Research Article

Structural and Electromechanical Characterization of Sol-Gel-Derived Lanthanum-Modified Lead Zirconate Titanate (PLZT) Ceramic

Anshu Sharma
Department of Physics, DAV College, Bulandshahr, Uttar Pradesh, India

Corresponding Author: *Anshu Sharma

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Abstract

In this study, lanthanum-substituted lead zirconate titanate (PLZT) ceramics with the general formula $Pb_{1-x}La_x (Zr_{1-y}Ti_y)_{1-x/4}O_3$ were prepared through a modified sol–gel route and densified by controlled sintering to yield dense ferroelectric bodies. The work systematically examines how progressive lanthanum addition affects the structural, microstructural, dielectric and electromechanical response of the resulting ceramics. X-ray diffraction (XRD) verified that a well-crystallised, single-phase perovskite had formed without any traceable secondary phase, while scanning electron microscopy (SEM) revealed a compact, uniform microstructure with refined grains. Density and porosity data confirmed that sintering produced effective densification. The dielectric study showed that raising the lanthanum level modified the ferroelectric transition, lowering the Curie point while increasing the permittivity in the vicinity of the morphotropic phase boundary (MPB). Piezoelectric testing recorded a marked rise in both the charge coefficient (d_{33}) and the planar coupling factor (k_p) at optimum lanthanum contents. These gains are linked to greater domain mobility, reduced lattice distortion and a finer microstructure introduced by lanthanum substitution. Overall, the findings establish sol–gel-derived PLZT as a strong candidate for advanced piezoelectric sensors, actuators, ultrasonic transducers, energy-harvesting units and related smart-electronic devices.

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1. INTRODUCTION

For several decades, lead zirconate titanate (PZT) ceramics have remained a focus of intense research owing to their remarkable ferroelectric, dielectric and piezoelectric behaviour. Because they interconvert electrical and mechanical energy so efficiently, such materials are widely used in sensors, actuators, ultrasonic transducers, fine-positioning stages and energy-harvesting units. Much of this superior behaviour stems from the way several crystallographic phases coexist close to the morphotropic phase boundary (MPB), a region in which polarization can be switched comparatively easily by an external field and the functional response is correspondingly amplified.

Within the broad family of modified PZT compositions, lanthanum-doped lead zirconate titanate (PLZT) stands out as one of the most industrially significant ferroelectric ceramics. Here, trivalent La^{3+} ions occupy a fraction of the A-sites normally held by lead in the perovskite framework. To offset the resulting charge imbalance, cation vacancies are generated; these alter domain-wall motion, lower the coercive field and strongly modify the dielectric and piezoelectric characteristics. For certain compositions the same substitution also raises optical transparency, so that PLZT serves electro-optic and photonic roles alongside its conventional piezoelectric functions.

The way the material is processed largely dictates the microstructure and, ultimately, the performance of PLZT ceramics. Conventional solid-state routes typically demand long calcination at high temperature and frequently lead to compositional non-uniformity, exaggerated grain growth and loss of lead through volatilisation. The sol–gel approach, by contrast, mixes the precursors at the molecular scale, giving better chemical uniformity, lower processing temperatures, a finer and narrower particle-size range and tighter control of stoichiometry. For these reasons sol–gel processing offers a practical means of obtaining dense PLZT with improved structural regularity and enhanced electrical behaviour.

Lanthanum content, the Zr:Ti ratio and the sintering schedule together exert a strong influence on how PLZT develops structurally. Adjusting any of these variables changes the crystal symmetry, grain growth, domain arrangement, dielectric response and electromechanical coupling. A thorough characterisation of both structure and electrical properties is therefore necessary before the links between composition, microstructure and functional behaviour can be properly understood.

In the present work, PLZT ceramics of differing lanthanum content were prepared by a modified sol–gel process followed by controlled sintering. Phase formation and crystal structure of the resulting samples were probed by X-ray diffraction (XRD), whereas grain shape and microstructural detail were examined under scanning electron microscopy (SEM). Density, dielectric response, Curie temperature, the piezoelectric charge coefficient (d_{33}) and the planar coupling factor (k_p) were also measured to gauge how far lanthanum substitution governs the overall behaviour of the ceramics.

The central aim of the study is to draw a clear link between processing conditions, structural features, dielectric response and electromechanical properties in sol–gel-derived PLZT. The results are intended to guide the tuning of PLZT compositions for high-performance piezoelectric sensors, actuators, medical ultrasonic components and emerging smart-electronic systems.

2. OBJECTIVES OF THE STUDY

This work set out to explore how partial lanthanum substitution alters the structural, dielectric and electromechanical response of lead zirconate titanate ceramics produced by the modified sol–gel route. Its specific aims were as follows:

1. To prepare lanthanum-modified lead zirconate titanate (PLZT) ceramics of several lanthanum concentrations by a modified sol–gel processing route.
2. To obtain well-densified ceramic pellets through carefully controlled calcination and sintering.
3. To examine the crystalline phases and structural features of the ceramics by X-ray diffraction (XRD).
4. To assess grain shape, microstructure and grain-size distribution using scanning electron microscopy (SEM).
5. To measure the bulk density, theoretical density and porosity of the sintered pellets and relate them to the processing conditions.
6. To characterise the dielectric behaviour dielectric constant, loss and Curie temperature across a wide range of temperature and frequency.
7. To determine the piezoelectric response through the charge coefficient (d_{33}) and the planar electromechanical coupling factor (k_p).
8. To correlate lanthanum content with crystal structure, microstructure and the resulting functional properties.
9. To judge the suitability of the prepared PLZT ceramics for applications such as piezoelectric sensors, actuators, ultrasonic transducers, MEMS devices and energy harvesters.

3. MATERIALS AND METHODS

3.1 MATERIALS

PLZT ceramics of varying lanthanum content were produced by a modified sol–gel method. To keep the stoichiometry accurate and limit contamination, high-purity analytical-grade reagents were selected. The starting materials employed were:

- Lead(II) acetate trihydrate, $Pb(CH_3COO)_2 \cdot 3H_2O$
- Lanthanum acetate hydrate, $La(CH_3COO)_3 \cdot H_2O$
- Zirconium (IV) propoxide, $Zr(OC_3H_7)_4$
- Titanium (IV) propoxide, $Ti(OC_3H_7)_4$
- n-Propyl alcohol (solvent)
- Acetylacetone (chelating and stabilizing agent)
- Distilled water for hydrolysis
- Polyvinyl alcohol (PVA) as a temporary binder during pellet fabrication

No further purification of the reagents was carried out. Analytical-grade chemicals were used throughout the experimental work to preserve compositional accuracy and ensure reproducible results.

3.2 Synthesis of PLZT Ceramics by Modified Sol-Gel Method

A modified sol–gel procedure was followed to obtain PLZT powders of high chemical uniformity and fine particle size. To begin with, weighed amounts of lead acetate, lanthanum acetate, zirconium propoxide and titanium propoxide corresponding to the target stoichiometry were dissolved in n-propyl alcohol under continuous stirring. Acetylacetone was then introduced to chelate the metal alkoxides and to suppress premature hydrolysis.

The mixture was held just below its boiling point for roughly two hours so that complexation among the precursors could proceed. Alkoxide complexes developed during this stage, accompanied by propyl acetate as a by-product; this volatile ester was afterwards stripped off by controlled distillation.

Once the solution had cooled, distilled water was added gradually with constant stirring to trigger hydrolysis and condensation. Through these reactions the clear solution was converted first into a uniform colloidal sol and then into a stable gel.

The gel was dried under controlled conditions to drive off residual solvent and moisture, after which it was calcined at 873 K. Calcination removed the organic species and left fine crystalline PLZT powder of nanometric particle size.

To break up agglomerates, the calcined powder was ground thoroughly and then sintered at 1473 K for 2 hours to encourage densification and growth of the perovskite phase. The sintered material was subsequently ball-milled together with under 0.5 wt.% polyvinyl alcohol (PVA) to raise green strength during compaction.

Circular pellets about 10 mm in diameter and 0.5 mm thick were shaped by uniaxial pressing. These green compacts underwent a final sintering step at 1100°C under optimised conditions, yielding dense PLZT specimens ready for structural, dielectric and electromechanical study.

The synthesis sequence adopted in this work is set out schematically in Figure 3.1.

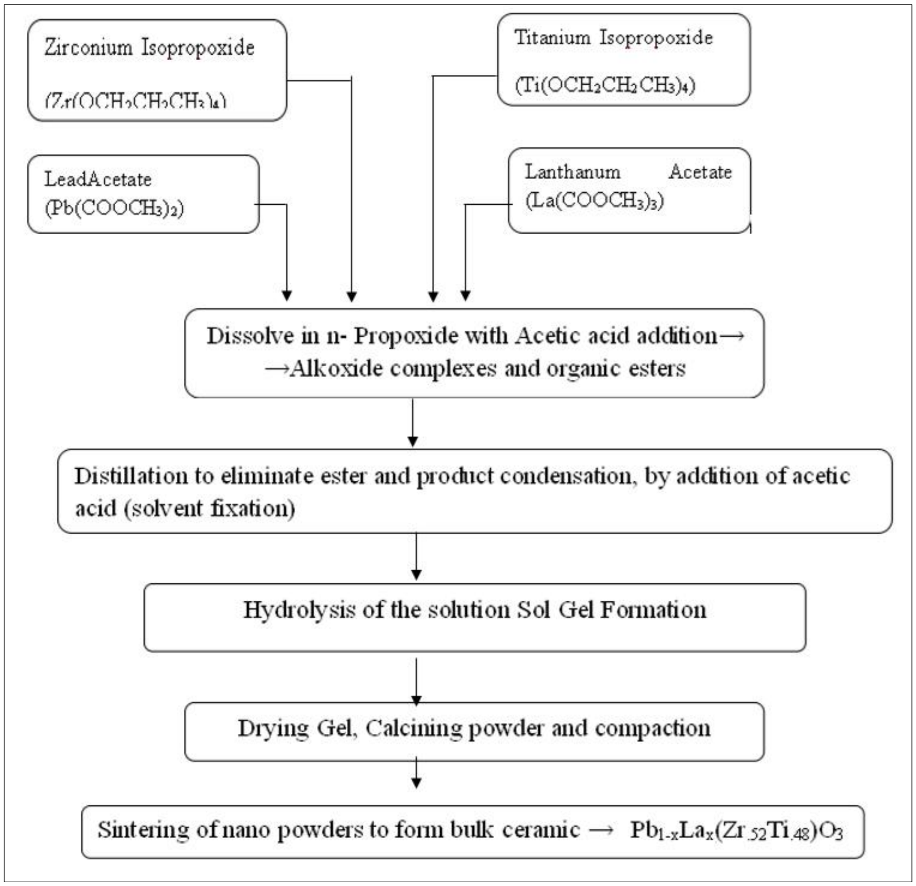

Fig 3.1: Preparation of PLZT Powders by modified Sol Gel Method

3.3 Composition of PLZT Samples

A total of eleven PLZT compositions were prepared, all sharing a fixed zirconium-to-titanium ratio matching the morphotropic phase boundary (Zr/Ti = 52/48). The lanthanum level was raised in steps of 2 mol%, from 0 up to 20 mol%.

The compositions studied are listed below:

Sample	Composition
PLZT-0	$Pb_{1.00}(Zr_{0.52}Ti_{0.48})O_3$
PLZT-2	$Pb_{0.98}La_{0.02}(Zr_{0.52}Ti_{0.48})O_3$
PLZT-4	$Pb_{0.96}La_{0.04}(Zr_{0.52}Ti_{0.48})O_3$
PLZT-6	$Pb_{0.94}La_{0.06}(Zr_{0.52}Ti_{0.48})O_3$
PLZT-8	$Pb_{0.92}La_{0.08}(Zr_{0.52}Ti_{0.48})O_3$
PLZT-10	$Pb_{0.90}La_{0.10}(Zr_{0.52}Ti_{0.48})O_3$
PLZT-12	$Pb_{0.88}La_{0.12}(Zr_{0.52}Ti_{0.48})O_3$
PLZT-14	$Pb_{0.86}La_{0.14}(Zr_{0.52}Ti_{0.48})O_3$
PLZT-16	$Pb_{0.84}La_{0.16}(Zr_{0.52}Ti_{0.48})O_3$
PLZT-18	$Pb_{0.82}La_{0.18}(Zr_{0.52}Ti_{0.48})O_3$
PLZT-20	$Pb_{0.80}La_{0.20}(Zr_{0.52}Ti_{0.48})O_3$

3.4 Preparation of Samples for Characterization

The way specimens were prepared depended on the measurement to be performed.

Pellets intended for X-ray diffraction were simply polished to flat, parallel faces; neither electroding nor poling was required.

For dielectric and other electrical measurements, both faces of the pellets were covered with conductive silver electrodes to give dependable contact.

Specimens for SEM were first polished, then cleaned ultrasonically to remove surface contaminants, and finally given a thin conductive gold coating before imaging.

Samples reserved for piezoelectric measurement were polished, painted with silver paste and heat-treated so that the electrodes adhered firmly. After curing, electrical leads were soldered to the electrodes, enabling reliable poling and electromechanical testing.

3.5 Density and Porosity Measurements

The degree of densification in the PLZT ceramics was judged from the bulk density, obtained by the Archimedes immersion method with distilled water as the immersion liquid. Before weighing, each pellet was polished and cleaned so that surface contamination would not distort the readings.

For every composition the theoretical density was computed from the lattice parameters extracted by XRD. Relative density then followed from the ratio of the measured bulk density to this theoretical value, as expressed below:

$$\text{Relative Density (\%)} = \frac{\text{Bulk Density}}{\text{Theoretical Density}} \times 100$$

Relative Density (%) = (Bulk Density / Theoretical Density) × 100

Open porosity of the sintered bodies was inferred from the density data. Together, the density and porosity results shed light on how effective the sintering had been and on the way lanthanum substitution affected densification. Figure 3.2 shows the variation of bulk density with lanthanum content.

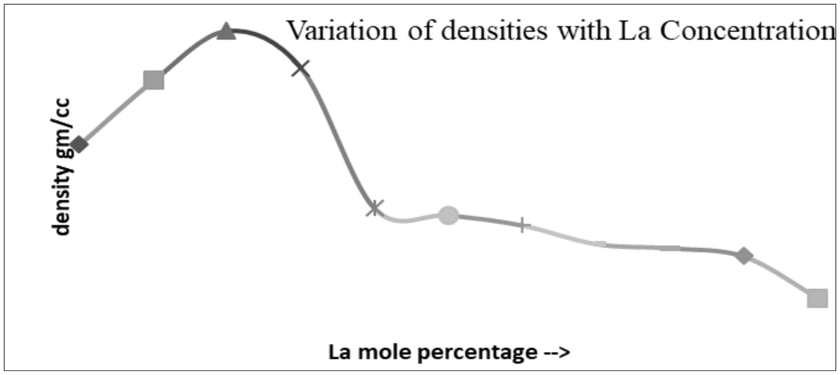

Fig 3.2: Plot of La mole percent in PLZT versus its density in gm/cc

3.6 Dielectric Characterization

Dielectric measurements on the PLZT ceramics were carried out over wide temperature and frequency ranges so as to trace how lanthanum substitution influences the ferroelectric behaviour.

Before testing, both faces of each pellet were coated with conductive silver electrodes and heat-treated for stable contact. A precision LCR meter was then used to record the dielectric constant (εr) together with the loss tangent (tan δ).

Data were collected from room temperature up to about 500 °C at three frequencies 1 kHz, 10 kHz and 100 kHz with the specimens heated at a controlled rate so that thermal equilibrium was maintained during acquisition.

The Curie temperature (Tc) of each composition was taken as the temperature at which the permittivity reached its maximum. The way the dielectric constant, loss and transition behaviour shifted with rising lanthanum content was then analysed to reveal how the compositional change affects ferroelectric performance. Representative dielectric spectra of the PLZT ceramics are shown in Figure 3.3.

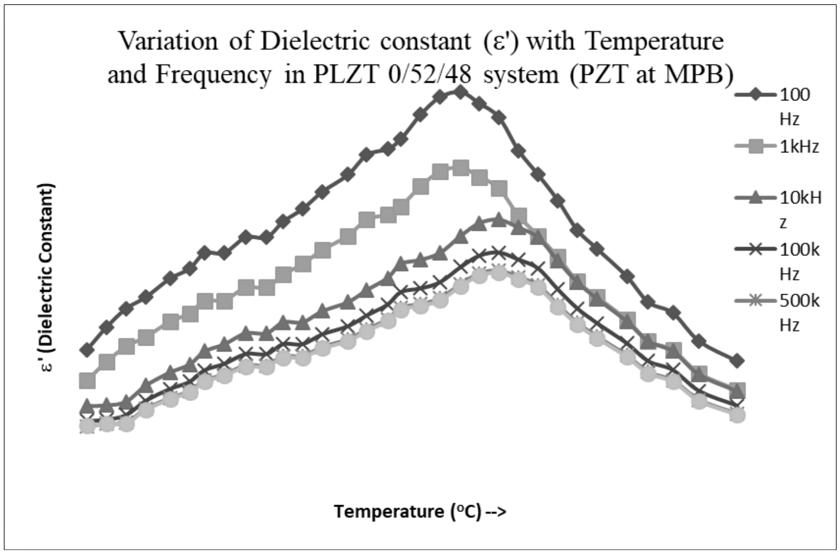

Fig 3.3: Plot of dielectric constant ε' as a function of temperature at different frequencies for PLZT 0/52/48 (PZT at MPB) ceramic systems

3.7 Electrical Poling and Electromechanical Characterization

Before electromechanical testing, the sintered pellets were electrically poled to switch on their piezoelectric response. Each sample was immersed in insulating silicone oil and exposed to a direct-current field of 2–4 kV mm^{-1} while held at roughly 100–120°C.

This field was maintained for 30–60 minutes so that the ferroelectric domains could align with its direction. Once poling was complete, the samples were cooled to room temperature with the field still applied, thereby locking in the domain alignment.

A Berlincourt-type d_{33} meter operating at room temperature was used to record the piezoelectric charge coefficient (d_{33}). The planar coupling factor (k_p) was extracted by the resonance–antiresonance method using an impedance analyser that scanned 50–200 kHz.

Further electromechanical quantities — the mechanical quality factor (Qm) and the elastic compliance — were derived from the resonance response of the poled pellets. To secure reliable, repeatable data, every measurement was carried out three times and reported as a mean value with its standard deviation.

A schematic view of the poling arrangement is given in Figure 3.4.

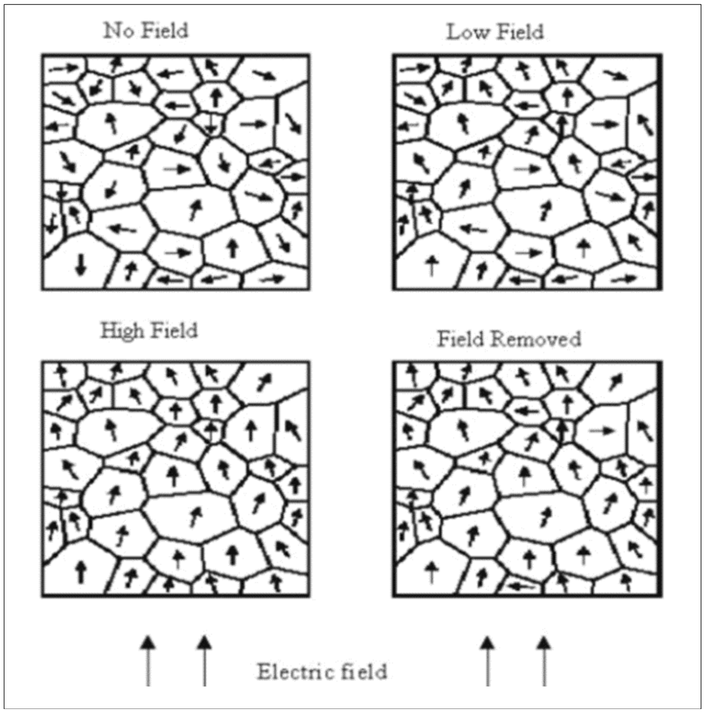

Fig 3.4: Concept of poling

4. RESULTS AND DISCUSSION

4.1 Structural Analysis

Phase formation and crystal structure were investigated by X-ray diffraction. For every composition the patterns confirmed that a crystalline perovskite phase had formed with no detectable impurity phase, showing that the modified sol–gel route yielded chemically uniform, phase-pure ceramics.

The reflections matched the characteristic PLZT peaks closely, indicating that lanthanum had entered the perovskite lattice without disturbing its basic structure. As lanthanum content increased, the peaks shifted slightly, pointing to small changes in the lattice parameters that occur when the larger Pb^{2+} ions are replaced by the smaller La^{3+}. Such distortion in turn alters the ferroelectric domain configuration and helps account for the observed changes in electrical response.

The diffraction data also pointed to structural change close to the morphotropic phase boundary, where tetragonal and rhombohedral phases occur together. This dual-phase region is widely recognised to boost dielectric and piezoelectric behaviour because polarization can rotate more freely under an applied field.

Figure 4.1 presents a representative pattern for the PLZT 0/52/48 composition, whose sharp peaks attest to good crystallinity and successful phase formation.

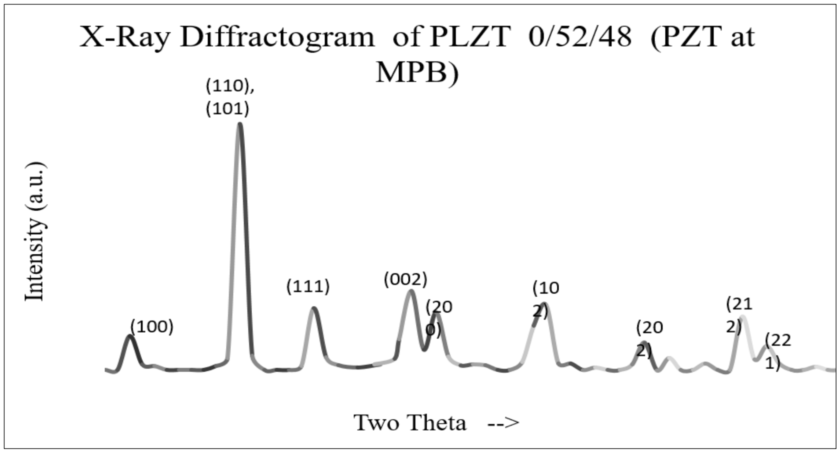

Fig 4.1: X-ray diffractogram of PLZT 0/52/48 (PZT at MPB)

4.2 Microstructural Analysis

Microstructure was studied by scanning electron microscopy (SEM). The micrographs revealed that the modified sol–gel route produced dense ceramic bodies with evenly distributed grains and comparatively little porosity.

Grain morphology proved highly sensitive to lanthanum content. Compared with undoped PZT, samples containing moderate lanthanum levels displayed finer, more uniform grains. This refinement is ascribed to lanthanum acting to inhibit grain growth during high-temperature sintering.

An even distribution of grains aids mechanical stability and eases the motion of ferroelectric domains under electrical loading. When the lanthanum content became too high, however, grain growth was suppressed still further and isolated pores occasionally appeared owing to incomplete densification.

Depending on composition and processing, the measured grain sizes ranged from below one micrometre to a few micrometres. Figure 4.2 illustrates how the average grain size varies with lanthanum content.

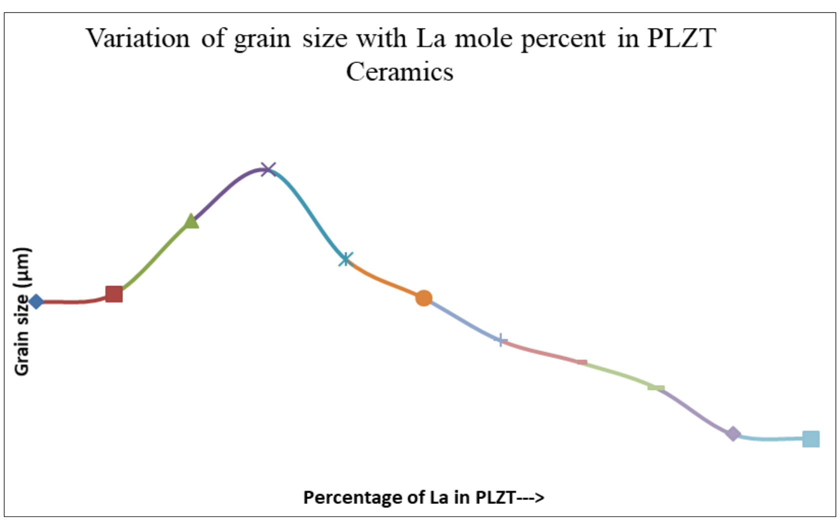

Fig 4.2: Dependence of grain size on La concentration in PLZT x/52/48 systems.

4.3 Dielectric Properties

The dielectric behaviour of the PLZT ceramics was studied in detail to gauge the effect of lanthanum incorporation on their ferroelectric character. Permittivity was recorded against temperature at several frequencies so that the phase-transition features and Curie temperature of each composition could be identified.

Every composition displayed a clear dielectric anomaly linked to the ferroelectric-to-paraelectric transition. The permittivity climbed steadily with temperature, peaked at the Curie temperature (Tc), and then fell as the material entered the paraelectric state behaviour typical of ferroelectric perovskites and confirming that ferroelectric order survives lanthanum substitution.

The Curie temperature dropped steadily as lanthanum content rose. This downward shift can be traced to changes in the crystal lattice and to the greater number of A-site vacancies formed during charge compensation. Because these changes lower the energy needed to reorient domains, polarization switching takes place at comparatively lower temperatures.

Permittivity increased markedly for compositions near the morphotropic phase boundary. In this region the coexistence of tetragonal and rhombohedral phases promotes polarization rotation and domain-wall mobility, which in turn raises the dielectric performance.

With increasing frequency the dielectric constant fell slightly. This trend reflects the inability of space-charge and dipolar polarization mechanisms to keep pace with a rapidly alternating field. Even so, the loss remained relatively low across the whole frequency range studied, signalling good insulating quality and little energy dissipation.

Taken together, the dielectric results show that carefully controlled lanthanum substitution improves the dielectric performance of PLZT while preserving good thermal stability. Figure 4.3 plots the corresponding variation of dielectric constant with temperature at the different frequencies.

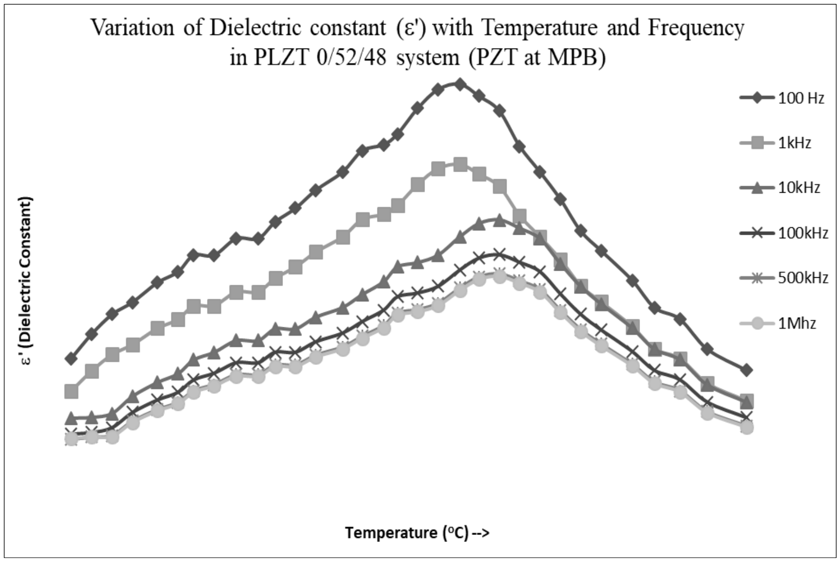

Fig 4.3: Plot of dielectric constant ε' as a function of temperature at different frequencies for PLZT 0/52/48 (PZT at MPB) ceramic systems

4.4 Electromechanical Properties

After poling, the electromechanical performance of the PLZT ceramics was evaluated through the piezoelectric charge coefficient (d_{33}), the planar coupling factor (k_p) and the mechanical quality factor (Qm). The measurements showed that moderate lanthanum substitution raised the piezoelectric response well above that of undoped PZT. Both d_{33} and k_p grew with lanthanum content up to an optimum composition, beyond which the electromechanical response gradually declined.

These improvements arise chiefly from greater ferroelectric domain mobility, a lower coercive field, a refined grain structure and the better densification achieved by the modified sol–gel process. The coexistence of several crystallographic phases close to the morphotropic phase boundary further assists polarization rotation under an applied field and so raises the efficiency of electromechanical conversion.

At elevated lanthanum levels, over-substitution created additional lattice defects and vacancy clusters that hindered domain-wall movement. The slight fall in density and the appearance of isolated pores also impaired the passage of mechanical energy through the ceramic, lowering the piezoelectric coefficients.

For the best compositions the d_{33} values lay within the range usually quoted for high-performance PLZT made by chemical routes, and the planar coupling factor likewise improved substantially clear evidence of efficient interconversion between electrical and mechanical energy.

Mechanical quality-factor data indicated moderate internal losses, implying that these ceramics suit sensing and precision actuation better than high-power ultrasonic use. On the whole, the combination of high permittivity, an improved piezoelectric coefficient and stronger electromechanical coupling demonstrates how effectively lanthanum substitution enhances the functional behaviour of PLZT.

5. CONCLUSION

Lanthanum-modified lead zirconate titanate ceramics were prepared successfully by a modified sol–gel technique followed by controlled calcination and sintering. This route delivered highly uniform powders and dense sintered pellets of excellent crystallinity and phase purity.

Structural study confirmed that a single-phase perovskite had formed across the whole compositional range examined. The microstructure was uniform, with grains evenly distributed and porosity kept low, showing that the chosen processing conditions favour dense ceramics of refined grain morphology.

Lanthanum addition had a pronounced effect on both the dielectric and the electromechanical behaviour. Higher lanthanum content steadily lowered the Curie temperature while boosting permittivity near the morphotropic phase boundary. The best compositions offered improved piezoelectric charge coefficients and electromechanical coupling, a consequence of greater domain mobility, better densification and favourable phase coexistence.

The findings reveal a firm link between chemical composition, crystal structure, microstructure and functional response. By adjusting lanthanum content appropriately, the electrical and electromechanical behaviour of PLZT can be tailored to particular engineering needs.

The strong dielectric response, high piezoelectric sensitivity and stable electromechanical behaviour recorded here mark the prepared PLZT ceramics as promising materials for advanced sensors, precision actuators, ultrasonic transducers, vibration-damping devices, energy harvesters and microelectromechanical systems (MEMS).

Future work could concentrate on refining the processing parameters, producing textured or thin-film PLZT, examining reduced-lead compositions and testing long-term durability under repeated electrical and mechanical loading. Such efforts would further widen the technological reach of PLZT-based ferroelectric ceramics in next-generation smart-electronic systems.

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