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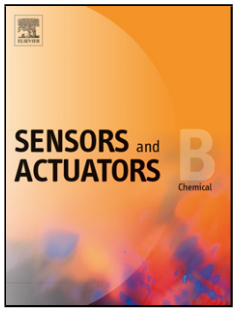

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Impact of high temperature H_2 pre-treatment on Pt-AlGaN/GaN HEMT sensor for H_2S detection

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Highlights:

- A H_2 gas flow pre-treatment at high temperature is utilized for Pt-AlGaN/GaN HEMT H_2S sensors
- The detection concentration range of H_2S is extended after pre-treatment
- The linearity of Pt-HEMT sensors is improved after pre-treatment
- The mechanism behind pre-treatment is discussed and characterized

Abstract:

In this paper, a method to extend the detection range of hydrogen sulfide (H_2S) gas sensor is demonstrated. The sensor is based on AlGaN/GaN high electron mobility transistors (HEMTs) with Pt gate. It is observed that the as-fabricated devices exhibited sensing signal saturation at 30 ppm H_2S exposure in dry air. A pre-treatment using H_2 pulses in dry air ambient at 250 ºC was applied to extend the detection range of the sensor. The H_2 treated H_2S gas sensor was able to detect a higher

H_2S concentration up to 90 ppm at 250 ºC without complete saturation.

Key words: AlGaN/GaN; Pt; HEMT; H_2S; Gas Sensor

1. Introduction

H_2S is colorless gas with the foul smell of rotten eggs. It can be emitted by many industrial activities, such as petroleum refining or paper mills [1]. Besides, it is a broad-spectrum poison and can severely affect the nervous system. Only 20 ppm H_2S is the acceptable ceiling concentration established by the Occupational Safety and Health Administration (OSHA) [2]. For 100-150 ppm H_2S, it can paralyze the olfactory nerve, and then the sense of smell will disappear [1]. Therefore, it is necessary to detect H_2S within low tens up to at least 100 ppm.

There are many ways to detect H_2S, such as metal-oxide semiconductor (MOS) [1, 3], electrochemical [4], Schottky diode [5] and field effect transistor sensors [6, 7]. Because industrial activity is one of the largest sources of H_2S, harsh environments like high temperature should be taken into consideration. Due to the wide bandgap, high chemical stability and corrosion resistance, the compound semiconductors like GaN [8, 9] and SiC [10, 11] have gained many interests. Compared with Schottky diode sensors, the transistor-based sensors usually have a larger sensing current and a higher signal-to-noise ratio. Especially for AlGaN/GaN based HEMT [12, 13], due to the presence of two-dimensional electron gas (2DEG), the response current is even larger than mA level.

Previously, Darmastuti et al. had fabricated a H_2S sensor based on SiC field effect transistor (FET) with Pt or Ir gate [10]. The device exhibited saturation at a low concentration of approximately 10 ppm, which is similar to our as-fabricated sensors. No pre-treatment was reported for their sensors. It was also demonstrated that the as-fabricated Pt-SiC MOS sensors showed little or no H_2 gas response [14]. In order to activate sensors, alternated exposures of 1% O_2 in N_2 (5 min) and 10% H_2 in N_2 (3 min) for 7 h at 610 ºC were once applied [14].

In this work, we report on the H_2 related pre-treatment for H_2S gas sensors based on the AlGaN/GaN HEMT. It clearly shows the effective extension of H_2S detection range.

2. Experimental

Fig. 1 (a) shows the cross-sectional view of our HEMT-sensors. The devices were fabricated on 2-inch sapphire wafers with epitaxial stacks grown by MOCVD. The epitaxial stacks started with a nucleation layer in order to reduce the lattice mismatch. Then it was followed by a 1.8 μm GaN buffer layer, a 1 nm AlN interlayer, an unintentionally doped 21 nm $Al_{0.26}Ga_{0.74}N$ barrier layer and a 1 nm GaN capping layer. The mesa etching was performed by BCl_3/Cl_2 plasma to isolate devices. Then a Ti/Al/Ti/Au (20/110/40/50 nm) stack was deposited on source/drain by e-beam evaporation and patterned by lift-off method. Annealing at 870 ºC for 47 s in N_2 ambient was performed to form Ohmic contacts. Next, the sensing gate electrode was formed by e-beam evaporation of a 10 nm Pt layer. Gate pattern was also defined by lift-off process. The bi-layer of 30/300 nm Ti/Au was also fabricated by lift-off process as wire bonding metal. The devices were passivated by 500 nm SiN_X using PECVD. Finally, Pt gate area was opened to detect the gas. The gate dimension exposed to gas was 40 μm × 400 μm (length × width) and the spacing between gate and source/drain was 6 μm [15].

The as-fabricated sensors were annealed at 150 ºC in dry air for 48 hours to test sensors' thermal stability. The sensors with no performance degradation were used for the formal measurement. A commercial gas mixing system was employed for H_2S sensing and H_2 pre-treatment. A stable gas flow at a rate of 310 sccm was continuously introduced into a 1.8 L chamber. H_2S or H_2 gas in dry synthetic air background with different concentration was used. The humidity in chamber was kept at 0% RH for all the measurements to inhibit moisture influence. For H_2 pre-treatment, the samples were exposed to alternated gas flows of dry air (50 min) and H_2 (20 min) for 6 hours at 250 ºC. The concentration of H_2 was increased with each exposure from 100 ppm to 900 ppm at 300 ppm increment. The electrical signals were measured by a pair of Keithley 2450 source meters with the measurement temperature varying from room temperature to 300 ºC . After each measurement cycle, the sensors were reset under room ambient conditions for at least 12 hours. Atomic force microscopy (AFM) was utilized to analyze the Pt gate surface morphology before and after H_2 pre-treatment.

3. Results and discussion

3.1 As-fabricated sensors

Our previous studies have shown that the sensor can operate from 150 to 250 ºC, and the best performance is obtained at an operation temperature of 250 ºC [16]. Fig. 1 (b) shows the output curves of a HEMT sensor exposed to 15 ppm H_2S gas at 250 ºC. Obviously, it shows linear and saturation regions with a pinch-off voltage of -3 V (gate-source voltage, V_{GS}). It is a normally-on device due to the 2DEG. When exposed to H_2S gas, the source-drain current I_{DS} increases even at a low concentration of 15 ppm, as shown in Fig. 1(b). The detection mechanism is related with the reaction between gas and catalytic metal Pt. After H_2S is adsorbed on Pt film, the S-H bond is then divided by Pt, resulting in the production of H atom or molecule [5]. Finally, the S atom can react with O_2 to form volatile SO_2 and the H atom can form a dipole layer. The dipole layer can affect the Schottky barrier height between Pt and GaN. Consequently, the threshold voltage and the drain current are altered.

Fig. 2 (a) shows the transient response curves for different H_2S concentrations at 250 ºC using a fresh sensor without H_2 pre-treatment. The transient response curves were measured at fixed voltages (V_{GS}=0 V and V_{DS}=5 V). The drain current (I_{DS}) was recorded with a 3s interval. Clearly, I_{DS} is saturated at a low concentration of 30 ppm. With a higher gas concentration, the H_2S atoms will cover larger area of Pt surface. Then the output signal intensity increases. But there is a limit for coverage, which may result in saturation of signal intensity. This phenomenon is similar to other studies based on Pt-SiC field effect transistor [10].

3.2 H_2 pre-treated sensors

Fig. 2 (b) shows the transient response curves for the H_2 pre-treated sensors at 250 ºC. The different H_2S concentration is clearly distinguished.

To compare the detection range more quantitatively, the gas response is calculated by

$$S(\%) = \frac{\Delta I_{DS}}{I_{DS,air}} \times 100\%, \quad (1)$$

where $\Delta I_{DS} = I_{DS,H_2S} - I_{DS,air}$ is the drain current variation between H_2S and air ambient. Fig. 3 (a) shows the gas response to H_2S for sensors with and without H_2 pre-treatment. Without H_2 pre-treatment, the gas response is nearly unchanged for different H_2S concentration. While, with H_2 pre-treatment it's almost linear. The large difference indicates that the H_2 pre-treatment can effectively improve sensor performance by extending its detection range.

Before sensing H_2S, the influence of H_2 pre-treatment on sensors is summarized in Fig 3(b) - (d). As shown in Fig. 3 (b), the H_2 pre-treatment obviously makes the drain current become smaller. The expression of drain current in saturation region is,

$$I_{DSS} = \frac{\mu C_b W_g}{2L_g}(V_{GS} - V_{TH})^2, \quad (2)$$

where μ is the 2DEG mobility, W_g/L_g is the ratio of gate width to length, C_b is the sum of capacitance contributions from each layer between the gate metal and 2DEG ($1/C_b = 1/C_{GaN} + 1/C_{AlGaN} + 1/C_{AlN} + 1/C_{2DEG}$). Threshold voltage ($V_{TH}$) in this work is extracted from $\sqrt{I_{DS}} - V_{GS}$ curve biased in the saturation region [17]. The V_{TH} shift can be observed in Fig. 3 (c), which shows a positive shift and results in I_{DS} and transconductance g_m reduction. Fig. 3 (d) is the extracted I_{DS} shift and V_{TH} shift at different measurement temperature. At a higher measurement temperature, the V_{TH} shift decreases and the I_{DS} shift is smaller too. The expression of V_{TH} for an AlGaN/GaN HEMT is [18],

$$V_{TH} = \Phi_b - \frac{\Delta E_c}{q} - \frac{qn_s}{C_b}, \quad (3)$$

where Φ_b is the Schottky barrier height, ΔE_c is the conduction band discontinuity, q is the electron charge and n_s is the sheet charge carrier density. The Schottky barrier height is dependent on the work function (Φ_m) of the gate metal and the semiconductor electron affinity (χ_s), i.e., $\Phi_b = \Phi_m - \chi_s$. Therefore, we believe the shift of V_{TH} should be related to the work function variation of the gate metal.

3.3 Explanation

For the V_{TH} positive shift after H_2 pre-treatment, there are three possible reported mechanisms [19]: a) the adsorbed H atoms give rise to a dipole layer; b) the existence of slow H trapping sites; and c) the lattice expansion due to H induced stress. The dipole layer is reversible and decreases the V_{TH} [20-22]. The existence of slow H trapping sites is also called Hydrogen-Induced Drift (HID), which is related to the metal-insulator interface [19, 23]. It is not temporary but it can decrease the V_{TH}. According to previous studies [19, 24], the stress induced by H can change the metal lattice parameters. Besides, it can even loosen contact between metal and substrate [24]. However, we didn't find any difference of sensor after H_2 pre-treatment by microscope. The AFM measurements shown in Fig. 4 also clearly indicate little change on Pt film. So, a), b) and c) cannot explain our

experimental results.

A new mechanism is proposed in this paper. Fig. 5 (a) and (b) show the H existence status in Pt gate for sensors with and without H_2 pre-treatment. There will be two statuses of H atoms within Pt film. One is trapped by the body defects in the Pt grains, losing its electron and existing as fixed positive H^+ ions. Then the induced negative charges will accumulate on Pt film surface as shown in Fig. 5(b). These negative charges will increase the work function of Pt film, resulting in the V_{TH} positive shift and I_{DS} reduction as mentioned in section 3.2. Since these H^+ ions within Pt grains are fixed, these induced negative charges on Pt film surface are also stable. So there is a larger V_{TH} for these transistors with H_2 pre-treatment as shown in Fig. 3(b). The other status of H is related with absorption by Pt surface defects [25], such as surface clusters, steps and grain boundaries. Once absorbed, H atoms will show positive polarity due to a weaker electronegativity comparing with Pt. Thus, a dipole layer may form around the Pt film surface and grain boundaries as shown in Fig. 5(c) [19]. These dipole layers will equivalently lower the Pt film work function, which will reduce the V_{TH}, as calculated in equation (3). However, these absorbed H atoms are not stable, they can diffuse out into the atmosphere when the sensed H_2 concentration is lower.

Since there are fixed H^+ ions within the Pt grains after H_2 pre-treatment, the induced negative charges on the Pt film surface and interface will stably exist there. While, the adsorbed H atoms on the Pt top surface will show positive polarity. Thus, the induced negative charges will attract these adsorbed H atoms. Consequently, on the Pt top surface the entrance for the adsorbed H atoms' diffusion into the Pt grain boundaries and interface of Pt/GaN will be partly blocked. The H atoms diffusion velocity at these entrances will be lowered. Hence, with the same sensing condition the amount of H atoms which can finally diffuse into the interface of Pt/GaN will decrease for the same H_2S concentration. So even for 90 ppm H_2S the I_{DS} is still not saturated for the sensor with H_2 pre-treatment. As seen in Fig. 2(b), the I_{DS} for 90 ppm H_2S is less than that for 30 ppm H_2S but without H_2 pre-treatment as shown in Fig. 2(a). Thus, the sensor detection range is extended.

Besides, Fig. 6 shows the transient response curves for samples pre-treated with H_2 at 250 ºC. Only after exposure to 900 ppm H_2, the I_{DS} in dry air obviously degrades. It means that the positive H^+ ions form within the Pt grains, which indicates that it is not easy to form these diffusion induced H^+ ions. A higher temperature and a higher H_2 concentration are preferred to activate and accelerate

the diffusion process within the grains. Once these H^+ ions form they will be much more stable than those adsorbed H atoms at surface or interface. It also implies the H^+ ions formation process can be ignored when the H_2 concentration is less than 100 ppm.

4. Conclusion:

In this paper, it is demonstrated that the Pt-AlGaN/GaN HEMT can detect H_2S at 250 ºC. However, the as-fabricated sensors exhibit saturation for low H_2S concentrations. A high temperature H_2 pre-treatment is proposed and successfully applied to extend the concentration detection range. The mechanism is believed to be correlated with the interaction between Pt film and H_2, which may introduce stable H^+ ions within Pt grains and result in the V_{TH} positive shift. The negative charges induced by these H^+ ions at the Pt top surface will attract the adsorbed H atoms with positive polarity. This will reduce the diffusion velocity of these adsorbed H atoms, finally decreasing the amount of H related dipoles at Pt/GaN interface. Thus a higher H_2S concentration is required to reach the same saturation level of the dipoles at Pt/GaN interface, which equivalently increases the H_2S concentration detection range.

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Figure captions

Fig 1. (a) The cross-sectional diagram of HEMT-sensor in this work. (b) The output curves of a fresh sensor exposed to air and 15 ppm H_2S at 250 ºC without H_2 pre-treatment.

Fig 2. (a) The transient response curves for H_2S at 250 ºC without H_2 pre-treatment. (b) The transient response curves for H_2S at 250 ºC with H_2 pre-treatment.

Fig 3. (a) Gas responses for sensors with and without H_2 pre-treatment measured at 250 ºC. (b) Output curves for sensors with and without H_2 pre-treatment measured at 250 ºC. (c) Transfer and g_m curves for sensors with and without H_2 pre-treatment measured at 250 ºC (V_{DS}=7 V). (d)With H_2 pre-treatment the measurement temperature influence on I_{DS} shift ($I_{DS,shift}$, V_{GS}=0 V, V_{DS}=5 V) and threshold voltage shift ($V_{TH,shift}$).

Fig 4. AFM image for Pt film surface (a) without and (b) with H_2 pre-treatment at 250 ºC.

Fig 5. The H existence status in Pt gate for sensor (a) without and (b) with H_2 pre-treatment. The H existence status in Pt gate after sensing H_2S for sensor (c) without H_2 pre-treatment and (d) with H_2 pre-treatment.

Fig 6. The transient response curve for samples treated with H_2 at 250 ºC.