PAMPA III ELECTRONIC NOSE: CONTROL ELECTRONICS DESIGN

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Abstract

The Argentine electronic nose PAMPA III is an equipment operating with a measurement system based on a set of twelve solid state gas sensors implemented on thin films deposited on a MEMS (Micro-Electro-Mechanical Systems) type microheater. Solid state gas sensors were designed and built by the Institute LAMEL-CNR (Bologna, Italy) while the thin film sensors were made in Argentina in the frame of the cooperation project CITEFA-CNEA-FI (UBA). In this work, the operating principle of control drivers for PAMPA III sensors is reported, mainly considering the chosen criterion to control the power supplied to each sensor heater and the signal conditioning circuit related to the sensing film itself.

The e-nose enables to program, in a flexible way, its functioning parameters, using control electronics and an associated software. Parameters to be changed are: heating power supplied to each sensor, bias voltage on each sensible film, velocity of the aspiration micropump etc. One of the main characteristics of the equipment is that the control circuits for sensors are of the "constant resistance" type.

All measurements data of PAMPA III e-nose are supplied to a PC which performs the statistical processing, employing the pattern recognition algorithm of Principal Components Analysis (PCA).

Resumen

La nariz electrónica argentina PAMPA III es un dispositivo que trabaja con un sistema de medición basado en la utilización de doce sensores de gas de estado sólido, implementados con sensores de película delgada, depositados sobre un microcalefactor de tipo MEMS. Los sensores de estado sólido fueron diseñados y construidos en el Instituto LAMEL, CNR (Boloña, Italia), mientras que los sensores de película fina fueron obtenidos en la Argentina en las instituciones que participan del convenio de cooperación CITEFA-CNEA-FI(UBA). En este trabajo, se presenta el principio de funcionamiento de los circuitos de control para los sensores de la nariz electrónica PAMPA III, poniendo especial énfasis en el criterio empleado para controlar la potencia entregada a los calefactores de cada uno de los sensores y el circuito de acondicionamiento de señal asociado con la película sensible propiamente dicha.

La nariz electrónica permite programar, de forma muy flexible, los parámetros de funcionamiento, lo que se logra con una electrónica de control y programación asociada.

Los parámetros a variar son: potencia de calefacción suministrada a cada sensor, tensión de polarización sobre cada película sensible, velocidad de aspiración de la microbomba, etc. Una de las características destacables del dispositivo es que el circuito de control de los sensores es del tipo de "resistencia constante".

La nariz electrónica PAMPA III entrega todos los datos de sus mediciones a una computadora personal que se encarga del procesamiento estadístico, empleando el algoritmo de reconocimiento de patrones denominado Análisis de Componentes Principales (ACP).

Introduction

Recent advances in gas sensors technology, together with electronics development and artificial intelligence knowledge, enabled the building of a new perceptive instrument: the electronic nose (e-nose). This new concept in the analytical instrumentation contributes to the fields of digitalization, simulation and extension of knowledge on human senses as it has been done before for vision and hearing. The measuring devices included by e-noses are gas sensors built with different technologies for different applications. Particularly, in case of the PAMPA III e-nose, semiconductive Metallic Oxide Gas Sensors were used. Thin film MEMS type (Micro-Electro-Mechanical Systems) sensors are built by evaporation deposition of thin semiconductors (SnO_2, ZnO or WO_3, among others) films, which must be heated locally in a temperature range from 300 to 450ºC to detect the different gaseous species. Metallic oxide semiconductors are usually doped with different elements (Al, In, Pd, Au, Sb, etc.) to increase sensor selectivity.

An e-nose is constituted by three important parts which are serially operated and act on the analyte: the specimen headspacer, the sensor sets and the system processesing the signal. E-nose data enables to discriminate odors, it is also possible, under certain conditions, to determine different concentrations of the analyte (responsible of the detected odor) [1], Figure 1. It is necessary to point out that an e-nose always operates by comparison and that it does not perform a chemical analysis but a discrimination of odors, enabling to separate different analytes by their odors.

Experimental Procedure

Microheating plate technology

Architecture of the thin film solid state sensor on the microheater

Selected thin films for the PAMPA III e-nose were deposited on a silicon supported membrane (Si_3N_2) Closed Membrane Type. The membrane was then heated between 300 and 450ºC with an integrated heating device (consisting of a thin Pt film, deposited by sputtering). The membrane was encapsulated and interconnected to a TO-8 capsule with a Au bonding wire (diameter: 50µm). The membrane was used to isolate thermically the hot thin film sensor from its substrate and capsule, thus improving considerably the yield of this heating system in comparison with that of other known sensor architectures. This ensamble also enabled the operation of bonding wires and microwelding at temperatures lower than 50ºC, i.e. under normal conditions, Figure 2.

To build the microheater: on a micromachined Silicon substrate (4mm long, 4 mm wide and ~500 µm thick) a non stoichiometric Si_xN_y coating was grown by LPCVD (Low Pressure Chemical Vapor Deposition) [2]. In the case of the PAMPA III e-nose, membranes obtained at the LAMEL Institute, CNR (Bologna, Italy) were actually used for the microheater although, simultaneously, a microheater completely designed in Argentina is being developed to replace it.

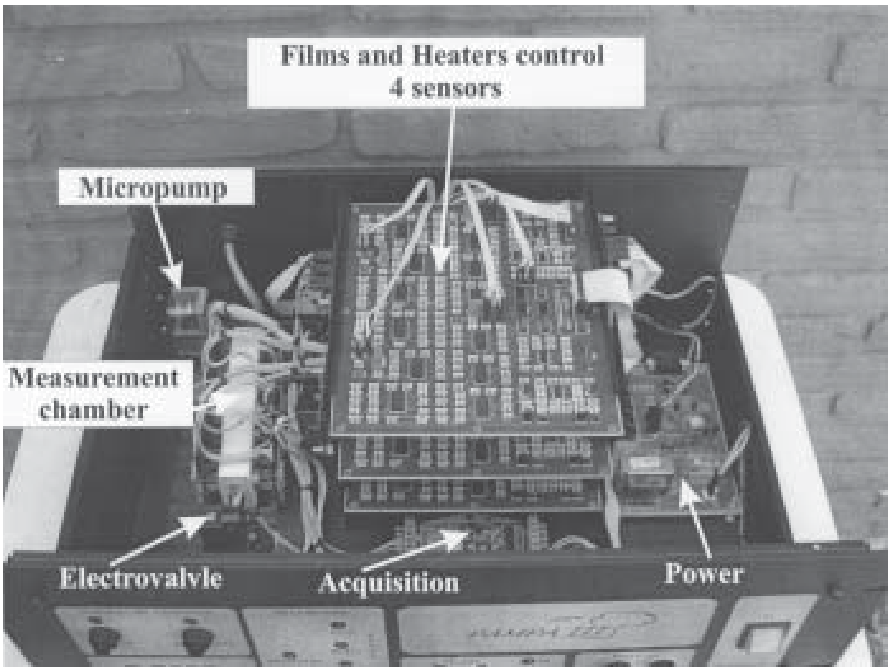

Fig. 1. *Photograph of the electronic nose PAMPA III, detailing different stages of the electronic circuit.*

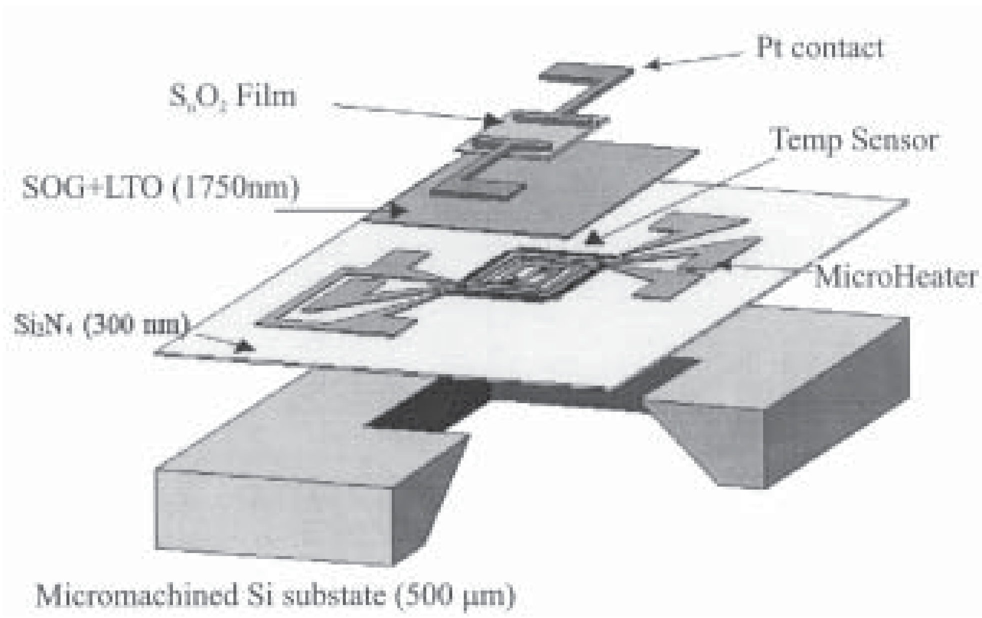

Fig. 2. *Micro-hot plate scheme (NNR-LAMEL, Bologna, Italy.*

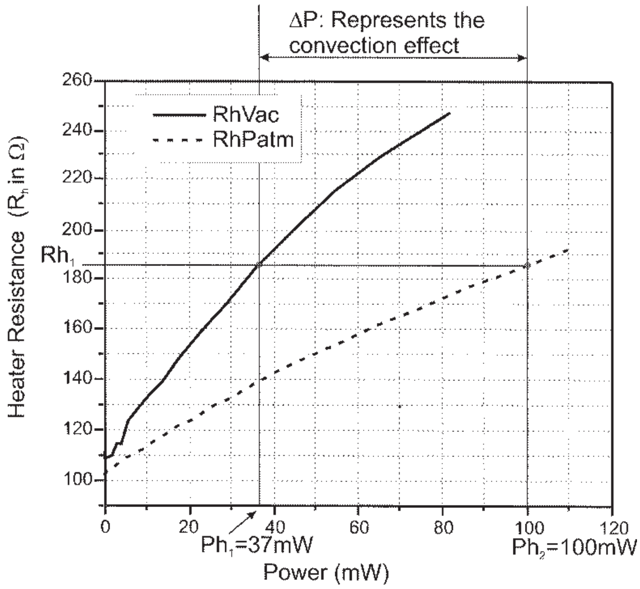

Fig.3. *Comparison of two different operating conditions for a same micro-hot plate.*

Electronic circuits

Control of supplied power to the heater:

The electronic circuit to control the microheater of micro-hot-plate was designed considering the device miniaturization and the necessary conditions to operate in the measurement chamber (air flow: ~0.5 $l.min^{-1}$). The design enabled an important temperature fluctuation on the sensitive film if abrupt changes in the dissipation conditions occurred, mainly due to the following contributions [3]:

- P_c: power transferred by conduction through the membrane to the silicon structure,
- P_k: power transferred by conduction and convection to the environment surrounding the hot membrane,
- P_r: power transferred by radiation.

Consequently, the power supplied to the heating device of a microheater (P_h) can be expressed as the sum of the three above mentioned contributions:

$$P_h = P_c + P_k + P_r$$

In the case of this work, the useful power was part of the dissipated by conduction power used to heat the sensitive film. A plot was done at our laboratory to compare the functioning conditions of a heating device operating in vacuum (10^{-5} Torr) and under normal conditions of pressure (AP: atmospheric pressure). Figure 3 shows the plot of the heating resistance versus the supplied power under the mentioned operating conditions. Measurement data indicated that, in order to reach a temperature of 400°C on the sensitive film, it was necessary to supply 100mW ($P_c + P_k + P_r$) under AP, while in vacuum only 37 mW were required ($P_c + P_r$) for the same temperature. Convection effects are responsible of ~ 60% or more of losses in the heating system at 400°C. This effect decreased if the heating power level decreased. Besides, if measurement data corresponding to two different operating conditions were compared, for example: for a sensor without and with forced convection (~0.5 $l.min^{-1}$), differences of approximately 6% for the same temperature were observed. Resistance is an indicator of the operational temperature of the heating device. Relation between temperature and resistance is not lineal since temperature is not homogeneously distributed on the heater [2]. This fact was proved by microthermographies [2]. If it is considered that temperature on the sensitive film is strongly dependent on thermal consumption of the heater surface, it is then to define operation points for sensors in the system constituted by the measurement chamber, the micropump and the sensors. This point is given by two easily measurable magnitudes: the heater resistance (R_h) and the supplied power to the heater (P_h), this operating point being an important factor to be considered as measurement parameter in PAMPA III. This fact shows the importance given to the temperature control of the heating driver. In Ref. [4] it is strongly pointed out the large influence of the operating temperature on the sensitive film conductivity. The sensing semiconductor exhibits a thermally activated conductivity mechanism; it is reported [6] that a temperature variation of 0.5°C in thick films generates a conductance change of 1%. This fact, also valid for thin films, points out that strict control of temperature variation is of the highest importance to increase the sensors sensitivity.

Consequently, a circuit maintaining a constant heating resistance regardless of variations of thermal solicitation on the heater, was chosen for this work as the heating driver [5]. The circuit consisted of a Wheatstone bridge, in which one of its resistors was the sensor heater (made of Pt and exhibiting a high positive temperature coefficient). The other resistors should be very stable with temperature. The bridge was fed with a Voltage Controlled Current Source (VCCS). The differential voltage between both, opposite bridge nodes was applied to an error/set-point amplifier, from which the controlling voltage to the VCCS was extracted. Then, different current values were supplied to the bridge so as to maintain at zero the error differential voltage, a situation only reached when the bridge was in equilibrium. If the value of heater resistor was modified, as a consequence of thermal solicitation from the measurement chamber, the circuit tends to compensate this effect, increasing or decreasing the power supplied to the heater and trying to maintain a constant heater resistance. This implied that its temperature was maintained also constant because the heater resistance only depended on temperature. The circuit is then called constant resistance driver.

The output of the error/set-point amplifier supplied a voltage proportional to the output differential signal of the Wheatstone bridge (error = $V_{nw} - V_{nl}$) plus a set-point (V_{nw}) obtained by a digital potentiometer placed on one of the bridge arms.

$$V_s = V_{nw} + (V_{nw} - V_{nl})\, R_G / R_A$$

The gain of this amplifier was approximately given by the ratio between the resistances R_G and R_A of the circuit of Figure 4. This digital potentiometer could be commanded by a

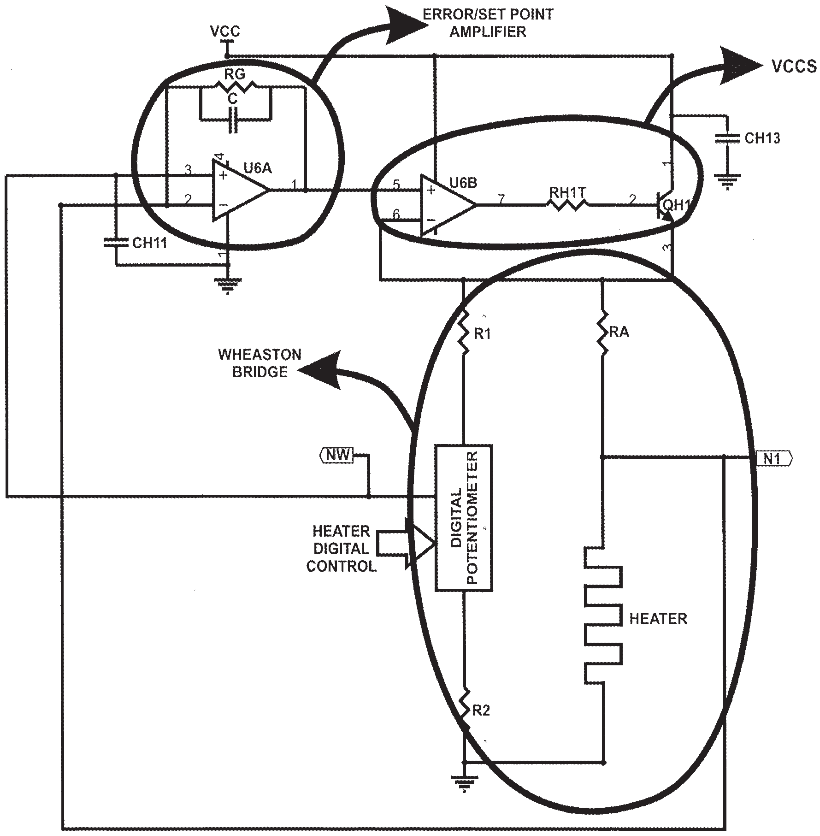

Fig. 4. *Electronic circuit for heating control*

microcontroller or a PC to digitally fix the heating power (maximal 100 mW) chosen to operate the sensor.

Device to condition the signal to the sensitive film:

A metal oxide semiconductive gas sensor is based on changes of its electrical conductivity due to the reaction between the semiconductor and the gases present in the gas chamber [7]. It is interesting to point out that these sensors are unspecific, i.e. they do not sense only one gaseous species but, they are sensitive to several species. For example: non doped SnO_2 sensors built by the RGTO (Rheotaxial Growth and Thermal Oxidation) technique are mainly sensitive to CO, CH_4, NO_2, VOCs, etc. however, they also exhibit a considerable response to humidity. In order to detect only one gaseous species, they are usually connected in arrays of multiple sensors or include filters to avoid the sensor exposition to the interfering species. In general, there are several strategies to diminish the effect of parameters upsetting the sensors response.

In Figure 5**,** it is shown the diagram of the circuit conditioning the signal to the thin film sensor. It is possible to observe that the output signal is proportional to the sensing film conductance. The circuit enables to fix:

I) the bias voltage of the sensitive film and, indirectly, the serial resistance hitched to the same branch where the sensor is connected,

II) the gain in the amplifying stage.

Both fittings are accomplished with digital potentiometers (consequently, to be commanded by a smart circuit, like a microcontroller, a PC, etc.). The observation of Figure 5 enables to deduce the following expression for the output voltage of the circuit, proportional to the sensitive film conductivity:

$$V_f = -V_R \, D(N_B) \frac{R_G(N_G)}{[R_S + R_B(N_B)]}$$

$R_G(N_G)$: *Gain Resistance (function of Digital Control number* N_G)

$D(N_B)$: *Divider Factor (function of Digital Control number* N_B)

$R_B(N_B)$: *Equivalent Resistance of Digital Divider (function of Digital Control number* N_B)

V_R : *Reference Voltage*

This configuration was chosen because of the dispersion of the sensitive films resistance data due to different metal oxides (SnO_2, ZnO, WO_3, etc.) for sensitive films, to different doping elements and to the dispersion of the fabrication processes. The values of films resistance to be connected to this circuit are between 30KΩ and 3MΩ (in air and at low temperature). The described circuits are useful for each sensor and in the PAMPA III the circuitry ended up considerably complex since this e-nose has 12 sensors. Besides, two commercial sensors were included in the measurement chamber: HYH3610 and LM35 to measure humidity and temperature, respectively. The driving circuits for these sensors are not described in this work.

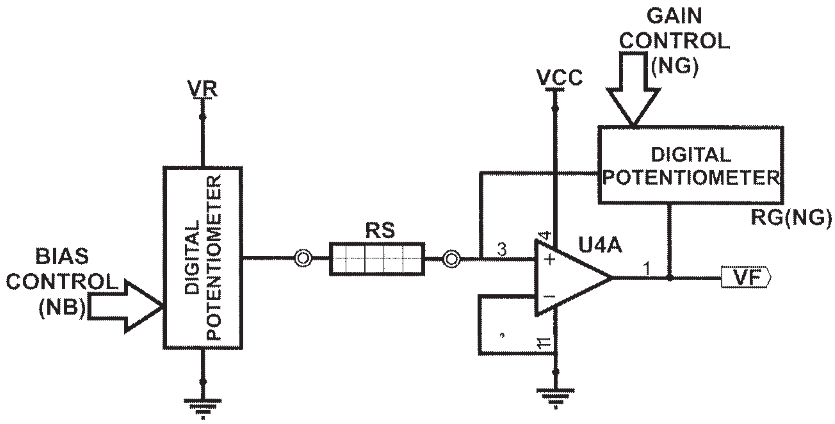

Figure 5: *Electronic circuit for a thin film gas sensor. It includes two potentiometers to control the amplifier gain and the voltage bias.*

Conclusions

The Argentine PAMPA III electronic nose constitutes a useful equipment built through a successful cooperation between the CNEA (Physics Department-MEMS Group) and CITEFA Microelectronics Division, Department of Applied Electronics. The functioning of the described circuit topologies was proved in the engineering e-nose prototype reaching stability in sensors operation and exhibiting a considerable versatility to choose the system measuring format because of the availability of digital controls for each sensor and the possibility of its control with a PC.

Acknowledgements

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