Temperature and Humidity Dependence of Response of PMGI-Encapsulated Pt-AlGaN/GaN Diodes for Hydrogen Sensing

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***Abstract*— Polydimethylglutarimide (PMGI), a photosensitive positive type resin used in photoresists, is shown to be an effective moisture barrier for mitigating the effect of humidity on the sensitivity of Pt-AlGaN/GaN Schottky diodes for hydrogen detection. The maximum relative current change observed in PMGI-encapsulated diodes forward biased at 1.3 V and exposed to 500 ppm of dry or humid H_2 is identical within experimental error over the temperature range 25 °C–300 °C and peaks at 200 °C in both cases (4.4×10^6% increase compared with the current under N_2 ambient). Unencapsulated diodes exhibit a decrease of approximately one ninth of the current signal for detection of 500 ppm of H_2 in the presence of water. The PMGI is easily spun-on to the sensors, and does not degrade until temperatures of 335 °C or higher, making it a suitable moisture barrier for most hydrogen sensing applications.**

***Index Terms*— Hydrogen, GaN, moisture barrier, encapsulation.**

I. Introduction

THERE are many applications in which detection of hydrogen leaks is critical [1], including industrial processing of ammonia, petrochemicals, and methanol, as well as fuel cell technologies, rocket propulsion systems, hydrogen-fueled vehicles and aircraft [1]–[4]. There is also new interest in the production of hydrogen through photocatalytic water splitting [4]. Hydrogen may be a significant component of renewable sources of clean energy, and in terms of removing the greenhouse gas carbon dioxide, it could be a feedstock gas for reactions which transform the sequestered CO_2 back into a useable fuel through catalytic hydrogenation [1]. All of these applications need robust and networked hydrogen sensors. Schottky diode semiconductor hydrogen gas sensors typically employ a Pd or Pt Schottky gate that catalytically decomposes molecular hydrogen to the atomic form [5]–[15]. The atomic hydrogen can be selectively absorbed in the gate, diffusing to the interface with the semiconductor and lowering the Schottky energy barrier [5], [16]. This produces an increase in diode current at fixed operating bias voltage. In particular, wide bandgap semiconductors such as SiC and GaN are attractive for their ability to operate at elevated temperatures where cooling systems are not practical and they can selectively detect low concentrations (ppm range) of hydrogen at room temperature and above [5]–[35]. Hydrogen is combustible at concentrations in air of >4.65% [4], [24], but much lower concentrations need to be detected to have early notice of leaks.

One advantage of GaN over SiC is the availability of AlGaN/GaN or InAlN/GaN heterostructures which can be used in transistor or diode mode to amplify the hydrogen sensing signal. These heterostructures produce a high density electron gas channel at the heterointerface whose concentration is strongly dependent on the presence of surface charges [5]–[7], [18]–[21], [36]. Thus, changes in the effective Schottky barrier height due to hydrogen absorption produce large changes in current of sensor channel at fixed operating voltage. While GaN nanowires are effective hydrogen sensors when contacted with Pd or Pt, thin film structures are preferred because of the availability of highly reproducible fabrication processes that lead to uniform performance and easy integration with wireless communication systems. Recent advances in enhancing the hydrogen detection sensitivity of GaN-based diodes include the use of catalytic metal nanonetworks with high surface-to-volume ratio [15], [25], [29], and the exploitation of the different sensitivity of particular crystal planes and surface terminations (N-face versus Ga-face) to the adsorption of hydrogen [2], [19], [28], [35].

In the practical application of Pt or Pd contacted GaN-based diodes, one issue has been the fact that the

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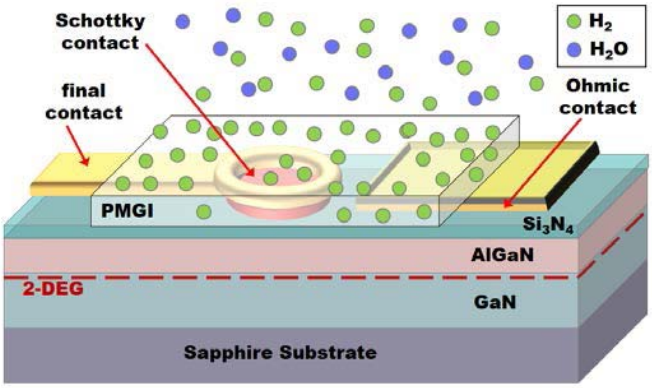

Fig. 1. Schematic of epi-layer structure and device layout of PMGI encapsulated Pt-AlGaN/GaN Schottky diode hydrogen sensor.

sensitivity for hydrogen detection is found to be significantly reduced in the presence of humidity in the ambient [31]. We have previously found that encapsulation with a common polymer film, poly(methyl methacrylate) (PMMA) can mitigate this problem by providing a moisture barrier that prevents water vapor from reaching the semiconductor surface [37]. There are other resist materials available that are based on polydimethylglutarimide (PMGI) which have even lower permeability coefficients for moisture [38]. PMGI is readily applied to semiconductor surfaces by simple spin-on techniques and it is stable to 335°C. Thus it is an option as a moisture barrier on Pt/GaN and Pd/GaN-based hydrogen sensors for sensing applications at humid or high temperature conditions.

II. Device Fabrication and Measurement

The AlGaN/GaN layer structures were grown on *c-plane* Al_2O_3 substrates with a low temperature GaN buffer by metal-organic chemical vapor deposition (MOCVD) as described previously [11], [15], [24]. Figure 1 shows a schematic of the layer and device structure. There is a two-dimensional electron gas (2-DEG) formed at the interface between the AlGaN asnd GaN layers and this is used as the conducting channel. Modulation of the current flowing in this channel between the Schottky and Ohmic contacts occurs by modification of the barrier height of the Pt Schottky gate induced by charge at the interface between and Pt and AlGaN from decomposed atomic hydrogen. This standard Ga-polar AlGaN/GaN heterostructure had a sheet carrier density of 1×10^{13} cm^{-2}. The device fabrication started with Ohmic contacts, which were formed by lift-off of e-beam deposited Ti (200 Å)/Al (800 Å)/ Ni (400 Å)/Au (800 Å) subsequently annealed at 900 °C for 60 s under a flowing N2 ambient. The surface was encapsulated with 2000 Å of plasma enhanced chemical vapor deposited SiN_x at 300°C for electrode isolation. Windows in the SiNx were opened by buffered oxide etchant wet etching, and 100 Å of Pt Schottky metal was deposited by e-beam evaporation for Schottky contacts. The final metal, Ti/Au (200 Å/2000 Å), was deposited by e-beam evaporator for interconnection contacts.

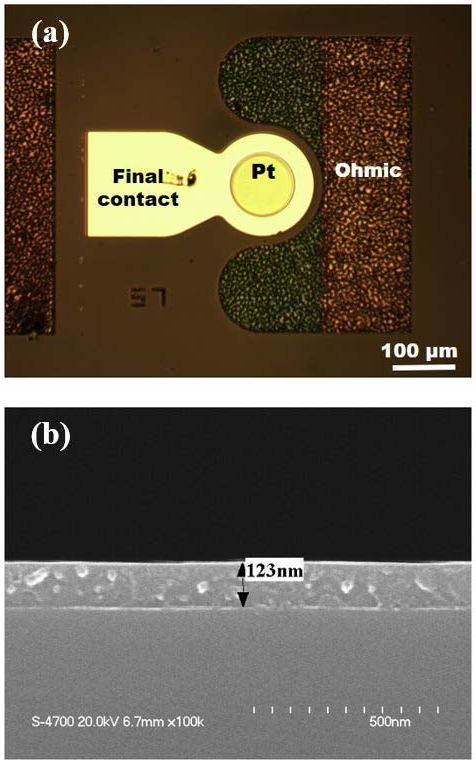

Fig. 2. (a) Optical micrograph of a typical sensor showing the central Pt Schottky contact surrounded by the Ohmic contacts and PMGI encapsulation. (b) Cross-sectional field-emission SEM image of 123 nm thick PMGI layer on top of the silicon substrate.

For some diodes, 123 nm of PMGI was spun-on to the device surface as a moisture barrier, and removed on the contact areas for electrical probing. The PMGI solution was obtained from Microchem (type SF13) and is a photosensitive positive-type resin whose composition was composed of the base polydimethylglutarimide, with cyclopentanone (65–85%) and tetrahydrofurfuryl alcohol (10–15%) to keep the resin in a liquid state. 2 mL of PMGI was diluted with 6.3 ml of cyclopentanone to obtain very thin 123 nm of PMGI layer on the device surface. The diluted PMGI solution was spun-on to the sensors at 2000 rpm for 40 secs and then baked at 180°C for 3 mins in air ambient. Figure 2 (a) shows an optical microscope image of a completed diode with the PMGI layer in place and the figure 2 (b) shows a field emission scanning electron microscope (FE-SEM) cross-sectional image of the PMGI coating on a silicon substrate, showing a nominal thickness of 123 nm. The completed diodes were placed in a test chamber and were exposed to 500 ppm concentration of wet or dry H_2 which is well below the lower flammable limit of hydrogen, but serves as a convenient test concentration. The gas flow rate and injection time were controlled by mass flow controllers in the chamber. Current-voltage (I–V) characteristics of both the bare and coated Schottky diodes in the gas test chamber were measured from 25 to 300°C using an Agilent 4155C semiconductor parameter analyzer. The 100% relatively humid hydrogen gas in nitrogen was produced in an auxiliary line passing through 2 consecutive water bubblers.

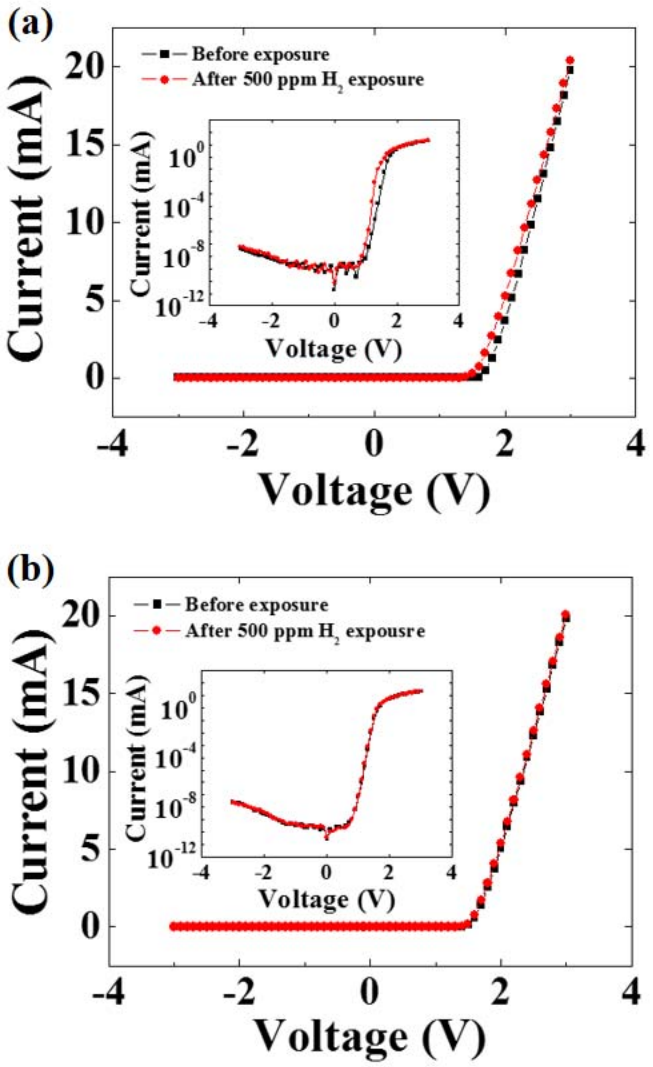

Fig. 3. Forward and reverse I–V characteristics from unencapsulated diodes at 25°C before and after exposure to (a) dry 500 ppm H_2 in N_2 or to (b) humid 500 ppm H_2. The insets show the same data on a semi-log scale.

III. Results and Discussion

Figure 3 (a) shows I–V characteristics on a linear scale with the same data on a log scale in the inset for an unencapsulated Schottky diode measured at 25°C before and during exposure to dry 500 ppm H_2 for 20 secs. The I–V trace shows a significant increase in forward current as a result of the Schottky barrier lowering caused by the hydrogen dissociation in the Pt Schottky gate. By sharp contrast, exposure to the same concentration of H_2 under humid condition shows no obvious change on this scale, as shown in Figure 3(b). This is consistent with the previous reports of suppression of response of Pt or Pd-gated GaN-based diodes to hydrogen when water vapor is present in the ambient. It is known that water molecules on the Pt surface block hydrogen molecules to be adsorbed on the catalytically active sites of Pt [31]. It is also important to note that the I–V characteristics were unaffected by the presence of the PMGI and the same absolute characteristics were obtained when detecting dry hydrogen.

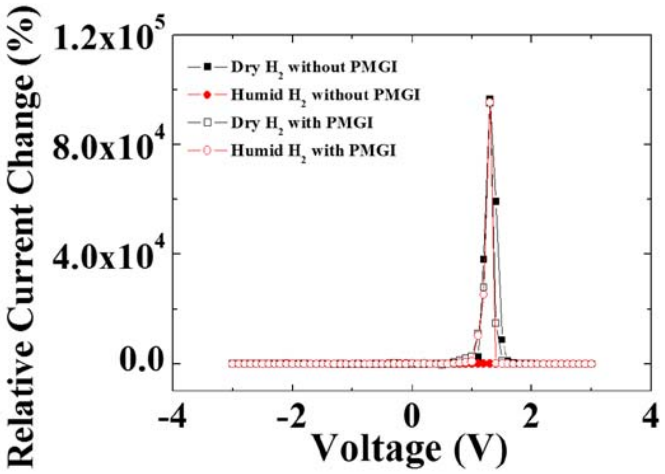

Fig. 4. Relative current change as a function of voltage on Pt-AlGaN/GaN diodes either with or without PMGI encapsulation exposed to dry or humid H_2.

Figure 4 shows the relative current change percentage of both bare and PMGI-encapsulated diodes as a function of bias voltage for exposure to 500 ppm H_2 either dry or with 100% humidity. The relative current change percentage is defined as $\frac{I_H - I_N}{I_N} \times 100\%$, where I_H is the current under hydrogen ambient and I_N is under nitrogen. The maximum relative current changes were 96 400, 95 100, and 95 300% for those of bare diode under dry, and of encapsulated diode under dry and wet hydrogen, respectively. The response of the bare or encapsulated sensors to dry H_2 is almost the same in the magnitude, as discussed above and there was no significant difference between these and the response of the encapsulated diode exposed to wet hydrogen. As we would expect, the un-encapsulated sensors show a greatly reduced sensitivity in the presence of the water vapor content in the hydrogen relative to dry conditions. The maximum relative current change percentage was merely 50% at 1.3V forward bias voltage. This shows that the PMGI encapsulation is completely successful in eliminating this decrease due to the increased humidity level, while still retaining the excellent detection sensitivity.

Figure 5 (a) shows the time-dependent response of the forward current measured at 1.3V bias voltage from an encapsulated diode as 500 ppm dry or humid H_2 at 25°C was cycled into the test chamber multiple times for 5 secs each time and purged with N_2 in between cycles. This again illustrates the strong effect of humidity on the sensitivity of unencapsulated diodes. The unencapsulated diode exhibited a decrease of approximately one ninth of the current change for detection of humid 500 ppm H_2. On the contrary, the PMGI encapsulated diode showed identical current signal for both dry and wet 500 ppm H_2 exposures, as shown in figure 5 (b). Also, the response and recovery behavior of the PMGI encapsulated diodes was not deteriorated by employing the thin PMGI layer, and the devices showed similar temporal characteristics for both dry and wet hydrogen exposures.

A key feature of the use of any moisture barrier material on a sensor is the thermal stability and therefore the device

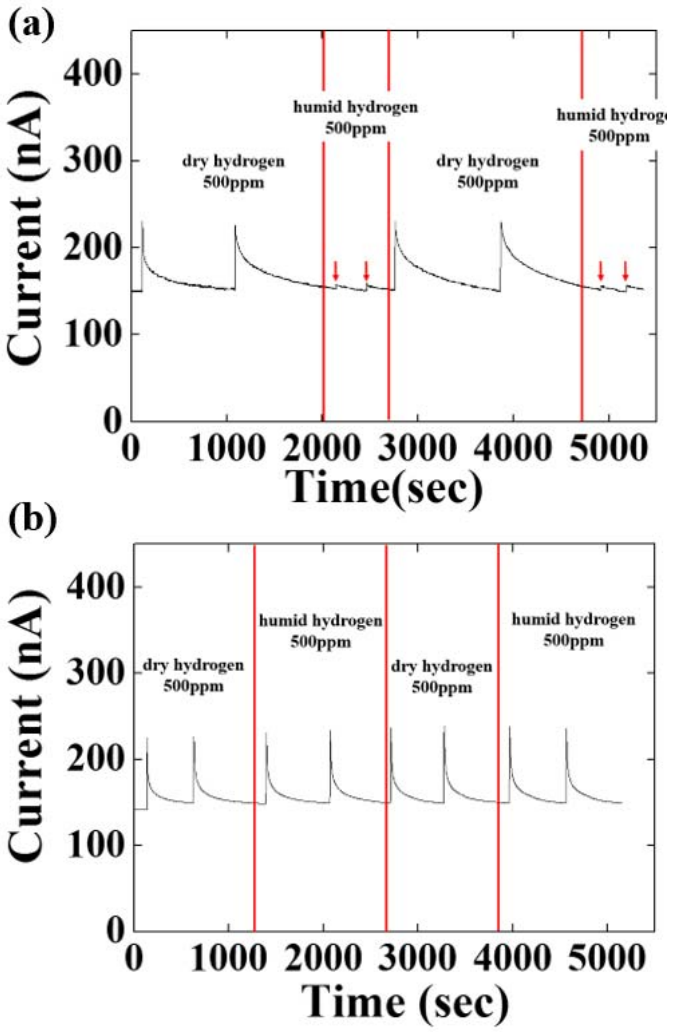

Fig. 5. Time dependence of current change at 1.3 V forward bias for (a) an unencapsulated sensor or (b) PMGI encapsulated diode as a function of cycling the ambient for 5 secs from N_2 to 500 ppm dry or humid H_2 in N_2.

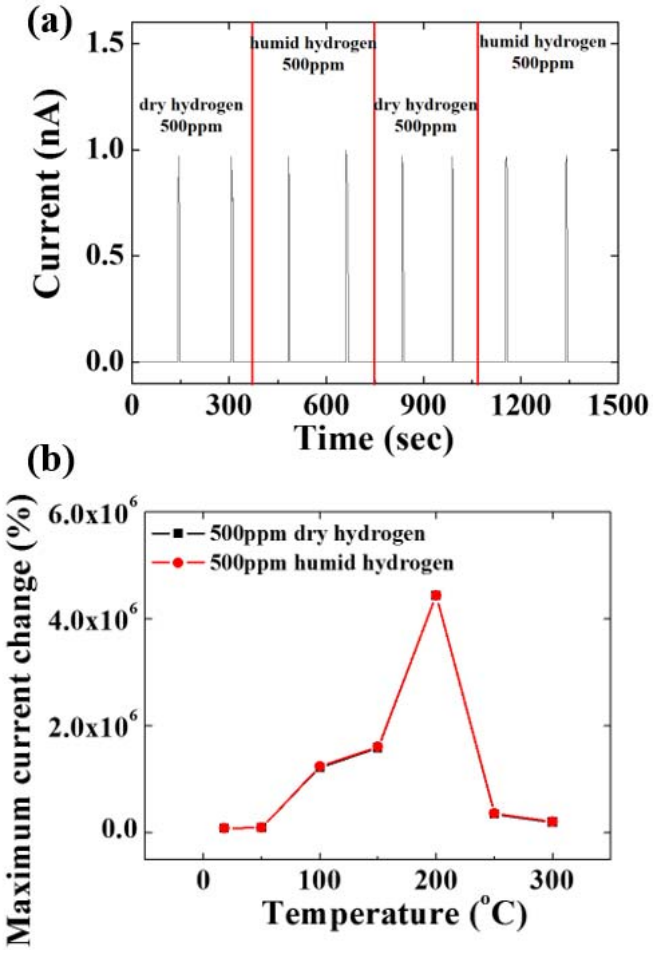

Fig. 6. (a) Time response of the forward current of PMGI encapsulated diodes at 200°C during cycling of 500 ppm dry and wet H_2 for 5 secs each (2 cycles of each) with purging of dry N_2 between each exposure. (b) Maximum relative current change percentage in current as a function of temperature in the encapsulated diodes resulting from exposure to either dry or wet 500 ppm hydrogen.

operating range with the encapsulant in place. In addition, the useful operating temperature range should be defined by the intrinsic sensitivity rather than the stability of the encapsulant. To answer these questions, we tested the response of the encapsulated diodes up to 300°C. Figure 6 (a) shows an example of the time response of the PMGI encapsulated diodes to repeated 5 sec cycles of 500 ppm dry or humid H_2 at 200°C. The sensor with a moisture barrier showed reliable current response to repeated dry and wet H_2 exposures from 25 to 300°C. The maximum relative current change percentage as a function of temperature in the encapsulated diode resulting from exposure to either dry or wet 500 ppm hydrogen is plotted in Figure 6 (b). Identical maximum relative current change percentages of the PMGI sensor were observed in this measurement temperature range. The sensor response peaks around 200°C and decreases at high temperatures. This is well below the degradation point of the PMGI (335°C) so that is not a limiting factor for the sensor useful operating range. At low temperatures, the sensitivity appears to be limited by the dissociation efficiency of the hydrogen, while above 200°C, the hydrogen-induced dipole layer is not stable enough to have a maximum influence on the barrier height of the catalytic Pt Schottky metal contact [39].

Figure 7 shows an Arrhenius plot of relative current change to 500 ppm H_2 exposures to PMGI encapsulated sensor, which was used to investigate the activation energy of H_2 detection. For the low temperature of 25–200°C, the extracted activation energy from the slope of the plot was 0.28 eV. This is the energy of the rate-limiting step in the formation of a charge at the interface between Pt and AlGaN from diffused hydrogen atoms through Pt Schottky layer after H_2 molecules are adsorbed and dissociated on catalytic Pt surface, and the values are similar to the activation energy of conventional Pt or Pd-AlGaN/GaN diode sensors without a moisture barrier, 0.18–0.26 eV [32,40]. Figure 8 shows that the PMGI encapsulated sensor were completely selective at 25°C for 500 ppm H_2 over N_2 (100%), CH_4 (4%), CO (0.1%), NO_2 (0.05%), CO_2 (10%), and O_2 (100%) under the same detection conditions as used for the H_2. The exposure time

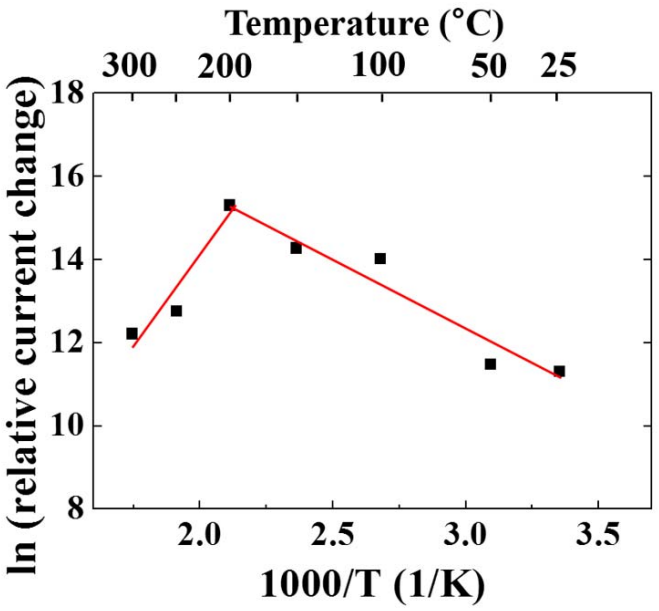

Fig. 7. Arrhenius plot of relative current change to 500 ppm H_2 exposures to PMGI encapsulated sensor.

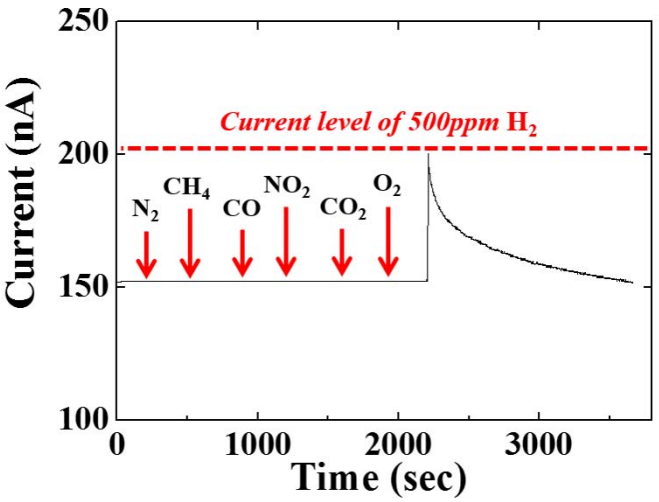

Fig. 8. Response of sensor to 5 sec sequential exposures of N_2 (100%), CH_4 (4%), CO (0.1%), NO_2 (0.05%), CO_2 (10%), and O_2 (100%) at 25°C. The bias voltage was fixed at 1.3 V.

to each of these gases was for 5 secs each and the forward bias voltage on the diode was 1.3 V.

IV. Summary and Conclusions

These results demonstrate that PMGI encapsulation enables Pt-gate AlGaN/GaN hydrogen sensor to eliminate the deleterious effect of moisture in the sensing ambient on the detection of hydrogen, maintaining the same sensitivity level to the device without a moisture barrier. The PMGI coated sensors are completely selective to the presence of other common gases such as N_2, CH_4, CO, NO_2, CO_2, and O_2 at 25°C. Also, by using thermally stable PMGI layer at high temperature, the device showed robust sensing characteristics up to 300°C. Since the PMGI is commonly available and easily applied to the semiconductor surface by simple spin-on processing, it represents a good choice as a moisture barrier layer on the hydrogen sensor applications.

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