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Application of plasma chemical treatment for manufacturing of instrument microstructures based on gallium nitride

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Abstract. The epitaxial layers of n-n⁺-GaN were processed by plasma-chemical etching using a Sentech SI 500 unit equipped with an inductively coupled plasma source. The regimes of gallium nitride processing in chlorine plasma have been established, which make it possible to remove epitaxial layers of the semiconductor down to a depth of 10 μm with a smooth surface. Based on the obtained processing results, prototype samples of Schottky diode microstructures with quasi-vertical contact geometry were manufactured. The effect of pretreatment on the characteristics of instrument microstructures is demonstrated.

1. Introduction

Materials based on A3B5 compounds, in particular, semiconductor nitrides, can be used to form instrument microstructures for electronic and optoelectronic equipment. Their advantages associated with the superiority of a number of electrophysical characteristics (large band gap, high electron mobility, high critical electric field strength and high electron saturation drift rate) lead to the possibility of their use in a wide range of applications. These include structures with Schottky barriers for power electronics devices, high-power microwave transistors and monolithic integrated circuits, injection lasers and LEDs in the range of short wavelengths [1, 2].

The growth of epitaxial layers of semiconductor nitrides is usually carried out on substrates of sapphire, silicon carbide, or silicon [3]. When forming instrument microstructures on such substrates, it is necessary to use the etching operation to form areas for contact metallization and inter-element isolation.

Significant limitations of liquid etching methods related to the properties of the source material lead to the need of developing of plasma etching methods.

For etching of semiconductor nitrides, it is necessary to use a denser plasma of high uniformity. This plasma can be created by an inductively coupled high-frequency (RF) discharge.

The use of such a plasma when using a chlorine-containing medium allows processing epitaxial structures based on nitrides. Gas mixtures including boron trichloride (BCl₃) and pure chlorine (Cl₂) with additives of inert gases such as argon (Ar) or nitrogen (N₂) are the most popular and allow obtaining a fairly wide range of changes in the etching rate [4, 5].

The purpose of this work is to develop a technology for processing structures based on gallium nitride in RF discharge plasma in the Cl₂/BCl₃/Ar medium to obtain instrument microstructures of Schottky diodes with quasi-vertical contact geometry.



2. Experimental methods

The structures were processed at the Sentech SI-500 plasma chemical etching unit (Figure 1) using an inductively coupled plasma source (ICP source).

By selecting the parameters of the plasma process (power of the source of inductively coupled plasma, high-frequency power and pressure in the reactor), processing modes were determined that allow removing the semiconductor to a depth of 10 microns.



Figure 1. Standard Sentech SI-500 etching unit.

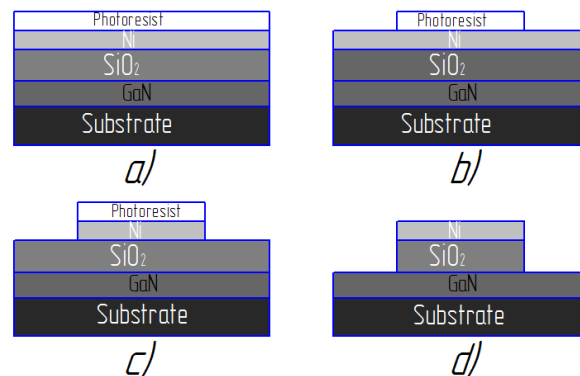


Figure 2. Formation of a SiO₂/Ni mask for etching gallium nitride: a – application of photoresist; b – creating a mask from photoresist; c – nickel ion etching; d – plasma-chemical etching of silicon dioxide and removing photoresist.

The test samples were undoped layers of gallium nitride with a total thickness of about two micrometers, grown on 2-inch diameter sapphire substrates by chemical deposition from the method of chemical deposition from the gas phase using metal-organic compounds.

To form the surface relief, a protective mask based on a layer of nickel (Ni) with a thickness of 0.25 microns and a sublayer of silicon dioxide (SiO₂) with a thickness of 0.3 microns was used. The sequence of technological operations for mask forming is shown in Figure 2.

The thickness of the layers and the etching profiles were controlled by using optical profilometer Talysurf CCI-Lite.

When choosing masking coatings, it should be considered that they have chemical resistance to the aggressive plasma of gas environment, have high adhesion to the substrate, and are easily removed after etching without changing the surface morphology of the semiconductor [6].

The formation of the mask begins with plasma chemical deposition of the SiO₂ film. Silicon dioxide films were grown using monosilane with a flow rate of 155 cm³/min and oxygen with a flow rate of 8.2 cm³/min at a source power of inductively coupled plasma of 150 W, a high-frequency power of 5 W, a chamber pressure of 5 Pa and a temperature of 250 °C. The nickel film was sputtered by magnetron method with heating of the substrate to 250 °C. After that, using optical photolithography, a photoresist mask was formed (Figure 2a), representing an alternation of open and closed surface areas. To preserve the anisotropic profile, the removal of nickel and silicon dioxide layers was performed using dry etching methods, namely, ion etching for nickel and plasma-chemical etching for SiO₂ (Figure 2c). After etching of the SiO₂/Ni layers, the photoresist was removed in dimethylformamide (Figure 2d).

In the experiment for the processing of structures based on gallium nitride, etching rate was determined on the semiconductor plasma process parameters, in particular, pressure in the reactor, a power of the source of inductively coupled plasma, RF power.

3. Determination of etching modes

3.1 influence of reactor pressure on etching parameters

The pressure in the reactor affects the main processes that are responsible for plasma etching. Lowering of the pressure leads to an increase in the average free path length, which in turn increases the energy of the ions bombarding the sample. The etching rate increases with intensification of ion bombardment and therefore lower pressure can contribute to etching process.

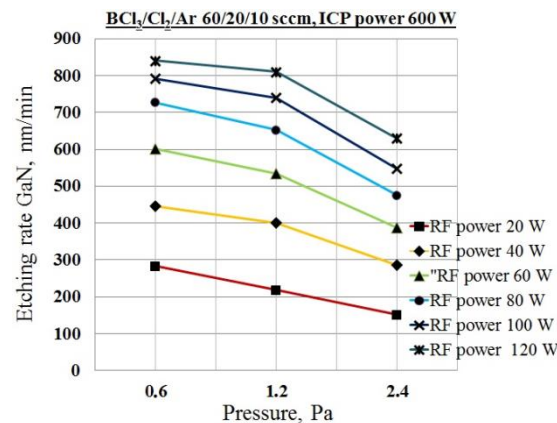


Figure 3. The dependence of etching rate of gallium nitride on the pressure in the chamber.

When the pressure in the chamber increases from 0.6 Pa to 2.4 Pa (Figure 3), the etching rate of gallium nitride decreases monotonously due to a decrease in the concentration of Cl_2 radicals in the plasma, or a decrease in the ion flow reaching the substrate surface.

3.2 Effect of inductively coupled plasma source power on etching parameters

Figure 4 shows the effect of inductively coupled plasma source power (ICP power) on the etching rate of gallium nitride. The ICP power in the plasma process regulates mainly the plasma density. As the plasma density rises with increasing of ICP power, the substrate receives a larger number of reactive particles that enhance the chemical aspect of etching. At the same time, the etching rate increases. Figure 4 shows that the etching rate of gallium nitride rises from ~ 15 to ~545 nm/min with an increase in ICP power from 25 to 700 W with a high-frequency power value (RF power) of 20, 40 and 60 W.

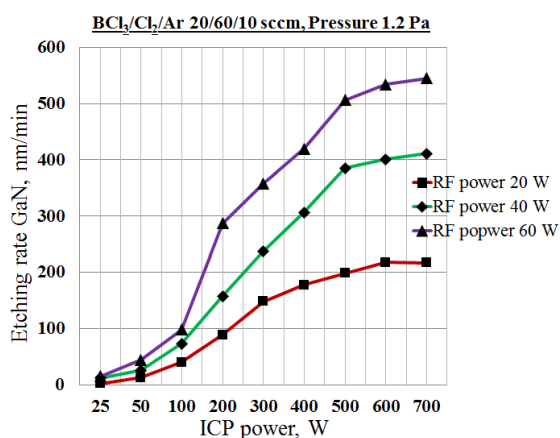


Figure 4. Dependence of the etching rate of gallium nitride on ICP power.

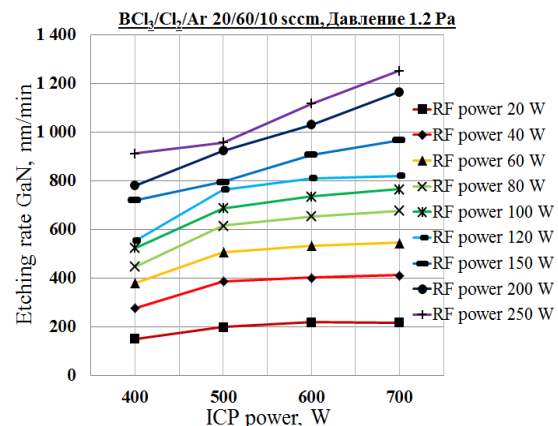


Figure 5. Dependence of etching rate of gallium nitride on ICP power.

At the same time, as can be seen from Figure 5, at small values of high-frequency power (up to values of the order of 60 W), the saturation of the etching rate is observed when the value of the ICP power of 600 W is reached. Saturation of the etching rate in this case can be caused either by saturation of reactive particles on the surface, or by desorption of reactive substances on the surface before reactions occur.

3.3 Effect of high-frequency power on etching parameters

High-frequency power in the plasma process increases the energy of electrons and, consequently, increases the probability of ionization. As a result, the etching rate increases with increasing RF power (Figure 6).

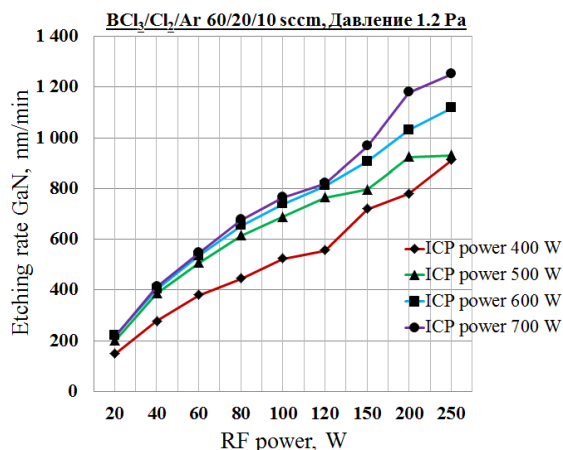


Figure 6. The dependence of etching rate of gallium nitride on RF power.

Figure 6 shows the dependence of etching rate of gallium nitride on RF power, where it is shown that the etching rates rise monotonously with increasing RF power. The etching rate rises by increasing the contribution of the physical etching component combined with a strong chemical etching component. An increase in the etching rate is associated with improved sputtering of etching products, and more effective destruction of bonds in the semiconductor with an increase in ion energy.

During the experiments, the etching depth and profile, as well as surface morphology, were monitored using optical profilometry, scanning electron microscopy, and atomic force microscopy (Figures 7-9). Figure 7 shows an example of measuring of the etching depth on a Talysurf CCI profilometer. Etching mode parameters: ICP power 600 W, RF power 100 W, pressure 1.2 Pa, gas consumption ($\text{BCl}_3/\text{Cl}_2/\text{Ar}$) 20/60/10 cm^3/min .

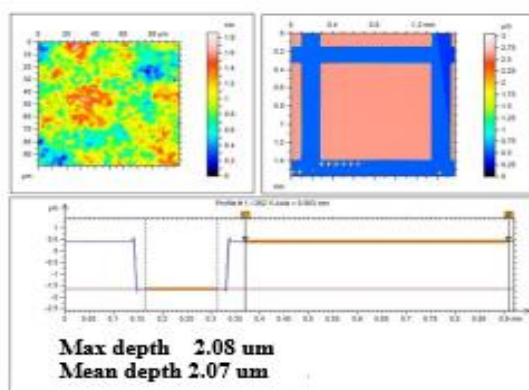


Figure 7. Results of etching depth measurements with a profilometer.

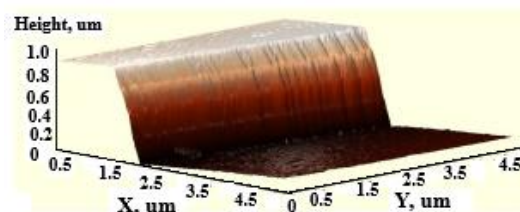


Figure 8. The measurement results of the etching depth using atomic force microscope.

Analysis of the etching profile and surface morphology (Figures 7-9) showed that the tilt angle of the side walls in the above mode was about 70° , and the value of the mean square roughness of the etching area is about 3 nm.

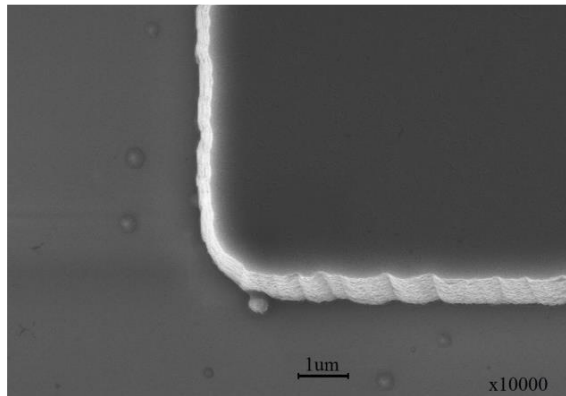


Figure 9. Image of the etching area obtained using a scanning electron microscope.

Thus, as a result of the experiments, the modes of deep etching of gallium nitride were determined: ICP and RF power 600 W and 100 W respectively, pressure 1.2 Pa, gas flow ratio $\text{BCl}_3/\text{Cl}_2/\text{Ar}$, equal to 20/60/10 cm^3/min . This mode provides optimal etching rate while maintaining the resistance of the protective mask and obtaining a plane-parallel and smooth surface of the semiconductor.

4. Formation of instrument microstructures

The results obtained during etching experiments were used in the formation of instrument microstructures of Schottky diodes based on gallium nitride. An epitaxial structure of the following type was used as the starting material: an undoped n-GaN layer 50 nm thick, an n-GaN layer 5.0 microns thick, and an n^+ -GaN layer with a thickness of 1.0 microns and a buffer layer. Directly in the growth chamber, the structures were passivated with a layer of silicon nitride 1.7 nm thick. The epitaxial structure was grown on 430 microns thick sapphire substrates by chemical deposition from the gas phase using metal-organic compounds

Mesa isolation was formed on such structures and windows were opened down to the n^+ -GaN layer by etching in the above mode. In this case, the mesa insulation was etched to the substrate (7.6 microns), and the etching depth down to the n^+ -GaN layer was about 5.6 microns. The value of the mean square roughness at this etching was ~ 3 nm.

Ohmic contacts were formed to the opened areas of n^+ -GaN by deposition of Si/Ti/Al/Ni/Au multilayer metallization followed by annealing at a temperature of 600°C for 45 seconds in a nitrogen atmosphere. Ohmic contacts were formed using a process of "lift-off" photolithography using a two-layer system of LOR 10A and S1813 photoresists. The use of a two-layer system based on LOR photoresist allows forming a negative wall slope, which facilitates the process of metallization removing and provides a smooth edge of the contact pads, as shown in Figure 10.

As a result, contacts with a specific contact resistance of $\sim 2 \times 10^{-6} \Omega \times \text{cm}^2$ were fabricated.

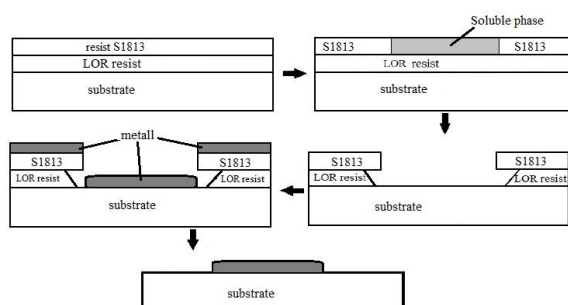


Figure 10. Stages of forming of a resistive mask under «lift-off» photolithography.

Before forming of the barrier contact, the surface was etched in order to determine the effect of introduced defects on the parameters of the Schottky contact. The Ni/Au system formed by lift-off photolithography was used as a barrier metallization. Etching was performed with the following parameters: the ratio of gas flows $\text{BCl}_3/\text{Cl}_2/\text{Ar} = 20/60/10 \text{ cm}^3/\text{min}$, pressure – 1.2 Pa, RF power – 20 W when changing the ICP power from 50 W to 400 W. The schematic illustration and appearance of the diode structure are shown in Figure 11.

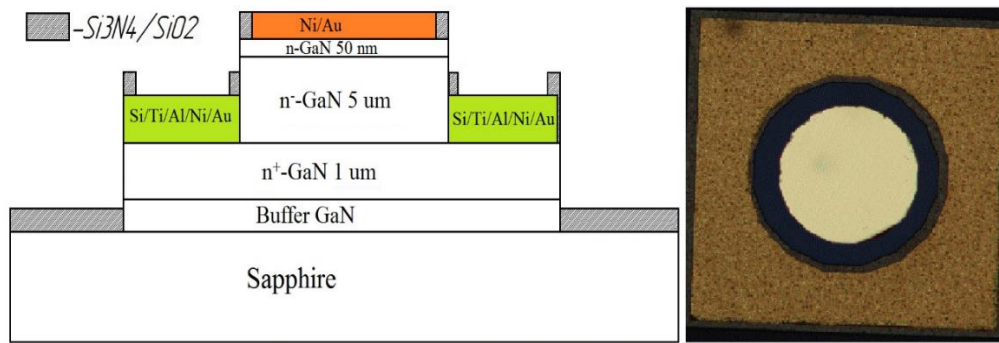


Figure 11. Schematic representation and appearance of the diode structure.

The quality of Schottky barriers was evaluated by the barrier height (ϕ_b) and the coefficient of non-ideality (n), determined from the current-voltage characteristics shown in Figure 12.

The voltage drop at the current density of 100 A/cm^2 and the reverse voltage at the reverse current level of 1 mA were estimated. The breakdown voltage of the diodes was more than 100 V.

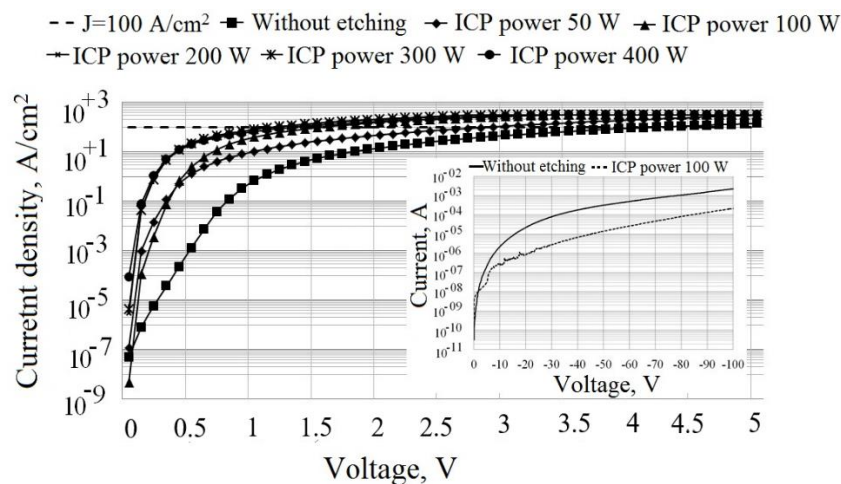


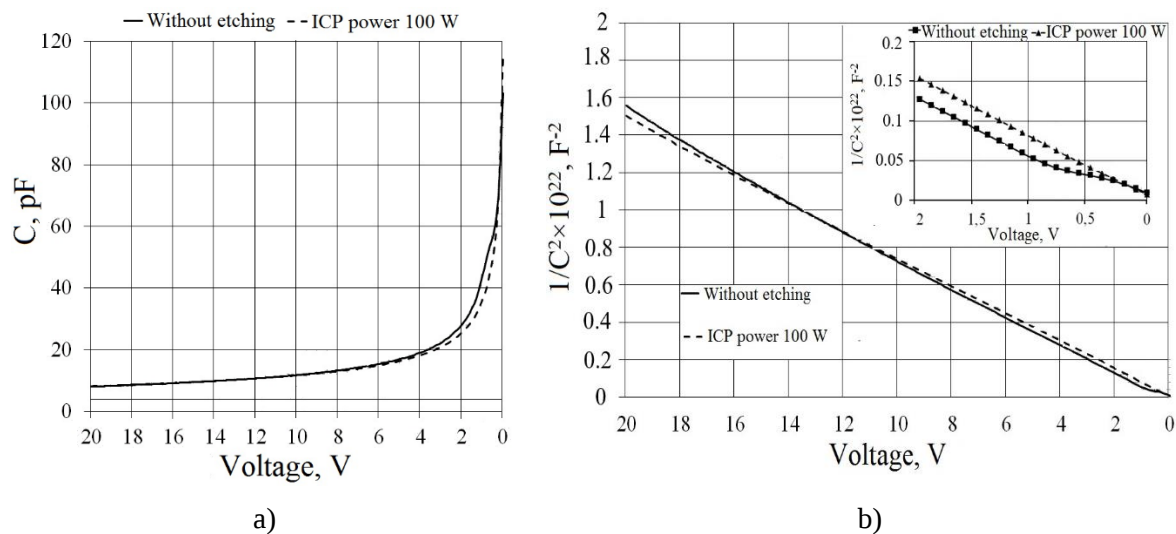
Figure 12. Direct I-V characteristics of diode structures. The inset presents the reverse I-V curves for structures without etching and after etching with ICP power of 100 W.

As follows from Figure 13, treatment in chlorine plasma based on $\text{Cl}_2/\text{BCl}_3/\text{Ar}$ allows improving the parameters of diode structures by removing protective layers and cleaning the surface.

The parameters of the obtained structures with the Schottky barrier are presented in Table 1. According to the capacitance-voltage characteristics (Figure 13), the profiles of charge carrier concentrations in gallium nitride layers were calculated, from which the doping level in the base region of the diode was determined – $7 \cdot 10^{15} \text{ cm}^{-3}$.

Table 1. Influence of etching modes on the parameters of the Schottky barrier.

ICP power, W	ϕ_b , eV	n	U_f , V $J_f=100 \text{ A/cm}^2$	U_{rev} , V $I_{rev}=1 \text{ mA}$
Without etching	0.68	2.0	4.25	78
50	0.82	1.2	2.95	92
100	0.87	1.0	1.55	> 100
200	0.75	1.1	1.18	95
300	0.64	1.5	1.15	80
400	0.62	1.5	1.35	92

**Figure 13.** Capacitance-voltage characteristics of the diode structures.

As follows from Table 1, the best results are obtained at a power of inductively coupled plasma of 100 W. At this, the barrier height is 0.87 eV, the coefficient of non-ideality is 1.0, and the reverse voltage is > 100 V.

5. Conclusion

Thus, in the course of experimental studies, etching modes of gallium nitride were established that allow etching the epitaxial layers of semiconductor to a depth of about seven microns to obtain a smooth surface and a tilt of the side walls of about 70° . Masks were obtained that protect the GaN surface from prolonged plasma chemical etching in the $\text{BCl}_3/\text{Cl}_2/\text{Ar}$ atmosphere. Using the obtained etching modes, instrument structures of Schottky diodes for various etching modes were fabricated. The mode of plasma chemical surface treatment before the formation of the Schottky barrier was determined.

As part of the work, the influence of treatment in chlorine-containing plasma on the parameters of the Schottky barrier was studied. It was shown that the best results are obtained at a power of inductively coupled plasma of 100 W. At this case, the barrier height is 0.87 eV, the coefficient of non-ideality is 1.0, the direct voltage drop over the current level of 100 A/cm^2 is 1.55 V, the breakdown voltage is more than 100 V, the serial resistance is $8.7 \text{ m}\Omega \times \text{cm}^2$.

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References

- [1] Balakirev A and Turkin A 2015 *Electronics: STB* **4** (00144) 64–8
- [2] Zhelannov A V et al 2014 Materials of the 5th International Scientific and Practical Conference on Physics and Technology of Nanoheterostructural Microwave Electronics MOKEROV READINGS (Moscow) pp 29–30
- [3] Bessolov V N 2012 *Technical Physics Letters* **38** (1) 21–6
- [4] Basak D 2000 *Solid-State Electronics* **44** 725–8
- [5] Zhelannov A V et al 2017 *Nano- and microsystem technology* **19** 399–405
- [6] Protasov D Yu 2014 *Tech. Phys.* **84**(9) 96–9