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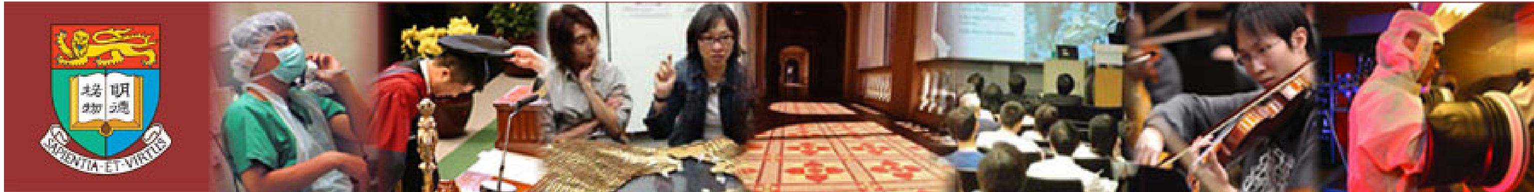

Title	Native donors and compensation in Fe-doped liquid encapsulated Czochralski InP
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Native donors and compensation in Fe-doped liquid encapsulated Czochralski InP

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Undoped and Fe-doped liquid encapsulated Czochralski (LEC) InP has been studied by Hall effect, current–voltage ($I-V$), and infrared absorption (IR) spectroscopy. The results indicate that a native hydrogen vacancy complex donor defect exists in as-grown LEC InP. By studying the IR results, it is found that the concentration of this donor defect in Fe-doped InP is much higher than that in undoped InP. This result is consistent with the observation that a much higher concentration of Fe^{2+} than the apparent net donor concentration is needed to achieve the semi-insulating (SI) property in InP. By studying the $I-V$ and IR results of Fe-doped InP wafers sliced from different positions on an ingot, the high concentration of Fe^{2+} is found to correlate with the existence of this hydrogen complex. The concentration of this donor defect is high in wafers from the top of an ingot. Correspondingly, a higher concentration of Fe^{2+} can be detected in these wafers. These results reveal the influence of the complex defect on the compensation and uniformity of Fe-doped SI InP materials. © *2001 American Institute of Physics.* [DOI: 10.1063/1.1331644]

I. INTRODUCTION

Semi-insulating (SI) InP is an important material used for the fabrication of microwave devices and integrated circuits. Nowadays commercial SI InP wafers are prepared by doping with a high concentration of deep acceptor Fe ($\sim 10^{16}\,\mathrm{cm}^{-3}$). It is well-understood that the Fe acceptors compensate donor defects in InP giving rise to SI conduction property.[1,2] In this case, Fe has two charge states in InP: Fe^{2+} and Fe^{3+}. Fe^{2+} is the ionized state of the Fe acceptor and is formed by capturing an electron to the neutral Fe^{3+} state. The concentration of Fe^{2+} is therefore expected to be equal to that of the net donor defects in SI InP. It is an advantage to have a low Fe concentration in SI InP since the out-diffusion of Fe has been observed to be harmful in the epitaxy process.[3]

A phenomenon that has been known for a long time is that the Fe^{2+} concentration in liquid encapsulated Czochralski (LEC) is far higher than the apparent concentration of net donor impurities, which is the difference of the concentration between donor N_D and acceptor N_A impurities.[4–6] Although the apparent net donor concentration of undoped LEC InP material is in the range of low to middle $10^{15}\,\mathrm{cm}^{-3}$, the doping concentration of Fe to get SI InP is still higher than $10^{16}\,\mathrm{cm}^{-3}$.[7–9] These facts imply the existence of native donor(s) in LEC grown Fe-doped SI InP and their total concentrations should be $[Fe^{2+}]-(N_D-N_A)$.

Recently, Zach has investigated the defect concentrations and compensation in Fe-doped SI InP.[10] In this work, the concentrations of Fe^{3+} and Fe^{2+} have been determined by electron paramagnetic resonance (ESR) and infrared absorption spectroscopy, respectively. The total concentration of Fe has been measured by secondary ion mass spectroscopy. Residual donors (mainly Si, S) and acceptors (Zn, Cd, etc.) have been measured by glow discharge mass spectroscopy (GDMS). The concentration of Fe^{2+} has indeed been found to be higher than N_D-N_A by about $(1-4)\times 10^{15}\,\mathrm{cm}^{-3}$, depending on the source of the samples. A native defect, which has a vibrational mode at 2315 cm^{-3}, has also been found to correlate with the change of the concentration of Fe^{2+}.

The above mentioned work has given some insights into the compensating donor in as-grown Fe-doped SI InP. However, the samples involved came from different sources with various growth conditions. Some uncertainty in the work has also resulted from the lack of information about the hydrogen complex defect. The comparison of the results was indirect and some inconsistency existed in this case. If this hydrogen complex defect is indeed a compensating defect in Fe-doped InP, the concentration of donor defect is expected to be high in Fe-doped InP when it is compared with undoped InP grown under the same condition. This will give a positive confirmation of the native donor defect in InP and explain the fact that the Fe^{2+} concentration is significantly higher than that of net donor impurities.

In our recent work, it has been found that the hydrogen complex is annihilated in annealed undoped and Fe-doped InP.[11,12] The apparent donor concentration of annealed undoped InP has decreased significantly. These results have indicated that the hydrogen complex is a donor defect in as-grown LEC InP. Further work has proved that this complex is a common defect in as-grown LEC InP.[13] It is necessary to study the abundance and influence of this defect on the compensation and properties of Fe-doped SI LEC InP.

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In this work, we report on the investigation of native hydrogen complex defects in undoped and Fe-doped LEC InP materials grown in our laboratory. The concentration of this hydrogen complex defect in Fe-doped InP has been found to be much higher than that in undoped InP material. The concentration of Fe^{2+} is also found to increase with increasing defect concentration by comparing infrared absorption spectra of the samples from different positions of an ingot. These results give direct evidence about the donor property of the hydrogen complex. By comparison with the results of undoped and Fe-doped LEC InP materials we deduce that the associated increase of donor concentration in the Fe-doped material comes from this complex defect. The influence of this donor complex on the compensation of Fe-doped SI InP is also studied.

II. EXPERIMENTAL PROCEDURES

Liquid encapsulated Czochralski (LEC) Fe-doped SI InP wafers grown in our laboratory were used for the measurements. The electrical parameters of the samples were obtained by Hall effect measurements. Ohmic contacts in Van der Pauw configuration were made using soldered indium dots.

To carry out $I-V$ measurements, both sides of the samples were first lapped and then degreased by acetone and methanol. The wafer was then etched, rinsed in deionized water, dried, and put into the metal deposition chamber. Gold spots of 1 mm in diameter were deposited on one side and of 6 mm in diameter on the other side of the sample to make electrical contacts. It was found that the annealing process normally required to get ohmic contacts was not necessary because no significant difference for the $I-V$ characteristics of samples with and without alloy annealing was observed. The sample thickness was 0.7 mm for this measurement. No guard ring was used since the electrical measurements were carried out above room temperature and in general leakage currents were negligibly small. Some small leakage current effects were noticeable around 300 K, but were not significant.

The $I-V$ measurement system consisted of a HP 3245A programmable voltage generator which drove a programmable EG&G ORTEC 556H high voltage power supply, a Keithley 485 digital programmable electrometer, and an IBM XT computer.

A Bruck ISF120 Fourier transform infrared absorption instrument was used for the infrared absorption measurement. 2.5 mm thick wafers polished on both sides were used for these measurements.

III. RESULTS AND ANALYSIS

A. Hall measurements

The electrical parameters of the samples are listed in Table I. These undoped and Fe-doped LEC InP samples are grown by using the same raw materials (6N indium and 6N red phosphorus, etc.). GDMS measurements show that the net donor impurity concentration (donors being mainly S and Si and acceptors being Zn, Cd, and Cu) (N_D-N_A) in undoped InP is less than $1\times10^{15}\,cm^{-3}$. This concentration is much lower than the net donor concentration of about $5\times10^{15}\,cm^{-3}$ measured by Hall effect. This fact implies the existence of native donor(s) in as-grown undoped LEC InP.

TABLE I. Room temperature electrical parameters of Fe-doped wafers at different positions of an ingot.

Sample No.	Dopant	Position	Carrier concentration (cm^{-3})	Mobility ($cm^2/V\,s$)	Resistivity (Ω cm)
S-01	undoped	top	4.6×10^{15}	4.42×10^{3}	
		tail	5.2×10^{15}	4.15×10^{3}	
S-02	Fe	top	4.63×10^{8}	1.35×10^{3}	1.00×10^{7}
		middle	2.48×10^{8}	1.56×10^{3}	1.61×10^{7}
		tail	6.61×10^{7}	1.43×10^{3}	6.63×10^{7}
S-03	Fe	top	7.06×10^{14}	2.91×10	4.01×10^{2}
		tail	4.19×10^{8}	1.28×10^{3}	9.26×10^{6}

It can also be seen that one Fe-doped InP sample sliced from the top of an ingot is not semi-insulating. In this sample, the concentration of Fe is 30% lower than the conventional concentration by which SI InP can be prepared reproducibly. The compensation in this part of the ingot is not strong enough and most Fe atoms are in the Fe^{2+} states.

B. Current–voltage ($I-V$) measurements

The Fe-doped samples have been studied by current–voltage ($I-V$) measurements. The $I-V$ characteristics of these samples are shown in Figs. 1 and 2. The $I-V$ characteristics are consistent with previous works on SI Fe-doped InP.[14–19] Such electrical transport behavior can be described by the space charge limited current theory of Lampert and Mark[20] which may be summarized as follows. For single carrier injection into a SI material having a single trap, the current density j in the ohmic and space charge limited regimes are expressed by:[20]

$$j=n_0 q\mu V/d, \tag{1}$$

$$j=\tfrac{9}{8}\theta\epsilon\mu V^2/d^3. \tag{2}$$

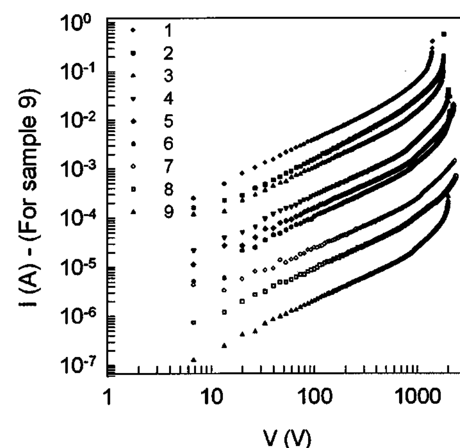

FIG. 1. Room temperature $I-V$ curves of Fe-doped SI InP samples. The curves numbered from 1 to 9 represent results of samples sliced sequentially from top to tail of the S-02 ingot in Table I. The curves have been separated intentionally for better clarity.

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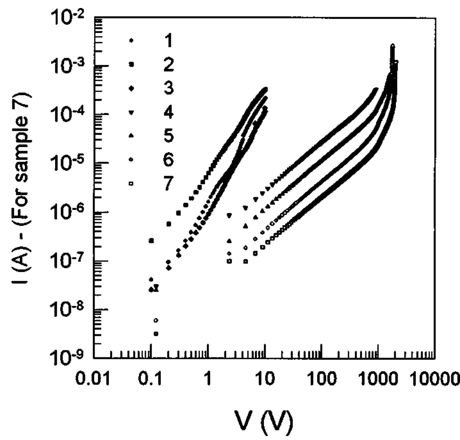

FIG. 2. Room temperature $I-V$ curves of a Fe-doped InP ingot labeled S-03 that is high resistivity in the top and SI at the tail. The curves numbered from 1 to 7 represent results of samples sliced sequentially from top to tail of the ingot. The curves have been separated intentionally for better clarity.

The transition between linear and quadratic regimes begins at a voltage V_{Ω} given by

$$V_{\Omega}=\frac{8}{9}qn_0d^2/\theta\epsilon \tag{3}$$

while the trap filling limited voltage V_{TFL} is described by

$$V_{\mathrm{TFL}}=\frac{qp_td^2}{2\epsilon}, \tag{4}$$

where d is the sample thickness, p_t is the concentration of empty deep traps, and $\theta=n/(n+n_t)$, where n_t is the concentration of electron occupied traps, other symbols having their usual meanings. It can be seen that the value of V_{TFL} depends on the concentration of empty traps, while the current in the transition regime (quadratic) is controlled by the ionized trap concentration. For the as-grown Fe-doped SI InP, the concentration of $Fe^{2+}(=n_t)$ and $Fe^{3+}(=p_t)$ can be known from Eqs. (3) and (4) by the measured current and voltage data.

The curves in Fig. 1 are measured from samples sliced from a Fe-doped SI InP ingot that is semi-insulating from top to tail. It can be seen that the $I-V$ curves of the samples from the top of the ingot do not have the apparent superlinear (quadratic) characteristics. The current increases almost linearly with applied voltage until it rises rapidly due to the heating effect. This fact can be ascribed to the high concentration of Fe^{2+} (or say low concentration of Fe^{3+}) in these samples. Thus the trap filling process cannot be observed clearly. In contrast, the $I-V$ curves of samples from the middle and tail of the ingot exhibit the typical characteristics of insulating material, i.e., with linear (ohmic), quadratic regions on the curves. It is clear that the Fe concentration in these samples is much higher than that in the sample from the top of the ingot. The Fe^{3+} concentration is also high in these samples. This is in agreement with the fact that Fe has a very low segregation coefficient ($\sim 1\times 10^{-3}$) and incorporates into the material in the form of solidified fraction g: $C=C_0k_{\mathrm{eff}}(1-g)^{k_{\mathrm{eff}}-1}$ during the growth process.[21] The concentration of Fe will increase rapidly as the crystal grows.

Figure 2 shows the $I-V$ curves of samples sliced from an Fe-doped InP ingot that is high resistivity ($10^2\ \Omega$ cm) at the top and SI ($10^7\ \Omega$ cm) at the tail. It is seen that there is no trap filling process for those high resistivity samples. However, such a process can be clearly seen by the $I-V$ of the samples from the ingot tail which are semi-insulating. This phenomenon is obviously caused by the fully ionized Fe acceptor in the samples from the top of the ingot in which the Fe concentration is not high enough. The compensation in the ingot top is much lighter than that in the ingot tail.

The $I-V$ characteristics of three samples in the top, middle, and tail from 295 to 400 K are shown in Figs. 3(a)–3(c). The resistance R at different temperature for each sample is obtained from the slope of linear region in the $I-V$ curve. Figure 3(d) shows the Arrhenius plots of $\ln R$ versus 1/kT for the three samples. The plots have a linear tendency at low 1/kT but become sublinear at higher 1/kT, probably as a result of leakage current effects (no guard ring was used on our samples). Taking the higher three temperature points for all the samples, an average activation energy of the value of 0.65 ± 0.05 eV was obtained, a value which is in good agreement with the Fe acceptor level in InP measured by other methods, such as deep level transient spectroscopy[22,23] and photoconductivity.[24] This indicates that the Fe acceptor is the dominant deep compensation center in Fe-doped InP materials. The trap filling process revealed by $I-V$ measurements must be related to the Fe acceptor in the SI samples.

C. Infrared absorption measurements

The infrared absorption spectra at 10 K of undoped and Fe-doped InP samples are shown in Fig. 4. An absorption peak around 2316 cm^{-1} can be seen on the spectra in Fig. 4(a) of undoped and Fe-doped InP. This absorption has been proved to be the local vibrational mode of a hydrogen indium vacancy complex $V_{\mathrm{In}}H_4$.[25] It can be seen from the spectra that this absorption is much stronger for the Fe-doped sample than the undoped InP sample. The integrated absorptions of undoped and Fe-doped InP samples are 0.2–0.3 and 0.4–0.6 cm^{-2}, respectively. The concentrations of this complex in undoped and Fe-doped InP are around 5×10^{15} and $(8-12)\times 10^{15}\ cm^{-3}$, respectively, if we use the calibration $2\times 10^{16}\ cm^{-3}$ of complex Zn–H in InP.[26] It is evident that the concentration of $V_{\mathrm{In}}H_4$ in Fe-doped InP is much higher than that in undoped InP. This fact implies that a high concentration of $V_{\mathrm{In}}H_4$ is formed when InP is doped with Fe. It is also noted that the concentration of $V_{\mathrm{In}}H_4$ in samples from the ingot tail is much lower than that in those from the ingot top.

The absorption spectra from 2700 to 3000 cm^{-1} of Fe-doped and undoped InP samples are shown in Fig. 4(b). The absorption peaks around 2848 cm^{-1} have been proved to originate from the intracenter transitions of Fe^{2+}.[27,28] The integrated absorption of these peaks can be used to measure the concentration of Fe^{2+} in InP.[28] It can be seen that these absorptions cannot be detected in undoped InP, indicating a very low concentration of Fe ($<10^{14}\ cm^{-3}$) in this sample. Compared with Fig. 4(a), the Fe-doped samples with a strong absorption of $V_{\mathrm{In}}H_4$ also have a strong absorption of Fe^{2+} intracenter transitions. This fact indicates that a high concentration of $V_{\mathrm{In}}H_4$ correlates with a high concentration of Fe^{2+} in Fe-doped material.

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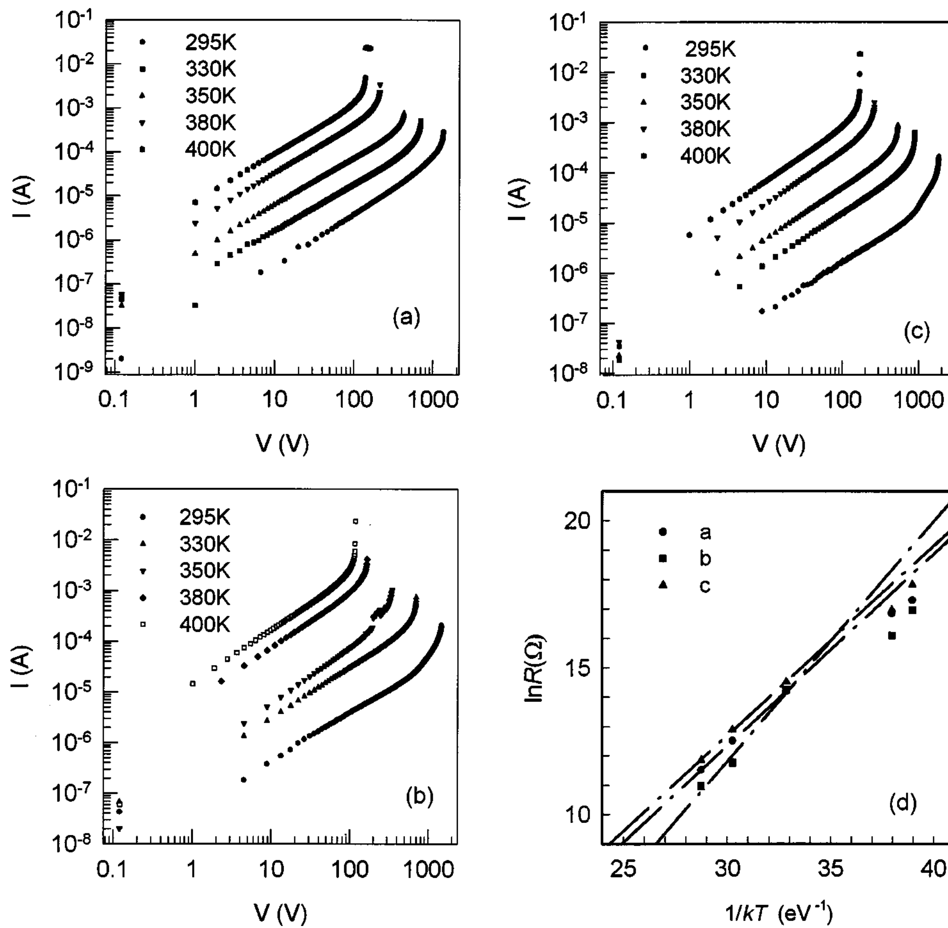

FIG. 3. I–V curves of Fe-doped InP sample labeled S-02 from 295 to 400 K. The three samples are from ingot top (a), middle (b), and tail (c), respectively. Curves in (d) are plots of $\ln R$ vs 1/kT which are used to give the thermal activation energy.

IV. DISCUSSION

The electron concentration and resistivity of semiconductor can be expressed by

$$n = N_C \exp\left[-\frac{E_C - E_F}{kT}\right], \tag{5}$$

$$\rho = (\mu_n n q)^{-1}, \tag{6}$$

where all symbols have their usual meanings. In Fe-doped InP, the charge neutrality condition gives

$$n + N_A + N_{\mathrm{Fe}^{2+}} = p + N_D + N_{\mathrm{ND}}, \tag{7}$$

where N_{ND} is the concentration of native donor. For SI material, $n \approx p$, thus $N_D + N_{\mathrm{ND}} - N_A = N_{\mathrm{Fe}^{2+}}$. From the statistical of acceptor level in semiconductor, we have

$$N_{\mathrm{Fe}^{2+}} = \frac{N_{\mathrm{Fe}}}{1 + \exp(E_A - E_F)/kT}. \tag{8}$$

From the above equations, the Fermi level in Fe-doped InP is

$$E_F = E_A + kT \ln\{(N_D + N_{\mathrm{ND}} - N_A)/[N_{\mathrm{Fe}} - (N_D + N_{\mathrm{ND}} - N_A)]\}$$

$$= E_A - kT \ln(\varphi - 1), \tag{9}$$

where $\varphi = N_{\mathrm{Fe}}/(N_D + N_{\mathrm{ND}} - N_A)$ is an indication of compensation. It can be seen from Eqs. (5), (6), and (9) that the resistivity of Fe-doped InP depends on the value of compensation φ. Since E_A is around 0.65 eV below conduction band,[22–24] $\varphi > 1$ is necessary to pin the Fermi level around $E_C - 0.65$ eV and give the SI property of the material.

The resistivity of Fe-doped InP is controlled by the initial doping concentration of Fe when InP is grown. From the above-mentioned analysis, such a concentration depends on the net donor concentration in as-grown InP. It has been proved that a native donor hydrogen vacancy complex exists in as-grown LEC InP materials. In undoped LEC InP, systematic discrepancies between the measured net donor impurity concentration and Hall electron concentration have been observed before.[29] The concentration of this complex defect on the top of the ingot is higher than that of ingot tail, as evidenced by the infrared absorption spectra shown in Fig. 4. Furthermore, the concentration of $V_{\mathrm{In}}H_4$ increases when Fe is doped in InP. Thus a high doping concentration of Fe, which is much higher than the net donor impurity concentration detected by mass spectroscopy, will be necessary for the preparation of SI InP. This has been a commonly observed phenomenon in Fe-doped LEC InP.

The $I-V$ results in Figs. 1 and 2 indicate that the trap filling process is not obvious in samples from the ingot top even for good Fe-doped SI LEC InP wafers. This indicates that most of the Fe atoms are in the Fe^{2+} state for the Fe-doped SI InP sample from ingot top. Thus compensation at the top is lighter than that at the tail of the Fe-doped InP ingot. Although the reason for this phenomenon can simply be ascribed to the segregation effect of an Fe atom, the in-

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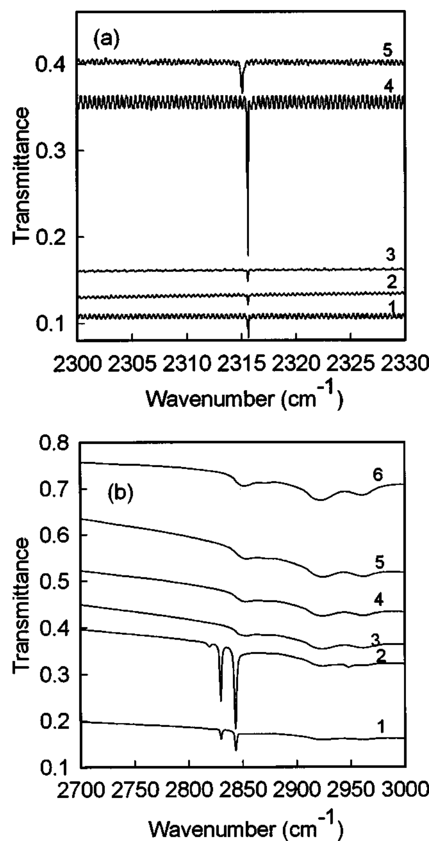

FIG. 4. Infrared transmission spectra of InP at 10 K. Curves in (a) are measured from samples of Fe-doped ingot (S-02) top (1,4), tail (5) and undoped ingot (S-01) top (2,3). Curves in (b) are transmission spectra of Fe^{2+} intracenter transitions measured from same samples of Fe-doped ingot top (1,2), tail (3) and undoped ingot top (4,5), tail (6). All curves have been separated intentionally for better clarity.

fluence of the donor defect $V_{In}H_4$ cannot be neglected since its concentration is much higher in the ingot top. When InP is doped with Fe, the resulting increase of concentration of this defect requires even a higher doping concentration of Fe to give the compensation level needed for forming a SI material. The significance of this result is, first, the doping concentration of Fe for SI InP is expected to decrease if the formation of $V_{In}H_4$ can be avoided; second, the longitudinal uniformity of Fe-doped SI InP is expected to improve since the concentration of $V_{In}H_4$ is lower in the ingot tail. The latter also implies that a moderate uniform doping concentration of Fe along an ingot can satisfy the compensation requirement for SI InP.

V. SUMMARY

A native hydrogen indium vacancy donor complex ($V_{In}H_4$) can be detected regularly in Fe-doped SI InP. By the comparison of the infrared absorption spectra of undoped and Fe-doped LEC InP, it is found that the concentration of $V_{In}H_4$ increases significantly when LEC InP is doped with Fe. The apparent compensation of Fe-doped SI InP is lighter in the ingot top although the concentration of Fe is much higher than that of the net donor impurities. The concentration of this $V_{In}H_4$ donor in the ingot top is higher than that in the ingot tail of Fe-doped SI InP. These facts give further evidence that $V_{In}H_4$ influences the compensation of LEC Fe-doped SI InP. The apparent light compensation in the ingot top is therefore ascribed to the existence of a high concentration of this special donor defect. It thus follows that any undesirable Fe doping concentration that we must have for achieving SI property may be minimized by controlling the growth condition so as to avoid the incorporation of this donor complex.

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