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LVM spectroscopy of carbon and carbon–hydrogen pairs in GaAs grown by MOMBE

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Abstract. Cryogenic (4.2 K) infrared absorption measurements of GaAs films grown by MOMBE and containing a high concentration of carbon acceptors ($\sim 2 \times 10^{20}$ cm⁻³) show an asymmetric Fano resonance at the frequency of the C_{As} local mode (582 cm⁻¹). After rapid transient annealing at 950 °C for 12 s in an atmosphere of helium and arsine, there is a reduction in the hole concentration, and localized vibrational mode (LVM) spectroscopy reveals the presence of nearest neighbour H-C_{As} passivated pairs. Previously unreported lines at 452.8 cm⁻¹ and 563.0 cm⁻¹ may be due to the vibrations of the passivated carbon atoms, or possibly C_{Ga} donors.

There is considerable current interest in doping thin films of GaAs with carbon impurities to produce stable p-type layers that may be incorporated into heterojunction bipolar transistors. When films are deposited by the metal organic molecular beam epitaxy (MOMBE) technique using beams of trimethyl gallium (TMG) and As₄ molecules, doping levels of up to 3.5×10^{20} cm⁻³ can be achieved. At this level the resulting hole concentration measured by the Hall effect is only some 40% of the total carbon content as measured by secondary-ion mass spectrometry (SIMS) [1]. It could be speculated that C_{Ga} donors are produced and compensate the C_{As} acceptors, although at low doping levels there is no evidence for the existence of the donor species.

An x-ray study carried out on these films [1] showed significant changes Δa_{\perp} in the lattice spacing normal to the (001) surface of a semi-insulating (SI) substrate. There was good agreement with the change expected from Vegard's law, provided the carbon atoms occupied substitutional lattice sites. However, comparison of the measurements with calculations was not sensitive to the particular site occupied as the difference between the C and Ga covalent radii, $\Delta r_{Ga} = 0.49$ Å, is hardly distinguishable from the corresponding difference between that of C and As with $\Delta r_{As} \approx 0.43$ Å. The epitaxial layers may have been strained since at the highest doping level a value of $\Delta a_{\perp}/a_0 \sim 2 \times 10^{-3}$ was measured, where a_0 is the lattice spacing for GaAs. In an attempt to reduce any such strain, samples have now been subjected to a rapid transient anneal (RTA) at 950 °C for 12 s in an atmosphere of 1.25 SLPM arsine (AsH₃) with 5 SLPM He (SLPM represents standard litre per minute). This treatment led to a reduction in the hole concentration [p] and an *increase* in the lattice parameter. The purpose of this paper is to report measurements using localized vibrational mode (LVM) infrared spectroscopy on these MOMBE layers, both in their as-grown and annealed states. Details of the samples examined are given in table 1. Infrared (IR) absorption measurements were made with a Bruker IFS 113v interferometer at a spectral resolution of either 0.5 or 0.1 cm⁻¹ over the spectral range 100 to 3500 cm⁻¹ with the sample at a temperature close to 4.2 K.

Carbon has a low mass and gives a well known LVM line [2] at 582 cm⁻¹ from ¹²C_{As} which comprises nine fine structure components [3] arising from the various isotopic combinations of Ga nearest neighbours (⁶⁹Ga 60%, ⁷¹Ga 40%) although only five features are resolved, even in lightly doped strain-free crystals. The overall width of this fine structure is only some 0.6 cm⁻¹. A line from ¹²C_{Ga} donors, if they were present, would be expected to lie in the same spectral region (cf the similarity [4] of the LVM frequencies for ²⁸Si_{Ga} donors at 384 cm⁻¹ and ²⁸Si_{As} acceptors at 399 cm⁻¹) but would show no fine structure since the ⁷⁵As nearest neighbours are 100% abundant. The concentration [C_{As}] in high

		Sample		
Parameter		2-16	2-20	2-31
Thickness (μm) s៲мs [C] (cm ⁻³)		7.7 1.7 × 10 ²⁰	2.5 3.5 × 10 ²⁰	0.75 Not uniform
Hall [p] (cm ⁻³)	As-grown After вта Difference	9.4×10^{19} 6.4×10^{19} 3.0×10^{19}	1.4×10^{20} 5.9×10^{19} 8.1×10^{19}	2.3×10^{20} 4.4×10^{19} 1.9×10^{20}
H–С _{Аз} LVM (2636 cm ⁻¹) after вта†	∆ (cm ⁻¹) ၊₄ (cm ⁻²) [H-C _{As}] (cm ⁻³)	$\begin{array}{l} \textbf{2.6} \\ \textbf{6.6} \times \ \textbf{10}^2 \\ \textbf{2.4} \times \ \textbf{10}^{19} \end{array}$	$\begin{array}{l} \textbf{3.8} \\ \textbf{2.3} \times \textbf{10^3} \\ \textbf{8.4} \times \textbf{10^{19}} \end{array}$	$\begin{array}{l} \text{4.2} \\ \text{6.3} \times 10^3 \\ \text{2.3} \times 10^{20} \end{array}$

Table 1.	Details c	f samples.
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† Defect concentration estimated for an apparent charge $\eta = 0.45e$ (see text).

resistivity GaAs can be determined from the integrated absorption (IA) in the 582 cm⁻¹ line, since IA = 1 cm⁻² corresponds to $[C_{As}] = (1.0 \pm 0.2) \times 10^{16}$ cm⁻³ [5, 6]. In highly doped p-type material there is strong electronic absorption in the form of a continuum that increases in strength as the wavelength increases and the carbon LVM has to be separated from that continuum.

Only sample 2-31 was sufficiently thin to give measurable transmission in the region near and just below 582 cm^{-1} in its as-grown state because of this electronic absorption. LVM absorption due to C_{As} was detected as an asymmetric Fano resonance (figure 1), similar in shape to that reported by us previously for ⁹Be_{Ga} acceptors at 482 cm⁻¹ in highly doped GaAs ([p] ~ 10^{19} cm^{-3}) grown by molecular beam epitaxy (MBE) [7].



Figure 1. Asymmetric Fano resonance due to C_{As} in (a) the as-grown sample 2-31 with the corresponding ordinate (left-hand side) calculated from the epitaxial layer thickness of 0.75 μ m and (b) the same sample after RTA, the spectrum being displaced upwards by 36.5 cm⁻¹. The inset shows mixed Ga isotopic fine structure due to absorption from C_{As} in the si substrate superposed on the Fano resonance in spectrum (b). The corresponding ordinate (right-hand side) was calculated from the substrate thickness of 0.46 mm.

The asymmetric shape results from electron-phonon coupling, but theoretical details have not been formulated. A similar interaction has been observed in Bedoped InSb [8], and the effect is also present in boron-doped silicon where anti-resonances are found [6].

A consequence of the interaction is that it is only possible to make qualitative estimates of IA since the LVM absorption is spread over a range of energies around the normal position of the line and 'dips' in the absorption count as negative contributions towards IA [9]. For the as-grown sample 2-31 we have estimated IA $\sim 6 \times$ 10^3 cm⁻², giving [C_{As}] ~ 6 × 10¹⁹ cm⁻³, which is of the same order of magnitude as the value of [p] measured by the Hall effect (table 1). The full-width-at-half-height of the line (Δ) was about 2.4 cm⁻¹—four times greater than that observed for low carbon concentrations in strainfree material [3]. No other LVM lines were observed in the as-grown sample, and so there was no evidence for the presence of C_{Ga} donors. However, weak lines at slightly lower energies than 582 cm^{-1} would not have been detected because of the high level of noise in the spectra resulting from the low sample transmission.

After RTA treatment, all three samples showed a significant reduction in the strength of the electronic continuum consistent with the measured reductions in [p] (table 1), and an additional strong LVM line at 2636 cm⁻¹ was observed. Samples 2-16 and 2-20 remained opaque in the spectral region near 580 cm^{-1} but the increased transmission of sample 2-31 allowed various weak features to be detected (figure 2 and table 2) as discussed below. The C_{As} linewidth narrowed to $\Delta =$ 2.2 cm^{-1} (figure 1) and the IA was estimated to have decreased by a factor of about two, leading to a lower carbon acceptor concentration $[C_{As}] \sim 4 \times 10^{19} \text{ cm}^{-3}$, which is in agreement with the value of [p] measured by the Hall effect (table 1). The Fano asymmetry was less pronounced, possibly due to some electrical compensation of the acceptors. Unfortunately, the presence of interference fringes in the spectra limited a quantitative assessment of the lineshape. Superposed on the Fano resonance was weak fine structure (figure 1), which was attributed to C_{As} acceptors in the si GaAs substrate in an estimated concentration $[C_{As}] \sim 2 \times 10^{15} \text{ cm}^{-3}$.

Concentration v EWHH IA (cm^{-2}) (cm^{-1}) Assignment Δ (cm⁻¹) (cm^{-3}) Lines in epilayer 0.9×10^{19} a 0.9×10^{3} 452.8 Х 1.4 1.4×10^{19} a Y 1.4×10^{3} 563.0 3.2 ¹²C_{A9} $\sim 4 \times 10^{19} \, {}^{a}$ $\sim 4 \times 10^3$ 583 2.2 2.3 × 10^{20 b} ¹²C_{As} 6.3×10^{3} 2636 4.2 н Lines in GaAs si substrate (NB narrow lines)^c ¹¹B_{Ga} 2.6×10^{16} 517.2 0.8 0.35 ¹⁰B_{Ga} 0.5×10^{16} 0.8 0.07 540.4 ¹²C_{As} 2.3×10^{15} 0.23 583 0.6 $^{11}\mathsf{B}_{As}$ 0.7×10^{16} 600.2 1.0 0.19

Table 2. LVM lines in sample 2-31 after RTA.

^a Estimated using the calibration appropriate for isolated C_{As}

impurities, where $A = 1 \text{ cm}^{-2} \equiv \text{impurity concentration} = 1.0 \times 10^{16} \text{ cm}^{-3}$ ($\eta = 3.0 \pm 0.3e$) [5, 6]. Higher values of the concentrations

would be obtained if a lower value of η were applicable.

^b Estimated using an apparent charge $\eta = 0.45e$ (see text).

^o See reference [11] for calibrations of defect concentrations.

The LVM line at 2636 cm⁻¹ (figure 3) has been assigned to the hydrogen stretching mode of $H^{-12}C_{As}$ pairs [10, 11]. Values of Δ for this line decreased with decreasing carbon content in the samples (table 1) as would be expected from the reduction in the internal strain. The concentration of H-12CAs pairs was estimated by assuming an apparent charge (dipole moment per unit displacement) $\eta \sim 0.45e$ (see [12]) to account for the reduction in [p] on annealing sample 2-20. Within experimental error, this same value of η leads to a selfconsistent explanation for the reduction in [p] in all three samples examined (table 1). For comparison, values of η determined more directly for the hydrogen stretch modes of H-Be_{Ga} and H-Si_{Ga} pairs in GaAs are $\eta = 0.7e$ [13] and $\eta = 0.5e$ [14], respectively (an integrated absorption of 100 cm⁻² was measured for a concentration of H-Si_{Ga} pairs of $3 \times 10^{18} \text{ cm}^{-3}$). Thus the assumed value of



Figure 2. LVM absorption lines in the 450 cm^{-1} to 600 cm^{-1} spectral region in sample 2-31 after RTA: the assignments are given in the diagram. Possible assignments for the new lines at 453 cm^{-1} and 563 cm^{-1} (see figure 4 also) are discussed in the text.

 $\eta = 0.45e$ for H-C_{As} pairs is not unreasonable. This value of η would imply that a value of IA = 1 cm⁻² corresponds to a defect concentration of 3.7×10^{16} cm⁻³.

No absorption due to the hydrogen wagging mode of $H^{-12}C_{As}$ pairs was detected in the spectral region near 1400 cm⁻¹ [15], in agreement with previous investigations [10, 11]. However, in sample 2-31, two previously unreported lines designated X and Y were observed at 452.8 cm⁻¹ ($\Delta = 1.4$ cm⁻¹) and 563.0 cm⁻¹ ($\Delta = 3.2$ cm⁻¹) respectively, with relative strengths of 0.6:1 (figures 2 and 4 and table 2). In addition, LVM lines from ¹¹B_{Ga} (517 cm⁻¹), ¹⁰B_{Ga} (540 cm⁻¹) [2] and ¹¹B_{As} (601 cm⁻¹) [16] were observed due to boron impurities in the substrate, with concentrations estimated to be [B_{Ga}] ~ 3 × 10¹⁶ cm⁻³ and [B_{As}] ~ 5 × 10¹⁵ cm⁻³, using previously reported calibrations [17].

The H-C_{As} complex is expected to have trigonal C_{3v} symmetry and so both the hydrogen and the carbon



Figure 3. LVM absorption due to the stretch mode of $H^{-12}C_{As}$ pairs in sample 2-31 after RTA. The inset shows possible BC and AB models for the defect.



Figure 4. Expanded spectra showing the new carbon modes X and Y for sample 2-31 after RTA.

atoms in the pair should have two IR active LVMs, corresponding to longitudinal (ω_{\parallel}) and transverse (ω_{\perp}) modes. Estimates of the frequencies of these four modes cannot be made until the model of the H-C_{As} pair has been established. It is inferred that there is a direct bond between the two atoms since the hydrogen stretch frequency is lowered to 2628 cm⁻¹ for H-¹³C_{As} pairs from 2636 cm⁻¹ for H-¹²C_{As} pairs, in lightly doped bulk material [10]. The frequency of a LVM can be written as [14, 17]

$$\omega_{\rm L}^2 = k_{\rm imp}(1/m_{\rm imp} + 1/\chi M_{\rm nn})$$

where m_{imp} and M_{nn} are the masses of the impurity and nearest neighbours respectively, k_{imp} is a constant which depends on the local interatomic force constants, and χ is a parameter which depends on the local angle bending and bond stretching force constants. Measurements usually lead to values of χ greater than 1.5, e.g. $\chi = 2.8$ for H-Si_{Ga} [14]. We deduce $\chi = 1.0$ from the H-¹²C_{As} and $H^{-13}C_{As}$ stretching frequencies; alternatively, we find $\chi = 0.65$ from the reported frequencies for H⁻¹²C_{As} and $D^{-12}C_{As}$ [11], although corrections for anharmonic effects (see [18]) lead to the somewhat higher value of 0.85. $\chi = 1$ is appropriate for a diatomic molecule and so it is implied that the motion of the carbon atom is not constrained by its nearest neighbours. We have no adequate explanation for this unexpected result. However, if the carbon atom relaxed into the plane of three Ga neighbours (see below), the restoring force to longitudinal displacements would be minimized as its origin would be bond bending only.

Since no H-wagging mode has been detected, it has been proposed [10] that the hydrogen atom occupies a bond centred (BC) position between the C_{As} and a neighbouring Ga atom (figure 3, inset). This conclusion was reached by analogy with the absence of such modes for hydrogen paired with group-II acceptors, including Zn and Be [13] where the hydrogen bonds predominantly to a nearest neighbour As atom. The frequency of the longitudinal mode ω_{\parallel} of the Be_{Ga} atom in the pair occurs

in the lattice continuum, while the corresponding doubly degenerate transverse mode ω_1 is at a higher frequency than that of an isolated Be_{Ga} atom [13]. These observations are in agreement with first principles calculations [19] but appear to disagree with the present results for $H-C_{As}$ pairs if the two new LVM lines X and Y are assigned to ω_{\parallel} and ω_{\perp} . The value of ω_{\parallel} should be low for a BC model for $H-C_{As}$ pairs since the bond between the H atom and the neighbouring Ga atom would have to be very weak for passivation to be effective. Alternatively, it might be argued that the H atom occupies an antibonding (AB) site and a C-Ga bond is lost (figure 3, inset). However, the frequency of the longitudinal mode of the carbon atom in the pair would again be expected to be low (cf LVM results for H-Si_{Ga} pairs in GaAs [14]). It would appear that on the basis of either model, one of the new lines (X or Y) could be ascribed to the transverse mode of the carbon atom in the pair, but a different assignment may be required for the second line. The line at 563 cm^{-1} (Y) is much too strong to be assigned to $^{13}C_{As}$ (natural abundance 1.1%), which gives rise to a LVM line at 561 cm⁻¹[2]. Another alternative is that one of the lines is due to C_{Ga} donors but the line broadening precludes discrimination on the basis of observing nearest neighbour isotopic fine stucture. Finally, the lines could in principle be due to C-C pairs, the existence of which was suggested by Shigetomi et al [20] for samples implanted with high doses of carbon ions. Irrespective of the uncertainties in the interpretation of the lines X and Y, the formation of H-C complexes must account for some, and possibly all, of the reduction in [p] resulting from the RTA treatment. The observation of corresponding LVM lines in samples containing enriched ¹³C and/or deuterium should provide information that would elucidate the interpretation, and it would be helpful to have first principles calculations of the vibrational frequencies of the H-C pairs.

An interesting observation was the appearance of a line from ¹¹B_{As} in the sample 2-31 given the RTA (figure 2). These defects must have been present in the substrate rather than the epitaxial layer as they give LVM absorption only in high resistivity or n-type material when the Fermi level is shifted away from the valence band by more than 200 meV [21, 22]. The defects are not found in stoichiometric GaAs even when it contains a high concentration of B_{Ga} impurities ($\sim 10^{18}$ cm⁻³). It seems likely that there was a loss of arsenic from the substrate during RTA leading to somewhat Ga-rich non-stoichiometric material and site switching of B_{Ga} atoms to arsenic lattice sites. This result was not expected since the RTA was carried out in an atmosphere containing AsH₃.

The present observations are not in agreement with a recent interpretation of the LVM spectrum of carbon in heavily doped MOMBE layers of $Al_xGa_{1-x}As$ with x = 0.24 where a sharp five-line fine structure was found at 582 cm⁻¹ [23]. It has already been stated that such structure would not be expected to be resolved in a highly doped layer because of the disorder produced by the carbon itself, which in that case was present at a

concentration of 1.6×10^{20} cm⁻³. The addition of aluminium would produce yet further disorder and a broadening of the line (see, for example, the work of Murray *et al* [24]). It would seem that the observed spectrum originated from C_{As} acceptors in the GaAs substrate at a concentration ~ 10^{16} cm⁻³ (similar to those observed in the substrate of our annealed sample 2-31).

In summary, it has been shown by LVM absorption measurements that CAs acceptors in thin highly doped GaAs grown by MOMBE give rise to a broad Fano resonance, similar to that found in other highly doped epitaxial p-type layers of III-V compounds and silicon. The introduction of hydrogen into GaAs by heating in arsine has been demonstrated by the observation of the $H^{-12}C_{As}$ stretch mode in the annealed samples. Although the processing temperature was high, the presence of this complex is not unexpected since it has been shown [11] that once formed it is not easily dissociated. To the best of our knowledge, the introduction of hydrogen into GaAs by this means is novel, though it is now well established for InP [25-28]. The introduction of interstitial hydrogen impurities should lead to an increase in the lattice parameter as was found here, but a quantitative analysis requires further measurements to be made. SIMS and x-ray data for the present samples will be presented in a later publication.

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