IOSR Journal of Applied Physics (IOSR-JAP)
e-ISSN: 2278-4861.Volume 6, Issue 5 Ver. II (Sep.-Oct. 2014), PP 20-29
www.iosrjournals.org

Effect of grain size on the dielectric properties of Lanthanum-doped $PbTiO_3$ perovskite ceramics.

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Abstract: *Lead lanthanum titanate, (PLT) ceramics were fabricated by conventional combustion technique and the influence of different sintering temperature (1170, 1200, 1230, 1260 and 1300 ^oC) at a fixed of sintering time for 2h on the microstructure and dielectric properties were investigated. The particle size of PLT ceramics which can be calculated by Scherer formula from XRD (x-ray diffraction) analysis is increased with increasing sintering temperature. The density of the samples also increases with increasing sintering temperature (Ts), which can be calculated from XRD pattern and can be observed from SEM (scanning electron microscope) technique. Dielectric properties of the samples were studied as a function of sintering temperature and frequency, where the maximum value of dielectric constant (ε_{max}) increased with increasing Ts, but it decreased with increasing frequency, but the transition temperature independent on the applied frequency. The versus imaginary permittivity and dielectric loss with frequency at room temperature were investigated. The diffusion coefficient (γ) of PLT ceramics are decreased with increasing sintering temperature.*

Keywords: *Lead titanate, Combustion technique, X-ray diffraction; sintering temperature, dielectric properties.*

I. Introduction

Lead titanate ceramics undergo a ferroelectric-to-paraelectric transition at 490 ^oC (Curie point) [1]. The crystal structure of PT in the paraelectric phase is cubic (space group Pm3m), while the ferroelectric phase below the Curie point exhibits a tetragonal phase (space group P4mm). When PT is cooled, across the phase transition it becomes fragile due to occurrence of significant changes in the unit cell dimensions which cause large internal stresses [2,3]. By adding suitable isovalent dopants such as (Ca^{2+}, Ba^{2+}, Cd^{2+}, ……etc) or off-valence (Sm^{3+}, Gd^{3+}, La^{3+}, Y^{3+}, ……etc) ions into the Pb sites of lead titanate, the lattice anisotropy is reduced [4,5], resulting in hard and dense ceramics with high mechanical strength. Ravender Tickoo et al [6] had reported that, the value of dielectric constant at room temperature was increased with increasing La content on the sample ($Pb_{1-x}La_xTi_{1-2y}Mo_yFe_yO_3$ with x=0.02, 0.05, 0.10, 0.15, 0.20 and y=0.02), also they have been observed that the transition temperature decreases at the rate of 14 ^oC per mol% of La substitution. Coercive field and c /a ratio are continuously decreased with increasing in La addition.

Jiang et al [7] studied varying average grain size of $PbTiO_3$ nanocrystalline ceramics with sintering temperature and they observed the dielectric phase transition more diffuse as the particle size decreased. Buscaglia et al [8], observed a similar behavior when they studied $BaTiO_3$ nanostructured ceramics prepared by a spark plasma sintering technique (SPS), they observed a shift in the phase transition temperature (Curie temperature) a shift towards lower values by decreasing the grain size was smaller than 100 nm.

Wei Liu et al [9] had reported that, the bulk density and grain size of porous PZT ceramics is increased with increasing of sintering temperature, consequently the porosity of the samples will decrease and the samples become more denser with increasing of sintering temperature which exert remarkable influence on the value of relative permittivity (ε_r) and piezoelectric strain coefficient (d_{31} and d_{33}).

Ukrit Chaimongkon, et al [10] studied effect of sintering temperature on the phase formation microstructure and dielectric properties ($Pb_{1-x}Ba_x$) TiO_3 PBT ceramics with $0.2< x< 0.8$ prepared by the combustion technique, under various of sintering temperature (1125-1275 ^oC) for 2 h, they mentioned that, both of value of average grain size and maximum value of dielectric constant for all the samples are increased when the firing temperatures is increased, but the Curie temperature is decreased when x is increased. Carreaued et al [11] studied ceramic samples with a lower density ($PbMg_{1/3}Nb_{2/3}O_3$) and they observed a decrease in the maximum value of dielectric constant with decreasing the average grain size.

The aim of the present work is to extend our previous investigation to other new material compositions, and provide an analysis of the effect of particle size on the structure and the dielectric behavior of (PLT20) ceramics samples.

Another goal was to fabricate these ceramics by conventional solid state reaction in air atmosphere at higher sintering temperature and time, while improving or at least maintaining the comparable dielectric, ferroelectric and piezoelectric properties of PLT ceramic.

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II. Experimental procedure

Commercially available PbO (.99.9% purity, Aldrich, USA), La_2O_3 (.99.9% purity, Aldrich, USA), and TiO_2 (.99.9% purity, Aldrich, USA) powders were used as the starting materials. The oxides were weighed in desired molar proportions according to molecular formula $[Pb_{0.7}\ La_{0.2}]\ TiO_3$ and mixed. The mixture was dry ball milled for 6h using zirconia balls, and then powders were pressed into 7-mm diameter green pellets and about 1.5 mm thickness using a hardened stainless steel die at 40 MPa. These disks were further sintered at different sintering temperature [1170, 1200, 1230, 1260 and 1300 °C] for 2h in a conventional furnace. Finally the disks were polished and coated by applying silver paste on both sides as electrode to study the electrical properties. Perovskite phase formation was confirmed by X-ray diffraction pattern of the sintered pallets, using Philips X-ray diffractometer (CuKα radiation λ=1.5405 A°) at room temperature. The microstructures of the sintered samples were analyzed by using (JEOL, JSM-5500LV) scanning electron microscope (SEM). The density of the PLT ceramics was measured by the Archimede's method. The dielectric and ferroelectric properties of the sintered PLT samples were measured using an LCR meter (TH2826) (in the range 20Hz-5MHz

III. Results and discussions

3.1. Powder morphology

PLT pellets sintered at (1170, 1230 and 1300 ^oC) were examined by x-ray diffraction Fig. (1). According to this figure all samples are single phase and have major peak at (110). The average particle size (D) of the samples was calculated using Debye-Scherer method from the broadening of the diffraction line using the expression [12]:

$$D = 0.94\lambda/\beta\cos(\theta) \quad (1)$$

Where λ is the wave length of the CuKα radiation, β is the full width at half maximum (FWHM) of the diffraction main peak and θ is the Bragg diffraction angle at this peak. The average particle size is increased from 20 nm to 40 nm, when the sintering temperature increased from 1170 to 1300 ^oC.

The behavior can be explained according to the phenomenological kinetic grain growth equation expressed as [13]:

$$\log G = (1/n) \log t + (1/n) [\log K_o - 0.434\ (Q/RT)] \quad (2)$$

Where G is the average grain size at the time sintering, n is the kinetic grain growth exponent, K_o a constant, Q the apparent activation energy, R the gas constant, and T is the absolute temperature. The last equation shows that, the average grain size is increased with increasing firing temperature and this means that the porosity of the samples are decreased with increasing sintering temperature. This behavior can be observed in [14-16].

X-ray density (d_x) various of sintering temperature of the PLT ceramic was calculated using the lattice parameters obtained from XRD patterns by this equation

$$d_x = (Z.M) / (N.V), \quad (3), \text{ where}$$

Z = No. of formula units per unit cell.
M = Molecular weight (gm).
N = Avogadro's number.
V = Unit cell volume $(A^o)^3$.

According to Eq(3), value of density depend only the volume of unit cell which consequently depend on the lattice parameter, however the sintering temperature increase the volume of unit cell is decreased, so that the density must be increased.

Fig. 2, shows the particle size and X-ray density versus the sintering temperature, where this fig indicated that the value both of grain size and density are increased with increasing the sintering temperature. The increase in density is related to increase the particle size and decreased the dislocation of the samples with firing temperature.

Fig. 3(a) and (b) shows the lattice constant and lattice anisotropy (c/a) of PLT ceramic with various firing temperature respectively. This figure shows that both value of the lattice constant c-axis (002), a-axis (200) are decreases and consequently the volume of the cell is decrease with increasing the sintering temperature, but the tetragonality (c/a) is increased. This behavior can be observed in [17]. Table 1. Show the particle size, density and lattice parameters for all the sintered PLT ceramics.

3.2 Microstructure

The microstructure of PT ceramics doped La 20 % with different sintering temperature (1170, 1230 and 1300 ^oC), were carried out by Scanning electron microscope (SEM) Fig 4(a-c). It can be observed that the structure of samples is very homogeneous at high firing temperature and diameter of the grain boundary increased with increasing sintering temperature; this means that the dislocations and porosity are decrease with

increase sintering temperature and the sample will be denser. So that we can conclude the firing behavior is optimum the crystal structure for the ceramic samples.

3.3. Room temperature electric parameters

Room temperature dielectric constant measured at 10 kHz increases with increasing sintering temperature as show in Fig. (5). this is attributed to increasing density and grain size [18].

Fig. 6 shows the variation of dielectric constant at room temperature with frequency of the applied a.c. field within a range 100 Hz to 1MHz for the samples sintered at (1170, 1230 and 1300 ^{o}C) of PLT ceramic.
The decrease in dielectric constant with increasing in frequency can be expressed by Kramers Kronig relation [1]

$$\acute{\varepsilon}(\omega) - \varepsilon_{\alpha} = A_1\omega^{s-1} \qquad (4)$$

Where $\acute{\varepsilon}$ is the dielectric constant measured at frequency ω, ($\omega = 2\pi f$), ε_{α} is the dielectric constant measured at high frequency and exponent s must be in the range $0 \leq s \leq 1$. The exponent s represents the degree of interaction between mobile with the lattices around them, and the prefactor exponent A determines the strength of polarizability. Writing Eq. (4) in the form,

$\log [\acute{\varepsilon}(\omega) - \varepsilon_{\alpha}] = (1\text{-}s)\log \omega + \log A1$. A plot of $\log [\acute{\varepsilon}(\omega) - \varepsilon_{\alpha}]$ versus $\log (\omega)$ show the result as shown in Fig. 7.
The value of parameter s calculated from the slope $\log [\acute{\varepsilon}(\omega) - \varepsilon_{\alpha}]$ vs. $\log(\omega)$ curves is found to be less than unity and decreases with increase in temperature, where s = 0.75, 0.79 and 0.82 for T_s = 1170, 1230 and 1300 ^{o}C.

On other words, the dielectric constant at low frequency is dependent on various polarization effects (electronic – ionic – orientation polarization), but at high frequencies this value is dependent on the electronic polarization only, so that the value of dielectric constant are less. This behavior can observe in [19, 20].

3.4. Phase transition

Variation in dielectric constant for all the sintered samples as a function of temperature under frequency of electric field 10 kHz is reported in Fig. 8. The value of permittivity is increased with increasing temperature and reach a maximum value (ε_{max}) at phase transition (ferro-para transition) (T_c), then it is decreased with increasing temperature. Also this figure shows that the value of phase transition (T_c) toward the higher temperature with increasing sintering temperature where, if the sintering temperature is increased from 1170 to 1300 ^{o}C, the T_c is increased from 413 to 438 ^{o}K. The increase in dielectric constant with the sintering temperature is due to increasing of density and grain size. According to the space-charge theory from

Okazaki and Nagata [21], there is a specific amount of space charge sites inside grain boundaries and domain wall, this space charge due to lattice vacancies is responsible for forming electric field which effects the movement of domain walls. Because the ceramic materials become more denser with increasing sintering temperature it is expected that the space-charge fields will decrease because this field is eliminated by the space charge of surrounding grains more easily than in more porous ceramics. When the grain size increases, the surface area of the space-charge layer will decrease, and consequently the space-charge field will decrease simultaneously. Hence, the domain walls are quite free in large grains and denser ceramics, as well as dielectric constant increases for samples with high density and large grains size.
A linear increase of phase of transition temperature with sintering temperature for PLT ceramic is observed in Fig.9.

3.5. Dielectric loss

The effect of sintering temperature on dielectric loss as a function of frequency in range of 20Hz to 1MHz at room temperature is shown in Fig.10. for the PLT ceramics had sintering temperature (1170, 1230 and 1300 ^{o}C). It is to be noted that, the value of dielectric loss is very small for all the samples and this value is decreased with increase the applied frequency reached to minimum value, then it increased with increase the frequency. This peak appears when the frequency of electric charge has approximately value equal to the value of external applied electric field. This peak is noticeable at high sintering temperature and shift toward lower frequencies. This is attributed to increase in grain size due to high sintering temperature and this behavior can observed [22-24].

Fig. 11 show the frequency dependence of imaginary part of dielectric constant at room temperature for PLT ceramics sintered at 1170, 1230 and 1300 ^{o}C. for all the range of frequency, behavior of imaginary dielectric constant is similar to behavior of dielectric loss, where the value of imaginary permittivity is decrease with increase frequency reached to minimum value, then it increase with increase frequency.
3.5. Sintering temperature effect-induced diffused phase transition
The degree of diffuseness of the transition is studied by using expression [25];

$(1/\varepsilon - 1/\varepsilon_{max}) = 1/C(T - T_c)^{\gamma}$, $(T > T_c)$, (5)

Where γ and C are assumed to be constants. The value of γ determines the degree of diffuseness of the phase transition which lies in the range of $1 < \gamma < 2$. The value of γ is equal to 1 for a system with a completely ordered transition while γ between 1 and 2 indicates diffused transition. According to Eq (5) the slope of $\log(1/\varepsilon - 1/\varepsilon_{max})$ versus $\log(T - T_c)$ gives the value of γ.

The graphs of $\log(1/\varepsilon - 1/\varepsilon_{max})$ versus $\log(T-T_c)$ for the samples $(Pb_{0.7}La_{0.2})$ TiO_3 ceramics have sintering temperature (1170, 1230 and 1300 ^oC) at an applied field of 10 kHz were plotted, giving the results shown in Fig12. The value of γ is (1.567, 1.824 and 1.956) for (1170, 1230 and 1300 ^oC), are respectively.

It is evident that the diffuseness on the phase transition of PLT ceramics is decreased with the increasing of sintering temperature. This behavior can be interpretation due to, in the ferroelectric material the diffuse phase transition increase with decrease of grain size and this behavior can be observed in [26,29].

Table 1. Particle size, density and lattice parameters for sintered PLT ceramics for all the samples

annealing temperature (^oC)	**1170**	**1200**	**1230**	**1260**	**1300**
Particle size (nm)	20	26	31	36	40
Density (gm/cm^3)	2.982	3.0022	3.03	3.044	3.056
a (A^o)	3.9079	3.8921	3.8715	3.8615	3.8505
c (A^o)	3.9399	3.9287	3.9234	3.9055	3.9005
V $(A^o)^3$	15.3971	15.2908	15.1507	15.081	15.0188
c/a	1.0082	1.0094	1.0108	1.0114	1.0129
έ	450	690	878	930	1000
$\acute{\varepsilon}_{max}$(10KHz)	2154	5500	8187	10230	12910
T_c (^oK)	413	420	428	433	438
γ	1.567		1.824		1.956

IV. Conclusion

PLT ceramics have been manufactured by solid state reaction method, and sintering at different temperature, microstructure and dielectric properties for the samples have been show. When the sintering temperature increased from 1170 to 1300 ^oC, the porosity of the samples decreased, and consequently the density and grain size increased obviously. The maximum value of dielectric constant increased with increasing sintering temperature attributed to an increased in grain size and density, which can be explained according to space-charge theory from Okazaki. The maximum value of dielectric constant decreased with increased the frequency but the phase transition of temperature is independent of frequency. The value of dielectric loss at room temperature decrease with increase frequency and reached to minimum value, then it increase with frequency and this peak related to the frequency of electric charge equal to the frequency of applied electric field, but both of value of real permittivity and imaginary permittivity at room temperature are decrease with increase frequency without appreciable peak .The diffused of phase transition of PLT ceramics is decreased with increasing sintering temperature, and this indicated that the quality of the samples is increased with sintering temperature. In other words, we can conclude that the sintering temperature is considered to be the essential factor for improving the quality of the ceramics samples.

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Caption of figures:

Fig.1. Shows XRD patterns of variation sintering temperature for the sample [($Pb_{0.7}$ $La_{0.2}$)] TiO_3 ceramics sintered at (1170, 1230 and 1300 ^oC).

Fig.2. Effect of sintering temperature of ($Pb_{0.7}La_{0.2}$)TiO_3 ceramic on grain size and density.

Fig. 3. Dependence of the (a) lattice constant and (b) lattice anisotropy (c/a) of [($Pb_{0.7}La_{0.2}$)] TiO_3 ceramics on the sintering temperature.

Fig. 4. SEM analysis of (a), (b) and (c) represented the sample [($Pb_{0.7}La_{0.2}$)] TiO_3 ceramics sintered (1170, 1230 and 1300 ^oC) are respectively.

Fig.5. Dielectric constant measured at room temperature of PLT ceramic versus with sintering temperature for all the samples.

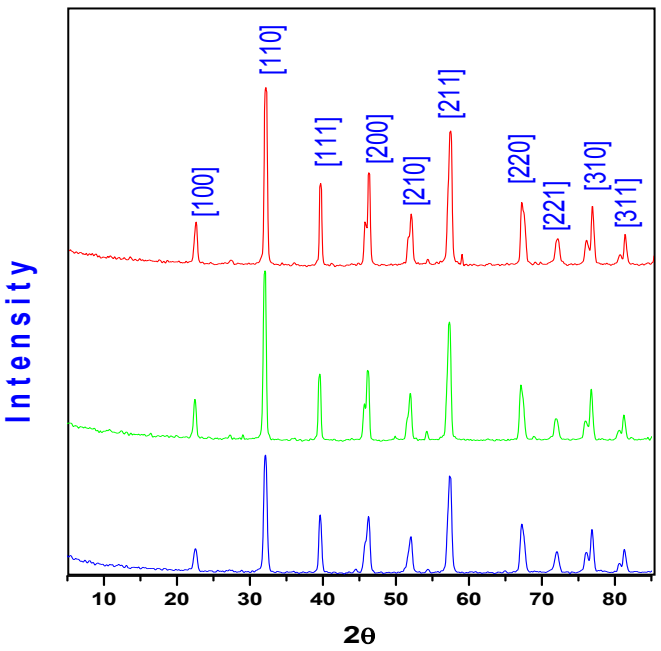
Fig.6. Dielectric constant measured at room temperature versus frequency of PLT ceramic sintered (1170, 1230 and 1300 ^oC).

Fig.7. log log [$\acute{\varepsilon}(\omega) - \varepsilon_\alpha$] versus log ($\omega$) of PLT ceramic Sintered (1170, 1230 and 1300 ^oC).

Fig.8. Variation of dielectric constant with temperature for the sample [($Pb_{0.7}La_{0.2}$)] TiO_3 ceramics sintered (1170, 1230 and 1300 ^oC) at 10 kHz.

Fig. 9. Effect of sintering temperature on transition temperature of La doped PT ceramic for all sintered the samples.

Fig. 12. A plot of $\log(1/\varepsilon - 1/\varepsilon_{max})$ versus $\log(T - T_c)$ for PLT ceramics, with sintering temperature (1170, 1230 and 1300 ^oC).



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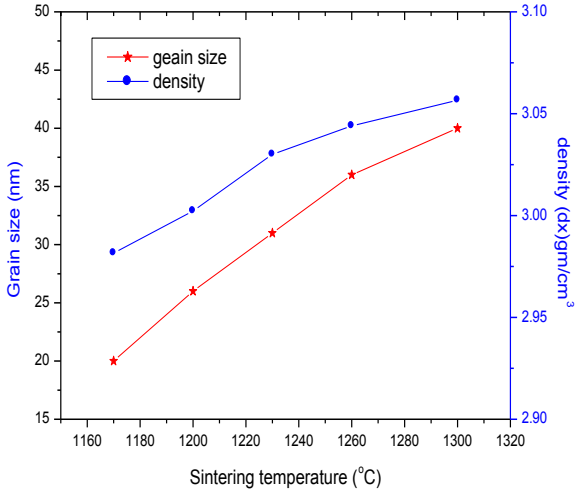
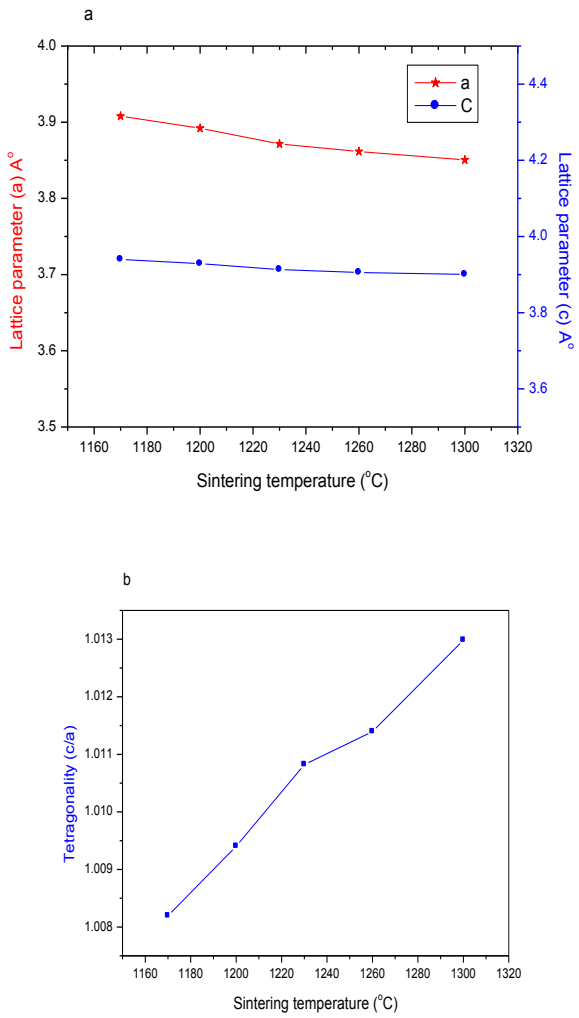


Fig.2. Effect of sintering temperature of $(Pb_{0.7}La_{0.2})TiO_3$ ceramic on grain size and density.



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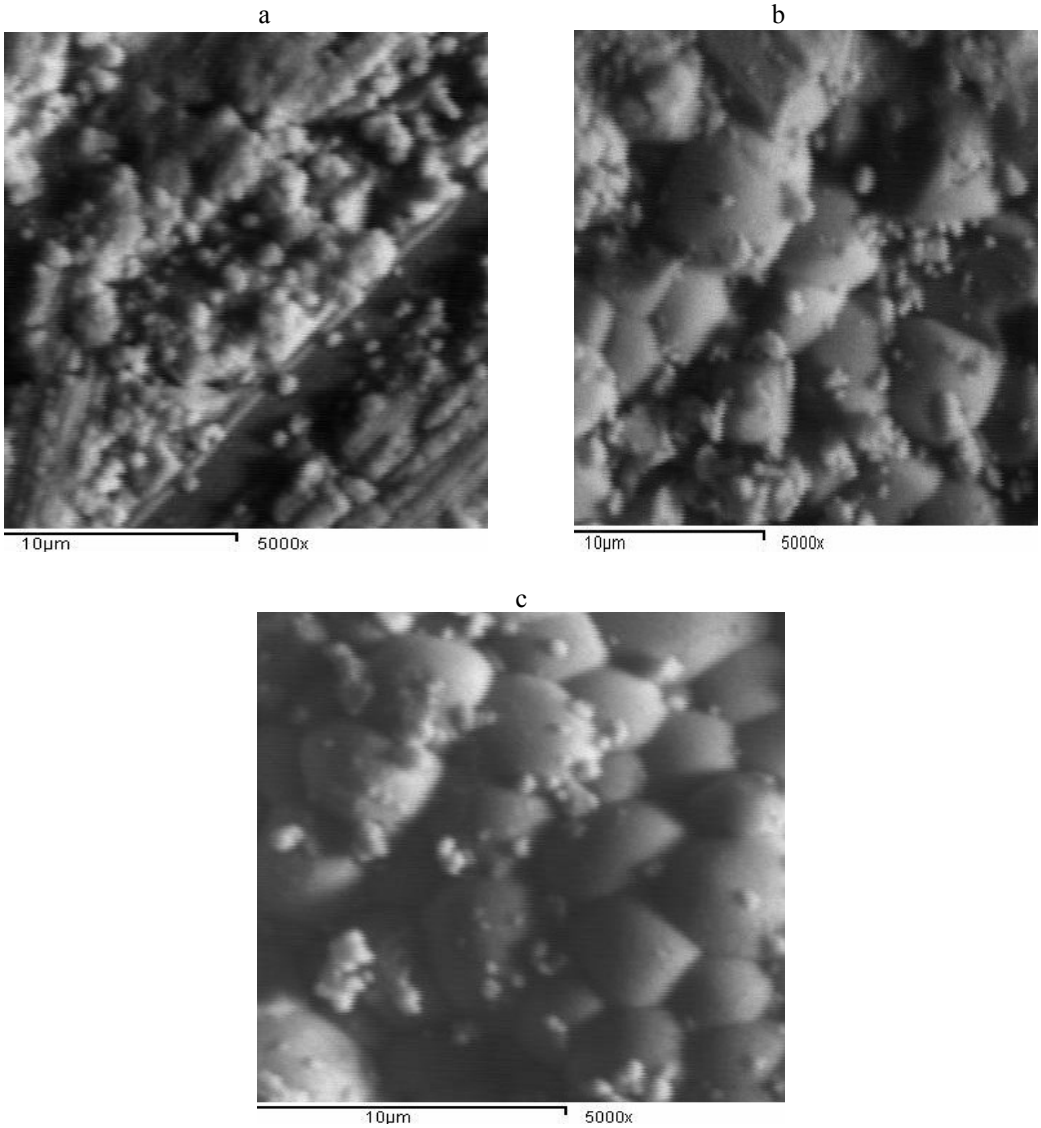

Fig. 4. SEM photographs of (a), (b) and (c) represented the sample [$(Pb_{0.7}La_{0.2})$] TiO_3 ceramics sintered (1170, 1230 and 1300 ^oC) are respectively.

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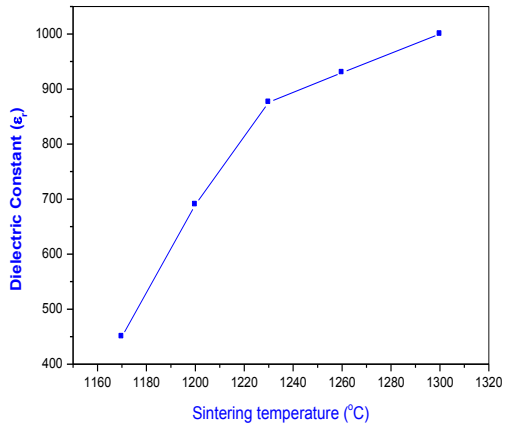
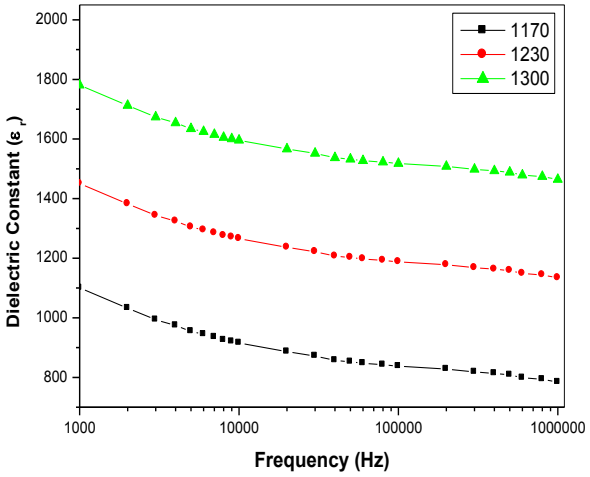


Fig.5 Dielectric constant measured at room temperature of PLT ceramic versus with sintering temperature for all the samples.



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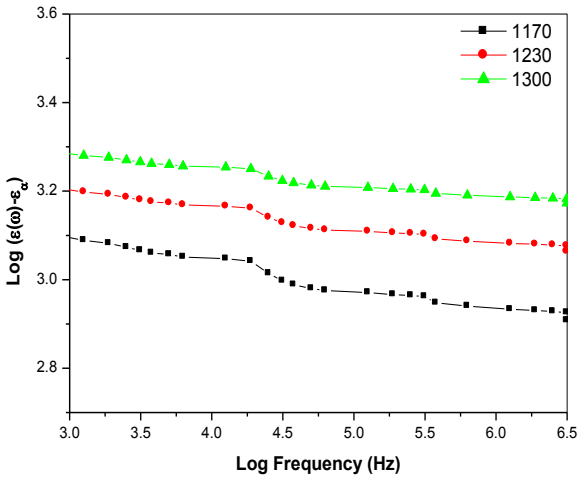
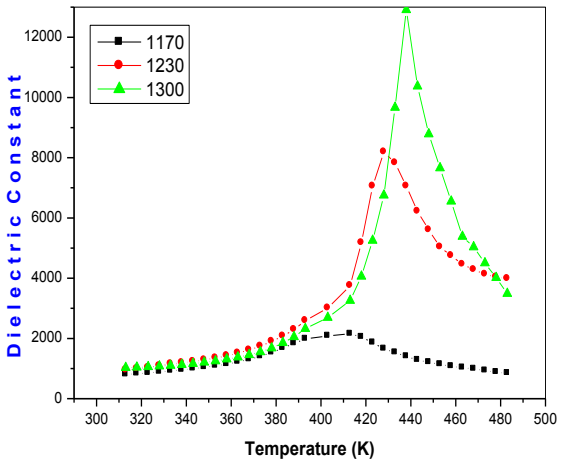


Fig.7. log log [έ(ω) - ε_{α}] versus log (ω) of PLT ceramic Sintered (1170, 1230 and 1300 ^{o}C).



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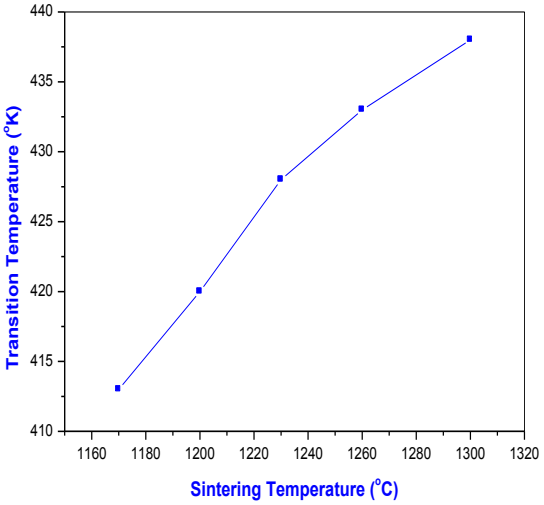

Fig. 9. Effect of sintering temperature on transition temperature of La doped PT ceramic for all sintered the samples.

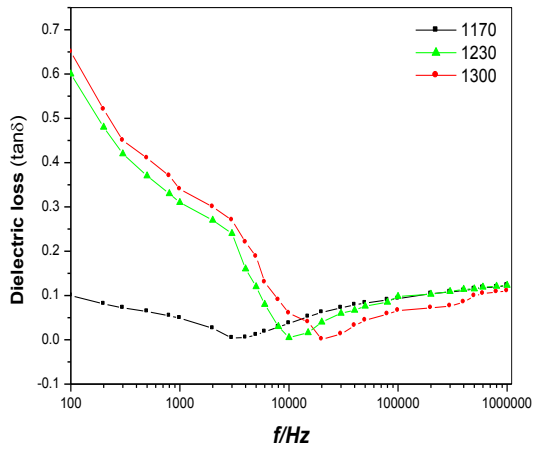

Fig. 10. Variation of the tanδ with frequency for PLT ceramic sintered at (1170, 1230 and 1300) at room temperature

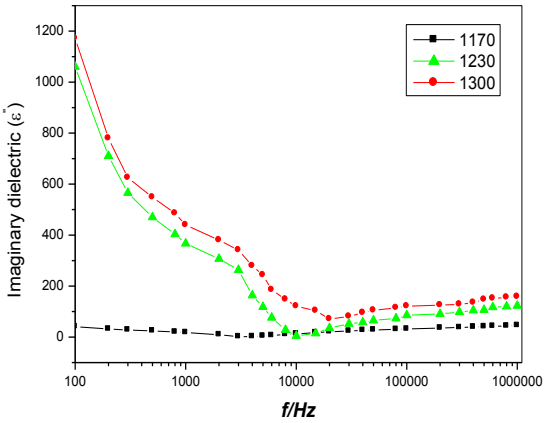

Fig. 11. Variation of the imaginary dielectric constant (ε'') with frequency for PLT ceramic sintered at (1170, 1230 and 1300) at room temperature

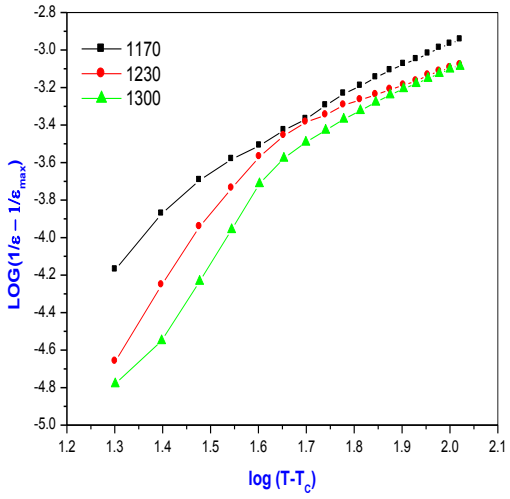

Fig. 12. A plot of $\log(1/\varepsilon - 1/\varepsilon_{max})$ versus $\log(T - T_c)$ for PLT ceramics, with sintering temperature (1170, 1230 and 1300 ^{o}C).