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Vibrational modes of carbon acceptors and hydrogen-carbon pairs in semi-insulating InP doped using CCl_4

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Infrared absorption measurements show that high-resistivity InP grown by metal-organic vapor phase epitaxy (MOVPE) at 500 °C and doped using CCl_4 contains comparably high concentrations ($\sim 3\times 10^{18}$ cm^{-3}) of isolated C_P acceptors and H-C_P pairs. These centers give rise to localized vibrational modes at 546.9 cm^{-1} (C_P) and at 2703.3, 521.1, and 413.5 cm^{-1} (H-C_P). A sharp weak line, also at 546.9 cm^{-1}, may be due to a much lower concentration of C_{In} donors. It is concluded that the doping introduces C_P acceptors that overcompensate grown-in donors and complete compensation results from the formation of H-C_P pairs. [S0163-1829(98)01648-8]

I. INTRODUCTION

Gardner *et al.*[1,2] have reported that semi-insulating InP may surprisingly be grown by metal-organic vapor-phase epitaxy (MOVPE) at low temperatures (460–500 °C) by using CCl_4 as a dopant source. Such high-resistivity material has potential applications in optoelectronic and high-speed electronic devices and its use as a buffer layer in $Ga_{1-x}In_xAs$/InP high-electron mobility transistors (HEMT's) has been demonstrated.[3] The reason why the material has a high resistivity, however, is not clear. Secondary ion mass spectrometry (SIMS) measurements showed that this material contained high concentrations of carbon and hydrogen in the range 10^{18}–10^{20} cm^{-3}, together with chlorine with a concentration at least one or two orders of magnitude lower.[1,2] The higher and lower concentrations refer to growth temperatures of 400 and 500 °C, respectively. It is generally assumed that carbon is incorporated in InP on the group-III site as a donor and this assignment has been supported by local-density-functional calculations.[4] However, spectroscopic evidence for these centers is limited to the observation of a Raman scattering line at 220 cm^{-1} that was attributed to a gap mode of C_{In}.[5] This measurement related to *n*-type InP grown by metal-organic molecular-beam epitaxy (MOMBE) at temperatures up to 430 °C and doped with carbon from the tertiary-butylphosphine precursor.[6] The assignment of the gap mode is not definitive, since an expected localized vibrational mode (LVM) for C_{In} was not detected.

We now present infrared LVM absorption measurements on high-resistivity InP grown by MOVPE and doped using CCl_4. We shall demonstrate that the majority of the carbon impurities occupy P-lattice sites as acceptors. About 50% of these centers are present as H-C_P pairs with LVM's that are analogous to those of H-C_{As} pairs in GaAs (Ref. 7) and other III-V compounds. We conclude that donors are also present in the material and discuss possible candidates for their identity.

II. EXPERIMENTAL DETAILS

InP was grown in a horizontal Metals Research MR350 reactor by low-pressure MOVPE on semi-insulating iron-doped (100) InP substrates orientated 0.35° to (110), using the precursors phosphine (PH_3) and trimethylindium [$In(CH_3)_3$] with a V:III ratio in the gas phase of approximately 100:1. The epilayers were doped using carbon tetrachloride (CCl_4) diluted to 500 ppm (parts per million) in a hydrogen carrier gas, with flow rates of 120, 225, or 450 sccm (standard cubic centimeter per minute). A substrate temperature of 500 °C was used, since the incorporation of chlorine into the epilayers at this temperature is expected to be negligible.[2] Another layer was grown under the same conditions but with no flow of CCl_4. The growth rate was in the range 4–5 μm/h.

SIMS profiling was carried out on a Cameca IMS 3f using a 10 keV cesium primary ion beam. Negative secondary ions of ^{1}H, ^{12}C, ^{16}O, ^{35}Cl, and ^{115}In were monitored and impurity concentrations (see Table I) were obtained by comparison with ion-implanted standards. The depth of the pit was measured with a Tencor alpha-step 200 surface profilometer and the epilayer thicknesses were obtained from the SIMS profiles. The concentration of chlorine (background level 2×10^{16} cm^{-3}) never exceeded 2×10^{17} cm^{-3} for any sample. The oxygen concentration never exceeded the background level of $\sim 1\times 10^{17}$ cm^{-3}, except for a spike at the epilayer/substrate interface.

High-resolution x-ray-diffraction (HRXD) measurements were carried out using a Philips triple-axis diffractometer with a 4-reflection Ge 220 monochromator and a 2-reflection Ge 220 analyzer with an instrumental resolution of 11 arc sec. The 400 ω-2θ scans of all the samples, with one exception, showed an InP epilayer peak with a full width at half maximum (FWHM) of 11 arc sec, which was indistinguishable from the peak from an epiready substrate. However, sample MR949 with the most heavily doped epilayer showed two overlapping peaks. The peak at lower angle was the

TABLE I. Sample details and estimates of the concentrations of C_P acceptors and H-C_P pairs obtained from the IA's of the LVM's at 546.9 and 2703.3 cm^{-1}, respectively.

Sample	CCl_4 flow (sccm)	Thickness (μm)	$[C]_{SIMS}$ (cm^{-3})	$[H]_{SIMS}$ (cm^{-3})	$[C]_{LVM}$ (cm^{-3})[a]	$[H\text{-}C_P]_{LVM}$ (cm^{-3})
MR919	0	2.8	5.7×10^{17}	2.1×10^{18}	5×10^{16}[b]	$<10^{16}$
MR923	225	4.5	2.9×10^{18}	4.1×10^{18}	1.8×10^{18}	1.5×10^{18}
MR950	120	10.3	4.2×10^{18}	5.0×10^{18}	2.3×10^{18}	1.4×10^{18}
MR949	475	8.0	1.0×10^{19}	1.9×10^{19}	5.4×10^{18}	4.6×10^{18}

[a] These data are from the total IA of the absorption feature at 546.9 cm^{-1} and include both the sharp and broad components.

[b] The line for sample MR919 consists of a sharp component only.

weaker of the two and was attributed to the substrate, since the epilayer thickness was comparable to the extinction depth. This HRXD profile was simulated using dynamical diffraction theory, assuming that the strain ($\Delta a/a_0 = -1.6\times10^{-4}$) was induced by carbon atoms present on either the P or the In lattice sites. A good fit was obtained for an epilayer with a thickness of 9 μm and $[C_P]=1.1\times10^{19}$ cm^{-3}, assuming Vegard's law to be valid and using a lattice constant for InC of 5.104 Å (calculated from tabulated tetrahedral covalent radii[8]). Alternatively, an equally good fit was obtained by using $[C_{In}]=5.7\times10^{18}$ cm^{-3} with a calculated lattice constant for CP of 4.318 Å. We note that the carbon concentrations for both assignments are comparable with that measured by SIMS (Table I).

Room-temperature Hall measurements were made on sample MR919 using the van der Pauw geometry for the nominally undoped sample, using the epilayer thickness given in Table I. This sample was *n*-type with a Hall electron concentration of 2×10^{17} cm^{-3} and mobility of 2500 $cm^2\,V^{-1}\,s^{-1}$. Due to their high resistivity, Hall measurements could not be made on the samples grown in a flow of CCl_4.

Fourier transform infrared (FTIR) absorption measurements were carried out using a Bruker IFS120HR interferometer operated at resolutions in the range of 0.02 and 0.5 cm^{-1} with the sample held at a temperature of approximately 10 K. The sample substrates were wedged at an angle of ~3% to eliminate interference fringes from the recorded spectra.

III. EXPERIMENTAL RESULTS AND INTERPRETATION

A. Modes of isolated carbon atoms

The IR absorption spectrum for sample MR950 (Fig. 1), within the gap in the two-phonon absorption,[9] reveals previously unreported lines at 546.9 and 521.1 cm^{-1}, together with lines due to B_{In} present in the substrate.[10] An expanded plot of the line at 546.9 cm^{-1} (Fig. 2) shows that it comprises two components, namely a broad line, with a FWHM of 1.3 and 1.0 cm^{-1} for samples MR949 and MR950, respectively, and a much sharper line, with a FWHM of 0.15 cm^{-1}. The ratio of the integrated absorptions (IA's) of these two components is approximately 20:1. We consider the possibility that the two components result from superposed LVM's of two impurity centers. We next demonstrate that the broad line is due to isolated substitutional $^{12}C_P$.

The spectrum with an expanded vertical scale (Fig. 1) also shows a line at 526.8 cm^{-1} with a FWHM of ~1 cm^{-1}, which is the same as that for the broad feature at 546.9 cm^{-1} within the uncertainty of the measurement. The ratio of the IA's of the two lines is 0.8 ± 0.2 % (the average for four samples). An assignment of the broad line to $^{12}C_P$ would imply an isotopic ratio of 0.9 ± 0.2 %, if the weak line is assigned to $^{13}C_P$, since the strength of a line is inversely proportional to the mass of the light impurity atom. This ratio is consistent with the natural abundance of ^{13}C (1.1%). Further information is obtained by consideration of the measured frequencies. If the two modes are due to ^{12}C and ^{13}C, their frequencies, ω_{imp}, should satisfy the relationship

$$\omega_{imp}^2 = k[1/m_{imp} + 1/(\chi M_{NN})], \tag{1}$$

where k is a force constant, m_{imp} is the mass of the impurity, and M_{NN} is the mass of the nearest neighbors (NN) to the impurity. χ is a numerical factor related to the ratio of the local stretch to bend force constants and has a value close to 2 for all known isotopic substitutions, consistent with theoretical analysis.[11] For example, our measured frequencies for $^{10}B_{In}$ and $^{11}B_{In}$ lead to a value for χ of 1.7. We obtain an acceptable value of $\chi=1.5$ if the lines are assigned to C_P ($M_{NN}=M_{In}=114.9$: 95.7% abundance); the presence of 4.3% of ^{113}In was not taken into account, as a frequency shift of only 0.3 cm^{-1} is deduced for $^{12}C_P$ [using Eq. (1)] even if

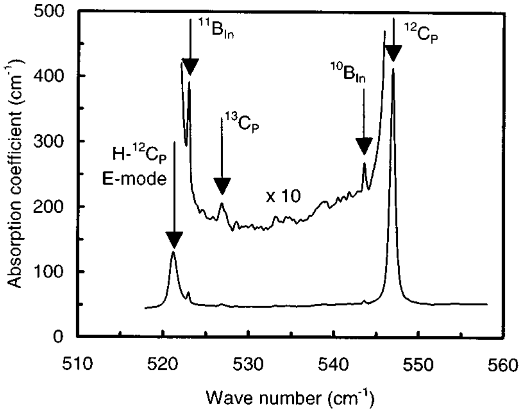

FIG. 1. IR absorption spectrum in the gap in the two-phonon density of states for sample MR950. The upper trace has been expanded by a factor of 10. Lines attributed to $^{12}C_P$, $^{13}C_P$, and H-$^{12}C_P$ defects are indicated. The boron lines are due to impurities in the substrate.

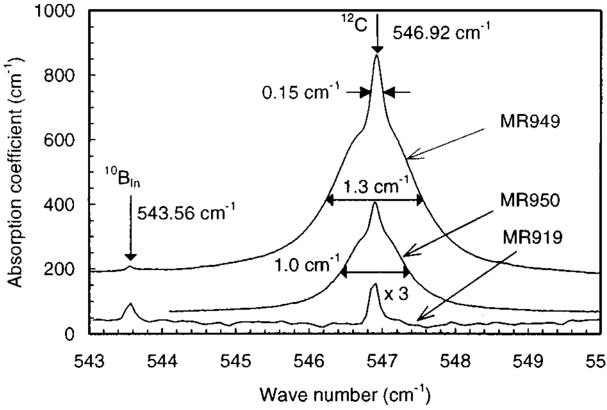

FIG. 2. IR absorption spectrum for samples MR949 and MR950 taken at a resolution of 0.02 cm^{-1} showing that the line at 546.9 cm^{-1} consists of two peaks with linewidths differing by nearly an order of magnitude. The magnified spectrum (resolution 0.1 cm^{-1}) for the nominally undoped sample MR919 also shows a narrow line at 546.9 cm^{-1}. The linewidth of the sharper line is slightly higher than that for the line due to ^{10}B$_{\mathrm{In}}$, from isoelectronic boron impurities present in the InP substrate.

all four indium NN are replaced with ^{113}In. However, an assignment of the two broad lines to C$_{\mathrm{In}}$ ($M_{\mathrm{NN}}=M_{\mathrm{P}}=31$) leads to an unacceptable value of $\chi=5.6$. This assignment must be rejected.[11]

If we assume that the sharp feature at 546.9 cm^{-1} is an LVM of a center other than C$_{\mathrm{P}}$ the mass of the impurity involved must be close to that of carbon. An obvious possibility is an assignment to carbon atoms occupying group-III sites, i.e., ^{12}C$_{\mathrm{In}}$. Assuming a value of $\chi=2$, the predicted frequency [Eq. (1)] for the ^{13}C$_{\mathrm{In}}$ mode is 528.9 cm^{-1}. The spectrum in Fig. 1 reveals a small nearby feature at 528.5 cm^{-1} with a FWHM of ~0.4 cm^{-1} and an intensity close to the background noise level: the same feature has also been observed in the spectra for other samples. Estimated IA's for this feature are close to 1% of that for the sharp line at 546.9 cm^{-1}, again consistent with the ratio expected for modes of naturally occurring carbon. If this interpretation is correct, the experimental value for χ is 2.3, which is still within the acceptable range.[11] In summary, we have shown that there is a possibility that the sharp line at 546.9 cm^{-1} is the ^{12}C$_{\mathrm{In}}$ LVM, consistent with its observation in the spectrum for the *n*-type nominally undoped sample MR919 (Fig. 2). No associated gap mode has been detected but this may be due to the low concentrations of the center.

B. Modes of hydrogen-carbon pairs

We now discuss modes of H-C pairs observed in the present samples. The line at 2703.3 cm^{-1} (Fig. 3) is identified as a stretch mode of a H-^{12}C$_{\mathrm{P}}$ pair by comparison with the modes of H-^{12}C$_{\mathrm{V}}$ in other III-V compounds for which this pair defect has been detected, namely for GaAs,[12] GaP,[13] and AlAs (Ref. 14) (Table II). In addition, a weak absorption feature at 2696.6 cm^{-1}, which can be identified as the stretch mode of H-^{13}C$_{\mathrm{P}}$, was observed with a strength ~1% of the line at 2703.3 cm^{-1}. The isotopic separation of 6.7 cm^{-1} is the same as that measured for the corresponding stretch modes of H-C$_{\mathrm{As}}$ in GaAs and is comparable with those for H-C$_{\mathrm{As}}$ defects in AlAs [6.2 cm^{-1} (Ref. 15)] and H-C$_{\mathrm{P}}$ in GaP (7.6 cm^{-1}). The assignment of the line at 2703.3 cm^{-1} to the stretch mode of H-^{12}C$_{\mathrm{P}}$ pairs in InP implies that the hydrogen atom occupies a bond-centered site with a strong hydrogen-carbon bond. Symmetric A_1^+ modes and transverse E modes of the H-C$_{\mathrm{P}}$ pairs are, therefore, also expected, by analogy with the data available for H-^{12}C$_{\mathrm{As}}$, H-^{13}C$_{\mathrm{As}}$, D-^{12}C$_{\mathrm{As}}$, and D-^{13}C$_{\mathrm{As}}$ pairs in GaAs (Ref. 7) and AlAs.[16] A second line observed in our InP samples at 413.5 cm^{-1} (Fig. 4) has an IA that correlates with the IA for the stretch mode of H-^{12}C$_{\mathrm{P}}$ pairs and is assigned to the symmetric A_1^+ mode of H-^{12}C$_{\mathrm{P}}$: a corresponding line in the spectra for GaP:C epilayers[17] has been attributed to H-^{12}C$_{\mathrm{P}}$ (see Table II). The spectra of the InP samples show a third H-C$_{\mathrm{P}}$ line at 521.1 cm^{-1} (Fig. 1) that can be attributed to a transverse mode of H-^{12}C$_{\mathrm{P}}$ pairs (see Refs. 7 and 16), since its IA also correlates with that of the stretch mode.

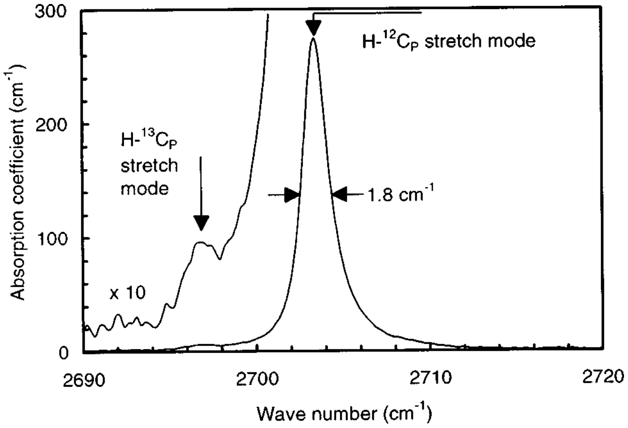

FIG. 3. IR absorption spectrum for sample MR950 taken at a resolution of 0.1 cm^{-1} showing the lines attributed to the stretch modes of H-^{12}C$_{\mathrm{P}}$ (2703.3 cm^{-1}) and H-^{13}C$_{\mathrm{P}}$ (2696.6 cm^{-1}).

Concentrations (cm^{-3}) of isolated or complexed impurities are obtained from the product of a calibration factor f and the IA of the LVM absorption.[18] Values of f for the C$_{\mathrm{P}}$ LVM and the stretch mode of H-C$_{\mathrm{P}}$ in InP have not been determined but values of $f_{\mathrm{C}}=(7\pm1)\times10^{15}$ cm^{-1} (Ref. 19) and $f_{\text{H-C}}=(9\pm2)\times10^{15}$ cm^{-1} (Ref. 20) have been obtained for the corresponding modes in GaAs. Use of these values

TABLE II. Comparison of the frequencies of the LVM's for isolated and hydrogen passivated carbon acceptors, C$_{\mathrm{V}}$ and H-C$_{\mathrm{V}}$, respectively.

Mode	C$_{\mathrm{V}}$ T_d (cm^{-1})	H-C$_{\mathrm{V}}$ A_1^- (cm^{-1})	H-C$_{\mathrm{V}}$ A_1^+ (cm^{-1})	H-C$_{\mathrm{V}}$ E (cm^{-1})
InP	546.9	2703.3[a]	413.5	521.1
GaP	606	2661	477	
(Refs. 17 and 13)		2660.2		
GaAs	582.8	2635.2	452.7	E^+ 562.6
(Refs. 7 and 12)				
AlAs	631.5	2555.4	487.0	E^- 656.6
(Ref. 16)				

[a] The peak position and FWHM of this line increase by 0.3 and 0.7 cm^{-1}, respectively, for the most heavily doped sample (MR949) and the line shape becomes slightly asymmetric.

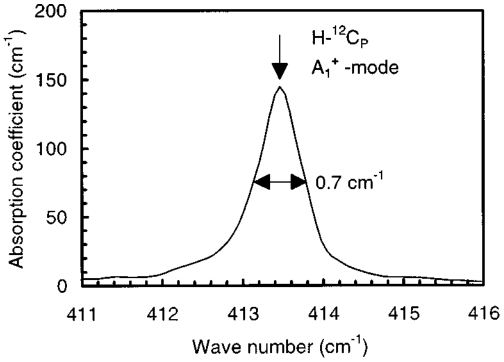

FIG. 4. IR absorption spectrum for sample MR950 taken at a resolution of 0.1 cm^{-1} showing the line attributed to the A_1^+ mode of H-$^{12}C_P$ at 413.5 cm^{-1}.

for InP leads to derived total concentrations of carbon, $[C]_{tot}=[C_P]_{LVM}+[H\text{-}C_P]_{LVM}$ significantly greater than those measured by SIMS, $[C]_{SIMS}$, but this discrepancy is removed to within ~10% by reducing $f_{H\text{-}C}$ to 2×10^{15} cm^{-1} (Table I). This value implies that the effective charge[21] for the H-C_P stretch mode is $\eta\sim2e$, compared with $\eta\sim e$ for H-C_{As} pairs in GaAs. The derived values for $[H\text{-}C_P]_{LVM}$ are smaller than $[H]_{SIMS}$ by factors of ~3.5 but hydrogen may be present in other forms that are not IR active. This is implicit in the results for sample MR919 since no LVM's from H complexes were detected although $[H]_{SIMS}=2.1\times10^{18}$ cm^{-3}. In this analysis the values of the IA relating to $[C_P]$ have been measured for the total absorption profile at 546.9 cm^{-1}: that is, no separate account has been taken of the sharp but weak superposed component. If this component is attributed to the LVM of C_{In} donors, we can deduce an LVM calibration factor $f=2.8\times10^{16}$ cm^{-1} and an effective charge $\eta=1.8e$ from the Hall measurements of the *n*-type sample MR919. These are plausible parameters but it would be implied that $[C_{In}]$ is much smaller than $[C_P]$ by a factor of ~5 for the samples doped using CCl_4. If the two concentrations were comparable, a stronger LVM, together with a gap mode, should have been detected for C_{In}.

IV. DISCUSSION

This LVM study of InP grown by MOVPE at 500 °C and doped using CCl_4 demonstrates that carbon atoms are incorporated on the group-V site. Approximately 50% of these acceptor centers are passivated with hydrogen during growth to form neutral hydrogen-carbon pairs and it is implied that the material must have been *p*-type during growth. Hydrogen atoms, derived from the dissociation of PH_3, could also be incorporated during growth and additional atoms may diffuse in during the cooling period. The latter process has been established for the passivation of Zn acceptors in InP.[22] It is clear that compensating donors or hole traps must also be present in a comparable concentration, to account for the semi-insulating nature of the material. We now consider possible identities for these defects.

It has been reported that the fully hydrogenated indium vacancy, $V_{In}H_4$ acts as a single shallow donor and gives rise to an LVM at 2316 cm^{-1}.[23,24] We have observed this line in the InP substrates used in the present work but no extra absorption was detected after the epilayer growth. We conclude that this is not the center that compensates the unpassivated C_P acceptors.

The strength of the sharp LVM feature at 546.9 cm^{-1} that may be due to C_{In} donors increases as $[C_P]_{LVM}$ increases but it has been shown that the estimated concentrations of these compensating donors are smaller than $[C_P]$ by a factor of ~5 in the doped samples. The possibility that complete compensation results from the presence of grown-in C_{In} donors therefore appears to be untenable for the present samples.

A third possibility is that P_{In} antisite defects are present, as suggested previously.[1,2] These defects have been positively identified, by a range of techniques, in InP grown at low temperatures (less than 350 °C) by gas-source molecular-beam epitaxy, using cracked phosphine and elemental indium.[25,26] They are double donors with energy levels at $E_C+0.12$ eV and at $E_C-0.23$ eV, above and below the conduction-band edge, respectively, and their presence can result in carrier concentrations of up to 2×10^{18} cm^{-3} in samples grown at temperatures less than 280 °C. The presence of the P_{In} antisite defects must result from the growth of nonstoichiometric, phosphorus-rich layers, similar to the As-rich layers found in low-temperature GaAs.[27] However, the identification of C_P acceptors in the present samples would imply that vacant phosphorus sites exist on the growth surface, such that carbon atoms can be incorporated on the group-V sites. This could occur if the concentration of indium atoms at the epilayer surface during growth is enhanced by the introduction of CCl_4, possibly due to the greater rate of dissociation of TMIn: this process might also contribute to the high carbon concentration. This proposal is similar to a previous suggestion for MOVPE growth of InP at 625 °C.[28] However, a high surface concentration of indium would be expected to inhibit the incorporation of P_{In} antisites.

V. CONCLUSIONS

In summary, we have presented IR absorption measurements that show that high-resistivity MOVPE InP grown at 500 °C and doped using CCl_4 contains high concentrations (~3×10^{18} cm^{-3}) of both isolated C_P acceptors and H-C_P pairs. These centers have been identified from their LVM's at 546.9 cm^{-1} (C_P) and at 2703.3, 521.1, and 413.5 cm^{-1} (H-C_P). A sharp weak line, superposed on the C_P LVM, may be due to C_{In} donors present at a much lower concentration. It is concluded that the carbon doping from CCl_4 leads to the incorporation of C_P acceptors that overcompensate grown-in donors and that the complete compensation results from the hydrogen passivation of the excess acceptors.

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