Silicon vacancy related T_{V2a} center in 4H-SiC

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Electron paramagnetic resonance (EPR) was used to study the T_{V2a} center in 4H-SiC, which was previously attributed to the isolated Si vacancy but with different charge states: neutral, single negative, and triple negative, corresponding to different spin states $S=1$, $S=3/2$, and $S=1/2$, respectively. The T_{V2a} EPR spectra observed in dark and under light illumination in as-grown high-purity semi-insulating 4H-SiC in the absence of the negatively charged Si vacancy (V_{Si}^-) provide direct evidence confirming the spin triplet ($S=1$) ground state of the center. A model with a triplet ground state and a singlet excited state is proposed to explain previously obtained results. The T_{V2a} center can be detected in as-grown material annealed at 1600 °C.

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I. INTRODUCTION

In irradiated n-type 4H- and 6H-SiC, several optically detected magnetic resonance (ODMR) spectra, labeled T_{V1x}, T_{V2x}, and T_{V3x} (with $x=a,b$), are often detected by monitoring a near-infrared photoluminescence (PL) band, which consists of zero-phonon lines (ZPL's) at 1.438 eV (labeled $V1$) and 1.352 eV ($V2$) in 4H-SiC and 1.433 eV ($V1$), 1.398 eV ($V2$), and 1.368 eV ($V3$) in 6H-SiC.[1] These ODMR centers have C_{3v} symmetry and an isotropic g value close to 2.003. These spectra were also observed by electron paramagnetic resonance (EPR) by several groups in 4H- and 6H-SiC.[2–5] One of these ODMR spectra, T_{V2a}, is related to the ZPL $V2$ at 1.352 eV in 4H-SiC.[1] In these works,[1–5] these spectra were suggested to originate from spin triplet states ($S=1$). The spectra were first observed by Vainer and Il'in in slightly n-type 6H-SiC (Ref. 4) (labeled $P3$ and $P5$) and were attributed to the far-distance vacancy pairs (V_{Si}-V_C) with different zero-field splitting (ZFS) parameter D depending on the distance between the two vacancies. Based on the hyperfine (HF) structure due to the interaction with 12 Si atoms in the next nearest neighbor (NNN) shell, the spin state, the correspondence between the number of the ZPL's (two in 4H- and three in 6H-SiC) and the number of inequivalent lattice sites in each polytype, and the possible level position in the band gap of 6H- and 4H-SiC, Sörman *et al.*[1] attributed the centers to the neutral isolated silicon vacancy (V_{Si}^0) at the quasicubic and hexagonal lattice sites. In a Zeeman study by Wagner *et al.*,[6] no splitting of any ZPL's in the magnetic field up to 5 T has been detected, indicating that the corresponding PL transitions are between the singlet states. The triplet spectra seen in ODMR were therefore tentatively explained as being detected indirectly via an excited triplet state located between the ground and excited singlet states.[6] However, recent EPR observations of the same spectra in 4H- and 6H-SiC under equilibrium conditions at low temperatures (1.2 to 4 K)[3,5] suggesting that the spectrum must arise from the ground state.

Recently, complete ligand hyperfine tensors of the interaction with four C atoms in the nearest neighbor (NN) shell have been determined,[2,7–9] supporting the isolated silicon vacancy model. There is an ambiguity about the spin $S=1$ of the T_{V2a} center since its EPR spectrum always appears together with the strong signal at $g\sim2.003$ of the undistorted negatively charged silicon vacancy (V_{Si}^-),[10–12] which may hide the central line of a possible $S=3/2$ spin. Recently, the spin state $S=3/2$ was indirectly determined for the T_{V2a} center in 4H-SiC by pulsed EPR,[7] whereas the high-field pulsed-EPR study by Orlinski *et al.*[3] suggested a spin $S=1$, in agreement with Sörman *et al.*[1] In a magnetic circular dichroism of the absorption (MCDA) and MCDA-detected EPR (MDCA-EPR) study of 6H- and 15R-SiC, Lingner *et al.*[13] observed the same PL band, but the MCDA-EPR spectrum measured on the $V3$ ZPL at 1.369 eV in 6H-SiC consists of only one line with $g=2.005$. This leads to a suggestion that the ground state of the silicon vacancy in SiC is triple negative (-3) with $S=1/2$.[13] Clarifying the spin state of the T_{V2a} center is important for understanding the electronic structure of this fundamental defect.

In this paper, we present our EPR studies of the T_{V2a} center in as-grown semi-insulating (SI) 4H-SiC. This center was observed in all the SI SiC wafers grown by high-temperature chemical vapor deposition (HTCVD), whereas the V_{Si}^- signal could not be detected in any sample. In the absence of the V_{Si}^- signal, the T_{V2a} spectrum with only two lines was detected, confirming the spin $S=1$ of the center. Based on our photo-EPR studies we will show that the T_{V2a} spectrum arises from a triplet ground state with a positive ZFS parameter D. The model will then be used to explain the Zeeman result in Ref. 6. The annealing behavior of the T_{V2a} center in as-grown material is presented.

II. EXPERIMENTS

In irradiated materials, the T_{V2a} spectrum always appears together with the dominating signal of V_{Si}^-, which has almost the same g value and similar HF structures. This makes the direct determination of the spin of the T_{V2a} center is difficult. In order to avoid the interference of the V_{Si}^- signal, in this study we used as-grown semi-insulating (SI) 4H-SiC substrates grown by HTCVD.[14] The typical concentration of some common residual impurities in HTCVD wafers measured by secondary ion mass spectrometry (SIMS) is N(nitrogen)$\sim7.6\times10^{15}$ cm^{-3}, N(boron)$\sim1.2\times10^{15}$ cm^{-3},

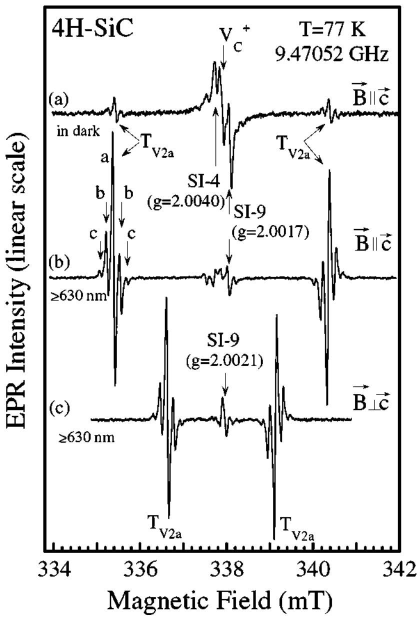

FIG. 1. EPR spectra in SI 4H-SiC grown by HTCVD measured for the magnetic field **B** along the c axis and at 77 K (a) in dark; (b) and (c) under illumination with light of wavelength $\lambda \geqslant 630$ nm.

N(aluminum)$\sim 6.6\times 10^{13}$ cm^{-3}, and N(vanadium)$\sim 8.0 \times 10^{12}$ cm^{-3}.[14] In this material, intrinsic defects are used to compensate nitrogen donor to obtain SI properties. The resistivity of the SI material at room temperature is in the range $\rho > 10^9$ Ω cm. EPR measurements were performed on a Bruker X-band ($\sim$9.47 GHz) EPR spectrometer. In photo-EPR measurements, a xenon lamp with a power of 150 W was used as the excitation source. Appropriate long-wavelength-passed and short-wavelength-passed optical filters were used to form band-passed filters at different wavelengths.

III. RESULTS AND DISCUSSION

The sample was mounted and cooled down to 77 K in the dark to obtain equilibrium conditions. Figure 1(a) shows the EPR spectrum measured in dark at 77 K for the magnetic field **B** along the c axis. As indicated in Fig. 1(a), the EPR signals of the positively charged carbon vacancy (V_C^+) (Ref. 15) and a new center, labeled SI-9, are predominant. Another line, labeled SI-4, also appears in this region of $g=2$ (Fig. 1). The SI-4 spectrum has C_{3v} symmetry and the g values are $g_\parallel = 2.0040\pm 0.0002$ and $g_\perp = 2.0024\pm 0.0002$.[16] The SI-9 center in Fig. 1 has C_{3v} symmetry with the g values $g_\parallel = 2.0017\pm 0.0002$ and $g_\perp = 2.0021\pm 0.0002$. We will not discuss further the SI-4 and SI-9 centers in this paper. In addition to these signals, two weak lines at $\sim$335.42 mT and $\sim$340.40 mT were detected [Fig. 1(a)]. Under illumination by light with photon energies $h\nu \geqslant 1.4$ eV ($\lambda \leqslant 890$ nm), the intensity of these two lines increase by two orders of magnitude. Figures 1(b) and 1(c) show the spectra measured for **B** parallel and perpendicular to the c axis, respectively, with a dramatic enhancement in intensity of these two lines under light illumination with $\lambda \geqslant 630$ nm ($h\nu \leqslant 1.97$ eV). [Light with this wavelength was used in order to suppress the V_C^+ (Ref. 17) and SI-4 signals.] The angular dependence study shows that these two lines belong to a spin $S=1$ center with an isotropic g value $g=2.0028\pm 0.0001$ and a ZFS parameter $D=46.75\times 10^{-4}$ cm^{-1} (or 70.1 ± 0.2 MHz). Within experimental error, both g and D values of this center are identical to that of the T_{V2a} center measured by Mizuochi *et al.*[7]

In this as-grown high-purity material, the linewidth (the distance between the maximum and minimum of the first derivative EPR line) is only $\sim$0.039 mT and a well-resolved isotropic HF structure is observed [Fig. 1(b)]. The splitting between the lines b is 0.305 mT, which is a half of the splitting between the lines c (0.61 mT). The intensity ratios between the HF lines and the main line are $b/a=0.25$ and $c/a=0.03$ if comparing the amplitude ($b/a=0.26$ and $c/a=0.034$ if comparing the integrated intensity). This HF structure is clearly the same structure due to the HF interaction with one and two ^{29}Si atoms (nuclear spin $I=1/2$ and a natural abundance of 4.67%) among 12 NNN Si atoms as observed for the T_{V2a} center in Refs. 1 and 7. The observed intensity ratios are in good agreement with the calculated ones: $b/a=0.273$ and $c/a=0.037$. From the same g and D values and the NNN ^{29}Si HF structure (within experimental errors), it is evident that we have observed the T_{V2a} center. As can be seen in Figs. 1(b) and 1(c), it is clear that the T_{V2a} spectrum consists of only two lines. There is no third central line of a possible spin $S=3/2$ corresponding to the previously assumed transition $|3/2,+1/2\rangle \leftrightarrow |3/2,-1/2\rangle$, which may be hidden by the strong V_{Si}^- signal always present in n-type irradiated material. The T_{V2a} and T_{V3a} spectra in 6H-SiC with clearly only two lines each have previously been observed in ODMR using resonant excitation at the corresponding ZPL's $V2$ and $V3$.[1] From the observations by ODMR[1] and EPR in this work, it is clear that the T_{V2a} spectrum arises from a spin triplet state ($S=1$).

We observed the T_{V2a} spectrum in all the studied samples (from few tens of different low doped n-type or SI wafers grown by HTCVD), but could not detected the undistorted (T_d symmetry, $S=3/2$) V_{Si}^- signal[7,11] in any sample even of n-type conductivity. We found that the T_{V2a} signal is often much weaker in slightly n-type samples than that in SI wafers. The failure in detection of the V_{Si}^- signal cannot be due to the measuring conditions since this center can easily be observed either in the dark or under light illumination. As also shown in Ref. 7, illuminating with a very powerful laser (up to 10 W) increases the T_{V2a} signal (up to the intensity level of V_{Si}^- line) but does not reduce the V_{Si}^- signal. The absence of the undistorted (T_d symmetry, $S=3/2$) V_{Si}^- signal in our case also suggests that T_{V2a} is not the V_{Si}^- center with the spin $S=3/2$ and C_{3v} symmetry as suggested in Ref. 7.

Both the low- and high-field lines of the T_{V2a} spectrum have the same phase corresponding to the absorption of microwave (MW) when measuring in the dark [Fig. 1(a)], but have opposite phases under light illumination [Figs. 1(b) and 1(c)]. This can be explained by the energy-level scheme illustrated in Fig. 2. Since the spectrum can be observed in dark and at low temperatures [even at 1.2–4 K (Refs. 3 and

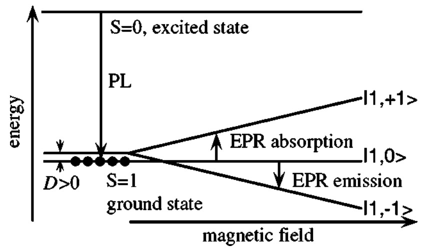

FIG. 2. The energy-level scheme for the T_{V2a} center in 4H-SiC. Under illumination by light with the photon energy $h\nu \geqslant 1.4$ eV, only the singlet $|1,0\rangle$ state is populated while the $|1,+1\rangle$ and $|1,-1\rangle$ states are empty since only the optical transition from the singlet $(S=0)$ excited state to the singlet sublevel $|1,0\rangle$ of the triplet ground state is allowed, giving rise to the ZPL (at 1.352 eV for the $V2$ ZPL) in PL. The low- and high-field EPR lines correspond to the MW absorption and emission, respectively.

5)] the triplet state must be the ground state. Under equilibrium conditions, all the states $|S,M_s\rangle$ of the triplet (the singlet $|1,0\rangle$ and doublets $|1,-1\rangle$ and $|1,+1\rangle$) are populated and the MW absorption occurs in both transitions $|1,0\rangle \leftrightarrow |1,+1\rangle$ and $|1,-1\rangle \leftrightarrow |1,0\rangle$. In low-frequency ($X$-band) EPR experiments, the splitting of these sublevels under the magnetic field is very small. At elevated temperatures, e.g., at 77 K as in our case, the difference in the population at these levels should be also small. Therefore, the EPR signal measured in the dark is rather weak [Fig. 1(a)]. Under illumination by light with the photon energies of ~ 1.352 eV (the energy of the ZPL $V2$ in 4H-SiC) or higher, electrons from the sublevels of the triplet ground state are pumped to the excited state of the center. The excited electrons rapidly relax down to the singlet $(S=0)$ excited state and then radiatively recombine to the singlet $|1,0\rangle$ level in the ground state, giving rise to the ZPL observed in PL. Only this singlet-singlet optical transition is allowed, while the optical transitions from the singlet excited state to the $|1,+1\rangle$ and $|1,-1\rangle$ sublevels of the ground states are forbidden. As a result, under light illumination only the singlet ground state $|1,0\rangle$ is populated, whereas the $|1,+1\rangle$ and $|1,-1\rangle$ states become empty (Fig. 2). Under this condition, the MW absorption transition $|1,0\rangle \rightarrow |1,+1\rangle$ and the MW emission transition $|1,0\rangle \rightarrow |1,-1\rangle$ will occur, giving rise to the EPR absorption and emission lines. Therefore, two EPR lines always have opposite phases. Since only the singlet state is populated under illumination, differences in population at sublevels of the triplet state become much larger compared to the case of equilibrium condition (in dark). This explains the dramatic increase of the EPR signal (approximately two orders of magnitude) under illumination.

As can be seen in Fig. 1, the EPR absorption gives rise to the low-field line. Therefore, the ZFS parameter D must be positive in this case. Since the transitions from the singlet $(S=0)$ excited state to the $|1,+1\rangle$ and $|1,-1\rangle$ sublevels of the triplet ground state are forbidden, only one ZPL line corresponding to the singlet-singlet transition can be detected. This explains why no splitting of the ZPL under the magnetic field could be observed in Zeeman experiments.[6] It should be noticed that our model with the triplet ground state and a singlet excited state is the only alternative which could explain the observed spin of the center and the Zeeman results. This is not an exceptional case for the T_{V2a} center but should be a typical property for all the spin $S=1$ centers with a triplet ground state and a singlet excited state. The V_C-C_{Si} pairs (or $P6/P7$ centers[4]) in 6H-SiC (Ref. 18) are typical examples. The V_C-C_{Si} centers have the spin $S=1$ and each configuration of these paired defects is related a ZPL of a PL band in the near-infrared spectral region.[18] Zeeman studies[19] showed that none of these ZPL's split under the magnetic field. In the case of spin $S=3/2$ ground state as suggested in Ref. 7, there would be more than one optical transition from the excited state (regardless of its spin multiplicity) to the states $|3/2,-3/2\rangle$, $|3/2,-1/2\rangle$, $|3/2,+1/2\rangle$, and $|3/2,+3/2\rangle$ of the quartet ground state detected in PL and hence the splitting of the ZPL under magnetic field would be observable. The explanation for the nonsplitting of the ZPL using an additional nonradiative level in between the excited and ground states, and assuming the same splitting in the triplet excited state and triplet ground state in Ref. 3, is also not satisfactory. It is possible in principle but difficult to have such coincidences of the energy levels in different SiC polytypes. Even for the cases of both the excited and ground triplet states having the same splitting, all the sublevels on the ground state will be populated under light illumination since more than one optical transition from the excited triplet state are allowed. This would lead to no difference in the intensity of the T_{V2a} signal when measuring in the dark or under light illumination, which is in disagreement with the observation in Ref. 7 and in our work (the intensity of T_{V2a} signal increases by about two orders of magnitude under light illumination). It should be noticed that terms ''low-field line'' and ''high-field line'' are applicable for the angles θ of the magnetic field in the range 0–54.74° (with respect to the direction of the c axis). For a spin $S=1$ center, the splitting induced by the crystal field becomes zero at the angle $\theta = 54.74°$ and the corresponding energy term, $[D(3\cos^2\theta - 1)]$, changes to the opposite sign afterward. Therefore at angles in the range 54.74°–90° the ''high-field'' line will becomes the ''low-field'' line and vice versa.

In Ref. 13, a very broad, structureless MCDA-EPR line with a g value of 2.005 was detected at the $V3$ ZPL at 1.359 eV in 6H-SiC. A similar spectrum was also detected in 15R-SiC.[13] For some unknown reasons, the ZPL $V2$ related to the T_{V2a} center was not observed in their MCDA experiments. The MCDA-EPR spectrum is completely different from that observed in EPR and ODMR. As can be seen in Fig. 2 of Ref. 13, the asymmetric MCDA-EPR line has a linewidth at half maximum of about 50 G, which is larger than the largest splitting induced by the crystal field of the T_{V3a} and T_{V3b} centers in 6H-SiC [~ 20 G for T_{V3a} and ~ 40 G for T_{V3b} (Ref. 1)]. If the T_{V3a} and T_{V3b} centers were actually observed in their experiments, both the low- and high-field lines should be well inside the broad MCDA-EPR line at all the angles of the magnetic field and cannot be resolved. The assignment of such a broad MCDA-EPR line to a single line from a spin $S=1/2$ center in Ref. 13 is irrelevant. We believe that the broad MCDA-EPR line is due to the unresolved lines of the T_{V3a} and T_{V3b} centers.

It is also possible that the MCDA-EPR signal is related to

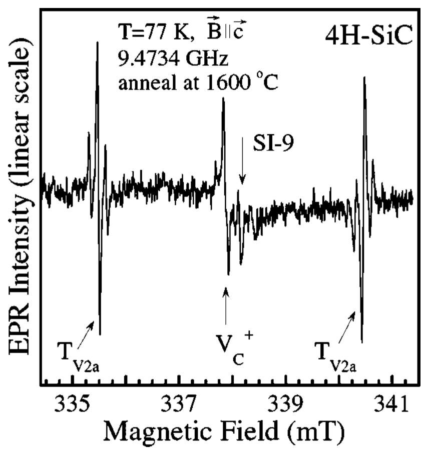

FIG. 3. EPR spectrum in a SI 4H-SiC sample annealed at 1600 °C for 30 min measured for the magnetic field **B** along the *c* axis under white light illumination.

other defects but not to the $V3$ PL line. In MCDA-EPR measurements, the sample is excited by light from a monochromator after going through different polarizers and filters. Such excitation is usually very weak. In some MCDA-EPR experiments, additional laser light in the visible region is also used.[18] If the additional visible light is used, then the absorption and emission processes occur also to other defects present in the sample. In irradiated material, there are strong and broad MCDA lines appearing in the region of 1.45–1.55 eV (Refs. 13 and 20) and their broad phonon bands may overlap with the $V1$-$V3$ lines and contribute to the MCDA-EPR signal. We know from our ODMR experiments that whenever above band gap light is used for excitation, then a broad ODMR line (the middle line seen in the ODMR spectra in Ref. 1 and related to a so-called lifetime limiting defect[21]) with an isotropic g value of 2.01 ± 0.01 will be detected together with vacancy-related signals. It is also possible that the MCDA-EPR line detected in Ref. 13 is related to this defect but not to the $V3$ ZPL. The data from EPR and ODMR experiments are more reliable. In the ODMR experiments using resonance excitation,[1] a laser line from a tunable Ti-doped sapphire laser was tuned exactly to excite only one transition corresponding to one ZPL and no other absorptions occurred. The observed ODMR[1,8,9] and EPR (Refs. 2, 7, and our work) spectra show clear HF structures, which unambiguously confirm the presence of a silicon vacancy in the defect.

Figure 3 shows the EPR spectrum in a SI 4H-SiC sample annealed at 1600 °C for 30 min in Ar gas. The spectrum was recorded for **B** along the *c* axis and under white light illumination. The T_{V2a} signal reduces but can be seen with its clear HF structure. In addition to T_{V2a}, the signals of V_C^+ and SI-9 centers were also detected. The annealing was performed in a CVD system so the sample was slowly cooled down from 1600 °C. The observation of the T_{V2a} center after annealing at such high temperatures is unexpected since it is known from previous studies that the center is annealed out at around 750 °C (Ref. 1) and has a similar annealing behavior as the V_{Si}^- signal.[10] From this annealing behavior and the fact that the V_{Si}^- signal has not been detected in any as-grown sample, it is possible that the T_{V2a} center may be not related to the isolated silicon vacancy as generally believed. The recent observation of the ligand HF structure due to the interaction with four nearest C neighbors[2,7–9] is strong support for the isolated V_{Si} model, but still cannot rule out the possibility of long-distance pairs along the *c* axis, which are possible in hexagonal SiC polytypes. To our knowledge, the V_{Si}^- signal has not been observed by magnetic resonance in as-grown materials. Its absence may be due to the process in which a Si vacancy traps one of its C neighbors to form a V_C-C_{Si} pair.[18] The energy barrier required for this process is about 1.7 eV,[18] which corresponds to the annealing temperature ~750 °C of the silicon vacancy.[10] This process can happen during the cooling down of the crystal after growth from the growth temperature (normally above 2200 °C in HTCVD or in sublimation techniques). In fact, we observe the EPR spectra of the V_C-C_{Si} pairs as the dominating signals in SI SiC wafers grown by HTCVD and by physical vapor transport (PVT) (see also Refs. 22 and 23).

In summary, we have observed the EPR spectrum of the T_{V2a} center in as-grown SI 4H-SiC grown by HTCVD in the absence of the undistorted V_{Si}^- signal. The spectrum with only two lines confirms the spin $S=1$ of the center. The T_{V2a} spectrum arises from a spin triplet ground state, and its associated PL line corresponds to the radiative transition from a singlet excited state to the singlet sublevel of the triplet ground state. The obtained result also explains the nonsplitting of the ZPL's of the center in previous Zeeman studies. The T_{V2a} center is present in all as-grown SI material grown by HTCVD with the concentration comparable to that of shallow dopants (N and B) and may play an important role in carrier compensation processes. The T_{V2a} signal reduces significantly but can still be detected after annealing at 1600 °C.

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