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Microfabricated Chemical Sensors for Safety and Emission Control Applications

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Microfabricated Chemical Sensors for Safety and Emission Control Applications

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ABSTRACT

Chemical sensor technology is being developed for leak detection, emission monitoring, and fire safety applications. The development of these sensors is based on progress in two types of technology: 1) Micromachining and microfabrication (MEMS-based) technology to fabricate miniaturized sensors. 2) The development of high temperature semiconductors, especially silicon carbide. Using these technologies, sensors to measure hydrogen, hydrocarbons, nitrogen oxides, carbon monoxide, oxygen, and carbon dioxide are being developed. A description is given of each sensor type and its present stage of development. It is concluded that microfabricated sensor technology has significant potential for use in a range of aerospace applications.

INTRODUCTION

Aerospace applications require the development of chemical sensors with capabilities beyond those of commercially available sensors. Three areas of particular interest are leak detection, emission monitoring, and fire detection. In launch vehicle safety applications, the detection of hydrogen leaks is important for applications involved with, for example, operation of the Space Shuttle. In 1990, the leaks on the Space Shuttle while on the launch pad temporarily grounded the fleet until the leak source could be identified. In response to the hydrogen leak problems, NASA endeavored to improve propellant leak detection capabilities during assembly, pre-launch operations, and flight. In particular, efforts were made to develop an automated hydrogen leak detection system using point-contact hydrogen sensors. However, no commercial sensors existed at that time that operated satisfactorily in this and other space related applications. For example, commercially available sensors often needed oxygen or depended upon moisture to operate.[1] Such sensors did not meet the needs of this application where hydrogen detection was necessary in inert environments or upon exposure to cryogenic environments.[2] Thus the development of new types of sensors was necessary.

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The development of a new class of sensors is also necessary for the monitoring of emissions from aircraft engines. The control of emissions from aircraft engines is an important component of the development of the next generation of these engines. The ability to monitor the type and quantity of emissions being generated by an engine is important in not only controlling those emissions but also in determining the status of the engine. Ideally, an array of sensors placed in the emission stream close to the engine could provide information on the gases being emitted by the engine. However, there are very few sensors available commercially which are able to measure the components of the emissions of an engine in-situ. The harsh conditions and high temperatures inherent near the reaction chamber of the engine render most sensors inoperable.

In fire safety applications, fire detection equipment presently used in the cargo holds of many commercial aircraft relies on the detection of smoke. Although highly developed, these sensors are subject to false alarms. These false alarms may be caused by a number of sources including: changes in humidity, condensation on the fire detector surface, contamination from animals, plants, or other contents of the cargo bay.[3] A second method of fire detection to complement existing techniques, such as the measurement of chemical species indicative of a fire, will help reduce false alarms and improve aircraft safety. Although, many chemical species are indicative of a fire, two species of interest are carbon monoxide and carbon dioxide.[4]

In order to meet the needs of these applications, a new generation of sensor technology must be developed. This paper discusses point-contact sensor technology being developed to address these needs. The development of these sensors is based on progress in two types of technology: 1) Micromachining and microfabrication technology (MEMS-based technology) to fabricate miniaturized sensors, and 2) The development of high temperature semiconductors, especially silicon carbide, to provide electrical components and sensors operable at the temperatures of interest. Using these technologies, point-contact sensors are being developed to measure hydrogen (H_2), hydrocarbons (C_xH_y), nitrogen oxides (NO_x), carbon monoxide (CO), carbon dioxide (CO_2) and oxygen (O_2). A description is given of each sensor type and its present stage of development. The silicon (Si) based hydrogen sensor is at a relatively mature stage of development while the state of development of the other sensors ranges from the proof of concept level to prototype stage. The number of dual-use commercial applications makes this general area of sensor development a field of significant interest.

SENSOR DEVELOPMENT

Hydrogen Sensor Technology

One component of the sensor development program at NASA Lewis Research Center (LeRC) and Case Western Reserve University (CWRU) involves the development of palladium (Pd) alloy Schottky diodes on silicon (Si) substrates. A Schottky diode is composed of a metal in contact with a semiconductor (MS) or a metal in contact with a very thin insulator on a semiconductor (MIS). For gas sensing applications, the metal is often a catalytic film. The advantage of a Schottky diode sensing structure in gas sensing applications is its high sensitivity: factors of over 1000 change in reverse current for 0.5% H_2.[5] Further, this type of sensor does not need oxygen for operation.

The sensor structure is shown in Figure 1. The structure includes a Pd alloy Schottky diode, a temperature detector, 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 and temperature detector are platinum resistors. Gold leads are applied by thermal compression bonding and the sensor is mounted on a TO5 header or on a ceramic flat package. The surface area of the Schottky diode is 6.1×10^{-3} cm^2 and the sensor dimensions are approximately 2.2 mm on a side. The response of the Schottky diodes was determined by measuring the diode's reverse current.

A hydrogen sensor using palladium chrome (PdCr) as the H_2 sensitive alloy is presently under development for use on the NASA experimental vehicle, the X-33, in its hydrogen leak detection system.[6] Palladium chrome was chosen due its ability to detect H_2 up to 100%. A resistor, whose resistance is dependent on the H_2 concentration, has been added to the sensor design to expand the detection range of the sensor (Figure 1); a Schottky diode provides sensitive detection of low concentrations of H_2 while the resistor (also made of PdCr) provides sensitivity up to 100% H_2. A complete system with hardware and software included with the sensor is included on the X-33 safety system. This complete system is also scheduled for a demonstration flight on the Shuttle for the fall of 1998.[6]

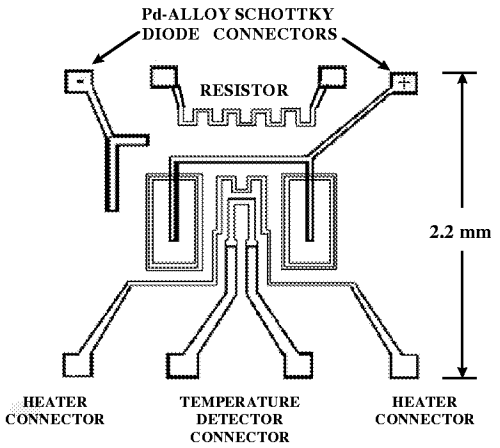


Figure 1. Schematic diagram of the Schottky Diode Hydrogen Sensor. The Pd alloy Schottky diode resides symmetrically on either side of a heater and temperature detector. The resistor expands the sensitivity range of the sensor package to 100% hydrogen.

Hydrocarbon Detection

The development of hydrocarbon sensors for use in harsh environments has centered on the development of a stable SiC-based Schottky diode. The advantage of SiC over Si is its ability to operate as a semiconductor at temperatures in excess of 600°C. This allows SiC-based gas sensors to operate at temperature high enough to allow the detection of hydrocarbons.[7] The first Schottky diode structure developed at NASA LeRC was Pd on SiC MS structures (Pd/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 between the Pd and SiC is necessary for long-term sensor stability. The sensor detects C_xH_y in inert or oxygen containing environments. The details of this work are reviewed elsewhere.[7]

Figure 2 illustrates the advantage of SiC over Si in C_xH_y sensing applications. Shown is the zero bias capacitive response of a Pd/SiC Schottky diode to one hydrocarbon (propylene) over a range of 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 air for 10 minutes.

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 C_xH_y gas sensing applications.

However, the Pd/SiC sensor response is affected by extended high temperature heating. Prolonged heating at 425°C has been shown to change the sensor properties and to decrease sensor sensitivity. The reason for this change in diode properties is likely due to reactions between the Pd and SiC at the interface upon heating.

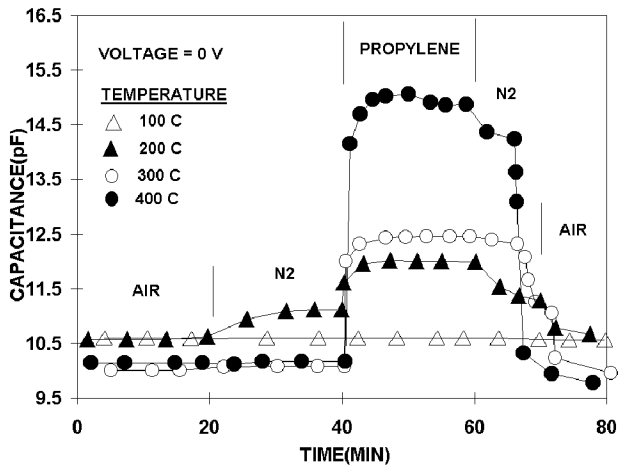


Figure 2. The temperature dependence of the zero bias capacitance to various gas mixtures. The response to propylene is seen to be strongly temperature dependent.

Efforts have been underway to stabilize the sensor structure for long-term, high temperature operation. Two new structures have been demonstrated which improve the stability of the Pd-based Schottky diode structure over that of Pd/SiC.[8] These structures involve 1) The use an alloy as the catalytic metal. 2) The use of a MIS structure where the "insulator" stabilizes the structure and is also reactive to the gases of interest. Preliminary testing of both of these structures suggests significantly improved stability and response over Pd/SiC.

Nitrogen Oxide and Carbon Monoxide Detection

A microfabricated and micromachined Si-based structure with a chemically sensitive resistor is being used to measure NO_x and CO detection. The Si is used as a platform on which the structure necessary for the sensor is fabricated. This sensor structure, shown in Figure 3, includes a temperature detector, heater, and sensing element. The microfabrication process allows the sensor to be small in size with low heat loss and minimal energy consumption. Energy consumption is further reduced by etching out the backside of the Si wafer so that the sensor components (temperature detector, heater, and sensing element) are over a diaphragm region. This minimizes the thermal mass of the sensing area thereby decreasing power consumption for heating and decreasing the time for thermal equilibrium. The temperature detector and heater are doped into the Si substrate for operation over a wide temperature range. The electrode material is Pt and the sensor dimensions are approximately 300 microns on a side with a height of 250 microns.

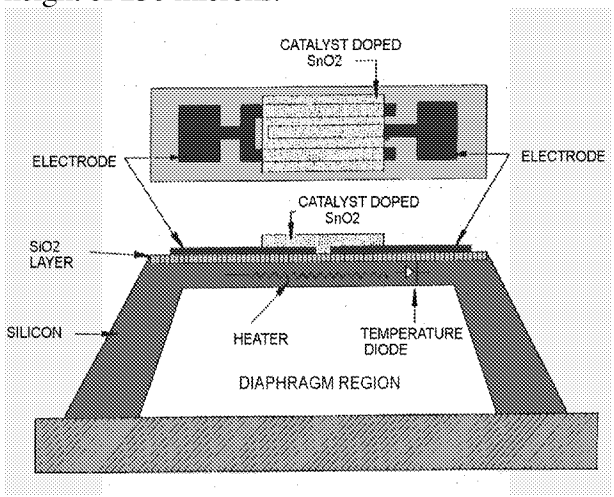


Figure 3. The structure of a tin-oxide NO_x and CO sensor including temperature detector, heater, and sensing element.

The sensing element is composed of interdigitated electrode elements across which is deposited SnO_2. Changes in conductivity of doped SnO_2 across the interdigitated electrodes is measured and correlated to NO_x

concentration. A major component of this development work is to stabilize the SnO_2 for long-term, high temperature operation. Drift in the properties of SnO_2 with long term heating due to grain boundary annealing have been previously noted.[9-10] This drift results in changes in the sensor output with time and reduces sensor sensitivity. In order to stabilize the SnO_2 structure for long term operation, the fabrication of nanocrystalline SnO_2 is being investigated. Nanocrystalline materials have several inherent advantages over conventionally fabricated materials including increased stability and sensitivity at high temperature.[11-12]

Several prototypes of these devices using nanocrystalline SnO_2 have been fabricated and evaluated. The sensitivity of the SnO_2 to NO_2 at 360°C is shown in Figure 4. The sensor is shown to be able to detect NO_2 down to the 5 ppm level. The sensor tends to saturate at higher NO_2 concentrations but the highest level of sensitivity is in the lower ppm range which is the region of interest. Similar results are seen with the detection of CO.

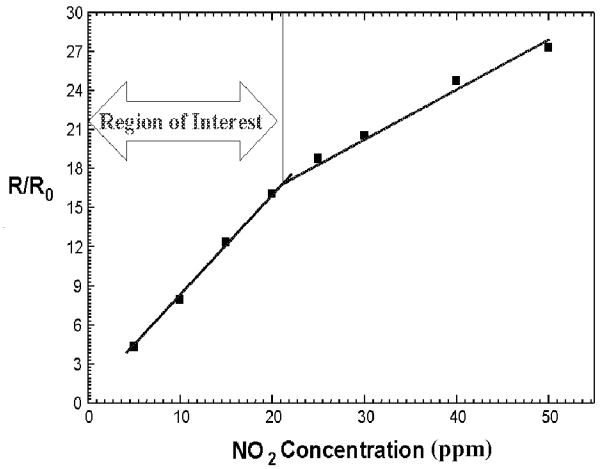


Figure 4. The response of a tin-oxide sensor to a range of NO_2 concentrations at 360°C.

Oxygen Detection

The development of a microfabricated O_2 sensor has been initiated for safety purposes in aerospace applications but significant applications also exist in the area of aeronautics emission control. Commercially available O_2 sensors are typically electrochemical cells using zirconium dioxide (ZrO_2) as a solid electrolyte and Pt as the anode and cathode.[13] The anode is exposed to a reference gas (usually air) while the cathode is exposed to the gas to be detected. Zirconium dioxide becomes an ionic conductor of $O^=$ at temperatures of 600°C and above. This property of ZrO_2 to ionically conduct O_2 means that the electrochemical potential of the cell can be used to measure the ambient oxygen concentration at high temperatures. However, operation of these commercially available sensors in this potentiometric mode limits the range of oxygen detection. Further, the current manufacturing procedure of this sensor is relatively labor intensive and costly resulting in a complete sensor package with a power consumption on the order of several watts.

The objective of this research is to develop a zirconium dioxide solid electrolyte O_2 sensor using microfabrication and micromachining techniques. The combination of a fuel with O_2 produces a hazardous environment. Thus, the measurement of both the fuel and O_2 simultaneously is useful in leak detection applications. Also, the presence of O_2 often affects the response of H_2, C_xH_y, and NO_x sensors. An accurate measurement of the O_2 concentration will help quantify the response of other sensors in environments where the O_2 concentration is varying. Therefore, the combination of an O_2 sensor with other microfabricated gas sensors is envisioned to optimize the ability to detect leaks and monitor emissions.

A schematic of the sensor design is shown in Figure 5. As discussed in the NO_x detection section above, microfabricating the sensor components onto a micromachined diaphragm region allows the sensor to be small in size and have decreased energy consumption and time for thermal equilibrium. When

operated in the amperometric mode, the current of this cell is a linear function of the ambient O_2 concentration. This linear response to oxygen concentration significantly increases the O_2 detection range of the sensor. A chamber structure with a well-defined orifice is micro-machined to cover the sensing area. This orifice provides a pathway to control oxygen diffusion which is important in amperometric measurements. This orifice also protects the integrity of the sensing electrode from impinging particles. Preliminary testing of the complete O_2 sensor has been accomplished and further improvements on the design are planned.

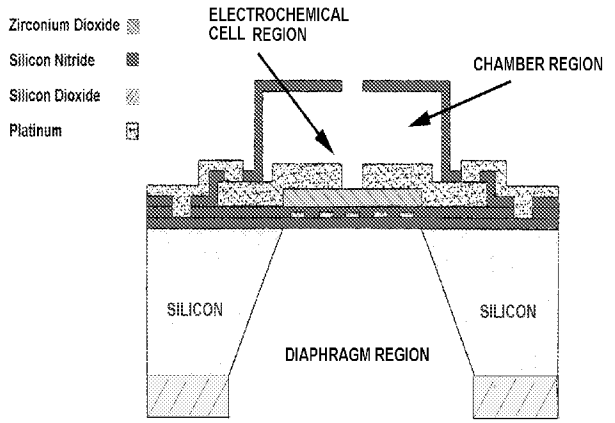


Figure 5. The structure of a microfabricated amperometric oxygen sensor. The dimensions of this sensor are comparable to that of the NO_x sensor shown in Figure 4.

CO2 Detection

The detection of CO_2 is, like the detection of oxygen, based on the use of a solid electrolyte. The significant difference from the O_2 sensor will be the use of NASICON (sodium super ionic conductor) as the solid electrolyte. NASICON is an ionic conductor composed of $Na_3Zr_2Si_2PO_{12}$ which has previously been shown to be sensitive for CO_2 detection. The preparation of the NASICON will be performed using a sol-gel technique. The sensor structure will be similar to that of Figure 5: a microfabricated, miniaturized sensor structure which can be incorporated with other sensors such as the CO sensor. A combined CO_2/CO sensor is of interest not only for fire safety applications but for combustion monitoring applications as well.

High Temperature Electronic Nose Concept

The successful development of the individual high temperature sensors discussed above will allow the formation a sensor array which will allow the detection of a number of gases on a single chip. For example, the formation of an array of the sensors discussed in this paper will detect H_2, C_xH_y, NO_x, CO, O_2 and CO_2. These gases will be detected using three different platforms: a Schottky diode, a resistor, and an electrochemical cell.

Development of such a micro-fabricated gas sensor array operable at high temperatures and high flow rates would be a dramatic step in allowing the monitoring and control of emissions produced by an aeronautic engine. This gas sensor array would, in effect, be a *high temperature* electronic nose and be able to detect a variety of gases of interest. Several of these arrays could be placed around the exit of the engine exhaust to monitor the emissions produced by the engine. The signals produced by this nose could be used to determine the constituents of the emission stream and this information then used to control those emissions. The microfabrication of these sensors is necessary: a conventional bulky system would add weight to the aircraft and impede the flow gases leaving the engine.

The concept of an electronic nose has been in existence for a number of years.[14] Commercial electronic noses presently exist and there are a number of efforts to develop other electronic noses. However, these electronic noses depend significantly on the use of polymers and other lower temperature materials to detect the gases of interest. These polymers are generally unstable above 400°C and thus would not be appropriate for use in harsh engine

environments. Thus, a separate development effort is necessary for a high temperature electronic nose.

COMMERCIAL APPLICATIONS

The gas sensors being developed at NASA LeRC and CWRU are meant for aeronautics and aerospace applications but can be used in a variety of commercial applications as well. For example, hydrogen sensors using palladium silver that were initially developed for application on the launch pad of the space shuttle. These sensors can be applied to an automotive application. GenCorp Aerojet Corporation, in conjunction with NASA Marshall Space Flight Center, has developed hardware and software to monitor and control the NASA LeRC/CWRU sensors. The system can be customized to fit the user's needs, e.g., to monitor and display the condition of the tank of a natural gas vehicle. Several of these systems have been purchased for use on the Ford Motor Company assembly line for natural gas vehicles (NGV). This complete system received a 1995 R&D 100 Award as one of the 100 most significant inventions of that year.[5]

Likewise, the high temperature H_2, C_xH_y, NO_x, and O_2 sensors are being developed for aeronautical applications but can be applied in commercial applications. For example, the conditions in an aeronautic engine are similar to those of an automotive engine. Thus, sensors that work in aeronautic engine applications may be operable in automotive engine applications. Other possible applications include catalytic reactor monitoring, alarms for high-temperature pressure vessels, and chemical plant processing.

SUMMARY

The needs of aerospace applications require the development of sensors with capabilities beyond those of commercial sensors. These requirements include operation in harsh environments, high sensitivity, and minimal size weight and power consumption. Sensor technology is being developed to address these requirements using microfabrication and micromachining technology as well as SiC semiconductor technology. Several types of sensors have been described all of which have aeronautic and space applications. Some of the sensor designs are relatively mature while the development of others is ongoing. The combination of these technologies may allow the development of a high temperature electronic nose to provide complex chemical analysis in harsh environments.

Sensors designed for aeronautic and space applications also have significant commercial applications. One example is the use of the hydrogen sensor in automotive applications. This application involved a sensor not completely developed for a space application but with excellent properties for use in the automotive application. Further, given the similarities of aeronautic engine environments to those of automotive engine environments and chemical process monitoring, sensors developed for aeronautic applications also have a wide range of applicability. Although each application is different and the sensor needs to be tailored for that environment, the base technology being developed for aeronautic and space applications can have significant impact on a range of fields.

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