Real-time mass spectroscopy of reflected fluxes during MBE growth of HgCdTe

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Abstract

The interaction between molecular beams present in the growth of HgCdTe was studied using reflection mass spectroscopy (REMS). The incident Hg flux was found to strongly influence the amount of Te and Cd reflected from the growth surface, and also, the incident Te flux influences the amount of Cd reflected from the surface. Based on these observations, it appears that the CdTe growth rate is influenced by the amount of excess Te and/or Hg available at the growth surface. A technique to control accurately the layer mole fraction by measuring the ratio of Cd to Cd+Te fluxes during growth has been investigated. Results of post growth layer characterisation by SIMS and infrared transmission indicate a strong correlation between the REMS ratio and the mole-fraction of the resulting layer. This technique has successfully been used to correct for long term and short term drifts in effusion cell output.

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I. INTRODUCTION

Advanced infra-red sensor devices based on $\text{Hg}_{(1-x)}\text{Cd}_{(x)}\text{Te}$ heterostructures are generally fabricated on MBE-grown material. The low growth temperature of MBE allows the formation of abrupt junctions and controlled doping profiles necessary for the high-performance¹, multi-spectral² and higher operating temperature devices³ that have been demonstrated in HgCdTe .

The defect density and mole-fraction x of a MBE-grown $\text{Hg}_{(1-x)}\text{Cd}_{(x)}\text{Te}$ layer is very sensitive to the molecular flux density and substrate temperature. Subsequently, the yield of MBE-grown HgCdTe is directly related to the control one has over these parameters. While several techniques exist for substrate temperature control,^{4,5} real-time control of molecular fluxes is more difficult. The molecular flux from a standard Te effusion cell is particularly difficult to control because the source material sublimates, and tends to break into smaller pieces over time. This changes both the total surface area of the charge and the temperature distribution within the charge, both of which lead to unstable flux densities. For typical HgCdTe growth rates, a change in the Te cell temperature of only 1.5°C is sufficient to change the mole-fraction x of $\text{Hg}_{(0.20)}\text{Cd}_{(0.80)}\text{Te}$ by ± 0.01 which corresponds to a shift in bandgap of 19% or peak response wavelength shift of $3.5\mu\text{m}$. Another difficulty in composition control is caused by the high Hg background pressure present in the MBE chamber between growth runs ($\sim 10^{-7}$ Torr) which causes large errors in beam equivalent pressure measurements of Te and CdTe fluxes due to the reaction between Hg and Te.

The most successful technique to date for controlling the molecular fluxes is via feedback from composition measurements made by *in-situ* ellipsometry⁶. While some success has been demonstrated using this method, ellipsometry requires wobble-free substrate rotation, and a significant amount of material to be grown before any flux correction can be made. Furthermore flux variations that cause a change in defect density without a concomitant change in x are not detected by ellipsometry. Direct measurement of Cd fluxes during growth have been demonstrated by Olson *et al.*⁷ using optical absorption techniques, but no further use of this technique has been published.

RHEED intensity oscillations are routinely used to calibrate growth rates and composition in other material systems, where epitaxy proceeds in a layer-by-layer growth mode. The preferred crystal plane for HgCdTe growth is (211)B, so that growth proceeds by a step-flow

process that suppresses the formation of twins. Unfortunately, RHEED intensity oscillations do not occur during step-flow epitaxial growth⁸, so growth rates and flux calibrations cannot be performed using this technique.

Reflection mass spectroscopy (REMS) is an *in-situ* method of measuring molecular fluxes which emanate from the growth surface. A quadrupole mass spectrometer (QMS) is typically mounted in an unused effusion cell port, so that the ioniser is in line-of-sight to the growth surface, while other molecular sources are blocked by cryopanel. The reflected and re-evaporated fluxes offer some insight into the MBE growth process, and, when combined with a suitable growth model and/or shuttering sequence, have been used to calculate the impinging fluxes in real-time for growth of III-V materials^{9–13} and group II-VI materials¹⁴.

In this work we investigate for the first time the use of reflection mass spectrometry (REMS) as a tool for flux control during growth of HgCdTe, and also describe some new insights into the growth process which are revealed by the results.

II. THEORY

We consider the REMS current recorded by the mass spectrometer to be the sum of several components, i.e.

$$I_X^{QMS} = A_{EM} K_X \left(F_X^{Ref} + F_X^{Rev} + F_X^{Bg} \right), \quad (1)$$

where F^{Ref} is a fraction of the incident molecular flux that is reflected from the surface (i.e. the substrate and/or molyblock) before being chemically absorbed into the layer, F^{Rev} represents re-evaporation from previously grown surface layer, and F^{Bg} describes background sources. F^{Rev} is in effect due to Langmuir type evaporation of the existing layer¹⁵, and so will be present when the incident molecular flux is shuttered. K_X is the sensitivity of the QMS to species X (coulombs m²/molecule), and A_{EM} is the gain of the electron multiplier inside the QMS. Assumptions implied in this equation are constant ionisation cross-sections, constant source-substrate-QMS geometry, constant angular distribution of reflected fluxes, and no other sources of ionisation other than the spectrometer itself. Note that as the reflecting surface begins to roughen, it is likely that the same reflected flux will be distributed across a wider angular range, thus reducing the ion current recorded by the QMS.

The substrate temperature for MBE growth of HgCdTe is close to 185°C with a high Hg

flux ($J_{Hg} \approx 2 \times 10^4$ Torr BEP). Under these conditions F^{Rev} of all species were found to be negligible when compared to F^{Ref} . At higher temperatures or very low incident fluxes, F^{Rev} becomes dominant.

The incorporation coefficient of a species may be written as,

$$\sigma_X = \frac{J_X - F_X^{Ref} - F_X^{Rev}}{J_X}, \quad (2)$$

where J_X is the incident flux of species X . Assuming single-phase growth, the mole-fraction of the growing $Hg_{(1-x)}Cd_{(x)}Te$ layer may then be written as

$$x = \frac{\sigma_{Cd} \times J_{Cd}}{(\sigma_{Cd} \times J_{Cd}) + (\sigma_{Hg} \times J_{Hg})} = \frac{\sigma_{Cd} \times J_{Cd}}{\sigma_{Te} \times J_{Te}} \quad (3)$$

The problem of calculating the mole-fraction of the growing layer from REMS data thus reduces to a problem of knowing the incorporation coefficients for each species, and keeping all other experimental conditions constant. In general, the incorporation coefficients are a function of surface temperature, surface composition, and incident fluxes of the other species being evaporated.

III. EXPERIMENTAL

Experiments were performed using a RIBER 32 MBE chamber, equipped with standard large aperture conical crucibles for CdTe and Te source materials. A valved cracker cell was used to generate As_2 and As_4 fluxes (a p-type dopant in HgCdTe) and a non-valved quartz crucible in communication with an external reservoir was used to evaporate Hg. HgCdTe layers described in this work were all grown on 10mm x 10mm lattice-matched $Cd_{(0.04)}Zn_{(0.96)}Te$ (211)B substrates. It is important to note that the CdZnTe substrates cover less than 5% of the surface area of the two inch molybdenum susceptor (molyblock), so that most of the reflected signal is actually reflected from polycrystalline HgCdTe growing on the molyblock. In later experiments a 2 inch Si wafer was mounted between the CdZnTe substrate and the molyblock to provide a consistent surface for the growth of polycrystalline HgCdTe. All substrates were rotated at 1 revolution per second during growth and measurement.

A SRS-RGA300 mass spectrometer was mounted on the MBE in place of an effusion cell, so that the ionizer was within a port in the LN_2 cryopanel which also surrounds the effusion cells, and was in line-on-sight to the substrate surface. The mass spectrometer was detuned

to a resolution of ~ 2 AMU, the electron multiplier was operated at 1800V and the emission current was set to 3.5mA for all experiments to improve the signal-to-noise ratio.

The spectrometer was programmed to perform a series of short scans across the mass peaks of interest. In these experiments they include As_2^{++} or As^+ at 75AMU, Hg^{++} at 101AMU, Cd^+ at 112AMU, Te_2^{++} or Te^+ at 130AMU, As_4^{++} or As_2^+ at 150 AMU, and Te_2^+ at 260 AMU. For each species, the area under the peaks was summed, and divided by the number of points to give an average REMS current for each species. Although the Te_2^+ signal at 260 AMU was the strongest Te signal (giving a slightly better signal to noise ratio), for the sake of consistency with earlier data, we use only the Te signal at 130AMU in this work. The width of the scan across the Hg peak was limited so that the electron multiplier did not saturate, hence relatively low REMS currents were recorded despite the reflected Hg flux actually being much greater than the reflected Cd or Te fluxes.

Some initial experiments were performed to periodically measure the gain of the electron multiplier by switching to Faraday cup mode and tracking the ratio of the results for the Hg peak. This showed that there was not any significant long term drift in the gain of the electron multiplier, but only short term instability caused by the calibration procedure itself, we therefore chose to treat the electron multiplier gain as being constant.

The average mole-fraction and thickness of the HgCdTe layers was measured after growth by a fit to room temperature infrared transmission data. Secondary Ion Mass Spectroscopy (SIMS) was performed on some samples to observe step changes in mole-fraction intentionally introduced during growth¹⁶.

IV. RESULTS

REMS data was recorded for a variety of growth conditions to (a) elucidate the HgCdTe growth process from a qualitative understanding of the reflected flux intensities, and (b) to investigate the possibility of using REMS data to control beam-flux intensities and thus layer composition.

A. Qualitative analysis

1. Effect of incident Hg flux on reflected flux densities

From REMS observations, the most significant factor influencing the reflected fluxes and incorporation coefficients of Cd and Te is the co-incident Hg flux. To understand this relationship a molybdenum target at a typical growth temperature of 185.5 °C was subjected to a varying Hg flux and constant Cd and Te incident fluxes generated from a CdTe effusion cell at 519°C. The relationship between the reflected Cd flux and the reflected Hg flux is shown in Fig. 1. Because there exists only a narrow range of conditions suitable for smooth epitaxy of HgCdTe, these experiments will generally result in polycrystalline (rough) growth of HgCdTe, and so analysis of the results must bear this in mind. Similarly, during normal growth of HgCdTe on small substrates, most of the reflected signal will arise from the rough polycrystalline layer growing around the substrate. We would expect the reflection from smooth surfaces to be greater in magnitude, as is observed in the first few minutes of growth when 2 inch Si carrier wafers are used. It remains to be answered if the reflections from smooth HgCdTe surfaces follow the same relations as those observed here for rough surfaces, although from the theory. In a similar fashion, a bare molybdenum was subjected to a constant Te flux and varying Hg flux, as shown in Fig. 2. In a final experiment REMS data was recorded as a function of varying Hg flux with *both* CdTe and Te cells open. The general shape of the reflected Cd and Te fluxes versus Hg flux in this experiment was similar to those shown in Figs. 1 and 2, and is not presented in this work.

In these experiments, the temperature of the Hg cell, and the total chamber pressure show a linear relation with the Hg REMS current, so we consider the Hg REMS current to be analogous to the incident Hg flux. Both the reflected Cd and Te fluxes are strongly influenced by the presence of a coincident Hg flux. This interaction between fluxes initially appeared to be evidence of thermal equilibrium between re-evaporated fluxes and the previously grown layer as described by the mass action law and equations given by Colin and Sakuli¹⁷. However, the REMS signals are not present when the source shutters are closed, consistent with the expectation that the re-evaporation of chemically bonded Cd will be very small at the 185°C growth temperature. If incident fluxes were simply elastically reflected from the surface, then it is unlikely that such a complex relation would exist between the

reflected fluxes. We therefore consider the reflected fluxes to arise from a physisorbed layer of species held to the growth surface by van der Waals forces. The nature of a physisorbed Near Surface Transition Layer (NSTL) in MBE is described in detail by Madhukar¹⁸ and Herman¹⁹. We propose that species in the NSTL which are not able to form chemical bonds with the existing layer are ejected to form the reflected fluxes which are measured in these experiments.

Figure 1 shows that as the incident Hg flux increases, more Cd is reflected from the surface, hence less Cd is incorporated into the layer. This suggests that the presence of Hg on the growth surface in some way prevents Cd from bonding to the surface, either as Cd metal or as CdTe. The reflected Te flux was also recorded during this experiment and shows a similar shape to the curve in Fig. 2. As the reflected Hg flux signal increases from 100 to 200pA, both the reflected Cd and Te flux signals increase, i.e. the Hg appears to be reducing the incorporation of *both* Cd and Te and not just replacing the Cd in telluride formation. For Hg flux signals above 200pA the Te reflected flux signal decreases, while the Cd reflected flux signal continues to increase. In this region, it appears that more Te is incorporating into the layer as HgTe, and even less Cd is being incorporated, either because there is less Te available for CdTe formation and/or because there is more Hg on the surface preventing incorporation. It would be useful to conduct this experiment using a Cd cell instead of the CdTe cell used here to further investigate this theory.

The relation between reflected Te and Hg is shown in Fig. 2 for Te and Hg fluxes alone although a similar shape is observed when Cd is being co-evaporated. As the Hg flux increases, the reflection of Te rapidly increases to a maximum, and then reduces as the Hg flux is increased further. For low Hg fluxes, we suggest that Te is incorporating primarily as metallic precipitates (as is sometimes observed in HgCdTe grown under low Hg flux), and that this incorporation is impeded by an increasing concentration of Hg in the NSTL. Beyond a certain level, the formation of Hg-Te bonds with the existing layer is the dominant route of Te incorporation, and thus the incorporation of Te increases with increasing Hg flux.

2. *Relation between reflected Cd and reflected Te flux*

To investigate the relation between reflected Cd and Te fluxes, a 2 inch silicon substrate at 187 °C was subjected to a range of Te and Cd fluxes supplied from Te and CdTe effusion

sources, as well as a constant Hg flux of $J_{Hg} \approx 2 \times 10^{-4}$ Torr BEP as is typically used for growth of HgCdTe. No significant change in the reflected Hg flux was observed during this experiment. As is evident from Figs 3 and 4 the reflected Cd flux increases when the incident Te flux is reduced. At the end of the growth of a $\text{Hg}_{(0.3)}\text{Cd}_{(0.7)}\text{Te}$ layer, a 100nm thick passivation layer of $\text{Hg}_{(0.1)}\text{Cd}_{(0.9)}\text{Te}$ is typically grown by closing the Te shutter only and leaving CdTe and Hg effusion cells open (a Hg flux is required to stabilise the HgCdTe layer). As can be seen in Fig. 4, the reflected Cd flux increases during growth of the passivation layer, solely due to a reduction in the incident Te flux. This effect again suggests either competition between Cd and Hg to bond with the available Te and/or a reduction in the Te flux leading to more unbound Hg on the surface which in turn reduces the incorporation of Cd.

Although the CdTe evaporates congruently from the effusion cell at 520°C, the condensation is not necessarily congruent, and it appears that Cd-Te condensation is not congruent under a high Hg flux, and in fact depends on the co-incident Hg (Fig. 1) and Te fluxes (Fig. 3). This complicates the relation between layer mole-fraction and effusion source temperatures, since it appears that the growth rate of CdTe in the alloy is not controlled solely by the temperature of the CdTe cell, as was previously thought, but also by the amount of ‘extra’ Te supplied from the Te cell and also by the concentration of Hg on the surface. Despite this, for typical Te and Hg fluxes used for $\text{Hg}_{(0.7)}\text{Cd}_{(0.3)}\text{Te}$, the reflected Cd flux is more strongly influenced by the temperature of the CdTe cell than the temperature of the Te or Hg cell, and thus more strongly controlled by the incident Cd flux than the incident Te or Hg flux.

The Te and Cd profiles shown in Fig. 4 show a decay in intensity at the start of growth, which we attribute to roughening of the silicon surface as it becomes coated in polycrystalline HgCdTe. This effect is less pronounced when the target is already rough (e.g. molybdenum coated from a previous growth run), but it is difficult to ensure a consistent reflecting surface in this way. For the sake of consistency between growth runs, we switched to mounting CdZnTe wafers on 2 inch diameter Si wafers, which were then mounted on the 2 inch diameter molybdenum blocks with Gallium.

B. Quantitative Analysis

From equations 1,2,and 3, and the results of the previous section, it is apparent that the intensities of the reflected fluxes are closely linked to the intensity of the incident fluxes, and thus might be used to control the composition of the growing layer through feedback to the effusion cell temperatures.

1. Average REMS current ratio compared to mole-fraction derived from FTIR

We recorded the reflected flux intensities for a series of HgCdTe growths, and found a correlation between the average ratio of $\text{Cd}/(\text{Cd}+\text{Te})$ and the mole-fraction x of the layer as determined from room temperature infrared transmission measurement. Figure 5 shows these results for layers where the substrate was mounted directly on the molybdenum block, and also where the substrate was mounted on a two inch Si carrier substrate. The numbers in brackets show the average Te cell temperature used for that growth. As is typically done in HgCdTe growth, the Hg flux is checked by measuring the total equivalent pressure in the growth chamber with a Bayard-Alpert ion gauge, mounted beside the substrate. In all these growths this total pressure was maintained at 1.0×10^{-5} Torr by slight adjustments to the level of Hg in the cell and the CdTe cell was set at 520°C . The range of Te cell temperatures used gives an indication of the poor run-to-run stability of this cell.

After a number of growths the correlation between the reflected flux ratio and the layer mole-fraction became more convincing, and so for later growths, the Te cell temperature was manually adjusted during growth to maintain the reflected flux ratio constant. In fact, the Te cell had become slightly blocked by this stage, but by increasing the cell temperature to above 320°C , we were able to maintain the reflected flux ratio within desired bounds and grow several more layers around $x=0.31$ and $x=0.29$.

2. SIMS profiles compared with REMS profiles

In one sample the Te cell temperature was raised by 2°C after 1 hour of growth, to create a sudden decrease in the mole-fraction x of the layer as shown in Fig.6, and a corresponding change of 0.015 was clearly observed in the REMS ratio during growth. After growth, the sample was analysed using Secondary Ion Mass Spectroscopy. The compositional profile

extracted from the SIMS data clearly shows the expected step in mole-fraction from $x=0.255$ to $x=0.24$.

V. CONCLUSIONS

The relations between reflected Hg, Cd and Te fluxes during HgCdTe growth reveal that low Hg fluxes act to prevent incorporation of Cd and Te through interactions in the near surface transition layer, and for higher Hg fluxes, the incorporation Te increases probably through the formation of Hg-Te bonds. For constant Cd and Hg fluxes, a decrease in the Te flux leads to an increase in the reflected Cd flux, which suggests that either the Cd incorporation is limited by the availability of Te, or that the concentration of Hg in the NSTL increases when Te is reduced since less is consumed in the growth of Hg-Te, and this increase in surface Hg acts to prevent incorporation of Cd.

The growth of very high x fraction HgCdTe passivation layers is difficult since a Hg flux is required to prevent decomposition of underlying HgCdTe layer, some of which incorporates into the passivation layer. Figure 2 suggests that a minimum flux of Hg is required for Te to bond with Hg and incorporate as Hg-Te into the layer. By reducing the Hg flux to just below this level, it may be possible to reduce the incorporation of Hg into the passivation layers (which are presently $\text{Hg}_{(0.1)}\text{Cd}_{(0.9)}\text{Te}$ when grown under a high Hg flux), while still providing enough Hg flux to prevent decomposition of the HgCdTe layer.

We have observed an initial decay in the strength of the REMS signals at the start of growth, which we attribute to roughening of the surface as polycrystalline HgCdTe covers the molybdenum or silicon carrier substrate. For full sized substrates this decay may not be so apparent. For smaller sized substrates a carrier wafer or molybdenum block with consistent roughness and surface properties should be used so that the REMS signals are consistent from run to run. Another approach would be to aperture the QMS so that only fluxes arising from the substrate itself are measured, but this would further reduce the intensity of the REMS signals. Further optimisation of the QMS tuning parameters may be able to improve the sensitivity of the technique.

A correlation has been shown between the ratio of reflected Cd/(Cd+Te) fluxes and the resulting x of the grown layer. This offers the possibility for implementing real-time control of layer mole-fraction by controlling the effusion cell temperatures via feedback from the REMS

measurements. We have demonstrated control over the mole-fraction of layers using this technique to correct Te cell temperatures. The SIMS depth profile of a two layer structure shows good agreement with the REMS ratio captured in real-time, which demonstrates the potential of this technique for controlling MBE growth of HgCdTe heterostructures.

VI. ACKNOWLEDGEMENTS

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Fig. 1: Average Cd REMS current vs. average Hg REMS current, with constant incident Te_2 and Cd fluxes (CdTe cell at 519°C Te cell closed). The target is a molybdenum block coated with $\approx 20\mu\text{m}$ of polycrystalline HgCdTe from previous growths.

Fig. 2: Average Te REMS current vs. average Hg REMS current with a constant incident Te_2 flux (Te cell at 320°C CdTe cell closed). The target is a molybdenum block coated with $\approx 20\mu\text{m}$ of polycrystalline HgCdTe from previous growths.

Fig. 3: Average reflected Cd REMS current vs. average Te REMS current for several CdTe cell temperatures, with a constant Hg flux. The temperature of the Te cell was varied from 260°C to 335°C for each series. The target is a silicon wafer, which was being coated with polycrystalline HgCdTe during these measurements.

Fig. 4: REMS profiles for Cd and Te vs. growth time, showing a sharp increase in the Cd REMS current when the Te cell is closed, and decay in the REMS signals as the target is coated.

Fig. 5: Average REMS $\text{Cd}/(\text{Cd}+\text{Te})$ ratio plotted against mole-fraction x as determined from a fit to room temperature FTIR measurements. Data is shown for $10\text{mm}\times 10\text{mm}$ CdZnTe wafers mounted directly on a molybdenum block and also for CdZnTe wafers mounted on 2 inch diameter silicon wafers. The numbers in brackets indicate the average Te cell temperature used for growth of each wafer.

Fig. 6: SIMS depth profile of mole-fraction x vs. sputter time, compared with REMS $\text{Cd}/(\text{Cd}+\text{Te})$ ratio during layer growth.

Figures

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FIG. 2: Average Te REMS current vs. average Hg REMS current with a constant incident Te_2 flux (Te cell at 320°C CdTe cell closed). The target is a molyblock coated with $\approx 20\mu\text{m}$ of polycrystalline HgCdTe from previous growths.

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FIG. 4: REMS profiles for Cd and Te vs. growth time, showing a sharp increase in the Cd REMS current when the Te cell is closed, and decay in the REMS signals as the target is coated.

FIG. 5: Average REMS Cd/(Cd+Te) ratio plotted against mole-fraction x as determined from a fit to room temperature FTIR measurements. Data is shown for 10mx10mm CdZnTe wafers mounted directly on a molybdenum block and also for CdZnTe wafers mounted on 2 inch diameter silicon wafers. The numbers in brackets indicate the average Te cell temperature used for growth of each wafer.

FIG. 6: SIMS depth profile of mole-fraction x vs. sputter time, compared with REMS Cd/Cd+Te ratio during layer growth.