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Sub-ppm NO₂ sensing in temperature cycled mode with Ga doped ZnO thin films deposited by RF sputtering

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1. Abstract

In this work Ga doped ZnO thin films have been deposited by RF magnetron sputtering onto a silicon micro-hotplate and their structural, microstructural and gas sensing properties have been studied. ZnO:Ga thin film with a thickness of 90 nm has been deposited onto a silicon based micro-hotplates without any photolithography process thanks to a low cost and reliable stencil mask process. Sub-ppm sensing (500 ppb) of NO₂ gas at low temperature (50 °C) has been obtained with promising responses R/R₀ up to 18.

2. Results

Micro-hotplates have been prepared using photolithographic process. The system is composed by a heating element and sensing electrodes. They are both integrated in membrane in order to have a localized heating and sensing spot onto which the sensitive thin film is deposited. The microhotplates can operate with low consumption and can heat up to 500 °C with a good stability. This system has been already published in [1]. The use of lift-of process to restrict the deposition of the thin film onto central electrodes can lead to the dissolution and/or contamination of the sensitive layer. That's why the photolithographic method was avoided and a stencil mask process was used (Figure 1).

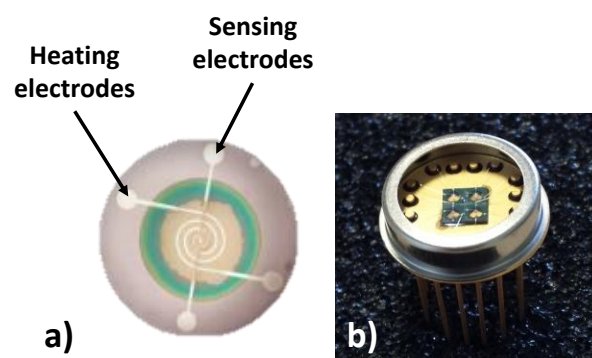


Figure 1: a) top-view of the membrane; b) mounted micro-sensor.

[1] L. Presmanes, Y. Thimont, I. El Younsi, A. Chapelle, F. Blanc, C. Talhi, C. Bonningue, A. Barnabé, Ph. Menini, and Ph. Tailhades, "Integration of P-CuO Thin Sputtered Layers onto Microsensor Platforms for Gas Sensing", *Sensors*, vol. 17, #1409, June 2017.

The deposition conditions are shown in the Table I.

Table I: *Deposition parameters of ZnO:Ga thin film by RF-sputtering.*

| | |
|------------------------------|---------|
| Target material | ZnO:Ga |
| Power (W) | (4 %at) |
| Magnetron | 30 |
| Argon pressure P (Pa) | Yes |
| Target to substrate distance | 2 |
| d (cm) | 7 |

The measurement protocol used in the test bench is a cycle of heating and cooling steps from 5 mW to 35 mW with a step of 5 mW for 5 min which correspond approximately to 50 °C to 350 °C. The tests were performed with 50% relative humidity. Alternation of air and air with 500 ppb of NO₂ has been applied. In presence of air, the resistance is very low, close to 300 Ω, due the high conductivity of ZnO:Ga. When 500 ppb of NO₂ are injected, the resistance increases strongly up to 7 kΩ at 50 °C. The ratio R/R₀ (where R is the resistance under NO₂ and R₀ the resistance under air) has been calculated using the last points at each temperature step (Figure 2).

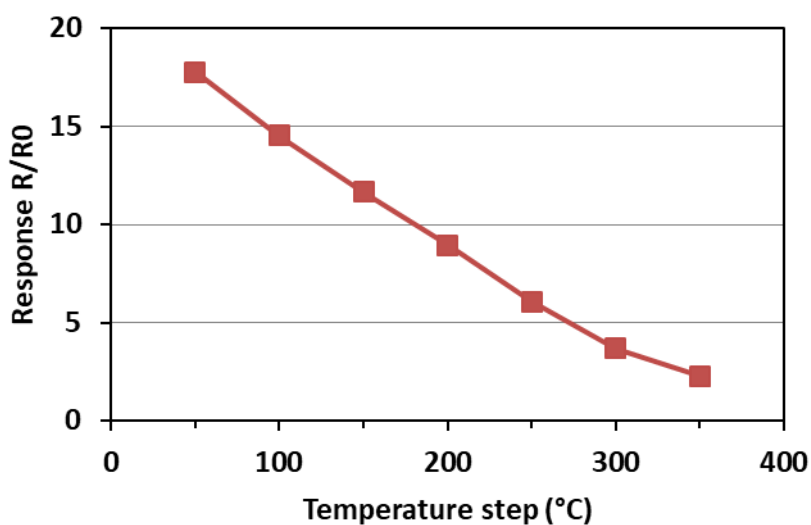


Figure 2: *Response of ZnO:Ga vs temperature step (in cycled temperature mode).*

Unlike the results we obtained in isothermal mode, the response in cycled temperature mode is much higher close to room temperature. Promising results with a response up to 18 for 500 ppb of NO₂ at 50 °C (R/R₀ ~ 36 /ppm) have been highlighted.

3. Acknowledgement

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