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EXPERIMENTAL TECHNIQUES FOR THE STUDY OF DIELECTRIC PROPERTIES OF MATERIAL

2.1 INTRODUCTION

There are various techniques, which could be used to study dielectric properties. The dielectric data are useful for telecommunication, lenses, radios and microwave integrated circuits, wave guides, lossy ceramics for microwave absorbers, lossy plaster for the design of food packages to put in microwave ovens for the purpose of heating, for diathermy and for the study of molecular structure of substances. The water content in crude oil, moisture in wood, sand, agriculture products are also the examples, where dielectric data are required.

The microwave energy in the frequency region from 300 MHz to 30 GHz has been used for many applications. As we are aware that the matter may exist in the gaseous state, liquid state, also it may have powder form, thick paste or different shapes such as sheets, grains, flat or rocks, therefore, different techniques are obligatory for dielectric measurements. Also, not one experimental technique is sufficient to measure the complex dielectric constant $(\varepsilon' = \varepsilon' - j\varepsilon'')$ as the dielectric permittivity $(\varepsilon')$ varies from 2.0 to several thousands and the dielectric loss $(\varepsilon'')$ varies from its fraction to several hundreds. Therefore, to understand all possible aspects, it is useful to consider a technique, which is most suited [1,2] and is fruitful from the
application point of view in addition to summarizing and referring to other methods [3,4]. The time domain method [5] is used where in for applications of dielectric data are required over a wider range of frequencies from 100 MHz to 20 GHz.

In view of understanding the physical basis of the dielectric relaxation, the response of dielectric loss ($\varepsilon''$) and of the dielectric permittivity ($\varepsilon'$) with temperature needs to be measured from the lowest possible temperature to its highest value are as accurate as possible. However, if the dielectric data with about 10% accuracy are available, the purpose for industrial, scientific, medical or many other applications proposed could be fulfilled but for the basic research and for the molecular structure study, precise and accurate data within one percent are inevitable.

2.2 EXPERIMENTAL TECHNIQUES

The experimental techniques can be classified mainly in two categories such as (i) Frequency domain and (ii) Time Domain. In the frequency domain, the experimental techniques are:

I. Free space propagation
II. Transmission line method
III. Automatic Network Analyzer
IV. Cavity perturbation.

These methods are further subdivided. The free space propagation into (a) reflection type and (b) transmission type among which the transmission line method includes (a) the use of waveguides and (b) use of coaxial line. The waveguides may be rectangular or circular. The free space methods are preferred for high temperature measurements and in this method it is not necessary to the machine with the sample
for the purpose to fit it exactly in the waveguide or cavity. Since, frequencies of the order of 2.50 GHz are required for heating and other applications, the free space method is not very much suitable for large number of applications. In the waveguide method, one needs a large quantity of sample and big size temperature controlled oven for high temperature measurements in addition to large dimensions of waveguide. Therefore, waveguide methods are also not very useful for these applications. However, if measurements are required at frequencies of the order of about 250 GHz, waveguides of very small dimensions are designed, but this results to high transmission losses. The cavity perturbation can be done into (a) single mode cavity and (b) multi-mode cavity. The single mode cavity may be rectangular, cylindrical, re-entrant and helical. The above-mentioned methods have been used for the last about fifty years except the automatic network analyzer, which has been recently exploited for the study of molecular structure and basic researches. The automatic network analyzer is classified as (a) transmission line method, since the sample is used in a transmission line and the dielectric response is found by the measurement of reflection or transmission coefficient or both. This method has emerged as one of the powerful modern methods with which, one can experimentally study dielectric responses over a wide range of frequencies in single measurement. (b) The scattering parameters are measured with a vector network analyzer consisting of a synthesized sweeper and an S-parameter test set. In this technique of dielectric measurement, there is an arrangement for data acquisition, printing of S-parameters and automation. In a vector network analyzer, errors in measurements are reduced by a calibration procedure significantly and there are many other methods to reduce these errors. Use of wide band sweepers and control by computer, dielectric data in a wide range of frequencies
could be obtained by this technique with minimum possible uncertainty in measurements.

The experimental techniques mentioned-above are related to frequency domain, however, there are Time domain experiments, which are subdivided as (a) Transmission, (b) Reflection and (c) Ratio. There may be single transmission, multiple transmission, single reflection or multiple reflection. These methods are growing in their use, because they provide dielectric data in the wide range of frequencies extending to 20 GHz and also for the consumption of very small volume of the sample for dielectric response, so as costly sample can be studied easily by this method.

2.3 MICROWAVE CAVITY SPECTROMETER

The microwave absorption has been studied so widely after the Second World War, when the necessary components for the observations became available. Among various microwave measurement techniques, the simplest one is to use the microwave bench with slotted line to measure the voltage standing wave ratio (VSWR) and thereafter derive equations for the dielectric permittivity and dielectric loss under different circumstances. The cavity perturbation technique is one of the most accurate method to study the absorption using cavity resonance. This resonance technique is used to study the acoustic properties of rooms, electron densities and collision frequencies of plasmas, phase transition studies in polar and non-polar liquids, ferroelectric – paraelectric phase transition in some crystalline solids, dielectric properties of ferroelectrics and biological molecules, gas phase system, liquid crystals, multi-photon ionization of highly excited hydrogen atom, electron scattering resonance, dielectric response of germinating seeds and in the study of quantum chaos.
in Billiards. Slater [6] and Johri et al [7] have shown that the ionic properties and the relaxation processes with in a sample of material loaded in a resonant cavity can be related to the change in resonant frequency and the change in quality factor (Q) of cavity when the sample is inserted in the cavity. The quality factor of the resonator, in a desired frequency range, is defined as 
\[ Q = \frac{\nu}{\Delta \nu}, \]
where \( \nu \) is the resonant frequency in that range and \( \Delta \nu \) is the typical width. The two successive resonant frequencies can be resolved, if they are separated by at least \( \Delta \nu \). A simple calculation shows that the total number of resonant frequencies can be regarded as of the order of \( Q_0 \).

The cavity perturbation technique has been used in variety of applications discussed in the above paragraph. Slater [6] has given the cavity perturbation equations involving shift in the resonant frequency and change in the width of the resonance profile. We give here in the ensuing paragraph the Slater perturbation theory [6].

### 2.3.1 Slater Perturbation Theory

A hollow cavity forms the analogy for a circuit element just as a waveguide forms the microwave transmission line of ordinary circuit theory. A cavity can be provided with one or more output leads. In general, in the theory of resonant cavities and the electromagnetic fields within them, one can consider an arbitrary number of output leads. In order to solve the Maxwell’s equations in a hollow cavity, one must have to apply certain boundary conditions around the surface. The Maxwell’s equations within a volume bounded by a certain surface based on two types of boundary conditions: the first, which may be called as the short circuited boundary conditions, requires that the tangential component of \( \mathbf{E} \) (i.e. \( E_{\text{tang}} \)), and the normal
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component of \( \mathbf{H} \) (i.e. \( \mathbf{H}_\text{norm} \)), be zero on the surface, while the second, which is called as the open circuited boundary conditions, requires that the normal component of \( \mathbf{E} \) (i.e. \( \mathbf{E}_\text{norm} \)), and the tangential component of \( \mathbf{H} \) (i.e. \( \mathbf{H}_\text{tang} \)), be zero on the surface. The reason behind this nomenclature is that a perfect conductor has zero tangential component of \( \mathbf{E} \), and forms the analog of a short circuit; while a perfect insulator carries no surface current, and hence, if \( \mathbf{H} \) is zero within it, it demands a zero tangential component of \( \mathbf{H} \), and forms the analog of an open circuit. We can use mixed boundary conditions over the part of the surface (denoted by \( S \)) for short-circuited boundary conditions, while over the rest of the surface, which is denoted by \( S' \), we have open circuited boundary conditions. In order to setup the orthogonal functions within the volume \( V \) bounded by \( S \) and \( S' \) suitable for expanding our fields within the volume.

By the general principles of vector analysis, any vector field can be broken up into field two fields, one of which is solenoidal, or has zero divergence, and the other of which is irrotational, or has zero curl. The electric field \( \mathbf{E} \) has been expanded by denoting the solenoidal functions as \( \mathbf{E}_a \) and the irrotational functions as \( \mathbf{F}_a \). Since, the magnetic field has no irrotational functions, hence, the solenoidal functions are used in expanding the magnetic field \( \mathbf{H} \) is represented by the symbol \( \mathbf{H}_a \). The functions \( \mathbf{E}_a \) and \( \mathbf{H}_a \), having no divergence, must be the curls of certain other vector functions. It is assumed that they satisfy the equations

\[
k_a \mathbf{E}_a = \text{curl} \mathbf{H}_a \quad (2.1)
\]

\[
k_a \mathbf{H}_a = \text{curl} \mathbf{E}_a \quad (2.2)
\]
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Where \( k_a \) is propagation constant \((= 2\pi/\lambda)\), associated with the \( a^{th} \) mode. Also, it is assumed that \( E_a \) and \( H_a \) satisfy the following boundary conditions:

\[
\mathbf{n} \times \mathbf{E}_a = 0 \quad \text{on } S \quad (2.3)
\]
\[
\mathbf{n} \times \mathbf{H}_a = 0 \quad \text{on } S' \quad (2.4)
\]

Where \( \mathbf{n} \) is the unit vector outer normal to the surface. In other words \( E_a \) has no tangential component over \( S \), and \( H_a \) has no tangential component over \( S' \). So, by using the Stoke's theorem on the above equations, it can be expressed as

\[
\mathbf{n} \cdot \mathbf{E}_a = 0 \quad \text{on } S \quad (2.5)
\]
\[
\mathbf{n} \cdot \mathbf{H}_a = 0 \quad \text{on } S' \quad (2.6)
\]

In order to setup separate differential equations for \( E_a \) and \( H_a \) instead of having them defined in terms of each other in Eq. (2.1) and (2.2) we can write these equations as

\[
\text{curl curl } \mathbf{E}_a = k_a^2 \mathbf{E}_a \quad (2.7)
\]
\[
\text{curl curl } \mathbf{H}_a = k_a^2 \mathbf{H}_a \quad (2.8)
\]

Using the vector identity that \( \text{curl curl } \mathbf{A} = \text{grad div } \mathbf{A} - \nabla^2 \mathbf{A} \), and that \( \text{div } \mathbf{E}_a = 0 \) and \( \text{div } \mathbf{H}_a = 0 \), we get the following familiar wave equations

\[
\nabla^2 \mathbf{E}_a + k_a^2 \mathbf{E}_a = 0 \quad (2.9)
\]
\[
\nabla^2 \mathbf{H}_a + k_a^2 \mathbf{H}_a = 0 \quad (2.10)
\]
These equations may be assumed to have an infinite set of solutions, corresponding to
different values of \( k_a \), subject to the boundary conditions Eq.(2.3). For each value of
\( k_a \), we have both functions \( E_a \) and \( H_a \). Both the functions correspond to the same set
characteristic numbers. The functions \( E_a \) and \( H_a \) have orthogonality properties of the
form

\[ \int_V E_a E_s \, dv = 0 \]  \hspace{1cm} (2.11)

\[ \int_V H_a H_s \, dv = 0 \]  \hspace{1cm} (2.12)

We can expand the electric and magnetic fields, and other related quantities, inside a
hollow cavity in terms of the orthogonal functions and by means of Maxwell’s
equations to find relations between expansion coefficients. The Maxwell equations are

\[ \text{curl} \, E + \frac{\partial B}{\partial t} = 0, \quad \text{div} \, B = 0 \quad \text{if} \quad a \neq b \]  \hspace{1cm} (2.13)

\[ \text{curl} \, H - \frac{\partial D}{\partial t} = J, \quad \text{div} \, D = \rho \quad \text{if} \quad a \neq b \]  \hspace{1cm} (2.14)

Where it is assumed that the wave guide is empty, so that within it we have

\[ D = \varepsilon_0 E, \quad B = \mu_0 H, \quad J = 0, \quad \rho = 0. \]  \hspace{1cm} (2.15)

Here, \( E \) is measured in V/m, \( D \) in Coul/m\(^2\), \( H \) in Amp-turns/meter, \( B \) in Weber/m\(^2\), \( J \)
in Amp/m\(^2\), \( \rho \) in Coul/m\(^3\). The quantities \( \varepsilon_0 \) is in farad/m and \( \mu_0 \) in henry/m and
\((\mu_0/\varepsilon_0)^{1/2} = 376.6 \text{ ohms, and } (\varepsilon_0 \mu_0)^{-\frac{1}{2}} = c \text{ (speed of light) } = 3.00 \times 10^8 \text{ m/s, where the}
symbols used are having their own meaning.}
Also, we get the following series:

\[
E = \sum_a (E_a \int E \cdot E_a \, dv + F_a \int E \cdot F_a \, dv) \quad (2.16)
\]

\[
H = \sum_a H_a \int H \cdot H_a \, dv \quad (2.17)
\]

\[
J = \sum_a (J_a \int J \cdot E_a \, dv + F_a \int J \cdot F_a \, dv) \quad (2.18)
\]

\[
\rho = \sum_a \psi_a \int \rho \psi_a \, dv \quad (2.19)
\]

Using these series of \( E, H, J, \) and \( \rho \) and Maxwell's equation \( \text{curl} \ E + \frac{\partial B}{\partial t} = 0, \) we have

\[
k_a \int E \cdot E_a \, dv + \mu_0 \frac{d}{dt} \int H \cdot H_a \, dv = - \int (n \times E) \cdot H_a \, da \quad (2.20)
\]

and from \( \text{curl} \ H - D = J \) we have,

\[
k_a \int H \cdot H_a \, dv - \varepsilon_0 \frac{d}{dt} \int E \cdot E_a \, dv = - \int J \cdot E_a \, dv - \int (n \times H) \cdot E_a \, da \quad (2.21)
\]

\[
- \varepsilon_0 \frac{d}{dt} \int E \cdot F_a \, dv = \int J \cdot F_a \, dv \quad (2.22)
\]

Now, combining the Eq. (2.20) and (2.21), we get separate equations for \( \int E \cdot E_a \, dv \) and \( \int H \cdot H_a \, dv \) i.e.

\[
\varepsilon_0 \mu_0 \frac{d^2}{dt^2} \int E \cdot E_a \, dv + k^2_a \int E \cdot E_a \, dv
\]

\[
= -\mu_0 \frac{d}{dt} \left( \int J \cdot E_a \, dv - \int (n \times H) \cdot E_a \, da \right) - k_a \int (n \times E) \cdot H_a \, da \quad (2.23)
\]
Equations (2.23) and (2.24) form the basis of most of our treatment of resonant cavities. They show that $\int E \cdot E_a dv$ and $\int H \cdot H_a dv$ are determined as functions of time by the type of differential equations encountered in elementary problems of simple harmonic motion. The terms on right-hand sides take the place of the external force in the problem of simple harmonic motion, so that we may get solutions of Eq.(2.23) and (2.24) showing the properties of forced oscillations and resonance, the forced motion resulting from the currents within the cavity, or from disturbances propagated from the walls. Equations (2.23) and (2.24) derived from Eq.(2.20) and (2.21) determine the solenoidal part of $E$ and $H$. The solenoidal part of the field that shows properties of wave propagation, is usually regarded as the radiation field. In further discussion the irrotational effect of $E$ will not be considered.

The Eq.(2.23) and (2.24) determine the coefficients $\int E \cdot E_a dv$ and $\int H \cdot H_a dv$ as a function of time in terms of the integrals, which appear on the right side of the equations. The quantity $\int J \cdot E_a dv - \int (n \times H) \cdot E_a da$ appearing on the right side is one of the components of $J$ (the volume integral), supplemented by the surface integral. In this surface integral $-(n \times H)$ is the tangential component of surface current connected with the discontinuity in the tangential component of $H$ at the surface, so that the surface integral gives us the contribution of the surface currents.

Similarly, the quantity $-\int (n \times E) \cdot H_a$ would have to be interpreted as the integral of $-(n \times E)$, a surface density of fictitious magnetic current, which appears at
a surface of discontinuity of the tangential component of \( \mathbf{E} \). Thus we have the interpretation of the surface integrals appearing in Eq.(2.20) to (2.24) respectively.

For free and damped oscillations of a resonant cavity, we considered Eq.(2.23) and (2.24). Assuming that the cavity contains no current density, so that \( \mathbf{J} = 0 \) and furthermore it is assumed that over that part of the surface \( S \) the tangential component of \( \mathbf{E} \) is zero \( i.e. \ E_{\text{tang}} = 0 \) for a perfect conductor and over \( S' \) tangential component of \( \mathbf{H} \) is zero \( i.e. \ H_{\text{tang}} = 0 \) for a perfect insulator having no surface current flow. The integrals on right side of the Eq.(2.23) and (2.24) vanish and we get following solutions

\[
\int \mathbf{E} \cdot \mathbf{E}_a \, dv = A e^{i \omega_a t} \quad \omega_a^2 \varepsilon_0 \mu_0 = k_a^2 \tag{2.25}
\]

Where \( A \) is some constant with similar solutions for \( \int \mathbf{H} \cdot \mathbf{H}_a \, dv \). The \( \omega_a \)'s are the angular frequencies of the resonant modes. From Maxwell’s Eq.(2.20) and (2.21) we can find the relation between the coefficients \( \int \mathbf{E} \cdot \mathbf{E}_a \, dv \) and \( \int \mathbf{H} \cdot \mathbf{H}_a \, dv \) for the normal modes \( i.e. \)

\[
\frac{\int \mathbf{E} \cdot \mathbf{E}_a \, dv}{\int \mathbf{H} \cdot \mathbf{H}_a \, dv} = -j \left( \frac{\mu_0}{\varepsilon_0} \right)^\frac{1}{2} \tag{2.26}
\]

The Eq.(2.26) shows that the time average electrical energy integrated through the cavity equals the time average magnetic energy. The phase difference, however, results in the magnetic energy being large when the electric energy is small, and \textit{vice versa}, just as with the kinetic and potential energy in simple harmonic motion. Therefore, the total energy is conserved. Also, it is clear from the Eq.(2.26) that the magnitudes of coefficients \( \int \mathbf{E} \cdot \mathbf{E}_a \, dv \) and \( \int \mathbf{H} \cdot \mathbf{H}_a \, dv \) are in the same ratios to each
other as the values of $E$ and $H$ in a plane wave in empty space, but the electric and magnetic fields are at $90^\circ$ apart in phase, a characteristic of standing electromagnetic waves. For damped oscillations of cavity, we should define the quality factor $Q$ of the cavity as the ratio of $2\pi$ times the total energy to the decrease in energy per period. The period means $2\pi/\omega_0$ as determined from the angular frequency, which would exist if the damping were absent.

There are three most common types of perturbations used to produce the damped oscillations in free oscillations of a cavity:

1. The walls of the cavity, instead of being perfect conductors, may have finite conductivity, resulting in resistive losses and $E_{\text{tang}} \neq 0$ over the surface $S$ for damped oscillations. Therefore,

$$\int_S (n \times E) \cdot H_s \, da \neq 0 \quad (2.27)$$

2. The cavity may have certain windows, or wave guide outputs, which allow the escape of energy, with consequent loss of the total energy. in this case the $H_{\text{tang}} \neq 0$ over the surface $S'$ and therefore,

$$\int_S (n \times H) \cdot E_s \, da \neq 0 \quad (2.28)$$

3. The cavity may contain currents in phase with the voltages producing them, as if they contained resistive material obeying Ohm’s law. Hence, in this case

$$\int J \cdot E_s \, dv \neq 0 \quad (2.29)$$

If the above mentioned conditions i.e. Eq.(2.27), (2.28) and (2.29) exist, then there results damped oscillations and the various integrals must be proportional to the
amplitude \( \int E \cdot E_d dV \) of the field component and must oscillate with the same period. In such a case, we may assume the solution of Eq.(2.23) as \( e^{iat} \), where \( \omega \) is in general complex.

Mathematically, under damped oscillation condition for a cavity, we can write

\[
\int \left( \frac{\omega - \omega_0}{\omega_0 - \omega} \right) + \frac{1}{Q} = 0
\]  \hspace{2cm} (2.30)

Which gives

\[
Q = -\frac{1}{j} \left( \frac{\omega \omega_0}{\omega^2 - \omega_0^2} \right)
\]  \hspace{2cm} (2.31)

Where, \( \omega_0 \) is the angular frequency of the free oscillations and \( \omega \) is that for any mode. For small perturbations, we can write the resonant frequency \( \omega_0 \approx \omega_0 \). In such a case, we can consider

\[
\omega_0 = \omega_a + \Delta \omega_a
\]  \hspace{2cm} (2.32)

where \( \Delta \omega_a \) is a small quantity. The Eq.(2.30) may be written in several forms, correct to the first order is

\[
\int \left( \frac{\omega}{\omega_a} - \frac{\omega_a}{\omega} \right) + \frac{1}{Q} - 2j \frac{\Delta \omega_a}{\omega_a} = 0
\]  \hspace{2cm} (2.33)

Therefore,

\[
Q = -\frac{1}{j} \left[ \frac{\omega(\omega_a + \Delta \omega_a)}{\omega^2 - (\omega_a + \Delta \omega_a)^2} \right]
\]  \hspace{2cm} (2.34)

Now, rearranging the Eq.(2.33) [with \((\Delta \omega_a)^2 \rightarrow 0\) and with \( \omega \equiv \omega_0 \)], we get
\[ \omega = \omega_a + j \frac{\omega_a \Delta \omega_a}{2Q} \quad (2.35) \]

or,

\[ \frac{j}{Q} \frac{2(\omega - \omega_a)}{\omega_a} + \frac{2\Delta \omega_a}{\omega_a} = 0 \]

\[ \frac{1}{Q} - j \frac{2\Delta \omega_a}{\omega_a} = -2 j \frac{(\omega - \omega_a)}{\omega_a} \quad (2.36) \]

Using the solution \( e^{iot} \) in Eq.(2.23) and Eq.(2.36), by dividing the right hand side of the Eq.(2.36) by \( \varepsilon_0 \mu_0 \int \mathbf{E} \cdot \mathbf{E}_a dv \)

\[ \frac{1}{Q} - j \frac{2\Delta \omega_a}{\omega_a} = -j \frac{\int (\mathbf{n} \times \mathbf{E}) \cdot \mathbf{H}_a da}{\omega_a (\varepsilon_0 \mu_0)^{1/2} \int \mathbf{E} \cdot \mathbf{E}_a dv} \]

\[ -\frac{1}{\varepsilon_0 \omega_a} \int (\mathbf{n} \times \mathbf{H}) \cdot \mathbf{E}_a da \int \mathbf{E} \cdot \mathbf{E}_a dv + \frac{1}{\varepsilon_0 \omega_a} \int \mathbf{J} \cdot \mathbf{E}_a dv \quad (2.37) \]

The first two terms of the Eq.(2.37) deal with the first and second type of perturbations \( i.e. \) Eq.(2.27) and (2.28) which depend mostly on the construction and connection of the cavity and may be considered as the perturbation when the cavity is not loaded. But, the third term on R.H.S. of Eq.(2.37) deals with two losses and frequency displacement due to currents in the cavity.

We can either assume the boundary condition such that first two terms on right hand side vanish \( i.e. \) for perfect conductor (over the surface \( S \)) and no energy loss, we make measurements relative to these terms under the circumstances of relative measurements.
Therefore, one can write

\[ \frac{1}{Q} - \frac{1}{Q_0} = \Delta \left( \frac{1}{Q} \right) \]  

(2.38)

Where,

\[ \frac{1}{Q_0} = \frac{- j}{\omega_a (\varepsilon_0 \mu_0)^{1/2}} \int_{\Sigma} \frac{(n \times E) \cdot H_a}{E \cdot E_a} d\sigma - \frac{1}{\varepsilon_0 \omega_a} \frac{\int_{\Sigma} (n \times H) \cdot E_a}{E \cdot E_a} d\sigma \]

Therefore, using Eq. (2.37) and (2.38) we get

\[ \Delta \left( \frac{1}{Q} \right) - j \frac{2 \omega_0}{\omega_a} \int_{\Sigma} \frac{J \cdot E_a}{E \cdot E_a} d\sigma = \frac{1}{\varepsilon_0 \omega_a} \frac{\int_{\Sigma} J \cdot E_a}{E \cdot E_a} d\sigma \]  

(2.39)

Equation (2.39) is applicable in the situation in which the perturbation is due to the currents produced within the cavity by introduction of the sample. We give here below a description of perturbation on type of the sample used.

(A). If the sample is in gas phase, it may be assumed that the cavity is uniformly filled with a material, which conducts according to Ohm’s law with the conductivity \( \sigma \), and dielectric constant \( \varepsilon \), rather than the value \( \varepsilon_0 \), which is characteristic of empty space. The current density \( J \) and conductivity \( \sigma \) are related to the applied electric field \( E \) as

\[ J = \sigma E \]  

(2.40)

and from the polarization (P), which is related to the currents in the sample

\[ P = (\varepsilon - \varepsilon_0) E \]  

(2.41)
There is a current density given by the time rate of change of $\mathbf{P}$ or $j\omega(\varepsilon - \varepsilon_0)\mathbf{E}$, if there is a sinusoidal field. Under these circumstance

$$\Delta\left(\frac{1}{Q}\right) = \frac{\sigma}{\varepsilon_0\omega_a}$$

$$= \frac{\sigma}{\omega_a} \quad \text{for } \varepsilon_0 = 1 \quad (2.42)$$

and

$$\frac{\Delta\omega_a}{\omega_a} = \frac{\varepsilon - \varepsilon_0}{2\varepsilon_0}$$

$$= \frac{\varepsilon - 1}{2} \quad (2.43)$$

Equation (2.42) indicates that the increase in losses depend upon increase in the conductivity. Eq.(2.43) gives first order correction to the frequency, if the cavity is filled with a dielectric. From Eq.(2.25) by putting $\varepsilon$ in place of $\varepsilon_0$

$$\omega_a^2 = \frac{k_a^2}{\mu_0\varepsilon}$$

$$\therefore \omega_a \propto \left(\frac{1}{\sqrt{\varepsilon}}\right)$$

Where, $k_a = 2\pi/\lambda$ and $\mu_0 = \text{permeability of free space}$.

For free oscillations put $\varepsilon = \varepsilon_0$, we get thus corrected formula for frequency shift, using the Eq.(2.43) and term $\mathbf{E}_a \int \mathbf{E} \cdot \mathbf{E}_a d\gamma$.

(B). Considering the case of liquid or solid in which the cavity is not completely filled, the distribution of the $\mathbf{E}$ and $\mathbf{H}$ might be violently altered. In this case, our assumption
that the field is nearly $E_a$ in the neighborhood of the $\alpha$th resonant frequency, on which parts of our deduction are based, would not hold. If the effect of the conductor is or dielectric material in the field distribution is not great, so that we can approximately replace $E$ by $E_a \int E \cdot E_a \, dv$, Eq.(2.39) becomes

$$\Delta \left( \frac{1}{Q} \right) - j \frac{2 \Delta \omega}{\omega_a} = \frac{1}{\varepsilon \omega_a} \int \sigma E \cdot E_a \, dv$$

or,

$$\Delta \left( \frac{1}{Q} \right) - j \frac{2 \Delta \omega}{\omega_a} = \frac{1}{\varepsilon \omega_a} \int \left[ \sigma + j \omega (\varepsilon - \varepsilon_0) \right] E_a^2 \, dv \quad (2.44)$$

That is, the effect of a conductor or dielectric is large at places where $E_a$ is large, small where it is small. Alternatively, we can solve this problem by considering the local field $E_s$ due to currents produced in the dielectric material and it is different from $E$.

Replacing $E$ by $E_s$ in Eq.(2.41) and treating $\varepsilon$ as complex

$$P = (\varepsilon^* - \varepsilon_0)E_s$$

$$= \left[ (\varepsilon' - j\varepsilon^*) - \varepsilon_0 \right] E_s$$

$$\therefore \varepsilon^* = \varepsilon' - j\varepsilon^*$$

Therefore, for $\varepsilon_0 = 1$

$$P = [(\varepsilon' - 1) - j\varepsilon^*]E_s \quad (2.45)$$

From Maxwell’s equation, we get

$$\Delta \times H = J + \frac{\partial D}{\partial t} = J + \frac{\partial}{\partial t} \left( \varepsilon_0 E_s + P \right)$$
Therefore, replacing \( J \) by \( J + j\omega(\varepsilon_0 E_s + P) \) or for relative measurement put \( \Delta J \) in place of \( J \) in Eq.(2.39).

\[
\Delta J = J + j\omega(\varepsilon_0 E_s + P) - J
\]

From Eq.(2.39) and (2.47)

\[
\Delta \left( \frac{1}{Q} \right) - 2j \frac{\Delta \omega_a}{\omega_a} = \frac{1}{\varepsilon_0 \omega_a} \int_v j\omega(\varepsilon_0 E_s + P) \cdot E_a dv
\]

\[
= \frac{j\omega}{\varepsilon_0 \omega_a} \int_v [\varepsilon_0 E_s + (\varepsilon = \varepsilon_0) E_s] \cdot E_a dv
\]

\[
\therefore \Delta \left( \frac{1}{Q} \right) - 2j \frac{\Delta \omega_a}{\omega_a} = \frac{\int_v E_s \cdot E_a dv}{\int_v E \cdot E_a dv}
\]

Where \( \varepsilon = \varepsilon^* = \text{complex} = \varepsilon' - j\varepsilon'' \).

Separating real and imaginary parts

\[
\Delta \left( \frac{1}{Q} \right) = \varepsilon'^* F(E) \quad (2.48)
\]

\[
2 \frac{\Delta \omega_a}{\omega_a} = -\varepsilon'' F(E) \quad (2.49)
\]
Where,

\[ F(E) = \frac{\int E_s \cdot E\, dv}{\int E \cdot E_s\, dv} \]  

(2.50)

2.3.2 Experimental Details of Microwave Cavity Spectrometer

The microwave cavity spectrometer is one of the accurate techniques and it needs only a small volume of the sample (≈ 0.001 cm³). The microwave cavity spectrometer [8-14] has been used at 9.0GHz. A block diagram of the microwave cavity spectrometer used [8,9] is given in Fig. 2.1. A signal frequency near the cavity resonance is generated by a reflex klystron by setting the voltage and mechanical tuning. The time base of a Tektronics Model 561A oscilloscope is used to derive a saw tooth ramp voltage, which sweeps the klystron over the range of frequencies desired. To produce an ac signal at the detector, simultaneously a chopper signal of 31 kHz is impressed upon the repeller electrode. This arrangement allows for a synchronized sweep of the frequency with the voltage scan of the oscilloscope. Moreover, the 31 kHz signal enables high gain tuned amplifiers to be employed to detect the signal arriving at the detector. The oscilloscope display shows markers on one channel and the signal derivative as desired on the other channel. The second derivative of the signal was chosen for this work. Signals from the klystron are sent to the marker mixer, along with the harmonics of the frequency standard. A tuned radio receiver (Hammerlund Model HQ 180A) is used for comparison of these two signals. Two markers \( f_k - nf_s \) and \( nf_s - f_k \), where \( f_k \) is klystron frequency and \( nf_s \) the proper harmonic of the standard signal source, were generated and displayed on one channel of the oscilloscope. The separation of these markers was set at 4 MHz.
Figure 2.1: The Block diagram of the Microwave cavity spectrometer
A cylindrical microwave cavity is shown in Fig. 2.2, with internal diameter 4.9 cm and a quality factor $Q \approx 5000$ operated in the $TM_{010}$ mode served as the test cavity. A permanent record of the signal and markers was produced on a dual chart recorder as shown in Fig. 2.3. The sample is subjected to temperature control by flushing chilled or steam heated air around the resonant cavity with the help of a copper pipe heat exchanger placed in a Dewar flask or in boiling water. The rate of flow was adjusted as required to maintain the sample and cavity at the equilibrium temperature. The cavity was thermally insulated and shielded in order to help to maintain the stable temperature. The measurement of temperature was done by thermocouple and it was read by an analog voltmeter.

The frequency calibration is achieved by the use of a general radio frequency standard set composed of and 1112A and 1112B system good to $1/10^6$. Thus, the frequency measurements are accurate to a few kHz for each reading of the chart on a dual – pen chart recorder. The data were taken by displaying the cavity resonance profile on one scan of a dual trace chart recorder while calibration markers generated from a mixer-multiplier displayed on the other scan. Each frequency interval was partitioned by using an interpolation radio receiver set for the desired frequency interval. The separation of the markers was set at $4\text{MHz}$. The center frequency of the resonance was set at $f_0 \pm \Delta f$, where the range of $\Delta f$ was determined by how much shift ensued when each sample was placed into the cavity. A permanent record of the signal and markers was produced on a dual – pen chart recorder as shown in Fig.3. The overall uncertainty of the measurements is less than 5%.
Figure 2.2: The sketch of the cylindrical cavity with top and side view of the cavity.
Figure 2.3: The trace of the chart recorder for a system under consideration. The separation between the markers is set to be 4MHz.
2.3.3 Thermodynamical Parameters

The thermodynamical parameters such as free energy of activation ($\Delta G$), enthalpy ($\Delta H$) and entropy ($\Delta S$) can be calculated by using relaxation time using Debye's single relaxation mechanism [15] with the help of loss tangent ($\tan \delta$).

The expression for the loss tangent ($\tan \delta$) is

$$\tan \delta = \frac{\varepsilon''}{\varepsilon'} \approx \frac{\Delta w}{2\Delta f}$$

for $\varepsilon' \gg 1$ (2.51)

Therefore, the relaxation time is calculated using Debye's single relaxation time mechanism [15] from the relation given below:

$$\tau = \left( \frac{1}{\omega} \right) \times \left( \frac{\Delta w}{2\Delta f} \right)$$

(2.52)

where, $\omega (= 2\pi f_0 ; f_0 = 9.0\text{GHz})$ is the angular frequency.

The expression for the activation energy ($\Delta G$) is written as

$$\Delta G = 2.303RT\log\left(\frac{\tau kT}{h}\right)$$

(2.53)

Also, we have

$$\Delta G = \Delta H - T\Delta S$$

(2.54)

where $R$ is the molar gas constant, $h$ is the Plank's constant, $k$ is the Boltzmann constant and $T$ is the absolute temperature, $\Delta H$ is the change in enthalpy and $\Delta S$ is the entropy change. Using Eq.(2.53) and (2.54) we get

$$2.303\log(\tau T) = \left( \frac{\Delta H}{R} \right) \frac{1}{T} + 2.303\log\frac{h}{k} - \frac{\Delta S}{R}$$

(2.55)
The slope of the plot of \( \log(\tau T) \) against \( 1/T \) gives \( \Delta H/R \) from which \( \Delta H \) can be calculated. Using Eq. (2.53) and (2.54) we can evaluate \( \Delta S \).

### 2.4 TIME DOMAIN REFLECTOMETER (TDR)

The Time Domain Spectroscopy (TDS) was first introduced by Fellner – Feldegg [16-18] in 1969 in the field of dielectric studies of liquid dielectrics. Since then, many important and useful modifications have been made in this technique to reduce errors in measurements and to attain a wide range of frequencies for the dielectric studies [19-21]. This technique provides information about the dielectric relaxation in just a single measurement for a wide range of frequencies and proofs itself more advantageous for measurements to be done on expensive materials, as it requires a very small amount of sample just about 1 ml. The measurements can be done in a very short duration \( i.e. \) within 30 seconds. So, time domain can be considered as truly spectroscopic technique because of its broad band nature and capacity to generate a continuous function of time or frequency.

The time domain reflectometry is the technique of observing time dependent response of system after the application of an electromagnetic field and finding the transfer function of the system when the input signal and the transient response both as a function of time are known. In 1960, Hewlett Packard Co. USA introduced a time domain system with a large frequency response up to 12.4 GHz. During its initial stages, this equipment was being used mainly for location faults in the wide-band transmission system such as co-axial lines [22,23].

The frequency response of the system is the ratio of the Fourier transforms of the transient response and the input signal. This operational method is especially for the
study of dielectric response at low frequencies [23]. For high frequency region, such as microwave, a fast rise time pulse of the order of pico second having fast detection system is used to get the characteristic information about the dielectrics.

For last few years the TDS technique has been used successfully for the dielectric study of liquids of biological importance [24-27], dielectric properties of agricultural samples [28]. The TDS technique is advantageous over the conventional frequency domain technique for dielectric studies as TDS technique, in principle, provides a wide range of frequencies without changing the experimental setup while, in frequency domain technique, every time a new frequency is required for the measurements. Soon after the development of fast pulse generation and sampling methods, the potential usefulness of time domain methods for the broad band measurements of dielectric behavior at several gigahertz frequencies was recognized.

In the time domain spectroscopy technique, there are two methods of measurements namely Reflection and transmission. The term, time domain reflectometry [29] (TDR), is used to describe a technique to observe the time dependent response of a sample of interest after the application of the time dependent electromagnetic field and its reflection from the sample. The response characteristic, whether measured in terms of current change or other related observable parameter, is thus a real quantity as a function of real-time in contradiction to steady state measurements of a complex quantity that expresses amplitude and phase relation of response to an alternating (AC) field at a specific frequency. The conventional AC method for the dielectric coefficient ranges from $10^7$ to $10^{10}$ Hz and it requires complex instrumentation and data processing. The time domain technique provides a better alternative for the study of the systems having time dependent behavior over a
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Figure 2.4: (A) Experimental arrangement of the basic unit of Time Domain Reflectometer measurement (B) Response to Step Pulse
wide range of frequencies. This technique has an important feature that a single data can give information over a considerable range of frequency. After development of the tunnel diode and sampling oscilloscope, at present it is possible to take measurements for a time resolution of few picoseconds to times of several nanoseconds or larger. If the system has a linear response characteristics, the measurements can be taken by two methods viz. reflection and transmission methods.

2.4.1 Theory of Time Domain Reflectometry

In this system a fast rising step voltage is propagated in an ideal (low loss) coaxial line having 50 Ω characteristic impedance. The shape of the step voltage remains unchanged as long as the propagation properties of the coaxial line remains the same. But, suppose a dielectric material is enclosed in a section of the coaxial line, then the step voltage will partly be reflected and partly be transmitted at the interfaces. A pulse generator produces a step voltage with a rise time of 40 picoseconds and is detected at the sample head, which is displayed on an oscilloscope. The travelling pulse on reaching the dielectric sample, gets reflected back and after some time reaches the sample head, which is then displayed on the oscilloscope screen.

The current $i(\omega)$ and voltage $V(\omega)$ in a coaxial line with characteristic admittance $G_c$ ($= 1/50\Omega$ for ideal 50Ω transmission line) are related to incident and reflected voltage $V_0$ and $r_x$ by

$$i(\omega) = G_c(V_0 - r_x)$$

$$V(\omega) = (V_0 + r_x)$$
The input admittance \( y(\omega) \) of a termination is obtained from

\[
y(\omega) = \frac{i(\omega)}{V(\omega)} = G_c \frac{V_0 - r_x}{V_0 + r_x}
\]  

(2.56)

In a particular case of the system exhibiting negligible conductivity, the admittance of the sample at the input of the transmission line is given by Cole [30]

\[
y(\omega) = \left( \frac{i \omega d}{c} \right) G_c \frac{\varepsilon^*}{\zeta \cot \zeta}
\]  

(2.57)

where \( \zeta = (\omega l/c)\sqrt{\varepsilon^*} \) and the term \( \zeta \cot \zeta \) can be written to explain the series expansion as

\[
\zeta \cot \zeta = 1 + \frac{1}{3} \zeta^2 - \frac{1}{45} \zeta^4 + \ldots \quad |\zeta| < \frac{1}{2}
\]  

(2.58)

In Eq. (2.57), \( G_c \) is the characteristic conductance of the coaxial line which is equal to \( (C_c/L_c)^{1/2} \), where \( C_c \) is the geometrical capacitance of ideal line of length \( l \) and \( L_c \) is its inductance per unit length, \( \varepsilon^* \) is frequency dependent complex permittivity, \( \omega \) is the angular frequency, \( d \) is the length of coaxial line section filled with dielectric and \( c \) is the speed electromagnetic radiation in air.

From Eq. (2.56) and (2.57)

\[
\varepsilon^* = \frac{c}{i \omega d} \frac{V_0 - r_x}{V_0 + r_x} \zeta \cot \zeta
\]  

(2.59)

The dielectric permittivity of an unknown sample can be found by this equation, if the time profile of the incident and reflected pulses are known within a frequency range determined by the time limits of \( V_0 \) and \( r_x \).
Rearranging Eq.(2.59) to get $V_0$ and $r_x$, 

$$V_0 = \frac{r \left[ z \cot z + \left( \frac{i \omega d}{c} \right) \varepsilon^* \right]}{\left[ z \cot z - \left( \frac{i \omega d}{c} \right) \varepsilon^* \right]}$$  \hspace{1cm} (2.60)$$

For air medium $\varepsilon^* = 1$ and Eq.(2.60) becomes 

$$V_0 = \frac{r \left[ z \cot z \right]}{\left[ z \cot z - \left( \frac{i \omega d}{c} \right) \varepsilon^* \right]}$$  \hspace{1cm} (2.61)$$

Equating the Eq.(2.60) and (2.61) we get,

$$\frac{r_1}{r_x} = \frac{\left[ z \cot z + \left( \frac{i \omega d}{c} \right) \varepsilon^* \right]}{\left[ z \cot z - \left( \frac{i \omega d}{c} \right) \varepsilon^* \right]}$$  \hspace{1cm} (2.62)$$

or,

$$\frac{c}{i \omega d} \frac{r_1 - r_x}{r_1 + r_x} = \frac{\varepsilon^* \left[ z \cot z - z \cot z \right]}{\left( z \cot z \right)^2 + \left( \frac{\omega^2 d^2}{c^2} \right) \varepsilon^*}$$  \hspace{1cm} (2.63)$$

The quantities $(r_1 - r_x)$ and $(r_1 + r_x)$ will be called as $p$ and $q$ respectively.

Since, Laplace transform is a linear operator, then,

$$p(i\omega) = LP(t) = L[R_1(t) - R_x(t)]$$  \hspace{1cm} (2.64)$$

and

$$q(i\omega) = Lq(t) = L[R_1(t) + R_x(t)]$$  \hspace{1cm} (2.65)$$

Neglecting the quadrupole resonance effect due to multiple reflections and using approximation $z\cot z = 1$, we can write Eq.(2.63) as 

$$\frac{c}{i \omega d} \frac{p}{q} = \frac{\varepsilon^* - 1}{1 + \left( \frac{\omega^2 d^2}{c^2} \right) \varepsilon^*}$$  \hspace{1cm} (2.66)$$
Figure 2.5: Photograph of HP54750A Time Domain Reflectometer setup.
Figure 2.6: Block diagram of Dual Channel TDR.
A. The Mainframe TDR unit
B. HP54754A TDR Plug-in module
C. Pulse generator
D. Sampling head
E. Sample holder – 1
F. Sample holder – 2
G. HP54750A Sampling oscilloscope
H. Floppy disk drive
L1, L2 – Transmission lines (50Ω)
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Left-hand side of this equation may be called Reflection Coefficient, which is a complex quantity given by

\[ \rho^* = \frac{c}{i\omega \gamma} \frac{p(\omega)}{q(\omega)} \]  

(2.67)

Therefore, Eq.(2.63) can be written as

\[ \varepsilon^* - 1 = \frac{(1 + A)\rho^*}{1 - B\rho^*} \]  

(2.68)

Since, for ideal coaxial transmission line \( A = B = (\omega dl/c)^2 \).

From Eq.(2.68) one can easily determine complex permittivity \( \varepsilon^* = \varepsilon' - j\varepsilon'' \), provided that the reflection coefficient \( d, \rho^* , p(\omega), q(\omega), A \) and \( B \) are known.

2.5 EXPERIMENTAL SETUP OF TDR AND PROCEDURE

The photograph of the TDR measurement system is shown in Fig.2.5. The experimental setup consists of a sampling oscilloscope HP54750A [30], TDR module HP54754A [31], a transmission line, sampling cell and temperature bath. The working of all these components has been discussed in the ensuing paragraphs.

The block diagram of the differential type TDR setup is given in Fig.2.6. HP54750A sampling oscilloscope is an instrument of high precision for the digital data acquisition of the instantaneous signals. The work function of the instrument is driven by the front panel keys as well as menus invoked after pressing the front panel keys. These menus are called the soft key manus. These soft key manus list various functions other than those accessed directly by the front panel keys. A function on soft
key menu can be accessed by pressing unlabeled key; immediately next to the annotation on the screen. The unlabeled keys next to annotation on display are called soft-keys. The front panel of the instrument consists of a display area and several functional areas, which include Control, Storage, Autoscale, Entry devices, Setup and System. The System and Control sections include three keys namely, Clear display, Run and Stop signal. These keys are used to clear the screen, start data acquisition and stop data acquisition respectively. The storage section includes four keys namely, Disk, Waveform, Setup and Print. The Disk key is used to access information from 1.44 MB Floppy Disk Drive. The waveform can be stored on the disk or loaded from the disk. The waveform key is used to store the current waveform in memory of the oscilloscope. At a time four waveforms can be stored in the memory of the oscilloscope. The Setup key is used for setting the waveform. The Print key is used to print the current waveform or waveform in the memory. The Autoscale section of the contains only one key Autoscale. This Autoscale key makes the instrument to analyze the signals quickly. Then, it sets up vertical and horizontal and triggers to best display the signal. Entry devices are the keypad, the arrow keys and the knob. Entry devices can change the numeric settings of some softkeys, such as trigger level, or to select an item from the list of choices. The Setup section includes seven keys namely, Time base, Trigger, Acquisition, Display, Marker, Define meas. and Math. With help of the time base key, we can change the horizontal position of waveform and also the time window. The Trigger can be used to change the trigger level of the signal. The Acquisition key is used to set number of data acquisition points and also number of times the averaging is to be done. The Marker key can be used for setting markers on waveform during the measurement of specific parameters. One can also put meas.
(measurement marker lines) during measurement. The Math function key is used to perform few mathematical operations such as addition and subtraction of two waveforms or even Fourier transform of the waveform. Additional functions listed in the bold typeface above and below some of the front panel keys. These functions are called shifted functions. By pressing the front panel shift key and front panel key next to the desired function, these shifted functions can be activated.

The mainframe TDR unit consists of blocks as marked in block diagram and their detailed functions are given in TDR user's guide [30,31]. The transmission line of $50 \Omega$ is used. The relations using lumped parameters are $R$, $L$, $C$, $\alpha$, $\beta$, $\gamma$, $Z_\sigma$, $\sigma$ (VSWR) could be found in any of the standard book on electronics [4].

The HP54750A sampling mainframe along with the HP54754A Plug-in unit has been used for the time domain reflectometer setup. The TDR unit is used for measurement after warming up for at least 30 min. This is necessary to get step pulse without drift. A fast rising pulse of $200mV$ with $40 \text{ ps}$ rise time was propagated through a coaxial cell of $3.5mm$ outer diameter and $1.35mm$ effective pin length. After connecting the transmission line and sample holder to TDR unit, the reflected waveform is observed carefully. The unwanted reflections in the reflected step at point of contact between the transmission line and TDR unit, as well as the transmission line and sample cell is minimized by ensuring proper contact between these components. The pulse reflected from the sample was monitored.

A time window of $5ns$ was used. The reflected pulse from the channel with the reference liquid $i.e. R_1(t)$ and with sample $R_x(t)$ were digitized with 1024 points and transferred to the floppy disk in the floppy drive attached in the sampling oscilloscope.
The temperature controller system with water bath and a thermostat have been used to maintain the constant temperature with the accuracy limit of ± 1°C. The sample cell is surrounded by a heat insulator container through which water at constant temperature is circulated. A digital thermostat was used to measure the temperature of the system.

The complete physical setup is viewed earlier in snap of the TDR Fig.2.5. A flexible or rigid transmission line is connected between the TDR unit and the sample holder. The measuring cell is SMA cell with effective pin length 1.35 mm. Considering the importance of the time window, to reduce the unwanted reflections, is kept to 5ns, which is changed depending on the nature of the dielectric liquid used. For the digitization of pulse, which is centered in the time window, 1024 points per waveform are used.

First the reflected pulse $R_1(t)$ of the sample cell without the sample is averaged 16 to 64 times and digitized with 1024 points per waveform and is stored in the oscilloscope memory. Secondly, by placing the sample in the cell, the reflected pulse $R_x(t)$ is again averaged 16/64 time and after digitizing with 1024 points, it is transferred to the floppy disk. A typical example of these reflected pulses without and with sample from the cell in a time window of 5ns is shown in Fig.2.7. To get the proper time referencing, the reflected pulse $R(t)$ was horizontally shifted properly.

After getting time referenced, the reflected pulses $R_1(t)$ and $R_x(t)$ are subtracted to get $p(t) = R_1(t) - R_x(t)$ and added to get $q(t) = R_1(t) + R_x(t)$ by using computer program.
Figure 2.7: (A) Reflected step pulse without sample and reflected pulse with sample $R_s(t)$, (B) Subtracted pulse $p(t)$ and added pulse $q(t)$.
2.5.1 Sample Cell

The sample cell holds the liquid under consideration. The physical dimensions of the cell are very important. So, one must be careful while designing the sample cell. The impedance of the cell should be matched with coaxial transmission line to which the cell is connected. If there is impedance mismatch, then unwanted reflections may disturb the wave thereby causing some errors in measurement. The proper design of the cell includes the inner conductor and outer conductor diameters. The length of inner conductor is called as 'pin length' of the cell and is very important factor in analysis. The sample length must be enough to avoid unwanted reflections.

In total reflection method, the sample length must be long enough to produce an adequate signal but short enough to keep less complications or resonance effects at frequencies above the range of interest.

The characteristic impedance of coaxial line is given by

\[ Z = \frac{138.2}{\sqrt{\varepsilon}} \log_{10} \left( \frac{b}{a} \right) \]  \hspace{1cm} (2.69)

This impedance for the transmission line is frequently 50 Ω. Here, 'a' is inner diameter of outer conductor equal to 3.5 mm and 'b' is diameter of inner conductor equal to 1.52 mm. \( \varepsilon \) is the relative permittivity of the dielectrics between the conductors. For air \( \varepsilon = 1 \) and for Teflon \( \varepsilon = 2.2 \), using above Eq.(2.69) with \( Z = 50 \) Ω and assuming either \( a \) or \( b \), it is easily possible to determine \( b \) or \( a \).

The SMA (Standard Military Application) type cell has 'a' equal to 3.5 mm. The 'inner conductor' of SMA cell itself considered as inner conductor and hex nut acts as an outer conductor. Since, these types of SMA connectors are already designed for
precise (50Ω) characteristic impedance, a special design for use with high frequency is not required. Therefore, SMA cell is considered to be the best cell for dielectric measurements. The physical length of the inner conductor can be changed without change of its characteristic impedance. The geometrical construction of this open-ended sample cell is given in Fig.2.8. These cells are used particularly in reflection method of time domain technique.

The effective pin-length $d$ can be determined as described below:

**Determination of effective pin-length:**

When a cell is filled with sample above the physical length of inner conductor, the fringing field effects take place. Due to the fringing field, the effective pin length will not be equal to the physical pin length. The effective electrical pin length will be more than the physical pin length. The accurate determination of the effective pin length $d$ is very important for the accurate evaluation of dielectric parameters. Fig. 2.8 shows the SMA cell dimensions with effective pin length. Experimentally it is found that for 7mm cell, the effective pin length is greater than actual physical length by 0.6 – 0.8mm and for SMA type cell it is greater than actual physical length by 0.1 – 0.2mm. This is due to the fringing field at the open end of the sample cell.

From the transmission line theory, the reflection coefficient can be defined as in Eq.(2.67) under limit $\omega \to 0$,

$$\lim_{\omega \to 0} \rho^* = \varepsilon_0 - 1 = \lim_{\omega \to 0} \frac{c}{j\omega l} \frac{p(\omega)}{q(\omega)}$$

$$= \frac{c}{j\omega l} \int_0^\infty p(t) \exp(j\omega t) dt$$

$$= \frac{1}{\omega} \int_0^\infty dq \frac{\exp(j\omega t) dt}{45}$$
Figure 2.8: (A) Geometrical construction and SMA cell dimensions showing the fringing field (B) Effective pin length of cell.
And finally, this expression reduces to

\[ \varepsilon_0 - 1 = \frac{c}{d} \left( \frac{P_{\text{area}}}{q_{\infty} - q_0} \right) \]  
(2.70)

or,

\[ \frac{d}{c} = \left[ \frac{P_{\text{area}}}{q_{\infty} - q_0} \right] \]  
(2.71)

where \( P_{\text{area}} \) is the area under the curve \( p(t) \), \( q_\infty \) and \( q_0 \) are the values of \( q(t) \) at \( t = \infty \) and \( t = 0 \) respectively.

Fig. 2.9 illustrates \( P_{\text{area}}, q_\infty \) and \( q_0 \). The values of \( P_{\text{area}}, q_\infty \) and \( q_0 \) can be determined experimentally by using liquids of known static permittivity (\( \varepsilon_0 \)). Therefore, for different liquids of known permittivity, we can plot a graph between \( P_{\text{area}}/(q_\infty - q_0) \) and \( (\varepsilon_0 - 1) \). The slope of the curve gives the right hand side of the Eq.(2.71), i.e.

\[ \frac{d}{c} = \text{Slope} \]

i.e.

\[ d = c \cdot \text{Slope} \]  
(2.72)

This slope determines the value of the effective pin length "d".

2.5.2 Fourier Transform

The reflection coefficient \( (\rho^*) \) is defined in Eq.(2.67), which needs \( d, p(\omega) \) and \( q(\omega) \). The \( p(\omega) \) and \( q(\omega) \) are Fourier transforms of \( p(t) \) and \( q(t) \) respectively as
Figure 2.9: Determination of Effective pin length
described in experimental procedure. The Fourier transformed values $p(\omega)$ and $q(\omega)$ are found as given below:

The data received by the PC from the TDR unit is in the form of voltage with respect to time. This data is then subtracted and added for obtaining the waveform of $p(t)$ and $q(t)$ and this waveform is stored in the file having extension “.wfm”. The data of $p(t)$ and $q(t)$ are then combined together after scaling (i.e. multiplying by a factor 10000 to each data point in each file). The combined files are called “.tdr”. Thus, “.tdr” file contains the array of data points of $p(t)$ and $q(t)$. This time domain data is converted into frequency domain data using Fourier Transform. While performing Fourier transform, one should be careful about the nature of the curve of which transform is to be obtained. Since, nature of curves $p(t)$ and $q(t)$ curves are different, therefore, the special methods of Fourier transform are used for $p(t)$ and also for $q(t)$.

The Fourier transform of $p(t)$ is obtained by a summation method [33] using equation

$$p(\omega) = T \sum_{n=0}^{N} \text{Exp}(-j\omega nT) p(nT)$$  \hspace{1cm} (2.73)

The Fourier transformation using summation method has some limitations that for all the sampling intervals $T$, the nature of the pulse form must be known. Furthermore, the transform $p(\omega)$ is simply the area under the curve $p(t)$, which has an initial peak followed by a decay to zero or a finite limiting value strictly to infinite time.

The pulse form of $q(t)$ is not known exactly. The $q(t)$ rises monotonically to a long time limit. Therefore, summation method of Fourier transforms can not be used
for \( q(t) \) curve. The Fourier transform of such type of curves can be done with the Samulon method [34] for which expression is as follows

\[
q(\omega) = \frac{T}{1 - \exp(-j\omega T)} \left[ \sum_{n=0}^{N-1} \{q(nT) - q(n-1)T\} \exp(-j\omega nT) \right]
\] (2.74)

In above equations, \( \omega \) is angular frequency, \( T \) is the sampling interval or time difference between two adjacent points and \( N \) is number of points. In our experiment \( N \) is 1024 points and \( T \) depends on time window and number of points per waveform (\( N \)). For example, if time window is 5 ns, then \( T \) is 4.88 ps for 1024 points per waveform. Thus, the time domain data is converted into frequency domain data in the frequency range of 10 MHz to 20 GHz.

The frequency domain data obtained from the Fourier transform is further used to calculate complex reflection function \( \rho^*(\omega) \). The single reflection method has the advantage of giving a reflection coefficient of magnitude \( 0.3 < |\rho| < 1 \) over the whole frequency spectrum present in the incident pulse. It can thus be considered as a true wide frequency method even reaching \( > 20 \text{GHz} \) if sufficient accuracy in \( \rho^*(\omega) \) can be achieved. The demands on the accuracy in \( \rho^*(\omega) \) are quite severe at high permittivity and high frequencies and the method has been mostly used for liquids of medium permittivity. The complex reflection coefficient spectra are called as 'raw' data. Using this complex permittivity spectrum as given in above equation, static dielectric constant \( (\varepsilon_0) \), dielectric constant at infinite frequency \( (\varepsilon_\infty) \) and relaxation time \( (\tau) \) can be calculated by using Havriliak – Nigami [35] expression.

\[
\varepsilon^*(\omega) = \varepsilon_\infty + \frac{\varepsilon_0 - \varepsilon_\infty}{1 + (j\omega\tau)^{-\alpha}}
\] (2.75)
2.5.3 Bilinear Calibration Method

The bilinear calibration method gives values of $A$ and $B$ as described below:

The problem in TDR experiments arises from the fact that the characteristic impedance of the transmission line connecting the measuring plane of the cell is generally not uniform along the propagation axis. This fact introduces reflections of the travelling pulses superimposed on the reflection from the cell. The expression mentioned above in Eq.(2.75) is obtained by considering the transmission line as ideal, so that $A = B = (\alpha d/c)^2$ but, under experimental conditions transmission can not be an ideal i.e. $A \neq B \neq (\alpha d/c)^2$.

To minimize the effect of unwanted reflections, several workers [35-37] have suggested different methods. Cole et al [38] has described a bilinear calibration method to eliminate these unwanted reflections at high frequencies. In the low frequency limit

$$\lim_{\omega \to 0} \rho^* = \varepsilon^* - 1$$

So, the permittivity at low frequency can be obtained directly by determining $\rho^*$.

Corrections in 'raw' data are necessary to get accurate values of permittivity at high frequencies. The unwanted reflections also depend on pin length of the sample cell used. For different pin lengths, the position of randomness of data shifts either to low frequency side or to high frequency side in the spectrum.

The calibration process suggested by Cole et al [38] involves experimental determination of values of $A$ and $B$ for getting reliable values of higher frequencies from $\rho^*$ i.e. using equation
We have to determine \( A^* = A' - jA'' \) and \( B^* = B' - jB'' \) using two or more standard liquids of known permittivity. The values of \( \varepsilon^* = \varepsilon' - j\varepsilon'' \) can be obtained theoretically by known values of dielectric parameters. The values of \( \rho^* \) can be determined experimentally by using above equation. After calculating \( \varepsilon^* \) and \( \rho^* \) for standard liquids, value of \( A \) and \( B \) can be evaluated by least squares fitting. At least two standard liquids are needed in order to evaluate four unknown parameters \( \varepsilon' \), \( \varepsilon'' \), \( B' \) and \( B'' \). Since, use of two standard liquids gives four linear equations, which can be solved to get above unknowns at all frequencies.

It is also important to select proper standard liquids. The selection of liquids should be such that the raw spectra of unknown sample should be matched with raw spectra of standard liquid. Otherwise accurate determination of permittivity is not possible. Cole et al [38] has suggested many calibrating liquids with different kind of dielectric spectra for determination of values of \( A^* \) and \( B^* \).

After getting the frequency dependent values of \( A \)'s and \( B \)'s, these values can are considered as constants and using Eq.(2.76) of unknown samples can be determined experimentally. Thus, by using the bilinear calibration process, one can eliminate non-ideal configuration of transmission line and imperfection of the system including spurious reflections.

The frequency dependent permittivity spectrum obtained by calibration process is called 'cor' data. Using 'cor' data, dielectric parameters can be obtained using the Eq.(2.75). The dielectric parameters \( \varepsilon_0 \), static permittivity (\( \varepsilon_0 \)), relaxation time (\( \tau \)
and \((\varepsilon_m)\) are calculated by fitting ‘cor’ data to suitable dielectric model using non-linear least squares fit method [36].

2.6 SYSTEM OPTIMIZATION USING TIME WINDOW, TIME REFERENCING AND ERROR TRUNCATION

2.6.1 Time Window

The time window chosen for the observation of reflected signals has to be related according to the frequency range of interest. The lower end of the frequency spectrum contained in the step pulse depends on the time window used, while the upper end is dependent on the rise time of pulse. The minimum frequency observable is \(f_{\text{min}} = 1/(\text{time window})\), whereas the maximum frequency observable is \(f_{\text{max}} = N/(2 \times \text{time window})\), where \(N\) is the number of points used to sample and digitize the signal. It is necessary to select number of points per waveform in the time window for the digitization of the signal. In order to reduce the noise, an averaging of signal for 16, 64 or 512 times can be done. If the signal is averaged over 64 times or more, noise will be reduced significantly.

2.6.2 Time Referencing and Error Truncation

When we observe the reflected pulses, the reference point, with respect to which we measure the amplitude of the pulse, is an important factor. The time reference of pulse reflected from cell without sample should match with the time reference of pulse reflected from the cell filled with the sample. The improper time referencing may give errors in reflection coefficient. Generally, reflected pulses with sample and without sample do not have the same relative time position for various
reasons. The effect of this error becomes serious at high frequencies. The correction of
timing errors can be carried out in a number of ways. One of the commonly used
methods for proper time referencing is to use numerical smoothing techniques, which
reduces effect of unavoidable reflections, in the portions where they are present in the
response signal.
REFERENCES


[34] H.A. Samulon, Proc, IRE., 39,175 (1951).


