



**HAL**  
open science

## **Inkjet printed SnO<sub>2</sub> gas sensor on plastic substrate**

Mathilde Rieu, El Hadji Malik Camara, Guy Tournier, Jean-Paul Viricelle,  
Christophe Pijolat, Nicolaas F de Rooij, Danick Briand

► **To cite this version:**

Mathilde Rieu, El Hadji Malik Camara, Guy Tournier, Jean-Paul Viricelle, Christophe Pijolat, et al..  
Inkjet printed SnO<sub>2</sub> gas sensor on plastic substrate. Eurosensors 2015, Sep 2015, Freiburg, Germany.  
pp.75-78, 10.1016/j.proeng.2015.08.569 . hal-01234940

**HAL Id: hal-01234940**

**<https://hal.science/hal-01234940v1>**

Submitted on 15 Dec 2015

**HAL** is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

# Inkjet printed SnO<sub>2</sub> gas sensor on plastic substrate

M. Rieu<sup>(a)\*</sup>, M. Camara<sup>(b)</sup>, G. Tournier<sup>(a)</sup>, J.P. Viricelle<sup>(a)</sup>, C. Pijolat<sup>(a)</sup>, N.F. de Rooij<sup>(b)</sup>, D. Briand<sup>(b)</sup>

<sup>(a)</sup> Ecole Nationale Supérieure des Mines de Saint-Etienne, SPIN-EMSE, PRESSIC Department, CNRS: UMR5307, LGF, 42023 Saint-Etienne, France

<sup>(b)</sup> Ecole Polytechnique Fédérale de Lausanne (EPFL), Institute of Microengineering (IMT), Sensors, Actuators and Microsystems Laboratory (SAMPLAB), Rue de la Maladière 71b, 2000 Neuchâtel, Switzerland

\*Corresponding author : rieu@emse.fr

## Abstract

We report on the development of a metal oxide (MOx) sensor prepared by inkjet printing technology onto polyimide foil. Gold electrodes and a gold heater were printed on each side of the substrate, respectively. SnO<sub>2</sub> based ink was developed by sol-gel method and printed onto the electrodes. A final annealing at 400°C compatible with the polymeric transducers allows to synthesize the SnO<sub>2</sub> film. Electrical measurements were carried out to characterize the response of fully printed sensor under different gases. The device was operated at a temperature between 200 and 300°C using the integrated heater. The sensor exhibited responses to carbon monoxide and nitrogen dioxide, under dry and wet air.

## Keywords

SnO<sub>2</sub> gas sensor; Sol-gel synthesis; Inkjet printing; Polyimide foil

## 1. Introduction

Development of sensing devices onto flexible foils is receiving special attention with the advances made in printed electronics [1-3]. However material synthesis and processes compatible with the low temperature induced by the use of flexible substrates are necessary. All the previous works related to preparation of metal-oxide gas sensors onto plastic substrate involve the use of photolithography process to shape metallic layers [2, 3]. Here, inkjet printing method was used to deposit all the functional layers of the sensor, as it is a low cost and flexible process to prepare digitally multilayer structures and sensor arrays.

In this work, we report on the preparation and the characterization of fully inkjet printed SnO<sub>2</sub> sensors with integrated heater onto polyimide (PI) film. For this, an ink was developed to synthesize SnO<sub>2</sub> at relatively low temperature and directly onto the PI foil. Gold electrodes and a heater were implemented in order to get an autonomous device. Finally, the gas sensing properties were evaluated.

## 2. Experimental

### 2.1. Inkjet printing

Flexible foil was used as substrate. Polyimide (PI) Upilex-50S 50 µm-thick has been chosen because it is a heat-resistant material and therefore keeps its physical and chemical properties under high-temperature conditions (up to 500°C).

Commercial gold ink (NPG-J from Harima) was ink-jetted onto the dry PI substrate. The printer used was a Dimatix DMP-2800 with 10 pL cartridges. Drops presented a diameter of around 150µm. A drop to drop spacing of 100 µm was chosen to obtain a uniform gold layer. The deposition was done 3 times in order to get a conductive layer. After printing, the coatings were placed in an oven at 250°C for 3 hours to cure and sinter the ink. Gold ink was used to print parallel electrodes on one side of the PI foil and the heater on the other side (figure 1). Then, tin ethoxide was prepared by sol-gel method [4] and the developed tin oxide based ink was printed between the two gold electrodes and sintered in air at 400°C-1h.

### 2.2. Characterization techniques

Thermal analyses of the xerogel, thermo-gravimetric analysis (TGA) and differential scanning calorimetry (DSC), were performed with a coupled TGA/DSC StareSystem from Mettler Toledo. Analyses were performed from room temperature to 800°C under 5 L h<sup>-1</sup> air flow.

Electrical characterization of the sensor was performed using a gas mixing system. The heater at the backside allowed to perform measurements at different operating temperatures. Several concentrations of CO and NO<sub>2</sub> were injected either in dry air or wet air (1% absolute humidity) as carrier gas.

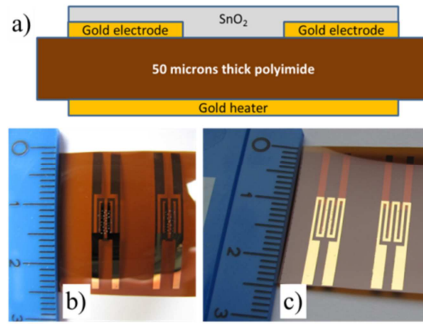


Figure 1: a) Cross section schematic view of the device, b) top side optical image of the sensor: electrodes and SnO<sub>2</sub>, and c) back side optical image: heater.

### 3. Results and discussion

#### 3.1. Characterization of SnO<sub>2</sub> precursor

Before using the solution in an inkjet printing process, the sol was hydrolyzed to obtain a gel in order to characterize it. The gel was then dried at 80°C for 24h. The decomposition in air of the xerogel was followed up by thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). Weight loss and heat flow are reported in function of the temperature in Figure 2.

TGA curve shows, with increasing temperature, a continuing loss of mass of the reaction products. Around 400°C, the total loss of mass constitutes 50 weight % of the xerogel mass. The shape of the DSC curve indicates a sharp exothermic process (T = 375°C) that occurs simultaneously with mass loss, which is associated with SnO<sub>2</sub> crystallization [5]. The sintering temperature was thus fixed at 400°C.

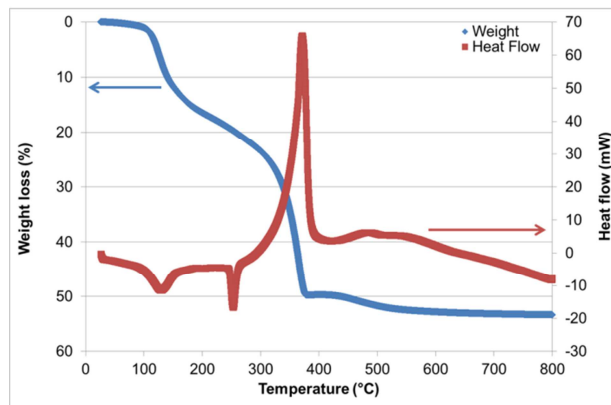


Figure 2: Thermo-gravimetric analysis (weight loss) and differential scanning calorimetry (heat flow) of SnO<sub>2</sub> sol-gel.

#### 3.2. Sensor characterization

The evolution of the conductance of the sensor at 300°C and 200°C, in dry air as carrier gas, is reported in Figure 3.

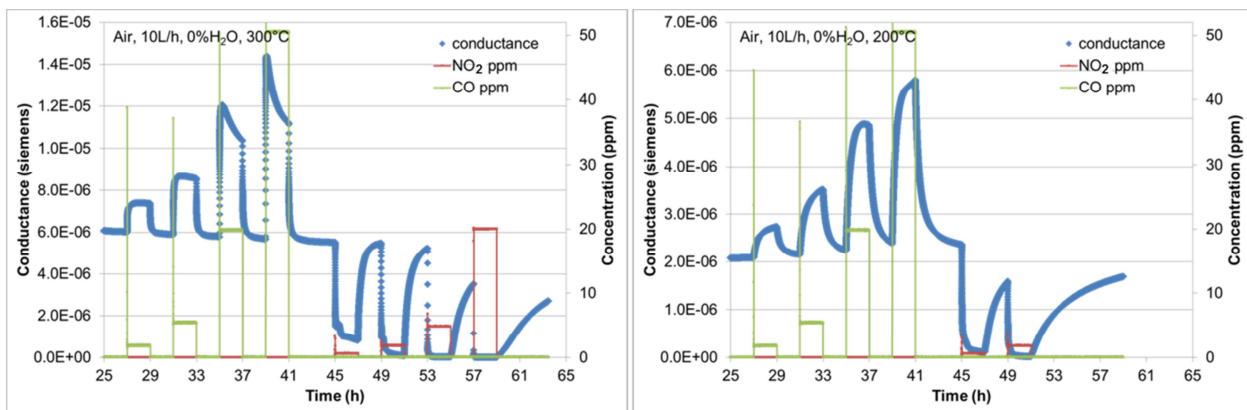


Figure 3: Evolution of the sensor conductance in dry air with CO and NO<sub>2</sub> injections at an operating temperature of 300°C (left) and 200°C (right).

It is evidenced that the pure SnO<sub>2</sub> sensor responds to the gases even at low concentrations: 1 ppm CO and 0.6 ppm NO<sub>2</sub>. Reaction kinetics are slower at 200°C compared to 300°C, that implies the response time and recovery time being lower at this temperature.

Chemoresistive gas responses ( $R_{\text{air}}/R_{\text{CO}}$  or  $R_{\text{NO}_2}/R_{\text{air}}$ ) of the sensor under dry and wet air were also calculated. Responses were smaller under wet air (1% absolute humidity), nearly divided by 2. However, they were still significant: indeed, responses to CO and NO<sub>2</sub> in the investigated temperature range were comparable to result reported in literature for SnO<sub>2</sub> sensors [2, 6].

#### 4. Conclusions

The reported technology allows simple metal-oxide sensors preparation with results comparable to usual SnO<sub>2</sub> sensors. We have demonstrated the first fully inkjet printed MOx sensor onto polymeric foil, including the gas sensitive layer and heating transducer. We are now working on the optimization of the heater design and on the synthesis of low-temperature operating MOx films to lower the sensor power consumption.

#### Acknowledgements

This work was supported by the LABEX MANUTECH-SISE (ANR-10-LABX-0075) of University of Lyon, within the program "Investissements d'Avenir" (ANR-11-IDEX-0007) operated by the French National Research Agency (ANR).

#### References

- [1] D. Briand, A. Oprea, J. Courbat, N. Bârsan, Making environmental sensors on plastic foil, *Mater Today*. 14 (2011) 416-423.
- [2] J. Courbat, D. Briand, L. Yue, S. Raible, N.F. de Rooij, Drop-coated metal-oxide gas sensor on polyimide foil with reduced power consumption for wireless applications, *Sensor Actuat B-Chem.* 161 (2012) 862-868.
- [3] S. Vallejos, I. Gràcia, E. Figueras, J. Sánchez, R. Mas, O. Beldarrain, C. Cané, Microfabrication of flexible gas sensing devices based on nanostructured semiconducting metal oxides. *Sens. Actuat A-Phys.* 219 (2014) 88-93.
- [4] S. Sujatha Lekshmy, K. Joy, SnO<sub>2</sub> thin films doped indium prepared by the sol-gel method: structure, electrical and photoluminescence properties, *J. Sol Gel Sci. Techn.* 67 (2013) 29-38.
- [5] L.F. Chepik, E.P. Troshina, T.S. Mashchenko, D.P. Romanov, A.I. Maksimov, O.F. Lutskaya, Crystallization of SnO<sub>2</sub> Produced by Sol-Gel Technique from Salts of Tin in Different Oxidation States, *Russian Journal of Applied Chem.* 74 (2001) 1617-1620.
- [6] S.H Hahn, N Bârsan, U Weimar, S.G Ejakov, J.H Visser, R.E Soltis, CO sensing with SnO<sub>2</sub> thick film sensors: role of oxygen and water vapor, *Thin Solid Films.* 436 (2003) 17-24.