Annealing behavior of vacancies and $Z_{1/2}$ levels in electron-irradiated 4H–SiC studied by positron annihilation and deep-level transient spectroscopy

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(Received 22 August 2001; accepted for publication 9 October 2001)

Annealing behavior of vacancies and the $Z_{1/2}$ levels in n-type 4H–SiC epilayers after 2 MeV electron irradiation has been studied using positron annihilation and deep-level transient spectroscopy. Isochronal annealing studies indicate that silicon vacancy-related defects are primarily responsible for positron trapping. The $Z_{1/2}$ levels are the predominant deep centers after irradiation and subsequent annealing at 1200 °C. Both the positron-trapping rate at vacancies and the $Z_{1/2}$ concentration decrease in a similar manner while annealing from 1200 to 1500 °C. It is thus proposed that the $Z_{1/2}$ levels originate from silicon vacancy-related defects. © *2001 American Institute of Physics.* [DOI: 10.1063/1.1426259]

The $Z_{1/2}$ levels in 4H–SiC are irradiation-induced deep acceptor states located approximately 0.7 eV below the conduction band.[1–3] To date, several researchers have argued that a correlation exists between these levels and the D_1 luminescence center.[1,4,5] The $Z_{1/2}$ levels in 4H–SiC also correspond to the $E_{1/2}$ levels in 6H–SiC, which exhibit negative-U characteristics.[6,7] Despite the intense amount of research in this area, it is still an open question as to whether or not vacancies are indeed constituents of the $Z_{1/2}$ defect.

Positron annihilation spectroscopy (PAS) is well suited to the characterization of vacancy-related defects in crystalline solids.[8] In this research, we studied the annealing behavior of vacancy defects in electron-irradiated 4H–SiC by PAS and deep-level transient spectroscopy (DLTS). By comparing PAS and DLTS data, we can clearly examine whether or not there is a relationship between vacancy defects and the $Z_{1/2}$ level.

Samples were cut from a high-quality nitrogen-doped n-type 4H–SiC epilayer (5 μm thick) on a 8.0° off-oriented 4H–SiC (0001) substrate from Cree Research Inc. The net donor concentration is approximately 5×10^{15} cm^{-3}. The samples were then irradiated at 50 °C with 2 MeV electrons to doses of 1×10^{15} and 3×10^{17} e$^-$/cm^2 for DLTS and PAS measurements, respectively. Isochronal annealing was conducted from 100 to 1700 °C for 30 min. After fabricating the Schottky and Ohmic contacts using Ni, DLTS measurements were performed in the temperature range from 100 to 700 K. Using an energy variable positron beam ($E_+ = 0.1–39$ keV), the Doppler broadening of annihilation radiation (511 keV) in the epilayers was measured with a single Ge detector. Peak and tail intensities of the Doppler spectrum, which are called S and W parameters, were determined in the energy windows of 511.0–511.8 and 515.0–522.0 keV, respectively. The positron lifetime measurements were also performed with a pulsed positron beam ($E_+ = 17$ keV).[9] The S and W parameters of the as-grown sample were 0.4327 and 0.0034, respectively. The positron lifetime was 143 ps, which agrees well with the bulk lifetime of SiC.[10] All the S and W parameters were normalized to those of the as-grown sample. To analyze the electron momentum distribution, coincidence Doppler broadening measurements were also carried out.[8]

After irradiation to a dose of 3×10^{17} e$^-$/cm^2, the positron lifetime and S parameter increased to 194 ps and 1.04, respectively. These results confirm the presence of vacancy defects. The depth distribution of the vacancy defects was found to be uniform due to the weak dependence of the S parameter on the incident positron energy at $E_+ > 5$ keV. Figure 1(a) shows the average S parameter at $E_+ = 20–30$ keV as a function of annealing temperature. The S parameter decreases at 400–500 and 1200–1500 °C. Vacancy defects are, therefore, annealing out within this temperature range. The average positron lifetime after irradiation (194 ps) is in good agreement with the expected lifetime of positrons at silicon vacancies.[10] From Fig. 1(b), the S–W correlation is almost straight between two circles for silicon vacancies ($S = 1.028$, $W = 0.834$) in 3C–SiC (Ref. 11) and bulk ($S = 1.0$, $W = 1.0$). The above results suggest that silicon vacancies are predominantly responsible for positron trapping.

Figure 2 shows the coincidence Doppler broadening spectra (normalized to the unirradiated sample) after irradia-

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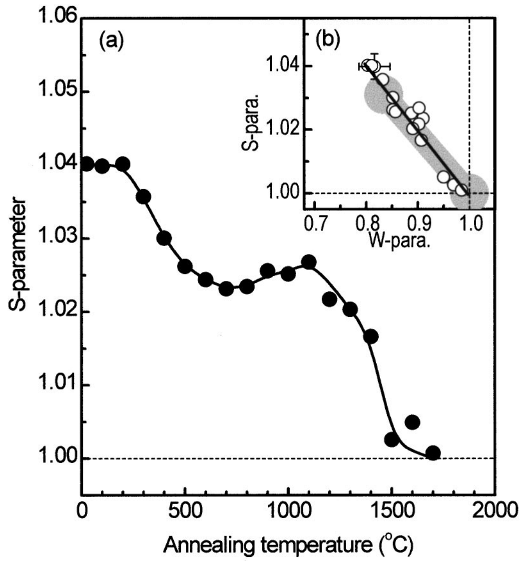

FIG. 1. (a) Average S parameter obtained at $E_+ = 20-30$ keV after electron irradiation to a dose of 3×10^{17} e^-/cm^2 as a function of annealing temperature. The horizontal line at $S=1.0$ denotes the as-grown level. (b) S vs W plotted from the start of annealing through to the end. The upper gray circle at ($S=1.028$, $W=0.834$) denotes the specific S and W parameters for silicon vacancies in 3C–SiC. Another gray circle is located at ($S=1.0$, $W=1.0$) corresponding to the as-grown level.

tion and additional annealing at 1000 °C for 30 min. The horizontal axis has been translated to electron momentum, $p=2\Delta E/c=3.9\times10^{-3}\ \Delta E\, m_0 c$, where ΔE is the Doppler broadening of annihilation radiation in keV, c the light speed, and m_0 the static electron mass. In the Doppler spectrum of SiC, the valence electron component is predominant at $p<1\times10^{-2}\ m_0c$. This component vanishes mostly up to $p=2\times10^{-2}\ m_0c$ and, hence, the core electron component turns up at $p>2\times10^{-2}\ m_0c$. As shown in Fig. 2, the increase and decrease in the respective intensities at $p<1\times10^{-2}$ and $p>2\times10^{-2}\,m_0c$ indicate both vacancy-related enhanced and reduced annihilation probabilities with valence and core electrons, respectively. A close fit between the two

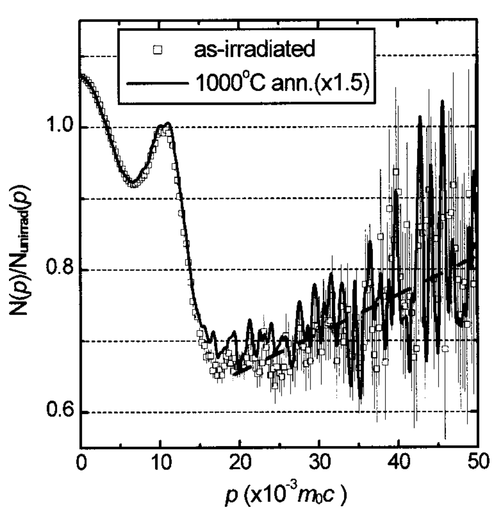

FIG. 2. Coincidence Doppler broadening spectra [ratio to unirradiated sample, i.e., $N(p)/N_{\mathrm{unirrad}}(p)$] obtained after electron irradiation to a dose of 3×10^{17} e^-/cm^2 and subsequent annealing at 1000 °C. For direct comparison of the two spectra, the intensity of the latter spectrum is amplified by a factor of 1.5 $\{[N(p)/N_{\mathrm{unirrad}}(p)-1]\times1.5+1\}$. The broken line is a guide for the eye.

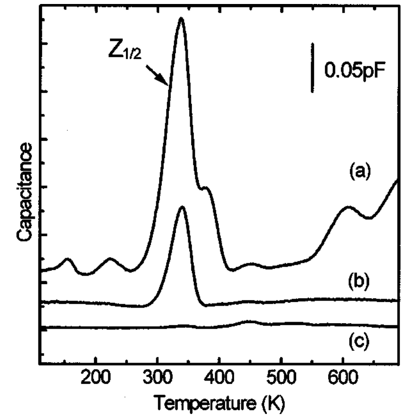

FIG. 3. DLTS spectra after electron irradiation to a dose of 1×10^{15} e^-/cm^2 and subsequent annealing at (a) 430 °C (after the first DLTS measurement) and (b) 1200 °C and (c) 1600 °C. The rate window is defined from 2 to 4 ms.

spectra suggests that the type of vacancy-related defect responsible for trapping is the same before and after annealing. This is consistent with the straight $S-W$ correlation shown in Fig. 1(b). The spectral intensities have a tendency to increase at $p>2\times10^{-2}\ m_0c$. Carbon core electrons ($1s$) give rise to greater Doppler broadening as compared to silicon core electrons (mainly, $2sp$).[12] The increased intensity in the high momentum region can, therefore, be attributed to a preferential annihilation of positrons with carbon core electrons. Hence, silicon vacancies are acting as the main source of positron trapping.

The above results show that there are two steps to the annealing process. Isolated silicon vacancies are normally annealed out below 1000 °C.[12–15] The first annealing process at 400–500 °C in Fig. 1(a) can be readily attributed to the disappearance of isolated silicon vacancies. The final annealing process above 1200 °C must be explained in terms of complexes, which have a higher thermal stability than isolated silicon vacancies. Similar results to these have been obtained for 6H–SiC epilayers.[16] During electron irradiation carbon vacancies are also generated.[14,15,17] However, no apparent annealing of carbon vacancies was observed in this case. This is probably due to a small increase in the S parameter for carbon vacancies because of the weak localization of the positron wave function and/or inefficient positron trapping at carbon vacancies.[18]

Figure 3 shows DLTS spectra after electron irradiation to a dose of 1×10^{15} e^-/cm^2 and subsequent annealing at 430 °C (after the first scan) and 1200 and 1600 °C. Based on its energy level (0.69 eV below the conduction band) and the capture cross section ($8\times10^{-16}\,cm^2$), the most prominent peak at 334 K can be assigned to the $Z_{1/2}$ level. Several small peaks not related to the $Z_{1/2}$ level are also observed after annealing at 430 °C. The concentrations of these small peaks are approximately one order of magnitude smaller than that of the $Z_{1/2}$ levels and drastically decrease after annealing at 1200 °C. Thus, in the high-temperature annealing process, the $Z_{1/2}$ levels can be correlated with PAS detected vacancies. Figure 4 shows the annealing behavior of the positron-trapping rate, which is proportional to the vacancy and concentration, and the concentration of the $Z_{1/2}$ levels above 600 °C. The positron-trapping rate of vacancies was calcu-

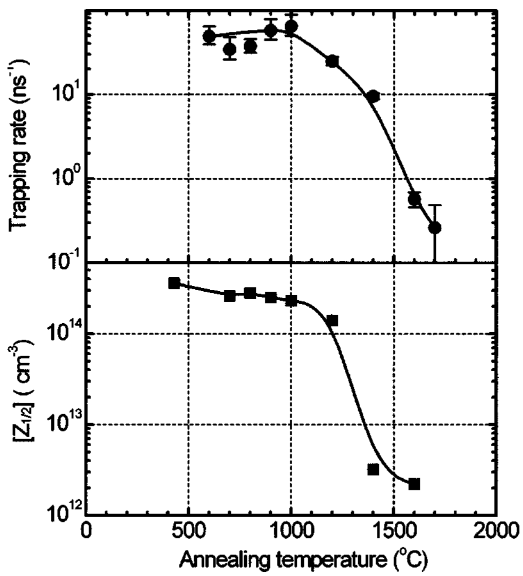

FIG. 4. Positron trapping rate of vacancy-type defects deduced from the S parameter shown in Fig. 1 and the concentration of the $Z_{1/2}$ levels shown in Fig. 2 as a function of annealing temperature.

lated using $\kappa = \lambda_B(S-1)/(S_V - S)$, where λ_B is the positron annihilation rate in the bulk (≈ 7 ns^{-1}) and S_V the specific S parameter for vacancies.[8] Here, we assumed $S_V = 1.028$ for silicon vacancies.[11] As shown in Fig. 4, the annealing behavior of the $Z_{1/2}$ levels is in good agreement with that of the positron-trapping rate. We can, therefore, conclude with some certainty that the $Z_{1/2}$ levels originate from silicon vacancy-related defects, as are the $E_{1/2}$ levels in the case of 6H–SiC.[16]

In summary, we examined the annealing behavior of vacancy defects and the $Z_{1/2}$ levels in 2 MeV electron-irradiated n-type 4H–SiC epilayers. A distinct correlation between the rate of annealing of the $Z_{1/2}$ levels and silicon vacancy-related defects suggests that silicon vacancies are primarily responsible for the existence of the $Z_{1/2}$ levels.

One of the authors (A.K.) would like to acknowledge the financial support of the Alexander von Humboldt Foundation.

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