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CHARACTERIZATION AND DIELECTRIC PROPERTIES OF ZINC TITANATE ($ZnTiO_3$) CERAMICS

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Abstract

In this study, zinc titanate ($ZnTiO_3$) was prepared by solid state reaction method using zinc oxide (ZnO) and titanium oxide (TiO_2) in a molar ratio of 1:1. Three calcination temperatures (800˚C, 900˚C, 1000˚C) at 2 h were selected to investigate the reaction of formation of zinc titanate ($ZnTiO_3$). Structural properties of $ZnTiO_3$ ceramics were characterized by X-ray Diffraction (XRD) technique. Scanning Electron Microscopy (SEM) technique was used to examine the surface morphology of ceramics. Frequency dependence of the dielectric properties of $ZnTiO_3$ was measured by LCR meter.

Keywords: $ZnTiO_3$ Ceramics, XRD, SEM, Dielectric Properties

Introduction

Ceramic materials are inorganic, non-inorganic materials made from compounds of a metal and non-metal. Ceramic materials may be crystalline or partly crystalline. They are formed by the acton of heat and subsequent cooling. Clay was one of the earliest materials used to produced ceramics, but many different ceramic materials are now used in domestic, industrial and building products. Ceramics can also be formed to serve as electrically conductive materials, objects allowing electricity to pass through their mass or insulators, materials preventing the flow of electricity. Electroceramics have numerous applications due to their specific structures and physical properties such as microelectronics or as individual circuit components, particulary as capacitors of sensor. These materials and their solid solutions are of a great interest for several applications to their easy synthesis by various methods (solid state reaction , sol-gel, hydrothermal or mechanochemical synthesis) and their properties (piezoelectricity, pyroelectricity, ferroelectricity). Due to their dielectric performances, they are mostly used in the electronic industry as capacitors, detectors, sensors, resonators, actuators, and memories.

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In the present work ZnTiO3 compound was prepared by solid state reaction method followed by calcimined at different temperatures 800C, 900C and 1000C for two hours each. The structure, microstructure and dielectric properties of sintered $ZnTiO_3$ have been investigated. $ZnTiO_3$ compound is an attractive dielectric ceramics owing to its interesting dielectric properties in high frequency range.

Experimental

The $ZnTiO_3$ compound was prepared by conventional solid state reaction method. The 99.99% pure ZnO and TiO_2 powders were weighed in a molar ratio 1:1 using digital balance as the stating materials. The stating materials were then grounded in an agate mortar for 24 h with the aid of ethanol to make homogeneous and fine powder. Finally the fine powder was calcined at different temperatures 800˚C, 900˚C and 1000˚C for 2h each in air chamber. The calcined powders at various temperatures were examined by X-ray diffractometry (XRD, Rigaku) using Cu Kα radiation to indentify the possible phases formed after heat treatment. The surface morphology was examined by scanning electron microscopy (SEM JEOL – JCM -6000 Plu). After examining the structural properties, the $ZnTiO_3$ compound powders at different temperatures were cold press into cylindrical pellet of size 2.4cm diameter and 0.5cm thickness using hydraulic press with a pressure of 5 ton. Saturated solution of polyvinyl alcohol (PVA) was used as a binder for pellets. These pellets were sintered at 500˚C for 2h each. The binder was burnt our during the sintering of the sample. The flat surfaces of pellets were be measured capacitance and resistance as a function of frequency (1kHz to 100kHz) using GW INSTEK LCR-8110G LCR Meter. The flow chart for preparation of $ZnTiO_3$ powders and ceramics were shown in figure (1).

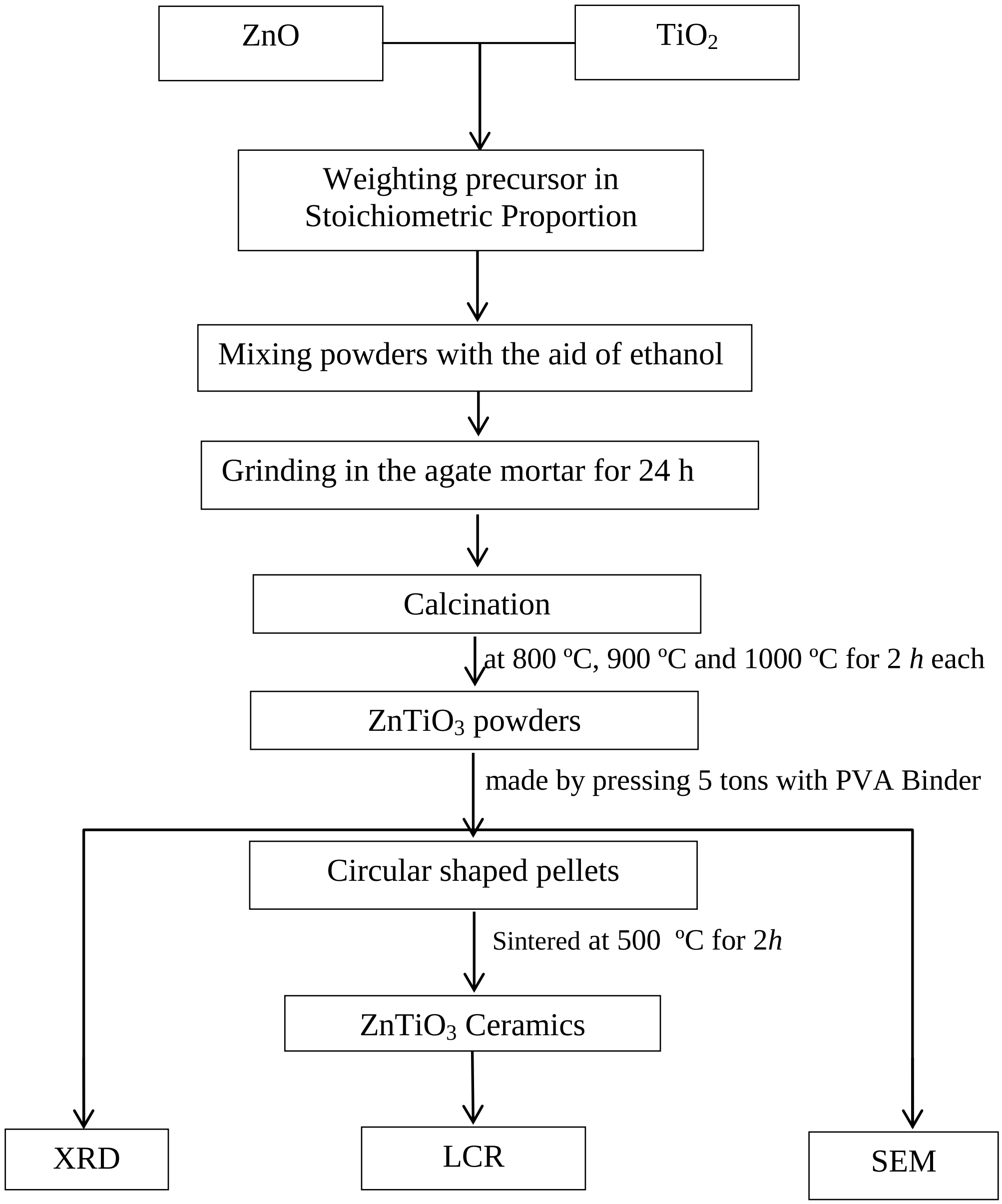

Figure 1: Block diagram of preparation of $ZnTiO_3$ powders and ceramics

Results and Discussion

The experimental results and discussion from XRD and SEM measurement of $ZnTiO_3$ powders prepared at different temperatures 800ºC, 900ºC and 1000ºC for 2 *h* each. The electrical properties of $ZnTiO_3$ ceramics were studied from the calculation of dielectric constant and resistivity.

Characterization of X–ray Diffraction

The X-ray diffractometry is mainly used for the identification and qualification of compounds by their diffraction patterns. The XRD patterns performed on the powder synthesized at various temperatures 800˚C, 900˚C and 1000˚C show the presence of $ZnTiO_3$ (cubic). The X-ray diffraction patterns of zinc titanate powder at different temperatures (800˚C, 900˚C and 1000˚C for 2*h)* were shown in figure 2(a-c). The XRD patterns of the powder showed (311), (220),(400), (422), (511) and (440) planes were produced. The diffraction angles, interplaner spacing, FWHM and crystallize size of (311) plane were listed in table (1).The crystallite sizes for all powder were calculated by Scherrer Formula,

$$D = \frac{0.899\lambda}{B\cos\theta}$$

where D is the crystalline size of the sample (nm), **λ** is the X-Ray wavelength (0.154056 nm), θ is the Bragg angle and B is observed FWHM (radian). As shown in XRD patterns, the diffraction line of (3 1 1) plane is found to the strongest in intensity (I=100%) and this plane (peak) is dominated on other peaks. XRD patterns revealed cubic phase of calcined zinc titanate powders. The obtained crystallite sizes can be taken as the nanosized crystalline materials due to the size lies between 1 nm –100 nm. The crystallite size of $ZnTiO_3$ powder is in nanometer range and apparently decreases with the increasing temperature.

Table 1: Data obtained by XRD analyses of $ZnTiO_3$ powders for (311) plane

Temperature (˚C)	Diffraction angle (deg)	Interplaner spacing (Å)	FWHM (deg)	Crystallite size (nm)
800˚C	35.341	2.538	0.191	45.61
900˚C	35.476	2.528	0.193	45.16
1000˚C	35.344	2.537	0.226	38.55

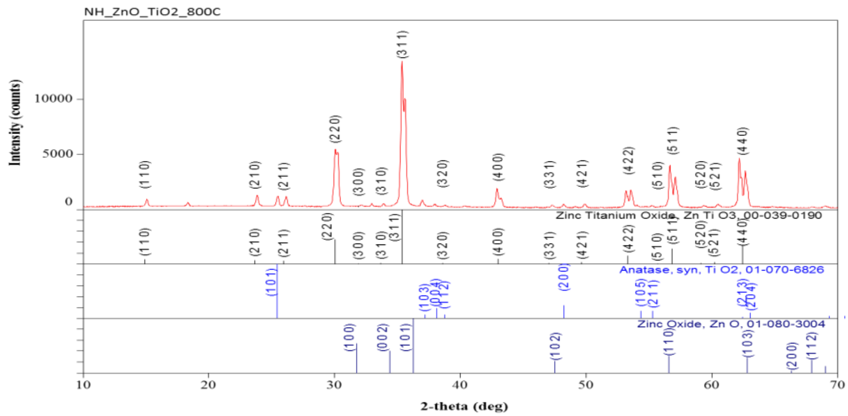

Figure 2: (a) XRD Pattern of $ZnTiO_3$powders prepared at 800˚C

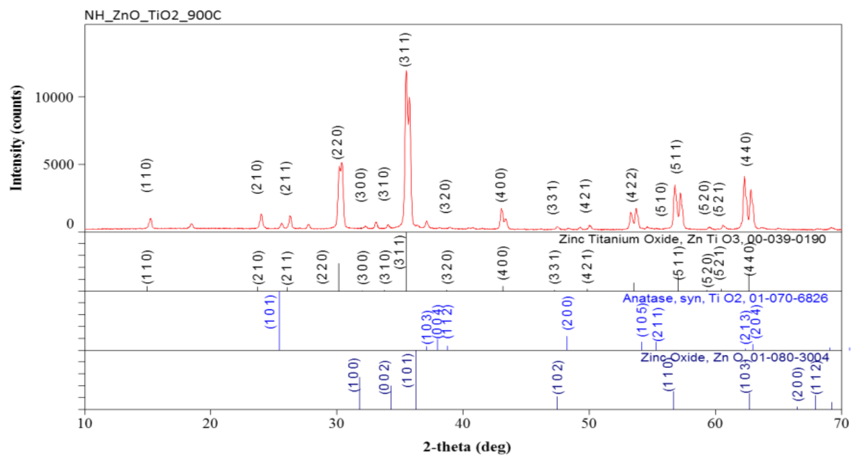

Figure 2: (b) XRD Pattern of $ZnTiO_3$ powders prepared at 900˚C

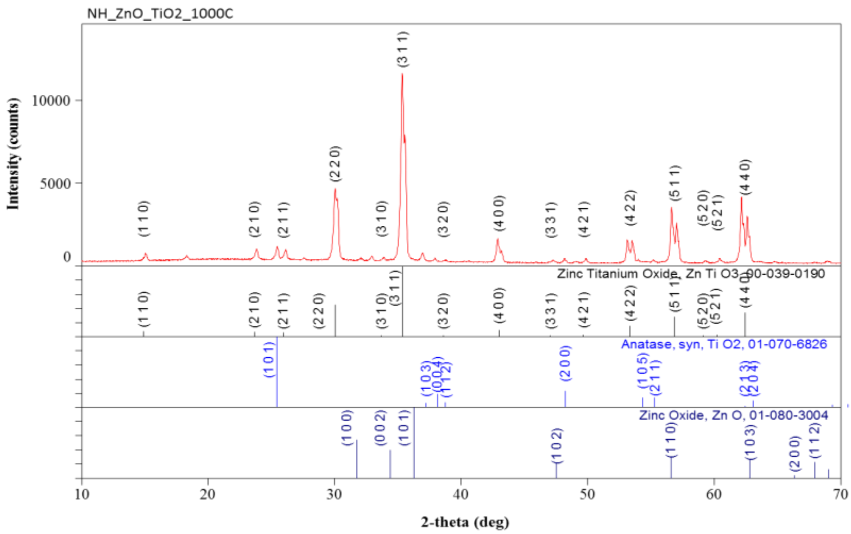

Figure 2: (c) XRD Pattern of $ZnTiO_3$powders prepared at 1000˚C

SEM Morphology of Zinc Titanate Powders

Morphology of zinc titanate powders was examined by scanning electron microscope. SEM image of the calcined $ZnTiO_3$ powders with various temperatures (800˚C , 900˚C and 1000˚C for 2 h) each are shown in Figure 3 (a-c).These images show nearly spherical with a very narrow distribution centered at around 0.3-0.4 µm. The variation of grain size with different annealing temperatures was shown in table (2).The sphere like particles seemed to distribute homogeneously and the particle size decreases with the increase in the calcination temperature.

Table 2: **Average grain size of $ZnTiO_3$ powders with different temperatures from SEM image**

Sr	Temperature (˚C)	Time (*h*)	Grain Size (µm)
1	800	2	0.44
2	900	2	0.40
3	1000	2	0.33

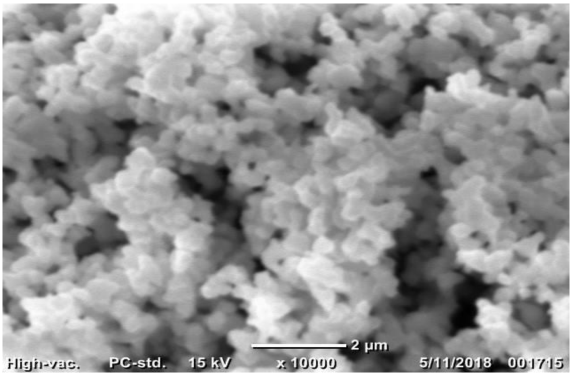

Figure 3: (a) SEM photograph of $ZnTiO_3$ powders prepared at 800˚C

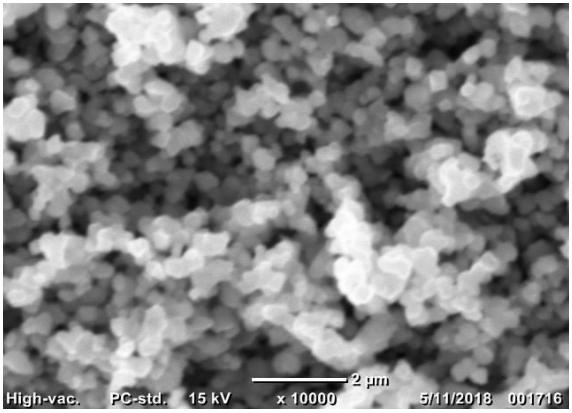

Figure 3: (b) SEM photograph of $ZnTiO_3$ powders prepared at 900˚C

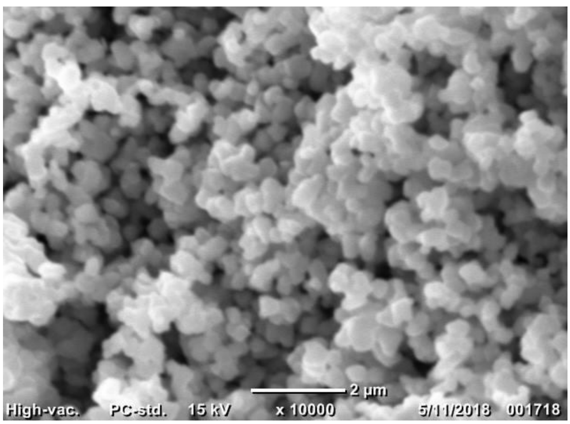

Figure 3: (c) SEM photograph of $ZnTiO_3$ powders prepared at 1000˚C

Dielectric Constant and Dissipation Factor of $ZnTiO_3$ Ceramics

Applied frequency dependence of resistance and capacitance value for $ZnTiO_3$ ceramics at various temperatures were measured by LCR meter. The higher dielectric constant makes these materials useful as capacitor and energy- storage devices materials. Variation of capacitance with respect to applied frequency for $ZnTiO_3$ ceramics at different temperatures (800˚C, 900˚C and 1000˚C) were shown in figure (4). .Decreasing of capacitance value was observed with respect to increasing of applied frequency. At the higher frequency region, saturated capacitance value was observed. The capacitance with the dielectric material is related to the dielectric constant ($\varepsilon_{r)}$ is showed in equation.

$$\varepsilon_r = \frac{Cd}{\varepsilon_0 A}$$

Where C is capacitance of a capacitor, d is thickness of sample, ε_0 is dielectric constant of vacuum ($8.854187818\times10^{-12} Fm^{-1}$), A is area and ε_r is dielectric constant of sample. The frequency variation of dielectric constant of $ZnTiO_3$ ceramics at different annealing temperatures are shown in figure (5). It was observed that dielectric constant decreases with increasing frequency in all temperatures. High dielectric value was observed at lower frequency (1 kHz). It is seen that all the parameter decrease with increasing frequency. The higher dielectric constant makes these materials useful as capacitor and energy storage devices materials.

The dissipation factor ($\tan\delta$) or DF is a measure of loss-rate of energy of a mode of oscillation (mechical, electrical, or electromechanical) in a dissipative system. DF will vary depending on the dielectric material and the frequency of the electrical signals. The frequency variation of dissipation factor of $ZnTiO_3$ ceramics at different annealing temperatures are shown in figure (6).

Electrical Resistivity of $ZnTiO_3$ Ceramics

Electrical resistivity is a fundamental property that quantifies how strongly a given material opposes the flow of electric current. The calculation of the resistivity was performed according to the following equation,

$$\rho = \frac{\pi d^2}{4}\frac{R}{L}$$

Where ρ *is* the resistivity, d is the diameter of the sample, R is the resistance and L is the sample thickness. The frequency variation of electrical resistivity of ZnTiO3 ceramics at different annealing temperatures are shown in figure (7). The variation of capacitance, dielectric constant, tanδ and resistivity values of of ZnTiO3 ceramics at different annealing temperatures are shown in Table (3).

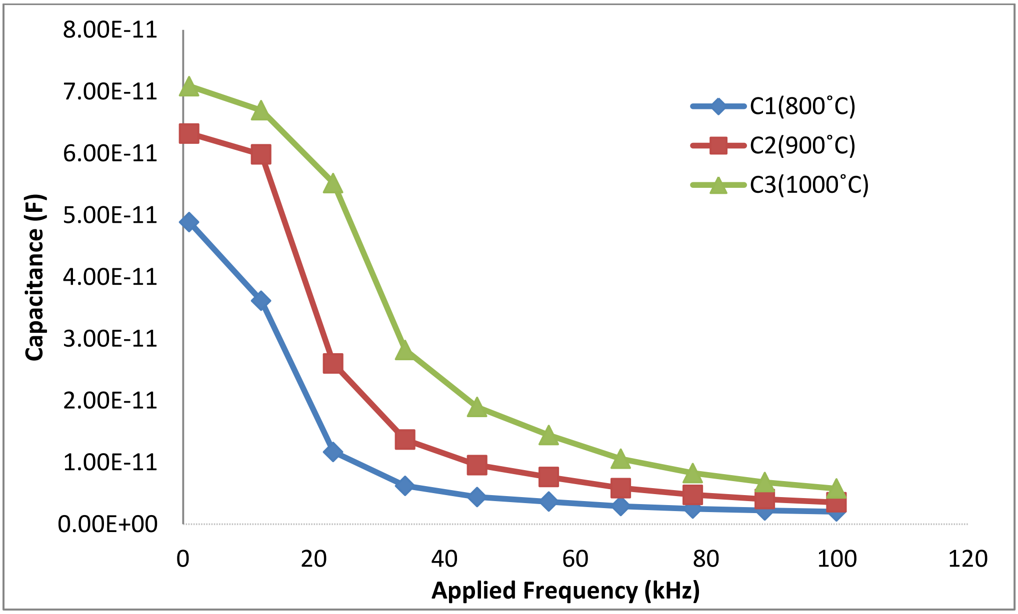

Figure 4: Frequency variation of capacitance of $ZnTiO_3$ ceramic with different temperatures

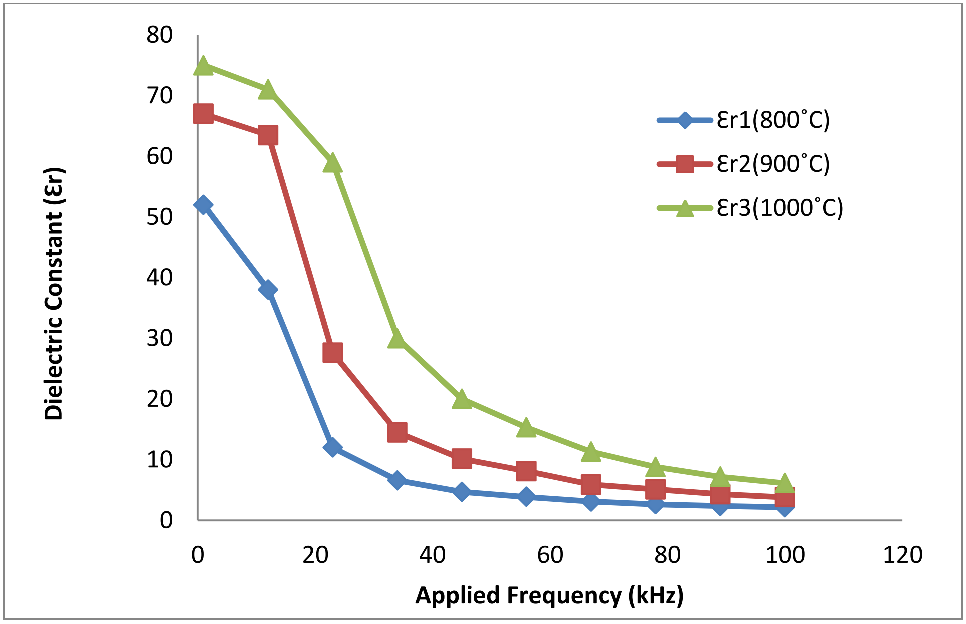

Figure 5: Frequency variation of dielectric constant of $ZnTiO_3$ ceramic with different temperatures

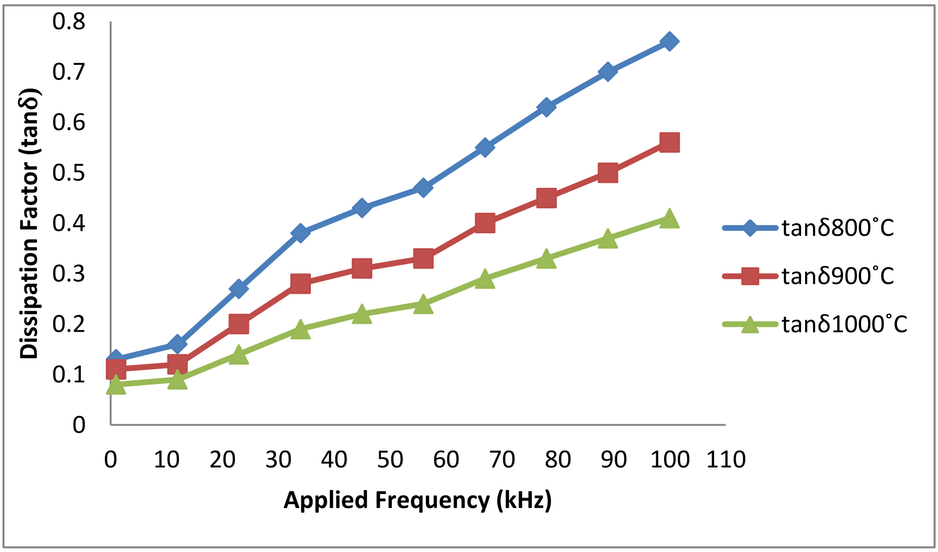

Figure 6: Frequency variation of dissipation factor of $ZnTiO_3$ ceramic with different temperatures

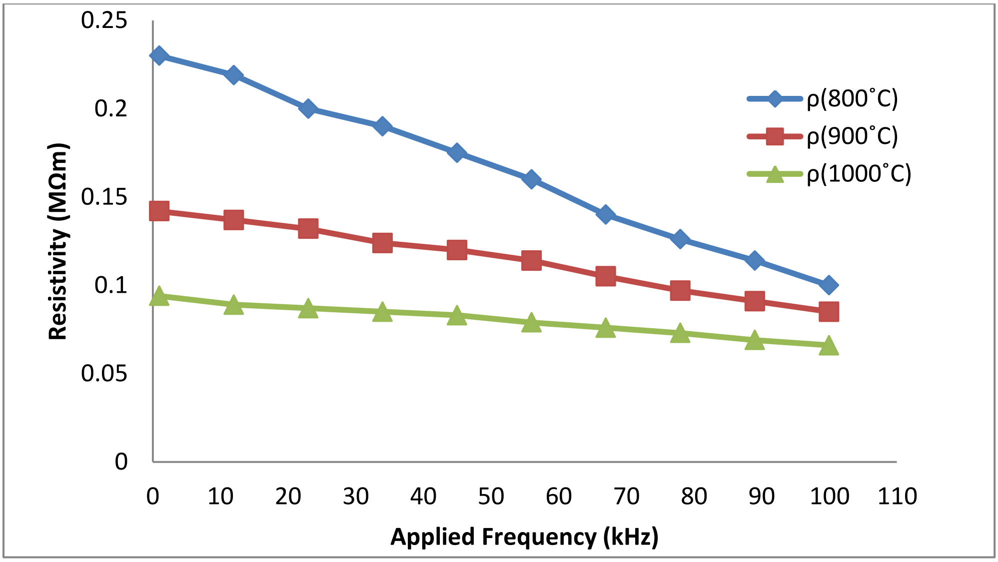

Figure 7: Frequency variation of resistivity of $ZnTiO_3$ ceramic with different temperatures

Table 3: The variation of capacitance, dielectric constant, dissipation factor and resistivity of $ZnTiO_3$ ceramics with different temperatures

Temperature (˚C)	f(kHz)	C(pF)	Dielectric constant (ε_r)	$\tan\delta$	Resistivity (ρ)
800˚C	1.00E+00	48.9	52	0.13	0.23
	1.20E+01	36.1	38	0.16	0.219
	2.30E+01	11.1	12	0.27	0.2
	3.40E+01	6.21	6.59	0.38	0.19
	4.50E+01	4.41	4.68	0.43	0.175
	5.60E+01	3.64	3.86	0.47	0.16
	6.70E+01	2.93	3.11	0.55	0.14
	7.80E+01	2.51	2.66	0.63	0.126
	8.90E+01	2.23	2.36	0.7	0.114
	1.00E+02	2.04	2.16	0.76	0.1
900˚C	1.00E+00	63.2	67	0.11	0.142
	1.20E+01	59.8	63.5	0.12	0.137
	2.30E+01	26.1	27.6	0.2	0.132

Temperature (˚C)	**f(kHz)**	**C(pF)**	**Dielectric constant (ε_r)**	**tanδ**	**Resistivity (ρ)**
	3.40E+01	13.7	14.5	0.28	0.124
	4.50E+01	9.56	10.15	0.31	0.12
	5.60E+01	7.63	8.1	0.33	0.114
	6.70E+01	5.84	5.9	0.4	0.105
	7.80E+01	4.76	5.1	0.45	0.097
	8.90E+01	4.05	4.3	0.5	0.091
	1.00E+02	3.55	3.8	0.56	0.085
1000˚C	1.00E+00	70.7	75	0.08	0.094
	1.20E+01	67.1	71	0.09	0.089
	2.30E+01	55.2	59	0.14	0.087
	3.40E+01	28.2	30	0.19	0.085
	4.50E+01	19.0	20	0.22	0.083
	5.60E+01	14.4	15.3	0.24	0.079
	6.70E+01	10.6	11.3	0.29	0.076
	7.80E+01	8.29	8.8	0.33	0.073
	8.90E+01	6.80	7.2	0.37	0.069
	1.00E+02	5.77	6.13	0.41	0.066

Conclusion

The crystallite size, micro structure and dielectric properties of $ZnTiO_3$ have been investigated. The solid state reaction method yields nano crystalline single phase $ZnTiO_3$ ceramics. The XRD pattern shows the formation of single phase cubic spinal structure for all the samples. The crystallite size of the samples decreases with the increase in temperature was observed in both XRD and SEM images. The decreasing trend of the dielectric constant and resistivity of $ZnTiO_3$ ceramics with increasing frequency are observed for all temperatures. These factors are normally taken into account when designing a capacitor for electronic application. Therefore $ZnTiO_3$ ceramic fabrication process and dielectric properties were quite suitable for ceramic capacitor.

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