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Deep level transient spectroscopy on as-grown and electron-irradiated *p*-type 4H-SiC epilayers

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The authors have investigated deep levels in as-grown and electron-irradiated *p*-type 4H-SiC epilayers by deep level transient spectroscopy. In as-grown epilayers, the D center and four deep levels are observed. In *p*-type 4H-SiC, reactive ion etching followed by thermal treatment (at 1150 °C) induces the HK0 ($E_V+0.79$ eV) and HK2 ($E_V+0.84$ eV) centers. By the electron irradiation, two deep levels at 0.98 eV (EP1) and 1.44 eV (EP2) are observed in all the samples irradiated at 116–400 keV, while two additional deep levels (EP3 and EP4) are observed only in the samples irradiated at 400 keV. After annealing at 950 °C, these centers are annealed out, and the HK4 ($E_V+1.44$ eV) concentration is increased. By the electron irradiation at more than 160 keV followed by annealing at 950 °C, three deep levels are always observed at 0.30 eV (UK1), 0.58 eV (UK2), and 1.44 eV (HK4). These centers may be defect complexes including carbon-displacement-related defects. All the centers except for the D center are reduced to below the detection limit ($1-3 \times 10^{11}$ cm⁻³) by annealing at 1550 °C for 30 min. © 2007 American Institute of Physics. [DOI: [10.1063/1.2730569](https://doi.org/10.1063/1.2730569)]

I. INTRODUCTION

Silicon carbide (SiC) is an attractive material for realizing high-power, high-temperature, and high-frequency devices, owing to its superior properties such as wide bandgap, high breakdown field, high thermal conductivity, and high saturation electron drift velocity.¹ 4H-SiC has been regarded as the most promising polytype for vertical-type high-voltage devices, due to higher bulk mobility and smaller anisotropy. To realize high-performance power devices which possess high blocking voltage (more than 5 kV) and low on-resistance at the same time, bipolar devices such as pin diodes, thyristors, and insulated gate bipolar transistors are more attractive than unipolar devices such as Schottky diodes and metal-oxide-semiconductor field effect transistor owing to the effect of conductivity modulation.²

Deep levels act as recombination and generation centers in such bipolar devices. The concentration of deep levels should be decreased because long carrier lifetime is essential to obtain sufficient conductivity modulation. Too long a lifetime, on the other hand, will cause considerably large reverse recovery, leading to limited switching frequency and excessive switching loss. Therefore, the concentrations of deep levels must be controlled to achieve an optimum lifetime value and its profile. Control of deep levels is also essential to reproducibly obtain high-purity semi-insulating substrates.^{3,4} Electron irradiation is one of the promising methods to control the concentrations of deep levels in silicon.^{5,6} For *n*-type SiC, electron irradiation has been performed by several groups.⁷⁻⁹ For *p*-type SiC, however, very few systematic investigations have been done and limited information is available about deep levels in *p*-type epilayers (hole traps).¹⁰⁻¹⁴ Study on hole traps is essential for devel-

oping SiC bipolar devices because hole traps possibly act as minority carrier (hole) traps in active layers (*n*-type layers) of most SiC bipolar devices.

In this work, the authors have investigated hole traps in as-grown and electron-irradiated *p*-type 4H-SiC epilayers by deep level transient spectroscopy (DLTS).

II. EXPERIMENT

Samples used in this study were Al-doped *p*-type 4H-SiC(0001) epilayers grown by chemical vapor deposition. The net acceptor concentration of the samples determined by capacitance-voltage (*C-V*) measurements was in the range from 6.5×10^{15} cm⁻³ to 2.8×10^{17} cm⁻³ and the thickness was 5–10 μm.

Titanium was thermally evaporated onto the sample surface as Schottky contacts with a thickness of approximately 70 nm. The typical diameter of Schottky contacts was 1500 μm. A Ti/Al/Ni (10 nm/150 nm/50 nm) layer annealed at 950 °C for 10 min was employed as backside Ohmic contacts in order to obtain reliable capacitance values in *C-V* and DLTS measurements.

Deep levels in *p*-type 4H-SiC epilayers were measured by DLTS in the temperature range from 150 to 700 K. If not specified, the reverse bias was kept at 5–10 V and the pulse voltage of 0 V with a pulse width of 1 ms was applied in DLTS measurements. A period width of 0.2 s was employed for all the DLTS measurements performed in this study. A Fourier transform analysis¹⁵ of the measured transients was employed. Temperature-independent capture cross section was assumed when analyzing the DLTS data. The error in determination of the activation energy is less than ±0.05 eV for low-temperature DLTS peaks and ±0.1 eV for high-temperature peaks. In this article, the trap concentrations have been corrected by considering the lambda effect.¹⁶

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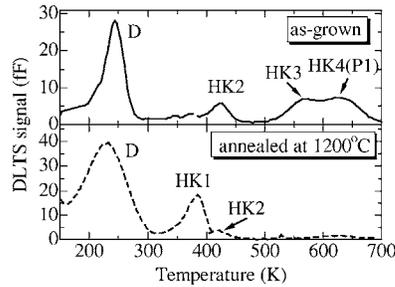


FIG. 1. DLTS spectra of the as-grown *p*-type 4H-SiC epilayer (solid line) and sample annealed at 1200 °C for 10 min (broken line).

The authors performed electron irradiation at 116–400 keV with a fluence of 3×10^{15} – 3×10^{18} cm⁻². By irradiation at lower than 200 keV, displacement of carbon atoms mainly takes place and at the energy higher than 200 keV, displacement of both carbon and silicon atoms is introduced.¹⁷ All the irradiation was performed in atmospheric nitrogen ambient without intentional heating. The annealing following the electron irradiation was performed at 950–1800 °C in Ar atmosphere for 30 min.

III. RESULTS

A. Deep levels in as-grown epilayers

Figure 1 shows the DLTS spectra of an as-grown epilayer and a sample annealed at 1200 °C for 10 min. In the as-grown sample, the D ($E_V+0.49$ eV, E_V : top of the valence band) (Refs. 18 and 19), HK2 ($E_V+0.84$ eV), HK3 ($E_V+1.27$ eV), and HK4 ($E_V+1.44$ eV) (Ref. 14) centers were dominant in this temperature range. The activation energy (E_a) and capture cross section (σ) of the centers obtained by the Arrhenius plot of emission time constant are summarized in Table I. The D center is a well-known trap detected in *p*-type SiC. This center originates from a B-related defect, boron at C site (B_C) or boron at Si site (B_{Si}) and carbon

TABLE I. Electrical properties of deep levels observed in as-grown and irradiated *p*-type 4H-SiC epilayers. The activation energy (E_a) and capture cross section (σ) are estimated from the Arrhenius plot of emission time constant. The temperature required for annealing out each trap (T_A) is also shown in the table.

Label	E_a (eV)	σ (cm ²)	T_A (°C)	Sample condition for trap observation
D	0.49	1×10^{-16}	>1800	As-grown
HK1	0.68	4×10^{-16}	~1500 ^a	Annealed at 1200–1400 °C
HK2	0.84	4×10^{-14}	1550 ^b	As-grown
HK3	1.27	3×10^{-14}	1350 ^b	As-grown
HK4	1.44	6×10^{-15}	1550	As-grown or irradiation+annealing
HK0	0.79	1×10^{-16}	1550	RIE+annealing
HS2	0.47	3×10^{-17}	1350	As-irradiated (≥ 116 keV)
EP1	0.98	8×10^{-17}	≤ 427	As-irradiated (≥ 116 keV)
EP2	1.44	6×10^{-14}	≤ 427	As-irradiated (≥ 116 keV)
EP3	0.73	2×10^{-15}	≤ 427	As-irradiated (≥ 400 keV)
EP4	1.18	2×10^{-15}	≤ 427	As-irradiated (≥ 400 keV)
UK1	0.30	6×10^{-19}	1350	Irradiation (≥ 160 keV)+annealing
UK2	0.58	8×10^{-16}	1350	Irradiation (≥ 160 keV)+annealing

^aNot shown.

^bData obtained for as-grown samples (Ref. 14).

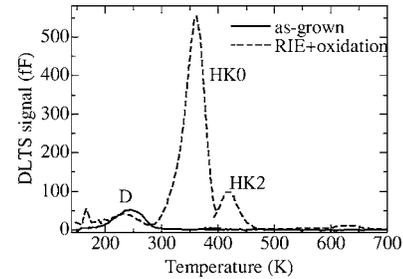


FIG. 2. DLTS spectra of an as-grown *p*-type 4H-SiC epilayer (without RIE, solid line) and sample treated with RIE followed by sacrificial oxidation at 1150 °C for 1 h (broken line). RIE was performed with radio frequency plasma (radio frequency power: 100 W) under CF₄ flow (15 sccm) at 25 Pa for 3 min.

vacancy (V_C).^{20,21} The concentration of the D center in this particular sample was 2.5×10^{13} cm⁻³. For the HK2, HK3, and HK4 centers, their concentrations were in the mid-high 10^{12} cm⁻³ range. These centers were detected in Al-doped epilayers as well as B-doped epilayers.²² The authors detected the trap P1 in Ref. 13, which is energetically located at $E_V+1.49$ eV. Since the energy position of the trap P1 in the band gap is close to that of HK4, the trap P1 might be the same as the HK4 center or the overlapping signals of the HK3 and HK4 centers. Fundamental properties of the HK2, HK3, and HK4 centers have been discussed elsewhere.¹⁴ In the samples annealed at 1200 °C, a trap could be detected at 385 K (HK1), while HK2, HK3, and HK4 concentrations were decreased as shown in Fig. 1. From the Arrhenius plot, the activation energy and capture cross section of the HK1 center were estimated to 0.68 eV and 4×10^{-16} cm², which are also listed in Table I. In this sample annealed at 1200 °C, the HK1 concentration was 1.4×10^{13} cm⁻³. The center was observed only in samples annealed at about 1200–1400 °C.

B. Reactive ion etching-induced deep levels

Reactive ion etching (RIE) is an essential technique to etch SiC surface because other etching techniques such as wet etching are difficult due to chemical and mechanical stability of the material. The authors found that a RIE process induces the generation of deep levels. Figure 2 shows the DLTS spectra of an as-grown sample (without RIE) and a sample treated with RIE and subsequent sacrificial oxidation at 1150 °C for 1 h. RIE was performed with radio frequency plasma (100 W) under CF₄ flow (15 sccm) at 25 Pa for 3 min (etched depth = 150 nm). In this as-grown sample, the D center was dominant. Just after RIE, the DLTS spectrum did not show a significant change (not shown). By performing sacrificial oxidation (or annealing in Ar ambient) at 1150 °C after RIE, however, the HK2 and a peak (HK0) at 360 K were introduced with high signal intensity. By the process, the concentrations were increased to 1.0×10^{15} cm⁻³ for the HK0 center and 2.1×10^{14} cm⁻³ for the HK2 center. The HK0 center could be detected not only near the surface but at a depth of 2 μ m by DLTS using a reverse bias voltage of 50 V. Since this increase was also observed in samples etched by plasma treatment using only O₂ gas and annealed at 1150 °C, the HK0 and HK2 centers may be a complex including a defect, which relates to plasma damage. During the

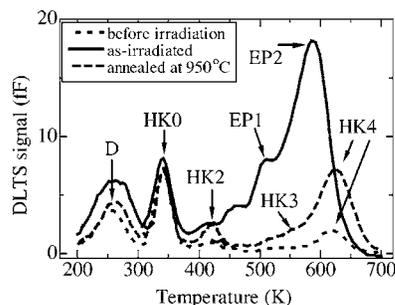


FIG. 3. DLTS spectra of unirradiated *p*-type 4H-SiC (treated by RIE, dotted line), the electron-irradiated sample (energy: 116 keV, fluence: $3 \times 10^{17} \text{ cm}^{-2}$, solid line), and the electron-irradiated sample annealed at 950 °C for 30 min in atmospheric Ar ambient (broken line).

RIE process, intrinsic defects like carbon vacancy, carbon interstitial, silicon vacancy, and silicon interstitial, can be formed by ion bombardment onto the surface. By thermal treatment, they may migrate and form complexes, resulting in formation of the HK0 and HK2 centers. It is of interest that the centers could be found even at a depth of 2 μm , which is deeper than etched depth. Therefore, the origins of the centers may be highly mobile. From the Arrhenius plot, the HK0 center was revealed to be energetically located at $E_V + 0.79 \text{ eV}$. The trap profiles of the HK0 center are listed in Table I. Although the peak temperature of the HK0 center (360 K) is close to that of the HK1 center (385 K), the center is different from the HK1 center because both HK0 and HK1 centers can be occasionally observed at the same time.

C. Deep levels in epilayers irradiated with low-energy electrons (below carbon-displacement threshold)

Figure 3 shows the DLTS spectra of an unirradiated sample (before irradiation), a sample irradiated at 116 keV with a fluence of $3 \times 10^{17} \text{ cm}^{-2}$, and an irradiated sample annealed at 950 °C for 30 min in atmospheric Ar ambient. In the unirradiated epilayer, the D, HK0, and HK4 centers were dominant. Since the samples used in this experiment were treated with RIE and subsequent sacrificial oxidation at 1150 °C, the HK0 center was observed in unirradiated samples, while the HK2 concentration was much smaller than the D, HK0, and HK4 concentrations. By the electron irradiation, two peaks, labeled EP1 and EP2, emerged. From the Arrhenius plot, the activation energy of the EP1 and EP2 centers was estimated as 0.98 and 1.44 eV, respectively, as summarized in Table I. Although the energy level of the EP2 center is very close to that of the HK4 center, they are different centers each other. For example, the capture cross section (Table I) and thermal stability are different between these centers. The EP1 and EP2 concentrations were higher than $5\text{--}16 \times 10^{13} \text{ cm}^{-3}$ in the as-irradiated sample. However, the centers were thermally unstable, and the concentrations were decreased below the detection limit ($1\text{--}3 \times 10^{11} \text{ cm}^{-3}$) after the DLTS measurement up to 700 K. By the low-energy electron irradiation at 116 keV, carbon vacancy (V_C) and carbon interstitials (C_i) can be mainly introduced in SiC samples.¹⁷ Since C_i may be thermally unstable due to its high mobility,^{23,24} EP1 and EP2 may be derived from C_i -related defects. The peak at around 265 K was

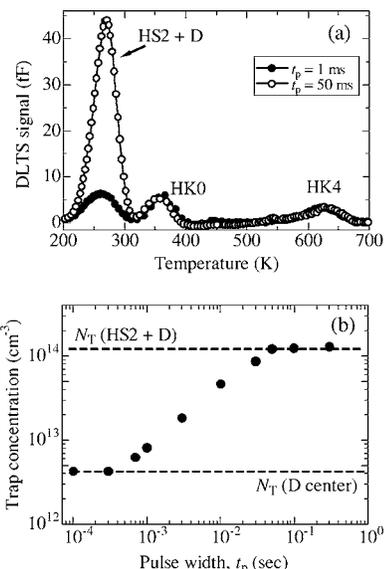


FIG. 4. (a) DLTS spectra of an irradiated 4H-SiC epilayer (annealed at 950 °C) measured with different pulse width (t_p) of 1 and 50 ms. (b) Pulse width dependence of apparent trap concentration (trap concentration calculated from the height of DLTS peak at 270 K).

slightly increased by the irradiation. This increase may not be due to the increase of the D center but due to the overlapping of the D and HS2⁸ centers. The details will be discussed in the latter part of this section. By annealing at 950 °C for 30 min, the HK4 concentration was increased from $1.5 \times 10^{12} \text{ cm}^{-3}$ to $4.0 \times 10^{12} \text{ cm}^{-3}$. The HK4 center may be a complex including V_C and/or C_i since the thermal annealing is required for the increase of the HK4 concentration.

DLTS measurements were performed with different pulse width (t_p) of 1 and 50 ms to study the deep levels detected at about 265 K. The DLTS spectra of the sample irradiated at 116 keV with a fluence of $3 \times 10^{17} \text{ cm}^{-2}$ (annealed at 950 °C) are shown in Fig. 4(a). The peak intensity at about 265 K was significantly increased by increasing pulse width (t_p) from 1 to 50 ms, while no significant change was observed for the other peaks. Since this increase was not observed in unirradiated epilayers but in irradiated ones, this result indicates the existence of an irradiation-induced trap, which has very small capture cross section. The trap (which became detectable with longer filling pulse, t_p) is energetically located at $E_V + 0.47 \text{ eV}$, which may be ascribed to the HS2 center.⁸ Storasta *et al.* detected the HS2 center in electron-irradiated 4H-SiC epilayers by minority carrier transient spectroscopy (MCTS) using t_p of 10 ms and the energy level has been estimated at $E_V + 0.4 \text{ eV}$.⁸ As shown in Fig. 4(a), the HS2 center was the most dominant hole trap in *p*-type epilayers irradiated at 116 keV, in good agreement with the MCTS results in the previous report.¹²

Figure 4(b) shows the relation between the apparent trap concentration calculated from the height of the DLTS peak at 265 K and the pulse width, t_p . The sample used for the plot was irradiated at 116 keV with a fluence of $3 \times 10^{18} \text{ cm}^{-2}$ without annealing [different from that used in Fig. 4(a)]. The apparent concentration was decreased by decreasing t_p . When t_p became shorter than 0.3 ms, the apparent concen-

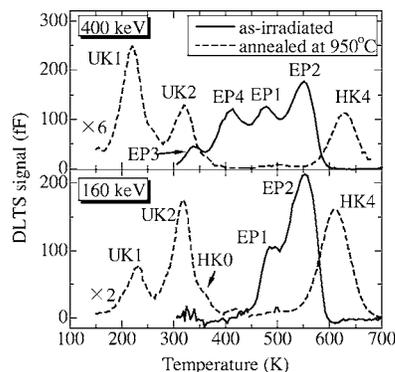


FIG. 5. DLTS spectra of *p*-type 4H-SiC epilayers irradiated at 160 and 400 keV. The solid lines denote the DLTS spectra of as-irradiated samples. The broken lines denote the irradiated sample annealed at 950 °C. The spectra for annealed samples are magnified as indicated in the figure.

tration saturated at $4.2 \times 10^{12} \text{ cm}^{-3}$, which coincides with the concentration of the D center in the as-grown sample. Therefore, the concentration of the D center is not increased by the electron irradiation. By increasing t_p , on the other hand, the apparent concentration was increased. The concentration saturated at $1.2 \times 10^{14} \text{ cm}^{-3}$ when t_p reached 50 ms. This apparent concentration represents the sum of the D and HS2 concentrations. When electron irradiation was performed at 116 keV with a fluence of $3 \times 10^{18} \text{ cm}^{-2}$, the HS2 concentration was $1.2 \times 10^{14} \text{ cm}^{-3}$. From the result, the introduction rate of the center is calculated as $4 \times 10^{-5} \text{ cm}^{-1}$, which is slightly smaller than those of the $Z_{1/2}$ (Ref. 25) and $\text{EH}_{6/7}$ (Ref. 7) centers ($7-8 \times 10^{-5} \text{ cm}^{-1}$).²⁶ Since the HS2 concentration was increased with increasing electron fluence (not shown), the HS2 center may be related to carbon displacement. The capture cross section of the HS2 center estimated from the pulse width dependence of the HS2 concentration is very low ($<10^{-20} \text{ cm}^2$). The capture cross section estimated from the Arrhenius plot of emission time constant is also low. The emission time constant of the HS2 center may be affected by the D center because both centers are detected at the same temperature range.

D. Deep levels in epilayers irradiated with high-energy electrons (above carbon-displacement threshold)

Figure 5 shows the DLTS spectra ($t_p=1 \text{ ms}$) of samples irradiated at 160 or 400 keV. The electron fluence is $3 \times 10^{17} \text{ cm}^{-2}$ for the irradiation at 160 keV and $4 \times 10^{16} \text{ cm}^{-2}$ for the irradiation at 400 keV. In the as-irradiated sample (160 keV), the EP1 and EP2 centers were dominant as in the case of the irradiation at 116 keV (trap concentration \sim high 10^{14} cm^{-3} to low 10^{15} cm^{-3}). When the irradiated sample was annealed at 950 °C for 30 min, the EP1 and EP2 centers were decreased and the UK1, UK2, HK0, and HK4 centers were increased (to low 10^{14} cm^{-3} for the UK1, UK2, and HK4 centers). From the Arrhenius plot, the activation energy of the UK1 and UK2 centers were estimated to be 0.30 eV (UK1) and 0.58 eV (UK2), respectively. The UK1, UK2, and HK0 centers were not increased by the irradiation at 116 keV (Fig. 3) but by the irradiation at 160 keV, although only carbon displacement was introduced

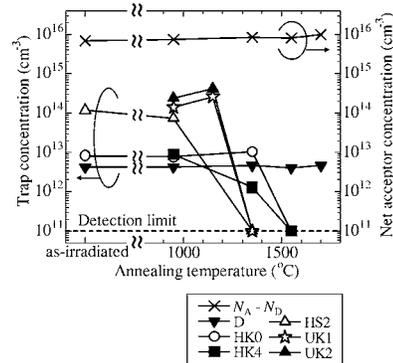


FIG. 6. Annealing temperature dependence of trap concentrations observed in irradiated *p*-type 4H-SiC. The samples irradiated at 116 keV with a fluence of $3 \times 10^{18} \text{ cm}^{-2}$ were used for all the traps except for the UK1 and UK2 centers and the sample irradiated at 160 keV with a fluence of $3 \times 10^{17} \text{ cm}^{-2}$ were for the UK1 and UK2 centers. The net acceptor concentration is also plotted.

by the irradiation at both 116 and 160 keV. In the samples irradiated at 116 keV, increase of the UK1 and UK2 centers might be too small to be detected by DLTS due to the low introduction rate of carbon displacement. For example, the HK4 concentration in the sample irradiated at 116 keV (shown in Fig. 3) was smaller than that at 160 keV by more than one order of magnitude. In this sample irradiated at 160 keV, the HS2 center was also increased by using long t_p of 50 ms (not shown) in DLTS as in Fig. 4.

By the irradiation at 400 keV, not only the EP1 and EP2 centers but also the EP3 ($E_V+0.73 \text{ eV}$) and EP4 ($E_V+1.18 \text{ eV}$) centers were introduced in the as-irradiated sample. Electron irradiation at 400 keV will induce both carbon and silicon displacement. Therefore, the EP3 and EP4 centers might be related to silicon displacement. By annealing at 950 °C, EP1–EP4 centers were annealed out, and the UK1, UK2, and HK4 centers became dominant as in the case of the sample irradiated at 160 keV.

E. Thermal stability of deep levels

Figure 6 shows the annealing temperature dependence of trap (D, HK0, HK4, HS2, UK1, and UK2) concentrations and the net acceptor concentration. The samples irradiated at 116 keV with a fluence of $3 \times 10^{18} \text{ cm}^{-2}$ were used for investigation of thermal stability of the D, HS2, HK0, and HK4 centers. The UK1 and UK2 centers were studied with the sample irradiated at 160 keV with a fluence of $3 \times 10^{17} \text{ cm}^{-2}$. Thermal annealing was performed in the temperature range from 950 to 1700 °C for 30 min in atmospheric Ar ambient. For the net acceptor concentration, it remained almost constant during these annealing experiments. In the as-irradiated sample, the D, HK0, and HS2 centers were observed. The presence of the UK1 and UK2 centers could not be confirmed in as-irradiated samples. The reliable DLTS data could not be obtained in the low temperature range ($<300 \text{ K}$) for the as-irradiated samples, because the samples showed high resistivity at low temperature. After annealing at 950 °C, the HK4, UK1, and UK2 centers emerged. For the UK1 and UK2 centers, their concentrations were slightly increased by annealing at 1150 °C. This result indicates that the UK1 and UK2 centers may originate from

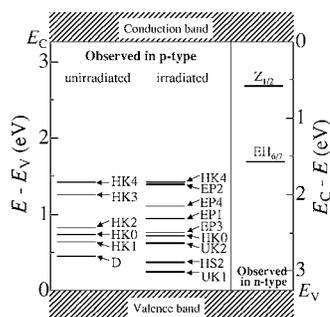


FIG. 7. Overview of ground states of deep levels detected in as-grown and electron-irradiated *p*-type 4H-SiC epilayers. The deep levels detected in *n*-type 4H-SiC are also shown (Refs. 7 and 25).

a complex (including carbon displacement). By annealing at 1350 °C, however, the UK1 and UK2 concentrations became lower than the detection limit ($1-3 \times 10^{11} \text{ cm}^{-3}$). The HS2 center was also annealed out at 1350 °C. Although the HK4 concentration starts to decrease by annealing at 1350 °C, it could be detected with a concentration of about $1 \times 10^{12} \text{ cm}^{-3}$. In the annealing experiment using as-grown samples, the concentration of the HK4 center was reduced to below the detection limit by annealing at 1350 °C.¹⁴ This may be attributed to the remarkably different trap concentrations in the starting materials: The trap concentration is one-order-of-magnitude higher in the irradiated samples than in the as-grown ones. In the samples annealed at 1550 °C, the concentrations of all the traps were reduced to below the detection limit except for the D center. The temperature required for annealing out each trap (T_A) is also summarized in Table I.

IV. DISCUSSION

Figure 7 illustrates an overview of ground states of deep levels detected in unirradiated or electron-irradiated *p*-type 4H-SiC. The major electron traps detected in *n*-type 4H-SiC are also shown. The levels presented by the bold lines [HK0, HK4, UK1, HS2, UK2, EP1, EP2, $Z_{1/2}$, and $\text{EH}_{6/7}$ (Ref. 8)] may be C-displacement-related defects, and the EP3 and EP4 centers may be Si-displacement-related defects. Since the EP1–EP4 centers and HS2 center emerged in as-irradiated samples, they may be derived from isolated defects (single vacancy or single interstitial) or complexes formed at low temperature (lower than room temperature). The origins of the HK0, HK4, UK1, and UK2 centers may be complexes including carbon displacement because their concentration is increased in epilayers irradiated by low-energy electron and annealed after the irradiation. Since theoretical study on the deep levels in the lower half of the bandgap is very limited, comparison between experimental and theoretical results is difficult. To clarify their origins, much more systematic studies are required.

V. CONCLUSIONS

The authors investigated deep levels in as-grown and electron-irradiated *p*-type 4H-SiC epilayers by DLTS. In as-grown epilayers, the D ($E_V+0.49 \text{ eV}$), HK1 ($E_V+0.68 \text{ eV}$), HK2 ($E_V+0.84 \text{ eV}$), HK3 ($E_V+1.27 \text{ eV}$), and HK4 (E_V

$+1.44 \text{ eV}$) centers were dominant. The HK0 center ($E_V+0.79 \text{ eV}$) was introduced by RIE followed by oxidation or thermal annealing at 1150 °C. By the electron irradiation, the EP1 ($E_V+0.98 \text{ eV}$), EP2 ($E_V+1.44 \text{ eV}$), EP3 ($E_V+0.73 \text{ eV}$), and EP4 ($E_V+1.18 \text{ eV}$) centers were introduced. The EP3 and EP4 centers were observed only in the sample irradiated at 400 keV, while the EP1 and EP2 centers were observed in all the samples when irradiation was performed at 116–400 keV. After the annealing at 700 K, the EP1, EP2, EP3, and EP4 centers were annealed out. In the samples annealed at 950 °C, the HK4 concentration was increased. The UK1 ($E_V+0.30 \text{ eV}$) and UK2 ($E_V+0.58 \text{ eV}$) centers were increased only in the samples irradiated at higher than 160 keV followed by annealing. All the centers except for the D center were annealed out at 1550 °C.

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