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Effect of pulse thermal treatments on the Ni(Ti)–*n*-21R(6H)-SiC contact parameters

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Abstract. We present experimental investigations of the effect of rapid thermal treatment with incoherent IR radiation, as well as electric-spark and electron-beam treatments, on the electric parameters of Ni(Ti) *n*-21R(6H)-SiC contacts. The results obtained show that pulse thermal treatment is an efficient technique for local change of parameters of heterogeneous metal/silicon carbide structures.

Keywords: Ni(Ti) *n*-21R(6H)-SiC contacts, Schottky barrier, rapid thermal treatment, electric-spark treatment, electron-beam treatment, atomic force microscopy, secondary ion mass spectroscopy.

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1. Introduction

The main line in development of manufacturing technology for semiconductor devices intended for high-temperature electronics and microsystem engineering is search for novel methods of controlled provision of required parameters of spatial-nonuniform heterogeneous structures, as well as refinement of the existing methods. Almost all semiconductor active elements are just such heterogeneous structures [1–4]. The parameters of such structures (especially of those with Schottky barrier) are determined by morphology and electronic properties of interfaces. Variation of structure parameters and interface morphology has a determining effect on the electric characteristics of contacts. Besides, it enables one to interpret the experimental results and elucidate the role of techniques applied for structure formation and treatment [5, 6].

Pulse thermal treatment with concentrated energy flows (e.g., laser processing, rapid thermal annealing (RTA) with incoherent IR radiation, electric-spark treatment (EST) and electron-beam treatment (EBT)) is one of those technological procedures that make it possible to provide sufficiently local effect on the properties of both discrete devices and integrated circuits [7–9].

The advantages and flaws of laser processing application in technology of microelectronic devices and micromechanical systems are well known (see, e.g., [3, 9]). At the same time other kinds of pulse action on the electrophysical properties of both silicon-carbide device structures with Schottky barrier and ohmic contacts to them has not been adequately investigated.

Here we present the results of experimental studies of the effect of RTA with incoherent IR irradiation, EST and EBT on the electric parameters and morphology of interfaces in Ni(Ti)–*n*-21R(6H)-SiC contacts. The reason for choosing the metal silicon carbide structure was that this structure is the main element of not only the devices of microsystem engineering which provide signal input/output but of all active elements of silicon-carbide electronics as well.

2. The samples and experimental procedure

We studied the structures whose Lely-grown substrates (SiC single crystals of 21R and 6H polytypes) were doped with nitrogen (up to concentration of ~10^{18} cm^{-3}). Both ohmic and barrier Ni–*n*-21R(6H)-SiC contacts were formed using resistive sputtering of ~0.1 μm thick Ni film

onto chemically cleaned (0001) or $(000\bar{1})$ faces (at substrate temperature of ~300°C) followed by RTA with incoherent IR radiation for 10 s at the temperatures of 450, 600, 750, 900, 1000 and 1100°C (a plant ИТО-18 MB [10]). The device structures were formed with photolithography.

To perform experiments on how EST affects the Ni n-6H-SiC contact parameters, we made ohmic contacts (using the above technology) on one face. On the opposite face, we perform EST of SiC surface through the windows (100×100 μm^2) in thermally grown SiO^2 layer 0.15 μm thick. We used a tungsten or aluminum probe 0.25 mm in diameter; the pulse energy was ~10^{-3} J. After this, we sputtered Ni film formed contacts and made RTA at the above temperatures.

The 6H-SiC single crystal samples (made by Bandgap Technologies Inc. 1428 Taylor St., Columbia, SC 29201, USA) intended for the EBT experiments have concentration of uncompensated donors equal to $(1.5–1.6)\cdot10^{17}$ cm^{-3}. They were exposed to standard cleaning procedure. To exclude graphitization, a Si layer was deposited onto the substrate surface. After EBT this layer was removed using the etchant HNO_3:HF (3:1). Ti *n*-6H-SiC contacts were formed on the substrates treated in the above way.

The test structures were either metallization-free or completely metallized SiC samples. We studied the processes proceeding at formation of SiC surface and contacts due to the above pulse treatments. The following techniques were used: atomic force microscopy (AFM), x-ray diffraction (XRD) and secondary ion mass spectroscopy (SIMS).

The parameters of ohmic (contact resistivity ρ_c) and barrier (Schottky barrier height φ_B) contacts were calculated from *I–V* curves taken for the test structures. An analysis of *I–V* curves was performed using traditional techniques [5, 6].

3. Experimental and discussion of results

Figure 1 presents resistivity of the Ni *n*-21R(6H)-SiC contacts (formed on Si and C faces) as function of RTA temperature. These temperature dependences were obtained from analysis of *I–V* curves.

After RTA at temperature up to 400°C, contacts have barrier-type *I–V* curves; after RTA at temperatures over 750°C, *I–V* curves become ohmic-type. The Schottky barrier height φ_B in Ni–*n*-21R(6H)SiC (0001) contacts lies within the 0.7…0.8 eV range (for different samples), both in initial samples and those after RTA at T = 400°C. After RTA at T = 600°C, φ_B decreases and is 0.5…0.6 eV; after RTA at T = 750, 900 and 1000°C, φ_B decreases consecutively: from 0.38…0.42 eV (after RTA at T = 750°C) down to 0.31…0.34 eV (after RTA at T = 1000°C).

The trend in φ_B dependence on the RTA mode remains for similar contacts made on the C face: after RTA at T = 600°C, it is practically the same as for the samples made on the Si face. However, φ_B value in the initial samples made on the C face is by 0.05 eV below that in the samples made on the Si face.

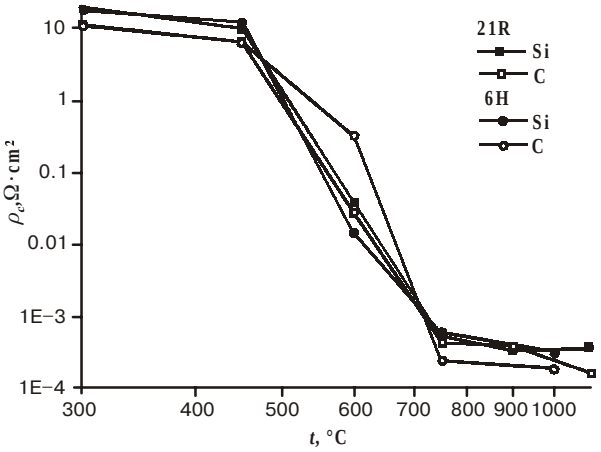

Fig. 1. Resistivity of the Ni–*n*-21R(6H)-SiC contacts (formed on Si and C faces) vs temperature curves obtained from analysis of I–V curves.

In the intermediate temperature range (400…750°C), according to the Ni-SiC phase diagram, the processes of formation of nickel silicides of various compositions occur in contacts.

At RTA up to 400°C, the electric parameters of contacts are due to presence of defects (produced during Ni film deposition onto SiC substrate heated up to 300°C) at the Ni-SiC interface. Variations of contact parameters in the RTA temperature range 400…750°C are related to interface smearing out due to diffusion of Ni and its interaction with SiC. At temperatures of the order of 750°C, a structure of stable morphology and composition is formed. In this case (as shown by us earlier), the contact resistivity value is stabilized at a level of several 10^{-4} Ω·cm^2; it slightly varies when RTA temperature is increased up to 1100°C [11–13]. The above variations practically do not depend on the SiC polytype and the face at which the contact was formed. This is evidenced by the component concentration depth profiles in the initial samples and those annealed for 10 s up to 1000°C (Ni 6H-SiC contact) and 1100°C (Ni–21R-SiC contact) – see Fig. 2.

At thermal annealing of the Ni SiC structures, nickel is spent for formation and transformation of metastable nickel silicide phases. One can see from Fig. 2 that, depending on the face where contact is formed, this process is accompanied with increase of number of either carbon or silicon vacancies at the interface. This is in agreement with the results of [13]. As a result, the structure of films obtained after thermal treatment is nonuniform. This conclusion is supported by the results of investigations of Ni–21R-SiC interface with AFM (Fig. 3) and XRD (Fig. 4) techniques.

It follows from the data given in Fig. 4 that both pure nickel (predominantly hexagonal modification) and nickel silicides $NiSi_2$ (cubic modification), Ni_2Si and NiSi (rhombic lattice) are present in the initial sample. Presence of nickel silicides is related to SiC substrate heating (up to 300°C) in the course of nickel sputtering. It was noted in [15] that the Ni_2Si phase (enriched in metal) appears on silicon substrates at comparatively low

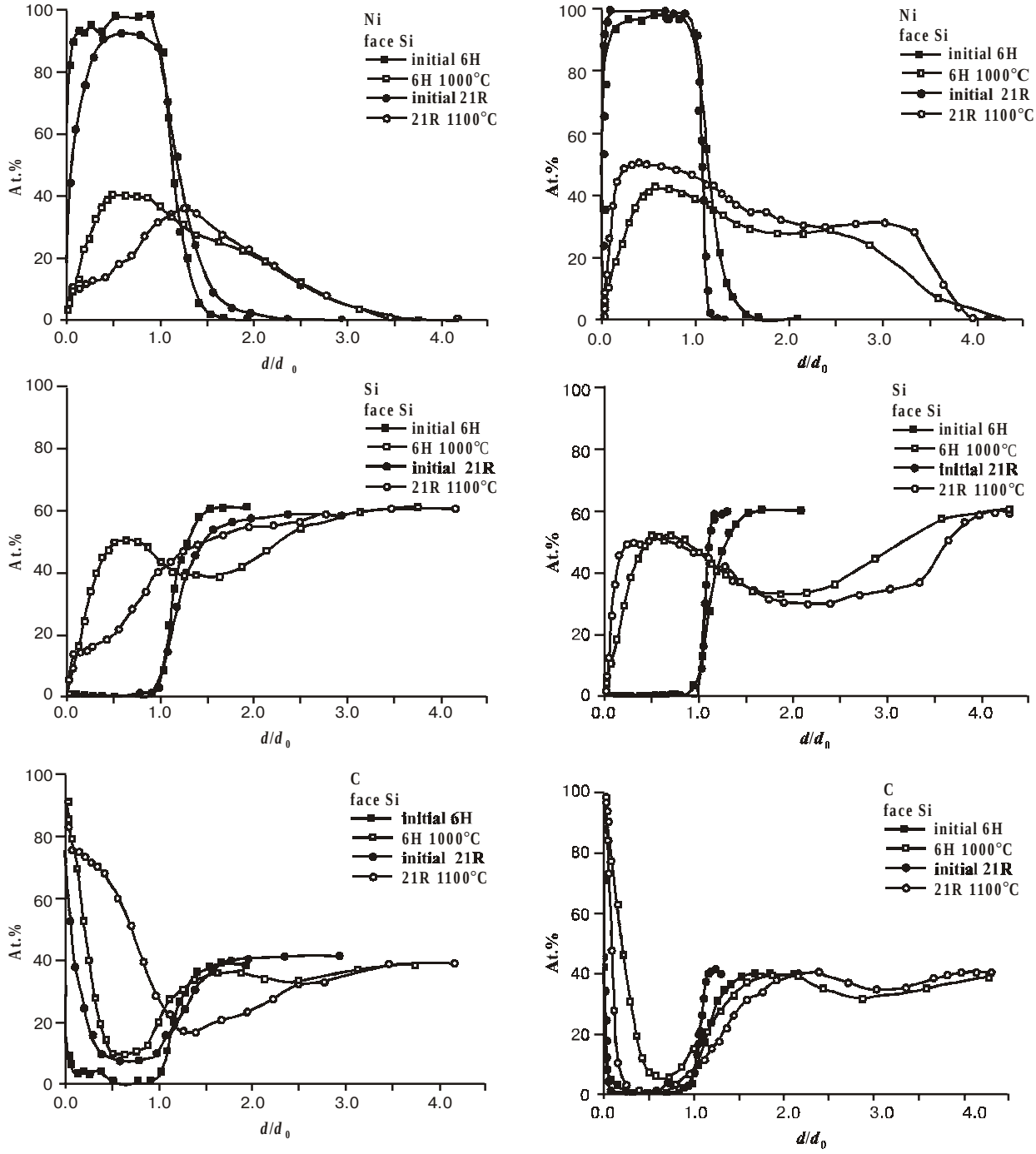

Fig. 2. Component concentration depth profiles in the initial samples and those thermally annealed for 10 s up to 1000°C (Ni–6H-SiC contact) and 1100°C (Ni–21R-SiC contact).

(~200…300°C) temperatures. In our case, this phase is formed on silicon carbide at ≈300°C. It is known [14, 16] that no phase formation is observed in the initial samples if Ni is deposited onto a "cold" 4H-SiC or 6H-SiC substrate ((0001) face for both polytypes). It should be noted also that concurrent existence of several silicide phases after RTA results from the fact that structure reconstruction has not been completed at short-term treatment.

Investigation of *I–V* curves of the Ni–6H-SiC contacts formed at the (0001) face exposed to EST before nickel deposition showed that the contacts were ohmic. Their sufficiently low (~$3 \cdot 10^{-3}$ Ω·cm^2) resistivity ρ_c practically did not change at RTA with temperatures from 400 up to 1100°C. The properties of Ni–6H-SiC contacts formed on the $(000\bar{1})$ face according to the above procedure were identical to those of contacts made on (0001) face.

Thus, exposition of substrate to EST changes completely the properties of metal SiC contacts, since (as was noted before) similar contacts that were formed on 6H-SiC surfaces without EST transformed from barrier to

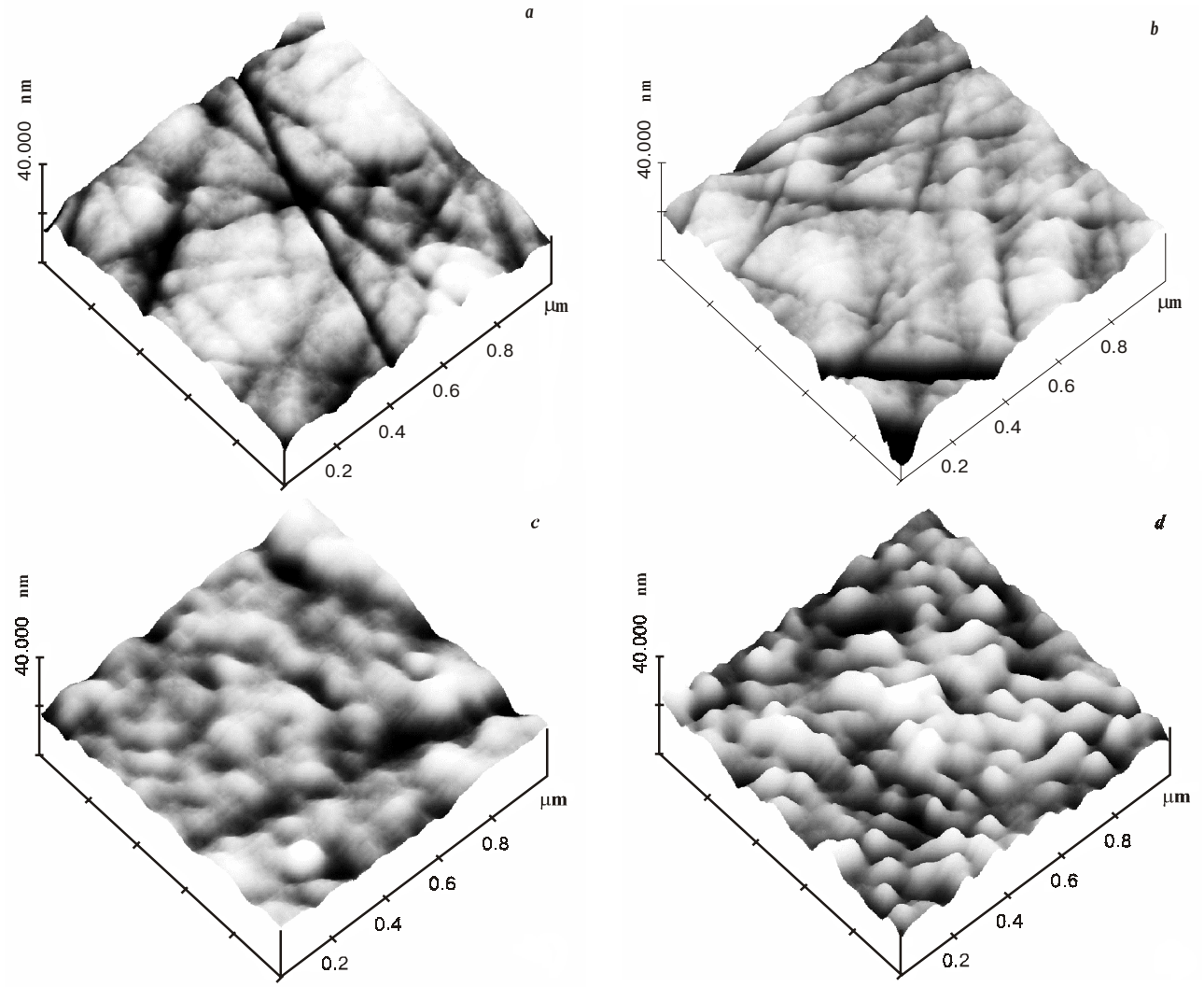

Fig. 3. Interface morphology variation in the Ni SiC structure due to RTA: *a* – initial sample; *b*, *c*, *d* – those annealed up to 450, 750 and 900°C, respectively.

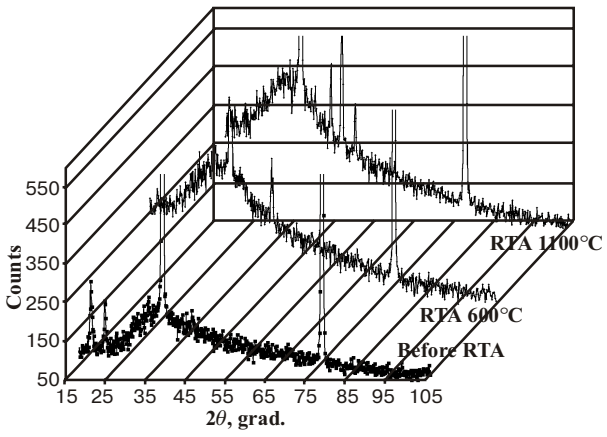

Fig. 4. XRD study of the Ni 21R-SiC interface before and after RTA.

ohmic after RTA at temperatures from the 400–1100°C range, and their resistivity ρ_c becomes minimal at RTA temperature $T = 750$°C. Further increase of RTA temperature practically does not change ρ_c value.

The effect of substrate pretreatment may be explained, first of all, by formation of a developed relief on the substrate surface after EST (see Fig. 5 and Table 1). This leads to enhancement of generation-recombination processes at current flow and formation of ohmic metal semiconductor contacts [5–7, 17]. Indeed, the initial SiC surface was a standard surface of single-crystalline semiconductor that was prepared for sputtering after the corresponding chemo-mechanical treatment (Fig. 5*a*), but after EST surface morphology became nonuniform: along with flat areas, those with very developed relief are present (Fig. 5*b*).

Another factor leading to variation of contact properties may be a complicated composition of SiC surface after EST. This results in structure-phase nonuniformity of the surface that provides non-barrier current-flow

mechanisms and results in decrease of contact resistance in such structures.

Study of near-surface composition of SiC layers after EST with a tungsten nib was made using sufficiently highly-sensitive SIMS technique. It enabled us to detect low concentrations of tungsten and its oxides that were absent at the initial surface. Shown in Fig. 6 is the yield diagram for single-charged positive ions of elements and compounds leaving SiC surface after EST. One can see that there are ions of tungsten and its isotopes, their oxides and positively-charged WOH^+ ions, as well as ions of tantalum and its oxides, in the SiC layer studied.

SiC treated with aluminum electrode has not been studied in such detail. However, an intense aluminum peak detected for it indicates at high possibility of presence, along with atomic aluminum, of its oxide phases. It seems that more purposeful studies of various surface oxides should be made for SiC samples exposed to such treatment, with variation of parameters and change of sign of potential applied to the probe.

The presented dependences show that application of EST makes it possible to form contacts with good ohmic characteristics without use of high-temperature annealing. This enables one to substantially simplify the manufacturing technology for active elements on the silicon carbide basis. Figure 7 presents our experimental results on contact resistivity ρ_c formed by Ni_2Si metallization as function of the dopant concentration in *n*-SiC. One can see that ρ_c values obtained in our experiments without and with RTA for 10 s at $T = 1000°C$ are in agreement with the results on ρ_c obtained by other authors [16, 24–29] who used thermal annealing – see [30].

Contrary to the above treatment, EBT makes SiC surface more flat and clean (see Fig. 5*c* and Table 1). But the metal (titanium) surface morphology on the initial and treated SiC surfaces is practically the same, both in microstructure and roughness (RMS roughness size ~ 4 nm, spread of roughness heights $Z_r \sim 41$ nm).

An analysis of *I–V* curves of the Ti *n*-6H-SiC contacts showed that the check sample had rectifying contacts (the Schottky barrier height of 0.63 eV), while the sample exposed to EBT had ohmic contacts (contact resistivity of $6.2 \cdot 10^{-4}$ $\Omega \cdot cm^2$) [18]. One of the reasons for the observed changes in contacts at such treatments may be

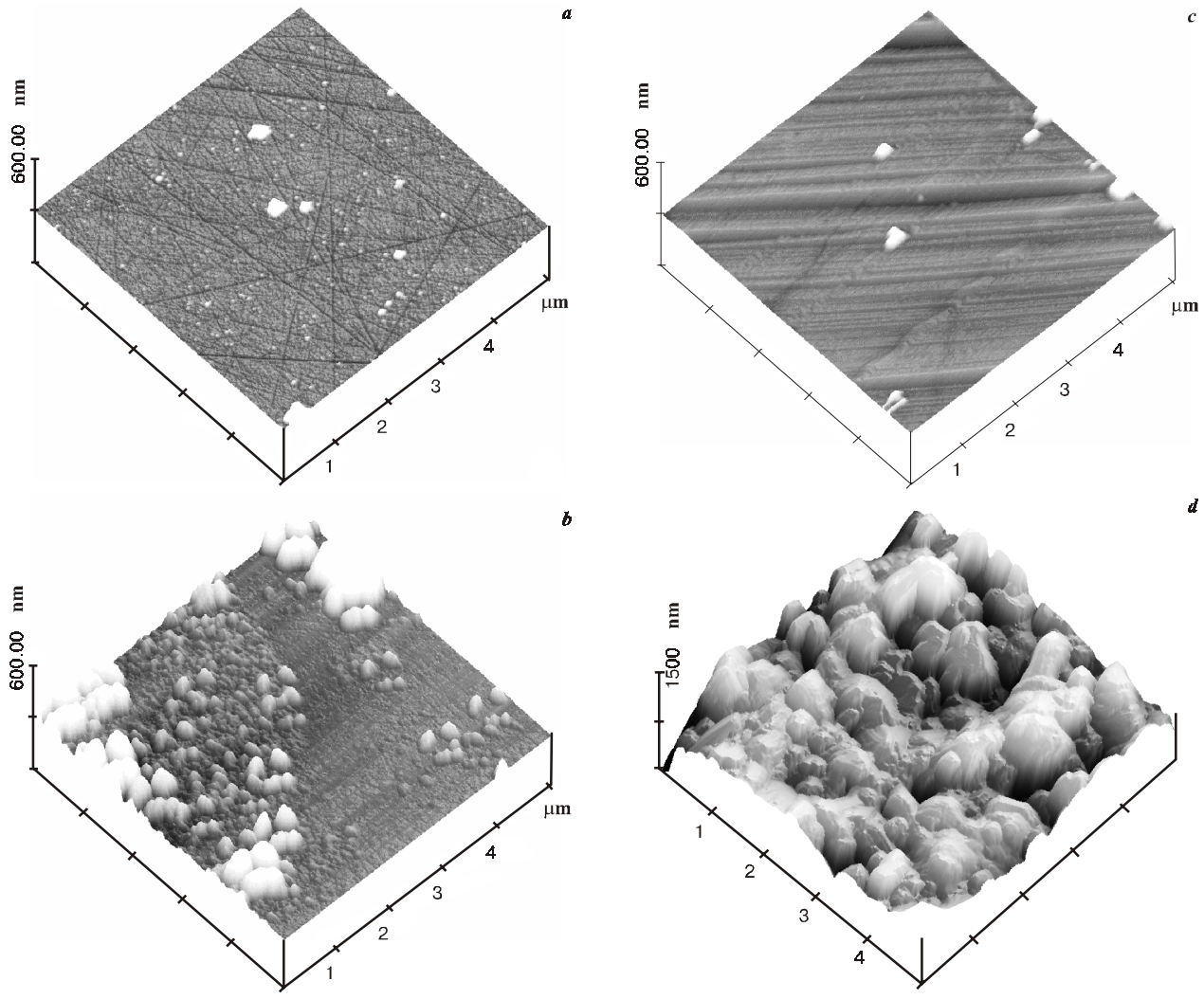

Fig. 5. SiC substrate surface: *a* – initial sample; *b*, *c* – those after EST and EBT, respectively.

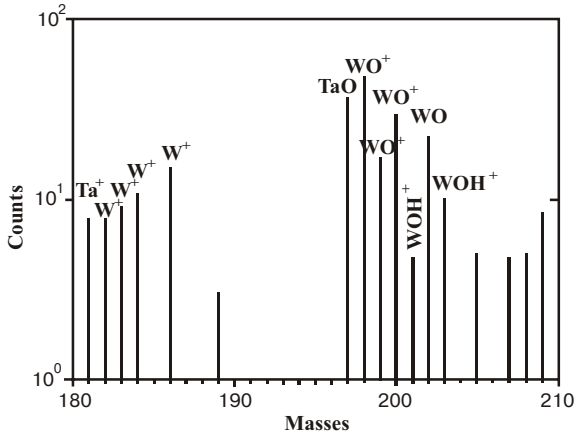

Fig. 6. Yield diagram for single-charged positive ions of elements and compounds leaving SiC surface after EST.

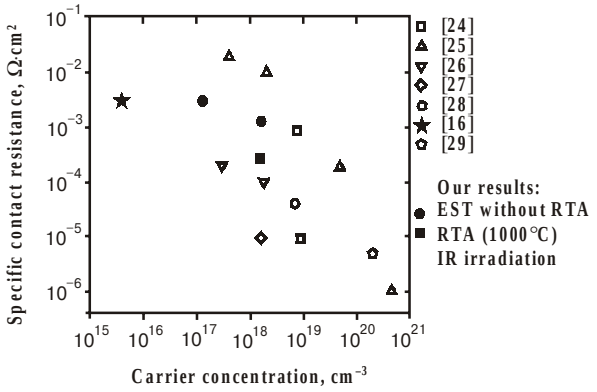

Fig. 7. Contact resistivity as function of dopant concentration in *n*-SiC.

reduction of the density of surface electron states (SES) due to removal of a damaged layer that was present at the surface of the initial samples. Indeed, an analysis of *I–V* curves of the samples after EBT showed that barrier height in the Ti *n*-6H-SiC contact has decreased by 0.3… 0.35 eV as compared to that of untreated samples. Assuming that the mechanism for current flow in the initial portion of *I–V* curve is thermionic, one may determine from the following expression [19]:

$$\rho_C = \frac{k}{qA^*T}\exp\left(\frac{q}{kT}\varphi_B\right), \quad (1)$$

where ρ_C is the contact resistivity, A^* is the Richardson constant (equal to 194 A cm^{-2} K^{-2} for n-6H-SiC), q is the electron charge, and k is the Boltzmann constant. Substituting in Eq. (1) φ_B = 0.33 eV (calculated from the forward branch of *I–V* curve), one obtains $\rho_C \approx 5.1\times 10^{-4}$ Ω·cm^2. This value is in agreement with the measured one ($\rho_C \approx 6.2\cdot10^{-4}$ Ω·cm^2).

Estimation of the SES density NSS was made according to the expression [20]

$$N_{SS} = \frac{\varepsilon_0}{qn}\left[\frac{\varepsilon_S}{l_-}(n^*-1)(n-1) - \frac{\varepsilon_S}{l_+}\right]. \quad (2)$$

Here $\varepsilon_0(\varepsilon_S)$ is the vacuum (semiconductor) permittivity, and $n^*(n)$ is the ideality factor of the reverse (forward) branch of I V curve at small biases. l_+ and l_- are is the space-charge region lengths at reverse and forward biases, respectively. They are determined from the expressions [29]

$$l_+ = \sqrt{\frac{2\varepsilon\varepsilon_0(\varphi_B - V)}{qN_D}} \text{ and } l_- = \sqrt{\frac{2\varepsilon\varepsilon_0(\varphi_B + V)}{qN_D}}$$

where N_D is the dopant concentration in *n*-6H-SiC (equal to $1.6\cdot10^{17}$ cm^{-3}). The barrier height φ_B after EBT is 0.33 eV.

When estimating N_{SS}, we assumed that the current flow mechanism in the initial portions of *I–V* curve is thermionic. The model for metal semiconductor interface took into account SES of two types: those whose filling is determined by interchange with semiconductor (N_{SN}) or with metal (N_{SM}). Allowance was also made to the fact that, at forward bias, charge transfer involves the states whose filling is provided by interchange with semiconductor (i.e., N_{SM} = 0), while at reverse bias N_{SN} = 0. Assuming that it was the same states that are empty (filled) in the first (second) case (i.e., $N_{SM} = N_{SN} = N_{SS}$), we obtained: $N_{SS} = 2.2\cdot10^{12}$ cm^{-2} eV^{-1}.

It was noted in [21] that, at SES density at the Ti n-6H-SiC interface ~10^{12} cm^{-2} eV^{-1}, the barrier disappears and the contact becomes nonrectifying. The results of [22, 23] also indicate at such possibility. Their authors applied various procedures for chemical treatment of substrate to change its structure perfection, and varied SES density in silicon carbide before formation of the Ti *n*-6H-SiC contacts. As a result, the contact characteristics changed from barrier ($\varphi_B \approx 0.6$ eV) to ohmic (resistivity of ~$6\cdot10^{-3}$ Ω·cm^2) [22, 23].

The results with ρ_C values close to the above were obtained for *n*-6H-SiC samples exposed to ion-plasma treatment (IPT) before Ni sputtering. A SiC layer (thickness of ~ 10 μm) has been removed from the surface during ion etching. A developed (strongly destructed) surface produced by such treatment is characterized by increased chemical activity, as compared to that of the initial sample. This leads to bigger possibility of interac-

Table 1. Effect of treatments on *n*-6H-SiC surface morphology (the analyzed surface area is 5×5 μm^2).

Type of sample	RMS roughness size, nm	Spread of roughness heights Z_r, nm
initial	3.43	47.17
after EST	34.53	255.36
after EBT	1.35	11.95

tion with Ni atoms that make chemical bonds (saturate dangling bonds of semiconductor) with the atoms of semiconductor. The result is a decrease of N_{SS} in the Ni-*n*-6HSiC contact and (related to this) reduction of φ_B. The latter factor provided formation of ohmic contact with $\rho_C \approx (1\ldots2)\ 10^{-3}\ \Omega\cdot cm^2$, without further high-temperature firing.

Thus the most apparent reason for ohmic contact formation at *n*-6H-SiC surface after EBT and IPT without further high-temperature annealing is reduction of Schottky barrier height due to decrease of N_{SS}.

4. Conclusions

In closing, we would like to note that, despite significant distinctions between the considered pulse treatments of SiC surfaces and contact structures, they can be applied for control over formation of nonrectifying Ni(Ti)–*n*-6H-SiC and Ni–*n*-6H(21R)-SiC contacts. Such control may be realized using RTA, as well as without it, after deposition of contact metallization onto silicon carbide surface previously exposed to pulse treatments (EST, EBT or chemo-ionic).

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