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Feasibility of Passive Gas Sensor Based on Whispering Gallery Modes and its RADAR Interrogation: Theoretical and Experimental Investigations

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Abstract: Feasibility of gas sensors based on Titanium Dioxide (TiO_2) dielectric resonator operating with whispering-gallery modes is presented. The gas or humidity adsorption modifies the TiO_2 dielectric permittivity and such modification induces variation in the resonant frequencies of high-Q whispering-gallery modes in the millimeter-wave frequency range. Full-wave electromagnetic simulation indicates that the measurement of this variation allows the derivation of few pars-per-million fluctuations in gas or humidity concentration. For validation purposes very first prototypes of resonator operating with whispering-gallery modes in the millimeter-wave frequency range are micro-machined. The obtained measured performances confirm that such high-Q resonant modes are very sensitive to small variations in dielectric resonator permittivity. Moreover we validate experimentally that these small variations can be remotely detected from the RADAR interrogation of an antenna loaded by the whispering-gallery modes resonator. *Copyright © 2010 IFSA.*

Keywords: RF gas sensor, Dielectric resonator, Whispering gallery mode, RADAR interrogation, wireless sensors network

1. Introduction

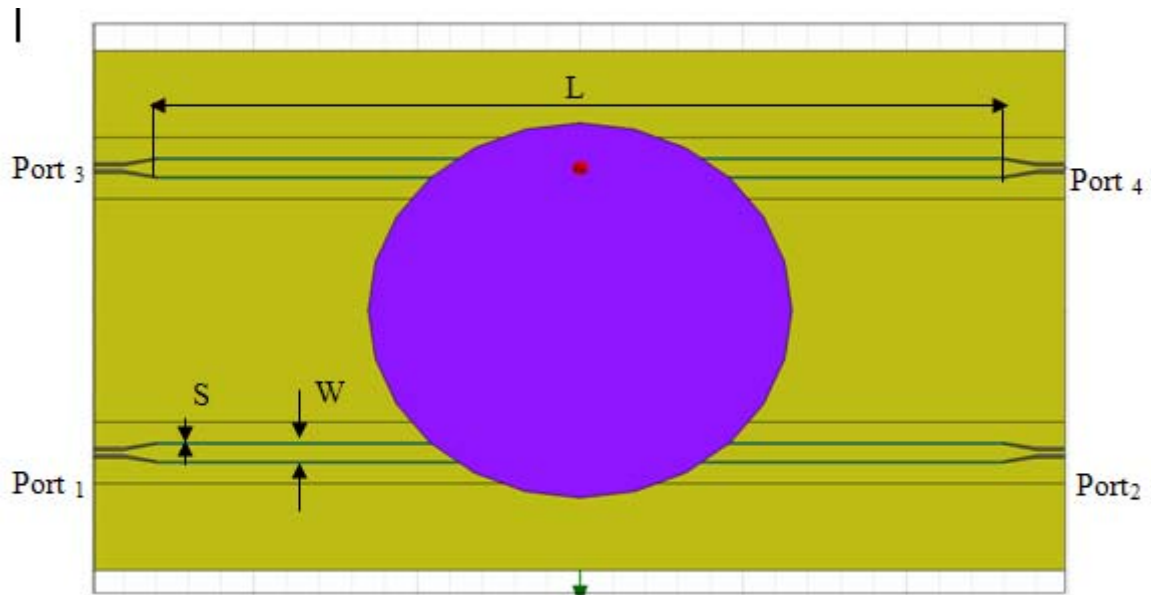
The gas sensors are the subject of intense research for many years. Recently demand for gas detection methods and measurement of their concentration has significantly increased. This interest is mainly due to environmental considerations, security and process control. Sensors based on metal oxides have

great potential and are of growing importance in the market for large volume applications and low precision requirements [1]. However such sensors require high temperatures (above 300°C), high-power consumption and their operating mode at low frequencies implies the use of modulation techniques for the wireless transmission of measured gas concentration [2]. Very recently passive sensors based on electromagnetic transduction have been reported [3-5]. For such passive sensors the wireless transmission of data and the measurement of physical quantities, such as pressure and temperature, share the same electromagnetic wave. This transduction mode requires no active power supply and no signal processing circuitry; this results in low power losses due to the low coupling coefficient between the electromagnetic and acoustic waves. The remote measurement from the wireless interrogation of such sensors by a frequency-modulated signal has been reported [6-7].

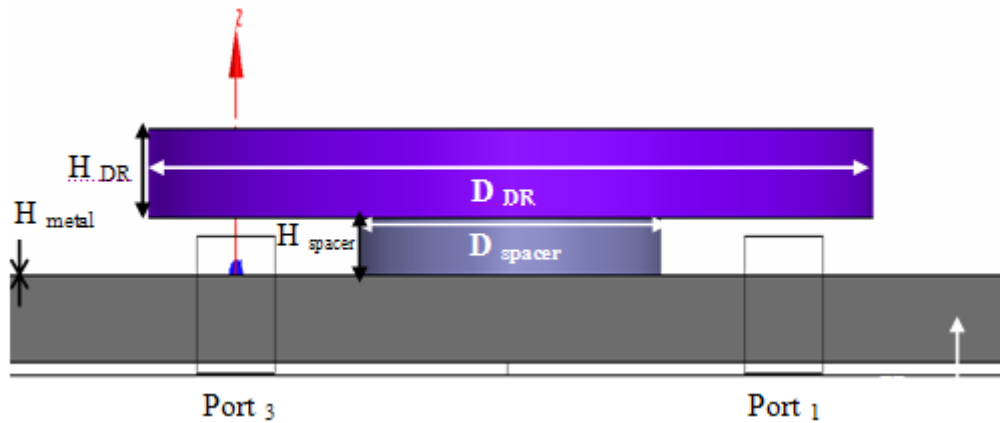
Based on theoretical and experimental investigations, we present here the feasibility study of gas sensors based on the electromagnetic transduction. This sensor uses Titanium Dioxide (TiO_2) dielectric resonators operating in whispering-gallery modes. This metal oxide has a permittivity relaxation at very high frequency due to the presence of gases [8]; this phenomenon is exploited here for our new gas detector in the millimeter-wave frequency range. The whispering-gallery-modes present a high quality factor Q [9-10], allowing in principle the design of high sensitive gas sensors. Moreover these modes can be excited in different ways and can be established inside the dielectric resonator in the azimuth as well as in the radial directions allowing quasi-isotropic gas detection. The operating principle of the proposed gas sensor is given as follows: (1) the gas adsorption modifies the TiO_2 permittivity of the dielectric resonator, (2) such modification induces variation in resonant frequencies of high- Q whispering-gallery modes in the millimeter-wave frequency range and, (3) the measurement of this variation allows deriving the fluctuation of few parts-per-million (p.p.m) in gas concentration. The design methodology of the proposed gas sensors and the very first electromagnetic simulation results have been already reported by the authors elsewhere [11-12]. Experimental validations of the design as well as additional simulation results are discussed in the present paper. Moreover, for the first time, we show that the frequency-Modulated Continuous-Wave (FMCW) RADAR interrogation of the proposed passive gas sensor may allow the remote detection of small fluctuations in gas concentration.

2. Gas Sensor Description and Electromagnetic Simulation Results

The proposed gas sensor is shown in Fig. 1. In the millimeter-wave frequency range, the Quasi-Transverse Electro-Magnetic (TEM) fundamental mode propagating along the Co-Planar Waveguides (CPW) is coupled with the whispering-gallery modes (WGM) in the TiO_2 dielectric cylindrical resonator (DR). Such structure has been proposed for millimeter-wave filter application [13] using High Resistivity Silicon DR. The design of this structure for gas sensing application using metal-oxide DR was described elsewhere [10-11]. The two parallel CPW are micro-machined on a thin dielectric membrane (thickness $H_{\text{membrane}} = 1.4 \mu\text{m}$ and relative permittivity: 1.09) with the following dimensions: thickness $H_{\text{metal}} = 1 \mu\text{m}$, length $L = 13 \text{ mm}$, slot width $W = 300 \mu\text{m}$ and strip width $S = 30 \mu\text{m}$. Such dimensions allow achieving 75Ω -characteristic impedance for the two membrane-supported CPW. The dielectric relative permittivity of the TiO_2 is 80 [14]. The DR presents a diameter $D_{\text{DR}} = 6.5 \text{ mm}$ and a thickness $H_{\text{DR}} = 360 \mu\text{m}$; it is mounted above the surface of the two CPW using alumina spacer of relative permittivity= 9.8, and with a diameter $D_{\text{spacer}} = 2.7 \text{ mm}$ and a height $H_{\text{spacer}} = 230 \mu\text{m}$. This height is adjusted to achieve high electromagnetic coupling between the WGM in the DR and the quasi-TEM mode in the two CPW. The whole sensor is laid out on a High Resistive Silicon substrate with relative permittivity of 11.6 and thickness $H_{\text{Si}} = 350 \mu\text{m}$. When loading the access 2 and access 4 by standard 50Ω -impedances and observing the transmission between access 1 and access 3 (see Fig. 1) the structure acts as a multi-band filter in the millimeter-wave range.



(a)



(b)

Fig. 1. Views of the fabricated gas Sensor: (a) top view; and (b) side view.

This behavior is illustrated in Fig. 2 where the transmission coefficient obtained from full-wave electromagnetic simulations is given versus frequency: transmission peaks at many resonant frequencies are apparent between 25 GHz to 40 GHz.

Some of these frequencies correspond to the resonances of whispering gallery modes in the DR. For example, at 26.01 GHz the $WGH_{5,0,0}$ mode resonates while the $WGH_{6,2,0}$ mode resonates at 33.24 GHz. The simulated loaded quality factor Q at these two transmission peaks is 370. These two WGM are unambiguously identified from Fig. 3 displaying characteristic magnetic field distribution at the two corresponding resonant frequencies. At 33.24 GHz low insertion losses (5.70 dB) are obtained indicating that the structure achieves a good electromagnetic coupling between the CPW quasi-TEM mode and the corresponding WGM in the DR.

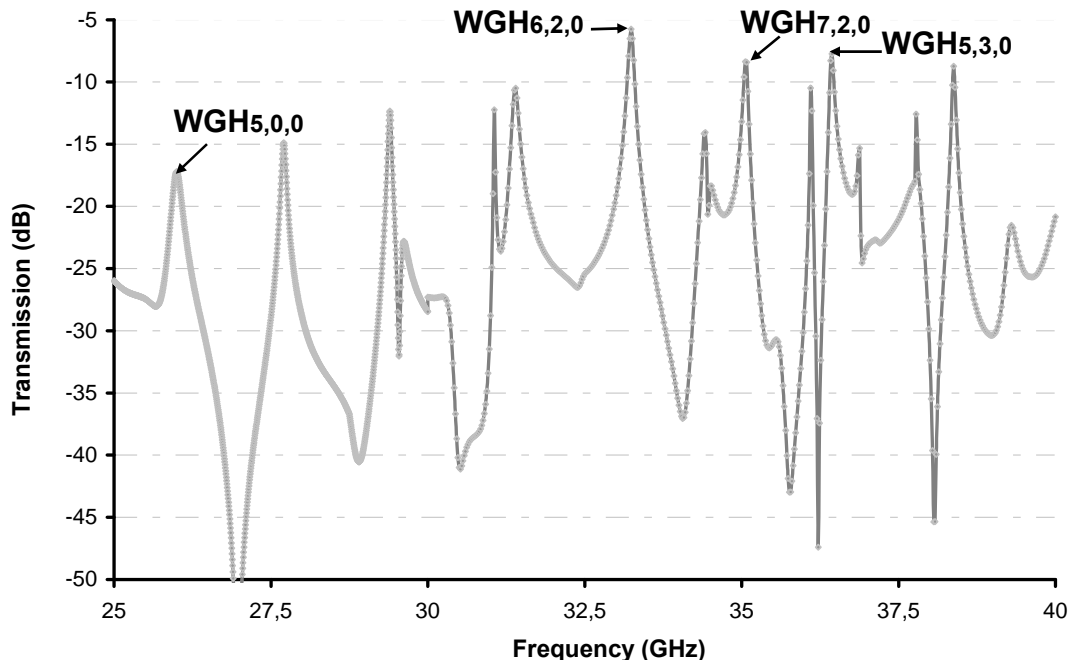


Fig. 2. Simulated transmission coefficient from port 1 to port 3 with ports 2 and 4 loaded by 50 Ω .

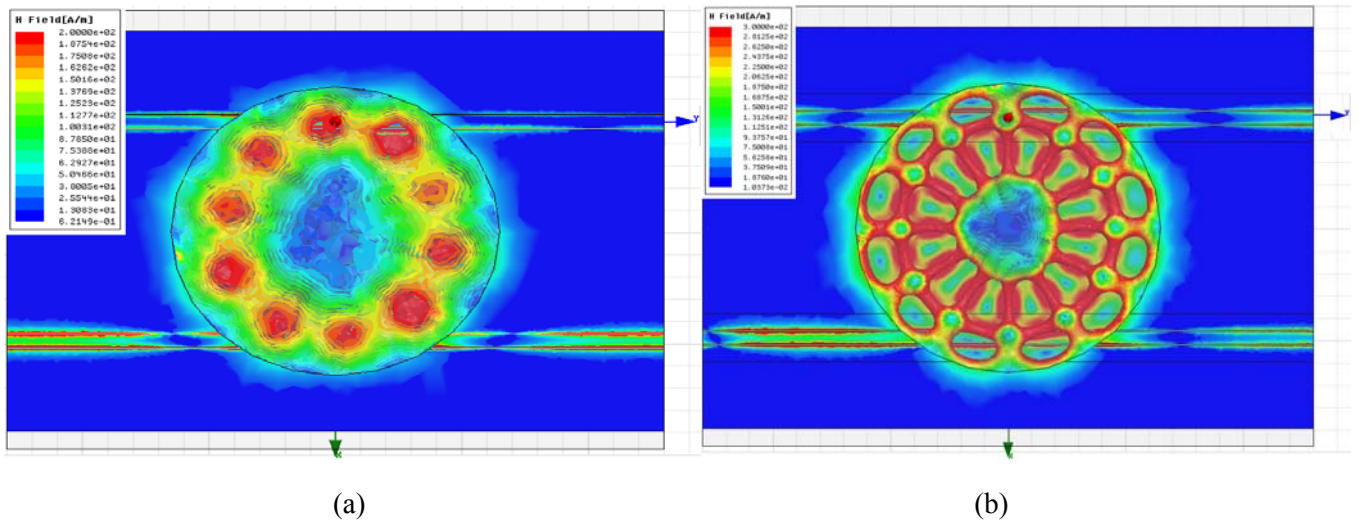


Fig. 3. Simulated magnitude of the magnetic field at the resonant frequency of the: (a) WGH_{5,0,0} mode (at 26.01 GHz); and (b) WGH_{6,2,0} mode (at 33.34 GHz)

The permittivity of TiO₂ material depends on the humidity (H₂O) [15]: typically if the proportion of H₂O increases by 30 %, the TiO₂ dielectric permittivity decreases by 18 %. To analyze the impact of such permittivity variability on the WGM resonant frequencies full-wave electromagnetic simulations based on the Finite-Element Method [16] were performed. Fig. 4 shows the transmission coefficient from port 1 to port 3 versus frequency for various DR relative permittivities. It could be observed that some transmission peak frequencies are affected by small variations in the DR permittivity. These transmission peaks occur at the resonant frequencies of WGM and can be selected for offering the maximum variability versus humidity concentration.

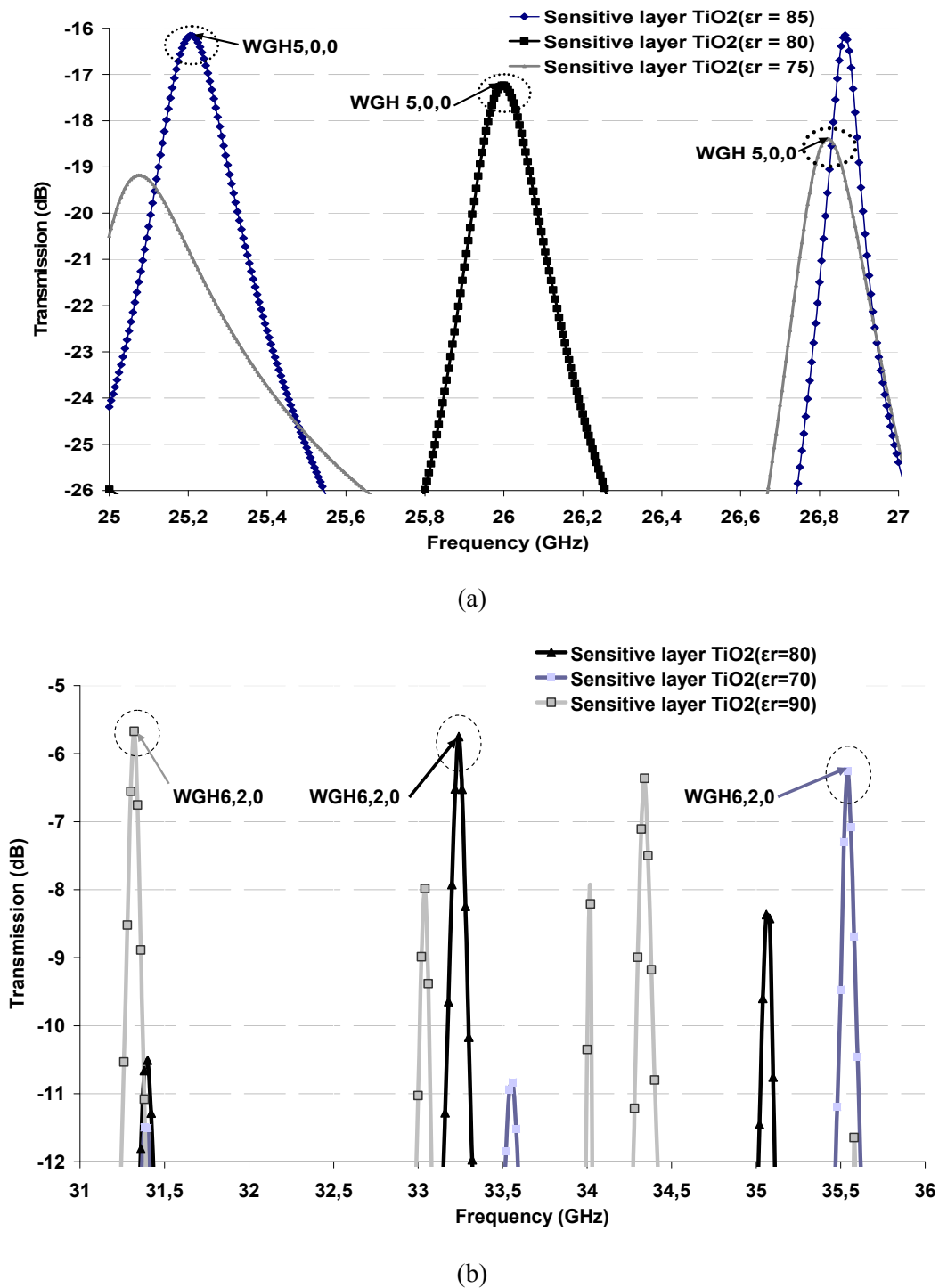
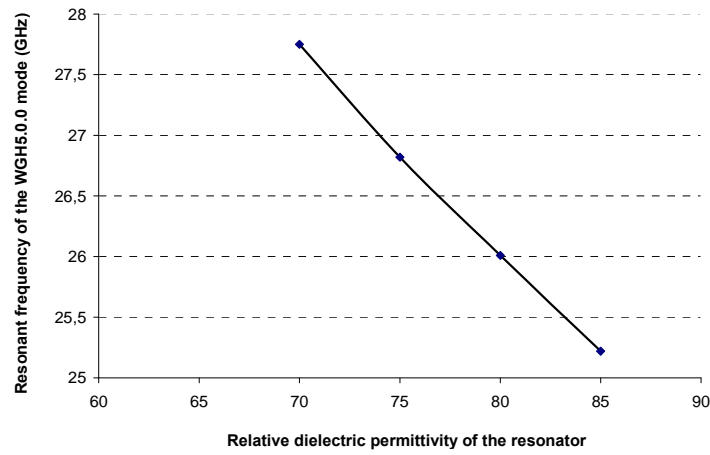
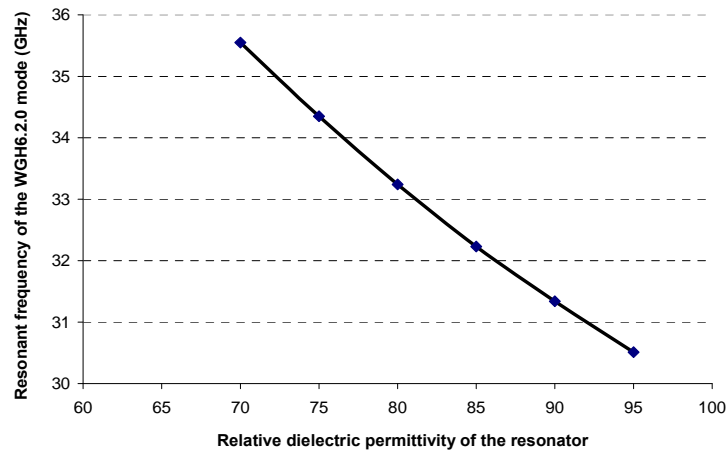


Fig. 4. Transmission coefficient from port 1 to port 3 versus frequency for various DR relative permittivities: (a) Permittivity $\epsilon_r = 75, 80$ and 85 ; and (b) Permittivity $\epsilon_r = 70, 80$ and 90 .

For example, as depicted in Fig. 5, the $WG_{5,0,0}$ resonant frequency decreases by 10 % when the TiO_2 dielectric permittivity increases by 20 %, due to the presence of humidity, while the resonant frequency of the $WG_{6, 2,0}$ mode decreases by 8 % when the DR relative permittivity increases by 30 %. Such attractive variability of WGM resonant frequency has been recently shown by simulation for other gaseous molecules such as ethylene gas for structures based on Tin dioxide (SnO_2) dielectric resonator [12].



(a)



(b)

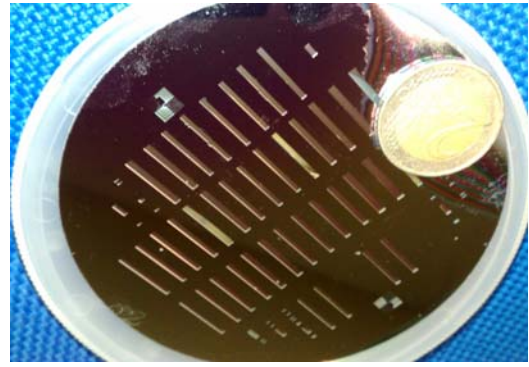
Fig. 5. Resonant frequency versus the DR relative permittivity of the: (a) WGH_{5,0,0} mode; and (b) WGH_{6,2,0} mode.

3. Fabrication Process of the WG M Resonator and Experimental Results

The substrate of the resonator is of High Resistive Silicon (resistivity higher than 3 K Ω .cm) with thickness of 400 μ m. On both sides of this substrate, were deposited thin layers of Silicon Dioxide (SiO₂, thickness: 0.8 μ m) and Silicon Nitride (SiN_x, thickness: 0.6 μ m) using standard thermal oxidation and Low-Pressure Chemical Vapor Deposition (LPCVD) process respectively. The metallization of the surface is then performed by Titanium/Gold evaporation (lift-off process). In order to minimize losses through coplanar lines, two processes were used for micromachining the CPW structure: (1) dry etching of the SiO₂/SiN_x membrane and (2) Deep Reactive-Ion Etching of the silicon substrate. Fig. 6 (a) and 6 (b) respectively show the top and bottom views of the resulting micro-machined CPW. Finally the alumina spacer and the dielectric cylindrical resonator (see Fig. 6 (c)) are mounted above the micro-machined surface. The experimental validation of the design and electromagnetic simulations was carried out using BaSmTiOxide ceramic as a dielectric material for the DR, as it is easier to fabricate by standard Sintering than the DR metal oxide: the dielectric property of this ceramic is not sensitive to gas concentration but its relative permittivity (78.6 \pm 2 from [17]) is very close to the one TiO₂. Consequently, the well-known and available BaSmTiOxide ceramic can be used for validation purposes of the WGM resonator design. The fabricated Whispering-Gallery mode resonator with its CPW excitation is depicted in Fig. 6 (d). Two coplanar test probes were used to measure the S13 transmission parameter of the gas sensor cell from the ports 1 to 3, with the ports 2 and 4 of the structure being loaded by 50 ohms



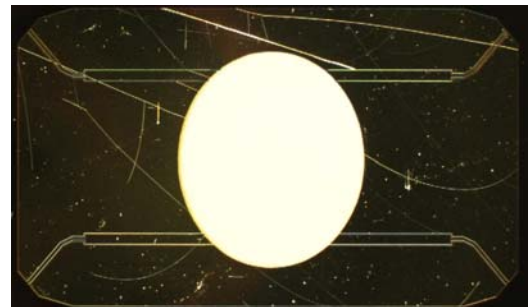
(a)



(b)



(c)



(d)

Fig. 6. (a) top and (b) bottom views of the fabricated micro-machined CPW; (c) the alumina spacer and the cylindrical dielectric resonator; (d) fabricated Whispering-Gallery mode resonator with its CPW excitation lines.

The measured transmission coefficient S_{13} for millimeter-wave frequency range is given in Fig. 7. As expected, the transmission peaks are observed at the resonant frequency of the $WGH_{5,0,0}$ and $WGH_{6,2,0}$ modes. Due to the technological inaccuracies and high-Q feature of the fabricated structures, the three resonators (with the same dimensions) micro-machined on the wafer do not have identical transmission peak magnitudes and, in a lesser extent, not the same resonant frequency. As illustrated in Fig. 8, the differences in peak magnitude may reach 7 dB. Keeping in mind this order of magnitude, the measurement and simulation results shown in Fig. 7 can be considered in good agreement. The slight difference (3.5 %) between the simulated and measured resonant frequencies is due to the fact that electromagnetic simulations have been performed using DR relative permittivity of 80 whereas measurements were done with the BaSmTi oxide dielectric resonator having an effective relative permittivity of 78.

We conclude that the measurements and simulation results of the S_{13} parameter are in good agreement and validate both the DR design, the simulation results and the technological process. The fabrication by 3-D stereo-lithography process of dielectric resonators with porous TiO_2 is under investigation at the Center for Technology Transfers in Ceramics Laboratory, Limoges, France. This process has been chosen for its ability to fabricate complex ceramic 3-D structures with required manufacturing tolerances for specific high frequency applications [18]. Pores in the TiO_2 -based resonator will be realized for insuring gas penetration inside the sensitive metal oxide.

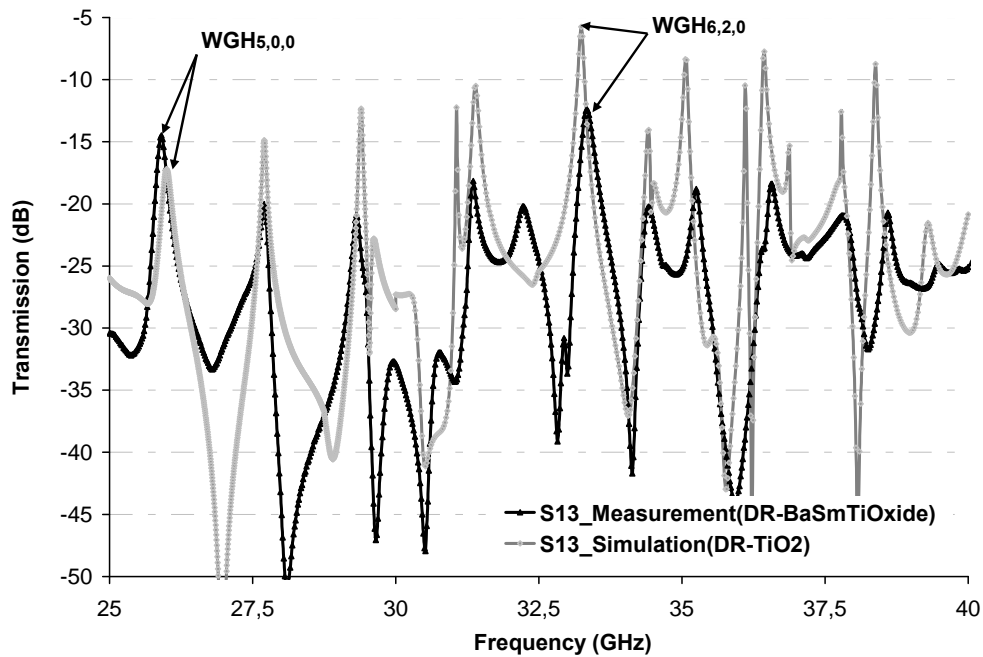


Fig. 7. Measured and simulated transmission coefficients versus frequency for the BaSmTiOxide dielectric resonator.

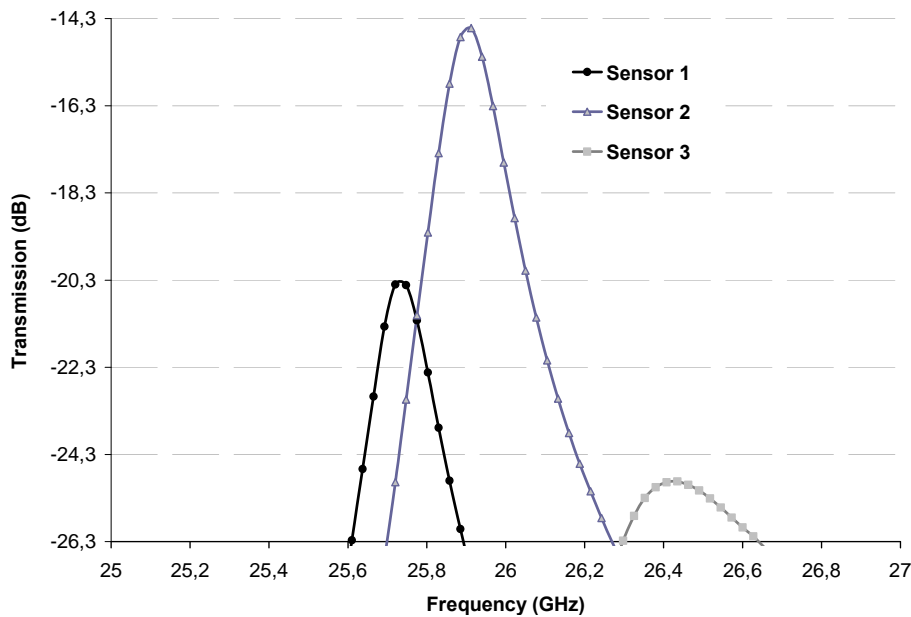


Fig. 8. Measured transmission coefficient versus frequency for three whispering-gallery mode resonators taken on the same wafer surface (these resonators share the same dimensions).

4. Wireless Interrogation of the Whispering-Gallery Mode Resonator

The experimental test set-up is described in Fig. 9. The port 3 of the resonator is loaded by 50Ω -impedance while port 1 is connected to a horn antenna via a delay line (here a coaxial line of 1m length). The resulting passive device is called here the Device Under Test (DUT). A Frequency-Modulated (FM) signal is incident upon the DUT (see Fig. 9) from FM continuous wave (CW) Radar. Within this system a Voltage Control Oscillator generates a frequency signal which increases linearly for a frequency sweep period by a 1 kHz sawtooth modulation signal [6]. This signal propagates from

the transmission horn antenna to the static gas sensor. The frequency band of the RADAR [25-27 GHz] includes the WGH_{5,0,0}-mode resonant frequency of the DUT. The signal back-scattered by the DUT is received by the RADAR and mixed with the transmitted signal.

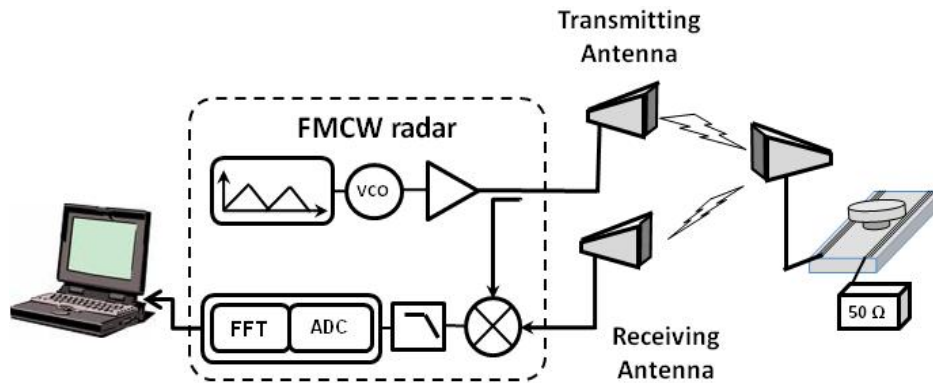


Fig. 9. Block diagram of the FMCW radar system.

The Intermediate Frequency or Beat Frequency is obtained by mixing the transmitted signal with the back-scattered signal. This step is performed by using A/D Conversion and Fast Fourier Transform by Hamming window at the mixer output. The resulting low-frequency spectrum is shown in Fig. 10. In this measured spectrum two peaks are of particular interest: peak No1 at 16.7 kHz corresponds to the structural scattering mode, i.e., to the echo of the DUT horn antenna: this beat frequency allows the remote derivation of the distance between the DUT antenna and the RADAR antenna (3 meters here). The peak No. 2 at 36.6 kHz corresponds to the antenna scattering mode, that is, to the echo depending on the DUT reflection coefficient. As this coefficient depends on the DR permittivity, variations in the peak No2 magnitude allows, at least in principle, the remote detection of variation in the DR permittivity due to fluctuations in gas concentration. This claim can be verified by measuring the magnitude of the peak No. 2 when loading the horn antenna by the three resonators considered in Section 3 (see Fig. 8). As a matter of fact, while their WGH_{5,0,0} mode does not resonate exactly at the same frequency, the frequency response of the three resonators can be viewed as frequency responses of a unique gas sensor in presence of three different gas concentrations.

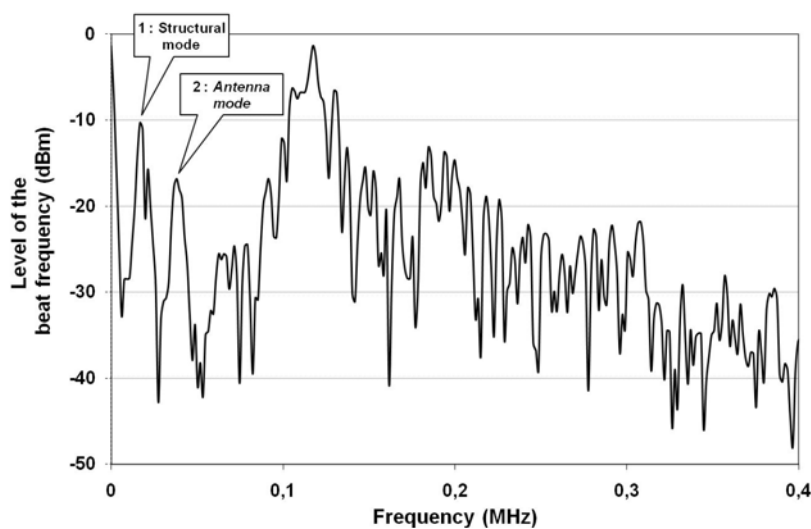


Fig. 10. Measured low-frequency spectrum resulting from the transmitted FM-signal mixed with the signal backscattered by the horn antenna.

Table 1 reports the measured magnitude of peak No. 2 when loading the horn antenna successively by the three resonators. As expected small variations in the resonant frequency of WGH_{5,0,0} mode modify significantly the magnitude of peak No. 2. This experimental result provides the proof-of-concept of remote measurement of gas concentration fluctuation from RADAR interrogation of passive gas sensors based on high-Q Whispering-Gallery modes. It concludes our feasibility study. The fabrication of dielectric resonators with porous TiO₂ is now under way for validating the remote measurement of gas concentration from RADAR interrogation of WGM-based sensors.

Table 1. Magnitude of peak No2 (antenna scattering mode) when the horn antenna is loaded by three resonators having the same dimensions.

	Magnitude of peak No. 2 (dBm)
WGM resonator No. 1	-16,9 ± 0.7
WGM resonator No. 2	-18,6 ± 1.2
WGM resonator No. 3	-20,2 ± 0.3

5. Conclusions

Theoretical and experimental investigations have been performed for studying the feasibility of passive gas sensors based on high-Q Whispering-Gallery modes. These investigations have been carried out by using BaSmTiOxide ceramic as a dielectric material for the DR: even if this ceramic did not present sensitivity to gas concentration, its dielectric permittivity is close to the one of TiO₂ and easy to fabricate by using standard stereo-lithography. The feasibility of the remote measurement of gas concentration from the FMCW RADAR interrogation of an antenna loaded by the passive sensor has been also studied. Work is now focused on a second generation of dielectric resonator based on porous TiO₂ dielectric resonator (one way is stereo-lithography) and the evaluation of the gas detection performances with a specific RF probe station with controlled atmosphere.

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