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Structural and Dielectric Study of a PLNZNT Ceramic Material Doped with Chromium

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ABSTRACT

The purpose of this work is to show the effect of Chromium (Cr) on the microstructural and dielectric properties of solid solution: $[Pb_{(0.975)}\ La_{0,\ 015}Nd_{0.01}][(Zr_{0,\ 524}Ti_{0,\ 476})_{(0.9875-\ (w/4))}Nb_{0.005}\ Cr_wO_3]$ with w = 0.0, 0.5, 1.0, 2.5 and 5.0 molar % (abbreviated PLNZNCT) were developed by solid state chemical reaction. The analysis by X-ray diffraction and the calculation of the tolerance factor (t) show a stable single-phase perovskite structure of tetragonal symmetry sintered at 1200°C. The SEM micrograph, the density test and the dielectric measurements indicate that the PLNZNCT ceramic achieves optimal values at 1 mol% Cr3 +. These obtained results, perhaps characterize the classic hard ferroelectric materials.

1. INTRODUCTION

The dielectric ceramics Pb (Zr, Ti) O_3, abbreviated (PZT) are very sensitive oxides to insertion or substitution at site A (and / or) site B, because of their simple perovskite ABO_3 structure. The purpose of doping is often related to the improvement of ceramic properties for their industrial applications (sensors, actuators and resonators) [1]. The properties of solid solutions with perovskite structure can be modified either by a cationic (and/or) anionic change according to the different substitutions and the different stages of development [2]. Doping of ions (La^{3+}, Nd^{3+}) at site A (Pb^{2+}) and ions (Nb^{5+}, Cr^{3+}) at site B (Zr^{4+}/Ti^{4+}) can change the properties of solid solutions (PZT) in order to obtain PLNZNCT ceramic from the complex perovskite structure ($AA'BB'O_3$) of the high performance [3]. The best electrical properties can be located around the concentration zone of x=52.4 for PLNZNT ceramic [Pb (Zr_X, $Ti_{(1-X)}$) O_3 doped with 1.5 mol% La, 1mol% Nd and 0.5mol % Nb] can be used in piezoelectric applications for the manufacture of sensors and motors [4]. Studies with other rare earth oxides are very limited. Therefore; in this work the Cr^{3+} element was chosen as a dopant to determine their effect on crystalline structure and dielectric properties for PLNZNT ceramic. Formulas and chemical compositions have been presented in Table 1 [2].

Table 1. Stoichiometric PLNZNCT ceramic compositions synthesized

General Formula: $[Pb_{(1-(x+y))}La_xNd_y][Zr_{0.524}Ti_{0.476}]_{(1-\ x/4\ -\ y/4\ -\ (5z/4)\ -\ (3w/4))}\ Nb_z\ Cr_wO_3$
Chemical Formula: $[Pb_{0.975}\ La_{0.015}Nd_{0.01}][Zr_{0.524}Ti_{0.476}]_{(0.9875-3w/4)}\ Nb_{0.005}\ CrwO_3$
with: x = 0.015, Y= 0, 01 and z = 0.005
w = 0.00 mol% $[Pb_{0.975}\ La_{0.015}\ Nd_{0.01}][Zr_{0.524}\ Ti_{0.476}]_{0.9875}\ Nb_{0.005}\ O_3$
w = 0.50 mol% $[Pb_{0.975}La_{0.015}\ Nd_{0.01}][Zr_{0.524}\ Ti_{0.476}]_{0.9837}Nb_{0.005}Cr_{0.005}O_3$
w = 1.00 mol% $[Pb_{0.975}\ La_{0.015}Nd_{0.01}][Zr_{0.524}\ Ti_{0.476}]_{0.980}\ Nb_{0.005}Cr_{0.01}O_3$
w = 2.50 mol% $[Pb_{0.975}\ La_{0.015}Nd_{0.01}][Zr_{0.524}Ti_{0.476}]_{0.9687}\ Nb_{0.005}Cr_{0.025}O_3$
w = 5.00 mol% $[Pb_{0.975}\ La_{0.015}Nd_{0.01}][Zr_{0.524}Ti_{0.476}]_{0.9500}\ Nb_{0.005}Cr_{0.050}O_3$

2. EXPERIMENTATION

2.1 Treatment of ceramics

In order to develop the desired material (PLNZNCT), commercial oxides of 99 % purity were used as powder base product (PbO, La_2O_3, Nd_2O_3, ZrO_2, Nb_2O_5, Cr_2O_3 et TiO_2). The latter were synthesized with solid state reaction method at high temperature. After grinding and compaction in the form of cylindrical pellets, the obtained PLNZNCT solid solution were calcined at 930 °C with a heating rate of 5 °C/min for 2 hours. This operation reduces the size of the grains and increases the reactivity PLNZNCT ceramic. The calcined powders were regrinded and compacted in the form of a disk of diameter 13 mm and 1.5 mm thick, and then treated at 1200 °C with a heating rate of 2 °C/min for 3 hours in order to complete the crystallization and increase the density of the material. The volatility of lead oxide (PbO) during sintering makes this material non-stoichiometric so it is important to add small amounts of $PbZrO_3$ to minimize the loss of PbO oxide

and to improve the dielectric properties of the final product [5-6]. To measure the dielectric properties, the electrodes were made by applying a silver paste on the two main faces of sintered pellets, followed by a heat treatment at 500 °C for 3hours [5].

2.2 Relative density and porosity measurement

Density is a physical quantity that influences the structural and electrical properties of ceramics. The relative density D (compactness) is given by the equation (1) of the material PLNZNCT sintered at 1200 °C:

$$D=\frac{\rho \exp}{\rho th} \quad (1)$$

such that:

ρ_{exp}: the experimental density calculated from the mass and the volume of fritted ceramics pellet.

ρ_{th}: the theoretical density obtained from the X-ray diffraction database providing the mesh parameters.

To verify the densification of the ceramic, the porosity (P) of the pellets were measured by the equations (2) [5]:

$$P=1-\frac{\rho \exp}{\rho th}=1-D \quad (2)$$

2.3 Structural characterization

An X-ray Diffractometer (XRD), D8 ADVANCE type *(*BRUKER-AX*)* with Cu-K α_1=1.5406 Å filtered radiation was used to characterize the crystal structure of the sintered samples. The scan was recorded in the range of 2θ varies from 5 to 85˚ with a scanning speed of 2˚/min.

2.4 SEM microstructure analysis

In order to study the microstructure of elaborate PLNZNCT samples, SEM images were obtained using a JEOL JSM-6390lv scanning electron microscope.

2.5 Dielectric characterization

The dielectric constant and the dissipation factor (tanδ) were measured using an HP/Agilent 4284A RCL-meter impedance bridge at a temperature ranging from 30 °C to 500 °C.

3. RESULTS AND DISCUSSION

3.1 X-ray diffraction studies

Figure1 illustrates X-ray diffraction patterns for Cr^{3+} doped PLNZNT ceramics. The results of the XRD for the samples under our study show a peak formation: (101) and (110), (002) and (200), (102) and (201), (112) and (211) were produced at 2θ = 30.930 ° and 31.351°, 43.828° and 44.978°, 49.735° and 50.486 ° and 54.992° and 55.743°, respectively correspond to a pure perovskite structure of tetragonal phase [7]. According to Hardtl and Hennings, it is estimated that (La^{3+}/Nd^{3+}) ions replace partially the Pb^{2+} ion at site A by producing defects site B. The ions (Nb^{5+}/Cr^{3+}) partially replace the (Zr^{4+}/ Ti^{4+}) ions at site B to maintain the charge balance of the tetragonal structure. A good distribution of the gaps (lacunes) ensures the equilibrium of the electric charges at site A (and / or) at site B in order to obtain the electro-neutrality of the perovskite of the PZT ceramic [8].

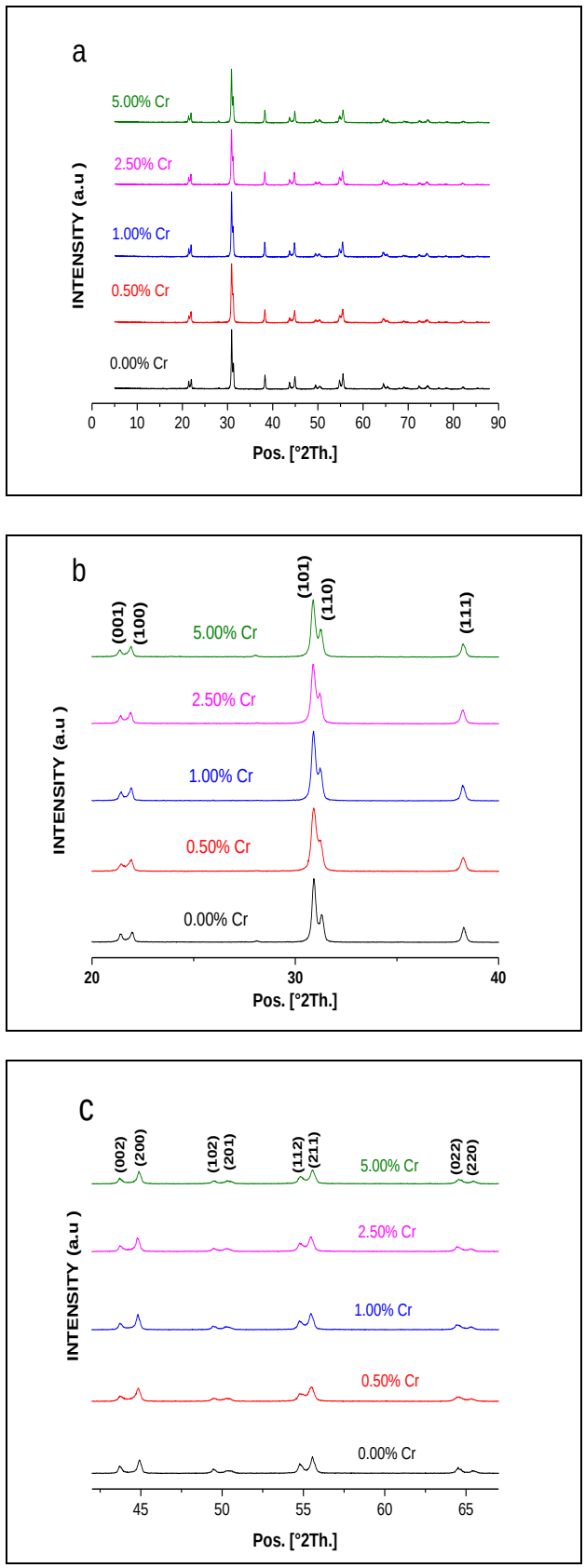


Figure 1. Representative X-ray diffraction of Cr-doped PLNZNT ceramic at the 2θ from: a - 05° to 90° b - 20° to 40°, c - 40° to 70°

The mesh parameters obtained from the PLNZNCT ceramics in Table 2, undergo expansion and then condensation in the direction of the (a_T) axis and also a small variation between the mesh parameters (a_T, c_T). The tetragonality ratio (c/a) changes with the chromium oxide substitution, which does not typically affect the tetragonality of the network structure.

Table 2. Structural parameters calculated from the XRD data

Cr(w)	a=b(A°)	c (A°)	c / a	V(A°) ^{3}
00.00	4.02811	4.01289	0.99622	66.9951
0.005	4.03423	4.12018	1.02130	67.056
0.010	4.03587	4.12452	1.02196	67.1812
0.025	4.03506	4.12137	1.02139	67.1029
0.050	4.02317	4.12001	1.02407	66.6860

3.2 Microstructural study of ceramics

To study the microstructural characteristics of PLNZNCT ceramic materials, SEM scanning electron microscopy is used to determine the most compacted structure. Figure 2 shows that the studied samples have a microscopic structure consisting of granules of different shapes and sizes. The grain boundary is compact and the appearance of pores is very low. Generally larger grains correspond to higher dielectric properties. At high size, homogeneous distribution and more dense grain was observed at 1 molar % Cr ratio. But changing this concentration will result in a decrease in grain size and affect negatively the dielectric properties, due to the deterioration of the microstructure [9].

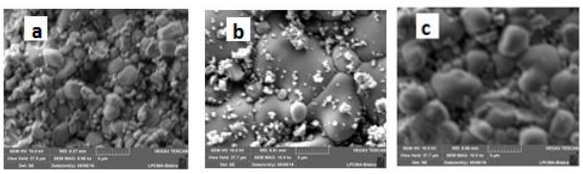


Figure 2. SEM micrographs of PLNZNCT ceramics with different amounts of Cr sintered for 2h at 1200 °C (a = 0.5; b = 1; c = 2.5) Cr %

3.3 Tolerance factor

The stability of the perovskite structure (ABO3) is characterized by the tolerance factor (t) which determines the compactness and freedom of movement of cations A and B. According to Goldschmidt, the structure is stable if 0.88 <t <1.09, but it exhibits a distortion in the stability of the structure beyond this range. The tolerance factor (t) could be determined by the following formula:

$$t=\frac{\sum Ra + Ro}{\sqrt{2}(\sum Rb + Ro)} \qquad (3)$$

where: ∑Ra, ∑Rb and Ro are respectively the ionic radii of cations; A, B and Oxygen [10]. The perovskite structure is more stable when the tolerance factor (t) is close to 1. The involved bonds play a strong ionic character [11]. The perovskite range showed a constant orientation with a slight constant change (Δt) of 0.00063 in tolerance values (t) [12]. According to the results in Table 3 it can be concluded that PLNZNT becomes more and more stable with the increase of the chromium content.

Table 3. Tolerance factor (t) of PLNZNCT ceramics

Cr.mol %	Tolerance(t)	Variation(Δt)
0	0.918546	//
0.005	0.9191832	0.0006372
0.01	0.9198213	0.0006381
0.025	0.921741	0.0019197
0.05	0.9249583	0.0032173

3.4 Relative density and porosity

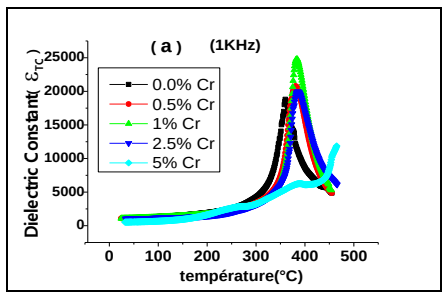
Table 4 shows the variations in relative density and porosity of PLNZNCT ceramic sintered at 1200 °C as a function of Cr concentration. It is distinguished that the PLNZNT ceramic doped with 1mol% of Cr reaches a maximum value of the apparent density and minimum value of the porosity. The high values of the density and the low values of the porosity confirm a disappearance of the pores, a reduction of the volume of the crystalline mesh and a more compact structure [13].

Table 4. The density and porosity of PLNZNT ceramics according to the rate of Cr

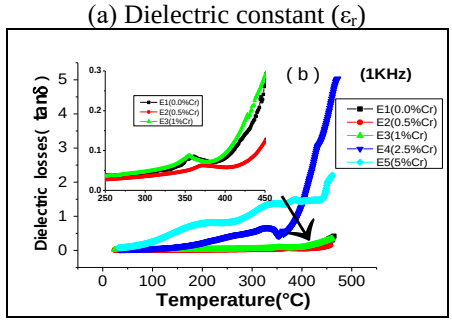
Cr(w)	ρ_{th} (g/cm^3)	ρ_{exp} (g/cm^3)	D %	1-D
0	8.02683	7.51050	93.567646	0.06044
0.005	8.00544	7.55357	94.355481	0.05645
0.01	7.9674	7.66444	96.197612	0.03803
0.025	7.9696	7.46776	93.702069	0.06298
0.05	7.9222	7.41952	93.654784	0.06346

3.5 Dielectric characterization

The two Figures, 3(a, b) and 4(a, b) give the experimental results, the measurements of the dielectric parameters including the dielectric constant (εr) and the dissipation factor (tanδ) of the PLNZNCT ceramic sintered at 1200 °C, at the frequency of 1kHz depending on the temperature.

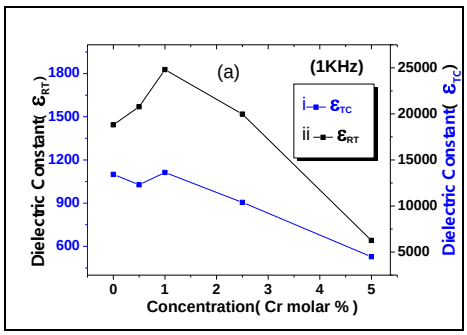


(a) Dielectric constant (ε_r)

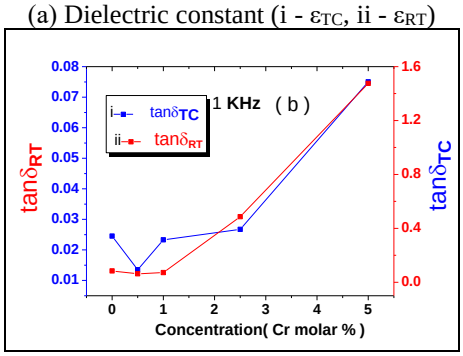


(b) Dielectric losses (tan δ)

Figure 3. Influence of the temperature in the dielectric proprietys (a-εr and b- tanδ) of the PLNZNCT ceramics



(a) Dielectric constant (i - ε_{TC}, ii - ε_{RT})



(b) Dielectric losses (i- $\tan\delta_{TC}$, ii- $\tan\delta_{RT}$)

Figure 4. Influence of the Cr ratio in the dielectric propertys (εr and tanδ) at: (i) - room temperature (R.T), (ii)- transition temperature (T.C) for PLNZ NCT ceramics

According to previous studies, the doping of the acceptor ions in the perovskite crystal lattice decreases the dielectric constant and increases the curie temperature (Tc) [14]. The strongest dielectric response is that associated with the doped sample of 1mol %Cr. The dielectric losses reach minimum values, for PLNZNT ceramic doped with; 0.0, 0.5 and 1 molar % of Cr, comparing with PLNZNT ceramic doped with 2.5 and 5 molar % of Cr [15]. Above the phase transition temperature, the dielectric losses of the PLNZNCT ceramic increase strongly and constantly throughout the series. This leads to the increase in conductivity, which could be due to the electro-dipolar polarization of Cr-doped PLNZNT ceramics [16].

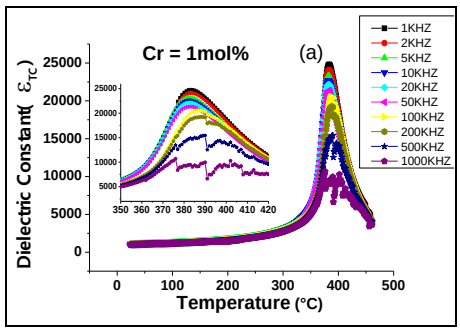
The increase in PLNZNCT ceramic dielectric constant doped with 1 % chromium may also be due to the multiple donor cations (La^{3+}, Nd^{3+} at site A and Nb^{3+} at site B) and the Cr^{3+} acceptor cations at site B [17]. The dielectric properties for the different systems studied have been summarized in Table 5 [18].

Table 5. Critical values of the dielectric properties of PLNZNT doped with Cr

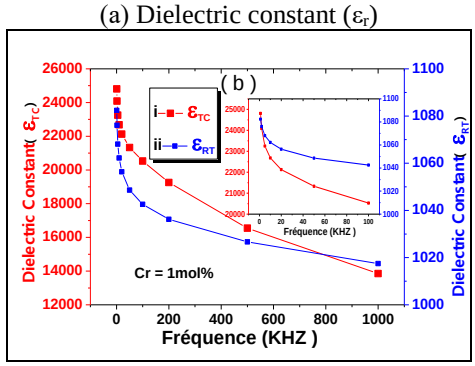
Cr molar%	ε_{RT}	$tn\delta RT$	T_C (°C)	εmax (T)	$tn\delta_{Tc}$
0.0	1099.72	0.0245	360	18811.66	0.0840
0.5	1027.84	0.0135	381	20772.64	0.0626
1.0	1113.21	0.0233	383	24805.80	0.0721
2.5	904.77	0.0267	385	19965.95	0.4865
5.0	527.59	0.0750	390	6259.96	1.4756

3.4.1 Variation of the dielectric constant (ε_r)

Figure 5 (a, b) illustrates the effect of the frequency on the dielectric constant (εr) of the PLNZNT ceramic doped with 1 mole% Cr sintered at 1200 °C as a function of temperature. It can be seen that the curves of (εr) pass through the same stage and reach maximum values at the transition temperature of the ferro - paraelectric phase. Therefore the PLNZNCT ceramic belongs to the classic ferroelectric material family hard, this perhaps explains the diminution of the dielectric permittivity above the Curie temperature (Tc) [19]. It has been observed that the dielectric constant decreases with the increase of the frequency, which can be attributed to the existence of different types of decreasing polarization [17].



(a) Dielectric constant (ε_r)

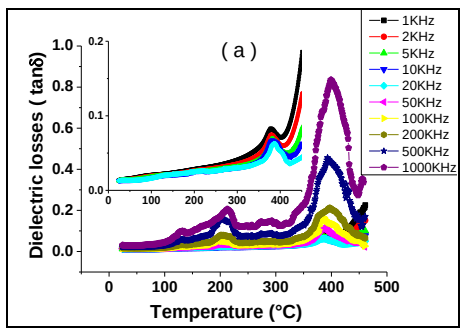


(b) Dielectric constant (i - ε_{TC}, ii - ε_{RT})

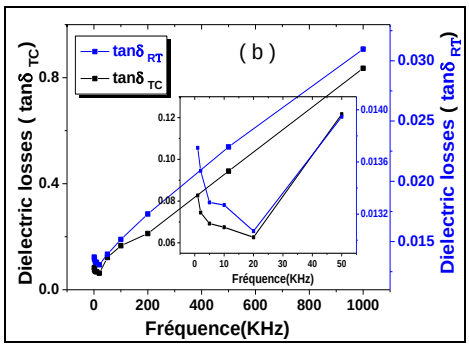
Figure 5. Influence of Frequency in the constant dielectric (εr) for 1molar% PLNZNCT Ceramics at: (i) - room temperature (R.T), (ii)- transition temperature (T.C)

3.4.2 Evolution of the dissipation factor (tanδ)

Figure 6 (a, b) shows the effect of the frequency on the dissipation factor (tanδ), which plays an important role in the high frequency circuits of the PLNZNT ceramics doped with 1 molar % of Cr and sintered at 1200 °C, depending on the temperature. It is seen that the maximum values of dielectric loss in the vicinity of the transition temperature (Tc) decreases as the frequency increases to 20 KHz; then they increase with the increase of the frequency from 50 KHz up to 1000 KHz, this is due to the movements of the dipole moments. The increase in temperature causes a deterioration of the properties of the material and distortion of the structure, which are related to the movement of the walls of the domains [20]. The dissipation factor is associated with a rate of electrical energy loss, often in the form of dissipated heat [21].



(a) Dielectric losses ($\tan\delta_{TC}$)



(b) Dielectric losses (i- $\tan\delta_{TC}$, ii- $\tan\delta_{RT}$)

Figure 6. Influence of Frequency in the dissipation Factor (tanδ) for 1molar% PLNZNCT Ceramics at: (i) - room temperature (R.T), (ii)- transition temperature (T.C)

4. CONCLUSION

Solid solutions of the type $[Pb_{0.975}La_{0.015}Nd_{0.01}][Zr_{0.524}Ti_{0.476}]_{(0.9875-3w/4)}Nb_{0.005}CrwO_3$ with w = 0.0, 0.5, 1.0, 2.5 and 5.0 molar % abbreviated PLNZNCT were synthesized by the conventional solid state method, sintered at 1200 °C. The X-ray diffraction pattern in the range of 05 ° to 85 ° shows that all compositions have a pure crystallized perovskite structure. The multilayer surface PLNZNCT at 1 molar% Cr ratio shows homogeneous uniform grain distributions in the SEM image. The calculation of the tolerance factor (t) of PLNZNCT material can confirm the stability of the perovskite structure. The doped sample of 1 molar% of Cr shows a denser microstructural development (7.6644g/cm^3), less porous (0.03803) and a higher dielectric permittivity (24805.8) at the transition temperature (Tc=383 °C) during sintering. The reduction of the dielectric constant and the dielectric loss with the increase of the frequency is due to the movements of the dipole moments, which explain the existence of the different types of polarization. Increasing the rate of chromium in PLNZNT ceramics is often associated with loss of electrical energy due to the growth of conductivity, resulting in deterioration of material properties that are related to the movement of the domain walls. According to the results of this study, PLNZNCT ceramic, considered a hard-ferroelectric material, can be used at different frequencies in several technological fields.

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