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# Effect of the combination of Cu and CdTe plasmas on the structural and optical properties of CdTe:Cu thin films deposited by laser ablation

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## ABSTRACT

In this work, thin films of semiconducting copper doped cadmium telluride (CdTe:Cu) were obtained by laser ablation. The effect of the combination of CdTe and Cu plasmas by the simultaneous ablation of Cu and CdTe targets, on the properties of CdTe:Cu films, when the copper plasma parameters (mean kinetic ion energy and density) are varied while CdTe plasma parameters were kept constant, is analyzed. The mean kinetic ion energy of Cu plasmas was increased from 73 to 100 eV. Cu plasma density increased linearly with mean kinetic ion energy. Mean kinetic ion energy and density of CdTe were kept constant at 75 eV and  $2 \times 10^{14}$  cm<sup>-3</sup>, respectively.

The chemical composition was measured by energy dispersive X-ray spectroscopy. It was observed that Cu content incorporated into the CdTe lattice varied from 1.4 to 1.6 at%. Structural characterization revealed the growth of a CdTe hexagonal structure with no preferential orientation for a reference CdTe film. For samples grown combining Cu and CdTe plasmas, it was observed that increasing density of Cu plasma produces a crystalline orientation of the films in the (110) direction. Optical properties of the samples were obtained by UV–Vis spectroscopy, the data was used to estimate band gap using Tauc plots yielding values that decreased slightly form 1.47 eV for the reference CdTe film, to 1.41 eV for the film grown using the highest Cu plasma density. Scanning electron microscopy was used to study surface morphology, were rougher surfaces were observed as the Cu content in the films was increased.

## 1. Introduction

Cadmium telluride (CdTe) has shown to be an excellent material for optoelectronic applications because of its remarkable optical absorption properties in the IR-visible region of the spectrum, together with electrical conductivity. CdTe has been synthesized by several physical and chemical techniques. However, deposition by laser ablation has proven to produce high quality CdTe films. Depending on the laser produced plasma conditions, the crystalline structure can be controlled even at room substrate temperatures.

CdTe is a semiconductor with a direct band gap of 1.5 eV which is

optimal for single-junction solar cells, at which the maximum theoretical efficiency for this cells can be found [1]. CdTe has a high absorption coefficient >  $5 \times 10^5$  cm<sup>-1</sup> which added to the band gap leads to a high quantum yield from ultraviolet to 825 nm light [2]. When using CdTe thin films as absorbing layer, just 1 µm is enough to absorb around 90% of the incident spectrum while having more than 3 µm ends up giving a constant value of efficiency [3]. CdTe thin films can exhibit both *n* and *p*-type conductivity, a cadmium excess produces *n*-type while tellurium excess produces *p*-type conductivity [2]. Among thin film deposition methods, CdTe can be deposited by close space sublimation (CSS), vapor transport deposition (VTD), sputtering, spray

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deposition [2], pulsed laser deposition (PLD) [4,5], electrodeposition [6]. PLD has demonstrated to be a suitable deposition method since it presents some advantages, for instance, it enables the deposition of compound materials by combining plasmas, it is also suitable for the growth of very thin films due to the possibility of having a precise control on the deposition rate. It could enable stoichiometry transfer from target to film if the right experimental conditions are used [7,8]. Furthermore, manipulation of plasma parameters in laser ablation deposition processes, offers the advantage of having control on the chemical composition of the films. In addition, as it is well known, the laser ablation technique allows the synthesis of high crystalline and optical quality thin films; which can be achieved with a precision that is not possible when using conventional deposition techniques such as sputtering or thermal evaporation.

As grown CdTe has shown to have a very high resistivity going from  $10^5$  to  $10^8 \Omega$  cm and it has been proved that Cu doping on *p*-type CdTe greatly reduces resistivity. Besides, Cu favors a better Ohmic contact and increases *p* carriers [9]. In the present work, we report the effect of the combination of CdTe and Cu plasmas by the simultaneous ablation of Cu and CdTe targets on the properties of CdTe:Cu films when the copper plasma parameters (mean kinetic energy and density of ions) were varied while CdTe plasma parameters were kept constant.

#### 2. Experimental details

CdTe:Cu thin films were grown on Corning glass substrates by multitarget pulsed laser deposition. A 1" CdTe target obtained by compressing high purity CdTe powders (99.99% from Sigma-Aldrich), was attached to a 2" Cu target (99.99% from Kurt J. Lesker). The targets were simultaneously ablated, in order to obtain a combined plasma. Fig. 1 depicts the used PLD configuration. A Nd:YAG laser with 600 mJ of maximum output energy, at a wavelength of 1064 nm, 6 ns pulse width, and repetition rate of 10 Hz, was used for the experiments. The beam was divided into two equal beams by the use of a beam splitter. Each laser beam were focused on their respective target while they were rotating at 15 rpm to avoid drilling. Thin films were deposited in vacuum at a pressure of  $4 \times 10^{-6}$  Torr and 10 min of ablation time. The distance from Cu and CdTe targets to substrate was 5 and 4.8 cm, respectively, for all the experiments.

Prior to each deposition, the individual laser produced plasmas were analyzed by Langmuir probe characterization. Measurements were carried out using a 6 mm diameter planar Langmuir probe biased at - 48 V. The probe current was obtained by measuring the voltage drop across a 20  $\Omega$  resistor; using a Tektronix 500 MHz digital oscilloscope. For these measurements, the substrate was replaced by the planar probe. The plasma parameters such as mean kinetic ion energy and plasma density were estimated by the time of flight curves (TOF). The mean kinetic energy of the ions ( $E_k$ ) present in the plasma was estimated using the TOF curve data and the procedure described in reference [10]. The calculation uses the following relationship:



Fig. 1. PLD system configuration.

$$\langle E_k \rangle = \frac{mL^2}{2} \frac{\int_0^T t^{-2} I(t) dt}{\int_0^T I(t) dt}$$

where m is the mass of the ion, L is the target to probe distance, and I(t) is the probe current as a function of time. For the plasma ion density calculation, the procedure described in [11] was used:

$$N_p = \frac{I_{max}}{evA},$$

where  $I_{max}$  stands for the maximum value of the current (saturation current), e is the electron charge, v is the plasma flow velocity and A is the area of the probe. A reference curve was set for the CdTe plasma; this curve was reproduced for all the experiments in order to have the same CdTe plasma conditions in all depositions. The laser fluence on the CdTe target was kept constant at  $1.7 \text{ J/cm}^2$ . Cu plasma parameters were varied by the modification of the fluence on the Cu target. Three samples were grown combining the plasmas with the aim of changing the copper content incorporated into the films. One CdTe sample was grown as reference.

UV–Vis measurements were obtained using a Genesys 10uv, Thermo Uv–Vis spectrophotometer. X ray diffraction (XRD) was carried out in a PANalytical Empyrean diffractometer. Raman spectroscopy measurements were taken with a Horiba Jobin Yvon HR 800 Raman spectrometer with the 532 nm excitation line, the beam was focused using a  $50 \times$  objective lens. Photoluminescence spectrum was obtained at room temperature (RT) using a Horiba spectrofluorometer model fluoromax-4. Morphology was observed by scanning electron microscopy (SEM) with a Tescan Mira 3 and atomic concentration measurements of the samples were determined by electron dispersive spectrometry (EDS) with an installed Bruker Quantax Xflash 6 and analyzed with Espirit software. The acceleration voltage was 20 kV; the same condition as for the images.

# 3. Results and discussion

It has been reported that, when doping/alloying a material by combining two plasmas in pulsed laser deposition, the variation of ion density of the dopant/alloying results in the modification of the final thin film composition [12,13], thus incorporation of a dopant/alloying material could be controlled by means of the ion density. On the other hand, interaction of both plasmas could have an influence on the growing films due to scattering processes. As it was mentioned in Section 2, for the present work, pulsed laser produced plasmas of CdTe and Cu were combined in order to achieve different copper contents into the CdTe films.

Three films were grown using different plasma densities for Cu, one CdTe sample was deposited in order to use it as a reference. Fig. 2 shows the TOF curves of the Langmuir probe measurements for Cu plasmas. The inset shows the TOF curve for CdTe. Mean kinetic energy  $(E_k)$  and density  $(N_p)$  of ions where calculated using the procedure described in references [11,14]. The CdTe plasma parameters were kept constant at 75 eV and  $2 \times 10^{14}$  cm<sup>-3</sup> respectively, for all the experiments as described in the experimental section. Plasma density depends on the maximum current value (saturation current) of the TOF curve [11], thus a change in the height of the TOF curve will produce a modification in the ion density of the plasma [12]. As it can be seen in Fig. 2 different heights in signal probe for Cu ions (Cu<sup>+</sup>, accounting for every copper ion species) were used; from these data the plasma density values were calculated to be in a range of  $3.4-6.5 \times 10^{13}$  cm<sup>-3</sup>. Plasma density was increased by increasing the Cu fluence from 2.9 to 3.7 J/ cm<sup>2</sup>. It can be expected that using an order of magnitude of difference between densities for CdTe and Cu plasmas would produce Cu incorporation in low quantities.

Regarding  $E_k$  for Cu plasmas, the values increased from 73 to 100 eV, showing a linear relation with  $N_p$ . In general, it is hard to independently vary  $E_k$  without  $N_p$  modification and vice versa, as they are



Fig. 2. TOF curves for the CdTe and Cu plasmas.



Fig. 3. Cu plasma density as a function of mean kinetic energy of  $Cu^+$ .



Fig. 4. Atomic contents from EDS measurements, of Cd, Te and Cu in the films, as a function of the Cu plasma density determined by TOF curves.

strongly correlated and depend on the ablation conditions [15]. A detailed study of density dependence on mean kinetic energy for Cu plasmas is out of the scope for the present work. Fig. 3 shows the linear relation between  $N_p$  and  $E_k$  for Cu plasmas.

In order to evaluate the Cu incorporation into the CdTe films, EDS measurements were performed. Several measurements were carried out, over different large areas on the thin film to get an average of the



**Fig. 5.** X ray diffraction patterns of the samples. The inset on the left shows the intensity ratios for the (110) to (002) peaks. The inset on the right shows the  $2\theta$  shift for the Cu containing films.



Fig. 6. Raman spectra of the samples grown at different Cu<sup>+</sup> density.



Fig. 7. Tauc plots for the samples. The inset shows the band gap values as a function of Cu plasma density.

atomic concentrations. Signals from both, CdTe:Cu thin film and Corning glass substrate, were observed in the EDS spectra, since the thickness of the samples is under 600 nm (@ 20 kV). However, the atomic percent ratios between Cd, Te and Cu hold, since they are distributed in the same analysis volume (thin film) and there are no traces



Fig. 8. PL spectra for the samples.

of these elements in the Corning glass substrate. The ratio values were correlated using the Bruker software Esprit v1.91, selecting to analyse only the Cd, Te y Cu contents; as well as calculating the concentration ratios by hand, from the EDS spectrum with all the elements. The analysis was performed after a calibration procedure, which includes using a Cu sample (99.99% pure), prior to data recording. The applied correction method was interactive PB-ZAF (standardless).

Fig. 4 shows the atomic composition for Cd, Te and Cu as a function of Cu<sup>+</sup> density. It can be noticed that the CdTe sample grew with a Te excess, having atomic concentration values of 58 and 42 at% of Te and Cd, respectively. When Cu is incorporated into the CdTe volume, the difference of Cd and Te concentrations decreases. These chemical composition values are consistent with previous reports for pulsed laser deposited CdTe thin films [15]. When Cu and CdTe plasmas are combined using a Cu plasma density of  $3.4 \times 10^{13}$  cm<sup>-3</sup> a value of 1.4 at% of Cu is detected in the film, further increase in Cu<sup>+</sup> density up to a value of  $6.5 \times 10^{13}$  cm<sup>-3</sup> slightly increases the Cu atomic content in the films.

Concerning to Cd and Te contents, note that for a Cu<sup>+</sup> density of  $3.4\times10^{13}\,\text{cm}^{-3}$  a Cd rich film is obtained with a content of 54.7 at%. It might be assumed that Te reacts with Cu, producing Cu-Te compounds at some stage of the deposition process. However, for Cu<sup>+</sup> densities of 4.9 and  $6.5 \times 10^{13}$  cm<sup>-3</sup> the situation is reversed, Cd/Te ratio is less than 1 which means higher Te content. It is worth mentioning that mean kinetic energy of Cu<sup>+</sup> changes with density, which means that plasma interactions are influenced by both energy and density of ions. Besides, it should be remembered that in general the ablation phenomenon is extremely complex and not completely understood, due to a series of equilibrium and non-equilibrium processes occurring during the deposition experiments. In order to have a better understanding of combined plasmas interactions additional diagnosis techniques such as optical emission spectroscopy, that allows identification of excited species contained in the plasma [15] or time of flight mass spectroscopy, that allows ions identification, should be carried out. Identification of excited species and types of ions together with its density and mean kinetic energy determination help to shed light on the reaction mechanisms that could yield the formation of a specific compound.

X ray diffraction patterns are shown in Fig. 5. As it can be seen hexagonal CdTe thin films were obtained. The pattern corresponding to the CdTe sample is in agreement with previous reports [5,15-19]. In

reference [15] which a CdTe film using a mean kinetic energy of 120 eV was deposited at room temperature. In the present work the CdTe film was grown at 75 eV which preserves the crystalline structure but induces a slight change in the orientation as seen by the increased intensity of the (110) peak. When CdTe plasma is combined with the Cu plasmas an enhancement of the (110) peak can be observed. The peak intensity increases with  $Cu^+$  density.

The left inset of Fig. 5 shows the ratio between intensities of the (110) and (200) peaks (I<sub>(100)</sub>/I<sub>(002)</sub>) which increases for increasing Cu<sup>+</sup> density, meaning that copper incorporation into the CdTe lattice induces a preferential orientation of the crystals in the (110) direction. The right inset displays a detail of the (110) reflection, where a slight shift to lower 20 values is observed in the CdTe samples with Cu content. This shift to lower 20 values means a compression of the lattice, which could indicate that Cu<sup>+</sup> and also Cu<sup>2+</sup> enter as Cd<sup>2+</sup> substitutes. The ionic radii of Cu<sup>+</sup> and Cu<sup>2+</sup> are 0.60 and 0.57 pm, respectively, and that of Cd<sup>2+</sup> is 0.78 pm. Consequently, Cu<sup>+</sup> and Cu<sup>2+</sup> in Cd<sup>2+</sup> sites compress the lattice.

Raman spectra of the films are presented in Fig. 6. The spectrum of the CdTe sample shows three peaks centered at 120, 140 and 163 cm<sup>-1</sup>. The signal centered at 120 cm<sup>-1</sup> appears in CdTe films when a Te excess is present and it is associated to the  $A_I$  vibrational mode of Te ( $A_I$  (Te)) [15]. The appearance of this mode confirms the results obtained by EDS measurements. The 140 cm<sup>-1</sup> signal corresponds to a combination of a *TO* vibrational mode of CdTe (*TO* (CdTe)) and the  $E_I$  vibrational mode of Te ( $E_I$  (Te)), resulting from the presence of Te excess in the film [15]. Finally, it is well known that the peak centered at 163 cm<sup>-1</sup> is associated to the *1LO* mode of CdTe. The inset shows in a larger scale the *1LO* mode of CdTe. For CdTe samples doped with Cu<sup>+</sup> and Cu<sup>2+</sup>, a shift of this mode to lower energies is observed, i.e., the mode hardens. This result is a reflect of the compression of the lattice.

Regarding the samples grown with the combination of plasmas, it can be seen in Fig. 6 that similar spectra were obtained regardless the Cu<sup>+</sup> density. Notice that the  $A_I$  (Te) mode disappeared from the spectra. The small peak centered at 140 cm<sup>-1</sup> for Cu containing samples is assigned just to *TO* (CdTe) and not to the combination the  $E_I$  (Te) and *TO* (CdTe), considering that samples are nearly stoichiometric or have Cd excess (see Fig. 3) when copper is incorporated into the CdTe lattice. A strong band has been reported centered between 123 and 132 cm<sup>-1</sup> for copper telluride depending on the Cu/Te ratio [20]; not evidence for a signal centered within these values was observed in any

# a) CdTe

SEM HV: 20.0 kV WD: 12.35 mm View field: 25.4 µm View field: 25.4 µm Det. 5E Sum Mac; 10.0 kb Tabsterind(s); 90°1317

c)  $N_p = 4.9 \times 10^{13} \text{ cm}^{-3}$ 





d)  $N_p = 6.5 \times 10^{13} \text{ cm}^{-3}$ 



Fig. 9. Scanning electron micrographs of the CdTe:Cu films grown using Cu<sup>+</sup> plasma densities of: a) 0, b) 3.4, c) 4.9 and d)  $6.5 \times 10^{13}$  cm<sup>-3</sup>.

of the Raman spectra for the CdTe:Cu samples.

It has been reported that Raman active modes for cubic and hexagonal CdTe have the same frequencies [5,15,17], this means that it is not possible to distinguish between hexagonal or cubic structure of CdTe by means of Raman spectroscopy measurements.

Fig. 7 shows the Tauc plots obtained from UV–Vis measurements. The band gap  $(E_g)$  was calculated from these plots by the extrapolation of the linear part of the curve to the photon energy axis. The inset of Fig. 7 shows the dependence of band gap values on the Cu<sup>+</sup> density. The  $E_g$  value for CdTe sample has a value of 1.47 eV, when Cu is incorporated it can be seen that the band gap slightly decreased with increasing Cu<sup>+</sup> density. This is consequence of the compression of the lattice. These values are consistent with previously observed when similar quantities of Cu were added to CdTe films [21].

Photoluminescence (PL) spectra at RT are shown in Fig. 8(*a*). A PL emission centered at wavelength ( $\lambda$ ) 827 nm (1.50 eV) can be observed for all the samples. No apparent shifts of the band is observed for increasing Cu<sup>+</sup> density. However, the Fig. 8(*b*) shows a slight shift to lower  $\lambda$  values (higher energies) according with  $E_g$  data. The 1.50 eV signal corresponds to the free exciton emission in hexagonal wurtzite

CdTe [21]. Note that there is a difference between  $E_g$  calculated from UV–Vis measurements and the PL free exciton emission, this effect is caused by the contribution to the absorption of the near band edge energy levels resulting from structural and compositional defects.

Finally, in Fig. 9 the SEM images for the samples are shown. As it has been observed before [15] pulsed laser deposition of CdTe produces smooth surfaces which present no micrometer sized grain boundaries at the surface (Fig. 9a). When Cu is incorporated into the films, the surfaces becomes rough and with a granular surface morphology. The density of the micro-grains increased with increasing Cu plasma density, thus incorporation of Cu into the films induces the formation of spherical-like micron sized grains at the surface of the films.

### 4. Conclusions

Copper doped CdTe thin films can be obtained by the combination of laser produced CdTe and Cu plasmas. It was found that the physical properties of the films are strongly dependent on both mean kinetic energy and density of Cu ions present in the plasmas. XRD results showed a preferential orientation of the films when Cu and CdTe plasmas are combined, due to the incorporation of Cu ions by substitution of Cd into the CdTe lattice.

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