3.1 INTRODUCTION

Time Domain Reflectometry (TDR) method is most suitable method for determination of frequency dependent dielectric parameters of material. This method was first introduced by Hugo Fellner-Feldegg et.al. in 1969 and developed by many workers in field of dielectric spectroscopy. In TDR method a fast rising step pulse is allowed to incident on sample under investigation. The reflected pulse from sample contains information regarding dielectric behavior of sample. The Fourier transformation of step pulse gives us frequency component contained in step pulse. Thus incident step pulse is treated as mixture of waves with different frequencies. The lower limit of frequency spectrum contained in step pulse depends on time window used, while upper limit depends on rise time of pulse. Response of dielectric sample to step pulse can be visualized as its response to different frequencies. Hence, frequency dependent parameters of sample can be determined from its response to incident step pulse. In order to obtain frequency dependent dielectric parameters one needs a step generator, a sampling oscilloscope, a sample holder and mathematical tools to process data. Dielectric study involves measurement of complex permittivity $\varepsilon' = \varepsilon' - i \varepsilon''$, where $\varepsilon'$ is dielectric dispersion and $\varepsilon''$ is dielectric loss. The complex permittivity also depends on temperature, pressure and intensity of applied field. From the observed frequency dependent permittivity, one can determine the relaxation time. These parameters then can be correlated to molecular structure and molecular dynamics.

The step generator must be capable of generating step pulse with rise time adequate enough to give the highest frequency components of interest with considerable magnitude. The broadband oscilloscope is required to handle broad frequency spectrum contained in step pulse. The oscilloscope must be capable of sampling high frequency signals with sufficient accuracy. A transmission line is needed to carry signal from step generator to sample holder. One end of transmission line is connected to sample holder while other end is connected to step generator. The transmission line as well as sample holder must be capable of holding high frequency signals. Further more characteristic impedance of these components must be matched for faithful transmission of signal from generator to sample holder. Any impedance mismatching in this signal path causes multiple reflections, which can distort signal of our interest. Practically multiple
reflections of signals, when it passes from one component to other cannot be avoided totally, but can be minimized to acceptable level by making some precautions.

The time domain data obtained from sampling oscilloscope is converted into frequency domain data using Fourier transmission. Then frequency dependent complex permittivity (ε*) is calculated from this data.

The traditional methods of dielectric permittivity were centered, around the measurements in the frequency domain. The common feature of all such methods being that the solution contained in a sample holder and its complex permittivity is measured at various discrete frequency points. It is, however not possible to device one piece of apparatus which can give the required frequency coverage. The experimental methods depend on range and type of system under consideration. At low frequency, the bridge techniques are used, with which one can obtain complex permittivity at frequency as low as 1 KHz. The upper frequency limit of bridges tends to be 100 to 250 KHz. At higher frequencies, transmission lines are used. Transmission lines may be coaxial line or waveguide. The frequency range of a coaxial line, is around 50 MHz to 20 GHz. Above this and below 100 GHz, waveguides are employed. Above 100 GHz, free space methods are used. At microwave frequencies 0.1 GHz to 100 GHz, traveling wave method, described by Buchanan is used.

For low dielectric loss materials, transmission measurement techniques, microwave bridges are used. The complex permittivity can be measured by probing standing wave pattern at regular intervals. The automated data processing improved the experimental sensitivity. Puranic et.al. has used X-band setup to measure complex permittivity.

3.2 TIME DOMAIN SPECTROSCOPY:

Dielectric study involves studying dielectric parameters at various discrete frequencies. A large range of apparatus and much experimental expertise is required. The Time Domain method is alternative to Frequency Domain method wherein a voltage pulse is passed through or reflected from the test liquid and the input output pulses, measured as function of time are compared. Fourier transmission of the data then gives frequency domain response of the system. Thus a complete frequency dispersion curve would be obtained from the analysis of a single pulse.
Originally, this method was used for low frequency dispersion by Davidson et al.\textsuperscript{10}. The technique consisted in studying the growth or decay of charge across a capacitor containing a dielectric substance. This range being from several seconds to milliseconds. However, in order to obtain information at a frequency of a few GHz, fast rise time pulse (of the order of ps) with an equally fast detection system would be required. In mid 60’s, the Hewlett-Packard introduced a time domain system with a frequency response up to 12 GHz\textsuperscript{11}. Initially, the apparatus was used mainly by the engineers, for making measurements on the solid state devices\textsuperscript{12,13,14}. TDS was introduced into the field of liquid dielectric by Fellner-Feldegg\textsuperscript{1,2,3}. TDS methods can be classified as slow response and fast response techniques\textsuperscript{15}. The slow response techniques have been reviewed by Vaughan\textsuperscript{16}, Suggett\textsuperscript{17}, and Hyde\textsuperscript{18}, are capable of measurements of time as low as 0.1 μS.

Time Domain Technique has been used to get complex permittivity spectra of organic liquids\textsuperscript{19-22} of aqueous solutions\textsuperscript{23,24} of solids\textsuperscript{25}, of electrolytic solutions\textsuperscript{26,27,28,29} of biological materials\textsuperscript{30}, of tissues, Heterogeneous substances and agriculture samples\textsuperscript{31}. Fellner-Feldegg suggested modifications in TDS equipment to minimize errors in the measurements and to increase the frequency range of dielectric study\textsuperscript{32-36}. Iskander and Stuchly\textsuperscript{37}, Nightingate et.al.\textsuperscript{38}, used TDS technique to study the dielectric properties of biological samples.

Time Domain Spectroscopy is classified into two groups -

(i) Time Domain Transmission method (TDT);

(ii) Time Domain Reflectometry method (TDR).

In the case of TDT, the wave shape of transmitted pulse through the sample is compared with incident pulse, whereas in the case of TDR, the wave shape of reflected pulse from the sample is compared with the incident pulse.

Gestblom et.al.\textsuperscript{39-41} has used TDT method for the study of low loss liquids. Cole\textsuperscript{19} has used TDR method for the dielectric relaxation. Suggett\textsuperscript{12,43} used TDR techniques to study amides and peptides. The advantage with TDS method is that one can get frequency response over a large range with a single shot measurement. The instrument setup is quite simple and less expensive than that used in frequency domain technique. The sample holder is usually a section of coaxial line or a coaxial line terminated by a cell, which is of a very small size requiring very small quantity of liquid for
measurements. Moreover, the time required for measurement is less than a 30 seconds, which avoids any degradation of the sample under consideration during reading.

3.3 THE EXPERIMENTAL SETUP:

The photograph of actual setup used is as shown in Fig 3.1. It consists of Digitizing oscilloscope HP 54750A, TDR module HP 54754A, a transmission line, sample cell and the temperature bath. The schematic block diagram of the experimental setup

![Schematic Diagram](image)

![Photograph of setup](image)

**Fig. 3.1:** Photograph of the actual experimental setup.
required for TDR measurements is shown in Fig 3.2. All these components in experimental setup are discussed in following sections.

**Fig. 3.2:** Schematic Diagram of Experimental Setup.

3.3.1 HP 54750A SAMPLING OSCILLOSCOPE:

The HP 54750A-sampling oscilloscope is very precise instrument for digital data acquisition of instantaneous signals. The working of instrument depends on front panel keys as well as menus invoked after pressing any front panel key. The menus of function are displayed along the right side of display screen. These menus are called as soft key menus. Soft key menus list functions other than those accessed directly by the front panel keys. To activate a function on soft key menu can be accessed by pressing unlabeled key immediately next to annotation on the screen. The unlabeled keys next to the annotation on display are called soft keys. Front panel of the instrument includes a display area and several functional areas, which includes control, storage, autoscale, entry devices, setup, and system. Control section includes three keys clear display, run and stop signal. These keys are used to clear screen, start data acquisition and start data
acquisition respectively. Storage section includes four keys disk, waveform, setup and print. Disk key is used to access information from 1.44MB Floppy Disk drive. We can store the waveforms on disk or load. Waveform key is used to store the current waveform in memory of oscilloscope. Four waveforms can be stored at a time in oscilloscope memory. Setup is used for setting waveform. Print key is used to print current waveform in memory. Autoscale section contains only single key autoscale. This autoscale key causes the instrument to quickly analyze the signal. Then, it sets up vertical, horizontal and triggers to best display that signal. Entry devices are the key board, the arrow keys, and the knob. Entry devices can change the numeric settings of some soft keys, such as trigger level, or to select an item from the list of choices. The setup section includes seven keys. Time base, Trigger, Acquisition, Display, marker, Define meas. and math. With time base key we can change horizontal position of waveform and also the time window. Trigger can be used to change trigger level of signal. Acquisition key is used to set number of acquisition points and also number of times the averaging is done. Marker key can be used for setting markers on waveform during measurement of specific parameters. One can also put meas. (measurement marker lines) during measurement. Math function key is used to perform few mathematical operations such as addition and subtraction of two waveforms or even Fourier transform of waveform.

3.3.2 HP 54754A PLUG-IN MODULE:

The HP 54754A TDR plug-in module\(^{45}\) capable of performing both, single ended TDR measurements as well as differential TDR measurements. These measurements include characterizing micro strip lines, PC board traces and coaxial cables. The plug in module takes up two, out of four mainframe slots. In single ended TDR measurement, a positive going step (a fast rising step voltage pulse of 200 mV with 49.50 ns rise time) is launched on one of the channels while the other channel is terminated using short. In differential TDR measurement, a positive going step is launched on channel 1 and effective negative going is launched on channel 2. The response controls are provided which shows the single ended or differential mode response of a TDR system under test.

i) PULSE GENERATOR
The tunnel diode pulse generator unit consists of trigger input, trigger generator, sequential delay generator and pulse filter. The plug-in's trigger input passes the trigger input directly into the mainframe. The trigger generator receives trigger input and sends a signal to sequential delay generator, whenever an edge meeting criteria is received, and the trigger is enabled. The sequential delay generator controls the time between when it receives trigger input from a trigger generator and when it sends a trigger output to the pulse filter. This delay time’s increase sequentially with each trigger event, allowing the trace to built up from left to right across the screen. The delay values depend upon the record length and time base setting, and are programmed by the system microprocessor.

ii) SAMPLING HEAD

The sampling head or sampler consists of a diode gate and sampling capacitor. The incoming signal is applied to the input of the diode gate, which is normally biased off. When the trigger event occurs, the pulse filter produces a pulse, which momentarily forward biases the gate and allows the input signal to charge the capacitor. It consist of an IF amplifier which senses the voltage on sampling capacitor and conditions it in preparation for digitizing the A/D converter.

The basic circuit used for sampling signal is shown in figure……. Closing sampling gate for short amount of time and all owing the signal to charge shunt capacitor through source resistance does sampling efficiency is given by

$$\frac{V_{out}}{V_{in}} = (1 - e^{-t/RC})$$

(3.1)

Shannon sampling Theorem states that to reliably extract all the information in the signal, it must be sampled at the rate at least twice the signal highest frequency.

Thus to sample 20 GHz signal, sampling rate must be 40GHz, which is not possible with fastest A/D converter available. This difficulty is overcome by using recitative sequential sampling. In the sampling processes, the input signal is sampled once per trigger event. The first trigger event in sequence causes the input signal to be sampled at an initial delay. On next trigger event (at a subsequent repetition of waveform), the sampling instance is delayed by small amount, relative to initial delay. Each additional trigger event causes the sampling instance to be delayed by greater amount of time, so that after many triggers, the input waveform builds on the screen from left to right.
3.3.3 THE 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 cell is connected. If there is impedance mismatch then unwanted reflections may disturb the wave thereby causing some errors in the measurements. The proper design of 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 difference signal but short enough to keep fewer complications of resonance effects at frequencies above the range of interest.

The characteristic impedance of a coaxial line is given by -

\[ Z = \frac{138.2}{\sqrt{\varepsilon}} \log_{10} \left( \frac{b}{a} \right) \]  \hspace{1cm} (3.2)

This impedance for our transmission line is frequently 50 Ω. Here ‘a’ is the diameter of inner conductor and ‘b’ is inner diameter of outer conductor, and \( \varepsilon \) is the relative permittivity of the dielectric between the conductors. The SMA type cell which is having ‘b’ as 3.5mm and ‘a’ as 1.52mm is used. The inner conductor of SMA cell itself considered as inner conductor and hex-nut acts as an outer conductor. Since these SMA connectors have already designed for precise 50 Ω impedance a special design when used with high frequency is not required. Therefore SMA cells are considered to be best cells for dielectric measurement. The physical length of inner conductor can be changed. The geometrical construction of this open ended sample cell is given in Fig. 3.3. These cells are used particularly in reflection mode of TDR technique.

When cell is filled with sample above the physical length of inner conductor the firing effect is takes place. Due to firing field the effective pin length will not be equal to physical pin length. The effective electrical pin length will be more than the physical pin length. The accurate determination of effective pin length ‘d’ is very important for the accurate evaluation of dielectric parameters. It is found that for SMA type cell effective
pin length is greater than actual physical length by 0.1 to 0.2 mm. The determination of
3.3.4 TEMPERATURE BATH

For temperature dependent dielectric measurements, the temperature of sample under consideration has to be checked and maintained. The temperature bath used is as shown in Fig. 3.5. The temperature of sample is maintained at desired value, within accuracy limit of ±1°C, by circulating constant temperature water through heat insulating jacket surrounding sample cell.

3.4 TDR FUNDAMENTAL

Measuring Standing Wave Ratio (SWR) checks the quantity of transmission line. But SWR measurement can not figure out the discontinuities separately. Also SWR measurement must be done at various frequencies to understand broadband response of the transmission line.

In TDR technique a voltage step up is propagated down the transmission line under investigation, and the reflected voltage waves are monitored by oscilloscope at particular point on line.

TDR measurement can give characteristics impedance of line and it shows both position and nature (resistive, inductive and capacitive) of each discontinuity along the line. TDR also demonstrate whether losses in transmission in system are series losses or shunt losses. Furthermore, TDR measurements give meaningful information regarding broadband response of transmission line.

3.4.1 PROPAGATION OF SIGNAL ALONG TRANSMISSION LINE

The equivalent circuit for transmission line\textsuperscript{46,47} is shown in figure 3.5. If C, G, L and R are defined per unit length for infinite long transmission line. Then we can write

\[
Z_m = Z_o = \sqrt{\frac{R + j\omega L}{G + j\omega C}}
\]

(3.3)

Where \(Z_o\) is characteristic impedance of transmission line.

A voltage pulse introduced at the input of transmission line requires finite time to
travel distance ‘x’ along the line. The phase of the voltage moving along the line lags behind the voltage introduced at input by amount $\beta$ per unit length. Furthermore, voltage will be attenuated by an amount $\alpha$ per unit length by series resistance and shunt conductance of the line. The phase shift and attenuation are defined by propagation constant $\gamma$ as

$$\gamma = \alpha + j\beta = (R + j\omega L)(G + j\omega C)$$

Velocity with which voltage propagates along the line can be written as

$$V_p = \frac{\omega}{\beta} \text{ unit length per sec}$$

The velocity of propagation approaches $V_c$, for transmission lines with air dielectric. For general case where $\varepsilon_r$ is dielectric constant of medium.

$$V_p = \frac{V_c}{\sqrt{\varepsilon_r}}$$

The voltage and the current at any distance ‘x’ along the transmission line can be written in terms of propagation constant $\gamma$ as

$$E = E_m e^{-\gamma x} \quad \text{and} \quad I = I_m e^{-\gamma x}$$

Since the voltage and current at any point ‘x’ is known, characteristics impedance of the line can be written as

$$Z_o = \frac{E_x}{I_x} = \frac{E_m e^{-\gamma x}}{I_m e^{-\gamma x}} = \frac{E_m}{I_m} = Z_{in}$$

When transmission line is terminated in a load whose impedance matches the characteristic impedance of the line, preceding equations satisfies the voltage and current relationships.

If $Z_L$ is not equal to $Z_o$, the incident energy is not fully delivered to load and propagates back towards source. The ratio of amplitude of reflected wave to incident wave is called reflection coefficient $\rho$.

$$\rho = \frac{E_L}{E_i} = \left( \frac{Z_L - Z_o}{Z_L + Z_o} \right)$$

The magnitude of steady state sinusoidal voltage along the line terminated in load other than $Z_o$ varies periodically as a function of distance between minimum and maximum
value. This variation is called standing wave. The ratio of maximum to minimum values of this voltage is called the Voltage Standing Wave Ratio (VSWR). It is related with reflection coefficient by equation

\[
\sigma = \frac{1 + |\rho|}{1 - |\rho|}
\]  

(3.10)

3.4.2 STEP REFLECTION FROM PURLEY RESISTIVE LOAD:

The TDR technique uses reflected pulse form sample to determine dielectric parameters. The block diagram for step reflection from load is shown in figure 4.6. It is very interesting to observe response of different type of loads to incident step. The shape of the reflected pulse is valuable since it reveals both, the nature and magnitude of mismatch. The typical examples for different values of purely resistive load are shown in figure 4.7. The knowledge of Er and E1 measured on oscilloscope allows ZL to be calculated in terms of Zo. Assuming Zo to be real (as in precision cables), it is seen that, mismatch reflects a voltage of the same shape as the driving voltage, with magnitude and polarity of E_r determined by relative values of Z_L and Zo.

3.3.3 STEP RERLECTIONS FORM COMPLEX LOADS:

The response of complex loads of different types to incident step pulse is shown in figure 3.8. The reflected voltage from complex loads is evaluated at t=0 and t=∞, by assuming any transition between two points to be exponential.

I) Series R-L

At t=0, reflected voltage is +E_r. This is because inductor will not accept sudden changes in current and initially it looks like infinite impedance. Then current in L builds up exponentially and its impedance drops down towards zero. At t=∞, E_r(t) is determined only by the value of R.

\[
\therefore \rho = \frac{R - Z_o}{R + Z_o} \text{ at } t = \infty
\]  

(3.11)

The exponential transition of E_r(t) has time constant determined by effective resistance seen by inductor. Since the output impedance is Z_o in series with R
\[
\tau = \frac{L}{R + Z_0} \quad (3.12)
\]

The reflected voltage can be written as
\[
E_r = E_i \left[ \left( 1 + \frac{R - Z_0}{R + Z_0} \right) \left( 1 - \frac{R - Z_0}{R + Z_0} \right) e^{-t/\tau} \right] \quad (3.13)
\]

II) Shunt R-C

At \( t=0 \), load appears as a short circuit since capacitor will not accept sudden changes in voltage and thus \( E_r = -E_i \). At \( t=\infty \), capacitor is effectively and open circuit and \( Z_L = R \).

\[
\therefore \rho = \frac{R - Z_0}{R + Z_0} \quad (3.14)
\]

Resistance seen by capacitor is \( Z_0 \) in parallel with \( R \). Therefore the time constant of exponential transition is
\[
\tau = \frac{Z_0R}{R + Z_0} C \quad (3.15)
\]

The reflected voltage can be written as
\[
E_r = E_i \left[ \left( 1 + \frac{R - Z_0}{R + Z_0} \right) \left( 1 - e^{-t/\tau} \right) \right] \quad (3.16)
\]

III) Shunt R-L

At \( t=0 \), \( Z_L = R \) (assuming \( R > Z_0 \)) and at \( t = \infty \), \( Z_L = 0 \). Impedance seen by inductor is parallel combination of \( R \) and \( Z_0 \). Thus
\[
\tau = \frac{R + Z_0}{RZ_0} L \quad (3.17)
\]

The reflected voltage \( E_r \) is given by equation
\[
E_r = E_i \left[ \left( 1 + \frac{R - Z_0}{R + Z_0} \right) e^{-t/\tau} \right] \quad (3.18)
\]

IV) Series R-C

At \( t = 0 \), \( Z_L = R \) (assuming \( R > Z_0 \)) and at \( t = \infty \), \( Z_L = \infty \). Impedance seen by
capacitor is series combination of R and \( Z_0 \). Thus

\[
\tau = (R + Z_0)C
\]

(3.19)

The voltage \( E_r \) is given by equation

\[
E_r = E_i \left[ 2 - \left( 1 - \frac{R - Z_0}{R + Z_0} \right) e^{-\tau/\tau} \right]
\]

(3.20)

3.4 SOURCES OF ERROR IN TDR MEASUREMENTS:

The primary sources of errors in TDR measurements are 1) Step Generator, 2) The Oscilloscope 3) Cables and connectors. 4) Selection of time window.

3.4.1 STEP GENERATOR:

The shape of the step pulse is important for accurate TDR measurements. The Device Under Test (DUT) responds not only to the step but also to aberrations on the step such as overshoot and non-flatness. Drift in step voltage with time causes remarkable change in frequency dependent permittivity spectra due to wrong time referencing. The use of proper warm up time reduces errors in measurement due to drift in pulse. The rise time of the step is very important. It decides the maximum frequency limit of frequency range in which complex permittivity spectra can be obtained. Measurement made at inappropriate rise time can yield invalid conclusions.

3.4.2 THE OSCILLOSCOPE

Oscilloscope introduces errors in measurement in several ways. The finite bandwidth of oscilloscope translates to limited rise tie. Edge with a rise times that approach the minimum rise time of oscilloscope is measured slower than they actually are. When we need to measure how a device responds to a very fast edge, the oscilloscope’s limited rise time may distort or hide some of the device response.

The oscilloscope can also introduce small errors that are due to trigger coupling into channels and channel cross talk. These errors appear as ringing and other non-flatness in display of measured channel baseline, which get superimposed on the
measured waveform. These effects are generally small and are only significant when measuring very small signals.

3.4.3 CABLES AND CONNECTORS:

Cables and connectors between step generator and DUT can significantly affect the TDR measurements. The impedance mismatch and imperfect conductors add reflections to the actual signal being measured. These reflections can distort signal and make it difficult to determine which reflections are from DUT and which are from other sources.

In addition, cables are imperfect conductors that become more imperfect as frequency of signal increases. Cable losses, which increase at higher frequencies, increase the rise time of edges and cause edges to droop as they approach their final value. Although the major sources of unwanted reflections are known in practice, it is extremely difficult to determine their exact contribution in the reflection coefficient. The effect of unavoidable reflections is reduced, by using numerical smoothening techniques in the portion in which they are present in the response signal.

3.4.4 SELECTION OF TIME WINDOW

The choice of time window through which the reflected signals are observed has to be related according to the frequency range of interest. The minimum frequency observable is \( f_{\text{min}} = \frac{1}{\text{time window}} \) while the maximum frequency observable is \( f_{\text{max}} = \frac{N}{2 \times \text{time window}} \), where \( N \) is the number of points used to sample and digitize the signal. The smaller time window causes loss of signal while larger time window includes unwanted reflections. Thus proper selection of time window is important to minimize these effects. In present work time window of 5 nS is used.

For digitizing the signal it is necessary to select number of points per waveform in the time window. To reduce the noise, an averaging of signal 16, 64 or 512 times can be done. If signal is averaged over 64 times or more, noise will be reduced significantly.
3.5 ERROR MINIMIZATION:

Using good quality cables and connectors can significantly reduce the errors due to unwanted reflections in measurement. Precision in connections can reduce errors in measurement. The errors due to drift in incident step are significant when instrument is switched on. Giving at least 30 min. warming up time for instrument can minimize these errors.

One way of error minimization is, to use of waveform subtraction. Response of a known good reference device is measured and reference waveform is stored in memory. The reference waveform could then be subtracted from the waveform measured from DUT. The result shows how response differs from reference response. This technique removes error terms common to both DUT and reference device such as trigger coupling, channels cross talk and reflections from cables and connectors. But subtracted waveform describes how DUT differs from reference device but not actual response of DUT, without errors introduced by test system. A digital error correction method called normalization can significantly reduce errors from TDR measurements. Normalization can predict how the DUT will respond to an ideal step of user defined rise time. This method can also increase bandwidth (i.e. decrease rise time) of system by some amount depending on the noise floor. The normalization method is not used in the present work.

3.6 EXPERIMENTAL PROCEDURE AND DATA ANALYSIS

The actual experimental setup is shown in Fig. 3.1 while block diagram of TDR is shown in Fig.3.2. The TDR unit is used for measurements after warming up for at least 30 min. This is necessary to minimize the time drift which occurs between two consecutive readings. The time window is kept 1.3nS to keep the unwanted reflections off screen and for the desired frequency coverage. The reflected step pulse is digitized in 1024 points and waveform is averaged over 64 times for each measurement. A flexible coaxial cable of 1 m length is connected between TDR unit and sample holder. The SMA cell had 3.5mm outer diameter and with 1.35 mm effective pin length is used as sample holder. After connecting transmission line and sample holder to TDR unit, the reflected waveform is observed carefully. The unwanted reflections in reflected step at point of contact between transmission line and TDR unit, as well as transmission line and sample
cell are minimized by ensuring proper contact between these components. The reflected pulse without sample $R_1(t)$ is averaged 64 times and acquisition is stopped. This acquired waveform is then transferred to floppy disk in TDR mainframe. Reflected pulse with sample $R_x(t)$ is recorded by the same method by putting liquid sample in SMA cell. The data files for reflected pulse without sample $R_1(t)$ and with sample $R_x(t)$, stored on 1.44 floppy disk, are transferred to computer for further processing. The 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. The file containing data array $p(t)$ and $q(t)$ is termed as TDR file.

Fig. Block diagram of Dual channel TDR unit.

Fig. 3.2 : Block Diagram of Dual channel TDR Unit

1. The mainframe TDR unit 2. HP 54754A TDR plus in module 3. Pulse generator
8. HP54750 A sampling oscilloscope 9. TDR Function 10. Display function keys
Fig. 3.10 reflected step pulse without sample $R_1(t)$

Fig. 3.11 reflected step pulse with sample $r_x(t)$

3.6.1 FOURIER TRANSFORMATION
This time domain data is converted to frequency domain data using Fourier transform. While performing Fourier transform one should be careful about the nature of the curve whose is to be obtained. Since nature of curves for \( p(t) \) and \( q(t) \) are not same, different methods of Fourier transforms are used.

The Fourier transform of \( p(t) \) is obtained by a summation method using equation

\[
p(\omega) = T \sum_{n=0}^{N} \exp(-i\omega nT) p(nT) \tag{3.21}
\]

The Fourier transformation using summation methods have some limitations that for all the sampling intervals, the nature of 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 obtained with the Samulon method which express as follows:

\[
q(\omega) = \frac{T}{1-\exp(-i\omega T)} \left[ \sum_{n=0}^{N} (q(nT) - q(n-1)T \exp(-i\omega T)) \right] \tag{3.22}
\]

In equation (4.3) and (4.4) \( \omega \) 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 Fourier transform is further used to calculate frequency dependent complex reflection coefficient \( \rho^*(\omega) \) given by equation

\[
\rho^*(\omega) = \frac{c}{i\omega d} \frac{p(\omega)}{q(\omega)} \tag{3.23}
\]

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 a true wide frequency method even reaching >20 GHz if
sufficient accuracy in $\rho^*(\omega)$ is 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, is called as ‘raw’ data. Using this ‘raw’ data complex permittivity can be determined as follows -

The basic equation for determining relative complex permittivity $\varepsilon^*$ of the sample derived from transmission line theory is conveniently written in simple form as -

$$\varepsilon^*(\omega) = \left( \frac{c}{i\omega d} \right) \frac{r_0 - r_x}{r_0 + r_x} Z \cot Z$$

(3.24)

Where, 

$$Z = \frac{\omega d}{c - \sqrt{\varepsilon^*}}$$

and $r_o$ and $r_x$ are the Fourier transform of the pulses from the cell without sample $[R_o(t)]$ and with sample $[R_x(t)]$ respectively.

If we consider only single reflection then $Z \cot Z = 1$ and eq.(4.6) can be written as -

$$\varepsilon^*(\omega) = \frac{c}{i\omega d} \frac{r_0 - r_x}{r_0 + r_x}$$

(3.25)

Eq. (4.25) indicates that the dielectric constant of unknown sample can be found if the time profile of the incident $R_o$ and reflected $R_x$ pulses are recorded within a frequency range determined by the time limits of $R_o$ and $R_x$.

A working equation more convenient than eq. (4.25) can be obtained as follows by rearranging it.

$$r_o = \left( \frac{\varepsilon^* + \frac{c}{i\omega d}}{\frac{c}{i\omega d} - \varepsilon^*} \right) r_x$$

(3.26)

If the sample cell is without any liquid i.e. with air is considered then $\varepsilon^* = 1$ and we get -
\[ r_0 = \left( \frac{1 + \frac{c}{i\omega d}}{\frac{c}{i\omega d} - 1} \right) r_1 \]  

(3.27)

where \( r_1 \) is reflected pulse in case of air with \( \varepsilon^* = 1 \). Eliminating \( r_0 \) from equations (4.26) and (4.27).

\[ \frac{r_1}{r_x} = \frac{\varepsilon^* + \frac{c}{i\omega d}}{\frac{c}{i\omega d} - \varepsilon^*} \]  

(3.28)

The equation can be rearranged to get -

\[ \varepsilon^* - 1 = \frac{\left( 1 + \left( \frac{\omega d}{c} \right)^2 \right) \rho^*}{\left( 1 - \left( \frac{\omega d}{c} \right)^2 \rho^* \right) \rho^*} \]  

(3.29)

or

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

(3.30)

Where \( A = B = \left( \frac{\omega d}{c} \right)^2 \).

Thus using eq.(4.30) one can obtain complex permittivity spectrum in the desired frequency range.

Using this complex permittivity spectrum static dielectric constant (\( \varepsilon_\infty \)), dielectric constant at infinite frequency (\( \varepsilon_\infty \)) and relaxation time (\( \tau \)) can be calculated by using Havriliak-Negami expression\(^9\) as -

\[ \varepsilon^*(\omega) = \varepsilon_\infty + \frac{\varepsilon_\infty - \varepsilon_\infty}{\left[ 1 + (i\omega \tau)^{\gamma - \alpha} \right]^\beta} \]  

(3.31)

**3.6.2 BILINEAR CALIBRATION METHOD**

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 traveling pulses superimposed on the reflection from the sample cell. The expression (4.31) above is obtained by considering the transmission line as ideal, so that \( A = B = \left( \frac{\omega d}{c} \right)^2 \) but under experimental conditions transmission line can not be an ideal and hence, \( A \neq B = \left( \frac{\omega d}{c} \right)^2 \).

To minimize the effect of unwanted reflections several workers\(^{51,52,53}\) have suggested different methods. Giese and Tieman described a method by which it is possible to take into account the influence of unwanted reflections quantitatively. Cole et. al.\(^{19}\), 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.\(^{19}\) involves experimental determination of values of \( A^* \) and \( B^* \) for getting reliable values of \( \varepsilon^* \) from \( \rho^* \) at higher frequencies. The experimental permittivity is given by:

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

(3.32)

The frequency dependent values of \( A^* \) and \( B^* \) are determined using two standard liquids. We have \( A^* = A' - i A'' \) and \( B^* = B' - i B'' \). For standard liquids the values of \( \varepsilon^* = \varepsilon_\infty - i \varepsilon_\infty\varepsilon_\infty \) can be obtained theoretically, by using suitable model such as Debye, Davidson-Cole etc.
Writing eq. (4.32) for two standard liquids -

\[
\epsilon_1^* - 1 = \frac{(1+A)\rho_1^*}{1-B}\rho_1^*
\]

\[
\epsilon_2^* - 1 = \frac{(1+A)\rho_2^*}{1-B}\rho_2^*
\]

(3.33)

(3.34)

Knowing the values of \(\epsilon_1^*, \rho_1^*\) and \(\epsilon_2^*, \rho_2^*\), eq.(4.33) and eq. (4.34) can be solved to determine the values of \(A^*\) and \(B^*\). At least two standard liquids are needed in order to evaluate four unknowns, \(A', A'', B'\) and \(B''\). Using two standard liquids gives four linear equations which can be solved to get these four unknowns at all frequencies.

It is also important to use proper calibrating liquids. It is observed that, the accuracy of calculated permittivity depends on the choice of the calibrating liquids. In order to improve the accuracy, the raw spectra of calibrating liquid should be closer to that of liquid under investigation. Cole et. al.\(^{19}\) has suggested many calibrating liquids with different kinds of dielectric spectra for determination of values of \(A^*\) and \(B^*\).

Once the values of \(A^*\) and \(B^*\) are known for give experimental set up, these values are then used to evaluate the permittivity \(\epsilon^*(\omega)\) of unknown sample using eq. (3.32). Thus using bilinear calibration process one can eliminate non-ideal configuration of transmission line and imperfections of the system including spurious reflections. The frequency dependent permittivity spectrum obtained by calibration process is called as ‘cor’ data. An example of ‘corrected spectra’ along with ‘raw spectra’ are shown in Fig. (3.6).

The dielectric parameters static permittivity (\(\epsilon_0\)), relaxation time (\(\tau\)) and (\(\epsilon_\infty\)) are calculated by fitting ‘cor’ data to suitable dielectric model using nonlinear least squares fit method \(^{54}\).
Fig. 3.6(a): Raw spectra.

Fig. 3.6(b): Corrected spectra.
3.7 DETERMINATION OF EFFECTIVE PIN LENGTH.

An effective pin length 'd' can be determined by considering equation (3.5) in the limit $\omega \to 0$

$$\lim_{\omega \to 0} \frac{p(\omega)}{q(\omega)} = \lim_{\omega \to 0} \left( \frac{c}{i\omega d} \right)$$

This expression reduces to

$$\varepsilon_\alpha - 1 = \left( \frac{c}{d} \right) \left( \frac{P_{\text{area}}}{q_\infty - q_0} \right)$$  \hspace{1cm} (3.35)

or

$$\left( \frac{d}{c} \right) = \left( \frac{P_{\text{area}}}{(q_\infty - q_0)} \frac{1}{(\varepsilon_\alpha - 1)} \right)$$ \hspace{1cm} (3.36)

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

The Fig.(4.7) 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 $\varepsilon_\alpha$.

Therefore for different known liquids we can plot a graph of $P_{\text{area}}/(q_\infty - q_0)$ versus $(\varepsilon_\alpha - 1)$, as shown in Fig.(5.8). The slope of the curve gives right hand side of equation (4.36).

$$\left( \frac{d}{c} \right) = \text{slope}$$

Thus, the effective pin length is given by:

$$d = c \times \text{slope}.$$
Fig. 5.7: $p(\text{area})$ and $q$, $q_\infty$ for a sample.

Fig. 3.8: Plot for determination of effective pin length.
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