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Electron irradiated liquid encapsulated Czochralski grown undoped gallium antimonide studied by positron lifetime spectroscopy and photoluminescence

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Abstract

Electron irradiated undoped liquid encapsulated Czochralski (LEC) grown GaSb samples were studied by positron lifetime spectroscopy (PLS) and photoluminescence (PL). In addition to the 315 ps component reported in the previous studies, another defect with a lifetime of 280 ps was also identified in the present electron irradiated samples. The bulk lifetime of the GaSb material was found to be 258 ps. The $V_{\text{Ga},280 \text{ ps}}$ and the $V_{\text{Ga},315 \text{ ps}}$ defects were associated with two independent Ga vacancy related defects having different microstructures. The well known 777 meV PL signal (usually band A) was also observed in the electron irradiated undoped GaSb samples. The band A intensity decreases with increasing electron irradiation dosage and it disappears after the 300 °C annealing regardless of the irradiation dosage. The origin of the band A signal is also discussed.

1. Introduction

Gallium antimonide is a III–V semiconductor having a narrow bandgap, a high carrier mobility and a small effective carrier mass. This material is thus a suitable material for fabricating high frequency electronic devices. Moreover, GaSb is the basic material for growing a variety of lattice matched optoelectronic materials working at the wavelengths of 0.8–4.3 μm .

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Detailed reviews on the growth, the properties and the device fabrication of this material are given in Milines and Polyakov [1] and Duta and Bhat [2]. Unintentionally doped melt grown GaSb is usually p type and has a hole concentration of 10^{16} – 10^{17} cm^{-3} . The residual acceptor has been reported to be a doubly ionizable [3] native defect and be related to Ga in excess [4–6]. This residual acceptor is usually regarded as the $V_{\text{Ga}}\text{Ga}_{\text{Sb}}$ complex [7–9], although direct evidence for this correlation is not yet available. Photoluminescence (PL) and cathodoluminescence (CL) measurements on undoped GaSb usually reveal a 777 meV luminescence signal (called band A) and this signal is usually related to the conduction band (CB) or the shallow donor (SD) to $V_{\text{Ga}}\text{Ga}_{\text{Sb}}$ acceptor transition [8, 10–16]. However, the results of some studies have disagreed with this assignment. Rühle and Bimberg [17] have studied the Zeeman effect of the excitons bound to the residual acceptor revealing a tetrahedral symmetry for this site, thus contradicting the idea that the residual acceptor is a $V_{\text{Ga}}\text{Ga}_{\text{Sb}}$ defect. By performing the first principles calculation, Hakala *et al* [18] have studied the native defects V_{Ga} , V_{Sb} , $V_{\text{Ga}}\text{Ga}_{\text{Sb}}$ and Ga_{Sb} in GaSb and concluded that Ga_{Sb} must be the most important acceptor in undoped GaSb because of its low formation energy and ionization energy. Based on thermodynamical considerations, Shaw [19] has suggested that the Ga_{Sb} defect must be the residual acceptor of undoped GaSb.

Positron lifetime spectroscopy has been extensively used to study neutral and negatively charged vacancy type defects in semiconductors in the past 20 years [20–23]. The principle of the positron lifetime technique is that neutral or negative vacancies present an attractive potential well to the positrons in the solid. Implanted positrons after thermalizing undergo diffusion and can readily be trapped by such potential wells. As the positron annihilation rate depends on the electronic density at which the positron annihilates, positrons annihilating at different vacancy sites have a lifetime specified by the vacancy type. Apart from defect identity, careful analysis of lifetime data can also give defect concentration and if taken as a function of temperature the charge state and the ionization energy of the defect. Positron annihilation spectroscopic studies on GaSb [24–30] are relatively fewer than other semiconductor materials like Si, GaAs or SiC. Mahony *et al* [24] observed a positron trap having a characteristic lifetime of 300 ps which they attributed to a monovacancy. Dannefaer *et al* [25] reported a 297 ps lifetime component and a bulk lifetime value of 253 ps in the Te-doped GaSb sample. In the study of Puska *et al* [26], lifetime values of 260, 287, 307 and 350 ps for GaSb bulk, V_{Ga} , V_{Sb} and $V_{\text{Sb}}V_{\text{Ga}}$ were respectively reported.

In our previous positron lifetime studies, a component with positron lifetime of ~ 315 ps was observed in non-irradiated heavily Zn doped and undoped GaSb materials [28, 29], whereas another lifetime component with $\tau_d \sim 280$ ps was identified in the electron irradiated undoped GaSb samples [30]. These two lifetime components were attributed to two independent different V_{Ga} related defects having different microstructures. The $V_{\text{Ga},315 \text{ ps}}$ component was found to anneal out at about 300 °C [28–30] while the $V_{\text{Ga},280 \text{ ps}}$ persisted after the 500 °C annealing [30]. For non-irradiated undoped samples annealed at 300 °C or above, the average lifetimes had constant value of 266–269 ps and the spectra were well fitted by a single component [29]. This value was thus attributed to the bulk lifetime of GaSb [28, 29], although it is significantly larger than the value reported by Dannefaer *et al* [25] (253 ps) and Puska *et al* [26] (260 ps). Correlation in the annealing behaviours of the band A PL signal and the 315 ps positron lifetime signal has been well established [29], suggesting that the band A signal is related to the 315 ps V_{Ga} related defect. Temperature dependent Hall (TDH) measurements showed that the most important acceptor responsible for the material p-type nature is at the position of $\sim E_{\text{v}} + 34$ meV [30]. Moreover, by considering the annealing behaviours and the ionization levels of the 34 meV acceptor and the two V_{Ga} related defects, it was concluded that the two V_{Ga} related defects were not related to the 34 meV acceptor [30].

In the present study, systematic positron lifetime and PL measurements have been employed to study the electron irradiated undoped GaSb materials. Particular attention was given to the correlation between the band A PL signal in the electron irradiated undoped samples and the V_{Ga} related defects, and the controversial reported values of GaSb bulk lifetime.

2. Experimental details

The raw samples used in the present study were cut from the two liquid encapsulated Czochralski (LEC) grown undoped GaSb wafers 042Un and 342Un having hole concentrations of $p \sim 2.5 \times 10^{17}$ and $2.0 \times 10^{17} \text{ cm}^{-3}$, respectively. The samples were irradiated with electrons having energy of 1.7 MeV while keeping their temperature below 20 °C. Each of the annealing steps was performed in the forming gas atmosphere (80% N_2 and 20% H_2) for a period of 30 min. Positron lifetime measurements were carried out with two conventional fast-fast positron lifetime spectrometers having resolutions of $\text{fwhm} = 200$ ps. The positron source used was either a 30 μCi ^{22}Na radioisotope encapsulated by two pieces of kapton foil or a directly deposited 5 μCi $^{22}\text{NaCl}$. The directly deposited low activity source was used to check the correctness of the source correction of the foil source. The sample-source ensemble was then loaded into an Oxford Instrument 10 K closed cycle He refrigerator system. Each of the positron lifetime spectra contained 4 million annihilation events. The lifetime spectra were analysed by the POSITRONFIT code [31], which fits the spectra to the expression $\sum I_i \exp(-t/\tau_i)$ taking into account background subtraction, source correction and instrumental resolution. In the PL measurements, the excitation light source used was the 514.5 nm line coming from a 500 mW Ar laser. Double monochromators each with focal length of 0.25 m were used to resolve the emitted light. The PL measurements were carried out at $T = 10$ K with the sample installed in an Oxford closed cycle He refrigerator. The emitted light was detected with a liquid nitrogen cooled InSb IR detector coupled to a lock-in amplifier. An 800 nm long pass filter was inserted in order to block the second order signal from entering the detector.

3. Results and analysis

3.1. Positron lifetime results

Room temperature positron lifetime measurements were conducted on the non-irradiated and the electron irradiated undoped GaSb samples annealed at different temperatures. The cross symbol in figure 1(a) represents the average positron lifetime as a function of the annealing temperature for the non-irradiated 342Un sample. For the non-irradiated sample, an annealing stage is seen at 270–370 °C, in which the average lifetime dropped from 274 to 265 ps. This annealing stage has been reported in our previous positron lifetime studies on non-irradiated undoped GaSb in which it was found to correspond with the annealing out of a V_{Ga} related defect having a characteristic lifetime of ~ 315 ps [28, 29]. In [29], it was also reported that at annealing temperatures higher than 300 °C the spectra contained only a single component with lifetime value of 265–268 ps. This value was attributed to the bulk lifetime of GaSb.

The circles in figure 1(a) show the positron average lifetime as a function of the annealing temperature for the 1.7 MeV 10^{16} cm^{-2} electron irradiated 342Un sample. Surprisingly, after the electron irradiation, the average lifetime does not increase, as may be expected when introducing new defects, but decreases as compared to the non-irradiated sample. Moreover, the annealing behaviour of this irradiated sample is different from that of the non-irradiated sample. Instead of having the abrupt drop at about 300 °C, the average lifetime increases from 268 to 272 ps at 300 °C and only at higher temperatures does it decrease to about 260 ps, a value

which is significantly smaller than our previously assigned GaSb bulk lifetime ~ 265 ps [28, 29]. This observation suggests that the 265 ps value is not the value of the GaSb bulk lifetime.

In order to check whether these features of the electron irradiated (1.7 MeV, 10^{16} cm $^{-3}$) 342Un samples shown in figure 1(a) are real and general features induced by electron irradiation, we irradiated a second sample of undoped GaSb wafer (042Un) with a lower 1.7 MeV electron dosage 10^{15} cm $^{-3}$. The average lifetime variation is shown in figure 1(b). Similar to the high dosage sample, the average lifetime drops after electron irradiation. Moreover, a similar peaking phenomenon occurs in the average lifetime, although in the slightly higher annealing temperature range of 300 – 400 °C. However, for this low dosage sample, after high temperature annealing, the high temperature average lifetime decreased to ~ 266 ps, which is the same value as found for the non-irradiated sample.

We have performed fitting to the lifetime spectra of the electron irradiated (1.7 MeV 10^{16} cm $^{-2}$) sample. It was found that a single defect component model could not represent the data throughout the whole annealing temperature range. Specifically, for the spectra annealed at 400 °C or above, a single-defect-component model could give a reasonably good fit to the experimental spectra with a characteristic defect lifetime of ~ 280 ps. In this higher temperature range, the modelled bulk lifetime is computed as 258 ± 1 ps using the single-defect simple trapping model formula: $1/\tau_{b, \text{mod}} = \lambda_{b, \text{mod}} = I_1\lambda_1 + I_2\lambda_2$ [20, 21, 23]. This bulk lifetime being close to the experimental and theoretical values of 253 and 260 ps reported by Dannefer *et al* [25] and Puska *et al* [26] respectively supports the picture of a single type of defect of trapping positrons in the irradiated samples annealed at 400 °C or above.

With the bulk lifetime value of $\tau_b = 258$ ps, the τ_d/τ_b ratios for the 280 ps and the 315 ps components are 1.09 and 1.22 respectively, implying that both these components are associated with monovacancies. As the sample is of p type and the V_{Sb} defect is a donor having ionization levels close to the conduction band, V_{Sb} is expected to be positively charged and unable to trap positrons. This implies that the currently observed 280 ps component ($V_{\text{Ga}, 280 \text{ ps}}$) and the 315 ps component ($V_{\text{Ga}, 315 \text{ ps}}$) are most probably associated with V_{Ga} related defects. Here two possibilities emerge. The first is that two V_{Ga} related defects exist having two different characteristic lifetimes. The second is that the two different positron lifetimes arise from a charge induced V_{Ga} vacancy relaxation, resulting from a change in the Fermi level position. To investigate the latter possibility, room temperature Hall measurements were performed on the irradiated samples annealed at different temperatures. The derived Fermi level position as a function of the annealing temperature is shown in figure 2(a). It is seen that the Fermi level position of the electron irradiated sample moves significantly towards the valence band with increasing annealing temperature and thus the charge state occupancy of the Ga vacancy defect may also be expected to change. However, in our previous study of heavily Zn doped GaSb material [28], the 315 ps V_{Ga} component also annealed at 300 °C, with no movement of the Fermi level. Moreover, in the present (shown in the discussion of next paragraph and the corresponding data in figure 2(c)) and previous positron lifetime studies on non-irradiated undoped GaSb [29], the 315 ps component annealed at 300 °C without any significant change in Fermi level position (hole concentration). These observations imply that the extinction of the 315 ps component is simply due to the thermal annealing of the defect rather than any change of its charge state occupancy. It is thus concluded that the two lifetime components 280 and 315 ps are related to two V_{Ga} related defect complexes having different microstructures.

Since the 315 ps component was unambiguously found in the non-irradiated samples annealed at 300 °C and below, it is natural to assume it probably also exists in the irradiated sample annealed at 300 °C or below. The 280 ps component was clearly identified in the irradiated sample annealed at temperatures higher than 300 °C. It is thus of interest to check whether there exists a coexistence of the two components, 280 and 315 ps, in the irradiated

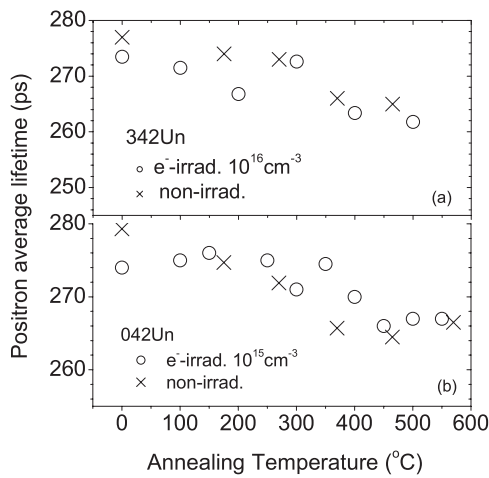


Figure 1. (a) Positron average lifetime as a function of the annealing temperature for the 342Un undoped GaSb sample. The cross and circle are respectively the non-irradiated and the electron irradiated ($1.7 \text{ MeV } 10^{16} \text{ cm}^{-2}$) samples. (b) Positron average lifetime as a function of the annealing temperature for the 042Un undoped GaSb sample. The cross and circle are respectively the non-irradiated and the electron irradiated ($1.7 \text{ MeV } 10^{15} \text{ cm}^{-2}$) samples. The statistical error of the average lifetime is less than 1 ps.

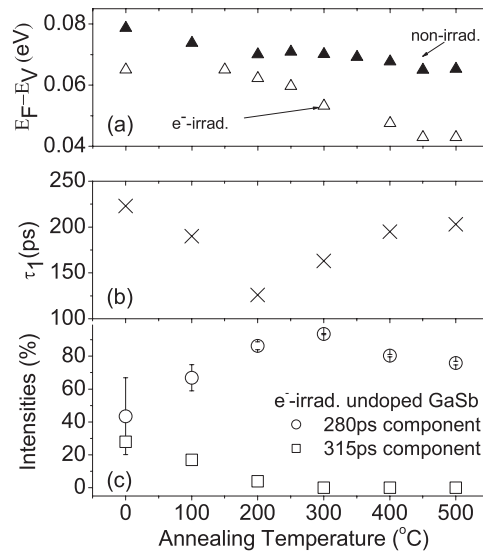


Figure 2. (a) Fermi level position as a function of the annealing temperature for the non-irradiated (solid triangle) and the electron irradiated (open triangle) undoped GaSb samples. (b) The fitted τ_1 and (c) the fitted defect component intensities as a function of the annealing temperature for the electron irradiated undoped GaSb samples. The characteristic lifetimes of the defect components were fixed at 280 and 315 ps during the fitting process. The electron irradiation process has an electron energy and dosage of 1.7 MeV and 10^{16} cm^{-2} .

samples annealed at 300°C or below. As fitting the lifetime spectra with three freely fitting components is difficult, the lifetimes of the two defect components were fixed at 280 and 315 ps respectively. Reasonably good fits were obtained with lifetime τ_1 and intensities of the two components as shown in figures 2(b) and (c). From figure 2(c), it is clearly observed that the $V_{\text{Ga},315 \text{ ps}}$ component disappears after the 300°C annealing. The intensity of the $V_{\text{Ga},280 \text{ ps}}$ component first increases with annealing temperature, reaches a maximum at about 300°C , and then slightly decreases with further increase in the annealing temperature.

The fact that the $V_{\text{Ga},280 \text{ ps}}$ component exists in the irradiated sample annealed at temperatures of 300°C and below could also explain the interesting feature that the positron average lifetime of the as-electron-irradiated sample is smaller than the as-grown sample. It is noted that the single-defect trapping model (with defect lifetime $\sim 315 \text{ ps}$) gives a good fit to the as-grown sample spectrum, but not to the as-irradiated sample. These observations suggest that the $V_{\text{Ga},280 \text{ ps}}$ does not exist (or exists in negligible amounts) in the as-grown sample and that this component has been induced by the electron irradiation process. The irradiation introduced 280 ps component would then act as a competitor with the 315 ps component in trapping positrons. The decrease of the positron average lifetime after electron irradiation would then be a consequence of a significant fraction of positrons annihilating in the 280 ps state.

3.2. Photoluminescence results

The PL spectra of the as-irradiated undoped GaSb samples with different irradiation dosages carried out at 10 K are shown in figure 3. The band A signal ($\sim 777 \text{ meV}$) that usually

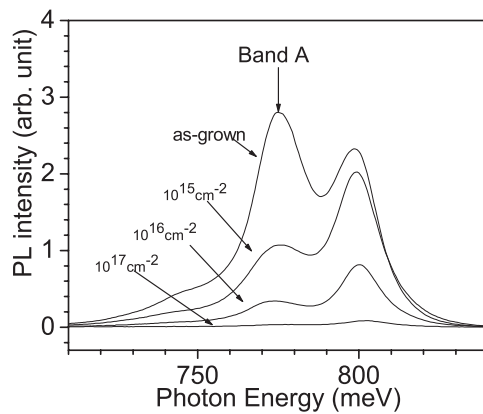


Figure 3. PL spectra of the electron irradiated (1.7 MeV) undoped GaSb samples with different dosages.

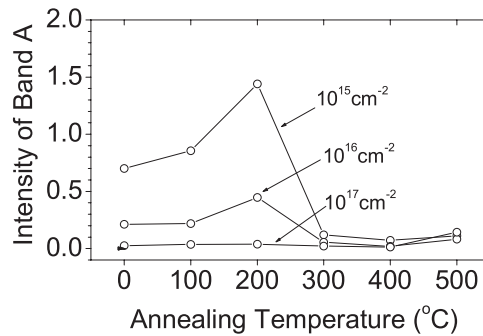


Figure 4. Intensity of the band A PL signals as a function of the annealing temperature for electron irradiated undoped GaSb samples with different irradiation dosages.

appears in p-type GaSb PL spectra, as well as the other two peaks at ~ 745 and ~ 800 meV, are readily seen. The 777 meV, the 745 meV and the 800 meV peak are usually attributed to the donor/conduction-band- $V_{\text{Ga}}\text{GaSb}$ transition, the LO phonon replica of 777 meV and the exciton bound to $V_{\text{Ga}}\text{GaSb}$. Their intensity decreases with increasing irradiation dosage, becoming negligible for the 10^{17} cm^{-2} dosage sample. The isochronal annealing dependence of the band A intensity for samples irradiated with different dosages is shown in figure 4. It is clearly seen that the band A signal anneals at about 300°C , in the same way that it does in the non-irradiated GaSb samples.

4. Discussion

In our previous positron lifetime study on non-irradiated undoped GaSb samples [29], the 315 ps lifetime component was identified in samples annealed at 300°C and below, while for samples annealed at above 300°C the lifetime spectra appeared to exhibit a single component, 267 ± 1 ps, that was tentatively attributed to the GaSb bulk lifetime [29]. The present lifetime study carried out with a better resolving lifetime spectrometer has revealed that this 267 ps component is in fact a composite of two closely spaced lifetimes (i.e. the τ_1 and the 280 ps components). In a previous study, positron lifetime measurements on a 500°C annealed electron irradiated sample at temperatures from 10 to 300 K (which corresponds to $E_{\text{F}} = E_{\text{V}} + 10 \text{ meV}$ to $E_{\text{V}} + 79 \text{ meV}$) were performed [30]. All the spectra were well described by the two-component model with constant long lifetime intensity ($I_2 \sim 76\%$) and constant defect lifetime ($\tau_2 \sim 280$ ps). This constancy over measurement temperatures indicates that the trapping centre is neutral in charge [20, 23] and it could not have an ionization level in the range of 10–79 meV above the valance band.

An important observation of the present study is that the annealing behaviours of the band A PL signal (as well as the 745 meV and the 800 meV PL lines) and the $V_{\text{Ga},315 \text{ ps}}$ defect observed in the electron irradiated undoped samples are similar, both annealing at 300°C . This correlation, which was also found on non-irradiated material [29], was interpreted as the involvement of the $V_{\text{Ga},315 \text{ ps}}$ defect in the optical transition responsible for the band A signal. This conclusion coincides with the conventional model that the band A line originates

from transition between a shallow donor (SD) or the conduction band (CB) and the $V_{Ga}Ga_{Sb}$ acceptor; the 745 meV line is the phonon replica of the band A line, and the 800 meV is related to the exciton bound to the $V_{Ga}Ga_{Sb}$ [10–16]. This being the case, the acceptor must have an energy level equal to or below 34 meV since the bandgap of the GaSb is 811 meV at 2 K. With the use of first principles calculation, Hakala *et al* [18] have studied the intrinsic defects in GaSb. They found that the Ga_{Sb} defect has an ionization level at $E_V + 40$ meV and it should be the most abundant of intrinsic defects. Moreover, the $V_{Ga}Ga_{Sb}$ or the V_{Ga} defects do not have any ionization state in the vicinity of $E_V + 34$ meV. Ling *et al* [30] have studied the ionization of the $V_{Ga,280\text{ ps}}$ and the $V_{Ga,315\text{ ps}}$ defects by performing the temperature dependent positron lifetime measurements, and did not find any ionization levels corresponding to these two V_{Ga} related defects located close to $E_V + 34$ meV. This implies that the expected value of the acceptor responsible for the band A PL line does not match the reported ionization energies of the $V_{Ga}Ga_{Sb}$ defect [11] or those of the two V_{Ga} related defects reported in [30]. However, it is in excellent agreement with the dominant acceptor Ga_{Sb} antisite [11].

If the band A signal (and also its phonon replica ~ 745 meV) is due to the Ga_{Sb} defect rather than any V_{Ga} complex, it would be interesting to investigate the cause behind the observed annealing correlation between the $V_{Ga,315\text{ ps}}$ defect and the band A signal. One possibility is that a PL quencher can be generated by the annealing out of the $V_{Ga,315\text{ ps}}$ defect. It is noted that in the electron irradiated samples the intensity of $V_{Ga,280\text{ ps}}$ reaches the maximum as $V_{Ga,315\text{ ps}}$ anneals out at annealing temperatures $\sim 300^\circ\text{C}$. This would suggest that the $V_{Ga,280\text{ ps}}$ defect is acting as a non-radiative PL quencher and agrees well with the observation that the band A intensity decreases with increasing electron irradiation dosage (see figures 3 and 4). However, this suggestion is speculative and requires further investigations.

Disagreement also exists between the understanding of the 800 meV PL lines and the Ga vacancy related defect derived from different studies. Conventionally, the 800 meV was thought to be related to the exciton bound to the $V_{Ga}Ga_{Sb}$ defect. The correlated anneal out of the 800 meV PL line and the $V_{Ga,315\text{ ps}}$ signal at 300°C in [29] and in the present study thus suggests that the $V_{Ga,315\text{ ps}}$ signal is related to the $V_{Ga}Ga_{Sb}$ defect. However, our previous temperature dependent positron lifetime study on the as-grown undoped GaSb sample showed that the $V_{Ga,315\text{ ps}}$ defect had an ionization level lying in the range of 70–85 meV above the valence band, which did not agree with Hakala *et al*'s calculated results that the stable ionization levels of $V_{Ga}Ga_{Sb}$ are at $E(-/2-) = E_V + 280$ meV and $E(2-/3-) = E_V + 520$ meV. Further works are also required to resolve this uncertainty.

5. Conclusion

Positron lifetime spectroscopy and PL have been carried out to study the electron irradiated undoped GaSb. In addition to the previously observed 315 ps component in non-irradiated undoped samples, a defect component having a lifetime of ~ 280 ps was identified in electron irradiated undoped samples. These two defects were attributed to two independent Ga vacancy related defects having different microstructures. The positron lifetime of the bulk GaSb has been corrected to be 258 ps, in contrast to the value of 266–268 ps reported in our previous studies. The 777 meV PL signal observed in the electron irradiated samples decreases with increasing electron irradiation dosage and it anneals out at 300°C . This phenomenon has tentatively been attributed to possible action of the $V_{Ga,280\text{ ps}}$ defect in quenching the luminescence.

Acknowledgments

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