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# Enhanced Sensing Performance of MISiC Schottky-Diode Hydrogen Sensor by Using HfON as Gate Insulator

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Abstract-MISiC Schottky-diode hydrogen sensor with HfON gate insulator fabricated by NO nitridation is investigated. The hydrogen-sensing characteristics of this novel sensor are studied by doing steady-state and transient measurements at different temperatures and hydrogen concentrations using a computer-controlled measurement system. Experimental results show that this novel sensor can rapidly respond to hydrogen variation and can give a significant response even at a low H<sub>2</sub> concentration of 48-ppm, e.g., a sensitivity of 81% is achieved at 450 °C and 2.5 V, which is two times higher than its  $HfO_2$  counterpart. The enhanced sensitivity of the device should be attributed to a remarkable reduction of the current of the sensor before hydrogen exposure by the NO nitridation because the NO nitridation can passivate the O vacancies in the insulator and facilitate the formation of a  $SiO_2$  interlayer to suppress the leakage current associated with high-k materials.

Index Terms— $HfO_2$ , hydrogen sensors, nitridation, Schottky diode, silicon carbide.

# I. INTRODUCTION

YDROGEN is widely used in our daily life. However, hydrogen is a very dangerous gas because it can cause a serious explosion if a spark is present. It can also weaken metals internally causing serious engineering problems. In order to prevent these accidents, hydrogen sensors have become increasingly important in leakage detection for industrial fabrication processes and hydrogen-fueled vehicles. Increasingly strict emissions regulations and heightened environmental awareness also make hydrogen sensors play an essential role in exhaust diagnosis and flue gas monitoring. The recent focus of SiC-based gas sensors has been on Metal-Insulator-SiC (MISiC) gas sensors because these sensors can have better sensitivity, stability and selectivity. Silicon dioxide has been widely used as the gate dielectric of metal-oxide semiconductor field-effect-transistors (MOSFETs) for more than three decades because it can be easily fabricated by conventional thermal oxidation and patterned by

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photolithography. However, pure  $SiO_2$  is not a good insulator for making MISiC Schottky-diode hydrogen sensors because the required insulator is very thin and the use of the traditional thermal oxide  $(SiO_2)$  will create a lot of problems. For example, intermixing of materials at the interfaces due to poor interfacial diffusion barrier (weak Si-Si and Si-O bonds of the oxide) can consume the insulator, and direct tunneling may occur in the thin film. Therefore, silicon oxynitrides (SiON) and high-k dielectrics have been extensively studied as an alternative to  $SiO_2$ to reduce the gate leakage current in the devices.

Several high-k dielectrics have been employed to fabricate hydrogen sensors, for example, TiO<sub>2</sub> [1], Ga<sub>2</sub>O<sub>3</sub>-ZnO [2],  $WO_3$  [3], and  $HfO_2$  [4]. The quality of the gate insulator as well as metal/insulator and insulator/substrate interfaces can greatly influence the sensor performance. The as-deposited high-k films are usually loosely packed and contain impurities and defects such as oxygen vacancies, oxygen interstitials and/or oxygen deficiency [5]. These defects and impurities will cause transient charge trapping in the high-k dielectric and leakage current [6]. In order to achieve high-quality devices, the deposited high-k films are always followed by surface passivation treatments such as annealing in  $N_2$  and ultraviolet ozone. Nitrogen incorporation is also a promising passivation method because nitrogen atoms are more mobile than oxygen atoms and hence can easily couple with oxygen vacancies in the high-k dielectrics, resulting in less mobile  $NO^-$  molecules and a remarkable reduction of leakage current [7]–[10]. There are various methods for N incorporation into Hf-based oxides, e.g., N plasma, N-bearing precursor chemistry,  $N_2^+$  implantation, and sputtering under  $N_2$  flow. In this work, a technique of nitridation in NO is used to incorporate nitrogen into  $HfO_2$  to form HfON as the gate insulator for the hydrogen sensors to reduce the interface states and oxide charges of the film and hence improve the device performance. This paper will first describe the fabrication procedure of the hydrogen sensors and the measurement methodology. Then, the effects of NO nitridation on the hydrogen-sensing characteristics of MISiC Schottky sensors are reported and analyzed.

# **II. EXPERIMENTAL DETAILS**

N-type (0001) Si-face 4H-SiC wafer, manufactured by CREE research, was used in this study. The SiC wafer had a 5- $\mu$ m epitaxial layer grown on heavily doped substrate. The doping level of the epitaxial layer was 5-6 ×10<sup>15</sup> cm<sup>-3</sup>. The wafer was cleaned using the conventional RCA method followed by a 60 s dipping in 5% hydrofluoric acid to remove the native oxide. The

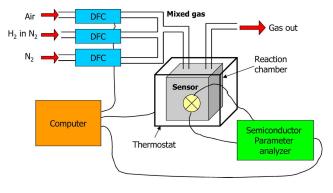


Fig. 1. Computer-controlled measurement system.

wafer was then loaded into a Denton vacuum LLC Discovery 635 sputterer, which was then pumped down to  $2 \times 10^{-6}$  Torr. HfO2 was then deposited at room temperature by DC sputtering of hafnium metal (99.99% purity) with a sputtering power of 17.52 W in a mixed Ar/O<sub>2</sub> ambient (Ar to O<sub>2</sub> ratio 4:1) for 20.9 min. An electrode consisting of 100-nm Pt with a diameter of 0.5 mm was then deposited on the wafer by DC-magnetron sputtering through a stainless steel shadow mask. The sample then underwent a nitridation in NO gas (1000 ml/min) by loading them into a furnace at 650 °C for 5 min to incorporate nitrogen into the HfO<sub>2</sub> film and also densify the insulator layer (denoted as HfON sample). For the purpose of comparison, a control sample underwent  $N_2$  annealing (denoted as HfO<sub>2</sub>) sample) at 650 °C for 5 min was also fabricated. The final oxide thicknesses of the HfON and HfO2 samples were 5.81 and 5.51 nm as measured by an ellipsometer. The steady-state and transient response measurements were performed using a computer-controlled measurement system (see Fig. 1). The measurement system consists of 5 main parts: 1) a thermostat; 2) a semiconductor parameter analyzer; 3) digital gas flow controllers (DFCs); 4) a reaction chamber; and (5) a computer. The test samples were mounted at the middle of the reaction chamber and gases were injected into the chamber through the DFCs. The reaction chamber was placed inside the thermostat so that a constant-temperature environment was provided for investigating the hydrogen-sensing characteristics of the sensors at stable temperature. The thermostat, the HP 4145B semiconductor parameter analyzer and the DFCs were all connected to a computer and were controlled by computer programs. The computer programs could provide different measurement conditions by altering the temperature of the thermostat and the flow rates of different gases, and measurement results were automatically stored in the computer.

## **III. RESULTS AND DISCUSSION**

Fig. 2 shows the I-V curves of the HfON sample measured in air and in different hydrogen concentrations (48, 238, and 810 ppm) at 50 °C and 150 °C. In order to obtain a steady response, the I-V curves for H<sub>2</sub> ambient are measured at 200 s after the introduction of the gases. The sensor can give a significant response even at low hydrogen concentration, e.g., 48-ppm H<sub>2</sub> in N<sub>2</sub> and the hydrogenation effect becomes more obvious at 150 °C. The I-V curves shift to the left upon exposure to H<sub>2</sub>

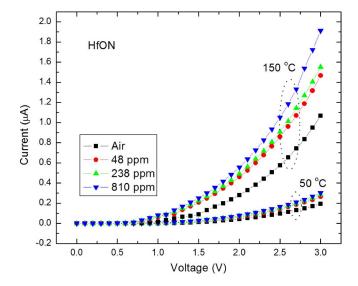


Fig. 2. I-V curves of the HfON sample measured in air and in different hydrogen concentrations (48, 238, 810 ppm) at 50 °C and 150 °C.

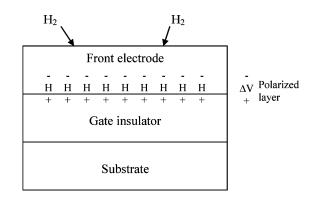


Fig. 3. Schematic representation of a MISiC Schottky-diode hydrogen sensor. A positive bias is applied to the front electrode.

due to the formation of a polarized layer at the electrode-insulator interface. When hydrogen-containing molecules come to the front electrode, they dissociate at the surface of the electrode and form hydrogen atoms. These hydrogen atoms then diffuse through the electrode to the surface of the insulator and form a polarized layer at the electrode-insulator interface. The polarized hydrogen layer, as shown in Fig. 3, can increase the electron concentration of the metal and hence its Fermi level near the insulator layer. As a result, the energy barrier at the metal-insulator interface is reduced, inducing an increase of electron transport [11]–[13].

Fig. 4 shows the I-V curves of the HfO<sub>2</sub> and HfON samples measured in air and in different hydrogen concentrations (48, 238, 810 ppm) at 450 °C. The HfON sample has larger turn-on voltage than the HfO<sub>2</sub> sample because it has a thicker gate insulator. The thicker insulator in the HfON sample is due to the NO nitridation process, which can oxidize the substrate to form a SiON interlayer. The current increase in H<sub>2</sub> ambient is governed by the polarized layer formed at the metal/insulator interface. A stronger polarized layer causes a larger barrier-lowering effect and hence a larger current variation. The table inside Fig. 4 shows the current shift  $\Delta I$  of the HfO<sub>2</sub> and HfON samples at 2.5 V under different H<sub>2</sub>-containing environments.

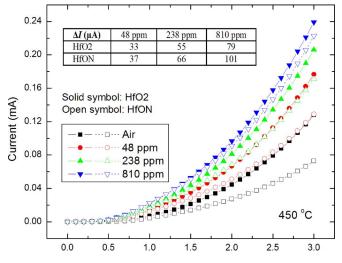


Fig. 4. I-V curves of the HfO<sub>2</sub> and HfON samples measured in different hydrogen concentrations at 450 °C.

It is found that the current variations of both studied devices increase with H<sub>2</sub> concentration. In addition,  $\Delta I$  of the HfON sample is larger than that of the HfO<sub>2</sub> sample under all studied hydrogen-containing ambients. At 2.5 V, 810-ppm H<sub>2</sub> in N<sub>2</sub>, the current shift of the HfON sample is 28% larger than that of its HfO<sub>2</sub> counterpart. Interface traps and oxide charges can screen the effect of the polarized layer formed by the hydrogen atoms at the electrode-insulator interface [14], resulting in less effective dipoles at the interface and hence smaller modulation of the Schottky barrier height. The improved sensor response of the HfON sample is due to the better insulator and HfON/SiC interface properties associated with nitridation-induced passivation, resulting in more effective dipoles at the electrode/insulator interface and hence a wider range of barrier-height modulation, as indicated in Fig. 5.

Based on the thermionic emission conduction mechanism of Schottky diode, for a forward bias V > 3kT/q, the *I*–V characteristics of a Schottky diode are given by [3], [15], [16]

$$I = I_o \exp\left(\frac{qV}{nkT}\right) \tag{1}$$

where k is the Boltzmann constant, T the temperature in K, n the ideality factor, and  $I_o$  the saturation current defined as

$$I_o = AA^{**}T^2 \exp\left(-\frac{\phi_b}{\phi_T}\right) \tag{2}$$

where A is the junction area,  $A^{**}$  the effective Richardson constant (194 A/cm<sup>2</sup>K<sup>2</sup> for SiC [17]),  $\phi_b$  the barrier height of the Schottky diode and  $\phi_T = kT/q$ . Plot of ln I versus V (in air and in 810 ppm H<sub>2</sub> in N<sub>2</sub>) of the HfON sample at 450 °C is shown in Fig. 6. The graph shows good linearity indicating that the current conduction mechanism of the Schottky-diode hydrogen sensor with HfON as gate insulator is controlled by thermionic emission. A relation between the current and temperature of the HfON sample is depicted in Fig. 7. Both the currents measured under hydrogen environment and air increase with temperature because at high temperature, more electrons have sufficient energy to overcome the potential barrier and flow through the Schottky-diode.

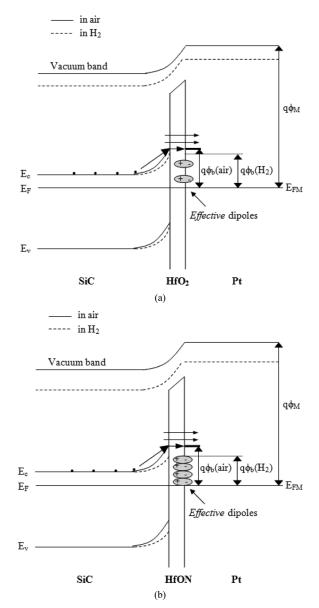


Fig. 5. Schematic energy-band diagram for MISiC Schottky-diode hydrogen sensors with (a) HfO<sub>2</sub> and (b) HfON as gate insulator upon exposure to H<sub>2</sub>. Electrons in the conduction band of the substrate (SiC) have to overcome an energy barrier  $\phi_b$  and then tunnel through the thin insulator to reach the gate electrode (Pt).

Presented in Fig. 8 is the sensor response of the  $HfO_2$  and HfON samples upon exposure to air and 800-ppm  $H_2$  in  $N_2$  at 450 °C. The applied forward voltage is 2.5 V. The HfON sample can give a larger response than the  $HfO_2$  sample. The response/recovery time, which is defined as  $e^{-1}$  times the final steady-state value, can also be obtained from the transient-response curve. The response (recovery) time is 2.2 (18.5) and 10.5 (16) s for the  $HfO_2$  and HfON samples respectively. The recovery time of the samples is longer than the response time. This means that more time is needed for hydrogen atoms to diffuse out of the electrode/insulator interface to the electrode surface and then recombine together to form hydrogen gas.

The sensitivity of the HfON sample upon exposure to different H<sub>2</sub> concentrations in N<sub>2</sub> at 50 °C, 150 °C, and 450 °C is compared with that of the HfO<sub>2</sub> sample in Fig. 9. The sensitivity is defined as  $(I_{H_2} - I_{air})/I_{air}$ , where  $I_{H_2}$  and  $I_{air}$  are

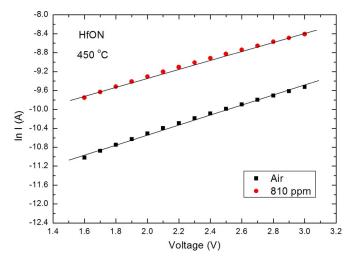


Fig. 6. I-V characteristics of the HfON sample at 450 °C in (1) air and (2) 810 ppm H<sub>2</sub> in N<sub>2</sub>.

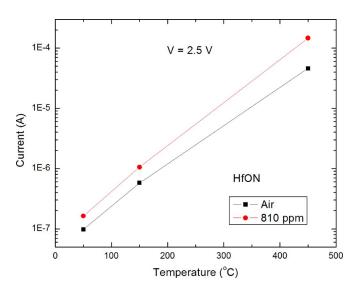


Fig. 7. Current of the HfON sample measured under hydrogen environment and air as a function of temperature.

currents measured under hydrogen environment and air respectively. The applied voltage is 2.5 V. The sensitivities of both samples increase with hydrogen concentration and temperature. This phenomenon can be explained as follows. When temperature increases, the hydrogen under higher pressure will bombard the surface of the electrode more frequently. Hence, more hydrogen molecules can absorb at the surface of the electrode and decompose into hydrogen atoms. More hydrogen atoms diffuse through the metal layer to the metal/insulator interface to form a polarized layer. This stronger polarized layer gives a larger  $\phi_b$  reduction, thus increasing the sensitivity. Increasing H<sub>2</sub> concentration can also cause more hydrogen atoms absorb at the metal-insulator interface to produce a stronger dipole layer, resulting in more barrier-height lowering, and hence larger current shift, thus higher sensitivity. At 450 °C, the HfO<sub>2</sub> sample has a sensitivity of 42% at 48 ppm H<sub>2</sub> and gets saturated gradually at 800 ppm with a sensitivity of about 99%. However, the HfON sample can give a higher sensitivity of 81% at 48-ppm  $H_2$ , which increases to 219% at 800 ppm and does not saturate

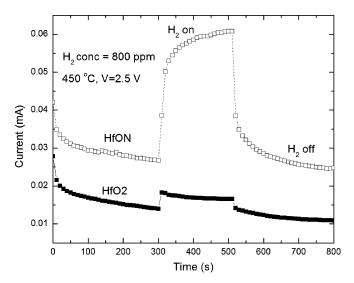


Fig. 8. Response of the  $\rm HfO_2$  and HfON samples upon exposure to air and 800-ppm  $\rm H_2$  in  $\rm N_2$  at 450  $^{\circ}C.$ 

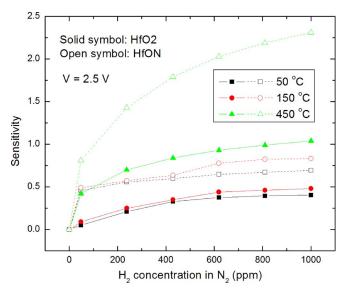


Fig. 9. Sensitivity of the HfO<sub>2</sub> and HfON samples (bias voltage = 2.5 V) versus H<sub>2</sub> concentration in N<sub>2</sub> at 50 °C, 150 °C, and 450 °C.

even at 1000 ppm. The enhanced sensitivity of the HfON sample is due to its smaller  $I_{air}$  compared with the HfO<sub>2</sub> sample. O and N species decomposed from the NO gas during the nitridation process can reduce the defects in the high-k dielectric and also facilitate the growth of a SiON interlayer between the high-kdielectric and the substrate. The SiON interlayer can improve the interface quality and block the injection of electrons from SiC into the high-k insulator [18]. In addition, the incorporation of N in the HfON sample can passivate the O vacancies in the insulator and remove the electron leakage path, resulting in significantly reduction of the leakage current [19]. NO nitridation forms a nitrogen-terminated passivation layer at the surface of the SiC wafer to help remove the dangling bonds there. Moreover, nitrogen can reduce the interfacial strain, and remove fixed oxide charges, carbon-related interface traps [20], carbon atoms, and clusters at the interface [21]. With better interfacial properties, the HfON sample has a better response to external stimuli.

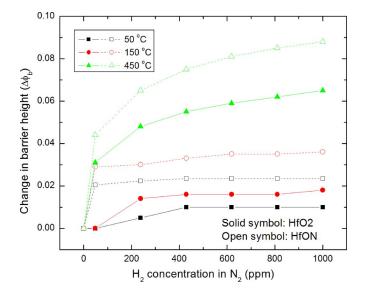


Fig. 10. Change in barrier height of the samples upon exposure to different  $\rm H_2$  concentrations in  $\rm N_2$  at 50 °C, 150 °C, and 450 °C.

Fig. 10 shows the change in the barrier height of the HfON and  $HfO_2$  samples upon exposure to different  $H_2$  concentrations. The barrier height is calculated using the following formula:

$$\phi_b = -\phi_T \ln\left(\frac{I_o}{AA^{**}T^2}\right) \tag{3}$$

 $I_{0}$  (in air and in H<sub>2</sub>) can be calculated from the corresponding y-intercept of the  $\ln I$  versus V plot. When hydrogen concentration increases, the barrier height of the sensor decreases and thus the barrier-height variation defined as  $\phi_b$  (air) –  $\phi_b$  (H<sub>2</sub>) increases. It is because with more hydrogen molecules dissociated at the front electrode, more hydrogen atoms can adsorb at the electrode-insulator interface to form a polarized layer. This stronger polarized layer causes a larger barrier-lowering effect. On the other hand, interface traps and oxide charges can screen the effect of the polarized layer [14]. The wider range of barrier-height variation in the HfON sample is due to the effect of NO nitridation that improves the dielectric and interfacial properties of the sensor. Temperature can also affect the variation of barrier height. The change of the barrier height increases when the temperature increases from 50 °C to 450 °C because at higher temperature, more adsorbed hydrogen can diffuse through the electrode to the electrode/insulator interface and hence produce a stronger dipole layer to lower the Schottky barrier more.

According to the Langmuir isotherm equation, the hydrogen coverage  $\theta$  at the interface under steady-state condition can be written as [22], [23]

$$\frac{\theta}{1-\theta} = k_o \sqrt{P_{\rm H_2}} \tag{4}$$

where  $k_o$  is the temperature-dependent constant which depends on the difference of adsorption between the surface and interface and  $P_{\rm H2}$  is the hydrogen partial pressure. The change in voltage across the hydrogen dipole layer  $\Delta V$  is proportional to the hydrogen coverage (i.e.,  $\Delta V \propto \theta$ ) and the proportionality

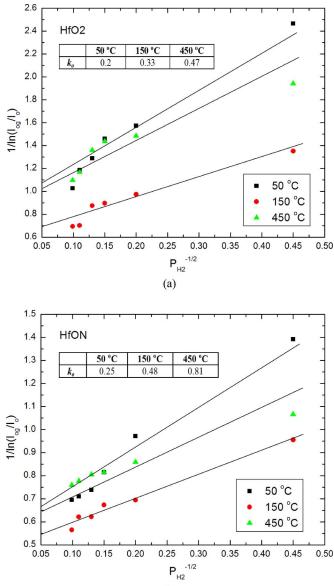


Fig. 11. Steady-state reaction kinetic analysis for hydrogen absorption of the (a)  $\rm HfO_2$  and (b)  $\rm HfON$  samples, at 50 °C, 150 °C, and 450 °C.

(b)

constant is  $\Delta V_{\text{max}}$  (maximum voltage change at a fixed temperature). By substituting  $\theta = \Delta V / \Delta V_{\text{max}}$  into (4)

$$\frac{1}{\Delta V} - \frac{1}{\Delta V_{\text{max}}} = \frac{1}{k_o \Delta V_{\text{max}} \sqrt{P_{\text{H}_2}}}.$$
 (5)

By using the equation  $I = I_o \exp(qV/nkT)$ , (5) can be written as

$$\frac{1}{\ln\left(\frac{I_{og}}{I_o}\right)} = \frac{1}{\ln\left(\frac{I_{og_{\max}}}{I_o}\right)} \left(1 + \frac{1}{k_o\sqrt{P_{H_2}}}\right) \tag{6}$$

where  $I_{og}$  and  $I_{ogmax}$  are, respectively, the saturation current and maximum saturation current of the sensor in hydrogen environment. Fig. 11 shows the plot of  $1/\ln (I_{og}/I_o)$  versus  $(1/P_{\rm H2})^{1/2}$  for the HfO<sub>2</sub> and HfON samples at 50 °C, 150 °C, and 450 °C. From the slope and y-intercept of the plot, the  $k_o$ value is calculated and shown in the inset tables of Fig. 11. It is

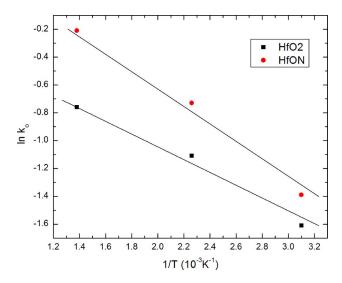


Fig. 12. In  $k_o$  as a function of the inverse of temperature for the HfO<sub>2</sub> and HfON samples.

found that the  $k_o$  values of both samples increase as the temperature increases. This implies that more hydrogen atoms can adsorb at the Pt/HfO<sub>2</sub> or Pt/HfON interface at higher operating temperature [24]. According to the van't Hoff equation [25]

$$\ln k_o = -\frac{\Delta H^o}{RT} + \frac{\Delta S^o}{R} \tag{7}$$

 $\Delta H^o$  is the enthalpy change;  $\Delta S_o$  is the entropy change; and R is the gas constant (8.314472 JK<sup>-1</sup>mol<sup>-1</sup>). Fig. 12 illustrates a plot of ln  $k_o$  versus 1/T for the studied devices. From the slope of the plot, the enthalpy change of the HfO<sub>2</sub> and HfON samples is determined as 4.1 kJ/mol and 5.7 kJ/mol, respectively. Since the hydrogen adsorption process is endothermic, it is favorable for high-temperature detection.

#### IV. CONCLUSION

A novel MISiC Schottky-diode hydrogen sensor with high-k dielectric HfON as gate insulator has been fabricated and studied. The influences of hydrogen concentration and temperature on the hydrogen-sensing performance of the device are investigated. Experimental results show that the sensor demonstrates higher sensitivity than its HfO<sub>2</sub> counterpart at different hydrogen concentrations and temperatures. Incorporating N into HfO2 by NO nitridation can result in fewer interface states and oxide charges and better interfacial diffusion barriers. In addition, NO nitridation can increase the thickness of the insulator to give a smaller  $I_{air}$  and hence higher sensitivity. It is also found that like other Schottky-diode hydrogen sensors, the current conduction of the sensor is governed by the thermionic-emission mechanism. In conclusion, HfON is a promising gate insulator for making high-performance MISiC Schottky-diode hydrogen sensor.

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