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Integration by shadow mask process of ZnO:Ga sputtered layers onto microsensor platform for detection of sub-ppm acetaldehyde concentration

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Summary

Micro-hotplates have been first prepared by using silicon microtechnologies onto a thin membrane, it was then possible to generate very fast temperature variations (from room temperature to 550 °C in 25ms) and a rapid temperature cycled mode could be applied. A low cost and reliable stencil mask method has been developed and successfully tested during the integration of ZnO:Ga layer. The variation of the resistance of this 50 nm thick sensitive layer has been measured under 500 ppb of acetaldehyde (C₂H₄O). The very high response obtained was between 10 and 1000 depending on the working temperature.

Motivation and results

Microsensors made by MEMS technologies have many advantages as for example, high performance, small size, low cost and low power consumption. Radio-frequency sputtering is compatible with these technologies and has many other advantages like the possibility to obtain very thin films with nanometric scale grain sizes and controlling very easily the inter-granular porosity by varying the deposition parameters [1]. Such films with controlled nanostructure are great of interest as sensitive layers and can be integrated in gas sensing devices. In this work, we show the interest of using fully compatible micromachining technologies to elaborate microheaters and deposit the sensitive layers to obtain sensors at the micron scale. Recent studies have highlighted the potential of pure [2] or doped [3] zinc oxide for the detection of acetaldehyde which is considered as air pollutant and carcinogen agent. However, for now there is no result about the detection of this gas by Ga doped ZnO sensitive layer. It is the reason why n-type ZnO:Ga has been chosen in this study first to demonstrate the integration feasibility and secondly to present its high level of sensing performances under acetaldehyde.

Optimized microhotplates (4-chip sensor) have been elaborated by standard photolithographic processes (Figure 1). They can work at high temperature and low power consumption (500 °C ; ~ 55 mW) with a very good stability and reproducibility. A circular design has been used to improve thermo-mechanical behavior. This technology is highly interesting to prepare multi-sensors (by integrating 4 different layers) which are especially suitable to operate in complex atmosphere containing various interfering gases and obtain a good selectivity. Structural characterizations of ZnO:Ga have been carried out on 100 nm thick films deposited on glass substrates. X-Ray diffraction confirmed that zinc oxide is crystallized with a single growth orientation along [001] direction (Figure 2). The average grain size determined by AFM on the 50nm thick film annealed at 400°C was 28 nm (Figure 3). After the integration of the ZnO:Ga layer into the microsensor by shadow mask method (Figure 1), it has been tested under sub-ppm concentration (500 ppb) of acetaldehyde. Five level of power have been applied to the heater to explore the sensing performances from 10 to 45 mW (50 to 400°C). This approach revealed a huge sensitivity between 10 and 1000, depending on the working pulsed-temperature level (Figure 4).

Word count: 486

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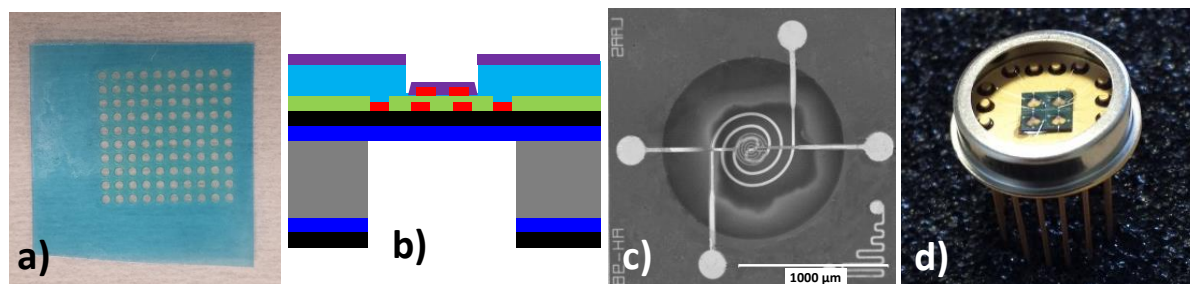


Figure 1: a) shadow mask made by cutting laser machine b) cross sectional schematic view, c) ZnO:Ga layer deposited onto the electrodes area after the removal of the stencil mask, d) multi-sensor (4 chips) packaged on a TO-9 support

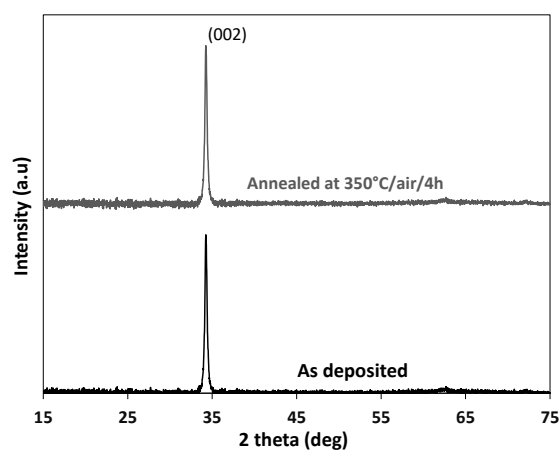


Figure 1: XRD pattern of a 100nm ZnO:Ga film deposited on glass, before and after annealing under air at 350 °C.

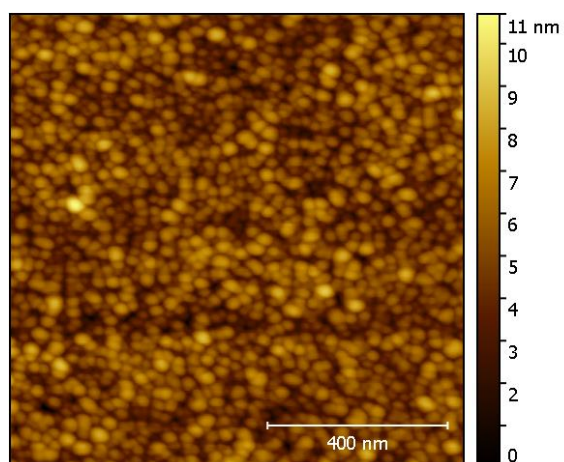


Figure 2: AFM image of a 50nm ZnO:Ga thick film annealed at 350°C for 1h under air atmosphere.

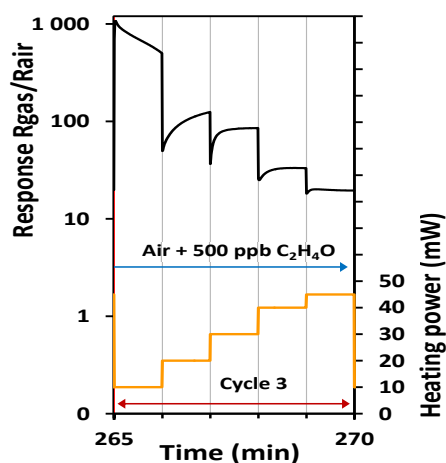


Figure 4: Response of the sensor (R_{gas}/R_{air}) under 500 ppb of acetaldehyde in temperature cycled mode.