

A Review of Characterization Techniques for Materials' Properties Measurement Using Microwave Resonant Sensor

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Abstract— This paper presents a compilation of important review in the development of microwave resonant sensor technology used in previous years. The major research work for each year is reviewed. Most of the resonators are designed for material characterization in specific application areas such as food quality control, medical, bio-sensing and subsurface detection. In the last few years, several resonant sensors based on the planar and non-planar structure are compared and examined in order to propose a new topology of microwave sensors designed. The weaknesses of conventional sensors such as bulky size, high cost manufacturing and consume high volumes of detectable sample have been reviewed. Most significantly, this new proposed structure must gain high quality factor to gain improvement in an accuracy of the sensing capability and can overcome previous design weaknesses. This device will discriminate the composition and properties of samples based on scattering parameters in certain operating frequency. The proposed system outlined in this paper, featuring new innovation in resonator structure as well as providing advanced capability design of future research works. The contribution of this study is useful for various types of applications where the characterizing of materials is very important, while improving its performance especially in terms of accuracy and sensitivity. The previous studies will be reviewed and critically compared in order to gain a better understanding in microwave resonant sensors and new ideas for further research improvement in application, which require characterizing of materials.

Index Terms— Accuracy, Characterization, Capability, Methods, Microwave, Resonators, Resonant, Sensitivity, Splitting, Sensor, Techniques.

I. INTRODUCTION

Accurate measurement of material characterization has gained significant importance over the last decade. The ability to analyze composition and properties of a material which undergoes physical and chemical changes has led to many applications in the industry such as food quality control, bio-sensing and subsurface detection. In the food industry, the research interest in dielectric properties of food materials has been growing, in which there has been an intensive research for predicting heating rates when the materials are subjected to

high frequency electric fields. Nevertheless, the subsurface detection of samples will affect the properties of materials when exposed to extremely high electric field distribution [1]. Other than that, the moisture content of the materials and its effect on the dielectric properties of materials also can be determined by using near-field sensors, which are highly sensitive. However, high lossy materials decrease the quality factor of the resonator drastically and at the same time reduce the measurement accuracy of microwave sensor [2]. On the other hand, microwave techniques for drying food products have also been very popular since the percentage moisture content of materials can be classified, although the permittivity of the samples will change as well as the water content of samples varies accordingly [3]. Those problems have been studied extensively in order to gain some knowledge for future research works.

Basically, the material characterization structure designs use mostly resonant methods, in which these methods can be classified into resonator methods and resonant perturbation methods. Resonant methods offer the potential of characterizing the properties of a material at a single frequency or a discrete set of frequencies with high accuracy in comparison to broadband methods. Conventionally, waveguide, dielectric and coaxial cavity resonators have been used for characterizing materials but in different topologies. Most of the methods and techniques applied are based on the applications used by the industry. A wide category concerning the dielectric properties of materials can be characterized into the resonator as transmission, reflection or absorption elements. Wideband permittivity measurements are mostly made of transmission sensors or reflection sensors, such as coaxial probes. However, this type of measurement is frequently overly complicated for industrial applications [4]. In this case, planar resonant sensors are taking place as the current complex permittivity measurements.

Sensors require a scalar measurement that allocates less cost and more robust instrumentation to be employed; resonant sensors can be fully described in terms of scalar measurements so that they provide themselves to industrial applications, whenever wideband characterization is not required. A typical

application of resonant sensors has been presented in the previous work [5]. In RF and microwave circuit design, the dielectric permittivity of the substrate plays an important role and requires precise evaluation over a broad range of frequencies. Knowledge of these properties plays a crucial role in the accurate design of a variety of circuit technologies. The following sections aim to provide a general discussion of some well-established dielectric measurement techniques, primarily applicable to moderate and high loss liquid samples and even for solid and gas samples [6].

Moreover, by introducing an accurate equivalent circuit model for the planar sensor, the experimental calibration procedure is avoided. The low cost and simplicity of manufacturing allows the use of the resonator in a disposable manner [7] and an effort has been made to present a recent technique on several phases of materials such as solid, liquid and mixture. To satisfy the current demands of many applications especially the biological and chemical industries, a mixture technique can be implemented by using planar resonant sensors developed by various researchers. In this paper, an effort has been made to describe and show the interesting advances in microwave resonator sensor developments. The structure to produce highly sensitive sensors is shown with some comparisons based on frequencies, methods, techniques, and technologies used. It is expected that the information from this paper will help researchers to have a broader perspective of resonator structure, and hopefully it can be considered as an alternative to produce a better technique design.

II. TYPE OF MATERIAL SAMPLES

A. Characterization of Solid

Several studies have been conducted on solid samples for materials characterization in order to introduce novel resonant sensor design with the improvements in accuracy and sensitivity of the device [1, 3, 4 and 8]. For example, [1] produced microwave planar resonant sensor based on the adoption of a scalar two-port measurement method. There are similarities between both papers in term of frequency. The operating frequency is 500-800 MHz on 2004 and the first paper produced on 2002 used 656.58 MHz as the operating frequency. The improvements in Q-factor, accuracy and robustness have been made. The method used is suitable for the detection of moisture content in wet powdered materials [1].

[4] designed a Cross-Shaped microstrip ring sensor with full planar microstrip technology. A scalar network analyzer was used to compute the resonant frequency and the matching amplitude of the transmission parameter for the fundamental mode. The complex permittivity was estimated by inverting the measured parameters using an algorithm based on Artificial Neural Network (ANN). This design was modified based on their research work in 2001 "full-wave modelling of microwave planar reflection sensors of material moisture testing". The design structure has three layers of substrate with different dimension specifications. The upper and second layers used RO4003 substrate and the bottom layer used FR4 materials. Each layer has its own function, which are input, hidden and output port of the sensors. The sensor was very

tough because it was a multilayer structure and can be easily produced by less expensive printed circuit technology. The relative error of the system was very small, which was less than 3% based on the test results in several samples [4]. Table 1 shows the comparison of the previous research works in solid materials characterization due to different types of methods and techniques.

Table 1
Comparison of previous research works in solid materials characterization

| No | Title/Author/Year | Remarks |
|------|--|--|
| 1[1] | A Planar Resonant Sensor for the Complex Permittivity Characterization of Materials. Elisa Fratticcioli, 2002 | The resonator was designed at frequency 656.58 MHz. Solid samples were used. The advantages are reducing costs, improving robustness and have excellent accuracy. Low cost manufacturing since it uses planar structure and easy to fabricate. |
| 2[3] | A Simple and Low-Cost Measurement System for the Complex Permittivity Characterization of Materials. Elisa Fratticcioli, 2004 | The resonator was designed at frequency 500-800 MHz. Solid samples were used. The advantages are less fabrication cost, robustness improvement and can be disposable. Suitable for measurement in compact areas. |
| 3[4] | Microwave permittivity measurements through cross-shaped ring sensors. Biffi Gentili, 2002 | The resonator sensor was designed at frequency 883 MHz. Solid samples were used such as Teflon, R4003, FR4, RF35 and glass. The advantages are high accuracy and extremely compact. Since the size is compact, the fabrication cost is less expensive. |
| 4[8] | Planar Resonator Sensor for Moisture Measurements. Jerzy Skulski, 1998 | The resonator sensor was designed at frequency 2.4 GHz. Solid samples were used such as grain, sand and concrete. The advantages are a highly sensitive detector of moisture contents in solid. Other than that, time measurement will be very short and able to measure practically on-line. The fabrication cost is cheaper since the resonator is planar based. |

B. Characterization of Liquid

The characterization of materials does not only focus on the solid phase, but also all of the phases such as liquids, gases and mixtures. Several studies on liquid materials characterization have been conducted and have been discussed in succeeding paragraphs [2, 7, 9, 10, 11, 12 and 28]. For example, [2] designed a split-ring resonator by combining the dielectric resonator coupling for accuracy enhancement. This resonator was designed in simulation software before coming up with fabrication process. The sensor was simulated using the 3D full-wave solver Ansys High-Frequency Structure Simulator (HFSS). The complex permittivity of water and saline solutions was modeled using a first order Debye model. The advantage of this filter over the resonant sensor presented by [1] was that its sensitivity was much better due to high Q-factor. However, this design had limitations in bandwidth

which operates at 20-40 GHz frequency range. Moreover, the Q-factor was still relatively low for a DR resonator, which was due to radiation. The high performance resonant sensor could be produced using this method with some modifications on its basic design. Other parameters, such as bandwidth and frequency ratio also varied accordingly [2].

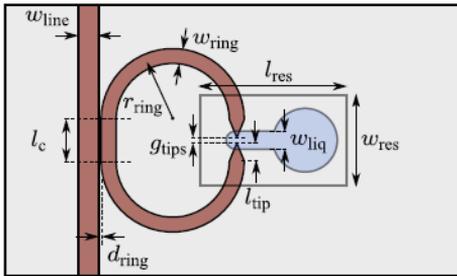


Figure 1: Geometrical parameters of the coupled DR-SRR sensor [2]

Another example is the integration of waveguide cavity resonator designed by [7] presented for Pharmaceutical industry application. The simulation results were obtained using the Full-wave finite-element method (FEM)-based High Frequency Structure Simulation (HFSS) commercial software package. The resonant frequency of the sensor was 8 GHz and substrate material used in fabrication process was RT/Duroid with dielectric constant 2.2 and loss tangent 0.00009. The high-resonant construction was a modernization of well-known measurement cells where the dielectric constant was deduced by cavity perturbation from the shift in resonant frequency and the change in the factor. The advantages of the sensor are low cost and it can be easily integrated with many other components. The error in the measured results was within plus minus 0.5% and this proves that the resonant sensor is much better in terms of sensitivity and accuracy [7]. Figure 2 and 3 show the structure of resonator sensor for liquid characterization.

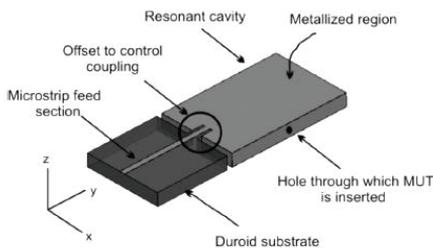
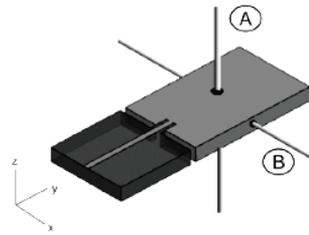


Figure 2: Layout of substrate integrated waveguide cavity resonator with microstrip feed section and hole within the cavity through which the MUT is inserted [7]



A - Capillary through broad walls of the waveguide (along z-axis)
B - Capillary through side walls of the waveguide (along x-axis)

Figure 3: Location of capillary and MUT inside the cavity [7]

[9] introduced a new idea for designing microwave sensor for precise permittivity characterization of liquids used for aqueous glucose detection in medical applications. This idea was based on the planar half-wavelength microstrip line (MSL) resonator. The resonator was a highly sensitive sensor for precise permittivity change detection of liquids present. The resonant frequency was 2 GHz using near-field sensor. A very sensitive concentration less than 0.01% was detectable. Even small mixture content can be detected for measuring purposes. The sensor was also applicable to other liquid mixtures independent of small, medium or high loss. A simple and highly sensitive microstrip sensor structure has been presented [9].

[10] proposed a new method for wide range applications by designing a microwave microfluidic sensor using a microstrip split-ring resonator based on perturbation theory. The resonant frequency and Q-factor depends on dielectric properties of resonator, which was the operating frequency at 3 GHz. The design used Rogers Corporation RT/Duroid 5880 laminate substrate for fabrication process, which had the permittivity at 2.2 and lost tangent at 0.00009. It was modified from [7] research work. COMSOL Multiphysics software has been used to perform the simulation of electromagnetic properties of the structure. Several common solvents were chosen for testing purposes such as hexane, chloroform, ethanol, methanol and water. The resonator was compact and planar, making it suitable for a lab-on-a chip approach and suitable for evaluation of the material properties [10].

[12] presented the concept of microwave resonator using micro fabricated sensor referring to liquids characterization. The design was compatible with a future lab-on-a-chip integration for convenience used. Some solvent has been tested such as de-ionized water-ethanol mixtures, which had the volume of ethanol ranging from 0% - 20%. The resonant frequency used was 20 GHz and standard match impedance 50 ohm. The associated transmission coefficient and maximum relative error were less than 0.1% and 6.5%, respectively, which was a highly accurate permittivity characterization. The dimensions of the design structure were presented in details, including procedures fabrication process. The microfluidic area was attached to the top surface of the structure to convey the liquid toward the sensing region. Simulated process for determining frequency and attenuation was done using High Frequency Structure Simulator (HFSS). The effective and accurate complex permittivity characterization has been done successfully in applications area [12].

[28] constructed a flow-type liquid sensor by using a 2 port SH-SAW resonator. SAW devices were used as sensor in a variety of fields. The waves propagating in this method were parallel between the surface and particle displacement, which means the sensor was suitable for sensing the liquids characterization. There are a lot of advantages of this kind of sensor, which is very effective due to the small size, highly stable operation, and high signal-to-noise ratio. The amounts of liquid during measurement were very small. The sensitivity of the sensor can be increased based on the operating frequency of the sensor. The resonant frequency of the SAW resonator was 40 MHz and insertion loss of the sensor was taken to be 60 dB with Q-factor 400 [28]. The summary of sensing techniques for liquid measurement is shown in Table 2.

Table 2
Comparison of previous research works in liquid materials characterization

| No | Title/Author/Year | Remarks |
|-------|---|---|
| 1[2] | Accuracy Enhancement of a Split-Ring Resonator Liquid Sensor Using Dielectric Resonator Coupling. Nora Meyne, 2014 | The resonator sensor was designed at frequency 20-40 GHz. Liquid samples were used such as water and saline solutions. This technique will improve Q-factor of the design and enhances accuracy of the sensor. However, the fabrication cost is quite expensive since the materials used are roger type. |
| 2[7] | Substrate Integrated Waveguide Cavity Resonators for Complex Permittivity Characterization of Materials. Kashif Saeed, 2008 | The resonator sensor was designed at frequency 8 GHz. Liquid samples were used such as ethanediol, acetone, methanol and dimethyl sulphoxide. The advantages are easily integrated with many other components and less expensive manufacturing cost. |
| 3[9] | Microwave Sensor for Precise Permittivity Characterization of Liquids Used for Aqueous Glucose Detection in Medical Applications. U. Schwerthoeffer, 2014 | The resonator sensor was designed at frequency 2 GHz. Liquid samples were used such as water and glucose solvents. The advantages are very sensitive to concentration with less than 0.01% detectable sample. Other than that, the structural design is simple and highly sensitive sensor structure. |
| 4[10] | Novel Microwave Micro fluidic Sensor Using a Microstrip Split-Ring Resonator. Ali A. Abduljabar, 2014 | The resonator sensor was designed at frequency 3 GHz. Liquid samples were used such as hexane, chloroform, ethanol, methanol and distilled water. The advantages are suitable for a lab-on-a chip approach and easily integrated with other components. |
| 5[12] | A Microwave and Micro fluidic Planar Resonator Efficient and Accurate Complex Permittivity Characterization of Aqueous Solutions. Thomas Chretiennot, 2013 | The resonator sensor was designed at frequency 20 GHz. Liquid samples were used such as water and ethanol mixtures. The advantages are efficient techniques for high Q-factor resonator and have high accuracy on permittivity characterization of aqueous solutions. It is also suited to a lab-on-a-chip approach |

C. Characterization of Mixture

Mixtures sample is the materials, which have different states of matter such as solids, liquids and gases. Since the phases are not fixed, the technique used for device structure must be applicable and have an ability to characterize variation of samples. Several studies have been carried out on the mixture samples [6, 13, 22, 23 and 45]. For example, [6] developed the complementary split-ring resonators (CSRRs). The design was constructed such as high measurements in sensitivity and accuracy, while at the same time eliminated the extensive sample preparation procedure needed. The sensors were working on a 0.8 – 1.3 GHz band of frequency. Several samples have been tested for performance results, which were on air, Teflon, RO3003 and FR4. The fabrication process was less expensive and easy to process with a very low loss. Minimum transmission and minimum reflection were observed depending on the permittivity of the sample under test. The structure was fabricated using printed circuit board technology [6].

[13] introduced a compact resonant near-field sensor for liquid characterization. It had a very low fabrication cost and the technique was based on a folded substrate-integrated half-mode resonator with a planar sensing tip. Several samples such as saline, ethanol, salt, sugar and water solutions can be discriminated by the sensor with a change of concentration of less than 1%. Besides lower cost, the advantages of the sensor were the simplicity of the structure and easy access to the tip from the top, which means that the construction of the sensor can be done in a shorter time compared to other resonant sensors, especially in biological and chemical application area. The resonant frequency was 5 GHz for test process using HFSS software in the simulation process [13].

[23] developed a gas-liquid separated micro-resonator for measuring the multifunctional characteristics of biological cells. Two resonator regions which are gas-phase and liquid-phase areas were used to separate from water-shedding wall and resonate the samples in the chamber at around 2.5 kHz. It proved that water-shedding wall can avoid leakage between both separated areas, as shown in Figure 4.

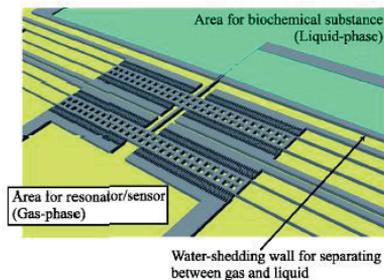


Figure 4: Schematic view of gas-liquid separated resonator [23]

The structure was developed using a micro electromechanical system (MEMS) technology. Silicon-on-insulator (SOI) wafer was used for fabrication process and the structures were formed using deep reactive ion etching (DRIE). Based on the experimental results, the performance of the sensor was very good and attempts were made to refine the

gas-liquid separated resonator [23]. Table 3 shows that the summary of previous studies in the mixture phase (solid, liquid, gas) characterization of resonant sensors.

Table 3
Comparison of previous research works in mixture materials characterization

| No | Title/Author/Year | Remarks |
|-------|--|--|
| 1[6] | Material Characterization Using Complementary Split-Ring Resonators. Muhammed Said Boybay, 2012 | The resonator sensor was designed at frequency 0.8-1.3 GHz. Mixture samples were used such as air, Teflon, RO 3003 and FR4. The advantages are high measurement sensitivity, less expensive fabrication cost, easy process of fabrication and very low loss detection structure. |
| 2[13] | Substrate-Integrated Half-Mode Resonant Near-Field Sensor for Liquid Characterization. Nora Haase , 2013 | The resonator sensor was designed at frequency 5 GHz. Mixture samples were used such as saline, ethanol, salt, sugar and water aqueous solution. The advantages are compact in size and act as resonant near-field sensor and have very low cost fabrication process. It is easy access to the tip from the top of sensor and also have the simplicity of structure. |
| 3[22] | A Circular Patch Resonator for the Measurement of Microwave Permittivity of Nematic Liquid Crystal. Diminic E. Schaub, 2011 | The resonator sensor was designed at frequency 4.8-8.7 GHz. Mixture samples were used such as nematic liquid-crystal mixture. The advantage of the design is simple resonator construction, but have quite expensive materials for the fabrication process. |
| 4[23] | Gas-Liquid Separated Resonator for Bio-Chemical Application. H. Hida, 2009 | The resonator sensor was designed at frequency 2.5 kHz. Mixture samples were used as the separation of gas from liquid. The technique used were most famous biochemical analysis – Quartz crystal microbalance (QCM) resonator. Yet, the cost is expensive during the manufacturing process. |

The separation of samples into 3 classification phases, namely the solid, liquid, and mixture are important for reviewing previous research works before proceeding to designing stage. Different types of phases can lead to different techniques to gain high quality. The accuracy and sensitivity of the resonant sensors depended on the Q-factor, while at the same time improved the performance of the device.

III. CONCLUSION

In this paper, an effort has been made to examine and discuss some of the advanced technologies in resonator sensor designs for many applications which are related to characterizing materials such as medical, food safety and

quality control, bio-sensing and chemical products. The advantages and disadvantages of the various techniques have been highlighted and thorough comparisons among various techniques have been presented. The focus of the discussion has been on the performance of each technique to seek the best sensitivity and accuracy for better measurement. This analysis presented in paper has been drawn from specific design sensor details and measurement results of each of the sensors. This has led to the tremendous development in both theoretical sensor design techniques and in the industry technology used. As a result, the resonant cavity method is known as the precise technique in the resonator sensor design for materials characterizing. However, the complexity of the manufacturing process leads to the high cost of fabrication, even though the sensitivity could be higher. Thus, the planar resonator sensor is proposed in this study. This technology can be used to enhance the weaknesses of conventional resonator design due to the planar structure. Further, it can be easily integrated with other components or devices. It is less expensive and consumes minimum volume of detectable samples. The design structures also are simple and easy to fabricate, although they can achieve higher Q-factor with certain techniques. The device can be expanded based on its achievement. Although the research in this area is not at its infancy stage, the research is still ongoing. Hopefully, the challenges faced by the researchers can be overcome and improved in order to gain advancement and improvement in this technology.

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