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Development of an Extreme High Temperature n-type Ohmic Contact to Silicon Carbide

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Keywords: silicon carbide, ohmic contact, high temperature, tungsten, nickel

Abstract. We report on the initial demonstration of a tungsten-nickel (75:25 at. %) ohmic contact to silicon carbide (SiC) that performed for up to fifteen hours of heat treatment in argon at 1000 °C. The transfer length method (TLM) test structure was used to evaluate the contacts. Samples showed consistent ohmic behavior with specific contact resistance values averaging 5 x 10^{-4} Ω-cm^2. The development of this contact metallization should allow silicon carbide devices to operate more reliably at the present maximum operating temperature of 600 °C while potentially extending operations to 1000 °C.

Introduction

Silicon Carbide (SiC) is widely recognized as one of the materials of choice for high temperature, harsh environment sensors and electronics due to its ability to survive and continue normal operation in such environments [1]. Sensors and electronics in SiC have been developed that are capable of operating at temperatures of 600 ^{o}C. However operating these devices at the upper reliability temperature threshold increases the potential for early degradation. Therefore, it is important to raise the reliability temperature ceiling higher, which would assure increased device reliability when operated at nominal temperature. There are also instances that require devices to operate and survive for prolonged periods of time above 600 ^{o}C [2, 3]. This is specifically needed in the area of hypersonic flight where robust sensors are needed to monitor vehicle performance at temperature greater than 1000 °C, as well as for use in the thermomechanical characterization of high temperature materials (e.g. ceramic matrix composites). While SiC alone can withstand these temperatures, a major challenge is to develop reliable electrical contacts to the device itself in order to facilitate signal extraction.

Experimental

Two to three μm thick homoepitaxial layers with nitrogen doping levels of 5-7 x 10^{18} cm^{-3} were commercially grown by Cree, Inc. on the silicon face of off-axis high-resistivity p-type substrates (4H-SiC with a resistivity of 7.5 Ω-cm [4.74 x 10^{17} cm^{-3}] and 6H-SiC with a resistivity of 2.4 Ω-cm [2.65 x 10^{18} cm^{-3}]). The epilayers were patterned into isolated mesas in a parallel-plate reactive ion etcher (RIE) system using SF_6 and Ar chemistry and a metal mask (titanium/nickel/aluminum). Metal contacts were deposited onto mesa surfaces prepared by extensive chemical cleaning. A sequence of equal volume HCl:HNO_3, equal volume 49%HF:HNO_3, and solvent cleaning was used to remove the metal mask and polymer residue introduced during the RIE plasma etch. An equal volume 30%H_2O_2:H_2SO_4 solution was used to remove any remaining organic contamination, followed by wet oxidation and HF strip to remove structural damage.

The contact metallization consisted of a sputtered 1000 Å layer of W (75 at. %):Ni (25 at. %), followed by a 200 Å layer of silicon (Si) which is used to prevent premature oxidation of tungsten after chamber venting and during annealing. The contact metals were chosen for their high

temperature capability, as well as historical results of ohmic behavior with SiC [4, 5]. The transfer length method (TLM) test structure, as shown in Fig. 1, was used to evaluate the metal contacts [6, 7]. TLM structures consisted of five rectangular contact pads, with three subsets. Dimensions/(edge-to-edge distances) were 100 x 40 μm^2/(35,70,105,140 μm), 100 x 45 μm^2/(30,60,90,120 μm), and 100 x 50 μm^2/(25,50,75,100 μm).

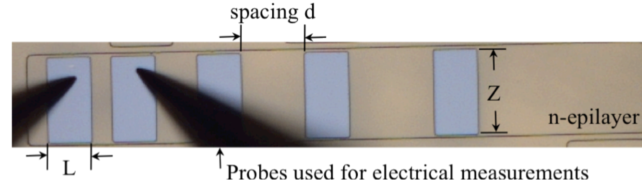

Fig. 1: Optical image of the linear TLM structures used to obtain specific contact resistance values (ρ_c).

Samples were initially annealed for one hour at 1100 °C in 5 slpm argon in a tube furnace, and subsequently heated intermittently for several hours at 1000 °C in argon in the same tube furnace. Room temperature measurements of current-voltage (I-V) and specific contact resistance (ρ_c) were made after deposition, first anneal, and after heating for several hours. Sample surfaces were also analyzed using Auger electron spectroscopy (AES) and X-ray diffraction (XRD). A detailed study of the contact metal/SiC interface after fifteen hours of heating was also performed using high-resolution transmission electron microscopy (HR-TEM) and energy-dispersive X-ray spectroscopy (EDS).

Results

All the samples remained consistently ohmic during the entire duration of the heat treatment. The I-V characteristics of the two polytypes as measured immediately after deposition, after the 1100 °C anneal, and after prolonged treatment at 1000 °C, are shown in Fig. 2a and b, respectively. The probed contacts in Fig. 2 measured 40 μm x 100 μm with a spacing of 30 μm. The I-V curves demonstrate the ohmic behavior of the contacts after first exposure to high temperature and that they remain so during the full heat treatment.

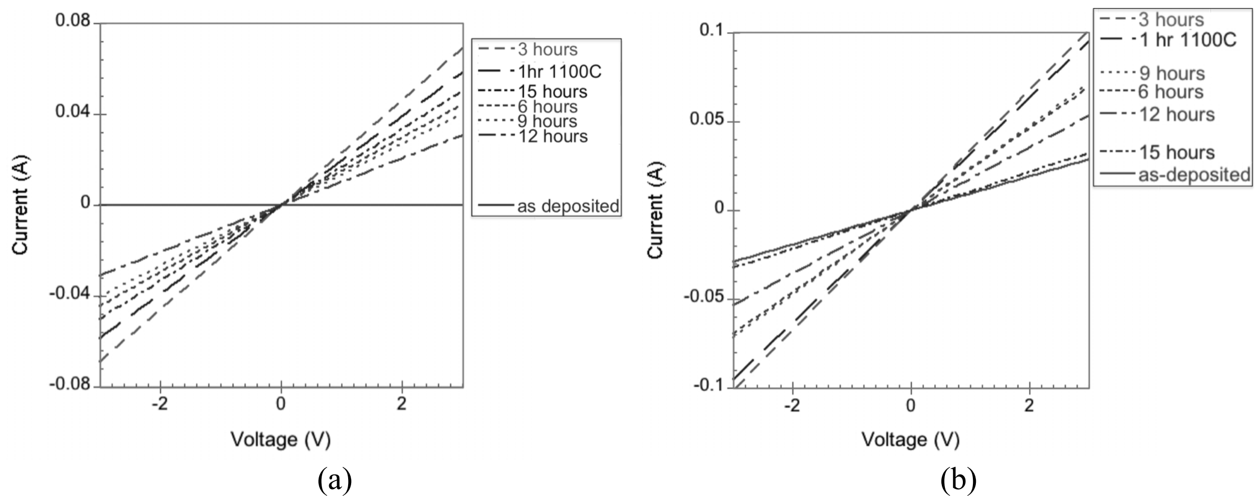

Fig. 2: Comparison of I-V curves after heating for a n-doped SiC epilayer (5.3 - 7.7 x 10^{18} cm^{-3}) on (a) 4H p-type SiC substrate and (b) 6H p-type SiC substrate.

Specific contact resistance, ρ_c, was measured by averaging across nine (6H SiC) and six (4H SiC) TLM subsets from each sample. Values were calculated using plots of total resistance (R_T) versus spacing (d). The linear transfer length L_T was extracted from the x-axis intercept, while the contact

resistance R_C was extracted from the y-axis intercept. The sheet resistance ρ_s of the epilayer was determined from the slope. Specific contact resistance was then calculated by applying Eq. 1 [7]:

$$\rho_c = R_C L_T Z. \quad (1)$$

Contact length, Z, is as defined in Fig. 1. Figure 3 displays specific contact resistance values obtained after heating for various lengths of time. The contact resistance values were initially observed to be high in the first couple of hours, but decreased and remained stable thereafter. The final ρ_c measured after 15 hours at 1000 °C was 5 x 10^{-4} Ω-cm^2 for both 4H and 6H substrates. This value compares reasonably with other contact schemes on similar polytype and doping levels commonly found in literature [8], with the added advantage of an extended temperature range.

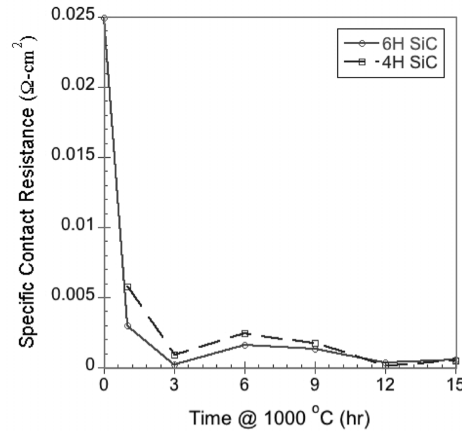

Fig. 3: Specific contact resistance of metallization on 5-7 x 10^{18} cm^{-3} n-doped SiC epilayers after prolonged heating at 1000°C.

In addition to the electrical characterization, the metal contacts were also analyzed by AES depth profiles in order to get a better understanding of the zonal reactions, intermetallic diffusion, and the roles they play in the stability of the electrical behavior at such extreme temperatures. Fig. 4a shows the Auger depth profile of a contact after the initial 1 hour anneal at 1100 °C. Nickel in the alloy has reacted with the SiC substrate to form Ni_xSi_y, thus freeing the carbon that reacts with

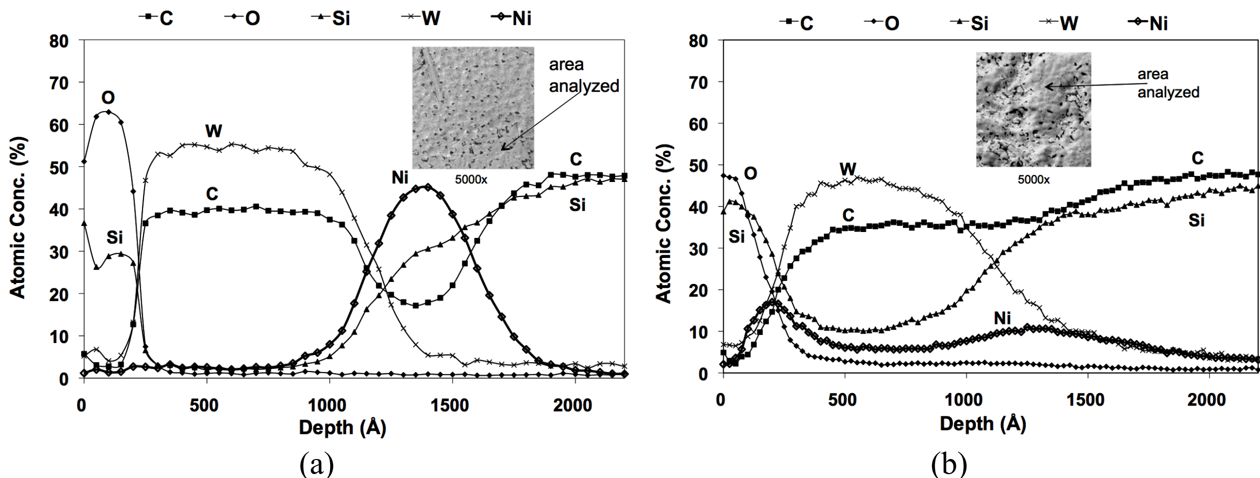

Fig. 4: (a) AES depth profile after 1 hour 1100 °C furnace anneal. (b) AES depth profile after 1 hour furnace anneal at 1100 °C and 14 hours at 1000 °C.

tungsten to form W_xC_y. XRD analysis also showed evidence of the presence of W_xSi_y compounds. The silicon oxide layer that formed on the surface during the initial anneal appears to have protected the tungsten from oxidation, maintaining approximately the same thickness while not interfering with electrical measurements. The AES of Fig. 4b shows that compounds that had formed during initial annealing remained segregated throughout the prolonged fourteen hour heating at 1000 °C,

with distinct crystalline features visible via optical microscopy. Fig. 5 shows a detailed image of the SiC interface after prolonged heating using HR-TEM. Nickel silicide and a matrix of tungsten silicide and carbide are observed to be in intimate contact with the SiC, making possible the formation of the stable ohmic contact.

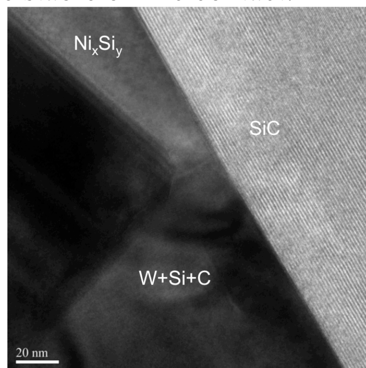

Fig. 5: HR-TEM micrograph showing formation of carbides and silicides at the SiC interface after high temperature anneal and prolonged heating at 1000 ^oC for 14 hours.

Summary

This work has demonstrated a metallization scheme with the capability to survive prolonged heating at 1000 ^oC. This metallization scheme shows that it is possible to achieve a highly stable ohmic contact to SiC within the 1000 ^oC temperature range. It expands the capabilities for SiC devices as well as potentially overcoming the reliability issues that currently limit high temperature operation. Further work planned includes longer duration heating tests, as well as annealing in environments other than argon. Although results are preliminary, the data is encouraging for potential increased operation time of devices at nominal (e.g., 600 ^oC) temperatures.

Acknowledgements

This work was funded by the Hypersonic Project under the Fundamental Aeronautics Program of the National Aeronautics and Space Administration (NASA). The authors greatly appreciate the contributions of A. Avishai of Case Western Reserve University for the HR-TEM analysis, C. Blaha, B. Osborn, P. Neudeck, C. Chang, G. Beheim, and L. Matus, of NASA Glenn Research Center.

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