SILICON CARBIDE GAS SENSORS FOR PROPULSION EMISSIONS AND SAFETY APPLICATIONS

G. W. Hunter, J. Xu, and P. G. Neudeck
NASA Glenn Research Center
Cleveland, OH

D. Lukco
ASRC Aerospace
Cleveland, OH

A. Trunek and D. Spry
OAI
Cleveland, OH

M. Artale, P. Lampard, and D. Androjna
Sierra Lobo, Inc.
Cleveland, OH

D. Makel and B. Ward
Makel Engineering, Inc.
Chico, CA

ABSTRACT

Silicon carbide (SiC) based gas sensors have the ability to meet the needs of a range of aerospace propulsion applications including emissions monitoring, leak detection, and hydrazine monitoring. These applications often require sensitive gas detection in a range of environments. An effective sensing approach to meet the needs of these applications is a Schottky diode based on a SiC semiconductor. The primary advantage of using SiC as a semiconductor is its inherent stability and capability to operate at a wide range of temperatures. The complete SiC Schottky diode gas sensing structure includes both the SiC semiconductor and gas sensitive thin film metal layers; reliable operation of the SiC-based gas sensing structure requires good control of the interface between these gas sensitive layers and the SiC. This paper reports on the development of SiC gas sensors. The focus is on two efforts to better control the SiC gas sensitive Schottky diode interface. First, the use of palladium oxide (PdO_x) as a barrier layer between the metal and SiC is discussed. Second, the use of atomically flat SiC to provide an improved SiC semiconductor surface for gas sensor element deposition is explored. The use of SiC gas sensors in a multi-parameter detection system is briefly discussed. It is concluded that SiC gas sensors have potential in a range of propulsion system applications, but tailoring of the sensor for each application is necessary.

INTRODUCTION

Silicon carbide (SiC) has high potential as the electronic semiconductor material for a new family of high temperature sensors and electronics. Silicon carbide can operate as a semiconductor in conditions under which silicon cannot adequately perform, such as at temperatures above 400°C. [1]. Silicon carbide gas sensors have been in development for a number of years using a range of device structures including capacitors [2], transistors [3], and Schottky diodes [4-8]. These sensors have been shown to be highly sensitive to several gases, including hydrogen and hydrocarbons, making them useful for a range of applications. One area where SiC semiconductor technology can be applied is in gas sensing for aerospace applications.

A range of aerospace applications require chemical sensing technology [9]. One application area is the monitoring of emissions from high temperature combustion environments such as propulsion systems. In aeronautic combustion emissions monitoring applications, sensitive detection of nitrogen oxides (NO_x) and hydrocarbons (C_xH_y) can be used to reduce emissions and potentially monitor the efficiency and health of the engine. For in-situ engine monitoring, high temperature operation is necessary. A significant challenge in this application is determining the relative emission constituents in a mixed and varying chemical environment. A SiC based gas sensor can be combined with other sensors forming a High Temperature Electronic Nose sensor array to characterize a range of emission species emitted by an engine [9].

A second application area is monitoring of fuel leaks in launch vehicles [10]. Detection of low concentrations of hydrogen and hydrocarbon fuels is critical in avoiding explosive conditions that could harm personnel and damage the vehicle. Reliable vehicle operation also depends on the timely and accurate measurement of these leaks. Detection of low concentrations of fuel is generally necessary from room temperature to cryogenic ambient and in air or inert gas atmospheres. Further, measurement of highly toxic propellants such as hydrazine in concentrations as low as the parts per billion (ppb) level is desired to safeguard the health of astronauts or ground personnel exposed to propulsion systems. This includes operation of manned systems such as the International Space Station or for ground operations of future Exploration Launch vehicles.

These applications require operation in a variety of challenging conditions: from cryogenic temperature to above 600°C, from chemically inert environments to highly corrosive engine conditions, and from the detection of one gas over a wide concentration range in inert environments to the detection of several gases over more narrow concentration ranges in the presence of interfering gases. These applications commonly require high sensitivity, long-term stability, good repeatability, and sensor operation at elevated temperatures to detect the gases of interest. The combination of these sensor requirements has led NASA Glenn Research Center (NASA GRC) SiC gas sensor development to concentrate on sensors based on the Schottky diode structure.

A Schottky diode is composed of a metal in direct contact with a semiconductor (MS), or a metal in contact with a very thin insulator or oxide on a semiconductor (MIS or MOS). For gas sensing applications, the metal is often catalytic. The advantage of Schottky diode gas sensors are their high sensitivity. The detection mechanism for hydrogen (H_2) involves the dissociation of H_2 on the surface of a catalytic metal leading to the formation of a dipole layer at the interface of the metal and the insulator (or metal-semiconductor interface depending on the structure). This dipole layer affects the effective Schottky barrier height of the diode resulting in an exponential change in the forward current and a quadratic change in the capacitance [11-12] while the diode is under fixed bias. The detection of hydrocarbons is possible if the sensor is operated at a high enough temperature to dissociate the hydrocarbon and produce atomic hydrogen at the sensor surface. The resulting atomic hydrogen affects the sensor output in the same way as molecular hydrogen [4, 13-14]. Predominately, the temperature for sensitive hydrocarbon detection is beyond the upper limit for silicon-based Schottky diode functionality and thus SiC enables high temperature detection of hydrocarbons with high sensitivity.

The successful use of the Schottky diode structure as a gas sensor depends on strict control of the metal-semiconductor interface [7]. One complicating factor in control of this interface is the operation of gas sensors at high temperatures. Higher temperature operation implies possible reactions, especially metal silicide formation, between the catalytic sensing metal thin film and the SiC. Overall, the choice of surface treatment or barrier layer(s) between the catalytic metal and the SiC substrate is complicated by simultaneous requirements of high sensor stability during high temperature operation while maintaining high sensitivity.

A second major complicating factor in the control of the SiC interface is the quality of the SiC semiconductor. In addition to high densities of extended crystalline defects such as micropipes, commercial SiC wafers also exhibit significantly rougher surfaces, and larger warpage than is typical for silicon wafers [15]. Thus, variability in the SiC surface itself significantly complicates efforts to control the catalytic metal/SiC interface.

The purpose of this paper is to provide an update on the development and application of SiC Schottky diode gas sensors related to emission and safety applications. In particular, this paper reports on the development of SiC gas sensors focusing on two efforts to better control the SiC gas sensitive Schottky diode interface. First, the use of palladium oxide (PdO_x) as a barrier layer between the metal and SiC is discussed. Second, the use of atomically flat SiC to provide an improved SiC semiconductor surface for gas sensor element deposition is explored. The use of SiC gas sensors in a multiparameter detection system is briefly discussed. It is concluded that SiC gas sensors have potential in a range of propulsion system applications of SiC gas sensor systems, but tailoring of the sensor for each application is necessary.

RESULTS AND DISCUSSION

Barrier Layer: Pd/PdO_x/SiC Structure

The use of a barrier layer between the catalytic metal and SiC was explored in previous publications. For example, the use of a platinum/chrome carbide/silicon carbide (Pt/Cr_3C_2/SiC) Schottky diode structure using commercial off-axis SiC epilayer was described in reference 8. Overall, the sensor showed stable operation with good sensitivity at 580°C, some migration of the chemical species within the structure during heating, and changing behavior during an initial break-in period.

In order to complement, and perhaps improve upon, the above approach using only palladium oxide as the barrier layer was investigated. It had been noted within our group that palladium oxide had formed naturally during heating processes in other palladium based SiC samples tested. Palladium oxide itself is a stable structure and its uncontrolled formation during heating was thought to disrupt the gas sensor structure. The objective of this work is to apply the palladium oxide in a controlled manner by standard deposition techniques resulting in a Pd/PdO_x/SiC structure. The approach is to form a barrier layer between the catalytic metal and SiC, thus stabilizing the sensor structure. This section presents the results of characterizing the properties of this Pd/PdO_x/SiC sensor structure.

The Pd/PdO_x/SiC sensor structure is fabricated as follows: a commercially available 2" diameter, 3.5° off-axis, 6H-SiC 400 microns (μm) thick substrate with 2 μm thick epilayer of n-type doping of 2.0 E+16 is patterned with photoresist and a Schottky diode photomask to form multiple diode contacts on the front side of the wafer. Sputtering is used to reactively deposit 50 Angstroms (Å) of PdO_x on the SiC substrate followed by the deposition of 450 Å of Pd on top of the PdO_x. A lift-off technique is then used to form circular Pd/PdO_x/SiC Schottky patterns of diameter 830 μm.   Backside contacts were achieved by sputtering 100 Å of titanium (Ti) and 5000 Å of nickel (Ni), followed by annealing at 1000°C for 5 minutes in nitrogen (N_2).

The gas sensor testing facility and sample connections have been described elsewhere [4]. The sample rested on a hot stage whose temperature was controlled from room temperature to near 600°C. Current-time (I-t) measurements were taken to characterize diode responses as a function of time during exposure to a variety of gases. The Pd/PdO_x/SiC Schottky diode structure was heated at 450°C for a total of 1400 hours. The sensor was periodically tested by first being exposed to air for 5 minutes, N_2 for 5 minutes, 0.5% hydrogen in N_2 for 10 minutes, pure N_2 for 5 minutes, and then air. The gain of the sensor was calculated as the difference in currents between hydrogen bearing gas and air divided by the current in air, i.e., the change in signal divided by the baseline.

The response of the Pd/PdO$_x$/SiC Schottky diode sensor to the air/nitrogen/hydrogen-nitrogen/air test cycle at a bias voltage of 1 V is shown in Figure 1 at two separate times: 305 and 979 hours. The data shows good repeatability of signal in form and magnitude at 305 and 979 hours. At both times, the sensor has limited response to changes from air to nitrogen followed by a large response of over two orders of magnitude change in current to 0.5% H_2/ 99.5% N_2. The response to hydrogen is much quicker than the recovery of the sensor signal after the sample is again exposed to nitrogen at 20 minutes. The overall recovery in inert environments is slower in N_2 than air, but previously this has been found to be test chamber dependent. This data shows a sensor with a highly sensitive response to hydrogen and a repeatable behavior over a significant period of time.

The long term behavior of the sensor over the full 1400 hours of testing at 450°C is shown in Figure 2. In this figure, the sensor current in air and in 0.5% H_2/ 99.5% N_2 is shown over time at 450°C measured at 1 V. Also shown in Figure 2 is the gain of the sensor derived from currents in 0.5 H_2/ 99.5% N_2 and air. Figure 2 shows that after a break-in period during approximately the first 40 hours, the sensor baseline current in air, current in 0.5 % H_2/95% N_2 and overall gain is shown to be generally stable with heating for 1400 hours.

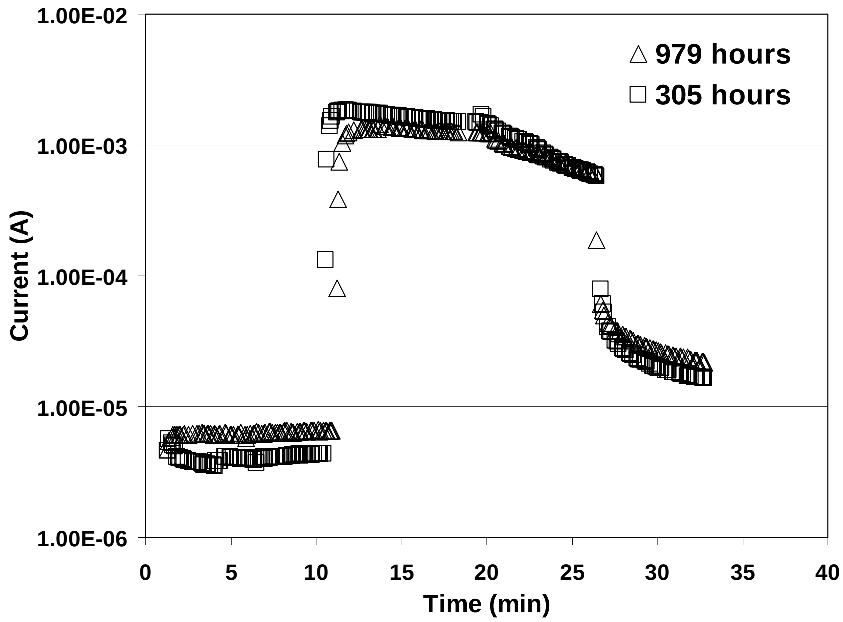


Figure 1. The Pd/PdO$_x$/SiC Schottky diode gas sensor tested at 450°C at 305 hours (□) and 979 hours (△) in 0.5% hydrogen and at 1V. The sensor response shows a strong and nearly repeatable response to 0.5% hydrogen in nitrogen over the heating period.

The overall result of this high temperature testing is that the Pd/PdO$_x$/SiC sensor has high sensitivity with prolonged stability and represents a marked improvement over a Pd/SiC Schottky diode sensor without the PdO$_x$ layer [4]. Surface analysis was conducted on the tested PdO$_x$ based sensor (not shown) and no significant silicide formation or species migration was observed. In other words, two of the major reasons for sensor degradation, silicide formation and species migration, are significantly inhibited. Thus, the barrier layer of PdO$_x$ prevents and minimizes chemical reaction between the catalytic sensing layer (metal or metal alloy) and the substrate layer (SiC). Palladium oxide prevents formation of metal silicides, an unwanted reaction product that forms between the catalytic sensing layer and the substrate layer. These silicide materials can adversely affect the sensitivity of the hydrogen detection. The barrier interlayer is resistant to further oxidation when made from palladium oxide. This resistance to oxidation prevents other degradation of the sensor at high temperatures.

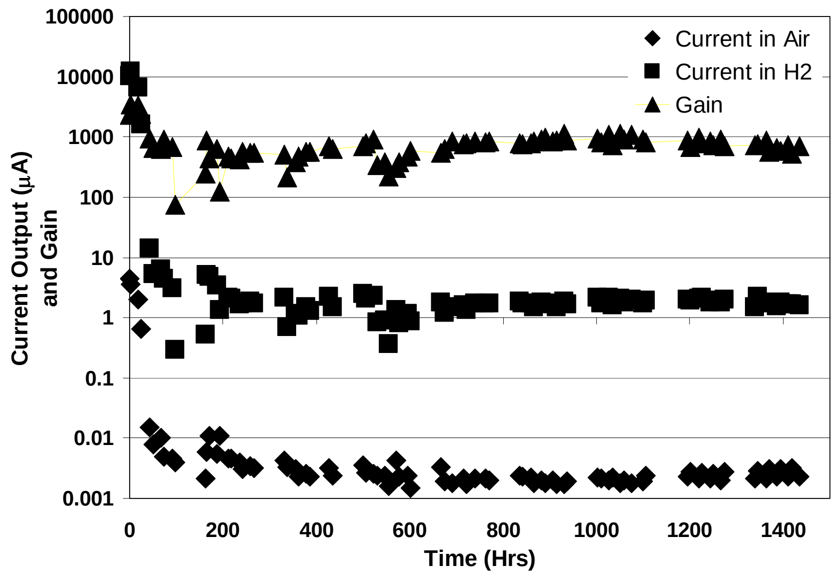


Figure 2. The Pd/PdO_x/SiC Schottky diode gas sensor response at 450°C and at 1V. Shown is the baseline current in air, current in 0.5% hydrogen in nitrogen, and the sensor gain (change in sensor signal/baseline signal). The sensor is seen to have nearly constant behavior throughout the extended test period after a break-in period.

Overall, this approach takes a reaction product, PdO_x, whose formation previously contributed to the disruption of the sensor structure and, by controlling its formation and position in the gas sensor structure, uses it to improve sensor stability and sensitivity. Oxidation of the barrier layer, which is sometimes problematic with other barrier layers, is not an issue with PdO_x because it is already oxidized and the PdO_x layer is very stable. In the extreme, the top surface layers of the PdO_x could be potentially reduced to Pd through combination with atomic hydrogen. If this occurs, it is believed but has not yet been verified, that this would likely increase the sensitivity of the device by creating more Pd which could later be reoxidized. However, it would not result in irreversibly degrading the structure with, e.g., the formation of silicides. Thus, it suggested that the use of PdO_x between the catalytic metal and the SiC is a highly viable approach to stabilizing the Schottky diode structure and such data as shown in Figure 2 suggests a notable advancement in the basic Schottky diode sensor structure. Future work will examine the use of PdO_x with other catalytic metals such as platinum (Pt) as well as in conjunction with reactive insulators such as tin oxide.

SiC Surface Treatment: Atomically Flat SiC

The first use of atomically flat SiC in sensing applications was discussed in reference 8. The result of that testing is shown in Figure 3 which compares the sensor response of an atomically flat (AF) and non-atomically flat (NAF) SiC Schottky diode gas sensor showing the data starting at 200°C and finishing at 300°C. The difference in response between these two sensors takes effect when the sensor is heated to 300°C and is easily evident in Figure 3. The AF sensor gain increases dramatically compared to the NAF sensor and stabilizes near 325 hours into the testing or after nearly 200 hours at 300°C. The gain of the AF sensor is near 7250 while that of the NAF sensor is near 50. Thus the gain of the AF sensor response averages nearly 145 times greater than that of the NAF sensor. These preliminary results have suggested a strong advantage to the use of on-axis and atomically flat SiC over the standard materials on which SiC Schottky diode gas sensors are fabricated [8].

a) b)

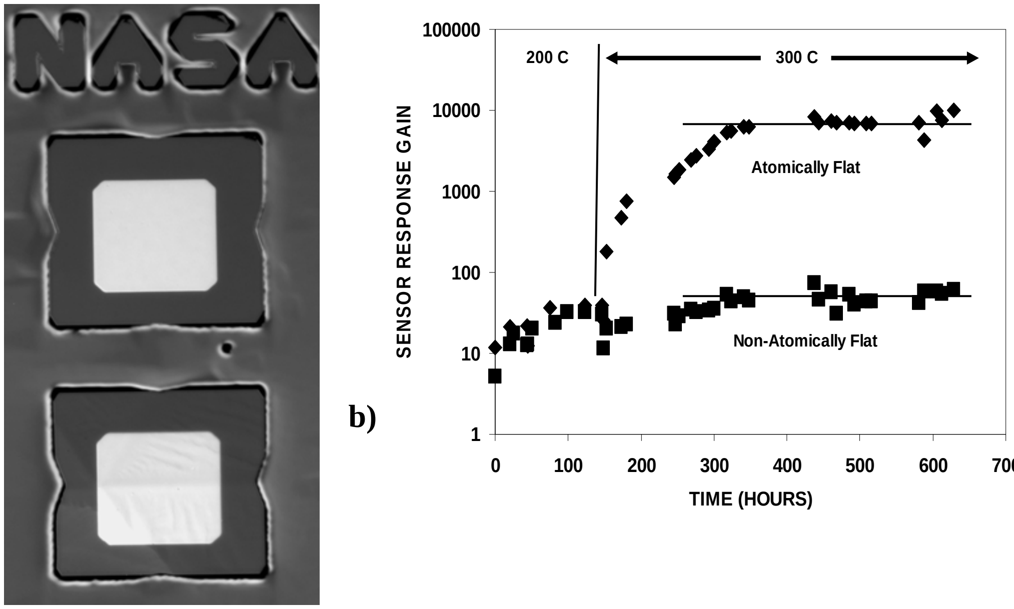


Figure 3. Demonstration of atomically flat SiC gas sensor responses from reference 8: a) Side by side sample of AF and NAF sensors. The bottom is the non-atomically flat sensor and the top is the atomically flat sensor. Surface differences between the two sensors are significant and include hillock morphology from a screw dislocation on the left mesa. b) Comparison of sensor gain to 0.5% hydrogen between Pt/SiC sensors deposited on atomically flat SiC (◆) and non-atomically flat SiC (■). At near 150 hours of testing at 200°C, both sensors are heated to 300°C. The difference in response between the sensors is easily observed.

Subsequent work being reported in this paper involves moving from the basic atomically flat mesa structure holding only a sensor, as shown in Figure 3, towards an atomically flat structure that includes the components necessary for an operational gas sensor. Such a structure would not only include the gas sensitive Schottky diode, but also a temperature detector and heater to control the sensor’s temperature in variable environments. The approach is to develop a process to fabricate a small, complete sensing structure with minimal thermal mass such as those formed in silicon-based structures [9]. Such processing capabilities are necessary if the full potential of atomically flat SiC gas sensors is to be achieved in compact microsystems.

Atomically flat or step-free surfaces were produced on commercially purchased on-axis 4H-SiC wafers in the following manner [16,17]. First, dry reactive ion etching (RIE) was used to form 30 µm deep trenches into the wafer surface to form an array of isolated growth mesas. Following an in-situ hydrogen pre-growth etch at 1630°C for 2 minutes, pure stepflow epitaxial growth is then used to grow all initial surface steps out to the edge of the mesa leaving behind a mesa with a topmost surface that is step-free. Only mesas that are not threaded by screw dislocations can be rendered step-free (i.e. atomically flat). Step-free mesas subjected to further growth will laterally evolve webbed cantilevered regions that extend out beyond the sidewalls at the top of the original mesa [16]. The sensor structure shown in Figure 4 resides on top of an atomically flat SiC “tabletop” that was mostly formed from lateral extension of SiC cantilevers grown from a much smaller pre-growth support mesa. As better described previously [16-17], the SiC epitaxial growth was carried out in a modified, commercial, horizontal flow, chemical vapor deposition (CVD) system with a tantalum carbide (TaC) coated susceptor. The growth was performed at a pressure of 200 millibar (mb), and a temperature of 1630°C. Propane (C_3H_8) and silane (SiH_4) were used for precursors in a hydrogen carrier gas with a Si/C ratio of 0.65. The growth time was five hours. A 2700Å thick Pt film was sputter deposited on both atomically flat and non-flat mesas. Photoresist was used to pattern the Pt film during an argon based dry etch and was then removed using solvents and an oxygen plasma. Backside contact was made by 1000Å Ti, 4000Å tantalum silicide ($TaSi_2$), and 2000Å Pt which has been shown to be a stable contact stack for high temperature applications [18].

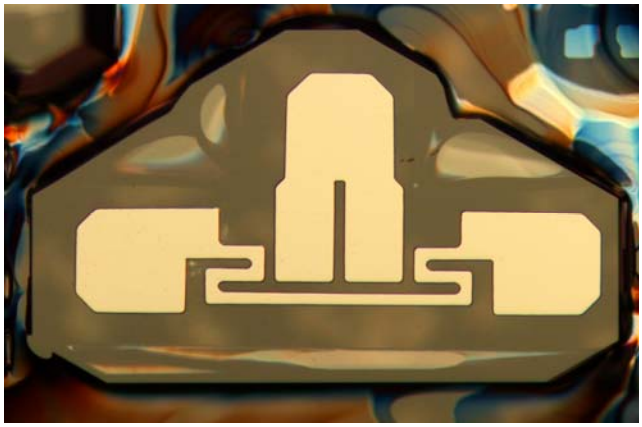


Figure 4. An atomically flat SiC mesa similar to the mesas shown in Figure 3, but now including both the Schottky diode gas sensor and a resistor for temperature control.

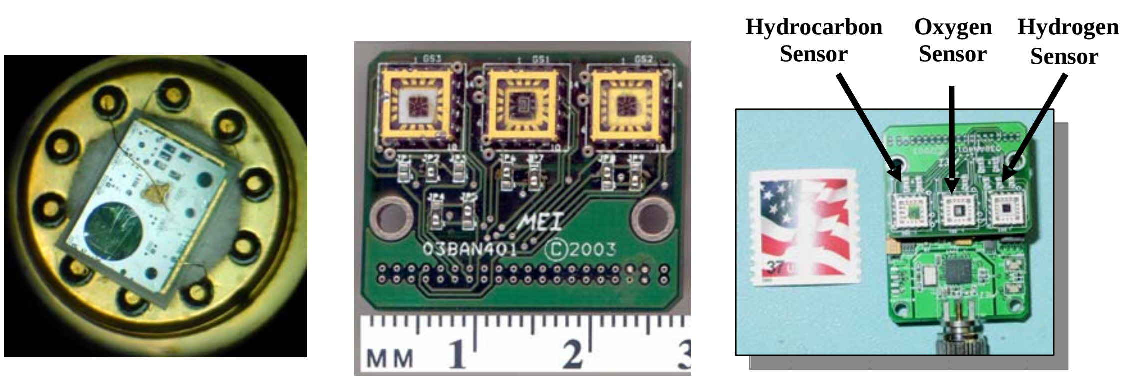
A sensor pattern with a combined temperature detector/heater has been deposited on an atomically flat mesa with dimensions near 0.6 x 0.3 mm and is shown in Figure 4. A Pt/SiC Schottky diode is shown in Figure 4 as the large contact pad in the center of the structure. A Pt resistor (with resistance ~ 50 ohms) forms the temperature detector/heater composed of the thin line metal pattern adjacent to the Schottky diode. Experience in fabrication of silicon based gas sensor systems has been used in the design of this SiC device. This and closely related structures are now in the process of testing and evaluation. Based on the results of this testing, modification of the atomically flat SiC device pattern will be performed. The long-term goal is to produce complete, operational SiC gas sensors that include the device structures pioneered in commercially available SiC, e.g., Pd/PdO_x/SiC, but retain the sensing advantages seen by using atomically flat SiC.

SENSOR APPLICATION

While work proceeds towards improving high temperature durability and sensitivity of SiC Schottky diode gas sensors, these sensors are useful in their present state for a range of applications. One area of development is an integrated smart leak detection system for a range of propulsion systems. The objective is to produce a microsensor array, that includes hydrogen, oxygen, and hydrocarbon sensors by microfabrication (MEMS) based technology [10]. Thus, a range of potential launch vehicle fuels (hydrogen or hydrocarbons) and oxygen can be measured simultaneously. The array is being incorporated with signal conditioning electronics, power, data storage, and telemetry. The final system will be self-contained with the surface area comparable to a postage stamp. Thus, this postage stamp sized “Lick and Stick” type gas sensor technology can enable a matrix of leak detection sensors placed throughout a region with minimal size and weight as well as with no power consumption from the vehicle. The sensors can detect a fuel leak from launch vehicles, and combine that measurement with a determination of the oxygen concentration to ascertain if an explosive condition exists. Sensor outputs are fed to a data processing station, enabling real-time visual images of leaks, and enhancing vehicle safety.

A prototype model of the “Lick and Stick” sensor system has been fabricated [8,10]. The complete system has signal conditioning electronics, power, data storage, and telemetry with hydrogen, hydrocarbon, and oxygen sensors. The assembly of this sensor system starting with the SiC Schottky diode gas sensor is shown in Figure 5. Figure 5a shows the packaging of a SiC hydrocarbon sensor in a TO5 header with a wire bond extending from the TO5 connecting post to the front side of the SiC gas sensor. While the SiC sensor structure shown in Figure 5a is a sensor test pattern and not optimized for production, packaging of these sensors has occurred for multiple applications and highlights the use of even these developmental SiC gas sensors in operational systems. Figure 5b shows the integration of the SiC gas sensor with a hydrogen and oxygen sensor in an electronics board meant for sensor control and conditioning. Figure 5c shows the complete electronics including microprocessor, memory, and telemetry integrated with the three sensors and control electronics board. These electronics parts have been chosen particularly for possible use in space flight applications.

This “Lick and Stick” sensor system, shown in Figure 5, has been demonstrated to detect the presence of various hydrocarbon fuel (RP-1) concentrations (in this case, using a chrome carbide barrier layer), while simultaneously measuring the oxygen and hydrogen concentration [9]. This basic “Lick and Stick” sensor approach using the hydrogen and oxygen sensors is being considered for possible implementation in Crew Launch Vehicle (CLV) applications. Presently the flight version of this “Lick and Stick” leak detection system does not include the SiC gas sensor, which is still being matured for integration into flight ready systems. Although operational and tested in applications, further development is under way to allow the SiC gas sensors to have the maturity of, e.g., the existing silicon-based hydrogen sensor [10]. This paper has described some of these maturation efforts.



a) b) c)

Figure 5. A prototype version of a “Lick and Stick” leak sensor system with hydrogen, hydrocarbon, and oxygen detection capabilities combined with supporting electronics including signal conditioning and telemetry. a) SiC Schottky diode gas sensor in a TO5 header. b) SiC gas sensor integrated with a hydrogen and oxygen sensor with control electronics. c) Complete “Lick and Stick” leak sensor system with hydrocarbon, hydrogen, and oxygen sensors integrated with microprocessor, memory, and telemetry.

SUMMARY AND CONCLUSIONS

SiC-based Schottky diodes have significant potential to meet the needs of a range of important aerospace applications. Reaching that potential remains a significant technical challenge although significant progress has been made. A major issue is control of the surface interface between the catalytic gas-sensitive metal and the SiC semiconductor. Control of this interface is a major step towards enabling application of the unique properties of SiC Schottky diode gas sensors. However, this surface interface control is problematic, especially given the lack of maturity of SiC semiconductors and the fact that the sensors are standardly exposed to higher temperatures. This paper presents two examples of attempts to control the interface of a SiC-based Schottky diode gas sensor. The first method was to introduce a PdO_x layer between the catalytic metal and a defect-containing SiC semiconductor, while the second was to employ an atomically flat SiC semiconductor surface.

The PdO_x barrier layer approach has shown significant potential in producing a sensitive sensor with stable response for more than 1400 hours. This is a significant step in achieving a stable baseline Schottky diode gas sensing structure. While this barrier layer approach has produced a sensitive and stable sensor, the use of atomically flat SiC holds the highest potential for significantly changing the way SiC Schottky diodes are fabricated. The atomically flat approach ideally provides the same surface each time for device fabrication and should significantly increase device reproducibility. Steps are being taken to fabricate this atomically flat gas sensor structure with the complete components necessary for a gas sensor, i.e., usually a temperature detector and heater. Nonetheless, SiC gas sensor have been integrated and tested in a sensor array including “Lick and Stick” leak detection system, whose basic design is being considered for flight applications. SiC gas sensors are still being matured for some applications such as integration into a flight-worthy “Lick and Stick” leak detection system, but significant steps have been made as discussed in this paper.

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