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# Optical and electrical properties of hydrogen-passivated gallium antimonide

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The effect of hydrogen-plasma passivation on the optical and electrical properties of gallium antimonide bulk single crystals is presented. Fundamental changes of the radiative recombination after hydrogenation in undoped, zinc-doped, tellurium-doped, and codoped (with Zn and Te) GaSb are reported. The results of optical measurements indicate that passivation of acceptors is more efficient than that of the donors and, in general, the passivation efficiency depends on the doping level. Passivation of deep nonradiative centers is reflected by the gain of photoluminescence intensity and decrease in deep-level transient spectroscopy peak height. Extended defects like grain boundaries and dislocations have also been found to be passivated. The thermal stability of the passivated deep level and extended defects is higher than that of the shallow level. The kinetics of thermally released hydrogen in the bulk has been studied by reverse-bias annealing experiments.

## I. INTRODUCTION

Hydrogen is known to passivate shallow and deep centers in bulk semiconductors by forming complexes. However, its behavior and passivation efficiency varies from material to material. Hydrogen passivation of various defects and impurities has been well studied in Si, GaAs, and InP.[1] It also passivates nonradiative recombination centers like dislocations and surface dangling bonds, which results in enhancing luminescence efficiency and mobility. The beneficial effects of hydrogenation have made this process an important step in device technology. Very little work has been done to investigate the passivating effect of hydrogen in GaSb,[2] a semiconductor which is now becoming important for the infrared sources and detectors in the 2-$\mu$m spectral regime.[3] In this paper, we present the effects of hydrogen passivation on the optical and electrical properties of bulk GaSb.

The knowledge of thermal stability of hydrogen-impurity complexes is of utmost importance in order to exploit the beneficial effects of passivation. However, the estimation of thermal stability becomes complicated due to the fact that thermally released hydrogen during annealing might get retrapped to form a hydrogen-impurity complex at the same site. Zundel and Weber[4] solved this problem by applying a reverse bias to the Schottky diodes during annealing. During the reverse-bias anneal (RBA), the thermally released hydrogen from the complex is drifted into the bulk due to the reverse-bias electric field and gets retrapped further into the bulk to form hydrogen-impurity complexes. This leads to a change in the net donor or acceptor concentration profile in the bulk. The RBA experiment also provides information regarding the charge state of hydrogen in different materials. Moreover, since most of the devices are operated with fields existing across them, it is important to know the conditions under which the active impurity profile changes in the bulk. With these motivations, we also performed a series of RBA and unbiased annealing experiments.

## II. EXPERIMENTAL DETAILS

The GaSb samples used in our studies were vertical Bridgman-grown single crystals.[5,6] Hydrogenation was carried out in a parallel-plate capacitor plasma reactor operating at a radio frequency of 13.56 MHz.[7] The rf power used was approximately 0.4 W/cm$^2$. The hydrogen pressure inside the reactor was maintained at 0.5 torr. It was found from our studies[8] that during hydrogenation, the bare surface of the sample was damaged, with the thickness of the damaged layer varying from 50–200 Å. Such a defect layer is highly leaky and is not suitable for any electrical measurements. Further, this defect layer introduces deep nonradiative recombination centers in the band gap resulting in a drastic decrease in the photoluminescence (PL) yield. Therefore, after hydrogenation, a 300-Å-thick layer was removed from the sample surfaces by controlled etching using an etchant consisting of potassium sodium tartrate ($C_4H_4O_6KNa\cdot 4H_2O$)/HCl/$H_2O_2$ in the ratio of (1:8:1).[9] The reference samples (as grown) were also treated the same way as the hydrogenated ones. To verify the thermal stability of the hydrogenated samples, they were heat treated in ultrapure hydrogen ambient. The samples were annealed face down on a GaSb substrate to minimize thermal degradation of the surfaces. Since annealing treatment reduces the overall PL efficiency of the sample (by introducing additional nonradiative centers), the annealing was simultaneously carried out on both the reference and the hydrogenated samples for comparison. The PL measurements were carried out using a MIDAC fourier transform photoluminescence spectrometer with a resolution of 0.5 meV. The room-temperature PL mea-

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surements were performed with the samples placed in a sealed He-filled chamber. For low-temperature measurements, the samples were freely suspended in liquid He at 4.2 K. An argon ion laser operating at 5145 Å was used as the excitation source with the power density of 3.5 $W/cm^2$ and 166 $mW/cm^2$ at 300 and 4.2 K, respectively. A liquid-nitrogen-cooled Ge photodetector was used for signal detection. For capacitance-voltage ($C$-$V$) and deep-level transient spectroscopy (DLTS) measurements, back ohmic contacts were prepared by evaporating a Au-Ge (88:12) eutectic mixture, followed by annealing at 300 °C for 2 min in an ultrapure $H_2$ atmosphere. The Schottky contacts were made by thermal evaporation of Au. All the electrical measurements were restricted only to $n$-GaSb as the Schottky barriers on $p$-GaSb are very low ($\sim 0.2$ eV) making it unsuitable for either $C$-$V$ or DLTS measurements. The reactivation kinetics of thermally released hydrogen from the hydrogen-acceptor or -donor complexes were studied by carrying out annealing experiments on Au/$n$-GaSb Schottky diodes for various durations with a constant reverse bias. Heat treatment was carried out in the temperature range of 100–250 °C. Since most of the Schottky diodes could withstand up to 2.5 V, the bias was kept constant at 2 V during all the RBA experiments. After annealing, the diodes were rapidly cooled by dipping the sample holder in liquid nitrogen while the bias was kept on. From previous studies on $p$-InP,[7] it is known that at low-bias conditions, the drifting of hydrogen is not effective and hence most of the thermally released hydrogen reassociate with the impurities at the same position. Therefore, we did not study the effect of RBA at low voltages. $C$-$V$ measurements (1 MHz) at room temperature were used to evaluate the depth profile of the net donor concentration after each reverse-bias annealing step. In order to see whether there is any degradation of the Schottky diodes on annealing, we annealed several Au/$n$-GaSb Schottky diodes till 300 °C. We did not find any significant change in the diode characteristics after annealing. In fact, Au contacts on GaSb deteriorate only after annealing above 350 °C for tens of hours.[10] Hall measurements were carried out at room temperature to evaluate the carrier concentration of various samples used in our studies.

## III. RESULTS AND DISCUSSION

In order to maximize the passivating effect, the substrate temperature and the duration of plasma exposure were varied from 150–250 °C and 2–6 h, respectively. Hydrogenation with a substrate temperature for 200 °C for 3 h was found to be optimum (as determined from room-temperature PL yield) and was subsequently used for further studies. Hydrogenation with a substrate temperature more than 200 °C or time exceeding 3 h did not improve the PL yield further. In general, after hydrogenation, the PL yield of all the samples increased due to reduction in nonradiative recombination centers. On annealing the hydrogenated samples, a decrease in PL intensity was observed due to reactivation of the passivated nonradiative centers. The annealing temperature and time were varied in the range of 200–350 °C and 2–30 min, respectively. As will be discussed later, the passivated shallow acceptors or donors could be retrieved completely by annealing the samples at 250 °C for 5 min, whereas only partial recovery of the deep level was possible even after annealing the samples at 300 °C for 30 min. Below we discuss the changes observed in the near-band-edge PL spectra on hydrogenation and after annealing the hydrogenated samples at 300 °C for 5 min. This annealing condition ensured that all the passivated shallow acceptors and donors were reactivated.

The room-temperature PL spectrum of an undoped GaSb (which is always $p$-type in nature with the native-acceptor concentration of $p \sim 10^{17}$ $cm^{-3}$ at 300 K) exhibited a single broad band-to-band transition around 720 meV as shown in Fig. 1. On hydrogenation, the intensity of this band increased approximately by 50 times. Even after annealing the hydrogenated sample at 300 °C for 5 min, the PL intensity did not come back to its original level implying that only a partial recovery of the passivated nonradiative centers had occurred. The low-temperature PL at 4.2 K is shown in Fig. 2. The dominant transition is at 777 meV corresponding to the neutral state of the doubly ionizable native-acceptor level.[11] This is accompanied by a LO-phonon replica at 746 meV, an excitonic peak at 792 meV, and a peak at 710 meV (approximately $\frac{1}{100}$th the intensity of the 777-meV peak) corresponding to the singly ionized state of the doubly ionizable native acceptors.[12] At 4.2 K, the 777-meV peak was found to shift towards higher energy by 2–3 meV per decade increase in laser power density. This indicates that the 777-meV peak corresponds to a donor-acceptor (D-A) pair transition. After hydrogenation, the intensity of the 777-meV peak increased by 25 times and the observed shift in the peak position was a 6-7 meV per decade increase in the excitation intensity. The large D-A pair peak shift may result from heavy compensation.[13] This is possible here only if hydrogen passivates acceptors more efficiently than donors. In addition to these

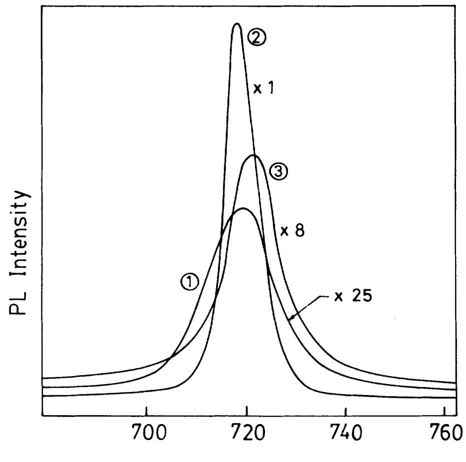

FIG. 1. PL spectra of (1) unhydrogenated, (2) hydrogenated, and (3) hydrogenated and annealed samples of undoped $p$-GaSb at 300 K.

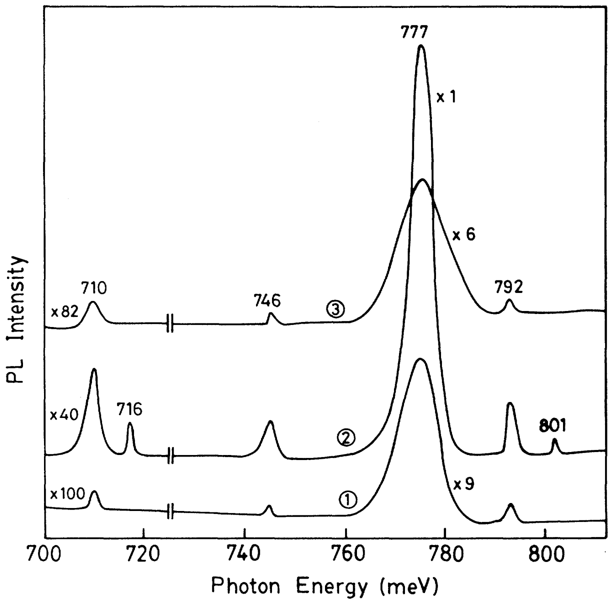

FIG. 2. PL spectra of (1) unhydrogenated, (2) hydrogenated, and (3) hydrogenated and annealed samples of undoped *p*-GaSb at 4.2 K.

changes, two new peaks at 716 and at 801 meV were observed. Nicholas *et al.*[14] reported these two peaks in their PL spectra of molecular-beam-epitaxy-grown GaSb with low native-acceptor concentration. They attributed the 716-meV peak to an exciton bound to the ionized native acceptor and the 801-meV peak to a donor-acceptor pair transition. The appearance of these peaks in our case after hydrogenation was simply due to the passivation of nonradiative channels for the photoexcited carriers and the reduction in high-background native-acceptor concentration. Annealing the hydrogenated samples leads to retrieval of the original native-acceptor concentration, due to which the two peaks disappear. However, the PL intensity does not come down to its original level indicating that the passivated nonradiative recombination centers are only partially reactivated (Fig. 2).

The PL spectra at 4.2 K of zinc-doped *p*-GaSb samples are shown in Fig. 3 for two carrier concentrations. The sample with low doping ($p = 2 \times 10^{17}$ cm$^{-3}$) [Fig. 3(a)] exhibits a broad peak at 775 meV along with a shoulder at 777 meV. Hydrogenation led to an overall increase in PL intensity by 70 times with the two peaks becoming better resolved. For the sample with slightly higher doping ($p = 4 \times 10^{17}$ cm$^{-3}$), a broad peak around 776 meV [Fig. 3(b)] is observed. On hydrogenation, a shoulder at 777 meV appears along with an overall PL intensity increase by approximately 100 times. This indicates that the Zn-acceptors are more effectively passivated than the residual native acceptors. This is due to the fact that the native acceptors are of defect origin (vacant gallium site $V_{\mathrm{Ga}}$ along with a gallium antisite $\mathrm{Ga_{Sb}}$) and not truly hydrogenlike acceptors like Zn. At the doping level of $10^{18}$ cm$^{-3}$, a broad symmetric band around 755 meV is observed. With increasing doping level, the peak shifts to the lower energy and the full width at half maximum (FWHM) increases. On hydrogenation, the FWHM decreases drastically and a peak shift of 15 meV towards the higher energy side is observed. This shows that the passivation efficiency becomes better with increasing Zn concentration in the crystal. The effect of annealing on the hydrogenated samples is also shown in this figure.

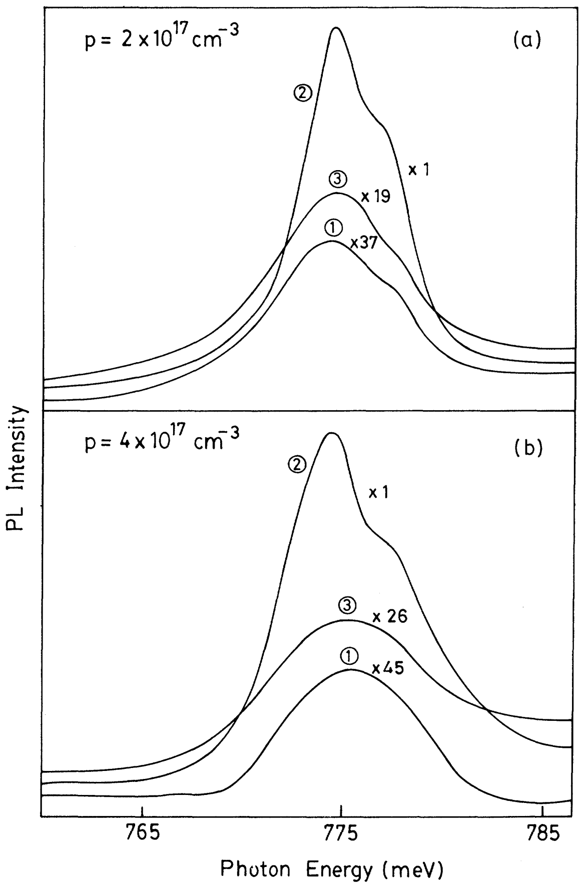

FIG. 3. PL spectra of (1) unhydrogenated, (2) hydrogenated, and (3) hydrogenated and annealed samples of Zn-doped *p*-GaSb at 4.2 K for (a) $p = 2 \times 10^{17}$ cm$^{-3}$ and (b) $p = 4 \times 10^{17}$ cm$^{-3}$.

The effect of hydrogenation on the tellurium-doped *n*-type samples is shown in Figs. 4(a) and 4(b) for two concentrations. Even though for both the samples, a shift in PL peak position (at 4.2 K) towards the lower energy side is observed after hydrogenation, the shift is 10 meV in case of the less doped sample ($n = 2 \times 10^{17}$ cm$^{-3}$) and 4 meV for the sample doped to $1.8 \times 10^{18}$ cm$^{-3}$. We attribute this peak shift to the passivation of donors by hydrogen. Systematic PL studies performed by us on *n*-GaSb have shown that with increasing carrier concentration, the PL peak shifts to the higher energy side. The large peak shift in the case of low-doped *n*-GaSb shows that the passivation efficiency is higher. This is in contrast to the case of *p*-GaSb wherein the passivation efficiency increases with the increase in doping concentration.

To directly measure the relative passivation efficiency of acceptors and donors, we also carried out PL measurements on GaSb codoped with Te and Zn to various con-

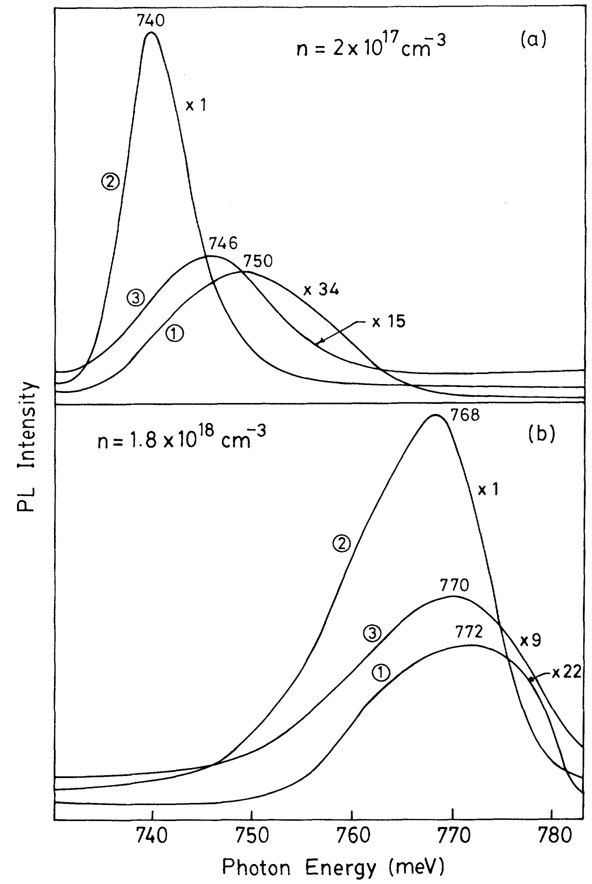

FIG. 4. PL spectra of (1) unhydrogenated, (2) hydrogenated, and (3) hydrogenated and annealed samples of Te-doped *n*-GaSb at 4.2 K for (a) $n = 2\times10^{17}$ cm$^{-3}$ and (b) $n = 1.8\times10^{18}$ cm$^{-3}$.

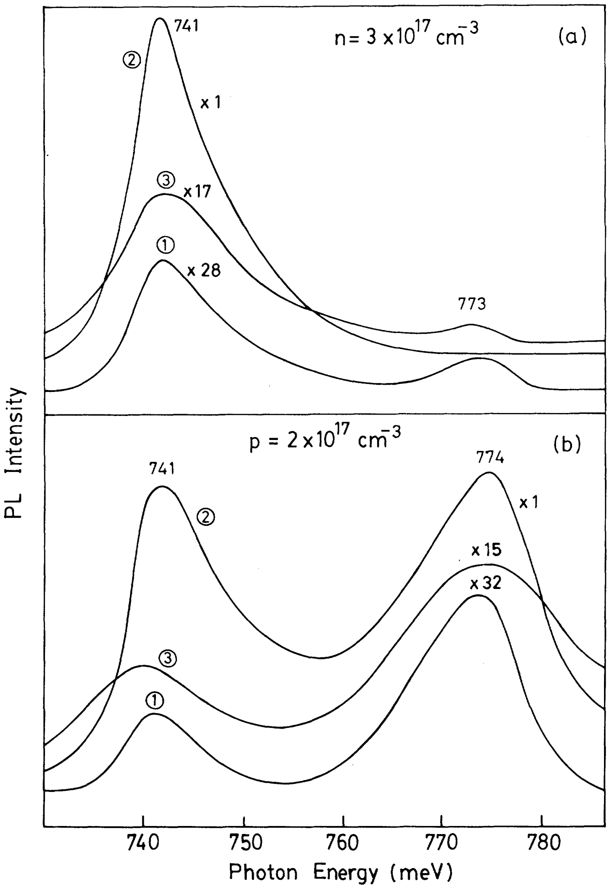

FIG. 5. PL spectra of (1) unhydrogenated, (2) hydrogenated, and (3) hydrogenated and annealed samples of Te-Zn codoped GaSb at 4.2 K for (a) $n = 3\times10^{17}$ cm$^{-3}$ and (b) $p = 2\times10^{17}$ cm$^{-3}$.

centrations to yield either *n*-type or *p*-type crystals. Figure 5(a) shows the PL spectra for an *n*-type GaSb before and after hydrogenation and after annealing. The unhydrogenated sample exhibits Te- and Zn-related peaks at 741 and 773 meV, respectively. On hydrogenation, the peak at 773 meV vanishes, indicating the passivation of acceptors to be more efficient than the donors. The PL spectrum of a *p*-type codoped sample is shown in Fig. 5(b). The intensity of the 774-meV peak was approximately 2.5 times higher than that of 741-meV peak. However, after hydrogenation the intensities of the two peaks became comparable. This once again shows the more efficient passivation of acceptors than donors.

The more efficient acceptor passivation can be explained if one takes into consideration that hydrogen is positively charged and hence attracted to the acceptors in Zn-doped *p*-GaSb, while in *n*-GaSb, it is primarily neutral hydrogen that does all the passivation and hence the efficiency of this process is much lower. Our results can be explained if one assumes that hydrogen in GaSb introduces a donor level close to the valence band edge and once it is crossed by the Fermi level in the hydrogenated sample, the passivation efficiency decreases. This is also true for lightly doped *p*-GaSb where the passivation efficiency is less than that of heavily doped ones. In InP, GaAs, and $In_xGa_{1-x}As$, the $H^+$-level position has been found to be close to the position of the so-called Hasegawa-Ohno hybrid orbital level, which determines the Fermi-level pinning at the semiconductor and/or metal interfaces and the Fermi-level position in heavily radiation-damaged semiconductors.[15] By analogy, the hydrogen donor level in GaSb should lie ~0.1 eV above the valence band edge, the position of the Fermi-level pinning at the surface of GaSb. It is worth noting that the $H^+$-level position in GaSb is within 0.2 eV of the position that would be calculated using the vacuum reference-level pinning model.[16]

Now we will discuss the results of our reverse-bias annealing experiments performed on Au/*n*-GaSb Schottky diodes at 220 °C for various durations with a constant reverse bias of 2 V. The depth profile of the net donor concentration, as determined from *C*-*V* measurements at room temperature after every reverse-bias annealing step, are shown in Fig. 6. Figures 6(a) and 6(b) correspond to tellurium-doped *n*-GaSb with a net donor concentration of $1.9\times10^{17}$ cm$^{-3}$ and $9\times10^{17}$ cm$^{-3}$, respectively. Figure 6(c) corresponds to Te-Zn-codoped *n*-GaSb with a net donor concentration of $2\times10^{17}$ cm$^{-3}$. Here the curves corresponding to the net-donor-concentration profile for

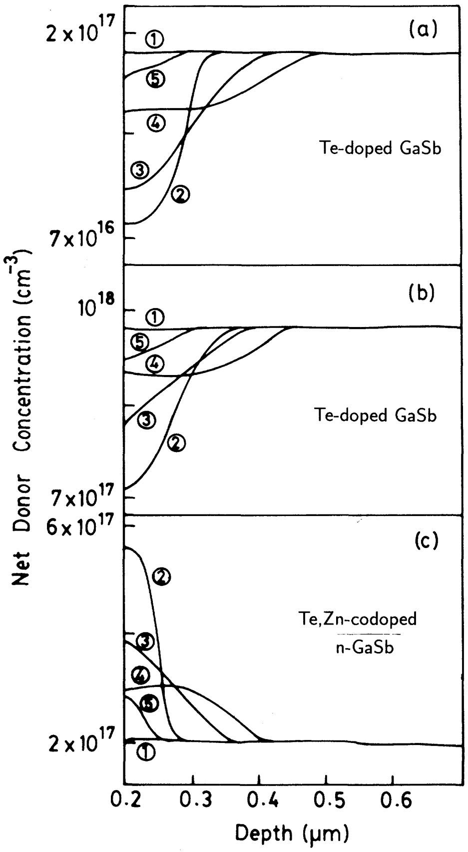

FIG. 6. Depth profile of net donor concentration for (a) and (b) Te-doped *n*-GaSb and (c) Te-Zn codoped *n*-GaSb. (1) As-grown sample, (2) hydrogenated sample, (3) after 40-min RBA, (4) after 60-min RBA, and (5) unbiased anneal for 15 min. (anneal temperature, 220 °C: reverse bias, 2V).

the unhydrogenated samples are labeled as (1) and those of the hydrogenated ones by (2). The net donor profile after RBA for various durations at 220 °C are shown by curves (3) and (4). Curve (5) is for the case where the diodes have been annealed at 220 °C for 15 min without applying any bias. It is clear from Figs. 6(a) and 6(b) that hydrogenation has led to donor passivation resulting in a decrease of net donor concentration near the surface. However, the changes are very low compared to those reported in GaAs and Si.[1] The RBA experiments indicate that hydrogen exists in negatively charged state in *n*-GaSb and is drifted into the bulk by the electric field. The increase in net donor concentration near the surface for the codoped *n*-GaSb [Fig. 6(c)] clearly indicates that the zinc acceptors are more efficiently passivated than the tellurium donors. The change is net donor profile after RBA [as shown by curves (3) and (4)] is attributed to the thermal dissociation of the hydrogen-dopant complexes and subsequent field-directed drift of charged hydrogen into the bulk followed by reassociation with the impurities inside. From curves (5), it can be seen that it is possible to regain the active donor concentration substantially by unbiased annealing experiments.

The result in Fig. 6(c) is striking, but rather odd. If the material is *n*-type, one would expect the H to be in a negative charge state and thus not interact with acceptors. Also it is known that in III-V compounds, minority dopants are not passivated by hydrogen. It is worth mentioning here that the dopant concentration of both tellurium and zinc in our crystal were of the order of $10^{17}$ cm$^{-3}$. The acceptor passivation can be explained by the charge-state conversion of $H^0$ or $H^-$ to $H^+$, a process known to occur in Si and GaAs.[1,17,18] Such a conversion is all the more favorable in the presence of a high concentration of minority carriers and in highly compensated samples. The $H^+$ would then passivate the zinc acceptors efficiently and that is what must be happening in our case. The charge-state conversion discussed above also results in considerable loss in the total amount of detected hydrogen during the annealing cycles, as seen in Fig. 6.

The carrier activation energies were calculated from the RBA experiments carried out at various temperatures in the range of 100–250 °C at a depth of 0.2 μm. The values were found to be 1.2 and 1.3 eV for the tellurium-doped and codoped *n*-GaSb, respectively. The dissociation frequencies were evaluated to be $2\times10^{13}$ s$^{-1}$ and $4\times10^{13}$ s$^{-1}$, respectively, for the above two cases. These values are comparable to the values of activation energies and dissociation frequencies observed in other III-V compounds.[19]

DLTS measurements were carried out on *n*-GaSb to see the effect of hydrogenation on the deep traps. A single electron trap with an activation energy of 312 meV and concentration close to $10^{15}$ cm$^{-3}$ was seen in the untreated samples as shown in Fig. 7.[20] The rate window and pulse width employed was 70.62 s$^{-1}$ and 10 ms, respectively. On hydrogenation, the DLTS peak height decreases by more than 3 times. Annealing the hydrogen passivated Au/*n*-GaSb Schottky diode at temperatures less than 300 °C for various times ranging from 2 to 3 min did not change the trap densities. The DLTS spectra for the hydrogenated sample after annealing at 300 °C for 30 min shows partial recovery of the deep trap (curve 3). It is worth noting that the passivated shallow acceptors or donors could be retrieved completely by annealing the

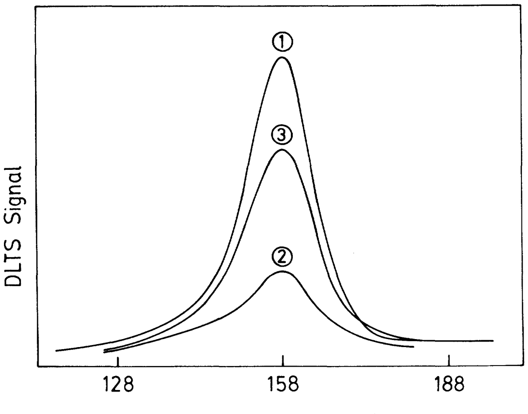

FIG. 7. DLTS spectra of (1) unhydrogenated, (2) hydrogenated, and (3) hydrogenated and annealed samples of Te-doped *n*-GaSb.

samples at 250 °C for 5 min, whereas the recovery of the deep level was possible only after annealing the samples above 300 °C. This shows the greater thermal stability of passivated deep level compared to shallow levels. Hence it is possible to regain the carrier concentration without losing the PL improvement (due to deep-level passivation). This is important for optoelectronic devices such as lasers wherein the PL efficiency can be increased without affecting the shallow-level concentration.

In addition to passivation of shallow and deep levels in GaSb, passivation of extended defects by hydrogen was also investigated. For this, we studied the *I-V* and *C-V* characteristics of Schottky diodes made from polycrystalline tellurium-doped *n*-GaSb. Reduction in reverse leakage current by more than an order of magnitude has been observed after hydrogenation. This reduction is attributed to the hydrogen passivation of grain boundaries. Such an effect has been observed in other III-V compound semiconductors also.[21] While very little change occurs in the forward *I-V* characteristics, the reverse breakdown voltage increases from approximately 1.4 to 2.5 V in most of our samples after hydrogenation. *C-V* measurements show a decrease in the net donor concentration from $3\times10^{17}$ cm$^{-3}$ to $9\times10^{16}$ cm$^{-3}$ near the surface after hydrogen passivation. A 250 °C anneal for 5 min restores the original carrier concentration, however the reverse breakdown voltage still remains higher than the as-grown samples. The increase in breakdown voltage can be attributed to hydrogen passivation of deep levels and extended defects that cause barrier lowering. The reverse breakdown voltage is found to decrease only after annealing the samples above 350 °C indicating higher thermal stability of the passivated extended defects.

## IV. CONCLUSIONS

In conclusion, we have evaluated the effect of hydrogenation on the optical and electrical properties of undoped and doped GaSb crystals. Passivation of shallow donors and acceptors were observed as a result of hydrogen treatment. The passivation efficiency was found to be maximum in heavily doped *p*-GaSb. Enhancement in PL yield due to passivation of deep level and other nonradiative centers was also observed. The thermal stability of passivated deep level and nonradiative centers was found to be much higher than the shallow level. Since there is very little change in electrical properties (carrier concentration), but drastic improvement in optical properties (large increase in PL yield) as a result of passivation, this can be of great importance in GaSb-based device technology.

## ACKNOWLEDGMENTS

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