Materials Science Forum
ISSN: 1662-9752, Vol. 1159, pp 31-36
doi:10.4028/p-NWvo3B

Submitted: 2024-09-20
Revised: 2025-02-07
Accepted: 2025-02-20
Online: 2025-09-09

Argon Plasma Treatment of 4H-SiC Surface before Nickel Ohmic Contacts Formation by UV Laser Annealing

Clément Berger[1,a], Daniel Alquier[1,b] and Jean-François Michaud[1,c *]

[1]GREMAN UMR-CNRS 7347, Université de Tours, INSA Centre Val de Loire, 16 rue Pierre et Marie Curie, 37071 TOURS Cedex 2, France

[a]clement.berger@univ-tours.fr, [b]daniel.alquier@univ-tours.fr, [c]jean-francois.michaud@univ-tours.fr

Keywords: Surface preparation, Ohmic contact, Laser annealing, 4H-SiC

Abstract. Laser Thermal Annealing (LTA) is a necessary fabrication step to improve the 4H-SiC devices by reducing their ON-state resistance. Because the LTA annealing is achieved at the end of the front-end fabrication, the classical Radio Corporation of America cleaning (RCA) cannot be used without affecting the material deposited on the frontside. Therefore, in this study, we investigate the argon (Ar) plasma surface treatment, achieved in our sputtering tool, before the ohmic contact fabrication, as an alternative surface preparation to the RCA sequence. As the Ar plasma modifies the SiC surface morphology, it affects its wetting properties. That can play a key role in the ohmic contact formation by LTA since the nickel turns into liquid phase during the laser irradiation. For an Ar plasma treatment of 30 min, a specific contact resistance of 5.0×10^{-5} $\Omega.cm^2$ has been obtained for an annealing at 5.0 $J.cm^{-2}$, which is in the same range than the contact fabricated by LTA involving a classical RCA cleaning.

Introduction

Silicon carbide (SiC) is a wide bandgap semiconductor, with superior physical and chemical properties allowing the fabrication of devices able to work at high voltage and high temperature [1]. To further improve devices, the fabrication of 4H-SiC vertical devices includes a grinding step of the substrate backside (C-face) reducing the device ON resistance. This implies a modification of the process flow and of the ohmic contact fabrication [2]. In this case, since the ohmic contact is performed at the end of the process flow, Rapid Thermal Annealing (RTA) cannot be used anymore without affecting the frontside materials. Then, to preserve the materials integrity, Laser Thermal Annealing (LTA) is currently used to achieve the backside ohmic contact. Several investigations were performed to study such contacts formed from titanium (Ti) or nickel (Ni) layers by LTA [3], [4], [5]. From the literature, nickel seems the most favorable metal to form efficient ohmic contact by LTA, with specific contact resistances in the 10^{-5} $\Omega.cm^2$ range. Based on our previous results on titanium and nickel, these contacts reached the ohmicity since they turn into their liquid phase. Therefore, the wetting properties of 4H-SiC C-face may play a role in the reaction between metal and SiC surface.

Generally, researches focused on the ohmic contacts involve a cleaning step with acid mixtures, like the Radio Corporation of America (RCA) sequence. Nevertheless, due to the elaboration of the backside ohmic contact at the end of the device fabrication, those chemical solutions can affect materials deposited on the wafer frontside. As a consequence, either a sacrificial layer has to be deposited on the front face before the cleaning sequence or a new surface preparation must be performed before the metal deposition, keeping the wafer frontside integrity.

In this study, we investigated the effect of a plasma treatment of the SiC surface before the nickel deposition though structural, morphological and electrical characterizations. Results will be compared with our best ohmic contact fabricated by LTA [6].

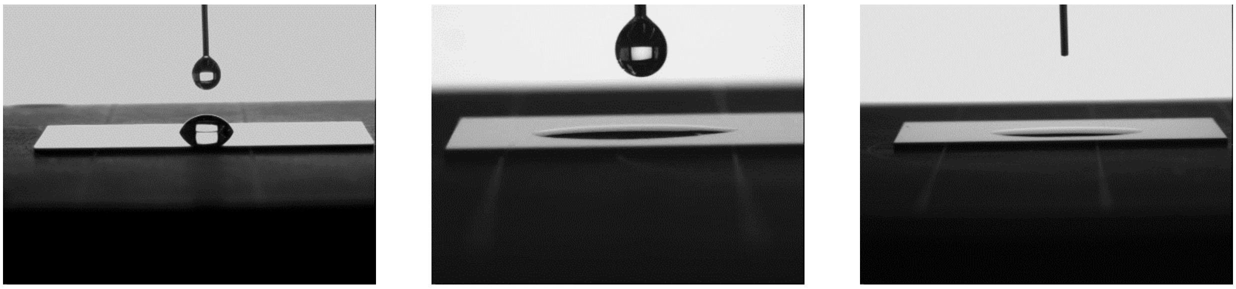
Experimental

For the physical characterizations, we fabricated the contacts on the C-face of 4H-SiC samples diced from bulk substrates, since C-face is classically used for the ohmic contact fabrication of the devices. Contrary to the classical ohmic contact achievement by RTA, we did not perform any acid cleaning such as Caro's or RCA sequence. Instead, we only dipped the sample in acetone to remove large particles on the surface. Then, samples were treated with an argon (Ar) plasma, generated in the chamber of the sputtering equipment (Plassys M650S) used for the metal deposition, for durations till 60 min. The chamber pressure was kept at 17 mTorr and the RF power was 100 W. In these conditions, the etching rate of the SiC substrate was estimated to 26 $nm.hr^{-1}$, by measuring a step with an Atomic Force Microscope (AFM). Consecutively to this Ar plasma processing, a 100 nm-thick nickel layer was deposited on the SiC at 5 mTorr under Ar. Finally, samples were irradiated with a tripled frequency YAG laser (355 nm) at a fluence of 5 $J.cm^{-2}$. The irradiations were performed at 30 kHz with a scanning speed of 800 $mm.s^{-1}$. Those irradiations conditions allow to form efficient nickel ohmic contact as presented in [6].

For the electrical characterizations, we used an isolated substrate configuration to prevent the current circulation through the substrate thickness that could affect the electrical performance determination [7]. Therefore, using the same protocol as described previously, contacts were fabricated on a highly nitrogen-doped epilayer (2 µm - 1×10^{18} $at.cm^{-3}$) grown onto a non-intentionally doped epilayer (10 µm thick - 5×10^{14} $at.cm^{-3}$) on the Si-face of a 4H-SiC substrate. After the irradiation of the unpatterned Ni layer, circular Transfer Length Method (cTLM) structures were fabricated by Ion Beam Etching (IBE). The measured real spacings vary from 14 to 50 µm with a constant inner electrode presenting a diameter of 205 µm. Finally, an aluminum layer was deposited on the cTLM patterns to thicken the contacts.

Results and Discussion

First, consecutively to the Ar plasma treatment, we evaluated the wetting properties of a deionized (DI) water drop on the treated SiC surface. To do so, we used the contact angle method on both SiC faces. The contact angle obtained for several SiC surface conditions are shown on the Figure 1 and on the Table 1. For the SiC only degreased in acetone, without plasma treatment, the water drop shape is almost spherical, resulting in a contact angle of 72° and 77°, respectively on the C- and Si-face, demonstrating a hydrophobic behavior. This measurement highlights the presence of a native oxide at the surface [8]. For the Ar plasma exposed samples, even for the smallest duration of 1 min, the drop spreading leads to an angle lower than 5° on both faces. As a comparison, we removed the native oxide by dipping a sample in hydrofluoric acid (HF) 1% during 5 min, rinsed with DI and dry under nitrogen flow, resulting in a contact angle of 20 and 24° on both C- and Si-face respectively. It is worth to mention that a high dispersion was observed with HF treatment because of erratic drop shape. Thus, we note that the plasma processing reaches to a higher SiC wettability than the oxide removal only. It is even clearer when the measurement is not performed directly after the treatment. Indeed, for the sample exposed to HF, after only 15 min, the contact angle significantly increases, probably due to the native oxide regrowth, whenever the 20 min Ar treated sample still shows a high wettability several hours after. Therefore, the plasma treatment leads to higher wetting properties than HF-treatment, even more at a long-time scale. This could be attributed to topological roughness changes that alter the native oxide regrowth. To investigate the origin of such changes, we analyzed the SiC surface at nanoscale by AFM measurement.



(a) (b) (c)

Fig. 1. Images of the contact angle of a water drop on SiC, with a camera tilt angle of 4°, (a) without treatment, (b) after a Ar plasma processing of 1 min and (c) 20 min.

Table 1. Measurements of contact angles of a water drop on SiC for several surface conditions. The measurements were performed immediatly, 15 min and 24 hours after the treatment. "-" symbol refers to condition not measured.

	Measured immediatly (C-face / Si-face)	After 15 min (C-face / Si-face)	After 4 hours (C-face / Si-face)	After 24 hours (C-face / Si-face)
Acetone cleaning	72° / 77°	-	-	-
HF(1%) – 5 min	20° / 24°	44° / 44°	50° / 32°	42° / 34°
Ar plasma – 1 min	<5° / <5°	<5° / <5°	12° / 16°	69° / 71°
Ar plasma – 20 min	<5° / <5°	<5° / <5°	17° / 18°	20° / 22°
Ar plasma – 30 min	10° / 10°	12° / 13°	26° / 29°	39° / 44°

The Figure 2 presents AFM characterizations of the 4H-SiC surface before and after different Ar plasma treatments. The surface of the shortest plasma treated sample is very similar to the one of the non-treated samples. Both present scratches, coming from the polishing stage. More, their roughness (Rq) value are identical and evaluated to 0.62 nm. Therefore, for this condition, the roughness and the surface morphology evolution do not seem to be responsible for the SiC wettability increase. The better wettability observed previously for this condition is then mostly attributed to the native oxide removal leaving a clean surface without the need of rinsing/drying that could bring some particles on the substrate. This might be why this sample behavior, 24 hours after the plasma exposure, was similar to the non-treated SiC due to the natural native oxide regrowth. After 10 min treatment, the minor scratches seem to be softened but the roughness was kept. From 20 min, the surface morphology strongly evolves by starting to present grains. The roughness slightly increases to 0.72 nm. Finally, for durations of 30 and 60 min, the grains become greater and greater, leading to a massive roughness increase for the longest plasma exposure (Rq = 2.76 nm). For long treatment duration, SiC surface is clearly affected by the Ar plasma, which might be responsible for the wettability modification observed. Then, it could influence the metal spreading during its melting under the high power laser annealing required to form ohmic contact.

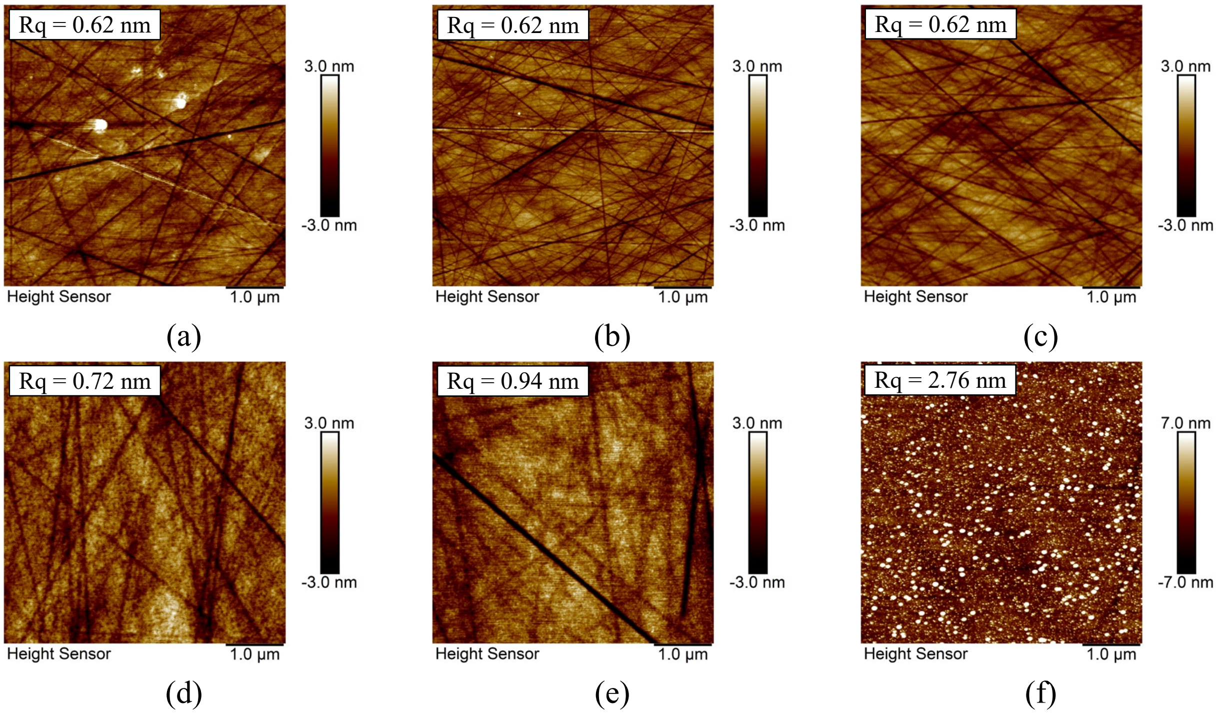


Fig. 2. AFM images of the 4H-SiC surface after an Ar plasma duration of (a) 0 min, (b) 1 min, (c) 10 min, (d) 20 min, (e) 30 min and (f) 60 min. The roughness (Rq) is noted for each condition.

Consecutively to the AFM measurements, a 100 nm-thick Ni layer was deposited on the treated SiC for several plasma durations. The Ni layer was then laser annealed at 5 $J.cm^{-2}$. Scanning Electron Microscope (SEM) images of contacts are presented on Figure 3. Without any surface treatment, the contact surface seems homogeneous, fully covering the SiC surface. However, a lot of nanoparticles are fully covering the contact after the irradiation (not visible at the magnification presented here). Then, surface morphology of the Ni contact evolves with the plasma duration. For a 20 min treatment, the gap between the irradiations is partially filled, suggesting a higher wetting of the liquid nickel onto the SiC. After 60 min of Ar plasma treatment, the contact morphology is the most regular among all the treated SiC, especially at the pulse center. Also, the nanoparticles density, covering the contact, is massively reduced compared to other conditions, highlighting a higher reaction between Ni and the substrate. In this case, the higher effective contact surface should be beneficial to reach better electrical performance and contact reliability.

Finally, electrical characterizations were performed on the Ni contacts by the means of TLM structures. The Figure 4(a) presents the current-voltage (I-V) characteristics for the contacts prepared on SiC exposed to an Ar plasma during 30 min. The I-V curves start to be linear from 4 $J.cm^{-2}$, highlighting the Schottky to ohmic transition. For the ohmic contacts prepared on the 30 min plasma treated SiC, the Specific Contact Resistance (SCR) was extracted for each fluence on the Figure 4(b). Reference contacts, presented in [6], fabricated with a classical cleaning, is also plotted. For the Ar plasma surface preparation sample, the SCR decreases with the increase of the fluence till 5 $J.cm^{-2}$ indicating an improvement of the ohmic properties.

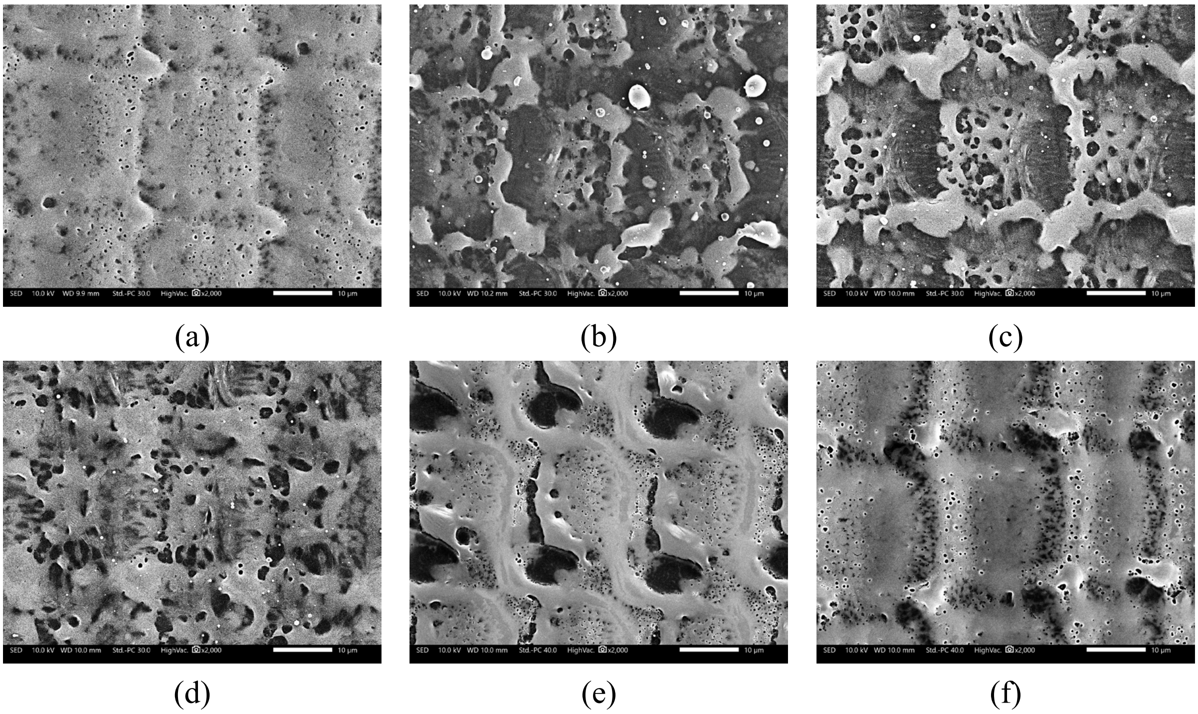


Fig. 3. SEM images of Ni(100 nm) contacts annealed at 5 J.cm^{-2} after a plasma treatment duration of (a) 0 min, (b) 1 min, (c) 10 min, (d) 20 min, (e) 30 min and (f) 60 min.

The optimal SCR value reaches $(5.0\pm2.0)\times10^{-5}$ Ω.cm^2, that is in the same range with the best reference value ($(2.4\pm1.1)\times10^{-5}$ Ω.cm^2). This result is hopeful considering that it is compatible with the device fabrication on thin substrate that prevents the use of chemical etching treatment before the metal deposition.

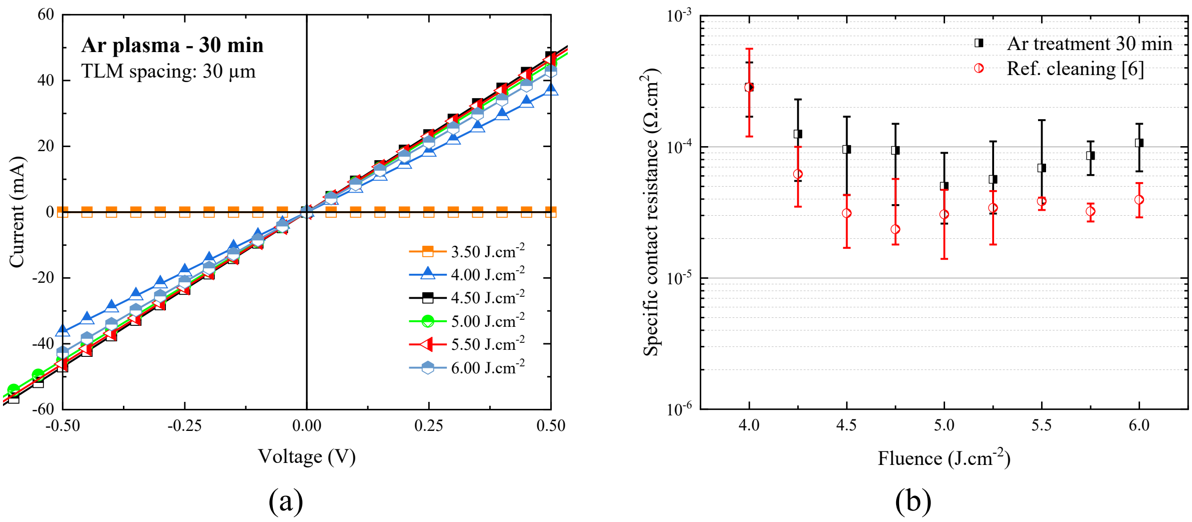


Fig. 4. (a) Current-Voltage characteristics of Ni contacts fabricated by LTA on a 30 min plasma exposed SiC surface. (b) Evolution of the specific contact resistance for the 30 min treated sample (black symbols) and a reference sample processed with a classical cleaning (red symbols) described in [6]. Error bars represent the min/max SCR value for each annealing condition.

Summary

The obtention of ohmic contact on thinned 4H-SiC substrate is currently achieved by laser irradiation, after the frontside fabrication. The classical acid cleaning could not be performed without affecting the materials located on the frontside. Therefore, we proposed to prepare the backside surface by using a plasma treatment before the metal deposition as an alternative to the wet cleaning. Surface characterizations demonstrated a modification of the SiC morphology, which might be responsible for its wetting properties modification. Therefore, when a nickel layer is annealed at high fluence, it reacts differently, after going through its liquid phase with SiC, depending on the Ar plasma treatment. Thanks to this plasma etching procedure, Ni ohmic contacts have been successfully fabricated with a 30 min treatment, presenting SCR values in the same range than the ones reached with a classical wet cleaning sequence. Samples with other plasma treatment durations are under electrical characterization.

Acknowledgements

The authors acknowledge the financial support provided by the "Agence Nationale de la Recherche" (ANR) through the project TempoScopy (ANR-19-CE42-0008).

References

[1] T. Kimoto and J. A. Cooper, "Fundamentals of Silicon Carbide Technology: growth, characterization, devices and applications", Wiley, Singapore, 2014.

[2] S. Rascunà, P. Badalà, C. Tringali, C. Bongiorno, E. Smecca, A. Alberti, S. Di Franco, F. Giannazzo, G. Greco, F. Roccaforte, M. Saggio, "Morphological and electrical properties of Nickel based Ohmic contacts formed by laser annealing process on n-type 4H-SiC", Mater. Sci. Semicond., vol. 97, p. 62, 2019, doi: 10.1016/j.mssp.2019.02.031.

[3] M. De Silva, T. Kawasaki, T. Miyazaki, T. Koganezawa, S. Yasuno and S.-I. Kuroki, "Formation of epitaxial Ti-Si-C Ohmic contact on 4H-SiC C face using pulsed-laser annealing", Appl. Phys. Lett., vol. 110, no. 25, p. 252108, 2017, doi: 10.1063/1.4987136.

[4] C. Berger, D. Alquier, M. Bah and J.-F. Michaud, "Electrical, morphological and structural properties of Ti ohmic contacts formed on n-type 4H–SiC by laser thermal annealing", Mater. Sci. Semicond., vol. 151, p. 106983, 2022, doi: 10.1016/j.mssp.2022.106983.

[5] P. Badalà, S. Rascunà, B. Cafra, A. Bassi, E. Smecca, M. Zimbone, C. Bongiorno, C. Calabretta, F. La Via, F. Roccaforte, M. Saggio, G. Franco, A. Messina, A. La Magna, A. Alberti, "Ni/4H-SiC interaction and silicide formation under excimer laser annealing for ohmic contact", Materialia, vol. 9, p. 100528, 2020, doi: 10.1016/j.mtla.2019.100528.

[6] J. F. Michaud, C. Berger and D. Alquier, "Nickel Ohmic Contacts Formed on 4H-SiC by UV Laser Annealing", Solid State Phenomena, vol. 359, p. 91, 2024, doi: 10.4028/p-xqh9xB.

[7] C. Berger, D. Alquier and J.-F. Michaud, "How to Accurately Determine the Ohmic Contact Properties on n-Type 4H-SiC", Electronics, vol. 13, no. 1, p. 217, 2024, doi: 10.3390/electronics13010217.

[8] J. G. Kim, W. S. Yoo, J. Y. Park and W. J. Lee, "Quantitative Analysis of Contact Angle of Water on SiC: Polytype and Polarity Dependence", ECS J. Solid State Sci. Technol., vol. 9, no. 12, p. 123006, 2020, doi: 10.1149/2162-8777/abcd0b.