# Project III. 2A: MECHANICAL AND CHEMICAL SENSORS

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# 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, "Integrated polymer-based micro fluidic micro system for DNA extraction, amplification, and silicon-based detection", P. Normand

- 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

# Main Activities in 2008:

In 2008, our main activities were focused on the following tasks:

- A. Low power Metal-Oxide (MOX) Chemical Sensors
- B. FET-type chemical sensors for wireless applications
- C. Polymer based chemical sensor arrays
- D. Capacitive Type Sensors
- E. Microfabricated electrochemical sensors

# **Research activities and main results within 2008**

#### A. Low power Metal-Oxide (MOX) Chemical Sensors\*

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

#### \* In collaboration with Department of Electronics, University of Barcelona (J. R Morante)

Solid state chemical sensors are one of the most common devices employed for the detection of hazardous gases, like  $NH_3$ , CO and NO. Their principle of operation is based on the changes of the conductivity of a sensitive material, which is deposited between two electrodes, due to the adsorption of reducing or oxidizing agents onto its surface. Many techniques have been developed for the deposition of catalytic materials. The most widely used techniques to deposit a sensitive material are either by sputtering or by microdropping. In the first case thin films were prepared by reactive r.f. magnetron sputtering using a 99.9%

pure SnO<sub>2</sub> target (Fig.1a). In the second case sensitive materials are prepared by a sol-gel solution with metal additives, in order to enhance its sensitivity, and then deposit the additive-modified nanostructured metal oxides on micro-hotplates, by microdroping. In this way, the use of Porous Silicon micro-hotplates allows for the fabrication of sensor arrays (Fig. 1b) that incorporate varying sensitive materials, while at the same time they exhibit a significant reduction of the power consumption.





Fig. 1. SEM image (a) of the sputtered SnO<sub>2</sub> deposited on a micro-hotplate and (b) of a sensor array with microdropped nanostructured sensitive materials SnO<sub>2</sub>:Pd and WO<sub>3</sub>:Cr, mounted on a package.

During this year, we developed gas sensors for food safety and quality applications as well as for environmental monitoring, fabricated by sputtering and micro-dropping. The sensors are based on suspended Porous Silicon micro-hotplates. Porous Silicon provides improved thermal isolation, thus reducing heat dissipation to the substrate. For further reduction of power consumption, various methodologies have been developed, such as alternative measuring techniques to constant temperature operation, such as pulsed-temperature mode. In Fig. 2a the response of the sputtered SnO<sub>2</sub> gas sensors towards NO is shown, operating in pulsed temperature mode, 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. Sensors were characterized in CO and NO ambient, for gas concentrations (100-500 ppm). Operation in pulsed temperature mode, results in higher sensor sensitivity and enhanced selectivity towards NO, with reduced power consumption. In Fig. 2b the response of the two nanostructured sensitive materials SnO<sub>2</sub>:Pd and WO<sub>3</sub>:Cr towards NH<sub>3</sub> is shown, for gas concentrations (2-15 ppm). The sensors operated in isothermal mode, by keeping constant the micro-hotplate temperature. The gas sensors with micro-dropped sensitive materials and especially SnO2:Pd exhibit the highest sensitivity towards NH<sub>3</sub>, with lower power consumption.



**Fig. 2:** (a) Sensor response towards NO for pulsed temperature operation mode for sputtered SnO<sub>2</sub> sensitive material, (b) response of gas sensors with SnO<sub>2</sub>:Pd and WO<sub>3</sub>:Cr micro-dropped sensitive materials, in isothermal operation, for low concentrations of NH<sub>3</sub> (2-15 ppm).

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#### B. FET-type chemical sensors for wireless applications

F. Farmakis, K. Alexandrou, C. Tsamis, M. Kompitsas<sup>1</sup>, I. Fasaki<sup>1</sup>, P. Jedrasik<sup>2</sup>, G. Petersson<sup>2</sup>, B. Nilsson<sup>2</sup>

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Chemical sensors for wireless applications are of major interest since they turn into reality the possibility to sense and monitor environmental changes in hard-accessible mediums or even to escort environment-sensitive products (such as food) in order to monitor and build environment-related database. Low-energy consumption (low current operation), room-temperature operation as well as integration in small dimension are some of the most important requirements of such sensors. To this end, we investigate two candidate devices: i) MOS capacitive sensors and ii) FET-type sensors with active catalytic layer.

During this year, we focused our efforts on the fabrication of interdigitated bottom-gate FET devices (gasFET) with various channel lengths (from 0.3  $\mu$ m to 2  $\mu$ m) (Figure 3a). On the top of the interdigitated electrodes, a 50-nm-thick zinc oxide layer was grown by pulsed laser deposition, as the active layer. Alternatively, Au-doped ZnO was also grown in order to investigate the influence of Au doping in ZnO. Figure 3b shows an AFM image of as-deposited thin ZnO film grown on oxidized silicon wafer. The average grain size of around 30-40 nm is not affected by the annealing process due to the low (400°C) temperature used. Surface roughness at 22 nm is slightly reduced after annealing. The indicated high surface-to-volume ratio favors the application of such films as gas sensors since the chemical active area is enhanced.

Figure 4a shows a comparison in CO sensing between a ZnO gas sensor without Au and with Au nanoparticles. It has to be noted that both sensors were held at  $200^{\circ}$ C during the experiments and that the CO was introduced with dry air. It is easily observed that the sensitivity is more than 1.5 times higher for the sensor with the Au nanoparticles. In addition, the response time t<sub>90</sub> (t<sub>90</sub> is defined as the time needed for the signal to achieve the 90% of its final value) is 3 times shorter for the ZnO/Au nanoparticles gas sensor. Figure 4b shows the comparison of the sensitivity between the two samples for various values of CO concentration. In both cases the gas sensor sensitivity seems to be linearly depended on the CO concentration in dry air at  $200^{\circ}$ C. However, for the Zn/Au gas sensor the slope of the sensitivity is 4.8 x10-4 ppm<sup>-1</sup>.





(b)

**Fig. 3:** (a) SEM images showing the interdigitated source and drain electrodes of the bottom gate FET device. (b) AFM image picture of as-deposited thin ZnO film grown on oxidized silicon wafer. The average grain size of around 30-40 nm is not affected by the annealing process due to the low (400<sup>o</sup> C) temperature used.



**Fig. 4:** (a) Sensitivity vs time for ZnO sensors with and without gold nanoparticles. The CO concentration is was 3800 ppm (VDS=6 V, VGS=0 V) and measurements were performed at 200°C. (b) Drain current increase (sensitivity) against CO concentration for ZnO sensors with and without gold nanoparticles. The sensors were at 200 °C (VDS=6 V, VGS=0 V).

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#### C. Polymer Based Gas sensors

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Capacitive-type chemical sensors rely on changes in the dielectric properties of the sensing polymeric layer due to absorption of VOCs. Such polymer-based chemocapacitive sensors are promising devices in terms of processability, low fabrication cost, reversibility and the wide range of material choice, commercially available or to be tailor-made, to meet the needs of specific VOC applications.

Our research aims at i) the development of capacitive sensor arrays based on InterDigitated Electrodes (IDEs) coated with polymers as sensing layers and ii) on the development of the necessary hardware – software for the measurement of these arrays. In this framework our research was focused on a) development and characterization of arrays with 4 sensors coated with different polymers b) development of methodologies for the sensitivity enhancement and c) development of PCA algorithms for substance identification.

Arrays of 4 IDEs were fabricated with conventional microelectronic processing technology and coated with polymeric films. The response of the sensor array in various analytes is illustrated in fig. 5. The response clearly depends on the polymer-analyte combination.





Fig. 5: Equilibrium capacitance response of Fig. 6: PCA analysis of the sensor array IDC array to 5000 ppm of each of the four analytes. Initial capacitance of the polymer layers Cp (pF): 11 (PBMA); 9 (PDMS-OH); 80 (PEI); 40 (PHEMA).

responses to 5000 ppm of the four analytes and their 1:1 mixture

Due to the inherently partial sorption selectivity of the sensing polymeric materials, it is necessary to apply a data processing technique to the responses of the sensor array, in order to enhance its discriminating ability towards complex vapor environments. A Principle Component Analysis (PCA) algorithm was developed to improve in the discrimination capability of the sensor array. PCA analysis of the capacitance responses upon exposure of the sensor array to single analytes and to mixtures is shown in Fig. 6. The plot, containing 92.7 % of the total variance of the data, depicts distinct separate clusters for the different analytes and their binary mixtures.

Furthermore, innovative InterDigital Chemocapacitive (IDC) sensors, based on polymer layers filled with various amounts of BaTiO<sub>3</sub> nanoparticles were investigated in order to evaluate the effect of incorporated BaTiO<sub>3</sub> on the sensitivity and selectivity of the pure polymer-based sensors. In fig. 7, optical micrographs of sensors with polymeric layers with and without BaTiO<sub>3</sub> are shown. The capacitance changes  $\Delta C$  of a pure PBMA sensor, as well as of composite PBMA -BaTiO<sub>3</sub> sensors, upon exposure to 5000 ppm of various analytes, are shown in Fig. 8. As expected, in the more hydrophobic PBMA polymer, exhibiting a higher sorption capacity for the less polar analytes, the capacitance response of the pure PBMAsensor decreases with increasing polarity of analyte.



Fig. 7: Optical micrograph of the interdigitated electrodes coated with pure PBMA and PBMA/ BaTiO<sub>3</sub> 10%v/v composite.



8: Effect of BaTiO3 load Fig. on capacitance response of PBMA- based sensors to 5000ppm of ethyl acetate, ethanol, methanol and water vapors.

# **D. Capacitive Type Sensors**

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#### Capacitive DNA Sensors Arrays

Unlabeled DNA detection has been the focus of great interest in recent years as it simplifies sample preparation and testing procedures. To this end and within the framework of the European Project Micro2DNA, we have developed a capacitive type chemical/biological sensor array organized in a 16 x 16 sensor matrix. Each sensor in the array consists of a single crystal silicon membrane which is able to sense surface stress changes which could be exerted by either a chemically sensitive layer or the interaction between immobilized biological species with other target biological species. In the first case the array is operated as a chemical sensor array (figure 9), while in the second case it is operated as a DNA sensor. In figure 10, CD19 bThalassemia normal and mutated probes were immobilized on the thin membrane surface. The sensor was then exposed to PCR containing the CD19 oligos and was hybridized.

Furthermore, in order to facilitate portable measurement the developed dedicated ASIC for the readout of the whole 16x16 array has been incorporated on a PCB together with a microcontroller. The board can then be controlled from a PC/laptop using Labview and a standard USB port.



Capacitive Strain Sensors

humidity and Volatile Organic Compounds (VOCs) have been deposited using the Laser Induced Forward Transfer (LIFT) technique. As analyte molecules are absorbed in the polymer film the membrane deflects causing a change in capacitance between membrane and substrate. The response to water vapors of membranes of 250µm diameter partially covered with four different polymers.

Fig. 9: Polymeric films sensitive to

Fig. 10: Hybridization of the CD19 bThalassemia oligonucleotide. CD19 normal and mutated probes (50uM) are first immobilized on the thin membrane surface of different sensors in the array at IMBB/FORTH. Then the PCR product (72nM) is inserted into the hybridization chamber and over the sensor. In the figure the region of the curves after the insertion of the PCR and obtaining the baseline is depicted. The sensor signal remains stable at first and then decreases for both mutated and normal as well as the reference. However the decrease in the normal CD19 is by far larger than the other two.

Online strain measurement is sought for in many industrial, aerospace and civil applications where monitoring of an engineering structure health is crucial in maintaining its integrity and avoiding catastrophic structural failure. Measuring strain on selected places of a structure can give information about overall deformation and lead to early detection of potential damage. This in effect reduces periodic inspection costs while, at the same time, increasing safety. In this work a robust capacitive type strain sensor is demonstrated which consists of a cavity

In this work a robust capacitive type strain sensor is demonstrated which consists of a cavity etched in a thick wet oxide, a fixed electrode and a thin Si membrane that seals the cavity and operates as a flexible electrode. When the flexible electrode is under stress, it deflects and the device capacitance changes. The whole structure is mounted onto a flexible film and is subsequently embedded into a solid layer of PDMS (polydimethylsiloxane) polymer. The latter covers the sensor in its entirety and is used to transfer stress to the flexible electrode from the surface under measurement. The device exhibits linear behavior with a gauge factor of 1.0 and sensitivity of 4.1 fF/mstrain.



*Fig. 11:* Capacitance *vs.* Strain for a downwards deflected cantilever end.



*Fig. 12:* Strain sensor mounted on the flexible structure: a) schematic, b) photographic view

# E. Microfabricated electrodes for stripping voltametry

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Stripping voltammetry (SV) has proved a powerful technique for the determination of trace metals in samples of environmental, clinical and industrial origin. Mercury film electrodes (MFEs) and the hanging mercury drop electrode have been traditionally used in SV, based on the ability of mercury to form amalgams with many heavy metals. However, the increased risks associated with the use, manipulation and disposal of metallic mercury or mercury salts have led to the search for alternative more environmentally friendly electrode materials. In this direction Bismuth electrodes appear to be an alternative environmental friendly approach exhibiting comparable performance to MFEs.

Methodologies were developed for the parallel production of sputtered bismuth electrodes with predefined geometry based on photolithography and paying particular attention to the preservation of sensing properties of the deposited thin bismuth films (fig. 13). Furthermore developed patterning approaches were further extended to the integration of the reference and counter electrodes on the same chip (fig. 14). With the developed technology disposable three-electrode cells were developed and successfully evaluated at Chemistry Dept., University of Athens for the trace determination of Ni.



*Fig. 13 :* Cross section and top-view of the bismuth sputtered sensor



# PROJECT OUTPUT in 2008

#### **Publications in International Journals**

- "Structural and sensing properties of nanocrystalline SnO2 films deposited by spray pyrolysis from a SnCl2 precursor", Gaiduk, P.I., Kozjevko, A.N., Prokopjev, S.L., Tsamis, C., Nylandsted Larsen, A., Applied Physics A: Materials Science and Processing 91 (4), pp. 667-670 (2008)
- [2] "Nanostructured Oxides on Porous Silicon Microhotplates for NH3 Sensing", R. Triantafyllopoulou, X. Illa, O. Casals, S. Chatzandroulis C. Tsamis, A. Romano-Rodriguez and J.R. Morante, Microelectronic Engineering 85 (5-6), pp. 1116-1119 (2008)
- [3] "Field-effect transistors with thin ZnO as active layer for gas sensor applications", F. V. Farmakis, A. Speliotis, K. P. Alexandrou, C. Tsamis, M. Kompitsas, I. Fasaki, P. Jedrasik, G. Petersson, B. Nilsson, Microelectronic Engineering 85 (5-6), pp. 1035-1038 (2008)
- [4] "Disposable mercury-free cell-on-a-chip devices with integrated microfabricated electrodes for the determination of trace nickel(II) by adsorptive stripping voltammetry" Ch.Kokkinos, A.Economou, I.Raptis, Th.Speliotis Anal. Chem. Acta 622 111(2008)
- [5] "Lithographically-fabricated disposable bismuth-film electrodes for the trace determination of Pb(II) and Cd(II) by anodic stripping voltammetry" Ch.Kokkinos, A.Economou, I.Raptis, C.E.Efstathiou Electroch. Acta. 53 5294(2008)
- [6] "Surface modification of polymeric thin films at SiO2 interfaces with vacuum ultraviolet light" E.Sarantopoulou, J.Kovač, Z.Kollia, I.Raptis, S.Kobe, A.C.Cefalas Surf. Interface Anal. 40 400(2008)
- [7] "Surface nano/micro functionalization of PMMA thin films by 157 nm irradiation for sensing applications" E.Sarantopoulou, Z.Kollia, A.C.Cefalas, K.Manoli, M.Sanopoulou, D.Goustouridis, S.Chatzandroulis, I.Raptis Appl. Surf. Sci. 254 1710(2008)
- [8] Design and fabrication of a Si micromechanical capacitive array for DNA sensing", V.Tsouti, S.Chatzandroulis, D.Goustouridis, P.Normand, D.Tsoukalas, Microelectronic Engineering, 85 (5-6), pp. 1359-1361 (2008)
- [9] "Direct laser printing of biotin microarrays on low temperature oxide on Si substrates", C.Boutopoulos, P.Andreakou, D.Kafetzopoulos, S. Chatzandroulis, I.Zergioti, Physica Status Solidi (a), vol. 205, no. 11, pp. 2505 – 2508, (2008)
- [10] "Liquid phase direct laser printing of polymers for chemical sensing applications", C.Boutopoulos, V.Tsouti, D. Goustouridis, S.Chatzandroulis and I.Zergioti, Appl. Phys. Lett. 93, 191109 ,2008.
- [11] "Detection of the Biotin-Streptavidin interaction by exploiting surface stress changes on ultrathin Si membranes", V.Tsouti, C.Boutopoulos, P.Andreakou, M.Ioannou, I.Zergioti, D.Goustouridis, D.Kafetzopoulos, D.Tsoukalas, P.Normand, S.Chatzandroulis, Microelectron. Eng., In Press, Available online 7 December 2008

#### Publications in International Conference Proceedings

- [1] "Detection of CO and NO Using Low Power Metal Oxide Sensors", R. Triantafyllopoulou, and C. Tsamis, Phys. Stat. Sol. (c) 5, No. 12, 3647–3650 (2008) / DOI 10.1002
- [2] F.V. Farmakis, K. Alexandrou, C. Tsamis, Th. Speliotis, I. Fasaki, M. Kompitsas, S. Kennou, S. Ladas and P. Jedrasik, "Gas sensing properties of ZnO filed-effect transistor enhanced by Au nanoparticles", EUROSENSORS XXII Proceedings, p. 1011-1013 (2008)
- [3] "Lithographically-Fabricated Bismuth-Film electrodes as disposable Mercury-Free voltammetric sensors for trace analysis of Pb(II)" Ch.Kokkinos, A.Economou, I.Raptis, Th.Speliotis Sensing in Electroanalysis, Uni. Pardubice (Czech Republic) 3 91(2008)
- [4] "Interdigital chemicapacitive sensors based on polymer/BaTiO3 composites" K.Manoli, P.Oikonomou, D.Goustouridis, E.Karonis, I.Raptis, M.Sanopoulou EuroSensors 2008 (Dresden, Germany, 09/2008)

- [5] "Evaluation of a chemocapacitive sensor array for the detection of vapor analytes and their mixtures" K.Manoli, E.Karonis, M.Chatzichristidi, D.Goustouridis, S.Chatzandroulis, I.Raptis, M.Sanopoulou IEEE Sensors 2008 (Lecce, Italy, 10/2008)
- [6] "A Capacitive Biosensor Based On Ultrathin Si Membranes", V.Tsouti, C.Boutopoulos, P.Andreakou, M.Ioannou, I.Zergioti, D.Goustouridis, S.Chatzandroulis, J.Hue, R.Rousier, D.Kafetzopoulos, D.Tsoukalas and P.Normand, The Seventh IEEE Conference On Sensors, IEEE SENSORS, October, 26-29, 2008 Lecce, Italy
- [7] "Capacitive Strain Sensors Using Polymer Embedded Thin Si Membranes<sup>\*</sup>, P.Broutas, D.Goustouridis, S.Chatzandroulis, P. Normand, D. Tsoukalas, EUROSENSORS XXII, Dresden, Germany 7-10 September 2008
- [8] "A Chemical Sensor Array Based On Si/Polymer Bimorphs", V.Tsouti, S.Chatzandroulis, D.Goustouridis, P.Broutas, P.Normand, C.Boutopoulos, I. Zergioti, D. Tsoukalas, EUROSENSORS XXII, Dresden, Germany 7-10 September 2008

# **Conference Presentations**

- [1] "Gas sensing properties of ZnO filed-effect transistor enhanced by Au nanoparticles", F.V. Farmakis, K. Alexandrou, C. Tsamis, Th. Speliotis, I. Fasaki, M. Kompitsas, S. Kennou, S. Ladas and P. Jedrasik, EUROSENSORS XXII, Dresden, Germany, September 7-10 2008 (poster)
- [2] "Direct laser printing of polymer materials for chemical sensing applications", C. Boutopoulos, V. Tsouti, S. Chatzandroulis, D. Goustouridis, P. Normand, D. Tsoukalas, and I. Zergioti, , poster presentation in the 34th International Conference on Micro and Nano Engineering (MNE08), Athens, Greece, September 15-18, 2008.
- [3] "Detection of the Biotin-Streptavidin interaction by exploiting surface stress changes on ultrathin Si membranes", V.Tsouti, C.Boutopoulos, P.Andreakou, M.Ioannou, I.Zergioti, D.Goustouridis, D.Kafetzopoulos, D.Tsoukalas, P. Normand, S.Chatzandroulis, poster presentation in the 34th International Conference on Micro and Nano Engineering (MNE08), Athens, Greece, September 15-18, 2008.
- [4] "Direct laser printing of polymers for chemical sensing applications", C.Boutopoulos, V.Tsouti, S.Chatzandroulis, D.Goustouridis and I. Zergioti, oral talk in the 16th international conference on Advanced Laser Technologies (ALT08), Siofok, Hungary, September 13-18, 2008.
- [5] "Laser microprinting on chemical and bio sensors", C. Boutopoulos, I. Zergioti, P. Andreakou, V. Tsouti, S. Chatzandroulis D. Goustouridis, D. Kafetzopoulos, oral talk in the 9th Symposium on Laser Precision Microfabrication (LPM 2008), Quebec, Canada, June 16 20, 2008.
- [6] "Laser induced forward transfer on capacitive sensors", I. Zergioti, C. Boutopoulos, P. Andreakou, D. Tsoukalas, D. Goustouridis, V. Tsouti, S. Chatzandroulis, D. Kafetzopoulos, oral talk in the symposium B, EMRS 2008 Spring Meeting, Strasbourg, France, May 26 May 30, 2008.
- [7] "A Biosensor Based on Surface Stress Changes of Ultrathin Silicon Membranes", V. Tsouti, C. Boutopoulos, P. Andreakou, M. Ioannou, I. Zergioti, D. Goustouridis, S. Chatzandroulis, D. Kafetzopoulos, D. Tsoukalas, P. Normand, XXIV Panhellenic Conference on Solid State Physics and Materials Science Heraklion, Crete, September 21-24, 2008

#### Ms Thesis

C.Boutopoulos, "Laser induced deposition of biomaterials with application to biosensors" (Εναπόθεση Βιοϋλικών με LASER με Εφαρμογές σε Βιοαισθητήρες), NTUA/SEMFE

# **Conference Organization**

*MRS-Spring Meeting 2008, Symposium PP: The Business of Nanotechnology*, March 24 – 28, San Francisco (USA),

Organizers: L. Merhari (Ceramec), A. Gandhi (Applied Materials), S. Giordani (TTP Lab), L. Tsakalakos (General Electric), **C. Tsamis** (IMEL/NCSR "Demokritos")