Investigation of the mechanical properties of epitaxial gallium nitride for applications in MEMS

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Abstract. This article presents the results of the experimental determination of the mechanical parameters of a GaN films grown by molecular beam epitaxy on silicon substrates. The values of Young’s modulus and internal stresses for GaN films with different growth parameters have been determined. The study was carried out by contact and optical methods on the formed cantilever and beam microstructures. The measured values can be used to develop the design and evaluate the characteristics of MEMS based on gallium nitride films.

1. Introduction
In the last decades, there has been a continuous development of the technology of gallium nitride epitaxial growth on inexpensive silicon substrates. The development of these technologies allows to obtain significantly low cost high-quality films. First of all, this fact contributes to the wide distribution of these films in the microelectronic industry as a material for optoelectronics and high-power radio-frequency microelectronics. However, its piezoelectric properties, high chemical, temperature and radiation stability make it a promising material for the development of sensitive and actuating microelectromechanical systems (MEMS) [1].

The design of such sensor devices requires the development of both technologies for the growth of high-quality GaN layers and post-processing [2]. In this case, the electric and elastic properties, as well as the internal stresses of the resulting films, will depend on their growth parameters [3]. Thus creating MEMS devices with structural layers of GaN requires preliminary measurements of mechanical properties and evaluation of the process parameters influence on their properties. In this work, we studied the Young’s modulus and internal stresses for gallium nitride films grown by molecular beam epitaxy on silicon substrates.

On the technical point of view, a technology was developed for the post-processing of gallium nitride films grown by molecular beam epitaxy on silicon substrates to form mechanical structures. This technology is based on plasma-chemical etching of GaN structural layers with the subsequent release of mechanical structures. According to this technology, micromechanical structures were produced in the form of cantilever and bridge arrays of elements.

2. Sample preparation technique
The objects of the study were the n-type GaN films grown on silicon wafers by plasma-assisted molecular beam epitaxy using Veeco Gen 200 industrial type setup equipped with RF (13.56 MHz) plasma source Riber RFN-50/63. Semi-insulating (R > 10000 Ohm·cm) Si(111) substrates were prepared according to the modified Shiraki method [4]. Before the GaN growth, silicon substrates
were annealed at \( T=850^\circ \text{C} \) in order to remove \( \text{SiO}_2 \) film. The growth procedure began with the deposition of several Ga monolayers onto the silicon surface to prevent \( \text{Si}_x\text{N}_y \) formation. Then, the array of GaN nanocolumns as a seed layer was formed. In order to obtain continuous GaN layer the coalescence overgrowth of GaN nanocolumns was carried out. Details of the epitaxial growth will be discussed elsewhere. The morphology of the samples grown was studied using scanning electron microscope (SEM) Supra 25 Zeiss (see figure 1).

Figure 1. SEM image of the epitaxial GaN layer: (a) top surface; (b) side view.

As it can be seen, the coalescence overgrowth of the GaN nanocolumns allowed to obtain continuous GaN layer. The samples have quite smooth surface morphology, the total height of the GaN is about 770 nm. The Ga-polarity of the samples was confirmed by wet etching in KOH aqueous solution [5].

Post-growth technology was developed to form GaN micromechanical structures (see figure 2). Gold pads with chrome adhesive layer were deposited on the GaN layer by lift-off lithography and magnetron sputtering. This step was followed by \( \text{SiO}_2 \) mask deposition and lithography patterning. Anisotropic etching of GaN layer were carried out using Oxford Plasma Lab ICP 380 dry etching system with reactive plasma-chemical etching mode based on \( \text{Cl}_2/\text{BCl}_3 \) chemistry [2]. Then selective isotropic etching of silicon in SF6 plasma was performed using the same equipment to release GaN mechanical structures. Last stage was dry removing of silicon dioxide mask.

![Figure 2. Technological sequence](image)

Figure 2. Technological sequence (a) and SEM image of the GaN microbeams array (b).

The elastic parameters of the grown epitaxial films were determined by contact methods using Hysitron nanoindenter. The morphology and curvature of micromechanical structures were studied using scanning electron microscope (SEM) Supra 25 Zeiss.
3. MEMS mechanical parameters measurements

An approved methods [6, 7] were used to determine the mechanical parameters of GaN films using. The elastic parameters of the films were studied using contact methods. Measurements were made using a Hysitron nanoindenter with a Berkovich tip needle on a 5 x 5 point field. The needle force was 2 - 4 mN (elastic deformations boundary), while the maximum needle displacement was 112 nm. The measurement results and comparison with published data are shown in Table 1. The obtained values of Young's modulus were in the range of 200 ± 25 GPa and hardness of 15 ± 4 GPa, which corresponds to published data [1]. The obtained values are comparable with the values typical for bulk GaN single crystals and differ by about 1.5 times [8,9].

| Table 1. The measurement results. | Formation method | Hardness, GPa | Young’s modulus, GPa |
|----------------------------------|------------------|---------------|----------------------|
| Measur. results                  | MBE on Si, thickness 0.5 um | 15 ± 4 | 200 ± 25 |
| Ref. | Bulk [8] | 18 - 20 | 295 ± 3 |
| | MBE on Si, thickness 1 um [1] | 22.3 ± 1.6 | 261 ± 19.2 |

The built-in internal stresses of the GaN film were determined on fabricated MEMS. As test samples, beam structures of the three types were made: bridge (figure 3a), cantilever (figure 3b), and ring-shaped (figure 3c). SEM images clearly show deformations corresponding to each type of structure caused by internal stresses.

![SEM images of micromechanical structures of epitaxial GaN: (a) array of beams; (b) array of cantilevers; (c) ring microstructure.](image)

In the MEMS with bilateral fixation of the beam (figure 3a, 3c), a sinusoidal arc-shaped deformation profile of the beam was revealed, which corresponds to the loss of stability [11]. This indicates that the film was in a compressed state prior to release. This can be caused both by the silicon and GaN crystal lattices mismatch, as well as by thermal stresses caused by the difference in thermal expansion coefficients. The release of the film leads to its stretching and relaxation of internal stresses. The corresponding longitudinal deformations $\varepsilon$ of the released structure (1) and its length (2) are defined as:

$$\varepsilon = \frac{l-L}{L},$$  \hspace{1cm} (1)

$$l = \int_0^L \sqrt{1 + y'(x)^2} dx \approx L + \frac{1}{2} \int_0^L y'(x)^2 dx,$$  \hspace{1cm} (2)

where $L$ and $l$ are the beam lengths before and after release, respectively.

Stresses caused by these strains show the total internal stresses in the film. However, the circular profile of the released cantilever structure (figure 3b) indicates the heterogeneity of internal stresses.
across the film thickness. Such heterogeneity can be caused by relaxation of internal stresses from the lower damaged layer to the upper as the film grows. This is also confirmed by the direction of the film curvature, where tensile deformations at the lower boundary are greater than at the upper one. The formation of a stress gradient over the film thickness leads to the formation of a bending moment $M_b$ proportional to its curvature $K$ (3):

$$K = \frac{M_b}{EI} = \frac{1}{R},$$

where $E$ is the effective Young's modulus, $I$ is the moment of inertia, and $R$ is the radius of curvature of the profile of the micromechanical structure.

Based on the measurement results obtained and deformation strained film structures values of the internal stresses were calculated. Measurements of the arc-shaped profile of the beam microstructures (Figure 3a, c) showed the presence of longitudinal deformations of 0.006. Based on the strain data and Young's modulus, the total longitudinal internal stresses of the structure can be determined. These values are in the range 1.39 ± 0.12 GPa and are in good agreement with the literature data for structures grown by molecular beam epitaxy [10]. The curvature of the cantilever-type microstructure profile is uniform for an array of beams of different lengths ($L$ from 200 to 1000 μm) and amounted to 6.06 mm⁻¹. Based on the obtained data on the film curvature and the methodology [6], it is possible to determine the ratio of internal stresses between the upper and lower boundaries of the film under the assumption of a linear change in its value. According to the results obtained this ratio is 1.72.

4. Conclusions
Post-growth dry technology was developed to form GaN micromechanical structures. An experimental evaluation of the mechanical properties of epitaxial films was performed in order to use them as functional layers of MEMS sensors. The results showed the high promise of this material as a structural layer of MEMS devices.

Acknowledgments
The work was done as a part of the state assignment (№FSRM-2020-0008) of the Ministry of Science and Higher Education of Russian Federation.

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