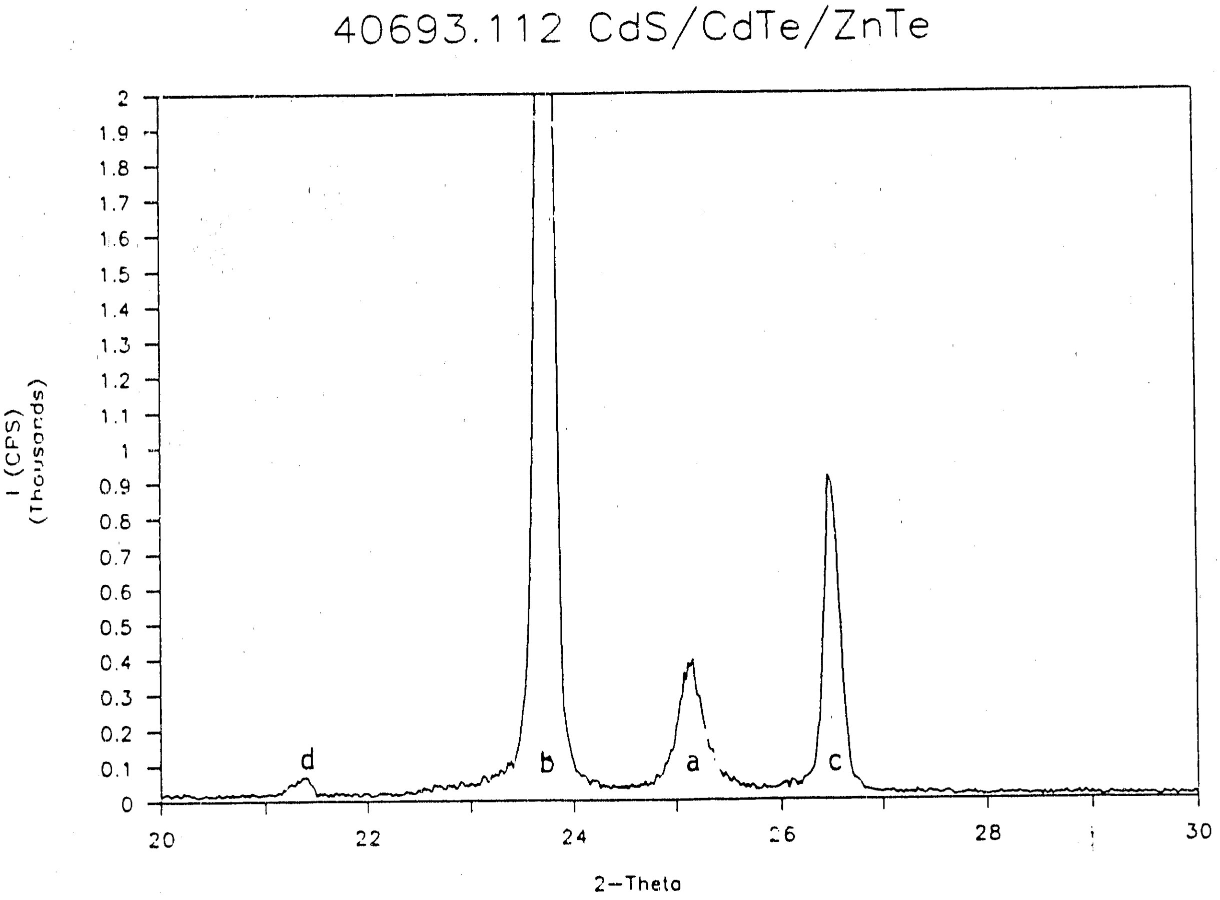

Figure 2-3. X-ray diffraction spectrum of ZnTe film on 7059/ITO/CdS/CdTe showing the ZnTe (111) peak (a), the CdTe (111) peak (b), the CdS (002) peak (c), and an ITO peak ((012)In_2O_3)(d).

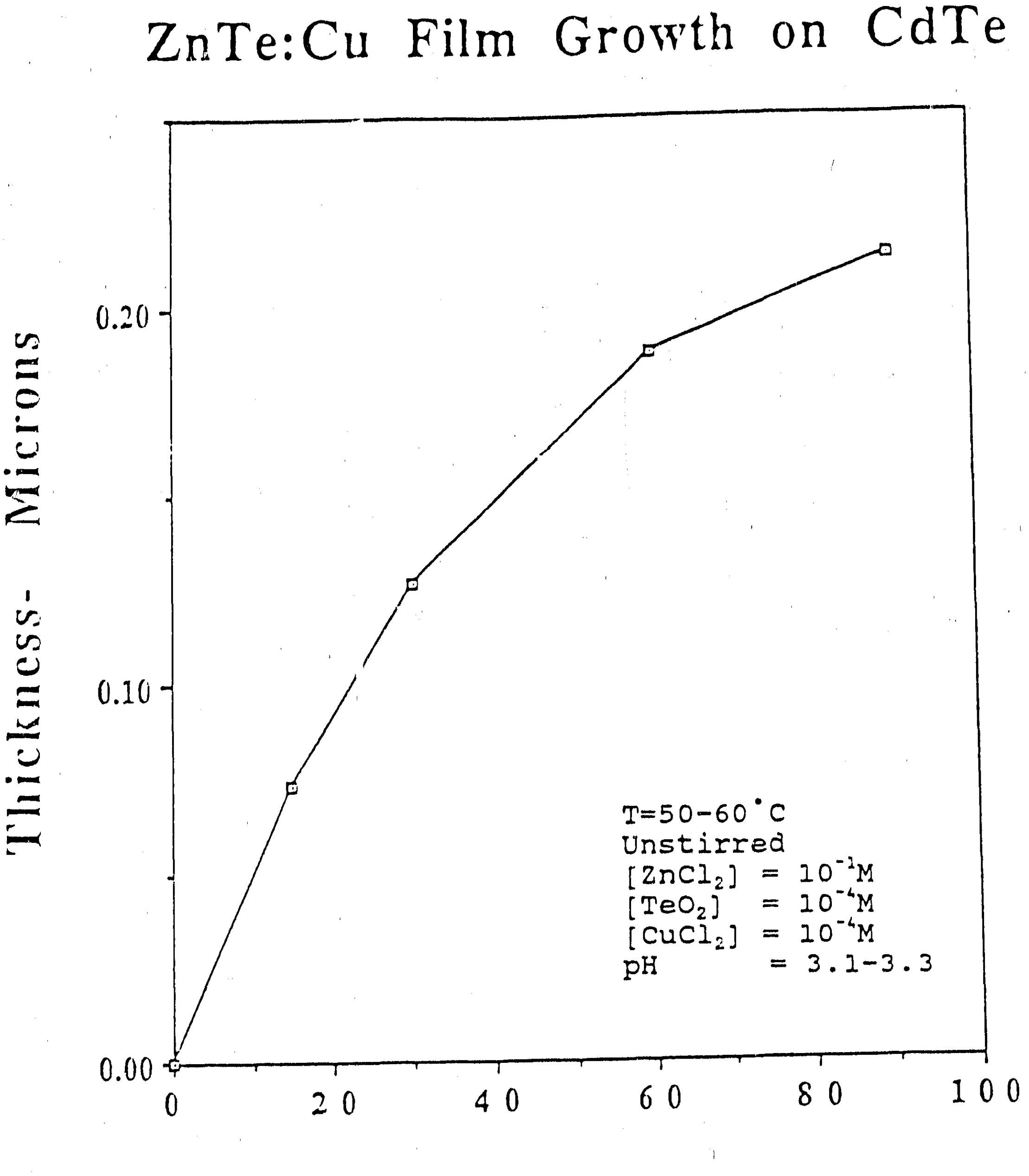

Figure 2-4. ZnTe:Cu film deposited as a function of deposition time.

SECTION 3.0

CdTe DEVICES WITH A ZnTe CONTACT

For solar cell fabrication, ITO/CdS/CdTe substrates were coated with a solution of methanol containing 1% by weight of $CdCl_2$ and then heat treated in dry air for 30 minutes at 400°C, as described elsewhere (6). A mild, 0.01%, Br_2 methanol etch for 5 seconds was then used to remove any surface residue resulting from the heat treatment since it was found that ZnTe did not grow uniformly on unetched surfaces. A ZnTe film, from 30 to 100 nm thick, doped with Cu was then electrochemically deposited as described above. A robust contact, either Cu/Au, Ni or ITO, was deposited onto the ZnTe:Cu to complete the solar cells. The area of the device was typically 0.08 cm^2 and was defined by scribing. The performance of the cells was optimized in a 150°C air heat treatment (6). I-V characteristics were measured at 32°C under 87.5 mW/cm^2 ELH illumination and the current was normalized to 100 mW/cm^2. A summary of the device results for cells with the best I-V parameters having different contacts to the ZnTe:Cu and three different ZnTe:Cu thickness is presented in Table 3-1. The device results are independent of the contact metal applied to the ZnTe:Cu indicating that no additional Cu is required to optimize the device. It should be noted that all thin film CdTe based solar cells having efficiencies greater than 9% utilize Cu, Ag, or Au in the processing, either as a p-type dopant for CdTe (7) or as a constituent of the ohmic contact to CdTe (6,8-15). Several devices with ZnTe:Cu/Cu/Au contacts were measured at SERI and the I-V and quantum efficiency curves are shown in Figures 3-1 and 3-2.

Table 3-1

Device Parameters for CdS/CdTe/ZnTe:Cu Cells Using ITO/Ni, Cu/Au, and Ni robust contacts measured under ELH simulation at 32°C but normalized for AM1.5 spectrum at 100 mW/cm^2.

V_{oc} (V)	J_{sc} (mA/cm^2)	FF (%)	Eff (%)
	ITO/Ni		
0.766	18.4	54.6	7.7
0.679	19.4	54.2	7.1
0.709	16.6	68.3	8.0
	Cu/Au		
0.754	17.8	60.5	8.1
0.616	20.5	55.1	6.9
0.703	17.2	69.0	8.3
	Ni		
0.676	20.6	51.7	7.2
0.628	18.6	54.9	6.4
0.616	15.6	74.8	7.2

Sample: 691.231-1

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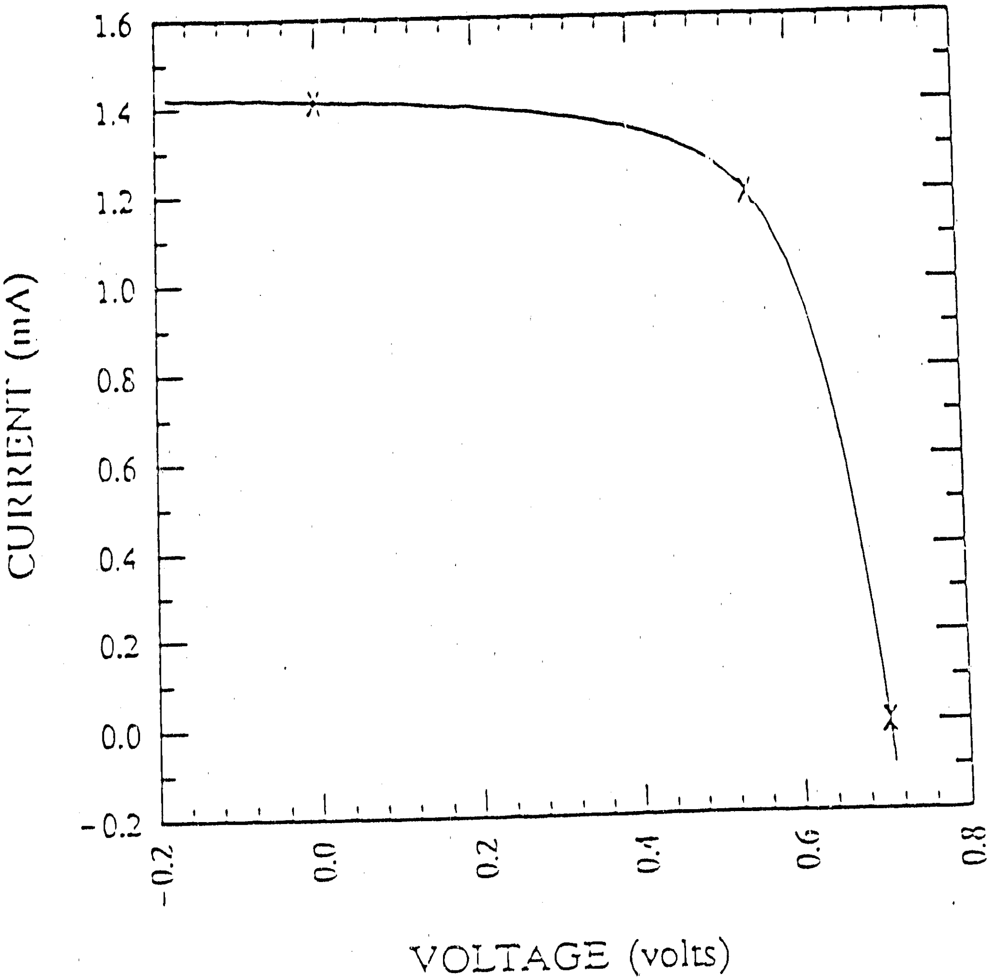
Temperature = 25.0°C

Area = 0.0752 cm²



V_{oc} = 0.7051 volts

J_{sc} = 18.79 mA/cm²

Fill factor = 65.66 %

Efficiency = 8.7 %

I_{sc} = 1.413 mA

P_{max} = 0.654 mW

I_{max} = 1.197 mA

V_{max} = 0.5465 V

Figure 3-1. I-V characteristics of CdS/CdTe/ZnTe:Cu cell #40691.231-1 using electrodeposited ZnTe:Cu layer measured under AM 1.5 global solar simulation at SERI.

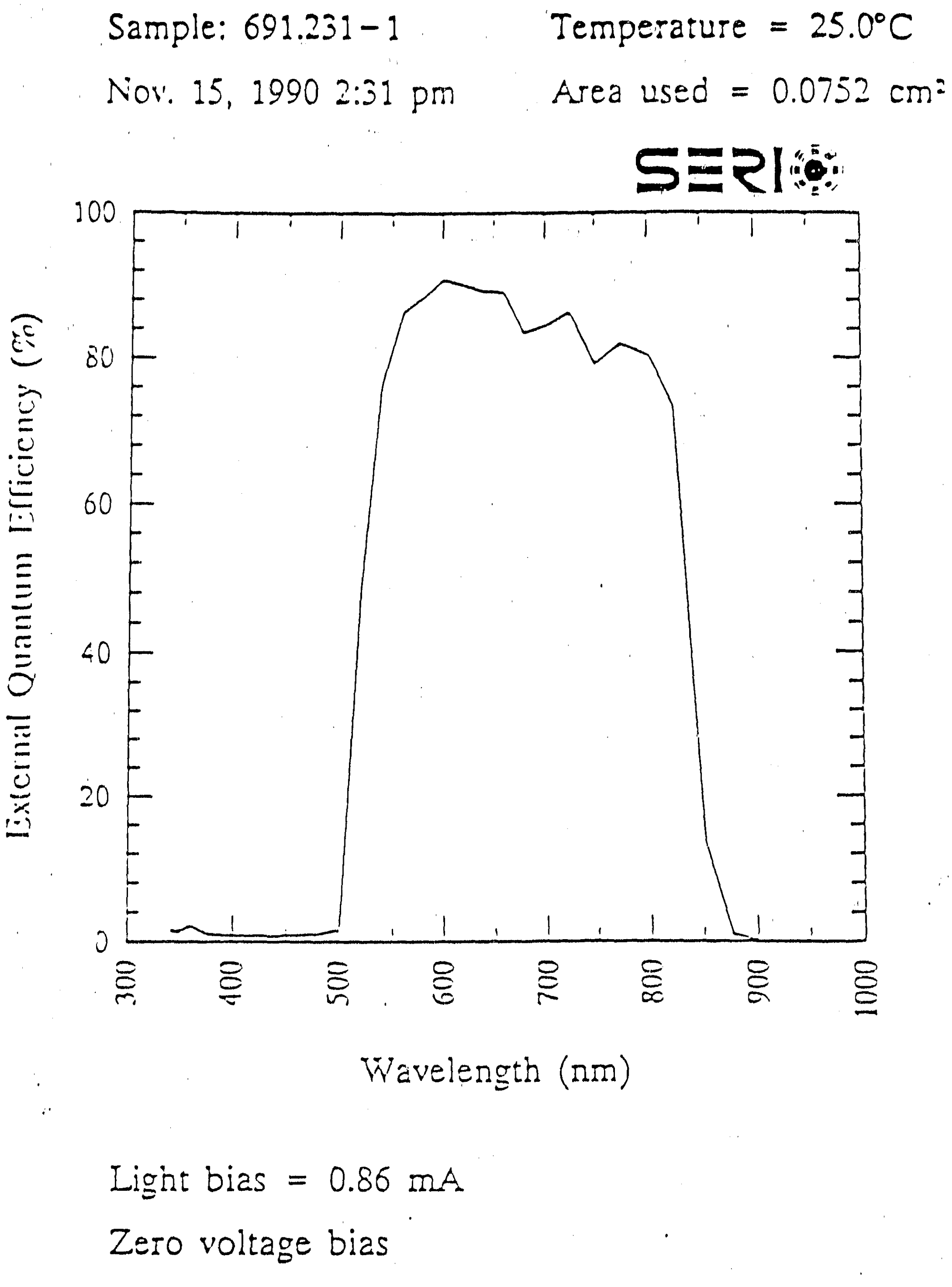


Figure 3-2. **Quantum efficiency versus wavelength of CdS/CdTe/ZnTe:Cu cell #40691.231-1.**

To evaluate the cells for tandem cell application the total transmission and reflection was measured for different deposition times, hence, thicknesses. The normalized transmission, T/(1-R), is shown in Figure 3-3 for two samples grown under similar conditions to that of Figure 2-4 but for 15 min and 60 minutes. The film deposited for 15 min was ~30 nm thick and had 70% normalized transmission at 900 nm. The film deposited for 60 min was ~110 nm thick and had 50% normalized transmission at 900 nm. These films bracket the deposition time/thickness used to make contacts to CdTe and the transmission results can be compared to the results reported by Meyers et al. (3) where the maximum T/(1-R) was ~30% at 900 nm. Thus, the electrodeposited ZnTe:Cu has higher transmission than the evaporated ZnTe:Cu demonstrating the advantage of the very thin electrodeposited films. The ZnTe:Cu thickness and the Cu doping level need to be optimized further to reduce absorption.

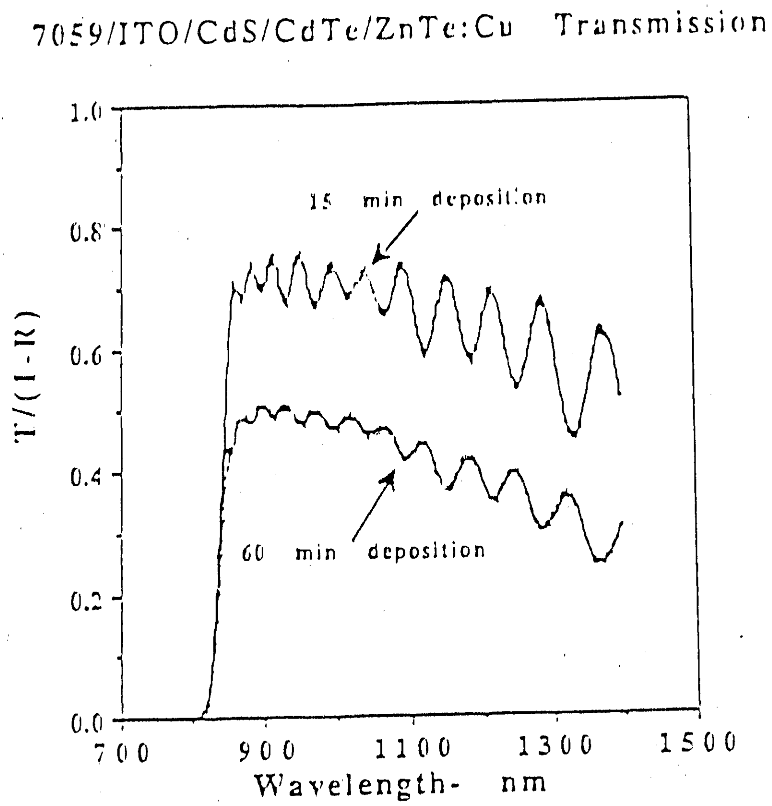


Figure 3-3. Normalized transmission, T/(1-R) spectrum of 7059/ITO/CdS/CdTe/ZnTe:Cu for two ZnTe:Cu deposition times.

SECTION 4.0

OTHER APPROACHES TO GROWING ZnTe

Three approaches for the formation of ZnTe were investigated: 1) forming a ZnTe layer from the CdTe surface by an exchange reaction; 2) growing a ZnTe film from solution in an analogous manner to CdS; and 3) forming ZnTe by successive deposition of Zn and Te followed by heat treatment. None of these approaches were successful; however, a brief description of each approach is presented below.

4.1 EXCHANGE REACTION

An exchange reaction was investigated to form the ZnTe film from a CdTe surface according to:

$$(CdTe)_{solid} + (ZnLn)_{aq} \rightarrow (ZnTe)_{solid} + (CdLn)_{aq}$$

where Ln is an appropriate ligand. Table 4-1 lists the reaction routes pursued along with the corresponding standard free energies of reaction. The following growth conditions were varied: solution temperature- 25 to 80°C, concentration- 0.03 to 3 molar, pH- 2.3 to 8.5 (basic solution only for $ZnCl_2$ reaction) and reaction time- 5 to 10 min. For these three reaction paths no ZnTe films were formed.

4.2 SOLUTION GROWTH

Two different chemistries were explored to form ZnTe by solution growth:

1. A solution of Na_2Te was prepared from Te powder, NaOH and $Na_2S_2O_4$ as a source of Te^{-2} ions. $Zn(C_2H_3O_2)_2$ was added as the source of Zn with NH_3 as a complexing agent. A small amount of Te precipitate was formed in the solution but no ZnTe was formed.

2. Zn metal was used as a reducing agent and a source of Zn using the following:
   $Zn(solid) \rightarrow Zn^{+2} + 2e^-$
   $TeO_2 + 6e^- \rightarrow Te^{-2}$
   $Zn^{+2} + Te^{-2} \rightarrow ZnTe(solid)$

Zinc dust was gradually added to a solution of TeO_2 and NaOH. This resulted in the formation of ZnO and crystalline Te. The solution had a reddish color suggesting the presence of elemental tellurium rather than the Te^{-2} ions needed for the reaction.

Table 4-1
Reaction Routes Followed to Deposit ZnTe by
Surface Exchange Reaction

Standard Free Energy
<u>of Reaction (Kcal/mole)</u>

<u>Cyanide Route</u>

$CdTe_{(s)} + Zn(CN)_4 \rightarrow ZnTe_{(s)} + Cd(CN)_4$

-25.6 -22.92 -28.05 -25.78 -5.31

<u>Chloride Route</u>

$CdTe_{(s)} + ZnCl_2 \rightarrow ZnTe_{(s)} + CdCl_2$

-25.6 -0.84 -28.05 -3.43 -5.04

<u>Acetate Route</u>

$CdTe_{(s)} + Zn(C_2H_3O_2)_2 \rightarrow ZnTe_{(s)} + Cd(C_2H_3O_2)_2$

-25.6 + -2.06 -28.05 -2.06 -2.45

4.3 Te AND $ZnCl_2$ REACTION

Experiments to form ZnTe by successive formation of Te and Zn followed by annealing were performed. Conductive substrates of 7059/Pt were used on which Te was electrochemically deposited from dilute solutions of TeO_2, 10^{-4} to 10^{-5} M, at a pH of 2 to 3 with an Al or Zn electrode externally connected to the substrate. The samples were then coated with $ZnCl_2$ in MeOH and heat treated in H_2/Ar at 400°C for 30 minutes. No ZnTe films were formed but PtTe was formed as determined from XRD. Further experiments using this approach with CdTe substrates did not yield ZnTe.

SECTION 5.0

CONCLUSIONS AND FUTURE WORK

Cu-doped ZnTe films, <100 nm, were deposited for the first time by an electrochemical method in which a CdTe/CdS/ITO/glass substrate is externally short circuited to a zinc counter electrode in an aqueous bath consisting of $ZnCl_2$ and TeO_2 to complete an electrochemical cell. Control of both pH and TeO_2 concentration was necessary to deposit single phase ZnTe films. A copper complex added to the bath allowed controllable p-type doping of the ZnTe films. CdTe/CdS solar cells using the ZnTe:Cu as the primary contact to the CdTe achieved efficiencies of 8.7% verified by SERI. The optical transmission of cells using ZnTe:Cu made in this manner is higher than on cells using evaporated ZnTe:Cu, making this an attractive contacting method for tandem cells and for use of optical back surface reflection enhancement techniques in conjunction with thin CdTe.

We have also obtained promising results using this method to deposit ZnSe from an aqueous bath containing $ZnCl_2$ and SeO_2 directly onto Mo/7059 and $CuInSe_2$/Mo/7059 substrates. We have also deposited CdS films onto ITO/7059 superstrates for CdTe/CdS cells. These applications of the method demonstrate its utility for depositing II-VI compound semiconductor window layers. Future work will include fabrication and characterization of ZnSe and CdS layers and evaluation of devices using them.

SECTION 6.0

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SECTION 7.0

ABSTRACT

Results of a research program to develop a method to deposit thin, <50 nm, ZnTe films as a transparent back contact to CdTe solar cells are presented. Several approaches to grow ZnTe film from aqueous solutions were investigated including electrodeposition, formation of ZnTe from the CdTe surface by an exchange reaction and direct formation of ZnTe from a chemical solution. Methods to dope the ZnTe films with Cu were an integral part of the program. Of the three approaches, only the electrochemical method was successful. The films were deposited from an aqueous bath containing $ZnCl_2$ and TeO_2 with the substrate externally short circuited to a Zn counter electrode to form a galvanic cell. Controlling both the pH and Te^{+4} ion concentration in the solution were critical for the formation of single phase ZnTe films. A copper complex was added to the bath to control the ZnTe conductivity and dope the films p-type. CdTe/CdS solar cells using the ZnTe:Cu as the primary contact to the CdTe have achieved efficiencies of ~9%.

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16. Abstract (Limit: 200 words)

This report describes a research program to develop a method for depositing thin (< 50 nm) ZnTe films to form a transparent back contact to CdTe solar cells. We investigated several approaches to growing ZnTe films from aqueous solution, including electrodepositing the films, forming the films from the CdTe surface by an exchange reaction, and forming the films from a chemical solution. Methods to dope the ZnTe films with Cu were another integral part of the program. Of the three approaches, only the electrochemical method was successful. In this approach, the films were deposited from an aqueous bath containing $ZnCl_2$ and TeO_2 with the substrate externally short-circuited to a Zn counter electrode to form a galvanic cell. Controlling both the pH and Te^{+4} ion concentration in the solution were critical for forming the single-phase ZnTe films. A copper complex was added to the bath to control the ZnTe conductivity and dope the films p-type. CdTe/CdS solar cells were developed incorporating the ZnTe:Cu as the primary contact to the CdTe. These cells have exhibited efficiencies of ~9%.

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