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SiC-based MIS gas sensor for high water vapor environments

Olga Casals^{a,*}, Thomas Becker^b, Philippe Godignon^c, Albert Romano-Rodriguez^a

^a *Universitat de Barcelona (UB), MIND-IN²UB-Department of Electronics, c/Martí i Franquès 1, 08028 Barcelona, Spain*

^b *EADS Innovation Works, Nesspriel 1, 21129 Hamburg, Germany*

^c *Institut de Microelectrònica de Barcelona, IMB-CNM-CSIC, Campus UAB, E-08193 Bellaterra, Spain*

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ABSTRACT

In this work we will prove that SiC-based MIS capacitors can work in environments with extremely high concentrations of water vapor and still be sensitive to hydrogen, CO and hydrocarbons, making these devices suitable for monitoring the exhaust gases of hydrogen or hydrocarbons based fuel cells. Under the harshest conditions (45% of water vapor by volume ratio to nitrogen), Pt/TaO_x/SiO₂/SiC MIS capacitors are able to detect the presence of 1 ppm of hydrogen, 2 ppm of CO, 100 ppm of ethane or 20 ppm of ethene, concentrations that are far below the legal permissible exposure limits.

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1. Introduction

Hydrogen- or hydrocarbon-based fuel cells are a subject of intense research due to the feasibility to generate electrical energy from the reaction of these gases with oxygen. As a result of this combustion reaction, the exhaust gases of the cell have a high concentration of water vapor, between 30% and 50% by volume ratio of water vapor to gas mixture, and a high concentration of carbon dioxide (CO₂), around 20%. However, if the combustion of the fuel is incomplete the exhausts could also contain rests of the fuel or byproducts of the reaction, such as smaller hydrocarbons, carbon monoxide (CO) and nitrogen oxide (NO_x) if nitrogen is also present. So, in order to monitor the proper operation of these devices gas sensors able to work in this harsh environment and sensitive to the gases involved in the process are required.

The control of the emission to the atmosphere of hazardous gases has substantially stimulated the research and development in the field of gas sensors in the last decades. Different types of solid-state gas sensors have been developed to monitor these

gases, among them metal–insulator–semiconductor (MIS) field effect devices have the advantage of being the most compatible with the most widespread electronic technology, that of the silicon.

A MIS device can be made sensitive to specific gases using a layer of a catalytic, such as palladium (Pd) or platinum (Pt), as gate electrode. The first gas sensor based on MIS technology is attributed to Lundström et al. [1], who in 1975 reported a hydrogen-sensitive Pd-MOS transistor fabricated on conventional silicon (Si) substrate. Later, the introduction of wide band-gap semiconductors, such as SiC, group-III nitrides (AlN, GaN and AlGaN) and diamond, made possible the increase of the operating temperature of the device above that of Si (limited to below 250 °C) [2–5]. The higher operating temperature allows the devices to work nearer the source of the hazardous gases, which are usually produced in high temperature conditions. Besides, an increase of the MIS devices' response to hydrogen and an improvement in their response and recovery times [6] are obtained with increasing the operating temperature. Among the wide band-gap semiconductors, SiC stands out because of its ability to grow thermal oxides of a quality comparable to that grown on Si in terms of breakdown electric field [7]. However, although the intense research carried out during the last decades, the reduction of interface defect density is nowadays an open field of research [8–13]. Nevertheless, its high chemical inertness, its physical resistance and its high thermal conductivity make of SiC a semiconductor suitable for harsh environmental applications.

* Corresponding author at: Universitat de Barcelona (UB), MIND-IN²UB-Department of Electronics, c/Martí i Franquès 1, 08028 Barcelona, Spain.
Tel.: +34 934034804; fax: +34 934021148.

E-mail address: ocasals@el.ub.es (O. Casals).

According to most accepted model for the sensing mechanism of MIS hydrogen sensors the changes in the electrical characteristics of the devices are due to the formation of H-induced dipole layer at the metal–oxide interface [1,14–18]. The hydrogen gas molecules are dissociatively adsorbed at the surface of the catalytic gate and then the atomic hydrogen diffuses quickly toward the metal–oxide interface through the dense metallic layer. The formation of the H-induced dipole layer results in a measurable displacement of the capacitance versus gate voltage curve toward lower gate voltages that is referred as the response to the hydrogen of the sensors devices. This model is supported by the poor or null response shown by MIS structures with a dense metallic gate for gases other than hydrogen or hydrogen-containing species. Nevertheless, the use of a porous and/or discontinuous catalytic gate has widened the range of gases to which these devices respond [19]. Thus, non-hydrogen-containing species, like CO or NO₂, can be detected by MIS devices with a porous and/or discontinuous gate, which indicates the direct interaction of the layer/s under the catalytic layer. Besides, better responses to hydrocarbons have been reported [19,20]. The improvement of the sensitivity of the MIS devices with the use of porous-and/or-discontinuous gate indicates that several or different sensing mechanism are implicated in the devices' response to hydrogen, hydrocarbons or CO, which are for the time being no well-understood and require of more research.

In this work we will demonstrate, for the first time to our knowledge, the ability of Pt/TaO_x/SiO₂/SiC MIS capacitors to detect hydrogen, CO and saturated and unsaturated hydrocarbons in atmospheres with extremely high concentrations of water vapor (up to 45% by volume ratio of water vapor to nitrogen) and high temperatures, and thus, check the viability of these devices to monitor the exhaust gases of hydrogen or hydrocarbon-based fuel cells.

2. Material and methods

MIS capacitors were fabricated on n-type (0001) Si-face 4H-SiC substrates purchased from CREE Inc. The wafers had a 10- μm epitaxial SiC layer with a doping level of 10^{16}cm^{-3} grown on a heavily doped SiC substrate with a resistivity of $0.021\ \Omega\ \text{cm}$. A 20-nm-thick silicon oxide (SiO₂) layer has been grown by dry oxidation of the SiC wafers. A post-oxidation annealing in 50% N₂O/Ar atmosphere has been performed in order to improve the electrical performance of the SiO₂ layer at high temperatures [21–23]. The catalytic gate electrode was formed by sputtering a 100-nm-thick layer of metallic platinum through a dot shadow mask with a diameter of 860 μm . A 20-nm-thick buffer layer of TaSi_x was deposited by sputtering on the SiO₂ layer prior the Pt deposition in order to increase the adhesion stability of this layer at high temperatures [6]. The back contact of the MIS structures was formed first by sputtering a nickel layer on the 4H-SiC substrate and then a platinum layer. The Ni layer forms an ohmic contact [24,25] with the heavily doped substrate and the Pt coating protected the Ni layer from the hazardous environment. A detailed description of this fabrication process has been published elsewhere [26]. After their fabrication the devices were annealing for some hours in an alternating reducing and oxidizing atmospheres at temperatures above 600 °C in order to obtain a suitable porosity of the gate layer [27] and stabilize the electrical behavior of the devices at high temperatures. This post-fabrication treatment not only transforms physically the metallic gate layer but yields to an oxidation of the deposited TaSi_x layer to Ta₂O₅ and several tantalum sub-oxides and a small quantity of SiO_x [26] that obviously result in different electrical characteristics with respect those of the as-fabricated devices. The final layer structure of the MIS capacitors is schematized in Fig. 1.

The MIS capacitor response to the presence of the different gases has been measured as the change of the gate

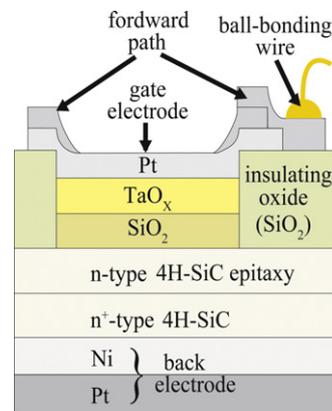


Fig. 1. Schematic diagram of the layers that comprise the MIS capacitors.

voltage ($\Delta V = V_G(\text{gas}) - V_G$) at constant capacitance. By means a proportional-integral-derivative (PID) software based on Labview, the gate voltage provided by a digital voltage source is changed as needed to maintain constant the capacitance value measured by a capacitance meter at 1 MHz. The MIS devices were heated to the operating temperature by means a platinum resistance (heater) attached under them. This Pt-heater is protected from the hazardous gases and electrically isolated from back contact by means of an alumina layer. All the system was mounted in a support specially designed to resist hazardous atmospheres and high temperatures.

All the gases mixtures were introduced in a gas test chamber, whose volume is 4 ml, at atmospheric pressure with a flux of 50 ml/min. The gas concentrations are expressed in volume ratio of the gas to the total gas mixture: parts per cent (%) for water vapor and part per million (ppm) for the rest of gases. Water vapor concentrations ranging from 3% (equivalent to 80% relative humidity at room temperature and 1 atm) to 45% were obtained by mixing ultrapure water with the gas flux in a Bronkhorst controlled evaporator and mixer (CEM) at 200 °C and at atmospheric pressure. The condensation of the water vapor was avoided maintaining the gas test chamber and all the pipes at 200 °C. A diagram of the experimental set-up is shown in Fig. 2.

3. Results and discussion

One of the characteristic of gas sensors based in MIS devices is their selectivity to the different gases according to the catalytic metal used as gate electrode and the operating temperature of the device. MIS capacitors with a Pt-layer show a positive response ($V_G(\text{gas}) - V_G > 0$) to oxidizing gases such as oxygen and a negative one ($V_G(\text{gas}) - V_G < 0$) to reducing gases such as hydrogen and hydrocarbons. MIS capacitors' response to hydrogen, hydrocarbons and CO has been studied for operating temperatures ranging from 200 to 350 °C in dry nitrogen atmospheres. The sensors respond to hydrogen and to CO at all the range of temperatures studied but the maximum sensor's response to hydrogen was obtained at around 260 °C and in the case of CO at 240 °C. In contrast, ethane and ethene are hardly detected below 290 °C and the maximum response was obtained at 300 °C and 320 °C, respectively. At the same time, MIS capacitors have not shown any significant response to the presence of high concentrations of CO₂ in all the range of temperatures studied.

Measurements of the admittance of the MIS capacitors in high water vapor environments have demonstrated their viability as electrical devices under the harsh environment of the present application. Fig. 3 shows the admittance versus voltage curves of a MIS capacitor working in dry nitrogen atmosphere and in presence of a 45% of water vapor. As it is seen, the presence of such high water

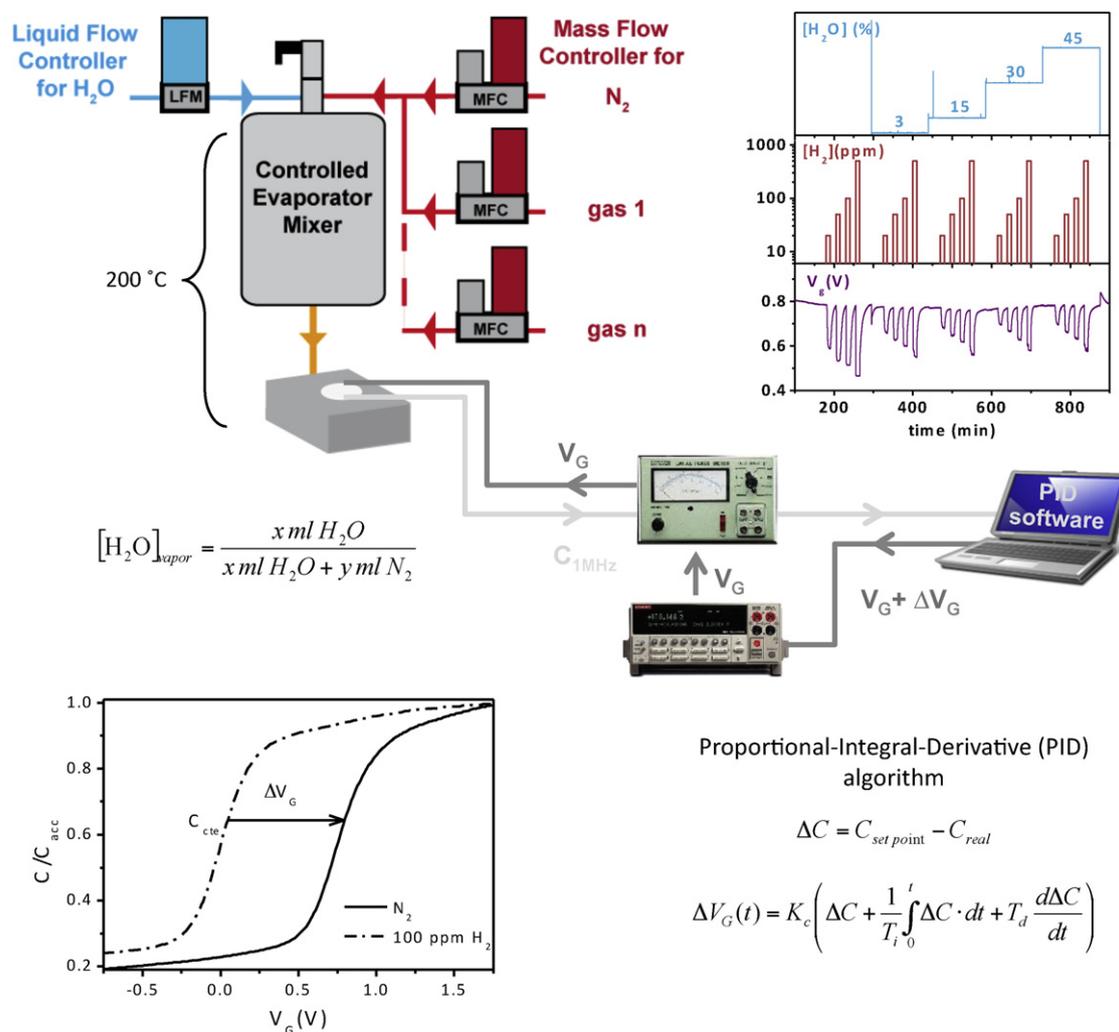


Fig. 2. Diagram of the experimental set-up used to characterize the MIS capacitor's response to gases in different water vapor containing atmospheres: a schematic diagram of the gas mixer system is shown in the upper left-hand corner of the figure; the equipment and PID algorithm needed for the gate voltage measurements at constant capacitance are included in the lower right-hand corner. The $C - V_G$ curves obtained in presence and in absence of 100 ppm of hydrogen are used to illustrate the concept of response to gases of a MIS capacitor (lower right-hand corner). A typical dynamic measurement of a MIS capacitor's response to gases is also shown in the higher left-hand corner of the figure: the upper panel shows the stepwise increasing water vapor concentrations; the middle panel shows the various pulses of increasing gas (H_2) concentration; finally, the sensor's gate voltage signal is shown in the lower panel.

vapor concentration hardly produces any electrical change in both $C(V)$ and $G(V)$ curves, that is, the MIS capacitors are insensitive to water vapor. Besides, no degradation of the electrical performance of the devices has been observed after several days of working under these high water vapor concentrations atmospheres.

Fig. 4 shows the dynamic response of the MIS capacitors to pulses of hydrogen from 20 to 500 ppm in different water vapor containing atmospheres at 300°C. Pulses with similar shape are obtained for all the water vapor concentrations, however, a decrease in the sensor's response is observed as the amount of water vapor in the gas mixture is increased. Nevertheless, the devices are able to detect down to 1 ppm of hydrogen even under the highest water vapor concentrations.

The sensor's response to hydrogen versus the gas concentration, $\Delta V[H_2]$, is plotted in Fig. 5a for the presence of the different concentrations of water vapor. Assuming that the response of the sensor is proportional to the fractional coverage of the hydrogen adsorption sites, θ_{H_2} , independently of whether the sites are localized at the metal-insulator interface or at the insulator surface (Eq. (1)), the behavior of the sensor's response to hydrogen

can be modeled according to the theoretical adsorption isotherms for gases on surfaces.

$$\Delta V([H_2]) \propto \theta_{H_2} \quad (1)$$

The simplest of these adsorption isotherms, the Langmuir's isotherm (Eq. (2)), assumes that the coverage cannot proceed beyond the monolayer coverage, that all sites are equivalent and that the surface is uniform, and finally, that the ability of a molecule to adsorb at a given site is independent of the occupation of neighboring sites [28]. According with these premises, the rate of adsorption is proportional to the gas pressure and to the probability that the n fragments of the gas molecule will find adsorption sites and the rate of desorption is proportional to the number of molecules adsorbed and to the probability of encounters of the adsorbed molecule fragments. Since at steady state the net rate of adsorption must be zero, we can solve for θ and obtain the

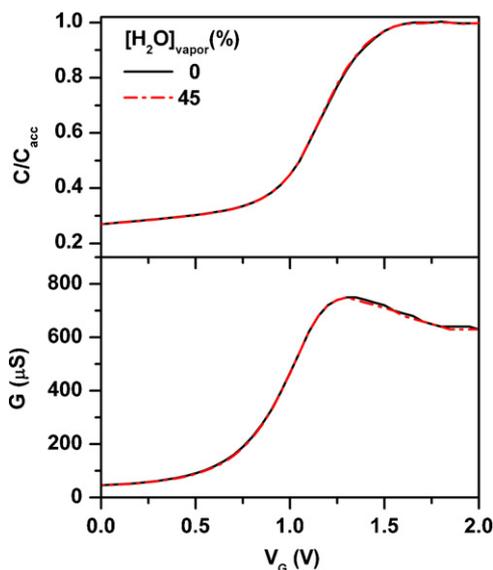


Fig. 3. Admittance versus gate voltage curves ($C-V_G$ and $G-V_G$) of a Pt/TaO_x/SiO₂/SiC MIS capacitor operating at 250 °C in dry nitrogen and in the presence of 45% of water vapor.

Langmuir's isotherm:

$$\left. \begin{aligned} \frac{d\theta}{dt} &= k_a P_{\text{gas}} [N(1-\theta)]^n \\ \frac{d\theta}{dt} &= -k_d \{N-\theta\}^n \end{aligned} \right\} \xrightarrow{\text{yields}} \theta = \frac{k^{1/2} P_{\text{gas}}^{1/n}}{1 + k^{1/n} P_{\text{gas}}^{1/n}} \quad \text{with } k = \frac{k_a}{k_d} \quad (2)$$

where k is the quotient between the temperature-dependent constants of adsorption, k_a , and desorption, k_b , and n is number of fragments into which a gas molecule is chemisorbed. Considering Eq. (1) and that the total pressure is 1 atm, Eq. (2) can be written as follows:

$$\Delta V([\text{H}_2]) = \Delta V_{\text{max}} \theta_{\text{H}_2} = \Delta V_{\text{max}} \frac{k^{1/n} [\text{H}_2]^{1/n}}{1 + K^{1/n} [\text{H}_2]^{1/n}} \quad (3)$$

where ΔV_{max} corresponds to the steady state value of the $\Delta V([\text{H}_2])$ curve, that is, the response of the MIS capacitor when all accessible sites are occupied. For all the water vapor atmospheres the experimental data fit quite well to Eq. (3) with an exponent n very close

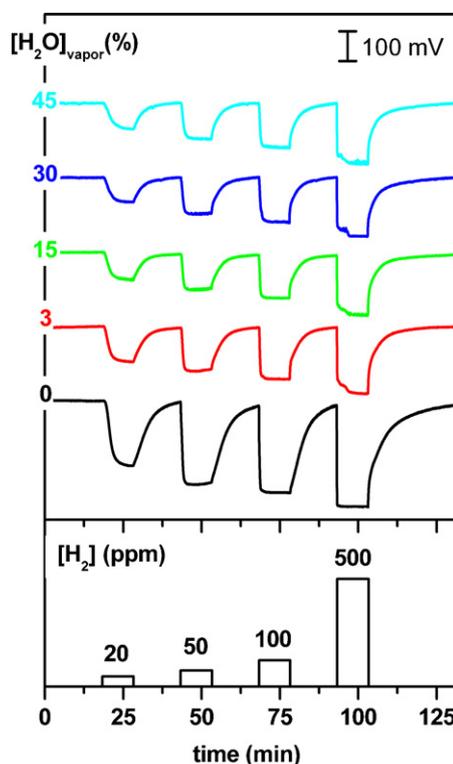


Fig. 4. Dynamic response of the Pt/TaO_x/SiO₂/SiC capacitors to pulses of hydrogen in different water vapor containing atmospheres at 300 °C.

to 2. The other parameters obtained for the nonlinear least squares fitting to Eq. (3) are shown in Table 1 and the corresponding fitted curves are included in Fig. 5a. According with Langmuir's isotherm an exponent n equal to 2 indicates that the adsorption of hydrogen is dissociative (i), which is consistent with studies of the hydrogen adsorption on Pt surfaces in dry atmospheres reported in the literature [29–34].



It is noteworthy that, in principle, for devices with a porous gate layer more than one adsorption mechanism for hydrogen molecules can occur since the insulator or the metal-insulator

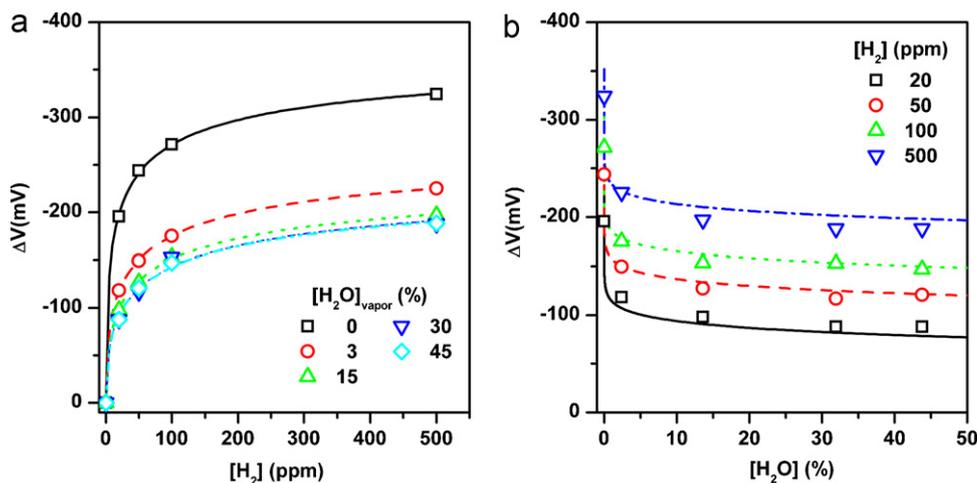


Fig. 5. (a) Symbols stand for the experimental response of the Pt/TaO_x/SiO₂/SiC capacitors to hydrogen as a function of the gas concentration in different water vapor containing atmospheres. Lines stand for the fitted curves according to the Langmuir's isotherm (Eq. (3)). (b) Symbols stand for the sensor's response to hydrogen as a function of the water vapor concentration present in the atmosphere for four different hydrogen concentrations. Lines stand for the fitted curves according to Eq. (4).

Table 1

Parameters obtained for the nonlinear least squares fitting to Eq. (3) of the sensor's response versus hydrogen concentration for different water vapour environments.

$[\text{H}_2\text{O}]_{\text{vapor}} (\%)$	$\Delta V_{\text{max}} (\text{mV})$	$K^{1/2}$	r
0	-387 ± 4	0.234 ± 0.008	0.99990
3	-292 ± 2	0.150 ± 0.002	0.99996
15	-264 ± 4	0.133 ± 0.005	0.9998
30	-263 ± 19	0.120 ± 0.01	0.996
45	-260 ± 10	0.122 ± 0.01	0.999

interface itself are directly accessible. This contradicts the premises of equivalence of the adsorption sites and of uniformity of the surface of the Langmuir's isotherm. So, either these adsorption mechanisms results in the same effect as the adsorption on the metallic surface or the contribution of the hydrogen molecules adsorbed through these other mechanisms hardly contribute to the sensor's response. In the latter case the larger sensor's response to hydrogen of the devices with a porous gate could be attributed to the increase of the effective surface of the Pt layer.

The decrease of ΔV_{max} means that the total number of sites available for the hydrogen adsorption decreases with increasing water vapor concentration in the atmosphere. The sites unavailable to hydrogen adsorption might be rendered useless or might be only inaccessible for hydrogen molecules or might be occupied by $\text{H}_2\text{O}_{\text{vapor}}$ molecules that do not generate any response in the sensors. In any case, the fraction of unavailable sites for hydrogen adsorption can be introduced in the equation of the ratio of adsorption by adding a term proportional to a power m of the water vapor partial pressure, $cP_{\text{H}_2\text{O}_{\text{vapor}}}^m$. By modeling the effect of the water vapor on the sensor's response by this simple term we assume that the water vapor molecules do not interact with gaseous or adsorbed hydrogen. So, solving again for the fractional coverage, considering Eq. (1) and that the total pressure is 1 atm we obtain an equation (Eq. (3)) based on a modified Langmuir's equation that describes the behavior of the sensor's response to hydrogen as a function of the hydrogen and the water vapor concentrations.

$$\left. \begin{aligned} \frac{d\theta_{\text{H}_2}}{dt} &= k_{a,\text{H}_2} P_{\text{H}_2} \{N(1 - \theta_{\text{H}_2} - cP_{\text{H}_2\text{O}_{\text{vapor}}}^m)\}^2 \\ \frac{d\theta_{\text{H}_2}}{dt} &= -k_{d\text{H}_2} \{N\theta_{\text{H}_2}\}^2 \end{aligned} \right\} \Delta(\text{H}_2)$$

$$= \Delta V_{\text{max}} \frac{K^{1/2} [\text{H}_2]^{1/2} (1 - c[\text{H}_2\text{O}]_{\text{vapor}}^m)}{1 + k^{1/2} [\text{H}_2]^{1/2}} \quad (4)$$

The exponent n has been fixed to 2 according to the results of the previous fittings. The hydrogen concentration, $[\text{H}_2]$, is expressed in ppm and the water vapor concentration, $[\text{H}_2\text{O}]_{\text{vapor}}$, in percent. The parameters obtained for the nonlinear least squares fitting of the experimental data to the Eq. (4) are summarized in Table 2 and the fitted curves are included in Fig. 5b. As it is seen Eq. (4) explains quite well the effect of the water vapor on the sensor's response to the different hydrogen concentration. Thus, the results confirm a kind of competition of the water vapor molecules with hydrogen for the adsorption sites and the premise of no interaction between the two t of molecules.

It is noteworthy that the decrease in the hydrogen response of the sensor almost saturated for water vapor concentration above 30% and, thus in the range of interest for the present application

Table 2

Parameters obtained for the nonlinear least squares fitting to Eq. (4) of the sensor's response versus hydrogen and water vapour concentrations.

$\Delta V_{\text{max}} (\text{mV})$	$K^{1/2}$	c	m	r
-430 ± 14	0.17 ± 0.01	0.32 ± 0.02	0.10 ± 0.01	0.99

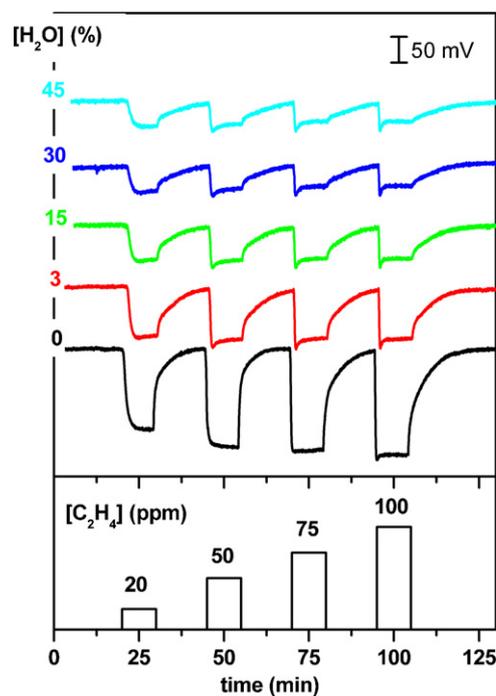


Fig. 6. Dynamic response of the Pt/TaO_x/SiO₂/SiC capacitors to pulses of ethene in different water vapor containing atmospheres at 300 °C.

we can consider the sensor's response to hydrogen independent of changes in the concentration of water vapor present in the atmosphere.

MIS capacitors have also demonstrated to be suitable sensors for detecting small hydrocarbon molecules such as ethane and ethene in high water vapor environments. An example of the dynamic sensor's response to pulses of ethene in the range from 20 to 100 ppm obtained in different water vapor environments is shown in Fig. 6. As in the case of hydrogen, a decrease in the sensor's response to pulses of the two hydrocarbons is observed with increasing the water vapor concentration. Besides, the presence of water vapor slows the recovery of the sensor's signal baseline when the hydrocarbons are removed from the test chamber. Nevertheless, the detection limits of the MIS capacitors to these gases, 100 ppm for ethane and below 20 ppm for ethene, are far below their legal permissible exposure limits, 1000 and 200 ppm time-weighted average (TWA) [35,36], respectively, even for the highest water vapor concentration tested.

The sensor's response to ethane and ethene as a function of their concentration also follows an equation based on the Langmuir's isotherm (Eq. (2)) for all the water vapor concentrations but the exponent n has been found to be around 1 for the two hydrocarbons. According with Langmuir's isotherm, an exponent $n=1$ implies a non-dissociative adsorption of the gas molecules. However, the value of this exponent can also mean that one the hydrogen atoms is leaving the molecule (ii) and diffuse toward the metal-insulator interface, giving rise to the sensor's response but that the rest of the adsorbed gas molecule, C_xH_{y-1} , does not produce any electrical change in the MIS capacitor. Nevertheless, the sensing mechanism of these two small hydrocarbons might be a combination of both adsorption mechanisms or the result of quite complicated one and, so, it is difficult to be deduced by only one type of measurement. Thus, additional studies with analytical techniques for atmospheric pressures, such as mass spectrometry [37] and/or in situ DRIFT (diffuse reflection infrared Fourier transform) spectroscopy [38,39], which have already provided very useful information to better understand the detection mechanism

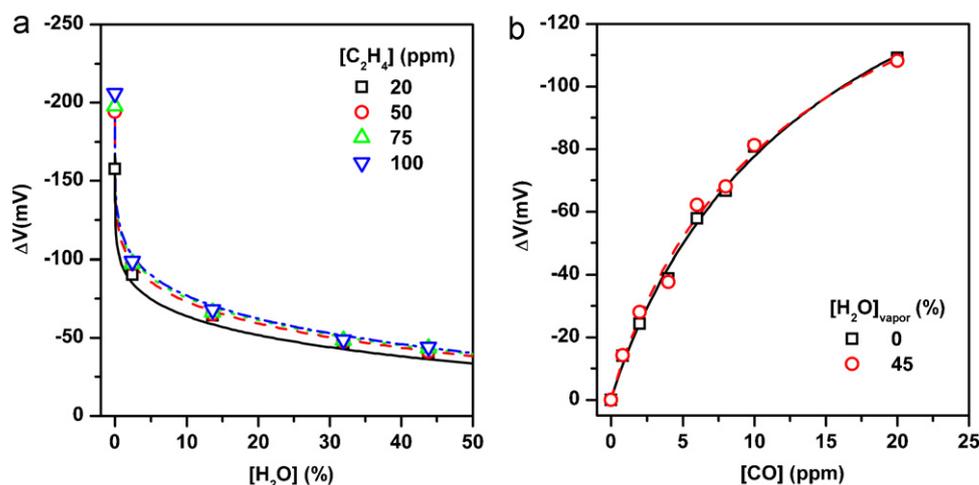


Fig. 7. Symbols stand for the response of the Pt/TaO_x/SiO₂/SiC capacitors to ethene as a function of the water vapor concentration present in the atmosphere for four different ethene concentrations. Lines stand for the fitted curves according to Eq. (5).

Table 3

Parameters obtained for the nonlinear least squares fitting to Eq. (5) of the sensor's response versus ethene and water vapour concentrations.

ΔV_{\max} (mV)	K	c	m	r
-212 ± 5	0.19 ± 0.03	0.10 ± 0.01	0.16 ± 0.01	0.996

of MIS devices to hydrogen and carbon monoxide, are needed to elucidate the sensing mechanism toward these gases.



The dependence of the sensor's response to ethene with the water vapor concentration for different concentrations of ethene from 20 to 100 ppm is shown in Fig. 7a. As in the case of hydrogen, the effect of the water vapor on the sensor's response can be modeled by an equation based on a modified Langmuir equation but with the exponent n around 1, that is

$$\Delta([C_xH_y]) = \Delta V_{\max} \frac{k[C_xH_y](1 - c[H_2O]_{\text{vapor}}^m)}{1 + k[C_xH_y]} \quad (5)$$

An example of the fitting of the experimental data to Eq. (5) is shown in Fig. 7a for the case of the sensor's response to ethene as a function of the water vapor concentration and the fitted parameters are listed in Table 3.

In order to study the effects of the water vapor in the sensor's response to non-containing-hydrogen molecules, the response to carbon monoxide has been measured. In contrast with the response to hydrogen or hydrocarbons, the response to CO has been found to be hardly dependent on the water vapor concentration. It has been found that the sensor's response to CO follows an equation based in the Langmuir's isotherm for non-dissociative adsorption (Fig. 7b). The detection limit of MIS capacitors for CO is around 2 ppm, which is far below the legal permissible exposure limits (25 ppm TWA) [35,36].

4. Conclusions

MIS capacitors have demonstrated that are able to work properly in high water vapor environments at high temperatures and, thus, are good candidates for monitoring the exhaust gases of hydrogen or hydrocarbons based fuel cells. The presence of high water vapor amounts in the environments does not produce any degradation of the electrical performance of MIS capacitors.

Besides, the devices do not respond to changes in the water vapor concentration. Under these harsh measuring conditions, the devices are sensitive to all the gases under study down to 1 ppm of hydrogen, 2 ppm of CO, 100 ppm of ethane and 20 ppm of ethene.

Although the sensor's response to hydrogen and to small hydrocarbons, such as ethane and ethene, decreases with the increasing of the water vapor concentration, the sensor respond properly to these gases even in the presence of the highest water vapor concentration tested (45% by volume ratio to nitrogen). It is noteworthy that for water concentration above 30% the sensor's response decrease is negligible, so the devices' response can be considered almost water vapor independent in the range of water vapor concentration usually found in the present application. The presence of water vapor does not affect the sensor's response to CO, which indicates that water vapor molecules only compete with hydrogen molecules or atoms for the adsorption sites.

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Biographies

O. Casals was born in Barcelona in 1973. She received her diploma in Optics and Optometry at the Polytechnic University of Catalonia in 1994 and her degree in Physics at the University of Barcelona in 2001. Currently she is student of the doctor's degree programme "Electronic Engineering and Technology". Her professional experience is set in development of new technologies on solid-state gas sensors.

Prof. Dr. Thomas Becker graduated at the university of Bremen/Germany in 1995. Subsequently he joined DaimlerChrysler Research and Technology (Munich/Germany), which became EADS Innovation Works in 2000. There he has been acting as researcher, key technology area manager for microsystems, electronics and microelectronics and as manager for sensor related topics. Currently he is focussing on energy harvesting and wireless sensing. In addition, since 2003 he is lecturer at the university of Isny/Germany. He is author of more than 60 publications, holds about 20 patents, acts as editor for scientific journals and served as chairman for international conferences.

Prof. Philippe Godignon received his PhD in electrical engineering in 1993 from INSA Lyon (France). Since 1990, he has been working in the Power Device and System group of CNM on Si and SiC IGBT/VDMOS devices design and technologies. His competences cover the power device and integrated circuit technological and electrical modeling, technological process development as well as the electro-thermal characterization. He is currently head of the systems integration department of CNM. He is co-author of more than 185 publications in international journals and conferences and of 12 patents.

Prof. Dr. Albert Romano-Rodríguez received his degree in physics at the University of Barcelona in 1986. In 1989, he joined the laboratory IMEC (Leuven, Belgium), where he worked out his PhD in physics, presented at the University of Barcelona (1991), on the application of transmission electron microscopy techniques for the development of ULSI devices. He continued in IMEC as postdoctoral fellow and returned to UB as researcher and was appointed in 1993 as Professor in electronics. His research activities are in the fields of structural, chemical and electrical characterization of electronic materials and processes employed in the electronic industries and in the development, characterization and test of chemical sensors. Prof. Romano has co-authored over 110 papers in international peer-reviewed journals and over 020 contributions to conferences.