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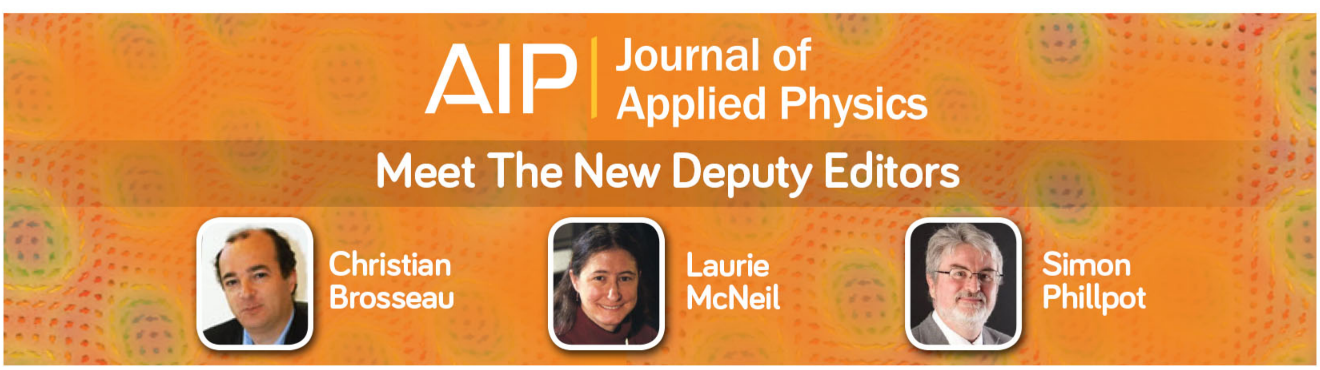

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JOURNAL OF APPLIED PHYSICS **111**, 014910 (2012)

Characterization of deep levels in n-type and semi-insulating 4H-SiC epitaxial layers by thermally stimulated current spectroscopy

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We have investigated deep level centers in n-type and semi-insulating (SI) 4H-SiC epitaxial layers by thermally stimulated current (TSC) spectroscopy. The epitaxial layers were grown using chemical vapor deposition utilizing a dichlorosilane precursor. Both epitaxial layers exhibited relatively shallow levels related to Al, B, L- and D-centers. A deep level center with an activation energy of 1.1 eV, peaked at ~400 K, was detected in the n-type epitaxial layer and correlated with the IL_2 level and the 1.1 eV center in a high purity bulk SI 4H-SiC. The TSC spectra of the SI epitaxial layer was dominated by the peaks at 525–585 K that we attributed to intrinsic defects and their complexes with energy levels close to the middle of the bandgap. The TSC spectra of SI epitaxial layer exhibited peaks with different current polarity which is explained by thermoelectric effect and the built-in electric field reversal. The results of the transfer length method measurements of the SI epitaxial layer and the room temperature current-voltage (I-V) characteristics of both epitaxial layers are also reported. © *2012 American Institute of Physics*. [doi:10.1063/1.3675513]

I. INTRODUCTION

Silicon carbide possesses a great potential for high power, high frequency, and high temperature electronics applications.[1] Chemical vapor deposition (CVD) is usually used to produce high quality SiC epitaxial layers for device fabrication. At present, high growth rate SiC CVD epitaxy has been under development to produce high quality thick epitaxial layers at low cost for high blocking voltage devices.[2–4] It is well known that the electrical properties of an epitaxial layer are controlled by defects and impurities. Both n- and p-type SiC epitaxial layers have been achieved by doping mainly with nitrogen and aluminum, respectively. Semi-insulating SiC films are being produced by impurity doping with deep levels, such as V-doping,[5] and by the natural compensation of residual shallow donors and acceptors by deep levels introduced by intrinsic defects.[6] The latter is used in high purity SI SiC epitaxial growth, which is essential for the development of high performance radiation detectors[7] and fast photo-switching devices.[8] This growth process represents a challenging task since the semi-insulating property is achieved by a compensation mechanism involving intrinsic defects and their complexes, which are difficult to control. Intrinsic defects or complexes involving impurities and intrinsic defects have been reported in as-grown SiC epilayers.[9,10] However, their influence on the properties of the SiC epilayers is not well established and still less is known about their incorporation or formation behavior during the CVD process.

Recently, a CVD epitaxial process utilizing a dichlorosilane precursor to achieve high growth rates has been demonstrated.[2] In this work, we report on the characterization of deep level centers in n-type and SI epitaxial layers grown using this process. To fulfill this task, we have successfully applied TSC spectroscopy, which is known to be a very sensitive technique for investigating point-defects and impurity-related traps in various semiconductors and it was used to characterize deep level centers in high purity bulk SI SiC substrates.[11–13]

II. EXPERIMENTAL PROCEDURES

In this study, we used n-type and SI epitaxial layers grown on 8×8 mm^2 4H-SiC (0001) substrates which were highly doped with nitrogen and were off-cut 8° toward the $[11\bar{2}0]$ direction. The epitaxial growth was carried out in a home-made hot-wall CVD system at 14–20 μm/h growth rate. Dichlorosilane (SiH_2Cl_2, DCS) and propane (C_3H_8) were used as the precursors and hydrogen of 6 slm was employed as the carrier gas. The dilution ratio during growth was ~1000. The flow rates of dichlorosilane and propane were maintained to obtain a C/Si ratio of 1.28 and 1.66 for the n-type and SI epitaxial layer, respectively. Before growth, *in situ* hydrogen etching was performed at 1550 °C for 5–20 min. The growth temperature and pressure were 1550 °C and 80–120 Torr, respectively. No intentional dopants were used during the growth of the semi-insulating epitaxial layer. The thicknesses of the n-type and SI epitaxial layers were ~28 and ~19 μm, respectively. The net doping concentration of the n-type epitaxial layer measured using the high frequency (100 kHz) capacitance-voltage (C-V) method was found to be 3×10^{14} cm^{-3}. The annular mercury probe with an 0.5 mm diameter contact (MSI electronics model Hg-402) was used for the C-V measurements. The high frequency capacitance of the SI epitaxial layer was very

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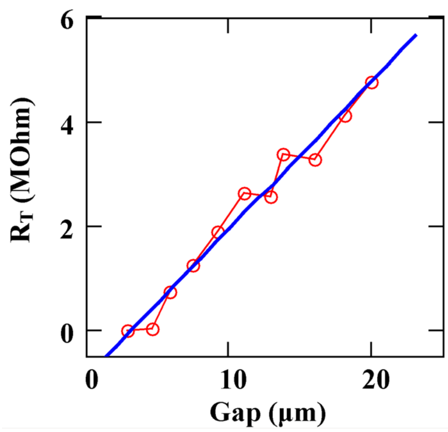

FIG. 1. (Color online) Total resistance vs gap width for the TLM structure on the SI SiC epitaxial layer revealing the resistivity $\geq 1 \times 10^5$ Ω cm. Symbols "o" in the plot indicate experimental data points.

low (~2 pF) and remained practically constant with applied bias indicating the semi-insulating nature of the epitaxial layer. The transfer length method (TLM) measurement performed on a similar epitaxial layer (Fig. 1) confirmed its semi-insulating property.

Nickel contacts, 10–20 nm in thickness, were deposited on top of both epitaxial layers for I-V and TSC measurements using electron beam evaporation (e-beam) system. The 3.2 mm diameter single contact on the n-type epitaxial layer was deposited using a shadow mask, while four contacts 1 mm in diameter on the SI SiC epitaxial layer were formed using the lift-off process. The standard RCA cleaning of the samples was used prior to contact deposition. No annealing was performed after the deposition of Ni contacts.

The room temperature I-V characteristics of the samples (Fig. 2) were measured using a Keithley 237 high voltage source measure unit. The forward I-V characteristic of the device on the n-type epitaxial layer (Fig. 2(a)) is typical for the Ni Schottky contact to the n-type 4H-SiC, revealing the barrier height, ~0.98 eV, and the ideality factor, ~1.36. The reverse I-V characteristic (Fig. 2(b)) exhibited a high leakage current, which might be attributed to the presence of crystallographic defects in the device active region. The I-V characteristics of the devices on the SI epitaxial layers are shown in Figs. 2(c) and 2(d). The current conduction in this case strongly depends on the carrier injection properties of the contact and the SI SiC/n^+-SiC interface and on the deep level centers and defects in the epitaxial layers.

The TSC measurements were conducted in the temperature range of 94–620 K in vacuum $<1 \times 10^{-4}$ Torr at 0 V and ±5 V bias voltages applied to the top Ni contact. The trap filling was achieved by illuminating the samples for ~3 min at 94 K using a UVP model UVM-57 handheld UV lamp specified to produce 302 nm of UV light. The detailed description of our TSC measurement set-up is available in our earlier publication.[11] We have used variable heating rate method[14] to determine the trap activation energy and capture cross-section. The details of the calculation can be found in our work[11] and in Refs. 12 and 15. Briefly, the TSC spectra were obtained at 4, 8, and 15 K/min heat rates revealing a given trap center by a peak with a maximum temperature,

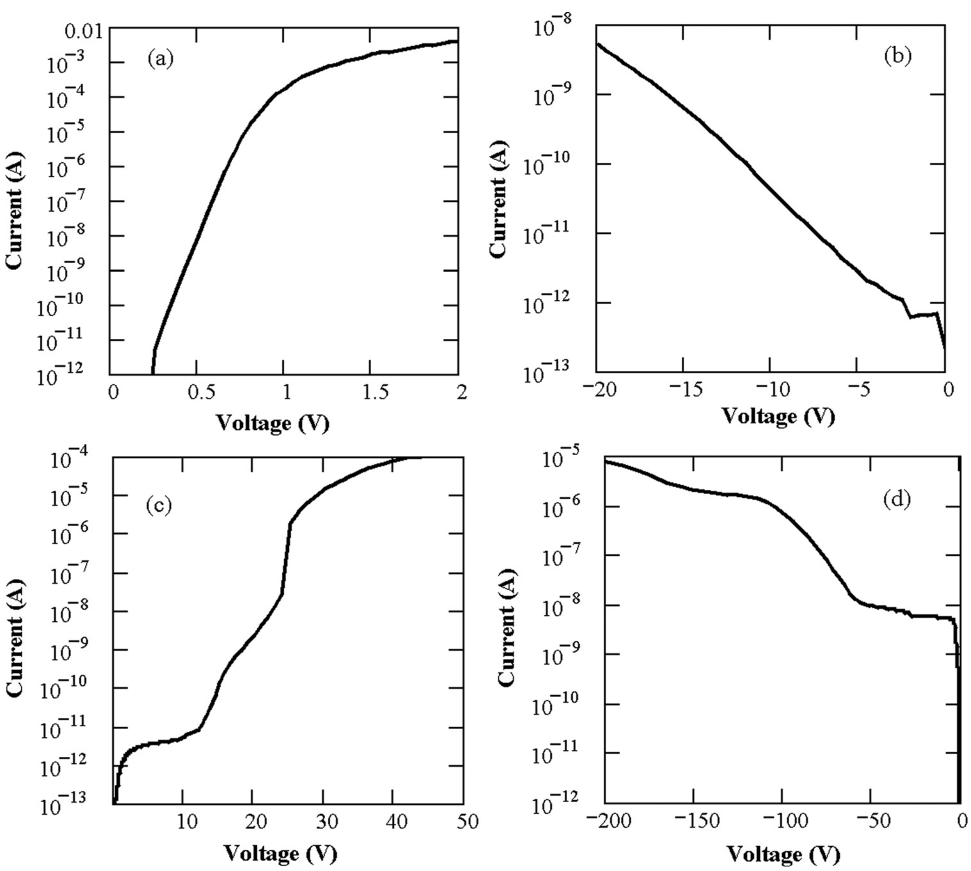

FIG. 2. Room temperature I-V characteristics obtained at (a) forward, and (b) reverse bias for the n-type SiC epitaxial layer, and (c) forward, and (d) reverse bias for the SI SiC epitaxial layer.

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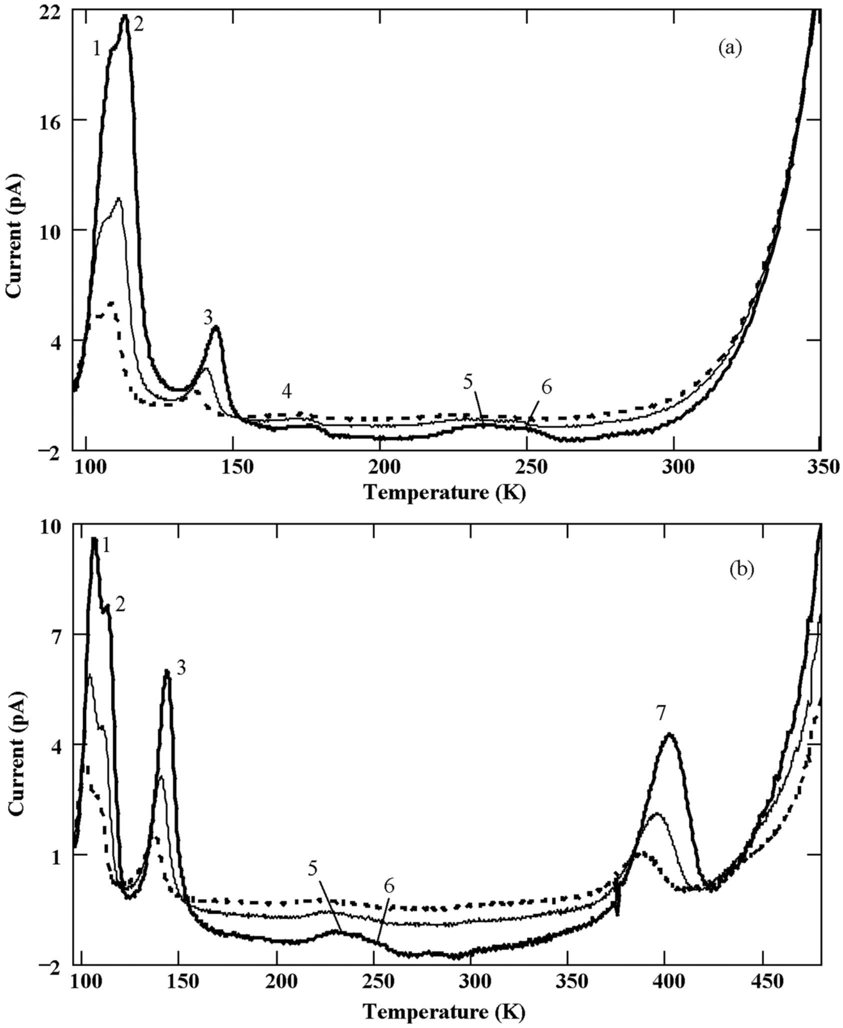

FIG. 3. TSC spectra of the n-type SiC epitaxial layer at (a) −5 V, and (b) 0 V bias voltages. Bold solid line: 15 K/min; thin solid line: 10 K/min; dotted line: 4 K/min.

T_m, at a given heat rate. Therefore, for a given trap center we have a set of three $T_{m,}$ which provided the Arrhenius plot for finding the trap activation energy from its slope. Using the activation energies, the temperature independent capture cross-sections were then found by fitting the experimental curves of the TSC spectra by the theoretical ones calculated by,

$$I_{TSC} = \sum_i I_i, \tag{1}$$

where I_{TSC} is the total TSC, i is the discrete energy level number, and I is the current produced by the individual discrete energy level, given by,[15]

$$I = I_0 \exp\left[-\frac{E_a}{kT} - \frac{kD_t}{\beta E_a} T^4 e^{-E_a/kT}\left(1 - 4\frac{kT}{E_a} + 20\left(\frac{kT}{E_a}\right)^2\right)\right], \tag{2}$$

where $I_0 = N_T e\mu A\tau E D_t T^2$, N_T is the density of the filled traps at the beginning of the temperature ramp, e is the

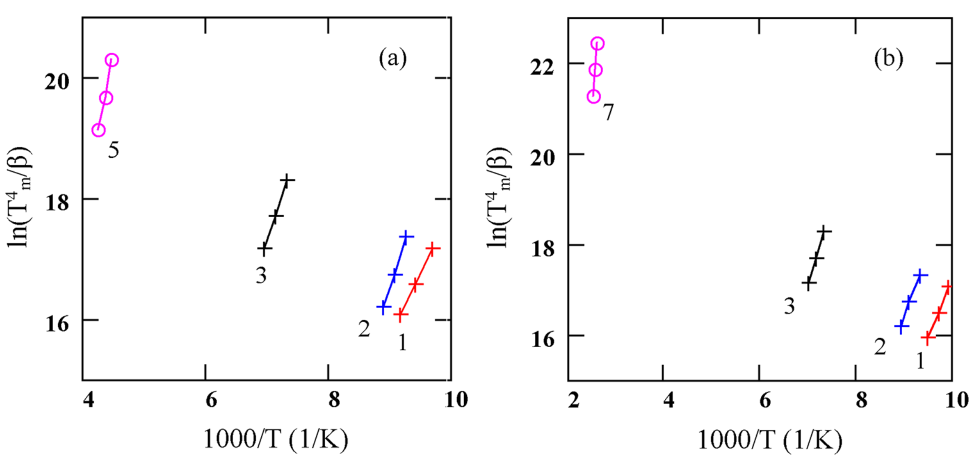

FIG. 4. (Color online) Arrhenius plots obtained from the TSC spectra of the n-type SiC epitaxial layer: (a) −5 V bias voltage, and (b) 0 V bias voltage.

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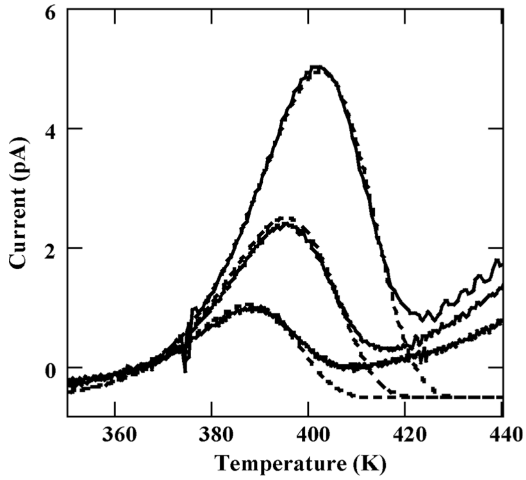

FIG. 5. Theoretical fit of peak 7 in Fig. 3(b) using 1.08 eV energy and 4×10^{-15} cm^2 capture cross-section. —: theoretically calculated curves; ---: experimental spectra. The theoretical and experimental curves are shifted to the same baseline at 350 K.

electronic charge, μ is the carrier mobility, A is the area of the electrode, τ is the free carrier lifetime, E is the electric field, k is Boltzmann's constant, T is the absolute temperature, $D_t = 3 \times 10^{21}\sigma(m^*/m_0)$, σ is the capture cross section, m_0 and m^* are the electron rest and effective mass, respectively, β is the heat rate, and E_a is the trap activation energy.

In order to minimize the leakage current which swamped the signal from the deep level centers at high temperatures, we conducted measurements at 0 V bias. During these measurements, the voltage source was excluded from the measurement set-up and calibration of the offset voltage of the electrometer was performed.[16] As a result, the voltage applied by the instrument to the sample was <30 μV. Note that similar measurements were successfully used to characterize deep levels in the SI GaAs samples[17] and the TSC was attributed to the thermoelectric effect (TEE) caused by a small temperature gradient between the front and back surfaces of the sample (~1 K across the ~500 μm thick sample).

III. RESULTS AND DISCUSSION

A. TSC measurements of n-type SiC epitaxial layer

Figure 3 shows the TSC spectra obtained for the device on the n-type SiC epitaxial layer at −5 and 0 V bias voltages. We have distinguished 7 peaks (1–7) in the spectra, as shown in Fig. 3. The intensity of peaks 1 and 2 increased with the bias voltages, which are associated with an increase of the width of the depletion region resulting in larger number of trap centers contributing to the TSC signal. Therefore, traps 1 and 2 behave as bulk traps. The Arrhenius plots for these traps are shown in Fig. 4, revealing activation energies of ~0.2 and ~0.25 eV for traps 1 and 2, respectively. These trap centers can be produced by the Al, B impurities, and L defect center, having close ionization energies of 0.23 eV (0.23–0.28 eV), and 0.24 eV above the valence band edge, respectively.[10,18] This correlation complies with the TSC studies of the SI 4H-SiC (Ref. 12) where the Al related trap with $E_a = 0.22$ eV was peaked at 105 K. The intensity of peak 3 was approximately the same at 0 V and at −5 V bias. This trap center has a peak temperature of ~140 K and had an activation energy close to that reported for shallow boron.[12] However, its independence on the bias voltage suggests that this peak represents the trap center at the Ni/SiC interface. The weak peak 4 at ~175 K can be caused by the HS1, D_I, and HH1 centers[12,19,20] with an activation energy of ~0.35 eV. The double peak at 220–250 K (features 5 and

TABLE I. Trap parameters in the n-type and the SI 4H-SiC epitaxial layers deduced from the TSC measurements.

	T_m (K)		Activation energy (eV)		Capture cross-section (cm^2)		
TSC peak number	0 V	5 V	0 V	5 V	0 V	5 V	Possible defects or impurities
1	101–106	103–109	0.22	0.19	2×10^{-16}	6×10^{-18}	Al[a]
2	107–112	108–113	0.25	0.26	6×10^{-16}	1×10^{-15}	Al,[a] B,[a] L-center[b]
3	136–143	137–144	0.31	0.26	1×10^{-16}	2×10^{-18}	Interface trap
4		172–176			…	…	D_I,[c] HS1,[c] HH1[d]
5	222–232	225–236	~0.5	~0.5	…	…	D-center[e]
6	…	240–250	…	…		…	
7	388–402	…	1.08	…	4×10^{-15}	…	IL_2,[f] SI-6,[g] SI-8[g]
A	105–112	…	0.19	…	$\sim 10^{-18}$	…	Al,[a] B,[a] L-center[b]
B	128–137	…	~0.2	…	…	…	Interface trap
C	236–247	…	0.57	…	$\sim 2 \times 10^{-16}$	…	D-center[e]
D	280–290	…	0.82–0.87	…	$\sim 10^{-13}$	…	*HK2*,[h] IL_1[f]
E	485–585	…	…	…	…	…	Intrinsic defects

[a]Reference 10.
[b]Reference 18.
[c]References 12 and 19.
[d]Reference 20.
[e]References 12, 18, and 21.
[f]Reference 22.
[g]Reference 23.
[h]Reference 21.

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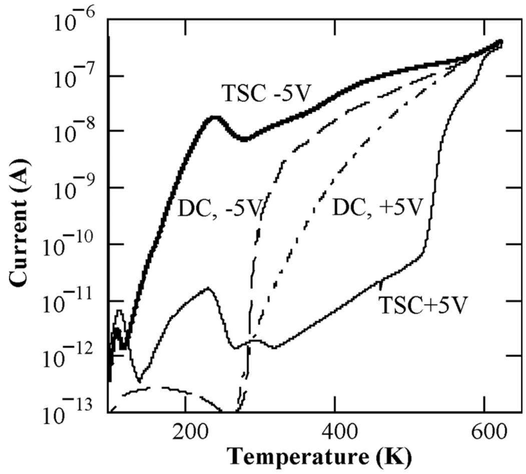

FIG. 6. TSC spectra and dark current (DC) of the SI SiC epitaxial layer at ±5 V bias voltages.

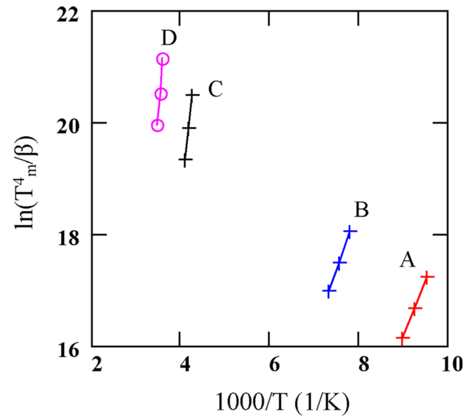

FIG. 8. (Color online) Arrhenius plots obtained from the TSC spectra of the SI SiC epitaxial layer at 0 V bias voltage.

6 in Fig. 3(a)) can be attributed to the well known boron related D-center.[12,18,21] The TSC measurements at 0 V bias revealed one more trap center peaked at ~400 K (peak 7 in Fig. 3(b)). This trap center had the activation energy of 1.08 eV and a capture cross-section $\sim 3 \times 10^{-15}\,\mathrm{cm}^2$. The theoretical fitting of this peak, shown in Fig. 5, indicates a very good representation of this trap by a discreet energy level with the aforementioned parameters. The candidates for this peak are electron trap, IL_2, that was found in the 4 H-SiC epitaxial layers grown at low C/Si (Ref. 22) and defects SI-6 and SI-8 detected by electron paramagnetic resonance in high purity SI 4H-SiC.[23] The 1.1 eV center was also found in our TSC studies of bulk SI 4 H-SiC and was related either to V-complexes or intrinsic defects. However, its peak temperature (456 K) was higher than for the peak 7. The trap parameters deduced from our TSC measurements of the n-type SiC epilayer are summarized in Table I.

B. TSC measurements of SI epitaxial layer

Figures 6 and 7 show the TSC spectra obtained on the SI SiC epitaxial layer at ±5 and 0 V bias voltages, respectively. The TSC spectra at ±5 V was affected by the leakage currents that strongly depend on the occupancy of deep level centers, as it is evident from the dark current plots, as shown in Fig. 6. Identification of the trap activation energy and the capture cross section was accomplished using the TSC spectra at 0 V, shown in Fig. 7. We have distinguished 5 peaks (A–E) in the TSC spectra in Fig. 7. The Arrhenius plots of the traps, A-D, are shown in Fig. 8. The summary of the trap parameters extracted from our TSC measurements of the SI epilayer is given in Table I. As follows from Table I and from a comparison of the TSC spectra of the n-type epitaxial layer and the SI epitaxial layer in Figs. 3(b) and 7, peaks A, B, and C are similar to peaks (1 and 2), 3, and 6 in the n-type epitaxial layer, respectively. Therefore, our correlation of these traps with defects and impurities is the same as for the n-type epitaxial layer (see Table I). Trap D peaked at temperatures typical of that of the $Z_{1/2}$ center,[10,18] a vacancy-type defect. However, its activation energy found in our experiments (~0.85 eV) is significantly higher than the 0.63 eV reported for the $Z_{1/2}$ center. Therefore, possible candidates for this peak could be the hole and electron traps, *HK2*,[21] and IL_1,[22] with reported energies ($E_V + 0.84$ eV) and ($E_C - 0.87$ eV), and

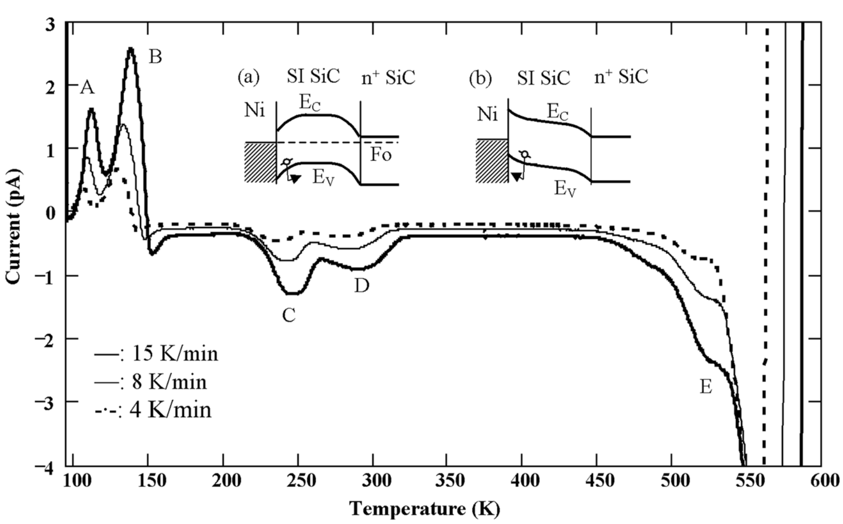

FIG. 7. TSC spectra of the SI SiC epitaxial layer at 0 V bias voltage.

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capture cross-sections 4×10^{-14} and $\sim 1 \times 10^{-14}$ cm^2, which are fairly close to that found from our TSC measurements. It is suggested that a complex involving two nitrogen atoms (N_C–N_i) or a split interstitial on the C site $(N\text{–}N)_C$ produce an IL_1 center, although V_C, Si_C, or Si_i related defects are also a possibility.[22] Further investigation is required to find the activation energy and capture cross section of the trap centers associated with feature E containing multiple peaks. Its highest intensity peak was swamped by a high stray current, associated with the thermoelectric effects. The high peaking temperature of E centers implies a high activation energy, possibly close to the middle of the bandgap. The trap centers with activation energies close to the middle of the bandgap were reported for the as-grown 4H-SiC epitaxial layers[21,22] and the high purity bulk SI 4H-SiC[11,23,24] and were attributed to intrinsic defects and their complexes.

The difference of the TSC spectra of the SI epitaxial layer from that of the n-type epitaxial layer is the appearance of negative peaks C, D, and E. For the SI epilayer the TSC at 0 V can be caused by the TEE similar to that reported by Huang *et al*. for the SI GaAs.[17] In this case, the current polarity depends on the type of carriers emitted by the traps and can be used to differentiate between the electron and hole traps. Assuming that during illumination donor traps are predominately filled with electrons and the acceptor traps are filled by holes, we should exclude the IL_1 acceptor-like center from consideration for trap D. However, the TEE approach does not account for the built-in electric field that is expected to be present in the SI SiC epilayer and may also affect the polarity of the TSC. Our alternative explanation of the negative peaks C, D, and E in the TSC spectra of the SI epilayer is depicted in insets (a) and (b) in Fig. 7. The schematic (inset (a)) shows a suggested band diagram of the studied structure in the thermodynamic equilibrium at low temperature. This energy diagram was obtained assuming no interface states and pinning of the Fermi level in the SI SiC close to the middle of the bandgap by the trap centers, E. We used $\sim$4.0 eV for the electron affinity of 4 H-SiC,[25] and $\sim$5.2 eV for the Ni work function.[26] During illumination of the structure at 94 K the electron-hole pairs are generated in the SI SiC epilayer by the incident light resulting in the photocurrent of about 0.3 μA. Holes propagating to the Ni electrode are trapped by acceptor-like centers, eliminating the negative charge initially present on acceptor deep levels in the vicinity of the Ni contact. Therefore, the band bending in the SI SiC will change to that shown in inset (b). As the temperature increases during the subsequent TSC measurement, the acceptor traps emit holes (peaks A and B) and become negatively charged. The band bending changes toward that shown in the inset (a) and the direction of the electric field in the region joining the Ni electrode also changes its direction, which led to the change in the direction of the TSC current. Also, note that the space charge region joining the n^+-SiC substrate may also contribute to the TSC with the opposite direction of the current, which also may result in the change of the current direction, if this current dominates the TSC current from the space charge region joining the Ni electrode.

IV. CONCLUSION

The deep level centers have been characterized in the CVD grown n-type and SI 4H-SiC epitaxial layers using TSC spectroscopy. Both epitaxial layers exhibited relatively shallow levels related to the Al, B, L- and D-centers. A deep level center with the activation energy 1.1 eV peaked at 400 K was detected in the n-type epitaxial layer and correlated with the IL_2 level and the 1.1 eV center in the high purity bulk SI 4H-SiC. The TSC spectra of the SI epitaxial layer was dominated by the peaks at 525–580 K that we attributed to intrinsic defects and their complexes with energy levels close to the middle of the bandgap. The TSC measurements conducted at 0 V bias voltage allowed us to detect deep level centers that were swamped by the high leakage current in the TSC spectra at a non-zero bias. The TSC spectra of the SI SiC epitaxial layer at 0 V bias exhibited peaks with different current polarity, which we attributed to TEE or to the built-in electric field reversal.

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