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Multi sensor platform on plastic foil for environmental monitoring

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Abstract

We report on multi-sensor platforms on plastic foils for environmental monitoring. Polymer-based capacitive sensors for humidity and volatile organic compounds (VOC)s, semiconducting metal oxides (MOX) based chemoresistive sensors for reducing/oxidizing gases and a Pt thermometer have been integrated together on a polyimide sheet and their performances characterized. The MOX gas sensors exhibited good sensitivity to CO and ethanol. The differential operation of the capacitive humidity sensors resulted in increased signals and reduced response/recovery times.

Keywords: Integrated gas sensors; Plastic foil; Environmental monitoring; Temperature sensor; Humidity sensor; Polyimide

1. Introduction

In order to meet the increasing demand for various gas sensors in mobile applications, a significant decrease in the production/exploitation costs and in energy consumption is required. In several cases (smart labels) the bendability of the devices are also important issues. The multi-sensor platform designed and manufactured by us attempt to make progress towards these goals.

Compared with the same kind of implementations reported before [1-3] the multi-sensor platforms presented here show significant advantages. They are smaller, have a higher degree of integration and are more economical. Bringing together, on the same flexible plastic substrate, gas sensors with different working principles, and providing temperature corrected responses we reached, on these platforms, roughly the same sensing potential as for the Si-based platforms/substrates. Thus, the detection of the reducing/oxidizing gases/vapors in low concentration ranges is ensured by two dedicated MOX (SnO₂:Pd) chemoresistors realized on low power hotplates while for the humidity and VOCs measurement, two capacitive interdigital structures, with almost no power consumption, have been preferred. To eliminate the substrate cross sensitivity towards humidity, which is a known drawback of the standard plastic materials used in electro-techniques and electronics (polyethylene naphthalate - PEN, polyimide - PI) the capacitive structures (CAP) have to be operated differentially [3]. Therefore, in the examples given below,

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only one capacitor, the sensing one, was covered with a sensing polymer film, while the other one was kept uncovered, as reference. However, the platform can be extended to include additional interdigital capacitors for other analytes. The accurate temperature values required in the data processing are delivered by the Pt thermometer of the device. All sensor signals can be acquired with high resolution analog/capacitive to digital converters. To the best of our knowledge the integration of these three different types of sensors on a flexible plastic foil was not reported in the literature yet. In following the platform performance is exemplified through CO/ethanol measurements with the MOX channels and ambient humidity with the CAP one.

Experimental

The configuration of the hybrid multi-sensor platform ($6 \times 6 \text{ mm}^2$) is presented in Fig. 1a. The device, which cross section is sketched in Fig. 1b, was realized on a $50 \mu\text{m}$ thick polyimide (PI) wafer (Upilex-50S from Ube Industries, Ltd) making use of a minimal number of conventional technological steps. The conducting elements of the sensors (thermoresistor, heaters, electrodes and pads) required only two levels of platinum (130 nm) with an adhesion layer of titanium (20 nm) deposited by DC sputtering.

The $100 \times 100 \mu\text{m}^2$ heaters of the MOX gas sensors are patterned with the first metallization by a lift-off technique. They have a nominal resistance of $\sim 150 \Omega$ at 0°C and need $\sim 18 \text{ mW}$ to reach an operation temperature of 300°C in a continuous heating mode of operation. Smaller devices would result in lower power consumption [4]. The electrical insulation from the top electrodes is made by a 700 nm thick photodefinable spin-coated polyimide film. The electrodes of the capacitive sensors and the resistance thermometer ($\sim 1 \text{ k}\Omega$) are produced on this additional PI film. Each interdigital capacitor covers an area of $1.4 \times 1.4 \text{ mm}^2$ and has $10 \mu\text{m}$ electrode width and spacing. The targeted nominal capacitance of about 10 pF allows for the direct readout of the humidity/VOCs induced responses with a commercial capacitance to digital converter (for example AD7746 from Analog Devices, Inc). After the fabrication, the transducers have been successively covered with gas sensitive materials by drop coating [5]: $\text{SnO}_2:2\%\text{Pd}$ (annealed in air at 400°C) for the MOX gas sensors, cellulose acetate butyrate (CAB) for humidity and polydimethylsiloxane (PDMS) for VOCs. The whole structure is flexible and has a total thickness of about $100 \mu\text{m}$.

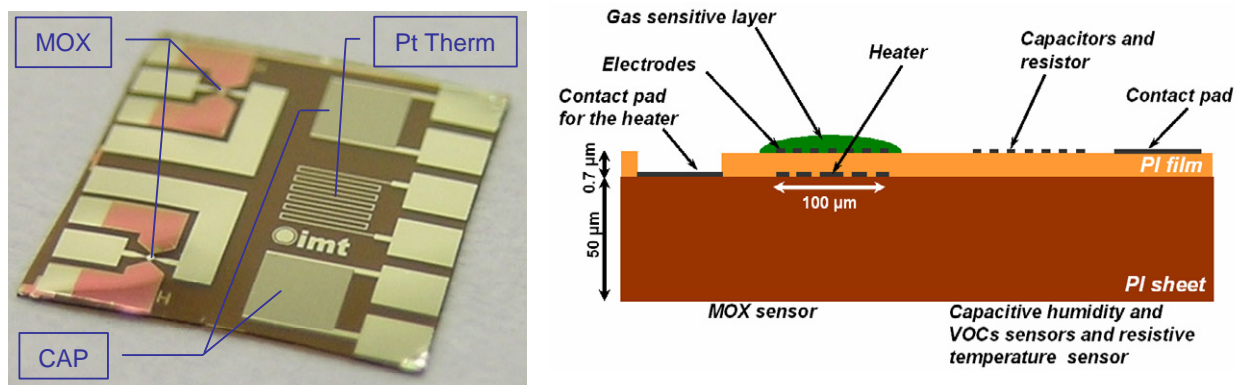


Fig. 1. Left: microscope photograph of the multi-sensor platform. Right: Schematic cross section of the sensor chip.

The platforms, packaged on printed circuit boards (PCB), have been tested and characterized in a computer controlled gas-mixing and measuring system. The MOX sensors have been exposed to CO and ethanol dosed at different concentrations in dry and humid synthetic air. Values in the TWA (time weighted average) range or below have been used. The exposure protocols are included on separated panels in the graphs displaying the sensor signals. The resistances of the MOX coatings, the capacitances of the measuring and reference capacitors and the Pt thermoresistor output have been acquired as raw data.

From these records the chemoresistive responses (CR) of the MOX sensors (the ratio of the resistances in the absence and presence of the target gas), the differential capacitance shift of the CAP and the ambient temperature

were evaluated. In an additional experimental step the heaters of the MOX sensors have been also calibrated. The Pt thermometer shows high linearity and a resistivity thermal coefficient of 2200 ppm/K.

2. Results and discussion

The main results are displayed in Fig. 2 and 3. One observes gas/humidity responses similar to those obtained with sensors produced by conventional technologies on ceramic/silicon substrates. Fig. 2 additionally shows the positive effect of the CAP differential operation leading to an increased sensor immunity to the substrate cross sensitivity towards detected analytes. In the same time, fast and almost linear responses of the CAPs have been obtained (for the investigated humidity levels). The sensitivity of the MOX to ethanol is good (CR ~ 20 for 20 ppm). In the case of CO a lower sensitivity was obtained (CR ~ 2 at 80 ppm). For both analytes a power low dependence of the CR on the concentration was observed. The humidity effect on the MOX sensors is high for the 0 – 10% r.h. interval (a factor of ~ 2.5) while in the whole 10 – 70% r.h. range, of interest in applications, it is less important (approximately 20%).

The reproducibility of the sensor responses was tested together with the sensor reliability during few weeks of uninterrupted operation for several platforms. No depreciation occurred for the CAP sensors, where the signals were statistically spread in the limit of the experimental errors only. The MOX sensors required a short stabilization period (few runs of 10 h following the protocols in Fig. 2, 3) and than showed a relatively stable operation. After many weeks of continuous exploitation a weak degradation occurred. The first, not optimized, sensor worked acceptably (a signal decrease by a factor less than 2) more than one month. For sake of space the corresponding results are not displayed but the trends in the sensor behavior can be observed on the calibration curves included in Fig. 4. The measurements with the PDMS coated devices reproduced the results previously obtained on simpler platforms [3] and are not presented here.

The simultaneous measurement of reducing/oxidizing gases and VOCs can be problematic under given circumstances. Increased ethanol concentrations, but still in the TWA range (~ 500 ppm), saturate the MOX channels of the platform, while well detectable by the differential CAPs. This issue is not specific to integrated sensors, being encountered for all type of MOX sensors with high sensitivity. The multi-sensor platform, however, has an important advantage. An intelligent driving electronics can switch off the MOX heaters and validate only the CAP outputs, preventing, in this way, inaccurate records and reducing the power consumption.

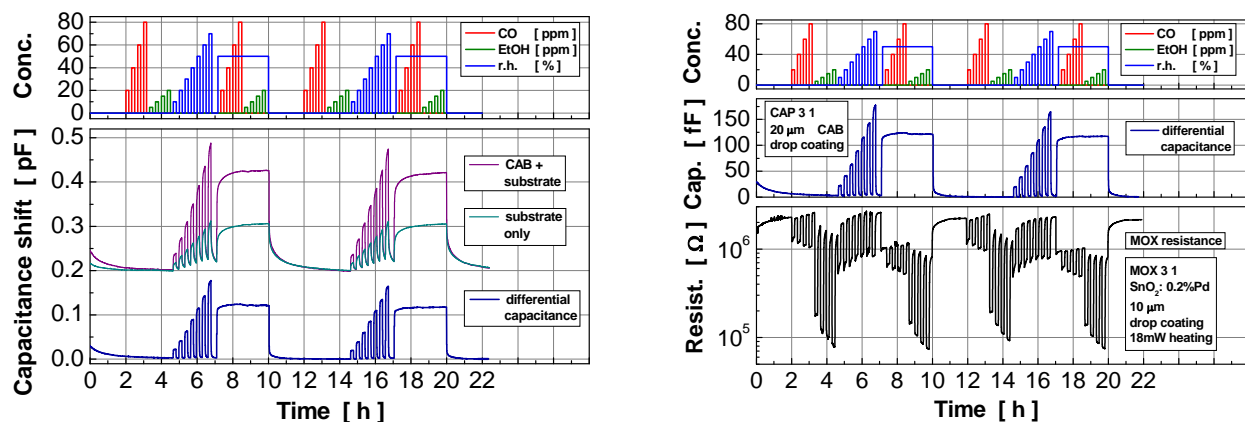


Fig. 2. Multi-sensor platform responses towards CO (20,40,60 and 80 ppm), ethanol (5,10,15,20 ppm) and humidity (10,20,30,40,50,60,70 % r.h.) in synthetic air at 200 sccm. In the left graph is sketched the evaluation of the differential response provided by the capacitive structures: from the signal of the CAB coated capacitor (CAB+substrate curve) is subtracted the parasitic contribution of the substrate response to humidity/VOCs, that is the response of the uncoated capacitor (substrate only curve) The procedure allows the detection of the measured analytes with higher accuracy and with reduced response and recovery times. In the right graph: the response of a MOX sensor together with the differential response of the CAP-sensor.

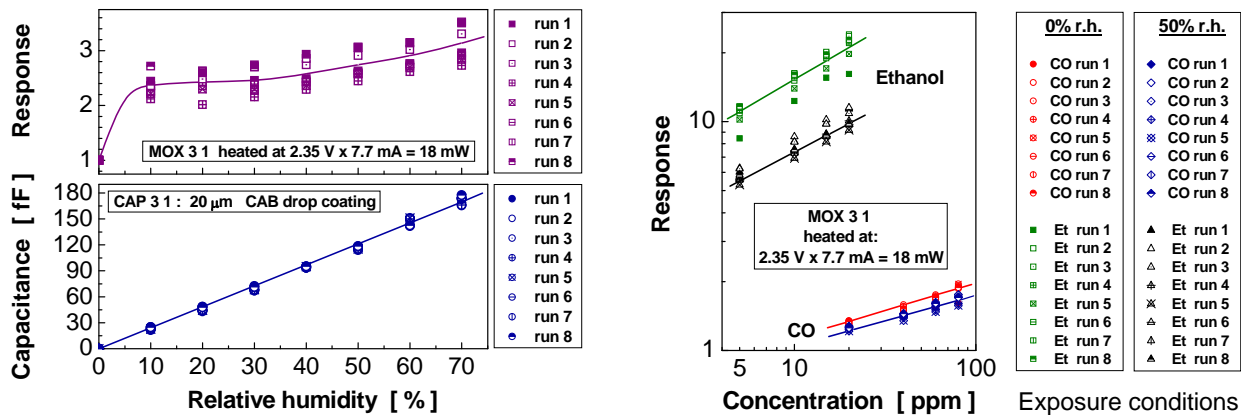


Fig. 3. Left: MOX (upper panel) and CAP (lower panel) calibration curves towards humidity. Right: Multi-sensor platform calibration curves (MOX channels) evaluated from the response partially presented in Fig. 2.

3. Conclusion

We realized, on a thin polyimide foil, a multi-sensor platform for the monitoring of environmental parameters. It contains a resistive Pt thermometer, two MOX sensors and two capacitive interdigital structures to be used as a differential humidity or VOCs sensor. The evaluation of the humidity with the differentially operated CAP sensors is precise and reproducible with reduced response/recovery times and parasitic effects from the substrate. The heating power of the MOX hotplates is less than 18 mW under standard exploitation conditions (continuous heating and reading). Through appropriate power and read-out management a platform average consumption below the milliWatt is easily achievable. The first generation of devices was devised to prove the sensor concept and can evolve towards hybrid sensor arrays with an increased number of elements. Moreover, by the use of plastic substrate we foresee the opportunity to produce such devices at large scale in a near future based on printed fabrication processes. Due to their reduced power dissipation and potentially reduced price, the environmental multi-sensors platforms could be widely used in wireless sensor networks and mobile/autonomous systems, such as RFID tags.

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References

1. D. Briand, S. Colin, A. Gangadharaiah, E. Vela, P. Dubois, L. Thiery, N.F. de Rooij. Micro-hotplates on polyimide for sensors and actuators. *Sensors and Actuators A* 2006;**132**:317-324.
2. D. Briand, S. Colin, J. Courbat, S. Raible, J. Kappler, N.F. de Rooij. Integration of MOX gas sensors on polyimide hotplates. *Sensors and Actuators B* 2008;**130**:430-435.
3. A. Oprea, J. Courbat, N. Bârsan, D. Briand, N.F. de Rooij, U. Weimar. Temperature, humidity and gas sensors integrated on plastic foil for low power applications. *Sensors and Actuators B*, 2009;**140**:227-232.
4. J. Courbat, D. Briand, L. Yue, S. Raible, N.F. de Rooij. Ultra-low power metal oxide gas sensor on plastic foil. in *Digest Tech. Papers Transducers 2009 conference*, Denver, USA, June 21-25, 2009, pp. 584-587.
5. D. Briand, A. Krauss, B. van der Schoot, U. Weimar, N. Barsan, W. Göpel, N. F. de Rooij. Design and fabrication of high-temperature micro-hotplates for drop-coated gas sensors. *Sensors and Actuators B* 2000;**68**:223-233.