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Thermal degradation of Au/Ni_2Si/n-SiC ohmic contacts under different conditions

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ABSTRACT

The thermal degradation of Au/Ni_2Si/n-SiC ohmic contact was investigated after long-time aging in air at 400 °C or rapid thermal annealing in Ar up to 700 °C. Current–voltage characteristics, sheet resistance measurements, Rutherford backscattering spectrometry, X-ray diffraction and scanning electron microscopy were used to characterize the contacts before and after heat treatments. Thermal stress at different conditions shows different influence on the properties degradation of Au/Ni_2Si/n-SiC ohmic contacts. It is shown that aging of the contacts in air at 400 °C resulted in complete degradation due to both oxygen penetration and inter-diffusion/reaction processes at the metal/SiC interface. In contrast, only a small change in contact morphology was detected on the contacts annealed in Ar at 700 °C.

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1. Introduction

Realization of the great application potential of silicon carbide (SiC) in the next-generation of high-power devices requires developing of reliable ohmic contacts [1]. Technology of low resistivity ohmic contacts with smooth surface morphology, preserving stability of their properties at high-temperature (>350 °C), must be developed for SiC-based devices operating at high-temperature. Most of the investigations in the last decade are dedicated to the study of the interaction at the metal/SiC interface in order to promote ohmic contact formation. Only few reports concern the long-term thermal stability of ohmic contacts to SiC [2–8]. In most of the previous reports [2–5], long-term reliability tests of ohmic contacts were performed in neutral ambience or vacuum. However, it is more important to have reliability tests of ohmic contacts in air, because devices operate generally in atmospheric environments. Thus, in this paper we report on the comparative study of the stability of ohmic contacts thermally treated in neutral and oxidizing environments.

Stoichiometric phase δ-Ni_2Si has been selected as optimal to form relatively low resistance ohmic contacts with smooth surface on n-SiC [9]. High-conductivity gold overlayer has been deposited after ohmic contact formation for interconnection or bonding metallization. In the next step one set of ohmic contact structures were long-time aged in air at 400 °C, second one were rapid thermal annealed in Ar up to 700 °C. We compare here composition, microstructure and electrical properties of the Au/Ni_2Si/n-SiC contacts before and after heat treatments.

2. Experimental

We used nitrogen-doped n-type ($\sim 2 \times 10^{17}\,cm^{-3}$) 4H-SiC (0 0 0 1) wafers from Cree Research Inc. The substrates were first cleaned according to the procedure described in [9] and then placed in the magnetron sputtering system with base pressure below 10^{-4} Pa.

First, the multilayer Ni/Si/n-SiC structures with thickness ratios of Ni:Si ∼ 1.1 were fabricated by dc magnetron sputtering of Ni and Si targets in Ar plasma. These structures were annealed at 600 °C (N_2, 15 min) to form stoichiometric phase of δ-Ni_2Si and next at 1000 °C (N_2, 3 min) to obtain Ni_2Si/n-SiC ohmic contacts [9]. Then the 150 nm-thick Au overlayers were deposited on Ni_2Si/n-SiC ohmic contacts by dc sputtering of Au target in Ar plasma. The Au/Ni_2Si/n-SiC contact structures were long-time aged in air at 400 °C or rapid thermal annealed (RTA) in neutral gas (Ar) up to 700 °C.

Current–voltage (I–V) characteristics of the contacts were measured by a Keithley 2400 source-meter. The circular transmission line method (c-TLM) was applied to measure contact resistivity (r_c). This method provides an upper estimate of the contact resistivity of ohmic contacts on substrates and the error of the method does not exceed 20% [10]. The sheet resistance (R_{sh}) of metallization was

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measured by a four-point probe. The element depth profiles in the contacts were examined by Rutherford backscattering spectrometry (RBS) using 2 MeV He^+ beam. The contacts phase composition was investigated by X-ray diffraction (XRD) using a Philips X'Pert diffractometer with the Cu K_α X-ray source. The contacts surface morphology was analyzed by a Philips XL-30 scanning electron microscopy (SEM).

3. Results

Electrical and structural characteristics of $Au/Ni_2Si/n\text{-}SiC$ ohmic contacts before and after heat treatments are summarized in Table 1. The contact resistivity (r_c) after fabrication of the contact was $0.4\,m\Omega\,cm^2$, which is one order lower than recently published resistivity of the Ni contact to n-SiC at similar electron concentration ($\sim 10^{17}\,cm^{-3}$) [11]. After aging in air at 400 °C, the r_c strong increases to $0.8\,m\Omega\,cm^2$ for 50 h, and next reveals rectifying behaviour of the contact for 150 h (Fig. 1a). The contact resistivity r_c of annealed at 600 °C and 700 °C contacts in Ar for 3 min remains unchanged (Fig. 1b). In order to explain features of the r_c changes under thermal stress at different conditions, R_{sh} sheet resistance, XRD phase composition, RBS elemental depth profiles and SEM surface morphology of contacts are compared.

The initial sheet resistance ($R_{sh} \sim 290\,m\Omega/sq.$) for as-deposited contacts exceeds sheet resistance for 150 nm-thick Au overlayer ($R_{sh} = \rho_{bulk}/d = 150\,m\Omega/sq.$), because of the electrons scattering by the film grains boundaries and the Au/contact interface. Subsequent aging in air at 400 °C of the $Au/Ni_2Si/n\text{-}SiC$ contact results in increase in R_{sh} by about 20% and 135% for 50 h and 150 h, respectively. Other changes are observed after RTA in Ar for 3 min. The sheet resistance decreases initially about 20% and then increases 20% after annealing at 600 °C and 700 °C, respectively. The changes of contact sheet resistance indicate mostly diffusion processes in metallization systems. However, initial reduction of R_{sh} after RTA at 600 °C is related to recrystallization of the Au layer resulting in decrease in the grain boundary density.

The XRD spectrum of the $Au/Ni_2Si/SiC$ contact includes peaks from the 4H-SiC single crystal, Au and orthorhombic $\delta\text{-}Ni_2Si$ polycrystals. After aging the contact in air at 400 °C for 50 h, a small shift of Au and $\delta\text{-}Ni_2Si$ peaks are detected, and for 150 h formation of NiSi phase and $Au_x(Ni,Si)_{1-x}$ solid solution is observed. After RTA at 600 °C and 700 °C, the $Au/Ni_2Si/n\text{-}SiC$ contacts preserve initial phase composition as unchanged XRD spectra suggest.

Fig. 2 shows the RBS spectra of the $Au/Ni_2Si/n\text{-}SiC$ contacts before and after heat treatments. An arrow in the spectra marks the surface energy of the respective element. Aging in air at 400 °C induces a considerable change in metallization as can be deduced from RBS spectra shown in Fig. 2(a). The RBS spectrum of the 50 h aged contact compared to the spectrum for as-deposited $Au/Ni_2Si/n\text{-}SiC$ contact shows changes of slope, width and shift of the low-energy edge for Au signal to lower energy, as well as appearance of Ni ($E \sim 1.5\,MeV$) and O ($E \sim 0.7\,MeV$) signals. This indicates on beginning of interaction at the Au/Ni_2Si interface: (i) in-diffusion of Au atoms into the contact, (ii) out-diffusion of Ni and Si atoms to the surface, as well as (iii) oxygen penetration into the contact. These inter-diffusion processes lead to disappearance of RBS signal from Ni_2Si and more strong interaction at the both Au/Ni_2Si and $Ni_2Si/n\text{-}SiC$ contact interfaces after aging for 150 h. As it is shown in Fig. 2(b), overlapping of the RBS spectra of the $Au/Ni_2Si/n\text{-}SiC$ contacts before and after annealing in Ar at 600 °C indicates on the same distribution profiles of the elements. Moreover, on comparing RBS spectra of the contacts before and after RTA at 700 °C only tiny Ni surface signal ($E \sim 1.5\,MeV$) is revealed, and interfaces are still abrupt.

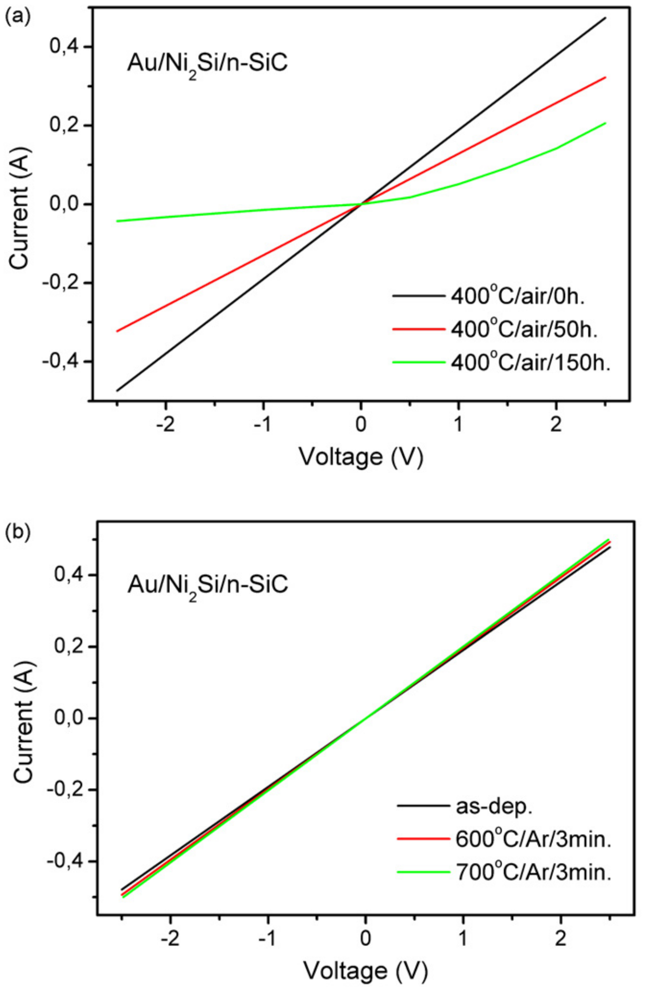

Fig. 1. I–V characteristics of the $Au/Ni_2Si/n\text{-}SiC$ ohmic contacts: before and after aging in air at 400 °C for 50 h and 150 h (a); before and after RTA in Ar for 3 min at 600 °C and 700 °C (b).

Fig. 3 shows the SEM micrographs of the $Au/Ni_2Si/n\text{-}SiC$ contacts before and after heat treatments. The surface morphology of initial contacts is smooth with surface roughness $H_s \sim 17\,nm$ and shiny gold colour (Fig. 3a). As shown in Fig. 3(b,c), after aging in

Table 1

Contact resistivity (r_c), sheet resistance (R_{sh}), surface roughness (H_s) and phase composition of the $Au/Ni_2Si/n\text{-}SiC$ ohmic contacts before and after heat treatments.

Treatment	As-deposited	Aging (400 °C, air)		RTA (Ar, 3 min)	
		50 h	150 h	600 °C	700 °C
r_c^* ($m\Omega\,cm^2$)	0.4	0.8	non-linear	0.4	0.4
R_{sh} ($m\Omega/sq.$)	290	350	680	230	270
H_s^{**} (nm)	~17	~156	~294	~21	~23
Phases (XRD analysis)	Au, $\delta\text{-}Ni_2Si$	Au, $\delta\text{-}Ni_2Si$	$\delta\text{-}Ni_2Si$, NiSi, $Au_x(Ni,Si)_{1-x}$	Au, $\delta\text{-}Ni_2Si$	Au, $\delta\text{-}Ni_2Si$

* The error of r_c determined from the spread of the experimental data over the area of the substrate did not exceed 10%.

** H_s peak-to-valley height measured on the 50 μm surface scan length by the TENCOR α-Step 200 profiler.

air at 400 °C the surface morphology becomes rough indicating the morphology degradation. For 50 h aged contact (Fig. 3b), the change of colour to dark gold, increasing of H_S by a factor of 9 and granular areas are observed. After further aging for 150 h (Fig. 3c), the colour changes to dark brown, H_S increase by a factor of 17 and visible depth craters in metallization all indicate on severe morphology degradation. The average surface density of craters appearing in the Au layer just for 50 h of aging is about 2000 mm^{-2}. Annealing of the Au/Ni$_2$Si/n-SiC contact in Ar at 600 °C (Fig. 3d), leads to changes of the gold surface colour to yellow shade and to small increase in H_S by a factor of 1.2, probably because of recrystallization leading to an increase in Au grains size. Furthermore, a small number of pores are observed in the Au layer. After annealing at 700 °C, only increase in density of pores is visible in the surface morphology modification (Fig. 3e).

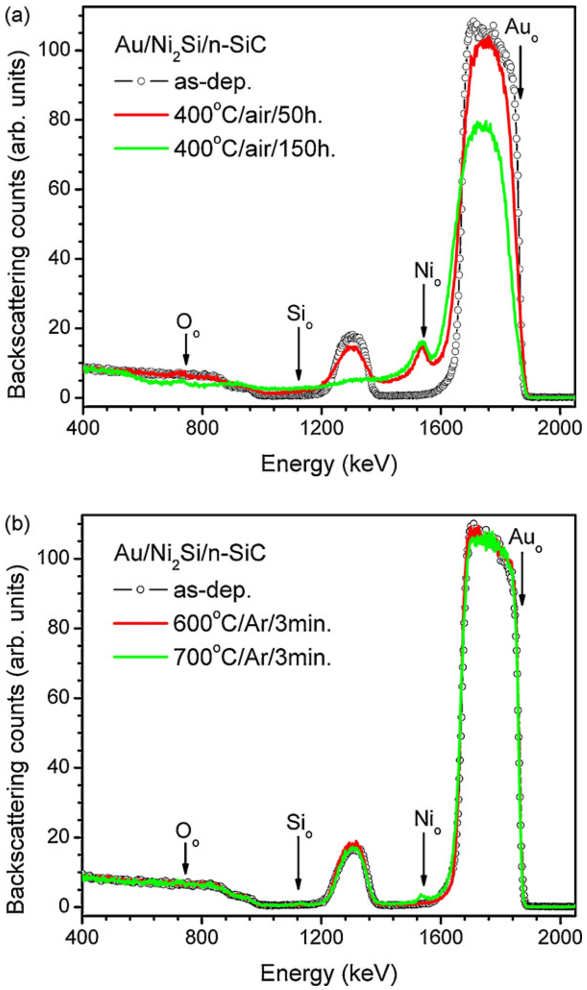

Fig. 2. The 2-MeV He$^+$ backscattering spectra of the Au/Ni$_2$Si/n-SiC ohmic contacts: before and after aging in air at 400 °C for 50 and 150 h (a); before and after RTA in Ar for 3 min at 600 °C and 700 °C (b).

4. Discussion

4.1. *Au/Ni$_2$Si/n-SiC: long-time aging in air at 400 °C*

Aging in air at 400 °C of Au/Ni$_2$Si/n-SiC ohmic contacts apparently induces (i) two times increase in contact resistivity for 50 h, and (ii) non-ohmic behaviour of *I–V* characteristics for 150 h. Strong degradation of the linearity of the *I–V* characteristics suggests influence of inter-diffusion processes into the contacts on metal/SiC interface. Moreover, it is observed, that time of aging has influence on the contact properties.

The change in the contact resistivity for 50 h aged contacts may be correlated mostly with oxygen penetration into the contacts, as was previously observed in papers [2,3]. In fact, the RBS depth profiling shows that width of the metal/SiC interface remains unchanged but the amount of oxygen increases after aging at 400 °C for 50 h. Appearance of the Ni surface signal in the RBS spectrum is related with formation of craters in the Au overlayer. These craters have depth in the range of Au thickness, which correlates well with surface roughness $H_S \sim 150$ nm. Unchanged phase composition of the metallization for 50 h aged contacts indicates that an increase in sheet resistance by only a factor of 1.2 is related to discontinuous structure of Au films (appearance of craters). Thus, we suppose that during aging in air at 400 °C for 50 h, oxygen diffuses into the Au/Ni$_2$Si/n-SiC ohmic

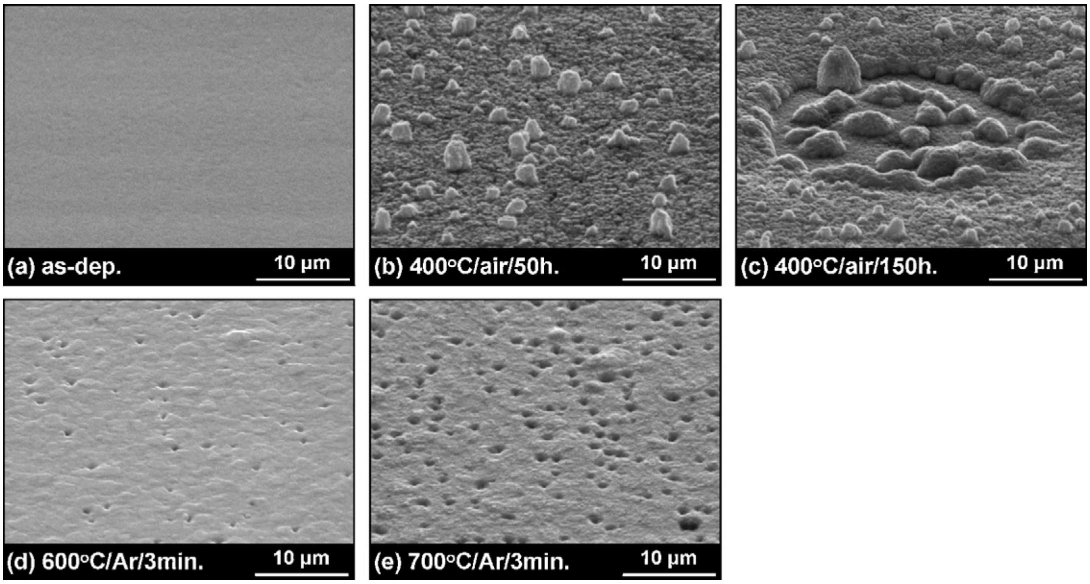

Fig. 3. SEM micrographs of the Au/Ni$_2$Si/n-SiC ohmic contacts: as-deposited (a), 50 h (b) and 150 h (c) aged in air at 400 °C; or 600 °C (d) and 700 °C (e) annealed in Ar for 3 min.

contacts and interacts preferentially via craters created in the Au films.

Transition to non-linear *I–V* characteristics for 150 h aged contacts is caused by both strong redistribution and reaction of components at the metal/SiC interface. Depth profiling from simulation of the RBS spectra shows that the width of the metal/SiC interface increases suggesting considerable inter-mixing of Au, Ni, Si, C and O. In fact, strong inter-diffusion of elemental components of the Au/Ni_2Si/n-SiC contacts during aging in air at 400 °C for 150 h correlates well with (i) decomposition of Ni_2Si, (ii) formation of NiSi and $Au_x(Ni,Si)_{1-x}$ phases, (iii) increasing of sheet resistance more than twice, and (iv) strong morphology degradation. Furthermore, the depth of the craters ($H_s \sim 294$ nm) exceeds the 250 nm thickness of the Au/Ni_2Si metallization, indicating on catastrophic degradation of the ohmic contact.

4.2. Au/Ni_2Si/n-SiC: rapid thermal annealing in Ar up to 700 °C

Rapid thermal annealing in Ar up to 700 °C does not induce detectable changes in the electrical characteristics of the Au/Ni_2Si/n-SiC ohmic contacts. The abrupt Au/Ni_2Si and Ni_2Si/n-SiC contact interfaces suggest non-redistribution of the elements over the depth after RTA annealing also. Additionally, the contacts preserve their as-deposited phase composition. Surface morphology retains relative smoothness up to 600 °C, but morphology degradation following pores formation at 700 °C is observed.

5. Conclusions

We observed that degradation of the Au/Ni_2Si/n-SiC ohmic contacts becomes stronger after long-time aging in air at relatively low temperature (400 °C), than after rapid thermal annealing up to 700 °C in neutral gas (Ar). Degradation process in air comes mainly from the diffusion of oxygen into the contacts. Apart from oxidation of the contacts, oxygen plays the role of a catalyst, which enhances inter-diffusion and/or reaction in metal/SiC contacts. Thus, a diffusion barrier that prevents both inter-diffusion between contact multilayers and in-diffusion of oxygen is suggested to improve long-time thermal stability of ohmic contacts to SiC.

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