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Effects of Reducing Annealing Temperature on Ni/Al Ohmic Contacts to n- and p-Type 4H-SiC †

ITO Kazuhiro*, TAKEDA Hidehisa**, SHIRAI Yasuharu**, MURAKAMI Masanori***

Abstract

Effects of reducing annealing temperature on a technique for the simultaneous formation of Ni/Al ohmic contacts exhibiting ohmic behavior to both n- and p-type SiC were investigated. The Ni/Al contacts reacted with the SiC substrates at 900° C, leading to formation of δ -Ni₂Si(Al). There was no contact consisting of δ -Ni₂Si(Al) after annealing at 900° C for 20 min exhibited ohmic behavior to both n- and p-type SiC. Since Al concentration in the δ -Ni₂Si(Al) grains increased as the annealing temperature decreased due to less evaporation of the Al layer during annealing, and thus it is difficult to control the narrow range of Al concentration appropriate to exhibit ohmic behavior to both n- and p-type SiC using a conventional deposition technique. The specific contact resistances of the contacts which exhibited ohmic behavior to n-type SiC after annealing at 900° C for 20 min were higher than those after annealing at 1000° C for 5 min. In contrast, those which exhibited ohmic behavior to p-type SiC did not vary with annealing temperature. On the other hand, the specific contact resistance of the Ni(50 nm)/Al(10 nm) contact consisting of δ -Ni₂Si(8 at%Al) to p-type SiC was reduced to be about 4.0×10^{-4} 0 cm^2

KEY WORDS: (Ni/Al contacts), (SiC substrates), (Ohmic contacts), (δ -Ni₂Si(Al)), (Annealing temperature effects)

1. Introduction

Silicon carbide (SiC) is better suited to the next generation of high-powered devices due to such excellent intrinsic properties as a higher electric field breakdown strength and a higher saturation electron velocity than those of Si¹⁾. However, several technical issues such as doping at high levels by ion implantation and development of formation techniques to create low resistance ohmic contacts²⁾ must be solved before the application of SiC in high-power devices can be realized. For power metal oxide semiconductor field effect transistor devices with a double-implanted vertical structure, the ohmic contacts on both n+-source region and p-well region are needed3). Nickel4) and Ti/Al5-11) contacts, where a slash symbol "/" indicates the deposition sequence starting with Ti, are well known as the low resistance ohmic contact to n- and p-type SiC, respectively.

Thus, the ohmic contacts to n- and p-type SiC are currently fabricated using different contact materials and different annealing processes. Simultaneous formation of ohmic contacts to both n- and p-type SiC using the same contact material and in a one-step annealing process will simplify device fabrication processes and miniaturize the

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cell size. Although a variety of ohmic contact materials to SiC have been developed, only a few materials are suitable for ohmic contacts to both n- and p-type SiC $^{12\text{-}15)}$. Fursin et al. $^{12)}$ reported that a Ni contact showed ohmic behavior after annealing at 1050°C to both n- and p-type SiC in which N and Al were implanted at 10^{19} cm $^{-3}$ and 10^{20} cm $^{-3}$, respectively. Tanimoto et al. $^{13)}$ reported that simultaneous formation of Ni-based contacts to both n- and p-type heavily doped SiC (Np $> 10^{20}$ cm $^{-3}$ and Na $> 10^{20}$ cm $^{-3}$) after annealing at 1000°C . Ito et al. $^{14)}$ reported that Ni/Al contact showed ohmic behavior after annealing at 1000°C to both n- and p-type SiC in which N and Al were doped at 1.3×10^{19} cm $^{-3}$ and 7.2×10^{18} cm $^{-3}$, respectively. Tsukimoto et al. $^{15)}$ reported that a Ni/Ti/Al ternary contact showed ohmic behavior after annealing at 800°C to both n- and p-type SiC in which N and Al were doped at 10^{19} cm $^{-3}$ and 4.5×10^{18} cm $^{-3}$, respectively.

The mechanisms responsible for the simultaneous formation of ohmic contacts to both n- and p-type SiC using the same material must be different from the contact materials. Ito et al. proposed that a fine microstructure and a large distribution in Al concentration in $\delta\textsc{-Ni}_2\textsc{Si}(Al)$ grains were found to be characteristic features of ohmic contacts to both n- and

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p-type SiC, and that the δ-Ni₂Si(Al) grains with low Al concentration and high Al concentration exhibited ohmic behavior to n-type SiC and p-type SiC, respectively¹⁴. Such a characteristic feature must be controlled by the initial Al-layer thickness and annealing temperature, and thus in this study we investigated effects of reducing annealing temperature on the simultaneous formation of ohmic contacts to both n- and p-type SiC. We investigated the electrical properties of Ni/Al contacts with various Al-layer thicknesses after annealing at 900°C for 20 min. Investigations of the microstructures at the contact/SiC interface were conducted using X-ray diffraction (XRD), cross-sectional transmission electron microscope (TEM) and energy dispersive X-ray (EDX) techniques.

2. Experimental

The n- and p-type 4H-SiC epitaxial layers, doped with N at 1.3×10^{19} cm⁻³ and Al at 7.2×10^{18} cm⁻³, were grown on 2-inch 4H-SiC wafers manufactured by Cree, Inc. (Durham, NC). The SiC substrates had 8° -off Si-terminated (0001) surfaces inclined toward the $<\overline{2}110>$ direction. Both the 4H-SiC wafers were diced into small square pieces of dimensions about 8mm × 8mm. The square 4H-SiC substrates were used for measurements of electrical properties. For current-voltage (I-V) and specific contact resistance measurements, circular electrode patterns on the substrates were prepared using a photolithographic technique to remove portions of the thermally grown SiO_x layers. The substrates were cleaned by dipping in diluted hydrofluoric acid solution and rinsing in deionized water prior to metal depositions. Ni and Al layers were deposited sequentially by evaporation in a high vacuum below 1.5×10^{-4} Pa (the base pressure prior to deposition was approximately 4.0×10^{-6} Pa). Thicknesses of the Ni and Al layers were controlled by crystalline film thickness meter, and those were 50 nm and 0-10 nm, respectively. After lifting off the photoresist, the samples were annealed at 900°C for 20 min in an ultra-high vacuum (UHV) chamber where the vacuum pressure was below 4.0×10^{-5} Pa (the base pressure prior to annealing was approximately 7.0×10^{-8} Pa). The UHV annealing was carried out to prevent oxidation of the Al top layer. The samples were heated by infrared-ray radiation from an electrically heated Pyrolytic Graphite heater.

The electrical property, microstructure and composition analyses were conducted using the annealed Ni/Al contacts. The I-V characteristics of Ni/Al contacts were measured by a two-point probe method using circular patterns with an interspacing of 8 μm . The specific contact resistances were measured using a circular transmission line model (TLM) four-point probe method 16 . The interspacings in the contacts employed for the circular TLM measurements were 4, 8, 16 and 24 μm . The contact microstructures were analyzed by XRD and TEM. The Al/Ni ratio of the Ni/Al contacts was measured by EDX in a scanning electron microscope (SEM).

3. Results and Discussion

3.1 Formation of δ -Ni₂Si(Al) in Ni/Al contacts after annealing

Figure 1 shows portions of XRD spectra of Ni/Al contacts with Ni-layer thickness of 50 nm and Al-layer thicknesses of 0, 2, 4, 6, and 10 nm (Hereafter, these contacts are called Ni(50nm)/Al(0nm), Ni(50nm)/ Al(2nm), Ni(50nm)/Al(4nm), Ni(50nm)/Al(6nm), and Ni(50nm)/Al(10nm), respectively) to n-type SiC after annealing at 900°C for 20 min, together with a portion of the XRD spectrum of the Ni(50nm)/Al(0nm) contact after annealing at 1000°C for 5 min¹⁴⁾ for comparison. Four broad peaks were observed in all the contacts. These peaks were in good agreement with a diffraction pattern of δ-Ni₂Si(Al). This indicates that the Ni/Al contacts reacted with the SiC substrates at 900°C, leading to formation of δ -Ni₂Si(Al). This is the same as the interface reaction after annealing at 1000°C for 5 min. The highest (013) peaks showed that a large number of the δ-Ni₂Si(Al) grains were grown along the [013] orientation. The (013) peak intensities after annealing at 900°C for 20 min were lower than those after annealing at 1000°C for 5 min in all Al-layer thicknesses, indicating that decreasing annealing temperature increased (013) scattering and/or formed lattice-space microstructure. While, peak intensity of the four peaks increased and especially the (013)-peak angle shifted to a low diffraction angle with increasing Al-layer thickness for annealing at 900°C and 1000°C, and the four peaks

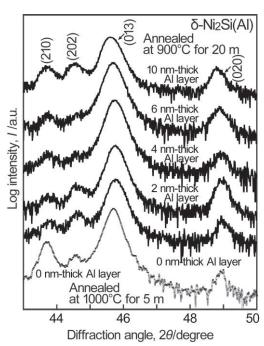


Fig. 1 Portions of XRD spectra of Ni(50nm)/Al(0nm), Ni(50nm)/Al(2nm), Ni(50nm)/Al(4nm), Ni(50nm)/Al(6nm), and Ni(50nm)/Al(10nm) contacts to n-type SiC after annealing at 900°C for 20 min, together with a portion of XRD spectrum of the Ni(50nm)/Al(0nm) contact after annealing at 1000°C for 5 min for comparison.

became sharp in the Ni(50 nm)/Al(10 nm) contact after annealing at 900°C for 20 min. This indicates that increase of Al concentration in δ -Ni₂Si(Al).grains contributes to form a better crystalline δ -Ni₂Si(Al).grain.

Some of Al layers were evaporated during annealing in UHV at 900°C and thus the Al(at.%)/Ni(at.%) ratios through the entire thickness of the annealed Ni/Al contacts were investigated using the SEM/EDS equipment. The Al/Ni ratios of the contacts using SEM/EDX were corrected based on the ratio of the Laser ablation inductively coupled plasma-mass spectrometry (LA-ICPMS) result (0.058) to the SEM/EDX result (0.042) for the Ni(50 nm)/Al(8 nm) contact after annealing at 1000°C for 5 min¹⁴). The corrected Al/Ni ratio of Ni/Al contacts with various Al-layer thicknesses after annealing at 900°C for 20 min is represented using white circles in Fig. 2, together with those after annealing at 1000°C for 5 min using black triangles for comparison. The Al/Ni ratio increased linearly with increasing the Al-layer thickness. The Al atoms were detected in only δ-Ni₂Si grains using TEM/EDX techniques in the previous study¹⁴⁾, and thus the average Al concentrations of the δ -Ni₂Si(Al) grains could be calculated based on the Al/Ni ratios through the entire thickness of the Ni/Al contacts (Fig. 2). Approximate Al concentrations of the δ-Ni₂Si(Al) grains after annealing at 900°C for 20 min were estimated in the range of 0 to 8 at.% (Al-layer thicknesses of 0-10 nm). The Al concentration of the δ-Ni₂Si(Al) grains after annealing at 900°C for 20 min was higher than that after annealing at 1000°C for 5 min in the investigated range of the Al-layer thickness, suggesting that decreasing annealing temperature increases Al concentration in the δ -Ni₂Si(Al) grains.

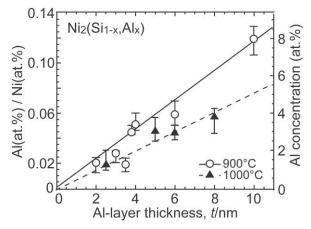


Fig. 2 Al(at.%)/Ni(at.%) ratios through the entire thickness of the Ni/Al contacts annealed at 900°C for 20 min with various Al-layer thicknesses (\circ), together with those annealed at 1000°C for 5 min°C (\blacktriangle) for comparison. The Al/Ni ratios obtained by SEM/EDX were recalculated based on LA-ICPMS results. The right axis shows approximate Al concentrations of the δ -Ni₂Si(Al) grains calculated based on the Al(at.%)/Ni(at.%) ratios through the entire thickness of the Ni/Al contacts.

3.2 Effects of decreasing annealing temperature on Ni/Al-contact resistance

The I-V characteristics of the Ni/Al contacts to nand p-type SiC after annealing at 900°C for 20 min are shown in Figs. 3(a) and 3(b), respectively. For n-type SiC (Fig. 3(a)), the contacts consisting of δ -Ni₂Si(Al) in the Al-concentration range of 0 to 1.8 at.% showed ohmic behavior, while the contact consisting of δ-Ni₂Si(Al) in the Al-concentration range of 3.0 to 8.4 at.% showed rectifying (nonohmic) behavior. For p-type SiC (Fig. 3(b)), the contacts consisting of δ-Ni₂Si(Al) in the Al-concentration range of 1.3 to 2.9 at.% showed nonohmic behavior, while the contacts consisting of δ-Ni₂Si(Al) in the Al-concentration range of 4.0 to 7.6 at.% showed ohmic behavior. The contact property of the Ni/Al contacts after annealing at 900°C for 20 min is given in Fig. 3(c), together with that after annealing at 1000°C for 5 min for comparison. As the Al concentration in δ-Ni₂Si(Al) was increased, ohmic contacts were less likely to form on n-type SiC and more likely to form on p-type SiC. There was no contact consisting of δ-Ni₂Si(Al) after annealing at 900°C for 20 min exhibited ohmic behavior to both n- and p-type SiC in contrast to the contacts consisting of δ-Ni₂Si(3.0 at.%Al) after annealing at 1000°C for 5 min which exhibited ohmic behavior to both n- and p-type SiC. With decreasing annealing temperature, the evaporation during annealing in UHV decreased and thus the appropriate range in the Al-layer thickness to exhibit ohmic behavior to both n- and p-type SiC became narrow. This suggests that it is difficult to control the appropriate range in a conventional deposition technique.

Specific contact resistances of the Ni/Al contacts consisting of δ-Ni₂Si(Al) to n- and p-type SiC after annealing at 900°C for 20 min are shown using white circles in Figs. 4(a) and 4(b), respectively, together with those after annealing at 1000°C for 5 min which are shown using black triangles for comparison. With increasing Al concentration in δ-Ni₂Si(Al), the specific contact resistances to n-type SiC increased and those to p-type SiC decreased in both cases after annealing at 900°C for 20 min and 1000°C for 5 min. The specific contact resistances of the contacts which exhibited ohmic behavior to n-type SiC after annealing at 900°C for 20 min were higher than those after annealing at 1000°C for 5 min. Especially, the specific contact resistance of Ni(50 nm)/Al(0 nm) contacts after annealing at 900°C for 20 min $(1.3 \times 10^{-4} \ \Omega \text{cm}^2)$ were higher by two orders of magnitude than that after annealing at 1000°C for 5 min $(3.2 \times 10^{-6} \,\Omega \text{cm}^2)$. This could be explained especially by integration of better crystalline (013) plane of the δ-Ni₂Si(Al) grains (due to high (013) XRD peak intensity). In contrast, the specific contact resistances of the contacts which exhibited ohmic behavior to p-type SiC did not vary with annealing temperature. On the other hand, decreasing annealing temperature to 900°C could increase Al concentration in δ-Ni₂Si(Al) grains, and the specific contact resistance of the Ni(50 nm)/Al(10 nm) contact consisting of δ-Ni₂Si(8 at%Al) to p-type SiC

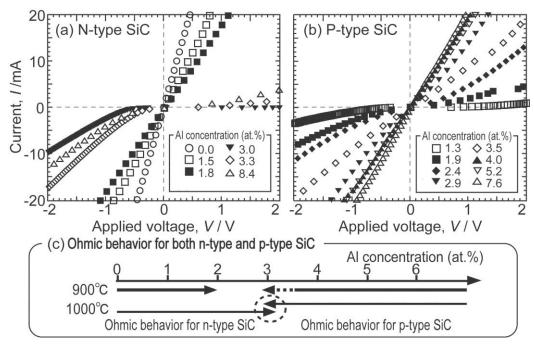


Fig. 3 Current-voltage (I-V) characteristics of Ni(50nm)/Al(0-10nm) contacts to (a) n- and (b) p-type SiC after annealing at 900°C for 20 min with various Al concentrations of the δ -Ni₂Si(Al) grains, which the Ni/Al contacts consisted of. (c) Schematic illustration of Al concentration dependence of ohmic behavior to n- and p-type SiC for the δ -Ni₂Si(Al) grains obtained after annealing at 900°C for 20 min, together with those after annealing at 1000°C for 5 min for comparison.

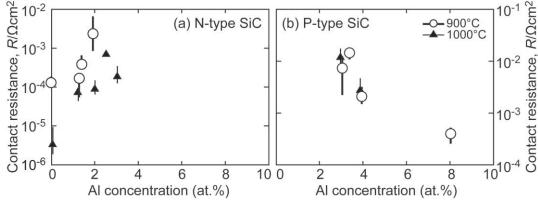


Fig. 4 Contact resistance of Ni(50nm)/Al(0-10nm) contacts to (a) n- and (b) p-type SiC after annealing at 900°C for 20 min with various Al concentrations of the δ -Ni₂Si(Al) grains (\circ), which the Ni/Al contacts consisted of, together with those after annealing at 1000°C for 5 min (\blacktriangle) for comparison.

was reduced to be about $4.0 \times 10^{-4} \ \Omega \text{cm}^2$, which is similar level to that of the Ni/Al contact to n-type SiC $(1.3 \times 10^{-4} \ \Omega \text{cm}^2)$

Decreasing annealing temperature to 900°C increased Al concentration in the δ -Ni₂Si(Al) grains. The XRD spectra of the Ni/Al contacts consisting of δ -Ni₂Si(8 at.%Al) had sharp four peaks. Especially, the XRD peak intensity for the (013) peak became the largest in the investigated Al concentration in the δ -Ni₂Si(Al) grains, and the (013) diffraction angle shifted to a low diffraction angle compared with δ -Ni₂Si(Al) with Al concentrations lower than 8 at.%. The (013) plane of the δ -Ni₂Si(Al) had an asymmetrical hexagonal shape as shown in **Fig. 5**, and its area expands with increasing Al concentration in the δ -Ni₂Si(Al) grains based on variation of the lattice

parameters of a, b, and c with Al concentration in the $\delta\text{-Ni}_2\mathrm{Si}(Al)$ grain¹⁷⁾ (Fig. 5). Thus, Al-concentration increase in the $\delta\text{-Ni}_2\mathrm{Si}(Al)$ grain reduce lattice mismatch between the (013) plane in the $\delta\text{-Ni}_2\mathrm{Si}(Al)$ grain and the (0001) plane in the SiC substrate. This could play a key role in decreasing the contact resistance.

3.3 Effects of decreasing annealing temperature on Ni/Al-contact microstructure

Figure 6 shows cross-sectional TEM bright field images of a Ni(50nm)/Al(4nm) contact and a Ni(50 nm)/Al(5 nm) contact to n-type SiC after annealing at 900°C for 20 min and after annealing at 1000°C for 5 min, respectively. An electron incident beam was almost parallel to the <\bar{2}110> orientation of the SiC substrate.

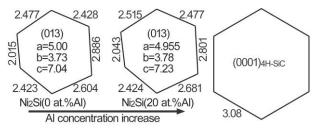


Fig. 5 Schematic illustrations of a (013) plane of the δ -Ni₂Si(0 at.%Al) and δ -Ni₂Si(20 at.%Al) grains calculated based on lattice constants variation with Al concentration in the δ -Ni₂Si(Al)^{ref.11}, together with that of a (0001) plane of 4H-SiC for comparison.

An about 100 nm-thick contact layer was uniformly formed on the SiC substrate after annealing at 900°C for 20 min and at 1000°C for 5 min. Thickness of the contact after annealing at 900°C for 20 min was thicker than that after annealing at 1000°C for 5 min due to less Al-layer evaporation during annealing. Also thickness oscillation of the Ni/Al contact layer formed after annealing at 900°C for 20 min was larger than that after annealing at 1000°C for 5 min. Based on selected area diffraction (SAD) analysis, polycrystalline δ-Ni₂Si(Al) grains were mostly observed for both samples after annealing at 900°C for 20 min and at 1000°C for 5 min. Also grains with white contrast (marked with arrows) were carbon based on its amorphous nature. Only δ-Ni₂Si(Al) grains were found to form at the contact/SiC interface and thus, the δ-Ni₂Si(Al) grains should play a key role in determining electrical transport properties at the contact/SiC interface. The δ-Ni₂Si grain was found to have grown along [013] orientation on the (0001) plane and the δ-Ni₂Si/SiC interface was found to be flat in HRTEM in previous study¹⁴⁾. Similarly, almost polycrystalline δ-Ni₂Si grains were found to have grown

along [013] orientation on the (0001) plane of the SiC substrate. Similar microstructures were observed in the Ni/Al contacts consisting of δ -Ni₂Si(Al) with other Al concentrations formed in both the annealing conditions.

study¹⁴⁾, that a fine in previous microstructure and a large distribution in Al concentration in δ-Ni₂Si(Al) grains were found to be characteristic features of ohmic contacts to both n- and p-type SiC, and that the fine microstructure was essential for the large distribution in Al concentration in δ -Ni₂Si(Al) grains. Thus, average δ -Ni₂Si(Al) grain sizes at the contact/SiC interface in the Ni(50nm)/Al(2nm) and Ni(50nm)/Al(4nm) contacts after annealing at 900°C for 20 min were investigated, and the results are shown in Fig. 7. The average δ -Ni₂Si(Al) grain size in the Ni/Al contacts after annealing at 900°C for 20 min is shown using white circles, together with those in the Ni(50nm)/Al(2nm), Ni(50nm)/Al(6nm), and Ni(50nm)/ Al(10nm) contacts to n-type SiC after annealing at 1000°C for 5 min using black triangles for comparison. Large average grain sizes of about 50 nm and 70 nm were found to form at the contact/SiC interface in the Ni/Al contacts after annealing at 900°C for 20 min with δ -Ni₂Si(1.3 at.%Al) and with δ -Ni₂Si(3.3 at.%Al), respectively. The former and latter distributions in the average δ -Ni₂Si(Al) grain size were similar to those in the Ni(50 nm)/Al(2 nm) contacts consisting of δ-Ni₂Si(0.9 at.%Al) and the Ni(50 nm)/Al(10 nm) contacts consisting of δ-Ni₂Si(4.5 at.%Al), respectively, formed after annealing at 1000°C for 5 min. A fine microstructure and a large distribution in average grain size were not observed in both the observed samples after annealing at 900°C for 20 min, and those might be existed in Al-layer thicknesses between 2 nm and 4 nm, and also in average Al concentrations between 1.3 and 3.3 at.% in δ-Ni₂Si(Al) grains. This suggests that decreasing

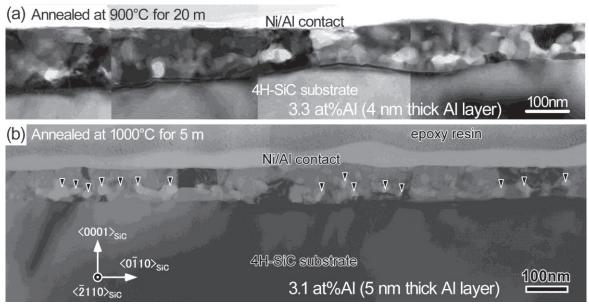


Fig. 6 A cross-sectional TEM bright field image of a Ni(50nm)/Al(4nm) contact to n-type SiC after annealing at 900°C for 20 min, together with that of a Ni(50nm)/Al(5nm) contact to n-type SiC after annealing at 1000°C for 5 min for comparison.

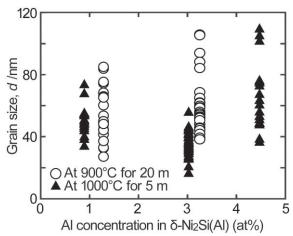


Fig. 7 The δ -Ni₂Si(Al) grain sizes at the contact/SiC interface in the Ni(50nm)/Al(2nm) and Ni(50nm)/Al(4nm) contacts to n-type SiC after annealing at 900°C for 20 min (\circ), together with those in the Ni(50nm)/Al(2nm), Ni(50nm)/Al(6nm), and Ni(50nm)/Al(10nm) contacts to n-type SiC after annealing at 1000°C for 5 min (\blacktriangle) for comparison.

annealing temperature reduces the range of both the Al-layer thickness and the average Al concentration in $\delta\text{-Ni}_2Si(Al)$ grains for exhibiting ohmic contacts to both n- and p-type SiC.

4. Conclusions

Effects of reducing annealing temperature for a technique of the simultaneous formation of Ni/Al ohmic contacts exhibiting ohmic behavior to both n- and p-type SiC were investigated. The Ni/Al contacts reacted with the SiC substrates at 900°C, leading to formation of δ-Ni₂Si(Al). The Al concentration of the δ-Ni₂Si(Al) grains was higher than that after annealing at 1000°C for 5 min in the investigated range of the Al-layer thickness, suggesting that decreasing annealing temperature increases Al concentration in the δ-Ni₂Si(Al) grains. As the Al concentration in δ-Ni₂Si(Al) was increased, ohmic contacts were less likely to form on n-type SiC and more likely to form on p-type SiC. There was no contact consisting of δ-Ni₂Si(Al) after annealing at 900°C for 20 min exhibited ohmic behavior to both n- and p-type SiC. Since Al concentration in the δ -Ni₂Si(Al) grains increased as the annealing temperature decreased due to less evaporation of the Al layer during annealing, and thus it is difficult to control the narrow range of Al concentration appropriate to exhibit ohmic behavior to both n- and p-type SiC using a conventional deposition technique. The specific contact resistances of the contacts which exhibited ohmic behavior to n-type SiC after annealing at 900°C for 20 min were higher than those after annealing at 1000°C for 5 min. In contrast, the specific contact resistances of the contacts which exhibited ohmic behavior to p-type SiC did not vary with annealing temperature. On the other hand, decreasing annealing temperature to 900°C could increase Al concentration in δ-Ni₂Si(Al) grains, and the specific

contact resistance of the Ni(50 nm)/Al(10 nm) contact consisting of δ-Ni₂Si(8 at%Al) to p-type SiC was reduced to be about $4.0 \times 10^{-4} \ \Omega \text{cm}^2$. Only δ -Ni₂Si(Al) grains were found to form at the contact/SiC interface. A fine microstructure and a large distribution in average δ-Ni₂Si(Al) grain size at the interface are essential for the simultaneous formation of Ni/Al ohmic contacts exhibiting ohmic behavior to both n- and p-type SiC. After annealing at 900°C for 20 min, such the microstructure was not observed, and those might be existed in Al-layer thicknesses between 2 nm and 4 nm, and also in average Al concentrations between 1.3 and 3.3 at.% in δ-Ni₂Si(Al) grains. Those suggest that decreasing annealing temperature reduce the range of both the Al-layer thickness and the average Al concentration in δ-Ni₂Si(Al) grains for exhibiting ohmic contacts to both n- and p-type SiC.

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