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Effect of surface catalysts on the long-term performance of reactively sputtered tin and indium oxide gas sensors

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Abstract

Long term performance of tin and indium oxide thin films with palladium and platinum deposited on their surface as catalysts was studied using a standardization method proposed recently by other researchers. A simple gas classification algorithm was used to investigate the validity of the standardization method proposed and to study the effect of doping type and thickness onto the surface of SnO_x and InO_x films on their selectivity and stability characteristics. It was found that: (a) the proposed method is reliable for the assessment of sensor degradation; and (b) sensors with relatively thick additive layer are more reliable. © 1997 Elsevier Science S.A.

Keywords: Metal oxide gas sensors; Surface catalysts; Gas classification; Sensor selectivity; Sensor degradation

1. Introduction

Metal oxide gas sensors offer many advantages as simple fabrication, low cost, high sensitivity to both reducing and oxidizing gases, easy integration, etc. Main disadvantages of these sensors are poor selectivity and stability. Noble metals used as catalysts enhance their characteristics but reliable sensors have not been reported yet. It is important to fabricate sensors not necessarily sensitive to one gas, but sensors exhibiting different responses to different gases of interest ('non-selective but different'), for their incorporation in sensor arrays in order to design systems selective to the desirable gases [1,2]. Researchers are trying to apply linear regression methods like PCA (principal component analysis) or PLS (partial least squares) to enhance selectivity of a linear sensor array. Non-linear regression techniques like MARS (multivariate adaptive regression splines) [3], PPR (projection pursuit regression) [4] or TLS (transformed least squares) [5,6] are used for non-linear sensor arrays. Even the application of linear regression techniques for non-linear but redundant sensor arrays can provide an adequate prediction accuracy [7]. All these methods use statistical tools to evaluate the performance of an array or individual sensor elements as parts of an array. In this work the performance of a single sensor was evaluated concerning its selectivity to four components with respect to its degradation, without considering the sensor as part of an array. This analysis can provide useful information for the characterization of a sensor, but is not adequate for the characterization of an array which would use this sensor.

The performance of a sensor was modeled using the distances between different classes with respect to the translation of the sensor response in the n-dimensional space. Each class (gas) occupies a specific volume in the n-dimensional space. The pattern recognition system used is called to map the points of the sensor space onto classification space. More than one sensors can be used, thus defining a sensor array. If the target gases do not provide well distinguished areas in sensor space, then the recognition algorithm cannot be expected to discriminate them. For example, if areas A and B in Fig. 1 overlap, it is not possible to distinguish the gases corresponding to these areas with a single measurement, especially if the measurement falls in the cross section of these areas. Consequently, the performance of individual sensors is of major importance for correct classification.

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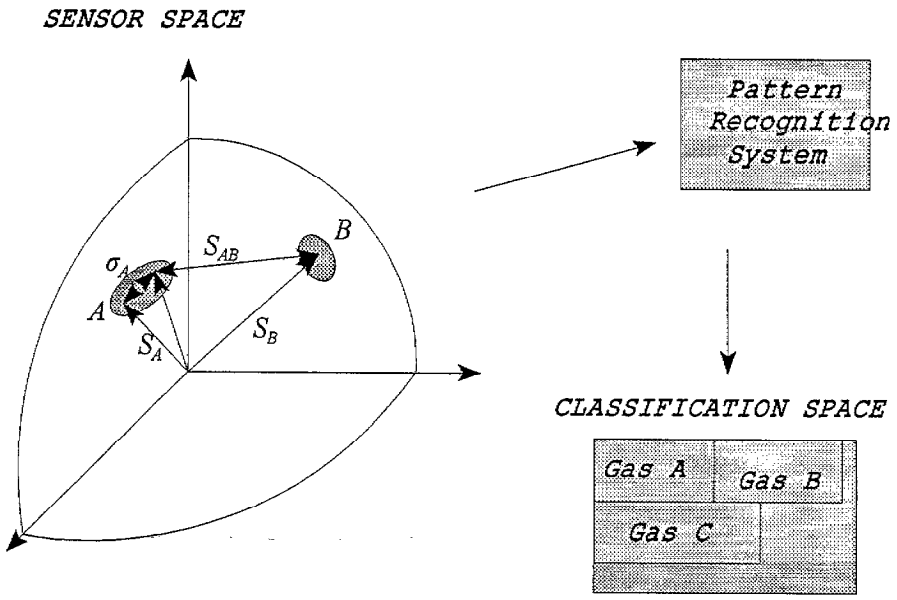

Fig. 1. Gas classification system.

The operation of metal oxide gas sensors is based on the interaction of ambient gases with sensor surface. This interaction leads to modification of chemical composition of the oxide and consequently to a change to its electrical properties. Since this kind of sensors operates at elevated temperatures, continuous degradation of sensor properties with operation is a usual phenomenon. The shaded regions of the hypersphere of Fig. 1 represent the regions in which the responses of a sensor to two gases A and B fall, for a number of consecutive measurements. Two measurements corresponding to gas A are shown. The area of these regions with respect to the distance between them can provide a measure of sensor reliability.

2. Experimental techniques

Deposition of SnO_x and InO_x films was performed with a Leybold Z-400 planar magnetron sputtering system, which was d.c. operated in a controlled, high purity Ar-O_2 mixture. Two different discharge modes may be obtained, the metallic mode and the reactive mode, where the target is respectively free from or covered with reaction products. The compound formation reactions are desirable in the metallic mode for high rate depositions, and for a more precise control of the O_2/metal (Sn or In) ratio of the deposited film. This necessitates the use of a plasma emission monitor (PEM) system, that allows us to select and maintain the degree of target oxidation [8], with the tin or indium emission line intensity at 450 nm as a measurand for the control loop of the reactive gas mass flow controller. This technique has been proven adequate for the fabrication of reproducible films.

Sputtering was carried out on Al_2O_3 (96%) substrates with dimensions 12 × 26 mm, heated at 300°C. Films were fabricated at a relatively high total gas pressure of approximately 1.0×10^{-2} mbar. The substrate holder was at a distance of 7 cm from the 10 cm in diameter target. The total power into the tin or indium target was 115 W with a total current of 0.36 A, thus achieving a deposition rate of approximately 100 nm min^{-1}. Argon flow was adjusted manually at 20 ml min^{-1}, while oxygen intake was controlled by the aforementioned PEM control unit, so that the intensity of the metal (tin or indium) emission line remained constant. An O_2 flow around 30 ml min^{-1} was found to give an appropriate oxygen to metal (tin or indium) ratio, so that the films fabricated can function as sensors with a base resistance below 30 MOhms at 100°C (low oxygen to metal ratio gives metallic films and high oxygen to metal ratio gives highly resistive films). The thickness of all films fabricated was equal to 1000 nm (10 min sputtering time). Films were not annealed. The eight lots of tin oxide films, which were fabricated under the same experimental conditions, showed the same electrical properties within a 10% tolerance. In addition, the eight lots of indium oxide films, which were fabricated under the same experimental conditions, showed the same electrical properties within a 10% tolerance.

Subsequently, palladium was electron beam evaporated on three of the aforementioned SnO_x films and on four of the aforementioned InO_x films. Platinum was deposited on the four SnO_x films and on the other four InO_x films. One tin oxide film was left plain (without

additive). Evaporation was performed with an Edwards 306 vacuum evaporation apparatus. Total pressure during electron beam evaporation was about 3×10^{-5} mbar, while the substrates were not heated during evaporation. Evaporation was carried out at a relatively low deposition rate, of the order of 3 Å s^{-1} for palladium and 1 Å s^{-1} for platinum. Palladium was deposited simultaneously on three SnO_x and three InO_x films with evaporation times of 5, 10 and 20 s. On the fourth InO_x film palladium deposition time was 30 s. Thus three SnO_x and four InO_x films with increasing Pd layer on each of them have been fabricated. We refer to these sensors as SnO_x + Pd1, SnO_x + Pd2, SnO_x + Pd3, InO_x + Pd1, InO_x + Pd2, InO_x + Pd3 and InO_x + Pd4, depending on palladium thickness. In the same manner but at a much lower rate (1 Å s^{-1}) platinum was deposited on four SnO_x and four InO_x films with evaporation times of 10, 20, 30 and 40 s, respectively. In an analogous sense we refer to these sensors as SnO_x + Pt1,..., InO_x + Pt4. The average thickness of the metal films deposited onto oxide surface has been evaluated by activation energy measurements, with the procedure described in [9], and has been found around 3–5 nm. For details concerning the physical properties and the structure of the additive layers one can refer to [9].

The sensor characterization set up was designed to measure the steady-state and the transient response of the samples, testing them at various working temperatures and under different gas compositions. The testing conditions were rigorously controlled via a fully automated, computer controlled system. All sensors have been characterized according to the following experimental procedure:

- The samples were heated to 450°C and cooled back to 150°C in zero grade air (ZGA, dry synthetic mixture of 80% N_2 and 20% O_2). This step was repeated three times before starting film characterization, in order to clean the samples from water vapor remains.
- The samples were heated to 450°C and cooled back to 150°C in 2000 ppm carbon monoxide diluted in ZGA.
- The samples were heated to 450°C and cooled back to 150°C in zero grade air.
- The last two steps were repeated four times again but with test gas compositions of 1% methane, 26 ppm ethanol and 2% butane diluted in zero grade air, instead of CO.
- The samples were submitted to 50 thermal cycles (heating to 450°C-cooling to 150°C), in the presence of ambient air.
- The samples were tested again to all target gases, repeating the aforementioned procedure. In this way a second set of sample responses was recorded, after 50 aging cycles.
- With the aforementioned procedure the response of the samples to test gases was recorded after 100, 150 and 250 subsequent aging cycles. In this way five sets of sample responses were recorded.

During the aforementioned measuring cycles, the resistance of the samples was recorded at 5°C intervals as the samples were cooled from 450 to 150°C; in this way 61 resistance values were recorded for the response of each sensor to each test gas. The cooling speed was 10°C/min. At temperatures above 250°C, this speed ensures that no significant transient effects have been recorded [10].

3. Mathematical treatment

3.1. Pre-processing of the data

The acquired experimental data include resistance values for both tin and indium oxide based sensors, covering a range of several orders of magnitude (indium oxide based sensors are by far more conductive than tin oxide based ones; particularly platinum doped tin oxide sensors exhibit non-measurable conductance values). As a consequence, a scaling technique is necessary in order to compensate the data for large resistance variances. A simple pre-processing technique that gives each variable equal weighting by adjusting the original data to remove inadvertent weighting on values covering a wide range was adopted and it is briefly described below.

Vectors were used in order to represent the response of the sensors. Each vector contains the resistance values of the sensor in the presence of the corresponding test gas in the temperature region from 150–450°C, recorded at 5°C intervals; thus the dimension of each vector is 61((450–150)/5 + 1).

The original data have been autoscaled so that the vector elements of each sensor are mean centered with a standard deviation of one. The following relationship gives the formula for the calculation of the autoscaled data [11]:

$$r_i' = \frac{r_i - r_{av}}{\sigma}, \quad i = 1,..., N \tag{1}$$

where r_{av} is the average resistance over all vector elements of each sensor at the presence of each test gas at each measuring cycle and σ is the standard deviation of the sample according to the following formula [11]:

$$\sigma^2 = \frac{\sum_{i=1}^{N} (r_i - r_{av})^2}{N-1} \tag{2}$$

where $N = 61$ (the dimension of the vector).

3.2. Performance standardization

In order to compare the performance of sensors it is necessary to define some standards. Recently J.W. Gardner and P.N. Bartlett [1] presented a simple method to represent with a number the ability of a sensor to discriminate two gases. Their method consists of the calculation of a factor, called resolving power (or electronic selectivity), which takes into account both the distance of the response vectors to two gases and the errors encountered. In a mathematical form, the resolving power of a sensor for two gases is defined by the following equation:

$$R = \frac{|S_{AB}|}{\sqrt{\sigma_A^2 + \sigma_B^2}} \qquad (3)$$

where $|S_{AB}|$ is the distance between in the response vectors $\boldsymbol{A}$ and $\boldsymbol{B}$ and σ_A and σ_B are the errors calculated along the direction of the distance metric. In the situation at hand, $|S_{AB}|$ is the Euclidian distance between the responses of the sensor to two test gases and σ_A and σ_B are the distances between the responses of the sensor to the test gases A and B, respectively, before and after a predetermined number of aging cycles (these distances are encountered as errors). Referring to Fig. 1, σ_A and σ_B denote the distance of the response points of two successive measurements under test gases A and B; the resolving power denotes the change of the distance between the two points with respect to the translation of the points with aging. Thus, if this factor is considerably larger than unity, then the sensor can discriminate the gases, while if it is smaller than unity the sensor should not distinguish the gases.

3.3. Classification rule

The pattern recognition engine used is a simple supervised learning algorithm. The response vectors to the four test gases are recorded during a measurement and are used as a decision rule for the successive measurement. The 'learned' response vectors are then replaced by the new measurement data. The decision rule consists of evaluating the four distances of the unknown vector from the four known classes and classifying the unknown response to the class from which this distance is minimum. Table 1d, Table 2d and Table 3d show the classification results according to this simple rule. The headings show the actual gases present and the four lines under the headings show the 'class' to which the test gas was attributed. If this gas coincides with the heading the classification is correct. The first row under the heading shows the classification for the response vectors taken after 50 aging cycles, with 'training data' the response vectors taken without aging. In an analogous sense the fourth row shows the classification results for the response vectors after 250 aging cycles, while the system was 'trained' with the response vectors after 150 aging cycles.

4. Results and discussion

Results from the most stable and selective sensors are presented in Tables 1–3. Tables 1–3 show the translation of the response vectors at each gas with aging (a), the resolving power for the response vectors after 100 and 150 aging cycles (b), the resolving power for the response vectors after 150 and 250 cycles (c), and the results of the gas classification algorithm (d). The headings of Table 1d, Table 2d and Table 3d show the actual gas that was present. From Tables 1–3, it is easy to investigate the behavior of the selectivity of each one of the sensors, in conjunction with the effect of degradation with continuous use, given that the resolving power reflects in practice sensor selectivity.

Table 1a shows that after 100 temperature cycles the tin oxide sensor with the thicker palladium layer becomes remarkably stable to all test gases. This stability

Table 1
Classification results for a palladium doped SnO_x sensor

	CO	CH_4	C_2H_5OH	C_4H_{10}
(a) Effect of aging				
Aging				
0–50	328	843	60	419
50–100	357	518	41	29
100–150	11	42	13	7
150–250	90	26	20	79
(b) Resolving factor, 100–150 aging cycles				
CO	0	746	1855	2667
CH_4	*	0	1167	1292
C_2H_5OH	*	*	0	307
(c) Resolving factor, 150–250 aging cycles				
CO	0	432	286	265
CH_4	*	0	1633	720
C_2H_5OH	*	*	0	134
(d) Classification results				
Aging				
0–50	CO	CO	C_2H_5OH	CO
50–100	C_4H_{10}	C_4H_{10}	C_2H_5OH	C_4H_{10}
100–150	CO	CH_4	C_2H_5OH	C_4H_{10}
150–250	CO	CH_4	C_2H_5OH	C_2H_5OH

The heading row shows the gas actually present and each row shows the output of the classification algorithm if the system has been trained with the data recorded prior 50 (rows 1, 2 and 3) and 100 (row 4) temperature cycles.
(a) Euclidian distances among the response vectors to the same test gases after a number of temperature cycles.
(b) Resolving factors denoting the selectivity of the sensor if it is submitted to 50 temperature cycles, with a pretreatment of 100 temperature cycles.
(c) Same as (b) if the sensor is submitted to 100 temperature cycles, with a pretreatment of 150 temperature cycles.
(d) Output of a classification algorithm.

Table 2
Classification results for a platinum doped SnO_x sensor

	CO	CH_4	C_2H_5OH	C_4H_{10}
(a) Effect of aging				
Aging				
0–50	1363	869	706	747
50–100	1134	9	515	743
100–150	1028	25	129	146
150–250	634	8	57	133
(b) Resolving factor, 100–150 aging cycles				
CO	0	99	128	85
CH_4	*	0	722	820
C_2H_5OH	*	*	0	685
(c) Resolving factor, 150–250 aging cycles				
CO	0	173	208	138
CH_4	*	0	1661	923
C_2H_5OH	*	*	0	951
(d) Classification results				
Aging				
0–50	C_2H_5OH	C_2H_5OH	C_4H_{10}	C_2H_5OH
50–100	C_4H_{10}	CH_4	C_2H_5OH	C_4H_{10}
100–150	CH_4	CH_4	C_2H_5OH	C_4H_{10}
150–250	CO	CH_4	C_2H_5OH	C_4H_{10}

The heading row shows the gas actually present and each row shows the output of the classification algorithm if the system has been trained with the data recorded prior 50 (rows 1, 2 and 3) and 100 (row 4) temperature cycles.
(a) Euclidian distances among the response vectors to the same test gases after a number of temperature cycles.
(b) Resolving factors denoting the selectivity of the sensor if it is submitted to 50 temperature cycles, with a pretreatment of 100 temperature cycles.
(c) Same as (b) if the sensor is submitted to 100 temperature cycles, with a pretreatment of 150 temperature cycles.
(d) Output of a classification algorithm.

results to high resolving power (shown in Table 1(b, c) and consequently the sensor can distinguish the test gases with the discrimination algorithm used (Table 1d). As it is obvious from Table 1a (first two rows) the sensor must be submitted to 100 temperature cycles in order to become stable. During this pre- treatment it is believed that severe changes take place, both to the structure and stoichiometry of the tin oxide layer, and to the form of the catalytic layer. Recall that the tin oxide film was not annealed and deposition of palladium took place at ambient temperature. Note that the sensor cannot distinguish butane from ethanol after 250 operation cycles (the recognition algorithm decided 'C_2H_5OH', whereas 'C_4H_{10}' was present, Table 1d). Note also that the relatively low resolving factor of 1.34 at Table 1c implies this error.

An analogous behavior can be observed in Table 2 for the platinum doped sensor. Note from Table 2a that the response of the sensor to CO is not stable, whereas its response to the other test gases is stable. This can also be observed at Table 2(b, c), where the first lines (corresponding to the resolving factors of CO with the other test gases) imply that this sensor should not recognize CO. Finally, classification results at Table 2d confirm the incapability of the sensor to recognize CO, whereas it can successfully distinguish the other three test gases.

Palladium doped indium oxide sensors exhibited serious degradation with temperature cycling and the classification algorithm was not able to correctly discriminate the test gases. The corresponding data are not presented.

The behavior of the platinum doped indium oxide sensor with the thicker layer of platinum is shown in Table 3. This is an example of a non-selective sensor, which although is stable (Table 3a) the low resolving factors of Table 3(b, c) imply that it is not capable to classify correctly the test gases. This happens indeed, as shown in Table 3d. Indium oxide based sensors were generally found non-selective. Their behavior continues to degrade even after they were submitted to 250 operation cycles.

The structure of palladium and platinum films deposited on tin and indium oxide films was estimated by

Table 3
Classification results for a platinum doped InO_x sensor

	CO	CH_4	C_2H_5OH	C_4H_{10}
(a) Effect of aging				
Aging				
0–50	1158	1539	1344	1051
50–100	229	223	735	247
100–150	172	170	151	104
150–250	136	149	48	208
(b) Resolving factor, 100–150 aging cycles				
CO	0	61	58	194
CH_4	*	0	82	123
C_2H_5OH	*	*	0	160
(c) Resolving factor, 150–250 aging cycles				
CO	0	45	58	194
CH_4	*	0	95	205
C_2H_5OH	*	*	0	218
(d) Classification results				
Aging				
0–50	C_2H_5OH	C_2H_5OH	C_2H_5OH	C_2H_5OH
50–100	CO	CO	CO	CH_4
100–150	CO	CO	C_2H_5OH	C_4H_{10}
150–250	CH_4	CH_4	CO	C_4H_{10}

The heading row shows the gas actually present and each row shows the output of the classification algorithm if the system has been trained with the data recorded prior 50 (rows 1, 2 and 3) and 100 (row 4) temperature cycles.
(a) Euclidian distances among the response vectors to the same test gases after a number of temperature cycles.
(b) Resolving factors denoting the selectivity of the sensor if it is submitted to 50 temperature cycles, with a pretreatment of 100 temperature cycles.
(c) Same as (b) if the sensor is submitted to 100 temperature cycles, with a pretreatment of 150 temperature cycles.
(d) Output of a classification algorithm.

activation energy measurements, with the use of conductance versus temperature plots. It was found that the thicker palladium (Pd3) and platinum (Pt4) films (average thickness > 30 Å) consist of discrete islands with particle dimension around 80 Å and particle separation in the order of a few tenths Å [12–14]. It is also known that when thin discontinuous metal films are subjected to high temperatures small metal islands grow together to form particles with larger dimensions [12]. This is a degradation mechanism for the sensors, which has a larger effect for thin catalyst films. After some thermal pretreatment this mechanism stops and does not contribute to sensor degradation. Films covered with thin catalyst layers showed worse sensing properties than the ones shown in Tables 1–3, and their response is not illustrated.

5. Advantages of the proposed method

The mathematical method proposed makes a compression of the information contained in the conductivity measurements of the sensors. Information presented in Tables 1–3 would require many conductivity graphs to be displayed, the study of which would be cumbersome and misleading. The most useful characteristics of the sensors, i.e. stability or selectivity, would be very difficult to be extracted from conductivity measurements. On the other hand, the tables with the resolving factors presented give an overview of the reliability of the sensors, taking into account both their selectivity and stability.

However, the classes to which the responses of the sensors were classified refer to single concentrations of five reducing gases. Similar results would presumably be obtained if different gas concentrations were used, but if the sensors were exposed to gas mixtures the results would be very different, since synergetic effects would occur [15]. Hence, the results of this mathematical method can be applicable if the gas concentrations are different. The proposed method is not suitable for the processing of data corresponding to the exposure of sensors to arbitrary gas mixtures.

6. Conclusions

The performance of tin and indium oxide sensors, doped with varying thicknesses of palladium and platinum was evaluated against accelerated temperature aging, using a simple mathematical formula proposed recently by other researchers; this formula was proven adequate to correctly describe their performance. It was found that the thickness of the additive plays an important role in sensor stability and its capability to discriminate the four gases tested. A simple supervised pattern recognition engine was used in order to assess the selectivity of the sensors. Sensors covered with very thin noble metal layers were found unreliable, while sensors covered with a thicker catalyst layer were found more stable and selective. Tin oxide based sensors were found more reliable than indium oxide based ones, for the temperature treatment they were submitted to.

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Biographies

John Avaritsiotis was born in Greece in 1948. He received his B.Sc. (Hon.) in physics from the Department of Physics of the University of Athens in 1972.

His M.Sc. and Ph.D. degrees were obtained from Loughborough University of Technology, UK, in 1974 and 1976, respectively, in the field of thin-film technology and fabrication of thin-film devices.

From 1976 to 1979 he was employed as a research fellow in the Thin Film Group of Loughborough University of Technology. From 1980 to 1986 he was appointed lecturer at the Electronics Laboratory of Athens University, and in 1986 he was elected associate professor in the Department of Electrical Engineering of the National Technical University of Athens. In 1990 he was elected professor of microelectronics in the same department.

He has worked as a technical consultant to various British and Greek industrial firms for the incorporation of new thin-film deposition techniques in their production processes.

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His research activities have been focused in the field of microelectronic materials and thin film deposition techniques for use in the fabrication of semiconductor devices. His present research interests concern study of the electrical properties of thin insulating and semiconducting films deposited by sputtering, on-chip interconnections and MCMs for high-speed circuits, design of ASICs, development of fabrication processes for the production of solid-state gas sensors and design and prototyping of smart sensors.

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C.A. Papadopoulos was born in Greece in 1966. He received the B.E. degree in electrical engineering from the National Technical University of Athens in 1990 and the PhD degree in solid-state gas sensors in 1996, from the same department. He is currently a research associate in the microelectronics laboratory of the National Technical University of Athens.

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