DEVELOPING ANALYSIS CRITERIA TO ADJUST THE GROWTH OF CdS AND CdTe THIN FILMS USING THE PLD TECHNIQUE, FOR SOLAR CELL PURPOSES

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The goal of this research is to obtain technical information of the conformation of the CdTe/CdS junctions deposited on an ITO/Glass substrate. Their physical conditions to deposit each single layer will enable appropriate configurations to be applied on electronic devices such as alternative solar cells on silicon technology. Firstly, CdS thin films were deposited upon an ITO/Glass substrate at room temperature, with 9,000 shots at the rate of 10 shots/s, at different pressures of 20, 40, 60, 65, 70, 75 and 90 mTorr. Afterwards, CdTe thin films were grown on glass substrates all with 100,000 shoots at the rate of 10 shots/s, at the same pressure of 100 mTorr, but at different substrate temperatures: 100, 200, 300 and 400 ^oC. For CdS at the rate of 0.1 nm/s good films of 150 nm were produced in 1,500 seconds (25 min), while for CdTe at the rate of 0.0696 \approx 0.07 nm/s, films of 696 nm in 10,000 s (166.66 min) were produced. Their morphologies, crystallite size and resistivities were also studied to propose the optimized CdTe/CdS junction for a photovoltaic application.

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1. Introduction

For a long time to make the most of solar energy on our planet has been sought by using non-pollutant renewable energies through the development of technology that takes advantage of solar radiation through solar cells. CdTe is used as an active layer together with CdS as an option for a window layer of electromagnetic energy [1-11]. The CdTe film is preferred due to its good adsorption characteristics, since this material adsorbs approximately 90% of the photons with energy higher than its forbidden band with thicknesses between 1-2 μ m in the region of the near infrared [12]. It has an adsorption coefficient of the order of 10⁵ cm⁻¹ [1-3], a forbidden band width of approximately 1.5 eV of photoconversion. With it, efficiencies near 16.5% have been obtained in the laboratory for glass substrates covered with Transparent Conductive Oxides (TCO) although efficiencies of 30% have been predicted for photovoltaic devices with this material [13, 14]. The deposit conditions, methods and annealing enable higher efficiencies [13]. The dependence on the

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power conversion on the semiconductor's forbidden band limited by the black body radiation reveals ground power generation under spectra of 1.5 to 1 eV. The optimum forbidden band energy is of 1.5 eV which is similar to that of semiconductors like CdTe, InP, GaAs and CuInSe₂ [15].

Previous works indicate that the substrate temperature influences the grain size and the crystallinity of the CdTe films. However, it cannot be arbitrarily raised since re-evaporation could be produced affecting the film's composition and the concentration of the carriers [3,4]. When recrystallization and grain growth is required, a post-deposit annealing is normally necessary. This improves the resulting CdTe film's crystalline quality and the electric performance [4]. Another important aspect is that the life expectancy of CdTe carriers is small (1x 10⁻⁷ s or less) with a mobility range from 10 to 100 cm⁻²V⁻¹s⁻¹. The small optical absorption length of systems II-VI is also present in relatively in carrier devices with insignificant diffusion length. The diffusion length of the granulated CdTe film is between 1 and 5 μ m, and thus the active layer thickness is the same [15, 16]. The reduced thickness in the absorbed layer increments the intensity of the electrical power and therefore a great fraction of carriers will be generated inside the depletion layer, making easier the efficient collection of carriers.

Among the methods to obtain photovoltaic CdTe for the development of thin film solar cells is the cathodic pulverization through RF magnetron, metal-organic chemical vapor deposition, electrochemical methods, pulsed laser deposition (PLD), among others [4-6]. The main advantages of this last method are that the deposit rates can be controlled in the rage of 0.1-0.25 μ m/min, in addition to fabricating the type n CdS window layer and the type p CdTe adsorbing layer in-situ just by exchanging the source objectives in the same vacuum chamber for the fabrication of the p-n junction. Since the in bulk forbidden bands for CdTe and CdS are 1.5 and 2.4 eV, respectively, the photons with energies inside this range can reach the CdTe layer, contributing to the photocurrent of the cell. CdS acts as a window layer in relation to the spectral response in regions of shorter wavelength. It plays an important role in the crystallinity of CdTe. It facilitates the formation of great forbidden bands of mixed crystalline layers of CdTe_{1-x}S_x near the CdS / CdTe interface, improving the properties of the absorbing layer.

In this work, we report a study of the required conditions of pressure, shot number and substrate temperature to obtain the thickness and the conformation of the CdTe / CdS junction deposited on a Indium-Tin Oxide (ITO)/Glass substrate with photovoltaic applications.

2. Experimental and Characterization

2.1. Experimental conditions for the deposit of CdS thin films

Cadmium sulfide thin films were deposited on glass substrates covered with Indium-Tin Oxide. To find the best experimental conditions for the deposit of the CdS films, a series of 8 samples was made by varying the pressure in the vacuum chamber and keeping the number of shots constant in the samples Sa1 through Sa7 from 9,000 and 15,000 pulses at 10 Hz. The substrate was at room temperature during the deposit, see Table 1.

SAMPLE	$\begin{array}{c} \textbf{SUSTRATE} \\ (SiO_2 = Glass) \end{array}$	MATERIAL	SHOTS (x10 ³) at 10 Hz	TEMPERATURE (⁰ C)	ARGON PRESSURE (mTorr)
Sa1	ITO/Glass	CdS	9	RT	20
Sa2	ITO/Glass	CdS	9	RT	40
Sa3	ITO/Glass	CdS	9	RT	60
Sa4	ITO/Glass	CdS	9	RT	65
Sa5	ITO/Glass	CdS	9	RT	70
Sa6	ITO/Glass	CdS	9	RT	75
Sa7	ITO/Glass	CdS	9	RT	90
Sa8	ITO/Glass	CdS	15	RT	40

Table 1. List of experimental conditions for the deposits of the CdS layers

2.2 Experimental conditions for the deposit of CdTe thin films

CdTe films were deposited on glass substrates. In order to find the best experimental conditions for the deposit of the CdTe, we made two series of samples. One series, depositing the CdTe films on glass, varying the substrate temperature and keeping the pressure constant in the vacuum chamber and the number of shots on the sample, 100,000 a 10 Hz, see Table 2 (samples Sb1 to Sb5). Other CdTe films were deposited on a CdS/ITO/Glass substrate, varying the number of deposition pulses: 37,500, 75,000 and 100,000 at 10 Hz. The substrate temperature and the chamber pressure were kept constant, see Table 2 (samples Sb6 to Sb8).

SAMPLE	SUSTRATE	MATERIAL	SHOTS (x10 ³) at 10 Hz	TEMPERATURE (⁰ C)	ARGON PRESSURE (mTorr)
Sb1	$SiO_2 = Glass$	CdTe	100	RT	100
Sb2	SiO_2	CdTe	100	100	100
Sb3	SiO ₂	CdTe	100	200	100
Sb4	SiO_2	CdTe	100	300	100
Sb5	SiO_2	CdTe	100	400	100
Sb6	ITO/Glass	CdTe/CdS	37.5/9	400 / RT	100 / 40
Sb7	ITO/Glass	CdTe / CdS	75/9	400 / RT	100 / 40
Sb8	ITO/Glass	CdTe/CdS	100 / 15	400 / RT	100 / 40

Table 2. List of experimental conditions for the realized deposits of the CdTe and CdTe/CdS layers

Replicas of sample Sb8 were submitted to the sequential processes of $i\mu\mu$ ersion in CdCl₂ solutions and thermal annealing, where the concentration of CdCl₂ was variable in order to improve the morphologic and structural properties of the CdTe film. Three different solutions were tested as it is shown on Table 3.

SATURATION %	CdCl ₂ GRAMS
75%	0.75
50%	0.5
40%	0.4

Table 3. Saturation percentage of CdCl₂ solutions

The solutions were prepared by dissolving the desired amount of $CdCl_2$ in 20 mL of deionized water and mixing this solution with ethanol until a total volume of 100 mL is reached. Generally, a typical solution of $CdCl_2$ and ethanol is used since ethanol is quickly evaporated, leaving behind a layer of $CdCl_2$ on the sample. We used a small percentage of water to guarantee the uniform distribution of $CdCl_2$ in the whole volume due to its insolubility on alcohol without compromising too much the sedimentation time.

3. Results and discussion

On Fig. 1, the behavior of seven CdS films is presented. The films were grown at 9,000 shots at a rate of 10 shots/s as it was designed on Table 1. The respective band gaps of each film are also calculated, associated with the growth pressure. As it can be seen, the thicker films were the films at an Argon pressure of 20 and 40 mTorr. This was a feedback for the analysis and thus we decided to create sample Sa8 by elevating the number of shots from 9,000 to 15,000, with the

goal of having a better balance in terms of thickness and optical transmission. The optical transmission of this last sample is presented on Figure 2, in comparison to the substrate used of glass covered with ITO.



Fig. 1. Optical transmission of seven CdS samples grown with 9 K (9,000) shots at a rate of 10 Hz, varying the deposition pressure

A selection of appropriate conditions for the use of a CdS layer grown by PLD to be implemented on a photo cell can be appreciated on Figure 2. The corresponding band gap is of 2.33 eV and its thickness, 150 nm. This means that this film can be utilized as a semiconductor window as far as its optical transmission is in the wavelength range between 500 and 1000 nm.



Fig. 2. Transmission spectra as a comparative reference between ITO/Glass system and the Sa8 CdS sample

The CdTe films grown according to the also feedback design shown on Table 2, are grouped the following way. The first five samples were grown on a glass substrate and we determined the favorable conditions for the formation of CdTe layers with a thickness of 700 nm through PLD. These conditions are the corresponding to maintaining the substrate at 400°C during the deposit (Sb5), an Argon pressure of 100 mTorr and 100,000 shots. This allowed to feedback the analysis and decided to create three more samples, Sb6, Sb7 and Sb8 with the parameters of Table 2, searching for a balance in terms of thickness and optical transmission, for an optimum absorbent performance.

Figure 3 shows two types of responses obtained for the material layers of CdS and CdTe, samples Sa8 and Sb8. These responses are the absorption coefficients (labeled 1 and 2) and the depths of the light penetration (labeled 3 and 4), both pairs of graphs are in function of the wavelengths in the range of 300 to 1000 nm, on logarithmic scales. The structure formed by contact/CdTe/CdS/ITO/Glass is referred to as superstrate. From the absorption coefficients, the band gap of each film is determined, while the depth of the light penetration is the inverse of the

absorption coefficient. As it can be seen in figure 3, the 150 nm layer of CdS has a sufficient photoconductive performance, allowing also the transmission of the wavelengths between 550 and 1000 nm. These energies interact with the absorption threshold of the next material, which is CdTe at 827 nm, labeled on the black line with a 2. It can be noted that the convenient thickness for the CdTe film in an active photovoltaic bilayer as this, would be of the order of 15 μ m for the total absorption. This correlation of parameters justifies the ranges of thicknesses published for photovoltaic cells with these materials.



Fig. 3. Absorption and light penetration depth for the independent CdTe and CdS thin films

Figure 4 shows the numerical calculation of the band gap for the CdTe film (Sb8) with the Tauc technique. This sample had a thickness of 696 nm and its band gap was of 1.5 eV.

A monitoring of the superficial morphologies of the layers of ITO, CdS and CdTe using AFM is shown on Figures 5, 6 and 7. On Figure 5, for the ITO surface we can observe a uniform morphology with disperse aggregates with partial roughness is no greater than 20 nm. On Figure 6, for the CdS surface, the partial roughness does not exceed 32 nm and the morphology is very uniform in a similar fashion as in Figure 5. This CdS surface shows a greater aggregate density, defined grain forms and cracking at a greater scale, in the limits of the resolution of AFM. On Figure 7 the morphology of the material layer of CdTe is shown by AFM. The distribution of aggregate sizes is less uniform than for the case of CdS and its partial roughness is lower than 24 nm.



Fig. 4. Calculation of the direct band gap of the CdTe thin film (Sb8) deposited through PLD, with a substrate kept at 400 °C through deposition, 100,000 shots at a rate of 10 shots/s and a pressure of 100 mTorr.



Fig. 5. Basic morphology of the ITO/Glass system (the substrate) where the CdS layer is deposited. (a) is a Top view and (b) is a perspective view of same region

Figure 6, the CdS film was grown on 25 minutes at a rate of 10 shots/s producing a thickness of 150 nm (0.1 nm/s), measured by ellipsometry. By performing surface XRD to this material information of the immediate inferior layer (ITO) was obtained. The area of the image is of $1\mu^2$.



Fig. 6. Top view (a), and perspective view (b) of the system CdS/ITO/Glass. Growth conditions at 40 mTorr of the chamber Argon pressure. The substrate was kept at room temperature with 15,000 shots during 1,500 seconds.

Fig. 7 corresponds to the morphology of the most external layer, CdTe, from a top view and in perspective of the same region with a height scale of 243 nm in a surface of 1 μ^2 . The CdTe film was grown on 166.66 minutes at 10 shots/s, producing a thickness of 697 nm (00696 \approx 0.07 nm/s) measured by ellipsometry.



Fig. 7. Top view (a), and perspective view (b) of the system CdTe/CdS/ITO/Glass. Grown conditions for the last layer (CdTe), 100 mTorr of the chamber Argon pressure. The substrate was kept at 400 °C with 100,000 shots during 10,000 seconds.

A more adequate technique which shows the morphologic differences in relation to the deposit temperatures of the CdTe layers is the scanning electronic microscopy. Figure 8 shows the micrographs of the samples Sb1 to Sb5 of Table 2. Image Sb1 is a very minute granular background, which we suppose is due to the lack of thermal annealing in situ during the formation stage. In this morphology the charge carriers should go through a greater number of grain frontiers to be electrically conducted on the CdTe film. The Sb2 image of Figure 8 shows a cracked morphology. We suppose this is due to the thermal annealing effect in situ during the formation stage promoting the coalescence of nanocrystals to form aggregates with a certain concurrence and cracking as a function of the intensity of the temperature at which the substrate is maintained. On the Sb3 image, a certain distribution of material obstacles of sizes of the order of the scale is shown on the micrograph. On the Sb4 image there is a homogenization at greater scale, although in granular form, where crystals of greater dimension are appreciated, confirming the still polycrystalline nature of the sample. Finally, the Sb5 image at 400°C is considered the most appropriate for solar cell applications.



Fig. 8. CdTe morphology changes, when the substrate temperatures were room temperature, 100, 200, 300 and 400 °C for Sb1, Sb2, Sb3, Sb4 and Sb5, respectively. All films were grown 100,000 shots and 100 mTorr of the chamber Argon pressure during 10,000 seconds.

The study of these material layers of CdS and CdTe through X-Ray Diffraction provides the following results. Figure 9 shows three X-Ray Diffractograms comparatively, labeling the material systems represented. The glass substrate covered with ITO corresponds to the pattern # PDF 39-1058, by depositing CdS, it coincides with pattern # PDF 02-0549 with a hexagonal structure, but the signals of ITO corresponding to the planes (222) and (400) are still very notorious. The CdTe is deposited on glass, since its growth depends only the deposit conditions and not on the substrate. The glass amorphous signal is shielded due to the sufficient thickness of $696 \approx 700$ nm of the cubic polycrystalline CdTe, which corresponds to # PDF 65-1082. All the crystallite sizes are compiled on Table 4, calculated with the Debye-Scherrer formula:

$$D = \frac{0.9\lambda}{\beta \cos\theta}$$

The XRD diffractograms depicted in Figure 9 pertain to the films without thermal annealing or chemical-thermal treatment.



Fig. 9. XRD characterization of the as ground material layers systems: CdTe /Glass, CdS/ITO/Glass and ITO/Glass, with growth conditions indicated.

The CdS and CdTe layers were analyzed under two thermal annealing at different temperatures that are shown on Figs. 10 and 11. Fig. 10 depicts a treatment of 1 hour at 150°C, where it can be observed that the intensities decrease, but the modulation is such that the medium width of the most intense peak almost remains constant, varying only 1nm with respect to the as ground samples for both, CdS and CdTe. The sizes of all the nanocrystals are on Table 4.



Fig. 10. XRD characterization for the thermal annealing for the material layers systems, in a forming gas atmosphere during 1 h at 150 °C: CdTe /Glass, CdS/ITO/Glass and ITO/Glass, with growth conditions indicated.

On Figure 11 the treatment was of 1 hour at 300°C. The intensity for CdS was not altered with respect to the previous treatment and there was a light increase of intensity for CdTe with respect to the previous treatment. There was a modulation such that the medium width of the most intense peak almost remains constant. The sizes of the nanocrystals did not change with respect to the as ground samples for both, CdS and CdTe. Their values are also shown on Table 4 in comparison to all the measured samples.



Fig. 11. XRD characterization for the thermal annealing of the material layers systems, in a forming gas atmosphere during 1 h at 300 °C: CdTe /Glass, CdS/ITO/Glass and ITO/Glass, with growth conditions indicated.

As it is shown on Figs. 9, 10 and 11 the characteristic peaks for ITO, (222) and (400) are marked on the CdS layer of 150 nm; while for the CdTe layers of around 700 nm the amorphous glass –where it was grown– signal is perfectly shielded.

A chemical-thermal treatment reported on literature is the described on the experimental section which consists on immerse the CdTe films on saturated solutions of cadmium chloride and exposing them to a thermal annealing. We found that the intermediate value of the essayed concentrations, denoted by 40, 50 and 75% showed a better efficiency to increase the crystallite grain size. A higher concentration could promote a molecular corrosion causing the size of the CdTe crystals to decrease, compared to the growth obtained with the intermediate concentration (50%). These values are shown on Table 4.



Fig. 12. Chemical immersion in three different solutions of 40, 50 and 75% of $CdCl_2$ followed by thermal annealing during 20 minutes at 400°C with a nitrogen atmosphere.

Figure Involved	CdS	CdTe
Figure 9 as ground	$\beta=0.47237^{\circ} \Rightarrow D=18 \text{ nm}$	$\beta=0.46485^{\circ} \Rightarrow D=18 \text{ nm}$
Figure 10 Therm. Aneal.1	$\beta=0.49615^{\circ} \Rightarrow D=17 \text{ nm}$	$\beta=0.45277^{\circ} \Rightarrow D=19 \text{ nm}$
Figure 11 Therm. Aneal.2	$\beta=0.47766^{\circ} \Rightarrow D=18 \text{ nm}$	$\beta=0.46575^{\circ} \Rightarrow D=18 \text{ nm}$
Figure 12 CdCl ₂ (40%)		$\beta=0.4641^{\circ} \implies D=18 \text{ nm}$
Figure 12 CdCl ₂ (50%)		$\beta=0.3458763^{\circ} \Rightarrow D=25 \text{ nm}$
Figure 12 CdCl ₂ (75%)		$\beta=0.390496^{\circ} \Rightarrow D=22 \text{ nm}$

Table 4. Calculated crystallite sizes of CdS and CdTe

Samples of pure CdTe films, Sb7 and Sb8, as well as the immersed samples with different concentrations of CdCl₂ Sb8: 40, 50, 75% were selected to analyze their electrical behavior, measuring sheet resistance (R_{sh}), Resistivity (ρ), Contact resistance (R_c) and Contact Specific Resistance (ρ_c). The values are displayed on Table 5.

Sample	Thickness	Sheet Resistance	Resistivity	Contact	Contact Specific
	(nm)	(Ω/sq)	$(\Omega \cdot cm)$	Resistance	Resistance
	t	$R_{\rm sh}$	ρ	R _c	$ ho_c$
Sb7	500	1.39×10^{12}	$6.95 \text{ x} 10^7$	$4.60 ext{ x10}^9$	$6.00 \text{ x} 10^4$
Sb8	696	4.63×10^{11}	$3.22 \text{ x}10^7$	$1.14 \text{ x} 10^9$	$1.11 \text{ x} 10^4$
Sb8:40%	696	$3.32 \text{ x}10^{11}$	$3.32 \text{ x}10^6$	3.35×10^8	$1.33 \text{ x} 10^3$
Sb8:50%	696	$9.75 ext{ x10}^{10}$	$9.75 \text{ x}10^5$	$5.12 \text{ x} 10^8$	$1.06 \text{ x} 10^4$
Sb8:75%	696	$4.07 \text{ x}10^{11}$	$4.07 \text{ x}10^6$	1.26×10^9	$1.55 \text{ x}10^4$

Table 5. Thickness, sheet resistance, resistivity, contact resistance, contact specific resistance for samples Sb5 at 40, 50 and 75% concentrations, Sb7 and Sb8

4. Conclusions

The PLD technique is convenient and beneficial for the deposit of CdS thin films at average rates of 0.1 nm/s and for CdTe deposition rates of the order of 0.07 nm/s, achieving layers of 150 nm and 700 nm, respectively. This led us to prepare a deposition scheme to correlate the optical absorption and light penetration depth, related with the thicknesses and the optical transmissions to predict a better performance of photovoltaic devices. Likewise, the as ground CdTe films have a resistivity of a magnitude order higher than the reported range [15], the chemical-thermal treatment lowers the resistivity a magnitude order, where the concentration of CdCl₂ is of 50% and the higher crystallite size is 25 nm. Also, there is a strong effect of substrate temperature on the morphology of CdTe films grown at a rate of around 0.07 nm/s.

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