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Dielectric Investigations of CoO Doped ZnO-TeO_2-B_2O_3 Glasses

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Abstract. A glass system with the composition 20ZnO-(25-x)TeO_2-55B_2O_3:x mol% CoO, where 'x' varies from 0.3 to 1.2, was synthesized using the conventional melt-quench technique. The prepared glass samples were characterized by XRD and SEM, confirming their non-crystalline state. This study experimentally measured three key dielectric properties over a frequency range of 1 to 100 kHz and a temperature range from room temperature to 200°C. The results show an increasing trend in these parameters with rising CoO content, indicating a higher degree of disorder due to the presence of trivalent cobalt ions, which act as network modifiers at higher concentrations. The variation in activation energy and other dielectric properties is analysed in detail, considering the oxidation states of cobalt ions and the resulting structural changes within the glass matrix. These findings suggest that the tailored dielectric properties make the glass system suitable for various electronic applications.

KEY WORDS: Cobalt ions; dielectric properties; tellurite; borate glasses; conductivity.

1 Introduction

To have more efficient devices for storing the data, attention is being concentrated on developing superior glasses and ceramics that can satisfy the stringent requirements of high-performance storage successfully [1–3]. Considering these factors, heavy metal oxide-based glasses, and particularly those containing tellurite and antimony oxides, are highly promising due to their unique atomic structure, which indicates superior electrical and optical properties [4–8]. The modification of glasses with modifier oxides, such as zinc oxide, improves dielectric properties, so these materials could be among the most appropriate candidates for being used in data storage applications [9–11]. To introduce zinc oxide, for instance, glass matrix modification is incorporated into the glass structure, which allows to achieve higher values of dielectric constants, stability over a broad range of temperatures, and lower dielectric losses.

One of the significant advances in this field is the incorporation of cobalt oxide, CoO, into such glasses, which imparts interesting structural and electronic properties to the glasses. The CoO doped glasses show significantly improved dielectric properties that can be used as storage media [12–15]. CoO doping is found to extend the network stability of the borate-tellurite network and, hence, enhance the densities and thermal durability, both of which are important characteristics for long-lasting data storage applications [16–18]. The divalent nature of cobalt ions will also provide a greater polarizability than that of zinc ions, providing further support for the promotion of the dielectric constant and giving increased strength of response to electric fields. The ions also stabilize the material by ensuring a consistent dielectric constant and loss values over a wide temperature and frequency range. This may be partly due to the dynamic dielectric relaxation effects of Co^{2+} ions, which cause space charge polarization; this significantly affects the dielectric response, especially at lower frequencies.

Recent studies have mainly dealt with the effect that cobalt oxide has on various glass systems; however, there is a scarcity of studies dealing specifically with cobalt-doped borate glasses in conjunction with tellurite and zinc oxide [19–28]. This particular compositional mixture is anticipated to offer a tellurite-borate glass network that would exhibit better dielectric behavior for the purpose of use in electronic and storage applications. Against this backdrop, there lies an open door to examine the effects of Co ions in the dielectric behavior of tellurite-borate glass networks. Complete knowledge of these could be the benchmark for designing optimized performance materials for future electronic devices as well as for high-capacity storage solutions.

2 Experimental Procedure

To prepare the selective composition of ZnO-TeO_2-B_2O_3:CoO glasses, conventional melt quenching, followed by annealing process have been used. The de-

tails of various samples prepared with their composition and labelling are provided below for better understanding of the investigations carried out

$$\mathrm{ZTBC_3} : 20\mathrm{ZnO} - 24.7\mathrm{TeO_2} - 55\mathrm{B_2O_3} : 0.3\mathrm{CoO}$$
$$\mathrm{ZTBC_6} : 20\mathrm{ZnO} - 24.4\mathrm{TeO_2} - 55\mathrm{B_2O_3} : 0.6\mathrm{CoO}$$
$$\mathrm{ZTBC_9} : 20\mathrm{ZnO} - 24.1\mathrm{TeO_2} - 55\mathrm{B_2O_3} : 0.9\mathrm{CoO}$$
$$\mathrm{ZTBC_{12}} : 20\mathrm{ZnO} - 23.8\mathrm{TeO_2} - 55\mathrm{B_2O_3} : 1.2\mathrm{CoO}$$

As per the above-mentioned compositions, TeO_2, B_2O_3 in the form of H_3BO_3, ZnO and CoO of analytical grade quality are weighed, then mixed all the chemicals until smooth powder is obtained using piston and agate mortar. Then, grain free mixed powder is transferred into platinum crucible from agate mortar, later crucible is placed in the PID controlled high temperature muffle vertical furnace maintained in the temperature range of 880°C to 930°C. After heating up, the powder slowly converted into liquid, ultimately after reaching the condition of bubble-free liquid, it is taken out carefully and poured on to the brass plate that has pre-arranged brass strips to get the proper dimensions of samples for further characterization. Immediately, as-prepared samples are directly transferred from brass plate to annealing furnace which was already maintained in the range 390°C to 430°C temperature to avoid breakage of glass samples due to abrupt decrease of temperature and facilitates gradual cooling of samples from high temperature to room temperature over a period of 8 to 12 hours. Later, samples are cut, grinded and polished to make them ready for following structural characterization and dielectric investigations [15, 18].

(a) Density measurements

Using o-xylene as buoyant liquid, the densities of the samples are measured by taking conventional Archimedes' principle.

(b) Physical parameters

After getting the experimental density values, theoretical densities are estimated and validated the densities of samples based on both values. These values are considered for calculation of important physical parameters which provides information about structural network, ionic radius, coordination behaviour etc. Investigating these parameters is essential to explore and correlate the variations in dielectric properties of glass samples against temperature and applied frequency.

(c) Basic characterization

Solid state behavior of prepared glass samples is tested by using standard X-Ray Diffraction (XRD) technique and surface morphology has been assessed using scanning electron microscopy (SEM) images with different magnifications.

(d) Dielectric investigations

It's the crucial part of the measurements. Different dielectric parameters such as dielectric constant, loss and conductivity are measured in the temperature range from 30°C to 200°C over a broad frequency range of 1, 10 & 100 kHz with HP Model 4263-B LCR meter.

3 Results and Discussion

X-ray diffraction scans did not show characteristic sharp peaks of crystalline materials. However, a broad bump appears at an angle of diffraction $2\theta = 26^\circ$ attributes to amorphous nature of glass samples. Figure 1 resembles the same XRD patterns for all test samples and no prominent changes in support of crystalline nature were seen. In agreement with XRD patterns, scanning electron microscopic pictures reveal that there are no clusters, grains with proper boundaries. Thus, both XRD scans and SEM images allow us to conclude that all the prepared glass samples do not show any kind crystalline nature that sometimes may be possible due to regular annealing process or gradual cooling treatment [15,29]. In the present work, the SEM images of two samples viz., $ZTBC_6$ and $ZTBC_{12}$ are given in Figure 2. The irregular shaped microcrystal grains in both the images of samples are seen which points out that those grains may be occurred due to annealing treatment; however, no symmetrical arrangement of

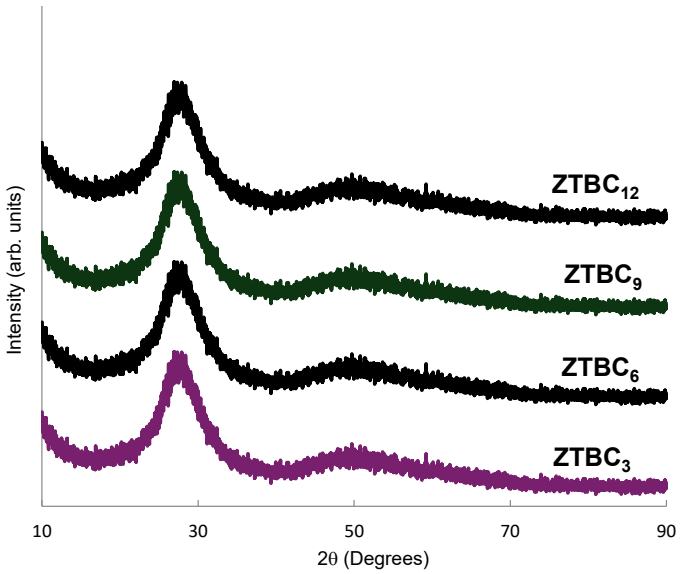

Figure 1. XRD patterns of ZnO-TeO_2-B_2O_3:CoO glasses.

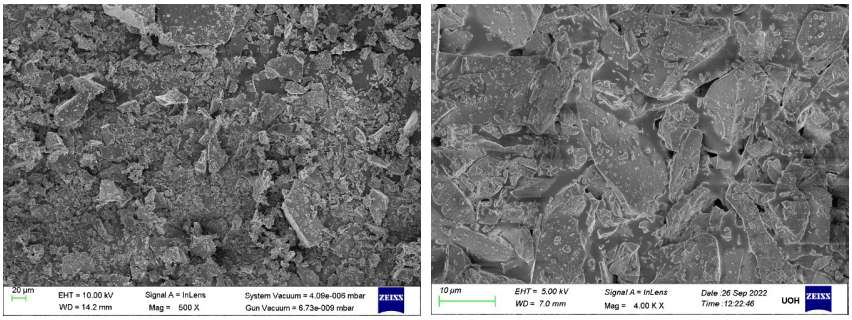

Figure 2. SEM pictures of $ZTBC_3$ & $ZTBC_6$ glass samples (Left to Right).

grains in any of the samples was observed. Thus, SEM images clearly confirm that samples are of amorphous in nature even though few microcrystals were present.

Table 1 displays the evaluated physical parameters of the B_2O_3–TeO_2–ZnO:CoO glasses, including density, average molecular weight, cobalt ion concentration (N_i), mean cobalt ion separation (r_i) and polaron radius (r_p). The detailed values are shown in the table depicted below.

Table 1. Experimental physical parameters of ZnO-TeO_2-B_2O_3:CoO glass samples

Glass	Avg. Mol. Wt.	Density (g/cm^3)	Concentration of 'Cobalt' ions N_i (10^{21}/cm^3)	Interionic distance of 'Cobalt' ions r_i (Å)	Polaron radius r_p (Å)
$ZTBC_3$	94.22	3.894	7.47	0.51	0.21
$ZTBC_6$	93.96	3.897	14.98	0.41	0.16
$ZTBC_9$	93.71	3.901	22.55	0.35	0.14
$ZTBC_{12}$	93.45	3.905	30.17	0.32	0.13

With an increase in CoO concentration from 0.3 to 1.2 mol%, several physical parameters of the ZnO-TeO_2-B_2O_3 glass system vary considerably, as shown in Table 1. The average molecular weight slightly decreases from 94.22 to 93.45, which indicates a denser network through the inclusion of CoO. Further, the density marginally increases as CoO concentration increases within the glass system from 3.894 to 3.905 g/cm^3, as reflected with a denser glass network.

In CoO content within the glass matrix, the concentration of cobalt ions (N_i) increases sharply from 7.47 to 30.17 ions/cm^3. This amount of ion concentration causes a reduction in interionic distance (r_i) from 0.51 to 0.32 Å and reduces the polaron radius from 0.21 to 0.13 Å. As the CoO concentration is increased, reduction in interionic distance (r_i) and polaron radius (r_p) implies that there is tight packing of cobalt ions within the glass network. Better packing between

cobalt ions may enhance the interaction between cobalt ions, which can lead to more easier hopping conduction of charge carriers. In other words, electrons or holes could transport more easily between neighboring cobalt ions, thereby improving the electrical conductivity of the glass. The localized electric fields increases the cobalt ion concentration within the glass matrix, thereby increasing the localized electric fields. This therefore further expands the material's dielectric properties. The overall glass structure is improved by a higher ionic and electronic interaction, making the material suitable for high electrical performance and stability [28, 29].

3.1 Dielectric constant

Figure 3 shows variation of dielectric constant ε' at 1 kHz with temperature for ZnO-TeO_2-B_2O_3 glass system doped with various concentrations of cobalt oxide. Experimentally obtained data exhibits an increase in the dielectric constant with respect to temperature in all the samples, but with some higher variations as between the low and high CoO concentration samples.

The low concentration sample $ZTBC_3$, 0.3mol% CoO, shows a dielectric constant starting at 7.37 at 30°C. There is a relaxation bump that appears between 90°C and 110°C, where the dielectric constant increases from 8.33 to 9.26. This is considered an indication of the dipolar relaxation processes acting within the glass matrix wherein the alignment of dipoles was found to be more effective at those temperatures. At 200°C, the dielectric constant reached 11.24, which has revealed enhanced polarization at high temperature levels, possibly through growing ionic mobility or structural changes in the glass. Similar trends have been found for the samples doped by rising concentrations of CoO [15–18].

The high concentration sample, $ZTBC_{12}$ (1.2 mol% CoO), had an initial dielectric constant value of 10.27 at 30°C. The relaxation bump shifts to an earlier position, between 60°C and 100°C, where the dielectric constant increases from 12.64 to 13.48. This reveals a relaxation that occurs at lower temperatures and would imply that the high concentration of CoO promotes higher active dipolar or ionic polarization mechanisms. The dielectric constant for $ZTBC_{12}$ increased significantly by the 200°C mark to reach a value of 20.36, which is another indicator of large polarization resulting from the increased cobalt density and their effect on the glass network.

This trend indicates that there is an increase in the dielectric constant at higher CoO concentrations with temperature. This behavior may be attributed to the increased availability of cobalt ions; these ions may assist the polarization process inside the glass. The higher value as well as earlier appearance of the relaxation bump in high concentration samples was evidence that more CoO also facilitate easier dipole alignment and increases the dielectric response. This glass system ZnO-TeO_2-B_2O_3 with higher content CoO has been found highly effective in electrical energy storage at higher temperatures. It is, thus, appropriate candidate

material for the applications requiring high-temperature dielectric properties.

The variation of ε' for $ZTBC_9$ sample with frequency at various temperatures from 30°C to 200°C and with frequency at specific temperatures depicts the frequency-dependent dielectric behavior as shown in the inset of Figure 3. From Figure 3, it can be seen that at 30°C, ε' has the tendency to decrease with an increase in frequency, where at 1 kHz; ε' is 9.63 and 7.57 at 100 kHz. This is because at high frequencies, dipoles and charge carriers within the glass matrix cannot realign or move fast enough to keep pace with the rapidly oscillating electric field, leading to lesser polarization and, thereby, a lower dielectric constant.

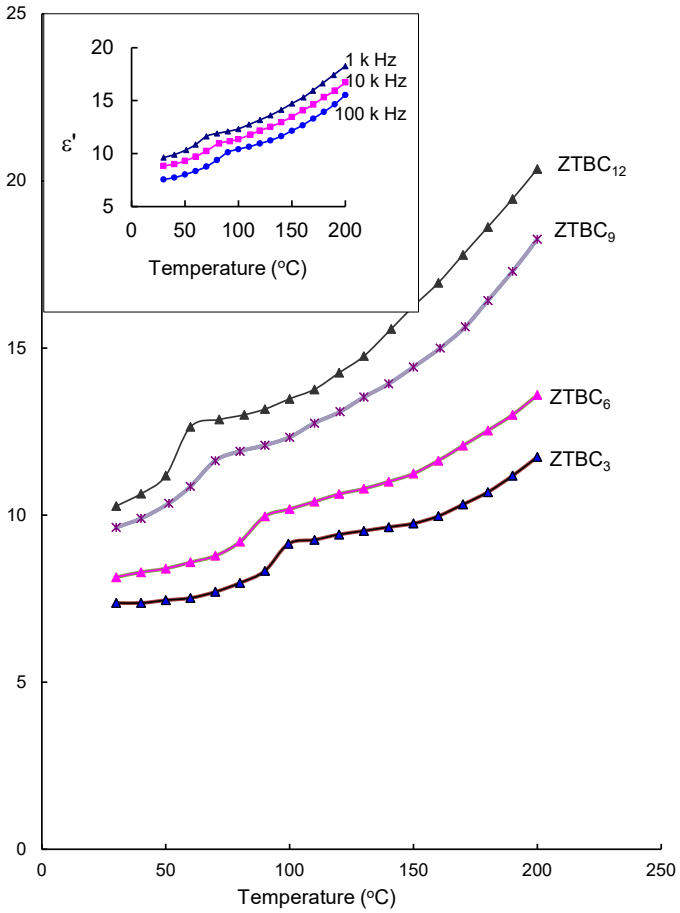

Figure 3. Comparision plot of variation of dielectric constant with temperature measured at 1 kHz for ZnO-TeO_2-B_2O_3:CoO glass samples. Inset shows the variation of dielectric constant with temperature of the glass ZTBC9 at different frequencies.

At higher temperatures of 200°C, the dielectric constant increases at all frequencies. These values are as follows: it is 18.26 at 1 kHz, 16.73 at 10 kHz, and 15.54 at 100 kHz. For the same reason, increased temperature provides more thermal energy, facilitating the movement of dipoles and ions as well as making it easier for them to align with the electric field, even at higher frequencies. Nevertheless, dielectric constant still decreases with frequency as higher frequencies also continue limiting the polarization response. This indicates both temperature and frequency have significant influence on the dielectric properties of the ZnO-TeO_2-B_2O_3 glass system. Due to better polarization mechanisms, lower frequency and higher temperatures result in higher dielectric constants.

3.2 Dielectric loss ($\tan\delta$)

Change of tan δ with temperature at frequency of 10 kHz for all the samples under investigation is shown in Figure 4. Temperature dependence of tan δ of sample $ZTBC_3$ at different frequencies of 1, 10 and 100 kHz is presented as inset of Figure 4. From the results of the experiment, in the case of dielectric loss, the variation of the sample $ZTBC_3$ was very apparent between 0.00455 at 30°C to 0.00885 at 200°C with strong relaxation effect like narrow peak between 95°C to 127°C having a peak value of 0.00811 at 110°C. The same trend can be observed for the rest of the glass samples. Though the broadening of relaxation peak with an increase in content of CoO has been observed, it has become significant in the high frequency side. The loss decreases with increase in frequency. As the frequency increases, the broad bump shifts to higher temperatures, and the frequency peak also moves to a higher range with rising temperature [15–18]. The increasing content of CoO in the ZnO-TeO_2-B_2O_3 glass system would shift the dielectric loss relaxation peak to relatively lower temperatures because a higher concentration of CoO introduced a higher concentration of cobalt ions into the glass matrix.

The ions give more sites for polarization and lower the activation energy required in the movement of the dipoles. Higher concentrations of cobalt ions render easier alignment of the dipole at lower temperatures, hence inducing an earlier onset of the relaxation peak. The lowered energy barrier pertaining to the reorientation of the dipoles presents one basis for why the dielectric loss peaks at higher temperatures as the concentration of CoO increases.

Table 2. Dielectric loss related parameters for ZnO-TeO_2-B_2O_3:CoO glass samples

Sample	$(\tan\delta_{max})_{avg}$	AE of dipoles (eV)	AE of conduction (eV)
$ZTBC_3$	0.0081	3.14	0.51
$ZTBC_6$	0.0086	2.87	0.42
$ZTBC_9$	0.0099	2.46	0.34
$ZTBC_{12}$	0.0122	2.15	0.29

Table 2 summarizes the dielectric loss maxima and activation energy (AE) for dipoles in electron volts for all glass samples. Data from this table indicates that sample $ZTBT_{12}$ had the highest dielectric constant and loss, and the lowest activation energy for dipoles. This should generally indicate that $ZTBT_{12}$ is more easily polarized in the electric field and may be associated with a gap of the band change. From the Table, it is clear that while maximum dielectric loss-tan δ-increases from $ZTBT_3$ to $ZTBT_{12}$, the activation energy for dipoles decreases from 3.14 eV to 2.15 eV. The result verifies that more energy is not needed for the rotation of dipoles due to increased dielectric loss. In the same way, activation energy for the conduction of electric currents also reduces from 0.51 eV to 0.29 eV while verifying that less energy is required for the conduction of electric currents by raising dielectric loss. In general, the table reflects the dielectric properties of the samples and expresses the relationship between the dielectric parameters.

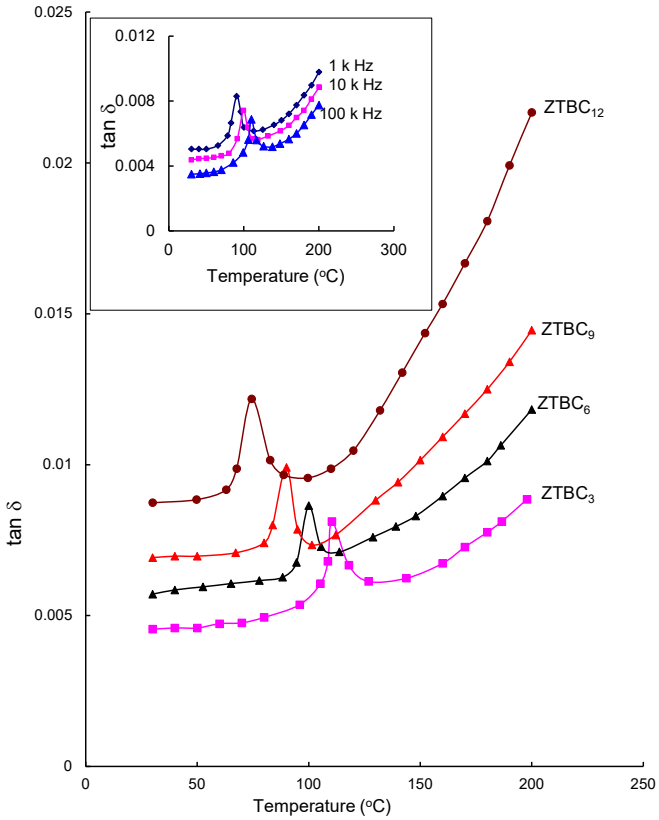

Figure 4. A comparision plot of variation of dielectric loss with temperature measured at 10 kHZ for ZnO-TeO_2-B_2O_3: CoO glass samples. Inset shows the variation of dielectric loss with temperature at different frequencies for the glass $ZTBC_3$.

3.3 A.C. conductivity

The estimation of the AE for dipoles is performed utilizing the subsequent equation:

$$f = f_0 \exp\left(-\frac{w_d}{kT}\right). \tag{1}$$

The a.c. conductivity values are evaluated by taking the experimental dielectric constant and loss values pertaining to different temperatures and frequencies using the mathematical relation given below [15, 29]:

$$\sigma_{\text{ac}} = \omega \varepsilon_0 \varepsilon' \tan\delta\,. \tag{2}$$

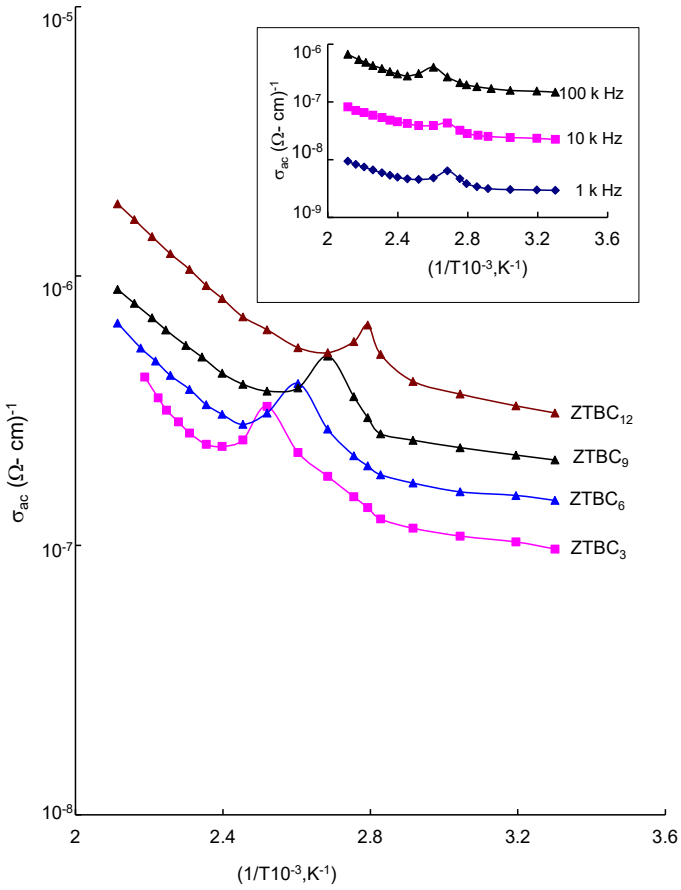

Figure 5. Variation of σ_{ac} with $1/T$ for ZnO-TeO_2-B_2O_3:CoO glasses at the frequency of 100 kHz. Inset shows variation of σ_{ac} with $1/T$ at different frequencies for the glass $ZTBC_6$.

The relationship between a.c. conductivity, frequency and temperature are depicted as log σ_{ac} against $1/T$ (Figure 5). From the figures, it can be seen that σ_{ac} increases with the inclusion of CoO at any given frequency and temperature. From the theoretical point of view, it is widely appreciated that the conductivity depends on the product of dielectric constant and dielectric loss, and, thus, the enhancement in conductivity comes due to the trends found in ε' and $\tan\delta$. At 100 kHz, at all temperatures, the a.c. conductivity of all samples rises with the incorporation of trivalent cobalt ions: this modification changes the network of tellurite borate glass. Since trivalent cobalt ions are modifiers, bonding defects in the glass contribute to their mobility as charge carriers. Therefore, it increases the CoO concentration with further increases in the Co^{3+} ion concentration. To be most pronounced, such increments occur in a sample with the highest concentration of CoO. The enhanced level of Co ions will more effectively contribute to the higher values of dielectric parameters, such as a dielectric constant and dielectric loss, as well as a conductivity. In addition, zinc and tellurite ions are modifier ions, like trivalent cobalt ions, which will also favour space charge polarization and thus the increase in overall a.c. conductivity [13–15, 29].

This differentiation in a.c. conductivity at different temperatures and frequencies with high concentration of CoO, is shown as an inset of Figure 5. These changes indicate a region of dielectric breakdown which would be by the reorientation of dipoles in the glass matrix. This behavior shows that cobalt ions are playing a more relevant role for enhancing the electrical properties of glass, which makes it more charge movement and polarization favor.

4 Conclusion

The dielectric constant (ε'), loss, and conductivity of ZnO-TeO_2-B_2O_3:CoO glass samples have been measured in the frequency range of 1-100 kHz and temperature range of 30–200°C. Samples prepared by the melt-quenching technique have been characterized by XRD and SEM, both showing that the samples were amorphous with no crystalline structure. The dielectric constant increases with temperature for all ZnO-TeO_2-B_2O_3 glass samples, but the effect is pronounced in the higher CoO concentration samples. For the low concentration sample $ZTBC_3$ (0.3 mol% CoO), there is a well-defined relaxation bump between 90°C and 110°C, which suggests effective dipolar relaxation. Even at 200°C, the dielectric constant is 11.24, suggesting increased polarization; an effect that might be mainly due to increased ionic mobility. This trend of increase in dielectric constant both with temperature and CoO content is observed consistently in all the samples. The dielectric constant for $ZTBC_{12}$ takes a value as much higher as 20.36 reflecting a high polarization due to the higher density of cobalt ions and their influence on the glass network. In all the samples, dielectric loss increases gradually from 30°C to 70°C after which there are remarkable dielectric relaxation effects between 70°C to 110°C as manifested through temperature-

dependent shifting peaks. The maximum dielectric loss value of 0.0087 has been reported at room temperature for $ZTBC_{12}$. As the maximum dielectric loss tan δ increases from $ZTBT_3$ to $ZTBT_{12}$, activation energy for dipoles decreases from 3.14 eV to 2.15 eV and for conduction from 0.51 eV to 0.29 eV, indicating that lesser energy is required for both dipole rotation as well as current conduction with higher dielectric loss. The rise in a.c. The conductivity in all the samples is attributed to the presence of trivalent cobalt ions, which influences the structure of the tellurite borate glass. The low activation energy of both dipoles and conduction in $ZTBC_{12}$ indicates a strong space charge polarization effect. These properties make them ideal candidates for capacitors, sensors, and several other electronic devices.

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