Synthesis and Characterization an Zn Doped $CaTiO_3$ Ceramics

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Abstract

In the present research study, the $ZnCaTiO_3$ samples were synthesized by using solid state reaction route with equal molar ratio using metal oxide powders of 99.9% purity. The samples were well grained and calcined at 900^{o}C for 10 hours and sintered at 950^{o}C for 6 hours. By varying the composition of Zinc then study the properties $CaTiO_3$. The characterization of the samples were carried out by using XRD for Structural characterization, SEM with EDAX for Micro structural and HIOKI 3532-50 LCR Hitester for Dielectric studies. The structures of the samples were estimated as rhombohedral by using XRD studies. From the SEM micrographs the avg. grain sizes were varied in the range of 0.25-5μm for all the compositions of Zinc. From EDAX the elemental compositions are well stoichiometric.

Key words: *$ZnCaTiO_3$, Solid state Reaction Route, Sintering, XRD, SEM, EDAX, Grain Size*.

Introduction:

In the last three decades there has been a phenomenal transformation in microwave communication systems such as mobile telecommunication systems, satellite communication and broadcasting systems, and global positioning systems. The rapid development in microwave communication systems was made possible with the use of dielectric ceramics as enabling materials for resonators, filters and other key components in microwave components. For these applications the ceramic materials are required to have a high relative permittivity (ε_r), low dielectric loss, and near zero temperature coefficient of resonant frequency (τ_f). The combination of these requirements greatly restricts the ceramic dielectrics available for applications in microwave systems (1).

In order to meet the demands of the next generations of communication systems, it is necessary to improve the performance of existing materials as well as to discover materials. System designers are particularly focusing on improving performance and miniaturization of microwave devices, which has increased the interest in the development of high permittivity materials with lower losses. The cost of production is also putting a considerable strain on the dielectric materials currently in use. High-permittivity ceramics currently used in state-of-the-art microwave systems are mainly based on solid solutions described by the general formula _RE is a rare-earth element_. Such types of ceramics exhibit relative permittivity of 80–90 and Q*f* between 5000 and 10 000 GHz (2, 3). High-permittivity materials based on $CaO – Ln_2O_3 – Li_2O – TiO_2$ ceramics with perovskite structures reported by Takahashi *et al (*4) show improved properties. Good quality microwave dielectric ceramics with relative permittivities in the range of 90–110 and Q*f* values 2000 GHz have been reported in CaTiO3 – $Ln1_{/2}Li_{1/2}TiO_3$ solid solutions where Ln = Nd^{3+} and Sm^{3+} (4, 5). These materials were developed following a common approach of mixing CaTiO3 dielectric ceramics with positive TC*f* and $Ln1_{/2}Li_{1/2}TiO_3$ ceramics with negative TC*f* to form temperature stable high-permittivity ceramics with moderate losses. Several authors have also reported attempts to improve the properties of $CaTiO_3$-based high- permittivity dielectric ceramics using a range of additives (6-9).

1. Preparation and Characterization methods

1.1 Synthesis

A series of Zinc doped Calcium Titanates ($Zn_xCa_{1-x}TiO_3$) were synthesized in air by the high temperature solid-state reaction mentioned elsewhere. Calcination of the samples was carried at 900^0C for 24 hours. Same conditions were maintained for all the samples. The resulted powders were made into pellets by hydraulic press at a pressure of 10 tons. The pellets were then sintered at 950^0C for 6 hours. These pellets were then annealed at ~300°C for about 2 hours under vacuum (10^{-2} torr) to remove the strain introduced due to mechanical stress. Formation of the compounds was confirmed by powder X-ray diffraction.

1.2 XRD

X-ray diffraction profiles were recorded at room temperature with Seifert X-ray diffractometer using Ni-filtered Cu-K_α radiation (λ = 1.54056A^o) at a rate of 2°/min. in the range of 10°-90°.

1.3 SEM and EDAX

Surface morphological studies (grain size, twin boundaries, stress, strain, dislocation etc.) are routinely carried by Scanning electron microscopy (SEM) with energy dispersive analysis of x-rays (EDAX). The scanning electron microscopy is used in determining the surface structure and EDAX is used in studying the composition of the compounds.

1.3 Dielectrical properties

The experimental variations of the dielectric constant (ε_r) and dielectric loss (tan δ) has been carried out by the two terminal capacitance methods. In the present study of ceramics, the dielectric parameters such as dielectric constant(ε_r), ac conductivity (σ_{ac}) and dielectric loss

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(tan δ) of the samples were measured using a HIOKI 3532 – 50 LCR Hitester in the frequency range of 100 Hz to 1MHz at different temperatures.

2 Results and Discussion

2.1 Crystal Structure

Fig.1 show the x – ray diffraction profiles of the prepared set of zinc doped calcium titanates $Zn_xCa_{1-x}TiO_3$ (x=0.1, 0.3 & 0.5). In these figures the intensity of the scattered x–rays is plotted against the scattering angle (2θ). From these figures it is observed that almost all the samples formed sharp peaks with high intensities. The sharp peaks in these figures confirm the formation of the compound. It also reveals that the compounds are Polycrystalline. These profiles have been used for the interpretation of the crystal structure. The crystal structure of the sample was found as rhombohedral with space group R_3^- (148).

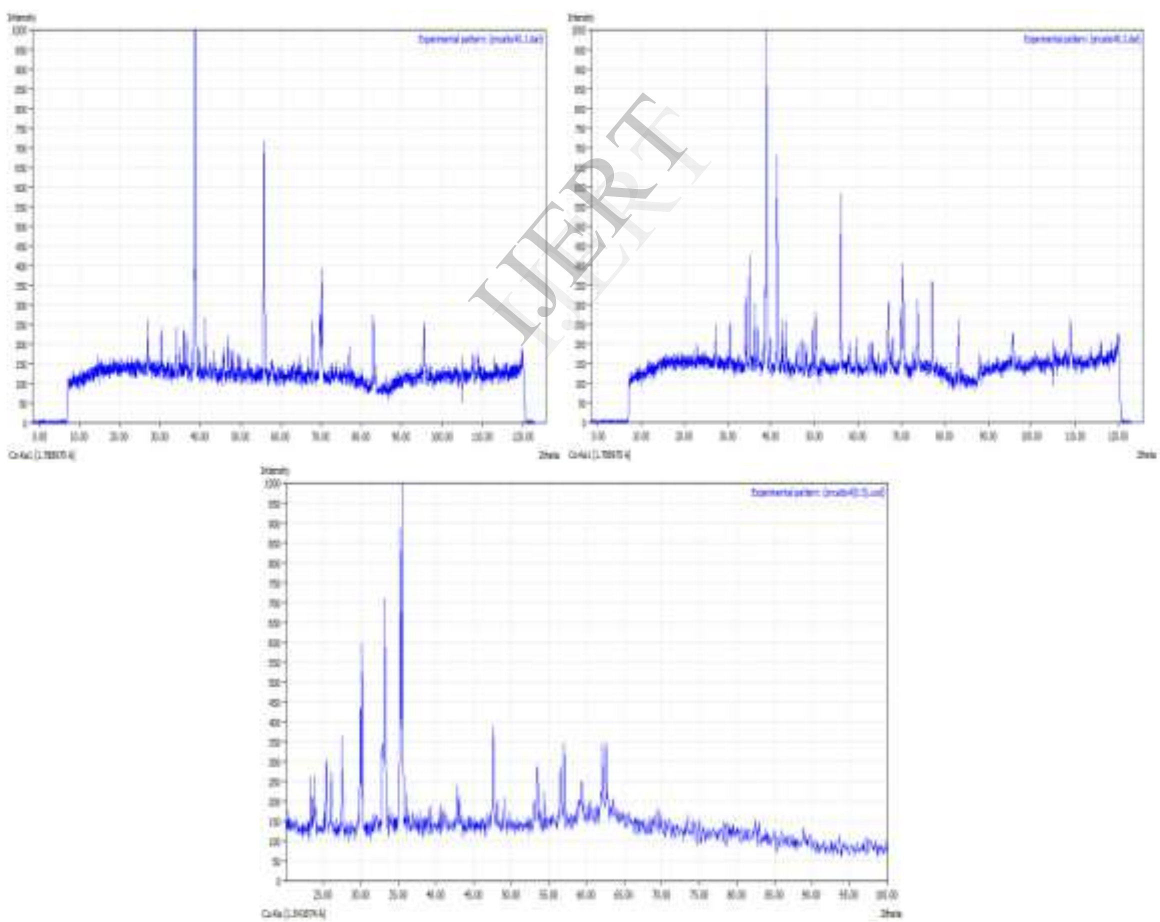

Fig.1 Shows the XRD plots for $Zn_xCa_{1-x}TiO_3$ at x=0.1, 0.3 & 0.5

2.2 SEM with EDAX:

Figure.2 depict the SEM micrographs of the prepared samples $Zn_xCa_{1-x}TiO_3$(x=0.1, 0.3 & 0.5) respectively for the magnification of 5000. It is assumed that all compounds have proper calcinations and sintering and it yields a perfect grain formed in Scanning electron micrographs. The average grain size is 5-10µm and has been calculated for all the compounds under same magnification. It is observed from SEM micrographs of $Zn_xCa_{1-x}TiO_3$ (x=0.1, 0.3 & 0.5) samples having high porosity. The grain size of the target samples depends on zinc concentration. The grain size increases as zinc concentration increases similar to that of Crystallite size. The grain size of the target samples depends on zinc concentration. From EDAX it is observed that the compounds are well Stoichiometric and perfect structures which were observed in fig.3.

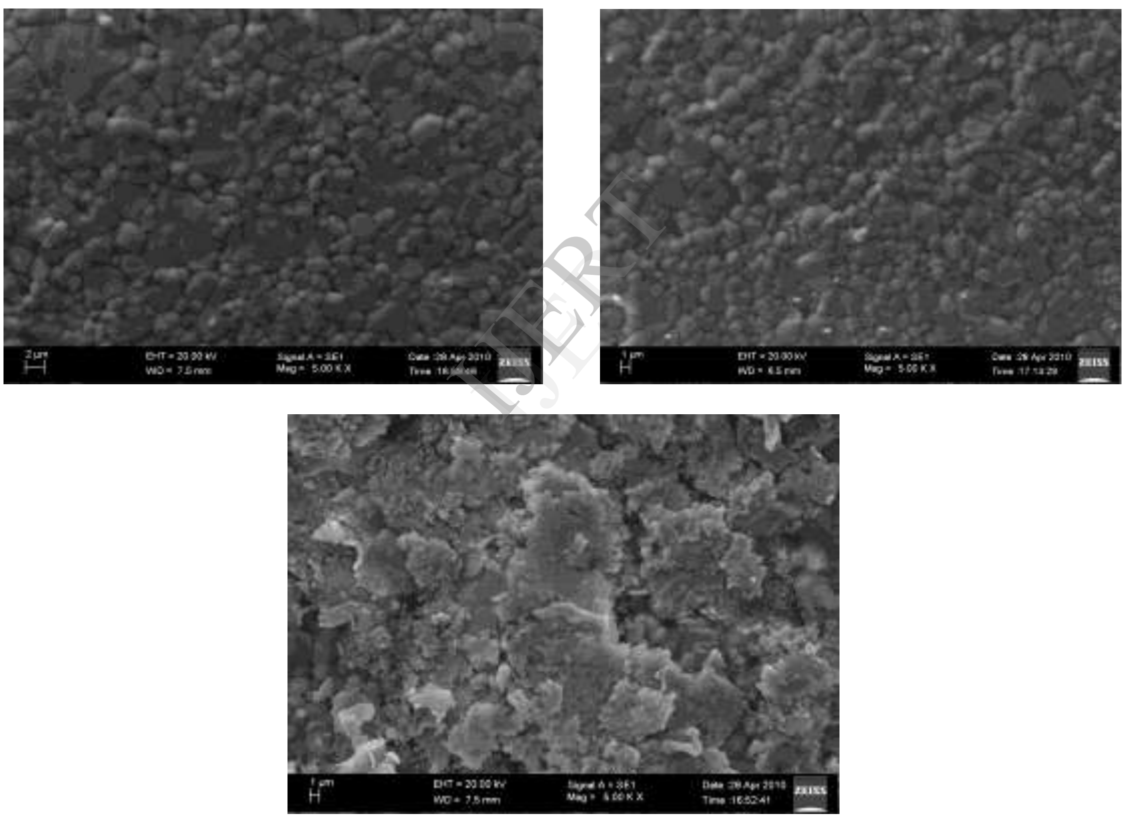

Fig.2 Shows the Micrograph plots for $Zn_xCa_{1-x}TiO_3$ at x=0.1, 0.3 & 0.5

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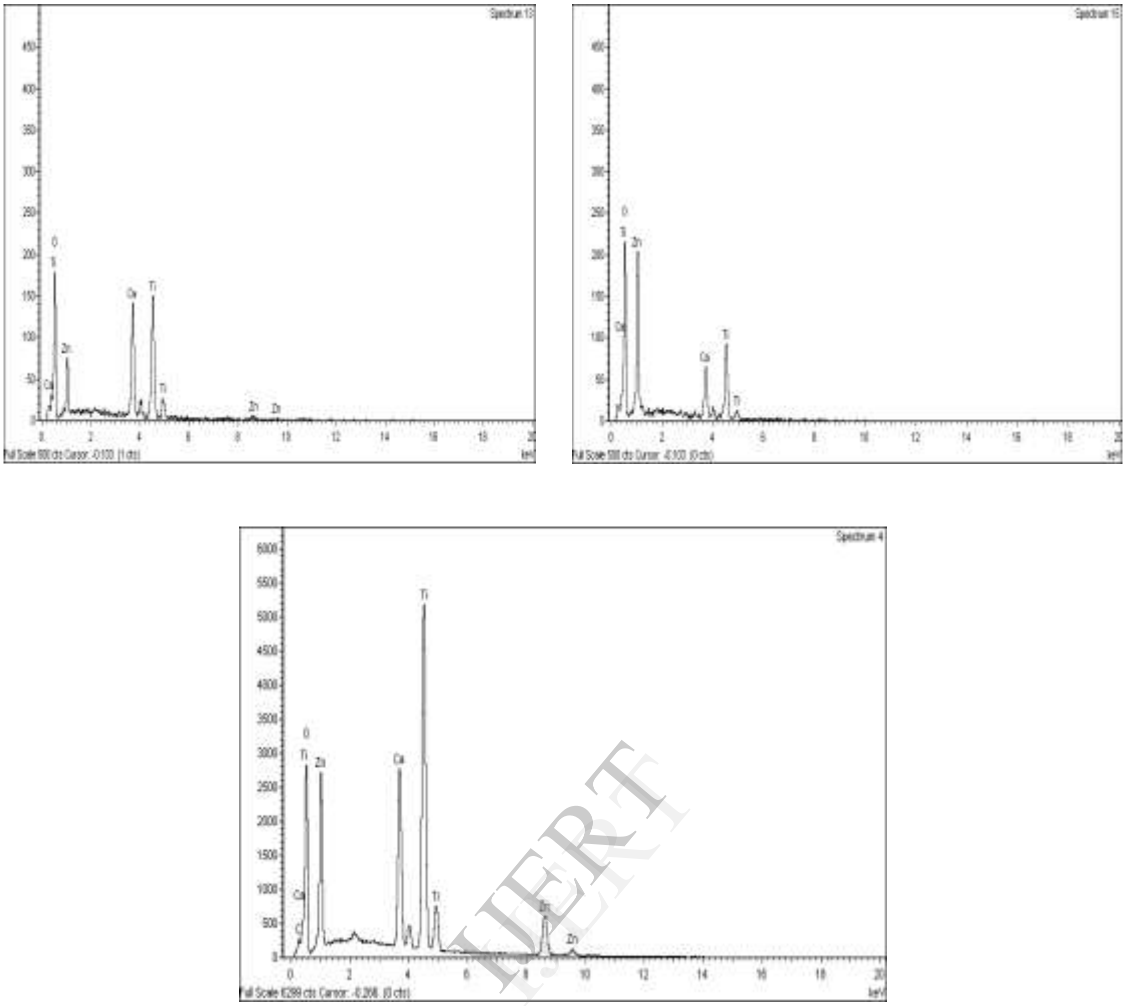

Fig.3 Shows the EDAX plots for $Zn_xCa_{1-x}TiO_3$ at x=0.1, 0.3 & 0.5

2.3 Dielectric measurements

2.3.1 Dielectric Constant:

Fig.4 shows the variation of dielectric constant (K) against the temperature (T) at five different frequencies of 100Hz, 1 KHz, 10 KHz, 1 MHz and 10 MHz. From these figures it is observed that the dielectric constant (K) decreases with the increase of temperature at lower composition of Zinc. As the Zinc composition increases the dielectric constant was increased with temperature and frequency. At x=0.5 the dielectric constant was increased with temperature up to ~650K then it will fall down because of the lagging of the dipoles present in it.

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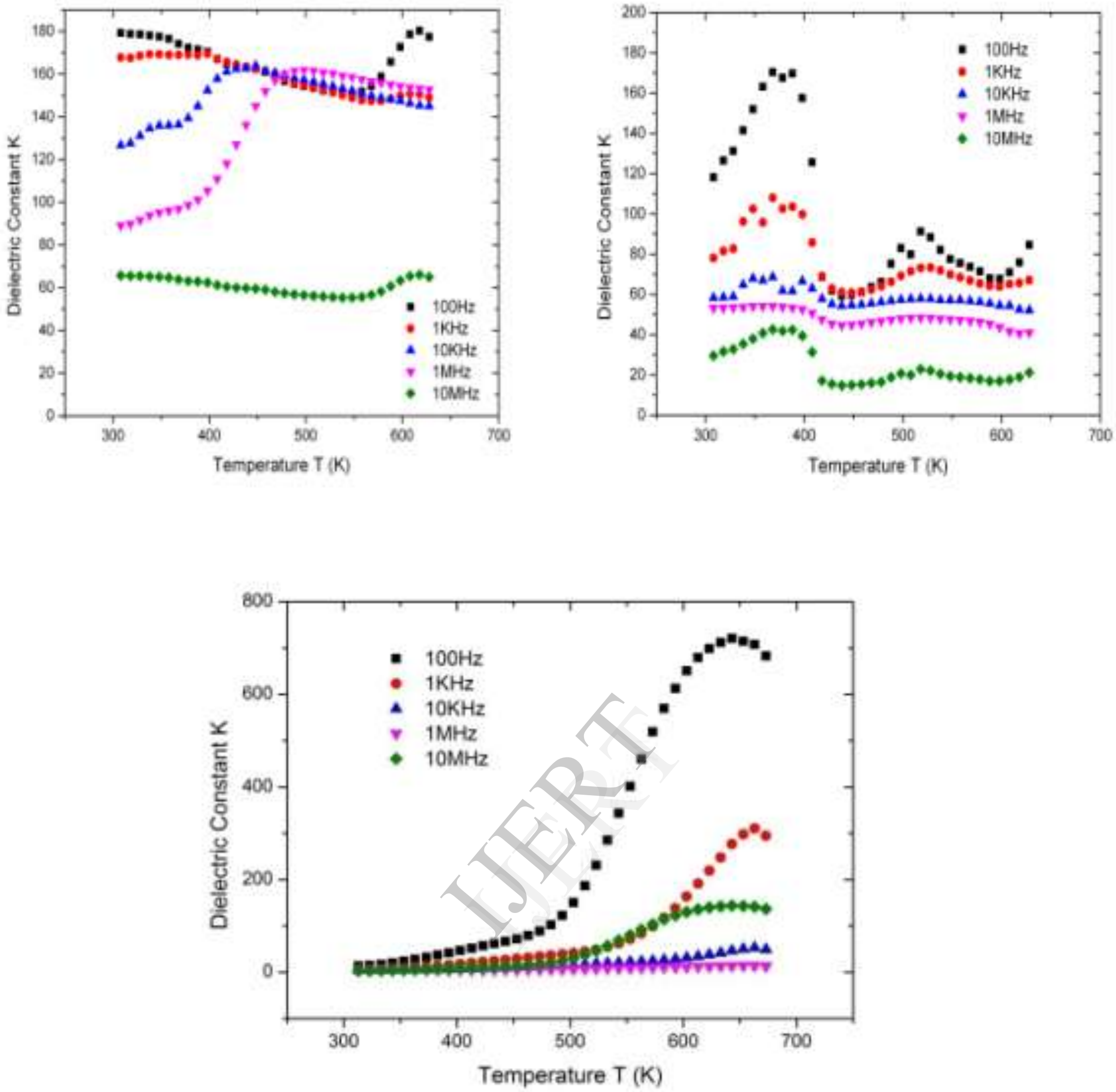

Fig.4 Shows the dielectric constant Vs temperature plots for $Zn_xCa_{1-x}TiO_3$ at x=0.1, 0.3 & 0.5

2.3.2 Dielectric Loss:

Fig. 5 shows the variation of dielectric loss with temperature at different frequencies at x=0.1, 0.3 and 0.5 of zinc composition. From the following plots, we observed that the dielectric loss was decreased with increase of temperature and frequency.

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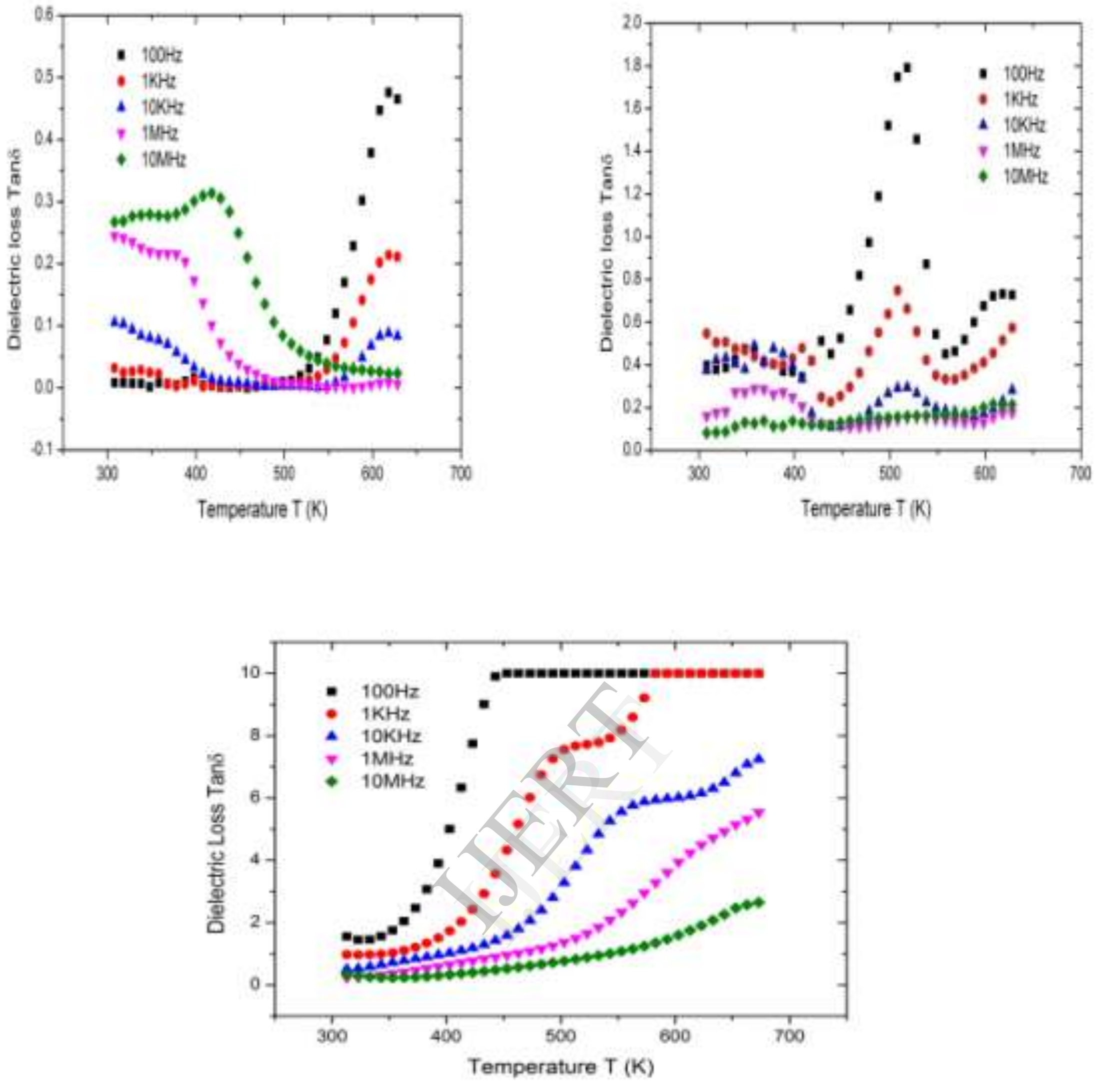

Fig.4 Shows the dielectric loss Vs temperature plots for $Zn_xCa_{1-x}TiO_3$ at x=0.1, 0.3 & 0.5

Conclusions:

A series of $Zn_xCa_{1-x}TiO_3$ (x=0.1, 0.3, 0.5) samples were prepared by using the solid state reaction technique with high purity powders and the structure of the sample were identified from the XRD plots as rhombohedral with space group R_{3}^{-} (148). The grain sizes of the sample were calculated as 0.25μm from SEM studies and the compound was well stoichiometric. The dielectric studies were carried out with HIOKI 3522-50 LCR Hi tester and observed that with the variation of the Zinc composition the dielectric constant and loss were studied with temperature and frequency. The dielectric constant increased with temperature and decreased with frequency. The dielectric loss were decreased with temperature and frequency. From this study it may also be predicted that Zn doping may improve dielectric properties and decrease sintering temperature.

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