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Optical reflectance spectroscopy for barrier thickness measurement of AlGaN/GaN heterostructures: comparison with X-ray reflectometry

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Abstract. This study evaluates optical reflectance (OR) spectroscopy as a rapid, cost-effective alternative to X-ray reflectometry (XRR) for measuring the thickness of the AlGaN barrier layer in AlGaN/GaN heterostructures. OR spectroscopy demonstrated excellent agreement with XRR, with deviations not exceeding 1 nm. The results highlight OR spectroscopy as an efficient and reliable method for routine characterization of GaN-based heterostructures.

Keywords: gallium nitride, AlGaN/GaN, heterostructure, optical reflectance spectroscopy, X-ray reflectance

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Измерение толщины барьерного слоя в гетероструктурах AlGaN/GaN методом спектроскопии оптического отражения: сравнение с рентгеновской рефлектометрией

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Аннотация. В данной работе исследуется возможность применения спектроскопии оптического отражения в качестве быстрой и недорогой альтернативы методу рентгеновской рефлектометрии (XRR) для измерения толщины барьерного слоя AlGaN в AlGaN/GaN гетероструктурах. Результаты, полученные двумя методами, хорошо согласуются друг с другом; отклонения не превышают 1 нм. Полученные данные демонстрируют, что спектроскопия оптического отражения может рассматриваться как практичный и надежный метод для регулярного контроля гетероструктур на основе GaN.

Ключевые слова: нитрид галлия, AlGaN/GaN, гетероструктура, спектроскопия оптического отражения, рентгеновская рефлектометрия

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Introduction

Gallium nitride (GaN) is becoming a cornerstone of modern high-power devices due to its wide bandgap (~ 3.4 eV), high electron saturation velocity (~ 2.5×10^7 cm/s) [1] and large breakdown electric fields (> 3 MV/cm) [2]. In Al(Ga)N/GaN heterostructures – key to high-performance transistors — a two-dimensional electron gas (2DEG) with a concentration up to 6×10^{13} cm^{-2} [3] and mobility up to 2500 $cm^2V^{-1}s^{-1}$ at room temperature [4] is formed at the heterointerface. Precise control of the barrier layer thickness is crucial, as this parameter affects the 2DEG density, mobility, and overall device performance.

X-ray reflectometry (XRR) is a standard, non-destructive technique capable of measuring layer thickness with sub-nanometer accuracy. However, it is relatively time-consuming, requires expensive complex equipment, and is often impractical for high-throughput measurements. Optical reflectance (OR) spectroscopy offers a simple, rapid, and more accessible alternative, particularly well-suited for routine measurement. Despite its advantages, the accuracy of OR for measuring barrier layers in AlGaN/GaN has yet to be evaluated.

In this work, we compare the performance of OR spectroscopy against XRR for determining the thickness of the barrier layer in AlGaN/GaN heterostructures. We demonstrate that OR spectroscopy provides reliable results with sub-nanometer agreement to XRR, while offering significant benefits in terms of cost, speed, and suitability for full-wafer analysis.

Materials and Methods

The heterostructures were grown on Si(111) substrates via metalorganic vapor phase epitaxy (MOVPE) in a custom Dragon-125 epitaxial system featuring an inductively heated horizontal reactor. Initially, AlN-on-Si templates were prepared in separate growth runs through the deposition of an AlN nucleation layer to prevent unintentional Ga incorporation. The AlN layer prevents Si wafer etching by gallium (referred to as the meltback etching effect [5]). The subsequent structure was grown in a second, main process as follows. First, a six-step compositionally graded AlGaN:Fe buffer layer was deposited on the templates. This design effectively addresses stress arising from the significant lattice mismatch and thermal expansion coefficient differences between GaN and Si, thereby reducing dislocation density and preventing crack formation during growth and cooling [6, 7]. Next, a ~1 μm-thick unintentionally doped GaN channel layer was grown, as we determined this thickness to be optimal for mitigating the detrimental effects of the Fe doping tail on the 2DEG properties, while maintaining good breakdown characteristics [8, 9]. Subsequently, a nominally binary ~1 nm thick AlN interlayer was deposited, followed by an AlGaN barrier layer with a different Al mole fraction and thickness. Finally, the structures were in-situ passivated with a Si_3N_4 layer (0.00 nm, 2.25 nm or 4.50 nm thick). A schematic cross-section of the grown structures is shown in Figure 1. Standard precursors – trimethylgallium (TMGa), trimethylaluminum (TMAl) and ammonia (NH_3), as well as ferrocene (Cp_2Fe) and monosilane (SiH_4), were used. A more detailed description of the growth conditions can be found in [6].

XRR measurements were performed with a PANalytical X'Pert PRO diffractometer (Cu Kα, λ = 1.5406 Å). The measured XRR spectra were analyzed and fit using X-ray Calc 3 software [10]. OR spectra were acquired at quasi-normal incidence using an Avantes AvaSpec 2048 fiber-optic

spectrometer with an AvaLight-DHc light source (deuterium mode only) and a 6-around-1 fiber optic reflection probe. The OR spectra were fit using the general transfer matrix method [11], with a custom Python-based implementation developed in-house. The complex refractive index for GaN were derived from the dielectric function in [12], while for AlGaN, data from [13] were used.

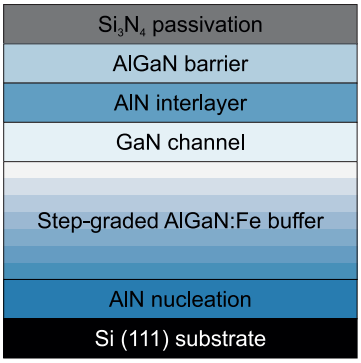

Fig. 1. Schematic cross-section of the epitaxial structures

Results and Discussion

The experimental XRR spectrum of one of the samples, along with the corresponding fitted curve, is shown in Fig. 2, *a*. Only the four topmost layers – namely, the Si_3N_4 passivation, AlGaN barrier, AlN interlayer, and GaN channel – were included in the analysis. The underlying buffer layers and substrate were found to have no impact on the results and were therefore omitted from the model. Consequently, the GaN channel layer was treated as an effective semi-infinite substrate. The obtained thicknesses of the Si_3N_4, AlGaN, and AlN are 4.49 nm, 16.93 nm and 0.71 nm, respectively. However, noted, the determined thickness of such ultra-thin AlN interlayer from XRR measurements may be ambiguous [14]; therefore, only the combined thickness of AlN and AlGaN barrier layers is meaningful.

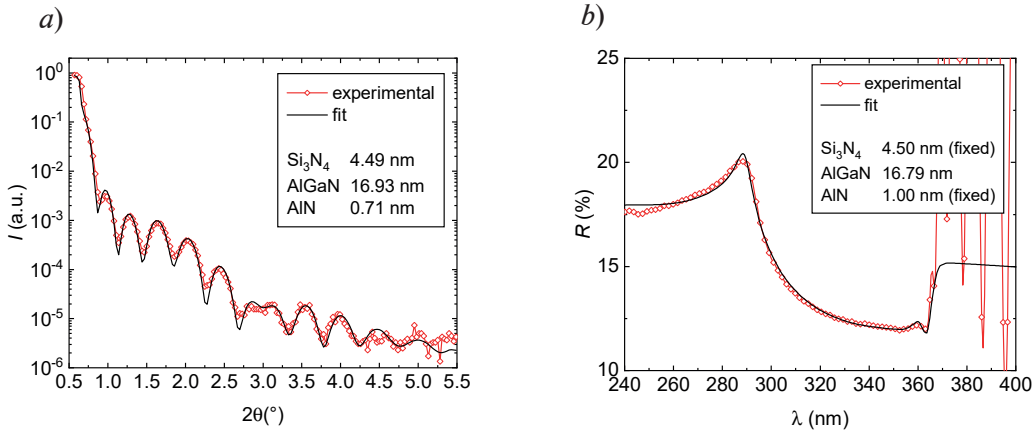

Fig. 2. Examples of the measured (red lines with symbols) XRR spectrum (*a*) and OR spectrum (*b*) along with the corresponding fitted curves (black lines)

Figure 2, *b* shows the measured optical reflectance spectrum of the previously discussed sample. Fabry-Perot oscillations are observed at wavelengths above around 365 nm, while absorption in the GaN layer suppresses these oscillations at shorter wavelengths. A distinct feature at around 290 nm corresponds to the bandgap of the AlGaN barrier layer. Due to the spectral range limitations of the equipment used (~240 nm), distinct spectral features attributable to the Si_3N_4 and AlN layers were not observed. Therefore, their nominal thickness values were fixed during the fitting procedures. Only the four topmost layers were included to the model, as in the XRR analysis. As shown in Fig. 2, *b*, the calculated OR spectrum (thin black line) accurately reproduces the shape and key features of the experimental spectrum. The fitted combined thickness of the barrier layers, 17.79 nm, is in excellent agreement with the XRR-derived value of 17.64 nm.

The fitting procedures described above were repeated for all investigated samples. A comparison of the combined thickness of the AlGaN and AlN barrier layers obtained using the XRR and OR methods is shown in Fig. 3. A good agreement is observed for all samples, with deviations not exceeding 1 nm, which is well within the estimated experimental uncertainty. These small differences may be attributed to spatial non-uniformities across the wafer, uncertainties in the optical constants, or the use of nominal thickness values for the AlN and Si_3N_4 layers in the OR modeling, which may slightly differ from their actual thicknesses.

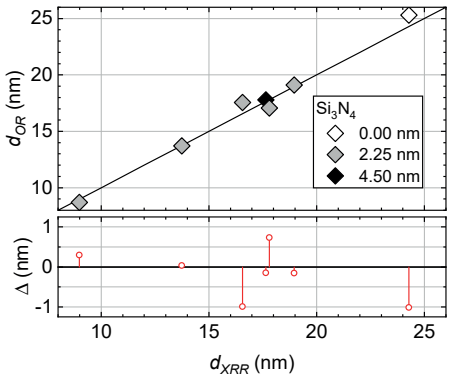

Fig. 3. Comparison of the combined thickness of the AlGaN and AlN barrier layers as determined by the XRR and OR method. The black line represents the ideal 1:1 agreement. Symbol fill denotes Si_3N_4 layer thickness: white for 0 nm, gray for 2.25 nm and black for 4.50 nm. The lower panel shows the difference between the values obtained by the two methods

Beyond the good agreement with XRR, the OR method is fast, non-destructive, and cost-effective. When combined with a CNC router or similar scanning setup, full-wafer mapping at dozens of locations can be completed within several minutes – a significant advantage over XRR, which is time-consuming and less suitable for large-area characterization. Additionally, the equipment required for OR measurements is relatively simple and inexpensive compared to the more complex and costly instrumentation needed for both XRR and spectroscopic ellipsometry, making OR highly accessible for routine use.

Nonetheless, the OR method has some inherent limitations. It requires prior knowledge of the optical constants of the materials involved, and the accuracy of the results strongly depends on the precision of these parameters. Moreover, OR is less sensitive to very thin layers. In contrast, XRR and ellipsometry techniques generally do not require such a priory information, often allowing the extraction of layer thicknesses and optical constants (in the case of ellipsometry) through model fitting. This restricts the flexibility of OR, especially when investigating novel or poorly characterized materials. However, even in the absence of precisely known optical constants, OR can still serve as a valuable tool for comparative “run-to-run” characterization. When a series of nominally identical structures is fabricated, or a previously grown structure is being reproduced, relative variations in OR spectra can be used to monitor process repeatability and detect early signs of drift in growth parameters. Thus, despite its limitations, OR remains a practical and efficient method for routine control in the production of semiconductor heterostructures.

Conclusion

In this work, optical reflectance (OR) spectroscopy was evaluated against X-ray reflectometry (XRR) for measuring the barrier thickness in AlGaN/GaN heterostructure. OR spectroscopy demonstrated excellent agreement with XRR, with deviations not exceeding 1 nm. These results highlight OR as reliable and cost-effective technique for routine, non-destructive analysis of GaN-based heterostructures, particularly in context requiring fast and/or large-area measurements.

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