CHAPTER V
Dielectrics may be classified according to the prevailing polarization mechanisms as: a) non-polar dielectrics; b) Polar dielectrics; c) dipolar dielectrics. When a DC electric field is applied on a dielectric material, various types of polarizations occur, and the dielectric permittivity of the material $\varepsilon$ can be written in general as,

$$\varepsilon = \varepsilon_s + \varepsilon_a + \varepsilon_i + \varepsilon_c$$

Where $\varepsilon_s$ arises from the polarization of ionic space charge as a result of the accumulation of charges at the electrodes and $\varepsilon_a$ arises from the orientational polarization of permanent dipole moments. $\varepsilon_i$ and $\varepsilon_c$ respectively arise from the ionic polarization of the positive and negative ion in ionic crystal and electronic polarization caused by the deformation of negative electron shells and positive nuclei in atoms.

The most important properties for dielectric resonator applications are dielectric constant ($\varepsilon'$), factor ($Q_o$) and temperature coefficient of resonant frequency.

The dielectric constant ($\varepsilon'$) of the material determines the resonator's dimensions at some specified frequency. Wavelengths within the dielectric resonator are shorter by a factor of $1/\varepsilon'$ than in free space. This gives rise to a first order working equation when $2\pi\varepsilon rD$:

$$D = C/f_0(\varepsilon')^{1/2}$$

where 'C' is a constant related to the velocity of light.
The unloaded Q factor ($Q_0$) depends on both dielectric and environmental losses. $Q$ is defined as the ratio of the stored energy to the dissipated energy per cycle. In terms of frequency and the impedance method, this ratio can be expressed as

$$Q_0 = \frac{f_0}{f_1 - f_2} = \frac{f_0}{\Delta f}$$

The temperature coefficient of resonant frequency ($T_f$) combines three independent factors: the temperature coefficient of the dielectric constant ($\tau_\varepsilon$); the thermal expansion of the material ($\alpha_L$) and the thermal expansion of the dielectric resonator's environment ($\tau_e$). Resonant frequency shifts due to intrinsic material parameters and the environment are related by the following equation

$$\tau_f = -\frac{1}{2} (\tau_\varepsilon + \alpha_L + A\alpha_e)$$

Here ‘A’ is independent of the position of the dielectric resonator relative to the top and bottom of the cavity walls. The typical range of values of A is between 0.05 and 1.0.

The dielectric constant ($\varepsilon'$) of the material is required to calculate the dimensions of the dielectric resonator for a given frequency. This parameter is typically measured by methods described by Hakki and Coleman (1) or the Courtney's (2) test fixture. The concepts of Q factor and dielectric loss tangent ($\tan \delta = 1/Q$) can be used interchangeably; the Q factor is used more commonly, however in microwave circuit design. The Q value of ceramics is structure-sensitive, strongly depending upon the sintering condition. The mechanism of dielectric loss of the ceramics has not been made clear, but it is certain that the major part of the loss should be ascribed to imperfections or defects in ceramics, in particular the grain boundaries.

Dielectric losses may be distortional, dipolar, conduction and interfacial. Distortional losses are related to electronic and ionic polarization and are sensed in the infrared, visible and ultraviolet regions. It would be more precise to refer to it as resonant absorption to distinguish it from
relaxation. Dipolar and conduction losses occur in processes of orientation polarization. The interfacial losses may be understood on the basis of the polarization mechanism. This model assumes the existence of a non-homogeneous structure where in the crystallite core is a better conductor than the surface layer; the later together with inter-granular layers, acts as an inter-crystallite barrier. This mechanism was analyzed in the Maxwell-Wagner capacitor model and in Volger's (3) and Krotzsch's (4) development of it.

From the equality of conductance activation energy and that of the temperature dependence of frequency corresponding to maximum tan δ, it would follow that relaxation is due to the jump motion of carriers. This idea was put forward in an earlier paper of Kamioshi(5).

Dielectric relaxation occurs during the polarization of a dielectric in an alternating electric field. This sudden application of an electric field (step field) is not followed by an instantaneous response of polarization. There is both a distortion polarization and a time dependent polarization, \( P_t(t) \), which tends asymptotically to an equilibrium value. This delayed component is generally given by dipolar and interfacial polarization. The tendency towards equilibrium of the system following the application or removal of the external field is called dielectric relaxation.

The dielectric relaxation of various types of polarizations is excited in the presence of an applied AC field on general dielectric matter. Each type of polarization has a characteristic relaxation time which is defined as the time for the attainment of equilibrium when an externally applied field is removed. The dielectric relaxation process of polarization becomes apparent when the frequency of the applied field \( E \) is not far from being of the same order of magnitude as its resonance frequency, \( \omega_r \). The resonance frequencies of electronic and ionic polarization are known to be about \( 10^{14} \) and \( 10^{12} \) Hz, respectively (6). On the other hand, the relaxation time of the polarization of ionic space charge typically varies from a few seconds to minutes (6).
The dielectric properties in the low frequency region have been explained to be based on the contributions of the space charge polarization and the dipolar polarization associated with either dipoles produced by lattice defects and impurities or permanent dipoles (7,8).

Rutile (TiO₂) has been studied for many years from two points of view, one as a semiconductor and the other as a dielectric. It has been reported that the values of the dielectric constants are from 10² to 10⁶ and the values of the loss factors, tan δ, are from 10⁻³ to 10².

These wide values are attributed mainly to differences in nonstoichiometric composition. The results in the previous paper conflict with each other, because their samples have various densities of oxygen vacancy.

It has been reported that the dielectric constants of Rutile (9) are 80 and 160 perpendicular and parallel respectively to the C’axis and that the dielectric constant of anatase (10) is 36. TiO₂ forms a basic constituent and building block of ferroelectric Titanates; it forms a link between insulator and semiconductor transitions. Previous studies have concluded that the absolute characteristic value of the dielectric constant (ε) depends on the history of the sample and also on experimental conditions (11). The relaxation of dielectric loss in these crystals around 10² to 10⁶ is found to be due to the influence of interfacial polarization (12). This polarization may be studied effectively by superimposing a suitable d.c. bias during capacitance measurements (13).
5.1. Brief review on the theory of dielectric properties of Titanates:

5.1.1. Polarization mechanism:

Dielectrics may be classified according to the prevailing polarization mechanisms as (a) Non-polar dielectrics (b) Polar dielectrics (c) Dipolar dielectrics. The first type includes substances consisting of one type of atoms. These substances become polarized when placed in an external electric field. This happens due to the relative displacement of the electronic charge with respect to the nucleus. Typical examples of such substances are gases, liquids and monatomic solids. The second type includes substances made up of molecules without permanent dipole moment. Besides electronic polarization there exists polarization induced by the modification of the relative positions of the ions in the external field (Ionic Polarization) in this class of substances. Some of the materials in this class are the ionic crystals (Halides, Oxides) paraffins, Benzene, Carbon Tetrachloride etc. the third class includes substances whose molecules possess a permanent dipole moment. In this class of materials polarization in an external field is predominately caused by the spatial reorientation of permanent dipoles. Continuation reorientation processes particularly occur in liquids and gases as well as in some solids. Not all these mechanisms may be operative simultaneously. Particular polarizability mechanisms may be dominant in certain frequency ranges of the spectral dielectric response of a solid. The atomic or ionic polarizability provides a dominant contribution to the dielectric constant (ε(0)) at very low frequencies (f). The atomic polarizability can be expressed by the Born equation

\[ \alpha_a = \frac{2\pi e^2}{\omega_0^2 a^3 \left( \frac{1}{m} + \frac{1}{M} \right) } \]

where \( \omega_0 \) is the infrared frequency for absorption maximum, a is the nearest neighbour distance of the material, m and M are the mass of the two ions. The electronic polarizability associated with atoms will provide the dominant
contribution to the dielectric constant, \( \varepsilon(\infty) \), at high frequencies where the contributions of ions or permanent dipoles are frozen out. Electronic polarizability is sometimes also called optical polarizability. This polarizability is a very rapid process and its frequency time is extremely small. The electronic polarizability can be treated from the classical as well as quantum mechanical points of view as the motion of harmonic oscillator and the expressions derived respectively for d.c. and a.c. fields takes the form

\[
\begin{align*}
\alpha_e &= \frac{e^2}{m \omega_0^2} \\
\alpha_e &= \frac{e^2}{m(\omega_0^2 - 
\omega^2)}
\end{align*}
\]

where \( e \) and \( m \) are respectively the charge and the mass of an electron, \( \omega_0 \) is the natural angular frequency of the dielectric and \( \omega \) is the frequency under consideration.

The dipolar polarizability contributes only at low frequencies. The dipolar polarizability is approximately given by

\[
\alpha_p \approx \frac{P^2}{3k_B T}
\]

where \( P \) is the dipole moment, \( k_B \) is the Boltzmann constant and \( T \) is the absolute temperature.

In experimental data of \( \varepsilon_i(\omega) \) obtained from capacitance measurements, there is often an extraneous contribution at very low frequencies due to space–charge polarization, arising from interfacial polarization at interfaces between sample and electrical contact, or between microcrystallite grains in the bulk of the sample.

5.1.2. Temperature dependence of dielectric constant

Both dielectric constant and dielectric loss are temperature dependent due to the change in the positional configuration of atoms or molecules with the rise of temperature. This leads to a change in the entropy or free energy of the system which can be related to permittivity and temperature by the equations

\[
S = S_0(T) + \frac{\delta \varepsilon \cdot E^2}{8\pi} = \Psi_0(T) + \frac{\delta E_\perp}{\delta T} \cdot E^2
\]
5.1.3. Temperature dependence of dielectric loss

As a general rule, dielectric loss (\(\tan\delta\)) of dielectric materials increases with the rise of temperature. This growth in \(\tan\delta\) is brought about by an increase in both the conduction of residual current and the conduction of absorption current. For the understanding of the change of \(\tan\delta\) with temperature, dipolar mechanism and electrical mechanism were given in the literature. The dielectric losses caused by the dipole mechanism reach their maximum at a certain definite temperature \(T_k\). This is explained as a result of drop in viscosity as temperature increases. At initial temperatures rise in \(\tan\delta\) is due to increase of degree of dipole orientation and at high enough temperature the energy required to overcome the resistance of the viscous medium called internal friction of matter decreases and hence the drop in the dielectric loss (\(\tan\delta\)).

5.1.4. Dielectric relaxations

Dielectric relaxation occurs during the polarization of a dielectric in an alternating electric field. The sudden application of an electric field (step field) is not followed by an instantaneous response of polarization. There is both a distortion polarization and a time dependent polarization, \(P_1(t)\), which tends asymptotically to an equilibrium value. This delayed component is generally given by dipolar and interfacial polarization. The tendency towards equilibrium of the systems following the application or removal of the external field is called dielectric relaxation. Thus, polarization comprises of two terms

\[ P(t) = P_0 + P_1(t) \quad \text{with} \quad P(t) \rightarrow P \quad \text{for} \quad t \rightarrow \infty \]

Where \(P_0 = \chi_0 E\), \(P_1(t) \rightarrow P_{1\infty} = \chi_1 E\)

After a sufficiently long time, \(P_1\) tends to its steady state value \(P_{1\infty}\) while the total polarization tends to \(P\). The delayed component tends to its steady state value at a rate given by the characteristic equation of relaxation phenomena.
\[
\frac{dP}{dt} = P_0 \alpha - P_0 - \frac{P}{\tau} = P_0 \alpha - \frac{P}{\tau}
\]

where \(\tau\) represents the time constant for an electric current and the macroscopic relaxation time for relaxation phenomena. The theory was first developed by Debye. The theory yields a complex polarization and a complex dielectric constant. The expressions are as follows

\[
\varepsilon' = \varepsilon_\infty + \frac{\varepsilon_s - \varepsilon_\infty}{1 + \omega^2 \tau^2}
\]

\[
\varepsilon'' = \frac{(\varepsilon_s - \varepsilon_\infty) \omega \tau}{1 + \omega^2 \tau^2}
\]

where \(\varepsilon_s\) and \(\varepsilon_\infty\) are the low frequency (static) and very high frequency dielectric constants respectively, \(\omega\) is the angular frequency and \(\tau\) is the relaxation time.

### 5.2. Results and Discussions

#### 5.2.1. Dielectric constant:

The dielectric constant \(\varepsilon\) and losses tan \(\delta\) of ceramic Titanates are not only determined by the composition but also by the processing conditions in particular the sintering and annealing have a large influence on \(\varepsilon\) and tan \(\delta\). The change of the dielectric constant depends on the grain size effecting stress distribution in the grain. The grain size dependence of \(\varepsilon\) has been investigated in ferroelectric materials (14-16).

Figures 5.1a,5.1b,5.1c depicts the dielectric constant at 100Hz and 1KHz and 10KHz whereas 5.1d,5.1e,5.1f will depicts the dielectric loss at 100Hz,1KHz and 10KHz respectively for LiTiO₃. The remaining experimental observation of variation of dielectric constant (ε) with temperature for the samples of Lithium zinc Titanate have 5.2a,5.2b,5.2c correspond to dielectric constant and 5.2d,5.2e,5.2f correspond to dielectric loss with frequencies 100Hz,1KHz and 10KHz respectively, Lithium Barium Zinc Titanate have 5.3a,5.3b,5.3c are dielectric constant figures and 5.3d,5.3e,5.3f for dielectric loss at 100Hz,1KHz and 10KHz respectively , Zinc Calcium Titanate have 5.4a,5.4b,5.4c for dielectric constant and 5.4d,5.4e,5.4f for dielectric loss with
frequencies 100Hz, 1KHz and 10KHz respectively and Magnesium calcium Zinc Titanate have 5.5a, 5.5b, 5.5c for dielectric constant and 5.5d, 5.5e and 5.5f with frequencies 100Hz, 1KHz and 10KHz respectively. In these figures dielectric constant (ε) has been plotted against the temperature (T) at three different frequencies of 100Hz, 1 KHz and 10 KHz. From these figures it was observed that the dielectric constant (ε) increases with the increase of temperature (T) and higher temperatures the peak nature was observed and it may be due to the phase transformation. It is also observed that similar nature of variation was observed in MgTiO₃-CaTiO₃ (3), TiO₂ (4), CaTiO₃-Ca(Zn₁/₃Nb₂/₃)O₃ (5), Yttrium Iron Garnet (6) and (Na₀.₅Bi₀.₅)₁₋ₓMₓTiO₃ (7).

The dielectric constant verses temperature plots exhibits maximum peak nature at around 900K in the case of Li₂TiO₃ at 100Hz, 1 KHz and 10 KHz frequencies. In Li₂Zn(TiO₃) exhibits peak nature at around 900K similar to Li₂TiO₃. But at 100Hz the discrepancy was observed, whereas 1KHz and 10KHz plots show sharp peaks. The sharp peak positions were shifted from 900K to around 800K in the case of Li₂BaZn (TiO₃) for all frequencies. It is a pronounced effect observed in this material due to Ba atom incorporation into the Li₂Zn(TiO₃) system. The temperature decreases nearly 100°C from the other materials to give rise phase transition peaks. In the case of CaZn (TiO₃) exhibits sharp peaks at 900K similar to Li₂TiO₃.

The last system Magnesium Calcium Zinc Titanate has sharp transitions at 800K for all frequencies. This effect may be due to the small grain size and particle sizes 31Å are responsible for this process. We observed almost all five compounds the dielectric constant verses temperature plots exhibits either 900 or 800K transitions it may be structural transition may be the responsible. The complex systems such as Lithium Zinc Barium Titanate and Magnesium Calcium Zinc Titanate show 800K peaks where as other three compounds Lithium Titanate, Lithium Zinc Titanate and Calcium Zinc Titanate shows 900K transitions. The dielectric constant decreases with increase of frequency. The dielectric constant almost constant up to the transition temperature was observed in all the materials.
Theoretical work was carried by Vugmeister and Glinchuk (10) and by Stachiotti and Migoni (11). Stachiotti and Migoni explained such an excess dipole moment by the interaction between a dipole and the surrounding lattice distortion.

5.2.2. Dielectric loss:

Figures 5.1d, 5.1e, 5.1f, 5.2d, 5.2e, 5.2f, 5.3d, 5.3e, 5.3f, 5.4d, 5.4e, 5.4f, 5.5d, 5.5e, 5.5f, depict the experimental observation of variation of dielectric loss (tanδ) with temperature for the samples Lithium Titanate, Lithium zinc Titanate, Lithium Barium Zinc Titanate, Zinc Calcium Titanate and Magnesium calcium Zinc Titanate at 100Hz, 1KHz and 10KHz respectively. The dielectric loss verses temperature plots of Lithium Titanate have sharp peak at 800K for 100 Hz and 750K peak for 1 KHz and around 900K peak for 10 KHz were observed. For three frequencies different relaxation peaks observed at different temperatures. In the case of Lithium Zinc Titanate the peak occurred at 900K for 100Hz, 750K and 900K for 1 KHz and 750K and 850K for 10 KHz. Still low temperature relaxation was recorded i.e., Lithium Barium Zinc Titanate around 550K for 100Hz around 780K for 1 KHz and around 700K for 10 KHz. In the case of Calcium Zinc Titanate the relaxation peak was around 670K for 100 Hz and around 600 to 700K for 1 KHz and 500K for 10 KHz. Magnesium Calcium Zinc Titanate, the dielectric loss have a sharp peaks at 820 K for 100Hz and 700K, 800K, 900K for 1 KHz and around 750K for 10 KHz was also observed. The relaxation process differs sample to sample and also exhibits 1 or 2 or 3 relaxations observed in dielectric loss verses temperature plots. In these figures dielectric loss (tanδ) has been plotted individually against the temperature (T) at three different frequencies of 100Hz, 1 KHz and 10KHz. In all frequencies the dielectric loss changes and shows relaxation peaks. It was observed nearly 800 to 900 K. A linear relationship exists between the concentration of defect and loss tangent (tanδ) at the relaxation frequency. According to Kingery (12) the relaxation frequencies and defect concentrations in the case of materials with discrete ion-jump relaxation, are \( \sim 10^3 \) Hz and \( \sim 10^{17} /\text{cm}^3 \). He also related the high
frequency relaxations to the migration of charged particles over several lattice sides through line defects. The relaxation is primarily governed by the migration of charged particles through the vacant lattice sites within the bulk portion of the grains. The broadening of the relaxation peak the possibility of more than one type of relaxation. Similar relaxation phenomenon in dielectric loss verses temperature was also observed in Ba₃ZnT₄₂O₉(17), LiO₂-Nb₂O₅-TiO₂ (18), Ca(Mg₁/₃Nb₂/₃)O₃-Ba(Zn₁/₃Nb₂/₃)O₅(19), Ba₆Ti₁₁O₄₀(20), Multilayer Ceramic Capacitors(21), BaOZnOB₂O₃(22), Al₀.5Ta₀.5O₂Mg₀.₃₃Ta₀.₆₇O₂(23), MgTiO₃CaTiO₃(24), Ca(Mg₁/₃Nb₂/₃)O₅(25,26).
Figure 5.1(a). Dielectric constant ($\varepsilon$) Versus temperature of Li$_2$TiO$_3$ for 100Hz
Figure 5.1(b). Dielectric constant ($\varepsilon$) Versus temperature of Li$_2$TiO$_3$
for 1KHz.
Figure 5.1(c). Dielectric constant ($\varepsilon$) Versus temperature of $\text{Li}_2\text{TiO}_3$ for 10KHz.
Figure 5.1(d). Dielectric loss (tan δ) Versus temperature of Li$_2$TiO$_3$ for 100Hz.
Figure 5.1(e). Dielectric loss (tan δ) Versus temperature of Li$_2$TiO$_3$ for 1KHz.
Figure 5.1(f). Dielectric loss (tan δ) Versus temperature of Li$_2$TiO$_3$ for 10KHz.
Figure 5.2(a). Dielectric constant ($\varepsilon$) Versus temperature of (Li$_2$Zn)TiO$_3$ for 100Hz
Figure 5.2(b). Dielectric constant (e) Versus temperature of (Li$_2$Zn)TiO$_3$ for 1KHz.
Figure 5.2(c). Dielectric constant(e) Versus temperature of (Li$_2$Zn)TiO$_3$ for 10KHz
Figure 5.2(d). Dielectric loss ($\tan \delta$) Versus temperature of $(\text{Li}_2\text{Zn})\text{TiO}_3$ for 100Hz.
Figure 5.2(e). Dielectric loss (tan δ) Versus temperature of (Li₂Zn)TiO₃ for 1KHz.
Figure 5.2(f). Dielectric loss (tan δ) Versus temperature of (Li₂Zn)TiO₃ for 10KHz.
Figure 5.3(a). Dielectric constant (ε) Versus temperature of (Li₂BaZn)TiO₃ for 100Hz
Figure 5.3(b). Dielectric constant($\varepsilon$) Versus temperature of (Li$_2$BaZn)TiO$_3$ for 1KHz
Figure 5.3(c). Dielectric constant (ε) Versus temperature of (Li₂BaZn)TiO₃ for 10KHz
Figure 5.3(d). Dielectric loss (tan δ) Versus temperature of (Li₂BaZn)TiO₃ for 100Hz.
Figure 5.3(e). Dielectric loss (tan δ) Versus temperature of (Li₂BaZn)TiO₃ for 1KHz
Figure 5.3(f). Dielectric loss (tan δ) Versus temperature of (Li₂BaZn)TiO₃ for 10KHz
Figure 5.4(a). Dielectric constant (ε) Versus temperature of (CaZn)TiO₃ for 100Hz
Figure 5.4(b). Dielectric constant ($c$) Versus temperature of (CaZn)TiO$_3$ for 1KHz
Figure 5.4(c). Dielectric constant ($\varepsilon$) Versus temperature of (CaZn)TiO$_3$ for 10KHz
Figure 5.4(d). Dielectric loss (tan δ) Versus temperature of (CaZn)TiO₃ for 100Hz
Figure 5.4(e). Dielectric loss (tan δ) Versus temperature of (CaZn)TiO₃ for 1KHz
Figure 5.4(f). Dielectric loss (tan δ) Versus temperature of (CaZn)TiO₃ for 10KHz
Figure 5.5(a). Dielectric constant ($\varepsilon$) Versus temperature of (MgCaZn)TiO$_3$ for 100Hz
Figure 5.5(b). Dielectric constant (ε) Versus temperature of MgCaZn)TiO$_3$ for 1KHz
Figure 5.5(c). Dielectric constant ($\varepsilon$) Versus temperature of (MgCaZn)TiO$_3$ for 10KHz.
Figure 5.5(d). Dielectric loss ($\tan \delta$) Versus temperature of $(\text{MgCaZn})\text{TiO}_3$ for 100Hz.
Figure 5.5(e). Dielectric loss (tan δ) Versus temperature of (MgCaZn)TiO₃ for 1KHz
Figure 5.5(f). Dielectric loss (tan δ) Versus temperature of (MgCaZn)TiO₃ for 10KHz
Table 5.1 depicts the Dielectric Constant and Dielectric Loss values at 700K.

<table>
<thead>
<tr>
<th>Materials</th>
<th>Dielectric Constant (c) at 10KHz</th>
<th>Dielectric Loss tanδ at 10KHz</th>
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<tbody>
<tr>
<td>Li₂TiO₃</td>
<td>5.2</td>
<td>0.04</td>
</tr>
<tr>
<td>(Li₂Zn)TiO₃</td>
<td>25</td>
<td>0.55</td>
</tr>
<tr>
<td>(Li₂ZnBa)TiO₃</td>
<td>46.5</td>
<td>1.9</td>
</tr>
<tr>
<td>(CaZn)TiO₃</td>
<td>11.4</td>
<td>1.2</td>
</tr>
<tr>
<td>(MgCaZn)TiO₃</td>
<td>895</td>
<td>0.8</td>
</tr>
</tbody>
</table>
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