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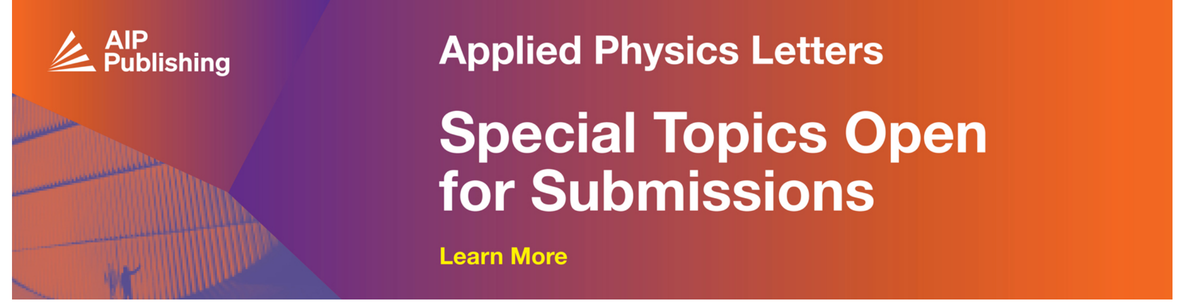

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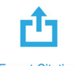
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ABSTRACT

It has been established that the formation of point defects and their behaviors could be regulated by growth details such as growth techniques and growth conditions. In this work, we prove that C doping approaches have great influence on the charge state of C_N, thus the interaction between H and C in GaN. For GaN with intrinsic C doping, which is realized by reducing the V/III ratio, C_N mainly exists in the form of C_N^- charged from the higher concentration of V_N and, thus, may attract H^+ by coulomb interaction. Whereas for the extrinsically C doped GaN with propane as the doping source, the concentration of V_N is reduced, and C_N mainly exists in neutral charge state and, thus, nearly does not attract H ions. Therefore, we demonstrate that the interplay between H and C atoms is weaker for the extrinsically C doped GaN compared to the intrinsically doped GaN, thus gives a clear picture about the different charge states of C_N and the formation of C–H complexes in GaN with different C doping approaches.

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The characteristics of defects in semiconductors such as the lattice location, charge state, and interaction with other defects are proved to vary sensitively with detailed growth environment including growth methods, growth conditions, and dopant sources.[1–4] For instance, carbon (C) and hydrogen (H), as the most important impurities in GaN, have been reported to have great influence on device reliability and stability.[5–8] However, the behaviors of C and H atoms in GaN, such as the lattice site, charge state, and the interplay between them, are very complicated, especially for the reason of the sensitivity to growth details.[9–13] It has been established that the lattice site of C in GaN is correlated with growth methods. In GaN films grown by HVPE, they mainly exist in the form of complexes, i.e., tri-carbon,[3] while in GaN grown by metalorganic chemical vapor deposition (MOCVD), isolated C_N is more plausible.[14] Even with the same growth method (MOCVD), it is reported that different C doping approaches also result in different C_N/C_{Ga} ratios.[4] C doping is a significant step in the growth of semi-insulating GaN, and it could directly impact the behavior of C related defects,[9,10,15] which is crucial not only for fundamental physics but also for device applications. Specifically, there mainly exist two kinds of C doping techniques in MOCVD. One is the traditional C doping approach in which C originates from the organic Ga source, and the concentration is regulated by changing the growth parameters including temperature, pressure, and V/III ratios,[16] thus called intrinsic doping. The other doping approach is realized by applying external carbon source such as propane or ethylene,[17] hence called extrinsic doping, which benefits to maintain the optimum growth conditions. Referring to device applications, it is reported that C doping approaches in MOCVD play a key role in affecting device

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dynamic and voltage withstanding properties, both are directly associated with the charge state of C and C–H related defects.[5,18] By adopting the extrinsic C doping,[15,19] better dynamic properties are realized in power electronic devices. However, the physics behind, especially for the effects of doping approaches on the charge states of C, and the formation of C–H complexes are still not well understood.

In this Letter, it is illustrated that the C doping approaches can greatly affect the charge state of C_N, therefore the interaction between H and C atoms in GaN. The microscopic mechanism is also revealed. In the intrinsically C doped GaN, C_N mainly exists in the form of C_N^-, which is charged from the high concentration of V_N^+, thus attracts more H^+ by coulomb force. While in the extrinsically C doped GaN, due to the lower concentration of V_N^+, C_N mainly exists in the form of C_N^0 and attracts fewer charged H atoms. We, thus, give a clear picture of the different charge states of C_N and the formation of C–H complexes in GaN vs different C doping approaches.

The investigated GaN layers were grown on top of AlN/AlGaN transition layers by MOCVD on Si (111) substrates. The GaN films were separated to two groups referring to intrinsic [using organic Ga source $Ga(CH_3)_3$ as the C precursor] and extrinsic (using propane as the C precursor) C doping approaches. The C and H concentrations were determined by secondary ion mass spectroscopy (SIMS) measurement. The concentrations of C and H in the intrinsically doped GaN films are in the range of 2.0×10^{16}–$9.0 \times 10^{19}\,cm^{-3}$ and 1.5×10^{17}–$1.2 \times 10^{18}\,cm^{-3}$, respectively. While for the extrinsically doped films, they are in the range of 2.0×10^{16}–$2.0 \times 10^{19}\,cm^{-3}$ and 1.4×10^{17}–$3.0 \times 10^{17}\,cm^{-3}$, respectively. Photoluminescence (PL) measurements were carried out using a continuous wave (CW) laser with a wavelength of 360 nm. Raman spectroscopy was performed with a backscattering geometry using a laser with a wavelength of 532 nm. Thermal annealing treatment was performed with a face-to-face pattern at various temperatures for 1 h in N_2-ambient tube annealing furnace. High frequency Electron Paramagnetic Resonance (EPR) measurements were performed on a locally developed superheterodyne quasi-optical pulsed/cw EPR spectrometer, which is located at the National High Magnetic Field Laboratory in Tallahassee, FL, USA.[20,21]

The concentrations of H and C of the two groups of GaN films are statistically shown in Fig. 1. It is found that the concentration of H (unintentionally doped) increases with C (intentionally doped) for the two groups of GaN films. We, thus, speculate that there exist some correlations for the incorporation of H and C in both C doping approaches. Additionally, it is interesting that the concentration of H increases faster with C incorporation in the intrinsically doped GaN than that in the extrinsically doped ones. We note here that the detection limit of H is about 10^{17} cm^{-3}. Based on the aforementioned facts, we, therefore, conclude that the C doping approaches have an important effect on the incorporation of H.

To analyze the discrepancy of the incorporation of H and the relation with C atoms in GaN films with different C doping approaches, PL measurements were performed on GaN films ([C] $\sim 10^{17}\,cm^{-3}$) with different doping approaches, and the related PL spectra at 80 K are shown in Fig. 2(a). In the spectra, there mainly exist two broad luminescence bands located at the YL (named as YL_1, peaked at 2.25 eV, and related to C_N $(-/0)$ level) and BL (peaked at 3.02 and 2.88 eV) regions. It is found that in the region of the BL band, the luminescence of the two groups of GaN is quite different.

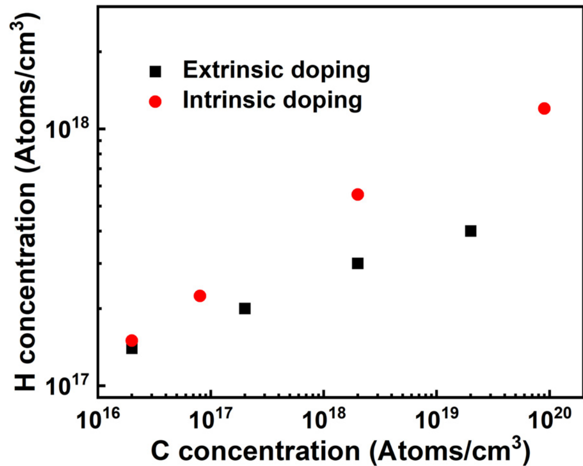

FIG. 1. SIMS measurement of the H concentration dependent on the C concentration for different C doped GaN films grown by intrinsic and extrinsic doping approaches.

Specifically, for the intrinsically C doped GaN, there only exists luminescence peaked at 3.02 eV (named as BL_2), which is proved to be associated with electron transitions between the conduction band or shallow donors, and 0/+ level of C_N–H_i complex, or the internal transitions from the excited state to the ground state of C_N–$H_i(0/+)$ donor.[22,23] While for the extrinsically C doped sample, there only appears luminescence peaked at 2.88 eV (named as BL_C), which is attributed to electron transitions from the conduction band, shallow donors, or an excited state to the ground state of $C_N(0/+)$ level under high density excitation.[24] Based on the above discrepancy between GaN with intrinsic and extrinsic C doping approach, we can speculate that the concentration of C_N–H_i complex in the intrinsically C doped GaN is much higher than that in the extrinsically C doped ones, though the C concentration is similar.

To explicitly identify the difference between the two groups of GaN, the annealing treatment at 1000 °C under N_2 ambient was also performed, as depicted in Fig. 2(b). For GaN with extrinsic C doping, the luminescence including the YL_1 and BL_C bands stays almost constant; while for GaN with intrinsic C doping, BL_2 totally disappears, and meanwhile, the BL_C band appears accompanied by the increase in the YL_1 intensity, revealing the dissociation of C_N–H_i complex to C_N^- and H^+ followed by the escape of the H atoms out from the GaN surfaces. The schematic band diagram of the transitions of electrons in different luminescence processes is also described in Fig. 2(c). In fact, we also found a similar phenomenon in the UV-VIS absorption spectra that the intensity of the C_N related absorption peak stays unchanged after annealing for extrinsically C doped GaN while enhances dramatically for intrinsically C doped ones, indicating the negligibly low decomposition of C_N–H_i complex for the extrinsically C doped GaN. The results are not shown here due to the space limit. It could, thus, be concluded from the coincident results of SIMS, PL, and absorption measurements that the extrinsic C doping approach reduces the incorporation of H and weakens the interplay between H and C atoms in GaN.

In order to further analyze the influence mechanism of the doping approaches, the doping mechanism should be deeply understood.

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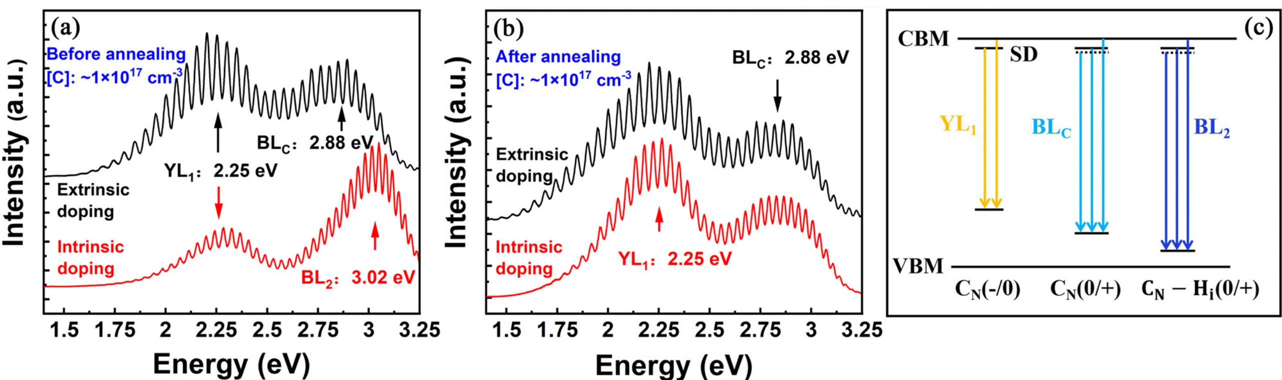

FIG. 2. PL spectra (80 K) of intrinsically and extrinsically C doped GaN films with C concentrations of around 10^{17} cm^{-3} (a) before and (b) after annealing treatment. The excitation power is 50 W/cm^2. (c) The schematic band diagram model of the transitions of electrons in different luminescence processes for C doped GaN films, in which SD is the abbreviation of shallow donor.

The doping process is as follows: in the intrinsically C doped GaN, C doping here is mainly achieved by reducing the V/III ratio, which will suffer from the deviation of the optimal growth conditions of GaN.[16] Especially, when the V/III ratio is reduced for more heavily C doping, a higher concentration of anion vacancy, i.e., nitrogen vacancy (V_N), would be formed.[11] In comparison, for the extrinsically C doped films, the C doping is achieved by changing the C source (here is propane) flow without changing the V/III ratio, which will benefit for maintaining the best growth conditions.[17] Hence, the concentration of V_N is lower under the latter growth conditions. Therefore, although the concentration of C atoms is comparable in the two groups of GaN films, the concentration of V_N will be speculated to be higher in the intrinsically C doped GaN than that in the extrinsically C doped samples. In order to verify the above speculation, Raman measurements on GaN films (with different C doping concentrations) grown by the two doping approaches were conducted, as depicted in Fig. 3. Except for the E_2 (high) peak of AlN, a new peak located at 671 cm^{-1} is also observed.

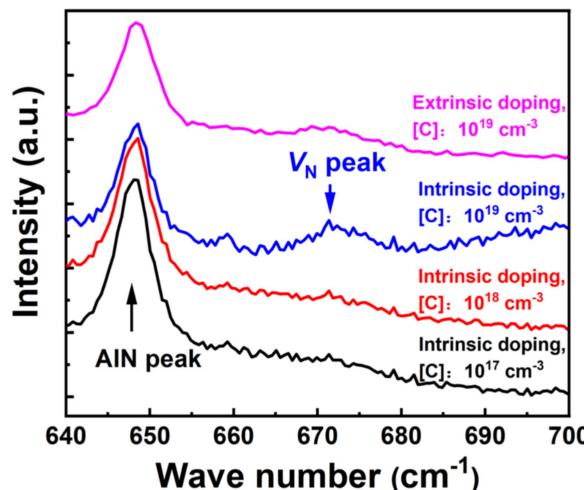

FIG. 3. Raman spectra for extrinsically and intrinsically C doped GaN films with different C and H concentrations.

According to previous reports,[25–27] the Raman peak located at 671 cm^{-1} is correlated with V_N. For the intrinsically C doped GaN films, it is found that the intensity of the V_N related peak increases with increase in the C concentration. While for the extrinsically C doped GaN films, the situation is changed. The intensity of the V_N related signal remains very weak and stays almost constant along with C doping, indicating the concentration of V_N donor is very low. Here, only the result of heavily extrinsically C doped GaN ([C]: 2×10^{19} cm^{-3}) is shown in Fig. 3 for comparison. The Raman results confirm the previous speculations that the GaN films with intrinsic C doping approach have higher concentration of V_N donor than that in the extrinsically C doped GaN, when the concentrations of C atoms are comparable. To step further, the crucial effect of the V_N donor on the charge state of acceptors such as C_N, thus C–H complex would be speculated to occur.

To reveal the charge state of C_N, photo-assisted high frequency Electron Paramagnetic Resonance (EPR) measurements under 3.3 K were performed on heavily C doped GaN films ([C] $\sim 9 \times 10^{19}$ cm^{-3} for the intrinsic doping sample and 2×10^{19} cm^{-3} for the extrinsic doping one, respectively). The light source is chosen to be an xenon lamp, for its ability to excite electrons from C_N acceptor level (0.9–1.0 eV above the VBM) to conduction band or shallow donors. Figure 4 illustrates the results of the two groups of heavily C doped GaN films with and without illumination. One can find that an EPR signal at 8.625 T for the intrinsically C doped GaN and a signal at 8.635 T for the extrinsically C doped GaN are observed. The signals are, however, not found in GaN with lower C concentration (not shown here). We should mention here that the asymmetric differential signal may be attributed to the saturation due to long spin lifetime. According to the formula $h\nu = g\beta H$, h is the Planck constant, ν is the microwave frequency (here is 240 GHz), g is the Lande g factor, β is the Bohr magneton, and H is the applied magnetic field. The g factor is calculated to be 1.988 and 1.986 for the two samples, respectively. Zvanut *et al.*[28] previously reported that the EPR signal with a g factor of 1.987 is attributed to the uncharged C_N (C_N^0) in C doped GaN grown by HVPE. We should mention here that only C_N^0 (with unpaired electrons) could be activated, while C_N^- and C_N^+ (without unpaired electrons) are inactive in EPR signal. Accordingly, we believe here the signal with g factor of 1.988 and 1.986 should also be

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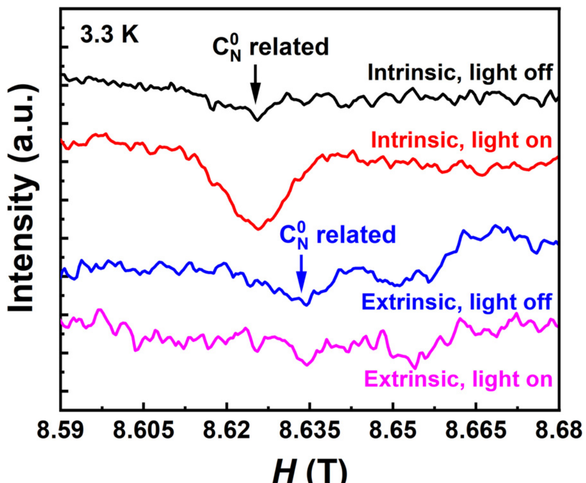

FIG. 4. Photo-assisted high field EPR spectra of heavily C doped GaN films ($>10^{19}$ cm^{-3}) grown by different doping approaches with and without illumination.

attributed to the uncharged C_N in $C_N(-/0)$ acceptor level, while the shift of which is considered to be related to the changes in the spin local environment due to the discrepancy of the growth method and doping approaches. Here, an additional EPR peak at 8.654 T (g factor is calculated to be 1.981) is also observed and needs to be further identified.

We now turn to analyze the change of the charge state of C_N in GaN before and after illumination. In Fig. 4, for the as-grown GaN with intrinsic C doping (light off), the C_N^0 related EPR signal is rather weak, indicating that the concentration of the uncharged C_N is very low. When the illumination is applied (light on), the intensity of the C_N^0 related signal dramatically increases. The increase is attributed to the transition of C_N^- to C_N^0 when the electrons on C_N^- level being excited to the conduction band by applying the illumination. Thus, the conclusion can be drawn that C mainly exists in the form of C_N^- in the as-grown intrinsically C doped GaN films. In contrast, the situation changes in the extrinsically C doped GaN. Before the light is applied, there exists a more intense C_N^0 related signal compared with the case in the intrinsically C doped GaN, indicating a higher concentration of C_N^0. After applying the illumination, the C_N^0 related signal is not improved but rather weakened to some extent, which we guess is attributed to the experimental error. It is, thus, suggested that C mainly exists in the form of C_N^0 in the extrinsically doped GaN. Combing with the Raman scattering results, we consider that the higher concentration of C_N^- in the intrinsically C doped GaN is attributed to the higher concentration of V_N that transfers electrons to compensate C_N. This is also in agreement with previous reports that V_N acts as the dominant compensation donors in C doped GaN.[11,29–31] Based on those facts, we, thus, conclude that doping approaches have an important influence on the charge state of C_N in GaN.

Finally, we will discuss the influence of C doping approach on the interaction between C and H impurities in GaN. C and H are known to interact with each other by Coulomb force; hence, the negative charge of C_N will act as the Coulomb attraction center to H atoms, based on the fact that H mainly exists in the form of positive charge state (H^+) in semi-insulating and n-type GaN.[32] Owing to the fact that C_N^- tends to attract positively charged H atoms, while C_N^0 has no Coulomb attraction to them, the higher concentration of C_N^- could, thus, interpret the higher concentration of C_N–H_i complex in intrinsically C doped GaN. Actually, the charge state of C_N and the formation of C_N–H_i complex in GaN could also affect the devices performance. On the one hand, it is reported that the dissociation of C_N–H_i complex under high electric field could deteriorate the device stability and reliability.[7,8] With the rapid scaling down of current electronic devices, they would withstand higher electric field and, thus, produce large numbers of hot electrons with high kinetic energy.[33] Under such circumstances, the atomic bonds of C_N–H_i complexes could be broken, and more charged defect states could be introduced, which may lead to the transconductance degradation and threshold voltage shifts.[8] On the other hand, the positive contribution of H to passivate the C deep level trap states associated with current collapse contrasts the negative aspects.[5,6] As such, by taking the effect of the C doping approach on the charge state of C_N and the formation of C–H complex as an example, this work provides necessary guidance for the investigation about the point defect behaviors with respect to growth details for high-quality GaN growth.

In summary, the influence of the C doping approaches on the charge state of C_N and consequently the interaction between H and C impurities in GaN have been explicitly investigated. In the intrinsically C doped GaN grown by reducing the V/III ratio, more intrinsic donors V_N^+ would be formed, and C_N, thus, mainly exists in the form of C_N^- charged from V_N^+ and, therefore, attracts more H^+ ions by coulomb force. While in GaN doped with external C source, the optimized growth conditions can be achieved without tuning the V/III ratio, C_N then mainly exists in a neutral charge state due to the lower concentration of V_N^+ donors and the attraction of fewer H atoms. Therefore, we reveal that the attraction of C to H atoms is weaker in the extrinsically C doped GaN compared to the intrinsically doped ones and clarify the charge state of C_N and the formation of C–H complexes in GaN with different doping approaches. Those results should guide the growth strategy for the demanded GaN layers in relevant electronic devices.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

Shan Wu: Conceptualization (lead); Data curation (lead); Formal analysis (lead); Investigation (lead); Methodology (lead); Writing – original draft (lead); Writing – review and editing (lead). **Xuelin Yang:** Formal analysis (equal); Funding acquisition (equal);

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Investigation (equal); Project administration (equal); Writing – review and editing (equal). **Zhenxing Wang:** Funding acquisition (supporting); Resources (supporting). **Zhongwen Ouyang:** Funding acquisition (supporting); Resources (supporting). **Huayang Huang:** Formal analysis (supporting). **Qing Zhang:** Resources (supporting). **Qiuyu Shang:** Resources (supporting). **Zhaohua Shen:** Formal analysis (supporting). **Fujun Xu:** Formal analysis (supporting). **Xinqiang Wang:** Formal analysis (supporting). **Weikun Ge:** Formal analysis (supporting); Writing – review and editing (supporting). **Bo Shen:** Formal analysis (equal); Funding acquisition (equal); Project administration (equal); Resources (equal).

DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding authors upon reasonable request.

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