

Comparison between MOX sensors for low VOCs concentrations with interfering gases

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Abstract—N-type Metal Oxide (MOX) sensors was developed to detect Volatile Organic Compounds (VOCs) at low concentration level. Sensitive layer like SnO₂, ZnO and WO₃ was deposited by reactive RF sputtering method. The sensors is based on a micro heater and a MOX sensitive layer on a silicon substrate. Gas sensing properties have been investigated toward isobutylene, as a typical VOC. The optimum working temperature was experimentally determined at 285°C for isobutylene. This work highlights the detection of VOC with interfering gas by MOX sensor at low level. This sensor will be used for a real time indoor air monitoring.

Keywords: gas sensor, MOX sensor, isobutylene, carbon dioxyde, carbone monoxyde.

I. INTRODUCTION

Some MOX sensors are investigated in order to detect VOCs thanks to their miniaturization and real-time monitoring capabilities. In this work, we have chosen three main n-type metal oxide used in this field: SnO₂ [1], WO₃ [2], and ZnO [3]. The aim of the comparison between the performances of these sensitive materials is to find the best sensitive layer for our low VOCs concentrations sensors and to evaluate the effect of interfering gases like CO and CO₂. In the Section 2, we will describe the sensitive layers and the sensor and in the Section 3 we will highlight the main results of this work.

II. MATERIAL AND METHODS

A. Sensitive layer

The three sensitive thin layers (~50nm) under study are deposited by reactive magnetron RF sputtering and annealed at 450°C during 1h30 to improve their Nano-crystallization and the stability of the sensors response. Fig.1 shows the XRD patterns of the three thin film. All the diffraction peaks show tetragonal rutile structure for SnO₂, monoclinic structure for WO₃ and hexagonal structure for ZnO (JCPDS cards No 72-1147, 41-1445, 36-1451 respectively).

Under isobutylene, the reducing molecules will react with the adsorbed oxygen ions and release the trapped electrons back to the metal oxide conduction band. This reaction leads to the decrease of electron depletion barrier and to increase the electrical conduction of the metal oxide.

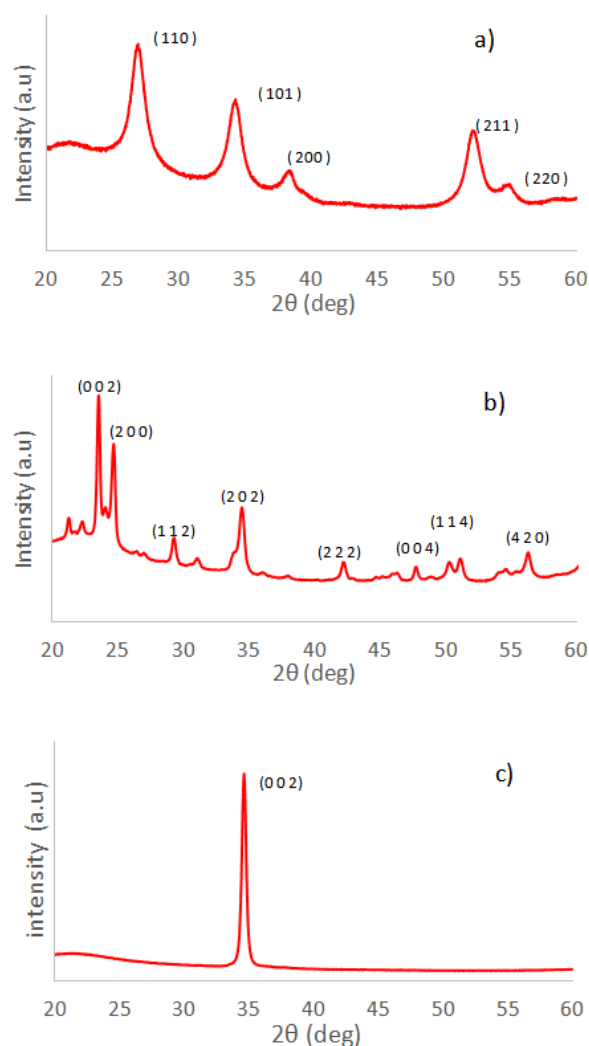


Figure 1. XRD patterns of a) SnO₂, b)WO₃ and c) ZnO made by reactive RF sputtering

B. Chip gas sensor

The gas sensors fabricated with SnO₂, WO₃ and ZnO layers as sensitive material is presented in Fig.2.

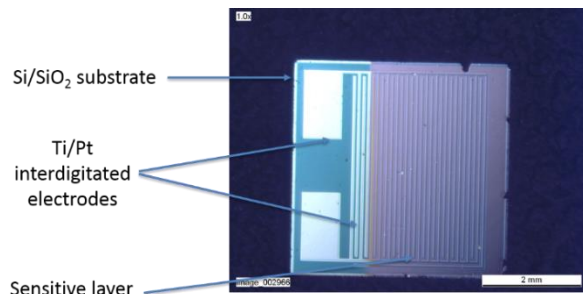


Figure 2. MOX Sensor with Si/SiO₂ substrate

This device has been tested with an automated gas bench with isobutylene. We used a power supply to control the operating temperature and a source meter for the data acquisition. This target gas was injected into a dilution system with or without interfering compounds. The outline was connected to a thermo-regulated test chamber. For each concentration, the sensor was exposed to isobutylene for 1 min then to dry air during 10 min. The sensors were maintained at the nominal heating voltage in dry air until the baseline was obtained to reach the response [4] under a flow rate of 500 sccm.

III. RESULTS AND DISCUSSION

A. Response to isobutylene

Fig.3 shows a typical responses with a wide range of detection from 50 ppb to 500 ppb of isobutylene. The best working temperature has been determined at 285°C.

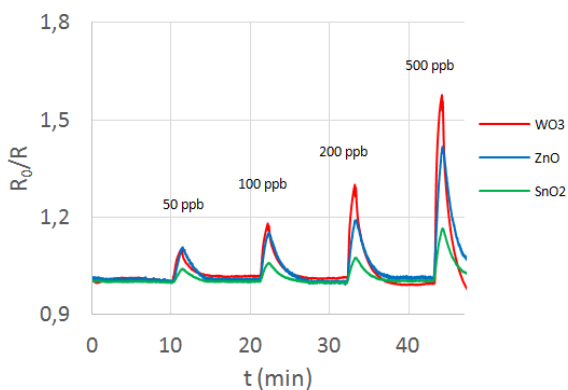


Figure 3. Sensors response for isobutylene concentrations, from 50 ppb to 500 ppb.

WO₃ and ZnO sensors seem to be the best devices for isobutylene. We have reached the highest responses from the three sensitive layer with low concentrations.

B. Influence of interfering gases

We have chosen 10 ppm of CO and 1% CO₂ as interfering gases concentrations. The Fig.4 shows the comparison of the responses of the three materials and for the two gases.

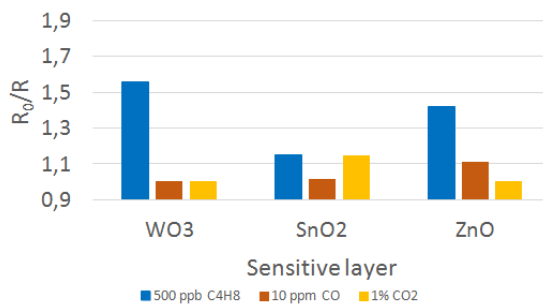


Figure 4. Sensor response under isobutylene and interfering gases

WO₃ and SnO₂ show low responses towards CO and CO₂ despite the better response for the target gas.

With tests under the same experimental conditions we can classify the right metal oxide for a target gas like isobutylene in presence of interfering gases.

IV. CONCLUSION

The gas measurement showed fast response / recovery times towards isobutylene. The best sensitive layers are WO₃ and ZnO because we have the highest responses for isobutylene and the weakest influence towards gases like CO and CO₂. This is the first step for air gas monitoring. We want to improve the selectivity towards others VOCs like benzene and toluene. On the other hand and after identifying the appropriate sensitive materials, we plan to study the improvement of the selectivity of these sensors.

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REFERENCES

- [1] Z. Tang , G. Jiang, P. C. H. Chan, J. K. O. Sin, S. S. Lau , Theory and experiments on r.f. sputtered tin oxide thin-films for gas sensing applications, Sensors and Actuators B vol. 43, pp. 161-164, 1997.
- [2] M. Bendahan, R. Boulmani, J.L. Seguin, K. Aguir , Characterization of ozone sensors based on WO₃ reactively sputtered films: influence of O₂ concentration in the sputtering gas and working temperature, Sensors and Actuators B, Chemical vol. 100, pp. 320-324, 2004.
- [3] V. Senay et al., ZnO thin film synthesis by reactive radio frequency magnetron sputtering, Applied Surface Science vol. 318, pp. 2-5, 2014.
- [4] C. Wang, L. Yin, L. Zang, D. Xiang, and R. Gao, Metal oxide gas sensors: Sensitivity and influencing factors, Sensors, vol. 10, pp. 2088-2106, 2010