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Thermal Modulation of SnO_2 Sensors for Alcohol Monitoring by Perspiration

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Abstract: In this paper a cyclical heating profile of Tin dioxide (SnO_2) sensor is investigated. We propose to reduce power consumption while maintaining high sensitivity and good stability during monitoring of alcohol elimination by the skin trough the perspiration. This approach consists to operate the sensor micro-hotplate in pulse mode by switching on – off. The sensitive thin layer of SnO_2 with a thickness of 50 nm was deposited by a magnetron reactive sputtering technique. Then, we studied the response of a SnO_2 sensor under ethanol exposure to enable alcohol monitoring under a number of different power conditions (continuous and cyclical heating) in order to investigate the impact of reduced system level power consumption on sensor sensitivity. We demonstrated that this operating mode allows dividing by four the power consumption but results in a slight decrease of the sensitivity of the sensor of about 25 %.

Keywords: Ethanol monitoring, Insensible perspiration, Non-invasive detection, Low power consumption, SnO_2 gas sensors.

1. Introduction

Alcohol is one of the most important factors of avoidable death across the world, and represents a high socio economic cost due to the alcohol related incidents and health concerns (Alcoholism, alcoholic cirrhosis, cancer of upper aero digestive tracts, etc…), which continue to increase. Currently, the most commonly used alcohol detection methods are the analysis of biological fluids such as blood, breath, or urine [1-3]. However, these detection techniques have a limit of efficiency by not allowing continuous monitoring or passive detection of the alcohol concentration in a non-invasive way [2, 4-6]. There is a growing need to monitor indirectly the blood alcohol level of people who have jobs at risks, or for abstention from alcohol for medical or legal reasons [7].

In this context research in the field of metal oxide chemo-resistive gas sensors (MOx Sensors) has been a growing area of interest for biomedical applications, such as the detection of Volatile Organic Compounds (VOC) released by the human body [8]. For the monitoring of alcohol vapor emitted by transdermal perspiration (Insensitive and non-visible vapor emitted by the skin continuously) after alcohol consumption, SnO_2 metal oxide sensors are used in this study. It is well known that Tin dioxide is one of the most sensitive layers which can be used for gas detection due to its high sensitivity and stability [9-13]. Thus, in the case of transdermal alcohol monitoring, the main criterion to be considered when developing a mobile/battery-operated sensor is the greatest sensitivity attainable with the minimum heater power consumption conditions which allow it to be incorporated into the power-constrained mobile component [14-19].

A MOx sensor requires two voltage inputs, the heater voltage and the sensor bias voltage [20-21]. The heater voltage allows the semiconductor to be maintained at a specific temperature in order to enable and enhance gas detection. A sensor bias voltage is applied to allow the measurement of the sensor response to gas exposure. One critical aspect to be considered is the power consumption required for the heating element, which is in the order of 70 mW or more on average. In the case of continuous monitoring, using power-constrained sensors, it is important to decrease heater consumption so that they can be incorporated in microsystems such as a wristband, patch and armband, or watch (this requires a power consumption in the order of 5 mW) [22]. One possible solution is to use cyclical heating, which means switching the heater on and off, thus minimizing the active period of energy consumption so as to save power [23].

Several works of power consumption reducing techniques for gas monitoring have been suggested. Most of this work attempts to reduce heat consumption by improving thermal isolation (to minimize the thermal losses related to heat conduction though solid parts) or reducing sensor size by using micro fabrication techniques [23-26]. One of these approaches is self-heating with potential for ultra-low power consumption but only on sub-micron devices. Different profiles of the pulsed heating exist such as the multi stage heating which is an approach that can drastically reduce heater consumption [27-30]. It is often composed of several stages of activation of heater; each stage corresponds to a specific value of the supply voltage. However its application results in sensitivity degradation. Otherwise Pulse–Width Modulation (PWM) is often used technique for heating the sensors up to working. PWM enable for changing the average heating voltage by changing period of the heating pulses [31-36].

Here in order to monitor the transdermal ethanol emission of the skin, we propose to apply cyclical heating (pulse heating). We will explore the various studies concerning the appropriate parameters for cyclical heating and we will compare the responses of the sensor in both cases, i.e. continuous heating and cyclical heating. The SnO_2 sensors were exposed to ethanol vapor with various concentrations ranging from 1ppm to 100 ppm.

2. Experimental

2.1. SnO_2 Sensors Fabrication

In this part we described the fabrication process of SnO_2 sensors. First we selected tin dioxide based sensitive layer relying on the results reported in the literature. Tin dioxide is n-type semiconductor oxide with a wide-band-gap (3.6 eV) [37-38], and a wide range of applications for various types of oxidizing or reducing gas/vapor detection. It is one of the most used sensitive layers. Our SnO_2 sensitive thin layers were deposited by RF reactive magnetron sputtering technique on transducers patented by AMU-CNRS-IM2NP laboratory [38]. These transducers are designed to have three sensors and two heaters in the same membrane.

In this study we will present only the response of the central sensor. A thickness of 50 nm of the sensitive layer was measured by Dektak-M6 profilometer. During le deposition process a mixture of Ar/O_2 (40 %/60 %) was used. Finally an annealing under dry air at 500 °C for 12 hours was done in order to improve the film oxidation the stability of the sensitive layer to enhance its sensitivity.

Indeed, when the ethanol molecules adsorption occurs to the surface of the sensitive layer (SnO_2) these molecules will react with the pre-adsorbed species on the metal oxide (essentially oxygen species). These reactions are mainly redox reactions. This will cause a variation of the depletion zone of the surface layer [39-40]. This variation contributes to the variation of the electrical resistance of SnO_2 (1).

$$C_2H_6O + 6O^- = 2CO_2 + 3H_2O + 6e^- \qquad (1)$$

The resistance of the sensor will decreases as SnO_2 is an n type semiconductor and ethanol is a reducing gas.

2.2. Sensor Tests

An innovative and automated sensor calibration system was designed and built thanks to the APPRISE (Personal Assistant of Health Prevention) project from Aix-Marseille University (Foundation A*MIDEX) in order to test and study the sensor performances. This platform generates ethanol and acetone vapor (acetone is one of the most important interference VOCs for the detection of alcohol in perspiration). The experimental set up (Fig. 1 and Fig. 2 the schematic representation) consists of three parts:

- Generation and control bench of ethanol vapor. Ethanol is generated by permeation system with different stages of dilutions via several automated mass flow controllers (Fig. 1(a)).
- An environmental chamber with temperature control with an integrated test cell made in our laboratory to characterize and calibrate the sensors (Fig. 1(b)).
- A control and data acquisition systems (Fig. 1(c)).

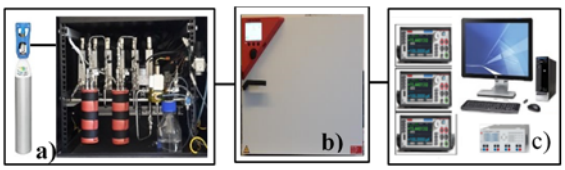


Fig. 1. Experimental set-up for ethanol microsensors characterization and calibration: (a) ethanol generation bench, (b) environmental chamber, (c) control and data acquisition system.

The gas flow rate is fixed at 250 sccm. Ethanol vapor is carried in the test chamber placed in the environmental chamber at 33°C which corresponds to the average temperature of the skin surface.

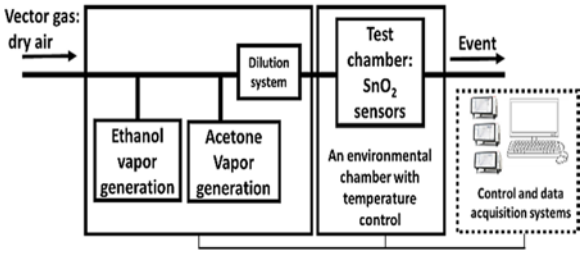


Fig. 2. Schematic representation of measuring bench.

The sensor resistances are measured directly by a Keithley 2450 source-meter. The sensor bias is fixed and the heater power is modulated to provide the operating temperature. The sensor response is defined as (2):

$$Response = \frac{R_{air}}{R_{ethanol}}, \quad (2)$$

where R_{air} is the sensor resistance measured under air and $R_{ethanol}$ is the sensor resistance measured under air and ethanol in the range of 1 to 100 ppm, every 2 minutes of exposure. When the concentration of alcohol in the blood after alcohol consumption reaches 0.5 g/l (2 standard glasses of alcoholic beverage), its equivalent in the breath to 139 ppm and lower concentrations varying between 1 and 100 ppm may be expected on the surface of the skin [1, 4, 41- 42].

2.3. Cyclical Heating

MOx sensors involve high working temperatures (220°C-300°C) in order to detect effectively gaseous species [18]. Temperature also influence the gas desorption. Fig. 3 illustrates the sensor response as a function of the heater voltage supply for 50 ppm of ethanol. 2V is the heater polarization which corresponds to the optimal heating temperature for the best response.

A frequency of cyclical heating was studied previously to determine the optimum active time of a heating element. Fig. 4 illustrates different heater activation times (6 s, 10 s, 15 s and 20 s).

Fig. 5 shows the sensor responses under different cyclical heating profiles. It demonstrates that as from a heater activation of 15 s, the response of the sensor stabilizes and the sensitive layer almost reaches the optimal temperature for the detection process.

During a period of 60s, the heater is switched off for 45 s, and on for 15 s. This is repeated throughout the measurements. The period of this cyclical heating is 60 s, its frequency is 16 mHz and the cyclical ratio is 0.25.

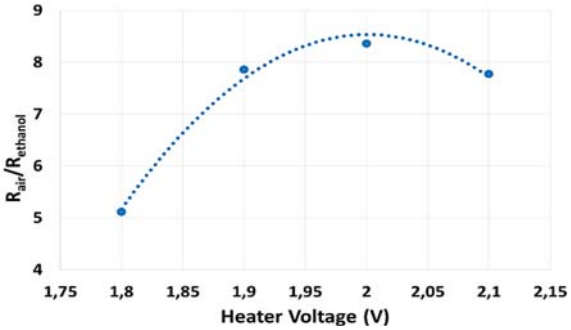


Fig. 3. SnO_2 sensor response under 50 ppm of ethanol as function of different heater voltages.

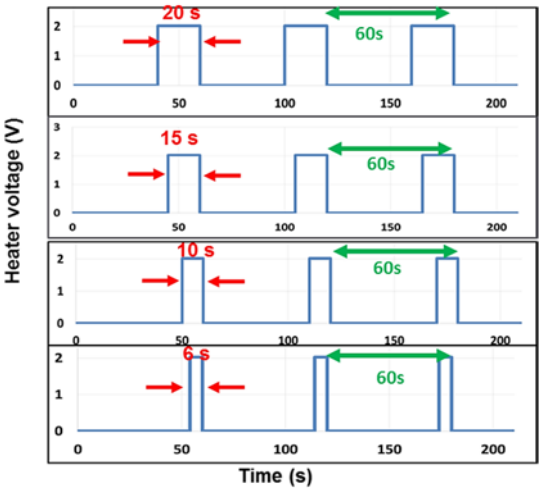


Fig. 4. Sensor responses for different cyclical heating profiles.

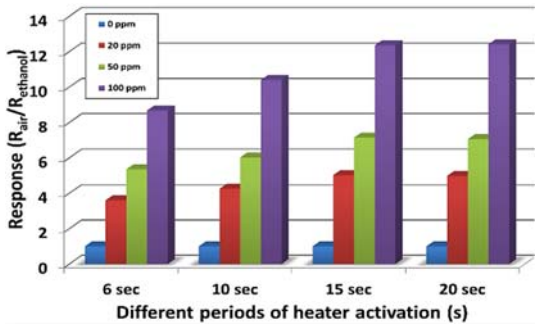


Fig. 5. Different cyclical heating profiles studied.

3. Results and Discussions

The SnO_2 sensor shows a good sensitivity to ethanol, mainly for low concentrations and a slight saturation of around 70 ppm. This slight saturation occurs when the coverage rate of the adsorption sites decreases. Fig. 6 shows the variation of sensor resistance in the case of continuous heating (53 mW).

Fig. 7 shows the variation of sensor resistance in the cyclical heating mode (13 mW). In both cases (continuous heating and cyclical heating), a decrease in resistance is observed as function of the different

concentrations of ethanol (reducing gas). The slight saturation is also observed. It can be noticed that the sensor baseline (0 ppm) is almost equivalent for a continuously heated sensor compared to the sensor in cyclical heating (heater on during 15 s during a period of 60 s).

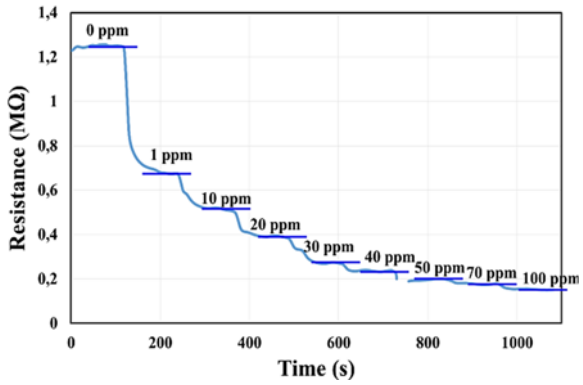


Fig. 6. SnO_2 sensor response under different ethanol concentrations: continuous heating.

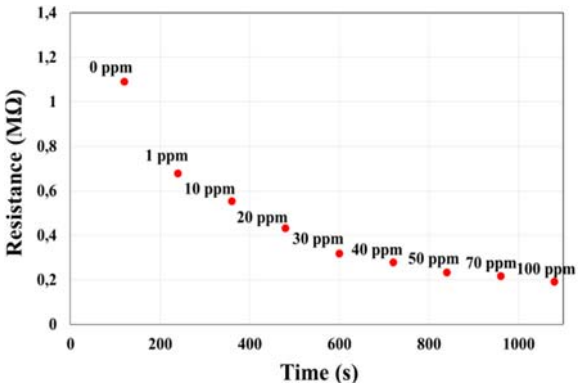


Fig. 7. SnO_2 sensor resposne under different ethanol concentrations: cyclical heating.

The power reduction (by a factor of 4) causes a slight decrease in sensor responses, as shown in Fig. 8. In fact, in the case of continuous heating the sensor operates at 280 °C and 53 mW. With cyclical heating, the heating time is decreased and thus power consumption too, without any reduction of the operating temperature (280 °C) during the detection process.

However, even if cyclical heating allows a reduction of power consuption of 75 %, it is important to note that pulse mode heating can also reduce slightly the sensor response (Fig. 8). This slight loss of sensitivity may be related to insufficient thermal energy for the complete desorption of the sensitive layer because the sensitive layer not being heated continuously. However for monitoring alcohol in perspiration cyclical heating can be interesting solution to highly reduce the power consumption of the gas sensor.

4. Conclusion

In this work, we find solutions to reduce the electrical consumption of the MOx sensors. In particular we have investigated cyclical heating apply to tin dioxide sensor. First we have studied the optimal time of operating sensor heater during 60 s of measurement. This allowed choosing 15 s of heating. Then we compared the responses of the SnO_2 sensor when the sensor operates in continuous heating and in case of cyclical heating. There was a slight loss of sensitivity around 25 %. However the response of the sensor during cyclical heating remains well exploitable. The main interest of this study is to note that we reduced the consumption of 75 % can be crucial for a monitoring application as in the case of this study.

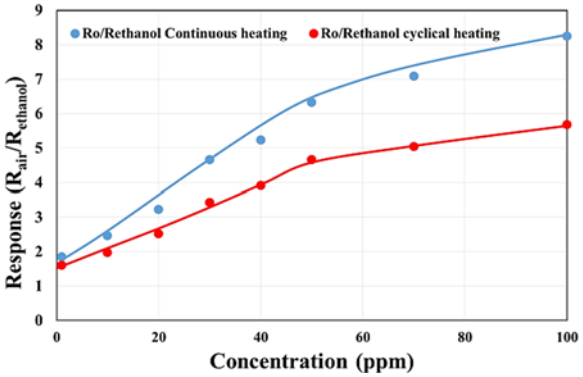


Fig. 8. Comparison of SnO_2 responses with continuous heating (53 mW) and cyclical heating (13 mW).

This method allows a reduction of power consumption in the integrated gas detection microsystems. Thanks to this heating modulation and to the short response time of the microsensors, energy consumption can be further reduced.

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ADVANCES IN SENSORS: REVIEWS 2



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Sensors and Biosensors, MEMS Technologies and its Applications

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