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# Characterization of GaN HEMT structures by combined SIMS & AFM approach

Running title: Characterization of GaN HEMT structures by SIMS & AFM

Running Authors: T. Spelta et al.

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## Abstract

This scientific paper describes the chemical study of different Al<sub>2</sub>O<sub>3</sub>/GaN interfaces found in new generation of high electron mobility transistors (HEMT) developed for power electronics applications. The stake for these interfaces is the limitation, ideally the absence, of an oxidized gallium layer hampering the good electrical behavior of the semiconductor. These structures have been studied through time of flight secondary ion mass spectrometry (ToF-SIMS), magnetic sims (M-SIMS) and atomic force microscope (AFM) analyses.

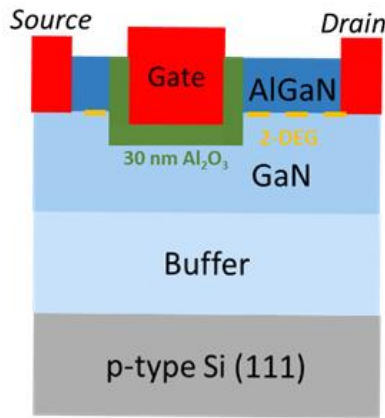
Two structures were considered: a bilayer Al<sub>2</sub>O<sub>3</sub> 10 nm – GaO<sub>x</sub> 2 nm on GaN and a Al<sub>2</sub>O<sub>3</sub> 10 nm single layer deposited on preliminary etched GaN. The first sample was used as a dedicated reference sample where an actual gallium oxide layer was grown whereas the second one was a technologically relevant structure. Several experimental conditions were compared for the ToF- and M-SIMS analyses; In particular, three angle of incidence for the primary Cs<sup>+</sup> sputter beam (65°, 61°, 45°) were used leading to

diverse depth resolution and roughness, as revealed by surface topography analysis provided by AFM. Among the different incidence angles, it was found that the best experimental conditions were those obtained by ToF-SIMS analysis with an incidence angle of  $45^\circ$ , generating the less roughness.

## I. INTRODUCTION

In recent years we have seen a remarkable growth of devices and technologies in power electronics. The main aims of scientific and technological research in the field are articulated around the study of new transistor architectures capable of working with high current flows required for the increasing electrified transport sector [1]. In the past, silicon-based devices such as insulated gate bipolar transistors (IGBTs) or metal oxide semiconductor field effect transistors (MOSFETs) have been and still are widely used transistor architectures. However, with technological progress, the physical and operational limitations regarding the properties of silicon in the power electronics field have been reached. For this reason, III-N materials are gaining a lot of interest due to their specific physical properties and more precisely, gallium nitride (GaN) is very promising by virtue of its high bandgap and its high breakdown field compared to silicon [2], [3]. GaN is thus particularly suitable to build new devices such as high electron mobility transistors (HEMTs) working at high voltages and high frequencies. The innovative aspect in GaN-based HEMT transistors lies in the AlGaN/GaN heterostructure. The contact between these two semiconductors generates a two dimensional electron channel (2-DEG) which increases the mobility of the charge carriers and consequently allows a greater current passage than silicon-based transistor architectures [4].

However, there are still some issues to address to enhance the final performances of GaN HEMTs. The presence of the 2-DEG leads to a continuous passage of current between the source and the drain, yielding to “normally-ON” devices. For safety reasons, especially in power applications, normally OFF structures are preferred [5]. One of the possible normally-OFF architecture solution is the fully recessed gate metal oxide semiconductor (MOS)-channel HEMT (MOSc-HEMT) shown in figure 1. However, this type of architecture leads to some GaN surface damage happening during the etching process of the AlGaN and GaN layers [6], [7]. To reduce the etch damage, before depositing the Al<sub>2</sub>O<sub>3</sub> dielectric via atomic layer deposition (ALD), different wet chemical treatments are employed to reduce the surface contamination such as carbon and chemical residues [8].



**Figure 1.** Scheme of the normally-OFF GaN MOSc-HEMT structure

In addition, the wet chemical cleaning step is crucial to remove the native gallium oxide, which impact negatively on the final performances of the device by shifting the transistor threshold voltage ( $V_{th}$ ) and therefore obtaining a normally-ON device, as reported by Vauche et. al [9]. Furthermore, the dielectric deposition step is also crucial and has to be optimized to improve the quality of the Al<sub>2</sub>O<sub>3</sub>/GaN interface [10]. Post-

deposition treatments, such as high temperature annealing, are also considered in order to improve the final electrical performances of MOSc-HEMTs [11].

Advanced physico-chemical characterization techniques are needed to better control the quality of the material and interfaces of such devices. Magnetic secondary ion mass spectrometry (M-SIMS) and time-of-flight secondary ion mass spectrometry (ToF-SIMS) have already been used for a precise chemical analysis of these structures [12]. In this paper, a complementary approach based on M-SIMS, ToF-SIMS and atomic force microscopy (AFM) techniques is implemented in order to produce reliable information about these GaN-based HEMTs structures. The experimental conditions related to the two types of SIMS instruments being different, especially regarding the primary sputtering beam incident angle, the M-SIMS/ToF-SIMS analytical performances in the chemical description of the  $\text{Al}_2\text{O}_3/\text{GaN}$  interface are compared. AFM is used to gain information about the roughness and the morphology of the samples surfaces caused by ion sputtering.

## II. EXPERIMENTAL

For the first sample (called A), representative of a real device considering the fabrication steps, GaN was grown by metal organic chemical vapor deposition (MOCVD) technique on top of several AlGaN/GaN buffer layers deposited on silicon. To get close to the real final device, a MOSc-HEMT structure, an etching process was conducted using an inductively coupled plasma reactive ion etching (ICP-RIE) with a chlorine chemistry directly on the GaN layer. Further, after the etching process, the GaN surface was treated with hydrofluoric acid (HF). This cleaning was reported by Caroline et al. [13] as an efficient wet chemical treatment for gallium oxide removal. Finally, a 10 nm-

thick Al<sub>2</sub>O<sub>3</sub> layer was deposited at 300 °C using trimethylaluminium (TMA) and H<sub>2</sub>O as precursors on the GaN etched layer. The thickness of the alumina was determined by ellipsometry with an error bar of ± 1 nm. A second sample (called B) was fabricated on purpose to constitute a reference material with the actual presence of a gallium oxide layer on the GaN surface. This gallium oxide layer was intentionally grown using Plasma-Enhanced Atomic Layer deposition (PE-ALD) at 300 °C with an O<sub>2</sub> plasma on the GaN surface. The thickness of the GaO<sub>x</sub> was measured by transmission electron microscopy (TEM) with a final value of 2.2 ± 0.3 nm. As for the previous sample, a 10 nm thick Al<sub>2</sub>O<sub>3</sub> layer was then deposited on top of the GaO<sub>x</sub> layer by ALD with TMA/H<sub>2</sub>O precursors. All the details regarding the studied samples are summarized in table 1.

**Table 1. Samples description**

<b>Sample name</b>	<b>A</b>	<b>B</b>
GaN oxidation	No	Yes
Al <sub>2</sub> O <sub>3</sub> thickness	10 nm	
Precursors	TMA/H <sub>2</sub> O	
Wet treatment	HF	

ToF-SIMS measurements were carried out using a ToF-SIMS 5 (IonTof GmbH) in positive mode with Cs<sup>+</sup> sputtering at 500 eV and a Bi<sub>3</sub><sup>+</sup> analyzing beam at 15 keV. Low energy caesium sputtering is known to be an adequate path to depth profiling of III-N structures [14]. The angle of incidence of both guns is 45° with respect to the surface

normal, the raster area was  $300 \mu\text{m}^2$  and the analyzed area  $80\mu\text{m}^2$ . The current values for both analyzing beams were checked at each measurement to always operate under the same experimental conditions.

M-SIMS experiments were carried out using a magnetic SC-Ultra SIMS instrument (CAMECA) in positive mode with a  $\text{Cs}^+$  sputter gun energized either at 500 eV or 3 keV. The raster area was  $200 \mu\text{m}^2$  and the analyzed area  $33 \mu\text{m}^2$ . In the SC-Ultra, the angle of the primary caesium column is  $60^\circ$  with respect to the surface normal. However, in order to collect secondary ions with positive polarity with a high efficiency, the sample is positively polarized leading to a more grazing incidence of the primary ion beam [15]. The angle of incidence of primary  $\text{Cs}^+$  ions beam were  $65^\circ$  and  $61^\circ$  respectively at 500 eV and 3 keV of beam energy. Finally, the two sample surfaces were metallized in order to avoid charge effects during the analysis.

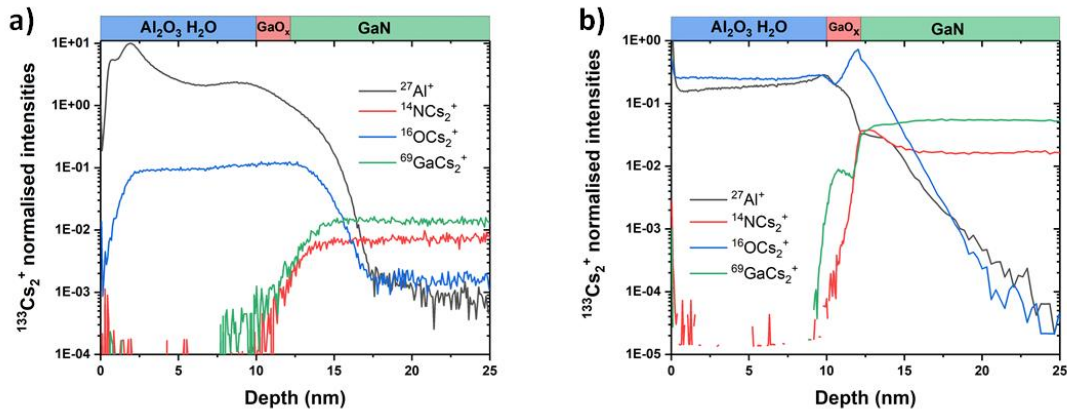
All SIMS analyzes were obtained in  $\text{MCs}_2^+$  mode in order to get depth profiles almost free from matrix effects that occur when heterostructures are analysed in conventional secondary ion negative polarity [16]–[18]. Indeed, as the composition changes, the ionization efficiency of an element can vary by several orders of magnitude and therefore mislead about the magnitude of the presence of an element in the heterostructure. For the depth profiling mode, the depth scale was calibrated with respect to the alumina thickness measured by ellipsometry with an uncertainty of  $\pm 1 \text{ nm}$ . The interface between the  $\text{Al}_2\text{O}_3$  and the GaN (sample A) or  $\text{GaO}_x$  (sample B) was set when reaching 50% of the average value of the  $^{69}\text{GaCs}_2^+$  intensity in the GaN matrix.

AFM analyzes were carried out on the FastScan System AFM instrument (Bruker Dimension Icon) in tapping mode with a silicon tip of a diameter of 10 nm. For each sample, 3 scans of 5  $\mu\text{m}^2$  size and 512 x 512 pixel resolution were performed, before and after SIMS measurements. Consequently, the root mean square (rms) roughness values were averaged over the 3 scans to get a accurate information about the surface morphology.

### III. RESULTS AND DISCUSSION

#### A. Comparison of ToF-SIMS and M-SIMS profiles

We compare here both ToF-SIMS and M-SIMS techniques with the aim of studying which are the best experimental conditions to be able to investigate the presence of gallium oxide at the  $\text{Al}_2\text{O}_3/\text{GaN}$  interface. In figure 2, we show the depth profiles of the  $\text{Al}_2\text{O}_3/\text{GaO}_x/\text{GaN}$  sample (B) obtained with both instruments with the same sputtering beam and energy ( $\text{Cs}^+$  at 500 eV).



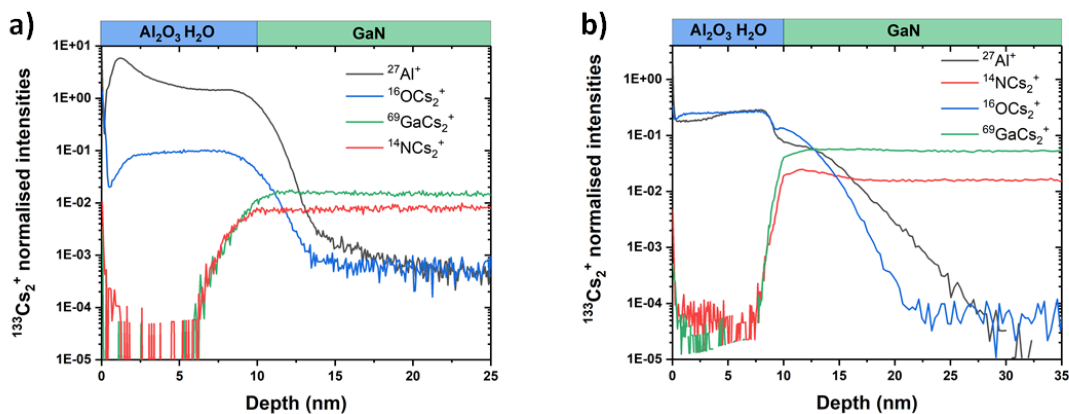
**Figure 2.** 500 eV  $\text{Cs}^+$  sputtering depth profiles of sample B obtained with a) M-SIMS and b) ToF-SIMS respectively at  $65^\circ$  and  $45^\circ$  incident angle.



From the depth profiles conducted at  $\text{Cs}^+$  500 eV on the reference sample (B), we note that the presence of gallium oxide is evidenced through ToF-SIMS analysis. Indeed, we can observe a first plateau between 10 and 12 nm in the  $^{69}\text{GaCs}_2^+$  signal in conjunction with a gradient of the  $^{16}\text{OCs}_2^+$  signal. The latter indicates a non-stoichiometric presence of oxygen along the thickness of the  $\text{GaO}_x$  layer. We can also notice a plateau regarding the  $^{16}\text{OCs}_2^+$  signal in the  $\text{Al}_2\text{O}_3$  matrix for which the stoichiometry has been verified by hard x-ray-photoelectron-spectroscopy (HAXPES) analysis with a final value of  $\text{O}/\text{Al} = 1.4$ .

The analyses carried out with the M-SIMS did not lead to the same observations, there is no clear highlighting of the gallium oxide at the interface through the  $^{69}\text{GaCs}_2^+$  and  $^{16}\text{OCs}_2^+$  signals. In addition, except for  $\text{Al}^+$ , higher ionization efficiencies are observed with ToF-SIMS compared to magnetic M-SIMS. This phenomenon could be explained by the differences in the analyzed area/raster area ratios between the two experimental setups.

Considering the real industrial case of sample A, the depth profiles given in figure 3 do not show a clear presence of gallium oxide for both instruments.

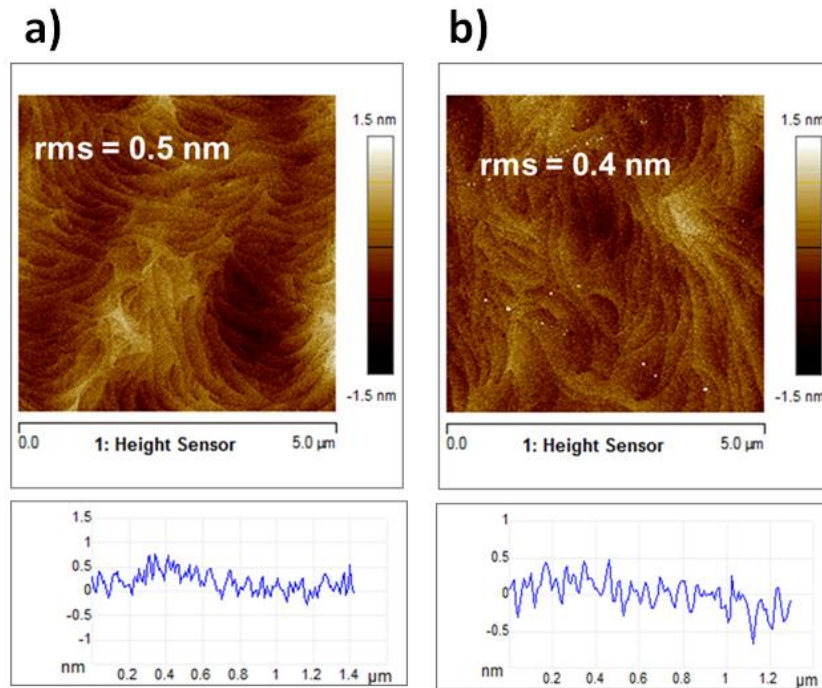


**Figure 3.** 500 eV Cs<sup>+</sup> sputtering depth profiles of sample A obtained with a) M-SIMS and b) ToF-SIMS respectively at 65° and 45° incident angle.

However, a plateau regarding the <sup>16</sup>OCs<sub>2</sub><sup>+</sup> signal is observed at the Al<sub>2</sub>O<sub>3</sub>/GaN interface with ToF-SIMS (see Figure 3b). Contrary to what was previously observed, this plateau is not correlated with a stable presence of gallium and it is therefore difficult to conjecture a real presence of gallium oxide. As for sample B, we observe differences regarding the ionization yields between the two analyzes in favor of the experimental conditions of the ToF-SIMS. Afterwards, we studied the impact of the incidence angle and roughness effect during SIMS analysis to better understand the previous depth profiles of our Al<sub>2</sub>O<sub>3</sub>/GaN structures.

### ***B. Effect of the incidence angle on surface roughness***

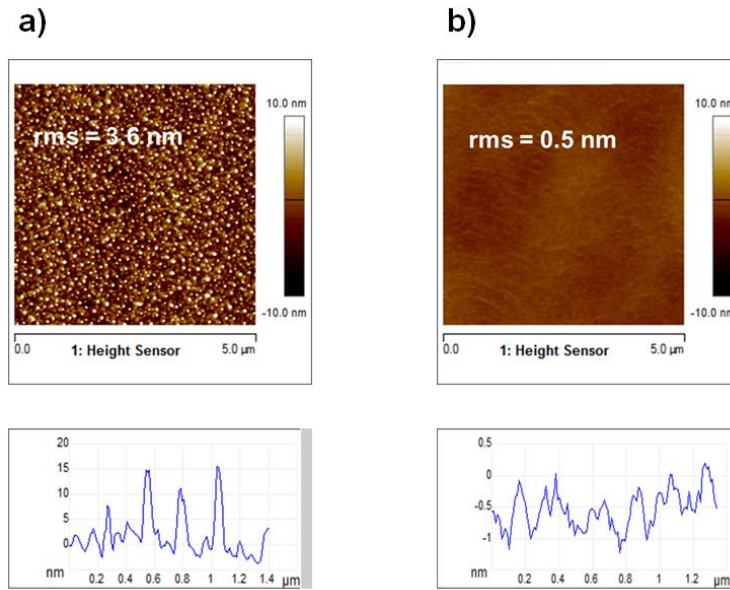
We firstly conducted AFM measurements before sims sputtering on samples A and B (see Figure 4).



**Figure 4.** a) AFM surface scans and height line scans on a)  $\text{Al}_2\text{O}_3$   $\text{H}_2\text{O}$  10 nm –  $\text{GaO}_x$  2 nm – GaN and on b)  $\text{Al}_2\text{O}_3$   $\text{H}_2\text{O}$  10 nm – GaN.

From the AFM analyses, we observe that the GaN atomic steps are clearly visible for both samples and that the rms roughness values are comparable. The dispersions of the peak-to-valley values on a 1.2  $\mu\text{m}$  length are also comparable. This therefore indicates that the initial morphology of the two samples is similar.

The results of the AFM measurements performed on sample B after sputtering are given in figure 5.

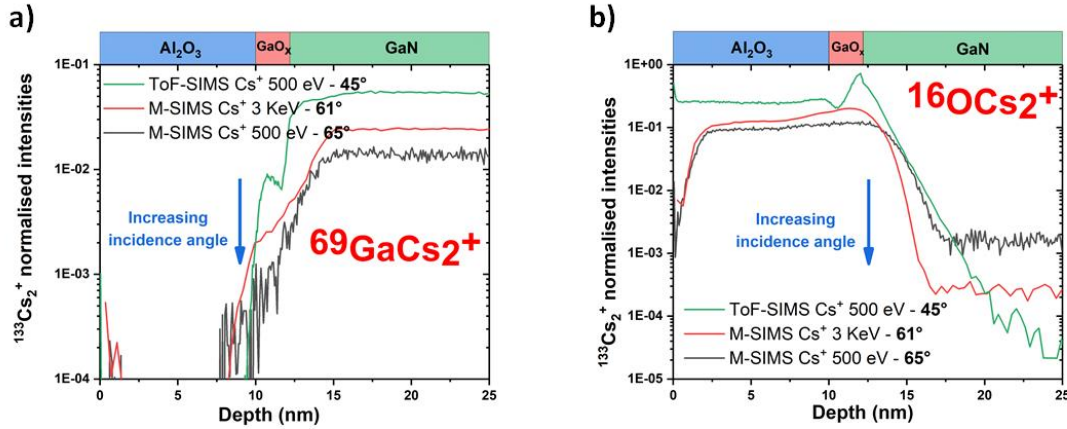


**Figure 5** a) AFM surface scans and height line scans on  $\text{Al}_2\text{O}_3$   $\text{H}_2\text{O}$  10 nm –  $\text{GaO}_x$  2 nm – GaN sample after 500 eV  $\text{Cs}^+$  sputtering at a)  $65^\circ$  and b)  $45^\circ$  incident angle when analysed respectively with M-SIMS and ToF-SIMS instruments.

AFM analyzes conducted after sputtering show two completely different morphologies. From one hand, the M-SIMS analyzes give rise to a surface with a high density of white points and an rms roughness equal to 3.6. The dispersion in terms of the height of the white dots varies between 5 and 15 nm. On the other hand, the ToF-SIMS analyzes show a surface free of white dots and consequently, an absence of peaks contrary to M-SIMS analysis

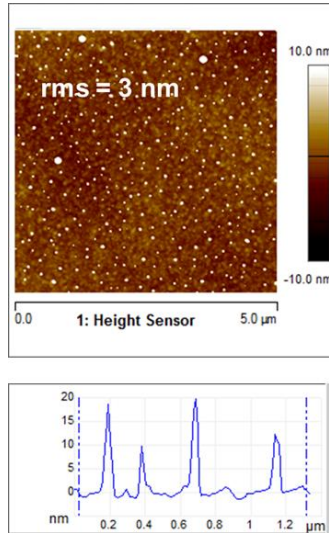
To confirm the strong contribution of the incident angle on the surface roughness and analytical depth resolution, we performed with the SC-Ultra M-SIMS instrument an additional depth profile at higher  $\text{Cs}^+$  beam energy, i.e. 3 keV. At this energy, the incident angle is slightly reduced, at  $61^\circ$ , but still significantly greater than the incident angle available in the ToF-SIMS 5 instrument.

In figure 6 are shown the depth profiles for  $\text{GaCs}_2^+$  and  $\text{OCs}_2^+$  secondary ions measured on sample B by ToF-SIMS (500 eV-45°) and M-SIMS (500 eV-65° and 3keV-61°).



**Figure 6.** a)  $\text{GaCs}_2^+$  and b)  $\text{OCs}_2^+$   $\text{Cs}^+$  sputtering depth profiles obtained with ToF SIMS at 500 eV – 45° incident angle and with M-SIMS at 500eV and 3 keV, 65° and 61° incident angle respectively, for sample B.

From the different profiles shown in figure 6, we observe that the incident angle has an effect on the ability to highlight the oxidized interface. Indeed, we note that the higher the angle of incidence, the lower the possibility to clearly evidence the thin  $\text{GaO}_x$  layer at the  $\text{Al}_2\text{O}_3/\text{GaN}$  interface. Moreover, it appears that the incident primary ions energy has only a weak contribution on depth resolution, increasing the energy leading to only a slightly degraded depth resolution.



**Figure 7.** AFM surface scan and height line scan on  $\text{Al}_2\text{O}_3$   $\text{H}_2\text{O}$  10 nm –  $\text{GaO}_x$  2 nm – GaN sample after 3 keV at  $61^\circ$  sputtering.

Post-sputtering AFM analyzes conducted on sample B after 3keV- $61^\circ$  sputtering show a different morphology and density from those previously illustrated at  $45^\circ$  and  $65^\circ$  (see Figure 7). Although the rms roughness value is quite similar to that of  $65^\circ$ , the scan exhibits a lower density of white points but from the height line we denote that the peaks are higher (over 20 nm). The angle of incidence seems to influence the density of white points thus impacting the ability to identify thin layers.

The difficulty in analyzing a 2.2 nm thin layer at  $65^\circ$  and  $61^\circ$  can be explained by the roughness created following the sputter. The rms roughness values are well above the value of the gallium oxide thickness thus preventing its observation. At  $45^\circ$  we observe how the surface was less impacted than in the two previous analyzes as the rms value is lower (0.5 nm) than the thickness of the gallium oxide and consequently, allowing its detection. The observations made regarding the presence

of white dots at 65° and 61° leads us to suppose that they could be the result of agglomerations of particles due to sputtering. Additional analyzes by Auger spectroscopy would allow to have sufficient lateral resolution to be able to analyze the chemical composition of the dots. From the preliminary analyzes conducted on a PHI700 (ULVAC-PHI), it is assumed that the observed peaks are the result of an agglomeration of particles mostly composed of carbon and caesium.

## **IV. SUMMARY AND CONCLUSIONS**

The electrical properties of GaN HEMT transistors strongly depend on the quality of the Al<sub>2</sub>O<sub>3</sub>/GaN interface. In this scientific work, we investigated the presence of gallium oxide at this interface through a combined approach between SIMS and AFM techniques. To be more precise, we compare M-SIMS and ToF-SIMS measurements at different angles of incidence and impact energies while analyzing by AFM the roughness caused by the primary ions beam. The intent is to improve the depth resolution to highlight a thin layer of gallium oxide at the Al<sub>2</sub>O<sub>3</sub>/GaN interface. Among the different angles of incidence, the ToF-SIMS at 45° angle highlighted the interface, a 2 nm-thick GaO<sub>x</sub> layer, which was not possible to see with M-SIMS at other angles of incidence. This observation is also supported by the AFM analyzes conducted after sputtering, showing RMS roughness values lower than the gallium oxide thickness for ToF-SIMS and consequently, the possibility of being able to highlight this interface.

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## DATA AVAILABILITY

Your manuscript must include a data availability statement. The data statement of your choice should be placed after the Acknowledgments and before the References and titled “Data Availability”. When data is available elsewhere, provide a citation of the data in your reference list. For instructions and templates on how to create your statement and reference, please visit [https://publishing.aip.org/wp-content/uploads/2020/02/AIPP-Data\\_Availability\\_Statement-Jan2020.pdf](https://publishing.aip.org/wp-content/uploads/2020/02/AIPP-Data_Availability_Statement-Jan2020.pdf).

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