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# A chemical sensor microarray realized by laser printing of polymers

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ABSTRACT

The fabrication of chemical sensor arrays using laser induced forward transfer (LIFT) as a means to deposit polymer layers to be used as chemically sensitive material is presented. LIFT is a direct, maskless technique offering high spatial resolution printing of a wide range of polymers and in this work it has been applied in the fabrication of a capacitive chemical sensor array with the ability to host up to 256 sensing sites. Each sensor in the array is composed of a Si membrane covered by a thin low temperature oxide layer and a polymer layer. Owing to the large number of sensing sites there is room for both sensitive polymer layer diversity as well as multiplicity, therefore expanding the capability for odour recognition. As a demonstration, a variety of polymers has been spotted on sensor arrays and tested during exposure to various concentrations of water, methanol and ethanol vapours. The deposited polymers showed good sensitivity depending on the analytes tested and the geometrical characteristics of the composite membranes.

## 1. Introduction

Chemical sensor arrays are of great interest due to their wide range of applications to food industry, medical diagnosis and environmental monitoring [1,2]. Composite structures, such as Si/polymer bimorphs, have been extensively used as chemical sensors either in the form of cantilever arrays covered with sensitive polymers [3–8] or in the form of thin membranes [9,10]. Their operation relies on the ability of the covering sensitive polymer to absorb gaseous chemical compounds (analytes). During the absorption, the polymer expands and induces a change in the surface stress of the underlying Si structure causing it to deflect or change resonance frequency. Upon desorption of the analyte the cantilever/membrane returns to its initial position. Parameters influencing the response of such sensors include the rate by which analyte molecules diffuse into the polymer layer, which mainly depends on the size and solubility of the molecules in the polymer layer. The cantilever/membrane response also depends on other parameters such as the mechanical properties of the structure as well as the changes inflicted on the mechanical properties and volume of the polymer during absorption of analyte molecules [11]. Therefore, depending on the potential application, a wide range of polymers can be selected for covering arrays of cantilevers or membranes and increasing the final selectivity.

The deposition of the chemical sensing material is one of the most important challenges for the fabrication of chemical sensors in general and Si/polymer bimorphs in particular, as different polymer materials have to be selectively deposited on different spots of the sensor arrays. The inkjet printing method has been widely applied in chemical sensing [12–14] but presents several limitations regarding the spatial resolution that may be achieved during printing, the properties of the solvents to be used as well as the polymer molecular weight and concentration [15]. On the other hand, alternative printing methods based on polymer lithography [16,17] are time consuming and expensive. Laser induced forward transfer (LIFT) is a technique, which enables the controlled transfer of a thin film of a liquid or solid material, from a transparent carrier, to a receiving substrate. It was first demonstrated by Bohandy et al. [18] for metal deposition and has become important for high resolution printing of a wide range of materials [19,20]. The dry printing of polymer materials by means of LIFT has been demonstrated using nanosecond pulses for the fabrication of organic light-emitting diodes [21] and organic thin-film transistors [22]. Recently, we have reported the possibility of using LIFT for liquid phase printing of polymer materials by using a pulsed Nd:YAG laser (266 nm wavelength, 4 ns pulse duration) [23]. Printing the chemically sensitive polymers on the sensor membrane by liquid phase LIFT offers significant advantages including high spatial resolution, contactless and maskless printing as well as material waste minimization.

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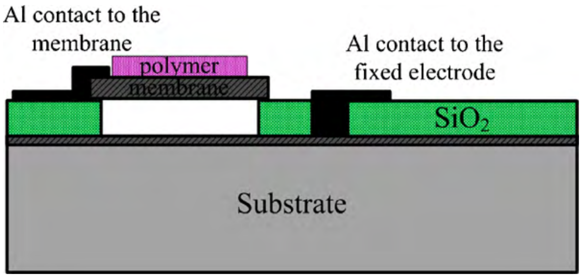

**Fig. 1.** Schematic of the polymer/membrane bimorph.

In the present work a 16 × 16 capacitive chemical sensor array is presented. The array consists of ultra-thin circular Si membranes onto which polymeric films sensitive to humidity and volatile organic compounds (VOCs) have been deposited in a controlled manner using the LIFT technique. Various polymers have been deposited on membranes of the same sensor array. These are poly(2-hydroxyethyl methacrylate) (PHEMA), polyacrylic acid (PAA), poly(4-vinylpyridine) (P4VP), poly(vinylpyrrolidone) (PVP), poly(vinyl chloride-co-vinyl acetate) (PVC-co-PVA), poly(hydroxyl styrene) (PHS) and poly(methyl methacrylate) (PMMA).

## 2. Experimental

### 2.1. Sensor description

The fabrication of the sensor array relies on the silicon fusion bonding of an SOI wafer and another wafer with a strain compensated SiGeB epitaxial layer. Details on the process may be found in [24]. The basic sensing element in the array (depicted in Fig. 1) is a polymer/Si membrane. The circumference of each membrane is supported onto a thick $SiO_2$ layer with a corresponding circular cavity underneath. A capacitive chemical sensor is created then between the membrane and a counter electrode, formed in the underlying substrate using phosphorus doping and by depositing appropriate chemically selective layers on the upper membrane surface. In addition, each membrane is passivated by a low temperature oxide (LTO) layer, which allows the use of the array in a liquid environment. Furthermore, due to its hydrophilic properties, the LTO provides a very suitable substrate for the polymer deposition technique. When the sensor is exposed to water or VOC vapours, the polymer absorbs the corresponding vapour molecules, swells and its mechanical properties change, resulting in a change of the membrane deflection and thus the measured capacitance. Each sensing element, like the one shown in Fig. 1, is part of a 256-sensor array arranged in 16 rows and 16 columns, while each sensor membrane may be covered by a different sensitive polymer. By collecting the varying response of each sensor in the array it is possible to obtain the “signature” of complex odours and eventually arrive at effective odour identification.

### 2.2. Polymer deposition

The deposition of the sensitive polymers onto the silicon micromechanical capacitive sensors was carried out by employing the experimental apparatus depicted in Fig. 2. The apparatus comprises a pulsed Nd:YAG laser (266 nm wavelength, 4 ns pulse duration) and a high power imaging micromachining system. A multi-target holder was mounted onto a computer-controlled *x*–*y* translation stage, allowing a maximum 25 mm × 25 mm movement and having a 1 μm positioning resolution. An in-house program was used in order to synchronize the *x*–*y* motion with the laser repetition rate at 2 Hz. The laser beam was first attenuated and expanded to irradiate a circular mask. Then, the deposition of the polymer droplets onto the membranes was achieved by using a 15× objective lens to focus the resulting spatially homogenous laser beam onto the polymer-coated quartz plates (targets). The targets were prepared by spin coating the polymer solutions onto quartz plates, coated with a 40 nm Cr layer, to attain a uniform layer thickness. The use of the Cr intermediate layer prevents the direct exposure of the polymer solutions to the laser irradiation and thus ensures that the transferred polymer patterns maintain their delicate physical and chemical properties. The transfer was carried out in such a way that each droplet was deposited by a single pulse and the distance between the target and the substrate was about 100 μm. By varying laser parameters, such as laser fluence (from 200 to 400 mJ/cm$^2$) and laser beam diameter (from 10 to 40 μm) we achieved the deposition of circular polymer layers onto the sensitive membranes with diameters ranging between 20 and 250 μm. During the printing process, the target-substrate distance was kept to 100 μm and this contactless approach prevented the damage of the sensitive membranes. However, liquid phase printing is difficult to implement in the case of polymer solutions with highly volatile solvents and this fact limited the selection of the sensitive polymer materials for our study. Thus we selected to prepare targets by spin coating sensitive polymer layers diluted either in $H_2O$ or low volatility solvents, such as ethyl lactate and tetrahydrofuran (THF). Close-up photos of a reference membrane and membranes partially covered with PHEMA, PVA, and PVP polymer layers are depicted in Fig. 3. The polymer solutions and the solvents used were as follows: PHEMA solution in ethyl lactate, PAA in $H_2O$, P4VP in ethyl lactate or $H_2O$, PVP in $H_2O$, PVC-co-PVA in ethyl lactate, PHS in THF, PVA in $H_2O$ and PMMA in THF. In Fig. 4a, a partial view of a 16 × 16 sensor array is shown whereas spots of different polymers over the membranes of the same array can be identified in Fig. 4b.

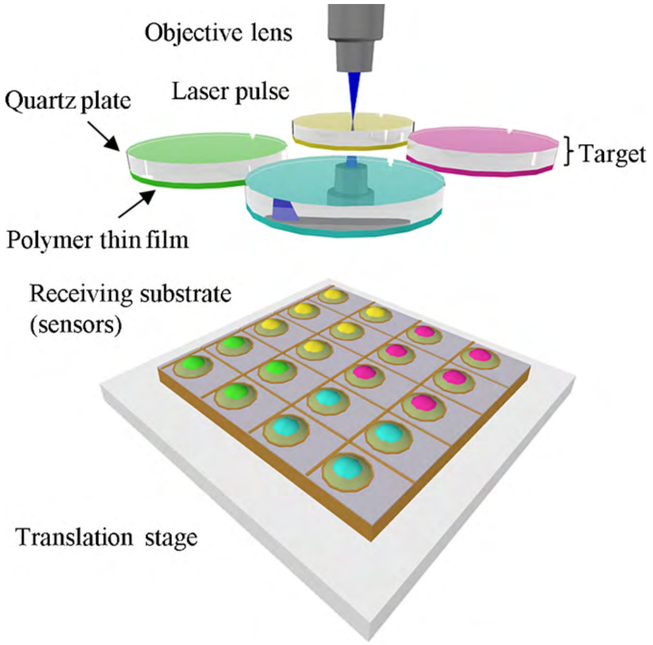

**Fig. 2.** Schematic illustration of the LIFT process.

### 2.3. Measurement setup

The measurement setup is illustrated in Fig. 5. The evaluation of the sensors' response was performed in a small volume (~7 cm$^3$) chamber where relative humidity and temperature are controlled to within 0.1% and 0.1 °C, respectively. Testing was carried out for controlled concentrations of water, methanol and ethanol vapours

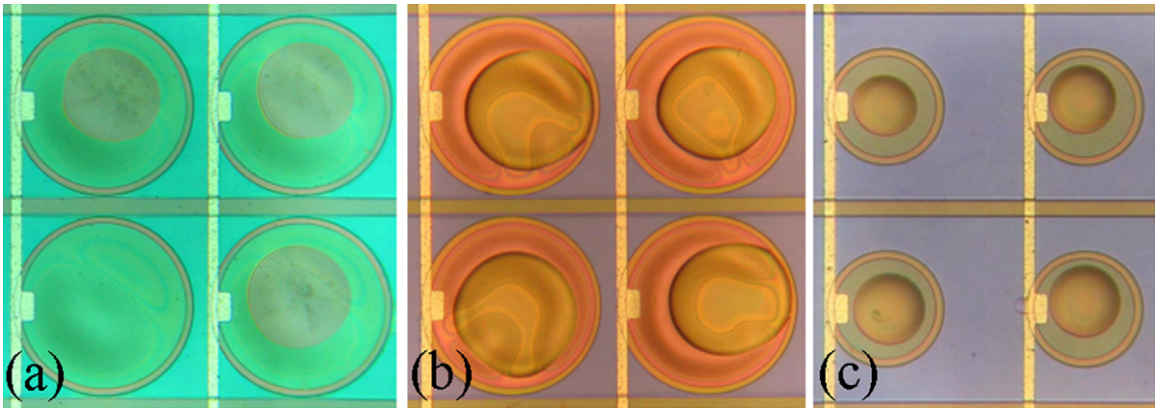

**Fig. 3.** Optical microscopy images of sensors partially covered with sensitive polymer layers by means of LIFT: (a) three membranes (250 μm diameter, 1.5 μm thick) partially covered with PHEMA and one reference membrane (b) membranes (250 μm diameter, 0.5 μm thick) partially covered with PVA (c) membranes (150 μm diameter, 1.5 μm thick) partially covered with PVP.

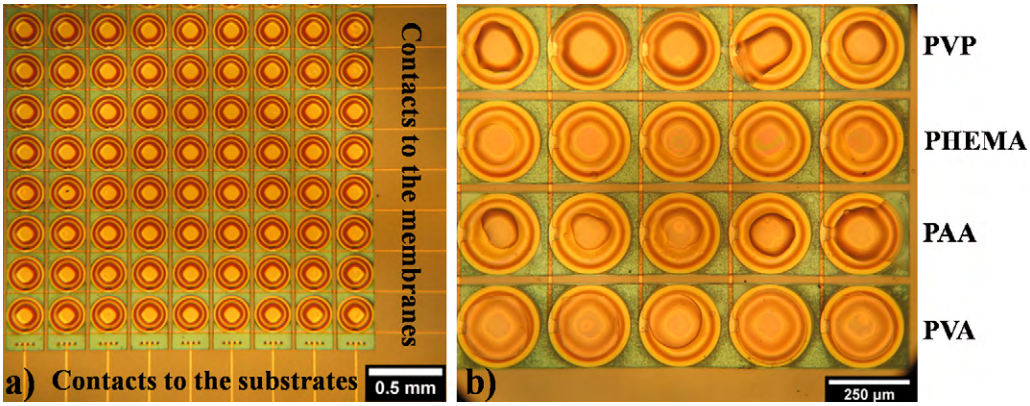

**Fig. 4.** Optical microscopy images of the sensor array. a) The Al contacts and b) the sensors covered with various sensitive polymer layers by means of LIFT can be identified.

in the 5000–35,000 ppm range. In the gas-delivering unit, initially a dry nitrogen flux was split in a carrier and a diluting part with the help of two mass flow controllers. The carrier part was then bubbled through the analyte of interest and subsequently mixed with the diluting part to achieve the desired concentration level in the chamber.

The sensor array was mounted onto a printed circuit board (PCB), which provides the necessary electrical connections to the outside world and is attached to the small volume chamber. In order to allow for the scanning of the whole array, the PCB edge is plugged in a standard connector on a 16 × 16 switch relay matrix. The concurrent measurement of multiple sensors was enabled through a Labview program running on a PC. The program controls the whole system, namely the switch matrix, the capacitance meter (HP 4278A) as well as the gas-delivering unit (mass flow controllers and valves).

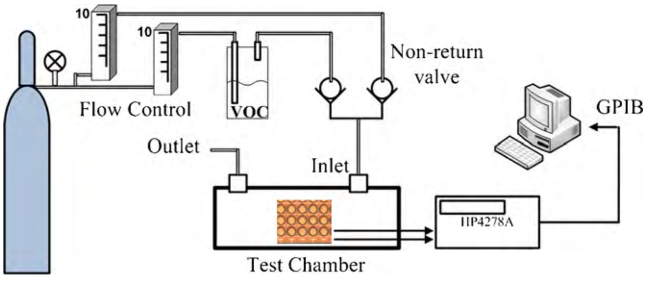

**Fig. 5.** Measurement experimental setup.

## 3. Results and discussion

For the evaluation of the sensor array a wafer with sensor membranes made of 0.5 μm thick SiGeB epitaxial layer was fabricated in three different diameters (150, 200 and 250 μm). All of the arrays were passivated by a 0.5 μm thick LTO layer. First, in order to evaluate the sensor response as a function of polymer coverage, a sensor array with 250 μm diameter membranes was used and its individual sensors were spotted with different diameters of 2% PHEMA spots using the LIFT technique. In Fig. 6, the response of one such PHEMA covered sensor to different concentrations of water, methanol and ethanol vapours is depicted.

In Fig. 7, the experimental response of the sensors when exposed to controlled concentrations (5000–20,000 ppm) of water vapours is plotted as a function of membrane coverage with PHEMA. In the same plot the results of a previous study [23], involving thicker (1.5 μm) SiGeB membranes is also plotted for comparison. The study was based on finite element analysis (FEA) and experimental results and revealed that partial coverage by 70% of flat or slightly deflected 1.5 μm thick membranes maximizes the sensitivity of the sensors. On the contrary, the thinner (0.5 μm) membranes of this study do not follow this trend. Rather the response of this second group of sensors maximizes at around 50%. This is attributed to the fact that although the thinner membranes yield more sensitive sensors, they are also more susceptible to the built-in stress induced during the fabrication process. This, in turn, forces the membranes to deflect asymmetrically, touches the bottom of the cavity (Fig. 4) and ultimately departs from the ideal case of flat membranes.

Following this initial evaluation, combinations of polymers between PHEMA, PAA, P4VP, PVP, PVC-co-PVA, PHS and PMMA,

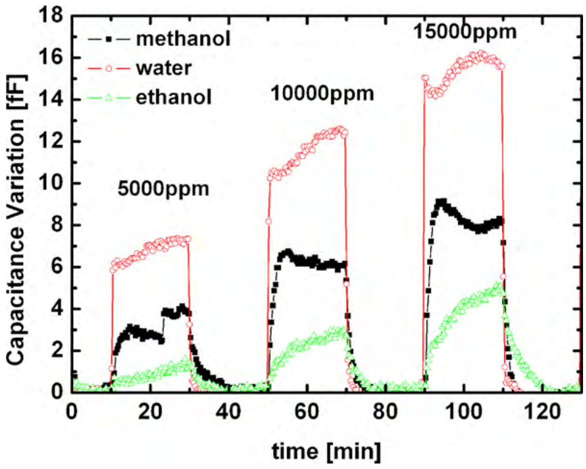

**Fig. 6.** The response in femto Farads (fF) of a PHEMA covered membrane of 250 μm diameter and 0.5 μm Si and LTO layer thickness, when exposed to various analytes.

were spotted on the surface of membranes (250 μm diameter, 0.5 μm thick SiGeB) in the same array. The verification of the optimum parameters for the polymer deposition included the laser parameters for each polymer. After polymer deposition, the arrays were tested by exposure to methanol, ethanol and water vapours. As an example, an array of four different polymers (PAA, PVP, PVC-co-PVA, P4VP) was made. In Fig. 8, the response of the array is depicted upon exposure to water vapours. Each sensor in the array responds in accordance with the polymer that has been printed on its Si membrane thus proving the suitability of laser printing of polymers in the fabrication of arrays of chemical sensors. In another example, a different set of polymers (PAA, PVP, PMMA and PHS) was deposited on sensor arrays with stiffer 200 μm diameter membranes. In this experiment, the thickness of the passivation layer was reduced to 0.2 μm by thinning down the covering LTO passivation layer using reactive ion etching. The response of these sensors to water and ethanol vapours is depicted in Figs. 9 and 10 respectively. As expected, reducing the LTO layer and thus the overall membrane thickness also increases the sensitivity (for example the PAA covered sensor exhibits 10 aF/ppm as compared to 0.7 aF/ppm for the 250 μm diameter with 0.5 μm thick LTO).

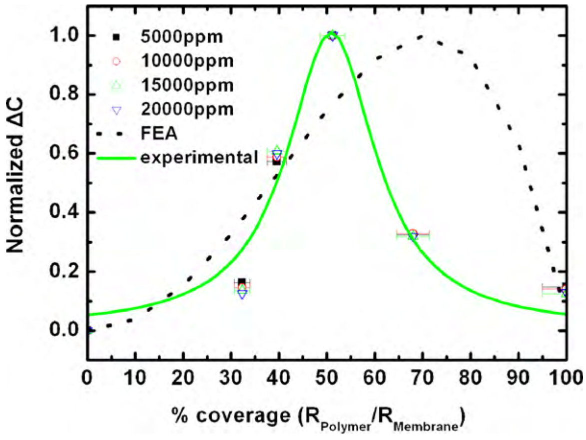

**Fig. 7.** Normalized experimental and simulation (finite element analysis, FEA) results of membranes (0.5 μm thickness and 250 μm diameter) partially covered with PHEMA. The simulation, and experimentally confirmed, results (dashed line) for flat membranes show that the sensor response is maximized when the ratio of the polymer over the membrane radius is about 70%. However experimentally, the maximum signal of the ultra-thin membranes is shifted to about 50% partial coverage. The experimental results correspond to 5000–20,000 ppm of water vapour.

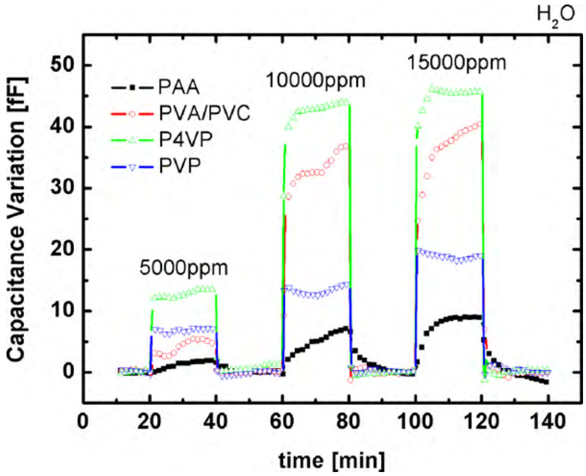

**Fig. 8.** The response of sensors with membranes of 250 μm diameter, and 0.5 μm Si and LTO layer, when exposed to humidity in concentrations 5000–15,000 ppm. The membranes are covered with four different polymers.

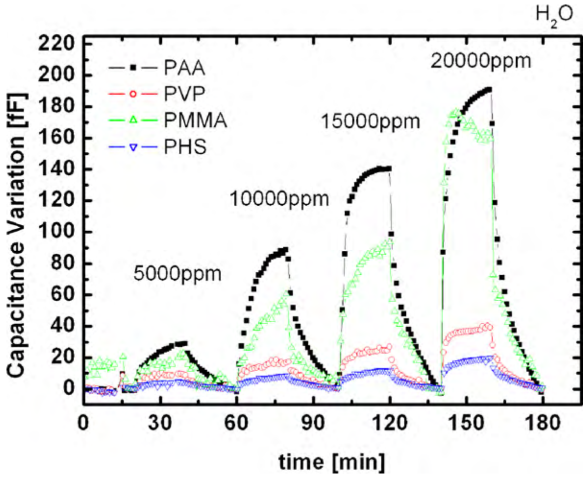

**Fig. 9.** The response of sensors with membrane diameter, 200 μm; Si thickness, 0.5 μm; and LTO thickness, 0.2 μm. The membranes are covered with four different polymers and are exposed to water vapour.

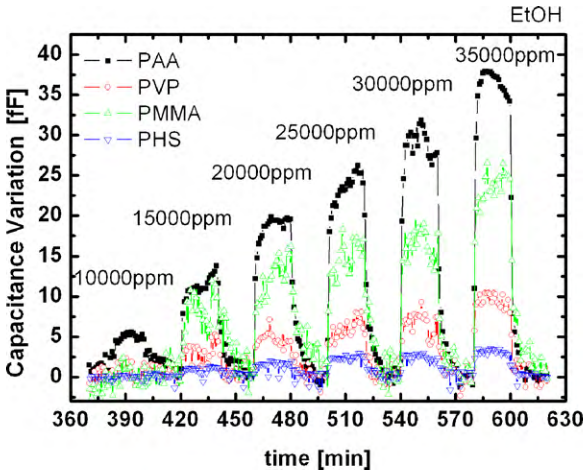

**Fig. 10.** The response of sensors with membrane diameter, 200 μm; Si thickness, 0.5 μm; and LTO thickness, 0.2 μm. The membranes are covered with four different polymers and are exposed to ethanol vapour.

Overall, in this work, liquid phase printing has been proven to be a useful tool for the printing of various polymer materials for the fabrication of chemical sensor arrays. However, the limitations imposed by the volatility of the polymer solvents constrain the range of polymer materials that may be used. In order to increase the selectivity, the sensitivity and the reproducibility of the array, future work will have to be directed towards dry laser printing. Dry laser printing is achieved by irradiating a dry polymer layer deposited on a quartz target and hence is applicable to a wide range of polymer materials, which may form uniform targets by spin coating, regardless of solvent properties. The technique is more controllable in terms of the size and the thickness of the deposited layer but requires close proximity conditions ($d < 1\,\mu$m) between the target and substrate that may be harmful for a delicate substrate, such as our sensor. However, the technique has been recently demonstrated for the fabrication of amperometric sensors [25] and we believe that the use of this dry laser printing method will provide us with a tool to investigate the sensitivity and selectivity limits of our sensor microarray.

## 4. Conclusions

A capacitive chemical sensor array with the ability to host up to 256 sensing sites and a polymer printing technique for the controlled deposition of the various sensitive layers have been demonstrated. Each chemical sensor in the array consists of a thin silicon/LTO membrane, which may be spotted with different polymers in a well defined way using LIFT. This laser printing technique is extremely suitable for the fabrication of micro-sensors, as the proper deposition of a specific polymer on a very confined area is the major concern for the development of miniaturized sensing devices. As a demonstration, a variety of polymers has been spotted on sensor arrays and tested during exposure to various concentrations of water, methanol and ethanol vapours. The deposited polymers showed good sensitivity depending on the analytes tested and the geometrical characteristics of the composite membranes.

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## Biographies

**V. Tsouti** was born in 1980. She received the BS degree in 2002 and MSc degree in Solid State Physics in 2005, both from the Department of Physics at the University of Athens, Greece. In 2009 she received the PhD degree from the Department of Applied Sciences of the National Technical University of Athens and the Institute of Microelectronics at NCSR "Demokritos". Her undergraduate and MSc thesis were focused on the electrical properties of SOI structures. Her PhD work included the development of chemical and biological micromechanical sensors. She is currently with the Institute of Microelectronics at NCSR "Demokritos" working on the development of silicon sensors (pressure, chemical and biosensors).

**C. Boutopoulos** was born in 1982. He received his BS in Applied Physics from the National Technical University of Athens in 2005 and his MSc in Microsystems and Nanodevices from the same University in 2008. His MSc thesis was focused on the development of protein microarrays via direct laser printing. He is now working at NTUA on his PhD thesis and his current research is focused on the combination of direct laser printing methods and sensing microsystems for the development of biosensors and chemical sensors.

**D. Goustouridis** was born in 1969. He received the BS in 1992 from the department of Physics of the University of Patras. In 2002 he received the PhD degree in microelectronics from the Department of Applied Sciences of the National Technical University of Athens for his work on capacitive-type pressure sensors. He is currently with the Institute of Microelectronics at NCSR "Demokritos". His interests include silicon micromachining, capacitive pressure sensors, chemical and biological sensing devices and measurements setup for sensors characterization.

**I. Zergioti** (Assistant Professor Physics, National Technical University of Athens) has more than 18 years experience in phenomena related to the interaction of electromagnetic radiation with matter and the development of laser materials processing for applications in nanotechnology and microdevices. During her PhD she worked at the University of California, Berkeley, and after her PhD she did research work at Max Planck Institute für Biophysikalische, and then at Philips CFT working on "Stereostiction of passive ceramic electronic 3-Dimensional structures using inkjet and laser sintering technologies". One of her main research achievements was the use of laser pulses to direct print biomolecules on biosensors and polymers on chemical sensors.

She was involved in many Research projects funded by the EC as a Project Coordinator and as Research team member, during the last 18 years. She has more than 90 publications in International peer reviewed journals and publications in conferences and she has contributed three book chapters on the laser printing technology.

**P. Normand** received his MS degree in microelectronics and microcomputing from Paris VII University in 1986, and his PhD degree in Physics of Semiconductors from the National Polytechnic Institute of Grenoble in 1992. Since then, he is with the Institute of Microelectronics of the National Center for Scientific Research “Demokritos” where he holds the position of Director of Research. He has been involved in several European projects with a focus on single-electron-, nanocrystal- and hybrid organic/silicon-memory devices as well as on the development of capacitive sensors for biomedical applications. He is author or co-author of over 100 publications in international journals, books and conference proceedings and 6 patents. His current research interests include the development of inorganic/organic memory devices, nanostructured materials for electronics, silicon nanotechnology and micromachining, as well as integrated sensors and MEMS.

**D. Tsoukalas** obtained the Electrical Engineering diploma from the National Technical University of Athens in 1979, the DEA degree in Electronics and Telecommunications in 1980, the Diploma of Docteur-Ingenieur in Electronics in 1983 and the Habilitation degree in 1994 all from the National Polytechnic Institute of Grenoble (INPG), France. From 1985 to 2002 he was a research scientist at NCSR “Demokritos”. In 2002 he joined the National Technical University of Athens where he is a professor. From 2009 he is Director of the Institute of Microelectronics, NCSR “Demokritos”. He was the Chairman of the Simulation Semiconductor Processes and Devices Conference in 2001 and of European Solid State Device Research Conference in 2009 both held in Athens. He has participated in numerous European projects in the area of Microsystems, nanoelectronic devices and silicon technology simulation. He is a Technical Committee member in several International Conferences in the above fields and he has authored or co-authored over 100 publications in International Journals.

**S. Chatzandroulis** received the Bachelor degree and the MSc in Electronic Automation from the Department of Physics at the University of Athens in 1990 and 1993 respectively. In 1999 he received the PhD degree from the same Department for his work on integrated silicon sensors. Since then he has been with the Institute of Microelectronics at NCSR “Demokritos” where he now holds the position of researcher. His interests include silicon micromachined physical sensors (pressure, strain), biosensors and chemical sensors, as well as electronic interfaces for sensors and sensor networking.