



Metalorganic vapor phase epitaxy of GaN-based structures grown on etched and non-etched Si(115) substrates for piezoelectric component separated devices

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Abstract. We propose the application of an Si(115) substrate for the growth of the GaN semi-polar layer, the c-axis of which is inclined by an angle of 40 degrees. The calculations concerning built-in polarization fields in AlGaIn/GaN heterostructures of various crystal orientations will be presented, focusing on the separation of piezoelectric and spontaneous components for high electron mobility transistor (HEMT) type structures that are insensitive to the applied stress. Two potential ways of growing semi-polar GaN by means of the metalorganic vapor phase epitaxy (MOVPE) technique will be presented, including etched (patterned) and non-etched Si(115) substrates. The selected optimized etching solution will be presented along with the GaN growth procedure. Scanning electron microscope (SEM) imaging supported by high resolution X-ray diffraction (HRXRD) characterization results of semi-polar GaN will be discussed.

Keywords: metalorganic vapor phase epitaxy; semi-polar GaN; patterned Si substrates.

1. INTRODUCTION

Due to the lack of large-diameter and inexpensive GaN substrates, nitride growth is most commonly performed heteroepitaxially on sapphire or SiC. Another substrate extensively studied for this application is silicon (Si). Si substrates exhibit a number of beneficial properties such as the availability of large diameters, low price, and good thermal and electrical conductivity. Moreover, GaN-on-Si epitaxial structures can be used to integrate GaN-based devices with electronic devices commonly fabricated in Si (e.g. complementary metal-oxide-semiconductor (CMOS) structures, micro-electro-mechanical systems (MEMS)) [1]. To date, the most successful growth of GaN-based structures has been performed on (111) oriented Si substrates [2]. The main cause of this is attributed to the three-fold symmetry, meaning a hexagonal-like in-plane surface atom arrangement. The application of Si(111) substrates leads to c-axis oriented GaN growth. Recently, there has been growing interest in semi-polar and nonpolar GaN growth due to the possibility of controlling large spontaneous polarization fields, oriented along the hexagonal c-axis, that arise from non-centrosymmetric nature of GaN compounds and the presence of polar faces in the wurtzite structure [3,4].

Alternation of built-in polarization field distribution is obtained by inclining the c-axis of GaN from the normal to the substrate surface.

The main application driving the development of the semi-polar and nonpolar GaN growth is the light-emitting diode (LED) fabrication, due to the reduced Stark effect [5,6].

Standard LEDs and μ LEDs, fabricated on semi-polar GaN layers grown either on freestanding GaN substrates or applying structured sapphire substrates, have shown improvements in wavelength stability and reduced efficiency loss at high current density [7,8]. Recently the successful growth of [9,10] semi-polar GaN buffer layers, InGaIn multiple quantum wells (MQWs) and light-emitting diode (LED) structure on patterned silicon-on-insulator (SOI) substrates, made from a 6° off-axis (001) Si layer, has been demonstrated for the fabrication of semi-polar standard LED and μ LED [11].

As for (0001) oriented GaN-based heterostructures, grown on Si substrates, the most common application is HEMT (high electron mobility transistor) devices. During operation HEMT's are exposed to additional stresses, e.g. thermal stress due to ineffective cooling of the active area or environmental factors. These stresses lead to variations in electrical properties of the devices. One way to minimize those variations is to reduce the influence of the piezoelectric component and thermal expansion coefficient mismatch of GaN and Si. We propose the application of Si(115) substrates to achieve a semi-polar GaN layer (11–24), whose c-axis is inclined by an angle of 40 degrees. Assuming

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that GaN will grow with its c-axis aligned perpendicular to the Si(111) plane in (11x) oriented substrate, it will become possible to obtain a semi-polar GaN layer for application in devices with separated piezoelectric components [9]. The manipulation of piezoelectric polarization is crucial in the ongoing research on the influence of mechanical stress, through the piezoelectric effect, on the performance of AlGaIn/GaN heterostructure-based devices. Previous studies have focused primarily on non-etched (0001) sapphire substrates [10], which, however, do not allow for the MOCVD growth of semi-polar GaN layers. In order to obtain such layers, complicated and inefficient methods, e.g. magnetron sputtering, need to be used [12]. Given the increasing capabilities of GaN-on-Si technology and the benefits of reducing piezoelectric polarization in GaN layers, the use of silicon substrates, with alternative crystallographic orientations emerges as a natural progression.

The original method proposed by N. Sawaki *et al.* [13] did not achieve satisfactory results and failed to unlock the full potential of semi-polar GaN layers. Considering the growing number of articles on the impact of piezoelectric effects on device performance, we have developed a new method to grow semi-polar GaN on Si(115) substrates. In this approach, the Si(115) surface is etched without the use of a dielectric mask, exposing Si(111) planes that act as nucleation facets for GaN (0001) growth. As a result, after the coalescence of GaN blocks, the semi-polar planes become perpendicular to the Si(115) substrate surface. To the authors' knowledge, no one to date has tried to use silicon substrates with (115) orientation for the growth of semi-polar GaN layers for device applications [3].

Additionally, the primary advantage of this method over the approach originally proposed by N. Sawaki *et al.* [5] is the elimination of the dielectric mask during the etching and growth process, which simplifies fabrication and enhances the viability of semi-polar GaN growth on silicon substrates. We show that this novel method allows for the reconsideration of the viability of Si(115) substrates for GaN growth. The usefulness of mask-less lithography and anisotropic wet etching using tetramethylammonium hydroxide (TMAH) solution for the growth of semi-polar (1-101) GaN stripes on patterned Si(001) substrates for the fabrication of InGaIn/GaN MQWs LEDs, has been presented in the article [14].

2. EXPERIMENTAL DETAILS

The article presents two potential methods of MOVPE (metalorganic vapor phase epitaxy) semi-polar GaN growth on etched and non-etched Si(115) substrates. From our calculations, it was found that the HEMT devices can operate even without induced piezoelectric charge. Specifically, according to theoretical calculations, for semi-polar AlGaIn/GaN heterostructure, with 40 degree c-axis inclination, the total polarization induced charge is reduced down to around 50% of the maximum total polarization charge value, which is sufficient for efficient operation of devices with 2DEG (two dimensional electron gas). An additional benefit is the smaller thermal expansion coefficient (TEC) mismatch in the horizontal direction. However, the described

case is also interesting for strain-gated piezotronic transistor (SGPT) applications due to the piezotronic phenomenon, similarly to ZnO-based SGTs presented by Yan Zhang, Ying Liu, and Zhong Lin Wang [15], and piezotronic transistors based on GaN [16].

By altering the inclination angle of the GaN c-axis with respect to the surface plane, it is possible to control both spontaneous polarization fields and piezoelectric fields in a non-linear way [17]. In Fig. 1, the calculations of built-in polarizations as a function of the inclination angle of the c-axis are presented for Al_{0.3}Ga_{0.7}N/GaN heterostructure exposed to additional strain. The detailed model definition used for this calculation can be found elsewhere [18].

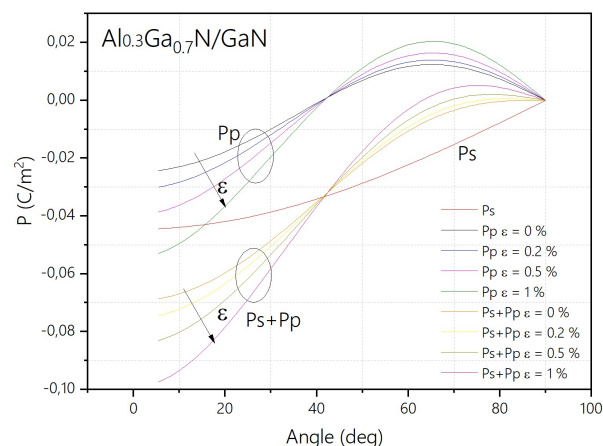


Fig. 1. Polarization charge in function of c-axis inclination angle to the normal to the surface for Al_{0.3}Ga_{0.7}N/GaN heterostructure. Piezoelectric (P_P) and spontaneous (P_S) components of polarization fields are denoted for selected induced strains, ε

It can be observed that for small c-axis inclination angles, the change in piezoelectric field-induced charge due to increasing stress exposure is larger, decreasing down to the point where the strain no longer influences the overall charge for the c-axis inclination angle of about 40 degrees. This leads to a conclusion that for a semi-polar AlGaIn/GaN heterostructure, where the c-axis is inclined by approximately 40 degrees with respect to the surface plane, it is possible to achieve HEMT-type structure insensitive to the induced stress. At this point the P_P component will be equal to zero.

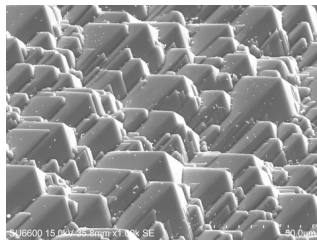
The GaN-based layers were grown using the MOVPE technique on 2", low-resistive, Si(115) substrates using an AIXTRON 3x2" close coupled showerhead system. The experiment was divided into two stages. The first one examined the growth of GaN directly on Si(115) substrates. In the second stage, GaN was deposited on Si(115) substrates etched in selected chemical solutions. The non-selective etching process was intended to expose the (111) planes of Si(115) substrate for the deposition of c-axis inclined GaN. The goal was to obtain a continuous semi-polar GaN layer. The following etching solutions were selected: potassium hydroxide (KOH), isopropanol (IPA), tetramethylammonium hydroxide (TMAH) and 1,5-pentanediol. The composition of solutions and etching time were subject of op-

timization. The solutions and etching conditions selected are presented in Table 1, along with the average radius of hillocks with exposed Si(111) planes. The radius was calculated using an image processing tool based on scanning electron microscopy (SEM) scans, presented in Fig. 2.

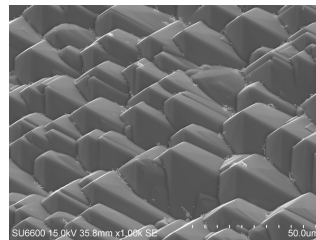
Table 1

Selected solutions and etching conditions, along with the average radius of etched hillock

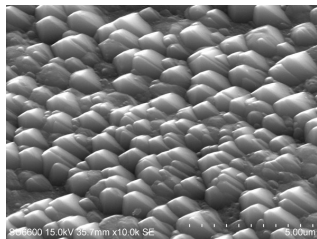
Etching solution	Etching temperature (°C)	Etching time (min)	Average radius of hillock with exposed Si(111) plane (nm)
A: 5M KOH + 10% IPA	80	27	1623
B: IPA saturated 5M KOH	80	33	752
C: 1M KOH + 2% 1.5-pentanediol	80	17	427
D: TMAH + 2% IPA	80	22	311



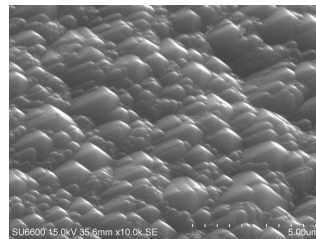
(a)



(b)



(c)



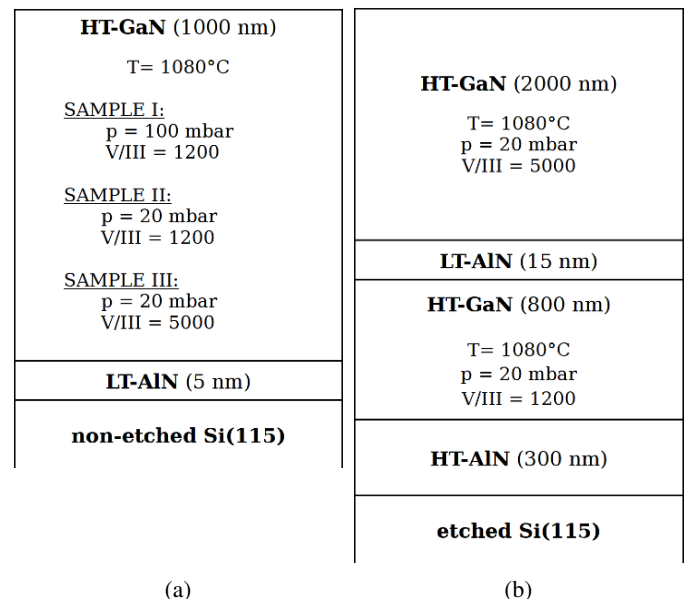
(d)

Please note that each image has a different scale

Fig. 2. SEM images of Si(115) substrates etched in four selected solutions: (a) A: 5M KOH + 10% IPA, (b) B: IPA saturated 5M KOH, (c) C: 1M KOH + 2% 1.5-pentanediol and (d) D: TMAH + 2% IPA

The selected results of GaN growth optimization on etched and unetched Si(115) substrates are presented in the next section. The two-layer schemes of GaN-based multilayer structures used on both: etched and non-etched Si(115) substrates are presented in Fig. 3. For both etched and unetched Si substrates, it was necessary to use a nucleation layer before GaN growth, in order to prevent etching of the substrate by gallium precursors during the MOVPE process. Typically, low- or high-temperature AlN layers are used for this purpose. The investigations demonstrated that for direct GaN epitaxy on non-etched Si(115), a thin

low-temperature AlN buffer layer is required to obtain semi-polar GaN [19], otherwise c-axis-oriented GaN is grown. On the other hand, using etched Si(115) substrates enables the utilization of HT-AlN seed layer and better protection from Ga-Si diffusion, which leads to melt-back etching [20], allowing at the same time to growth semi-polar GaN layers. Epitaxial growth of all investigated samples was conducted using ammonia (NH₃), trimethylgallium (TMGa) and trimethylaluminum (TMAI) in a hydrogen atmosphere. Prior to the epitaxial growth, the silicon substrates were thermally cleaned in H₂ atmosphere at 1100°C for 10 minutes. In order to minimize the parasitic nitridation of the silicon surface by ammonia, the AlN deposition was started with a 30-second pre-flow of the Al precursor. Only after this period was ammonia supplied to the reactor. In the case of unetched Si(115), a 5 nm thick AlN layer, grown at 680°C/100 mbar with V/III group mole fraction equal to 9000, was used as the nucleation layer. Subsequently, a 1000 nm thick GaN layer was deposited at 1080°C. Three type of samples, differing in growth pressure (20/100 mbar) and molar ratio of group V and III precursors (5000/1200), were examined. The growth conditions of samples deposited on non-etched Si(115) substrates are marked on the scheme (Fig. 3a). Epitaxial structure grown on etched Si(115) substrates was more complex. Due to high built-in tensile stress arising from the lattice mismatch between the substrate and layer, the growth of GaN layers thicker than 800 directly on Si results in cracking of the epitaxial structure. This undesirable phenomenon can be controlled to some extent by adjusting the growth conditions, but, as shown in the following discussion, it limits the production of semi-polar GaN on non-etched Si(115) substrates. For the structures grown on etched substrates, a stress engineering approach involving the use of AlN spacer was applied [21,22]. The epitaxial process was initiated with a 300 nm thick AlN layer deposited



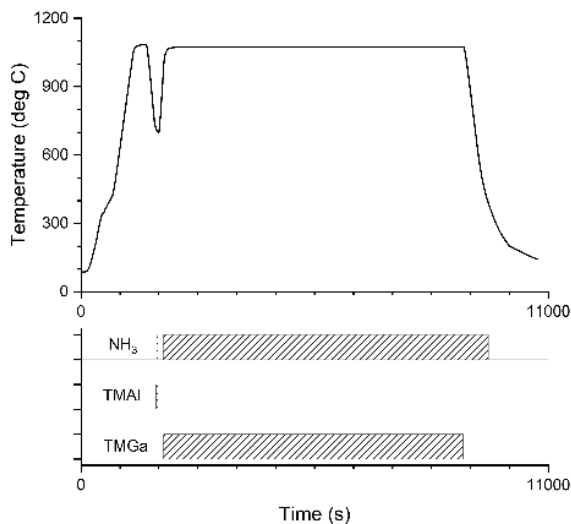
(a)

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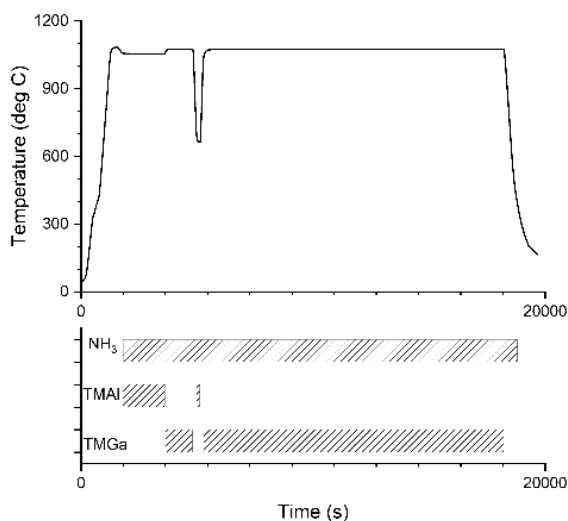
Fig. 3. GaN-based structures scheme for two sets of layers grown on: (a) non-etched and (b) etched Si(115) substrates; HT states for high temperature and LT states for low temperature

at 1060°C/100 mbar with V/III group mole fraction of 2600. Next, two GaN sub-buffer layers (800 nm and 2000 nm) were grown, separated by a 15 nm thick low-temperature AlN (LT-AlN) spacer. The layer schematics of the investigated structures with selected growth parameters are presented in Fig. 3b.

The epitaxial process, along with the temperature profile and precursor dosing times, is shown in Fig. 4a (on non-etched substrate – sample III) and in Fig. 4b (on etched substrate). Deposition of the material was interrupted during the temperature ramps.



(a)



(b)

Fig. 4. MOVPE growth scheme of investigated heterostructures deposited on: (a) non-etched and (b) etched Si(115) substrates

3. RESULTS AND DISCUSSION

All fabricated GaN samples were investigated using scanning electron microscopy (SEM). The SEM images of the GaN surfaces of the three selected samples grown on non-etched Si(115)

substrates are presented in Fig. 5. It can be observed that at lower reactor pressures (sample I: $p = 100$ mbar, II and III: $p = 20$ mbar) the degree of coalescence is higher, and for higher V/III precursor molar ratios (samples I and II: V/III = 1200, III: V/III = 5000), a fully planar layer is obtained.

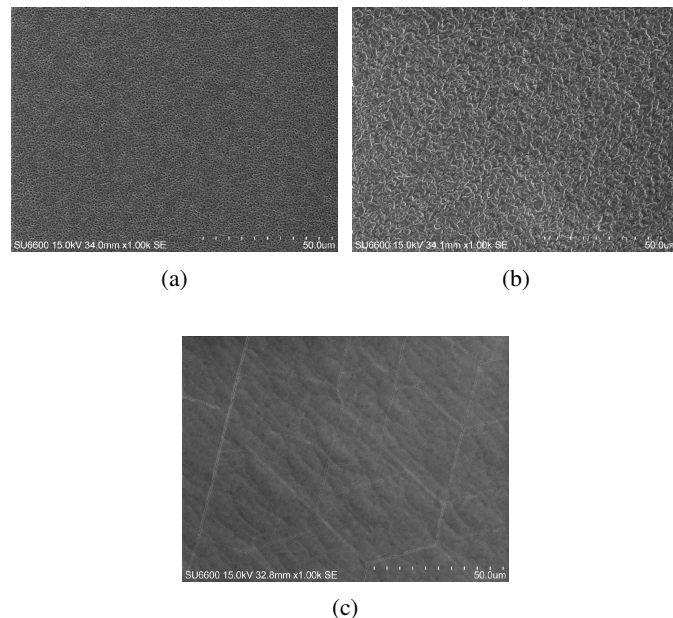


Fig. 5. SEM images of GaN-based samples (Fig. 3b) grown on non-etched Si(115) under three different sets of growth conditions: (a) $p = 100$ mbar, V/III = 1200, (b) $p = 20$ mbar, V/III = 1200, (c) III: $p = 20$ mbar, V/III = 5000

The SEM investigation showed highly misoriented GaN layers grown on Si(115) substrates etched in solutions A and B. When growth was performed of smaller etched Si hillocks (obtained using solutions C and D), it was possible for GaN to manifest a single dominant crystallographic orientation. However, complete coalescence remains an issue, as can be observed from SEM images presented in Fig. 6.

The HRXRD (high resolution X-ray diffraction) measurement of the samples grown was performed in a double-axis configuration, without an analyzer in front of the detector. Rocking curves were recorded for the (0006) reflection. The measurements were conducted at four rotation angles of the sample holder (on which the sample was mounted), allowing the determination of shifts in the GaN peak maximum positions, which enabled the evaluation of the c-axis inclination angle. The HRXRD plots for sample D (etched substrate) and sample III (non-etched substrate) are presented in Fig. 7.

The most promising result for GaN growth on etched Si(115) substrates was achieved using a 1M KOH + 2% 1.5-pentandiol solution on top of which an optimized GaN/LT-AlN/GaN/HT-AlN multilayer structure was deposited. The achieved inclination from the (0001) plane was 33.3°, resulting in (11-25) GaN with an ω scan (0006) FWHM of approximately 1800 arcsec, for GaN total thickness of 2800 nm. On the other hand, GaN-based sample grown under condition III ($p = 20$ mbar,

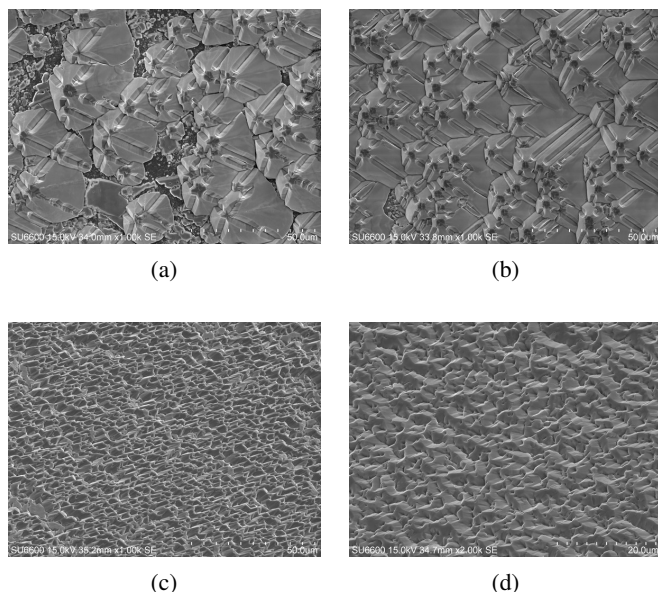


Fig. 6. SEM images of GaN-based samples (Fig. 3a) grown on Si(115) substrates etched in four selected solutions: (a) A: 5M KOH + 10% IPA, (b) B: IPA saturated 5M KOH, (c) C: 1M KOH + 2% 1.5-pentanediol and (d) D: TMAH + 2% IPA

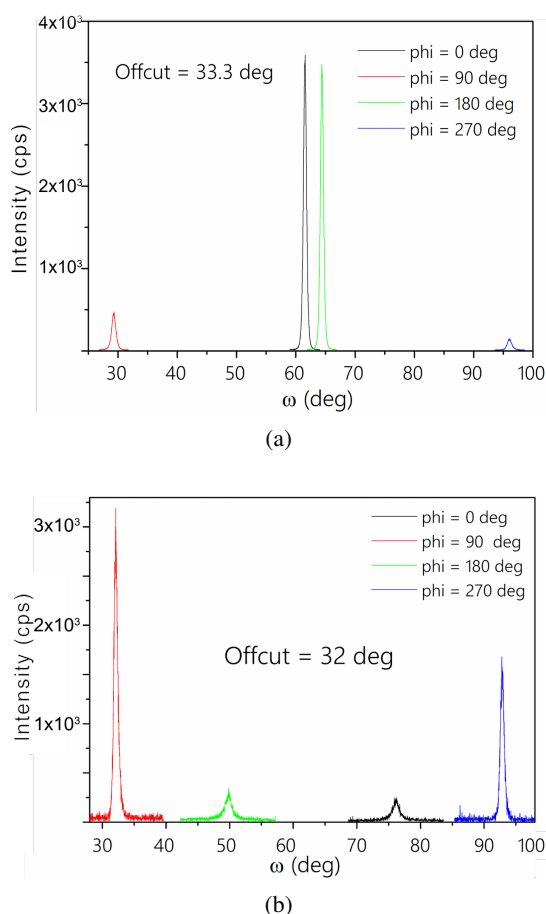


Fig. 7. HRXRD plots of GaN-based structures deposited on Si(115) substrate: (a) etched in TMAH + 2% IPA solution and (b) non-etched Si(115) substrate grown under conditions III: $p = 20$ mbar, $V/III = 5000$

$V/III = 5000$) on non-etched Si(115) reached an inclination from the (0001) orientation of 32° , resulting in (11-25) GaN with an omega scan (0006) FWHM around 1700 arcsec for GaN total thickness equal to 1000 nm. High FWHM values for the samples grown reflect the rather poor crystallographic quality of the deposited material, however an inclination of GaN(0001) toward Si(115) was achieved.

Finally, the GaN layer deposited on non-etched Si(115) under conditions III was continuous with optical flatness, whereas the other investigated non-etched samples exhibit island-like or polycrystalline structure. However, sample III is cracked due to high internal stress incorporated in the GaN/Si heterostructure. Further investigations, focused on the optimization of growth conditions, are necessary to achieve GaN crystal quality improvement.

4. CONCLUSIONS

The studies presented herein proved that it is possible to achieve GaN layer growth directly on Si(115), as well as on etched Si(115) by MOVPE with an inclination angles between 30° and 40° , closer to 40° . However, for etched surfaces the GaN layer exhibits a continuous but hillock-like structure.

High-resolution X-ray diffraction (HRXRD) and scanning electron microscopy (SEM) analyses confirmed that GaN grown on non-etched Si(115) under optimized conditions ($p = 20$ mbar, $V/III = 5000$) achieves an inclination of 32° toward the (0001) orientation with improved coalescence and optical flatness. However, high internal stress in these structures leads to cracking, indicating the need for further optimization. In contrast, GaN layers grown on etched Si(115) substrates, particularly those treated with a 1M KOH + 2% 1.5-pentanediol solution, exhibit a slightly higher inclination (33.3°) but suffer from incomplete coalescence and higher defect densities. The high FWHM values in HRXRD measurements reflect the challenges in achieving high crystallographic quality, emphasizing the necessity for additional refinement of the growth process.

This article also presents the application potential of such layers. According to the presented theoretical calculations and experimental results obtained using GaN structures grown on Si(115), it is possible to fabricate devices with a separated piezoelectric component of the built-in piezoelectric polarization field. This unique characteristic offers new opportunities in the design of GaN-based piezotronic devices, where controlled polarization fields are essential for improving device performance and functionality.

However, due to surface defects and the observed hillock-like morphology, the presented structures require further optimization to be viable for practical applications in piezotronic devices. Future research should focus on refining growth parameters, optimizing surface morphology, and reducing defect densities to enhance the structural and electrical properties of the layers. Additionally, exploring alternative substrate treatments or buffer layers may further improve GaN quality on Si(115), rendering it more suitable for piezoelectric component-separated structures. The results of this study provide a foundation for future advance-

ments in GaN/Si heterostructures with semi-polar polarization axis orientation, paving the way for innovative applications in electronics and optoelectronics.

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