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Radiation-induced defects in GaN bulk grown by halide vapor phase epitaxy

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Defects induced by electron irradiation in thick free-standing GaN layers grown by halide vapor phase epitaxy were studied by deep level transient spectroscopy. In as-grown materials, six electron traps, labeled D2 ($E_C-0.24$ eV), D3 ($E_C-0.60$ eV), D4 ($E_C-0.69$ eV), D5 ($E_C-0.96$ eV), D7 ($E_C-1.19$ eV), and D8, were observed. After 2 MeV electron irradiation at a fluence of $1 \times 10^{14} \text{ cm}^{-2}$, three deep electron traps, labeled D1 ($E_C-0.12$ eV), D5I ($E_C-0.89$ eV), and D6 ($E_C-1.14$ eV), were detected. The trap D1 has previously been reported and considered as being related to the nitrogen vacancy. From the annealing behavior and a high introduction rate, the D5I and D6 centers are suggested to be related to primary intrinsic defects. © 2014 AIP Publishing LLC.

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With its outstanding optical and electrical properties such as a direct wide bandgap (3.44 eV at 300 K), high breakdown field ($\sim 5 \times 10^6 \text{ V cm}^{-1}$ at 300 K), and high electron mobility ($\leq 1000 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ at 300 K),¹ GaN has long been considered as the most promising material for optoelectronics and high-frequency power devices. In recent years, a rapid development of GaN-based devices has been made. However, due to the lack of native substrates, most of GaN-based electronics and optoelectronics used foreign substrates, such as sapphire and SiC, which give rise to high dislocation densities, limiting and worsening the performance of devices. Using native substrates to reduce dislocation density is therefore highly desired. GaN bulk grown by halide vapor phase epitaxy (HVPE)^{2,3} has become available only recently and knowledge on defects in the material is still rather poor. Identifying deep level defects and understanding their electronic structure are important for defect control and, hence, the success of device applications.

Electron irradiation is often used to create intrinsic defects in crystals in a controlled manner so that their deep energy levels can be conventionally studied by capacitance transient techniques such as deep level transient spectroscopy (DLTS). Several deep level defects induced by electron irradiation in GaN have been previously reported.^{4–9} Fang *et al.* found a deep defect level at 0.18 eV below the conduction band in electron-irradiated GaN, but its origin has not conclusively been identified.⁵ Polenta *et al.*⁹ did a detailed study of electron irradiated metalorganic chemical vapor deposition (MOCVD) grown n-type GaN Schottky diodes in the temperature range of 80–400 K, revealing two severely overlapped DLTS peaks with the thermal activation energies of $E_C-0.06$ eV and $E_C-0.11$ eV, respectively. Another observation by Goodman *et al.* suggested that there are at least three defects whose DLTS spectra overlapped with each other to form a broad peak with different activation energies (0.06 eV, 0.10 eV, 0.20 eV).⁷

In this letter, we present results from DLTS studies of thick HVPE-grown GaN layers irradiated with electrons at an energy of 2 MeV and a fluence of $1 \times 10^{14} \text{ cm}^{-2}$. DLTS

centers in as-grown and irradiated GaN, and their annealing behavior at high temperatures (up to 1000 °C) are presented. It is known that the threading dislocation density (TDD) can influence the electrical properties and concentration of traps,^{10,11} therefore, we have used thick (~ 0.4 mm) n-type GaN layers grown by HVPE^{2,3} with low TDD ($\sim 5 \times 10^6 \text{ cm}^{-2}$ as determined by cathodoluminescence) for the study. The Ga-face was polished for improving the rectifying properties of the Schottky contacts. 1000 Å thick Au dots with a diameter of 1.2 mm were used as Schottky contacts. The result from current versus voltage (IV) measurement showed a good rectifying property. For Ohmic contacts, silver paint was used on the backside of the samples. The samples were then irradiated with 2-MeV electrons at a fluence of $1 \times 10^{14} \text{ cm}^{-2}$. In order to avoid heating, the samples were put on a water-cooled copper plate during electron irradiation. The Schottky contacts were made prior the electron irradiation in order to avoid unintentional annealing of radiation-induced defects due to heating during the evaporating process. IV and capacitance versus voltage (CV) measurements were performed to study the influence of irradiation on the rectifying characteristic and the free-carrier concentration in the samples. Diodes with a leakage current less than 10 μA at a reverse bias of -10 V were used for the DLTS measurements. Finally, the samples were annealed at the high temperature of 1000 °C in N_2 ambient for 30 min.

The DLTS data were collected by a homemade system using a 1 MHz Boonton 7200 capacitance bridge and a 100 MHz Tabor 8024 pulse generator. In our measurement, Schottky diodes were under a reverse bias of -10 V, the filling pulse amplitude was 10 V, the filling pulse width was 10 ms, and the temperature was in the range of 77–700 K.

IV measurements show that the leakage current of Schottky diodes at room temperature before irradiation and after irradiation was about -2 nA at the reverse bias of -10 V. The IV measurement after irradiation is shown in the inset of Fig. 1. Thus, the leakage current was not affected by the irradiation. The depth profile from CV measurements after irradiation is slightly different in comparison with that in

FIG. 1. Depth profile of net donor concentration of the Schottky diode on n-GaN before irradiation (square) and after irradiation (circle). The inset shows IV characteristics of the Schottky diode after irradiation.

the as-grown sample (see Fig. 1). However, the change is very small and the average net donor concentration is approximately $1.4 \times 10^{16} \text{ cm}^{-3}$ in both cases.

Fig. 2 illustrates the DLTS spectra of the sample measured before electron irradiation (Fig. 2(a)), after irradiation (Fig. 2(b)), and after irradiation and annealing at 1000°C for 30 min (Fig. 2(d)). The DLTS peak amplitudes are proportional to the trap concentration which can be estimated by the expression¹²

$$N_T = 2 \times \frac{S(T) \times (N_d - N_a)}{C_0} \times \frac{r^{x/(r-1)}}{(1-r)}, \quad (1)$$

where $S(T)$ is the amplitude of the DLTS peak, $N_d - N_a$ is the net donor concentration, and C_0 is the background capacitance. The factor r is the ratio t_2/t_1 where t_1 and t_2 are the capacitance sampling times. In order to take into account a small change of $N_d - N_a$ and C_0 in the different cases, the spectra are scaled according to Eq. (1) where r is equal to 8 by choosing $t_2/t_1 = 2$. After rescaling of the DLTS spectra, the absolute value of the peak amplitude corresponds to the trap concentration. By taking the difference between the scaled signal before and after irradiation, the increase of trap

FIG. 2. The DLTS spectra after rescaling according to Eq. (1): (a) as-grown sample; (b) after electron-irradiated by 2 MeV electron with a fluence of $1 \times 10^{14} \text{ cm}^{-2}$; (c) the spectrum in irradiated sample after subtracting the signal measured before irradiation; and (d) the spectrum after annealing at 1000°C for 30 min in N_2 gas flow.

concentration (ΔN_T) were determined, see Fig. 2(c). Before irradiation, we observe six traps labelled D2, D3, D4, D5, D7, and D8. The traps D2 and D3 form two distinct peaks while the traps D4, D5, D7, and D8 give rise to a broad band in the temperature range 340–550 K where peak D7 dominates the spectra. After electron irradiation, three traps, labelled D1, D5I, and D6, were observed. Peak D1 was not observed before irradiation. However, we cannot rule out that traps D5I and D6 are present before irradiation since they may be immersed by the strong D5 and D7 signal. Peak D1 is partly overlapping with peak D2, while peak D6 together with peak D5I dominates the spectra after irradiation. Most interestingly, we have observed that peak D8 was absent after irradiation.

In order to study the observed DLTS peaks in more detail, the difference between the DLTS signals before and after irradiation were evaluated, see Fig. 2(c). We observed that the concentration of traps D2, D3, and D4 does not change while the concentration of traps D5I and D6 increase significantly. As can be seen in the figure, the right shoulder of the broad peak does not show any signal from the trap D7 suggesting that the concentration of trap D7 is not affected by irradiation.

After annealing at 1000°C for 30 min in nitrogen ambient (Fig. 2(d)), the traps D2, D3, D4, and D5 can still be observed, whereas traps D1, D5I, D6, and D7 are completely annealed out and in addition, peak D8, which was observed before irradiation, reappears. An interesting observation is a significant reduction in the concentration of the traps D5I and D6 after annealing. This means that the deep defect concentration in as-grown GaN can be improved by the heat treatment.

The activation energies and the intercept capture cross-section of the trap levels were obtained from an Arrhenius plot which illustrates the dependence of the thermal emission rate e_n on the reciprocal temperature $1000/T$ with T being the temperature corresponding to the DLTS peak at different rate windows, as shown in Fig. 3. The slope of the plot gives information about the activation energy, and the capture cross section is determined from the intercept point when $T \rightarrow \infty$. From the peak amplitudes, the concentration of traps was determined as in Ref. 13 where we have considered the

FIG. 3. Arrhenius plots of the electron emission rates for different deep levels observed in n-type GaN grown by HVPE before and after electron-irradiation.

TABLE I. The activation energy E_a ($E_c - E_t$), the intercept capture cross section σ_{int} and the trap concentration N_t deduced from the peak amplitudes before irradiation, after electron irradiation by 2 MeV electrons with a fluence of $1 \times 10^{14} \text{ cm}^{-2}$ and after annealing at 1000 °C for 30 min. ΔN_T is the increase of traps due to irradiation.

Trap level	E_a (eV)	σ_{int} (cm ²)	N_T (cm ⁻³) Before irradiation	N_T (cm ⁻³) After irradiation	N_T (cm ⁻³) After annealing	ΔN_T (cm ⁻³) After irradiation
D1	0.12	1.7×10^{-18}	0	4.3×10^{13}	0	4.3×10^{13}
D2	0.24	2.0×10^{-16}	1.5×10^{13}	$\sim 1.5 \times 10^{13}$	8.4×10^{12}	0
D3	0.60	2.5×10^{-15}	5.4×10^{13}	$\sim 5.4 \times 10^{13}$	1.6×10^{13}	0
D4	0.69	1.5×10^{-15}	$\sim 2.4 \times 10^{13}$	$\sim 2.4 \times 10^{13}$	8.5×10^{12}	0
D5	0.96	3.0×10^{-14}	$\sim 3.4 \times 10^{13}$	...	8.3×10^{12}	...
D5I	0.89	1.1×10^{-15}	0	$\sim 1.0 \times 10^{14}$	0	$\sim 1.2 \times 10^{14}$
D6	1.14	7.7×10^{-14}	0	$\sim 1.6 \times 10^{14}$	0	$\sim 1.5 \times 10^{14}$
D7	1.19	1.5×10^{-13}	$\sim 5.3 \times 10^{13}$	$\sim 5.3 \times 10^{13}$	0	0
D8	...	...	$\sim 1.7 \times 10^{13}$	...	$\sim 1.9 \times 10^{13}$	...

influence of the free electron carrier tail. The obtained activation energies, capture cross sections and trap concentrations before and after irradiation and after annealing are presented in Table I.

Subtracting the signal measured before irradiation (Fig. 2(c)), we obtain the increase of the trap concentration (ΔN_T) after irradiation. However, since it was observed that peaks D5I and D6 were annealed out already during the high temperature DLTS measurements, the concentrations given for these levels are slightly underestimated.

Fig. 4 shows the concentrations of levels D1, D5I, and D6 extracted from sub-sequential DLTS measurements. The concentrations of traps show a tendency of decreasing to the values as in the as-grown sample where N_0 is the concentration of traps in the first scan and N_t is the concentration of trap measured in each scan. As can be seen in the figure, the annealing process starts already at temperatures above 550 K for all defects. However, the process is slightly slower for trap D5I and the signals from the traps do not disappear completely until after annealing at 1000 °C for 30 min (the fifth scan).

Peak D1 is introduced by electron irradiation and has been reported several times.^{5,7,9} Fang *et al.*⁵ suggested the level to be related to the N vacancy (V_N). From simulation, this peak is suggested to consist of several overlapping levels.

FIG. 4. The reduction in concentration of the traps D1 (square), D5I (round), and D6 (triangle) after each DLTS scan. The 1st scan was done in the temperature range 77–550 K; the 2nd scan in 77–600 K, the 3rd and 4th scans in 77–700 K; the 5th scan was performed after annealing at 1000 °C for 30 min in N_2 .

Polenta *et al.*⁹ fitted this DLTS peak using two components with a thermal activation energy of 0.06 and 0.11 eV, respectively. In a later study on MOCVD grown GaN, Godmann *et al.*⁷ suggested that this peak is resulted from three overlapping levels. From numerical fitting, the activation energies were determined to $E_c - 0.06$ eV, $E_c - 0.10$ eV, and $E_c - 0.20$ eV, respectively.

The trap D2 with an activation energy of 0.24 eV detected before irradiation is commonly detected in as-grown GaN regardless of growth method.^{14–16} There have been different reports on this trap; however, its origin is still under debate. By using two kinds of precursors (TMGa and TEGa), Lee *et al.*¹⁷ proposed D2 to be carbon or hydrogen related defects. In a later study, Fang *et al.* suggested the peak to be related to the divacancy $V_N - V_{Ga}$.¹⁸

The trap D3 at $\sim E_c - 0.60$ eV has been previously reported^{10,11,16,19,20} and one of the suggested defect models is the N antisite (N_{Ga}).¹⁹ However, later studies of electron-irradiated HVPE-grown GaN²¹ ruled out the model of a simple intrinsic defect since no change of the concentration was observed after irradiation. This is in agreement with our observation. It was suggested that the defect is associated with some common impurity such as Si, O, or C.

The signal from the trap D4 is very weak and has been previously observed in as-grown material.^{21,22} However, the concentration of the defect is unaffected by irradiation and thermally stable, which suggests that it is related to an impurity or possibly an impurity complex.

The trap D5 ($E_c - 0.96$ eV) is observed before irradiation and after annealing at 1000 °C. Due to the overlap with D7, it is difficult to characterize the peak in detail. After irradiation, peak D5 is completely immersed by the strong peaks D5I with an activation energy of 0.89 eV and by D6.

In Ref. 6, Goodman *et al.* studied electron irradiated MOCVD grown GaN and they observed a broad peak with an activation energy of 0.913 eV. By numerical fitting it was suggested that the peak consisted of at least 4 closely overlapping features coinciding with peaks D5 and D5I. However, no suggestion of its origin was given. In n-type HVPE-grown GaN irradiated by 25 GeV H^+ ions, Castaldini *et al.*⁴ observed a level at $\sim E_c - 0.90$ eV. The peak was severely overlapped with signals from other levels, hindering the identification. Based on the thermal stability at high temperatures, we suggest D5 to be associated with an isolated impurity or its associated complex. Judging from low

annealing temperatures (~ 550 K) and high introduction rate ($\sim 1.2 \text{ cm}^{-1}$), the D5I center may be related to a primary intrinsic defect.

Trap D6 at $E_C - 1.14$ eV is only clearly observed after irradiation. However, due to overlapping with other peaks, we cannot rule out that the defect is already present before irradiation and so far there has not been any report about this trap. The defect has a high introduction rate ($\sim 1.5 \text{ cm}^{-1}$) and starts annealing out already at 550 K, i.e., it has a similar introduction and annealing behavior as the trap D5I. Therefore, we suggest that the trap D6 may also be related to a primary intrinsic defect.

Trap D7 ($E_C - 1.19$ eV) is observed before irradiation (Fig. 2(a)) and annealed out at $\sim 1000^\circ\text{C}$ with the trap concentration being unaffected by irradiation. It has previously been observed by Ito *et al.*²³ but no defect model has been suggested for this trap. Since the concentration is unaffected by electron irradiation, the level is unlikely to be associated with a primary intrinsic defect. On the right shoulder of peak D7, we observe the weak peak D8 which has an interesting annealing behavior. After irradiation and a partial anneal due to the thermal DLTS scan (Fig. 2(b)), the signal disappeared, but then reappears after annealing at 1000°C . The peak is observed at temperatures where intrinsic defects introduced by the electron irradiation become mobile (~ 550 K). Therefore, we tentatively suggest that the defect associated with the level D8 may form a complex with a primary intrinsic defect during the DLTS scan leading to the vanish of the signal in the DLTS spectrum. After annealing at 1000°C (Fig. 2(d)), the complex is disassociated and the level D8 is observed again. It was not possible to determine the activation energy and capture cross section of this level due to high leakage current at the high temperature but its peak position coincides with a level labeled E5 in Ref. 22.

In conclusion, freestanding bulk GaN was irradiated with 2 MeV electrons at a fluence of $1 \times 10^{14} \text{ cm}^{-2}$ at room temperature. In as-grown materials, six electron traps were observed for as-grown GaN, D2 ($E_C - 0.24$ eV), D3 ($E_C - 0.60$ eV), D4 ($E_C - 0.69$ eV), D5 ($E_C - 0.96$ eV), D7 ($E_C - 1.19$ eV), and D8 where D4, D5, D7, and D8 form a broad band in the temperature range of 350–600 K. After electron irradiation, three traps were observed. Among these, the trap D1 ($E_C - 0.12$ eV) was associated to N vacancy and the traps D5I ($E_C - 0.89$ eV) and D6 ($E_C - 1.14$ eV) were suggested to be related to primary intrinsic defects based on their high introduction rate and relatively low-temperature annealing behavior. The concentration of irradiation-induced traps (D1, D5I, D6) decreased already during the high-temperature DLTS scans and after annealing at 1000°C for 30 min in a N_2 environment they were completely annealed

out. The annealing process started at ~ 550 K; thus, primary defects are mobile already during the measurements. Most interestingly, after irradiation and a partial anneal due to the thermal DLTS scan, peak D8 disappeared and after annealing at 1000°C , the peak reappeared. Thus, we suggest that the defect associated with peak D8 forms a complex with a primary intrinsic defect and the complex may be dissociated by annealing at 1000°C . However, in order to verify this and understand the annealing process, further studies are necessary.

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