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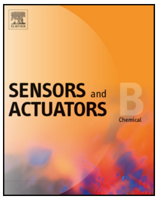

Highly sensitive nonpolar *a*-plane GaN based hydrogen diode sensor with textured active area using photo-chemical etching

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ABSTRACT

In this work, a highly sensitive *a*-plane $(11\bar{2}0)$ GaN (*a*-GaN) based hydrogen sensor with a large active surface area on the Schottky contact region was fabricated and characterized. By using a simple photo-chemical etching technique, a striated surface morphology with triangular prisms consisting of *m*-plane facets on the *a*-GaN surface was obtained. The maximum relative current change of the etched *a*-GaN diode was as high as 3.8×10^7%, and the reduction of the effective Schottky barrier height was 0.49 eV upon 4% hydrogen exposure. The photo-chemically etched *a*-GaN sensor showed a remarkably improved hydrogen response and good repeatability for cyclic exposure to hydrogen. The photo-chemically textured GaN surface with enlarged surface area increased the number of adsorption sites available for hydrogen molecules to catalytically-decompose into surface atoms, lowering the effective Schottky barrier height, thereby increasing the measured current. Furthermore, the hydrogen sensing properties of the etched *a*-GaN diodes at different values of humidity and temperature were investigated.

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1. Introduction

In recent years, hydrogen has attracted growing interest as an alternative energy source and carrier. As a pollution-free fuel, when reacted with oxygen, hydrogen can produce more heat of combustion per unit mass than gasoline and diesel [1]. Conventionally, hydrogen is used in petrochemical plants for hydrodealkylation, hydrodesulfurization, and hydrocracking processes [2]. Besides, it is employed as a coolant in power stations, owing to its large specific thermal conductivity, which is the highest among all gases [3]. However, colorless and odorless hydrogen gas presents some safety concerns, as it is extremely reactive with oxygen, owing to its low ignition energy [4]. In addition to preventing accidental explosions, the sensing of hydrogen gas is a very critical issue to monitor the efficiency of the energy conversion of the feed stream in fuel-cell vehicles [5].

Gallium Nitride (GaN) is one of the most suitable materials for the fabrication of semiconductor-type hydrogen gas sensors. Its superior material properties, such as wide bandgap, excellent carrier transport, and radiation hardness, enable the GaN-based gas sensors to exhibit high signal-to-noise ratios, as well as reliable and stable operations at high temperature and in harsh radiative environments [6,7]. In addition, the mechanical and chemical robustness of GaN can ensure great sensing reliability and durability [8,9]. Various types of GaN-based hydrogen sensing devices have been developed, including Schottky diodes, metal oxide semiconductor diodes, GaN nanowires and AlGaN/GaN high electron mobility transistors [10–15]. The crystal plane and polarity of the GaN film play vital roles in hydrogen sensing [14–16]. As reported by Wang et al. and Baik et al., Schottky diode hydrogen sensors based on nonpolar *a*-plane$(11\bar{2}0)$ GaN (*a*-GaN) exhibited a marked improvement in sensitivity, compared with those based on conventional Ga-polar *c*-plane (0001) GaN (*c*-GaN) due to the strong affinity of nitrogen on the *a*-GaN surface with hydrogen [14,16]. Kim et al. also dramatically improved the hydrogen sensing characteristics of the device by depositing catalytic platinum nanostructures in the active region, thereby creating a larger surface area that provided more active sites for hydrogen molecules to be adsorbed and decomposed [17,18]. Hence, the used of photo-chemically etched *a*-GaN with more available hydrogen adsorption sites is expected to provide a more effective way to enhance the hydrogen response levels. In such devices, thermal reliabilities higher than those of GaN-based sensors with platinum nanostructures can be also achieved, as the deformation of the metal nanostructure, which induces a sensitivity drop, occurs at much lower temperatures than that of the metal film [19].

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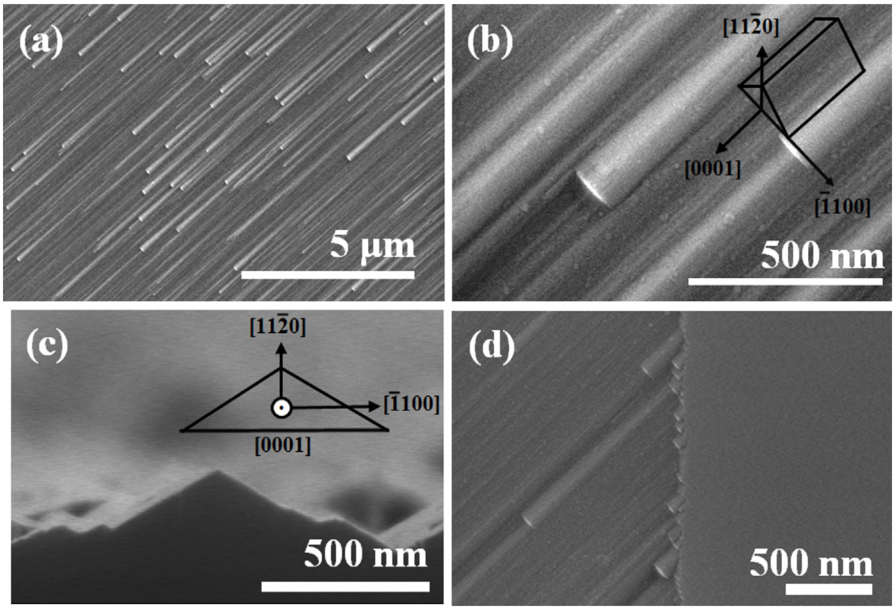

Fig. 1. (a), (b) Top and (c) cross-sectional view SEM images of photo-chemically etched *a-GaN*. (d) Top-view SEM image of selective area etched *a-GaN*.

In this work, the active region of the diode sensor was roughened by using a facile photo-chemical etching method, and an *a*-GaN sensor with a large area Schottky contact was fabricated. The crystal plane and polarity of the etched surface were studied, and the hydrogen sensing characteristics of the etched *a*-GaN Schottky diode were investigated. Notably, compared with the unetched *a*-GaN sensor, the etched nonpolar *a*-GaN diode exhibited a remarkable response to hydrogen exposure and the excellent hydrogen sensitivity up to the high temperature of 500 °C.

2. Experimental

Nonpolar *a*-GaN epitaxial layers of 4.5 µm in thickness were grown on *r*-plane $\left(1\bar{1}02\right)$ sapphire substrates by an 11 × 2 inch AIX 2400 G3 metal organic chemical vapor deposition system with a planetary reactor [16,20]. Trimethylgallium and ammonia were used as gallium and nitrogen sources, respectively. Prior to the growth, the substrate was thermally cleaned at 1050 °C for 10 min, and the surface nitridation was conducted with an ammonia flow of 8 slm (standard liter per minute). A 150-nm-thick GaN nucleation layer was grown in mixed atmosphere of H_2 and N_2 at the temperature of 1050 °C to achieve the minimum surface roughness and reduce dislocation density. Subsequently, an *a*-GaN layer with high crystalline quality surface was obtained by the conventional two-step growth method. To improve crystalline quality, during the three-dimensional island growth, multiple SiNx layers were inserted by flowing SiH_4 and NH_3. The surface orientation and crystalline quality of *a*-GaN films were characterized by high-resolution X-ray diffraction, using Jordan Valley QC3 system with a Cu $K_{\alpha 1}$ X-ray target source ($\lambda = 1.5406$ Å). The full width at half maximum values of the X-ray rocking curve for the grown *a*-GaN films were measured to be 450 and 620″ along the *c*-axis and *m*-axis, respectively.

The device fabrication was initiated with Ti/Al/Ni/Au ohmic contact formation by performing electron-beam evaporation (the deposition rate of 2 Å/s), lift-off, and annealing at 850 °C for 45 s in N_2 ambient. A 200-nm-thick Si_3N_4 layer was deposited for the diode isolation by plasma enhanced chemical vapor deposition. The windows for active area opening were prepared by using a buffered oxide etchant. For the photo-chemical etching, the sample was immersed in a potassium hydroxide solution and exposed to ultraviolet light (UV) by using an Excelitas X-cite 120PC Q, 120 W mercury lamp. The concentration of the KOH solution was 1 M, and the solution temperature and etching time were 85 °C and 5 mins, respectively. Then, for the catalytically active Schottky contact, a 10-nm-platinum film was deposited by electron-beam evaporation. Finally, Ti/Au contact pads were deposited for probing and wire bonding. A reference diode sensor was also fabricated with the same structure, but without using photo-chemical etching.

The surface morphology of the photo-chemically etched *a*-GaN film was analyzed by field-emission scanning electron microscopy (SEM). The current-voltage (I–V) hydrogen response characteristics of the *a*-GaN Schottky diode exposed to various concentrations of hydrogen balanced with nitrogen at 25 °C in a gas test chamber were measured using an Agilent 4155C semiconductor parameter analyzer.

3. Results and discussion

Photo-chemical etching of nitrogen polar *c*-GaN $\left(000\bar{1}\right)$ is a widely used method in vertical light emitting diode fabrication to enhance the light extraction through surface texturing [21,22]. This very simple wet etching process results in the formation of damage-free surfaces. Photo-chemical etching of GaN is a repetitive process involving the formation of gallium oxide and dissolution of the oxide in an alkali etchant solution; most of the stable specific crystal planes are exposed after the etching [23]. Fig. 1 shows top and cross-sectional SEM images of photo-chemically etched *a*-GaN. Straight stripes along the [0001] *c*-axis direction are observed in Fig. 1(a), while the magnified SEM images of Fig. 1(b) and (c) show triangular prisms lying on the *a*-plane with two *m-plane* $\left\{10\bar{1}0\right\}$ facets and the *a*-plane $\left(11\bar{2}0\right)$ base. The angle between the exposed facets was measured to be 120° in Fig. 1(c), which confirms the presence of *m*-plane facets of the $\left(10\bar{1}0\right)$ and $\left(01\bar{1}0\right)$ planes. The surface energy of a semiconductor is defined by the density of atoms and number of dangling bonds on the surface. Stable crystal planes with low atomic density and surface energy are etched slower than unstable planes; this determines the exposed specific crystal planes after photo-chemical etching. The atomic surface configurations of the nonpolar *a*-plane and nonpolar *m*-plane GaN (*m*-GaN) consist of alternate gallium and nitrogen atoms in 1:1 ratio, but the

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