Thermal annealing effect on electrical and structural properties of Tungsten Carbide Schottky contacts on AlGaN/GaN heterostructures

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Abstract
Tungsten carbide (WC) contacts have been investigated as an original gold-free Schottky metallization for AlGaN/GaN heterostructures. The evolution of the electrical and structural/compositional properties of the WC/AlGaN contact has been monitored as a function of the annealing temperature in the range from 400 to 800 °C. The Schottky barrier height ($\Phi_B$) at the WC/AlGaN interface, extracted from the forward current-voltage characteristics of the diode, decreased from 0.82–0.85 eV in the as-deposited and 400 °C annealed sample, to 0.56 eV after annealing at 800 °C. This large reduction of $\Phi_B$ was accompanied by a corresponding increase of the reverse leakage current. Transmission electron microscopy coupled with electron energy loss spectroscopy analyses revealed the presence of oxygen (O) uniformly distributed in the WC layer, both in the as-deposited and 400 °C annealed sample. Conversely, oxygen accumulation in a 2–3 nm thin W-O-C layer at the interface with AlGaN was observed after the annealing at 800 °C, as well as the formation of W$_2$C grains within the film (confirmed by x-ray diffraction analyses). The formation of this interfacial W-O-C layer is plausibly the main origin of the decreased $\Phi_B$ and the increased leakage current in the 800 °C annealed Schottky diode, whereas the decreased O content inside the WC film can explain the reduced resistivity of the metal layer. The results provide an assessment of the processing conditions for the application of WC as Schottky contact for AlGaN/GaN heterostructures.

Keywords: AlGaN/GaN heterostructures, Au-free metallization, Schottky contacts, Tungsten Carbide, current-voltage characteristics

1. Introduction

In recent decades, AlGaN/GaN heterostructures have been receiving great attention from the semiconductor community. A unique feature of these systems is the carrier confinement at the AlGaN/GaN heterointerface in a two-dimensional electron gas (2DEG) having a high sheet density ($\sim$10$^{13}$ cm$^{-2}$) and mobility ($\sim$2000 cm$^2$ V$^{-1}$ s$^{-1}$) [1]. This characteristic, combined with the large band gap and high critical electric field of GaN-based materials, allows the fabrication of high electron mobility transistors (HEMTs) operating at a high-frequency and high-voltage, suitable for power electronics applications [2]. An interesting aspect is the possibility to grow
AlGaN/GaN heterostructures onto large area Si substrates, which makes these systems very promising for the monolithic integration of GaN-based devices with Si technology. In this context, making the fabrication of GaN-devices fully compatible with Si technology requires the use of Au-free Ohmic and Schottky metallizations, in order to prevent undesired sources of contamination and to reduce the fabrication costs.

In previous literature, several works discussed the key role of Au in the achievement of a low contact resistance and, hence, the possible routes to form good Au-free Ohmic contacts on AlGaN/GaN heterostructures [3–10].

On the other hand, Schottky contacts are less studied, although they are equally important in AlGaN/GaN HEMTs, as they modulate the output current in the device and have a strong impact on the leakage current in the off-state [2]. For that reason, a good understanding of the carrier transport mechanisms in Au-free Schottky contacts and their response to thermal budgets are required [11, 12].

Typically, Schottky contacts to GaN-based materials are formed using Ni/Au [13, 14] or Pt/Au [15] bilayers. However, many Au-free Schottky metallizations for GaN-based heterostructures have been proposed as an alternative, e.g. Ti [16], Cr [16], Pt [17, 18], Pd [17, 19], Ir [19, 20], Mo [21], Pd/Mo [22], TiSi2/Cu [23], ITO [24, 25], TaN [25, 26], and TiN [27, 28]. The latter (TaN and TiN) exhibited some interesting features, such as a low leakage current combined with a good thermal stability [26, 27]. The electrical properties of these systems are strongly influenced by the deposition technique, as their work function can vary in wide range (i.e. 4.13–5.05 eV for TaN [29] and 3.8–4.6 eV for TiN) [30–32].

Recently, tungsten-based compounds (W [33], WSi [19] or WN [34]) have been proposed as Schottky contacts on AlGaN/GaN heterostructures, as they showed promising results in terms of ON/OFF current ratio and turn-on voltage uniformity. Among W-based compounds, tungsten carbide (WC) is known for its high mechanical and wear resistance properties [35, 36]. In recent years the use of WC has also been considered to obtain stable Schottky contacts on silicon carbide [37, 38] and diamond [39, 40]. However its possible use on GaN-based materials and heterostructures has not been investigated yet.

In this paper, the electrical behavior of tungsten carbide (WC) Schottky contacts on AlGaN/GaN heterostructures has been studied and correlated with the structural and compositional evolution of the WC/AlGaN system after thermal annealing. This study revealed good electrical performances of the diodes up an annealing temperature of 600 °C and a degradation of the forward and reverse bias characteristics after 800 °C, which was explained by the reduction of the WC/AlGaN Schottky barrier height associated to the formation of a thin oxygen-rich W-O-C layer at the interface.

2. Experimental

AlGaN/GaN heterostructures grown on Si, with an AlGaN thickness of 16 nm and an Al content of 26%, have been used in this work. The choice of this heterostructure parameters was done in order to achieve a good trade-off between the 2DEG sheet carrier density and the Ohmic contacts performances. The fabrication of Schottky diodes started with the definition of the Ohmic contact by optical lithography and the lift-off technique. Non-recessed Au-free Ohmic contacts have been formed by Ti/Al/Ti multilayer annealed at 600 °C for 3 min [10]. Then, Schottky contacts have been obtained by the deposition of 100 nm of the WC layer. The WC films have been deposited by DC magnetron sputtering, carried out at a base pressure (before deposition) in the order of $10^{-5}$ mbar and a working pressure of $5 \times 10^{-3}$ mbar. Such structures consist of an inner circular Schottky contact with a radius of 40 μm and an Ohmic contact formed by a circular ring, which surrounds the Schottky electrode. Also the Schottky contacts were defined by optical lithography followed by the lift-off technique. Both Ohmic and Schottky metals have been deposited using a Quorum Q300T D sputter system. Post deposition rapid annealing (60 s) treatments have been performed in the temperature range 400 °C–800 °C, by using a Jipelec JetFirst 150 furnace. Annealing treatments have been carried out in an Ar atmosphere in order to prevent any possible surface reaction with the metal. Atomic force microscopy (AFM) scans have been acquired to monitor the surface morphology of the investigated material using a Dimension 3100 microscope with a Nanoscope V controller. The current–voltage ($I–V$) measurements were performed on a Karl Suss Microtec probe station equipped with a HP 4156B parameter analyser. The structural analyses were conducted by x-ray diffraction (XRD) and Transmission Electron Microscopy (TEM). XRD measurements were carried out using a Malvern Panalytical Empyrean High Resolution x-ray Diffractometer. TEM analyses in a cross-section were performed using a 200 kV JEOL 2010F microscope, equipped with a Gatan imaging filter (GIF) spectrometer allowing electron energy loss spectroscopy (EELS).

3. Results and discussion

Firstly, the surface morphology of the WC layer deposited onto the AlGaN/GaN heterostructure has been monitored by AFM. Figure 1 shows four representative AFM images acquired on 50 × 50 μm² scan areas of the bare AlGaN surface (a), of the as-deposited WC contact (b) and after annealing at 600 °C (c) and 800 °C (d). Figure 1(e) summarizes the evolution of the root mean square (RMS) surface roughness of the WC as a function of the annealing temperature. Each value in the plot is the average of the RMS obtained on several AFM scans and the error bar is the standard deviation. The as-deposited WC layer exhibits a similar surface morphology to that of the AlGaN underneath (RMS = 5.1 nm ± 1.1 nm). It can also be noticed that the starting RMS value of the as-deposited WC is preserved up to the annealing temperature of 600 °C, whereas a further annealing temperature increase to 800 °C results in a higher surface roughness (RMS = 7.4 nm ± 1.6 nm).

The evolution of the resistivity of the WC layer deposited on AlGaN/GaN heterostructures was also monitored by four-point probe measurements as a function of the annealing...
Figure 1. AFM images of the bare AlGaN surface before metal deposition (a), of the as-deposited (b), the 600 °C (c) and 800 °C annealed (d) WC contacts. Average values of the surface roughness (RMS) of the WC contact, determined by the AFM images, as function of the annealing temperature (e).

Figure 2. Resistivity of the WC layer deposited on AlGaN/GaN heterostructure as function of the annealing temperature. The literature data of WC films on sapphire, taken from [39], are reported for comparison.

temperature (see figure 2). In particular, a resistivity of about $2 \times 10^{-4}$ Ωcm was evaluated for the as-deposited WC layer. The metal resistivity decreased down to $1 \times 10^{-4}$ Ωcm after the thermal treatment at 800 °C. Resistivity values in the order of $10^{-4}$ Ωcm are typical of sputtered WC thin films, as can be seen from the good agreement with literature values measured on WC films on sapphire [39], which is reported in the same graph for comparison.

Then, the electrical behavior of the diodes fabricated on AlGaN/GaN heterostructures by using a WC Schottky contact have been investigated. Figure 3(a) shows the forward current–voltage ($J$–$V$) characteristics of diodes with the as-deposited WC contact and after different annealing temperatures (up to 800 °C). A decrease of the forward current was initially observed from the as-deposited contact to the one annealed at 400 °C, whereas a higher current injection was found at the annealing temperature of 600 °C and 800 °C. For all samples, the semilog plot of the $J$–$V$ curves displays a linear increase of the current in the bias range from 0 to $\sim$1 V, followed by a saturation behavior at higher forward bias values ($V > 1.0–1.5$ V) due to the series resistance contribution. In ideal Schottky barrier diodes, the carrier transport is typically ruled by the thermionic emission (TE) model, which in the linear region can be approximated as:

$$J = J_S \left[ \exp \left( \frac{qV}{nkT} \right) - 1 \right] \approx J_S \exp \left( \frac{qV}{nkT} \right) \quad (1)$$

with

$$J_S = A^* T^2 \exp \left( - \frac{q\Phi_B}{kT} \right) \quad (2)$$

where $A^*$ is the Richardson constant (32 $A/(cm^2K^2)$ for AlGaN) [14, 41], $T$ is the measurement temperature, $k$ is the Boltzmann constant, and $\Phi_B$ and $n$ are the Schottky barrier height and the ideality factor, respectively.

From the fit of the experimental curves with the TE model, an ideality factor of 1.59 and a barrier height of 0.82 eV were evaluated for the as-deposited WC Schottky diode. The theoretical barrier height value $\Phi_B$ for an ideal WC/AlGaN interface depends on the WC work function $\Phi_{m(WC)}$ according to the Schottky-Mott relation $\Phi_B = \Phi_{m(WC)} - \chi_{AlGaN}$, where $\chi_{AlGaN}$ is the value of the electron affinity for an AlGaN layer with 26% Al content. For the WC, a work function of 5.2 eV can be estimated [42, 43]. In this condition, an ideal barrier height of 1.1 eV is expected, which is much higher than that obtained by the fit of the experimental $J$–$V$ curves. A similar deviation from the ideal barrier value have been observed in Schottky contacts to AlGaN/GaN heterostructures for different metalizations [44, 45]. Despite the physical origin of this deviation is still debated, many works ascribe this behavior to the material quality, the presence of surface states, effects of polarization charges or the presence of the 2DEG [14, 46–48].
After thermal annealing at 400 °C, a decrease of the ideality factor from 1.59 to 1.50 is observed, accompanied by a slight increase of $\Phi_B$ up to 0.85 eV. After thermal annealing at 400 °C, a decrease of the ideality factor from 1.59 to 1.50 is observed, accompanied by a slight increase of $\Phi_B$ up to 0.85 eV. A decrease of the ideality factor after annealing of the Schottky contact has been in some case associated with the reduction of surface state density caused by the annealing treatment [49]. By further raising the annealing temperature to 600 °C and 800 °C, the ideality factor significantly increased to 2.13 and 2.65, while the barrier height decreased to 0.72 eV and 0.56 eV (figure 3(b)). Such a trend observed at these annealing temperatures is an indication of an electrical degradation of the metal/semiconductor interface.

The current-voltage characteristics of the WC/AlGaN/GaN Schottky diodes under negative (reverse) bias have been also investigated. Figure 4(a) shows the reverse $J$–$V$ curves acquired after different annealing temperatures. All the acquired characteristics exhibited a first region at a lower negative bias where the current increases with the voltage, followed by a second region at a higher bias with a constant current (i.e. independent of the bias), corresponding to the complete depletion of the 2DEG. Evidently, the annealing treatments have a notable impact on the reverse characteristics of the diodes. In particular, the leakage current density at a reverse bias $V_R = -10$ V firstly decreased from $4.1 \times 10^{-4}$ A cm$^{-2}$ in the as-deposited WC contact to $2.2 \times 10^{-5}$ A cm$^{-2}$ after annealing at 400 °C, while it increased again to $1.3 \times 10^{-2}$ A cm$^{-2}$ in the sample annealed at a higher temperature of 600 °C. A further increase of the leakage current density to $3.3 \times 10^{-2}$ A cm$^{-2}$ was observed after the contact was annealed at 800 °C (figure 4(b)).

The increase of the reverse current can be justified by the reduction observed in the Schottky barrier height. For comparative purpose, the reverse leakage current density values measured at $V_R = -10$ V on as-deposited and 400 °C annealed Ni/Au Schottky contacts, fabricated on the same material, are displayed in figure 4(b). It is worth noting that a factor of two lower leakage current density can be observed in the case of the WC contacts. For higher annealing temperatures, this comparison is not particularly significant, due to the intermixing of Ni and Au layers occurring in the Ni/Au bilayer [50].

Figure 3. (a) Forward J–V characteristics of WC Schottky diodes subjected to annealing processes at different temperatures; (b) ideality factor and Schottky barrier height as a function of the annealing temperature.

Figure 4. (a) Reverse J–V characteristics of WC Schottky diodes subjected to annealing processes at different temperatures. (b) Reverse leakage current density at $V_R = -10$ V as a function of the annealing temperature. For comparison, the leakage current density values of as-deposited and 400 °C annealed Ni/Au contacts are also reported.
can be observed from the TEM analyses on the as-deposited and annealed samples at 400 ◦C. The WC layer (see figure 6(c))) EELS analysis confirmed the presence of both W, C and O in this thin layer. Moreover, at this annealing temperature the presence of W-rich grains was detected in the metal film, which is likely associated with the W2C phase detected by XRD analysis. In order to investigate the origin of the oxygen incorporated at the interface layer, EELS analyses have also been carried out to monitor the oxygen content within the as-deposited and annealed WC metal films. Figure 6(d) displays three typical EELS spectra collected within the metal film in the as-deposited sample and after annealing at 400 ◦C and 800 ◦C. It is worth noting that the leakage current values vary in a wide range, thus making a correlation between these electrical parameters not straightforward. In fact, the leakage current can be influenced by several materials parameters (e.g. defect density, doping level, buffer, etc.). It is worth noting that the proposed WC contact provides a good trade-off between the Schottky barrier (0.82 eV) and the leakage current density (4.1 × 10−4 A cm−2). However, further investigations are still required to assess the breakdown and reliability behaviour of this alternative Schottky metallization.

In order to understand the origin of the electrical behaviour of the Schottky contacts, a structural investigation of the WC layer has been carried out by the means of XRD and TEM analyses.

In particular, no significant structural changes of the WC layer were shown by XRD analysis (see figure 5) in the as-deposited contact and after the thermal annealing at 400 ◦C. On the other hand, by increasing the annealing temperature to 800 ◦C, some peaks associated to the presence of W2C appear in the XRD spectrum, despite the WC remaining the dominant phase in the metal layer. The bank of inorganic structures ICSD has been used to identify the diffraction peak of WC (ICSD No5212) and W2C (ICSD No619097). As a matter of fact, some works report on the formation of the W2C phase after thermal annealing [51] and, in general, on the possible formation of other W-C phases at temperatures higher than 500 ◦C [52].

In figure 6 cross-sectional bright field TEM micrographs of the WC/AlGaN interface are shown for the as-deposited and annealed samples at 400 ◦C and 800 ◦C. No significant differences in the WC film and in the WC/AlGaN interface can be observed from the TEM analyses on the as-deposited (see figure 6(a)) and on the 400 ◦C annealed sample (see figure 6(b)). Instead, after annealing at a temperature of 800 ◦C, a 2–3 nm thick interface layer appears between the AlGaN and the WC layer (see figure 6(c)). EELS analysis confirmed the presence of both W, C and O in this thin layer. Moreover, at this annealing temperature the presence of W-rich grains was detected in the metal film, which is likely associated with the W2C phase detected by XRD analysis. In order to investigate the origin of the oxygen incorporated at the interface layer, EELS analyses have also been carried out to monitor the oxygen content within the as-deposited and annealed WC metal films. Figure 6(d) displays three typical EELS spectra collected within the metal film in the as-deposited sample and after annealing at 400 ◦C and 800 ◦C. It is worth noting that the

### Table 1. Survey of literature data on as-deposited Au-free Schottky contacts on AlGaN/GaN heterostructures.

| Schottky Metal | Ideal factor | Schottky Barrier height (eV) | Leakage current density (A cm−2) @ VR = −10 V | Ref |
|---------------|-------------|-----------------------------|-----------------------------------------------|-----|
| Ir            | 1.72        | 1.22                        | 2.6 × 10−3                                   | [19]|
| Pd            | 1.84        | 1.13                        | 8.1 × 10−4                                   | [19]|
| Ni            | 2.94        | 0.71                        | 0.24                                         | [19]|
| WSi           | 2.28        | 0.58                        | 0.3                                          | [19]|
| Pd/Mo         | 1.75        | 0.68                        | 2.5 × 10−3                                   | [22]|
| TiSi2/Cu      | N.A.        | 1.07                        | 2.6 × 10−4                                   | [23]|
| Ni/Au         | N.A.        | 0.51                        | 0.13                                         | [25]|
| ITO           | N.A.        | 0.54                        | 0.2                                          | [25]|
| TaN           | N.A.        | 0.62                        | 0.07                                         | [25]|
| TaN           | 4.42        | 0.68                        | 2.0 × 10−4                                   | [26]|
| TiN           | N.A.        | 1.1                         | 8.0 × 10−4                                   | [28]|
| W             | 1.08        | 0.65                        | 0.01                                         | [33]|
| W             | 1.15        | 0.89                        | N.A.                                         | [34]|
| W6GeN34       | 1.42        | 1.07                        | N.A.                                         | [34]|
| W32Ni8        | 1.24        | 1.21                        | 9.0 × 10−4                                   | [34]|
| Ni/Au         | 1.16        | 1.37                        | 8.0 × 10−4                                   | [34]|
| Ni/Au         | 1.68        | 0.85                        | 4.1 × 10−3                                   | Our work|
| WC            | 1.59        | 0.82                        | 4.1 × 10−4                                   | Our work|

In past many Au-free solutions have been proposed as an alternative to the standard Ni/Au Schottky contact. Table 1 shows a survey of literature data on as-deposited Au-free Schottky contacts. As can be seen, the barrier heights and the leakage current values vary in a wide range, thus making a correlation between these electrical parameters not straightforward. In fact, some works report on the formation of the W(5212) and W(34) ant phase in the metal layer. The bank of inorganic structures ICSD has been used to identify the diffraction peak of WC (ICSD No5212) and W2C (ICSD No619097). As a matter of fact, some works report on the formation of the W2C phase after thermal annealing [51] and, in general, on the possible formation of other W-C phases at temperatures higher than 500 ◦C [52].

Figure 5. XRD patterns of WC/AlGaN/GaN system after metal deposition (as-deposited) and after annealing at 400 ◦C and 800 ◦C.
Figure 6. Bright field TEM micrographs in cross section for WC/AlGaN/GaN contacts (a) as-deposited and annealed at temperature of (b) 400 °C and (c) 800 °C. (d) The electron energy loss spectra related to the signal of oxygen acquired in the regions 1, 2 and 3 of the as-deposited and annealed WC layer.

characteristic oxygen-related edge at 532 eV is clearly visible in the as-deposited and 400 °C annealed WC layer, thus indicating the presence of oxygen distributed within the film.

Figure 7. Schematic representation of the evolution of the WC layer during annealing at 800 °C.

Oxygen incorporation into the WC layer represents a long standing problem in deposited WC layers [53, 54]. Several techniques have been used to produce WC layers, such as
magnetron sputtering [55], mixing W and C plasma streams [56] or evaporation [57]. Indeed, the difficulty to obtain pure carbide films relies on the high affinity of WC for oxygen, which can often be revealed inside the deposited WC films [37, 58, 59]. Instead, the oxygen edge could not be detected in the EELS spectra collected in the WC layer annealed at 800 °C. A possible explanation can be found in the diffusion of the oxygen toward the surface and AlGaN interfaces during the annealing treatment at a very high temperature (800 °C). In this scenario, the oxygen reaching the AlGaN interface leads to the formation of the oxygen rich W-O-C layer detected by TEM. Despite some works proposing a plasma oxygen surface cleaning prior to metal deposition in order to reduce the leakage current in AlGaN/GaN Schottky contacts [60–62], the presence of oxygen at the interface with the AlGaN is more often the cause of high reverse current and early stage degradation in AlGaN/GaN HEMTs [63]. In fact, the oxygen contamination is often indicated as the origin of a high concentration of shallow donors at the AlGaN surface [64, 65]. Figure 7 displays a schematic representation of the evolution of system, to explain the correlation between the electrical behaviour with the structural and compositional properties of the WC/AlGaN contact during annealing. In particular the degradation of the current–voltage characteristics in forward and reverse bias can be correlated to the modification of the WC/AlGaN interface after annealing at high temperature (800 °C), with the formation of a W-O-C containing layer. The presence of W2C grains detected inside the films, which are far from the interface, plays probably no role on the electrical characteristics of the diodes. On the other hand, the oxygen desorption from the WC layer can explain the decrease of the film resistivity observed upon annealing.

4. Conclusion

In conclusion, the evolution of the electrical and structural properties of WC Schottky contacts on AlGaN/GaN heterostructures has been investigated by cross correlating different analyses. The WC layer exhibited a smooth morphology even after annealing at 600 °C, which increased at higher temperatures (800 °C). As-deposited WC Schottky contacts exhibited a barrier height of 0.82 eV and a reverse leakage current of 4.1 × 10−4 A cm−2 at VR = −10 V, which is a promising trade-off for Au-free Schottky contacts on AlGaN/GaN heterostructures. Their electrical behaviour remains stable after annealing at 400 °C. The Schottky barrier height, determined from the forward characteristics of the diodes, decreased to 0.56 eV after annealing at 800 °C, thus being accompanied by an increase of the reverse leakage current. A microstructural and chemical analysis revealed the presence of oxygen in the WC layer, which accumulates in a 2–3 nm thick W-O-C layer at the interface with AlGaN after the annealing at 800 °C. Moreover, XRD showed the formation of W2C grains within the film. This scenario and in particular the presence of the oxygen content at the AlGaN surface can be the main origin of the decreased barrier height and the increased leakage current at 800 °C annealed Schottky diode. These results provide useful insights for the practical application of this Au-free metallization as Schottky contact for AlGaN/GaN heterostructures.

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