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XPS and SIMS studies of MBE-grown CdTe/InSb(001) heterostructures

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Abstract. A series of MBE-grown CdTe/InSb(001) heterostructures with different substrate growth temperatures is studied using x-ray photoelectron spectroscopy (XPS) and secondary-ion mass spectrometry (SIMS). XPS shows that Te exists mainly as TeO₂ at the sample surface, while In exists as In₂O and In₂O₃. The Cd/Te ratio is greater than one indicating that the surface is Cd rich. Both XPS and SIMS reveal In interdiffusion directly. The SIMS depth profiles and the XPS In 3d and 4d spectra indicate that the In concentration in the CdTe film grown at high substrate growth temperature, T_s , is significantly higher than at low T_s . This is attributed to the diffusion of indium across the CdTe/InSb interface from the InSb substrate. Dislocations also enhance the diffusion of In from the InSb substrate. It is deduced that CdTe and InSb have an exact lattice match at growth temperatures of about 180 °C since the surface In concentration is a minimum for samples grown at $T_s \sim 180$ °C. The SIMS depth profiles for Te⁺ also give evidence of some diffusion of tellurium across the interface into the substrate for samples grown at high T_s . Furthermore, the SIMS data suggest that some In_xTe_y compound, probably In₂Te₃, forms at the interface, but further studies using other analytical techniques are required to confirm this.

1. Introduction

CdTe is an important II–VI semiconductor compound with a bandgap of 1.56 eV at room temperature. It is an attractive material due to its numerous and potential uses in gamma-ray and infrared detectors, solar cells and other applications in optoelectronics, integrated optics and CdTe–HgTe, CdTe–CdMnTe superlattices. It is also used as a substrate and buffer material for HgCdTe-based devices. Therefore, much work has been invested in the growing of high-quality CdTe layers on various substrates by liquid phase epitaxy (LPE), vapour phase epitaxy (VPE), metallorganic chemical vapour deposition (MOCVD) and molecular beam epitaxy (MBE).

InSb is a suitable substrate for the growth of CdTe films due to its close lattice match with CdTe. At 298 K, the lattice constant for bulk CdTe is 6.4829 Å, and for InSb is 6.4798 Å [1], giving a lattice mismatch of only 0.05%. Their thermal expansion coefficients are also fairly similar between 77 and 600 K [2].

Previous studies for MBE-grown CdTe films on (001) InSb substrates have shown that the quality of the CdTe films is affected by substrate growth temperature, T_s , the surface

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preparation prior to MBE [3] and other growth-related parameters [4–7]. As T_g is varied during the growth, there are large variations in structural defects in the CdTe film, the existence of which have been confirmed by planar and cross-sectional transmission electron microscopy (XTEM), x-ray double-crystal rocking curves (DCRC), photoluminescence (PL), etch profiling and scanning electron microscopy (SEM) [8–13]. It has been reported that in CdTe/InSb heterostructures, indium diffuses across the interface and modifies the optical and electronic properties of epitaxial CdTe [14–16]. In this work, the indium interdiffusion in MBE-grown CdTe/InSb heterostructures at different substrate growth temperatures is studied using the surface analytical techniques of XPS and SIMS.

2. Experimental details

The CdTe films were grown on (001) InSb substrates by MBE using a Westinghouse-built MBE machine equipped with a Varian 360-style vacuum load lock and sample manipulator, a Varian scanning ion gun (Type 981-2043) and a reflection high-energy electron diffraction (RHEED) system. The system was ion pumped (Varian triode pump, 400 l s^{-1}) and sublimation pumped. A liquid-nitrogen-cooled cryoshroud surrounded the source region but not the deposition region. Base pressures were in the 10^{-10} mbar range prior to growth, and in the 10^{-9} mbar range during growth. The InSb substrates were (001)-oriented wafers polished on both sides by the supplier (Metal Specialties, Fairchild, CT). They were free etched before growth with a lactic acid solution of $\text{H}_2\text{O}:\text{HNO}_3:\text{HF}$ in the ratio of 25:4:1. The method of *in situ* cleaning was ion bombardment and subsequently annealing at $\sim 200^\circ\text{C}$ before epitaxial growth. The CdTe source material was high-purity polycrystalline material made by crushing Bridgman-grown CdTe crystals. The purity of this material can be judged from its room-temperature carrier concentration of around $1 \times 10^{-15} \text{ cm}^{-3}$, n type. It was extensively outgassed by heating to 700°C prior to growth [5]. The growth rates were in the range of $0.5\text{--}0.7 \mu\text{m}$ per hour. Film thicknesses were determined from step height measurements using a contact stylus profilometer ('Talystep') to scan across the step produced by removing the epitaxial CdTe film to the initial substrate level using a chemical etch selective to CdTe. Thicknesses were precise to 3%. The CdTe film thicknesses for this set of samples are in the range of $0.8\text{--}1.4 \mu\text{m}$, as listed in table 1. The substrate growth temperature was measured by a thermocouple attached to the substrate block, with absolute temperature accurate to about 5°C . Reproducibility in measurement was probably better than 1°C . The temperature was varied between 170°C and 285°C for different samples in this study. Different techniques, such as x-ray double-crystal rocking curves (DCRC), transmission electron microscopy and photoluminescence, have been employed for the characterization and investigation of these or similar MBE CdTe films on InSb [8–13].

XPS measurements were made in a VG ESCALAB MkII spectrometer (base pressure $<2 \times 10^{-10}$ mbar) using a Mg $K\alpha$ x-ray source (1253.6 eV, 120 W) at a constant analyser pass energy of 20 eV. The instrument has been calibrated at high resolution on both the Au $4f_{7/2}$ peak at binding energy 84.0 eV and the Cu $2p_{3/2}$ peak at binding energy 932.7 eV. This procedure ensures that the error at midrange of the energy scale will not exceed 0.05 eV [17]. All XPS binding energies are calibrated to the C 1s peak of adventitious carbon at 284.8 eV [18]. A take-off angle (angle between the detected photoelectrons and surface) of 75° was used for all the samples.

The SIMS experiments were performed in a VG SIMSLAB (base pressure $<2 \times 10^{-10}$ mbar) which is connected to the XPS chamber via a preparation chamber. A 9 keV 100 nA rastered O_2^+ beam produced from a differentially pumped VG DP50B duoplasmatron ion gun was incident at 45° to the sample normal. O_2^+ instead of Ar^+ was chosen as the

Table 1. Summary of data and growth parameters of six MBE-grown CdTe films with substrate temperatures from 173 °C to 285 °C.

Sample number	Substrate temperature T_s (°C)	Film thickness (μm)	Growth rate ($\mu\text{m h}^{-1}$)
203	173	1.17	0.52
206	180	1.21	—
233	185	1.23	—
249	225	1.32	0.66
218	265	1.40	0.70
220	285	0.88	0.70

primary-ion beam since the secondary-ion counts are significantly enhanced using O_2^+ , and furthermore oxygen is not an element of interest in the SIMS studies. Energy-prefiltered secondary ions were detected normally by a VG MM12-12 quadrupole mass spectrometer (0–800 amu). Electronic gating was used to minimize crater edge effects. The sample bias (approximately 10 V for positive ions) was chosen to optimize yields at cluster ions.

3. Results and analysis

3.1. X-ray photoelectron spectroscopy (XPS)

High-resolution XPS spectra from Cd 3d, Te 3d, In 3d and Sb 3d regions were obtained for the samples. Due to spin-orbit coupling, the 3d peaks are split into two components of $3d_{5/2}$ and $3d_{3/2}$ with an area ratio of 3:2. Figure 1 shows the Cd 3d, Te 3d and In 3d spectra for a typical sample (No220) used in this study. The Sb signals (Sb 3d and 4d) were weak indicating that there is little Sb on the surface, and the spectra are not presented here. Detailed curve fitting was only performed on the stronger $3d_{5/2}$ peak, with the background line going through the mean of the noise on either side of the peak at the point of lowest average intensity. The Cd $3d_{5/2}$ spectrum is found to consist mainly of a single peak with a binding energy of (405.2 ± 0.1) eV which corresponds to that of elemental Cd or CdTe [18]. The In $3d_{5/2}$ spectrum also consists of one component with a binding energy of (444.2 ± 0.1) eV which corresponds to In_2O or In_2O_3 [18]. The Te $3d_{5/2}$ spectrum, on the other hand, exhibits two peaks which are assigned to Te in CdTe at (572.7 ± 0.2) eV and TeO_2 at (576.1 ± 0.1) eV [18].

Table 2. Ratios of selected peak areas.

Sample				
Substrate temp. (°C)	Sample number	In/Cd	In/Te	Cd/Te
173	203	0.028 ± 0.005	0.09 ± 0.02	3.1 ± 0.3
180	206	< 0.007	< 0.07	10.0 ± 1.0
185	233	< 0.016	< 0.15	9.4 ± 0.9
225	249	< 0.013	< 0.03	2.4 ± 0.2
265	218	0.026 ± 0.005	0.16 ± 0.03	6.2 ± 0.6
285	220	0.22 ± 0.02	0.47 ± 0.05	2.2 ± 0.2

All the spectra are decomposed into their appropriate components by using a mixed

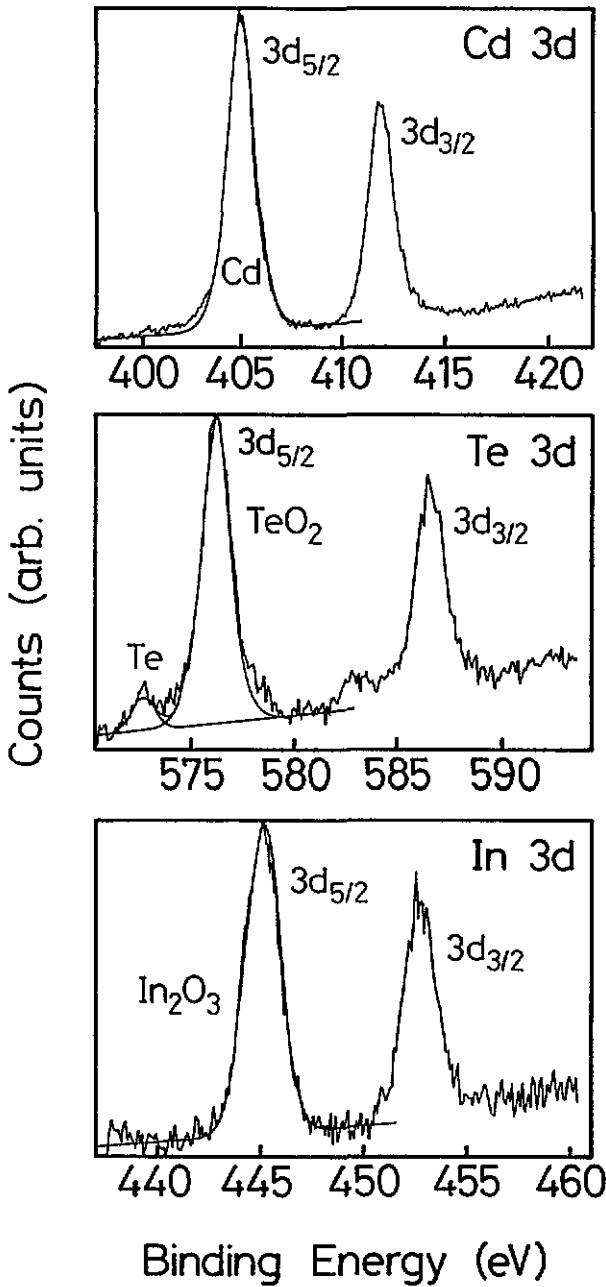


Figure 1. XPS spectra showing the Cd 3d, Te 3d and In 3d transitions from a typical MBE-grown CdTe/InSb heterostructure (No220). Smooth solid curves are composites obtained by adding the deconvoluted curves for each component.

Gaussian-Lorentzian function with suitable full width at half maximum (FWHM). The area of each peak is determined and corrected using the manufacturer's relative sensitivity factors (RSFs) [19]. This set of RSFs is based on the empirical RSFs published by Wagner *et al* [20] and corrected to the VG ESCALAB's $E^{-0.5}$ response function. The accuracy of

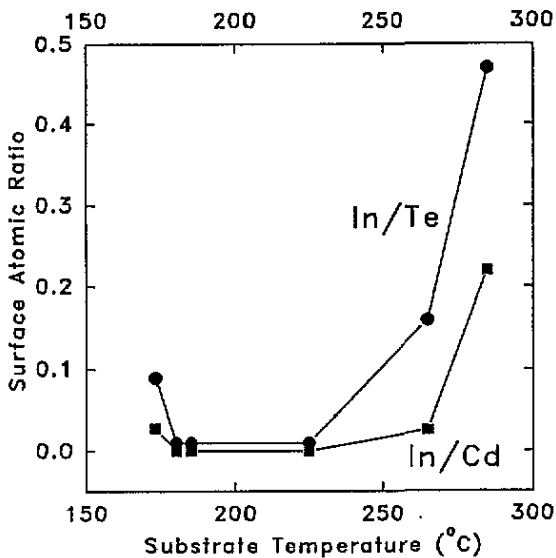


Figure 2. Surface atomic ratios of In/Cd and In/Te versus the substrate growth temperature, T_s .

this set of RSFs has been tested using samples of known stoichiometry. The RSFs used for Cd $3d_{5/2}$, Te $3d_{5/2}$ and In $3d_{5/2}$ are 4.3, 6.1 and 4.6 respectively. The ratios of the In atomic concentration to that of Cd and Te as well as the Cd/Te ratio are calculated and presented in table 2. The Cd/Te ratios for the samples are greater than one, indicating that the surface is Cd rich. It is also observed that for samples which have high In concentration in the CdTe film, the Cd/Te ratios are generally smaller. This suggests that there is substitution of surface Cd atoms by In. The In/Cd and In/Te ratios are plotted against the substrate temperature in figure 2. Both these plots clearly indicate that the indium surface concentration relative to either Cd or Te increases with increasing substrate growth temperature T_s , with the exception of the $T_s = 173$ °C sample. The In 3d XPS spectra for all the samples are also shown on a single plot for comparison in figure 3.

These data indicate that strong In signals appear for samples that are grown at high T_s ($T_s > 225$ °C) while relatively low signals are obtained for those grown at lower T_s , the minimum In intensity being at 180 °C. It was confirmed from previous studies that these In signals are not due to subsequent surface contamination [21]. The high In concentration in the CdTe film grown at high T_s can be attributed to indium diffusion from the InSb substrate. The diffusion constant of any species in a perfect crystal is given by the following relation:

$$D = D_0 \exp(-E/kT) \quad (1)$$

where D_0 = diffusion constant when $T = 0$ K, E = diffusion activation energy, k = Boltzmann constant, and T = temperature. We would expect, therefore, that higher substrate growth temperatures would cause more indium to diffuse across the CdTe/InSb interface and result in a higher indium surface concentration. The observed increase in indium surface concentration for the $T_s \sim 173$ °C sample as compared to the $T_s \sim 180$ °C sample will be discussed in the light of the SIMS results.

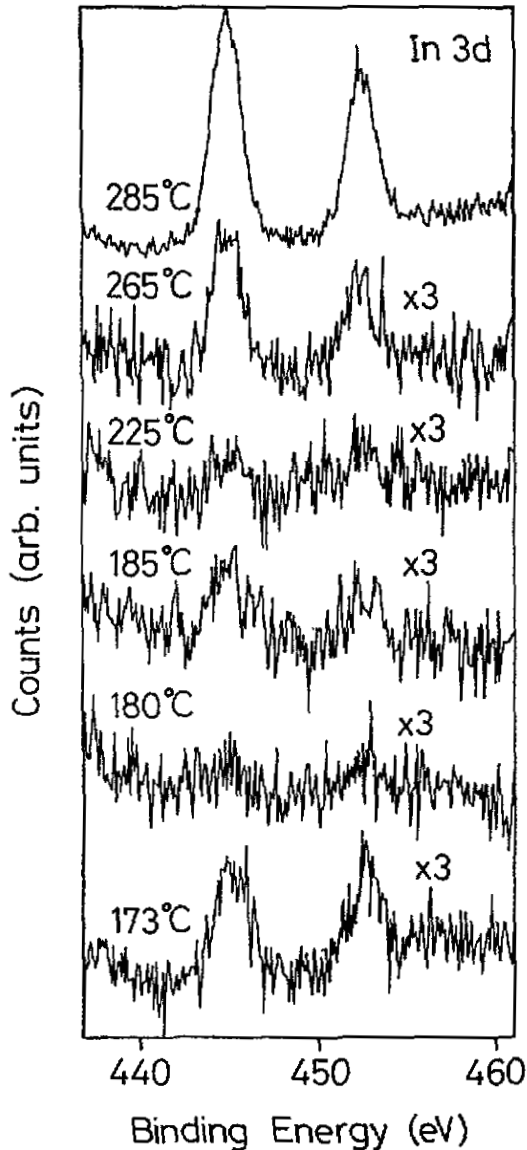


Figure 3. xps In 3d signals of MBE-grown CdTe/InSb samples at different T_s .

3.2. Secondary-ion mass spectrometry (SIMS)

The positive SIMS depth profiles of the six samples are shown in figure 4. The CdTe/InSb interface is indicated by the abrupt increase in the In^+ and Sb^+ signals. The Cd^+ signal, however, showed an increase in yield at the interface as well when it should be expected to decrease. This observation can be explained by the so-called 'SIMS matrix effect', i.e. the yield of Cd^+ is higher in the InSb matrix than in the CdTe matrix.

Interfacial reactions have been reported to occur which result in the formation of interfacial layers [22–24]. These CdTe/InSb interfacial layers were found to be rich in indium and tellurium. Previous studies using XPS and Raman spectroscopy indicate that the

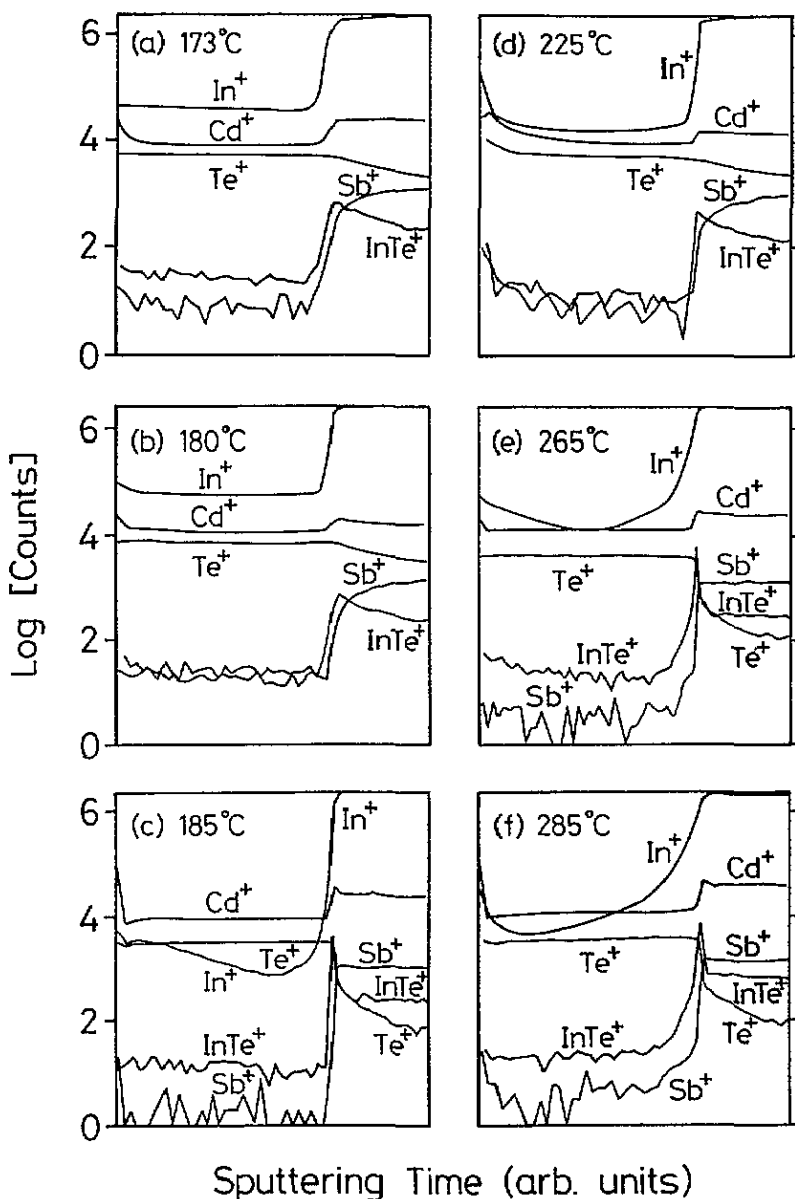


Figure 4. SIMS depth profiles obtained for MBE-grown CdTe/InSb heterostructures at different T_s . The CdTe/InSb interface is clearly visible from the rise in the In^+ signal.

interfacial layer might contain In_2Te_3 [23, 24]. It was also reported from thermodynamic considerations that the formation of In_2Te_3 at the interface is unfavourable and that the interfacial compound is stressed InTe and not In_2Te_3 [15]. In order to investigate this, the profiles of InTe^+ , InTe_2^+ and In_2Te_3^+ , which are possible secondary ions produced from In_2Te_3 , were obtained (figure 5). These signals showed an increase at the interface. This may suggest the presence of In_2Te_3 at the interface but the possibility of matrix effects cannot be ruled out. Hence, further studies using other suitable analytical techniques which can probe the interface, such as more detailed high-resolution transmission electron microscopy

(TEM) studies are necessary to confirm whether In_2Te_3 is indeed formed at the interface.

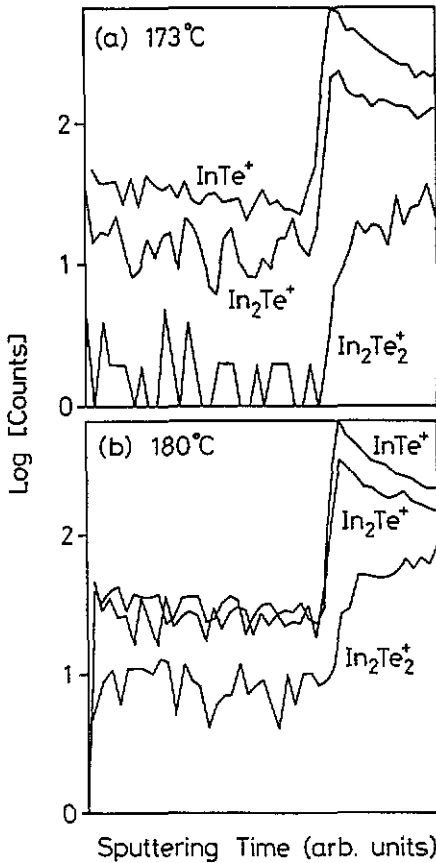


Figure 5. SIMS depth profiles showing InTe^+ , In_2Te^+ and In_2Te_2^+ signals of MBE-grown CdTe/InSb at (a) $T_s = 173^\circ\text{C}$ and (b) $T_s = 180^\circ\text{C}$.

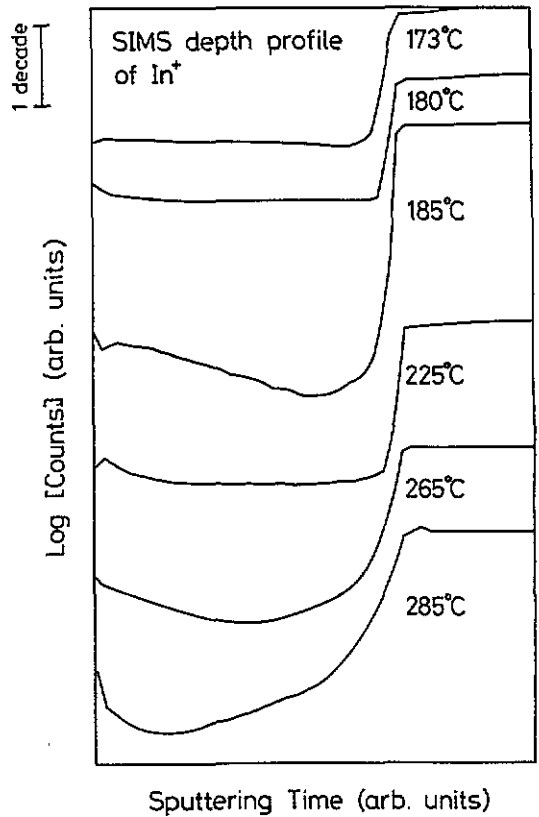


Figure 6. SIMS depth profile showing In^+ signals of MBE-grown CdTe/InSb heterostructures at different T_s . The In^+ profiles are extracted from figure 4.

From the depth profiles in figure 4, it can be seen that indium diffuses outwards from the InSb substrate and accumulates at the CdTe surface for samples that are grown at high T_s . Some diffusion of Te into the InSb substrate is also observed. However, the diffusion of other elements does not appear to occur in significant amounts and this can be explained by the diffusion constants shown in table 3. It is noted from this table that the diffusion constant of In in CdTe is the highest, followed by Te in InSb.

The In^+ profiles of all the six samples are plotted together in figure six for ease of comparison. The diffusion of In across the CdTe/InSb interface from the InSb substrate may be described mathematically by Fick's second law of diffusion:

$$\frac{\partial C(x, t)}{\partial t} = D \frac{\partial^2 C}{\partial x^2}. \quad (2)$$

Since there is a constant supply of In from the InSb substrate, the boundary conditions will be $C(0, t) = C_s = \text{constant}$ and $C(-\infty, t) = 0$ assuming an infinitely thick CdTe overlayer.

Table 3. Diffusion constants of In, Te, Sb and Cd. [15]. Data on diffusion of Sb in CdTe could not be found. $D = D_0 \exp(-E/kT)$

	D_0 ($\text{m}^2 \text{s}^{-1}$)	E (eV)	D at 185 °C	D at 285 °C
Cd in InSb	1×10^{-9}	1.1	7.9×10^{-22}	1.2×10^{-19}
Te in InSb	1.7×10^{-11}	0.57	9.1×10^{-18}	1.2×10^{-16}
Sb in InSb	5×10^{-6}	1.94	2.2×10^{-27}	1.5×10^{-23}
In in InSb	5×10^{-6}	1.81	6.0×10^{-26}	2.2×10^{-22}
In in CdTe	4×10^{-6}	0.9	5.0×10^{-16}	3.0×10^{-14}

The solution to (2) is

$$C(x, t) = C_s \operatorname{erfc} \left(\frac{x}{\sqrt{4Dt}} \right) \quad (3)$$

where erfc is the complementary error function.

By taking $t = 60$ min (typical growth time), a graph of $C(x, t)/C_s$ against distance from the CdTe/InSb interface x at different T_s is obtained (figure 7). In this theoretical model, $C(x, t)$ reaches a value of $\frac{1}{2}C_s$ further from the CdTe/InSb interface than that obtained experimentally (figure 6), i.e. the diffusion of indium is actually slower than that predicted by Fick's law of diffusion. This may be because this simplistic model assumes that indium diffusion occurs in a static CdTe overlayer when in fact indium diffuses during CdTe growth. The time $t = 60$ min chosen is also rather arbitrary in such a growth situation. Furthermore, the presence of dopants, dislocations and defects may significantly alter the diffusion kinetics. Nevertheless, in the absence of a more dynamic model, this simple model serves to illustrate the qualitative growth temperature effects on indium diffusion.

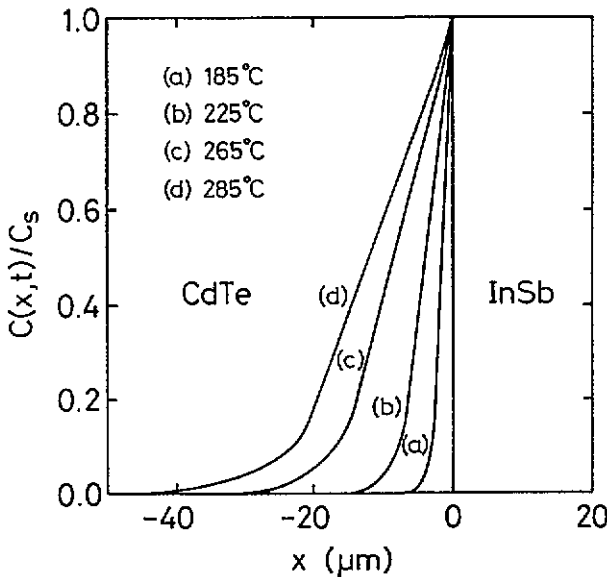


Figure 7. Plot of $C(x, t)/C_s$ against distance from the CdTe/InSb interface for (a) $T_s = 185$ °C; (b) $T_s = 225$ °C; (c) $T_s = 265$ °C; and (d) $T_s = 285$ °C.

The CdTe film thicknesses of our samples are of the order of $1 \mu\text{m}$. Hence, from the results of our model in figure 7, indium should easily diffuse to the sample surface. The

concentration of indium at the surface depends on the evaporation rate of indium. If the evaporation rate is fast, then there will be depletion of indium at the sample surface. If it is slow, then there will be accumulation of indium at the surface. The SIMS depth profiles of In^+ (figure 6) together with the XPS data (figure 3) show that there is actually accumulation of indium at the sample surface.

In discussing the indium interdiffusion phenomenon, it would be appropriate to include possible interfacial chemical reactions between CdTe and InSb resulting in the release of free indium. The large variation in the indium diffusion profiles as a function of substrate temperature suggests that the diffusion process, and not the interfacial indium bond breaking process, is rate limiting [25]. Moreover, our XPS and SIMS data do not reveal any direct information on this interfacial reaction and the relevant bond strengths.

A series of photoluminescence studies together with other techniques have previously been done on the same series of samples with T_s varying from 170 °C to 285 °C [8-10, 12] and an optimum T_s of 185 °C was obtained. Taking the experimental errors into account, our optimum T_s of 180 °C corresponding to the minimum amount of indium diffusing to the surface is well matched to the previously determined optimum T_s of 185 °C obtained from optical, x-ray and electron microscopy studies. The CdTe films grown at temperatures below 200 °C show high crystalline perfection. Their cross-sectional TEM (XTEM) images across the CdTe/InSb interfaces are very ordered and without obvious dislocations. However, numerous dislocations, stacking faults and microcracks were found at the interfaces of the $T_s > 250$ °C samples, and the amount increased with increasing T_s between 225 °C and 285 °C [9].

It can be deduced then that there exists an exact lattice match between CdTe and InSb at about 180 °C (or 185 °C) and that $a(\text{CdTe}) > a(\text{InSb})$ for $T_s < 180$ °C and $a(\text{CdTe}) < a(\text{InSb})$ for $T_s > 180$ °C. This difference between the lattice constants becomes larger as T_s increases beyond 225 °C. When the CdTe film is grown on InSb at high T_s , the larger lattice mismatch will cause dislocations to be produced together with the formation of the CdTe/InSb interface. Hence, indium from the InSb substrate will diffuse more efficiently along these dislocation lines into the CdTe film and reach the surface during the film growth. The results obtained from XPS and SIMS are consistent with the above observations as seen from the large In signals in the XPS spectra for the $T_s = 265$ °C and 285 °C samples. Also, the SIMS profiles of these samples show high concentrations of In in the CdTe film due to In diffusion from the InSb substrate.

The slightly higher indium surface concentration in the $T_s = 173$ °C sample as compared to the $T_s = 180$ °C or 185 °C samples appears to support our hypothesis that lattice mismatch also occurs at $T_s < 180$ °C. However, since indium diffusion is thermally activated, it is not surprising to observe less surface indium at $T_s = 173$ °C than at $T_s = 265$ °C or 285 °C.

4. Conclusions

This study of a series of MBE-grown CdTe/InSb heterostructures at different substrate growth temperatures using the surface analytical techniques of XPS and SIMS yields the following results.

(i) XPS shows that Te exists mainly as TeO_2 at the sample surface, while In exists as In_2O or In_2O_3 . The Cd/Te ratio is greater than one, indicating that the surface is Cd rich.

(ii) Both XPS and SIMS reveal indium interdiffusion directly. The SIMS depth profile and the XPS In 3d and 4d spectra indicate that the indium concentration in the CdTe film grown at high substrate growth temperature, T_s , is significantly higher than at low T_s .

This is attributed to the diffusion of indium across the CdTe/InSb interface from the InSb substrate. Higher T_s enhances the interdiffusion, and hence samples grown at $T_s = 265^\circ\text{C}$ and 285°C have a high indium concentration in the CdTe layer. However, dislocations also play an important role in indium diffusion from the InSb substrate. Samples with lattice mismatch will have more dislocations creating low-energy pathways for indium diffusion. It is deduced that CdTe and InSb have an exact lattice match at about 180°C , and have lattice mismatch at $T < 180^\circ\text{C}$ and $T > 180^\circ\text{C}$ since the surface indium concentration is a minimum for samples grown at $T_s \sim 180^\circ\text{C}$.

(iii) The SIMS depth profiles of the Te^+ signals also show some diffusion of tellurium across the interface into the substrate for samples grown at high T_s .

(iv) The SIMS depth profiles suggest that some In_xTe_y compound, probably In_2Te_3 , forms at the interface, but further studies using other analytical techniques are needed to confirm this

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