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Schottky Diodes with Thin Catalytic Gate Metals for Potential Use as Ammonia Sensors for Exhaust Gases

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Selective catalytic reduction (SCR) is a method in which ammonia reacts with nitric oxides in a catalytic converter to form water and nitrogen. We show that catalytic Metal Insulator Silicon Carbide (MISiC) devices can be used as ammonia sensors for a SCR system in a diesel engine. Different catalytic metals, Pt and Ir, with a thickness of 30 or 50 nm were investigated. The temperature dependence of the ammonia response of the sensors was characterized. Maximum responses were found at temperatures between 225–250°C. Preliminary experiments were performed to investigate how annealing in different gas ambient influences the response-temperature curve of the sensors. In synthetic diesel exhausts with ammonia added, the sensors showed very good selectivity for ammonia and a small interaction effect with oxygen. The influence of other gas components was almost negligible. Temperature in the diesel exhaust system can reach 550°C; however, operating at temperatures above 400°C limited the lifetime of the sensor. Auger electron spectroscopy (AES) revealed that island formation of the metal due to structural changes was the main reason for failure of the sensor.

1. Introduction

Nitric oxides (NO_x) are poisonous compounds formed at high temperatures in an internal combustion engine when N_2 is oxidized. In the presence of hydrocarbons and

sunlight, NO_x may form smog, containing for example, ozone, which is dangerous to humans and will damage green plants. Ammonia (NH_3) can be used to reduce NO_x using selective catalytic reduction (SCR) which is a technique developed for stationary power plants. However, research is under way to enable the application of this technique to vehicles. The concept is to reduce NO_x emission by injecting NH_3 or a molecule containing amine such as urea into the exhaust system of a diesel engine. In a catalytic converter, NH_3 and NO_x will react with each other to form N_2 and H_2O.

When using SCR in vehicles, temperature and flow of the exhaust are problems that should be solved. An efficient ammonia sensor must be able to monitor the NH_3 concentration accurately before and after passage through a catalytic converter so that it can be used as a feedback control for the amount of NH_3 injected. A fast and accurate ammonia sensor would lead to a more efficient reduction of NO_x without emitting excess NH_3 to the atmosphere. A thin catalytic gate of platinum or iridium on a metal insulator semiconductor device is known to perform very well as an ammonia sensor, while a metal gate of dense platinum does not show sensitivity to NH_3 in air.[1]

A comparison of the physical properties of SiC with those of Si indicates that SiC is superior to Si in many application areas. For example, Si-based metal-oxide semiconductor field-effect transistor (MOSFET) sensors can only be operated up to about 200°C, while the optimum temperature for NH_3 detection is between 225°C and 250°C, as shown for SiC-based Schottky diode devices in § 3. This tendency was also shown for Si-based capacitor devices, which have the disadvantage of requiring expensive electronics for measurements.[2] In general, SiC extends the temperature range for exploration of the properties of catalytic metals using field-effect devices. It is also possible to heat SiC-based sensors to regenerate them; moreover, the material has very good chemical stability, which makes it suitable for use in harsh environments, such as the exhaust systems of cars and trucks.

In this paper, metal insulator silicon carbide (MISiC) Schottky diodes with thin catalytic metal gates have been tested as ammonia sensors. Their electrical properties were investigated from the *I-V* curves, and sensitivity to NH_3 was monitored as a function of temperature. Some preliminary studies have been carried out on the sensor response to NH_3 after exposure to different gases at different temperatures. Experimental design of synthetic diesel exhausts was used to investigate the sensitivity to NH_3 in various exhaust gases. Concentration levels of NH_3 corresponding to those prior to passage through the converter were used in the experiments. Failed sensors were analyzed by Auger electron spectroscopy (AES) and scanning electron microscopy (SEM) and compared with fresh samples.

2. Experimental

2.1 *Gas sensing principle*

When MISiC Schottky diode sensors are exposed to hydrogen, hydrogen molecules dissociate on the metal surface and hydrogen atoms diffuse rapidly (within microseconds) through the metal and form a polarized layer at the metal-oxide interface. This polarized

layer shifts the current-voltage (*I-V*) curve to a lower voltage, and the gas response is measured as a shift in the forward bias at a constant current. This process is well known since Lundstöm *et al.* reported the phenomenon in 1975.[3] It was also described in a recent review article.[4]

The change in the forward bias at a constant current is also a function of the concentration of NH_3 in the ambient; however, the sensing mechanism for NH_3 is not fully understood. The existing model is that the NH_3 molecule adsorbs on the metal, or metal oxide boundaries, and reacts with oxygen. Reaction products diffuse out onto the oxide exposed by holes and cracks in the metal film (Fig. 1). Reaction products with strongly polarized adsorbate complexes on the metal and/or on the oxide will give rise to a shift in the forward *I-V* curve.[1, 2] In the case of Ir as the gate metal, recent research suggests that hydrogen from the decomposed NH_3 molecules is the measured species.[5]

2.2 *Sensor preparation*

The sensors used in these experiments are based on MISiC Schottky diodes. They are manufactured in the following way: silicon carbide, 6H wafers, n-type (nitrogen doped to 1.7×10^{18} cm^{-3}) with a 10 μm n-type epilayer (4.5×10^{15} cm^{-3}) were used.[6] The cleaning procedure involved several steps ended by a final etching in 50% HF.[7] Thereafter, the wafer was oxidized at 1,245°C in dry oxygen and annealed in argon. The oxide formed was etched off to leave a fresh SiC surface and the wafers were reoxidized following the same procedure. The obtained oxide thickness was measured by ellipsometry to be 100–150 nm. The oxide on the back of the wafer was etched, while the front side was protected by photoresist. After removing the photoresist, ohmic back contacts consisting of 200 nm $TaSi_x$ and 400 nm Pt were deposited. The Pt coating of $TaSi_x$ served as protection of the

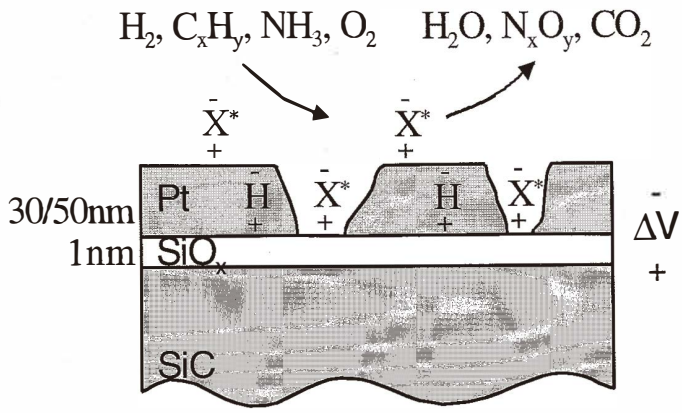

Fig. 1. Schematic cross section of a porous Pt-gate sensor. The SiO_2 layer typically has a thickness of approximately 1 nm and the Pt film is 30 or 50 nm thick. Some possible catalytic reactions on the sensor surface are also indicated.

back contact against oxygen during subsequent high-temperature measurements in air. The $TaSi_x$ formed an ohmic contact with the heavily doped substrate and simultaneously increased the adhesion to the substrate. Contact pads of 200 nm $TaSi_x$ and 400 nm Pt were deposited on the oxide on the front of the wafer. The back contacts and contact pads were deposited by DC magnetron sputtering. After removing the oxide in the gate areas with 50% HF, the SiC was cleaned with ozone, which formed a native oxide with a thickness of approximately 1 nm.[8] Finally, the electrodes that are active in the gas sensing process, the gate electrodes, were deposited by thermal evaporation. In this case 30 or 50 nm Pt and 30 or 50 nm Ir was deposited through a shadow mask of copper. A cross section and a top view of the sensor can be seen in Fig. 2. The thin metal film does not cover the oxide completely and there are holes and cracks in the film (Fig. 1).

Electrical connections were made with Pt ribbons that were welded to the sensors, and the sensor chip containing two devices was mounted with ceramic glue on a ceramic rod that could be heated. The temperature of the ceramic rod was measured and regulated with a chromel/alumel thermocouple. The ceramic rod was mounted in a stainless steel holder through which gas was flowing. A computer with an A/D card recorded the sensor signal, i.e., the forward bias, at 0.5 mA. The computer also controlled the valves and the mass flow controllers on the gas mixing board. The gases were of 99.99% purity or better.

Regarding the influence of different annealing conditions on the sensor response, other experimental equipment was used. Tests were performed with an improved mounting, heating and measurement procedure. The sensors were glued three by three to a platinum heater[9] together with a SiC pn diode for temperature control, and the assembly was welded to a 16-pin holder. The sensors and the pn diode were gold-bonded to the 16-pin holder, which was placed in an aluminum block where the sensors were exposed to the gases.

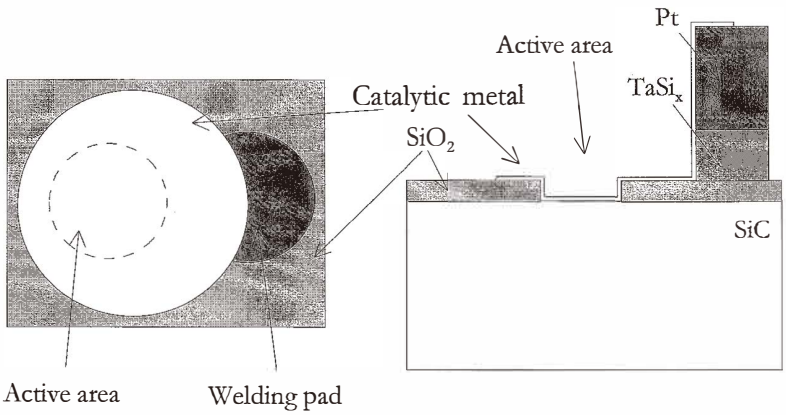

Fig. 2. Schematic top and side view of the completed sensor device (note the drawings are not to scale).

2.3 *Measurements*

The gases were mixed using eight mass flow controllers and five valves controlled by a computer. The *I-V* characteristics of the sensors were measured. The current was measured as the voltage over a 1000 Ω resistor. When measuring gas responses, a constant current (0.1 or 0.5 mA), controlled with external electronics, was passed through the sensors and the voltage was recorded as sensor signal.

The two-level factorial design[10] is a simple way of performing experiments and provides highly informative evaluation. The output (sensor signal) of an experiment depends on different factors (gases). The two-level factorial design means that two levels (concentrations) are selected for every factor. For n gases, there are 2^n different gas mixtures and hence 2^n outputs for each sensor. The different gas mixtures are applied randomly. The gases used and their high and low levels are shown in Table 1. The concentrations represent those of typical diesel exhaust. For these measurements, we have used an experimental design called the two-level fractional factorial design with resolution IV, which is generated by commercially available software.[11] We have used this software to evaluate the results. The reduced number of runs (i.e., mixtures) is sufficient for calculating the necessary information.

The two-level factorial design predicts *main* and *interaction* effects. The main effect of a factor is defined by taking into account the change of the output at all levels of all other factors. For example: *main effect* (factor 1) = average of outputs at the high level of factor 1 minus average of outputs at the low level of factor 1. This is visualized in Fig. 3. *Interaction effect* (factor 1 × factor 2) = effect (factor 1) at the high level of factor 2 minus effect (factor 1) at the low level of factor 2.

We have used a two-level fractional factorial design with six midpoints. For seven gases (Table 1) and six midpoints, a total of $2^7/2 + 6 = 70$ runs are generated for each experiment. The midpoints contain identical gas mixtures with concentrations of gases between the high and low levels; the midpoints are used to calculate the standard deviation and thereby the 95% confidence interval in the measurement. Each run lasted for 90 seconds, which was the minimum time required for the mass flow controllers to stabilize. Each set of runs was randomly tested for a certain operation temperature of the sensors. In total, four different operation temperatures were tested twice.

Table 1
Concentrations for different gas components in simulated car exhausts from a diesel engine.

Type of gas	Low level	High level
Carbon dioxide	3%	9%
Carbon monoxide	50 ppm	250 ppm
Oxygen	6%	12%
Nitric oxides	200 ppm	1,000 ppm
Hydrocarbons	130 ppm	260 ppm
Ammonia	200 ppm	1,000 ppm
Water	1%	4%

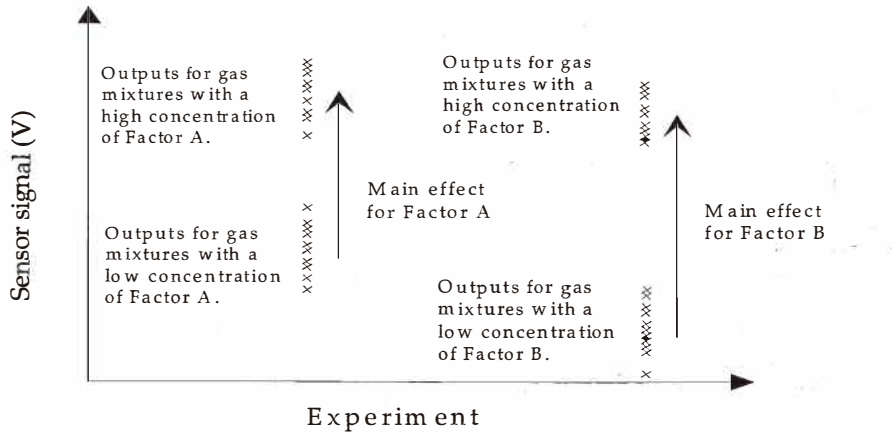

Fig. 3. Principle of a main effect when using experimental design. The main effect for a specific gas component is the average of all sensor signals at a high concentration of the specific gas component minus the average of all sensor signals at a low concentration of the same gas component.

3. Results

The sensors in this study were tested in three ways. First, the electrical properties were investigated based on the *I-V* curves. The sensitivity to NH_3 was tested at different temperatures and after different annealing procedures. The aim was to evaluate how exposures at different temperatures affected the response versus temperature curve and at what temperature the sensors stopped operating. Finally, the sensors were exposed to synthetic diesel exhausts. The above-described experimental design was used for synthetic diesel exhausts measurements.

3.1 I-V *measurement*

I-V measurements were carried out to check the sensor characteristics for O_2 and H_2. Three *I-V* curves, in 1% O_2/N_2, 3% H_2 in 1% O_2/N_2, and then in 1% O_2/N_2 again, were recorded at two temperatures, 300°C and 400°C. A good sensor should display a diode-like *I-V* curve with a reversible shift for H_2. In Fig. 4, a relatively good *I-V* curve from a 30 nm Pt sensor is shown. The ideality factor of this sensor is approximately 2. The ideality factor varies considerably between different sensors. Earlier findings have shown that even resistivity changes in the gate contact might contribute to the gas response.[12]

3.2 *Response vs. temperature curves*

Response versus temperature curves were measured for 250 ppm NH_3 in 20% O_2/N_2 at different temperatures. The response is measured as the difference between the sensor signal in 20% O_2/N_2 for 3 min and 250 ppm NH_3 in 20% O_2/N_2 for 2 min. The signal is an

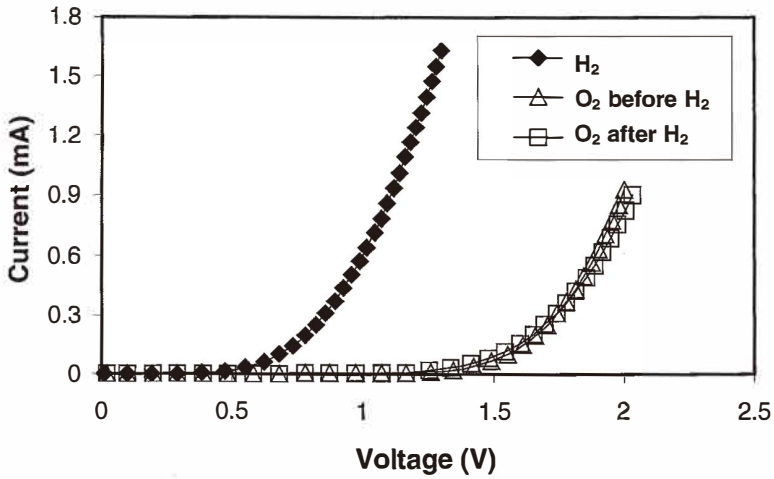

Fig. 4. Typical *I-V* curves in the forward direction for a MISiC Schottky diode sensor with 30 nm Pt as gate metal at 400°C. The sweep velocity was 0.1 V/s. The three curves are measured in the following order: in 1% O_2/N_2, in 3% H_2 in 1% O_2/N_2, and finally in 1% O_2/N_2 again. The curve on the left is measured with H_2 and the two curves on the right are measured without H_2. The voltage difference at 0.5 mA is used as the sensor response.

average of the signal during the last 5 s of each experiment. The response below 200°C was not the steady state response since the response time increased at lower temperatures. The sensitivity maximum for NH_3 was obtained at approximately 225°C for both Pt and Ir sensors (Fig. 5). Some temperature variation of the sensitivity maximum occurred, which could be due to a rather sharp response peak at the maximum temperature and large temperature steps or due to variations between sensors.

3.3 *Dependence on annealing of the response vs. temperature curves*

Pt and Ir sensors were formed with a gate metal thickness of 50 nm. They were exposed to H_2 and NH_3 pulses, for 15–30 min to 3 days at different temperatures. The NH_3 response versus temperature was analyzed after every exposure. For the annealing experiments in different ambients the mounting on the 16-pin holders was used (§ 2.2).

The response to NH_3 of the Ir sensors showed a large dependence on the history of the gas exposure. Higher temperature annealing tends to shift the response maximum for NH_3 towards higher temperatures. After a 30 min exposure at 350°C to 30 s pulses of 5% H_2 in a carrier gas of 2% O_2/N_2 with 60 s between the pulses, the response maximum for NH_3 was approximately 175°C, which rose to 225°C after the same treatment at 500°C (Fig. 6). Exposures to H_2 pulses tend to suppress the response to NH_3, thereby flattening the response versus temperature curve. Long runs with NH_3 pulses appear to lead to the restoration of the response curve to its original state. This can be seen in Fig. 7, where the

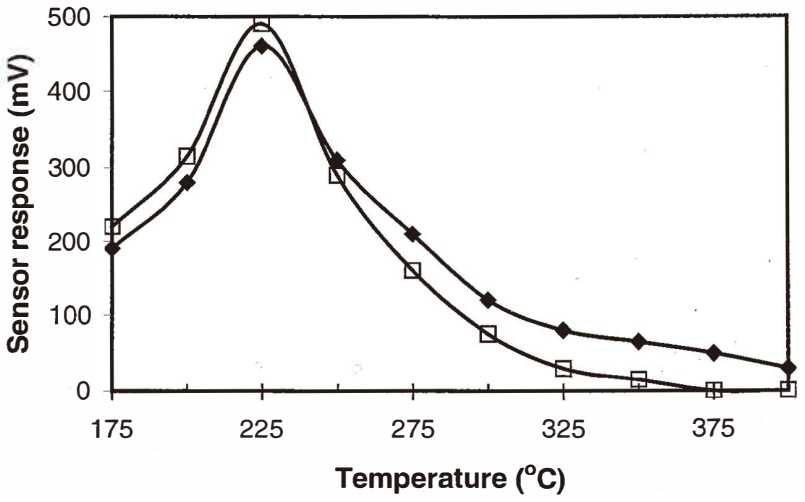

Fig. 5. Response vs temperature curves of two sensors with Pt gates for 250 ppm NH_3 in 20% O_2/N_2, with the maximum response at approximately 225°C. The sensors are mounted in pairs on ceramic rods and the current flowing through these sensors is 0.5 mA.

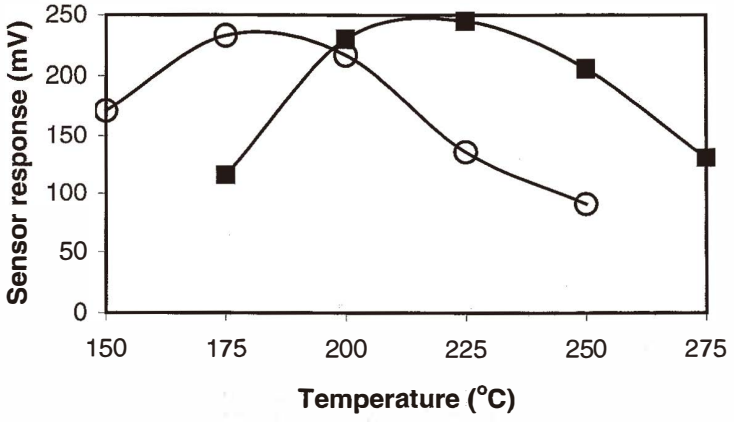

Fig. 6. Response vs temperature curves of a sensor with a 50 nm Ir gate to 250 ppm NH_3 in 20% O_2/N_2 after different pre-treatments. In the first curve (○), the device was annealed at 350°C in 5% H_2 pulses in 2% O_2 in N_2 for 30 min, and in the second curve (■), the device was annealed at 500°C in 5% H_2 pulses in 2% O_2/N_2 for 30 min. The response maximum is shifted by 50°C after annealing at a higher temperature.

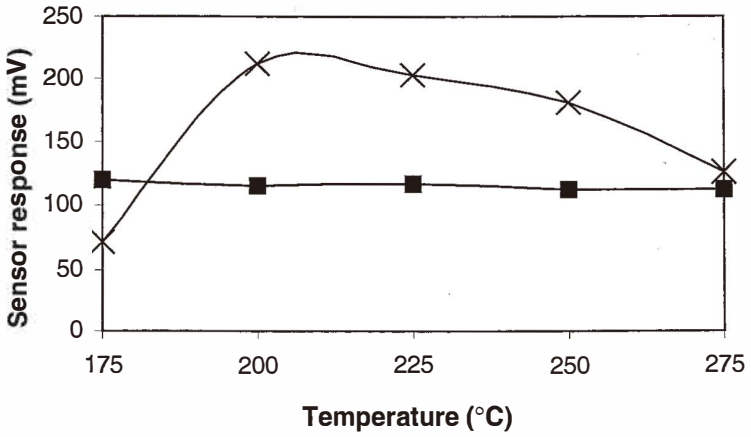

Fig. 7. Response vs temperature curves of a sensor with a 50 nm Ir gate to 250 ppm NH_3 in 20% O_2/N_2 after different pre-treatments. The first curve (■) is measured after exposure for 60 min at 450°C to 30 s pulses of 5% H_2 in 2% O_2/N_2 with 60 s of 2% O_2/N_2 between each pulse. The second curve (×) is measured after further exposure for 24 h at 225°C to 250 ppm NH_3 pulses in 20% O_2/N_2. It seems like H_2 exposures flatten the curve and long NH_3 exposures restore the curve.

flat curve is the NH_3 response versus temperature after 1 h exposure to H_2 pulses at 450°C, and the curve with a clear maximum is measured after further exposure for 24 h to NH_3 pulses in synthetic air (20% O_2/N_2) at 225°C. It should be noted that at 200°C and above the sensor signal always returns to the baseline after both H_2 pulses and NH_3 pulses.

The maximum operating temperature depends on several factors, such as the total time and the temperature at which the sensors have been used. A typical sensor was operational for approximately 15 min at 550°C, while at 450°C it was operational for more than 24 h. At temperatures close to the response maximum, the device can be operated for 2–3 months.

The Pt sensors did not show the same dependence on gas exposure as the Ir sensors. The temperature at which maximum response is realized only varies between 200°C and 225°C, which is slightly less than the Ir sensors. The shape of the response-temperature curve did not vary as much as that of the Ir sensors, i.e., the effect of H_2 and NH_3 exposures on the shape of the curve is much smaller. The Pt sensors were operational for 30 min at 450°C and for approximately 6 h at 400°C, but lasted for 2–3 months at approximately 250°C.

3.4 *Measurement in synthetic diesel exhausts*

During the tests with synthetic diesel exhausts, the temperature of the sensors was only measured with an accuracy of ±25°C due to experimental difficulties. The temperatures selected were approximately 200, 250, 300 and 350°C. We would like to point out that

twin sensors, see §2.2, show very similar results, while sensors mounted on different rods differ somewhat more. This is probably due to the difficulty to control the temperature.

3.4.1 *Pt sensors*

The sensors with Pt as gate metal showed the highest selectivity for NH_3 at about 250°C, which is consistent with the experimental results in § 3.3. The influences of other gases are small except for O_2, which also shows an interaction effect with NH_3. Figure 8 shows all main effects and some interaction effects of one Pt sensor at 250°C. As the temperature decreases, the main effect for NO_x becomes larger and no other significant change occurs. At higher temperatures, 300–350°C, the main effect for NH_3 decreased and the main effects for HC (C_3H_6, propen) and O_2 became larger.

3.4.2 *Ir sensors*

Sensors with Ir as gate metal show the largest main effect for NH_3 at approximately 200°C but also significant effects for O_2 and NO_x (Fig. 9). At approximately 250°C NH_3 has a slightly smaller effect, the NO_x effect is lowered markedly and a small HC effect is noticeable. Also a small significant interaction effect is observed for H_2O and NH_3. At temperatures of approximately 300°C and 350°C, the sensors show no consistent responses. Small significant main effects were observed for NH_3, O_2 and NO_x equal in size and with large standard deviations.

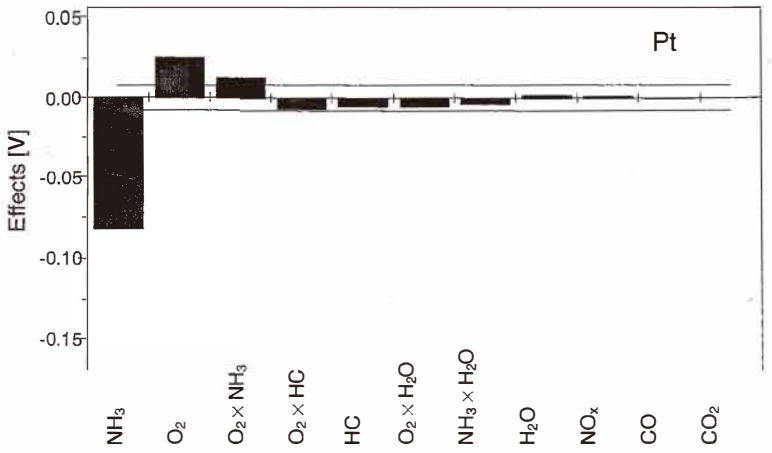

Fig. 8. Main effects and interaction effects for a 30 nm Pt sensor at 250 ±25°C. Some non-significant interaction effects were removed. Horizontal lines represent the ±95% confidence level. Only NH_3, O_2 and $NH_3 \times O_2$ show significant main effects. Propene, C_3H_6, is used as the hydrocarbon, HC.

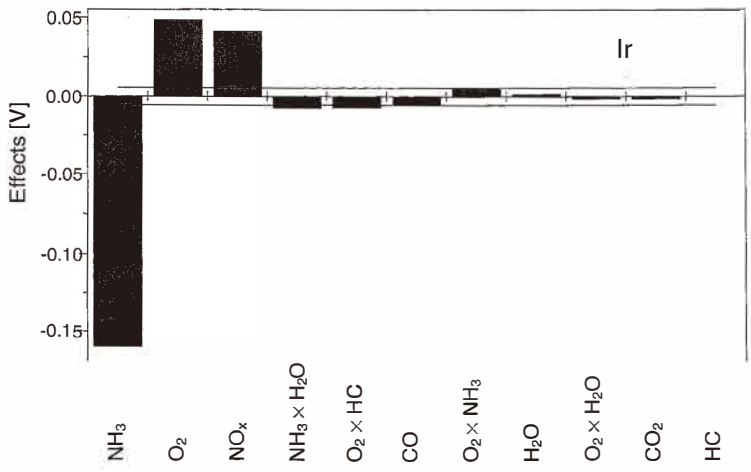

Fig. 9. Main effects and interaction effects for a 30 nm Ir sensor at 200 ±25°C. Some non-significant interaction effects have been removed. The horizontal lines represent the ±95% confidence level. NH_3, O_2, and NO_x show significant effects. Propene, C_3H_6, is used as the hydrocarbon, HC.

3.5 *AES and SEM*

Since our sensors have limited lifetimes, we wanted to investigate why they failed. Both Ir sensors and Pt sensors failed in a similar way: the resistance in the diode increased in a stepwise manner towards infinite resistance. The reason behind this behavior was investigated by AES, and SEM. We had two hypothesis: the metal film formed isolated islands on the oxide or the metal film formed a metal oxide. Both could explain the increase in resistance in the diode when it stopped operating.

Investigations with SEM revealed the formation of isolated islands. The islands were more evident on Pt sensors than on Ir sensors. The location of the islands varied between the sensors. Some sensors had isolated islands between the contact pad and the active area and other sensors had isolated islands over the entire active area. Further investigation on Pt and Ir sensors with AES sputter profiles revealed no oxide in the film. These results indicate that the formation of isolated islands is the main reason why the sensors stopped working.

4. Discussion

MISiC sensors with thin (30 or 50 nm) Pt or Ir gates have surprisingly good selectivity for NH_3 in synthetic diesel exhausts at the temperature interval of 200°C to 250°C. The ideal temperature varies slightly with the type of catalytic metal and the history of the sample.

The temperature for the optimum NH_3 response is in good agreement with that obtained from Si-based MOS capacitors with thin (6 nm) Pt and Ir gates.[2] Capacitor devices can be operated at higher temperatures than transistor devices based on Si. However, the long-term stability of Si-based capacitor devices operated at about 250°C is very poor. The SiC-based Schottky diode devices, both with Pt and Ir gates, could be operated even at, *e.g.*, 300°C, for more than two months in the laboratory.

SiC is a very inert material. It requires, for example, a higher temperature to form an oxide on SiC than on Si.[13] This makes SiC material suitable for use as a sensor material for applications in exhaust and flue gases. Silicon carbide as a semi-conducting material considerably extends the temperature range for the investigation of catalytic reactions on metal surfaces by field-effect devices, as presently performed by silicon-based devices.[4] Short periods of exposure to high temperatures may also be used to restore the gas response when it has been affected by, *e.g.*, long H_2 exposure, as shown in Fig.7. However, here it was a long exposure to NH_3 pulses at a fairly low temperature that restored the NH_3 response. The mechanism behind this behavior is presently unknown.

The reason why the ideal operation temperature is about 225–250°C for Pt and Ir gate sensors has yet to be determined. Adsorbed NH_3 molecules on the metal and/or the oxide are polar species that may be detected by these sensors. However, this does not explain the decrease of the response at lower temperatures. NH_3 is known to react with oxygen on a catalytic metal surface.[14] Competing reactions could thus be the reason for the temperature dependence. The reactions that dominate at high temperatures reduce the amount of detectable species, while the reactions at low temperatures increase the amount of the species. Even the same reaction could give rise to temperature dependence. The reaction may have a certain activation energy and the detected species may simply remain for too short a time on the surface at high temperatures. Recent results indicate that Ir does not have to be porous to be NH_3 sensitive.[5] One interpretation of this is that in the case of Ir, the detected species are actually derived from hydrogen atoms. This is puzzling in relation to the results in Fig. 7 where exposure to H_2 had consequences other than those due to exposure to NH_3.

Even though both Pt and Ir sensors have almost the same response versus temperature characteristics, they show differences in some aspects. The effects, as determined from measurements performed according to our experimental design (Figs. 8 and 9), for gases other than NH_3 are different in the temperature range of 200°C to 250°C, and the response for NH_3 on Ir is more history-dependent than that of NH_3 on Pt.

Sensitivity to gases other than NH_3 is inevitable for the chemical sensors described here. The effect of HC (C_3H_6, propen) increases with increasing temperature.[15] At high temperatures, the HC will dissociate on the metal surface,[16] and hydrogen atoms will diffuse through the metal to form a polarized layer that is the origin of the response.[17]

The main effect for O_2 also increases with increasing temperature. The effect of oxygen is probably indirect. Oxygen atoms will react with hydrogen atoms to form water at the catalytic surface and lower the effect of hydrogen-containing molecules, and this reaction will occur more rapidly with increasing temperatures.[18] Similarly, oxygen oxidizes carbon to CO and CO_2 and NO to NO_2. A small response to nominally pure O_2 of MISiC sensors was also noted in Ref.(19). In most cases, however, low levels of background

hydrogen may influence the result.

The main effect for NO_x can only be seen at low temperatures, i.e., the effect decreases with increasing temperature. NO may react with one oxygen atom and desorb as an NO_2 molecule. If this reaction involves polarized intermediates and is sufficiently slow, it might be visible in the sensor response. NO_x may also decompose to N and O atoms but the effect from the oxygen atoms will hardly be visible in an atmosphere of 10% oxygen (typical diesel exhaust).

AES and SEM studies indicate that the formation of isolated islands is the main reason why the sensors stop working. Since the sensors fail at high temperatures and apparently form isolated islands, this indicates that the melting point of the metal influences the maximum operating temperature. Due to the basic physical properties, the islands were formed at the thinnest part of the film, which could be at different locations on different sensors. Sensors with islands between the pad and the active area usually fail at a lower temperature than sensors with metal islands over the entire active area. This behavior is probably due to sensor preparation problems; the film is not uniform over the active surface area of the sensor. The film is often very thin at the border of the bonding pad and this causes failure at low temperatures. AES profiles were also taken for failed devices since it is known from the literature that Ir forms IrO_2 readily at 327°C[20] while Pt may form an oxide above 500°C.[20] However, the AES profiles showed oxygen only at the surface of the failed sensors. This is encouraging since by improving processing details of the MISiC device, the upper limit of the operating temperature and lifetime may be increased.

5. Conclusions

Schottky diodes with a catalytic gate metal (Pt or Ir) used as NH_3 sensors show good selectivity for NH_3 in synthetic diesel exhausts. The sensitivity of the sensors to several compounds in the exhausts shows a temperature dependence, and it is suggested that an array of different sensors at different temperatures combined with a linear lambda sensor, could be used for monitoring the NH_3 level in a diesel exhaust system. Further experiments and the use of artificial neural networks (ANN) may enable quantitative measurements of specific gases in diesel exhausts.

Sensors with Ir or Pt gates have NH_3 response maxima at 225–250°C but they show differences with respect to other properties:

- Ir shows better stability at temperatures above 400°C.
- Both Ir and Pt show significant main effects for NH_3, O_2 and NO_x at 200°C in synthetic diesel exhausts. Ir has larger main effects for O_2 and NO_x than Pt.
- At 250°C, Pt shows almost no effect for NO_x while Ir does. Pt also has a significant interaction effect for NH_3 and O_2 at this temperature.
- The response to NH_3 of Ir is strongly history dependent. Previous annealing and gas exposures affect the NH_3 response in various ways. Pt shows a much smaller history dependence.

Acknowledgements

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