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Fabrication and Characterization of GaN-Based Two Terminal Devices for Liquid Sensing

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Abstract Gallium Nitride (GaN) based materials are highly suitable for liquid-phase sensor applications due to their chemical stability and high internal piezoelectric polarization. The sensitivity of GaN surfaces in aqueous solutions and polar liquids has been investigated. For this purpose, two terminal devices fabricated on bulk Si doped-GaN structures and undoped-AlGa_{0.3}N/GaN heterostructures with unpassivated open area are used to measure the responses to the changes of the H⁺ concentration in aqueous solutions and the dipole moment in polar liquids. The I-V characteristics show that the devices are able to distinguish the variations of pH. It is observed that the drain current decreases linearly with pH for both device structures. Evaluating the sensitivity in aqueous solutions at V_{DS} = 2V, a quite large current change is obtained for both structures. For the response to polar liquids, it is also found that the drain current decreases with the dipole moments. The results indicate that both devices are capable of distinguishing molecules with different dipole moments.

1. 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 [1-3]. 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 [4, 5]. 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.

AlGa_{0.3}N/GaN heterostructures have been extremely useful for gas and liquid sensor due to primarily three reasons: 1) a high electron sheet carrier concentration channel induced by piezoelectric polarization of the strained AlGa_{0.3}N layer, 2) the carrier concentration which is strongly depends on the ambient [6, 7] 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 [8-12]. The ability of electronic devices fabricated on these materials to operate in high temperature, high power and high flux/energy radiation conditions enable performance enhancements in a wide variety of spacecraft, satellite, homeland defense, mining, automobile, nuclear power, and radar applications.

In this work, the sensitivity of GaN surfaces in aqueous solutions and polar liquids has been investigated. For this purpose, two terminal devices fabricated on bulk Si doped-GaN structures and undoped-AlGaIn/GaN heterostructures with unpassivated open area are used to measure the responses to the changes of the H⁺ concentration in aqueous solutions and the dipole moment in polar liquids.

2. Design and fabrication process

2.1 Material structure

In this study, the GaN-based material structures used are bulk Si doped-GaN and undoped-AlGaIn/GaN heterostructure, as shown in Figure 1.

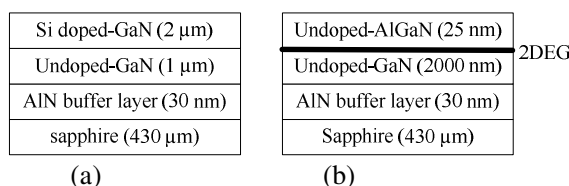


Figure 1. Cross sectional of material structure for (a) bulk Si doped-GaN (b) Undoped-AlGaIn/GaN heterostructure.

Both structures are grown on 430 μm sapphire substrates. The heteroepitaxial growth of the group III-nitrides on sapphire substrate allows the application of simple planar device structures [7]. The following AlN buffer layer is necessary as it dramatically improves the surface morphology and crystalline quality of the following undoped-GaN layer [13]. Furthermore, the thermal expansion of the sapphire substrate is close to that of AlN and AlN is also often used as packaging materials for high temperature sensor [7]. For the bulk structure, Si doped-GaN channel is formed near to the surface of the top layer. For the heterostructure, undoped-GaN layer is formed on the buffer layer and is followed by the growth of undoped-AlGaIn layer with 25% of Al composition. As a result, the two adjacent III-Nitride layers form a two-dimensional electron gas (2DEG) in between. From Hall measurement at room temperature, the carrier mobilities of bulk Si doped-GaN and undoped-AlGaIn/GaN heterostructure are 222 cm²/Vs and 1860 cm²/Vs, respectively. The 2DEG sheet carrier density in the heterostructure is 6.61×10¹² cm⁻².

2.2 Fabrication process

Figure 2 shows the top view and the cross sectional of GaN-based two terminal devices. The first fabrication process is mesa patterning. The mesa pattern of GaN-based wafer is obtained from photolithography, which is followed by the process of dry etching by reactive ion etching (RIE).

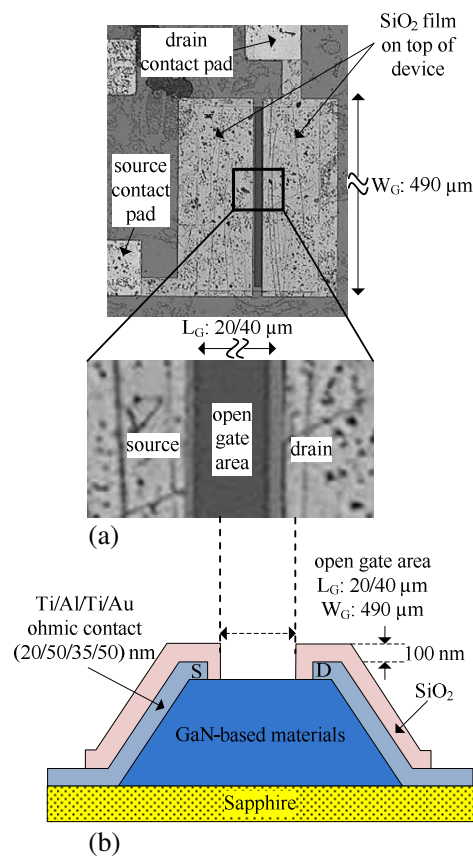


Figure 2. (a) Top view (b) Cross sectional of GaN-based two terminal devices.

In order to form source and drain ohmic contact, photolithography is done again before depositing metal layers of Ti/Al/Ti/Au (20/50/35/50nm). Methyleneethyl ketone (MEK) is then used for the lift off process. The alloyed Ti/Al/Ti/Au is obtained by the process of rapid thermal annealing (RTA) at 850°C in N₂ ambient for 30 seconds. Then, the ohmic contacts are coated with 100 nm of SiO₂ layer to insulate these contacts from being contacted to electrolyte. SiO₂ layer is deposited at 300°C by plasma-enhanced chemical vapor deposition (PECVD). Lastly, the SiO₂ layer at the opening area is etched out by wet etching using buffered hydrofluoric (BHF) solution.

The two-terminal devices chosen for measurement have the open area dimension of $490 \times 20 \mu\text{m}$ for bulk Si doped-GaN structures and $490 \times 40 \mu\text{m}$ for undoped-AlGaN/GaN heterostructures.

2.3 Measurement

Figure 3 shows the measurement circuit consisting of two working electrodes formed by copper lines on printed circuit board (PCB) and connected to source measure unit (SMU). The fabricated device is attached on the PCB and immersed in the electrolyte contained Teflon beaker.

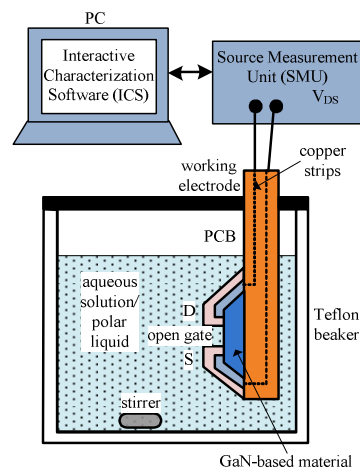


Figure 3. Schematic of measurement circuit

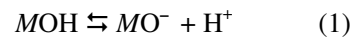
The drain-source of the fabricated devices are biased with drain-source voltage, V_{DS} by SMU and the drain current, I_D is measured by a computer installed with an interactive characterization software (ICS). Measurements are first done by immersing the working electrode in aqueous solutions with different H^+ concentration (pH) and then are followed by polar liquids with different dipole moment, at room temperature ($25^\circ C$).

3. Experimental results

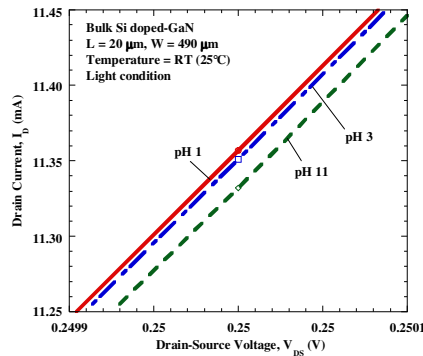
3.1 Current response to variation H^+ concentration

Variation of H^+ is represented by pH . Higher pH value shows the lower H^+ concentration present in the aqueous solution. Figure 4 shows the I - V characteristics of devices fabricated on bulk Si doped-GaN structure and undoped-AlGaIn/GaN structure in aqueous solutions with different pH . Figure 4(a) does not start at $V_{DS} = 0V$ because its scale has to be enlarged in order to observe clearly the micro-range current variations.

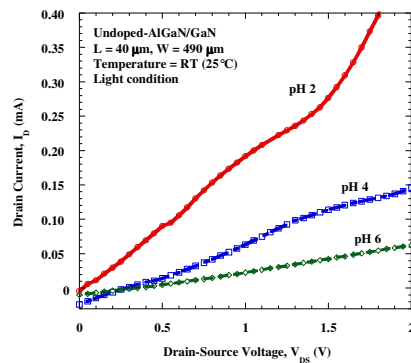
In order to analyze the current response of H^+ concentration, data of aqueous solutions with pH 1, pH 2 and pH 11 are used for bulk Si doped-GaN device, whereas data of aqueous solutions of pH 2, pH 4 and pH 6 are used for undoped-AlGaIn/GaN device. From the I - V characteristics, higher pH which contains less H^+ concentration is observed to have lower current. This phenomenon can be explained by using site-binding model [14]. It was reported that amphoteric hydroxyl groups (MOH : M may represents Ga) are formed at the oxide surface in contact with aqueous solutions. The amphoteric hydroxyl groups may be neutral, protonized or deprotonized, depending on the H^+ concentration. The equilibrium constants for the relevant reactions are:



When H^+ concentration decreases in aqueous solution, charges on the oxide surface become negative due to deprotonized hydroxyls (MO^-), as represented by Eq. (1). On the other hand, the increase of H^+ concentration in aqueous solution induces positive charges on oxide surface due to protonized hydroxyls (MOH_2^+), as represented by Eq. (2). These result in a potential change at GaN surface and change the current of the devices.



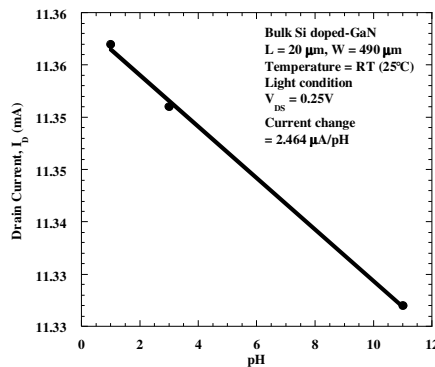
(a)



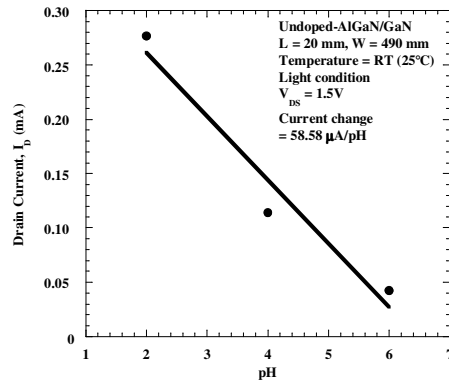
(b)

Figure 4. *I-V* characteristics of (a) bulk Si doped-GaN device (b) undoped-AlGaIn/GaN device in aqueous solutions with different *pH*.

The linearity of current as a function of *pH*, which indicates systematic potential change at GaN surface of both devices, is shown at $V_{DS} = 0.25\text{V}$ with current change (ΔI) = 2.464 $\mu\text{A/pH}$ for bulk Si doped-GaN device and $V_{DS} = 1.5\text{V}$ with $\Delta I = 58.58 \mu\text{A/pH}$ for undoped-AlGaIn/GaN device, as shown in Figure 5.



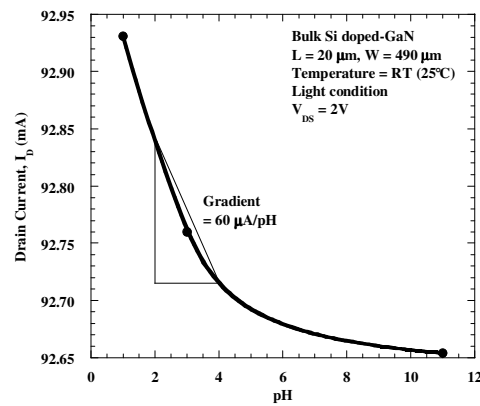
(a)



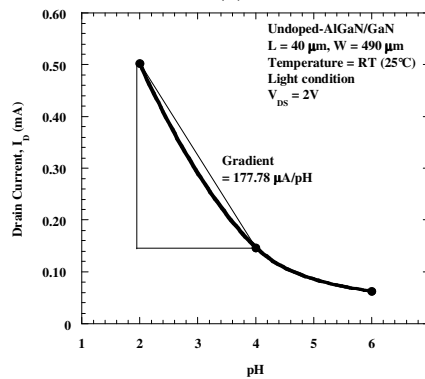
(b)

Figure 5. Drain current as a function of pH measured at (a) $V_{DS} = 1.5V$ for Si doped-GaN device (b) $V_{DS} = 1.5V$ for undoped-AlGaIn/GaN device.

For device performance comparison, Figure 6 presents the drain current measured at $V_{DS} = 2V$ for both devices.



(a)



(b)

Figure 6. Drain current as a function of pH measured at $V_{DS} = 2V$ for (a) Si doped-GaN device (b) undoped-AlGaIn/GaN device.

Since the graphs obtained are non-linear, the curve gradient from pH 2 to pH 4 is calculated, so that current change can be compared. It can be seen that the undoped-AlGaIn/GaN device has a larger current change of 177.78 $\mu A/pH$, as compared to bulk Si doped-GaN device with a smaller current change of 60 $\mu A/pH$.

3.2 Current response to polar liquids

The liquids used to investigate the current response of dipole moments are DI water, IPA and acetone. Figure 7 shows the drain current as a function of p/ϵ values for both devices (p is the dipole moment and ϵ is the dielectric constant of the liquid).

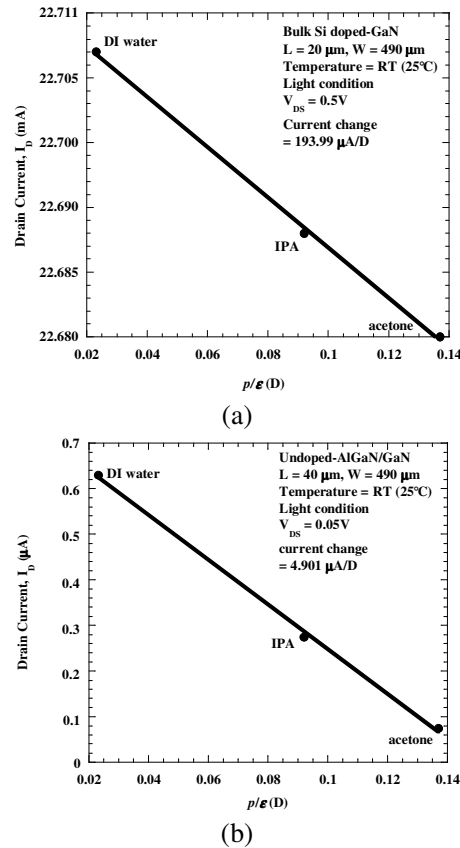


Figure 7. Drain current as a function of p/ϵ value measured at (a) $V_{DS} = 0.5V$ for Si doped-GaN device (b) $V_{DS} = 0.05V$ for undoped-AlGaIn/GaN device.

Systematic potential change (ΔV) at GaN surface of both devices is observed where current decreases linearly with the increase of p/ϵ value. Current changes, ΔI of $193.77 \mu A/D$ at $V_{DS} = 0.5V$ for bulk Si doped-GaN device and ΔI of $4.901 \mu A/D$ at $V_{DS} = 0.05V$ for undoped-AlGaIn/GaN device are obtained.

The potential drop (ΔV) at the polar liquid/GaN interface can be described by the Helmholtz model [15, 16]:

$$\Delta V = \frac{N_s p (\cos \theta)}{\epsilon \epsilon_0} \quad (3)$$

Where p is the dipole moment, N_s is the dipole density per unit area, θ is the angle between the dipole and the surface normal, ϵ is the dielectric constant of the polar liquid, and ϵ_0 is the permittivity of free space. Helmholtz model describes that p/ϵ value causes voltage drop at GaN surface and thus the relationship between current and dipole moment can be observed. Polar liquid with high p/ϵ value gives more voltage drop, which decreases the current.

4. Conclusion

In aqueous solutions with H^+ concentration (pH), higher pH give lower current. This relationship is explained by using the site-binding model. In polar liquids with dipole moments (p), higher p/ϵ value also give lower current. This phenomenon is described by using the Helmholtz model. In short, this work has successfully proven the ability of GaN-based two terminal devices to distinguish aqueous solutions with different pH and polar liquids with different dipole moments. For liquid sensor applications, systematic potential change in GaN-based two terminal devices is very important to ensure stable operation. Linearity of current change is observed indicating systematic potential change in both structures.

Acknowledgements

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