A Fully Integrated Wide Dynamic-Range
Read-Out and Temperature Control Circuit
for Microhotplate Thin Film Gas

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Anno Accademico 2007–2008
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INTRODUCTION

The research activity reported in this thesis has been carried out during the period January 2006 - February 2008 at the Integrated Microsystems Laboratory (IMS), University of Pavia. This activity has been carried out in the framework of the Italian Government PRIN project 2005092937, entitled “Interface and control circuits for high-selectivity gas sensors operated with temperature pattern”. Three research groups have been involved in the realization of this project under the coordination of the Department of Innovation Engineering of University of Lecce, which directed the activity. The Research Center IMM-CNR of Lecce developed the microsensor, the Department of Electronics of University of Rome Tor Vergata studied the algorithms for gas concentrations extraction and the University of Pavia realized the integrated interface circuit for the sensor. The presented manuscript is mainly about the integrated interface circuit, that consists of a temperature control circuit and a wide dynamic range read-out circuit. The micromachined sensor and some algorithms for gas concentration extraction are also presented, since they are useful to understand the requirements of the interface circuit. The design, the implementation and the characterization of the wide dynamic range integrated interface circuit for gas-sensors and in particular of the temperature control circuit will be presented. These circuits have been realized to be compatible with the requirements and demands of the other blocks of the gas-sensing system and, in particular, they match at the input with IMM-CNR sensor specifications and at the output with University of Rome data analysis algorithms. The manuscript is organized as follows.

Chapter 1 is an overview about the aim of the research activity. Gas sensors applications, spacing from food inspection, to environmental air control, to the ratio between air and fuel in automobile engine, are presented. Gas sensor technological trends and working principles are presented and the motivations that push in developing semiconductor gas sensor are explained. State of the art fabrication techniques and materials used are then reported. At the end the actual sensor exploited in the project for the chemical measurements is presented, showing its main specifications, including preliminary chemical measurements.
In Chapter 2 particular focus is given to sensor arrays. At first chemometrics techniques exploiting the cross-correlation between the sensor responses will be presented. The idea on which those techniques are based, i.e. to use linear transformation to determine the inverse calibration function, is reported and compared to the electronic nose approach based on artificial neural network. Indeed electronic noses recognize gas components and concentrations exploiting a biologically inspired neural architecture, based upon human learning. Latest research results in pattern recognition algorithms for odor recognition and gas classification are reported and different approaches are quickly explained.

Chapter 3 describes the integrated read-out circuit for thin film gas-sensors. At the beginning of the chapter possible solutions are presented, in order to show several different approaches to the read-out task and, then, the chosen solution is reported and motivated. Since the main target application is indoor environmental gas sensing, considering the dynamic range required in resistive sensor measurement, a calibration free circuit has been developed. The sensor is biased with a constant buffered reference voltage and the current flowing through the sensor is read exploiting the current to frequency conversion technique. An internal ad-hoc frequency-meter completes the analog to digital conversion.

Chapter 4 illustrates the temperature control circuit. It begins reminding the importance of the effectiveness of the temperature control in metal oxide sensors, and presents a quick overview of most common control techniques. Some possible solutions are then illustrated and the chapter ends reporting the block diagram of the implemented temperature control circuit.

Chapter 5 presents in detail the most significant parts of the design of the interface circuit. Then some layout details are reported. It was crucial in order to obtain good performance to provide enough insulation between the power and digital circuits with respect to the analog parts of the circuit, in particular the precision read-out circuit. Both the electronic and chemical measurement set-up are shown, focusing also on the user interface that drives the chip. Electrical silicon testing results and chemical measurements are then presented.
Finally, brief conclusion recalls the outline of the thesis and summarizes the measurements obtained with the developed integrated circuit.
Chapter 1

Gas-sensors

1.1. Introduction

This chapter introduces the objectives of the research activity reported in this thesis. At first we will present the aim of the research activity and why the research in this area is useful, presenting some gas-sensors applications. Then, the gas-sensors development trend that leads to semiconductor gas-sensors devices and their detection principles will be presented. State of the art techniques for gas-sensors, with particular focus on Tin Oxide sensors, including micromachining and an introduction to the most used doping elements, will be exposed. The chapter will be concluded presenting the gas-sensors exploited in this work.
1.2. Aim of the research activity

Target of the research project described in this thesis is the development of the fundamental blocks necessary for the realization of a high-selectivity gas detection system. It has to be based on the use of semiconductor chemical sensors and the high selectivity has to be obtained by using an array of gas sensors working with modulated temperature. In particular, the research target will be the development of a gas detector for methane (CH₄), carbon monoxide (CO) and some of their interfering species, as for example ethanol (C₂H₅OH) and water steam (H₂O), for domestic applications. The system will consists of a certain number of semiconductor sensing devices, arranged in array, by an A/D multichannel integrated circuit including a dedicated conditioning network for the read-out of the data coming from the sensors and a system of digital processing of the acquired data. In order to increase the selectivity, a temperature controller for each sensor is necessary to implement the chosen temperature profile. This manuscript mainly focuses on this temperature controller. It has been realized as a closed loop circuit driven by a 9 bit digital set-point that allows the desired temperature pattern to be easily obtained, with sufficient accuracy, exploiting two platinum-titanium embedded resistors (a heater and a thermometer). The reported system design faces the following aspects among the most critical ones in the development and realization of a complete portable gas sensing system:

- realization of a family of miniaturized semiconductor gas-sensors manufactured with sol-gel technique having sufficient stability over time, good sensitivity, moderated noise and, as a consequence, high resolution;
- development of a smart mixed-signal A/D front-end circuit with a very large dynamic range, able to read-out different gas-sensor types, taking into account also unavoidable technological spreads;
- design of an integrated temperature controller able to regulate the sensor temperature with suitable accuracy;
• integrate on the same chip a high precision circuit (the A/D front-end circuit) and a power circuit (the temperature controller), limiting the disturbances on the first deriving from the latter;

• development of novel algorithms of pattern recognition for the processing of the acquired data.

The research activity has been assigned to three groups, which operated in parallel but in strong cooperation, as described in the following. A first group has developed various semiconductor gas-sensors. These demonstrated to detect gases like CO, CH₄, NOₓ, and O₃, which are critical in ambient monitoring applications. These sensors have been realized and fully characterized for what concerns their functional and electrical performance, with particular attention to the drifts of the characteristics over the time and to the noise. Such sensors have then been connected to the developed electronic front-end circuit in strong cooperation with a second group. The activity performed by this group has been the electronic front-end circuit which processes the signals coming from the gas-sensors and the development of the temperature controller. A third team has been assigned to the development of novel pattern recognition algorithms to elaborate the digital data supplied by the A/D interface. Feature extraction, beside the not already trivial steady-state response, takes also into consideration the behaviour over time of the sensor signals. The extraction of further and advanced features has then been considered as a possibility to improve the information content extracted from each single sensor. Concerning the data analysis, this has been mostly focused on the study of sensor arrays and on the possibility to determine both qualitative and quantitative characteristics of analysed samples. Beside the use of standard chemometrics techniques, such as principal component analysis and partial least squares, and neural networks, like radial basis functions and self organising maps, the research went also in the direction of constructing hybrid models. This additional approach allowed also the optimisation of quantitative models through the definition of partial models, each valid in a specific sub-manifold of the sensor space, determined through a qualitative analysis that may be either supervised or unsupervised. Furthermore, a certain emphasis has
been given on the possible discrimination, at data analysis level, of spurious signal sources not correlated with the chemical information characterising the sample. For this scope the analysis of independent components has been considered. Algorithms have been developed via software and a certain attention has been paid to the complexity control in order to give the possibility of an eventual hardware implementation either in an integrated circuit or in a microcontroller, like FPGA through VHDL platform. A validation of the algorithms through their implementation into integrated circuit is not fundamental for two reasons:

- the integration should not be critical due to the very low system throughput operating frequency;
- the comparison between the developed algorithms should be easier if these are implemented via software.

The peculiarity of realizing a miniaturized system can then give the advantage of connecting the developed system with a wireless transmission system (Bluetooth, for instance), in order to create a distributed remotely controllable net of gas-sensors of several kinds, each one with optimized performances.

### 1.3. Gas-sensors applications

A gas-sensor is the main block of a more complex system composed also by an electronic interface and conditioning circuit and by a data analyzer. A gas-sensor is a device that changes at least one electrical parameter in relation to the gas composition at which it is exposed. The first purpose of the electronic interface and conditioning network is to create the physical conditions, usually in terms of a certain temperature and of a reference voltage or current, to allow the sensor to work. The second aim of the electronic interface is to acquire the electrical parameter variation of the sensor. The data analyzer that completes the system discriminates the different gases. One of the most interesting application fields for gas-sensors is the environmental monitoring. It includes the analysis and the identification of household odors,
air-quality monitoring of factory emissions, work environment and air-quality monitoring in general [1]. The analysis of gaseous environments is not the only task that requires a gas-sensor. Another important application is the measurement of oxygen partial pressure, as required in combustion control systems, in the feedback control of the air/fuel ratio of automobile engine exhaust gases, in order to improve the fuel economy efficiency and reduce the harmful emission of gases as CO, NOx, and hydrocarbons. Anyway, nowadays, the largest market for gas-sensors, embedded in systems called electronic noses, is the food industry. Applications of electronic noses in the food industry include: quality assessment in food production, inspection of food quality by odor, control of food-cooking processes, inspection of fish, monitoring the fermentation process, checking the rancidity of mayonnaise, verifying if orange juice is natural, monitoring food and beverage odors, grading whiskey, and inspection of beverage containers, often avoiding human operator exposure to dangerous environments. In fact, in some instances, electronic noses can be used to augment or replace panels of humans experts. In other cases, electronic noses can be used to reduce the amount of analytical chemistry that is performed in food production, especially when only qualitative results are needed. Moreover, the European community has defined strict standards for food quality and significant efforts have been devoted to the development of new techniques that could complement the traditional sensorial and analytical analysis of foodstuffs. The attention has been turned to electronic noses which, in mimicking the human nose, offer an objective way of detecting aromatic fingerprints. Olive oil was the first foodstuff to be classified by both chemical and sensorial analysis according to the EU Normative. This means that an olive oil can be labeled as extra virgin only if both its chemical and sensorial characteristics are within certain standards established by law. Thus, the market requires urgently reproducible, reliable, inexpensive, easy to train and to use, objective “sniffing” electronic devices dedicated to a large variety of goods, mainly for classification and degradation. Moreover, because the sense of smell is an important sense to the physician, an electronic nose also has applicability as a diagnostic tool. An electronic nose may in fact examine odors from the body
(e.g., breath, wounds) and identify possible problems. Odors in the breath can be indicative of gastrointestinal problems, sinus problems, infections, diabetes, liver, and even cancer problems. The list of examples reported in the above paragraph is only a subset of the possible applications of smelling and gas-sensing devices, which are the main application of the circuits reported in this work.

**1.4. Gas-sensors trend**

Analytical techniques as gas chromatography (GC) and mass spectroscopy (MS), which are complex, expensive and difficult to operate are not suitable for a portable cheap gas sensing device. In addition, most of the analyses require sample preparation, so on-line and real-time analyses are difficult. The interest of the industrial and scientific world in gas-sensors in mainly focused on solid semiconductor gas-sensors, because of their numerous advantages, like small size, high sensitivity also while detecting very low concentrations (at levels of ppm or even hundreds of ppb) of a wide range of gaseous chemical compounds, the possibility of on-line operation and, due to possible batch production, low cost. Solid-state gas-sensors are the best candidates for the development of commercial gas-sensors for a wide range of applications. In contrast to optical processes, which employ infra-red absorption of gases, chemical processes, which detect the gas by means of a selective chemical reaction with a reagent, mainly utilize solid-state chemical detection principles. Solid-state chemical sensors in general have been widely used, but they suffer from limited measurement accuracy and problems of long-time stability. However, recent advances in nanotechnology, i.e. in the cluster of technologies related to the synthesis of materials with new properties by means of the controlled manipulation of their microstructure on a nanometer scale, produce novel classes of nanostructured materials with enhanced gas sensing properties, providing the opportunity to dramatically increase the performance of real solid-state gas-sensors. A characteristic of solid state gas-sensors is the reversible interaction of the gas with the surface of a solid-state material. The conductivity change of a
gas-sensing material is the main way to detect a reaction, but it can be revealed also by measuring the change of capacitance, work function, mass, optical characteristics or reaction energy released by the gas/solid interaction. Organic (as conducting polymers [2], porphyrins and phthalocyanines [3]) or inorganic (as semiconducting metal oxides [4, 5]) materials, deposited in the form of thick or thin films, are used as active layers in such gas sensing devices. The read-out of the measured value is performed via electrodes, diode arrangements, transistors, surface wave components, thickness-mode transducers or optical arrangements. Indeed, although the basic principles behind solid-state gas-sensors are similar for all the devices, a multitude of different technologies have been developed and nowadays the number of different solid-state based gas-sensors is very large. Solid state sensors depend strongly on the development of technologies mainly driven by other than sensor applications. A steering technology for chemical sensors, which has led to the development of gas-sensor devices with small power consumption and short time constants both for temperature regulation and response, greater portability and easy integration with electronics, is the micromachining technology.

1.5. Semiconductor gas-sensors

Semiconductor gas-sensors (SGS), known also as chemoresistive gas-sensors, are typically based on metal oxides (e.g. SnO₂, TiO₂, In₂O₃, WO₃, NiO, etc.). The gas-sensing mechanisms of SGS are gas/semiconductor surface interactions that occur at the grain boundaries of the polycrystalline oxide film. The interactions generally include reduction/oxidation processes of the semiconductor, adsorption of the chemical species directly on the semiconductor and/or adsorption by reaction with surface states associated with pre-adsorbed ambient oxygen, electronic transfer of delocalized conduction-band electrons to localized surface states and vice versa, catalytic effects and in general complex surface chemical reactions between the different adsorbed chemical species. These surface phenomena produce a reversible and significant change in electrical resistance (i.e. a resistance increase or decrease
under exposure to oxidizing or reducing gases, respectively, referring as example to an n-type semiconductor oxide). This resistance variation can be used to detect chemical species in the ambient. The changes in the electrical resistance of the sensor can be described by the formation of depletion space-charge layers at the surface and around the grains, with upwards bending of the energy bands [5]. Surface energy barriers for conduction electrons, whose height and width are variable, depend on the occupancy of surface states related to adsorbed species. The gas/solid interactions exploited in SGS can involve changes in the bulk conductance or changes in the surface conductance; correspondingly two principal types of metal-oxide based gas-sensors can be distinguished [6, 7]. The first kind of mechanism exploits the fact that a semiconductor oxide is in general non-stoichiometric and the oxygen vacancies are the main bulk defects. At high temperatures (600-1000 °C) the oxygen vacancies can quickly diffuse from the interior of the grains to the surface and vice versa and the bulk of the oxide has to reach an equilibrium state with ambient oxygen. The main application of this first kind of sensors is the measurement of oxygen partial pressure as required in combustion control systems, in particular in the feedback control of the air/fuel ratio of automobile engine exhaust gases near the λ point, in order to improve the fuel economy efficiency and to reduce the harmful emission of gases as CO, NOx and hydrocarbons [8, 9]. The second principal type of semiconductors gas-sensors is based only on changes in surface conductivity at lower temperatures, depending on the specific target gas in the ambient and on the selected sensor material in conjunction with its properties, usually in the range 200-400 °C, and at quasi-constant oxygen partial pressure. In this condition the sensor detects small concentrations of reactive gases in air by a displacement from the constant oxygen pressure equilibrium state, induced by gas interfering effects at the surface of the sensor. Here we will discuss about this latter class of resistive-type gas-sensors. A simple SGS is thus basically composed of a substrate in alumina or silicon (on which the sensing layer is deposited), the electrodes (to measure the resistance changes of the sensing film) and the heater (commonly a Pt resistive type heater) to reach the optimum sensing temperature. Semiconductor gas-sensors offer low cost, high
sensitivity, and a real simplicity in operation. The possibility of combining in the same device the functions of sensitive element, signal converter and control electronics constitutes the main advantage of chemoresistive-type sensors over biochemical, optical, acoustic, and other gas sensing devices.

1.5.1 Analysis of gas detection limits

The sensor response is defined as:

\[ X = \begin{cases} 
\frac{X_S}{X_0}, & \text{if } X_S > X_0 \\
\frac{X_0}{X_S}, & \text{if } X_S < X_0 
\end{cases} \]  

(1.1)

where \( X_S \) and \( X_0 \) are the electrical signal values at equilibrium in the presence of a gas/odor and in a reference gas, respectively, depending on the reducing or oxidizing character of the chemical species interacting with the sensor surface. A general trend, which has been experimentally verified, is that the sensor response to a particular gas increases up to a maximum corresponding to an optimum working temperature, then falls off toward zero at higher temperatures. In spite of the numerous advantages of resistive-type gas-sensors, they show different disadvantages, such as poor reproducibility and selectivity and long-time instability due to aging. There are in fact two undesired aging effects that may appear when the sensor works for a long period: a drift of the baseline signal defined as the conductance in air or in a reference gas (offset drift) or a drift in the sensor sensitivity (gain drift). Long-time instabilities are of considerable importance for the practical use of the sensor; pre-ageing thermal treatments and cycle calibration checks have to be carried out in order to avoid incorrect use of the device. The causes of instabilities are mainly microstructural and morphological changes (change in size, number, and distribution of grains and intergranular boundaries) of the sensing elements, but also irreversible reactions with chemical species in the ambient, modifications of the sensor heating element, or of the electrodes have to be taken into account. Metal-oxide-based gas-sensors are normally sensitive to more than one chemical species in air, and usually show cross
sensitivities. Moreover metal oxides are nonlinear sensors; the change in sensor response due to a defined change in gas concentration depends generally on the concentration of the gas to be monitored \( y_k \), and also on the concentration of other gases \( y_j \neq k \) (cross-sensitivity effect). In this case, more than one sensor or more than one operation mode of the same sensor are required to determine the concentration level \( y_k \). The (partial) sensitivity \( S \) describes the change in the sensor response due to a specific change in the stimulus. In particular, if \( y_k \) is the concentration of the chemical species \( k \), the (partial) sensitivity \( S_k \) of the sensor to detect the species \( k \) is defined by the slope of the sensor calibration curve at concentration \( y_{k_0} \):

\[
S_k = \left( \frac{\partial X}{\partial y_k} \right)_{y_k = y_{k_0}, y_j \neq k \text{ const.}}
\]

Another important disadvantage is the sensitivity to water. The sensitivity to different species in air so far considered an intrinsic property of metaloxide-based gas-sensors. When different reactive gases are present simultaneously in the same atmosphere interference effects between them can occur. Unselectivity cannot be eliminated completely, but selectivity can be improved in different ways, such as:

- the use of filters [10, 11];
- the use of chromatographic columns to discriminate between gases on the basis of their molecular size or other physical properties [12];
- the use of catalysts and promoters or more specific surface additives [13, 14];
- the selection of the material for the sensing layer and its physical preparation [15, 16];
- the analysis of the transient sensor response [17];
• the selection of a fixed temperature to maximize sensitivity to a particular analyte gas [18];

• the use of a temperature-modulated operation mode [19, 20].

A different approach to the problem of unselectivity is based on the development of an electronic nose, which consists of an array of different sensing elements with partially overlapping response features and a pattern recognition system [21]. Basically, the idea of an electronic nose is to exploit the unavoidable cross sensitivity of the sensors instead of trying to eliminate it. Linking the sensors in an array configuration and analyzing the responses of the sensors in a subsequent data-processing step allows us to perform a qualitative and/or quantitative analysis of the ambient under examination. Sensitivity, selectivity, and stability are the three principal parameters that have to be improved in order to develop more accurate devices.

1.5.2 Gas-sensor fabrication techniques

Semiconductor gas-sensors (SGS) can be divided into two classes: thick-film or thin-film SGS. Thick-film sensors are rather thick (a few to several hundred micrometers) and porous, thus a high active surface area is exposed to the gas interactions. Thin-film sensors, by contrast, can be prepared by established preparation methods mutated from semiconductor technology, with a typical thickness in the range of a few to several hundred nanometers. In this case, gas interactions are mainly restricted to the surface layer. Thick-film metal-oxide-based sensors are typically more responsive than thin-film sensors. Currently, SnO₂ is the most used sensitive layers for both thin and thick film sensors. SnO₂ is usually prepared starting from commercial tin oxide, tin, tin salt (mostly SnCl₄), or organo-tin compounds (mostly tin alkoxides). The starting materials are used to prepare powders, colloid solutions (gels), or sprayable solutions. The sensitive layer is then fabricated based on these mixtures. The often used term sol-gel is referred to this
special preparation route, an aqueous tin salt solution forms a sol with poly SnO₂ clusters. They can be precipitated by basic solutions, or they may undergo a gelation step when clusters grow, but still dissolve as a colloid. A critical step in this process is the calcinations of the powders because this will define the grain size of the material. Different methods are used to deposit the powders or solutions onto electrode-equipped substrates with a heater at the back side. The most common methods are dipping, painting, screen printing, and drop coating for the deposition of thick films, and spraying and spin coating for the deposition of thin films. These methods use a thermal treatment after deposition to create the sensitive layer. The sensors can be optimized by chemically modifying the SnO₂ material by adding dopants, as will be shown in the following sub-section. Various methods have been developed for doping. These methods can be divided into two groups; in the first, a compound containing the dopant is added to the precursor; in the second, the SnO₂ powders are treated with such compound. The sol-gel technique is a wet chemical deposition method that is well suited for the preparation both of thick and thin films. It offers several advantages with respect to conventional processing technologies:

- it is simple;
- it is inexpensive;
- it is easy to control the morphology and to modify the composition with uniformly dispersed dopants and modifiers;
- it produces films with a high porosity and a large surface area that improves the efficiency of the sensors in the mechanism dominated by surface phenomena.

Nowadays, the sol–gel method is mostly applied for the preparation of thick films through a dip-coated or drop-coated deposition procedure of the base material paste onto the transducer structure. However, great interest is devoted to the preparation of sol-gel metal-oxide thin films because thin-film devices are perfectly compatible with silicon technology and Si-micromachined heater substrates. Most of the commercially available gas-sensors based on thick-film metal oxides use ceramic heater substrates.
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(mainly alumina substrates). A common defect of such thick-film is the high level of heating power consumption (up to 1 W). This level can be reduced by about one order of magnitude using Si-micromachined heater substrates. A complicating feature in employing such miniaturization techniques, however, is that these are not compatible with standard screenprinting technologies, and hence, it is difficult to integrate miniaturized low-power-consumption devices with product-oriented thick-film sensing layers. In this context, novel approaches such as drop coating have been proven to be fruitful and advantageous thick-film deposition techniques for the coating of Si microhotplates [22-25]. The lower heating power consumption and the availability of the silicon semiconductor technology for the development of gas-sensor micromachined substrates push in the direction of thin film sensors. Additionally, since the electrical sensor signals have to be acquired, amplified, and evaluated, the sensor technology has to be compatible with modern electronics, like CMOS. Thus, another cost reduction may be achieved by the integration of the sensing part and electronic circuitry on the same chip, and this provides a second argument for fabrication by microelectronic technologies. In this context, micromachined gas-sensing devices are acquiring an ever-growing importance in the field of gas-sensors [26-29]. The heart of this structure is the active area that consists of the heater, the sensor electrodes, and the gas-sensitive layer situated at the center of a thin membrane which is supported by an outer frame of silicon. This resistively heated dielectric membrane (a–SiON or LPCVD Si$_3$N$_4$) provides the thermal insulation between the active area heated up to high temperatures and the silicon frame that remains at almost room temperature. The attractive features and advantages offered by using such membranes in gas-sensing devices are numerous. They can be handled easily in the electronic industry, and enable integration with electronics; in particular, they need a voltage source both for resistance measurement and for the necessary heater compatible with standard digital electronics (e.g., CMOS). Furthermore they allow a low power consumption not exceeding 100 mW for operation. This small amount of heating power is caused by the reduction in the heated surface area, as well as by the excellent thermal isolation provided by the thin
dielectric membranes. They allow the production of tiny sensor devices of small size and low weight (portability). They allow the reduction of manufacturing costs. They can be easily grouped into battery-operated arrays (i.e., sensor arrays integrated on a sensing chip).

1.5.3 **Gas-sensor dopants**

Noble metals (e.g., Ag, Pd, Pt, etc.), uniformly distributed and finely dispersed as catalytic clusters on the surface of the oxide-sensing layer or added as dopants into the oxide bulk, are often used in semiconductor gas-sensors. It is well known and experimentally proven that catalysts added to the sensor may influence its response to specific gases, may speed up the surface reaction, or may impart selectivity to the reaction so that some reaction processes are favoured over others [30-33]. Because semiconductor gas-sensors depend on surface chemical reactions, they are strongly influenced by the activation energy needed to initiate the reaction. The activation of a species involved in a surface reaction may be the dissociation of a molecule, the ionization of the species, or some other intermediate reaction that presents the species in a form ready for an exothermic reaction on the sensor surface. The activation energy necessary to promote a reaction, usually provided as thermal energy, depends on the route of the reaction. If the activation energy using a particular route is high, the rate of the reaction will be low, and vice versa, if a route for the reaction can be found with a low activation energy, the reaction will be fast. Energetically, the effect of catalysis is to provide a more favourable reaction path. In such a way, a lower operating temperature for the sensor may be needed, and the rate of only some reactions also can be greatly enhanced in contrast to the rate of others, so that selectivity is provided to the system. Thus, the use of catalysts/dopants is a feature of great importance in semiconductor sensor design. Platinum and palladium are the most commonly used dopants to improve the performance of SnO$_2$ for CO detection; it seems that Pt promotes CO oxidation via the spillover mechanism, while it seems that Pd acts via electronic interaction (Fermi-level control) [32, 34].
1.6. Exploited gas-sensors

The gas-sensor used in this project consist of a micromachined sol-gel tin-oxide sensor (1.1 mm × 1.1 mm membrane) with a 450 nm thick silicon nitride/oxide membrane and an active area of 170 µm × 170 µm. Heater/thermometer interdigitated electrodes were realized on the same device layer using a front-side approach that simplifies the production process. The SnO₂ layer was realized with the sol-gel technique and deposited by spin coating on silicon micromachined substrates.

![Figure 1.1: Pictures of the developed gas-sensors.](image)

The heater and thermometer resistance are realized in Ti-Pt. Their value at room temperature is respectively 25 and 75 Ω approximately. Their values vary with the temperature as indicated by the following expression:

\[ R_{T_A} = R_{T_0} \times [1 + \alpha \times (T_A - T_0)] \]

(1.3)

where:
- \( \alpha \) is the thermal coefficient, \( \alpha=0.0022 \, ^\circ C^{-1} \);
- \( T_0 \) is the room temperature, \( T_0=25 \, ^\circ C \);
- \( T_A \) is the working temperature, comprise between 200 °C and 400 °C;
- \( R_{T_0} \) is the value of the resistance at room temperature;
- \( R_{T_A} \) is the value of the resistance at the temperature \( T_A \).

The sensor resistance may vary with gas more than two decades.
Measurements confirmed the effectiveness of the sensors. Figure 1.2 shows the system transient response varying CH$_4$ concentration in quite dry air (RH≈30%) from 500 ppmvol to 1000 ppmvol, while Figure 1.3 represents the system characterization with respect to methanol, varying methanol concentration between 4.2% and 12.5%.

Figure 1.2: System transient response to CH$_4$ concentration in wet air.

Figure 1.3: System transient response to methanol.
Figure 1.4 shows the system transient response to NO$_2$ in wet air from 10 ppmvol to 30 ppmvol, RH=30%.

![System transient response to NO$_2$ in wet air](image)

Figure 1.4: System transient response to NO$_2$ in wet air.

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2.1 Introduction

This chapter introduces the state of the art gas-sensor array data analysis, in particular electronic noses. At first we discuss why electronic noses are so widely used and how an electronic nose works, through the description of major pattern recognition techniques. Even if larger space has been allocated for describing basic static techniques, also important dynamic approaches are reported.

2.2 Electronic noses

An electronic nose [1, 2] incorporates an array of chemical sensors. The term “array” is not intended as a particular spatial arrangement of the sensors in the device but in the sense of an organization of the output of the single sensors under exposure to the same environment. In this way the contribution of each array element is a component
of a multidimensional general problem. In these systems a single sensor in the array may also not be highly specific and selective in its responses, but could respond to a broad range of compounds, such that different patterns are expected to be related to different odours. The response of an array composed by non-ideal gas sensors with partially overlapping features can be improved by elaborating the data from the sensors together. A typical gas sensor response is shown in Figure 2.1, ideal case, where the sensor is exposed to a certain odorant \( j \) with a certain concentration \( c_j(t) \). Usually the rise time, reported as “\( \tau_r \)” in the figure and the decay time “\( \tau_d \)” are different. However, the output signal \( R_{ij}(t) \) is subjected to divergence from the ideal case by interfering signals. There are several interfering inputs, the most common being changes in temperature and relative humidity. Usually in most state of the art systems, the heater of a chemoresistor is maintained at constant voltage in open loop control, then in reality the operating temperature varies due to any changes in ambient temperature. Humidity has also a strong effect on most sensors. The baseline resistance usually decreases as the humidity increases, although the exact slope depends on the operating temperature of sensors. One of the important characteristics of an array of chemical sensors is again its long-term stability. The stability is considered as the amount of variation of the steady-state response that is represented by \( R_S \) and the baseline \( R_0 \). The baseline is referred to the value of the reference gas injected into the a test chamber before injecting the sample odorant, for which the response changes until reaching the saturation value of \( R_S \). Seasonal and environmental drifts occur in the behaviour of some odour sensors. Drift is a dynamic process, caused by physical changes in the sensors and the chemical background, which gives an unstable signal over the time. It is in general a slow change in sensitivity and baseline that occurs in time due to ageing, slow morphological aspects of sensor material, poisoning and other reasons. Sensor poisoning occurs when the sensor is exposed to a chemical that irreversibly binds the sensing surface leading to a reduction or the total loss of sensitivity. An example of drift is shown in Figure 2.2 where a sensor is exposed to the same odorant over time. Drift could be both reversible, e.g. condensation of vapour on the sensors, and irreversible, e.g. ageing
that causes the pattern recognition system to be very short-lived. Memory effect could be considered similar to drift effect; which means that a measurement at time \( t \) is highly influenced by measurements at time \( t-k \). This leads to the fact that the same gas mixture will not give a well defined pattern. If no drift correction of the sensor signals is made, the model will have a continuous need for re-calibration. Since the training phase of the pattern recognition model should reflect the variance of the populations, many samples are necessary. In real application processes, the samples may be very expensive to acquire, which makes it impossible to re-calibrate the pattern recognition model very often.

Figure 2.1: Typical sensor response to ideal gas concentration stimulus.
2.3 Chemometrics based technique

The signals of a sensor array can be evaluated by means of pattern recognition (PARC) and multicomponent analysis (MCA) methods which, exploiting the cross correlations between the sensor responses, extract information contained in the sensor-output ensemble [3-7]. In particular, a PARC procedure performs a qualitative analysis of the environment; it evaluates the multidimensional dataset from a gas-sensor array, seeking the underlying main relationships in the dataset itself in order to analyze the data structure and discriminate between different data classes (clusters) belonging to different “chemical patterns” (classification). A PARC procedure has to assign a sensor array output from an unknown gas/odor to a class, recognizing in such a way the occurrence of a particular chemical pattern.
(identification). An MCA procedure, based on multivariate techniques of regression, performs a quantitative analysis of the environment, determining the concentration of one or more compounds in a mixture. These analytical regression methods provide a general correlation between the analytical information (composition of the chemical pattern) and the output of the sensor array. Of course, such multisensor systems need calibration; by presenting many different chemicals to the sensor array, a database of patterns is built up. This database of labelled patterns is used to train the pattern recognition system. The goal of this training process is to configure the recognition system so as to classify and eventually quantify each chemical compound in a gas mixture. Data processing techniques adapted to the data analysis for an electronic nose have a long history, and in parallel with the first well established approach provided by chemometrics, a new approach based on artificial neural networks (ANNs) is also available in literature [5]. Chemometrics, which has been in existence since the 1960s, is the chemical discipline, that uses mathematical and statistical methods to provide the maximum chemical information by analyzing chemical data. These chemometric techniques, applied to data from a sensor array, seek to determine the distribution of the data and the underlying relationships between one set of independent variables (i.e., the preprocessed sensor responses from a sensor array) and another set of dependent variables (i.e., the gases/odors classes or the component concentrations). This statistical approach is sometimes referred to as parametric since it assumes that the data can be described by a probability density function, such as the multinormal distribution. In chemometric techniques, the correlation between the measuring data and the composition or concentrations of the analytes in the chemical pattern during the calibration procedure may be described by a calibration function using an exactly defined set of parameters. These parameters normally are related to physical variables (such as partial sensitivity, i.e. local slope of the calibration function). Most chemometric techniques use linear transformations so that, by explicitly determining the inverse calibration function, it is possible to calculate the concentrations of unknown samples from the measured sensor signals.
2.4 **Modern electronic noses**

The second approach to data analysis in the electronic noses, based on artificial neural networks, is a relatively new one, that arose in the 1980s. It seeks to solve multivariate problems in a manner similar to the human cognitive process by using a biologically inspired neural architecture and rules based upon human reasoning. This approach is also referred to as nonparametric, because it uses an implicit representation of the correlation sensor signal/analyte concentrations without a definition of parameters which can be identified as physical variables. For both the chemometrics-based and ANN-based approaches, pattern recognition for qualitative analysis and the multicomponent analysis techniques for quantitative analysis are available [7]. An important distinction in data analysis techniques concerns their unsupervised or supervised character. Unsupervised methods make no a priori assumption about the sample classes, but they try to discriminate between clusters of unknown gases/odors by enhancing the differences between their associated input vectors; on the contrary, the task of a supervised technique is to classify unknown gases/odors as known ones that have been learned during an earlier calibration procedure. Numerous data analysis techniques are commonly employed to analyze data from electronic nose instrumentation. Multiple linear regression (MLR), principal component regression (PCR), and partial least squares (PLS) are quantitative data processing techniques which are well known in analytical chemistry and in the sensor array area, while the most used conventional statistical pattern analysis techniques are principal component analysis (PCA), cluster analysis (CA), and discriminant function analysis (DCA). Among the neural-network-based techniques, the most important ones are back propagation (BP), radial basis function (RBF), Kohonen self-organizing map (SOM), and learning vector quantization (LVQ). Moreover, there are other more recent dynamical methods, that seek to improve the performance of the pattern recognition method through the use of the dynamic (transient) response of the sensors, or through the use of models that evolve with time. The latter type of technique is known as adaptive, and deals with non-
stationary sources of data, for example, a sensor output that, as mentioned, may systematically age or become poisoned with time. Adaptive techniques are not well established, but currently are an important topic of research; we mention among them the methods based on genetic algorithms (GAs) or the adaptive resonance theory (ART). Finally, we mention the fuzzy neural networks (FNNs) that apply fuzzy logic to electronic nose data.

2.4.1 The basic Self Organizing Map algorithm

The principal goal of the self-organizing map (SOM) [8] is to transform an incoming signal pattern of arbitrary dimension into a one or two-dimensional discrete maps and to perform this transformation adaptively in a topological ordered manner. The embedded competition paradigm for data clustering is done by imposing neighbourhood constraint on the output units, such that a certain topological property in the input data is reflected in the output unitary weight. Figure 2.3 shows a two-dimensional self-organizing map. The Euclidean distance is considered as a measure of similarity and the winning neuron is the one with the largest activation, i.e. the lowest distance. The updating procedure takes into account the neighbourhood function, in order to self-organize the network around the winner. Inter-neuron connections assure lateral plasticity to preserve the topology of the map, which for simplicity has been reported rectangular. The Kohonen network translates the neurons toward the input probability distribution. Input neurons are represented by the abstracted features which are mapped onto a lower dimensional space represented by the output neurons. In a first stage the net is trained with a few objects by presenting each abstracted feature and selecting the winner neuron. This is actually made to roughly approximate the input probability distribution of the feature vectors. Then the winner neighbourhood is adapted in order to move each neuron, weighted with a Gaussian function, toward the input vector.
Figure 2.3: Kohonen model or transform of a two-dimensional lattice of neurons.

The algorithm may be summarized as follows:

- initialize randomly all the codebook vectors $w_i$ with $i=1, \ldots, N$ where $N$ is the size of the full map;
- once given an input vector $x$, find the winner $c$ which satisfies:
  $$\| x - w_c \| = \min_i \{ \| x - w_i \| \} \quad (2.1)$$
- adapt the neurons as follows:
  $$w_i(t+1) = w_i(t) + h_{ci}(t)[x(t) - w_i(t)] \quad (2.2)$$

where $h_{ci}(t)$ is what we call *neighbourhood function* or also *kernel*, which plays a central role in the relaxation process. For convergence it is necessary that $h_{ci}(t) \to 0$ when $t \to \infty$. Actually, it is a function defined over the lattice point, defined as:

$$h_{ij}(t) = \alpha(t) \cdot e^{\frac{||r_c-r_i||^2}{2\sigma^2(t)}} \quad (2.3)$$

where $(r_c, r_i) \in \mathbb{R}^2$ defines the position in the grid of the winning neuron $c$ and a neighbouring neuron $i$ respectively, $0<\alpha(t)<1$ is the learning rate parameter and $\sigma(t)$ is the width of the kernel.
With increasing $||r_c-r_i||$, follows that $h_{c,i}(t)\to 0$. The average width and shape of $h_{c,i}$ together define the *stiffness* of the elastic surface to be fit to the data points. Let us define with $N_c(t)$ the neighbourhood set of array points around node $c$ at time $t$. Assuming a rectangular topology of the network, Figure 2.4 shows three successive steps of the neighbourhood set. We thus have that $h_{c,i}(t)=\alpha(t)$ if $i\in N_c(t)$ and $h_{c,i}(t)=0$ elsewhere. Both $\alpha(t)$ and the radius or width $\sigma(t)$ of $h_{c,i}(t)$ are usually decreasing monotonically over time during the ordering process.

### 2.4.2 The multiple Self Organizing Map algorithm

A labelling phase follows the unsupervised phase, in order to classify data based on the Euclidean distance. However in the context of electronic nose measurements, a single map often rapidly becomes useless due to drift. If a neuron is not often activated, it would not map the new probability density of the odour. This means that, if a cluster moves to a new position, it is not obvious that all the neurons belonging to that cluster will be updated simultaneously. This behaviour could give rise to confusion, since in the middle of a cluster there could be a neuron that belongs to another cluster and that has not been activated since very long time. For this reason systems of multiple self-organized maps have been developed in literature that enable a self-adjusting process to all the neurons in each local map, and autonomous adaptation to new situations. One of these approaches preserves the self-organization
paradigm by considering as many maps as the various odour classes, which are taken under consideration, in order to accomplish the classification task. The peculiarity in this architecture is the possibility of adjustment of the individual maps step by step in order to be able to predict gas measurements which have suffered from drift. Figure 2.5 shows the mSOM neural architecture. Mainly the processing is made in two phases: training and testing with self-training. In the initial training phase each map is trained with observations belonging to the same odour, i.e. pre-processed measurement vectors $x$. The pre-processing is actually carried out by extracting the first useful principal components over the training set composed of vectors $x_j(t)$ where $j=1, \ldots, m$ represents the odour class.

Once the maps are self-organized, a refining process is performed, in order to reduce the high uncertainty accumulated at the borders of two or more different clusters, which in this case are actually the boundaries of the maps. The refining process is discussed below and it is based on the well-known learning vector quantization (LVQ) algorithm [8, 9]. At the end of this two stages of training (SOM+LVQ), the maximum quantization error for each map is found ($\max_{x \in M_j} q_{err}$), based on the initial presented data given by a teacher, then stored into local memories $M_j$ for $j=1, \ldots, m$. Now the testing phase can take place, where autonomous classification is carried out.

![Figure 2.5: mSOM architecture.](image-url)
The testing phase is not driven by a normal teacher but is completely autonomous and is based on the past history of each map that is contained into local memories. In fact, local memories are simply used to store a certain number of recognized patterns needed for the retraining process and to control the input probability distribution by means of the quantization error. They have a fixed dimension and work as a chain, containing a certain number of recognized patterns occurred in a given period of time.

At a certain time step $t$, a pre-processed measurement vector $x$ is presented to the network. The Euclidean distance measure is computed over all neurons of the maps, and the winner map, which is the one with the minimum distance, is considered as the estimated recognized odour. The quantization error is defined with the Euclidean distance and is computed as the average of the distances between all data vectors and the neurons in the codebook of each map. Each unknown vector $x$ that is presented to the network and then recognized with the winning map $j$ by the nearest neuron $c$, is stored in the corresponding local memory $M_j$ if and only if the following condition is verified:

$$\| x - w_c' \| \leq \max_{s \in M_j} q_{err}(last\_training)$$  \hspace{1cm} (2.4)

After a certain number of unknown pre-processed vector presentations $x$, if the data are subjected to drift, the distribution of data stored into local memories changes, and a self-training process is needed to re-adapt the network to reflect the new odour distribution. In fact, if no re-adaptation takes place, mSOM algorithm decreases its performance. The self-training process is performed using recognized vectors stored into local memories during the testing phase. Furthermore, misclassification is mainly caused by noisy data that can be wrongly stored into the local memories, ending up with an unstable network behaviour. To prevent this problem, the maximum quantization error is computed at the end of the training and each self-training process. Note that drift may be not a slow process, then it is possible to track odours even if the constraint of storing patterns into local memories for further self-training holds true. To test the network, the objective function to be reduced for each map
j = 1, ..., m is the average quantization error computed both at every presentation and at the end of each self-training process, defined as follows:

\[
q_{\text{err}_{\text{ave}}}^{(j)}(t) = \text{mean}_{x \in M_j(t)} \left\{ \sum_{w \in \text{map}_j(t), x \in M_j(t)} \| x - w \|_2 \right\}
\] (2.5)

This value gives always information about the input probability distribution partially represented into local memories of the “jth” map, in order to check for enabling a new retraining process.

### 2.4.3 Learning Vector Quantization process (LVQ)

Learning vector quantization (LVQ) is a supervised learning method based on a reward-punishment scheme, in order to improve the quality of the decision surface. Once the maps have been organized in order to approach the input probability distribution, LVQ algorithm is carried out. LVQ is a useful method to refine clusters and to reduce the areas where two or more clusters with highest uncertainty are present. This area type is also known as Bayesian border. The use of LVQ for fine tuning prototypes, e.g. finding optimal class separation, learned by the basic SOM algorithm, has also been applied in speaker-independent speech recognition problems. The idea behind LVQ is that a data vector \(x\) is presented to the maps where competitions into each map take place for the winner neuron selection. Then the second competition process selects the nearest map to the input vector \(x\) based on the selected winners. If the label of the input vector \(x\) agrees with the label of the winning map \(j\), then the corresponding winner neuron \(w_c^j\) is moved in the direction of the input vector. If, on the other hand, the class label of the input vector \(x\) and the label of the winning map \(j\) disagree, the winner neuron \(w_c^j\) is moved away from the input vector. This may be seen as an adapted version applied to multi maps of the first version of the learning vector quantization algorithm referred to as LVQ1 by Kohonen [10] since two improved versions, LVQ2 and LVQ3, have been introduced. In these additional versions, two codebook vectors \(w_c^{(i)}\) and \(w_i^{(i)}\) of a certain map \((i)\) that are the nearest neighbours to \(x\) are now updated simultaneously. The input vector \(x\) must
fall into a zone called window, defined by a mid-plane of \( w_c \) and \( w_i \). Assuming that \( d_c \) and \( d_i \) are the Euclidean distances of \( x \) from \( w_c \) and \( w_i \) respectively, then \( x \) is said to fall into a window of relative width \( s \) if \( \min(d_c/d_o, d_i/d_o)>t \) where \( t=(1-s)/(1+s) \). In mSOM, the refining process is carried out by using all the data stored into local memories. Thus for each map \( \text{Map}_j \) with \( j=1, \ldots, m \) each vector \( x \in M_j \) stored in the corresponding local memory \( M_j \) is taken to find the winner neuron \( W_c^{(j)} \) and the second winner \( w_i^{(j)} \), in order to update the codebooks in one of the following cases:

1. if \( x, w_c^{(j)}, w_i^{(j)} \) belong to the same map, which actually means same odour class, but fall into the window:

\[
\begin{align*}
    w_c^{(j)}(t+1) &= w_c^{(j)}(t) + \varepsilon \lambda(t) [x - w_c^{(j)}(t)] \\
    w_i^{(j)}(t+1) &= w_i^{(j)}(t) + \varepsilon \lambda(t) [x - w_i^{(j)}(t)]
\end{align*}
\]

2. if \( x \) and \( w_c^{(j)} \) belong to the same map \( j \), while \( x \) and \( w_i^{(j)}, \) actually the second nearest, belong to different classes and thus are different odours we have:

\[
\begin{align*}
    w_c^{(j)}(t+1) &= w_c^{(j)}(t) + \lambda(t) [x - w_c^{(j)}(t)] \\
    w_i^{(j)}(t+1) &= w_i^{(j)}(t) - \lambda(t) [x - w_i^{(j)}(t)]
\end{align*}
\]

where the learning rate \( \lambda \) is usually made to decrease monotonically with time and \( 0.1<\varepsilon<0.5 \) is related to the width of the chosen window \( s \). It is recommended to initialize the learning rate as a quite low factor, because it may happen that initially no local minima are found, leading to an increasing quantization error. Figure 2.6 shows an example of feature extraction of water, propanol, acetonitrile, acetone, butanol and methanol compounds by projecting a data set from a 32-dimensional space to the first tree principal components exploiting first mSOM technique and then Learning Vector Quantization.
Figure 2.6: Example of feature extraction result exploiting both mSOM and LVQ.

2.4.4 Adaptive resonant theory (ART)

Adaptive resonant theory (ART) was introduced as a theory of human cognition in information processing. It is based on the fact that a human brain can learn new events without necessarily forgetting events learnt in the past [11]. ART networks are intelligent systems that are capable of autonomously adapting in quasi real time to changes in the environment. They have been studied to solve the stability-plasticity dilemma and so they are solid enough to incorporate new information without destroying the memories of previous learning. More details may be found in literature in the work of Carpenter [12]. ART networks have been applied to metal oxide sensor based electronic noses. The results are very similar to those obtained with multilayer back-propagation trained networks [13], but the training time is typically an order of magnitude faster if small data set is used.

2.4.5 Linear dynamic models or techniques

The techniques that are typically used for modelling dynamic sensor response are borrowed from the field of system identification. This field is actually the process developing a mathematical representation of a physical or chemical dynamic system or phenomenon using experimental input-output data. The majority of methods that
have been developed to study engineering problems assume linearity and stationary. However, almost all real chemical transducers are characterised by nonlinear laws and response drift. This sub-section shortly reviews a couple among the most significant dynamic linear techniques employed for the extraction of odour classes.

### 2.4.5.1 ARMA, ARX, ARMAX and Box-Jenkins

Linear methods have been applied in various fields like econometrics, biological systems and control systems. Their application in electronic smelling for the identification of sensor array systems is quite recent. The objective of the dynamic model is actually to forecast the sensor response from knowledge of the input signals in dynamic conditions, also named *forward modelling*. Only the inversion of the model could allow to identify the input, e.g. the gasses or aromas once given the output signals, i.e. *inverse modelling* approach. The most popular models are the *Auto-Regressive Moving Average* ARMA, the *Auto-Regressive with Extra Input* ARX, a combination of them, named ARMAX or *Box-Jenkins* technique. These models are of interest in digital signal processing because the time series can be considered to be the output of a linear filter with a rational transfer function. In the following their mathematical expressions are given, where \( x(n) \), \( y(n) \) and \( e(n) \) are the sampled input, the numerical output and the noise or error term, respectively:

\[
ARMA(q, p): y(n) = \sum_{i=1}^{q} \alpha_i y(n-i) + \sum_{j=0}^{p} \beta_j e(n-j)
\]  \hspace{1cm} (2.8)

The current value of the output is modelled using \( q \) past values of the output and the present and \( p \) past values of the noise. Two different sub-models of this one can be considered: the *Auto-Regressive only* (AR) and the *Moving Average only* (MA):

\[
AR(q): y(n) = \sum_{i=1}^{q} \alpha_i y(n-i) + e(n)
\]  \hspace{1cm} (2.9)

\[
MA(p): y(n) = \sum_{j=0}^{p} \beta_j e(n-j)
\]  \hspace{1cm} (2.10)
The ARX model, instead, uses a linear combination of the past $q$ values of the output and the present and past $r$ values of the input as follows:

$$\text{ARX}(q,k): y(n) = \sum_{i=1}^{q} \alpha_i y(n-i) + \sum_{k=0}^{r} \gamma_k x(n-k) + e(n) \quad (2.11)$$

ARMAX algorithm, instead, is similar to the ARX, but it includes a moving average term, as reported below:

$$\text{ARMAX}(q,k,p): y(n) = \sum_{i=1}^{q} \alpha_i y(n-i) + \sum_{k=0}^{r} \gamma_k x(n-k) + \sum_{j=0}^{p} \beta_j e(n-j) \quad (2.12)$$

Finally in the Box-Jenkins model, the prediction of the output is made without the use of past values of the output. It only uses present and past values of the input in addition to filtered noise, as explained in the following expression:

$$\text{Box–Jenkins}(r,p): y(n) = \sum_{k=0}^{r} \gamma_k x(n-k) + \sum_{j=0}^{p} \beta_j e(n-j) \quad (2.13)$$

### 2.4.5.2 State-space models

In the state space form, the relationship between the input, noise and output signals is written as a system of first order difference equations using an auxiliary state vector $\xi_n$. This description of linear dynamical systems became increasingly important after Kalman’s work [14] on forecast and linear and quadratic control. Insights into the physical mechanism of the system can usually more easily be represented using space-state models than into the models described in the previous paragraph. The state-space model can be summarized as:

$$\xi_{n+1} = A(\vartheta) \xi_n + B(\vartheta)x(n) + e_p(n) \quad (2.14 \ a)$$

$$y(n) = C(\vartheta) \xi_n + e_m(n) \quad (2.14 \ b)$$

where $A$, $B$ and $C$ are matrices of appropriate dimensions and $\vartheta$ is a vector of parameters that typically correspond to unknown values of physical coefficients. As
example, in Figure 2.7, the trajectories of an element of a sensor array in the phase space for different analytes is shown. The data processing has been carried out by the project partner team of University of Rome, Tor Vergata.

Figure 2.7: The sensor element response trajectories span in the phase space in presence of different volatile analytes (arbitrary units).

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Chapter 3

Read-out circuit

3.1. Introduction

This chapter presents the gas sensor interface circuit requirements and some possible solutions to implement the gas-sensor read-out circuit. The first possible solution presented is a range compression-based front end circuit; then a second solution is reported, which is instead a state of the art resistance-to-frequency conversion based circuit, while the last reported solution is based on an auto-ranging circuit mainly oriented for high-resolution outdoor air measurements. At the end of the chapter the solution developed within the considered project, is described more accurately, but avoiding to go too much in detail. This is a low-cost, wide-range front-end circuit for resistive gas sensors, able to operate also without circuit calibration, particularly oriented for indoor applications also based on resistance-to-frequency conversion. This measurement method improves the state of the art by decoupling the Resistance
value Controlled Oscillator circuit (RCO) from the resistive sensing device, thus leading to constant voltage bias sensor and hence to higher overall performance. A second interesting feature of this solution is the embedding of a novel digital frequency measurement control system able to extend the sensor read-out range interval. The improved version reported in this work presents an even higher input range and has been optimized in order to allow the possibility to integrate this precision circuit in the same chip with a temperature control circuit, including power stage. Circuit details are presented in chapter 5.

3.2. Interface circuit requirements and state of the art

As mentioned in previous chapters, portable gas sensing systems are becoming everyday more and more essential to put into effect world-wide new legislative initiatives with the aim of monitoring pollution and air quality as well as to keep human beings far away from dangerous environments [1]. The interest in portable systems regards both outdoor and indoor gas detection and concentration measurements. For these reasons, being the development of integrated gas sensing circuits mandatory for embedding in portable devices, manufacturers are now dropping the employment of expensive ad-hoc instruments or multi-chip board solutions, preferring the micromodule or ASIC approach [2], justified also by the opportunity of embedding novel digital data post-processors. The innovative algorithms on which these new feature extraction circuits are based may lead to significant power saving, because they are able to extract information also from dynamic sensors response, thus needing a shorter sensor query time to reach the same performance [3]. Combining this advantage together with pattern modulated temperature techniques, a further efficiency improvement arises. This latter operation mode exploits the additional information given by sensors when they are read-out at specific different temperatures, taking in consideration also temperature transient edge profiles [4, 5]. In view of the large spread of the electrical characteristics of gas sensors, it is evident that the interface and signal processing circuitry becomes very
important in order to achieve a robust system. In most gas monitoring systems available at present in literature, the most significant design effort has always been devoted to the development of the sensors, while “off the shelf” discrete components mounted on printed circuits boards have always been used, even in most of the latest implementations for the interface circuit. The resulting circuits have to be trimmed every time to match the characteristics of the sensor, have large power consumption and dimensions, thus being unsuitable for portable applications. The implementation of the interface circuits in integrated form could overcome this problems, leading to a highly innovative gas monitoring system. Gas sensors which have been used in this project are semiconductor devices, which from the electrical point of view behave as resistors, whose resistance varies with gas concentration. The resistance value in the absence of gas, named baseline, can vary for the same sensor over time as much as two orders of magnitude, while among nominally equivalent sensors this variation can become three orders of magnitude due to technology spread [6]. Obviously, this introduces big challenges in the design of an interface circuit capable of working over the whole resulting resistance range, without requiring any calibration phase performed by the operator, as it is the case in the systems reported in literature. In the interface circuits implemented with discrete components this problem can be solved by replacing or trimming some components depending on the characteristics of the sensor. This, however, cannot be done for obvious reasons in integrated interface circuits. Indeed, so far there are several integrated circuits for reading out resistive sensors, although with much lower input ranges. Typically, these interface circuits are based on bridges or on volt-amperometric methods which exploit operational amplifiers in feedback configuration or on resonators. All of these approaches have advantages and disadvantages, which have to be carefully considered. Concerning the control of sensors parameters, considerable attention has been paid to temperature control and actuation. Temperature is a key parameter for semiconducting gas sensors mostly because the interactions between analytes and sensitive materials are always governed by processes characterized by defined activation energies and hence they are extremely sensitive to temperature. For this reason, the temperature control
sub-system can play an important role in determining the sensors characteristics such as sensitivity, selectivity and baseline, which are of great importance for sensor arrays. From this point of view, many documents, appeared recently in literature, pointed out how in metal-oxide, such as Tin-Oxide (SnO$_2$), semiconductor resistive sensors it is possible to virtually increase the number of sensors of an array by changing from time to time the sensor temperature. From this point of view, temperature is not only a parameter to be controlled in order to ensure a stable behaviour of the sensor, but also a parameter to be actuated in order to modify the sensor performance, thus increasing the information provided by the sensor array [7]. For instance, the application of well defined temperature profiles, like ramps or thermal cycles, recently allowed spectral techniques exploiting both Fourier and wavelet transforms. This new approach for studying the sensor signal opens new perspectives on the kind of information which can be extracted from a chemical sensor. All the reported gas-sensing innovative technique require, as briefly underlined in the second chapter, an interface circuit sufficiently fast, i.e. having a data throughput of the order of 10Hz or faster, in order to manage these exploitable transient data, introducing minimum distortion. For indoor gas monitoring applications, the resistance value range to be measured is comparable to the one needed for outdoor gas sensing purpose, but the absolute mean resistive value of the microsensors customized for this particular approach (pure SnO$_2$ sensors or Tin-Oxide devices with ad-hoc additional dopant concentrations are once more the best candidates) is actually higher of about one decade. In addition, the resistance value measurement precision required by indoor pattern data processing algorithms is slightly relaxed. In fact, for indoor gas monitoring applications, in order to detect every kind of gas of interest with enough accuracy, usually in the order of ten ppm after pattern analysis, it is important to measure the sensor resistance value ($R_{sens}$) with a precision around few percent. Furthermore, considering the mentioned state of art in Tin-Oxide sensors manufacturing, the sensor resistance value may once more vary across decades, being the effect of the three typical sensor response components: the baseline, which in indoor oriented sensors typically ranges, depending on the
device type, from a moderate value, like 100 kΩ, to a much higher one, like 100 MΩ, the deviation from the baseline, due to technology spread and sensor aging as well as temperature and finally the resistance variation, due to gas concentration, which may be as large as a couple of decades below or above the baseline, depending on the presence of oxidizing or reducing gases respectively.

### 3.3. Compression based front-end circuit

A wide-range interface circuit without calibration may be realized performing a compression of the sensor current value (I_sens) as shown in Figure 3.1. In this case, as an example, the current flowing through a constant voltage, e.g. V_{REF}, biased sensor could be mirrored into a MOSFET or BJT transistor in diode configuration. In this scheme, a sensor driven current I_sens is mirrored into a diode D1, while a precise reference current I_{REF} flows in a matched diode D2; thus the difference between D1 and D2 voltage drops, V_0, may be read as a compressed value for R_{sens}:

$$V_0 = -V_T \times \ln \left( R_{sens} \times \frac{I_{REF}}{V_{REF}} \right)$$

(3.1)

Unfortunately, even if the wide-range is guaranteed by compression, it is difficult to achieve an accuracy better than few percents in resistance measurement, due to the intrinsic behaviour of such devices, for resistance values ranging in [10kΩ-100MΩ] applied to the circuit of Figure 3.1.

Other implementations of the same approach have already been developed leading approximately to the same performance: a precision around 2% over 4 decades [8]. A main limitation of the circuit is the minimum measurable current through the diode due to unavoidable parasitics which affect linearity performance.
### 3.4. Oscillator based front-end circuit

The block diagram of a front-end circuit based on an oscillator [9, 10] is reported in Figure 3.2. The resistance to time conversion is performed by an integrator stage composed of operational amplifier OP$_1$ and capacitor C = 100 pF; it integrates the current flowing through the sensor, which is modelled using a resistance R$_{SENS}$ in parallel with a capacitance C$_{SENS}$. Since the sensor excitation voltage V$_{exc}$, for a time period, is constant, the integrator output V$_{OP}$ is, in the same period, a falling or a rising ramp, depending on the sensor current direction.
To better explain the system behaviour, a timing diagram is shown in Figure 3.3. The ramp is compared, by the comparator COMP\text{th}, with a threshold V_{th} that follows, with the opposite sign, the excitation voltage V_{exc} and COMP\text{th} generates a square-wave voltage signal V_{Cth}, whose amplitude is equal to the total supply voltage and whose period T_{C} is proportional to sensor resistance R_{SENS} according to:

$$T_C = 4GC R_{SENS} \left(1 - \left(\frac{C_{SENS}}{GC}\right)\right)$$  \hspace{1cm} (3.2)

where C is the integrating capacitor and G is the ratio between R_{2} and R_{1}. Sensor capacitance C_{SENS} involves a charge transfer, which affects the ramp signal when a voltage commutation occurs, through a vertical edge on V_{OP}, as shown in Figure 3.3.

![Figure 3.3: Timing diagram of the oscillator circuit.](image_url)

The comparator COMP_{0} separates the ramp signals in two parts: The first part, immediately after the commutation, presents the charge transfer effect due to C_{SENS}.
whereas the second part depends on $R_{\text{SENS}}$ only. In detail, the exclusive-OR logic block $\text{XOR}$ generates a square-wave signal $V_{\text{XOR}}$, which allows the estimation of both the $C_{\text{SENS}}$ and $R_{\text{SENS}}$ values:

\[
R_{\text{SENS}} = \frac{T_{C2} + T_{C4}}{2GC} \quad (3.3)
\]

\[
C_{\text{SENS}} = GC \frac{(T_{C2} + T_{C4} - T_{C3})}{2T_{C2} + 2T_{C4}} \quad (3.4)
\]

where the increasing and decreasing ramp contributions have been averaged.

This state of the art oscillator based front end circuit realized with discrete components shows a theoretical ultra-wide dynamic range, from $100 \, \text{k\Omega}$ to $1 \, \text{T\Omega}$. The precision obtained in Spice® simulations is around 2% on about 6 decades (from $1 \, \text{M\Omega}$ to $1 \, \text{T\Omega}$) but decreases to 8% on 7 decades (from $100 \, \text{k\Omega}$ to $1 \, \text{T\Omega}$).

Again, in the actual implementation of the circuit, there is a stronger limitation in the maximum measurable resistance value, due to unavoidable parasitic components which limit the accuracy of the oscillator frequency with respect to input resistance value. By “off the shelf” components implementation a reasonable maximum measurable resistance value, if the desired accuracy is of the order of few percent, is few $\text{G\Omega}$.

### 3.5. Auto-scaling front-end circuit

The block diagram of a different solution that has been implemented for high-accuracy outdoor applications and which shows very high performance is shown in Figure 3.4 [11, 12]. It is composed by a single-ended continuous-time Programmable Trans-resistance Amplifier (PTA) that converts the current $I_{\text{SENS}}=V_{\text{REF}}/R_{\text{SENS}}$ flowing in the sensor into a voltage and by a fully-differential switched-capacitor oversampled 13-bit incremental A/D converter (ADC) that digitizes the output of the first stage. In addition two 8-bit DACs and a digital section allow the PTA features to be reconfigured to match the sensor specifications.
The overall schematic of the PTA is shown in Figure 3.5. The gas sensor, modelled as a resistance $R_{sens}$, is connected between a reference voltage $V_{REF}$ and the operational amplifier virtual ground, biased at $V^+$. A current $I_{sens}$ is then flowing across the sensor, given by:

$$I_{sens} = \frac{V_{REF} - V^+}{R_{sens}} \quad (3.5)$$

$V^+$ is biased at 2V, while $V_{REF}$ is nominally biased at 2.5 V, even if this value will be adjusted during calibration. This leads to a nominal 500 mV voltage drop across the sensor resistance.

The operational amplifier output voltage, ignoring $I_{CAL}$ contribution is then given by:
\[ V_0 = -V_{REF} \times \frac{R_f}{R_{sens}} + V^+ \times \left(1 + \frac{R_f}{R_{sens}}\right) \] (3.6)

\[ V_0 = (V^+ - V_{REF}) \times \frac{R_f}{R_{sens}} + V^+ \] (3.7)

In this application if the resistance value range to be measured is of the order of decades, it would give a comparable and, then, extremely large dynamic range at the amplifier output voltage and a direct approach could not be implemented, also due to the unavoidable noise floor, which of course limits the minimum absolute resolution. In order to accommodate this large \( R_{sens} \) range, the feedback resistance \( R_f \) is adjusted with the aim of keeping it of the same order of magnitude of \( R_{sens} \). In this way the output swing is comparable to \( V_{REF} \) and can be managed by the operational amplifier. The absolute resistance range for indoor applications is different but the concept is the same and this circuit could work at higher resistance values simply by using different values for \( R_f \). This first stage re-configuration is done by splitting the resistance values into different sub-ranges, each of them covering about half decade. These different scales are generated by realizing the feedback resistor with an array of different possible \( R_f \) to be properly connected for each sub-range. The use of multiple scales requires to face the problems arising from the mismatches (mainly in terms of offset and gain error) between consecutive sub-ranges. A calibration technique, which exploits a partial overlap of adjacent scales of about a quarter of decade, is used for this purpose. Notice that the above approach of adjusting the \( R_f \) value to match the \( R_{sens} \) value does not give a full resistance matching, due to quantized values of \( R_f \). Any mismatch between adjacent \( R_f \) with respect to the \( R_{sens} \) corresponding value leads to a deviation of the output voltage and then limits the accuracy. For this reason a calibration current \( I_{CAL} \) is added. The output voltage expression can then be written as:

\[ V_0 = (V^+ - V_{REF}) \times \frac{R_f}{R_{sens}} + V^+ + I_{CAL} \times R_f \] (3.8)

The use of \( I_{CAL} \) allows us to recover the optimum value for \( V_0 \), which is 2 V after startup calibration, value near to upper voltage range end, taking into account that gas presence has almost always the effect to decrease \( V_0 \), being most gases of interest...
oxidizing. Thus the trans-resistance stage output swing for a given scale, including upper and lower over-range is [1.05 V-2.25 V], which corresponds to the resistance measurement interval [−Rf/3-2Rf]. Notice that ICAL allows us to calibrate the stage with respect to any imperfections, like global system offset (Voffset), and to cancel any bias value of the sensor resistance, i.e. for cancelling baseline (Rbl) and baseline deviation (ΔRbl) at system start-up. In this way the effective V0 deviation is correlated only to the sensor resistance deviation due to the gas presence (−ΔRgas). Remembering that, for oxidizing gases in the following expression ΔRgas is positive:

\[ R_{sens} = R_{bl} + ΔR_{bl} - ΔR_{gas} \]  \hspace{1cm} (3.9)

and referring all to V+=2V, the output voltage V0 is:

\[ V_0 = a \times V_{REF} + b \times V_{offset} \]  \hspace{1cm} (3.10)

\[ a = -\frac{R_f}{(R_{bl} + ΔR_{bl} - ΔR_{gas})} \]  \hspace{1cm} (3.11)

\[ b = 1 + \left[ \frac{R}{R_{bl} + ΔR_{bl} - ΔR_{gas}} \right] \]  \hspace{1cm} (3.12)

Then, introducing the controlled current ICAL, we obtain:

\[ V_0 = a \times V_{REF} + b \times V_{offset} - ICAL \times R_f \]  \hspace{1cm} (3.13)

The output voltage deviation in the absence of gas can then be reduced by using the optimum current ICAL, given by:

\[ I = \left( \frac{V_{REF} - V_{offset}}{R_{bl} + ΔR_{bl}} \right) \]  \hspace{1cm} (3.14)

Figure 3.5 shows the complete schematic of the read-out circuit. In this circuit, the regulated current source used to compensate inter-scale system offset mismatch is realized with an 8-bit buffered resistive DAC (DAC1 in the schematic) and a programmable resistor R_{DAC}, that also needs to be selected from an array.
In this design $R_{DAC} = R_f$ in order to keep the operational amplifier working with gain and feedback factors of the same order of magnitude over the entire dynamic range and to guarantee a good integrated component matching for $R_{DAC}$ and $R_f$. Notice that $I_{\text{cal}}$ may range from 50 nA to 1.5 mA, while $I_{\text{sens}}$ may range from 25 nA to 5 mA. Furthermore, by regulating the sensor voltage reference ($V_{\text{REF}}$) through an additional buffered DAC (DAC2), it is possible to correct separately the gain-error of each of the 10 scales available in the circuit. Again, as shown in Figure 3.5, the two 8-bit D/A converters (DAC1, DAC2) and the two selector circuits for $R_f$ and $R_{DAC}$ (SEL1, SEL2) are all controlled by a common digital unit, whose tasks are of course the choice of the current measurement range and the actual correction of offset and gain error for each scale using the “calibration words” determined during initial setup phase. This read-out circuit shows impressing performance with an accuracy over the overall dynamic range of more than 5 decades near to 0.1 %, but the calibration cost to join the different scales is quite high and it is not justified for the purpose of the system considered in this thesis for end-user portable applications, especially for indoor gas measurements.

![Figure 3.5: Complete schematic of the PTA based read-out circuit.](image-url)
3.6. **Overview of the implemented interface circuit**

The implemented interface circuit schematic is reported in Figure 3.6 [11, 13]. The resistive sensor, $R_{\text{sens}}$, is biased with an accurate reference voltage $V_{\text{REF}}$, applied exploiting a low-noise, high-gain (100 dB), low output resistance amplifier in V-to-I configuration. A couple of high-linearity cascoded current mirrors alternately push or pull the current $\delta(V_{\text{REF}}/R_{\text{sens}})$ into or from the virtual ground of a resettable Miller integrator, which has also the aim of keeping constant the voltage drop across the output current mirrors, while $V_0$ varies, thus rising the linearity of the response. The reference voltage has been set to the nominal value $V_{\text{REF}}=1$ V after simulation level system evaluation. The 2-stage operational amplifier employed in the integrator exhibits a DC gain of 60dB and a unity-gain bandwidth of 280 MHz. By comparison of the output of the integrator with two constant boundary voltages $V_H$ and $V_L$ ($V_H=2.2$ V, $V_L=1.1$ V), by means of two high-speed continuous-time comparators ($T_{\text{response}}<2$ ns) the control signals for the switches that shunt the mirrored current are generated. An additional control logic finally grants that comparators switch alternately (half-infinite equivalent hysteresis principle) and feeds an internal counter, whose transition period, $T_{\text{OSC}}$, is consequently directly proportional to the value of $R_{\text{sens}}$:

$$T_{\text{OSC}} = \frac{2C\times\Delta V\times R_{\text{sens}}}{\delta\times V_{\text{REF}}}$$

(3.15)
3.6.1 A frequency based measurement technique

The developed logic, embedded in the interface circuit, takes care of the frequency measurement. The implemented technique allows us to handle a dynamic range which is two times (in terms of decades) with respect to previously published frequency measurement methods. This circuit works as follows: the ratio between a reference counter, whose clock frequency is set at the midrange of the possible oscillator frequency interval \( f_{\text{mid}}=(f_{\text{min}} \cdot f_{\text{max}})^{1/2} \) and a counter, whose clock frequency is the output signal of the resistance dependent oscillator, represents the digitized value of \( R_{\text{sens}} \). Therefore, the measured resistance value is given by:

\[
R = \alpha \times R \times \frac{N_{\text{ref}}}{N_{\text{osc}}} + \beta
\]

where \( N_{\text{ref}} \) and \( N_{\text{osc}} \) indicate the reference and the oscillator frequency dependent counter values at the end of the conversion, respectively, \( R_{\text{mid}} \) the logarithmical center of scale, while \( \alpha \) and \( \beta \) represent the additional front-end circuit gain and offset contribution terms. These coefficients may be left uncompensated in gas sensing because they do not affect linearity in gas concentration extraction. The measurement ends when the slower of the two counters reaches a given value \( N^* \) which is sufficient to achieve the desired accuracy, i.e. \( N^*=256 \) for a minimum guaranteed 8-bit equivalent precision, as implemented in the presented interface system. As a
consequence, if we need to measure resistance values in the range of at least ±2 decades centered around \( R_{\text{mid}} \), over a single read-out scale, the minimum number of bits needed for each of the two counters registers is given by:

\[
N_{\text{BIT}} > \log_2 \left( N \times \frac{R_{\text{max}}}{R_{\text{mid}}} \right) = \log_2 (256 \times 10^2) > 14
\]  

(3.17)

More in detail, in the implemented circuit, a larger number of bits is employed for the oscillator-dependent counter to allow resistance measurement 3 decades below \( R_{\text{mid}} \). Then, if the applied \( R_{\text{sens}} \) value is lower than midrange, the slower of the two counters reaching \( N^* \) is going to be the reference one and the measurement time is constant. By contrast, the slower counter is going to be the one controlled by the oscillator if the measured \( R_{\text{sens}} \) is larger than midrange, leading to a measurement time proportional to the resistance value itself. Therefore, the output digital word will be composed by 15 bits, which represent the value reached by the faster of the two counters during the measurement and by an additional bit, which indicates if the converted resistance value is either higher or lower than \( R_{\text{mid}} \). In order to extend the dynamic range of the system, three different current scaling ratios (\( \delta = 1:200 \), \( 1:20 \), \( 1:2 \)) are available for the input of the Miller integrator by means of a programmable set of current mirrors, allowing a full-range measurement in terms of resistance value varying from 1 k\( \Omega \) to 1 G\( \Omega \) by exploiting different overlapped scales. Programmability in the integrator capacitance has also been introduced in order to improve accuracy and conversion speed for the maximum resistance values, choosing a value for \( C \) among two: \( C = 10 \text{pF} \) or \( C = 1 \text{pF} \). The nominal value for the reference external clock is \( f_{\text{mid}} = 2.5 \text{kHz} \), thus leading to a constant conversion time for \( R_{\text{sens}} < R_{\text{mid}} \) close to 100 ms for every \( \delta \)-C combination, while for higher resistance values the time needed for conversion is, for example, setting \( \delta \) to 1:200 and \( C = 10 \text{pF} \), \( R_{\text{sens}} \) \textit{microseconds}, if \( R_{\text{sens}} \) is expressed in Ohm, as reported in Table 3.1.
Table 3.1: Summary of available sub-ranges with $f_{\text{mid}}=2.5\text{kHz}$, for generic $f_{\text{mid}}$: $R_{\text{min}}$, $R_{\text{max}}$ and $R_{\text{mid}}$ must be normalized to $2.5\text{kHz}/f_{\text{mid}}$.

<table>
<thead>
<tr>
<th>Mode</th>
<th>$\delta$</th>
<th>C</th>
<th>$R_{\text{min}}$</th>
<th>$R_{\text{max}}$</th>
<th>$R_{\text{mid}}$</th>
<th>Time ($R&gt;R_{\text{mid}}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>00</td>
<td>1:200</td>
<td>10pF</td>
<td>1k$\Omega$</td>
<td>10M$\Omega$</td>
<td>100k$\Omega$</td>
<td>$R$ $\mu$s</td>
</tr>
<tr>
<td>01</td>
<td>1:20</td>
<td>10 pF</td>
<td>1 k$\Omega$</td>
<td>100 M$\Omega$</td>
<td>1 M$\Omega$</td>
<td>$(R/10)$ $\mu$s</td>
</tr>
<tr>
<td>10</td>
<td>1:2</td>
<td>10 pF</td>
<td>10 k$\Omega$</td>
<td>1 G$\Omega$</td>
<td>10 M$\Omega$</td>
<td>$(R/100)$ $\mu$s</td>
</tr>
<tr>
<td>11</td>
<td>1:2</td>
<td>1 pF</td>
<td>100 k$\Omega$</td>
<td>1 G$\Omega$</td>
<td>100 M$\Omega$</td>
<td>$R$ ns</td>
</tr>
</tbody>
</table>

3.6.2 Available front-end resistance value measurement ranges

Notice that, as appears in Table 3.1 not all sub-ranges $[R_{\text{min}}^{\ast}, R_{\text{max}}^{\ast}]$ are symmetrical with respect to $R_{\text{mid}}$. In fact these ends, as a difference from mentioned $[R_{\text{min}}, R_{\text{max}}]$ geometrical bounds take also in account further physical limitations and eventual over-ranging capabilities of the interface circuit. The lower limit is typically given by the fact that the maximum internal recommended oscillator frequency is $f_{\text{osc,max}}=1/T_{\text{osc,min}}=2.5$ MHz, while the maximum current to be measured should be 1mA ($R_{\text{sens}}=1$ k$\Omega$) due to $V_{\text{REF}}$ buffer linearity specifications, in order to respect the requirements for indoor monitoring purpose. For example, in mode “01” the lower limit conditions coincide. The upper limit in resistance measurable value comes instead when the current lost towards bulk from the input analog pad is no longer negligible for the accuracy of interest: this happens for $R_{\text{sens}}>1$ G$\Omega$, which means in terms of sensor current, $I_{\text{sens}}=1$ nA.

REFERENCES:


Chapter 4

Gas-sensors temperature control circuit

4.1. Introduction

This chapter introduces the gas-sensor temperature control. It begins with a summary of the most common control techniques and continues presenting some examples of state of the art solutions. The first architecture studied is an analog implementation of a proportional controller exploiting an operational transconductance amplifier. The second approach is based on a resistance to frequency conversion with a constant power source. The third presented solution exploits a digital PID controller in feedback loop, while the linearization of the relationship between set point temperature and the heater power is performed in the analogue domain, using a trans-linear MOS geometric mean circuit. Finally, the solution developed in the research work reported in this manuscript, based on a on/off closed loop control, is presented.
4.2. Temperature control for microsensors

As underlined in the gas-sensor overview, micromembrane sensing devices contain an embedded resistive Joule-effect based heater. Sometimes the heating resistor is used also as a thermometer, thus reducing the device lead number of a couple of pins, while the heating resistance is usually in platinum and its temperature coefficient is linear over the gas-sensor operative temperature range. On the other hand, instead, the heater and thermometer parts may be separated, even if interdigitated, thus allowing separate optimization of the heating efficiency and the temperature measurement accuracy. The importance of controlling the membrane temperature, as mentioned, comes from the fact that, since the sensor response is strongly temperature-dependent, a good stability of this variable over time would lead to a higher gas-sensing accuracy, with strong reduction of the drift effect. Consequently, electronic control systems for temperature stabilization should be developed exploiting also the fact that large scale integration technologies, like CMOS, are well suitable for this purpose, even if in some cases the available technology low supply voltage $V_{DD}$ is not sufficient to deliver enough power to heat up the available sensor. This happens when the heater resistance is not sufficiently low or when the sensor membrane with embedded heater is not properly thermally insulated, thus requiring a large amount of power to raise the sensor to a given temperature. On the other hand a very low voltage supply technology together with a very low heater resistance sensor, even if working properly, may lead to a more complex chip layout and may be cause of electro-ionic migration due to the high current value towards the heater if operated with constant polarity, thus reducing the lifetime of the circuit. For these reasons in the developed temperature control circuit we employed a 0.35µm technology with a supply voltage of 3.3V together with a heater sensor resistance of about 30Ω. The heater control circuit, which actually is both a sensor and an actuator interface, may be integrated together with the sensor or in a separate chip. In the first option a greater compactness is achieved while the second solution, the so-called micromodule approach, permits once more to optimize the system with respect to
temperature working conditions. In this section after a quick overview on different common types of control systems, the most suitable technique is justified and then chosen for the temperature control system development.

4.2.1 Quick overview on control systems techniques

Different theories have been developed on how to design controllers for the most various applications. A summary of the possible control techniques is roughly listed:

- **On-off controllers**: are simple and consequently used in a wide range of applications, spanning from domestic heat regulators to industrial level controllers. The output of an on-off controller, i.e. the control signal, is either on or off and therefore it can be implemented easily. Indeed, the actuator part can be even a simple contactor. Since actuators are often among the most complex elements of the control loop, on-off control systems can be utilized reasonably.

- **Classical control**: this technique deals primarily with linear, constant coefficient systems, commonly known as time-invariant systems. Few real systems are exactly linear over their whole operating range, and few systems have parameter values that are precisely constant over longer time spans. However, many systems are approximately linear in a sufficiently narrow operating range of interest. The treatment of linear, stationary systems is greatly simplified by the use of transform techniques and frequency domain approaches, like Bode plots, Nyquist theory and Laplace or Z transform for continuous and discrete signals processing respectively. The most popular classical control techniques are Proportional (P) control, proportional-integral (PI) control, proportional-derivative (PD) control, and proportional-integral-derivative (PID) control, all widely used also for the control of industrial processes and servomechanisms.

- **Optimal control**: this technique is used when specific performance or cost criteria, like time or energy must be minimized. The given constraints or cost
functions are used to derive an appropriate control law, which is then implemented with a controller. The state-space system representation is used in the calculation of the controller. Linear quadratic regulator (LQR) control, linear quadratic Gaussian (LQG) control are examples of optimal control techniques.

- **Robust control**: the controller is designed with due consideration of both, the nominal model of the system to be controlled and the characterization of model uncertainties and disturbances. The system to be controlled has usually the technical name of *plant*.

- **Nonlinear control**: the controller is designed to handle the nonlinearities of a system in a large operation range or to handle systems with discontinuous nonlinearities that do not allow for linear approximation.

- **Intelligent control**: this technique is used in systems with insufficient information about the plant parameters, making it impossible to derive a plant model. It is also used in systems where plant parameters or plant models change over time. Knowledge-based control, adaptive control, fuzzy logic, are good representatives of this last approach.

In summary, a suitable control technique is selected depending on the system dynamics, the control goals, and the controller implementation, which in most cases may have a monolithic implementation, like, for example, FPGA or ASIC DSP. Micromembrane sensors systems may be classified in first approximation as time-invariant single-input/single-output (SISO) systems from the temperature control point of view and they allow for nominal operating point linear approximation. Classical control techniques like proportional (P) control and proportional-integral-derivative (PID) control may be chosen to regulate the heater power of these systems because they are suitable for effective control and thus may be realized as monolithic implementations. The other side of medal is that to implement a classical control technique would mean to force flowing in the heating resistance a current whose value is related with the difference between the imposed
temperature and the actual one, while the power that has always to be available for heating the sensor needs to be as high as necessary to allow the sensor to reach the highest temperature among the possible set-points. In static conditions, when the temperature needs only to be maintained, the difference between the imposed temperature and the actual one is tiny and so is also the power the control really uses. Simple ways to limit the power delivered to $R_H$ are to use voltage or current dividers but those solutions waste power. Other more complicated solutions could use, for example, a power stage buffered DAC to bias the heating resistance. Those solutions ideally do not imply a large amount of wasted power but at the end it again depends on which buffer class has been employed to design the power DAC driver and on the DAC design itself. All above reasons and the goal of a high efficiency led us to decide to use an ON/OFF control technique for the prototype described in this work.

4.3. **Examples of monolithic temperature controllers for microsensors**

4.3.1 **Analog implementation of the proportional temperature controller**

The first example reported is the heater control circuit that has been implemented in the first version of this project. Since the micromodule solution has been preferred, the first prototype of the heater driver has been fabricated on a stand-alone chip [1]. A simplified schematic of this analog proportional controller prototype is reported in Figure 4.1. The circuit exploits an operational transconductance amplifier in closed loop configuration and may be connected directly to microsensors having separated heater-thermometer. Actually the source degenerated power transistor $M_1$ delivers the demanded power to the platinum heater ($R_{HEATER}$) which could be for instance about 100Ω, regulating its current. Thus, the degeneration resistance $R_{DEG}$ may be set by design to a nominal value of about 20Ω, having also the function to limit the output current and thus the maximum actual set-point, enhancing as a consequence the sensor life which could be dramatically reduced by ionic electromigration at very
high temperature and current, even if for a short time. The resistive temperature sensor, alias thermometer or $R_{\text{SENSOR}}$, may be biased by a temperature-independent current source, like an external P-MOS current mirror. The voltage drop across the thermometer resistance provides the feedback signal for the temperature controller. The dominant pole of this system is determined by the thermal time constant of the hotplate, which usually is of the order of tens or hundreds of milliseconds, while the simplified forward path gain of the controller is:

$$A_{FP} = -A_{OL,OP} \times \frac{R_{\text{HEATER}}}{R_{\text{DEG}}}$$  \hspace{1cm} (4.1)

where $A_{OL,OP}$ is the open loop voltage gain of the operational amplifier, which usually is of the order of 100 dB. The degeneration resistance $R_{\text{DEG}}$ makes $A_{FP}$ and consequently $G_{\text{loop}}$ of the controller almost independent of the transconductance $g_{m}$ of transistor $M_1$ and thus of the heater current and temperature.

![Figure 4.1: Analogue implementation of the proportional temperature controller.](image)

A drawback of this scheme is the power loss on $R_{\text{DEG}}$ and on $M_1$, which must work in saturation region and thus exhibits a non negligible $V_{DS}$ voltage. Furthermore, the efficiency is dependent on the characteristics of the sensor. In fact, the ratio between
the power loss and the power delivered to $R_{HEATER}$ depends on the value of both the resistances and consequently the efficiency changes significantly with $R_{HEATER}$ and can vary a lot when the control is used with different sensors, if a programmability of $R_{DEG}$ is not provided. The reported analogue proportional controller does not compensate the fact that the relationship between the voltage control $V_{CONTROL}$ and the power furnished to the heater is not linear, but quadratic. This may be taken into account in design by generating the analogue control signal by means of a D/A converter driven by an additional digital controller which should perform a root square extraction operation to achieve a linear relationship between set point digital value and heater power. A further enhanced solution [2], studied at ETH, Zurich, is reported in Figure 4.2. In this approach a digital PID controller in the feedback loop is exploited, while the linearization of the relationship between temperature set point ($T_{REF}$) and heater power is performed in the analogue domain using a trans-linear MOS geometric mean circuit ($M_1, ..., M_4$), while $T_{MEM}$ is instead the digitized value of the membrane temperature measured by the thermometer $R_{SENSOR}$. The linearization is realized as follows: the control voltage drives a V-to-I converter through a reference resistor $R_{REF}$, located into the membrane in order to have the same temperature of $R_{HEATER}$. In this way, the temperature dependence of the proportionality coefficient between $V_{CONTROL}$ and the heater power is cancelled. The output current of the V-to-I converter feeds the trans-linear loop $M_1, ..., M_4$ and the geometrically averaged current is amplified by a factor $m$ and applied to the heater. This current is used also to adjust the open loop gain of the system by changing the value of the gain current $I_{GAIN}$.

$$P_{HEATER} = \left( I_{GAIN} \times \frac{m^2}{4} \times \frac{R_{HEATER}}{R_{REF}} \right) \times V_{CONTROL} \quad (4.2)$$

Again, in this circuit, the heater driver transistor is operated in saturation region, with a continuous time control and thus power loss due voltage drops is not negligible.
Another possible solution is represented by the schematic reported in Figure 4.3. Temperature is again read-out through a resistance that operates as a thermal sensor [3-4]. By operating the heater at constant power or by turning it off it is possible to control the sensor temperature without interferences. The current (or more generally) the power delivered to the heater resistance must be such that the temperature has to remain constant at its desired value. The value of the thermometer resistance allows through a resistance to frequency conversion the measurement of the instantaneous temperature and therefore the control of the current flowing into the heater resistance.

Figure 4.2: Mixed signal temperature controller.

Figure 4.3: Temperature control system based on resistance to frequency conversion.
The R-T converter scheme is reported in Figure 4.4. It produces a square-wave signal whose period is directly proportional to the $R_{SENS}$ value.

![R-T converter diagram](image)

Figure 4.4: R-T converter with non inverting amplifier and without buffers.

The digital counter is enabled for a given temporal window that is decided by design, taking into account several factors, like the desired measurement resolution and the required $R_{SENS}$ sensitivity against output digital value. The output of the counter is related to the actual temperature of the sensor and a logic control block takes care of enabling or not the constant power heater. The schematic of this temperature control circuit has been reported in Figure 4.5. The temperature control system is driven by a constant power supply, which generates a constant power signal, that is controlled through an external voltage. The two pairs of transistors M6, M8 and M7, M10 realize a trans-linear loop through a negative feedback, that ensures the following relation:

$$4I_{Bias}^2 = I_{D7sat} \cdot I_{D10sat}$$

(4.3)

Transistor M5 acts as a current buffer for transistors M9 and M10 to insulate them from the supply voltage, while $V_{BIAS}$, applied to the gate of M5, sets the required power on $R_{HEATER}$. The drain currents of M9 and M10 are summed and mirrored.
through the cascoded current mirror M1–M4. The current which flows into $R_{\text{HEATER}}$ is equal to $I_{\text{diode}}$, suitably mirrored by M13–M16. By comparing the voltages at nodes A and B, M17 drives the current into M19 and M20, which acts directly on the trans-linear loop. The reported temperature control circuit is more efficient than the previously reported ones because it exploits a kind of ON-OFF technique, but still a large amount of power is dissipated by M15 and M16, operated in saturation region, in order to regulate the amount of power to the heater during the ON phase.

![Heater circuit schematic.](image)

**Figure 4.5:** Heater circuit schematic.

### 4.4. Developed temperature control circuit structure

The leading concept that has been followed in order to develop the temperature control is to realize a circuit as robust, flexible and power efficient as possible [5-7]. The sensor temperature control that have been developed, whose block diagram is shown in Figure 4.6, is a closed loop circuit driven by a 9-bit digital set-point that allows the desired temperature pattern to be easily obtained with sufficient accuracy, exploiting two platinum-titanium embedded resistors (a heater, $R_H$, and a thermometer, $R_T$). The resistor $R_H$ has one terminal connected to ground and the other connected to the power stage output and when it is necessary heats the sensor by
Joule effect. The resistor $R_T$ is a thermometer, whose value, processed by a suitable continuous time conditioning network, is sampled by an ADC, whose 9-bit output, compared with the temperature set-point, controls the gate of the p-MOS power switch.

**Figure 4.6: Sensor temperature control block diagram.**

It has been considered also the possibility to use instead of the p-MOS an n-MOS transistor to deliver power to the heater, because its resistivity in the linear region is lower due to higher carrier mobility. In fact, a lower resistivity would allow us to use a smaller chip area for the switch and, additionally, to further save area for the driver of the switch, being its input load smaller. The circuit, in this case should have been realized as reported in Figure 4.7. This solution has, however, few drawbacks.
First of all in a typical CMOS technology, a p-MOS switch can be realized in a dedicated and insulated n-well, with a dedicated VDD for well biasing, thus avoiding to disturb other parts of the circuit. With the employed 0.35µm technology the insulated n-MOS in a triple n-well is not available and so using a n-MOS power switch would have generated intolerable noise in the substrate. Moreover, in general, in a multi-instrument measurement set-up, like the one that has been realized for ASIC characterization and that will be presented, it is better to refer all the devices that are being used at the voltage generator ground. This is especially true when the VDD is obtained, like in this case, by exploiting on board LDO (Low Drop-Out regulator). It is also better to shield the outer part of the coaxial cable which interconnects the active bipoles of the gas-sensor to the ASIC test board with ground rather than VDD for electromagnetic compatibility issues and to reduce the risk of ground loops.
4.5. **Developed temperature control circuit details**

The continuous time conditioning network is shown in Figure 4.8. High flexibility has been obtained using a digitally programmable current generator for thermometer bias and by giving the possibility to modify the value of $V_A$:

$$V_+(T) = V_A + I_{DC} \times R_T(T)$$  \hspace{1cm} (4.4)

$$R_T(T) = R_{T0}[1 + \alpha \times (T - T0)]$$  \hspace{1cm} (4.5)

where in the nominal case is

$$R_{T0} = R_T(25°C) = 75Ω$$  \hspace{1cm} (4.6)

and

$$\alpha = 0.00216°C^{-1}$$  \hspace{1cm} (4.7)

It is easy to see that, if it is necessary to use a sensor characterized by a $R_{T0}$ of, for example, $150Ω$ instead of $75Ω$, we can restore the nominal conditions simply using a value of $I_{DC}$ that is half with respect to the nominal case.

![Diagram of Continuous time conditioning network](image)

Figure 4.8: Continuous time conditioning network.
Moreover, it is possible to exploit the current DAC in order to change the temperature range that corresponds to the ADC input. In fact, the input range of the ADC, depending on the values of $I_{DC}$ and $V_A$ can vary from room temperature to the maximum sensor temperature, around 450°C, or to the most common temperature working range that is between 250°C and 400°C or in other cases to a more limited temperature range spreading 50 °C around a certain temperature. Of course, being the number of bit fixed and the input range of the ADC given by design, the absolute precision in temperature regulation increases when the temperature range is narrower. Some configuration examples are reported in Table 4.1.

<table>
<thead>
<tr>
<th>First configuration</th>
<th>Second configuration</th>
<th>Third configuration</th>
</tr>
</thead>
<tbody>
<tr>
<td>$R_T=75\Omega$</td>
<td>$R_T=75\Omega$</td>
<td>$R_T=150\Omega$</td>
</tr>
<tr>
<td>$I_{DC}=1.4mA$</td>
<td>$I_{DC}=3.0mA$</td>
<td>$I_{DC}=1.4mA$</td>
</tr>
<tr>
<td>$\alpha=0.00216$ °C$^{-1}$</td>
<td>$\alpha=0.00216$ °C$^{-1}$</td>
<td>$\alpha=0.00216$ °C$^{-1}$</td>
</tr>
<tr>
<td>$V_A=1.5V$</td>
<td>$V_A=1.25V$</td>
<td>$V_A=1.3V$</td>
</tr>
<tr>
<td>$V_s(25^\circ C)=1.605V$</td>
<td>$V_s(25^\circ C)=1.586V$</td>
<td>$V_s(25^\circ C)=1.602V$</td>
</tr>
<tr>
<td>$V_s(450^\circ C)=1.703V$</td>
<td>$V_s(450^\circ C)=1.685V$</td>
<td>$V_s(450^\circ C)=1.684V$</td>
</tr>
<tr>
<td>$V_{ADC}(25^\circ C)=1.335V$</td>
<td>$V_{ADC}(25^\circ C)=1.205V$</td>
<td>$V_{ADC}(25^\circ C)=1.310V$</td>
</tr>
<tr>
<td>$V_{ADC}(450^\circ C)=1.605V$</td>
<td>$V_{ADC}(450^\circ C)=1.586V$</td>
<td>$V_{ADC}(450^\circ C)=1.602V$</td>
</tr>
<tr>
<td>$OUT_{ADC}(25^\circ C)=109$</td>
<td>$OUT_{ADC}(25^\circ C)=49$</td>
<td>$OUT_{ADC}(25^\circ C)=100$</td>
</tr>
<tr>
<td>$OUT_{ADC}(450^\circ C)=429$</td>
<td>$OUT_{ADC}(450^\circ C)=371$</td>
<td>$OUT_{ADC}(450^\circ C)=367$</td>
</tr>
<tr>
<td>$STEP=1.3^\circ C$</td>
<td>$STEP=0.76^\circ C$</td>
<td>$STEP=0.84^\circ C$</td>
</tr>
</tbody>
</table>

Table 4.1: Different configurations of the continuous time network

In order to avoid the loss of power, exploiting for example voltage dividers or MOS current generators to furnish the right amount of power in a continuous way to $R_H$, VDD is directly connected through a power p-MOS transistor to $R_H$ when the actual temperature of the sensor is lower than the desired one (set-point), while $R_H$ is disconnected otherwise. Due to the on-off topology of the temperature control a key parameter of the ADC is the conversion time, because between two successive updates of the output the digital comparator, the direction of the temperature variation
cannot be inverted. Approximating the trend of the temperature over time with a first order exponential equation, it is possible to obtain the maximum allowed conversion time in order to respect the desired temperature ringing of less than 1.5 °C. The first source of temperature ringing is the fact that it is not possible to completely bound the behaviour of the temperature inside the temperature range that correspond to an LSB of the ADC. How much the instantaneous temperature deviates from the desired value depends on the time interval between two successive updates of the output of the ADC. In Figure 4.9, the points when the A/D converter updates the output are underlined by vertical chopped lines. It is possible to note that, for example, when the temperature crosses the upper limit of the selected temperature, the system keeps furnishing heat till the successive update of the ADC output, increasing the amount of the temperature ringing.

Using different sensors with different values of $R_T$ or different values of $I_{DC}$ leads to a variation of the temperature interval that corresponds to an LSB. The following calculation are done with a typical configuration in which the LSB corresponds to 1°C. The unavoidable minimum ringing is therefore 1 °C and since the system needs to show an overall ringing smaller than 1.5 °C, we force by design $T_{CONV}$ to be as small as necessary to limit the temperature to move less than 0.5 °C between two ADC output updates. When the temperature is at very high values the slope of the temperature variation is the largest because the difference between the initial temperature and the temperature the system would go to (room temperature), is the maximum. At high temperatures the ringing towards even higher temperatures is
negligible because the difference between the initial temperature and the temperature the sensor is going to is much lower. By contrast when the temperature is around 300°C, in the middle between room temperature and the maximum reachable temperature, the ringing effect is important and almost equivalent in both directions. In the first mentioned case, of which an actual example will be reported below, the set-point temperature is 450 °C and the system on the basis of ADC output considers the temperature as the desired one while it is between 449.5 °C and 450.5 °C. When the temperature is over 450.5 °C the system stops to deliver power and the temperature starts to decrease. It happens that the ADC checks the temperature when it is just above 449.5 °C, at the time $t_I$, and so for an entire ADC conversion cycle the temperature will decrease starting from 449.5 °C. The temperature would decrease toward room temperature, $T_F$, around 25 °C, with the thermal time constant of the sensor, $\tau = 30$ ms. The maximum distance from the set-point temperature would be reached at $t_{UD}$ that is the instant occurring $\Delta_{\text{CONV}}$ after $t_I$. The maximum ringing, $T(t_{UD})-T(t_I)$ can be fixed to 0.4 °C with 0.1°C margin and thus $\Delta_{\text{CONV}}$ can be calculated.

$$T(t_{UD}) = (T(t_I) - T(t_F)) \exp\left(\frac{t_I-t_{UD}}{\tau}\right) + T_F$$

(4.8)

$$449.1 \, ^\circ C = (449.50 \, ^\circ C - 25 \, ^\circ C) \exp\left(-\frac{\Delta_{\text{CONV}}}{\tau}\right) + 25 \, ^\circ C$$

(4.9)

$$\Delta_{\text{CONV}} \approx 28 \mu s$$

(4.10)

In the second mentioned case, $T(t_I)=300^\circ C$, $T(t_{UD})=299.75^\circ C$, leads to $\Delta_{\text{CONV}} \approx 27 \mu s$. In order to grant flexibility with respect to the thermal time constant of the sensor, both considering technology drift among same brand of sensors and also that the efforts of sensors developers are in the direction of scaling down the power needed by decreasing the size and the mass of the sensors themselves, it has been decided to set $\Delta_{\text{CONV}}$ to 3 μs (an order of magnitude lower) which means that the output has to be updated with a frequency of about 300kHz. The basic ADC block diagram is reported in Figure 4.10. It is a first order
sigma-delta modulator, in which the digital decimator filter is an accumulator that counts the number of times in which the output of the comparator has been positive during the entire conversion cycle.

The main limitation of this solution is that, in order to respect the output throughput specification obtained above, by taking into account temperature ringing specifications, the circuit would have to work with a clock frequency around 100MHz, to perform a complete conversion in less than 3μs because 512 clock cycles are needed for 9-bit resolution. A circuit that work at such high frequency besides requiring a challenging design, dissipates a larger amount of dynamic power and also serious switching noise interference problems may arise in the chip between the high frequency temperature control circuit and the low frequency sensor read-out circuit. A solution to overcome these problems, paying in terms of area occupancy, is to develop a more complex decimation digital filter at the output of the comparator, to relax the specifications for the rest of the circuit. This solution, shown in Figure 4.11, presents 32 counters that process the output of the comparator. Every counter starts to count 16 clock cycles after the previous one and, as a consequence, the output of adjacent counters will exhibit a timing offset of 16 clock cycles.

Figure 4.10: Block diagram of the employed sigma-delta ADC.
A logic control circuit, that drives the counters and selects the last updated output for the following block driving a MUX, has thus necessarily been developed. This block has been designed as shown in Figure 4.12.

It is a simple but robust control circuit composed by two counters: the first one is a 4 bit counter that works on the rising edge of the clock, while the second one is a 5 bit counter that works on the falling edge of the clock. Only when the output of the first counter is equal to “1111” the AND network will enable the second counter, which will change its output producing the control signal for the MUX gate.

In order to limit the frequency of the control signal that drives the power p-MOS a finite state machine (FSM) has been added between the system set-point digital comparator and the power p-MOS. The FSM is characterized by 1024 states and during one cycle the state C and B appear just once, while state A appears 1022 times. The function of the FSM, depending on the state, is reported in Table 4.2.
Table 4.2: FSM state characterization

<table>
<thead>
<tr>
<th>State</th>
<th>Output</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>NOT(IN)</td>
</tr>
<tr>
<td>B</td>
<td>0</td>
</tr>
<tr>
<td>C</td>
<td>1</td>
</tr>
</tbody>
</table>

The effect of the FSM is to force the control signal to change at least two times every 1024 clock cycles, that correspond to about 2\(\mu\)s, as may be seen in the example of Figure 4.13. In this way the minimum frequency component of the frequency spectrum of the control signal, after the DC, is 5 kHz. A signal at 5 kHz is at a frequency high enough to be attenuated by the nominal thermal time constant of the sensor, so it does not affect the temperature or gas resistance measurements.

![Figure 4.13: FSM timing example.](image)

REFERENCES:


Chapter 5

Temperature control circuit details and measurements results

5.1. Introduction

This chapter reports the gas-sensor temperature control circuit details: in particular the power stage, the conditioning network and the ADC will be presented. The chapter continues showing the layout, the measurement set-up, the labview interface, that drives the chip through a FPGA, and the chemical test-bench. The chapter ends showing the measurements that have been carried out to validate the design and the chip.
5.2 Gas sensor temperature control overview

The block diagram of the sensor temperature control circuit, already shown in chapter 4, is reported again for simplicity in Figure 5.1. Every block of the control circuit will be presented in detail in the next paragraphs [1-4].

![Sensor temperature control block diagram](image)

Figure 5.1: Sensor temperature control block diagram.

5.3 Controller and actuator

The aim of the controller and actuator part of the circuit is to let the current flow from VDD to GND through $R_H$ when it is necessary to increase the sensor temperature, reducing to the minimum the power losses. For this reason, the W/L ratio of the p-MOS transistor, that connects to VDD when required the terminal of $R_H$ not connected to ground, is very important, because it determines the on-resistance of the transistor in linear region (triode). The rule of thumb in dimensioning the W/L of the power transistor has been first to set the length of the transistor at a quite low value, but not at the minimum of the technology, in order to increase the device life, which may be reduced by short channel effects, like hot carriers. In our design we selected a
value of \( L = 0.5\mu m \), with a technology with \( L_{\text{min}} = 0.35\mu m \). Then, the width of the transistor has been chosen in order to grant about 1\(\mu m \) for 10\(\mu A \) of heater current. Since the maximum average power over time for the highest temperatures is of the order of 100mA, the width has been set to \( W = 10000\mu m \). This huge geometry has been split in 5 parallel modules of 50 fingers each, leading to a finger width of 40\(\mu m \). This grants an on-resistance \( R_{\text{on}} \) of about 1.7\(\Omega \). Being the nominal resistance value of the heater about 30\(\Omega \), this is acceptable in terms of power efficiency.

![Figure 5.2: p-MOS power switch](image)

The shape of the drain and source power rails metallization has been realized with a V-shape, as highlighted in Figure 5.3, in order to optimize the current density in the rails themselves.

The switch, because of its large size, shows a great capacitance to its driving circuit. The capacitance has been calculated considering the unitary capacitance per unit area, given by the technology datasheet:

\[
C_{ox} \approx 3 \frac{fF}{\mu m^2}
\]  

(5.1)

Area and \( C_{ox} \) lead to an overall capacitance that is around 15pF, called \( C_f \), that cannot be driven in a reasonable time by an elementary buffer or inverter. In order to have a rough idea of the number of inverters necessary in the chain, the calculation to minimize the delay time an input signal needs to get through the inverter chain has been done.
The output gate of the digital control circuit is an elementary inverter, named INV.0 in the schematic in Figure 5.4, with a gate capacitance around 3 fF referred later as \( C_i \). The output of the control stage, is at high level when the set-point temperature is higher than the actual temperature, i.e. when the switch needs to be closed. For this reason the number of inverters that follow INV.0 has to be an odd number and then the number of stages, \( N \), that guarantees the minimum delay between output and input is the nearest even number given by the formula:

\[
N = \ln \left( \frac{C_f}{C_i} \right)
\]  

(5.2)
The optimum number of stages turns out to be 8 and the inter-stage dimensions ratio, \( R \), is:

\[
R = \left( \frac{C_f}{C_l} \right)^{\frac{1}{N}}
\]

leading to a value for \( R \) that is around 2.88. The simulations run with 8 stages have shown a response time extremely low, but the area occupied by the inverter chain is large, almost comparable to the power switch area. From simulations, the best compromise between area and response time turned out to a solution with \( N \) equal to 4 and hence three inverters between INV.0 and the switch, with \( R=8.32 \) exploiting again equation 5.3 with \( N=4 \). The transistor length is 0.35\( \mu \)m, the minimum, for all the transistor (Table 5.1).

<table>
<thead>
<tr>
<th>INV.0</th>
<th>INV.1</th>
<th>INV.2</th>
<th>INV.3</th>
</tr>
</thead>
<tbody>
<tr>
<td>( W_P=1.8 \mu m )</td>
<td>( W_P=15 \mu m )</td>
<td>( W_P=125 \mu m )</td>
<td>( W_P=1040 \mu m )</td>
</tr>
<tr>
<td>( W_N=1.18 \mu m )</td>
<td>( W_N=10 \mu m )</td>
<td>( W_N=80 \mu m )</td>
<td>( W_N=680 \mu m )</td>
</tr>
</tbody>
</table>

Table 5.1: Inverter chain dimensions

In this way the magnitude order of the response time changed from microseconds, obtained with elementary inverter, to nanoseconds. The layout detail of the inverter chain is shown in Figure 5.5 while the figure 5.6 shows together the layout of the inverter chain and of the p-MOS switch to give the possibility to compare their area.
5.4 Conditioning network

The conditioning network block diagram, shown in Figure 5.6, is composed by two parts: a digitally programmable current generator (DAC) and an operational amplifier. The aim of the DAC is to allow the use of different gas sensors.
characterized by different $R_T$ or zoom in or out the temperature range that is under test. This gives the possibility to control the temperature over a wide range with a certain precision or to control the temperature over a narrower range but with a higher precision, as already discussed in chapter 4.

![Digital Conditioner Diagram](image)

**Figure 5.6:** Continuous time conditioning network connected to the ADC.

The DAC has been realized by using 4 cascaded current mirrors, that provide 1.6mA, 0.8mA, 0.4mA and 0.2mA, respectively. Figure 5.7 shows a cascode current mirror and also the circuit that switches it on or off. With this circuit it is possible to bias $R_T$ with a current that varies from 0.2mA to 3mA. The accuracy of the ratios between current mirrors is not very important, because during one measurement the input code of the DAC remains constant.

It is very important instead that $I_{DC}$ remains constant with the variations of $R_T$ due to the temperature changes.
Figure 5.7: Thermometer bias DAC detail for a single current generation.

The purpose of the operational amplifier is to adapt the voltage swing of node $V_+$ to the input voltage range of the ADC. The same effect could be obtained by changing $V_A$ and using a DAC with currents 7 times larger, but in this way the power consumption due to the thermometer would increase more than the current drawn by the additional amplifier. The implemented amplifier, shown in Figure 5.8, is a two-stage structure with Miller compensation. The current flowing through the second stage ensures a good driving capability and hence it is not necessary to implement a source follower output stage. In fact, the output impedance thanks also to the feedback is low enough. The gain of this amplifier is around 60dB, the gain bandwidth product is 280MHz with 1pF capacitive load, the phase margin is 70° and the power dissipated is about 3mW. Such a large bandwidth and driving capability are required to handle the kickback of the input stage of the ADC. In fact, during every clock cycle, the input capacitance of the ADC (2pF) must be charged with complete settling in 100ns.
5.5 ADC

The analog core of the A/D converter is reported in Figure 5.9 [5, 6]. It is a switched capacitor ADC that consists of a fully-differential resettable integrator followed by a comparator. The input sampling network of the integrator has also the function of converting the single-ended (s.e.) signal into fully differential (f.d.). The operational amplifier, being fully differential, needs a common mode feedback circuit, also implemented with a switched-capacitor circuit, not reported for simplicity.

Figure 5.8: Operational amplifier schematic.

Figure 5.9: ADC analog core.
The amplifier embedded in the integrator is based on a folded cascode topology, as shown in Figure 5.10. It achieves a full scale linearity performance in switched capacitor signal processing of 79dB when driven by 1MHz clock. It exhibits a DC-gain of 79dB and a unity-gain bandwidth of 85MHz, while consuming 500µW including bias branches.

![Figure 5.10: ADC folded cascode operational amplifier.](image)

Particular care in operational amplifier design has been taken for the generation of the bias voltages of the folded cascode active loads in order to improve the dynamic swing performance of the amplifier which is, in simulation, 3.5V peak-peak differential, with 3.3V supply. The phase margin is 57°, when the capacitive load is \( C_f = 1\, \text{pF} \). Figure 5.11 shows the schematic of the latched comparator used in the ADC, which nominally consumes 40µA. Different timing are used in the two paths of the integrator input circuit, to implement the s.e. to f.d. conversion. Furthermore, during PH2 the comparator samples the signal from the integrator, while it updates its output on the rising edge of PH1, actually selecting the reference value \( V_{\text{LOOP}} \), which will be subtracted from the input signal in the successive cycle. After 512 clock periods the value stored in the 9-bit register, incremented of one unit every time the output of the comparator is high, gives the digital converted value of the single ended input \( V_{\text{in,s.e.}} \).
5.6 Layout

A picture of the layout of the chip is shown in Figure 5.12. The chip, realized in a 0.35μm technology occupies an area of 10mm². There are 94 PADs, divided in 3 groups, on the basis of the signals they deal with. The PAD subcategories are power, analog and digital and they have been separated to minimize interferences and cross-talk noise in general. Starting from the left corner of the layout the first block that has been highlighted is the “Heater Power Switch” that includes the p-MOS power switch and its driver. The second block, called “Probe MUX”, is used during test to allow checking the voltage level of some interesting nodes of the circuit and to help to understand the cause of an eventual disturbance. The block called “Voltage References”, creates the voltage references necessary for the whole chip starting from on-chip precise resistive dividers. Only a reference current of 200μA has then to be delivered from the test board.
The block called “Heater CTRL” is composed by a digital comparator that subtracts the 9 bit output of the ADC from the 9 bit set-point and by the finite state machine that processes the output of the comparator. The “Output Mux” is characterized by one bit selection, 32 inputs and 16 outputs. Thanks to this block it is possible to chose to get out of the ASIC either the output value of the ADC that represents the measured temperature of the sensor or the output of the “Chemical Sensor Read-Out” that is a fixed-points 16 bit word that represents the value of the gas sensor resistance. The block referred as “Gain Restorer” in the picture is the DAC which biases the thermometer, followed by the amplifier that adapts the thermometer swing to the ADC input range. In the “9-Bit Thermometer A/D Converter” it is possible to note that the digital part occupies most of the chip area. This is not really a problem because this first test chip is pad-limited due to a large set of test selectors. In a final device the ADC digital area would not again be a problem, since it will be small.
compared to the pattern recognition algorithm circuit. A photograph of the chip is shown in Figure 5.13. Unfortunately, most of the circuit is covered with metal dummy fillers, but still it is possible to recognize some of the parts, comparing this picture with Figure 5.12.

![Photograph of the chip](image)

Figure 5.13: Photograph of the chip.

### 5.7 Measurements set-up

An important feature that was crucial to meet was the easiness for the user to interface with the chip and to reduce to the minimum the number of laboratory instruments necessary to drive it. The chip can be totally controlled by a compact USB-based FPGA board featuring a Xilinx Spartan 3 FPGA software, controlled by a Labview® routine. This board has been used to generate and deliver the clock signals necessary to drive both the temperature control part and the read-out part of the chip, to set in real-time the desired temperature set-point for the gas-sensor and to acquire
the output of the digital output bus, which may be either the measured temperature of the sensor or the value of the gas sensitive resistance. A photograph of the control board is shown in Figure 5.14 (the green one on the left). On the same picture there is also the printed circuit board (PCB) that hosts the integrated circuit.

The on-board PLLs are very flexible and can be set to obtain directly signals between 9kHz and 400MHz. The clock necessary for the read-out circuit should be tunable and of the order of 2.5kHz. It has been obtained generating a clock at 320kHz and dividing by 128 its frequency by using 7 flip-flop contained in the FPGA. Choosing a different main carrier or enabling a different number of registers, the read-out circuit clock frequency may be changed, thus varying the measurement range, according to the description of the interface circuit given in chapter 3. The clock for the temperature control circuit could be between 1MHz and 5MHz. The more the clock is
fast the more frequently the output of the ADC is updated, but at higher frequency the linearity of the ADC slightly decreases and, furthermore, cross-talk with the precision read-out circuit increases. The clock that allowed to achieve the best results is around 3.5MHz. The PLL configuration is reported in Figure 5.15, while Figure 5.16 shows the control screen of the USB-based FPGA.

![PLL Configuration](image)

Figure 5.15: PLL configuration.
Figure 5.16: USB-based FPGA control screen.

Figure 5.17 shows the calibration gas-sensing test-bench used to test the system in controlled atmosphere in dynamic constant flow mode. The micromachined SnO$_2$ thin-film device was introduced into a teflon-made test cell, which was placed in series with a multichannel mass flow programmer/display (MKS mod. 647B), driving four separate gas-channels connected to four distinct mass flow controllers (MFCs of MKS mod. 1179A), one for reference gas, a second for obtaining humidified air by saturation method and the other two for gases under test. The accuracy of each MFC was of 1% of its full scale and 1% of reading. In our measurements, dry air was used both as reference gas and as diluting gas to obtain CO mixtures in air at different concentrations (5÷50 ppm). All chemical measurements have been carried-out at the research center IMM-CNR of Lecce.
5.8 Measurements results

5.8.1 Gas sensor temperature control measurements results

Figure 5.18 and Figure 5.19 show the transient behaviour of the temperature of the sensor when the set-point is varied with steps of about 13°C. The measurements that have been reported here have been carried-out changing the set-point from 9-bit word combination 150 to about 325 with steps of 7. As a consequence, the temperature of the sensor, as can be seen from the figures, changed from about 140°C to 400°C, exploring all the temperature dynamic range of the sensor. Similarly, it is also possible to generate every kind of temperature profile shape having a spectral content within the low-pass frequency barrier of the sensor time constant.
Figure 5.18: Example of a temperature profile “stair” for a chemical measurement.

Figure 5.19: A particular of the temperature “stair” between two fixed values.

Figure 5.20 shows the steady state of the sensor temperature when the set-point is 264°C and the gas-sensor has reached the desired temperature and, thus, the temperature control circuit has only to deliver the average power necessary to keep the sensor temperature constant. The ringing around the mean temperature value is
about ±0.75°C and can be considered negligible, since it does not affect the gas-sensing operation.

The linearity of the system, measured over the range 190°C ÷ 400°C is fully compliant with gas-sensing requirements, since the error is always lower than 1.5°C, as reported in Figure 5.21.
The power consumption of the temperature control circuit is independent of the set-point temperature while the power necessary to maintain the sensor at a certain temperature is dependent on its value. The current drawn by the digital part is around 1mA while the current required by the analog part is around 8mA. The analog core of the ADC needs around 1mA, the resistance dividers about 1mA, the inverters chain around 0.5mA and the conditioning network needs about 1.5mA for nominal sensors. The remaining part of the overall analog current flows into the gas-sensing read-out circuit. In Table 5.2 the power efficiency of the temperature control circuit is reported for different set-points, taking also into account the power loss by the p-MOS driver. The circuit exhibits an efficiency of about 80% for nominal operating temperature of gas-sensors (around 300°C).

<table>
<thead>
<tr>
<th>Temperature [°C]</th>
<th>I_H [mA]</th>
<th>Efficiency [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>188</td>
<td>32</td>
<td>74</td>
</tr>
<tr>
<td>221</td>
<td>39</td>
<td>77</td>
</tr>
<tr>
<td>300</td>
<td>51</td>
<td>81</td>
</tr>
<tr>
<td>328</td>
<td>64</td>
<td>84</td>
</tr>
<tr>
<td>384</td>
<td>81</td>
<td>86</td>
</tr>
</tbody>
</table>

Table 5.2: Power efficiency

### 5.8.2 Gas sensor read out measurements results

Figure 22 shows the linearity performance of the chemical sensor read-out circuit over 5.3 decades range, obtained by an electrical measurement using different precision resistance samples to obtain a simulated controlled variation of the test resistance. The achieved precision is better than 3% over the entire range at low temperatures (dynamic range, DR = 136.5dB). The precision degrades at high temperature, being 6% at 400°C, DR=130.4dB. The precision is limited by unavoidable residual cross-talk noise generated by the temperature control system power circuit, that affects the read-out part. The reason that causes the precision at higher temperature to decrease is that the larger amount of power that needs to be
delivered to the sensor causes a larger switching noise on the precision read-out circuit. Particular care has been taken in layout design in order to minimize this degrading effect.

The MATLAB® routine that calculates read-out accuracy starting from a data set of results, corresponding to an input resistance sweep over the entire dynamic range is the following:

```matlab
clear;
R=[100e3 999e3 9.99e6 12e6 100e6 1e9]';
DATA(1,:)=[28770 2936 300 258 2129 21222];
SIGN(1,:)=[0 0 0 1 1 1];
DATA=DATA';
SIGN=SIGN';
alfa=1;
gamma=1;
Rmid=10e6;
Nbit=8;
L=length(R);
for j=1:L
    if SIGN(j,1)==0
        Rmeas(j,1)=alfa*Rmid*(2^Nbit/DATA(j,1));
    else
        Rmeas(j,1)=gamma*Rmid*((DATA(j,1)+1)/2^Nbit);
    end
end
Rmeas1=Rmeas(:,1);
Gappl=1./R;
Gmeas1=1./Rmeas1;
Gnorm1=Gmeas1./Gappl;
Gfitting1=polyfit(Gappl,Gnorm1,1);
GnormID1=Gfitting1(1)*Gappl+Gfitting1(2); Gfit1=GnormID1.*Gappl;
Rfit1=1./Gfit1;
ERRnorm1=Gnorm1-GnormID1;
ERRabs1=ERRnorm1.*Gappl;
ERRrel1=ERRabs1./Gmeas1;
ERRperc1=ERRrel1*100;
DoubleFit1=polyfit(R,Rfit1,1);
m1=DoubleFit1(1);
q1=DoubleFit1(2);
Rcorrected_m1=Rmeas1./m1;
Offset1=Rcorrected_m1(1)-R(1);
Rcorrected_mq1=Rcorrected_m1-Offset1;
figure(1);
semilogx(R, ERRperc1,'b');
```

The code that has been reported needs also to know if the resistance under evaluation is larger or not with respect to $R_{\text{mid}}$ (referring to chapter 3). This information is the
additional output bit of the chip called *OSC_WHICH* that tells if the value of the input measured resistance is either lower or higher than geometrical midrange \((R_{\text{min}} \cdot R_{\text{max}})^{1/2}\). Three measurements examples on different ranges have been reported in Figure 5.22, Figure 5.23 and Figure 5.24, respectively. Of course, enlarging the measured range window, the accuracy decreases.

![Figure 5.22: Relative error in gas sensor resistance measure on a wide range (10kΩ ÷ 2GΩ).](image-url)

**Figure 5.22:** Relative error in gas sensor resistance measure on a wide range (10kΩ ÷ 2GΩ).
Figure 5.23: Relative error in gas sensor resistance measure on a small range (5MΩ ÷ 500MΩ).

Figure 5.24: Relative error in gas sensor resistance measure on a medium range (500kΩ ÷ 500MΩ).
5.8.3 Chemical measurements results

The system has been chemically tested choosing a constant temperature of 350°C as working temperature. The sensor response to CO has been characterized by exposing it to different concentrations from 5 ppm to 50 ppm, alternating every different concentration with pure air, as shown in Figure 5.25.

![Figure 5.25: System response at different concentrations of CO alternated to pure air at constant working temperature of 350°C.](image)

In order to evaluate the capability of the SnO$_2$-micromachined sensor to detect small CO concentration variation as occurs in a real application, the same gas-sensing test was repeated without a recovery period in dry air after each gas concentration step and the results are shown in Figure 5.26.
The sensor features a low conductance ($10^{-9}$A), probably due to a very low SnO$_2$ layer thickness of about 50nm. Moreover, even if the response (defined as $I_{\text{gas}}/I_{\text{air}}$) to CO is not so remarkable, it is valuable and useful as signal for the detection of carbon monoxide. The calibration curve to CO is showed in Figure 5.27, where it can be observed a trend to saturation at high concentration. The linearity error in the response between the two test is below 8%.
Temperature control circuit details and measurements results

Chapter 5

Figure 5.27: Gas sensing system calibration curve at constant working temperature of 350°C

REFERENCES:


CONCLUSIONS

In this thesis the design and the characterization of an integrated read-out and temperature control interface circuit with digital I/O for a gas-sensing system, based on a SnO$_2$ microhotplate thin film sensors, has been reported. The research activity has been carried out since the beginning of 2006 within the Italian Government PRIN project 2005092937, entitled “Interface and control circuits for high-selectivity gas sensors operated with temperature pattern”. The integrated interface circuit have been entirely developed at the Integrated Microsystem Laboratory of the University of Pavia, while the complete gas-sensing microsystem has been realized in cooperation with the University of Rome Tor Vergata, and the Research Center IMM-CNR of Lecce, under the supervision of the University of Lecce as Project Coordinator. Modern odor recognition techniques for environmental electronic noses and micromachined based Tin Oxide sensors with embedded heater and thermometer are presented, before starting with the interface circuit design discussion. Then the gas-sensor read-out circuit able to operate without calibration is described. This circuit is a redesign of a state of the art interface circuit modified in order to achieve a larger resistance measurement range and better robustness with respect to switching noise due to digital and power switching circuits. This last feature is particularly important because in the presented chip a precision and a power circuit must live together on the same die. The performance obtained from this read-out circuit are compatible with the demands of the other modules of the microsystem. The throughput of 100Hz, available over more than 3-decades resistance measurement range, makes the developed solution suitable for all current environmental gas-sensing applications, including those requiring dynamic pattern recognition operation on acquired data. The precision obtained varies with the temperature that the sensor has to reach and also with the input range required. The highest dynamic range measured is 138dB. The accuracy of the temperature control, better than 1.5°C, and the ringing of the temperature in steady state, again lower than 1.5 °C peak-to-peak, are really
satisfactory. The chip has been tested with real sensors. Both electrical and chemical measurements have been presented and prove the effectiveness of the interface circuit. For instance, the microsystem is able to reveal the presence of 5 ppm of CO with a single sensor before pattern recognition processing. Latest algorithm studies from unit of Rome grant a detection of CO lower than 1 ppm employing 4 SnO$_2$ sensors, taking into account the specifications of the read-out circuit presented in this work. Algorithms and sensors arrays also for NO$_2$, CH$_4$, CO$_2$ and alcohols have been developed. Temperature modulated profiles study for gas sensors arrays in time and in space, exploiting the temperature control circuit reported in this work in order to improve selectivity is the next goal.
ACKNOWLEDGMENTS

This work has been carried out and funded within the PRIN project 2005092937, entitled “Interface and control circuits for high-selectivity gas sensors operated with temperature pattern”, issued by the Italian Government. Chemical measurements have been carried out at Research Center IMM-CNR of Lecce, Italy. Experimental results reported for sensor devices overview are again provided by Research Center IMM-CNR of Lecce. Special acknowledgment is also due to project partner unit of Rome Tor Vergata. University of Lecce unit was represented by professor Andrea Baschirotto, Director of the PRIN project.