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Flexible Gas Sensors Fabricated by Ultrasonic Spray Deposition

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Abstract — Sensors are changing the way people live and considerable efforts are under way for their integration into daily devices. Recently, much attention has been given to sensor fields for flexible and printed electronics. Among all the processes, ultrasonic spray deposition is a simple and precise technique giving enormous potential for applications with low cost production, low waste of material and thin film uniformity. In this work, we are focusing on Zinc Oxide nanoparticles deposited by ultrasonic spray on a flexible substrate as sensitive layer for air quality monitoring. The flexible platform consists of Ti/Pt interdigitated electrodes for gas detection and a micro-heater device, both on Kapton HN substrate. A brief description of the process steps will be provided. Gas sensing properties have been investigated and the flexible sensors present repeatable responses toward ammonia (NH₃) and ozone (O₃) with fast responses and recovery times in a wide range of gas concentrations. The optimum working temperatures were experimentally determined at 300 °C for NH₃ and 200 C° for O₃. Overall, this work highlights the ultrasonic spray potential for fast processing flexible gas sensors based on zinc oxide solution.

Keywords-Flexible gas sensor; ultrasonic spray deposition; ozone sensor; ammonia sensor; kapton substrate; ZnO ink.

I. INTRODUCTION

The market for printed, flexible and organic electronics is growing exponentially with huge potential and new applications in our daily life. New sensors are emerging in various applications, and the fabrication techniques could be key to reach some benefits such as improved performance, flexibility. transparency, reliability, and better environmental credentials. Ultrasonic spray deposition is a good candidate to fabricate thin film at a lower cost in a large area with low material waste avoiding expensive chemical solutions and high temperatures, which are not suitable with flexible substrates [1][2]. Moreover, "ultrasonic technology nozzle-less" delivers a thinner and more precise coating application than conventional spray nozzles, film coaters, roll coaters, and jetting technology.

In this work, ZnO nanoparticle films were deposited by ultrasonic spray nozzle–less on DuPontTM Kapton HN polyimide substrate using ink solution from Genes'Ink

company (reference ZnO5F12). The interdigitated electrodes and the micro-heater device were fabricated using photolithography [3]. In Section II, the sensor process will be described and the results will be discussed in Section III.

II. DESCRIPTION OF APPROACH AND TECHNIQUES

Our flexible gas sensor consists of a 75µm thin Kapton polyimide film, with Ti/Pt interdigitated electrodes for gas detection, and a Ti/Pt micro-heater device allowing an efficient control of temperature, useful for detection of gases. Kapton polyimide film presents the advantage of working up to 400 °C with an excellent thermal stability. Furthermore, it is solvent resistant and flexible. The metal electrodes Ti/Pt were deposited by magnetron sputtering with thicknesses of 5 nm and 100 nm, respectively. In this work, ZnO nanoparticles were used as sensitive material with a thickness of 100 nm deposited by ultrasonic spray. It is a simple technique ideal for the thin and uniform application of several materials with low viscosity. Furthermore, it ensures very little waste of coating material. The deposition was done with a programmable nozzle-less spray machine Prism BT Benchtop X-Y-Z Coating System from Ultrasonic Systems, Inc. (USI manufacturer) that gives thin film thickness homogeneity with an error around ± 4 % on A4 sheet of Kapton HN. The operational principle consists in the product being applied into a rectangular tip through a liquid applicator. The spraying takes place by the vibration of a titanium transducer and due to the rectangular tip, the coating pattern is rectilinear, flat and uniform [4]. The ZnO films obtained were annealed for 3 hours at 300°C to improve their quality and stability. The gas measurements were carried out in a closed chamber by measuring the resistance through the sensitive material at different temperatures from 25°C to 350°C under a target exposure of 1 minute in order to find the best operating conditions. We used a power supply to control the operating temperature and a source meter Keithley 6430 for the data acquisition.

III. RESULTS AND DISCUSSION

A. Flexible gas sensors

The gas sensor fabricated with ZnO nanoparticles as sensitive material and deposited by ultrasonic spray technique is presented in Figure 1.





B. Response to Ammonia and Ozone

Figure 2 shows good sample responses and a wide range of detection from 10 ppm to 100 ppm for ammonia. The best working temperature has been determined at 300°C.



Figure 2. Sensor responses deposited by ultrasonic spray under NH₃ at 300 °C.

The response time is defined as the time taken by a sensor to achieve 90% of the total signal change when the sensor is exposed to a target gas [5][6][7]. Similarly, the recovery time is defined as the time it takes to reach 90% of the difference between the equilibrium resistance under target gas and the sensor resistance in dry air after the gas injection was stopped (i.e. in dry air with no gas) [8]. We observed good and fast responses, with recovery times of less than 2 minutes, and a good detection range for ammonia. Responses to ozone during the same exposure time have also been registered and the best working temperature has been found at 200°C. Figure 3 presents the response at 200°C under ozone from 5ppm to 300ppb. This exposure time did not allow reaching the response peak saturation as in the case of ammonia. However, for an exposure time of 1 minute the responses are always fast for all concentrations as well as the time to come back to the baseline.



Figure 3. Sensor responses deposited by ultrasonic spray under O3 at 200 °C.

This coating technique presents important benefits such as good thin film distribution and good control of the deposit parameters.

IV. CONCLUSION

The gas measurements in our experiments showed good responses with fast response / recovery times towards ammonia (at 300 °C) and ozone (at 200 °C) in a wide range of gas concentrations. The obtained gas sensing properties on Kapton HN substrate and the use of ultrasonic spray deposition highlight the promising opportunity in the flexible electronic field to fabricate quality devices with fast and low cost production.

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