

Negative-U behavior of the Si donor in $\text{Al}_{0.77}\text{Ga}_{0.23}\text{N}$

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Electron paramagnetic resonance (EPR) spectrum of a shallow donor is observed at low temperatures in darkness in Si-doped $\text{Al}_{0.77}\text{Ga}_{0.23}\text{N}$ epitaxial layers grown on 4H-SiC substrates. It is shown from the temperature dependence of the donor concentration on the neutral donor state measured by EPR that Si is a DX (or negative-U) center but behaves as a shallow donor due to a small separation of only ~ 3 meV between the neutral state E_d and the lower-lying negative state E_{DX} . The neutral state is found to follow the effective mass theory with $E_d \sim 52$ – 59 meV.

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High-Al-content $\text{Al}_x\text{Ga}_{1-x}\text{N}$ ($x > 0.7$) is the principal alloy system for the development of deep ultraviolet light emitting diodes (LEDs)^{1–5} with contemplated enormous social impact if considering their intended implementation in portable units for water/air purification and sterilization.⁶ Electrical pumping of LEDs requires both *n*- and *p*-type doping. Silicon (Si) is the only technologically important *n*-type dopant in the $\text{Al}_x\text{Ga}_{1-x}\text{N}$ alloy system ($0 < x < 1$). Achieving high *n*-type conductivity in high-Al-content $\text{Al}_x\text{Ga}_{1-x}\text{N}$ is proven difficult. The presence of readily incorporated oxygen (O) impurity, especially in high-Al-content $\text{Al}_x\text{Ga}_{1-x}\text{N}$, complicates the doping study even further. It is known that both Si and O are shallow donors in GaN but their electronic structure in high-Al-content $\text{Al}_x\text{Ga}_{1-x}\text{N}$ is still much debated.

Theoretical studies by Park and Chadi⁷ suggested that O and Si undergo a transition from a shallow donor in GaN to a deep DX center in $\text{Al}_x\text{Ga}_{1-x}\text{N}$ when $x \sim 0.20$ for O and $x \sim 0.26$ for Si. The DX behavior has also been found by other calculations for Si (at $x \geq 0.6$)⁸ and O (at $x \geq 0.4$).^{9,10} In these DX (or negative-U) centers, the donor in its neutrally charged state d^0 prefers to capture another electron and relaxes to a deeper negatively charged state DX^- , forming an ionized donor state d^+ according to the process: $2d^0 \rightarrow d^+ + \text{DX}^-$.⁷ The DX behavior occurs in connection with a large lattice relaxation caused by a Coulomb attraction between the impurity and third nearest neighbors.¹⁰ This is relevant for O occupying the anion site, which is negatively charged in the DX^- state, interacting with the third neighbor cation (Ga or Al), but not for Si substituting for the cation site. It was, therefore, suggested that Si should remain to be a shallow donor even in AlN.¹⁰ The conclusion was supported by a Raman scattering study of Si and O in GaN under hydrostatic pressure¹¹ which increases the band gap similar to increasing the Al content in $\text{Al}_x\text{Ga}_{1-x}\text{N}$. This study predicted the transition from a shallow donor to a DX center to occur at $x \sim 0.40$ for O, but no DX formation for Si up to $x \sim 0.56$ (corresponding to the highest pressure used in their experiments).¹¹ Hall-effect and persistent photoconductivity measurements suggested that O becomes a DX center for $x \sim 0.27$, whereas no DX behavior has been detected for Si up to $x \sim 0.44$.¹² In a later transport study of Si-doped $\text{Al}_x\text{Ga}_{1-x}\text{N}$ with higher Al content ($0.5 < x < 0.6$), O and Si

were suggested to become DX centers for x exceeding ~ 0.3 and ~ 0.5 , respectively.¹³

Concerning O, previous studies seem to agree that O becomes a DX center in $\text{Al}_x\text{Ga}_{1-x}\text{N}$ with x exceeding ~ 0.3 – 0.4 , and hence, is a carrier compensating defect, hindering the *n*-type doping in the material. For Si, reported results are controversial. More recently, several groups have obtained *n*-type conductivity in $\text{Al}_x\text{Ga}_{1-x}\text{N}:\text{Si}$ with $x \sim 0.5$ – 1.0 and concluded that Si is a shallow donor even in AlN.^{14–19} However, in transport measurements, it is not possible to know if free carriers come from the neutral d^0 or negative DX^- states of the donor. In electron paramagnetic resonance (EPR) studies of Si-doped AlN, the EPR signal of the shallow donor could only be observed under or after illumination at temperatures below 60 K.²⁰ The behavior was explained by the negative-U properties of the Si donor with the DX^- level at ~ 0.32 eV below the conduction band E_C .²⁰ Similar DX behaviors were observed for the donor in $\text{Al}_x\text{Ga}_{1-x}\text{N}:\text{Si}$ with $x \sim 0.75$ or larger, but the location of the DX^- level was not determined.²¹ In EPR studies of undoped AlN, O was also suggested to be a DX center.^{22,23} However, in the EPR study of AlN:Si by Irmscher *et al.*,²⁴ Si was suggested to be a shallow donor and the failure of detecting the signal in darkness was explained as due to carrier compensation by other lower-lying defect levels. In a more recent EPR study of unintentionally Si-doped AlN, the EPR spectrum of the Si shallow donor could be detected in darkness at temperature above ~ 95 K and from the temperature dependent study of the donor concentration it was suggested that Si is a DX center with the DX^- level at ~ 78 meV below the neutral d^0 state.²⁵ More recent hybrid functional calculations suggest that both Si and O are DX centers in AlN.²⁶

In this letter, we report EPR study of $\text{Al}_x\text{Ga}_{1-x}\text{N}:\text{Si}$ with $x \sim 0.77$. By monitoring the change of the concentration of the neutral state d^0 with temperature in darkness, we show that Si already becomes a DX center as previously suggested by Bayerl and co-workers²¹ but behaves as a shallow donor due to a small separation of only ~ 3 meV between the DX^- and d^0 states, and its shallow states follow the effective mass theory (EMT).

The $\text{Al}_x\text{Ga}_{1-x}\text{N}:\text{Si}$ epitaxy was performed by metal-organic chemical vapor deposition (MOCVD) on semi-insulating 4H-SiC substrates following an *in situ* template

growth. Typical performance of the MOCVD system as to the AlN growth,²⁷ and Si-doped²⁸ high-Al-content AlGa_N layers can be found elsewhere. The dopant gas silane (SiH₄) was delivered with a gas-flow-rate ratio to the total metal-organic precursor flow in the range of $\sim 1.8\text{--}3.3 \times 10^{-4}$. The Al content, the thickness of the Al_xGa_{1-x}N:Si layer, and the atomic concentration of Si, [Si], and O, [O], were determined by secondary ion mass spectrometry (SIMS) by Evans Analytical Group (the notation [] is used in this paper to denote the concentration obtained from SIMS). The deposition parameters were tuned to obtain [Si] in the range of low 10^{18} cm^{-3} , which gives good EPR signals while the conductivity is still not too high so that EPR measurements with high Q-factors can be performed at high temperatures. The typical thickness of the layers is $\sim 400\text{ nm}$ and the Al content is $x \sim 0.77$. The energy of the near band edge (NBE) emission in the studied Al_xGa_{1-x}N structures as measured by cathodoluminescence at room temperature is $\sim 5.37\text{ eV}$. EPR measurements were performed on an X-band ($\sim 9.4\text{ GHz}$) E500 Bruker spectrometer equipped with a continuous He-flow cryostat, allowing the regulation of the sample temperature from 4 to 295 K. For illumination, halogen (200 W) or xenon (150 W) lamp was used as excitation source. The donor concentration or the number of spins were determined from double integration and measurement conditions using the spin counting application provided and calibrated by Bruker.

In the Al_{0.77}Ga_{0.23}N:Si layers with the free carrier concentration, n , in the range of low 10^{18} cm^{-3} as determined by either capacitance-voltage or microwave-based contactless measurements, an EPR line was observed in darkness at low temperatures. Fig. 1 shows the EPR spectra observed in darkness at different temperatures in the range of $\sim 6\text{--}80\text{ K}$ in a Si-rich sample ([Si] $\sim 2 \times 10^{18}\text{ cm}^{-3}$ and [O] $\sim 3 \times 10^{17}\text{ cm}^{-3}$). The inset shows the integrated EPR spectra with their intensity being corrected for the Boltzmann distribution between the two spin levels $M_S = \pm 1/2$ and the Q-factor at different temperatures so that the area under the curve is directly proportional to the number of spins. The g-value of the spectrum decreases with

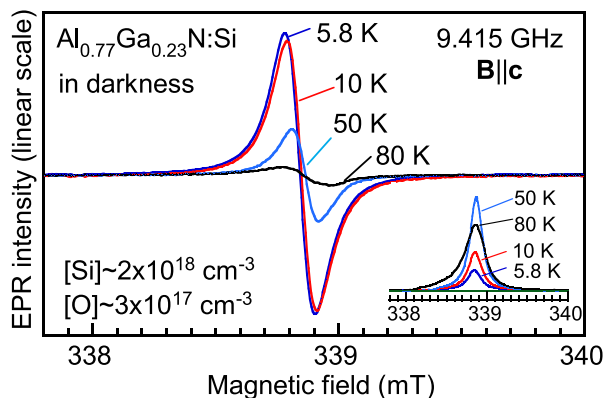


FIG. 1. EPR spectra measured in darkness at different temperatures for $B||c$ in a Si-doped Al_{0.77}Ga_{0.23}N layer with [Si] $\sim 2 \times 10^{18}\text{ cm}^{-3}$, [O] $\sim 3 \times 10^{17}\text{ cm}^{-3}$, and $n \sim 1.6 \times 10^{18}\text{ cm}^{-3}$. The inset shows the integrated EPR spectra with their intensity being corrected for the Boltzmann distribution between the two spin levels $M_S = \pm 1/2$ and the Q-factors at different temperatures so that the area under the curve is directly proportional to the number of spins.

increasing temperature ($g = 1.9851$ at $\sim 6\text{ K}$ and $g = 1.9849$ at $\sim 80\text{ K}$) and increases with increasing the Al content as expected for a shallow donor in Al_xGa_{1-x}N.²¹

As can be seen in Fig. 1, the EPR spectra of the thin ($\sim 400\text{ nm}$), low-residual-oxygen-content Al_{0.77}Ga_{0.23}N layers grown by MOCVD on 4H-SiC substrates are characterized by a narrow ($\sim 1.5\text{ G}$) linewidth. The EPR linewidth shows no narrowing effect with increasing temperature in the range of 5–30 K as previously observed in thick ($\sim 1\text{ }\mu\text{m}$) Al_xGa_{1-x}N films grown by plasma-induced molecular beam epitaxy²¹ and thick ($\sim 3\text{--}10\text{ }\mu\text{m}$) GaN films²⁹ grown by MOCVD on sapphire substrates. Apparently, there is a difference in the implemented growth details—including deposition method, process temperature, choice of substrates, thickness and composition of the doped layers, [Si] and [O] content—which may cause various strain effects in the layers for which there is a lack of systematic study on the EPR signal features.²¹ The concentration of free carriers at room temperature in this Si-rich sample is $n \sim 1.6 \times 10^{18}\text{ cm}^{-3}$ (i.e., $\sim 80\%$ of Si shallow donors activated). In this sample, [Si] is about an order of magnitude higher than [O] and should be the principal source of free carriers. The observation of the EPR signal of the shallow donor in darkness at low temperatures indicates that Si behaves as shallow donor in the material.

Fig. 2 shows the EPR spectra measured at 5 K in darkness and under illumination by white light in an O-rich ([Si] $\sim 2 \times 10^{18}\text{ cm}^{-3}$ and [O] $\sim 2 \times 10^{18}\text{ cm}^{-3}$) Al_{0.77}Ga_{0.23}N sample. As has also been observed in many samples, the change of the signal intensity induced by illumination in this O-rich layer is very small, indicating that carrier compensation, if present, should mainly be caused by electron traps in the lower half of the band gap.

From the temperature dependence of the donor concentration on the neutral state d^0 , it is possible to estimate the energy distance between the shallow state and the first excited state as previously shown for the P shallow donor in SiC.³⁰ If a donor transforms into a DX center, the Fermi level E_F is expected to move close to the DX⁻ state, E_{DX} , at low temperatures. Monitoring the temperature dependence of the population on d^0 (or E_d) also allows determining the energy distance between d^0 and E_{DX} . The population on d^0 can be described by the Boltzmann distribution³¹

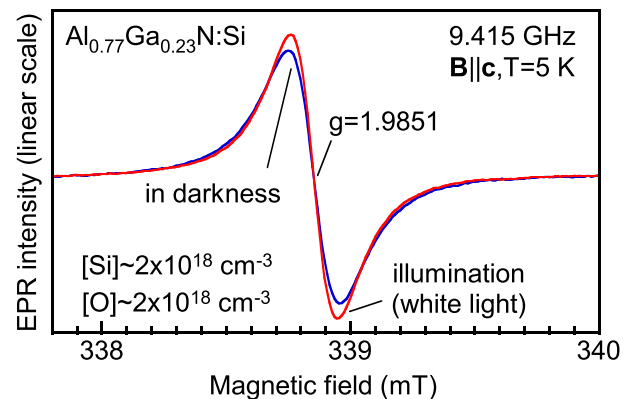


FIG. 2. EPR spectra measured at 5 K for $B||c$ in darkness and under illumination with white light in an O-rich Si-doped Al_{0.77}Ga_{0.23}N layer with [Si] $\sim 2 \times 10^{18}\text{ cm}^{-3}$, [O] $\sim 2 \times 10^{18}\text{ cm}^{-3}$, and $n \sim 2.3 \times 10^{18}\text{ cm}^{-3}$.

$$n(T) \propto \frac{N2e^{-(E_d-E_F)/k_B T}}{1 + 2e^{-(E_d-E_F)/k_B T} + \sum_i G_i e^{-(E_i-E_F)/k_B T}} \quad (1)$$

$$n(T) \propto \frac{N}{1 + 0.5e^{(E_d-E_F)/k_B T} + 0.5\sum_i G_i e^{-(E_i-E_d)/k_B T}}.$$

Here, N is the total concentration of the donor, k_B is the Boltzmann factor, the degenerate factor $G = 2$ for E_d (taking into account the two spin levels $M_S = \pm 1/2$) and E_i are the excited states. The energy is taken to be zero at the conduction band ($E_C = 0$), and hence, $E_d - E_F > 0$, and $E_i - E_d > 0$. The transition from d^0 to the first excited state ($2p$ or $2s$) is the most important for the process of removing electrons from the level since once electrons reach the first excited state, they can move up to higher-lying states within $k_B T$. Considering only the first excited state, Eq. (1) can be rewritten as

$$n(T) \propto \frac{N}{1 + 0.5e^{(E_d-E_F)/k_B T} + Ce^{-\Delta E/k_B T}}. \quad (2)$$

Here, ΔE is the energy distance between d^0 (E_d or E_{1s}) and the first excited state, and C is a factor taking into account the thermal excitation from the first excited state to higher-lying excited states (including their degeneration factors). At low temperatures, E_F is close to E_d for a shallow donor and to E_{DX} for a DX center.

Figs. 3(a) and 3(b) show the temperature dependence of the number of spins on d^0 determined by EPR for two representative Si-rich and O-rich $\text{Al}_{0.77}\text{Ga}_{0.23}\text{N}$ layers whose EPR spectra were shown in Figs. 1 and 2. Unlike a shallow donor

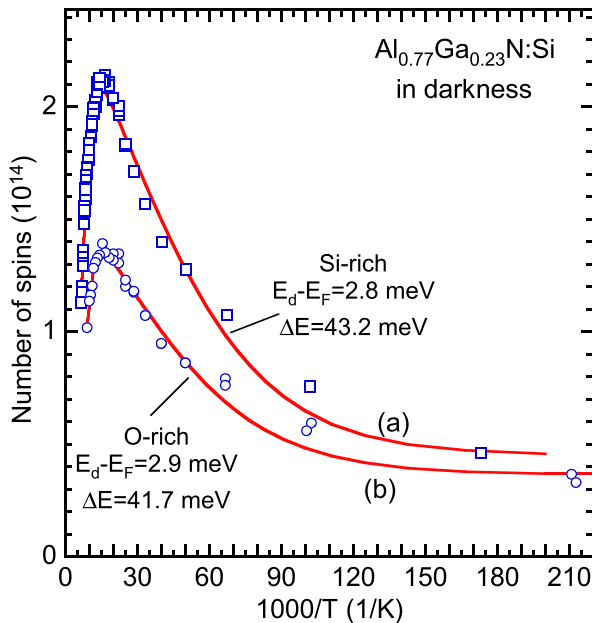


FIG. 3. Temperature dependence of the number of spins on the d^0 state in (a) Si-rich ($[\text{Si}] \sim 2 \times 10^{18} \text{ cm}^{-3}$ and $[\text{O}] \sim 3 \times 10^{17} \text{ cm}^{-3}$) and (b) O-rich ($[\text{Si}] \sim 2 \times 10^{18} \text{ cm}^{-3}$ and $[\text{O}] \sim 2 \times 10^{18} \text{ cm}^{-3}$) $\text{Al}_{0.77}\text{Ga}_{0.23}\text{N}$ layers. The solid curves represent the fits using Eq. (2). The concentration was determined from spectra measured in darkness with different microwave powers of 0.1 and 1.0 mW. The concentrations estimated by EPR in two samples are similar and the difference in the number of spins is due to different volume of their Si-doped layers. (The number of spins is used instead of the concentration in order to avoid errors induced when estimating the volume of these thin layers.)

in semiconductors, which has higher concentrations on the neutral state at lower temperatures, $n(T)$ in these $\text{Al}_{0.77}\text{Ga}_{0.23}\text{N}$ layers is smallest at lowest temperatures and increases with increasing temperature (Fig. 3). Such behaviors at low temperature are typical for a DX center since the Fermi level E_F lies close to E_{DX} and below d^0 . As long as the process of thermal excitation of electrons from E_{DX} to E_d is dominating, $n(T)$ increases with increasing temperature. When the thermal energy can excite electrons from E_d to the first excited state, $n(T)$ will be governed by two competing processes. At higher temperatures, the removal of electrons from d^0 to the excited states by the latter process becomes dominant, leading to the decrease of $n(T)$.

From the best fits to the experimental data using Eq. (2), we obtained the energy distances ($E_d - E_F$) and ΔE : ($E_d - E_F$) ~ 2.8 – 3.2 meV and $\Delta E \sim 39$ – 44 meV for several $\text{Al}_{0.77}\text{Ga}_{0.23}\text{N}:\text{Si}$ samples including thick (1.2 and 2.1 μm) Si-doped layers which show a similar linewidth narrowing effect as reported in Refs. 21 and 29. In this case, the Fermi level is expected to locate close to the DX state and we can approximate $E_d - E_F \sim E_d - E_{DX}$. Locating only few meV above the DX^- level, the shallow d^0 state can be populated even at low temperatures, giving rise to the observed EPR signal in darkness similar to the behavior of a normal shallow donor. The main source of errors in determination of the donor concentration comes from the temperature measurements. In a He-flow cryostat, at very low temperatures (< 10 K), the sample temperature can be ~ 1 – 1.5 K higher than the reading from the temperature sensor which is placed separately 1.5–2 cm below the sample. (According to Boltzmann distribution and for an energy separation of 2.8 meV, $n(T)$ at 6 K is three times larger than that at 5 K.) This leads to higher $n(T)$ values than predicted as can be seen in the low temperature range in Fig. 3 (three data points at $T < 15$ K). Since the errors from temperature measurements and the linewidth narrowing effect are pronounced only at very low temperatures, their influence on the obtained results is small.

With the thickness of Si-doped layer obtained from SIMS and the number of spins measured by EPR, the spin density or the concentration of the donor on the d^0 state can be determined. The highest spin densities in both O-rich and Si-rich samples are about ~ 1.5 – $1.8 \times 10^{18} \text{ cm}^{-3}$ at 70–80 K and the larger number of spins in the Si-rich sample in Fig. 3 was due to a larger volume of Si-doped layers. In the Si-rich sample, $[\text{Si}]$ is significantly higher than $[\text{O}]$, and thus, the obtained energy separations should be related to Si.

Assuming that the neutral state and excited states of the donor follow the EMT, i.e., the E_d/i^2 rule ($i = 1, 2, \dots, n$) or $E_{2p} \sim E_d/4$ and $|\Delta E| = |E_{2p} - E_d| = |(E_d/4) - E_d| = 3E_d/4$, we can estimate the energy of the neutral state E_d from the observed ΔE value: $E_d = 4\Delta E/3 \sim 52$ – 59 meV for the studied $\text{Al}_{0.77}\text{Ga}_{0.23}\text{N}$ samples. Taking into account the formation of the DX^- state at ~ 3 meV below the shallow d^0 state, the activation energy E_a of Si is estimated to be: $E_a \sim E_{DX} \sim 55$ – 62 meV, which is considerably higher than the value of ~ 41 meV determined from the temperature dependence of the resistivity for Si in heavy Si-doped $\text{Al}_{0.77}\text{Ga}_{0.23}\text{N}$ (the Si concentration $\sim 4 \times 10^{19} \text{ cm}^{-3}$).¹⁶ (The observed activation energy of the donor in $\text{Al}_x\text{Ga}_{1-x}\text{N}$ measured by transport measurements was

found to depend strongly on the Si concentration in heavy Si-doped samples.³²⁾

In order to compare the obtained results with the EMT, we calculate the corresponding EMT values for an effective mass shallow donor in $\text{Al}_x\text{Ga}_{1-x}\text{N}$ with $x = 0.77$. The electron effective masses and dielectric constants for $\text{Al}_x\text{Ga}_{1-x}\text{N}$ were obtained by linear interpolation from the corresponding values in GaN (Refs. 33 and 34) and AlN (Refs. 33 and 35) and we followed the calculation used in Ref. 36. The EMT values obtained from calculations are: $\Delta E = 40.5$ meV and $E_d = 54.6$ meV. These EMT values are within the range of the corresponding energies determined by EPR ($\Delta E \sim 39$ – 44 meV and $E_d \sim 52$ – 59 meV), suggesting that in $\text{Al}_x\text{Ga}_{1-x}\text{N}$ with x up to 0.77, although Si becomes a DX center, its neutral state follows the EMT.

In summary, we have observed an EPR signal of a shallow donor in Si-doped $\text{Al}_{0.77}\text{Ga}_{0.23}\text{N}$ layers grown on 4H-SiC substrates in darkness at low temperatures. The temperature dependence of the donor concentration on the neutral state d^0 shows that Si already becomes DX center when $x \sim 0.77$ but behaves as a shallow donor due to a small separation (~ 3 meV) between the DX^- and d^0 states. The neutral donor state is determined to be $E_d \sim 52$ – 59 meV, which follows the EMT. Taking into account the DX formation, the activation energy of Si is estimated to be in the range of $E_a \sim 55$ – 62 meV. The energy separation between E_d and E_{DX} is much smaller than the accuracy of calculations and a slight difference in the charge correction between calculations would put the DX^- level above or below d^0 corresponding to a positive-U (shallow donor) or negative-U (DX) Si center. This explains the controversial results in calculations as well as the shallow donor behavior of Si in high-Al-content AlGa_N as indicated in transport measurements.

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