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Neutron irradiation effects on metal-gallium nitride contacts

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We have measured the effect of fast and thermal neutrons on GaN Schottky barriers and ohmic contacts using current–voltage and transmission line method electrical techniques, optical, atomic force and scanning electron microscopy morphological techniques, and X-ray photoemission spectroscopy chemical techniques. These studies reveal a $10^{12} \text{n/cm}^2$ neutron threshold for Schottky barrier ideality factor increases, a $10^{15} \text{n/cm}^2$ fast plus thermal neutron threshold for ohmic contact sheet and contact resistance increases, and $10^{16} \text{n/cm}^2$ neutron fluence threshold for major device degradation identified with thermally driven diffusion of Ga and N into the metal contacts and surface phase changes. These results demonstrate the need for protecting metal-GaN contacts in device applications subject to neutron radiation. © 2014 AIP Publishing LLC. [http://dx.doi.org/10.1063/1.4869552]

I. INTRODUCTION

The interaction of neutrons with GaN is a subject of increasing interest for both radiation-tolerant electronics and for neutron detection devices. GaN is attractive for radiation-tolerant electronics because it can operate at high power and high temperature while exhibiting nearly the highest atom displacement energies of all compound semiconductors.1 Thus, while some measurements indicate comparable displacement energies for GaN vs., e.g., GaAs,2 other direct measurements show GaN displacement energies that are nearly twice those of GaAs that display an inverse relation since neutron emission is a signature unique to radioactive materials and one that is not easily shielded from detection.3,4 Because of its wide bandgap (3.4 eV at 300 K),1,15 GaN’s intrinsic carrier concentration at room temperature is very low, resulting in low off-state diode leakage current. Low leakage currents are important for operation at elevated temperatures, where such currents increase exponentially in semiconductors, as well as for detecting small electrical changes induced by neutron-detector interactions. Likewise, electron bombardment studies show that the threshold for radiation-induced displacement of the GaN lattice is high. Ga displacement in GaN requires 19 eV, and N displacement is minimal because the N sublattice self-heals efficiently.1,6

Neutron detectors have become vital for national security since neutron emission is a signature unique to radioactive materials and one that is not easily shielded from detection.5,6 The detection of neutrons are also involved in nuclear reactor near-core flux monitoring, nuclear accident rescuing and response, and fusion facilities.5 He, BF3, and 235U are currently used to detect neutrons but they are either in limited supply6 or are environmentally toxic. Hence, new solid state methods are needed for both slow and fast neutron detection, the latter using detectors wrapped in hydrogen-enriched materials to convert fast to slow neutrons and then into charged particles. Given its radiation tolerance, GaN-based electronic devices represent a promising alternative for such detectors. However, still unknown are the mechanisms by which neutrons interact with such devices and, in particular, with their metal contacts.

Besides extensive studies on the effect of high energy photons, protons, and electrons on GaN devices, there has been significantly less for neutron irradiation on these devices. These have focused primarily on neutron-induced deep trap spectra, electrical and luminescence changes within the GaN per se9–16 as well as neutron transmutation doping effects.17–19

Recently, we described the effect of fast (1 MeV) and thermal (25 meV) neutron irradiation on GaN Schottky diodes.20 That work focused on the defect densities and depth distributions produced by fast and thermal neutrons in epitaxial n-GaN after irradiation. Using a combination of depth resolved cathodoluminescence spectroscopy (DRCLS), surface photovoltage spectroscopy (SPS), transient (t-) SPS, and current-voltage (I-V) measurements, we showed that fast neutron fluences $\geq 10^{15} \text{n/cm}^2$ increased densities of deep level defects, specifically the well-known yellow band (YB) and blue band (BB) defects.6 In contrast, thermal neutron fluences of $<10^{15} \text{n/cm}^2$ actually reduced these defect densities and thereby improved the GaN crystalline quality. Nevertheless, fast plus thermal neutron fluences $\geq 10^{15} \text{n/cm}^2$ increased these defects and...
degraded I-V characteristics at the Schottky and ohmic contacts of the irradiated diodes. In order to understand this electrical degradation, it is important to separate the effects of neutrons on the GaN per se versus on the metal-GaN interface. To this end, we have used DRCLS, t-SPS, and I-V measurements to characterize how neutrons interact with GaN Schottky and ohmic contacts electrically and metallurgically for two metals, titanium, and nickel, after fast and thermal neutron irradiation. These measurements reveal that neutrons induce both electronic defects and metallurgical reactions at the metal-GaN interfaces.

II. EXPERIMENT

This study consists of two parts: (1) the changes in electrical properties of GaN Schottky diodes and ohmic contacts with neutron irradiation and (2) the metallurgical and morphological effects of neutron irradiation on specific metal-GaN Schottky and ohmic contacts. The Schottky diodes consisted of 42 μm² metal contacts on 2 μm n-type (Si doped ~3 × 10¹⁶ cm⁻²) epilayer GaN on sapphire in the layout shown in Fig. 1(a). The ohmic contacts (Fig. 1(b)) consisted of 20 nm Ti/150 nm Al/37.5 nm Ni/50 nm Au, followed by a rapid thermal anneal (RTA) at 850 °C for 30 s. In both cases, the GaN was grown by metal-organic chemical vapor deposition (MOCVD). The Schottky contacts (Fig. 1(c)) consisted of 30 nm Ni/400 nm Au. Both ohmic and Schottky contacts were deposited using electron beam lithography and deposition.

Irradiation experiments were performed with a pneumatic “rabbit” system which is designed to move samples rapidly in and out of close proximity to the reactor core for neutron fluence 10¹⁴ n/cm² (fast/thermal ratio ~4) and with in-core irradiation for neutron fluence 10¹⁶ n/cm² (fast/thermal ratio ~2) at The Ohio State University Research Reactor (OSURR). At both irradiation locations, the fast neutron energies peak at 0.73 MeV with an average energy of 1.98 MeV, which together are labelled “1 MeV.” Thermal neutron energies are less than 0.4 eV and peak at 0.0253 eV. Total neutron flux is 2.5 × 10¹¹ n/cm²-s, 1.25 × 10¹² n/cm²-s, and 1.8 × 10¹² n/cm²-s, respectively, for 10¹⁴ n/cm², 10¹⁵ n/cm², and 10¹⁶ n/cm² fluences. Specimens were at elevated temperatures only during the fluence times, i.e., several hundred to several thousand seconds. At the core proximity position, sample temperatures were measured to be ≤80 °C at “rabbit” position versus ≤150 °C at in-core irradiation position. We used a Four-Position Temp-Plate (Wahl Inc.) to measure the near-core temperature. This temperature indicator was calibrated to 1% NIST-traceable accuracy from 27 to 593 °C. Heat-sensitive indicator “positions” turn permanently black when the rated temperature thresholds are reached or exceeded. These time and temperature values suggest that effects of annealing after removal from irradiation are small for these studies. We irradiated the GaN contacts on six different dies using the fluences and the fast versus thermal neutron ratios listed in Table I. To distinguish the effects of thermal versus fast neutrons, we placed our samples in a cadmium capsule in order to filter out thermal neutrons and thereby irradiate with fast-only neutrons.

We used temperature-dependent I-V and transmission line method (TLM) measurements to extract electrical parameters including dominant transport mechanisms, ideality factor n, Schottky barrier height φᵣ₀, sheet resistance Rₛ, and contact resistance Rᶜ. For I-V and transmission line measurements of Rₛ and Rᶜ, we used a Keithley 2400 signal generator and two probes from a four point probe system. I-V measurements acquired using Labview software ranged from −6 V to 3 V whereas TLM measurements ranged from −3 V to 3 V.

<p>| TABLE I. Fast and thermal neutron irradiation fluences received by GaN ohmic and Schottky diodes. |</p>
<table>
<thead>
<tr>
<th>Sample (Die) identification</th>
<th>Fast neutrons ( \text{n/cm}^2 )</th>
<th>Thermal neutrons ( \text{n/cm}^2 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>( 10^{14} )</td>
<td>( 4 \times 10^{14} )</td>
</tr>
<tr>
<td>B</td>
<td>( 10^{15} )</td>
<td>( 4 \times 10^{15} )</td>
</tr>
<tr>
<td>C</td>
<td>( 1.4 \times 10^{16} )</td>
<td>( 2.6 \times 10^{16} )</td>
</tr>
<tr>
<td>F</td>
<td>( 10^{14} )</td>
<td>...</td>
</tr>
<tr>
<td>G</td>
<td>( 10^{15} )</td>
<td>...</td>
</tr>
<tr>
<td>H</td>
<td>( 1.4 \times 10^{16} )</td>
<td>...</td>
</tr>
<tr>
<td>K (reference)</td>
<td>...</td>
<td>...</td>
</tr>
</tbody>
</table>

FIG. 1. (a) Top view of ‘plus sign’ Ni/Au Schottky diode surrounded by ohmic contact and separated by a bare GaN border. Metal stacks used for the (b) ohmic and (c) Schottky contacts.
The second set of experiments consisted of monitoring the metallurgical and morphological effects of high neutron fluence irradiation on Ti/GaN and Ni/GaN contacts. Ti and Ni are the metals in intimate contact with GaN at the ohmic and Schottky contacts, respectively, so that any chemical interactions induced by neutrons at these junctions will have the strongest effect on electrical properties of those contacts. GaN grown at Kynew by hydride vapor phase epitaxy (HVPE) consisted of 5 μm epitaxial c-plane GaN layers (resistivity < 0.5 Ohm-cm) on 100 nm sapphire substrates. These n-type GaN wafers were doped with Si at ~10^{17} Si/cm^3. We then deposited 40 nm of Ni on two samples and 40 nm Ti on another two. The metal was deposited at room temperature via electron beam evaporation at The Ohio State ENCOMM NanoSystems Laboratory (ENSL) using a Kurt J. Lesker Lab-18 magnetron sputtering system. Metal layers were grown at ~2 nm per minute. Subsequent neutron irradiation at OSURR was performed using 10^{16} fast neutrons (2 samples) and 3.3 x 10^{15} fast + 6.6 x 10^{15} thermal neutrons (2 samples).

We used x-ray photoemission spectroscopy (XPS) to measure chemical changes of the irradiated metal-GaN contacts. We used a PHI 5000 Versaprobe with an Al Kα radiation source (1486.6 eV) for the XPS measurements, which were conducted in UHV at 2 x 10^{-7} Pa (1.5 x 10^{-9} Torr). High resolution core level spectra employed 0.05 eV, 200 ms energy steps. Atomic force microscopy (AFM) (Park XE-70) measurements provided morphological changes of the irradiated Ti- and Ni-GaN contacts.

III. ELECTRICAL RESULTS

A. Schottky contacts-ideality factor and barrier height

Figure 2(a) represents the log(I)-V results for our reference and irradiated diodes over the applied voltage V_a range, −5 V < V_a < 3 V. The most pronounced changes in the I-V curves were for 1.4 x 10^{16} fast n/cm^2 (blue) and 1.4 x 10^{16} fast + 2.6 x 10^{16} thermal n/cm^2 (yellow). In both cases the current dropped significantly, the I-V shape was no longer rectifying, and there was a shift in the curves (the current minimum shifted away from V_a = 0.) All I-V characteristics were obtained in darkness so the shifts were not photoinduced. Based on these shifts, 1.4 x 10^{16} n/cm^2 fast fluence irradiation (blue) induced negative charge trapping, whereas 1.4 x 10^{16} fast + 2.6 x 10^{16} n/cm^2 fast plus thermal (yellow) irradiation induced positive charge. These orders-of-magnitude I-V current and bias changes represent additional capacitances produced by formation of insulating interface layers and/or metal detachment. They represent two types of interface interactions and substantial contact degradation to be discussed in Secs. IV–VII. Contact degradation resulting from these 10^{16} n/cm^2 fluences precluded measurements of n, φ_{SB0}, R_S, and R_C.

We used the lower forward voltage bias region displayed in Figure 2(b), 0.05 V < V < 0.15 V to extract n and φ_{SB0} values for the Schottky contacts. The slope change evident for higher voltage is also present for the reference sample and indicates another current path and possible inhomogeneity at the intimate metal-GaN interface rather than effects due to irradiation. Current within the lower voltage range was low enough for IR_S (voltage loss due to internal sheet resistance) to be negligible but higher than the 2kT/q threshold for thermionic emission to occur. Based on Eq. (1),

$$\ln(I) = \ln\left[A^*T^2 \exp\left(-\frac{q\phi_{SB}}{k_BT}\right) + \left(\frac{qV_a}{nk_BT}\right)\right],$$

with diode area A, the slope of ln(I) in this range can be used to calculate n and its extrapolation to V_a = 0 V can be used to find φ_{SB}. In Eq. (1), we used A^* = 26 A/cm^2 K^2 for GaN and room temperature T = 300 K. Table II shows the resulting n and φ_{SB} before and after neutron irradiation. The φ_{SB} values contrast with a neutron irradiated E_c−1.0 eV value reported previously possibly due to differences in interface formation treatments and their effect on barrier heights.

While only I-V measurements of Schottky barrier heights were made, temperature-dependent I-V studies revealed that thermionic emission was the dominant transport mechanism between 200 and 300 K, extending down to 100 K for low fluence (1 x 10^{15} n/cm^2 fast and 5 x 10^{14} n/cm^2 fast + thermal) neutron irradiation. Furthermore, these results suggest

![Image](image_url)

FIG. 2. The log(|current|) vs. voltage characteristics for the neutron-irradiated GaN Schottky diodes for voltages (a) −5 V to 3 V and (b) 0.01 V to 0.15 V.
TABLE II. Ideality factor and Schottky barrier height versus neutron fluence on GaN Schottky diodes.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Fast neutron fluence (n/cm²)</th>
<th>Thermal neutron fluence (n/cm²)</th>
<th>Ideality factor, n</th>
<th>Schottky barrier height, φ_{SB0} (V)</th>
</tr>
</thead>
<tbody>
<tr>
<td>K</td>
<td>...</td>
<td>...</td>
<td>1.07</td>
<td>0.607</td>
</tr>
<tr>
<td>A</td>
<td>10^{14}</td>
<td>4 x 10^{14}</td>
<td>1.05</td>
<td>0.610</td>
</tr>
<tr>
<td>B</td>
<td>10^{15}</td>
<td>4 x 10^{15}</td>
<td>1.36</td>
<td>0.609</td>
</tr>
<tr>
<td>F</td>
<td>10^{14}</td>
<td>...</td>
<td>1.04</td>
<td>0.615</td>
</tr>
<tr>
<td>G</td>
<td>10^{15}</td>
<td>...</td>
<td>1.15</td>
<td>0.612</td>
</tr>
</tbody>
</table>

IV. ELECTRICAL ANALYSIS

A. Schottky contacts

Using Eq. (1), we obtained $\phi_{SB0} = 0.607$ eV and $n = 1.07$ for the reference Schottky diode without irradiation (K). For all fluences $\leq 10^{15}$/cm², $\phi_{SB0}$ remained nearly the same to within 0.008 V. However, neutron fluence had a significant effect on $n$. For an ideal Schottky barrier with only thermionic emission, $n = 1$. If defects are present, other mechanisms can occur: nonradiative recombination near the interface,\(^8\) tunneling through the Schottky barrier,\(^9,11\) and formation of intermediate layers with new dielectric properties. These processes raise $n$ and reduce current.\(^9,34\) From Table II, at neutron fluences of $10^{15}$/cm² (F), $n$ slightly decreased to 1.04 in comparison to the non-irradiated reference diode. This decrease is an indication of improved crystal quality and hence reduced defects.\(^5\) However, $10^{15}$ n/cm² fast neutrons (G) produced a significant increase in $n$, and the addition of thermal neutrons (B) increased it further. In the fast only case, $n$ increased by 0.08 in comparison to the reference whereas $n$ increased by 0.28 for the fast + thermal case (B). A 0.08 ideality factor increase suggests that when fast neutron fluence exceeds $10^{15}$/cm², the positive benefits of GaN re-crystallization are outweighed by new neutron induced defects. These new defects are likely caused by elastic scattering of metal and GaN atoms with incident high-energy fast neutrons. For example, knocking a metal atom into GaN may lead to the formation of a deep-level trapping site. A 0.28 increase in ideality factor signifies that thermal neutrons interact with metal/GaN differently than fast neutrons and are much more detrimental to device operation. Thermal neutrons have very low energy, suggesting less damage due to elastic defects but more damage caused by heat, either directly or indirectly via neutron/atom fission reactions and fission byproducts. Overall, the changes in ideality factor suggest that neutron fluence should be $<10^{15}$/cm², especially if thermal neutrons are present, in order to avoid degrading the GaN Schottky contact.

B. Ohmic contacts

Neutron irradiation effects on the ohmic contacts are consistent with those described in Sec. IV A. Similar to the data points, we were able to derive $R_C$ and $R_S$ for the reference as well as for our neutron irradiated samples. The calculated results for $R_C$ and $R_S$ appear in Table III.

TABLE III. $R_S$ and $R_C$, calculated values versus neutron irradiation fluence using the transmission line method on GaN ohmic contacts.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Fast neutron fluence (n/cm²)</th>
<th>Thermal neutron fluence (n/cm²)</th>
<th>Sheet resistance $R_S$ (Ω)</th>
<th>Contact resistance $R_C$ (Ω)</th>
</tr>
</thead>
<tbody>
<tr>
<td>K</td>
<td>...</td>
<td>...</td>
<td>2050</td>
<td>57.7</td>
</tr>
<tr>
<td>A</td>
<td>$10^{14}$</td>
<td>$4 \times 10^{14}$</td>
<td>2376</td>
<td>70.82</td>
</tr>
<tr>
<td>B</td>
<td>$10^{15}$</td>
<td>$4 \times 10^{15}$</td>
<td>39929</td>
<td>969.7</td>
</tr>
<tr>
<td>F</td>
<td>$10^{14}$</td>
<td>...</td>
<td>1720</td>
<td>59.95</td>
</tr>
<tr>
<td>G</td>
<td>$10^{15}$</td>
<td>...</td>
<td>6712</td>
<td>307.4</td>
</tr>
</tbody>
</table>

FIG. 3. Overall resistance versus contact spacing for the non-irradiated sample. The red line is the best fit line ($R^2 \sim 0.99$) for the four points of data.
Schottky diodes, 10^{14} fast n/cm^2 (F) and 10^{14} fast plus 4 \times 10^{14} thermal n/cm^2 (A) caused no significant changes to \( R_C \) and \( R_S \). Additionally, the \( R_S \) decrease with 10^{14} n/cm^2 fast neutrons suggests improved GaN crystal quality.

At 10^{15} n/cm^2 fluence, however, \( R_S \) and \( R_C \) change significantly. In the fast neutron only case, \( R_C \) increased \~5x and \( R_S \) increased \~3x. The \( R_S \) increase can be attributed to crystal disorder caused by neutron collisions within the GaN lattice and the resultant lower carrier mobility. The stronger \( R_C \) increase can be attributed to a combination of (1) this lower mobility within the GaN below the metal as well as (2) metallurgical changes at the intimate metal-GaN interface. Regarding (1), \( R_C \) is dependent on the resistance of GaN directly underneath the contact, denoted by \( R_{SK} \), according to

\[
R_C = R_{SK}L_T/W
\]

for contact width \( W \) and transfer length \( L_T \). The proportionally larger \( R_C \) increase versus \( R_S \) indicates that metallurgical changes contribute significantly to \( R_C \) in addition to the changes in GaN below the metal.

At 10^{15} fast + 4 \times 10^{15} thermal n/cm^2, \( R_C \) increases by \~16x and \( R_S \) increases by \~19x. This dramatic increase of both \( R_C \) and \( R_S \) indicates that \( R_C \) dominates the TLM measurements of \( R_S \) in this case. The metallurgical effects on \( R_C \) and indirectly on \( R_S \) can be attributed to the reaction at the Ti-GaN, which can create new alloys, native point defects, and impurities.

Overall, these TLM results show that neutron fluences \( \geq 10^{15} \) n/cm^2 degrade ohmic contact functionality, and the disproportionate increase in \( R_C \) and \( R_S \) with additional thermal neutrons indicates their importance in promoting metallurgical reactions.

V. METALLURGICAL INTERFACE RESULTS

The pronounced current decreases and voltage shifts in Fig. 2(a) indicate that fluences of 10^{16} n/cm^2 substantially degrade these Schottky diodes so that metal-GaN contacts irradiated at this fluence can be used to observe morphological and chemical effects associated with device degradation. Indeed diodes irradiated with 1.4 \times 10^{16} fast + 2.6 \times 10^{16} thermal n/cm^2 exhibit a ‘melted’ morphology under an optical microscope. In contrast to Fig. 4(a), Fig. 4(b) shows that the ohmic contact developed wave-like diffraction regions suggestive of melted metal. The edges of the ohmic contact are no longer sharper, and the metal has spread to the previously bare GaN separating the ohmic from Schottky contact. Likewise, Fig. 4(b) shows that the Schottky contact developed circular regions where the metal has deformed as if melted, and/or is detaching from the GaN and ballooning outward. In order to understand these changes, we examined Ti-GaN and Ni-GaN ohmic and Schottky contacts, respectively, with these fluences using optical microscopy, scanning electron microscopy (SEM), and XPS.

A. Ni/GaN microscopy results

We acquired optical and SEM images of the Ni surface after fast and fast + thermal neutron irradiation. Figures 5(a) and 5(b) show that fast neutrons caused bumps with characteristic \~30–40 \mu m diameters (Fig. 5(b)) on the Ni surface. Scratches (black regions) on the surface were likely caused by sample loading/unloading. In contrast, the optical microscope image of fast + thermal neutron irradiated Ni in Fig. 5(c) shows qualitatively different features, i.e., large circular regions of localized “melting.” Figure 5(d) shows a higher magnification of such a circular region with a large center bump surrounding by cracks. Outside of the cracked area are circular rings of rainbow/diffracted colors. This diffracted light region resembles the ‘melted’ ohmic contact regions in Fig. 4 for our ‘melted’ ohmic contacts—suggesting that the ‘melted’ metal in Fig. 4 was nickel. The diameter of this specific localized ‘melted’ area was \~100 \mu m or \~5x larger with thermal neutrons than without. The AFM image, Fig. 5(e), confirmed that the center of the Ni ‘melted’ region consists of a cluster of bumps \~200 nm in height. The depths of the cracks were not measured but may extend through the 40 nm Ni thickness.

B. Fast + thermal Ni/GaN XPS

Besides the morphology changes observed via microscopy, the metal films undergo chemical changes due to neutron irradiation. We acquired XPS elemental spectra both at ‘melted’ and un-‘melted’ regions of the irradiated metal surface using the PHI Versaprobe’s scanning x-ray image (SXI) capability to focus on such micron-scale regions. We used Ar^+ etching to (1) remove surface contamination and (2) determine if Ga, N, and O were present in the Ni layer. We etched the Ni film in 30 s intervals for the first 6.5 min of etching, then in 1 min intervals for a total etch time of 9.5 min. AFM provided a measure of the Ar^+ etch rate equal to 1 nm per minute so that spectra obtained after 9.5 min corresponded to the outer \~10 nm of the 40 Ni film.

Figure 6 presents sets of spectra for Ga 2p, Ni 1 s, and O 1 s at ‘melted’ regions and, for O 1 s, an un-‘melted’ region, all with etch times ranging from 30 s to 9.5 min. Figure 6(a) shows that Ga 2p^{3/2} emission appears after \~4 min in the ‘melted’ Ni region and both Ga 2p^{3/2} and 2p^{1/2} emission intensities continue to increase with etch depth up to 9.5 min. N 1 s emission in Fig. 6(b) is present at all depths but relatively weak, consistent with ‘chemical trapping’ by reaction with Ni. Away from the un-‘melted’ regions, XPS spectra yield no evidence of Ga or N (not shown).
XPS spectra also showed significant oxygen contamination of the Ni film in both ‘melted’ and non-‘melted’ areas. Figure 6(c) shows O 1s core level emissions in a ‘melted’ region vs. etch depth. Beyond the first 30 s (0.5 nm) etch depth, the O 1s core level shifts by 0.5 eV to higher binding energy $E_B$, indicating O in two different chemical environments—adventitious oxygen near the free Ni surface at $E_B = 531.8$ eV and oxygen fully coordinated with Ni atoms deeper inside the film at $E_B = 532.3$ eV. The latter’s intensity appears higher and relatively constant with depth below the free surface.

Figure 6(d) shows analogous O 1s spectra for an un-‘melted’ area. In this case, the oxygen intensity is $\geq 2x$ lower than in Fig. 6(c) and decreasing with increasing etch depth. The constant 532.6 eV binding energy but decreasing intensity with etch depth suggests O diffusion into the Ni film. The qualitative differences between O 1s core level spectra in Figs. 6(c) and 6(d) suggests a chemical reaction in the ‘melted’ region but O indiffusion in the un-‘melted’ region.

C. Ti/GaN visual results

We obtained optical and SEM images of Ti on both GaN and sapphire surfaces. Figure 7(a) shows a relatively featureless SEM image of a Ti film on sapphire after $10^{16}$ fast n/cm$^2$. In contrast, the Ti film on sapphire optical image shown in Fig. 7(b) from the same wafer after $10^{16}$ n/cm$^2$ fast plus thermal shows elongated structures distributed across the entire Ti surface and in some cases aligned diagonally. The further optical magnification inset in Fig. 7(b) shows the elongated as well as smaller bubble-like features. Figure 7(c) shows the same Ti film after $10^{16}$ n/cm$^2$ fast plus thermal and at the same magnification as in Fig. 7(a), showing the same ~5–20 nm diameter bubbles and elongated structures with many of the latter attached at a central point. The AFM image in 7(d) shows the elongated structures with lateral dimensions on a ~10 $\mu$m scale and vertical heights of ~200 nm. The new features imaged in Figs. 7(b)–7(d) illustrate the pronounced morphological effects of thermal neutrons on Ti films deposited on GaN.

D. Fast + thermal Ti/GaN XPS

XPS survey scans acquired from the structures generated by thermal neutrons in Fig. 7 show the presence of Ga and N on a Ti film deposited on GaN. Figures 8(a) and 8(b), respectively, show peak features of N 1s and Ga 3d. Neglecting the adventitious C and O, the Ga, N, and Ti percentages...
appear in the ratio 19:33:48. The high proportion of Ga and N at the Ti surface indicates strong intermixing and segregation induced by thermal neutrons.

VI. METALLURGICAL INTERFACE ANALYSIS

A. Neutron irradiated Ni/GaN analysis

The morphological and chemical changes shown in Fig. 5 are consistent with previous studies of neutron-irradiated Ni, in which similar microscopic features were interpreted according to thermal mechanisms. Thus, Teodorescu et al. observed that thin film Ni irradiated with fast neutrons in vacuum changed the lattice from face centered cubic (fcc) to mixtures of fcc and hexagonal close-packed (hcp), which they interpreted as fast neutrons causing thermal spikes with temperatures high enough to amorphize the Ni. Depending on the duration of subsequent annealing, this amorphous phase could recrystallize in either fcc or hcp phase or a mixture of the two. Such thermal effects can account for the ‘melted’ and raised features imaged in Fig. 5 and be due to both kinematic energy transfer to as well as fission reactions with the metal. The irregular features and cracking evident in Fig. 5 may be due to inhibited recrystallization due to Ga, O, and N
impurities incorporated during heating as well as the differences in crystal symmetry.

Our SEM measurements also provide evidence of localized charging, indicative of an insulating phase at the irradiated Ni surface. Image instabilities and dark regions that moved within images were suggestive of electron pooling at the surface into extended negatively charged regions. Such insulating regions could certainly degrade the Ni contact to GaN, particularly at the intimate interface.

The detachment of metal from the GaN surface corresponds well with the I-V characteristics in Fig. 2(a) for the $1 \times 10^{16}$ fast and $1 \times 10^{16}$ whole neutron spectra I-V curves. Both no longer exhibit Schottky-like characteristics. Instead, both curves appear ohmic with very low current flow. The gap between metal and GaN adds an additional barrier to current flow along with insulating morphological changes to the metal, as exhibited by Fig. 5.

B. Neutron irradiated Ti/GaN analysis

The morphological and chemical changes shown in Fig. 7 are consistent with previous studies of TiN morphology under various magnetron sputtering conditions. Jayachandran et al. reported similar bubble precipitates and needle crystallites that varied with Ti-N stoichiometry and film thickness. Depending on the N$_2$ concentration, needle crystallites appeared within the Ti at 11%–17%, bubble precipitates formed up to 22%, and surface roughening occurred at higher concentrations. According to the XPS intensities in Fig. 8, the comparable N concentration relative to Ti is consistent with formation of TiN but also the formation of Ti-rich regions within the Ti layer that could account for the observed bubbles and needles. Perhaps most significant is that an annealing temperature $>500$ °C is required in order to diffuse N into the Ti film. Therefore, since our Ti/GaN contacts were not intentionally annealed, the presence of N within the Ti demonstrates that thermal neutron irradiation produced temperatures of $500$ °C or higher. Since the Fig. 8 XPS spectra also show that Ga as well as N diffused into the Ti overlayer, voids can form at the Ti/GaN interface. This is likely caused by radiation-enhanced diffusion (RED) at the metal-GaN interface. The RED coefficient is known to increase with irradiation-induced surface defects and is several orders of magnitude higher than the normal diffusion coefficient. Indeed such voids were reported for the annealed Ti/GaN interface. Similar voids would certainly degrade the metal-GaN ohmic contacts.

Regarding how the density of bubbles and other precipitates relate to the fast neutron-induced collision cascades, we can estimate the elastic scattering reactions produced in a GaN wafer based on the total elastic cross section of 1 MeV fast neutrons for Ga and N atoms, which are 3.1 and 2.3 barns, respectively. These values translate to an elastic scattering rate of $2.9 \times 10^{11}$/cm$^2$/s for $10^{16}$/cm$^2$ fast neutron fluence. However, the correspondence with the density of surface features induced by neutron irradiation is difficult to quantify since the nuclear reaction time is $<10^{-11}$ s whereas the resulting defect evolution can take from msec to months or longer in a multi-step dynamic process.

XPS results of bare GaN showed no evidence for the well-known neutron induced transmutation of Ga into Ge after $1 \times 10^{16}$/cm$^2$ fast $+ 6.67 \times 10^{15}$/cm$^2$ thermal neutron irradiation. XPS spectra in the 1210–1260 eV binding energy region showed no clear Ge 2p core level emission, the energy region showed no clear Ge 2p core level emission, the strongest XPS feature for Ge. This result is reasonable, given that the concentration of Ge atoms formed after thermal neutron irradiation is given by:

$$N_{TD} = \phi \Sigma_i n_i \sigma_{ic},$$

where $N_{TD}$ is the net concentration of transmuted atoms after thermal neutron irradiation. $\phi$ is the thermal neutron fluence, $n_i$ is the atomic concentration for isotope i, and $\sigma_{ic}$ is the thermal neutron capture cross section for isotope i. The estimated Ge production with respect to Ga $\rightarrow$ Ge transmutation is $\sim 8.5 \times 10^{14}$ Ge atoms/cm$^2$, which is well below the 0.1 at. % detection limit of XPS as well as the Si doping density of $3 \times 10^{16}$ Si/cm$^3$.

In general, such contacts should be protected from thermal neutron irradiation in radiation environments to prevent their degradation. Methods of radiation protection for
monolithic circuits have been well studied, e.g., for CMOS devices. Similar methods can be applied to GaN devices, for example, by depositing a radiation-absorbing coating on chip surfaces and by new metal contact elements that have minimized radiation cross sections and interface diffusion.

VII. CONCLUSIONS
Using a combination of electrical, chemical, and microscopy techniques, we determined fluence thresholds and the nature of device degradation for conventional GaN Schottky barriers and ohmic contacts by neutron irradiation. These studies showed that both fast and thermal neutrons degrade GaN device structures by introducing insulating phase and recombination centers that raise ideality factors for Schottky GaN device structures by introducing insulating phase and reaction with metal contacts. These results highlight the need for protecting both the ohmic and Schottky contacts in device applications subject to high neutron radiation environments.

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