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Defect Characterization of 6H-SiC Studied by Slow Positron Beam

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The defect formation and annealing behavior in as-grown and electron-irradiated 6H-SiC wafers were investigated by variable-energy slow positron beam. For the n-type as-grown samples, it was found that annealing decreased the defect concentration due to recombination with interstitial, and when it was annealed at 1400 °C for 30 min in vacuum, a 20 nm thick Si layer was found on the top of SiC substrate, which is a direct proof of the Si atom diffusing to the surface when annealed at the high temperature stages. During the high temperature annealing stage, we found an obvious surface effect occurred that induced the higher S parameter close to the surface. This may be caused by the diffusion of the Si atoms to the surface during annealing. After 10 MeV electron irradiation of the n-type 6H-SiC, the positron effective diffusion length decreased from 86.2 nm to 39.1 nm. This shows that there are some defects created in n-type 6H-SiC. But in the p-type 6H-SiC irradiated by 10 MeV electrons, the change is very small. This may be because of the opposite charge of the vacancy defects. The same annealing behavior as that of as-grown 6H-SiC samples was also observed for the 1.8 MeV electron-irradiated 6H-SiC samples except that after being annealed at 300 °C, its defect concentration increased. This may be explained as the generation of carbon vacancies, due to either the recombination between divacancies and silicon interstitial, or the charge of the charge states.

Key words: Positron annihilation, Defect, Semiconductor

I. INTRODUCTION

Silicon carbide is a wide-band-gap semiconductor. As a semi-conduction material, silicon carbide holds great potential for power devices that are functional under extreme conditions, such as high temperature, high-frequency, and radiation environments [1-4]. The behavior of defects in SiC greatly impacts the chemical, mechanical, thermal, and electronic properties of the semiconductor material. In order to improve the device performance and wafer die yields, it is necessary to reveal the nature of the induced defects. Various methods have been used to study the defects induced by irradiation, such as electron spin resonance (ESR), photoluminescence (PL), deep-level transient spectroscopy (DLTS), and positron annihilation spectroscopy [2]. Many works have suggested that silicon and carbon monovacancies and also the Frenkel pairs 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 [2].

With the early photoluminescent lines (PL) studies [6-11], a series of luminescence lines designated D1 were observed in 3C, 4H, and 6H-SiC crystals due to the annealing at the temperature 1000-3000 °C following electron or ion irradiation. The D_1 luminescence was reported to persist even after the annealing at 1700 °C. Patrick and Choyke proposed that the D_1 luminescence was related to pure defect complex [7], such as divacancies, since its intensity increased irrespective of irradiated ion species. Deep level-transient spectroscopy (DLTS) studies for nitrogen-doped 6H-SiC revealed a series of deep levels, termed E1/E2, E3/E4, and Z1/Z2, were introduced by electron or ion irradiation at room temperature [12-14]. Ballandovich and Violina showed that E1/E2 and E3/E4 were annealed to 1100 °C [14], while the Z1/Z2 levels remained even after annealing at 1700 °C [12,13]. To identify defect species introduced by irradiation, several ESR studies have been performed [15-20]. 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 [13,14]. 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. Balona and Loubser also found that carbon and silicon vacancies in electron-irradiated 6H-SiC were annealed at approximately 200 and 750 °C [15]. The ESR spectra related to divacancies and vacancy-impurity complexes involving V_{Si} and nitrogen atoms at the carbon sublattice were found in quenched 6H-SiC by Vainer and Ii'in [19,20]. They reported $V_{Si}N_C$ complexes and divacancies were annealed at 1400-1500 °C and above 1700 °C, respectively.

Positron annihilation spectroscopy is now a well-established tool for the study of electronic and defect properties of solid [5]. Several positron investigations of defects in electron-irradiated SiC have also been carried out in recent years. Different annealing stages were

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TABLE I Treatment conditions of the first group samples and the fitted results of S and L_{eff}. N_2:H_2 is 80%:20%.

Sample	Type	Irradiation	Annealing	S parameter	L_{eff}/nm
AN0	n	No	No	0.4694±0.0003	59.7(±3.4)
AN1	n	No	1400 °C, 15 min in vacuum	0.4728±0.0003	60.7(±1.5)
AN2	n	No	1400 °C, 30 min in vacuum	0.4696±0.0003	44.6(±3.3)
AN3	n	No	1000 °C, 30 min in forming gas	0.4685±0.0003	65.0(±2.9)
AN4	n	No	1400 °C, 15 min in forming gas	0.4739±0.0003	87.2(±2.5)
EN0	n	No	As above	0.4739±0.0003	86.2(±3.6)
EN1	n	10^{17}e$^-$/cm^2	No	0.4822±0.0002	39.1(±0)
EP0	p	No	No	0.4696±0.0002	12.1(±1.6)
EP1	p	10^{17}e$^-$/cm^2	No	0.4713±0.0002	13.9(±1.3)

determined in these studies. Girka *et al.* and Rempel *et al.* investigated the annealing behavior of electron-irradiated 6H-SiC and detected three stages at 150-500, 1400-1500, and 1700-2900 °C and one negative annealing stage between 1000 and 1100 °C [22-24]. Girka *et al.* tentatively assigned the former stages to the annihilations of the Frenkel pairs and complexes containing Si vacancies and N dopant atoms. The negative annealing stage was explained in terms of clustering of vacancies. Atsuo *et al.*'s work indicated that after irradiation, defects related to silicon and carbon vacancies and divacancies were observed [2]. 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 [1]. The motion of vacancies and the dissociation of complex between vacancies and impurities can explain the high temperature stages.

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 here. An annealing stage of C vacancies and divacancies by mobile interstitials at 500 °C had been detected in electron-irradiation (3 MeV, 60 °C, 10^{17} cm^{-2}) 6H-SiC by Kawasuso *et al.* [21]. The annealing of defects related to Si vacancies at 750 and 1400 °C was also reported [2]. The former annealing stage was inferred to be due to migration of silicon vacancies to internal sinks or nitrogen atoms to forming complexes of silicon vacancies and nitrogen atoms. The latter annealing stage was explained as due to annihilations of the complexes as well as the case of as-grown specimens. No positron trapping was observed in electron-irradiated p-type 6H-SiC and thus, no vacancies were detected in this material [2,25]. This can be caused by the positive charge of vacancy defects.

In this work, we investigated defect formation and annealing behavior in as-grown SiC wafers and wafers irradiated with 1.8 MeV electron by positron annihilation spectroscopy. In order to study the electron irradiation effect, the 10 MeV electron irradiated n- and p-type 6H-SiC were both investigated.

II. EXPERIMENTS

The samples used in this work were cut from the modified Lely grown nitrogen-doped (n-type) and Al-doped (p-type) research grade 6H silicon carbide wafers purchased from CREE Research Inc. Two series of samples were used. The first group included n-type and p-type 6H-SiC in which doping concentrations were 1.1×10^{18} and 1.8×10^{18} cm^{-3}, respectively. The second is n-type 6H-SiC samples with doping concentration 8.5×10^{15} cm^{-3}. All the samples were degreased with acetone and methanol, and then rinsed with deionized water.

For the first group samples, a series of n-type as-grown SiC specimens were annealed at different temperatures and environments, and the other n-type and p-type samples were irradiated by 10-MeV electrons at room temperature. The irradiation dose was 10^{17} e$^-$/cm^2. In the second group samples n-type SiC samples were irradiated by electron dose 1.8×10^{18} e$^-$/cm^2 and annealed at 300 and 450 °C. In order to investigate if a surface effect occurred during electron irradiation, a chemical etch was also made for the 300 °C sample. The treatment conditions are listed in Table I and Table II. Variable-energy positron measurements were performed for the first group samples at the University of Hong Kong and the same treatment was made for the second group samples in the University of Science and Technology of China. The vacuum in the target chamber is 1.33 μPa. The Doppler broadening technique of the annihilation radiation is applied to the measurements. Photo-peak energy spectra of annihilation 511 keV γ-ray from target were measured using a high purity Ge detector, which had an energy resolution of 1.2 keV (FWHM) at 514 keV γ-ray of ^{85}Sr. The line-shape S parameter of a spectrum is defined as the integral of γ-ray counts in the central energy region at the 511 keV divided by the total counts of spectrum, in which 5×10^5 counts is contained

$$S=\frac{\displaystyle\int_{-a}^{a}c(E)\mathrm{d}E}{\displaystyle\int_{-\infty}^{\infty}c(E)\mathrm{d}E} \tag{1}$$

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TABLE II Treatment conditions of the second group samples which were irradiated by 1.8 MeV electron with dose of 8.5×10^{18}e$^-$/cm^2 and the fitted results of S and $L_{\rm eff}$. N_2:H_2 is 80%:20%.

Specimen	Type	Treatment	S parameter	$L_{\rm eff}$/nm
BN0	n	As-grown	0.4750±0.0007	140(±13.1)
BN1	n	Irradiated by 1.8 MeV	0.4826±0.0004	13.7(±1.4)
BN2	n	300 °C, 30 min in forming gas	0.4839±0.0002	3.6(±0.4)
BN3	n	BN2 with chemical etched	0.4976±0.0004	54.7(±3.2)
BN4	n	450 °C, 30 min in forming gas	0.4961±0.0003	74.8(±4.5)

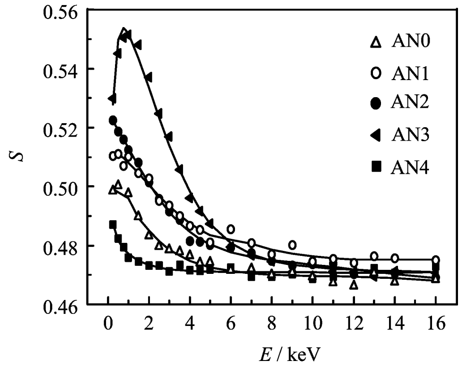

FIG. 1 Mean Doppler broadening S parameter as a function of incident positron energy E for the n-type SiC specimens AN0-AN4. The solid lines are the fitted curves.

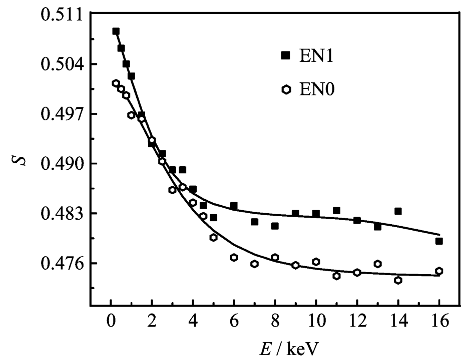

FIG. 2 Mean Doppler broadening parameter S parameter as a function of incident position energy E for n-type SiC specimens EN0-EN1. The solid lines are the fitted curves.

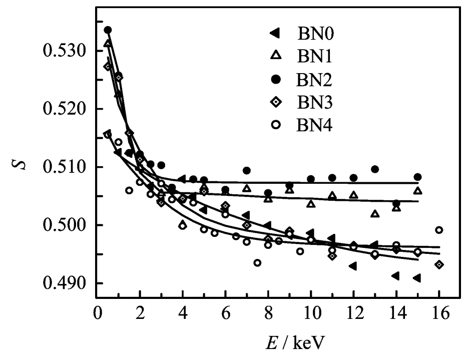

FIG. 3 Mean Doppler broadening parameter S parameter as a function of incident positron energy E for n-type electron-irradiated specimens of the second group. The solid lines are the fitted curves.

where $c(E)$ is the measured spectrum with the background subtracted, and $(-a, a)$ is an energy region in the coordinate axis with original point at the peak energy 511 keV. We take an energy shift of about 1 keV at both sides of the peak energy. The variation of the S parameter is mainly influenced by the relative counts around the peak, so it provides the information about the positron annihilating with the lower momentum electrons. At the defect region, the atoms lacking in vacancy regions will increase the S parameter for the larger fraction of lower momentum electrons.

III. RESULTS AND DISCUSSION

The measured S-E curves for all specimens are presented in Fig.1, Fig.2, Fig.3, and Fig.4 respectively. All the S-E curves were analyzed using the VEPFIT model 5 program [26]. The fitted results of S parameter and $L_{\rm eff}$ for the two groups are also listed in Table I and Table II respectively.

The $L_{\rm eff}$ indicates effective diffusion lengths of positrons in the material. It is related to the trapping of defects in the sample [3] as follows:

$$L_{\rm eff}=\left(\frac{D_+}{\lambda_b+k}\right)^{1/2} \qquad (2)$$

where D_+ is positron diffusion coefficient, λ_b is the positron annihilation rate in the bulk, and k_i is the trapping rate in a concentration of defects c_i. c_i is proportional to the defect-concentration: $k_i=\mu_i\times c_i$, μ_i is the positron trapping coefficient. The relative values of k_i for different samples are calculated as follows:

$$k_i=\left(\frac{L_0}{L_i}\right)^2 k_0+\left[\left(\frac{L_0}{L_i}\right)^2-1\right]\lambda_b \qquad (3)$$

where L_0 and L_i are the effective diffusion lengths of the basic sample and the compared sample respectively. k_0 is the positron trapping rate of the basic sample.

Ling *et al.* have observed that in n-type 6H-SiC, when the annealing temperature is above 1000 °C, the V_{Si}

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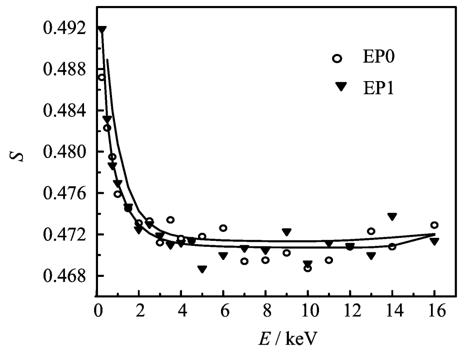

FIG. 4 Mean Doppler broadening parameter S as a function of incident position energy E for p-type SiC of specimens EP0-EP1. The solid lines are the fitted curves.

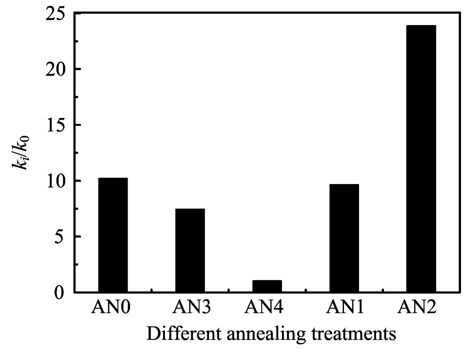

FIG. 5 The calculated k_i/k_0 of AN0-AN4 of the first group samples as a function of different annealing treatment conditions.

was almost annealed out, and the remaining kind of vacancy was mostly the divacancy $V_{Si}V_C$ [3]. Meanwhile, the trapping rate in it remains constant. In this work, if taking the sample annealed at 1400 °C for 15 min in forming gas as a basic sample, the L_0=87.2 nm. According to Brauer *et al.* [5], the $\lambda_b=1/\tau_{bi}$ is 7.092 ns^{-1} for n-type 6H-SiC. All of the relative positron trapping rates of n-type 6H-SiC samples AN0-AN4 can be calculated using Eq.(3). Their relationships can be expressed as shown in Fig.5.

From Fig.5, it is obvious that the relative positron trapping rates of the samples AN3 and AN4 that annealed in the forming gas were all smaller than that of the AN0. But for samples AN1 and AN2 that annealed in vacuum, one can see that the trapping rate of AN1 was slightly smaller than that of AN0, and the trapping rate of AN2 was much larger than that of AN0. Considering the relative positron trapping rate of AN1 and AN4, which annealed at the same temperature with the same annealing time, it is seen the relative positron trapping rate of AN1was much higher than that of AN4. All these variations will be discussed below.

For the samples annealed in the forming gas (AN3, AN4), comparing those relative positron trapping rates with that of non-annealed AN0, it can be seen that some vacancies in the samples were eliminated by the annealing, which is probably due to the migration of the pure silicon vacancies. The ESR studies have shown that pure silicon vacancies become mobile at around 750 °C. The mobile silicon vacancies seem to annihilate at sinks or to form complexes of silicon vacancies and nitrogen atoms. In this work, when the sample was annealed at 1000 °C in the forming gas, mostly V_{Si} vacancies annealed out, and more complexes of silicon vacancies and nitrogen were formed, thus the total concentration decreased. When the annealing temperature rose to 1400 °C, more complexes vacancies annealed out. Considering AN1 and AN4, which both annealed at 1400 °C for 15 min but in different environments, it is clear that the relative positron trapping rate of sample annealed in vacuum AN1 was about 9.6 times of that annealed in the forming gas AN4. This indicates that there are more vacancies created after this annealing process. This may be due to the fact that after annealing at high temperature in vacuum, some of the Si atoms diffuse to the surface and then V_{Si} can form. This process can be compared with the recovery of original V_{Si} during the annealing. More Si atoms can diffuse to the surface of sample when it is annealed in vacuum than when annealed in the forming gas. Considering the curve of AN2 in Fig.1, when annealed at 1400 °C for 30 min in vacuum, it was observed that the S parameter increased with the increasing energy till the energy reached about 1 keV. Considering this part of S-E relations in AN2, we found it has the same tendency with that of the original Si, as previous work obtained [28]. This can be explained by a surface effect, which will be discussed below. Using a three layers model (Si/interface/SiC), setting the interface region to be 48 nm, a good fitting can be given. It was found that a 20-nm thick Si layer formed on the top. This is a direct proof that the Si atoms diffused to the surface when annealed at high temperature. The high interface relative positron-trapping rate k_i/k_0 (56.33), calculated from its fitted effective diffusion length of 30.9 nm, indicates that the migration of silicon atoms created more vacancies in this layer.

It is very clear that contrary to the annealing effects that occurred in the SiC bulk, after annealing, the S parameters close to specimens' surface all increased. This indicates that the high temperature annealing induces a surface effect in SiC specimens. The S parameters at 1.5 keV for AN0-AN4 are presented in Fig.6. By comparing the data in Fig.6 one can see that the S parameter increased with the higher annealing temperature. When annealing at the same temperature, it is also increased with longer annealing time. The S parameter 0.5082 for AN1 is slightly larger than the 0.5045 for AN4, which indicates that when annealing in vacuum, this surface effect is more distinct. It is probably due to the migration of Si atoms. In the previous discussion, we have estimated that during the annealing some of Si

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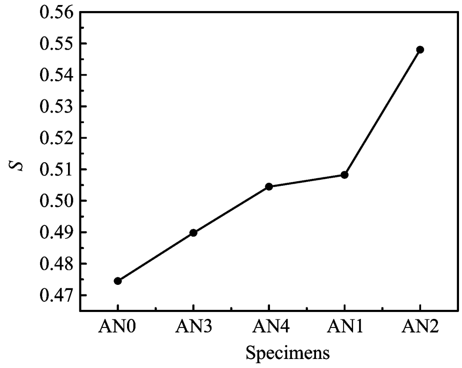

FIG. 6 Mean Doppler broadening parameter S at 1.5 keV for specimens AN0-AN4.

atoms diffuse to the surface so V_{Si} can form, and this process can be compared with the recovery of original V_{Si}. To some extent, during the annealing process, two reactions exist: the creation of the Si vacancies induced by some Si atoms diffusing to the surface, and the annealing out of the Si vacancies because of annihilation at sinks or forming complexes of silicon vacancies and nitrogen atoms. The former is stronger near the surface than that in bulk, so the V_{Si} concentration near the surface was larger than that in bulk. When the silicon atoms diffuse to the surface, some V_{Si} and complexes vacancies related to silicon atoms such as Si−N complexes formed. The concentration of the silicon atoms was reasonably proportional to the annealing temperature and its corresponding annealing time. With the longer annealing time and the higher annealing temperature, more silicon atoms will diffuse to the surface layer, thus more vacancies related to it will form. It is obvious that when annealed at 1400 °C for 30 min in vacuum, the excessive silicon atoms, which are not combined by complexes vacancies, can form a silicon film layer in the SiC surface.

A number of extensive research projects have been performed to clarify the properties of radiation-induced defects in 6H-SiC [1,2,25]. Those works indicate that vacancy-type defects have important roles in irradiated and also in as-grown SiC. Silicon vacancies, carbon vacancies, Frenkel pairs, and small amount of divacancies are directly created by irradiation [1].

In this work, for the first group samples, the S-E measured curves for the as-grown samples (no irradiation) and the 10-MeV electron irradiated n-type 6H-SiC are shown in Fig.2. The S parameter of irradiated sample increased from 0.4739 to 0.4822. It is shown that after 10 MeV electron irradiation, more vacancies have been created. Dannefaer *et al.* found that two vacancy types have been created after electron irradiation [25], but the balance between these two defects is energy dependent. Change of the S parameter after 10-MeV electron irradiation in this work was about 1.75%, which is lower than the result presented by Dannefaer *et al.* [25]. This may be caused by the lower irradiation dose for the sample. Kawasuso *et al.* found that the trapping rates of positrons in vacancies increased linearly with the dose in the initial stage of irradiation [1]. After the linear increase, the trapping rates were proportional to the square root of the dose for the n-type 6H-SiC. We obtained that L_{eff} decreased from 86.2 nm to 39.1 nm after irradiation, which means that the concentration of the vacancies increased.

For the second group of electron irradiated n-type 6H-SiC samples, their measured S-E curves and the fitted L_{eff} and S parameter are listed in Fig.3 and Table II, respectively. From Table II, we can see that: after irradiation by 1.8 MeV, the effective diffusion length L_{eff} of BN1 decreases from 140 nm to 13.7 nm. This agreed well with the result of the electron irradiated SiC samples in the first group. When annealed at 300 °C, compared with that of irradiated sample BN1, the L_{eff} of BN2 decreased from 13.7 nm to 3.6 nm. This effect may be explained as the generation of carbon vacancies, due to either the recombination between divacancies and silicon interstitials, or the charge of the charge states. This fact seems to be contrary to the annealing behaviour of the original sample studied in previous work [29]. For original SiC, it is found that after annealing at 300 °C, the defects related to carbon vacancies were annealed. In order to investigate if there is a surface effect, we chemically etched the 300 °C annealed sample. It was found that after chemical etching the 300 °C annealed sample, the effect diffusion length L_{eff} increased from 3.6 nm to 54.7 nm. This reflects that there is a surface effect during the annealing process. It indicates that the generation of carbon vacancies was mostly created near the surface layer. We also performed a 450 °C annealing for the electron irradiated sample. After annealing, its effective diffusion length L_{eff} increased to 74.8 nm. This indicates that this temperature annealing can eliminate some defects, which may be attributed to the annihilation of carbon vacancies to their sinks. These results are similar to those for the original 6H-SiC. More annealing temperature experiments have been done in other work for different electron irradiation n-type 6H-SiC, but no surface effect has been observed.

For the p-type 6H-SiC, the sample was irradiated under the same condition with the n-type one in the first group. Figure 4 shows the S-E data and the fitting curves. The fitting L_{eff} and the S parameter are listed in Table I. It is interesting that different behavior of L_{eff} occurs in p-type. Here L_{eff} increased from 12.1 nm of the as-grown sample to 13.9 nm after the same irradiation. The S parameter only slightly changed from 0.4696 to 0.4713. This may be explained by the fact that irradiation-induced vacancies V_{Si} and V_C in the p-type were positively charged, thus they are not capable of trapping positrons. Obviously it is possible since Itoh *et al.* detected a paramagnetic resonance signal they ascribed to a positive charged carbon vacancy

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V_C^+ only in p-type 3C-SiC. Also, in a previous study, Ling *et al.* pointed out that in the p-type 6H-SiC material, the Fermi level was close to the valence band, which probably made the silicon vacancy V_{Si} positively charged and the acceptor neutrally charged [27]. The second reason is that the introduction rate of vacancies may be much smaller in materials than that in n-type, since p- or n-type dependences on irradiation damage is commonly observed for semiconductors. To make the process of irradiation in p-type SiC clear, more experiments should be carried out.

IV. CONCLUSION

Annealing behavior of vacancy-type defects in as-grown and electron-irradiated 6H-SiC were studied using a slow positron beam. The defects related to silicon vacancies, possibly complexes of silicon vacancies and nitrogen atoms were found to be present in as-grown n-type samples. It was found that after annealing, the defect concentration decreased because of recommendation with interstitials, and when it was annealed at 1400 °C for 30 min in vacuum, a 20-nm thick Si layer was found on the top of SiC substrate. This proved that the Si atom diffuse to the surface when annealed at high temperature. During the high temperature annealing stage, we found an obvious surface effect which induced the higher S parameter close to the surface, which may be caused by the diffusion of the Si atoms to the surface during annealing. After 10 MeV electron irradiation was applied to the n-type 6H-SiC, the positron effective diffusion length decreased from 86.2 nm to 39.1 nm. This shows that there are some defects created in n-type 6H-SiC. But for the p-type 6H-SiC irradiated by 10 MeV electrons, the change is very small. This may be because of the opposite charge of the vacancy defects. The same annealing behavior as that of as-grown 6H-SiC samples was also observed for the 1.8 MeV electron-irradiation 6H-SiC samples except that after being annealed at 300 °C, its defect concentration increased. This may be explained as the generation of carbon vacancies, due to either the recombination between divacancies and silicon interstitials, or the charge of the charge states.

V. ACKNOWLEDGMENTS

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