

Surface structural analysis using grazing incidence x-ray reflectometry

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Grazing incidence X-ray diffraction and reflectometry are used to characterize chemically treated cadmium telluride single crystal (c-CdTe) surfaces, amorphous silicon (a-Si) thin films and amorphous oxygenated cadmium telluride (a-CdTe:O) thin layers. In the case of CdTe single crystal surface treated with an oxidizing agent (a solution of Br₂ in CH₃OH), the superficial layer was found to be less dense than its support with a profound alteration of CdTe in the volume. After rinsing in KOH solution, the properties of single crystalline CdTe are obtained. In the case of a-Si thin layers, we show that the simulation of the reflectometry curves enables not only the determination of the layer thickness but also the detection of an ultra-thin superficial oxide layer. Finally, in the case of a-CdTe:O films it is found that the oxygen contents of the surface increases with increased exposure time. Also, this exposure leads to an increase of the oxide thickness and a net decrease in the surface roughness.

I. INTRODUCTION

The technique of studying thin layers by the interference of X-rays is becoming more and more reliable. Being an optical interference technique, grazing incidence X-ray reflectometry presents several advantages, namely, non destructive character, no preliminary sample preparation and easy sample handling (layers are examined in air) [1, 2]. The samples should be, however, deposited on substrates with excellent flatness (optical-grade finish) and with an area of a few cm².

Owing to the small wavelength of X-rays, this method allows a precise determination of film thickness of less than a few thousands of Angstroms. It is also extremely sensitive to surface roughness. A detailed analysis of the reflectivity spectra enables to reveal the existence of ultra thin layers at the surface or at the interface. However, X-ray reflectometry can not be used to identify compounds unless their refractive indices are known.

As a result, this technique will be useful for a variety of problems concerning annealing, corrosion, oxidation (or surface states), optical properties, etc. Also, the method enables the study of buried interfaces otherwise inaccessible by other techniques [3], and the characterization of multilayers when these are well stratified [4, 5].

In this paper we are interested in two cases for which X-ray reflectometry at grazing incidence is potentially useful. The first deals with the effect of a chemical treatment on the surface of c-CdTe. The second concerns the study of thin a-Si and a-CdTe:O layers deposited by rf-sputtering. These situations have been the subject of large number of investigations using diverse optical, electronic, and nuclear characterization techniques [6,7,8,9,10].

II. EXPERIMENTAL PROCEDURE

The principle of the technique consists of sending a filtered parallel x-ray beam onto the sample at a grazing incidence angle, and recording the change in the intensity

of the specularly reflected beam as the incidence angle (α) is varied. For increasing α , the experimentally obtained reflectivity curve first shows a slow decrease for angles less than the critical angle (α_c) which is of the order of a few tenths of a degree. This is followed by a sudden decrease of the intensity of the reflected beam, then a slow decrease again upon which, in the case of thin films or superficial layers, a series of maxima and minima (Kiessig interference fringes) may be superimposed until the incidence angle reaches 2 or 3 degrees. The period of the oscillations is directly related to the layer thickness [2, 4, 11].

The density of the material can be found from the measured critical angle for total reflection [1,12]. In addition, using the Fresnel equations and a model, it is possible to accurately simulate the measured reflectivity curves [13,14]. To this end, the sample is assumed to be composed of a series of strata each with a uniform density and refractive index. For every angle (α), the Fresnel equations are applied at the interfaces to calculate the reflection and transmission coefficients. The reflected intensity is then calculated by taking into account the phase shifts and the attenuation of the X-ray beam in the sample. The parameters (thickness of the layers, densities, and roughness) are then found from a least-square fit of the model to the experimental data [15].

We used an X-ray generator with a copper (Cu) anode equipped with a 0.1x10 mm² linear focus. A nickel (Ni) filter enables us to choose the Cu-K _{α} line. The beam is collimated using a system of 40 μ m tantalum (Ta) slits resulting in a small angular divergence (0.02°). The goniometer and the acquisition of the data are controlled by a computer. The angular precision of the goniometer positioning system is 0.003°. Other experimental and theoretical aspects of this technique will be discussed in light of the following very important examples

where X-ray reflectometry is useful: the surface of CdTe single crystal and that of a-Si and a-CdTe:O thin films.

III. RESULTS AND DISCUSSIONS

A-Single crystalline CdTe Surface

The surface plays a critical role in the electronic properties of devices based on CdTe such as solar cells [16, 17] and detectors of nuclear radiation [18]. Cleaving in vacuum [19, 20] or in air [21, 22] and mechanical polishing followed by chemical etching [7,8,23] are the methods usually used to prepare electronic-grade CdTe surfaces. In general, the chemical treatment consists of reacting the surface with an oxidizing agent such as a solution of Br₂ in methanol (CH₃OH) followed by rinsing with potash (KOH). This technique is usually used to prepare large area surfaces.

The properties of CdTe surfaces prepared by chemical treatment have been the subject of several physico-chemical investigations [19, 20]. Despite the numerous results obtained on these surfaces, problems pertinent to the chemical composition and to the density of states at the surface still remain open.

Fig. 1 presents the reflectometry curves measured at different stages of the chemical treatment. The critical angle, corresponding approximately to the angle at which the reflected intensity dropped by half (inflection point), varied in this case from 0.18 to 0.32°.

The critical angle is directly related to the density of the material through the following equation [24] :

$$\alpha_c = \sqrt{\frac{e^2}{4\pi\epsilon_0 mc^2} \frac{\lambda^2}{\pi} N(Z + \Delta f')}} \quad (1)$$

where λ is the wavelength of the X-rays, N is the number of atoms per unit volume and Z is the atomic number of the material. $\Delta f'$ is correction factor for the abnormal dispersion of the refractive index, e and m are the usual absolute charge and mass of an electron.

Fig. 1a shows the intensity of the reflected beam obtained from the surface of a mechanically polished single-crystal CdTe sample with a mirror-like finish. The critical angle (0.29°) found from this curve is less than the theoretical value (0.32°) for CdTe, suggesting that the material is perturbed over a depth of a few thousands of Angstroms. This is attributed to the damage introduced by the mechanical polishing. No superficial layer can be conclusively identified in this case since only one penetration front (i.e., critical angle) is observed. Indeed, it is well known that the surface of CdTe reacts slowly with oxygen and that the thickness of the superficial layer (oxides of tellurium) will only be on the order of a few nanometers [6]. Curve (b) in Fig. 1 corresponds to the results obtained on the above sample after a treatment with a 2% Br₂ in reagent-grade CH₃OH for one minute.

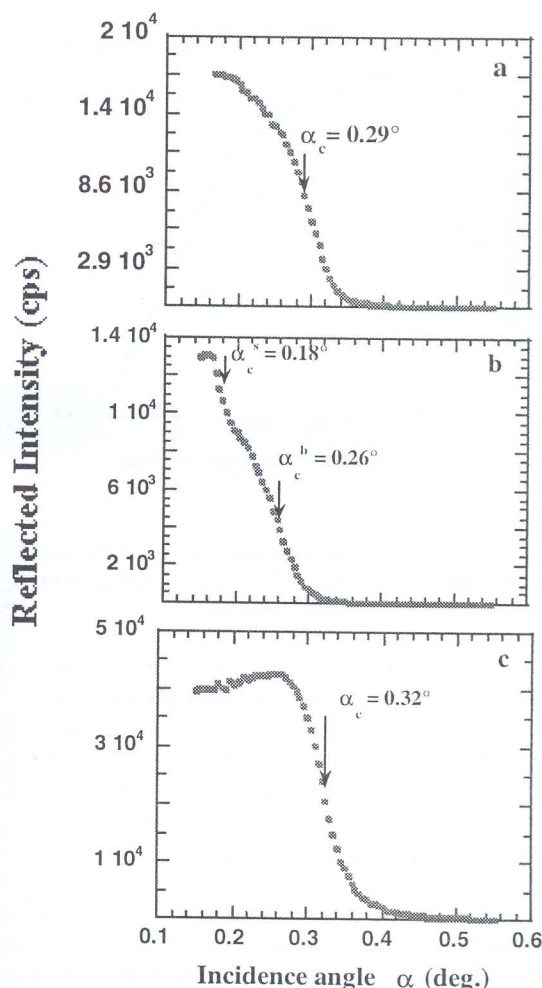


FIG. 1. Variations of the specularly reflected intensity versus the incidence angle α measured on the CdTe sample at different stages of the chemical treatment. a) untreated surface ; b) surface treated with a 1% Br₂ in CH₃OH solution ; c) surface rinsed with KOH.

The etching was performed at room temperature and without stirring the solution. In this case, the reflectivity curve reveals two X-ray penetration fronts and consequently two critical angles of 0.18° and 0.26°. These angles correspond to the angular position where the reflected intensity dropped by half from its maximum value at the onset of each penetration front. This result suggests that the surface of CdTe (after treatment) is covered by a layer markedly less dense than the underlying CdTe (only one critical angle would have been observed if the film were buried in the volume of the sample). The density of this layer with a critical angle of 0.18° was found to be 1.92 g/cm³. We note that the critical angle of 0.26° is also lower than that of single-crystal CdTe (0.32°), suggesting that the chemical treatment has further perturbed the CdTe crystal at least over a depth of a few thousands of Angstroms.

The thickness of superficial layer was found by fitting the experimental specular reflection data over the entire

range of α (Fig. 2). The value of 350 Å for

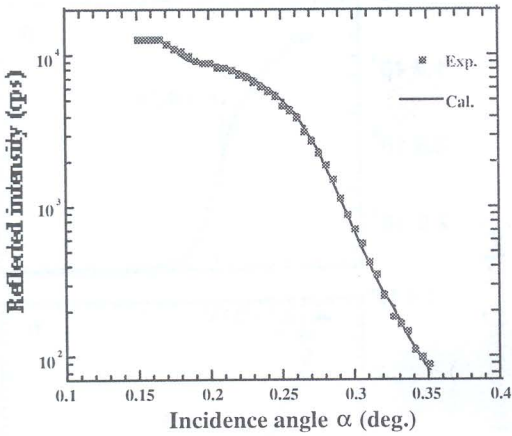


FIG. 2. Variation of the experimental (.....) and calculated (—) specularly reflected intensity of the CdTe surface treated with the a with a 1% Br2 in CH3OH solution.

thickness resulted in an agreement between the experimental and the calculated reflected intensities.

The reason behind the absence of the interference fringes in the case of the CdTe sample subjected to the Br₂-CH₃OH etch is that the critical angles of the layer and the support differ only by 0.09° which less than the period of the interference pattern expected for a layer thickness of 350 Å (0.13°).

The nature of this surface layer was revealed by X-ray Photoelectron Spectroscopy and Photoluminescence measurements showing that it is deficient in Cadmium and composed mainly of disordered oxides of tellurium as well as other impurities from the etching solution [6,7, 25]. The present results, on the other hand, only confirm the presence of this layer (less dense than the CdTe support) and are not conclusive with respect to its nature.

Subsequently, this CdTe sample is etched with a 1M KOH in reagent grade methanol. This steps was carried out at room temperature and without stirring the solution. The experimental results obtained in this case are shown in Fig. 1c. Only one critical angle of 0.31° can be observed in this case. In addition, this angle is very close to that of a CdTe single crystal (0.32°). These results suggest that the KOH treatment has removed the surface layer that resulted from the Br₂-CH₃OH etch as well as the damage that originated from the mechanical polishing. Furthermore, this treatment has decreased the roughness of the CdTe surface as the magnitude of the intensity increased and its subsequent decrease is much steeper than in the two previous cases. In addition, by taking into account the abnormal dispersion ($\Delta f = -0.255$) [26], the density (5.67 g/cm³) deduced from this angle matches well that of single-crystal CdTe (5.85 g/cm³).

B. Surface of a-Si thin Films

The samples, in this case, were prepared by rf-sputtering onto a Si(100) substrate as reported in detail elsewhere [27, 28]. The deposition was carried out at ambient temperature

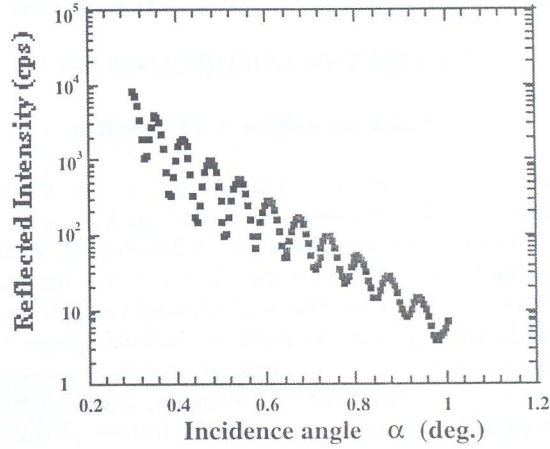


FIG. 3. Variation of the experimental specularly reflected intensity as a function of the incidence angle α for the a-Si/c-Si structure.

from a 5N, 10 cm diameter polycrystalline Si target at a rate of 30 Å/min. The sputtering gas is Argon (10⁻² mbar) and the deposition power was 250 W. The thickness of the deposited layer was around 700 Å. Fig. 3 shows a typical reflectivity curve obtained on the as-deposited samples. We can see that the curve presents a series of maxima and minima (Kiessig interference fringes) whose positions (α_p) enable us to determine the thickness (t) of the layer. These positions are related to the interference order (p) through the following relationship [29] :

$$\sin^2 \alpha_p = \alpha_c^2 + \left(\frac{p\lambda}{2t} \right)^2 \tag{2}$$

where α_c is the critical angle, and p is an integer for a maximum and half integer for a minimum. As shown in Fig. 4, plotting $\sin^2 \alpha_p - \alpha_c^2$ as a function of p² results in a straight line whose slope is inversely proportional to the thickness. Using this procedure, the value of the thickness of the layer is 630 Å. The method is more precise as the number of fringes is large. The fringes appearing at high angles are practically equidistant and the measure of the spacing between fringes ($\Delta\theta$) gives approximately the

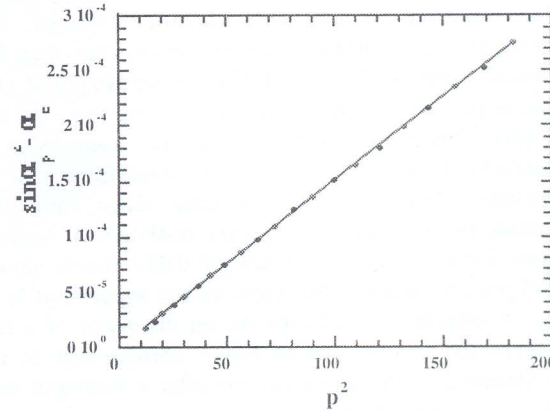


FIG. 4. Plot of $\sin^2 \alpha_p - \alpha_c^2$ versus p^2 to extract the thickness of the layer. (6) maxima (7) minima.

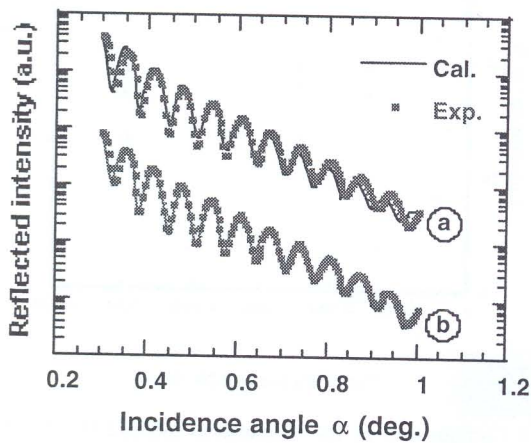


FIG. 5. Comparison of the experimental (.....) and calculated (—) reflectivity curves for the a-Si/c-Si structure. a) a-Si layer considered as uncovered; b) a-Si film considered as covered a silicon oxide layer.

thickness through the relationship $t = \frac{\lambda}{2\Delta\theta}$. This value of t fixes the interference order without ambiguity. Further information on the a-Si film can be determined by simulating the experimental reflectivity curves. Assuming only a one layer model, the best fit obtained is shown in Fig. 5a (continuous line). However, the agreement between the theory and the experiment when the incidence angle is greater than 0.7° is not acceptable. Since the sample was examined in air, we then took into account the unavoidable existence of a superficial native oxide (SiO_x) on the a-Si layer. As shown in Fig. 5b (continuous line), this two-layer model leads to a satisfactory agreement with the experimental results. The characteristics obtained for the two layers are given in Table 1.

We have also studied the reflectivity spectra of 100 Å thick a-Si films covered by a thicker oxide layer. The oxide was prepared by reactive rf-sputtering of Si in an oxygen partial pressure of 10^{-4} mbar. As shown in Fig. 6, the results reveal the existence of a modulating effect between the interference patterns produced by the two layers. Two lobes and three nodes can be observed in Fig. 6. This is a result of the superposition of two waves of respective

frequencies $\omega_1 = 2\frac{t_{ox}}{\lambda}$ and $\omega_2 = 2\frac{t_{tot}}{\lambda}$ where t_{ox} is the thickness of the oxide layer and t_{tot} is the overall thickness of the film. The mean frequency ω_m is given by $\frac{\omega_1 + \omega_2}{2} \approx \frac{1}{\Delta\theta_i}$, where $\Delta\theta_i$ is the distance between successive fringes. The beat or modulation frequency

ω_{mod} is given by $\frac{\omega_2 - \omega_1}{2} \approx \frac{1}{\Delta\theta_{mod}}$, where $\Delta\theta_{mod}$ is the period of the beats. The oxide thickness can be readily obtained from these two measured parameters as follows:

$$t_{ox} = \frac{\lambda}{2} \left(\frac{1}{\Delta\theta_i} - \frac{1}{\Delta\theta_{mod}} \right) \quad (3)$$

The thickness of the a-Si layer follows from:

$$t_{si} = t_{tot} - t_{ox} = \frac{\lambda}{\Delta\theta_{mod}} \quad (4)$$

with $\Delta\theta_i = 0.06^\circ$ and $\Delta\theta_{mod} = 0.58^\circ$, the obtained thicknesses are: $t_{ox} = 630$ Å and $t_{si} = 150$ Å. More valuable information can be extracted from the experimental data by fitting the results to a two- or three-layer model. This part of the work is in progress and will be the subject of a future report on the transition region between the a-Si layer and the oxide layer.

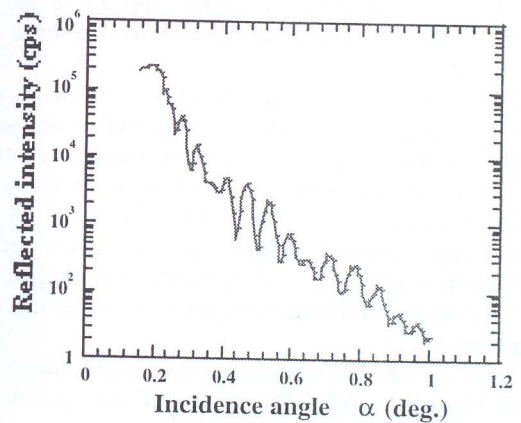


FIG. 6 Variation of the specularly reflected intensity with the incidence angle α for the a-Si sample covered by a thick silicon oxide film.

C. CdTe and a :CdTe :O thin films

1) General considerations

The thin films used in this study were prepared by RF-sputtering in an ALCATEL SCM 451 deposition system described in details elsewhere [30,31]. Prior to each deposition, the vacuum chamber was evacuated down to a base pressure of $5 \cdot 10^{-7}$ mbar.

The layers were deposited from a 10 cm diameter and highly pure (5N) polycrystalline CdTe target. Before the deposition, the target was pre-sputtered for 15 min in order to remove superficial layers resulting from exposure to ambient gases. The sputtering atmosphere consisted of a mixture of high purity (5N) argon (Ar), oxygen (O_2), and nitrogen (N_2). The overall deposition pressure was maintained at 10^{-2} mbar. Table 2 summarizes the deposition parameters for various samples.

Figure 7 shows the X-ray diffraction (XRD) spectra of samples prepared under various partial pressures of nitrogen. The XRD spectrum of the sample X11A1, deposited without intentionally adding O_2 or N_2 to the

Table 1. Parameters obtained from the calculation model used to fit the data in Fig. 3.

Layers	Thickness (Å)	Density (g/cm^3)	Roughness (Å)
SiO_x	50 ± 10	1.92 ± 0.06	13
a-Si	620 ± 10	2.08 ± 0.06	9
c-Si	-	2.33	13

sputtering gas (Ar), is marked by the presence of Table 2. Deposition parameters of the various samples.

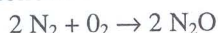
Sample	Pressure (mbar)			Power (mW.cm ⁻²)	Target bias (V)
	O ₂	N ₂	Ar		
X10A1 ^a	4,0 10 ⁻⁵	1,6 10 ⁻⁴	9,8 10 ⁻³	159	-600
X11A1 ^a	4,0 10 ⁻⁵	1,6 10 ⁻⁴	9,8 10 ⁻³	159	-600
X11A2	4,0 10 ⁻⁵	9,6 10 ⁻⁴	9,0 10 ⁻³	159	-600
X11A3	4,0 10 ⁻⁵	5,0 10 ⁻³	5,0 10 ⁻³	159	-600
X11A4 ^b	4,0 10 ⁻⁵	9,9 10 ⁻³	≅0	159	-600
X11G1 ^a	4,0 10 ⁻⁵	1,6 10 ⁻⁴	9,8 10 ⁻³	159	-600
X11G3 ^a	4,0 10 ⁻⁵	1,6 10 ⁻⁴	9,8 10 ⁻³	159	-600
X11G4 ^a	4,0 10 ⁻⁵	1,6 10 ⁻⁴	9,8 10 ⁻³	159	-600

- (a) without intentional addition of neither O₂ nor N₂.
 (b) without intentional addition of argon.

diffraction peaks at $\theta=11.8^\circ$ and $\theta=19.62^\circ$ characteristic of CdTe. On the other hand, the XRD spectra of samples deposited in the presence of nitrogen show that these films are all amorphous.

Without exception, all the samples deposited in the presence of nitrogen contain oxygen. Figure 8 shows the variations of the composition of these samples (obtained from XPS measurements) as a function of the partial pressure of nitrogen during the deposition. It can be seen that the oxygen content of these films increases with the partial pressure of N₂ and tends towards a limiting value of 41 %. This observation was interpreted using the following assumptions [32] :

1-Nitrogen in the deposition chamber plays the role of catalyst favoring, under the effect of the plasma, the formation of a strong oxidizing agent which is most likely N₂O [33,34,35] as follows :



N₂O, in turn, oxidizes the sputtered CdTe species according to the reaction :



2-The kinetics of the reactions involved are responsible for the variation of the oxygen contents of the films with the amount of catalyst in the deposition chamber during the deposition. The limiting composition is reached when the amount of nitrogen introduced is sufficient to react with all

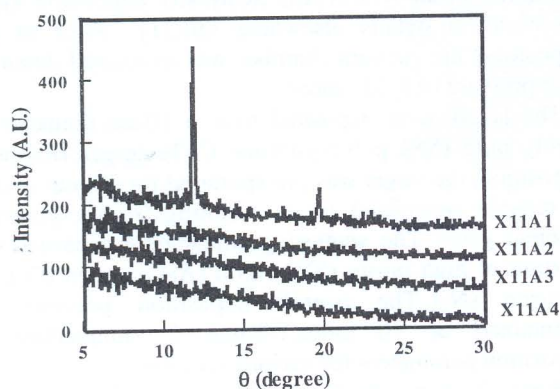


FIG. 7. X-ray diffraction patterns for some representative a-CdTe:O samples.

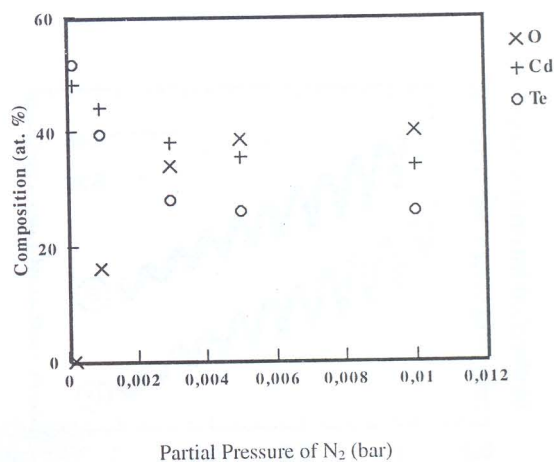


Fig. 8 Composition of the rf-sputtered a-CdTe:O samples as a function of the nitrogen partial pressure.

the residual oxygen. We can conclude that the CdTe thin films prepared by diode rf-sputtering contain oxygen when deposited in an atmosphere composed of argon, nitrogen and oxygen

2) Some properties of the surface of CdTe and a-CdTe : O films.

Figures 9 and 10 show the X-ray reflectivity spectra of samples deposited without intentional addition of N₂ and O₂. As opposed to X11G4, the sample X10A1 was exposed to the oxidizing plasma for 2 hours after the deposition. The reflectivity spectrum of X10A1 reveals two penetration fronts around the critical angles $\alpha_{c1}=0.231^\circ$ and $\alpha_{c2}=0.314^\circ$, while the spectrum of the sample X11G4 presents only one penetration front at $\alpha_c=\alpha_{c2}=0.314^\circ$ corresponding to cadmium telluride. The observation of a reflectivity front at 0.231° in the case of the sample X10A1 shows that the surface of the sample exposed to the oxidizing plasma is formed of a layer of lower density than CdTe corresponding most likely to an oxide layer. This conclusion was further supported by XPS measurements.

3) Surface Structure of the Layers

The texture of the samples was studied using X-ray reflectometry measurements on samples X11G1 and X11G3 all deposited in the absence of N₂ and O₂. However, sample X11G3 was exposed after deposition to the oxidizing plasma for 15 min. The reflectivity spectra of these samples presents Kiessig oscillations after the total reflections plateaus. The oscillations result from the interference of waves reflected from various interfaces in the thin film [36,37,38].

The thickness' as well as the surface and interface roughness were obtained by simulating the reflectivity curves using a two layer model and a Debye-Waller-type roughness model [39]. Figure 11 and 12 show that there is an excellent agreement between the experimental and the simulation results. The first parameter that is obtained from this fit is the thickness of the CdTe oxide layer. This thickness is 2.9 nm for the sample X11G3 which was not

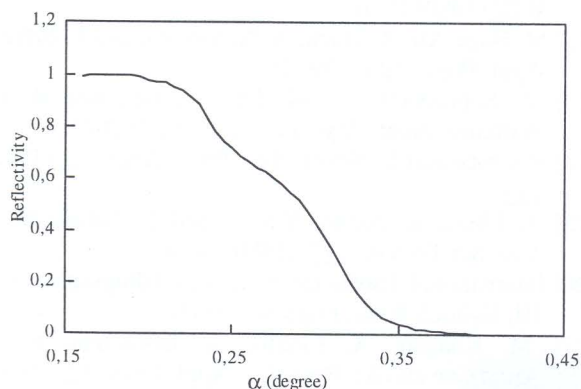


FIG. 9. X-ray reflectivity spectrum of a CdTe thin film exposed to an oxidizing plasma ($\text{Ar}+\text{N}_2+\text{O}_2$) for 15 min.

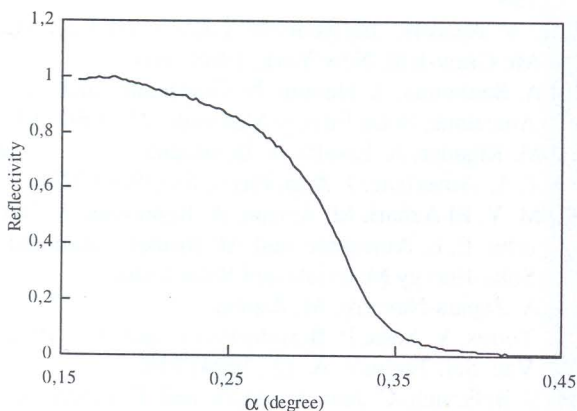


FIG.10. X-ray reflectivity spectrum of a CdTe thin film not exposed to the ($\text{Ar}+\text{N}_2+\text{O}_2$) plasma for 15 min.

exposed to the plasma, while it reaches 10.9 nm for X11G1 which was exposed to the plasma for 15 min.

The reflectivity curves also allows to determine the surface roughness of the films. For the unexposed sample X11G3, the roughness is 1.1 nm while it does not exceed 0.6 nm for the sample X11G1 which was exposed for 15 min to the oxidizing plasma.

The above results suggest that the exposure of the CdTe surfaces to the $\text{Ar}+\text{N}_2+\text{O}_2$ plasma leads to the oxidation of the surface with the oxide thickness increasing with exposure time. In addition, it mainly leads to net decrease in the roughness of the film surface

IV. CONCLUSIONS

The surfaces of CdTe single crystal and a-Si thin films have been studied by X-ray reflectometry at grazing incidences. In the case of CdTe treated with $\text{Br}_2\text{-CH}_3\text{OH}$ solution, a superficial layer of thickness 350 Å was revealed. The density of this layer is smaller than that of known Te oxides, suggesting that the surface layer is probably porous. Subsequent treatment with KOH solution resulted in a surface comparable with that of cleaved CdTe. For a-Si, a thin native oxide layer was revealed on the surface of the thin film.

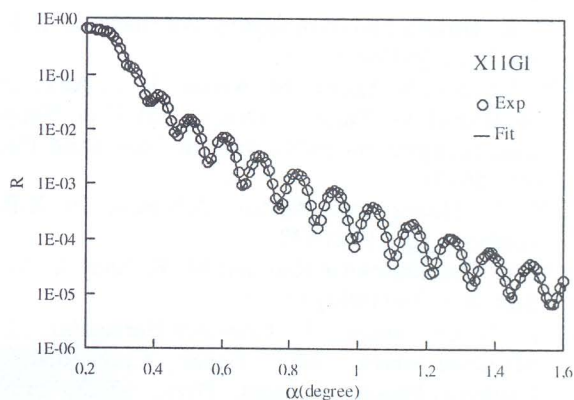


FIG. 11. . X-ray reflectivity spectrum of a CdTe thin film not exposed to the ($\text{Ar}+\text{N}_2+\text{O}_2$) plasma.

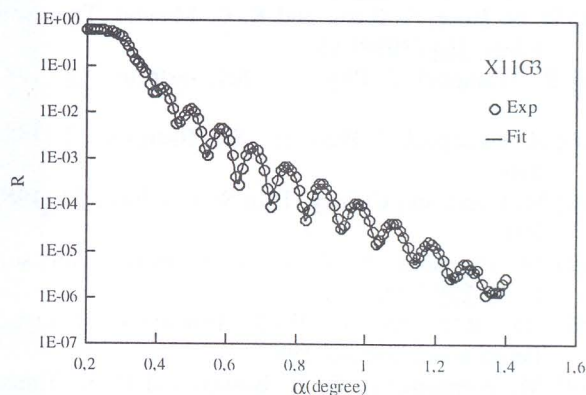


FIG. 12. X-ray reflectivity spectrum of a CdTe thin film not exposed to the ($\text{Ar}+\text{N}_2+\text{O}_2$) plasma for 15 min

Therefore a direct analysis of the X-ray reflectivity spectra allows the determination of the density and the thickness of the layer. Additional information provided by other surface sensitive techniques such as X-ray Photoelectron Spectroscopy (XPS), Auger electron spectroscopy or Rutherford Back Scattering (RBS) can be used to obtain an approximate model of the layered structure of the sample. This model is then used to fit the reflectometry spectra to obtained the depth profile and the composition of the sample.

In the case of CdTe and a-CdTe:O thin films prepared by diode rf sputtering, we showed that the surface of the layers exposed to the oxidizing plasma are affected in two ways : 1) they are oxidized to a depth which is a function of the exposure time ; 2) their surface roughness is noticeably decreased. It will be interesting to study the effect of this last observation on the electrical activity of deep levels induced near the surface region.

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