Development of Fluorine Plasma Treatment for AlGaN/GaN Device Isolation

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1 Abstract

The objective of this project was to develop a fluorine plasma treatment (FPT) process for isolating AlGaN/GaN heterostructure devices using the PlasmaTherm Oxide Etcher (PT-OX) in the Stanford Nanofabrication Facility. A design of experiments (DOE) was conducted to determine the most significant parameters to be studied in the FPT recipe. Several factors were taken into account for the DOE, including prior art in literature, PT-OX fluorine plasma uniformity characterization results, PT-OX capabilities, test results from bare AlGaN/GaN pieces, and FPT isolation mechanisms. The DOE resulted in a set of experiments where the plasma treatment time was chosen to be the variable (varied from 100 seconds to 300 seconds) while CF_4 flow rate, ICP power, bias power and chamber pressure were set to be 80 sccm, 1000 W, 10 W and 10 mTorr, respectively. Circular test structures were fabricated in this set of experiments to examine and compare the current-voltage (I-V) characteristics of FPT isolations with different treatment times, mesa etch isolations and no isolations. Unexpected current-voltage characteristics were observed including nonlinear I-V response, large current variations across the wafer, and lack of isolation from mesa etch. Troubleshooting was carried out using atomic force microscopy (AFM), Auger electron spectroscopy (AES), and experimenting with multiple rapid thermal anneals (RTA). These results indicate that the original AlGaN/GaN substrate was of low quality; however, the troubleshooting results are insufficient to draw definite conclusions. Future work will be done to analyze the quality of the commercial AlGaN/GaN heterostructure wafers, to calibrate the RTA process for the formation of ohmic contacts to AlGaN. Additionally, thermal storage tests were conducted on fluorine plasma treated bare AlGaN/GaN samples at 600^{o}C for 10 hours in air to study the behavior of fluorine in FPT isolation in high-temperature oxidizing environments. XPS depth profiles showed that after thermal storage significant fluorine diffusion had occurred, indicating the feasibility of this technology for high-temperature applications needs to be further studied.

2 Introduction

Gallium nitride (GaN) has been researched as a material platform for high-temperature electronics and sensors due to its superior thermal stability resulting from its wide bandgap and strong atomic bonds. The AlGaN/GaN heterostructure has been utilized to fabricate high electron mobility transistors (HEMTs) thanks to the high electron mobility and density in the two-dimensional electron gas (2DEG) in GaN close to the AlGaN/GaN interface. The 2DEG is formed due to a quantum-well in the energy band structure at AlGaN/GaN interface. The quantum well is formed due to the bandgap differences between AlGaN and GaN, the surface states of AlGaN, as well as the piezoelectric and spontaneous polarization charges of the two materials.

A critical step in fabricating AlGaN/GaN heterostructure based devices is to isolate individual devices from each other by removing 2DEG between devices. One commonly used method, which has been used in our group previously, is to remove AlGaN layer by chlorine based plasma etching resulting in an AlGaN mesa structure (mesa etch process). Another method that has been reported in literature is to use fluorine plasma to incorporate negatively charged fluorine ions into the AlGaN layer [1][2]. Based on simulation and experimental results, fluorine ions are believed to bend the conduction band of AlGaN upwards and to raise the conduction band of GaN above the Fermi level at the AlGaN/GaN interface, thus depleting the electrons in 2DEG [2]. And it was demonstrated that the fluorine plasma only etches AlGaN negligibly resulting in a planar isolation [2]. Compared to the three-dimensional mesa etch process which can impose problems on photolithography and metallization, the fluorine plasma treatment (FPT) method allows for planar integration of AlGaN/GaN devices, which promises higher density and higher uniformity of device integration [1].

The objectives of this project are to develop a fluorine plasma treatment (FPT) based isolation technique using one of the plasma etcher (the PlasmaTherm Oxide Etcher, PT-OX) in Stanford Nanofabrication Facility (SNF). The developed FPT isolation will be compared with the previously-developed mesa etch isolation using the Oxford III-V etcher in SNF. As our group is focused on developing GaN based sensors for high-temperature environments, the high-temperature characteristics of the developed FPT isolations will also be investigated. This paper will first go into the design of experiments and test sturctures fabricated for isolation. Then results will be discussed from I-V measurements, auger electron spectroscopy (AES), atomic force microscopy (AFM), and x-ray photoemission spectroscopy (XPS). The results will be debrief comparison isolation techniques, thermal effects on FPT, and on troubleshooting to explain unexpected results.

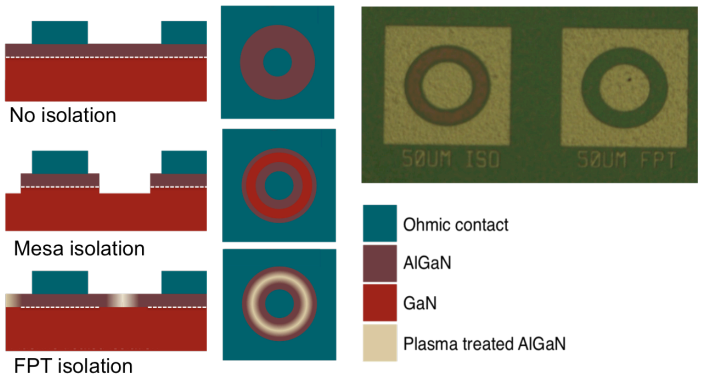


Figure 1: Schematic illustration cross section and optical image of top view of circular test structures with no isolation, mesa etch isolation, and FPT isolation.

3 Experimental Methodology

3.1 Test structure and microfabrication

Leakage current was chosen as the figure of merit to compare the various isolations mechanisms (no isolation, mesa etch isolation, and FPT isolation). Circular test structures were designed to compare the isolation mechanisms, as seen in Figure 1. Three test structures were used for comparing the two different isolation techniques, as well as no isolation. Each structure had an inner circle and an inner contact and outer contact and a 30 μm ring between the two contacts. The first contact had no isolation between the two devices and the other devices were isolated by a 10 μm inside the ring separating the two contacts. One of the isolated test structure was isolated via completely etching away the AlGaN layer removing 2DEG. The second isolated structure was isolated by applying the FPT between the contacts. The three test structures were microfabricated by first etching the mesa to develop the first type of isolation. The AlGaN was removed with BCl_3 (25 sccm) and Cl_2 (10 sccm) etch using the Oxford III-V inductively-coupled plasma (ICP) etcher (ICP power 250 W, forward power 80W, 10 mTorr, 75 seconds). Next, ohmic contacts were deposited via electron beam evaporation and lift-off of Ti (20 nm)/Al (100 nm)/Pt (40 nm)/Au (80 nm). A rapid thermal anneal was conducted at 850°C in nitrogen for 35 seconds to form ohmic contacts. Finally, FPT was done with PlasmaTherm Versaline LL-ICP Oxide etcher (PT-OX). The fabrication process of the three test structures is illustrated in Figure 2.

3.2 Design of experiments (DOE)

In this work, an ICP etcher was used with CF4 as the source for fluorine. The parameters involved in the process are 1) ICP power, 2) bias power, 3) CF_4 flow rate, 4) pressure inside the etch chamber, and 5) plasma treatment time. Given the constraints imposed by the limited time and the limited supply of substrates, experiments were carefully designed in order to expand the meaningful experimental space and to obtain useful results.

Several different factors were taken into account for the design of experiments, including 1) the prior art in literature, 2) the capabilities of PT-OX and processing considerations, 3) the results obtained from the PT-OX fluorine plasma characterization, 4) the bare AlGaN/GaN treatment results, and 5) the FPT isolation mechanism. Details on the five factors will be elaborated in the five following subsections, and a summary on design of experiments will be given at the end.

3.2.1 Prior art in literature

The FPT isolation process reported in the literature used a reactive ion etching (RIE) system with CF_4 as the source for fluorine [1-2]. The plasma power was 300 W and the treatment time was 100 seconds. The gas flow was 150 sccm and the plasma bias was set to 0 V. The bias was set to zero to

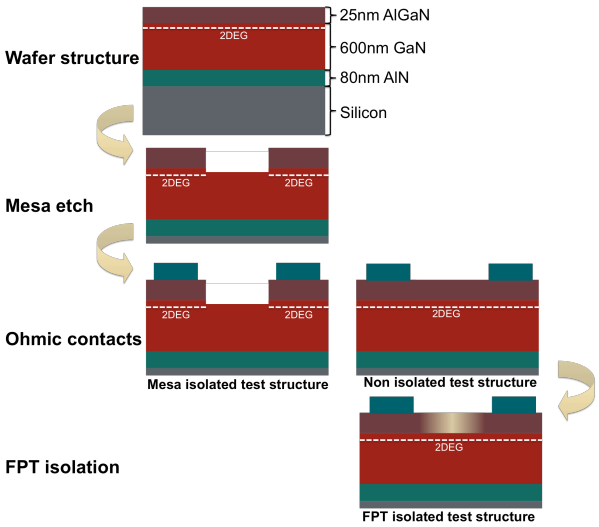


Figure 2: Schematic of isolation test structure fabrication process

minimize etching of AlGaN. ICP systems are different from RIE systems, but nonetheless the literature provided a starting point.

RIE fluorine plasma has also been used to partially deplete 2DEG to build enhancement-mode AlGaN/GaN HEMTs [2]. The plasma power and the treatment time were varied to investigate their effects in the threshold voltage of AlGaN/GaN HEMTs. It was revealed that the higher the plasma power and the longer treatment time, more shifts to positive voltage values were observed, which indicates more effective depletion of 2DEG. It indicates that the plasma power and the treatment time are two important variables for FPT processes.

3.2.2 PT-OX capabilities and processing considerations

In order to develop a low-variance recipe, the parameters (ICP power, bias power, flow rate, pressure) set on PT-OX should be within certain ranges. The extreme limits of PT-OX capabilities were avoided. Typical values in established recipes were considered. Pressure was chosen to be set at 10 mTorr (typical value) and the flow rate was chosen to set at 80 sccm (the maximum is 100 sccm).

For the bias power, a small value is desired to minimize etching of AlGaN on one hand; but on the other hand, we need to make sure the bias power is large enough so that the plasma would light and stay stable through the whole treatment. The bias power was chosen to be set at 10 W, which is a small value but has been proven to work for other PT-OX recipes. For ICP power, it should not be smaller than 400 W for repeatable processing in PT-OX but it should not exceed certain values where etching of AlGaN is no longer negligible. In addition, the plasma should be uniform across four-inch wafers.

3.2.3 PT-OX CF_4 plasma characterization

The fluorine plasma was characterized in terms of uniformity in thermal silicon dioxide etch rate across 4-inch wafers. The effects of ICP power and CF_4 flow rate on the uniformity were investigated. The ICP power was varied from 400 W to 1000 W and the CF_4 flow rate was varied from 20 sccm to 80 sccm. The chamber pressure was set at 10 mTorr and the bias power was set at 10 W.

It was found that different ICP powers yielded similar uniformity in oxide etch rate (Table 1), and

Table 1. oxide etch rate variance across 4-inch wafers by CF4 plasma of different ICP powers. The bias power, the CF4 flow rate and chamber pressure were set at 10 W, 80 sccm and 10 mTorr, respectively.

Wafer ID	ICP power (W)	Avg etch rate (A/min)	Max etch rate (A/min)	Min etch rate (A/min)	Etch rate variance* (%)
1	400	781	789	775	0.92%
2	700	1296	1310	1286	0.93%
3	1000	1596	1605	1579	0.82%
4	400	802	813	792	1.34%
5**	1000	1585	1646	1550	3.02%

*Etch rate variance = (Max etch rate – Min etch rate)/(2 X Avg etch rate). **Wafer 5 experienced huge reflective ICP power during 3 seconds of plasma lighting step (about 450W).

Table 2. Effects of flow rate on SiO2 etch rate. The bias power and chamber pressure were set at 10 W and 10 mTorr, respectively.

ICP power (W)	CF_4 flow rate (sccm)	etch rate (A/min)
400	80	931
400	20	883
1000	80	1874
1000	50	2054
1000	20	1891

that the flow rate does not exert a large effect on the oxide etch rate (Table 2). Therefore, we decided to fix the flow rate at 80 sccm in the subsequent experiments. In addition, the plasma uniformity is no longer a limitation on the choice of the ICP power.

3.2.4 Bare AlGaN/GaN FPT results

In order to gain more insights into fluorine incorporation behavior before conducting FPT on fabricated devices, some testing runs of FPT were carried out on bare AlGaN/GaN pieces and the treated samples were interrogated with X-ray photoelectron spectroscopy (XPS). In the testing runs, the CF_4 flow rate was set at 80 sccm, the bias power was at 10 W and the chamber pressure was set at 10 mTorr. The ICP power was varied from 400 W to 1000 W with a treatment time of 100 seconds. In addition, another test run was conducted where the ICP power was set at 1000 W but the treatment time was extended to 300 seconds.

XPS was used to investigate the surface concentrations and the depth profiles of fluorine in AlGaN. It was shown that the surface concentration is directly related to the treatment time while ICP power does not have significant influence on the surface concentration. When the treatment time was increased from 100 seconds to 300 seconds, the fluorine concentration at AlGaN surface was increased from about 4% to about 15%. However, the fluorine surface concentrations were all around 4% for ICP powers of 400 W, 700 W and 1000 W. On the other hand, the ICP power is related to the penetration depth of fluorine: the higher the ICP power, the deeper fluorine was incorporated. Therefore, a higher ICP power and a longer treatment time are simultaneously required in order to obtain more and deeper fluorine incorporation.

3.2.5 FPT isolation mechanism

As mentioned in the introduction section, fluorine ions are believed to bend the conduction band of AlGaN upwards and to raise the conduction band of GaN above the Fermi level at the AlGaN/GaN interface depleting 2DEG. Therefore, in order to obtain more effective depletion of 2DEG, a higher concentration of fluorine ions at deeper locations in AlGaN layer is required. Based on the bare AlGaN/GaN FPT testing results, a higher ICP power is required to drive fluorine deeper and a longer treatment time is required to increase the concentration of fluorine. Therefore, the ICP power was decided to be set at 1000 W while the treatment time would be varied from 100 seconds to 300 seconds.

3.2.6 Summary of design of experiments

The DOE flow is schematically shown in Figure 3. The literature provides us with a starting point, and indicates that the plasma power and the treatment time are two very important parameters. The bias power and the chamber pressure were chosen to be fixed parameters (10 W and 10 mTorr, respectively) after considering the PT-OX capabilities, the parameter values of some of the well-established recipes on PT-OX, and some processing considerations (e.g., minimizing etching of AlGaN, a reliable plasma that can be lighted). The CF_4 flow rate was set to be 80

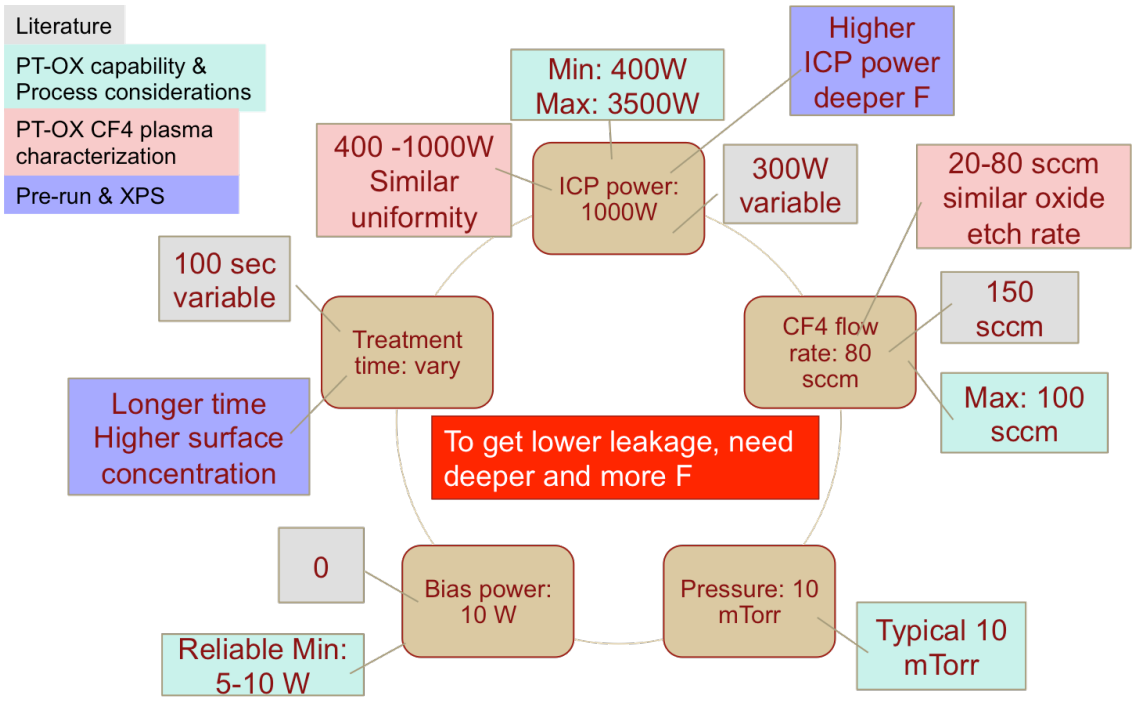


Figure 3: Schematic illustration of DOE to determine primary FPT variables

sccm with the results from characterization of CF4 plasma showing that the flow rate does not affect oxide etch rate, which indicates the plasma density is not affected by the flow rate. Different ICP powers showed similar plasma uniformity as well as similar concentration of fluorine on the AlGaN surface, but resulted in different penetration depth of fluorine into AlGaN. The plasma treatment time, on the other hand, determines the fluorine surface concentration. Taking into account of the FPT isolation mechanism, a more effective isolation would result from higher fluorine concentration and deeper fluorine incorporation. Therefore, the ICP power was chosen to be 1000 W while the treatment time was chosen to be the variable and was varied from 100 seconds to 300 seconds in our final experimental runs.

4 Results and discussion (C)

4.1 Initial results and device response to anneal time

After the mesa isolation etch and the ohmic contact deposition and RTA, the current-voltage responses was measured for the devices with no isolation. The I-V characteristics were expected to be linear due to the expected formation of ohmic contacts after RTA of Ti/Al/Pt/Au, which has been experimentally demonstrated by this group [3] and other researchers [4]. A circuit model to describe the expected response can be described as several resisters in series: the contact resistance between the AlGaN and metal, the 2DEG, and the contact resistance of the second contact. However, there was considerable variation across the wafer in terms of I-V linearity and the current levels from devices with the same geometry but in different locations on the wafer; the results are shown in Figure 4. The center of the wafer showed considerably higher current at the same voltage. Additionally, diode-like I-V characteristics were seen from devices on the outer dies, where the current would suddenly spike at high voltages; however the

devices on the center dies displayed more linear I-V characteristics. Some variations in the physical appearance of the metallization was seen, thus the RTA process was suspected to cause the nonlinear I-V curves and variations.

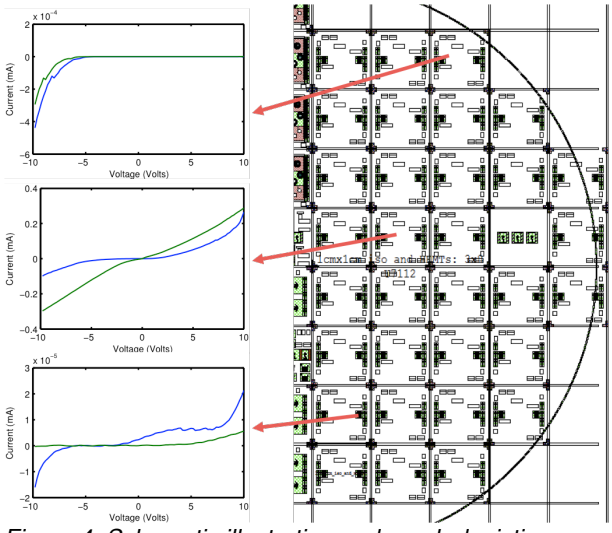


Figure 4: Schematic illustration and graph depicting nonuniformity of current voltage response across the wafer

In order to test the hypothesis that the RTA process was causing variation, the wafer was diced into dies and subsequent RTA processes were conducted on individual dies to experiment the effects of additional RTA on the I-V linearity and current level. Further annealing did increase the current and linearity of the devices as shown in Figure 5. The graph illustrates continued annealing led to increased current and linearity for two different non-isolated devices.

AES was used to investigate the depth profiles of atomic concentrations of different chemical elements of two samples: one that had only been annealed a single time for 35 seconds and the other that underwent 215 seconds of total anneal. Argon at 1 kV was used to sputter the sample surface away to provide atomic concentration with depth profile, as seen in Figure 6. The results of the atomic concentration depth profiling show that different anneal time resulted in different alloying of metals. In the sample that was annealed for a shorter period the Au concentration decreased sharply before it reaches the AlGaN surface. However, in the second sample which was annealed for a total of 215 seconds rather than just 35 seconds Au diffused further into the sample reaching the AlGaN surface. Due to the small thickness of AlGaN layer (about 25 nm), it is difficult to determine exactly where the AlGaN layer begins and ends. Due to the Al-containing metal alloy, it is hard to differentiate between the contribution of Al from the metal alloy and AlGaN layer.

4.2 Comparing Isolation Structures

After studying the effects of RTA time on I-V linearity and the current level, the different isolation mechanisms were compared. The results are seen in Figure 7. The graphs report the current response to voltages between -10 V and 10 V for non isolated devices, mesa etch isolated devices, and FPT isolated devices (before and after) FPT. The FPT recipe used was 80 sccm CF4, 1000 W ICP power, 10 W bias power, 10 mTorr pressure, and times of 100, 200, and 300 seconds. It should be noted that isolation (either mesa etch or FPT) did not appear to be effective; no consistent trend was observed among the test structures. It was hypothesized that fluorine was not incorporated

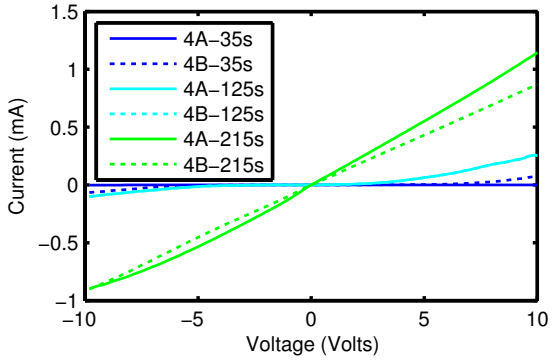


Figure 5: Graph of how increased anneal time effect current-voltage response of multiple devices on a die

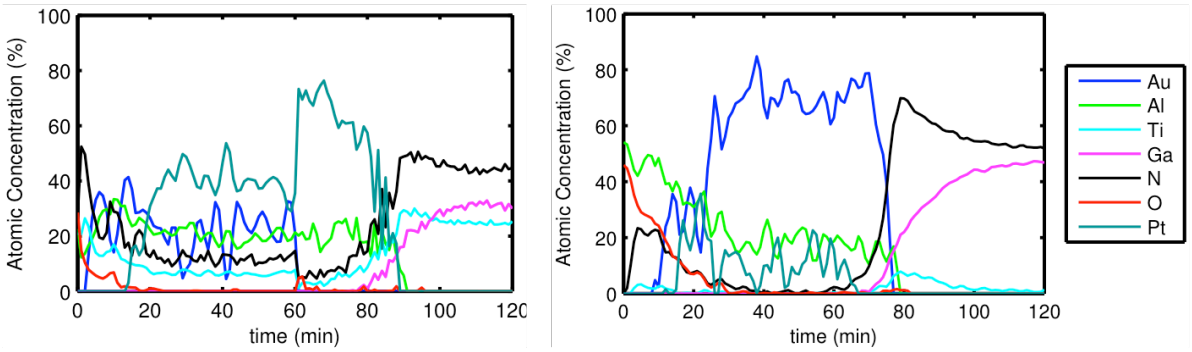


Figure 6: Graphs of atomic concentration with sputter time as surface is slowly removed via argon sputtering for single RTA (35 seconds) sample (left) and a sample with a total RTA time of 215 seconds (right).

deeply into AlGaN enough after FPT. Additionally, another hypothesis proposed to explain the lack of mesa isolation is that current was not conducted through 2DEG, either due to the absence of 2DEG or due to a more conductive path generated by the multiple RTA processes.

In an effort to test the first hypothesis that FPT did not result in fluorine incorporation that was deep enough, a 600C anneal in nitrogen of one of the samples that underwent a 300 second FPT was conducted. The sample was annealed in nitrogen for 10 minutes at 600°C, using the FGA furnace. The results for test structures of different widths of isolation are presented in Figure 8. The anneal caused little change or in some cases even increased the leakage currents FPT test structures, as opposed to a reduction of current.

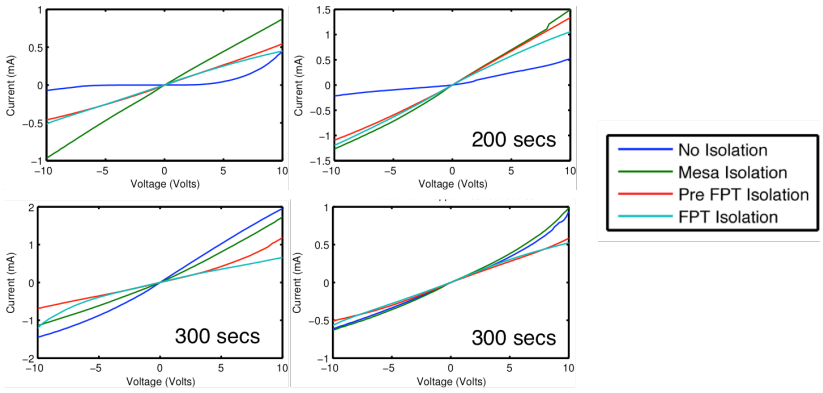


Figure 7: Comparing isolation techniques and device performance before and after FPT

The lack of difference between devices that were isolated via a mesa etch and devices that were not isolated was concerning because this group has previously shown that mesa etches are effective in AlGaN/GaN device isolation. Mesa etch isolation works on a simple principle that the AlGaN layer is etched away to remove the 2DEG, removing the conductive path at the interface of AlGaN and GaN. To verify that the mesa etch was deep enough, atomic force microscopy (AFM) was used to measure the step height on a mesa etch structure. The depth was shown to be approximately 80 nm, more than enough to remove the AlGaN layer (25 nm), as illustrated in Figure 9. The AFM illustrated the AlGaN was etched deep enough to be removed, and the nonisolated and isolated structures show no

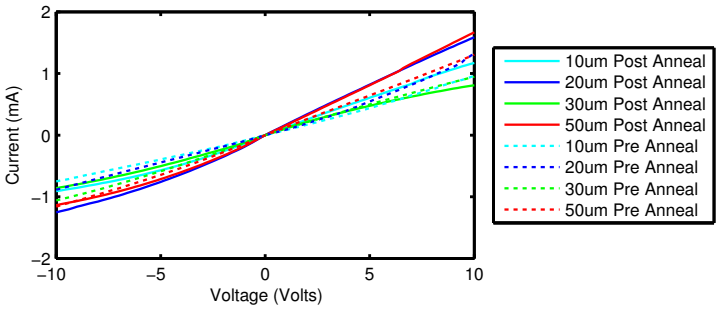
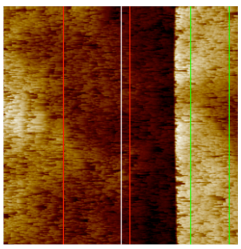
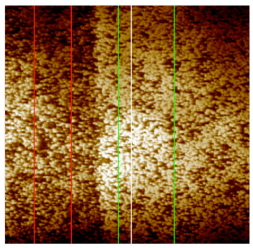


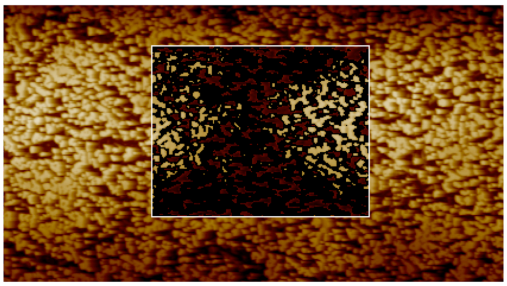
Figure 8: Comparing FPT performance before and after 10 minute, 600C anneal in nitrogen



Step height for mesa: 77 nm



Step height for FPT: 31 nm



Roughness Measurements: 14 nm

Figure 9: AFM measurements of mesa step height, FPT step height, and AlGaN wafer roughness

difference in current, therefore the electrons must be following similar conduction that is unaffected by isolation (mesa or FPT). This indicates there is either a conduction path more conductive than the 2DEG or the 2DEG is absent.

The roughness of the AlGaN surface and the GaN etched surface were measured with the AFM. Both measurements showed a roughness of about 14 nm, over 50% of the AlGaN thickness. This further brings the AlGaN/GaN substrate quality into concern.

The step height of the FPT was also measured and observed to be approximately 30 nm. This is also more than enough to remove the AlGaN. The FPT is not intended to be a physical removal of the AlGaN, rather a doping method to modify the band structure of the heterostructure. This step height is too large and future work will be done to modify FPT recipe to reduce removal of AlGaN. This might include reducing the ICP power, etch time, or increasing the pressure.

4.3 Thermal storage tests

A bare AlGaN/GaN-on-silicon piece was treated with CF_4 plasma in PT-OX (ICP power 1000 W, bias power 10 W, treatment time 300 seconds, CF_4 flow rate 80 sccm, pressure 10 mTorr) and then was diced into two halves. One half was subjected to 10 hours of thermal storage at 600^{o}C in air while the other half stayed as-is. XPS was used to characterize the fluorine concentration depth profiles in the two halves. The results demonstrated successful fluorine incorporation into AlGaN layer after the plasma treatment (Figure 10a). However, significant fluorine diffusion occurred during thermal storage resulting in undetectable fluorine concentrations (Figure 10b). This indicates the current FPT process may not be capable of providing stable and reliable isolation for AlGaN/GaN devices at 600^{o}C in air for extended periods of time. More studies need to be carried out to investigate the feasibility of FPT at elevated temperatures, and auxiliary

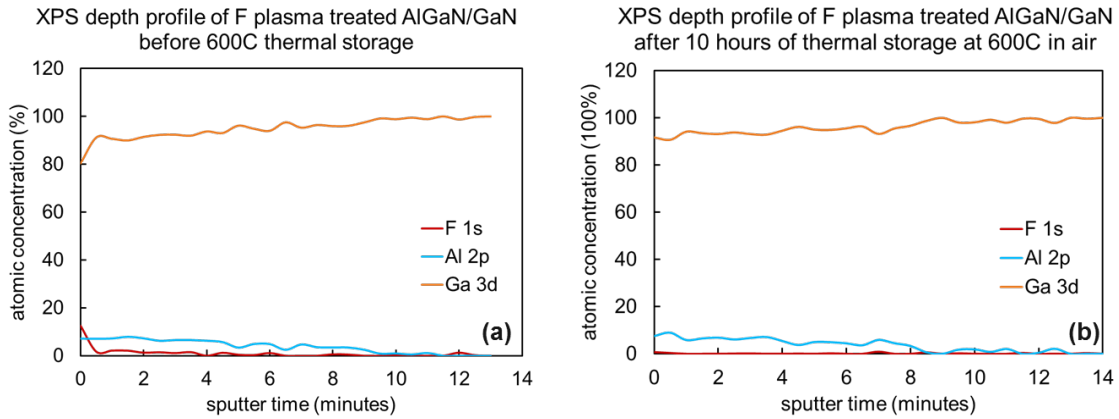


Figure 10: XPS depth profiles incorporated fluorine of FPT AlGaN/GaN samples befoere and after 10 hours of thermal storage at 600C in air.

processing (e.g., capping AlGaN with silicon nitride) can be researched to extend the FPT isolation capability.

5 Conclusions

In order to develop a FPT process to isolate AlGaN/GaN heterostructure devices and compare leakage current of mesa etch isolation and FPT isolation, circular test structures were microfabricated with mesa etch, contact metallization and FPT. A DOE was carried out to identify the primary recipe variables to be investigated among ICP power, treatment time, CF_4 flow rate, chamber pressure and bias power. FPT isolated devices were compared with no-isolation devices and mesa etch isolated devices. Unexpected results, including nonlinear I-V characteristics, large variations of I-V characteristics, and lack of mesa isolation led to extensive troubleshooting. The trouble shooting results have led to inconclusive understanding of the AlGaN/GaN wafer quality and the RTA process. Further investigation is needed. However, initial thermal storage test on bare AlGaN samples and XPS characterization show poor stability of the fluorine at elevated temperatures, as the fluorine was no longer detectable after 10 hours of heating at 600°C in air. This initial thermal storage testing result suggests that further study is required on the application of FPT for AlGaN/GaN high-temperature devices.

6 Acknowledgements

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