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Electrical properties of highly nitrogen-doped 6H-SiC single crystals: Microwave cavity perturbation study

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Abstract. The silicon carbide (SiC) single crystals of 6H polytype with nitrogen donor concentration $(N_D - N_A) \approx 1\cdot10^{17}... 4\cdot10^{19}$ cm^{-3} grown using the modified Lely method were studied applying the cavity perturbation method. From the temperature dependence of the resonant frequency shift and microwave loss of the cavity loaded with samples under study, the temperature dependence of the conductivity was estimated. From the temperature dependence of the natural logarithm of conductivity *versus* 1000/*T*, the activation energies for processes corresponding to electron transitions from impurity levels to the conduction band (ε_1) and electron hopping over nitrogen donors in the D^0 bands (ε_3) were determined. It was found that in 6H-SiC $\varepsilon_1 = 50$ meV for $(N_D - N_A) \approx 1\cdot10^{17}$ cm^{-3}, $\varepsilon_1 = 32$ meV and $\varepsilon_3 = 6$ meV for $(N_D - N_A) \approx 1\cdot10^{19}$ cm^{-3}, $\varepsilon_1 = 13.5$ meV and $\varepsilon_3 = 3.5$ meV for $(N_D - N_A) \approx 4\cdot10^{19}$ cm^{-3}.

Keywords: conductivity, SiC, cavity perturbation method, activation energy.

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1. Introduction

Silicon carbide (SiC) is a wide bandgap IV-IV compound semiconductor that is considered as a promising material for high-power electronics due to its unique electrical properties. In particular, the high breakdown electric field strength of SiC allows producing SiC-based high-voltage power devices (*e.g.*, metal-oxide-semiconductor field-effect transistors) possessing low on-state resistances [1, 2]. Moreover, the high donor concentration in SiC leads to efficient wavelength conversion, making it a perspective candidate for a wavelength converter in white light-emitting diodes [3]. Therefore, SiC crystals highly doped with nitrogen (N) donors with ultra-low resistivity are of great industrial interest.

The transport measurements often require putting contacts on the sample, and the impact of the contact resistance and capacitance on the measurements becomes troublesome; therefore, contactless probes are beneficial, mainly for the case of small-size samples [4]. One known contactless measurement methods is the cavity perturbation technique [5], which has been used successfully in many studies [6–8]. The cavity perturbation method eliminates the need in contacts to the sample and allows it to work with low signal levels [9]. Thus, the electrical transport parameters of semiconductors can be very easily determined by the microwave (MW) cavity perturbation method [10].

In [11], an improved method of MW conductivity measurements based on the MW absorption measurements in the resonant cavity was proposed. In this method, the conductivity can be estimated from the resonant frequency shift and change of cavity quality factor (*Q*-factor) after the insertion of the sample. The benefits of this approach are: higher accuracy and a wider application range, which allows studying the materials with higher conductivity [12, 13]. According to [11], cavity perturbation measurements may be simple and fast technique for determining the main impurity states responsible for the conductivity in the semiconductor materials and their ionization energies with 10…15% accuracy, with no need for clear understanding the scattering mechanisms.

The previous study of nitrogen-doped SiC crystals of 6H polytype by using the contactless MW conductivity method based on the fact that the conductivity is

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reverse proportional to the loaded cavity Q-factor value was performed in [14], where the change of Q-factor was taken into consideration in these studies only.

This paper is aimed to show how the microwave-cavity perturbation technique, considering the resonant frequency shift and change of cavity quality factor, may be used to investigate the electrical properties of monocrystalline n-type SiC of 6H polytype highly doped with nitrogen donors.

2. Materials and methods

The n-type 6H-SiC wafers with $(N_D - N_A) \approx 1{\cdot}10^{17} \ldots 4{\cdot}10^{19}$ cm^{-3} were grown applying the modified Lely method at 2200…2400 °C and 30…50 mbar of Ar pressure with 1.2 mm/h growth rate on [0001] Si face by using polycrystalline SiC as a source material [15].

The cavity perturbation measurements were performed using the X-band (operation MW frequency $f_0 \sim 9.4$ GHz) Bruker ELEXSYS E580 spectrometer equipped with ER 4122 SHQE SuperX High-Q cylindrical TE_{011} cavity and ER 4112HV variable temperature helium-flow cryostat. The Q-factor value of the cavity, when unloaded and loaded with the sample, was estimated at a MW power level of 0.07518 mW (33 dB).

3. Results and discussion

It is known that after inserting the sample, the empty cavity's resonant frequency and Q-factor change owing to variation in the overall capacitance and conductance of the cavity without perturbing the inductance [16]. Thus, when a small sample is inserted into a MW cavity, the changes of its resonant frequency δ and its MW loss Δ are given by [10]:

$$\delta = \frac{f_0 - f_L}{f_0}, \tag{1a}$$

$$\Delta = \frac{1}{Q_L} - \frac{1}{Q_0}, \tag{1b}$$

where f_0, f_L are the resonant cavity MW frequency of the unloaded and loaded cavity with the sample, Q_0, Q_L are the Q-factors of the unloaded and loaded cavity with the sample, respectively. The cavity Q-factor is determined as $Q = f/\Delta f$, where f is the resonance frequency of the cavity, and Δf is the width at half of the resonance amplitude. Most samples possess non-resonant MW absorption *via* the electric field, and an increase in the dissipated energy will decrease the Q-factor value.

Fig. 1 shows the temperature dependence of microwave loss and frequency shift in 6H-SiC single crystals with different donor concentrations.

In the quasi-static approximation, the frequency shift δ and MW loss Δ-values are related to the complex dielectric constant $\tilde{\varepsilon} = \varepsilon' + j\varepsilon''$ by the following expressions (see, *e.g.* [10]):

$$\delta = \frac{\alpha}{N}\cdot\left(1 - \frac{1 + N\cdot(\varepsilon' - 1)}{[1 + N\cdot(\varepsilon' - 1)]^2 + (N\cdot\varepsilon'')^2}\right), \tag{2a}$$

$$\Delta = \frac{2\cdot\alpha}{N}\cdot\frac{N\cdot\varepsilon''}{[1 + N\cdot(\varepsilon' - 1)]^2 + (N\cdot\varepsilon'')^2}, \tag{2b}$$

where α is the filling factor (for TE_{011} mode cavity: $\alpha = 2V_s/V_c$, V_s – sample volume, V_c – cavity volume), N – sample depolarization factor.

In [10, 17], it was proposed to consider ε′ as a constant or to neglect it as compared to ε″ in the denominator of Eqs (2a) and (2b) over the whole temperature range. Thus, within the Drude formalism, one can write that [10]:

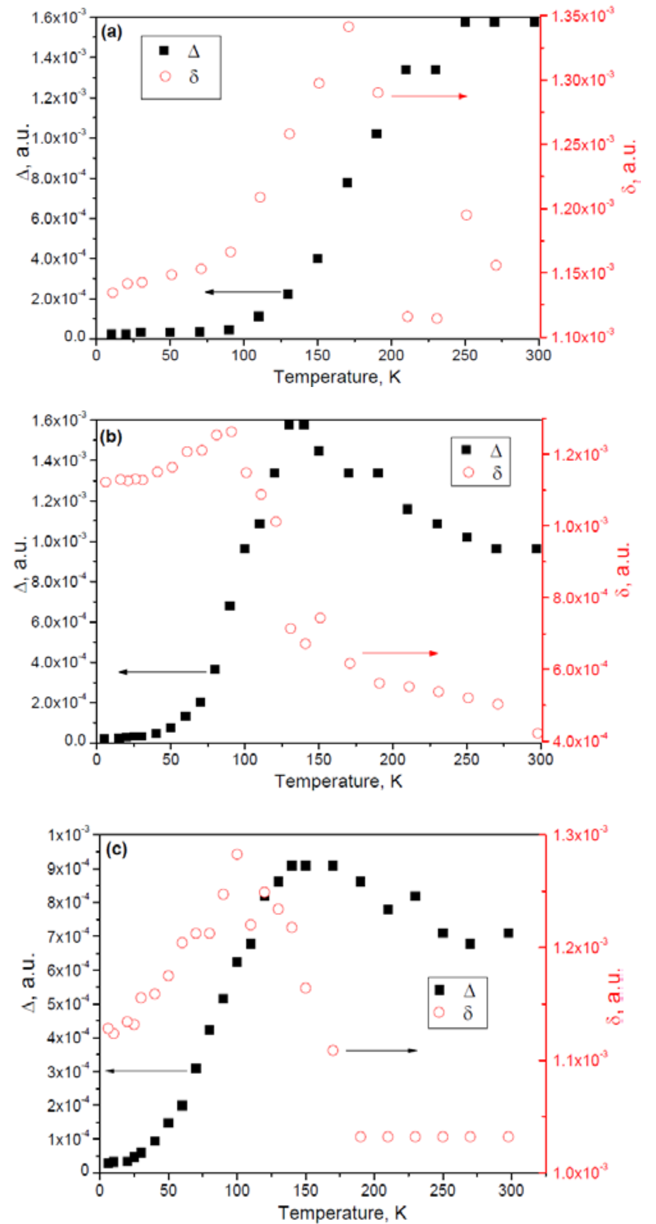

Fig. 1. Temperature dependence of microwave loss (■) and frequency shift (○) for 6H-SiC single crystals with $(N_D - N_A)$ close to $1{\cdot}10^{17}$ cm^{-3} (a), $1{\cdot}10^{19}$ cm^{-3} (b) and $4{\cdot}10^{19}$ cm^{-3} (c) calculated using Eqs (1b) and (1a), respectively.

$$\frac{\varepsilon'_D}{\varepsilon''_D} = -\frac{f}{f_\tau}, \tag{3}$$

where ε'_D is the contribution of free carriers to ε', $f = 2\pi f_0$ is the MW frequency, $f_\tau = e/m\cdot\mu$ is the free carrier collision frequency.

For most semiconductors $f_\tau > 10^{12}$ s^{-1} [10], thus for $f_0 = 9.4$ GHz: $f/f_\tau < 0.06$, and as a result, we can estimate that [10]:

$$\varepsilon''_D = \frac{\varepsilon_L \cdot f_p^2 \cdot f_\tau}{f \cdot \left(f^2 + f_\tau^2\right)} \approx \frac{\varepsilon_L \cdot f_p^2}{f_\tau \cdot f} = \frac{\sigma}{\varepsilon_0 \cdot f}, \tag{4}$$

where ε_L is the lattice dielectric constant, f_p is the plasma frequency, $\varepsilon_0 = 8.854\cdot10^{-12}$ F·m^{-1} – vacuum electric constant, σ is the DC conductivity.

According to [16], the dielectric constant ε′ and loss ε″ from Eqs (2a) and (2b) can be expressed as:

$$\varepsilon' - 1 = \frac{1}{N}\cdot\delta\frac{\left(\frac{\alpha}{N}-\delta\right)-\left(\frac{\Delta}{2}\right)^2}{\left(\frac{\Delta}{2}\right)^2+\left(\frac{\alpha}{N}-\delta\right)^2}, \tag{5a}$$

$$\varepsilon'' = \frac{\alpha}{N^2}\frac{\frac{\Delta}{2}}{\left(\frac{\Delta}{2}\right)^2+\left(\frac{\alpha}{N}-\delta\right)^2}. \tag{5b}$$

Using Eqs (4) and (5b) and putting $\varepsilon'' = \varepsilon''_D$, which allows us to neglect all the loss in the sample that are not caused by free carriers, gives us the expression for the temperature dependence of the conductivity similar to those reported in [11]:

$$\sigma(T) = 2\pi\cdot f_0\cdot\varepsilon_0\cdot\frac{\alpha}{N^2}\frac{\frac{\Delta(T)}{2}}{\left(\frac{\Delta(T)}{2}\right)^2+\left(\frac{\alpha}{N}-\delta(T)\right)^2}. \tag{6}$$

According to [10], an insignificant error in α and N values affects the absolute value of ε″ significantly, however, it has only a tiny effect on $\varepsilon''(T)$ and thus on $\sigma(T)$, which is essential for determining the energy gap and activation energies from the $\sigma(T)$ dependence.

Fig. 2 represents the natural logarithm of MW conductivity obtained from Eq. (6) *versus* $1000/T$ for 6H-SiC single crystals with different donor concentrations. It is well known that the $\sigma(T)$ in n-type semiconductors in a general case is described by the sum of the following terms [18, 19]:

$$\sigma(T) = \sigma_1\exp\left(\frac{-\varepsilon_1}{-k_\mathrm{B}T}\right)+\sigma_2\exp\left(\frac{-\varepsilon_2}{-k_\mathrm{B}T}\right)+\sigma_3\exp\left(\frac{-\varepsilon_3}{-k_\mathrm{B}T}\right) \tag{7}$$

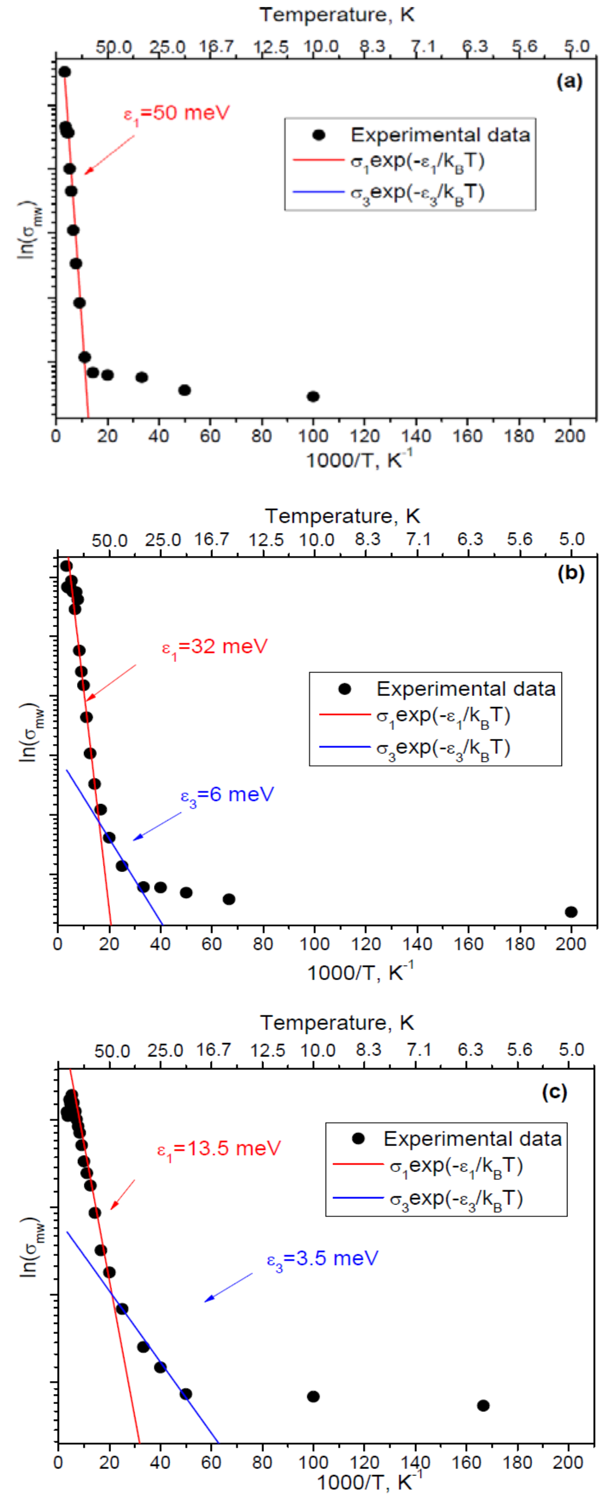

Fig. 2. Natural logarithm of MW conductivity *versus* $1000/T$ for 6H-SiC single crystals with $(N_D - N_A)$ close to $1\cdot10^{17}$ cm^{-3} (a), $1\cdot10^{19}$ cm^{-3} (b) and $4\cdot10^{19}$ cm^{-3} (c). Dots are the data obtained from Eq. (6), and the solid lines are the results of the fitting of terms from Eq. (7).

where σ_1, σ_2, σ_3 denote electrical conductivity values, extrapolated to zero reciprocal temperature ($1/T \to 0$); σ_1 is due to electron transitions from impurity levels to

Table. Activation energies ε_1 and ε_3 in 6H-SiC single crystals with different donor concentrations obtained using various experimental methods.

$(N_D - N_A)$, cm^{-3}	$\geq 10^{17}$	10^{17}		10^{19}		$4 \cdot 10^{19}$	
ε_1, meV	69…193	50	31	32	23	13.5	11.5
ε_3, meV	6.4…13	–	–	6	5	3.5	2.5
Method for measure-ments	Hall measure-ments	Cavity perturbation method	Estimation from ~$1/Q$	Cavity perturbation method	Estimation from ~$1/Q$	Cavity perturbation method	Estimation from ~$1/Q$
Reference	[21]	This work	[14]	This work	[14]	This work	[14]

the conduction band, σ_2 is defined by electron transitions between Hubbard zones (electrons from the D^0 to the D^- band), σ_3 is associated with electron hops over impurity atoms in the D^0 band; ε_1, ε_2, ε_3 are thermal activation energies of the corresponding charge transfer channels [20], k_B is the Boltzmann constant. The activation energy ε_2 is observed only when minor impurities weakly compensate for the main impurities.

Fitting the corresponding terms from Eq. (2) with the data represented in Fig. 2 enabled to obtain the ε_1 and ε_3 values in 6H-SiC single crystals with different donor concentrations (see Table).

From Fig. 2, it follows that the process of electron transitions from impurity levels to the conduction band takes place in 6H-SiC single crystals within the range from 297 to 130 K for $(N_D - N_A) \approx 1 \cdot 10^{17}$ cm^{-3}, from 297 K to 60 K for $(N_D - N_A) \approx 1 \cdot 10^{19}$ cm^{-3} and from 190 K to 50 K for $(N_D - N_A) \approx 4 \cdot 10^{19}$ cm^{-3}. Moreover, at $T = 297…190$ K in 6H-SiC with $(N_D - N_A) \approx 4 \cdot 10^{19}$ cm^{-3}, the conductivity rises slightly due to the scattering process of conduction electrons by ionized nitrogen donor impurities. At the same time, the electron hopping process over the nitrogen impurity atoms in the D^0 bands takes place in 6H-SiC with $(N_D - N_A) \approx$ $1 \cdot 10^{19}… 4 \cdot 10^{19}$ cm^{-3} at $T < 45$ K, while no hopping process in 6H-SiC with $(N_D - N_A) \approx 1 \cdot 10^{17}$ cm^{-3} was observed.

The obtained values are represented in Table along with those derived from temperature dependent Hall measurements [21] and temperature-dependence of MW conductivity estimated as $1/Q$ value in [14]. Thus, we can conclude that the cavity perturbation method gives the ionization energies closer to Hall data than those obtained from $1/Q$ estimation.

According to [21], the activation energy for nitrogen residing the hexagonal site (N_h) in 6H-SiC lattice with the shallowest donor energy level in the bandgap can vary from 34 up to 84 meV in the samples with $(N_D - N_A) \geq 10^{17}$ cm^{-3}. Thus, the ε_1 value obtained in this work for 6H-SiC single crystals with $(N_D - N_A) \approx$ 10^{17} cm^{-3} and $(N_D - N_A) \approx 10^{19}$ cm^{-3} agrees well with these data and corresponds to the N_h donor energy level in the bandgap. The fact that the ε_1 value in 6H-SiC single crystals with $(N_D - N_A) \approx 4 \cdot 10^{19}$ cm^{-3} was found to be smaller, which can be explained by the fact, that for the heavily doped semiconductors, we should take into account the broadening of the impurity donor level and extension of conduction band long tails towards lower energies. Therefore, the ε_1 value should not be considered as the activation energy between a single donor impurity energy level and conduction band edge [22]. At the same time, the obtained ε_3 values in 6H-SiC single crystals fit well with the Hall data shown in [21].

4. Conclusions

We have studied the *n*-type 6H-SiC single crystals grown applying the modified Lely method with different nitrogen donor concentrations $(N_D - N_A)$ from $1 \cdot 10^{17}$ up to $4 \cdot 10^{19}$ cm^{-3} by using the cavity perturbation method. With account of the temperature dependence of conductivity estimated from the resonant frequency shift and microwave loss of the cavity, we have found that the process of electron transitions from nitrogen impurity atoms to the conduction band takes place in 6H-SiC single crystals at $T = 297…130$ K for $(N_D - N_A) \approx 1 \cdot 10^{17}$ cm^{-3} with the activation energy $\varepsilon_1 = 50$ meV, at $T = 297…60$ K for $(N_D - N_A)$ $1 \cdot 10^{19}$ cm^{-3} with $\varepsilon_1 = 32$ meV and at $T =$ $190…50$ K for $(N_D - N_A) \approx 4 \cdot 10^{19}$ cm^{-3} with $\varepsilon_1 = 13.5$ meV. The electron hopping process over impurity nitrogen atoms in the D^0 bands occurs in 6H-SiC with $(N_D - N_A) \approx$ $1 \cdot 10^{19}…4 \cdot 10^{19}$ cm^{-3} at $T < 45$ K with the activation energy $\varepsilon_3 = 6…3.5$ meV, while no hopping process in 6H-SiC with $(N_D - N_A) \approx 1 \cdot 10^{17}$ cm^{-3} was observed. The activation energy values in 6H-SiC single crystals obtained using the cavity perturbation method in this work agrees better with previously reported Hall data than the values obtained earlier with the simple assumption that conductivity is reverse proportional to loaded cavity *Q*-factor value.

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Genkin O.M.: methodology, validation.
Nosachov Yu.F.: conceptualization, writing – review & editing.
Drozdenko O.V.: conceptualization, writing – review & editing.
Moiseenko V.I.: data curation (partially), writing – original draft.
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Електричні властивості монокристалів 6H-SiC з високим вмістом азоту: дослідження мікрохвильовим резонаторним методом

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Анотація. Резонаторним методом досліджено монокристали карбіду кремнію (SiC) політипу 6H з концентрацією донорів азоту $(N_D - N_A) \approx 1\cdot10^{17}...4\cdot10^{19}$ cm^{-3}, вирощені модифікованим методом Лелі. За температурною залежністю зсуву резонансної частоти та мікрохвильових втрат резонатора, завантаженого досліджуваними зразками, оцінено температурну залежність електропровідності. За температурною залежністю натурального логарифма провідності від $1000/T$ визначено енергії активації процесів, що відповідають переходам електронів із домішкових рівнів у зону провідності (ε_1) та перестрибування електронів по донорах азоту в зонах D^0 (ε_3). Установлено, що в 6H-SiC $\varepsilon_1 = 50$ меВ для $(N_D - N_A) \approx 1\cdot10^{17}$ cm^{-3}, $\varepsilon_1 = 32$ меВ і $\varepsilon_3 = 6$ меВ для $(N_D - N_A) \approx 1\cdot10^{19}$ cm^{-3}, $\varepsilon_1 = 13{,}5$ меВ і $\varepsilon_3 = 3{,}5$ меВ для $(N_D - N_A) \approx 4\cdot10^{19}$ cm^{-3}.

Ключові слова: провідність, SiC, резонаторний метод, енергія активації.