Synthesis and Characterization of PbZrTiO$_3$ Ceramics

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ABSTRACT. Lead zirconate titanate (PZT) ceramic powders were prepared via conventional solid-state reaction method. The prepared samples were sintered at 900°C for 2 h. The sintered materials were characterized for structural analysis. The diffraction pattern reveals the pure phase formation of perovskite PZT structure. The morphology is analyzed by scanning electron microscope and average grain size is evaluated to be of 3.3 μm. The frequency and temperature dependence of electrical properties such as dielectric constant and dielectric loss was studied using LCR controller. In addition, the ferroelectric behavior was investigated by P-E loop tracer.

Introduction. Lead titanate is a ferroelectric ceramic material. It shows distinct applications in various fields and is used for non volatile memories, medical ultrasound imaging and actuators and data storage devices [1, 2]. The well populated applications of ferroelectric materials are observed in the fields of dielectrics for capacitor applications. In particular, these materials are candidates for ferroelectric thin film technology. The perovskite materials will have a general chemical formula of the type ABO$_3$ [2]. Many ferroelectric materials such as; barium titanate (BaTiO$_3$), lead titanate (PbTiO$_3$), lead zirconate titanate (PZT), lead lanthanum zirconate titanate (PLZT), have this perovskite type structure. These materials can work as good dielectric materials. In addition, the barium titanate, strontium titanate, strontium copper titanate, strontium lanthanum titanate, strontium lead titanate, strontium zinc manganese titanate, strontium magnesium titanate and strontium bismuth titanate are investigated recently for the dielectric and ferroelectric properties by several researchers [3-9]. In the current study, the structure, morphology, dielectric and ferroelectric behaviour are discussed for PZT ceramics.

Experimental Procedure. In this study the precursors are chosen as PbO, ZrO$_2$ (99.6% purity, Sigma Aldrich), TiO$_2$ (99.4% purity, Sigma Aldrich) to prepare the ferroelectric PZT ceramics. Initially, the raw materials are weighed and mixed uniformly according to their stoichiometric ratio. The mixed powder is ball milled for approximately 12 h using ball miller (Retsch PM200). Furthermore, the uniformly grounded powder is pre-sintered at 700°C for 12 hr. The pre-sintered powder is again grounded for nearly 2 hr. The pellets of radius 0.58 cm and thickness 0.282 cm are prepared after applying 2 ton pressure using hydraulic press. The pellets are sintered at 850°C for 2 hr in conventional furnaces. Further, the pellets are characterized using XRD at room temperature (Bruker X-Ray Powder Diffract Meter, CuKα = 0.15418 nm), SEM (Hitachi: S-4700), LCR controller (Hioki 3532-50) and P-E loop measurement (Marine India) for structural, surface morphological, dielectric and ferroelectric properties respectively.

Results and Discussions.
Structural Analysis. The diffraction pattern of PZT ceramic powder is shown in Fig. 1. It can be understood from figure that the formed single crystalline phases that belong to cubic perovskite structure of pure lead zirconate titanate. The maximum intense plane is noticed at the diffraction or two-theta angle of 32.254°. The value of lattice parameters 'c' of PZT ceramic composition were found to be 4.131 Å where as “a = b” is 4.088 Å conforming the tetragonal perovskite structure. Furthermore, the average crystalline size (D) is calculated as 41.2 nm using the Scherer formula [10-13].

![Fig. 1. PZT perovskite structure.](image)

Surface Morphology. The Scanning Electron Microscope (SEM) provides the surface morphology of powder specimen. It is seen from Fig. 2 that all the grains are of almost spherical in shape. The average grain size (Gₐ) is found to be 3.3 µm using linear intercept method using the following equation [14].

\[ Gₐ = \frac{1.5L}{MN} \]  

(1)

where \( L \) is the test line length, \( N \) is the number of intersecting grains and \( M \) is the magnification.

SEM image of the PZT sample prepared is shown in Fig. 2. It contains well defined grains of spherical in shape. Moreover, an apparent porosity is observed to be very small.

![Fig. 2. SEM image of PZT.](image)
Dielectric Properties. The variation of dielectric constant ($\varepsilon'$) and loss ($\varepsilon''$) of PbZrTiO$_3$ is shown in Fig. 3 and Fig. 4 respectively as a function of both frequency (100 Hz-1 MHz) and temperature (300-573 K). It is understood from the figures that the dielectric constant and loss were slowly increasing with increase of temperature up to 573 K and further a sharp increasing trend in both the cases was observed. The sharp increase is attributed to the interfacial or space-charge polarization effect. Similar trend is observed in the literature [15-18]. In addition, $\varepsilon'$ and $\varepsilon''$ were decreasing with increase of frequency. This was happened due to in effective space-charges at the grain boundary interface. At room temperature for frequency ~ 1 MHz the present specimen showed dielectric constant of ~15. The loss was also showing the similar trend as that of permittivity in all respects. But interestingly, loss versus temperature plot shows dielectric relaxations with increase of temperature. The relaxation is shifted towards the right. These relaxations are generally formed due to the presence of oxygen vacancies with temperature. The high loss of 14 is attributed at 1 MHz frequency. This kind of high $\varepsilon'$ and high $\varepsilon''$ values noticed at room temperature were most suitable for filter, charge stored capacitors and absorber applications.

Ferroelectric properties. The ferroelectric behavior of PZT is investigated with the help of P-E loop tracer. While doing measurement the pellet is connected parallel to 2 µF capacitor for compensation. Fig. 5 depicts the ferroelectric hysteresis loops of PZT under an applied frequency of 600 Hz at an operating voltage of 600 V. It is understood from Fig.5 that the sample shows a well-behaved hysteresis loop distorted into ‘banana’ shape performed at distinct temperatures such as 303K. The response of dipoles per unit field is in general regarded as polarization. It is an observed fact that the saturation polarization ($P_s$) and remanance polarization ($P_r$) of PZT at all temperatures is found to be constant value of $\approx$ 21.33 µC/cm$^2$ and coactivity field ($E_c$) is 0.79 Kv/Cm.
Summary. Lead zirconate titanate (PZT) ceramic powders were prepared via conventional solid-state reaction method. The diffraction pattern reveals the pure phase formation of perovskite PZT structure. The average crystallite diameter is of 41.2 nm. The average grain size is evaluated to be of 3.3 micrometer. The high dielectric constant and high dielectric loss was observed for dielectric absorbers applications. The P-E loop analysis showed the highest $P_s$ of 21.3 µC/Cm$^2$ coactivity field (Ec) is 0.79 Kv/Cm.

References


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