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Structural and electrical properties of Ni/Ti Schottky contacts on silicon carbide upon thermal annealing

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The evolution of the structural and electrical properties of Ni/Ti/SiC Schottky contacts upon thermal treatments was investigated. The samples were prepared by sequentially evaporating titanium and nickel layers onto silicon carbide (6H-SiC) substrates and were annealed in vacuum in the temperature range 400–650 °C. Above 450 °C a solid state reaction sets in, giving rise to the formation of nickel silicides (i.e., $Ni_{31}Si_{12}$ and Ni_2Si). During reaction, by increasing annealing temperatures, the electrical characteristics of the contacts showed an increase of the Schottky barrier, along with a decrease of the device leakage current. An inversion of this trend was observed at around 600 °C, which can be attributed to the inhomogeneity of the nickel silicide/SiC barrier. The scenario of the reaction of the Ni/Ti/SiC system is presented. The physical information obtained from the study of this bilayer can be extremely important in the control of the electrical properties of Schottky barriers for advanced devices on SiC. *© 2004 American Institute of Physics*. [DOI: 10.1063/1.1787138]

I. INTRODUCTION

The fabrication of either ohmic and rectifying contacts on silicon carbide (SiC) may be achieved by using metal silicides.[1] In particular, different reactions can take place in a metal/SiC system, depending on the metal and on the annealing conditions. Some metals, such as silver or gold, do not react with SiC, other metals can form silicides and/or carbides (i.e., Co, Ni, Cr, Fe, Pt, Pd, Ti, W,…), while some others are also able to form ternary compounds (i.e., Mo, Ta, Ti, Zr,…).[2] The metals which are most widely used in the technology of SiC power diodes, for Schottky and/or for ohmic contacts, are titanium and nickel.[3–5] Titanium gives values of Schottky barrier height lower than those given by nickel, both on 6H-SiC and 4H-SiC.[1,6]

As concerns the technological fabrication processing, Schottky diodes are the most progressed SiC power devices and, in fact, they are already commercially available. Nevertheless, their performances require to be further improved by optimizing the efficiency of the existing edge terminations, as well as by developing new metallization concepts for the Schottky contact. The last concern led recently to several efforts in the fabrication of dual-metal Schottky diodes,[7–9] i.e., devices in which the Schottky contact was formed by two metals (Ni, or nickel silicide Ni_2Si, and Ti). The dual-metal diodes were able to combine a low forward voltage drop V_F (i.e., that of the lower barrier of Ti) with a low reverse leakage current I_L (i.e., that of the higher barrier of Ni or Ni_2Si). However, the control of the electrical properties of dual-metal contacts, beyond the device geometry, may also be strictly related to the effects of thermal annealings on the metal/semiconductor systems.

In silicon, the control and the improvement of the electrical properties of silicides contacts were object of study in the last years. In particular, it has already been observed that the addition of thin metallic interlayers at the metal/Si interface can affect the silicidation mechanism[10–12] and lead to lower resistivity values and to a better thermal stability of the contacts. The use of metallic interfacial layers was also applied in silicon carbide, where a reduction of the specific resistance of sintered Ta and Mo ohmic contacts on *p*-type 6H-SiC was achieved by using an Al interlayer.[13]

A bilayer contact Ni (∼100 nm)/Ti (∼10 nm)/SiC has been already used by Vassilevski *et al.*[8] in the fabrication of high performance dual-metal SiC Schottky diodes. The electrical behavior of these contacts after annealing showed a voltage drop V_F as that of a Ti/SiC diode and a leakage current I_L comparable to that of a Ni/SiC diode that the authors attributed to the presence of the high Schottky barrier of Ni at the device edges. However, in spite of the potential technological applications, an analysis of the structural changes of the Ni/Ti/SiC system subjected to thermal annealing was not reported in the literature.

The aforementioned reasons make the knowledge of the properties of Ni/Ti bilayers on SiC upon thermal treatments an extremely interesting issue for SiC devices technology. In fact, thermal annealing of this system can result in the formation of phases with different barrier heights at the interface. Hence, the forward and reverse *I*-*V* characteristics may depend both on the fraction area of the different barriers and on the size of the low barrier regions (patches).[14] In this case, as predicted by Tung's model,[14] if the average dimensions of the low barrier regions are comparable to those of the depletion width, the low barrier regions rule the current flow under forward bias, leading to a low V_F. In reverse bias, instead, "pinch off" of the low barrier regions by the high ones can occur, thus resulting in a low leakage current. Therefore, this kind of inhomogeneous barrier can be useful for device fabrication.

In this paper, the behavior of Ni/Ti bilayers on 6H-SiC after thermal annealing is reported, and the structural properties of the system are correlated to the electrical characteristics of Schottky diodes. A solid state reaction sets in above 450 °C, leading to the formation of nickel silicides, thus strongly influencing the Schottky barrier height and hence the electrical properties of the contacts.

II. EXPERIMENTAL DETAILS

Silicon carbide (6H-SiC) substrates by CREE Research Inc. were used for the structural characterization of the Ni/Ti/SiC system. The metallic layers were sequentially deposited in an electron gun ultrahigh vacuum evaporator, at a base pressure of 3×10^{-9} mbar. The thickness of the Ti layer was ≈5 nm while that of the Ni layer 100 nm. Thermal annealings of the samples were performed in a conventional vacuum furnace, operating at a pressure of 4×10^{-6} mbar, between 400 and 650 °C for 30 min. The structural characterization of the system was carried out by combining transmission electron microscopy (TEM) analysis performed using a 200 kV JEM 2010 JEOL microscope, and x-ray diffraction (XRD) carried out with a Bruker AXS D5005 diffractometer. In particular, energy filtered transmission electron microscopy (EFTEM) was used to monitor the evolution of the system by extracting the chemical maps of the elements. This technique allows to map the elemental chemical composition of the sample, without losing the spatial resolution typical of the TEM analysis, by subjecting the transmitted electron beam to an energy filtering process. Since the energy losses of the electron beam are the characteristics of the interaction with the specimens present in the matrix, selecting electrons that have lost energy by specific scattering effects results into an element specific contrast in the image, i.e., the chemical map of the element.

The evolution of the electrical properties of the Ni/Ti/6H-SiC contact after thermal annealing processes was monitored by measuring the current-voltage (I-V) characteristics of test Schottky diodes. the I-V curves were acquired in a Wentworth probe station equipped with a Keithley 236 source meter. The diodes were fabricated onto 4 μm thick epitaxial layers (n-type doped) with an epilayer carrier concentration of about 3×10^{15} cm^{-3}. The carrier concentration of the heavily doped substrate was $\sim5\times10^{18}$ cm^{-3}. A silicon dioxide layer (1 μm thick) was first deposited via CVD onto the sample front side. Nickel silicide (Ni_2Si), formed by rapid thermal annealing in nitrogen at 950 °C before processing the wafer front, was used as low resistance back side ohmic contact. Then, standard optical photolithography and wet etches were used to define the circular contact geometry of the diodes with an active area of 3.4×10^{-4} cm^2. The diodes were annealed in vacuum in the same annealing runs together with the blanket samples for the structural analysis.

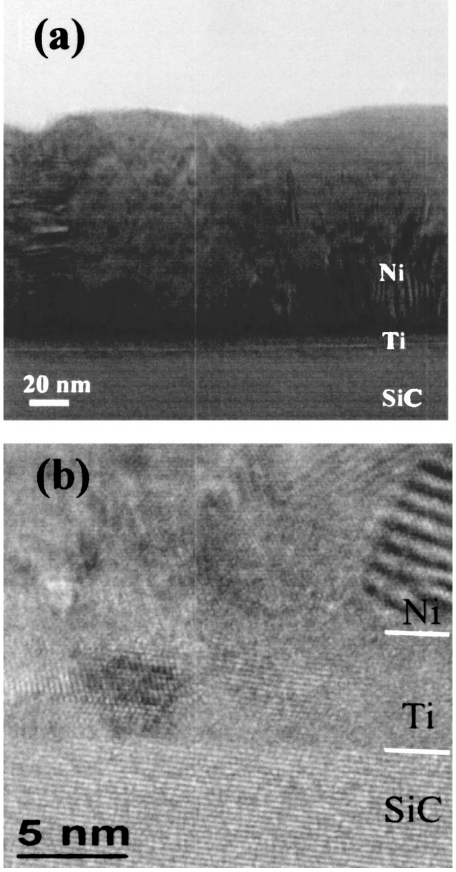

FIG. 1. (a) Cross section TEM image of an as-deposited Ni/Ti/6H-SiC sample. (b) High resolution TEM of the same sample.

III. RESULTS

A. Structural characterization

A cross section TEM image of the “as-deposited” Ni/Ti/SiC bilayer is reported in Fig. 1(a), from which the thickness of the Ti and Ni layers were determined to be 6 and 100 nm, respectively. Figure 1(b) shows the high resolution cross section TEM picture of the same sample, taken along the $[11\bar{2}0]$ zone axis, which is perpendicular to the [0001] direction of 6H-SiC. From this image, it can be seen that the planarity of the interface is almost completely preserved after deposition, but the deposited Ti film has not grown epitaxially onto the 6H-SiC substrate. Conversely, Porter *et al.*[15] demonstrated that the deposition of Ti on 6H-SiC (0001) results in epitaxial films, since both 6H-SiC ($a=3.08$ Å, $c=15.11$ Å); and Ti (Ti, $a=2.95$ Å, $c=4.68$ Å) have hexagonal structures with a lattice mismatch of 4%. The different behavior observed in our samples can be attributed to a non-optimized surface preparation and cleaning prior to Ti deposition or to different vacuum conditions during evaporation, which in turn are critical factors for achieving a good epitaxy.

After deposition, the bilayers were undertaken to thermal annealing in vacuum up to 650 °C and the structural changes of the system were monitored both by XRD and EFTEM.

The XRD spectra of the Ni/Ti/SiC samples, taken in Bragg-Brentano configuration, at various annealing temperatures between 450 and 650 °C are reported in Fig. 2. Below

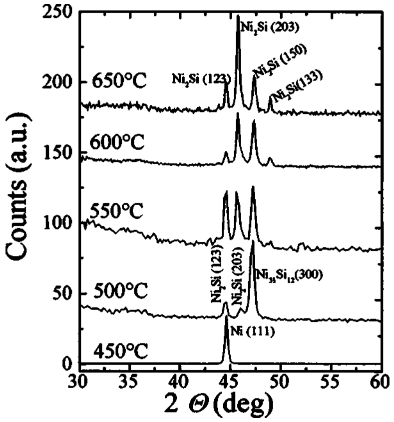

FIG. 2. X-ray diffraction spectra of the Ni/Ti/6H-SiC sample, after annealing in vacuum for 30 min between 450 and 650 °C.

the onset temperature of 450 °C, XRD analysis indicated that no reaction between the metal layer and the SiC substrate occurred. In fact, up to 450 °C, the only Ni (111) signal was detected, thus also indicating the texturing of the deposited film. By increasing the annealing temperature the presence of other features in the XRD spectra reveals the occurrence of a solid state reaction. In particular, the diffraction patterns detected at 500 °C reveal the coexistence of two silicide phases, i.e., the $Ni_{31}Si_{12}$ and the Ni_2Si. At higher temperatures the diffraction signal of $Ni_{31}Si_{12}(300)$ shift towards that of $Ni_2Si(150)$ at 47.3°, indicating the transformation of the Ni rich phase into the more stable Ni_2Si. At the annealing temperature of 550 °C, the XRD spectra also show the presence of a peak located at around 49°, which grows in intensity by increasing annealing temperature. This diffraction signal could arise either from titanium silicide $Ti_5Si_3(220)$ [which is known to form under similar annealing conditions on 4H-SiC (Ref. 16)] or from nickel silicide $Ni_2Si(133)$. However, since the following EFTEM analysis was not able to reveal the presence of Ti at the interface with SiC after annealing, it is more plausible that the signal arises from the diffraction from the (133) planes of the Ni_2Si phase. Therefore, the XRD analyses indicate a structural evolution of the nickel silicides layer, both in terms of the phases (transformation of $Ni_{31}Si_{12}$ into Ni_2Si) and of the orientation of the polycrystalline film. Moreover, from the XRD analyses, there was no evidence of nickel- or titanium-carbide formation. The Ti peaks could not be detected by the XRD spectra acquired in two different configurations. In particular, the texturing of the film did not allow Ti to be detected in glancing angle configuration, while this thin layer (6 nm) could not be detected even in Bragg-Bentano configuration because of the resolution of the technique.

Further insights into the reaction mechanism and, in particular, on the distribution of the elements inside the layer were provided by the EFTEM spectra of the samples which are reported in Figs. 3–5. In these chemical maps, a bright contrast indicates the presence of the element in the sample. The EFTEM spectra of the bilayer system were taken after deposition (Fig. 3) and after annealing at 500 °C (Fig. 4) and 550 °C (Fig. 5).

Figure 3 shows the chemical EFTEM maps of Ti and Ni in the as-deposited sample, which clearly show the presence of the Ti layer (at the interface with SiC), and the thicker Ni layer on the top. Annealing of the samples up to the temperature of 450 °C did not lead any difference in the chemical maps, i.e., the deposited metallic species did not diffuse up to this temperature. On the other hand, as can be clearly seen from the maps reported in Fig. 4, annealing at 500 °C makes

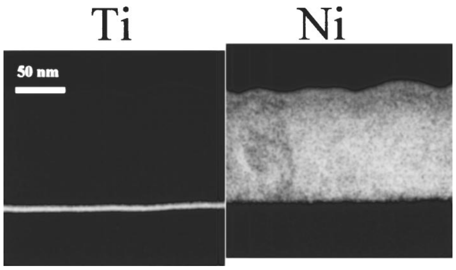

FIG. 3. Ti and Ni maps determined by EFTEM analysis of the as-deposited Ni/Ti/6H-SiC sample.

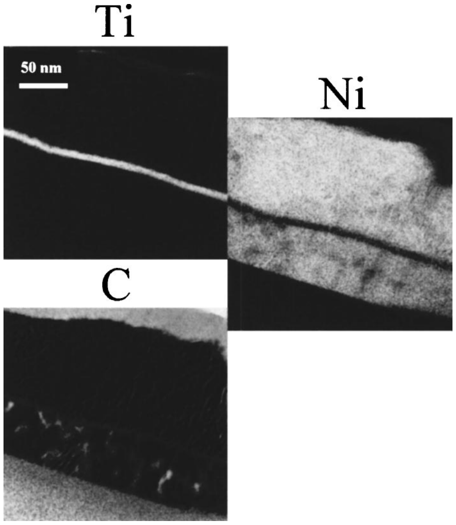

FIG. 4. Ti, Ni, and C maps determined by EFTEM analysis of the sample annealed at 500 °C.

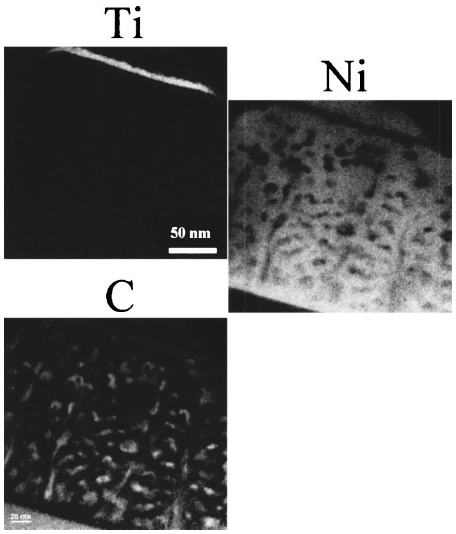

FIG. 5. Ti, Ni, and C maps determined by EFTEM analysis of the sample annealed at 550 °C.

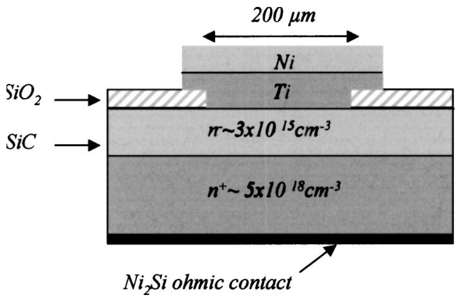

FIG. 6. Schematic of the Schottky diodes used to monitor the electrical properties of the contacts.

the metallic species of the bilayer mobile, i.e., interdiffusion of Ni and Ti occurs. Then Ni reaches the interface with SiC and reacts by forming the two silicides phases, as previously pointed out by discussing the XRD spectra. Indeed, the maps of the sample annealed at 500 °C show the presence of the thin Ti layer embedded in the thick Ni layer. The thickness of this Ti layer is nearly similar to that obtained after deposition. Near the interface with SiC, Ni has reacted by forming silicides, while at the sample surface no reaction occurred. The silicidation of the Ni layer in the proximity of the interface with SiC can be deduced by the maps of carbon, which was liberated during reaction and is now present in this layer (see Fig. 4). In particular, from the carbon map, it can be seen that carbon has formed precipitates inside the silicide layer, as it usually occurs in the case of the simple Ni/SiC system.[17–19] The formation of carbon precipitates when a SiC layer is consumed in the silicidation process of nickel is a consequence of the instability of the ternary phases Ni-Si-C as can be seen from the phase diagram reported in Ref. 2.

After annealing at 550 °C, almost all the Ni film has reacted, forming a silicide layer with a uniform distribution of carbon precipitates, while the Ti layer is now located on the top of the metallic contact, as visible from the EFTEM shown in Fig. 5. No substantial changes were observed in the chemical maps of the samples annealed above this temperature.

B. Electrical characterization

The structural changes of the Ni/Ti/SiC system observed after annealing affect the electrical properties, i.e., of Schottky barrier height. For that reason, the electrical characteristics of Schottky diodes were analyzed. In Fig. 6 schematic of the diode structure is reported. The active area of the diode was defined by the circular windows (whose diameter is about 200 μm) opened onto the oxide and the Schottky contact was formed by the bilayer Ni/Ti. The presence of the oxide, in turn, avoided the formation of a Ni ring at the diode edge, caused by the Ti overetch in the sequential Ni-Ti wet etch, as described in Ref. 8, which can give additional perimeter effects in the electrical characteristics of the contacts.

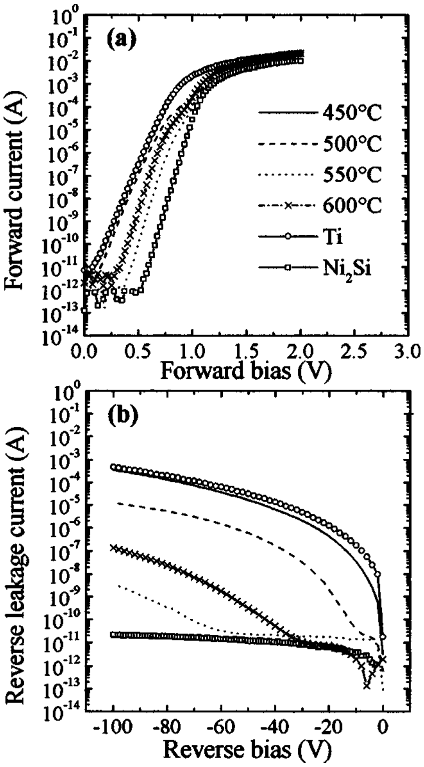

FIG. 7. Current voltage (*I-V*) electrical characteristics of Ni/Ti/6H-SiC Schottky diodes under (a) forward and (b) reverse bias, after deposition and after annealing between 450 and 600 °C. For comparison, the *I-V* characteristics of Ti/6H-SiC and Ni_2Si/6H-SiC diodes are also reported.

Figures 7(a) and 7(b) show the forward and reverse *I-V* characteristics of the Ni/Ti/6H-SiC Schottky diodes, annealed at various temperatures. In the same graph, the electrical characteristics of reference Ti/6H-SiC and Ni_2Si/6H-SiC (formed by a rapid thermal annealing of Ni/6H-SiC at 600 °C) Schottky diodes are shown. As can be seen, after annealing at 450 °C the forward and reverse characteristics of the Ni/Ti/6H-SiC Schottky diodes are almost the same as those of a Ti/6H-SiC contact prepared in the same annealing conditions. The latter indicates that the onset temperature for interdiffusion and/or reaction of the metallic species was not reached, i.e., the Schottky barrier is uniquely determined by the presence of titanium at the interface with SiC. Indeed, the value of the Schottky barrier height, determined by fitting the *I-V* curves in the linear region by means of the thermionic emission theory,[20] was 0.91 eV, i.e., a typical value of the barrier height of the Ti/6H-SiC contact. By increasing the annealing temperature (500–550 °C) the diodes exhibit an "anomalous" forward characteristics, i.e., they show the presence of a shoulder at low forward bias values instead of a wide linear region. In particular, after annealing at 500 °C, at lower bias (below 0.5 V) the curves are still close to those of the as-deposited diode, while at high biases (above 0.5 V) the presence of a second (higher) barrier can be observed. This behavior is typical of an "inhomogeneous" Schottky barrier at the metal/SiC interface, as similarly reported in the case of annealed Ti/4H-SiC contacts.[4] At 550 °C the forward *I-V* characteristics moves towards those of a Ni_2Si contact, whose barrier height on 6H-SiC is 1.39 eV.[18] The reverse leakage current I_L of this inhomogeneous Schottky contact decreased about four orders of magnitude by annealing from 450 °C ($I_L=3.6\times10^{-4}$ A at −100 V) to 550 °C ($I_L=3.5\times10^{-9}$ A at −100 V). At 550 °C the reverse characteristic at low bias, up to −60 V, is almost coincident with that measured in Ni_2Si

diodes. Finally, after annealing at 600 °C the *I-V* curves exhibited again a decrease of the average barrier height, accompanied by an increase of the leakage current.

IV. DISCUSSION

On the basis of the experimental results a possible scenario of the reaction mechanism of the Ni/Ti/6H-SiC bilayer system is the following. According to thermodynamic considerations concerning the ternary phase diagrams and the enthalpy changes during reactions,[2,15,21,22] Ni and Ti can react with SiC by forming silicides (i.e., $Ni_{31}Si_{12}$, Ni_2Si, Ti_5Si_3, etc.), carbides (TiC), or ternary phases (Ti_3SiC_2). In particular, Ni reacts with SiC above 450 °C by forming nickel silicides ($Ni_{31}Si_{12}$ and Ni_2Si), while the carbon liberated during reaction form precipitates.[22] On the other hand, above 500–600 °C Ti can form the metal rich silicide (Ti_5Si_3) and TiC. These two phases can coexist and, under certain annealing conditions, can even arrange in a two-layer structure.[2,15] At higher temperatures (900–1000 °C), Ti can also form the ternary phase Ti_3SiC_2.[2]

In the case of the Ni/Ti/SiC system, since Ti reacts with SiC only at higher temperatures (forming silicides and/or carbides[2,15]), below 450 °C no substantial structural change of the system occurs. Moreover, the stack sequence of the metallic layers remains the same like in the as-deposited sample, thus indicating that this thermal budget is even not sufficient to activate the diffusion of Ni or Ti atoms. Above this onset temperature, interdiffusion of the two elements in the bilayer occurs, with Ni diffusing downwards and Ti upwards. In this way, the Ni atoms can reach the interface with SiC and react by forming silicides ($Ni_{31}Si_{12}$ and Ni_2Si), as observed by XRD analyses at 500 °C.

Presumably, in the early stages of the annealing process, when the metallic species become mobile, a condition in which both Ti and Ni are present at the interface with SiC occurs. Therefore, an “inhomogeneous” Schottky barrier (Ti-$Ni_{31}Si_{12}$-Ni_2Si/SiC) forms, as confirmed by the anomalous forward *I-V* characteristics of the diodes observed at 500 °C. In fact, as pointed out by Ohdomari, Kuan, and Tu,[23] it must be remembered that even a few percent fraction of the whole contact area covered by a low Schottky barrier (Ti in our case) is sufficient to rule the conduction through the contact. By further increasing the processing temperature, as indicated by EFTEM, a further indiffusion of Ni through the Ti film and towards the interface with SiC (and/or a further outdiffusion of Ti through the Ni film and towards the surface) occurs. At the same time XRD analysis shows that the nickel silicide phases gradually transform into the most stable one (Ni_2Si), with an increase of the Schottky barrier. The final situation showed by EFTEM is that of a Ti thin film at the sample surface and a thicker silicide layer (with carbon precipitates inside) at the interface with SiC.

At around 550 °C the transformation of the $Ni_{31}Si_{12}$ phase into the Ni_2Si phase is also accompanied by the appearance of the Ni_2Si (133) signal in the XRD spectra, thus confirming that, at these temperatures, a structural evolution of the polycrystalline Ni_2Si is occurring. Obviously, these structural changes of the system in the near interface region affect the electrical properties of the barrier. Hence, it is not surprising that the structural evolution of this inhomogeneous silicide contact, by further annealing at 600 °C, gives rise again to a decrease of the Schottky barrier and to an increased leakage current, as experimentally observed by the *I-V* curves.

However, previous experimental results on the electrical characteristics of the Ni/SiC system after rapid thermal treatments,[18,24,25] showed that the silicidation mechanism of Ni results into an improvement of the barrier homogeneity and into an increase of the Schottky barrier accompanied by a decrease of the leakage current. Therefore, it can be argued that the different annealing conditions along with the presence of the Ti interlayer (which in turn acts as a barrier for Ni diffusion towards SiC) result into a different electrical behavior of the Ni/Ti/6H-SiC barrier upon annealing with respect to the Ni/6H-SiC. Hence, it can be concluded that in the case of the Ni/Ti/SiC system, by optimizing the film thickness and the annealing conditions, an inhomogeneous Schottky barrier (with both Ti or nickel silicide phases at the interface) may be intentionally formed in order to achieve a favorable compromise between the low forward voltage drop of a low barrier Ti/6H-SiC diode and the low leakage current values of a high barrier Ni_2Si/6H-SiC diode.

On the basis of the results presented in this paper, we believe that the reaction mechanism of the Ni/Ti/SiC system upon annealing must be taken into account also to explain the results on dual-metal Schottky diodes reported in Ref. 8, where similar Ni/Ti/SiC contacts where annealed in the same temperature range.

V. SUMMARY

In this work the evolution of the structural and electrical properties of the bilayer Ni/Ti/6H-SiC system at different annealing temperatures was studied. The experimental results allowed to find an onset temperature of about 450 °C above which the elements of the metallic bilayer become mobile, thus allowing the Ni atoms that reach the interface with SiC to react and form nickel silicides. The Schottky barrier height, determined by the electrical characteristics of test diodes, increased because of the silicidation of nickel with a consequent reduction of the leakage current. The inversion of this trend observed at high temperatures (600 °C) can be attributed to the inhomogeneity of the Schottky contact and to the structural changes occurring at the interfacial nickel silicide. This study provides some insights into the complicated reaction mechanism of the Ni/Ti/6H-SiC system. These results may be also useful to control and optimize the electrical properties of advanced Schottky diodes.

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