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Mechanism of free electron concentration saturation phenomenon in Te-GaSb single crystal*

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Te-doped GaSb single crystal grown by the liquid encapsulated Czochralski (LEC) method exhibits a lag of compensating progress and a maximum carrier concentration around 8×10^{17} cm^{-3}. The reason for this phenomenon has been investigated by a quantity concentration evaluation of the Te donor and native acceptor. The results of glow discharge mass spectrometry (GDMS) and Hall measurement suggest that the acceptor concentration increases with the increase of Te doping concentration, resulting in the enhancement of electrical compensation and free electron concentration reduction. The acceptor concentration variation is further demonstrated by photoluminescence spectra and explained by the principle of Fermi level dependent defect formation energy.

Keywords: GaSb, defect compensation, Hall effect measurement, photoluminescence spectroscopy

PACS: 71.55.Eq, 78.55.Cr, 81.05.Ea, 81.70.Jb **DOI:** 10.1088/1674-1056/28/5/057102

1. Introduction

With excellent electrical and optical properties, III–V compound semiconductors have become fundamental materials for the photoelectronic and microelectronic industries, among which gallium antimonide (GaSb) is of particular importance. GaSb-based devices such as laser diodes, photovoltaic cells, microwave devices, and infrared (IR) detectors are promising for applications in mid-infrared regions.[1–5] GaSb has also become an excellent substrate for epitaxial growth of various ternary and quaternary III–V compounds due to their well-matched lattice parameters.[6–9]

Irrespective to crystal growth methods, undoped GaSb is p-type with an intrinsic acceptor defect concentration up to 10^{17} cm^{-3}.[10,11] This high concentration of native defects in GaSb substrates may lead to an enhancement of IR absorption and electrical compensation which has a negative effect on fabricating high performance IR detectors. The p-type conductivity in undoped GaSb is usually believed to arise from gallium vacancies (V_{Ga}) and gallium in antimony site (Ga_{Sb}). These acceptors are sometimes seen as $V_{Ga}Ga_{Sb}$ complexes with a doubly ionizable nature.[12,13] Other researchers such as Ling *et al.*[14] and Slotte *et al.*[15] have ruled out the V_{Ga}-related origin hypothesis using positron annihilation spectroscopy. Unlike GaAs and InP,[16,17] GaSb has a narrow bandgap of 0.73 eV with no deep donors nor deep acceptors, thus it may not be possible to make GaSb semi-insulating for manufacturing high-frequency devices. GaSb single crystal with various resistivities can be obtained by controlling the compensation degree via doping. In addition, GaSb single crystal with high compensation, which has a relatively high resistivity, exhibits better transmittance in the infrared region.[18,19] However, GaSb single crystals grown by liquid encapsulated Czochralski (LEC) method usually can only achieve a maximum carrier concentration around 8×10^{17} cm^{-3}.

Researches on the compensating mechanism and doping concentration have been developed for decades and still need to be continued. In this paper, to comprehend the mechanism of defect compensation and the effects of impurity on the electrical and optical properties, undoped and Te-doped GaSb single crystal samples were prepared by the LEC method. The concentrations of Te dopant in the samples were measured by glow discharge mass spectrometry (GDMS). The electrical and optical properties of the samples were analyzed by Hall effect measurements and photoluminescence (PL) spectroscopy. The result shows evidence that the doping of Te impurity may promote the ionization of the native acceptors and slow down the compensation process.

2. Experiment

Undoped and Te-doped GaSb single-crystal ingots were grown by the LEC method using high-purity (99.9999%) Ga and Sb metals as the raw materials. Various doses of high-purity Te elementary substance were used as dopant to achieve

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different doping levels. After growth, 2-inch [100] oriented substrates were then cut from these ingots and applied with chemical mechanical polishing on both sides. The substrates were later washed in a series of organic and inorganic solutions to be epi-ready. The samples used in this work were all sliced into 1 cm×1 cm squares before other measurements.

The Hall effect measurements were carried out at 300 K using the Van der Pauw method to test and analyze the electrical properties of different GaSb samples. A four-point probe was placed at the four corners of the square samples. By soldering indium dots to the surfaces of the samples, ohmic contacts could be achieved.

To determine the concentrations of Te in the doped samples, GDMS measurements were taken using a VG9000 system which provides a ppb/ppt (weight) level detection limit.

For PL spectra, a Bruker Vertex 80 V Fourier infrared spectrometer (FTIR) with a 647 nm Kr^+ laser excitation source was used. And a HgCdTe photodetector with a resolution of 0.5 meV was employed to detect the signal. The PL measurements were all taken at 10 K.

3. Results and discussion

For Te-doped GaSb samples at different doping levels, the Te concentrations measured by GDMS as well as the electrical properties gained from the Hall effect measurements are listed in Table 1. The Te-doped samples show n-type conductivity with a net electron concentration varying from as low as 3.09×10^{16} cm^{-3} to as high as 7.50×10^{17} cm^{-3}.

Table 1. Te concentrations measured by GDMS and electrical properties of Te-doped samples.

Sample	Sampling position of ingot	Te concentration/cm^{-3}	Type	Carrier concentration/cm^{-3}	Ionized acceptor concentration/cm^{-3}	Mobility /cm^2·V^{-1}·s^{-1}
G01H	head	2.41×10^{17}	n	3.09×10^{16}	2.10×10^{17}	3.04×10^{3}
G02H	head	5.04×10^{17}	n	1.24×10^{17}	3.80×10^{17}	2.44×10^{3}
G03H	head	7.42×10^{17}	n	3.35×10^{17}	4.07×10^{17}	3.15×10^{3}
G04H	head	7.16×10^{17}	n	4.14×10^{17}	3.02×10^{17}	3.24×10^{3}
G05T	tail	1.93×10^{18}	n	7.50×10^{17}	1.18×10^{18}	2.50×10^{3}

Basically, the carrier concentration increases with the Te concentration. Te is a shallow donor with an impurity energy level above the conduction band minimum. We assume that the donor impurities are fully ionized at 300 K, therefore the ionized donor concentration roughly equals the donor concentration, i.e., $N_D^+ \approx N_D$, and the ionized acceptor concentration $N_A^- = N_{A1}^- + N_{A2}^-$, depending on the ionization degree of the doubly ionizable native acceptor. After linear fitting of the data, in relatively lightly doped samples, which has a Te concentration lower than 10^{18} cm^{-3}, the net carrier concentration can be estimated as

$$n_0 \approx N_D - N_A^- = 0.72\times C_{Te} - 1.72\times10^{17}\ \text{cm}^{-3}. \quad (1)$$

For sample G05T with a Te concentration up to 1.93×10^{18} cm^{-3}, the result deviates off the trend shown in Eq. (1), indicating that the net carrier concentration tends to saturate under heavily doped circumstance.

Based on the dosages of Te elementary substance added to the raw materials in the crystal growth process, the approximate Te concentrations in the Te-doped samples can also be calculated using a modified version of Scheil equation[20]

$$C_S = a\times k\times C_0\times(1-f_S)^{k-1}, \quad (2)$$

where C_S is the concentration of Te impurity in the GaSb crystal after a fraction f_S of the melt is solidified, C_0 stands for the Te concentration in the initial GaSb melt, and the distribution coefficient of Te is taken as $k = 0.37$.[21] It should be noted that due to the volatility of the Te element at high temperature, the actual concentration of Te in the GaSb melt is always much lower than the ratio of elemental Te added in the raw materials in our crystal growth circumstance, thus a correction factor of $a = 0.39$ is taken into account according to the GDMS result.

Table 2. Calculated Te concentrations and electrical properties of undoped and Te-doped GaSb samples.

Sample	Sampling position of ingot	Te concentration/cm^{-3}	Type	Carrier concentration/cm^{-3}	Ionized acceptor concentration/cm^{-3}	Mobility /cm^2·V^{-1}·s^{-1}
G06H	head	0	p	1.58×10^{17}	1.58×10^{17}	6.13×10^{2}
G06T	tail	0	p	1.58×10^{17}	1.58×10^{17}	6.14×10^{2}
G07H	head	2.62×10^{17}	n	1.81×10^{16}	2.44×10^{17}	1.76×10^{3}
G08H	head	2.71×10^{17}	n	3.09×10^{16}	2.40×10^{17}	3.04×10^{3}
G09H	head	3.80×10^{17}	n	1.24×10^{17}	2.56×10^{17}	2.45×10^{3}
G10H	head	3.84×10^{17}	n	1.28×10^{17}	2.56×10^{17}	3.23×10^{3}
G11H	head	6.87×10^{17}	n	3.35×10^{17}	3.52×10^{17}	3.15×10^{3}
G12H	head	7.11×10^{17}	n	3.49×10^{17}	3.62×10^{17}	2.89×10^{3}

Table 2 shows the calculated Te concentrations of more undoped and Te-doped samples using Eq. (2) and their net carrier concentrations measured at 300 K. For convenience of calculation, the Te-doped samples are taken from the head of the ingots. The relationship between the net carrier concentration and the calculated Te concentration C'_{Te} can be approximated by

$$n_0 \approx N_{\mathrm{D}} - N_{\mathrm{A}}^{-} = 0.72 \times C'_{\mathrm{Te}} - 1.61 \times 10^{17}\ \mathrm{cm}^{-3}. \quad (3)$$

These results are also summarized in Fig. 1. The data points representing lightly doped samples have a linear distribution in general. The best fit curve of all data points shows a trend of saturation of the free electron concentration. As can be seen from the intercepts, the intrinsic hole concentration in undoped p-type GaSb crystals would be approximately 1.61×10^{17}–$1.72 \times 10^{17}\ \mathrm{cm}^{-3}$, which is comparable to the carrier concentration measured in our undoped samples ($1.58 \times 10^{17}\ \mathrm{cm}^{-3}$) and data from other researchers, such as Sunder *et al.*[22] have reported carrier concentrations of undoped GaSb crystals in the range $\sim 1.6 \times 10^{17}\ \mathrm{cm}^{-3}$ and Lui *et al.*[23] have reported a LEC grown undoped GaSb wafer with a $2 \times 10^{17}\ \mathrm{cm}^{-3}$ hole concentration.

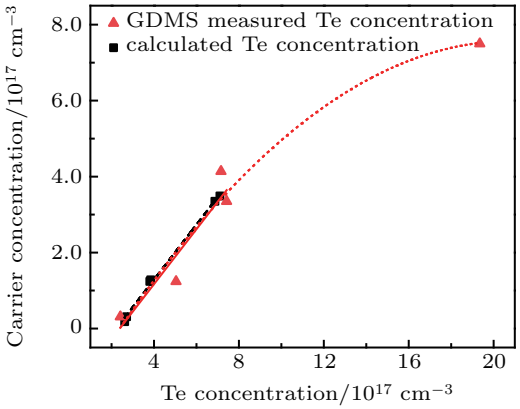


Fig. 1. Carrier concentration as a function of Te concentration. Triangles: data gained from the GDMS and Hall effect measurements. Squares: data gained from Te concentration calculation and Hall effect measurements, straight line: data linear fitted by Eq. (1), dashed line: data linear fitted by Eq. (3), dotted line: best fit curve for data gained from the GDMS and Hall effect measurements.

Because of the volatility of Sb during the process of crystal growth, a Ga-rich condition is usually obtained and Sb vacancies ($\mathrm{V_{Sb}}$) are easy to form under this condition. The $\mathrm{Ga_{Sb}}$ antisite is found to have a fairly low formation energy of 1.37 eV,[24] thus, the $\mathrm{V_{Ga}Ga_{Sb}}$ acceptor complexes can originate from Sb vacancies through the nearest-neighbor diffusion mechanism[25] as

$$\mathrm{V_{Sb}} \leftrightarrow (\mathrm{V_{Ga}Ga_{Sb}}). \quad (4)$$

Theoretically, Te impurities occupying the sites of $\mathrm{V_{Sb}}$ will reduce the probability of the formation of $\mathrm{Ga_{Sb}}$. But, as a matter of fact, the concentration of ionized acceptors measured still sees a growth under Te-doping conditions. For lightly doped samples, a slope of 0.72 exists in both Eqs. (1) and (3), meaning that the compensation process is slower than the increase of Te concentration. We speculate that this slope is related to the doubly ionizable nature of the native acceptors. While some native acceptors are compensated, the introduction of Te dopants also reduces the ionization energy of the $\mathrm{V_{Ga}Ga_{Sb}}$ acceptor complexes, promoting their ionization process. In other words, the amount of the singly ionized acceptors $(\mathrm{V_{Ga}Ga_{Sb}})^{-}$ or the doubly ionized acceptors $(\mathrm{V_{Ga}Ga_{Sb}})^{2-}$ increases with the concentration of Te impurity, which needs more donors if full compensation is expected. If the fully ionized Te impurities are all the donors in the samples, the ionized acceptor concentration could increase to

$$N_{\mathrm{A}}^{-} \approx 0.28 \times C_{\mathrm{Te}} + 1.61 \times 10^{17}\ \mathrm{cm}^{-3}. \quad (5)$$

For the heavily doped sample, the lag of compensating progress is even worse. The slope decreases significantly showing the evidence for saturation of free electron concentration. Instead of the increase of the $\mathrm{V_{Ga}Ga_{Sb}}$ ionization rate, a boost for the formation of native acceptors must be responsible for this phenomenon. Undoped samples G06H and G06T taken from both the head and tail of a same ingot have the same carrier concentration, indicating that the ratio of Ga to Sb, whether in the crystal or the melt, remains unchanged at different stages of crystal growth. This means that the high concentration of Te facilitates the generation of $\mathrm{V_{Ga}Ga_{Sb}}$ acceptor complexes.

Meanwhile, the carrier mobility listed in Tables 1 and 2 shows a growth and decline as the Te concentration increases. For samples with a net carrier concentration lower than $1.28 \times 10^{17}\ \mathrm{cm}^{-3}$, the mobility increases with the Te concentration. This may be caused by the electron screening effect as the electrons fill up the higher lying conduction band valley.[26] When the Te concentration keeps increasing, more native acceptors and Te dopants are ionized, thus the ionized-impurity scattering becomes the dominant scattering mechanism and the electron–electron scattering also comes into effect, resulting in the decrease of mobility.

Figure 2 shows the variation of the ionized acceptor concentration whose overall line shape is near hyperbolic. The slope of the asymptotic line at low Te concentration is 0.28 corresponding to Eq. (3). The growth of ionized acceptor concentration accelerates at a higher doping level. For the asymptotic line at high Te concentration, its slope should be close to 1 if the maximum carrier concentration is $8 \times 10^{17}\ \mathrm{cm}^{-3}$.

This result can be further verified by our PL spectra demonstrated in Fig. 3. For the undoped GaSb sample, we see an intense peak at 779 meV commonly known as band A corresponds to the doubly ionizable native acceptor defect

$V_{Ga}Ga_{Sb}$ at its neutral state.[27–29] A weaker peak, known as band G, at 756 meV is caused by another acceptor associated with Ga_{Sb}.[28,30] This peak annihilates in the lightly doped samples suggesting that the Ga_{Sb} defects are suppressed by Te compensation. Other research has shown that band G could also be eliminated when the GaSb samples were annealed in Sb ambient.[31] And band D which shows a peak at 797 meV can be assigned to the reported excitons bound to the neutral acceptor.[32]

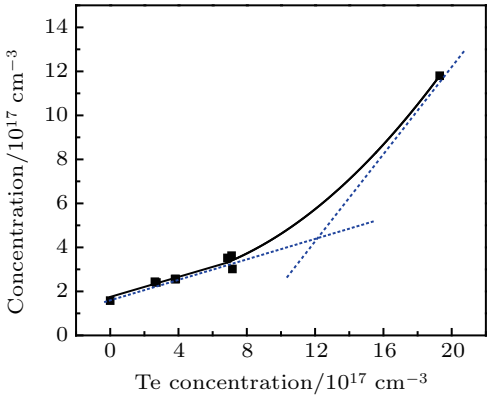


Fig. 2. The variation of ionized acceptor concentration. Squares: calculated concentration of ionized acceptors at different Te concentration. Dotted line: asymptotic lines for the fitted hyperbolic curve.

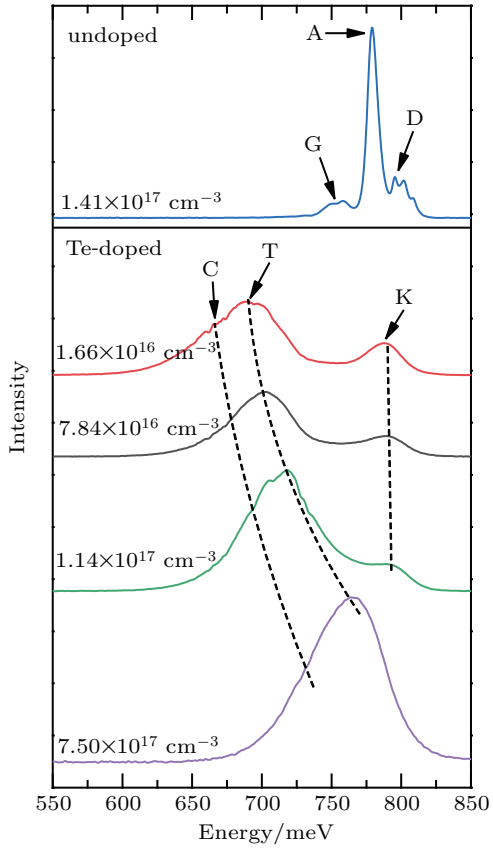


Fig. 3. PL spectra of undoped and Te-doped GaSb samples measured at 10 K. The undoped GaSb sample is p-type. The net carrier concentrations of the Te-doped GaSb samples listed on the left are measured at room temperature. The same emission peaks of different PL curves are linked by dotted lines.

For lightly doped GaSb samples, a broad band containing band C and band T appears. This combination then becomes the main peak with the increase of Te doping level. It has been suggested that band C corresponds to the singly ionized native acceptor $(V_{Ga}Ga_{Sb})^-$, and band T arises from a Te_{Sb} donor complexing with a doubly ionized $(V_{Ga}Ga_{Sb})^{2-}$ center.[28] The specific process can be described as follows:

$$(V_{Ga}Ga_{Sb})^{2-} + Te_{Sb}^{+} \rightarrow (V_{Ga}Ga_{Sb}Te_{Sb})^{-}. \quad (6)$$

We suggest that the Fermi level moves upward when Te impurity is introduced into GaSb, resulting in an increase of the occupation probability of the residual native acceptor. That is, more $V_{Ga}Ga_{Sb}$ acceptor centers, which are at a neutral state at first, capture a single electron and become $(V_{Ga}Ga_{Sb})^-$ centers. Thus, as can be seen from Fig. 3, a strong peak of band C appears. As the doping level slightly increases, the intensity of band C drops off a bit. This means with a higher Te concentration, more $(V_{Ga}Ga_{Sb})^-$ acceptors are further ionized into $(V_{Ga}Ga_{Sb})^{2-}$; therefore, more $(V_{Ga}Ga_{Sb})^{2-}$ acceptors are transformed into $(V_{Ga}Ga_{Sb}Te_{Sb})^-$ complexes. Hence the peak intensity of band T rises.

For the heavily doped sample, the intensity of the broad peak sees a significant enhancement, which means the amount of all the ionized acceptors is way more than that in the lightly doped samples. This change corresponds to the decrease of the slope in Fig. 1 and confirms that a high concentration of Te promotes the formation of more $V_{Ga}Ga_{Sb}$ acceptor defects.

In addition, both band C and band T show a blue shift as the doping concentration increases. This phenomenon can be explained by the Moss–Burstein effect.[33] GaSb is easy to become degenerate at relatively low electron densities, as the doping level get higher, the Fermi level rises into the conduction band, which increases the optical band gap.

Comparing the doped samples to the undoped sample, the intensities of band A and band D decrease rapidly, and the two peaks merge into a new one we call band K at the position of 787 meV. Rather than annihilating immediately, this newly formed peak weakens gradually which proves that although Te impurity at a concentration much higher than that of the initial native acceptors has been doped into the GaSb crystal, a certain number of uncompensated native acceptors still exist.

4. Conclusion

We prepared undoped and Te-doped GaSb samples by the LEC methods. The net carrier concentration versus Te concentration (fitted lines) shows that the defect compensation process lags behind the impurity doping process and the net carrier concentration tends to saturate, indicating that the introduction of Te dopants may promote the formation and ionization process of the doubly ionizable $V_{Ga}Ga_{Sb}$ acceptor complexes. The results of the PL spectra further corroborate this

hypothesis. At relatively low doping levels, when the Te concentration increases, more $V_{Ga}Ga_{Sb}$ acceptor centers are ionized and new acceptor complexes of $(V_{Ga}Ga_{Sb}Te_{Sb})^{-}$ form. As the doping level keeps growing, more $V_{Ga}Ga_{Sb}$ acceptor complexes are generated, making the compensating progress slower.

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