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# Planar Microwave Sensors for Complex Permittivity Characterization of Materials and Their Applications

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## 1. Introduction

Accurate measurement of material properties has gained considerable importance over the last decade. The ability to non-destructively monitor specific properties of a material undergoing physical or chemical changes has led to many applications in industry, medicine and pharmaceuticals.

In the food industry, the interest in dielectric properties of agricultural and food materials has been principally for predicting heating rates when the materials are subjected to high frequency electric fields (Oliveira, 2002). Microwave volumetric heating for food preservation and processing applications has been popular since the early 1970s and products such as rubber, wood, paper and many other agricultural products have been studied extensively (Nelson, 1991). An important application of microwaves in the food industry has been the non-invasive determination of moisture content (Kraszewski, 1998) and its effect on the dielectric properties of materials such as granular solids, meats, vegetables and fruits (Mudgett, 1995). Microwave techniques for drying of food products have also been very popular (Schubert, 2005).

The use of microwaves in therapeutic medicine has increased dramatically in the last few years (Golio, 2003). The ability to discriminate between normal and malignant cancerous tissues using microwaves is well reported and has led to the development of many non-invasive techniques for early detection of cancer and harmful tumors in the human body. Moreover, the ability of microwaves for deep tissue heating has resulted in the development of many novel microwave therapeutic treatments. An important and a growing application is the treatment of cardiac arrhythmias using microwave ablation (Rosenbaum, 1993, Greenspon, 2000). Furthermore, novel techniques such as microwave aided liposuction (Rosen, 2000) and the RF and microwave enhancement of drug absorption are currently being investigated.

In the field of chemistry, dielectric measurements serve as a fundamental tool for the characterisation and evaluation of solvent materials, and have proven to be very convenient

for investigating the molecular dynamics of materials. The dielectric analysis of pharmaceutical materials (Benoit, 1996) has increased in importance in recent years and is vital to the understanding and application of microwaves to synthetic organic chemistry (Tierney, 2005). Microwave dielectric heating has been very popular since the early 1980s due to the many benefits it offers over conventional heating (Galema, 1997, Gabriel, 1998). Solvents with higher loss factors lead to higher heating rates than can be achieved conventionally. Moreover, the interaction of microwaves with metal powders is used extensively to create hot-spots which serve as catalysts to accelerate chemical reactions of metals with gases, other inorganic solids, and organic substrates. The reduced reaction times also assist in the synthesis of radiopharmaceuticals (Galema, 1997) and inorganic complexes, and have had a significant impact in drug discovery (Larhed, 2001, Wathey, 2002). Microwaves are also employed during many organic reactions which require dry reaction conditions. In ceramic processing, microwave heating reduces cracking and thermal stress (Michael, 1991), allows quick curing of polymers, and is more economical than conventional heating methods.

In RF and microwave circuit design the dielectric permittivity of 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 multi-layered circuits, Monolithic Microwave Integrated Circuits (MMICs) and Low Temperature Co-fired Ceramic (LTCC) circuits (Edwards, 1991). 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.

## 2. Dielectric measurement techniques

The measurement methods relevant for any desired application depends on the nature of the dielectric material to be measured, both physically and electrically, the frequency of interest, and the degree of accuracy required. Being able to design an appropriate holder for the sample and to obtain an adequate model of the circuit for reliable calculations of the permittivity from electrical measurements often proves to be an important challenge.

At low and medium frequency ranges, bridge and resonant circuits have often been used for characterising dielectric materials. At higher frequencies however, transmission line, resonant cavity, and free-space methods are commonly used and have been illustrated in an early review (Altschuler, 1973). In general, dielectric measurement techniques can be categorised as reflection or transmission type, using resonant or non-resonant systems, with open or closed structures.

### 2.1 Non-resonant methods

In non-resonant methods, the properties of the materials are fundamentally deduced from their impedance and wave velocities therein. When an electromagnetic wave propagates from one material to another, both the characteristic wave impedance and the wave velocity change, resulting in a partial reflection of the wave from the interface between the two materials. Measurements of the reflections from such an interface, and the transmission through it, can provide information for the deduction of permittivity and permeability relationships between the two materials. Non-resonant methods mainly include reflection

and reflection/transmission methods. In reflection methods, the properties of the material are deduced from the magnitude and phase measurement of the reflected signals. In reflection/transmission measurements the properties of the material are deduced from measuring the magnitude and phase of both reflected and transmitted signals. To improve confidence in findings it is generally preferred to use results from both reflection and transmission measurements. Non-resonant methods require a means of directing the electromagnetic energy towards a material, and then collecting what is reflected and transmitted through it. For this purpose, any type of transmission line could be used; for instance a coaxial line, a hollow metallic waveguide, a planar transmission line etc.

### 2.1.1 Waveguide and coaxial transmission line techniques

The use of waveguide and coaxial transmission line cells for complex permittivity measurements was first reported by Nicholson-Ross (Nicholson, 1970) and then Weir (Weir, 1974) who analysed the structure in the time and frequency domains respectively. Following this, Baker-Jarvis (Baker-Jarvis, 1990) produced iterative methods of solutions. The analysis of Nicholson, Ross and Weir led to the development of explicit formulae for the calculation of permittivity and permeability which is commonly referred to as the NRW algorithm. The technique essentially involves filling a section of a transmission line of a certain length with the sample to be measured (figure 1). The change in the propagation constant  $\gamma$  and the characteristic impedance  $Z_0$  leads to partial reflections of the wave at the interfaces. The propagation constant is related to the attenuation coefficient  $\alpha$  and propagation coefficient  $\beta$  through the relation  $\gamma = \alpha + j\beta$ . For a dielectric material the propagation coefficient of a wave within a transmission line is related to the complex permittivity of the filling material through the relation,

$$\gamma = j\sqrt{\frac{\omega^2 \mu_r \epsilon_r}{c^2} - \left(\frac{2\pi}{\lambda_c}\right)^2} \quad (1)$$

Where  $\omega$  is the angular frequency,  $\mu_r$  is the permeability of the material which is equal to 1,  $c$  is the speed of light and,  $\lambda_c$  is the cutoff wavelength of the transmission line. In case of a coaxial transmission line supporting TEM propagation the cutoff wavelength is taken to be equal to infinity.  $\epsilon_r = \epsilon_r' - j\epsilon_r''$  is the complex permittivity of the material filling the line where,  $\epsilon_r'$  is the dielectric constant and  $\epsilon_r''$  is the dielectric loss of the medium. From equation 1 it becomes possible to extract the complex permittivity of the material filling the transmission line.

$$\epsilon_r' = \left(\frac{c}{\omega}\right)^2 \left[ \left(\frac{2\pi}{\lambda_c}\right)^2 - \alpha^2 + \beta^2 \right] \quad (2)$$

$$\epsilon_r'' = \frac{c^2 2\alpha\beta}{\omega^2} \quad (3)$$

The complex propagation coefficient within the section of the line filled with the material is obtained from the S-parameter measurements with a network analyser. Reference planes are

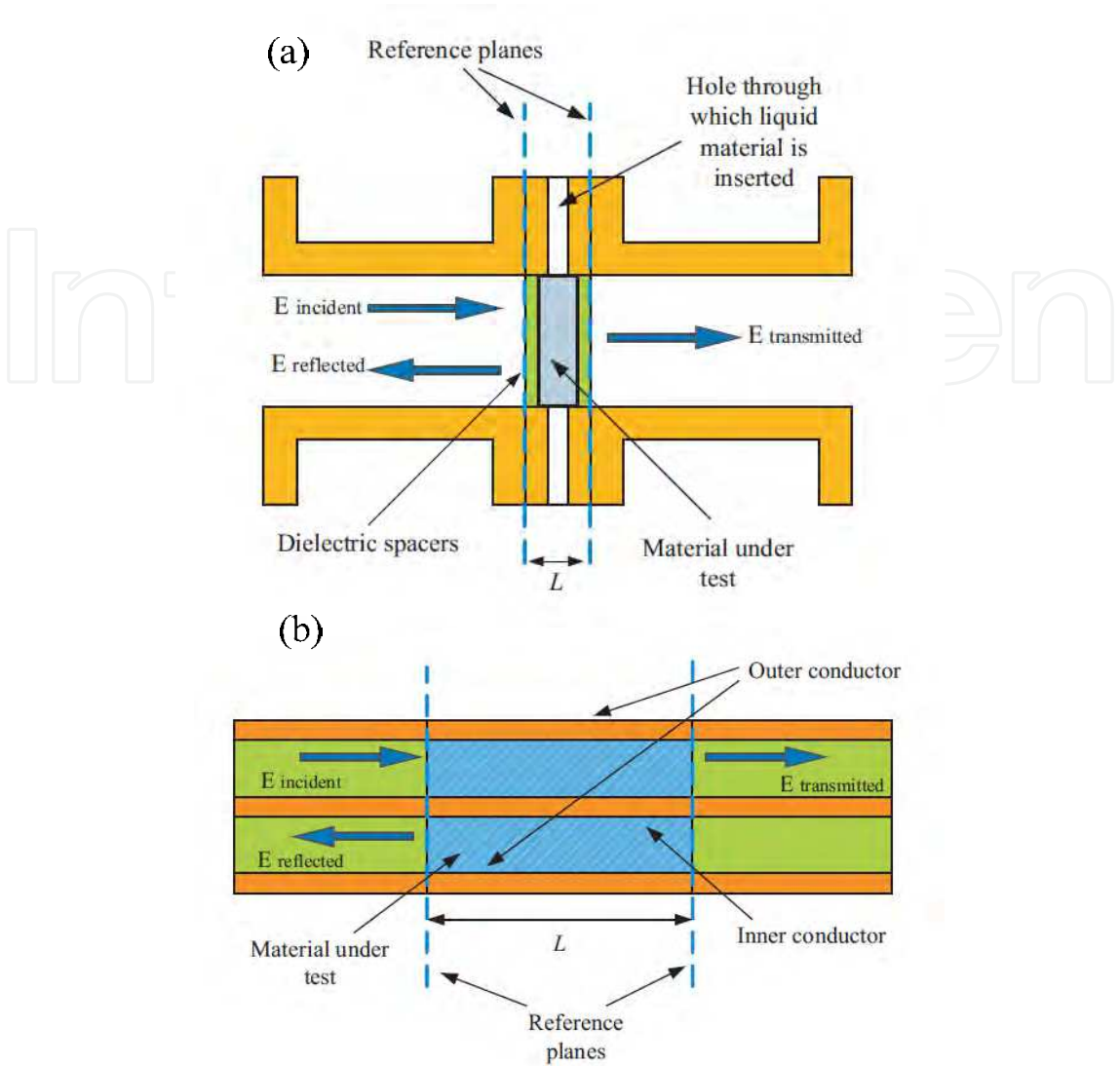


Fig. 1. (a) Waveguide fixture and (b) Coaxial fixture for transmission measurements.

established using well known calibration methods such as the Thru-Reflect-Line (TRL) or the Short-Open-Load-Thru (SOLT) techniques (Bryant, 1993). Owing to the simplicity of this technique, it has been widely adopted and many improvements have been made over the years which allow the permittivity of high and moderately lossy samples to be evaluated with a good degree of accuracy.

This technique is applicable to almost any kind of transmission line but waveguides and coaxial lines are generally preferred at frequencies below 30 GHz. Waveguide transmission cells can be constructed from either rectangular or circular waveguides and the mode of transmission depends on the structure. At frequencies below 2.45 GHz, the use of waveguides is not well suited due to the large volume of the test sample required. Coaxial transmission lines are preferable under these circumstances as they are comparatively small in size and cover a broader bandwidth. It has to be ensured that higher order TE and TM modes do not propagate as they lead to errors in the measured permittivity. For this purpose, standard cell dimensions are used. These are 3.5 mm (0 - 34.5 GHz), 7 mm (0 - 18.2

GHz), and 14 mm (0 - 8.6 GHz). Transmission line cells also suffer from problems with air gaps when the size of the cell becomes too small. The presence of air gaps in the cell leads to significant measurement errors. Over the past 30 years there have been several improvements and variations in the design of transmission line cells each being well suited to a particular class of dielectrics. At frequencies above 30 GHz, the dimensions of cells become too small and one has to resort to using free-space measurement techniques.

### 2.1.2 Free space transmission techniques

Free-space techniques are also grouped under non-destructive and contactless measuring methods, and are generally employed at higher frequencies (above 10 GHz) (Varadan, 1991). They do not require any special sample preparation, and are particularly suitable for measuring materials at high temperatures and for inhomogeneous dielectrics. They have also been implemented in many industrial applications for continuous monitoring and control. In a typical free-space transmission measurement technique, a sample is placed between a transmitting and a receiving antenna, and the attenuation and phase shift of the signal are measured, from which the dielectric properties of the sample can be determined. Accurate measurement of the permittivity over a wide range of frequencies can be achieved by free-space techniques.

In most systems, the accuracy of the determined permittivity depends mainly on the measuring system and the validity of the model used for calculations. The usual assumptions made with this technique are that a uniform plane wave is normally incident on the flat surface of a homogeneous material, and that the planar sample extends to infinity laterally so that diffraction effects at the edges of the sample can be neglected. Figure 2 shows a typical arrangement of a free-space measurement setup. Multiple reflections, mismatches, and diffraction effects at the edges of the sample are generally considered to be the main sources of errors and have to be accounted for appropriately. To enhance the measurement accuracy, special attention must be paid to the choice of the radiating

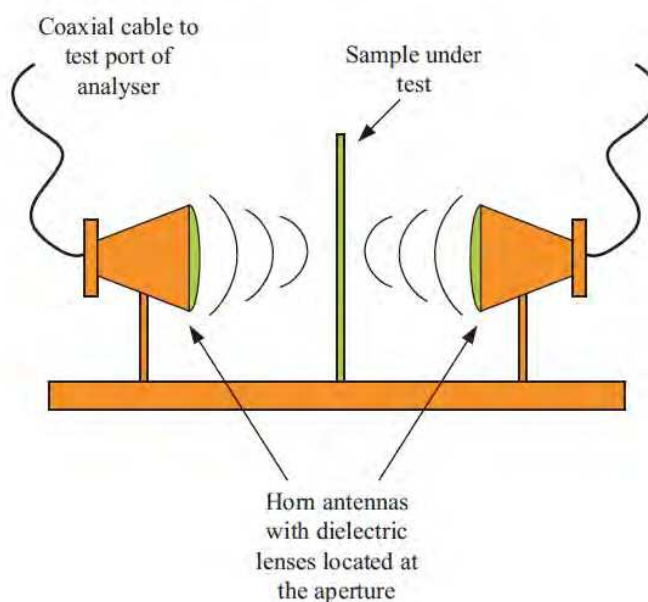


Fig. 2. Typical arrangement of a free space dielectric measurement setup.



elements, the design of the sample holder, and the sample geometry and location between the radiating elements.

### 2.1.3 Open ended transmission line techniques

Open ended transmission line techniques provide a very convenient and a non-invasive fixture for the evaluation of dielectric permittivity of liquids and semi-solids with no sample preparation. This technique was pioneered by Stuchly and Stuchly (Atthey, 1982), who used it to measure the dielectric properties of biological materials.

Over the years, this technique has been improved. In this method, the material to be tested is placed against the cut-off section of the transmission line, and the magnitude and phase of the reflected signal are measured (figure 3). Over the past decade or so several models have been used to relate the permittivity of the material to the reflection coefficient measured at the aperture of the probe. Both waveguide and coaxial transmission lines have been used, although coaxial lines are preferable since they are very broadband and have small dimensions. This technique is well suited to measuring high dielectric constant and high loss samples, and is ideal for characterising lossy solvent materials. It requires calibration reference planes to be obtained at the probe aperture, which are challenging in the case of a coaxial probe. Well known reference materials are often required for calibration.

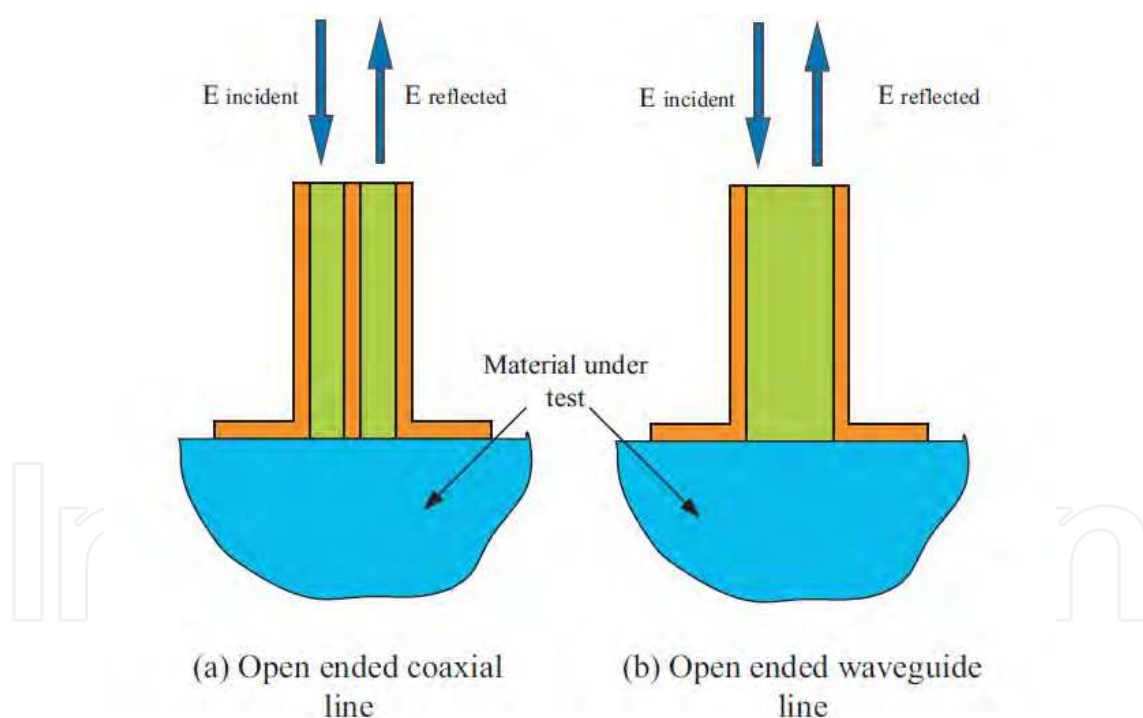


Fig. 3. Open ended coaxial and waveguide transmission lines.

### 2.1.4 Dielectric waveguide techniques

Dielectric waveguide techniques (Abbas, 2001) have recently been proposed which allow the determination of the dielectric permittivity of low loss planar sheet materials such as, teflon or perspex. The sample to be measured is placed in direct contact between two dielectric waveguides which can be of circular or rectangular geometry (figure 4). The sample can be

of arbitrary shape, but its transverse dimensions must be greater than, or in the limiting case equal to, the transverse dimensions of the dielectric waveguide. The system is based on two basic properties of dielectric waveguides. The first property is that the energy of the wave propagating within the waveguide is entirely concentrated inside the waveguide. The other property is that the phase velocity  $v_\phi$  is equal to the phase velocity of a plane wave in an unbound dielectric medium  $v_\phi = \frac{c}{\sqrt{\epsilon_r}}$ . Suitable expressions which allow determination of the complex permittivity from reflection and transmission measurements can be found in (Abbas, 2001). In order to launch energy into a dielectric waveguide suitable transitions are needed. These are generally made from metallic horn antennas which can be difficult to design.

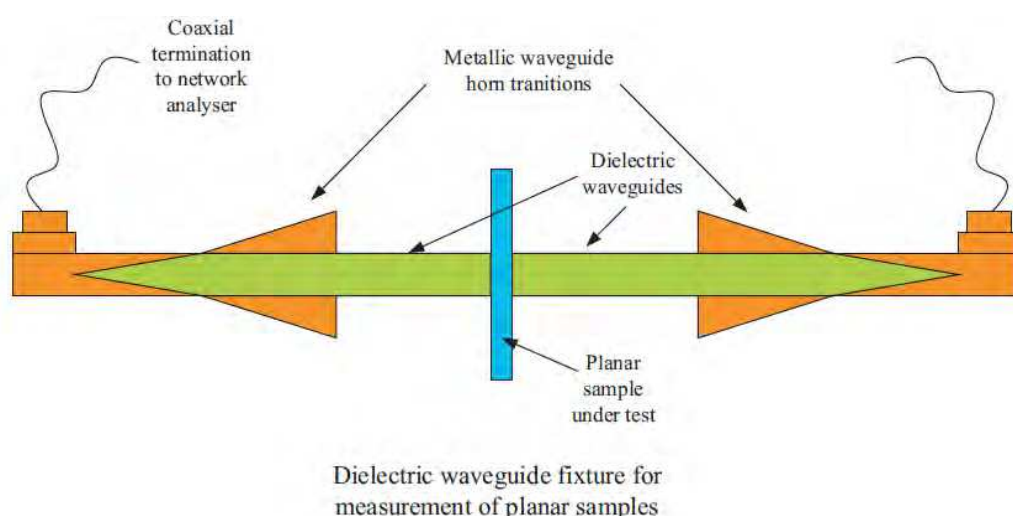


Fig. 4. Dielectric waveguide fixture for the complex permittivity measurement of low dielectric constant materials.

### 2.1.5 Planar transmission line techniques

Planar transmission lines such as microstrip and coplanar waveguides have long been used as microwave components. They allow ease of fabrication, low cost of manufacture, and compactness, which makes them suitable for industrial applications which use dielectric permittivity measurements. In planar transmission line methods, the material to be tested usually serves as a superstrate or as a substrate, or as a part of either. In the case of solids, the dielectric sample can serve both as the substrate or the superstrate. However, in the case of liquids and semi-solids, it is easier to have the sample as a superstrate. There have been many investigations into the use of planar circuits for complex permittivity measurements of liquids (Stuchly, 1998, Raj, 2001, Facer, 2001, Queffelec, 1994, Hinojosa, 2001, Wadell, 1991, Chen, 2004). Figures 5 and 6 show typical planar cells for dielectric permittivity measurements of liquid materials. The liquid to be measured completely covers the planar circuit and is enclosed inside a low loss container which is fixed firmly or epoxied on top of the board. The enclosure introduces mismatches which can easily be calibrated. The introduction of the liquid dielectric changes the effective permittivity  $\epsilon_{eff}$  and the characteristic impedance  $Z_o$  of the line. The dielectric properties are then extracted from the change in effective permittivity using suitable expressions which can be found in literature (Wadell, 1991).



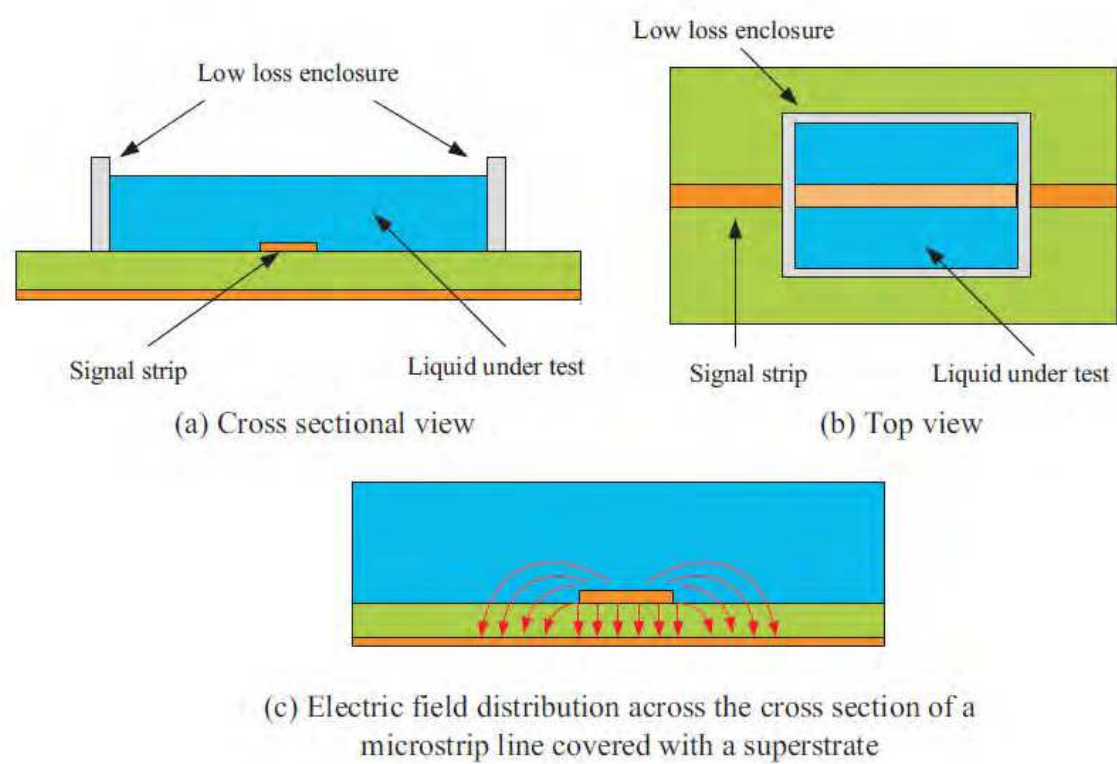


Fig. 5. Microstrip cell for complex permittivity measurement of liquids.

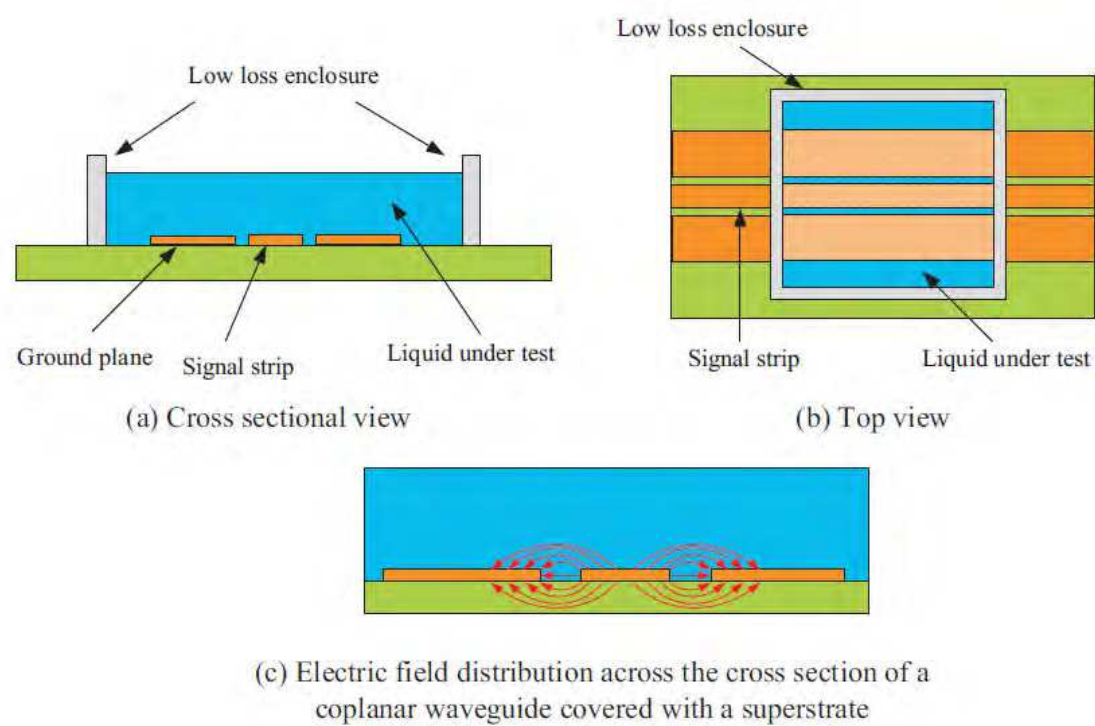


Fig. 6. Coplanar waveguide cell for complex permittivity measurement of liquids.

2.2 Resonant methods

Resonant methods offer the potential of characterising the properties of a material at a single frequency or a discrete set of frequencies with high accuracy in comparison to broadband methods. They can be classified into resonator methods and resonant perturbation methods. *Resonator methods* are those in which the material to be measured serves as a resonator and are only applicable to extremely low loss samples. The different types of resonators used for this purpose can be further classified into dielectric, coaxial surface-wave, and split resonators. Details on these methods can be found in (Chen, 2004). *Resonant perturbation methods* are those in which the sample is introduced into a resonant structure causing a perturbation in the response. The perturbation results in a shift of the resonant frequency and a decrease in the unloaded quality factor of the resonator from which the dielectric properties can be evaluated. The resonant perturbation technique is well suited for low and moderate loss samples. Both reflection and transmission type resonators can be employed for this purpose.

2.2.1 Waveguide cavity resonators

Waveguide cavity resonators are frequently employed for resonant perturbation measurements because of their high quality factors. The resonant cavities are designed in the standard TM (transverse magnetic) or TE (transverse electric) modes of propagation of the electromagnetic fields. The choice of cavity used depends on the particular field distribution of interest. The material to be tested is inserted at a particular location within the cavity where the electric field is at a maximum. Often a rod shaped sample is used, and in the case of liquid and semi-solid materials an appropriate cylindrical enclosure is needed to hold the sample. The insertion of the sample within the cavity causes perturbation to the system which results in a shift in the resonant frequency and a decrease in the unloaded quality factors. This perturbation in the response of the cavity is related to the material properties through cavity perturbation theory which is the most common technique used owing to its simplicity and accuracy. The cavity must be designed for a particular frequency of interest and at lower frequencies the cavity resonators are often large.

Figure 7 shows a rectangular TE<sub>101</sub> and a cylindrical waveguide TM<sub>010</sub> cavity operating in the two most widely used modes of propagation for permittivity measurements. Also shown in figure 7 is the magnitude of the electric field distributions for each mode.

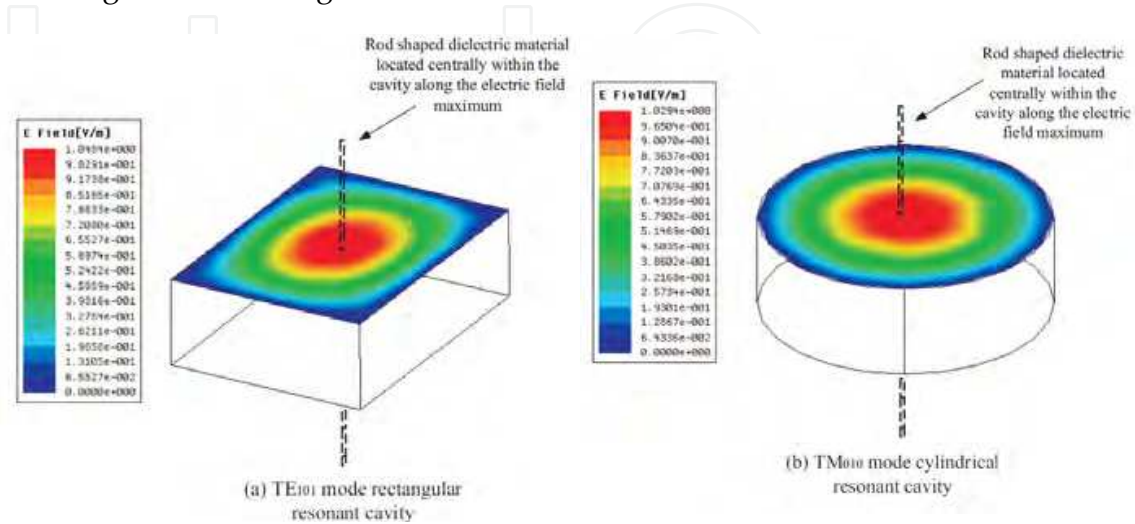


Fig. 7. Waveguide cavity resonators for complex permittivity measurements.

2.2.2 Coaxial cavity resonators

A coaxial cavity shown in figure 8 can also be used for complex permittivity measurements of both moderate and high loss samples. This technique has been demonstrated by Raveendranath and colleagues (Raveendranath, 2000), and can also be analysed using perturbation theory. It offers high quality factors and thus can be used for measuring lossy samples. The coaxial cavity resonator is essentially a straight piece of coaxial transmission line which is coupled to another coaxial line from one end with the other end either shorted or left open. The cavity exhibits resonance at the fundamental with only one electric field maximum and multiple higher order resonances which could also be used for measurements at discrete frequencies. A rod shaped material is inserted within the cavity through a slot cut into the outer conductor, which can slide across the length of the cavity. Since coaxial lines are broadband in nature, resonant measurements can be carried out over a very broad bandwidth.

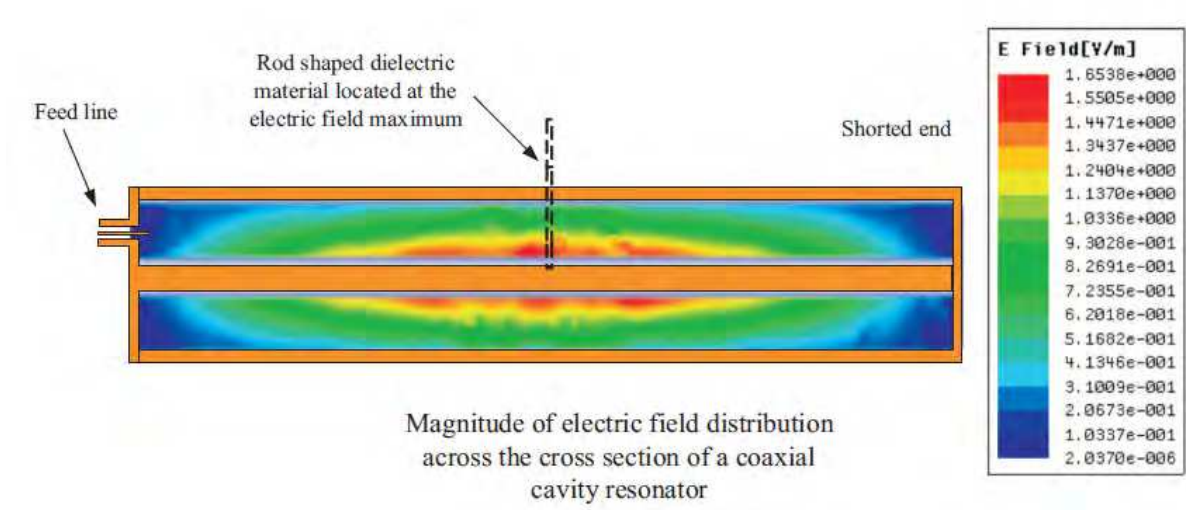


Fig. 8. Coaxial cavity resonators for complex permittivity measurements.

2.2.3 Open ended resonant lines

A very important but not so widely used resonant method of complex permittivity measurement is the resonant open ended line method first demonstrated by Johnson and colleagues (Johnson, 1992). The sensor consists of a long coaxial line with the input feed line located a distance  $L_1 = \lambda/4$  away from the shorted end and  $L_2 = \lambda/2$  away from the open end section (figure 9). At resonance, the fields are concentrated at the open end aperture of the resonator where the material to be tested is brought into contact. This method is ideally suited for measuring high loss samples and does not require any sample preparation. The sensor is calibrated in air before bringing it in contact with the material under test. The effect of the material in contact with the aperture of the sensor detunes the response.

The response is then tuned back to the original frequency by adjusting length  $L_1$ . This change in the length is directly proportional to the permittivity of the material. The deterioration in the quality factor of the resonator after tuning provides information on the loss of the sample.

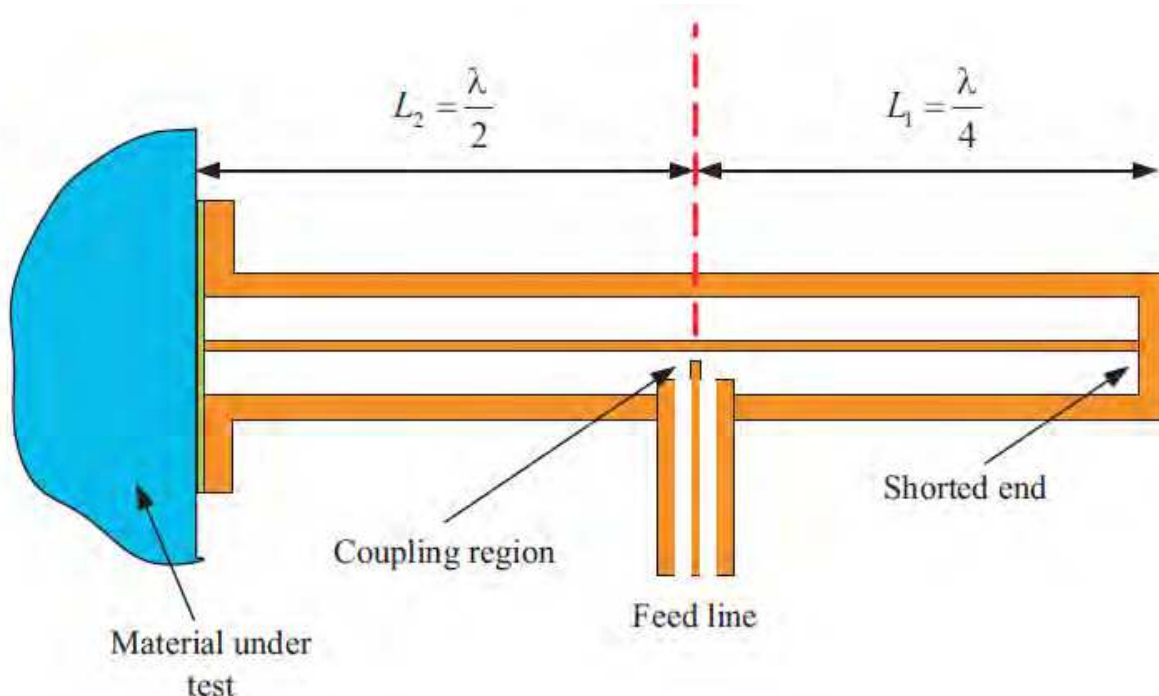


Fig. 9. Open-ended resonant line for complex permittivity measurements.

### 2.2.4 Planar transmission line resonators

Almost any kind of planar topology can be used for permittivity measurements. Unfortunately, all planar methods suffer from low quality factors (less than 500) and as a result measurements on low to moderate lossy samples have been common. Just as in the case of broadband planar transmission line methods in planar resonator perturbation measurements, the sample under study forms either a substrate or as a superstrate or as a part of either. The perturbation to the system is governed by the properties of the sample and the extent of interaction with the electric fields. Figure 10 shows a planar straight ribbon microstrip resonator fixture proposed by Abdalnour and colleagues (Abdalnour, 1995). This structure is well suited for resonant perturbation measurements on liquid samples. The sample is inserted in a small hole drilled inside the substrate. Because of the higher concentration of the electric fields at the tips of the resonator the sample can be located near the tips. The electric fields interact with the dielectric causing a capacitive perturbation in the response. The advantage of using such a technique is that only a small amount of sample (less than a few microlitres) is required for characterisation. This is highly beneficial, for instance, in the pharmaceutical industry, where accurate characterisation of materials in small quantities is vital. Another well known microstrip resonant measurement fixture applicable to moderately lossy samples was proposed by Boosanovich [Bogosanovich, 2000]. The sensor is essentially a circular disk resonator which is coupled to a coaxial transmission line that extends through the substrate from the bottom. The measurement fixture is shown in figure 11. The sample to be measured serves as a superstrate and the dielectric properties are measured from the shift in resonance and a deterioration in the quality factor of the response. The sample size required for measurements is generally large at low frequencies as the material has to completely cover the patch. Closed forms expression allow the permittivity to be calculated without requiring any reference calibration samples.



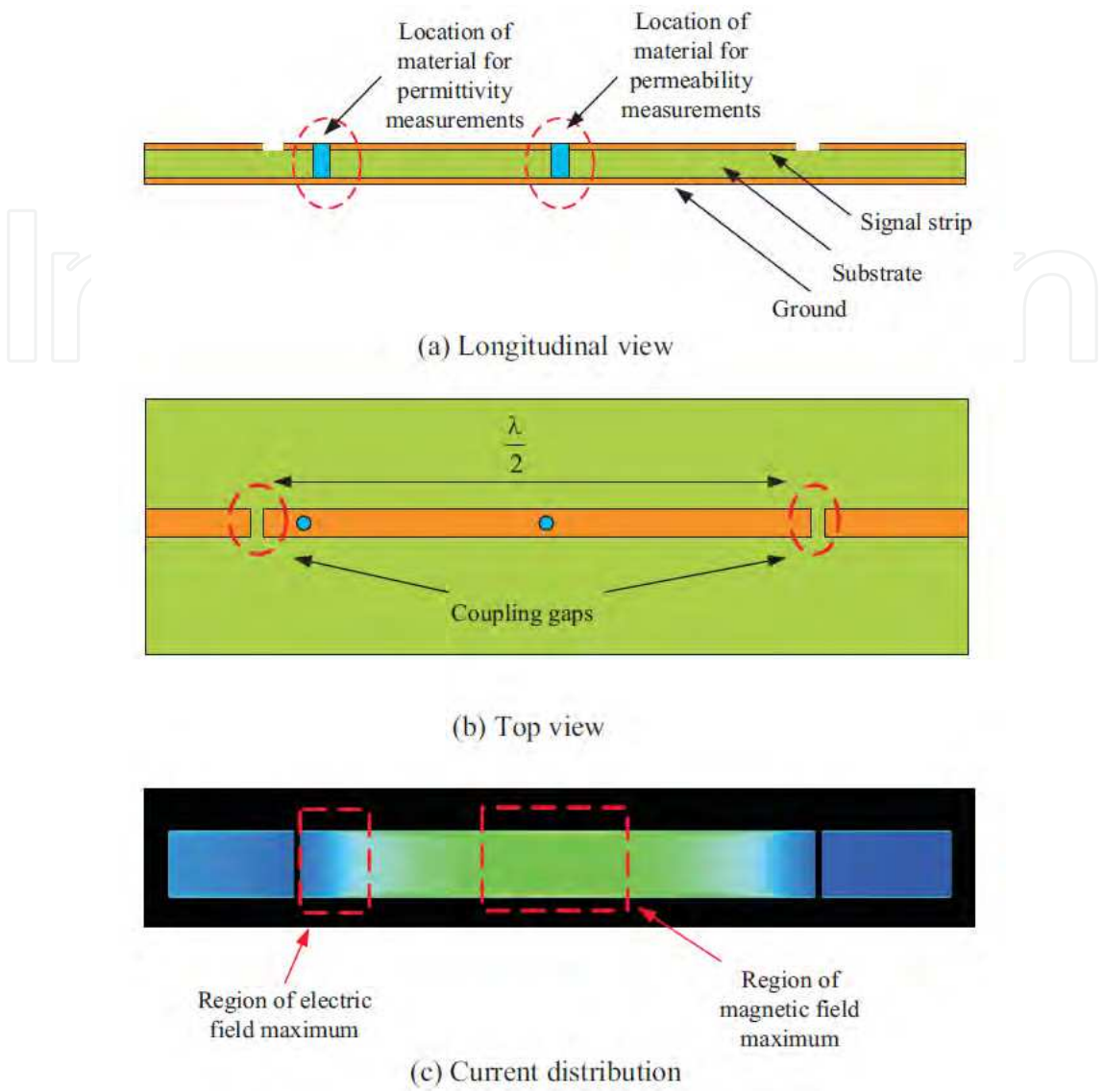


Fig. 10. Microstrip fixture for resonant perturbation measurements on liquid samples.

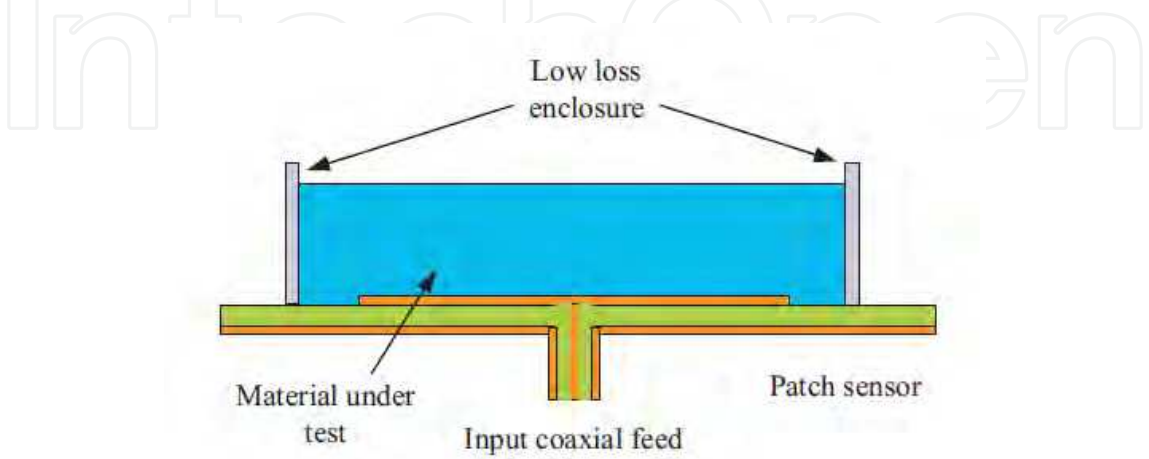


Fig. 11. Patch resonator sensor for complex permittivity measurements.



### 3. Microstrip resonators

Planar transmission lines have been used extensively in the past to measure electromagnetic properties of materials including thin films, sheet samples and substrate samples. They can offer many attractive features which include compactness, ease of fabrication and, use in a disposable manner. The three most common types of planar transmission lines used widely for materials measurements are stripline, coplanar waveguide and microstrip. Similar to their coaxial and waveguide counterparts, planar circuit methods can also be characterised into broadband and resonant methods. Resonant methods offer higher sensitivity and accuracy and therefore have been the focus of attention. Planar resonator methods are generally applicable to low loss samples such as thin films and substrates. In resonant perturbation methods part of the substrate is replaced by the sample under study. The interaction of the electric field lines with the material under test leads to a perturbation of the system thereby changing its resonant frequency and the unloaded quality factor. Since our aim is primarily to characterise lossy solvent materials using planar technology we will therefore focus our attention on resonant perturbation methods. The use of microstrip resonators for measuring material properties has been investigated by many researchers (Bernard, 1991, Abdulnour, 1995, Bogosonovich, 2002, Fratticcioli, 2004). These methods usually include the material under test (MUT) as a part of the substrate or as an overlay. The complex permittivity of the MUT is related to the resonant frequency  $f_r$  and the unloaded-Q factor  $Q_u$ , through closed form expressions which have limitations. The techniques can be destructive or non-destructive and in the past have been limited to measuring materials with low or moderate permittivities. In this section we demonstrate how a microstrip perturbation method could be used to characterise common polar and non-polar lab solvents (Saeed, 2007).

#### 3.1 Microstrip transmission line

A microstrip transmission line consists of a strip conductor and a ground plane separated by a dielectric substrate as shown in figure 12(a).  $w$  and  $t$  are the width and the thickness of the strip conductor respectively.  $h$  and  $\epsilon_d$  are the thickness and the dielectric constant of the substrate. Since the dielectric constant of the substrate is much higher than that of air the field is concentrated near the substrate. The field distribution on a microstrip line is shown in figure 12(b). The effective permittivity  $\epsilon_{eff}$  and the characteristic impedance  $Z_o$  of a microstrip line can be determined using the well known closed form expressions found in the literature (Wadell, 1991).

Microstrip resonators such as the ring resonator (figure 12(c)) and the open resonator also sometimes referred to as the straight ribbon resonator (figure 12(d)) form an integral part of many microwave circuits such as oscillators and filters. The ring resonator is a basic resonant structure which offers high unloaded  $Q$  factors due to reduced radiative losses. For rings constructed on substrates which have a much higher thickness than that of the strip conductor, we have, at resonance,

$$l = \frac{n\lambda_g}{2} \quad (4)$$

where  $n$  is an integer order of resonance and  $\lambda_g$  is the wavelength of the guided structure. To take into account the dispersive nature of a microstrip line the above equation can be rewritten as,

$$l = \frac{nc}{2f\sqrt{\epsilon_{eff}}} \tag{5}$$

where  $c$  is the speed of light,  $f$  is the frequency and  $\epsilon_{eff}$  is the effective permittivity of the structure. Similarly, the open loop resonator is resonant when its electrical length is half that of the wavelength in the guided structure. The open resonator does however, suffer from increased radiative losses due to the presence of the fringe fields at the ends which result in the inaccurate determination of the resonant frequency. In order to take into account the fringing fields, the length of the resonator is generally taken to be slightly longer.

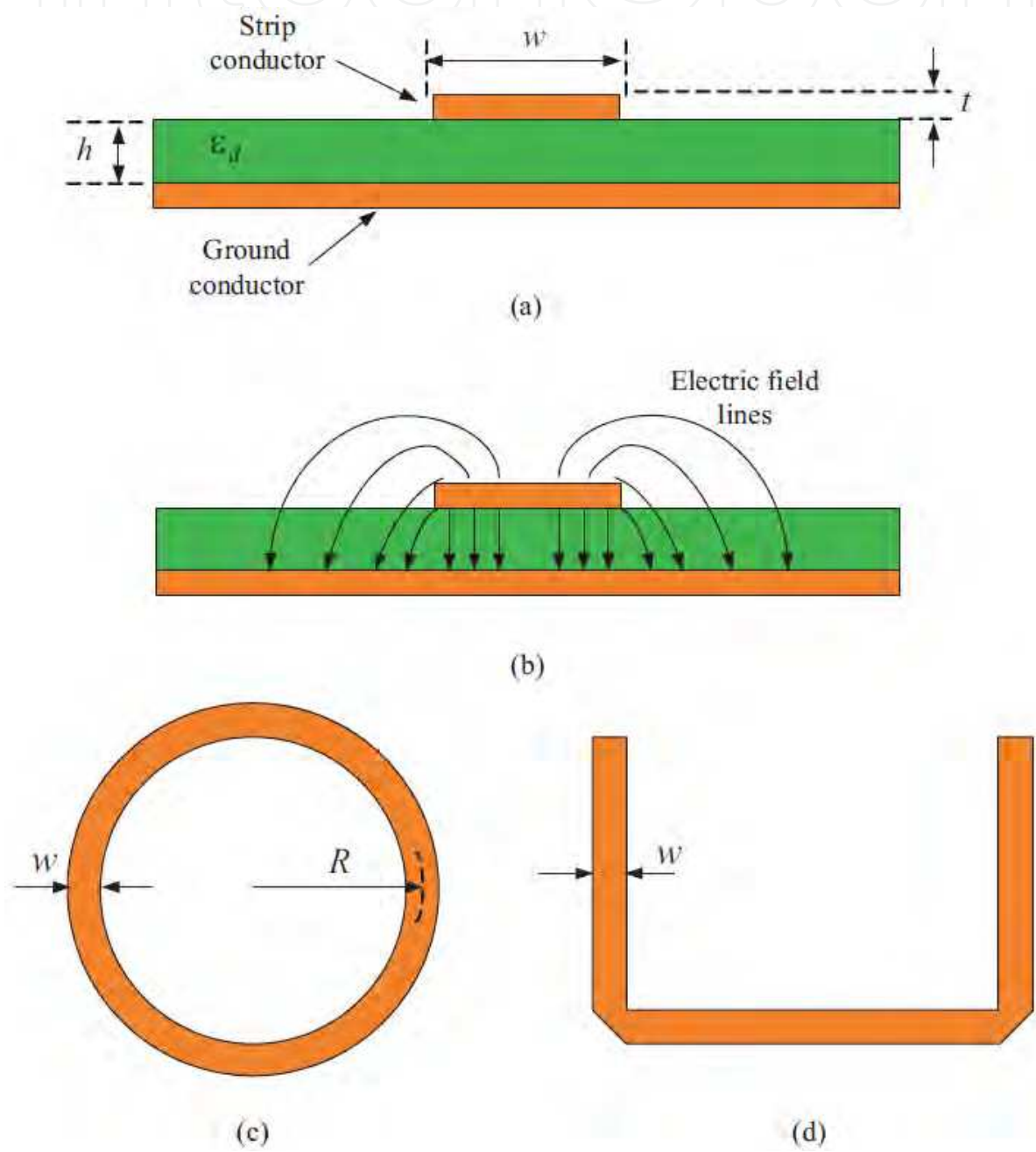


Fig. 12. (a) Geometry of a microstrip; (b) Electric field distribution at the cross section of a microstrip line; (c) Ring resonator; (d) Open loop resonator.

### 3.2 Microstrip ring resonator for complex permittivity measurements

The earliest use of ring resonators for dispersion measurements was reported by Troughton (Troughton, 1969). Later on it was realised that this simple structure could also be used to characterise the dielectric properties of thin sheet materials. Bernard and his colleagues (Bernard, 1991) used a microstrip ring resonator in a multilayer substrate topology to measure the dielectric constant of thin wafers. This technique was suitable for measuring low dielectric constant and low loss wafers.

The ring resonator could also be used to measure the dielectric properties of solvents. When the material under test is brought in close vicinity to the resonator. The fringing fields interact with the dielectric and energy is coupled into it causing a shift in the resonant frequency  $f_0$ . This shift depends on the relative permittivity  $\epsilon_r'$  of the solvent and is independent of the dielectric loss  $\epsilon_r''$ . The relationship between  $f_0$  and  $\epsilon_r'$  can be modelled using a 2nd order polynomial  $f_0 = a + b_1\epsilon_r' + b_2\epsilon_r'^2$  which provides a suitable fit. The coefficients  $a$ ,  $b_1$  and  $b_2$  can be found by using standard solvents such as methanol, ethanol and 1-propanol of which the dielectric properties are well known. Reducing the perturbation even further gives a linear response and only two calibration media are now required (air and water for which the dielectric permittivity is well known). For demonstration purposes we will use a one port coupled ring resonator as shown in figure 13 (Saeed, 2007). The resonator is modelled as an equivalent  $RLC$  network where  $R$  is the resistance,  $L$  is the inductance and  $C$  is the capacitance. Figure 14(a) is the simulated response of a square ring resonator designed on an RT/Duroid 5880 substrate with a dielectric constant of 2.2 and a loss tangent of 0.0009. The length of the resonator is 208 mm with the fundamental resonance at 1 GHz. Higher order modes occur at integer multiples of the fundamental frequency at 2 GHz, 3 GHz and so on. Enhanced coupling method with extended input transmission line was used with a coupling gap of 0.4 mm. This is a critically coupled device with a 3dB bandwidth of approximately 10 MHz and a return loss of up to 60 dB. The use of critical coupling allows more accurate measurement of the resonant frequency. The response can be tuned by selectively adding/removing copper.

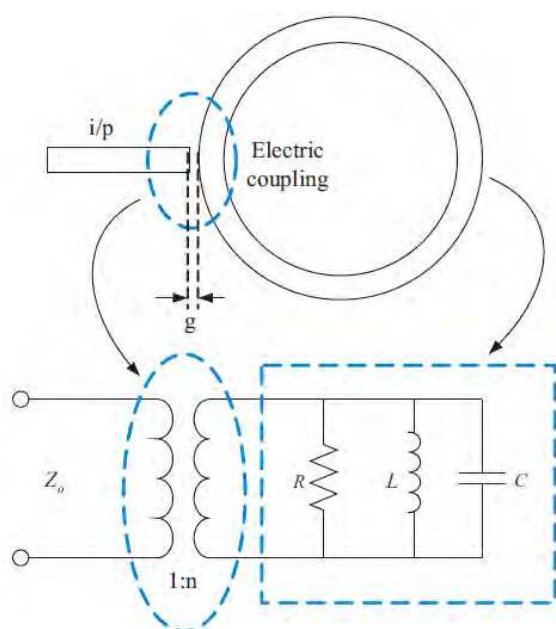


Fig. 13. Ring resonator electrically coupled to a microstrip transmission line through a gap.

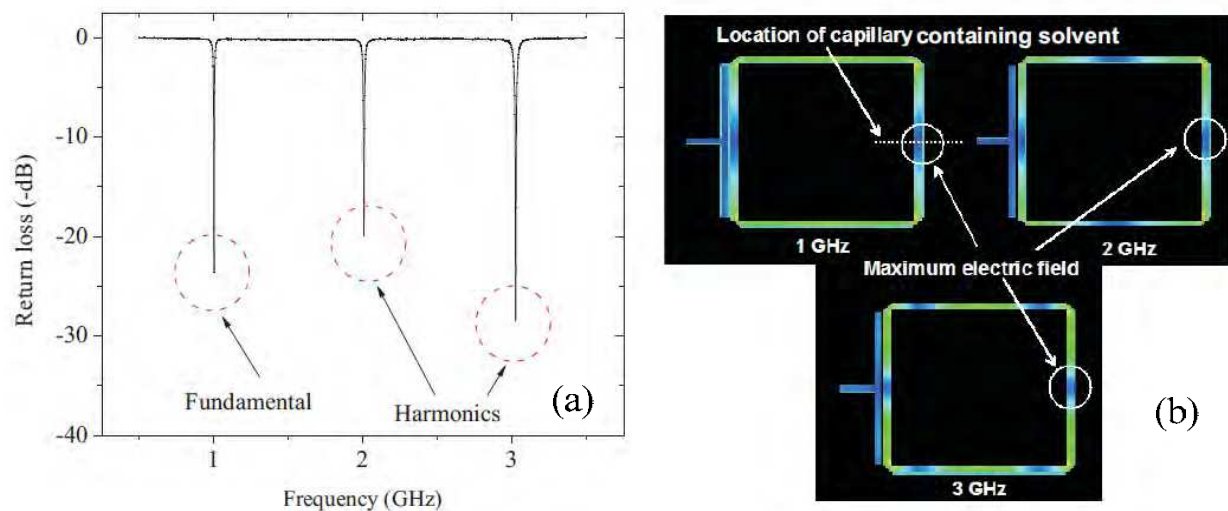


Fig. 14. (a) Simulated return loss performance of ring resonator; (b) Current distribution on ring resonator for first three resonant modes.

Also shown in figure 14(b) is the current distribution on the ring for the first three resonances. It can be seen that the coupling is electric in nature and that several electric field maximums are located along the length of the resonator. For lossy materials only a small portion of the ring can be covered by the material under test. The size and the location of the material governs the perturbation to the system. Liquid samples which can have high dielectric constant and/or high loss can be contained inside a small low loss capillary made of quartz or borosilicate glass and then introduced on the ring. Fixing the capillary on the microstrip in this manner ensures the same amount of perturbation to the system each time the measurement is made. A good location on the ring where the capillary can be located is shown in figure 14(b). Here the electric field maximum is common in all modes.

### 3.2.1 Location of capillary containing material under test

The capillary containing the material under test can be located either on top of the copper track or inside the substrate. The sensitivity depends on the extent of field penetration inside the material. Figure 15 illustrates two possible locations of the capillary for material measurements. It can be seen that the field lines are more concentrated within the substrate than in air. Therefore it should be obvious that by locating the material inside the substrate (below the copper track) results in higher sensitivity. To do this we can drill a small hole inside the substrate and insert the capillary through it. However, this might not be easily achievable in case of thin substrates. Figure 16 shows the change in the transmission response of a microstrip line when a capillary of 0.25 mm inner radius and 0.3 mm outer radius containing the material under test is placed on top of copper track and inside the substrate. The difference in the transmission responses is shown in figure 17. We can clearly see that higher sensitivity is achieved when the sample is located within the substrate.



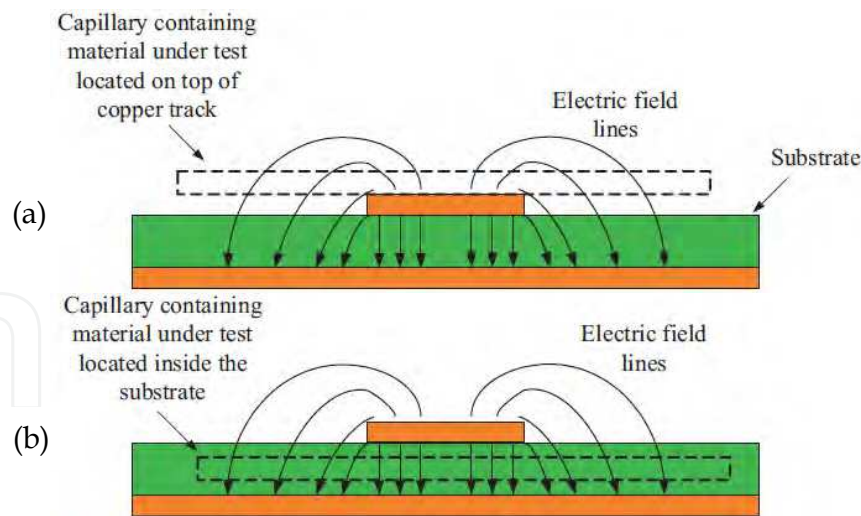


Fig. 15. (a) Capillary located on top of a microstrip line and (b) inside the substrate.

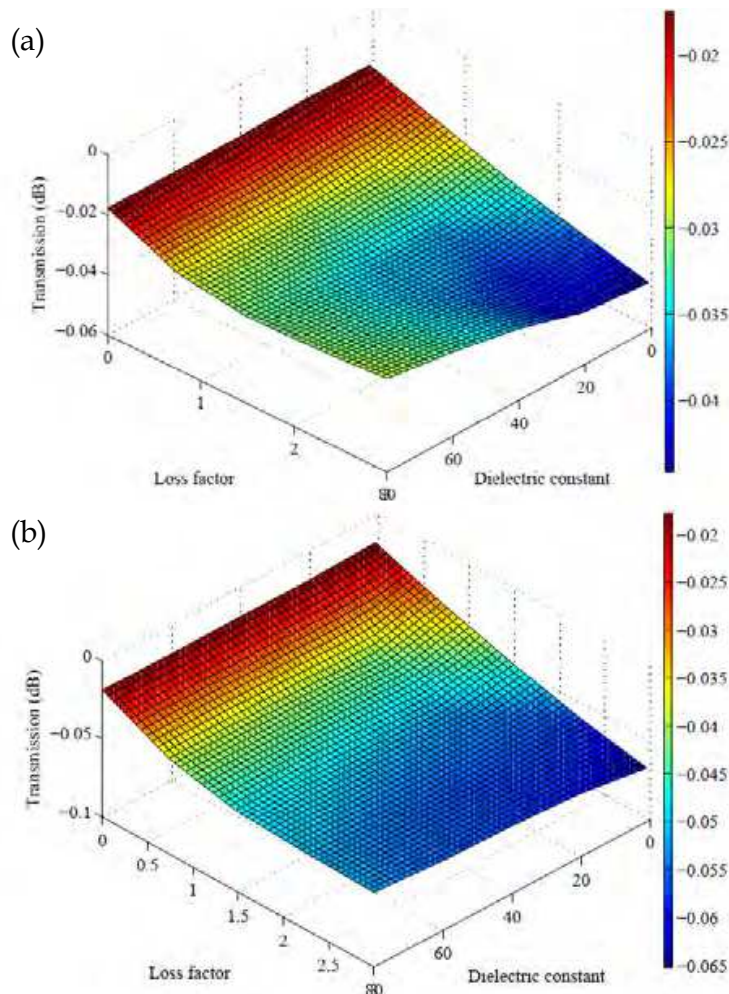


Fig. 16. Transmission response of a microstrip line at 1 GHz with a borosilicate glass capillary containing material located (a) on top of microstrip line and (b) located inside the substrate. Capillary made of borosilicate glass with outer radius of 0.3 mm and inner radius of 0.25 mm. RT/Duroid 5880 substrate with  $\epsilon_r' = 2.2$  and  $\tan(\delta) = 0.0009$  with thickness  $h = 0.787$  mm. Copper thickness  $t = 0.035$  mm and width of microstrip line  $w = 2.5$  mm.



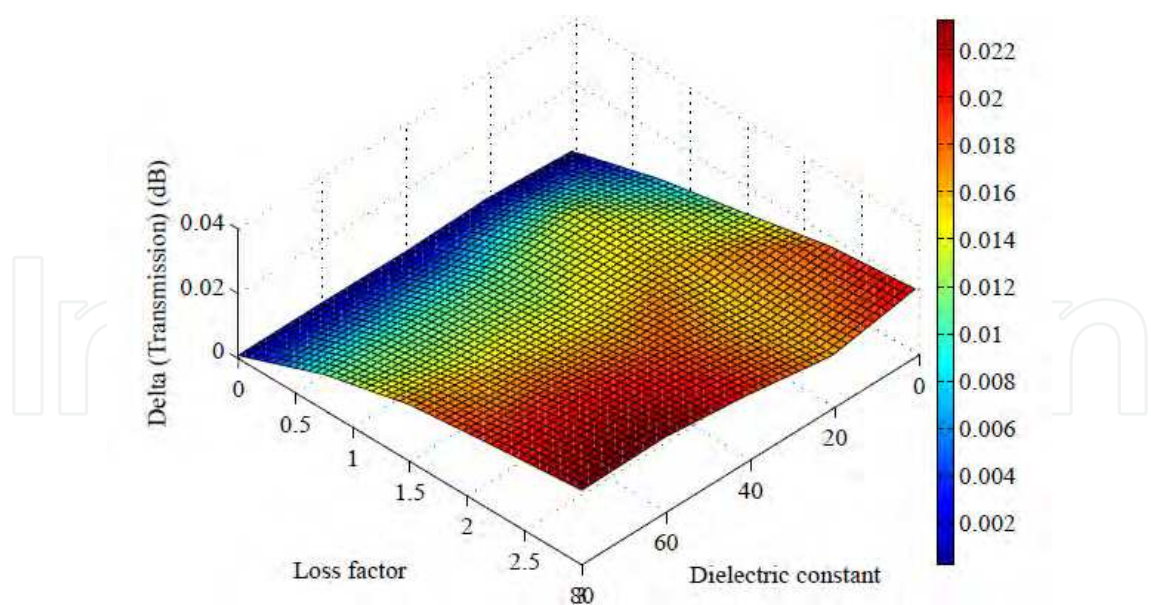


Fig. 17. Difference in the transmission response of a microstrip line when the capillary is located on top of copper track and inside substrate.

3.2.2 Measurements

The solvents used as standards and the unknowns are listed in Table 1 along with literature (Gregory, 2001) and measured values of dielectric permittivity. All samples obtained were of ANALAR standard and have purity greater than 99.5%. The measurements were carried out at 1 GHz, 2 GHz and at 3 GHz. All measurements were performed using an Agilent 8510C network analyzer at room temperature. The analyzer was used to average the data 256 times and the measurements repeated 4 times. Once the dependence of resonant frequency on dielectric permittivity was modelled and fitted using a 2nd order polynomial, the permittivity of the unknown solvents was determined. Four standards namely air, ethanol, methanol and dimethylsulphoxide were used to characterize 1-propanol and ethanediol. Air, 1-propanol and ethanol were then used as standards to characterize xylene, trichloroethylene, butyl acetate,

Solvent	$\epsilon_r'$ (Literature)			$\epsilon_r'$ (Measured)		
	1 GHz	2 GHz	3 GHz	1 GHz	2 GHz	3 GHz
Ethanol	14.14 ± 0.07	8.26 ± 0.05	6.34 ± 0.04	-	-	-
Methanol	30.17 ± 0.08	24.83 ± 0.17	19.71 ± 0.17	-	-	-
Dimethylsulphoxide	45.91 ± 0.07	44.29 ± 0.16	41.84 ± 0.26	-	-	-
1-propanol	6.98 ± 0.04	4.69 ± 0.03	4.14 ± 0.03	7.9 ± 0.16	4.86 ± 0.08	4.26 ± 0.06
Ethanediol	28.49 ± 0.16	17.34 ± 0.19	12.6 ± 0.18	27.8 ± 0.56	17.83 ± 0.3	12.51 ± 0.23
Xylene	-	-	-	2 ± 0.01	1.69 ± 0.31	1.84 ± 0.02
Trichloroethylene	-	-	-	3.08 ± 0.05	2.73 ± 0.218	3.02 ± 0.02
Butyl acetate	-	-	-	4.4 ± 0.06	4.25 ± 0.105	4.21 ± 0.03
Chlorobenzene	-	-	-	5.1 ± 0.01	4.82 ± 0.125	5.18 ± 0.05
Ethyl acetate	-	-	-	5.4 ± 0.03	5.3 ± 0.233	5.67 ± 0.11

Table 1. Measured dielectric permittivity of solvents.

chlorobenzene and ethyl acetate. Figure 18 is the measured and plotted results at 1 GHz, 2 GHz and 3 GHz respectively. The graphs clearly indicate that the resonant frequency is a good fit to the reciprocal of the square root of the dielectric permittivity.

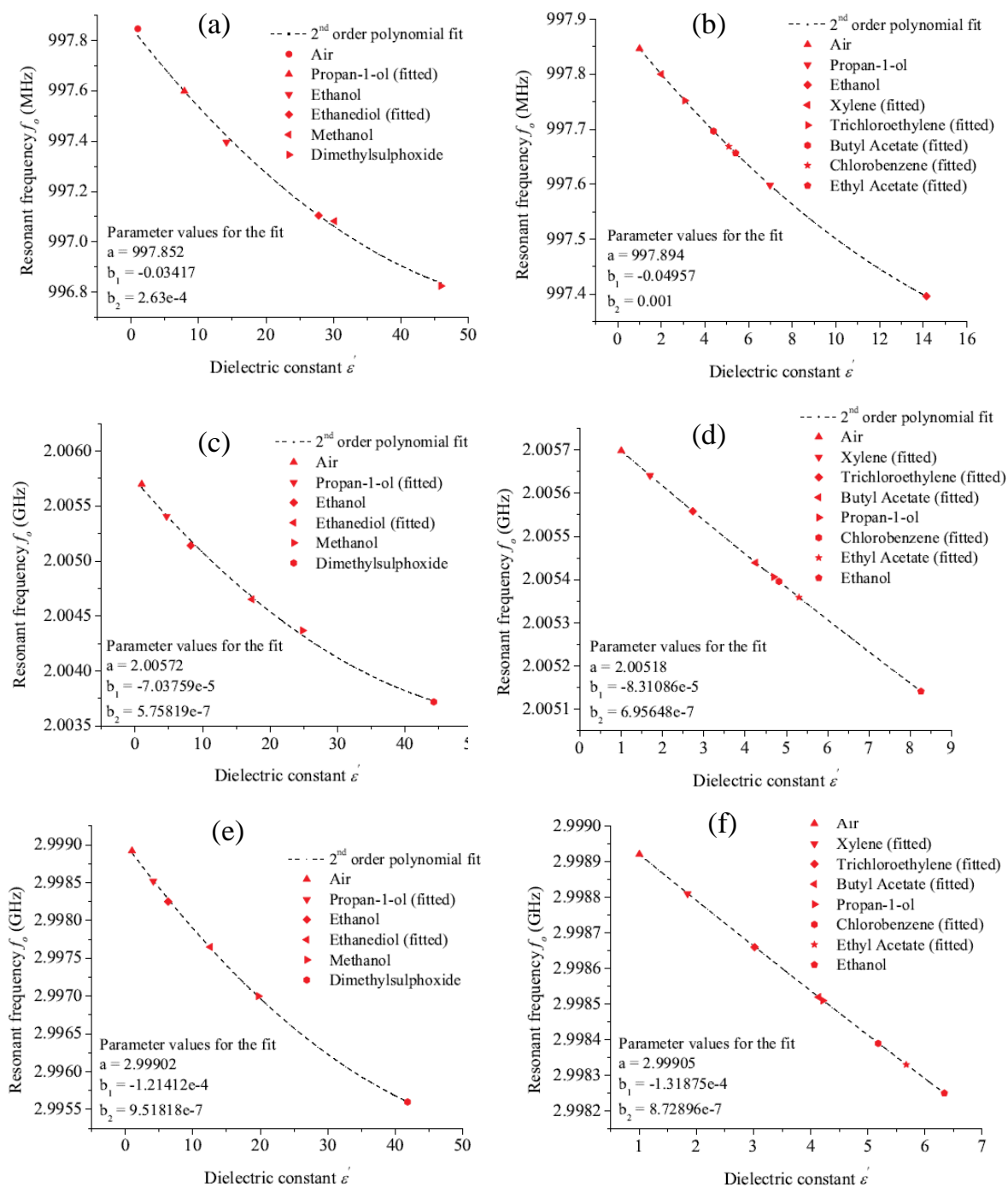


Fig. 18. Resonant frequency  $f_0$  versus dielectric permittivity  $\epsilon_r'$  for measurement of 1-propanol and ethanediol at (a) 997 MHz (c) 2 GHz (e) 3GHz. Resonant frequency  $f_0$  versus dielectric permittivity  $\epsilon_r'$  for measurement of xylene, trichloroethylene, butyl acetate, chlorobenzene and ethyl acetate at (b) 997 MHz (d) 2 GHz and (f) 3GHz.

### 3.3 Microstrip open loop resonator

To demonstrate the use of a microstrip resonator for solution sensing application we use an open loop resonator to characterise glucose-water solutions (Saeed, 2007). The resonator was designed at 1 GHz using RT/Duroid 5880. The length of the resonator is 111.98 mm with a width of 2.4 mm. The resonator is magnetically coupled to a transmission line. The electric field is concentrated at the two tips of the resonator as shown in Figure 19(a). Perturbation can be introduced to the system by placing the capillary across the tips and can be controlled by perturbing a single arm or both.

To demonstrate the high sensitivity of the resonators and the ability to measure small quantities of dissolved impurity, glucose-water solutions have been detected. This could be done either by noticing the change in the resonant frequency of the device or the unloaded-Q or both. The concentration of glucose-water solutions was varied from 0% to 4% (w/v) i.e. 0 g/100 ml to 4 g/100 ml. Figure 19(b) shows the variation in the unloaded-Q and the resonant frequency with varying glucose concentration. It can be seen that as the glucose concentration increases the resonant frequency increases whereas the unloaded-Q decreases. This indicates that the dielectric permittivity decreases and the dielectric loss increases with the addition of glucose. This could be anticipated from dielectric mixing theory (Steeman, 1990). Another conclusion that can be drawn from the graph is that it is simple to relate the concentration to the resonant frequencies and the unloaded-Q as the response is approximately linear. The device shows a decrease of 0.348 in the unloaded-Q factor and an increase of 20 kHz in the resonant frequency for every 1% increase in the concentration of glucose; making this a very sensitive method of detecting impurity concentration. Nevertheless, the sensitivity can be further increased by increasing the perturbation. From the linear relationship the sensitivity  $\Delta f/(g/100ml)$  of the device is 25.313kHz/(g/100ml) and in terms of the unloaded-Q  $\Delta Q_u/(g/100ml)$  is 0.348/(g/100ml).

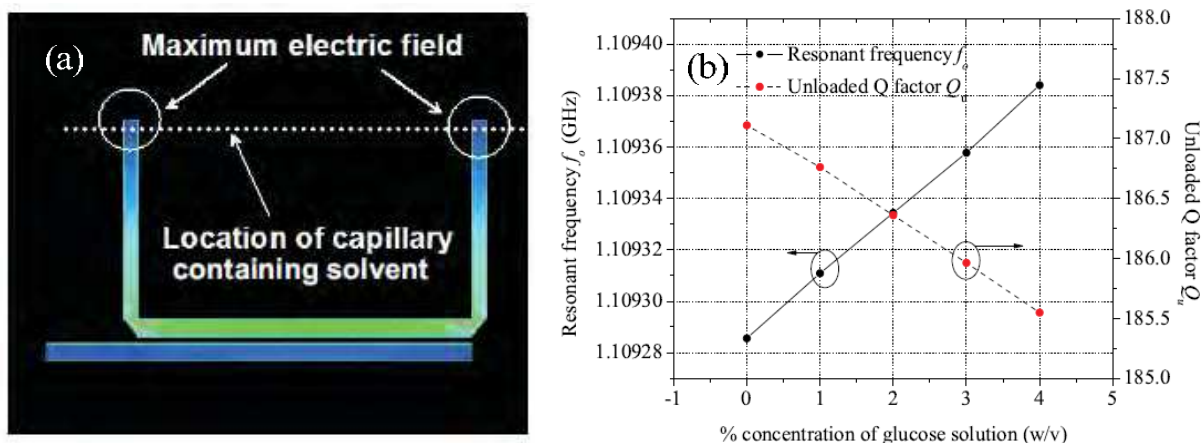


Fig. 19. (a) Current distribution on an open loop resonator at 1 GHz; (b) Resonant frequency  $f_0$  and unloaded-Q  $Q_u$  for varying concentration levels of glucose solutions.

### 4. Substrate integrated waveguide cavity resonators

Traditionally, waveguide cavity resonators have been used to characterise materials through resonant perturbation methods. They offer high quality factors, accuracy, and sensitivity

and the volume of the material required for characterisation can be as small as a few microlitres. However, waveguide resonators can often be bulky, and in some cases difficult to construct which greatly limits their use. In such circumstances, one has to resort to using planar resonant devices which are generally compact, less expensive and easier to construct. Substrate integrated waveguide resonators are a relatively new class of planar resonant devices which offer higher quality factors in comparison to other planar devices. They are compact and can be constructed easily on a substrate along with the transitions. The devices can be made extremely compact and low loss by fabricating them on a high dielectric constant and low loss substrate. This makes them a suitable candidate for many industrial applications which require measurement of dielectric properties. The compactness of these devices can allow measurements to be made on sample volumes as small as a few nanolitres with better sensitivity. Their high quality factors can allow measurements to be made on a broad range of materials.

The substrate-integrated-waveguide is an artificial waveguide structure that is fabricated on a planar substrate with periodic metallised via-hole cylinders or metallised grooves which serve as sidewalls and solid top and bottom walls of metallization (figure 20). Since their first introduction (Hirokawa, 1998, Deslandes, 2001) a large number of microwave circuits based on this technology have been constructed to demonstrate the validity of the concept in easy integration with planar devices and its significant size reduction. The SIW resonant cavity is an important component in microwave circuits, e.g. filters and oscillators etc., and is constructed in the same way as a conventional waveguide cavity but with smaller dimensions. Due to finite losses in the substrate the quality factor of the cavity is reduced but is still significantly higher than its planar counterparts. If the integrated waveguide is to

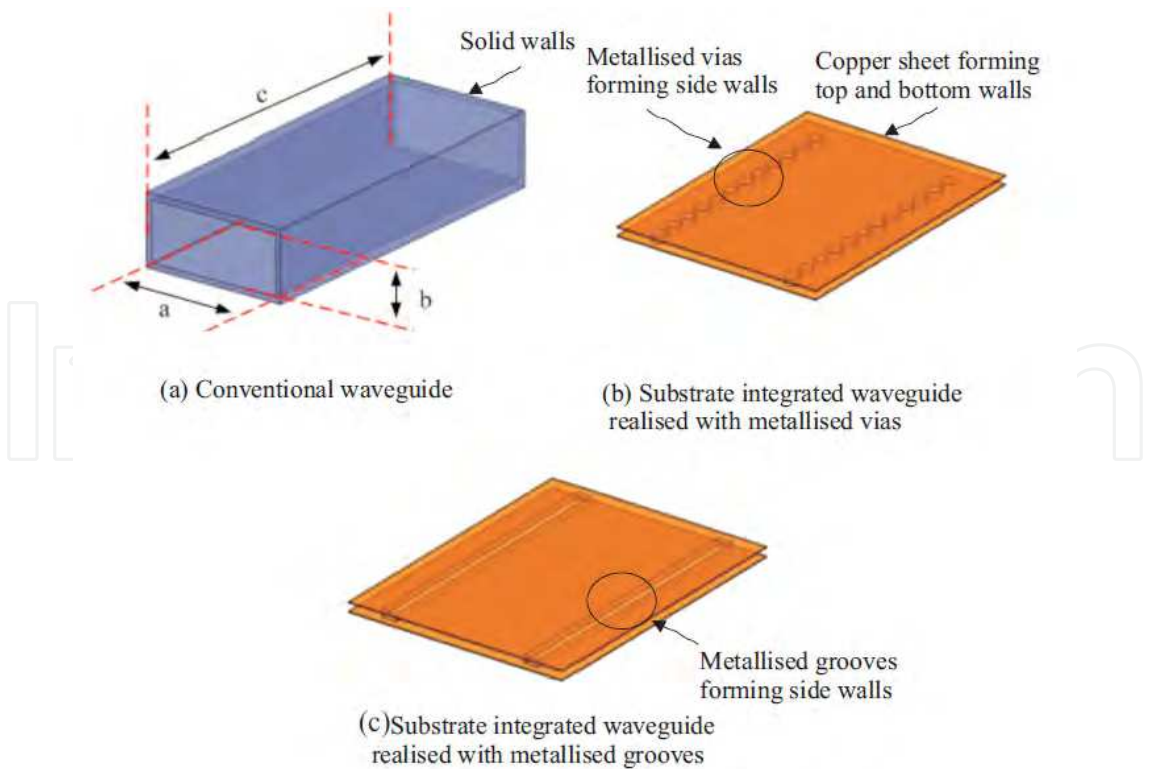


Fig. 20. Construction of a substrate integrated waveguide from metallised grooves and vias on a substrate.



be constructed from metallised grooves (figure 20(c)) the structure can be treated as a rectangular waveguide filled with a dielectric and the dimensions can be calculated easily using equations valid for a conventional rectangular waveguide. If however, the walls of the integrated waveguide are constructed using metallised vias (figure 20(b)) the exact calculations for the dimensions become rather more complex. The exact cut-off frequency of a particular mode in the integrated waveguide then becomes dependent upon the diameter of the vias and the spacing between them. In the past many authors have made use of modelling techniques such as the finite element method and the method of moments to compute the appropriate dimensions of the integrated waveguide and simulate the structures (Deslandes, 2001, Hill, 2001). Cassivi and colleagues (Cassivi, 2002) report on the dispersion characteristics of a substrate integrated rectangular waveguide for the first time and provide approximate equations to estimate the cut-off frequency of the first and second propagating modes. In this section we report on how the substrate integrated waveguide cavity resonator could be used to characterize solvents in small volumes (Saeed, 2008). The sensor is compact and highly sensitive which makes it an ideal candidate to be used for dielectric measurements in the pharmaceutical industry.

#### 4.1 Design of SIW cavity

The dimensions of the TE<sub>101</sub> SIW resonant cavity constructed from complete metallised walls/grooves can be estimated from  $\lambda_o = \frac{2}{\epsilon_r' \sqrt{a^2 + l^2}}$ . For an empty rectangular waveguide

cavity resonating at 8 GHz the typical dimensions for the width *a*, length *c* and height *b* would be 23 mm, 32 mm and 10 mm respectively. However, the corresponding substrate integrated waveguide cavity constructed using complete metalised walls and fabricated using RT Duroid 5880 laminate with a thickness of 2.8 mm would have a width of 15 mm and a length of 23 mm. This results in a great reduction in the overall volume of the cavity. Moreover, the electric field is concentrated within a small portion of the cavity. The unloaded Q factor for an empty waveguide cavity is approximately 3500 whereas for a substrate integrated waveguide cavity it is reduced to approximately 700-800 owing primarily due to the losses in the substrate. However, the use of low loss ceramics or polymer substrates will significantly improve the Q. For demonstration purposes an X-band SIW cavity resonator was designed to measure the complex permittivity of solvents. The cavity was a one-port reflection type cavity and the energy was coupled into it by means of a microstrip line [48]. The extent of coupling could be controlled through the use of an offset which makes it possible to tune into the resistive losses of the cavity and achieve return losses in excess of 50 dB. Figure 21(a) is a layout of the cavity resonator. The cavity was formed by complete metallisation of the top, bottom and sidewalls of the substrate. At the intersection of the microstrip and the cavity the metallisation extends towards the microstrip so as to form a complete cavity.

#### 4.2 Location of material under test and sensitivity analysis

As discussed in previous sections for the fundamental TE<sub>101</sub> resonant mode the electric field is dominant at the center of the cavity and a common practice is to locate the sample at this position for maximum sensitivity. The sample can be inserted either through the broad walls of the cavity or through the side walls (figure 21(b) positions A and B). In case of



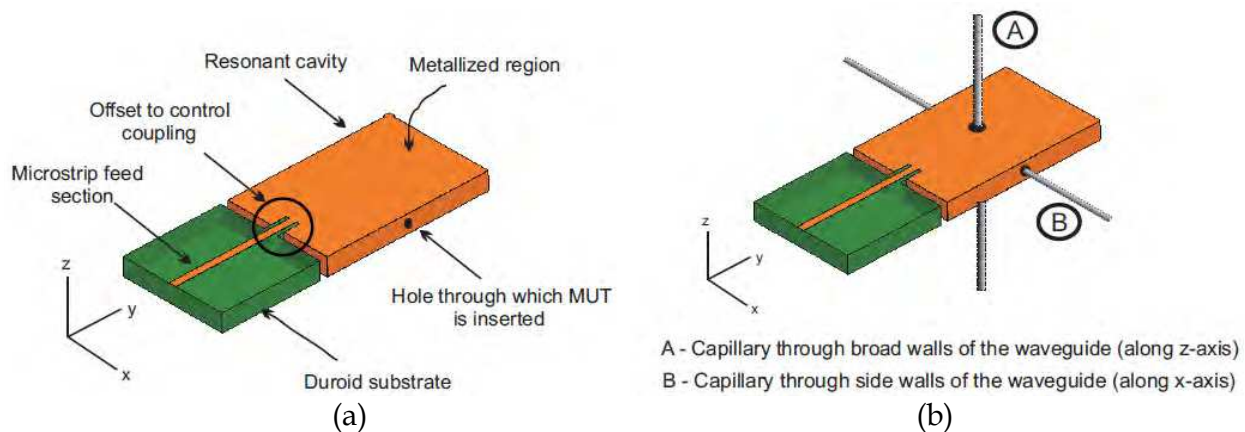


Fig. 21. (a) Layout of substrate integrated waveguide cavity resonator with microstrip feed section and hole within the cavity through which the material under test (MUT) is inserted; (b) Location of capillary containing material under test inside the cavity.

liquid dielectrics the material is contained inside a capillary made of low loss material such as quartz or borosilicate glass and then inserted into the cavity. We choose to insert the capillary through the sidewalls of the cavity through a small hole drilled into the substrate. This gives good mechanical stability and repeatability as the width  $a$  of the cavity is much larger than the height  $b$ . Care must be taken to ensure that no air bubbles are created in the portion of the capillary inside the cavity. The capillary does not need to be filled entirely as only the portion of material inside the cavity interacts with the fields. Therefore, the volume of sample required for measurements depends on the inner diameter of the glass capillary and the width  $a$  of the cavity.

The perturbation to the system is governed primarily by the location and size of the capillary. As an example we show simulation results for an X-band substrate integrated cavity resonator (8 GHz) made from an RT/duroid substrate of dielectric constant of 2.2, loss tangent of 0.0009 and a thickness of 2.8 mm with a width of 15 mm and a length of 23 mm. The simulation results were obtained using HFSS. Figures 22 and 23 show the fractional change in the resonant frequency  $F_s = \frac{f_{r0} - f_{rs}}{f_{rs}}$  and the sensitivity  $\frac{dF_s}{d\epsilon_r}$  to a lossless

dielectric material with varying dielectric constant located within a borosilicate glass capillary ( $\epsilon_r' = 3.4$ ,  $\tan(\delta) = 0.0015$ ) of various inner diameters (outer diameter = 0.7 mm).  $f_{r0}$  is the resonant frequency of the cavity without the sample and  $f_{rs}$  is the resonant frequency of the cavity with the sample. From perturbation theory it is anticipated that the fractional change in resonant frequency increases with increasing dielectric constant of material (figure 21). For capillaries with small inner diameters this increment is linear. As the inner diameter of the capillary increases the linearity is no longer valid due to higher perturbation to the system. For even larger inner diameters of the capillary the fractional change in resonant frequency increases very significantly with increasing dielectric constant. As would be expected the sensitivity is higher for low dielectric constant materials for any given diameter of the capillary (figure 22).

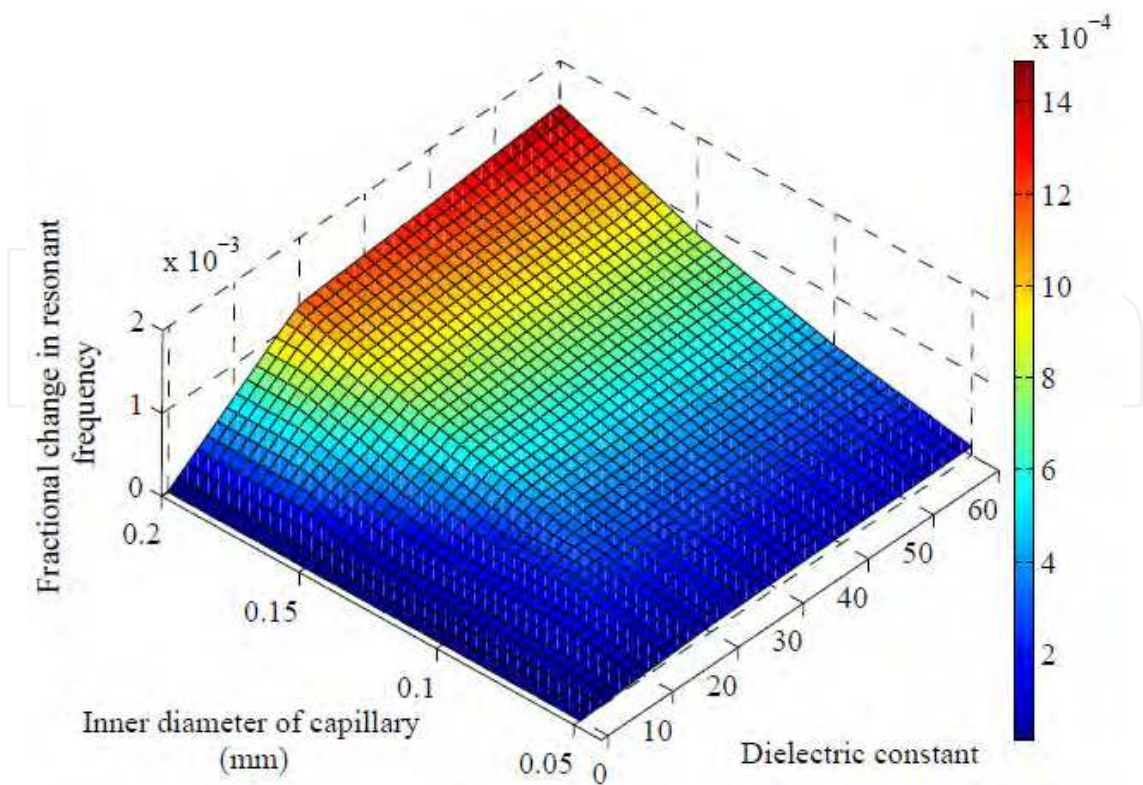


Fig. 22. Fractional change in resonant frequency with varying dielectric constant of an ideal lossless material for different inner diameters of capillary (Outer diameter of capillary = 0.7 mm).

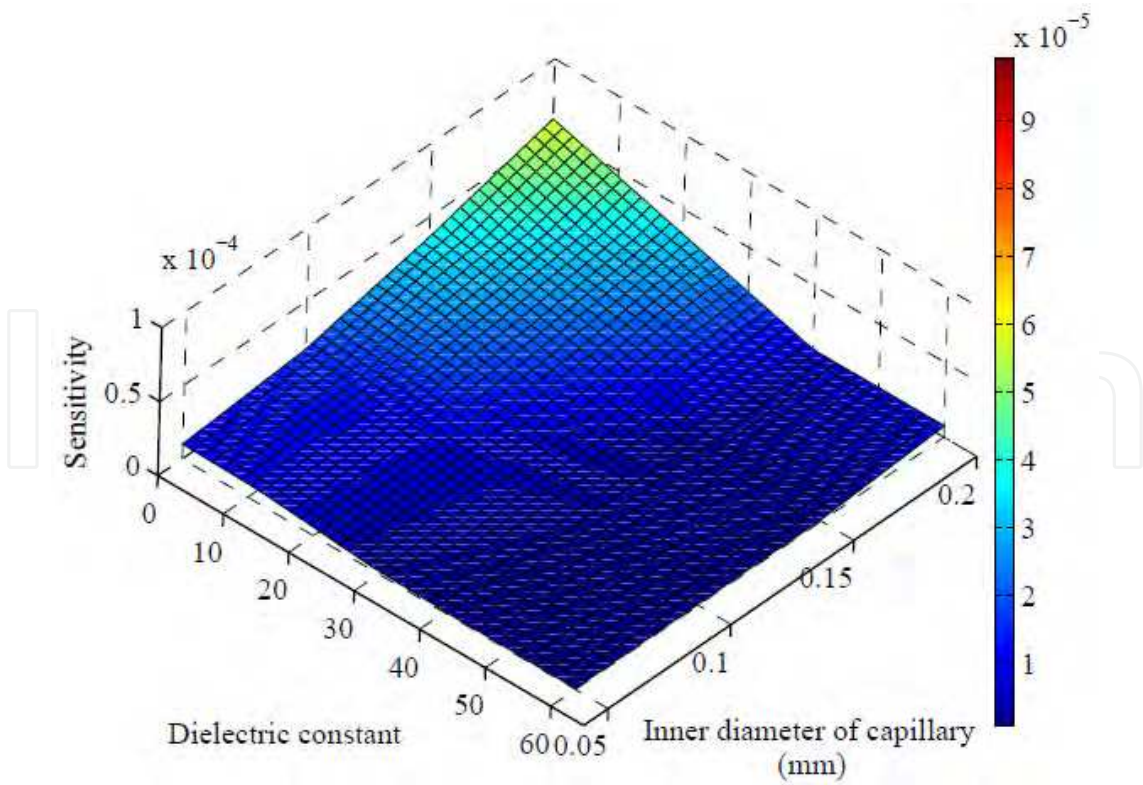


Fig. 23. Sensitivity versus the dielectric constant of material under test with varying inner diameter of capillary (Outer diameter of capillary = 0.7 mm).

For a capillary with an inner diameter of 0.05 mm the volume of material required is 29 nL. The shift in resonant frequency is 0.86 MHz for a dielectric constant change of 60 which is significantly high for samples in nanolitre volumes. This change can be measured easily with a network analyzer. In our case the volume required was less than 0.47  $\mu\text{L}$  as we chose to use a capillary which had an inner diameter of 0.2 mm. The graph given in figure 22 also provides good insight into the range of solvents that can be characterized using a specific size of capillary. It can be concluded that for a capillary of an inner diameter of 0.07 mm this technique is most suitable for measuring solvents with a moderate dielectric constant i.e.  $1 \leq \epsilon_r' \leq 20$  as the sensitivity is high. This technique is also suitable for measuring small dielectric differences in a range of solvents which can be of high dielectric constant. This is achieved by careful adjustment of coupling. The cavity containing the reference material is tuned by varying the coupling offset until the load is critically coupled to the input feed section. The solutions are then compared to the reference material by studying the relative change in the response.

#### 4.3 Perturbation of higher order modes

In order to study the dispersive dielectric properties of solvents over a desired bandwidth it is also possible to observe the perturbation of higher order modes in the resonant cavity. However, this requires the ability to vary the location of the material which can be achieved by drilling holes at various locations along the length of the cavity. To illustrate this we show simulation results on an integrated transmission type cavity formed using the same method discussed in the previous sections but with a longer length of cavity ( $l = 95$  mm). The cavity is loosely coupled to the input and output microstrip feed sections. Figure 24(a) and 24(b) are the reflection and transmission responses of the cavity. The graphs show the perturbation of five resonant modes observed when a cylindrical sample of a dielectric constant  $\epsilon_r' = 20$  is located at the centre of the cavity inserted through the sidewalls. Also shown is the magnitude of the electric field distribution at each resonance. It can be clearly seen that several electric field maximum occur along the length of the cavity. The extent of perturbation of each resonance can be controlled by varying the location of the capillary.

#### 4.4 Fabricated resonant cavity

To study the dielectric properties of solvents an X-band substrate integrated waveguide cavity resonator operating at 8 GHz has been fabricated with complete metallised walls on an RT/Duroid 5880 substrate with a thickness of 2.8 mm. Metallisation of the sidewalls of the cavity was achieved by uniformly depositing a thin layer of silver epoxy. At the intersection of the microstrip and the cavity the metallization extends towards the microstrip by 6.5 mm from either side so as to form a complete cavity. The width and the length of the cavity were optimized using HFSS. The optimized width of the cavity  $a$  was 15 mm and the length  $l$  was 23 mm. A microstrip to substrate integrated waveguide transition was used and the reflection type cavity was coupled to a microstrip line having a width of 1 mm through an offset length of 3.1 mm. The offset length for critical coupling was optimized to a borosilicate glass capillary located within the substrate through the side walls of cavity (Figure 21(b) position B with capillary along the x-axis). The capillary had an outer diameter of 0.5 mm and an inner diameter of 0.2 mm (0.47  $\mu\text{L}$  volume of material) respectively. The optimised response showed a resonance close to 8 GHz and the electric field distribution in the cavity at resonance is shown in figure 25(a). The fabricated resonator is shown in figure 25(b) and 25(c).



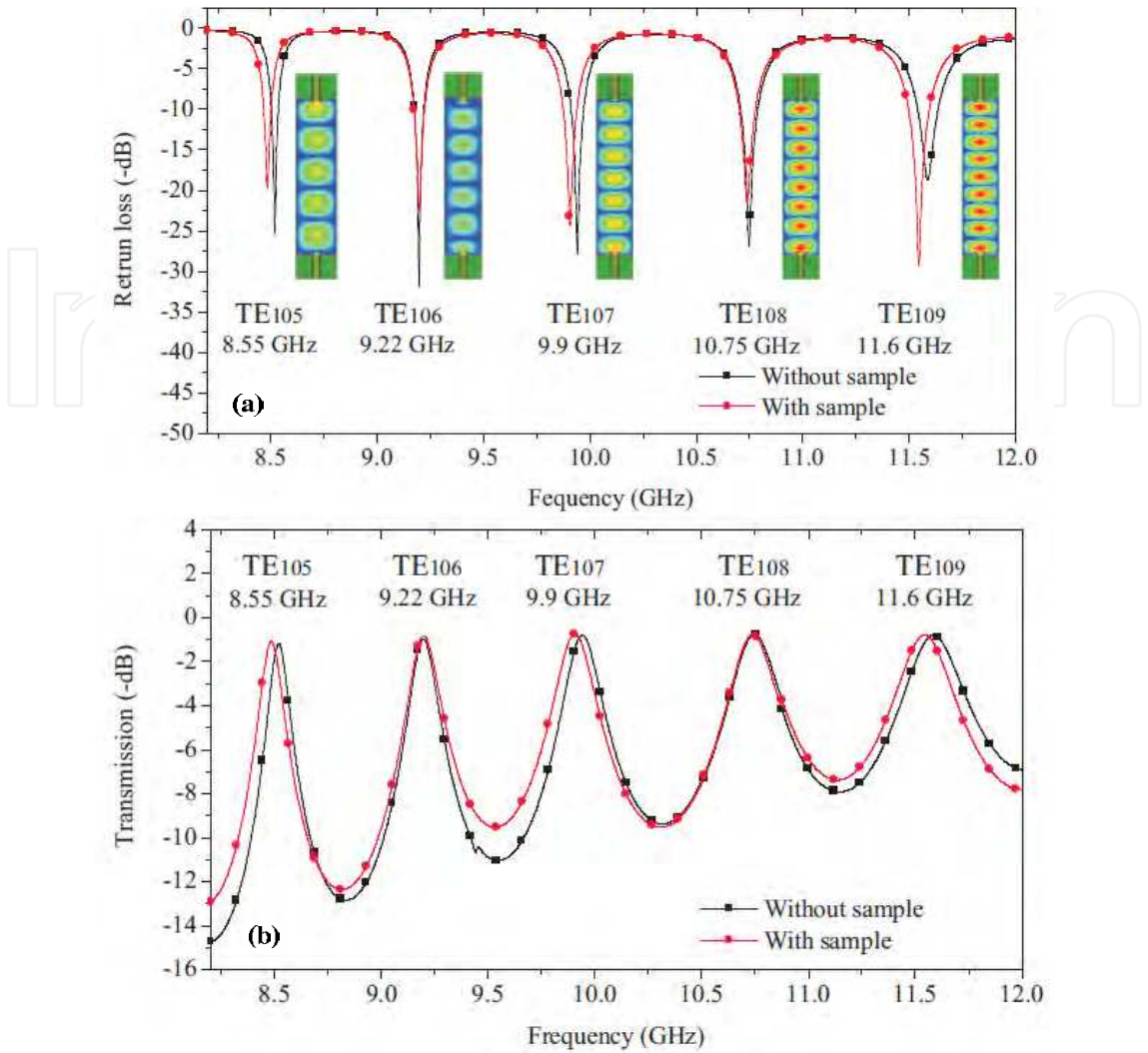


Fig. 24. Simulated (a) Return loss performance and (b) transmission response of integrated waveguide resonant cavity of 95 mm length with sample of dielectric constant  $\epsilon_r'= 20$  located at the center of the cavity.

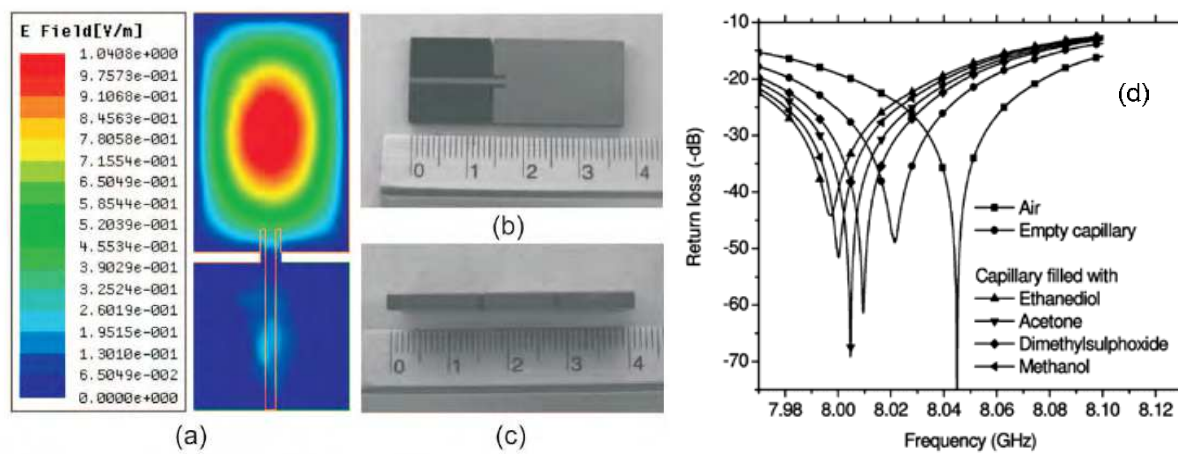


Fig. 25. (a) Magnitude of electric field distribution on the substrate integrated waveguide resonator for the fundamental TE<sub>101</sub> resonant mode; (b) Top view of fabricated resonator; (c) Side view of fabricated resonator; (d) Measured return loss performance of various solvents.

4.5 Measurements

To illustrate the ability of the compact resonator to discriminate between solvents the capillary was filled with various solvents and their response was measured using an Agilent PNA E8361A at room temperature. The data presented was the result of averaging 256 times and the measurements were repeated 4 times. Figure 25(d) is the measured response for various materials clearly indicating its ability to discriminate among solvents. The resonant frequency shifts and the Q factor changes as various materials are inserted in the capillary. The measured unloaded Q for the empty capillary was approximately 700 and the resonant frequency was close to 8 GHz. As stated in previous discussion the resonator is highly sensitive to moderate dielectric constant materials. Therefore, this technique can be used to characterize and study many alcohol solutions. For demonstration purposes we choose to measure the dielectric properties of isobutanol-isopropanol mixtures. Mixtures with varying volume fractions of dissolved isopropanol were carefully prepared and their response was measured using an Agilent PNA E8361A at room temperature. The shift in the resonant frequency was linearly related to the change in the dielectric constant (figure 26(a)) whereas the change in the 3 dB bandwidth was linearly related to the change in the loss tangent of the mixture (figure 26(b)). The measured return loss performance of the mixtures is shown in figure 26(c). The dielectric properties of the mixtures were measured and compared to those obtained using a TE<sub>101</sub> transmission type rectangular waveguide cavity and an open ended coaxial probe. A comparison is presented in table 2. From figure 26(a) it can be seen that the resonant frequency decreases as the volume fraction of dissolved isopropanol increases. This is expected as the dielectric constant of the mixture increases with increasing concentration of isopropanol since isopropanol has a higher dielectric constant than that of isobutanol. There is also an increase in the 3 dB bandwidth with increasing volume fraction of dissolved isopropanol due to an increase in loss tangent (figure 26(b)).

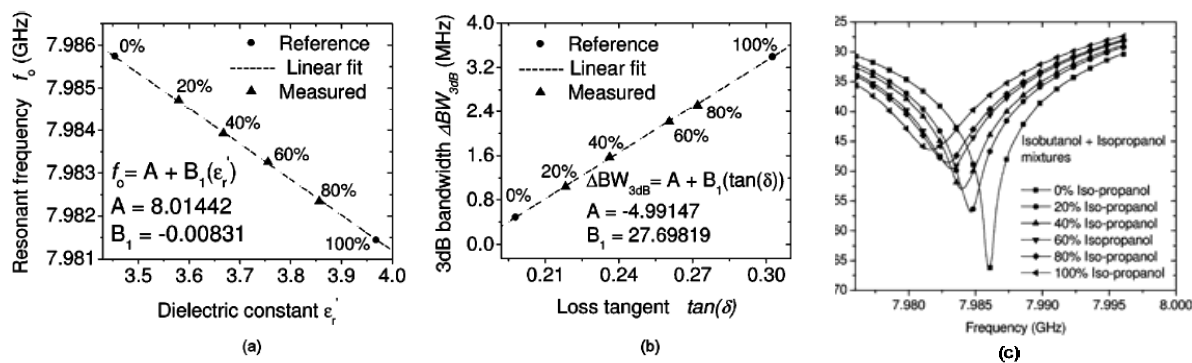


Fig. 26. (a) Measured resonant frequency versus dielectric constant of mixture for increasing volume fraction of dissolved isopropanol (%). (b) Measured 3-dB bandwidth versus loss tangent of mixture for increasing volume fraction of dissolved isopropanol (%). (c) Return-loss performance of isobutanol and isopropanol mixture.

The dielectric data measured using the integrated waveguide technique is in excellent agreement to that obtained using a rectangular waveguide resonant cavity method. For a change in dielectric constant of 0.5 there is a shift of almost 4.4 MHz in the resonant frequency indicating a high sensitivity. Moreover, a change of 2.9 MHz in the 3 dB bandwidth is observed for a change in loss tangent of 0.1. The error in the measured results for both resonant cavity and the integrated waveguide cavity is within  $\pm 0.5$  %.



$\epsilon_r'$			
$\phi_f$	Waveguide cavity	SIW	Coaxial probe
0	$3.45 \pm 0.04$	—	$3.18 \pm 0.09$
0.2	$3.56 \pm 0.02$	$3.579 \pm 0.02$	$3.25 \pm 0.14$
0.4	$3.642 \pm 0.03$	$3.667 \pm 0.011$	$3.32 \pm 0.1$
0.6	$3.78 \pm 0.023$	$3.755 \pm 0.012$	$3.39 \pm 0.13$
0.8	$3.814 \pm 0.02$	$3.856 \pm 0.017$	$3.46 \pm 0.11$
1	$3.96 \pm 0.04$	—	$3.52 \pm 0.07$
$\tan(\delta)$			
$\phi_f$	Waveguide cavity	SIW	Coaxial probe
0	$0.197 \pm 0.002$	—	$0.288 \pm 0.03$
0.2	$0.22 \pm 0.012$	$0.218 \pm 0.021$	$0.311 \pm 0.03$
0.4	$0.236 \pm 0.03$	$0.236 \pm 0.011$	$0.326 \pm 0.034$
0.6	$0.26 \pm 0.01$	$0.26 \pm 0.015$	$0.345 \pm 0.03$
0.8	$0.28 \pm 0.01$	$0.27 \pm 0.01$	$0.367 \pm 0.035$
1	$0.3 \pm 0.013$	—	$0.38 \pm 0.035$

Table 2. Comparison of measured complex permittivity of isobutanol-isopropanol mixtures using a substrate integrated waveguide cavity resonator method, a rectangular waveguide cavity resonator method and an open ended coaxial probe method.

5. Conclusion

In conclusion, we have presented a thorough introduction into the various resonant and non-resonant methods for the characterization of complex permittivity using a range of various transmission line topologies. We have highlighted the advantages and disadvantages of the various techniques and have presented a thorough comparison. Discussion has been focused primarily on the use of planar resonant perturbation methods for dielectric characterization. Two topologies namely the microstrip resonator technique and the substrate integrated waveguide technique have been highlighted as potential candidates for use as compact sensitive sensors for use within the pharmaceutical industry. The discussion has been supported through careful design of sensors and measurements on polar and non-polar common lab solvents for each of the sensors. It has been concluded that the substrate integrated waveguide sensors are highly compact offering high unloaded-Q factors and are capable of performing accurate and sensitive measurements on solvents in volumes of less than a few nano-litres making them ideally for use within the pharmaceutical industry.

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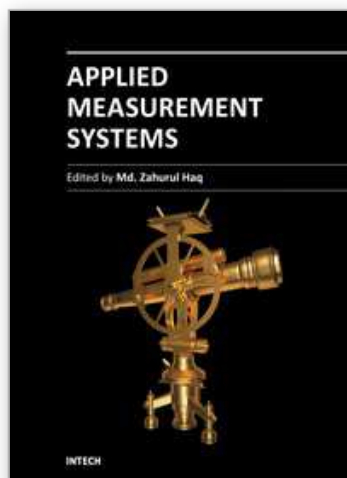
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