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IN-SITU CHARACTERIZATION OF GaN MATERIAL USING REFLECTANCE SPECTROSCOPY

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Abstract

This paper presents a measurement system for in-situ characterization of semiconductor structures fabricated by the *Metalorganic Chemical Vapor Deposition* (MOVPE) method using *Reflectance Spectroscopy* (RS). The construction of the developed measurement set-up is presented, along with a description of individual functional blocks. As part of the experiment, the parameters of the deposited gallium nitride (GaN) layer such as thickness (d), roughness (R_{EMA}), optical energy bandgap (E_g^{opt}) were monitored in-situ, and the complex refractive index ($n + ik$) of GaN was determined at temperatures above 1000°C. The *Effective Medium Approximation* (EMA) method was employed to characterize the surface roughness of the layer during the growth process. Based on this data, the exact moment of full coalescence and subsequent growth in two dimensions was determined.

Keywords: AIIIN, MOVPE, in-situ measurements, reflectance spectroscopy, optical properties.

1. Introduction

Heterostructures based on third-group nitrides from the periodic table (AIIIN), specifically gallium nitride (GaN), gallium oxide (Ga_2O_3), and scandium nitride (ScN), are gaining prominence in the fabrication of advanced microelectronic devices. Achieving precise control over the crystallization process of the semiconductor material necessitates the use of advanced in-situ characterization methods [1–3].

The largest share of the global GaN semiconductor market belongs to high frequency wireless communication [4], power and high temperature [5,6] and optoelectronic devices [7–9]. Successful integration of GaN and Si transistors on 300 mm substrates [10] has become a milestone for development of modern hybrid integrated circuits.

The conventional method employed for in-situ characterization of epitaxial processes involves *Laser Reflectometry* (LR). This technique allows for analysis of the monochromatic light signal

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reflected from the deposited structure. The parameters of the layer are determined by analysing the duration of oscillations corresponding to the growth rate. Additionally, the signal amplitude is influenced by factors such as the surface roughness, refractive index of the layer and substrate, and the number of defects within the layer. To ascertain the material parameters of the fabricated layer, the recorded LR signal is analysed with a virtual interface model [11, 12].

The discussed measurement method, despite its simple measurement system design, presents notable drawbacks. A substantial portion of information related to the investigated structure within the reflection spectrum is lost. Consequently, determining critical parameters, such as the material's energy gap, chemical composition of the deposited layer, and its surface roughness, becomes challenging. Additionally, to ascertain the layer growth rate accurately, at least one complete oscillation of the analysed measurement signal is required.

The extension of the previously described measurement method involves analysing the reflection spectrum across a broad range of wavelengths. This approach facilitates the assessment of the above-mentioned material parameters, as well as the determination of the complex refractive index $(n + ik)$ of the layer at the process temperature. Thanks to the spectral analysis, measurements can be conducted at specific time points to extract relevant material properties. Notably, in the case of multi-component materials, which are not well-studied, in-situ analysis of the reflection spectrum allows for precise characterization of the deposited layers and their composition. Consequently, this method reduces the need for extensive technological experiments when developing next-generation semiconductor devices.

Metalorganic Chemical Vapor Epitaxy (MOVPE) stands as one of the most prevalent epitaxial techniques in semiconductor material growth. Its popularity arises from several key advantages: good scalability, precise control over layer thickness and composition, and the capability to produce layers spanning a range from approximately one nanometre to several micrometres. Consequently, MOVPE epitaxy finds widespread use in fabricating heterostructures based on AIIIN materials. The MOVPE reactors, typically constructed from stainless steel, introduce unique measurement challenges. They operate at high temperatures exceeding 1000°C and involve the use of toxic gases. As a result, in-situ characterization of semiconductor structures grown via MOVPE relies exclusively on optical methods.

2. Description of the measurement system

The work presents a measurement system for in-situ characterization of semiconductor structures fabricated within an AIXTRON CCS FT 3x2" MOVPE epitaxial reactor, featuring a *Close Coupled Showerhead* (CCS). A characteristic feature of this type of reactors is the use of a "showerhead" homogenizing head and a rotating graphite susceptor on which the substrates are placed. This is intended to enhance gas dosing uniformity across the substrate, thereby increasing the uniformity of the manufactured semiconductor structures. Optical characterization of the grown structure is carried out through a measurement window located in the cover of the epitaxial reactor.

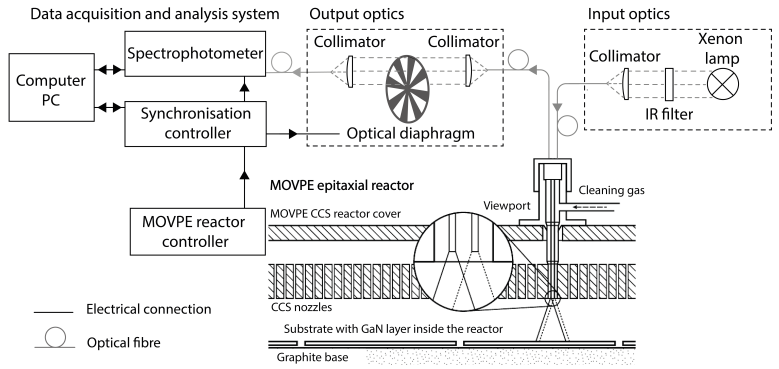
The most commonly employed measurement technique is laser reflectometry, which is comprised of four fundamental components. The initial component is a light source, which may take the form of a *laser diode* (LD) or *light-emitting diode* (LED), with an emission wavelength characteristic for the material group under study. The subsequent component is a beam splitter, which is responsible for dividing the optical power and redirecting the light reflected from the tested surface to the optical detector. The third component is a controller for synchronising the rotation of the graphite susceptor on which the semiconductor substrates are placed. Furthermore,

the device is responsible for processing the signal obtained from the photodiode. Measurements are made through a quartz window situated within the MOVPE epitaxial reactor.

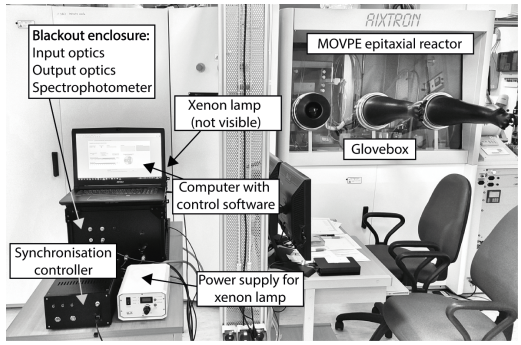
The straightforward configuration of laser reflectometry was attained through the examination of discrete wavelengths. In the case of the measurement system described in this article, it is possible to analyse a wide spectrum of radiation resulting in a more complex design, which can be divided into four key functional blocks:

1. Input optical system intended to illuminate the deposited structure,
2. Epitaxial reactor in which the layer deposition process is carried out,
3. Output optical system enabling sampling of reflected light,
4. Measurement data acquisition and analysis module.

The block diagram of the measuring system is shown in Fig. 1a), while Fig. 1b) shows a photograph of the measuring station.



(a)



(b)

Fig. 1. Measurement system for in-situ reflectance spectroscopy of semiconductor structures fabricated in a MOVPE CCS reactor, a) block diagram, b) photo of measuring station.

The input optical system consists of a LOT LSN150 xenon illuminator with a power rating of 75 watts. The xenon lamp emits light, which is then transmitted through the ThorLabs TF1 optical filter. The filter has spectral characteristics within the 375–650 nm range. Subsequently, the light is focused onto a ThorLabs FG600AEA optical fibre using a ThorLabs LA4380 lens. Within the reactor, a transmitting and receiving optical fibre is connected via a gas-tight capillary. The optical fibres are positioned approximately 12 millimetres above the examined structure, and the

spot illuminating the structure has a diameter of approximately 6 millimetres. The reflected light from the measurement structure is transmitted through the receiving optical fibre to the optical receiving system, where the signal is sampled.

The optical shutter [13] is controlled via self-developed software using a system for synchronizing the rotation of the graphite base, which allows precise selection of the measurement area and acquisition time for optical signals. In our experiments, the optical shutter remained open for 12 milliseconds, corresponding to a rotation of the graphite base by approximately 4.5 degrees. The measurement frequency primarily depends on the rotational speed of the graphite base, typically set at 60 revolutions per minute. Additionally, the optical diaphragm not only samples the optical signal but also protects the optical detector from intense light, reducing thermal noise and thereby improving the *Signal-to-Noise Ratio* (SNR).

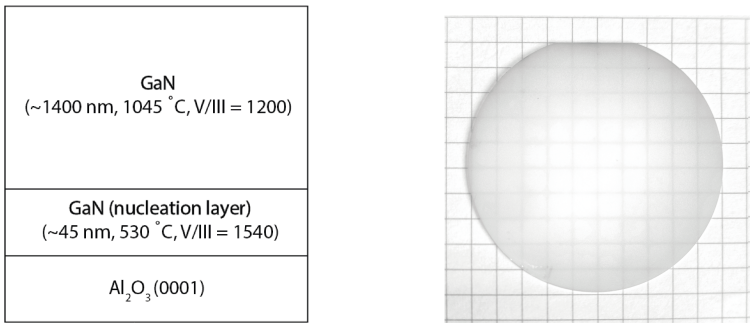
The spectral data were acquired using an Ocean Optics USB4000 spectrophotometer, while the measurement system was controlled using a PC computer and self-developed software.

3. Test structure

As part of the experimental study, in-situ characterization of gallium nitride (GaN), which is a fundamental material utilized in device structures based on AIIIN heterostructures, was conducted. A thin layer of gallium nitride was manufactured using the MOVPE technique in the AIXTRON FT CCS 3 × 2″ system. The layer growth was carried out at a reduced pressure of 100 mbar in a hydrogen atmosphere, using trimethylgallium (TMGa) and ammonia (NH_3) as precursors for gallium and nitrogen, respectively.

Prior to GaN deposition, a single-sided polished 2″ sapphire substrate (Al_2O_3) with the (0001) orientation was subjected to a thermal cleaning process at 1070°C for 5 minutes in hydrogen. Subsequently, the substrate surface was nitrided using ammonia at 530°C for 5 minutes. The nitridation step immediately preceded the deposition of a low-temperature LT-GaN seed layer at 530°C, with a NH_3/TMGa molar ratio of 1540:1. Based on previous calibration procedures, the estimated thickness of the GaN seed layer was approximately 45 nanometres.

In the subsequent step, the nucleation layer was annealed by increasing the temperature from 530°C to 1045°C i.e., to the conditions for deposition of the actual GaN layer (1045°C, NH_3/TMGa molar ratio of 1200:1). The layer diagram in the manufactured structure is shown in Fig. 2a), while Fig. 2b) shows a photograph of the produced structure.



(a) (b)

Fig. 2. Layer composition of the test structure a) schematic diagram, b) photograph of the analysed structure characterised in-situ with the developed measuring system for the reflection spectroscopy method.

4. Measurement procedure

In order to measure the reflection spectrum, a 100 nm thick aluminium (Al) mirror was fabricated on a *Si*(111) silicon substrate using the UHV PVD225 metal layer deposition system manufactured by the K. J. Lesker Company. The thickness of the substrate on which the metallic layer was created and the diameter of the substrate (2") are compatible with the MOVPE epitaxial reactor of the CCS type and do not introduce additional measurement errors resulting from the modification of measurement conditions.

Before the deposition of GaN/Al_2O_3 test structure, a reference spectrum was measured using an aluminium mirror. Simultaneously, the epitaxy process was monitored using an independent, commercial measurement system that performed laser reflectometry measurements at a wavelength of 635 nanometres. On this basis, the layer thickness was determined to be approximately 1400 nm. Figure 3 illustrates the process, including the temperature profile, reagent dosing, and reflectogram at the 635 nm wavelength. In Fig. 3, Stages 1 to 4 correspond to individual measurement points:

1. Measurement of the reflection spectrum from the reference mirror (I_{REF}).
2. Measurement of the radiation spectrum of the graphite base and Al_2O_3 substrate at the process temperature (I_{TEMP}).
3. Reflectance spectrum at the moment of full coalescence of the layer, determined for the time during which there is no change in the oscillation period and the stabilized process temperature ($I_{\mathrm{MEAS3,4}}$).
4. Measurement of the reflection spectrum at the end of layer growth (TMGa dosing), (I_{MEAS5}).

Figure 3 omits the reflection spectrum measurement at 30°C.

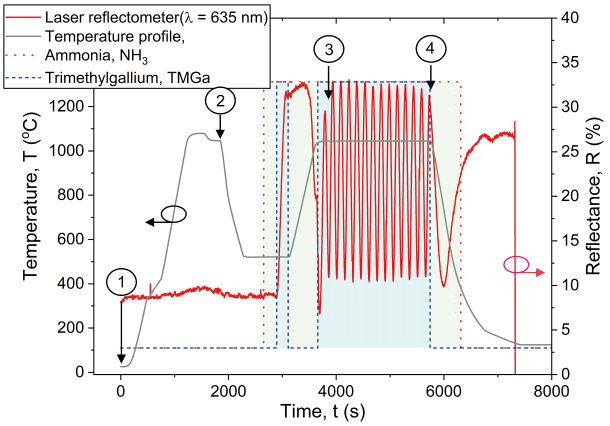


Fig. 3. Course of the MOVPE process during the growth of the GaN/Al_2O_3 test structure.

5. Experimental results and discussion

Reflectance spectrum measurements were made in accordance with the description of the measurement procedure presented in the previous section. The measurements carried out at individual stages of the epitaxial structure deposition process made it possible to determine the reflection spectrum using (1).

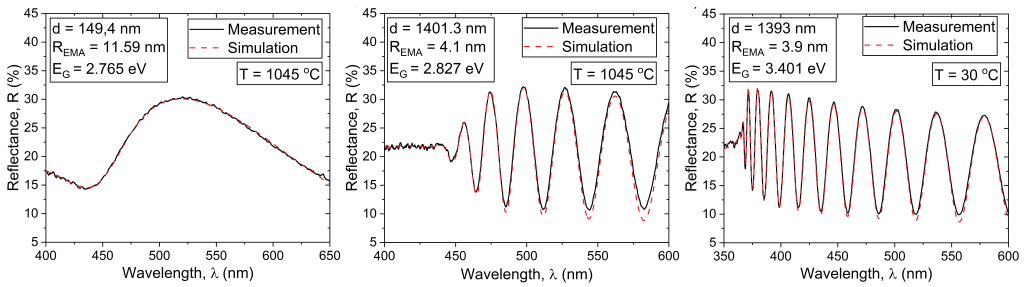
$$R = \frac{I_{\mathrm{MEAS}} - I_{\mathrm{TEMP}}}{I_{\mathrm{REF}}} \cdot 100\%, \tag{1}$$

where: R – Reflectance (%); I_{REF} – measurement of the reference spectrum, measured at Point 1; I_{TEMP} – measurement taken at the process temperature before the layer growth, Point 2; I_{MEAS} – measurements made during the process, Points 3, 4 and 5.

The reflection spectrum analysis of the GaN/Al2O3 test structure was conducted using the V.A.S.E. software developed by J. A. Woollam, along with the parametric model of semiconductor oscillators (PSEMI, Psemi-0 and Psemi-Tri and one Gaussian oscillator), which was developed by Johs and Herzinger [14, 15]. The PSEMI model uses the summation of oscillators described by polynomials of the spline function at each critical point, ensuring compliance with the Kramers-Kronig relation. The quality of fit of the numerical model to the experimental data was assessed on the basis of *Mean Squared Error* (*MSE*). Specifically, the reflection spectra were analysed at Points 3 and 4, as indicated in Fig. 3. Additionally, a measurement was performed after cooling the epitaxial reactor to a temperature of 30°C, although this condition was not marked in Fig. 3. By simulating the reflection spectrum (Fig. 4), it was possible to determine material parameters layer thickness (d), roughness (R_{EMA}), optical energy bandgap (E_g^{opt}), and the complex refractive index ($n+ ik$). These parameters are summarized in Table 1 and visualized in Fig. 5.

Table 1. Summary of the results obtained at various moments of the GaN/Al_2O_3 test structure growth process.

Measurement point	d (nm)	R_{EMA} (nm)	E_g^{opt} (eV)	T (°C)	*MSE*
Point 3	149.4	11.6	2.764	1045	0.017
Point 4	1401	4.1	2.827	1045	0.105
After the deposition process	1393	3.9	3.401	30	0.065



(a) (b) (c)

Fig. 4. Reflectance spectra measured a) at the moment of transition from 3D to 2D growth (Point 3), b) just after the end of the growth of the epitaxial layer (Point 4), c) inside the epitaxial reactor at a temperature of 30°C.

The analysis of the reflection spectrum made possible to determine the optical energy bandgap. An underestimation of the E_g value for GaN by approximately 10-40 meV was observed compared to the literature data [16].

This discrepancy is likely attributable to the influence of stress and defects in the LT-GaN layer, which exhibits different material and optical properties compared to the GaN layer grown at high temperature [17, 18]. The significant density of defects in the heterostructure can also influence the shape of the reflection spectrum, thereby affecting the precision of the simulation fit to experimental data. Another equally crucial factor affecting the determined energy gap value is the method of measurement itself. As demonstrated by J. F. Muth *et al.* in their paper [19], there is a divergence of approximately 20-50 meV between the energy gap and the optical energy gap of the analysed material.

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To validate the determined E_g, the photoluminescence spectrum was measured at room temperature (Fig. 5). This measurement was conducted using a CryLas laser with a wavelength of $\lambda = 266$ nm and a Horiba microHR spectrophotometer. The analysis enabled the determination of the energy gap width, which was found to be $E_g = 3.421$ eV. The determined E_g for GaN confirmed the discrepancy between the measurement data and the literature data.

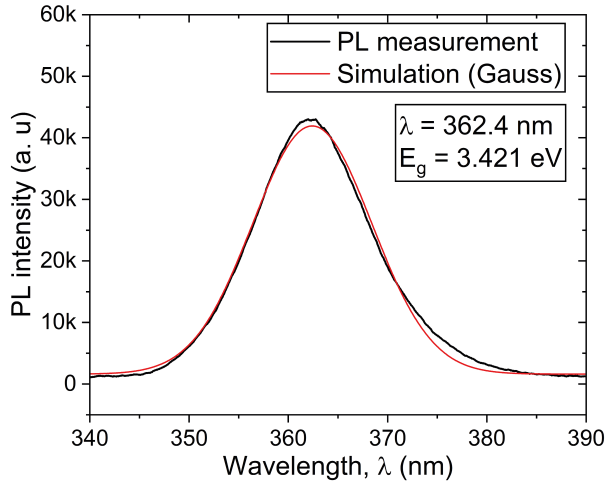

Fig. 5. Photoluminescence spectrum of the GaN layer at RT.

The reflection spectrum analysis conducted at various stages of the process enabled the determination of both refractive and extinction coefficients (Fig. 6) at the process temperature (1045°C) and post-process temperature (30°C). The blue line represents the measurement taken at Point 3, while the red line corresponds to the measurement at Point 4 during the MOVPE epitaxy process. The black line denotes the refractive index at room temperature.

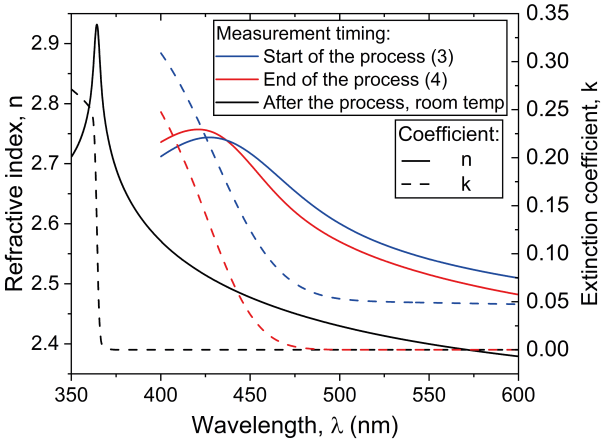

Fig. 6. Determined refractive indices for the real part (n) and imaginary part (k).

Differences between the values of the $(n + ik)$ for measurement at Points 3 and 4 result from the percentage of the nucleation layer characterized by a significant number of defects, poor crystallographic quality and strong stresses in relation to the GaN layer produced at high temperature. The E_g^{opt} and $(n+ik)$ analysis enables the assessment of the degree of recrystallization of the nucleation layer, and in turn, enables the optimization of the examined layer to achieve superior optical and electrical properties in the final semiconductor device.

The layer thickness, initially estimated from the reflection spectrum, was subsequently verified by cross-sectional images of the GaN/Al2O3 structure using a Hitachi SU6600 scanning electron microscope (SEM). To enhance measurement accuracy, 20 consecutive measurements were conducted, from which an average layer thickness of 1397 nm was determined. The analysis accounted for the thermal expansion coefficient of GaN, which is 3.17×10^{-6} 1/K. This coefficient corresponds to a thickness change of 4.6 nm for 1015°C increase in temperature (from 30°C to 1045°C). An image of the cross-section of the test structure is shown in Fig. 7.

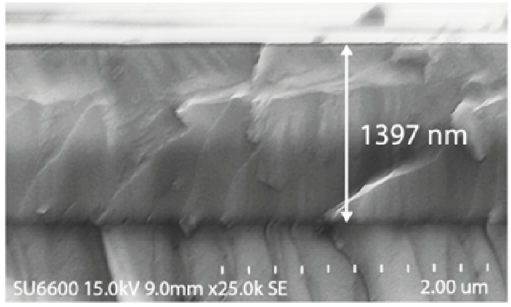

Fig. 7. Cross-sectional SEM image of the investigated GaN/Al_2O_3 structure.

The Effective Medium Approximation (EMA) method was employed to characterize the surface roughness of the layer [20]. In the case of measurements taken at Points 3 and 4, the roughness values were determined to be around 11.6 nm and 4.1 nm, respectively. By analysing selected measurements taken during growth, a characteristic was plotted to show the change in roughness as a function of layer thickness. Based on this data, the exact moment of full coalescence and subsequent growth in two dimensions was determined. Notably, as the thickness of the deposited GaN layer increased, the roughness decreased. This behaviour can be attributed to the initial island growth of the nucleation layer, which has an approximate thickness of 45 nm. The function that describes changes in surface roughness enables the determination of the minimum layer thickness at which surface development reaches the desired low value. The corresponding characteristics are illustrated in Fig. 8, while the function parameters are listed in Table 2.

Table 2. The parameters characterizing the function of roughness reduction in the thickness domain.

Parameter	y_0	A_1	t_1
Value	–2.05	15	–1614

The obtained measurement results were compared with those obtained using an atomic force microscope for areas of 5 μm × 5 μm and 50 μm × 50 μm, respectively, as presented in Fig. 9. Notably, significant differences in roughness were observed between the measurements. In the case of the 5 μm × 5 μm measurement, the GaN layer exhibited a typical terraced structure. However, the root mean square roughness value was 0.82 nm. Conversely, for the 50 μm × 50 μm surface,

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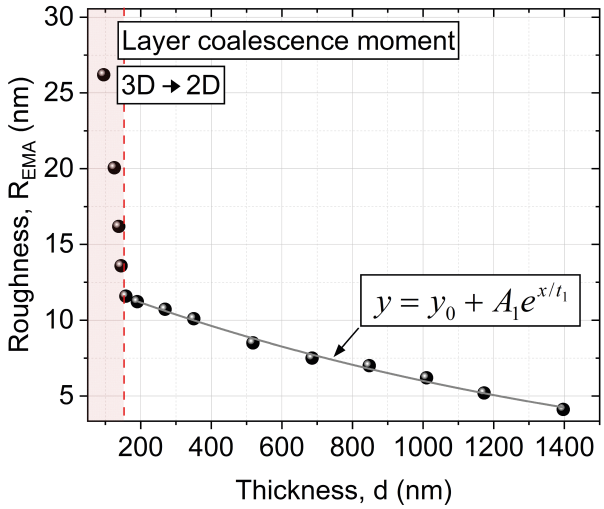
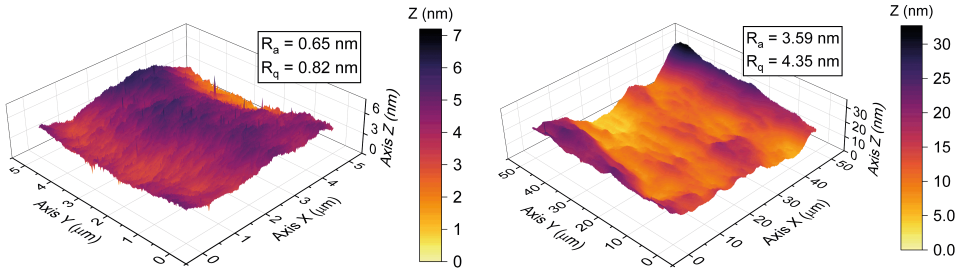


Fig. 8. Variation of roughness as a function of the thickness of the grown layer.

this parameter increased to 4.35 nm. These roughness disparities arise from the initial island growth during nucleation layer formation. Analysing layer roughness during growth, particularly in materials like GaN, is crucial for achieving smooth interfaces between layers. Such interfaces have a crucial role in optoelectronic applications and epitaxial production of two-dimensional materials, where surface development is a critical manufacturing consideration.



(a) (b)

Fig. 9. Atomic force microscope images showing the surface development of the GaN layer, for the measured areas a) 5 µm x 5 µm, b) 50 µm x 50 µm.

6. Conclusions

The presented research findings validate the feasibility of utilizing the developed in-situ measurement system for characterizing semiconductor layers produced via the MOVPE technique in CCS reactors. The measurement system enables the analysis of many layer parameters such as: thickness (d), roughness (R_{EMA}), optical energy bandgap (E_g^{opt}), and the coefficient assessment.

Notably, existing literature of the subject lacks comprehensive data on refractive index and extinction measurements across a wide spectral range at high temperatures. This limitation arises

due to layer decomposition and changes in surface roughness under high-temperature conditions. Therefore, *n+ ik* analysis should be carried out during or immediately after the end of the process.

Moreover, the introduced measurement system may be useful for future applications in the analysis of multi-component and multi-layer semiconductor structures. For instance, it can be employed for in-situ examination of manufacturing processes involving Bragg mirrors, gradient structures, and the observation of physical phenomena (such as the Purcell effect) in modern materials like GaN, Ga_2O_3, or ScN. Importantly, this system offers a valuable alternative to conventional techniques for in-situ characterization of MOVPE epitaxy, providing more information than traditional methods.

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