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The Development of Hydrogen Sensor Technology at NASA Lewis Research Center

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THE DEVELOPMENT OF HYDROGEN SENSOR TECHNOLOGY AT NASA LEWIS RESEARCH CENTER

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ABSTRACT

The detection of hydrogen leaks in aerospace applications, especially those involving hydrogen fuel propulsion systems, is of extreme importance for reasons of reliability, safety, and economy. Motivated by leaks occurring in liquid hydrogen lines supplying the main engine of the Space Shuttle, NASA Lewis has initiated a program to develop point-contact hydrogen sensors which address the needs of aerospace applications.

Several different approaches are being explored. They include the fabrication of PdAg Schottky diode structures, the characterization of PdCr as a hydrogen sensitive alloy, and the use of SiC as a semiconductor for hydrogen sensors. This paper discusses the motivation behind and present status of each of the major components of the NASA LeRC hydrogen sensor program.

INTRODUCTION

Hydrogen leak detection sensor technology is important in aerospace applications for reliability, safety, and economic reasons. The conditions under which the sensor must operate vary depending on the application. Important factors include the sensor sensitivity, selectivity, and calibration stability, temperature of the gas to be measured, ambient atmosphere, response time necessary, and sensor size, weight and power consumption. There are a large number of sensors available to detect hydrogen (1-3). Each sensing method has its advantages and range of applications.

An ideal sensor would be one with zero size, weight, and power consumption which is operable in a wide range of environments for a long period of time with negligible calibration change. The sensor sensitivity, selectivity, and response time would be adequate to meet the needs of any application. The position of the leak could be determined by placing a number these sensors at locations near the source of the potential leak and monitoring the relative signal from each of these sensors. Such point-contact sensors that meet all the needs of aerospace applications do not presently exist (2-3).

We have begun work at NASA LeRC on the development of point-contact hydrogen sensors. This work, which began in response to hydrogen leaks on the Space Shuttle while on the launch pad, addresses the needs of aerospace applications in general. There are three major components of this program. One component involves the fabrication of PdAg Schottky diodes on Si substrates. A second is the investigation of PdCr as a hydrogen sensitive alloy for use in MOS systems or as catalytic resistors. The third involves the use of SiC rather than Si as the semiconductor in a metal-oxide-semiconductor (MOS) or metal-semiconductor (MS) structure. Each component of the program addresses different aspects of the fabrication of hydrogen sensors and each is at a different stage of development.

Two types of sensors which hold promise as point-contact sensors are those based on MOS technology and those based on the change of resistance of catalytically active resistors. The MOS type sensors are based on semicon-

ductor technology. The MOS structures are composed of a hydrogen sensitive metal deposited on an insulator adherent to a semiconductor. A Schottky diode is formed for a very thin layer of insulator. The most common MOS structure for hydrogen detection is the Pd-SiO_2-Si structure. Hydrogen disassociates on the Pd surface and diffuses to the Pd-SiO_2 interface affecting the electronic properties of the MOS system (4-6).

The use of catalytically active resistors is based on the concept that hydrogen migrates into the resistor and changes the resistance of the sensor. Common resistor materials are Pd or its alloys (7-10).

The speed and magnitude of response depends on the material and such factors as temperature, the ambient gas, and previous history of the sensor. The use of Pd as the hydrogen sensitive metal is problematic for several reasons. The most serious of these involves a phase change that occurs at high hydrogen concentrations. This stresses the film and can lead to hysteresis or film damage.

This paper discusses the motivation behind and present status of each of the major components of the NASA LeRC hydrogen sensor program. We will first discuss the testing facility used to test the sensors as well as the proposed expansion of the facility. We will then discuss the Schottky diode prototype sensors, the present characterization of PdCr, and the use of SiC as a semiconductor for a hydrogen sensor. An overview of future directions for the program will then be presented. It is concluded that results thus far are encouraging and further development work is necessary.

NASA LeRC HYDROGEN SENSOR TESTING FACILITY

The facility used for sensor testing is shown schematically in Figure 1. The facility can supply a continuous flow of a gaseous hydrogen and nitrogen mixture to a chamber containing the sensor under test. The hydrogen content of the mixture is adjustable from approximately 3000 parts per million (ppm) to 1,000,000 ppm (pure hydrogen). The test facility is outfitted with commercially available hydrogen detectors and a roof vent/fan to detect and

remove any gas that may leak from the system plumbing.

This system is intended to simulate hydrogen leakage from a rocket engine. A chilled gas mixture can be used to simulate cryogenic hydrogen leaking from an engine. A hydrogen-nitrogen mixture is used since often the atmosphere into which hydrogen from an engine will leak is mainly nitrogen. Also, the hydrogen-nitrogen mixture minimizes the ignition hazard during testing. A flowing system is used since it is assumed that leaking hydrogen would come from a pressurized system, resulting in streams of hydrogen.

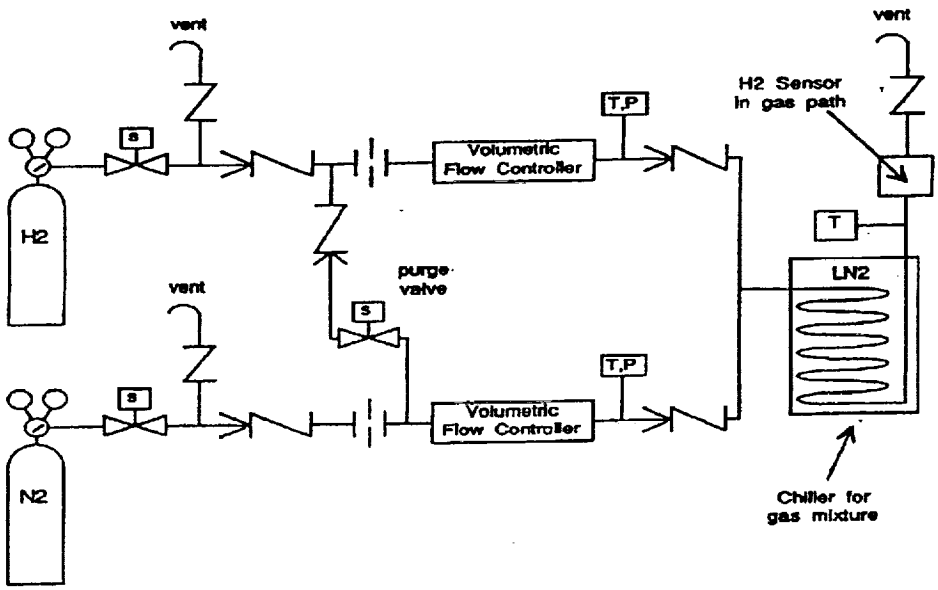

Figure 1. Schematic of the NASA LeRC H_2 sensor test facility. Bottled gases are regulated, flow controlled, mixed and pass through a chiller prior to flowing over the hydrogen sensor. T and P represent temperature and pressure measurements.

The flow of the gas stream is seen in Figure 1. The hydrogen and nitrogen are supplied from facility bottles with gas purity of 99.99%. Gas pressure is reduced and regulated to approximately 50 PSI. The gases pass through check valves and flow limiting orifices before entering the flow controllers. Hydrogen volumetric flow is adjustable from 0 to 100 standard cubic centimeters per minute (SCCM), and nitrogen volumetric flow from 0 to

500 SCCM by the computer controlled flow controllers. Gas temperature and pressure are measured at the controllers exits. Mass flow of the individual gases is calculated from the volumetric flow rate, and temperature and pressure of the gases. After passing through a set of check valves, the gases are mixed and enter a chiller. The chiller consists of a coiled section of copper tubing placed in a dewar which can be filled with liquid nitrogen. The temperature of the mixture can be controlled by regulating the liquid nitrogen level in the dewar. The mixture passes through the chamber containing the hydrogen sensor being tested, and exits via a roof vent. The computer is used to set the flow controllers, calculate mass flow, calculate the hydrogen concentration of the mixture, and record data.

The major features of the proposed expansion of the hydrogen sensor testing system are the expansion of the test chamber and addition of a mass spectrometer. The enlarged test chamber will allow the simultaneous testing of more than one sensor. A mass spectrometer will monitor the gas in the test chamber. This will allow an independent check on the gas composition in the test chamber and the performance of the sensor. Mass flow controllers will be used which will allow the flow of H_2, N_2, and He. The range of the mass flow controllers will also be expanded to 1000 SCCM to increase the range of flows and hydrogen concentration levels available.

RESULTS AND DISCUSSION

PdAg SCHOTTKY DIODES

Schottky diodes using Pd13%Ag as the hydrogen sensitive metal are presently being fabricated at Case Western Reserve University (CWRU) and tested at CWRU and NASA Lewis. The use of PdAg in hydrogen sensing applications was pioneered by Hughes (11-12). The advantages of PdAg over Pd include the ability to be exposed to higher concentrations of hydrogen without damage to the film and improved sensor speed and aging characteristics. The 13% Ag concentration is considered to be the optimum concentration for balancing these improved properties and the need the have a large enough Pd concentration to allow sensitivity to hydrogen (13).

The configuration of the CWRU structure is seen in Figure 2. The system includes a Pd13%Ag Schottky diode, a temperature sensor, and a heater all incorporated in the same chip. The sensor is fabricated using a n-type silicon wafer on which approximately 50 Å of SiO_2 is thermally grown in the sensor region. The heater is formed by diffusing p type boron into the substrate forming a resistance heater while the temperature sensor is formed by diffusing p type boron and n type phosphor into the substrate forming a diode. Appropriate mixtures of Pd and Ag are deposited by thermal evaporation onto the SiO_2 to yield films 300 Å thick. Gold leads are applied by thermal compression bonding and mounted on a TO5 header or on a ceramic flat package.

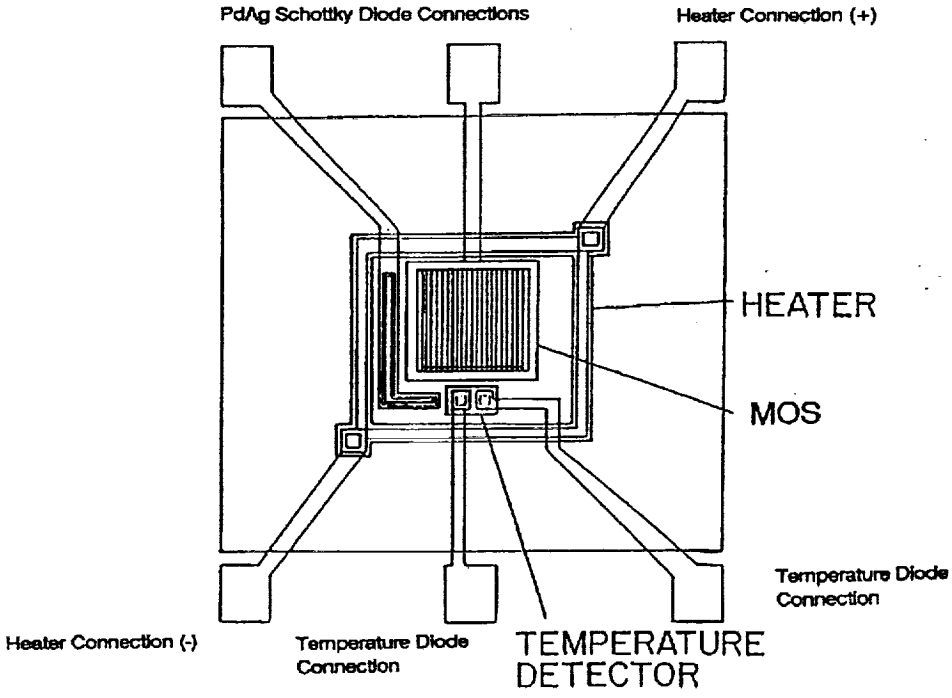

Figure 2. Schematic diagram of the Pd13%Ag Schottky Diode Hydrogen Sensor. Included on the sensor are a Pd13%Ag Schottky diode, a heater, and a temperature diode. Noted on the figure are the location of each device and their respective connections. The sensor is a square with sides 5 mm in length.

The response of the diode to changes in hydrogen concentration is

measured by monitoring the reverse current. The reverse current is measured by applying 5 V across a circuit composed of a resistor in series with the sensor diode and measuring the voltage drop across the resistor. The behavior of the reverse current in an air oven is shown in Figure 3 at temperatures from 20 °C to 160 °C. The reverse current is near zero at room temperature and, as would be expected theoretically for thermonic emission in a Schottky diode, increases exponentially with temperature (14). At temperatures greater than 120 °C the reverse current is very sensitive to temperature changes and some scatter in the data is noted. The operating temperature of the sensor must be chosen so as to maximize sensitivity and speed without degrading the stability or repeatability. Thus, in order to insure that the measured reverse current is a stable and repeatable baseline, the diode temperature is kept below 110 °C.

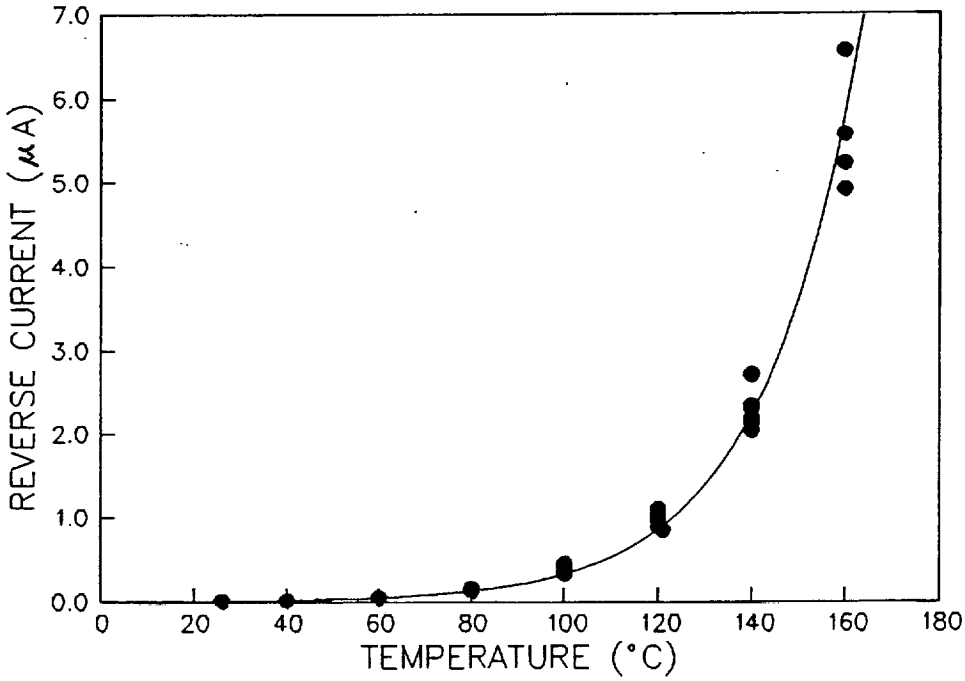

Figure 3. The reverse current of the PdAg Schottky diode over several cycles in an air oven from room temperature to 160 °C.

The response of these temperature controlled Schottky diodes to changes in ambient gas was investigated. The insert in Figure 4 shows the behavior

of the reverse current at 100 °C to 100 SCCM flowing N_2. A significant change in the reverse current is noted with exposure to flowing N_2. Figure 4 shows that further changes in the reverse current occur when a diode, stabilized for overnight in flowing N_2, is exposed to 5200 ppm hydrogen in nitrogen. The response is rapid with the reverse current in flowing hydrogen mixed with nitrogen nearly double that of nitrogen alone and 2 orders of magnitude greater than that measured in air. When the hydrogen flow is stopped, the recovery of the sensor in nitrogen is slow but a rapid recovery is noted when the sensor is removed from the test cell and exposed to air (oxygen). These results show the high sensitivity of the sensor to hydrogen as well as the vital role oxygen, which draws hydrogen out from the metal, plays in assuring a rapid recovery time. The sensor response in pure nitrogen (an oxygen deficient environment) is unexpected and will be investigated in the future.

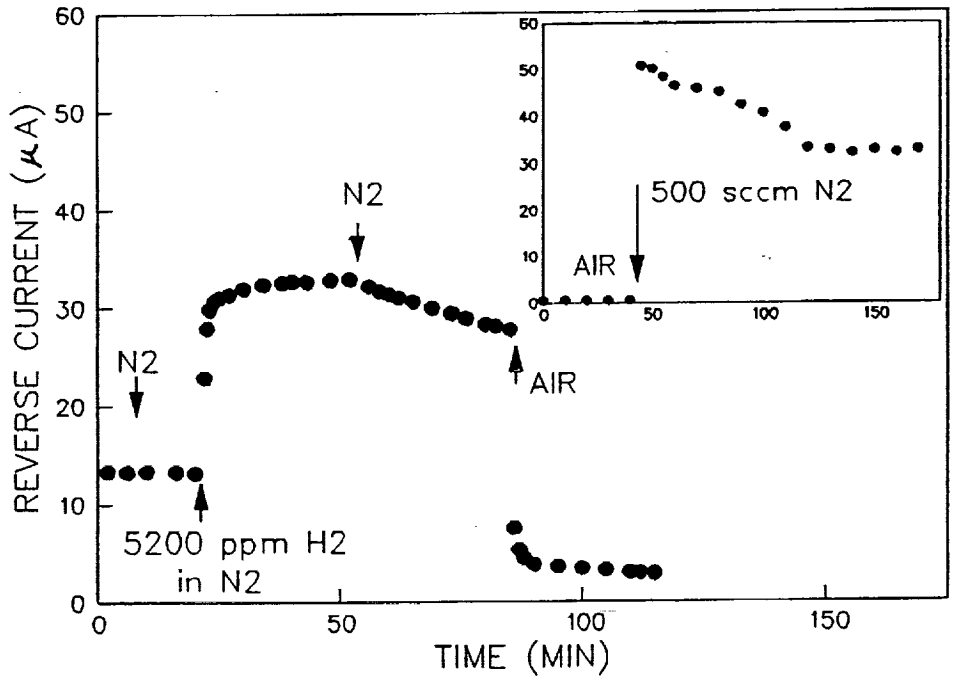

Figure 4. The response of the PdAg Schottky diode sensor at 100 °C to changes in gas composition. Inset: The sensor responds to the flow of pure nitrogen (500 SCCM). Figure: The sensor is stabilized overnight in 500 SCCM N_2. The gas composition is changed to 5200 ppm H_2 in N_2, then to pure N_2, followed by exposure to air.

CHARACTERIZATION of PdCr

Recently, PdCr has been developed as a strain gage material for high temperature applications (15). Temperature compensated with a Pt resistor, thin film PdCr strain gages have small apparent strain with good reproducability upon repeated thermal recycling to 1000 °C. The optimum Cr concentration for this application is 13 weight percent.

We have begun investigating the hydrogen sensitivity of PdCr for two reasons. The alloying of Pd with other metals has previously produced improved hydrogen sensing alloys (7, 11-12). An alloy that is as mechanically and electrically stable as PdCr may have increased durability to the expansion/contraction that accompany hydrogen adsorption/desorption and may thus provide an improved hydrogen sensing material.

A second motivation in investigating the response of PdCr to hydrogen is to characterize possible complications in using this material as a strain gage in applications where it might be exposed to hydrogen. An example of such an application is use in an hydrogen-oxygen engine.

In order to characterize the hydrogen sensitivity of PdCr, a number of Pd13%Cr and Au films were sputter deposited on to alumina substrates. The Au films were deposited as hydrogen insensitive reference resistors. Resistive elements in the form of strain gage patterns were formed using standard photolithography and etching techniques. Gold pads were deposited at the base of the resistor pattern for lead wire connections. Copper lead wires were bonded to the Au film using silver epoxy. The alumina substrate was then bonded to a 8 pin DIP connector which was mounted in the testing system.

The resistance of the thin film was determined using a constant current source and measuring the voltage drop across the film. The baseline resistance of the Pd13%Cr, R_0, was determined in flowing nitrogen. The flow rate of the N_2 was varied from 100-500 SCCM to verify that the effect of the flow rate on R_0 was less than 0.05% and thereafter kept constant at 100 SCCM. After R_0 was established, the hydrogen concentration was increased from 0 to

100% and back to 0%. Twenty minutes elapsed between hydrogen concentration changes although a steady state value was reached within 3 minutes. The input power was kept nearly the same for all resistors tested.

Preliminary results suggest that the resistance of Pd13%Cr is sensitive to hydrogen concentration. Figure 5 shows the percent change in resistance from the baseline, $\%_{\Delta}R/R_0$, of a 1000 Å thick Pd13%Cr resistor and an approximately 100 Å Au resistor at room temperature. There is a change in the Pd13%Cr resistance with hydrogen concentration. These changes are reversible and repeatable as shown in the figure. The maximum percent change in resistance is near 1.1% at hydrogen concentration of 100%. After the hydrogen flow is stopped, the resistance drifts back to R_0 with a time constant on the order of 50 minutes. The limited response of the Au reference resistor suggests that the Pd13%Cr response is not due to factors other than the interaction of the hydrogen with the Pd13%Cr.

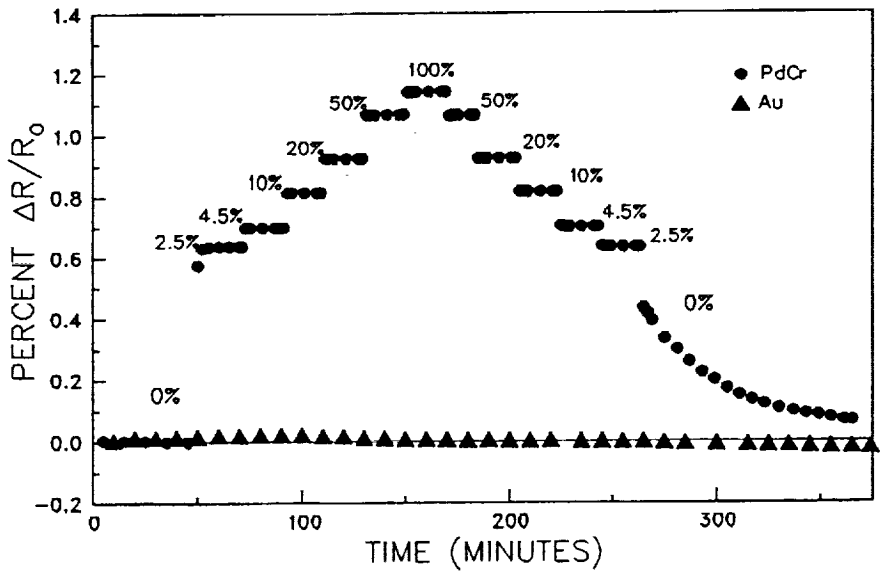

Figure 5. The percent change in resistance vs time of a Pd13%Cr resistor at room temperature. The hydrogen concentration is changed from 0% to 100% and back to 0% in set intervals.

Figure 6 shows the relation between $\%\Delta R/R_0$ and hydrogen concentration for the data of Figure 5 as well as that for lower concentration measurements of this sample. The response of the resistor saturates at higher hydrogen concentrations and is sensitive at lower concentrations. No degradation of the resistor was observed due to the cycling in hydrogen.

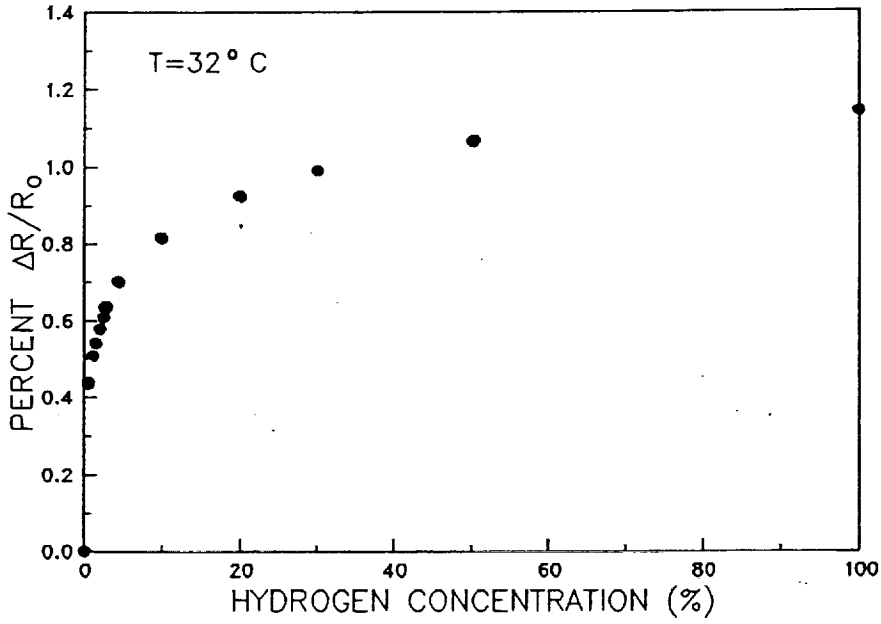

Figure 6. The percent change in resistance vs hydrogen concentration for the resistor shown in Figure 5.

Sensors Based on Silicon Carbide as a Semiconductor

The sensitivity of MOS or MS hydrogen sensors depends on a number of factors including the properties of the semiconductors. Silicon is the dominant semiconductor for most sensor applications. However in some gas sensing applications, there are significant advantages to using silicon carbide (SiC) as the semiconductor instead of silicon.

In particular, material properties of silicon carbide make it suitable for operation in hostile conditions which would strain or exceed the inherent

limitations of silicon-based electronics (16-17). The demonstrated ability of silicon carbide devices to operate at temperatures unattainable in silicon technology should enable the fabrication of high-temperature semiconductor sensors. SiC device functionality at temperatures higher than 600 °C is made possible by its wide bandgap and low intrinsic carrier concentration; silicon devices are limited to operation below 300 °C. The leakage current of Schottky diodes, which could potentially limit the sensitivity range of some sensor designs, is generally orders of magnitude less in SiC than silicon. Another advantageous property of silicon carbide is its superior mechanical toughness (it is often used in drill bits and saw blades), which should enable it to better withstand the possible thermal shocks associated with cryogenic fuel systems. The increased thermal conductivity of SiC should aid heat distribution in on-chip heating schemes (like the one presented in Figure 2). Silicon carbide is also a superior material in environments where nuclear radiation would severely degrade the performance of Si based semiconductors (18).

Palladium MS Schottky diodes with SiC as the semiconductor were prepared in the following manner. An 8 μm thick 6H-SiC epilayer (doped n-type near 1 x 10^{16} cm^{-3}) was grown by chemical vapor deposition on a commercially available n^+ 6H (0001) carbon-face 3-5° off-axis SiC substrate (19-20). A few thousand angstroms of palladium metal were then sputter deposited onto the as-grown 6H-SiC epilayer surface. Circular Pd Schottky patterns were then produced using standard photolithographic pattern definition and wet chemical etching in 1 HCl : 1 HNO_3 : 2 H_2O. Device fabrication was completed by sputtering nickel onto the bottom of the wafer to form a backside contact. Contacts to the Pd surface were made with a tungsten probing needle mounted on the sample holder.

The first results using SiC as a semiconductor in a hydrogen sensitive Schottky device are shown in Figure 7. The forward current is measured at 1 V while the sensor is exposed to 500 SCCM of flowing nitrogen at room temperature. The sample is then exposed to 5200 ppm H_2 in flowing nitrogen. The forward current is seen to increase by nearly a factor of 5. Figure 7 shows the response of the SiC based diode when the hydrogen flow stops and

the diode is exposed to pure nitrogen then air. The qualitative similarities to Figure 4 are evident: the SiC based Schottky diode responds to hydrogen rapidly, has a slow recovery time in pure nitrogen, and a much quicker recovery time in air.

Typical I-V curves taken while the diode is stabilized in nitrogen and hydrogen are shown in Figure 8. The magnitude of the reverse current and the forward current below 0.5 V were below the resolution of the instrument. As Figure 8 shows, the introduction of hydrogen into the Pd lattice has significantly changed the I-V characteristics of this MS structure. The forward voltage of 1 V is seen to be a reasonable voltage at which to measure of the changes occurring in the forward current. Further results and analysis will be presented in an upcoming paper.

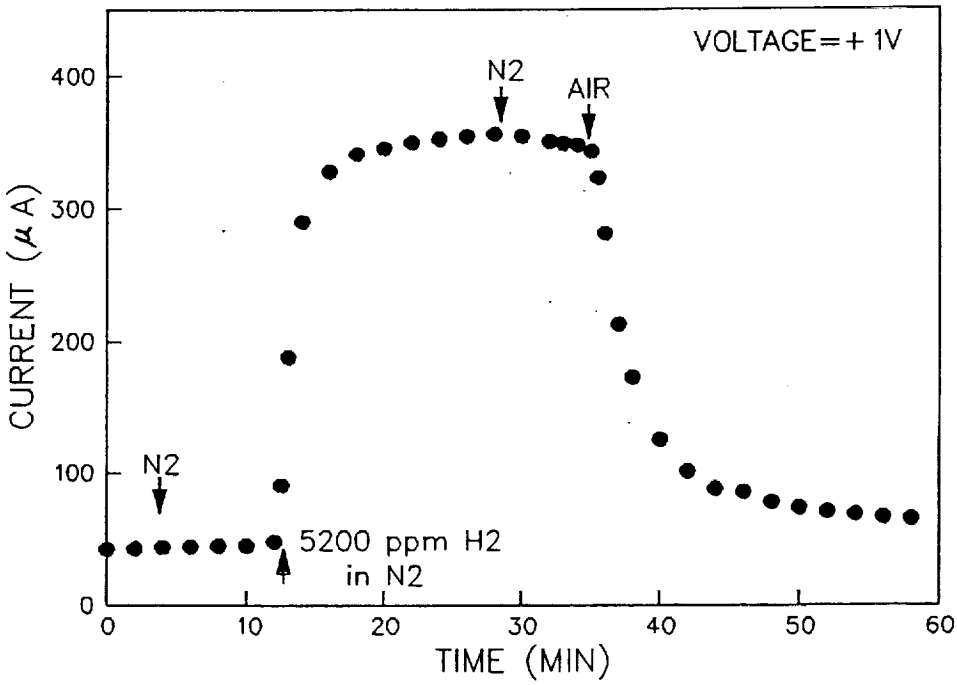

Figure 7. The response of the Pd-SiC Schottky diode at room temperature to changes in gas composition. The sensor is stabilized in 500 SCCM N_2. The gas composition is changed to 5200 ppm H_2 in N_2, then to pure N_2, followed by exposure to air. The diode's response is qualitatively similar to that of the PdAg-SiO_2-Si diode shown in Figure 4.

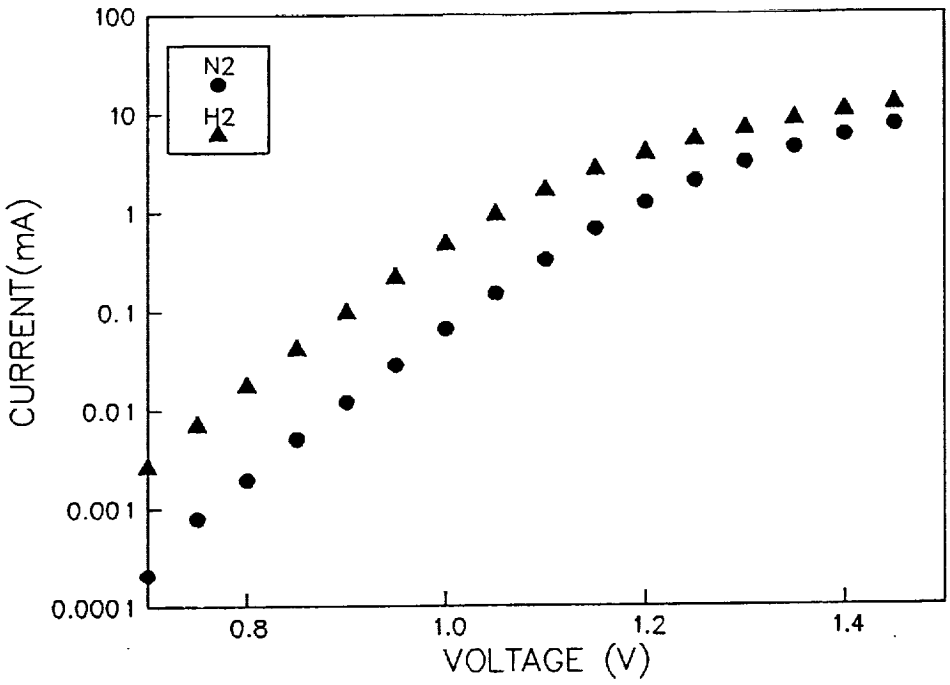

Figure 8. Comparison of the forward I-V characteristics for a Pd-SiC Schottky diode in nitrogen and hydrogen.

CONCLUSIONS AND FUTURE PLANS

The program at NASA Lewis in cooperation with Case Western Reserve University is still in its formation period. The progress thus far on all three components of the program are very encouraging. The Pd13%Ag Schottky diode structure combines a hydrogen sensitive sensor with the basic elements necessary for low temperature operation: a temperature sensor and a heater. The preliminary results suggest that the resistance of the Pd13%Cr alloy changes with hydrogen concentration. The resistance change is reproducible within a single cycle and approaches saturation as the hydrogen concentration approaches 100%. The demonstrated introduction of SiC as a semiconductor for MS or MOS devices opens a range of new possible application for hydrogen sensors.

The future direction of this program is to determine which type of

sensor(s) would best meet the needs of aerospace applications and continue development work in this area. From the preliminary results discussed above, the near term research will emphasize the following areas.

We will continue to characterize the Pd13%Ag Schottky diode sensors in a variety of conditions. These include operation in high concentrations of hydrogen, in pure nitrogen and pure helium, and at low temperatures. Investigation of the sensor response in oxygen deficient environments will help define the range of applicability of these sensors. The production of a number of different sensor types that can span the range of hydrogen concentrations, are very reproducible, and can operate at low temperatures is a goal of this part of the program.

The hydrogen sensing properties of PdCr will continue to be explored. The change of resistance with temperature and hydrogen concentration will be measured for several Cr concentrations and surface preparations. A second characterization method will include depositing PdCr on a quartz crystal. When hydrogen flows over the PdCr, the PdCr-quartz crystal resonant frequency will change in proportion to how much hydrogen is adsorbed. The amount of hydrogen absorbed by the PdCr can be determined by measuring the frequency change. This would provide an independent confirmation of the hydrogen sensitivity of PdCr as well as more detailed information on the hydrogen phase diagram of this alloy.

The response of the SiC based Schottky diode to hydrogen is encouraging. These results show the use of SiC in hydrogen sensing structures is feasible. The near term goals of this part of the program will be to compare the behavior of the SiC based diodes to the Si based diodes and examine the sensor response in a range of temperatures and hydrogen concentrations. Long term goals include integrating the SiC-based hydrogen sensor into a MOSFET structure (possibly with its own temperature sensor and heater) and testing in hostile environments in which Si-based sensors would not function.

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13. ABSTRACT ***(Maximum 200 words)***

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