

## Gateless-FET pH Sensor Fabricated on Undoped AlGa<sub>N</sub>/Ga<sub>N</sub> HEMT Structure

(Penderia pH FET tanpa Get difabrikasi di atas Struktur HEMT AlGa<sub>N</sub>/Ga<sub>N</sub> Tidak Didop)

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### ABSTRACT

Gallium nitride with wurtzite crystal structure is a chemically stable semiconductor with high internal spontaneous and piezoelectric polarization, which make it highly suitable materials to create very sensitive and robust sensors for the detection of ions, gases and liquids. Sensing characteristics of an open-gate liquid-phase sensor fabricated on undoped-AlGa<sub>N</sub>/Ga<sub>N</sub> high-electron-mobility-transistor (HEMT) structure in aqueous solution was investigated. In ambient atmosphere, the open-gate undoped AlGa<sub>N</sub>/Ga<sub>N</sub> HEMT clearly showed only the presence of linear region of currents while Si-doped AlGa<sub>N</sub>/Ga<sub>N</sub> showed the linear and saturation regions of currents, very similar to those of gated devices. This seems to show that very low Fermi level pinning by surface states exists in undoped AlGa<sub>N</sub>/Ga<sub>N</sub> sample. In aqueous solution, the typical current-voltage (I-V) characteristics of HEMTs with good gate controllability were observed. The potential of the AlGa<sub>N</sub> surface at the open-gate area is effectively controlled via aqueous solution by Ag/AgCl reference gate electrode. The open-gate undoped AlGa<sub>N</sub>/Ga<sub>N</sub> HEMT structure is capable of stable operation in aqueous electrolytes and exhibit linear sensitivity, and high sensitivity of 1.9 mA/pH or 3.88 mA/mm/pH at drain-source voltage,  $V_{DS} = 5$  V was obtained. Due to large leakage current where it increases with the negative reference gate voltage, the Nernstian's like sensitivity cannot be determined. Suppression of current leakage is likely to improve the device performance. The open-gate undoped-AlGa<sub>N</sub>/Ga<sub>N</sub> structure is expected to be suitable for pH sensing application.

*Keywords:* AlGa<sub>N</sub>/Ga<sub>N</sub>; HEMT; liquid-phase; open-gate structure; pH sensor

### ABSTRAK

Galium nitrat dengan struktur hablur wurtzite adalah semikonduktor kimia yang stabil dengan polarisasi dalaman spontan dan polarisasi piezoelektrik yang tinggi, membuatnya menjadi bahan yang sangat sesuai sebagai penderia yang sensitif dan kuat untuk mengesan ion, gas dan cecair. Pencirian penderia fasa-cecair get terbuka yang difabrikasi di atas struktur AlGa<sub>N</sub>/Ga<sub>N</sub>-tidak didop transistor-pergerakan-elektron-tinggi (HEMT) dalam larutan akuas dikaji. Dalam keadaan ambien, AlGa<sub>N</sub>/Ga<sub>N</sub>-tidak didop HEMT get terbuka menunjukkan dengan jelas hanya ada kawasan arus linear sementara AlGa<sub>N</sub>/Ga<sub>N</sub>- Si-dop menunjukkan kawasan arus linear dan tepu, hampir serupa dengan peranti berget. Hal ini menunjukkan bahawa tahap pengepitan Fermi sangat rendah oleh permukaan dalam sampel AlGa<sub>N</sub>/Ga<sub>N</sub>-tidak didop. Dalam larutan akuas, ciri arus-voltan (I-V) HEMTs dengan pengawalan get yang baik diperhatikan. Keupayaan permukaan AlGa<sub>N</sub> di kawasan get terbuka dikawal secara berkesan melalui larutan akuas dengan elektrod get rujukan Ag/AgCl digunakan. Struktur AlGa<sub>N</sub>/Ga<sub>N</sub>-tidak didop HEMT get terbuka mampu beroperasi stabil di dalam elektrolit akuas dan menunjukkan sensitiviti linear, dan diperoleh sensitiviti yang tinggi bernilai 1.9 mA/pH atau 3.88 mA/mm/pH pada voltan salir-punca,  $V_{DS} = 5$  V. Disebabkan kebocoran arus besar dan ia meningkat dengan voltan rujukan negatif, sensitiviti Nernstian tidak dapat ditentukan. Pengurangan kebocoran arus akan meningkatkan prestasi peranti. Struktur AlGa<sub>N</sub>/Ga<sub>N</sub>-tidak didop HEMT get terbuka dijangkakan sesuai untuk aplikasi penderiaan pH.

*Kata kunci:* AlGa<sub>N</sub>/Ga<sub>N</sub>; fasa cecair; HEMT; penderia pH; struktur get terbuka

### INTRODUCTION

Many semiconductor materials have been tested for their suitability as ion sensors. There is an emerging interest in the use of wide band gap semiconductors as sensitive chemical sensors. Group III-nitrides with wurtzite crystal structure are chemically stable semiconductors with high internal spontaneous and piezoelectric polarization, which

make them highly suitable materials to create very sensitive but robust sensors for the detection of ions, gases and polar liquids (Eickhoff et al. 2003; Stutzmann et al. 2002). Solids with a large band gap such as diamond or gallium nitride are among the prime candidates for a variety of sensor applications, particularly at high temperatures and in harsh environments. AlGa<sub>N</sub>/Ga<sub>N</sub> high-electron-mobility-

transistor (HEMT) structures have been extremely useful for gas and liquid-phase sensor due to primarily three reasons: (1) a high electron sheet carrier concentration channel induced by piezoelectric polarization of the strained AlGa<sub>x</sub>N layer, (2) the carrier concentration which is strongly depends on the ambient (Alifragis et al. 2007; Eickhoff et al. 2003) and (3) an opportunity of on-chip co-integration with signal processing and communication circuit. In addition, sensors fabricated from these wide band gap semiconductors could be readily integrated with solar blind UV detectors or high temperature, high power electronics with wireless communication circuits on the same chip to provide high speed transmission of the data.

For these reasons, GaN-based HEMT structures are versatile structures that may be used for a variety of sensing applications. Due to their low intrinsic carrier concentrations, wide band gap semiconductor sensors based on GaN can be operated at lower current levels than conventional Si-based devices and offer the capability of detection up to 600°C (Baranzhi et al. 1995; Lloyd et al. 2001; Luther et al. 1999; Schalwig et al. 2001, 2002). The pH response of GaN surfaces using ISFET structure was recently reported by Steinhoff et al. (2003). A work on the pH response to n-doped AlGa<sub>x</sub>N surfaces was recently reported by Kokawa et al. (2006). However, no work on the pH response to undoped-AlGa<sub>x</sub>N surfaces was done, and mechanism of pH response to such surfaces are not understood yet.

The purpose of this study is to investigate pH-sensing characteristics of open-gate undoped AlGa<sub>x</sub>N/GaN HEMT structures. We have investigated the basic transistor characteristics and liquid-phase sensing capability of open-gate devices with bare undoped-AlGa<sub>x</sub>N surfaces in aqueous solutions. The results obtained seem to open up the feasibility of cointegration with AlGa<sub>x</sub>N/GaN HEMT circuits for sensor network applications.

Early reports have suggested that the pH sensitivity of gateless AlGa<sub>x</sub>N/GaN heterostructure transistors to electrolyte solutions results from the presence of metal oxide surface in gate region such as native oxide (Eickhoff et al. 2001; Schalwig et al. 2002). Steinhoff et al. (2003) found a linear response to changes in the pH range of 2–12 for ungated GaN-based transistor structures and suggested that the native metal oxide on the semiconductor surface is responsible on these characteristics. The observed pH sensitivities can be explained in terms of the site binding model which has been suggested by Yates et al. (1974) and has been further developed and applied to ion-sensitive field-effect transistors (ISFETs) later (Bousse & Bergveld 1983; Siu & Cobbold 1979). According to this model, amphoteric hydroxyl groups which are dependent on the ambient pH lead to a pH dependent net surface charge, thereby cause an additional voltage drop at the solid/liquid interface.

In AlGa<sub>x</sub>N/GaN HEMT structure, the sheet carrier concentration is formed by the two-dimensional electron gas (2DEG) at AlGa<sub>x</sub>N/GaN interface induced by spontaneous

and piezoelectric polarization, even in undoped AlGa<sub>x</sub>N. A 2DEG layer is formed because of the discontinuity in the areal density of microscopic dipoles in the wurtzite crystal lattice at the heterointerface. The 2DEG is separated from the free surface by the insulating AlGa<sub>x</sub>N barrier layer. The conduction band offset at the heterointerface plus the polarization-induced internal electric field gives rise to a strong confinement of the 2DEG within approximately 2 nm, despite of the large carrier density (Stutzmann et al. 2002).

The sensing mechanism for solution of these materials is completed by the action between polarization-induced positive surface charge and ion in electrolyte, and then, this change on the gate region of AlGa<sub>x</sub>N/GaN HEMTs affects the surface charges of the device. The change in the surface charge is transduced into a change in the concentration of the 2DEG causing a change in drain-source current. As a result, we can measure the pH of the solution with the related change in current.

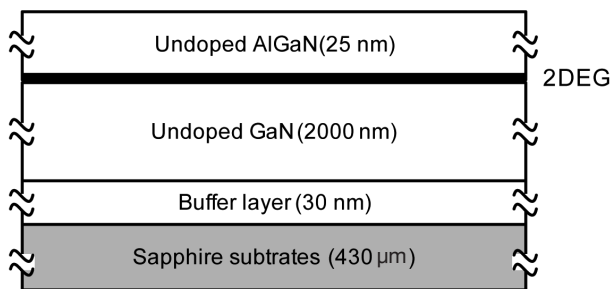
#### MATERIAL, DEVICE STRUCTURE AND FABRICATION

AlGa<sub>x</sub>N/GaN heterojunction has been shown to form a potential well and a 2DEG at the lower heterointerface. These structures are well known for possessing high electron mobility in the 2DEG channel, highest sheet carrier concentration among III-V material system, high saturation velocity, high breakdown voltage, and thermal stability. When wide band Al<sub>x</sub>Ga<sub>1-x</sub>N and narrow band GaN are brought into contact, thermal equilibrium align their respective Fermi levels,  $E_F$  that both conduction band,  $E_c$  and valence band,  $E_v$  are bent and cause the GaN conduction band at the interface to drop below  $E_F$ . Free electrons will fill the triangular well and form 2DEG. From another viewpoint, as described by Ambacher et al. (2000), 2DEG is the compensation to a fixed sheet charge induced by both spontaneous polarization and piezoelectric or strain-induced polarization.

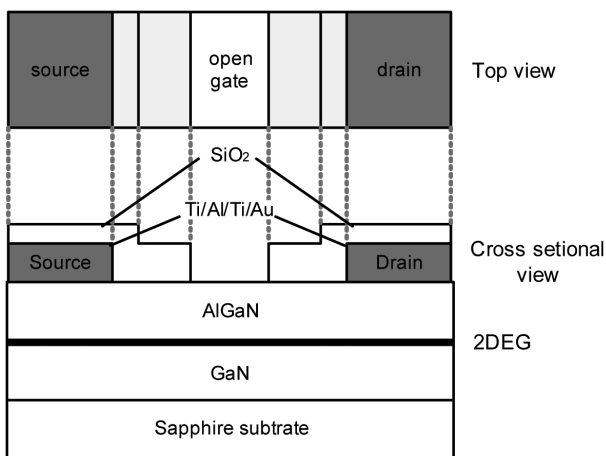
In addition to the pyroelectric and piezoelectric material properties of AlGa<sub>x</sub>N thin films, which allow the fabrication of HEMT structures with a polarization induced 2DEG, the heteroepitaxial growth of the group III-nitrides on electrically insulating sapphire substrates allows the application of simple planar device structures. In addition, the low pyroelectric coefficients combined with the wide band gap of more than 3.4 eV are advantageous for sensor devices based on AlGa<sub>x</sub>N/GaN heterostructure field-effect transistors, which display high thermal stability (Eickhoff et al. 2003).

In this study, we propose the use of undoped-AlGa<sub>x</sub>N/GaN HEMT structure for pH sensing device. The expected advantages of using undoped-AlGa<sub>x</sub>N/GaN as compared with doped structures are lower gate leakage current, lower pinch-off voltage and less noise due to the no donor in AlGa<sub>x</sub>N. These are the reasons why many groups prefer non-modulation doped nitride HEMT structures (Ambacher et al. 2000; Rizzi & Luth 2002).

Figure 1a shows our proposed material structure. The AlGaIn/GaN samples are grown by metal organic chemical vapor deposition (MOCVD) on 430  $\mu\text{m}$  c-plane sapphire substrates. The growth of the group III-nitrides on electrically insulating sapphire substrates allows the application of simple planar device structures. Furthermore, the thermal expansion coefficient of the sapphire substrate is close to those of aluminium oxide or aluminium nitride ceramics frequently used as packaging materials for high temperature sensors. Thereby the complicated packaging technologies for high temperature sensors can be simplified. As shown in Figure 1a, the epitaxial structure consisted of a 30 nm-thick GaN buffer layer, a 2  $\mu\text{m}$ -thick undoped GaN layer and 25 nm-thick undoped-AlGaIn barrier layer with an Al composition of 25%. The electron mobility and density of the two dimensional electron gas (2DEG) were 1860  $\text{cm}^2/\text{Vsec}$  and  $6.61 \times 10^{12} \text{ cm}^{-2}$ , respectively, at room temperature. The GaN buffer is necessary to achieve a uniform Ga face polarity of the III-nitride epilayer across the entire substrate and also improves the structural quality of the following GaN layer. As discussed above due to the macroscopic polarization in the nitride heterostructure, a 2DEG with a high electron density forms at the AlGaIn/GaN heterointerface created by the deposition of a thin AlGaIn barrier layer.



(a)



(b)

FIGURE 1. (a) Schematic of material layer structure (cross-sectional view) and (b) schematic device structure (top and cross-sectional view)

The schematic of device structure is shown in Figure 1b. The device fabrication process started with 100 nm-thick SiO<sub>2</sub> deposition using plasma-enhanced chemical vapor deposition (PECVD) at 280°C with a SiH<sub>4</sub>/NH<sub>3</sub>/He gas system. Then, isolation patterning using inductive-coupled plasma(ICP)-assisted reactive ion beam etching with a Cl-based gas system consisting of BCl<sub>3</sub>, Cl<sub>2</sub> and Ar. The etching pressure is 5 mTorr and the etching rate is around 0.1  $\mu\text{m}/\text{min}$ . The drain and source electrodes were formed by deposition of Ti/Al/Ti/Au (20 nm/50 nm/20 nm/150 nm) multilayers and annealed at 850°C for 30 seconds under a flowing of N<sub>2</sub> ambient by rapid thermal annealing system. Although the present device is a two-terminal device, electrodes are called source and drain electrodes in this article so that the results on the gateless device can be correlated with behavior of the gated device. The drain will be positively biased, and the voltage and current are called the drain voltage,  $V_{DS}$ , and drain current,  $I_{DS}$ , respectively. Next, the device surface was covered with SiO<sub>2</sub> film to a thickness of 300 nm using PECVD to prevent a chemical reaction between electrolyte and metal electrodes. Finally, the open-gate area, width of 490  $\mu\text{m}$  and length of 40  $\mu\text{m}$ , was defined through standard photolithography and wet etching processes in a buffered HF solution. The fabricated device is shown in Figure 2.

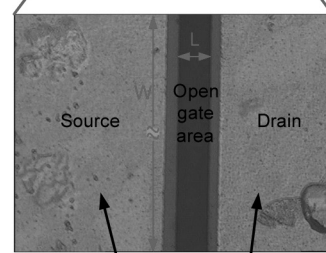
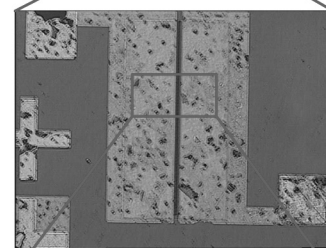
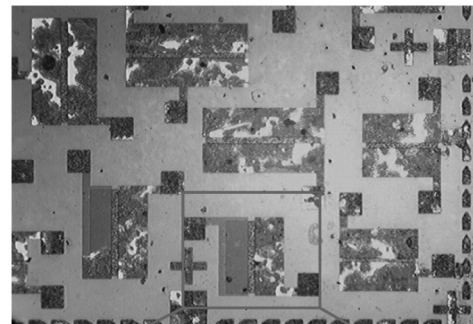
SiO<sub>2</sub> layer on top of device

FIGURE 2. Photo of fabricated device (top view)

## RESULTS AND DISCUSSIONS

Figure 3a shows the sample holder. The sample was mounted using photoresist on a PCB strip having a contact pad for the sample and conductor strips for source and drain connection. Wire bonding is carried out as shown in the diagram. Photoresist is applied carefully on the wires and contact region as well as on the sample sides, keeping the open-gate region exposed for interaction with the electrolyte.

Figure 3b shows a simple electrochemical system and a measurement circuit consisting of three source measure units (Keithley 236 SMU) and lab view control system. The gate bias is applied from the source measure unit to the electrolyte/AlGaN interface at the open-gate area via a Ag/AgCl electrode. For pH-sensing measurements, we prepared a mixed solution with HCl and NaOH in de-ionized (DI) water. The pH values in solutions are measured using a digital pH meter (Fisher Acumet AB15) after calibration with standard reference solution. All measurements in solutions were performed at room temperature (25°C) under light condition.

The typical DC current-voltage ( $I$ - $V$ ) characteristics of the open-gate undoped-AlGaN/GaN and Si-doped AlGaN/GaN HEMT structure in air-exposed condition under light environment at room temperature is shown in Figure 4. Here, the data of open-gate Si-doped AlGaN/GaN HEMT structure is also presented as comparison. The epitaxial structure of Si-doped AlGaN/GaN HEMT structure consisted of a 30 nm-thick GaN buffer layer,

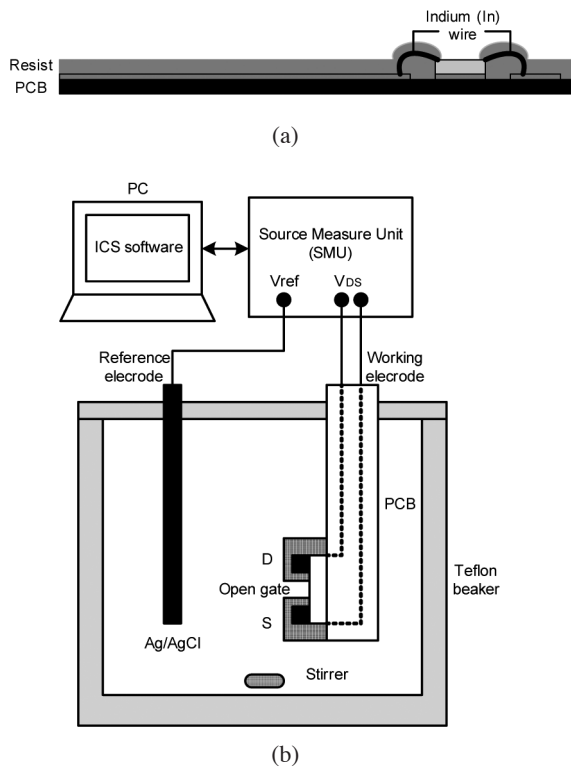


FIGURE 3. (a) Schematic of sample holder and (b) schematic of the electrochemical system and the measurement circuit

a 2  $\mu\text{m}$ -thick undoped GaN layer, a 3 nm-thick undoped AlGaN spacer layer, 15 nm-thick Si-doped-AlGaN barrier layer and 10 nm-thick undoped-AlGaN cap layer with an Al composition of 25%. The electron mobility and density of the 2DEG are 221  $\text{cm}^2/\text{Vsec}$  and  $2.554 \times 10^{13} \text{ cm}^{-2}$ , respectively at room temperature.

It is clearly seen that for all tested undoped-AlGaN/GaN samples, the presence of the linear and saturation regions of currents are not observed. On the other hand, in spite of an ungated structure, the curves of most tested Si-doped AlGaN/GaN samples showed the presence of linear and saturation regions of currents similar to those of the gated device. The possible mechanism of the appearance of current saturation and pinch-off behavior was proposed by Hasegawa et al. (2003). They negated the possibility of velocity saturation because the average electric field strength is too small to expect significant velocity saturation effect in such a long gate device. The proposed interpretation is that it is due to the presence of strong Fermi level pinning by surface states which tends to fix the surface potential at a particular position and makes the entire surface behave like a virtual gate (Hasegawa et al. 2003). In fact, they have shown that the data could be reasonably well fitted to the theoretical DC  $I_{DS}$ - $V_{DS}$  curves based on the gradual channel approximation. From this result, it can be simply said that the undoped AlGaN/GaN structure may have produced very low Fermi level pinning by surface states. It also may be due to large open-gate dimension which induced parasitic resistance. Thus, the drain current does not reach the saturation even up to 10 V.

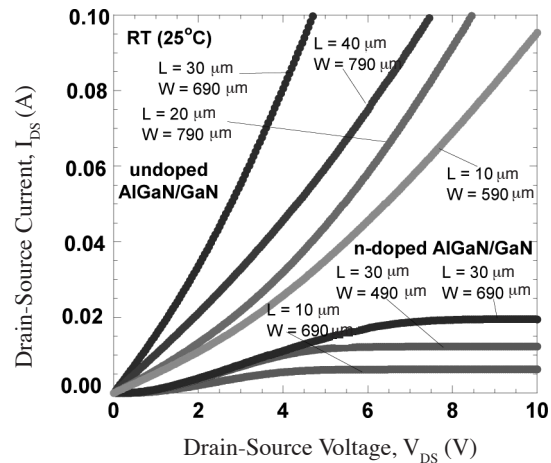
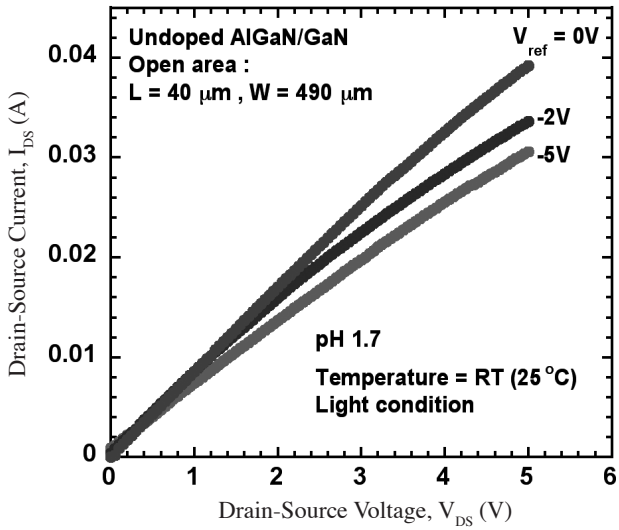


FIGURE 4. Typical  $I_{DS}$ - $V_{DS}$  characteristics of the open-gate HEMT in air condition

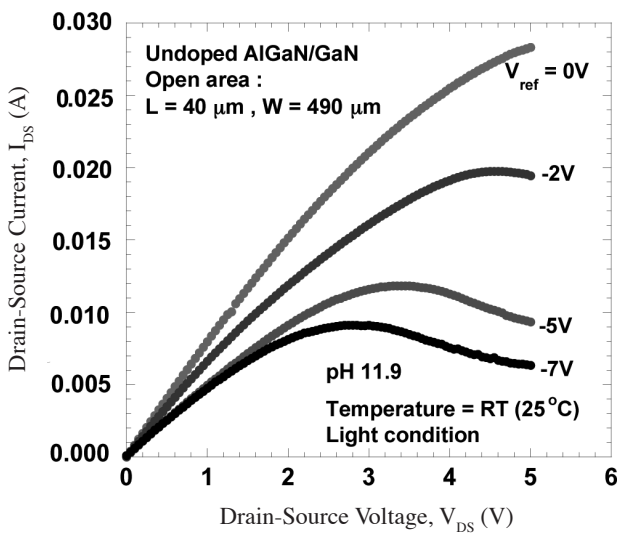
Figures 5a and 5b show the typical  $I_{DS}$ - $V_{DS}$  characteristics of the open-gate undoped AlGaN/GaN HEMT in a mixed solution of HCl and NaOH in water with pH value of 1.7 and 11.9, respectively. The measurement was done at room temperature in room's light environment. It can be seen in Figure 5a that the pinch-off behavior is hard to be achieved in low pH solution compared to high

pH solution. In addition, it is observed that large leakage current exists during measurement in low pH solution compared to high pH solution, and will be presented in the following figure. Despite the existence of leakage current, the device showed the conventional FET behavior with good gate controllability.

The  $I_{DS}$ - $V_{DS}$  characteristics as a function of pH values is shown in Figure 6a. The drain-source current decreases with the pH values as expected. Figure 6b shows the drain-source current measured under  $V_{DS} = 1$  V and 5 V, and reference gate voltage,  $V_{ref} = -5$  V. As expected, it clearly shows that the drain-source current decrease with the pH value. We obtained a large current change,  $\sim 1.9$  mA/pH or  $\sim 3.88$  mA/mm/pH at  $V_{DS} = 5$  V because of high mobility and 2DEG density of the undoped-AlGaIn/GaN HEMT. In addition, a linear sensitivity is clearly observed,

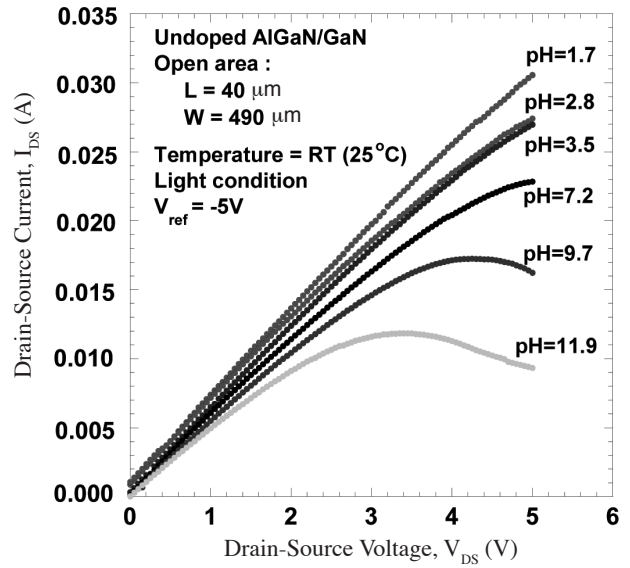


(a)

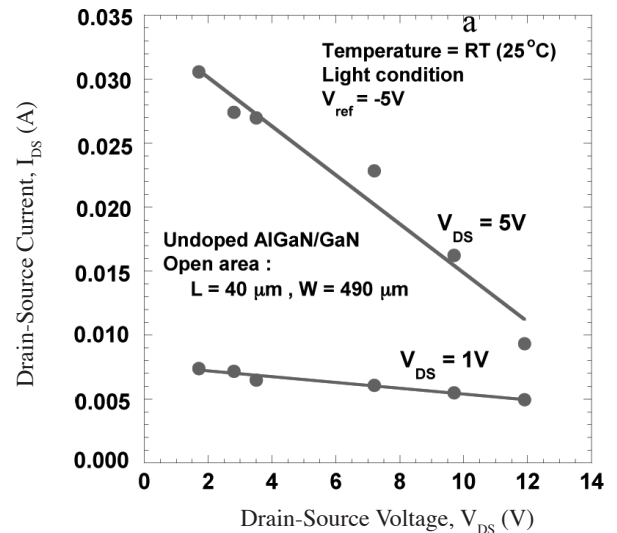


(b)

FIGURE 5. Typical  $I_{DS}$ - $V_{DS}$  characteristics of the undoped open-gate HEMT in (a) pH of 1.7 and (b) pH of 11.9



(a)



(b)

FIGURE 6. (a)  $I_{DS}$ - $V_{DS}$  characteristics as a function of pH values and (b) measured  $I_{DS}$  under  $V_{DS} = 1$  V and 5 V and  $V_{ref} = -5$  V

reflecting systematic change in potential at the AlGaIn surface in the both linear and saturated bias regions. Thus, it seems to show that undoped AlGaIn/GaN open-gate HEMT devices are capable of stable operation and exhibit linear sensitivity. The exact mechanism of how these changes occur is still unknown but similar tendency is also commonly observed in other reports. However it can be explained using electrolyte-insulator interfaces ( $\text{SiO}_2$ ,  $\text{SiN}_x$ ,  $\text{Al}_2\text{O}_3$ , AlN, etc.) in Si-based ion-sensitive FETs, where a site-binding model is generally accepted (Bousse et al. 1983; Esahi et al. 1978; Yates et al. 1974). According to this model, hydroxyl groups (MOH: M represents Si or metals) are formed at insulator surfaces in contact with aqueous solutions, and can be dissociate to or combine with  $\text{H}^+$ , depending on the  $\text{H}^+$  concentration and the equilibrium constants for the relevant reactions, as follows:



When  $\text{H}^+$  concentration decreases in the solution, the right-direction reaction in the equilibrium Eq. (1) becomes dominant, resulting in negative charges at the insulator surfaces due to deprotonized hydroxyls ( $\text{MO}^-$ ). On the other hand, the increase of  $\text{H}^+$  can induce positive charges at the surfaces due to protonized hydroxyls ( $\text{MOH}_2^+$ ), represented by Eq. (2). This leads to pH dependent net charge at the insulator surfaces, and the liquid-solid interfacial potential thereby follows the Nernst equation.

Figure 7 shows the the drain-source current at  $V_{DS} = 0$  V as a function of the reference gate voltage. Large drain-source current presents at low pH value and it increased with the negative reference gate voltage although no drain-source voltage is applied. Figure 8a shows the gate-leakage characteristics of the open-gate undoped AlGaIn/GaN HEMT as a function of pH value. For comparison, the gate-leakage characteristics of the open-gate n-doped AlGaIn/GaN HEMT in DI water and a typical  $I_{GS} - V_{GS}$  curve of the Ni/Au Schottky-gate HEMT from Kokawa et al. (2006), also shown together. The fabricated device shows large leakage current and it increases with the decrease of pH value, which is probably due to the high carrier concentration (electrons and holes) in the electrochemical system exist under room's light condition. Figure 8b shows the changes of reference gate-leakage current under various pH value at  $V_{DS} = 0$  V and  $V_{ref} = -5$  V. The reference gate current shows drastic reduction from pH of 1.7 to 7.2, but increased from 7.2 to 12 (Figure 8b). This results show that the leakage-current depends strongly on the concentration of  $\text{H}^+$  ions in the electrolyte. The cause of the presence of large leakage-current is not understood yet. Due to large leakage current where it increases with the negative reference gate voltage, the Nernstian's like sensitivity cannot be determined as what normally reported by the other researchers.

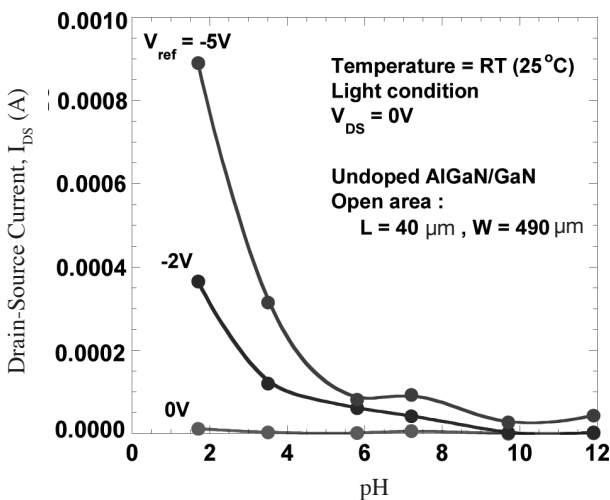
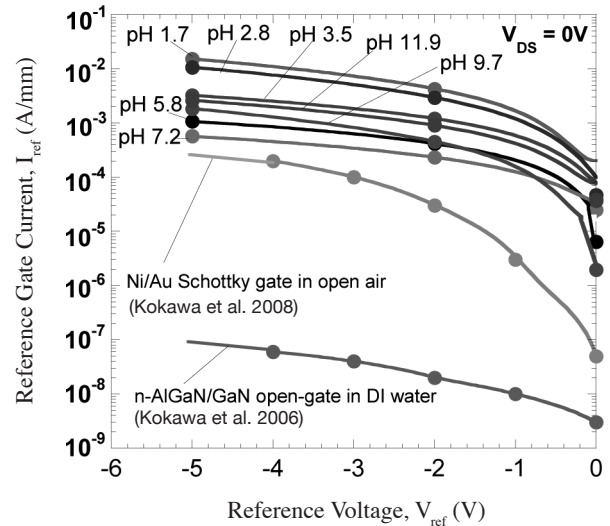
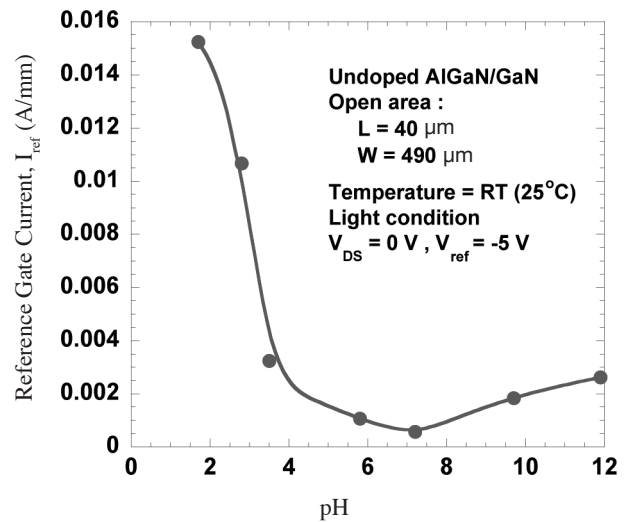


FIGURE 7. Drain-source current as a function of the reference gate voltage at  $V_{DS} = 0$  V



(a)



(b)

FIGURE 8. (a) Gate-leakage characteristics of the open-gate undoped AlGaIn/GaN HEMT at  $V_{DS} = 0$  V and (b) changes of gate leakage current at various pH value

Because the sensor was wetted by the liquid electrolyte, it is critically important to isolate its electrical contacts for source and drain from the test liquid sample for reliable measurements. The adhesion of the photoresist is a major issue because with repeated use the photoresist may wear off, exposing the wires and pads, and causing device malfunctioning or gate-leakage.

## CONCLUSIONS

This investigation shows that undoped-AlGaIn/GaN open-gate HEMT devices is capable of stable operation in aqueous electrolytes and exhibit linear sensitivity. High sensitivity of 1.9 mA/pH or 3.88 mA/mm/pH at  $V_{DS} = 5$  V was obtained. However, due to large leakage current where it increases with the negative reference gate voltage, the Nernstian's

like sensitivity cannot be determined. Further optimization of the open-gate region is likely to improve the device performance in term of current leakage suppression. The fabricated open-gate undoped-AlGaIn/GaN structure is expected to be suitable for pH sensing application.

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