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Self-adapted temperature modulation in metal-oxide semiconductor gas sensors

Eugenio Martinelli*, Davide Polese, Alexandro Catini, Arnaldo D'Amico, Corrado Di Natale

Department of Electronic Engineering, University of Rome "Tor Vergata", Via del Politecnico 1, 00133 Roma, Italy

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ABSTRACT

Sensing mechanisms of gas sensors depend on temperature, and this is in particular true for metal-oxide semiconductors where the peculiar role of temperature suggested the modulation of temperature as a viable method to tune selectivity and sensitivity. This principle was widely investigated in the past, and methods to design *ad hoc* temperature behaviors have been proposed. In this paper, instead of a priori temperature profiles, a self-adaptive temperature modulation is proposed. For the scope, a closed-loop circuit connecting the sensor resistance to the sensor heater is designed. In this condition changes in sensor resistance are reflected into changes of operating temperature. Herewith, the method is implemented with an oscillatory circuit, so with a steady resistance value the signal driving the temperature modulation converges to a periodic pattern of pulses that is specific for the sensor state. Since the relationship between resistance and temperature may depend on the quality and quantity of the gas at which the sensor is exposed, the temperature modulation signal is likely dependent on the kind of gas and its concentration. As a consequence, features describing the temperature modulation signal pattern can be used as a multicomponent variable that can allow for gas identification and quantification. The hypothesis is confirmed by simulations with electronics CAD software and experiments with a commercial metal-oxide semiconductor gas sensor. Results show that optimal gas identification and concentration are simultaneously possible with a unique sensor device.

1. Introduction

Although various sensing technologies are continuously introduced, metal-oxide based gas sensors are still considered, due to their combination of robustness and facility of use, as one of the best technological options for many real applications. Along the years, different methods have been proposed to increase the performance of these sensors. Most of the sensor characteristics depend on the physical properties of the material. To this regard, it is known that the sensitivity is influenced by the physical properties of the material such as the grain size dimension, and the studies of nano-sized structures emphasized the role of grain dimensions and surface-to-volume ratio [1,2]. The lack of specificity is an intrinsic property of these sensors, it is brilliantly solved with the principle of combinatorial specificity as demonstrated by sensor arrays applications [3]. The optimal application of this method requires sensors with different selectivity, and the practiced approach to modify the selectivity is the addition of ultra-thin layers of catalytic metals [1].

Besides the physical characteristics, the sensors properties are strongly influenced by the operative conditions, and since sensing processes in these sensors are temperature dependent, changes in the operating temperature can give rise to very different sensor behaviors [4–6]. As a consequence, the application of temperature modulation profiles can give rise to sensor signals that contains qualitative and quantitative information about the gases under measure [7–11]. It is important to note that thermal modulation requires an appropriate feature extraction method to be efficiently applied [12,13]. Eventually these studies evidence that optimal thermal modulation and features extraction strongly depend on the type of sensor and the particular application at hand.

Although, the operating temperature is mainly connected to sensitivity and selectivity, several studies have demonstrated that temperature may also affect the stability. In particular, proper modulation of the temperature may lead to better reproducible devices [14–17].

In this paper, an alternative approach to temperature modulation is proposed. This method implements the concept of self-adapted temperature modulation, and it is based on the evidence that the sensitivity to the gas of the sensor resistance depends on the operating temperature, and, conversely, the sensitivity to the temperature depends on the gas. Hence, a complete assessment of the sensor state can be obtained considering simultaneously both of the variables. It is worth to remark here that dependence on gas involves both qualitative (kind of gas) and quantitative (concentration) aspects. The method is demonstrated placing the sensor with

* Corresponding author. Tel.: +39 06 72597259; fax: +39 06 2020 519.
E-mail address: martinelli@ing.uniroma2.it (E. Martinelli).

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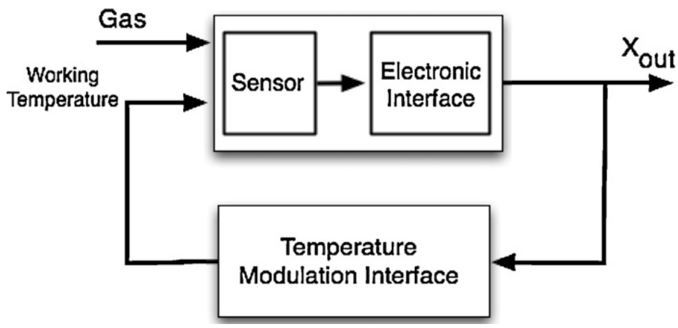

Fig. 1. Conceptual scheme of the closed-loop circuit implementing the self-adapted thermal modulation.

its sensitive and heater terminals in a closed-loop circuit. The loop is formed connecting the sensitive terminals to a circuit interface, and using the circuit output as the input signal of the sensor heater. In such a way, the thermal modulation depends on the resistance of the sensor changing according to the interaction with the gas molecules.

In the following sections, details of the proposed method are given (Section 2), and results of simulated and real experiments are provided (Sections 3 and 4, respectively). Finally, Section 5 presents some conclusions derived from this work, and provides the reader with information about future outlook.

2. Self-adaptive thermal modulation

The concept of self-adaptive thermal modulation is illustrated in Fig. 1. It consists of a closed-loop system, in which the first block comprises the sensor itself together with its circuit interface, whereas the second block is the one in-charged to process the output signal of the first block, x_{out}, in order to obtain a signal suitable to drive the sensor heater with a defined range of voltage and frequency values.

Fig. 2 shows one of the possible practical implementations of the self-adaptive thermal modulation scheme presented in Fig. 1, in which the sensor resistance is part of an astable multivibrator circuit. The choice of an oscillator circuit is mainly because of the necessity of both simplifying the circuit complexity and generating the periodic signal used to conduct the temperature modulation interface. The astable multivibrator is an elementary circuit characterized by a simple relationship between the output signal frequency and the sensor resistance.

The oscillator circuit is based on the LM555 timer (National Semiconductor), an integrated circuit mainly used as a clock generator in consumer electronics [18]. In the scheme of Fig. 2, the timer provides a square wave signal, in which the duration of the two semi-periods depends on the sensor resistance according to the following equation: for the upper semi period, on the one hand, ($V_{out} = V_{supply}^{high}$), we have

$$\int_{t_1}^{t_2} \frac{1}{(R_1 + R_{sens}(\text{gas},\ T(t)))\,C_1}\,dt = \int_{V_{threshold}^{low}}^{V_{threshold}^{high}} \frac{dV_{C_1}}{V_{supply}^{high} - V_{C_1}} \tag{1}$$

where t_1 is the time when the voltage across the capacitance C_1 is $V_{c1}(t_1) = V_{threshold}^{low}$; t_2 is the time when $V_{c1}(t_2) = V_{threshold}^{high}$ and V_{out} switches from V_{supply}^{high} to V_{supply}^{low}.

For the lower semi-period, on the other hand, ($V_{out} = V_{supply}^{low}$) is

$$\int_{t_2}^{t_3} \frac{1}{(R_{sens}(\text{gas},\ T(t)))\,C_1}\,dt = \int_{V_{threshold}^{high}}^{V_{threshold}^{low}} \frac{dV_{C_1}}{V_{supply}^{low} - V_{C_1}} \tag{2}$$

where t_2 is the time when $V_{c1}(t_2) = V_{threshold}^{high}$; t_3 is the time when $V_{c1}(t_3) = V_{threshold}^{low}$ and V_{out} switches from V_{supply}^{low} to V_{supply}^{high}.

Above, the threshold values $V_{threshold}^{low}$ and $V_{threshold}^{high}$ are fixed and defined by the LM555 [18]. The V_{supply}^{high} and V_{supply}^{low} values are the higher and lower voltage supply values, respectively, whereas R_1 and C_1 are the resistance and capacitance values shown in the circuit of Fig. 2. The $x_{out}(t)$ serves the purpose of both the oscillator output signal and the thermal modulation interface input signal. The circuit driving the thermal modulation is composed by a

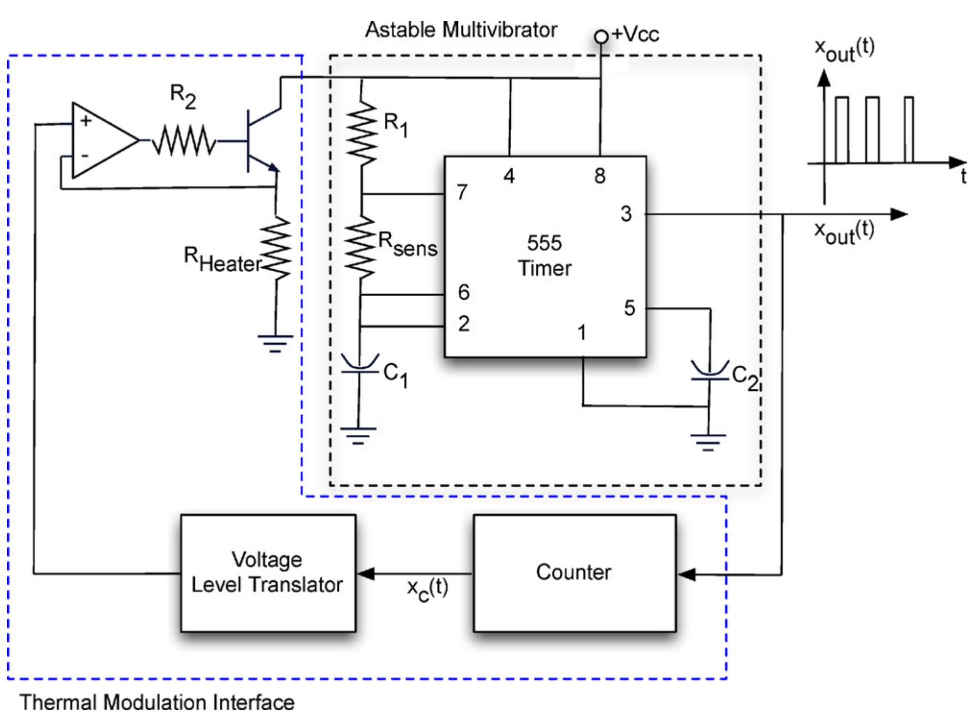

Fig. 2. Electronic circuit put into practice the scheme of Fig. 1.

digital counter, followed by a voltage level translator, and, finally, an amplifier that provides the signal of the needed power to actuate the sensor temperature.

After a certain number of input pulses, a transition between the two logical states of the digital counter output ($x_c(t)$) arises. For each one of the two logic states, a defined voltage value is applied to the heater and kept constant until another transition of $x_c(t)$ occurs.

In this way, the digital counter allows to adapt the modulation signal frequency in order to satisfy the dynamic property of the sensor thermal circuit. Actually, a fast evolution of the driven signal of the heater would not guarantee a sufficiently spanning of the working temperature that would be almost constant and proportional to the mean value of the $x_{out}(t)$ signal.

For these reasons, the length of the digital counter, k, should be optimized considering both the thermal response of the sensor and the conditions of the application. However, for the scope of this paper such an optimization is not investigated. To illustrate the properties of the proposed method, a practical instance of $k = 16$ has been chosen. With this choice, the counter changes its output value every eight input pulses. In correspondence to the two output states (0 and 1 states), the counter sets the equivalence voltage of the heater by means of the remaining part of the temperature modulation block. In this way, the temperature modulation is characterized by two semi-periods that are related to the two heater voltages and the resulting output signal pattern is composed by 16 pulses.

The temperature modulation circuit also sets the limit values of the heater voltage applied during the temperature cycles. In correspondence to these voltages, after a sufficiently long time, the sensor temperature reaches the limit temperatures (T_{min}, T_{max}).

During the measurement, the dynamical change of the sensor resistance is reflected in an unsteady temperature pattern; when the steady state is reached, a steady pattern of pulses is expected (Fig. 3a). The length of the x_{out} pattern is function of both the frequency of the output signal (and then of the sensor resistance) and the length of the counter. In particular, the larger the value of k is, the longer the pattern pulses are.

When the signal driving the heater, $x_{heater}(t)$, changes from minimum to maximum value, the temperature increases towards T_{max}, following the usual exponential behavior determined by the thermal constant of the sensor (Fig. 3b). However, before the sensor's operating temperature reaches its final value (T_{max}), $x_{heater}(t)$ switches from the highest to the lowest value and the temperature decreases towards its initial values. Then the temperature decrease also follows the exponential behavior.

Comparing Fig. 3a and b it is also possible to put in evidence that the number of pulses composing the pattern is equally divided for the two semi-periods of the thermal modulation (8 + 8) although they are characterized by different time lengths. This evidence further suggests the importance of the digital counter that changes its output only after eight input pulses independently by their time lengths. In this way, when the sensor reaches a dynamic equilibrium with the close environment the digital counter, by means of the temperature modulation interface, is able to set a periodic thermal evolution to the sensor also in presence of a modulated $x_{out}(t)$ signal.

It is worth to note that in standard thermal modulation the temperature affects the resistance according to the gas at which the sensor is exposed. In this method the terms are exchanged. Here, instead, the sensor resistance determines the temperature in a way that it depends on the sensed gas. In practice, it is expected that, at the equilibrium, the limit temperatures and the pattern of pulses are both correlated with the quality and quantity of the measured gas. Then, if the relationship between temperature and resistance depends on the gas at which the sensor is exposed, it should be possible selecting the optimal range of temperatures (T_{min} and T_{max} values) and the digital counter, to obtain different pulses patterns that can discriminate the gases.

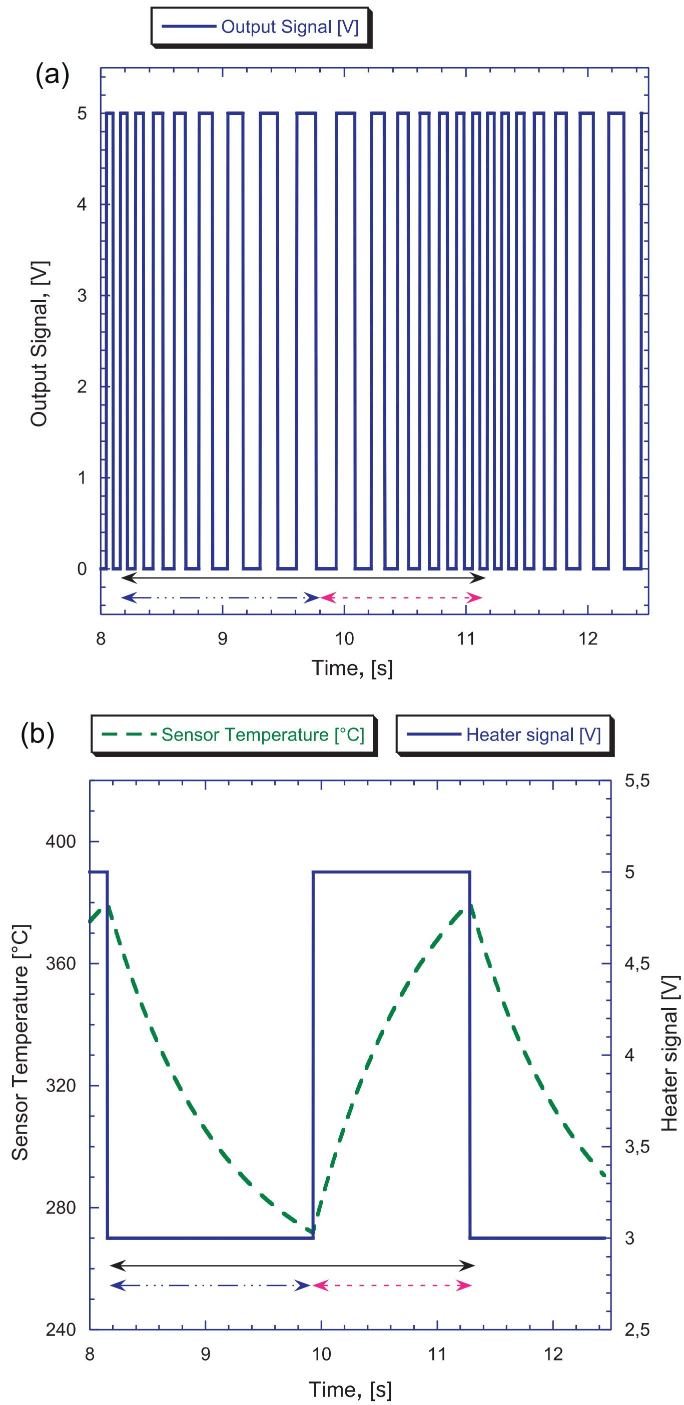

Fig. 3. (a) The signal x_{out} as a pattern of pulses. (b) The signal x_{out} is transformed by the digital counter into a slower signal x_{heater} that applied to the temperature modulation interface forces the sensor temperature to oscillate between two limits. In case of a simple thermal circuit with one thermal constant the temperature follows an exponential behavior. In both figures the lengths of the two semi-periods of the temperature modulation are also shown.

3. Circuit simulation

The method has been studied simulating the circuit of Fig. 2 in ORCAD, a standard CAD software for electronic circuits [19]. For this scope, the metal oxide sensor has been simulated by an *ad*

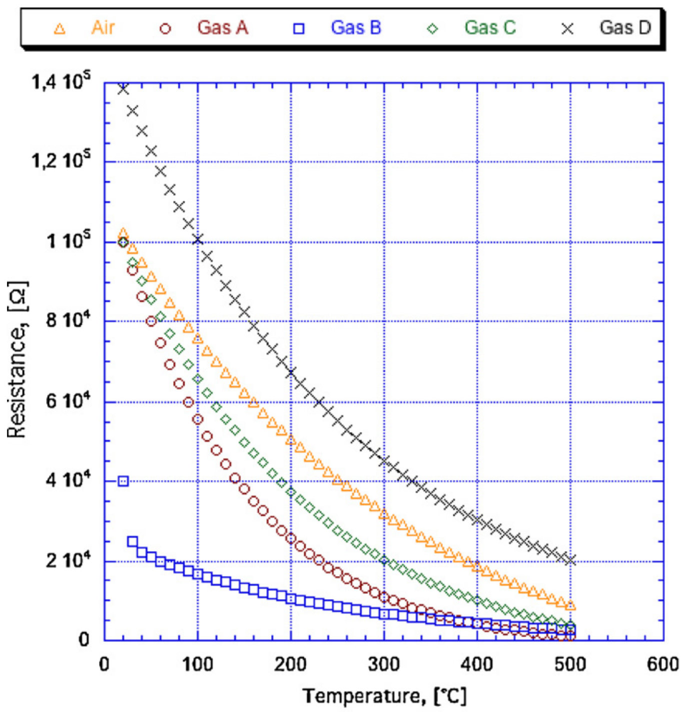

Fig. 4. Resistance vs. temperature curves used in the electrical simulation.

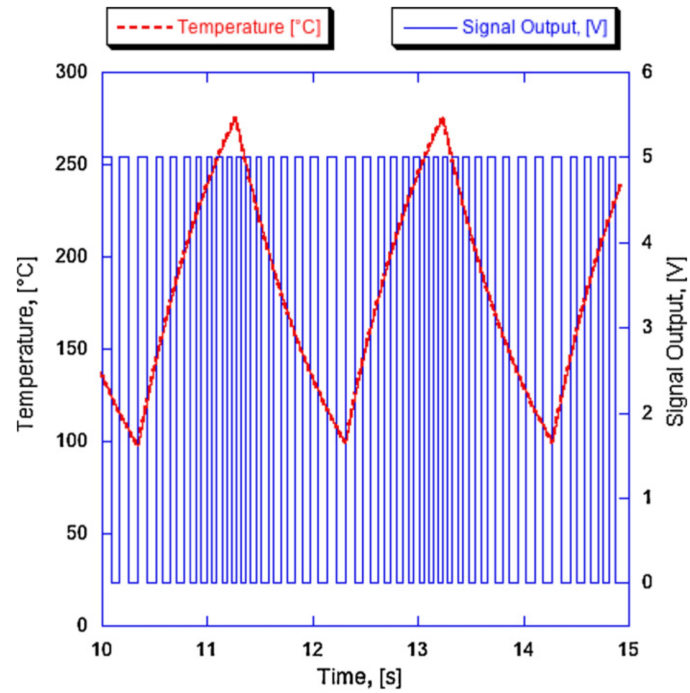

Fig. 5. Calculated output signal and sensor temperature as a function of time.

hoc defined software component. The main issue of the simulated sensor is the resistance as a function of temperature and gas quality and quantity. To this regard, a literature example of resistance vs. temperature for different gases has been considered, where the behavior of doped SnO_2 sensor is reported [20]. In that paper, resistance vs. temperature curves are reported for four gases (methane, butane, CO, and H_2) and at different concentrations. Fig. 4 shows the extrapolated curves for CO, H_2 and methane (gases A, B, C respectively) and another curve *ad hoc* created to mimic the NO behavior (gas D). For sake of simplicity, a generic label for gases is used, and the curves describe the sensor in saturation conditions. To reproduce the effect of concentration, it has been supposed that, for each gas, the resistance–temperature curves is given by the following equation:

$$R_{\text{sensor}}(T) = \alpha \cdot R_{\text{air}}(T) + \beta \cdot R_{\text{gas}}(T) \quad \text{where} \quad \alpha + \beta = 1 \tag{3}$$

where R_{air} is the sensor resistance in a reference inert air and R_{gas} is the sensor resistance in saturation conditions. The coefficients α and β are weight parameters that describe the gradual transition from R_{air} to R_{gas}. Then these parameters depend on the sensor sensitivity and the gas concentration.

Simulation was limited to the steady-state condition, then an ideal step response to gas of the sensors was assumed. Namely, the sensor reached immediately the steady state after the gas application. Since sensor dynamic was not taken into account, only the steady-state output signal was considered. In this condition, the temperature immediately reaches a periodic behavior and $x_{\text{out}}(t)$ is a stable pattern of pulses as shown in Fig. 5.

Fig. 6 shows the temperature modulation for different gases obtained by the circuit simulations. The figure clearly shows that different time patterns are obtained confirming that the modulation depends on the gas and in particular on the gas dependent temperature–resistance relationship. It is important to remark that the temperature values are derived by the voltages applied to heater.

Simulation shows that different temperature modulations produce x_{out} patterns that are formed by the same number of pulses but with different lengths. A comparison between patterns then requires an alignment of the pulses sequences that is obtained defining the first pulse of the sequence. Herewith, the shortest pulse was considered as the first pulse where the first semi-period of the pulse was characterized by the high voltage level. The length of the each semi period of pulses is the more obvious feature that can be defined to describe the pattern of pulses. All the features were then arranged in a vector that is the multivariate response of the sensor

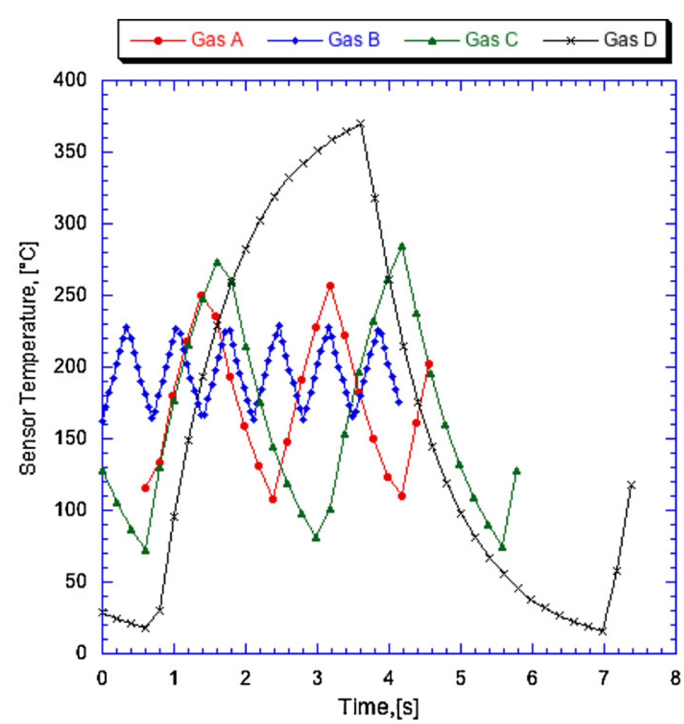

Fig. 6. Calculated pattern of temperature in the cases of three gases. The temperature behavior depends on the resistance vs. temperature relationship of Fig. 4. The temperature profiles differ on intensity, frequency, and phase.

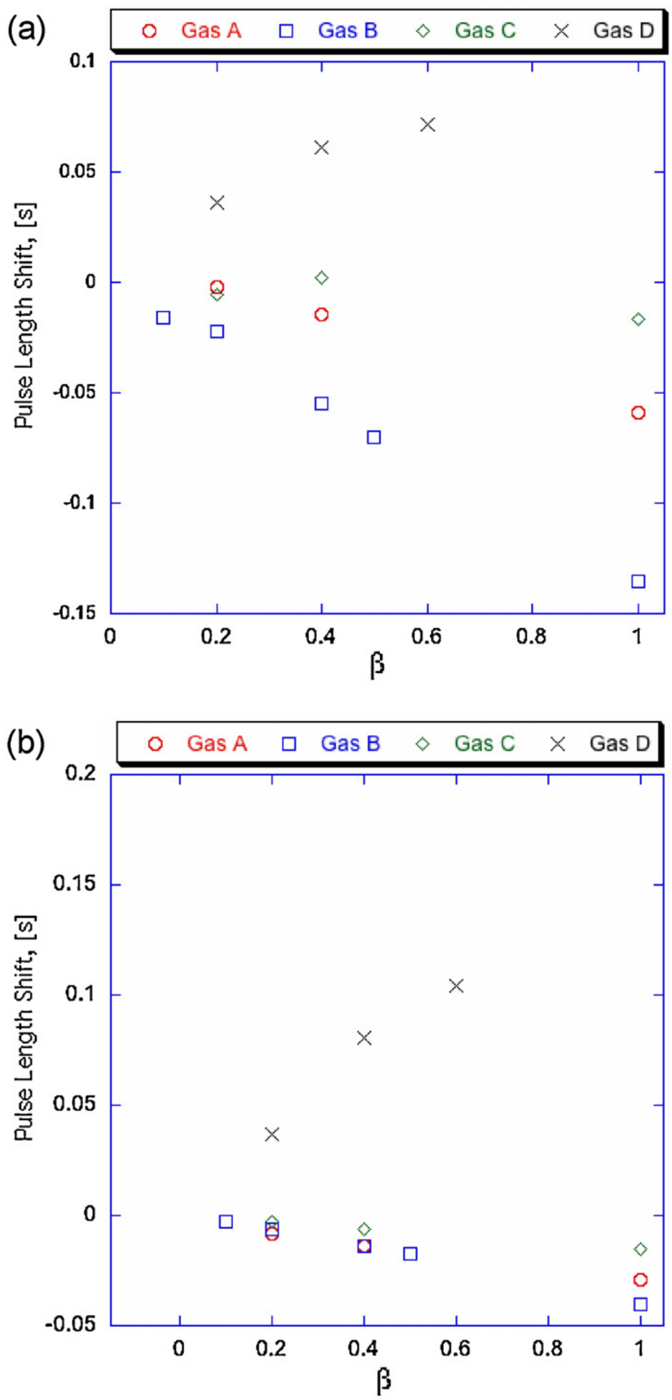

Fig. 7. Dependence of two of the features describing the periodic pulse sequence plotted vs. the parameter β of Eq. (3) and for different simulated gases.

to a single measure. A differential sensor response was then calculated subtracting the pulse lengths vectors measured with the sensor exposed to gas, and with the sensor in reference air (pulse length shift).

Fig. 7a and b shows the plots of two elements of the vector of differential sensor response plotted vs. the β coefficient of Eq. (3). Considering the definition of this coefficient, these plots provide an indication of the variation of the features with gas concentration.

Fig. 7 illustrates the selectivity and sensitivity of these sensor response features, for instance the first of these features (Fig. 7a) shows opposite sensitivities for gases B and D, while the other

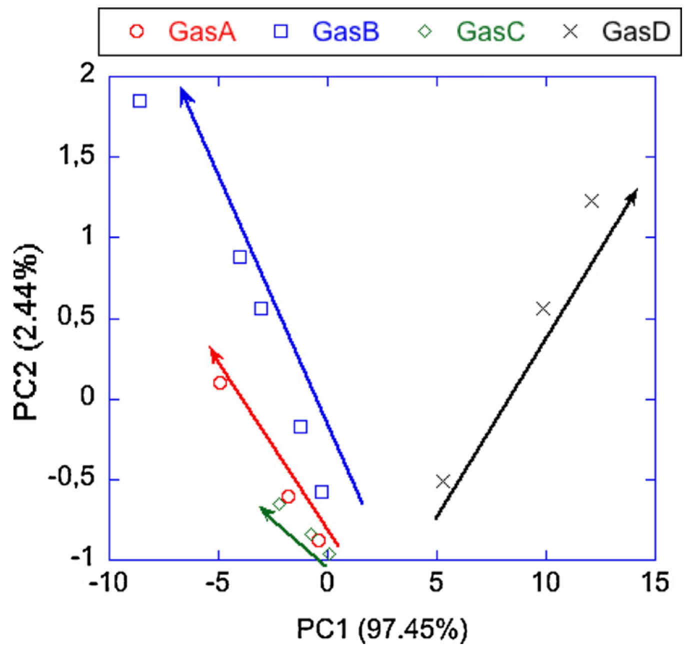

Fig. 8. Scores plot of the first two principal components of the PCA model calculated on the features vector matrix of the simulated data. The progression of concentrations of the different gases points towards different directions of the scores plane.

feature (Fig. 7b) is almost sensitive only to gas D. A synthetic appraisal of the feature discrimination properties is offered by the principal component analysis of the features vectors. Fig. 8 shows the scores plot of the first two principal components (PC1–PC2) where a clear quantitative and qualitative separation of the gases is achieved [21]. A deeper investigation about the relationship between resistance vs. temperature curves of Fig. 4 and the results reported in Figs. 6–8 is necessary, here it may be sufficient to observe that gases with similar curves in Fig. 4 are plotted close in Fig. 8. On the contrary, gas D, for which a net segregation in the principal component plot is observed, is also characterized by distinct resistance vs. temperature curve (Fig. 4) and different temperature modulation (Fig. 6).

4. Experimental

Following the results of the simulations, the circuit in Fig. 1 was assembled using as a sensor, a commercial tin-oxide device from Figaro Inc. The sensor was a TGS2600, a sensor indicated for air contaminants detection [22].

To test the method in a real context, an experiment aimed at identifying different concentrations of nitrobenzene (26 and 52), carbon monoxide(10, 25 and 50 ppm), nitric oxide (10, 25 and 50 ppm) and a mixture of nitric oxide (25 ppm) and nitrobenzene (26 ppm) was performed.

The sensor was placed into a 20-ml volume sealed chamber with orifices for gas inlet and outlet. Gas delivery was controlled by a mass flow controller system. Different gas concentrations were obtained diluting the volatile compounds in a synthetic air carrier; the same carrier was also used to clean the sensor. Certified bottles were used for carbon monoxide and nitric oxide. The concentration of nitrobenzene was estimated considering the dilution factor of the saturated vapor pressure calculated from the Antoine equation with the parameters reported by the NIST database [23]. Ten and thirty minutes were chosen for the measuring and cleaning phases respectively. All measures were performed in triplicate.

During the experimental measurements, the time evolution of the output sensor signal is stored in a computer by means of a data

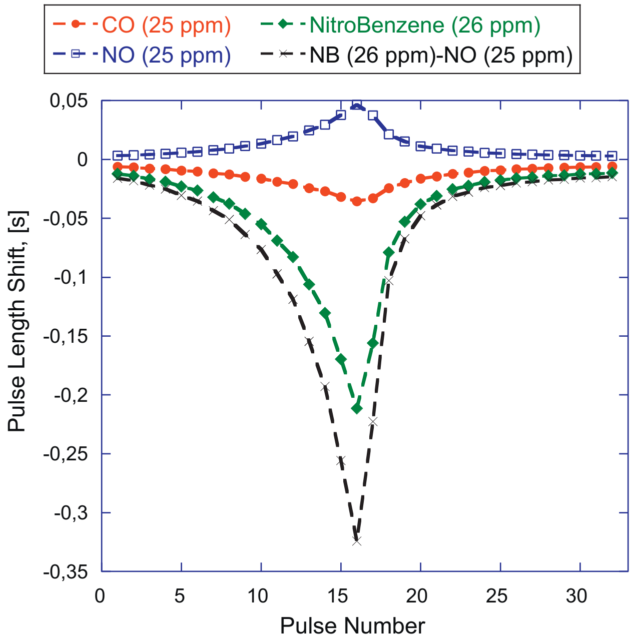

Fig. 9. Changes in pulses time length measured for the gases and the mixture.

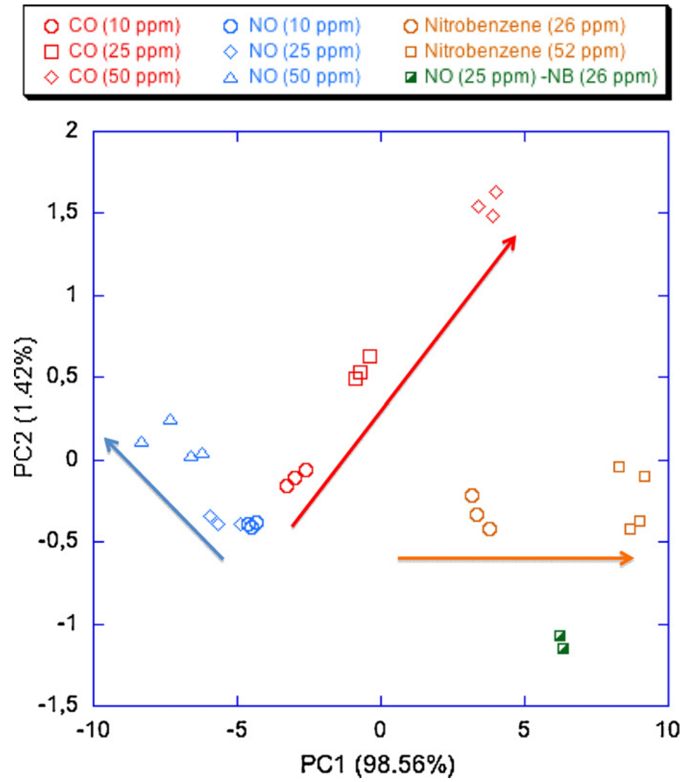

Fig. 10. Scores plot of the first two principal components of the PCA model of the whole set of experimental data. Each gas is characterized by a well-defined direction for the progression of concentrations.

acquisition board (NI-USB-6211, National Instruments [24]). All the routines for the processing and the analysis of the experimental and simulated data have been developed in the MATLAB environment [25].

5. Results

The circuit parameters have been adapted to the characteristics of the used sensor, in particular the following limit voltages applied to the heater have been chosen: $V_{min} = 3$ V and $V_{max} = 5$ V. These values correspond to an equilibrium temperature of $T_{min} \approx 250\,^{\circ}$C and $T_{max} \approx 400\,^{\circ}$C, respectively. The main difference between simulation and real experiment is the finite response time of the sensor resistance to the gas, and the sensor temperature to the signal applied to the heater terminals. For the parameter of the digital counter of the temperature modulation interface the value $k = 16$ was used in the real experiment.

After transient time due to gas exposure, the measured signal sequences were qualitatively similar to those obtained by the simulation and following the method outlined in Section 3, a multicomponent vector formed by the length of the 32 semi-periods of the 16 pulses of the pattern was used as sensor response. The sensor response was then expressed by the differential features as defined in Section 3.

Fig. 9 shows the changes in pulses length for different gases and the mixture, for simplicity only one measure per gas is shown. It is interesting to observe that these behaviors differ in quantity (case of CO, nitrobenzene, and mixture) and quality (NO with respect to the others). A better display of the discrimination properties is obtained processing with PCA the whole set of data. Fig. 10 shows the scores plot of the first two principal components calculated on the autoscaled data matrix. Data are well separated according to their quality and quantity. The discrimination property of the sensors data is shown by the fact that vectors indicating for each gas the progression of concentrations point towards different directions of the scores plane. This result is qualitatively similar to that obtained with simulated data that was obtained assuming a simplified sensor model.

6. Discussion

With respect to the literature studies, the temperature modulation here presented is not driven by a fixed signal, a priori defined, but it is determined by the sensor itself through its proper resistance vs. temperature relationship. Here a practical implementation of the concept has been illustrated. It is based on a close loop circuit designed connecting the sensor resistance to an astable multivibrator oscillator and using the oscillator output to drive the heater circuit. In such a circuit the resistance vs. temperature relationship builds a stable sequence of pulses as the output of the oscillator. Changes in the resistance vs. temperature relationship can then modify the sequence of pulses. Since the relationship between sensor resistance and temperature intrinsically depends on the gas, the sequence of pulses is specific for the kind of gases and for its concentration. The sequence of pulses can be represented by a vector formed with the time length of the semi-periods of each pulse of the sequence.

Both simulations and real experiment demonstrate that the vector of features can discriminate gases and their mixtures according to their quality and quantity. Moreover, simulated experiment also evidenced that the ranges of temperature modulations are specific for the kind of gas. This evidence confirms the fundamental principle of this approach where, for a given sensor, the gas sets the temperature modulation.

Nevertheless, some aspects of this strategy have to be clarified, such as the role of the digital counter in the gas discrimination. The counter defines the time scale of temperature cycles, and it adjusts the pulse period to the frequency response of the thermal circuit of the sensor. Here an empiric choice was adopted, as a consequence, the length of the digital counter is a non-adaptive part of the circuit and at this stage of development of self-adapted modulation it is still based on an a priori analysis of the sensor and its application.

Another important aspect is concerned with the fact that the discrimination properties of the system here discussed are based on the steady-state condition of the sensor. This condition that is standard in laboratory sensor tests is hardly met in real world application where gas concentration can change faster that than the sensor response time and then the sensor never reach its steady-state condition. Furthermore, the dynamics of the sensor signal between two steady-state conditions can also be used to retrieve information about the related change of gas quality and quantity. During this transient time, the pulse pattern is not stable and the feature to describe an evolving pattern may be of difficult definition. However, it will be interesting in the future to consider how to extend the method to the cases of unsteady sensor signals.

7. Conclusions

In this paper an alternative method of connecting a metal oxide sensor to an electronic circuit is considered. Differently from the standard use of these sensors, measuring and heater terminal are both connected to the same circuit and the sensor temperature is determined by the sensor resistance. This method implements the concept of self-adaptation that allows a sensor to set the temperature modulation according to the variation of its conductivity when it interacts with volatile compounds. The concept is here presented with a simple implementation based on an oscillator circuit in order to obtain, in correspondence of a stable sensor state, a periodic sequence of pulses.

Simulation and experiments have been performed and compared, and both shown that a multicomponent variable formed by the length of the two semi-periods of the pulses composing the pattern sequence can discriminate both the quality and quantity of gases and mixtures.

The method has been here demonstrated with a commercial metal-oxide semiconductors, however, it could be extended to any conductometric sensor where the sensitivity of the resistance with respect to gas concentration is a function of the sensor temperature. In case of metal-oxide semiconductors this dependence is relevant to the detection, but in principle it could also be applied to other materials, such as conductive polymers.

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Biographies

Eugenio Martinelli is an assistant professor in electronics at the Faculty of engineering of the University of Rome Tor Vergata. His research activities are concerned with the development of chemical and biological sensors, artificial sensorial systems (olfaction and taste) and their applications, sensor interfaces, data processing. He authored more than 130 peer-reviewed papers on international journals and conference proceedings. He is member of the editorial board of Journal of Sensors and he is regular referee for a number of journals of the sector.

Davide Polese received an mathematical engineering degree from the University of Rome Tor Vergata in 2008. Currently he is a PhD student in Engineering of Sensorial and Learning Systems at the University of Rome Tor Vergata. His research interests are in the bio-inspired algorithms and multivariate data analysis of gas chemical sensor array.

Alexandro Catini was born in Rome, Italy, on June 06, 1978. Since 2003, he works for Sensors and Microsystems Group at the University of Rome "Tor Vergata", on physical, chemical and biological sensors mainly devoted to the monitoring of natural environment. In 2007 he received the Ph.D. degree in Microsystems Engineering from University of Rome Tor Vergata. From 2009 he got a Post Doc grant on the theme: Innovative sensorial system for security and environmental applications. Main areas of expertise and interest include electro-optical and chemical sensor systems, electronic sensor interfaces, wireless sensors networks.

Arnaldo D'Amico received the Laurea degrees in physics and in electronic engineering from the University La Sapienza, Rome, Italy. For several years, he has been with the National Research Council (CNR) leading the Semiconductors Laboratory at the Solid-State Electronics Institute, Rome. In 1988, he was appointed Full Professor of electronics at the University of L'Aquila, and, since 1990, he has been with the University of Rome Tor Vergata where he leads the Sensors and Microsystems Group and is Full Professor of electronics. He teaches courses on electronic devices, micro- and nano-systems, and sensors at the Faculty of Engineering. Currently, his main research activities are concerned with the research and development of physical and chemical sensors, low voltage electronics, noise, and advanced electronic devices. He is author of more than 550 papers in international journals and conference proceedings. He has been Chairman of several conferences on sensors, electronics, and noise and a member of the editorial board of the journals Sensors and Actuators A (physical) and Sensors and Actuators B (chemical). He served as Chairman of the Steering Committee of the Eurosensors conference series from 1999 to 2004 and currently he is member of the steering committee. At national level, he is Chairman of the National Society of Sensors and Microsystems (AISEM) and Director of the "Corbino Institute of Acoustic" of the National Research Council (CNR).

Corrado Di Natale is an associate professor at the Faculty of Engineering of the University of Rome Tor Vergata where he teaches courses on Sensors and Detectors. His research activities are concerned with the development and application of chemical, bio-sensors, and artificial sensorial systems (olfaction and taste), and with the study of the optical and electronic properties of organic and molecular materials. He authored more than 420 papers on international journals and conference proceedings. He chaired the 9th International Symposium on olfaction and electronic nose (Rome, 2002) and Eurosensors XVIII Conference (Rome, 2004) and was member of the organizing committee of national and international conferences in sensors. He serves as component of the steering committee of the Eurosensors conferences series and as associate editor of IEEE Sensors Journal. He edited several proceedings volumes, and is a regular referee for a number of journals of the sector. In September 2006 he received the Eurosensors Fellow Award for his contribution in the fields of chemical sensors and artificial senses.