# Vacancy production by 3 MeV electron irradiation in 6*H*-SiC studied by positron lifetime spectroscopy

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The vacancy production in 6H-SiC by 3 MeV electron irradiation at room temperature was studied using positron lifetime spectroscopy combined with annealing experiments. It was found that the trapping rates of positrons in vacancies increased linearly with the fluence in the initial stage of irradiation. After the linear increase, the trapping rates were found to be proportional to the square root of the fluence. The linear and nonlinear fluence dependences of the trapping rates are explained by the reduction of vacancies due to recombination with interstitials during irradiation. The positron trapping rate for the admixture of silicon vacancies and divacancies showed a tendency to saturate in the higher fluence range. The trapping rate for carbon vacancies decreased after reaching a maximum. These results are explained in terms of the shift of the Fermi level due to the irradiation process. It was found that, for the lightly irradiated specimen, an annealing stage caused by recombination between close vacancies and interstitials was observed. However, such an annealing stage was not observed when using a heavily irradiated specimen. These different results are explained as the reduction of interstitials during irradiation. © 1997 American Institute of Physics. [S0021-8979(97)03619-0]

## I. INTRODUCTION

Silicon carbide (SiC) holds great potential as a semiconducting material for power devices that are functional under extreme conditions, such as high temperature and radiation environments.<sup>1</sup> Recently, the development of devices that employ the ion implantation technique has been preferred since this technique allows for full control of the electrical properties. In light of this, it is necessary to reveal the nature of radiation-induced defects.<sup>2,3</sup> Despite extensive studies, described below, the understanding of radiation-induced defects in SiC is apparently far from complete.

In previous works, photoluminescent (PL) lines, designated  $D_1$ , were found in 3C-, 4H-, and 6H-SiC crystals after annealing at 1000-1300 °C following electron or ion irradiation.<sup>4–8</sup> The  $D_1$  luminescence was reported to persist even after annealing at 1700 °C. Deep level transient spectroscopy (DLTS) studies for nitrogen-doped 6H-SiC revealed that a series of deep levels, termed E1/E2, E3/E4, and Z1/Z2, were introduced by electron or ion irradiation at room temperature.<sup>9-11</sup> The E1/E2 and E3/E4 levels were annealed to 1100 °C, while the Z1/Z2 levels remained even after annealing at 1700 °C.<sup>9,10</sup> Considering that the  $D_1$  luminescence appeared irrespective of the irradiated ion species, it is hypothesized that the  $D_1$  originated from simple point defects. Choyke and Patrick proposed that  $D_1$  luminescence was related to divacancies  $(V_{Si}V_C)$ .<sup>4</sup> To identify defect species and their annealing stages, electron spin resonance (ESR) studies were performed.<sup>12–17</sup> From the detailed analyses of the spectra, Itoh et al. succeeded in identifying silicon and carbon vacancies ( $V_{Si}$  and  $V_C$ ) in electron-irradiated 3C-SiC.<sup>13,14</sup> It was revealed that these two types of defects were annealed via two major stages (at 150 and 750 °C) and via a single stage at approximately 200 °C, respectively. Their results also suggested the presence of the Frenkel pairs  $(V_{\rm Si} \cdot I_{\rm Si}, V_{\rm C} \cdot I_{\rm C})$ .<sup>15</sup> Carbon and silicon vacancies in electron-irradiated 6*H*-SiC were found to be annealed at approximately 200 and 750 °C, respectively, by Balona and Loubser.<sup>12</sup> The ESR spectra related to divacancies and vacancy-impurity complexes involving  $V_{\rm Si}$  and nitrogen atoms at the carbon sublattice were found in quenched 6*H*-SiC by Vainer and II'in.<sup>16,17</sup> They reported that the  $V_{\rm Si}N_C$  complexes and divacancies were annealed at 1400–1500 and above 1700 °C, respectively.

The previous results indicate that monovacancies and Frenkel pairs in SiC are stable at room temperature and divacancies have an even higher stability. These are quite different from the situation in conventional semiconductors such as Si. For instance, monovacancies and single interstitials in Si are mobile far below room temperature.<sup>18,19</sup> Divacancies in Si are known to be annealed up to 350 °C.<sup>20</sup> There is great interest in understanding the kinetics of intrinsic defects in SiC. Several positron annihilation studies have been performed to date.<sup>21-26</sup> Recently, we investigated the annealing processes of vacancies in electron-irradiated nitrogendoped 6H-SiC using positron lifetime measurement.<sup>27</sup> After irradiation, defects related to silicon and carbon vacancies and divacancies were observed. These vacancies were found to be partly annealed at several stages below 500 °C and were ultimately diminished by high temperature annealing up to 1450 °C. The high temperature stages can be explained by the motion of vacancies and the dissociation of complexes between vacancies and impurities. Meanwhile, the low temperature stages can be explained by the recombination between vacancies and interstitials due to the motion of interstitials. This is supported by the fact that vacancies are immobile there.

Considering the fact that interstitials start to migrate at low temperatures, e.g., 100–200 °C, the vacancy-interstitial

reaction is expected to occur even at room temperature in a radiation field. In our research, we focused on the production of vacancies due to electron irradiation. The fluence dependences of positron lifetime and their annealing characteristics were investigated. The results strongly indicate that vacancyinterstitial recombinations are enhanced during irradiation and this plays a critical role in the production of vacancies.

### **II. EXPERIMENT**

The specimens used in this work were cut from a modified-Lely-grown 6*H*-SiC wafer doped with nitrogen. The net concentration of nitrogen atom was  $5.5 \times 10^{17}$  cm<sup>-3</sup>. The wafer was one commercially supplied by Cree Research Inc. The specimens were irradiated with 3 MeV electrons to a fluence of  $1.5 \times 10^{18} e^{-1}$  cm<sup>2</sup> at 60 °C using a dynamitron accelerator. The Fermi level of the specimens was determined from the Hall measurement using the van der Pauw method at room temperature. Isochronal annealing was performed in the temperature range between 75 and 1500 °C for 5 min in a dry argon ambience.

The positron source was prepared by depositing <sup>22</sup>NaCl ( $6 \times 10^5$  Bq) on a titanium thin film with a thickness of 3  $\mu$ m. It was sandwiched by two specimens and the positron lifetime measurement was carried out using a conventional fast-fast spectrometer with a time resolution of about 230 ps [full width at half-maximum (FWHM)] at room temperature. About  $5 \times 10^6$  counts were accumulated in each spectrum. The source components were determined to be  $130\pm10$  and  $520\pm30$  ps with intensities of  $12\pm2\%$  and 0.5  $\pm 0.3\%$ , respectively, from a measurement of an unirradiated floating-zone grown Si crystal. These components appear to come from the positron annihilations in the titanium film and in the sodium, respectively. To check the validity of these source components we analyzed various semiconductors and metals using the source components. The bulk lifetimes obtained agreed with those reported in previous works, suggesting that the source components were determined properly.<sup>28</sup> To discriminate between bulk and defect components, after subtracting the source and background components, a lifetime spectrum L(t) was decomposed into two terms using the computer program PATFIT-88:<sup>29</sup>

$$L(t) = (I_1/\tau_1) \exp(-t/\tau_1) + (I_2/\tau_2) \exp(-t/\tau_2).$$
(1)

Here,  $\tau_i$  are the lifetimes and  $I_i$  are the intensities  $(I_1+I_2 = 1)$ . The average positron lifetime  $\langle \tau \rangle$  is defined as

$$\langle \tau \rangle = I_1 \tau_1 + I_2 \tau_2. \tag{2}$$

This quantity is sensitive to the change in positron lifetime. With the framework of the two-state trapping model<sup>28</sup> in which positrons are assumed to annihilate through the bulk state or through the trapped state in vacancy-type defects, the lifetimes  $\tau_1$  and  $\tau_2$  have the following physical meanings:

$$\tau_1 = \frac{1}{\tau_R^{-1} + \kappa},\tag{3}$$

$$\tau_2 = \tau_V. \tag{4}$$

Here,  $\tau_B$  is the positron lifetime in the bulk (bulk lifetime) which is determined to be 138±2 ps from a measurement of



FIG. 1. Positron lifetime spectra for the unirradiated specimen and for irradiated specimens with fluences of  $1.0 \times 10^{17}$  and  $1.5 \times 10^{18} e^{-}/cm^{2}$ .

an unirradiated *p*-type SiC (doped with aluminum),  $\tau_V$ , the positron lifetime in vacancy-type defects, and  $\kappa$ , the net positron trapping rate due to the defects are

$$\kappa = \frac{I_2}{I_1} (\tau_B^{-1} - \tau_2^{-1}). \tag{5}$$

The trapping rate is proportional to the concentration of defects (*C*) and the specific trapping rate ( $\mu$ ):  $\kappa = \mu C$ . Equation (3) represents the effective bulk lifetime when positrons are trapped by defects. In the following discussion, when referring to defect concentration, we will use the trapping rate rather than intensity  $I_2$  since  $I_2$  is not proportional to the defect concentration. The validity of the two-state trapping model can be checked by Eq. (3) since each side of the equation is determined independently.

#### **III. RESULTS AND DISCUSSION**

Figure 1 shows the positron lifetime spectra obtained for the unirradiated specimen and for irradiated specimens with a fluence of  $1.0 \times 10^{17}$  and  $1.5 \times 10^{18} e^{-1}$  cm<sup>2</sup>. The average lifetimes for these specimens were determined to be 142, 179, and 202 ps, respectively (error within 2 ps). The positron lifetime of the unirradiated specimen is slightly longer than that of the unirradiated p-type specimen (138 ps). As reported in the previous research,<sup>27</sup> the prolonged positron lifetime for the unirradiated n-type specimen may be due to the presence of vacancy-type defects. The positron lifetime increases as the fluence increases. This shows that vacancytype defects are created by the irradiation and that their concentrations also increase with the fluence. To obtain more detailed information, the lifetime spectra were decomposed into two terms (bulk and defects) using Eq. (1). Figure 2 shows the dependence of the lifetimes ( $\tau_1$  and  $\tau_2$ ) and the intensity  $I_2$  on the fluence. The experimental error is within the circle. The lifetime  $\tau_2$  increases from 183 ps with an intensity of 45% at the unirradiated state to  $\sim$ 215 ps with an intensity of 90%.



FIG. 2. Positron lifetimes ( $\tau_1$  and  $\tau_2$ ) and intensity  $I_2$  as a function of the fluence. The lifetime  $\tau_1^{\text{TM}}$  denotes the lifetime  $\tau_1$  expected from the trapping mode [Eq. (3)].

A recent theoretical calculation by Brauer et al. showed that the positron lifetimes for a carbon vacancy, a silicon vacancy, and a divacancy in 6H-SiC are 150-153, 183-194, and 214 ps, respectively.<sup>30,31</sup> The lifetime for a  $V_{\rm Si}N_{\rm C}$  complex was reported to be similar to that of an isolated silicon vacancy. The lifetime  $\tau_2 = 183$  ps obtained for the unirradiated specimen in the present work was previously attributed to complexes between silicon vacancies and nitrogen atoms through the annealing experiment.<sup>27</sup> The average energy of primary knock-on atoms (PKAs) is estimated from an elementary displacement calculation to be approximately 100 eV for 3 MeV electron irradiation.<sup>32–34</sup> Thus, simple defects such as monovacancies, single interstitials, and Frenkel pairs are mainly produced by irradiation. In addition, divacancies are also created directly through the successive displacement of two adjacent atoms. After the creation of divacancies, most PKA energy is expected to be lost. Hence, the displaced atoms will stop in the vicinity of the divacancies created. On the basis of this assertion and the result that the value of the lifetime  $\tau_2$  varied between those for silicon vacancies and divacancies, the lifetime  $\tau_2$  after the irradiation is considered to be a weighted average between defects related to silicon vacancies and those related to divacancies. As the fluence increases, the lifetime  $\tau_2$  approaches the theoretical lifetime for a divacancy. This indicates that divacancies



FIG. 3. Trapping rates  $\kappa_s$  (for carbon vacancies) and  $\kappa_2$  (for silicon vacancies+divacancies) as a function of the fluence.

work as strong positron trapping centers compared to the other types of vacancies.

Figure 2 also shows that the lifetime  $\tau_1$  considerably deviates from the value expected in the two-state trapping model ( $\tau_1^{\text{TM}}$ ). The deviation well exceeds experimental uncertainties. As explained in previous work,<sup>27</sup> this is because the  $\tau_1$  component is a weighted average between the lifetime  $\tau_1^{\text{TM}}$  and the additional lifetime  $\tau_s$  caused by carbon vacancies. Positron trapping rates for the  $\tau_s$  (carbon vacancies) and  $\tau_2$  (silicon vacancies+divacancies) components are calculated by

$$\kappa_{S} = \frac{\tau_{1}(\tau_{B}^{-1} - I_{2}\tau_{2}^{-1}) - I_{1}}{\tau_{S} - \tau_{1}},\tag{6}$$

$$\kappa_2 = \frac{I_2}{I_1} \cdot \frac{\tau_B^{-1} - \tau_2^{-1} - I_1 \tau_S^{-1} (1 - \tau_1 \tau_2^{-1})}{1 - \tau_1 \tau_S^{-1}},\tag{7}$$

respectively, instead of by Eq. (5).<sup>28</sup> Here,  $\tau_s$  is assumed to be 150 ps for carbon vacancies.<sup>30,31</sup> Figure 3 shows the fluence dependence of the trapping rates. The trapping rate  $\kappa_2$ increases rapidly in the initial stage of irradiation. Although the data scattering is relatively large in the higher fluence range, the trapping rate  $\kappa_2$  seems to have a tendency to saturate within the experimental uncertainties. The trapping rate  $\kappa_s$  first increases with the fluence but decreases after reaching a maximum. It should be noted that the trapping rates show nonlinear behaviors.

If the produced defects do not react to each other during irradiation, their concentrations should be simply proportional to the fluence. On the contrary, the nonlinear fluence dependence of the trapping rates was observed as shown above. This result can be explained by the loss of vacancies during irradiation. Such a loss of vacancies is known to be observed when interstitials are mobile.<sup>35</sup>

We argue the general features for the fluence-dependent vacancy concentration using the chemical rate equations, considering the recombination between vacancies and interstitials and their migration to sinks. Although the formation of divacancies by the combination of mobile vacancies was suggested for 3C-SiC irradiated with 1 MeV electrons, it was found to be obvious in a quite high fluence range(>5 $\times 10^{18} e^{-/cm^2}$ .<sup>36</sup> In the case of 3 MeV electron irradiation up to  $1.5 \times 10^{18} e^{-1}$  cm<sup>2</sup>, it is likely that the primary production of divacancies is predominant. Thus, the production of divacancies via the combination of vacancies is not considered here. The generation of monovacancies through recombination between divacancies and interstitials is also not considered since the concentration of divacancies is thought to be considerably lower than those of monovacancies. Even so, it becomes problematic to find the solution to the chemical rate equations when we include all types of defects. We therefore do not distinguish between different types of vacancies or interstitials for simplicity. In this case, the chemical rate equations for vacancies and interstitials during the irradiation are

$$\frac{dC_V}{dt} = K - K_{V \cdot I} C_I C_V - K_{SV} C_{SV} C_V, \qquad (8)$$

$$\frac{dC_I}{dt} = K - K_{V \cdot I} C_I C_V - K_{SI} C_{SI} C_I, \qquad (9)$$

where  $C_V$ ,  $C_I$ ,  $C_{SV}$ , and  $C_{SI}$  denote the concentration of vacancies, interstitials, and their internal sinks, respectively, K is the production rate of Frenkel pairs due to the irradiation, and  $K_{V\cdot I}$ ,  $K_{SV}$ , and  $K_{SI}$  are the reaction constants for the vacancy-interstitial recombination, annihilations of vacancies, and interstitials at their sinks, respectively. The fluence is given by  $\phi = it$ , where i and t are the flux of the incident electrons and the irradiation time, respectively. The general trends to the solution to the above rate equations were shown by Sizmann assuming  $C_{SI} = C_{SV} \equiv C_S$  (constant) as described below.<sup>37</sup>

The characteristic time constants for the beginning of vacancy-interstitial recombination after the accumulation of vacancies and interstitials, annihilations of interstitials, and vacancies at sinks are defined as  $t_1 = (KK_{V.I})^{-1/2}$ ,  $t_2 = (K_{SI}C_S)^{-1}$ , and  $t_3 = (K_{SV}C_S)^{-1}$ , respectively. From the analogy of studies for metal defects,35 the rate of recombination of Frenkel pairs is expected to be much faster than that of the annihilation of interstitials at sinks. Interstitials are also expected to be much more mobile than vacancies. Thus, it is natural to assume  $t_1 < t_2 < t_3$ . In the initial stage of irradiation, the second and the third terms of the right-hand side of Eqs. (8) and (9) are negligible since  $C_V$  and  $C_I$  are low. This yields  $C_V = C_I = Kt$ , i.e., the concentration of vacancies and interstitials is proportional to the fluence  $(C_V = C_I \propto \phi)$ . As vacancies and interstitials pile up sufficiently so that t $\geq t_1$ , there is an increased chance for vacancy-interstitial recombination. Then, the production and annihilation of vacan-



FIG. 4. Logarithmic plot of the fluence dependence of the trapping rates  $\kappa_2$  and  $\kappa_s$ . The symbols  $\phi$  and  $\phi^{1/2}$  represent the slopes of the lines.

cies and interstitials are temporarily balanced and the concentrations reach a plateau,  $C_V = C_I = (K/K_{V\cdot I})^{1/2}$ . No plateau appears when the vacancy-interstitial recombination and the annihilation of interstitials at sinks occur almost simultaneously, i.e.,  $t_1 \sim t_2$ . At  $t \ge t_2$ , interstitials start to annihilate at sinks and the concentration decreases as  $C_I$  $= (K/K_{V\cdot I}K_{SI}C_St)^{1/2}$ . Then, the concentration of vacancies increases as  $C_V = (KK_{SI}C_St/K_{V\cdot I})^{1/2}$ . Namely, the concentration of vacancies is proportional to the square root of the fluence  $(C_V \propto \phi^{1/2})$ . At  $t \ge t_3$ , vacancies also start to annihilate at sinks, hence, the concentration of vacancies and interstitials have a tendency to saturate. Then the concentration of vacancies and that of interstitials can be represented by the following equations:  $C_V = (KK_{SI}/K_{V\cdot I}K_{SV})^{1/2}$  and  $C_I$  $= (KK_{SV}/K_{V\cdot I}K_{SI})^{1/2}$ . This corresponds to the stationary state.

Figure 4 shows the logarithmic plot of the fluence dependence of trapping rates  $\kappa_2$  and  $\kappa_s$ . (Note that the trapping rate is proportional to the concentration of vacancies). Both trapping rates seem to be proportional to the fluence  $(\kappa_2 \propto \phi)$  in the fluence range below  $1 \times 10^{17} e^{-1}$  cm<sup>2</sup> and to the square root of the fluence  $(\kappa_2 \propto \phi^{1/2})$  in the fluence range from  $1 \times 10^{17}$  to  $4 \times 10^{17} e^{-1}$  cm<sup>2</sup> within experimental errors. These features closely match that expected from the chemical rate equations shown above. The absence of a plateau after the linear growth of the trapping rates indicates  $t_1 \sim t_2$ . Thus, it is probably that vacancies are reduced during irradiation through mutual recombination with interstitials. It is likely that interstitials migrate a long distance during irradiation. Although the close Frenkel pairs and interstitials in SiC are thought to be stable at room temperature,<sup>15</sup> the mi-



FIG. 5. The Fermi level as a function of the fluence. Here,  $E_C$  represents the energy of the bottom of the conduction band.

gration of interstitials may be enhanced by the ionization effect during irradiation. It is inferred that interstitials can migrate in a radiation field by gaining excess energy released from electron-hole recombinations and/or through successive transformation of the stable configuration due to the change in the charge state.<sup>38,39</sup>

With reference to the chemical rate equations mentioned above, the saturation level of the trapping rate  $\kappa_2$  in the fluence range above  $5 \times 10^{17} e^{-1}/cm^{2}may$  be explained as the annihilation of silicon vacancies and divacancies at sinks. However, it is questionable, since silicon vacancies and divacancies are known to be immobile below  $\sim 750 \ ^\circ C.^{12-14}$ The migration of carbon vacancies may be assisted by irradiation since they are mobile at approximately 200 °C.<sup>13,14</sup> Even so, the decrease in the trapping rate  $\kappa_s$  in the fluence range above  $5 \times 10^{17} e^{-}/cm^{2}$  is not the expected result based on the above rate equations. Here we should consider the change in the electrical properties of the specimen. Figure 5 shows the fluence dependence of the Fermi level at room temperature. (Above  $8 \times 10^{17} e^{-1}$ /cm<sup>2</sup>, the Hall measurement at room temperature was difficult to record due to the extreme reduction of the free carrier density). The Fermi level approaches the middle of the band gap with increasing fluence. This result shows that residual defects work as acceptors to compensate free electrons and that their concentrations increase as the fluence increases.<sup>40</sup> A series of acceptor levels is known to be introduced by electron irradiation in the upper half of the band gap from DLTS measurements.<sup>9-11</sup> Although the energy levels associated with carbon and silicon vacancies and divacancies have not yet been established, these defects are expected to have acceptor levels. It is probable that the charge states of vacancies change as the fluence increases due to the shift of the Fermi level. Considering the fact that the specific trapping rate for a vacancy is known to depend on its charge state due to the Coulomb attraction,<sup>41</sup> the saturation of the trapping rate  $\kappa_2$  and the decrease of the trapping rate  $\kappa_s$  in the higher fluence range may be caused by the change in the charge state of vacancies as the fluence increases.

The above arguments imply that the concentrations of residual interstitials in the low fluence range may be higher than those of the high fluence range. This may affect the annealing kinetics of vacancies. For confirmation, we performed annealing experiments for the lightly (1.0



FIG. 6. Positron lifetimes ( $\tau_1$  and  $\tau_2$ ) and intensity  $I_2$  obtained for the specimens irradiated with  $1 \times 10^{17}$  (open circle) and  $1.5 \times 10^{18} e^{-/cm^2}$  (closed circle) as a function of annealing temperature. The lifetime  $\tau_1^{TM}$  denotes the lifetime  $\tau_1$  expected from the trapping model.

×10<sup>17</sup>  $e^{-/cm^2}$ ) and heavily (1.5×10<sup>18</sup>  $e^{-/cm^2}$ ) irradiated specimens. Figure 6 shows the annealing behavior of lifetimes ( $\tau_1$  and  $\tau_2$ ) and intensity  $I_2$  for these specimens. Figure 7 shows the annealing behavior of the trapping rates  $\kappa_s$ and  $\kappa_2$  calculated from Eqs. (6) and (7) which are related to carbon vacancies and the admixture of silicon vacancies and divacancies, respectively.

First, let us consider the approximate annealing features for the lightly irradiated specimen and then compare them with those for the heavily irradiated specimen. The trapping rate  $\kappa_s$  of carbon vacancies first decreases at approximately 75 °C and finally diminishes at 250-450 °C. Since close Frenkel pairs are considered to be less stable than isolated vacancies, the annealing stage at  $\sim$ 75 °C is probably caused by the mutual recombination of Frenkel pairs ( $V_{\rm C}$  $\cdot I_{\rm C} \rightarrow$  null). The annealing temperature for the higher stage coincides with that observed by ESR studies.<sup>12,14</sup> The annealing stage at 250-400 °C can probably be attributed mainly to the motion of carbon vacancies ( $V_{\rm C} \rightarrow \text{sinks}$ ). The lifetime  $\tau_2$ related to the admixture of silicon vacancies and divacancies decreases from 210 to 189 ps from 75 to 500 °C. The trapping rate  $\kappa_2$  also decreases from 75 to 500 °C. The lifetime  $\tau_2 = 189$  ps observed after the annealing at 500 °C is almost



FIG. 7. Annealing behaviors of the trapping rates  $\kappa_s$  and  $\kappa_2$  obtained for the specimens irradiated with  $1 \times 10^{17}$  (open circle) and  $1.5 \times 10^{18} e^{-/cm^2}$  (closed circle).

the same as the theoretical lifetime of a positron in a silicon vacancy. These results show that silicon vacancies and divacancies already start to disappear at 75 °C. Most likely, the low temperature annealing of vacancies occurs because of recombination with interstitials rather than by their migration and dissociation. In fact, the annealing experiments by ESR showed that silicon vacancies partly disappeared at approximately 200 °C due to recombination with interstitials.<sup>13,14,42</sup> When the temperature is greater than 500 °C, the trapping rate  $\kappa_2$  decreases at 700–800, 1050, and finally reaches the detection limit at 1450 °C. These processes were attributable to the migration of silicon vacancies, and residual impurities such as  $V_{Si}N_C$  complexes.<sup>27</sup>

For the heavily irradiated specimen, the trapping rate  $\kappa_S$  for carbon vacancies first increases at 250–350 °C and diminishes at approximately 450 °C. The increase in the trapping rate may be explained as the generation of carbon vacancies, due to either the recombination between divacancies and silicon interstitials, or the change of the charge states. The final annealing stage at 450 °C may be attributed to the annihilation of carbon vacancies to their sinks. Here, it should be noted that the annealing stage at approximately 75 °C was observed for the lightly irradiated specimen but

not for the heavily irradiated specimen. As previously noted, the annealing stage at approximately 75 °C was attributable to the disappearance of close Frenkel pairs between carbon vacancies and interstitials. The above result therefore suggests that the concentration of the Frenkel pairs is fairly low in the heavily irradiated specimen. Close Frenkel pairs are thought to have already been annihilated during irradiation due to the motion of interstitials.

The lifetime  $\tau_2$  and the trapping rate  $\kappa_2$  decrease with the annealing temperature. The annealing process above 500 °C is similarly explained as in the case of the lightly irradiated specimen. The decreases in the lifetime  $\tau_2$  and that of the trapping rate  $\kappa_2$  up to 500 °C again show the disappearance of silicon vacancies and divacancies in this temperature range. However, the low temperature annealing stage at 75-200 °C observed for the lightly irradiated specimen is not observed for the heavily irradiated specimen. Instead, its annealing temperature shifts to 200-300 °C. The annealing stage at 75-200 °C was attributed to the disappearance of silicon vacancies and divacancies due to recombination with interstitials. Thus, the above result again allows us to assume that the concentrations of close vacancyinterstitial pairs are significantly low in the heavily irradiated specimen due to the motion of interstitials.

Thus, it becomes clear that the annealing behaviors of vacancies at low temperatures (<500 °C) are different depending on the irradiation fluence: The annealing stages of vacancies related to the recombination with close interstitials below 200 °C is observed for the lightly irradiated specimen but not for the heavily irradiated one. It is concluded that vacancies recombine with interstitials during irradiation due to the motion of interstitials. As a result, the concentration of residual interstitials close to vacancies is reduced as the fluence increases. This conclusion is consistent with that deduced from the fluence dependent positron trapping rates shown above.

#### **IV. SUMMARY**

In this research, we investigated the vacancy production in 6H-SiC by 3 MeV electron irradiation through the fluence dependence of positron lifetime combined with annealing experiments. The results of this research are summarized as follows. After irradiation, defects related to carbon and silicon vacancies and divacancies were found. The positron trapping rates for these vacancies showed nonlinear fluence dependences. The nonlinear fluence dependences are partly explained using the chemical rate equations taking into account the reduction of vacancies due to recombination with interstitials and long-range migration of interstitials during irradiation. The annealing experiments showed that the low temperature annealing of vacancies (<200 °C) due to recombination with interstitials was not observed for the heavily irradiated specimen. This result is consistently explained as the reduction of such defects through recombination. It is concluded that the recombination of close vacancyinterstitial pairs and the long-range migration of interstitials are enhanced during irradiation and, hence, such defects are reduced during prolonged irradiation.

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