Australian Journal of Basic and Applied Sciences, 9(1) January 2015, Pages: 76-85

AENSI Journals

Australian Journal of Basic and Applied Sciences

ISSN:1991-8178

Journal home page: www.ajbasweb.com

Comparative Study of Materials Characterization using Microwave Resonators

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ARTICLE INFO

Article history:
Received 19 September 2014
Received in revised form
19 November 2014
Accepted 22 December 2014
Available online 2 January 2015

Keywords:
Microwave Resonators, Material characterization, Dielectric measurement techniques.

ABSTRACT

In the last few years, several designs of the microwave resonator, which has satisfied various objectives, have been proposed for materials characterization. Special attention is devoted to resonance techniques that are more accurate and sensitive compared to the transmission-reflection methods. Several specific methods for the materials measurement are described. Some of them are new concepts, whereas others are inspired from the previous works. This compilation of studies can change those designs towards the development and enhancement of materials characterization. This paper begins with a review of the common characterization techniques using different resonator designs at various frequencies followed by an explanation of the microwave resonant methods. Furthermore, a discussion of various designs in term of measurement techniques, performance, and application use. This paper is concluded with a comparison between the advantages and disadvantages of previous works for the important used techniques. Finally, designs can be developed to produce accurate resonator with high Q-factor and good performance in term of return loss, permittivity, and resonant frequency.

To Cite This Article: A.A. Rammah, Z. Zakaria, E. Ruslan, A.A.M., Isa Comparative Study of Materials Characterization using Microwave Resonators. ***Aust. J. Basic & Appl. Sci.,*** *9(1): 76-85, 2015*

INTRODUCTION

Material characterization techniques have been developed in recent years. There are many techniques available for the measurement of the real part of the complex permittivity (dielectric constant). However, each of these techniques is useful for certain kinds of materials and certain frequency range. Cavity techniques, and transmission line techniques are the two most commonly used techniques for measuring material properties at microwave frequencies. The cavity technique involves modeling a cavity in some geometry with boundaries of finite conductivity filled with the materials under test. This method is used to relate the measured transmission and reflected signals of the cavity to the characterized materials from which it was constructed. On the other hand, the transmission line techniques use an assumed model of the response of the dielectric materials, which filled in transmission line section. Then, either use time or frequency domain measured techniques in order to match the response of the real transmission line to the model.

Permittivity is considered as an important material characterization used for electrical engineers. The permittivity of materials effects the response of a material to electric signals. Therefore, precise permittivity determination is important for microwave engineering. Furthermore, materials characterization or detecting changes in the electrical properties of materials has applications in areas such as food industry, subsurface detection, quality control, or bio sensing (Nelson *et al*., 2007), (Nelsdon *et al*., 2008), (Guo *et al*., 2007), (Nelson and Trabelsi, 2009), (Dalmay *et al*., 2008), and (Kim *et al*., 2008). Most of the application of the sample under test carry valuable information such as material composition, water content, or moisture. Several methods have been proposed to characterize materials. Certain method can be categorized as near-field sensors, transmission-line method, resonant cavity methods, and free-space methods (Kinayman and Aksun, 2005). In near-field sensors, the open-ended coaxial lines are the most common near-field sensor used for materials characterization (Tabib *et al*., 1993), (Ganchev *et al*., 1995), (Stuchly *et al*., 1982), and (Athey *et al*., 1982). The material properties of the sample under test can be extracted using the measured reflected coefficient from the open end when the sample under the test is placed at the right in the opening. That is due to the response of an

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open-ended coaxial line structure, which can be obtained analytically (Misra, 1987., Baker *et al*., 1994), and (Bakhtiari *et al*., 1994). In addition, this method can be used for extracting material properties because of the method is not limited by the diffraction limit. Irregularities on the sample surface deteriorate the accuracy of the extraction even though the near-field sensors provide an inexpensive method for materials characterization. On the other hand, the modeling for the measurement of the reflected coefficient as a function of the dielectric properties remains a difficult task (Gabriel and Grant, 1994).

Transmission-line method is another method for materials characterization. The sample under test is employed as filling materials for transmission lines in this method. For instance, a slab of material can be inserted to a waveguide (Erntok *et al*., 2007), and (Akhtar *et al*., 2006), or the insulating materials of a coaxial line can be replaced by the sample under test (Jones *et al*., 2000). The transmission and reflection from the sample-filed region provides the needed information to extract the materials properties. The cost of this method is considered cheaper compared to free-space methods due to the elimination of the used lenses. However, the sample preparation remains as a challenging task. Stripline and Microstrip-line structures can be employed using this method (Barry, 1986), (Yue *et al*., 1998), and (Queffelec *et al*., 1994). Among the characterized techniques, the resonant cavity technique is the most accurate method. The cavity resonator is filled with the sample under test and the shift in the resonance frequency and the change in the quality factor are measured (Baker *et al*., 1998), and (Milovanoic *et al*., 1998). Furthermore, partial filled resonators can be used to characterize material properties. Not only the conventional box resonator used for this purpose, but also the Microstrip-line resonators and circular resonators (Shimin, 1986), (Itoh, 1974), and (Bernard and Gautray, 1991). It is considered as the most precise characterization techniques even though the resonant cavity method is applicable over a narrow band. This method also needs a precise sample preparation. The classical bio-static measurement setup consists generally of a pair of spot focusing lens antenna connected to a vector network analyzer (VNA) (Ghodgaonkar *et al*., 1990), (Kadaba, 1984), (Khosrowbeygi *et al*., 1994), and (Aris and Ghodgaonkar, 2004). The free-space reflection and transmission coefficients of the sample placed between the antennas are measured with this technique. Being a non-destructive and contactless is, its advantages, but it uses expensive lenses, horn antennas, and needs a large sample.

In the microwave resonator design, the permittivity of the dielectric substrate plays an important role and requires precise evaluation over a broad range of frequencies. In this paper, findings and analysis of the recent development for material measurement techniques are discussed and various factors are considered in order to obtain an appropriate technique, which provides high accuracy, low cost, easy procedures, and rapid measurement for the desired testing material.

Overview of the Microwave Methods for the Electromagnetic Materials Characterization:

A. Non-Resonator Methods:

In non-resonant methods from the material's impedance and wave velocity, the properties of an electromagnetic such as permittivity and permeability can be derived from the partial reflection of an incident wave from the interface between two materials. The reflected measurement can provide an information for the deduction of permeability and permittivity relationships between the two materials. In non-resonator methods, the transmission line is needed to direct the wave to the material under test. Then, collect the reflected and transmitted energy. Metallic waveguide, dielectric waveguide, free space, planar transmission line, and coaxial can be used as a transmission line. The reflection and transmission methods can be mainly included in the non-resonator methods. The properties of the materials are deduced from the phase and magnitude measurement of the signal reflections for the reflection methods. However, the properties of the materials are deduced from measuring the phase and magnitude of both transmitted and reflected signals in the transmission methods. Furthermore, the results from both transmitted and reflected measurements are preferred to be used in order to improve the confidence in the findings.

1. Coaxial Transmission Line and Waveguide Techniques:

In non-resonator methods, the sample under test is placed in a segment of transmission line, which is usually a waveguide or a coaxial line. The properties of the electromagnetic for the materials under test, its permeability and permittivity, can be derived from the scattering parameters. All the four scattering parameters can be measured in such method. Where the complex permittivity and permeability, the positions of the two reference planes and the sample length are the main variables contained in the relevant scattering equations of transmission or reflection measurement. The more difficult placement of a sample in the coaxial line even though the cutoff wavelength of coaxial line is infinity which leads to more often use the waveguides especially rectangular ones. Figure 1 illustrates the segment of a rectangular waveguide while the sample has been placed. Filling the line and leaving no air gaps is typically measurement configuration. Furthermore, it shows the axis direction which lies in x-direction which is same as the propagation direction.

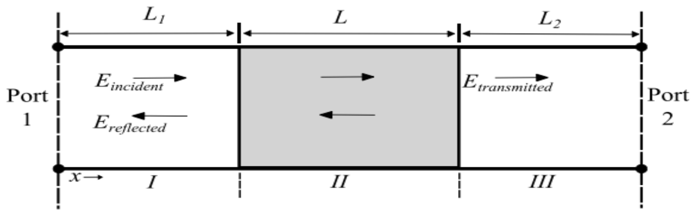

Fig. 1: Incident, transmitted and reflected electromagnetic waves in a failed transmission line (Christos, SE-221 00).

The advantage of this method is suitable for the broadband frequencies, but the arrangement of the sample is somehow complex. The coaxial lines and waveguide are normally used to measure samples as illustrated in Figure 2. In (Baena *et al*., 2005), the authors used the reflected transmission and short circuit line method measured the dielectric materials and presented the uncertainty analysis of this method

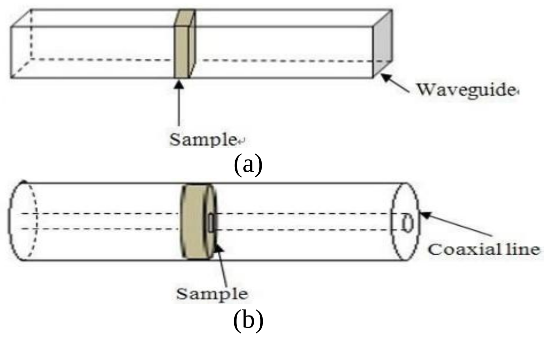

Fig. 2: Transmission Line Methods (a) Sample inside the waveguide line, (b) Sample inside the coaxial line (Xiang and Jiang, 2010).

For the TM mode, it can also propagate in a coaxial cable. The coaxial line for characterizing material properties should work in a pure TEM mode in order to ensure the measured accuracy and its sensitivity. Table 1 demonstrates the ranges of working frequencies for coaxial airline with different dimensions. It can be observed that the coaxial line with a smaller outer diameter has a wider range of working frequency, while it has strict requirements on sample fabrication than the coaxial line with a larger outer diameter (Chen *et al*., 2004)

Table 1: The 50 Ω coaxial line for the working frequency ranges with different outer diameters (mm).

Working Frequency Range (GHz)	Outer Diameter (mm)
0 – 24.5	3.5
0 – 18.2	7.0
0 – 8.6	14.0

Open reflection method:

Figure 3 demonstrates the basic configuration measurement for the open reflection method. The materials under measurement are assumed nonmagnetic in this method and the probe does not sense the interacted electromagnetic field with the non-contacted boundaries of the sample. The coaxial lines are generally preferable due to their usage for broadband and have smaller dimensions. It can measure high dielectric constant and high loss samples. It is also ideal to characterize loss solvent materials (Li and Chen, 1995) and (Stuchly M and Stuchly S, 1980).

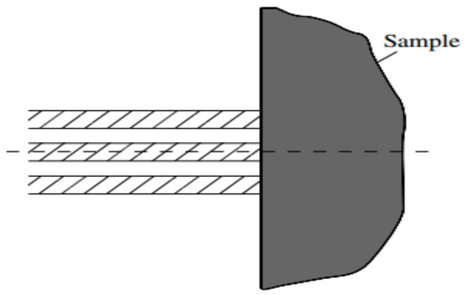

Fig. 3: Coaxial open-circuit reflection (Chen *et al*., 2004).

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Shorted reflection method:

Figure 4 illustrates the coaxial short circuit reflection. This method is used to measure magnetic permeability and the sample under test is electrically short. The permittivity of the material is not sensitive to the measurement results (Guillon, 1995), and (Fannis *et al*., 2002).

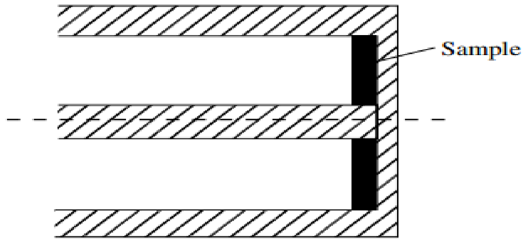

Fig. 4: Coaxial short circuit reflection (Chen *et al*., 2004).

The advantages of Transmission and Reflection Line Method is that both permeability and permittivity can be determined using this method for the material under test (MUT) and the samples has loss range from medium to high can be measured using waveguide and coaxial line. However, the disadvantages of transmission and reflection line method is that the air-gap affects the measurement accuracy and there is bound to be low accuracy for a sample whose length is a multiple of one-half.

2. Free Space Techniques:

It is possible to make accurate free-space measurements at microwave frequencies due to the precision availability of horn lens antenna that has far-field focusing ability. There are several advantages using Free-space techniques for material property measurements, which are the unwanted high order modes can be excited at an air dielectric interface in the hollow metallic waveguides cause of material's inhomogeneity such as ceramic, and composites while this problem does not exist in free-space measurement. Then, it can be used for measuring samples at high temperature and it does not need to machine the sample to fit the waveguide cross section with negligible gaps, which requires accuracy measurement limitation for materials that cannot be machined precisely such as a hollow metallic waveguide method. Figure 5 illustrates the typical free-space measurement setup, which mainly consist of two horn lens antennas and a sample under test (MUT). The using of lens antennas is for spot focusing in order to minimize the effects of sample boundaries and measurement environment (Chen *et al*., 2004), (Rustam, 2013), and (Varadan *et al*., 2003).

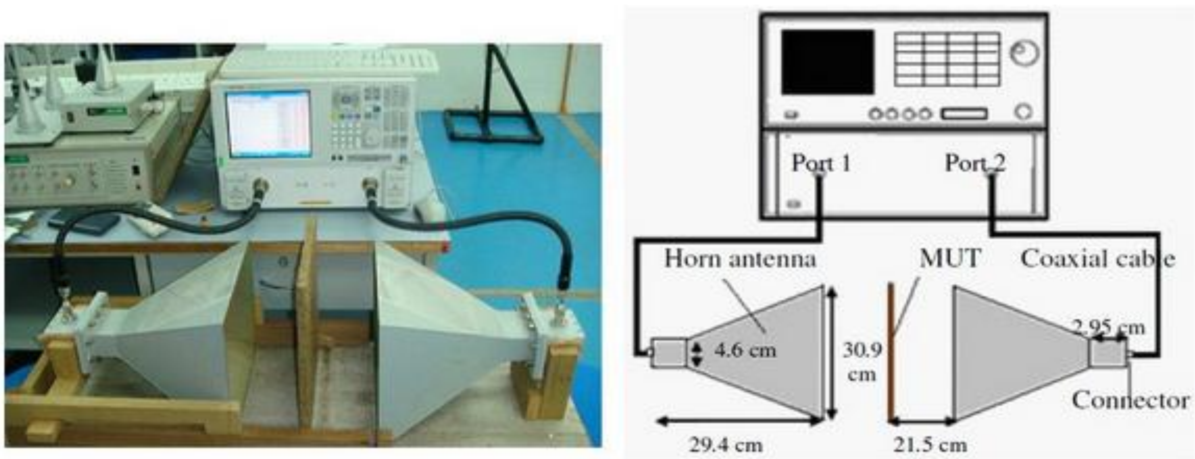

Fig. 5: Free space measurement setup using VNA (Wee *et al*., 2012).

B. Resonators Methods:

Resonant methods have high accuracy and sensitivity compare to non-resonator methods others. They include dielectric resonator methods and resonant-perturbation methods:

1. Dielectric Resonator Methods:

Dielectric resonator methods are also called resonators methods and they are widely used for characterizing the low-loss dielectric materials (krupka *et al*. 1994), anisotropic materials (Geyer and Krupka 1995), and high dielectric constant (cohn and Kelly 1966). This method can be used for measuring the permittivity of dielectric materials and the surface resistance of conducting materials. The sample under measurement serves as a resonator in the measurement circuit while the loss tangent of the sample and dielectric constant are determined from its resonant frequency and quality factor (kobayashi, tanaka 1980). The configuration often used in the dielectric resonator method is shown in Figure 6.

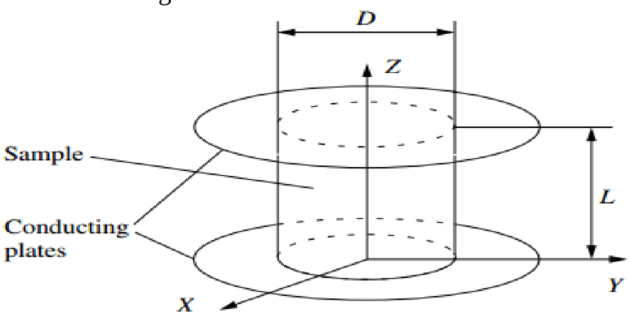

Fig. 6: A cylindrical dielectric sandwiched between two conducting plates (Chen *et al*., 2004).

2. Resonator Perturbation Methods:

The quality factor and resonant frequency of the resonator will be changed when the sample is introduced to the resonator. Then, the electromagnetic properties of the sample can be derived. There are three types of resonator perturbation methods, which are cavity shape perturbation, material perturbation, and wall-loss perturbation. Cavity shape is usually used for adjusting the resonant frequency of a cavity, whereas the material perturbation is used for measuring the low-loss materials and wall-loss perturbation is usually used for measuring the surface resistance of the conductors. Measuring the surface resistance of the conductors is shown in Figure 7 by using cavity perturbation method.

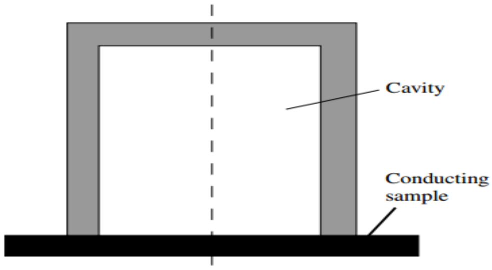

Fig. 7: Measurement for the surface resistance of conductor using cavity perturbation method (Chen *et al*., 2004).

Recent Development on Microwave Resonators for Materials Characterization:

Given that technology has rapidly developed, demands for microwave resonators still exist due to different application that use different methods for materials characterization. Therefore, the design for microwave resonators is very critical in order to achieve high accuracy, Q-factor, return loss, and testing measurement. By considering the high demands, researchers have begun searching for the best method, design with appropriate specifications, and implemented them. Numerous designs have been successfully developed for different application to characterize the materials. In this paper, several designs with different technological methods have been reviewed. The summary of researches is presented in Table 2. These researches are arranged chronologically in order to obtain the information on trends and development of microwave resonators for materials characterization. Moreover, the present study focused on which methods are extensively used by researchers in designing microwave resonator.

The analysis showed that the ring resonator is mostly used which is simple, cost effective and faster compared to other techniques. In (Jacob *et. al.,* 2008), the authors measured the microwave properties of two green tape materials with different thickness, which is 0.08mm and 0.13mm. They used high frequency (109 GHz) dielectric properties of Green tape materials in the temperature range from 10K to 290K by using spilt post dielectric resonator. They found that the permittivity measurement of uncertainty is assessed to be 0.5% with an error due to the uncertainty in measurement of thickness for the thin film samples. Furthermore, the spilt cylinder resonator is introduced in (Karsten and Uwe, 2012) and it is used for measuring materials with uncertainty budgets. The complementary spilt ring resonator is presented in (Boybay and Ramahi, 2012) and (Xi-Cheng *et. al.,* 2013). The authors, in (Boybay and Ramahi, 2012), used it for planar materials because it is easy, inexpensive fabrication, produced high measurement sensitivity, and eliminated the extensive sample preparation procedures which needed in resonance based method. On the other hand, the (Xi-Cheng *et. al.,* 2013) used it for substrate materials with terahertz range frequencies 110 GHz – 180 GHz. It is consistent and accurate measurement for the permittivity of the substrate and it has the potential to be used at higher terahertz frequencies by reducing the thickness of substrates.

Table 2: Microwave Resonator Designs for Several Researchers.

Year	Type of Microwave Resonator	Application	References
2008	Split Post Dielectric Resonator	Low Temperature Microwave Characterization of Green Tapes	(Jacob *et. al.*, 2008)
2010	Substrate Integrated Waveguide Resonators	Dielectric Characterization of PCB Materials	(Dmitry and Vincent, 2010)
2011	Circular Patch Resonator	Measurement of Microwave Permittivity of Nematic Liquid Crystal	(Dominic and Derek, 2011)
2011	Cavity Resonators	Automated Dielectric Constant and Loss Tangent Characterization	(Pasunoori and Engin, 2011)
2012	Complementary Split-Ring Resonators	Material Characterization	(Boybay and Ramahi, 2012)
2012	Modified Microstrip Ring Resonator Technique	Dielectric Characterization of Materials	(Atabak *et. al.*, 2012)
2012	Substrate Integrated Waveguide Resonators	Millimeter-Wave Printed Circuit Board Characterization	(Dmitry *et. al.*, 2012)
2012	Dielectric Resonator-Based Microwave Probe	Liquids Characterization	(María and Thomas, 2012)
2012	Split-Cylinder Resonator	Measurements for Uncertainties	(Karsten and Uwe, 2012)
2013	Cavity Resonator Measurement	Dielectric Materials Accounting for Wall Losses and a Filling Hole	(Emre *et. al.*, 2013)
2013	Multi-resonance Cell	Microwave Characterization of Powders	(Kapilevich *et. al.*, 2013)
2013	Complementary Split Ring Resonators at Terahertz Frequencies	Characterization of Substrate Materials	(Xi-Cheng *et. al.*, 2013)
2014	Rectangular Patch Resonator Sensors	Characterization of Biological Materials	(Nabila *et. al.*, 2014)

In (Pasunoori and Engin, 2011), and (Emre *et. al.*, 2013), the authors presented the cavity resonator method which is efficient, high accuracy, high Q-factor, and demonstrating the reliability with identical results. The used frequency range is from 2 GHZ to 20 GHz. The (Nabila *et. al.*, 2014) used rectangular resonator for biological materials which is used for characterizing non-invasive medical application and frequency range from 40 MHz to 65 GHz, whereas the powder characterization is introduced by (Kapilevich *et. al.*, 2013) using multi-resonance cell based on rectangular resonator with frequency range from 8 GHz to 12 GHz. It has medium losses and the method can be extended to other powder materials and application such as food and chemical. Authors in (María and Thomas, 2012), and (Dominic and Derek, 2011), introduced liquid characterization. The (María and Thomas, 2012) used the dielectric resonator based on microwave probe with operating frequency 5.72 GHz and their method can be applied for low loss liquid materials while (Dominic and Derek, 2011) used circular patch resonator with finite element software and the range of used frequency is 4.8 GHz to 8.7 GHz. On the other hands, the Microstrip ring resonator is introduced by (Atabak *et. al.*, 2012). Its frequency range from 2 GHz to 40 GHz and it can remove the conductor and radiation losses and measure materials in non-substrate forms such as powder and liquids. The authors, in (Dmitry and Vincent, 2010), and (Dmitry *et. al.*, 2012), introduced the substrate integrated waveguide resonator. Its operating frequency is 10 GHz and it has many advantages over other techniques such as, accurate attraction of unloaded resonant frequency for single port resonator, robust, used for characterization at the different substrate thickness, and its resonant cavity can be used with higher unloaded Q-factor (700-800).

Comparative Analysis Summary for Measuring Techniques:

Usually the choice of techniques depends on the required measurement accuracy, the frequency of interest, the expected value of permittivity (ε_r), material properties (such as, isotropic, homogeneous), the material form (such as, powder, liquid, sheet, solid), sample size restrictions, contacting or non-contacting, destructive or non-destructive, and temperature.

As mentioned before in the selection of dielectric measuring technique, which is basically depended onto several factors, this selection is associated with the test material. Electrics characterization for every materials are different to each other that depending on its dielectric properties. Furthermore, this dielectric material's behavior can be varied with temperature, size, and testing frequency. Therefore, the frequency is increased and the dielectric constant is decreased, which makes it dependent on the frequency. As a result, the dielectric constant will be changed from high measured frequency value if the materials are measured at low frequency. Table 3 and 4 represent a comparison of measuring techniques and the advantages and disadvantages for each technique.

For the non-resonant methods (coaxial probe technique), it can be worked over the broad range of frequencies and can be used for high-loss samples. However, the air-gaps can make some errors and it needs repetitive calibrations. Transmission line or waveguide method is used for the high frequency range to measure the electric and magnetic properties. In addition, it can be used for liquid and solid samples, but it is quite difficult for preparing samples compared to others. Wide frequency range with non-contacting testing can be used in free-space method as well as small and flat sample, but the problem is caused by the large size of the samples and the diffraction from the edges of the sample, which can cause errors. The resonant cavity is the most accurate method that supports liquid and solid samples and it is suitable for measuring single or set of frequencies. However, it is limited to low loss for small size samples only and it does not require repetitive calibration.

Table 3: Extracting the dielectric properties of different materials at various measurement methods (Rustam, 2013).

Measurement Methods	Materials	Dielectric properties	S-Parameters
Transmission and Reflection Line	- Coaxial line - Waveguides	μ_r and ε_r	S11 and S21
Open-ended coaxial Probe	- Liquids - Biological Specimen - Sem-Solids	ε_r	S11
Free Space	- High Temperature materials - Large Flat Solid - Gas - Hot Liquid	μ_r and ε_r	S11 and S21
Resonant Methods (Cavity)	- Rod Shaped Solid Materials - Waveguides - Liquids	μ_r and ε_r	Frequency and Q-Factor

Table 4: Comparisons between the most popular techniques and their advantages and disadvantages (Rustam, 2013), (Yaw, 2006), (Agilen, 2006), (Venkatesh *et al*., 2005), (Shyam *et al*., 2011), (Wee *et al*., 2009), and (Clerjon and Damez, 2009).

Measurement Techniques	Advantages	Disadvantages
Transmission and Reflection line	- Used to measure samples with medium to high loss - Used to determine both permittivity and permeability	- Limitation of measurement accuracy of the air-gap effects - Low accuracy for sample whose length is multiple of one-half wavelength in the materials
Open-ended Coaxial Probe	- Easy sample preparation - Measurement for the large number of samples in short time after the calibration - Measurement can be performed in a temperature controlled environment	- Support only reflection measurement - Affected by air-gap for measurement in the specimen
Free Space	- Suitable for high frequency measurement - Allows non-destructive measurement - Measure material under test in hostile condition - Evaluate both permittivity and permeability properties	- Need large and flat material under test - Multiple reflection between the surface of the sample and antenna - Diffraction effects at the edge of the sample
Resonant Methods (Cavity)	- Suitable to measure small material under test - Use approximate expressions for field in both sample and cavity	- Required high frequency resolution Vector Network Analyzer (VNA). - Limited to only narrow frequency bands

Conclusion:

The material characterization has gained a significant role in industrial application. Design a modification can yield a high Q-factor, compact size, high accuracy, high performance, and good measurement results. Several designs provide useful information to improve design, processing, quality and control product, whereas others are being developed based on the previous designs or a combination of certain designs. Most of the researchers tend to favor a ring resonator structure. This structure is easy to simulate, easy to fabricate, and easy to enhance the accuracy of the sensor with respect to materials characterization. Furthermore, several designs preferred medium and lower resonant frequency whereas at higher frequencies, the transmission line, coaxial, resonant-cavity and free-space methods are generally used. Open coaxial probe and free-space method have illustrated a good performance for the high loss materials while the cavity resonator methods have a higher accuracy for the low permittivity materials. It is required to consider many factors, such as measuring frequency, testing materials, losses and measurement temperature, which associated to materials while selecting the appropriate measurement techniques. Therefore, this work is helpful in understanding the recent development of materials characterization techniques and introducing a good technique, which has a high accuracy, low cost, easy procedure, rapid measurement for the desire testing materials. It can be used for many applications such as food industry, quality control, bio sensing, medicine, and pharmaceuticals. It aims to provide a reference for researchers who are interested in microwave resonators for materials characterization.

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ACKNOWLEDGMENT

The authors would like to thank Universiti Teknikal Malayisa Melaka (UTeM). This work is funded by RAGS/1/2014/TK03/UTEM//14 research grants.

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