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ABSTRACT

The impact of oxidation temperature on the formation of single photon-emitting defects located at the silicon dioxide (SiO_2)/silicon carbide (SiC) interface was investigated. Thermal oxidation was performed in the temperature range between 900 and 1300 °C. After oxidation, two different cooling processes—cooling down in N_2 or O_2 ambient—were adopted. Single photon emission was confirmed with second-order correlation function measurements. For the samples cooled in an N_2 ambient, the density of interface single photon sources (SPSs) increased with decreasing oxidation temperature with a density that could be controlled over the 10^5 to 10^8 cm^{-2} range. For the O_2 cooled samples, on the other hand, many interface SPSs were formed irrespective of the oxidation temperature. This is attributed to the low-temperature oxidation during the cooling process after oxidation.

I. INTRODUCTION

A single photon source (SPS) is recognized as one of the most promising key components to realize quantum sensing and quantum cryptography. Some point defects in wide bandgap semiconductors are known to exhibit single photon emission and are known as "color centers." They have several advantages compared to fluorescence of a wide range of quantum systems (e.g., trapped ions, single molecules, and carbon nanotubes), such as room temperature operation and integration with semiconductor devices. The negatively charged nitrogen-vacancy in diamonds emits single photons at room temperature and is widely acknowledged as the most investigated and advanced color center.[1–3] Silicon carbide (SiC) also harbors color centers, and the negatively charged silicon vacancy (V_{Si}^-) receives much attention.[4–8] Controlling the spin of a single V_{Si}^- has been reported.[9] Although such color centers are promising, one of the disadvantages is their relatively low brightness and low spin contrast. SPSs also exist at the interface of silicon dioxide (SiO_2) and SiC, and they exhibit photons at least an order of magnitude brighter than the other color centers in SiC.[10–14] However, the defect structure of the interface SPS has not been clarified and its density is not well controlled. Since the interface SPSs can be easily formed by thermal oxidation, the formation process of thermal oxides differs from one laboratory to another, leading to a poor understanding of the interface SPSs.

In this study, the impact of oxidation temperature on the density of the interface SPS was investigated. The density of the interface SPSs can be controlled over a broad range from 10^5 to 10^8 cm^{-2} by simply changing the oxidation temperature. The samples oxidized at low temperatures include high densities of the interface SPS. The similarity in the oxidation temperature dependence of the interface SPS to that of the interface states near the conduction band edge indicates that the origin of the interface SPSs is likely a carbon-related defect. In addition to that, defects at the SiO_2/SiC interface play an important role as not only SPSs for quantum technology but scattering centers in a metal–oxide–semiconductor field-effect tran-

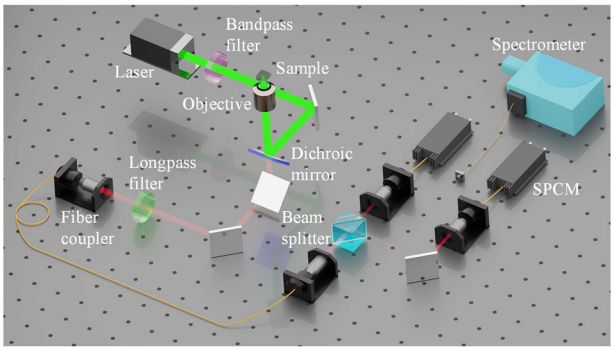

FIG. 1. Experimental setup. Laser light is focused using an objective lens. The fluorescence collected with the objective lens is analyzed using the single-photon counting modules or the spectrometer.

sistor for power electronics. Since the interface SPSs originate from some kinds of SiO_2/SiC interface defects, several research groups have recently proposed that the photoluminescence (PL) mapping for the interface SPSs can be an alternative method to characterize the interface quality.[13,15,16] From this point of view, the relationship between the SPS density and interface state density (D_{it}) obtained in this study is also briefly discussed.

II. EXPERIMENTS

High-purity semi-insulating 4H-SiC(0001) substrates grown by high-temperature chemical vapor deposition were used in this study. The substrates were dipped in H_2SO_4 + H_2O_2, HCl, HCl + HNO_3, and HF solutions. Then, the substrates were rinsed in de-ionized water and loaded into an oxidation furnace. The furnace temperature was raised to the oxidation temperature at a ramping rate of 20 °C/min with an O_2 gas flow. The oxidation temperature was changed from 900 to 1300 °C. The oxidation time varied in the range of 5 min to 20 h. After the thermal oxidation, we adopted two different cooling processes: (i) cooling down in O_2 ambient (O_2 cooling) and (ii) changing the gas flow from O_2 to N_2 before cooling (N_2 cooling). The cooling rate was 5 °C/min for both cooling processes. The SiO_2 thickness was in the range of 2 to 35 nm, which was measured by spectroscopic ellipsometry.

The optical experimental setup consisted of a stage scanning confocal microscope, as shown in Fig. 1.[17,18] The excitation laser was a continuous-wave laser (λ = 532 nm), and the laser power was 6–9 mW before an objective lens. Note that the defects characterized and analyzed in this study are limited to those accessible by the 532-nm laser. When a laser with another wavelength is used, different defects can be measured.[10] The samples were mounted on a three-axis piezo actuator and scanned at a step size of 200 nm with an objective lens with a numerical aperture of 0.9. The luminescence from the samples was collected with the same objective lens and measured by a

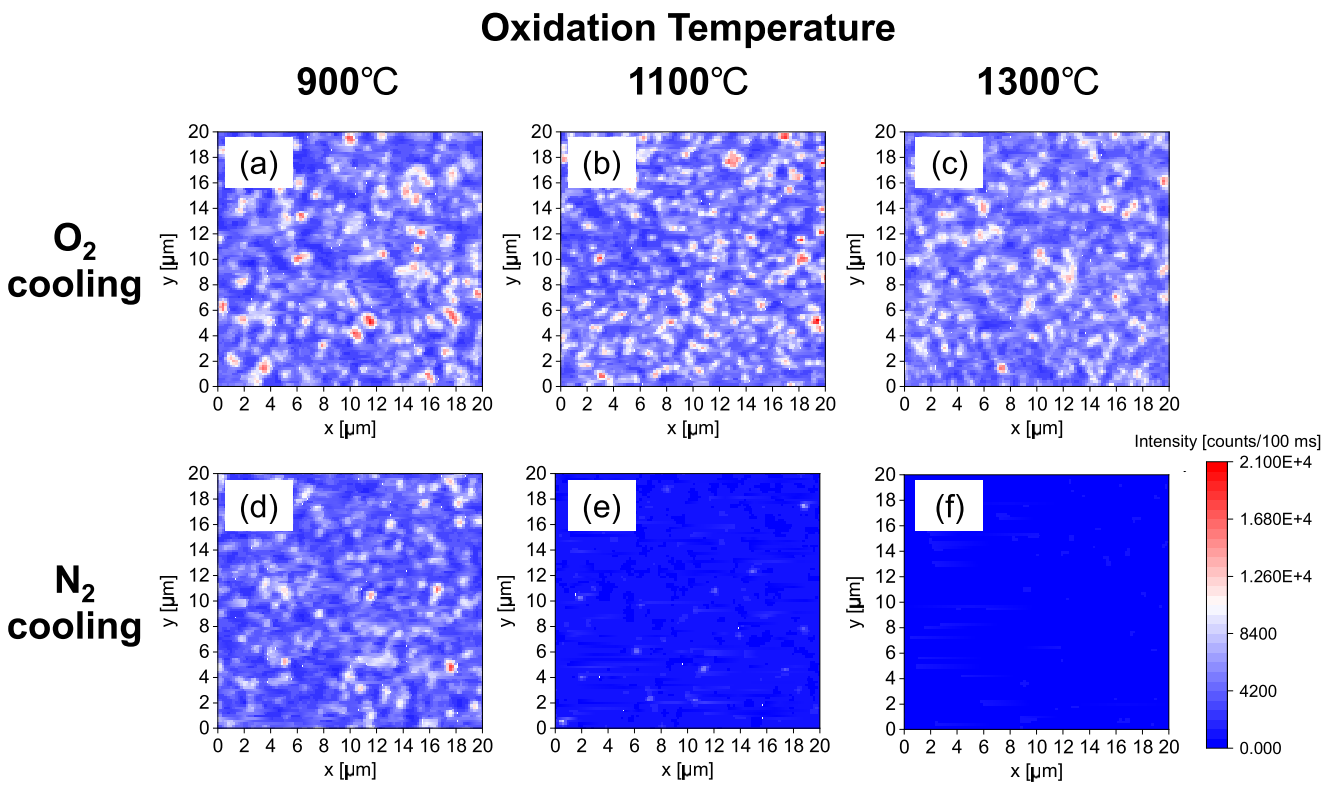

FIG. 2. Confocal scanning images (20 × 20 μm^2) of the SiC samples oxidized at the temperatures of 900, 1100, and 1300 °C with (a)–(c) O_2 cooling and (d)–(f) N_2 cooling.

single-photon counting module (SPCM) or a spectrometer equipped with a charge coupled device camera. The second-order correlation function $g^2(\tau)$ was also measured using two SPCMs. In order to cut the excitation light, a 575 or 600-nm long-pass filter was placed in front of the multi-mode fiber with a core diameter of about 10 μm (SMF-28) to propagate the photons to the SPCMs or the spectrometer, respectively. We manually counted the number of bright spots in the PL mapping image. Note that a bright spot may include more than one SPS when SPSs are located within the diffraction-limited volume. All measurements were performed at room temperature.

We also prepared n-type 4H-SiC (0001) epilayers with a donor density of 1×10^{17} cm^{-3} on n-type substrates to form metal–oxide–semiconductor capacitors. The n-type epilayers were chemically cleaned and oxidized at 1300 °C at the same time as the semi-insulating substrates were oxidized. After the oxidation, circular Al electrodes were deposited (diameter: 300–500 μm). The quasi-static and 1 MHz capacitance-voltage measurements were performed to extract the D_{it}.

III. RESULTS

Figure 2 shows confocal scanning images of the samples oxidized at temperatures of 900, 1100, and 1300 °C. Figures 2(a)–2(c) and 2(d)–2(f) are acquired from the samples with O_2 cooling and N_2 cooling, respectively. In the case of O_2 cooling, lots of bright spots are confirmed and no significant difference can be seen among the samples oxidized at the different oxidation temperatures. On the other hand, bright spots remarkably decreased with elevating the oxidation temperature for the N_2 cooled samples [Figs. 2(d)–2(f)]. We did not observe any single bright spots from the sample oxidized at 1300 °C with N_2 cooling even when the scanning area was expanded to 50 ×50 μm^2.

The second-order correlation function $g^2(\tau)$ of bright spots in the samples oxidized at 1100 °C with O_2 cooling and N_2 cooling is depicted in Figs. 3(a) and 3(b), respectively. Note that the experimental data were normalized and corrected in the way described in Ref. 19. A dip at around zero delay is not clearly obtained from the sample with O_2 cooling. The bright spot of the sample with O_2 cooling may consist of a few SPSs owing to its high density [Fig. 2(b)]. On the other hand, from the sample with N_2 cooling, a sharp dip is observed, meaning that well isolated SPSs were formed. The red curve shown in Fig. 3(b) shows the fitting result using a three-level model.[20] The $g^2(0)$ is estimated to be 0.2, which is less than 0.5. Then, bright spots observed in the scanning images are regarded as interface SPSs. Note that one main reason why $g^2(0)$ is not zero even for the fitting result is due to the timing jitter of the phonon detectors.[21] The photoluminescence (PL) spectra from bright spots in the samples oxidized at 1100 °C with O_2 cooling and N_2 cooling are shown in Figs. 3(c) and 3(d), respectively. The spectra exhibit wide variability in the emission peaks. Note that the peaks include zero-phonon lines and phonon sidebands. The peak wavelength is in the range

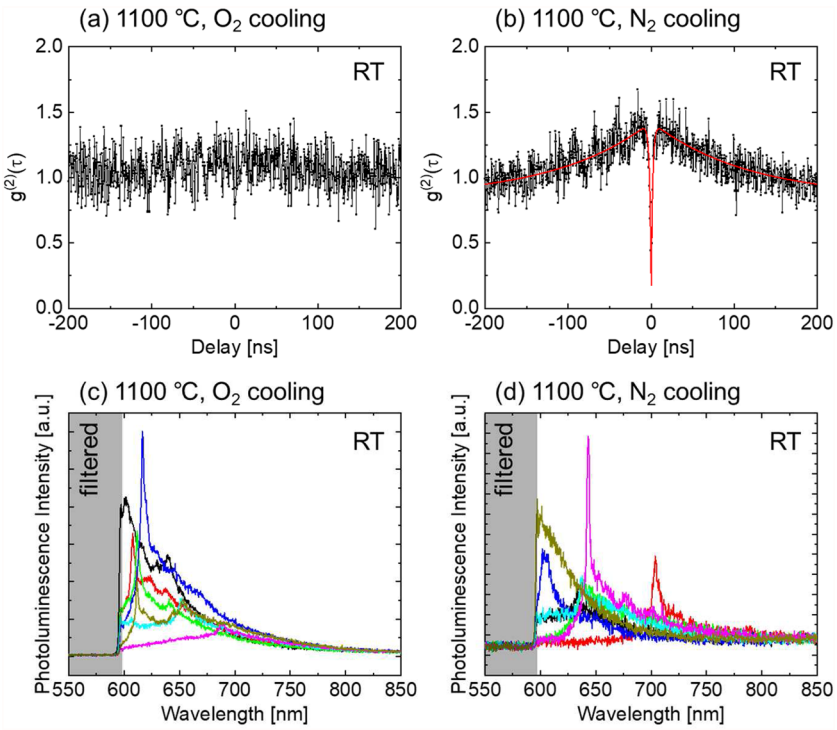

FIG. 3. A second-order correlation function $g^2(\tau)$ of bright spots in the samples oxidized at 1100 °C with (a) O_2 cooling and (b) N_2 cooling. The red curve shown in (b) represents the fitting result using a three-level model. Examples of photoluminescence spectra from bright spots in the sample oxidized with (c) O_2 cooling and (d) N_2 cooling.

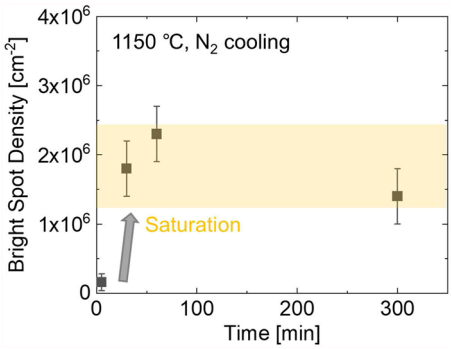

FIG. 4. Dependence of bright spot density on oxidation time at the oxidation temperature of 1150 °C.

between 600 and 750 nm, which is consistent with previous studies using an excitation laser with a wavelength of 532 nm.[12,13] Many peaks in the PL spectra from the O_2 cooling sample are detected, whose number is larger than that from the N_2 cooling sample, supporting that $g^2(0)$ becomes unity in the O_2 cooling sample [Fig. 3(a)] due to several SPSs located at bright spots within the excitation volume of the laser.

Figure 4 depicts the oxidation time dependence of the bright spot density. The oxidation was performed at a temperature of 1150 °C with N_2 cooling, and the oxidation time varied from 5 to 300 min. The bright spot density does not strongly depend on the oxidation time and reaches around 2×10^6 cm^{-2} except for the sample that was oxidized for 5 min. The relatively low density of the 5-min oxidation sample is attributed to the very short oxidation time (SiO_2 thickness: 3 nm). Bright spot density saturates at a certain value soon after oxidation proceeds.

The oxidation temperature dependence of the bright spot density is shown in Fig. 5(a). As shown in Figs. 2(a)–2(c), the bright spot density of the samples with O_2 cooling is high and does not depend on the oxidation temperature. On the other hand, the bright spot density of the samples with N_2 cooling dramatically decreases with elevating the temperature and control of the bright spot (interface SPS) density in the range from 10^5 to 10^8 cm^{-2} is achieved by changing the oxidation temperature.

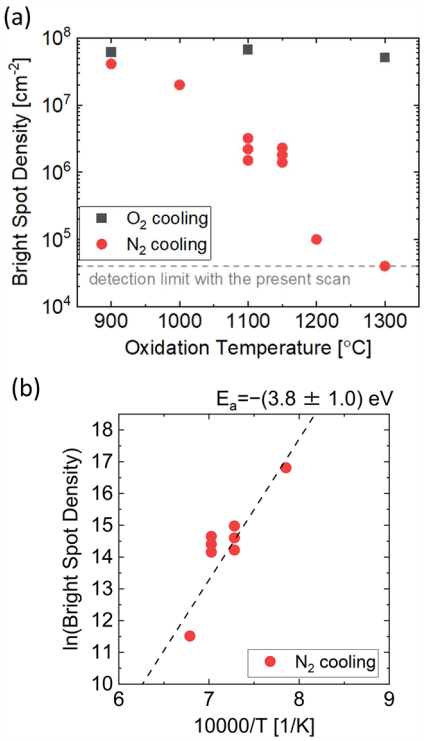

FIG. 5. (a) Bright spot density vs oxidation temperature in the SiO_2/SiC structure. Black squares and red circles represent the samples with O_2 cooling and N_2 cooling, respectively. (b) Arrhenius plot of the bright spot density (N_2 cooling).

IV. DISCUSSION

First, we discuss why the bright spot (interface SPS) density does not vary depending on the oxidation temperature for the samples with O_2 cooling. During the cooling period after the oxidation with an O_2 gas flow, oxidation at the SiO_2/SiC interface slightly proceeds. The bright spot density in the samples with O_2 cooling was close to that in the sample oxidized at the lowest temperature (900 °C) with N_2 cooling. According to previous studies, the SiC(0001) surface is known to be oxidized at 900 °C, whereas oxidation hardly proceeds at 800 °C.[22] Considering that all the samples with O_2 cooling were slightly oxidized at 900 °C or lower during the cooling process, the present result suggests that the bright spot density is determined by the temperature at which the oxidation takes place at last, regardless of what temperature the sample was previously oxidized at. To examine this hypothesis, we prepared a SiC sample oxidized at a low temperature (900 °C) with O_2 cooling and subsequently oxidized the SiC at a high temperature (1300 °C) with N_2 cooling. Confocal scanning images of this sample (not shown) revealed that bright spots, whose density was consistent with the density extracted from Fig. 2(a) (900 °C, O_2 cooling), were formed at the first oxidation step, and they were removed by the next high-temperature oxidation, which clearly indicates that the bright spot density is determined by the oxidation condition performed at last.

The defect structure of SPSs at a SiO_2/SiC interface has not been identified yet, and we briefly discuss the origin of the SPSs. A SiO_2/SiC interface works as a channel of a SiC metal–oxide–semiconductor field-effect transistor, which is of importance as a next generation power device to reduce energy loss, and intensive research has been done to investigate defects at the SiO_2/SiC interface.[23] It has been reported that unintentional oxidation during cooling down after oxidation introduces interface states whose density is higher than that in the sample without oxidation during the cooling process.[24] Reference 24 reported that the D_{it} (and flat band shift) does not depend on the oxidation temperature for the samples oxidized with O_2 cooling. The reason for a lower density of interface states in the sample oxidized without unintentional oxidation during cooling down was explained by favorable out-diffusion of CO_x, whereas excess carbon atoms remained at the SiO_2/SiC interface when low-temperature oxidation occurred.

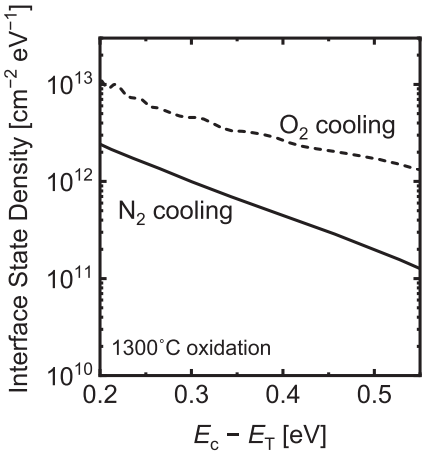

FIG. 6. Energy distribution of the interface state density of the sample oxidized at 1300 °C with O_2 and N_2 cooling extracted by the high (1 MHz)-low method.

High-temperature oxidation effectively reduces such excess carbon-related defects (interface states).[25] The energy distributions of the D_{it} estimated by a high (1 MHz)-low method are given in Fig. 6. The lower D_{it} was obtained from the sample oxidized with N_2 cooling, which is the same trend given in the literature.[24] Taking account of the similarity between the temperature dependence of the D_{it} and the bright spot density, the SPSs observed in this study may originate from carbon-related defects. Figure 7 illustrates excitation and emission polarization measurements of bright spots observed at the SiO_2/SiC interface. As previously reported,[11,26] we also confirmed that the interface SPSs measured in this study hold emission and absorption dipoles that are aligned with the SiC lattice, indicating that the defects are located on the SiC side (not in the oxide layer). First-principles calculations predict that dicarbon antisites (defects), which are located on the SiC side, are easily introduced by low-temperature oxidation and form multiple energy levels inside the energy bandgap of SiC.[27,28] Such a carbon-related defect is a possible origin of the interface SPSs. Note that the Arrhenius plot of the bright spot density [Fig. 5(b)] yields the activation energy of $-(3.8 \pm 1.0)$ eV, which is close to the absolute value of the activation energy of the SiO_2 growth rate (2.9 eV).[24] The removal rate of the SPSs, or the reduction rate of the excess carbon-related defects, may be determined by the oxidation rate.

The origin of the SPS interface is a controversial issue. Hijikata *et al.* proposed that oxygen-vacancy defects are the possible origin of the interface SPSs by comparing their experimental results with the *ab initio* study.[14] Although we believe that the results obtained in this study can be explained by considering the origin of the interface SPSs as carbon-related defects, our study does not exclude oxygen-vacancy defects as a candidate for the origin of the interface SPSs. Further studies are required to clarify the specific defect structure of the interface SPS.

As briefly discussed in the Introduction, PL mapping receives attention as a technique to characterize the SiO_2/SiC interface quality. Woerle *et al.* reported that the interface SPS density depends on the D_{it}, which was electrically measured.[15] In this study, the higher D_{it} was obtained from the sample with the highest SPS density

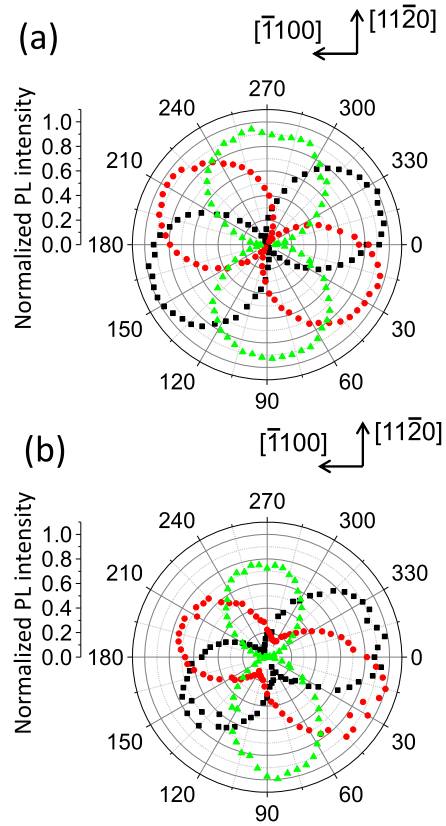

FIG. 7. (a) Excitation and (b) emission polarization measurements of three bright spots observed at the SiO_2/SiC interface. The red, blue, and yellow dots show the measurement results for different bright spots.

(1300 °C with O_2 cooling) compared with the sample with almost no SPSs (1300 °C with N_2 cooling), which partly agrees with the results given in the literature.[13,15] It is well known that the as-oxidized SiO_2/SiC interface includes high D_{it} and post-oxidation annealing in nitric-oxide (NO) effectively reduces D_{it}. Even for the sample with low SPS density (1300 °C with N_2 cooling) obtained in this study, the D_{it} is still high compared to that of the NO-annealed SiO_2/SiC interface.[29] Then, we conclude that a low SPS density indicates that the SiC was oxidized at a high temperature but does not ensure that the D_{it} is as low as that with NO annealing. Although PL mapping using a confocal microscope could be a noncontact, nondestructive, spatially resolved technique to assess the quality of the SiO_2/SiC interface, interface states consist of many types of defects and further studies are required to clarify which type of defects can be characterized by the PL mapping method.

V. CONCLUSION

In this study, the oxidation temperature of SiC was systematically varied and its impact on the density of photon-emitting defects was investigated. For the samples cooled in an N_2 ambient, the bright spot density monotonically decreased with increasing the oxidation temperature and was controlled in the wide range from 10^5 to 10^8

cm^{-2}. The SPSs tended to be formed by oxidation at low temperatures. The similarity between the temperature dependence of the SPS density and that of the SiO_2/SiC interface state density indicates that the origin of SPSs is likely a carbon-related defect. Identification of the atomistic structure of the defect is required for understanding and controlling the physical properties of the defect.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

Mitsuaki Kaneko: Conceptualization (lead); Data curation (lead); Formal analysis (lead); Investigation (lead); Project administration (lead); Validation (lead); Visualization (lead); Writing – original draft (lead). **Hideaki Takashima**: Data curation (supporting); Formal analysis (supporting); Investigation (supporting); Methodology (lead); Software (lead); Writing – review & editing (supporting). **Konosuke Shimazaki**: Data curation (supporting); Investigation (supporting); Methodology (supporting); Visualization (lead); Writing – review & editing (supporting). **Shigeki Takeuchi**: Funding acquisition (lead); Resources (lead); Supervision (lead); Writing – review & editing (lead). **Tsunenobu Kimoto**: Funding acquisition (lead); Resources (lead); Supervision (lead); Writing – review & editing (lead).

DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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