



# Investigating Highly Doped Marker Layers in GaN on Sapphire Using Scanning Microwave Microscopy

## Application Note

Matthias A. Fenner,  
Agilent Technologies

Rachel A. Oliver,  
Department of Materials Science and Metallurgy, University of Cambridge

### Abstract

Gallium nitride films grown on sapphire substrate were investigated using Scanning Microwave Microscopy (SMM). During the growth thin, highly doped layers were included to mark the shape of the surface at regular intervals. The SMM's capability to measure dopant densities was employed to reconstruct cross sections of these surfaces. An unintentionally doped region was found for the initial stages of the growth. The growth surface at this stage is rough with most parts of the surface tilted out of the substrate plane. This suggests a model in which inclined surfaces promote the unintentional uptake of dopant material. Later stages of the growth result in smooth surfaces without unintentional doping.

### Introduction

GaN is a III – V semiconductor with a wide band gap. It is used in optoelectronics especially for the production of blue and green light emitting diodes (LED), high power, high temperature, and high frequency devices. It can also be doped with magnetic impurities which has possible applications in spintronics. A main challenge for the production of GaN based devices is the lack of suitable substrate materials. It remains very difficult to grow large GaN single crystals so that devices are mainly fabricated on sapphire and SiC substrate wafers. The heteroepitaxial growth

of GaN layers on sapphire substrate can be compromised by unintentional doping during the growth process. Identification of the origin and the mechanism of incorporation of dopants is needed to optimize the GaN based heterostructures for electronic devices.

Scanning Microwave Microscopy can be used to measure the density of charge carriers in semiconductors at a high spatial resolution. SMM combines the very high capacitance sensitivity of a Vector Network Analyzer (as the Agilent PNA) with the high spatial resolution of a beam deflection Atomic Force Microscope (AFM)<sup>1</sup>. Working on semiconductors the capacitance of the tip sample junction is influenced by the applied tip sample bias. This is well known behavior in semiconductors and especially in Metal-Insulator-Semiconductor junctions (MIS). Many semiconductors like Silicon or GaAs form an insulating oxide layer when exposed to oxygen or air. This so called native oxide generally is very thin on the order of Angstroms but thickness can be increased by thermal treatment with several hundred degrees<sup>2</sup>. A metallic SMM tip scanning a semiconductor surface in ambient conditions forms a MIS junction. When applying a bias voltage  $V_t$  to the SMM tip charge carriers in the semiconductor are attracted or depleted at the surface. A space charge region is formed. For a given semiconductor the thickness of the space charge region varies with



Agilent Technologies

$V_t$  which affects the capacitance of the MIS junction<sup>ii</sup>. The width of the space charge region is also a function of the charge carrier density in the semiconductor which in many cases is equal to the concentration of impurity donor or acceptor atoms i.e. the dopant density.

### Investigation of Gallium Nitride Growth

For the investigation of unintentional doping of GaN an overgrowth technique was employed<sup>iii</sup>. Nominally undoped material was grown. At regular intervals dopant material was introduced into the growing GaN for short periods thus forming thin layers of highly doped GaN. The sample was then cleaved to expose a cross section of the grown film and the marker layers. Figure 1 shows SMM topography, capacitance map, the dopant density map of the film cross section, and a line profile across the dopant density map.

The sapphire substrate is located on the left edge of the data set, the wafer surface would be further towards the right but it is not within the scan range shown. The topography shows a step from the substrate to the GaN layers, several steps within the GaN, and some undefined contaminations at the right edge. The capacitance map shows some contrast at the substrate/film interface and a regular pattern of bright and dark lines towards the wafer surface. In SMM dopant density maps undoped as well as highly doped material yield lower signal or darker regions. The dark regions comprise the sapphire substrate, the highly doped marker layers, and the undoped GaN layers. The regions are indicated in figure 1d. The bright features are regions with a low but not too low density of charge carriers. This is the case in the regular stripes towards the wafer surface. Between the layers dubbed "undoped" a layer of highly doped material was grown. Both materials have little dC/dV signal and show dark. Due to diffusion of carriers from the doped into the undoped layers a low density of carriers is present at the edge of the undoped region and thus shows a high dC/dV signal. The straight

regular stripes indicate that the film growth of these layers was regular and smooth.

In between the substrate and the smooth layers we find another region of doped material. This region was unintentionally doped during the growth process. In this region we find one to

three dark bands meandering from left to right. The bands are highly doped marker layers. They mark the position of the growth surface at those times when dopant material was introduced. In the unintentionally doped region the doped layers show a strong fluctuation indicating a rough surface during growth<sup>iii</sup>.

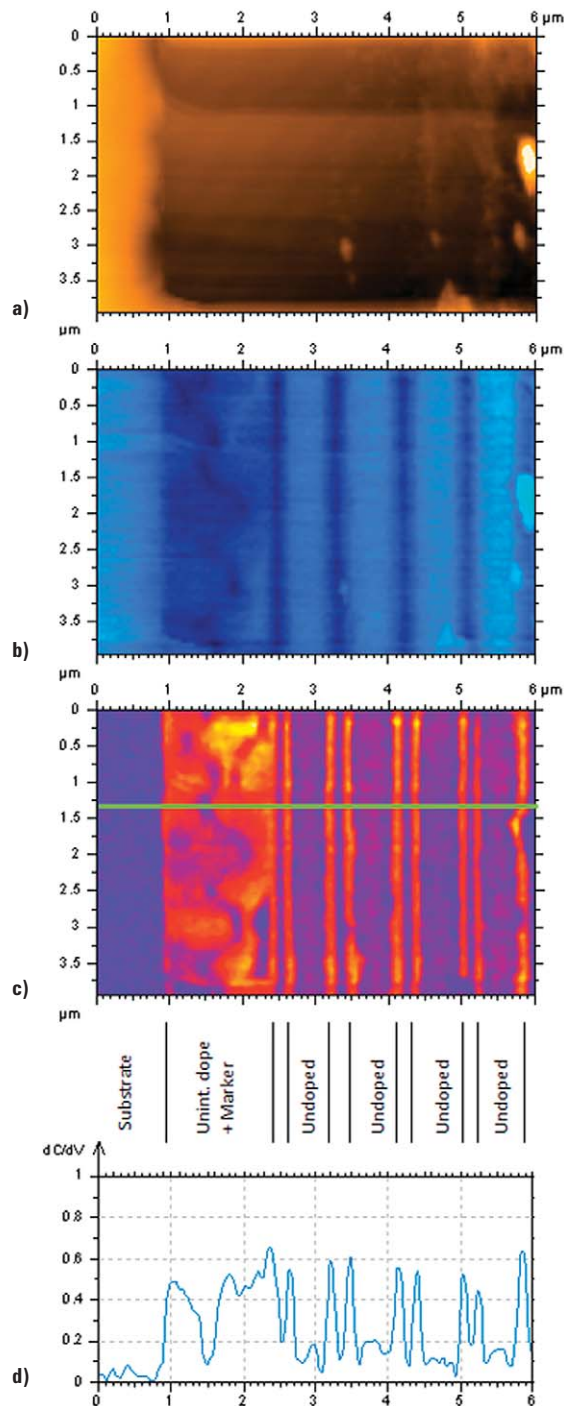


Figure 1. a) to d) SMM topography, capacitance map, dopant density map and cross section of dopant density map along the green line in c).

The model assumption that inclined surfaces are crucial for the growth of unintentionally doped material can be tested with dopant density maps of larger regions. Figure 2 shows topography and dopant density map of a 64  $\mu\text{m}$  wide scan. Additionally a schematic shows the positions of the substrate, three marker layers, the unintentionally doped region and its boundary as yellow, red, lines, black hashed areas, and blue lines respectively. Due to the cleavage steps and contaminations the marker layers and the doped region cannot be traced across the whole cross section so that some gaps remain in the lines.

The assumption that inclined surfaces are crucial for the growth of unintentionally doped material is supported by the fact that this material is present mainly in the regions where the marker layers fluctuate. Locations marked "A" and "B" are of particular interest for the analysis. The letter "A" marks regions where inclined surfaces persisted longer than in the surrounding material. Here the unintentionally doped material extends further towards the wafer surface. The letter "B" marks regions where the unintentionally doped material extends further towards the wafer surface, too, but the straight marker layers indicate a smooth growth surface. Despite the straight marker it is still possible that during growth the surface was inclined in an out of plane direction. Therefore locations marked "B" do not preclude the inclined surface model. A detailed analysis of the model can be found in the article by R. A. Oliver<sup>iii</sup>.

### Conclusion

Scanning Microwave microscopy was employed to investigate the origin of unintentionally doped regions in Gallium Nitride grown on sapphire substrate. During the growth process thin marker layers were introduced which are snapshots of the surface configuration. Cross sections through these surfaces reveal that unintentionally doped regions grow at initial stages of the growth when the surface is rough and most parts of the surface are tilted out of the substrate plane.

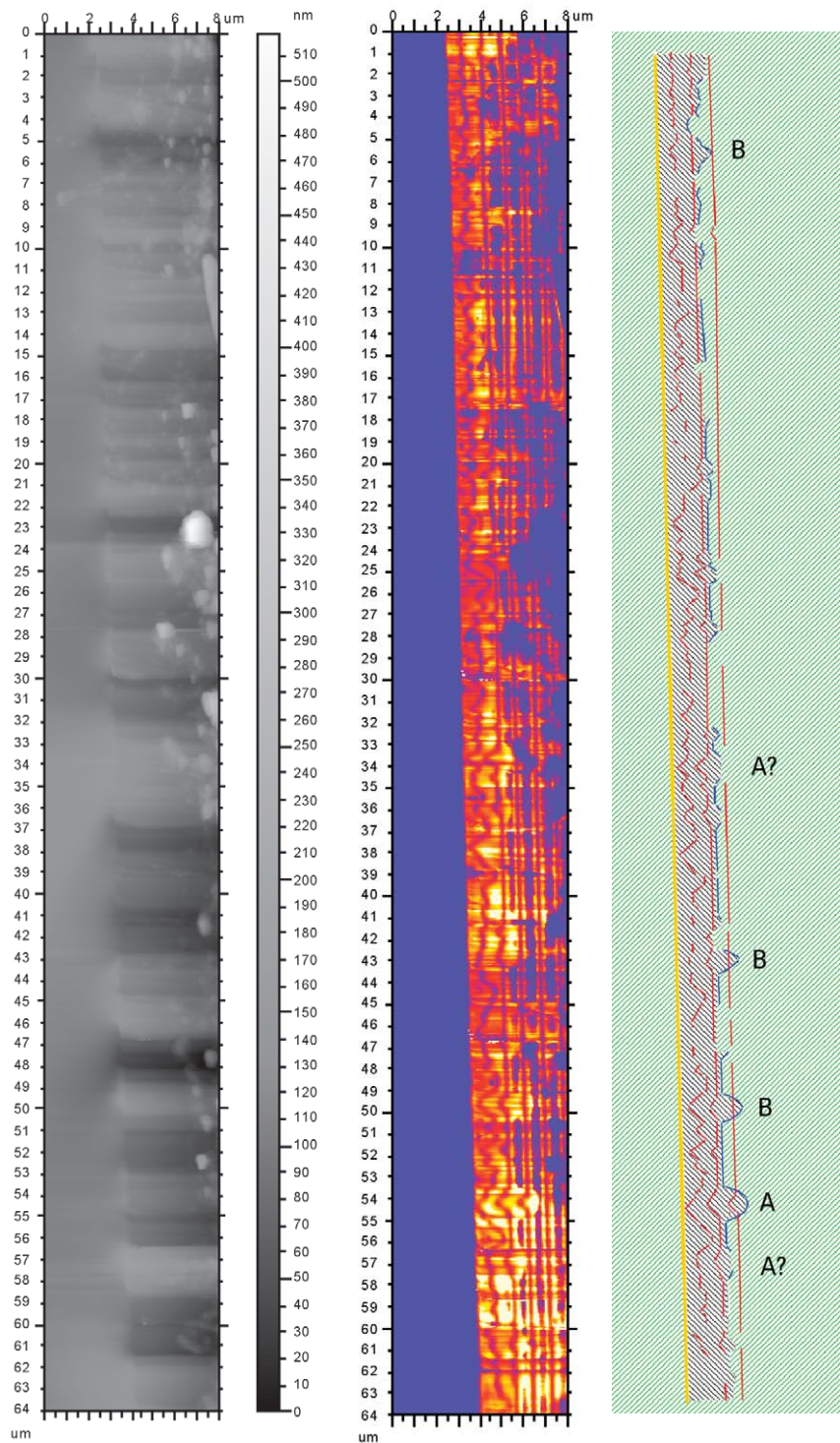


Figure 2. Topography, dopant density map, and schematic of GaN layers on sapphire. The positions of the substrate, marker layers, the unintentionally doped region and its boundary are shown as yellow, red lines, black hashed areas, and blue lines respectively. Due to cleavage steps and contaminations the marker layers and the doped region cannot be traced across the whole cross section.

## References

- i H.P. Huber, M. Moertelmaier, T.M. Wallis, C.J. Chiang, M. Hochleitner, A. Imtiaz, Y.J. Oh, K. Schilcher, M. Dieudonne, J. Smoliner, P. Hinterdorfer, S.J. Rosner, H. Tanbakuchi, P. Kabos, and F. Kienberger, Rev. Sci. Inst. 81, 1 (2010). Calibrated nanoscale capacitance measurements using a scanning microwave microscope.
- ii Physics of Semiconductor Devices, S.M. Sze, John Wiley & Sons, New York, 1981.
- iii R.A. Oliver, Ultramicroscopy 111, (2010), Application of highly silicon-doped marker layers in the investigation of unintentional doping in GaN on sapphire.

## AFM Instrumentation from Agilent Technologies

Agilent Technologies offers high-precision, modular AFM solutions for research, industry, and education. Exceptional worldwide support is provided by experienced application scientists and technical service personnel. Agilent's leading-edge R&D laboratories are dedicated to the timely introduction and optimization of innovative and easy-to-use AFM technologies.

[www.agilent.com/find/afm](http://www.agilent.com/find/afm)

### Americas

---

Canada	(877) 894 4414
Latin America	305 269 7500
United States	(800) 829 4444

### Asia Pacific

---

Australia	1 800 629 485
China	800 810 0189
Hong Kong	800 938 693
India	1 800 112 929
Japan	0120 (421) 345
Korea	080 769 0800
Malaysia	1 800 888 848
Singapore	1 800 375 8100
Taiwan	0800 047 866
Thailand	1 800 226 008

### Europe & Middle East

---

Austria	43 (0) 1 360 277 1571
Belgium	32 (0) 2 404 93 40
Denmark	45 70 13 15 15
Finland	358 (0) 10 855 2100
France	0825 010 700*
	*0.125 €/minute
Germany	49 (0) 7031 464 6333
Ireland	1890 924 204
Israel	972-3-9288-504/544
Italy	39 02 92 60 8484
Netherlands	31 (0) 20 547 2111
Spain	34 (91) 631 3300
Sweden	0200-88 22 55
Switzerland	0800 80 53 53
United Kingdom	44 (0) 118 9276201

Other European Countries:

[www.agilent.com/find/contactus](http://www.agilent.com/find/contactus)

Product specifications and descriptions in this document subject to change without notice.

© Agilent Technologies, Inc. 2012  
Printed in USA, January 3, 2012  
5990-9693EN



**Agilent Technologies**