

# Auger electron spectroscopy analysis of oxidation states of Te in amorphous CdTe oxide thin films

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Amorphous CdTe oxide (*a*-CdTe:O) thin films with different concentrations of oxygen were grown by rf sputtering and analyzed by Auger electron spectroscopy. A slight change of shape in the Cd *MNN* peak as a function of oxygen content in *a*-CdTe:O is observed. This peak resembles the Cd *MNN* peak of CdTe, at one extreme, and that of CdTeO<sub>3</sub>, at the other. The Te *MNN* and the O *KLL* peaks have similar shapes and lower intensities for *a*-CdTe:O oxygen saturated films than those in CdTeO<sub>3</sub>. On the other hand, there is a large and gradual difference in shape, intensity, and energy observed in the Te *MNN* peak among *a*-CdTe:O with low, intermediate, and high concentration of oxygen. Different Te oxidation states, as Te<sup>-2</sup> and Te<sup>+4</sup>, contribute to this change as evidenced by a simulation with combinations of CdTe and of CdTeO<sub>3</sub> spectra, or by combinations of *a*-CdTe:O spectra with low and high oxygen content. © 1997 American Vacuum Society. [S0734-2101(97)01405-X]

## I. INTRODUCTION

Oxide films have generated scientific interest and have many practical applications, for example, Si and GaAs oxides have been used in metal/insulator/semiconductor and semiconductor/insulator/semiconductor devices.<sup>1-5</sup> Although there have been many studies of oxide formation on CdTe and Hg<sub>1-x</sub>Cd<sub>x</sub>Te surfaces,<sup>6-12</sup> not much work has been done on CdTe oxide films. Recently some studies on CdTe:O films growth by rf sputtering deposition have appeared.<sup>13,14</sup> Those studies show that it is very difficult to determine the chemical composition of this kind of material.

Auger electron spectroscopy (AES) is normally used to determine the elemental composition of solid surfaces. However, in the case of multielement samples, the Auger peaks related to the elements may have serious overlapping, especially in samples with multiple chemical states for an element. This makes the quantitative analysis of AES and line shape analysis very difficult. An extensive study by Wagner<sup>15,16</sup> has shown that there are many elements where the chemical shift of the Auger line is larger and thus more useful in distinguishing chemical states than the photoelectron line chemical shift. In addition to the energy shifts, it has been suggested that the Auger line shapes may provide useful chemical information for the analyst.<sup>17-19</sup> Some studies have been reported to obtain chemical information, basically by factor analysis,<sup>20,21</sup> a kind of linear multivariable analysis method.

In this work we have studied the oxidation states of Te in

amorphous CdTe oxide (*a*-CdTe:O) thin films grown by rf sputtering deposition with various concentrations of oxygen by using AES. Using these results we estimate the elemental composition of the films.

## II. EXPERIMENTAL DETAILS

A series of six samples of *a*-CdTe:O were deposited on silicon wafers by magnetron rf sputtering using a CdTe target, 99.99 at. % purity, from Balzers, in an argon-nitrous oxide (Ar-N<sub>2</sub>O) atmosphere. The base pressure of the deposition system was  $1.5 \times 10^{-6}$  Torr and the gases used were Ar—99.999 at. % purity, from Linde and N<sub>2</sub>O—99.999 at. % purity, from MG Industries. Samples were grown with a total pressure in the chamber of  $1 \times 10^{-2}$  Torr. The N<sub>2</sub>O partial pressure for the samples, named S1–S6, was  $8 \times 10^{-6}$ ,  $1 \times 10^{-5}$ ,  $6 \times 10^{-5}$ ,  $9 \times 10^{-5}$ ,  $1 \times 10^{-4}$ , and  $9 \times 10^{-4}$  Torr, respectively. Under these conditions, sample S1 was grown with very low concentration of oxygen and sample S6 was grown with an oxygen content at saturation, as reported before.<sup>14</sup> Substrate temperature during growth increased slowly from room temperature at the beginning, up to about 70 °C at the end. Before deposition, the CdTe target was sputter-cleaned for 15 min. Growth time was 20 min for all samples and the rf power used was 30 W. Also, a CdTe single crystal, 99.99 at. % purity from Balzers and CdTeO<sub>3</sub> powder 99 at. % purity from Cerac were measured with AES as reference samples.

AES analyses of *a*-CdTe:O films, CdTe single crystal, and CdTeO<sub>3</sub> powder were performed in a Perkin-Elmer PHI 560/ESCA-SAM system equipped with a double-pass cylindrical mirror analyzer, with a base pressure of  $\sim 2 \times 10^{-9}$  Torr. Auger spectra in the scanning mode were obtained with a 2 keV electron beam with typically 0.2  $\mu$ A

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current, incident at  $45^\circ$  to the surface normal, in the  $E^*N(E)$  mode and numerically differentiated using a five point convolution. No smoothing of the signal was attempted. A scanning step of 0.5 eV/step with an interval of 50 ms was utilized and 40 energy sweeps were added for each spectrum. An energy window for the Cd MNN Auger transition, and another for the Te MNN and O KLL transitions were selected. The energy resolution was  $\Delta E/E = 0.27\%$ , which correspond to a  $\Delta E$  of 1.0, 1.3, and 1.4 eV for Cd MNN, Te MNN, and O KLL, respectively. All Auger spectra were obtained after 7 min of  $\text{Ar}^+$  sputtering. Argon ion sputtering was performed with a beam energy of 4 keV and a beam current of  $0.36 \mu\text{A}/\text{cm}^2$ , yielding a sputtering rate of about 8–10 nm/min. It is known that preferential sputtering in CdTe is not considerable, since there it is reported a reduction of the Cd MNN intensity with respect to the Te MNN intensity of about 10% under wide experimental conditions.<sup>22</sup> In a similar way, Davis *et al.*<sup>23</sup> found no preferential sputtering in  $\text{CdTeO}_3$ ,  $\text{TeO}_2$ ,  $\text{CdO}$ , and  $\text{Cd}(\text{OH})_2$ . Since our films have different oxygen concentration and considering the results of Stahl *et al.*<sup>22</sup> and Davis *et al.*,<sup>23</sup> we assume no preferential sputtering in all our samples. All spectra presented only Cd, Te, and O peaks, indicating a lack of contamination in the films.

X-ray diffraction (XRD) spectra were measured by means of a Siemens D5000 diffractometer with scan rate of  $2^\circ/\text{min}$ . The source used was Cu ( $K_\alpha$ ,  $\lambda = 0.15406 \text{ nm}$ ).

### III. RESULTS

The *a*-CdTe:O films obtained have thicknesses between 0.8 and 1.5  $\mu\text{m}$ , the thicknesses decrease when the partial pressure of  $\text{N}_2\text{O}$  increases. The Auger signals of tellurium and oxygen of the cadmium telluride oxides have serious overlapping, which makes difficult the elemental quantification using the integrated area under Auger peak related to each element. On the other hand, the peak-to-peak intensity method in the derivative Auger spectra for elemental quantification is very common, then we analyze the derivative spectra.

The obtained derivative Auger spectra of the *a*-CdTe:O films show different shifts in energy, presumably due to charging effects caused by the very high resistivity characteristic of the samples. In order to compensate this effect and since no noticeable energy shift has been reported in the Cd MNN peaks for a CdTe (110) surface,<sup>11</sup> we adjusted all derivative spectra to the obtained Cd MNN Auger transition for crystalline CdTe at 372.5 eV. In order to compare corresponding intensities among all samples, since intensities changed from sample to sample, we adjusted all derivative spectra to the main Cd MNN Auger transition to have the same intensity.

In Fig. 1 we present the CdTe crystal and the powder  $\text{CdTeO}_3$  derivative Auger spectra for (a) the Cd MNN transition and (b) the Te MNN and O KLL transitions. In part (a) we can observe some changes between both samples, mainly in the intensity of the peak located at about 378.5 eV; this intensity is larger for CdTe. However, in part (b) the

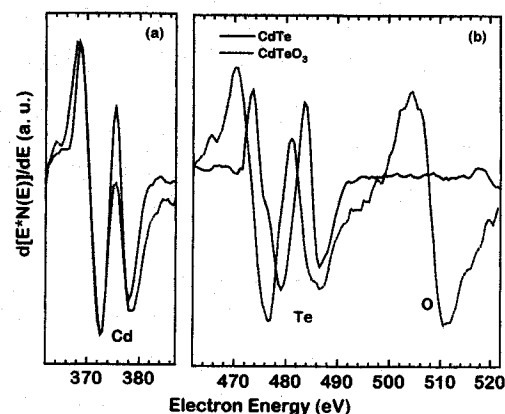


FIG. 1. CdTe and  $\text{CdTeO}_3$  derivative Auger spectra for (a) Cd MNN transition and (b) Te MNN and O KLL transitions.

spectra are very different. As expected, there is a large peak related to oxygen in the  $\text{CdTeO}_3$  spectrum which is not present in the CdTe spectrum, and there is an important shift of 2.5 eV between their main Te MNN peaks, 479 eV for CdTe and 476.5 eV for  $\text{CdTeO}_3$ . Also, although the relative stoichiometry between Cd and Te has not changed from one sample to the other, there is a remarkable increase in the Te MNN peaks of  $\text{CdTeO}_3$ .

In Fig. 2 we show the *a*-CdTe:O derivative Auger spectra of samples S1–S6 for (a) the Cd MNN transition and (b) the Te MNN and O KLL transitions. In part (b) the spectra show a gradual increase in the peak related to oxygen from samples S1–S6. On the other hand, we notice in part (a) that for samples S1–S6 cadmium spectra are alike in shape except for a small gradual change in the intensity of the peak at about 378.5 eV which becomes smaller as a function of the increasing oxygen content of the samples. Also, in part (b) the peaks related to Te present different shapes for samples S1 and S6 and what appears to be a gradual combination of both extremes in between.

The derivative Auger spectra for the CdTe crystal and sample S1 (a) the Cd MNN transition, and (b) the Te MNN and O KLL transitions are presented in Fig. 3. We appreciate

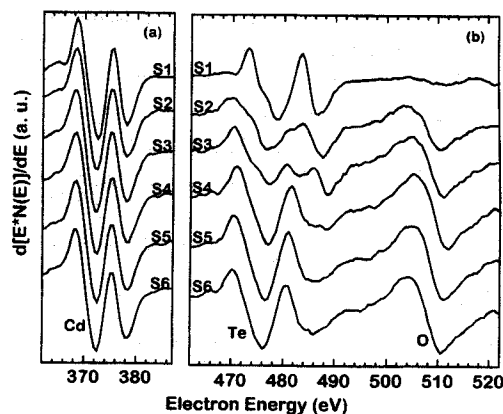


FIG. 2. *a*-CdTe:O derivative Auger spectra of samples S1–S6 for (a) Cd MNN transition and (b) Te MNN and O KLL transitions.

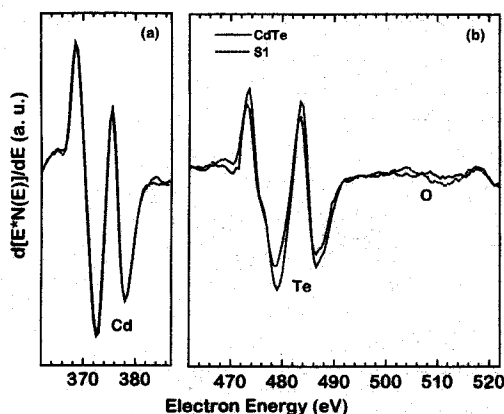


FIG. 3. CdTe and sample S1 derivative Auger spectra for (a) Cd *MNN* transition and (b) Te *MNN* and O *KLL* transitions.

that both spectra are very similar, and we can also observe a small peak (at 508.5 eV) related to oxygen for sample S1. Also, the Te peaks of sample S1 are smaller than those of the CdTe crystal.

A comparison between derivative Auger spectra of CdTeO<sub>3</sub> and sample S6 for (a) the Cd *MNN* transition and (b) the Te *MNN* and O *KLL* transitions are presented in Fig. 4. There are smaller differences in the high energy peak of the Cd *MNN* transition, and we notice that tellurium and oxygen peaks for CdTeO<sub>3</sub> are larger than for sample S6, with approximately similar shapes.

All films were measured with XRD and the results, not presented here, show that in all cases except in sample S1 there is no indication of crystallinity with very flat diffractograms at the noise level. Sample S1 also presents a flat diffractogram and a very small peak [related to the (111) reflection], about five times the noise level, indicating that the samples are basically amorphous.

#### IV. DISCUSSION

There is a change in the Cd *MNN* peaks between CdTe and CdTeO<sub>3</sub> (see Fig. 1) showing that these peaks are wider for CdTeO<sub>3</sub>. Intermediate shapes occur for samples S1–S6

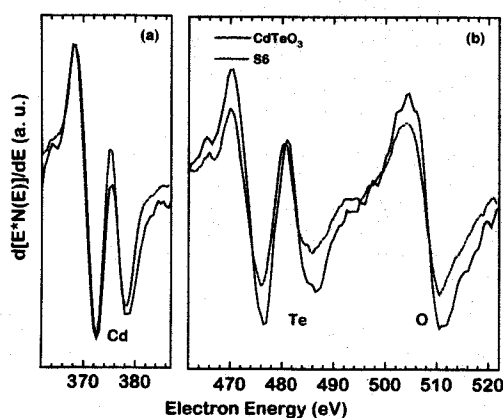


FIG. 4. CdTeO<sub>3</sub> and sample S6 derivative Auger spectra for (a) Cd *MNN* transition and (b) Te *MNN* and O *KLL* transitions.

(see Fig. 2) which show a continuous change in these Cd peaks as a function of oxygen peak intensity, sample S1 being the closest to CdTe (see Fig. 3) and sample S6 the closest to CdTeO<sub>3</sub> (see Fig. 4). This can have implications for quantification in these compounds if measurements with low analyzer resolution are made that could affect the sharper peaks more, as we can see in the results of Goldstein *et al.*<sup>24</sup> For example, see the cadmium and tellurium peaks in Figs. 6 and 7 in Ref. 11. It should be noted that the expected relative stoichiometry between Cd and Te among the S1–S6 samples is the same as in CdTe (or CdTeO<sub>3</sub>) since in the deposition process the only variable is the relative concentration of N<sub>2</sub>O in the residual gas environment.

The large difference in shape, size and energy observe between the Te *MNN* peaks of CdTe and CdTeO<sub>3</sub> (see Fig. 1) might be attributed to two different oxidation states of this element, Te<sup>-2</sup> for CdTe and Te<sup>-4</sup> for CdTeO<sub>3</sub>. Difference in the corresponding peaks between CdTe and CdTe oxide have been reported before.<sup>11</sup> The energy for the main Te *MNN* peak of CdTe and CdTeO<sub>3</sub> observed (see Fig. 1) are 479 and 476.5 eV, respectively. The corresponding energy for the same Te peak of pure Te reported is 483 eV.<sup>25</sup> On the other hand, the main Te *MNN* peak intensity for CdTe is smaller than the corresponding one for CdTeO<sub>3</sub>. This change of intensity of the Te *MNN* peak can mislead attempts to quantify atomic concentrations in these or related compounds if no consideration is made for different sensitivity factors for Te. Moreover, even larger changes in shape and intensity of the same Te peak are present for different *a*-CdTe:O films as a function of different oxygen peak intensities (see Fig. 2) causing a continuous shift to lower energies and deformation of the Te peaks.

A comparison between CdTe (without oxygen) and sample S1 (with very small content of oxygen) spectra (see Fig. 3) shows that, even in this case, there is a change in the Te *MNN* main peak intensity between these two compounds. On the other hand, sample S6 was prepared with the largest pressure of N<sub>2</sub>O during growth with a consequent saturation of the oxygen content in the film, as was reported before in similar samples.<sup>14</sup> A comparison between CdTeO<sub>3</sub> and sample S6 spectra (see Fig. 4) indicates that the Te *MNN* peaks are smaller for sample S6, although the shapes are similar. A similar situation occurs for the O *KLL* peaks in the last case. This could indicate that the sensitivity factors for sample S6 are different from those of CdTeO<sub>3</sub> powder, or that this amorphous film has a different composition than CdTeO<sub>3</sub>, having a lower concentration of oxygen.

The previous x-ray photoelectron spectroscopy (XPS) study on similar films<sup>14</sup> show that the Te are bonding with Cd and O, the fraction of each bonding depending of the oxygen concentration, and the Te–O bonds increase with oxygen while Te–Cd decrease. The possibility of Te–Te bonds, although they cannot be totally ruled out, seems to be very slim since XPS study did not show evidence for it under the limits of this spectroscopy.<sup>14</sup> Similar results were obtained in native oxide of CdTe by Choi and Lucovsky.<sup>12</sup> This result can be explain in terms of the relative bond energy

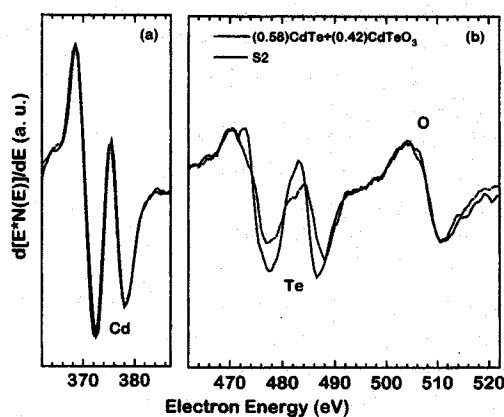


FIG. 5. Average derivative Auger spectra of CdTe and CdTeO<sub>3</sub>, and derivative Auger spectra of sample S2 for (a) Cd *MNN* transition and (b) Te *MNN* and O *KLL* transitions.

between the elements, and the Te–O bonds are favored and the rest of tellurium are bonded with cadmium. Based on this analysis we can expect that the Te *MNN* peaks in S1–S6 samples are formed basically by a combination of the corresponding Te *MNN* peaks of Te<sup>–2</sup>, as in CdTe, and Te<sup>–4</sup>, and as in CdTeO<sub>3</sub>, respectively. Assuming this possibility, the gross changes observed in the Te *MNN* peaks of samples S1–S6 could be explained basically in terms of the coexistence of different oxidation states of Te (for instance, Te<sup>–2</sup> as in CdTe, and Te<sup>–4</sup> as in CdTeO<sub>3</sub>) in these films causing an overlap of signals, which have shifts in energy and different shapes, and since they could have different intensities, depending on their relative concentrations of each oxidation state, the shape of the curve is heavily distorted. This fact makes the quantification of these compounds difficult, especially for intermediate concentrations of oxygen. An apparent reduction of the tellurium peak for intermediate concentrations of oxygen, and a further increase for the oxygen saturated samples appears in a recent report with similar samples.<sup>14</sup> These apparent changes in relative stoichiometry between Cd and Te are unreal and are due to the coexistence of two oxidation states of Te in those samples.

An argument in favor of the idea that there are different oxidation states of Te in these samples is presented graphically in Fig. 5. We show a simulation of a spectrum with two oxidation states with a combination of 58% of CdTe and 42% of CdTeO<sub>3</sub> spectra, by adding point-by-point the deriva-

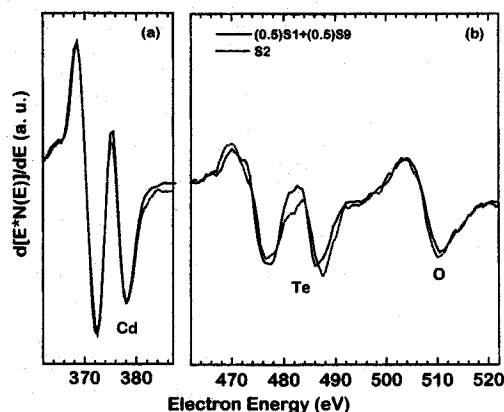


FIG. 6. Average derivative Auger spectra of samples S1 and S6, and derivative Auger spectra of sample S2 for (a) Cd *MNN* transition and (b) Te *MNN* and O *KLL* transitions.

tive Auger spectra. This simulation compares favorably with the spectrum of sample S2 (with an intermediate oxygen signal) in the regions of Te *MNN* and O *KLL* [see part (b)] and very well in the region of Cd *MNN* [see part (a)]. We believe that the comparison is not perfect, possibly because we are comparing the spectrum obtained from a crystalline compound and an amorphous film spectrum, and because we are considering only two phases. A better match is obtained when an average with equal weights of spectra of samples S1 (with a small oxygen signal) and S6 (with the highest oxygen signal) by adding the derivative Auger spectra point-by-point, presented in Fig. 6. The better match in all their peaks (Cd, Te, and O) is probably because in this case, we are comparing amorphous materials. The fact that there is a shift in energy between the Te *MNN* peaks of CdTe and CdTeO<sub>3</sub>, and that we can reproduce the *a*-CdTe:O spectra with intermediate concentrations of oxygen by combinations of CdTe and CdTeO<sub>3</sub> spectra, indicates that indeed there are different oxidation states of Te in *a*-CdTe:O films. This situation makes difficult the quantification of this kind of material by AES, and makes it impossible to use the peak-to-peak intensity method with an elemental sensitive factor. However, with a more detailed data analysis one can distinguish the different oxidation states of Te and improve quantification.

To show that this last possibility is feasible, we compare in Table I the different intensity ratios, with respect to the Cd *MNN* main peak intensity, for all the studied samples and

TABLE I. The different intensity ratios, with respect to Cd *MNN* main peak intensity, and the possible relative concentrations of the two coexisting oxidation states of tellurium, Te<sup>–2</sup> and Te<sup>–4</sup>, for all the studied samples.

	$I(\text{Te})/I(\text{Cd})$	$I(\text{O})/I(\text{Cd})$	$I(\text{O})/I(\text{Te})$	Te <sup>–2</sup> (%)	Te <sup>–4</sup> (%)
CdTe	0.68	0	0	100	0
S1	0.54	0.06	0.01	92	8
S2	0.39	0.34	0.96	58	42
S3	0.44	0.47	1.05	42	58
S4	0.59	0.55	0.93	31	69
S5	0.65	0.57	0.87	29	71
S6	0.61	0.59	0.96	27	73
CdTeO <sub>3</sub>	0.87	0.80	0.92	0	100

based on the idea that these amorphous films would have an intermediate concentration of oxygen, we decided to consider the possibility that the Auger spectra of  $\alpha$ -CdTe:O could be simulated by a combination of the spectra of the two compounds with the more common oxidation states of Te, with extreme concentrations of oxygen (CdTe and CdTeO<sub>3</sub>), i.e., Te<sup>-2</sup> and Te<sup>+4</sup>. Assuming that Te has the same concentration as Cd in all samples we also calculated possible concentrations of the two coexisting oxidation states of Te in these films as follows: first, for sample CdTeO<sub>3</sub> the ratio  $I(O)/I(Cd)$  is 0.8, and in this sample all tellurium is in state Te<sup>+4</sup>, then we divided the  $I(O)/I(Cd)$  ratio of each sample by 0.8 and assigned the resulting amount as the percentage of tellurium in state Te<sup>+4</sup> in that sample. We present that result in the last column of Table I, together with the difference to 100%, which was assigned to tellurium in state Te<sup>-2</sup>. One can see that for low concentration of oxygen (sample S1) the oxidation state of tellurium is basically Te<sup>-2</sup>, which is reasonable because the tellurium bonds with the cadmium. Besides we can notice that the Te<sup>+4</sup> concentration increases with the oxygen while the Te<sup>-2</sup> decreases because some Te atoms bond with Cd and the rest bond with O. For sample S6, the majority oxidation state of Te is Te<sup>+4</sup> because the samples were grown with a high concentration of oxygen.

Although more elaborate ways to quantify this kind of material can be developed, this method would be easy to apply in many laboratories for the basic quantification of the concentration of elements and their oxidation states.

## V. CONCLUSIONS

From the AES measurements of the series of  $\alpha$ -CdTe:O films with different concentrations of oxygen grown by rf sputtering, CdTe single crystal, and CdTeO<sub>3</sub> powder, and using as a reference the Cd MNN transition, we can conclude the following: There is a slight change of shape in the Cd MNN peaks as a function of oxygen content that resembles the Cd MNN peak of CdTe at one extreme and that of CdTeO<sub>3</sub> at the other. The Te MNN and the O KLL peaks have similar shapes but lower intensities for oxygen saturated films of  $\alpha$ -CdTe:O than for CdTeO<sub>3</sub>. There is a large difference in shape, intensity, and energy observed between the Te MNN peaks between  $\alpha$ -CdTe:O with a very low concentration of oxygen and oxygen saturated, and a continuous change of the same peaks for  $\alpha$ -CdTe:O samples in between. The change is attributed to contributions of different Te ox-

idation states like Te<sup>-2</sup> and Te<sup>+4</sup> which occur in CdTe and CdTeO<sub>3</sub>, respectively, and these induce a poor elemental quantification of the films by using peak-to-peak intensity method with a sensitive factor. This change can be approximately simulated by making linear combinations of CdTe and CdTeO<sub>3</sub> spectra, or by linear combinations of the spectra of  $\alpha$ -CdTe:O with low and high oxygen content.

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