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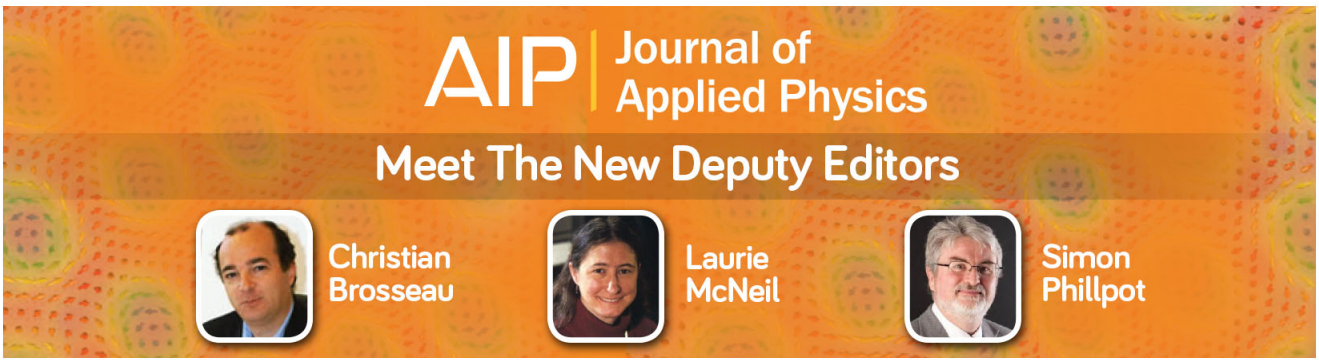

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Doping of *n*-type 6H–SiC and 4H–SiC with defects created with a proton beam

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Deep centers in *n*-type 4H–SiC and 6H–SiC irradiated with 8 MeV protons have been investigated by capacitance spectroscopy and electron paramagnetic resonance (EPR). Samples were fabricated by sublimation epitaxy or commercially produced by CREE Inc. Research Triangle Park, NC. It is showed that irradiation of wide-band gap semiconductors may lead to an increase in the concentration of uncompensated donors in an *n*-type material. The spectrum of deep centers in both SiC polytypes is independent of the technology of material growth or type of charged particles. However, the parameters and behavior of the radiation defects in 6H– and 4H–SiC are different. A conclusion about the possible nature of the irradiation induced centers is made on the basis of annealing behavior and EPR data. The obtained results show that proton irradiation can be used in SiC device fabrication technology for producing local high-resistance regions in the semiconductor.
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I. INTRODUCTION

It is known that proton irradiation can be used to passivate the periphery of semiconductor $p-n$ structures.[1] Previously, SiC layers semi-insulating at room temperature have been produced by means of proton irradiation.[2–4] However, the parameters and concentrations of radiation defects (RDs) formed during irradiation have not been studied. In other works,[5,6] RD parameters were studied but the compensation appearing in the course of irradiation was not analyzed.

The aim of this work was to fabricate semi-insulating 6H– and 4H–SiC layers by proton irradiation and to determine the parameters of RDs responsible for the compensation.[7]

II. SAMPLES

All samples used were SiC *pn* structures and epitaxial layers commercially produced by CREE Research, Inc.,[7] or fabricated at the Ioffe Institute by sublimation epitaxy.[8] The *n*-type layer thickness was about 5 μm; that of the *p*-type layer was about 1 μm, and the substrate thickness was about 400 μm. The diameter of Schottky barriers or mesa structures of the diodes was in the range 300–1200 μm. The concentration of uncompensated donors in the *n*-type layer (Nd–Na) was $(0.8-4)\times10^{16}\,\mathrm{cm}^{-3}$, that in the substrate $(3-5)\times10^{18}\,\mathrm{cm}^{-3}$, and the concentration of acceptors in the *p*-type layer was $\sim5\times10^{18}\,\mathrm{cm}^{-3}$. Thus, the doping level in the substrate and *p*-type emitter exceeded that in the *n*-type base layer by no less than 2 orders of magnitude. Since the rate of RD generation in the semiconductor is practically independent of its doping level,[9] we assumed that the resistance of forward-biased structures of this kind (R_b) was determined by the carrier concentration in the *n*-type layer (actually by its compensation by RDs). Deep centers were studied by electron paramagnetic resonance (EPR) spectroscopy on Lely substrates about 0.5 mm thick with initial nitrogen concentration of $2\times10^{17}\,\mathrm{cm}^{-3}$ (6H–SiC) or CREE substrates with Nd–Na of $\sim2\times10^{18}\,\mathrm{cm}^{-3}$ (4H–SiC).

Proton irradiation was performed in an MGTs-20 cyclotron. Protons having energy of 8 MeV were used, with irradiation dose D in the range $1\times10^{14}-2\times10^{16}\,\mathrm{cm}^{-2}$.

III. EXPERIMENTAL RESULTS

A. Capacitance–voltage ($C-V$) characteristics

The $C-V$ characteristics were measured on a standard $C-V$ setup with a parallel equivalent circuit and sinusoidal frequency of 10 kHz. A study of irradiated samples demonstrated a decline in the Nd–Na value measured at room temperature, with Nd–Na markedly increasing on heating a structure to 650 K. For 6H–SiC the Nd–Na value measured at 650 K was even higher than that in the initial structures prior to irradiation. With increasing irradiation dose, this difference became more pronounced [Figs. 1(a) and 1(b)].

Irradiation also led to an increase in R_b. With increasing irradiation dose, the growth of R_b made capacitance measurements impossible—the measured capacitance ceased to depend on applied voltage.[10] On heating, the R_b value decreased exponentially with activation energy ε_A [Figs. 2(a)

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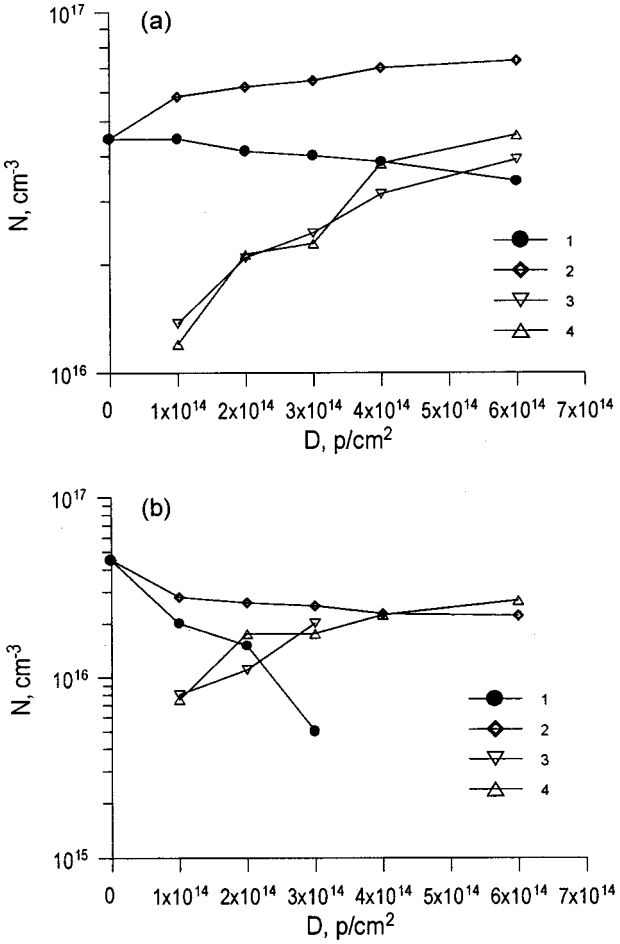

FIG. 1. (A) 6H–SiC, Nd–Na at $T=300$ K (1) and $T=650$ K (2), difference of these (3), and concentration of the center located at $E_c=1.1-1.22$ eV (4) vs the irradiation dose. (B) 4H–SiC, Nd–Na at $T=300$ K (1) and $T=650$ K (2), difference of these (3), and total concentration of the centers (RD1/2+RD3+RD4) (4) vs the irradiation dose.

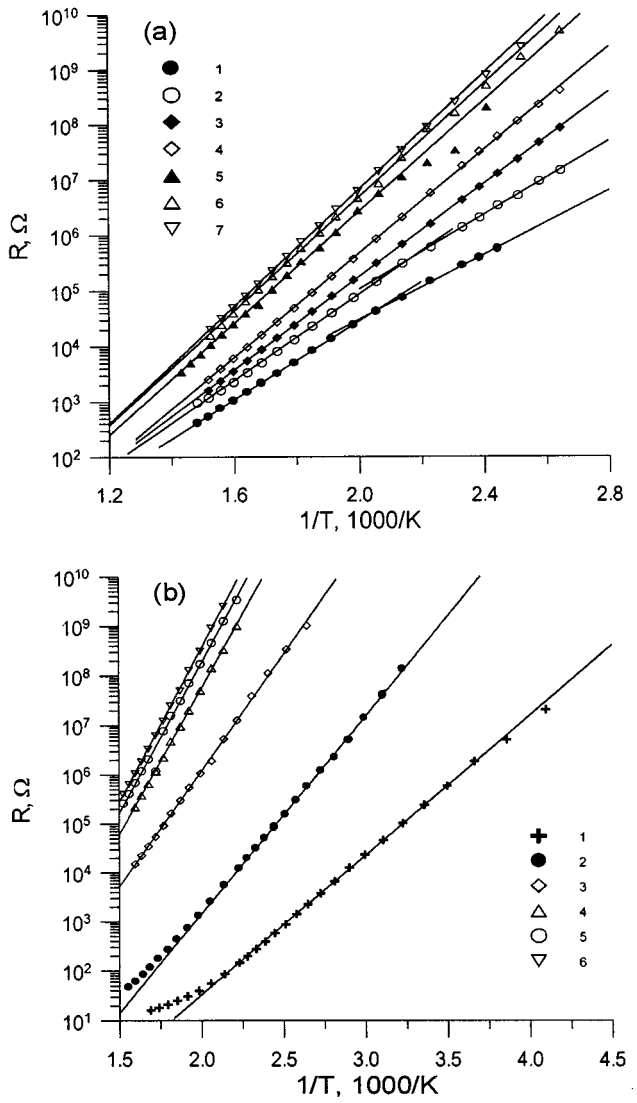

FIG. 2. (A) 6H–SiC; Temperature dependences of the forward resistance of a Schottky diode at different irradiation doses, 10^{14} cm^{-2}: 1–3; 2–4; 3–6; 4–10; 5–20; 6–50; 7–100. (B) 4H–SiC; Temperature dependences of the forward resistance of a Schottky diode at different irradiation doses, 10^{14} cm^{-2}: 1–6; 2–10; 3–20; 4–50; 5–100; 6–200.

and 2(b)]. With increasing irradiation dose, the ε_A value became higher, asymptotically approaching the values of ~1.1 eV for 6H–SiC and 1.25 eV for 4H–SiC [Figs. 3(a) and 3(b)]. Such behavior of the resistance under irradiation (Fermi level ''pinning'') is also characteristic of other semiconducting materials (see, e.g., Ref. 11 for the case of GaAs). That is, with increasing irradiation dose, the Fermi level can be pinned near the radiation defect whose introduction rate into the material is the highest.

B. DLTS measurements

In studying deep centers in the upper half of the forbidden gap in *n*-type 6H–SiC, six kinds of deep centers were found. Most of these were observed in electron-irradiated samples[12] or had parameters close to intrinsic structural defects. Two of the observed centers ($E_c=0.16-0.2$ eV and $E_c=0.5$ eV) were completely annealed out at 500–650 K. In some samples, the concentration of the center located at $E_c=0.7$ eV increased upon annealing at 800 K.

Investigation of the deep centers in the upper half of the forbidden gap in *n*-type 4H–SiC revealed five deep centers. Most of these were found in samples implanted with He^+ ions[5] or had parameters close to those of intrinsic structural defects. One of the observed centers ($E_c=0.18$ eV) was completely eliminated by annealing at 500–650 K.

For both of the polytypes studied, no pronounced difference was observed between the spectrum of deep centers formed in CREE epitaxial layers and those grown by sublimation epitaxy.

Tables I and II list ionization energies of the observed centers (E_c-E_0), their electron capture cross sections σ_n, and concentrations upon irradiation with a dose of 2×10^{14} cm^{-2}. Figures 4(a) and 4(b) present Arrhenius dependences for the revealed RDs. The introduced RDs were completely annealed out at ~1800–2100 K.

C. EPR studies of deep centers created by proton irradiation

Bulk samples were irradiated under the same experimental conditions: irradiation dose 4×10^{15} cm^{-2} (6H) and 2

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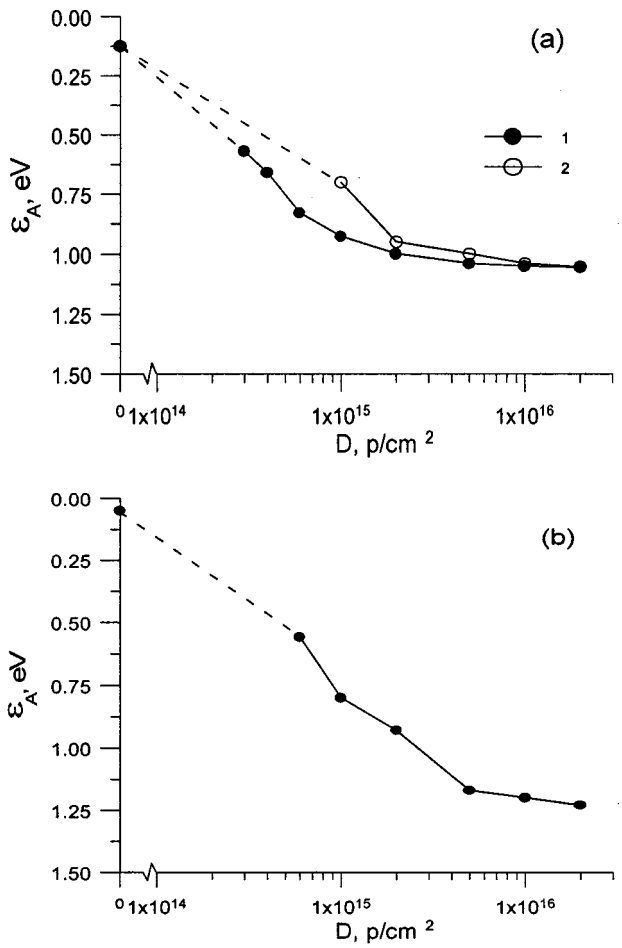

FIG. 3. (A) 6H–SiC, ε_A vs irradiation dose for (1) CREE *pn* structure and (2) Schottky diode based on an epitaxial layer grown by sublimation epitaxy. (B) 4H–SiC, ε_A vs irradiation dose for a Schottky diode based on an epitaxial layer grown by sublimation epitaxy.

$\times 10^{16}\,\mathrm{cm}^{-2}$ (4H). The measurements were done on a Varian E-112 EPR spectrometer at 77 K. As a result of irradiation, the EPR spectra of the polytypes in question changed significantly. Irradiation gave rise to additional RD-related lines, whereas prior to irradiation, only signals from electrons on impurity nitrogen atoms were observed.

Complete spectra of irradiated samples are presented in Fig. 5: spectrum 1 for 4H–SiC and spectrum 2 for 6H–SiC. The positions of lines correspond to the angle between the *c* axis and the magnetic field direction at which the spectrum splitting is the strongest (about 35°). In region ''a,'' denoted in the figure by vertical lines, the amplification was lowered by an order of magnitude for the 6H polytype and 2 orders of magnitude for the 4H polytype in order to demonstrate the presence of an EPR signal from electrons on impurity nitrogen atoms. The presence of these resonances indicates that at the irradiation dose used in this work the Fermi level remains close to the nitrogen level, with all lower lying RD-related levels filled with electrons. However, comparison of the intensities of EPR spectra of nitrogen in irradiated and unirradiated samples demonstrates that upon irradiation with the chosen dose the electron concentration on nitrogen centers decreases by a factor of 1.8 for the 6H and 2.6 for the 4H polytype, i.e., the samples become rather strongly compensated.

It should be noted that, in contrast to the 6H polytype which has such high resistance at 77 K, no changes are introduced into the cavity *Q* factor, where the unirradiated 4H polytype shows pronounced conductivity leading to the Dyson's shape line and impairing the *Q* factor of the cavity of the EPR spectrometer. As a result of irradiation, this line narrows and becomes more symmetrical, and weak additional peaks appear on its background. Part of these peaks can be related to the emergence of an EPR signal from isolated nitrogen atoms and part to the appearance of new resonances associated with RDs. However, because of the considerable difference between the concentrations of nitrogen centers and RDs there is no way of tracing the behavior of RD-related spectra in the region where nitrogen resonances are observed.

Figure 6 shows on a larger scale single lines from RD-related spectra (1, 4H polytype; 2, 6H polytype), which allow more substantiated conclusions about the defect structure in different SiC polytypes. The lower and upper scales correspond to curves 1 and 2, respectively. The figure clearly demonstrates weak satellite hyperfine lines, with two pairs of lines observed for the 6H polytype: two stronger, lying 14

TABLE I. Parameters and properties of RDs observed in 6H–SiC.

Parameters of observed RDs				Correspondence to published data		
$E_c - E_0$ (eV)	$\sigma(n)$ (cm^2)	N_g (cm^{-3})	T_{ann} (K)	Electron irradiation[a]	Structural defects	Possible structure
0,16–0,2	6×10^{-17}	3×10^{14}	800–950	L_1		Primary defects[d]
0.36/0.4	2×10^{-15}	3.3×10^{15}	1100<..<1800	L_3L_4	$E1/E2$,[b] S[c]	
0.5′	5×10^{-15}	2.2×10^{15}	800–950	L_6		V_C[a,d]
0.7	4×10^{-15}	1.3×10^{15}	1100<..<1800	L_7/L_8	Z_1/Z_2[b]	V_C+V_{Si}[b,d]
0.8	4×10^{-15}	6×10^{14}	1100<..<1800	L_9		
1.1–1.22	2×10^{-15}	2×10^{16}	1100<..<1800	L_{10}	R[c]	V_C+V_{Si}[d,e]

[a]See Ref. 12.
[b]See Ref. 5.
[c]See Ref. 13.
[d]Present work.
[e]See Ref. 15.

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TABLE II. Parameters and properties of RDs observed in 4H–SiC.

Parameters of observed RDs				Correspondence to published data		
E_c-E_0 (eV)	σ_n (cm^2)	N_g (cm^{-3})	T_{ann} (K)	Implantation of He^{+}[a]	Structural defects	Possible structure
0.18	6×10^{-15}	2×10^{14}	800–950	*P*1/*P*2		Primary defects[d]
0.63–0.7	5×10^{-15}	5×10^{15}	1100<..<1800	*Z*1	*Z*1[a,b]	V_C[d]
0.96	5×10^{-15}	6.3×10^{15}	1100<..<1800	RD1/2		$\{V_C+V_{Si}$[d]
1.0	1×10^{-16}	6.3×10^{15}	1100<..<1800	RD3	$E_c-1.1$ eV[c]	
1.5	2×10^{-13}	5×10^{15}	1100<..<1800	RD4		

[a]See Ref. 5.
[b]See Ref. 16.
[c]See Ref. 17.
[d]Present work.

Oe apart and two weaker, spaced by 4 Oe. For the 4H polytype the single line is broader than that for the 6H case. Also broader are the satellite lines lying 18 Oe apart. No weaker satellites are observed in 4H.

IV. DISCUSSION

A. Compensation

As shown by the investigation performed, proton irradiation creates a defect with the highest introduction rate in *n*-type 6H–SiC ($E_c=1.22$ eV) with parameters close to those of the known structural defect—the *R* center.[13] As follows from the parameters of the center located at $E_c=1.1-1.22$ eV, the recharging time τ_R for this center is about 2 weeks at 300 K. Thus, the charge state of this and deeper lying centers does not change during $C-V$ measurements at room temperature. At the same time, the τ_R value for the center which is the closest to this level, ($E_c=0.8$ eV), is 3.3 s. Consequently, the center $E_c=0.8$ eV (and all shallower centers) can be considered completely depleted in $C-V$ measurements at room temperature.

In *n*-type 4H–SiC, several deep centers are formed in the energy range 0.96–1.5 eV. The time constant of charge exchange for the RD1/2 center is 5×10^3 s at 300 K. Similarly, in the case of 6H–SiC, the charge state of this and deeper lying centers remains unchanged in the course of $C-V$ measurements at room temperature. At the same time, the time τ for the *Z*1 center, the closest to the above level is 36×10^{-3} s at 300 K. Consequently, the *Z*1 center (and all shallower centers) in 4H–SiC can be considered completely depleted at room temperature.

At higher irradiation doses, the sample conductivity is determined by thermal excitation of electrons from the RDs, and the activation energy of resistivity approaches the ionization energy of the deepest center. The available experimental data do not allow any unambiguous conclusion about whether the observed RDs are donors or acceptors (see Ref. 14 for more details). However, irrespective of the RD nature, the maximum activation energy of the base resistance will

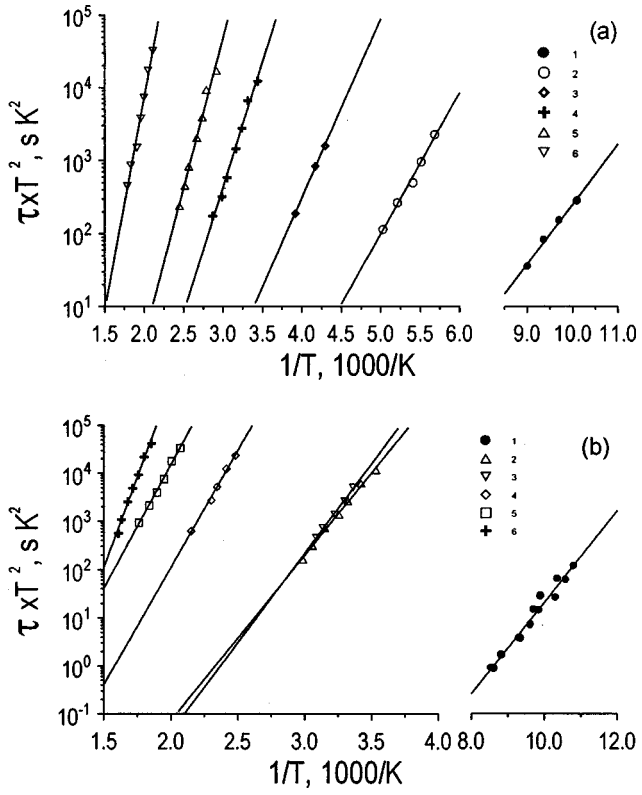

FIG. 4. (A) 6H–SiC, Arrhenius dependences for the observed RDs: (1) $E_c=0{,}2$ eV; (2) *E*1/*E*2; (3) $E_c=0{,}5$ eV; (4) $E_c=0{,}7$ eV; (5) $E_c=0{,}8$ eV; (6) $E_c=1{,}1$ eV. (B) 4H–SiC, Arrhenius dependences for the observed RDs: (1) *P*1/*P*2, (2) *Z*1 after irradiation, (3) *Z*1 before irradiation, (4) RD1/2, (5) RD3, and (6) RD4.

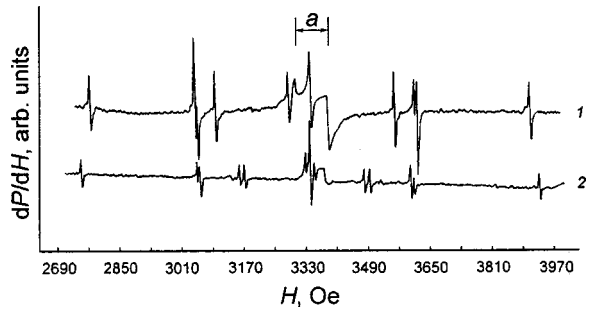

FIG. 5. EPR spectra of irradiated 4H–SiC (spectrum 1) and 6H–SiC samples (spectrum 2) at the maximum distance between outermost lines. In region ''a'' the spectrometer amplification was lowered by 2 orders of magnitude for 4H–SiC and an order of magnitude for 6H–SiC.

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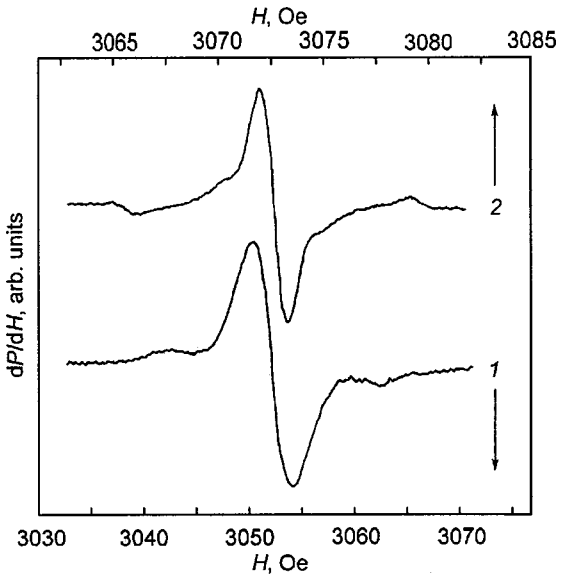

FIG. 6. A single line from an EPR spectrum for (1) 4H and (2) 6H polytypes. The corresponding magnetic field scales are shown by arrows.

tend to the ionization energy of the deepest of the RD centers, ~1.25 eV (6H) and 1.5 eV (4H), with increasing D.

Thus, the difference between the Nd–Na values measured at 300 and 650 K must be equal to the concentration of the center located at $E_c = 1.1-1.22$ eV for 6H–SiC and that of the centers RD1/2+RD3+RD4 for 4H–SiC, determined from DLTS spectra. As seen from Figs. 1(a) and 1(b), this equality agrees well with experiment for doses $\leqslant 3 \times 10^{14}\,\mathrm{cm}^{-2}$. At higher irradiation doses, room-temperature $C-V$ measurements were impossible because of the high R_b value. On heating, the resistance of irradiated structures must decrease exponentially which, indeed, has been observed experimentally.[2]

B. Identification and possible structure of centers

As seen from Tables I and II, there is good agreement between the spectra of the RDs appearing in SiC upon proton and electron irradiation (6H) or proton irradiation and implantation with He^+ ions (4H). Also, a correlation exists between the annealing temperatures of the introduced centers.

Thus, it can be concluded that in SiC, as in other semiconducting materials, different kinds of irradiation cause formation of the same RDs, but with different concentration ratios.

Despite the coincidence of the RD parameters with the parameters of some native defects [R, $E1/E2$, $Z1/Z2$ (6H); $Z1$, $E_c = 1.1$ eV (4H)], a conclusion that the given RDs and native defects are completely identical seems, in our opinion, preliminary. We recall that RDs are completely annealed out at ~1500 K, whereas native defects with nearly the same parameters exist in sublimation-grown SiC layers up to temperatures as high as 2100 K.

As mentioned in Ref. 6, where the mechanism of RD formation and annealing in SiC was studied in experiments with positron lifetime, proton irradiation leads to formation of carbon and silicon vacancies and other primary defects, which recombine on annealing at 400–600 K to give thermally stable complexes. Thus, our DLTS investigations before and after annealing suggest that the centers located at $E_c = (0.16-0.2)$ eV and $E_c = 0.5$ eV(6H) and $E_c = 0.18$ eV(4H) are related to primary RDs, and that at $E_c = 0.7$ eV(6H) is, similarly to the D center, a vacancy complex.

The RD structure is well determined on the basis of EPR data; however, to relate the structure and electrical properties, additional conditions are necessary. In particular, this becomes possible when defects are photosensitive.

All attempts to relate the structure of RDs generated in SiC upon irradiation to their electrical characteristics in the general case have failed. For some structural defects appearing in SiC upon sample quenching this could be done owing to the photosensitivity of their EPR spectra.[15] A study of the effect of light on the intensity of EPR spectra in our samples revealed their insensitivity to light. In order to identify the structure and electrical properties of the RDs, we compared the parameters of the EPR spectra taken in this work with those obtained in studying thermal defects in quenched 6H–SiC samples[15] and radiation defects in 4H–SiC samples irradiated with protons and α-particles.[18] As shown in these works, defects in SiC give rise to two kinds of EPR spectra: with $S = 1/2$ and $S = 1$ spins. The latter show fine structure. The EPR parameters of centers in both polytypes are close, which suggests the occurrence of a similar correlation of the electrical properties.

In our case, irradiation also generates paramagnetic centers of two kinds in both polytypes. The center with spin $S = 1/2$ corresponds to a weakly anisotropic line with the g factor nearly coinciding with that of nitrogen. Since the spectrum of nitrogen atoms in the samples studied does not disappear, but remains rather intense, weak hyperfine lines, possibly present in the spectrum and associated with the interaction of the electron spin with nuclear spins of the isotopes ^{29}Si and ^{13}C, are indistinguishable against its background. In this connection, it has been impossible to identify unambiguously the structure of this center. However, comparison with the spectrum of thermal defects suggests that this line is related to an isolated vacancy on the carbon sublattice, with thermal activation energy of 0.5 eV for the 6H polytype and 0.63–0.7 eV for 4H. (The optical activation energy measured for the 6H polytype in Ref. 15 was 0.65 eV).

Much more precise identification could be done for the center with spin $S = 1$. Taken in arbitrary orientation, the spectrum contains 12 lines for both the polytypes. Since these spectra are shown in Fig. 5 at the strongest fine splitting for one of the directions, the positions of some other lines coincide. This means that the angle between the magnetic field and the principal axis of fine splitting for other symmetrical positions of this RD is about the same and is determined by the position of principal axes in the c plane. The symmetry axis of such a center lies in the plane containing the c axis and one of twofold axes. The strongest observed fine splitting constant $D = 580$ Oe ($520 \times 10^{-4}\,\mathrm{cm}^{-1}$) for the 6H polytype and $D = 567$ Oe ($508 \times 10^{-4}\,\mathrm{cm}^{-1}$) for

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the 4H polytype. These values are observed with the field at about 30° to the *c* axis.

Comparison of the spectrum parameters with the known parameters of RDs in Ref. 18 and thermal defects in Ref. 15 suggests that similar spectra are observed for the RD $G2$ (in 4H– and 6H–SiC) and the photosensitive thermal defect $P7$ (in 6H–SiC). The latter has optical activation energy of 1.17 eV. On this basis, the RD spectrum with $S=1$ can be identified with high probability as a center incorporating carbon and silicon vacancies (V_C+V_{Si}) on adjacent lattice sites, having an activation energy of 1–1.2 eV.

Analysis of a single fine line in Fig. 6 allows us to reveal some additional characteristics of these centers. It can be seen from Fig. 6 that two pairs of satellite lines with different intensities are observed in the 6H polytype. Stronger lines, spaced by $A_1=14$ Oe, have an intensity constituting about 5% of that of the main line. A pair of weaker lines whose intensity is difficult to measure lie on wings of the main line, spaced by $A_2=4$ Oe. The stronger pair is due to a hyperfine interaction of the spin with nearest neighbors of ^{29}Si nuclei, since the intensity ratio of the satellite and main lines corresponds to the natural abundance of the ^{29}Si isotope (4.7%). Its nuclear spin $S_{Si}=1/2$, with gyromagnetic ratio $\gamma=-0.53\times10^{-4}\,\mathrm{rad\,G^{-1}}$. The second pair should presumably be attributed to the interaction with the nearest neighbors of ^{13}C with natural abundance of 1.1% ($S_C=1/2$; $\gamma=0.67\times10^{-4}\,\mathrm{rad\,G^{-1}}$). It is well known that the hyperfine splitting (HS) is proportional to the probability of finding a spin at a certain point of the nucleus (squared wave-function density) and the gyromagnetic ratio. With knowledge of this, the probability ratio of finding an electron on silicon and carbon nuclei can be evaluated to as

$$\psi_{Si}^2\psi_C^2=A_{Si}\gamma_C/A_C\gamma_{Si}=4.42.$$

Taking into account that silicon atoms surround a carbon vacancy and carbon atoms surround a silicon vacancy, we can conclude that even though the spin is spread between the carbon and silicon vacancies, it spends nearly 4.5 times longer time on the carbon vacancy, as compared with the silicon vacancy.

As for the 4H polytype, the HS on the nearest C atoms cannot be determined because of the much broader linewidth. However, it can be considered to be not very different from that for the 6H polytype, and the spin also mainly resides on the carbon vacancy. Mention should also be made of the fact that the HS is stronger in the 4H polytype as compared with the 6H. This means that the probability of finding an electron on a carbon vacancy in 4H–SiC exceeds that for the 6H polytype. This coincides with a similar phenomenon for impurity nitrogen when the HS is 11 and ~20 Oe for 6H– and 4H–SiC, respectively. One more difference between the 6H and 4H polytypes is that the EPR line is broader in the latter. Since at the experimental temperatures and electron concentrations the EPR line in SiC is Gaussian and its width is determined by inhomogeneous broadening, the broader linewidth can be attributed to a higher probability of finding an electron on remote atoms in the 4H polytype, as compared with the 6H.

Thus, we could compare, on the basis of EPR data for two proton-irradiated SiC polytypes, the structure and electrical properties of two deep centers. These are the carbon vacancy with activation energies of 0.5 eV (6H–SiC) and 0.6–0.7 eV (4H–SiC) and a pair of vacancies in carbon and silicon sublattices with activation energies of 1.1 eV (6H–SiC) and 1.0 eV (4H–SiC). The different energies of thermal ionization of centers with supposedly the same structure (V_C+V_{Si}) can presumably be explained by different distances between pair components (vacancies) characteristic of each of these RDs.

The conclusions made about the structure of the observed RDs are summarized in Tables I and II. Even though the number and properties of the RDs appearing in SiC are not completely identical, three RDs can be distinguished, whose ionization energies and supposed structure are close in both the polytypes. These are $L1$, $L7/L8$, $L10$ (6H) and $P1/P2$, $Z1$, and RD3 (4H), respectively.

V. SUMMARY

Comparison of the properties of 8 MeV proton-irradiated 6H– and 4H–SiC suggests the following.

The spectrum of RDs introduced into each polytype of silicon carbide is practically independent of the material growth technology and charge particles used for irradiation (protons, α particles, and electrons).

Irradiation generates deep acceptor centers in both polytypes to which electrons from shallower donor levels pass. This shifts the Fermi level downward to give *n*-type 6H– and 4H–SiC layers semi-insulating at room temperature.

The number and properties of RDs appearing in two different SiC polytypes under irradiation are not completely identical.

The Nd–Na concentration increases in irradiated 6H–SiC samples. In contrast, the total concentration of uncompensated donors in proton-irradiated 4H–SiC decreases. This indicates that acceptor centers are formed in 4H–SiC in the lower half of the forbidden gap or donor centers are destroyed in its upper half. At the same time, the concentration of donor RDs formed in 6H–SiC exceeds that of the acceptor centers.

The obtained results may be applicable in SiC device fabrication technology for creating local high-resistance regions in the semiconductor. This result is particularly important for devices not intended to operate at high temperature: e.g., photodetectors or various radiation detectors.

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