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Creating metal saturated growth in MOCVD for CdTe solar cells

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Highlights

- MOVPE growth of CdTe under Cd saturated conditions
- Technique for establishing metal saturated conditions in MOVPE
- Method for determining changes in the II/VI precursor ratio with depletion in a horizontal MOVPE reactor
- Improvement in the open circuit voltage of cadmium telluride photovoltaic solar cells by changing the II/VI ratio

Abstract

Determining the thermodynamic conditions in MOCVD growth of II-VI semiconductor materials is not as **straightforward** as in III-V growth where Group V hydrides are generally used. This paper establishes a technique, using in situ laser reflectometry, to ensure that the thermodynamic equilibrium is under metal saturated growth. This has been applied to the arsenic doping of CdTe solar cells where it was shown that increasing the II/VI precursor ratio resulted in an increase in As dopant incorporation. The growth kinetics were determined by the diisopropyl tellurium (DIPTe) concentration for II/VI precursor ratios above 2. A method is presented where the change in II/VI precursor ratio can be predicted for different positions in a horizontal MOCVD chamber that has, in turn, enabled variation in N_A and the solar cell open circuit voltage (V_{oc}) to be determined as a function of the II/VI precursor ratio. This gives new insight to the thermodynamic drivers in MOCVD growth for improved solar cell V_{oc} and is a method that could be applied to MOCVD of other II-VI semiconductors.

1.0 Introduction

Metal Organic Chemical Vapour Deposition (MOCVD) is often viewed as a non-equilibrium process where the chemical potential of the precursor can be considerably **more than** the equilibrium **value** at the surface. This is useful in driving the growth process and normally becomes regulated by gas phase diffusion through a boundary layer. This means that the known input partial pressures of the precursors do not readily relate to the equivalent equilibrium partial pressures at the growth interface¹. However, the ratio of the cation to anion in the vapour will govern point defect concentrations in the film, a well-known phenomenon with both III-V and II-VI MOCVD. In general, we don't know the equilibrium concentration at the growth interface, and this can be a problem if the equilibrium ratio influences dopant incorporation and point defect formation. The precursor decomposition at the surface will generate an equivalent vapour pressure of the elemental species such as Cd and Te₂. These partial pressures of Cd and Te₂ will drive the equilibrium condition according to the following thermodynamic equation,

$$K_{CdTe} = \frac{a_{CdTe}}{p_{Cd}(p_{Te_2})^{\frac{1}{2}}} \quad (1)$$

where K_{CdTe} is the equilibrium constant for the reaction of CdTe, a_{CdTe} is the activity, p_{Cd} is the partial pressure of Cd and p_{Te_2} is the partial pressure of Te_2 . The variables here are the partial pressures so to balance the equilibrium condition of one of the component partial pressures, such as Cd is increased, then the equilibrium pressure of the other component, Te_2 , must decrease. They cannot work independently. A pressure-temperature phase diagram for CdTe was published by Greenberg² in 1996 and shows that the Cd and Te rich phase boundaries correspond to a good approximation to the saturated vapour pressures of Cd and Te, respectively, for temperatures well below the melting point. The pressure phase width at 400 °C, for single phase CdTe, varies by more than a factor of 10 and will influence both point defect formation and dopant incorporation.

An example of known equilibrium condition is for cadmium mercury telluride (CMT) where the mercury component is introduced as a mercury vapour³. The equilibrium state is more difficult to determine when all the constituents are introduced as organometallic precursors. In the case of CdTe the arsenic incorporation increases with increasing p_{Cd}/p_{Te} ratio but this is within the single phase field for the normal MOCVD growth temperature of 400 °C. The purpose of the present study is to investigate what would happen if the Cd:Te ratio was taken all the way to the Cd saturated phase boundary and to determine the Cd:Te precursor partial pressure ratio at different positions on the substrate.

MOCVD was used in early work on arsenic (group V) doping of CdTe for PV cells and it was demonstrated by Rowlands et al.⁴ that the reaction of the precursor (tris-dimethylaminoarsenic) was a second order reaction introducing arsenic into the CdTe lattice up to concentrations in excess of $1 \times 10^{19} \text{ cm}^{-3}$. Group V doping combined with Cd-rich stoichiometry in polycrystalline (px) CdTe thin film solar cells is a promising alternative route to high performance devices, due to its potential for increased p-type doping concentration^{5,6,7}. Doping is achieved via substitution on the anion (Te) site under thermodynamic growth conditions that favours V_{Te} creation^{7,8}. McCandless et al., demonstrated CdTe thin films having acceptor concentration exceeding 10^{15} cm^{-3} with group V (As, Sb) doping⁸. Ma and co-workers reported the impact of CdTe stoichiometry on device minority carrier lifetime, as significantly improved lifetimes were demonstrated in Cd-rich CdTe, compared to a Te-rich sample⁹, the reason being that Te_{Cd} recombination centres are suppressed in Cd-rich conditions. Nagaoka et al., reported growth of As-doped Cd-rich CdTe single crystals exhibiting p-type doping concentrations between $10^{15} - 10^{18}$, with increasing As concentration¹⁰. Recent results have also been reported where efficient As incorporation in px CdTe:As via in-situ Cd-saturated growth by MOCVD, promoted by varying Cd:Te precursor partial pressure ratio in the range 2 – 4¹¹. The optimum Cd:Te ratio combined with optimised process parameters resulted in significantly improved PV parameters. Therefore the Cd-rich condition is clearly a desirable route for efficient group V doping in CdTe thin films.

2.0 Experimental

MOCVD deposition was carried out in an atmospheric pressure horizontal (Thomas Swan) reactor onto fluorine doped tin oxide (FTO) coated glass substrates provided by First Solar Inc. The precursors and growth sequence are described elsewhere^{7,11} and are summarised here. The buffer layer was $Cd_{0.3}Zn_{0.7}S$ deposited from the precursors dimethylcadmium (DMCd), diethylzinc (DEZn) and ditertiarybutylsulphide (DtBS) at 360 °C. The CdTe absorber layer was grown from the precursors DMCd and diisopropyltelluride (DIPTe) and doped with As using *tris-*

dimethylaminoarsenic (tDMAAs) at temperatures between 350 and 390 °C. Growth nucleation and growth rate was monitored using a triple wavelength laser interferometer supplied by ORS Ltd.

2.1 Characterization

Illuminated current – voltage (J-V) measurements of CdTe:As solar cells, from which open circuit voltage (V_{oc}) was measured, were performed with a Keithley 2400 source measure unit. Cells were illuminated with AM 1.5 (1 sun) light intensity from an Abet Technologies Ltd. solar Sun 2000 solar simulator (Class A), with the light power density calibrated using a GaAs reference cell (ReRa solutions). Secondary-ion mass spectroscopy (SIMS) of As concentration was carried out using a Cameca IMS-4f instrument with Cs⁺ ion source operating at 10 keV energy and 20 nA current (LSA Ltd). Acceptor doping concentration (N_A) was obtained from capacitance – voltage (C-V) characteristics measured in the dark using a Solartron Modulab Impedance Analyzer (Ametek Inc.), at a frequency of 300 Hz, with a constant 20 mV r.m.s signal superimposed on a DC bias voltage in the range of – 4 to +2 V. N_A values reported herein were read from the trough of C-V scans, converted to carrier concentration vs. depletion width. CdTe thin film thickness profiles were obtained using a Dektak 150 profilometer (Veeco).

The method for determining the Cd-rich phase boundary at different substrate temperatures in the MOCVD reactor was described in a recent paper by Oklobia et al.¹¹ where a laser interferometer was used to detect the onset of Cd condensation onto a glass substrate. For the p_{Cd} to be sufficiently high to achieve Cd supersaturation it was necessary to reduce the deposition temperature from the usual 400 °C to 350 °C using a DM Cd flow in the range from 35 sccm to 115 sccm at a bubbler temperature of 2.3°C. The flow was increased in steps of 5 sccm and held for a period of 5 mins. before increasing to the next flow and again turning on the DM Cd flow at the new setting. The reflectance data collected for a fixed substrate temperature at 350 °C is shown in Fig. 1. The initial exposure at 34 sccm gave no measurable decrease in reflectance, indicating that at this concentration the p_{Cd} was not saturated. By 39 sccm a small decrease could be seen, indicating the onset of Cd saturation. For growth experiments there would be depletion due to growth of the CdTe film so an excess flow of DM Cd was selected at 64 sccm for all the Cd-saturated CdTe growth experiments. A photograph of the glass coupons following the Cd saturation experiment can be seen in Fig. 2 where a plume can be seen from the Cd deposit that has accumulated on the surface.

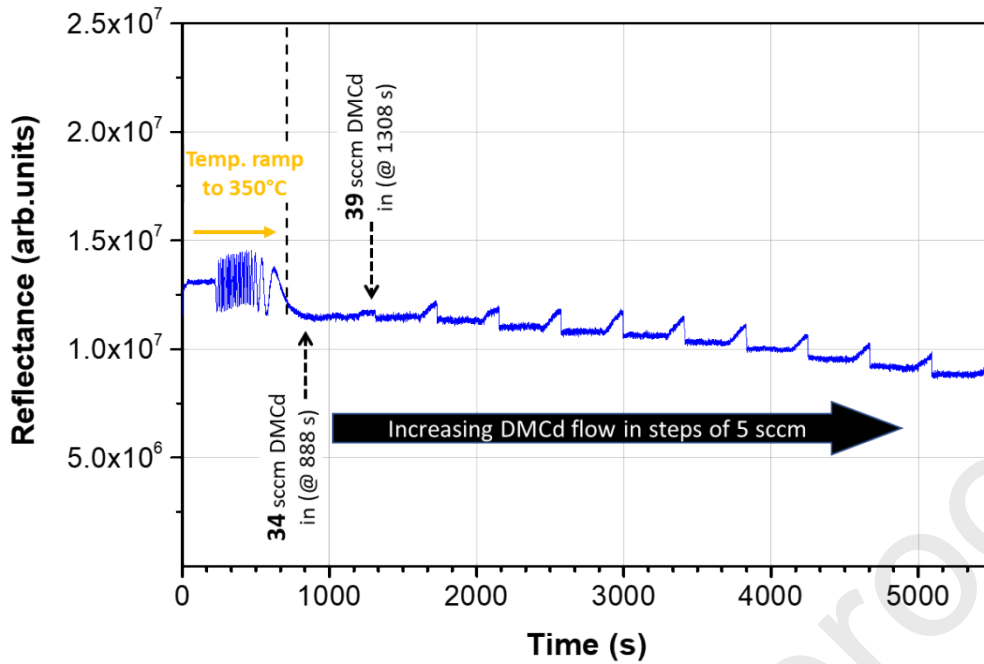


Fig. 1: Laser reflectance monitoring of DMCD exposure to glass substrate in a horizontal MOCVD reactor at a fixed temperature of 350 °C and with varying the DMCD flow.

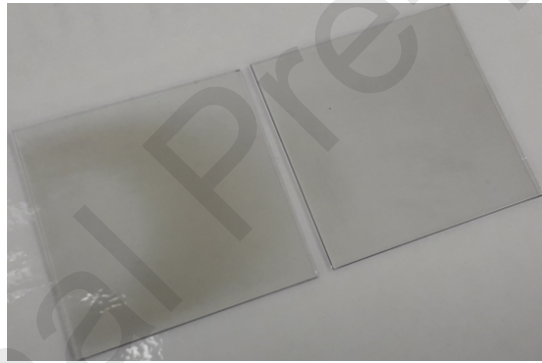


Fig. 2: The Cd coating plume on the 5x5 cm square glass coupons after the Cd saturation experiment.

3.0 Results and Discussion

3.1 Growth as a function of reactor inlet concentrations

A series of MOCVD CdTe:As growth runs, with complete PV devices, were carried out where the DMCD flow was kept constant at 64 sccm (equivalent to partial pressure of 31.5 Pa) and the DIPTe partial pressure was varied between 6.5 and 15.8 Pa to change the reactor inlet II/VI ratio. This ensured that the Cd partial pressure was kept on the Cd rich phase boundary while the DIPTe partial pressure changes were used to change the II/VI ratio. The range over which the p_{DIPTe} determined the growth kinetics is shown in the linear region in Fig. 3. The growth rate was determined by in situ laser reflectometry at a position 60 mm downstream from the leading edge of the first glass substrate (Fig. 4). The maximum p_{DIPTe} for linear growth rate dependence was 10.8 Pa, corresponding to a $p_{\text{DMCD}}/p_{\text{DIPTe}}$ ratio of 3. At the ratio of 2, the growth rate is sublinear in p_{DIPTe} indicating that growth rate was dependant on the DMCD as well as the DIPTe partial pressures.

SIMS depth profile analysis was carried out on these device structures to determine the bulk As concentration for As precursor flow kept in the range of 1.0 to 1.5 sccm. The dependence of As incorporation as a function of the tDMAAs precursor concentration has been reported previously

and was shown to be a second order reaction in As concentration⁴. Fig. 5 shows the calibrated SIMS concentrations, taken from depth profiles, for the different inlet $p_{\text{DMCd}}/p_{\text{DIPTe}}$ ratios. This shows a clear trend for increasing As dopant incorporation for higher ratios which is consistent with increasing the Te vacancy concentration, under thermodynamic equilibrium.

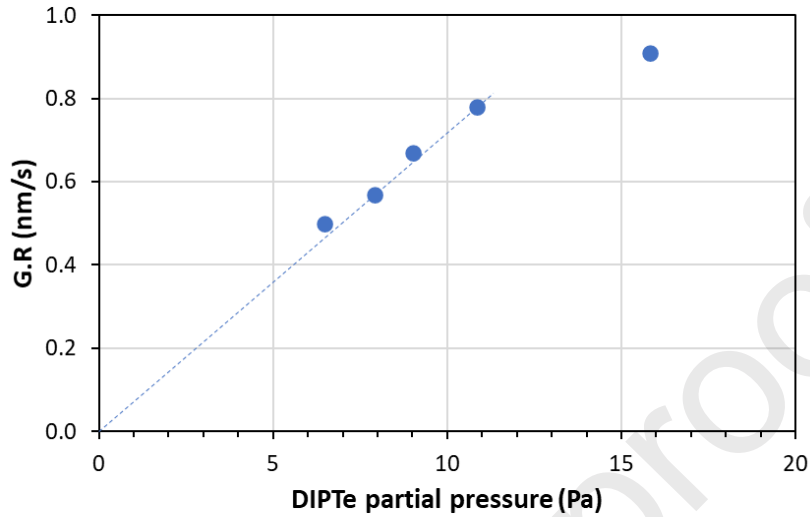


Fig. 3: Growth rate of CdTe films grown onto CdZnS/FTO coated glass substrates as a function of DIPTe inlet partial pressure in Pascals

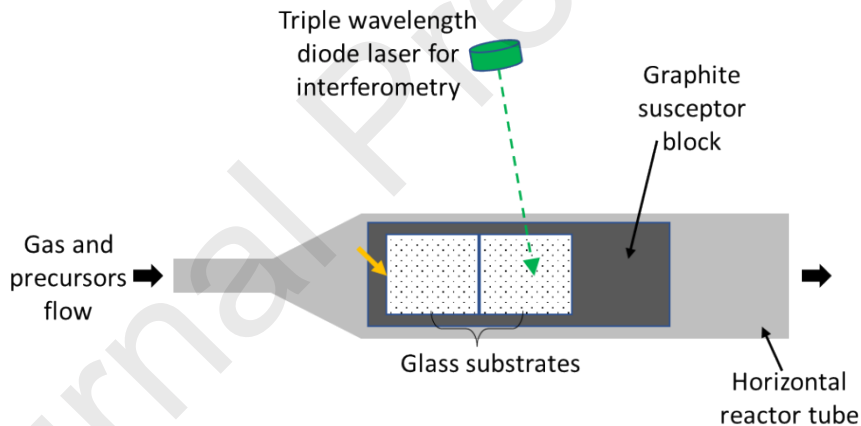


Fig. 4: Schematic showing the arrangement of glass substrates on a graphite susceptor block in a horizontal reactor for CdTe:As device structure growth (orange arrow indicating leading edge of first glass substrate)

The acceptor doping concentrations (N_A), determined from C-V measurements on devices from the upstream (u/s) and downstream (d/s) substrates as a function of $p_{\text{DMCd}}/p_{\text{DIPTe}}$ is shown in Fig. 6. The value of N_A increases strongly with $p_{\text{DMCd}}/p_{\text{DIPTe}}$ but appears to saturate above or possibly decrease above 4. There is also a consistently higher value of N_A for the d/s substrate. While depletion of DM Cd, and to a greater extent DIPTe, occurs d/s it is observed that the As concentration increases. This might be due to a variation in the $p_{\text{DMCd}}/p_{\text{DIPTe}}$ going d/s due to different proportional rates of depletion of DM Cd and DIPTe occurring. All the values of p_{DMCd} and p_{DIPTe} quoted so far are the inlet concentrations but the thermodynamic condition, controlling the As incorporation and value of N_A , will be changing between as the surface Cd and Te concentrations change.

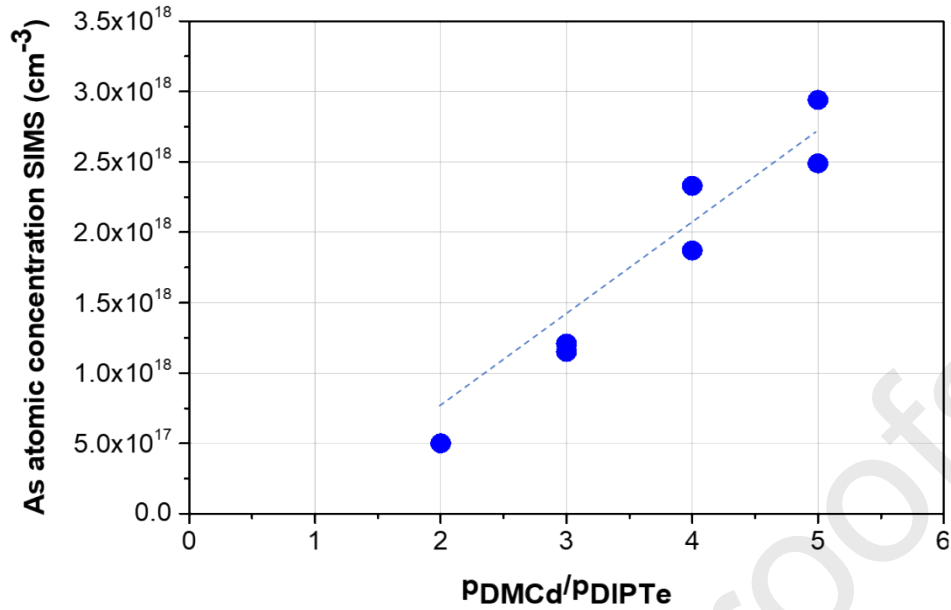


Fig. 5: Arsenic doping concentration determined by SIMS as a function of the inlet ratio of $p_{\text{DMCd}}/p_{\text{DIPTe}}$ partial pressures. The tDMAAs flow was kept constant between 1.0 and 1.5 sccm. The dotted line provides a guide but does not imply a linear trend.

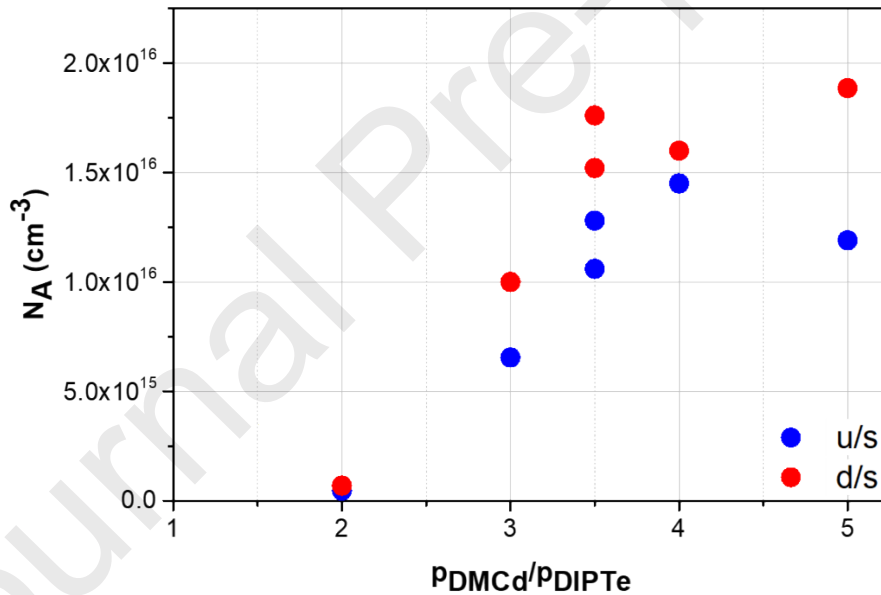


Fig. 6: Acceptor concentration measured by C-V taken from devices on the upstream (u/s) and downstream (d/s) substrates for different $p_{\text{DMCd}}/p_{\text{DIPTe}}$.

3.2 Estimating precursor concentrations at different positions on the substrate

The depletion of precursor concentrations going downstream in a horizontal reactor can be turned to advantage to explore the true dependence of thermodynamic conditions for defects and device performance. A method was developed whereby an estimate could be made based on the known inlet precursor concentrations, relating this to the initial film thickness at the inlet end of the susceptor. The method is applicable to the linear region of Fig. 3 where it can be assumed that the growth rate, and therefore CdTe thickness, is proportional to the local partial pressure of DIPTe. $p_{\text{DIPTe}}(x)$ is the partial pressure at distance x from the leading edge of the first substrate. The known

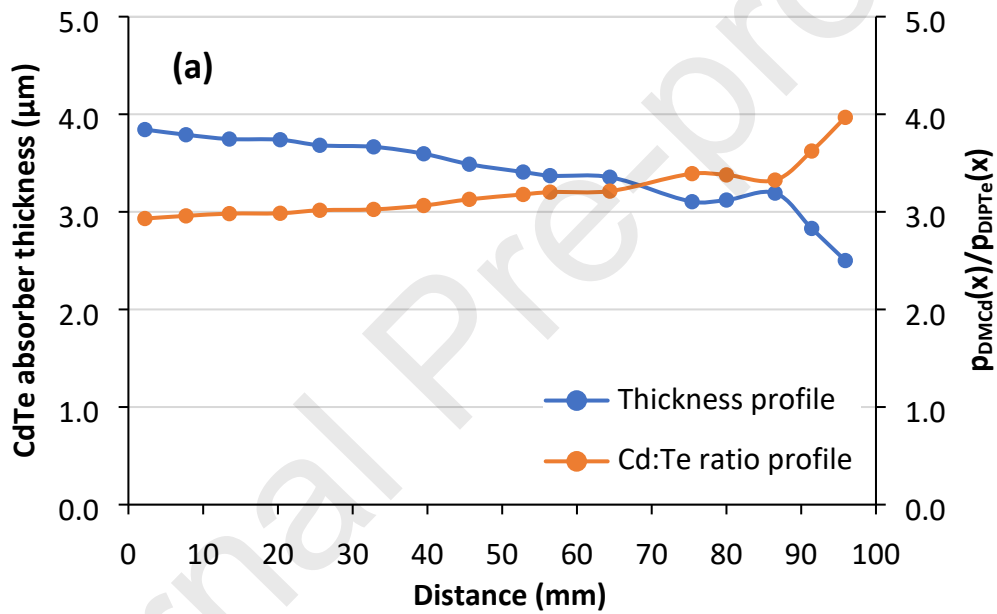
partial pressure of DIPTe for the inlet is assumed to be the value of $p_{DIPTe}(0)$. Therefore, a simple correction factor can be calculated for estimating $p_{DIPTe}(x)$ based on the different film thicknesses, $d(0)$ and $d(x)$. Examples of thickness measurement using a profilometer along the axis of the horizontal reactor is given in Fig. 7. The estimated partial pressure of DIPTe for any generalised position along the axis of the reactor is given by the following equation.

$$p_{DIPTe}(x) = \frac{p_{DIPTe}(0)d(x)}{d(0)} \quad (2)$$

The partial pressure of DMCD ($p_{DMCd}(x)$) can be calculated assuming 1:1 depletion of DMCD and DIPTe. This is justified for CdTe grown in the kinetic temperature regime from work by Mullin et al.¹² Therefore, $p_{DMCd}(x)$ is determined by the following equation.

$$p_{DMCd}(x) = p_{DMCd}(0) - (p_{DIPTe}(0) - p_{DIPTe}(x)) \quad (3)$$

Where $p_{DMCd}(0)$ is known from the inlet partial pressure of DMCD and $p_{DIPTe}(x)$ is determined by equation (2).



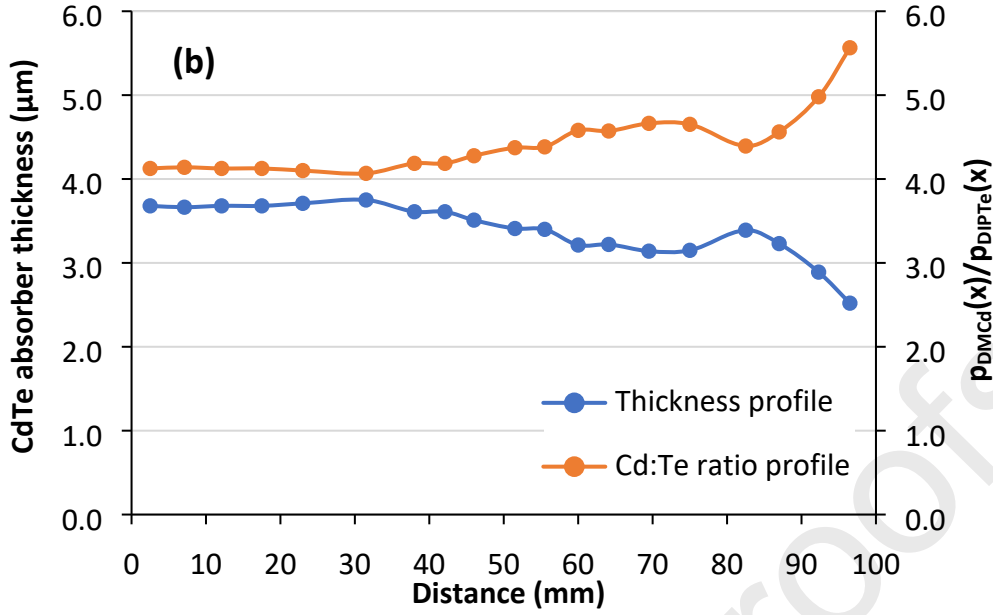


Fig. 7: Variation in measured film thickness and calculated $p_{DMCd}(x)/p_{DIPTe}(x)$ as a function of axial position in the horizontal reactor from the leading edge of the first substrate for an inlet $p_{DMCd}(0)/p_{DIPTe}(0)$ of (a) 3 and (b) 4. The separation between the u/s and d/s glass substrates is at 50 mm.

Finally, the precursor ratio $p_{DMCd}(x)/p_{DIPTe}(x)$ can be calculated for each position along the growth zone of the horizontal MOCVD reactor by combining equations (2) and (3).

$$p_{DMCd}(x)/p_{DIPTe}(x) = \frac{d(0)[p_{DMCd}(0) - p_{DIPTe}(0) + p_{DIPTe}(0)d(x)/d(0)]}{p_{DIPTe}(0)d(x)} \quad (4)$$

The ratio for different positions on the glass substrates is shown in Fig. 7 (a) for inlet precursor ratio of 3.0. It can be seen that the actual precursor ratio increases from around 3 to close to 4, a substantial change from the inlet ratio. Another example is given in Fig. 7(b) for an inlet ratio of 4 where the ratio increases to in excess of 5. In both examples the first 30 mm shows relatively little change where it is expected that the concentration can be maintained from the free flow. However, once the flow becomes fully developed significant depletion occurs.

The estimate of the precursor ratio for different positions on the substrates can now be related to variations in PV device parameters. In particular the paper by Oklobia et al. showed significant increase in V_{oc} with increase in precursor ratio at the Cd saturated phase boundary¹¹. The V_{oc} for different positions on the u/s and d/s substrates can now be plotted as a function of the precursor ratio $p_{DMCd}(x)/p_{DIPTe}(x)$ and is shown in Fig. 8.

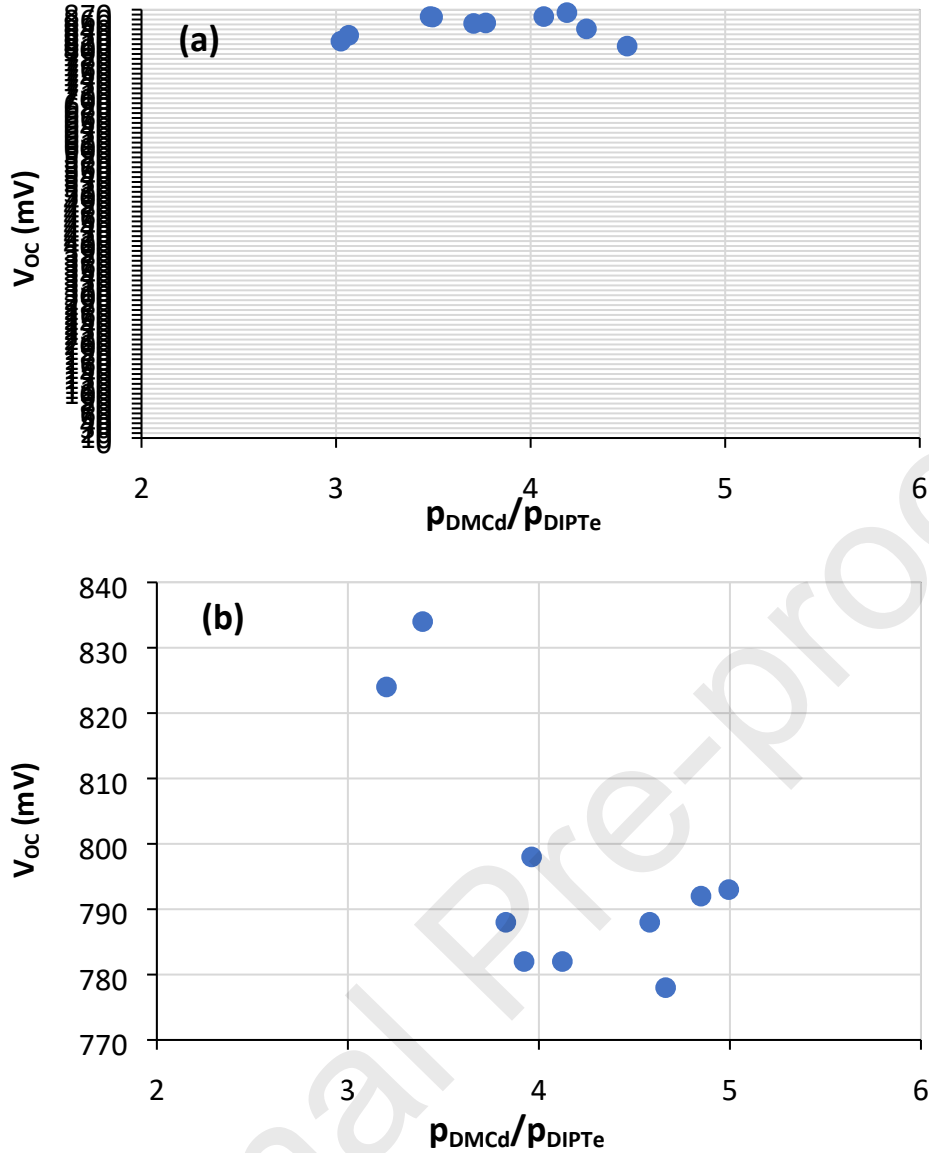


Fig. 8: V_{oc} for 5x5 mm PV devices as a function of $p_{DMCd}(x)/p_{DIPTe}(x)$ for (a) u/s substrate and (b) d/s substrate.

The results are plotted for the u/s and d/s substrates and show a different behaviour for each. In both cases the higher V_{oc} values (>800 mV) are only achieved for a very specific range of precursor ratios. This demonstrates the importance of the thermodynamic conditions in achieving good device performance. For the u/s substrate the higher values of V_{oc} are achieved for $p_{DMCd}(x)/p_{DIPTe}(x)$ from 3.5 to 4.2 and for the d/s substrate for ratios of 3 to 3.5.

The difference in optimum precursor ratios between u/s and d/s substrates could be related to the dependence of the As dopant incorporation with precursor ratios shown in Fig. 5 and the acceptor concentration in Fig. 6. A consistent behaviour of increasing As concentration from the u/s to the d/s substrates has been observed. It has also been observed that for N_A above $1-2 \times 10^{16} \text{ cm}^{-3}$ a decrease in V_{oc} occurs which is related to an increase in associated trap defects^{7, 13, 14, 15}. On this basis it is expected that for higher precursor ratios that the As concentration and N_A increase to the point where V_{oc} is being dominated by the concentration of deep level defects in the bulk of the CdTe film.

4.0 Conclusions

MOCVD has the capability of a wide range of thermodynamic conditions through changing precursor partial pressures and the growth temperature. It is often not well understood what thermodynamic conditions exist at the growth interface as this requires a knowledge of precursor decomposition and the resultant element yield. This paper sets out a method for establishing metal rich growth to drive Group V substitutional doping. It has been shown in this paper that it is possible for the Te precursor partial pressure to govern the growth kinetics while the Cd precursor partial pressure can control the thermodynamics.

A more detailed analysis of the relationship between the V_{oc} of As doped CdTe solar cells and the depleted precursor partial pressure ratio along a horizontal reaction tube has been made. This method could be applied to other MOCVD growth materials and depends on measurement of growth rate along the axis of the horizontal reaction tube. For the CdTe solar cells in this study it has been possible to correlate V_{oc} with $p_{DMCd}(x) / p_{DIPTe}(x)$ for the upstream and downstream devices.

A different optimum value of the precursor ratio is seen for upstream and downstream, 4.0 and 3.3 respectively. This method of estimating local variations in the precursor II/VI ratio can help to better inform the relationship between the thermodynamic conditions and device performance.

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CREDIT AUTHOR STATEMENT

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S. J. C. Irvine¹ - conceived the method, developed the model, wrote the manuscript

O. Oklobia¹ – design of experiments, carried out experimental work, assisted with writing manuscript

S. Jones¹ – Carried out MOCVD growth experiments

D. A. Lamb¹ – contributed to design of experiments, interpretation of results and review of manuscript

G. Kartopu² - contributed to design of experiments and interpretation of results

D. Lu³ - contributed to design of experiments and interpretation of results

G. Xiong³ - interpretation of results and review of manuscript

Declaration of interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

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