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**GaN/AIGaN DEVICES FOR
SPACE-BASED DEFENSE SYSTEMS**

FINAL REPORT
Contract F33615-99-C-5426
Project GWSML997200860
11 June 1999 - 11 December 1999

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Prepared for

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13. ABSTRACT (Maximum 200 words) Report developed under SBIR contract for topic BMDO99-005. The objective of this effort was to design and develop GaN MODFETs with minimal semiconductor volume to minimize radiation damage, particularly due to neutron radiation. High-quality GaN and AlN films were grown on Si and sapphire; MODFETs were fabricated on sapphire which exhibited a transconductance of ~ 150 mS/mm. We succeeded in selectively removing Si substrates using an HNO ₃ :HF solution; methods to remove sapphire substrates using laser ablation were investigated. After obtaining the GaN films, we developed a scheme for mounting nitride films on a high-thermal-conductivity substrate such as AlN. Electron-radiation studies with Schottky-barrier diodes fabricated on GaN layers were carried out and a baseline for defects established. MODFETs were also subjected to electron radiation. In an effort to develop efficient methods for etching, we used GaN samples in a 13.56-MHz RF parallel-plate reactor with well-characterized electrical parameters. Gas mixtures used were HBr/Ar, CH ₃ Br/Ar, and HBr/He. The optimum etch rate achieved was 73 nm/min using a 30% HBr/70% He gas mixture at 80-W RF power and at 150-mTorr pressure. The effort described lays the groundwork for manufacturing radiation-resistant transistors for commercial and military space applications.			
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1. CONTRIBUTION OF PROF. HADIS MORKOÇ

For the Phase I effort, we proposed to produce MODFETs with minimal semiconductor volume to minimize radiation damage--particularly damage due to neutron radiation, which cannot be alleviated except by reduced volume. Because of the ease with which Si can be removed from GaN layers, we opted to propose the use of a Si substrate for growth, followed by its removal and mounting of the wafer to an good dielectric substrate with good thermal conductivity. After submission of our proposal, experiments conducted in other laboratories succeeded in laser removal, in a selective fashion, of sapphire substrates. This paved the way for us to consider sapphire substrates as well, since GaN layers on sapphire exhibit better transport properties than those on Si.

We proceeded on two fronts to accomplish the above-mentioned goals--1) growth of nitride layers on Si and 2) fabrication and epitaxial removal and mounting.

Growth of Nitride Films on Si Substrates

In pursuit of the first goal, we developed a method for growing high-quality GaN on AlN buffer-coated Si substrates. Since the Si surface is reactive to nitrogen, a new approach was developed whereby a monolayer of Al was grown on a 3-in. Si substrate. The Al monolayer is deposited at very low temperature on a thermally cleaned Si surface. An alternative approach involves growing the Al monolayer at higher temperatures where excess Al naturally desorbs from the surface. Our experiments were concerned more with the former method, although both techniques were effective. The Al layer is

characterized by a change from the 7×7 [111] RHEED associated with Si to a 1×1 RHEED image associated with an Al layer with its characteristic rod spacing.

Following deposition of the Al monolayer, a high-temperature process was used during which additional layers of Al combined with ammonia were deposited on the surface to initiate the AlN growth process. AlN layers with smooth surfaces and very good luminescence characteristics were obtained. AlN layers with a thickness of nearly 0.5μ were sent to Prof. W. Jim Choyke (University of Pittsburgh) for analysis by variable-temperature cathodoluminescence (CL). The CL scans obtained on Si substrates are shown in Fig. 1; for comparison purposes, the CL scans of AlN layers of comparable thickness on sapphire substrates are shown in Fig. 2. Based on a comparative analysis with AlN supplied from other laboratories, it appears that the AlN layers thus grown are of relatively high quality. However, as is the case with all nitride semiconductor research and development, much room for improvement remains.

Following this, GaN was grown on $\sim 25 - 50$ -nm-thick AlN buffer layers. Optimization issues have been addressed concerning enhancement of the quality of the layers, and a C source has also been installed on the system to allow us to compensate the normally n-type films.

As in the case of AlN, we have made significant progress in attempts to grow high-quality GaN on Si substrates. Unlike the case of AlN, however, the GaN films grown on Si substrates are inferior to those grown on sapphire substrates. We are confident that device-quality layers can be grown, as shown recently in a presentation at the 1999

International Electron Devices Meeting. The advantages of Si such as the large area and low cost are overwhelming to the point where even a slight reduction in quality may not be construed as discouraging.

The goal is to grow low-doped GaN, compensated with an acceptor, for buffer layers of GaN/AlGaN modulation-doped heterostructures on Si substrates. A prudent approach would be to reduce the defect and impurity concentration as much as possible and then compensate the residual donors with a C acceptor. Even though C is deep and cannot be used successfully to create holes, its acceptor nature is sufficient to compensate the residual donors in GaN.

In summary, we have achieved high-quality AlN layers and have initiated the growth of GaN layers on AlN buffer layers on Si substrates. The experiments were performed with ammonia and RF nitrogen sources since the latter tends to result in the smoother surfaces that are preferred for MODFETs. While we have made substantial progress, additional effort is required on compensating the films. On a parallel front, we have grown high-quality MODFET layers on sapphire; the output characteristics are shown in Fig. 3. We used these devices for experiments on laser removal of sapphire substrates and on radiation experiments. Mechanical problems with our system, which have recently been solved, prevented us from making additional achievements during the course of this nine-month program.

Selective Removal of Si from Nitride Films

Wet-chemical selective etching of Si from GaN has been successful. We were able to remove Si at room temperature at a rate of 15 μ /min. Depending on its thickness, the entire substrate can be removed in 10-20 min. We have employed this technique to etch windows in Si down to GaN selectively.

In summary, Si substrates can be removed selectively and efficiently from nitride films in a HNO_3 :HF (1:2 by volume) solution, with an etching rate of $\sim 20 \mu\text{m}/\text{min}$.

Mounting Experiments

We have developed a method for removing the substrate from nitride films and replacing that substrate with a high-thermal-conductivity ceramic substrate such as AlN. The processing steps are shown in detail in Fig. 4 for the case of Si substrates. In short, the nitride films, either before or after fabrication into devices, are flip-chip bonded on a temporary template with wax. The experiments on Si have been successful. The substrate is then removed chemically in the case of Si and by laser evaporation in the case of sapphire. Next, a thin SiO_2 film is deposited via low-pressure plasma-enhanced CVD (PECVD) on the freshly exposed substrate side of the nitride buffer layer. A thin SiO_2 film is also deposited with low-pressure PECVD on the surface of a ceramic-grade AlN substrate. The SiO_2 layers on both sides are then fused together. A scanning-electron-microscope (SEM) image of the finished process is shown in Fig. 5. Next, the temporary template on the surface of the epitaxial layer is removed. The salient feature of this

approach is that a dielectric with high thermal conductivity is used as the substrate, which allows efficient heat removal while maintaining a low RF-loss medium. The thin SiO₂ layers are not expected to degrade the heat-conduction properties of the stack noticeably.

After submission of our proposal, a laser-evaporation method for selective removal of sapphire substrates was reported in the literature. The group at Walter Schottky Institute in Garching, Germany, successfully removed nearly 2-in. wafers. Prof. Hadis Morkoç visited that laboratory and made plans to collaborate with Drs. M. Kelly and O. Ambacher. While the technique was successful in lifting off thick GaN films (on the order of tens of microns) from sapphire, the residual strain present in the thin MODFET films caused them to crack. This technique should be effective if only a fabricated device chip were lifted. Because of time limits, we were unable to confirm this, but we feel confident that it can be accomplished. The group of Prof. T. Sands (University of California, Berkeley) was able to remove device chips using a similar technique.

In summary, we have developed a scheme for removing Si substrates from nitride films and for mounting the nitride films. This method can be utilized after device fabrication since this is a low-temperature process, with the maximum temperature being 400°C, on ceramic-grade high-thermal-conductivity insulating AlN substrates. The experiments on removing the sapphire substrates for this particular case have been stymied by residual strain and ensuing cracking. Removal of fabricated chips, where strain is expected to play a lesser role, should be successful. More experiments are needed in this area.

Radiation Studies

We have undertaken radiation experiments on p-n junction diodes and MODFETs in collaboration with Drs. Z. Fang and D. C. Look of Wright State University to ensure that the mechanisms will be in place when we have produced the final device structures on ceramic-grade AlN substrates. Electron irradiation--owing to its availability and the fact that it provides a window for defect-generation processes in semiconductors--has been chosen for this purpose. In addition, device layers on sapphire were chosen because we have not yet carried out experiments dealing with compensating the donors in GaN layers on Si. The MODFET devices on sapphire substrates were well characterized before electron irradiation; the output characteristics are shown in Fig. 3. To date, considerable analysis has been carried out on GaN films on sapphire substrates before and after electron irradiation. The devices exhibited characteristics similar to those obtained before our change to Virginia Commonwealth University. The output and transfer characteristics indicate a transconductance of ~ 150 mS/mm. Transconductances as high as 200 mS/mm have been observed in 1- μ m gate FETs. The drain breakdown voltage with small drain current is ~ 100 V.

Schottky-barrier diodes fabricated on GaN layers on sapphire substrates before and after radiation have been used for baseline studies to pave the way for similar studies on MODFETs. To facilitate the experiments, the layers were doped with Si to a level of $\sim 10^{17}$ cm⁻³. As mentioned earlier, similar experiments on MODFETs are underway. Deep centers in Si-doped n-GaN achieved using different ammonia flow rates (AFRs) have been studied by deep-level transient spectroscopy prior to electron-beam irradiation;

for the purpose of establishing a baseline. In samples investigated prior to electron irradiation, two new centers C_1 (0.43-0.49 eV) and E_1 (0.25 eV) were observed, in addition to five electron traps [A_1 (0.89 eV), A (0.67 eV), B (0.62 eV), C (0.45 eV), and D (0.24 eV)], which were also found in n-GaN layers grown by both metalorganic chemical-vapor deposition and hydride vapor-phase epitaxy. C_1 , whose parameters show strong electric-field effects and anomalous electron-capture kinetics, may be associated with dislocations. E_1 , which is very dependent on the AFR, exhibits an activation energy near that of a center created by electron irradiation and is thought to be a defect complex involving the nitrogen vacancy. Deep-level characterization of a set of samples after electron-beam irradiation is underway.

In summary, the baseline for the defects was established. While efforts are underway to reduce and/or eliminate those defects, similar experiments after electron-beam irradiation are in progress. These experiments will establish the nature of the defects and the resilience of the films and reduced-volume MODFET structures to defect generation under radiation.

2. CONTRIBUTION OF ISSI PERSONNEL IN COOPERATION WITH DR. J. SCOFIELD AND LT. J. STRICKER

In efforts to develop an efficient etching recipe for GaN and also to understand the interrelationship of the plasma characteristics and the resultant etching, we used a parallel-plate reactor and associated calibrated discharge-current and -voltage measurements. This allowed selection of the optimum operating parameters with regard to DC bias, phase angle, and discharge impedance. In many cases the gas pressure

selected for minimum phase angle, power-coupling efficiency, and minimum impedance at a reasonably high DC offset voltage also results in optimum etch rates. While these relationships are valid for fluorine-containing gas mixtures, they did not seem to be so clear-cut for the gas mixtures used here. Initially an HBr/Ar mixture was used; however, because difficulties arose with contamination of the gas flow meters and pump, we switched to CH₃Br in Ar. This mixture exhibited reasonable electrical-discharge characteristics; however, instead of etching being achieved, a film was deposited. The HBr/Ar plasma mixture was used again after alteration of the etching system to make it more compatible with the HBr gas. The DC voltage bias and power-coupling efficiency of the plasma were measured at pressures ranging from 150 to 1250 mTorr. These results are presented in Fig. 6 to illustrate that the 30% HBr plasma mixture, as compared to the other mixtures, has a low DC bias and a high power-coupling efficiency throughout the pressure range. Because of these characteristics, the 30% HBr plasma mixture was the used first in attempts to etch GaN

GaN samples were etched with the 30% HBr/Ar plasma mixture at 60 W and 150 mTorr and, when measured with the Dektak profilometer, appeared to have considerable surface roughness. The samples were analyzed via SIMS and Auger spectrometers before the mask was removed to determine whether the surface roughness was due to deposition. No clear evidence was found of a significant amount of foreign elements being deposited on the surface of the sample. Traces of C and F and large quantities of O were found; the deposition of O is unusual. We suspect that the sample may have been contaminated with oil from the roughing pump; the F may have been present in the oil from past experiments. The presence of these elements may also be the result of contamination after

the sample was removed from the etching chamber and exposed to the air in the lab. The sample was then deposited in a HCl solution to remove the Al mask. Data from the Dektak profilometer indicated that the GaN sample had much less surface roughness after deposition in HCl. The step height on the GaN sample was measured to be 695 nm, which indicates that some etching did occur--but not a significant amount.

The next step was to substitute He for Ar in the plasma mixture. The same power measurements were made at 60 W for the HBr/He plasma mixtures; the results are presented in Fig. 7. The DC bias values of the 30% HBr plasma mixture ranged from the lower values of the 10% HBr plasma mixture to the higher values of the 50% HBr plasma mixture throughout the pressure range. The power-coupling-efficiency values for the 30% HBr plasma mixture varied widely but tended to remain above the values for the 10% HBr plasma mixture and stay within the range of the values for the 50% HBr plasma mixture.

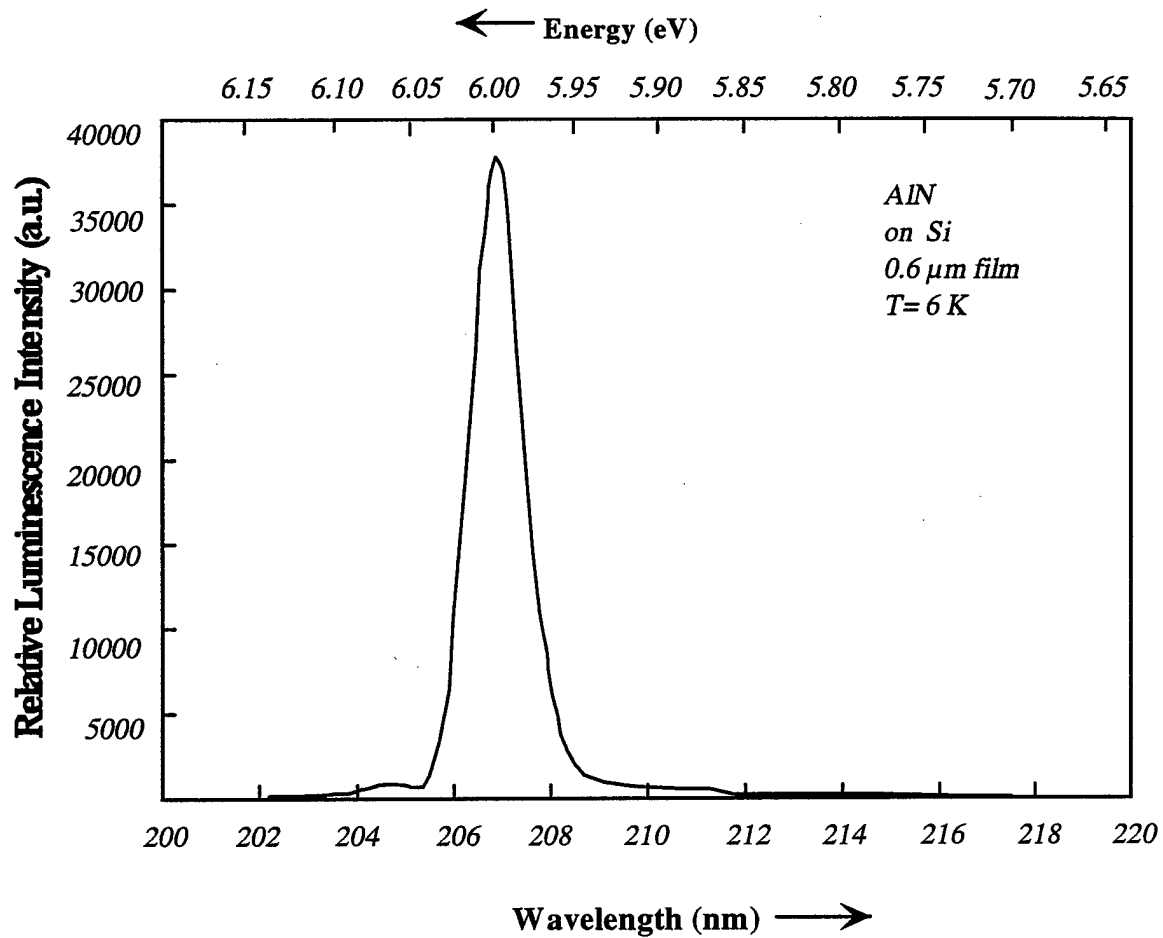
All three plasma mixtures were used at various pressures in attempts to etch GaN. Surface roughness was observed to be significantly less than that of the Ar etched samples when measured with the Dektak profilometer. The optimum etch rate of GaN was 73 nm/min and was achieved with the 30% HBr/70% He plasma mixture at settings of 80 W, 150 mTorr, and 20 min. A step on the GaN sample, displayed in the SEM images in Fig. 8, was measured with the Dektak profilometer to be 1460 nm. Further observation of the sample surface indicated substantial surface roughness, which may be due to ion bombardment.

GaN was first etched at 60 W and then at 80 W to determine whether increasing the power would affect the etch rate significantly. The etch rate increased from 67.25. to 73 nm/min, which is an 8.6% increase in etch rate with a 33.3% increase in power. Another characteristic observed was that the etch rate decreased with an increase in pressure. The etching of GaN was attempted at pressures of 150, 250, 350, and 500 mT.

A recently acquired corrosion-resistant pump will enable etching at pressures lower than 150 mT, and we can determine the increase in etch rate with lower pressures. The pump will also allow us to determine whether higher flow rates will improve the etch rate.

3. SUMMARY

In this effort, techniques were developed for growing high-quality GaN and AlN films on Si and sapphire substrates. Also, a MODFET was fabricated on a sapphire substrate exhibiting a transconductance of ~ 150 mS/mm. Schottky diodes and MODFETs were exposed to electron radiation, and the radiation effects were measured. Methods for removing the device structures from the substrates on which they were grown and mounting them on high-thermal-conductivity radiation-insensitive substrates were developed. Etch recipes were tested in a parallel-plate reactor, and a 30% HBr/70% He gas mixture yielded an etch rate of 73 nm/min. The work reported constitutes an important step toward the fabrication of radiation-resistant GaN and AlGaIn semiconductors and makes achievement of a high degree of radiation resistance feasible.



e⁻beam: 7 kV, 0.1 μA
Spectrometer blaze: 300 nm, 1200 lines/mm, slits: 250 μm
step : 0.2, integration time: 3 seconds

Figure 1. Cathodoluminescence scan of AlN on Si substrate obtained by Prof. W. J. Choyke (University of Pittsburgh).

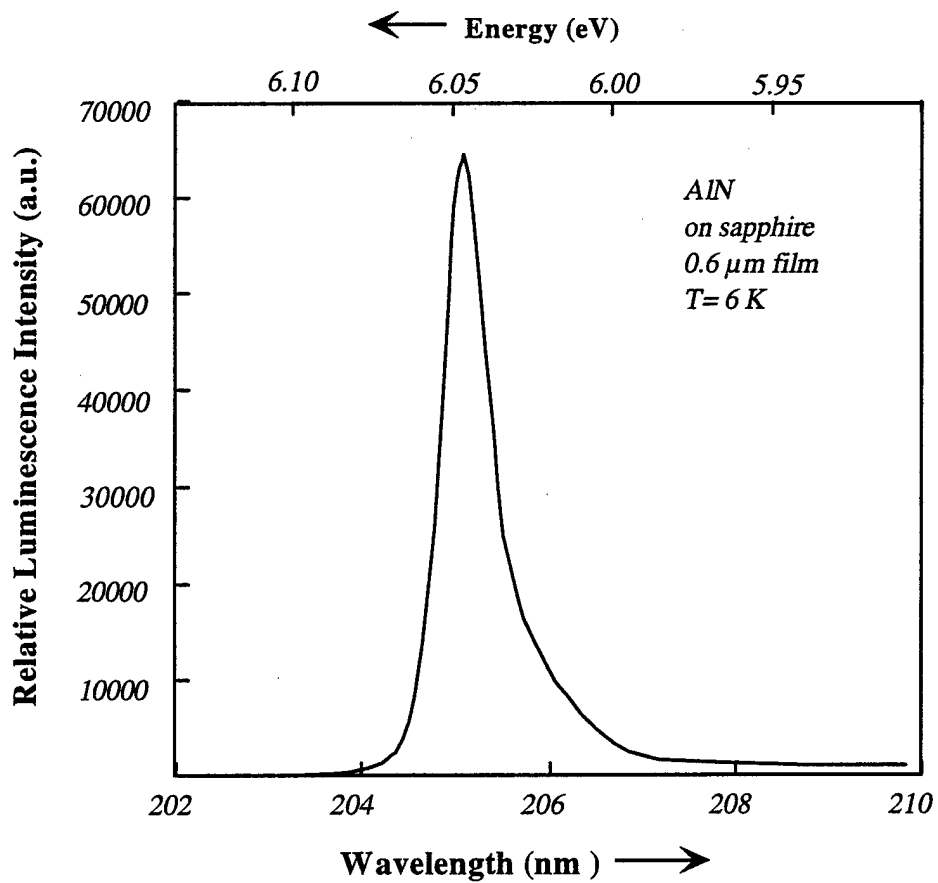


Figure 2. Cathodoluminescence scan of AlN on sapphire substrate obtained by Prof. W. J. Choyke (University of Pittsburgh).

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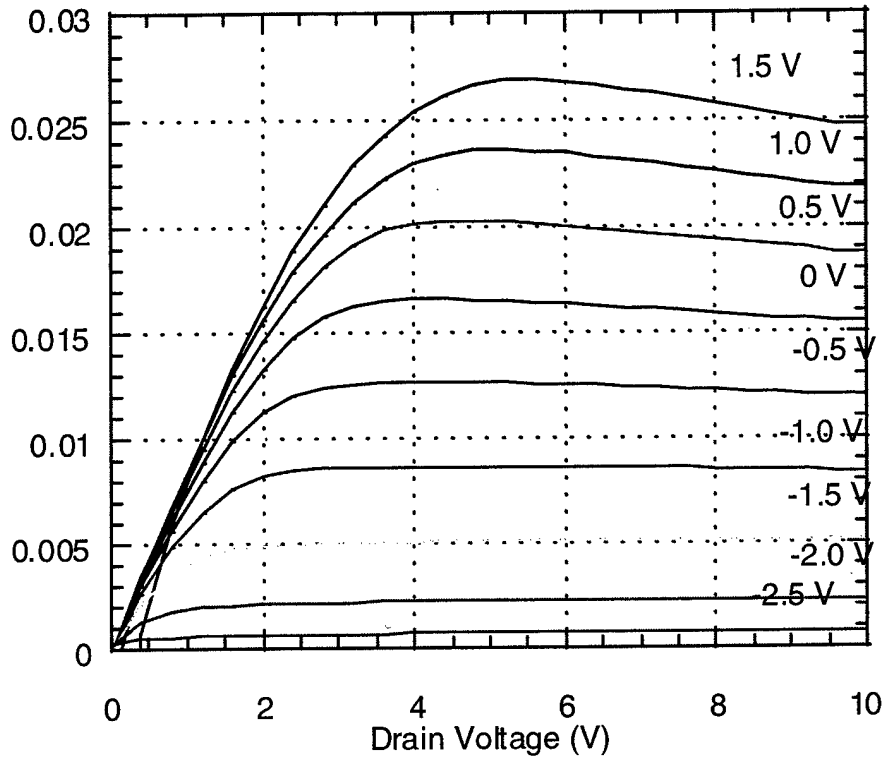
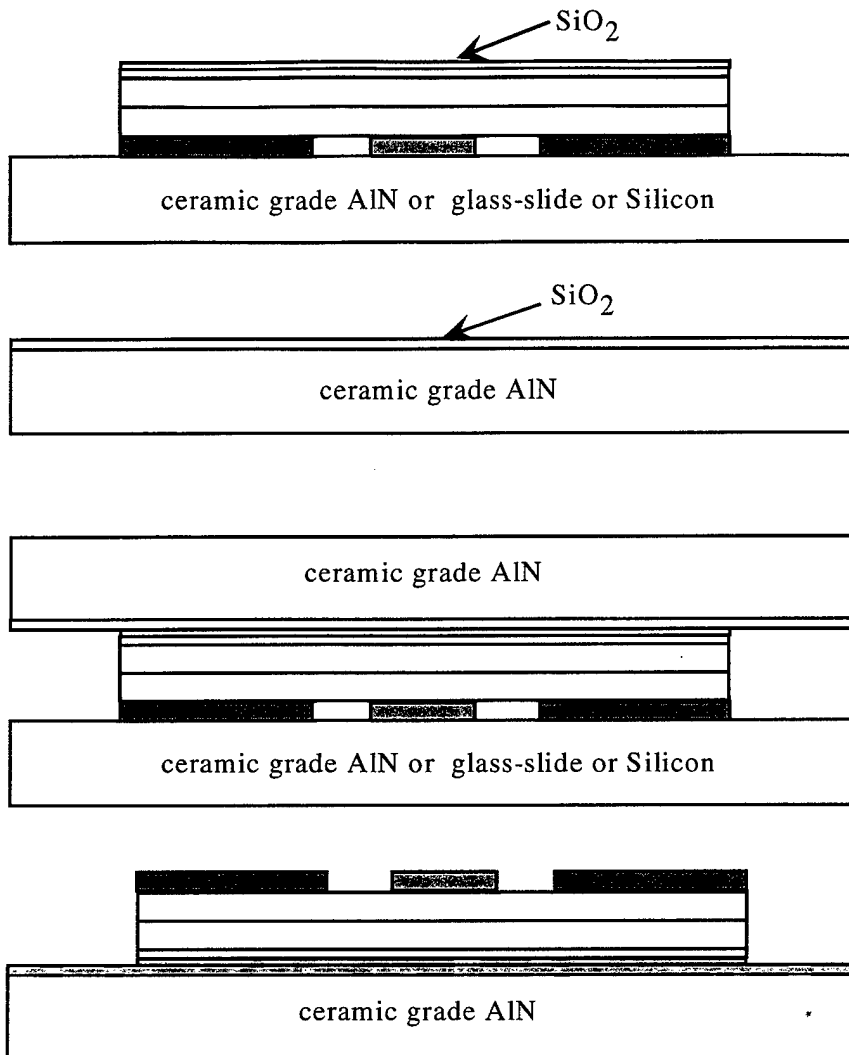


Figure 3. Output characteristics of 1- μm gate MODFET device on sapphire, with 40- μm gate width exhibiting transconductance of ~ 150 mS/mm.

Flip Chip mount for AlGaIn/GaN MODFET grown on Sapphire substrates- with minimal semiconductor body



Step.4. Deposit SiO₂ on the back side of the buffer layer by PECVD at 100 C, 30 min, 0.3 μm.

Step. 5. Deposit SiO₂ on ceramic grade AlN by PECVD at 250 C, 30 min, 0.3 μm.

Step6. Fuse the ceramic grade AlN on the device wafer as shown using SiO₂ as the bonding layer with mitigation from NaOH or KOH.

Step.7. Remove the waxed substrate which was for mechanical strength.

Figure. 4. Processing steps used to remove the Si substrate and mount the wafer--either before or after fabrication of the device--on a high-thermal-conductivity insulating AlN substrate.

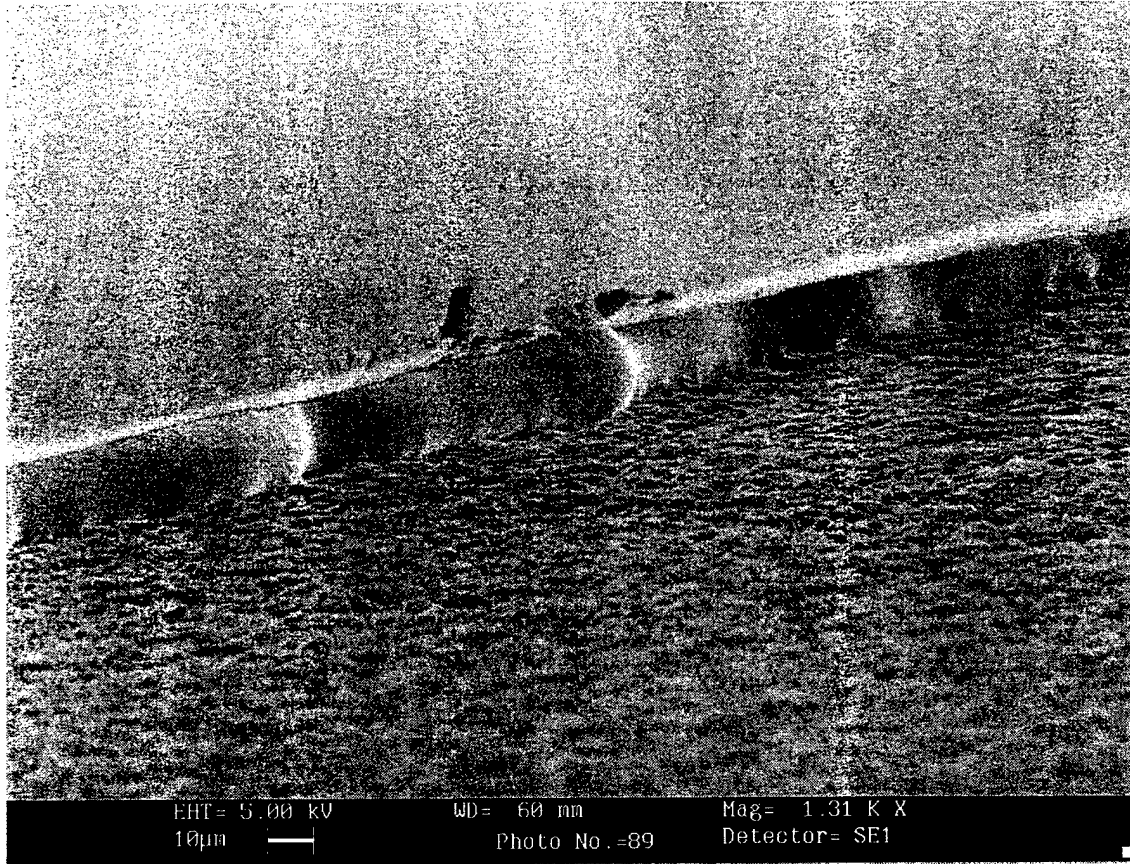


Figure 5. SEM image of GaN layer transferred from Si substrate onto ceramic-grade high-thermal-conductivity AlN substrate.

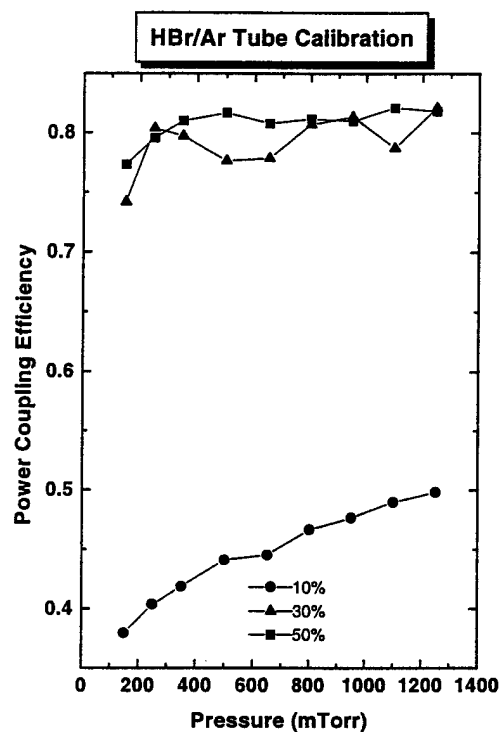
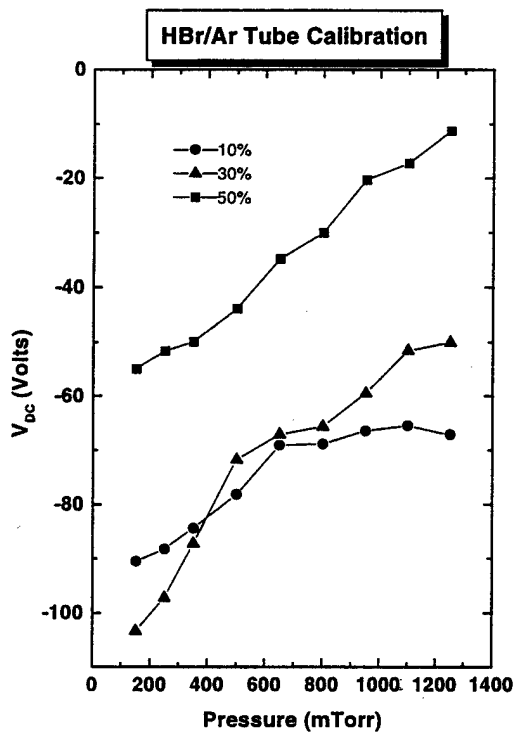


Figure 6. Plasma characteristics of HBr/Ar gas mixture.

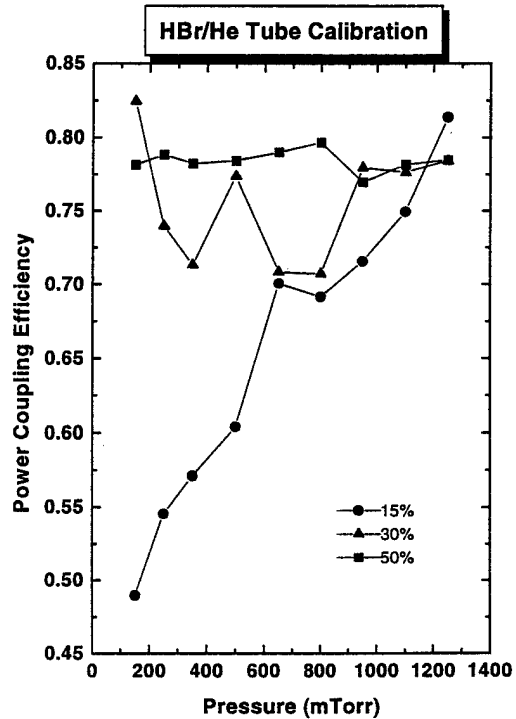
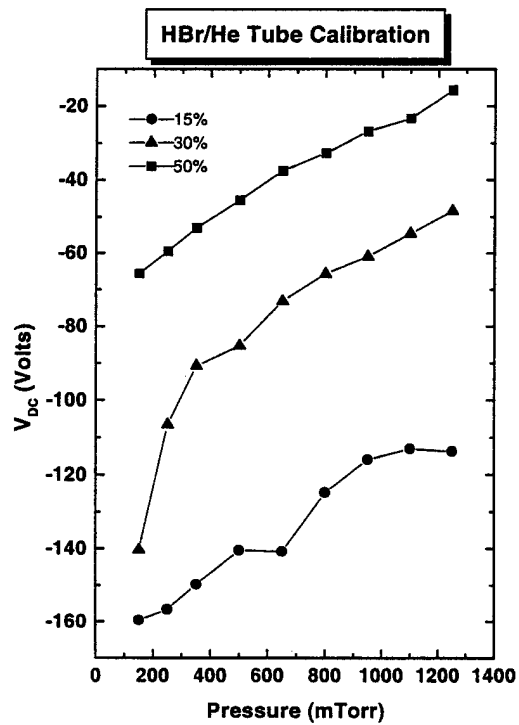


Figure 7. Plasma characteristics of HBr/He gas mixture.

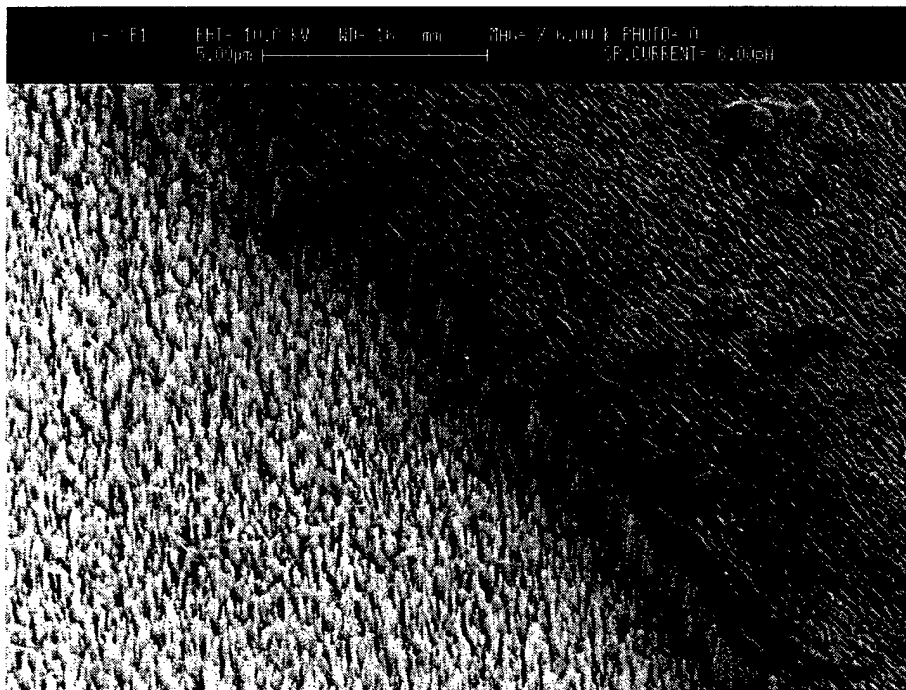
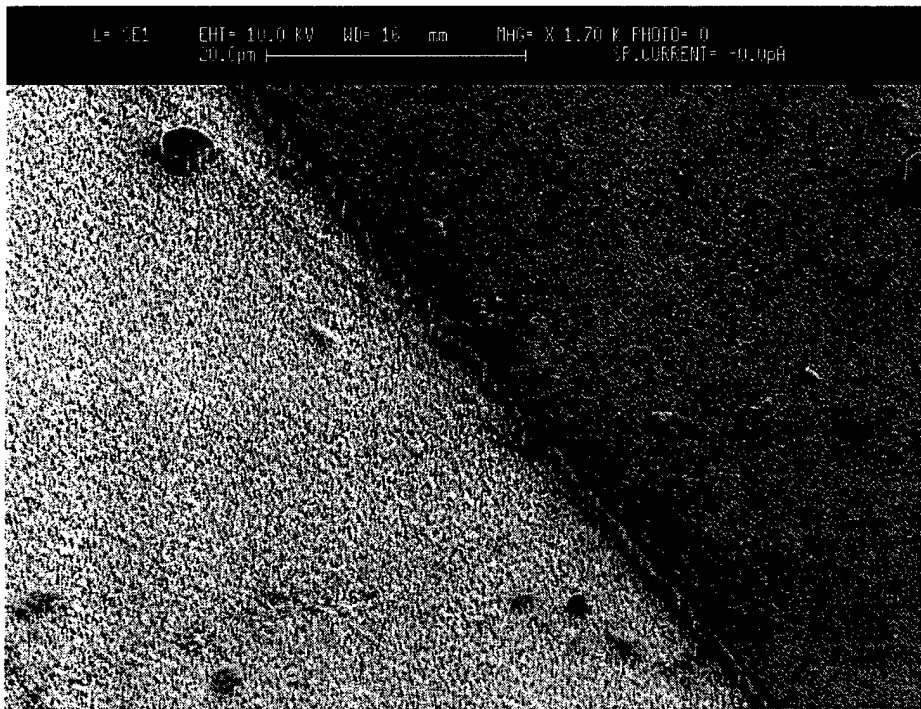


Figure 8. SEM image of etched GaN wafer.