Investigation of the piezoresistive properties and temperature coefficient of resistance of epitaxial GaN layers for applications in MEMS and thermal flow sensors

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Abstract. The paper presents the results of an experimental determination of the piezoresistive gauge factor and temperature coefficient of resistance of a GaN films grown by molecular beam epitaxy on sapphire substrates. The measured values were used to calculate the design and characteristics of the GaN hot-wires anemometer for the MEMS gas flow sensor.

1. Introduction
Due to its high chemical, temperature and radiation resistance, gallium nitride is a promising material for electronics and sensors for harsh environments [1]. For deposition of gallium nitride films, along with the metalorganic chemical vapor phase epitaxy (MOCVD) [2] and hydride vapor phase epitaxy (HVPE) [3], which are widely used in commercial manufacturing of devices, molecular beam epitaxy (MBE) is also used [4]. Reactive nitrogen in this process can be obtained both by thermal cracking of ammonia (NH\textsubscript{3}-MBE) [5, 6] and by activation of N\textsubscript{2} in plasma (PA MBE) [7]. The advantage of the latter method is the ability to grow films at low substrate temperatures and in the absence of hydrogen. Also as has been shown in [8, 9] the method allows growing GaN films with different polarities. The progress achieved in recent years in the field of synthesis and post-processing of epitaxial GaN films opens up promising opportunities for their use in microelectromechanical systems (MEMS) [10].

GaN sensitive elements can be used in miniature gas sensors, temperature, pressure and strain sensors, flow sensors, microphones and other MEMS. The metrological characteristics of such sensors depend on the GaN films synthesis method and its technological regimes, substrate type, electrode materials, film etching methods for the formation of sensing elements. For example, the experimental values of the gauge factor of GaN piezoresistors differ by an order of magnitude by different authors, who used different methods of GaN growth [11, 12]. Therefore, before developing a specific sensor design, you must first carry out an experimental estimation of the sensitivity of the grown GaN film electrical characteristics to changes in some environmental parameter. These experimental data are necessary to create an adequate numerical model of the prospective sensor and calculate its parameters.

The first part of this article presents the results of preliminary experiments to determine the piezoresistive and temperature sensitivity of conductivity of GaN films grown by the PA MBE on a sapphire substrate. The obtained data are used in the second part of the article, where the results of numerical calculations of a thermoanemometer with a sensitive element in the form of GaN strings are presented. Such a device is the basis of a new class of MEMS - thermal flow sensors [13], and can be
used in flow sensors for corrosive gases and liquids and thermal microphones for remote diagnostics of equipment operating in aggressive environments.

2. Experiments and results

2.1. Samples fabrication
The objects of the study were the silicon doped n-type GaN films with thickness of 300 nm, grown by PA MBE using Gen 200 (Veeco) industrial type setup on GaN/c-Al₂O₃ templates [9,14]. The latters were preliminary formed by MOCVD. According to the room-temperature Hall measurements the electron concentrations and motilities in the n-GaN layers were about \(3.75 \times 10^{19} \text{ cm}^{-3}\) and \(107 \text{ cm}^2/(\text{V} \cdot \text{s})\), respectively.

The synthesized samples were diced into rectangular pieces, 9 mm × 3 mm in dimensions, by laser scribing method. Ti(20 nm)/Al(500 nm) metal stack was then deposited on the GaN surfaces of the samples by magnetron sputtering with preliminary ionic treatment of the surface to form non-alloyed ohmic contacts. Wirebonding was carried out using sintered silver paste to connect metal pads to the external instrumentation. The measured current-voltage characteristics of the samples were linear up to 16 V, which proves the non-rectifying nature of the contacts. A typical sample resistance was close to 200 Ω.

2.2. Measurement of TCR
A test bench was assembled to measure the temperature coefficient of resistance of GaN films. The bench consisted of a thermal chamber with temperature control in the range of 20–350 °C, a unit for measuring the sample resistance and a unit for measuring the sample temperature. A sample with a GaN film was mounted in close proximity to a platinum temperature sensor (Pt1000) inside a metal-glass package. Each resistance measurement was carried out after temperature stabilization for 15 minutes and was continued for 10 minutes to determine possible deviations. The temperature dependence of the GaN film conductivity in the temperature range 20-160 °C is shown in Figure 1.

![GaN conductivity versus temperature](image)

**Figure 1.** GaN conductivity versus temperature

The choice of the measurement range corresponded to the distinctive temperature range of Pt sensitive elements of conventional hot-wire and calorimetric MEMS thermo-anemometers. In this range, the temperature dependence of the GaN conductivity was close to linear. This is an advantage for the subsequent processing of the sensor output signal by an electronic circuit. The GaN TCR values are close to the TCR of platinum thermistors in order of magnitude.
In the future, the assembled test bench and measurement technique are planned to be used to determine the TCR of the grown GaN films. These data are important for the development of the temperature sensors, heat and mass flow sensors, as well as for making appropriate corrections to compensate for temperature deviations of other GaN MEMS.

2.3. Measurement of gauge factor
The piezoresistive effect of the materials is described by a gauge factor (GF). It’s the ratio of relative change in electrical resistance to the mechanical strain in the film.

In our work a special test bench was assembled in order to measure the GF of GaN. In this test bench the synthesized samples were placed on a special pedestal without rigid fixation. This arrangement avoids stress concentrations near fixing points. In the center area, the sample was subjected to external loading in the transverse direction using calibrated weights. In this case, longitudinal deformations (tensile or compressive strain, depending on the direction of sapphire deformation) appear in a thin GaN film. The magnitude of these deformations was calculated using numerical simulations in the COMSOL Multiphysics program, based on the known elastic properties of the sapphire. The resulting stress gradient in the GaN film led to a change in its resistance, which was measured using the test circuit.

In the experiments, the samples were loaded with weights in the range of 400 - 1200 g. At higher loads, the destruction of the samples was observed which is probably due to the fact, that sapphire has a high stiffness with a sufficiently high brittleness, which limits the range of allowable values of sample deformation. The GaN film strain gradient range, corresponding to the used loads, was 1.0003 - 1.0007. Each sample was subjected to multiple cyclic loads in this range and the measured resistances were averaged. Thus, we measured the static value of GF. It should be noted that in our experiments the dynamic GF associated with the piezoelectric properties of GaN [11] was detected at the moments of loading and unloading, but was not measured. All measurements were carried out at room temperature.

After a mathematical calculation of the experimental results, the GF of GaN was estimated as 24 ± 2. This value is significantly inferior to monocrystalline silicon (GF = 150-200), but exceeds the GF of metals (GF = 1-5) by an order of magnitude and is comparable to the GF of some semiconductor materials (GF = 30 for poly-Si and SiC), which are successfully used in modern pressure sensors for harsh environments.

Estimation of GF GaN is important for the development of GaN resonators, strain and pressure sensors, as well as for calculating the characteristics of temperature sensors in which the change in resistance due to external deformations can lead to an increase in measurement errors.

3. FEA of thermal flow sensor prototype
The obtained values of TCR and GF prove that GaN epitaxial films can be used as a sensitive elements of MEMS thermal transducers, such as a thermo-anemometers. These devices, based on conventional materials (Pt, poly-Si), have already become widespread in the field of measurement of the dynamic characteristics of mass and heat flows in neutral gases and liquids. On the basis of calorimeter-type transducers, it is possible to manufacture acoustic velocity sensors with a narrow polar pattern for miniature direction-finding devices. They are also applicable for remote sound diagnostics and early detection of faults in industrial lines and mechanisms, gas pipelines, turbomachines, chemical reactors, etc. GaN thermistors, possessing high strength and resistance to aggressive environment, can in some tasks replace conventional materials of thermo-anemometers sensitive elements.

Therefore, on the basis of our experimental data on the temperature dependence of GaN conductivity given in section 2.2, we developed the design of a GaN thermo-anemometer and performed its numerical simulation in the COMSOL Multiphysics program. The sensor design is shown on the geometric model in Figure 2. The device includes the semiconductor structure which consists of 800 nm thick Ga-polar GaN layer, grown by PA MBE atop of 300 µm thick semi-insulating silicon substrate which was preliminary nitridated in order to form thin Si₃N₄ interlayer. The
details of the PA MBE synthesis of the structure are described in [9]. The square contact pads (Ti-Al-Ni-Au) were formed on the GaN surface of the structure by lift-off lithography. Sensitive elements in the form of narrow GaN strings were formed by plasma etching. The cavity under the strings was made by selective chemical etching of silicon. The geometric dimensions of the GaN strings were 800 x 20 x 0.7 µm, and their height above silicon surface is one of the adjustable parameters of the numerical model. An example of a thermal convection cloud that forms around strings when a current is passed through them is shown in Figure 3. The thermo-anemometer operating principle is based on measuring the difference in resistance of two GaN hot-wires, which occurs when the thermal cloud is shifted from one string to another by the ambient gas flow. Depending on the vector of the flow velocity, the temperature difference between the two strings will be different. The calculation of the GaN anemometer characteristics required an interconnected solution of the problem in the mechanical and electrical domains, and in the domains of heat and mass flow. The reference data on the mechanical and thermal properties of GaN, Si, Si₃N₄ and our experimental data on TCR and GF of GaN were used. In our calculations, we studied the effect of laminar air flow at low flow rates (up to 1 cm/s) on the sensor output signal and took into account the influence of the angle of attack (AoA), i.e. flow velocity vector relative to the plane of the hot-wires.

Figure 2. Model of GaN hot-wires anemometer

Figure 3. Thermal cloud around the GaN strings (at U = 5 V, v_gas = 1 cm/s, AoA = 5˚)

Unlike the classical macroscopic case of flow measurements (for example, in turbomachines), in the MEMS anemometer, hot-wires are located near the silicon surface, the distance to which is determined by the substrate etching depth (of the order of several tens of microns). This fact and the viscous effects in the gas lead to a significant drop in the velocity of the flowing surface of the GaN strings. The distribution of the velocity of the air flowing around the sensor at an input flow rate of 1 cm/s and AoA of 5˚ is shown in Figure 4. It can be seen that in the air layers, which are at the level of the hot-wires, the flow velocity drops by an order of magnitude compared with the undisturbed flow, and, therefore, does not lead to a significant displacement of the convection cloud and a changes in the temperature and resistance of both GaN strings. At the same time, changing the angle of attack changes the flow velocity distribution. Figure 5 shows the two-dimensional distribution of the flow velocity over the center of the chip (vertical line in Fig. 4), depending on the height above the chip surface and the value of AoA. It can be seen that the highest flow velocity in the region of the location of hot-wires occurs at an angle of attack of 45˚.
Figure 4. Laminar flow velocity distribution over the chip surface (“0” – level of GaN hot-wires)

These regularities are typical for MEMS anemometers and are important for a better understanding of some aspects of their technology, depending on the application. So, with an etching depth of the silicon substrate under GaN strings over one hundred microns, this design can be used as a gas flow meter with a sensitivity at the level of conventional analogs. In addition, with a different arrangement of GaN strings with respect to the flow, this design can be used as a gas molecules velocity vector sensor or as a gas acoustic sensor (thermal microphone). The result of our calculations demonstrates that the magnitude of the displacement of the thermal cloud above the GaN strings due to a change in the angle of attack is sufficient to form a narrow directional pattern of such sensors. In Figure 6, this polar pattern is shown as the dependence of the difference in resistance of hot-wires on the angle of attack. The calculations also showed that the change in hot-wires resistance due to the piezoresistive effect in GaN under mechanical impact of the incident gas flow is negligible (the previously obtained value of GF = 28 was used as the upper estimate, since with increasing temperature GaN GF drops [12]).

To determine the frequency characteristics of the thermal microphone, numerical simulation was performed in the time domain with sinusoidal oscillations of air molecules surrounding the strings. The results of the study are shown in Figure 7. It can be seen that in the low-frequency range, which is typical for acoustic diagnostics of industrial equipment (at frequencies up to 500 Hz), the output signal amplitude remains at a fairly high level.

Figure 5. Flow velocity versus AoA and height above the chip (“0” – level of GaN hot-wires)

Figure 6. The difference in resistance of GaN hot-wires versus AoA (sensor polar diagram)

Figure 7. The difference in resistance of GaN hot-wires versus acoustic frequency (at U=10V)
Thus, the obtained experimental and data testify to the prospects of using epitaxial gallium nitride synthesized by PA MBE as a material for sensing elements of thermal flow meters and acoustic velocity sensors. The work will be continued further to ensure the technological production of prototypes of these devices.

4. Conclusion
The temperature coefficient of resistance of GaN film synthesized by PA MBE has been measured and its piezoresistive gauge factor has been estimated. The obtained data were used to carry out FEA simulation of the characteristics of a MEMS anemometers with gallium nitride hot-wires sensing elements. The low piezoresistive sensitivity of the GaN layers eliminates the change in hot-wires resistance under mechanical influence of the incident gas flow. The results show the prospect of using of GaN films in a gas flow sensor.

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References
[1] Rais-Zadeh M, Gokhale V J, Ansari A, Faucher M, Cordier Y and Buchaillot L 2014 J. Microelectromech. Syst. 23 1252–71
[2] Wu X H, Brown L M, Kapolnek D, Keller S, Keller B, DenBaars S P and Speck J S 1996 J. Appl. Phys. 80 3228–37
[3] Bessolov V, Konenkova E, Shcheglov M, Sharofidinov Sh, Kukushkin S, Osipov A and Nikolaev V 2013 Phys. Stat. Sol. C 10 433–6
[4] Arthur J R 2002 Surf. Sci. 500 189–217
[5] Mesrine M, Grandjean N, and Massies J 1998 Appl. Phys. Lett. 72 350–2
[6] Mizerov A M, Jmerik V N, Kaibyshev V K, Komissarova T A, Masalov S A and Ivanov S V 2009 Semiconductors 43 1058–63
[7] Jmerik V N, Mizerov A M, Shubina T V, Listoshin S B and Ivanov S V 2007 Tech. Phys. Lett. 33 333–6
[8] Shubina K Yu, Berezovskaya T N, Mokhov D V, Mizerov A M and Nikitina E V 2017 Tech. Phys. Lett. 43 976–8
[9] Mizerov A M, Timoshnev S N, Sobolev M S, Nikitina E V, Shubina K Yu, Berezovskai T N, Shstrom I V, and Bouraveulev A D 2018 Semiconductors 52 1529–33
[10] Shubina K Yu, Berezovskaya T N, Mokhov D V, Morozov I A, Kotlyar K P, Mizerov A M, Nikitina E V and Bouraveulev A D 2018 Semiconductors 52 2117–9
[11] Bykhovski A D, Kaminski V V, Shur M S, Chen Q C and Khan M A 1996 Appl. Phys. Lett. 68 818–9
[12] Tilak V, Vertiatchikh A, Jiang J, Reeves N and Dasgupta S 2006 Phys. Stat. Sol. C 3 2307–11
[13] Balakrishnan V, Phan H-P, Dinh T, Dao D V and Nguyen N-T 2017 Sensors 17 2061-91
[14] Kukushkin S A, Mizerov A M, Osipov A V, Redkov A V and Timoshnev S N 2018 Thin Solid Films 646 158–62