THE SOURCES AND BEHAVIOUR OF IMPURITIES IN LPE-GROWN (Cd,Hg)Te LAYERS ON CdTe(111) SUBSTRATES

M.G. ASTLES, H. HILL, G. BLACKMORE, S. COURTNEY and N. SHAW

Royal Signals and Radar Establishment, St. Andrews Road, Great Malvern, Worcs. WR14 3PS, UK

The sources of background impurities in LPE layers of (Cd,Hg)Te grown on CdTe(111) substrates are investigated using Spark-source mass spectrometry (SSMS) and secondary-ion mass spectrometry (SIMS). The principal sources of impurities were (i) the compounds HgTe and CdTe used in the LPE solution synthesis, (ii) the graphite sliding boat system and (iii) the CdTe substrate. The main impurities and their typical levels in the LPE layers were found to be Li (0.2 ppma), Na (0.3–1.0 ppma), Si (0.1–0.3 ppma), Cl (0.3–0.4 ppma) and K (0.03–0.06 ppma). To reduce the levels various changes in procedure were adopted such as cleaning of the graphite boat parts in aqua regia, the use of elemental starting materials (Hg, Cd, Te), substrate purification and the use of in-situ wash melts. The effect of Hg saturated isothermal annealing on the impurity concentrations and distribution has been studied, revealing rapid surface segregation of the Group IA impurities. The values of the distribution coefficients for several impurities have been calculated from the analytical results, these values being generally in good agreement with other published work.

1. Introduction

While much work has been reported on the LPE growth of (Cd,Hg)Te alloys from Te-rich [1–4] and Hg-rich solutions [5,6], this has mainly concentrated on such factors as surface topography, compositional control and uniformity and electrical results before and after annealing treatments, while little has been reported on the nature or the sources of background impurities in typical LPE layers. This is important as the photoconductive and photovoltaic IR detectors made from this material require extremely low levels of electrically active impurities in the low 10^{14} cm^{-3} (< 0.01 ppma) range.

In this paper we report chemical analysis work to investigate the levels and sources of background impurities in LPE layers of (Cd,Hg)Te grown on CdTe (111)B substrates from Te-rich solutions by the horizontal graphite sliding boat system. The sources of impurities have been identified and various remedial actions are described to reduce the impurity levels. While the exact impurity levels found in this work are specific to the particular growth procedure used, the results

are likely to be applicable to other laboratories' growth methods. The behaviour of impurities during Hg-rich isothermal annealing of the LPE layers has also been investigated using SIMS profiling.

2. Liquid-phase epitaxy

The LPE layers were grown from Te-rich solutions at 460°C in a graphite sliding boat system operating at atmospheric pressure in an open flow system with separate Hg pressure control. The apparatus is shown schematically in fig. 1. The sliding boat is heated by one furnace, and a graphite capsule containing the liquid mercury source by a separate furnace. The temperatures of the two furnaces were carefully linked throughout the growth cycle to ensure that the Hg partial pressure in the growth system was maintained at the correct value according to the published Hg vapour pressure data for Te-rich solutions [7]. This is extremely important to prevent loss of mercury from the growth solution which could affect the liquidus temperature (T_L) and also the composition of the grown layer. The whole growth system
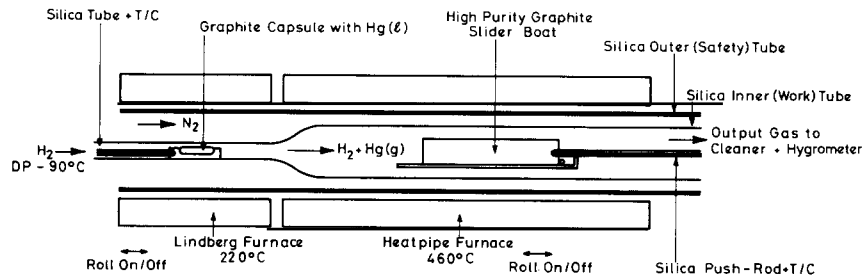
Fig. 1. Liquid-phase epitaxy system for (Cd,Hg)Te growth.

is computer-controlled, with the mercury source temperature being kept within $0.2°C$ of its target value. The substrates were (111)B CdTe polished by diamond and $Syton/H_2O_2$ which produced a flat surface with minimal ($< 1 \mu m$) surface damage [8]. A short etch with Br_2–methanol solution was used immediately prior to loading into the LPE boat. The growth solutions were pre-synthesised in a sealed "Spectrosil" synthetic silica ampoule in a rocking furnace at $600°C$ using either (i) Te (Cominco QZR grade), CdTe and HgTe or (ii) Te (QZR), Cd (Cominco QZR) and Hg (Cominco 99.99999%). After mixing, the solution was quenched a broken into pieces for use in the LPE growth runs.

The temperature–time cycle used to grow the layers is shown diagramatically in fig. 2, consisting

of a dissolution phase at $\sim 20°C$ above T_L, followed by a stabilisation phase at T_L for ~ 1 h. A temperature ramp of typically $0.5-1.0°C$ min^{-1} was then initiated and at a temperature of $T_L - 2°C$, the solution was moved onto the substrate. Typical growth times were 10–15 min to minimise interdiffusion effects at the interface. Wipe-off of the Te solution at the end was generally 98%, the slider movement being controlled by a stepper motor.

3. Chemical analysis

The bulk materials (Te, CdTe, HgTe, graphite, etc.) were analysed by spark-source mass spectrometry (SSMS) using an AEI MS702 instrument
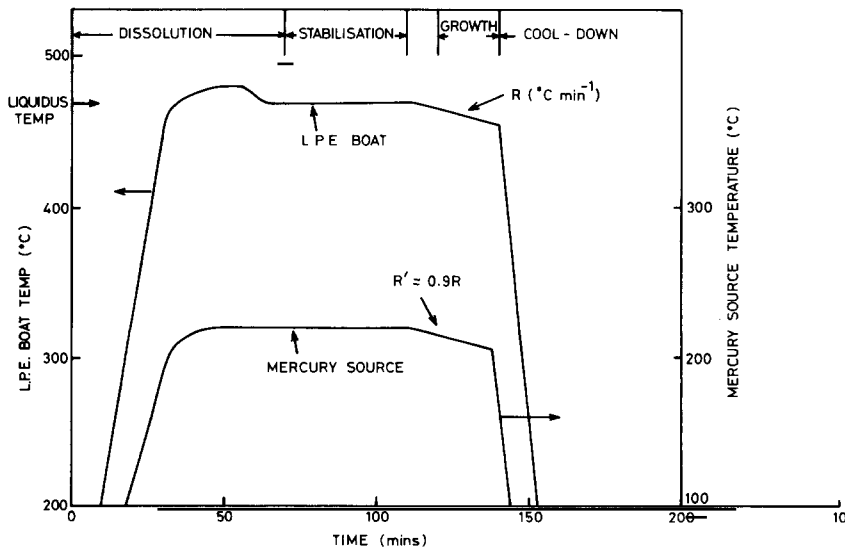

Fig. 2. Temperature–time cycle for LPE boat and mercury source.

which was capable of detecting most elements down to concentrations of 1 part in 10^9. The sample was ionised under a vacuum of 10^{-8} Torr by a 40kV RF spark. The ions were focussed onto a photographic plate by a sequence of electrostatic and magnetic analysers in the mass spectrum range of 5–250 AMU. Liquid helium cryogenic pumping of the source was used to reduce the level of residual gases in the system. Impurity concentrations were obtained by microdensitometer measurements on the plate. The elements were assumed to have equal sensitivities except for Na and K which had normalising factors of 0.1. The results of this method of mass spectrometry have been shown to be accurate to within a factor of 3 of their true values [9].

The epitaxial (Cd,Hg)Te layers were depth-profiled by SIMS using a Cameca IMS3F machine with 12.5 kV O_2 primary ions [10]. The beam was focussed to 10–20 μm diameter and rastered over an area 500×500 μm giving an erosion rate for (Cd,Hg)Te of ~ 4 μm h^{-1}. Only secondary ions from the central 60μm diameter of the rastered area were mass analysed to avoid edge effects near the crater walls. Quantitative analyses were obtained by calibrating the SIMS instrument with doped standards of bulk (Cd,Hg)Te which were analysed by SSMS or atomic absorption spectrometry.

4. Annealing

Annealing of grown layers was carried out in sealed silica ampoules containing liquid mercury, the ampoules being sealed off at a vacuum of 10^{-6} mbar. Prior to annealing, the LPE samples were etched in 2% bromine–methanol solution to remove ~ 2 μm from the surface. This annealing process is commonly used to remove the mercury vacancies believed to be present in (Cd,Hg)Te grown under Te-rich conditions. Normal anneal conditions for 10–15 μm thick LPE layers were 250°C for 48 h.

Because of the variable impurity concentrations in commercially available CdTe substrates and the potential effect of these impurities on the epitaxial layers, the purification of CdTe has been investi-

gated using a high temperature Cd-rich anneal at 800–880°C for 48 h in a sealed silica ampoule. After this anneal treatment, the specimens were analysed by SIMS depth profiling to study the surface segregation of impurities.

5. Results

5.1. Analysis results

Fig. 3 shows the results of the chemical analyses made by SSMS and SIMS at the various stages of the LPE process. In the original starting materials, the major sources of impurities are the compounds HgTe and CdTe used for the solution synthesis, of which the HgTe (containing Na, Al, Si, Cl and K) is the most significant as it is a high proportion of the charge mixture. Levels of C and O were found to be high in all the starting materials but this was
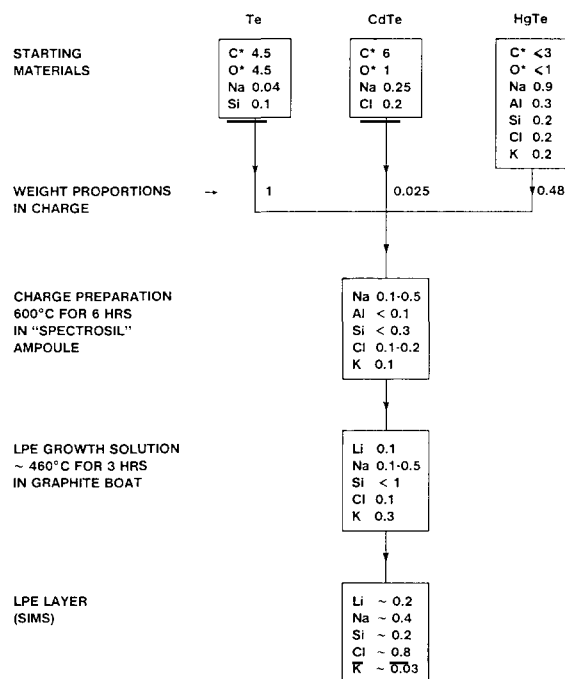

Fig. 3. Typical analysis results obtained by Spark-source mass spectrometry (SSMS) and secondary-ion mass spectrometry (SIMS) at various stages of the LPE growth process. * Surface contamination. All figures in parts per million atomic (ppma): 1 ppma $\equiv 3 \times 10^{16}$ cm^{-3} in CMT.

thought to be surface contamination. The analysis results for the mixed charge are consistent with what would be expected from the starting materials mixed in the proportions shown except for a slight increase in Na and Cl which is presumably coming from the ampoule used for the charge mixing. After the solution has been used for LPE growth, the major feature in the analysis is the appearance of Li at a level of ~ 0.2 ppma and an increased amount of K. The most likely source of these is the graphite boat in which the LPE growth is carried out. When the LPE layer is analysed by SIMS, the dominant impurities found are the same five elements Li, Na, Si, Cl and K. Thus it can be seen that there is a consistent picture which gives confidence in the analytical results.

As a result of this work, it was decided to try to remove the impurities in (i) the graphite boat and (ii) the HgTe, CdTe starting materials. To investigate the graphite, samples of the material as supplied and after machining were analysed by spark-source mass spectrometry. The results are shown in table 1 and indicate that after machining the levels of all the detected impurities have risen sharply. The group IA elements probably originate from the general environment of the machine shop, while the transition metals V, Fe, Ni, Cu probably come from the steel tools used in the machining process. The high level of Cl in the as-received material is probably due to the halogen baking process used by the graphite manufacturers as a final purification step. Obviously, the extent to which these impurities contaminate the Te growth solution will depend on (i) the efficiency of the cleaning procedure employed on the graphite

Table 1
Analysis of graphite by SSMS (raw data) in ppma

	Before machining	After machining
Li	< 0.1	0.5
Na	0.3	5
Al	2	3
Cl	60	130
K	0.1	1
V	< 0.03	10
Fe	1.5	15
Ni	< 0.07	0.7
Cu	< 0.15	0.35

Table 2
Analyses of Te by SSMS (in ppma)

	Melted in graphite given a bake in H_2	Melted in graphite given a chemical clean
Li	0.1	0.02
Na	1	1 [a]
Al	0.05	0.02
Si	1	0.5
Cl	0.07	0.05
K	0.1	0.02

[a] Surface contamination.

components before they are used and (ii) the solubility of the impurity species in the Te solution. The cleaning procedure initially used for the graphite components was a surface degreasing with methanol followed by a high temperature bake at 800°C in H_2 for 48 h. This was obviously not completely effective and hence a new method was used consisting of a chemical treatment in aquaregia for 12 h, followed by extensive cleaning in de-ionised water until conductivity measurements on the water showed no ionic contamination. The graphite was then carefully baked to dryness in H_2. To test the effectiveness of this procedure, samples of high purity Te were placed in graphite components (i) which had been cleaned by the original technique and (ii) which had been given the chemical cleaning process. The Te was melted and held at ~ 470°C for 2 h to simulate the pre-growth stages of the LPE process. The samples of Te were then analysed by SSMS and the results are shown in table 2. A reduction in the elements of most concern (Li and K) by a factor of ~ 5 or more was found. The result for Na was not clear cut, due to surface contamination on the specimen.

Table 3
Analyses of LPE charges by SSMS (in ppma)

	Using Te + HgTe + CdTe	Using Te + Hg + Cd
Li	0.003	0.004
Na	0.9	0.5
Si	0.15	0.04
Cl	0.45	0.2
K	0.1	0.03

The effect of using elemental starting materials for the solution charges was investigated and table 3 shows a comparison of LPE charges (prior to use) made from (i) Te : CdTe : HgTe and (ii) Te : Cd : Hg. In all cases, the impurity concentration was reduced by a factor of 2 to 4, when the elements are used. The levels of Na and Cl are still rather high and as mentioned earlier, it is suspected that contamination with Na and Cl may be coming in during the charge preparation.

5.2. Impurities in LPE layers

During LPE growth, impurities can be introduced into the layer in two main ways (i) by segregation from the grown solution and (ii) from the substrate. Fig. 4a shows typical SIMS depth profiles for Li and Al through an as-grown LPE layer of (Cd,Hg)Te on CdTe. The interface position can be ascertained from the Hg profile. The peaks in the impurity concentrations at the interface are found for several elements. This effect has also been seen in MOVPE grown layers of (Cd,Hg)Te [11]. There are several possible causes of this (i) gettering of impurities to the heterojunction during growth (ii) an instrumental enhancement effect in the SIMS machine due to a change in the erosion rate in the interface region or (iii) surface contamination of the original CdTe substrate either by out-diffusion from the substrate prior to growth or by residual contamination from polishing and etching processes. To have this high concentration of impurities at the interface is wor-

rying since there is the possibility of diffusion of these impurities into the layer during the growth or subsequent annealing treatments. In order to remove this problem we have developed an in-situ wash–melt technique [12] which removes ~ 10 μm depth from the surface of the CdTe substrate immediately prior to the start of growth. This is done by having a well of pure Bi in the sliding boat, which is brought into contact with the substrate for 1 min immediately prior to moving the growth solution onto the substrate. This technique effectively removes the interfacial impurities as can be seen in fig. 4b, and suggests that the cause of the interface peaks is the build up of impurities on the CdTe substrate surface. In order to ascertain the extent to which these impurities affect the electrical properties of the epitaxial layers, two LPE layers of $x = 0.3$ were isothermally annealed at 250°C for 24 h, one grown using the wash–melt technique, the other without it. The solution charge and the substrate source were the same in both cases. The electrical results at 77 K after annealing as measured by the Hall effect were: standard sample $n = 1.7 \times 10^{15}$ cm^{-3}, $\mu = 4.0 \times 10^4$ cm^2 V^{-1} s^{-1}; wash–melt sample $n = 1.5 \times 10^{14}$ cm^{-3} cm^{-3}, $\mu = 5.8 \times 10^4$ cm^2 V^{-1} s^{-1}. This improvement in the electrical properties suggests that the interfacial impurities are affecting the electrical properties either by the diffusion of those impurities into the layer as it grows or by producing a high n-type region near the interface which is being sampled in the Hall measurement.
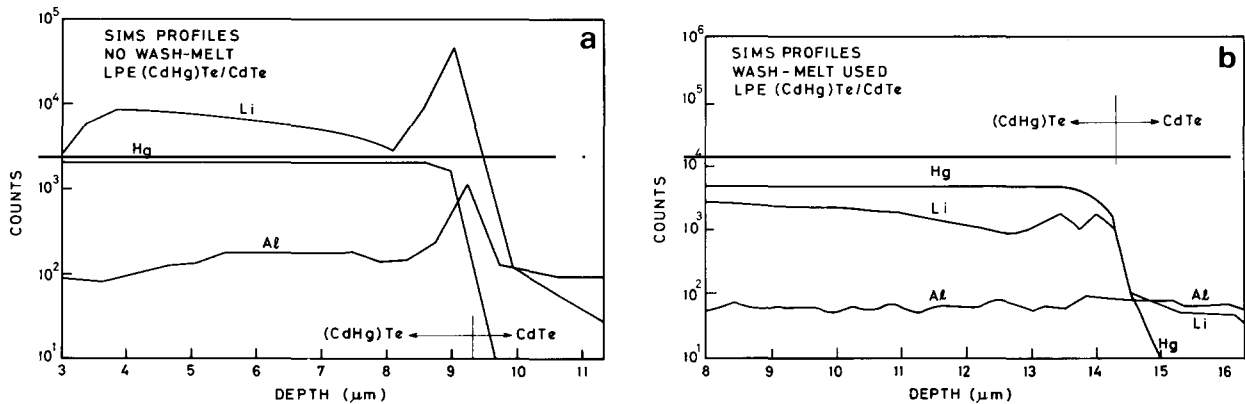

Fig. 4. SIMS profiles for Li and Al through (CdHg)Te (layer)/CdTe (substrate) structure (a) without and (b) with the use of an in-situ wash melt during the LPE growth process.
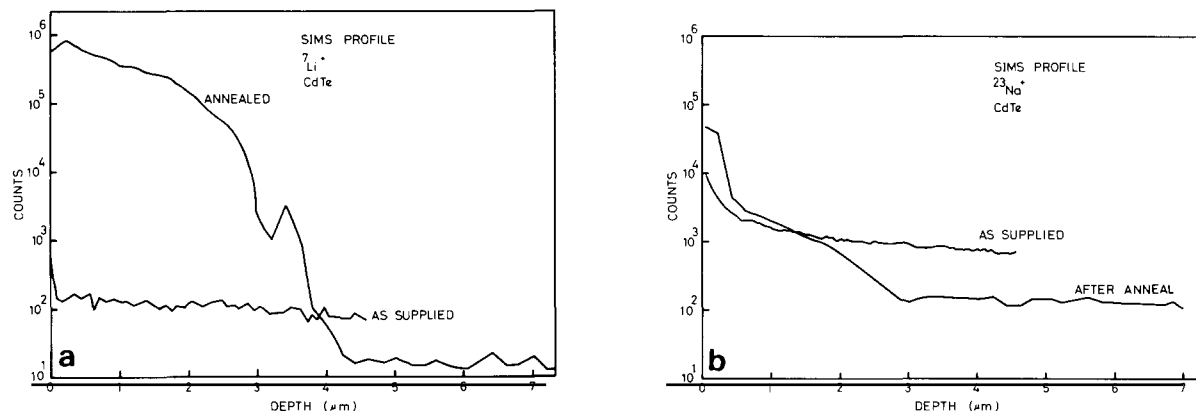
Fig. 5. SIMS profiles in near surface region of CdTe substrate material before and after heat-treatment for (a) lithium and (b) sodium.

5.3. Substrate purification

The effect of the high temperature Cd-rich annealing of CdTe substrate material was studied by doing near-surface SIMS profiling before and after the heat-treatment. Fig. 5 shows the results for Li and Na, and indicates that during the heat treatment significant surface segregation of impurities has occurred with the levels in the bulk of the material being reduced. The results for all the detected impurities are shown in table 4. Apart from the level for Ga, all the impurity concentrations have been reduced significantly by the heat treatment, although the result for Cu is not definitive due to the high detectivity limit. The use of SIMS in the imaging mode has shown that several of the impurities showing this surface segregation effect are present at the surface in particulate form, although the exact chemical nature of these

Table 4
Analysis of CdTe substrate material by SIMS (in ppma)

	As supplied	After heat treatment at 800 °C (48 h)
Li	0.01	0.001
Na	0.1–0.2	0.03
K	0.2–0.3	0.07
Al	0.2–0.3	0.08
Si	1	0.2
Cu	⩽ 0.5	⩽ 0.7
Cr	0.002	0.001
Ga	0.03	0.06

has not been established. Work is currently underway to assess the effect of this reduction in impurities on epitaxial layers grown on such purified substrates.

5.4. Effect of isothermal annealing

The effect of Hg-rich annealing on the background impurities in the LPE layers was investigated using SIMS profiling before and after the anneal treatment of 250°C for 48 h. The results for Li and Na are shown in fig. 6 and it can be seen that there is a drastic reduction in the levels of the impurities in the bulk of the layer with a build up towards the surface. The concentrations of Li and Na in the layers after isothermal annealing are $\sim 5 \times 10^{12}$ cm^{-3} and $\sim 10^{14}$ cm^{-3}, respectively. In some layers, the Li concentration in the as-grown layer was constant through the layer thickness. In the case of the layer shown in fig. 6, it can be seen that there is a dip in the Li profile which probably indicates that some Li has begun to be lost to the surface during the cool-down process from the growth temperature to room temperature. Similar, though less dramatic, effects occur for other impurities such as Si and K. No movement of Al or Cl has been seen during isothermal annealing.

It thus appears that during type-conversion of as-grown p-type layers to n-type by Hg-rich annealing, a process of purification also occurs. Fortuitously, the purification effect is most dramatic
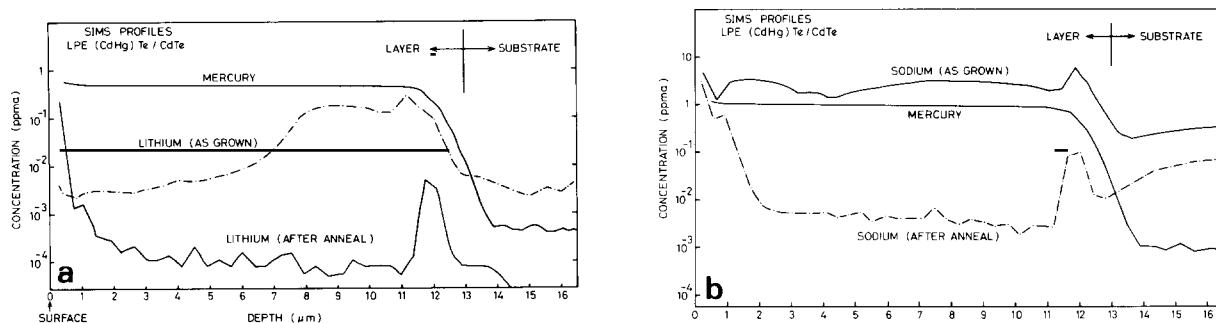
Fig. 6. SIMS profiles through (Cd,Hg)Te (layer)/CdTe (substrate) structure before and after isothermal Hg-rich annealing at 250°C for 48 h in the case of (a) lithium (b) sodium.

for the Group IA elements which are persistent impurities in the (Cd,Hg)Te layers.

6. Discussion and conclusions

This work has shown a consistent pattern of impurities in the LPE growth process for (Cd,Hg)Te. Five particular species have been found to be the dominant background impurities, namely Li, Na, Si, Cl and K. Table 5 lists these impurities, the range of concentrations typically found in our LPE layers and the sources of the impurities as identified in this work. Of these five, Li and Na were effectively removed by an isothermal Hg-rich annual treatment leaving Si, Cl and K as the major impurities. The electrical effects of

Table 5
Dominant background impurities in as-grown LPE grown (Cd,Hg)Te layers

Impurity	Typical concentrations in LPE layers (ppma) [a]	Sources of impurities
Li	0.1–0.4 [b]	Graphite boat
Na	0.3–1.0 [b]	Compound sources + charge preparation
Si	0.1–0.3	Compound sources
Cl	0.3–0.4	Compound sources + charge preparation
K	0.03–0.06	Compound sources + graphite boat

[a] 1 ppma $\equiv 3 \times 10^{16}$ cm^{-3}
[b] Drastically reduced after isothermal anneal

these impurities in Te-rich LPE has not been established, although Si and Cl have been found to be active as donors in bulk (Cd,Hg)Te [13]. One potentially important impurity that has not been discussed here is Cu. This is commonly found in CdTe substrate material and is known to be a very fast diffuser in CdTe and (CdHg)Te [14]. In the SSMS And SIMS analysis techniques used in this work, there is an interference on the ^{63}Cu$^+$ and ^{65}Cu$^+$ isotope lines from the double charged matrix species ^{126}Te^{2+} and ^{30}Te^{2+}. In the SIMS analyses, background levels of ~1 ppma are found. However, recent work as RSRE has produced a technique for Cu determination by SIMS in (Cd,Hg)Te down to 3 ppba (~1 × 10^{14} cm^{-3}) level [15]. Analysis of an LPE layer with this technique has given a value of ~10 ppba (3 × 10^{14} cm^{-3}) suggesting that Cu is not a serious contaminant.

From the analytical work reported here and the results of doping experiments reported elsewhere [16], values of the distribution coefficient for several impurities have been calculated. These are listed in table 6 together with values from previous work [17–20]. The values of k for Li and Na do not agree well with the values of Brice et al. [17]. However, in the latter work, the LPE growth was carried out at 500°C and it is quite likely that during the cool-down to room temperature after growth that any Li or Na in the layers would be rapidly lost to the surface, thus giving a low apparent value of k. For the other impurities, the agreement is reasonable. The high values of k for Li, Na and Cl mean that these are the most important to avoid in the growth process.

Table 6
Distribution coefficients (k) in Te-rich LPE growth of (Cd,Hg)Te

Element	k (this work)	k (previous workers)	
Li	2	0.1–0.3	[17]
Na	1	0.01–0.03	[17]
Al	0.3	0.1–0.3	[17]
Si	0.1	0.03–0.1	[17]
Cl	8	–	
K	0.1	0.03–0.1	[17]
Cr	0.05	0.01–0.03	[17]
In	0.9	< 0.01	[17]
		2.8	[18]
Sb	$\leqslant 1 \times 10^{-4}$	10^{-3}	[19]
		$0.3–1.0 \times 10^{-4}$	[20]

The surface segregation of Li and Na seen in this work has been seen by previous workers in the case of Li in ZnTe [21], ZnSe [22], and (Cd,Hg)Te [23]. The work of Bubulac et al. [23] looked at the distribution of Li in LPE grown (Cd,Hg)Te before and after annealing as was done here. In that work, a short high temperature Hg-rich anneal (400°C, 3 h) which reduced the acceptor concentration caused the Li level in the bulk of the LPE layer to layer to fall and the Li to concentrate in a surface region of 3–6 μm depth which is much larger than in this work. When a low temperature isothermal anneal was used (250°C, 24 h) a redistribution similar to that seen in this work was found whereby the Li was redistributed into a narrow near-surface region. The explanation for this was that the Li was being gettered into a surface region which contained dislocations and Te precipitates either in the as-grown condition or during the high-temperature anneal. In the present work, the surface region of the as-grown layers was removed before annealing. However it is not known if the Hg-rich anneal treatment introduced surface defects into the layers which could act as gettering sites. Although Bubulac et al deduce from their results that the Li is attracted to native acceptor sites, it would be expected that as an as-grown LPE layer containing Hg vacancy acceptors is converted to n-type by the in-diffusion of Hg interstitials then Li would be driven deeper into the layer. Some other mechanism is required to explain why the Li dif-

fuses so rapidly "uphill" to the surface. The behaviour of Li has been studied in several II–VI compounds to ascertain its lattice location and the types of complexes it forms with native defects and other impurities. In CdTe diffused with Li, Ko and Spitzer [24] have obtained strong evidence from localized vibrational mode infra-red absorption measurements for the present of $Li_{Cd}-Li_i$ pairs. At long diffusion times, evidence for a second phase, possibly Li_2Te precipitates, was found. In CdTe containing the Group III impurity Al, Zielinska et al. [25] have found evidence for a complex consisting of $(Al_{Cd}-V_{Cd}-Li_i)$ which at long diffusion times form $(Al_{Cd}-Li_{Cd})$ pairs. In the case of (CdHg)Te, it is likely that similar behaviour of Li is found, e.g. $(Li_{(Hg/Cd)}-Li_i)$. If such complexes are broken up by the in-diffusion of Hg during isothermal annealing [23], Li_i would be released which is not a stable defect even at room temperature [25] and would rapidly diffuse at the anneal temperature. The question remains as to why Li appears to diffuse towards the layer surface rather than towards the interface. One explanation is that the Li-interstitial probably has a low ionisation energy and once ionised would be small and able to move rapidly under the influence of internal electric fields. There are several possible sources of such a field near the (Cd,Hg)Te surface. For example, the Dember effect [26] has been postulated by Tregilgas and Gnade [27] to explain the surface segragation of Ag in (Cd,Hg)Te at room temperature. This entails the absorption of band-gap radiation producing electron–hole pairs. Due to the higher mobility of electrons compared to holes in (Cd,Hg)Te, the electrons move further away from the surface, leaving the near surface region with a net positive charge which establishes the so-called Dember field. Charged mobile species such as Li interstitials can migrate in this field to the surface where they may be lost from the sample or agglomerate into precipitates as seen in our SIMS imaging work.

7. Summary

We have found a consistent picture of background impurities in the LPE growth process for

(Cd,Hg)Te. Five dominant impurities are found, namely Li, Na, Si, Cl and K and the sources of these have been identified. Of these, Li and Na are effectively removed by the Hg-rich isothermal anneal treatment used for type converting the as-grown p-type layers. The other three impurities are not affected by the anneal process, and of these Si and Cl are known to be active as donors in bulk (Cd,Hg)Te, although their electrical effects in Te-rich LPE are unknown. A chemical treatment for graphite is shown to be effective in removing impurities left in the graphite by machining, and the use of elemental starting materials instead of the HgTe and CdTe components for the solution synthesis has been found to give purer solutions.

High temperature heat treatment of CdTe substrates has been found to be effective at removing impurities from this material. This is extremely important as the quality of commercial CdTe is very variable and it is vital to remove impurities which might diffuse into the epitaxial layers. An in-situ wash melt technique has been developed which effectively removed the interfacial impurities found at (Cd,Hg)Te/CdTe (substrate) interfaces by etching off ~ 10 μm from the substrate surface. This removes a potential source of impurities which could diffuse into the layer during growth or annealing and also could produce a high n-type region near the interface. Hall effect measurements on annealed layers suggest that this wash-melt technique dramatically improves the electrical properties of the layers.

The distribution coefficients of several impurities in Te-rich LPE have been calculated from the analytical results of this work. The agreement with the values of previous workers is quite good except for Li and Na. This discrepancy may be due to the rapid loss of Li and Na from the (Cd,Hg)Te layers in earlier work giving erroneously low values of k. The values of k are particularly high for Li(2), Na(1) and Cl(8) which makes these elements the most critical to remove in the growth process.

The likely mechanism for the surface segregation of Li and Na in (Cd,Hg)Te during Hg-rich isothermal annealing has been discussed. From published results on the behaviour of Li in other II–VI compounds, the most likely process is the

release of Li-interstitials from complexes such as $(Li_{Hg}-Li_i)$ or $(V_{Hg}-Li_i)$ during isothermal annealing with the highly mobile Li ions migrating to the surface in an electric field generated by, for example, the Dember effect or by the presence of a thin surface oxide.

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