Formation and preferential orientation of Au-free Al/Ti-based ohmic contacts on different hexagonal nitride-based heterostructures

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Formation and preferential orientation of Au-free Al/Ti-based ohmic contacts on different hexagonal nitride-based heterostructures

I. INTRODUCTION

Power-conversion systems are a crucial segment of the worldwide electric network. Nitride-based semiconductors are currently being developed for innovative power-transistor applications. A nitride-based heterostructure enables high electron mobility and high-voltage compatibility, which results in devices that are far superior to Si-based power transistors.

A nitride-based high-electron-mobility transistor (HEMT) relies on a two-dimensional electron gas (2DEG) that is formed by the spontaneous polarization at the interface between gallium-, aluminum-, or indium-nitride-based layers of different composition. Although the state-of-the-art HEMT devices are based upon an AlxGa1−xN/GaN heterostructure, a further improvement can be achieved with InxAl1−xN/GaN or AlN/GaN heterostructures due to a larger polarization field. Unfortunately, the wide bandgap of the top nitride layer such as AlxGa1−xN, ALN, or InxAl1−xN makes it challenging to electrically connect the 2DEG to the source and drain contacts. An increase of the Al concentration in the barrier layer of the heterostructure increases the Schottky barrier height, which is present at the metal–barrier interface, and it is, therefore, a significant barrier for the electrical connection of the 2DEG to the source and drain terminals.

Wide-bandgap nitride semiconductors are currently in development for high-power electronic applications. Compositional layered heterostructures of such nitrides result in a high polarization field at the interface, enabling a higher electron mobility, a higher power density, and a higher conversion efficiency. Further optimization of such GaN-based high-electron-mobility transistors can be achieved by evolving from a top AlxGa1−xN barrier toward AlN or even InxAl1−xN. An ongoing challenge in using such hexagonal nitride semiconductors is the formation of a low-resistive, Au-free, ohmic contact far below 1 Ω mm. In this paper, we investigate the formation of ohmic contacts by Ti–Al–TiN-based metalization as a function of different annealing temperatures (up to 950 °C), Ti–Al ratios (from 15 up to 35 at. %) and nitride barrier composition (AlxGa1−xN, GaN, AlN, and InxAl1−xN). Contacts processed on AlxGa1−xN/GaN, and AlN/GaN heterostructures result in low contact resistance of, respectively, 0.30 and 0.35 at. %) and nitride barrier composition (AlxGa1−xN, GaN, AlN, and InxAl1−xN). The observation that the solid-phase reaction is identical on all investigated nitrides suggests that a further decrease of the contact resistance will be largely dependent on an optimization of the nitride barriers themselves.
not straightforward to transfer the metalization scheme from an AlGaAsN - or GaN-based technology to AlN or InAlN -based HEMTs.

Two strategies are required to achieve low-resistive ohmic contacts. First, the contact stack needs to be optimized in terms of composition and annealing conditions. Second, the underlying nitride barrier also needs to be optimized with respect to its thickness and composition, e.g., Al and In concentrations.\(^6\) For example, Takei et al.\(^7\) demonstrated that the contact resistance depends on the AlGaAsN barrier thickness. However, their study shows that the optimal annealing temperature for each specific AlGaAsN barrier was identical and that optimal annealing conditions are unaffected by the changes in the barrier. As such, a first step to engineer the optimal contact process conditions can be performed independently of the barrier structure optimization. However, a question remains whether or not the contact formation is altered upon drastically changing the barrier composition (e.g., AlGaAsN vs InAlN). Therefore, the formation mechanism of low-resistive ohmic contacts needs to be studied with a direct comparison between different nitride-based barriers. The contact formation itself can be influenced by numerous parameters, such as annealing conditions, initial composition of the metal stack, and protective capping layer. By consequence, one requires to control those parameters in order to understand the formation of ohmic contacts.

Nowadays, the most commonly reported contacts stacks on AlGaAsN /GaN heterostructures contain elements with a low metal work function such as Ti, Ta, or Al.\(^1\)–\(^7\) These contact stacks require a protective capping layer to prevent out-diffusion and undesired oxidation from the top of the contact stack. A large amount of the demonstrated low-resistive contacts rely on a Au-based capping layer to avoid oxidation of the contact.\(^7\) However, Au is an undesired contaminant in an industrial Si-CMOS fab. The formation of Au-free ohmic contacts is, therefore, a critical issue and is of utmost importance for high-volume and low-cost production of HEMT devices in Si-CMOS fabs. Capping layers such as W, TiN, or TiN,\(^4\),\(^1\) therefore, are highly interesting candidates for the formation of industrially-relevant ohmic contacts. Alternatively, Ni or Pt as capping layer can also be effective,\(^1\)–\(^4\)–\(^7\) but these noble metals are also seen as contaminants in Si-CMOS fabs.

We previously reported on the Ti–Al–TiN contact stack\(^4\) for AlGaAsN /GaN heterostructures. We demonstrated that the Ti–Al ratio defines the optimum annealing temperature for ohmic contact formation,\(^8\) which confirms earlier insights on similar metal contact schemes.\(^8\) The ratio can be either Ti-rich (the Ti-to-Al ratio of > 25 at.%) or Al-rich (the Ti-to-Al ratio of < 25 at.%), a differentiation which is based on the binary Al–Ti phase diagram.\(^8\) Each Ti–Al ratio corresponds with an optimum annealing temperature. Ti-rich contacts require an annealing temperature above 850 °C to reach a low contact resistance, whereas Al-rich contacts require an annealing temperature below 600 °C. Unfortunately, most papers are limited in the number of samples with different Ti-to-Al ratios in the metal stack or limited in the number of different annealing temperatures. A study that systematically compares these contact stacks as a function of the Ti-to-Al ratio, annealing temperature, and of the heterostructure barriers would be of great interest. In order to improve our physical understanding of such contacts, this paper reports on the phase transitions of Ti–Al-based contact schemes for contacts between 15 and 35 at. % Ti and for temperatures up to 900 °C and on different nitride barriers.

We first show that the Ti–Al–TiN contact stack can indeed result in low-resistive ohmic contacts on different nitride barriers, although the contact performance is slightly better on AlGaAsN - than on AlN- or InAlN -based heterostructures. We then provide a detailed overview on the observed crystalline phase formation of the different Ti–Al–TiN contact stacks during annealing by comparing Al-rich contacts with Ti-rich contacts. The investigation of the formed crystal structures allows us to correlate the contact compositions with the lowest resistive ohmic contacts. Intriguingly, the formed contacts have the same crystalline solid-phase reactions and preferential alignment in respect to the epitaxial barriers, independent on that barrier composition. Therefore, the observed differences in electrical performance of the contacts can be attributed to the intrinsic differences of the nitride barrier properties.

II. EXPERIMENTAL

The GaN-based heterostructures were grown on 6-in. (111)-oriented, Si wafers by metal organic chemical vapor deposition (MOCVD). Several nitride barriers were investigated: AlGaAsN , AlN, GaN, or InAlN . An in situ grown SiN layer was used as a protective capping layer. Ti–Al–TiN contacts were deposited through magnetron sputtering [Physical Vapour Deposition (PVD)] on nitride-based heterostructures in two different deposition strategies. The first strategy is a standard PVD deposition of metal stacks with a fixed, uniform Ti–Al ratio across the wafer. The thickness of each individual layer was determined by x-ray reflectivity measurements after deposition. These stacks were deposited uniformly on AlGaAsN , AlN, GaN, InAlN and SiN and capped with a reactively-sputtered protective TiN layer without breaking the vacuum.

A second approach is the PVD deposition of a linear Ti–Al compositional gradient on top of an AlGaAsN /GaN-heterostructure wafer. The deposition flux can be tailored by positioning masks in-between the sputter target and the wafer. The deposited layers then have a controlled, linear variation of composition across the wafer.\(^9\) On such a combinatorial gradient, the Ti-to-Al ratio linearly increases as a function of position on the wafer, as displayed in Fig. 1. As such, metal stacks could be systematically investigated with Ti–Al ratios from 15 up to 35 at. % Ti on a single wafer. This approach enables exploring different compositions in parallel on a single wafer, which enables a cost-effective screening on these expensive substrates and assures identical processing conditions such as chemical cleaning or deposition background pressure for all contacts. These stacks were subsequently capped with a TiN layer without breaking the vacuum.

Both the gradient-deposited wafers as well as uniformly-deposited wafers were then mapped for electrical and morphological properties by optical profilometry (Veeco NT9080 surface profiler) and scanning electron microscopy (SEM) after rapid thermal annealing (RTA) under N\(_2\) ambient at different temperatures for 90 s. The electrical characteristics of the contacts were assessed by transmission line method (TLM) structures from which the ohmic contact resistance (R\(_c\)) and the 2DEG sheet resistance (R\(_{sh}\)) were extracted.
Lab- and synchrotron-based x-ray diffraction (XRD) measurements were used to investigate the crystal structure of the formed contacts. The first part of our results, Sec. III B, investigates the solid-phase reaction (SPR) of the deposited metal stacks as a function of temperature. The samples were heated at a constant rate of $1{\degree}C/s$ up to 950 $^\circ$C in a purified N₂ ambient, while being monitored through XRD using a laboratory setup. A thermocouple installed just below the sample allows us to monitor the temperature during the ramp anneal. A CuKα ($\lambda = 1.54$ nm) x-ray source illuminated the sample under a fixed incident angle, while the Bragg diffraction was monitored using a linear detector in a Bruker D8 system, which covered the diffraction angles $2\theta$ from 30$^\circ$ up to 50$^\circ$ (i.e., inter-planar distances from 2.85 down to 1.8 Å).

As XRD is also an orientation-sensitive technique, the fixed setup geometry used in these in situ measurements will only detect crystal planes that are oriented (nearly) parallel to the sample surface. This feature was used to avoid over-exposure of the detector due to the highly-intense GaN(002) diffraction peak by introducing a small out-of-plane tilt of the sample of only a few degrees.

A further numerical improvement of the data's signal-to-noise ratio was achieved by subtracting the minimum intensity recorded by each detector pixel (thus correcting for a constant background intensity originating from, e.g., fluorescence).

The fixed-geometry limitations of the in situ setup makes it challenging to study the epitaxial nature of the formed contact phases. The preferential alignment of the contact grains is an important aspect of the microstructure of the ohmic contacts, since it is known that the best ohmic contacts often correspond to an epitaxial alignment. Ex situ XRD pole figures are, therefore, a perfect addition in order to inspect the preferential orientation of the crystal grains. For obtaining an XRD pole figure, the diffracted x-ray signal is recorded while the sample is being tilted and rotated both out-of-plane with respect to the surface normal (defined as the elevation angle $\chi$) and in-plane (azimuth angle $\phi$). The incidence angle of the x rays with the sample is set to the value $\theta$, and the angle between the x rays and the detector is set to $2\theta$ such that the Bragg law is valid for a desired inter-planer distance $d_{hkl}$. The resulting dataset is then represented as an intensity map in polar coordinates [Fig. 2(a)]. A high intensity can thus be translated into particular orientations of the corresponding crystalline phase within the probed area of the sample. XRD-pole figures are able to evaluate the crystalline alignment within the x-ray footprint, e.g., order of $250 \times 250 \mu m^2$, which is an important difference with (high-resolution) transmission electron microscopy (TEM) imaging, which would evaluate the crystalline alignment of a very small area (order of $100 \times 100 \mu m^2$).

These ex situ XRD measurements were acquired at the DifAbs beamline of the SOLEIL Synchrotron (Gif-sur-Yvette, France). The incidence x-ray beam was monochromatized using a double-crystal Si(111) monochromator to a wavelength of $\lambda = 0.68$ nm (i.e., an energy of $\approx 18$ keV) with a beam Full Width Half

![Figure 1](https://scitation.aip.org/content/aip/journal/jap/127/215701-3)

**FIG. 1.** The Ti concentration of a binary Ti–Al layer monotonously increases as a function of position across the wafer.

![Figure 2](https://scitation.aip.org/content/aip/journal/jap/127/215701-3)

**FIG. 2.** X-ray diffraction intensities of an Al$_x$Ga$_{1-x}$N /GaN heterostructure on top of a Si(111) wafer. (a) The diffraction intensities of both Si(220) and GaN (102) planes while tilting the sample both in- and out-of-plane (i.e., azimuth $\phi$ and elevation $\chi$, respectively, see the inset) for a fixed incident and diffraction angles (respectively, $\theta$ and $2\theta$). (b) The diffraction intensities were displayed as a function of both diffraction- and elevation angles (the displayed intensities were averaged along the in-plane, azimuth, tilting angle $\phi$).
Maximum (FWHM) of 250 × 250 μm². The sample was mounted on a 6-circles diffractometer (kappa geometry), with a hybrid pixel area detector (XPAD detector²⁰) on a 6-circles diffractometer (kappa geometry), with a hybrid area detector of two sequential steps in the elevation angle of 10° up to 32° (i.e., inter-planar distances from 2.82 down to 1.3 Å). The area detector acquired in a so-called fly-scan recording mode, acquiring a snapshot every 100 ms during a continuous in-plane rotation of the samples’ azimuth. The use of the area detector enables us to acquire diffraction from the full range of rotation of the samples, thus, the sample only needs to rotate for a few discrete steps in the elevation angle of 10°. The discrete step size of 10° in the second part of our work (Sec. III C), we discuss the preferential orientation of the crystalline grains directly after deposition and after a RTP-based heat treatment to 400, 500, 600, 700, 800, 900 °C. We display the obtained data in two ways: pole figures show the geometry-dependence for specific diffraction angles (i.e., specific crystalline planar distance), whereas color-intensity plots allow us to determine the preferential alignment for a broad range of diffraction angles. Figure 2 shows such plots for a AlₓGa₁₋ₓN / GaN reference sample. As Si(111) and AlₓGa₁₋ₓN (001) are oriented in the same direction, one can further calculate the expected elevation angles χ between different diffraction planes. From these angles, one can derive the alignment of preferentially-oriented crystals, as tabulated in Table I. The supplementary material contains a concise introduction to these plots.

### III. RESULTS AND DISCUSSION

#### A. Electrical performance of ohmic contacts formed on AlₓGa₁₋ₓN, AlN, and InₓAl₁₋ₓN barriers

The reported contact resistances R_c of the Ti–Al-based, Au-free, ohmic contacts on AlₓGa₁₋ₓN barriers are displayed in Fig. 3 and includes data from this study as well as the literature. The graph shows that there are two regions of interest (ROI) to obtain low-resistive ohmic contacts on AlₓGa₁₋ₓN-barriers. Contacts below 1 Ω·mm can be formed either at relatively low

![Graph showing contact resistances on AlGaN heterostructures, incl. values from literature](image)

**FIG. 3.** The average values of contact resistance of Ti–Al–TiN metal stacks with various Ti–Al ratios (excluding the capping layer) and annealing temperatures on AlₓGa₁₋ₓN heterostructures. The data show two regions-of-interest (ROI) in order to form low-resistive ohmic contacts with the Ti–Al–TiN contact scheme on AlₓGa₁₋ₓN: near 15–20 at. % for 550 – 600 °C or 25–30 at. % upon annealing up to 850 °C. This work explored the contact formation within the ROI for InAIN and AlN, as indicated by the arrows. The shapes of the icons correspond with the origin of the datapoint: x, our data on AlₓGa₁₋ₓN; +, AlN or InₓAl₁₋ₓN (this work); *, Ref. 5; ■, Ref. 13; †, Ref. 10; *′, Ref. 26; †′, Ref. 11; †′′, Ref. 9; †′′′, Ref. 25; x′, Ref. 6; x′′, Ref. 26; +′, Ref. 27.

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**TABLE I.** Crystalline data on the observed epitaxial structures of as-deposited Ti, Al, and TiN. Those planes that were observed parallel to Si(111) (i.e., elevation angle χ = 0°) are displayed in bold. The diffraction angles for the two different wavelengths used in this article are also displayed.

<table>
<thead>
<tr>
<th>Compound</th>
<th>Plane (Å)</th>
<th>d-spacing (Å)</th>
<th>2θ_exp (°)</th>
<th>2θ_th (°)</th>
<th>Calc. χ (°)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti</td>
<td>(002)</td>
<td>2.34</td>
<td>38.4</td>
<td>16.7</td>
<td>0</td>
</tr>
<tr>
<td></td>
<td>(100)</td>
<td>2.56</td>
<td>35.1</td>
<td>15.3</td>
<td>90</td>
</tr>
<tr>
<td></td>
<td>(101)</td>
<td>2.24</td>
<td>40.2</td>
<td>17.4</td>
<td>61.4</td>
</tr>
<tr>
<td></td>
<td>(102)</td>
<td>1.72</td>
<td>53.0</td>
<td>22.7</td>
<td>42.5</td>
</tr>
<tr>
<td></td>
<td>(103)</td>
<td>1.33</td>
<td>70.6</td>
<td>29.6</td>
<td>31.4</td>
</tr>
<tr>
<td>Al</td>
<td>(111)</td>
<td>2.34</td>
<td>38.5</td>
<td>16.7</td>
<td>0</td>
</tr>
<tr>
<td></td>
<td>(200)</td>
<td>2.02</td>
<td>44.7</td>
<td>19.3</td>
<td>54.7</td>
</tr>
<tr>
<td></td>
<td>(220)</td>
<td>1.43</td>
<td>65.1</td>
<td>27.5</td>
<td>35.3</td>
</tr>
<tr>
<td>TiN</td>
<td>(111)</td>
<td>2.45</td>
<td>36.6</td>
<td>16.0</td>
<td>0</td>
</tr>
<tr>
<td></td>
<td>(200)</td>
<td>2.12</td>
<td>42.6</td>
<td>18.5</td>
<td>54.7</td>
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</tbody>
</table>
temperature, e.g., 550–600 °C, for Ti-to-Al ratios of 15–20 at. % or after annealing at higher temperature, e.g., 850–950 °C, for 25–35 at. %. We will further discuss the phase formation as a function of Ti-to-Al ratios. Ti-to-Al ratios below 25 at. % will be addressed as Ti-rich, and Ti concentrations above 25 at. % will be addressed as Ti-rich. Our previous work reported that a deviation from these regions-of-interest results in a degradation of the ohmic contact resistance.

Similar to the contacts realized on Al$_x$Ga$_{1-x}$N /GaN heterostructures, Al-rich and Ti-rich contacts were processed on AlN and In$_x$Al$_{1-x}$N barriers. It is intriguing that those same contact stacks that result in a low-resistive ohmic contact on Al$_x$Ga$_{1-x}$N barriers also perform well on AlN barriers. However, a further improvement to the results can be expected through an optimization of the barriers, e.g., with recessing (not performed here). Higher contact resistances were measured with metal stacks processed on In$_x$Al$_{1-x}$N barriers.

The above classification was further investigated in terms of phase formation, crystal structure, and crystal alignment between the contact stacks and the nitride barriers (Al$_x$Ga$_{1-x}$N , AlN, GaN, and In$_x$Al$_{1-x}$N ).

B. Solid-phase reaction of Ti-Al-based contacts

A total of 10 different positions were diced out of the deposited Ti–Al–TiN compositional gradient on the Al$_x$Ga$_{1-x}$N / GaN heterostructure, and each sample was measured by in situ x-ray diffraction (XRD) during a heating procedure. These measurements allow us to identify the phase sequence during the contact formation and to determine the exact temperatures at which phase transitions are observed. From these ten samples, we selected two representative measurements to be displayed for, respectively, 16 and 29 at. % Ti (Figs. 4 and 5). These two concentrations are representative for the two main phase sequences observed for the Ti–Al–TiN contact stack. We will start to discuss the phase sequence in detail for these two Ti-to-Al ratios in Secs. III B 1 and III B 2. Thereafter, a summary is provided on the observed phase sequence for all measured Ti-to-Al ratios in Sec. III B 3. We finalize this section on the solid-phase reaction of Ti–Al–TiN contact stacks by comparing the solid phase reaction on different nitride barriers in Sec. III B 4.

1. Solid-phase reaction of an Al-rich metal stack on AlGaN (16 at. % Ti)

The XRD-patterns in Fig. 4 are displayed as a function of increasing temperature for a sample with a Ti-to-Al ratio of 16 at. % Ti. At room temperature, two intense XRD peaks are present: a peak near 2θ = 36.8°, originating from TiN(111), and a peak near 2θ = 38.5°. The latter can be related to both Ti(002) and Al(111), two crystal planes that have coinciding diffraction peaks due to a similar crystal plane spacing (see also Sec. III C 1 for an in-depth discussion based on XRD pole figures). The Ti (002)/Al(111) diffraction peak remains present up to a temperature of ≈665 °C, but already loses half of its intensity at a temperature of ≈530 °C. When the Ti(002)/Al(111) diffraction peak has its first major intensity reduction, it is clear from the XRD recording that this is because Ti and Al are consumed in order to form a tetragonal Al$_3$Ti compound [as seen by the occurrence of the (118) and (008) peaks]. As the as-deposited layer contains insufficient Ti to fully convert all Al into Al$_3$Ti, we can suppose that the remaining low-intense diffraction peak at 2θ = 38.5° can be attributed to the remaining Al above 530 °C. At 660 °C, we observe a complete decrease of this remaining Al(111) diffraction peak, and a new, highly-intense peak arises at 2θ = 42.3°, which we correlated to a transformation of the Al$_3$Ti phase to the high-temperature variation of the crystal structure. The tetragonal Al$_3$Ti phase is known...
to occur in two different crystal structures with identical stoichiometry\textsuperscript{15,28,29} and is denoted in this article as a low-temperature (Al\textsubscript{3}Ti\textsubscript{l}, observed in Fig. 4 below 665 °C) and a high-temperature (Al\textsubscript{3}Ti\textsubscript{h}, observed in Fig. 4 above 665 °C) structure. The unit cell of Al\textsubscript{3}Ti\textsubscript{l} can be considered in a first approximation as four stacked unit cells of Al\textsubscript{3}Ti\textsubscript{h} [with \(a_0 (0.3877 \text{ nm}) \approx a_6 (0.3849 \text{ nm})\) and \(c_1 / 4 (0.8458) \approx c_6 (0.8104 \text{ nm})\)]. The above observations show that the disappearing Al peak near 660 °C coincides with a significant change in the Al\textsubscript{3}Ti crystal structure. Some interesting observations will be made by discussing these measurements more detailed in the following paragraphs.

First, one notices that only a few diffraction peaks can be observed for each phase. We will show in Sec. III C that this originates from preferential orientation of these layers. Second, it is yet an open question where the excess of Al reacts into above 660 °C, as there is no sufficient amount of Ti to convert all Al into Al\textsubscript{3}Ti. The Ti–Al binary phase diagram\textsuperscript{15,28} shows that the Al\textsubscript{3}Ti structure is the most Al-rich compound possible, and it is a line phase with a fixed 3:1 ratio of Al to Ti. If elemental Al has not formed a new compound above a temperature of 660 °C, the binary phase diagram suggests that this Al will melt and form a liquid, a material state which is not observable through XRD. Indeed, when the sample cools down after annealing, as seen in Fig. 4(b), one does notice that an Al(200) peak appears at \(2\theta = 44°\). This confirms the presence of an Al liquid at high temperatures that solidifies during cooling. Note that the orientation of the solidified Al after annealing is different from the as-deposited Al in the beginning of the measurement. Third, the Ti(111) peak of the protective capping layer remains present throughout the measurement but its intensity is not constant. Numerical analysis of the XRD intensities (Fig. 6, blue curve indicates the currently discussed contact stack) shows a decline of the TiN (111) diffraction intensity of over 40% above 800 °C, in favor of an increase in the intensity of Al\textsubscript{3}Ti. A shift in diffraction angle of TiN(111) is also observed toward lower diffraction angles, i.e., showing that the TiN lattice expands. This shift in the diffraction angle was previously reported as typical behavior, indicating a transformation toward a more N-rich composition.\textsuperscript{30,31} As the Al\textsubscript{3}Ti peak is simultaneously increasing in intensity, it indicates that the liquid Al reacts with the capping layer and is partially transformed into Al\textsubscript{3}Ti.

Comparing the in situ XRD data with the electrical data displayed in Fig. 3 suggests that at 16 at. % Ti, the region of interest for low-resistive ohmic contacts is limited due to the melting temperature of Al. Above this temperature, the contact drastically degrades as seen by a change in the Al’s plane alignment, a transition of Al\textsubscript{3}Ti crystal structure and a reduced integrity of the TiN capping layer.

2. Solid-phase reaction of a Ti-rich metal stack on AlGaN (29 at. % Ti)

For a Ti-to-Al ratio above 25 at. % Ti in the as-deposited stack, we observed a different phase sequence (Fig. 5). The as-deposited diffraction patterns are similar and show the presence of Ti, Al, and TiN. Now, the presence of Al is limited up to 538 °C, when Al\textsubscript{3}Ti is formed. Near 800 °C, the formed Al\textsubscript{3}Ti is accompanied by two other compounds: ternary Ti\textsubscript{2}AlN and binary Al\textsubscript{2}Ti. A crystal transition of the Al\textsubscript{3}Ti phase, which was clearly observable for the lower Ti concentration regime above 660 °C, does not occur: the initial Al\textsubscript{3}Ti (118) and (008) peaks are stable throughout the measurement. The high-temperature Ti\textsubscript{2}AlN phase only shows the (006)-peak, although the measured range of diffraction angles

![Stability of the TiN capping layer of Ti\textsubscript{x}-Al\textsubscript{1-x}-TiN contact stacks](image-url)
would also include higher intensity peaks such as (100) and (103) peaks. Again, this is an indication of crystallographic preferential orientation, i.e., that there is a geometrical link between the epitaxial Al\textsubscript{x}Ga\textsubscript{1-x}N substrate and the formed ohmic contacts, which limits the observed orientations of the crystal structures.

From Fig. 5, we can observe that excess of Ti reacts into a combination of Al\textsubscript{3}Ti and Al\textsubscript{2}Ti so that both Ti and Al are fully consumed. No consumption of the TiN capping layer was observed through any reduction of the diffraction intensity of the TiN(111) peak for these higher Ti concentrations (red and green lines in Fig. 6).

3. Solid-phase reaction diagram

Similar measurements as discussed above were performed on all other compositions present in the compositional Ti–Al gradient (15 to 35 at. % Ti) on an AlGaN/GaN heterostructure. A summary is plotted in Fig. 7. The figure allows us to identify two different regimes of phase sequence

- Below 25 at. % Ti, also denoted here as an Al-rich contact stacks, Ti + Al → Al\textsubscript{3}Ti + Al
- Above 25 at. % Ti, also denoted here as a Ti-rich contact stacks, Ti + Al → Al\textsubscript{3}Ti + Ti\textsubscript{2}AlN + Al\textsubscript{2}Ti

The observed phases in Fig. 7 illustrate the differences between a bulk phase diagram, which represents the phases in thermal equilibrium at elevated temperatures of the binary Ti–Al system, and the constructed thin-film phase diagram of Ti–Al stacks on an Al\textsubscript{x}Ga\textsubscript{1-x}N barrier. The latter shows the phases, which are formed during a ramped thermal anneal and in close proximity of an epitaxial nitride barrier. Similar to the bulk phase diagram\textsuperscript{15,28} one can refer to the atomic composition of the Al\textsubscript{3}Ti compound as a reference to define a Ti-rich or an Al-rich metal stack. A remarkable consequence of the occurrence of these two regimes can be seen by comparing the surface morphology of these contacts after an RTP-anneal up to 800°C. For metal stacks with an excess of Al, the molten Al re-solidifies during cooling. For such samples, we have observed severe cracks and holes in the surface of the samples [Fig. 8(a)], while for samples with an excess of Ti, the top surface remains relatively smooth after anneal. In Fig. 8(c), the average roughness parameter \( R_a \) follows the same two-regime behavior as observed with the XRD measurements. The roughness is the highest for the lowest Ti concentrations (around 45 nm, but quickly decreases with increasing titanium content, until it converges to a minimum value of \( R_a = 8.9 \) nm above concentrations of 25 at. % Ti). Previously, Constant \textit{et al.} reported that samples below 25 at. % Ti result in bad contact resistance when annealed at 850°C, an observation that was correlated with a poor sheet resistivity and morphology.\textsuperscript{4}

We can then compare the obtained thin-film phase diagram in Fig. 7 with those temperatures and compositional combinations that result in low-resistive contact stacks in Fig. 3. The regions of low ohmic resistance are indeed situated at regions with a unique area on the phase diagram. In the Al-rich regime, low-resistive ohmic contacts are formed for temperatures between 550 and 600°C just below the melting temperature of Al. This corresponds with a

**FIG. 7.** A schematic overview of all crystalline phases as observed through in situ XRD of the Ti–Al–TiN contact stack on an AlGaN/GaN heterostructure. The average composition of each sample is indicated by a horizontal, dashed, black line at the arrow positions.

**FIG. 8.** Top: SEM images of the top morphology of the investigated samples show a clearly rougher surface for e.g., Al-rich [point (a)] samples than for Ti-rich samples [point (b)]. Bottom: Ex situ morphology assessment was performed by optical profilometry on samples annealed up to 800°C and show a drastic change when increasing the Ti concentration above 25 at. %.
4. Variation of nitride barriers

Similar metal stacks of Ti–Al–TiN with relatively low (e.g., 16 at.%) and high (e.g., 26–29 at.%) Ti-to-Al ratios were deposited on several nitride layers (hexagonal) Al\textsubscript{1-x}Ga\textsubscript{x}N, GaN, AlN, and In\textsubscript{1-x}Al\textsubscript{x}N and (amorphous) Si\textsubscript{3}N\textsubscript{4}. In situ XRD measurements on these samples are displayed in Fig. 9, clearly showing that the phase sequence is identical for the same metal stack on different nitride layers with the sole exception of the depositions on (amorphous) Si\textsubscript{3}N\textsubscript{4}. Here, no Ti\textsubscript{3}AlN (006) peak was observed upon annealing the samples, while the (101) peak was observed (at a diffraction angle of 35° as seen in a second measurement window, not shown).

C. Crystalline orientation of Ti-Al-based contacts

1. As-deposited metal stack on AlGaN

While discussing the in situ XRD measurements, we have noticed that not all diffraction peaks were observed for some compounds. For example, in Fig. 4, one could expect some additional diffraction peaks: Al(200) (d = 2.02 Å, 2θ = 44.8°), TiN(200) (d = 2.12 Å, 2θ = 42.6°), Ti(101) (d = 2.24 Å, 2θ = 40.2°), and Ti(100) (d = 2.56 Å, 2θ = 35.0°). The reason for this peculiarity is related to the measurement geometry of the in situ XRD setup. As the X-ray source, the sample and the detector are in fixed geometry, the setup only probes those planes which are nearly parallel to the sample’s surface. To eliminate this blind spot in our XRD measurements, we have performed 3D XRD color maps for samples annealed to specific temperatures. In Fig. 10, we display the diffraction intensity as a function of a diffraction angle 2θ and pattern elevation angle χ (note that the diffraction angle 2θ in our XRD pole figures does not correspond with the diffraction angle from our in situ XRD, due to a different x-ray wavelength). From these plots, we can determine whether a crystal has a random orientation in respect to the substrate (which results in a vertical line) or a preferential alignment to the substrate (which results in a local spot).

We can immediately recognize the features related to the Si substrate and the Al\textsubscript{1-x}Ga\textsubscript{x}N layers, as indicated by the circles and which were previously discussed in the experimental section with Fig. 2. The Ti, Al and TiN diffraction patterns are limited at specific values of elevation angle χ, which indicates a preferential orientation of those crystals. One can then use the known geometry of the crystal unit cells to determine the specific alignment of those crystal grains. For example, if one considers a hypothetical situation where only Al(111) planes are parallel to the samples’ surface, then all other crystal planes will also have a specific orientation in respect to the samples’ surface. This orientation can be calculated and is equal to the inclination angle with the Al(111) plane, as defined by the crystal structure of Al. Indeed, the geometric angles between the planes of Al(111) on the one hand and Al(111-1), (200), or (220) on the other hand are, respectively, 70.5, 54.7, and 35.3° (see also Table 1). Therefore, if all Al(111) planes are parallel to the samples surface, then e.g., the Al(200) diffraction would only be observed at elevation angle χ = 54.7°, which is the case. Note that this diffraction dataset does not include diffraction of Al at any other χ values, thus eliminating any other orientation for all Al grains probed by the x-ray footprint. We can, therefore, conclude from these measurements that every compound in the as-deposited Ti–Al–TiN stack is preferentially aligned with the Al\textsubscript{1-x}Ga\textsubscript{x}N-barrier.

Two types of preferential orientations are possible: epitaxial-alignment and fiber alignment. To differentiate between these two alignments, one need to take a closer look to the individual pole figures in Fig. 11. Here, we observe that the diffraction of Ti, Al, and TiN are also limited in the in-plane angle φ-angle, thus showing that the as-deposited layers have an epitaxial alignment. The specific alignments are defined through

\[ Ti(002)\parallel Al\textsubscript{1-x}Ga\textsubscript{x}N(002) \quad \text{and} \quad Ti(100)\parallel Al\textsubscript{1-x}Ga\textsubscript{x}N(100), \]
\[ Al(111)\parallel Al\textsubscript{1-x}Ga\textsubscript{x}N(100) \quad \text{and} \quad Al(1 – 10)\parallel Al\textsubscript{1-x}Ga\textsubscript{x}N(100), \]
\[ TiN(111)\parallel Al\textsubscript{1-x}Ga\textsubscript{x}N(002) \quad \text{and} \quad TiN(1 – 10)\parallel Al\textsubscript{1-x}Ga\textsubscript{x}N(100). \]

These alignments were observed both for as-deposited stacks with 16 and 27 at. % Ti on Al\textsubscript{1-x}Ga\textsubscript{x}N barriers, even after an RTP-based anneal up to 400 °C. Those stacks that were annealed up to 500 °C result in different XRD-pole figures and are dependent on the initial Ti–Al ratio, as expected from our in situ XRD-based phase identifications.

2. Annealed Al-rich metal stacks on AlGaN (16 at. %)

The XRD pole figures of an Al-rich contact stack with 16 at. % Ti, annealed by RTP up to 600 °C is displayed in Fig. 12. This combination of annealing temperature and Al concentration is of peculiar interest, as it was previously identified as one of the two regions for formation of low-resistive ohmic contacts. We can again recognize the diffraction peaks related to the single-crystal Si(111) wafer and the Al\textsubscript{1-x}Ga\textsubscript{x}N/GaN-based heterostructures. The diffraction spots of as-deposited Al are situated at those diffraction angles and elevation angles χ as displayed in Table 1. In addition to those epitaxial diffraction features, one now can recognize the diffraction of Al\textsubscript{1-x}Ti as a vertical line, i.e., for all values of χ, which indicates that those crystal grains are randomly-aligned. From these measurements, we can now be certain of the assumption previously made in Sec. III B 1 that Ti is first fully consumed and that residual, epitaxial Al remains present up to it’s melting temperature. Therefore, the low-resistive ohmic contact achieved at 600 °C is thus correlated to one of, or a combination of, the following conditions:

- the presence of randomly-aligned Al\textsubscript{1-x}Ti;
- the consumption of the initial Ti-layer; and
- the presence of epitaxial layer (epitaxial Al for the Al-rich region).

In Sec. III C 3, we will show that the fulfillment of only the first two conditions does not suffice to form a low-resistive ohmic contact. As such, the third condition seems to be critical to form a low contact resistance in the Al-rich regime. We propose that some
FIG. 9. In situ XRD measurements of Ti-rich as well as Al-rich contact stacks on different nitride barriers —Al$_x$Ga$_y$N, AlN, GaN and In$_x$Al$_{1-x}$N—and (amorphous) Si$_3$N$_4$. For these measurements, there is always an undesired background present due to diffraction of the hexagonal crystalline barrier (AlN, GaN, In$_x$Al$_{1-x}$N, Al$_x$Ga$_y$N) by low-intensity W-based x-rays, which is typically observed for aging lab-based Cu-x-ray sources due to W re-deposition on the Cu anode.
FIG. 10. Ex situ XRD intensity color maps of a non-annealed Ti–Al–TiN metal stack with a Ti-to-Al ratio of 16 at. % as a function of diffraction angle 2θ (λ = 0.688 nm) and elevation angle with respect to the substrate surface χ (i.e., the elevation angle). The data clearly show that Al, Ti, and TiN diffraction is only observed for specific values of χ, which is typical behavior for epitaxial or fiber-textured compounds.

FIG. 11. Ex situ XRD pole figures of specific diffraction angle 2θ (λ = 0.688 nm): 2θ = 17.3° [d = 2.29 Å; contains diffraction from AlGaN(010) and TiN(002)], 2θ = 18.7° [d = 2.12 Å; contains diffraction from TiN(200)], 2θ = 22.9° [d = 1.73 Å; contains diffraction from AlGaN(101) and Al (220)], and 2θ = 27.7° [d = 1.44 Å; contains diffraction from AlGaN(111) and Al (220)]. The data clearly show that Ti, Al, and TiN are epitaxially aligned.

FIG. 12. Ex situ XRD intensity color maps of a Ti–Al–TiN metal stack with a Ti-to-Al ratio of 16 at. % annealed up to 600 °C as a function of diffraction angle 2θ (λ = 0.688 nm) and inclination angle with the substrate surface (i.e., the elevation angle χ). The data clearly show that Al and TiN diffraction is only observed for specific values of χ, which is typical behavior for epitaxial or fiber-textured compounds. By contrast, Al3Ti diffraction is observed for all elevation angles χ, which indicates that those crystal grains are randomly-aligned. The red arrows indicate the diffraction positions where Ti was observed for a non-annealed sample.

FIG. 13. Ex situ XRD intensity color maps of a Ti–Al–TiN metal stack with a Ti-to-Al ratio of 16 at. % annealed up to 800 °C as a function of diffraction angle 2θ (λ = 0.688 nm) and inclination angle with the substrate surface χ (i.e., elevation angle). The data clearly show that Al, Al3Ti and TiN diffraction is only observed for specific values of χ, which is typical behavior for epitaxial or fiber-textured compounds.
reaction with Ti is required in combination with the presence of epitaxial Al. We can assume that the reaction of Ti can result in a cleaning at the Al$_x$Ga$_{1-x}$N barrier, where undesired contaminants are dissolved in the solid solution with Al$_3$Ti, or that the reaction of Ti can also result in out-diffusion of nitrogen from Al$_x$Ga$_{1-x}$N toward Ti and the formation of Ti–N complex and/or N vacancies in Al$_x$Ga$_{1-x}$N.

When heating an Al-rich sample above the melting temperature of Al, a transformation occurs of Al$_3$Ti into a different preferential orientation, as shown through pole-figure XRD data in Fig. 13: now the diffraction of the most-intense (103), (004), and (204) peaks of Al$_3$Ti, which were rather uniform as a function of elevation angle $\chi$ in Fig. 12, now have higher diffraction intensities near $\chi$ angles of 58°, 0°, and 45°, respectively. The diffraction spots of Al are also situated at different elevation angles: Al(111), Al(200), and Al(220) diffractions are now limited at $\chi$ 54°, 0°, and 45°. The crystal alignment of both Al$_3$Ti and (re-solidified) Al are clearly significantly different from the sample annealed at lower temperatures. An inspection of the individual pole figures in Fig. 14 shows a new crystal preferential orientation, i.e., a fiber alignment, for which the axis of rotations are determined by

$$\text{Al}(200)/\text{Al}_x\text{Ga}_{1-x}\text{N}(002),$$

$$\text{Al}_3\text{Ti}(004)/\text{Al}_x\text{Ga}_{1-x}\text{N}(002).$$

The fiber alignment of Al$_3$Ti is also evident from a cross-sectional TEM image of an Al-rich contact in Figs. 15(b) and 15(c). The planes of the Al$_3$Ti layer show a clear planar alignment parallel to the surface of the sample after an anneal up to 800°C, which is not present after an anneal up to 600°C. As a fiber alignment does not result in a systematic geometry at the interface between semiconductor and contact, in contrast to epitaxy, one can expect that a fiber-aligned contact has poorer contact resistivity values.
3. Annealed Ti-rich metal stacks on AlGaN (27 at. %)

The reciprocal color map of a Ti-rich metal stack with 27 at. % is displayed in Fig. 16 for an RTP-based anneal up to 600 °C, and Fig. 17 displays the data after an anneal up to 800 °C. At 600 °C, we observe that the Ti–Al stack has fully converted to randomly-oriented Al1Ti. So, here, we have a situation where the metal stack has formed randomly-aligned Al1Ti and has consumed the initial Ti-layer. This corresponds with two out of three conditions from Sec. III C 2 that correlated with a low-resistive ohmic contact. As the contact stack here does not result in a low-resistive ohmic contact, it appears that the presence of epitaxial Al is crucial for the contact formation.

At higher temperatures, Fig. 17 shows the formation of Ti2AlN in addition to this randomly-oriented Al1Ti. This is also the second region with ideal temperature and compositional conditions for low-resistive ohmic contact. The reciprocal space maps show that Ti2AlN, which has a hexagonal structure, is epitaxially aligned with the nitride barrier. The alignment is defined by parallel a, b and c-axes between Ti2AlN and AlxGa1-xN, as shown in Fig. 18. Cross-sectional TEM investigation shows that the Ti2AlN is formed at the interface between the AlxGa1-xN barrier and the contact stack (Fig. 19). Again we observe that the formation of a low-resistive ohmic contact occurs with the presence of randomly-aligned Al1Ti, the consumption of the initial Ti-layer and with the presence of an epitaxial layer, this time the intermediate Ti2AlN layer between the barrier and Al1Ti.
4. Phase alignment differences between nitride barriers

Pole figure measurements of a Al-rich and a Ti-rich metal stack was obtained for different barriers: Al\(_{x}\)Ga\(_{1-x}\)N , AlN and GaN. No difference could be observed between the contact stacks in terms of formed compounds and their individual alignment in respect to the underlying barrier. This includes the formation of ternary Ti\(_2\)AlN even on a pure GaN barrier. As such, we can conclude that not only the phase sequence is identical for the same contact stack on different nitrides, as observed in Sec. III B 4, but also their alignment in respect to the barrier.

IV. SUMMARY AND CONCLUSIONS

This work compares the formation mechanism of ohmic contacts with different Ti–Al ratios, as well as on different nitride barriers. Two optimal zones were identified as a function of annealing temperature as well as the Ti-to-Al ratio: 600 °C for Ti concentrations below 25 at. % and above 850 °C for Ti concentrations above 25 at. %. Remarkably, these contact stacks also result in low-resistive (<1 Ω mm) ohmic contacts on other barriers such as AlN.

In-depth investigation was performed on the solid-phase reaction of the annealed contact stack with different initial Ti–Al compositions. Our x-ray based investigation for several representative Ti-to-Al ratios showed that the phase sequence is identical on the different nitride barriers (Al\(_{x}\)Ga\(_{1-x}\)N , GaN, AlN, and In\(_{y}\)Al\(_{1-y}\)N). The preferential orientation of the formed crystalline grains shows that the epitaxial or fiber alignment of the formed ohmic contacts are identical for the different nitride barriers, as observed by full-reciprocal space XRD pole figures. The best performing contact stacks coincides with the following observations:

- the presence of randomly-aligned Al\(_3\)Ti;
- the consumption of the initial Ti-layer; and
- the presence of epitaxial layer in direct contact with the barrier.

The obtained results in this paper illustrate that the interaction between the nitride layers and the metallic contact can create an epitaxial crystal alignment of the metallic contacts. As the contact resistances can be lowered by either optimizing the contact stack or optimizing the barrier, it is clear that a similar contact stack alone is not sufficient to obtain a low-resistant contact on various nitride semiconductors. Further optimization of the advanced AlN or even In\(_{y}\)Al\(_{1-y}\)N -barriers will be required for the enhancement of the electron tunneling through the barrier.

ACKNOWLEDGMENTS

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DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

APPENDIX: PLOTTING OF ORIENTATION-RESOLVED X-RAY DIFFRACTION

An XRD pole figure displays, in polar coordinates, elevation angle \(\chi\) and azimuth angle \(\phi\), the recorded diffracted intensity for a specific diffraction angle 2\(\theta\). For example, Fig. 2(a) shows the polar plot of the diffraction near the Si(220) and GaN(102) planes (d = 1.92 Å and 1.89 Å, respectively) of a bare Al\(_{x}\)Ga\(_{1-x}\)N -based heterostructure. The spots of higher intensity (yellow) indicate that the observed planes are oriented at those very specific angles. The observed elevation \(\chi\) angles correspond with the typical geometric angles in the crystal unit cell of Si and GaN. Table II contains useful crystalline data for the epitaxial Si and GaN diffraction. Since GaN(002)//Si(111), the inclination angle for all other diffraction planes can be calculated from the reported crystal unit cells.
TABLE II. Crystalline data on the observed epitaxial structures of Si and GaN. Those planes that were observed parallel to Si(111) (i.e., elevation angle \(\chi = 0^\circ\)) are displayed in bold. The diffraction angles for the two different wavelengths used in this article are also displayed.

<table>
<thead>
<tr>
<th>Compound</th>
<th>Plane</th>
<th>d-spacing (Å)</th>
<th>(2\theta_{\text{inc}}) (°)</th>
<th>(2\theta_{\text{PV}}) (°)</th>
<th>Calc. (\chi) (°)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td>(111)</td>
<td>3.14</td>
<td>28.4</td>
<td>12.6</td>
<td>0</td>
</tr>
<tr>
<td></td>
<td>(220)</td>
<td>1.92</td>
<td>47.3</td>
<td>20.7</td>
<td>35.3</td>
</tr>
<tr>
<td></td>
<td>(311)</td>
<td>1.64</td>
<td>56.1</td>
<td>24.2</td>
<td>29.5</td>
</tr>
<tr>
<td>GaN</td>
<td>(002)</td>
<td>2.59</td>
<td>34.5</td>
<td>15.3</td>
<td>0</td>
</tr>
<tr>
<td></td>
<td>(101)</td>
<td>2.43</td>
<td>36.8</td>
<td>16.3</td>
<td>62.0</td>
</tr>
<tr>
<td></td>
<td>(102)</td>
<td>1.89</td>
<td>48.1</td>
<td>21.0</td>
<td>43.3</td>
</tr>
<tr>
<td></td>
<td>(103)</td>
<td>1.46</td>
<td>63.4</td>
<td>27.3</td>
<td>32.1</td>
</tr>
<tr>
<td></td>
<td>(100)</td>
<td>2.76</td>
<td>32.4</td>
<td>14.3</td>
<td>90.0</td>
</tr>
<tr>
<td></td>
<td>(110)</td>
<td>1.59</td>
<td>57.8</td>
<td>25.0</td>
<td>90.0</td>
</tr>
</tbody>
</table>

Note that these figures can also be used to evaluate preferential crystal orientations such as a random alignment or a fiber alignment, which would result in a uniformly increased intensity or a concentric ring of higher intensity, respectively. A fiber alignment is a preferential orientation where all grains share a fixed orientation of a specific plane, which is always parallel to the samples’ surface. Individual crystal grains can still rotate freely along the normal of this plane. A single pole figure would contain concentric circles, where the radius of the concentric circle corresponds with the elevation angle \(\chi\) between the diffraction plane and the plane that is the rotation axis.

An XRD color plot [Fig. 2(b)] is obtained by averaging the recorded intensities of a single pole-figure over the whole rotation angle \(\phi\) and was previously introduced by Gaudet et al.\(^{1,2}\) As such, one obtains an averaged intensity value for every concentric circle on the pole figure with equal elevation angles \(\chi\). By calculating these averaged values for every value of \(\chi\) and \(\theta\), one obtains a two-dimensional color plot such as that displayed in the bottom part of Fig. 2(b). In this graphical representation one can perform standard XRD phase identification by determining the positions of higher diffracted intensity as a function of \(\theta\). In addition, these diffraction maps can also be used to investigate the alignment of randomly, epitaxially, or fiber-oriented compounds. A randomly aligned sample would diffract at specific values of diffraction angle \(\theta\) and for every value of elevation angle \(\chi\). The diffraction color map would therefore display vertical lines at these \(\theta\) positions. A highly-textured, epitaxial film would only diffract at specific, and well defined, regions in diffraction angles \(\theta\), elevation angle \(\chi\), and azimuth \(\phi\). The corresponding diffraction maps, therefore, contains only local spots of higher intensity as a function of \(\theta, \chi\), and \(\phi\) (e.g., Fig. 2). Finally, one can also observe fiber orientation: an XRD color plot of a fiber texture will also correspond to local spots at specific \(\theta, \chi\), and \(\phi\) angles (Fig. 20).

**REFERENCES**