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Single-step synthesis process of Ti_3SiC_2 ohmic contacts on 4H-SiC by sputter-deposition of Ti

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We report a single-step procedure for growth of ohmic Ti_3SiC_2 on 4H-SiC by sputter-deposition of Ti at 960 °C, based on the Ti–SiC solid-state reaction during deposition. X-ray diffraction and electron microscopy show the growth of interfacial Ti_3SiC_2. The as-deposited contacts are ohmic, in contrast to multistep processes with deposition followed by rapid thermal annealing. This procedure also offers the possibility of direct synthesis of oxygen-barrier capping layers before exposure to air, potentially improving contact stability in high-temperature and high-power devices.

Keywords: Silicon carbide; MAX phase; Physical vapor deposition; High temperature

Silicon carbide holds a combination of useful properties [1], e.g. wide band gap, high breakdown electric field strength, high thermal conductivity and chemical inertness, making it ideal for electronic devices for high temperature and high power applications. One of the most basic electronic elements in such devices is a suitable ohmic contact. To this end, Ti/Al based contacts are widely studied for 4H-SiC [2–7].

The synthesis method of this class of ohmic contacts requires a post-deposition process with rapid thermal annealing at around 950 °C. This process results in the formation of new phases in the contact, mainly Ti_3SiC_2 and Ti_3Al. Transmission electron microscopy has shown that Ti_3SiC_2 grows at the interface. Therefore, it is the main reason for ohmic properties [8–10]. Moreover, first-principles studies have also attributed the efficient lowering of the Schottky barrier, and the corresponding ohmic behavior, to the formation of Ti_3SiC_2 [11–13].

Ti_3SiC_2 is a member of the family of layered carbides and nitrides known as MAX phases [14,15], where M is an early transition metal, A is an element from groups 12–16, and X is carbon or nitrogen. This family exhibits an unusual combination of metallic and ceramic-like properties [16], such as high-temperature stability and high electrical conductivity. Ti_3SiC_2 thin films are commonly grown by physical vapor deposition, primarily sputtering from elemental [17,18] or compound targets [19]. However, growth of this phase on 4H-SiC was reported to still require rapid high-temperature thermal annealing to exhibit ohmic properties [20]. Transmission electron microscopy studies before and after the annealing process of Ti_3SiC_2 films have shown that annealing results in a more ordered interface between the film and the 4H-SiC [20].

Eliminating the annealing process-step, i.e. synthesizing as-deposited ohmic contacts through a single-step process, would be beneficial for ohmic contacts with SiC-based devices [21]. In addition, this approach puts forward the possibility of directly synthesizing oxygen-barrier capping layers after the main contact deposition without exposing the devices to air for a process step like annealing, thereby avoiding any risk of oxidation or contamination, or any need for a cleaning step. This can improve the long-term stability of devices using ohmic contacts, especially those that operate at high temperatures and in a corrosive environment. Here, we report a straightforward procedure for that purpose, by deposition of Ti at a shigh substrate temperature. The ohmic contacts form during deposition because of the reaction between the sputter-deposited Ti and the substrate to form Ti_3SiC_2.

The depositions were performed in an ultrahigh-vacuum stainless steel chamber with a base pressure lower than 1.3×10^{-6} Pa. The deposition sources were two sputtering targets, Ti (99.995%) and Pt (99.99%), 5.08 cm in diameter, run in power-regulated DC mode. We used the Pt target only for the synthesis of the capping layers. Temperature was calibrated before the series of depositions using a

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thermocouple placed at the substrate position. The substrates were mechanical grade n-type (10^{18} cm^{-3}) 4H-SiC, (0001), 4° off-axis, diced to 10 × 10 mm in size. Prior to deposition, they were ultrasonically cleaned by acetone and isopropanol for 10 min each, blown dry in pure nitrogen and inserted directly into the load-lock of the chamber. The sputtering gas was Ar, with pressures of 0.32 and 0.1 Pa for Ti and Pt depositions, respectively. We used the minimum possible pressure of 0.1 Pa for deposition of Pt in order to eliminate film roughness. X-ray diffraction (XRD) was performed using a Philips PW 1820 instrument (Cu K_α, θ–2θ scan, aligned with the substrate (0004) peak). Scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX) were performed in a LEO 1550 microscope for surface imaging, chemical analysis and film thickness measurement via cross-sectional samples. We used 5 and 2 kV accelerating voltages for the SEM and EDX, respectively. The choice of this low accelerating voltage for the latter method was made to obtain highly surface-sensitive mapping of C. Transmission electron microscopy (TEM) and selected area electron diffraction (SAED) studies were done in a Tecnai G2 TF20UT FEG microscope. Cross-sectional samples were first mechanically polished to a thickness of about 60 μm, followed by ion-beam milling with Ar^+ in a Gatan precision ion polishing system at 5 keV ion energy, with a final polishing step at 1.5 keV. For electrical measurements, we deposited contacts on samples using a shadow mask, resulting in two distinct 4 × 10 mm^2 coated areas on the substrate separated by 1.2 mm. The shadow mask was made of 4H-SiC to rule out any potential reaction between the mask and the 4H-SiC substrate. To improve electric current spreading in the film, we deposited a 250 nm thick Pt layer on top of the main contacts before removing the shadow mask. Pt deposition was performed at room temperature to exclude any further influence of high temperature on the interface between the contact and the 4H-SiC. In addition, it was done in the same chamber without breaking the vacuum to prevent any oxidation or gas adsorption at the contact-capping layer interface. Current–voltage measurements (I–V) were performed using a Keithley 2601 source meter equipped with two removable gold-coated electrodes placed directly on the two separated contact areas on the substrate. In this set-up, the current flows through three different interfaces in each contact area. These interfaces are removable contact–Pt, Pt–main contact and main contact–substrate. Any potential non-ohmic behavior would only correspond to the latter since the first two are metal–metal contacts and are ohmic. Therefore, this relatively simple measurement set-up allows the ohmic

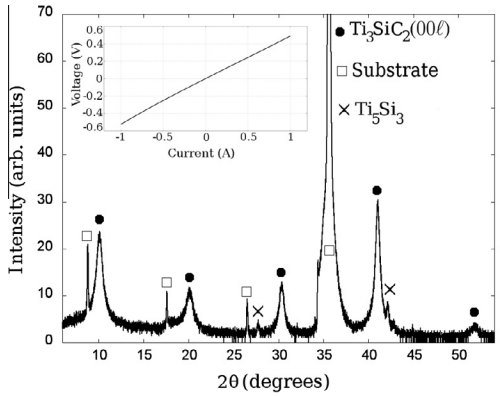

Figure 1. X-ray diffractogram of a 4H-SiC substrate coated with Ti at 960 °C for 10 min, inset: I–V curve.

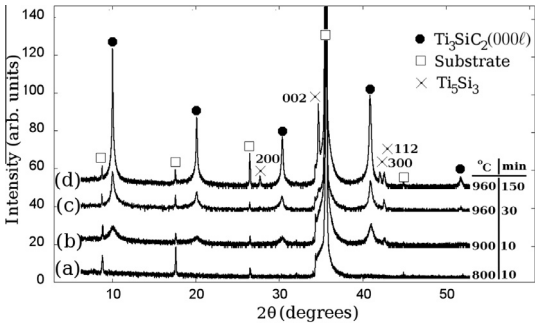

Figure 2. X-ray diffractograms of samples deposited at (a) 800 °C and (b) 900 °C for 10 min, and of samples deposited at 960 °C for (c) 30 min and (d) 150 min.

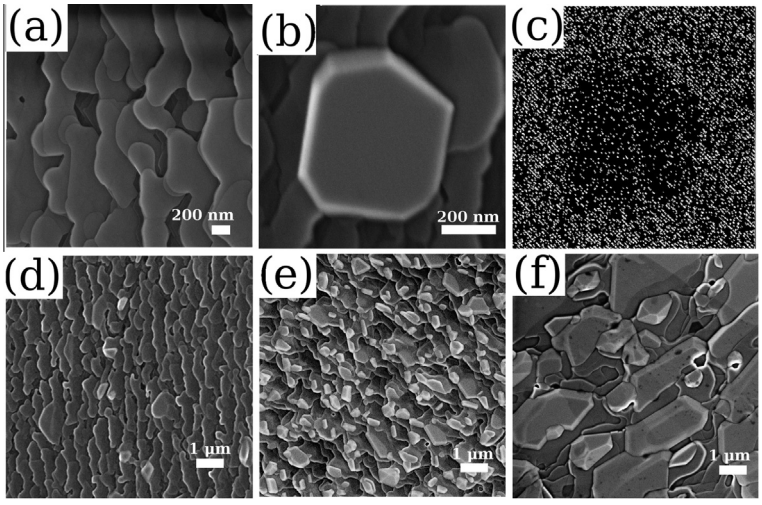

Figure 3. SEM images and EDX mapping of 4H-SiC substrates coated with Ti at 960 °C: (a) 10 min deposition, showing plate-like Ti_3SiC_2 grains; (b) 10 min deposition, showing a faceted Ti_5Si_3 grain; and (c) 10 min deposition, showing the EDX data of the C K_α peak. Surface morphology of samples deposited for (d) 10 min, (e) 30 min and (f) 150 min.