6 Techniques for Measuring Dielectric Properties

In general materials can be classified into conductors, semiconductors and insulators or dielectric materials. Dielectric materials play an important role in our daily life especially every electronic circuit, which needs a dielectric medium to build the circuit. Typically high frequency electronics circuits are built on dielectric materials and the operation of all high frequency circuits depends on the dielectric properties of the material. In order to design high frequency circuits it is essential to have vital understanding of the properties of the dielectric materials especially the dielectric constant (real part of complex permittivity) and loss tangent at the operating conditions. Dielectric property is also a characteristic of plant materials and fruits mainly due to the structure of the biomaterials and the large amount of water content. A 'dielectric material' (or dielectric) is an electrically insulating material that will be polarized under an electric field and the phenomenon is called dielectric polarization. The dielectric properties of the material provides valuable information about the storage and dissipation of electric and magnetic fields in materials and also provides insight into the feasibility of using the material in potential applications. The polarizability of the material is expressed by permittivity. The permittivity is a complex number and the real part is often called as dielectric constant. Though a "perfect dielectric" is a material with zero electrical conductivity, all insulators are not dielectrics.

6.1 Dielectric Properties

The two parameters which determine electromagnetic field propagation in any space are the electrical permittivity and magnetic permeability of the space. Except in the case of ferromagnetic materials, the magnetic permeability of space is usually unaltered by the presence of objects; however the dielectric properties of the material profoundly affect the electrical permittivity of the space occupied by any material object.

The dielectric properties of materials, namely permittivity, are typically measured as a function of frequency and are called dielectric/ impedance spectroscopy. The permittivity values show the interaction of an external field with the electric dipole moment of the sample (Griffiths 1999, Baker-Jarvis, *et al.* 2010, Yaw 2012). Dielectric measurement is an important tool to understand the material behavior especially at high frequencies because it can provide the electrical or magnetic characteristics of the materials, which is a critical parameter required to implement the material in many applications. The measurement of complex dielectric properties of materials at radio frequency (RF) and microwave frequency is very relevant especially in the research fields, such as material science, communication, microwave circuit design

and biological research (Burdette, *et al.* 1980, F.H.Wee, *et al.* 2009). A number of methods have been developed to measure the complex permittivity of materials in time domain or frequency domain using transmission (2 port) or reflection (1 port) methods. Each technique is limited to specific frequencies, materials and applications and has its own limitations. Often, data obtained by dielectric spectroscopy is expressed graphically in a Bode plot or a Nyquist plot.

Maxwell's equations can be used to understand and explain the dielectric properties of materials. Four differential equations proposed by James Clerk Maxwell in 1864 form the basis of the theory of electromagnetic waves (Griffiths 1999). They may be written, in vector notation, as:

$$\nabla \times E = -\frac{\partial B}{\partial t} \qquad \oint_c E \cdot dl = -\frac{\partial \Phi}{\partial t} \text{ Faraday's law}$$

$$\nabla \times H = J + \frac{\partial D}{\partial t} \qquad \oint_c H \cdot dl = J + \oint_s \frac{\partial D}{\partial t} \cdot ds \text{ Ampére's law} \qquad (6.1)$$

$$\nabla \cdot D = \rho \qquad \oint_s D \cdot ds = Q \text{ Gauss's law}$$

$$\nabla \cdot B = 0 \qquad \oint B \cdot ds = \quad \text{No isolated magnetic charge}$$

where **D** is the electric displacement, **B** the magnetic flux density, **E** the electric field strength or intensity, **H** the magnetic field strength or intensity, ρ the charge density, and **J** the current density.

In addition to Maxwell's base equations, the Lorentz condition is:

$$\nabla \cdot \vec{A} + \mu\varepsilon\frac{\partial V}{\partial t} = 0 \qquad (6.2)$$

$$\nabla \times \vec{B} = \mu\vec{J} + \mu\frac{\partial \vec{D}}{\partial t} = \nabla \times \nabla \times \vec{A} \qquad (6.3)$$

$$= \mu\vec{J} + \mu\varepsilon\frac{\partial}{\partial t}(-\nabla V - \frac{\partial \vec{A}}{\partial t}) \qquad (6.4)$$

$$= \nabla(\nabla \cdot \vec{A}) - \nabla^2 \vec{A} \qquad (6.5)$$

$$= \mu\vec{J} - \nabla(\mu\varepsilon\frac{\partial V}{\partial t}) - \mu\varepsilon\frac{\partial^2 \vec{A}}{\partial t^2}$$

$$\Rightarrow \nabla^2 \vec{A} - \mu\varepsilon\frac{\partial^2 \vec{A}}{\partial t^2} = -\mu\vec{J} + \nabla(\nabla \cdot \vec{A} + \mu\varepsilon\frac{\partial V}{\partial t}) \qquad (6.6)$$

If the Lorentz Condition holds, then:

$$\nabla^2 \vec{A} - \mu\varepsilon \frac{\partial^2 \vec{A}}{\partial t^2} = -\mu \vec{J} \tag{6.7}$$

$$\nabla \cdot \vec{D} = \rho = \nabla \cdot \varepsilon \vec{E} = \rho = \nabla \cdot \varepsilon(-\nabla V - \frac{\partial \vec{A}}{\partial t}) \tag{6.8}$$

$$\Rightarrow \nabla^2 V + \frac{\partial}{\partial t}(\nabla \cdot \vec{A}) = \nabla^2 V + \frac{\partial}{\partial t}(-\mu\varepsilon \frac{\partial V}{\partial t}) = -\frac{\rho}{\varepsilon} \tag{6.9}$$

$$\nabla^2 V - \mu\varepsilon \frac{\partial^2 V}{\partial t^2} = -\frac{\rho}{\varepsilon} \tag{6.10}$$

From the above set of equations effective permittivity can be derived:

$$\nabla \times \vec{H} = \vec{J} + \varepsilon \frac{\partial \vec{E}}{\partial t} = \sigma \vec{E} + j\omega\varepsilon\vec{E} \tag{6.11}$$

$$\nabla \times \vec{H} = j\omega(\varepsilon + \frac{\sigma}{j\omega})\vec{E} = j\omega\varepsilon_c\vec{E} \tag{6.12}$$

$$\Rightarrow \varepsilon_r = \varepsilon - j\frac{\sigma}{\omega} = \varepsilon' - j\varepsilon'' \Rightarrow \sigma = \omega\varepsilon'' \tag{6.13}$$

Measurement of dielectric properties involves measurements of the complex relative permittivity (ε), which consists of a real part and an imaginary part. As mentioned earlier, the real part of the complex permittivity, also known as the dielectric constant is a measure of the amount of energy from an external electrical field stored in the material. The imaginary part is zero for lossless materials and is also known as loss factor. It is a measure of the amount of energy loss from the material due to an external electric field.

Loss tangent:

$$\tan \delta_C = \frac{\varepsilon''}{\varepsilon'} = \frac{\sigma}{\omega\varepsilon} \tag{6.14}$$

The term tanδ is called loss tangent (dissipation factor or loss factor) and it represents the ratio of the imaginary part to the real part of the complex permittivity.

6.2 Polarization

Polarization is an ordering in space of an electrically charged unit under the influence of an external electric field. The charges become polarized to compensate for the electric field such that the opposite charges move in opposite directions. The external field causes the formation of an electric moment in the entire volume of the dielectric material in each polarizing units namely atom, ion or molecule. Linear dielectrics show a direct proportionality between the induced electric dipole moment p acquired by the polarizable unit during the process of polarization and the intensity E of the field acting on it as given by $p = \alpha E$, where α is the polarizability, which reflects the properties of individual polarizable units. Polarizability is independent of the dielectric volume and this parameter is very important to define the electrical properties of a dielectric material. As a result of polarization the charges that are displaceable will accumulated at physical barriers like the grain boundary and hence interfacial polarization or space charge polarization occurs. At the microscopic level, several dielectric mechanisms can contribute to dielectric behavior. At different frequency regions the mechanism of polarization, which gives the dielectric constant, is different. For example, dipole orientation and ionic conduction interact strongly at microwave frequencies like the dipole of water molecules, which rotate to follow an alternating electric field. Atomic and electronic mechanisms are relatively weak. Each dielectric mechanism has a characteristic "cutoff frequency".

As frequency increases, the slow mechanisms drop out in turn, leaving the faster ones to contribute to ε'. The loss factor (ε_r'') will correspondingly peak at each critical frequency. The magnitude and "cutoff frequency" of each mechanism is unique for different materials. Based on the dipolar effect the dielectric constant changes significantly at certain frequencies or will remain stable. For example the dielectric constant of water decreases significantly at 22 GHz where as Teflon exhibits constant dielectric properties. Figure. 6.1 depicts the different frequency region and different polarization mechanism.

6.2.1 DIPOLAR POLARIZATION

The formation of a molecule due to the combination of atoms by sharing electrons will cause an imbalance in charge distribution and hence a permanent dipole moment is created. When an electric field is applied due to the torque on the electric dipole, the dipole will rotate to align with the electric field causing dipolar (orientation) polarization to occur. The friction accompanying the orientation of the dipole will contribute to the dielectric losses. The ε_r' and ε_r" changes due to dipolar rotation. Below 1 kHz, dipolar polarization occurs due to the molecules containing permanent dipole moment or by the rotation of dipoles between two equilibrium positions, and this relaxation is around 10 kHz to 10 MHz.

6.2.2 IONIC POLARIZATION

The ionic polarization occurs due to the displacement of the positive and negative ions against each other, and it relaxes in the frequency range of 10^{12} to 10^{13} Hz. The different types of atoms in a molecule (or crystal) create a positive or negative charge and the centres of these charges can be displaced. The locations of the centre of charges are affected by the symmetry of the displacements. When the centres do not correspond, polarizations arise in molecules. This polarization is called ionic polarization.

6.2.3 ELECTRONIC AND ATOMIC POLARIZATION

Electronic polarization occurs due to the displacement of electrons with respect to the atomic nucleus, and it relaxes at high frequencies ~ 10^{15} Hz. Atomic polarization occurs when adjacent positive and negative ions "stretch" under an applied electric field. For many dry solids, electronic and atomic polarizations are the dominant polarization mechanisms especially at microwave frequencies, although the actual resonance occurs at a much higher frequency. The amplitude of the oscillations will be small for any frequency other than the resonant frequency. Far below resonance, the electronic and atomic mechanisms contribute only a small constant amount to ε_r' and are almost lossless. The resonant frequency is identified by a resonant response in ε_r' and a peak of maximum absorption in ε_r''. Above the resonance, the contribution from these mechanisms disappears.

6.2.4 INTERFACIAL OR SPACE CHARGE POLARIZATION

Electronic, atomic, and dipolar polarization occur when charges are locally bound in atoms or molecules. Charge carriers also exist that can migrate over a distance through the material when a low frequency electric field is applied. Interfacial or space charge polarization occurs when the charge carriers moving over a distance through the material interfaces under electric field and the motion of these migrating charges are impeded. This is the space charge polarization. The charges can become trapped within the interfaces of a material. Motion may also be impeded when charges cannot be freely discharged or replaced at the electrodes. This can also results in charge accumulation and hence higher capacitance effect.

6.2.5 DIELECTRIC LOSS

Dielectric loss (loss tangent or tan δ) quantifies a dielectric material's inherent dissipation of electromagnetic energy (for example as heat due to the charging

and discharging of capacitor). Dielectric losses depend on frequency and the dielectric material. Heating through dielectric loss is widely employed industrially for heating thermosetting glues, for drying lumber and other fibrous materials, for preheating plastics before moulding, and for fast jelling and drying of foam rubber. In communication systems, higher the dielectric loss means higher the attenuation and hence it is a limitation for long range transmission. At low frequencies, the overall conductivity can be made up of many different conduction mechanisms, but ionic conductivity is the most prevalent in moist materials. The ionic conductivity of materials will contribute to the dielectric loss.

6.2.6 RELAXATION TIME

Relaxation time τ is a measure of the mobility of the molecules (dipoles) that exist in a material. The dielectric relaxation is caused by the delay in molecular polarization with respect to a changing electric field in a dielectric medium. The movement of dipoles under the field causes collisions and hence internal friction so that the molecules turn slowly until it reaches the final state of orientation polarization with relaxation time constant τ. When the field is switched off, the sequence is reversed and random distribution is restored with the same time constant. The relaxation frequency fc is inversely related to relaxation time. The dielectric loss is proportional to the frequency up to fc. Above the relaxation frequency both ε_r' and ε_r'' decreases as the electric field is too fast to influence the dipole rotation and the orientation polarization disappears.

6.3 Cole-Cole diagram

The complex permittivity may also be shown on a Cole-Cole diagram by plotting the real part (ε_r') on the horizontal axis and the imaginary part (ε_r'') on the vertical axis with frequency as the independent parameter.

6.3.1 Bode' plots and Nyquist Plots

There are several ways of displaying frequency response data, including Nyquist plots, which is invented by Nyguist and Bode' plots. Bode' plots use frequency as the horizontal axis and use two separate plots to display amplitude and phase of the frequency response (Figure 6.1). A Nyquist plot is a polar plot of the frequency response function of a linear system. Nyquist plots display both amplitude and phase angle on a single plot, using frequency as a parameter in the plot.
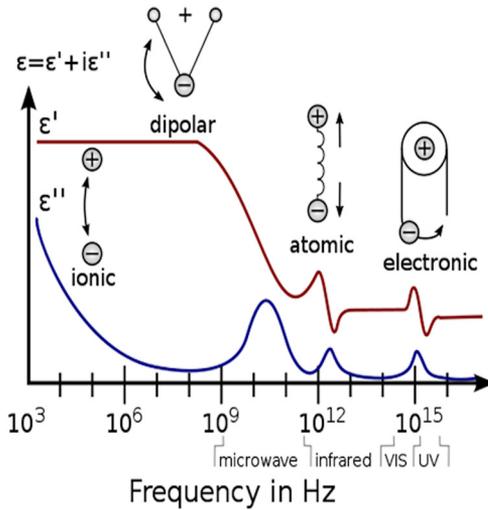
Figure 6.1: The various phenomenon at different frequencies (Technologies 2010, Yaw 2012).

6.4 Microwave Measurement Methods

There were number of dielectric characterization technique developed over the last couple of decades. This is mainly due to:

Communication technologies have grown to unprecedented heights during the last decade. Increased popularity rising due to mobile communications, wireless data transfers and instant access technologies – such as the internet, has given rise for the need for faster data rates and more data channels for an increased number of users. To cater for the increasing number of users, demanding more data in a shorter period of time, circuits must be made smaller, and perform faster than ever before. In order to facilitate this requirement, materials with good dielectric properties (Complex permittivity) must be used in the circuit. Manufacturers will give a value of permittivity for the materials, which is often at low-frequency however this measure is not adequate for use in RF (microwave frequency) applications. Dielectric characterization of biomaterials could provide information about the quality of agricultural produces and the information can be collected non-destructively. This information can be used for determining the quality of wood, fruits and vegetables, especially for wood, which is commonly used in electric poles. Degradation or termite infestation can be determined before critical damage to the system occurs.
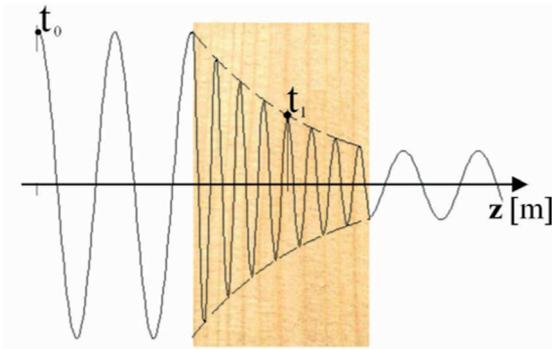
Figure 6.2: Propagation of microwave energy through wood showing the attenuation.

Decay in the tissue can cause severe damage to all biological samples including any type of wood or plant materials. Early non-destructive detection of biological degradation in wood is important if remedial treatments are to be effective. Wood and wood based materials are relatively transparent at microwave frequencies. The microwave transmission through the material depends on the moisture content. For example, when microwaves are transmitted through wood, the wave will be partially reflected, attenuated and delayed (Figure 6.2). Wave attenuation; reflections from the surface; and internal scattering from embedded objects or cavities in the wood causes a "shadow" on the opposite side of the material from the microwave source. Phase delay also depends on the bulk properties of the material through which the wave travels.

Many methods exist for determining the dielectric constant and loss tangent of a sample. Typically the methods used for the microwave characterisation of dielectric materials are classified into different groups based on the measurement structure implemented, namely Free Space Methods, Transmission Line and Reflection Methods, Resonant Techniques. These include one port coaxial and waveguide cells, open-ended probes, free-space transmission and/ or reflection methods, microwave microscope methods, microwave cavity methods, stripline and microstrip methods (Afsar, *et al.* 1986, Baker-Jarvis, *et al.* 1990, Courtney 1998, Yue, *et al.* 1998, Courtney and Motil 1999, Wang, *et al.* 2002, Murata, *et al.* 2005, Krupka 2006b). Most of these techniques are widely used. Each technique has its own limitations including the frequency at which the measurements can be performed and the type of material that can be measured. Frequency-domain methods are however preferred when measurement resolution is of concern, which is of importance for low-loss materials (Afsar, et al. 1986). Coaxial and waveguide methods are commonly used for determining the electromagnetic properties of materials, however rely on additional measurement fixtures and specific geometric dimensions of the sample to obtain accurate results. The accuracy of characterization is much higher for resonant techniques especially the modified cavity techniques (Janezic and Williams 1997, Baker-Jarvis, *et al.* 2010).

Transmission line techniques, including reflection techniques utilize transmission line concepts, where a piece of dielectric material is placed inside a transmission line, and an electromagnetic wave is directed at the sample. The disadvantage of this technique is that frequencies above 10 GHz are usually immeasurable, due to parasitic losses at higher frequencies. Majority of microwave devices operate at frequencies significantly higher than 10 GHz, hence, these techniques are not popular for characterizing PCB materials at microwave frequencies (Krupka, *et al.* 2000, Technologies 2010)

Resonant techniques are widely used to determine the permittivity and loss tangent of low loss PCB materials operating at microwave frequencies. Cavities are used to shield the dielectric, and to perform the measurement process. Cavities resonate specific to the mode for which they were manufactured. Most cavities are defined by their mode of operation. In TE_{01n} mode for example, electromagnetic field lines are tangential to the surface of the dielectric material. Resonant techniques encompass five main families of resonant metrology techniques: Microstrip, Dielectric probe, cavity, dielectric resonators and Open resonators. Cavities can be either TM_{0n0} mode, or TE_{01n} mode, while open resonators include the fabry-perot resonator. Dielectric Resonators were used for these experiments and consist of: TE_{0111} mode, Whispering Gallery Mode, $TE_{01\delta}$ Mode, and Split Post Dielectric Resonator. (Sheen 2005, Technologies 2006, Sheen 2007)

Parallel Plate Holder, is an accurate and popular method of measuring permittivity and dielectric loss of solid materials for the 8 GHz – 40 GHz operating range. The advantage of these methods lies in accessing the material from the resonator, as it is much simpler than in other devices, meaning the material is less likely to become damaged via insertion or removal. Operating range is from 50 MHz, while the permittivity measurement uncertainties are at 0.3%. (Krupka 1999)

Dielectric probes are advantageous given their ease of use, and non-invasive measurement technique. While easy to use, 3 sources of error; Air gaps, cable stability and sample thickness, can affect the measurement such that uncertainty in the technique is +/- 3% for larger specimens.

Two measurement techniques namely; Split Post Dielectric Resonator (SPDR) and Dielectric Probe, were used (Krupka 1999, 2006a). SPDR is a very accurate measurement which can be performed as a function of temperature, however as it is a resonant technique it can only be measured at one frequency. The second technique used, was the dielectric probe, even though the error in measurement is high, it allows the measurement to be carried out over a wide range of frequencies.

In general the different methods developed for complex permittivity measurement can be classified into (Baker-Jarvis, *et al.* 2010, Technologies 2010):

– Transmission/reflection line method,
– Open ended coaxial probe method,
– Free space method,
– Resonant method.

6.4.1 Transmission/Reflection Line Method

The Transmission/Reflection line method (Janezic and Williams 1997) is a popular broadband measurement method. In this method, only the fundamental waveguide mode (TEM mode in coaxial line and TE mode in waveguides) is assumed to propagate. Transmission line techniques, including reflection techniques utilize transmission line concepts, where a piece of dielectric material is placed inside a transmission line, and an electromagnetic wave is directed at the sample. The disadvantage of this technique is that frequencies above 10 GHz are usually immeasurable, due to parasitic losses at higher frequencies. The majority of microwave devices operate at frequencies significantly higher than 10 GHz, hence, these techniques are not popular for characterizing PCB materials at microwave frequencies.

A measurement using the Transmission/Reflection line method involves placing a sample in a section of waveguide or coaxial line and measuring the two ports complex scattering parameters with a vector network analyzer (VNA). The method involves measurement of the reflected (S_{11} or S_{22}) and transmitted signal (S_{21} or S_{12}). The relevant scattering parameters relate closely to the complex permittivity and permeability of the material by equations. The conversion of s-parameters to complex dielectric parameter is computed by solving the equations using a computer program. In many cases, the method requires sample preparation so that the samples fit tightly into the structure, typically waveguide or coaxial line. For accurate dielectric measurement, the maximum electric field is required within the sample. Calibrations in transmission line measurements use various terminations (standards such as open, short or 50 ohm load) that produce different resonant behavior in the transmission line.
Advantages of Transmission/Reflection line method (Yaw 2012)

– Coaxial lines and waveguides are commonly used to measure samples with medium to high loss.
– It can be used to determine both the permittivity and permeability of the material under test.
– Disadvantages of Transmission/Reflection line method
– Measurement accuracy is limited by the air-gap effects.
– It is limited to low accuracy when the sample length is the multiple of one-half wavelength in the material.

6.4.2 Resonant Technique

The resonant method provides high accuracies and assumes the TE or TM propagation mode. Resonant techniques are widely used to determine the permittivity and loss tangent of low loss PCB materials operating at microwave frequencies (Jacob, *et al.* 2002, Jacob, *et al.* 2005, Baker-Jarvis, *et al.* 2010). Cavities are used to shield the dielectric, and to perform the measurement process. Cavities resonate specific to the

mode for which they were manufactured. Most cavities are defined by their mode of operation. In TE_{01n} mode for example, electromagnetic field lines are tangential to the surface of the dielectric material.

Resonant techniques encompass five main families of resonant metrology techniques: Dielectric probe (Burdette, *et al.* 1980, Ellison and Moreau 2008, Technologies 2010), cavity /dielectric resonators and Open resonators. Cavities can be either TM_{0n0} mode, or TE_{01n} mode. Dielectric Resonators were used for these experiments and consist of: TE_{0111} mode, Whispering Gallery mode resonator $TE_{01\delta}$ Mode, and Split Post Dielectric Resonator.

Resonant measurement is one of the most accurate dielectric characterisation methods but at the expense of the limited frequencies and low loss characteristics of the materials. There are many types of resonant methods available such as reentrant cavities, split cylinder resonators, cavity resonators, Fabry-Perot resonators etc. There are two types of resonant measurements commonly used. Perturbation methods are suitable for all permittivity measurements, magnetic materials and medium to high loss material measurements. Low loss measurement method is a measurement on low loss materials using larger samples. However, the perturbation method is more popular especially using a TM cavity geometry. With resonance characteristics depending on the MUT in a cavity its, quality factor and resonance frequency can be monitored to determine the dielectric parameters. The dielectric properties can be determined by first measuring the resonant frequency and quality factor of an empty cavity. The second step is to repeat the measurement after filling the cavity with the MUT. The permittivity or permeability of the material can then be computed using the frequency, volume and q-factor.

Advantages of resonant method
– Ability to measure very small MUT.
– Use of approximate expression for fields in sample and cavity.

Disadvantages of resonant method
– Need high frequency resolution VNA.
– Limited to narrow band of frequencies only.

6.4.3 Dielectric Resonator

The dielectric resonator typically consists of a metallic cavity and a dielectric material of cylindrical shape (Figure 6.3). The measured loss consists of loss from the metallic walls and dielectric loss. By properly designing the cavity, the loss from the lateral walls can be eliminated. Also by using low loss materials like superconducting materials as the end plate, all the losses associated with the cavity can be substantially reduced and hence the measured loss will be from the material under test. The Dielectric resonator technique with High Temperature Superconducting (HTS) plates

is a modification of the metallic dielectric resonator and the cavity perturbation techniques used in the past to characterize dielectric materials. The dielectric sample to be measured was enclosed in a copper cylindrical cavity between metallic (or two High Temperature Superconducting films). As the dielectrics under test exhibit low relative permittivity, the diameter 'd' of the samples was chosen to be sufficiently large to ensure that electromagnetic fields are evanescent in the air region.
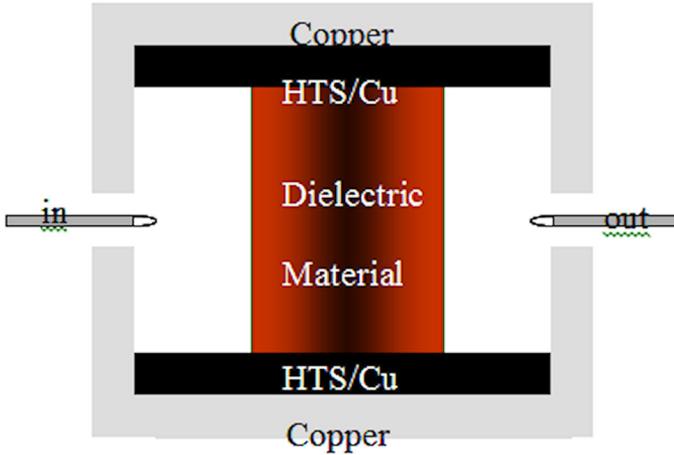
Figure 6.3 Schematic of a TE$_{011}$ mode dielectric resonator.

Since the materials under test are isotropic, the TE$_{011}$ mode of operation was employed in the measurements. The real part of relative permittivity ε_r of a dielectric was determined as the first root of the following transcendental equation (Jacob, *et al.* 2002):

$$k_{\rho 1} J_0(k_{\rho 1} b) F_1(b) + k_{\rho 2} J_1(k_{\rho 1} b) F_0(b) = 0 \tag{6.15}$$

where:

$$F_0(\rho) = I_0(k_{\rho 2}\rho) + K_0(k_{\rho 2}\rho) \frac{I_1(k_{\rho 2}a)}{K_1(k_{\rho 2}a)}$$

$$F_1(\rho) = -I_1(k_{\rho 2}\rho) + K_1(k_{\rho 2}\rho) \frac{I_1(k_{\rho 2}a)}{K_1(k_{\rho 2}a)} \tag{6.16}$$

$$k_{\rho 1}^2 = \frac{\omega^2 \varepsilon_r}{c^2} - k_z^2, \quad k_{\rho 2}^2 = k_z^2 - \frac{\omega^2}{c^2}, \quad k_z = \pi / L$$

and ω is the angular frequency ($2\pi f$), c is velocity of light, ε_o is free space permeability, ε_r is real relative permittivity of the sample and J_0, J_1, I_0, I_1, K_0, K_1, denote corresponding Bessel and Hankel functions.

The loss tangent of a dielectric under test is found on the basis the loss equation of the resonator and measured values of the Q_0-factor of the resonator, namely:

$$\tan \delta = \frac{1}{\rho_e}\left[\frac{1}{Q_0} - \frac{R_{SS}}{A_S} - \frac{R_{SM}}{A_M}\right] \tag{6.17}$$

where Q_0 is measured unloaded Q-factor of the entire resonant structure, R_{SS} and R_{SM} are the surface resistance of the end and lateral metallic walls of the cavity respectively, A_S and A_M are the geometric factors of the end and lateral metallic walls of the cavity and r_e is the electric energy filling factor.

Geometric factors A_S, A_M, and r_e to be used in were computed using incremental frequency rules as follows:

$$A_S = \frac{\omega^2 \mu_0}{4} / \frac{\partial \omega}{\partial L} \tag{6.18}$$

$$A_M = \frac{\omega^2 \mu_0}{2} / \frac{\partial \omega}{\partial a} \tag{6.19}$$

$$p_e = 2\left|\frac{\partial \omega}{\partial \varepsilon_r}\right|\frac{\varepsilon_r}{\omega} \tag{6.20}$$

6.4.4 Dielectric Post Resonator

The accuracy of measurements using HCDR (Jacob, *et al.* 2002, Jacob, *et al.* 2005) is high but the main difficulty is to machine samples precisely to realize the same height as that of the copper cavity. Also the linear thermal expansion coefficient of the metallic wall is different from that of the dielectric material. This constitutes a problem especially during low temperature measurement. Hence a TE_{01d} mode post dielectric resonator is suitable. The dielectric under test is placed over a low loss support material. The DP resonator can be used for characterizing the material at different frequencies by exciting the higher order modes. The electromagnetic field distribution of TE_{011} mode is shown in Figure 6.4. The field distribution studies will assist to estimate the permittivity of the material at different frequencies. This technique also needs machining of the sample but the height of the sample is more

flexible. This technique can be used for timber or fresh biological samples machined in cylindrical shape.
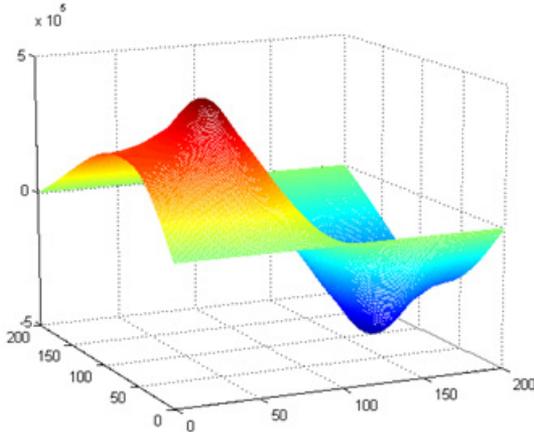
Figure 6.4: H_r Fields for different quasi TE_{0mn} modes in MgF_2 loaded DP resonator (Jacob, et al. 2005).

6.4.5 Whispering Gallery Mode Resonator

The Whispering Gallery Mode (WGM) Resonators (Krupka, *et al.* 1999) enable the most accurate measurements of very low loss dielectric materials. The WGM technique is a very complex one and requires large dielectric samples of specific shapes. WGM describes the electromagnetic wave that circulates around the inner surface of a dielectric sphere or cylinder as the result of total internal reflection. At millimeter wavelengths, the conventional dielectric resonators (as listed above) operate in their TE or TM modes have quite small dimensions, lower Q-factor and are difficult to machine. The WGM operates at higher frequencies and the Q-factor is very high. But it is difficult to identify the correct modes and hence it is advisable to know the approximate value of the permittivity before using WGM resonators for microwave characterization using another technique.

6.4.6 Open-ended co-axial probe method

The open ended co-axial probe method (Gabriel, *et al.* 1996, Ellison and Moreau 2008, Technologies 2010, Yaw 2012) is a non-destructive method and the method assumes only the TEM or TE mode is propagating. In this method the probe is pressed against a specimen or immersed into the liquids and the reflection coefficient is measured

and used to determine the permittivity. Since the sample must be in contact with the probe, it is critical to have the sample polished to avoid air gaps for solid specimens. In many cases it is not possible to machine or prepare the samples so that we can use some techniques such as dielectric resonator or waveguide. This is especially important in the case of biological specimens to perform in-vivo measurements because the material characteristics may change. Therefore, with this method the sample can be placed in close contact with the probe without causing any changes in the material properties.

The reflection coefficient is measured using a vector network analyzer (VNA). The VNA with a probe system is first calibrated so that the reflection coefficient measurements are referenced to the probe's aperture plane. This can be done by using reference liquids for direct calibration at the open end of the probe. Though simple, the uncertainties in the measurement are due to the uncertainties in the characterization of the reference liquids and the selection of reference liquids as calibration standard. In this method, all measurements are performed by placing the standards (a short, an open and a referenced liquid) at the end of the probe. The referenced liquid is used as a calibration standard and must be a liquid with known dielectric properties. Water, saline and methanol are usually selected as the reference liquids. Standard one port full calibration is then applied. The s-parameters measured on the MUT can be post-processed to obtain the dielectric parameters using a program.

Advantages of open ended coaxial probe method
– Require no machining of the sample, easy sample preparation.
– After calibration, the dielectric properties of a large number of samples can be routinely measured in a short time.
– Measurement can be performed in a temperature controlled environment.
– Disadvantages of open ended coaxial probe method
– Only reflection measurement available.
– Affected by air gaps for measurement on specimen.

6.4.7 Dielectric Probe (Coaxial probe)

The open-ended coaxial probe is a cut off section of transmission line (Figure 6.5). This technique is ideal for a variety of materials including solids and liquids, and also for broadband measurement. Over all, the design is rugged and can withstand many physical conditions especially a wide range of temperatures.
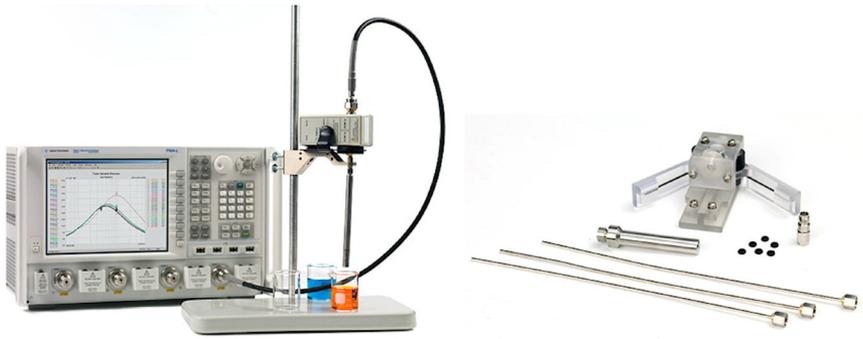
Figure 6.5: Dielectric Probe Setup (Agilent).

Most of the characterization techniques discussed so far are very good for a single frequency. In order to evaluate the sample over a wide range of frequencies wave guide techniques or dielectric probe (Agilent) techniques could be used. The open-ended coaxial probe is a cut off section of transmission line. The material is measured by touching the flat face of a solid material to the probe. The EM fields at the probe end penetrate into the material under test and the reflected signal (S_{11}) can be measured. The complex permittivity is computed from the reflected signal. However the accuracy and repeatability of the dielectric probe technique is very poor.

The Dielectric Probe (Figure 6.6) was used to measure the complex permittivity over a wide range of frequencies, thereby allowing the response over various frequencies for a particular dielectric to be viewed. It is a convenient measurement technique as it does not require manipulating the geometry of the dielectric material to be tested, however, the accuracy in the permittivity measurement is poor.
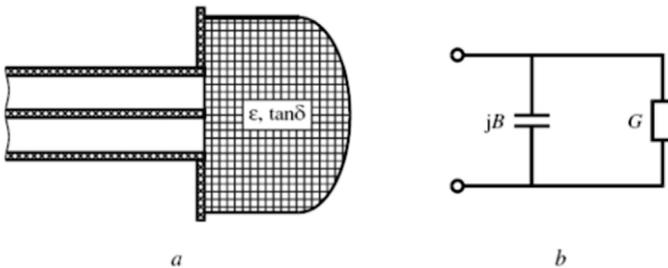
Figure 6.6: a – Schematic of a dielectric probe with open end, b – equivalent circuit of dielectric probe (Mekhannikov, et al. 2007).

The principle of operation involves the propagation of a TEM travelling wave, launching fringing EM fields from the open end, into the dielectric material. The reflection coefficient can be related to the complex permittivity by using; modal analysis of the fields in the transmission line, or by analyzing the fields in the dielectric material. Equations below govern the determination of permittivity and loss tangent for a dielectric probe. (Mekhannikov, *et al.* 2007)

$$\varepsilon = \frac{2T \sin\left[2B + \frac{4\pi\left(L_2 - L_1\right)}{\lambda}\right]}{B\left\{1 + T^2 + 2T \cos\left[2B + \frac{4\pi\left(L_2 - L_1\right)}{\lambda}\right]\right\}} \tag{6.21}$$

$$\tan\delta = \frac{1 - T^2}{2T \sin\left[2B + \frac{4\pi\left(L_2 - L_1\right)}{\lambda}\right]} \tag{6.22}$$

where:

T - Is magnitude of reflection coefficient of the probe with sample.

B - Standing wave ratio.

The dielectric probe technique calculates the dielectric properties from the phase and amplitude of the reflected signal at the end of an open-ended coaxial line inserted into a sample to be measured. The coaxial probe has a tip used for sensing the signal reflected from the material. The tip is brought into contact with the substance by touching the probe to a flat face of a solid or by immersing it in a liquid. This method is easy to use and makes it possible for the dielectric properties to be measured over a wide range of frequencies (500 MHz – 110 GHz). The operations of the open-ended coaxial lines to measure the dielectric constant of unknown materials is documented in papers by Athey, Stuchly, & Stuchly. The input reflection coefficient at the probe tip is given by:

$$\rho = \frac{Z_L - Z_o}{Z_L + Z_o} = \frac{Y_o - Y_L}{Y_o + Y_L} \tag{6.23}$$

where Z_0 is the line impedance and Z_L is the load impedance. When placed in contact with a homogenous material whose thickness is sufficient to simulate a slab of infinite electrical thickness, an open coaxial line has an admittance, Y_L, of:

$$Y_L\left(\omega, \varepsilon\right) = Y_i\left(\omega\right) + Y_e\left(\omega, \varepsilon\right) \tag{6.24}$$

This admittance value is comprised of two terms; the first is the internal admittance, Y_i, corresponding to the fringing capacitance that accounts for the fringing field in the Teflon region between the inner and outer conductors of the line. The second term is the external admittance, Y_e, which is a function of the frequency and the dielectric constant of the material being examined. A calibration is required when operating the measurement probe in order to define values concerned with the capacitance, conductance and geometry of the probe. This enables an iterative process to find a value for the dielectric constant, ϵ.

Ulaby and El-Rayes operated the dielectric probe over a wide frequency range extending from 0.1 GHz up to 20 GHz, which requires a small radii probe, and also there is a need to have strong sensitivities to variations in the dielectric properties, which require a probe with a large radii.

One of the typical issues is to obtain good surface contact and hence the measured reflection may not represent full reflectance from the sample. Surface preparation of solid samples must be done very carefully to insure that no air gaps remain where the probe is applied. The change in reflected signal is dependent on the electrical properties of the impedance which terminates the line. In the case of the coaxial probe, the sample material terminates the line and its properties are mirrored in the reflection coefficient.

6.4.8 Free Space Method

The free space method (Courtney 1970, Athey, *et al*. 1982, El-Rayes and Ulaby 1987, Ghodgaonkar, *et al*. 1990, Amiet and Jewsbury 2000, Trabelsi and Nelson 2003, F.H.Wee, *et al*. 2009, Juan-García and Torrents 2010, Cataldo, *et al*. 2011) is for broadband applications and assumed only the TEM propagation mode. Free space technique is good for materials that can remain unmodified for characterization purposes. Free space metrology is used for characterizing materials in an un-enclosed environment (Figure 6.7). Reflective mirrors are set up, such that diverging beams are directed onto a piece of dielectric material. Though surface modification or separate enclosure is not required, measurements performed in open space will increase possibility of obtaining inaccurate results.

Free space measurement allows measurements on MUT under many environmental or physical conditions such as high temperatures and wide band frequencies. The measurement requires the MUT to be large and flat. It usually utilizes two antennas placed facing each other and the antennas are connected to a network analyzer. Before starting the measurement, the VNA must first be calibrated. There are a number of calibration methods that can be used, such as the through-reflect-line (TRL), the through-reflect-match (TRM) and the line-reflect-line (LRL). However, the LRL calibration method can produce the highest calibration quality. The line standard can be achieved by separating the focal plane of the two antennas to approximately a

quarter of wavelength. The reflect standard can be obtained by placing a metal plate on the sample holder in between the antennas. Once calibrated, the s-parameters of an empty sample holder are measured by placing the sample holder midway between the two antennas. The MUT is then placed on the sample holder between the antennas and the s-parameter measurement is performed again. Using the de-embedding function of the VNA, the influence of the sample holder can be cancelled out and only the s-parameter of the MUT can be determined. The s-parameter for both the reflection and transmission coefficients can be determined.

Time domain gating should also be applied to ensure there are no multiple reflections in the sample itself, though appropriate thickness should able to avoid this. It also eliminates the diffraction of energy from the edge of the antennas. The dielectric properties can be determined by post processing the measured reflection and transmission coefficient using a program.

Advantages of free space method
– Can be used for high frequency measurement.
– Allows non-destructive measurement.
– Measure MUT in hostile environment.
– Both the magnetic and electric properties can be evaluated.

Disadvantages of free space method
– Need large and flat MUT.
– Multiple reflections between antenna and surface of sample.
– Diffraction effects at the edge of sample.

6.4.9 Antenna

In the case of many materials it is hard to use dielectric probe or cavity resonators because the probes cannot be kept in contact. For materials such as vegetables or soils etc, a non-destructive technique involving a pair of horn antennae can be used; this is a free-space method operating in the far-field region employing spot-focusing horn lens antennae. This technique measures reflection coefficients and from the measured data dielectric constants, loss factors, and complex permeability as a function of frequency (microwaves) can be estimated.

This technique is suitable for precise, accurate and reproducible microwave measurements on materials under various environmental conditions and complex electromagnetic environmental conditions due to contactless feature of free-space measurements. Composite materials such as timber, vegetable and soil which are lossy and anisotropic cause a linearly polarized electromagnetic field to be depolarized (i. e. elliptically polarized) upon transmission through the material.

The measurement system consists of a pair of spot-focusing horn lens antennas, mode transitions, coaxial cables and a vector network analyzer. The inaccuracies in

free-space measurements are due to two main sources of errors. The spot-focusing antennae are used for minimizing diffraction effects and free-space LRL (line, reflect, line) calibration method implemented on VNA eliminates errors due to multiple reflections. The time domain gating or smoothing feature of VNA is used to reduce post calibration errors in reflection and transmission measurements. The main errors are due to:

i. Diffraction effects at the edges of the material specimen/sample.
ii. Multiple reflections between horn lens antennas and mode transitions via the surface of the sample.

The sample is kept in between the two focused antennas, which are connected to the two ports of the vector network analyzer by using precision coaxial cables, rectangular-to-circular waveguide adapters and coaxial-to-rectangular waveguide adapters.
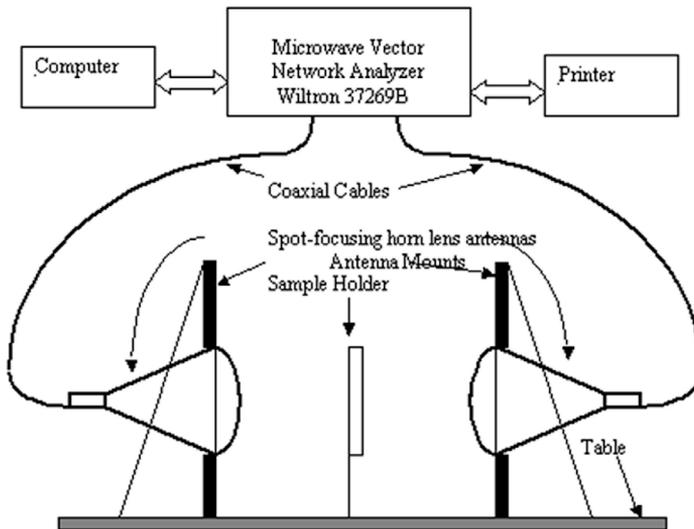
Figure 6.7: Microwave Nondestructive Testing using Free-Space Microwave Measurement Techniques (Ghodgaonkar, et al. 1990).

6.4.10 Near-field Microwave Probe

Local properties of thin films can be measured by focusing the EM fields on to the film surface. This can be done by using a near field microwave probe (Gao and Xiang 1998), which scans thin films or measures submicron materials because the resolution can be much smaller than $\lambda/2$. The near-field microwave probe usually consists of

a resonator connected to a probe on the bottom, so that as the probe approaches a material, it shifts the cavity resonance. The measurement requires measurement of resonant frequency and Q with and without the specimen and analysis of the shift in measured parameters. In order to obtain precise accuracy in measurement a theoretical and numerical model that relates shifts in the system's cavity resonator to the material under test is needed.

6.4.11 Reentrant Cavity

The reentrant cavity consists of a coaxial line or other transmission line with a gap in the inner electrode. The specimen is inserted into this gap. The cavity is then resonated and the capacitance of the gap produces a frequency shift. Depends on the specimen gap region the cavity can be a singly reentrant cavity or a doubly reentrant cavity. The resonant reentrant cavity typically estimates the permittivity component normal to the face of the material. This Technique is ideal for low microwave frequency region.

6.4.12 Fabry-Perot Resonator

Fabry-Perot Resonators (Courtney 1970, Clarke and Rosenberg 1982) are characterised by very high Q factors (typically around 200,000) and are ideal for characterisation of low loss materials in the millimetre range region. Fabry-Perot Resonators, a form of open resonator, uses two sufficiently large mirrors to direct a Gaussian beam of light at a sample material to determine the complex permittivity. In the confocal setup both mirrors are concave, whereas in the semi-confocal arrangement only one of the mirrors is concave and the other is flat Since they are open structures they suffer from leakage of radiation. This can be reduced by large samples such that the Gaussian beam incidents the sample at full strength which is usually taken to be an integral multiple of ½ the wavelength (λ). The tensor permittivity values can be obtained by measuring at different angles of incidence. When combined with a 'Bragg Reflector' these resonators become excellent at measuring complex permittivity of gasses.

6.5 Conclusions

Figure 6.8 shows the different types of measurement technique used and the feasibility of methods as a function of frequency. Based on the nature of samples required for this project, dielectric probe and free-space measurement using transmitting and reflecting antenna are ideal measurement systems for biological samples and plant materials. Table 6.1 shows the dielectric constant of many materials.
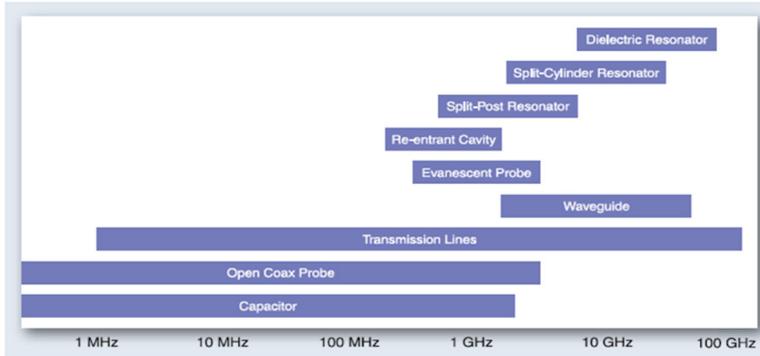
Figure 6.8: Different microwave characterization techniques used as a function of frequency (Baker-Jarvis, et al. 2010, Yaw 2012).

Table 6.1: The real part of the Dielectric Constant of Various Materials.

Material Name	Relative Dielectric Constant	Material Name	Relative Dielectric Constant
Acetic acid	6.2	Fat	16
Air	1	Fibre	5
Alcohol, ethyl (grain)	24.55	Flour (dry)	4.1 - 6.2
Alcohol, methyl (wood)	32.7	Formica	3.6
Amber	2.6 - 2.7	Freon 12 (vapour)	2.4
Asbestos	4	Freon 12 (liquid)	3.5
Asbestos fibre	3.1 - 4.8	Germanium	16
Asphalt	2.6	Glass	5.0 - 10.0
Bakelite	5	Glass pyrex	4.6
Barium titanate	100	Glycerine	42.5
Beeswax	2.4	Gutta percha	2.4
Benzene	2.284	Isolantite	6.1
Bone, cancellous	26	Jet fuel (jet a)	1.7
Bone, cortical	14.5	Kevlar	3.5 - 4.5
Brain, gray matter	56	Lead magnesium niobate	100
Brain, meninges	58	Lead oxide	25.9
Brain, white matter	43	Lead sulfide (galena)	200
Calcite	8	Lead titanate	200
Calcium carbonate	8.7	Liquid ammonia(-78°C)	25

_{Continued}**Table 6.1:** The real part of the Dielectric Constant of Various Materials.

Material Name	Relative Dielectric Constant	Material Name	Relative Dielectric Constant
Cambric	4	Lithium deuteride	14
Carbon tetrachloride	2.17	Lucite	2.5
Cartilage, ear	47	Mica	4
Cartilage, general	22	Mica, canadian	6.9
Celluloid	4	Mica, muscovite	5.4
Cellulose	3.7 - 7.5	Micarta	3.2 - 5.5
Cellulose acetate	2.9 - 4.5	Muscle, smooth	56
Cement	~2	Muscle, striated	58
Cocaine	3.1	Mycalex	7.3
Cotton	1.3	Mylar	3.1
Diamond, type I	5.87	Neoprene	4
Diamond, type iia	5.66	Nylon	3.4 - 22.4
Durite	4.7 - 5.1	Oil, linseed	3.4
Ebonite	2.7	Oil, mineral	2.1
Epoxy resin	3.4 - 3.7	Oil, olive	3.1
Ethyl alcohol	6.5 - 25	Oil, petroleum	2.0 - 2.2
Eye, aqueous humor	67	Oil, silicone	2.5
Eye, cornea	61	Oil, sperm	3.2
Eye, sclera	67	Oil, transformer	2.2
Paper	3.3, 3.5	Slate	7
Paraffin	2	Soil	44
Plexiglas	3.4	Soil dry	2.4
Plexiglass	2.6 - 3.5	Steatite	5.2
Polycarbonate	2.9 - 3.2	Strontium titanate	332
Polyester	3.2 - 4.3	Styrofoam	1.03
Polyethylene	2.25 - 2.5	Sulfur	3.7
Polyimide	3.4	Tantalum pentoxide	27
Polypropylene	2.2 - 2.3	Teflon	2.1
Polystyrene	2.55	Tin antimonide	147
Polystyrene	2.4	Tin telluride	70
Polyvinyl chloride	3.18 - 4.5	Titanium dioxide (rutile)	114

_{Continued}**Table 6.1:** The real part of the Dielectric Constant of Various Materials.

Material Name	Relative Dielectric Constant	Material Name	Relative Dielectric Constant
Porcelain	4.0 - 8.0	Tobacco	1.6 - 1.7
Potassium niobate	700	Tongue	38
Potassium tantalate niobate, 20°C	6000	Uranium dioxide	24
Quartz	5	Vacuum	1
Quartz, crystalline	4.5 - 4.6	Vaseline	2.16
Quartz, fused	3.8	Vinylite	2.7 - 7.5
Rubber	2	Water	80.4
Rubber, butyl	2.4	Water distilled	34
Rubber, neoprene	6.6	Water, ice, −30°C	99
Rubber, silicone	3.2	Water, liquid, 0°C	87.9
Rubber, vulcanized	2.9	Water, liquid, 100°C	55.5
Ruby mica	5.4	Water, liquid, 20°C	80.2
Salt	5.9	Water, liquid, 40°C	73.2
Selenium	6	Water, liquid, 60°C	66.7
Shellac	2.9 - 3.9	Water, liquid, 80°C	60.9
Silicon	11.8	Wax, beeswax	2.7 - 3.0
Silicon carbide (αsic)	10.2	Wax, carnuba	2.9
Silicon dioxide	4.5	Wax, paraffin	2.1 - 2.5
Silicone	3.2	Waxed paper	3.7
Silicone oil	2.7 - 2.8	Waxes, mineral	2.2
Skin	33 - 44	Wood dry	1.4 - 2.9

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