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On a GaN-based ion sensitive field-effect transistor (ISFET) with a hydrogen peroxide surface treatment

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ABSTRACT

A GaN-based ion sensitive field-effect transistor (ISFET) prepared by a hydrogen peroxide (H_2O_2) treatment is fabricated and studied. A 3-nm-thick Ga_xO_y layer formed by an immersion in H_2O_2 solution is examined and confirmed by EDS and XPS analyses. Experimentally, the studied pH-ISFET presents a higher voltage sensitivity (54.88 mV/pH), a higher current sensitivity (−56.09 μA/pH mm), a lower drift rate (1.41 μA/h mm), an extremely low hysteresis (0.4 mV), and a lower voltage decay rate (−0.14 mV/pH day) after 28 days. Moreover, insignificant interference effects from Na^+ and K^+ ions were observed. Thus, the studied GaN-based ISFET utilizing an H_2O_2 treatment promises to fabricate high-performance pH sensing applications.

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1. Introduction

Semiconductor-type chemical sensors can detect polar molecules and ions, which are extremely attractive for various chemical and biological sensing applications. Numerous semiconductor materials utilizing heterostructures and quantum structures have been studied and applied for chemical sensor applications [1–3]. The first ion-sensitive field-effect transistor (ISFET) with a SiO_2 insulator as a sensing membrane was reported by Bergveld [4]. Since then, many applications and improvements of ISFET have been widely studied [5–7]. However, the application of Si-based ISFET in aqueous solutions still suffers from various difficulties such as chemical instability and degradation of silicon nitride or silicon oxide gate insulators [8,9]. In addition, because the pH value is an important index in friendly environments and human beings, highly sensitive and stable pH sensors are indispensable for many applications such as clinical and biological analyses, medical detection, chemical analyses, environmental monitoring, etc. For instance, the body fluids of living organisms usually have a specific pH range. The pH value of normal human blood is regulated within the narrow range of 7.35–7.45 [10]. If the pH value of blood changes by as little as 0.03 pH units or less, the function of the human body will be greatly impaired [10]. On the other hand, a Group III-nitride such as gallium nitride (GaN) with a wurtzite crystal structure is a chemically stable semiconductor with high internal spontaneous and piezoelectric polarizations [11]. These properties of GaN are suitable to manufacture highly sensitive and durable sensors for chemical detection at higher temperatures and in harsh environments [12]. Based on the inherent advantages of GaN material, GaN-based ISFET was initially developed to detect H^+ ions in aqueous solutions [13]. Combined with surface functionalizations on GaN sensing membranes, various ion transports and specific recognitions could be performed for GaN-based ISFETs.

To improve chemical stability and long-term stability during ion-detection measurements, many kinds of sensing membranes (e.g., Ga_xO_y, SiO_2, Sc_2O_3, TiO_2, etc.) on GaN-based ISFETs were investigated and demonstrated [12,14,15]. A variety of methods are frequently to deposit metal-oxide membrane layers, such as sputtering, chemical vapor deposition, atomic layer deposition, and chemical wetness technique. [13–16]. However, the former three methods belonging to high-energy processes might damage the chemical composition and induce surface defects, which would cause the long-term instability and poor reliability of an ISFET [17]. Moreover, they usually need longer deposition time and higher cost apparatus. In this work, a chemical wetness technique, i.e., H_2O_2 surface treatment is proposed to form a Ga_xO_y sensing membrane on the GaN-based ISFET. The relationship between H_2O_2 immersion

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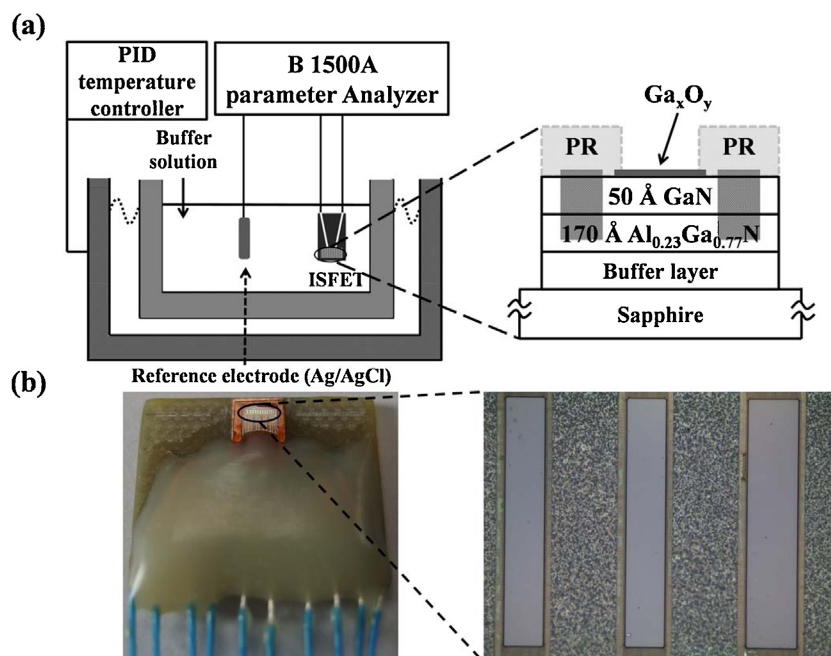

Fig. 1. (a) Schematic diagram of device's cross section and the related measurement setup. (b) Optical microscopy (OM) top view of the studied ISFET.

time and pH sensitivity is investigated for the studied ISFETs. Moreover, the related non-ideal effects on hysteresis, drift, life time, and ion interference are demonstrated in this work.

2. Experimental

The structure of the studied GaN-based ISFETs was grown on a sapphire substrate using a metal organic chemical vapor deposition (MOCVD) system. The epitaxial structure consisted of a GaN buffer layer, a 170 Å $Al_{0.23}Ga_{0.77}N$ active layer, and a 50 Å GaN cap layer. The epitaxial structure of the studied ISFETs is depicted in Fig. 1(a). After epitaxial growth, mesa isolations were performed by an inductively coupled-plasma reactive ion etching (ICP-RIE) system in a chlorine ambiance. Drain/source Ohmic contacts were sequentially evaporated by 10/100/10/100-nm-thick Ti/Al/Ti/Au metals, respectively. Then, the devices were annealed by a rapid thermal annealing (RTA) process at 900 °C in N_2 ambiance for 60 s. For the surface treatment, the studied device was immersed in an H_2O_2 solution (volume percent: 34.5%) for 7.5 min. The thickness of the native oxide (Ga_xO_y) grown on the GaN surface was about 3 nm. Furthermore, the photoresist (PR) was utilized to define the sensing area and prevent other areas from exposure to the electrolyte solution. For pH sensing, the GaN-based ISFETs were packaged using an aluminum-wire bonder and epoxy. Schematic diagrams of the cross section of the studied device and the measurement setup are illustrated in Fig. 1(a). In addition, the optical microscopy (OM) top view of the studied ISFET device is shown in Fig. 1(b). The pH buffer solutions were prepared using commercial products (Fluka Analytical). Before pH measurement, the commercial buffer solutions were individually calibrated by a commercial pH meter (CyberScan pH 510). The pH meter with a high resolution of ±0.01 pH provides precise pH measurement. The chemical composition of the studied devices was characterized by a scanning electron microscope (SEM; HITACHI SU8000) equipped with an energy-dispersive X-ray analysis system (EDS; Bruker) and a PHI VersaProbe Scanning X-ray Photoelectron Spectrometer (XPS) System coupled with a monochromatic Al Kα ($h\upsilon$ = 1486.6 eV) X-ray source. The current–voltage (*I*–*V*) characteristics were measured by a semiconductor parameter analyzer (Agilent B1500A).

3. Results and discussion

GaN-based ISFETs prepared with and without H_2O_2 surface treatments for 7.5 min are denoted as Devices A and B, respectively. Fig. 2(a) and (b) shows the EDS analyses of Devices A and B, respectively. Fig. 2 (c) shows the binding energy of Ga 3d for Devices A and B. The binding energy was scanned using a 0.2 eV/step, ranging from 14 to 28 eV for the Ga element. Fig. 2(d) shows the depth profiles of Devices A and B, which were analyzed by the XPS. Table 1 lists related data of EDS analyses for Devices A and B. The energy of the incident electron beam was kept at a high voltage (HV) of 10 kV. Clearly one can see from Table 1 and Fig. 2(d) that oxide atomic composition of Device A (3.35%) is substantially higher than that of Device B (0.35%). This confirms that the surface oxidation is indeed performed by the employed H_2O_2 treatment. From Fig. 2(c), it could be found that the binding energy of Ga_2O_3 is increased from 20.29 eV (Device B) to 20.74 eV (Device A) after the H_2O_2 treatment. It is well known that an atom in an outer shell will transfer toward the high binding energy level when it loses its electron [18]. Therefore, this shift in binding energy confirms the occurrence of Ga oxidation [18]. The higher signal intensity of Ga_xO_y on the GaN surface shows the increased deficiencies which provide more electrically active dangling bonds to easily detect hydrogen ions [19]. Therefore, Device A is expected to present a better sensing performance.

Fig. 3 shows the voltage sensitivity versus the immersion time of H_2O_2 surface treatment. The sensing electrode area is 70 μm × 450 μm. For each immersion time, 4 ISFET devices were characterized in this work. In Fig. 3, a highest average sensitivity of 54.88 mV/pH is obtained at an immersion time of 7.5 min. And the corresponding relative standard deviation (RSD) is about 1.6%. The ISFET without H_2O_2 treatment only exhibits a sensitivity of 43.55 mV/pH (RSD: 0.82%). An enhancement of about 26.0% on the voltage sensitivity is obtained after the employment of 7.5 min H_2O_2 surface treatment. Using H_2O_2 surface treatment, the GaN surface roughness and dielectric capacitance could both be increased by the insertion layer of Ga_xO_y. Due to the increased deficiencies from the Ga_xO_y surface, more electrically active dangling bonds are beneficial to detect hydrogen ions in an aqueous solution. This substantially improves the pH sensitivities of the treated ISFETs. Unfortunately, the series capacitance

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