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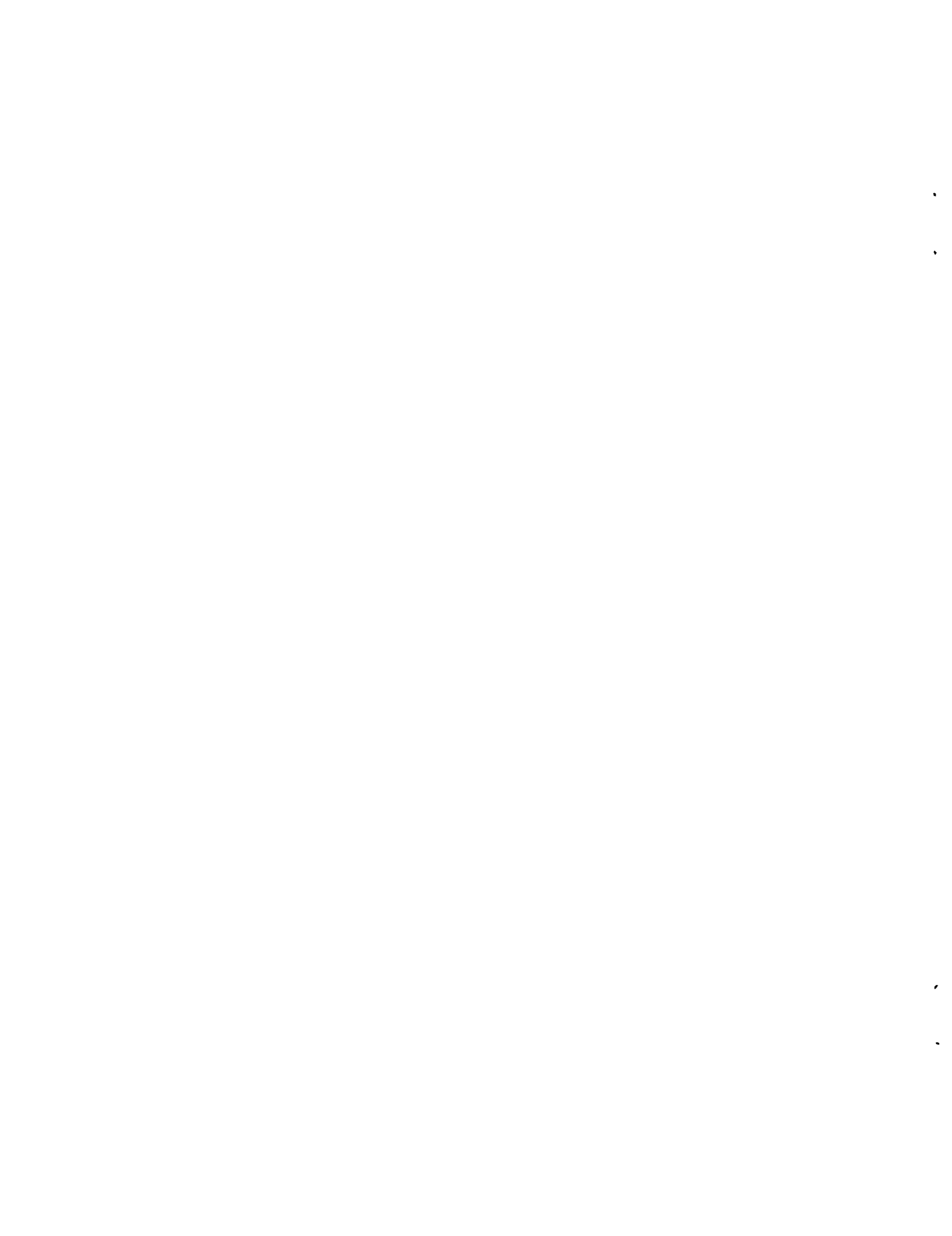
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ABSTRACT

Electronic grade Silicon Carbide (SiC) is a ceramic material which can operate as a semiconductor at temperatures above 600°C. Recently, SiC semiconductors have been used in Schottky diode gas sensor structures. These sensors have been shown to be functional at temperatures significantly above the normal operating range of Si-based devices. SiC sensor operation at these higher temperatures allows detection of gases such as hydrocarbons which are not detectable at lower temperatures. This paper discusses the development of SiC-based Schottky diode gas sensors for the detection of hydrogen, hydrocarbons, and nitrogen oxides (NO_x). Sensor designs for these applications are discussed. High sensitivity is observed for the hydrogen and hydrocarbon sensors using Pd on SiC Schottky diodes while the NO_x sensors are still under development. A prototype sensor package has been fabricated which allows high temperature operation in a room temperature ambient by minimizing heat loss to that ambient. It is concluded that SiC-based gas sensors have considerable potential in a variety of gas sensing applications.

INTRODUCTION

The requirements of aeronautic, aerospace, and commercial applications increasingly demand component parts to operate in harsh environments under conditions which exceed the capabilities of traditional materials. Due to their inherent stability, this has led to the development of ceramic materials as structural materials, electronic semiconductors, and sensor components. Silicon carbide (SiC) is a ceramic material which can be used for all three functions. For many years, SiC has been used as a structural material due to its

durability and hardness. SiC is also becoming an electronic material of considerable importance (1). Due to its wide band gap and low intrinsic carrier concentration, SiC operates as a semiconductor at temperatures significantly higher than that possible with silicon (Si) semiconductor technology. SiC is now available commercially and processing difficulties associated with its production are being addressed.

Recently, SiC has been included as a sensor component in gas sensing applications. In particular, the capabilities of SiC as a semiconductor in gas sensing metal-insulator-semiconductor (MIS) or metal-semiconductor (MS) structures have been investigated. For example, Lundstrom et al. have investigated MIS capacitor structures with SiC as the semiconductor. SiC-based capacitors using platinum (Pt) as the gas sensitive metal have detected hydrogen concentrations as low as 2.5 ppm and have operated at temperatures as high as 800°C (2). The capacitors respond to hydrocarbons such as methane, ethane, and propane, as well as NO_x. Response times of less than 100 ms have been obtained (3-4). Although the majority of the work done by this group has concentrated on the MIS capacitor structure, the development of a Schottky diode structure has also been reported (5-6).

In contrast to the MIS capacitive sensor development, the emphasis of the work at NASA Lewis Research Center (LeRC) and Case Western Reserve University (CWRU) has centered on the effort to develop a Schottky diode sensor using SiC as the semiconductor. The advantage of the Schottky diode structure is its high sensitivity (i.e. large change in signal) for low concentration measurements. The ability to detect low concentrations of gases with a strong change in signal is especially important in, for example, safety monitoring, emissions measurements, and chemical process control. The ambient environments for each of these applications may vary significantly.

The purpose of this paper is to give an overview of the development of SiC-based gas sensors at NASA LeRC and CWRU. First, we will discuss sample preparation and the gas sensor testing system at NASA LeRC. We will then discuss the development of hydrogen and hydrocarbon sensors. Specifically, the properties of Schottky diodes composed of palladium (Pd) directly deposited on SiC will be discussed. The long term stability of these diodes at high temperature will also be briefly discussed. An overview of the sensor packaging effort and the use of SiC-based sensors to detect nitrogen oxides (NO_x) will then be given. It is concluded that SiC-based gas sensors with a Schottky diode configuration have excellent potential to provide very sensitive measurements in a variety of environments, but further development work is necessary.

DEVICE FABRICATION AND TESTING

Palladium MS Schottky diodes on SiC (Pd/SiC) were prepared for hydrogen and hydrocarbon detection in the following manner. An 4-5 μm thick 4H or 6H-SiC epilayer was grown by chemical vapor deposition on a commercially available 4H or 6H silicon-face SiC substrate (7-8). Approximately 400 angstroms of Pd metal was sputter

deposited onto the as-grown SiC epilayer surface. Circular Pd Schottky patterns of diameter 200 μm were then patterned by a lift-off technique. Aluminum (Al) was then sputtered onto the bottom of the wafer to form a backside electrical contact.

The facility used for sensor testing can supply a continuous flow of gaseous hydrogen, hydrocarbons, helium, nitrogen, or air, either individually or as a mixture, to a chamber containing the sensor under test. Computer-controlled mass flow controllers give a continuous gas flow at a range of flow rates from 0 to 4000 standard cubic centimeters per minute (sccm). A three-way valve allows the gas to bypass the test chamber and go directly to the vent. This feature allows the mass flow controllers to be stabilized without flowing gases through the test chamber. The composition of the gas in the chamber is monitored by a mass spectrometer. The mass spectrometer provides an independent measure of the relative concentration of the gases in the test chamber as a function of time.

The Pd/SiC Schottky diodes were characterized in the test chamber containing a probing station and a heated sample stage. As shown in Figure 1, the SiC substrate rested on a gold-covered alumina substrate so that the Al-covered backside of the chip made contact with the gold. Sample contact was made using tungsten probes. One probe made contact with the diodes' patterned Pd surface while another probe made contact with the gold and thus the backside of the chip. The alumina substrate rested on the hot stage whose temperature was controlled from room temperature to 425 $^{\circ}\text{C}$.

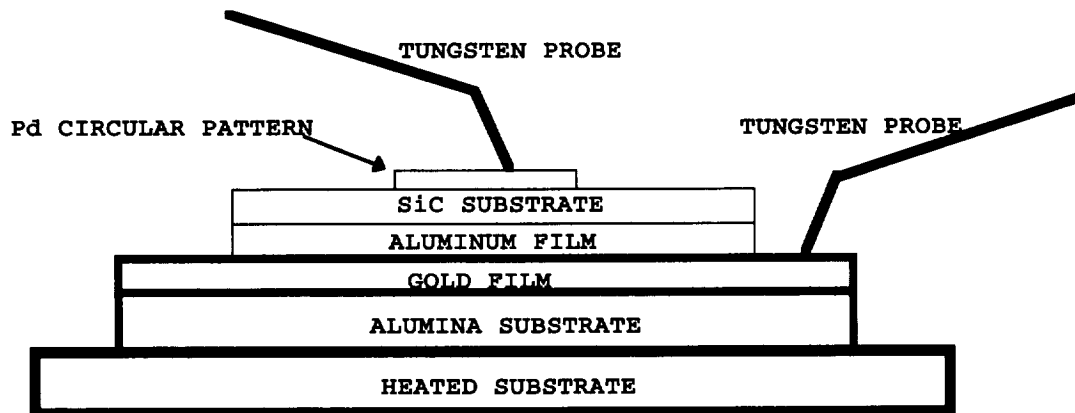


Figure 1. Schematic of testing system for Pd/SiC Schottky diode (not to scale).

The electrical properties of the Pd/SiC Schottky diodes were characterized using capacitance and current measurements. The measurement of the diode's capacitance was conducted as follows: The response of the diode capacitance measured at 0 V with time (C-t) was used to characterize the time dependent behavior of the system upon exposure to various gases. The barrier height of the diode was determined using capacitance-voltage (C-V) measurements by finding the intercept on the voltage axis of the plot $1/C^2$

vs. V (9). Corresponding current-time (I-t) and current-voltage (I-V) measurements were also taken in separate tests. The forward voltage across the diode was held constant and the current was measured as a function of time for the I-t measurements. The forward voltage was chosen in order to maximize the diode response to the hydrogen-bearing gas and to minimize series resistance effects.

HYDROGEN AND HYDROCARBON SENSOR DEVELOPMENT

Sensor Characterization

In order to understand the capabilities of SiC-based Schottky diode using Pd or a Pd alloy, we have extensively studied the properties of diodes composed of Pd directly deposited on SiC. We have chosen Pd-based systems for hydrogen and hydrocarbon detection due to the superior ability of Pd to absorb hydrogen and the substantial previous knowledge of Pd and its alloys in hydrogen detection (10-13). The simplest Pd with SiC system is Pd directly deposited on SiC. Direct contact between the catalytic metal and the semiconductor allows changes in the catalytic metal to have maximum effect on the semiconductor. Studies of this baseline system help determine limits of diode sensitivity, potential material interactions between Pd and SiC, and whether a barrier layer is necessary between the Pd and SiC for long-term sensor stability.

The proposed mechanism for hydrogen detection in SiC-based devices using Pd is the same as that commonly accepted for Pd on Si devices: the dissociation of hydrogen on the metal surface leads to the formation of a dipole layer composed of hydrogen at the metal-semiconductor or metal-insulator interface. This dipole layer changes the work function of the Pd and affects the electronic properties of the device in proportion to the amount of hydrogen and other gas species (especially oxygen) present in the surrounding ambient atmosphere (11). The dissociation of the hydrocarbons is thought to release hydrogen and it is predominately the hydrogen which is detected. Thus, for hydrocarbon detection to occur, the hydrocarbon must dissociate on the metal surface (14-15).

Evidence for this mechanism of hydrogen and hydrocarbon detection is given in Figure 2. The inset of the figure shows the detection of hydrogen and hydrocarbons at 400°C: The diode is first exposed to air for 10 minutes, nitrogen for 40 minutes, followed by 300 ppm of hydrogen in nitrogen (N_2/H_2) for 40 minutes and then 10 minutes of nitrogen and 10 minutes of air. The second and the third cycles are the same as the first except that the 300 ppm of hydrogen is replaced by 300 ppm propylene in nitrogen (N_2/C_3H_6), and then 300 ppm of propylene in nitrogen and 1% oxygen ($N_2/C_3H_6/O_2$) respectively.

The inset of Figure 2 shows a small change in the capacitance as the ambient is changed from air to N_2 . A significant change in capacitance is noted with exposure to both hydrogen and propylene. The response decreases as the hydrogen-bearing mixture is changed from N_2/H_2 , to N_2/C_3H_6 , and then to $N_2/C_3H_6/O_2$. These results are reflected in

Figure 2 where the corresponding curves of $1/C^2$ vs. V taken in air, N_2/H_2 , N_2/C_3H_6 , and $N_2/C_3H_6/O_2$ are shown. The difference in the barrier height from air to hydrocarbon mix as derived from the intercept of $1/C^2$ vs. V decreases in the same pattern as the sensor response: from 916 mV, to 807 mV to 389 mV as the mix is changed from N_2/H_2 to N_2/C_3H_6 to $N_2/C_3H_6/O_2$ respectively.

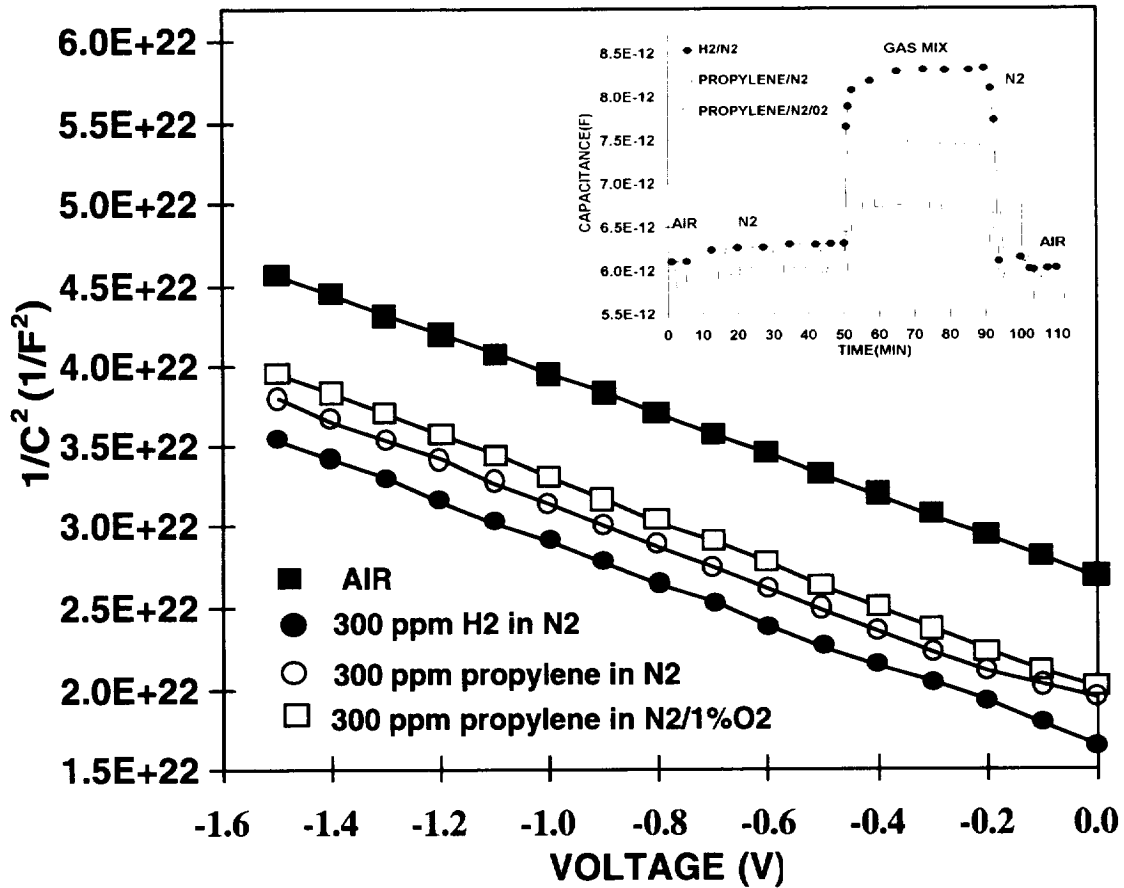


Figure 2. Inset: The capacitance vs. time at 400°C upon exposure to air, nitrogen plus 300 ppm hydrogen (●) or propylene (○), or nitrogen plus 300 ppm propylene plus 1% oxygen (□). The diode is first exposed to air, nitrogen, then the hydrogen bearing mixture followed by nitrogen, then air. The figure shows the corresponding $1/C^2$ vs. voltage curves for air (■) and each of the hydrogen bearing mixtures.

The data in Figure 2 is consistent with the mechanism of hydrogen and hydrocarbon detection discussed above. We note that the air curve is parallel to the N_2/H_2 curve. This suggests that the major effect of the hydrogen is to change the work function of the Pd,

and not, for example, interact with dopants in the SiC. The curves for N_2/C_3H_6 and $N_2/C_3H_6/O_2$ are also predominately parallel to that in air and N_2/H_2 . This suggests that the mechanism of detection in both cases is the same as that for hydrogen: a change in the Pd work function due to the presence of hydrogen. The presence of oxygen changes the magnitude of the sensor response, but does not change the basic mechanism of detection. Finally, the decreasing sensor response as the gas mixture is changed from N_2/H_2 to N_2/C_3H_6 to $N_2/C_3H_6/O_2$ is likely due to decreasing amounts of hydrogen available to be absorbed into the Pd. The exact surface science still remains to be investigated, but less hydrogen would migrate into the lattice if the dissociation of the propylene is incomplete, or if less sites are available on the Pd surface for dissociation from propylene than from molecular hydrogen. The presence of oxygen further decreases the amount of hydrogen available to migrate into the Pd lattice by either taking up sites used by the propylene for dissociation, or by combining with the released hydrogen to form water. Thus, this data is consistent with the idea that the mechanism of detection is due to the dissociation of hydrogen and hydrocarbons at the metal surface evolving hydrogen which changes the work function of the Pd.

The dissociation of hydrocarbons is a temperature dependent effect even in the presence of a catalyst. We have studied the response of a Pd/SiC Schottky diode to one hydrocarbon, propylene, at a range of temperatures. Such studies help define the operating temperature of the sensor, as well as its long-term stability. Figure 3 shows the zero bias capacitive response of the sensor to 360 ppm propylene at various temperatures. The sensor temperature is increased from 100°C to 400°C in steps of 100°C and the response of the sensor is observed. At a given temperature, the sensor is exposed to air for 20 minutes, N_2 for 20 minutes, 360 ppm of propylene in N_2 for 20 minutes, N_2 for 10 minutes, and then 10 minutes of air.

There are two points to note in the sensor behavior as shown in Figure 3. First, the baseline capacitance in air does not change between 100°C and 200°C, but decreases to a lower value as the temperature is raised to 300°C and above. This decrease in capacitance as the sensor is heated above 200°C is consistent with the results reported in reference 16. Both results suggest the possibility that temperature dependent chemical reactions which affect the electronic properties of the diode may have an onset at temperatures above 200°C. Second, Figure 3 clearly shows that the magnitude of sensor response to 360 ppm propylene depends strongly on the operating temperature. A sensor operating temperature of 100°C is too low for propylene to dissociate on the Pd surface, so the device does not respond at all. The three other curves for 200°C, 300°C, and 400°C show that elevating the temperature increases the sensor's response to propylene. The presence of propylene can be detected at any of these higher temperatures with 200°C being the minimum operating temperature determined in this study. Since the standard long-term operating temperature of Si is usually below 200°C, these results demonstrate the significant advantages of using SiC rather than Si in gas sensing applications.

The sensor response to other hydrocarbons at a given temperature will vary depending on the hydrocarbon. This is shown in Figure 4 where the capacitive response of the Pd/SiC Schottky diode at 300°C is shown for three different hydrocarbons. Two of the hydrocarbons, propylene and ethylene, are alkenes while the third, methane, is an alkane. Thus, this test involves two different classes of hydrocarbons. The sensor is exposed to air for 20 minutes, N_2 for 20 minutes, 360 ppm of propylene, ethylene, or methane in N_2 for 20 minutes, N_2 for 10 minutes, and then 10 minutes of air. The

temperature, 300°C, is significantly above the minimum operating temperature of 200°C discussed above. The sensor response to propylene is just slightly larger than that of ethylene. In contrast, the sensor response to methane shows a smaller, short-term increase in capacitance followed by a drift towards the baseline.

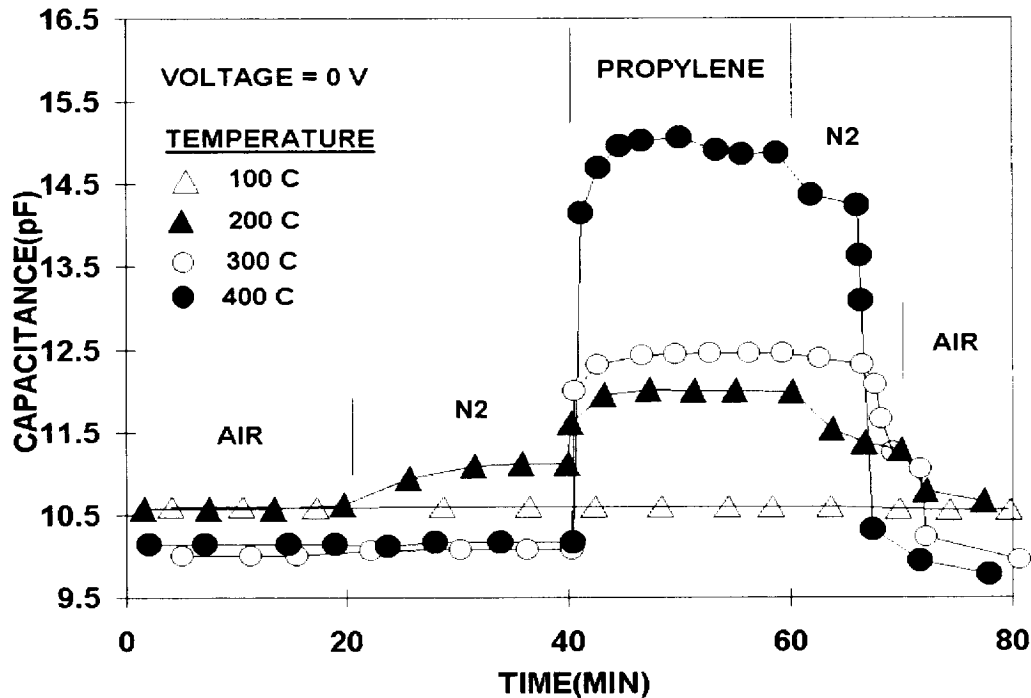


Figure 3. The temperature dependence of the zero bias capacitance to various gas mixtures. The response to propylene is seen to be strongly temperature dependent.

Figure 5 shows the sensor response to the same test as in Figure 4 but measured in the I-t mode. There are two points to note in Figure 5. First, the change in the forward current upon exposure to propylene and ethylene is very large: more than a factor of 1000. This is in contrast to the capacitive response in Figure 4 which showed an increase in capacitance of near 35%. This is due to the fact that a change in the Pd work function increases the forward current exponentially while the capacitance only changes quadratically. Second, the response to methane, although larger when measured with the forward current, is still significantly different from that of propylene and ethylene: a short term increase in signal followed by a decrease back toward baseline. Thus, the sensor response differs depending on the class of hydrocarbon.

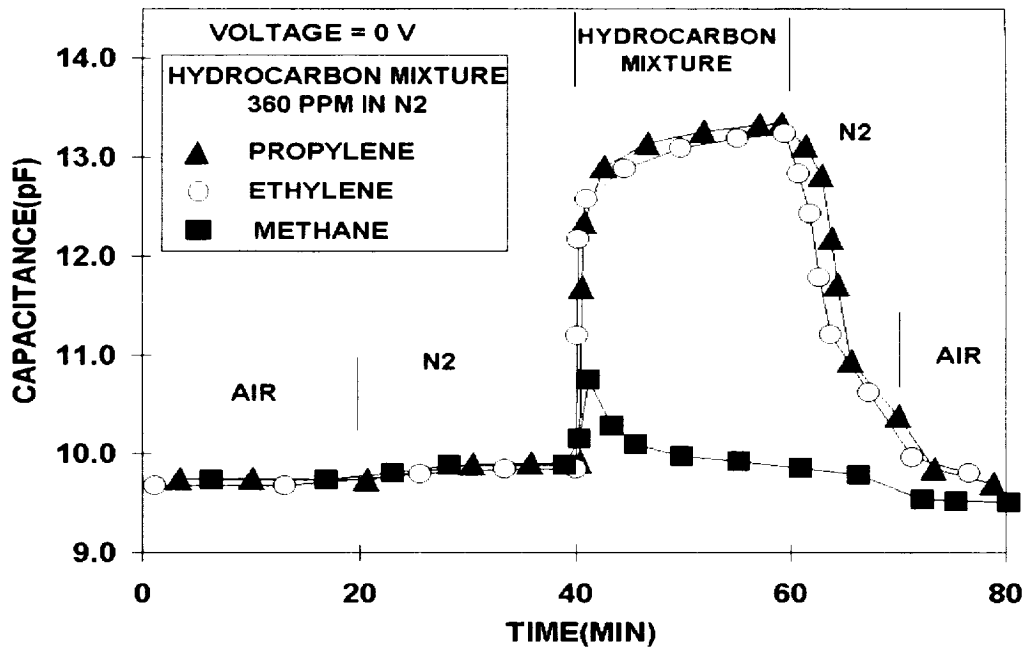


Figure 4. The zero bias capacitance vs. time at 300°C upon exposure to propylene (▲), ethylene (○), and methane (■). The sensor response depends on the class of hydrocarbon.

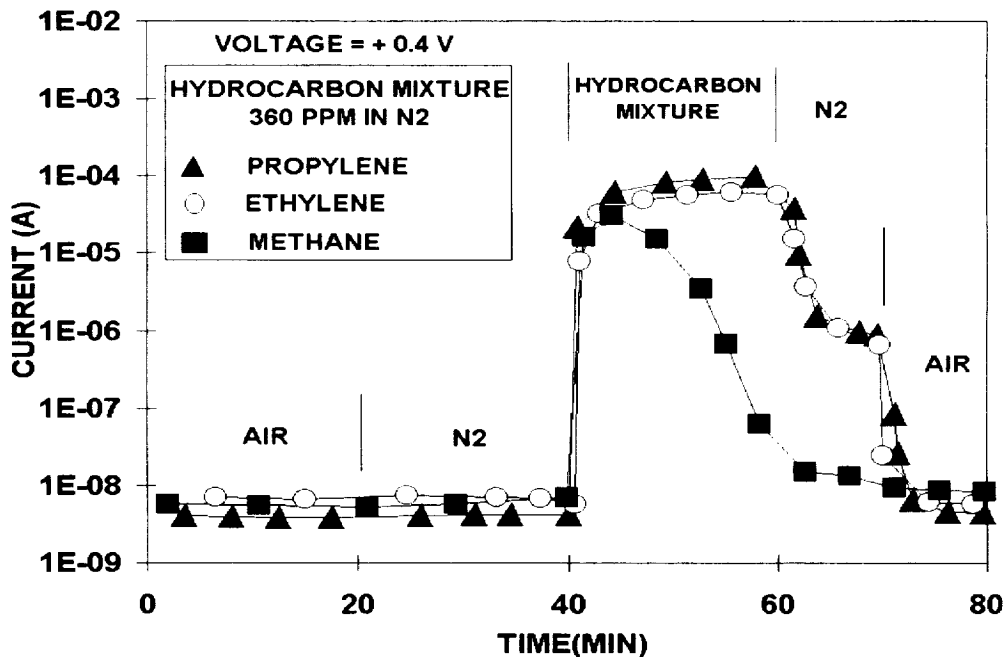


Figure 5. The forward current at 0.4V vs. time at 300°C upon exposure to propylene (▲), ethylene (○), and methane (■). The sensor response is much larger in the forward current mode than in the capacitive mode but still depends on the class of hydrocarbon.

Therefore, this data shows that Pd/SiC Schottky diodes respond to hydrogen and hydrocarbons up to 400°C. The data strongly suggests that the mechanism of detection is the dissociation of the gas on the Pd surface and subsequent detection of hydrogen. The effect is both temperature and hydrocarbon dependent. The sensor can be extremely sensitive, especially when the forward current is measured. Thus, the SiC-based Schottky diode structures have high potential for the measurement of a variety of chemical species.

In order to use these sensors in high temperature applications, such as in emissions measurements, it is necessary to determine their long-term stability in high temperature environments. We have studied the stability of Pd/SiC Schottky diodes at 425°C in air for extended periods. A detailed description of this work may be found elsewhere (17-18) so we will only summarize the results here.

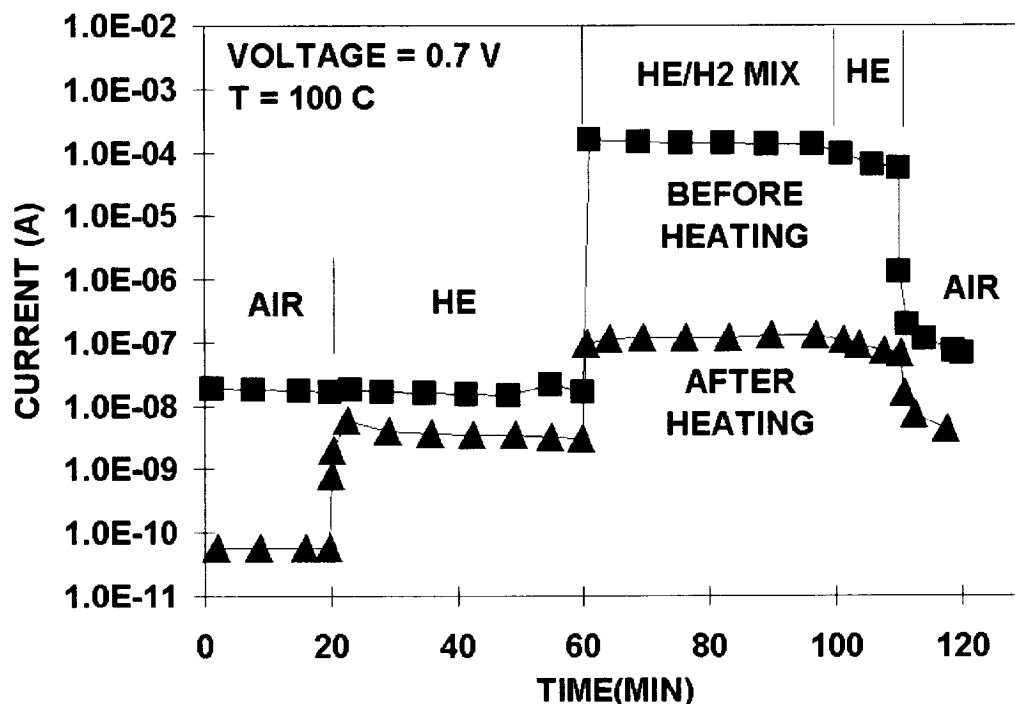


Figure 6. The forward current at 0.7 V vs. time at 100°C before (■) and after heating (▲) for 140 hours in at 425°C. The sensor is exposed to air, He, 1000 ppm H₂ in He, followed by He and air. The sensor is sensitive to hydrogen although its properties have changed.

A major result of this work is shown in Figure 6. The logarithmic forward current at 0.7 V is shown before and after heating at 425°C for 140 hours in air. The sensor is exposed to air for 20 minutes, He for 20 minutes, 1000 ppm H₂ in He (He/H₂ mix) for 20 minutes, N₂ for 10 minutes, and then 10 minutes of air. Before heating, the current

shows no significant change in the oxygen-deficient He environment while after heating the current increases from the air baseline nearly two orders of magnitude upon exposure to He. Before heating, the current changes from the baseline in air (near 10^{-8} A) by nearly four orders of magnitude in response to the He/H₂ mix. After heating, the current change is about three orders of magnitude from the baseline in air (near 10^{-10} A) and about one order of magnitude from the level in the He environment. In both cases, the current in the He/H₂ mix is significantly larger than that measured in air. The recovery of the diode's current towards baseline is much more rapid in air than in He. Therefore, after 140 hours at 425°C in air, the diode current still responded sensitively to the He/H₂ mix and thus the diode still functioned as a hydrogen sensor. However, the diode's sensitivity and characteristics have changed. Clearly, the diode structure will have to be stabilized for long-term, high temperature applications. Attempts to stabilize the structure by including a silicon dioxide (SiO₂) barrier layer between the Pd and the SiC have, surprisingly, led to an accelerated degradation of the diode's sensitivity (19). Further attempts to stabilize the diode's structure are under way.

SiC-BASED SENSOR PACKAGING

The packaging of a complete integrated sensor chip for operation outside of a probe stage environment is obviously essential for real world applications. One major component of our packaging effort is the formation of structures on the SiC substrate necessary to make a complete integrated sensor chip. These structures must allow the sensor chip to operate in a variety of environments without interfering with sensor operation. Since the sensing reactions involved are temperature dependent, one necessary aspect of complete sensor functionality is the ability to control the temperature of the Schottky diode sensing element. The minimum additional structures necessary for temperature control are a heater and temperature detector. The temperature detector and heater circuits must be electrically isolated from the Schottky diode sensing element to prevent electrical cross-talk.

We have investigated two approaches to packaging a complete integrated sensor including a temperature detector and heater. The first approach was to place the heater away from the sensing area on the opposite, backside of the SiC substrate. Isolation of the heater and temperature detector was achieved by depositing an insulator on the backside of the SiC and then sputter depositing a Pt-based temperature detector and heater onto the insulator. Initial processing problems resulted in pinholes in the insulating layer causing unstable sensor operation due to electrical cross-talk between the heater and the SiC substrate. These processing problems are being addressed. The second approach to providing a heater and temperature detector to the SiC-based Schottky sensing element is to apply the temperature detector and heater onto a separate piece of Pyrex glass and then mount the SiC sensor onto the Pyrex substrate using silver epoxy. This approach has provided stable temperature control to the sensor without electrical cross-talk between the sensing diode and heater.

Another major component of the packaging effort is to mount the complete sensor chip into an electronic package which can provide connections with external hardware. The type of electronic package necessary depends on the application. For applications in which a high temperature sensor is operated in room temperature ambient conditions, a modified version of a traditional electronic package may be sufficient. However, if the application involves sensor operation in harsh conditions such as an engine, traditional electronic packaging may be inadequate.

Our work has centered on packaging a complete, integrated sensor chip for operation in a room temperature ambient where the Schottky sensing element is temperature controlled up to 600 °C. Such a package would allow characterization of the sensor at high temperatures without testing in a high temperature environment. Early in our sensor packaging work, we found that mounting our integrated sensor design onto the base of a commercially available TO-5 header unacceptably restricted the maximum sensor temperature. The high thermal conductivity of SiC combined with the intimate contact of the sensor chip with the TO-5 package led to considerable heat loss from the sensor chip to the surrounding header. The maximum temperature achievable under these conditions was near 225°C. Increasing heating power to several watts increased the sensor temperature but led to degradation and failure of the heater and temperature detector.

This problem was addressed by changing the way the complete sensor is mounted in the TO-5 header. The complete sensor is suspended with the wires used for electrical contacts to four poles of a TO-5 type base. Thus, the only contact that these elements had with the outside world was through the wires. Platinum wire of 25 μm diameter was used for the electrical contact and suspension. This approach significantly decreased the amount heat loss to the ambient and allowed heating of the sensor to temperatures as high as approximately 550°C in a room temperature ambient. However, it is anticipated that improvements in processing combined with SiC micromachining techniques may provide an alternative solution to this problem in the future. By micromachining the backside of a SiC substrate with an incorporated temperature detector and heater, the heat loss to the sensor package will decrease. This would likely allow standard mounting of the sensor to a TO-5 header.

The most important aspect of a sensor packaging effort is that the resulting packaged sensor not be damaged or have decreased capabilities due to the packaging. Figure 7 shows the response of a packaged Pd/SiC Schottky diode sensor to air, N₂, 1000 ppm H₂ in N₂, and then air. The change of the sensor forward current to 1000 ppm hydrogen is near a factor of 10. This response is a significant decrease from the response of diodes examined in the probe chamber as seen, for example, in Figures 5-6. Possible causes for this decrease in sensitivity upon packaging include: 1) The diode surface area in the packaged diodes is usually larger than that of diodes examined in the probe station. The presence of micropipes (1,20) in the SiC might dominate the current flow in a packaged sensor and decrease the effect on the sensor of changes in Pd work function. 2) Processing the sensor for packaging may influence diode properties crucial to the sensors sensitivity. The reason for decreased sensor sensitivity after packaging is an area of continuing investigation.

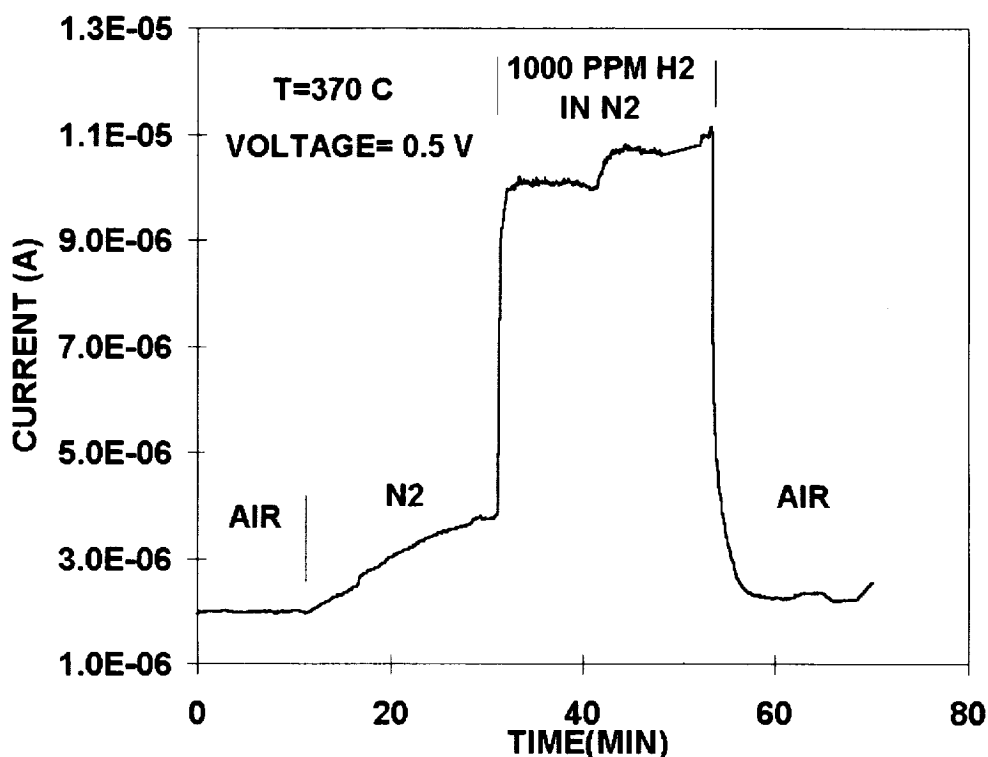


Figure 7. The response of packaged Pd/SiC Schottky diode sensor to air, N₂, 1000 ppm H₂ in N₂, and then air at 370°C. The sensor response is seen to be smaller than those seen in an unpackaged sensor.

SiC-BASED NO_x DETECTION

A natural extension of the work on SiC-based hydrogen and hydrocarbon sensors is the development of a NO_x sensor using the same baseline structures. Lunstrom et al. have demonstrated the use of capacitive structure for NO_x detection (3). However, NO_x sensitive Schottky diode structures have not been demonstrated.

We are taking two approaches in this work. The first approach is to change the catalytic gate of the Schottky diode to a material more sensitive to the presence of NO_x. A prime candidate material is platinum (Pt). Changes in the Pt upon exposure to NO_x is thought to change the electronic properties of the diode. These changes can be correlated to changes in the NO_x concentration in the ambient and thus be used to quantitatively measure the NO_x concentration. However, the presence of interfering gases such as hydrocarbons and oxygen must be accounted for in order to obtain an accurate reading of the NO_x concentration.

The second approach is the incorporation of a NO_x sensitive insulator into a Schottky diode structure. This approach allows the combination of SiC semiconductor technology with more traditional methods of NO_x detection such as the use of metal oxides. This would allow SiC to act as a platform for gas sensing over a range of temperatures which would not be possible with lower temperature and more reactive

Si-based devices. For example, a thin insulating layer of tin oxide (SnO_2) can be placed between a porous catalytic gate and the SiC. Upon exposure to NO_x , it is surmised that changes will occur in both the catalytic gate and the insulating layer. The combined effect of these changes will yield a more sensitive sensor than changes in the gate alone. Further, the sensor structure can be tailored to allow sensor characteristics to be determined by the sensitivity of the insulating layer alone by, for example, using a NO_x insensitive gate. Prototypes of these systems are under development and the results of testing of these sensors is planned for a future publication.

SUMMARY AND FUTURE PLANS

The development of SiC-based structures allows for sensor operation in environments and applications where Si-based technologies are inoperative. In particular, gases such as hydrocarbons can be detected by operating the SiC structure at temperatures beyond the capabilities of Si. The work at NASA LeRC in conjunction with CWRU has centered on the development of a SiC Schottky diode sensor structure. This structure is very sensitive to hydrogen and hydrocarbons. The mechanism of detection is based on the dissociation of the gas on the catalytic gate of the diode and the subsequent detection of hydrogen. The ability to detect hydrocarbons is temperature dependent and differentiation of classes of hydrocarbons has been demonstrated.

Areas of future development include improvements on the stability of the sensor for long-term, high temperature operation and incorporation of the sensor in a stable sensor package. The use SiC-based sensors for NO_x detection is also being investigated. This work relies on the Schottky diode structure with a different gate material or the combination of SiC with more traditional NO_x detection materials such as SnO_2 . The use of SiC holds significant promise for the development of new sensors and sensing approaches in gas sensing applications.

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