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Open-Gate Liquid-Phase Sensor Fabricated on Undoped-AlGaN/GaN HEMT Structure

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Abstract: Sensing responses of an open-gate liquid-phase sensor fabricated on undoped-AlGaN/GaN high-electron-mobility-transistor (HEMT) structure are investigated in aqueous solution. In air-exposed ambient, the open-gate undoped AlGaN/GaN HEMT shows the only presence of linear region of currents while Si-doped AlGaN/GaN shows 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 AlGaN/GaN sample compared to Si-doped sample. In aqueous solution, the typical current-voltage (I-V) characteristics of HEMTs with reasonably good gate controllability are observed. The potential of the AlGaN surface at the open-gate area is effectively controlled via aqueous solution by Ag/AgCl gate electrode. The open-gate undoped AlGaN/GaN HEMT structure is capable of distinguishing pH level in aqueous electrolytes and exhibits linear sensitivity, where high sensitivity of 1.9 mA/pH or 3.88 mA/mm/pH at drain-source voltage, V_{DS} = 5 V is obtained. Due to large leakage current where it increases with the negative gate voltage, the Nernstian's like sensitivity cannot be determined as what commonly reported in literatures. This large leakage current may be caused by the technical factors rather than the characteristics of the devices themselves. Suprisingly, although there is imperfection in the device preparation, the fabricated devices work very well in distinguishing the pH levels. Suppression of current leakage is likely to improve the device performance. The fabricated open-gate undoped-AlGaN/GaN structure is expected to be suitable for pH sensing application.

Key words: AlGaN/GaN, pH sensor, open-gate structure, liquid-phase, HEMT

INTRODUCTION

Many semiconductor materials have been tested for their suitability as ion sensors; especially 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 (Stutzmann *et al*., 2002; Eickhoff *et al*., 2003). Solids with a large band gap such as diamond or gallium nitride (GaN) are among the prime candidates for a variety of sensor applications, particularly at high temperatures and in harsh environments. AlGaN/GaN 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 AlGaN layer, (2) the carrier concentration which is strongly depends on the ambient (Eickhoff *et al*., 2003; Alifragis *et al*., 2007) 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.

The pH response of GaN surfaces using ion-senstive field-effect transistor (ISFET) structure is recently reported by Steinhoff *et al*. (2003). A work on the pH response to n-doped AlGaN surfaces is recently reported by Kokawa *et al*. (2006). However, no study on the pH response to undoped-AlGaN surfaces is done and

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mechanism of pH response to such surfaces is not understood yet. The expected advantages of using undoped-AlGaN/GaN as compared with doped structures are lower gate leakage current, lower pinch-off voltage and less noise due to the no donor in AlGaN. These are the reasons why many groups prefer non-modulation doped nitride HEMT structures (Ambacher *et al*., 2000; Rizzi and Luth, 2002).

The possible sensing mechanism for these materials can be related to the action between polarization-induced positive surface charges and ions in electrolyte on the gate region of AlGaN/GaN HEMT and then, this will affect the surface charges of the device. The change in the surface charge will result in the 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.

The study presents the investigation on pH-sensing characteristics of open-gate undoped AlGaN/GaN HEMT structures. We have investigated the basic transistor characteristics and liquid-phase sensing capability of open-gate devices with unpassivated undoped-AlGaN surfaces in aqueous solutions. The results obtained seem to open up the feasibility of cointegration with AlGaN/GaN HEMT circuits for sensor network applications.

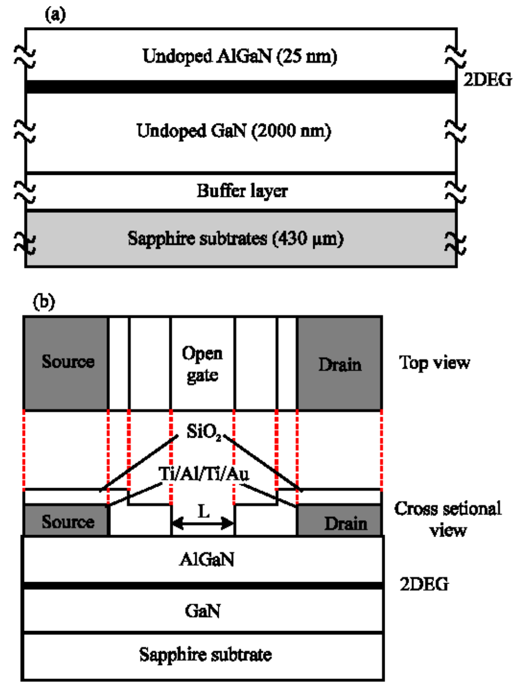

Fig. 1: (a) Schematic of material layer structure (cross-sectional view) and (b) schematic device structure (top and cross-sectional view)

EXPERIMENTAL

Sample preparation: Figure 1a shows proposed material structure. The AlGaN/GaN samples are grown by metal organic chemical vapour deposition (MOCVD) on 430 µm 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 Fig. 1a, the epitaxial structure consisted of a 30-nm-thick GaN buffer layer, a 2-µm-thick undoped GaN layer and 25-nm-thick undoped-AlGaN barrier layer with an Al composition of 25%. The electron mobility and density of the two-dimensional electron gas (2DEG) are 1860 cm^2/Vsec and 6.61×10^{12} cm^{-2}, respectively, at room temperature. The GaN buffer is basically 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.

The schematic of device structure is shown in Fig. 1b. The device fabrication process starts with 100-nm-thick SiO_2 deposition using plasma-enhanced chemical vapour deposition (PECVD) at 280°C with a SiH_4/NH_3/He gas system. This SiO_2 dielectric layer plays a role as a mask for mesa patterning in the following dry etching process. This dielectric layer is removed out after that. A mesa patterning is formed using inductive-coupled plasma (ICP) assisted reactive ion beam etching with a Cl-based gas system consisting of BCl_3, Cl_2 and Ar. The etching pressure is 5 mTorr and the etching rate is around 0.1 µm min^{-1}. The drain and source electrodes are formed by deposition of Ti/Al/Ti/Au (20 nm/50 nm/20 nm/150 nm) multilayers, annealing process at 850°C for 30 sec under a flowing of N_2 ambient by rapid thermal annealing system and lift off process. 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-source voltage, V_{DS} and drain-source current, I_{DS}, respectively. Next, the device surface is covered with 300-nm-thick SiO_2 film using PECVD to prevent a chemical reaction between electrolyte and metal electrodes. Finally, the open-gate area, width, W of 490 µm and length, L of 40 µm, is defined through standard photolithography and wet etching processes in a buffered HF solution. The fabricated device is shown in Fig. 2.

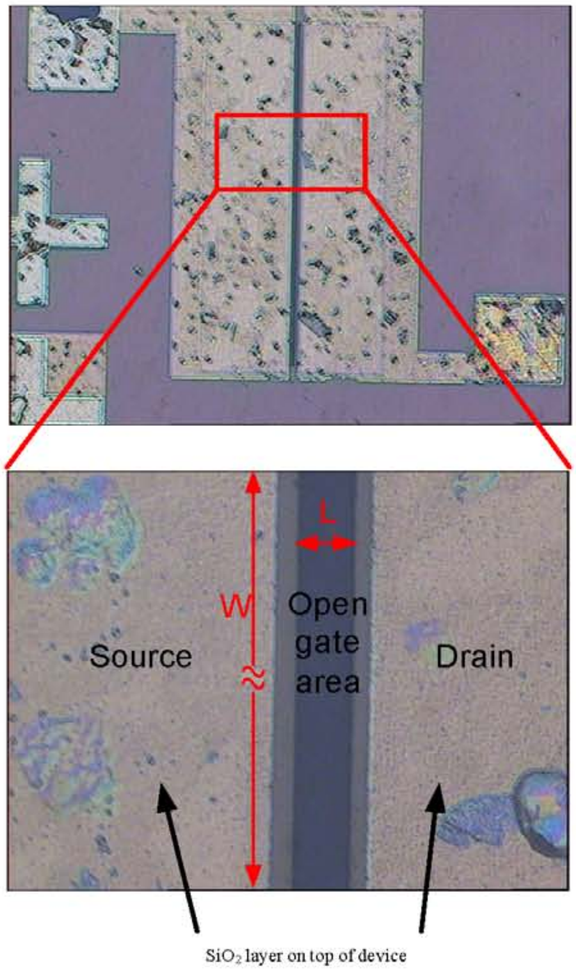

Fig. 2: Photo of fabricated device (top view)

Figure 3a shows the sample holder. The sample is mounted using photoresist on a printed circuit board (PCB) having a contact pad for the sample and conductor strips for source and drain connection. Wire bonding is made using In wire as shown in the diagram. Photoresist is applied carefully on the wires and all metal contact area, 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 a 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 are performed at room temperature (25°C) under light condition.

pH measurement: 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 Fig. 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, a 2-μm-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 for the Si-doped sample are 221 $cm^2/Vsec$ and 2.554×10^{13} cm^{-2}, respectively, at room temperature.

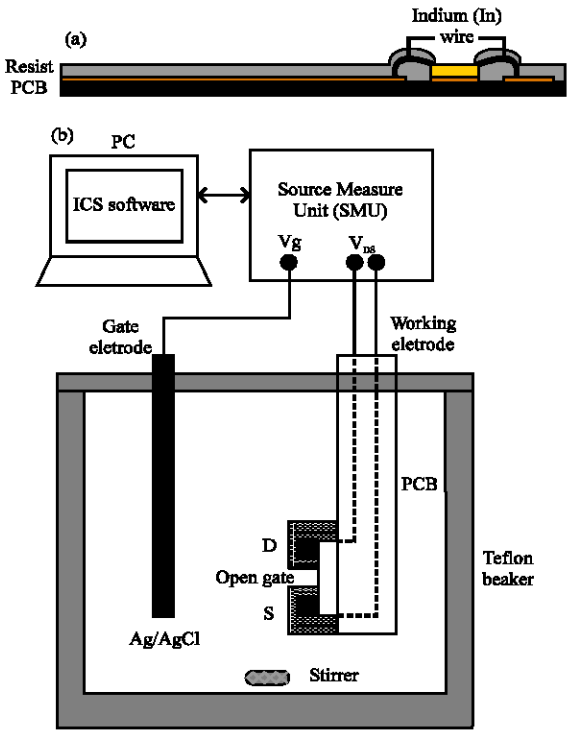

Fig. 3: (a) Schematic of sample holder and (b) schematic of the electrochemical system and the measurement circuit

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 the tested Si-doped AlGaN/GaN samples show the presence of the linear and saturation regions of currents similarly to those of the gated device. The possible mechanism of the appearance of current saturation and pinch-off behavior is proposed by Hasegawa *et al.* (2003). They deny 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 intepretation 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 produce very low Fermi level pinning by surface states. It also may due to large open-gate dimension which induces parasitic resistance. Thus, the drain current does not reach the saturation even up to 10 V.

Figure 5a and b 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 is done at room temperature in room's light environment. It can be seen in Fig. 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. This large leakage current may be caused by the technical factors rather than the characteristics of the devices themselves. Despite of the existence of leakage current, the device shows the conventional FET behavior with reasonably good gate controllability.

The I_{DS}-V_{DS} characteristics as a function of pH values is shown in Fig. 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 gate voltage, V_g = -5 V. As expected, it clearly shows that the drain-source current decrease with the pH value. We obtained a large current change, ~ 1.9 mA/pH or ~ 3.88 mA/mm/pH at V_{DS} = 5 V because of high mobility and 2DEG density of the undoped-AlGaN/GaN HEMT. In addition, a linear sensitivity is clearly observed, reflecting systematic change in potential at the AlGaN surface in the both linear and saturated bias regions. Thus, it seems to show that undoped AlGaN/GaN open-gate HEMT devices are capable of distinguishing pH level 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 (Kokawa *et al.*, 2006). But it can be explained using electrolyte-insulator interfaces (SiO_2, SiN_x, Al_2O_3, AlN, etc.) in Si-based ion-sensitive FETs, where a site-binding model is generally accepted (Yates *et al.*, 1974; Bousse *et al.*, 1983; Esahi and Matsuo, 1978). 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 H^+, depending on the H^+ concentration and the equilibrium constants for the relevant reactions, as follows:

$$MOH \rightleftarrows MO^- + H^+ \quad (1)$$

$$MOH + H^+ \rightleftarrows MOH_2^+ \quad (2)$$

When H^+ concentration decreases in solution, the right-direction reaction in the equilibrium Eq. 1 becomes dominant, resulting the negative charges at the insulator surfaces due to deproonized hydroxyls (MO^-). On the other hand, the increase of H^+ can induce positive

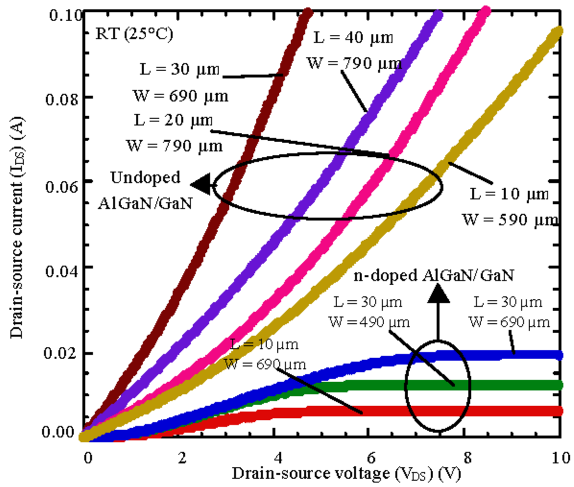

Fig. 4: Typical I_{DS}-V_{DS} characteristics of the open gate HEMT in air condition

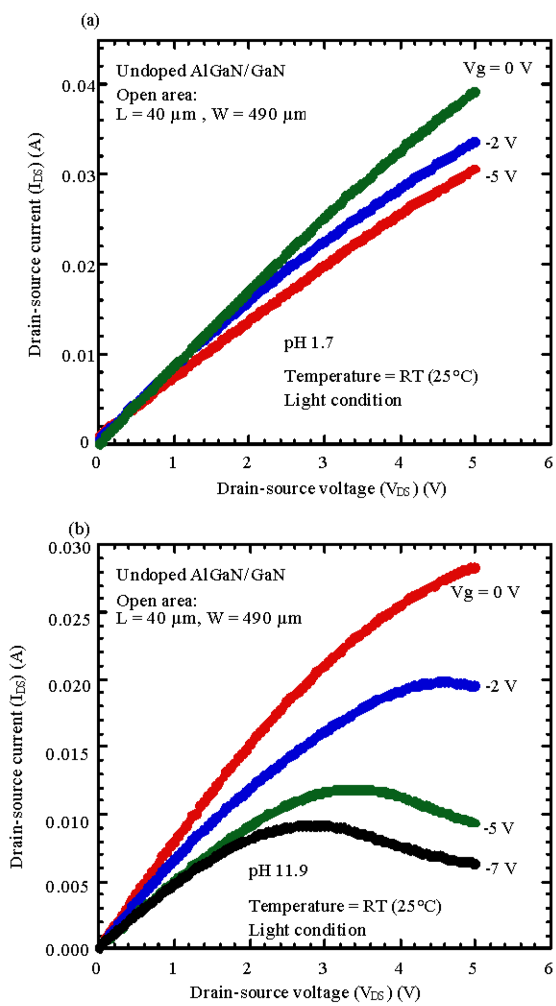

Fig. 5: Typical I_{DS}-V_{DS} characteristics of the undoped open gate HEMT in (a) pH of 1.7 and (b) pH of 11.9

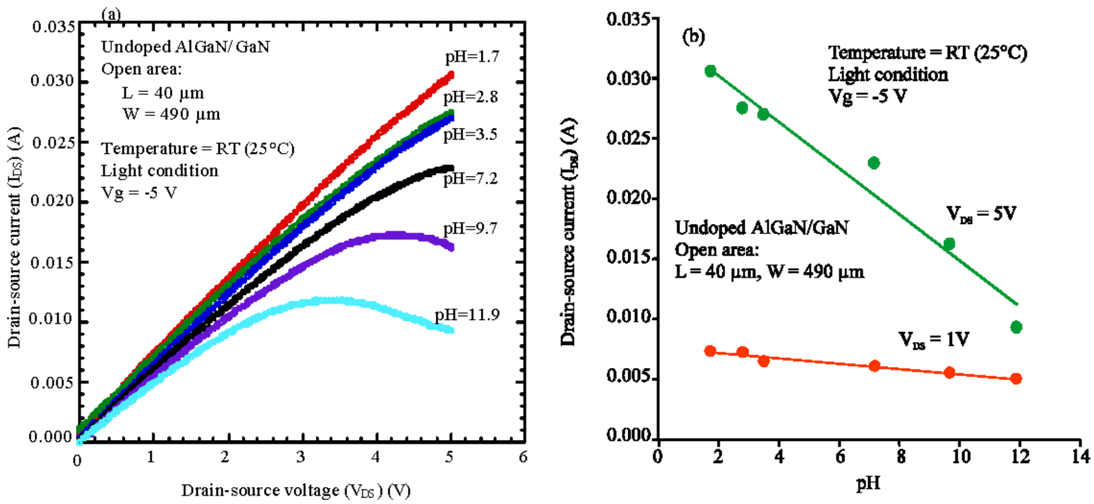

Fig. 6: (a) I_{DS}-V_{DS} characteristics as a function of pH values and (b) measured I_{DS} under V_{DS} = 1 V and 5 V and Vg = -5 V

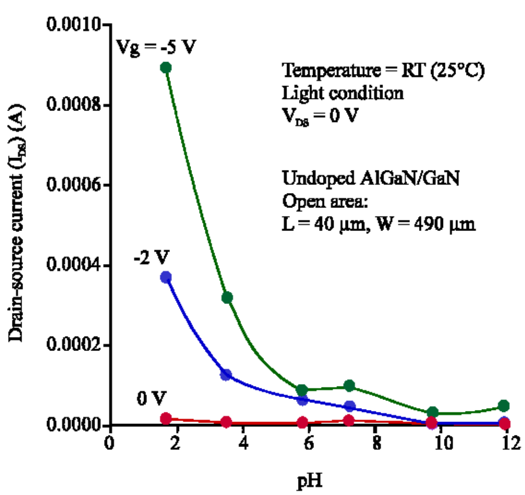

Fig. 7: Drain-source current as a function of the reference gate voltage at V_{DS} = 0 V

charges at the surfaces due to protonized hydroxyls (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 drain-source current at V_{DS} = 0 V as a function of the gate voltage. Large drain-source current presents at low pH value and it increases with the negative gate voltage although no drain-source voltage is applied. Figure 8a shows the gate-leakage characteristics of the open-gate undoped AlGaN/GaN HEMT as a function of pH value. For comparison, the gate-leakage characteristics of the open-gate n-doped AlGaN/GaN HEMT in de-ionized water and a typical I_{GS}-V_{GS} curve of the Ni/Au Schottky-gate HEMT in air (Kokawa *et al*., 2006) are also shown together. The fabricated device shows large leakage current and it increases with the decrease of pH value, which probably due to lots of carriers (electrons and holes) in the electrochemical system exist under room's light condition.

Figure 8b shows the magnitudes of gate-leakage current under various pH value at V_{DS} = 0 V and V_g = - 5 V. As shown in Fig. 8b, gate current shows drastic reduction from pH of 1.7 to 7.2, but increases from 7.2 to 12. This results show that the leakage-current depends strongly on the concentration of H^+ ions in the electrolyte. We believe that this large leakage current may be caused by the technical factors rather than the characteristics of the devices themselves. Because the sensor is 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. Due to large leakage current where it increases with the negative gate voltage, the Nernstian's like sensitivity cannot be determined as what normally reported by the other researchers (Kokawa *et al*., 2006). Although, there is imperfection in the device preparation, the fabricated devices work very well in distinguishing the pH levels. Therefore, the fabricated open-gate undoped-AlGaN/GaN structure is expected to be suitable for pH sensing application.

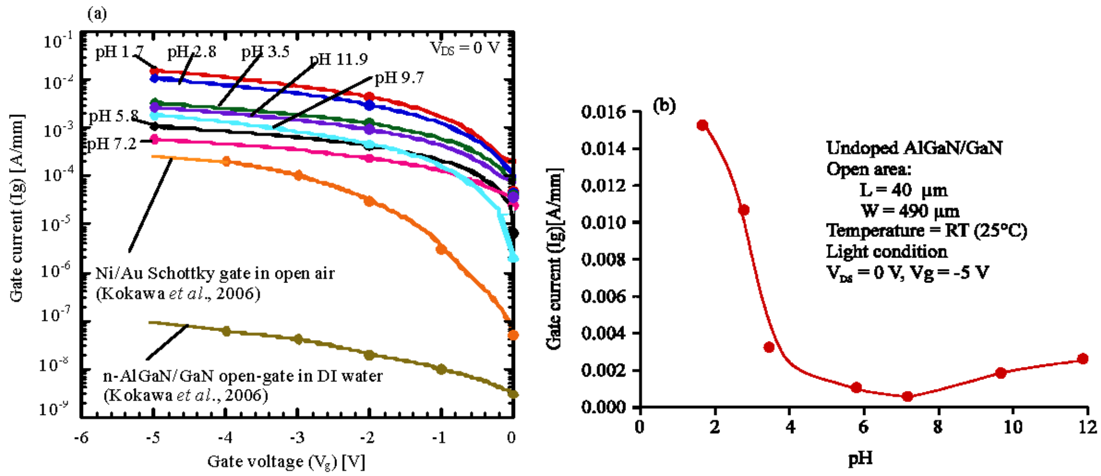

Fig. 8: (a) Gate-leakage characteristics of the open gate undoped AlGaN/GaN HEMT at V_{DS} = 0 V and (b) Changes of gate leakage current at various pH value

CONCLUSIONS

This investigation shows that undoped-AlGaN/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 is obtained. However, due to large leakage current where it increases with the negative reference gate voltage, the Nernstian's like sensitivity can not determined. Further improvement on device preparation is likely to improve the device performance in term of current leakage suppression. The fabricated open-gate undoped-AlGaN/GaN structure is expected to be suitable for pH sensing application.

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