*Proc. of the International Conference on Electrical, Computer, Communications and Mechatronics Engineering (ICECCME)*
*7-8 October 2021, Mauritius*

2021 International Conference on Electrical, Computer, Communications and Mechatronics Engineering (ICECCME) | 978-1-6654-1262-9/21/$31.00 ©2021 IEEE | DOI: 10.1109/ICECCME52200.2021.9590945

# Microwave-based Nitrogen Dioxide Gas Sensor For Automotive Applications

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***Abstract*— As a consequence of global industrialization and the increased use of cars powered by gasoline, the level of environmental pollution (mostly by nitrogen dioxide) is increased year by year. There are a number of studies, where ambient nitrogen dioxide concentrations were measured, and currently, in many cities, smart air pollution sensors are installed. However, the problem of monitoring the nitrogen dioxide concentration on highways has not been solved. Herein, the preliminary results on the microwave-based $NO_2$ gas sensor for such the application are presented.**

***Keywords—microwave gas sensors, nitrogen dioxide detection, automotive application, gas-sensing measurements, metal oxides***

## I. INTRODUCTION

The need for monitoring environmental pollution increases every year due to the increased air pollution all over the world, including nitrogen dioxide ($NO_2$) monitoring [1]. $NO_2$ is a well-known pollutant with a harmful effect on human health, as well as, various other organisms. Aptly to the outlines delivered by the WHO (World Health Organization) the exposition to $NO_2$ concentration above 200 µg/m$^3$ may cause inflammation in the respiratory tract [2]. Epidemiological research has provided that lengthened inhalation by children with asthma to $NO_2$ worsen the effects of bronchitis [3]. From the negative effect of nitrogen dioxide on human being, several standards have been developed, such as Euro 6 where the emission of $NO_2$ was limited to 0.08 g/km and the mass fraction of $NO_2$ in $NO_x$ emissions should not exceed 10 [4].

Up to date, many types of gas-sensing systems have been developed for nitrogen dioxide detection, such as electrochemical systems [5-6], catalytic sensors [7-8], optical systems [9-10], as well as, sensors based on the semiconductors MOX (metal oxides) as a gas-sensitive layer [11-12].

Various metal oxides have been proposed for nitrogen dioxide detection, including Zn oxide (ZnO) [13], Sn dioxide ($SnO_2$) [14], W trioxide ($WO_3$) [15], In oxide ($In_2O_3$) [16], Ti dioxide ($TiO_2$) [17], and Cu oxide (CuO) [18-20]. Generally, the MOX-based gas sensors operate in the DC mode. In addition, higher temperatures are required such as 300-500$^o$C. Recently, some papers have suggested that nitrogen dioxide detection could be obtained at room temperature, however, the long-term stability effect is poor [21].

The sensors that exhibit good long-term stability and work at low temperatures, including room temperature are microwave-based gas detectors. They are commonly fabricated by using the interdigital capacitor topology, where metal oxide gas-sensitive layer is deposited with the utilization of various methods in the 'active' part of the gas sensor. Recently, organic-based gas-sensitive materials such as phthalocyanines have been tested for gas-sensing application in the microwave frequency range [22]. On the other hand, the metal oxides have been tested at the microwave frequency range as well [23].

In this study, microwave-based gas detectors operated in a wide range are proposed. The gas-sensitive layer was fabricated with the utilization of magnetron sputtering technology, i.e. copper oxide deposition under argon-oxygen atmosphere. The gas sensor substrate was realized as a snake co-planar transmission circuit. The reflection coefficient measurements over a freq. range of 1.5 GHz - 4.5 GHz have been conducted. The nitrogen dioxide from 2.5 to 5 ppm and various relative humidity concentrations were used to simulate various environmental conditions that may occur. The results have been obtained during the international project entitled “Highly Accurate and Autonomous Programmable Platform for Providing Air Pollution Data Services to Drivers and the Public”.

## II. Methods and Materials

### A. *Microwave circutis*

The gas sensor is build of four key elements: sensor substrate with the electrode, gas-sensitive layer, package, and front-end electronics. Within this study, the developed gas detectors operate in the microwave range thus the microwave circuit as a gas sensor substrate was designed. Briefly, a coplanar waveguide (CPW) was designed, simulated, and fabricated on a 60-mil thick laminate (RO4003C, Rogers). The transmission line width of 1.28 mm was used, which results in a 50 Ohm (characteristic) impedance of the line. To enable the increased sensor's sensitivity, the transmission line was designed in a form of a meander with the arm's portion and turn radios of 3 mm and 1.28 mm, respectively. More details are presented in reference [24].

### B. *Gas-sensitve layer*

The gas-sensitive layers were deposited in the ultra-high vacuum systems from Kurt J. Lesker equipped with a multi-magnetron system and glancing angle deposition manipulator. The deposition was conducted under the reactive mode, i.e. 70%argon-30%oxygen mixture, controlled by mass flow controllers (GF40, Brooks). The high purity (99.99%) metallic target (i.e. copper, Cu) was used. The DC-MF power supply with 50W was applied. The thickness of the samples was controlled by adjusting the deposition time and verified by off-line measurement with the utilization of a mechanical profilometer. The material properties of the deposited layers have been studied and previously reported [25].

### C. *Gas-sensing setup*

The gas-sensing measurement setup is equipped with four mass flow controllers (MFC) GF40 (Brooks). The MFCs are 100 sccm full-scale models with 0.5 +/- accuracy of the flow measurement. The gas-dosing system consists of four gas lines including synthetic air and three lines with nitrogen dioxide: 1 ppm, 5 ppm, and 10 ppm; target gas is balanced with synthetic air as well to avoid the influences from the ballast. The gas chamber was optimized to achieve a renewal rate of approximately 1 minute, and the measurements were done at least three times to check the repeatability. All measurements were taken at room temperature (23ºC, 30%RH) stabilized by the AC (air-conditioning) system installed in the lab. Fig. 1presents the sketch of the measurement setup, where mass flow controllers, Dreschel bottle, and pipelines to DUT (device under test) are illustrated.

The following measurement protocol was set: at the beginning the sensors were stabilized in pure synthetic air for two hours, to remove any contamination during the installation as well as to stabilize the atmosphere inside the measurement chamber. Then, gas was introduced to the measurement chamber as presented in Fig. 2. and Fig. 3. During the gas-dosing procedure, some fluctuation in the gas-dosing can be observed which results in slightly signal changes. However, the fluctuations are not critical and can be neglected at this point.

## III. Results Analysis

The proposed sensors were verified in terms of possible application in the automotive application for monitoring environmental pollution. The microwave-based measurement system is planned to be installed in the trucks that drive on the highways in Europe. Therefore, the data collected within the system enables the monitoring of the current nitrogen dioxide levels in many places. The main part of the system is the gas-sensing element that will enable $NO_2$ detection in the concentration range present normally, as well as, at various changes of ambient conditions such as humidity and temperature.

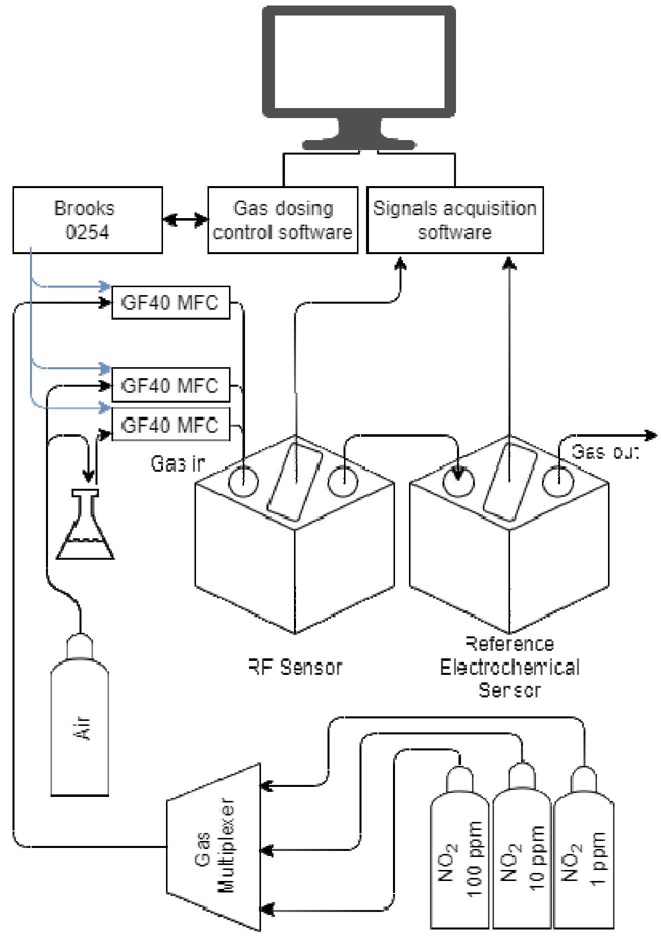


Fig. 1. A sketch of the gas-dosing system

In the case of long-term stability and the possibility to work in the wide range of ambient condition changes, the gas sensors based on the p-type semiconductor gas-sensitive layers have been proven as a good candidate. Within this study, the gas sensor responses defined as normalized phase slope changes have been measured at specific conditions, i.e. the ambient conditions were stabilized (23ºC, 50%RH), while relative humidity level in the gas stream was changed – 25%, 50%, and 75%. As can be observed in Fig. 2 (the gas-sensitive layer with 200 nm thickness), and in Fig.3 (the gas-sensitive layer with 300 nm thickness), the sensor response is highly depending on the relative humidity concentration and increases when relative humidity increases. Therefore, from the practical point of view a reference relative humidity sensor needs to be used as well as accurate algorithm that include the RH influence. Although, recently it was proven that relative humidity impact can be reduced in the wide range concentration range by the utilization of $CuO/SnO_x$ and $SnO_x/CuO$ [26] heterostructres for $NO_2$ detection, p-type or n-type and n-type or p-type, respectively. However, such measurements were conducted only in DC mode, and in the microwave frequency range will be a subject of research in the next tasks of the project. Another, crucial parameter is the thickness of the active layer. As can be observed in Fig.4, the gas sensor signal goes up with increased thickness of the active layer, however, the optimal thickness should not exceed 300 nm for the deposition technique used, i.e. glancing angle deposition technique. Above the optimum thickness the effect of nanorods structure will not be achieved.

At this point, it is worth to be mentioned that the primary goal is to develop the gas-sensitive material that can be deposited by the magnetron sputtering technology during the integrated circuit fabrication to enables cheap mass production. Therefore, even if other sensing techniques exhibit better results, the possibility to integrate and minimalize the number of processes (reducing the overall cost) is the only solution for using such sensors globally on the automotive market.

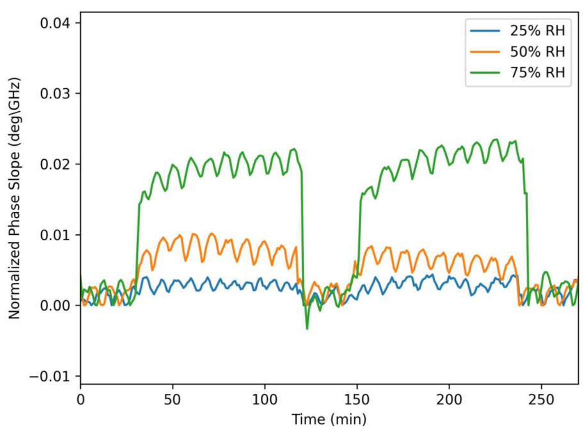


Fig. 2. The gas-sensor response under the exposure to 5 ppm of $NO_2$. The thickness was 200 nm (gas-active material).

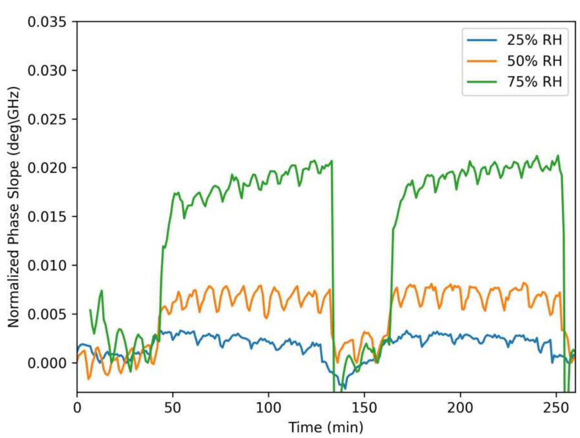


Fig. 3. The sensor response under the exposure to target gas: 5 ppm of $NO_2$. The thickness of the active layer: 300 nm.

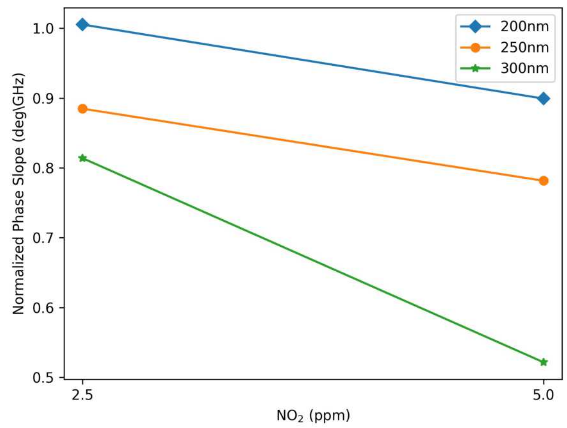


Fig. 4. The gas-sensor response under the exposure to 2.5 ppm and 5 ppm of $NO_2$. The thickness of the detecting layer: 200, 250, and 500 nm.

## IV. CONCLUSIONS

The microwave-based gas sensors, where metal oxides are used as a gas-sensitive layer offer indisputable features - the possibility to work at room temperature under various environmental conditions. Within this study, the gas detector response of the copper oxide-based gas sensors to nitrogen dioxide was shown. Firstly, copper oxide was used due to the previous outstanding results in the field of various gas-sensing applications, including nitrogen dioxide detection. Secondly, the primary goal of the research is to find the gas-sensing material that will work at the microwave frequency excitation in a wide range of ambient conditions. Finally, the material needs to be cheap and can be easily deposited during the integrated circuit technology processes. Taking into account all these requirements, CuO seems to be very promising results, however, further experiments are needed to reduce the impact of the relative humidity.

## ACKNOWLEDGMENT


The research was supported by the Norway Grants 2014–2021 via the National Centre for Research and Development. The work was carried out in the Biomarkers Analysis Laboratory AGH at the Institute of Electronics AGH.


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