Solution-processed van der Waals heterojunction as the damage-free gate contact for high performance GaN HEMTs

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Abstract:

The junctions formed between gate contact and III-nitride are crucial components of GaN-based electronics and optoelectronics. In this work, solution-processed inorganic Ti$_3$C$_2$Tx MXene films were spray coated on the AlGaN/GaN epitaxial wafer as the gate contact. The workfunction of MXene films was effectively enhanced by partial oxidization process, and accordingly, the gate leakage current and off-state drain current were significantly suppressed. The van der Waals heterojunction between MXene films and III-nitride without direct chemical bonding retained the pristine atomically flat native oxides of III-nitride, record high $I_{on}/I_{off}$ current ratio of $10^{13}$, and near ideal subthreshold swing of 61 mV/dec was achieved in the Schottky-Type gate GaN HEMTs. In addition, the MXene gate GaN HEMTs display superior electron mobility and transconductance, high uniformity with 20 measured transistors. The unprecedented performance can be correlated to the damage-free interface between MXene and III-nitride, negligible atomic diffusion of MXene into the semiconductor layer, and excellent adhesion between MXene and III-nitride. This work provides an alternative method to create contact not only in GaN HEMTs but also in other electronic and optoelectronic devices which uniquely features cost-effective, non-vacuum, high deposition rate, and damage-free properties.

Keywords: Ti$_3$C$_2$Tx MXene, GaN HEMTs, damage-free interface, record high $I_{on}/I_{off}$ ratio, near ideal subthreshold swing

1. Introduction

GaN-based high-electron-mobility transistors (HEMTs) are capable of delivering high current, high voltage, and high switching speeds characteristics and they have been widely used in power and radio frequency devices. Ohmic contact in the source and drain regions of GaN HEMTs received ample attention and the attempt to achieve a low contact resistance plays a key role in affecting the output current density, power-added efficiency, transconductance. For the Schottky-Type gate contact, it has equally crucial importance as it can impact the threshold voltage, $I_{on}/I_{off}$ current ratio, and subthreshold swing of GaN HEMTs. Moreover, the electric field peaks at gate edge in the drain side, thus hot electrons easily trapped by the defects around the gate edge can degrade the output power, device breakdown and dynamic performance. The gate contact quality is important factors related to these issues, however efforts are not addressed sufficiently yet in this area.

The requisite of the high performance Schottky-Type gate contact of GaN HEMTs includes high Schottky barrier height, low gate leakage current, large gate voltage swing, and good adhesion with III-nitride. Among typical Schottky-Type contact materials with III-nitride, such as Cu, Pt, Ti, Cr, Pb, Ir, Mo, ITO, TaN, TiN, Tungsten-based compounds (W, WN or WC), and Ni, Ni is the most commonly adopted gate materials in GaN HEMTs due to the high work function (5.1 eV) and good adhesion between Ni and III-nitride. Ni/Au bi-layer films are more widely used to further prohibit the oxidation of Ni films. Except Ni, Cu was reported to reduce the reversed gate leakage current by two orders of magnitude compared with traditional Ni/Au metal films. However, copper can diffuse into the underlying AlGaN layer easily, jeopardizing the interfacial quality of Schottky junction and increasing the
defect states. Pt is also the potential materials to replace Ni as the gate contact in GaN HEMTs, nevertheless, Pt typically presents an adhesion problem with III-nitride. Except Ni, Cu, and Pt, other gate electrode materials can suffer from a large gate voltage current densities.

MXenes, the new emerging family of 2D materials, have the atomic-thin layers of transition metal carbides, nitrides, or carbonitrides with promising electronic and optical properties. These years, MXenes have entered the field of by energy storage, optoelectronic, biomedical, and catalyst by virtue of its unique property such as metallic conductivity, hydrophilicity, negatively charged surface, and tunable work. By modifying the surface of MXene with different functional groups (-F, -OH, =O), the tunable work function can be achieved with a broad range between 2 and 8 eV. The metallic conductivity (10000 S/cm) of MXene films combined with its high work function by terminating the surface with more =O groups renders this material as the potential gate contact material in GaN HEMTs.

2. Results and Discussions

2.1 MXENE Film characterization

Atomic force microscopy (AFM) was deployed to characterize the surface morphology of MXene films, the thickness of single nanoflake was calculated to be around 1.6 nm as depicted in the inset of Fig. 1(a) which is consistent with previous report. The X-ray diffraction (XRD) and Raman spectra of MXene films are shown in Fig. 1(b) and (c). The XRD peak located at 6.6° corresponds to (002) planes of MXene films. Raman spectra displays broad peaks at 207, 376, 575, and 739 cm\(^{-1}\) which indicates the coexistence of various surface functional groups on the surface of MXene films, coinciding well with previous report. The XRD pattern combined with the Raman spectra confirm the successful preparation of Ti\(_3\)C\(_2\)T\(_x\) MXene films.

MXene films coated on AlGaN/GaN epitaxial wafer were annealed at 500 °C for 50 s in ultra-high vacuum conditions (10\(^{-7}\) Torr). The chemical composition of MXene films as well as the annealing effects were investigated using high resolution X-ray photoelectron spectroscopy (XPS). XPS spectra of Ti 2p, C 1s and O 1s core levels from fresh MXene films and annealed ones have been displayed in Fig. 1(d)–1(f) and Fig. 1(g)–1(i), respectively. The Ti 2p core level was fitted with six doublets (Ti 2p\(_{3/2}\) – Ti 2p\(_{1/2}\)) with a fixed area ratio equal to 2:1 and doublet separation of 5.7 eV (except for Ti_C the doublet separation is equal to 6.2 eV). The Ti 2p\(_{3/2}\) components were located at 454.9 eV, 455.6 eV, 457.0 eV, 458.5 eV, 459.4 eV, and 460.3 eV corresponding to Ti-C, Ti\(^{2+}\), Ti\(^{3+}\), Ti\(^{4+}\) (TiO\(_2\)), Ti (TiO\(_{2-x}\)F\(_x\)), Ti (C-Ti-F\(_x\)) respectively. After annealing, we did not observe a significant change in the Ti 2p spectra. The C 1s core level was fitted using seven components located at 281.8 eV, 282.8 eV, 284.2 eV, 284.8 eV, 285.7 eV, 288.1 eV and 289.9 eV corresponding to C-Ti, C-Ti-O, C=C (sp2), C-C/C-H (sp3), C-O, C=O,O-C=O/C-F , respectively.
After thermal annealing, we can observe a decrease of the carbon contribution to C 1s spectrum from contaminants (C=C, C-C, C-O, C=O, O-C=O/C-F) compared to fresh MXene films.

The O 1s core level was fit using five components located at 529.7 eV, 530.7 eV, 531.8, 532.7 eV, and 533.8 corresponding to TiO₂, C-Ti-Oₓ (moiety) and/or organic compounds due to atmospheric surface contaminations adsorbed hydrocarbons (OR), C-Ti-(OH)ₓ and/or (OR), adsorbed hydrocarbons (OR), and H₂O adsorbed (moiety) and/or (OR). The surface contaminations show an obvious drop after thermal treatment. The ratios of O(C-TiOₓ)/O(C-Ti(OH)ₓ) in MXene films does not show obvious change after thermal treatment. As the ratio of
O(C-TiO$_x$)/O(C-Ti(OH)$_x$) is related to the work function of MXene films,\textsuperscript{33} this result indicates the stability of the work function before and after thermal annealing in high vacuum conditions.

### 2.2 Characterization of the interface quality of MXene/ III-nitride

![Image](image_url)

Fig. 2. (a) Microscope image of the GaN HEMT. (b) Cross-sectional TEM image of MXene gate GaN HEMTs. EDS maps of (d) Ti and N signals and (e) Ti and N signals of the MXene/III-nitride junctions. (f) EELS spectrum profiles across the interface from III-nitride to the MXene films.

The solution processed MXene were spray coated on AlGaN/GaN epitaxial wafer, lift-off process was carried out in an ultra-sonic acetone bath with controlled power. Thermal annealing was applied to the devices at 500°C in ultra-high vacuum for 50 s. The microscope image of the GaN HEMT in Fig. 2(a) shows the uniform surface morphology of the 5 μm width MXene film gate contact. Fig. 2 (b) and (c) show the cross-sectional TEM image of GaN HEMT in the gate contact region. Van der Waals heterojunctions (vdWHs) were formed between MXene and III-nitride. The sandwich-like MXene fillms display typical two-dimensional morphology with the thickness of ~1.5 nm for a single-layer. An atomically flat and uniform interface between MXene fillms and III-nitride is observed due to the damage-free gate contact fabrication process. The traps at the junctions and rough interface which can degrade the electron mobility can be significantly suppressed due to the superior interface quality between MXene and III-nitride.\textsuperscript{34,35} Additionally, Cross-sectional X-ray spectroscopy (EDS) in Fig. 2 (d) and (e) show the continuous and homogeneous N, Ga, and Ti signals in III-nitride and MXene films, respectively. Atomic spatial distribution collected by electron energy loss spectroscopy (EELS) in Fig. 2 (f) shows the minimal overlap of the above elements, implying negligible atomic diffusion of
Ti$_3$C$_2$T$_x$ MXene into underlying III-nitride. Moreover, compared with the high-energy metallization techniques like e-beam evaporation or magnetron sputtering, disordered chemical bonds between two dissimilar materials can be significantly reduced due to the sharp vdWHs.$^{36,37}$

2.3 Transistor performance before and after RTP

![Graphs showing transistor performance](image)

Fig. 3. (a) Transfer and transconductance, (b) gate leakage current density, and (c) output current density of GaN HEMTs with MXene films as the gate contact before and after annealing.

After finishing the device fabrication process, the wafer was annealed in high vacuum conditions at 500°C for 50 s. As shown in Fig. 3(a) and (b), significant improvement of transistor performances were observed after thermal treatment with one order reduced off-state drain leakage current, improved on-state drain current and grossly suppressed transconductance and drain current sweep hysteresis. Accordingly, the $I_{ON}/I_{OFF}$ current ratio increased from 1.2×10$^7$ to 1.7×10$^8$. It is important to achieve a low off-state drain leakage current and high on-state drain current as they play a key role in determining the flicker noise and power consumption of GaN HEMTs.$^{38,39}$ In addition, the sub-threshold swing (SS) also reduces from 69 to 63 mV/dec after thermal treatment. The suppressed transconductance and drain current sweep hysteresis, in conjunction with the steeper SS, is the indication of decreased defects after thermal treatment which is attributed to the reduced carbon related defects in the MXene films in above-mentioned XPS results. As the gate contact were fabricated with solution and spray coating processes at room temperature, leaving carbon residue in MXene films,$^{37}$ we thus conclude thermal annealing is an effective method to reduce these contaminations and thus improve the transistor performances. In addition, the gate leakage current does not show obvious decreasing after thermal annealing, therefore, the reduced off-state drain leakage current cannot be attributed to the change of gate leakage current with thermal treatment.

As displayed in Fig. 3(c), the on-state resistance extracted from the output current curves reduce from 12.3 Ω·mm to 9.1 Ω·mm after thermal treatment. The decreasing of on-state resistance can be ascribed to the Ohmic contact quality ameliorating with thermal annealing. Accordingly, the output current increased with improved contact in the source and drain region accordingly. In addition, the decreasing of defects induced traps can also be responsible in part for the reduced on-state resistance.$^{40}$
2.4 Uniformity test

Fig. 4. (a) Dual-sweep logarithmic-scale transfer and gate leakage current density, (b) dual-sweep transconductance, and (c) dual-sweep field-effect mobility characteristics of MXene gate GaN HEMTs.

The $I_{ON}/I_{OFF}$ ratio, gate leakage current, transconductance, and output current density of MXene gate GaN HEMTs were collected with 20 different transistors to confirm reproducibility and uniformity in the wafer. Before measurements, the wafer receives post gate annealing at 500°C for 50 s in ultra-high vacuum conditions as discussed before. As shown in Fig. 4(a)–(c), MXene gate GaN HEMTs show high uniformity with regard to threshold voltage, $I_{ON}/I_{OFF}$ ratio, SS, and transconductance. The corresponding highly uniform output curves are displayed in Fig. S1. (Supporting Information).

Gate-recessed and fluorine-implanted GaN HEMTs often suffer from non-uniformity issues and implant damage concerns. For example, the threshold voltage of gate-recessed GaN HEMTs can range from -2 to 1 V even after device fabrication and material deposition processes optimization. Ni/Au bilayer was commonly adopted as the gate contact in depletion-mode Schottky-Type gate GaN HEMTs, however, the $I_{ON}/I_{OFF}$ ratio was limited below $\sim 10^7$ to $10^8$ due to large gate leakage current density. As shown in Fig. 4(a), for Schottky-Type gate GaN HEMTs with MXene films as the gate contact, the threshold voltage demonstrates a narrow hump between -4.9 and -5.1 V of the 20 measured transistors. Gate leakage current of MXene gate GaN HEMTs was suppressed to a very low value, and thus $I_{ON}/I_{OFF}$ ratios range over the values from $2.1 \times 10^8$ to $3.3 \times 10^9$ were achieved which was two orders improvement compared with traditional Ni/Au gate GaN HEMTs. In addition, from the transfer characteristics, a negligible small hysteresis was observed between the up and down sweep measurements. The small hysteresis implies low interface or border traps at the junctions between MXene films and III-nitride. Moreover, a steep SS was achieved with a small variation from 61 to 65 mV/dec. As discussed above, the value of SS is closely related to the density of defects, the steep SS of MXene gate GaN HEMTs also indicates the superior interfacial quality. Frequency dependent hysteresis of MXene gate GaN capacitor was further deployed to confirm the low density of traps as shown in Fig. S2, (Supporting Information).

The extracted transconductance and field-effect mobility as a function of $V_{GS}$ was shown in Fig. 4(b) and (c). The field-effect mobility was calculated by using the equation:
\[ \mu_{FE} = \frac{L}{W} \frac{1}{C_{ox} V_D} \frac{1}{g_m} \]

where \( L \) is the gate length, \( W \) is the gate width, \( C_{ox} \) is the capacitance per unit area, \( V_D \) is the drain voltage, and \( g_m \) is the transconductance.\(^{45,46}\)

The scattering originated from the thickness fluctuation of the surface native oxide as well as diffused atoms induced charges can degrade the 2DEG mobility in the channel,\(^{35,40,47}\) thus the high field-effect mobility can also be attributed to the superior interfacial height homogeneities and the diffusion-free gate contact owning to the vDWHS contact between MXene films and III-nitride. Furthermore, the transconductance and field-effect mobility display high uniformity between different devices on the wafer and the small hysteresis during the up and down sweep measurements also signify the damage-free surfaces of III-nitride play a key role in determining the transistor performances. The abovementioned results verify our solution-processed \( \text{Ti}_3\text{C}_2\text{Tx} \) MXene gate GaN HEMTs are capable of delivering superior performances in terms of reproducibility, uniformity, and unprecedented single transistor performance.

**Oxidation of MXene films**

![Fig. 5. (a) UPS spectra of GaN HEMT, fresh-\( \text{Ti}_3\text{C}_2\text{Tx} \)/HEMT, and 500-\( \text{Ti}_3\text{C}_2\text{Tx} \)/HEMT. (b) Ti 2p, and (c) O 1s XPS spectra of MXene films after thermal oxidation. (d) Transfer and gate leakage current density, (e) transconductance, and (f) output current density of GaN HEMTs.](image)

MXenes are vulnerable oxidized in humid ambient even at room temperature which could impact the device performances.\(^{48}\) The MXene gate GaN HEMTs were put in air for one month
to test the thermal stability of the devices. Unexpectedly, the $I_{\text{ON}}/I_{\text{OFF}}$ ratio can be improved by one order after one month soft oxidization in ambient at room temperature measured on the same device while the output current remained unchanged. (Details in Fig. S3, Supporting Information). MXene films coated on the AlGaN/GaN epitaxial wafer were transferred to a RTP chamber, thermal treatment was carried out for 50 s at 500 °C to accelerate the oxidization process. Fig. 5(a) shows the ultraviolet photoelectron spectra (UPS) from GaN HEMT, fresh MXene films, and annealed Mxene films. The work function value of 3.95, 4.33 eV and 4.45 eV were obtained, respectively. The energy difference between valance band top ($E_v$) and the Fermi level ($E_F$) of AlGaN/GaN epitaxial wafer derived from the UPS spectra in the low binding energy region is 3.09 eV. (Fig. S4, Supporting Information). Based on these results, as shown in Fig. 5(b) and (c), the calculated build-in potential $qV_0$ between MXene and III-nitride increased from 0.38 to 0.50 eV after thermal oxidization, and the Schottky barrier height also increased from 0.68 to 0.80 eV. The gate leakage current can be suppressed to a lower value as a higher Schottky barrier height formed.

XPS analysis were performed to characterize the chemical composition of MXene films after partial oxidization. The XPS spectrum of the Ti 2p core level in Fig. (5b) shows an increase of Ti$^{4+}$ (TiO$_2$), Ti (TiO$_2$xF$_x$) contributions compared with fresh Mxene films (Fig .1(d)), indicating the partial oxidization of the films. As shown in in Fig. 1 (e) and Fig. 5(c), the ratio of O(C-TiO$_x$)/O(C-Ti(OH)$_x$) determined from the XPS spectrum of O 1s core level increased from 0.3 to 0.9 after the partial oxidization of MXene films. The peculiar and abundant surface functional groups of MXene films confer them with specific electrical and optical properties. For example, compared with -F and -OH terminated groups, MXenes terminated with =O groups exhibit a higher work function. Therefore, the increased work function after thermal oxidization is consistent with the observed increase ratio of O(C-TiO$_x$)/O(C-Ti(OH)$_x$). As shown in in Fig. 5(f), after thermal oxidation, the carbon related contaminations such as C=C, C-C, C-O, C=O, O-C=O/C-F increased compared with the fresh MXene films shown in Fig. 1(e).

As shown in Fig. 5(e) and (f), after thermal oxidization of the wafer, the gate leakage current of MXene gate GaN HEMTs can be significantly reduced without sacrifying the output current, and accordingly, $I_{\text{ON}}/I_{\text{OFF}}$ ratio can be increased by four orders of magnitude to $\sim 10^{13}$ which is six orders higher than the traditional Ni/Au gate GaN HEMTs. As discussed before, the increased work function of MXene films after thermal oxidization is responsible in part for the significantly reduced gate leakage current. Additionally, TiO$_2$ from partially oxidization of MXene films can act as a gate dielectric, further reducing the gate leakage current, thereby enhancing the $I_{\text{ON}}/I_{\text{OFF}}$ current ratio. However, the threshold voltage shifts to negative value after thermal treatment which can be attributed to the positive charges at the TiO$_2$/ III-nitride interface. As shown in Fig. 5(b), the peak transconductance of MXene gate GaN HEMTs display a higher value than Ni/Au gate GaN HEMTs.
3. Conclusion

In this study, solution-processed MXene films were spray coated on GaN HEMTs as the gate contact. Diffusion-free and atomically flat interface was achieved owing to the formation of vdWHs between MXene films and III-nitride. Highly uniform MXene gate GaN HEMTs deliver high \( I_{on}/I_{off} \) current ratio, transconductance, field-effect mobility and near ideal SS. This work provides a cost-effective, non-vacuum, high deposition rate, and damage-free method to create contact not only in GaN HEMTs but also in other GaN-based electronic and optoelectronic devices. Moreover, except for the Schottky-Type gate material of depletion-model GaN HEMTs, MXene films also have great potential as the gate contact material in other type of GaN HEMTs like the P-GaN and GaN metal-insulator-semiconductor HEMTs which are under study now.

Methods

Materials Preparations:

GaN HEMT growth

The GaN HEMT structure used in this paper was grown on a 6 inch Si substrate by using the metal–organic chemical vapor deposition system. 4.7 μm GaN buffer layer was sandwiched between the Si substrate and 300 nm GaN channel. And then 21 nm Al\(_{0.25}\)Ga\(_{0.75}\)N barrier was deposited on top of the GaN channel. 2 nm GaN cap was covered on the surface of AlGaN barrier.

MXENE synthesis

The Ti\(_3\)C\(_2\)T\(_x\) colloidal solution was synthesized with the same method as previous report\(^{22,31}\) and then it was stored in vacuum ready for spray coating.

MXene characterizations

XPS studies were carried out in a Kratos Axis Supra DLD spectrometer equipped with a monochromatic Al K\(\alpha\) x-ray source (hv=1486.6 eV) operating at 75 W, a multichannel plate and delay line detector under a vacuum of 1~10\(^{-9}\) mbar. The survey and high-resolution spectra were collected at fixed analyzer pass energies of 160 eV and 20 eV, respectively and quantified using empirically derived relative sensitivity factors provided by Kratos analytical. Samples were mounted in floating mode in order to avoid differential charging. Charge neutralization was required for all samples. UPS spectra were obtained using a He-I excitation (21.22 eV) source at pass energy 10 eV. The samples were mounted in contact mode for UPS measurements. Raman spectra were collected with the Wintec Apyron Raman spectrometer equipment with a 633 nm laser source excitation. X-ray diffraction (XRD) patterns of the MXene films were measured with Bruker D2 PHASER. The STEM lamellae samples were fabricated by a FIB technique (Helios G4, FEI). The STEM images were acquired with Titan G2 60-300 (FEI) at an acceleration voltage of 300 kV. For the EELS measurement, the current was kept around 41pA to avoid beam-induced damage. The electrical measurements including were conducted by a
Keysight B1500 Semiconductor Parameter Analyzer in the dark under ambient conditions at room temperature.

**Device fabrication and measurements**

Starting from the Mesa etching to isolate the single HEMT device, a piranha cleaning process have been deployed to fully clean the residue photoresist and remove the organic contaminations. Before the bi-layer films Ti/Al/Ti/Au deposition in the source and drain region, diluted hydrochloric acid was used to remove the native oxide on the surface of HEMT structure. Rapid thermal annealing process at 870°C in Ar atmosphere was used to form ohmic contact. AZnLOF 2020 was used as the negative photoresist to define the gate region. Subsequently, MXene was spray coated on the whole wafer and lift-off process was used to peel off the MXene outside the gate region. Post-gate annealing at was used in a RTP chamber at 500 °C for 50 s in 1000 sccm Ar atmosphere. The other set of samples were subjected to the pulsed laser deposition chamber and annealed at 500 °C for 50 s in ultrahigh vacuum conditions. The Al₂O₃ thin films fabricated by atomic layer deposition at 300 °C was adopted as the passivation layer and buffered oxide etching was used to remove the Al₂O₃ films in selective area to expose the MXene films for probing.

**Supporting information**
**Fig. S1.** Output current of 20 measured transistors show the high uniformity of MXene gate GaN HEMTs.

**Fig. S2.** Frequency dependent hysteresis of GaN capacitor with MXene films as gate contact. The small hysteresis indicate the high interface quality between MXene films and GaN.

**Fig. S3.** (a) Transfer and transconductance, (b) output current density of GaN HEMTs with MXene films as the gate contact after one month exposure to open-air conditions. The measurement was carried with the same device shown in Fig. 3. The off-state gate leakage current show one order decreasing without sacrificing the output current density after one month later.

**Fig. S4** UPS spectra of GaN HEMT in the low binding energy region.
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