Critical Influence of Dielectric Sensitive Material and Manufactured Process in Microwave Gas-Sensing: Application of Ammonia Detection with an Interdigital Sensor

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ABSTRACT: In this paper, authors propose a study on microwave gas sensors and the influence of critical key parameters such as the sensitive material and the circuit conception process. This work aims to determine the influence of these parameters on the quality of the final response of the microwave gas sensor. The fixed geometry of the sensor is a microstrip interdigital capacitor coated with a sensitive layer excited with two 50 Ω SMA ports. The sensitive material has been chosen in order to interact with the target gas: ammonia. Indeed, this gas interacts with phthalocyanine and metal oxides like hematite, TiO₂. To explore the effect of the circuit manufacturing process, three series of samples are prepared. The first series of sensors is produced by classical UV photolithography (process) in the laboratory. The second series of sensors is produced by a subcontractor specialized in rf circuits. The third series is obtained by the experimental platform of the FEMTO-ST laboratory with EVG620 Automated Mask Alignment System Nanoimprint lithography in a clean room. To examine the reliability of this gas sensor at room temperature, it was exposed to different ammonia gas concentrations from 100 to 500 ppm in an argon flow to eliminate coadsorption phenomena. According to the recorded frequency responses, the reflection and transmission coefficients show a change of resonance amplitude due to electrical characteristic modification. This can be correlated to the presence of gaseous ammonia. The chemical nature of the sensitive material layer has a major influence at the excited frequency range. The process of conception influences the sensor sensitivity. The analysis of the results shows a strong correlation between the injected ammonia concentration and its frequency response. The influence of the critical key parameters cited is discussed here.

INTRODUCTION

In 2015, the UNFCCC parties agreed in Paris on the mitigation of the greenhouse gas emission levels by 2025. As a matter of fact, nitrous oxide (N₂O) has a global warming potential 265−298 times greater than CO₂.¹ N₂O is produced by ammonia (NH₃) transformation in the air generated from various domains like agricultural activities or agri-food industry.² The environmental monitoring of volatile chemical species requires in situ and real time measurements. Consequently, numerous types of gas sensors were developed over the last century based on direct or indirect sensing methods. A recent review³ highlights the evolution of gas sensing by microwave transduction, which appeared 20 years ago. In liquid or gas detection, this transduction has the advantage of being adaptable to conductive,⁴⁵ semiconducting⁶ (metal oxide at nanometric level), or insulating sensitive material⁷−¹³ (phthalocyanine,⁷−⁹,¹² metal organic framework,¹⁰,¹¹ imprinted molecular polymer¹³). The principle is based on variations within dielectric and conductive properties of a gas sensitive material at microwave frequencies. The main
parameters of this technology are intuitively the geometry of the microwave circuit and the characteristics of the sensitive layer (material, width). The geometry defines the resonant frequency to characterize the sensor, whereas the sensitive material induces the type of interaction with pollutants (adsorption, absorption...). However, few works evaluate the different parameters coming into play in the response of gas sensing and microwave gas sensing especially. To evaluate the influence of other key parameters, authors fixed the geometry and the width of the sensitive layer. In this paper, the used microwave gas sensor is an interdigital capacitor (IDC). This type of circuit is commonly used as a conductimetric or surface acoustic wave gas sensor. In sensing applications, IDC circuits can be a good alternative especially when miniaturization, integration within lab-on-chip systems and low manufacturing cost criteria are needed. This work evaluates the influence of parameters such as the manufacturing process or the type of sensitive material on the microwave response. Afterward, the sensor response is evaluated thanks to a random profile of ammonia concentration.

**MATERIAL AND METHOD**

**Gas Sensing Measuring System.** Prior to the measurement, no processing was carried out on the gas-sensitive layer of the sensor. It was left uncovered at ambient humidity. During gas detection measurements, the sensor is placed into a hermetic cell maintained at room temperature with constant relative humidity (as shown in Figure 1). The cell is enclosed in a compact mini electromagnetic compatibility (anechoic) chamber to reduce the effect of potential electromagnetic disturbances. To remove mechanical vibrations, the chamber is fixed on a marble table. The gas supply unit consists in three different parameters coming into play in the response of gas sensing. To remove mechanical vibrations, the chamber is fixed on a marble table. The gas supply unit consists in three different parameters coming into play in the response of gas sensing. To remove mechanical vibrations, the chamber is fixed on a marble table. The gas supply unit consists in three

![Figure 1. Experimental setup and sensor design.](image)

in a compact mini electromagnetic compatibility (anechoic) chamber to reduce the effect of potential electromagnetic disturbances. To remove mechanical vibrations, the chamber is fixed on a marble table. The gas supply unit consists in three mass flow controllers (EL-Flow meter Bronkhorst) to manage the flow of carrier gas (argon here) and pollutants. The flow is fixed at 0.500 ± 0.025 L·min⁻¹ throughout the experiment. A pneumatic fast switching valve (5 s) enables a quick changeover between exposure to carrier gas flow with pollutant and carrier gas flow only. The sensor is exposed to concentrations from 500 to 100 ppm with 100 ± 7 ppm steps. Ammonia diluted with argon and pure argon [from certified bottles mixed with argon (Air Liquide)] are alternatively to the sensor and maintained during 1 and 4 min, respectively. The renewal rate in the hermetic cell is close to 20 s. In order to regulate the gas flow and avoid sudden pressure variations inside the cell, a proportional-integrated-derivative controller is thereby used. All of the measurements are conducted with a Vector Network Analyzer (Rohde & Schwarz ZVB20) after a SOLT calibration (short-open-load-thru) at the input of the sensor. The calibration kit is a ZV-Z32 from constructor. The whole experimental setup is controlled by a National data acquisition device.

**Type of Sensor and Response. Design of the Sensor.** The sensor is based on a microwave transduction system performed by a PCB IDC model printed on a 0.76 mm thickness h of Rogers/Duroid RT6002 substrate (εr = 2.94, tan δ = 1.2 × 10⁻³) and coated with a gas sensitive material (sorbent in physicochemistry approach). The IDC consists in two interpenetrating comb-shaped electrodes. Each 2 mm width electrode is fed by a 50 Ω port and presents an N/2 number of fingers (N = 6), length (15 mm), and width (0.18 mm) (Figure 1). The gap between two successive fingers as well as the gap between a finger and the electrode ahead are equal and have 0.18 mm width. A complete description of the design is given in ref 6. The IDC dimensions are calculated to target a reflection coefficient frequency band located around 2–4 GHz. The simulated values are then optimized with CST 5-Parameter Measurement of Sensor. The scattering parameters of the IDC prototype are measured using a vector network analyzer. The frequency accuracy is close to 100 Hz and the uncertainty of magnitude is 0.02 dB. Figures 2 and 3 show a relative correlation between simulated and measured S-parameters (here S₁₁) results without sorbent. Because of the symmetry of the IDC design, reflection coefficient S₁₁ and transmission coefficient S₂₁ are used only, assuming a symmetrical S parameter matrix (S₁₁ = S₂₂ and S₂₁ = S₁₂). Only S₁₁ is shown in this article. Results will be analogous in the case of insertion loss (S₂₁). All of the observed dips in the measurement are clearly visible and similar to the ones obtained in the simulation plots. As for the observed differences, like the amplitude and the resonance frequency value accuracy, it could be explained by a possible inaccuracy with dimensions due to manual production of the prototype. The first frequency peak is simulated to 2.46 GHz. The difference between the simulated frequency and the experimental data is 90 MHz which represents 3.7% of the simulated value. The second tip is close to 2.98 GHz with a difference

![Figure 2. Return Loss (S₁₁) vs frequency with the different dip frequencies identified: comparison between simulated results (CST) and experimental data of sensor response without sensitive material.](image)
crystallites, the most commonly polymorphs. The commonly composition. In fact, P25 is composed of anatase and rutile However, the supplier does not report the crystalline which is often used as a reference in laboratory studies. Degussa P25 is one such commercially available material S network analyzer is required to discriminate the complex component variation of the response. In these cases, a vector network analyzer is required to discriminate the complex component variation of the response. To eliminate the influence of gas presence and its concentration over a large frequency band, we propose to display the difference between the spectra obtained for different concentrations and the reference spectrum (without NH3 gas). To eliminate the influence of the design, we choose to represent this difference with its absolute value and according to the following expressions

$$\Delta S_{11} = S_{11}(\text{Ar} + \text{NH}_3) - S_{11}(\text{Ar})$$ (1)

The molecular material17 and hematite19 induce a weak variation of the response. In these cases, a vector network analyzer is required to discriminate the complex component response (real + j imaginary) of S11. In TiO2 case, a scalar network analyzer is sufficient to track the signal (magnitude of S11 in dB). The response variation is close to 0.12 dB. Thus, to evaluate the influence of the material, the metal oxides are here chosen with the same deposition protocol. The first metal oxide studied is Degussa P25 (Evonik), Aeroxide TiO2. Degussa P25 is one such commercially available material which is often used as a reference in laboratory studies. However, the supplier does not report the crystalline composition. In fact, P25 is composed of anatase and rutile crystallites, the most commonly polymorphs. The commonly reported ratio being typically 70:30. According to Sola et al., BET surface area of P25 is close to 50 m²·g⁻¹, pore volume to 0.31 cm³·g⁻¹, and mean crystallite size is 26 nm (A) or 49 nm (R).

The other sensing material is hematite α-Fe2O3. These particles have been produced with microwave thermohydrol-
ysis by the authors.23 The XRD results demonstrated that the only phase obtained during the synthesis is pure hematite α-Fe2O3. Three morphologies have been chosen: pseudocube, rhomboheda, and spindle. The pseudocube terminology is due to a few degrees difference in the polyhedron angles with respect to those of a perfect cube (86 and 94° instead of 90°). The rhomboheda shape is perfectly faceted, whereas spindle particles have an aspect ratio of 10. The chosen sample has a specific area close to 50 m²·g⁻¹, allowing the study of morphological effects on gas sensing properties, regardless of the size effects. All morphology analyses have been made with scanning electron microscopy (JEOL JSM-7600F) and transmission electron microscopy (JEOL JEM-2100, LaB₆).

The deposition process is also a key factor for the sensor response.14 The TiO₂ sensitive layer is deposited according to a typical procedure where 1.5 g of TiO₂ P25 is properly dispersed in 5 mL of aqueous solution of polyethylene glycol 20k, acetyl acetone, and Triton X-100. Then, one drop from this solution is applied on the sensor surface and spread following a “doctor blade”26 protocol. The coated area is controlled with a mask and its thickness is evaluated to 25 ± 3 μm by mechanical profilometry. After the deposition process, the sensor is placed in a ventilated oven (40 °C) to remove the water and acetyl acetone. The polyethylene glycol 20k plays the role of binder. The sensing material is the major constituent of the film (up to 95%). This protocol leads to robust films that can be reproduced in thickness. Consequently, it is not necessary to carry out thermal densification treatments. In any case, these heat treatments would damage the microwave circuit and will induce growing of particle size. As part of frequency applications, it is important to know the dielectric properties of the sensitive layer inducing variation of S parameters of sensor. However, they are highly correlated to the deposit process conditions (annealing, spin coating...) and the particle morphology.23

Previous permittivity investigations were conducted, indicating that relative permittivity of this TiO₂ superstrate (in electromagnetic propagation approach, i.e., sensitive material) is around 6.4 ± 0.1, whereas the hematite superstrate is close to the substrate.

The dielectric permittivity of the sensitive material is obtained by the reverse-fitting process. This process consists to determine the value of permittivity by recurrent CST simulation to result the same S parameters of the real sensor. The sensing material is a composite material based on TiO₂ and its associated binder. Thus, the estimated permittivity differs from the value of crystal oxide. In the case of the PcCo, the value is consistent with Soliman et al.25 The TiO₂ estimation is close to the value of permittivity of a mix of

| sensitive material | PcCo | α-Fe₂O₃ | TiO₂ |
|--------------------|------|---------|------|
| permittivity       | 220  | 1.3 - 1.6 | 6.9 |
| S₁₁ response 300 ppm | real/imaginary ≈ 3 × 10⁻⁴ | real/imaginary ≈ 2 × 10⁻⁴ | magnitude ≈ 0.12 dB |

Table 1. Relative Magnitude of the Response of Sensor toward 300 ppm NH₃ with Different Three Sensitive Materials (TiO₂,14 PcCo,15 Hematite19)

![Figure 3. Insertion loss (S₁₁) vs frequency with the different dip frequencies identified: comparison between simulated results (CST) and experimental data of sensor response without sensitive material.](https://dx.doi.org/10.1021/acsomega.0c00596)
The hematite value of the permittivity is consistent with the ref 22.
The PcCo20 and the used polymer present few loss tangent of
dielectric permittivity. For the metal oxides, estimation of loss
tangent is close to 0.1−0.3 and affects the signal magnitude
specifically.

A downward frequency shift can be observed when the IDC
is coated with the gas sorbent thanks to the representation of
the frequency evolution of S11 (experimental) parameters with
and without the gas-sensitive layer (Figure 4) with the same
design. In the following, the analysis of the first peak of S11
is developed. A similar study is possible with the second peak.
The permittivity in this frequency range (2−4 GHz) is almost
constant.

While TiO2 frequency shift is about 150 MHz (in first peak)
for the reflection coefficient (S11), it is about 800 MHz for the
transmission coefficient (S21). In the case of hematite, S11 shift
is close to 80 MHz and S21 frequency shift is 40 MHz. Then,
the results are consistent with the previous results (Table 1).
They highlight the influence of sensitive material permittivity
which needs to be greater than substrate permittivity.

Manufacturing Process. The sensor performance is linked
to the sensor manufacturing process. Sensor characteristics
from the same series can be very different.27 A Photolithography
is one of the key processing steps in electronics today (semiconductor
and IC industry). Thus, the quality of the manufacturing process
for circuits plays a major role on the characteristics of the sensor
in presence of gas.

This study aims to explore the effect of the circuit manufacturing
process by means of three series of experiments.
(a) The first series of sensors is produced by classical UV
photolithography in laboratory. The mask is obtained by
ink-jet printing. The IDC sensor is made following a
standard PCB chemical etching process using a deposit
of positive resin layer (S1813) spread with a spinner.
This method is of low cost and easy-to-do; however, the
accuracy of these masks and sensors depends on
operator’s experience, temperature, and room humidity.
The reproducibility requires a careful use during each
step (photoresist coating, soft bake, exposure, exposure,
development, hard bake). However, the best spatial
resolution is close to 50 μm.

(b) The second series of sensors is produced by a
subcontractor specialized in rf circuits. The spatial
resolution is around one mil (∼25 μm). The industry
traditionally uses a lithography process based on a
polyester mask in contact with a large, resist-coated
substrate. A mini-series of 100 tailor-made sensors is
obtained after 2 weeks and the price is about 106 per
unit.

(c) The third series is obtained by the experimental platform
of FEMTO-ST laboratory with EVG620 Automated
Mask Alignment System Nanoimprint lithography in a
clean room. With the optional tools for UV-nanoimprint
lithography, the pattern can be under 100 nm size. This
type of manufacturing is the first step for nanoimprint
lithography (e-beam, RIE...). However, this method
needs devices and operator’s specific skills.

To clarify the influence of the manufacturing process, the
evolution of magnitude sensor response (dB S11) at the first
peak of frequency 2.32 GHz is plotted as a function of
ammonia concentration, as shown in Figure 5. In fact, gas
interaction with the sensitive material layer could only take
place on the surface which might cause a permittivity
modification on a thin TiO2 stratum. Therefore, we assume
that injected gas concentration with such low levels is not able
to trigger a frequency shift on the sensor scattering parameters
so far. However, based on the observed collected data, it is
clearly obvious that gas concentration influences S-parameters
in terms of amplitude. The presence of pollutant does not
induce a resonant frequency shift but an amplitude shift.
The evolution of each series is found to be a near-linear function
with ammonia concentration between 200 and 500 ppm. The
frequency is 2.32 GHz.

Figure 4. Frequency evolution of sensor’s response without and with
two types of sensitive material coating (TiO2, hematite).

Figure 5. Impact of the manufactured processes (clean room,
subcontractor, classic UV lithography) on the sensibility of the sensor.
The ammonia concentration is between 200 and 500 ppm. The
frequency is 2.32 GHz.
influence the response in this frequency range. The sensor developed by a specialized subcontractor appears to be a reasonable compromise. This conclusion is validated with the Figure 6 which represents the evolution of the tailor-made sensor response with decreasing concentration from 500 ppm to 10 ppm. The associated figure is the calibration curve.

At 100 ppm, it should be noticed that the response with a tailor-made sensor is very close to with a clean room sensor. A similar behavior can be assumed under 100 ppm. The sensitivity of tailor-made sensor is around $2.1 \times 10^{-4} \text{dB/ppm}$, whereas the clean room sensor has a sensitivity close to $5 \times 10^{-4} \text{dB/ppm}$. It represents 2.5 times the sensitivity of the first series of sensors.

Then, the representation of NH$_3$ gas concentration influence on S-parameters for arbitrary selected frequency (here 2.32 GHz) highlights a strong correlation between the sensor response and the gas concentration (Figures 5−8).

**RESULTS AND DISCUSSION**

**Cycle of Concentration and Desired Characteristics.** As a result, the optimal configuration of the sensor by microwave transduction in this work uses TiO$_2$ (P25) as sensitive material with a “Dr Blade” deposition. The thickness of the sensitive material is close to electrodes thickness. The manufactured or tailor-made design is obtained by subcontracting. To ensure the reliability of this proposed sensor in terms of reversibility, sensitivity, and stability, the sensor is exposed to cycles of increasing and decreasing NH$_3$ gas concentrations. Ammonia exposure is divided into three temporal parts.

The first section is dedicated to an increasing concentration cycle gradually every 100 ppm. Each step of concentration is repeated. Thus, this part is dedicated to estimate the signal stability (ammonia adsorption) and the baseline evolution without pollutant concentration. The second section is linked to the quantitative behavior of the sensor. The pulse for increasing concentration is shorter than the pulse for decreasing concentration. This decided and chosen difference tends to follow the drift of the response and its recovery time. The response time of the sensor is calculated as the duration for the sensor response to reach 90% of its saturation value under ammonia exposure mixed with argon. The recovery time is calculated as the duration corresponding to the decrease of sensor response by 90% of its base value after ammonia emission has been stopped.

The third section is dedicated to the signal stability and sensor reversibility.

**Analysis of Response of Each Part of Cycle.** In the first section (Figure 7), the baseline of the signal drift is 0.02 dB, #TABLE 2. COMPARISON BETWEEN MANUFACTURED PROCESS ON THE SENSIBILITY (dB/PPM) OF THE SENSOR AT 100 PPMA

| Process            | Manufactured | Clean Room | Subcontractor (tailor-made) | Classic UV lithography |
|--------------------|--------------|------------|------------------------------|------------------------|
| Resolution         | <100 nm      | <10 nm     | >50 nm                       |                        |
| Cost/unit          | ≫10€         | 10€        | 5€                           |                        |
| Sensibility        | $10^{-4}$    | $2 \times 10^{-4}$ | $5 \times 10^{-4}$          |                        |

$^a$The frequency is 2.32 GHz.

**Figure 6.** Relative variation of $S_{11}$ (dB) sensor versus concentration (NH$_3$). The frequency is 2.32 GHz.

**Figure 7.** Quantitative performance and drift baseline of response sensor: temporal evolution of response sensor (2.32 GHz) at increasing pulse of concentration.

**Figure 8.** Reversibility characteristics and time response of sensor: temporal evolution of sensor response for exposure of decreasing and increasing ammonia concentration.
whereas the lower concentration induces a signal variation (peak-baseline) around 0.1 dB. At each concentration, the difference between two responses is close to 0.04 dB (−10.86 dB). For this worse case, the signal-to-noise ratio is close to 50. For the highest concentration, the variation between two similar responses is close to 0.04 dB with a signal amplitude (peak-baseline) close to 0.2 dB.

Consequently, the raw signal supplied by the sensor is operable to discretize the concentration with a relative stability of the signal and its baseline.

In the second section (Figure 8), the drift of the baseline is also close to 0.02 dB. At first sight, the evolution of the response follows the application of concentration. However, when looking further into it by zooming at each concentration, we notice that each concentration presents a slight amplitude modification (−0.02 dB) between response associated with the increasing and decreasing concentration. The thermal variation of the setup is close to 1 °C during 2 h. It does not explain the significant variation of 0.02 dB. The desorption kinetics is governed by the adsorption process. This variation underlines a slow desorption which can be explained by the presence of two mechanisms on TiO\textsubscript{2} surface (physisorption and chemisorption). Then, microwave transduction is a convenient tool to follow the adsorption phenomena and to distinguish them.

Argon is used as a carrier gas to exclude the influence of oxygen and coadsorption phenomena in the recovery process. The use of another carrier gas other than argon (example air) would induce the above phenomena. The short duration of increasing concentration pulse highlights the ability to tend to 90% of the response for this pulse duration. This time is close to 25 s. Contrariwise, the recovery time of gas sensor is longer than the response time (∼120 s). Characteristic times of entrance and departure of ammonia are quite different. This difference could be easily explained. First, and as already reported, ammonia is adsorbed on TiO\textsubscript{2} as coordinated NH\textsubscript{3} and NH\textsubscript{4} due to Lewis acidity of surface. These two species are induced by two acid sites of different strength. Moreover, P25 shows a type II isotherm characteristic of macroporous materials. A H\textsubscript{2}-type hysteresis loop is observed at high relative pressure and can be related to the typical capillary condensation and evaporation processes that take place in the presence of large pores.

The departure of ammonia NH\textsubscript{3} needs to reverse the reaction which has produced ammonium NH\textsubscript{4}. This conclusion is similar to Xia’s work about hydrogen adsorption by TiO\textsubscript{2} thin film sensor. Thus, there is a correlation between gas concentration and sensor response.

Obviously, the tiny adsorbed ammonia is sufficient to induce a dielectric change able to be detected. It is difficult to discriminate the effect of coordinated NH\textsubscript{3} and NH\textsubscript{4} due to Lewis acidity of surface. These aspects must be studied with several samples of TiO\textsubscript{2} with various macroporous structures induced by thermal treatment and deposition conditions.

An important aspect should be mentioned. The P25 sample includes adsorbed water which is strongly bonded to the TiO\textsubscript{2} surface. These results gave experimental evidence of ammonia detection concomitantly with water presence. Water is the most abundant interferent in ambient air, with the concentration of saturated water vapor pressure at 30 000 ppm at room temperature (100% relative humidity, RH). Thus, if a new sensor under development is thought to detect 1 ppm of a toxic vapor at 50% RH air (water vapor at 0.5P/P\textsubscript{r}), this new sensor must operate at a 15 000-fold overload from water vapor interference. The sensibility of almost all sensors to water vapor represent the largest challenge for their practical applications. Chemical interferences represent one of the key environmental noise parameters of the sensed environment. These results show potentiality of microwave sensing in real-world scenarios which significantly complicate the detection capabilities of laboratory sensor prototypes.

The third section (Figure 9) of the cycle highlights the drift of the baseline which is also present in the sensor response with pollutant concentration (drift of 0.02 dB). However, this figure highlights a satisfactory sensor reversibility at 200 ppm here. Under this concentration, the response is similar beyond 500 ppm exposition (case A) or 0 ppm exposition (case B).

Consequently, the optimal sensor configuration is evaluated by the mean of this cycle of concentrations. The drift of the baseline is estimated to be 0.02 dB with a signal to ratio close to 50. Nevertheless, the response is quantitative and underlines a significant chemisorption. This chemisorption influences the difference between response time and recovery time. The reversibility of the sensor is also demonstrated. The characteristics of the sensor are depicted on the following Figure 10.

**CONCLUSIONS**

This paper presents a microwave gas sensor using a microstrip IDC coated with a sensitive material for ammonia gas detection in argon flow. The electromagnetic simulations of...
the sensor with and without gas sensitive layer presented good agreement with the measurement. It has been demonstrated in this work that there is a strong correlation between the injected targeted gas concentration and the sensor response through microwaves transduction. This paper also highlights the influence of the sensitive layer dielectric permittivity on the magnitude evolution of the response. The proposed sensor based on microwaves transduction showed interesting and promising results about the characteristics of the gas sensor (drift, stability...). However, minimizing the dimensions of the sensing system remains the challenge of our future works on this gas sensor system. The next step of this work is dedicated to evaluating the response with coadsorption phenomena (in air flow).

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Notes

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