

**5^{èmes} Journées Franco-
Espagnoles**
**5^{as} Jornadas Franco-
Españolas**



IBERNAM - CMC2

Micro-Nano Systems

November 25,26th 2010

Residència d'Investigadors

C/Hospital 64, Barcelona (Spain)

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Preface

From the Spanish Network of Micro and Nanosistemas IBERNAM we greet the assistants to this scientific event in Barcelona " The fifth Spanish -French meeting in nano-microsystems IBERNAM-CMC2 ".

This year IBERNAM with CNM-B host the join meeting with CMC2, the parallel French Network with similar objectives.

Since 2002, every two years (Madrid 2002, Biarritz 2004, San Sebastian 2006, Toulouse 2008) we hold a join meeting with our colleagues to discuss about the recent and common advances and to foresee future join projects.

The IBERNAM is born in 2002, as renovation of the former Spanish Group of Sensors-GES in order to incorporate the technologies of nano - microsystems and in this way set up small intelligent instruments capable of realizing functions of detection, measurement and performance on real systems, as well as vigilance, forecast and monitoring their evolution in very diverse environments and with wide applications of social impact from the health, safety, quality of life up to the exploration of the space.

From the associated groups, a high number of competitive projects of research: regional, national and international have been generated with valuable results, such as: patents, publications of impact, presence and organization of scientific prestigious congresses and promotion of the take-off of small enterprises of technological base (spin-off).

At present IBERNAM is formed by 27 National Groups of scientific and technological type with industrial presence, supporting a wide cooperation among groups, sharing material and personal resources, all this with the conviction of that we are following a adapted way to improve our knowledge and to promote the technological innovation.

In this meeting recent accomplishments and technological potentials in format of oral and posters are presented. It is important to emphasize the fact of that many of them belong to the collaborative projects between laboratories from both Networks.

We want express our gratitude to the Organizing Committee, local Committee (CNM-Barcelona) and the participants by helping the opportunity to celebrate together this meeting that we wish very fruitful for all.

M^a Carmen Horrillo Güemes
Current President IBERNAM

F.Javier Gutiérrez Monreal
Honorary President of IBERNAM

Programme

Thursday, 25th of November 2010

12h30 Reception

13h00 Opening

Carles Cané, CNM-CSIC, Chairman

Nicole Jaffrezic - Renault, President CMC2, LSA, Univ. Claude Bernard, Lyon

Javier Gutiérrez/Carmen Horrillo, President IBERNAM, IFA- CSIC, Madrid

13h30 Lunch and Posters

Session 1

15h00 Plenary 1 (France) **F. Baco-Antoniali**, IFP Lyon, "Trends and needs for microanalysis for industrial R&D"

15h30 Oral 1 (Spain) **A. Vilà**, U. Barcelona, "Microtechnologies for fully transparent transistors"

15h50 Oral 2 (France) **A. Salaün**, U. Rennes, "Synthesis of silicon nanowires for chemical sensor applications"

16h10 Oral 3 (Spain) **P. Pina**, U. Zaragoza, "Zeolite films and membranes on Si-based microstructures"

16h30 Coffee break and Posters

Session 2

17h00 Oral 4 (France) **P. Temple-Boyer**, LAAS-CNRS Toulouse, "Dialysis dose monitoring using ChemFET-based detection Microsystems"

17h20 Oral 5 (Spain) **D. Matatagui**, IFA-CSIC Madrid, "Love-wave sensors to detect low-levels of CWA simulants"

17h40 Oral 6 (France) **A. Errachid**, LSA University Lyon, "Unconventional fabrication technologies for the elaboration of Lab-on-Chip in electrochemical bio(chemical) sensors"

18h00 End of First Jour

20h30 Dinner (city center)

Friday 26th of November 2010

Session 3

- 9h00 Plenary 2 (Spain) **J.L. Pons**, Bioengineering Group CSIC Madrid, "Windows to the human nervous system"
- 9h30 Oral 7 (France) **C. Pijolat**, EMSE Saint-Etienne, "Current developments of gas sensors and associated microsystems"
- 9h50 Oral 8 (Spain) **M. Duch**, IMB-CNM-CSIC, Barcelona, "Micro-barcodes for biological applications"
- 10h10 Oral 9 (France) **R. Lakhmi**, IMS U. Bordeaux, "Functionalization of screen-printed PZT microcantilevers for gas detection"
- 10h30 Coffee break and Posters

Session 4

- 11h15 Oral 10 (Spain) **P. Marín**, IMA-UCM, Madrid, "Magnetic microwires and their applications"
- 11h35 Oral 11 (France) **K. Aguir**, IM2NP U. Aix-Marseille, "Fabrication and characterization of fluidic microsystems for gas detection"
- 11h55 Oral 12(Spain) **A. Romano**, U. Barcelona, "Integration of nanowires onto microhotplates for the fabrication of low power consumption and fast operated gas nanosensors"
- 12h15 Presentation of CDE-2011 Conference, Palma de Mallorca., E. García Moreno, CDE-2011 Chairman
- 12h30 Closing Ceremony, Emilio Lora-Tamayo, Director of IMB-CNM
- 13h00 Lunch

Plenary Presentations:

- Trends and needs for on-line micro analysis for R&D. **INNOVAL: Innovative Project for Micro Process Analytical Technology**

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- **Windows to the Human Nervous System**

J.L. Pons

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Trends and needs for on line micro analysis for R&D. INNOVAL: Innovative Project for Micro process analytical technology

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Keywords: Green chemistry. Process analytical chemistry

Over the last years, on line analysis has become currently used for the instrumentation of industrial plants (process monitor and control) as well as the equipment of research and development pilot plants (chemical, petrochemical, green chemistry, ...). In the last years, new technologies were put in the market like NeSSITM and miniaturized systems which enable new on line analysis concepts.

For industrial applications, numerous benefits of these new concepts can be foreseen : reduce greatly the response time, increase the safety level of analysers (reducing transport and volume of samples), decrease the detection limits (no sampling, no contamination), open the way for new optimisation applications in order to perform new measures at low investment cost, define new standards of components, reduce the cost of maintenance with reduction of spares, increase flexibility of analytical applications with easy addition or replacement of micro analysers using the ISA SP 76 standard.

Trends in R&D is toward downscaling and process intensification (micro reactors, string pellet reactors), which put new constraints on analytical systems. Downscaling results in less product available and requires the development of new analytical tools and strategies: bring the analysis directly on the process streams. To implement these tools in or as close as possible of the reactor, they must become smaller with no loss of performance.

For many years the Process Analytical Technology (PAT) has been of high added value for industry.

In situ analysis (at the process' heart) represents the future of industrial analysis nevertheless this technology faces difficulties to penetrate the industrial sites as the only tools currently available on the market are not adapted to the needs.

INNOVAL project aims at fostering innovation in the field of industrial analysis by catalyzing the development of adapted in situ tools. It gathers 13 partners from R&D (academic centers, instrumentalists) to the market (industrialists).

This large project shall directly benefit to the different partners involved:

- Process control improvement for technology users with a direct positive impact on competitiveness
- Value and jobs creation for the instrumentalists
- Tech transfer and skills improvement for the academic sites

This paper will present some major actual and future trends and needs for on line micro analytical instruments and micro captors for R&D (process intensification) and industrial plants. INNOVAL project aims at fostering innovation in the field of micro PAT will be also presented.

Windows to the Human Nervous System

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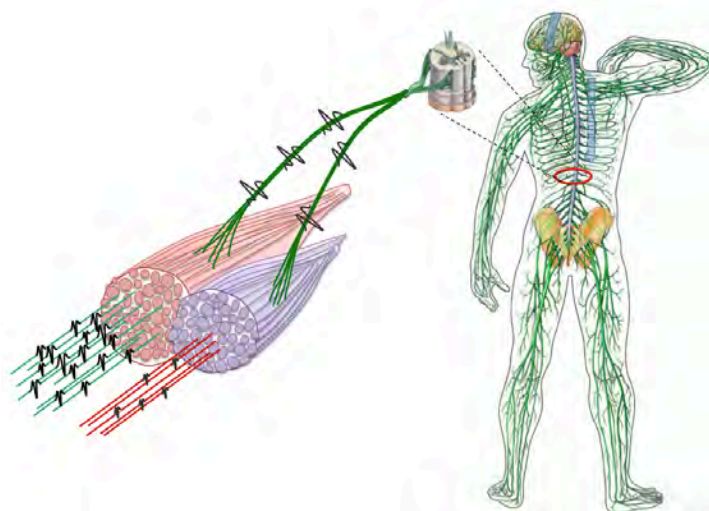
Keywords (up to 5 as maximum):

EEG, ECoG, ENG, EMG, Motor Neuroprostheses, Exoskeletal robots

Technologies for functional substitution in motor disorders have been applied in chronic phases of neurological conditions, whenever no functional recovery is possible [1]. Amongst these technologies, exoskeletal robots and motor neuroprostheses might find an application niche. Exoskeletal robots and motor neuroprostheses are technologies worn by patients, immediately thereafter issues related to wearability, safety and dependability emerge.

Target populations for both rehabilitation and functional substitution exoskeletal robots are patients suffering from neurological conditions leading to severe motor disorders, in particular cerebrovascular accident (CVA), spinal cord injury (SCI), cerebral palsy (CP) and pathological tremor. All these conditions lead to altered sensorimotor capabilities and one of the most disabling sequels is diminished locomotion capability.

Relaying information on intention and volition (in terms of desired motor tasks) to drive, command, and control a robot is a difficult task whenever it has to be achieved in a natural way. Intention to move is a cognitive process that may comprise high-level functions carried out by the human brain. The functions include comprehension, visual perception and construction, the ability to calculate, attention (information processing), memory, executive functions (such as planning, self-monitoring, and perception), and motor control [2].



In the context of motor control, this cognitive process leads to planning and execution of motor action, thus involving activity at central structures (i.e., brain and spinal cord) and peripheral structures (i.e., motor units and the musculoskeletal system), eventually leading to coordinated limb movement. Therefore, in principle, valuable information to decode human intention can be obtained from all these processes.

New trends are moving in the direction of sourcing the information directly from the human cognitive processes involved in the normal execution of tasks. These are called natural interfaces. We will briefly highlight the most salient characteristics of systems for relaying commands to the exoskeletal robot while sourcing the information from the stages of motor planning to motor execution.

Research in brain-machine interface (BMI) technology has expanded dramatically in the last decade, with impressive demonstrations of nonhuman primates and humans controlling robots in real time through signals collected from cortical areas. There are several important questions regarding the control of artificial actuators directly from brain-derived signals. These include the type of brain signals (single unit, multiple unit, or field potentials) that would provide the optimal control signal for the device and the number of channels that may be necessary to operate a BMI efficiently for long periods of time. Single- and multiple-unit recording interfaces are only attainable through implantable microarray electrodes. Surface and noninvasive BMIs based on field potentials are much more common in practical control of rehabilitation exoskeletons [3].

Natural control of wearable robots has been recently approached by directly interfacing with the human PNS. Several neural interfaces have been developed with different characteristics. Cuff electrodes have proven very reliable and robust, and they have the advantage of reduced invasiveness but suffer from limited selectivity. Sieve electrodes could present a very interesting solution for the development of neuroprosthetic and hybrid bionic systems, but still there are a number of problems limiting their usability, and they are only applicable to sectioned nerves.

The last step in the motor control process in humans is motor execution. Also, information obtained at this step can be used to relay control signals to the exoskeleton. In the context of this oral presentation these technologies are introduced as windows to the human nervous system on which rehabilitation technologies can be build.

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Oral Presentations:

- InkJet for large-area electronics: fully transparent transistors

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- Synthesis of silicon nanowires for sensor applications

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- Zeolite films and membranes on Si-based microstructures

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- Dialysis dose monitoring using ChemFET-based detection microsystems

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- Love-wave sensor to detect low-levels of CWA simulants

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- Unconventional fabrication technologies for the elaboration of Lab-on chip in electrochemical bio(chemical) sensors

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- Current developments of gas sensors and associated micro-systems

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- Micro-Barcodes for Biological applications

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- Functionalization of screen-printed PZT microcantilevers for gas detection

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- Magnetic microwires and their applications

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- Fabrication and characterization of fluidic microsystems for gas detection

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- Integration of Nanowires onto Microhotplates for the Fabrication of Low Power Consumption and Fast Operated Gas Nanosensors

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J. Santander(4), C. Calaza(4), L. Fonseca(4), C. Cané(4),

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InkJet for large-area electronics: fully transparent transistors

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Keywords (up to 5 as maximum): inkjet, large-area electronics, TFTs, transparent electronics, GIZO

Inkjet printing of functional materials initiated the developments for giving this technology an important role as an efficient, low-cost and versatile way to produce electronic circuits. This selective deposition process can address several different areas of electronic applications, such as solar cells¹, sensors², thin film transistors (TFTs)³, etc. and uses printing materials in form of a jettable solution (ink). Amorphous metal-oxide semiconductors have shown to give high performance electronic components, as despite their amorphous state can provide electrical properties superior to their covalent semiconductor counterparts. Additionally, these materials are transparent in the visible range, addressing an even wider range of applications in the field of transparent electronics.

In this work we present the capabilities of inkjet technology in the fabrication of printed circuits of thin film transistors based on the amorphous ternary Ga₂O₃-In₂O₃-ZnO (GIZO) semiconductors⁴. We examine the production of inks capable of providing functional GIZO semiconductors. By mixing the three basic inks under different ratios we obtain different compositions of the GIZO semiconductor that affect the electrical properties of the printed films.

In order to test the semiconductor's performance as an electronic component, TFT devices were fabricated by printing the desired materials on Si-SiO₂ or glass substrates. Tests showed good behaviour as transistor, generally with high threshold tension. Moreover, since one of the major applications of these devices is focused on displays, the same TFT devices were build along other electronic components such as light emitting diodes (LEDs) in order to assess their ability to control them and fabricate a fully functional electronic circuit.

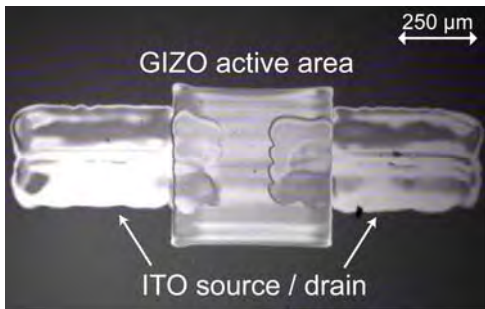


Fig. 1. Inkjet printed TFT on SiO₂/Si.

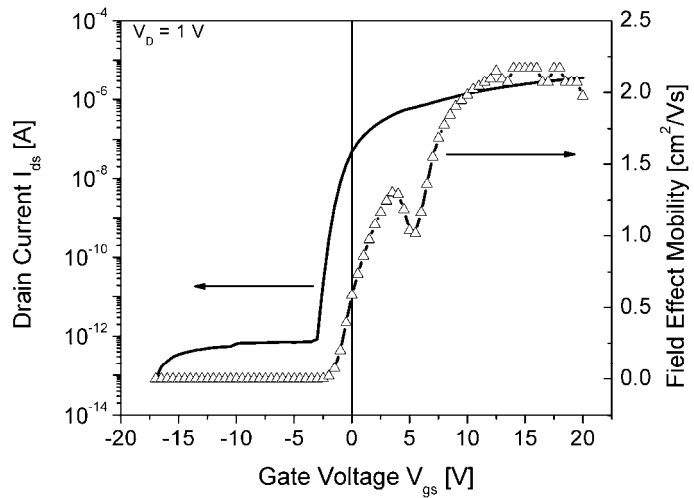


Fig. 2. Transfer characteristics and field-effect mobility of the printed TFT.

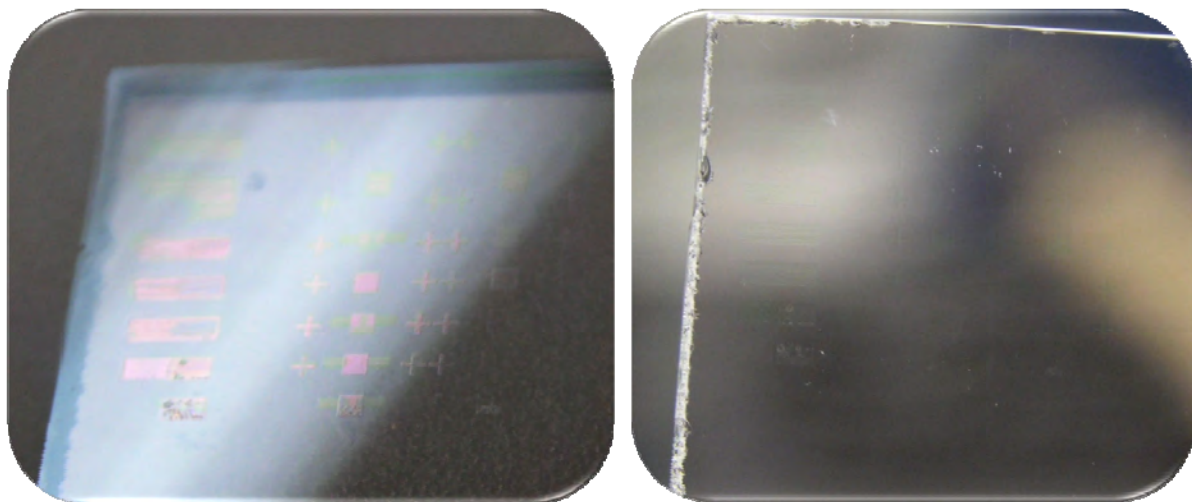


Fig.3. GIZO printed TFTs on glass/ITO/ATO

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Synthesis of silicon nanowires for sensor applications

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Keywords: Silicon nanowires, synthesis, gas sensor

Summary

Silicon nanowires (SiNWs) are synthesized following two methods: i) the VLS (Vapor-Liquid-Solid) growth technique (bottom up approach), and ii) the sidewall spacer realization (top down approach) commonly used in microelectronic industry. The VLS growth technique uses gold nanoparticles to activate the vapor deposition of the precursor gas and initiate a 100 nm diameter SiNWs network growth. In the case of the sidewall spacer method, a polysilicon layer is deposited by LPCVD (Low Pressure Chemical Vapor Deposition) technique on SiO₂ wall patterned by conventional UV lithography technique. Polysilicon film is then plasma etched. Accurate control of the etching rate leads to the formation of spacers with a curvature radius below 100 nm that can be used as polysilicon NWs. Each kind of nanowires is integrated into resistors. Electrical measurements in vacuum show the potential usefulness of these SiNWs as chemical sensors.

Motivation and results

SiNWs are currently attracting much attention as promising components for future nanoelectronic devices such as nanowire field effect transistors [1], photonic and optoelectronic devices [2], and more particularly as chemical or biological sensors [3, 4]. They can be prepared using various advanced methods such as e-beam, AFM or deep UV lithography. In this work, SiNWs are synthesized without requiring these previous costly lithographic tools.

SiNWs present large advantages: high surface to volume ratio, surface functionalization, synthesis compatible with large area silicon technology, leading to the development of innovative sensors. Main interest of SiNWs rests on their high surface that can be sensitive to charges. Two structures are used to fabricate devices acting as sensor for ambiance detection.

High density Au-catalyst VLS-SiNWs network is synthesized by LPCVD at 480°C and 40 Pa. The SiNWs grow with high densities anchored at the dedicated catalyst islands defined using classical optical lithography technique. Due to the length of the SiNWs which can exceed 20 μm, bridges are achieved to connect two heavily doped polysilicon islands leading to the formation of resistors in a 3D configuration as shown on figure 1. Another configuration is used to fabricate resistor in a coplanar structure by using polysilicon NWs made by the spacer method.

Contacts electrodes are made of aluminum thermally evaporated and defined by wet etching as shown on figure 2.

Figure 3 shows results of simple tests carried out on both structures that consists in exposing SiNWs to smoke or gas in a cryostat. Upon exposure, resistance is found to dramatically decrease. These first demonstrations serve only as proof-of-concept for a new kind of chemical sensor based on silicon nanowires. Indeed, SiNWs are of special interest because they are expected to play an important role as their surface can be modified with functional group.

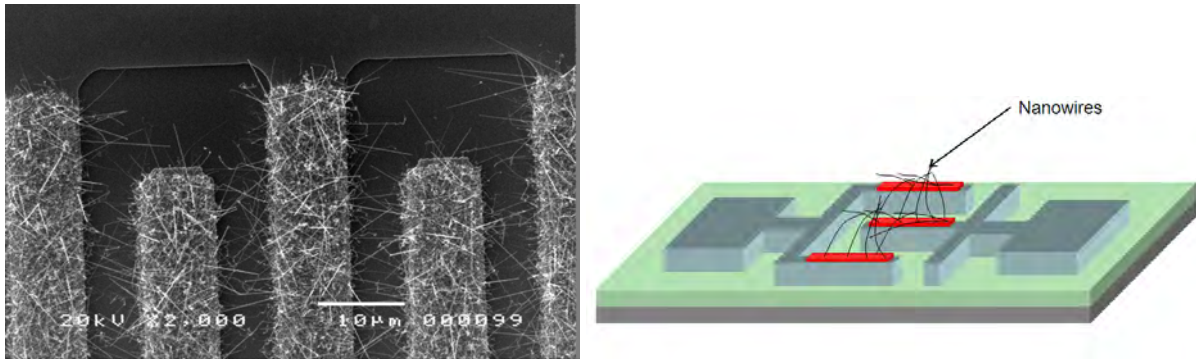


Fig. 1: VLS-SiNWs based resistor, (a) schematic view, (b) SEM picture (Au is deposited on local areas on half teeth)

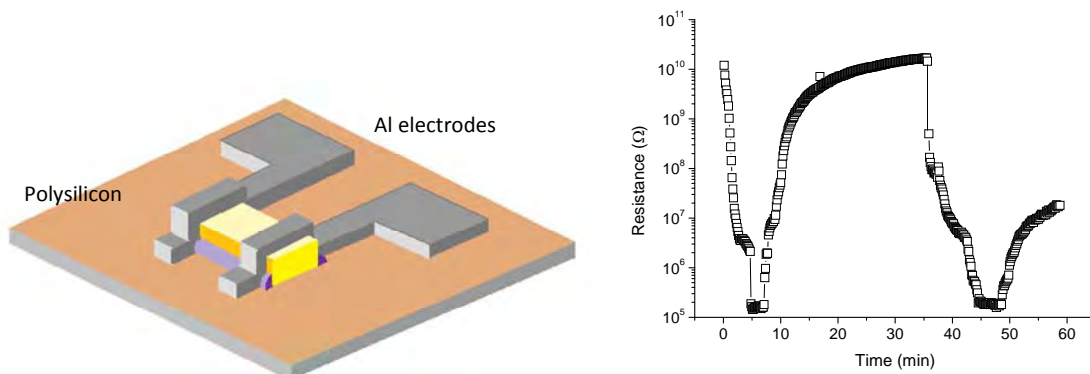


Fig. 2: Spacer method polysilicon NW based resistor, (a) schematic view, (b) Resistance variations versus exposure duration to smoke of the polysilicon NWs based resistor (spacer method)

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Zeolite films and membranes on Si-based microstructures

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Keywords (up to 5 as maximum): catalytic zeolite coatings, ultra-high surface/volumen ratio, microreactors, VOCs abatement

During the last two decades considerable progress has been achieved in controlling the growth of zeolite films of a varied nature, as described in a number of recent reviews [1-3]. These zeolite films constitute a special type of nanostructured interface capable of very specific interactions with individual molecules. Because of this, in many cases it is possible to implement molecular recognition functions, in addition to exploiting other well known zeolite properties such as their catalytic, adsorption and diffusion/permeation characteristics. Therefore, the characteristics of zeolite films can be very different depending on the intended application, and the preparation procedures have to be tailored accordingly. In particular, this work reviews the progress attained on zeolite synthesis over Si based microstructures.

Yeung and coworkers have developed several strategies for the preparation of zeolite layers onto micro-chemical systems and microchannels etched on silicon wafers [4-5] ranging from the simple de-position of zeolite powder in the microchannel to methods involving hydrothermal synthesis, which on a Si wafer presents significant difficulties of its own, related to chemical attack to the support under the highly alkaline synthesis conditions. In a different approach [5] a uniform zeolite layer was grown on the silicon wafer and then patterned in a variety of shapes using photolithography etching processes adapted from those employed in the semiconductor industry. The same authors [6-7] went on to coat 5 micron thick TS-1 zeolite layers on microchannels 33 mm long, 500 μm wide, fabricated onto silicon wafers. Different configurations of the microreactors (single channel, T-shaped and multichannel microreactor) were used for the epoxidation of 1-pentene.

In our group, photolithography processes have also been used to prepare zeolite-coated microreactors with ultrahigh external surface to volume ratios [8-9]. To this end, regular structures of SiO_2 micromonoliths or microneedles with a diameter of 3-5 microns has been created on silicon wafers by photoassisted electrochemical etching [8-9] and covered with a uniform zeolite layer using seeded hydro-thermal synthesis [10] (see Figure1) The thin zeolite coating (< 1 micron) provides a short diffusion path length, and the external area of the zeolite films is 400000-750000 m^2/m^3 of reactor volume, underlying the potential of these microstructures as highly efficient contactors. In an attempt to reduce hydrothermal synthesis time, and therefore broaden the family of zeolite structures capable to a successfully growth onto Si wafers, microwave- assisted heating is being fully studied. Figure 2 summarizes some of these recent results attained with ZSM-5 zeolite (Si/Al= 100).

The catalytic performance of the as prepared zeolite coatings onto Si micromonoliths microreactors after conventional Pt-exchange have been evaluated in the combustion of hexane at ppm level, and also compared with published literature indicating the potentialities of the system.

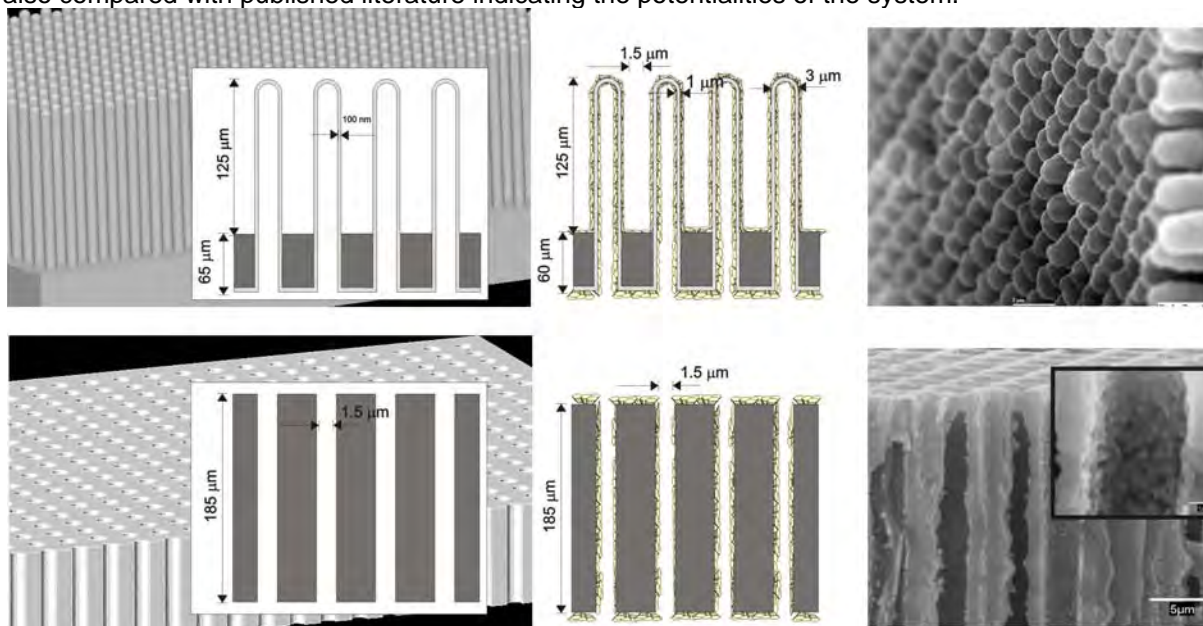


Figure 1. MFI type zeolite coatings by conventional hydrothermal growth onto Si-based: up) microneedles; and, bottom) micromonoliths.

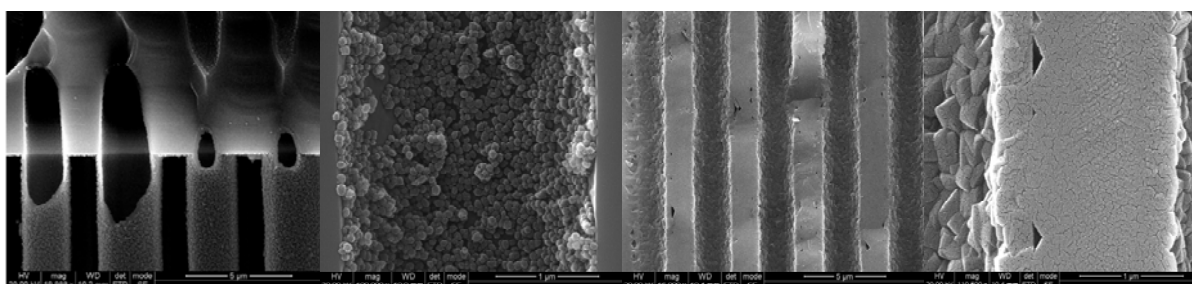


Figure 2. MFI type zeolite coatings by microwave-assisted hydrothermal growth (3 min synthesis time) onto Si-based microstructures. From left to the right: seeded surface by dip-coating, seeded surface by LBL technique with PDDA, intergrowth ZSM-5 zeolite layer top view, ZSM-5 layer thickness onto micromonoliths.

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Dialysis dose monitoring using ChemFET-based detection microsystems

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Keywords: ChemFET microsensor, urea detection, haemodialysis, health-care microsystems

Chronic end-stage kidney failure is a widely-spread health disorder and becomes therefore a priority for the development of health-care systems. In particular, the dialysis dose has to be quantified with the on-line monitoring of urea. This has been realised with enzyme-modified electrodes but expensive costs and non-usability have limited such developments^{1,2,3}. In this context, this paper presents the urea measurement in artificial dialysis conditions using low-cost, disposable pH-ChemFET-based microsensors.

In collaboration with the HEMODIA company, a technological process was developed in LAAS-CNRS cleanroom facilities. These works showed the feasibility of a ChemFET-based microsensor for detecting urea in biological solutions⁴. This fabrication process was optimised and transferred by the HEMODIA company in an industrial foundry in order to reach fully mass fabrication at low cost, reproducibility, and high fabrication yield⁵. Then, pH-ChemFETs were adapted to the urea detection with an urease-rich PVA layer deposited thanks to an automatic ink jet system. Finally, in order to perform differential analysis, both urea-ChemFET and pH-ChemFET were integrated on a flexible module with a gold gate electrode, and were packaged into a specific flow cell adapted to haemodialysis (fig. 1).

First experiments were applied to the pH monitoring. Similar detection properties (sensitivity: ~53 mV/pH) have been obtained for the pH-ChemFETs microsensors on wide concentration ranges, validating the ChemFET/ReFET differential analysis. Thus, urea microsensors were tested in EDTA-based solutions. The urea-ChemFET/pH-ChemFET differential analysis showed good urea detection properties (sensitivity: ~70 mV/pUrea) on the [1 - 25 mM] pathological range. Then, tests have been performed in dialysate solutions. Again, good urea detection properties have been obtained on the studied range even if lower sensitivity (~21 mV/pUrea) was evidenced. This decrease should be related to the dialysate buffer properties. Finally, the microsensors were tested in order to monitor continuously urea in artificial dialysis conditions. Thus, during the dialysate purification, the expected urea concentration decrease with time was characterized and a good fit was found with optical absorbance measurements (fig. 2).

In summary, the dialysis dose monitoring using urea-ChemFET microsensors has been successfully demonstrated for the first time. Such technology is fully compatible with any pH-related biochemical detection and can be extended to other medical applications.

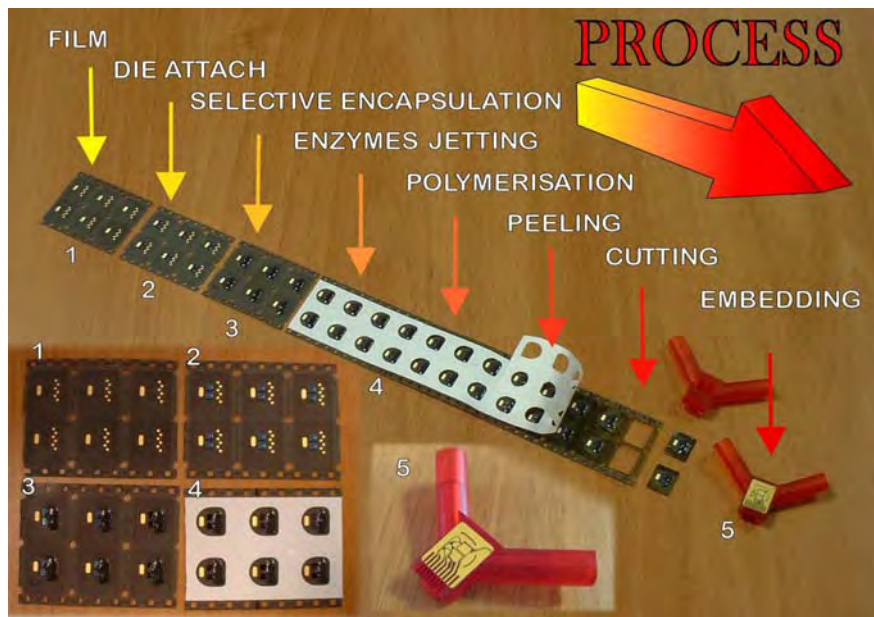


Fig. 1: urea-EnFET mass-fabrication

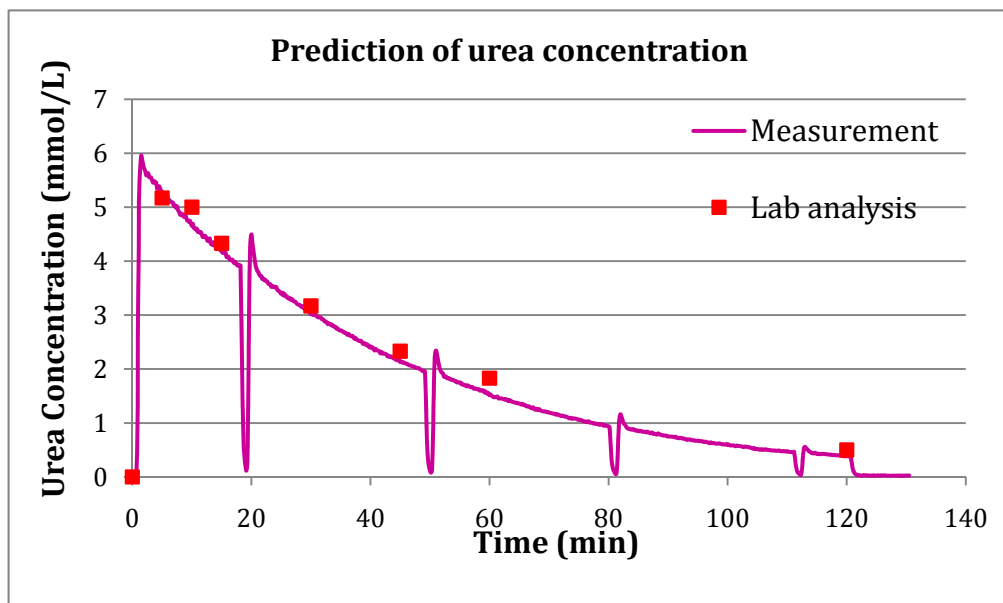


Fig.2: urea-ChemFET dialysis dose monitoring

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Low-wave sensor to detect low-levels of CWA simulants

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Keywords: Chemical warfare agents; Sensor array; Love wave; Novolac

Chemical warfare agents (CWAs) are highly dangerous for human health. Therefore, an early and clearly detection is of enormous importance for the people safety. An array based on Love-wave sensors has been developed, and specific polymer coatings have been deposited to detect simulants of CWAs [1].

The array has been made up of six Love-wave sensors which consist of a delay line (DL) composed of a ST-x 90° cut quartz piezoelectric substrate and a Novolac guiding layer [2]. Interdigitated traducers (IDTs) of 200 nm of aluminium are made by sputtering. A structure of four fingers per wavelength ($\lambda = 28 \mu\text{m}$) is repeated 75 times to form each IDT. Both, the spacing between IDTs and the acoustic aperture are 75λ . The guiding layer is a Novolac photoresist deposited by spinner and processed by photolithography, in such a way that a high uniformity and a low surface roughness are achieved. Sensor sketch is shown in Fig. 1. Different polymers (PCPMS, PECH, CARBOWAX, PDMS, PEI, PMFTPMS), [3], have been deposited by spin coating as sensitive layers with an approximately 100 nm thick.

The sensing properties of the CWAs have been investigated using their well known simulants: dimethylmethyl phosphonate (DMMP), dipropylenglycol methyl ether (DPGME), dimethylmethyl acetamide (DMA), dichloroethane (DCE), dichloromethane (DCM), dichloropentane (DCP) and toluene (TOL) and very low concentrations of them (Table 1) have been measured. A first set of measurements was made for testing sensors array performance. Fig. 2 illustrates the response of the PECH coated sensor for very low concentrations of DMMP, and the linear relation between frequency shift and the concentration. Fig. 3 shows that these simulants have been clearly discriminated by Principal Component Analysis (PCA).

Excellent properties with respect to stability, linearity, reversibility, fast response, repeatability and good sensitivity have been achieved for this kind of sensor coated with the polymers presented. Therefore, very low concentrations have been detected, such as DMMP 0.025 ppm and DPGME 0.25 ppm.

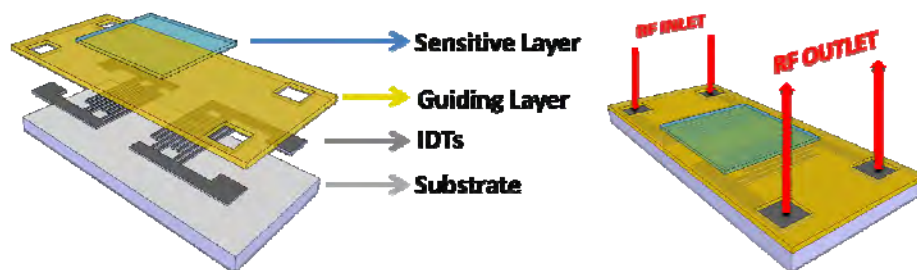


Fig. 1: Love-wave sensor.

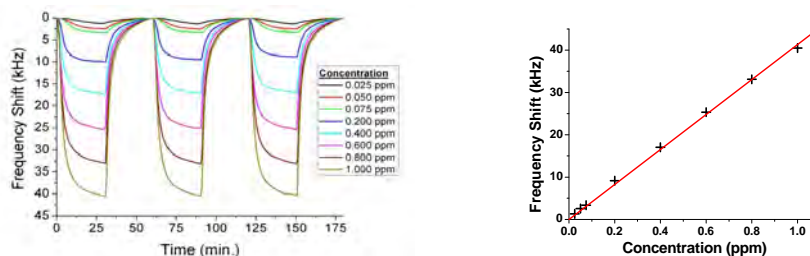


Fig. 2: Left, response curves of the sensor coated with PECH for DMMP and right, linear correlation between frequency shift and concentrations.

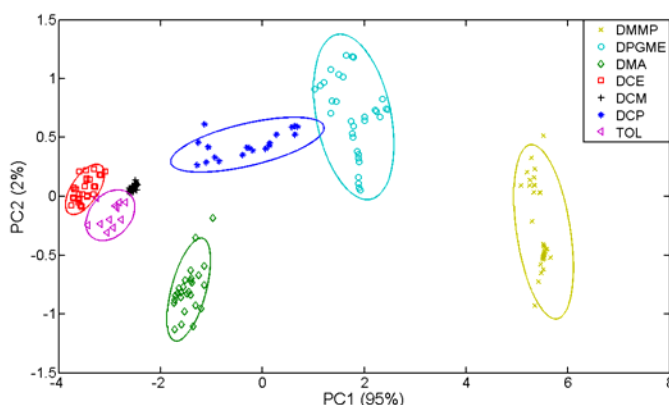


Fig. 3: Principal Component Analysis applied to data for discrimination of CWA simulants.

Table 1: Chemical warfare simulants.

ABBREVIATION	CONCENTRATION (PPM)
DMMP	0.025 – 1
DPGME	0.25 – 10
DMA	25 – 250
DCE	75 – 250
DCM	125 – 300
DCP	5 – 25
TOL	100 – 200

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Unconventional fabrication technologies for the elaboration of Lab-on chip in electrochemical bio(chemical) sensors

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Keywords: Lab-on Chip, Biosensors, PDMS

Lab-on-chip systems are nowadays one of the most interesting subjects of flexible devices especially for medical fields and biological applications. The elastomeric polymer Poly(dimethylsiloxane) also known as PDMS, is widely used almost for almost all manufactured microfluidic systems, and has become an important material for biomedical applications. Furthermore due to its biocompatibility, PDMS has several important properties such as its permeability, elasticity, good thermal stability and low surface tension [1]. Bonding PDMS with polymer substrate to make functional micro fluidic system is the principal problem of PDMS-polymer adhesion. To address this challenge, in this paper a new technique is presented that is designed to decrease fabrication time, cost, as well as many other process stages to manufacture microfluidic channels on polymer substrate. Biocompatible polymers used in this paper are polyimide (PI), polyethylene naphthalate (PEN), and polyethylene terephthalate (PET). These three polymers are chosen for their many attractive material properties, such as their excellent thermal and mechanical stability, high chemical resistance, and good optical properties [2-3].

The substrates with gold electrodes were fabricated on PI. This is done by absorption of thiols onto structured PDMS (a,b), followed by microcontact printing onto gold deposited PI substrates (c) leaving the desired pattern (d) for wet etching to produce the microelectrodes (Fig 1). After making the microelectrodes on the polyimide substrate, the microfluidic systems based on PDMS was bonded on polymer substrates (PI, PEN and PET) by Si-o-Si binding between inorganic silane groups of PDMS and silane groups which have been grafted on polymer surfaces by 3-mercaptoptrimethoxysilane (3-MPTMS) treatment, and followed by oxygen plasma treatment. The PDMS was treated then by oxygen plasma to generate OH group on its surface, and brought immediately after plasma treatment on the surface of the treated polymers. The adhesion between polymers-PDMS was defined first by concentration of 3-MPTMS, and the time of oxygen plasma treatment of PDMS and 3-MPTMS polymers treated (Fig.2).

The developed Lab-on-Chip will be used for the detection of clinical inflammation of pro- and anti-inflammatory cytokines that are experienced within heart failure (HF) patients. The interaction of biomarkers with the antibodies and the antibodies immobilized onto functionalized gold microelectrodes, will be measured by impedance spectroscopy.

Acknowledgements

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Figures:

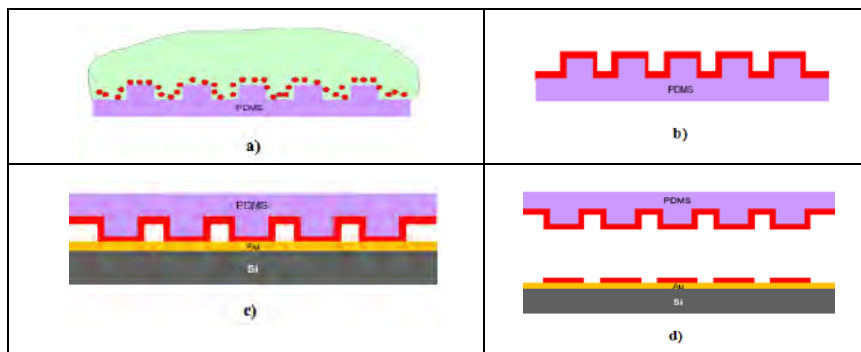


Fig. 1: Micro contact printing process

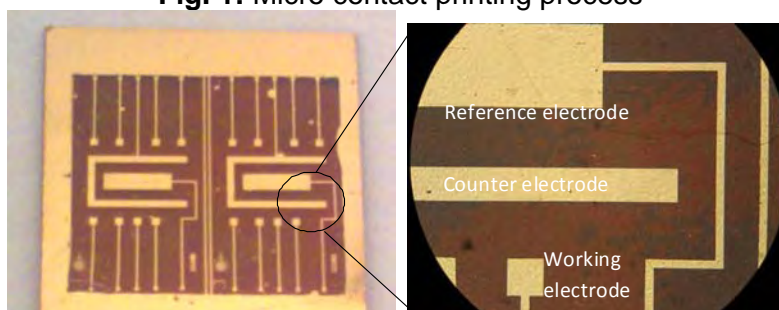


Fig. 2: Gold Micro electrodes on polyimide substrate fabricated by microcontact printing.



Fig. 3: Optical image of the Lab-on-chip

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Current developments of gas sensors and associated micro-systems

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Keywords: gas sensors, preconcentrators

The presentation concerns the studies on gas sensors and associated micro-systems which are currently performed at the department MICC (Microsystèmes, Instrumentation et capteurs Chimiques) du centre SPIN.

A part of the activity is focussed on the development of gas sensors for automotive exhausts. On one hand, planar potentiometric NO_x sensors based on a solid electrolyte YSZ and metallic electrodes (platinum and gold) are fabricated by using screen-printing technology. The sensing properties were tested in a laboratory test bench for different concentrations of NO and NO₂ in oxygen rich atmosphere at 400-550°C. Interferences from co-existing gases in automotive exhaust such as CO and light hydrocarbons could be successfully eliminated by depositing a catalytic filter layer over the sensing elements.

On the other hand, tin dioxide gas sensors are also tested for automotive exhaust gas application. It consists in elaborating robust sensors on alumina substrate by screen-printing technology. Sensors are tested on a synthetic gas bench which is able to produce high gas velocity and high gas temperature near those of real exhaust gas conditions. Results show the possibility to detect a hydrocarbon like C₃H₈ for high sensors temperatures (600°C) while sensitivity to NO₂ is low.

Another application concerns the use of a mini fuel cell for the detection of low or high levels of CO in the reformat gas. A prototype of a miniaturized fuel cell has been studied in order to detect carbon monoxide in hydrogen-rich atmosphere for PEM-FC (Protonic Exchange Membrane fuel - cell) applications. It consists in a home-made Membrane –Electrode – Assembly (MEA) developed by the CEA. Experiments have been carried out on a laboratory testing bench with simulated reformed gases. For low CO concentrations (≤ 20 ppm), an amperometric mode is suitable but regeneration in air is necessary to obtain a good reversibility of the sensor response. On the contrary, for higher CO concentrations (250 to 4000 ppm), a good reversible response is observed without air regeneration by using a potentiometric or quasi-potentiometric mode.

The presentation will also include the development of a gas preconcentrator based on a micro-channel in porous silicon filled with carbon nanopowders by a micro-fluidic process. The particularity of this device is its applicability in the fields of atmospheric pollution monitoring by targeting VOCS (volatiles organic compounds). Various designs of micro-devices have been investigated and a special focus has been dedicated to the carbon adsorbent. The optimization of the device and its operation were driven by its future application in outdoor environments. The benefits of using porous silicon to ease the fixing of the carbon adsorbent in micro-channels and to modify the gas desorption kinetic are also investigated. Results on a device based on a carbon adsorbent powder filled in a porous silicon micro-channel for benzene preconcentration are presented.

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Micro-barcodes for biological applications

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Keywords (up to 5 as maximum): Polysilicon, silicon, barcode, microfabrication, embryo labeling

1. Abstract

Single cell labeling and tracking is growing interest in cell biology. The development of small barcodes will allow investigating single cell behavior under optical microscopes. These codes have to fulfill special biological, optical and fabrication requirements. Nowadays, silicon microtechnologies advances allow fabricating small structures. In this paper, we show the design and fabrication of polysilicon barcodes based on standard MEMS technologies and their feasibility to be used as intracellular cell barcodes.

2. Introduction

In the last decade, it has been a growing interest in developing different barcodes systems to track cells, and nowadays, the development of in vitro reproduction techniques has presented a great concern about the identification of the embryos [1][2]. Typically, systems to track cells were designed to label subpopulations of cells with individual characteristics, as metallic barcodes [3] or quantum-dots microbeads [4]. However, many of them require the use of fluorochromes and their visualization at confocal scanning laser microscopes. In this work, we propose the use of new small biocompatible 2D and 3D silicon based barcodes for cell tracking and embryo labelling.

3. Design and Fabrication Process

Two different kinds of silicon based barcodes were designed based on standard semiconductor techniques (Fig 1). The 2D devices consist of a start marker and 8 digits that represent 8 bits, giving 256 different encoding values. The device dimensions were fixed to 10 μm x 6 μm x 1 μm . The smallest features were limited to ~ 1 μm close to the resolution limit of light microscopes. A 1 μm thick silicon oxide layer is used as sacrificial layer and a 1 μm thick polysilicon layer as a device layer. After device patterning, the barcodes are released by a sacrificial etching in HF vapours and finally collect in a biocompatible medium.

4. Applications

Cell tracking

As a proof of concept, silicon barcodes were internalized on human macrophages differentiated in vitro, taking profit of their phagocyte capability. Figure 2 shows a barcode read by optical microscopes, demonstrating their functionality. In this image we can also see a schematic representation of the barcode internalized by the living cell. In addition, our biocompatibility studies of internalized codes report similar values in survival rates of macrophages with and without phagocytosed silicon barcodes.

Embryos Micro-Labeling

2D and 3D barcodes have been introduced into mouse embryos by microinjection technique. The barcodes were kept between the pellucid zone and the cell membrane without affect the embryo cell division. In all cases was possible to read the binary code by optical microscopy. In addition, cryopreservation studies show that the barcoded embryos can continue their development after this process.

5. Conclusion

Preliminary results evidence the usefulness of these barcodes for future applications as single cell tracking and embryo labelling. At the same way we demonstrate the possibility to employ semiconductor fabrication techniques to produce different types of biocompatible silicon based barcodes to be use in cell biology.

[Additional text, graphs or images]

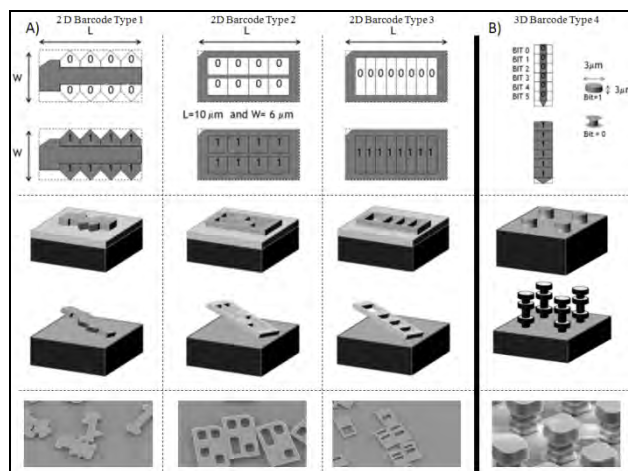


Fig. 1: A) Schematic view of 2D silicon barcodes, the asymmetric corner indicates the starting point to the barcode reader. Below, schematic view of 2D barcode fabrication process: first silicon wafers are used as starting material, second silicon oxide layer is deposited as sacrificial layer. LPCVD polysilicon oxide layer is deposited as structural layer. Finally, barcode patterning is achieved by a photolithographic step and a polysilicon dry etching. Then the barcodes are released in HF vapors. B) Schematic view of 3D silicon barcodes design. Below, schematic view of 3D barcode fabrication process: First, Silicon wafer are used as substrate, then a silicon oxide layer is patterned and used as mask for silicon DRIE with vertical profile for bit=1, and non-vertical etching for bit=0. Finally the barcodes are release by long isotropic overetching. At the bottom, SEM images of different types of fabricated barcodes are shown.



Fig. 2. Optical image a human macrophage with an intracellular silicon barcode and schematic view of the internalized code with its binary and decimal representation.

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Functionalization of screen-printed PZT microcantilevers for gas detection

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Keywords: microcantilever, thick-layer, benzene, polyaniline, polypyrrole, active carbon, tin oxide

The gas detection at a low level (ppm to ppb) is a great challenge in order to guarantee safe environments with low pollution and prevent against toxic gases at low concentrations, such as methanol, toluene, nitric oxide, benzene, etc. Because of its high sensitivity and low power consumption, resonant piezoelectric cantilever sensor has been chosen for this purpose (Fig. 1a) [1,2]. Furthermore, the high resonance frequency of the 31-longitudinal mode of the PZT-cantilever might lead to good gas sensitivities. This has been achieved with screen-printed PZT cantilevers shown on Figure 1b [3]. Electrical measurements of poled samples confirm the piezoelectric behavior of cantilevers and the presence of longitudinal modes. The resonant frequency and the quality factor of a PZT beam of 8mm x 2mm x 0.05mm, which are respectively $f_R=65\text{kHz}$ and $Q=300$ for the longitudinal mode, justify the choice of the latter for gas sensitivity (Fig.2). In order to evaluate the mass sensitivity of screen-printed PZT cantilevers before benzene detection, toluene absorption on PEUT polymer film (partition coefficient of 1000) has been undertaken. For PZT cantilevers coated with a sprayed $44\mu\text{m}$ PEUT layer, a linear variation of the frequency shift with the toluene concentration is obtained for the first and second longitudinal 31 modes (Fig.3). Using an estimated minimum measurable frequency shift of 0.1 Hz, a limit of detection of 15 ppm for toluene has been calculated for the first longitudinal mode and 6 ppm for second one.

The well known gas sensing properties of polypyrrole, polyaniline and SnO_2 thin films make them suitable candidates to be used for the fabrication of gas sensing devices [4]. Hence, in order to obtain gas sensing devices based on screen-printed PZT cantilevers, polypyrrole, polyaniline, active carbon and SnO_2 layers were deposited onto the PZT cantilevers. The fabrication of polyaniline coatings onto the gold electrodes was carried out by electrochemical synthesis [5], while polypyrrole films were obtained by electrodeposition onto the gold electrodes [6]. Active carbon or SnO_2 coating process consisted of drop coating.

SEM images of the different coatings confirmed the proper deposition of the polymers onto the substrate (Fig. 4,5). Coating influences on the resonant properties with both the first and the second 31's longitudinal modes also corroborated that the coatings had been well-adhered to the gold substrate (Table 1).

The deposition of described coatings onto the cantilevers presents a first step towards the detection of gases, which could permit good sensitivities to many different compounds. More specifically, SnO_2 gives promising results for the detection of benzene.

Acknowledgements: Generalitat de Catalunya, Région Aquitaine and Gobierno de Navarra have supported this research through CTP Research Grants.

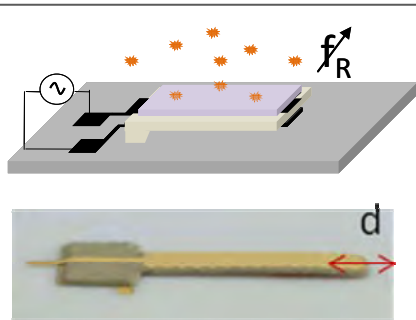


Fig. 1. Top: Schematic of PZT-based cantilever gas sensor with sensitive layer. Bottom: 31-Longitudinal displacement (in-plane) d for a polarized PZT screen-printed cantilever (size $8 \times 2 \times 0.08 \text{ mm}^3$)

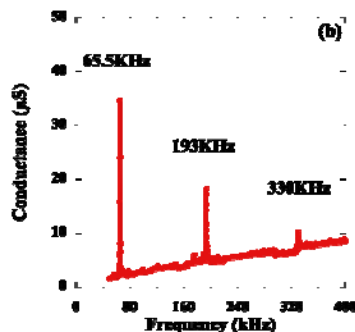


Fig 2 : Measurements of the three first longitudinal modes of a $8 \times 2 \times 0.08 \text{ mm}^3$ screen-printed PZT cantilever

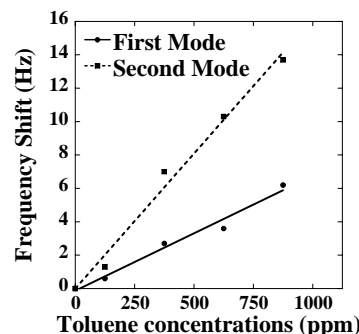


Fig.3 : Resonance shift of 1st and 2nd resonances for different concentrations of toluene in N_2 performed with the $8 \times 2 \times 0.08 \text{ mm}^3$ screen-printed PZT cantilever

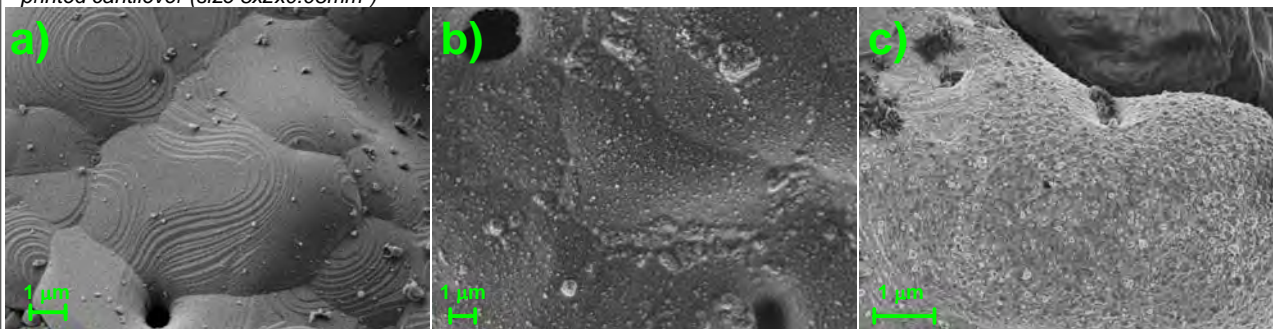


Fig. 4 : SEM image of: (a) the gold electrode; (b) the polyaniline-coated gold electrode and (c) the polypyrrole-coated gold electrode.

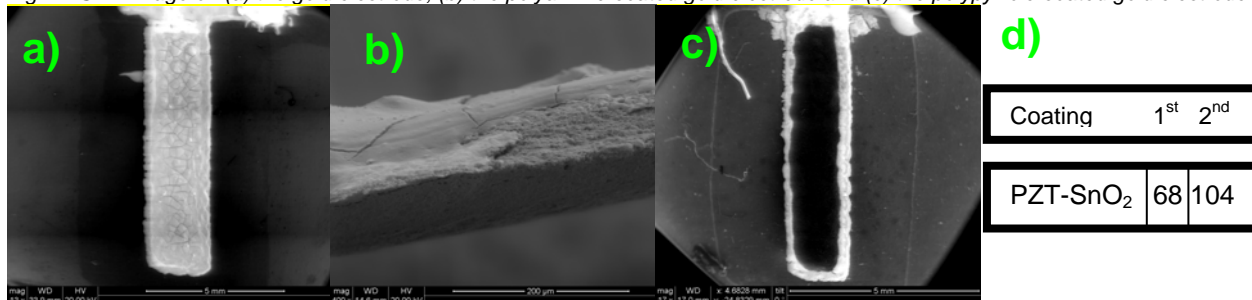


Fig.5 : PZT cantilever coated with SnO₂ (a) top and (b) side views (c) coating with active carbon (d) Resonance shift (Hz) of 1st and 2nd resonances for 1 ppm benzene in air.

	Before Deposition (KHz)		After Deposition (KHz)		Shift (KHz)	
	1 st resonance	2 nd resonance	1 st resonance	2 nd resonance	1 st resonance	2 nd resonance
Polyaniline	69.380	205.704	74.297	218.994	4.917	13.29
Polypyrrole	70.607	211.184	71.830	212.414	1.223	1.23
SnO₂	69.614	207.451	73.333	220.987	3.719	13.536

Table. 1 : Variation of the 1st and 2nd resonance modes before and after the deposition of the polymeric and CNT coatings.

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Magnetic microwires and their applications

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Keywords (up to 5 as maximum): magnetic microwire, amorphous and nanocrystalline materials, magnetoelasticity, magnetoelastic sensors

A magnetic microwire is a continuous filament of total diameter less than 100 μm of circular cross-section consisting in an inner metallic magnetic nuclei covered by a glassy outer shell. Methods, which rely on producing microwire in one operation directly from the melt, are intrinsically inexpensive and have been used successfully to produce a wide variety of metals and alloys in sizes ranging from more than 100 μm to 1 μm . Amorphous magnetic microwires with outstanding magnetic properties are, usually obtained by Taylor's technique [1]. This method allows the obtaining of tiny wires having a metallic nucleus covered by insulating pyrex-like coating. The diameter of metallic nucleus and the thickness of the coating are typically of a few microns.

Figure 1 shows several aspects related with magnetic microwire as fabrication process (a), SEM image showing the metallic nuclei and pyrex cover (b) and a magnetic microwire coil (c)

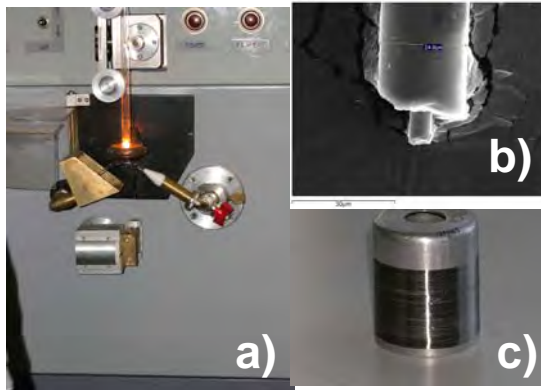


Figure 1. Amorphous microwire fabrication process (a), SEM image (b) and coil (c)

The interesting magnetic properties of magnetic microwires are strongly connected with its high axial magnetic anisotropy. This is a consequence of amorphous structure; cylindrical geometry and mechanical stresses induced during fabrication process due to the difference between the thermal expansions coefficients of glass and metal.

Magnetic sensors developed using amorphous magnetic microwires are based in operating principles strongly connected with composition and uniaxial anisotropy through magnetostriction constant like large Barkhausen effect, Mateucci magneto-impedance effect. They are also characterized by two kind of resonances i.e.: magnetoelastic resonance and ferromagnetic resonance [2].

Due to its magnetoelastic nature, amorphous magnetostrictive microwires exhibit a mechanical resonance when exposed to a time-varying magnetic field. The frequency, amplitude and damping of the vibration gives information of the sensor environment. Properties of a media, such as its viscosity, density, pressure or flow speed can be determined by analyzing the response of a magnetoelastic element placed in the media. It is also possible to follow a dynamic event, for instance the curing process of glue and paint. The magnetoelastic resonance can be monitored by using a pickup coil without the use of direct physical contacts. Because of this unique advantage, numerous applications have been proposed for this kind of sensor elements. Experiments have been performed by using a resonance technique in which the complex susceptibility was measured as a function of frequency [3] and then compared to that presented by amorphous ribbons [4]. Sample immersion in ethanol, oil and petrol allowed us to quantify the influence of medium viscosity on the coupling factor, k , and resonance frequency [5].

Other interesting property under the point of view of applications is ferromagnetic resonance, observed in the range of GHz. Based on this effect it has been possible the development of wireless sensors with possibilities of being detected at high distances. The detection of stress and strain in building structures has been possible by means of tagging it using a magnetic microwire [6,7].

Composites based on this kind of microwires present very interesting properties at high frequency. The Institute of Applied Magnetism in collaboration with the "spin-off" company Micromag 2000, S.L. has developed a radar absorbing material based on magnetic microwires [8,9].

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Fabrication and characterization of fluidic microsystems for gas detection

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Keywords (up to 5 as maximum): Gas sensors, micro-fluidic, WO₃, thermal creep, stability

The performance of gas microsensors such as selectivity and stability are among the most studied properties [1, 2]. In fact, the analysis of pollutants is generally in a real atmosphere and it is often difficult to have constant parameters of the ambient gas, such as temperature, volume and flow. In some applications such as tunnels the air is often very disturbed. However, these parameters must be controlled to obtain a reproducible analyze in different environmental conditions [3]. Our work allows fixing these parameters with a minimal space and energy. Moreover our fluidic microsystem may be use for several applications which demand stable flow and a low electric consumption. A gas microsensor based WO₃ and its heater were inserted at one end of the microchannel. Thanks to the heater that creates a gas pumping in the channel by the thermal creep phenomenon. (fig 1).

During a first step, the microsystem dimensions were calculated to obtain and enhance the thermal creep phenomenon (fig. 1). Concerning the microchannel depth, it should be closed to the free molecular pathway and should correspond to a Knudsen number between 10⁻³ and 10⁻¹ [2]. The second step was to find the best materials for the design and realization of the fluidic microsystem. Indeed, these materials affect the temperature gradient along the microchannel which is very important for the thermal creep. Thus, thermal simulations were done using Comsol Multiphysics software. The simulations results showed better properties compared Pyrex to silicon. Pyrex is the most advantageous material for the support fabrication due to its low thermal conductivity. The silicon was chosen for the microchannel fabrication because it can be more easily etched (fig. 2).

After these theoretical studies, the microsystem was fabricated with the classical microelectronic process. Then, the thermal calibration and characterization were performed with and without the microchannel. The thermal calibration has allowed observing the heater electric behaviors and the temperature variations (fig. 3). The heater thermal stability and the thermal equilibrium in the entire microsystem were done.

Finally, gas detections were done with the gas sensor alone, ie without closing the microchannel. The working temperature was determined to obtain a good sensitivity and fast response. The gas sensor showed a response with an optimum detection temperature of 473 K. Then, the sensitivity function of the ammonia concentration was studied. The results showed a high sensitivity which varies linearly with the gas concentration (fig. 4) [2, 5]. The reproducibility was also tested (figure 5).

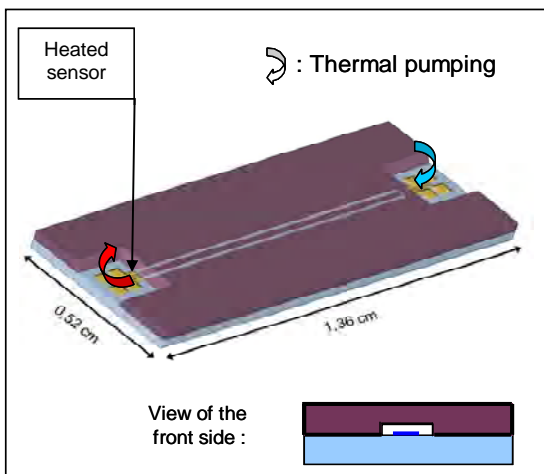


Fig. 1: Schema of the microfluidic system during activated heater

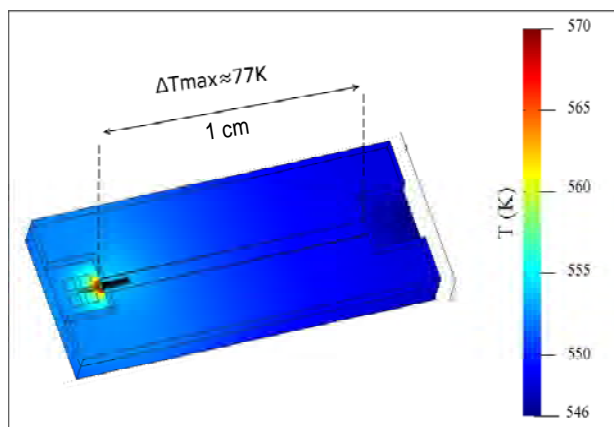


Fig. 2: Thermal simulation of the heat gradient for heater at 623 K: Representation of shell heat between 546 K and 570 K.

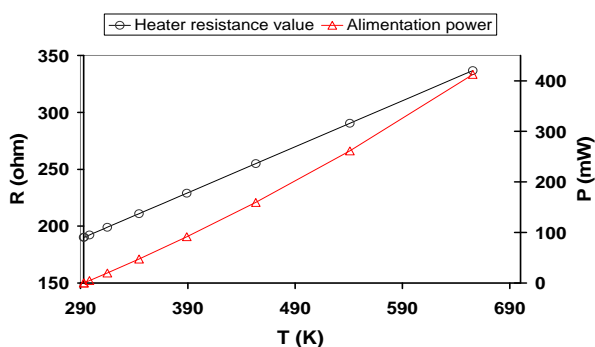


Fig. 3: Heater resistance variations and power of alimentation several temperatures of the gas sensor.

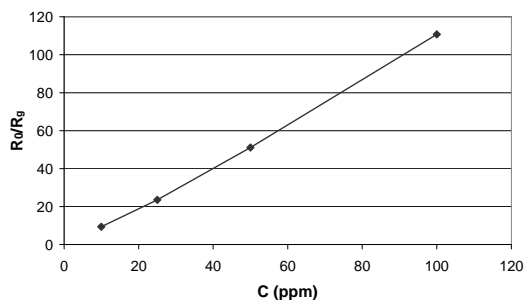


Fig. 4: Normalized sensor response in presence of for different NH₃ concentrations: 10, 25, 50 and 100 ppm. (Operating temperature: 473K)

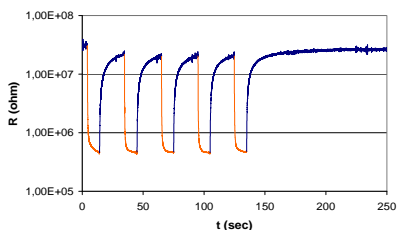


Fig. 5: Sensor response reproducibility to 50 ppm of ammonia at 473 K.

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Integration of Nanowires onto Microhotplates for the Fabrication of Low Power Consumption and Fast Operated Gas Nanosensors

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Keywords (up to 5 as maximum): Nanowires, gas sensors, Microsystems, nanofabrication, integration

Nanowires have emerged as potential building blocks for future electronic devices [1]. However, significant requirements arise from the use of elements with dimensions in the nanometer scale: large scale synthesis of structures with homogeneous properties and reliable, affordable and fast contact fabrication, among others. In this work, a methodology for the fabrication of gas sensors by integrating individual metal oxide nanowires (NWs) as sensing elements and microhotplates for low power consumption and fast operation is presented.

First, monocrystalline CVD-grown SnO₂ nanowires were dispersed on ethanol. And later, a droplet of this solution was spread onto over suspended MEMS hotplates which contained integrated microheaters. To guarantee the formation of good electrical contacts between pre-patterned microelectrodes and nanowires, Electron Beam Assisted Deposition and Ion Beam Assisted Deposition processes were performed. These nanowires were electrically contacted using a FEI Strata 235 dual beam instrument equipped with an injector to deposit Pt. The details of this fabrication method were explained in detail elsewhere [2]. Finally, two- and four-probe dc electrical measurements were done using a Keithley 2602 Source Measure Unit, enabling the estimation of the key-parameters of these nanowires. On the other hand, the integration of microheaters expedited the use of these nanowires as gas sensors. Some of them were tested using well-controlled environmental conditions of gas and temperature. This kind of measuring platform enables an optimal control of the working temperature allowing fast and reproducible modulation of the temperature up to 600 K with reduced power consumptions due to the thermal isolation and reduced dimensions of these microhotplates. The obtained results demonstrate the huge potential of nanowires as building-blocks of a new generation of devices with improved performances. For this reason microhotplate-based technologies are a promising approach for the fabrication of nanosensors in a scalable process.

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Poster Presentations:

- Innovative 3D polymer packaging of CI

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- Development of PDMS Compliant Multilayer Microstructures

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- 2D FEM model for integrated GMR current sensors

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- Optical fiber biosensor based on lossy mode resonances

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- Performance of integrated microelectrodes for the electrochemical detection

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- New sensing layers for food analysis

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- Biomimetic biosensor based on tyrosinase imbibed in a fatty acid bilayer for the detection of antioxidants

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- Micro Planar Ion Mobility Spectrometer Modelling for Security Applications

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- Feasibility of Wireless Gas Detection with an FMCW RADAR Interrogation of Passive RF Gas Sensor

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- Development of gas sensors by microwave measurement

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- Selective detection of benzene in presence of butadiene using a pre-concentrator in front of silicon μ -column/metal oxide gas sensor

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Innovative 3D polymer packaging of CI

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Keywords: Tridimensionnal Packaging; stacking Integrated circuits; interconnection; Vias; Polymer; Microsystems

The new trend of mobile electronic products is moving towards a growing need for miniaturization, weight reduction, increased performance and low cost electronic systems. The packaging technology is one of the key technologies to improve these challenges. Special attention is focused on SiP (System in Package) resulting from integration of several ICs and components from different technologies (RF, analog, digital and GaAs on Si) into a single "package".

Recently, new methods of wafer thinning allowed the development of technology "Through Silicon Via (TSV) technology". Its principle is based on the stack of thinned wafers whose chips are vertically interconnected through vias filled with a conductive material. This technology has serious drawbacks such as yields, difficulty of implementation, etc...; for small diameter vias, the silicon behaves as a composite material and loses some of its properties.

In this work, we propose an alternative technology by developing a Polymer Through Via (TPV) technique for vertical stacking of CI with a particularly intention to increase the routing density for ultra compact systems by integrating more complex chips, featuring an important number of I / O. We chose the SU8, a particular photosensitive epoxy polymer to surround the IC chips and to drill holes with high aspect ratio (until 50). The first results allowing the stress reduction give a noticeable advantage to SU8 compared with other polymers routinely used.

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R. Maheshwary , *3D Stacking: EDA Challenges & Opportunities*
SEMATECH Symposium, September 2009 , Tokyo

Development of PDMS Compliant Multilayer Microstructures

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Keywords: PDMS, multilayer, molding, hot embossing, microstructures

In the biomedical scenario, some neurosensory diseases require the use of active microimplants for electrical stimulation of the ending nerves¹. This is the case of retinal prosthesis for the treatment of the retinitis pigmentosa (RP) and the age related macular degeneration (AMD); or cochlear implants for the treatment of the sensorineural hearing loss. These microimplants have to contain a relatively high number of microelectrodes and have to be soft and flexible to adapt to a reduced human stimulation area. Mechanical and dimensional requirements constitute a challenge in the development of new planar microimplants.

In this work, a multilayer structure with alternating microelectrodes and insulating layers is proposed to increase the number of microelectrodes in a small/limited area. This paper studies two main structural aspects of these microdevices: The first one is the polymer processing to obtain a flexible multilayer microstructure. The second one is related to the polymer removing and the drilling of holes through the layers to expose the stimulation electrodes.

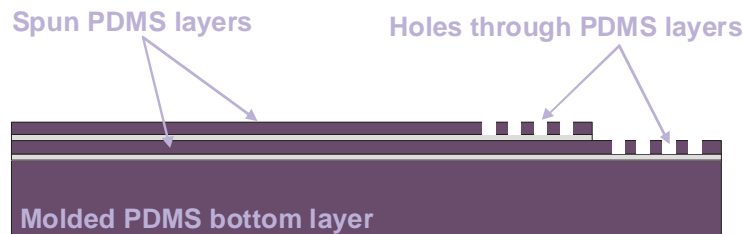


Figure1. Multilayer structure

PDMS multilayer

The silicone elastomer MED 4011 (Nusil Technology) is chosen as structural material. The consistency and viscosity of this biocompatible PDMS allows structuring a layer by molding or spin coating. The thick bottom layer is obtained by molding. The process involves a SU8 mold (SU8-100 from Microchem) fabricated on a Borofloat (Schott) glass wafer. This mold is filled with the PDMS' base polymer and its cross-linking agent and degasified for 1 hour. In a next step, a hot embossing system model EVG510 is used to cure the polymer under pressure. To obtain 300 μ m-thick substrates, a temperature of 150°C and a force of 1500N are applied for 1 hour.

The formation of the thinner successive PDMS layers is accomplished by a spin coating system. The PDMS mixture is spun at 5000rpm for 30s. Then, PDMS layers are degasified for 10 min and cured at 150°C for 1h on a hot plate. The achieved layers are 30 μ m-thick.

Multilayer structures made up of one thick bottom layer and three thinner spun layers have been fabricated. First surgery tests have been successfully performed.

PDMS removing

Regardless of the number of stacked layers, a selective PDMS elimination is necessary to access to the underlying microelectrodes. An accurate etching depth control is needed. For this goal, four procedures have been considered. The use of a photoPDMS, a mixture PDMS/Benzophenone, has been excluded because the new compound loses the biomedical grade. Wet etching using organic solvents such as a mixture Tetra Butyl Ammonium Fluoride /N Methyl Pyrrolidone is very aggressive and requires a strong etching mask. Laser drilling has also been rejected because of the damage caused to the underneath metallic layer by the beam energy. Then reactive ion etching (RIE) technique has been taken into account. RIE processes have been performed in a Plasmalab 80Plus RIE system from Oxford Technologies. Different values for RF power, total gas flow and SF₆-O₂ gas ratio have been considered. Aluminium has been employed as etching mask.

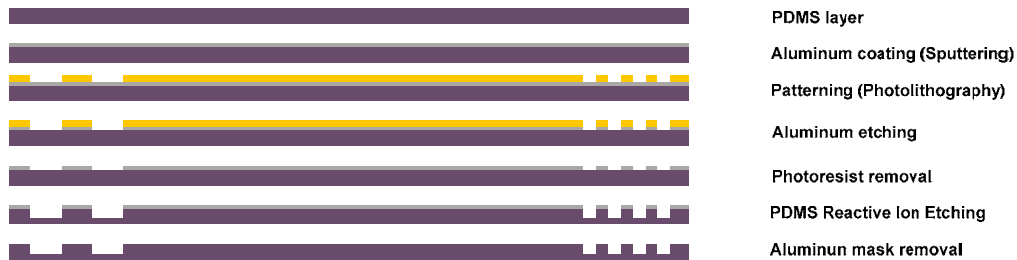


Figure2. RIE process steps

Etching rates between 0.15 and 0.91 $\mu\text{m}/\text{min}$ have been achieved. Roughness of the etched surface ranges between 0.04 and 0.52 μm .

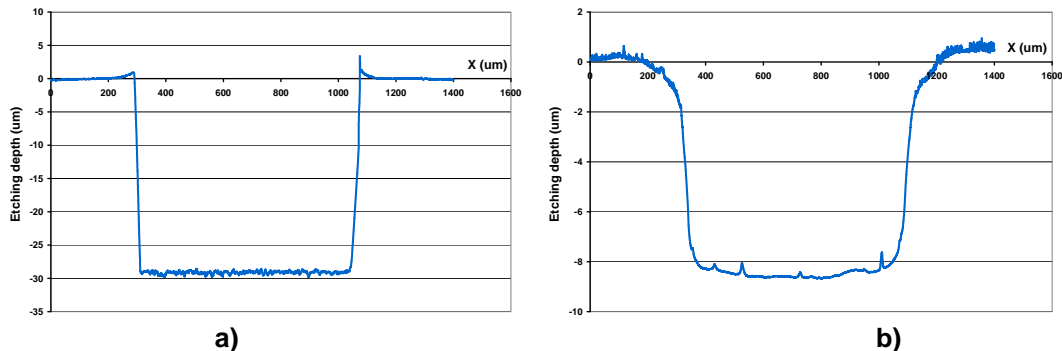


Figure3. RIE processes a) 300W, 60sccm, SF₆-O₂ (4:1) and b) 100W, 30sccm, SF₆-O₂ (1:9)

As conclusion we can summarize: a) A flexible multilayer microstructure made up of a thick substrate processed by moulding with successive thin layers applied by spin coating has been achieved, b) The consistency and mechanical robustness of such multilayer microstructures are adequate to sustain surgery probes performed in human temporal bones and c) The perforation of thin PDMS layers for exposing the underneath stimulation electrodes has been achieved by a controlled RIE process.

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2D FEM model for integrated GMR current sensors

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Keywords (up to 5 as maximum): FEM, modelling, current sensor, magnetoresistance, spin valve

Indirect measurement of the electrical current from the magnetic field that is generated is a well-established concept. Regarding electrical current measurement at the IC level, the utilization of solid state magnetic sensors (mainly Hall effect and giant magnetoresistance ones) is often preferred, due to their inherent higher level of integration. Nevertheless, the incorporation of current strips into the IC during the fabrication process of monolithic devices introduces additional handicaps (high frequency couplings, power dissipation limitations ...) and it is currently under research [1].

In this sense, in the present work, and as a very useful analytical tool, we describe and validate a 2D FEM model applied to giant magnetoresistance based current sensing devices, as described in Fig. 1.

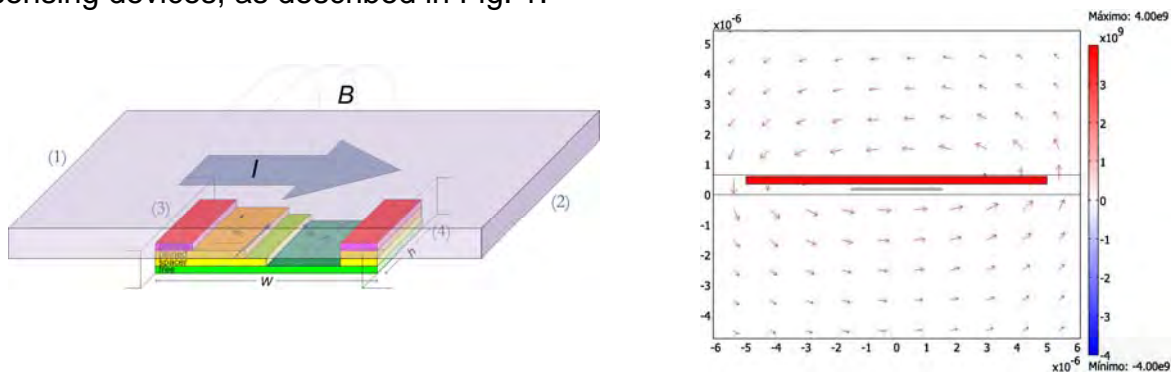


Fig. 1 Device principle and modelling

The devices used in the present study were fabricated at the clean room facilities of INESC-MN at Lisbon (Fig. 2). The microfabrication details can be found in [2]

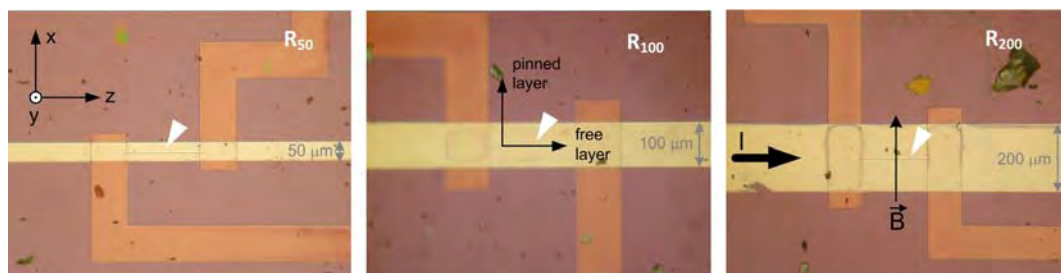


Fig. 2 Devices micrographs

If low to moderate magnetic fields are considered, we can assume:

$$R = R_0 + MR \cdot B$$

where R_0 is the spin-valve resistance at zero magnetic field, MR is the linear magnetoresistive parameter and B is the magnetic field component in the direction of the easy axis of the pinned layer. R_0 and MR can be experimentally extracted.

For the numerical modelling we used the FEM-based COMSOL Multiphysics software package. This package is the current evolution of the well-known FEMLab, which has long been successfully applied to the modelling of general physical problems but, to the authors' knowledge, has only recently been applied to the calculation of magnetic field-related sensing structures [3]. For DC characterization, an automated measurement system, similar to the one used for magnetic field characterization, was arranged. For checking the devices functionality, the resistance values were measured for each different device, as a function of the driven current, from -10 mA to +10 mA, following a hysteresis detecting scheme. Results are shown in Fig. 3.

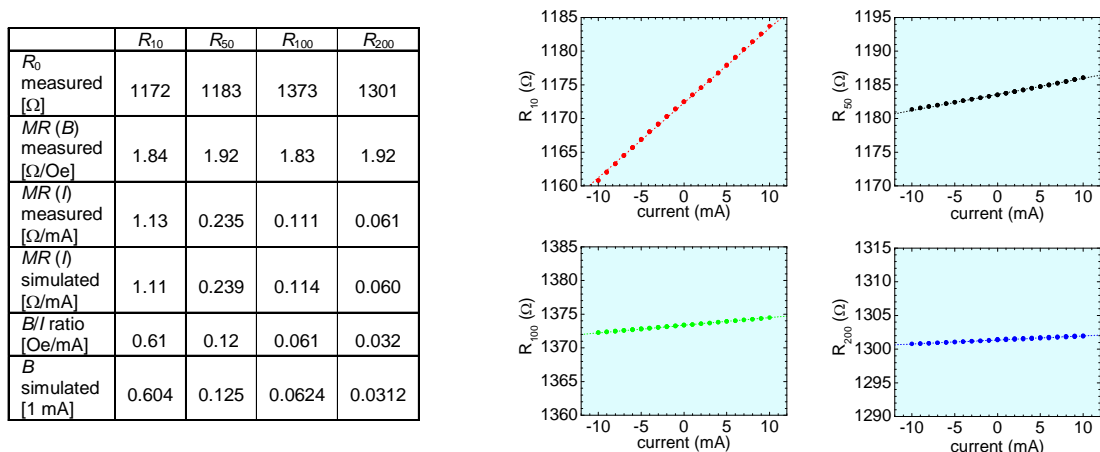


Fig. 3 Extracted parameters (left) and model (lines) versus measurements (dots) (right)

In summary, we have successfully developed a 2D FEM model for spin valve based electrical current sensing elements. The model has been validated against experimental results from specifically designed test devices. The model is flexible and can be easily applied to quantify the effects of misplacements that are inevitably introduced during the fabrication process, such as inhomogeneities in the isolation layer thickness. Knowledge of these errors can shorten the final prototyping process, with its associated benefits. The application of the model can be extended to electrical feasibilities such as frequency response analyses.

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Optical fiber biosensor based on lossy mode resonances

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Keywords: optical fiber, antibody, biosensor, lossy mode resonance, layer-by-layer

The detection of the specific binding between antigen and antibody is perhaps the most used technique for diagnosis in medicine and biology. More specifically, the utilization of optical devices whose spectral response is sensitive to this specific antigen-antibody binding is extensively exploited in immunoassays. For instance, the coating of optical waveguides with semiconductor or metallic materials has become a widely explored technique for the fabrication of devices such as surface plasmon resonance sensors (SPR) [1]. Usually, these biological sensors are fabricated by synthesising a thin film on the sensor probe whose refractive index is sensitive to a target analyte. This alteration of the refractive index leads to a modification of the sensor spectral response [2]. Among other techniques, the layer-by-layer self assembly method (LbL) has been proposed as a promising method for the fabrication of these thin films which can operate as substrates for the immobilization of immunoglobulin G (IgG) and anti-IgG [2,3].

Moreover, devices with a spectral response similar to the SPR have been recently presented in the literature, named Lossy Mode Resonance (LMR) sensors [4,5]. The LMRs are obtained by the coupling of light from a multimode optical waveguide to a supporting layer of an optical absorbing material such as ITO or TiO₂/PSS films [4,5]. Each of these resonances owns a particular sensitivity to the external refractive index.

In this work, a LMR-based optical fiber biosensor which consists of a 200 μm optical fiber core coated with titanium oxide nanoparticles and poly (sodium 4-styrenesulfonate) (PSS) is studied. The experimental setup and details of the sensor head are depicted in Figure 1. The cladding of a fragment of a multimode optical fiber (FT200EMT, Thorlabs Inc.) was chemically removed and then the core was sonicated and cleaned in both detergent and acetone and rinsed in ultrapure water. After that, the fragment was perpendicularly cleaved and coated with reflective film (Ag) forming a mirror on the end of the surface of the fiber and protected with a plastic cap. Then, the LMR supporting overlay composed of [TiO₂/PSS] bilayers was fabricated by the Layer-by-Layer (LbL) electrostatic self-assembly technique [5], see Figure 2 to observe the generated LMR. This polymer/inorganic film was used as a precursor film for the immunosensing layer and IgG molecules were adsorbed onto the polymeric surface by hydrophobic and electrostatic interactions [6]. The sensor was then immersed in goat IgG solution (50μg/ml) for 90 min at room temperature. To reduce the non-specific binding, the sensor was rinsed with phosphate buffered saline (PBS) buffer solution. Finally, the sensor was immersed in anti-goat IgG (50μg/ml) and the results are plotted in Figure 3.

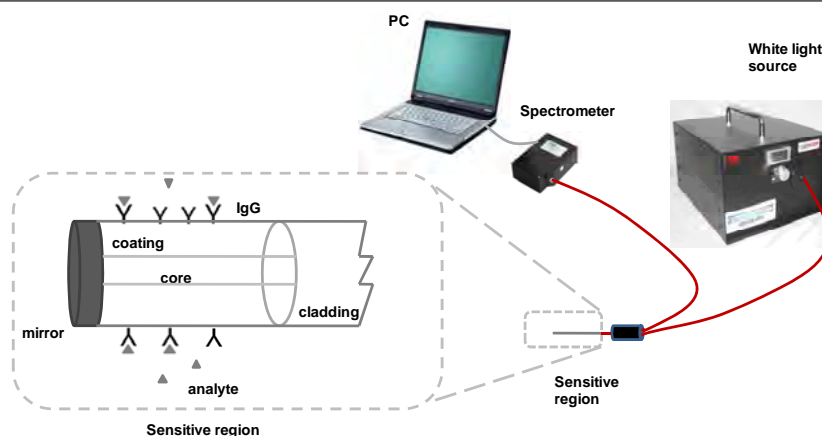


Figure 1. Experimental set-up

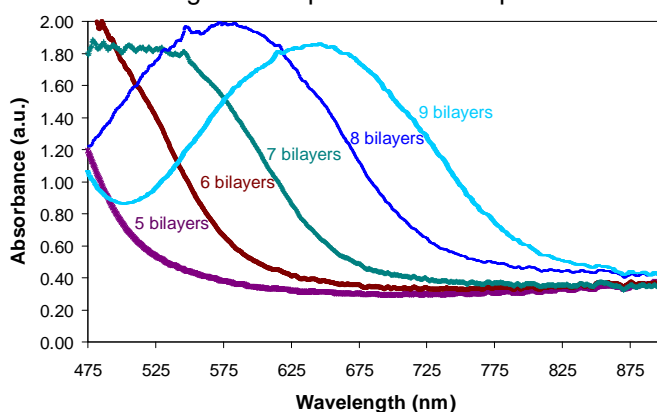


Figure 2. Spectral response of the LMR devices

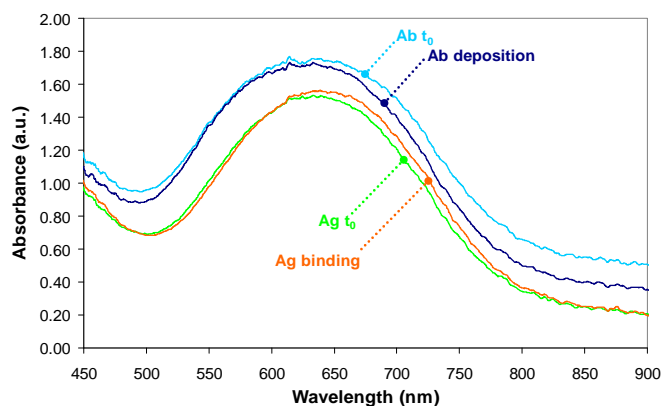


Figure 3. Spectral response before (Abt_0) and after (Ab deposition) the deposition of antibodies and before (Abt_0) and after (Ag binding) the attachment of antigens

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Performance of integrated microelectrodes for the electrochemical detection

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Keywords (up to 5 as maximum) : Microelectrodes, Electrochemical cell, Oxidative stress, Cyclic voltametry

Electrochemical sensors present a growing interest for analytical control in various application fields (health, environment, food industry...). These devices tend to be miniaturized to answer current issues - low volumes, reduced analysis time, portability - and to take advantage of the potentiality of microelectrodes: sensitivity, detection limit improvement, ability to measure in resistive media. The microtechnologies can now manufacture collectively generic microelectrodes and electrochemical cells with multiple geometries [1]. However, to obtain reliable and reproducible results, the electrochemical measurements require a prior knowledge of the microelectrodes materials, and more especially of the surface states and properties.

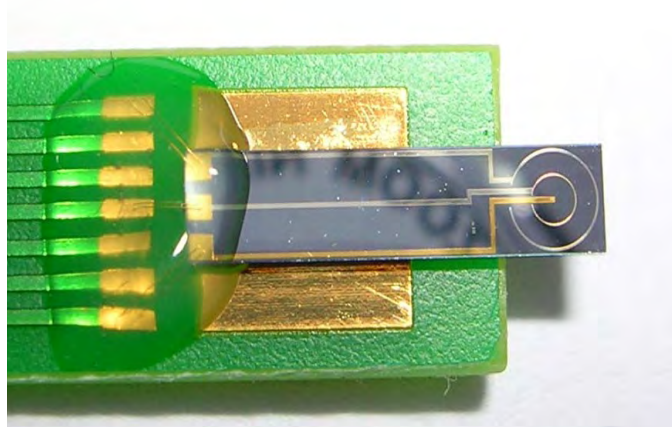
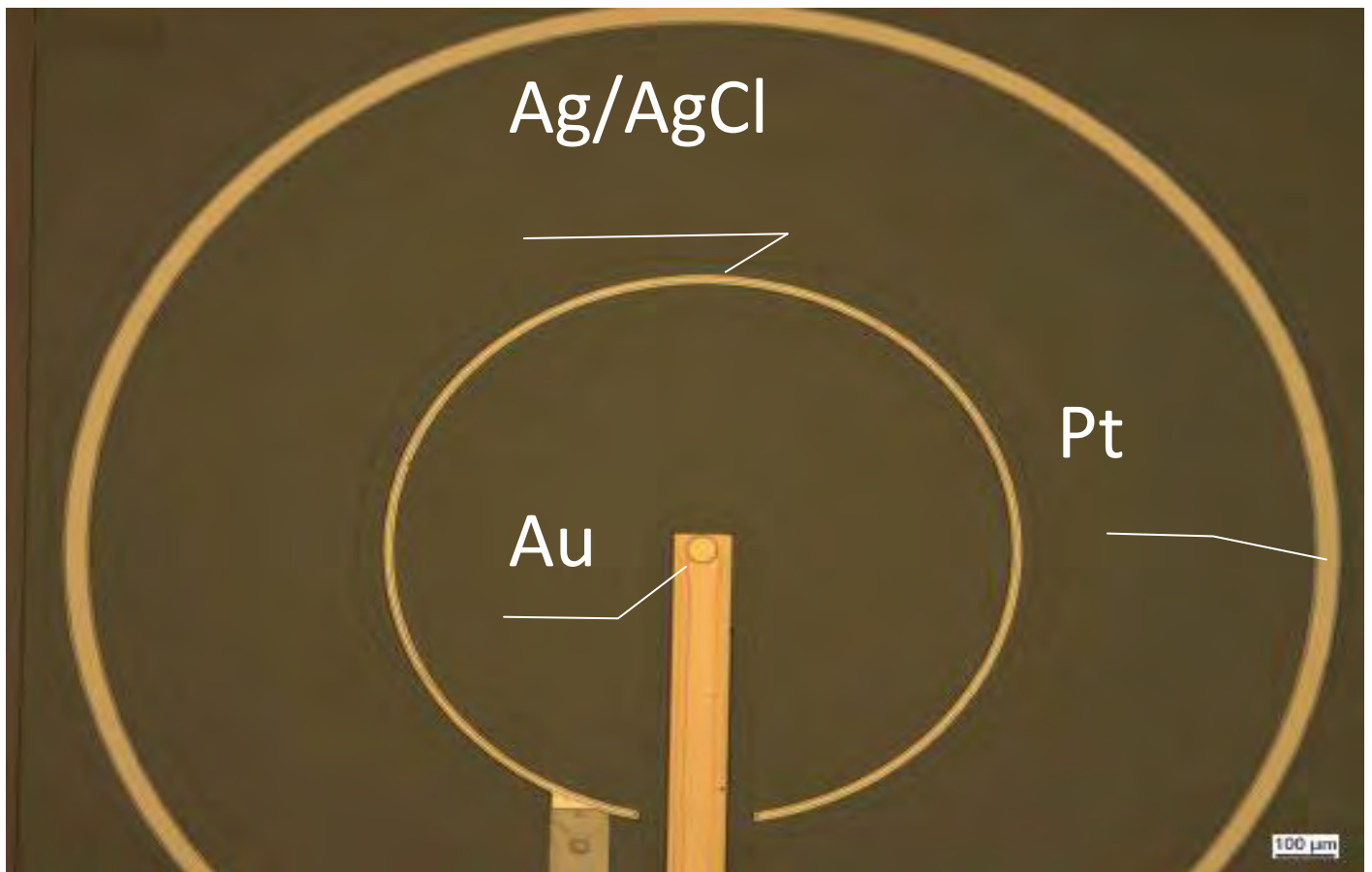
Our work consisted in integrating an electrochemical cell comprising a traditional three electrodes system on a silicon substrate and in characterizing all the materials used. The fabrication process has been realized thanks to "Silicon & Polymers" microtechnologies and has been more precisely focused on the "lift-off" photolithography technique. It allowed the integration of metallic thin films such as gold, platinum and silver while using metal sub-layers to assure the adhesion on the silicon surface. The encapsulation techniques have been finally required to passivate the underlying layers in order to avoid contact with the studied environment and to delineate the active areas. Our choice concerning this passivation material has been brought on the biocompatible SU-8 photoresist, knowing that this photosensitized epoxy-based polymer with triaryl sulfonium salts could be responsible for some interfering electrochemical reactions.

The quality of passivation and metal deposition has been studied by characterizing the different microelectrodes using cyclic voltammetry. The voltammograms obtained in model solutions are similar to those recorded with solid materials. They allow to show the quality and the definition of the different active metal surfaces. Finally, an Ag/AgCl reference electrode is necessary to complete the electrochemical cell.

The results have shown the possibility to mass integrate reproducible electrochemical cells from techniques of microelectronics. These devices can be finally adapted to specific bio-electrochemical applications. For example, they have been used to detect the uric and ascorbic acids, molecules involved in the defense system against oxidative stress. To evaluate their performance, results were compared to those obtained with traditional hand-made microelectrodes while using commercial macroscopic auxiliary and reference electrodes.

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New sensing layers for food analysis

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Keywords (up to 5 as maximum):

Aptamer, Molecular Imprinted Polymer, Antibody, Biosensor

Effective food control systems are essential to protect consumers from any possible health risk and to ensure the quality of the final product. These procedures are usually been performed with traditional laboratory tools that in most cases are time consuming, expensive and not fast enough.

Recent advances in micro- and nano-technologies have provided new tools which can be applied to the food analysis as well. In this sense, biosensors are gaining more and more importance and the applications in the food industry are increasing due to their powerful characteristics: sensitivity, selectivity, speed...

When making a biosensor, a critical issue is to develop an appropriate sensing layer where the target analyte recognition occurs. Moreover, a signal (optical, electrical...) ready to be detected should be generated. With this purpose different biomaterials such as antibodies have been reported to be adequate and achieve very good results [1].

In this work 3 different biomaterials (antibodies, molecular imprinted polymers, aptamers) are presented which have been developed for sensing pesticides and biogenic amines.

1. An **immunosensor** development is reported for immunochemical screening of small organic molecules based on the use of an innovative three-dimensional interdigitated electrode array. Four pesticides from different chemical groups have been selected for the performance evaluation of the multiplexed device. With that purpose, the sensor surface of the transducer has been chemically modified to achieve the covalent union of appropriate haptenized-proteins. Subsequently a cocktail of immunoreagents that selectively binds to the corresponding antigen has been added. These molecules recognize specifically each antibody and a selective change in the impedance of the electrodes was observed.

2. **Molecular imprinted polymers** (MIPs) are generated by co-polymerization of a functional monomer with a cross-linking monomer in the presence of a template molecule. MIPs are easily and rapidly synthesized with a low cost and present a high thermal and chemical stability. Moreover, some of the characteristics of the polymers (size, shape...) can be controlled by selecting appropriate polymerization conditions [2]. In this work two organophosphorous pesticides: azinphos-methyl and

chlorpirifos-ethyl have been used as template molecules. The polymers can be prepared with well controlled physical forms in different size ranges so as to be used in different applications (separation columns, sensing layers...). Several MIPs have been achieved with different particle size and appearance. Their affinity towards the selected pesticides has been evaluated and some applications are shown.

3. **Aptamers** are small oligonucleotide acid molecules that can bind to various molecular targets such as proteins (thrombin...) and small molecules (vitamins, cocaine, amino acids...) with strong affinity and specificity, based on their unique three-dimensional structure. Therefore they can be considered as strong candidates to become affinity ligands and bio-recognition elements, being an alternative to antibodies.

Aptamers have been used in the development of different kinds of biosensors (aptasensors) employing electrochemical, optical and mass-sensitive transducers. They have been successfully coupled to Piezoelectric Quartz Crystal Microbalance and Surface Plasmon Resonance (SPR) devices as transducers.

Aptamers can be selected by means of the Systematic Evolution of Ligands by EXponential enrichment method (SELEX) developed by Ellington and Szostak (1990) (1). This methodology starts from a random synthetic nucleic acid library of 10^{15} oligonucleotides, followed by repeated rounds of in vitro selection consisting of binding, washing, elution and PCR-amplification of selected oligonucleotides. Once optimal aptamers are selected, their sequence must be defined in order to be able to both introduce optimization modifications as well as to produce them indefinitely by chemical synthesis.

In AZTI we have fulfilled the selection process of specific aptamers for histamine molecules by means of FlumagSELEX, a modified SELEX selection protocol that employs fluorescence molecules and magnetic particles to facilitate selection (2). Future actions will include coupling the selected aptamers to two different kinds of transducer systems available Piezoelectric Quartz Crystal Microbalance and Surface Plasmon Resonance (SPR).

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Biomimetic biosensor based on tyrosinase imbibed in a fatty acid bilayer for the detection of antioxidants

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Keywords (up to 5 as maximum): Biosensor, Langmuir-Blodgett, Antioxidant, Voltammetric sensor

The development of phenol oxydase biosensors is of great interest due to the importance of phenols and polyphenols in the food industry. To retain the enzyme specific biological function, the appropriate immobilisation of the enzyme on a solid matrix is a key factor. Tyrosinase and Lacasse biosensors have been developed using a variety of techniques that include covalent immobilization, screen printing or the use of the Langmuir-Blodgett (LB) technique (1, 2). The LB technique is of special interest for enzyme immobilization because it allows preparing biomimetic systems, where the enzyme is adsorbed in a lipidic layer. It has been established that the incorporation into the LB films of electron mediators can improve the sensitivity of the electrochemical sensor.

In this work, biosensors based on LB films of a phenol oxydase (the Tyrosinase) for the detection of phenol derivatives have been developed. In order to mimic biological systems, the enzyme has been incorporated in a monolayer of arachidic acid using the Langmuir-Blodgett technique (Figure 1). The enzyme immobilization has been improved by cross-linking with glutaraldehyde. Lutetium bisphthalocyanine has been admixed in the nanostructured films in order to facilitate the electron transfer. Cyclic voltammetry has been applied to study the detection of five phenolic species including vanillic acid, catechol, pyrogallol, gallic acid and caffeic acid.

The electrochemical behaviour of the electrodes has been analyzed and the specificity of the biosensors has been discussed.

It has been demonstrated that the presence of the lutetium bisphthalocyanine as electron mediator, increases the sensitivity of the sensors. The biosensors presented here have a promising future for the detection of antioxidants of interest in the food industry.

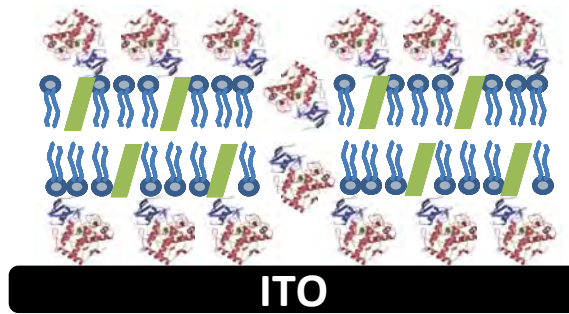


Figure 1. Structure of the biosensor

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Micro Planar Ion Mobility Spectrometer Modelling for Security Applications 5^{èmes} Journées Franco-espagnoles IBERNAM – CMC2

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*Keywords (up to 5 as maximum): IMS; Ion Mobility Spectrometry; FAIMS; FEM
Multiphysics, COMSOL*

Summary

Modelling of a Micro Planar Field Asymmetric Ion Mobility Spectrometer (P-FAIMS) is developed for sensing acetone (Ac) and dimethyl methylphosphonate (DMMP) for security applications. In P-FAIMS target ions are discriminated by the application of the proper separation voltages to the electrodes of the system. Using FEM Multiphysics software, optimum voltages for achieving a correct separation of ionized molecules have been obtained for the studied compounds.

Motivation and results

Security has become a very attractive application for gas chemical sensing systems as many of the potentially risky substances may be detected by sensing their vapors at low concentrations. Nowadays, the main concerns are the detection of chemical warfare agents, explosives and health risky volatiles. Systems for detecting these volatiles must be fast, and selective while working at very low concentrations (ppb's). Traditional gas sensing systems based on resistive, optical or acoustic measurements and on microbalances do not easily accomplish such requirements and Ion Mobility Spectrometers are good alternatives, already used in airports, public spaces and industries [1]. Thus, the development of such systems using micro-technologies for smaller systems integration is very challenging.

In this paper, the modelling of a micro differential IMS is presented with the aim of being used for the detection and discrimination between DMMPH⁺ and Ac₂H⁺, as representative examples of possible cohabitation of various target vapours in Security applications. IMS show good separation capability for multiple ions with short response time, and especially μ IMS's allow low temperature operation with higher sensitivity compared to other IMS implementations [2].

In figure1 a simplified diagram of the proposed micro IMS is presented. The target vapour is 100% ionized by an external source prior to enter the IMS and flows through the gas chamber defined by two electrode plates on which a voltage is applied. A separation AC voltage (V_{RF}) and a compensation DC voltage (V_C) component are applied to the electrodes with the objective of attracting non desired ions to the plates while allowing the fly of the target vapour ions until the detector electrode, at the end of the chamber.

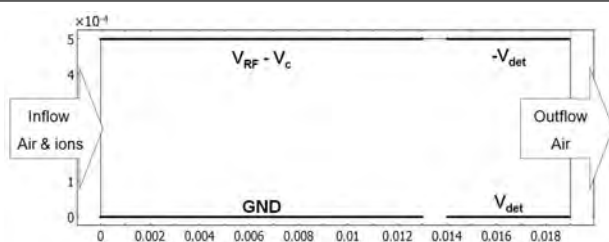


Fig. 1: micro Ion Mobility Spectrometer schematic.

Defining the proper dimensions of the chamber and the proper voltages it is possible to differentiate the ions that will reach the detector electrode. The dimensions of the IMS chamber considered for simulations are [3]: $13 \times 5 \text{ mm}^2$ for electrodes in the drift region, $5 \times 5 \text{ mm}^2$ for electrodes in the detection region with a separation of 0.5 mm between plates and 1 mm between regions. A two-harmonic waveform with amplitude ranging between 875 and 1125 V and 2 MHz frequency has been applied for the separation voltage. Compensation voltages in the range of -4 V to 2 V have been considered. Concentrations have been fixed to 1 ppm in all studied cases. In figure 2, concentrations of the Ac and DMMP gases are presented for a $V_{RF} = 875$ and 1000 V, showing two different situations: A) interferences in results: for a $V_{RF} = 875$ V, Ac and DMMP ions reach the detector for the same value of V_C (-2.3 V) so, for just certain values of the applied voltage ions with the same DC voltages will not be separated. B) Separated measurement: for $V_{RF} = 1000$ V the V_C that allows the detection of each component is well differentiated.

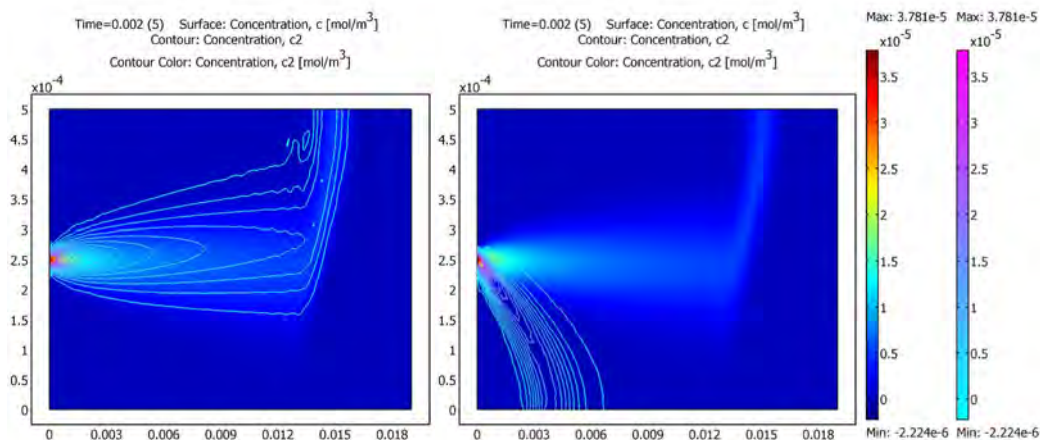


Fig. 2: Simulations of dimer ion Ac_2H^+ concentrations -represented by colour variations- mixed with monomer ion DMMPH^+ -represented by isoconcentration lines-. For a separation voltage of LEFT) $V_{RF} = 875$ V, both ions have the same $V_C = -2.3$ V and are attracted to the same detector electrode. No differentiation is obtained. RIGHT) $V_{RF} = 1000$ V, and for a $V_C = -1.9$ V only Ac_2H^+ detection is obtained. Differentiation is achieved.

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Feasibility of Wireless Gas Detection with An FMCW RADAR Interrogation of Passive RF Gas Sensor

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Keywords (up to 5 as maximum): RF gas Sensors, Dielectric Relaxation, TiO₂ resonator, RADAR interrogation

Summary

The feasibility of the remote measurement of gas detection from an RF gas sensor has been experimentally investigated. It consists of a Frequency-Modulated Continuous-Wave (FMCW) RADAR interrogation of an antenna loaded by the passive sensor. The frequency band of the RADAR [28.8-31GHz] allows the detection of the resonant frequencies of Whispering Gallery Modes that are sensitive to gas concentration. Reported experimental results provide the proof-of-concept of remote measurement of gas concentration fluctuation from RADAR interrogation of this new generation of passive gas sensors.

Motivation

Recent results obtained from electromagnetic simulations¹ indicated that a TiO₂ dielectric resonator (DR) can adsorb gas molecules (like CO) and such adsorption induces a frequency shift in resonant frequencies of high-sensitive Whispering Gallery (WG) modes. This transduction is given by the dielectric relaxation effect at high frequency. The experimental validation of the design and electromagnetic simulations was carried out using BaSmTiOxide ceramic as a dielectric material for the DR instead of TiO₂, as it is easier to fabricate by standard Sintering technique than metal oxide DR: the dielectric property of this ceramic presents a relative permittivity (78.6 ± 2 from¹) is very close to TiO₂ one.

Results

The fabricated Whispering-Gallery mode resonator with its coplanar waveguide (CPW) feeding lines is shown in Fig.1. The measured transmission coefficient S_{13} in the millimeter-wave frequency range is reported in Fig. 2. As expected, transmission peaks are observed at the resonant frequency of the WGH_{5,0,0}, WGH_{6,0,0} and WGH_{7,0,0} modes. Due to the technological inaccuracies, the three resonators (with the same dimensions) micro-machined on the same wafer do not have identical transmission peak magnitudes and frequencies : for example the WGH_{7,0,0} mode resonates at 29.4 GHz for the resonator 1, at 29.5 GHz for the resonator 2 and at 29.9 GHz for the resonator 3 (see fig.3). These three resonators can be considered as a gas sensor that resonates at three different frequencies, each frequency corresponding to a specific gas concentration. These three resonant frequencies (or gas concentration) can be measured from the FMCW RADAR interrogation of an antenna loaded by the resonator (passive sensor). As shown in Fig.4, the sensor interrogated by this reader, use an identification technique¹ which depends on the backscattered signal of the horn antenna (structural mode) and the cables delay line, a length of coaxial cable, connected to the cell (antenna mode). The three WGH_{7,0,0} resonant frequencies can be scanned by the RADAR because they belong to its specific band (28.8GHz to 31GHz).

In the FMCW radar output spectrum, the beat frequency that allows measuring the location of the sensor can easily be detected (here the distance between the radar antenna and the sensor horn antenna is 3.4 m and consequently the beat frequency is 30 KHz). Depending on load which is applied to its output, on Fig.5, the presence or absence of the sensor is checked. Table 1 shows the variation of radar amplitude between the three structures with the output open circuit. These first measures validate the feasibility of a radar measurement of gas detection and their quantity by wireless communication between the reader and a totally passive sensor.

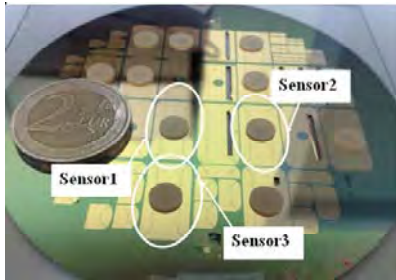


Fig. 1: Fabricated sensors on 4" Silicon wafer: resonator with its CPW excitation lines.

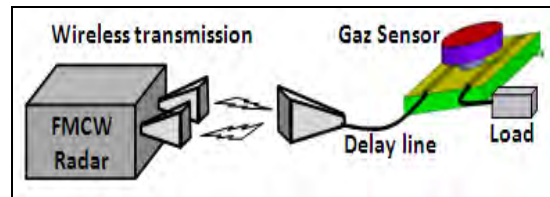


Fig. 4: Experimental assembly between the radar and gas sensor

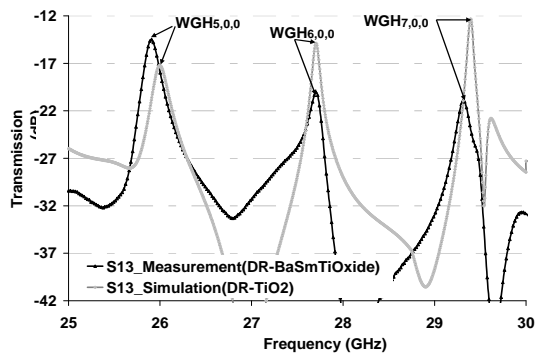


Fig. 2: Measured and simulated transmission coefficients versus frequency for the BaSmTiOxide and TiO₂ dielectric resonator.

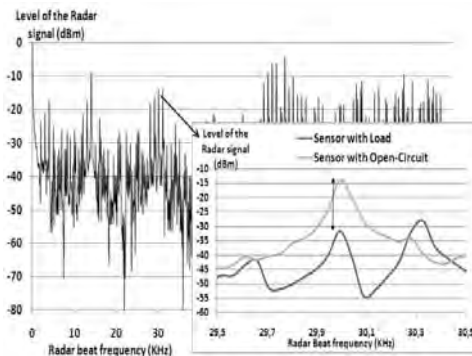


Fig. 5: Spectrum of the radar signal: Identification of the sensor variations (with and without load)

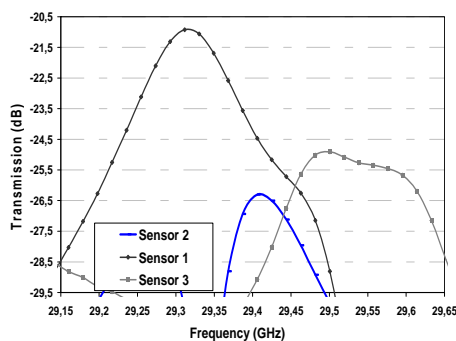


Fig. 3: Measured transmission coefficient versus frequency for three sensors at WGH7, 0, 0.

Level of the Radar signal	Sensor 1	Sensor 2	Sensor 3
Sensor with open-circuit (dBm)	-10,4	-12,7	-13,9
Sensor with Load (dBm)	-43	-18,5	-31,7

Table 1: Level of the beat frequency radar for each sensor (1, 2, and 3)

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Development of gas sensors by microwave measurement

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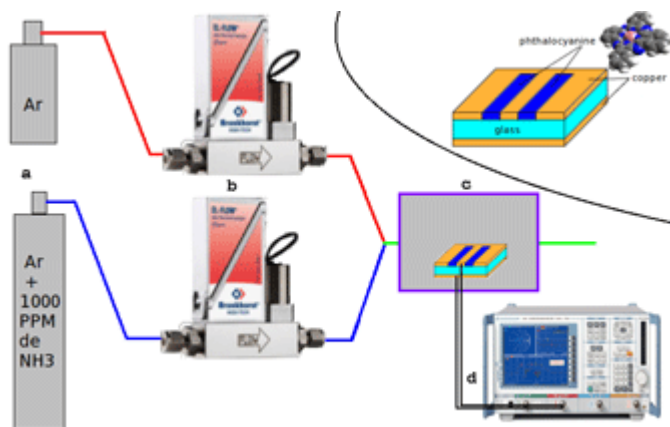
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Keywords gas sensor, Microwave transduction, room temperature, molecular material

This work presents a novel approach in gas detection by an original method of microwave transduction. The shape of the sensor incorporating the microstrip resonant line and a sensing material was designed and its sensitivity to ammonia was studied.

The sensitive material can play the role of the substrate or can be deposited as a thin layer on a microstrip structure used in electronics. Submitted to an electromagnetic excitation in microwave energies, the sensor response in the presence of a gas results in a specific modification of the reflected wave (real and imaginary parts). We showed how, in the presence of ammonia, the reflected wave is related to its concentration. The response to the material-gas interaction depends on the excitation frequency. The parameter used as the sensor response is the reflected wave on incidental wave ratio at each frequency. The study deals with the influence of sensitive materials, inorganic (SnO_{2-x}) or molecular (CoPc), on the response of the sensor in the presence of ammonia in air. All the studies were carried out at room temperature.



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Selective detection of benzene in presence of butadiene using a pre-concentrator in front of silicon μ -column/metal oxide gas sensor

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Keywords: Pre-concentrator, silicon micro-column/ commercial metal oxide gas sensor, benzene, butadiene, selective detection

Abstract

Among the hazardous chemical compounds that can be found in the atmosphere, as a result of petroleum industry and transport, we can quote benzene and butadiene. Both gases are known as carcinogenic causing several complications such as leukemia.

Conventional analytical methods such as chromatography have been used successfully to selectively monitor different compounds at the ppb level. Their main drawback is their big size and high cost which make them impractical for in situ analysis [1]. The use of miniaturized chromatographs for the analysis of complex vapors was reported. Those chromatographs rely on silicon etched micro-columns related to a microsensor array [2]. Some other authors reported the coupling of gas pre-concentrators with such GC systems for the selective detection of BTEX [3]. Our final objective is to develop a detection micro-system based on the three coupled elements: a micro-concentrator, a GC micro-column and a metal oxide gas sensor for the selective detection of benzene in presence of butadiene in air.

The system used in this study is composed by a gas pre-concentrator [4], connected to a GC column in front of a detector. The GC column was either a commercial capillary column or a circular spiral silicon micro-column fabricated following the procedure detailed in [2]. While the detector consisted either of a commercial flame ionization detector or a TGS metal oxide gas sensor from Figaro. The three units were connected to each other using a six-way valve.

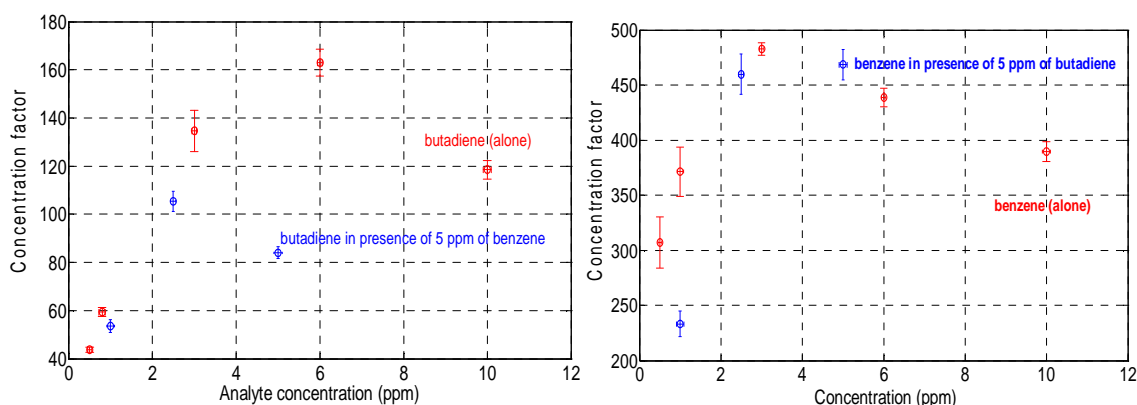


Fig. 1. Concentration factor versus analyte concentration (a) benzene (b) butadiene

In this study, we started by characterizing the gas pre-concentrator device in order to concentrate benzene and 1,3-butadiene. Using the GC/FID, the experimental conditions to operate the preconcentrator were optimized for a successful desorption of both analytes. The analytes were adsorbed at 100 ml/min during 10 min and then desorbed at 175°C during 10 min. Thanks to the pre-concentration stage, the detection limit of the system for both analytes was lowered by at least 6 times. However, the adsorbent showed more affinity for benzene than for butadiene, resulting in a concentration factor (CF) for benzene much higher than that for butadiene (cf. Red label-Fig. 1a and b). When analyzing a mixture of one analyte in the presence of a fixed concentration of the other (blue label-Fig. 1a and b), we noticed the existence of a mutual interference between both compounds in the pre-concentrator.

Once the pre-concentration of both analytes was adjusted, the capillary GC column was replaced by a silicon micro-column and the FID by the TGS.

We adjust at first the sensor temperature so as to obtain a chromatographic peak response. This temperature was found at 400 °C. By calibrating the system with and without the concentrator, we found that the detection limit of the system to each analyte separately was lowered by a factor up to 5 times for benzene and 2.5 for butadiene. When the mixture was analyzed, the micro-column was operated at ambient temperature for a best separation of both analytes and the detection limit of the system was lowered as before. We observed the elution of four peaks from the micro-column instead of two, that must be due to both desorbed analytes (Fig. 2 a). The two other peaks observed are due to the benzene and butadiene remaining in the pre-concentrator chamber during analyte adsorption. A part from this problem, the successful operation of the pre-concentrator/micro-column/Gas sensor system was demonstrated regarding the possibility to selectively detect benzene in presence of butadiene in mixture at ambient temperature (Fig. 2 b), which is interesting for the cost of the application. A decrease of the detection limit of the system towards both analytes was also observed.

The performance of the system can be further enhanced by optimizing the micro-column design.

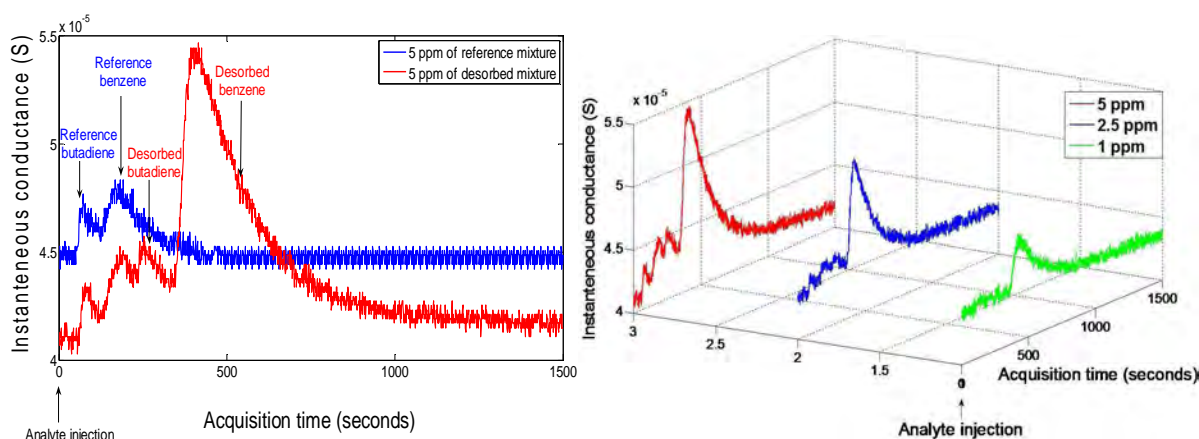


Fig. 2. Chromatograms of desorbed mixture (a) compared to reference mixture at 5 ppm (b) for the dilution of both desorbed analytes at room temperature

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With the collaboration of: