Isochronal annealing studies of *n*-type 6*H*-SiC with positron lifetime spectroscopy

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n-type 6H silicon carbide has been studied using positron lifetime spectroscopy with isochronal annealing temperatures of 400, 650, 900, 1200, and 1400 °C. In the as-grown sample, we have identified the V_{Si} vacancy, the V_CV_{Si} divacancy, and probably the V_C vacancy. The silicon vacancy and the carbon vacancy were found to anneal out in the temperature range 400–650 °C. The V_CV_{Si} divacancy was found to persist at an annealing temperature of 1400 °C.

I. INTRODUCTION

Silicon carbide is a wide-band-gap semiconductor which has attracted considerable attention as a material for high-temperature, high-power, and high-frequency devices. As defects play an important role in influencing the electrical properties of the material, defects in 6H-SiC have been extensively studied with the use of different techniques like deep-level transient spectroscopy (DLTS),[1–4] electron-spin resonance (ESR),[5] positron annihilation spectroscopy,[6–11] and photoluminescence.[12] A detailed review of the electrical and optical characterization of SiC was given by Pensl and Choyke.[13]

With the use of DLTS, a number of deep levels in the SiC band gap have been found in as-grown or irradiated samples. For example, Z_1/Z_2 and E_1/E_2 levels have been observed in many *n*-type 6H SiC studies, like those of Zhang *et al.*,[14] Hemmingsson *et al.*,[2] Gong *et al.*,[3] and Aboelfotoh and Doyle.[4] However, regarding the interpretation of the microstructures associated with these deep levels, there are presently no consensus. Zhang *et al.*[14] reported a double peak at $E_C-0.62$ eV and $E_C-0.64$ eV which they called Z_1/Z_2. The Z_1/Z_2 defect level was proposed to be due to the V_CV_{Si} divacancy. The V_CV_{Si} site was also previously observed in an ESR study, and it was related to the D_1 center observed in a photoluminescence study[12] because they were all stable at an annealing temperature of 1700 °C. However, with DLTS, Aboelfotoh and Doyle[4] also observed a double peak at the same position, but the defect was found to anneal out at a temperature of 850 °C and was thus suggested to be a silicon vacancy. Gong *et al.*[3] observed a signal close to the Z_1/Z_2 position, but failed to obtain a reliable position as the signal was too small. The peak was found to be annealed out at a temperature lower than 1150 °C. The authors also pointed out the problem of simply assigning the Z_1/Z_2 levels to the V_CV_{Si} divacancy, as its generation rate in the electron irradiation process varies considerably from sample to sample in different studies, although the electron implanting energies were similar (see, for example, Hemmingsson *et al.*[2] and Zhang *et al.*[14]).

For the case of E_1/E_2, most studies agree that this level doublet is not annealed out even at temperatures as high as 1600 °C but the assignment of the defect microstructure differed between investigators.[1,3,4,14] Aboelfotoh and Doyle,[4] for example, found deep acceptors at $E_C-0.34$ eV and $E_C-0.41$ eV, and identified them as E_1/E_2. The deep acceptors were assigned to a negatively charged carbon vacancy occupying different sites, and it was suggested that such sites were stable after a 1000 °C annealing. Gong *et al.*,[3] found deep acceptors ED_3 and ED_4 at $E_C-0.36$ eV and $E_C-0.44$ eV respectively. Again, it was pointed out that the ED_3 and ED_4 levels were the E_1/E_2 levels as seen by others. Moreover, the $E_C-0.36$ eV was found to consist of two overlapping peaks ED_{3L} and ED_{3H}. The ED_3L level was found to be annealed out at a temperature of 700 °C. It was suggested that the deep levels ED_{3H} and ED_4 were attributed to the V_CV_{Si} divacancy, as they were not completely annealed out at a high temperature of 1600 °C.

Positron lifetime spectroscopy has been used extensively as a nondestructive probe in semiconductors because of its excellent sensitivity toward neutral or negatively charged vacancies.[15–17] The principle of the positron lifetime technique is based on the fact that vacancies in the semiconductor trap implant positrons into localized states which have a longer positron lifetime compared to delocalized positrons, because the electron density at the vacancy site is lower. Different vacancies can be identified by their characteristic positron lifetimes, which can thus serve as a fingerprint of the defect. Moreover, additional information, such as defect concentration, charge state, and ionization energies of different charge states can often be extracted from positron lifetime data.

In a previous study,[11] we examined as-grown *n* and *p*-type 6H-SiC with the positron lifetime technique. In *p*-type material, a 225-ps component was found, and was attributed to positron annihilating in neutral V_CV_{Si} divacancies. The V_CV_{Si} concentration was estimated to be 4×10^{16} cm^{-3}. For *n*-type 6H-SiC, a 200-ps lifetime component was observed, and this was proposed to be the overlapping of coexisting silicon vacancy and V_CV_{Si} divacancy components, which have characteristic lifetimes smaller than and larger than 200 ps respectively. A positron shallow trap was also identified from its low-temperature competition with vacancy trapping. This Rydberg-like state of the positron results from the positron binding with a negative impurity or acceptor. It was also pointed out that in *p*-type material, the Fermi level was close to the valance band, which probably made the silicon vacancy positively charged the acceptor neutrally charged;

thus neither is capable of trapping positrons.

In the present study, we aim to examine previous proposals on defect assignment, and also to study the annealing behavior of the vacancy defects in the material. We have performed positron lifetime measurements on *n*-type 6H SiC annealed at 400, 650, 900, 1200, and 1400 °C. At each of the annealing temperatures, positron lifetime spectra were accumulated at temperatures ranging from 20 K to room temperature.

II. EXPERIMENT

The material used in the study was a Lely-grown nitrogen-doped research grade *n*-type 6H silicon carbide wafer purchased from Cree Research Inc. The doping concentration of the wafer was $1.2\times10^{18}\ \mathrm{cm}^{-3}$. Four samples with a size of $5\times8\ \mathrm{mm}^2$ were cut from the wafer, degreased with acetone and methanol, and then rinsed in deionized water. The positron radioactive source was made by encapsulating 20-μCi^{22}NaCl with a kapton foil. The source foil was then sandwiched by four pieces of sample with two pieces at a side. The sample assembly was then installed into a closed-cycle He fridge. Each of the lifetime spectra were accumulated up to 4×10^6 counts, with the standard fast-fast lifetime spectrometer having a resolution of 220 ps. Lifetime measurements were performed on samples annealed at 400, 650, 900, 1200, and 1400 °C. The annealing process was performed in a forming gas environment for a period of 30 min. For each of the annealing temperatures, lifetime spectra were collected at different temperatures from 20 K to room temperature.

III. RESULTS AND ANALYSIS

A positron lifetime spectrum is a linear combination of exponential terms contributing from the corresponding annihilating sites, i.e.,

$$S(t)=N\sum_i \frac{I_i}{\tau_i}\exp\left(\frac{-t}{\tau_i}\right), \tag{1}$$

where τ_i and I_i are the characteristic lifetime and the intensity of the corresponding component, respectively. The lifetime spectra were decomposed into components by the program POSITRONFIT,[18] with all the parameters free.

It was found that in all of the spectra, a three-component fit could give a good representation of the spectra. A nonvarying long lifetime component, having a lifetime value of about 1000 ps at the 1.5% level, was found to exist in spectra irrespective of the annealing temperature and the measurement temperature. This lifetime component was thus attributed to positronium annihilation at voids or at the surface of the sample, and does not fall within the scope of interest of the present study. This component was thus removed, and data analyzed with just two components.

The fitted results of the lifetime parameters τ_2, τ_1 and I_2, as a functions of the measurement temperature for the as-grown sample, and that after the 900 °C anneal, are shown in Fig. 1. The sample used in the present study was the same sample as that employed in a previous study.[11] For the as-grown and the 900 °C annealed spectra, the values of τ_2, the characteristic lifetime of the positron trapping center, were

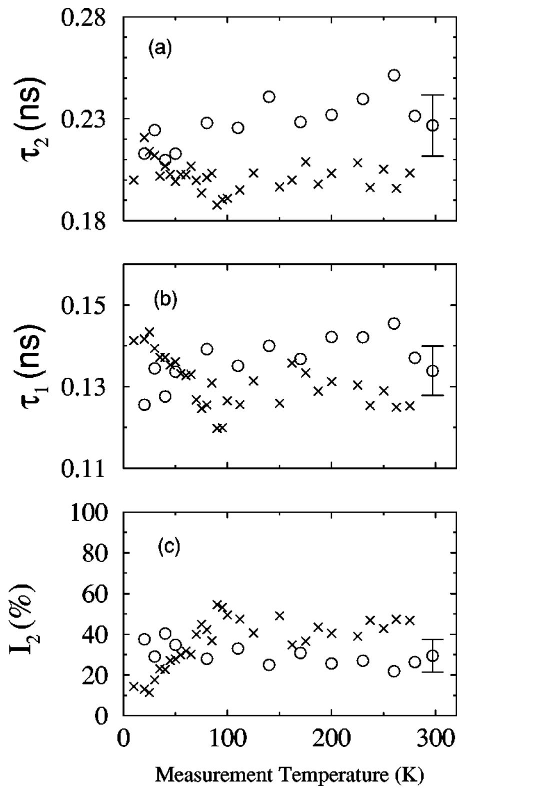

FIG. 1. Fitted results of τ_2, τ_1, and I_2 as functions of the measuring temperature. The cross and the circle are the results obtained from the as-grown and the 900 °C annealed spectra, respectively.

essentially constant (with values of about 200 and 230 ps, respectively), at all of the measurement temperatures. This behavior was also true for all other annealing temperatures. This constancy, which was also observed in the annealed samples, implies that there is no change in the defect microstructure as the measurement temperature is lowered from room temperature. The average τ_2 values plotted as a function of annealing temperature are shown in Fig. 2(a), from which it is seen that the mean τ_2 value varies with the annealing temperature. This variation will be discussed below.

In order to eliminate the correlation between the fitting parameters in the fitting process, the spectra were refitted by fixing the τ_2 values according to the values shown in Fig. 2(a). The resulting I_2 and τ_1 variation, plotted as a function of the measurement temperature for the different annealed samples, is shown in Fig. 3. For the as-grown sample it is observed that the long lifetime intensity I_2 stays at a constant value of about 40% for temperatures above 80 K, but falls to about 12% as the temperature decreases to 10 K. This dropping of I_2 at low temperature is accompanied by an increase of τ_1. In Ref. 11, this low-temperature behavior was attributed to a positron shallow trap. A positron shallow trap is a Rydberg state formed by the positron binding to a negatively charged impurity ion, such states having been observed in both GaAs (Ref. 19) and Si.[20] The binding energy of this state varies typically from 10 to 100 meV. Thus the state is thermally detrapped at room temperature, and only plays a significant role at low temperature (i.e., $T<80$ K). From Fig. 3 it is seen that this low-temperature behavior vanishes when the sample is annealed to 650 °C.

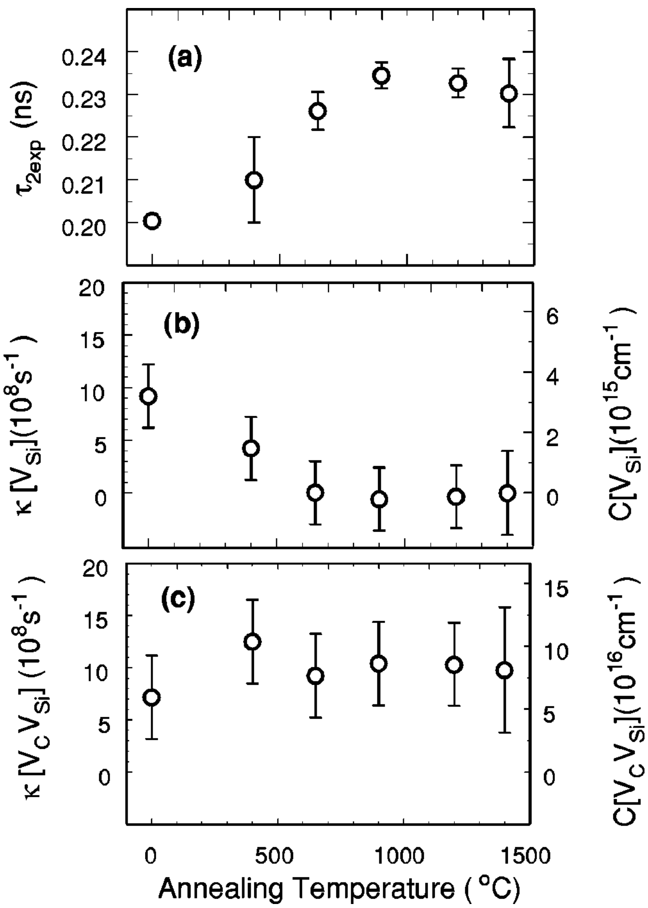

FIG. 2. (a) Characteristic lifetime τ_2 and (b) positron trapping rate and concentration of V_{Si}, and (c) positron trapping rate and concentration of $V_C V_{Si}$ measured at room temperature as functions of the annealing temperature.

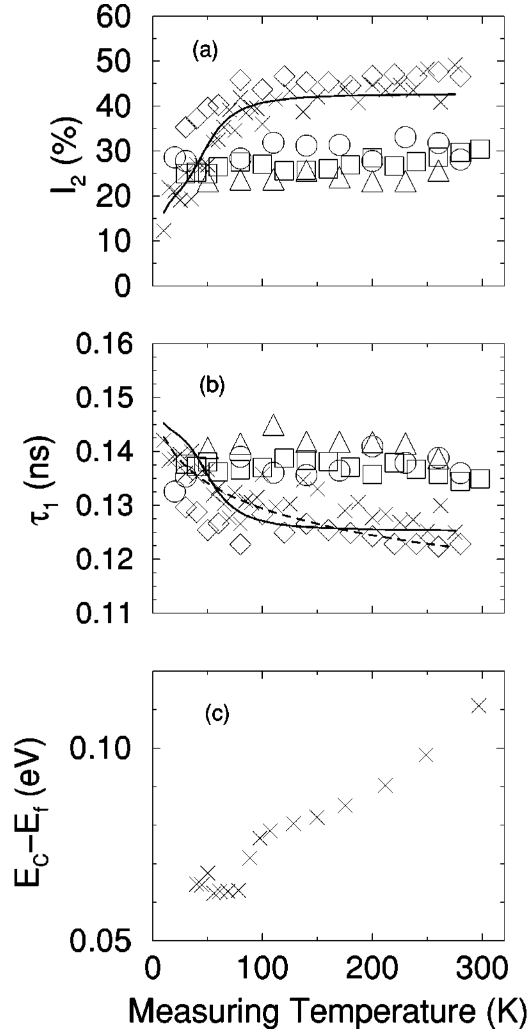

FIG. 3. I_2 and τ_1 as functions of the measuring temperature with τ_2 fixed as described in the text in order to eliminate the correlation between the fitting parameters. The cross, diamond, square, circle, and triangle represent the data obtained from the as-grown, 400°, 650°, 900 °C, and 1200° annealed samples. The solid and dashed lines are the modeled curves, assuming a shallow trap and a negatively charged carbon vacancy, respectively.

A. Long lifetime component ($\tau_2 = 200-232$ ps)

In investigating the annealing behavior of positron trapping, we first consider the variation of τ_2 with annealing temperature. At each annealing temperature, an average value of τ_2 was obtained by averaging the temperature measurement scan data from 80 K to room temperature. With reference to Fig. 2, for the as-grown sample, it can be seen that the defect lifetime τ_2 is 200 ± 9 ps. This then increases up to about 225 ps as the annealing temperature is increased up to 650 °C. Only a minor increase in τ_2 is then observed as the annealing temperature further increases up to 900 °C, at which temperature τ_2 has saturated at about 232 ps.

Brauer *et al.*[21] calculated different vacancy type defects in 6H-SiC with the use of the linear muffin orbital in the atomic spheres approximation. The lifetime values of the carbon vacancy, the silicon vacancy, and the Si+C divacancy were calculated to be 153, 191, and 212 ps, respectively. For experimental work, positron lifetime values of V_{Si} and $V_C V_{Si}$ have been reported to be 183 ps (Ref. 8) and 235 ps,[10] respectively. According to our previous study,[11] the 200-ps lifetime component found in the as-grown sample was the overall result of positrons annihilating in the V_{Si} monovacancy and the $V_C V_{Si}$ divacancy coexisting in the sample, the lifetime values of V_{Si} and $V_C V_{Si}$ being too close to be resolved. This being the case the increase of the observed τ_2 upon annealing up to 650 °C can be explained by the annealing out of the silicon vacancy. The τ_2 value of 232 ps observed at annealing temperatures at 650–1200 °C is thus understood as the lifetime of $V_C V_{Si}$. The disappearance of V_{Si} after annealing to 650 °C is close to the observation of Ref. 22, where the F spectrum in an ESR measurement, attributed to V_{Si}, was annealed out at 750 °C, which is close to the value observed here.

It is possible to calculate the concentrations of V_{Si} and $V_C V_{Si}$ as functions of the annealing temperature. In the higher measurement temperature range, which is free from low temperature traps ($T \geqslant 80$ K), positron trapping into the silicon vacancy and the Si+C divacancy can be described by the simple trapping model with two competing trapping centers. The solution of this model is given by[15]

$$\lambda_{D2} = 1/\tau_{V_{Si}}, \tag{2}$$

$$\lambda_{D3} = 1/\tau_{V_C V_{Si}}, \tag{3}$$

$$I_{D2}=\frac{\kappa_{V_{Si}}}{\lambda_b-\lambda_{V_{Si}}+\kappa_{V_{Si}}+\kappa_{V_C V_{Si}}}, \tag{4}$$

$$I_{D3}=\frac{\kappa_{V_C V_{Si}}}{\lambda_b-\lambda_{V_C V_{Si}}+\kappa_{V_{Si}}+\kappa_{V_C V_{Si}}}, \tag{5}$$

where I_{D2} and I_{D3} are the intensities of the two lifetime components corresponding to the silicon vacancy and the Si+C divacancy, respectively, and the λ_{Di}'s are the respective annihilation rates from the two defect states. λ_b is the positron annihilation rate of the bulk. $\kappa_{V_{Si}}$ and $\kappa_{V_C VSi}$ are the trapping rates into V_{Si} and $V_C V_{Si}$, respectively.

In the present case, as the lifetime values of the two positron trapping centers are too close to be separated, the observed lifetime would be equal to[15]

$$\tau_{2,exp}=\frac{I_{D2}}{I_{D2}+I_{D3}}\times\frac{1}{\lambda_{D2}}+\frac{I_{D3}}{I_{D2}+I_{D3}}\times\frac{1}{\lambda_{D3}}. \tag{6}$$

Thus the values of $\kappa_{V_{Si}}$ and $\kappa_{V_C V_{Si}}$ can be found by solving Eqs. (2)–(6). The lifetime value of $V_C V_{Si}$ has been reported in studies of ion-implanted 6H SiC (Ref. 21) and as-grown *p*-type 6H-SiC,[11] and was found to be 235 and 225 ps respectively. The lifetime value of the silicon vacancy was reported to be 185 ps (Ref. 10) and 183 ps.[8] Therefore, the trapping rates into V_{Si} and $V_C V_{Si}$ at room temperature as functions of the annealing temperature can be calculated by taking $\tau_{V_{Si}}=184$ ps and $\tau_{V_C V_{Si}}=230$ ps, which are the average values of these previous results.

The calculated room-temperature trapping rates of V_{Si} and $V_C V_{Si}$ are plotted as function of the annealing temperature in Figs. 2(b) and 2(c). The positron trapping rate is proportional to the defect concentration $\kappa_i=\mu_i\times c_i$, where μ_i is the specific trapping coefficient of the defect *i*.[15] From Fig. 2, it is seen that the trapping rate (and thus the concentration) of V_{Si} dropped from $\kappa_{V_{Si}}=9.2\times10^8$ s^{-1} to zero for annealing temperatures of 650 °C or above. In contrast, the trapping rate of $V_C V_{Si}$ remains essentially constant at about $\kappa_{V_C V_{Si}}=10^9 s^{-1}$, within statistical error over the whole annealing temperature range.

The vacancy and divacancy concentrations can be calculated if the values of their specific trapping coefficient $\mu_{V_{Si}}$ and $\mu_{V_C V_{Si}}$ are known. Although there are no reports on the experimental or theoretical values of the $\mu_{V_{Si}}$ and $\mu_{V_C V_{Si}}$ in SiC, the Si vacancy and Si+C divacancy concentrations can still be estimated with the use of the previous findings in Si. As it was reported that $\mu^0_{V_2}:\mu^-_{V_2}=1:35$, and $\mu^-_{V_2}=4\times10^{16}$ s^{-1} in Si,[23,24] the specific trapping coefficient of neutral Si divacancy in Si was found to be 1.14×10^{15} s^{-1}. With this value, the concentration of the $V_C V_{Si}$ divacancy can be estimated as $c_{V_C V_{Si}}=8.3\times10^{16}$ cm^{-3}.

In calculating the concentration of silicon vacancy, we assume the silicon vacancy in the *n*-type 6H SiC sample is negatively charged. This seems reasonable based on the following: (i) A negatively charged silicon vacancy was observed in 4H SiC with the use of ESR.[25] and (ii) According to the calculation of Zywietz *et al.*,[26] the (−/0) level is close to the midgap position, and the Fermi-level position of the present *n*-type sample is close to the conduction band. Mäkinen *et al.*[27] reported a value of about $\mu_V=10^{17}$ s^{-1} for the negatively charged Si vacancy at $T=20$ K. For the negatively charged vacancy, the specific trapping coefficient follows a $T^{-0.5}$ dependence.[15] Based on these assumptions, the concentration of V_{Si} in the as-grown sample is estimated to be 3.2×10^{15} cm^{-3}.

B. Low-temperature trap: shallow trap or carbon vacancy?

For the as-grown sample, as shown in Fig. 3, I_2 was found to decrease for temperatures below 80 K. This decrease in I_2 corresponded with an increase in the fitted value of τ_1. This effect cannot be explained by the change of the charge state of the vacancy defect induced by a decrease of the measurement temperature, since a lowering of the temperature moves the Fermi level closer to the conduction band, which would imply a more negatively charged defect and thus a increase of the I_2 value. Neither can it be explained by the temperature dependence of the rate of positron trapping into the vacancy states discussed in Sec. III A, because neutral and negatively charged vacancies have either a constant or $T^{-0.5}$ dependence on temperature, respectively, in which case a decreasing I_2 with decreasing temperature cannot be obtained. In the following sections, we discuss some possibilities that may introduce such a low-temperature behavior, namely, (a) the presence of a negatively charged impurity acting as a positron shallow trap, (b) the charge state transition of the carbon vacancy V_C induced by the change of the Fermi-level position, and (c) the $T^{-0.5}$ temperature dependence of the positron trapping coefficient of V_C.

1. Positron shallow trap

In Ref. 11, we attributed this low-temperature effect to a positron shallow trap. A positron shallow trap is a hydrogen-like state formed by a positron binding to a negative ion. The binding energy of such a trap is typically about 10–100 meV, which is small compared to that of about 1 eV for a vacancy type defect. As the positron is weakly bound to the negative ion, the lifetime of the positron is very close to the bulk state positron lifetime. As the temperature decreases, thermal detrapping of the positron from the shallow trap becomes insignificant, and thus more positrons annihilate in the shallow trap state. This is reflected by the decrease of I_2 and the corresponding increase of τ_1, as shown in Fig. 3. According to a model involving competitive positron trapping into a shallow trap and a vacancy-type defect, which is presented in Ref. 11, the concentration and the binding energy of the shallow trap was found to be $c_{st}=8\times10^{15}$ cm^{-3} and $E_b=13$ meV. The fitted curves of I_2 and τ_1 and shown as the solid lines in Figs. 3(a) and 3(b), respectively. Although we can explain the low-temperature trapping by the shallow trap model, it is important in making a comprehensive discussion to consider some other alternative possibilities.

2. Charge state transition of V_C

Brauer *et al.*[21] calculated the lifetime value of V_C as 153 ps, which, like the shallow trap state, is very close to the bulk lifetime. This implies that even if a carbon vacancy exists, we may not be able to separate it from the observed τ_1 component. If the carbon vacancy has an ionization level $(0/+)$ or $(-/0)$ close to the conduction band, then the moving up of the Fermi level induced by the lowering of the temperature may cause the carbon vacancy to capture an electron, thus becoming more negative and positron attractive. The increased positron trapping into the carbon vacancy would result in a decrease of V_{Si} and $V_C V_{Si}$ trapping, and a reduced value of I_2.

In order to test the validity of this interpretation, Hall-effect measurements were performed on the sample at temperatures ranging from 30 to 300 K. The Fermi-level position was calculated using the equation $n = N_C \exp[-(E_C - E_F)/kT]$, where N_C is the effective density of states of conduction band, and $N_C = 2g(2\pi m_d^* kT)^{3/2} h^{-3}$ and $m^*_d = (m^{*2}_t m^*_l)^{1/3}$.[28] The values of m^*_t and m^*_l were taken as 0.25 and 1.5 (see Ref. 28, and references therein). The calculated Fermi-level position $E_C - E_F$ is shown in Fig. 3(c). It was found that $E_C - E_F$ in general dropped with decreasing temperature from 0.11 eV at 300 K. A transition was found at the temperature range of 80–100 K, in which $E_C - E_F$ decreased from 0.078 to 0.053 eV. At lower temperatures, the Fermi level was found to pin at $E_C - E_F = 0.053$ eV. The transition found at $T = 100$ K results from a ''freeze out'' of the carriers onto the main nitrogen donor. The rapid change of the Fermi level at this temperature may imply a charge state transition of a defect. Although this transition temperature is close to the transition temperature of the positron lifetime data, it is probable that they are not the same transition. This is because the transition seen in the Hall measurement is fully completed by $T = 80$ K, and then the Fermi level remains constant, in contrast to the variation in I_2 and τ_1 which persists down to 10 K.

Zywietz *et al.* performed first-principles calculations on the neutral and charged carbon vacancy in 4H-SiC.[26] According to their calculation, the carbon vacancy exhibited a negative-U behavior. The ionization levels (-/0) and (- -/-) were at $E_V + 2.81$ eV ($E_C - E_F = 0.29$ eV) and $E_V + 2.38$ eV ($E_C - E_F = 0.72$ eV), respectively. As compared with the measured Fermi-level position shown in Fig. 3(c), this implies that V_C in the sample is always in a (- -) charge state, and probably has no charge transition in our measuring temperature range. Furthermore, Dannefaer *et al.*[7] studied electron-irradiated 6H-SiC with positron lifetime and Doppler broadening techniques. A 160-ps lifetime component was found, and attributed to the carbon vacancy. From their reported data down to a temperature of 30 K, no charge state transition was observed. Thus we tend not to favor this model, which involves the transition of the V_C charge state as the temperature decreases.

3. $T^{-0.5}$ dependence of positron trapping coefficient of negatively charged V_C

Another effect requiring consideration is whether the carbon vacancy in a negatively charged state could bring about the observed low-temperature I_2 and τ_1 behaviors. As mentioned in Sec. III B 2, according to the calculation of Zywietz *et al.*,[26] and our measured Fermi-level position, the charge state of the V_C in our sample is 2, which implies a positron trapping coefficient dependence of $T^{-0.5}$.[16] As the lifetime value of positron annihilating in V_C is very close to that of the bulk, this lifetime component could be indistinguishable from the bulk lifetime. Thus, if a carbon vacancy does exist, as the measurement temperature decreases the positron trapping rate into V_C increases; consequently I_2 decreases and τ_1 increases in much the same manner as in a nonopen volume positron shallow trap, as discussed in Sec. III A.

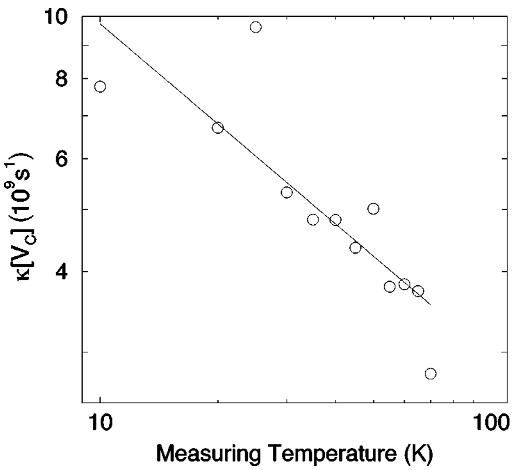

FIG. 4. Rate of positron trapping into the negatively charged carbon vacancy as a function of the measuring temperature. The fitted slope of the straight line is -0.52 ± 0.01, which is close to the expected value -0.5 of a negatively charged vacancy.

In a case when the lifetime value of a positron trapping center (i.e., V_C) is indistinguishable from the bulk lifetime and there is another well-separable long lifetime component, the positron trapping rate into the carbon vacancy κ_{V_C} and the observed $\tau_{1,expt}$ can be expressed as[15]

$$\kappa_{V_C} = \frac{\tau_{1,expt}(\lambda_b - I_{2,expt}\lambda_{2,eff}) - I_{1,expt}}{\tau_{V_C} - \tau_{1,expt}}, \tag{7}$$

$$\kappa_{2,eff} = \frac{I_{2,expt}}{I_{1,expt}}(\lambda_b - \lambda_{2,eff} + \kappa_{V_C}), \tag{8}$$

$$\tau_{1,mod} = \frac{1 + (\kappa_{V_C}/\lambda_{V_C})[1 + \kappa_{2,eff}/(\lambda_b - \lambda_{2,eff} + \kappa_{V_C})]}{\lambda_b + \kappa_{V_C} + \kappa_{2,eff}}. \tag{9}$$

We have calculated the positron trapping rate into the carbon vacancy κ_{V_C} at $T < 80$ K using Eq. (7), while taking $\tau_{V_C} = 153$ ps, $\tau_b = 141$ ps,[21] and $\tau_{2,eff} = 200$ ps. The results of $\log \kappa_{V_C}$ as a function of the $\log T$ is plotted in Fig. 4. The slope of the fitted straight line in Fig. 4 was found to be -0.52 ± 0.01, which is very close to the predicted value of -0.5 with our present assumption. With these results, the modeled $\tau_{1,mod}$ was calculated and plotted in Fig. 3(b). The modeled curve also shows a good representation of the experimental data.

We now consider whether the annealing behavior as shown in Figs. 3(a) and 3(b) can help in distinguishing any of the above models. As the annealing temperature goes up to 650 °C, the observed I_2 becomes independent of the temperature, indicating that defect center responsible for the low-temperature anomaly has been annealed out. No further change was observed as the annealing increased up to 1400 °C. In previous sections, the low-temperature behavior observed in samples with annealing temperatures lower than or equal to 400 °C originated from two possibilities, namely, the presence of a positron shallow trap or the presence of a negative carbon vacancy. The annealing out of this low-temperature defect site is thus due to the removal of either a carbon vacancy or a charge neutralization reaction of a positron shallow trapping negative impurity.

IV. DISCUSSION AND CONCLUSION

In the analysis of the lifetime spectra, the following observations have been made. In the as-grown *n*-type 6H SiC sample, we observed a lifetime component $\tau_2 = 200$ ps which was attributed to positrons annihilating from V_{Si} and $V_C V_{Si}$, which have lifetime values lower and greater than 200 ps, respectively. At annealing temperatures of 650 °C or above, the observed τ_2 changed to about 232-ps as the silicon vacancy was annealed out. This 232-ps lifetime component was attributed to a positron purely annihilating from $V_C V_{Si}$. This value is close to the $V_C V_{Si}$ lifetime value of 214 ps calculated by Brauer *et al.*,[21] and the 235-ps lifetime component found after Ge implantation, which was attributed to a $V_C V_{Si}$ divacancy.[10]

In an earlier study,[11] we also performed lifetime measurements on as-grown *p*-type 6H-SiC as a function of the measurement temperature. A temperature-independent lifetime component of 225 ± 11 ps was observed. The Fermi level of this *p*-type sample at room temperature is estimated to have been $E_V + 0.045$ eV. The $(++/+)$ level of V_{Si} was calculated to be $E_V + 0.17$ eV by Zywietz *et al.*[26] As already pointed out by Ling *et al.*[11] and further supported by the result of Zywietz *et al.*, V_{Si} in the *p*-type sample should be positively charged, which implies that the 225-ps observed component was totally free from any positrons annihilating from V_{Si}. In this respect it is noted that the as-grown *p*-type lifetime data (225 ± 11 ps) are statistically consistent with that of the *n*-type sample annealed at temperatures of 650 °C or above (232 ± 5 ps) as regards the long lifetime component. This supports our viewpoint, namely, that the 232-ps lifetime component observed in the present annealed *n*-type sample originates from the $V_C V_{Si}$ divacancy. Assuming the correctness of this interpretation, and considering that the positron trapping rate of $V_C V_{Si}$ is found to be independent of the measurement temperature, the Si+C divacancy should be a neutral defect. Moreover, it can also be stated that the $V_C V_{Si}$ divacancy is stable at an annealing temperature of 1400 °C. These observations are consistent with the report of Polity *et al.*,[29] in which the annealing of electron irradiated *n*-type 6H SiC was monitored by the average positron lifetime without performing spectral decomposition. These authors reported a positron trapping center, probably a divacancy which annealed out in the region 920 °C–1470°.

For the as-grown and 400 °C annealed sample, our study revealed that the observed I_2 decreases with decreasing measuring temperature, with a corresponding increase in τ_1, at temperatures lower than 80 K. This low-temperature behavior can be attributed to the presence of either a positron shallow trap or a negatively charged carbon vacancy. This feature was found to anneal out in the temperature range 400–650 °C. Kawasuso *et al.*[8] and Polity *et al.*[29] studied electron-irradiated *n*-type 6H SiC, though their electron dosage and implanting energy were different. In Kawasuso *et al.*'s work,[8] the lifetime spectra were decomposed into two lifetime components. The experimental τ_1 values were found to deviate from the simple trapping model, and this was attributed to the presence of the carbon vacancy. This observed carbon vacancy was annealed out at 450 °C. In Polity *et al.*'s[29] work, the average lifetime was measured as a function of the annealing temperature. The average lifetime was found to slightly increase with increasing annealing temperature in the range of 480–930 °C, and this was attributed to the annealing out of a shallow trap. The present study and these previous studies share a common observation that in an *n*-type 6H SiC sample, there is a positron trapping site with characteristic lifetime close to the bulk that competes with trapping into the longer lifetime component (i.e., the silicon vacancy and $V_C V_{Si}$). It is unlikely that positron lifetime data can on their own distinguish this positron trapping site, although its low temperature trapping behavior shows it to be a negatively charged site. We are inclined, however, to believe the center to be a negatively charged carbon vacancy. This is because it is possible for a vacancy to be annealed out, whereas it is difficult to imagine a mechanism for charge neutralization of an ionized acceptor impurity.

Photoluminescence studies of ion-implanted 6H SiC show a D_1 spectrum which is thermal stable up to 1700°,[12] while DLTS in 6H SiC reveals a series of deep levels labeled as Z_1/Z_2, E_1/E_2 and E3/E4.[1–4] In Zhang *et al.*'s work,[14] Z_1/Z_2 was found to be thermally persistent up to 1700 °C, causing them to relate the Z_1/Z_2 level to the D_1 spectrum and to the $V_C V_{Si}$ divacancy. Gong *et al.*[3] and Aboelfotoh and Doyle[4] studied electron-irradiated 6H SiC, and obtained a different result. Similar to the findings of Zhang *et al.*, Gong *et al.* and Aboelfotoh and Doyle also observed the deep levels Z_1/Z_2, but their Z_1/Z_2 annealed out at a temperatures of 850 °C and less than 1150 °C, respectively. However, in all these DLTS studies E_1/E_2 deep levels are found to have a high thermal stability. In Gong *et al.*'s study, E_1/E_2 started to anneal out at a temperature of about 1500°, and the level was attributed to a $V_C V_{Si}$ divacancy, while in Aboelfotoh and Doyle's work E_1/E_2 persisted after a 1000 °C anneal, although in this case the levels were attributed to a negative carbon vacancy related defect.

In Gong *et al.*'s work,[3] it was reported that the observed level E_1 (referred to as ED_3 in Ref. 3) in electron-irradiated *n*-type 6H SiC has two annealing stages. The concentration of ED_3 first decreased at 700 °C to a certain plateau value which was sustained up to 1600 °C. It was suggested that the ED_3 deep level consisted of two defects, whereas the first one (ED_{3L}) annealed out at 700 °C and the second one (ED_{3H}) annealed out above 1600 °C. As this (ED_{3H}) had the same annealing behavior as E_2 (referred to as ED_4 in Ref. 3), and the ratio of ED_{3H} to ED_4 is the same for different electron-implanting dosages, it was concluded that (ED_{3H}) and ED_4 were due to the same defect taking up different site configurations, and these were attributed to the $V_C V_{Si}$ divacancy. Frank *et al.*[30] studied He-implanted 6H SiC with DLTS and photoluminescence (PL) in order to corelate the DLTS deep levels to the PL lines. It was found that at an annealing temperature of 1400 °C, the E_1/E_2 observed in the DLTS measurement and the D_1 lines found in the PL measurement were found to persist. On the other hand, the Z_1/Z_2 DLTS signal and a 4349 Å PL line were found to simultaneously extinguish at this annealing temperature. It was suggested that the E_1/E_2 DLTS signal and the D_1 PL lines originated from the same defect. As our present lifetime data show that the concentration of the V_CV_{Si} divacancy remains unchanged at an annealing temperature of 1400 °C, this implies that Z_1/Z_2 and thus the 4349 Å are not related to the V_CV_{Si} divacancy. It is still plausible to maintain the viewpoint that the thermally stable E_1/E_2 observed in the DLTS and the corresponding D_I in PL are associated with the V_CV_{Si} divacancy.

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