On the chemistry of epitaxial Ti3SiC2 formation on 4H-SiC using Al-Ti annealing

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Making a low resistance ohmic contact on *p*-type SiC is known to be difficult. High-temperature annealed Ti and Al-containing alloys have been reported to give good results [1]. Among the reasons for explaining this, the formation of Ti_3SiC_2 phase is frequently proposed. This material, of the MAX phase family, has a very interesting combination of metallic and ceramic properties, together with an excellent chemical stability with SiC. However, the chemical path leading to its formation from Al-Ti annealing on SiC is not well understood, leading to some non-reproducibility of the process.

In order to form Ti_3SiC_2 on 4H-SiC(0001) 8°off, 200 nm of $Ti_{30}Al_{70}$ was deposited onto SiC substrates by magnetron sputtering from pure $Ti_{30}Al_{70}$ targets in a high vacuum system. The deposition was carried out with an Ar constant pressure (5×10^{-3}mbars) at room temperature. The samples were then annealed at 1000°C for 10 min under Ar atmosphere in a rapid thermal annealing oven (heating rate of about 20°C/s).

Figure 1 shows XRD theta-2theta scan of a typical sample annealed in these conditions. As seen, the diffraction peaks unambiguously reveal the formation of the hexagonal Ti_3SiC_2 structure which is in epitaxial relation with the substrate. The rocking curves obtained on the (0008) diffraction planes of the Ti_3SiC_2 phase present a full width at half-maximum (FWHM) of 0.4° showing the good crystallinity of the film. Using XPS analysis, high amount of Al and O elements were also detected inside the deposit, which was not seen from XRD measurements. In order to better understand this, TEM investigations were carried out. Figure 2 displays a low magnification bright field (BF) filtered image of a typical region of an as-annealed sample. This general view shows that the 4H-SiC substrate is entirely covered by a Ti_3SiC_2 layer with varying thickness from ~95 to 140 nm. On top of this is found an almost continuous layer which was attributed to amorphous to polycrystalline Al oxide (probably Al_2O_3). Obviously, the formation of Ti_3SiC_2 is accompanied by unwanted Al oxide, probably due to some unwanted oxygen residual in the RTA chamber.

By using proper backing steps before the annealing, the results are clearly different. As can be seen from XRD measurements (figure 1b), several compounds (Ti_3SiC_2, Al_3Ti, and Al) are now detected. As a result, oxygen seems to play a key role during the Ti-Al/SiC interaction for obtaining only one Ti-based phase (Ti_3SiC_2). The following chemical reactions are proposed to summarize globally the results obtained:

Without oxygen : $20Al + 9Ti + 2SiC \rightarrow Ti_3SiC_2 + 6Al_3Ti + 2Al + Si$

With oxygen : $14Al + 6Ti + 4SiC + 27O \rightarrow 2Ti_3SiC_2 + 7Al_2O_3 + 3SiO_2$

Apparently, oxygen is "pumping" preferentially the Al atoms from the Al-Ti alloy so that the system moves towards the single Ti_3SiC_2 phase instead of a mixture with Al_3Ti.

On the oxide free sample, the specific contact resistivity ρ_c of the Ti_3SiC_2 based contact on 4H-SiC will be measured by the TLM technique.

[1] F. Roccaforte, A. Frazzetto, G. Greco, F. Giannazzo, P. Fiorenza, R. Lo Nigro, M. Saggio, M. Leszczynski, P. Pristawko, V. Raineri, " Critical issues for interfaces to p-type SiC and GaN in power devices ", Applied Surface Science 258 (2012) 8324– 8333.

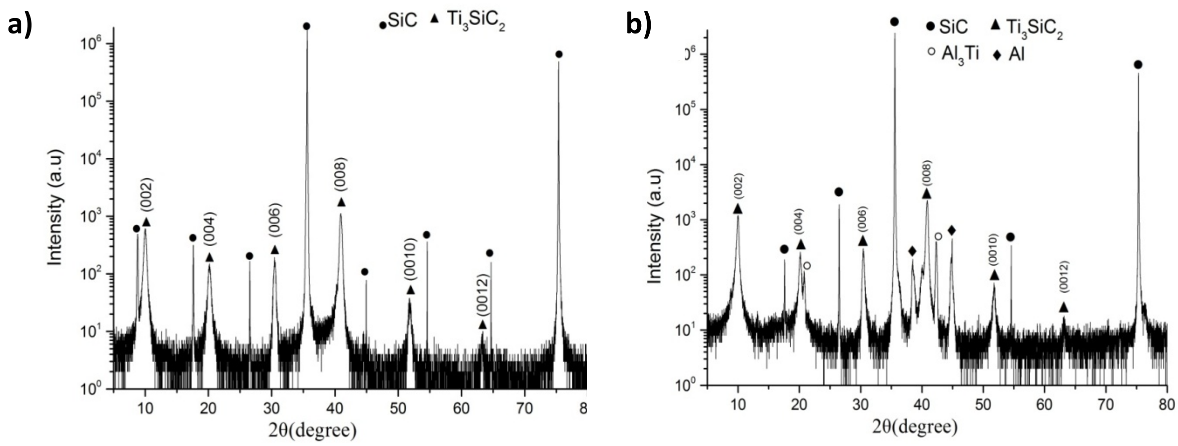

Figure 1. X-ray diffractograms of the TiAl contacts (200nm) onto (0001)Si-face 4H-SiC annealed at 1000°C for 10min a) without and b) with proper backing of the system

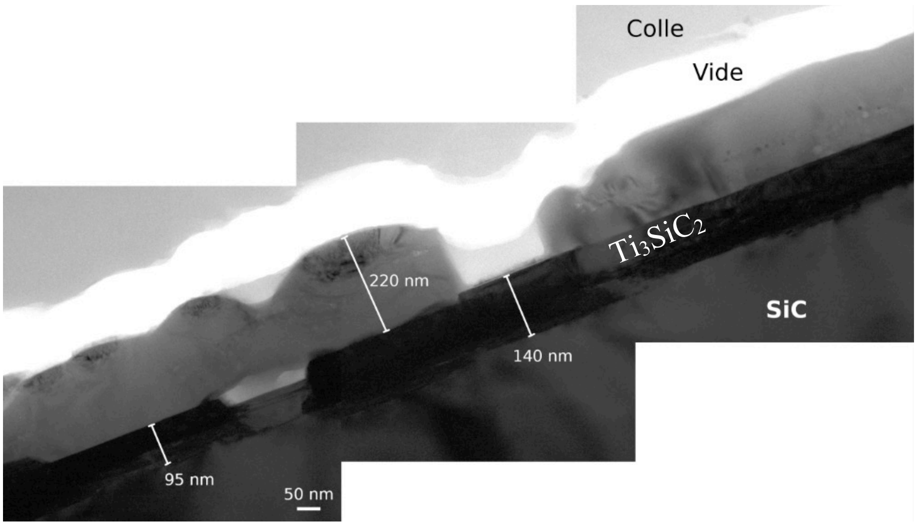

Figure 2. Cross-sectional TEM image of the same sample as in figure 1. Ti_3SiC_2 covers completely the surface of the SiC substrate and is itself covered by a Al oxide layer.