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Current-voltage characteristics and DLTS spectra of high voltage SiC Schottky diodes irradiated with electrons at high temperatures

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A R T I C L E I N F O

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A B S T R A C T

The effect of the electron irradiation at high temperatures T_i (300 and 500 °C) and of the subsequent annealing on the current–voltage characteristics and DLTS spectra of high-voltage integrated 4H-SiC Schottky diodes is compared for the first time. The optimal annealing modes of the structures irradiated with 0.9 MeV electrons are determined. It is shown that the irradiation with a relatively low fluence $\Phi = 10^{16}$ cm^{-2} at 300 °C leads to an increase in the base resistance R_b by approximately an order of magnitude, and the initial value of R_b can be completely restored by a single annealing in a nitrogen atmosphere at a temperature of 250 °C for 60 min. At the same time, however, annealing affects only slightly the DLTS spectra measured after the irradiation. With increasingΦ, the longer annealing is required to fully restore the R_b. However, with the fluence increasing to $\Phi \geq 5 \times 10^{16}$ cm^{-2}, the effect of a "reverse annealing" was observed in SiC for the first time: the resistance of the base grows as a result of the annealing. Irradiation at temperature of 500 °C leads at $\Phi \geq 5 \times 10^{16}$ cm^{-2} to an even more pronounced effect of reverse annealing. It is demonstrated that the presence of DLTS-detected acceptor centers arising as a result of the "hot" irradiation in the upper half of the band gap can explain neither the change in the current–voltage characteristics nor the results of annealing.

1. Introduction

High-voltage 4H-SiC Schottky diodes have found wide application in such areas as high-temperature and radiation-hard electronics, including applications in space and automotive electronics, solar converter switches, nuclear-power-plant equipment, computer power devices [1–6].

The radiation hardness of devices is an important criterion for determining the possibility of their practical application. The resistance of devices to various types of irradiation is of particular importance in such areas as equipment for nuclear reactors, aviation and space electronics, and charged particle accelerators. Studies of the radiation resistance of devices to various types of radiation, in addition to their practical importance, make it possible to analyze the physical processes of the defect formation, as well as the response of the already formed defects to various types of annealing. The effect of the room temperature electron irradiation and the subsequent annealing on the parameters of high-voltage 4H-SiC Schottky diodes has been studied extensively [7–11]. Experiments show that the electron irradiation affects only slightly the parameters of the current–voltage (*I-V*) characteristics of diodes at small forward biases, in the region of their exponential part. The parameter that is the most sensitive to the dose and energy of radiation is the base resistance of a diode R_b [12]. Conduction electrons are captured by acceptor centers created by the electron irradiation, which leads to monotonic growth in R_b with increasing dose (fluence). In this connection, the study of DLTS spectra formed as a result of irradiation plays an important role in the interpretation of results [13–16].

The effect of electron irradiation performed at elevated temperatures ("hot irradiation") on the parameters of 4H-SiC Schottky diodes was done by us in Refs. [12,17]. It was shown that the radiation resistance grows monotonically with increasing irradiation temperature T_i (similar results had been obtained earlier under irradiation for A_3B_5 materials [18]).

In the present work the change of parameters of high voltage Schottky diodes under the influence of high-temperature electron

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irradiation and subsequent annealing is compared with the results of DLTS spectra measurement for the first time.

2. Materials and methods

The effect of the high temperature irradiation on high-voltage (blocking voltage 1700 V) integrated Junction Barrier Schottky (JBS) diodes CPW3-1700-S010B-WP was studied. The diodes were irradiated with 0.9 MeV electrons in a pulsed mode. The pulse repetition rate was 490 Hz, and the pulse duration 330 μs. The mean free path of 0.9 MeV electrons in SiC is approximately 1 mm. With a diode base thickness $L \approx$ 20 μm, the defects formed by the irradiation are introduced uniformly over the sample volume. The high-temperature (hot) irradiation was carried out in a purpose designed target chamber, which made it possible to carry out irradiation in air in the temperature range from room temperature to 600 °C. During the irradiation, the accuracy of temperature maintaining was ± 5 °C. The heating rate was 0.5 deg/s. The cooling rate was ~0.25 deg/s. Electrons were extracted from the evacuated volume of the accelerator through a 50-μm titanium foil.

The isothermal *I-V* characteristics of the diodes were measured at room temperature in the single-pulse mode with a base resistance $R_b \leq 10^3$ Ohm. At $R_b \geq 10^3$ Ohm measurements were carried out with a direct current.

The parameters of the formed radiation defects were determined by the DLTS method. The measurements were carried out both in the initial samples and after each irradiation and/or annealing.

To avoid spontaneous annealing, the upper temperature limit for DLTS spectra measurements was limited to $T = 400$ K [13]. On control samples, the DLTS spectra were studied up to a temperature of 650 K.

The basic annealing mode was experimentally chosen such that, after the irradiation with a fluence leading to an increase in the base resistance R_b by approximately an order of magnitude, the initial value of R_b could be almost completely restored by a single annealing. The mode consisted of annealing the samples in a dry nitrogen atmosphere at a temperature of 250 °C for 60 min.

3. Results and discussion

Fig. 1 shows the forward *I-V* characteristics of an unirradiated diode ($\Phi = 0$), a diode after the irradiation with a fluence $\Phi = 1 \times 10^{16}$ cm^{-2} at a temperature T_i = 300 °C, and that after the optimized subsequent annealing. The inset in Fig. 1 shows the results of the corresponding DLTS measurements.

As seen from Fig. 1, the irradiation in the indicated mode leads to a significant increase in the differential base resistance R_b from initial $R_b \approx 0.1$ Ohm to $R_b \approx 1.2$ Ohm. If the mobility of electrons in the initial and irradiated diodes is considered to be the same [19], it can be concluded that the irradiation leads to a decrease in the concentration of electrons in the base from the initial value $n_0 \approx 3.4 \times 10^{15}$ cm^{-3} [16,20] to $n \approx 2.8 \times 10^{14}$ cm^{-3}. It would be reasonable to assume that acceptor levels with a total concentration $(n_0 - n) \approx 3.1 \times 10^{15}$ cm^{-3} are formed as a result of the irradiation.

The inset in Fig. 1 shows the DLTS spectra of the levels in the upper half of the forbidden gap. Curve 2′ in the inset corresponds to the DLTS spectrum of the irradiated sample. For three acceptor levels, E0/E1, Z1/Z2, and E3 [16], the values measured for the concentrations, N_t are 1.1×10^{13} cm^{-3}, 9×10^{13} cm^{-3}, and $\sim 1.3 \times 10^{13}$ cm^{-3}, respectively. Measurements of DLTS spectra carried out on the control samples heated to a temperature of 650 K reveal in the irradiated samples the appearance of the well-known EH6/7 level [15,16,21]. The concentration measured for this level nearly coincides with N_t for the Z1/Z2 level, as it has been repeatedly observed earlier [15,21]. As noted in [15], the concentrations of the Z1/Z2 and EH6/7 levels are very similar in all types of samples: immediately after the *n*-type 4H-SiC epitaxial films are grown, after the irradiation, and after the annealing. It is assumed that both levels correspond just to the same defect, namely a carbon vacancy.

Thus, the total concentration of all levels recorded by DLTS in the upper half of the energy gap,$N^D_{t\Sigma}$, is $\sim 2 \times 10^{14}$ cm^{-3}, an order of magnitude less than the value of $N^R_{t\Sigma}$ estimated from the change in R_b.

The current–voltage characteristic of the sample after the annealing (curve 3) coincides with good accuracy with the *I-V* characteristic of an unirradiated diode (curve 1).

Curve 2 in Fig. 2 is a current–voltage characteristic of a diode irradiated with a fluence $\Phi = 4.5 \times 10^{16}$ cm^{-2} ($T_i = 300$ °C).

The resistance base, R_b for the *I-V* characteristic represented by curve 2 is ~48 Ohm. An increase in fluence Φ by a factor of 4.5, from $\Phi = 1 \times 10^{16}$ to 4.5×10^{16} cm^{-2}, leads to a rise in R_b by a factor of ~40 (compare with curve 2 in Fig. 1). The amplitude of the peak corresponding to the Z1/Z2 level (curve 2′ in the inset) also noticeably increases. The value measured for the concentration N_t of the Z1/Z2 level in this case is $\sim 3.3 \times 10^{14}$ cm^3, i.e. ~3.7 times that for the dose $\Phi = 1 \times 10^{16}$ cm^{-2} (Fig. 1, curve 2′). The concentrations N_t of the levels E0/E1 and E3 increased in

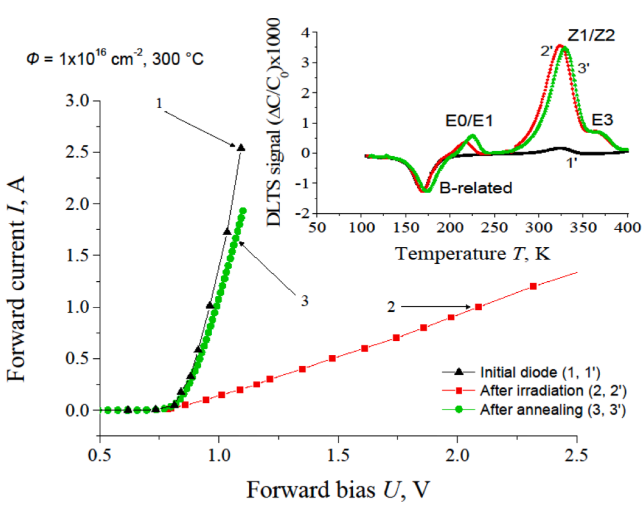

Fig. 1. Forward current–voltage characteristics of the initial diode (curve1), the diode after the irradiation with electrons at a temperature of 300 °C with a fluence $\Phi = 1 \times 10^{16}$ cm^{-2} (curve 2), and that after the subsequent annealing in the basic annealing mode (1 hr at a temperature of 250 °C in the dry nitrogen atmosphere, curve 3). The inset shows the DLTS spectra (1′, 2′, and 3′) related to curves 1, 2, and 3. Rate window 51 s^{-1}.

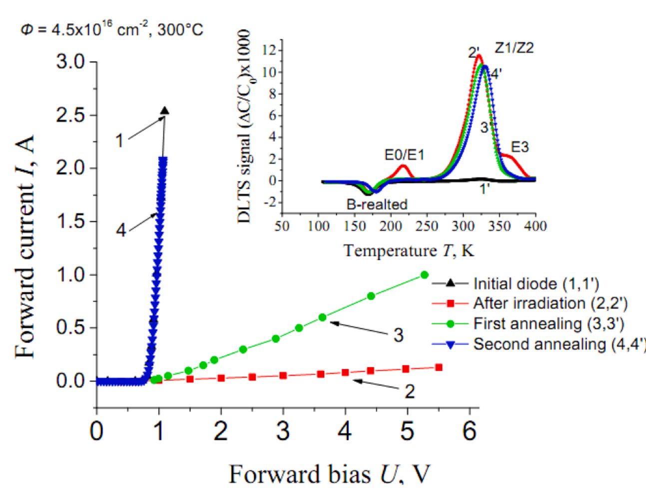

Fig. 2. Forward current–voltage characteristics of the unirradiated diode (curve 1), that after the irradiation at a temperature of 300 °C with a fluence $\Phi = 4.5 \times 10^{16}$ cm^{-2} (curve 2), and that after the first annealing in the basic annealing mode (curve 3), and after the second annealing in the same mode (curve 4). The inset shows DLTS spectra 1′– 4′ corresponding to curves 1–4, respectively. Rate window is 51 s^{-1}.

comparison with the case in Fig. 1, by approximately a factor of 3. The concentration of the E6/7 level measured on control samples increased by approximately a factor of 3.6.

The concentration of the E3 level (inset in Fig. 2) is small compared with N_t for the Z1/Z2 and E6/7 levels and does not make any noticeable contribution to the total concentration of all the levels recorded by DLTS, which for the data in Fig. 2 is $N_{t\Sigma}^{D} \approx 7 \times 10^{14}$ cm^{-3}. Thus, the value of $N_{t\Sigma}^{D}$ is somewhat smaller than one third of the initial electron concentration $n_0 \approx 3.4 \times 10^{15}$ cm^{-3} in the unirradiated sample. At the same time, the *I-V* characteristics indicate a decrease in the free-electron concentrations by approximately a factor of 500 in comparison with the electron concentration in the unirradiated sample.

The first annealing (curve 3) reduces the base resistance to $R_b \approx 3.8$ Ohm, i.e. by a factor of 13. In this case, however, it can be seen from the inset that the DLTS amplitude of the Z1/Z2 maximum remains nearly unchanged (curve 3′). The levels E0/E1 and E3 are almost completely annealed out. This circumstance is surprising, since, as can be seen from the inset in Fig. 1, after the same annealing of the sample irradiated with a fluence $\Phi = 1 \times 10^{16}$ cm^{-2}, the amplitudes of these peaks remain nearly unchanged. As noted above, the total concentration of these levels is small compared to the total concentration $N_{t\Sigma}^{D}$. Thus, annealing of these levels cannot account for the decrease in the resistance R_b.

After the second annealing, the current–voltage characteristic of the sample (curve 4) almost completely coincides with the *I-V* characteristic of the unirradiated diode. As a result of the second annealing, the base resistance R_b decreases by a factor of almost 40.

With the fluence increasing further to $\Phi = 1.3 \times 10^{17}$ cm^{-2} (Fig. 3), there occurs a radical rearrangement of the DLTS spectra. Besides, the effect of a “*reverse annealing,*” [22,23], i.e. an increase in the base resistance is observed in SiC as a result of the annealing.

An increase in fluence Φ from 4.5×10^{16} cm^{-2} to $\Phi = 1.3 \times 10^{17}$ cm^{-2} leads to a rise R_b from 48 to 270 Ohm after the irradiation (curve 1). Such a change in the resistance is accompanied by a radical restructuring of the low-temperature part (100–250 K) of the DLTS spectrum (upper inset in Fig. 3). After the irradiation (curve 2′), three peaks are observed at $T = 140$, 175, and 212 K which were not observed at smaller values of Φ (compare with the insets in Figs. 1 and 2). It is impossible to interpret these peaks in terms of the commonly accepted classification scheme [6,24].

The temperature dependence of the Z1/Z2 peak after irradiation is shown by curve *c* in the lower inset to Fig. 3, where it is compared with the corresponding dependencies obtained for this level after irradiation with doses of 1×10^{16} и 4.5×10^{16} cm^{-2}. It can be seen that an increase in the fluence at $T_i = 300$ °C leads to a monotonic increase in the amplitude of the Z1/Z2 peak and some shift of the peak towards lower temperatures.

An increase in fluence from $\Phi = 4.5 \times 10^{16}$ cm^{-2} to $\Phi = 1.3 \times 10^{17}$ cm^{-2} is accompanied by a significant (more than threefold) rise in the amplitude of the Z1/Z2 peak. However, as also in the case of lower fluences, neither the first nor the second annealing has any significant effect either on the amplitude or on the temperature position of this peak. It should be noted that the “linewidths” of the lines at half maximum for peaks *a* and *b* are $\Delta T \approx 32$ K. Approximately the same ΔT is also characteristic of the peak width of Z1/Z2 at much lower fluences [16]. However, an increase in the fluence to $\Phi = 1.3 \times 10^{17}$ cm^{-2} is accompanied by a noticeable broadening of the peak (ΔT for peak *c* is 55 K) and by its noticeable shift to lower temperatures.

The first annealing (curve 2) leads to a significant decrease in R_b from 270 to 3.4 Ohm. However, such a decrease in R_b is not accompanied by a significant decrease in the amplitude of the peaks at $T = 140$ and 175^{0}K. The peak corresponding to $T \approx 212$–230 K decreases significantly in amplitude and shifts towards higher *T*.

The repeated annealing leads not to a decrease, but to a certain increase in R_b from 3.4 to 3.9 Ohm (curve 3) (reverse annealing). In this case, no significant rearrangement of the DLTS spectrum occurs (compare curves 2 ’and 3′ in the inset).

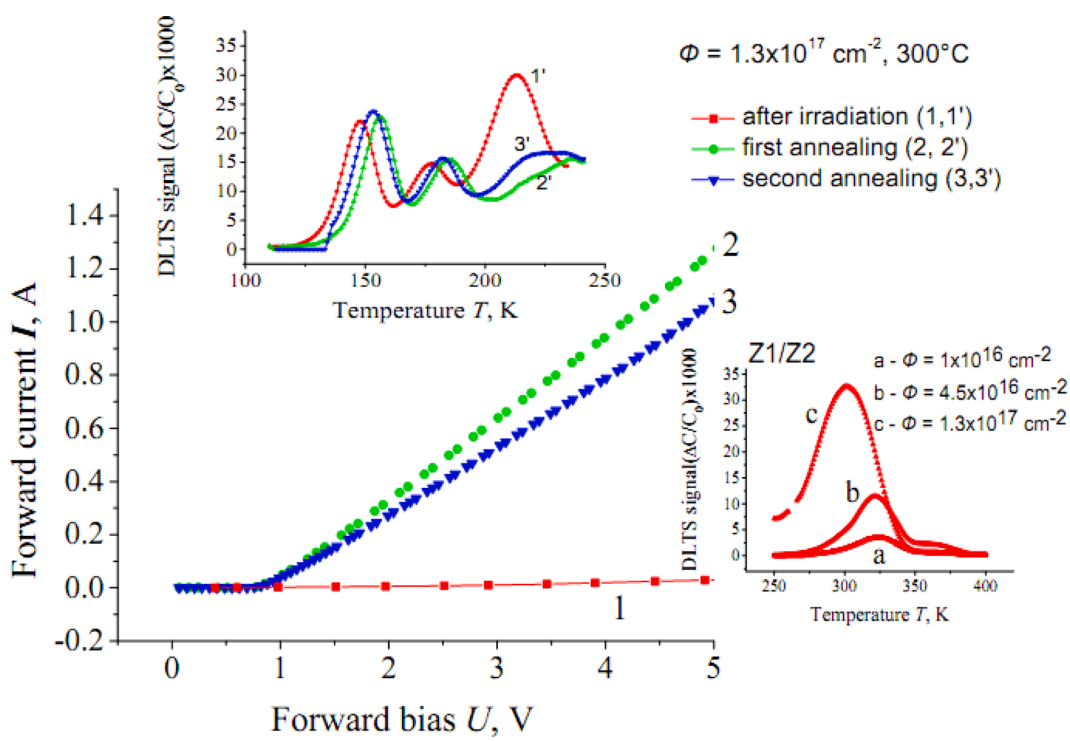

Fig. 3. Forward current–voltage characteristics of the diode after the irradiation with electrons at a temperature of 300 °C with a fluence $\Phi = 1.3 \times 10^{17}$ cm^{-2} (curve 1), after the first annealing in the basic annealing mode (curve 2) and after the second annealing in the same mode (curve 3). The upper inset shows DLTS spectra obtained in the temperature range 100–250 K. Curves 1′, 2′, and 3′ correspond to curves 1, 2, and 3. The lower inset shows the shapes of the peaks corresponding to the Z1/Z2 level at three fluences $\Phi = 1 \times 10^{16}$, 4.5×10^{16}, and 1.3×10^{17} cm^{-2} immediately after the irradiation at $T_i = 300$ °C (curves *a*, *b*, and *c*, respectively). The rate window is 51 s^{-1}.

As noted above, raising the irradiation temperature T_i is accompanied at a given fluence Φ, by a decrease in the base resistance R_b. Fig. 4 shows the results obtained by irradiating the diode at $T_i = 500$ °C with the same fluence $\Phi = 1.3 \times 10^{17}$ cm^{-2} as that in Fig. 3 for $T_i = 300$ °C.

Since the heating of diodes to temperatures exceeding ~ 370 °C may lead to a partial degradation of devices [25], we carried out a series of control experiments to make sure that the irradiation mode we used ($T_i = 500$ °C) did not affect significantly the parameters of the devices. For this purpose, unirradiated samples were heated in a target chamber to a temperature of 500 °C, kept at this temperature, and then cooled in the same mode as that in irradiation experiments. The current–voltage characteristics and DLTS spectra of the devices after such a stress coincided over the entire biases range with the initial characteristics of the primary samples to within the thickness of the line (see curves 1 and 1′ in Fig. 1).

After the irradiation, R_b is approximately 1.5 Ohm, i.e., it is 180 times smaller than that in the case of irradiation with the same fluence Φ at $T_i = 300$ °C (compare with Fig. 3). Raising the temperature to 500 °C also leads to a significant decrease in the amplitudes of the DLTS peaks and to a certain shift in the temperatures corresponding to the position of the peaks (curves 1′ and 2′ in the upper inset in Fig. 4). However, the decrease in the amplitude of the peaks (by approximately a factor of 5) is incomparable with the decrease in R_b, which exceeds two orders of magnitude. Comparison of the amplitudes of the peaks corresponding to the Z1/Z2 level for T_i 300 °C and 500 °C shows that rising in the irradiation temperature also affects very slightly the amplitude of the corresponding peak.

The first annealing after the irradiation with a fluence of $\Phi = 1.3 \times 10^{17}$ cm^{-2} at 300 °C leads to a decrease in R_b by a factor of ~ 13, whereas the same annealing after the irradiation at $T_i = 500$ °C results in that R_b decreases by only a factor of ~ 1.1. During the subsequent annealing, the sample resistance grows rather rapidly (Fig. 4).

The obtained experimental data are summarized in Tables 1 and 2.

Table 1
Irradiation.

	Irradiation temperature, T_i				
	300°C				500°C
	Fluence $\Phi/10^{16}$, cm^{-2}				Fluence $\Phi/10^{16}$, cm^{-2}
	0	1	4.5	13	13
Base resistance, R_b (Ohm)	0.1	1.2	48	270	1.5
Level Z1/Z2 $\Delta C/C_0 \times 1000$	0.15	3.5	11	30	27

As can be seen from Table 1, irradiation with a fluence $\Phi = 1 \times 10^{16}$ cm^{-2} at a temperature = 300 °C (Fig. 1) increases the base differential resistance R_b by more than an order of magnitude. In this case, DLTS measurements demonstrate a multiple increase in the concentration of the Z1/Z2 acceptor level. However, as noted above, after a single annealing in experimentally chosen mode the current–voltage characteristic of the sample coincides with good accuracy with the *I-V* characteristic of an unirradiated diode. Thus, it should be concluded that the acceptor centers arising as a result of the irradiation and responsible for the capture of free electrons are almost completely annealed out. However, as can be seen from the inset in the Fig. 1, there is no noticeable change either in the position or in the amplitudes in the DLTS spectra. It can be assumed that, in addition to the acceptor levels detected by the DLTS method in the upper half of the band gap, the electron irradiation also creates acceptor levels in its lower half of the gap, which are completely annealed out in the mode described above.

An increase in the irradiation dose to $\Phi = 4.5 \times 10^{16}$ cm^{-2} ($T_i = 300$ °C) leads to a further increase in R_b and an increase in the concentration of acceptor levels Z1/Z2 and E3 (Fig. 2). The *I-V* characteristics indicate a decrease in the free-electron concentrations by a factor of 480 in comparison with the electron concentration in the unirradiated sample. In this case, the concentration of the Z1/Z2 level increases by

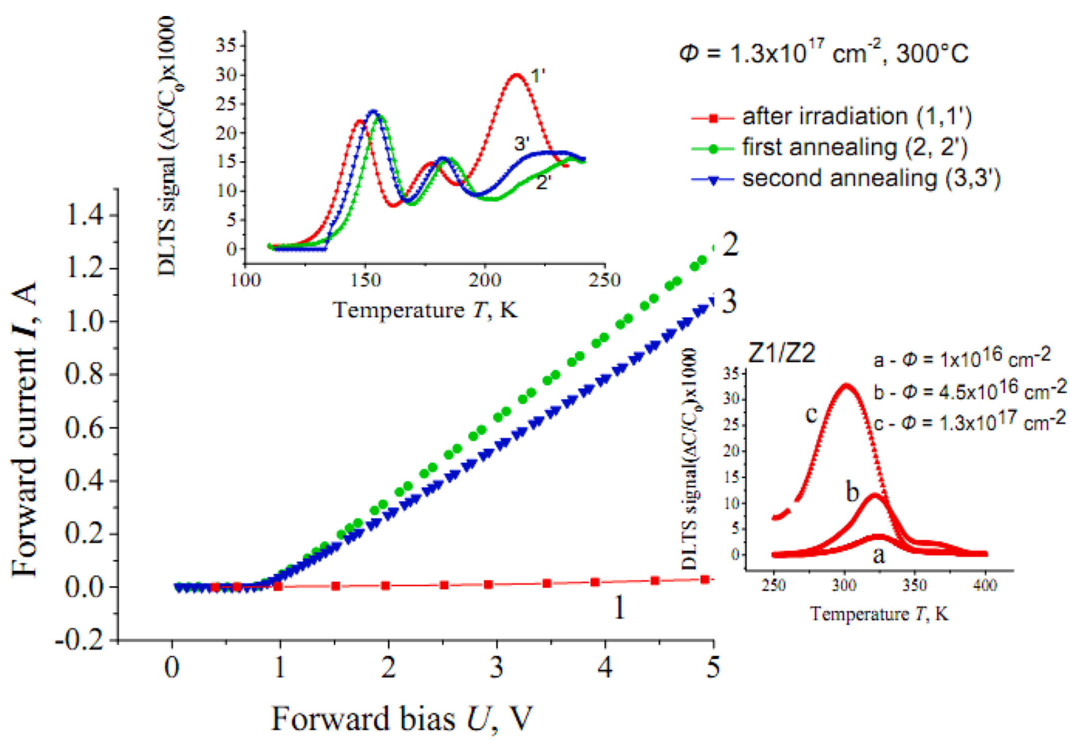

Fig. 4. Forward current–voltage characteristics of the diode after the irradiation with electrons at a temperature of 500 °C with a fluence $\Phi = 1.3 \times 10^{17}$ cm^{-2} (curve 1), after the first annealing in the basic annealing mode (curve 2), after the second and third annealing in the same mode (curves 3 and 4, respectively). The upper inset shows the DLTS spectra measured after the irradiation with a dose of $\Phi = 1.3 \times 10^{17}$ cm^{-2} at 300 °C (curve 1′) and 500 °C (curve 2′). Curve 3′ corresponds to the DLTS spectrum after the second annealing (irradiation at 300 °C); curve 4′ demonstrates a DLTS spectrum after the third annealing (irradiation at 500 °C). The rate window is 51 s^{-1}.

Table 2
Annealing.

	After irradiation at T_i=300°C After fluence $\Phi/10^{16}$, cm^{-2}						After irradiation at T_i=500°C After fluence, $\Phi/10^{16}$, cm^{-2}		
	0	1	4.5		13		13		
Annealing N°	–	1st	1st	2nd	1st	2nd	1st	2nd	3rd
Base resistance, R_b (Ohm)	0.1	0.11	3.8	0.1	3.4	3.9	1.35	2.4	4.1
Level Z1/Z2, ΔC/C$_0$×1000	0.15	3.45	10.5	10.2	28	27	29	27	27

less than 4 times compared with the $\Phi = 1 \times 10^{16}$ cm^{-2}. The total concentration of all acceptor levels created by irradiation in the upper half of the bandgap, $N^D_{t\Sigma}$ is smaller than one third of the initial electron concentration $n_0 \approx 3.4 \times 10^{15}$ cm^{-3} in the unirradiated sample. The first annealing reduces the base resistance by more than an order of magnitude. However, the concentration of the Z1/Z2 level after annealing remains almost unchanged, and the annealing of the E0/E1 and E3 levels with a low concentration cannot explain the observed radical increase in the electron concentration in the conduction band. After the second annealing, the *I-V* characteristics coincide with good accuracy with the characteristics of the unirradiated samples. Thus, the second annealing leads to an increase in the electron concentration in the conduction band by a factor of ~40. However, it can be seen from the inset in Fig. 2 that the second annealing, just like the first one, leads only to a slight decrease in the amplitude and to a small shift of the peak corresponding to the Z1/Z2. That, it can be assumed that under high-temperature (T_i = 300 °C) irradiation, the levels created by irradiation in the lower half of the band gap can play a decisive role.

A further increase Φ to 1.3×10^{17} cm^{-2} (T_i = 300 °C) leads to a relatively small increase in the base resistance, but is accompanied by a radical change in the DLTS spectrum (Fig. 3). After irradiation, peaks appear that cannot be interpreted within the framework of the generally accepted classification. The first annealing leads to a noticeable decrease in R_b. However, as a result of the second annealing, R_b does not decrease, but increases; that is the "reverse annealing" is observed. As far as we know, it is the first observation of the reverse annealing in SiC.

The results obtained suggest that an increase in the fluence is not reduced simply to an increase in the concentration of introduced defects. It can be assumed that under a "hot (300 °C) irradiation", a group of radiation *complexes* appears as the fluence grows. The appearance of such complexes is apparently responsible for the observed phenomenon of reverse annealing.

An increase in the irradiation temperature to T_i = 500 °C at a dose of $\Phi = 1.3 \times 10^{17}$ cm^{-2} is accompanied by a decrease in the R_b resistance by more than two orders of magnitude compared to irradiation with the same dose at T_i = 300 °C. Although the amplitude of the Z1/Z2 level at T_i = 500 °C significantly decreases, the decrease in the concentration of this level (approximately by a factor of 5) cannot explain the radical difference in the R_b values corresponding to the indicated T_i values. The first annealing has almost no effect on the R_b value. The second and third annealings of the sample lead to a noticeable increase in its resistance (Fig. 4), however, the annealings hardly affect the DLTS dependences (inset in Fig. 4).

4. Conclusion

The results obtained primarily indicate that the presence of acceptor centers detected by the DLTS after a hot irradiation in the upper half of the band gap, can explain neither the changes in the current–voltage characteristics nor the results of annealing. Indeed, at the dose $\Phi = 1 \times 10^{16}$ cm^{-2} and T_i = 300 °C, the total number of electrons captured by the recorded acceptor centers is an order of magnitude smaller than the value determined from the change in the base resistance R_b. The subsequent annealing, which completely restores the initial value of R_b, changes only slightly the position and amplitude of the DLTS peaks. Raising the fluence to $\Phi = 4.5 \times 10^{16}$ cm^{-2} at the same T_i leads to an increase in R_b by a factor of 480, compared to the value in the unirradiated sample. Meanwhile, the total concentration of acceptor centers (in the upper half of the band gap),$N^D_{t\Sigma}$), is, in this case, only 5 times lower than the initial electron concentration in the base of unirradiated diode. A double annealing, which restores the initial value of R_b, entails only a very slight change in the total concentration $N^D_{t\Sigma}$.

At the same irradiation temperature (300 °C) and the dose further raised to $\Phi = 1.3 \times 10^{17}$ cm^{-2}, the phenomenon of "reverse annealing" was observed in silicon carbide for the first time: annealing leads to an increase, rather than to a decrease, in R_b. Thus, it is obvious that an increase in the dose (at a constant irradiation temperature) gives rise to a new type of defects. It can be assumed that, at very high values of Φ, the irradiation leads to the formation of radiation complexes. The decomposition of such complexes upon annealing and the formation of point defects may be responsible for the reverse annealing phenomenon. At the same fluence of $\Phi = 1.3 \times 10^{17}$ cm^{-2}, raising the irradiation temperature to 500 °C leads to a sharp (~180-fold) decrease in R_b after the irradiation. However, the phenomenon of reverse annealing after such an irradiation is even more pronounced than that after the irradiation with the same dose at 300 °C. Apparently, it should be concluded that, at large fluences, an increase in the irradiation temperature leads to a more intense formation of radiation complexes. For an adequate interpretation of the results obtained, it is necessary to study the characteristics of the local levels created by the hot irradiation in the lower half of the band gap of silicon carbide. No information of this kind is currently available in the literature. Of undoubted interest is also an analysis of the nature of the radiation complexes created by the hot irradiation at large fluences.

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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