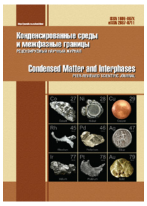

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Study of semi-polar gallium nitride grown on m-sapphire by chloride vapor-phase epitaxy

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Abstract

In this study, we analyzed the result of the influence of the non-polar plane of a sapphire substrate on the structural, morphological, and optical properties and Raman scattering of the grown epitaxial GaN film.

It was found that selected technological conditions for the performed chloride-hydride epitaxy let us obtain the samples of structurally qualitative semi-polar wurtzite gallium nitride with $(11\bar{2}2)$ orientation on m-sapphire. Using a set of structural and spectral methods of analysis the structural, morphological, and optical properties of the films were studied and the value of residual bi-axial stresses was determined. A complex of the obtained results means a high structural and optical quality of the epitaxial gallium nitride film.

Optimization of the applied technological technique in the future can be a promising approach for the growth of the qualitative GaN structures on m-sapphire substrates.

Keywords: GaN, AlN, m-Al_2O_3, chemical vapor-phase epitaxy

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1. Introduction

The growing interest in $A_{III}N$ semiconductors is caused by the capabilities of these compounds ensuring their application as a basis for the development of a wide range of new devices forming the backbone of the electronic component base[1].

Epitaxial $A_{III}N$ films in several cases are grown on affordable alien substrates (sapphire, silicon, silicon carbide) having wurtzite hexagonal modification. Nitrides from the third group Periodic table are polar materials meaning that they lack inversion symmetry.

The lack of inversion symmetry in the hexagonal crystal lattices of $A_{III}N$ compounds stipulates their polarization properties which are extremely important while considering the mechanisms of recombination in light emitting devices, for characterizing 2D electron gas in microwave transistors and other applications.

Large values of piezoelectric constants in the conditions of strong misfit stresses in III-N heterostructures result in the large values of piezofields ($< 10^7$ V/cm) and, hence, in a considerable spatial separation of charge carriers in the quantum-size heterostructures. This effect is widely used in microwave transistors based on AlGaN to obtain 2D electron gas with enhanced mobility. On the other hand, such separation in heterostructures with quantum wells (QW) is accompanied not only by the red shift of the effective width of the band gap but results in a decrease of the radiative recombination as well, due to a decrease of overlapping the electron and hole wave functions (Stark effect) [2–4].

That is why nowadays along with the studies of the synthesis features of the polar $A_{III}N$ compounds active investigations are performed concerning the development of technological approaches for the epitaxial synthesis of high-quality non-polar or semi-polar $A_{III}N$ compounds [5, 6].

To perform epitaxial synthesis of $A_{III}N$ nitride films with different orientations various technologies are used: molecular beam epitaxy (MBE), vapor phase epitaxy from metal-organic compounds VPE-MOS) and chloride vapor-phase epitaxy (Cl-VPE). The latter makes it possible to synthesize $A_{III}N$ layers with the highest growth rate and it proves to be the most advanced technology for the fabrication of bulk $A_{III}N$ layers of high quality [7–14].

In this work, the results of the study for GaN/AlN heterostructure are presented and obtained with the use of a set of structural-spectroscopic methods. The structure was grown on the substrate of m-Al_2O_3 by the Cl-VPE technique.

2. Experimental

Optimal technological conditions used in this work for growing AlN and GaN layers were determined in the series of preliminary experiments concerning the synthesis of AlN and GaN compounds by the Ch-VPE technique on the substrates of c-sapphire (Al_2O_3), silicon (Si), and silicon carbide (SiC/Si). It was also experimentally determined that the use of buffer AlN layers allows for avoiding the appearance of cracks in the process of growth of the main GaN layer considerably reduces the values of elastic deformations as well as it can specify the crystallographic direction of growth. For example, in [15] the authors managed to grow a semi-polar GaN layer in the direction of $[10\bar{1}3]$ on the substrate of the Si (100) plane with the use of an oriented buffer layer of AlN layer with a thickness of 600 nm in the direction of $[10\bar{1}2]$, $[10\bar{1}3]$ plane. While performing a series of experiments related to the deposition of the buffer AlN layers on the substrates of sapphire and Si optimal temperatures of the substrates in the range of 1080–1100°C were determined. The optimal temperature for deposition of the buffer AlN layer on sapphire substrate proved to be equal to $T = 1080$ °C, while for silicon it was equal to $T = 1100$°C. More detailed information on the epitaxial growth of AlN on the substrate of silicon is presented in [16].

In the present work, the growth of GaN/AlN heterostructure using the Ch-VPE method was performed on the substrate of m-sapphire (m-Al_2O_3) in two stages. In the first stage of deposition of the buffer, the AlN layer was performed at the temperature of $T = 1080$ °C. The time of deposition for the buffer layer was equal to 3 minutes. In the second stage deposition of the main GaN layer was realized on the buffer AlN layer at the temperature of $T = 1050$ °C.

Diagnostics of the samples were made with the use of a set of structural-spectroscope methods of analysis.

Microscopic studies were performed with the use of a scanning electron microscope (SEM) JSM-7001F (Jeol) and scanning probe microscope Femtoscan-001 (NT MDT).

X-ray diffraction data were obtained at room temperature with the use of diffractometer DRON-4-07 employing characteristic radiation of the cobalt tube.

Micro-Raman scattering spectra were obtained with the use of Raman microscope RamMix 532 employing laser excitation with the wavelength of 532 nm.

Photoluminescence spectra of the samples were obtained with the unit for measuring of photoluminescence and optical reflection Accent RPM Sigma. The studies were made at room temperature under laser excitation with a wavelength of 266 nm and W = 5 W/cm^2.

3. Results and discussion

Fig. 1 depicts an SEM image of the cross-section (cleavage) for the GaN/AlN/m-Al$_2$O$_3$ heterostructure. SEM data made it possible to determine not only the nominal thickness of the Ch-VPE grown AlN and GaN layers (presented in Fig. 1), but also to estimate qualitatively the features in the development of the layers morphology during the process of their growth by Ch-VPE technique. From Fig. 1 it is seen that AlN and GaN in the obtained heterostructures were grown in the form of a continuous layer. Buffer AlN layer was characterized by rather rough morphology while in the growth of the main GaN layer a gradual change of morphology occurred from the rough one observed near the GaN/AlN interface to more smooth morphology of the GaN layer. One should also note the lack of microcracks for both cases.

Images of the surface microareas of GaN epitaxial film grown on the m-plane of the sapphire substrate are presented in Fig. 2. It is seen that the film morphology can be characterized as a step-wise terrace with certain features of the surface in the form of feathers (V-shaped elements), oriented along (0001) direction of a sapphire substrate. This result coincides with the known literature data not only for GaN layers[10,17,18] but also for those of α-(AlGa)$_2$O$_3$ [19] grown on an m-plane of sapphire. In the chosen conditions of the epitaxial growth merging of semi-polar{11¯03} faces proved to be energy efficient that just resulted in

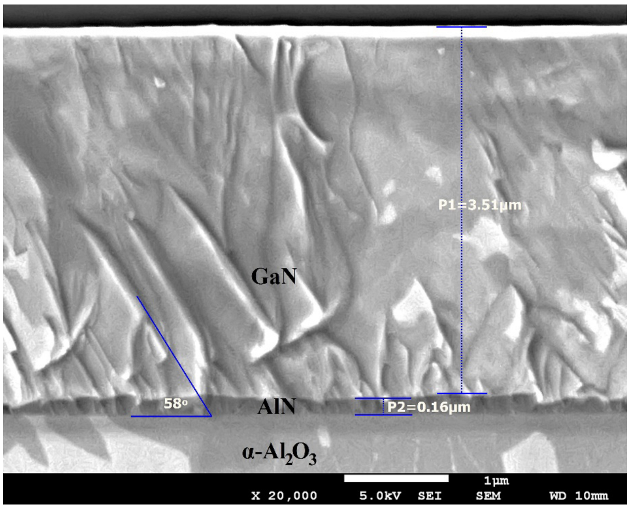

Fig. 1. SEM image of the cleavage for GaN/AlN/m-Al$_2$O$_3$ heterostructure

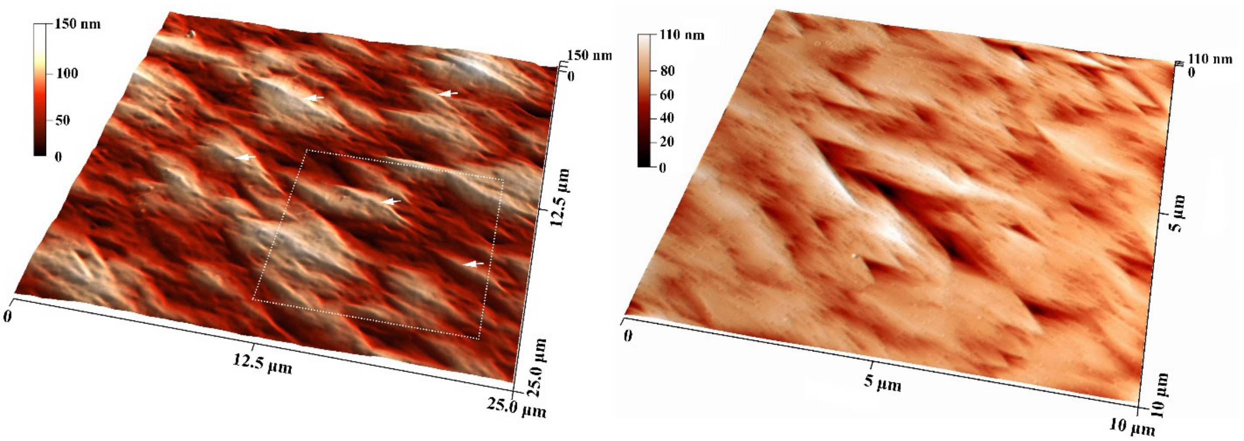

Fig. 2. AFM images of relief for microareas at the surface of GaN/AlN/m- Al$_2$O$_3$ heterostrucures

the formation of the observed surface relief [20]. This is just a coincidence with the discussions presented in [17], where semi-polar growth of GaN on m-sapphire was employed with the use of a vapor-phase deposition.

X-ray ω/2θ scan of GaN/AlN/m-Al_2O_3 heterostructure and m-Al_2O_3 substrate is given in figure 3 in the logarithmic scale of intensity. The diffraction profile is presented in the range of angles 2θ = 10–100°, where a number of the main diffraction reflexes is arranged.

The analysis demonstrates that the most intensive reflection here is related to $(30\bar{3}0)$ line of a sapphire substrate. It should be noted that there are other reflections in ω/2θ scan attributed to the diffraction from m-Al_2O_3 (this range of the angles is not shown in Fig. 3), which is connected with the presence of deviation of the substrate orientation from its primary flat. As for the epitaxial film, our experiments show that in θ/2θ scan there are two reflections observed at the angles greater than those inherent for sapphire substrate, which correspond to the diffraction from the plane $(11\bar{2}2)$ of wurtzite GaN and AlN layers.

Using elasticity theory [21–24] for the crystals with wurtzite crystal lattice deformation in the growth plane ε_{xx} *(along a axis)* can be determined in terms of the following relation [25]:

$$\varepsilon_{xx} = \varepsilon_{11-22} = \frac{d - d_0}{d_0} \qquad (1)$$

Here d and d_0 is the experimental interplanar spacing for the reflection $11\bar{2}2$, d_0 – experimental interplanar spacing for the reflection of $11\bar{2}2$ for the non-strained crystal of GaN, in accordance with the literature data d_0 = 1.3588 Å [26,27].

Next, according to the known correlation [28] it is possible to calculate bi-axial strains in the growth plane σ_{xx}:

$$\sigma_{xx} = M\varepsilon_{xx}. \qquad (2)$$

Here M is the biaxial modulus of elasticity for the crystalline material with a wurtzite crystal lattice and its value for GaN ~478.5 GPa. Calculations show that deformations in the growth plane are stretching ones (ε_{xx} ~ 0.00088, while the appearing biaxial stresses in the growth plane σ_{xx} are close to ~ 420 MPa.

It was repeatedly shown that Raman spectroscopy is a method for the determination of fine structural crystal properties and it is very sensitive to impurities, composition, crystalline structure, crystal orientation, and mechanical stresses.

Micro-Raman scattering spectra from the epitaxial heterostructure and the used m-sapphire substrate are presented in figure 4. Taking into account sample orientation and in accordance with the selection rule in the Raman spectrum of semi-polar GaN several maxima are observed that can be attributed to the phonon modes A_1(TO), E_1(TO), E_2^{high} for the crystal with a wurtzite

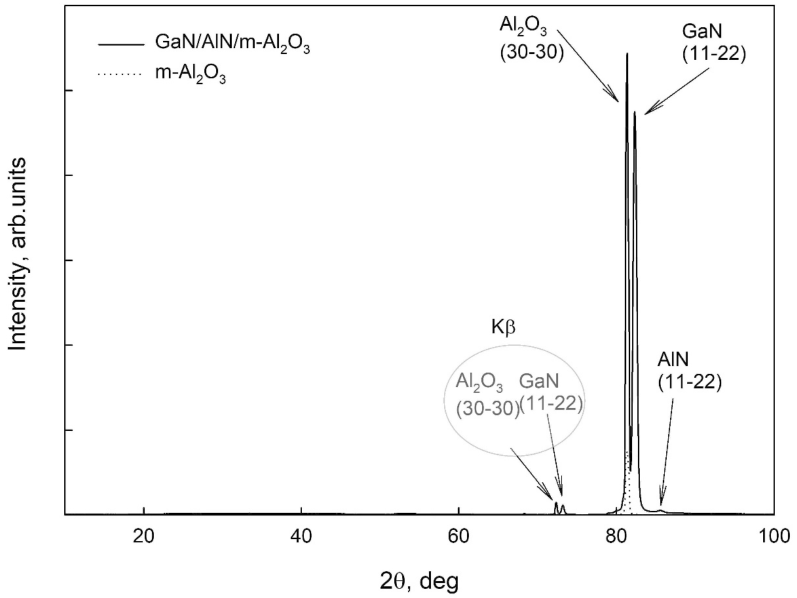

Fig. 3. X-ray ω/2θ scan of GaN/AlN/m-Al_2O_3 heterostructure

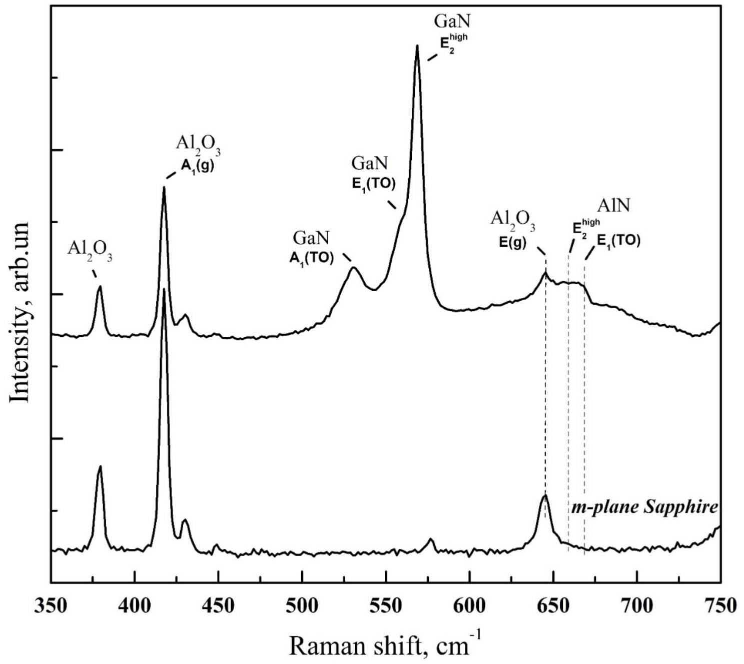

Fig. 4. Raman spectra from the epitaxial GaN/AlN/m-Al_2O_3 heterostructure and sapphire substrate.

structure [29]. Maxima in the scattering Raman spectra at 370, 417, 644 cm^{-1} represent typical characteristic combination-active modes of sapphire (Al_2O_3) with symmetry A_{1g} and E_{1g} [30].

Moreover, in the spectrum of the sample, there are two low-intensive vibrations in the range of 655 and 670 cm^{-1}. They can be attributed to the mode of E_2^{high} AIN, the strong among the allowed modes in the films of wurtzite AlN films, for the geometry of backscattering employed in our experiment *z*(*xy*)*z*′, and E_1(TO) mode of AlN, respectively [31, 32].

It is known that the intensity and full width at the half maximum (FWHM) of the phonon mode E_2^{high} in GaN represents the structural quality of the crystals in the epitaxial film. GaN films with a high dislocation density usually demonstrate the spectrum with a greater full width at the half maximum[29]. Based on the obtained results on micro-Raman scattering FWHM of the phonon mode E_2^{high} in the spectrum it is ~7 cm^{-1}. Comparison with the results of similar previous works shows that the width of E_2^{high} mode in the Raman spectrum of semi-polar $(11\bar{2}2)$ GaN was noticeably greater and it was within the 9.5–12 cm^{-1} range.

The presence of a narrow peak of E_2^{high} in GaN and an intensive peak of E_1(TO) in the Raman spectra indicate that high-quality semi-polar $(11\bar{2}2)$ GaN films were obtained in fact, almost without their deformation.

Estimation of the structural quality of the epitaxial film, namely, the value of stresses in the GaN layer, can be obtained based on the determination of the main maximum shift in the Raman spectrum.

The value of biaxial stress for semi-polar GaN film grown on m-sapphire can be estimated according to the following formula:

$$\Delta\omega = k\sigma \qquad (3)$$

where где s – is a value of residual biaxial stresses in the epitaxial layer, *k* – is the coefficient of the transformation for biaxial stress into Raman shift. The transformation coefficient for the E_2^{high} mode of GaN is equal to 4.3 (cm^{-1}·GPA^{-1}) [33]. The value of biaxial stress in the GaN layer calculated from expression (3) is of ~ 117 MPa and it means effective relaxation of elastic stresses in the epitaxial GaN layer. It should be noted that the determined value of residual biaxial stresses is 2,5 times lower than that one calculated based on the results of the XRD w-scan.

The optical quality of the epitaxial layer can be assessed from photoluminescence (PL) [34–36]. Figure 5 represents the PL spectrum of the GaN layer obtained at room temperature. It is seen as the main intensive peak with a maximum

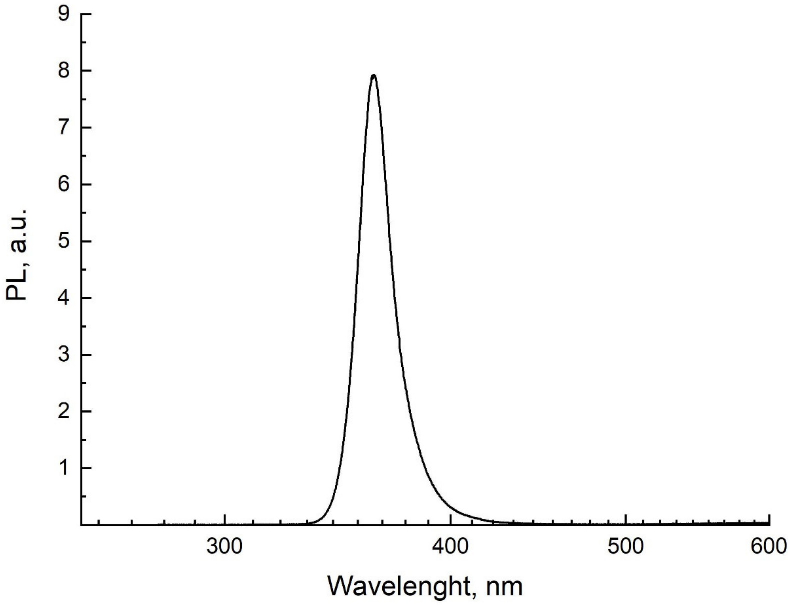

Fig. 5. PL spectrum of GaN/AlN/m-Al_2O_3 heterostructure at room temperature

localized near 3.4154 eV. The full width at half maximum of this photoluminescence peak is ~0.16 eV which is much less than the values presented earlier in the literature for GaN grown on m-sapphire. Nevertheless, the observed rather broad PL spectrum can indicate the relatively high density of defects in the grown layer [8, 9, 17].

It should be noted that no additional emission is observed in the spectrum in the low-energy range near 3.0–3.1 eV which is attributed either to highly localized excitons [9] or is connected with yellow luminescence thus indicating the quite good crystalline quality of the GaN layer.

It was repeatedly shown that PL is one more optical method providing information about residual stresses in the epitaxial film.

Quantitative level of biaxial stresses σ_{xx}, arising in GaN film, can be determined based on the fact that the band-gap width of the layer is sensitive to the deformation degree [37]. To do this the following empirical linear correlation was applied:

$$\Delta E = E_{PL} - E_0 = K_{PL}\sigma_{xx}. \qquad (4)$$

Here E_{PL} is the value of the band gap experimentally measured from the PL spectrum, E_0 is the energy of peak of the non-deformational PL at room temperature, while K_{PL} is the coefficient of the transformation of biaxial stresses into the linear shift of the material's band-gap value in the spectrum of photoluminescence. Energy value of E_0 = 3.4180±0.0008 eV, and the value of K_{PL} = -0.017±0.0025 eV/GPa; these values are based on the results obtained in [37].

Our calculations show that the value of residual stresses in the GaN layer determined from the PL spectrum, is about ~ 150 MPa, and this is comparable with the data of Raman spectroscopy. Note, that the PL spectrum at room temperature allows for a determination of stresses in a quite thin surface GaN layer since penetration depth for selected laser radiation wavelength is up to 100 nm.

4. Conclusions

The result of the effect of non-polar m-plane in the sapphire substrate on the structural, morphological, and optical properties as well as on Raman scattering of the epitaxial GaN film grown by Ch-VPE was analyzed in our studies.

It was found that selected technological conditions of Cl-VPE epitaxy made it possible to obtain samples of structurally qualitative semi-polar wurtzite gallium nitride with $(11\bar{2}2)$ orientation on m-sapphire. With the use of a set of structural-spectroscopic methods for analyzing various structural, morphological, and optical properties of the films and the level of residual biaxial stresses was determined as well.

Optimization of the applied technological technique can become a rather challenging approach to the growth of the qualitative GaN structures on the substrates of m-sapphire in the near future.

Contribution of the authors

The authors contributed equally to this article.

Conflict of interests

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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