Methods for processing structures based on solid solutions of $\text{A}^{\text{III}}\text{B}^\text{V}$ compounds

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Abstract. The technologies of plasma-chemical etching of $\text{Al}_x\text{Ga}_{1-x}\text{N}$ epitaxial layers in chlorine-containing mixtures, which allow forming a microrelief on the surface of a semiconductor in the range from tens of nanometers to tens of micrometers, are presented. The modes of selective etching of GaN layers with respect to $\text{Al}_x\text{Ga}_{1-x}\text{N}$ ($x = 0.25$) are determined.

1. Introduction
When developing device structures based on the $\text{Al}_x\text{Ga}_{1-x}\text{N}$ solid solutions system, one of the key tasks is the development of a technology for local material removal. Such a technology is necessary for the formation of mesa-insulation of active regions of device structures, recesses for ohmic contacts [1] and for gate metallization.

Both chemical (“fluid”) and plasma (“dry”) etching methods can be used for etching of materials of the $\text{A}^{\text{III}}\text{B}^\text{V}$ group. "Liquid" methods of removing material involve the use of liquid solutions for chemical etching of masked areas of a semiconductor. These methods are simple to implement and do not require sophisticated technological equipment. But when working with AlGaN / GaN heterostructures, the “liquid” method did not become popular, because of the strong chemical bonding in nitride semiconductors, it is difficult to remove solutions that chemically etch the material. It was reported about photo-stimulated electrochemical etching of GaN, however, this method depends on many parameters and is poorly reproducible [2, 3].

A significant limitation of "liquid" etching methods for group III nitrides has caused considerable interest in the development of dry etching methods. Various methods of dry etching related to ion influence are used for the treatment of nitride semiconductors. Among them are ion etching, reactive ion etching, reactive ion etching using electron-cyclotron resonance and reactive ion etching in inductively coupled plasma, chemical ion-beam etching. As the most effective method of plasma etching, reactive ion etching with a source based on inductively coupled plasma can be distinguished, which allows to obtain a dense plasma with high homogeneity and controllability.

2. Experimental technique
The aim of this work is to obtain the technology of deep etching of gallium nitride using a mixture based on $\text{Cl}_2/\text{BCl}_3/\text{Ar}$ with rates of about 0.8–1.0 $\mu\text{m/min}$, and the technology of selective etching of GaN relative to AlGaN in a gas mixture based on $\text{Cl}_2/\text{Ar}/\text{O}_2$. 

To establish the modes of gallium nitride etching, experimental studies of reactive ion etching were performed on a Sentech SI-500 unit (figure 1) equipped with a source of inductively coupled plasma. The testing of etching processes involved the selection of modes that ensure sufficiently high etching rates (up to 1 μm/min) to remove the semiconductor to a depth of 10 μm. As test samples, structures with an undoped layer of gallium nitride and a thickness of ~2.0 μm, grown by chemical vapor deposition using organometallic compounds on a sapphire substrate 2 inches in diameter, were used.

When choosing masking coatings, it is necessary that they have chemical resistance to aggressive plasma gaseous media, have high adhesion to the substrate, and are easily removed after etching without changing the semiconductor surface morphology [4].

In our experiments, a combined mask consisting of nickel (Ni) with a thickness of 0.25 μm and a sublayer of silicon dioxide (SiO₂) with a thickness of 0.3 μm was used. The sequence of technological operations for the formation of the mask is presented in figure 2.

Mask formation begins with plasma-chemical deposition of a 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 with an inductively coupled plasma source power of 150 W, high-frequency power 5 W, pressure in the chamber 5 Pa and temperature 250°C. The nickel film was sputtered by the magnetron method with the substrate heated to 250°C. After that, using optical photolithography, a photoresist mask was formed (figure 2a), which consists of alternating open and closed areas of the surface. To preserve the anisotropic profile, the removal of nickel and silicon dioxide layers was carried out using dry etching methods, namely: ion etching for nickel and plasma-chemical etching for SiO₂ (figure 2c). After etching the SiO₂/Ni layers, the photoresist was removed in dimethylformamide (figure 2d).

In the course of experiments on the etching of gallium nitride, the dependences of the etching rate on the parameters of the plasma process were established, including pressure in the reactor, gas flow rate, power of the source of inductively coupled plasma, high frequency power.

3. Measurement results

3.1. Effect of pressure in the chamber on the etching parameters

The pressure in the chamber affects the basic processes that are responsible for plasma etching. The mean free path is inversely proportional to pressure, so that the potential of the shell increases the energy of the ions bombarding the sample. The etching rate increases with increasing ion bombardment and therefore lower pressure may contribute to etching.
Increasing the pressure in the chamber leads to a decrease in the free path of the ions and an increase in the probability of electron and ion collisions; therefore, the plasma density decreases (figure 3).

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

The etching rate of gallium nitride decreases monotonically with increasing pressure in the chamber from 0.6 Pa to 2.4 Pa, due to a decrease in the concentration of Cl₂ radicals in the plasma, or a decrease in the flux of ions coming to the surface of the substrate.

3.2. Effect of gas mixture flow on etching parameters

The correct choice of reactive gas or mixture of gases has a strong influence on the etching rate. However, the selection of the optimal gas environment is determined not only by the productivity of the process, but also by the achievement of high selectivity of etching. The etching rate increases rapidly with increasing feed rate or gas flow rate (figure 4).

It reaches a maximum, and then decreases with a further increase in gas consumption. Low etching rate at low gas consumption is determined by the insufficient number of chemically active particles formed in the discharge due to the lack of the original substance. The drop in rate at high flows can be explained by the fact that the active plasma particles do not have time to interact with the material being processed.

**Figure 4.** Dependence of etching rate of gallium nitride on the flow rate of the gas mixture components.

3.3. Effect of inductively coupled plasma source power on etching parameters

Figure 5 shows the change in the etching rate of gallium nitride with an increase in the power of an inductively coupled plasma source (ICP power). ICP power in the plasma process mainly regulates plasma density. As the plasma density increases with increasing ICP power, more reactive particles will be applied to the substrate, increasing the chemical aspect of etching. This is accompanied by the increase in etching rate. As can be seen in Figure 5, the etching rate of gallium nitride increases with ~15 nm/min to 545 nm/min with an increase in ICP power from 25 to 700 W at a high-frequency power value (RF power) of 20, 40 and 60 W.
Figure 5. Dependence of the rate of gallium nitride etching on ICP power

At the same time, as can be seen from Figure 6, for small values of high-frequency power (up to values of about 60 W), the etching rate is saturated when the ICP power of 600 W is reached. The saturation of the etching rate in this case can be caused either by saturation of the reactive particles on the surface, or by desorption of reactive substances on the surface before the occurrence of the reactions.

3.4. Effect of high-frequency power on etching parameters

The high-frequency power of the system increases the energy of the electrons and, therefore, increases the probability of ionization. As a result, the etching rate increases with increasing RF power (figure 7).

Figure 6. Dependence of etching rate of gallium nitride on ICP power

Figure 7 presents the dependence of the etching rate of gallium nitride on RF power, where it is shown that the etching rate monotonously increases with increasing RF power. The etching rate is increased by increasing the contribution of the physical component of etching in combination with a strong component of chemical etching. The increase in the etching rate can be explained by the im-
proved sputtering of the etching products, as well as by more efficient breaking of bonds in the semiconductor with an increase in the ion energy.

3.5. Selective etching of GaN / AlGaN for creating device structures
The etching modes discussed above do not provide for etching selectivity between gallium nitride and AlGaN solid solution. The development of such a process is necessary when using heterostructures with protective heavily doped layers during the formation of areas for barrier metallization [5]. The selectivity of etching of GaN/AlGaN heterostructures, as a rule, can be achieved by introducing oxygen into the gas mixture Cl₂/Ar. As soon as the AlGaN layer is exposed, the oxygen contained in the plasma reacts with aluminum to form an oxide barrier on the surface. This prevents further etching and also protects the surface from ion bombardment [6].

To implement this kind of process, structures with alternating GaN (80 nm)/Al₀.₂₅Ga₀.₇₅N (80 nm)/GaN (2.2 μm) layers were used.

The samples were etched in Cl₂/Ar medium with the addition of various amounts of oxygen (from 0 to 10 cm³/min) with an inductively coupled plasma source power of 75, 100 and 200 W and RF power 10, 30, 60 and 90 W. The chamber pressure is 1.2 Pa with a Cl₂/Ar flow of 60/10 cm³/min. Figures 8 and 9 show the dependences of the etching selectivity on the oxygen consumption.

As it can be seen from the plots, the addition of oxygen to the gas mixture reduces the etching rate of AlGaN. This is due to the oxidation of the AlGaN layer upon interaction with oxygen and the formation of a compound that is resistant to etching. In this case, the dependence of the etching selectivity on the oxygen consumption has a maximum at a flow rate of 5 cm³/min and is about 30:1. A further increase in oxygen consumption leads to a decrease in selectivity due to a decrease in the etching rate of gallium nitride. In this case, the magnitude of the selectivity is also affected by the magnitude of the high-frequency power. Since the RF power determines the physical etching mechanism, then, at a certain its value, Al₂O₃ compound will be also etched. Thus, there is a maximum in the dependence of the etching selectivity on high-frequency power, as shown in figure 10.
The influence of the power of an inductively coupled plasma source on the rate and selectivity of etching of GaN and AlGaN was also investigated (figure 11). Other parameters of the etching mode remained constant: RF power of 30 W, a Cl$_2$/Ar flow of 60/10 cm$^3$/min, a chamber pressure of 1.2 Pa. The ICP power value in this case was varied from 75 to 200 W with different oxygen flow (0 and 5 cm$^3$/min). As one can see from the plots, the dependence of the rate and selectivity of etching has a maximum at an ICP value of 100 W. Smaller power values do not provide the necessary etching rates due to sufficiently low plasma density. When the power value is greater than 100 W, the AlGaN etching rate increases due to the denser plasma and, accordingly, the selectivity decreases.

### 4. Conclusion

Thus, in the course of the work, the processing modes of materials of the A$^{	ext{III}}$B$^{	ext{V}}$ group have been developed, allowing to obtain a surface microrelief in the range of up to ten micrometers. The influence of the main parameters of the plasma process on the etching rate of a semiconductor is considered. The modes of selective etching of GaN with respect to Al$_x$Ga$_{1-x}$N ($x = 0.25$) were studied and the etching selectivity was obtained on the order of 30:1.

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