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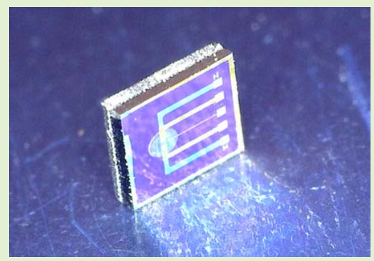
Enhanced Sensitivity Pt/AlGaN/GaN Heterostructure NO_2 Sensor Using a Two-Step Gate Recess Technique

Jianwen Sun, Teng Zhan, Robert Sokolovskij, Zewen Liu, Pasqualina M. Sarro, *Fellow, IEEE*, and Guoqi Zhang, *Fellow, IEEE*

***Abstract*—Based on our proposed precision two-step gate recess technique, a suspended gate-recessed Pt/AlGaN/GaN heterostructure gas sensor integrated with a micro-heater is fabricated and characterized. The controllable two-step gate recess etching method, which includes O_2 plasma oxidation of nitride and wet etching, improves gas sensing performance. The sensitivity and current change of the AlGaN/GaN heterostructure to 1-200 ppm NO_2/air are increased up to about 20 and 12 times compared to conventional gate device, respectively. The response time is also reduced to only about 25 % of value for conventional device. The sensor has a suspended circular membrane structure and an integrated micro-hotplate for adjusting the optimum working temperature. The sensitivity (response time) increases from 0.75 % (1250 s) to 3.5 % (75 s) toward 40 ppm NO_2/air when temperature increase from 60°C to 300°C. The repeatability and cross-sensitivity of the sensor are also demonstrated. These results support the practicability of a high accuracy and fast response gas sensor based on the suspended gate recessed AlGaN/GaN heterostructure with an integrated micro-heater.**

***Index Terms*—AlGaN/GaN, gate recess, gas sensor, NO_2.**



I. Introduction

RECENTLY, there have been growing concerns about environment pollution, such as photochemical smog and acid rain, was caused by the growing nitrogen dioxide (NO_2) emission mainly from combustion of automotive exhaust [1], [2], industrial processes [3]. Especially, the monitoring of exhaust gas of the automotive vehicles was required for the feedback control of the catalytic reduction system (SCR) in order to reduce NO_2 emission. Accordingly, the NO_2 sensor installed in the automobile exhaust gas after-treatment-system have to work in the harsh environment of high temperature. The harsh environment, low power, continuous NO_2 monitoring sensors with excellent sensing performance was desired.

Among the various sensing technologies,the chemical resistive type is one of the most comprehensively researched and developed for NO_2 detection over the last few decades. Various materials of transducer such as metals, polymers, carbon-based nanomaterials (carbon nanotubes, graphene) and metal oxide semiconductors have been employed as NO_2 sensitive layers [4]–[6]. For further enhancing the gas performance, nanostructures of the sensitive materials (nanoparticles, nanowires, nanosheets, nanobelt etc.) have been investigated due to high surface-to-volume ratio, high electrical or heat conductivities, chemical inactivity [7]. Silicon-based field effect devices such as Schottky diodes and metal oxide semiconductor field-effect transistor (MOSFET)are extensively studied for NO_2 detection [8], [9]. The narrow energy bandgap of Si limits the maximum operation temperature of MOSFETs to approximately 200 °C [15].

In order to improve sensing characteristics and operation in harsh environment, other semiconductor materials includ-

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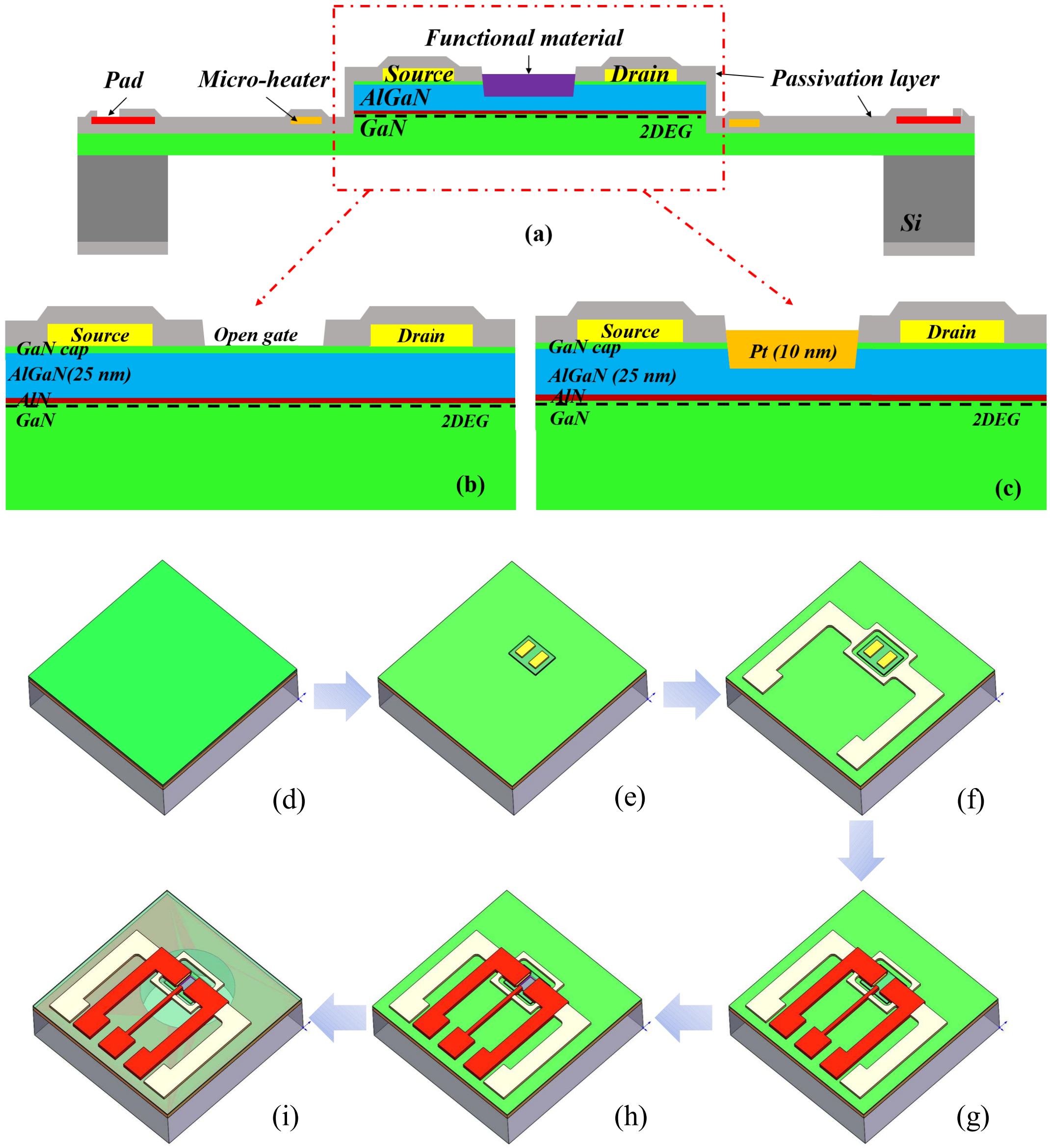

Fig. 1. (a) Cross-section schematic representation of GaN-based HEMT sensor. Cross-section schematic of (b) open gate and (c) 10 nm Pt thin film on recess gate.Main steps of fabrication process: (d) silicon substrate with epitaxial layers; (e) mesa and ohmic contact forming; (f) micro-heater deposition and passivation; (g) interconnect metal deposition and passivation; (h) gate recess and functional materials deposition; (i) backside etching form suspended membrane.

ing GaAs, InP or SiC were investigated for NO_2 sensor applications [10]–[14]. Gallium nitride (GaN) based sensors have been drawing attention due to their unique properties, such as high thermal resistance and chemical stability [16]. Compared to AlGaN/GaN Schottky diode sensors [17]–[19], AlGaN/GaN high electron mobility transistor (HEMTs) sensors provide several advantages: higher current changes [20], lower theoretical detection limits [21] and the modulated sensitivity by changing the gate bias [23]. AlGaN/GaN heterostructures exhibit great potential for the development of a high performance sensing platform, due to the high carrier density two-dimensional electron gas (2DEG) at the interface, which is sensitive to the changes in surface potential [24]. By functionalizing the gate area of a HEMT sensor for H_2 [25], NO_2 [26], NH_3 [27], acetone [28], glucose [31], DNA [32], protein [33] and ions [34]–[36] have been reported.

A locally thinned (20-30 nm) AlGaN barrier recess has also been applied for enhancement mode HEMTs [37], Au-free ohmic contact [38], CMOS compatible ohmic resistance reduction [39] and to improve the sensitivity of HEMT-based sensors [21], [40]–[42]. The AlGaN/GaN recess would be commonly done by reactive ion etching (RIE) using Cl_2/BCl_3 plasma with low power or thermal oxidation at 650 °C coupled with KOH oxide etching at 70 °C [43].However, the use of dry RIE etching often exhibits difficulties of depth control, non-uniformities, etching residues and lattice damage due to ion bombardment. Furthermore, cyclic oxidation using oxygen plasma, followed by wet etching to fabricate gate recess of GaN device was reported [44]. However, the etching rate (~0.38 nm/cycle) was too slow for practical application [44]. Our early work investigated the oxygen plasma oxidation and HCl wet etch-

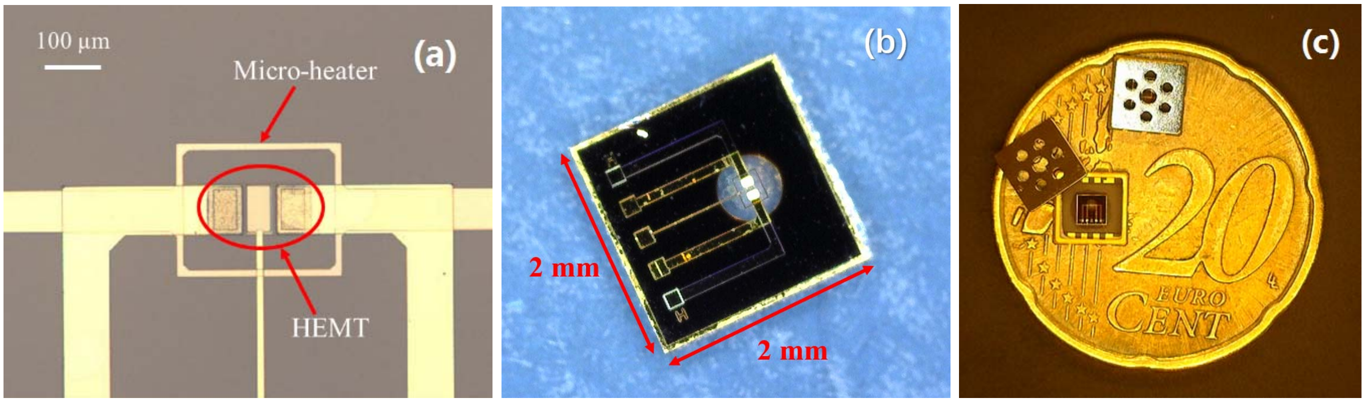

Fig. 2. (a) Optical image of recess gate Pt/AlGaN/GaN device; (b) optical image of complete sensor; (c) Sensors with CQFN package on 20 Euro cent coin.

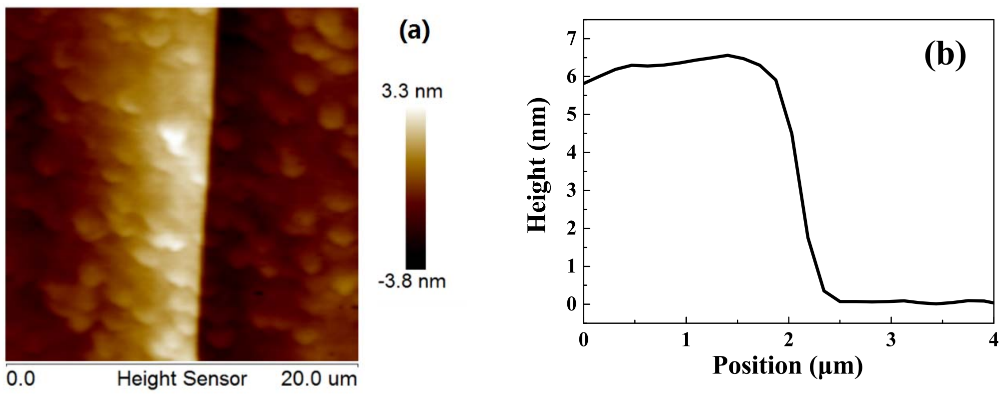

Fig. 3. (a) AFM image of GaN step on testing wafer (b) Step profile of two-step gate recess technique.

ing for AlGaN/GaN, and obtained a controllable etching rate 0.6-11 nm/cycle [45].

A gate recess of AlGaN/GaN heterostructure sensor using ICP-RIE dry etching with low-ppb level sensitivity was demonstrated by Peter Offermans *et al.* [21], [22], [42], [46]. However, the current change is nA level and response time is about 30 mins for 100 ppb NO_2/N_2, which is not easy for practical application but offer nevertheless indisputably high potential [21], [42]. The authors later developed a gate recess AlGaN/GaN heterostructure sensor integrated a micro-heater. The response time and recovery time were optimized to 1 min and 5 mins for 10-100 ppb of NO_2 [22]. The sensitivity of a urea biosensor based on gate-recess AlGaN/GaN adapted by photoelectrochemical etching method was improved about 40 % [47].Nevertheless, three-terminal HEMT-based gas sensors for NO_2 response with recessed barrier and the catalytic metal gate have not been studied.

In this research, we have the successful implemented a suspended gate recessed Pt/AlGaN/GaN heterostructure gas device integrated with a micro-heater. The gate recess is etched by a dedicated developed precise two-step etching method, including O_2 plasma oxidation and wet etching. We find that the sensitivity and current change to NO_2 gas of these devices are boosted with the additional benefit of faster response time. The temperature of the membrane is modulated by the micro-heater unit based on Joule heating. The sensing performance of the sensor at different temperatures are studied. The repeatability and selectivity of sensor are also demonstrated.

II. Experimental Section

Figure 1 illuminates a schematic representation of the cross-section of GaN based membrane sensor with integrated micro-heater. Figure 1(b) and (c) show the enlarged active area of open gate and recess gate structure. The AlGaN/GaN heterostructure was grown on a (111) silicon wafer, 100 mm in diameter and 1 mm thick using Metal-organic Chemical Vapor Deposition (MOCVD).The epitaxial structure consisted of an undoped GaN buffer layer (2 μm), followed by a AlN interlayer (1 nm), an undoped $Al_{0.26}Ga_{0.74}N$ barrier layer (25 nm), and a 3 nm GaN cap layer. The electron mobility of the 2DEG was 1500 cm^2/V-s, with a sheet electron density of 1×10^{13} cm^{-2}.

The fabrication process started with a mesa etching to define the active area. Then, Ti/Al/Ti/Au (20/110/40/50 nm) metal contacts were evaporated, followed by a rapid thermal anneal at 870 °C for 45 s under N_2 ambient. Next, an evaporated Ti/Pt (30/200 nm) layer was patterned by lift-off to form the micro-heater, followed by a 200-nm PECVD SiO_2 layer for isolation from the interconnect layer. The evaporated Ti/Au (20/300 nm) layer stack is then used to form metal interconnect. The topside of the wafer was passivated with a 300 nm PECVD SiO_2 layer and the backside was polished to 400 μm. The

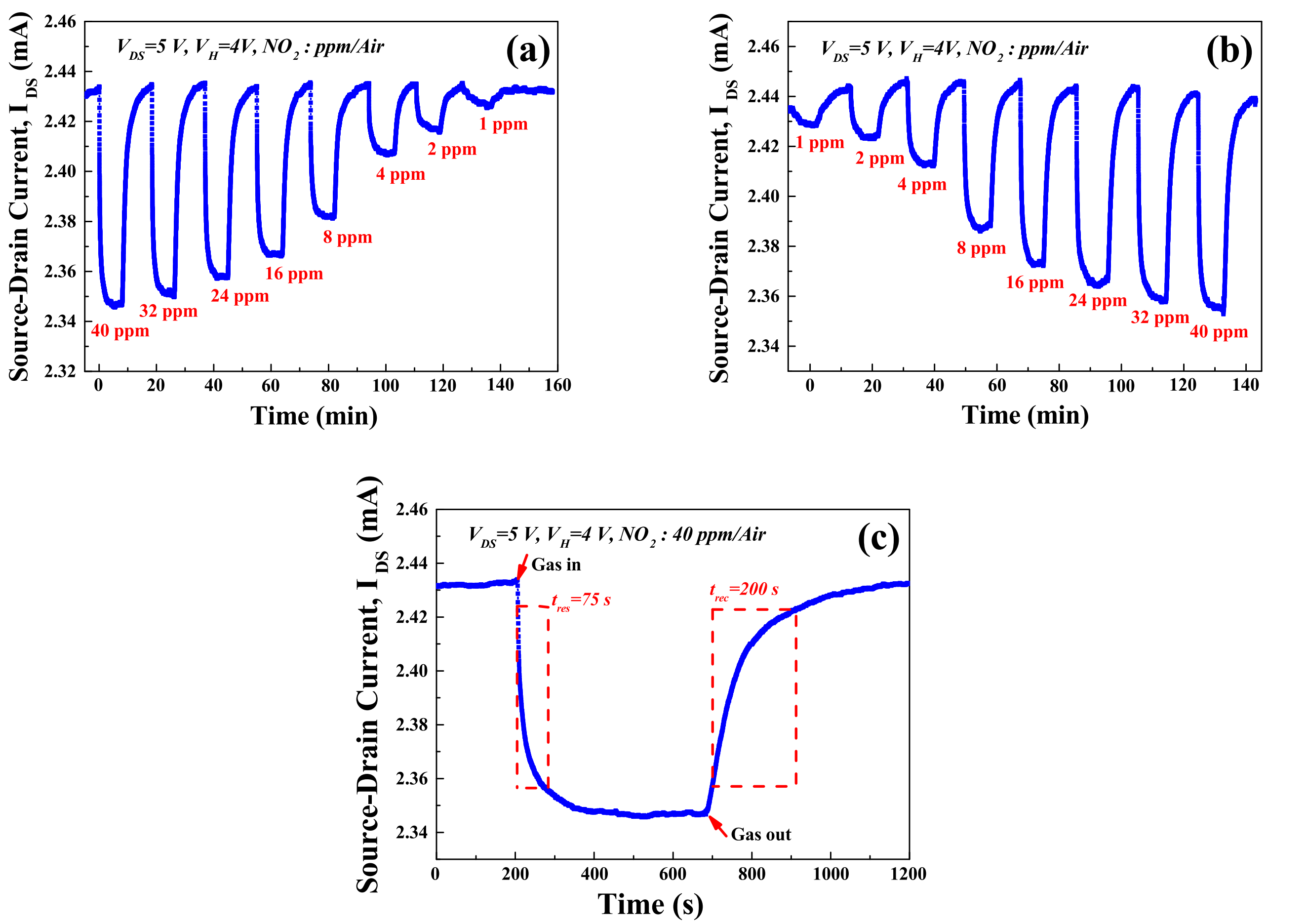

Fig. 4. (a) Transient response characteristics upon injection and purge of NO_2 in dry air ambient. During all measurements $V_{DS} = 5$ V, $V_H = 4$ V (a) with NO_2 gas concentration decreasing (b) with NO_2 gas concentration increasing (c) Enlarge part of the response curve of 40 ppm.

topside SiO_2 layer was etched in the BOE solution to open the contact pads and gate windows. To fabricate the gate-recessed structure, nitride oxidation was done in an ICP-RIE etcher using O_2 plasma for 3 min, followed by 1 min oxide etching in a 1:4 HCl:H_2O solution at room temperature. Then a 10 nm-thick Pt layer was evaporated and patterned on the 80 μm $\times$ 40 μm gate area. The silicon substrate was etched from the backside by deep reactive ion etching (DRIE) using 5 μm-thick SiO_2 layer as hard mask to form a circular membrane (650 μm in diameter). The main steps of fabrication process are shown in Figure 1 (d)-(i).

Figure 2 (a) shows the topside optical image of gate re-cess Pt/AlGaN/GaN device. Figure 2 (b) shows the opti-cal image of the complete sensor with the size of 2 mm*2 mm. Then, the chip was attached to ceramic quad flat no lead (CQFN) package, and Au-wire bonding was utilized to interconnect the bond pads of the Pt/AlGaN/GaN device to the electrical con-tact points of CQFN, as shown in Figure 2(c). The packaged sensor was placed in a chamber and electrically connected to a Keithley 2400 source meter. Before testing, the sensors were preheated at different temperature for about 30 mins to get the stable output. The target gases were injected into the chamber through a rubber plug by a syringe. After the current reaching a new saturated value, the gas in test chamber was drawn out by micro air pump. The sensitivity is given by following equation

$$S(\%) = \frac{\Delta I_{DS}}{I_{DS,air}} = \frac{\left|I_{DS,Gas} - I_{DS,air}\right|}{I_{DS,air}}. \tag{1}$$

where $I_{DS,Gas}$ and $I_{DS,air}$ are the drain current of sensor in target gases and air ambient, respectively. The response time (t_{res}) and recovery time (t_{rec}) are defined as the time for the drain current to change from 10 % to 90 % of its saturated value to gases or vapor. The AFM image of the surface morphology and the step profile of the gate recess on a test wafer are shown in Figure 3 (a) and (b). The depth (Δd) of the gate-recessed region after etching was about 6 nm. More details about the two-step gate recess technique [45] and the AlGaN/GaN sensors can be found in our earlier publications [20], [28], [48]–[52].

III. Results and Discussion

The surface membrane temperature is modulated by Joule heating of the micro-heater when the current passes the Ti/Pt layer. To calculate the membrane temperature, a calibration is required at various heating voltages. Ac-cording to the measurement results in our previous publications [28], [50], the surface temperature of device under the work mode (V_{DS} = 5 V) is 60 °C, 82 °C, 135 °C and 300 °C when

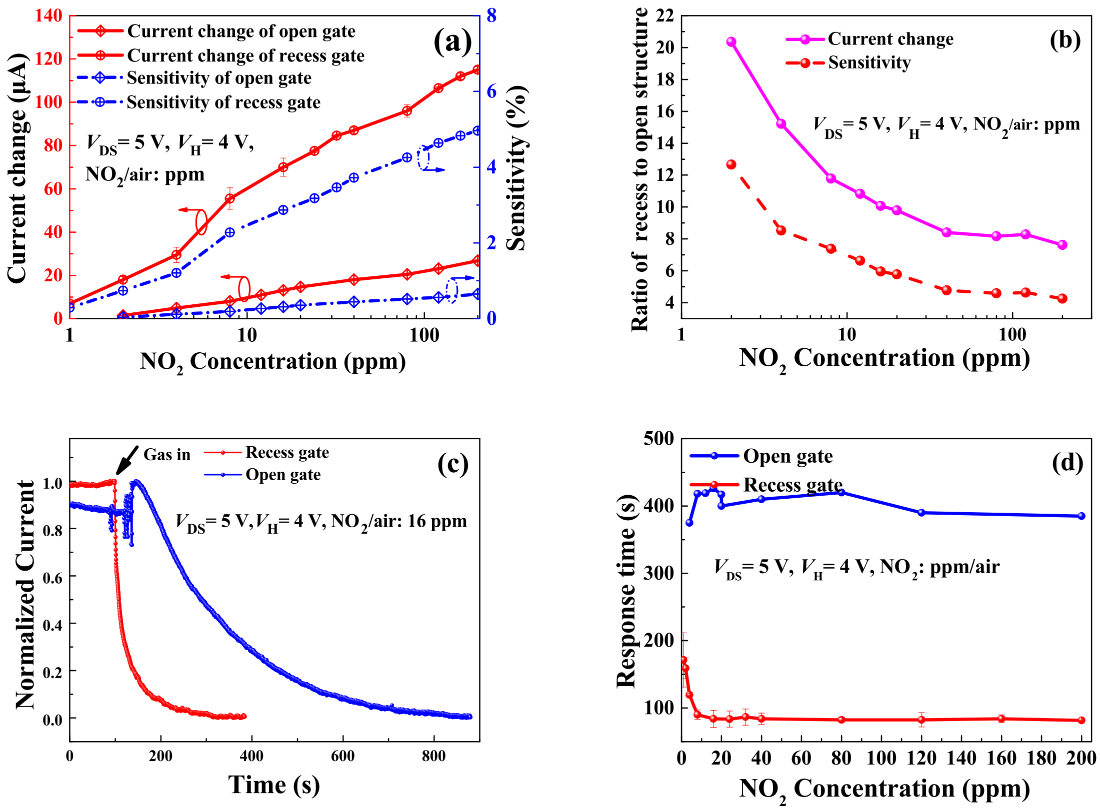

Fig. 5. (a) Current change/sensitivity and (b) corresponding ratio of the sensor with recess gate and open gate structure to-ward 1-200 ppm NO_2/air gas. (c) Normalized drain current response of the sensor with open gate and recess gate to-ward 16 ppm NO_2/air gas. (d) Response time of the sensor with open gate and recess gate. During the measurement the $V_{DS} = 5$ V and $V_H = 4$ V.

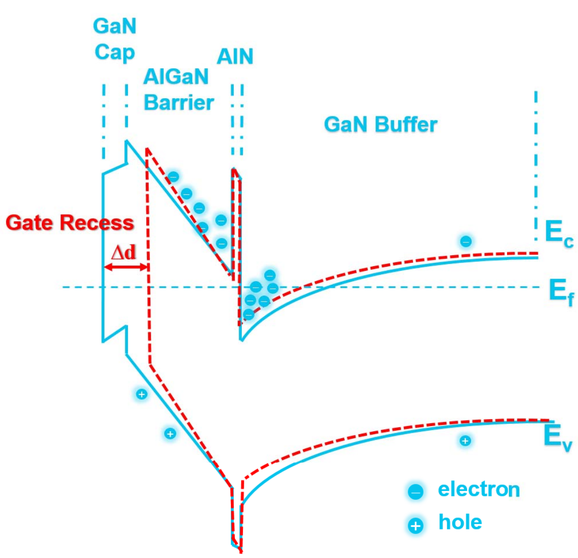

Fig. 6. Band energy diagram of AlGaN/GaN heterostructure before and after gate recess.*Δd*:recess depth.

voltages of 1 V, 2 V, 3 V and 4 V, respectively, are applied to the micro-heater.

Figure 4 shows the transient response characteristic of the gate recessed Pt/AlGaN/GaN heterostructure sensor to 1-40 ppm NO_2/air at the temperature of 300 °C ($V_H = 4$ V). In case of a HEMT used as a gas sensor, one takes advantage of the fact that interface states exist at the interface between the gate materials (Pt or metal oxide et.al.) and semiconductor (GaN or AlGaN layer), which has a direct effect on the space charge region beneath the gate. It is known that NO_2 gets dissociated into NO and oxygen ion (O) when it exposed to Pt surface resulting in high coverage of oxygen on the Pt surface [29], [30].The negatively charged oxygen ions interact capacitively with AlGaN and diffuse via pores or grain boundaries on the Pt surface and get adsorbed at the Pt/AlGaN interface. The presence of these ions generates negative surface potential which compensates the existing positive charges on the AlGaN surface. This results in the depletion of 2DEG causing the current reduction.Similarly, NO molecule having one unpaired electron gets adsorbed as a free radical resulting in negative surface potential, thereby contributing to further reduction in the current. Also, surface donor states are considered to be the source of electrons in the formation of 2DEG [53]. NO_2 molecule capture electrons from the non-ionized donor states, reducing the positive surface charge and thereby decreasing the 2DEG density. Also, the 2DEG mobility may be affected by surface trapping of electrons by NO_2 [54]. The sensor shows stable operation both for increasing [Figure 4(a)] and decreasing [Figure 4(b)] gas concentration. Figure 4(c) shows the enlarged part of the response curve in Figure 4(a) measured at 40 ppm/air of NO_2 to reveal the response and recovery processes of the gas in and out. The response time was found to be 75 seconds, with recovery time about 200 seconds.

TABLE I
COMPARISON OF THE FABRICATED SENSOR AND PRIOR WORKS ON ALGAN/GAN NO_2 SENSORS

Sensor structure	Pt thickness	NO_2 range	Sensitivity (%)	Integrated micro-heater	Recess gate	Response time	Recovery time	Reference
Pt/AlGaN/GaN	10 nm	1-200 ppm	3.5@40 ppm	Yes	Yes	75 s	200 s	This work
Pt/AlGaN/GaN	15 nm	10-800 ppm	1@100 ppm	No	No	~180 s	~200 s	[26]
Pt/AlGaN/GaN	20 nm	0.5-10 ppm	5.5@10 ppm	No	No	120 s	300 s	[57]
Pt/AlGaN/GaN	15-20 nm	100 ppm	12.6@100 ppm	No	No	61 s	~40 s	[58]
AlGaN/GaN	No	60-500 ppb	40000@500 ppb	No	No	~1.5 h	~4 h	[46]
Recess gate/AlGaN/GaN	No	7-100 ppb	30000@100 ppb	No	Yes	~30 min	~5 h	[21]
Recess gate/AlGaN/GaN	No	10-100 ppb	1500@100 ppb	Yes	Yes	~1 min	~5 min	[22]

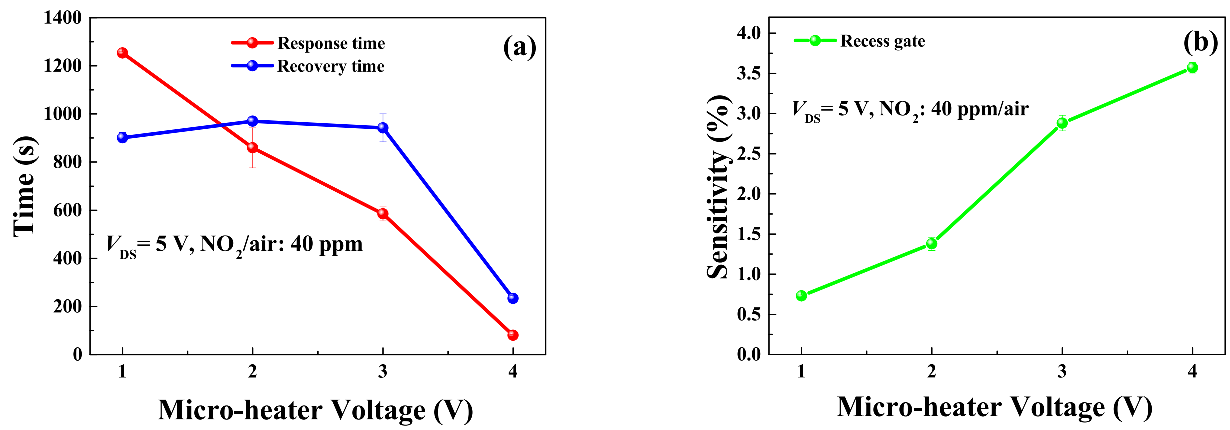

Fig. 7. (a) Response/recovery time and (b) Sensitivity with gate-recess as a function of micro-heater voltage toward 40 ppm NO_2/air gas.

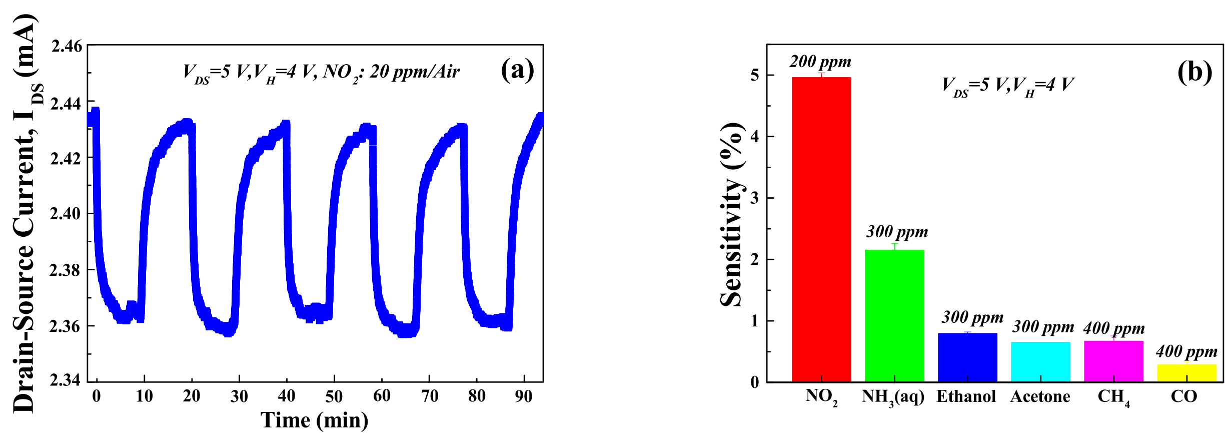

Fig. 8. (a) Repeatability of sensor to 20 ppm NO_2/air at 300 °C and (b) Gas response to NH_3(aq), ethanol, acetone, methane and CO in air at 300 °C.

The use of a gate recess is well known for improving the device characteristics of AlGaN/GaN HEMT. [21] As shown in Figure 5(a) and (b), the current change values (ΔI_{DS}) and sensitivity S (%) of recessed gate AlGaN/GaN heterostructure to 1-200 ppm NO_2/air gas, are about 7.5 to 20 times and 4.5 to 12 times compared to open gate device, respectively. In a thinner AlGaN layer, the reduced surface barrier potential causes fewer surface states to be ionized [53], [55]. The boost in sensing response may be explained by the increase in the number of non-ionized surface states that become available to interact with NO_2. Figure 6 shows the band energy diagram of AlGaN/GaN heterostructure before and after gate recess. The removal cap layer and thinner AlGaN layer on gate area result in lower barrier height [56]. Also, the gate recessed structure with shorter distance between the sensing surface and 2DEG layer make it much easier to modulate by NO_2 molecule. Consequently, the sensitivity and current change of gate recessed AlGaN/GaN can be effectively improved. This could also explain the observed decrease in response time as shown in Figure 5(c) and (d). The response time to 40 ppm NO_2/air decreased from 400 s to 75 s.

As we known, the working temperature have a considerable influence on the sensitivity and response rate of the gas sensor. The response time, recovery time and sensitivity to 40 ppm/air NO_2 as a function of the micro-heater voltage are shown in Figure 7(a) and (b). The response time decreases with increasing micro-heater voltage, which is attributed to faster gas molecule adsorption rate at the surface at higher temperature [26], [28]. The recovery time almost keep stable when the micro-heater voltage increases from 1 V to 3 V. However, it is greatly reduced down to 200 s when the temperature is up to 300 °C ($V_H = 4$ V). The sensitivity (response time) increases from 0.75 % (1250 s) to 3.5% (75 s) to 40 ppm NO_2/air when temperature increases from 60 °C to 300 °C.

The repeatability and selectivity of the sensor measured at $V_H = 4$ V are shown in Figure 8. The drain current response when the NO_2 gas concentration is swept repeatedly from 0 to 20 ppm is demonstrated in Figure 8(a). Figure 8(b) presents the cross-sensitivity performance of the AlGaN/GaN sensor to other gases such as NH_3(aq), ethanol, acetone, CH_4 and CO in air at $V_H = 4$ V.

The comparison of the fabricated sensor and prior works on AlGaN/GaN NO_2 sensors was shown in Table I. The sensors with the gate recess have higher sensitivity and lower detection range. That is because of the trade-off between sensitivity and current change (ΔI). In general, the Pt/AlGaN/GaN sensors have a higher detection range and fast response despite the lower sensitivity. Therefore, the comparison of the sensitivity has reference value at the same layout and operation conditions. Compared to the Pt/AlGaN/GaN, The device in this work is integrated with a micro-heater for in-situ heating for fast response and recovery, and provide higher sensitivity by gate recess method. Overall, the performance results of our device are a first main step towards implementation of AlGaN/GaN heterostructure sensor for NO_2 detection in harsh environment by adopted the combination of the gate recess and catalytic metal or functional materials.

IV. Conclusion

In summary, suspended gate recess Pt/AlGaN/GaN heterostructure NO_2 gas sensor integrated with a micro-heater was fabricated and characterized. Proposed precision two-step gate recess technique dramatically enhances the performance of AlGaN/GaN devices. The sensitivity and current change of AlGaN/GaN heterostructure to 1-200 ppm NO_2/air are increased up 20 times and 12 times compared to conventional gate device respectively with faster response time. The suspended membrane structure and integrated micro-hotplate also improve response time and sensitivity by adjusting the optimum working temperature with low power consumption. The sensitivity (response time) increases from 0.75% (1250 s) to 3.5% (75 s) toward 40 ppm NO_2/air when temperature increases from 60 °C to 300°C. The repeatability and selectivity of sensor are also demonstrated. The characteristics of the here presented suspended gate recess AlGaN/GaN devices integrated with a micro-heater, form an encouraging first step towards the development of a high accuracy and fast response gas sensor in harsh environment.

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