Twofold Porosity and Surface Functionalization Effect on Pt–Porous GaN for High-Performance H₂-Gas Sensors at Room Temperature

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ABSTRACT: The achievement of H₂ detection, up to 25 ppm, at room temperature using sulfur-treated, platinum (Pt)-decorated porous GaN is reported in this study. This achievement is attributed to the large lateral pore size, Pt catalyst, and surface treatment using organic sulfide. The performance of H₂-gas sensors is studied as a function of the operating temperature by providing an adsorption activation energy of 22 meV at 30 ppm H₂, confirming the higher sensitivity of the sulfide-treated Pt–porous GaN sensor. Furthermore, the sensing response of the sulfide-treated Pt–porous GaN gas sensor increases with the increase in porosity (surface-to-volume ratio) and pore radii. Using the Knudsen diffusion–surface reaction equation, the H₂ gas concentration profile is simulated and fitted within the porous GaN layer, revealing that H₂ diffusion is limited by small pore radii because of its low diffusion rate. The simulated gas sensor responses to H₂ versus the pore diameter show the same trend as observed for the experimental data. The sulfide-treated Pt–porous GaN sensor achieves ultrasensitive H₂ detection at room temperature for 125 nm pore radii.

INTRODUCTION

Of late, hydrogen has been established as a promising, clean, renewable, and pollution-free alternative energy source and found applications in several fields such as spacecraft, transportation, and petrochemical plants. However, hydrogen gas poses certain safety concerns because it is odorless, highly volatile, explosive, invisible, and extremely reactive with oxygen. In H₂ applications, the safety during transportation, storage, and usage is crucial. Safe monitoring of H₂ leakage with compact, long-life, low-power-consumption sensors at room temperature is highly desired. However, H₂-sensing elements require a typical temperature of 100–200 °C to accelerate certain chemical reactions, leading to high energy consumption. GaN is a suitable candidate for several harsh applications, including gas sensors. However, GaN-based H₂ sensors have disadvantages of a long response time, high limit of detection of approximately 1000 ppm in the presence of oxygen, and high operating temperature. To avoid these problems, GaN nanostructures among other semiconductor materials, having structures such as pores, nanopores, nanorods, and nanotubes, are good solutions because of their large surface-to-volume ratio as well as the nanosize effect.

The significant increase in the surface-to-volume ratio subjects them to high trap-state densities if the surfaces are imperfectly passivated. Additionally, it can increase fast charge-carrier recombination rates and create additional energy barriers at the semiconductor surface. Furthermore, band bending on the lateral surfaces of GaN nanostructures due to space-charge-related surface depletion can increase the surface recombination rates and cause Fermi-level pinning. To overcome these problems of GaN nanostructures, various surface passivation technologies using organic and inorganic materials have been extensively investigated. Among them, sulfur-based compounds, such as sodium sulfide (NaS₂) and ammonium sulfide ((NH₄)₂S), are mainly used for passivating n-type GaN, enhancing its optical and electrical properties.

On the other hand, short-carbon-chain 1,2-ethanedithiol (EDT) (C₂H₂(SH)₂) is used for ultrathin bifunctional surface treatment, in which the S atoms can replace the surface oxide, simultaneously acting as a ligand for attaching the metal ion co-catalysts by forming strong covalent bonds. Recently, researchers have investigated the contact resistance, emission enhancement of InGaN/GaN nanowire light-emitting diodes, and enhanced photochemical water splitting, which have been treated with 1,8-octanethiol and EDT (1,2-ethanethiol) solutions. However, the performance of compact, high-
efficiency H₂-gas sensors based on sulfur-treated porous GaN fabricated by planar technology and operating at room temperature, without input power for the sensor, has not yet been explored.

In this work, combining the sulfur passivation of porous GaN nanostructures at different porosities (surface-to-volume ratios) with Pt nanoparticles, the H₂-gas detection performance is enhanced at room temperature. The sensitivity of the H₂-gas sensor is compared, with and without sulfur passivation. The sensitivity of the sensor is studied as a function of the GaN porosity (surface-to-volume ratio) and temperature. A model is proposed to theoretically describe the effect of the surface-to-volume ratio and passivation on the sensor performance.

### RESULTS AND DISCUSSION

Figure 1 shows the morphology of the porous GaN surface after etching for 10, 20, and 30 min. The pore-size distribution and porosity were determined for different etching times, using ImageJ software, as shown in Figure 1. The error in the pore diameters was estimated to be ±10 nm. The porosity was estimated from the enhancement of the threshold tool to select the areas of porosity from the scanning electron microscope (SEM) images using the same software. The error in the porosity was estimated to ±5%. In the SEM images, it was observed that for a short etching time of 10 min (Figure 1a) the pores were circular with an average diameter of 160 nm and a porosity of approximately 29%. As the etching time was increased to 20 min (Figure 1b), the pores collapsed with a nearby pore, growing and increasing the average pore diameter to 180 nm and the porosity to 42%. These larger pores were still circular. For an etching time of 30 min (Figure 1c), the pores collapsed, forming longer pores with lengths of a few hundred nanometers. The average pore diameter was approximately 250 nm, and the porosity was 58%.

To determine the changes in the chemical properties, before and after sulfur treatment, X-ray photoelectron spectroscopy (XPS) measurements were performed for porous GaN and sulfur-treated porous GaN. Figure 2a,b depicts the Ga 3s/S 2p region and (c, d) S 2s region. With two peaks located at 226.7 and 227.7 eV, attributed to the Ga−S bonds and C−S−H bonds, respectively. It is to be noted that on analyzing samples, as-grown and after sulfur treatment and Pt deposition (Figure S3 in the Supporting Information), traces of Pt element were detected after sulfur treatment and Pt deposition (Figure S3 in the Supporting Information). Comparison between XPS spectra of porous GaN and sulde-treated porous GaN: (a, b) Ga 3s/S 2p region and (c, d) S 2s region.

Figure 3a shows the response of a Pt−porous GaN gas sensor with a porosity of 58% (higher surface-to-volume ratio), before and after sulfur treatment at room temperature. It is determined that prior to passivation the sensitivity varies from 40 to 59%, whereas the variation of the H₂ concentration from 30 to 300 ppm exhibits a repeatable response. However, after treatment, the device response increased to 65% for a H₂ concentration of 30 ppm. This increase in response, for a lower H₂ concentration, is due to the effect of sulfur. The untreated gas sensor device shows saturation at a concentration of 130 ppm (Figure 3b). This may be attributed to the saturation of the adsorbed H₂ atoms at the porous surface, resulting in the full coverage of the porous surface area, which may block further adsorption and interaction of the H₂ atoms with the sensing surface. However, for the sulfur-treated sensor, the response is significantly more on increasing the H₂ concentration. Thus, such treatment drastically improves the performance of H₂-gas sensors at room temperature. This enhancement is due to the presence of sulfur, which effectively removes the surface defects on porous GaN by forming gallium ethanedithiolates, thus improving the surface stability. Due to

Figure 1. (a–c) Surface morphology of the porous GaN structure, generated after photochemical etching for 10, 20, and 30 min, respectively. Scale bar: 1 μm.

Figure 2. Comparison between XPS spectra of porous GaN and sulde-treated porous GaN: (a, b) Ga 3s/S 2p region and (c, d) S 2s region.
the presence of Pt nanoparticles on the surface, the two nearest gallium ethanedithiolates and the sulfide atom can combine with hydrogen. This process is based on hydrosulfurization, which is catalyzed by metal sulfides, as per the following equation:

$$R - S - R + 2H_2 \xrightarrow{Pt-sulfides catalyst} 2RH + H_2S.$$
Furthermore, the H₂ gas molecules near the sensor surface are dissociated and the hydrogen atoms combine with the sulfide atom. Thus, the electrical response of the gas sensor device is improved because of the decrease of the charge recombination rates, thereby enhancing charge extraction.

Figure 4a,b depicts the effect of temperature on the sensitivity of a sulfur-treated Pt–porous GaN device with a porosity of 58% at a H₂ gas concentration of 60 ppm. The effect of temperature on the performance of the sensor was investigated at 23, 60, and 80 °C. The responses of the sensor at 80 °C were higher compared to those at 23 °C because the thermal energy at a higher temperature is sufficiently high to overcome the activation energy barrier of the chemical reaction between sulfur (S) and the disassociated H₂-gas molecules. On increasing the working temperature up to 80 °C, we note that the sensor responses between 30 and 300 ppm H₂ were approximately 2-fold higher than those measured at room temperature. The parameters that limit the gas-sensing characteristics of porous materials are the surface-to-volume ratio and the surface morphology. The enhancement in the response is evident at higher temperatures.

The EDT/Pt–porous GaN sensor shows excellent response compared to that of GaN nanostructures, proving that sensors based on EDT/Pt–porous GaN are more promising for detecting low concentration of H₂ at room temperature.

The conductivity of an n-type semiconductor at room temperature is expressed by the following relationship: \( \sigma = \sigma_0 \exp(-E_a/(k_bT)) \), where \( \sigma \) is the electrical conductivity, \( E_a \) is the activation energy, \( k_b \) is the Boltzmann constant, and \( T \) is the room temperature in Kelvin. By plotting the logarithm of the response versus \( 1/T \), as shown in Figure 4b, the activation energy of the EDT/Pt–porous GaN sensor calculated from the slope of the Arrhenius plot is approximately 22 meV. This value is smaller than the activation energy value of 25 meV obtained for GaN NWs and 29.4 meV obtained for GaN nanotubes. This smaller value of the activation energy due to the sulfur treatment confirms the higher sensitivity of the sulfide-treated Pt–porous GaN sensor. Increasing the porosity (surface-to-volume ratio) of porous GaN increases the specific surface area, strongly influencing the sensor detectability.

Figure 5a shows the response of sulfide-treated Pt–porous GaN with respect to time, for different porosities of 29, 42, and 58% at room temperature. It was observed that the response to H₂ increases with the increase in porosity. Therefore, a porosity of 58% drastically enhances the response to H₂ gas.

Figure 5b shows that on increasing the pore radius (increasing the porosity), at 30 ppm H₂ gas, the exposure area of EDT/Pt–porous GaN increases, which is responsible for the higher response. The diffusion rate of H₂ gas is very high for larger pore sizes on the upper layer of a thin film. To clarify the porosity outcomes measured experimentally from sulfide-treated Pt–porous GaN gas sensors, the influence of H₂-gas transport on the sensitivity of porous GaN was investigated theoretically.

The mechanism of gas diffusion through a porous material depends on the size of pores, the surface diffusion, and the Knudsen diffusion. The Knudsen diffusion coefficient \( (D_k) \) is given by eq 1 for a mesoporous film

\[
D_k = \frac{4r}{3} \sqrt{\frac{2RT}{\pi M}}
\]

where \( T \) is the temperature, \( r \) is the pore radius, \( R \) is the gas constant, and \( M \) is the molecular weight of the diffusing H₂ gas.

To simulate the H₂ concentration within the porous GaN layer, depending on the pore size, we use the diffusion equation, eq 2, which depends on the concentration of the gas in the layer and the gas diffusion and surface reaction rates

\[
\frac{\partial C}{\partial t} = D_k \frac{\partial^2 C}{\partial x^2} - KC
\]

where \( C \) is the concentration of H₂, \( t \) is the time, \( x \) is the thickness of porous GaN, \( D_k \) is the diffusion coefficient, and \( K \) is the rate constant of the surface reaction. Solving the equation in the steady-state condition \( (\partial C/\partial t = 0) \), the gas concentration profile is given by eq 3

\[
C = C_r \frac{\cosh \left( \frac{1 - x}{L} \right)^m}{\cosh(m)} , \quad m = L \sqrt{\frac{K}{D_k}}
\]

where \( L \) is the porous layer thickness. The gas concentration profile depends on the pore size, if \( K \) is constant. Figure 6 shows the gas concentration profiles simulated using eq 3 for a 300 nm thick porous GaN layer at various pore diameters of 160, 180, and 250 nm and at \( K = 100 \, 000 \, \text{s}^{-1} \). The film thickness (L) and surface concentration \( (C_r) \) were set to 300 μm and 30 ppm, respectively. (The inset shows the cross-sectional SEM image; scale bar: 500 nm.)

Figure 6. Simulated H₂ concentration profiles within a porous GaN layer at different values of \( \sqrt{K/D_k} \) corresponding to pore diameters of 160, 180, and 250 nm and at \( K = 100 \, 000 \, \text{s}^{-1} \). The film thickness (L) and surface concentration \( (C_r) \) were set to 300 μm and 30 ppm, respectively. (The inset shows the cross-sectional SEM image; scale bar: 500 nm.)
The conductance of the porous layer is given by the integral of $\frac{1}{D_k}$, where $D_k$ is the diffusion depth, because of Knudsen diffusion. The simulation results revealed that H$_2$ diffuses easily from pores having a large diameter. The sensor responses with respect to the pore diameter showed the same trend for the simulation and experimental data. The sulfur-treated Pt–porous GaN sensor involves low-technology fabrication steps, is of low cost, and provides high performance at room temperature. Hence, the sulfur-treated device is promising for improving hydrogen sensitivity in sensor applications.

### EXPERIMENTAL SECTION

**Synthesis of Porous GaN Nanostructures.** Porous gallium nitride was fabricated by metal-assisted electroless etching. The unintentionally doped n-type GaN film used in this study was grown on a c-axis (0001) sapphire substrate with a resistivity below 0.05 $\Omega \cdot$ cm, purchased from Xiamen Powerway Advanced Material Co., Ltd. The thickness of the GaN film was approximately 30 $\mu$m, and its carrier concentration was $3.4 \times 10^{17}$ cm$^{-3}$. The GaN samples were cleaned by sonication in acetone followed by 2-propanol for 5 min in each solution. Furthermore, the samples were immersed in nitric acid (HNO$_3$) at 65 °C for 15 min, after which they were rinsed with deionized (DI) water and methanol. Two narrow stripes of 10 nm thick Pt, separated by a few millimeters, were deposited on the GaN samples using a sputtering system. The samples were then etched in the H$_2$O$_2$/HF/CH$_3$OH (2:1:2) solution, under UV illumination, for different etching times (10, 20, and 30 min). After chemical etching, the samples were removed from the solution and rinsed with DI water. The X-ray diffraction pattern of wurtzite porous GaN nanostructures etched for 30 min is depicted in Figure S1 in the Supporting Information. For surface passivation, the porous GaN samples were immersed in EDT (99.99%, Sigma-Aldrich) for 5 min, after which they were rinsed with ethanol and dried with N$_2$. Pt nanoparticles of 5 nm radii were then deposited on the surface of the porous GaN layer using an ultrahigh vacuum chamber for nano-
particles production (Mantis Deposition Ltd.). Before the sensing measurements, Pt/Ni electrical contacts were deposited by a sputtering system on the top surface, with a thickness of 100 nm, using an electrode hard mask. The image of the fabricated sensor device is depicted in Figure S2 in the Supporting Information.

**Physical Characterization.** An FEI’s Magellan 400-FEG scanning electron microscope (SEM), operating at 5 keV beam energy, was used for analyzing the morphology of porous GaN. X-ray photoelectron spectroscopy (XPS) was carried out to analyze the surface property of porous GaN, before and after surface treatment. The XPS data were obtained using an Axis Ultra DLD system (Kratos, U.K.) with an Al Kα radiation source ($hν = 1486.8$ eV), under a vacuum of $10^{-9}$ mbar. The binding energy was calibrated according to the C 1s peak at 284.8 eV. The data were analyzed using “CasaXPS”, and the peaks were fitted using a Gaussian–Lorentzian function. The sensor was fixed on a test stage consisting of a heater within a temperature-controlled Teflon chamber. H₂ gas was diluted with N₂ gas within a gas mixer and injected into the chamber at controlled flow rates, using Bronkhorst mass flow meters and a controller. As dry gases were used within the sealed-off test chamber, the relative humidity within the chamber was assumed to be negligible. The response signal of the device was measured using a computer-controlled Keithley Instruments source measurement unit (K1 236). The source and gas flow rates were controlled to a computer, using LABVIEW software. The sensor response was defined as $S = R_L/R_R$, where $R_L$ and $R_R$ are the resistances of the gas sensor in air and H₂ gas, respectively.

**ASSOCIATED CONTENT**

**Supporting Information**

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acsomega.8b02730.

H₂ gas sensor device, X-ray diffraction pattern of porous GaN, and survey of XPS spectra (PDF)

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**Notes**

The authors declare no competing financial interest.

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