Project III. 2

MECHANICAL AND CHEMICAL SENSORS

Key Researchers: C. Tsamis, I. Raptis, P. Normand
Other Collaborating researchers: A. Tserepi
Post-doctoral scientists: S. Chatzandroulis, D. Goustouridis, E. Makarona, F. Farmakis
Phd candidate: R. Triantafyllopoulou,

Objectives:

- Development of micromachining processes for the realization of novel chemical and mechanical sensors
- Development of low power silicon sensors based on new materials and new processes
- Design, fabrication and testing of microsystems using silicon sensors
- Realization of sensors for specific industrial applications with emphasis on medical, food and automotive fields

Funding:

- EU - IST-FP6-STREP-027333 Micro2DNA, "I*ntegrated polymer-based micro fluidic micro system for DNA extraction, amplification, and silicon-based detection*", P. Normand
- GSRT – Technogenesis no 67, "*Miniature sensor for the control dangerous tire pressure and malfunction of ball bearings and breaks*"
- EU, IST, IP, GOODFOOD, "*Food Safety and Quality Monitoring with Microsystems*", contract No. 508774, C. Tsamis
- GSRT Greece-Italy bilateral cooperation "*Fabrication and characterization of an array of transparent conductive thin film polymeric composite as multiparametric sensitive layers for a new e-nose*", D. Goustouridis
- GSRT-PENED 03ED630, "*Micromachined chemical sensors for controlling food safety and quality*", C. Tsamis
- GSRT- ENTER 05EP032, "*Development of MOSFET type chemical sensors for wireless sensor networks*", C. Tsamis

RESEARCH in 2006

Research in 2006 focused in the following:

a) Alternative micro-hotplate design for low power metal-oxide (MOX) sensor arrays
b) Electronic ASIC for MOX Chemical Sensors
c) Polymer based chemical sensors
d) Capacitive Type Sensors
e) Suspended Cantilevers for Electromagnetic Energy Harvesters

RESEARCH RESULTS

A. Low power Metal-Oxide (MOX) Chemical Sensors based on Porous Silicon Micro-hotplates

R. Triantafyllopoulou, C. Tsamis, S. Chatzandroulis, F. Farmakis, and A. Tserepi

Solid state gas sensors based on SnO_2, are widely used in both indoor and outdoor applications, due to their low cost and high sensitivity. The sensing mechanism of these sensors is based on the changes of the conductivity of the SnO_2 sensitive layer, which is deposited between two electrodes, due to the adsorption of reducing or oxidizing agents onto its surface. One disadvantage of this type of sensors is that their operating temperature is in the range of 300-400^{0}C, which results in a considerable amount of power being consumed on an embedded heater. In order to reduce the power requirements of the sensor, the sensitive SnO_2 layer has been integrated on Porous Silicon Micro-hotplates, developed in the previous years. Porous Silicon provides improved thermal isolation, thus reducing heat dissipation to the substrate (Fig III.2.1).

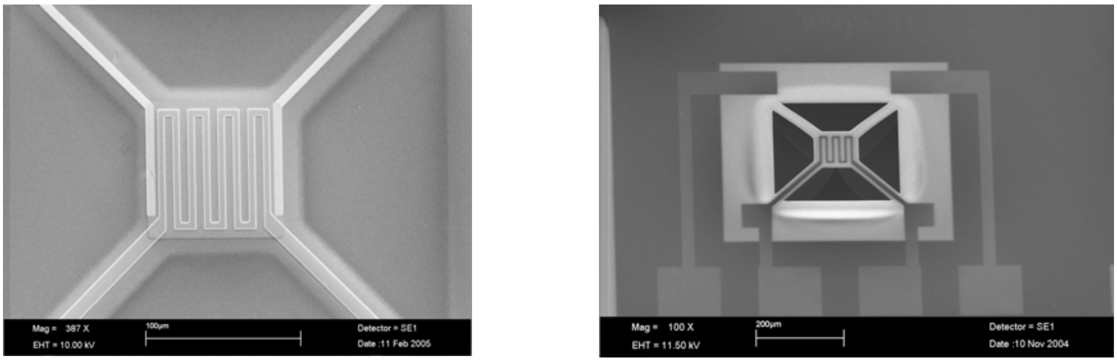

Fig. III.2.1: *(a) SEM image of a not-released PS micro-hotplate. We can see the sensitive material deposited of the membrane. (b) SEM image of a released PS micro-hotplate, with integrated heater and electrodes*

During this year, we have fabricated gas sensor devices for food safety and quality applications as well as for environmental monitoring. The response of the SnO_2 gas sensors was measured for the various gases (NH_3, CO and NO) and gas concentrations (50-500 ppm). Analysis was performed in isothermal mode (Fig. III.2.2(a)), by keeping constant the micro-hotplate temperature and in pulsed temperature mode (Fig. III.2.2(b)), by applying voltages pulses to the heater. In this case, the sensitivity and selectivity of the sensors was estimated as a function of the total shape of the pulse cycle, the duration of the pulses and the temperatures of the "hot" and the "cold" part of the measuring cycle. Very good sensitivity has been achieved with very low power consumption.

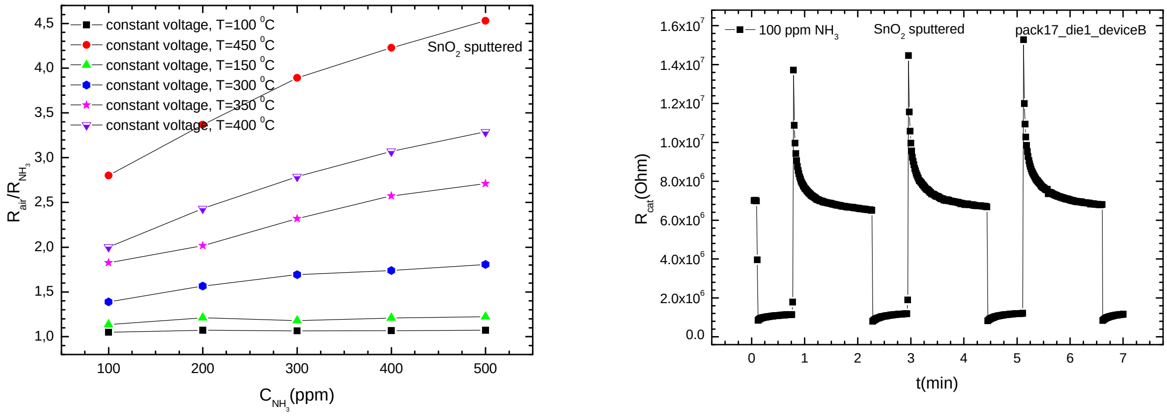

Fig. III.2.2: *(a) Sensor sensitivity as a function of the NH_3 concentration for temperatures between 100-450 ^{0}C, (b) Sensor resistance in pulsed operation mode for 100 ppm NH_3 and T_{hot}=350 ^{0}C, t_{hot}=40s and T_{cold}=100 ^{0}C, t_{cold}=90s.*

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B. Control and Electronics for MOX Chemical Sensors

P. Robogiannakis, S. Chatzandroulis and C.Tsamis

The correct operation of metal oxide (MOX) sensors requires that precise control over the operating temperature of the device is exercised simultaneously with the read-out of the chemically sensitive resistance. To this end, an electronic ASIC (Fig. III.2.3) has been developed able to interface a quad gas sensor array to a microcontroller and thereon to a PC or E-nose system. The chip contains in a single IC all the necessary analog electronics to operate four MOX sensors while the control logic will be implemented on the microcontroller (fig III.2.4). The ASIC has successfully been implemented in a single-poly, double-metal, 0.7um CMOS process (fig III.2.3) and first measured results are in agreement with simulated values. Provision has been taken for both polysilicon and Pt heater control.

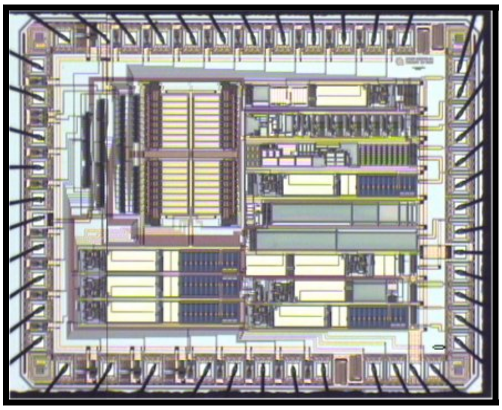

***Fig. III.2.3.*.** *Fabricated ASIC chip*

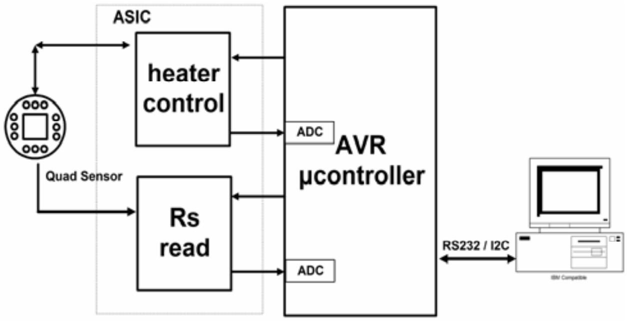

Fig. III.2.4: *Schematic of the MOX electronic control system.*

After testing of the basic functions of heater control and sensitive resistance readout of the gas sensor interface ASIC were tested, the chip was used with an actual commercial MOX sensor from Microsens. The sensor operates on a steady heater voltage of 1.68V (isothermal mode) provided from the ASIC. Changes in the sensitive resistance were sensed and transformed into a voltage signal (Vout). In Fig. III.2.5, measurements during exposure of the sensor to 220 ppm of NO gas are depicted.

Subsequently a PCB for the MOX control board which contains the ASIC and a microcontroller was designed. The completed PCB for the control and read out of a four MOX sensor array is depicted in Fig. III.2.6. The development of the firmware is underway and the microcontroller is able to transmit sensor values through the RS-232 serial port. The firmware includes independent heater driving in continuous mode, independent sampling for each sensor in the array and averaging of sensor measurements.

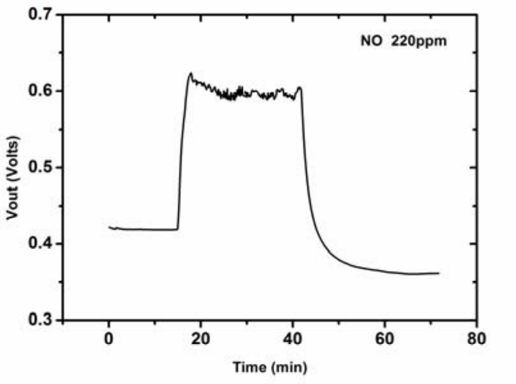

Fig. III.2.5: *Output of the ASIC with one commercial metal oxide sensor connected*

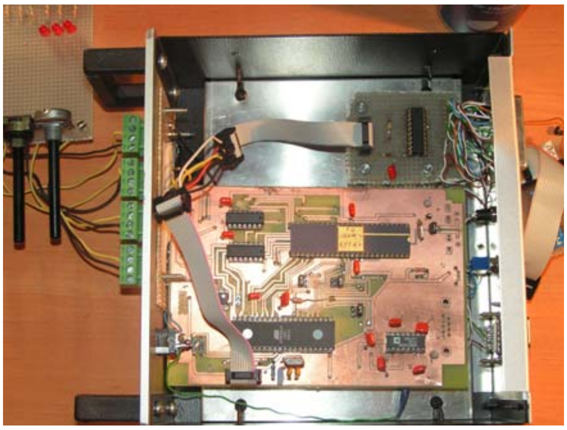

Fig. III.2.6: *ASIC and microcontroller PCB*

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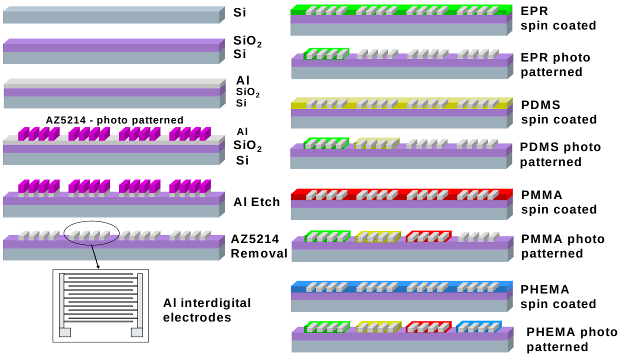
C. Polymer based chemical sensors

M.Kitsara, K.Manoli, D.Goustouridis, S.Chatzandroulis, I.Raptis

c_1 Capacitive Interdigitated Electrode Chemical Sensors

One of the simplest chemical sensing devices makes use of polymer coated InterDigitated Electrodes (IDEs) where the transduction mechanism relies on the permittivity changes and swelling of the coating polymer, to inflict a change in the capacitance between the two interdigital electrodes.

A single chip chemical sensor array based on IDEs has successfully been fabricated with conventional semiconductor processes (thermal oxidation, Al evaporation, optical lithography and wet etching). On those IDEs, polymers with photolithographic patterning processes have been applied as sensing layers. The use of a polymer as sensitive layer provides specific advantages compared to other chemical sensors. The major benefits include the ease of depositing the polymeric sensitive film to the desired geometric form and thickness due to use of lithographic methods. The whole fabrication, it is a batch process, compatible with microelectronic processes. In addition, polymers enable a good compromise between response time, selectivity and reversibility. The implementation of the whole sensor on Si wafers allows for future integration with CMOS signal conditioning circuits.



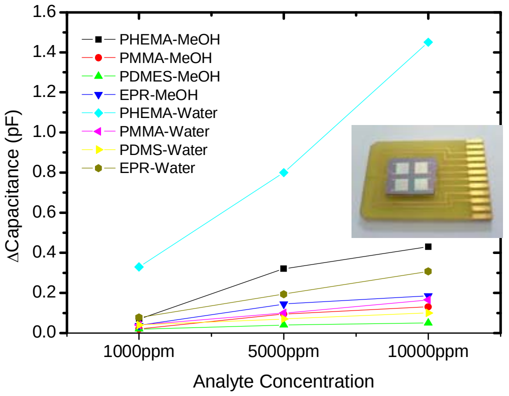
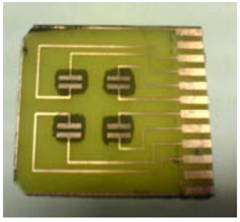


Fig. III.2.7: *Fabrication flowchart of the Inter Digitated Capacitor array*

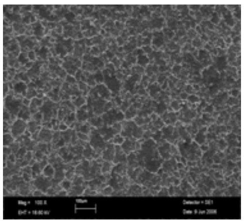
Fig. III.2.8: *a) Finished sensor array b) Response of the array to controlled concentrations (1000, 5000, 10000 ppm) of water and methanol vapors.5.*

c_2 Conductometric chemical sensors

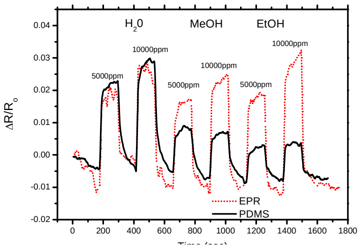
Conductometric chemical sensors based on polymer composite films, synthesized by adding conductive fillers to the polymer solutions and deposited between two predefined electrodes, are well established. Deposition of the sensitive composites is usually applied to sensor devices by solvent casting methods such as spin coating, spray coating and drop casting. These methods lack in pattern precision and repeatability. In order to overcome pertinent problems the application of conventional patterning methods for the fabrication of the conductive sensing array is proposed. In this work, we present the deposition of two conductive polymer composites (poly(dimethylsiloxane) /carbon black and epoxy polymer / carbon black on the same substrate. Each polymer composite is deposited onto two respective electrodes, effectively creating a conductive polymer chemical sensor pair. The two sensors performance is evaluated and considered as a first step towards the fabrication of a conductometric polymer composite array. Electrical vs. dimensional sensitivity issues and the significance of electrode configuration are considered.



Photograph of final sensor chip with four PDMS conductive polymer chemical sensors



SEM image of the patterned surface: PDMS



Response of both PDMS/CB and EPR/CB sensors to 5000 and 10000 ppm of water, methanol and ethanol vapors.

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D. Capacitive Type Sensors

S. Chatzandroulis, D. Goustouridis, D. Tsoukalas, P. Normand

d_1 *Pressure Sensors*

In 2006 part of our activities on silicon micromachined capacitive pressure sensors were devoted to the development of advanced finite element models to approximate experimental behaviour. Pressure sensors typically consist of a thin silicon diaphragm which deflects when a pressure differential is exerted, and are usually modelled as having completely immovable edges and rigid unbendable substrates. The model we developed follows a realistic device approach by taking into account the whole sensor die body, including the substrate, the intermediate layers and the foundation onto which the sensor diaphragm stands. It also includes all nonidealities and stress components of the micromachined device.

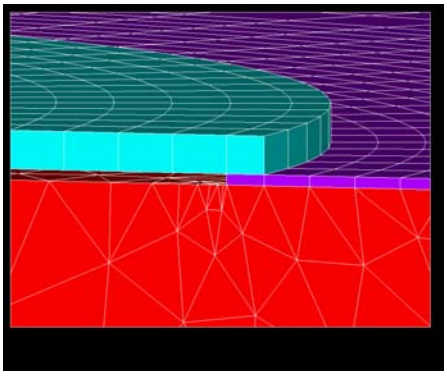

Fig. III.2.9: *Model detail of the sensor diaphragm rim lying on the oxide.*

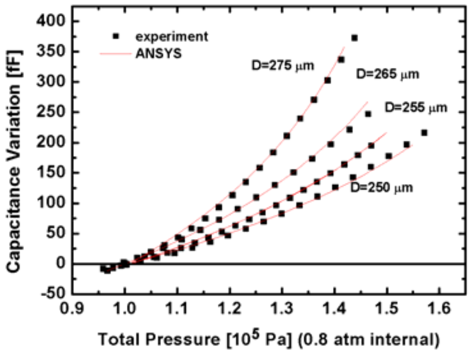

Fig. III.2.10: *Simulated and experimental sensor response after including residual stress for four sensor diaphragm diameters.*

d_2 *Capacitive DNA Sensors Arrays*

Unlabeled DNA detection has received a lot of attention in recent years as it simplifies sample preparation and testing procedures. To this end, we work towards exploiting surface stress changes and subsequent bending of microelectromechanical structures combined with capacitive detection. A first sensor employing capacitive detection and silicon micromachined membranes has been developed. The sensor accommodates in a single chip a capacitive DNA sensor array of 256 elements. Each sensor in the array consists of a single crystal silicon membrane that is covered with receptor DNA after surface functionalization and deflects upon exertion of surface stress hybridization. Membrane deflection is detected as a change in device capacitance. These activities are conducted within the frame of the European Project Micro2DNA.

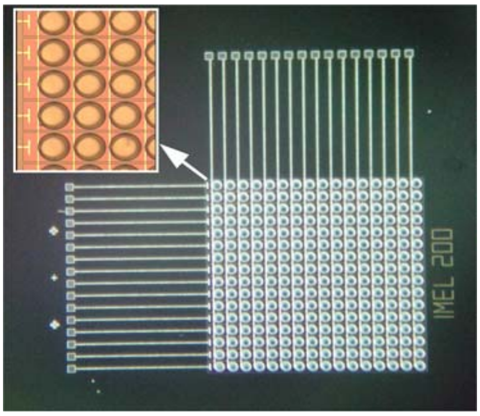

Fig. III.2.11: *Capacitive DNA array photo (top view) before Al deposition and functionalization.*

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E. Suspended Cantilevers for Electromagnetic Energy Harvesters

E. Makarona, C. Tsamis, S. Chatzandroulis, T. Speliotis, and D. Niarchos**

The proliferation of Micro-Electro-Mechanical Systems (MEMS) envisioned to be placed in remote locations, hazardous environments, or even within the human body raises the problem of power supply and data transmission. To solve this problem, sustainable power generation has to be achieved by harvesting ambient energy and converting into electrical energy. Our focus towards such a solution is the development of inertial electromagnetic micro-generators based on standard silicon technology.

The goal of this present work was the in-depth study of the engineering constraints and challenges one faces when integrating magnetic materials with silicon microstructures, as well as the determination of suitable fabrication conditions that would eventually allow the combination of optimal magnetic (fig III.2.12a) and mechanical (fig. III.2.12b) properties of the materials.FEM analysis of the structures was performed using Coventorware (fig. III.2.13a). Figure III.2.13b shows the simulated deflections as a function of the “effective” stresses that develop in the film. Estimation of the stresses that develop as a function of the growth conditions is obtained by comparison of the experimental and the simulation results. A process window has been determined in order to integrate magnetic material on suspended microcantilevers, with optimum magnetic and mechanical properties, suitable for electromagnetic microgenerators.

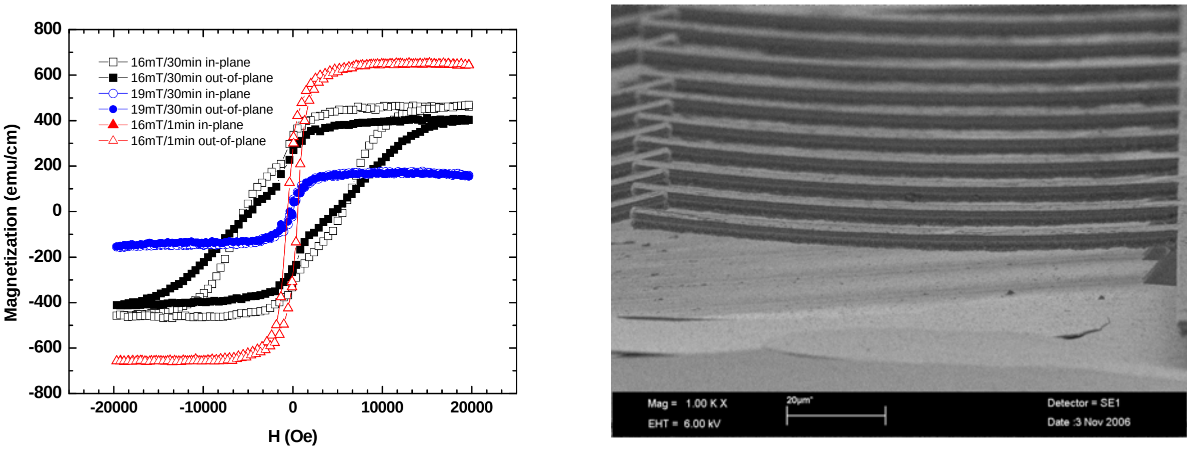

Fig. III.2.12: *(a) Magnetization loops for the in-plane (open) and out-of-plane (solid) magnetization as a function of the processing conditions, (b) Control of the stresses within the structure can be achieved through a compromise at the length of the annealing time and hence the magnitude of the magnetization of the magnetic film*

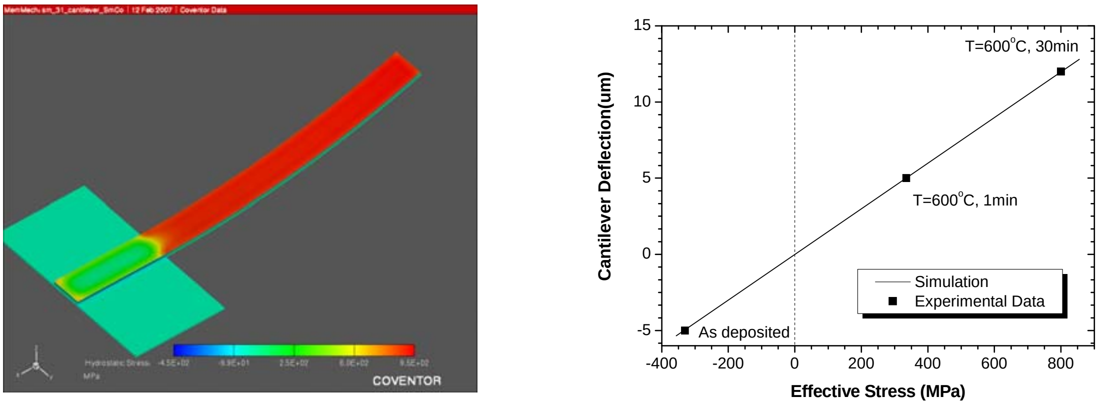

Fig. III.2.13: *(a) Stress distribution on the cantilevers as predicted from FEM analysis, (b) Estimation of the intrinsic stress from the comparison between simulated and experimental deflections.*

**This work is performed in collaboration with the Inst. of Material Science, NCSR “Demokritos”*

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PROJECT OUTPUT in 2006

Publications in International Journals and Reviews

1. “A Lithographic Polymer Process Sequence for Chemical Sensing Arrays”, M.Kitsara, D.Goustouridis, S.Chatzandroulis, K.Beltsios, I.Raptis, Microelectron. Eng. 83 1192(2006)
2. “Vapor sorption in thin supported polymer films studied by white light interferometry”, K.Manoli, D.Goustouridis, S.Chatzandroulis, I.Raptis, E.S.Valamontes, M.Sanopoulou, Polymer 47 6117(2006)
3. “Capacitive Pressure Sensors And Switches Fabricated Using Strain Compensated SiGeB”, S.Chatzandroulis, S.Koliopoulou, D.Goustouridis, D.Tsoukalas, Microelectron. Eng. 83, 1209 (2006).
4. "Capacitive Sensors", D.Tsoukalas, S.Chatzandroulis, D.Goustouridis, invited article in Wiley Encyclopedia of Medical Devices and Instrumentation, Second Edition, vol. 2, pp. 1-12 (2006).
5. “Alternative microhotplate design for low power sensor arrays”, R. Triantafyllopoulou, S. Chatzandroulis, C. Tsamis and A. Tserepi, Microelectronics Engineering, Vol. 83 , 1189 (2006)
6. “Integrated circuit interface for metal oxide chemical sensor arrays”, P. Robogiannakis, S. Chatzandroulis and C. Tsamis, Sensors and Actuators A: Physical, Volume 132, Issue 1, Pages 252-257 (2006)

Publications in Conference Proceedings

Conference Presentations

1. “Single chip interdigitated electrode capacitive chemical sensor arrays”, M.Kitsara, D.Goustouridis, S.Chatzandroulis, I.Raptis, R.Igreja, C.J.Dias, EuroSensors 2006, Sept. 2006, Goeteborg, Sweden (Poster)
2. “Fabrication of conductometric chemical sensors with a novel lithographic method”, N.Andreadis, S.Chatzandroulis, D. Goustouridis, K.Beltsios, I.Raptis, MNE 2006 Micro & Nano Engineering 2006, September 2006, Barcelona, Spain (Poster)
3. “Patterning of PDMS/carbon black conductive polymer composite for chemical sensors fabrication”, N.Andreadis, S.Chatzandroulis, D.Goustouridis, K.Beltsios, I.Raptis, Micro Process & Nanotechnology 2006 October 2006, Kanagawa, Japan (Poster)
4. ”Impact Of Structural Parameters On The Performance Of Silicon Micromachined Capacitive Pressure Sensors”, G. Bikakis, V. Tsouti, S. Chatzandroulis, D. Goustouridis, P. Normand, D. Tsoukalas, in Eurosensors XX in Göteborg, Sweden, September 17-20, 2006.
5. “SnO2 sensors integrated on porous Si microhotplates to detect NH3”, M. C. Horrillo, I. Sayago, J.P. Adrados, J. Gutiérrez, R. Triantafyllopoulou, S. Chatzandroulis, C. Tsamis, Eurosensors XX, Goteborg, Sweden, September 17-20, 2006 (Oral)
6. “Pulsed mode operation of low power SnO2 sensors for improved gas selectivity”, R. Triantafyllopoulou, C. Tsamis, S. Chatzandroulis, M. C. Horrillo, J. Gutiérrez, Micro- and Nano-Engineering, MNE 2006, 17-20 September 2006, Barcelona, Spain (Poster)
7. “Implementation of hard magnetic thin films on suspended cantilevers for electromagnetic energy harvesters”, E. Makarona, T. Speliotis, A. Darsinou, C. Tsamis, S. Chatzandroulis and D. Niarchos, SPIE Conference on “Microtechnologies for the New Millenium 2005”, 2-4 May 2007, Maspalomas, Spain (Accepted for Oral presentation)

M. Sc. theses

1. “Sequential polymer lithography for chemical sensor arrays”, M. Kitsara, Chemistry Dept./Uni. Athens, December 2006, Supervisors: I. Raptis, K. Beltsios
2. “Design and optimization of silicon nitride micro-hotplates for chemical sensors”, N. Tokpasidou, Dept. of Informatics/Univ of Athens (April 2006), Supervisor: C. Tsamis
3. “Micromachining techniques in Germanium substrates for sensors and nano-devices”, A. Konstantopoulou, Dept. of Informatics/Univ of Athens (July 2006), Supervisor: C. Tsamis

Diploma theses

1. “Characterization and modeling of suspended microstructures for sensor applications”, N. Andreadis, Materials Dept./Uni. Ioannina, September 2006, Supervisors: I. Raptis, K. Beltsios
2. “Fabrication and Characterization of Micromachined Chemical Sensors”, J. Kokkinis, Materials Dept./Uni. of Ioannina, October 2006, Supervisor: C. Tsamis
3. “Fabrication and characterization of vibrational energy harvesters based on piezoelectric elements”, P. Papandreou, Materials Dept./Uni. of Ioannina, October 2006, Supervisors: E. Makarona/ C. Tsamis

Patents

1. “A Method to Deposit Multitude Polymer Materials for Chemically Sensitive Arrays”, I.Raptis, D.Goustouridis, S.Chatzandroulis, M.Kitsara (OBI 20060100040)
2. “Capacitive pressure-responsive devices and their fabrication”, S. Chatzandroulis, D. Goustouridis, D. Tsoukalas, P. Normand, Israel Patent No 151277, Publication date: 01-08-2006.