Microwave Technique for the Corrosion Detection

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ABSTRACT

We present in this paper a new passive ceramic corrosion sensor which can be integrated into a structure submitted to a corrosive environment. The corrosion detection mechanism is based on the modification of a resonator frequency as a function of the corrosion level. An alumina substrate is covered with a metal enclosure that is protected against corrosion apart from the middle of the top face. This latter, patterned as a grid, is made of a metal sensitive to corrosion. The cracks caused by the corrosion in the grid introduce a capacitive effect and cause a frequency shift of the resonance mode (f_r), and a degradation of unloaded quality factor (Q_0) as a function of the corrosion level.

Key words: corrosion; passive; sensor; ceramic; grid.

1. INTRODUCTION

Corrosion is the destructive attack of a metal and its properties by chemical or electrochemical reaction with its environment [1]. The corrosion of structures exposed to a corrosive environment leads to failures of mechanical systems and thereby to serious problems of security. The direct cost of corrosion in countries throughout the world is estimated to be between 3% and 4% of the Gross Domestic Product (DGP) of each country [2]. To avoid such risks, it is of first importance to be able to monitor the corrosion process of structures and so to predict their failure. Several methods (chemical and electrical techniques) can be used for corrosion characterization. Electrochemical impedance spectroscopy (EIS) is an electrochemical technique, which can be used to test corrosion rate [3]. However these electrochemical tests include the need for comparatively expensive equipment and high levels of technical expertise for data analysis [4]. It is also not an ideal tool for in-situ corrosion detection and monitoring. Another electronic active sensor developed by Luna innovation [5] a system designed to monitor the corrosion of an aircraft. This sensor is based on the classic sensor architecture composed of a part for the corrosion sensor and another part for the data transmission. Although it uses very low power components, a power battery should be integrated in the sensor. As the corrosion is a very long process (several years), the sensor should have a long lifetime. This is very restrictive for active sensors when they are integrated in the structure and inaccessible. In addition to its lifetime, the sensor must be economical to produce, maintain and install, since many sensors are needed to monitor any given structure effectively (bridge, aircraft, ship…). And since it is often difficult to have direct access to the sensor or to take it off, it is therefore interesting to develop corrosion sensors permanently attached to a structure which give remote information on the state of corrosion of this structure, by wireless techniques. For all this reasons there is a real interest to use passive sensors.

In this article, we present a new design for a passive ceramic corrosion sensor which can be integrated into the structure submitted to a corrosive environment, the corrosion detection being based on the modification of a resonator frequency in function of the corrosion level. The sensor has been fabricated and tested in our laboratory. The simulated and experimental results are presented and discussed at the end of this paper.

2. PRINCIPLE OF CORROSION DETECTION

The close-packed microstructure of metals dictates that electrons are shared among the constituent atoms. For this reason they are great electrical conductors. Most metals are thermodynamically unstable in atmospheric conditions. In order for them to maintain the metallic form they must absorb and store energy. If this needed energy is not supplied, the metal will revert to a lower energy state, such as a covalent-bonded metal oxide. This transition from a metal to a metal oxide exemplifies corrosion which leads to material loss and is often the precursor of fatigue cracking [6]. We were based on these consequences to build our new design for the corrosion sensor.

3. CORROSION SENSOR DESIGN AND SIMULATION

Our sensor is presented in figures 1.a and b. It is a 3D ceramic resonator, based on a ceramic substrate covered with metal. The corrosion detection is based on the modification of the resonance frequency of this resonator as a function of the corrosion level. The sensor is composed of 96%-purity alumina substrate (ε_r=9.4, tanδ=5*10-4) and has 0.5mm of thickness, 25.5mm of length, and 25.5mm of width. Alumina was selected for its low loss tangent which leads to a high unloaded quality factor and therefore a high selectivity of our sensor. A second reason of this choice is that this material is not sensitive to corrosion. This substrate is covered with a metal layer enclosure having a 5µm of thickness and protected against corrosion apart from the middle of the top face. This latter, patterned as a grid, is made of a metal sensitive to corrosion, having a 13mm of length and 13mm of width as shown in figure 1.a. The vertical and horizontal lines that compose the grid have a width of 250µm and are separated by a distance of 700µm. The grid form is selected to be easily degraded by the corrosion causing cracking in the grid. This crack introduces a capacitive effect and causes a frequency shift of the resonance mode (f_r), a and degradation of its unloaded quality factor (Q_0).
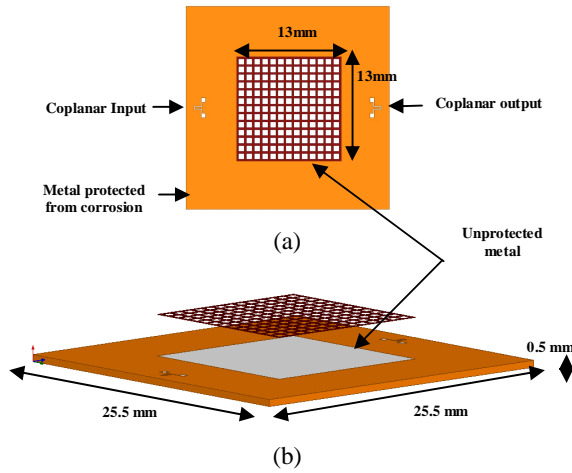
Figure 1: Sensor design (a) top view, (b) 3D view

With the increasing number of cracks in the grid, the frequency shift increases, allowing monitoring the level of corrosion. The metal chosen for the grid can be changed (zinc, aluminum, steel), depending on the studied corrosion mechanism. In our sensor we choose copper. The employed I/O excitation system is composed of short circuited coplanar lines placed on the top of resonator. The short-circuited lines allow concentrating the electromagnetic field in this point and consequently the coupling with the electromagnetic field of the TE_{101} resonance mode in the cavity. The TE_{101} mode has been selected because of its high Q_0, good frequency isolation from the higher modes and because the magnetic fields produced by the I/O coplanar lines are collinear with this mode magnetic field, making it easily coupled by such planar accesses.
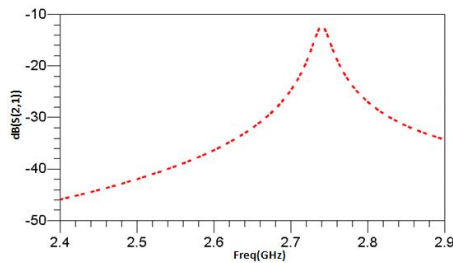

Figure 2: Transmission coeficient S_{21}(dB) simulated for non-corroded sensor

The sensor is designed and simulated with software Ansofts HFSS. The resonance frequency of the sensor when the grid is not attacked by corrosion is 2.74GHz, the unloaded quality factor is 165 and the level of S_{21} is -12.3 dB as shown in figure 2, we chose this level on purpose to keep a low coupling level in order to make the measurement (part V) easy. When the sensor is attacked by corrosion, cracks are formed in the grid. The cracks were modeled by slots in the grid. Corrosion can attack different areas of the grid and this is

why the distribution of slots was randomly chosen for the shown simulated results.

The number of cracks increases with time and this can be modeled by the increase of the number of slots as shown in figures 3.a, b, c and d.
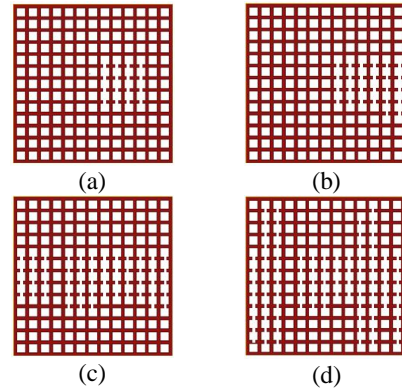

Figure 3: Different levels of the corrosion from low (a) to high (d), (a)=16 slots, (b)=26 slots, (c)=53 slots, (d)=91 slots.

Figure 4 shows the resonant frequencies of the simulated sensor that is not corroded and in the presence of the slots due to corrosion. Each curve shows one of the four cases shown in figure 3. The blue curve shows an 11MHz frequency shift with respect to the frequency of resonance of non-corroded sensor, and an unloaded quality factor of 160, this frequency shift and this degradation of quality factor are due to the presence of the 16 slots presented in figure 4.a. The pink curve shows a 31MHz frequency shift with respect to the resonance frequency of non-corroded sensor, an unloaded quality factor of 155 and a decrease in the level of S_{21} by 2 dB. The increase in frequency shift, the degradation in unloaded quality factor and the decrease in the level of S_{21} are caused by the increased number of the slots as shown in figure 4.b. Brown and black curves show a frequency shift of 52MHz and 275MHz respectively, an unloaded quality factor of 145 and 115 respectively, and a decrease in the level of S_{21} by 2.5 dB and 13.7 dB respectively. The observed frequency shift is linked to the creation of small capacitance at the surface of the resonator each time the grid gets a crack. Each crack increases the total of these added capacitances and therefore lowers the frequency of the TE_{101} mode. The loss in S_{21} level and in Q_0 is attributed to radiation losses (Q_0 starting from 165 for a non-corroded state and goes down to 115 with 91 slots).

$$\Delta f=3.6N-54 \text{ (1.a)} \qquad Q_0=-0.6N-171 \text{ (1.b)}$$

The relationships between number of slots (N) and the frequency shift Δf (MHz) on the one hand and the unloaded quality factor Q_0 on the other hand are given by two linear equations (1.a and 1.b).
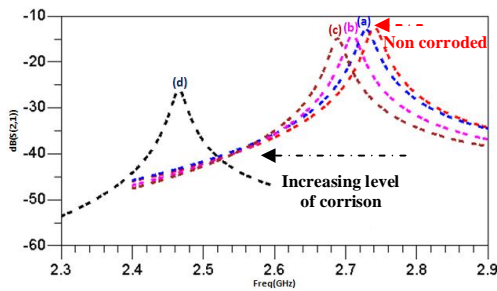
Figure 4: Transmission coeficient S_{21}(dB) simulated for corroded sensor

Thus the detection of the level of corrosion can be based on the change of frequency shift, degradation of unloaded quality factor, and the decreased level of S_{21}. As a summary, as the rate of corrosion increases, the frequency shifts increases, the degradation of Q_0 increases and the level of S_{21} decreases.

4. FABRICATION OF SENSOR

The fabrication of the sensor was held in our laboratory through two steps. The first step is the metallization of a blank alumina substrate; this step is achieved through an electroless copper plating. The second step is engraving the exciting patterns and finally the preparation of the grid by a laser ablation machine.

(a)   (b)

Figure 5: (a) Alumina substrate, (b) Alumina substrate by copper electroless plating.

A blank alumina substrate (25mm*25mm*0.5mm, ε_r=9.4, $\tan\delta$=5*10^{-4}) is plated by electroless copper plating (figures 5.a and b). This process requires the use of different chemical solutions to obtain the desired thickness of the copper layer (5µm in our case). The substrate to be metallized was immersed successively in three chemical solutions: the first one is a conditioner, used to "super-wet" the dielectric material, and to provide this area a uniformity of polarity such that the later adsorption of activator will be facilitated. The second solution is the catalyst which allows the activation of dielectric surface before the deposition step and finally the third solution is dedicated the electroless copper layer growth. The second step is the realization of the excitation system. For this purpose we used a laser ablation machine using a λ=1µm laser beam. This latter can etch the coplanar line based excitation system on the surface of the metalized substrate in an extremely flexible way. This technique was also used to achieve the grid as shown in figure 6.
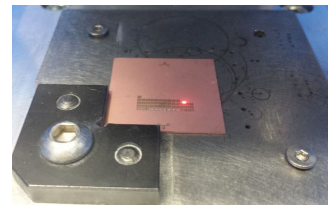

Figure 6: Etching of excitation system and grid by a laser beam
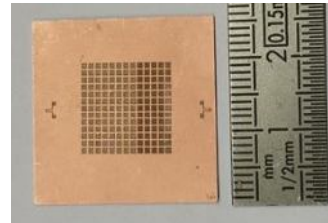

Figure 7: Ceramic Corrosion sensor after fabrication

Figure 7 shows our sensor after the metallization and the etching of exciting system and the grid.

5. MEASURMENT RESULTS

After being fabricated, the experimental investigation of the sensor is performed at Xlim laboratory utilizing a Rohde and Schwarz ZVA 24-4 VNA. ACP40AGSG125 RF probes that are compatible with the I/O coplanar lines (250µm) are used for the structure measurement. A SOLT (Short-Open-Load-Thru) full 2-port calibration technique is performed using ISS 101-190 Cascad Cal Kit. The resonance frequency is found to be 2.74GHz, the measured Q_0 is 169 (theoretical Q_0 is 165) and the level of S_{21} is 12.2 dB as shown in figure 8.
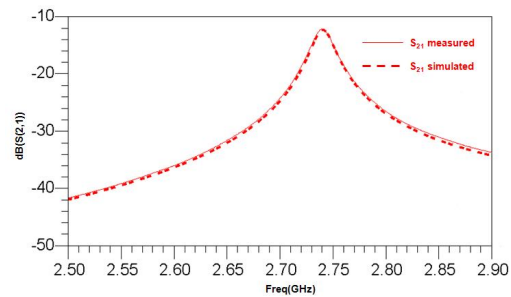

Figure 8: Measured results versus EM simulation of transmission coeficient S_{21}(dB) of non-corroded sensor

The use of salt spray chambers or thermal climatic chambers is the best solution to test this type of sensors. This type of testing being not available at the moment in our laboratory, a first demonstration step was here performed using our ablation laser to create cracks in the grid of our sensor, mimicking the expected effect of corrosion. Cracks in the grid are engraved in the same place as simulated, which allows us to validate our EM model. Figure 9.a, b, c and d shows a portion of the

engraved grid, the number of the slots and their position are similar to the simulated cases (Figure 3.a, b, c and d).
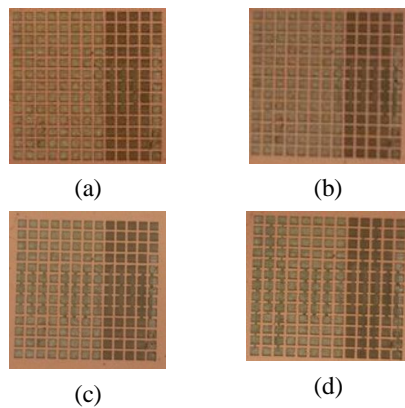

(a)   (b)

(c)   (d)

Figure 9: Grid engraved by a laser beam is order to reproduce figure. 3a ,b ,c and d cases

Figure 10 shows the measured resonance frequencies of the sensor with and without cracking. Each curve shows one of the cases shown in figures 3.a, b, c, and d. The blue line shows an 11MHz frequency shift with respect to the resonance frequency of the sensor without cracking and an unloaded quality factor of 162. This frequency shift and this degradation of quality factor are due to the presence of the 16 slots as predicted by simulations.
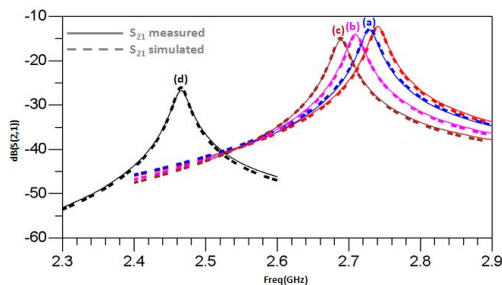

Figure 10: Measured results versus EM simulation of transmission coeficient S_{21}(dB) of corroded sensor for different corrosion states

The pink curve shows a 31MHz frequency shift with respect to the resonance frequency of the non-corroded sensor and an unloaded quality factor of 155 and a decrease in the S_{21} level by 1.7 dB. The increase in frequency shift, the degradation of Q_0 and the decreased level of S_{21} are due to the increased number of the slots. The black and brown curves show a shift of 52MHz and 275MHz respectively, an unloaded quality factor of 147 and 117 respectively and a decrease in the S_{21} level by 14.5 dB and 3.1 dB respectively. These changes are also a result of the increase of slots number. To be noted that the simulated and measures values are very close, and that these measurements confirm the behavior of this sensor once the grid is more and more getting cracks.

6. CONCLUSION

In conclusion, we proposed a new model of a ceramic-based passive resonator acting as a sensor for the detection of corrosion. The monitoring of the level of corrosion is based on the change in the resonant frequency of the sensor due to the presence of cracks caused by corrosion in a metallic grid acting as the sensitive part of this sensor. The increase of the corrosion rate increases the number of cracks which increases the frequency shift of the ceramic resonator TE_{101} mode frequency and causes a decrease in the level of S_{21}. The sensor was fabricated in our laboratory using a flexible manufacturing process. The measurement of non-corroded sensor shows a very good agreement with EM simulation. Testing this type of sensor in good conditions in salt spray chambers or thermal climatic chambers is not available at this time in our laboratory. Thus we used in a first stage a laser to create the cracks in the grid of our sensor just as the corrosion is expected to act. The obtained results are promising with a significant and measurable change in frequency (from a few MHz for a minor corrosion to more than 250 MHz for 91 slots in the top grid) and in good agreement with the simulations. In a future work we will select specific metals which are sensitive to corrosion for the fabrication of the grid which is the sensitive part of this sensor, and we will optimize the pattern given to the grid in order to precisely monitor the sensor carrion level. The sensor next generation will be tested in salt spray chambers to validate the approach.

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