Ultra miniaturized InterDigitated electrodes platform for sensing applications


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Ultra miniaturized InterDigitated Electrodes platform for sensing applications


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Abstract

InterDigitated Electrodes (IDEs) is a generic platform for a wide range of diverse applications with their implementation in sensing modules being a major one. We propose the use of IDCs with deep sub-micron critical dimension; equally spaced electrodes of 200nm width for enhanced sensing performance and also the method of fabrication thereof. The transducer configuration was studied theoretically with a finite element method simulation by using COMSOL Multiphysics. The miniaturization of the IDEs up to 200nm critical dimension with an adequate sensing area for the deposition of the polymeric materials is considered beneficial in terms of sensitivity gain. The IDCs were designed to deliver capacitance values of few pF in order to be compatible with already developed miniaturized low-power readout electronics. The transducers fabrication is performed with conventional microelectronic/micromachining processing and then coated with several semi-selective polymeric films. Besides the fabrication of multiple sensor arrays (chips) on the same silicon wafer, the miniaturization offers the integration with the readout electronics on the same chip. The evaluation of the sensing performance of the semi-selective polymer coated sensors is performed upon exposure to vapours of pure and binary mixtures of VOCs and humidity in various concentrations. The sensors demonstrate high sensitivity to the examined analytes as a result of the miniaturization, while their semi-selectivity is a key for applications in complex vapour environment discrimination.
1. Introduction

The maturation of microelectronic devices and communication devices and protocols gave the boost for the Internet of Things (IoT). In this huge application domain, one of the most demanding building blocks are the sensors which should fulfill a wide range of diverse characteristics such as: ultra small size, long term operation, very low power requirements, very low cost and easy integration with other integration circuits. One very important application for such systems is the air quality continuous monitoring for indoor (including automotive cabin air quality) and outdoor environments. For example, the humidity sensor market is expected to double in the period 2018-2023 while the automotive temperature and humidity market is expected to overcome the $2B $ size soon. In the race for the design and implementation of sensors that fulfill these specifications many transducers and principles of operations have been proposed and implemented.

One very promising sensing concept is the chemocapacitor [1-4] where the change in the dielectric permittivity of the layer between the capacitor’s electrodes results in change in the capacitance value. The chemocapacitor is fabricated with mature and easy microelectronic processes and has a sensing layer between the two electrodes. This type of devices has the potential for monolithic integration with readout electronics, while their power consumption is limited to the measurement of the sensor response only. In the majority of applications of chemocapacitors, the sensing layer is made from a suitable polymeric material, while in some cases other materials, e.g. SnO₂ for the detection of ethylene [5] have been also explored. In addition, this type of devices has been also explored for biosensing applications, e.g. biofunctionalized planar capacitors for the measurement of the concentration of CRP biomarker [3] or for ABO blood group typing [6].

In the area of gas sensing applications chemocapacitors have already found wide application in the detection of humidity [7], Volatile Organic Compounds (VOCs) e.g. [8], toxic chemicals e.g. [9]. Due to the limited selectivity of polymeric materials against similar analytes, an array of chemocapacitors need to be employed, followed by appropriate signal processing for the identification of the analyte and its concentration. On the other hand, other chemical sensors, like the ones based on metal oxides, require power for their operation which makes them of limited usage in several applications, e.g. long term monitoring of analytes through battery-operated sensing systems. Thus, this bunch of unique characteristics, low-cost & large volume production by mainstream microelectronics processes and low power requirements, make them appropriate for a wide range of applications in our daily life with the most characteristic case to be the measurement of humidity which is the dominant device for this application.

There are several designs [10-14] that have been proposed and implemented for the realization of the related transducers. The transducers that have been mainly explored are: a) the Top-Down capacitor [10] where the capacitor’s electrodes are surfaces with the polymeric sensing film be in between the bottom and the top electrode while the top electrode is perforated to allow for fast interaction with the analyte molecules, b) Vertical Interdigitated Electrodes [11] where the electrodes are two arrays of posts and with the polymeric film covering the space between the electrodes, c) Planar InterDigitated Electrodes (IDES) where the polymeric sensing material covers the gap between the electrodes and its thickness is high enough to cover the electric field that extends over the dielectric substrate [12-13], d}
nanogap Interdigitated Electrodes where the nanosized electrode edges, contribute most to the capacitance of the system [2].

From all suggested designs, the planar InterDigitated Electrodes (IDEs) is the mostly studied transducer concept. It is implemented either on rigid substrates, e.g. [13-15] or on flexible ones [16] with critical dimension (electrode finger width and gap between the electrodes) to be 1μm or wider.

So far, very few applications of IDE transducers with critical dimension in the nanoscale are reported. Their applications are mainly for biomolecules detection and are currently limited by low-yield production and packaging processes [17-19], but to our knowledge they have not been employed as chemocapacitors. However, as it has been shown [20], the use of IDEs with smaller critical dimension, in the deep sub-micron regime, improves further the sensing performance of the device by a) smaller size which is extremely beneficial in the case of integration with the readout electronics on the same chip, saving precious Si real estate and b) using thin sensing polymeric layers, saving material and boosting the sensing speed since the analyte’s molecules reach equilibrium faster in the sensing zone of the polymer.

In the present work, we introduce the design and realization of IDE arrays with critical dimension of 200nm for higher sensing performance and for smaller footprint. After the deposition of selected polymeric layers the chemocapacitors are evaluated as sensors for humidity and certain volatile organic compounds.

2. Transducer design

A typical IDE layout is illustrated in Fig. 1a. The fundamental geometrical characteristics are the width of each finger (W), the gap between adjacent fingers (G) and the total sensing area (A). The capacitance of the IDE device is measured by applying voltage at the contact pad of the two electrodes. In the present work, the critical dimension, W and G, was selected to be 200nm to allow for significant reduction of the sensor footprint for the same capacitance value. For example, the capacitance of an IDE device made of fingers with W=G=200nm is five times higher than the capacitance of the IDE device made from fingers with W=G=1μm for the same area. Therefore, the transition from 1.0μm critical dimension to 200nm one allows for the integration of multiple IDE structures in a very small area increasing this way the integration level. In addition, such a small critical dimension requires also much thinner dielectric layers compared to the dielectric layers required when critical dimension is in the micron range and above.

The capacitance value of the IDE transducer was calculated by considering the actual layer stack and by employing the COMSOL Multiphysics (www.comsol.com) finite element simulation package. This simulation environment allows for detailed simulation of all design parameters providing useful information for the layout design as well as for the selection of the geometrical characteristics of the layers involved, e.g. minimum thickness of the dielectric layer, minimum thickness of the sensing layer. The sensing performance increases with increasing the sensing film thickness because more electric field lines pass through the sensing film, and saturates as the film thickness overcomes the value (W+G) [21, 22]. In fig. 1b the cross section of the electric field lines for the selected geometry is illustrated and
indeed the electric field lines do not surpass at heights higher than \(W+G=0.4\mu m\). Thus, for the selected geometry, sensing layer thickness of 0.5\(\mu m\) thickness is thick enough to ensure that the sensing performance is maximized. Such a thin sensing layer provides also with faster sensing response since the analyte molecules reach the equilibrium faster and can monitor concentration changes faster. In the simulation study, we have employed stationary electrostatics models with one electrode set to ground and the other set to 1V.

By employing this model, simulation of IDE structures at various geometries was carried out and for critical dimension in the \(W=G=0.2-1.0\mu m\) range. The simulation results are illustrated in fig. 2a for the same sensing area of 0.04mm\(^2\). In fig. 2b, the relative capacitance change, i.e. the increase in capacitance over the initial capacitance (at air) due to the increase of the dielectric constant of the sensing layer is shown. As it can be clearly seen the response’s slope increases with the decrease of the critical dimension of the IDE structure.

### 3. Transducer fabrication

Following the simulation study, the transducer was designed with 200nm critical dimension, i.e. 200nm IDEs spaced by 200nm gap. For the realization of these structures high resolution e-beam lithography was employed as patterning method. The process flowchart is illustrated in fig. 3. Initially a thick dielectric layer, is grown on the Si wafer. The thickness of the layer should be high enough to allow for the electric filed lines to pass through it and to ensure high Q values and lack of cross-talk between the IDE structures on the same chip. For the needs of the present work, 2.0\(\mu m\) thick SiO\(_2\) layers were either grown via thermal oxidation or deposited via PECVD. Both layers provided similar electrical performance and similar Q values in the examined frequency range. Then a PMMA bilayer was deposited on the Si wafer consisting of a thick 495K PMMA layer and followed by a thin 950K PMMA layer. The thicknesses of the individual PMMA layers were selected to provide the necessary undercut of the base PMMA layer for the next processing steps. The exposure was carried out by a JEOL-6300FS at 100KeV to ensure the high resolution patterning followed by the development step in MIBK:IPA 1:3 developer at 23\(^\circ\)C. After the lithographic step, deposition of Al layer followed via sputtering at a thickness \(~70nm\). In the lift-off step, in ultrasonic bath in acetone, the resist areas are dissolved and finally the IDEs structures are revealed on the SiO\(_2\) surface. In fig. 4a, a SEM image of characteristic IDE structures is illustrated revealing both the SiO\(_2\) surface and the aluminum electrodes. Each transducer offers a sensing area of 0.04mm\(^2\). The chips are then diced at size suitable for the DIL package. In fig. 4b, such a Si chip with 6 transducers is illustrated.

### 4. Sensor fabrication & evaluation

The efficiency of the fabrication process was evaluated with capacitance measurements of the uncoated IDEs. The IDEs were characterized in dry environment, i.e. upon exposure to inert gas (N\(_2\)), and their \(C_{air}\) values were found to be equal to the theoretically estimated values (\(C_{air}=2.4pF\)) with minimum standard deviation of \(\pm0.2pF\). The standard deviation is calculated over 16 sensor arrays comprising of 6
transducers in each one. The real resistance of IDEs, with DC excitation signal, was measured to be over 100Mohm and measurements with an impedance analyzer at 1MHz, showed Q values >30.

Then, the transducers were coated with different polymeric materials and the evaluation of the sensing performance of each polymer coated sensor of the sensor array was performed with measurements upon exposure to controlled concentrations of pure Volatile Organic Compounds (VOCs), humidity and selected binary mixtures.

In order to deal with the challenging task of monitoring the concentration levels of selected analytes- targets in complex vapour environment, a sensor array of semi-selective components is used [13, 23]. Each sensor of the array will respond to a certain group of chemical elements showing in most cases a broad and accordingly an overlapping response to the individual compounds. The response of a chemocapacitive sensor is mainly determined by two factors: (i) the sorption capacity of the polymer to analyze and (ii) the difference in dielectric constants between polymer and analyte. Therefore, for complex vapor environments the sensor array will produce a unique response pattern.

The polymers used for sensing materials in the present study are: poly(butylmethacrylate) PBMA, poly(hydroxyethylmethacrylate) PHEMA, polyisobutylene PIB, polydimethylsiloxane PDMS and a blend of PDMS with 30%w/w poly(ethylene oxide)/poly(propylene oxide)/poly(ethylene oxide) triblock copolymer commercially available as Poloxamer, PDMS-POE. The selection of the polymeric materials was initially based on solubility parameters used to identify the chemical affinity between the polymers and the analytes. For example, solubility parameters, \( \delta_t \), are calculated by group contribution method [24] to be 16.4, 18.8 and 24.1 (MPa)\(^{1/2}\) for PDMS, PBMA and PHEMA respectively and 47.8, 29.9 and 18.2 (MPa)\(^{1/2}\) for water, methanol and toluene respectively. In addition, based on our previous studies on the polymer sorption capacity to various VOCs and humidity by White Light Reflectance Spectroscopy (WLRS) method [25, 26], PHEMA is expected to be more sensitive to small volume polar analytes (i.e. methanol and humidity) than PBMA and PDMS which show the opposite behavior. All analytes were purchased from Sigma Aldrich and used as received. The analytes, in ascending dielectric constant value, that are used for the assessment of the performance of the IDE arrays are: n-Hexane, Toluene, Ethyl Acetate (EtOAc), Ethanol (EtOH), Methanol (MeOH) and humidity. All measurements were performed with the use of a fully automated, temperature stabilized set up, for the production of constant gaseous environments, by means of the bubbling technique, in continuous flow through the sensors. The capacitance measurements were performed by a previous developed electronics module, based on the commercial 24-Bit Capacitance-to-Digital Converter that allows for the measurement of capacitance changes of 1fF [8].

The evaluation of the polymer coated sensors (chemocapacitors) was performed with dynamic measurements under laboratory conditions. In particular, the sensors were purged with Nitrogen (N\(_2\)) to reach the baseline and then their response at various vapour concentrations of humidity and different VOCs was recorded in dynamic mode. The duration of each concentration step was selected to be long enough to allow for equilibrium. In the dynamic plot below, each step corresponds to a different analyte concentration. In addition to single analyte studies, the sensors were characterized in binary mixtures and in particular at high levels of humidity in the presence of low concentrations of different VOCs.

Typical dynamic measurements upon exposure to pure components and their mixtures are presented in fig. 5. The illustrated measurements were performed at 21°C, upon exposure to various vapour concentrations of: a) humidity in a wide concentration range (10-55% in terms of relative humidity), b) low concentration range of pure analytes of ethanol and methanol (1000-4000ppm) and c)
in binary mixture of high humidity level (55%) in the presence of the lowest examined concentrations of ethanol and methanol vapours (1000ppm for ethanol and 2000ppm for methanol respectively).

The sensors responses upon exposure to controlled concentration of analyte vapor are defined as 
\[ \delta C(\text{fF}) = C_{\text{eq}} - C_p, \]
where \( C_{\text{eq}} \) is the capacitance value in equilibrium with the analyte vapor of a certain concentration (expressed in ppm) and \( C_p \) is the capacitance of the polymer coated sensor at dry environment (exposure to \( N_2 \)). In fig. 6 the linear plots of sensors capacitance change upon exposure to various concentrations of: pure MeOH (a polar analyte) and pure EtOAc (a weakly polar analyte) are presented. For all polymer-analyte systems the sensitivity \( S \, (\text{fF/ppm}) \) is calculated by linear extrapolation of the \( \delta C(\text{fF}) \) vs. analyte concentration graphs and shown in Table 1. The corresponding limit of detection (LoD) is calculated by the \( 3\sigma/S \) ratio, where \( \sigma \) is the standard deviation of the blank capacitance value (noise). Since the blank capacitance value is 0.2-0.3 fF, the LoD for these sensors is their reciprocal sensitivity. Thus, an actual response of 1 fF of the sensor divided by the individual sensitivities \( S(\text{fF/ppm}) \) corresponds to the LoD in analyte concentration values. Additionally, the selectivity is determined by the ratio of sensitivities of one polymer for two different analytes or as the ratio of sensitivities of different polymer coated sensors for the same analyte.

PHEMA-coated sensor has the highest sensitivity to humidity and MeOH vapours due to its hydrophilic nature and the high dielectric constant of these analytes. PIB coated sensor, is almost inert to all analytes other than humidity, due to the generally highly impermeable nature of this polymer. The response to humidity is explained by the high dielectric constant of water rather than the polymer’s water sorption capacity. Comparing the PDMS-coated sensor and the PDMS-POL coated sensor, in all examined analytes, the latter shows higher responses, therefore higher sensitivity than the former.

For binary vapour mixtures, consisting of high humidity levels and low concentrations of different analytes, the equilibrium capacitance change for the majority of this group of polymer coated sensors is approximately equal to the sum of the capacitance changes that are induced by the pure components of the mixture: 
\[ \delta C(\text{mixture}) = \delta C(\text{component})_1 + \delta C(\text{component})_2. \]
As a result, in most cases, the presence of MeOH and EtOH is clearly detected in the presence of 55% RH, as shown in fig. 5.

As it is shown the sensitivities of the different polymer coated sensors to each analyte is often similar while the selectivity to each analyte differs, e.g. even though for PBMA and PDMS-POL-coated sensors the sensitivities to ethyl acetate and ethanol are almost equal, the PBMA is more selective to toluene vapours.

5. Conclusions

The simulation and fabrication of ultra miniaturized IDEs i.e. with electrodes in the deep submicron regime, and their implementation in polymer-coated capacitive sensors, is successfully demonstrated. So far, the nano-IDEs transducer surface has found limited application mainly for biosensing purposes.

The transducer configuration was studied theoretically with a finite element method simulation by using COMSOL Multiphysics. The miniaturization of the IDEs up to 200nm critical dimension with an
adequate sensing area for the deposition of the polymeric materials is considered beneficial in terms of sensitivity gain.

The transducers fabrication is performed with conventional microelectronic/micromachining processing and then coated with several semi-selective polymeric films. Besides the fabrication of multiple sensor arrays (chips) on the same silicon wafer, the miniaturization offers the integration with the readout electronics on the same chip.

The evaluation of the sensing performance of the semi-selective polymer coated sensors is performed upon exposure to vapours of pure and binary mixtures of VOCs and humidity in various concentrations. The sensors demonstrate high sensitivity to the examined analytes as a result of the miniaturization, while their semi-selectivity is a key for applications in complex vapour environment discrimination.

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References


Figures Captions:

Figure 1: a) Simplified top view of InterDigitated Electrodes (IDEs). b) Zoom-in image of electric potential profile around the electrodes obtained through simulation with the COMSOL platform. The device under simulation consists of electrodes with W=G=200μm, lateral dimensions of 30 * 45 μm² footprint and thickness of the substrate and of the overlayer of 5 μm. The layer stack consists of: (a) Si substrate (ε=11.8), b) >500nm thick SiO₂ (ε=3.9) as dielectric layer, (c) 60 nm thick Al as electrodes (ε= 10.0) and (d) polymeric or air overlayer (ε, in the 1.0–6.0 range).

Figure 2: Simulation data (a) Capacitance vs. dielectric constant of overlayer for IDEs with different critical dimension and (b) relative capacitance change vs. dielectric constant of overlayer.

Figure 3: Process flowchart for the realization of IDE arrays at wafer level scale.

Figure 4: a) Top-down SEM image of the IDE structures. Both the Al layer and the SiO₂ layer (deposited by PECVD in this case) beneath can be clearly seen. b) The Si transducer with the six IDE structures, imaged by a stereoscope. The pads of each transducer are close to the edge of the chip to facilitate for wire bonding with the DIL package.

Figure 5: Dynamic measurement of the different polymer coated sensors to various concentrations of pure analytes and selected binary mixtures of them, a) PBMA, PDMS, PIB, PDMS-POL and b) PHEMA coated sensor.

Figure 6: Equilibrium responses of different polymer coated sensors upon exposure to various concentrations of a) methanol and b) ethyl acetate vapours.
Tables Captions:

**Table 1:** Linear coefficients for the sensitivity (mean value) of each polymer-analyte system with the corresponding limit of detection (LoD).
Figure 1:

a) 

b)
Figure 2:

a) Capacitance (pF) vs. dielectric constant of overlayer ($\varepsilon_r$) for different $W (\mu m) / G (\mu m)$ ratios.

b) Relative Capacitance Change $(C-Co)/Co$, $Co=C$ vs. dielectric constant of overlayer ($\varepsilon_r$) for different $W (\mu m) / G (\mu m)$ ratios.
Figure 3:
Figure 4:

a)

b)
Figure 5:

a) 

Measurement at 21°C

Capacitance Change $\Delta C$ (fF) vs. Time (min)

- PBMA
- PDMS RTV 615
- PIB
- PDMS-POL

Relative Humidity (R.H.%): 20%, 30%, 40%, 50%

Steps: 1000 ppm, 2000 ppm, 3000 ppm, 4000 ppm

b) 

Measurement at 21°C

Capacitance Change $\Delta C$ (fF) vs. Time (min)

- PHEMA

Relative Humidity (R.H.%): 20%, 30%, 40%, 50%

Steps: 1000 ppm, 2000 ppm, 3000 ppm, 4000 ppm

N2
Figure 6:

a)

![Graph a)](image)

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Capacitance Change $\Delta C$ (fF) vs. MeOH (ppm)

b)

![Graph b)](image)

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Capacitance Change $\Delta C$ (fF) vs. EtOAc (ppm)
Table 1:

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Credit author statement
Declaration of interests

☒ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐ The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:
Declaration

☐ The authors declare that they don’t have any conflict of interest.
Highlights

- Simulation with a finite element method and microfabrication of IDE arrays in the deep sub-micron regime
- Chemocapacitors arrays for the detection of VOCs and humidity
- High sensitivity as a result of the miniaturization of the IDE critical dimension.